SHELLAC.

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ABSTRACT.

A description is given of the source, manufacture, uses, and common methods of testing shellac. It is shown that the generally accepted iodine value method for determining rosin in flake shellac may be subject to very large errors, and this method can not be applied to cut shellac. The amount of material soluble in a light petroleum distillate and the acid number of this material are shown to be quite constant for pure shellac, and a method of determining adulteration, both by rosin and other substances, which can be as easily applied to cut shellac as to flake shellac, has been developed. Suggested specifications for pure orange flake shellac and orange shellac varnish are given.

CONTENTS.

	Page.
I. Introduction	277
II. Source, preparation, and use of shellac.....	278
1. Properties of orange shellac	279
2. Uses of shellac.....	280
III. The methods in use for testing shellac.....	281
1. The detection of adulterants in shellac.....	281
IV. A new method for detecting adulteration in orange shellac.....	283
1. Modification of the McIlhiney method for the detection and estimation of adulterants in dry shellac and shellac varnish....	284
V. Material insoluble in alcohol.....	292
1. Method for the determination of material insoluble in hot alcohol.	293
VI. The determination of nonvolatile matter in shellac varnish.....	294
VII. The color of orange shellac.....	295
VIII. Suggested specification for pure orange shellac.....	296
IX. Suggested specification for pure orange shellac varnish.....	296

I. INTRODUCTION.

The need for a publication on shellac by the Bureau of Standards has been shown by the numerous inquiries which have been received in recent years. The continued high price of shellac during and after the World War has been the cause of the appearance of an unusual number of adulterated shellac varnishes on the market.

There are certain standard methods¹ for the estimation of rosin and of material insoluble in alcohol in dry orange shellac, but these methods are not applicable to the examination of shellac varnishes for adulteration.

¹ See A. S. T. M. Standards, 1921, p. 663.

A brief summary is given in this paper of the source, preparation, and uses of shellac, together with a description of certain methods which are used at the Bureau of Standards for the examination of both flake shellac and shellac varnish.

II. SOURCE, PREPARATION, AND USES OF SHELLAC.

Shellac is a manufactured material produced from lac, the excretion of the East Indian insect "*Tachardia Lacca*." The young insects swarm twice a year, alight on the twigs of various species of trees on which they feed, and pierce the bark with their beaks. The assimilated sap excreted by the insect soon dries, and the multitude of tiny animals, becoming cemented to the twig, lay myriads of minute eggs and ultimately die. In a short time the eggs burst forth into life and the swarm of young insects appear on the branches like blood-red dust. Generation after generation dwell upon the same twig until the lac coating often attains a thickness of half an inch.

In May and June, and again in October and November, the incrustated twigs are gathered by natives and broken into short pieces. These are the stick lac of commerce. Prior to the introduction of synthetic dyestuffs stick lac was collected, primarily, for the production of the red lac dye, and shellac was a by-product. Lac dye is now obsolete, and the stick lac is collected only for the manufacture of shellac.

The stick lac is crushed, washed to remove most of the dye, and dried, the so-called seed or grain lac resulting. The grains are ruby red and about the size of wheat. Shellac is usually made by melting dry seed lac with approximately 0.05 to 0.25 per cent of arsenic sulphide (orpiment), with or without the addition of small amounts of rosin, in long narrow bags over a charcoal fire. The softened lac mixture is forced through the bag by twisting it. The material forced through the bag is then softened again and made into thin sheets which are broken into the familiar flakes of the shellac of commerce.

The grading of shellac is based upon freedom from rosin and dirt and upon the color of the flakes. Rosin has been the most common adulterant of shellac because of its low price and especially because it greatly facilitates the melting of the seed lac. The best grades of shellac are free from rosin, but material containing from 3 to 5 per cent of rosin is a common grade and for many purposes serves as well as the pure material. Lower grades of shellac frequently contain even higher percentages of rosin.

The addition of orpiment to shellac seems to be regular practice, and some authorities have claimed that arsenic sulphide causes a vulcanizing effect analogous to the action of red sulphide of antimony on rubber. A portion, at least, of the orpiment remains unchanged in the shellac and tends to give the flakes a lighter appearance. This effect is apparent only in the flake shellac, because on dissolving the flakes in alcohol the orpiment settles out slowly and its tinting power is thereby lost.

Garnet lac is a product usually made from the residue of impure lac left in the bag after the squeezing process. The sand and other impurities are usually eliminated by dissolving this impure lac in alcohol or dilute sodium carbonate solution, filtering, and then driving off the solvent or liberating the lac from the alkaline solution by acid, respectively. The dark-colored product is fused into lumps of various sizes. Button lac is essentially shellac fused into a mass the shape of a button. Bleached shellac is manufactured by dissolving orange shellac in dilute sodium carbonate solution and bleaching with sodium hypochlorite solution. The red lac dye is thereby destroyed and the shellac is then precipitated by mineral acid as an amorphous yellowish-white mass. White shellac prepared in this way is quite sensitive to heat and must be dried with care at rather low temperatures, or it may become partially or wholly insoluble in alcohol. Bleached shellac which has been stored for a time tends to become more or less insoluble in alcohol.

1. PROPERTIES OF ORANGE SHELLAC.

The specific gravity of orange shellac varies from 1.08 to 1.13, owing to minute air bubbles inclosed during its manufacture. The melting point is indefinite, and decomposition starts if shellac is heated much above 100° C. It is insoluble in dilute mineral acids, but dissolves readily in ammonia or solutions of sodium carbonate, borax, and other alkalis. It dissolves (with the exception of a small amount of natural wax and mineral matter) in cold methyl or ethyl alcohol or a mixture of these two (denatured alcohol). It should be noted that high-proof alcohol must be used. Ethyl or denatured alcohol much weaker than 95 per cent by volume does not dissolve shellac perfectly. Shellac is also soluble in isopropyl and amyl alcohols (fusel oil) but is quite insoluble in petroleum hydrocarbons and turpentine, a property possessed to the same degree by no other common resin soluble in alcohol. Orange shellac is very stable and does not depreciate in quality even during long storage.

2. USES OF SHELLAC.

Shellac has a diversity of uses in the industries. Orange and bleached shellac are components of composition articles, such as buttons, phonograph records, imitation ivory, poker chips, sealing wax, and many similar products. Shellac is used in making electrical instruments, in various types of insulation, in fireworks and explosives, and as a binder in abrasive wheels. One of the largest single uses for shellac is in the vehicle of the anticorrosive and antifouling paints specified by the Navy Department and the Shipping Board for the protection of the hulls of steel vessels.

Shellac varnish is very commonly used as a quick-drying coating for floors, furniture, interior decoration, musical instruments, toys, etc. It is extensively used for stiffening straw hats, for insulating and waterproofing, in various leather dressings and polishes, and upon willow and rattan goods. The resistance of a dried film of shellac to turpentine and turpentine substitute (mineral spirits) is of great value where shellac is employed as a first coating to fill the pores of wood and cover resinous knots and streaks, because the varnish or wax polish applied afterwards does not soften the shellac. This valuable property is not possessed by most of the so-called shellac substitutes upon the market.

So-called standard shellac varnish is made by dissolving 5 pounds of the resin in 1 gallon of special denatured alcohol (U. S. Internal Revenue Bureau formula No. 1 for special denatured alcohol, used in making shellac varnish, consists of 100 gallons ethyl alcohol 190° proof and 5 gallons of approved methyl alcohol (wood alcohol)). Commercial shellac varnish usually contains a smaller percentage; 4 to 4½ pounds of shellac per gallon of alcohol is a common proportion. As already noted, shellac requires alcohol of high proof for its solution. The formulas for completely denatured alcohol of the United States Internal Revenue Bureau in every case call for "100 gallons ethyl alcohol not less than 160° proof," mixed with small amounts of materials like kerosene or pyridine bases which render the mixture nonpotable. Alcohol of 160° proof is not strong enough to properly dissolve shellac, and since the maker of completely denatured alcohol is not required to use ethyl alcohol stronger than 160° proof it follows that the ordinary denatured alcohol purchased in the open market is generally unsuitable for mixing with shellac. The specially denatured alcohol (formula No. 1) should be used in shellac when it is obtainable, otherwise completely denatured alcohol, 190° proof, should be employed.

In making shellac varnish the weighed resin in flake form should stand overnight in a closed vessel with the proper volume of 190° proof alcohol. The mixture should then be stirred at intervals until no more lumps are present. Mechanical stirring devices are usually employed in factories.

Shellac varnish dries only through evaporation of the solvent. In humid weather it may happen that the rapid evaporation of alcohol from the film will sufficiently lower the temperature to cause condensation of moisture. This may turn the film white temporarily, but eventually the coating should dry to its normal color. A shellac film does not possess the waterproof qualities and resistance to weather of a good long oil spar varnish, and hence is not suitable for outside exposure. If it were possible to prepare an oil varnish from shellac and linseed or tung oil, it should be an excellent product, owing to the hardness and permanence of this resin; but unfortunately shellac will not mix with such drying oils or with turpentine.

III. METHODS IN USE FOR TESTING SHELLAC.

1. THE DETECTION OF ADULTERANTS IN SHELLAC.

It has been recognized for some time that the regular methods in use for testing shellac are in many cases inadequate to show that given samples are unadulterated. Shellac varnishes have been more difficult to examine for adulterants than flake shellac.

For many years the purity of a given sample of shellac varnish has been decided upon the results of a qualitative test for rosin. The test commonly used is the familiar Liebermann-Storch reaction, in which a solution of a sample containing rosin in acetic anhydride should give a fugitive lavender color with sulphuric acid of specific gravity 1.53.

Livache and McIntosh² state that the Liebermann-Storch test gives a decided response when shellac contains as much as 10 per cent of rosin, but only a doubtful test may result when 5 per cent of this adulterant is present.

Some experimental work done at this bureau indicates that powdered rosin, which has become partially oxidized through exposure to air, will not give a positive test by the Liebermann-Storch reaction. It was found that an oxidized sample of rosin will, however, give a purple color by the Halphen-Hicks reaction.

² *Manufacture of Varnishes*, 3, p. 306.

The reagent consists of two solutions: (A) 1 part by volume of phenol dissolved in 2 parts of carbon tetrachloride, and (B) 1 part by volume of bromine in 4 parts of carbon tetrachloride. The most convenient procedure is the following: Dissolve a small quantity of the substance to be tested in 1 to 2 cc of solution A. Pour this solution into one of the cavities of an ordinary porcelain color-reaction plate and fill an adjacent cavity with solution B. Cover the plate with a watch glass and note whether the bromine vapors develop a purple or blue color in the solution of the sample to be tested.

The quantitative estimation of rosin in dry orange shellac by a determination of the Wijs iodine number was recommended in 1907 by a subcommittee on shellac of the American Chemical Society. This method was made a tentative standard by the American Society for Testing Materials in 1914 and was adopted as standard in 1917.³ It has been extensively used but is open to a number of criticisms. The method depends upon the fact that pure shellac resin normally shows a very low iodine absorption value (18 is arbitrarily taken as the maximum figure), while rosin shows an abnormally high value (arbitrarily taken as 228). When a sample of shellac is found to have an iodine value greater than 18, a calculation by a simple proportion is made to estimate the percentage of rosin present.

One of the most serious objections to the method is the fact that it is not applicable to shellac varnishes. The presence of alcohol interferes with the iodine number determination, while removal of all traces of alcohol by heating a sample of shellac varnish tends to change the iodine value of the shellac. The determination of the Wijs iodine value of shellac requires unusual care and skill in order to obtain reliable results. There are samples of shellac with an iodine number as low as 14, and it is conceivable that an addition of as much as 2 per cent of rosin could be made to such a sample without increasing the iodine value above the allowable limit for pure shellac. At the same time this small percentage of rosin might be undetectable by a qualitative test of the sample. There is evidence that the rosin used to adulterate shellac tends to decrease in iodine value with increased age, due to partial oxidation. A sample of T. N. shellac containing rosin, which showed a Wijs iodine value of 30.4 in 1912, altered in a sealed bottle so that its iodine value in 1921 was found to be 26.8. F. P. Veitch,⁴ of the Bureau of

³ See A. S. T. M. Standards, 1921, p. 663.

⁴ Private communication.

Chemistry, Department of Agriculture, has recently noted that the change in iodine number of rosin exposed to the air in powdered form is more rapid than commonly supposed, and that marked changes often take place in less than a month's time.

From another point of view the estimation of rosin from the iodine number of a sample of shellac is not theoretically sound. It can be shown that the iodine numbers of rosin and of manila gum (a resin sometimes used to adulterate shellac) are dependent upon the weight of sample taken for the test. Several determinations of the iodine numbers of rosin and manila gum were made, using different weights of sample with all other conditions unchanged. The determinations were made by the Standard A. S. T. M.⁵ method, using 20 cc of the Wijs solution and allowing the reaction to proceed for one hour in the dark at 21–23° C.

TABLE 1.—Wijs Iodine Values of Rosin and Manila Resin with Varying Weights of Sample.

Manila sample No. 1.		Manila sample No. 2.		Rosin (WW grade).	
Weight of sample taken.	Iodine value.	Weight of sample taken.	Iodine value.	Weight of sample taken.	Iodine value.
0.2004	126.8	0.2054	127.1	0.1446	185.0
.1944	128.5	.1948	130.4	.1258	195.5
.1550	135.5	.1754	136.3	.1029	216.7
.1469	138.0	.1698	136.0	.1016	220.1
.1033	151.0	.1003	160.6	.0571	249.5
.0979	155.3	.0988	161.1	.0476	255.0

IV. A NEW METHOD FOR DETECTING ADULTERATION IN ORANGE SHELLAC.

It is apparent that some new method is needed which can be applied to both flake shellac and shellac varnish for the detection and estimation of adulterants like rosin and manila gum. Some years ago Langmuir made the observation that pure shellac is very nearly insoluble in petroleum ether, while rosin dissolves readily in this solvent. McIlhiney⁶ developed a method for the quantitative estimation of rosin in shellac, based on the above observation, but apparently the method has not been used to any extent.

A study of McIlhiney's method led to certain changes which shortened and simplified the procedure and finally resulted in a modified method by which it is believed to be possible to detect adulteration and give quality ratings for both dry shellac and shellac varnish.

⁵ See A. S. T. M. Standards, 1921, p. 663.
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⁶ J. Am. Chem. Soc., 30, p. 867; 1908.

1. MODIFICATION OF THE McILHINEY METHOD FOR THE DETECTION AND ESTIMATION OF ADULTERANTS IN DRY SHELLAC AND SHELLAC VARNISH.

Petroleum ether distilling between 55 and 75° C.⁷ was obtained by the distillation on the steam bath of either commercial petroleum ether or aviation gasoline, fighting grade.⁸ Glacial acetic acid was diluted with water until its solidification point was between 13 and 14° C.

METHOD—Accurately weigh 2 g of flake shellac (± 0.1 g) and transfer to a 2-liter Florence flask, the neck of which has a volume somewhat over 100 cc. In case of a shellac varnish, determine the percentage of nonvolatile matter (see p. 294). Place a portion of the well mixed sample in a stoppered container. Weigh the container and sample. Transfer a weight of the sample corresponding as closely as practicable to 2 g of nonvolatile matter to the flask already described and restopper the container. Weigh the container again and by difference calculate the exact weight of the portion transferred to the flask. Calculate the exact weight of shellac in the sample taken from the percentage of nonvolatile matter in the varnish. The procedure from this point is the same for dry and cut shellac. Add 20 cc of the special acetic acid and heat the flask until the shellac resin and wax are dissolved. With certain shellac adulterants there may be a small amount of resin which can not be dissolved. Cool the flask to room temperature (19–21° C.), whereupon a part of the natural shellac wax usually separates. Add slowly from a pipette 50 cc of the petroleum ether cooled to 19–21° C., with constant shaking of the flask, allowing one to two minutes for the addition. Add all at once from a graduated flask 100 cc more of the petroleum ether kept at 19–21° C., stopper the flask, and shake vigorously. While shaking the flask, slowly add tap water kept at 19–21° C., until the shellac has separated as an amorphous mass. Half fill the flask with water and agitate so as to thoroughly wash the ether layer. Add water until the petroleum ether layer nearly fills the neck of the flask, cork and let the flask stand until the ether layer is free from suspended particles. Transfer 100 cc of the ether layer to a 100 cc graduated flask. It is convenient to fit the large flask with a two-hole stopper with tubes arranged like a wash bottle, so that the ether solution can be blown into the graduated flask.

⁷ A fraction with initial boiling point of 40° C. should be satisfactory, provided it is not used in extremely hot weather.

⁸ Bureau of Mines Technical Paper No. 323, p. 1.

Evaporate the 100 cc of petroleum ether solution, portion wise if necessary, in a small weighed Erlenmeyer flask on a hot plate. When the dry point is reached, suck out the residual solvent vapors, cool the flask, and weigh. The weight of the residue multiplied by 150 and divided by the weight of shellac taken is the percentage of "matter soluble in petroleum ether." Dissolve this residue in 25 cc of a mixture of equal volumes of 95 per cent denatured alcohol and benzol (the mixture should be previously titrated to a faint pink color with dilute alkali, using phenolphthalein as an indicator) and titrate in the cold with 0.1 *N* alcoholic sodium hydroxide with phenolphthalein as an indicator. Calculate the acid number of the "matter soluble in petroleum ether" (milligrams of KOH required for 1 g of petroleum ether residue). Transfer most of the petroleum ether layer remaining in the Florence flask to a small beaker or flask and evaporate to dryness. Test this residue for rosin by means of the Halphen-Hicks test already referred to. Report a faint purple or blue coloration as "faint test for rosin" and a deep purple or blue as "decided test for rosin."

A series of orange shellac samples was examined by the modified McIlhiney method as outlined, and the analytical data are given in Table 2. The samples numbered Con. 10479 to 10495 were collected in 1911 by representatives of the Bureau of Chemistry, United States Department of Agriculture, at the United States customs-house, New York, N. Y. The samples numbered B. S. 70939 to 70951 were obtained in 1921 from a large importer of shellac in New York, N. Y. The iodine value of each sample by the Wijs method, a qualitative test for rosin on the original sample and on the petroleum ether residue, and a calculation of the percentage of rosin from the Wijs iodine value of the adulterated samples are also given in Table 2.

TABLE 2.—Data on Orange Shellac.

Trade name of shellac.	Identification number.	Matter soluble in petroleum ether.	Acid number of petroleum ether soluble material.	Iodine number of shellac.	Qualitative test for rosin on shellac.	Qualitative test for rosin on petroleum ether residue.	Rosin calculated from iodine number.	Classification according to Table 3.	Material insoluble in hot denatured alcohol.
D. C.	Con. 10479.	Per cent. { 6.4 } { 6.1 }	{ 65.5 } { 66.5 }	{ 15.6 } { 16.2 }	Negative.	Negative.	Per cent. { 0.73 } { 1.072 }	Pure.	{ 0.68 } { 1.32 }
Ralli superfine.	Con. 10480.	6.3 } 6.4 }	69.5 } 70.5 }	17.9 } 17.4 }	do.	do.	{ 1.32 } { 1.39 }	do.	{ 1.32 } { 1.43 }
Ralli No. 3.	Con. 10481.	6.4 } 6.3 }	68.5 } 69.0 }	17.1 } 17.0 }	do.	do.	{ 1.39 } { 1.40 }	do.	{ 1.39 } { 1.43 }
Ralli.	Con. 10482.	8.0 } 7.7 }	93.0 } 91.5 }	20.4 } 21.2 }	Positive ²	Positive ²	{ 1.30 } { 1.35 }	Slightly adulterated.	{ 1.30 } { 1.35 }
T. N.	Con. 10483.	9.8 } 9.8 }	99.5 } 100.5 }	28.2 } 28.1 }	do ³	do ³	{ 2.86 } { 2.50 }	{ Somewhat a adulterated.	{ 2.86 } { 2.48 }
D. C.	Con. 10484.	6.5 } 6.7 }	63.5 } 65.0 }	16.6 } 16.6 }	Negative.	Negative.	{ .84 } { .94 }	Pure.	{ .84 } { .94 }
R. P. special.	Con. 10485.	6.3 } 6.3 }	79.0 } 76.5 }	16.2 } 16.4 }	do.	do.	{ 1.10 } { 1.11 }	do.	{ 1.10 } { 1.11 }
Ralli superfine No. 1.	Con. 10486.	6.0 } 6.2 }	74.0 } 70.5 }	17.9 } 17.9 }	do.	do.	{ 1.59 } { 1.55 }	do.	{ 1.59 } { 1.57 }
Ralli fine No. 3.	Con. 10487.	6.2 } 6.3 }	67.0 } 64.0 }	17.5 } 17.5 }	do.	do.	{ 1.42 } { 1.46 }	do.	{ 1.42 } { 1.44 }
Ralli good No. 9.	Con. 10488.	6.6 } 6.7 }	70.0 } 70.0 }	17.7 } 17.8 }	do.	do.	{ 1.72 } { 1.75 }	do.	{ 1.72 } { 1.74 }
Pure T. N.	Con. 10489.	7.9 } 7.7 }	56.5 } 59.0 }	16.3 } 16.7 }	do.	do.	{ 2.13 } { 2.05 }	Suspicious.	{ 2.13 } { 2.09 }
D. C.	Con. 10490.	6.6 } 6.6 }	67.0 } 67.0 }	16.0 } 15.9 }	do.	do.	{ .85 } { .86 }	Pure.	{ .85 } { .86 }
Ralli superfine.	Con. 10491.	7.5 } 7.2 }	84.5 } 84.0 }	21.0 } 20.6 }	Positive ²	Positive ²	{ 1.43 } { 1.51 }	Slightly adulterated.	{ 1.43 } { 1.47 }
V. S. O.	Con. 10492.	6.2 } 6.1 }	70.0 } 71.0 }	16.5 } 16.8 }	Negative.	Negative.	{ .89 } { .91 }	Pure.	{ .89 } { .90 }
Ralli fine.	Con. 10493.	6.5 } 6.7 }	70.5 } 71.0 }	18.1 } 18.5 }	do.	do.	{ 1.34 } { 1.35 }	do.	{ 1.34 } { 1.35 }
Ralli good.	Con. 10494.	6.5 } 6.5 }	66.0 } 65.0 }	17.7 } 18.1 }	do.	do.	{ 1.92 } { 1.98 }	do.	{ 1.92 } { 1.95 }
T. N.	Con. 10495.	11.3 } 11.1 }	107.5 } 107.0 }	26.9 } 27.3 }	Positive ²	Positive ²	{ 4.4 } { 4.4 }	{ Somewhat a adulterated.	{ 4.4 } { 4.4 }

Ralli.....	B. S. 70939.....	{ 8.4 } { 8.2 } { 8.4 } { 8.2 }	{ 83.0 } { 88.0 } { 90.0 } { 92.0 }	{ 85.5 } { 17.2 } { 19.1 } { 20.0 }	{ 18.0 } { 17.6 } { 19.6 } { 15.0 }	Negative..... Positive ² do. ³ do. ³	do.....do.....do.....do.....	{ 2.02 } { 2.07 } { 1.87 } { 1.86 }	2.05 1.87
Ralli fine.....	B. S. 70940.....	{ 8.4 } { 8.2 } { 7.9 } { 8.0 }	{ 83.0 } { 88.0 } { 90.0 } { 92.0 }	{ 85.5 } { 17.2 } { 19.1 } { 20.0 }	{ 18.0 } { 17.6 } { 19.6 } { 15.0 }	Negative..... Positive ² do. ³ do. ³	do.....do.....do.....do.....	{ 2.02 } { 2.07 } { 1.87 } { 1.86 }	2.05 1.87
Ralli good.....	B. S. 70941.....	{ 8.4 } { 8.2 } { 7.9 } { 8.0 }	{ 83.0 } { 88.0 } { 90.0 } { 92.0 }	{ 85.5 } { 17.2 } { 19.1 } { 20.0 }	{ 18.0 } { 17.6 } { 19.6 } { 15.0 }	Negative..... Positive ² do. ³ do. ³	do.....do.....do.....do.....	{ 2.02 } { 2.07 } { 1.87 } { 1.86 }	2.05 1.87
Diamond I.....	B. S. 70942.....	{ 7.7 } { 7.3 } { 6.5 } { 6.7 }	{ 97.0 } { 102.5 } { 83.0 } { 84.0 }	{ 100.0 } { 18.9 } { 15.4 } { 15.1 }	{ 18.8 } { 18.9 } { 15.3 } { 15.3 }	Negative..... Positive ² do. ³ do. ³	do.....do.....do.....do.....	{ 1.53 } { 1.44 } { 1.30 } { 1.21 }	1.49 1.25
V. S. O.....	B. S. 70943.....	{ 7.7 } { 7.3 } { 6.5 } { 6.7 }	{ 97.0 } { 102.5 } { 83.0 } { 84.0 }	{ 100.0 } { 18.9 } { 15.4 } { 15.1 }	{ 18.8 } { 18.9 } { 15.3 } { 15.3 }	Negative..... Positive ² do. ³ do. ³	do.....do.....do.....do.....	{ 1.53 } { 1.44 } { 1.30 } { 1.21 }	1.49 1.25
Ralli superfine.....	B. S. 70944.....	{ 7.7 } { 7.3 } { 6.5 } { 6.7 }	{ 97.0 } { 102.5 } { 83.0 } { 84.0 }	{ 100.0 } { 18.9 } { 15.4 } { 15.1 }	{ 18.8 } { 18.9 } { 15.3 } { 15.3 }	Negative..... Positive ² do. ³ do. ³	do.....do.....do.....do.....	{ 1.53 } { 1.44 } { 1.30 } { 1.21 }	1.49 1.25
U. S. A. T. N.....	B. S. 70945.....	{ 9.3 } { 9.5 } { 7.5 } { 7.4 }	{ 91.0 } { 88.0 } { 72.5 } { 74.0 }	{ 89.5 } { 20.8 } { 20.4 } { 20.0 }	{ 18.7 } { 20.4 } { 20.2 } { 20.0 }	Negative..... Positive ² do. ³ do. ³	do.....do.....do.....do.....	{ 1.60 } { 1.59 } { 1.59 } { 1.59 }	1.60 1.60
Pure T. N.....	B. S. 70946.....	{ 7.5 } { 7.4 } { 6.9 } { 7.1 }	{ 72.5 } { 73.0 } { 76.0 } { 72.5 }	{ 73.0 } { 14.1 } { 14.7 } { 14.4 }	{ 20.2 } { 14.1 } { 14.7 } { 14.4 }	Negative..... Positive ² do. ³ do. ³	do.....do.....do.....do.....	{ 1.60 } { 1.59 } { 1.59 } { 1.59 }	1.60 1.60
D. C.....	B. S. 70947.....	{ 7.1 } { 7.3 } { 6.1 } { 6.1 }	{ 76.0 } { 72.5 } { 75.5 } { 77.0 }	{ 73.0 } { 14.1 } { 14.7 } { 14.4 }	{ 20.2 } { 14.1 } { 14.7 } { 14.4 }	Negative..... Positive ² do. ³ do. ³	do.....do.....do.....do.....	{ 1.60 } { 1.59 } { 1.59 } { 1.59 }	1.60 1.60
Grade A Navy.....	B. S. 78813.....	{ 7.3 } { 7.3 } { 6.1 } { 6.1 }	{ 76.0 } { 72.5 } { 75.5 } { 77.0 }	{ 73.0 } { 14.1 } { 14.7 } { 14.4 }	{ 20.2 } { 14.1 } { 14.7 } { 14.4 }	Negative..... Positive ² do. ³ do. ³	do.....do.....do.....do.....	{ 1.60 } { 1.59 } { 1.59 } { 1.59 }	1.60 1.60
Angelo T. N.....	B. S. 78814.....	{ 6.1 } { 6.1 } { 11.5 } { 11.1 }	{ 86.0 } { 85.5 } { 111.5 } { 112.5 }	{ 86.0 } { 85.5 } { 112.0 } { 112.5 }	{ 18.4 } { 18.4 } { 26.8 } { 26.7 }	Negative..... Positive ² do. ³ do. ³	do.....do.....do.....do.....	{ 1.60 } { 1.59 } { 1.59 } { 1.59 }	1.60 1.60
Committee sample T. N.....		{ 11.1 } { 11.1 } { 11.1 } { 11.1 }	{ 111.5 } { 112.5 } { 112.5 } { 112.5 }	{ 112.0 } { 112.5 } { 112.5 } { 112.5 }	{ 26.8 } { 26.8 } { 26.8 } { 26.8 }	Positive ² Positive ² Positive ² Positive ²	do.....do.....do.....do.....	{ 1.60 } { 1.59 } { 1.59 } { 1.59 }	1.60 1.60

¹ Extraction by A. S. T. M. method (see p. 292).² Faint test for rosin.³ Decided test for rosin.

An examination of the results given in Table 2 indicate that a large number of the samples of shellac were free from rosin, as shown by a qualitative test and by an iodine value below the arbitrary accepted limit of 18. Such samples can probably be safely considered as unadulterated shellacs. These samples of pure shellac, with few exceptions, yielded values between the limits of 6.0 and 7.0 per cent for material soluble in petroleum ether and acid values for this residue between the limits of 60 and 90. This material soluble in petroleum ether is believed to be chiefly a natural shellac wax, which is stated by Livache and McIntosh⁹ to be present to the extent of approximately 6 per cent in shellac and is composed of ceryl and myricyl alcohols, partly in combination with fatty and resinous acids.

These limiting values are believed to be characteristic of pure shellac. Every sample in Table 2 which showed a positive test for rosin yielded a petroleum ether residue greater than 7.0 per cent, and the acid number of the residue in nearly every case was higher than in the case of the pure shellacs. A sample of pure shellac (Con. 10492) was intentionally adulterated by grinding a weighed sample with 5.1 per cent of its weight of lump rosin, WW grade. This mixture showed a petroleum ether residue of 11.2 and an acid value for the residue of 114. Since this sample before adulteration contained 6.2 per cent of material soluble in petroleum ether, it was evident that the increase in percentage of material soluble in petroleum ether was practically equal to the percentage of rosin added to the sample. The marked increase of the acid value of the above residue due to the rosin is also worthy of note.

It appeared that a good approximation of the percentage of rosin in a given sample of adulterated shellac could be obtained by a simple subtraction of the normal percentage of material soluble in petroleum ether for pure shellac from the observed value in case of a rosin-bearing sample.

A complication was introduced when the adulteration of shellac by partially oxidized rosin or by manila resin was considered. A sample of WW rosin, fine enough to pass a 100-mesh sieve, which had been exposed to the air for over a year, was examined by the modified McIlhiney method. A petroleum ether residue of 48.4 per cent was obtained, with an acid value for this residue of 153.0. Lump rosin of the same grade, which

⁹ *Manufacture of Varnishes*, 3, p. 305.

was unappreciably oxidized owing to the small surface exposed to the air, was examined in the same manner and showed a petroleum ether soluble residue of 98.8 per cent and an acid number for this residue of 160.0. Two different samples of manila resin on hand in the laboratory, the histories of which were unknown, were also examined. They showed petroleum ether residues of 46.5 and 60.1 per cent, with acid values for the residues of 146 and 134.5, respectively. A mixture of 19 parts by weight of pure shellac (Con. 10492) and 1 part by weight of manila resin (showing 60.1 per cent of material soluble in petroleum ether) was found to give a value of 9.0 per cent material soluble in petroleum ether and an acid value of 105 for this residue.

From a consideration of all these data it was concluded that if a sample of shellac contains unoxidized rosin as an adulterant, the material soluble in petroleum ether of the sample will exceed the percentage 7.0 by a figure closely approximating the percentage of adulterant present. If a sample of shellac is appreciably adulterated by partially oxidized rosin or by manila resin, the difference between the percentage of material soluble in petroleum ether found and 7.0 per cent will represent a minimum figure for adulteration, and in many cases the real adulteration may be twice or, in extreme cases, three times this minimum figure. The consideration of the acid value of the residue soluble in petroleum ether of samples of shellac under examination may give valuable information. This value for a normal pure shellac lies between 60 and 90. Any marked departure from these limits should place a sample under suspicion. In general, adulteration with acid resins like rosin or manila gum should tend to increase this value above the normal, while neutral resins or waxes, or resins of low acid value like coumarone, paraffin, and ester gum (rosin glyceride), respectively, would tend to lower the acid value of the petroleum ether residue below the normal for pure shellac. In Table 3 will be found a suggested method for rating samples of shellac from the data of the modified McIlhiney method.

TABLE 3.—Suggested Method for Rating Samples of Shellac.

Petroleum ether soluble percentage.	Rosin test.	Acid number of residue.	Purity.
7.0 or less.....	Negative.....	60-90.....	Pure.
7.0-8.0.....	Positive.....	May be greater or less than 90.	Slightly adulterated; probably 1-2 per cent rosin, as in "Superfine."
7.0-8.0.....	Negative.....	do.....	Suspicious; no adulteration proved.
8.0-12.0.....	Positive.....	Generally, but not always over 90.	Somewhat adulterated; probably a "T. N." shellac.
8.0-12.0.....	Negative.....	do.....	Somewhat adulterated with an adulterant other than rosin.
12.0-20.0.....	Positive or negative.	do.....	Badly adulterated; possibly as high as 40 per cent adulteration.
Over 20.0.....	do.....	do.....	Grossly adulterated; possibly no shellac present.

In the next to the last column of Table 2 may be found the ratings of the various samples of shellac obtained by the application of the suggested method in Table 3. The modified McIlhiney method for the determination of material soluble in petroleum ether in shellac was developed with particular reference to orange shellac and orange shellac varnishes. In Table 4 are some data on samples of white shellac, garnet, button, stick, and seed lac, and several commercial orange shellac varnishes. It is worthy of note that the sample of orange shellac (Con. 10492), when cut in alcohol to make a shellac varnish, gave the same percentage of material soluble in petroleum ether (6.2) as was obtained with the sample in flake form.

TABLE 4.—Data on Shellac Varnishes, Bleached Shellac, Garnet Lac, etc.

Trade name of sample.	Identification number.	Matter soluble in petroleum ether.	Acid number of petroleum ether residue.	Iodine number of shellac.	Qualitative test for rosin on original sample.	Qualitative test for rosin on petroleum ether residue.	Classification according to Table 3.
		Per cent.					
V. S. O. cut-in alcohol.....	Con. 10492	{ 6.1 } 6.2	{ 64.0 } 66.0		Negative..	Negative..	Pure.
Orange shellac varnish ¹	B. S. 79511	{ 24.5 } 25.0	{ 136.5 } 136.5		Positive ²	Positive ² ..	Grossly adulterated.
Do. ¹	B. S. 79566	{ 8.8 } 9.0	{ 33.0 } 32.5		Negative..	do. ²	Somewhat adulterated.
Do. ¹	B. S. 73626	{ 10.8 } 10.9	{ 90.0 } 91.0			do. ²	Do.
Do. ¹	B. S. 79750	{ 12.6 } 13.0	{ 95.0 } 94.5		Positive ³	do. ²	Badly adulterated.
White shellac varnish ¹	B. S. 63058	{ 13.3 } 14.0	{ 24.5 } 23.0			do. ³	Do.
Dry white shellac ¹	B. S. 67839	{ 7.6 } 7.9	{ 87.0 } 88.0	{ 11.3 } 11.1	Negative..	do. ³	Slightly adulterated.
Garnet Lac.....	B. S. 70948	{ 13.5 } 12.2	{ 150.5 } 149.0	{ 39.8 } 40.0	Positive ²	do. ²	Badly adulterated.
"C" Garnet Lac.	B. S. 78815	{ 5.8 } 5.6	{ 111.5 } 114.0	{ 18.9 } 18.8	Negative..	do. ²	Very slightly adulterated.
Button Lac.....	B. S. 70951	{ 7.8 } 7.7	{ 83.5 } 79.0	{ 13.8 } 15.5	do.....	Negative..	
Stick Lac.....	B. S. 70949	{ 11.9 } 12.1	{ 99.0 } 97.5	{ 16.9 } 18.2	do.....	do.....	
Seed Lac.....	B. S. 70950	{ 7.4 } 7.3	{ 81.5 } 78.5	{ 14.4 } 15.0	do.....	do.....	

¹ Sample submitted to bureau for test by another branch of the Government.² Decided test for rosin.³ Faint test for rosin.

TABLE 5.—Examination of Commercial Orange Shellac Varnishes.

Num- ber.	Vola- tile.	Pounds per gal- lon.	Petro- leum ether residue.	Acid num- ber.	Rosin.	Turpentine film test.	Rating of sample.
	Per ct.		Per ct.				
3.....	62.1	4.00	8.9	125.2	Very positive.....	Unaffected..	Somewhat adulterated.
5.....	59.9	4.42	16.3	128.0	Positive.....	do.....	Badly adulterated.
9.....	55.0	5.40	37.3	126.5	do.....	Removed....	Grossly adulterated.
13.....	68.5	3.04	15.2	129.5	do.....	Unaffected..	Badly adulterated.
15.....	57.4	4.91	25.5	139.1	Very positive.....	Softens.....	Grossly adulterated.
17.....	55.8	5.21	9.3	53.4	Positive.....	Unaffected..	Somewhat adulterated.
21.....	62.2	4.02	33.1	147.3	do.....	Removed....	Grossly adulterated.
24.....	59.4	4.51	58.2	95.8	do.....	do.....	Do.
29.....	59.7	4.45	16.0	130.2	do.....	Unaffected..	Badly adulterated.
33.....	64.2	3.69	44.2	114.7	do.....	Removed....	Grossly adulterated.
40.....	64.4	3.65	44.4	129.5	do.....	do.....	Do.
51.....	44.7	8.18	44.9	80.5	do.....	do.....	Do.
56.....	49.0	6.87	43.2	110.6	do.....	Unaffected..	Do.
59.....	53.0	5.85	37.7	105.8	do.....	do.....	Do.
60.....	56.5	5.08	27.9	108.0	do.....	do.....	Do.
63.....	54.1	5.58	51.5	110.8	do.....	Removed....	Do.
67.....	41.5	9.20	43.8	122.2	do.....	do.....	Do.
68.....	58.8	4.62	36.6	143.5	do.....	do.....	Do.
74.....	58.6	4.66	7.4	39.1	Slight.....	Unaffected..	Slightly adulterated.
75.....	56.8	5.01	28.2	126.1	Positive.....	Removed....	Grossly adulterated.
79.....	59.5	4.49	16.1	122.5	do.....	Unaffected..	Badly adulterated.
80.....	64.1	3.69	16.5	125.3	do.....	do.....	Do.
81.....	33.3	13.25	46.1	132.1	Very positive.....	Removed....	Grossly adulterated.
87.....	65.5	3.48	9.7	29.1	Positive.....	Unaffected..	Somewhat adulterated.
88.....	56.7	5.05	7.4	70.7	Negative.....	do.....	Suspicious; probably pure.
89.....	60.0	4.40	46.3	123.8	Positive.....	Removed....	Grossly adulterated.
90.....	52.9	5.88	44.2	116.0	do.....	do.....	Do.
91.....	46.0	6.60	50.0	133.1	do.....	do.....	Do.
92.....	70.3	2.79	42.6	143.5	do.....	do.....	Do.
99.....	65.3	3.51	7.2	65.3	Slight.....	Unaffected..	Practically pure.
100.....	66.5	3.32	8.3	53.4	Negative.....	do.....	Somewhat adulterated.
102.....	53.5	5.74	30.8	147.5	Positive.....	Removed....	Grossly adulterated.
103.....	51.0	6.34	37.1	150.2	do.....	do.....	Do.
104.....	57.1	4.95	5.0	63.0	Negative.....	Unaffected..	Pure.
106.....	56.9	5.00	25.0	162.0	Positive.....	do.....	Grossly adulterated.
108.....	57.1	4.95	29.4	153.4	do.....	Removed....	Do.
110.....	59.1	4.56	8.7	109.8	Negative.....	Unaffected..	Somewhat adulterated.
112.....	52.2	6.06	32.3	147.0	Positive.....	Removed....	Grossly adulterated.
113.....	51.2	6.30	35.1	151.0	do.....	do.....	Do.
114.....	61.3	4.16	6.8	43.0	Negative.....	Unaffected..	Slightly suspicious; prob- ably pure.
117.....	55.7	5.25	5.3	82.8	Slight.....	do.....	Very slightly adulterated.
118.....	55.0	5.40	6.9	67.3	do.....	do.....	Do.
119.....	56.6	5.06	4.2	97.1	Negative.....	do.....	Suspicious; probably pure.
120.....	54.9	5.43	6.1	77.0	Slight.....	do.....	Very slightly adulterated.
121.....	56.2	5.15	6.7	63.7	Negative.....	do.....	Pure.
122.....	59.7	4.46	13.6	120.5	Positive.....	do.....	Badly adulterated.
124.....	62.8	3.91	6.4	57.6	Negative.....	do.....	Pure.
125.....	59.3	4.53	10.7	28.6	Positive.....	Softens.....	Somewhat adulterated.
126.....	59.6	4.47	10.4	58.4	do.....	Unaffected..	Do.
130.....	60.2	4.36	11.8	105.8	Very slight.....	do.....	Do.
131.....	54.9	5.43	14.1	114.1	Positive.....	do.....	Badly adulterated.
135.....	63.8	3.74	6.5	46.0	Positive (slight).....	do.....	Very slightly adulterated.
136.....	62.1	4.02	7.4	72.1	Very slight.....	do.....	Do.
138.....	60.2	4.36	15.2	120.8	Negative.....	do.....	Badly adulterated.
140.....	59.4	4.51	8.8	70.8	Positive.....	do.....	Somewhat adulterated.
141.....	64.0	3.71	6.7	61.6	Negative.....	do.....	Pure.

The modified McIlhiney petroleum-ether method has been used recently by H. A. Gardner, of the Institute of Paint and Varnish Research, Washington, D. C., in the examination of a series of orange shellac varnishes. The turpentine film test was made by

flowing the various samples on strips of clean tin plate, allowing the coating to dry for one day, immersing the panels in turpentine overnight, and noting the condition of the film. With his permission, the data obtained by him on these samples are given in Table 5.

In the last column of Table 5 are found ratings of the samples according to the method of Table 3. It will be observed that a large number of the samples were found to be badly or grossly adulterated, but the conclusion should not be drawn that these samples are necessarily representative of shellac varnishes upon the market, because it is believed that many of the samples were selected for test because of suspected adulteration.

In every case where the dried film of a sample of shellac varnish was reported as removed in the turpentine film test the examination of the sample indicated gross adulteration. Some samples that were found to be grossly adulterated were reported to be unaffected in the turpentine film test, and for this reason this qualitative test is of value only where it is positive. When a dried film of shellac varnish is removed by immersion overnight in turpentine, it is believed to be safe to say that the sample is badly adulterated. It might be preferable to make this test upon a baked film of shellac—for example, a film that has been baked at 100 to 105° C. for two hours.

V. MATERIAL INSOLUBLE IN ALCOHOL.

Shellac normally contains a small percentage of mineral matter, partly dirt and partly orpiment added during the process of manufacture. The Navy Department requires in its specification for grade A shellac a content of not more than 1.75 per cent of material insoluble in hot 95 per cent ethyl alcohol and similarly not more than 3 per cent of insoluble matter in their grade B shellac. No method for making the determination of the material insoluble in alcohol is given in the Navy Department specification.

Committee D-1 on preservative coatings of the American Society for Testing Materials adopted in 1917 a standard method for determining the material insoluble in alcohol in orange shellac.¹⁰ In this method a weighed sample of shellac is extracted for two hours in a paper cartridge in a Knoefler extraction apparatus with hot 99.5 per cent methyl alcohol. At different times difficulty has been experienced at the Bureau of Standards with this method, and it has been learned that other laboratories have had difficulty

¹⁰ A. S. T. M. Standards, 1921, p. 665.

in obtaining satisfactory agreement in duplicate determinations by this method. In general, the results are higher when 99.5 per cent methyl alcohol is used than with 95 per cent specially denatured alcohol under the same conditions.

The regulations of the U. S. Internal Revenue Bureau, together with the high price of methyl alcohol as compared with denatured alcohol have practically restricted the solvent for commercial shellac varnishes to the specially denatured alcohol (formula No. 1). It seems logical that the determination of the material insoluble in alcohol should be made with this solvent, composed of a mixture of 100 gallons of 190° proof ethyl alcohol and 5 gallons of approved wood (methyl) alcohol.¹¹

A method for matter insoluble in alcohol in shellac, used for a number of years in Government laboratories¹² was selected because of its simplicity as compared with the A. S. T. M. method. Determinations of material insoluble in alcohol were made on most of the shellac samples described in Table 2, using the specially denatured alcohol previously described.

1. METHOD FOR THE DETERMINATION OF MATERIAL INSOLUBLE IN HOT ALCOHOL.

Thoroughly grind a representative sample of at least 20 g of shellac in a mortar, accurately weigh about 2 g and transfer to a small beaker. (In the case of a shellac varnish thoroughly mix the sample, weigh an amount containing approximately 2 g of nonvolatile residue, and transfer to a beaker.) Heat the sample with 25 cc of 95 per cent specially denatured alcohol (U. S. Internal Revenue Bureau formula No. 1) until the resin and wax are in solution. Draw a small volume of boiling alcohol through a weighed Gooch crucible (prepared in the usual way) and at once filter the boiling shellac solution, using suction. Transfer the insoluble material from the beaker to the crucible with hot alcohol from a wash bottle, using a "policeman," if necessary. Wash the residue in the crucible with boiling alcohol until the washings are colorless, and then wash three times more, nearly filling the crucible each time (it will be necessary to shut off the suction momentarily to fill the crucible). Wash off any film of shellac on the bottom or sides of the crucible and dry to constant weight at 105–110° C. The weight of the residue multiplied by 100 and divided by the weight of the sample represents the percentage of material insoluble in hot alcohol.

¹¹ Regulations No. 61 of the U. S. Internal Revenue Bureau.

¹² B. S. Misc. Pubs. No. 15.

The samples of orange shellac described in Table 2 were examined by the Gooch crucible method for material insoluble in alcohol and the results obtained are given in the last column of that table. It is worthy of note that the shellac samples which were rated as pure contained on the average appreciably less material insoluble in hot alcohol than the samples rated as adulterated or suspicious. Only one sample of the 14 "pure" shellacs yielded over 1.75 per cent of material insoluble in alcohol, while 11 samples showed less than 1.50 per cent. Only 3 samples of the 12 adulterated or suspicious shellacs showed less than 1.50 per cent of material insoluble in alcohol, while 5 samples showed over 2.00 per cent.

The samples of shellac numbered Con. 10479 and Con. 10484, both pure materials of a grade known as D. C., offered difficulty in the filtration on a Gooch crucible. Some constituent of the insoluble material tended to clog the asbestos and hinder the filtration. These samples were submitted to a continuous extraction in an apparatus of the Knoefler type. The extraction proceeded very slowly with both samples, eight hours being necessary for completion. The results for material insoluble in alcohol on these two samples given in Table 2 were obtained by a continuous extraction for eight hours with the 95 per cent specially denatured alcohol.

It is believed that samples of orange shellac will seldom be found that will offer difficulty in the filtration upon a Gooch crucible. It is recommended, in case such samples are encountered, that determinations of material insoluble in alcohol be made with a continuous extraction apparatus of the Knoefler type, using 95 per cent specially denatured alcohol (U. S. Internal Revenue Bureau formula No. 1). In such cases, extraction should be continued for one hour after the alcohol runs colorless through the thimble.

VI. THE DETERMINATION OF NONVOLATILE MATTER IN SHELLAC VARNISH.

The following method is recommended for the determination of the percentage of nonvolatile matter in shellac varnish. Place a portion of the well-mixed sample in a stoppered bottle or weighing pipette. Weigh the container and sample. Transfer 1.5 g (± 0.5 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 transferred to the weighed dish. Heat the dish and its

contents in an oven maintained at 100–105° C. (212–221° F.) 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. To calculate the ratio of shellac to alcohol in a shellac varnish in terms of pounds of resin to a gallon of solvent, it is necessary to know the percentage of nonvolatile residue and the specific gravity at 15.5° C. of the alcohol. Distill a sample of the shellac varnish nearly to the dry point, but not far enough to decompose the shellac residue. Determine the specific gravity of the distillate at 15.5° C./15.5° C. by a hydrometer or any other accurate method. Calculate the weight of a gallon of this distillate by multiplying the specific gravity by 8.33 (weight of a gallon of water at 15.5° C.). Calculate the pounds of shellac cut in a gallon of alcohol (y) from the following formula:

$$y = \frac{a \times b}{100 - b}$$

where a is the weight in pounds of a gallon of the alcohol and b is the percentage of nonvolatile matter in the sample. For example, a shellac varnish is found to contain 40.0 per cent of nonvolatile matter, and the distillate is found to have a specific gravity of 0.816 at 15.5° C./15.5° C. (95 per cent alcohol).

$$a = 0.816 \times 8.33 = 6.80 \text{ pounds}$$

$$y = \frac{6.80 \times 40}{100 - 40} = 4.53 \text{ pounds per gallon}$$

Where only approximate values are required, it can be safely assumed that the alcohol in a shellac varnish weighs 6.8 pounds per gallon.

VII. THE COLOR OF ORANGE SHELLAC.

Normal samples of orange shellac contain small percentages of mineral matter, wax, and inclosed bubbles of air. As previously mentioned, a small amount of orpiment is usually added in the manufacture of shellac. The shellac flakes are therefore not transparent to light, and for this reason color standards for comparison with the flakes are not practicable. The apparent light color of some samples of orange shellac may be misleading where such effect is caused by air bubbles or the use of orpiment. When such a sample is dissolved in alcohol, the orpiment tends to slowly settle, the inclosed air bubbles are released, and the resulting solution may be darker than the solution of a flake shellac containing less inclosed air and orpiment and apparently of a darker shade when in flake form.

VIII. SUGGESTED SPECIFICATION FOR PURE ORANGE SHELLAC.

Orange shellac must be in flake form, and any flakes which have stuck together must separate readily under hand pressure. When tested by the modified McIlhiney method, a sample must not show a residue of material soluble in petroleum ether greater than 7.0 per cent, and the acid number of this residue must be between the limits of 60 and 90.

The residue of material soluble in petroleum ether must give a negative test for rosin with the Halphen-Hicks reagent. The residue of material insoluble in hot 95 per cent denatured alcohol (U. S. Internal Revenue Bureau formula No. 1) must not exceed 1.75 per cent.

IX. SUGGESTED SPECIFICATION FOR PURE ORANGE SHELLAC VARNISH.

Orange shellac varnish must contain not less than 40.0 per cent by weight, of nonvolatile matter, the volatile matter to be specially denatured alcohol of 190° proof (U. S. Internal Revenue Bureau formula No. 1). The film of shellac on clean, bright tin, after drying over night, must be as hard as and resist the action of turpentine as well as a varnish made from pure orange flake shellac and 190° proof specially denatured alcohol.

When tested by the modified McIlhiney method, a sample must not show a residue of material soluble in petroleum ether greater than 7.0 per cent, based upon the weight of nonvolatile matter in the sample, and the acid number of this residue must be between the limits of 60 and 90.

The residue of material soluble in petroleum ether must give a negative test for rosin with the Halphen-Hicks reagent.

The residue of material insoluble in hot 95 per cent specially denatured alcohol must not exceed 1.75 per cent, based upon the weight of nonvolatile matter in the sample.

WASHINGTON, December 20, 1922.