"Protected with color and pigment and var
To the uppermost tip of the topgallant spar."

PAPERS ON PAINT AND VARNISH

AND THE MATERIALS USED IN THEIR MANUFACTURE

By

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PREFACE.

In the present work the author brings up to date the series of technical papers which he has prepared as circulars of the Educational Bureau of the Paint and Varnish Manufacturers Association of the United States, since January, 1919, covering his researches in the technology of paint and varnish.

Every thinking individual must be impressed with the importance in everyday life of the protective coatings which are depended upon to preserve wealth and health, and to add to the beauty of our surroundings. It is hoped that this volume will bring a fuller realization of the importance of surface protection and a better understanding of how to obtain and maintain it.

The author wishes to acknowledge the contributions to the work by his various colleagues, and the helpful suggestions of the members of the Educational Bureau as well as the members of the Paint and Varnish Associations.

Washington, D. C., November 1, 1920.

TO THE MEMBERS OF THE
EDUCATIONAL BUREAU
PAINT MANUFACTURERS ASSOCIATION OF THE UNITED STATES
NATIONAL VARNISH MANUFACTURERS ASSOCIATION
(CO-OPERATING)
THIS BOOK IS DEDICATED

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PAPERS ON PAINT AND VARNISH

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CHAPTER I

Resume of Soya Bean Oil Investigations

Soya bean oil has become one of the staples of the paint trade. Due to the rapidly climbing imports and increased domestic production, quantities are being made available for more extended use. It is believed that an immediate presentation of summarized data on soya bean oil, as previously published by the writer, in circular form, may be of interest at this time.

In Circular No. 50, entitled "Soya Oil in Paints," published in June, 1917, the following information was presented:

> For many years the makers of high-grade prepared paints have used pure linseed oil as a liquid for such paints, adding the usual small amount of drier and thinner required to form a practical paint. Whenever rosin oil, gloss oils, or neutral petroleum base oils have been used as such, partially or wholly to replace linseed oil in such paints, disastrous results may follow. There is, however, a class of pure vegetable oils produced from seed in the same manner as linseed oil, which have a thoroughly legitimate use in the paint industry. Of these oils, soya bean oil is the most prominent. From the standpoint of resistance to exposure when used in paints, it compares favorably with linseed oil, but on account of its somewhat lower drying value, it does not become as hard, and has, therefore, a tendency to hold particles of dust present in the

atmosphere. The retention of such dust causes a white paint made with soya oil to assume a slightly darker appearance than a similar paint made with linseed oil. It has been found, however, that the use of additional drier overcomes this tendency to a great extent.

While it is possible that the use of soya oil in paints is contemplated by manufacturers who are unable, on account of the present flax shortage, to obtain sufficient linseed oil for their requirements, it is probable that this oil will always be used in admixture with preponderating amounts of linseed oil. When used in this fashion, its purpose would be to assist in overcoming the present shortage rather than to lower the price of the product in which it is used, since soya oil sells at a price substantially the same as that of linseed oil.

Suggestions have been made for the use of paint oils containing from 2 to 3 parts of linseed oil mixed with one part of soya oil. Such oils would doubtless give very good results, and when used, the amount of drier may be slightly increased, so that the soya oil paint shall contain one-third more drier than the amount used for linseed oil. Lead and manganese driers such as are now used with linseed oil also give excellent results with soya oil. Cobalt driers are, however, even more efficient with soya oil and can be used in many cases to advantage. Some results on drying tests with soya oil are presented in the latter part of this circular.

One of the most promising uses for soya oil is for the grinding of oil colors. When used for this purpose, soft pastes are obtainable which do not become hard even upon long standing. Moreover, the amount of soya oil that would be introduced into a white paint base as a result of the use of such colors would be extremely small, since relatively small amounts of color are required for making most tints or shades.

A series of extensive tests was made at Washington, by the writer, to determine the wearing value of various oils when used with a standard pigment mixture consisting of white lead, zinc oxide and a moderate percentage of inert pigment. In these tests, several mixtures containing soya bean oil were included. The results of the tests on these paints after exposure for various periods of time are shown in Table I. Repainting tests covering nearly three years exposure were made on each paint.

RESUME OF SOYA BEAN OIL INVESTIGATIONS

TABLE 1

Panel.	Formula.	Year.	Color.	Chalking.	Checking.	General Film Condition.
2.	100% Soya Bean Oil	1912	Darkened	Very slight	Slight	Very good
		1913	Very dark	Medium	Medium	Good
		1914	Fairly white	Considerable	Medium	Fairly good
		*R-1916	Slight darkening	Medium	Medium	Fairly good
		*R-1917	Fairly white	Slight	Medium	Fairly good
10	50% Raw Linseed Oil 50% Soya Bean Oil	1912	White	Medium	Very slight	Good
		1913	Fairly white	Medium	Very slight	Good
		1914	White	Medium	Very slight	Good
		*R-1916	White	Medium	Slight	Fairly good
		*R-1917	White	Medium	Medium	Fairly good
18	75% Raw Linseed Oil 25% Soya Bean Oil	1912	Fairly white	Medium	Slight	Good
		1913	Fairly white	Considerable	Very slight	Fairly good
		1914	White	Medium	Slight	Fair
		*R-1916	White	Medium	Medium	Fairly good
		*R-1917	White	Slight	Medium	Fairly good
27	50% Blown Linseed Oil 50% Soya Bean Oil	1912	Very white	Slight	Slight	Good
		1913	White	Slight	Medium	Good
		1914	White	Medium	Medium	Good
		*R-1916	White	Considerable	Very slight	Very good
		*R-1917	White	Slight	Slight	Good

*R—abbreviation for "Repainting Tests"

POSSIBLE PRODUCTION:

Since it has been found that soya beans have a higher percentage of digestible protein* than practically any other food, and can be used in the production of soya flour, milk and other valuable food products, it is probable that a very large acreage will be planted this year. The crop of seed may be extracted or pressed for its oil content at any of the cottonseed mills in the South. The oil may be used in the paint and soap industries and the press cake in the manufacture of various food products of great value.

Composition of Missouri Soya Beans Crushed at The Institute of Industrial Research

	No. 635.	No. 1219.	No. 2253.
Oil	18.63%	15.00%	18.94%
Cake	81.37%	85.00%	81.06%
	100.00%	100.00%	100.00%

Analysis of Oils

	No. 635.	No. 1219.	No. 2253.
Iodine Number	132.1	135.2	127.3
Acid No.	0.4	0.4	0.4
Saponification No.	192.2	192.3	191.6
Specific Gravity	.916	.915	.915

Analysis of Cake (oil-free)

	No. 635.	No. 1219.	No. 2253.
Nitrogen	6.98%	6.96%	7.51%
Nitrogen = Protein	39.68%	39.65%	42.71%
Potash (K_2O)	2.52%	2.12%	2.18%
Soda (Na_2O)	.10%	.14%	.13%
Phosphoric acid (P_2O_5)	2.06%	2.07%	2.24%

The writer has examined a number of representative samples of the oil that have come into this country, and results obtained are shown in Table 2.

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*See Bull. 312, Ohio Agric. Exp. Station.

TABLE 2

Chemical Characteristics of Soya Bean Oil

Sample No.	Specific gravity.	Acid No.	Saponification No.	Iodine No.	Per cent of foots.
1	0.9233	1.87	188.4	127.8	3.81
2	0.9240	1.92	188.3	127.2	......
3	0.9231	1.90	187.8	131.7	......
4	0.9233	1.91	188.4	129.8	......
5	......	......	......	130.0	......
6	......	......	......	132.6	......
7	......	......	......	136.0	......
Av.	0.9234	1.90	188.2	130.7	......

It is evident that the iodine value of soya bean oil is the only chemical characteristic that markedly differentiates it from linseed oil. Therefore, in the detection of soya bean oil and its estimation, the iodine values of several samples of mixed oils are given, as being of interest in this connection.

TABLE 3

Iodine Values of Linseed Oil and Mixed Oils

Sample No.	Straight linseed. North American.	Soya. 25 per cent. Linseed. 75 per cent.	Soya. 50 per cent. Linseed. 50 per cent.	Soya. 75 per cent. Linseed. 25 per cent.
1	190.3	175.2	160.7	140.4
2	189.5	175.9	161.7	140.8
3	188.0	175.4	160.3	139.0
Av.	189.3*	175.5	160.9	140.4

A series of tests were conducted to determine the rate of drying of soya oil treated with lead and manganese driers. A definite quantity of oil was placed in weighed friction-top can covers, which were reweighed after receiving the oil, to determine the amount used for the test in each case. The tin covers containing the oil were then placed in a large box under definite temperature control and humidity for a certain period. Weighings were made at different periods, and the increase in weight, due to absorption of oxygen, was calculated to percentages.

Tables 4, 5 and 6 give the results obtained. The time of drying was somewhat longer than with linseed oil. Later tests show that driers prepared from cobalt resinate or linoleate are much more efficient than lead or manganese driers for drying soya oil.

TABLE 4

Soya Bean Oil and Lead Drier

Per cent. PbO.		0.05	0.10	0.30	0.50	0.70	1.00	1.30	1.60
Per ct. gain	1 day	....	0.07	0.63	1.34	1.05	1.53	0.93	1.35
	3 days	....	0.07	3.52	4.31	2.75	4.86	4.82	4.12
	5 days	....	0.09	5.04	6.06	6.09	6.75	6.66	5.52
	12 days	....	....	6.88	7.34	7.43	7.76	7.32	6.47
	15 days	....	....	8.84	8.93	8.59	8.81	8.44	7.46
	20 days	0.05	0.20	9.02	9.08	8.90	9.03	8.65	7.83

---

*South American flax often produces an oil having an Iodine value of about 175.

TABLE 5

Soya Bean Oil and Manganese Drier

Per cent. MnO_2.		0.01	0.05	0.15	0.26	0.30
Per cent. gain........	1 day........	....	....	0.02	0.02	0.01
	10 days........	....	5.06	6.48	6.10	5.97
	20 days........	0.05	9.07	8.80	6.78	6.51

TABLE 6

Soya Bean Oil, Manganese and Lead Drier

Per cent. PbO.		0.20	0.30	0.50
Per cent. MnO_2.		0.05	0.15	0.25
Per cent. gain........	1 day	3.04	3.77	3.74
	8 days....................	5.96	6.43	6.47
	12 days....................	6.33	6.78	6.67

The writer recently submitted to a number of investigators on a sub-committee of Committee D-1, American Society for Testing Materials, several samples of commercial soya bean oils for the examination and determination of their suitability for use in paints. The following directions were also submitted for guidance in the testing work:

Heat Test.—Heat 2 oz. of the oil at 450° F. in a glass receptacle until bleaching is noticed. Then blow a slow current of dry air through the oil until the specific gravity has increased to .950. This may require a period of 7 hours. The blowing should be conducted at a temperature between 300° and 370° F. The oil should become light and fairly rapid drying.

The heat tests to which the soya oils were subjected gave interesting results, but did not in any case seem to increase the drying value of the oils, and this indicates the necessity of using the same amount of driers for heated as for raw soya oils. It is fairly well established by the tests, however, that most soya oils will bleach under the heat treatment and become viscous.

In the determination of the analytical constants of soya oil, results which agree very closely were obtained by nearly all of the observers.

Average Constants of Nine Samples of Raw Soya Bean Oil as Taken from the Results of Nine Observers

Specific gravity	0.9247
Saponification number	192.0
Iodine number	134.5
Acid number	2.14

In Circular No. 63, entitled "Legitimization of Soya Bean Oil," published in June, 1919, the following statements were made:

> The rapidly growing production of soya bean oil in the United States is to a very great extent due to the early work of the Educational Bureau. During the past year there has been produced in North America over five million gallons of this oil, and an additional large quantity has been imported.
>
> Exposure tests have shown that 20% of soya bean oil may be mixed with 80% of raw linseed oil to produce a paint liquid that will wear quite as well as pure linseed oil. The above mixture will show an iodine number (index of drying value) of approximately 170 when North American linseed oil is used as the base. In the writer's opinion, the above mixture (with an iodine value not less than 170) may be used with successful results in high grade paints. The adoption of this mixture for at least many types of paint in which it is customary to use linseed oil wholly, would be suggested. There should be no hesitation in disclosing, where analysis labels are now used, the actual composition of this liquid, as it is a thoroughly legitimate product which has proved satisfactory in scientific and practical tests.
>
> Soya bean oil mixtures are excellent for the grinding of certain paste tinting colors. The raw oil is of lighter color than raw linseed oil, and may be made even lighter by refining. Soya oil grinds easily with pigments and produces pastes that remain soft and less liable to stiffening in the package than those made with pure linseed oil. The amount of soya oil introduced into a house paint, for instance, through the use of painter's paste tinting colors made on the above basis, would be extremely small and would not have any substantial influence upon the drying of the paint. Because of the above properties of soya oil, it would appear that it is an oil that is even more desirable than straight linseed oil for color grinding, and that colors produced with this oil should really have a higher sales value than those ground wholly in raw linseed oil.
>
> Soya oil has a higher flash point than any other vegetable oil used in the paint industry. It may be heat treated and blown to a viscous form. Its value in var-

nish making has already been indicated, and it is probable that it will soon be established firmly in the industry. Its further use is suggested.

Further data on Soya Bean Oil in Paste Colors is given in an article presented by the writer on January 15, 1920, before the Pennsylvania State Association of Master Painters. This article was entitled "Livering of Paste Colors" and extracts therefrom are presented below:

> It may be well to describe an example of the livering conditions that have been observed by some painters. For instance, a paste made of linseed oil and pigments is at first found soft, smooth, and easily reducible with oil. Subsequently the paste becomes stiff, gelatinous, and hard to break up. The initial cause of this condition may be due to the formation and solution of certain products resulting from chemical and physical reactions between the pigments and the liquids. The reaction which brings about the initial effect is in many instances dependent upon free acid in the oil or upon the presence of certain portions of the liquid that are readily oxidizable. Normal linseed oil, which is substantially neutral, seldom causes any trouble, but occasionally oil may contain much foots which, in turn, holds considerable amounts of water. The water will hydrolize or split up oil into free fatty acid. The rapidity of this action depends upon the amount of water, intimacy of the mixture which exposes the oil to greater surface action, and to temperature. The acids formed react with the color pigment until the liquids become saturated with the products that evidence their presence by a thickened condition. When these products reach a certain concentration, or when their physial condition is disturbed, they separate or precipitate. Similar conditions have been observed when a poorly designed paint liquid of high resin content held in unstable solution by inferior processing of solvents, is added to an oil color. Small amounts of rosin or other highly acid resinous substances, or acid driers may also be active causes which contribute to the hardening effect. The complexity of the problem is, however, evidenced by the fact that large quantities of rosin, large quantities of free acid, or large quantities of water may sometimes entirely prevent livering.

Without pursuing a technical description of the phenomenon of livering, it is apparent that foots-free and substantially acid-free oils or thinning liquids should be used by painters for thinning linseed oil paste colors. Let us also consider an allopathic treatment even more advanced for eliminating the livering ailment.

The grinding of colors in oil is a difficult task. Each individual fine grain of pigment must be thoroughly wetted with the oil in order to get a smooth paste. All these pigments are characterized by surface tension which tends to resist wetting. Great pressure developed by properly run mills is necessary to overcome this force. Different liquids differ in their wetting power, and the surface tension of the grinding liquid must bear a certain relation to the surface tension of the color pigment, if a smooth paste is to result. While bodied oils may have a greater tension than raw oils and for this reason are sometimes used in small percentage to assist in grinding colors and to increase color strength by dispersion, we are at present dealing with the ordinarily used raw oils. Soya bean oil, for instance, has been found to have a much greater wetting power than linseed oil, and a color mill running on a soya paste color can turn out with lower power consumption much more wetted color than one running on a linseed paste. Furthermore, the resulting soya oil paste almost invariably will show a much softer condition. This is to some extent due to the fact that soya bean oil is substantially free of those oxidizable acid glycerides of the linolinic type. Moreover, soya bean oil, because of its chemical make-up, prevents the precipitation of the particles that may be in some type of solution. Consequently such pastes, if well packed, will remain very soft.

The fact that raw soya oil is not as good a drying oil as linseed oil will at once occur to you as a reason for questioning the use of paste colors ground with soya oil. While this objection might apply where a solid color paste is reduced with varnish for making an enamel, it should be remembered that the amount of tinting color added to a white paste to produce nearly any tint and most light shades of interior or exterior paint, is very small. As such color in oil generally contains less than 25% of oil, the balance being pigment, it will be seen that the amount of soya oil added to the paint would be extremely small and insufficient to assert any non-drying tendency. For instance, to produce a fairly substantial tint of yellow, blue, gray or green, the amount of oil color added to a white paint would in most instances run not more than 3% or 4% of the total. If soya oil is used in these paste tinting colors, there would be present in the paint not more than 1% of soya bean oil, which is evidently insufficient to cause any detraction in the drying powers of the paint.

Because of the fact that blacks such as are used for solid color work require such extremely large amounts of oil for grinding and seldom show the hardening conditions above referred to, painters will probably continue to prefer such colors ground in linseed oil. This might also apply to lake colors.

Soya bean oil also has possibilities as a medium for grinding red lead. At present it is sometimes difficult to secure paste red lead in linseed oil that will keep indefinitely. While some samples obtainable may remain soft for long periods of time, other samples may become hard. With soya oil, however, a soft paste results, that has excellent keeping properties. The writer recently ground some red lead in the proportion of 87½% dry red lead and 12½% soya bean oil. This paste was thinned for brushing according to the following formulae:

No. 1.

Paste Red Lead	20 lbs.
Raw Linseed Oil	4½ lbs.
Drier	2 gills
Turpentine	2 gills

No. 2.

Paste Red Lead	20 lbs.
Linseed Oil (boiled)	5 pints

When applied to metal surfaces, these paints dried to touch in 12 hours, without showing sagging. They became fairly hard at the end of 24 hours. While this time of drying is not as rapid as may be shown by red lead ground in linseed oil and thinned in the same manner, it might be considered satisfactory. While no recommendation at present is made for the use of such red lead pastes, the experiments recorded above are at least suggestive.

In conclusion, it is suggested that manufacturers produce a line of soya bean oil tinting colors and that painters become acquainted with these products for their requirements.

In Circular No. 60, entitled "Changes in Oil Upon Storage," a series of analyses were presented, showing the changes taking place in various oils upon storage. An extract from this circular, showing the results on soya oil, is given below. It will be noted that the changes taking place over the long period of storage are not great, indicating that the oil is stable and not subject to rapid decomposition.

> During the early part of 1911 the writer secured a quantity of a number of commercial oils for use in experimental paints that were to be exposed to the weather to determine the efficiency of various oil mixtures as paint ingredients. After the painting tests were made, samples of the pure oils were placed in pint glass bottles having ground glass stoppers. The bottles were well filled; an air space above the oil of not more than one inch being allowed in any instance. The oils were placed upon a shelf in the laboratory where they were exposed to indirect light and to ordinary room temperature (in the summer not over 105° F. and in the winter not less than 35° F.). In November, 1914, portions of the oils were removed from the bottles and examined as a check against the original determinations. Air was of course admitted during this procedure. During September, 1916, the oils were again examined; further quantities being removed for this purpose. The bottles were again placed upon the shelf and allowed to remain there until March, 1919, when further quantities were removed for examination. The results obtained on these samples are given below:

TABLE 7

Soya Bean Oil—					
March, 1911	.924	129.	189.	2.3	....
November, 1914	.925	130.2	193.1	4.7	1.4813
September, 1916	.937	122.0	192.1	7.0	1.4733
February, 1919	.939	121.7	193.4	7.8	1.4721

In Circular No. 37, June, 1916, there was reprinted an address by Mr. L. P. Nemzek, Special Technical Representative of the Educational Bureau, before the Mississippi Cottonseed Crushers' Association, at New Orleans. Extracts from this interesting circular, which outlined the early work carried on by the Educational Bureau to stimulate the production of soya bean oil in this country, are given below:

> As early as 1907 the Bureau interested itself in a campaign to promote the increased production of flaxseed in the United States. The rapidly increasing demand for linseed oil, by the industries of this country, indicated that we should soon face a shortage of this commodity. It remained for the 1910 flaxseed crop failure to demonstrate what a menace to the paint trade such a shortage meant. Due almost entirely to the resultant scarcity of linseed oil, the prices commenced to soar during the latter part of 1910. Linseed oil reached the high price of $1.00 per gallon during 1911 and high prices prevailed throughout most of the year 1912. This condition forcibly showed the necessity for a more profound investigation of oils which might find application as substitutes for linseed in the different industries which use the oil in large quantities. In view of the fact that the work which was done toward promoting an increased production of flaxseed had been so marvelously effective, it was decided to do work of similar magnitude with miscellaneous oils.
>
> Soya oil was one of the oils decided upon for the experiments largely because it was already available in quantities and could readily be imported. After looking into the matter it was found that the production of it might be developed on a satisfactory basis in this country. Soya beans were already being grown in considerable quantities, but up to that time domestic oil on a commercial scale had not been produced.
>
> Soya oil is crushed from the bean bearing that name. The bean is commonly referred to by the agriculturists in this country as "Soy," but the oil, ever since it was first imported, has been known as "Soya Bean Oil" or "Soya Oil." Largely for the sake of convenience the title "Soya Oil" has been used. The title "Soya" was, very likely, introduced from Manchuria, where the bean is grown in enormous quantities, and is a derivation from the Japanese "Shoyu" or "Soja." "Soya" is replacing "Soy" when used in reference to the bean as such, even in the language of the agriculturist, and it is only a question of time when the word "Soya" will be adopted generally.

The first step in connection with the Bureau's endeavor consisted of the importation of soya beans from Manchuria for oil-extraction tests and the distribution of this seed, together with seed from a number of varieties already grown in this country, and the soliciting of the assistance of the State Agricultural Experiment Stations and other interested persons to urge the farmer to grow more soya beans for seed. While soya beans had been grown in this country for a good many years, this was done as a forage crop and for fertilization purposes. Only enough seed was being harvested to take care of the succeeding year's planting. The far-reaching propaganda, through the active co-operation of the State Agricultural Experiment Stations and the different Bureaus of the U. S. Department of Agriculture, was solely for the purpose of inducing the farmer to increase his production.

It was agreed that the best way to reach the farmer is through the State Agricultural Experiment Stations, as these institutions give the matter expert attention from a local standpoint and have proved to be the most valuable sources of information the farmer enjoys. They are capable of giving the results of their work the greatest publicity, which was a big factor to be considered. Their Bulletins carry a prestige which no other means of publicity along this line can develop. For this reason more than anything else it was considered of the greatest importance to obtain the co-operation of the Agricultural Experiment Stations.

While growing tests during 1911 and 1912 were limited to North Dakota, Minnesota, Missouri, New Jersey and Kentucky, they were extended in 1913 to every State, with the exception of a very few, where conditions are unfavorable from an agricultural standpoint.

The tests were also extended to the Philippine Islands and to several places in Canada. It is noteworthy in this connection that there are at least two varieties which can be successfully matured as far north as the southern portions of Quebec.

During 1912, forty-eight varieties were experimented with, including four imported from Manchuria. This number was greatly increased in 1913, so that all available types would be included. It was considered likely that varieties previously discarded as unsuitable for forage purposes may prove desirable for seed production, so new varieties were included in the oil extraction tests whenever brought to our attention.

In view of the fact that there are hundreds of varieties, many of which differ in only a few minor respects from others, it was necessary, after making preliminary tests, to eliminate many of them in order to bring the number down to a practical working basis. There is really nothing to be gained by growing so many different varieties. The number has been reduced to about fifteen already popular varieties, namely: Mammoth, Medium, Yellow, Ito San, Holly Brooks, Haberlandt, Peking, Wilson, Auburn, Black Eye Brow, Arlington, Tokio, Mikado, Virginia, Chiquita, Sable and the Yellow Manchurian bean, which growing tests have shown to be very satisfactory. In time some of these will be eliminated, so that the number will gradually be brought down to four or five varieties which prove to be the most satisfactory for forage purposes as well as for the production of oil.

No sooner were the farmers interested in the larger growth of soya beans than they began to ask for information as to how and where the beans could be disposed of to be converted into oil.

It was soon evident that the cotton seed mills in the South were best situated for crushing the beans. The location of the hundreds of these mills is fortunate, inasmuch as the South is naturally adapted to become the great soya bean-producing section of the country. Definite plans have not been evolved in sections where the cotton seed mills are not located, but in the northern section of the country the linseed oil mills will, very likely, crush the beans as soon as the production is large enough to warrant their going into the matter. In some sections, notably Tennessee, it was suggested that the farmers in a certain locality club together and conduct their own mill, disposing of the oil in the regular manner, and each farmer to use the cake from his crop for feeding purposes. In this way there would be returned to the land the full fertilizing values of the crop. The promoters of successful agriculture see in this a means to increase the production of soya beans, mainly because of the value which the farmer obtains by the improvement of his soil.

During August and September, 1913, I made a trip of nearly twenty-four thousand miles, visiting most of the Agricultural Experiment Stations, to discuss matters relating to the increased production of beans for oil-crushing purposes and to determine whether or not the cotton seed mills were in a position to handle the crop without materially altering the machinery they use for the crushing of cotton seed or going to the expense of installing new equipment. Most of my time was spent in the States growing cotton and where cotton seed mills were already established. From the information obtained at that time I felt safe in stating in my 1914 report to the Association that the cotton seed mills were equipped to crush the beans with few minor and inexpensive changes in their equipment. Since that time reports from different mills have varied somewhat, but they are all more or less agreed that the bean can be successfully crushed and that the cost of changes to accommodate this crop need not be counted on as an item of expense.

You are naturally interested most in the crushing of the bean, what they can be bought for, and how the oil and the by-product can be disposed of profitably.

An important detail of the work that has been done so far is the determination of the oil-bearing properties of different varieties of beans and an examination of the oils, particularly for drying properties. Hundreds of laboratory crushings have been made, and it was found that the oil content varies from 16 to 25 per cent. One of the interesting facts which was developed in connection with the extraction of the oil is the relation of the physical structure of the soya bean to the amount of oil it contains and the release of this oil by expression. It was found that of two varieties containing exactly the same percentage of oil one would yield its oil by expression a great deal easier than another; in some cases, although two varieties contained the same amount of oil, one would yield, by expression, not nearly as much of it as another. A careful microscopic study of the structure of the oil cells of the different varieties of the beans may lead to the development of a variety which is particularly adapted for crushing purposes.

The average oil content of soya beans is 19 per cent. While some varieties will uniformly run a little higher, two lots of the same variety will vary from 18 to 21 per cent., while occasionally a lot may run a little below 18 per cent.

During the past six or seven months there has been produced in this country in the neighborhood of one hundred thousand gallons of soya oil. Samples from the different crushings have been examined in comparison with the imported oils. It was found that the chemical constants of the domestic oils compared very closely with those of the imported oils. A comparison of the chemical constants is here shown:

Identification No.	Specific Gravity	Iodine No.	Acid No.	Saponification
FI, No. 9355 (Domestic)......	.929	124.3	0.8	194.
Lab. No. 383 (Domestic)......	.921	129.	0.6	193.8
Lab. No. 411 (Domestic).........	.924	126.	1.3	201.
Lab. No. 409 (Domestic)......	.925	123.5	2.	196.1
Lab. No. 335 (Domestic)......	.924	126.3	1.2	190.4
Imported Oil, FI, No. 8940...	.925	123.8	1.9	190.
Imported Oil, FI, No. 9029...	.925	133.8	3.1	190.

Some of the samples examined showed a considerable amount of foots, indicating that the oil had not been allowed to settle long enough. Upon permitting these oils to settle, or after filtering, they were found to be equal to the best shipments of imported oil. Two or three samples showed at the end of three days' standing a foots separation amounting to between 8 and 10 per cent. by volume. Some of this separation consisted of oil, but the foots matter was distributed through it. Had the foots been removed by filtration it would have amounted to less than 2 per cent. by volume of the oil; the gelatinous nature of this substance keeps it distributed over so large a volume of oil and thereby depreciates the value of the oil.

The crude or raw oil should be clear, of good bright color, and possess a pleasant odor. Its chemical constants should fall within the limits set for soya oil. None of the domestic oils produced on a commercial scale have been found to show an iodine number as high as that of most of the Manchurian oils. The majority of the oils expressed in a laboratory way had iodine numbers fully as high as the Manchurian oils, which would indicate that it is possible to produce beans yielding an oil equal in this respect to the imported oils. The iodine number may be taken as an index as to the relative drying time of oils, although this does not hold absolutely, and soya oil, having an iodine number of 125, may prove just as satisfactory as one having an iodine number of 135.

In the crushing process about 75 per cent. of the oil is expressed, the rest remaining in the cake. Cold pressing will not return quite as much oil as when the ground beans are slightly heated. The bean yields its oil in a satisfactory manner if handled similar to the methods now in use for crushing cotton seed.

After the oil has been expressed it should be permitted to settle thoroughly before tanking or placing in barrels.

A ton of the beans will produce between 35 and 38 gallons of oil, depending upon the variety of the bean, which, to a certain extent, determines the percentage of oil. Taking 19 per cent. of oil as a good average, and figuring that between 4 and 5 per cent. of the oil remains in the cake, as there is some loss in the process of manufacture through moisture which is driven off, cleaning of machinery, etc., it is advisable to figure the cake returns from a ton of the beans to be about 75 pounds less than the difference between the amount of oil produced and the beans pressed.

In order that, under normal conditions, the mills can operate at a satisfactory profit the beans should be available for about $1.00 per bushel. They should not cost more than $1.25, although during periods of vegetable oil shortage the mills may find it possible to pay a little more.

It may prove profitable for the mills to import the beans from Manchuria. Due to the war and its effect on water freights it would not prove a desirable undertaking at the present time, but when the matter was looked into two or three years ago it was found that the beans could be imported for $1.00 per bushel. The mill operator should keep this in mind, because the future production in this country may be such at certain periods, at least, to warrant importation or, on further and more careful investigation, it may be found possible to crush imported beans almost continually regardless of what this country's crop develops into.

In portions of Manchuria, where the best results are obtained, the yield per acre varies from 20 to 30 bushels. The average height of the plant is about 36 inches; the plants carry from 30 to 100 pods, and each pod carries from two to three seeds. The variety cultivated most extensively is a medium-size bean, yellow in color, and almost round. The yield in this country from data up to date varies from 12 to 53 bushels; twenty-five to thirty bushels can be considered a fair average.

From the present outlook the price of soya beans under normal conditions will be determined very largely by the market value of seeds from which competing oils are made, chiefly flaxseed, cotton seed and corn. The price of soya beans will always be lower than flaxseed and higher than corn. Soya beans as a crop can be grown under very much the same conditions as corn, and while the yield in bushels is 15 to 25 per cent. less, an advance of 25 to 30 cents per bushel over corn should make them a profitable crop, taking into consideration the fertilizing value of soya beans, which is of vital importance to the farmer, and will continue to be as the fertility of the land is decreased by continual cropping and the cost of artificial fertilizers increases because of the growing scarcity of materials from which they are made.

In those sections of the South where the cotton fields are infested by the boll weevil, the growers may find it to their advantage to produce soya beans on a large scale. The Alabama Cotton Seed Crushers' Association has the matter under careful consideration at the present time. The cotton crop of certain sections of that State has been seriously affected by the disastrous results which accompany the boll weevil.

It is at once apparent that it is to the advantage of the cotton seed mills to take up the crushing of soya beans. In the first place it will help to give the mills a longer season and thereby shorten the period of idleness. This period generally varies from four to six months every year with different mills.

The crushing of soya oil has advantages over cotton seed; it is a cleaner and easier material to handle preparatory to crushing, and the bean releases its oil as freely, at least, as cotton seed.

The cake as such, or in the form of meal, should be disposed of just as readily as cotton seed cake or meal for fertilizer purposes or for stock feed. As a stock food soya bean meal is even better than cotton seed meal. The crude or raw oil, after it has settled, finds application chiefly in the manufacture of soap, paints, linoleums and in foundries in the manufacture of cores. Quantities of soya oil have been used in the manufacture of cores with very satisfactory results. The oil has been found superior to what is commonly known as "core oil." Soya oil for this purpose has been found much superior to corn and cotton seed oil, which have also been tried out on a large scale.

While practical paint tests started in 1911 have not yet been completed, and the Bureau, in line with its adopted policy, does not intend to make a definite report until the investigation has been finished, the writer feels safe in stating that large quantities of soya oil will eventually be consumed in the manufacture of paint and varnish. Because of the inferior drying properties of soya oil as compared with linseed oil it cannot entirely displace linseed, and its use in connection with linseed oil will be limited to from 25 to 50 per cent., depending on the product in which it is used. You understand that the drying properties can be taken care of when used in such quantities by the addition of driers, or the oil may be treated with metallic driers in the varnish factory, and in this manner the drying properties can be made to resemble those of linseed oil. For some time to come and, depending somewhat upon the price of cotton seed oil, soya oil will find its largest consumption in the manufacture of soaps. At the present time it is more adapted for use in this industry than in any other.

The oil can be refined and, when this is done, it is suitable for edible purposes. It can be used in a manner exactly similar to cotton seed and, in some respects, it may even be superior to refined cotton seed. Tests have been made by the Baskerville Process with excellent results.

The cake and meal can be disposed of as fertilizer and as stock food. In both respects it is the equal, at least, of cotton seed cake or meal. Here again the Agricultural Experiment Stations can point out the advantages to the farmer and stock grower.

Soya oil foots finds a market in exactly the same manner as cotton seed oil foots.

It is an assured fact that the demand for the oil will increase. The importations for the United States for the past five years have varied from fifteen to forty-five million pounds, and, as the oil becomes better known, more will be used, so that within a few years the consumption in this country should be at least several times the amount which has been used during any year heretofore.

CHAPTER II

Driers for Soya Bean Oil

The writer has received many requests for information as to the most efficient driers for soya bean oil. The results of a laboratory investigation that has just been completed, justify, in so far as these tests are concerned, the conclusions given below. It is believed that similar results with soya oil may be obtained by paint manufacturers who are skilled in the treatment of oils.

1. Through the use of proper driers, raw soya bean oil may be dried almost as rapidly as boiled linseed oil.

2. Heat treatment of raw soya bean oil produces a material that dries more rapidly than the raw oil and is more receptive to the action of driers.

3. When used alone as driers, the maximum amounts of cobalt, manganese or lead that it is desirable to incorporate in raw soya bean oil are 0.03%, 0.07%, and 0.50%, respectively. The actual amount of salts of the above metals, corresponding to such metallic content, may be calculated by reference to Table 8.

4. Linoleates are perhaps the best form in which driers may be incorporated in soya bean oil.

5. Cobalt and manganese have a tendency to produce hard films, whereas lead and zinc driers produce somewhat more elastic films. Consequently a combination of driers, in proper proportions, gives a more suitable result. This appears true not only with raw or treated soya bean oil, but also with mixtures of these oils with linseed oil.

6. The drier combinations that produced the best results, in the order named, are given below:

D { Manganese linoleate containing 0.03% manganese
Lead linoleate containing 0.20% lead
Cobalt linoleate containing 0.01% cobalt

B { Lead linoleate containing 0.20% lead
Cobalt linoleate containing 0.02% cobalt

Previously published data on the subject of driers record the results of tests made on glass plates that were re-weighed from day to day to determine the increase in weight due to oxygen absorption. In such tests the necessity of obtaining accurate weighings precluded the possibility of touching the films during the eight- or ten-day drying periods, to find out when the oils had set. Although affording considerable scientific data, tests of the above type have been of limited practical application.

In the new series of tests that have just been completed, the writer has omitted the weight tests and made determinations of the drying of the films simply by touching the painted surfaces every few hours until they had become hard. The tests have been made with great care and it is believed the information that has resulted therefrom will be of considerable service to the manufacturers of paint and varnish at this time. All the driers reported on are obtainable commercially and may be added by the paint manufacturer to liquids containing soya oil, in order to obtain the desired degree of drying.

The driers used in the tests were as follows:

Lead Linoleate
Lead Resinate
Manganese Linoleate
Manganese Resinate
Manganese Borate
Cobalt Linoleate
Cobalt Resinate
Cobalt Acetate
Zinc Resinate.

The lead linoleate and lead resinate were prepared by saponifying linseed oil or rosin with caustic alkali and then treating the clear aqueous solution of the soaps thus formed with acetate of lead solution. The insoluble precipitates were filtered off, washed thoroughly with water, and dried in vacuo over calcium chloride. The manganous, cobaltous, and zinc soaps were prepared by similar methods, using manganous, cobaltous, and zinc chlorides, respectively, as the precipitants. The manganese borate was prepared by adding a solution of borax to a solution of chloride of manganese; the white precipitate being washed with water and dried as above. Cobalt acetate was obtained in chemically pure crystal form. Throughout the tests it was decided to base the efficiency of the driers on their metallic content. Analysis was therefore made of each drier prepared, and there is given below a chart showing the theoretical metallic content of each drier, as well as the content that was actually found by careful chemical analysis of the products as prepared:

TABLE 8

Name of Drier	Theoretical Metal Content	Metal Content Found by Anaylsis	
Lead Linoleate	28.42%	Lead	26.15%
Lead Resinate	25.65%	Lead	24.12%
Manganese Linoleate	9.54%	Manganese	8.72%
Manganese Resinate	8.40%	Manganese	7.13%
Manganese Borate	23.07%	Manganese	24.18%
Cobalt Linoleate	10.16%	Cobalt	8.36%
Cobalt Resinate	8.95%	Cobalt	6.92%
Cobalt Acetate	23.69%	Cobalt	24.18%
Zinc Resinate	9.77%	Zinc	7.83%

It will be observed that there are some slight differences between the theoretical metallic contents and those shown by analysis. These minor differences are due to such causes as slight oxidation during the drying of the soaps, and to traces of moisture that could not be removed from the curdy masses. In the case of cobalt acetate, the slightly high percentage of cobalt present was probably due to the loss by the crystals of a small amount of water of hydration.

Two series of tests were made on each drier. One series was made by applying the oils containing definite amounts of drier, to glass plates inclined at an angle of about 20°. The other series was made by applying the same types of oils to boards that had already received two coats of paint thoroughly dried. The painting tests on boards were allowed to dry in a horizontal position. The application in each test was made with a brush.

The oils used in the tests were prepared by dissolving the drier in raw soya oil with the aid of gentle heat. Stress should be laid on the proper method of solution of the drier in the oil, when strong stock solutions are required. It frequently happens that a given amount of drier added to the oil is not completely dissolved. Apparent solution may take place while hot, but on cooling a sediment deposits, consisting chiefly of uncombined drier. The best way of dissolving the drier is to heat the oil slowly to about 300° C., then adding the drier in small portions during a period of about an hour, and continuing the heating for about one-half hour after all the drier has been added. In this way the maximum amount of drier combines with the oil.

Sufficient quantities of drier were added by the above process to give stock solutions having a 1% metal content. After preparation, the oils were filtered and a portion of each was ashed to determine the exact amount of metal present. Adjustments were then made by adding further amounts of oil or of drier, in order to make the solutions standard. From these stock solutions, by dilution with raw soya oil, solutions for test were made, having a metal content noted in Tables 9 to 14, which also show the number of hours required to dry the applied films.

TABLE 9.

Drying Tests on Glass

Driers dissolved in 100% Raw Soya Oil.
Time of drying reported in hours.

Metal Content of Drier in oil. *Percent.*	Cobalt Linoleate.	Cobalt Resinate.	Cobalt Acetate.	Manganese Linoleate.	Manganese Resinate.	Manganese Borate.
0.01	49	52	51	53	54	53 hours to dry
0.03	*36	*38	*39	44	45	44 hours to dry
0.05	27	29	30	39	41	40 hours to dry
0.07	24	26	26	*35	*38	*36 hours to dry
0.09	23	24	25	33	35	33 hours to dry
0.11	22	22	24	31	33	32 hours to dry
0.15	21	21	23	30	31	30 hours to dry
0.20	20	21	21	29	30	28 hours to dry

Metal Content of Drier in oil. *Percent.*	Lead Linoleate.	Lead Resinate.	Zinc Resinate.
0.10	73	75	84 hours to dry
0.20	60	63	75 hours to dry
0.30	54	56	65 hours to dry
0.40	49	51	60 hours to dry
0.50	*42	*44	55 hours to dry
0.60	39	40	*52 hours to dry
0.70	36	38	49 hours to dry
0.80	34	35	46 hours to dry

The (*) in each instance indicates the highest metallic content giving a smooth, hard film of dried oil. Driers used in excess of amount so noted cause the oils to set up well at first and dry rapidly, but to develop wrinkles and other phenomena, characteristic of surface drying. Moreover, such mixtures do not harden properly. Possibly an excessive amount of drier causes a reversible action to occur.

TABLE 10.

Drying Tests on Painted Wood.

Driers dissolved in 100% Raw Soya Oil.
Time of drying reported in hours.

Metal Content of Drier in oil. *Percent.*	Cobalt Linoleate.	Cobalt Resinate.	Cobalt Acetate.	Manganese Linoleate.	Manganese Resinate.	Manganese Borate.
0.01	38	39	41	42	44	42 hours to dry
0.03	*24	*26	*29	34	37	35 hours to dry
0.05	18	20	24	29	30	29 hours to dry
0.07	15	17	20	*26	*28	*27 hours to dry
0.09	13	15	17	23	24	23 hours to dry
0.11	12	14	15	21	22	20 hours to dry
0.15	11	12	14	19	21	18 hours to dry
0.20	10	11	12	18	19	17 hours to dry

Metal Content of Drier in oil. *Percent.*	Lead Linoleate.	Lead Resinate.	Zinc Resinate.
0.10	60	63	76 hours to dry
0.20	49	51	72 hours to dry
0.30	43	45	69 hours to dry
0.40	37	39	64 hours to dry
0.50	*32	*34	58 hours to dry
0.60	28	29	53 hours to dry
0.70	25	26	*50 hours to dry
0.80	23	24	48 hours to dry

The (*) in each instance indicates the highest metallic content giving a smooth, hard film of dried oil. Driers used in excess of amount so noted cause the oils to set up well at first and dry rapidly, but to develop wrinkles and other phenomena, characteristic of surface drying. Moreover, such mixtures do not harden properly. Possibly an excessive amount of drier causes a reversible action to occur.

TABLE 11.

Drying Tests on Glass

Driers dissolved in a mixture of 75% Raw Linseed Oil and 25% Raw Soya Oil.
Time of drying reported in hours.

Metal Content of Drier in oil. *Percent.*	Cobalt Linoleate.	Cobalt Resinate.	Cobalt Acetate.	Manganese Linoleate.	Manganese Resinate.	Manganese Borate.
0.01	30	32	32	37	39	37 hours to dry
0.03	*25	*28	*27	32	33	33 hours to dry
0.05	23	26	26	29	31	30 hours to dry
0.07	21	23	23	*27	*28	*27 hours to dry
0.09	18	20	21	25	26	26 hours to dry
0.11	17	19	19	23	24	23 hours to dry

Metal Content of Drier in oil. *Percent.*	Lead Linoleate.	Lead Resinate.	Zinc Resinate.
0.10	60	63	77 hours to dry
0.20	51	52	64 hours to dry
0.30	45	48	59 hours to dry
0.40	39	40	55 hours to dry
0.50	*33	*34	50 hours to dry
0.60	31	33	46 hours to dry
0.70	30	32	*42 hours to dry
0.80	29	30	39 hours to dry

Under same conditions of test, raw linseed oil requires 61 hours to dry, and raw soya oil requires 172 hours.

*Indicates best film.

TABLE 12.

Drying Tests on Painted Wood.

Driers dissolved in a mixture of 75% Raw Linseed Oil and 25% Raw Soya Oil.
Time of drying reported in hours.

Metal Content of Drier in oil. *Percent.*	Cobalt Linoleate.	Cobalt Resinate.	Cobalt Acetate.	Manganese Linoleate.	Manganese Resinate.	Manganese Borate.
0.01	19	21	22	25	26	26 hours to dry
0.03	*14	*15	*16	19	19	18 hours to dry
0.05	11	13	14	17	18	17 hours to dry
0.07	9	10	12	*16	*17	*16 hours to dry
0.09	7	9	11	12	14	13 hours to dry
0.11	7	8	10	10	11	11 hours to dry

Metal Content of Drier in oil. *Percent.*	Lead Linoleate.	Lead Resinate.	Zinc Resinate.
0.10	46	48	64 hours to dry
0.20	38	39	52 hours to dry
0.30	32	34	47 hours to dry
0.40	26	27	41 hours to dry
0.50	*21	*23	38 hours to dry
0.60	20	21	35 hours to dry
0.70	18	20	*33 hours to dry
0.80	16	19	28 hours to dry

Under same conditions of test, raw linseed oil requires 50 hours to dry, and raw soya oil requires 150 hours.

*Indicates best film.

TABLE 13.

Drying Tests on Glass

Soya Bean Oil heated to 600° F. for 30 minutes, then dropped to 400° F. and air blown through it for 30 minutes.
Driers were then added to this oil.
Time of drying reported in hours.

Metal Content of Drier in oil. *Percent.*	Cobalt Linoleate.	Cobalt Resinate.	Cobalt Acetate.	Manganese Linoleate.	Manganese Resinate.	Manganese Borate.
0.01	43	46	46	48	49	47 hours to dry
0.03	*31	*32	*33	38	39	38 hours to dry
0.05	21	23	24	32	36	34 hours to dry
0.07	18	19	20	*30	*32	*31 hours to dry
0.09	17	17	18	27	29	28 hours to dry
0.11	16	16	17	23	25	24 hours to dry

Metal Content of Drier in oil. *Percent.*	Lead Linoleate.	Lead Resinate.	Zinc Resinate.
0.10	66	69	80 hours to dry
0.20	53	56	70 hours to dry
0.30	48	50	61 hours to dry
0.40	42	44	56 hours to dry
0.50	*35	*36	51 hours to dry
0.60	33	35	48 hours to dry
0.70	30	31	*44 hours to dry
0.80	27	28	41 hours to dry

Under the same conditions, soya oil treated as above and without the addition of driers, dries in 144 hours. The heat treatment has improved the drying rapidity of the oil when mixed with drier.

*Indicates best film.

TABLE 14.

Drying Tests on Painted Wood.

Soya Oil heated to 600° F. for 30 minutes, then dropped to 400° F. and then air-blown for 30 minutes. Driers were then added to this oil.

Time of drying reported in hours.

Metal Content of Drier in oil. *Percent.*	Cobalt Linoleate.	Cobalt Resinate.	Cobalt Acetate.	Manganese Linoleate.	Manganese Resinate.	Manganese Borate.
0.01	33	35	36	39	40	40 hours to dry
0 03	*20	*21	*23	31	33	31 hours to dry
0.05	15	17	19	26	26	28 hours to dry
0 07	13	13	15	*22	*23	*23 hours to dry
0.09	11	12	12	20	22	21 hours to dry
0.11	10	10	11	18	18	18 hours to dry

Metal Content of Drier in oil *Percent.*	Lead Linoleate.	Lead Resinate.	Zinc Resinate.
0.10	53	56	70 hours to dry
0.20	43	45	67 hours to dry
0.30	38	40	63 hours to dry
0.40	31	32	56 hours to dry
0.50	*27	*28	51 hours to dry
0.60	23	25	47 hours to dry
0.70	22	22	*44 hours to dry
0.80	21	21	42 hours to dry

Under same conditions, soya oil treated as above and without the addition of driers, dries in 108 hours.

*Indicates best film.

From the foregoing, it would appear that the most efficient individual driers of the cobalt, manganese, or lead type that can be used with soya oil, are as follows:

Cobalt linoleate in amount sufficient to show 0.03% metallic cobalt in the soya oil.

Manganese linoleate in amount sufficient to show 0.07% metallic manganese in the soya oil.

Lead linoleate in amount sufficient to show 0.50% metallic lead in the soya oil.

Zinc driers apparently react so slowly that they are not of great value. Because of their light color, however, they may be of service in white enamels.

It should be noted that no matter in what form the metal is combined in a drier, its drying value is about the same, with perhaps a slight preference to be given the linoleates.

It should be pointed out that paints dry more rapidly than oils, due to the accelerating action of the pigments. An oil, for instance, that dries in 16 hours will show a drying time of from 15 to 14 hours when made up into a paint with suitable lead or zinc pigments.

Particular attention is directed to the notes attached to the charts reporting the experimental work, showing the effects of the addition of excess drier. It is gratifying to observe the corroboration evidenced in repeated experiments, on the maximum amount of drier that it is desirable to incorporate in soya oils. Excess drier may be more harmful than insufficient drier.

In each instance the oils containing cobalt and manganese formed films having a hard, smooth finish; the lead and the zinc driers giving a more elastic film. It would seem desirable, therefore, in order to get a hard and yet elastic film to combine these driers in proper proportions. With this in view, out of the innumerable combinations possible, a series of tests were made. Raw soya oil was used, containing the following driers, and the mixtures were applied to painted boards:

		Metal.	
A	Cobalt linoleate	0.02%	Dries in 17 hours to a fair film, but contains too much drier.
	Manganese linoleate	0.02%	
	Lead linoleate	0.30%	
B	Cobalt linoleate	0.02%	Dries in 21 hours to an excellent film.
	Lead linoleate	0.20%	
C	Manganese linoleate	0.03%	Dried in 23 hours to a very good film.
	Lead linoleate	0.30%	
D	Manganese linoleate	0.03%	Dries in 18 hours to a most excellent film. Apparently the best combination.
	Lead linoleate	0.20%	
	Cobalt linoleate	0.01%	
E	Manganese borate	0.02%	Dries in 22 hours to a fairly good film; not quite as hard as those containing less lead.
	Cobalt resinate	0.01%	
	Lead resinate	0.40%	
F	Manganese resinate	0.01%	Dries in 19 hours. Apparently too much drier.
	Cobalt acetate	0.03%	
	Lead resinate	0.40%	

The best combinations are D and B, in the order named.

Applying driers D and B to mixtures of 75% linseed and 25% of raw soya oil, drying takes place in 12 and 14 hours, respectively, the resulting film in each instance being firm and smooth. When applied to soya oil that has been heated to 600° F. and subsequently air-blown (see tests on this oil), drier D causes drying to take place in 17 hours, and drier B in 19 hours; good films resulting in each case. Side by side with these last two tests, and under exactly parallel conditions, painted boards were coated with two samples of pure commercial boiled linseed oil Nos. 1 and 2. No. 1 contained 0.26% lead and 0.034% manganese. No. 2 contained 0.21% lead and 0.041% manganese. No. 1 dried in 13 hours; No. 2 in 12 hours.

The results show that by using drier D, it is possible to incorporate 25% of soya oil with linseed and get a film that dries as rapidly as commercial boiled linseed oil. The six tests outlined above are charted below.

DRIERS FOR SOYA BEAN OIL

Linseed oil 75% Soya bean oil 25% With Drier D	Dries in 12 hours to a firm, smooth film.
Linseed oil 75% Soya bean oil 25% With Drier B	Dries in 14 hours to a firm, smooth film.
Heat-treated soya oil With Drier D	Dries in 17 hours.
Heat-treated soya oil With Drier B	Dries in 19 hours.
Boiled linseed oil .26 lead, .034 manganese	Dries in 13 hours.
Boiled linseed oil .21 lead, .041 manganese	Dries in 12 hours.

CHAPTER III

Marine Animal and Blubber Oils

Although whale and herring oils have been used to some extent in the manufacture of paint, menhaden oil has so far proved much more satisfactory. The better grades of menhaden oil often approach in constants the oil crushed from South American flax of a lower grade, samples having an iodine value up to 170 being sometimes obtainable. The quantity of menhaden oil produced depends upon the activity of the fisheries. From the figures in Table 15, furnished by the U. S. Bureau of Fisheries, it will be noted that the production during 1918 exceeded that of 1917 in spite of war-time conditions. With the return to peace and the present high price of oils, it is quite possible that a greatly increased production may be made available.

Refined menhaden oil has been in use for many years as a constituent of the liquid part of certain types of paint. For use in barn and roof paints and coatings for structural steel, all of which are usually of a dark color, it has proved successful. In white paints it has so far proven less satisfactory, since it has the property of taking up dust. This is due to the fact that the raw fish oil does not dry permanently hard and has a tendency to become slightly soft in warm, moist atmospheres. This property may to some extent account for the elasticity of film that has been attributed to it. The use of red lead and other pigments which stimulate drying is advisable in such paints in order to reduce the softening tendency.

Fish oil, like many other oils, gives much better results when blown and heat treated. The addition of driers during this operation is advisable. When thus treated, products of a bodied nature result, which produce a much more satisfactory film than does raw oil.

TABLE 15

STATEMENT OF THE NUMBER OF GALLONS OF VARIOUS AQUATIC OILS PRODUCED IN 1917 AND 1918

Year and Kinds	North of Chesapeake Bay	Chesapeake Bay	North Carolina	Florida	Texas.	Total.	Pacific Coast States.	Alaska.	Grand Total.
1917:									
Menhaden	368,200	1,750,000	1,200,000	231,500	70,000	3,619,700			3,619,700
Whale								900,883	900,883
Sperm								197,670	197,670
Herring								205,992	205,992
Miscellaneous							283,160	25,150	308,310
Total	368,200	1,750,000	1,200,000	231,500	70,000	3,619,700	283,160	1,329,695	5,232,555
1918:									
Menhaden	*	*	*	*	*	3,900,000			3,900,000
Whale							232,500	671,310	903,810
Sperm							43,000	338,931	381,931
Herring								138,012	138,012
Miscellaneous							386,362	4,624	390,986
Total						3,900,000	661,862	1,152,877	5,714,739

*Distribution not tabulated.

Note.—The figures for the menhaden oil are estimated on a basis of 50 gallons to the barrel. Miscellaneous oil is that obtained from by-products.

The odor of fish oil is claimed to be due to the presence of small percentages of clupanodonic acid. The removal or destruction of this acid would possibly do away with the objectionable odor of the oil. Great difficulty, however, has attended the attempts that have been made to remove the odor. It is probable that the real odor is due to the presence of amines that are not readily broken up. Although it is well known that marine animal oils, such as whale oil, may be made perfectly neutral in odor by hydrogenation, the degree of hydrogenation required to remove the odor is apparently so great as to considerably lower the iodine value and stiffen the oil to a degree that would make it unsuitable for use in paints.

Some of the many attempts that have been made to treat fish oil to reduce the odor are described in the following patents:

English Patent 12,525, 1905; German Patent 169,410: A. de Hemptine. Treatment of fish oils with ozone.

German Patent 162,638: Sanberg. Concentrated sulphuric acid.

U. S. Patent 823,361: Potolowsky. Concentrated sulphuric acid plus nitric acid.

English Patent 7901, 1906: E. Böhm. Heating in vacuo to 350°-400° C.

German Patent 202,576, 1908: E. Peterson and H. Holstein. Treatment with superheated steam, chalk and charcoal.

English Patent 23,534, 1906: Bottaro. Odorless fatty acids obtained from fish oil by saponification with lime and subsequent decomposition of the lime soap with sulphur dioxide gas.

French Patent 394,456: C. H. J. Melfling. The fish oil is emulsified with alcohol and after separating the alcohol the oil is washed with hot water to remove the alcohol retained by the oil. The washed oil then is treated with fuller's earth or animal charcoal.

French Patent 394,530: G. van der Heyden. Treating the fish oils with six to eight times the volume of alcohol at 70°. washing with hot water and treating the washed oil with fuller's earth at 75°-80° C.

French Patent 448,517: S. H. Goldschmidt and C. Jorgensen. By treating the "sebacic acid" of whale and fish oils with sodium-hypochloride containing as little sodium hydrate as possible.

English Patent 18,925, 1913: F. Bergins. Fish oil is deodorized by a single heating to 250°-300° C. for several hours.

German Patent 294,136, 1913: G. Weiss. The oil is heated to 200° C. under atmospheric pressure and subsequently treated with steam in the presence of basic substances, especially oxides or hydroxides of alkali, alkaline earth, or other metals.

U. S. Patent 1,260,072, 1918: Walter P. Shuck.

While experiments are being conducted by the writer along other lines, which may yield interesting results, it is suggested that for the immediate present at least the disagreeable odor of fish oil be offset by using with it other ingredients of a pronounced but more agreeable odor. For instance, when fish oil is mixed with from 5% to 10% of pine oil (the water white or straw yellow final distillate in turpentine production), a product having a rather pleasant turpentine odor results.

There are given below some results showing the changes that took place in menhaden oil upon standing in the writer's laboratory for a number of years:

TABLE 16

	Specific Gravity	Iodine No.	Sapon No.	Acid No.	Refractive Index.
March, 1911	.932	158.	187.	3.9	....
November, 1914	.934	156.3	193.7	16.1	1.4850
September, 1916	.938	....	191.4	19.2	1.4768
February, 1919	.940	156.9	191.5	21.3	1.4838

TABLE 17

RAW MENHADEN OIL

(Untreated and Sterilized)

	Specific Gravity	Iodine No.	Sapon No.	Acid No.	Refractive Index.
Original Oil (not sterilized) examined 1911	.932	158.	187.	3.9	....
Original Oil (not sterilized) examined November, 1914	.934	156.3	193.7	16.1	1.4850
Original Oil (not sterilized) examined February, 1919	.940	156.9	191.5	21.3	1.4802
Original Oil (sterilized November, 1914*) examined February, 1919	.938	156.2	190.1	5.1	1.4802

*Sterilized by heating to 105° C.

It will be noted from the above charts that fish oil may undergo rather rapid changes on storage, due to the decomposition of the glycerides and formation of substantial percentages of free fatty acid. This would indicate the advisability of careful storage of the oil. In the latter chart the results obtained by sterilizing the oil are of interest, since very slight changes in the constants of such oil take place.

The wearing value of menhaden oil in white lead and zinc paints is shown by the results of some exposure tests started in 1911. These included repainting tests covering a period of nearly three years. The color, chalking, and checking of the paints is recorded below. It will be noted that with raw menhaden oil the darkening is considerable, but that where this oil is used in admixture with substantial percentages of linseed oil very much cleaner paints result.

TABLE 18

Panel.	Formula.	Year.	Color.	Chalking.	Checking.
3	100% Menhaden Oil	1912	Very dark	Very slight	None
		1913	Very dark	Considerable	Very slight
		1914	Very dark	Medium	Slight
		R–1916	Very dark	Medium	Slight
		R–1917	Very dark	Slight	Slight
11	50% Raw Linseed Oil 50% Menhaden Oil	1912	White	Medium	None
		1913	Fairly white	Medium	Considerable
		1914	White	Medium	Considerable Deep in places
		R–1916	White	Medium	Considerable
		R–1917	Fairly white	Slight	Heavy
19	75% Raw Linseed Oil 25% Menhaden Oil	1912	Darkened	Medium	Slight
		1913	Fairly white	Medium	Slight
		1914	White	Medium	Medium
		R–1916	White	Medium	Medium
		R–1917	Fairly white	Slight	Medium

Shark, Dog Fish, and Other Marine Oils.—The writer has been informed by the U. S. Bureau of Fisheries that there has been established a by-products plant on the Pribilof Islands, where an output of twenty-five to forty thousand gallons of fur seal oil may be produced annually.

Information has also come to the effect that fisheries in the State of Washington now handle considerable quantities of dog fish for conversion into oil and fertilizer, and that plants now located at Morehead City, N. C., and Fort Myers, Fla., work up large catches of sharks for the liver oil content. It is possible that these same plants will be able to handle skate liver oil and other types of fish oil, and that the West Coast plants may be able to produce considerable tuna fish oil and salmon oil. Samples of all the above types of oil are being obtained and will be used in an investigation of their drying properties and suitability for use in paints.

With the apparent shortage of animal skins, attempts have recently been made in the leather industry to use aquatic skins. It is probable that these skins, when properly tanned, will find a wide application. This industry would receive considerable encouragement should it be found that the liver oils or other oils obtained from the fish from which skins are stripped, could be worked up into suitable drying oils.

In a memorandum on shark and dog fish oils, compiled by Dr. G. F. White, special investigator for the Bureau of Fisheries, considerable data on the yield, composition and uses of liver oils are given. In this paper the chemical constants are given for cod liver oil, dog fish liver oil, sand shark liver oil, and hammerhead shark liver oil, showing saponification numbers ranging from 171 to 188, acid numbers from 1.52 to 4.84, and iodine numbers ranging from 155.5 to 213.6. The latter iodine number, which is very high, was obtained on a sample of sand shark liver oil. Although most oils of the above type are rather dark in color and of disagreeable odor, it is possible that treatment may remove these objectionable characteristics.

One of the present writer's associates, who has had considerable experience with liver oils, claims that the odor of such oils is due to the presence of such amines as butylamine, hexylamine, dehyrolutidine, and morrhurine, which are present as a result of careless handling of the livers, permitting putrefaction. He states that his investigations made several years ago showed that the oil from fresh livers is free from nitrogen, of fairly light color and unobjectionable odor.

Blubber Oils: A preliminary investigation has been made of certain fish oils obtained from the Commissioner of Fisheries, Department of Commerce, with a view to determining their value in the paint and varnish industry. These oils have heretofore found application in other industries and may be used in the manufacture of soap, leather dressings, core oils, and for hydrogenation. It is probable that they will become available in greater quantities and be of use in the manufacture of certain types of coatings. So far, however, they have not, when used alone, proved satisfactory as drying oils, and it is probable that they will find application only in admixture with real drying oils. The results of analysis of the samples are given herewith:

TABLE 19

	Grayfish Oil	Shark Liver Oil	Fur Seal Oil	Skate Liver Oil
Refractive Index	1.4703	1.4708	1.4772	1.4712
Specific Gravity	.9157	.9222	.9254	.9318
Acid Value	2.0	1.3	9.0	1.8
Saponification Value	180.1	62.2	182.4	179.9
Iodine Value (Hanus)	135.7	135.9	132.4	151.6

Iodine value of hydrocarbon extracted from Shark Liver Oil = 141.5.

Data resulting from drying tests are presented below. In these tests information is given regarding the drying value of the raw oils, with and without the addition of drier, as well as the drying value of the oils after blowing and heating for a period of two hours at a high temperature. The oils resulting from the heat treatment were not thick and viscous like linseed oil that has been given similar treatment. This would indicate that the above named fish oils would not be satisfactory for the manufacture of varnishes.

TABLE 20

DRYING TESTS ON FISH OILS.

Tests started on September 10, 1919.

1	Grayfish Oil	All oils without drier required three days to become tacky. At the end of 21 days they were still soft and "greasy" to the touch. All oils with 10% drier became tacky in two days. At the end of 21 days they were still soft, but presented somewhat better films than those without drier.
2	Grayfish Oil with 10% drier	
3	Fur Seal Oil	
4	Fur Seal Oil with 10% drier	
5	Skate Liver Oil	
6	Skate Liver Oil with 10% drier	
7	Shark Liver Oil	
8	Shark Liver Oil with 10% drier	

TABLE 21

DRYING TESTS ON BLOWN OIL

Oil heated to 220° C. and air bubbled through for 2 hours.
Temperature maintained at 200-240° C.

9	Blown Grayfish Oil	All oils without drier required nearly three days to become tacky. At the end of 21 days they were still soft. All oils with 10% drier became tacky in less than 2 days and had set at end of 21 days to films that were rather soft.
10	Blown Grayfish Oil with 10% drier	
11	Blown Fur Seal Oil	
12	Blown Fur Seal Oil with 10% drier	
13	Blown Skate Liver Oil	
14	Blown Skate Liver Oil with 10% drier	
15	Blown Shark Liver Oil	
16	Blown Shark Liver Oil with 10% drier	

TABLE 22

DRYING TESTS ON FISH OIL MIXTURES

Mixture No. 1. 45% Fish Oil, 45% Linseed Oil, 10% Drier.
Mixture No. 2. 50% Fish Oil, 50% Tung Oil Spar Varnish WD 6.

Grayfish Oil Mixture No. 1	Almost dry in 1 day, but present a somewhat tacky film. After 4 days the films were still slightly tacky. The maximum set apparently occurs during the first day of drying period.
Fur Seal Oil “ “ 1	
Skate Liver Oil “ “ 1	
Shark Liver Oil “ “ 1	
Grayfish Oil Mixture No. 2	Almost dry in 1 day, but films are slightly “greasy” to touch. After 3 days they were slightly harder than films shown by Mixtures No. 1, above, in 4 days. The Grayfish Oil mixture is harder than the others, having shown much better drying values.
Fur Seal Oil “ “ 2	
Skate Liver Oil “ “ 2	
Shark Liver Oil “ “ 2	

From the results presented above, it would appear that the marine animal oils experimented with might be used in admixture with linseed oil and drier or with spar varnish in certain types of paint where rapid drying is not essential. Roof paints, paints for rough lumber, and certain metal coatings might be considered in the above class. It is believed that the films presented by the above mixtures are, when dry, quite elastic and water repellant. They are, however, apparently subject to softening action during moist weather, and in this condition tend to pick up dust.

The drying results given above would indicate that the iodine number of a marine animal oil does not constitute a reliable index of the drying value of the oil. The fish oils examined showed, for instance, iodine numbers of from 132 to 151, but possessed only slight drying properties, whereas vegetable oils showing iodine numbers as low as 120-130 (cottonseed oil, soya bean oil, etc.) are capable of being dried in a relatively short period of time. While most blubber oils consist largely of glycerides, some of the acids forming these compounds may be of a rather saturated type. Volatile acids may also be present as constituents of the glycerides. The latter products may have a retarding effect upon the oxygen absorption of the drying glycerides. Such oils also may contain stearin or spermaceti. The shark liver oil examined precipitated a considerable amount of a white, flocculent mass resembling spermaceti.

The presence of highly unsaturated hydrocarbons in marine animal oils apparently does not add to their drying value. Such a hydrocarbon was found present in substantial quantity in the sample of shark liver oil examined, which was produced from sharks caught off the North Carolina coast. The presence of this hydrocarbon was indicated by the low saponification value and large amount of unsaponifiable matter present. The hydrocarbon was isolated from a sample. It was of a pale yellow color, limpid, and without pronounced odor. It had poor drying properties, but showed an iodine number of 141.5. This low iodine number should be compared with that of the hydrocarbon extracted from Japanese shark liver oil (388) by Mitsumaru Tsujimoto. In his paper entitled "A Highly Unsaturated Hydrocarbon in Shark Liver Oil"* the following statements are made:

> "The results of the present investigation have confirmed that two Japanese shark liver oils, Aizamé and Heratsunozamé oils, contain very high proportions of unsaponifiable matter which mainly consist of a new highly unsaturated hydrocarbon of the formula $C_{30}H_{50}$. It will be rather premature to make any further statement, but certainly a remarkable quantity of a hydrocarbon, which in reality is the essential constituent of the oil itself, occurs in oils of such vital organs as the liver of the fish. It may be added here from the author's latest experiments that the hydrocarbon also occurs in the liver oils of so-called 'black sharks,' basking sharks and another shark (probably *Triakis scyllium*—Japanese, 'Korozamé). Sharks belonging to Squal-

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***J. Ind. & Engrg. Chem.*, October, 1916, p. 889.

> idæ appear to contain the hydrocarbon more frequently. But, the data obtained hitherto are yet too scanty to allow a generalization as to the genus and species of sharks.
>
> "To solve the question, why such a hydrocarbon is present in the liver of some fish, is probably very difficult, and presents an important problem for physiological chemistry. It is interesting to note that the bromine content of the bromine additive compound of the hydrocarbon is nearly identical with those of the polybromides of the highly unsaturated fatty acids in marine animal oils, viz., about 70 per cent.
>
> "In the case of the present shark liver oils, the place of the highly unsaturated fatty acids is taken, as it were, by the hydrocarbon of a nearly equally high unsaturation. The liver is a storage organ for fat and consequently a source of energy of the fish. If a speculation may be allowed, the hydrocarbon, which evolves far more heat of oxidation than the glycerides, must have been formed in the liver of the fish from the necessity of local conditions. Little as the habit of sharks is known, it is pretty certain that they are comparatively deep-sea dwellers."

In describing the properties of the hydrocarbon which he has found to have the formula $C_{30}H_{50}$, with a molecular weight of 408, the following information is given:

> "The hydrocarbon is a colorless, oily liquid which refracts light somewhat strongly. In the pure state, it is nearly odorless; but the distilled sample possesses a faint odor, apparently of decomposition products, which can be removed by washing with alkali. On keeping, it evolves an odor which recalls some terpenes. Its taste is peculiar, but not so unpleasant. It is readily soluble in ether, petroleum ether, carbon tetrachloride and acetone; in cold alcohol and glacial acetic acid, it is sparingly soluble. When heated over a flame, the hydrocarbon burns with a smoky, luminous flame, emitting a resinous smell.
>
> "BOILING POINT—Under ordinary pressure, the hydrocarbon cannot be distilled without decomposition. Boiling point (10 mm. pressure), 262-264° C; (5 mm. pressure) 252-254° C.
>
> "SOLIDIFYING POINT—Kept at -20° C. for one hour, it remained clear; at -75° C., it solidified to a white wax-like mass.

"SPECIFIC GRAVITY—At 15°/4° C., 0.8587.

"IODINE VALUE—388.12 (Wijs), 389.70 (Hübl).

"Theory requires for $C_{30}H_{50}I_{12}$, iodine value = 371.10.

"REFRACTIVE INDEX—(20° C.) 1.4965.

"OPTICAL ROTATION—Inactive.

"HEAT OF COMBUSTION—(Hempel's calorimeter), 10,773 cal. per g.

"COLOR REACTION—By Liebermann's test (solution in acetic anhydride and addition of a drop of concentrated sulfuric acid), the fresh hydrocarbon gave only a faint pink coloration in the lower (acetic anhydride) layer.

"DRYING PROPERTY—The hydrocarbon dries like the vegetable drying oils. The pure hydrocarbon requires a long time to dry at the ordinary temperatures. That which had been treated with 1 per cent of cobalt resinate, dried in about 10 days (in winter) to a colorless, smooth film which possessed a firmness superior to those of the fatty oils. At 100° C., a thin layer of the hydrocarbon dried in about 2 hours."

In conclusion, it is suggested that the four oils reported on above deserve further investigation when applied in admixture with strong drying oils (linseed, tung, etc.) in the manufacture of special paints such as barn paints, oxide metal paints, and roofing paints. Distillation in vacuo of the shark liver oil might be carried out commercially if the hydrocarbon is found useful in paint making.

CHAPTER IV

Miscellaneous Fish Oils

Tuna Fish Oil: A sample of tuna fish oil forwarded from the U. S. Commissioner of Fisheries, H. M. Smith, was recently received at the writer's laboratory. It is understood that this type of oil is available on the Pacific Coast and that a substantial quantity might be produced for use in the paint industry. Examination of the sample has given the following results:

Color	Pale
Odor	Not Objectionable
Taste	Very bland for a fish oil

TABLE 23

CONSTANTS OF TUNA FISH OIL

Iodine Value (Hanus)			184.0
Saponification Value			190.0
Acid Value			0.37
Specific Gravity	Westphal Balance	.9327	.9327
	Hydrometer	[illegible]	
	Pyrometer		

TABLE 24

DRYING TIME ON GLASS IN HOURS
Tuna and Linseed Oil.

Oil.	Tacky	Dry	Character of Film.
Tuna Oil	20	58	Firm and clear.
Raw Linseed Oil	72	96	Firm and clear.
Tuna Oil with 10% Drier*	......	10	Very firm, clear and lustrous.
Raw Linseed Oil with 10% Drier	......	12	Firm and clear.
Tuna Oil—50% Raw Linseed Oil—50%	50	66	Very firm, clear and lustrous.
Tuna Oil—45% Raw Linseed Oil—45% Drier—10%	......	11	Firm, clear and lustrous.

* Drier used was Liquid Lead—Manganese Linoleate in Turpentine.

Information originally obtained from the Bureau of Fisheries of the Department of Commerce indicates that approximately 100,000 gallons of tuna oil are annually produced on the west coast. Information obtained from the Pacific-American Fisheries indicates that it is usual for the west coast fisheries to market tuna oil in the emulsion form in which it is produced during the process of manufacture. Apparently attempts to break the emulsion are not usually made. Such emulsions are difficult to use. It is understood that this oil is produced by the South Coast Canning Co. at Long Beach, Cal., and by F. Van Camp, of San Pedro, Cal.

Yellow Tail Fish Oil: The results of a preliminary investigation of yellow-tail fish oil are given below. The sample of oil used in the tests was forwarded by the U. S. Commissioner of Fisheries. It was received from the West Coast Laboratory of the Bureau of Fisheries in November, 1919. It is understood that the species from which the oil was obtained (*Seriola dorsalis*) is abundant on the Southern and Lower California coast. According to "California Fish and Game," the catch in 1918 amounted to 12,000,000 pounds.

The oil contains a large quantity of "stearine" which gives it a very opaque appearance. Upon filtration, a very clear, pale oil is obtained.

Color	Light
Odor	Not strong
Taste	Not pleasant

TABLE 25

CONSTANTS OF YELLOW TAIL OIL

Iodine Value (Hanus)	177.*
Saponification Value	190.
Acid Value	0.6
Specific Gravity	.9322

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*Iodine value of filtered oil 180.

TABLE 26
DRYING TIME ON GLASS
Yellow Tail and Linseed Oil.

OIL	Tacky (hours)	Dry (hours)	Character of Film
Yellow Tail Oil	75	105	Fairly firm
Raw Linseed Oil	70	95	Firm and clear
Yellow Tail Oil with 10% Drier*		15	Fairly firm
Raw Linseed Oil with 10% Drier*		12	Firm and clear
Yellow Tail Oil.........50% Raw Linseed Oil.........50%	72	100	Firm and clear
Yellow Tail Oil.........45% Raw Linseed Oil.........45% Drier.........10%		14	Firm and clear

*Lead and manganese linoleate liquid drier.

There is given below a list of fisheries operating on the West Coast. This was furnished by the Bureau of Fisheries. It is believed that various types of West Coast fish oils are obtainable from many of these fisheries.

FISH OIL AND FERTILIZER COMPANIES OPERATING IN 1918
ALASKA

NAME	Location.	Product.
Alaska Fish Salting and By-Products Co.	Killsinoo	Fish meal, fish oil.
Fish Canners' By-Products, Ltd.	Ward's Cove	Fertilizer, fish oil.
Royden, W. H.	Petersburg	Shark liver oil.

MISCELLANEOUS FISH OILS

PUGET SOUND

Japanese-American Fert. Co.	Lummi Island	Fertilizer, fish oil.
Marani Products Co.	Anacortes	Fish meal, fertilizer, glue, fish oil.
Pacific American Fisheries	Eliza Island	Fish meal, fish oil.
Pacific Products Co.	Port Townsend and Friday Harbor	Fertilizer, fish oil.
Robinson Fisheries Co.	Anacortes	Fish meal, fish oil.
Sea Products Co.	Everett	Fish meal, fish oil.
Wannenwetsch Reducing Co.	Blaine	Fish meal, fish oil.

COLUMBIA RIVER

De Force Oil Works	Astoria	Fertilizer, fish oil.
Northwest Fish Products Co.	Astoria	Fish meal, fish oil.

CALIFORNIA

American Tuna Packing Co.		Fish meal, fish oil.
Booth, F. E., Co.	Monterey	Fish meal, fertilizer.
Globe Cotton-Seed Oil Works	East San Pedro	Fish meal, fertilizer, fish oil.
Halfhill Tuna Packing Co.	Long Beach	Fish meal, fertilizer.
Hovden, K. Co.	Monterey	Fish meal, fertilizer, fish oil.
Imperial Glue Co.	San Francisco	Fish meal, fish oil, fertilizer and glue.
International Pkg. Corp.	San Pedro, San Diego	Fish meal, fish oil.
Monterey Canning Co.	Monterey	Fish meal, fish oil.
Monterey Fish Meal & Oil Co.	Monterey	Fish meal, fish oil.
Los Angeles Tuna Canning Co.	Long Beach	Fish meal, fertilizer, fish oil.
Nielson & Kittle	San Pedro	Fish meal, fish oil.
San Pedro Packing Co.	East San Pedro	Fish meal, fish oil.
Schaefer, M. N. Co.	San Rafael, Monterey	Fish meal, fish oil.
South Coast Canning Co.	Long Beach	Fish meal, fish oil.
Southern California Fish Co.	East San Pedro	Fish meal, fish oil.
Southern Reduction Co.	San Diego	Fish meal, fish oil.
Spano Packing Co.	San Pedro	Fish meal, fish oil.
Van Camp Sea Food Co.	San Pedro	Fish meal, fish oil.
West Coast Canning Co.	Long Beach	Fish meal, fertilizer, oil.
White Star Tuna Packing Co.	San Pedro	Fish meal, fish oil.

BRITISH COLUMBIA

Evans, Coleman & Evans	Ladner	Fish oil, fertilizer.
Fish Oil and Products, Ltd.	Prince Rupert	Fish meal, fish oil.

Channel Cat Fish Oil.—A sample of channel cat fish oil extracted by the Welaka Fish Company of Welaka, Fla., was recently obtained by the writer from the Acting Commissioner of the Bureau of Fisheries at Washington. The results of an examination of this oil are presented herewith:

Color: Very pale yellow.
Odor: Not objectionable.
Appearance: Turbid. Contains considerable "stearine."

Specific gravity	.9234
Acid number	10.9
Saponification number	192.
Iodine number	123.
Refractive Index at 25°c	1.4741

Drying test: The oil alone or with 10% drier produces a film that remains wet after three days.

It is apparent from the drying tests that this oil is not suitable in its raw condition to use in the manufacture of paints or varnishes. Heat treatment renders it much more suitable for this purpose.

Salmon Fish Oil.—A sample of No. 1 Salmon Oil, obtained by the writer from the Pacific-American Fisheries at South Bellingham, Wash., has recently been examined, with the following results:

Color: Similar to boiled linseed oil.
Odor: Characteristic fish odor.
Appearance: Clear.

Moisture	0.2%
Specific gravity	.9268
Acid value	9.8
Saponification number	183.
Iodine number	159.
Refractive index at 25°c	1.4788

Drying value: Raw oil tacky after three days. With 10% drier, it sets to a fairly firm film in three days.

It is apparent that this oil is from the King Salmon which generally runs about 158 in iodine value. The oil from Humpback Salmon generally shows an iodine value of 128.

Mr. F. W. Nestelle, chemist of the Pacific-American Fisheries, states that the constants of an oil from the Silver Salmon is represented by the following data:

Specific gravity	.9183
Refractive index	1.4753
Solidification temperature	–3 to –11° C
Free fatty acids (oleic)	0.62%
Iodine number	150.6
Saponification number	193 3
Reichert Meissel No.	00.00
Acetyl value	17.83
Soluble acids	00.0046%
Insoluble acids	92.59%
Unsaponifiable residue	1.69%
Titer test	29.20° C.
Maumene No.	83.40° C.
Hexabromide tests	45.03

Mr. Nestelle states that he has conducted a series of experiments on the west coast with various types of salmon oil. He finds that with suitable drier a paint made with salmon oil will dry in 18 hours to a good surface. Paint of this character has apparently been used by him on the hulls of scows and boats in Alaskan waters as well as on Puget Sound. The results of his tests on the durability of these paints will be available during the latter months of this year.

The writer has found that salmon oil will body readily under heat treatment or with blowing. The products thus obtained, dry better than the raw oil.

CHAPTER V

Changes In Oils Upon Storage

In various papers* the writer has called attention to the storage changes that may take place in oils when ground with pigments. For instance, when paints made with pure raw linseed oil having a certain iodine number are stored for long periods of time, subsequent analysis may show that the oil has a lower iodine number than that called for by the specifications upon which the paints were made. Reactions that are responsible for such changes are of course greatly stimulated at high temperatures. As a result of some investigations that have just been completed, it would appear that similar but less marked changes may take place in pure oils without the presence of pigments, and that such changes will depend to a very great extent upon the method of storing and certain other factors.

During the early part of 1911 the writer secured a quantity of a number of commercial oils for use in experimental paints that were to be exposed to the weather to determine the efficiency of various oil mixtures as paint ingredients. After the painting tests were made, samples of the pure oils were placed in pint glass bottles having ground glass stoppers. The bottles were well filled; an air space above the oil of not more than one inch being allowed in any instance. The oils were placed upon a shelf in the laboratory where they were exposed to indirect light and to ordinary room temperature (in the summer not over 105° F. and in the winter not less than 35° F.). In November, 1914, portions of the oils were removed from the bottles and examined as a check against the original determinations. Air was

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*"The Effect of Pigments upon the Constants of Linseed Oil." J. Frank. Inst., Oct. 1912, pp. 415-423. "Changes Occurring in Oils and Paste Paints, Due to Autohydrolysis of the Glycerides." **Ibid.** May, 1914, pp. 533-540. "A Study of Some Curious Painting Phenomena." **Ibid.** June, 1915, pp. 681-695.

of course admitted during this procedure. During September, 1916, the oils were again examined; further quantities being removed for this purpose. The bottles were again placed upon the shelf and allowed to remain there until March, 1919, when further quantities were removed for examination. The results obtained on these samples are given in Table 27. In Table 28 are shown the results on a series of oils obtained during 1914 and kept under similar conditions as those shown in Table 27. In Tables 29, 30 and 31 are given the results on special oils, the history of which is given in each table.

Previous to the examination of the oils in March, 1919, a record was made of the approximate contents of each bottle so that some information might be on record as to the amount of air space above the oil and the appearance of the oil. This record is given below:

Appearance of Oils in Containers in March, 1919, Previous to Analysis

Oil	No.	1—1/4	full.	Very slight sediment.
"	"	2—3/4	"	Very clear.
"	"	3—3/4	"	White sediment at bottom. Globular-like oxidation at surface.
"	"	4—2/3	"	Completely solidified to white mass, crystalline at surface.
"	"	5—1/3	"	Very clear.
"	"	6—1/4	"	Highly viscous. Clear.
"	"	7—2/3	"	Clear. Slight film at surface.
"	"	8—3/4	"	Highly viscous.
"	"	9—3/4	"	White sediment at bottom. Globular-like oxidation at surface.
"	"	10—1/2	"	Clear.
"	"	11—1/2	"	Slight white sediment at bottom. Clear.
"	"	12—1/4	"	Heavy white sediment at bottom. Clear.
"	"	13—2/3	"	Dark sediment.
"	"	14—2/3	"	Clear. Film at surface.
"	"	15—2/3	"	Very clear.
"	"	16—1/3	"	Very clear.
"	"	17—1/3	"	Very clear.
"	"	18—1/4	"	Dark sediment at bottom.

Oil No. 19—1/5 full.	Dark sediment at bottom.
“ “ 20—3/4 “	Lower third of oil completely solidified to white mass. Upper part clear.
“ “ 21—1/3 “	Completely solidified to white mass.
“ “ 22—3/4 “	Clear also in this can, which was 9/10 full, but showed fungus streaks in oil.
Oil No. 23—2/3 full.	Completely solidified to milk-white mass.
“ “ 24—1/4 “	Very clear.
“ “ 25—1/2 “	Very clear.
“ “ 26—3/4 “	Clear.
“ 27—2/3 “	Lower third of oil black mass with white specks throughout and a blanket of milk-white granules at top.

The precipitated foots and curious form of oxidation products at the surface of the fish oils was of a distinctive nature. The solidification of the tung oils to a white granular mass is characteristic of these oils when exposed to light for long periods. This condition makes necessary the determination of refractive index at 60° C. The matter settled out from the corn and cottonseed oils was very flocculent and white.

It will be noted from a study of the charts that the ageing of nearly every oil causes a drop in the iodine number, which is accompanied by a rise in the specific gravity, saponification number and acid number. Two special exceptions to the rise in saponification number are shown by oils Nos. 8 and 13. It is probable that the highly over-oxidized condition of oil No. 8 may be held responsible for the results shown. In the case of oil No. 13 the low result may be due to the condition of the oil, a considerable amount of material having precipitated out. This precipitate was of a highly acid character and consequently may have been the means of lowering the acid value of the clear oil.

Special attention is directed to the rapid increase in acid number shown by oils Nos. 3, 9 and 19 (fish oils). It would appear that marine animal oils are very susceptible to changes which develop considerable percentages of free fatty acid. The subsequent drop in the acid number of No. 19 may have been caused by the precipitation of matter during 1917.

On oils Nos. 1, 3 and 5 the September, 1916, iodine numbers have been omitted, since they had been recorded as being lower than is shown in the 1919 examination. This result not being in accord with the progressive decrease in iodine number would lead to the conclusion that some error was made in these determinations rather than to the assumption that the iodine number of an oil may drop and then increase.

Special attention should be focused on the rather moderate changes in constants shown by corn oil, cottonseed oil, lumbang oil, sunflower oil, and hempseed oil (Nos. 11, 12, 15, 16, and 17 respectively). These oils were all received in excellent condition and were perfectly clear and apparently free from moisture. These factors may have had much to do with their keeping properties.

Most interesting results were obtained with oils Nos. 20 and 21 (American grown tung oils), Table 29. These oils have shown but very moderate changes in acid value, although both developed rather high saponification numbers, and oil No. 21 showed a substantial decrease in iodine number. A comparison of the value of glass and tin for storage purposes is shown in the case of oil No. 22. It seems rather curious that the oil stored in tins should have shown more change than that stored in glass. This may possibly be due to the action of the fungus growth that was in 1919 observed to be present in the tinned sample. Similar action on the same oil in the glass bottled sample may have been inhibited by the action of light.

In Table 30 some data is given on the effect of sterilization of linseed and menhaden oils by heat treatment. The rapid rise in acid value shown by the unsterilized sample of menhaden oil, and the very moderate rise in acid value shown by the sterilized oil would indicate that properly treated oils may be made more or less immune from changes of an undesirable nature. In the writer's opinion the changes that take place in oil upon standing are due very largely to autohydrolysis caused by the presence of either moisture or in some instances to fat splitting enzymes. Whenever oil is heated to a temperature of 105° C. for a sufficient period of time to remove the moisture, and then filtered, a moisture-free, clear and sterile oil will result. Such oil will apparently keep for a long period of time without showing any marked changes.

There was unfortunately no analytical data originally obtained on the samples of oils shown in Table 31, but at the end of nearly three years' storage the constants would indicate that but slight changes have taken place in the oils, with the exception of tung oil 29. This sample was from an unknown source and may have been adulterated.

TABLE 27.

1911-1919 Oil Tests

Analyses were made when oils were first obtained and again at three later periods, as shown below:

	Sp. Gr. at 60° F.	Iodine No.	Sapon. No.	Acid No.	Refrac. Index, 60° F.
1. Linseed Oil—					
March, 1911	.931	186.	188.	2.0	....
November, 1914	.933	185.4	189.6	2.8	1.4867
September, 1916	.936	....	190.2	3.3	1.4798
February, 1919	.943	182.1	192.3	4.8	1.4870
2. Soya Bean Oil—					
March, 1911	.924	129.	189.	2.3	....
November, 1914	.925	130.2	193.1	4.7	1.4813
September, 1916	.937	122.0	192.1	7.0	1.4733
February, 1919	.939	121.7	193.4	7.8	1.4721
3. Menhaden Oil—					
March, 1911	.932	158.	187.	3.9	....
November, 1914	.934	156.3	193.7	16.1	1.4850
September, 1916	.938	....	191.4	19.2	1.4768
February, 1919	.940	156.9	191.5	21.3	1.4838
4. Tung Oil—					
March, 1911	.944	166.	183.	3.8	....
November, 1914	.946	161.5	190.3	5.7	1.5050
September, 1916	.944	158.6	188.7	5.6	1.5138
February, 1919	.948	141.1	191.6	6.0	1.5024

CHANGES IN OILS UPON STORAGE

	Sp. Gr. at 60° F.	Iodine No.	Sapon. No.	Acid No.	Refrac. Index, 60° F.
5. Perilla Oil—					
March, 1911	.94	180.	188.	2.	....
November, 1914	.94	172.	195.4	7.4	1.4874
September, 1916	.939	....	193.3	14.8	1.4767
February, 1919	.941	168.9	192.1	10.5	1.4817
6. Perilla Oil (Special)*—					
March, 1911	.94	192.	189.	3.2	....
November, 1914	.981	123.8	219.4	20.8	1.4978
September, 1916	1.000	122.4	220.9	31.2	1.4840
February, 1919	....	....	....	....	....
7. Heavy-bodied Linseed Oil—					
March, 1911	.968	133.	189.	2.8	....
November, 1914	.992	130.5	200.	6.3	1.4966
September, 1916	....	124.4	206.3	9.0	1.4876
February, 1919	....	133.5	192.2	11.1	1.4892
8. Lithographic Linseed Oil—					
March, 1911	.97	102.	199.	2.7	....
November, 1914	.96	103.4	150.9	13.4	1.4978
September, 1916	.974	108.5	137.7	15.2	1.4890
February, 1919	....	100.2	131.5	18.3	1.499
9. Whale Oil—					
March, 1911	.924	148.	191.	9.2	....
November, 1914	.926	138.2	191.2	17.4	1.4820
September, 1916	....	....	....	....	....
February, 1919	.929	136.9	193.3	20.2	1.4793
10. Boiled Linseed Oil—					
March, 1911	.941	172.	187.	2.7	....
November, 1914	.943	170.	188.	3.1	1.4895
September, 1916	....	....	....	....	....
February, 1919	.948	172.7	193.7	10.9	1.4890

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*This is a special grade of bleached oil that has become highly viscous —possibly through oxidation—as result of exposing to air for a long period.

TABLE 28.

1914-1919 Oil Tests

Analyses were made when oils were first obtained and again at two later periods as shown below:

	Sp. Gr. at 60° F.	Iodine No.	Sapon. No.	Acid No.	Refrac. Index, 60° F.
11. Corn Oil—					
November, 1914	.921	124.8	190.1	4.1	1.4800
September, 1916	.924	121.3	191.1	4.6	1.4707
February, 1919	.926	127.2	191.4	5.3	1.4742
12. Cottonseed Oil—					
November, 1914	.920	111.7	194.3	0.9	1.4781
September, 1916	.924	110.6	192.9	1.4	1.4681
February, 1919	.925	109.4	193.0	0.82	1.4718
13. Rosin Oil (Low Grade 4th Run)**—					
November, 1914	.964	68.9	35.5	32.4	......
September, 1916	.964	66.0	36.6	31.6	......
February, 1919	.965	64.4	22.6	17.9	1.5202
14. Treated Tung Oil***—					
November, 1914	.882	56.4	101.3	7.7	1.4767
September, 1916	.884	53.2	103.2	8.0	1.4660
February, 1919	......	......	......	......	......
15. Lumbang Oil—					
November, 1914	.927	162.	189.	1.0	1.4789
September, 1916	.926	164.0	188.9	1.9	1.4748
February, 1919	.927	161.2	189.4	2.4	1.4749
16. Sunflower Oil—					
November, 1914	.924	124.6	189.3	7.5	1.4796
September, 1916	.923	122.2	190.2	9.0	1.4712
February, 1919	.922	130.1	194.2	8.2	1.4747
17. Hempseed Oil—					
November, 1914	.927	149.4	191.1	3.9	1.4822
September, 1916	.930	146.1	191.0	5.0	1.4745
February, 1919	.930	151.3	191.8	4.9	1.4777
18. Shark Oil—					
November, 1914	.910	132.8	158.9	5.2	1.4815
September, 1916	.915	127.4	163.3	6.2	1.4722
February, 1919	.918	121.4	168.9	8.9	1.4749
19. Sardine Oil—					
November, 1914	.919	134.6	177.3	10.4	1.4800
September, 1916	.962	91.4	180.2	31.1	1.4755
February, 1919	......	76.5	188.7	......	1.4787

**Considerable matter of highly acid character settled out during 1917.

***Heat treated with driers and thinned with mineral spirits; as used in the liquid portion of interior flat paints.

TABLE 29

A. S. T. M. American Grown Tung Oils

	Specific Gravity	Iodine No.	Saponification No.	Acid Number	Refractive Index
20. Oil No. 3-A—					
December, 1914		167.4	190.0	.9	
March, 1919		168.6	193.6	.6	1.5042*
21. Oil No. 4-A—					
December, 1914		168.5	189.6	.6	
March, 1919		163.3	194.8	.5	1.5037*

A. S. T. M. Tung Oils

	Specific Gravity	Iodine No.	Saponification No.	Acid Number	Refractive Index
22. Oil No. 1-B Commercial — December, 1913	.9395	168.7	192.1	3.8	1.5195
March, 1919 (in glass)	.939	169.4	193.4	2.1	1.5177
March, 1919 (in tin)**	.945	160.6	192.9	2.0	1.5178
23. Oil No. 2-B American Tallahasse—					
December, 1913	.939	169.5	191.	.9	1.5210
March, 1919		166.9	193.	.6	1.5037*

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*Because of condition of oils, 60° C., required for determination of Refractive Index.

**Oil in can streaked with fungus growth.

Oils Nos. 20 and 21 were crushed by the Bureau of Chemistry from two lots of tung nuts grown during 1914 in Leon County, Florida. For data on character of nuts, percentage of oil contained therein, evaluation of kernels, etc., see report of Sub-Committee III of Committee D-1 on Testing of Paint Vehicles, Proc. Amer. Soc. for Test. Mater., 1915, p. 211.

Oil No. 22 was a portion of a sample submitted to the Amer. Soc. for Test. Mater. by L. P. Nemzek as representing a large commercial shipment of imported oil that had proved satisfactory in making paints and varnishes.

Oil No. 23 was crushed by the Bureau of Chemistry from nuts grown in China and obtained through the Agricultural Explorer of the Bureau of Plant Industry. For data on character of nuts, percentage of oil contained therein, evaluation of kernels. etc., see report of Sub-Committee III of Committee D-1 on Testing of Paint Vehicles, Proc. Amer. Soc. Test. Mater., 1914, p. 237.

TABLE 30

RAW LINSEED OIL

(Untreated and Sterilized)

	Specific Gravity	Iodine No.	Saponification No.	Acid Number	Refractive Index
24. Original Oil (not sterilized), examined 1911	.931	186.	188.	2.0	....
Original Oil (not sterilized), examined November, 1914	.933	185.4	189.6	2.8	1.4867
Original Oil (not sterilized), examined February, 1919	.943	182.1	192.3	4.8	1.4831
Original Oil (sterilized November, 1914*), examined February, 1919	.935	181.5	191.8	0.9	1.4816

RAW MENHADEN OIL

(Untreated and Sterilized)

	Specific Gravity	Iodine No.	Saponification No.	Acid Number	Refractive Index
25. Original Oil (not sterilized), examined 1911	.932	158.	187.	3.9	....
Original Oil (not sterilized), examined November, 1914	.934	156.3	193.7	16.1	1.4850
Original Oil (not sterilized), examined February, 1919	.940	156.9	191.5	21.3	1.4802
Original Oil (sterilized November, 1914*), examined February, 1919	.938	156.2	190.1	5.1	1.4802

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*Sterilized by heating to 105° C.

TABLE 31

MISCELLANEOUS TESTS

Analyses made after allowing oils to remain in glass bottles for nearly three years. No determinations were made at start of test.

		Specific Gravity	Iodine No.	Saponification No.	Acid Number	Refractive Index
26.	Peanut Oil	.916	87.1	192.4	3.5	1.4696
27.	Poppy Seed Oil	.931	133.2	194.3	7.4	1.4767
28.	Alfalfa Seed Oil	.926	152.1	186.4	3.8	1.4791
29.	Tung Oil**	.937	153.5	190.1	.8	1.4995

Suggestions Regarding the Selection of Drying Oils.—The selection and use of oils for the products of various industries requires the constant care and skill of the chemist and the factory superintendent who have been trained to know the properties of each type and the service they will perform. The use of oils without such information often results in the production of inferior articles and suggests the advisability of using products that are made under the guidance of skilled technicians.

Moisture in such oils as linseed may be held responsible as the active agent that often causes the formation of free acid. The removal of moisture by heating raw linseed oil to at least 105° C. will guard against such development. Products with which the oil is used should be kept in a thoroughly dry condition so that moisture from this source will likewise be excluded.

Sediment or foots in raw oil are made up largely of mucilaginous and nitrogenous substances, and moisture. These products are objectionable and are often the cause of drying troubles. They further serve the purpose of ideal sprouting media for microorganisms. The use of foots-free, well filtered oil is suggested.

**Source unknown. Very heavy granular deposit settled out, streaked with fungus growth.

NOTE: All of the above determinations were made at 60° F., except in the case of Tung Oil, which had become so viscous by February, 1919, that a temperature of 60° C. was required for the determination of its refractive index. In determining the iodine numbers of the oils, Hanus solution was used in every case except for the Tung Oil, in which case Hubl solution was used.

The use of rosin and rosin driers of an acid nature in a promiscuous manner is to be avoided, as they are often the cause of troubles that later develop. For this reason, boiled oils of the linoleate type are to be preferred in some products, to those containing resinate driers.

Refined oils should be used with care. Acid refined oils often contain the sulphonic acid radical that may continuously affect the formation of free fatty acid over a long period. While refined oils having an acid value of 9-10 are desirable for some work, those having an acid value of not more than 6 are generally preferred. Alkali refined oils are substantially neutral and may be used with much greater safety. The lack of grinding properties possessed by alkali refined oils would suggest their use rather as thinning media to be added to products ground with moisture-free raw oil of normal acid value.

CHAPTER VI

New Foreign Oils

An Interesting Foreign Seed Oil (*Palo maria.*)—The writer recently secured samples of an oil that had been shipped into this country as tung oil, in 5-gallon tin containers, together with a large shipment of commercially pure tung oil in similar containers. The peculiar aromatic and resinous odor and the dark color of the oil led the manufacturer who had purchased this shipment to believe that the cans contained an impure grade of tung oil. Samples were accordingly examined, and the writer's laboratory results are shown below.

The oil was clear, but of a dark green tint. It possessed a highly aromatic odor. It was soluble in the usual oil solvents, but upon admixture with 10% of lead and manganese drier it became cloudy and highly viscous, precipitating out a green mass, apparently due to reaction with the lead. When spread upon glass, four days were required before tackiness was shown and a globular film resulted. When admixed with 10% of drier, the film was still tacky at the end of four days. When admixed with equal quantities of linseed oil and 10% of drier or with equal quantities of tung oil varnish, drying occurred in four days' time. On heating to 200° C. for 3 hours and incorporating in the mass 1% of red lead as drier, a highly viscous and rather cloudy mass was obtained, which did not show good drying properties. It is apparent that special treatment of the oil would be required to make it applicable to the paint industry. Analysis of the oil is shown herewith.

ANALYTICAL RESULTS.

Refractive index	1.4743
Specific gravity	.934
Acid value	46.1*

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*Phenolphthalein was used as indicator when making the acid value determinations, but difficulty was experienced in observing the end point. Using litmus as an indicator, acid values of 5 and 6 were obtained.

Saponification value	193.0
Iodine value	96.5
Volatile at 200° C.	Negative
Rosin test	Negative
Petroleum test	Negative
Tung oil heat test (Browne)	Negative

The constants of this oil were so different from those of tung oil as to make it quite certain that the sample was not an adulterated tung oil but consisted of some foreign seed oil that had been shipped, possibly by mistake. A search of the literature revealed the product as apparently identical with an oil grown in the Philippines and known as Palo maria, the botanical name of which is Calophyllum inophyllum. In this connection, the following quotation from page 444 of a paper entitled "Commercial Utilization of Some Philippine Oil-Bearing Seeds," by George F. Richmond and Mariano Vivencio del Rosario, published in The Philippine Journal of Science, Vol. II, No. 6, Section A (December, 1907), may be of interest. The present writer showed a sample of the oil referred to above to Mr. Richmond, one of the authors of the above paper, who believed it to be identical with the sample described in his paper.

> *Calophyllum inophyllum* (*Palo maria*).[7]—A large hardwood tree belonging to the family *Guttiferæ*, found along the seashores throughout the tropics of both hemispheres. In the Philippines it is known as *palo mari de la playa* to distinguish it from *Calophyllum wallichianum*, the widely distributed form found in upland forests, known as *palo maria del monte*. It has a beautiful, dark green, thick, fine-nerved leaf, 4 to 5 inches long, from whence is derived the name *Calophyllum* or *Schönblatt*. The fruit is the size of a walnut, with a fleshy rim containing a thin-shelled seed which incloses a hard, oily kernel. The fruit of the mountain variety is only two-thirds to three-fourths the size of that growing near the seashore. Each tree yields several bushels of nuts per year. There is no es-

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[7] Tavera: Medicinal Plants of the Philippines, Manila (1901), 38.

tablished industry, although in some localities the oil is expressed and used for lights.

Oil from the expressed seeds of *Calophyllum inophyllum* is called domba, and in Indo-English, improperly, laurel-nut oil. The kernels on extraction yield 70 to 75 per cent of a greenish-yellow oil. The oil is not serviceable as an edible fat, since it contains a poisonous resin to which the color and odor are due. On the other hand, it finds application as a natural remedy in skin diseases and rheumatism, and it is used for that purpose in many districts of India; it is exported in considerable amounts from Travancore, particularly from Burma, and under the name of "udiloöl" it has been experimented with in Europe for some time in the treatment of rheumatism.

The seeds of *Calophyllum inophyllum*,[8] a forest tree widely distributed in the eastern tropics, furnish an oil known by various names (dilo, domba, pinnay, poon seed or tamanu oil); when mixed with pigments, this forms a paint that dries in twelve hours, without any previous boiling. Owing to the large yield of oil and the plentifulness of the trees in India, Ceylon, the Malay Archipelago and Java, and the South Pacific islands, etc., this oil appears likely to be an important article in the future.

Domba oil[9] is obtained from the nuts of *Calophyllum inophyllum*. It is chiefly used as an embrocation for rheumatism, and for illuminating purposes. Domba oil is sold in Burma at four times the Calcutta price of castor oil, which it resembles. G. Fenler[10] has made a chemical examination of the oil from *Calophyllum inophyllum*. He describes it as greenish-yellow in color, of a bitter, pungent taste, soluble in all proportions in the usual solvents, but insoluble in absolute alcohol. The following values are recorded:

Specific gravity at 15° C	0.942
Reichert Meissel number	.13
Acid value	28.45
Saponification value	196
Iodine value	92.8

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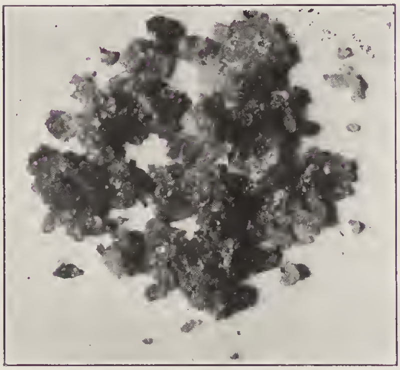
[8] Alder Wright, C. R.: Fixed oils, fats, butters and waxes (1903), 348.

[9] *J. Soc. Chem. Ind.* (1901), 20, 624.

[10] *Chem. Ztschr.* (1905), 29, 15.

By treating with caustic soda the oil yields a greenish resin of semiliquid consistency, soluble in alcohol. The fatty acids consist mainly of palmitic, oleic and stearic. It is stated that the oil is also used in the manufacture of soap.



Meal from Polymerized Tung Oil.

Meal from Soft Lumbang Oil.

Aleurites Trisperma, "Soft Lumbang" Oil.—What appears to be an excellent drying oil that has not yet found its way into the paint and varnish industry was recently delivered to the writer by Mr. R. A. Young, Plant Introducer of the Bureau of Plant Industry, U. S. Department of Agriculture. This oil comes from the seeds of the same botanical species as tung oil and lumbang oil. It is known as Aleurites trisperma or "soft lumbang," which differentiates it from true lumbang, Aleurites moluccana. Aleurites trisperma or soft lumbang is a tropical species apparently of limited distribution. It is grown in the Philippine Islands where it is said to fruit irregularly. According to Mr. Young, the Porto Rico Experiment Station reports that "soft lumbang" fruits in Porto Rico even more freely than true lumbang, and it is possible, therefore, that substantial quantities of the oil might be obtained for importation into the United States.

The only published data to be found regarding this oil appears in the Philippine Journal of Science, Vol. II, No. 6, Section A, page 443, as follows:

> "*Aleurites trisperma.*—Another species of candle nut, called *lumbang banucalag* in Cavite and *baguilumbang* in Tayabas, is entirely confined to the Philippines, where it is widely distributed, but not so abundant as the first variety. The tree is about 40 feet high, and the fruit matures in June and July. Both species are propagated by seed or by cuttings and are of very rapid growth. The oil expressed from the *Aleurites trisperma* is more viscous and darker colored than that from *Aleurites moluccana.* Locally it is used principally for painting boats. The seeds of this variety of candle nut are comparatively thin shelled and are easily crushed.
>
> "One kilo of unshelled nuts produced 357 grams of shells and 643 grams of kernels. One kilo of unshelled nuts, crushed and pressed, yielded 327.28 grams of oil or 32.72 per cent, which corresponds to a yield of 50.9 per cent calculated on the kernels. The oil dries more rapidly than linseed oil or the lumbang bato previously described, and is so closely allied to the Chinese wood oil as to make its differentiation difficult."

Two samples of the oil were received. One was crushed from the seed and one from the whole seed; the former, of course, being lighter in color. Both, however, were of a light amber color, somewhat paler than the commercial grades of lumbang or tung oil obtainable in the market. The odor of each was similar, strongly resembling that of tung oil. In body they also resembled tung oil, being rather viscous. Analysis of the oil in comparison with pedigreed samples of tung oil and lumbang oil were made. The results are charted below:

TABLE 32

	Aleurites Fordii, Tung Oil, Chinese Wood Oil	Aleurites Moluccana, Lumbang Oil, Candle Nut Oil	Aleurites Trisperma, Soft Lumbang Oil crushed from seed	Aleurites Trisperma, Soft Lumbang Oil crushed from whole seed
Refractive Index	1.5092	1.4765	1.4929	1.4927
Specific Gravity	.941	.927	.9380	.9383
Acid Value	4.0	2.3	4.4	7.1
Saponification Value	192.	192.3	194.	190.
Iodine Value (Hanus)	Unsatisfactory Method	162.	Unsatisfactory Method	Unsatisfactory Method
Iodine Value (Hübl)	167.8	....	164.2	160.5
Tung Oil Heat Test (Browne Method)	Firm, crumbly jelly in 10 minutes	Negative	Negaitve	Negative

It is to be noted that the Hanus iodine value is unsatisfactory on "soft lumbang" oil, while the Hübl method gives results comparable to those obtained on tung oil. This is another feature which distinguishes "soft lumbang" from ordinary lumbang oil and indicates the similarity between "soft lumbang" and tung oil.

When the "soft lumbang" oil was spread on glass it dried in 34 hours to an opaque, crystalline film, resembling the film that is produced by raw tung oil. When 10 per cent of lead-manganese drier was added to the "soft lumbang" oil a perfectly clear, firm film was obtained on glass in 14 hours. This result indicated that the oil could be used without heat treatment in the same manner as linseed oil in the manufacture of paints. This result differentiates the oil from tung oil which is very difficult to use without heat treatment if perfectly clear and hard films are desired.

A small amount of the oil was exposed to the air and sunlight in a partially filled test tube for a period of one week. At the end of that time, an opaque, crinkled mass, light in color, was shown. This mass was removed from the tube. Upon pressure it readily granulated to a meal resembling the meal formed by crushing heat polymerized tung oil. This peculiar physical condition would suggest its use as a filler for inlay linoleum. A photograph of the material is shown above.

It was found that the granular mass referred to above is of a different nature than the mass formed by the heat polymerization of tung oil. Granulated polymerized tung oil is practically insoluble in all solvents and even little affected by boiling caustic soda. The mealy mass from the "soft lumbang" oil was found to be readily soluble in caustic soda (10% solution) with the formation of a clear soap. It was also found somewhat soluble in boiling alcohol.

As will be noted above in the chart of analytical data, "soft lumbang" oil, like true lumbang oil, is not affected by heat as is tung oil, and does not polymerize when brought to 280° C. and held at that temperature for a period of even 15 minutes. During this process, however, the oil becomes viscous (bodied) and apparently is well suited to use in the manufacture of varnishes. Experiments were made in the production of a varnish with "soft lumbang" oil on the same formula used in the manufacture of a tung oil varnish. For this purpose there was used with both oils a percentage of linseed oil and rosin with lead as the drier and mineral spirits as the thinner. Both oils were treated in the same fashion. The tung oil varnish had better body and slightly more rapid drying properties than the soft lumbang varnish. Both gave very waterproof films.

Stimulation of the production of "soft lumbang" oil in both the Philippines and Porto Rico should result in available quantities of this oil for use in the United States. Its properties make it most desirable for use in the paint and varnish industry.

Philippine Lumbang Oil Industry.—In the March, 1919, issue of the Philippine Journal of Science, appears an article entitled "The Lumbang Oil Industry in the Philippine Islands," by R. H. Aguilar, of the Bureau of Science at Manila. In this article much information is given regarding the proper methods of shelling the nuts and treating the kernels in order to prevent hydrolysis and discoloration of the oil. The comparatively ready separation of the kernels from soft lumbang nuts is pointed out. Production figures are presented, which are of interest. Because of the importance of the above described paper and the limited distribution in this country of the Journal in which it appears, some of the more important paragraphs are presented below as extracts. These will doubtless be of interest to the paint and varnish technologist.

> * * * "There are two kinds of lumbang nuts known in the Philippine Islands from which oils are obtained; lumbang bato (*Aleurites moluccana*)[1] and lumbang banucalag (*Aleurites trisperma*). Lumbang bato is of wider distribution, more abundant, and better known than lumbang banucalag, and the attention of manufacturers at present is devoted to the production of oil from the former. Lumbang banucalag is almost unknown in the Manila market, and when the word 'lumbang' is employed it is generally taken to mean lumbang bato. The Chinese, who are the largest dealers in lumbang oils,[2] are not willing to handle the banucalag variety, because it is supposed that contact causes skin eruptions;[3] but, in my opinion, there is no foundation for this belief.
>
> "The supply of banucalag nuts is irregular and until a definite supply can be depended upon, the extraction of oil from these nuts would hardly be profitable as a separate industry; but no doubt it could be carried on profitably in connection with the extraction of lumbang bato oil. The close similarity[4] between lumbang banucalag oil and Chinese wood oil, and the fact that the latter is so highly appreciated[5] by United States paint manufacturers, constitute inducements for the production of banucalag oil. * * *
>
> "An estimate of the yearly Philippine crop of lumbang nuts is very difficult to make. It would not be legitimate

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[1] Richmond, G. F., and Rosario, M. V., Philip. Journ. Sci., § A 2 (1907), 441-443.

[2] For further data see Brill, H. C., and Agcaoili, F., Philip. Journ. Sci., §A 10 (1915), 113.

[3] Aguilar, R. H., Philip. Journ. Sci., §A 12 (1917), 236.

[4] Richmond, G. F., and Rosario, M. V., Philip. Journ. Sci., §A 2 (1907), 443.

[5] Drugs, Oils, and Paints 30 (1914), 207.

to base such on the annual production of lumbang oil, because the portion of the crop now used for oil manufacture is very small. In 1911 one manufacturer claimed that there was a sufficient available supply with proper machinery to produce 5,000 kilograms of oil per day; this amount would represent about 9,000 kilograms of kernels or about 26,000 kilograms of dry unshelled nuts. On this basis the available nuts would supply a modern oil factory with a monthly capacity of 234 metric tons of nut kernels. The Bureau of Forestry is encouraging the planting of lumbang trees throughout the Archipelago, and it is estimated that approximately a half million trees are being planted annually. Therefore, an increasing supply of raw material for the manufacture of lumbang oil is assured. The number of lumbang trees in the Islands is not definitely known, but the information at hand would seem to indicate that it is possible to secure an abundant supply of lumbang nuts and to make of the lumbang oil industry a profitable business. * * *

"Lumbang bato nuts may be stored for a year or more in a cool dry place without their undergoing an appreciable change in the amount or the composition of the oil; but lumbang banucalag nuts stored for the same length of time, probably due to oxidation, will be greatly affected in both the amount and the composition of the oil, with an increase in its acidity. No mold or fungus was observed on any of the nuts stored. Due to the comparatively thin shell the kernels of lumbang banucalag nuts can be easily separated from the shell after cracking; but considerable difficulty was experienced in trying to find some adequate means of separating the kernel from the shell of lumbang bato nuts; nuts gathered over a year ago showed, when opened, that the kernel still adhered firmly to the shell. Steaming has been used successfully with certain varieties of cocoanuts, but is unsuccessful in this case and, furthermore, darkens the kernel and the oil. The commonest procedure now in use is to crack the nuts and pick out the kernels by means of a pointed instrument, a very tedious operation.

Lumbang Bato (Aleurites Moluccana)

"*Biau* (Misamis, Davao); lumbang (Rizal, Laguna, Zamboanga, Batangas); *lumbang bato* (Cavite). Lumbang is the name officially adopted by the Bureau of Forestry. * * *

"Among the various procedures now in use for the purpose of minimizing the work involved in removing the kernel from the shell of lumbang bato nuts, none of which has been found wholly satisfactory, are the following:

"In some localities the Chinese place large quantities of nuts on the ground, cover them with straw and after burning the straw immediately sprinkle the nuts with cold water. They claim that with this method the nuts burst. In Laguna, Tayabas, and Batangas Provinces, the nuts are placed in tanks of boiling water and left there for from five to six hours. This loosens the kernel, and when sufficiently cool the nuts are cracked and the kernels are separated from the shells. These two methods produce brown kernels from which only brown oil can be expressed.

"In Moro Province, along the coast of Davao, the nuts are dried in the sun until the kernels loosen sufficiently, which may be ascertained by occasionally cracking a few nuts for trial. The drying takes from five to ten days or more, depending upon the condition of the weather; the nuts are then cracked and the kernels removed. This process is very slow, although the kernel usually comes out whole and is of the best quality.

"The method that has been developed in the Bureau of Science laboratory reduces somewhat the time and the labor involved in the preparation of the kernel and has no injurious effect on the oil. The nuts were heated in an oven at 95° C. for from three to four hours, dumped rapidly into cold water, and left over-night. The next morning the shells had burst, and the kernels were separated without difficulty. * * *

TABLE 33.

Constants of Three Samples of Oil

	Oil expressed from fresh kernels	Oils obtained from the market	
		Grade I.	Grade II
Appearance	Light colored	Brown	Dark brown
Specific gravity at 15.5° C.	0.9261	0.9253	0.9237
Saponification value	188	193	194
Iodine number	154	157	160
Acid number	0.55	64.25	106.48

* * * *

"Lumbang Banucalag (Aleurites Trisperma)

"*Baguilumbang, balucalad* (Laguna); *banucalag, lumbang banucalag, lumbang gubat* (Cavite); *balucanag* (Batangas); *lumbang* (Oriental Negros, Camarines). Baguilumbang is the name officially adopted by the Bureau of Forestry. * * *

"Freshly gathered nuts, free from moisture, produce kernels of the best quality, and the yield of oil by expression at

800 kilograms per square centimeter may be as much as 56 per cent. of the weight of the kernels. This amount represents about 35 per cent. of the weight of the dry nuts, whereas the oil obtained from lumbang bato kernels represents only about 19 per cent. The banucalag oil so prepared is of very good quality, light amber in color, and altogether satisfactory in its physical and chemical properties. If the oil is placed in air-tight containers while fresh, it will keep longer and no appreciable change in its composition will be noticed. On the other hand, unshelled nuts that were kept for sixteen months underwent so great a change in their oil value that the yield by expression was reduced from 56 per cent. to 40 per cent. of the weight of the kernel, and the oil was high in free fatty acids and much darker in color. The results obtained are shown in the following table:

TABLE 34

"Constants of Bunucalag Oil

	Oil expressed from the kernels of fresh nuts	Same oil after being kept for 16 months in a well-stoppered bottle	Oil expressed from the kernels of nuts stored in a sack in a cool, dry place for 16 months
Per cent of oil based on the weight of kernels	56		40
Color of oil	Light Amber	Amber	Dark Amber
Specific gravity at 15.5° C.	0.9362	0.9385	0.9441
Saponification value	191	192	200
Iodine value	166	170	160
Acid value	2.22	5.51	49.14

* * * *

"Summary and Conclusions

"The lumbang oil industry, if properly developed, will become a profitable business.

"It is much less desirable for a factory devoted to the manufacture of lumbang oils to purchase kernels than the nuts from which to prepare its own kernels, because oil from kernels that are being sold in the market is almost always of lower grade than that from freshly prepared ones.

"No appreciable change can be noticed in the available oil content of uncracked lumbang bato nuts stored for over a year; but the content and the quality of the oil of unshelled banucalag nuts stored for the same length of time is considerably reduced. In the latter case even the shell is insufficient to protect the oil in the kernel from oxidation by the air, and shelled kernels deteriorate very rapidly.

"Both oils after expression may be kept satisfactorily in proper containers. In view of these facts, a lumbang-oil factory engaged in the commercial expression of both bato and banucalag oils, in order that the maximum yield and best quality of both bato and banucalag oils may be obtained, preferably should commence the season with the expression of banucalag oil and store the bato nuts until the other species is finished."

CHAPTER VII.

Standards for Tung Oil

There is reprinted herewith the standard specifications of the American Society for Testing Materials for Tung Oil, with the revisions proposed in June, 1920. There is also presented a reprint of the adopted report of Sub-Committee III of Committee D-1, of which the writer is chairman, on tests of Tung Oil carried out during the year.

In the past, much Tung Oil has been purchased by the paint, varnish and allied industries, with very little guarantee of purity. Some jobbers in Tung Oil have offered oil subject to arbitrary test. The chemist in the purchaser's plant might thus have no means of checking the results. With the purpose of overcoming this condition, the attached specifications and methods, which were worked out by paint and varnish chemists in many of the most prominent plants in the country, are presented. It is the writer's suggestion that all paint and varnish manufacturers in the future use the results of this work when making purchases of Tung Oil. The oil jobbers who first assist this work by discarding old time trade practices and by offering Tung Oil subject to these specifications, should merit the confidence of the whole industry.

STANDARD SPECIFICATIONS

For Purity of Raw Tung Oil (Chinese Wood Oil) With Revisions Proposed in June, 1920.

I. Properties and Tests

1. Raw Chinese wood oil shall conform to the following requirements:

	Maximum.	Minimum.
Specific Gravity at $\frac{15°.5}{15°.5}$ C.	0.943	0.939
Acid Number	6	....
Saponification Number	195	190
Unsaponifiable Matter, per cent	0.75	....
Refractive Index at 25° C.	1.520	1.515
Iodine Number (Hübl 18 hours)	....	165
Heating Test, minutes	12	....

II. Methods of Testing

2. The recommended methods of testing are as follows:

Specific Gravity.—Use a pykometer accurately standardized and having a capacity of at least 25 cc., or any other equally accurate method, making the test at 15°.5 C., water being 1 at 15°.5 C.

Acid Number.—Weigh 10 g. of oil in a 200-cc. Erlenmeyer flask, add 50 cc. of neutral alcohol, connect with a reflux air condenser (or place small funnel in neck of flask), and heat on a steam bath for ½ hour. Remove from the bath, cool, add phenolphthalein, and titrate the free acid with N/5 sodium hydroxide. Calculate as the acid number (milligrams of potassium hydroxide to 1 g. oil).

Saponification Number.—Weigh from 2 to 3 g. of oil in a 200-cc. Erlenmeyer flask, add 30 cc. of a N/2 alcoholic solution of potassium hydroxide, connect with a reflux condenser, heat on a steam bath for 1 hour, then titrate with N/2 sulfuric acid, using phenolphthalein as indicator. Always run two blanks with the alcoholic potash. From the difference between the number of cubic centimeters of acid required by the blanks and the determinations, calculate the saponification number (milligrams of potassium hydroxide to 1 g. of oil).

Unsaponifiable Matter.—To 25 g. of oil add 15 cc. of an aqueous solution of KOH (200 g. of KOH dissolved in water and made up to 300 cc.) and 35 cc. of 95 per cent alcohol. Connect with a reflux condenser and heat on the water bath for 1 hour with occasional shaking. Transfer to a separatory funnel containing a little water and wash out flask with water, using in all 125 cc. Cool, add 200 cc. of ether and shake vigorously for 1 minute. In a few minutes the ether solution will separate perfectly clear. Draw off the soap solution into another separatory funnel. Shake the soap solution with three 100-cc. portions of ether. Unite all the ether portions and wash with three 30-cc. portions of water. Filter the ether solution (small portions at a time) into a tared flask, distill off the ether and dry flask and contents to constant weight at 95 to 100° C. in a steam oven. Report the percentage of unsaponifiable matter.

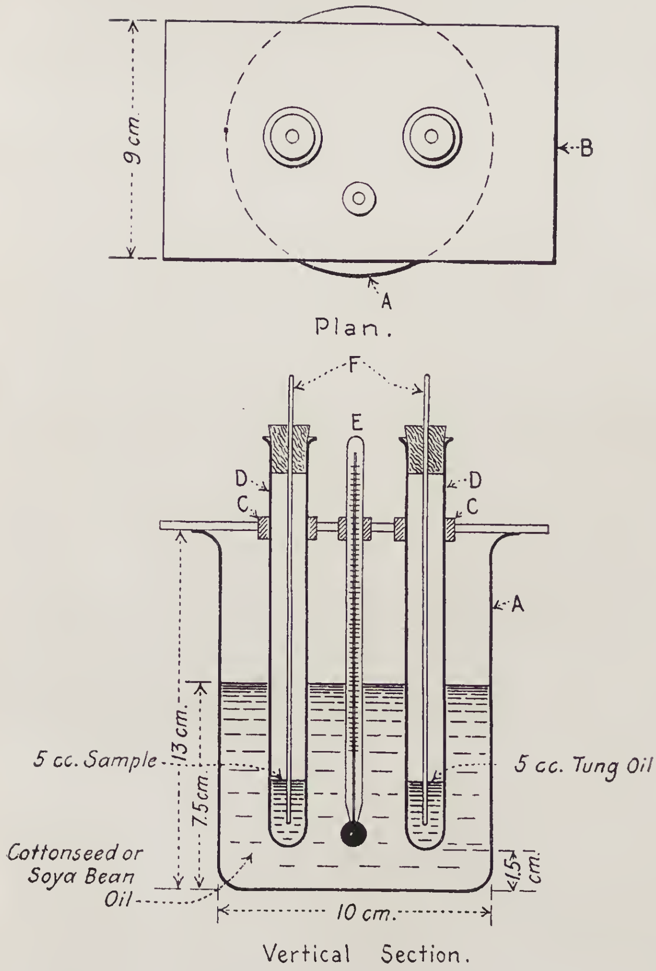
Refractive Index.—Use a properly standardized Abbe refractometer at 25° C., or any other equally accurate instrument.

Iodine Number (Hübl).—Place a small quantity of oil into a small weighing bottle or beaker. Weigh carefully. Transfer by dropping from 0.2 to 0.3 g. to a 500-cc. bottle having a well-ground stopper, or a specially flanged neck, iodine-test Erlenmeyer flask. Reweigh the weighing bottle or beaker to determine the amount of oil used in the test. Then dissolve the oil in 10 cc. of chloroform and add an amount of Hübl solution containing twice the amount of iodine that will be absorbed by the oil. Stopper the flask, shake well, and place in a dark closet for 18 hours. Add 20 cc. of a 10-per-cent solution of potassium iodide and 100 cc. of distilled water. Titrate with N/10 sodium thiosulfate, using starch as an indicator. Blank tests must be made. From the difference between the amounts of sodium thiosulfate required by the blanks and the determination, calculate the iodine number (centigrams of iodine to 1 g. of oil).

On account of the fact that Hübl solution after preparation is apt to deteriorate in strength, it is considered advisable to have prepared the two component parts of Hübl solution, namely, a solution of mercuric chloride in alcohol and a solution of iodine in alcohol, of the proper strength, as outlined in text-books. The proper amounts of these solutions may be mixed on the day of use.

Heating Test.—Test tubes containing the oil should be 15 cm. by 16 mm., with a mark near the bottom to indicate 5 cc., and closed by a cork so perforated that a glass rod 3 mm. in diameter can move freely.

Fill an 800-cc. glass beaker (height, 15 cm.; diameter, 10 cm.), with cottonseed oil or soya bean oil to a height of 7.5 cm. Place a thermometer so as to be 1.5 cm. from the bottom of the bath. (See Fig. 1.)



— SPECIFICATIONS —

A. Beaker Glass Pyrex, 800 cc.

B. Cover Plate (Iron or Wood.)

C. Collar Support (Rubber Stopper No. 6.)

D. Test Tubes; 15 cm. x 16 mm.

E. Thermometer, - Small Range.

F. Glass Rods, (3 mm. with Cork Stoppers)

FIG. 1.—Tung Oil Heat Test Apparatus (Revised 1920).

Note.—Collars C may be omitted and tubes supported in present place by aid of wire gauze placed in bottom of oil bath and resting on bottom of beaker.

Use a nitrogen-filled, chemical thermometer; engraved stem; total length 4 to 4½ in., graduated from 210 to 310° C. in 2° intervals; the length between 210° and 310° C. not less than 2½ in. Thermometer glass shall be well annealed.

When the bath temperature is 293° C. (560° F.) and very slowly rising at this point, place the tube containing 5 cc. of the oil to be tested and the tube containing 5 cc. of the control sample, so that the bottom of each tube is level with the lowest part of the bulb of the thermometer. If desired, the collars C may be omitted and the tubes allowed to rest upon a piece of wire gauze placed in the bottom of the oil bath so that the tubes will be 1.5 cm. from the bottom of the bath. Note the time. Remove the source of heat for about 45 seconds and then reapply. Before 2 minutes have elapsed the temperature of the bath will have fallen to 282° C. (540° F.), at which point it should be kept as steady as possible. When the samples have been in the bath 9 minutes, raise the glass rods at intervals of ¼ minute. Note the time when each sample becomes firmly set. At this period the oil will be so stiff that the entire tube may be lifted by aid of the rod if the collar C is omitted from the apparatus. As setting or jellying takes place within a few seconds of fluidity, a good end determination is afforded. Remove the specimens. Heat the bath again to 293° C., and repeat the experiment with fresh portions of the sample.

No stirrer is used in the bath. A screen around the bath enables the temperature to be more easily reached. When the bath oil has become tarry and viscid, it should be renewed; otherwise heating may be irregular.

REPORT OF SUB-COMMITTEE III

Tests of Tung Oil

The subject of the heat test in the present Standard Specifications for the Purity of Raw Tung Oil (D 12-16)[1]

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[1] 1918 Book of A. S. T. M. Standards, p. 576.

has received much consideration during the past year. At the suggestion of members of the committee, the apparatus has been slightly modified. For tests with the modified apparatus there was forwarded to each member of the committee samples of oil as follows:

100 per cent Pure A. S. T. M. Tung Oil.
95 per cent Pure A. S. T. M. Tung Oil— 5 per cent Soya Bean Oil.
90 per cent Pure A. S. T. M. Tung Oil—10 per cent Soya Bean Oil.
85 per cent Pure A. S. T. M. Tung Oil—15 per cent Soya Bean Oil.

No. 1 J. G. Commercial Tung Oil 100 per cent.
No. 2 J. G. Commercial Tung Oil 90 per cent—Soya Bean Oil 10 per cent.

No. 1 A. A. Commercial Tung Oil 100 per cent.
No. 2 A. A. Commercial Tung Oil 90 per cent—Soya Bean Oil 10 per cent.

The oil referred to above as pure A. S. T. M. Tung Oil was of the same batch reported in the Proceedings for 1914.[2] This oil was pressed at the Bureau of Chemistry from Chinese tung-oil nuts delivered by the agricultural explorer of the Bureau of Plant Industry; the nuts having been imported from China by L. C. Gillespie and Co., New York City. This oil had been kept for a period of six years in a black bottle at the Bureau of Chemistry and the Bureau of Standards. It had been opened on only one occasion, about a year ago, at which time it was perfectly clear and fluid. A small sample was then withdrawn. The container was again sealed and kept for a further period of one year, at which time the bottle was opened to obtain samples for the present work. It was then noted that the oil was turbid. Upon placing a vessel containing a sample of the oil in warm water, the oil would immediately clear up.

The soya bean oil used was a commercially pure sample previously used in committee work. The sample of J. G.

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[2] Proceedings, Am. Soc. Test. Mats., Vol. XIV, Part I, p. 237 (1914).

Commercial Tung Oil was recently obtained from one of the largest tung-oil producers in America, who stated that the oil was of the usual high-grade quality. The sample of A. A. Commercial Tung Oil used was recently obtained from one of the largest oilcloth firms in America from a lot that had proved very satisfactory, and which

Table 35.—Constants of Various Oils Used in 1920 Tests—Report by Bureau of Standards

Properties	Linseed Oil	Soya Bean Oil	Pure A. S. T. M. Tung Oil No. 2, 1914.	No. 1 A. A. Commercial Tung Oil	No. 1 J. G. Commercial Tung Oil
Specific Gravity at 15°.6/15°.6 C......	0.9314	0.9297	0.9406	0.9393	0.9404
Acid Number........	4.6	10.75	1.3	4.4	4.8
Saponification Number	191.5	194.2	192.4	193.6	193.2
Iodine No. (Hanus 25 cc., ½ hour)	178.7	135.8	....	....	....
Iodine No. (Hubl 18 hours).............	....	....	170.4	170.0	170.6
Iodine No. (Wiij ½ hour room temperature)....	....	....	163.4	161.1	163.1

It is very interesting to compare the constants obtained for the 1914 A. S. T. M. No. 2 Pure Tung Oil with the results reported by the committee in 1914.

Properties.	Tung Oil No. 2 1914 Analysis.	Tung Oil No. 2 1920 Analysis.
Specific Gravity at 15°.5/15°.5 C......................	0.9396	0.9406
Acid Number	0.90	1.3
Saponification Number	193.02	192.4
Iodine Number (Hubl 18 hours)	169.6	170.4

The Browne heat tests on this oil as conducted in 1914 correspond closely to those obtained in 1920.

Taking in account the limit of accuracy of the determinations, the analysis of this tung oil after an interval of six years seems to show that it is unchanged, with the possible exception that the sample has increased to a very slight degree in acid number.

The conclusion to be drawn from these results is that the formation of white solid material evident in the sample after the storage in a black bottle for six years is not due to polymerization, but to an isomeric change in the elaeostearic glyceride molecule.

in their opinion represented the highest grade tung oil commercially obtainable.

There is given in Table 35 a report of the Bureau of Standards on the analytical constants of these oils. The linseed oil referred to was used in the Hexabromide tests reported separately. It was obtained from a tank-car shipment known to be pure.

The instructions sent out to the sub-committee for work on these oils are presented below:

> The illustration (Page 87) depicts the type of improved apparatus to be used by the members of Sub-Committee III in testing the pure and adulterated samples of Tung Oil recently forwarded for co-operative work. The method of running the test is essentially that given below (Browne method). It should be noted however that a glass cup 11 centimeters in diameter is recommended. It should also be noted that two test tubes are inserted. One tube contains a known or control sample of pure or satisfactory oil as a standard by which to judge the value of any sample that may be placed in the other tube. An iron or wood cover plate is used. A Tagliabue thermometer according to the specification (see page 87) is used.
>
> The method of procedure is given below. By this method the polymerization period for the pure control sample of Tung Oil may, for instance, run 10 minutes. Every additional 5% of adulteration may increase the time of setting approximately 1¼ minutes.
>
> Heating Test Procedure.[1]—Test tubes for containing the oil should be 15 cm. by 16 mm., with a mark near the bottom to indicate 5 cc., and closed by a cork so perforated that a glass rod 3 mm. in diameter can move freely.
>
> "In the experiments the thermometer is placed so as to be 1.5 cm. from the bottom of the bath. In ascertaining the temperature of the bath due attention was given to the cool column of the mercury, so that corrected temperatures are recorded.
>
> "When the bath temperature is 293° C. (560° F.) and very slowly rising at this point, the tube containing 5 cc. of the oil to be tested is fixed in so that the bottom of the tube is level with the lowest part of the bulb of the thermometer. The time is noted, and the source of heat re-

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[1] Quoted from Proceedings, Am. Soc. Test. Mats., Vol. XIV, Part I, p. 242 (1914).

moved for about 45 seconds and then reapplied. Before 2 minutes have elapsed the temperature of the bath will have fallen to 282° C. (540° F.), at which point it should be kept as steady as possible. When the tung oil has been in the bath about 9 minutes, the glass rod is raised at intervals of ½ minute, and when the rod is firmly set the time is again noted.* As setting or jellying takes place within a few seconds of fluidity, a good end determination is afforded. The specimen is at once removed, the bath is again heated to 293° C., and the experiment is repeated with another portion of the sample. There is no difficulty in obtaining results which do not differ by more than ½ minute. No stirrer is used in the bath. A screen around the bath enables the temperature to be more easily reached. When the cotton-seed oil has become tarry and viscid, it should be renewed; otherwise heating may be irregular. The jelly may be removed by half filling the tubes with strong sulfuric acid and placing in a warm place a few hours."

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*If apparatus is used with wire gauze support for tubes in place of C collar support, the end point is noted when oil stiffens so that entire tube may be lifted through opening by aid of the glass rod. (See results of Lindsay and Ross.)

Table 36—Heat Test on Tung Oils

Oils.	Gardner and Reilly.	Bailey, Baldsiefen and Whitescarver	Kohr, McFadden, Fasig and Marling.	Pickard.	S. and P. Waldstein.	White and Crowley.	Steele.	Lindsay and Ross.	Maximum.	Minimum.	Average.
100 per cent Pure American Pressed A. S. T. M. Tung Oil	9¼	9½	9¼	9½	9¾	9 1/12	9½	9¼	9¾	9¼	9½
95 per cent Pure American Pressed A. S. T. M. Tung Oil .... 5 per cent Soya Bean Oil	10½	10¼	9 5/6	10¼	11	10¼	11¼	10¼	11¼	9 5/6	10⅜
90 per cent Pure American Pressed A. S. T. M. Tung Oil .... 10 per cent Soya Bean Oil	11½	11⅛	11½	11¾	12¼	11½	12¼	11½	12¼	11⅛	11¾
85 per cent Pure American Pressed A. S. T. M. Tung Oil .... 15 per cent Soya Bean Oil	13¼	13¼	12 5/6	13	13¼	12 5/6	14	13¼	14	12 5/6	13⅛
100 per cent J. G. Commercial Tung Oil	11¾	12⅜	....	12	12	11½	11¼	11	12⅜	11	11¾
90 per cent J. G. Commercial Tung Oil .... 10 per cent Soya Bean Oil	14	14⅜	....	14	14	13¾	14½	13¼	14½	13¼	14
100 per cent AA Commercial Tung Oil	12	12⅜	....	12½	12¼	12	12¾	11¾	12¾	11¾	12 1/3
90 per cent AA Commercial Tung Oil .... 10 per cent Soya Bean Oil	14½	14⅝	....	14¾	14¾	14 1/6	14¾	13¾	14¾	13¾	14½

The reports sent in by the various observers are presented in Table 36.

With the reports that were sent in comments were included by various observers. These are digested below:

In the work by White and Crowley the results shown represent the work of at least three check determinations on each sample.

S. and P. Waldstein noted that the pure A. S. T. M. tung oil when received was in a cloudy and semi-solid condition. When heated it became clear, but subsequently reverted to its original condition on recooling. This caused them to wonder whether this condition was the natural one for absolutely pure tung oil. They commented upon the fact that the pure American Pressed A. S. T. M. tung oil bodied in approximately 9 minutes whereas all commercial samples received in paint and varnish factories give a heat test of not less than 11 minutes. They approve of the glass beaker which was substituted for the copper beaker as enabling the operator to observe the temperature more closely.

Kohr, McFadden, Fasig and Marling make brief comment on the condition and vicosity of the first four samples.

Bailey, Baldsiefen and Whitescarver suggest that in laboratory work a number of determinations be run on a pure oil and the average gelatinizing time determined. When an unknown oil is tested the standard should be run at the same time and in the same bath. The gelatinizing time of the unknown oil is represented by the observed time plus or minus the same correction which it is necessary to make to the observed gelatinizing time of the standard to correct it to the "average gelatinizing time" as previously determined.

Lindsay and Ross find it very inconvenient to use a rubber stopper for holding the test tubes in the plate, stating that the stopper becomes soft and spongy and does not hold the tubes in place. They found a cork stopper preferable. They also state that they believe the method might be greatly improved by taking as the end point that point at which it is possible to lift by means of the rod the entire test tube through the opening at *C*. For this determination it will be necessary to do away with the rubber stoppers *C* in the plate openings for the tubes. As supports for the tubes to keep them 1.5 cm. from the bottom of the beaker, they recommend the use of a table placed in the oil bath and made of a small bent wire screen. The rods within the tubes are placed in corks having sufficiently large openings so that the rods move freely.

Conclusions.—Through the use of the Browne Heat Test as revised, more concordant results are shown in the hands of the various operators than appear in the work done with the original test in 1914-1915.

It is further apparent that by this revised method tung oil containing as low as 5 per cent soya bean oil can be differentiated from pure tung oil. The use of this test with revisions noted is therefore recommended to the paint and varnish industry for the examination of tung oil.

It would further appear from the results of the heat test run by the committee from 1914 to date, that most of the imported commercial tung oils contain appreciable quantities of other oils. In order to determine whether this conclusion is justified, the committee has arranged through different sources to obtain absolutely pure tung oil expressed in China under responsible supervision. Samples thus obtained will be used during the coming year in co-operative work.

It is recommended that the committee be instructed to propose a revision of the present Standard Specifications for Purity of Raw Tung Oil (Chinese wood oil) (D 12-16) substituting the modified Browne Heat Test for the Heat Test as now issued. It is also recommended that the Iodine Jelly Test be omitted until further work is done thereon.

CHAPTER VIII

Hexabromide Test for Determining Purity of Linseed Oil

Work on Hexabromide Values of Linseed Oil

A new method of determining the hexabromide value of linseed oil having been worked out by Dr. Steele and Mr. Washburn* of the Bureau of Standards, and a modification of this method by Mr. Bailey, it was the desire of the sub-committee to determine their value and whether fairly close checks might be obtained with these methods when followed by various operators. Accordingly, samples of the following oils were sent out to the sub-committee for this work by the writer as chairman of the committee.

Linseed Oil	100 per cent
Linseed Oil	85 "
Soya Bean Oil	15 "
Linseed Oil	75 "
Soya Bean Oil	25 "
Linseed Oil	65 "
Soya Bean Oil	35 "

The linseed oil and soya bean oil used are described below. Copy of the instructions sent to the sub-committee together with the methods by Steele and by Bailey are given below:

Information has often been requested as to the best method of determining the purity of raw soya bean oil and the purity of raw linseed oil. The iodine numbers of these oils are not always the indices of their purity. Similarly because of the fact that raw linseed oil from North American seed may often have an iodine number as high as 185 while raw linseed oil from South American seed may have an iodine number as low as 170, it has

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*Journal of Industrial and Engineering Chemistry, Vol. 12, p. 52, January, 1920.

been suggested that there would be difficulty in determining whether oil having an iodine number of approximately 170 was obtained from South American seed or really compounded by mixing North American linseed oil with smaller amounts of soya bean oil. Since the ordinarily used methods of chemical analysis would not differentiate between certain mixtures, it has been the desire of the analytical chemist to use some other method of identification. It would appear that such means are available with special Hexabromide Tests. These tests measure the percentage of ether-insoluble brominated products yielded by the oil in question. Raw linseed oil, for instance, has a hexabromide number of approximately 46 whereas soya bean oil has a hexabromide number of usually less than 5.

In order to determine the usefulness of the hexabromide method, the writer as chairman of Subcommittee III on the Testing of Paint Vehicles, has forwarded to members of the committee, samples of pure linseed oil and pure linseed oil mixed with varying amounts of soya bean oil. The co-operative analytical work that is being done on these oils should give information of great interest. The committee will co-operate with Sub-Committee V on Linseed Oil.

There is attached herewith the two methods (Steele and Washburn or Bailey's modification thereof) by which the hexabromide test may be made. The Bailey modification differs from the Steele and Washburn method in that an acetic acid-bromide solution is used instead of a chloroform-bromide solution. The Bailey modification also entirely eliminates the use of chloroform from the reaction mixture and does not require "amyline" or other reagent for removing the excess of bromide after the bromination of the fatty acids. The Bailey modification, however, requires the precipitated hexabromides to first stand over night in an ice chest instead of immediate washing.

A NEW HEXABROMIDE TEST FOR LINSEED OIL

By L. L. Steele and F. M. Washburn, Bureau of Standards

A. *Preparation of Reagents.*

The following reagents are necessary:

1. *Chloroform.*—Shake ordinary U. S. P. chloroform with several portions of water to wash out all the alcohol. Dry the product with granulated anhydrous calcium chloride over night in order to remove all traces of water. Decant from the calcium chloride and distil. Add to the distillate 3 cc. of absolute ethyl alcohol for every 100 cc. of chloroform. Keep in a stoppered brown bottle.

2. *Bromide Solution.*—Mix one part by volume of C. P. bromine* with two parts by volume of chloroform, prepared as above. This solution must be made up fresh each day because it deteriorates upon standing.

3. *Wash Ether.*—Shake ordinary ethyl ether with ten per cent of its volume of ice cold distilled water. Separate and repeat the washing three times. Dry the washed ether with fused calcium chloride over night. Decant the ether through a folded filter into another flask and add thin slices of sodium. Warm gently on a steam bath under a reflux condenser until the evolution of gas by action of the sodium has practically ceased and bits of freshly cut sodium remain bright in the ether. Distil the ether into a dry bottle and add an excess (at least three grams per liter) of finely powdered hexabromide of the fatty acids of linseed oil previously prepared. If no hexabromide is on hand from previous determinations it may be easily prepared as follows: In a centrifuge tube dissolve about 5 grams of the fatty acids of linseed oil in 15 to 20 cc. of chloroform. Place the tube in a freezing mixture and add slowly with shaking, bromine solution until a slight red color is permanent. Add a few drops of amylene to take up excess of bromine. Whirl in a centrifuge until the precipitate has settled and then pour off the chloroform. Rub up the precipitate with 20 cc. of cold absolute ether, whirl in a centrifuge and pour off the wash ether. Repeat the washing with 3 more 20 cc. portions of ether. After drying, the hexabromide is pure enough for the preparation of wash ether. Shake at intervals for two or three hours or allow the mixture to stand over night. Then place the bottle in ice water so that the ether solution will be at zero or not above 2°C for three hours. Decant the ether solution rapidly through a folded filter into a dry bottle and keep tightly corked in order to prevent loss of ether by evaporation.

4. *Amylene.*—This material may be purchased from the East-

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*The authors have observed that samples of bromine marked "C. P." often contain considerable amounts of non-volatile material. All bromine which is used must be redistilled unless it is found that 5 gms. leave no weighable residue upon evaporation.

man Kodak Company. It is one of the organic chemicals prepared in the laboratory of the University of Illinois. It may be prepared in small quantities from amyl alcohol by the method of Adams, Jour. Am. Chem. Soc., 1918, page 1950.

B. *Preparation of the Fatty Acids.*

Weigh approximately 50 grams of linseed oil into a 1½ liter Florence flask, and add 40 cc. NaOH solution (sp. gr. 1.4) and 40 cc. of alcohol. Place the mixture on a steam bath and heat for about ½ hour. Add 1 liter of hot distilled water and insert into the neck of the flask a 2-hole rubber stopper carrying a tube which projects into the flask so that its end is slightly above the liquid, and pass a stream of CO_2 through the tube into the flask. The soap mixture may then be heated, to remove the alcohol, either over a free flame or on the steam bath. If the free flame is used, a capillary "boiler" must be placed in the liquid, since otherwise the soap solution will bump badly. If excessive foaming takes place, the current of CO_2 should be increased until it is strong enough to break up the foam. If the solution is heated on the steam bath, usually about 2 to 3 hours is required to remove the alcohol, while if it is boiled over a free flame, one-half hour is usually sufficient. After the alcohol has been removed, cool the soap solution and acidify with dilute HCl (1-1). Insert a 3-hole rubber stopper, carrying two glass tubes arranged as for a wash bottle, leaving the third hole in the stopper open for an outlet for the CO_2. The inlet tube should extend to just above the layer of fatty acids, and the outlet tube should extend to the bottom of the flask. It is essential that the outlet tube should not extend down more than an inch or two outside of the flask, as otherwise siphoning would take place, causing the liquid to boil inside the tube.

Pass a stream of CO_2 through the system, and boil gently, using a capillary boiler to prevent bumping, until the layer of fatty acids is clear. Plug the hole in the stopper which acts as an outlet for the CO_2. The lower layer will be forced out through the outlet tube by the pressure of the CO_2. In this manner remove as much water as possible without losing any of the fatty acids, then remove the stopper and add about 500 cc. of hot distilled water, shake thoroughly so that the fatty acids are well washed, allow the fatty acids to separate and siphon off the wash water as before. Repeat the washing until the wash water does not give an acid reaction with methyl orange. Before removing the last washing, insert a capillary boiler and boil gently until the fatty acid layer is clear. After the last washing, remove the stopper and suck up with a pipette the last few globules of water. Filter the hot fatty acids through a folded filter under an evacuated bell jar and keep in a well stoppered bottle.

C. *Preparation of Hexabromides.*

Weigh accurately in a weighed centrifuge tube (approximately 6½ inches long by 1 inch in diameter) 1.00 gram (plus or minus 0.05 gram) of linseed fatty acids, prepared as given above. Dissolve in 10 cc. of chloroform and place the tube in a freezing mixture kept as near − 5°C as possible, made by adding a little dilute hydrochloric acid to finely cracked ice. Add bromine solution from a burette at the rate of one or two drops per second, shaking the tube well during the addition. At first the bromine color will be rapidly discharged, but later the mixture will assume a permanent orange color indicating a slight excess of bromine. For most fatty acids of linseed oil about 1 cc. of the bromine solution will be found necessary to give the orange color. At this point run in rapidly 0.5 cc. more of the bromine solution, shake well, and allow the tube to stand in the ice mixture for ten minutes. Remove the tube from the freezing bath and add amylene drop by drop with shaking until the bromine color has entirely disappeared. Usually five to six drops of amylene are sufficient, but a slight excess does no harm. The addition of bromine solution must never be done in direct sunlight.

Attach the tube to a good water vacuum pump (one which will indicate a pressure not greater than 40 mm. of mercury) by means of a new one-hole rubber stopper. Evaporate the chloroform in a vacuum, warming the tube in water at 50 to 60°C to hasten evaporation. The tube must be constantly shaken to prevent bumping of the chloroform. Towards the end of the evaporation, when the contents of the tube become more viscous, rotate and tilt the tube so that the oil will flow about half way up the sides and thus present more surface for evaporation. When practically all the chloroform has been evaporated, place the tube in a bath at 55 to 60°C for fifteen minutes, keeping the suction on.

Detach from the pump and place the tube in a bath of finely cracked ice and water. When the tube is cold pour down its sides 20 cc. of cold wash ether, as prepared above. The wash ether should have been previously placed in four corked test tubes graduated at 20 cc. by a file mark and kept at 0°C in an ice bath. Thoroughly stir and rub up the bromide mixture with a rod, breaking up all the lumps. Return the tube to the ice bath for two minutes and then whirl in a centrifuge until the precipitate has settled into a hard cake and the supernatant liquid is clear. Return the tube to the ice bath for two minutes and then pour off the wash ether, making sure that no solid material is lost. Repeat the washing of the hexabromide precipitate three times in exactly the same way, using three 20 cc. portions of ice-cold wash ether and rubbing up the precipitate thoroughly each time. Use a weighed stirring rod and wash the precipitate adhering to the rod into the tube with the wash ether at each washing of the hexabromide. Afterwards dry and weigh the rod plus the slight coating of precipitate and add the weight of material on the rod to the weight of the main portion of hexabromide. After the fourth ether washing has been poured off, carefully incline and tap the tube and spread the hexabromide precipitate part way up the sides. Warm the tube in water at 50 to 60°C until most of the ether has evaporated. Attach to the suction pump and place the tube in a bath at 60 to 70°C for fifteen minutes. Detach the tube from the pump, cool in cold water to room temperature, wipe dry with a towel and weigh at once. Dry the tube to constant weight in an oven at 100-110°C. The total weight of the precipitate times 100 divided by the weight of fatty acids taken, gives the hexabromide percentage. The hexabromide should dry pure white.

Special Precautions:

1. Have the chloroform dry and adjust its alcohol content to three per cent.
2. Make sure that all the chloroform is evaporated from the impure hexabromide before adding wash ether. This will be accomplished if the water pump indicates a pressure not greater than 30-40 mm. and the tube is heated in the bath at 60°C for two-thirds of its length.
3. Make sure that the wash ether is anhydrous and free from alcohol and that it is saturated with hexabromide at 0°C. Unless the wash ether is allowed to stand at 0°C for a sufficient length of time before filtering off excess hexabromide, it will be supersaturated at 0°C and will give high results. Care should be taken to prevent appreciable loss of ether by evaporation; it is well to cool the stock bottle of wash ether on hot days before uncorking.
4. Make sure that the centrifuge tube containing hexabromide and the wash ether are kept as near 0°C as possible during the process of washing. The finely cracked ice should be replenished at intervals.
5. It has been found that low and non-concordant hexabromide results on pure linseed oil by the new method can nearly always be traced to the use of a faulty vacuum pump or to faulty rubber connections. Therefore, the operator should convince himself by test with a mercury manometer immediately before making the determination that his pump, as used, will give a vacuum of not greater than 30 mm. mercury. Heavy walled pressure tubing should always be used for connections. A faulty pump or faulty rubber connections usually means that the chloroform is not all

evaporated from the impure hexabromide and will exert a solvent action on the hexabromide later on, thus giving low yields.

6. If a Nelson rotary oil pump is available, it is an excellent plan to remove the last traces of chloroform by attaching the centrifuge tube to this type of pump for 15 minutes. The bulk of the solvent should be removed first by means of the water pump.

7. With either pump the tube should be heated in a bath of 60-65°C., during the evaporation of the last traces of chloroform.

BAILEY'S MODIFICATION OF STEELE AND WASHBURN METHOD

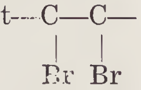
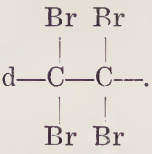
Hexabromide Number

References:

The literature on the determination of hexabromide numbers is not very extensive. The following are the recent references of value:

Chem. Tech. and Anal. of Oils, Fats & Waxes, by Lewkowitsch, 5th Ed., Vol. I, p. 568.
Farben Zeitung (1912) No. 3 ff.
Muggenthaler, Inaug. Dissert. 1912, Augsburg.
Bailey and Johnson, J. I. E. C. 10, 999.

Principle:

The unsaturated fatty acids when treated under proper conditions with bromine absorb at each unsaturated linkage two or more atoms of bromine depending on the degree of unsaturation. Thus at a double bond—C=C— there is obtained a saturated bromo product  $-\underset{Br}{\underset{|}{C}}-\underset{Br}{\underset{|}{C}}-$ and at a triple bond—C=C—four bromine atoms are absorbed to give a saturated compound  $-\overset{Br}{\overset{|}{\underset{Br}{\underset{|}{C}}}}-\overset{Br}{\overset{|}{\underset{Br}{\underset{|}{C}}}}-$. The solubility in ether of the bromo derivatives decreases rapidly with increase of bromine content. Thus the di and tetra bromo compounds are easily soluble, whereas the hexa (and octo) compounds are only very sparingly soluble. This fact is made use of to separate the hexa (and octo) bromo derivatives in carrying out the analytical determination.

Status:

The following method is applicable to the determination of the hexabromide number of saponifiable oils. It must be remembered that the hexabromide number depends upon the method employed in making the determination. It is, therefore, important that in reporting results, the particular method must be specified.

Reagents and Apparatus:

(*a*) *Reagents:*

(1) C. P. sodium hydroxide solution of 1.4 sp. gr.
(2) 95% alcohol.
(3) Distilled water.
(4) C. P. hydrochloric acid.
(5) CO_2 or nitrogen.
(6) C. P. bromine containing no non-volatile matter.
(7) Glacial acetic acid showing no reduction with dichromate or permanganate in the usual test.
(8) Wash ether.

Shake ordinary ethyl ether with 10% of its volume of ice-cold distilled water. Separate the water and repeat the washing three times. Dry the washed ether with fused calcium chloride over night. Decant the ether through a folded filter into another flask and add thin slices of sodium. Warm gently on a steam bath under a reflux condenser until the evolution of gas by action of the sodium has practically ceased and bits of freshly cut sodium remain bright in the ether. Distill the ether into a dry bottle and add an excess (at least 3 grams per liter) of finely powdered hexabromide of the fatty acids of linseed oil previously prepared. (If no hexabromide is on hand from previous determinations, it may be prepared as follows: In a centrifuge tube dissolve about five grams of the fatty acids of linseed oil in 25 cubic centimeters of ether. Place the tube in a freezing mixture and add slowly with shaking bromine solution until a red color is permanent. Let stand for at least fifteen minutes and then whirl the tube in a centrifuge until the precipitate has settled and then pour off the ether. Rub up the precipitate with 20 cc. of cold absolute ether, whirl in a centrifuge and pour off the wash ether. Repeat the washing with 3 more 20 cubic centimeter portions of ether. After drying, the hexabromide so obtained is pure enough for the preparation of the wash ether.) Shake at intervals for two or three hours or allow the mixture to stand over night. Then place the bottle in ice water so that the ether solution will be at zero or not above 2°C. for at least three hours. Decant the ether solution rapidly through a folded filter into a dry bottle and keep tightly corked in order to prevent the loss of ether by evaporation.

(*b*) *Apparatus:*

(1) Steam bath.
(2) Gas burner.
(3) Iron tripod, ring stand and wire gauze.
(4) Round bottom flask of 2 liters capacity.
(5) Separatory funnel, 500 cc.
(6) Bell jar.
(7) Well annealed test tubes 5″ x 1″.
(8) 50 cc. burette.
(9) Glass stirring rods 6″ x 3/16″.
(10) Glass battery jars.
(11) Graduated cylinders 10 and 50 cc. capacity for weighing out samples.
(13) Centrifuge giving about 3,000 R. P. M.
(14) A vacuum showing no higher than 40 mm. pressure.

Determination:

(*a*) *Preparation of fatty acids:*

Weigh approximately 50 grams of oil into a 2 liter round-bottom flask and add 40 cc. of NaOH solution (sp. gr. 1.4=36.50% sol.) and 40 cc. of alcohol. Place the mixture on a steam bath and insert a 2-hole rubber stopper into the neck of the flask carrying a tube which projects into the flask so that its end is just above the liquid. Heat for about one-half hour, passing a stream of CO_2 through the apparatus all the while. Add one liter of hot distilled water and boil the soap solution to remove the alcohol, either over a free flame or on a steam bath. If a free flame is used about one-half hour's boiling will be sufficient, but it may be necessary to insert capillary tubes to prevent bumping of the liquid. If the solution is heated on the steam bath, usually 2 to 3 hours are required. After removing the alcohol, the solution is cooled somewhat and then acidified with dilute HCl (1:1). Warm the mixture until the fatty acids form a clear layer, continuing to pass CO_2 through the system all the time. The fatty acids are separated from the aqueous layer by means of a 500 cc. separatory funnel. The funnel is filled with the mixture, and the fatty acids will float on top, and the aqueous portion is run off. The remainder of the mixture in the flask is added to the funnel and the aqueous portion again run off. A brisk stream of CO_2 is passed into the funnel to replace the air. 300 cc. of hot distilled water is added and the mixture is vigorously shaken. After the fatty acids collect on top the aqueous portion is run off. This washing is repeated until the water is neutral to methyl orange, three washings usually being sufficient. The warm fatty acids are run into a centrifuge tube (1″ x 5″) and whirled for about one minute to collect any remaining water at the bottom. They are then filtered by decantation on to a folded filter under an evacuated bell jar and kept in a well-stoppered bottle.

(*b*) *Preparation of the hexabromides:*

Weigh accurately in a weighed centrifuge tube (1″ diam. x 5″ long) as nearly as possible one gram of fatty acids. It was found that in the case of linseed oil better results are obtained by keeping the weight of the sample as near to one gram as possible, so the deviations from this should not be more than plus or minus 0.02 gram.

Dissolve the fatty acids in 25 cc. of the specially prepared ether and place the tube in a freezing mixture kept at about —5°C. made by adding a little HCl to finely cracked ice. Add bromine solution* from a burette at the rate of about one or two drops per second, shaking the tube well during the addition until a deep red color is produced. This should not be done in direct sunlight. The tube is then allowed to stand in an ice chest over night (about 14 hrs.), the proper precautions being taken to prevent the loss of solvent by evaporation by inserting a stopper.

It is necessary to let the tube stand for this period of time because in the case of oils which contain only a small amount of linolenic acid (soya bean oil is a good example) the precipitation of the hexabromide proceeds more slowly than in the case of an oil with a larger content of linolenic acid (linseed for example).

Next morning cool the tube by immersion in a bath of cracked ice and rub up the precipitate by means of a weighed glass rod, being sure to loosen any material adhering to the side of the tube. Whirl the tube in a centrifuge till the precipitate forms a hard cake on the bottom, cool in the ice bath, and decant the ether. Add 20 cc. of the wash ether previously prepared and cooled to 0°C. and rub up the precipitate with the glass rod. Return the tube to the ice bath and when cold whirl it in the centrifuge. Return the tube to the ice bath and then remove the ether by decantation. Repeat this washing twice more. After the last washing incline the tube and carefully tap it to spread the hexabromide precipitate part of the way up the sides. Warm the tube in water at 60°C. until most of the ether has evaporated, then attach it for 15 minutes to a vacuum line showing a pressure of 30-40 mm. keeping the temperature around 60°C. Wipe the tube dry and allow it to stand in the balance at least 15 minutes before weighing. To the weight of the precipitate in the tube add the weight of the slight amount

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*5 cc. bromine, 25 cc. glacial acetic acid made up just before use.

adhering to the glass rod. This total weight of precipitate multiplied by 100 and divided by the weight of fatty acids taken, gives the hexabromide percentage.

Notes:

(1) The fatty acids are used instead of the glyceryl esters because the latter give inconcordant results.

(2) In the case of linseed oil, the weight of the sample should be kept as near one gram as possible.

(3) Care should be exercised in preparing the wash ether for if it is unsaturated with hexabromides according to directions, the results will be low.

The results of the various observers are given in the table below:

TABLE 37
Hexabromide Values

Oils.	Steele-Washburn Method.									Bailey Modification of Steele-Washburn Method.		
	Gardner and Bielouss.	Steele.	Came.	White and Crowley.	Bailey.	P. and J. Waldstein.	Maximum.	Minimum.	Average.	Came.	Bailey.	Average.
100 per cent Pure Raw Linseed Oil	45.9	46.4	46.6	45.4	46.0	46.2	46.6	45.4	46.1	40.5	42.7	41.6
85 per cent Pure Raw Linseed Oil 15 per cent Soya Bean Oil	39.3	......	41.8	39.1	41.9	39.8	41.9	39.1	40.4	34.9	37.5	36.2
75 per cent Pure Raw Linseed Oil 25 per cent Soya Bean Oil	36.4	38.4	38.2	36.0	37.2	36.1	38.4	36.0	37.0	31.5	34.5	33.0
65 per cent Pure Raw Linseed Oil 35 per cent Soya Bean Oil	30.8	33.4	34.8	30.5	33.8	33.5	34.8	30.5	32.8	26.7	31.4	29.6

Comments made by the various observers are abstracted herewith:

S. and P. Waldstein state that when a large percentage of soya bean oil is present in an oil there is formed a large percentage of tetrabromide which may not be completely washed out. They recommend in such instances to increase the portions of ether to 25 or 30 cc. or to increase the number of washings to 5 or 6 in order to overcome high results.

The chairman of the sub-committee has done some experimental work with the idea of developing a method of determining the hexabromide value of oils volumetrically. It was found that the bromine absorption number of the fatty acids of linseed oil were higher than for soya bean oil in some preliminary volumetric work. It is known that the bromine substitution number of linseed oil fatty acids is low while the fatty acids of soya bean oil have a low addition number. Considerable further work, however, will be necessary in order to develop a method that will be satisfactory.

Conclusions.—The Steele-Washburn method of determining hexabromides has given closely concordant results in the hands of several different operators, and is apparently well adapted for determining the purity of raw linseed oil. It is recommended that further work be carried on with the object of shortening the method.

Addenda

Through the kindness of Mr. Herbert S. Bailey and Mr. Baldsiefen of the Experimental Station. E. I. du Pont de Nemours & Co., there are presented herewith two tables showing the iodine and hexabromide numbers of raw and treated linseed oils, and of soya bean oil, tung oil, and various mixtures thereof. It is believed that the data presented in these tables will be of great service to the paint chemist who intends to carry on hexabromide work.

TABLE 38.

Iodine and Hexabromide Numbers of Raw and Treated Linseed Oils

OL No.	Substance	Iodine No. Hanus	Hexabromide No. Steele Method		Hexabromide No. Bailey Method
			By A.	By B.	By A.
553	Linseed oil from North American seed	176.2	42.0	43.3	42.6
553	“ “ “ “ “ “		45.6	41.3	41.9
553	“ “ “ “ “ “		46.3	45.1	
553	“ “ “ “ “ “		46.5	43.8	
553	“ “ “ “ “ “		46.3	45.5	
553	“ “ “ “ “ “		43.6	44.4	
553	“ “ “ “ “ “		39.2		
553	“ “ “ “ “ “		39.1		
565	Linseed oil, commercial refined	176.5	44.0		
563	“ “ “ raw	185.0	43.6	40.0	
563	“ “ “ “		43.8		
563	“ “ “ “		40.5		
574	Linseed oil (Bu. of Stds. sample)	183.0	47.6	47.5	
574	“ “ “ “ “ “		49.6	45.2	
574	“ “ “ “ “ “		47.1	47.3	
574	“ “ “ “ “ “		46.9		
574	“ “ “ “ “ “		46.2		
574	“ “ “ “ “ “		45.7		
535	Linseed oil Ext’d from Argentine seed	189.8	50.0	49.7	
535	“ “ “ “ “ “			50.8	
535	“ “ “ “ “ “			50.7	
641	OL 553 + 5% (1) tung oil drier	166.8		45.2	42.6
641	“ “ “ “ “ “			45.9	41.1
642	“ “ + 10% “ “ “	161.5		44.4	41.0
642	“ “ “ “ “ “			44.1	40.9
643	“ “ + 20% “ “ “	152.5		41.8	39.2
643	“ “ “ “ “ “			42.2	39.0
644	“ “ + 5% grinding japan	164.8		39.7	40.8
644	“ “ “ “ “			39.6	39.2
645	“ “ + 10% “ “	158.9		42.3	39.9
645	“ “ “ “ “			41.1	40.3
646	“ “ + 20% “ “	147.4		40.4	39.9
646	“ “ “ “ “			39.4	39.4
588	“ “ + 10% (1) linoleate drier	165.6	43.6		
588	“ “ “ “ “		42.7		
589	“ “ + 20% (1) “ “	169.3	45.3		
589	“ “ “ “ “		42.7		
589	“ “ “ “ “		43.6		
583	“(2)“ + 0.2% manganese drier	168.6	32.4		

(1) Added without treating oil.
(2) Manganese linoleate boiled in at 250° C.

TABLE 39.
Iodine and Hexabromide Numbers of Soya Bean, Tung and Linseed-Soya Bean Mixtures

OL No.	Substance	Iodine No. Hanus	Hexabromide No. Steele Method		Hexabromide No. Bailey Method
			By A.	By B.	By B.
583	OL 553 + 0.2% manganese drier		34.7		
584	" " + 0.05% " "	171.0	32 6		
584	" " " " "		33.2		
561	Linseed oil, commercial	173.8	41.8		42.6
561	" " boiled				41.9
359	" " heavy bodied	80 8	11.8		
310	Soya bean oil, raw	133.2	5.1	6.5	4 2
310	" " " "		6.1		4 9
310	" " " "		6.3		3.6
565	Soya bean oil, raw commercial	133.2	1.5		
565	" " " " "		5.0		
658	Soya bean oil, cold pressed			8.41	7.27
658	" " " " "			9 38	7.54
659	Soya bean oil, cold pressed			5.95	5.35
659	" " " " "			5.76	5.65
670	Soya bean oil, extracted			3.9	
622	Tung oil			0.0	0.0
572	50% OL 553 + 50% OL 310		26.0	23.0	
572	" " " " " "			23.6	
572X	50% OL 553 + 50% OL 670			24.3	23.3
572	" " " " " "			24.6	24.3

CHAPTER IX

A Method for Determining the Acid Values of Varnishes and Boiled Oils

The acid values of oils or varnishes that are practically non-colloidal (*such, for instance, as raw linseed oil or a spirit varnish made by dissolving a resin such as rosin in benzol*) may be determined by the usual procedure of boiling the product with alcohol and titrating with a dilute alkali in the presence of an indicator such as phenolphthalein.

When an oil has been bodied by heat or by blowing, or when a resin has been fused into an oil, products of a colloidal nature are formed. The acid numbers of these products cannot be satisfactorily determined by the ordinary methods now in use, as described above for non-colloidal oils or varnishes. It has been found, however, that true determinations may be obtained by using a mixture of alcohol and benzol. The action of the benzol is to break down the colloidal condition in which the acids may be occluded, and form a true solution in which the acids are readily soluble. The chemical explanation of the reaction is presented in this paper. The method for determination of acid value is described below.

Method for Making Determination

Benzol-Alcohol Mixture.—Purified 90° benzol is preferable. Denatured alcohol may be used. It must, however, be free from phenol or other materials which would give it a high acid value. Equal parts by volume of the alcohol and benzol constitute the mixture. This mixture should be neutralized with dilute alkali.

Procedure.—One to two grams of the sample of oil or varnish to be examined is weighed into a 200 cc Erlenmeyer flask. 40 cc of the neutralized benzol-alcohol mixture is added. The flask is placed in a water bath and attached to a reflux condenser. The mixture is allowed to boil 30 minutes and then cooled to room temperature. Four drops of standard phenolphthalein solution are added and the mixture titrated with standard N/10 NaOH and KOH solution. The acid number is then calculated in the ordinary manner (the number of milligrams of KOH required to neutralize the free fatty acid in one gram of oil.)

Remarks.—The sharpest end point is obtained when a permanent pink tinge is apparent rather than a dark pink color. As the titration progresses, the mixture tends to become cloudy due to the presence of the water in the standard solution. As the end point is approached, there is generally a noticeable change of color, the solution becoming more or less darkened just before the end point is reached. If during titration there is uncertainty as to whether the end point has been reached, the mixture should be allowed to remain quiet for a minute or two. There will then be a separation in which the oil and solvent will come to the top. The water will remain below. The upper solvent layer will then clear. A very faint pink color may be detected if present.

Explanation and Theory of Tests

Almost all varnishes and bodied oils are colloidal, that is, they are in the colloidal state. This being so with all but a very few exceptions, necessitates that consideration be given to the laws operating in a material in this state, when determining certain of its chemical properties and factors. This is especially true in determining the acid number of a bodied oil or varnish.

In determining the acid number in the ordinary manner, (by heating several grams of the oil with neutralized alcohol for a short time, cooling, and then titrating the mixture with standard KOH or NaOH solution, using phenolphthalein as an indicator) it is often very difficult to get a definite end point or check results. With colloidal varnishes and oils, when following the standard procedure, the first pink color that appears is fugitive, disappearing almost at once. Subsequent additions of the caustic solution cause slightly increased permanency of the pink color. This color even with the addition of a considerable amount of caustic solution, remains only a very short time, rarely more than from two to five minutes. This indefinite end point leads to varying and inaccurate results. If the first appearance of the pink color is taken as the end point, the acid number will be low, as an insufficient amount of solution has been added to neutralize all the free acid existing in the oil or varnish. If a sufficient quantity of solution is added to cause a more stable end point, the results will be high. Due to accompanying reactions, the amount of alkali required to accomplish this, is greater than the amount required to neutralize all the free acid. Therefore the amount of caustic solution required to accomplish the end point is no indication of the amount of alkali required to neutralize all the free fatty acid in the material.

The condition described above does not apply to non-colloidal oils or varnishes. In determining the acid number of these by the standard method, the end point is sharp and definite and the pink color remains for a considerable length of time, generally from 20 to 45 minutes. The sharp, definite end points obtained with non-colloidal oils and varnishes indicate that the reactions proceed in a normal manner. On the other hand, the indefinite end points and inaccurate results obtained with oils and varnishes of colloidal condition indicate that the reactions do not occur in a normal or definite manner.

The principle of the acid number determination is first the solution (molecular) of the free acid in the alcohol or other solvent and second the neutralization of this acid by combination with the alkali. These reactions must proceed normally in order to secure definite and accurate results. All of the free fatty acid must be dissolved by the solvent and the neutralization must be allowed to occur in accordance with the law of mass action governing reactions of this nature.

With colloidal oils and varnishes, conditions are often present which prevent the solution of all of the free acid by the alcohol. Further, as the law of mass action is not operative when a colloidal condition is present, the combination does not occur in a normal manner. When a colloidal condition is present, there is also the possibility of physical reactions between the alkali and the colloidal bodies instead of its combination with the free acid. In view of this it is apparent that the effect of the colloidal condition of an oil or varnish must be given consideration in adapting a method for acid number determination. An extensive investigation of this subject indicates that the following conditions are probably present.

Occlusion of Acids.—A prominent characteristic of colloidal solutions is the property of the disperse phase to occlude within it a portion of the disperse means. As the free acids are generally part of the means, it is probable that a portion of them are occluded by the phase, and are not dissolved by the alcohol. The extraction of these occluded acids and their subsequent solution can sometimes be effected by continued boiling or by successive boilings, titrating after each boiling. This method is, however, only successful when the oil or varnish is only slightly colloidal. With those that are highly colloidal this procedure does not give accurate results, as all of the acid is not extracted and dissolved in the alcohol.

Effect of Chemical Equilibrium.—The presence of a colloidal condition prevents the operation of the law of mass action and tends to establish a chemical equilibrium which prevents the complete combination of the acid and alkali. Such an equilibrium would allow the existence of both free acid and alkali in the mixture without their combination. It is probable that the uncertainty of the results and the indefinite end point is due to some extent to such an equilibrium.

Absorption of the Alkali.—The disperse phase of colloidal oils and varnishes has the property of readily absorbing alkaline bases. In this manner the alkali can be taken up and occluded within the oil or varnish without at once combining with the free acid. When this occurs the alkali cannot indicate its presence by reaction with the indicator and consequently the pink color is discharged. That this reaction generally occurs with colloidal oils and varnishes is indicated by the fact that a quantity of the alkaline solution, considerably in excess of the amount required to neutralize the free acid, can be added without causing a stable pink color. This fact has also been substantiated by investigations of the viscosity increase of oils and varnishes with the addition of alkaline solutions and the treatment of these materials with alkali.

It is apparent from the foregoing that the presence of a colloidal condition prevents the combination of the free acid and alkali in a normal manner and causes indefinite and inaccurate results. It also follows that definite results can only be obtained when this condition is eliminated and the material brought to the condition of a molecular or true solution.

With slightly colloidal oils and varnishes, the colloidal condition can be broken down and the occluded acids dissolved, by repeated boilings, to an extent sufficient to give fairly satisfactory results. The most satisfactory method for this accomplishment is, however, by the use of a solvent in which the colloidal oil or varnish forms a true solution and in which the caustic solution is also soluble. When this condition obtains, the colloidal condition is eliminated, the occluded free acids become dissolved in the solvent and the formation of an equilibrium by the colloidal condition is prevented. The combination of the acid and alkali is then allowed to proceed in a normal manner.

Benzol as the solvent for the above purpose fulfills the necessary requirements. Practically all colloidal oils and varnishes either form a molecular (true) solution with it, or the colloidal condition is reduced to an extent or a state where its presence has no effect on the results. It is not, however, suitable for use alone as the aqueous solution of alkali is not soluble in it and a condition is caused, which is equally as bad as the presence of the colloidal condition. A mixture of one-half each of benzol and alcohol has been found very satisfactory for the purpose. With this mixture of solvent, the colloidal condition is destroyed, and the alkaline solution is readily soluble in the mixture.

Experimental Evidence.—The following results on three bodied oils varying in their extent of colloidal condition will serve to show the comparison between benzol, alcohol, and the benzol-alcohol mixture.

TABLE 40.

	Benzol solvent.	Alcohol solvent.	Alcohol-benzol solvent.
Raw Linseed Oil		3.4	
Same Oil, slightly polymerized—slightly colloidal only	2.1		3.6
Polymerized and oxidized—highly colloidal	9.15	13.1	16.5

With alcohol the end point was not definite or stable.

With benzol the end point was indefinite.

With the benzol-alcohol mixture the end point was sharp and definite.

Below is shown a comparison of the acid values obtained with alcohol and with the benzol-alcohol mixture on a tung oil-rosin mixture at various stages of the bodying. The mixture contained 20% of rosin that had not been treated in any manner. The rosin was melted in the oil and the mixture heated at 450° F. for the time noted.

TABLE 41.

Time of cooking.	Acid value in alcohol.	Acid value in alcohol-benzol mixture.
Melted	36.2	36.4
At 450° F.	35.7	36.4
" " 15 minutes	34.9	35.6
" " 30 minutes	33.4	35.4
" " 45 minutes	32.9	34.8
" " 60 minutes	29.4	35.2
" " 75 minutes	12.9	31.6

The low acid values on the high heats, when alcohol was used for the determination of acid value, explain the general belief among varnish chemists that heating wood oil varnishes always lowers the acid value. The results with the alcohol-benzol mixture show that such a phenomenon does not necessarily take place.

The table below shows the effect of the different solvents and the time of boiling with a slightly colloidal oil and an oil of high colloidal condition.

"A" Polymerized Linseed Oil. Slightly heat treated. Low colloidal viscosity. Only slightly colloidal.

"B" Blown Linseed Oil. High colloidal viscosity. Very colloidal.

TABLE 42.

Oil	Solvent.	Time of boiling.	Nature of end point.	Acid number.
"A"	Alcohol	To boiling point only	Slow	6.37
"A"	Alcohol-benzol mixture	To boiling point only	Quick	6.17
"A"	Alcohol	½ hour	Quick	6.17
"B"	Alcohol	5 minutes	Slow. Indefinite	8 8*
"B"	Alcohol	½ hour	Slow. Indefinite	8.82*
"B"	Alcohol-benzol mixture	5 minutes	Quick. Definite	7.3
"B"	Alcohol-benzol mixture	10 minutes	Quick. Definite	7.3
"B"	Alcohol-benzol mixture	½ hour	Quick. Definite	7.3

It will be noted that with oil "A" which was only slightly colloidal, a true end point was obtained if boiled for a sufficient length of time, indicating that in this case the continued boiling extracts all the acids and that the colloidal condition is not sufficient to affect the results. It will be further noted that the benzol-alcohol mixture eliminated the colloidal condition without the necessity of continued boiling. With oil "B," however, the condition was different. With alcohol alone as the solvent, continued boiling did not break down the colloidal condition. There was practically no difference in the number between the five-minute and the half-hour boiling. In

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*Sufficient caustic solution added to make a fairly stable end point.

each determination an excess of alkali was necessary in order to establish a fairly stable end point. With the benzol-alcohol mixture the end point was quick and definite, occurring with less alkali than with the alcohol. It will be noted that the acid number was the same in each determination, regardless of the boiling time. In the two determinations made with alcohol the pink color remained about ten minutes only. In those made with the benzol-alcohol mixture the pink color was still apparent after standing one hour.

The concentration of the varnish in the solvent has some bearing on the results with the benzol-alcohol mixture. As it is necessary to have the varnish as near to molecular solution in the solvent as possible, it is important that the solution be sufficiently dilute to accomplish this. The effect of the degree of concentration is illustrated by the results shown below.

TABLE 43.

40 cc of benzol-alcohol mixture used for each test on Bodied Linseed Oil

Weight of oil.	Acid number.
.5362 grams	15.28
.5461 "	15.48
.9578 "	15.4
1.7343 "	15.08
3.2005 "	14.5
7.7951 "	14.2
7.8153 "	14.2
11.7862 "	13.9

Further results, presented below, were obtained on a sample of flat wall grinding liquid composed of tung oil, linseed oil and treated rosin. It was quite highly colloidal.

TABLE 44.

Sample.	Benzol-alcohol combination.		Strength alkali.	Number of determinations.	Average.
10 gms..........	25 cc.	25 cc.	N/2	6	10.4
5 "	25 cc.	25 cc.	N/2	6	14.5
1 gm..........	25 cc.	25 cc.	N/10	10	16.0
1 "	45 cc.	30 cc.	N/10	4	15.5
1 "	30 cc.	20 cc.	N/10	4	15.7
1 "	20 cc.	30 cc.	N/10	4	16.0
1 "	100 cc.	50 cc.	N/10	6	14.3

It is apparent from the results shown that the quantities often used in standard methods for acid number determinations, i. e., 10 grams of oil or varnish to 50 cc of solvent is too great to assure accurate results. The proportion of one to two grams of the oil or varnish to 40 cc of solvent gives best results. With most oils and varnishes two grams to 40 cc of the solvent is satisfactory. If, however, the material is very viscous and contains a small amount of thinner, approximately one gram could be used. With the smaller amount, however, there is a greater opportunity for error in manipulation.

CHAPTER X

The Effect of Moisture on the Drying of Oils and Varnishes

Paint or varnish applied during moist weather often shows a more rapid set than when applied during very dry weather. It would appear, however, that the initial speed of drying shown in an atmosphere containing a high degree of humidity is followed by a secondary reaction, during which water is taken up by the partially dried film. This would possibly account for the lesser resistance of such films to the further action of the weather. On the other hand, films dried in moisture-free air may lack elasticity, due to the rapid progressive oxidation that takes place. These results would point toward the presence of a limited amount of moisture in the air as a condition affording the best results. It is the writer's opinion that an atmospheric humidity of from 20° to 30° gives the best results in the drying of paint and varnish.

The curious phenomena noted above led the writer to conduct a series of drying tests to determine, if possible, whether the introduction of different types of drier would show any differences in the drying of films in moist atmosphere. For the purpose of the test, a series of metals were selected, and made up into driers of the types that are ordinarily used in paint and varnish manufacture. These driers were introduced into raw linseed oil in such proportion that there would be present in each sample 0.2% of metallic oxide, regardless of the amount of drier in various forms added. In no case was the temperature of the oil brought to over 100° C. during the introduction of the drier.

The tests were all conducted in a special cabinet which was built for the purpose, and which consisted of an inner box of galvanized iron fitted with glass shelves. The box was perforated and fitted with flues so as to allow the freest possible circulation of air. This inner box was surrounded at a distance of about 4 inches by an outer wooden box fitted with glass doors. Inside the wooden box and just beneath the central flue of the inner box was placed an electric hot plate by means of which a constant temperature could be maintained. Arrangements were also included for saturating the air with moisture when desired.

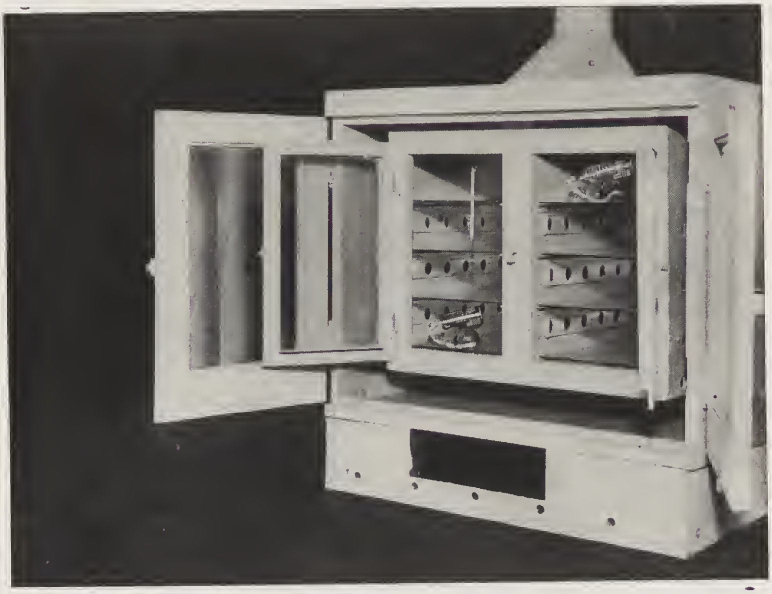


Fig. 2.

DRYING CABINET USED IN TESTS

Perfectly clean, dry, glass plates four inches square were weighed on an analytical balance to a tenth of a milligram. A few drops of one of the oils described above was poured on the centre of each plate and flowed evenly over its surface. It was allowed to drain for five minutes in a nearly vertical position and the edges were then carefully wiped so as to leave a margin of about one-half inch all around for convenience in handling. The plates were again weighed, the amount of oil noted, and then placed flat on the shelves in the cabinet. The amount of oil carried by each plate was approximately 0.2 gram. The plates were then weighed every twenty-four hours for a period of about fourteen days, or until they had become fairly constant in weight.

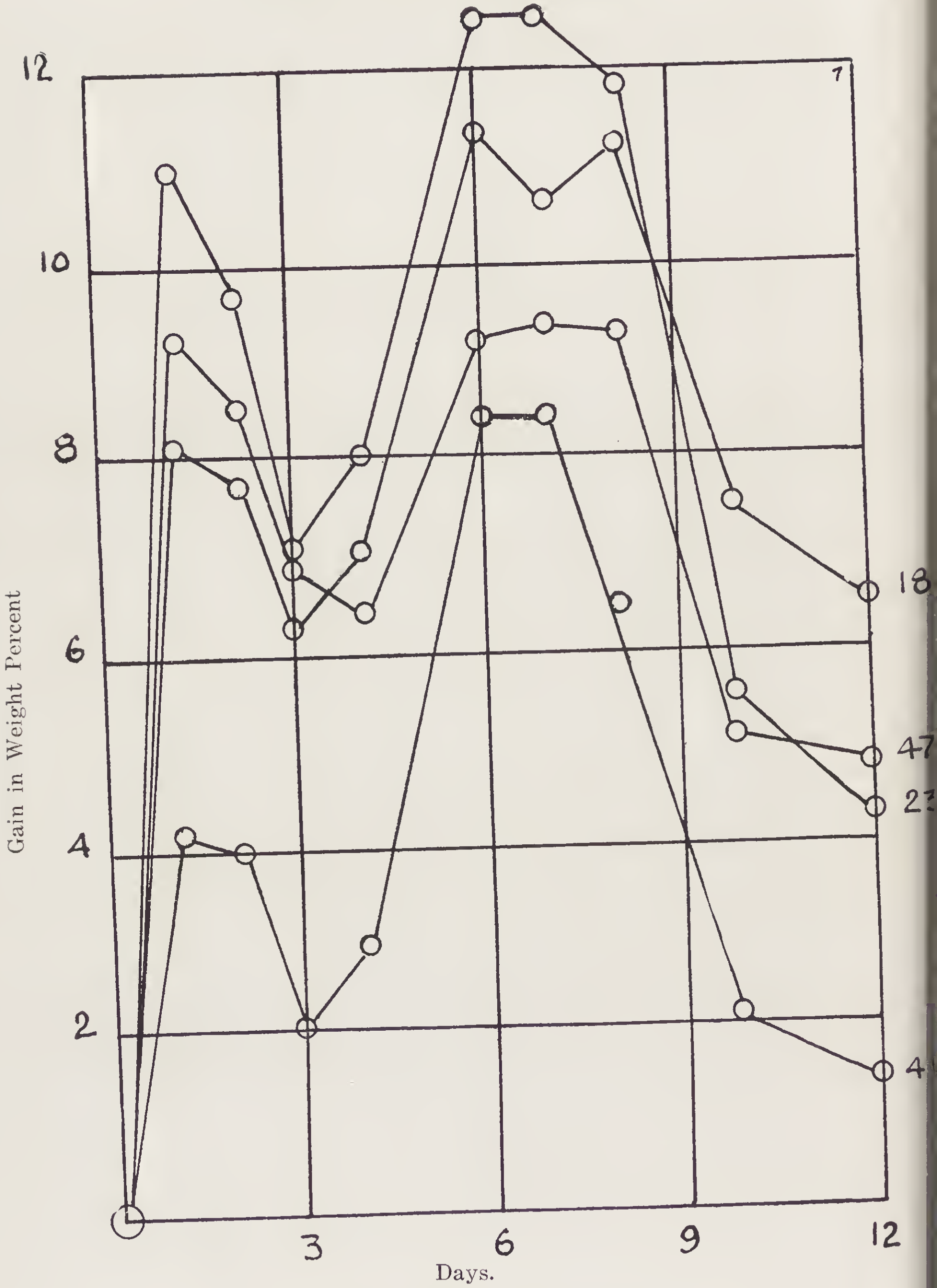


Fig. 3.

LINOLEATE DRIERS (Moist Cabinet).
No. 18—Aluminum Linoleate.
No. 47—Lead Linoleate.
No. 23—Zinc Linoleate.
No. 41—Cobalt Linoleate.

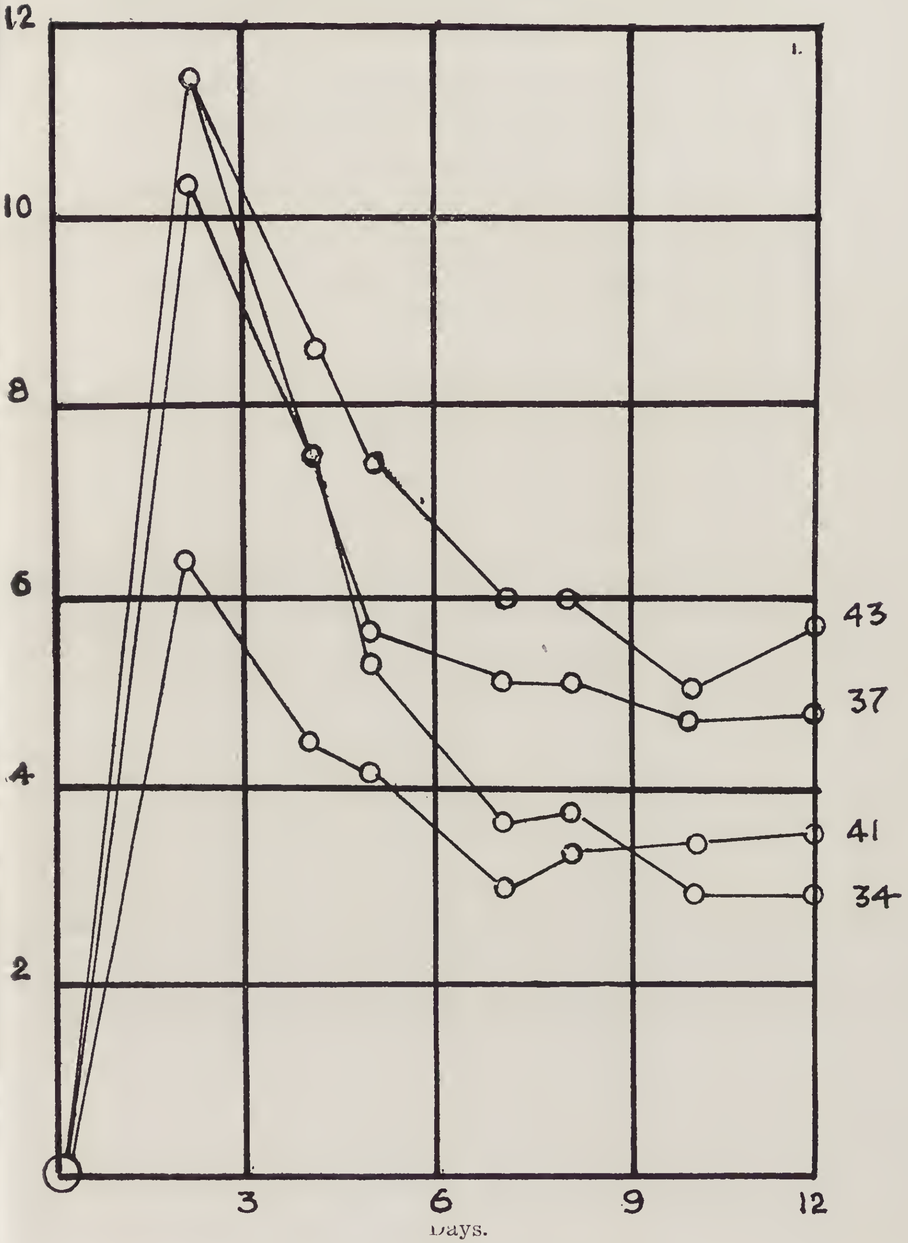


Fig. 4.

LINOLEATE DRIERS (Dry Cabinet).
No. 43—Lead Linoleate.
No. 37—Zinc Linoleate.
No. 41—Cobalt Linoleate.
No. 34—Aluminum Linoleate.

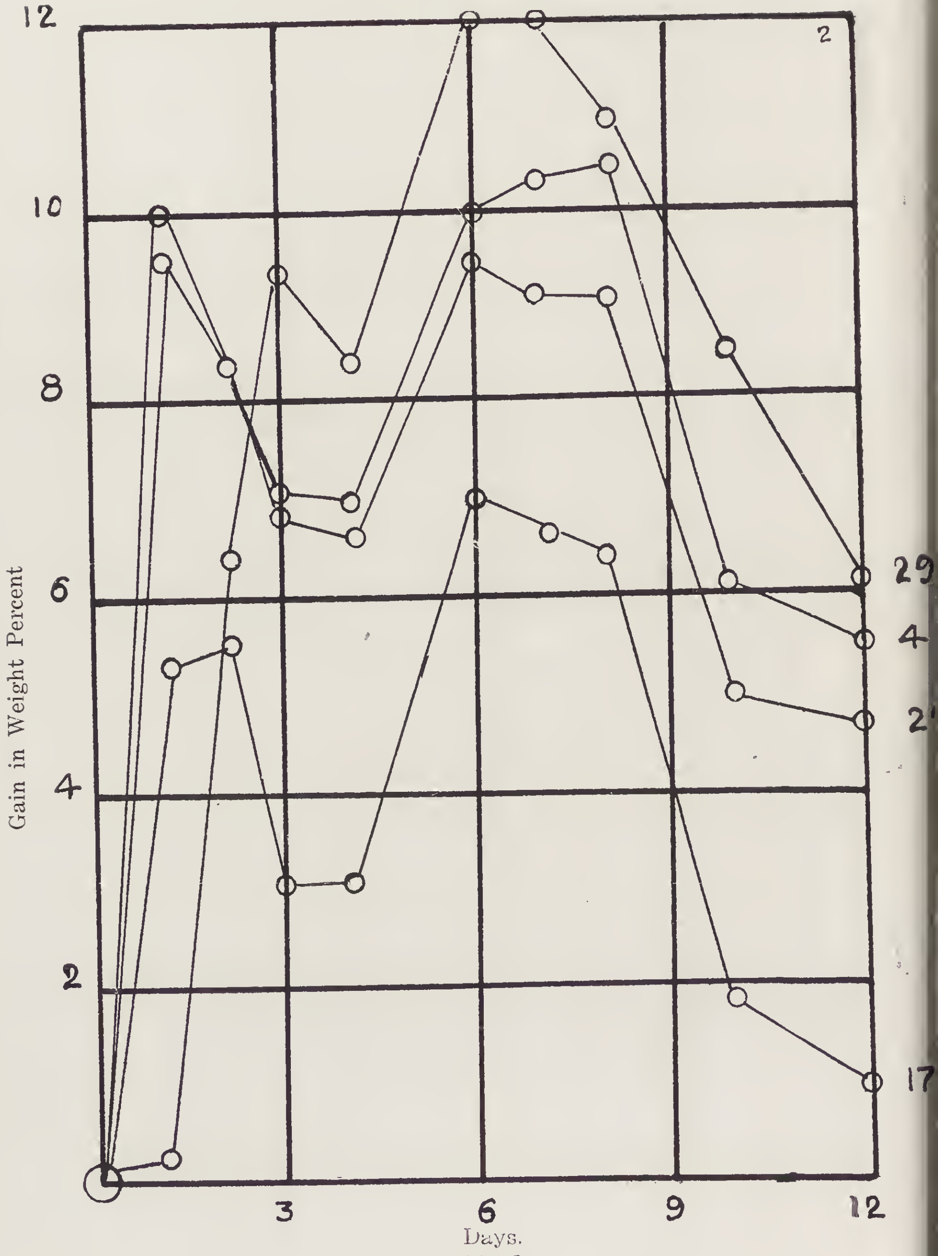
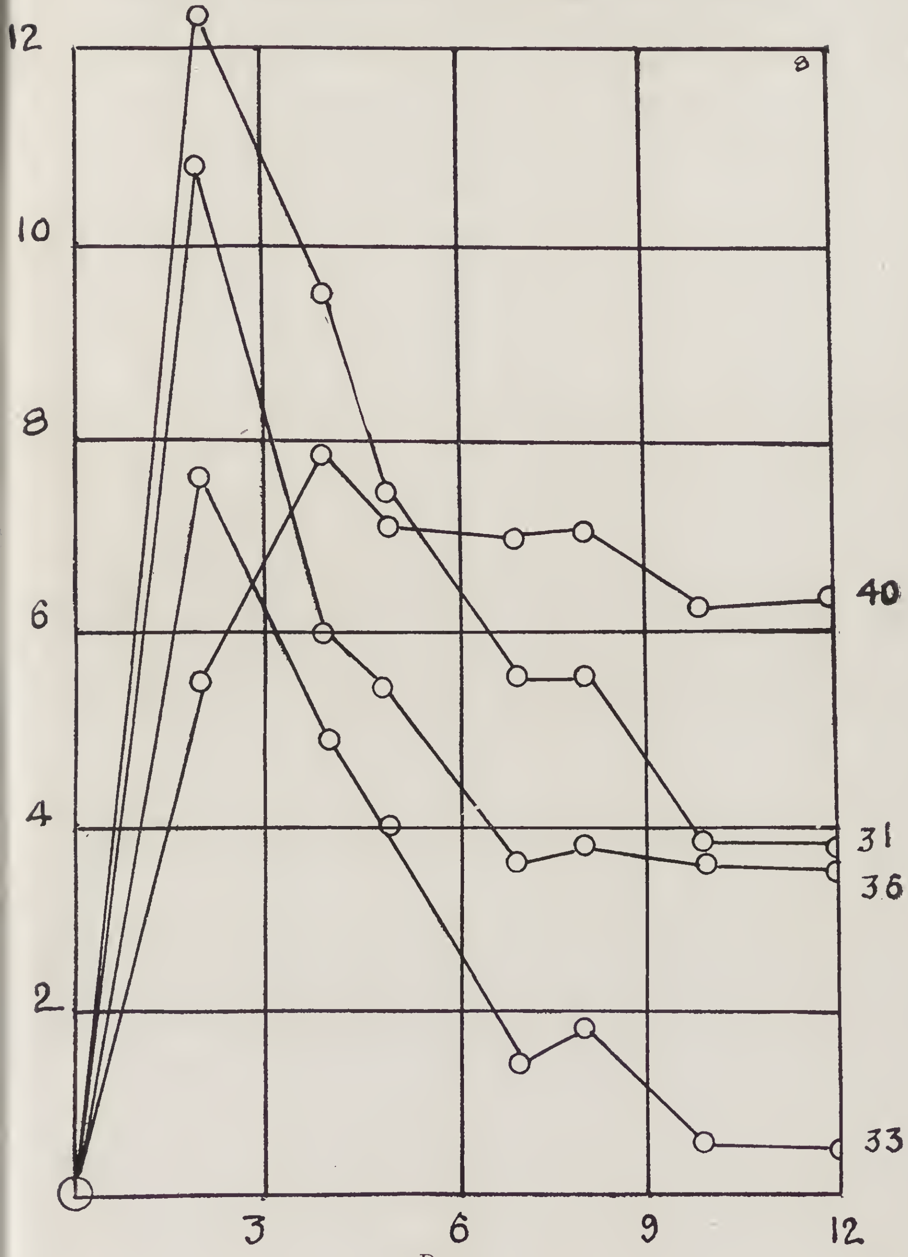


Fig. 5.

RESINATE DRIERS (Moist Cabinet).
No. 29—Aluminum Resinate.
No. 4—Lead Resinate.
No. 22—Chromium Resinate.
No. 17—Iron Resinate.



Days.

Fig. 6.

RESINATE DRIERS (Dry Cabinet).
No. 40—Aluminum Resinate.
No. 31—Lead Resinate.
No. 36—Chromium Resinate.
No. 33—Iron Resinate.

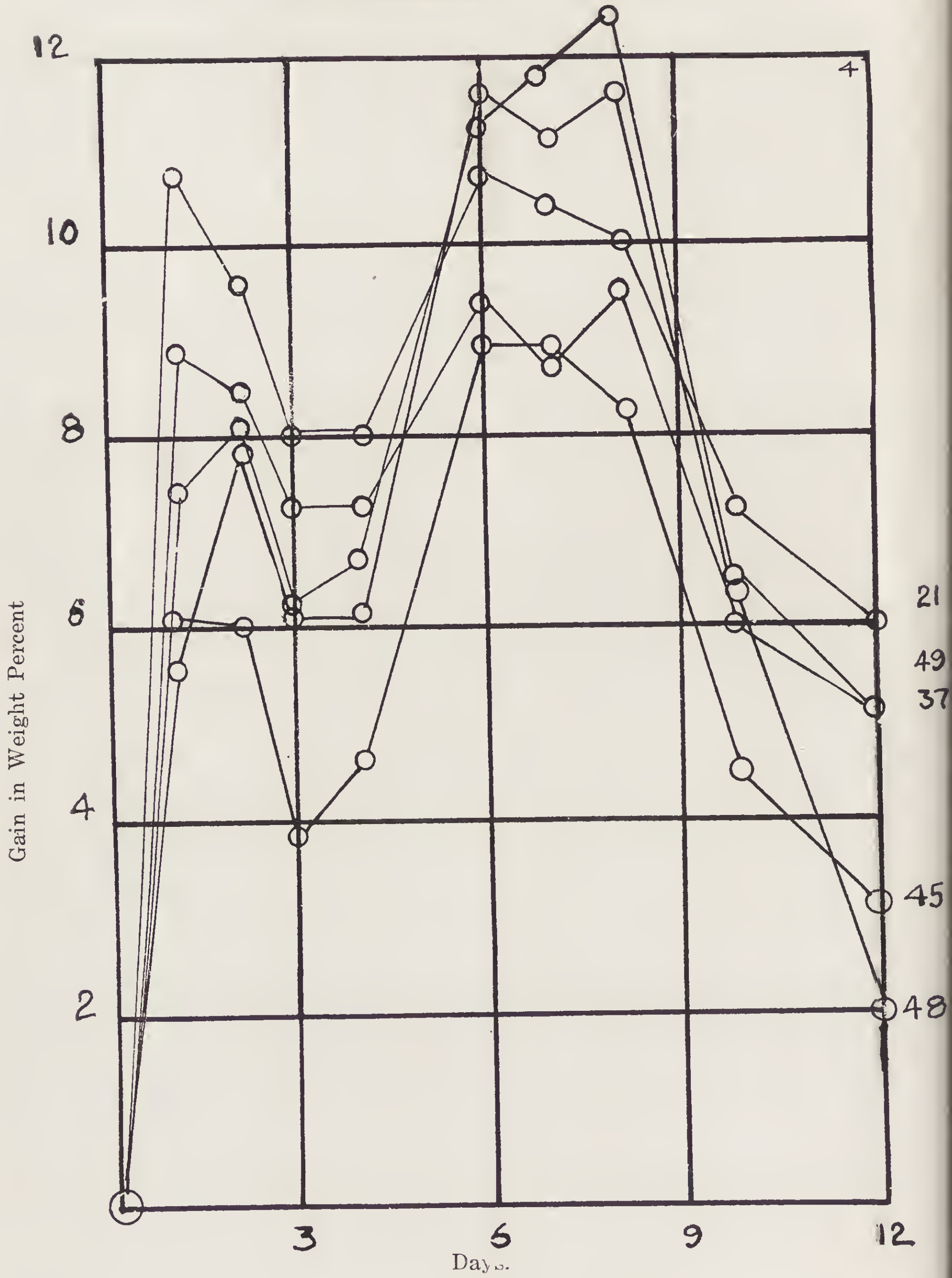


Fig. 7.

TUNGATE DRIERS (Moist Cabinet).
No. 21—Lead Tung Oleate.
No. 49—Aluminum Tung Oleate.
No. 37—Lead Menhadenate.
No. 45—Manganese Tung Oleate.
No. 48—Lead Tungate.

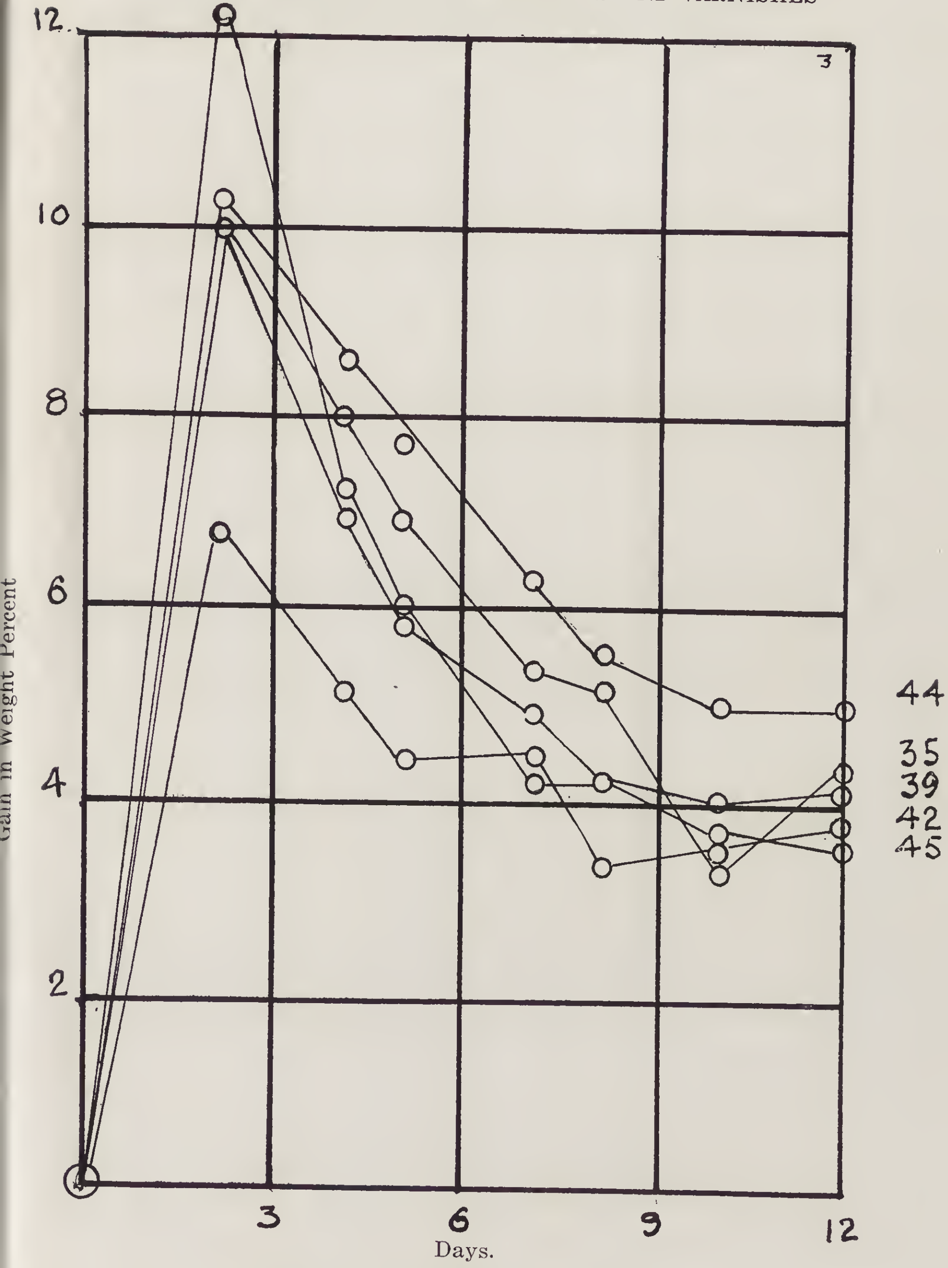
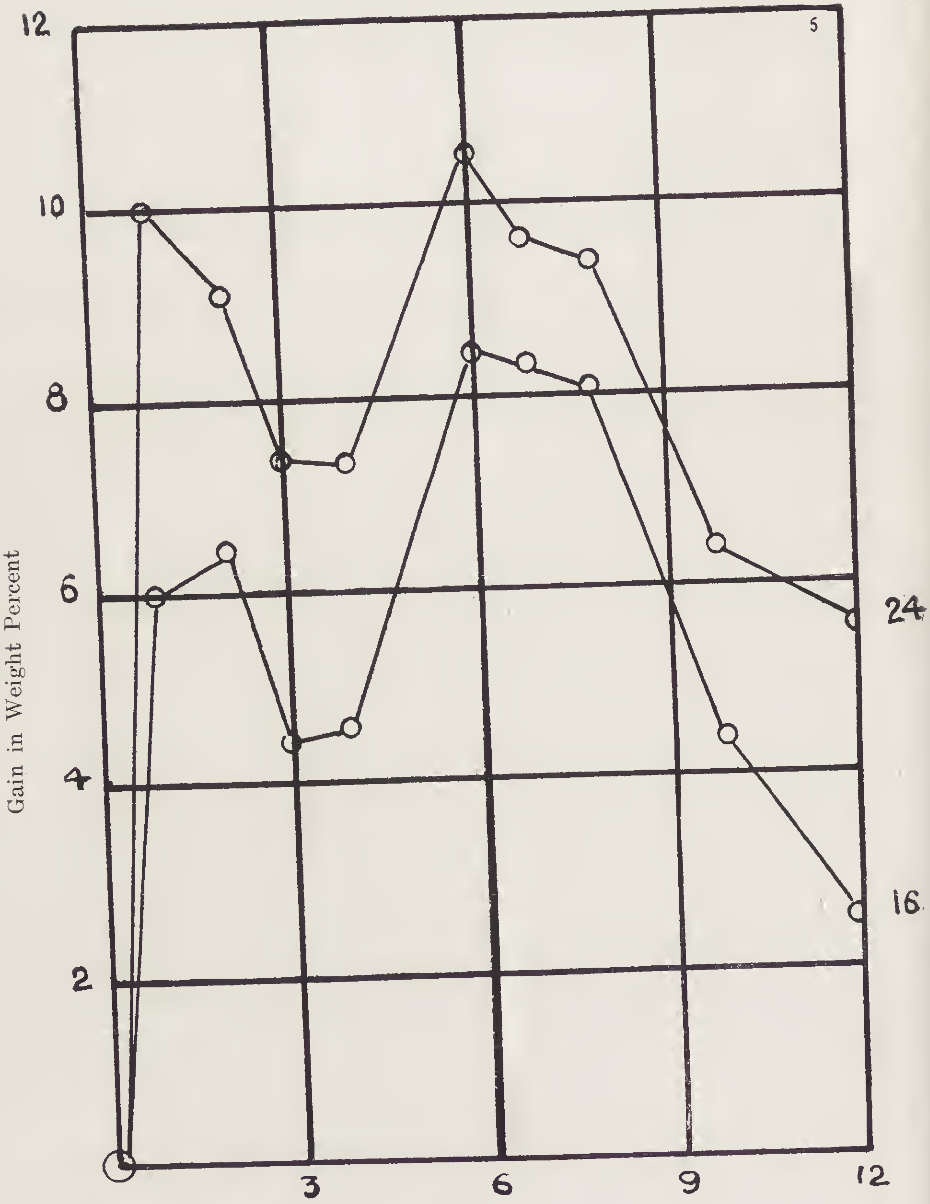


Fig. 8.

TUNGATE DRIERS (Dry Cabinet).
No. 44—Lead Tungate.
No. 35—Lead Tung Oleate.
No. 39—Lead Menhadenate.
No. 42—Manganese Tung Oleate.
No. 45—Aluminum Tung Oleate.



Days.
Fig. 9.

BORATE DRIERS (Moist Cabinet).
No. 24—Lead Borate.
No. 16—Manganese Borate.

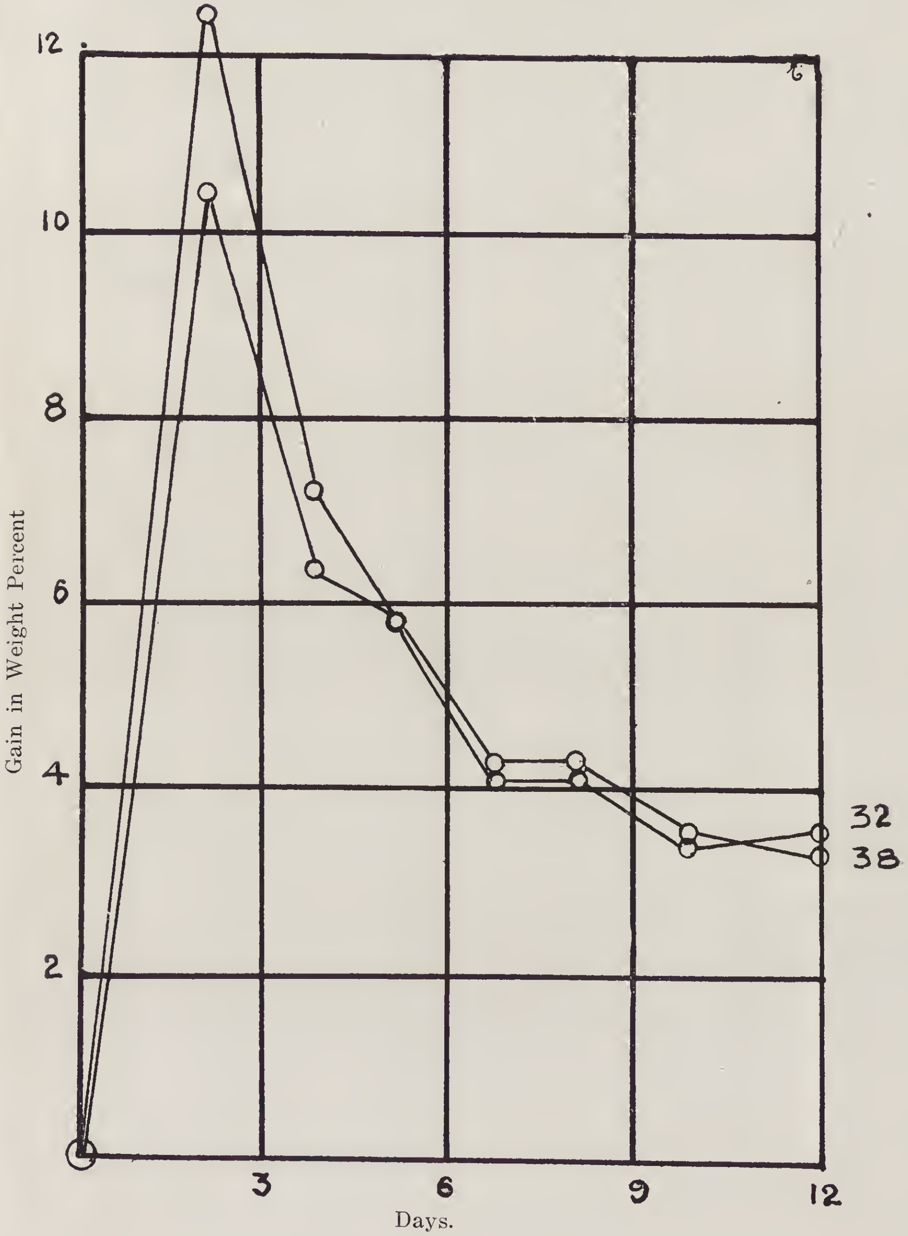


Fig. 10.

BORATE DRIERS (Dry Cabinet).
No. 32—Manganese Borate.
No. 38—Lead Borate.

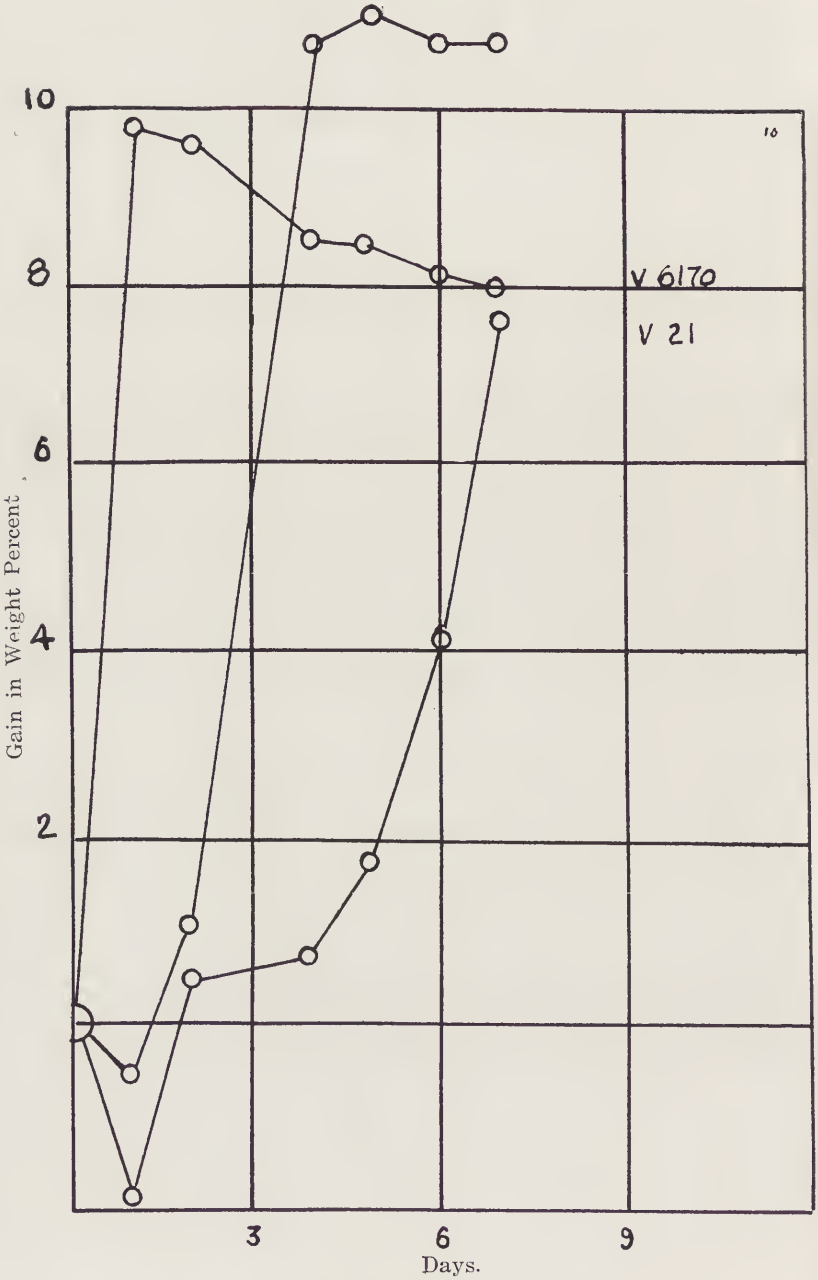


Fig. 11.
VARNISHES (Moist Cabinet).

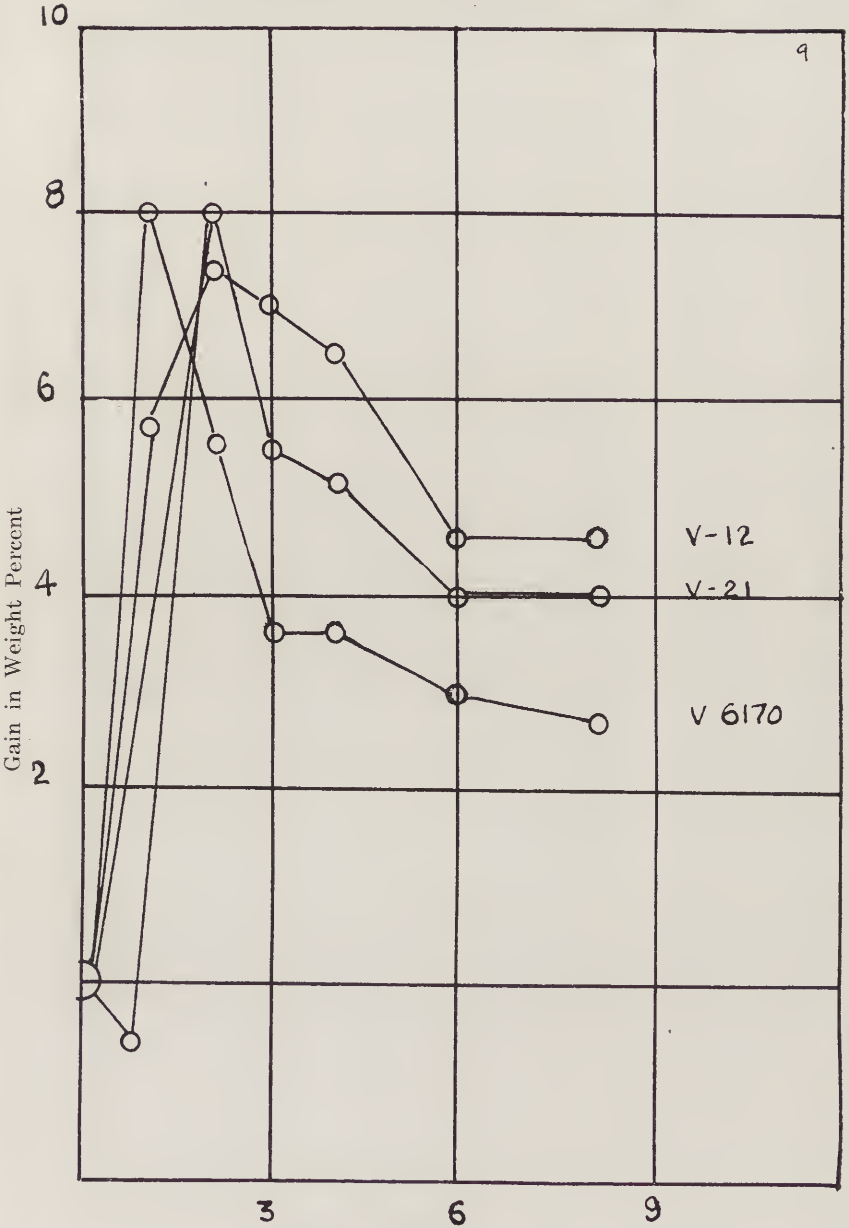


Fig. 12.

VARNISHES (Dry Cabinet).

The oil films secured in this manner were extremely thin, and it is probable that all of the oil employed was subjected to whatever reactions and changes may be involved in the process of drying. Had the films been thicker, this would probably not have been the case, as the hardening of the surface would have protected the underlying layers at least for a considerable time. At the conclusion of the tests all of the films were found to be thoroughly dry and exhibited resistance to scraping with a knife blade.

A consideration of the results obtained shows that in every case there was a very great increase in weight during the first twenty-four or forty-eight hours. The films dried in moist air almost invariably showed a gain in twenty-four hours that was equal to that shown by the same films in dry air in forty-eight hours. This gain was followed by a decrease, in some cases amounting to more than the original gain. Still later, in the majority of cases, the weight increased again and a second maximum was reached before the oils settled down to constant weight. The formation of a second maximum is particularly noticeable in those cases in which drying occurred in a humid atmosphere, it being often greater than the first maximum. In dry air the second rise is usually slight and sometimes entirely lacking. Evidently there is a great deal going on besides a simple absorption of oxygen. In fact, it has been shown by previous experiments* that during the drying of oil there is a large evolution of carbon dioxide, carbon monoxide, water and various organic substances. It seems fair to suppose that the absorption of oxygen continues throughout the whole process of drying, but that during the process certain highly perishable compounds are formed. While these compounds are decomposing, the amount of vapors evolved are much greater than the weight of the oxygen that is taken up. Furthermore, the

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*Paint Researches and Their Practical Application, pp. 218-230.

present experiments seem to show that the final hardening is arrived at more quickly and directly in dry air than in an atmosphere saturated with moisture. The very marked second rise that occurred in the moist cabinet is a very interesting feature. It is lacking in all those experiments conducted in a dry atmosphere. The fact that it appears in a rudimentary form in some of our dry cabinet experiments is probably due to the fact that these experiments were made during the summer and at a time when there was some humidity in the air. As no extreme precautions were taken to desiccate the air entering the cabinet, it was of course not perfectly dry.

There is every reason to believe that in the drying of linseed oil a rather complex series of chemical reactions take place. It is evident that when a certain stage has been reached, beginning at about the sixth day, the oil is in a condition to take up water if it be present in the surrounding atmosphere. No water seems to be taken up during the initial stages of the drying process, as the increase in weight is no greater, although more rapid, during the first few days in the moist cabinet than it is in the dry cabinet. Therefore, partial oxidation must be necessary to put the oil in a condition to absorb water. Furthermore, it is practically certain that the water taken up at this secondary period is chemically combined, as it is not given up when a plate is taken from the moist cabinet and kept for several consecutive hours in dry air.

It is quite generally believed at the present time that at some stage during the drying of oil the fatty acids separate from the glycerine with which they are combined. If this is true, it is evident that the glycerine would require three hydroxyl groups and each fatty acid molecule would need one hydrogen atom in order to establish themselves as separate entities. Accordingly, each molecule of tri-glyceride would take up three molecules of water on separating into its component parts.

$$C_3H_5(RO)_3 + 3H_2O = C_3H_5(OH)_3 + 3(ROH)$$

In this equation R represents the acid radical of whatever fatty acid may be present.

If all of the fatty acid present were linolenic acid the increase in weight due to the above reaction would be almost exactly 6.2%. In the case of fatty acids of lower molecular weight the increase would be proportionately greater. The above reaction would then account for the second rise in the curve. It is known that formic and acetic acids, aldehydes and perhaps other volatile compounds are formed and given off and these can hardly come from any other source than the free glycerine. This gives a clue to the nature of the second fall in the curves. It may be supposed that in the moist cabinet the drying of linseed oil proceeds somewhat after this fashion. First an absorption of oxygen by the unsaturated fatty acids causing a rise in the curve; then an evolution of carbonic acid and perhaps other products which causes the curve to fall. When this has gone on for a certain time the fatty acids lose their hold on the glycerine and the consequent taking up of water causes the second rise. Finally the decomposition of the glycerine into volatile products causes the second fall.

In the dry cabinet it may be assumed that the liberation and decomposition of the glycerine take place simultaneously or at least more nearly so. Certainly any volatile products which are formed would be dissipated more rapidly in dry air. As the gain and loss take place at the same time they neutralize each other and the second increase in weight is therefore lacking. In both the moist and dry cabinet the end product is probably the same: an oxidized and altered form of the fatty acids. However, it should be pointed out that oil dried in air saturated with moisture produces a film that is less likely to be as durable as that produced in dry air or air with a moderate moisture content.

In all of this intricate series of changes it is supposed that the drier—lead, cobalt, manganese, or whatever it may be—acts as a catalyzer, promoting chemical action, yet remaining unchanged itself. A surprising feature of the results is the very high apparent efficiency of aluminum compounds as driers. Although slow in initial effect, they seem to have even greater drying power than the corresponding compounds of lead. Further investigation of aluminum salts as driers would be indicated.

The results with linoleate, resinate, tungate and tung oleate do not show sufficient difference to indicate superiority of any class. As a rule, however, the linoleates are preferred for use in paints. As a matter of fact, an accurate relation between the oxygen absorption and the firmness of a film does not always exist. For this reason the efficiency of driers is compared much better through the use of the practical methods outlined in Chapter II. For the purpose of the investigation contained in this present circular determination of the increase in weight of each sample was necessary.

CHAPTER XI

Fume Loss in Boiling Oils

When oils are heated, they give off vapors and substantial losses are shown; the loss increasing with temperature and duration of processing. Some experiments have just been conducted, which show the fume loss of six oils when heated at varying temperatures for different periods of time. In the table presented below the gravity of the original oil, as well as of the processed oil, is given. The table also shows the weight loss as well as the volume loss. The tests were conducted in quart size open metal pans. The surface area exposed, to some extent would represent that shown by some types of oil boiling kettles. It is possible, however, that in factory practice, lower losses would result on account of the blanketing effect of foaming and smaller radiation areas.

In only one instance were irregular results shown. This was with sunflower-seed oil, which developed a higher loss on heating at 550° for 3 hours than for 4 hours. These results were shown on repeat determinations, but will not be reported until further tests are made. It is probable, however, that in the case of this oil the longer period of heating caused sufficient oxygen absorption to account for the difference.

The oils resulting from the above experiments have been preserved in large well filled and stoppered containers. They will be used to determine the changes in constants and viscosity.

FUME LOSSES IN BOILING OILS

TABLE 45.

	Specific Gravity	Percentage Weight Loss	Percentage Volume Loss
RAW LINSEED OIL	.9325		
1. 500°F for 2 hours	.9408	2.0 %	2.86%
2. 500°F for 3 hours	.9446	2.7 %	3.94%
3. 500°F for 4 hours	.9477	2.9 %	4.46%
4. 550°F for 2 hours	.9508	3.9 %	5.75%
5. 550°F for 3 hours	.9610	5.9 %	8.69%
6. 550°F for 4 hours	.9690	8.6 %	12.05%
VARNISH MAKERS LINSEED OIL	.9320		
1. 500°F for 2 hours	.9405	1.2 %	2.10%
2. 500°F for 3 hours	.9446	1.9 %	3.22%
3. 500°F for 4 hours	.9482	2.2 %	3.88%
4. 550°F for 2 hours	.9502	2.7 %	4.57%
5. 550°F for 3 hours	.9608	4.6 %	7.47%
6. 550°F for 4 hours	.9666	5.4 %	8.79%
FISH OIL (Menhaden)	.9335		
1. 500°F for 2 hours	.9561	3.73 %	6.00%
2. 500°F for 3 hours	.9630	4.63 %	7.55%
3. 500°F for 4 hours	.9671	5.16 %	8.45%
4. 550°F for 2 hours	.9725	6.78 %	10.51%
5. 550°F for 3 hours	.9770	8.04 %	12.14%
6. 550°F for 4 hours	.9800	9.16 %	13.47%

TABLE 45 (Continued).

	Specific Gravity	Percentage Weight Loss	Percentage Volume Loss
SOY BEAN OIL	.9265		
1. 500°F for 2 hours	.9312	2.54%	3.03%
2. 500°F for 3 hours	.9375	4.16%	5.28%
3. 500°F for 4 hours	.9391	4.52%	5.80%
4. 550°F for 2 hours	.9398	5.74%	7.07%
5. 550°F for 3 hours	.9438	6.76%	8.47%
6. 550°F for 4 hours	.9498	8.57%	10.81%
ORIENTAL PERILLA OIL	.9360		
1. 500°F for 2 hours	.9475	3.72%	4.90%
2. 500°F for 3 hours	.9502	3.92%	5.35%
3. 500°F for 4 hours	.9560	4.79%	6.79%
4. 550°F for 2 hours	.9592	6.18%	8.45%
5. 550°F for 3 hours	.9720	7.54%	10.97%
6. 550°F for 4 hours	.9800	8.73%	12.83%
SUNFLOWER SEED OIL	.9240		
1. 500°F for 2 hours	.9307	3.78%	4.48%
2. 500°F for 3 hours	.9321	4.69%	4.53%
3. 500°F for 4 hours	.9345	4.04%	5.12%
4. 550°F for 2 hours	.9370	8.92%	10.19%
5. 550°F for 3 hours	.9400	..........*	..........*
6. 550°F for 4 hours	.9430	9.33%	11.16%

*Higher results obtained than for four-hour period.

CHAPTER XII

Fume Control in the Varnish Industry

By-Product and Solvent Recovery

Industrial economies often result from the influence of external conditions. For instance, the aromatic pungency of atmosphere that evidences the presence of an oil or gum using industry has been responsible for attempts, more or less successful, to "bottle the smell." The resulting elimination of objectionable fumes or odors has often been accompanied by recovery of useful products.

Industrial fumes may be of the uneconomic types that are condensible through the installation of apparatus and processes that do not yield products of sufficient value to pay interest charges on the installation and the cost of operation. The fumes from smelters and acid factories might in some instances be of this type. Again, industrial fumes may be of the economic type, that may be abated with an accompanying yield of by-products of considerable value. It is believed that the fumes from the varnish industry are often of the latter kind.

As is well known, there are serious fume losses in the gums (resins) that are used in the manufacture of varnish. The amount of loss varies from moderate to heavy percentages, depending upon the nature of the gums and the temperature and duration of the cooking process. Unfortunately, very little data as to the amount of gum loss has been accurately determined or at least made available by disclosure of general information to the industry. Extensive laboratory and factory investigations are, however, under way to accumulate such data. Similar but comparatively lower losses are shown in the heat treatment of drying oils. Some tests just conducted by the writer presented losses of approximately 2 to 9% on a few commonly used oils when heated to several different temperatures that are used in factory practice. (See Chapter XI.)

The data on varnish fume loss that is available is usually on losses made on blended gums and determined over a period of time under general factory conditions for a variety of products, or some given type of varnish. The usual method of determining the disappearance or loss of weight is by difference in the weight of materials used and products obtained. The data thus secured is, of course, subject to error and may often mask heavy losses on expensive gums. Moreover, there may be gains in weight through chemical reactions such as oxidation, to offset and conceal the total weight of fume loss. For instance, a very serious fume loss on a high-priced gum may be greatly obscured by the undetermined but relatively small loss of weight of other components of the formula, such as oil and rosin.* In a carefully conducted test made in the writer's laboratory, rosin of H grade was treated with 6% of lime at 350° F., then brought up to 525° F. and held there for 15 minutes. The fume loss was 6.8% of the rosin used. The mere fact, however, that the expensive hard fossil gums used in varnishes may in ordinary practice lose from 10% to 40% of their weight as a result of the heat treatment necessary to render them soluble in oil, is enough to indicate to the varnish manufacturer the gravity of the situation.

In order to emphasize how much money loss in gum fume may be obscured through determining the over-all

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*Rosin shows a comparatively low fume loss as compared to resins requiring a high temperature for fusion and oil solution. The following example has, for instance, been furnished by a varnish maker as an illustration: If 500 pounds of rosin are treated with 30 lbs. of lime, a total batch of 530 lbs. of material results, which, after boiling, yields only 495 lbs. of limed rosin. The actual rosin being eliminated as fume would therefore amount to 35 lbs. or 7% of the amount originally used. With 35 lbs. of rosin given off as fume, an average of 21 lbs. may be recovered as a primary condensate by surface condensation in flues. Other varnish manufacturers have furnished data indicating that good practice shows a fume loss on rosin of about 8% for grades F, G and H; about 9% for grades M and N; and about 10% for grades WW and WG.

loss or difference between total of materials used and product obtained, a table is appended of losses as actually determined and furnished for this report by one well-known varnish maker of high standing. The type formulas given in the appended table are selected to cover a wide range of varnishes and to emphasize the fact that the over-all figure of loss conceals very large losses of gum as fume. From the appended formula and factory practice it will be noted that the over-all loss runs from 12% to 20% but that the actual gum loss may run from 30% to 42% of the amount used in the varnish. These figures are all on losses shown by the varnsh bases previous to thinning with turpentine or other thinners.

TABLE 46.
Gum Loss or Disappearance in Kettle

	Over-all loss by weight	Loss of gum used by weight
Long Oil Kauri Varnish	12.4%	30%
Short Oil Kauri Varnish	20.0%	34%
Long Oil M Varnish*	16.0	40
Short Oil M Varnish	20.0	42

The proportions contributed to the fume loss by the oil and by the gum are indicated by the following data.

TABLE 47.
Showing the Proportion by Weight of Oil and of Gum in the Fume Losses

	Oil	Gum
Long Oil Kauri Varnish	33 1/3%	66 2/3%
Short Oil Kauri Varnish	11.5%	88.5%
Long Oil M Varnish	27.3	72.7
Short Oil M Varnish	12.5	87.5

Of these losses it is shown that the gum loss is greater in weight. The monetary value of the gum losses would show even more startling figures.

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*Based largely upon Manila.

TABLE 48.

Showing the Proportion by Money Value of Oil and of Gum in the Fume Losses

	Oil	Gum
Long Oil Kauri Varnish	15%	85%
Short Oil Kauri Varnish	5.4%	94.6%
Long Oil M Varnish	24.7	75.3
Short Oil M Varnish	11.	89.

Having thus indicated the percentage of "over-all" loss, the higher percentage of gum loss, the proportions of gum and oil in the fume loss, and the comparative economic values thereof, the next question of importance is the specific money loss per kettle batch.

A varnish factory averages on treated oil 2 batches per day and on untreated gums 1½ batches per day. A yearly average of 250 days is a conservative allowance for activity. On such a basis an average output per kettle would be about 400 batches. The equivalent of a varnish kettle running continuously on one of these type varnishes would thus indicate a yearly fume loss on gum and oil equivalent in money to the cost of the products lost. It should, however, be pointed out that even entire recovery of fumes would not necessarily mean recovery of the original value of the products from which they were derived. This is due to the fact that oil and gum fumes recovered as condensates by any system are of different composition than the materials of their origin and of much less money value. Nevertheless the above charts show the importance of fume recovery, and it is quite possible that certain products might be fractionated from condensed gum fumes, that would have an even greater money value than the original gums.

The recovery of such portions of turpentine or other liquids used as thinners, as may become volatile during the thinning process, is a matter of growing importance. Some varnish factories have for several years made more or less successful attempts at such practice, through the installation of draft hoods and simple condensers conveniently placed in the thinning room. Installation of suitable apparatus for this purpose will quickly pay for itself in a few months' time. The condensed thinners may be obtained in excellent yield and in good condition for reuse.

Historical Data on Fume Recovery.—Systems for at least partial recovery of gum and oil fumes from varnish plants have been installed and experimented with from time to time over a great many years and as early as a century ago. These methods, such as Tingary's, Andre's, Hackathorn's, Lehmann's, Neil's, Koerting's, and others, are described in well-known books on varnish technology. Through their use considerable quantities of copal oil (condensible fume from varnish resins) have been obtained. These have been used not only as a solvent for resins but as a constituent of certain types of varnishes and paints. Some of these systems depended upon the condensation of the fumes carried through sheet metal flues. A considerable portion of the fume that was not condensible and which was of the most objectionable odor may have been passed through chambers containing spray jets of water. This system was not always sufficient to remove the objectionable or odoriferous portions which consequently passed out into the air.

The writer has made a search of available Patent Office records on fume recovery and is presenting herewith a brief abstract of the result.

U. S. Patent 222,983 of December, 1879, to D. D. Cattanach; IMPROVEMENT IN TREATING LINSEED AND KINDRED OILS. During the thinning of the cooked oil, the solvent in the form of vapor is conducted away by a pipe in the top of the hood and may be condensed in a coil so as to be used repeatedly without material loss.

U. S. Patent 286,528 of October, 1883, to Bernhard Borowsky, entitled "APPARATUS FOR THE MANUFACTURE OF VARNISH." In the method of cooking varnish described, the vapor of the turpentine or other solvent is carried by a strong draft of air into a condenser where the lighter portion of the vapor condenses.

U. S. Patent 312,854 of February, 1885, to D. R. Hostetter, entitled "COVER FOR VARNISH KETTLES." In this is described a cover for a varnish kettle, having attached thereto a coil pipe provided with means for air or water cooling, so that the vapors from the varnish will be condensed and returned to the kettle.

U. S. Patent 318,642 of May 1885, to S. W. Mayer and R. J. Bungart, entitled "APPARATUS FOR THE MANUFACTURE OF VARNISH." In this is described special varnish kettles with furnace beneath such kettles, suction pipes connected to the discharge pipes of the kettles, trunk pipes connected to the suction pipes, a suction fan connected to the suction pipe, a condenser, and distributing pipes leading to the condenser from the varnish kettles, etc.

U. S. Patent 663,594 of December, 1900, to R. A. Worstall and C. T. Hackathorn, entitled PROCESS OF MAKING VARNISH. In this is described a process of manufacturing varnish, which consists in subjecting the gum or resin to the action of heat, condensing the vapors of the gum, formed during the process of heating, and subsequently using the distillate obtained thereby.

U. S. Patent 708,935 of September, 1902, to E. H. Strange and Edward Graham, entitled "MANUFACTURE OF VARNISHES." In this, provision is made for recovery by condensation, of the turpentine used for thinning purposes.

U. S. Patent 711,596 of October, 1902, to A. L. Tedesco, entitled PROCESS OF MANUFACTURING VARNISHES. In this patent is described the depolymerization of copal resins by heat, the volatilized terabenthines being condensed in a special apparatus and returned to the varnish mass.

U. S. Patent 1,011,659 of December, 1911, to E. H. Strange, entitled "MANUFACTURE OF VARNISHES." In this is described a process of manufacturing varnishes, involving a special type of apparatus, which consists in dissolving copal by heating the latter under pressure in the presence of a relatively small amount of oil distillate, simultaneously distilling off the solvent and thereafter adding the requisite quantity of turpentine.

U. S. Patent 1,027,563 of May, 1912, to George W. Priest, entitled "THE MANUFACTURE OF VARNISH FOR COATING LEATHER." In this is described special types of apparatus, consisting of a portable kettle and stationary dome, specially designed stirring devices and condensation equipment.

U. S. Patent 971,071 of September, 1910, to G. W. Priest, entitled "MANUFACTURE OF VARNISH FOR COATING LEATHER." In this special means are provided for condensing the vaporized solvent and returning it to the dome of the apparatus.

U. S. Patent 1,066,794 of July, 1913, to Howard Cassard, entitled "PROCESS OF REFINING SHELLAC." In this is described a condenser attached to the solution kettle, for recovery of solvent used.

U. S. Patent 1,298,159 of March, 1919, to C. H. Bennett, entitled "APPARATUS FOR MANUFACTURING VARNISH." In this a special type of apparatus for thinning varnishes is described, with suitable equipment for recovery of vaporized fume material.

U. S. Patent 1,329,076 of January, 1920, to E. H. Frohner, entitled "METHOD OF AND APPARATUS FOR TREATING OIL AND OBTAINING BYPRODUCTS THEREFROM." This describes a method of passing the gases arising from the blowing of fatty oils, directly through solvents to recover the oils contained in said gases, and then passing the gases through an alkaline solution to remove the acrolein.

U. S. Patent 883,842 of April, 1908, to Henri Terisse entitled "TREATMENT OF HARD AND SEMI-HARD COPALS AND THE PREPARATION OF VARNISHES. Describes process of heating oil, resin and naphthalene; boiling to remove naphthalene and then adding thinner. See description below of Cruickshank-Smith article.

Probably the most interesting paper presented to date on the subject of varnish manufacture with fume elimination is that by Cruickshank Smith and Gaston de Pierres, entitled "The Technology of Varnish Manufacture, with Notes on an Improved Process." This paper, which was presented as an original communication to the Eighth International Congress of Applied Chemistry, describes work based on the original experiments carried out nearly seventy years ago by Schutzenberger, who heated mixtures of copal, linseed oil, and turpentine under pressure in a closed vessel. The authors point out that the gums used in varnish manufacture are polymerized terpenes and that what the varnish maker attempts to accomplish by heating, is to depolymerize the terpenes so that they may become soluble in oil. In order that this may be accomplished without fume loss of any type, they recommend the use of an autoclave pressure-temperature system. The following extracts from their article present this subject clearly:

> "If we consider the fusion of resin as it is effected in the ordinary process of 'running' in the gum-pot, it may be regarded as essentially a process involving partial dry distillation of polymerized terpenes. It may be likened to dry distillation of wood or coal with the differences that in the case of the resin the distillation is arrested at a definite point and that the valuable product is contained, not in the distillate (as in the case of wood or oil), but in the residue which is left in the gum-pot. As in the distillation of wood or coal the vaporized portion contains carbon dioxide and ethylic compounds while the distillate contains acetic and pyroligneous acids, naphthalene, hydrobenzene and, in short, that whole range of compounds derived from the polyterpenes to which we apply the name 'copal oil' or 'varnish fumes.' It must not be imagined that the acids which are found in the copal oil exist as such in the original resin, any more than acetic or oxalic acid is contained as such in wood or cellulose. The lower acids named are decomposition products, in the one case of cellulose and in the other of the polymerized terpenes. Nevertheless in the conventional method of varnish making this fusion and incipient distillation process could not be avoided because the resin had to be rendered wholly soluble not only in spirits of

> turpentine but also in linseed oil, and this result could only be achieved (under the conditions which surround the conventional process) by subjecting the resin to strong direct heat. The practical result aimed at in subjecting a hard resin to heat is to depolymerize it, that is, to convert it into bodies of simpler constitution and lower molecular condensation than those possessed by the higher members of the polyterpene series. Thus the polymerized terpenes of high molecular weight which may be represented by the general formula $(C_{10}H_{16})$ are in effect polymers of dipentine $C_{10}H_{16}$ which again is a polymer of pentine (isoprene) C_5H_8, the first members of the series."

Smith and de Pierres further point out that hard resins are capable of being de-polymerized with perfect ease under pressure, and they illustrate the value of having present an intermediate substance with a low fusion point to act as a solvent. For this purpose they have experimented successfully with naphthalene* which subsequently may be removed from the manufactured varnish by distillation. They comment upon the ease of fusion and solution by this process, the absence of fume, and condensate (copal oil). They sum up their paper with the following statement which should stimulate the varnish chemist to further efforts in similar directions:

> "We claim that there is abundant evidence at hand to warrant the dissipation of the fiction that the historic conventional method of gum-running is a necessary or even a defensible process inasmuch as the 'acid' which the old fashioned method strove to separate was to a large extent produced by the incipient destructive distillation of the resin itself. We claim also that the depolymerization (on which the solubility of the fused resin depends) can be brought about by a method which is more gentle in its effects, more under scientific control and capable of producing better and cheaper varnishes than the method which has been the

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*See U. S. Patent 883,842 of April, 1908, to Henri Terisse.

> fetish of the mystery-maker and secret-monger for upwards of two hundred years. In conclusion we should like to point out that in spite of the apparent simplicity of the process and of the principles on which it depends it must be controlled with skill and nicety in order that satisfactory and uniform results may be obtained. The merely theoretical worker who attempts to make good varnish either by this or by any other process will find that he has a long and costly novitiate before him. While it will be granted that the modern varnish factory has little use for the hide-bound rule-of-thumb, so-called practical man, it is equally true that to the dough of organized science must be added the leaven of practical skill—that indefinable turn of the wrist which will always distinguish the clever cook, the good varnish maker, and the successful technical expert."

No data is apparently available regarding the commercial use of the autoclave system described above and as to whether it is now on a successful basis. It is believed, however, that Smith's process has been used successfully in England. The writer has been informed of certain unsuccessful attempts in the United States to make varnish under similar procedure. These early failures may, however, point the way toward later successes. However, in some European as well as American varnish factories there still exist types of apparatus that were used for early experiments on fume control. The general plan of the earlier attempts was to draw the fumes through fume hoods and thence through a system of condenser flue pipes by the aid of a fan or suction blower. The waste gases from the coke fires, as usual, continued to pass up the heavy varnish stacks, although in one instance the expensive stack was omitted and a metal stack of proper size and moderate cost used. These installations represented separate individual efforts or experiments to recover fume products or to absorb repulsive fume odors. In many of them, non-movable, non-adjustable hoods were located above the kettle so that a considerable open space existed between the varnish kettle and the bottom of the hood. Great volumes of air necessarily were admixed with the fume, and at least partial oxidation of the fume resulted. Outlets were provided for discharging the condensate and cleaning out the fume deposits from the condenser pipes. The remainder or uncondensed portion of the fumes were carried along by the fan and discharged into the air. These earlier installations disclosed certain important facts and defined the problems to be overcome in order to obtain successful fume and odor recovery control. Some of these systems also disclosed the important fact that a large amount of the fume produced in varnish making will not liquify at the temperatures resulting from the heat conductivity of the metal walls of the pipe flues. Notably those fractions of the fumes which are characterized by repulsive odor do not to any marked extent liquify or condense.

The unlimited admission of air between the kettle and the metal hood will maintain and make even more pronounced the danger of fire flash through the presence of oxygen. In the heavy stack type of plant, for instance, where fume control is not in operation the construction of the building is designed with reference to the dangers from fire flash at the kettle and the loss through fire of the goods in process. This is evidenced in the isolated location of the boiling or kettle house; the heavy construction of the stack to permit burning out of fume accumulations, and in the building, which is either of very heavy, non-burning construction or of a very cheaply replaceable form of construction. As illustrative of the above, the following instructions are found in a text book on varnish manufacture by a prominent European technologist:

> "Every person intending to manufacture varnish on a profitable scale ought to procure suitable premises some distance out of town and sufficiently large for the business intended. The building or shed wherein varnish is made ought to be quite detached from other buildings, in order to avoid danger from fire."

A full discussion of the fire hazards in varnish plants, together with some suggestions for their elimination, are given further on in this article.

Recent Developments in Fume Control. In the spring of 1917 the president of a large paint and varnish manufacturing company in Rhode Island often found that fumes from his plant were carried by the wind to a nearby hospital. He determined, therefore, for his own interest and for the public advantage to investigate the possibilities of suppressing or eliminating the odors from his varnish stacks. He put his problem before his technical staff which was headed by W. W. Rice. After considerable research work had indicated how the fumes might be removed, they placed their recommendations before a technical man formerly associated with the early scientific developments in American paint manufacture.

Fig. 13.

Fume Hood on Kettle (First Design)

This man and his associates had already been investigating the subject of industrial fumes, having applied certain scientific principles in the acid industry for the elimination of acid fumes. Their study of varnish fumes indicated to them that while important fractions of the gum and oil fume were condensible when cooled to moderate temperatures, those portions of the fumes with the most repugnant odors did not generally respond to surface condensation. Further investigations determined that these end product fumes would not wholly respond even to the scrubbing action in spray towers when passed through water or other liquids neutral to and not chemically reactive with the fumes. It was found, however, that the odorous end fractions of the fumes could be converted into solid or liquid materials and thus be entirely removed from the fume gases if brought in intimate contact with liquids holding certain very inexpensive reagents of an alkaline nature, as, for instance, milk of lime. As a result of these investigations the plan of action called for the unification of fumes from a battery of kettle fires, to be drawn through a manifold flue and then through a unified system for treatment and recovery of separate fume products. Such manifolding of the fumes necessitated special equipment for controlling the air which in many previous attempts had been admitted to the top of the kettle and passed with the fumes into the hood and through the flues, as well as preventive equipment designed to stop accidental fire flashes from communicating through the manifold. An experimental system was installed on the broad general principles that were suggested in the early development previously referred to. After several thousand kettle batches had been produced under the system with a record of only two kettle flashes, (which incidentally were instantly smothered) and with a record of complete odor suppression, additional improved equipment was installed.

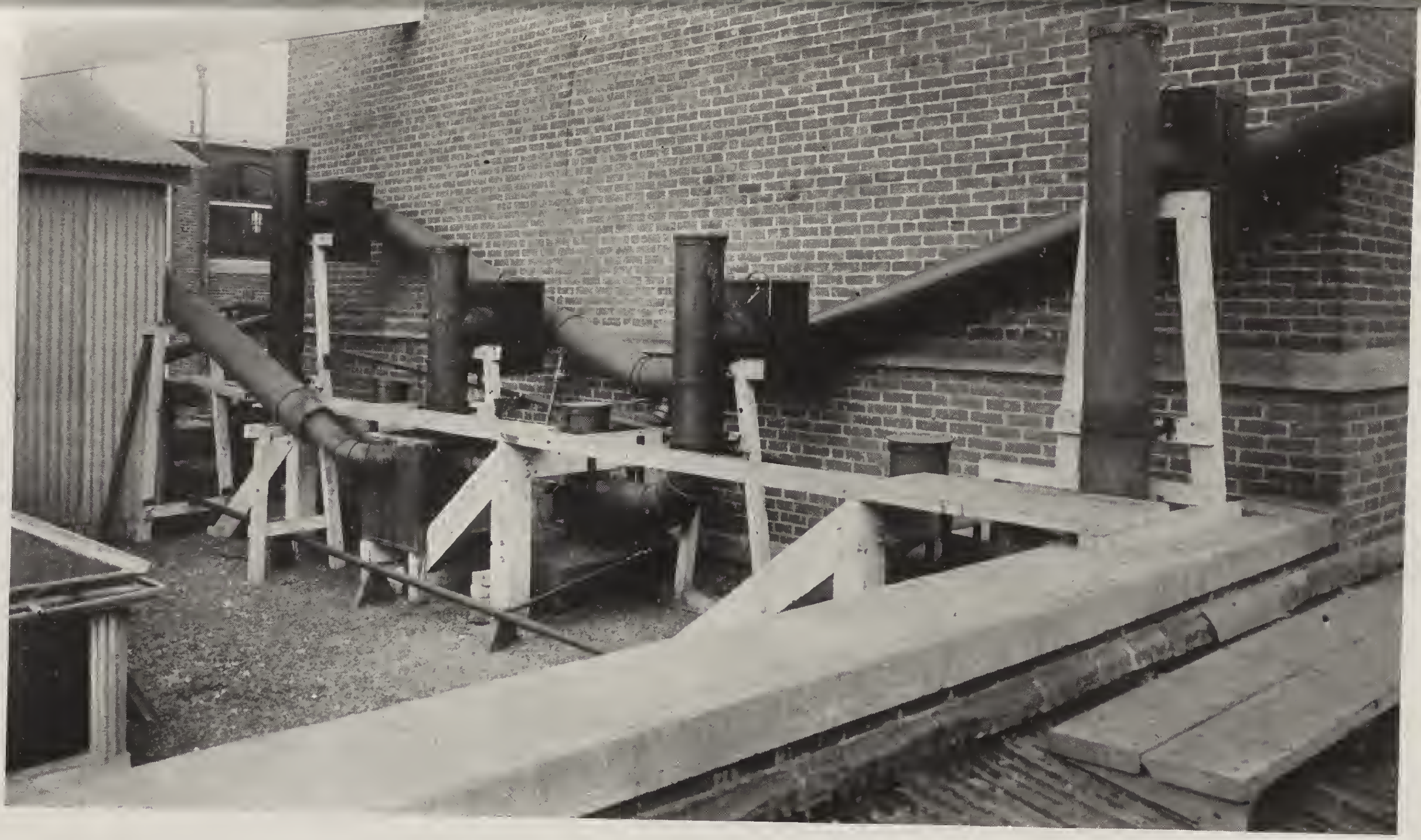

Fig. 14.
Original Installation of Perry Fume Collecting and Treatment System in Old Stack Plant.

As a result of factory practice upon the installation of this system, it was determined that three or more products could be fractionated from the fume. The primary condensate was recovered at atmospheric temperature and with a minimum of air dilution. This product probably represents the major portion of the copal oil or fume distillate from the gum content of the varnish.* An intermediate condensate, consisting of some of the gum fumes admixed with some of the not ordinarily condensible oil fumes, was obtained. An end product consisting of practically all of the fumes which were not fractionated out by other means, was obtained by absorption in alkaline materials.

The writer obtained three of the products recovered by the above-mentioned system and made a preliminary examination thereof. They represented the recovered fume from gum and oil only. The results of examination are given below.

TABLE 49.
Results of Preliminary Investigation of Fume Products

	Primary Gum Condensate	Secondary Oil Condensate	Final Absorption Product
Refractive Index	1.477	1.476	....
Specific Gravity	.958	.924	....
Iodine Number	141.	124.	67.
Saponification Number	80.	164.	76.
Acid Number	53.	134.	34.
Water Soluble Acids (Acetic, etc.)	1.4	1.4	....
Solubility in Water	slight	slight	slight
Solubility in Benzine	readily	readily	very slight
Solubility in Benzol	readily	readily	very slight
Solubility in Turpentine	readily	readily	very slight
Solubility in Alcohol	readily	readily	very slight
Drying on Glass	Tacky in 1 day Fairly dry in 3 days	Wet after 3 days	Tacky for 3 days Opaque character of film gradually lost.
Drying on Glass when reduced with:			
50% Benzol	Fairly dry in 1 day	Wet after 3 days	
50% Benzine	Fairly dry in 1 day	Wet after 3 days	
50% Turpentine	Fairly dry in 1 day	Wet after 3 days	
50% Alcohol	Fairly dry in 1 day	Wet after 3 days	

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*These recovery products are about equal to rosin gloss oil according to one producer.

TABLE 50.
Dry Distillation Results

PRIMARY GUM CONDENSATE:		SECONDARY OIL CONDENSATE:	
Water	1%	Water	1%
Distillate, 100 to 150°C	3%	Distillate, 100 to 150°C	2%
Distillate, 150 to 200°C	38%	Distillate, 150 to 200°C	3%
Distillate, 200 to 220°C	25%	Distillate, 200 to 300°C	12%
Distillate, 220 to 300°C	5%	Residue	82%
Residue	28%		
	100%		100%
Distillates have average Refractive Index 1.456.		Distillates have average Refractive Index 1.466.	
Distillates cloudy and odorous.		Distillates cloudy and odorous.	
Distillates have high solvent action on resins.		Distillates have high solvent action on resins.	
Distillates have high solvent action on rubber.		Distillates have solvent action on rubber.	
Distillates have high solvent action on oils.		Distillates have high solvent action on oils.	
Distillates miscible with turpentine, benzine, benzole and alcohol.		Distillates miscible with turpentine, benzine, benzole, and alcohol.	
Acid Number of Residue 24.		Acid Number of Residue 117.	
Residue is clear red brown in color, almost solid. Soluble in turpentine, benzine, benzol, but only partly soluble in alcohol, possibly due to polymerization during heating.		Residue is dark brown liquid. Soluble in turpentine, benzine, benzol and to large extent in alcohol.	
Residue dissolved in equal volume of benzine, tacky in two hours but not dry in 20 hours.		Residue dissolved in equal volume of benzine still wet after 24 hours.	
Same mixture + 10% drier, dries hard in eight hours.		Same mixture + 10% drier still wet after 24 hours.	
Residue on polymerization 20%.		Residue on polymerization 10%	

TABLE 51.
Steam Distillation of the Gum Condensate

100° to 180°C	30%	Clear and light amber colored liquid having a pleasant terpene odor.* Resembles oxidized turpentine.
180° to 330°C	45%	Clear liquid having greenish yellow color and not very objectionable odor. Viscous and somewhat resembles pine oil.
Residue	25%	Clear amber red mass almost solid. Resembles fused resins. Acid No. 20.

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*Most of this portion distilled between 160° to 175°C. It contained a small amount of an oil that was found to be "terebene," an antiseptic substance having medicinal properties when used for the treatment of bronchitis.

The above-described system, after operation for over two years, has offered opportunities for improvements and developments of a mechanical nature.

The present equipment consists of:

1. Kettle hoods.
2. Fume piping with telescopic and universal joints.
3. Fire control and safety equipment.
4. Collecting manifold.
5. Surface condenser for primary condensate.
6. Scrubbing condenser tank for intermediate condensate.
7. Blower and motor for exhaust and blowing of gases.*
8. Chemical reaction tank.

A brief description of the above items is given below.

Kettle Hoods: The kettle hoods are of several types and are designed to permit of the usual art and the unhampered operations of varnish manufacture such as stirring, ladeling, whipping, temperature control, etc., and are so designed that the fumes are readily removed, thus preventing blanketing and the consequent greater surface tension and temperature that would increase the danger of fire flash. The covers are raised and lowered over the kettles by means of telescoping and other suitable joints in the connecting piping, thus giving freedom for placing on or withdrawing the kettles from the fire. One type of hood is made to separate from the piping so that it may be easily attached to and detached from the kettle with a cone snout for control of the fume. It also furnishes a cover for the kettle during its progress to the cooling house and for use in the recovery of volatiles in the reducing or thinning room.

Kettle Hood Flues or Pipes: These move freely up and down and forward and backward at the gentle pressure of the varnish maker's hand. The counterweighting is simple. Together with the hood they restrict the inflow of air and its admixture with the fume. In practice they are

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*It is understood that the cost per kettle per day for power required for fan and blower is 81 cents. A 25-kettle plant would, therefore, show a yearly power cost of over $5,000. This sum would represent interest on a considerable amount of money. In some instances, therefore, a "stack" might be cheaper than a fume condensing plant but would not do away with the odor.

understood to be substantially free from the building up of fume deposits.

Automatic Double Damper Boxes: These consist of precautionary or safety equipment to protect the flue system. They prevent the communication of flash from one kettle to another through the flues. They are equipped with explosion heads. Should ignition and combustion accidentally occur in a flue the flame becomes automatically directed toward the separate flue used ordinarily to remove the combustion gases from the fires. The large area and the suction present, are designed to make excessive pressures impossible. The fusing of the fire link causes the attached damper to fall shut, thereby preventing flame from reaching the manifold. This damper in turn, by its weight and motion and by the wire rope connection automatically opens the other damper connecting with the combustion gas flue. The system of control, although automatic, is also subject to hand operation so that it can be tested frequently to insure its free working. The damper boxes are further provided with drips to prevent the accumulation of condensed inflammable materials.

Screen Boxes: Following the automatic damper is a box divided by a double screen of fine mesh which serves as an auxiliary protection against communication of flame. The action of this screen box is analogous to that of the well known Davey miner's safety lamp. These screens are not permanent fire arresters but are designed to act for the short space of time necessary to fuse the fire link on the preceding damper, which gives a positive permanent cut-off. A drip is also provided in the box to prevent the accumulation of condensed inflammable materials.

Collecting Manifold: The several screen boxes are joined to a common fume collecting manifold by means of suitable lengths of flue. The manifold is carried out of the building at a slope to allow drainage of the condensate, and connects with the condenser flue. These flues are drained at the low point of junction to prevent the accumulation of condensate and to provide a means of collecting condensate.

Surface Condenser and Primary Condensate: This flue is preferably placed outside of the building and receives the fume from the manifold. It is installed with a slight inclination from the horizontal, in order to collect the condensate at a suitable collecting point. Those portions of the fume that are not condensed therein are discharged for treatment and recovery in the scrubbing equipment and odor reaction equipment. These two latter equipments have usually been housed on the roof of the plant. For this purpose a pent house of light and inexpensive construction for protection from the weather is provided.

Scrubbing Condenser and Intermediate Condensate: This involves, of course, a wet process. The fume fractions discharged from the primary condenser are carried to an appropriate manifold at the bottom of the treatment tank. This manifold is submerged under an appreciable depth of liquid. By creating a suction beyond this part of the system, the fume is carried up and through the liquid, and the soluble constituents of the fume are removed. The remaining fraction of the fume is drawn from this tank and is carried through the odor reaction equipment.

Odor Reaction Equipment and End Product: This also involves a wet process. The end fume passes into a moderate size tank and at the bottom of this tank escapes upward through the liquid contents, being broken into a series of bubbles to give reactive surface. The tank is filled either with an alkaline solution made with a soluble alkaline salt or an emulsion of milk of lime. The fume reacts with the reagent, and alkaline solid or liquid compounds of the organic acids of the fume are produced. The vent gases from these tanks are practically free from objectionable odor. The end product referred to is usually collected weekly.

Fume Control Applied to Stack Plants: For those plants where heavy stacks are in existence it is stated that the standard apparatus may be provided, the stacks continuing their task of discharging the products of combustion from the kettle fires. The equipment necessary for such plants would be kettle hoods, cooling fume hoods, sheet metal flues, fire safety devices of sheet metal, tanks and tubs for scrubbing and chemical reaction, and a power driven motor and blower to move the fumes through the system.

Equipment for New Varnish Plants to be Provided With Fume Control: Later developments in the operation of the process and apparatus described above have resulted in the building up of a flexible system of a standard design of equipment. It is referred to as multi-story type plants. These plants provide for the storage of raw materials on floors above the kettle or boiling room. The finished product is stored on the floor beneath the kettle room. The advantages are stated to be better control of inventory and formula, with installation of costing systems and incident economies in materials. A new type of building is provided, with certain economies in construction and investment, such as entire elimination of bulky varnish stacks. Reduction of ground and roof area is accomplished. The above advantages, together with fume recovery, odor elimination, and reduction of fire hazard are presented. The multi-story system for a varnish department in paint factories has also been developed. The following description of the design of one such plant now under construction would be of interest.

The varnish department is stackless, being similar in assemblage to the paint department. On each of the four floors the flow sheet or steps in varnish manufacture approximates the flow sheet or steps in paint manufacture. On the fourth floor are located the stocks of raw materials. On the third floor is located the boiling room with its kettle fires and kettles and also the reducing room for introducing the thinners. The hoods for fume collecting, as well as the thinning room hoods are located on this floor for fume control and recovery of both varnish fumes and volatiles. On the second floor are located the centrifugals or other types of filters used in varnish manufacture, as well as the ageing or storage tanks. On this floor may be placed apparatus for accessory manufacture such as tumbling barrels used for making spirit varnishes. The ground floor is usually a portion of the plant factory; the filling, labeling, packing and shipping being carried on at this point. The basement under both paint and varnish departments is available for the usual purposes such as storage for oil tanks and excess raw materials not needed for immediate use on the fourth floor. A fire wall from the building foundations through all the floors, separates the paint department from the varnish department, and extends above the roof of the building as a parapet wall. This wall has no breaks or openings for communication above the ground floor. Communication between floors is, however, provided by exterior stairways and open balconies between floors. There is, therefore, no communication whatsoever within the building between the varnish department and the paint department. Even the pipes which permit of transfer of liquids from one department to the other and between floors are run through exterior conduits designed for protection from fire.

A coke bin is provided on the fourth floor of the plant. All the raw materials such as liquids, gums and coke are obtained on the third floor by valves and simple hopper levers operated from the kettle room.

Some of the advantages claimed for the above system are efficiency of labor, with favorable conditions of light and air for occupational comfort; elimination of unnecessary handling and hauling of the kettles; weight and measure control of the materials delivered into the kettles; recovery of all kettle fumes during the cooking and cooling; recovery equipment for handling volatile thinners distilled from the kettles in the reduction room; protection of plant from fire hazard, etc.

In the operation of the plant described above, the raw materials are obtained from the fourth floor through the hoppers in specified quantities in accordance with the standard formulae that are used. This avoids the storage of excess materials on the third floor where the fires are maintained. From the fires, the kettles are hauled to the cooling equipment. Separate hoods or covers might be preferred by some manufacturers for this purpose. Others might prefer to keep the kettles covered during the entire period of cook, cooling and reduction. In such case recovery equipment is connected with each of the three locations of cook, cooling and reduction. When cool the kettles are removed into the reduction room by passing out of the building on to the exterior balcony, and thence into the reduction room. The recovery of thinners is accomplished by a system entirely separate from the fume recovery system used during the cooking and cooling. From the reduction room the varnish is dropped by gravity to the filtering and storage room on the second floor, by the aid of conduits passing on the outside of the building. The filtering equipment is located on a mezzo floor or balcony from which the varnish flows by gravity into the ageing or storage tanks.

Fire Hazard in Varnish Plants: Fire hazard may possibly be inherent in certain old style varnish plants and the manufacturing methods used therein. Claims are made that this hazard may be reduced to a minimum by the new system of fume control. A summarized discussion of the means by which these results are obtained is believed warranted.

In the old style stack plants, fires occur usually from spills or from kettle flash. Kettle flash may come from over-heating or from the escaping fumes being blown by some chance air current into the open fire under the kettle. In any case, one of the most effective ways to subdue fire in the kettle is to smother the flame with a tight cover. This method is efficacious but hazardous. Too long an interval may elapse between the flash and the placing of the cover on the kettle by the workmen. Considerable interesting data on the subject of fire fighting devices in varnish plants is presented in Service Bulletin No. 60 of the National Varnish Manufacturers' Association. In this Bulletin the comparative value of the various types of sprinklers and extinguishers is presented. The equipment recommended, especially the type that throws a "chemical blanket" upon burning liquids, constitutes a very valuable adjunct toward fire elimination.

Fires resulting from spills, with subsequent ignition, are rarely, if ever, of sufficiently large volume to in themselves constitute a serious risk or to do any damage other than to the materials directly involved. Nevertheless spills from open kettles become a menace because of the probability that flame from them will be communicated to other open kettles. With the close hooding of the kettles and the removal of all fumes, fire hazard from the above cause is largely removed. Should a kettle flash from over-heating, the tight fitting cover provided by the fume system is already in place, and the closing of the stirring openings and pipe damper, both of which are readily approached, completes the blanketing and the fire may be smothered at once. It is understood that this apparatus has been equally successful in preventing communication of fires in the case of open kettles of highly heated oil that were deliberately set on fire in the room in which the regular varnish making operations were being conducted. This indicates that the danger from accidental fire in the melting room may be largely eliminated.

Consideration of the possibility of fire traversing the fume piping is next in order. The pipes leading from the kettle hood terminate in an automatic double damper box heretofore described. This box, in turn, communicates to the screen box. Leaving the screen box, the fume is drawn by suction into the manifold and through the surface condenser into the water condenser and scrubber. The scrubber wets down the gases and interposes a water barrier in the system.

By providing a non-inflammable building with proper heat resisting materials, it is claimed that no additional risk is introduced by the storage of inflammable material, provided any fire occurring on the lower floor from the causes already noted is positively prevented from communicating to the upper or storage floor. In the construction of such buildings, reinforced concrete, brick and steel, with steel sash and wire glass, are used. The floor separating the upper and lower stories is of solid reinforced concrete construction, without openings except for the hoppers. The hoppers are of cast iron and steel with self-closing doors. They are grouted into the floor. If, for instance, the storage tanks on the upper floors should be ruptured, flooding of the lower floors could be prevented by means of the curbs placed about hopper openings and by outside floor drains.

The idea of locating open or closed fires or furnaces in upper stories has been practiced in certain other industries for many years, and the engineering design and materials necessary apparently present no new or untried problems. By completely enclosing the fire pits with steel lined with fire brick, they may project beneath the floor below. While it may be advanced that in case the brick lining of the fire pits is destroyed the steel casing would burn through. this probably could only happen after long warning as to the condition of the brick. Care and reasonable inspection should prevent such happenings.

Some consideration should now be given to the risks involved in reducing the cooked material with the volatile and inflammable liquids used in this art. Common practice has heretofore been to carry out this operation in the open or under a small shed roof for weather protection. The reduction is usually done in the uncovered kettles with the vapors being given off freely to the air. These vapors when mixed with air may become increasingly inflammable, and being heavier than air will tend to settle down around the more highly heated truck frame and wheels. For this reason it is common practice in some factories to cool the truck and wheels with a stream of water before reducing the varnish, when reduction is contemplated at relatively high temperatures. By the introduction and use while reducing, of properly designed kettle hoods, from which all vapors are continuously removed, the risk of flash or ignition is practically eliminated. The reducing agents may be introduced through pipes sealed in the hood. An exhaust pipe leads from the hood to a condensing system and is so regulated as to practically maintain a condition of balanced draft. Further safeguards in this method are accomplished by having the reducing room completely walled off from the boiling room with a fire wall. The exhaust pipes from the reducing house are led outside the building into suitable condensers equipped with suction fans located in the fume treatment house or any other suitable place.

When a multi-story varnish plant is incorporated as an integral part of a larger building which, for instance, may contain a separate paint factory, fire protection is given by the fire wall extending without openings through the roof from the ground. This fire wall may be expected to completely protect the varnish department from fire in the remainder of the building or the building from possible fires in the varnish department.

The results obtained from the above-described system of fume control and varnish factory design will be watched with great interest.

CHAPTER XIII

Tests of Moisture and Water Resistance of Various Coatings on Small Boat Construction

It is obviously necessary to render fairly waterproof the pontoon, float, or hull surfaces of seaplanes, flying boats, or other craft constructed of wood. If this is not accomplished, considerable water will be admitted, which will add greatly to the weight and materially diminish the speed obtainable and the general efficiency of the craft. Unfortunately, the word "waterproof" has been used to describe the film that is produced by protective coatings of nearly every class, whereas, some should not be so classed. In order to determine the actual value of various types of coatings that are used by the boat builder for finishing small craft a series of tests were made, which have afforded considerable data of a valuable nature.

Before describing these tests it might be well to point out that wood is a cellular substance, the fibrous part of which absorbs large quantities of moisture. When the fiber becomes saturated with moisture, further quantities of water may be admitted in liquid form. This water will be held in the cellular cavities of the wood. When wood that has absorbed either water vapor into its fiber or liquid water into its cellular cavities, is submitted for a considerable period of time to drying, both the water and the moisture pass out and the wood is restored to its original so-called air-dry form which under normal conditions of testing and fabrication, should contain in the neighborhood of 11 to 15 per cent of moisture. Spruce, for instance, such as is used in the manufacture of the various parts of seaplanes, is always conditioned to the above moisture content before working.

For making the tests, there was selected a series of carefully dressed panels of airplane spruce and of yellow poplar. The edges of each panel were carefully rounded and all surfaces made smooth. The panels were of the following dimensions (4″ x 8″ x ½″), there being thus afforded by each panel a total area of approximately one square foot. After drying the panels for ten days in the laboratory at a temperature of 70° F., they were given three coats of the compounds that were used in the tests, allowing ample periods for the hard drying of each coat. After the third coat was dry the panels were allowed to dry for a further period of ten days, and were then carefully weighed and placed in a specially constructed moisture cabinet (See Fig. 2) kept at a temperature of 100° F., with a relative humidity of 95 per cent. At the end of a period of seven days the panels were removed from the cabinet and the surfaces wiped off, to remove any extraneous water that had been deposited by condensation. The panels were then carefully weighed and the gain in weight due to moisture absorption was recorded. After a period of seven days' drying at 70° F. they were reweighed and immersed in a specially constructed tank (Fif. 15) containing distilled water; being hung from the bottom so that they would float upward. The completely immersed panels were allowed to soak for a period of seven days. They were then removed, the surfaces carefully wiped, and weighed. The gain in weight due to water absorption was recorded. The panels were again allowed to dry for seven days at a temperature of 70° F. and reweighed to determine the amount of water retained within the structure of the wood.

The materials used for coating the wood panels need but little description. The raw linseed oil and boiled linseed oil conformed to Navy Department Specifications. The shellac, acetate airplane dope, nitrate airplane dope, spar varnish, and enamels conformed to Aeronautical Specifications of the Navy Department. All of the enamels were made with No. 3 varnish containing 25—30—40 and 15 per cent respectively, of pigment. In the charted results these enamels are referred to as No. 4 a, b, c, and d, respectively. The first three contained lead and zinc pigments, while the last contained aluminum powder as the pigment. The oil (N) dope was a cellulose nitrate dope containing a drying oil, and the same material was used in combination with gray pigment to form the pigmented oil (N) dope. The panel painted with oil-graphite was first treated with a flat drying carbon black-linseed oil paint. The graphite was then dusted on while the film was tacky. This operation was repeated until a smooth surface was built up, two coats being necessary for this purpose. The panel coated with varnish-graphite was treated with two coats of No. 3 spar varnish containing 20 per cent of graphite. Dry graphite was then dusted on the film while it was still tacky, and by polishing with burlap a bright, smooth surface was developed.

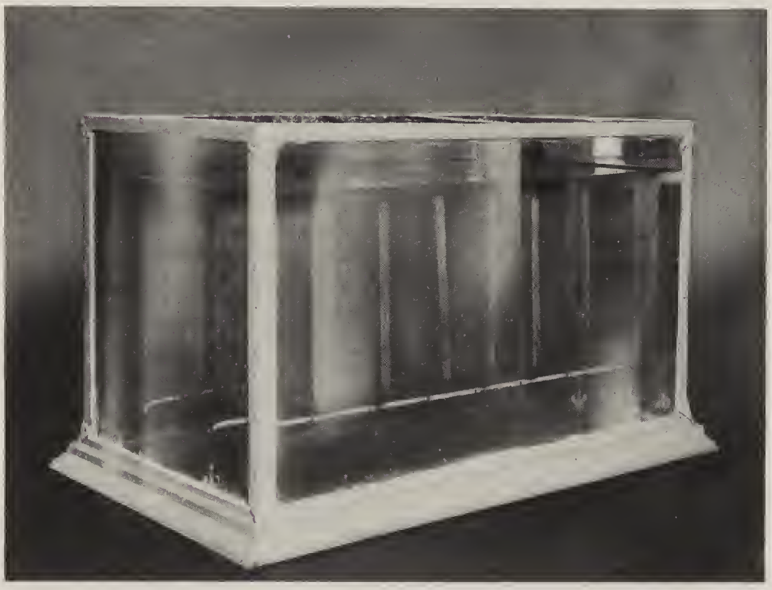

Fig. 15.
SOAKING TANK

The results which are tabulated below, expressed in ounces of water gained per square yard, show that there is a marked difference in the amount of moisture and the amount of water which may be absorbed through films of different types. The results also show that while a film may be relatively moisture-proof, it may not be waterproof.

Another interesting feature is shown by the apparent ability of some films to allow large quantities of moisture or water to pass through into the wood but relatively small quantities to pass out after being submitted to drying, thus allowing the wood to retain large quantities of water. The acetate and nitrate dopes and the oil-graphite are representatives of this class.

Special attention is directed to the very large amount of water absorbed through raw linseed oil films, as compared to the amount absorbed by the films of spar varnish. It would seem evident from these results that spar varnish forms either a more continuous and less porous film, or one that is less subject to intermolecular absorption. The passage of water through a paint film is probably due to osmotic action, the film acting as a semi-permeable membrane. With increase of temperature and vapor pressure, increase of penetration is shown. The water may also pass through in some cases by first forming an emulsion with the film, although such action may probably be considered as playing a minor role in the phenomenon. If a paint should form a film that is absolutely impermeable it might be unsuited for use on certain types of wood construction (dwellings), as the moisture in the wood is continually being drawn out to the surface by the action of the sun and would tend to cause blistering of an impermeable film. A film that shows only slight permeability to water but which allows water which is in the wood to escape freely, without affecting the film, should be considered satisfactory for most small boat protection.

It should be noted that some panels coated with No. 4

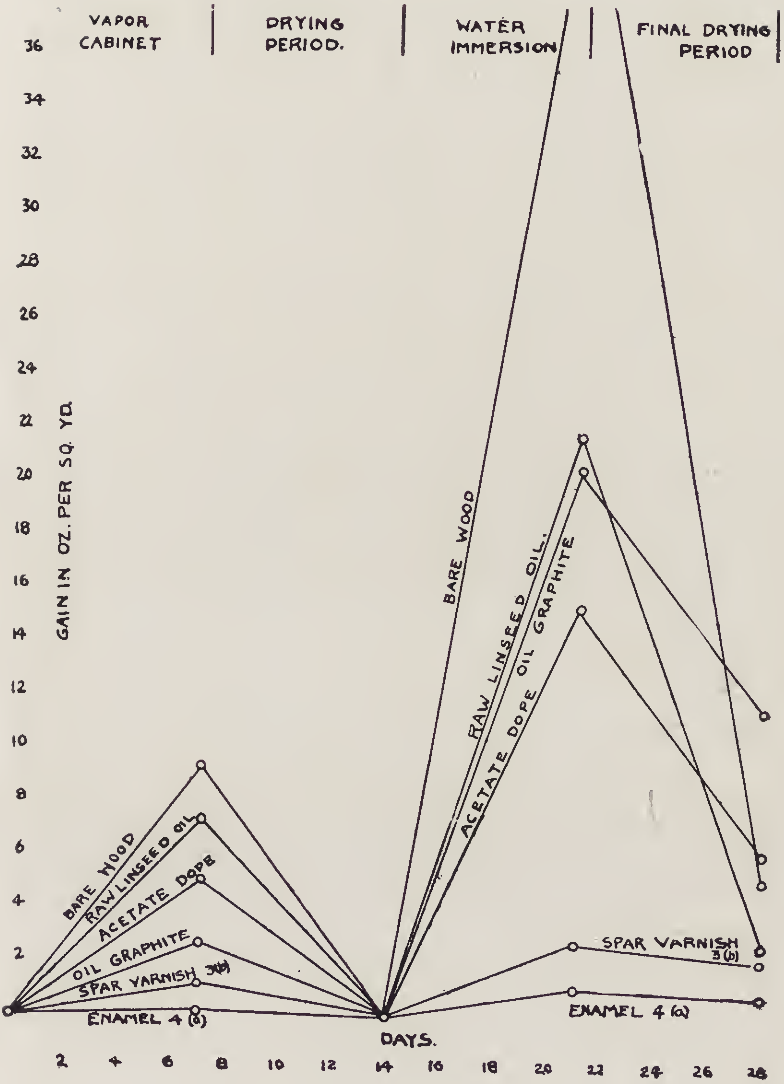
VAPOR CABINET
DRYING PERIOD.
WATER IMMERSION
FINAL DRYING PERIOD
GAIN IN OZ. PER SQ. YD.
36
34
32
30
28
26
24
22
20
18
16
14
12
10
8
6
4
2
BARE WOOD
RAW LINSEED OIL
ACETATE DOPE
OIL GRAPHITE
SPAR VARNISH 3(b)
ENAMEL 4(a)
BARE WOOD
RAW LINSEED OIL.
OIL GRAPHITE
ACETATE DOPE
SPAR VARNISH 3(b)
ENAMEL 4(a)
DAYS.
2
4
6
8
10
12
14
16
18
20
22
24
26
28

Fig 16.

wing enamel,* which is composed of No. 3 spar varnish and pigment, gave even better results than the spar varnish alone, thus indicating the value of a percentage of pigment in rendering films more waterproof. This may to some extent be due to the thicker films which result from the application of an enamel. It should be further recorded that of the four grades of No. 4 wing enamel used, slightly better results were shown by those containing a high percentage of pigment as compared with those containing a relatively low percentage. The amount of water shut out, however, through the addition of a large amount of pigment may not be sufficient to justify the use of a heavier enamel, as such an enamel would increase the initial weight of a flying-boat considerably. Very good results were however shown by the No. 4 (d) enamel which contained a relatively small percentage of pigment (aluminum powder). The results obtained with the oil-graphite dust treatment of hull surfaces, which has been used by the British to some extent, were poor as compared to the present American practice of using No. 4 enamel. When No. 3 spar varnish was used in the British treatment, in place of oil, very good results were shown.

It will be observed that collodion dopes, or acetate dopes, are not to be compared with spar varnish as water excluders, although the dope designated as N, which contained a substantial percentage of varnish oil, gave much better results than the other dopes, which, incidentally, did not contain oil. Since some aircraft makers have often used dope in place of varnish for moisture-proofing woodwork, the above results would indicate that such practice should be discontinued.

The low moisture and water absorption shown by the shellac-coated panels is most interesting, as it has heretofore been believed that shellac was subject to rapid water absorption. The fact that shellac coatings almost immediately turn white when placed in water may be responsi-

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*Navy Gray Enamel—Aeronautical Specifications C. and R. No. 4-B.

ble for such belief. This whitening of the film should apparently not be attributed to water that has passed through the coating but probably to the hydration of the outer surface of the shellac film. From the standpoint of durability however, shellac has proved very inferior to spar varnish when exposed to sunlight and the atmosphere for long periods of time.

A further series of experiments were made with some of the enamels by applying four coats to new panels and then submitting the panels to the same type of water absorption apparatus as described above. A remarkable reduction in absorption was shown. The results, which are rather astounding, would indicate that the application of four coats of enamel to boat construction would be well repaid by the very high water-resisting films thus obtained.

It is the writer's belief that the type of enamel that has proved so successful in these experiments should be used to a much greater extent than heretofore. Its highly successful use on the hull surfaces and also as a protective over the dope on the fabric covered wings of several hundred seaplanes has demonstrated its value and possibilities. It may have application as a general protective for exterior marine work and prove more successful than some of the paints now in use for such purpose.

Tests were not made of the newly devised process of the Forest Products Laboratory, of waterproofing wooden surfaces, such, for instance, as propellers. In this process the wood is coated with a varnish and then aluminum leaf is applied. The aluminum leaf is subsequently finished with varnish or paint. It is understood that the highest degree of moisture and water resistance is afforded by this process which might find application for use on certain parts of small boat construction.

TABLE 52.
Spruce Panels

Material.	Gain in moisture cabinet, oz. per sq. yd.	Gain in water tank, oz. per sq. yd.	Water retained after drying, oz. per sq. yd.
Bare Wood	7.04	37.83	1.45
Raw Linseed Oil	6.01	23.20	2.18
Boiled Linseed Oil	4.80	12.50	2.74
Acetate Dope	3.85	20.85	6.70
Nitrate Dope	4.18	21.50	4.73
Oil N. Dope	2.34	5.45	3.32
Pigmented Oil N. Dope	2.04	5.14	3.88
Spar Varnish No. 3 (*a*)	.87	.57	.32
Spar Varnish No. 3 (*b*)	.78	3.36	1.27
Enamel No. 4 (*a*)	.13	.68	.36
Enamel No. 4 (*b*)	.48	1.39	.77
Enamel No. 4 (*c*)	.77	1.75	1.37
Enamel No. 4 (*d*)	.24	1.60	1.46
Shellac	.52	1.48	1.21
Oil-Graphite	2.64	33.22	14.10
Varnish-Graphite	1.31	5.32	1.46

TABLE 53.
Yellow Poplar Panels

Material.	Gain in moisture cabinet, oz. per sq. yd.	Gain in water tank, oz. per sq. yd.	Water retained after drying, oz. per sq. yd.
Bare Wood	9.54	47.78	5.20
Raw Linseed Oil	7.43	21.81	2.70
Boiled Linseed Oil	5.37	15.80	5.30
Acetate Dope	5.20	15.40	6.20
Nitrate Dope	5.00	14.10	5.50
Oil N. Dope	2.40	4.76	3.24
Pigmented Oil N. Dope	.29	3.02	2.95
Spar Varnish No. 3 (*a*)	1.40	3.42	2.81
Spar Varnish No. 3 (*b*)	1.32	2.88	2.20
Enamel No. 4 (*a*)	.16	1.18	.96
Enamel No. 4 (*b*)	.76	1.52	1.21
Enamel No. 4 (*c*)	1.54	3.20	2.42
Enamel No. 4 (*d*)	.47	1.04	.72
Shellac	.47	2.01	1.89
Oil-Graphite	2.85	20.60	11.80
Varnish-Graphite	.82	2.42	1.50

TABLE 54.

Water Absorption of Panels with 3 and 4 Coats of Enamel.

3-Coat Work.		4-Coat Work.
Gain in water, ozs. per sq. yd.		Gain in water, ozs. per sq. yd.
1.18	Enamel No. 4 (*a*)	.19
1.04	Enamel No. 4 (*b*)	.27

CHAPTER XIV

New Exposure Tests at Atlantic City

A new series of test panels exposed on May 15th at Atlantic City are expected to afford considerable information on certain paint problems of general interest to the industry. While the tests are not of a competitive nature, as have been most of those heretofore made, some of the panels are coated with products which have been developed in recent years and not included in previous tests. The panels have been arranged on the upper deck over the old wave motor located near the outer reaches of Young's Million Dollar Pier.

Fig. 17.
VIEW OF WOOD PANELS

The tests are in groups which are described below. Among them are some which are designed to show the comparative life of uncoated and coated structural materials. As is well known, the durability of such materials depends on surface protection. Bare steel, wood, or other structural materials of the lowest or of the highest grade are all rapidly acted upon by the elements, but all may be preserved indefinitely if their surfaces are protected by occasional renewals of paint or varnish coatings. In order to show the actual effect of surface coatings, some of the panels have been exposed to the trying conditions existing along the seacoast with half of their surfaces uncoated, the area of the other half being given surface protection. Exposure over a short period will indicate the comparative value of the untreated and treated surfaces.

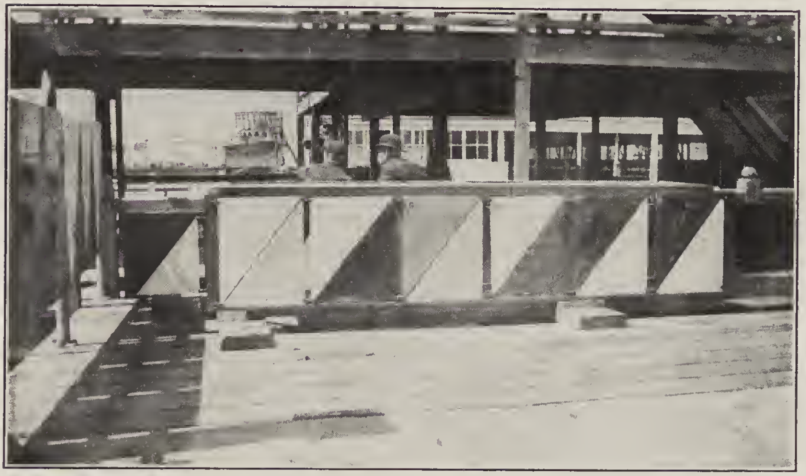

Fig. 18.

VIEW OF METAL PANELS

TABLE 55.

Series I—Tin Plate

Panel No.	Description of panel	Section of panel	Treatment
1	E-X (Tin Coated)	Upper Lower	Vermilion Paint, 3 coats Bare
2	E-X (Tin Coated)	Upper Lower	Spar Varnish Bare
3	E-X (Tin Coated)	Upper Lower	Collodion Dope D, 1 coat Bare
4	A-P 40 lb. (Heavy Tin Coated)	Upper Lower	Vermilion Paint, 3 coats Bare
5	A-P 40 lb. (Heavy Tin Coated)	Upper Lower	Spar Varnish, 3 coats Bare
6	A-P 40 lb. (Heavy Tin Coated)	Upper Lower	Collodion Dope N, 1 coat Bare

The above sheet tin roofing plates are exposed in a horizontal position. The vermilion paint contains 50% basic chromate of lead, 40% raw linseed oil, 5% turpentine and 5% drier. The spar varnish is W. D. 6.* The dopes used are collodion dopes containing substantially 6 oz. of nitrocellulose per gallon of solvent. No. 3 contains approximately 6% castor oil, and No. 6 contains approximately 6% treated tung oil.

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*W. D. stands for War Department formulas published in War Department Paint Pamphlet No. 1.

TABLE 56.

Series II—Black Iron Metal Powder Tests

Panel No.	Description of panel	Section of panel	Treatment
7	Open Hearth Steel (Black Sheet)	Upper	Aluminum Powder in Spar Varnish, 2 coats
		Lower	Aluminum Powder in Linseed Oil, 2 coats
8	Open Hearth Steel (Black Sheet)	Upper	Aluminum Powder in Collodion Dope D, 2 coats
		Lower	Bare
9	Open Hearth Steel (Black Sheet)	Upper	Zinc Powder in Spar Varnish, 2 coats
		Lower	Zinc Powder in Linseed Oil, 2 coats
10	Open Hearth Steel (Black Sheet)	Upper	Zinc Powder in Collodion Dope D, 2 coats
		Lower	Bare

Aluminum and zinc powders have high protective action when placed in contact with iron and steel. They are strongly electro-positive and protect the underlying ferrous metal from corrosion. In the above series, exposure will indicate whether spar varnish, linseed oil or collodion dope is the most satisfactory vehicle for their application. Because of the viscous nature of spar varnish and dope, smaller quantities of the metallic powders are required than when raw linseed oil is used. Only 20% of aluminum powder was used with 80% of either spar varnish or dope, whereas 40% of aluminum powder was required with 60% of linseed oil in order to make a paint that would not show running. The drying of linseed oil is apparently greatly retarded through the use of aluminum powder. For that reason, better results might have been obtained by the use of boiled linseed oil which is more viscous and rapid drying than the raw oil, and better suited as a carrier for the aluminum powder.

With 50% of zinc powder, 50% of spar varnish or dope was used, whereas 86% of zinc powder was required for 14% of raw linseed oil in order to make a paint of brushing consistency. For this reason, boiled linseed oil would be preferable to raw linseed oil for use with zinc powder, as a lower amount of zinc dust could have been used.

TABLE 57.

Series III—Special Metal Tests, Including Galzanized Sheets.

Panel No.	Description of panel	Section of panel	Treatment
11	Open Hearth Steel (Galvanized)	Upper Lower	Spar Varnish, 3 coats Bare
12	Open Hearth Steel (Galvanized)	Upper Lower	Linseed Oil, 3 coats Bare
13	Open Hearth Steel (Galvanized)	Upper Lower	Copper brushed, then vermilion paint, 3 coats Bare
14	Open Hearth Steel (Black Sheet)	Upper Lower	Vermilion paint, 3 coats Bare
15	Copperized Steel (Galvanized)	Upper Lower	Copper brushed, then vermilion paint, 3 coats Bare
16	Copperized Steel (Black Sheet)	Upper Lower	Vermilion paint, 3 coats Bare
17	R-R Iron (Galvanized)	Upper Lower	Copper brushed, then vermilion paint, 3 coats Bare
18	R-R Iron (Black Sheet)	Upper Lower	Vermilion paint, 3 coats Bare
19	R-R Iron (Heavy Tin Coating)	Upper Lower	Vermilion paint, 3 coats Bare

The same type of vermilion paint and spar varnish as in Series I were used in this series. The copper brush treatment consisted of applying an aqueous solution of copper salt (6 oz. copper acetate to 1 gallon of water). This was applied in order to remove the highly spangled surface of the galvanized iron, which in some cases will cause flaking of paint after exposure.

TABLE 58.
Series IV—War Paints on Metal

Panel No.	Description of panel	Section of panel	Treatment
20	Open Hearth Steel (Black Sheet)	Upper	Spar Varnish (W. D. 6) 3 coats
		Lower	Bare
21	Open Hearth Steel (Black Sheet)	Upper	Spar Varnish, 3 coats
		Lower	Linseed Oil, 3 coats
22	Open Hearth Steel (Black Sheet)	Upper	Camouflage O. R. 333, 3 coats
		Lower	Camouflage N. O. R. 334, 3 coats
23	Open Hearth Steel (Black Sheet)	Upper	Yellow W. D. 37 and Olive Green W. D. 33 Projectile Paints, 3 coats
		Lower	Blue W. D. 34 and Black W. D. 38 Projectile Paints, 3 coats
24	Open Hearth Steel (Black Sheet)	Upper and Lower	Brown Enamel Primer W. D. 13, 1 coat; Red Oxide W. D. 11, 2 coats
25	Open Hearth Steel (Black Sheet)	Upper	Reinforced Red Lead, Ready Mixed, W. D. 40, 3 coats
		Lower	Red Lead, Hand Mixed, 3 coats
26	Open Hearth Steel (Black Sheet)	Upper	Helmet Paint, 3 coats
		Lower	Navy Camouflage, Gray, 3 coats.
27	Open Hearth Steel (Black Sheet)	Upper	Standard Olive Drab W. D. 28, 3 coats
		Lower	Quick Drying Olive Drab W. D. 29, 3 coats

In this series are included many war time paints such as were specified in Paint Pamphlet No. 1 of the War Department. The red lead hand-mixed paint consisted of 22 lbs. red lead, 5 pints linseed oil, 2 gills drier, and 2 gills petroleum spirits. The helmet paint consisted of 60% of pigment composed of:

Ochre	60%
Lithopone	30%
Magnesium silicate	10%

The liquid consisted of:

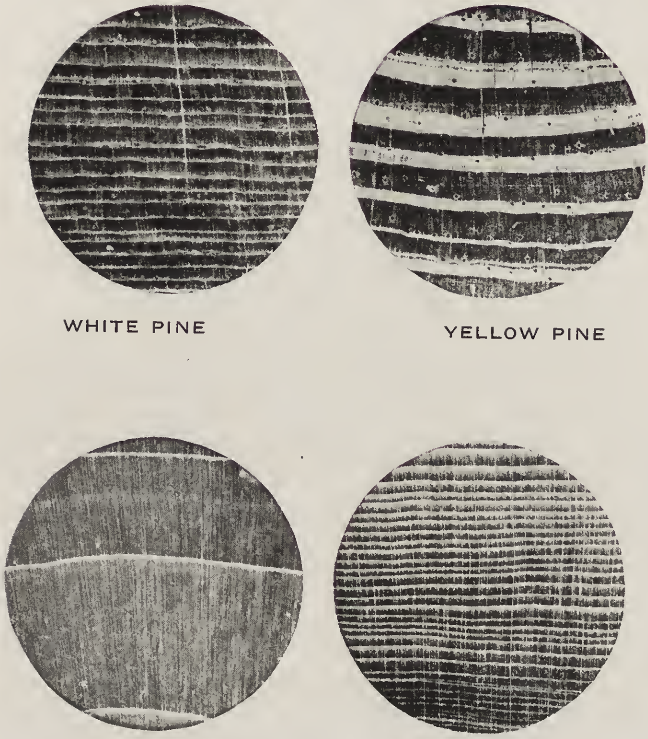
Linseed oil, tung oil, and varnish resins	40%
Drier and thinner	60%

The Navy Camouflage Gray consisted of: 65% of pigment composed of zinc oxide 47%, white lead 23%, magnesium silicate 30%. The liquid consisted of linseed oil 40%, spar varnish 20%, mineral spirits and drier 40%.

TABLE 59.

Series V—Wood Panel Tests

Panel No.	Description of panel	Section of panel	Treatment
50	White Pine	Upper Lower	White Paint, 3 coats Bare
51	Yellow Pine	Upper Lower	White Paint, 3 coats Bare
52	Cypress	Upper Lower	White Paint, 3 coats Bare
53	Red Wood	Upper Lower	White Paint, 3 coats Bare
54	White Pine	Upper Lower	Spar Varnish, 3 coats Bare
55	Yellow Pine	Upper Lower	Spar Varnish, 3 coats Bare
56	Cypress	Upper Lower	Spar Varnish, 3 coats Bare
57	Red Wood	Upper Lower	Spar Varnish, 3 coats Bare



WHITE PINE

YELLOW PINE

CYPRESS

REDWOOD

Fig. 19.

PHOTOMICROGRAPHIC REPRODUCTION OF WOODS

In this series the upper diagonal half of each panel was coated and the lower half left uncoated. Spar varnish (W. D. 6) was used on one set, and a prepared lead and zinc paint (W. D. 50) on the other.

TABLE 60.

Series VI—War Paints on Wood

Panel No.	Description of panel	Section of panel	Treatment
58	Cypress	Entire Panel	Primed with Red Lead, then
		Upper	Exterior Paint Pearl Gray W. D. 50, 3 coats
		Lower	Exterior Fire Resisting Paint, Gray Green W. D. 21, 3 coats
59	Cypress	Entire Panel	Primed with Red Lead, then
		Upper Board 1	Anti-Actinic Wing Enamel Army Olive Drab, 3 coats
		Upper Board 2	Anti-Actinic Wing Enamel Light Gray, 3 coats
		Lower Boards 1 and 2	Anti-Actinic Wing Enamel Semi-Flat Navy Department Aero. Spec. C. & R. No. 3, 3 coats

In this series special war time paints were included. As will be shown by Pamphlet No. 1, many of the War Department paints were of a quick drying nature, necessitating the use of spar varnish in the liquid portion. Since linseed oil paints have heretofore been used by the War Department, it will be interesting to observe the degree of durability to be obtained with varnish paints.

TABLE 61.

Series VII—Special Primer Tests on Wood

Panel No.	Description of panel	Section of panel	Treatment
60	Yellow Pine	Upper 2 Boards	Vermilion Paint, 1 coat
		Lower 2 Boards	Lampblack, 1 coat
		Right half of panel	Then coated with White Paint, 3 coats
		Left half of panel	Coated with Spar Varnish, 3 coats
61	Cypress	Upper 2 Boards	Vermilion Paint, 1 coat
		Lower 2 Boards	Lampblack, 1 coat
		Right half of panel	Then coated with White Paint, 3 coats
		Left half of panel	Coated with Spar Varnish, 3 coats
62	White Pine	Upper	Lampblack, 1 coat; Then White Paint, 3 coats
		Lower	Bare, then White Paint, 3 coats

The object of these special priming tests was to determine the results to be obtained through priming yellow pine and cypress with a hard drying primer made of basic chromate of lead, or with a primer made of lampblack. Over these primers white paint (W. D. 50) and varnish (W. D. 6) were applied. Exposure will determine whether the results obtained through the initial priming treatment are as good as or superior to those which are sometimes shown when this special priming treatment is omitted. It is the writer's theory that practically any type of wood may be painted with greater success if it is primed with a very fine pigment, such, for instance, as lampblack.

TABLE 62

Series VIII—Special Tests

Panel No.	Description of panel	Section of panel	Treatment
63	White Pine	1st Board 2nd Board 3rd Board 4th Board	Red Paint, 3 coats Green Paint, 3 coats Yellow Paint, 3 coats Black Paint, 3 coats
64	White Pine	Upper 2 Boards Lower 2 Boards	Red Oxide Machine Ground, 3 coats Red Oxide Hand Mixed, 3 coats
65	White Pine	Upper 2 Boards Lower 2 Boards	White Paint Pure Oil, 3 coats White Paint Foots Oil, 3 coats
66	White Pine	1st Board 2nd Board 3rd Board 4th Board	White Paint B-T-X Yellow Paint B-T-X Gray Paint B-T-X Blue Paint B-T-X
67	White Pine	1st Board 2nd Board 3rd Board 4th Board	White Paint L-Z-X Yellow Paint L-Z-X Gray Paint L-Z-X Blue Paint L-Z-X
68	White Pine	Upper 2 Boards Lower 2 Boards	Spar Varnish, 5 coats Linseed Oil, 5 coats

In this series are included tests of various colors on which temperature readings will be made to determine the heat absorption and comparative degree of solar radiation. Panel 64 was coated with two paints of exactly the same composition, one being machine ground and the other mixed by hand from the raw ingredients.

It is believed that exposure will demonstrate the value of machine grinding. On panel 65 there were applied two paints of exactly the same composition, using in one a pure raw linseed oil free from moisture and foots and in the other a linseed oil with considerable foots and a high moisture content. The use of oil of the latter type has in the past been responsible for cases of washing.

On panel 66 was applied a titanium oxide paint containing 60% of pigment and 40% of liquid. The pigment consisted of "titanium-barium sulphate" pigment 70% and zinc oxide 30%. The liquid portion consisted of linseed oil 85% and combined turpentine and drier 15%. In order to determine the wearing value of this paint there was applied on Panel 67, for control purposes, a high grade lead and zinc paint consisting of 66% pigment and 34% liquid. The pigment consisted of 47% white lead and 53% zinc oxide. The liquid consisted of 85% linseed oil and 15% combined turpentine and drier. The titanium oxide pigment used in the paint applied to Panel 66 is a pigment made by precipitation somewhat in the same manner as lithopone. It contains 16% of titanium oxide and 84% of barium sulphate. The hiding power of this pigment is comparable with that of white lead and zinc oxide. Its durability to severe exterior exposure is high. Both paints 66 and 67 were in white, yellow, gray, and green of the same tints.

The following additional wood and metal tests were exposed on June 8, 1919:

TABLE 63.

Panel No.	Description of panel	Section of panel	Treatment
69	White Pine	1st Board	White Paint B-T-XX
		2nd Board	Yellow Paint B-T-XX
		3rd Board	Gray Paint B-T-XX
		4th Board	Blue Paint B-T-XX
70	Cypress	1st Board	White Paint M-L
		2nd Board	Yellow Paint M-L
		3rd Board	Gray Paint M-L
		4th Board	Blue Paint M-L
71	Cypress	1st Board	White Paint N-L
		2nd Board	Yellow Paint N-L
		3rd Board	Gray Paint N-L
		4th Board	Blue Paint N-L

The paint used on panel 69 was made with a titanium-barium sulphate pigment containing 34% of titanium oxide and 66% of barium sulphate. The proportions were as follows: Pigment 55%, liquid 45%. The pigment composition was: Titanium-barium-sulphate 40%, barytes 40%, zinc oxide 20%. The liquid portion consisted of linseed oil 85% and combined turpentine and drier 15%.

The paint applied to panel No. 70 consisted of pigment 50%, liquid 50%. The pigment consisted of lithopone (type M) 70% and zinc oxide 30%. The liquid portion consisted of linseed oil 85%, combined turpentine and drier 15%.

The paint applied to panel 71 was of exactly the same composition as that on panel 70, except that lithopone (type M) was used.

Exterior tests with lithopone paints have heretofore indicated failure due to rapid chalking. The addition of zinc oxide overcomes this tendency to some extent, especially if the paints are tinted, and at the same time tends to prevent darkening due to light action. Panels 70 and 71 may indicate the approximate wearing value of two very good grades of lithopone now used in the paint industry.

TABLE 64.

Additional Metal Tests

Panel No.	Description of panel	Section of panel	Treatment
28	Steel Sheet (Parkerized)	Upper Lower	Vermilion Paint, 3 coats Bare
29	Steel Sheet (Parkerized)	Upper Lower	Spar Varnish, 3 coats Bare
30	Open Hearth Steel (Black Sheet)		1 coat Primer No. 1 over total surface, then 2 coats Finish No. 3 on upper diagonal half
31	Open Hearth Steel (Black Sheet)		1 coat Primer No. 2 over total surface, then 2 coats Finish No. 4 on upper diagonal half

Panels 28 and 29 were treated by a rust-proofing process, the surface of the metal being black due to the iron phosphide formed by boiling with dilute phosphoric acid compounds. The panels were dipped in a mixture of 1 part linseed oil and 7 parts naptha to give them the customary finish. After drying, they were painted. The vermilion paint and spar varnish were of the same grade as used in Series I. Paints No. 1 and No. 2 were reinforced red leads containing a small amount of red iron oxide to increase their hiding properties. Paints No. 3 and No. 4 were black and gray finishing coats.

A panel of special aluminum alloy (duralumin) with one-half of its surface coated with 3 coats of spar varnish W. D. 6 was also exposed on June 8, 1919.

Examination of Tests After Four Months Exposure.—The test panels were inspected on September 15, 1919, after a period of four months' exposure. At the time of inspection, brief notes were made of the condition of each panel and the results are presented below in chart form. Nearly all the metal panels, which were near the floor of the exposure deck, had been abraded during exposure, by workmen on the pier. These metal panels were therefore given one finishing coat of surface protection on September 22, 1919. The wood panels were not recoated as they were in a well-preserved condition.

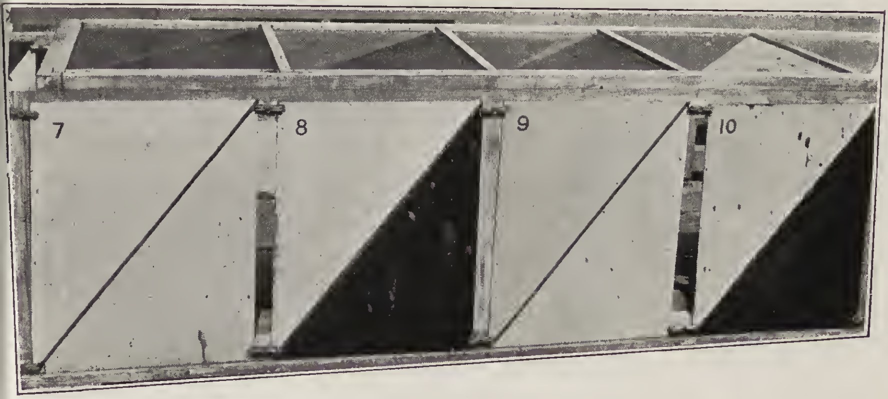


Fig. 20
Metal Panels on September 15, 1919
Corrosion of Uncoated Specimens Evident

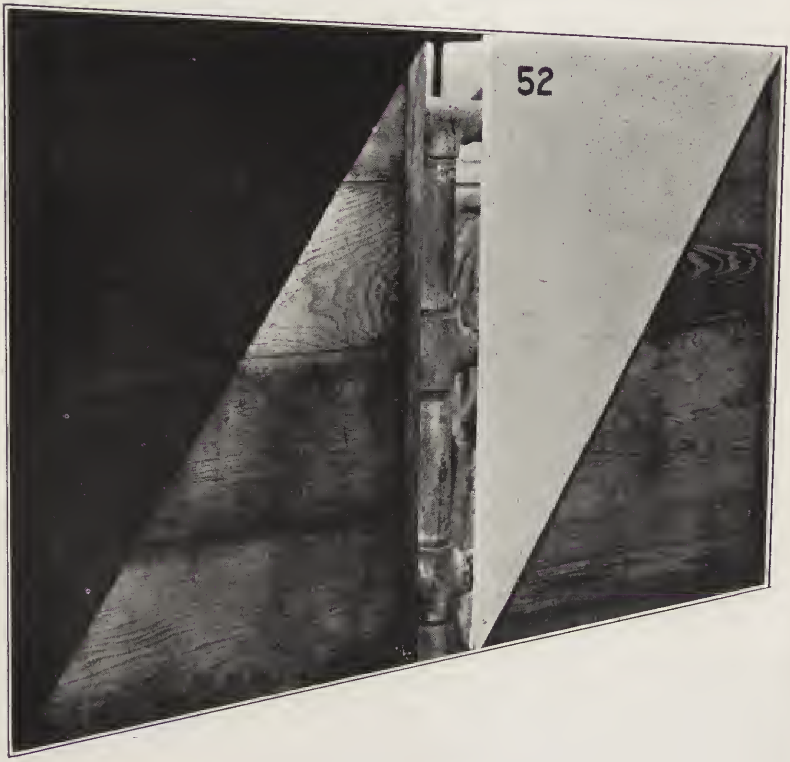


Fig. 21

Wood Panels on September 15, 1919
Eroded and Spotted Condition of Uncoated Specimens Evident

NEW EXPOSURE TESTS AT ATLANTIC CITY

TABLE 65.
Series I—Tin Plate

Panel No.	Description of panel	Section of panel	Treatment	Condition after 4 months' exposure
1	E-X (Tin Coated)	Upper	Vermilion Paint, 3 coats	Okeh
		Lower	Bare	Very rusty
2	E-X (Tin Coated)	Upper	Spar Varnish	Okeh
		Lower	Bare	Very rusty
3	E-X (Tin Coated)	Upper	Collodion Dope D, 1 coat	Okeh
		Lower	Bare	Very rusty
4	A-P 40 lb. (Heavy Tin Coated)	Upper	Vermilion Paint, 3 coats	Okeh
		Lower	Bare	Few small pin-hole rust spots
5	A-P 40 lb. (Heavy Tin Coated)	Upper	Spar Varnish, 3 coats	Okeh
		Lower	Bare	Few small pin-hole rust spots
6	A-P 40 lb. (Heavy Tin Coated)	Upper	Collodion Dope N, 1 coat	Peeling
		Lower	Bare	Few small pin-hole rust spots

TABLE 66.
Series II—Black Iron Metal Powder Tests

Panel No.	Description of panel	Section of panel	Treatment	Condition after 4 months' exposure
7	Open Hearth Steel (Black Sheet)	Upper	Aluminum Powder in Spar Varnish, 2 coats	Okeh
		Lower	Aluminum Powder in Linseed Oil, 2 coats	Okeh
8	Open Hearth Steel (Black Sheet)	Upper	Aluminum Powder in Collodion Dope D, 2 coats	Slight rust at abrasion points*
		Lower	Bare	Very rusty
9	Open Hearth Steel (Black Sheet)	Upper	Zinc Powder in Spar Varnish, 2 coats	Okeh
		Lower	Zinc Powder in Linseed Oil, 2 coats	Okeh
10	Open Hearth Steel (Black Sheet)	Upper	Zinc Powder in Collodion Dope D, 2 coats	Very slight rust at abrasion point
		Lower	Bare	Very rusty

*Protection of abraded areas by the electropositive aluminum apparently counteracted y the underlying black mill scale which is strongly electronegative to the iron surface and therefore rust stimulative in action.

TABLE 67.

Series III—Special Metal Tests, Including Galvanized Sheets

Panel No.	Description of panel	Section of panel	Treatment	Condition after 4 months' exposure
11	Open Hearth Steel (Galvanized)	Upper	Spar Varnish, 3 coats	Okeh
		Lower	Bare	Okeh
12	Open Hearth Steel (Galvanized)	Upper	Linseed Oil, 3 coats	Okeh
		Lower	Bare	Okeh
13	Open Hearth Steel (Galvanized)	Upper	Copper brushed, then vermilion paint, 3 coats	Okeh
		Lower	Bare	Okeh
14	Open Hearth Steel (Black Sheet)	Upper	Vermilion paint, 3 coats	Okeh
		Lower	Bare	Very rusty
15	Copperized Steel (Galvanized)	Upper	Copper brushed, then vermilion paint, 3 coats	Okeh
		Lower	Bare	Okeh
16	Copperized Steel (Black Sheet)	Upper	Vermilion paint, 3 coats	Okeh
		Lower	Bare	Very rusty
17	R-R Iron (Galvanized)	Upper	Copper brushed, then vermilion paint, 3 coats	Okeh
		Lower	Bare	Okeh
18	R-R Iron (Black Sheet)	Upper	Vermilion paint, 3 coats	Okeh
		Lower	Bare	Very rusty
19	R-R Iron (Heavy Tin Coating)	Upper	Vermilion paint, 3 coats	Okeh
		Lower	Bare	Rust spotted

TABLE 68.

Series IV—War Paints on Metal

Panel No.	Description of panel	Section of panel	Treatment	Condition after 4 months' exposure
20	Open Hearth Steel (Black Sheet)	Upper	Spar Varnish (W. D. 6), 3 coats	Okeh
		Lower	Bare	Very rusty
21	Open Hearth Steel (Black Sheet)	Upper	Spar Varnish, 3 coats	Okeh
		Lower	Linseed Oil, 3 coats	Rusty below oil film
22	Open Hearth Steel (Black Sheet)	Upper	Camouflage O. R. 333, 3 coats	Okeh
		Lower	Camouflage N. O. R. 334, 3 coats	Okeh
23	Open Hearth Steel (Black Sheet)	Upper	Yellow W. D. 37 and Olive Green W. D. 33 Projectile Paints, 3 coats	These four paints show very dull surfaces
		Lower	Blue W. D. 34 and Black W. D. 38 Projectile Paints, 3 coats	
24	Open Hearth Steel (Black Sheet)	Upper and Lower	Brown Enamel Primer W. D. 13, 1 coat; Red Oxide W. D. 11 2 coats	Okeh
25	Open Hearth Steel (Black Sheet)	Upper	Reinforced Red Lead Ready Mixed W. D. 40, 3 coats	Okeh but slightly dull
		Lower	Red Lead Hand Mixed, 3 coats	Okeh
26	Open Hearth Steel (Black Sheet)	Upper	Helmet Paint, 3 coats	Slight checking
		Lower	Navy Camouflage, Gray 3 coats	Okeh
27	Open Hearth Steel (Black Sheet)	Upper	Standard Olive Drab W. D. 28, 3 coats	Okeh
		Lower	Quick Drying Olive Drab W. D. 29, 3 coats	Okeh

TABLE 69.
Series V—Wood Panel Tests

Panel No.	Description of panel	Section of panel	Treatment	Condition after 4 months' exposure
50	White Pine	Upper Lower	White Paint, 3 coats Bare	All painted or varnished surfaces in excellent condition. All bare wood surfaces are rough, showing weather-etched grain and dark spots due to fungous growths
51	Yellow Pine	Upper Lower	White Paint, 3 coats Bare	
52	Cypress	Upper Lower	White Paint, 3 coats Bare	
53	Red Wood	Upper Lower	White Paint, 3 coats Bare	
54	White Pine	Upper Lower	Spar Varnish, 3 coats Bare	
55	Yellow Pine	Upper Lower	Spar Varnish, 3 coats Bare	
56	Cypress	Upper Lower	Spar Varnish, 3 coats Bare	
57	Red Wood	Upper Lower	Spar Varnish, 3 coats Bare	

TABLE 70.
Series VI—War Paints on Wood

Panel No.	Description of panel	Section of panel	Treatment	Condition after 4 months' exposure
58	Cypress	Entire Panel	Primed with Red Lead, then:	All okeh
		Upper	Exterior Paint, Pearl Gray W. D. 50, 3 coats	
		Lower	Exterior Fire Resisting Paint, Gray Green W. D. 21, 3 coats	
59	Cypress	Entire Panel	Primed with Red Lead, then:	All okeh except where bird shot have penetrated 2 upper boards
		Upper Board 1	Anti-Actinic Wing Enamel Army Olive Drab, 3 coats	
		Upper Board 2	Anti-Actinic Wing Enamel Light Gray, 3 coats	
		Lower Boards 1 and 2	Anti-Actinic Wing Enamel Semi-Flat Navy Department Aero. Spec. C. & R. No. 3, 3 coats	

TABLE 71.

Series VII—Special Primer Tests on Wood

Panel No.	Description of panel	Section of panel	Treatment	Condition after 4 months' exposure
60	Yellow Pine	Upper 2 Boards	Vermilion Paint, 1 coat	All okeh
		Lower 2 Boards	Lampblack, 1 coat	
		Right half of panel	Then coated with White Paint, 3 coats	
		Left half of panel	Coated with Spar Varnish, 3 coats	
61	Cypress	Upper 2 Boards	Vermilion Paint, 1 coat	All okeh
		Lower 2 Boards	Lampblack, 1 coat	
		Right half of panel	Then coated with White Paint, 3 coats	
		Left half of panel	Coated with Spar Varnish, 3 coats	
62	White Pine	Upper	Lampblack, 1 coat; then White Paint, 3 coats	Slight checking noted over lampblack
		Lower	Bare, then White Paint, 3 coats	

TABLE 72.

Series VIII—Special Tests

Panel No.	Description of panel	Section of panel	Treatment	Condition after 4 months' exposure
63	White Pine	1st Board	Red Paint, 3 coats	All okeh
		2nd Board	Green Paint, 3 coats	
		3rd Board	Yellow Paint, 3 coats	
		4th Board	Black Paint, 3 coats	
64	White Pine	Upper 2 Boards	Red Oxide Machine Ground, 3 coats	All okeh
		Lower 2 Boards	Red Oxide Hand Mixed, 3 coats	
65	White Pine	Upper 2 Boards	White Paint Pure Oil, 3 coats	All okeh except where birdshot have penetrated 2 upper boards
		Lower 2 Boards	White Paint Foots Oil, 3 coats	
66	White Pine	1st Board	White Paint B-T-X	All okeh
		2nd Board	Yellow Paint B-T-X	
		3rd Board	Gray Paint B-T-X	
		4th Board	Blue Paint B-T-X	
67	White Pine	1st Board	White Paint L-Z-X	All okeh
		2nd Board	Yellow Paint L-Z-X	
		3rd Board	Gray Paint L-Z-X	
		4th Board	Blue Paint L-Z-X	
68	White Pine	Upper 2 Boards	Spar Varnish, 5 coats	Disappeared from test rack
		Lower 2 Boards	Linseed Oil, 5 coats	

TABLE 73.
Additional Tests Exposed June 8, 1919

Panel No.	Description of panel	Section of panel	Treatment	Condition after 4 months' exposure
69	White Pine	1st Board	White Paint B-T-XX	All okeh
		2nd Board	Yellow Paint B-T-XX	
		3rd Board	Gray Paint B-T-XX	
		4th Board	Blue Paint B-T-XX	
70	Cypress	1st Board	White Paint M-L	All okeh
		2nd Board	Yellow Paint M-L	
		3rd Board	Gray Paint M-L	
		4th Board	Blue Paint M-L	
71	Cypress	1st Board	White Paint N-L	All okeh
		2nd Board	Yellow Paint N-L	
		3rd Board	Gray Paint N-L	
		4th Board	Blue Paint N-L	

TABLE 74.
Additional Metal Tests Exposed June 8, 1919

Panel No.	Description of panel	Section of panel	Treatment	Condition after 4 months' exposure
28	Steel Sheet (Parkerized)	Upper	Vermilion Paint, 3 coats	Okeh
		Lower	Bare	*
29	Steel Sheet (Parkerized)	Upper	Spar Varnish, 3 coats	Okeh
		Lower	Bare	*
30	Open Hearth Steel (Black Sheet)		1 coat Primer No. 1 over total surface, then 2 coats Finish No. 3 on upper diagonal half	Okeh
31	Open Hearth Steel (Black Sheet)		1 coat Primer No. 2 over total surface, then 2 coats Finish No. 4 on upper diagonal half	Okeh

*Very slight pin hole rust spots. Surface is rough and has assumed a light gray color.

Examination of Tests after One Year's Exposure.—The test panels exposed were again examined on May 17, 1920, after a period of exposure of one year. Some very interesting developments had taken place during that time. After the inspection the panels were rearranged for further exposure. The results of inspection are presented below.

Fig. 22

BACK OF PANEL No. 2

Test Illustrating Great Durability of Tin Plate Coated with Varnish. Lower Portion Showing Decay was Unprotected.

Fig. 23
BACK OF PANEL No. 6
Test Illustrating Great Durability of Tin Plate Coated with Varnish.
Lower Portion Showing Decay was Unprotected.

TABLE 75.

Series I—Tin Plate

Panel No.	Description of Panel	Section of Panel	Treatment	Condition after exposure for 1 year
1	E-X (Tin Coated)	Upper	Vermilion Paint, 3 coats	Okeh
		Lower	Bare	Very rusty
2	E-X (Tin Coated)	Upper	Spar varnish	Slight rust spot at center
		Lower	Bare	Very rusty
3	E-X (Tin Coated)	Upper	Collodion Dope D, 1 coat	Film scaled off. Some rust
		Lower	Bare	Very rusty
4	A-P 40 lb. (Heavy Tin Coated)	Upper	Vermilion Paint, 3 coats	Okeh
		Lower	Bare	Few small pin-hole rust spots
5	A-P 40 lb. (Heavy Tin Coated)	Upper	Spar Varnish, 3 coats	Okeh
		Lower	Bare	Few small pin-hole rust spots
6	A-P 40 lb. (Heavy Tin Coated)	Upper	Collodion Dope N, 1 coat	Film scaled off but no rust
		Lower	Bare	Few small pin-hole rust spots

One-half of the back surface of each panel from No. 1 to No. 6 was originally given one coat of spar varnish. Being protected from direct sunlight, the varnish stood up in most excellent condition, preserving the underlying surface of the panels from corrosion. The uncoated portion showed even greater corrosion than the upper sides which were exposed to sunlight. This is probably due to the constant presence of moisture on the unexposed surfaces. The moisture had, however, but little effect upon the spar varnish in the absence of direct sunlight, although only one coat was used as contrasted with three coats on the surfaces exposed to the sun. These tests indicate that sunlight is probably the most destructive factor in the decay of varnish films. They also indicate the great value of varnish in preserving tin plate or other metals from corrosion. Even the heaviest coated tin plate requires surface protection.

Fig. 24

Tests Illustrating Durability of Painted or Varnished Tin Plate. Lower Portions Showing Rust were Unprotected. See Series I.

TABLE 76.

Series II—Black Iron Metal Powder Tests

Panel No.	Description of Panel	Section of Panel	Treatment	Condition after exposure for 1 year
7	Open Hearth Steel (Black Sheet)	Upper	Aluminum Powder in Spar Varnish, 2 coats	Okeh
		Lower	Aluminum Powder in Linseed Oil, 2 coats	A few pin-hole rust spots
8	Open Hearth Steel (Black Sheet)	Upper	Aluminum Powder in Collodion Dope D, 2 coats	A few slight rust spots
		Lower	Bare	Very rusty
9	Open Hearth Steel (Black Sheet)	Upper	Zinc Powder in Spar Varnish, 2 coats	Okeh
		Lower	Zinc Powder in Linseed Oil, 2 coats	A few slight rust spots
10	Open Hearth Steel (Black Sheet)	Upper	Zinc Powder in Collodion Dope D, 2 coats	Scaled off in many spots
		Lower	Bare	Very rusty

These tests indicate that aluminum powder and zinc powder are both valuable metal protectives, but it is apparent that spar varnish or linseed oil are better liquids for exterior exposure than collodion dope, especially in the case of the zinc powder. The very much greater durability of collodion dope containing aluminum powder as compared to clear collodion dope on panel No. 3 or No. 6 should be given attention. The aluminum powder present in the dope has a direct effect in reflecting the sun's rays and thus protecting the dope film from decay.

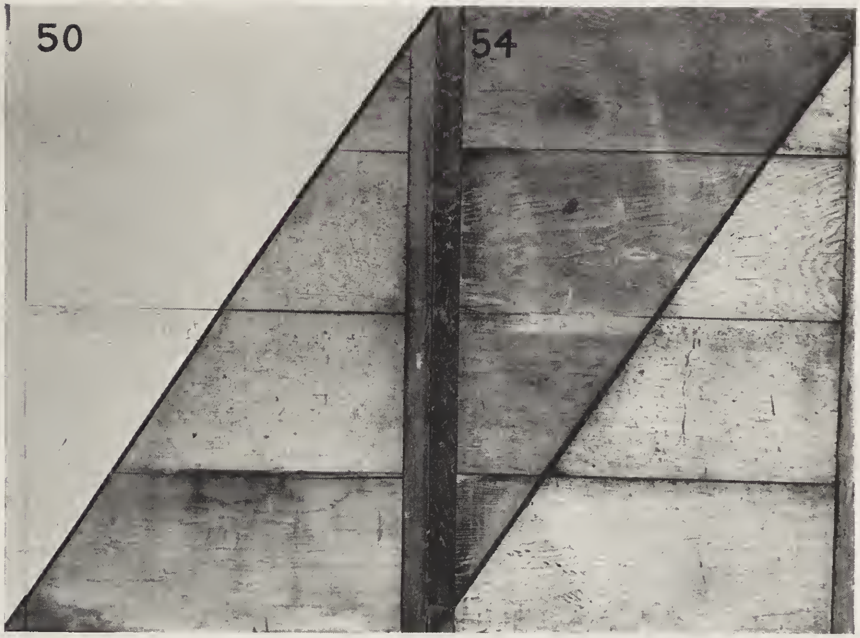


Fig. 25

Tests Illustrating Great Durability of Painted or Varnished Wood. Lower Portions Showing Decay were Unprotected. See Series V.

TABLE 77.

Series III—Special Metal Tests, Including Galvanized Sheets

Panel No.	Description of Panel	Section of Panel	Treatment	Condition after exposure for 1 year
11	Open Hearth Steel (Galvanized)	Upper	Spar Varnish, 3 coats	Film nearly gone
		Lower	Bare	Okeh
12	Open Hearth Steel (Galvanized)	Upper	Linseed Oil, 3 coats	Okeh
		Lower	Bare	Okeh
13	Open Hearth Steel (Galvanized)	Upper	Copper brushed, then vermilion paint, 3 coats	Film showing slight scaling
		Lower	Bare	Okeh
14	Open Hearth Steel (Black Sheet)	Upper	Vermilion paint, 3 coats	Okeh
		Lower	Bare	Very rusty
15	Copperized Steel (Galvanized)	Upper	Copper brushed, then vermilion paint, 3 coats	Film showing slight scaling
		Lower	Bare	Okeh
16	Copperized Steel (Black Sheet)	Upper	Vermilion paint, 3 coats	Okeh
		Lower	Bare	Very rusty
17	R-R Iron (Galvanized)	Upper	Copper brushed, then vermilion paint, 3 coats	Okeh
		Lower	Bare	Okeh
18	R-R Iron (Black Sheet)	Upper	Vermilion paint, 3 coats	Okeh
		Lower	Bare	Very rusty
19	R-R Iron (Heavy Tin Coating)	Upper	Vermilion paint, 3 coats	Okeh
		Lower	Bare	Badly spotted with rust

In this series of tests the value of a paint coating in protecting metal from corrosion is demonstrated. It is apparent that even the highest grades of carefully made black sheets cannot be exposed for even a short period without surface protection. Tests Nos. 11 and 12 show that linseed oil has a longer life than spar varnish, although the varnish is initially more water-resisting.

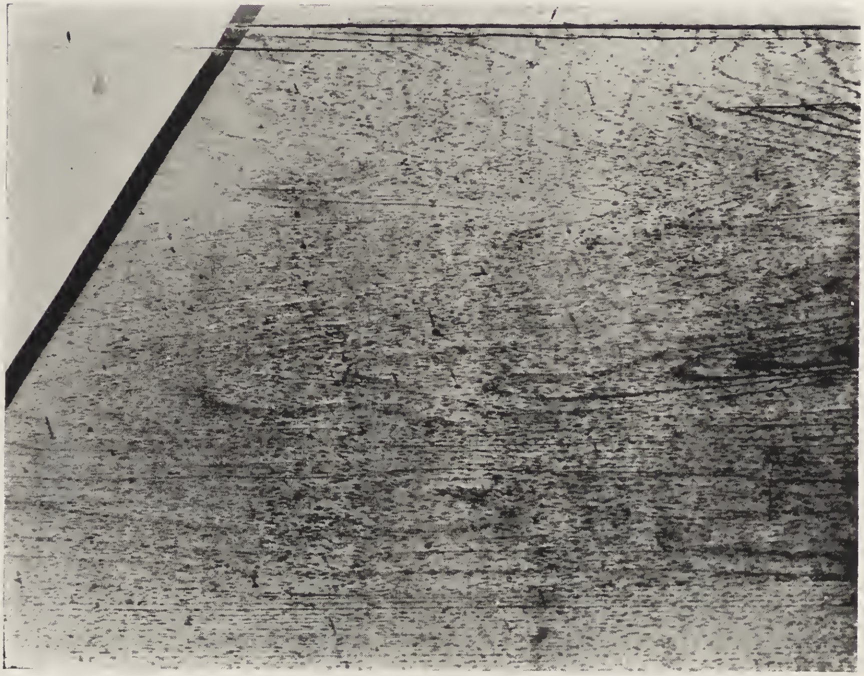


Fig. 26

Detail View of Unprotected Wood Panel. See Series V.

TABLE 78.

Series IV—War Paints on Metal

Panel No.	Description of Panel	Section of Panel	Treatment	Condition after exposure for 1 year
20	Open Hearth Steel (Black Sheet)	Upper	Spar Varnish (W. D. 6), 3 coats	Film disintegrated and rust developed
		Lower	Bare	Very rusty
21	Open Hearth Steel (Black Sheet)	Upper	Spar Varnish, 3 coats	Film disintegrated and rust developed
		Lower	Linseed Oil, 3 coats	Rusty below oil film but film is in good condition
22	Open Hearth Steel (Black Sheet)	Upper	Camouflage O. R. 333, 3 coats	Okeh
		Lower	Camouflage N. O. R. 334, 3 coats	Okeh
23	Open Hearth Steel (Black Sheet)	Upper	Yellow W. D. 37 and Olive Green W. D. 33 Projectile Paints, 3 coats	These four paints show very dull surfaces. Only a very few slight rust spots discernible
		Lower	Blue W. D. 34 and Black W. D. 38 Projectile Paints, 3 coats	
24	Open Hearth Steel (Black Sheet)	Upper	Brown Enamel Primer W. D. 13, 1 coat;	
		Lower	Red Oxide W. D. 11 2 coats	Okeh
25	Open Hearth Steel (Black Sheet)	Upper	Reinforced Red Lead Ready Mixed W. D. 40, 3 coats	Okeh but slightly dull
		Lower	Red Lead Hand Mixed, 3 coats	Okeh
26	Open Hearth Steel (Black Sheet)	Upper	Helmet Paint, 3 coats	Heavy checking
		Lower	Navy Camouflage, Gray, 3 coats	Okeh
27	Open Hearth Steel (Black Sheet)	Upper	Standard Olive Drab W. D. 28, 3 coats	Okeh
		Lower	Quick Drying Olive Drab W. D. 29, 3 coats	Few slight pin-hole rust spots

Test No. 23 indicates the value of grinding pigments in spar varnish to increase the durability of the coating. The light reflecting value of the pigment accomplishes the purpose of protecting the varnish film from decay.

Fig. 27

Detail View of Unprotected Wood Panel. See Series V.

TABLE 79.

Series V—Wood Panel Tests*

Panel No.	Description of Panel	Section of Panel	Treatment	Condition after exposure for 1 year
50	White Pine	Upper Lower	White Paint 3 coats Bare	Painted or varnished wood surfaces are in generally excellent condition. Uncoated wood surfaces are rough, pitted, weather-etched, and covered with spots of black fungus growth.
51	Yellow Pine	Upper Lower	White Paint, 3 coats Bare	
52	Cypress	Upper Lower	White Paint, 3 coats Bare	
53	Red Wood	Upper Lower	White Paint, 3 coats Bare	
54	White Pine	Upper Lower	Spar Varnish, 3 coats Bare	
55	Yellow Pine	Upper Lower	Spar Varnish, 3 coats Bare	
56	Cypress	Upper Lower	Spar Varnish, 3 coats Bare	
57	Red Wood	Upper Lower	Spar Varnish, 3 coats Bare	

TABLE 80.

Series VI—War Paints on Wood

Panel No.	Description of Panel	Section of Panel	Treatment	Condition after exposure for 1 year
58	Cypress	Entire Panel	Primed with Red Lead, then:	Okeh. Moderate chalking only
		Upper	Exterior Paint, Pearl Gray W. D. 50, 3 coats	
		Lower	Exterior Fire Resisting Paint, Gray Green W. D. 21, 3 coats	
59	Cypress	Entire Panel	Primed with Red Lead, then:	Okeh, except where bird shot have penetrated 2 upper boards Moderate chalking developed
		Upper Board 1	Anti-Actinic Wing Enamel Army Olive Drab, 3 coats	
		Upper Board 2	Anti-Actinic Wing Enamel Light Gray, 3 coats	
		Lower Boards 1 and 2	Anti-Actinic Wing Enamel Semi-Flat Navy Department Aero. Spec. C. & R. No. 3, 3 coats	

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*The coated portions of these 6 panels only, received after the inspection and photographic work on May 17, 1920, one additional coat of surface protection.

Fig. 28

Tests Illustrating Great Durability of Painted or Varnished Wood. Lower Portions Showing Decay were Unprotected. See Series V.

TABLE 81.

Series VII—Special Primer Tests on Wood

Panel No.	Description of Panel	Section of Panel	Treatment	Condition after exposure for 1 year
60	Yellow Pine	Upper 2 Boards Lower 2 Boards Right half of panel Left half of panel	Vermilion Paint, 1 coat Lampblack, 1 coat Then coated with White Paint, 3 coats Coated with Spar Varnish, 3 coats	White paint checking over black and red. Panel removed for laboratory study
61	Cypress	Upper 2 Boards Lower 2 Boards Right half of panel Left half of panel	Vermilion Paint, 1 coat Lampblack, 1 coat Then coated with White Paint, 3 coats Coated with Spar Varnish, 3 coats	Okeh
62	White Pine	Upper Lower	Lampblack, 1 coat; then White Paint, 3 coats Bare, then White Paint, 3 coats	Heavy checking noted over lampblack. Panel removed for laboratory study

TABLE 82.
Series VIII—Special Tests

Panel No.	Description of Panel	Section of Panel	Treatment	Condition after exposure for 1 year
63	White Pine	1st Board 2nd Board 3rd Board 4th Board	Red Paint, 3 coats Green Paint, 3 coats Yellow Paint, 3 coats Black Paint, 3 coats	Panel removed for laboratory study of thermal properties
64	White Pine	Upper 2 Boards Lower 2 Boards	Red Oxide Machine Ground, 3 coats Red Oxide Hand Mixed, 3 coats	Okeh
65	White Pine	Upper 2 Boards Lower 2 Boards	White Paint Pure Oil, 3 coats White Paint Foots Oil, 3 coats	Good condition except where birdshot have penetrated 2 upper boards. Panel removed for laboratory study
66	White Pine	1st Board 2nd Board 3rd Board 4th Board	White Paint B-T-X Yellow Paint B-T-X Gray Paint B-T-X Blue Paint B-T-X	Very slight checking and moderate chalking. Few slight scale spots at joints
67	White Pine	1st Board 2nd Board 3rd Board 4th Board	White Paint L-Z-X Yellow Paint L-Z-X Gray Paint L-Z-X Blue Paint L-Z-X	Very slight checking and moderate chalking. Few slight scale spots at joints
68	White Pine	Upper 2 Boards Lower 2 Boards	Spar Varnish, 5 coats Linseed Oil, 5 coats	Disappeared from test rack

TABLE 83.

Additional Tests Exposed June 8, 1919

Panel No.	Description of Panel	Section of Panel	Treatment	Condition after exposure for 11 mos.
69	White Pine	1st Board 2nd Board 3rd Board 4th Board	White Paint B-T-XX Yellow Paint B-T-XX Gray Paint B-T-XX Blue Paint B-T-XX	Panel broken from test rack
70	Cypress	1st Board 2nd Board 3rd Board 4th Board	White Paint M-L Yellow Paint M-L Gray Paint M-L Blue Paint M-L	Fairly good condition. Sloughing off at few spots. Considerable chalking. Tints show slight fading probably due to chalking
71	Cypress	1st Board 2nd Board 3rd Board 4th Board	White Paint N-L Yellow Paint N-L Gray Paint N-L Blue Paint N-L	Fairly good condition. Moderate chalking. Harder film and better tints than No. 70

TABLE 84.
Additional Metal Tests Exposed June 8, 1919

Panel No.	Description of Panel	Section of Panel	Treatment	Condition after exposure for 11 months
28	Steel Sheet (Parkerized)	Upper	Vermilion Paint, 3 coats	Okeh. Holding paint well
		Lower	Bare	Very rough surface and covered with small reddish brown rust spots
29	Steel Sheet (Parkerized)	Upper	Spar Varnish, 3 coats	Okeh. Holding varnish well.
		Lower	Bare	Very rough surface and covered with small reddish brown rust spots
30	Open Hearth Steel (Black Sheet)		1 coat Primer No. 1 over total surface, then 2 coats Finish No. 3 on upper diagonal half	Very few slight pinhole rust spots on primer but black painted surface is in excellent condition with no rust present
31	Open Hearth Steel (Black Sheet)		1 coat Primer No. 2 over total surface, then 2 coats Finish No. 4 on upper diagonal half	Disappeared from test rack

CHAPTER XV.

Production of Rosin Tung Esters (Tunga Resin)

In the Open Varnish Kettle and Their Application in Varnish Manufacture.

Esters of rosin, commonly known as ester gums, are widely used in the manufacture of varnishes. Although some varnish manufacturers may be producers of these esters, it is the practice of others to purchase them. In this paper there is presented an outline of a research that has resulted in the production of an ester-like resin of low acid value, suitable for the manufacture of highly water resisting and other types of varnishes. The fact that this resin, which for purposes of identification the author has called "Tunga Resin," may easily be produced in an open kettle will probably be of special interest to the varnish maker.

The usual process of making esters is to heat rosin under special conditions and in special apparatus, with from 10% to 18% of its weight of glycerin until a clear esterified resin results. It should be pointed out that while the aim of producers of ester gums may be to form the tri-glyceride of rosin, the mono- and di-glycerides may also result. The chart below indicates the theoretical amount of glycerine necessary to produce the various glycerides, assuming that rosin consists wholly of abietic acid.

Abietic Acid (Rosin)	Glycerin
$C_{20}H_{30}C_2$	$C_3H_5(OH)_3$
Molecular Weight 302	Molecular Weight 92

100 parts by weight require 30.5 parts by weight to form the monoglyceride
100 parts by weight require 15.25 parts by weight to form the diglyceride.
100 parts by weight require 10.17 parts by weight to form the triglyceride

A search of patent records referring to the production of ester gums and their use is very briefly presented below. In the various processes described in the patents referred to, the main purpose in some appears to be that of effecting the combination of glycerin and rosin under such conditions as will exclude contact with air, and that will eliminate the water formed during the process. This latter condition is referred to in most of the patents cited. Other modifications are stated, such as heating the rosin before combining it with the glycerin, presumably for the formation of rosin anhydride; distilling off the volatile or softer portion of the resin; and formation of compound esters of rosin and fossil resins. For the most part, the patents cover the manufacture of esters under pressure, although some describe the manufacture without pressure. Apparatus that will remove by suction the water formed by the esterification and provision for the presence of neutral gases which will exclude contact with air are outlined in others. Reference is also made to the advantages of using an excess of glycerin, indicating that better combination is thus effected. Some patents refer also to the addition of lime after the ester has been formed, in order to lower the acid value to a point below that formed by the ester. The combining temperatures referred to in the patents range from 280° C. to 300° C. for rosin-glycerin combinations, and up to 320° C. for the fossil resin combinations.

Class 1.

Esters Produced by a Combination of Rosin or Resin Acids with Glycerin.

U. S. Patent to Eugene Schaal, No. 335,485 of Feb. 2, 1886, *Manufacture of Resin Acid Ethers;* and reissued letters patent to Eugene Schaal, No. 10,823, dated March 29, 1887 (original No. 335,485, dated Feb. 2, 1886), *Manufacture of Resin Acid Ethers.* Rosin and other acid resins are distilled in vacuo to drive off volatile and soft constituents. Glycerin or other alcohols are added, and heating is continued with or without pressure. Water is eliminated and esters are produced. These may be subjected to distillation to separate them into soft or hard resinous products. Acid or acid salts favoring the above reactions may be employed in the process.

U. S. Patent to Eugene Schaal, No. 501,446, of July 11, 1893, *Manufacture of Resin Acid Esters.* Discusses the art of making ester-like products from acid resins such as copal, anime, and similar resins, and rosin, by combination with alcohols or hydroxyl derivatives, such as glycerin, resorcin, mannite, phenol, cresols, and sugars. Combination is effected under pressure in an apparatus that will allow elimination by suction of the aqueous vapors produced during the formation of the esters or ester-like products. The alcohols eliminated are replaced by the introduction of a fresh supply, until the formation of the ester or ester-like product is completed.

U. S. Patent to C. Ellis and Louis Rabinowitz, No. 1,226,-760, of May 22, 1917, entitled *Process of Making Varnish Bases.* Discusses the formation of resin anhydrides and combination thereof with glycerin to form esterified resins. The use of various gases favorable to the reaction is claimed.

U. S. Patent to Henry Terrisse, No. 1,236,996, of Aug. 14, 1917, entitled *Esterified Fossil Resin and Process of Making Same.* Discusses the art of making glycerin esters from acid fossil resins. Process consists of first depolymerizing or solubilizing the fossil resin, preferably by heating under pressure in an autoclave, with or without the addition of naphthalene or phenol; then combining this depolymerized resin with glycerin or other high boiling alcohol with or without pressure and to such temperatures as will effect combination.

U. S. Patent to C. Ellis No. 1,242,161, of October 9, 1917, entitled *Process of Making Varnish Material.* The process refers to the esterification of rosin and other acid resins such as copal with glycerin or similar materials in the presence of finely divided aluminum or aluminum salts. Resins may first be "cracked" to render them soluble in oil. After esterification, the ester may also be "cracked."

CLASS 2.

Patents Referring to the Esterification of Acids Other Than Rosin or Hard Resin Acids.

U. S. Patent to William C. Arsem, No. 1,098,776, of June 2, 1914, *Resinous Condensation Products and Process of Making the Same.* Relates to the esterification of organic acids particularly the polybasic acids, with glycerol and other polyhydric alcohols. Such organic acids as thallic acid and succinic acid are referred to. Explains the combination of glycerin with these acids, in essentially molecular proportions. Further refers to the combination of the esters of thallic acid with rosin.

U. S. Patent to Edward S. Dawson, Jr., No. 1,085,112, of Jan. 27, 1914, *A Resinous Condensation Product.* Relates to the production of a composition suitable as an impregnating varnish or insulating material, by producing a glycerin or similar ester with citric acid. Descriptive formula is given, showing method of forming ester of citric acid and glycerin in the presence of a flexibilizing agent such as meta-dinitrobenzol, ethyl benzoate, acetanilid or beta-napthol.

U. S. Patent to Reinhold Fruter, No. 1,195,673, of Aug. 22, 1916, entitled *Varnish, Lacquer or the Like, and Process of Making Same.* Relates to a process of making varnishes or lacquers by combining nitrocellulose with esters of lactic acid in conjunction with hydrocarbons. Patent indicates that lactic acid esters are excellent solvents for the organic cellulose esters and for various resins such as rosin, shellac, copal and sandarac, all of which may be used in certain of the processes.

CLASS 3.

Patents Relating to Products Which Include the Use of Rosin-Glycerin Esters In Their Manufacture, But Do Not Include or Refer to the Production of These Esters.

U. S. Patent to William Painter, No. 684,521, of Oct. 15, 1901, entitled *Composition of Matter.* Relates to a composition of matter consisting of glue or gelatine mixed with water-insoluble substances such as waxy, resinous, oily or pitchy substances. Illustrative formula stipulates the use of ester-gum and vaseline as the water-insoluble materials. The addition of glycerin is also referred to as an added ingredient.

U. E. Patent to C. Ellis, No. 1,189,550, of July 4, 1916, entitled *Coating Composition.* A coating compound for cement surfaces, consisting essentially of unsaponifiable resinous bodies among which are resin esters.

U. S. Patent to C. Ellis No. 1,189,551, of July 4, 1916, entitled *Treating Concrete.* A coating compound for cement surfaces, in which resin esters may be used as an ingredient.

U. S. Patent to Charles A. Hollenbeck and Aaron C. Horn, No. 1,190,765, of July 11, 1916, entitled *Waterproof Varnish Composition and Process of Making Same.* Hard varnish resins, including rosin esters, may be melted and heated to render soluble in a varnish liquid. After cooling they may be remelted and incorporated with sulfured Chinese wood oil, a drier and thinner.

U. S. Patent to Henri Terrisse, No. 1,214,611, of Feb. 6, 1917, entitled *Manufacture of Varnishes, Paints and Other Protective or Coating Preparations.* Relates to the production of a liquid suitable for use as a varnish or paint liquid, made by combining esterified Congo copal prepared in accordance with specification Serial No. 23,164, filed April 22, 1915, U. S. Patent No. 1,236,996, dated Aug. 14, 1917 (referred to in Class 1) with suitable drying oils and driers and thinning with suitable volatile thinner.

Characteristics of Marketed Esters.—As is well known, commercial rosin has an acid value usually approaching 150 and is readily dissolved by ethyl alcohol, while rosin that is well esterified will have an acid value of not over 20, and will be very resistant to the action of alcohol. Those of low acid value are preferred as they make substantially neutral varnishes that have much less tendency to thicken with basic pigments than varnishes of high acid value.* Unfortunately, however, some products marketed as ester gums, apparently consist almost wholly of uncombined rosin. The laboratory examination of six samples recently obtained is of interest in this connection:

TABLE 85.
COMMERCIAL ESTERS.

No.	Brand	Acid Numbers		Color	Action of Alcohol
		In Alcohol	In Alcohol and Benzol		
1		161.2	158.9	Very pale	Dissolves readily and completely in cold.
2		100.8	119.6	Light in color. Opaque.	Dissolves readily and completely in cold. Solution is milky. (Colloidal solution.)
3		13.8	27.4	Medium light color. Opaque.	Partially dissolved by alcohol. Solution dark in color.
4		16.4	22.3	Medium light color.	Unaffected by alcohol.
5		12.9	17.2	Medium light color.	Unaffected by alcohol.
6		5.	8.18	Very pale color. Slightly cloudy.	Softened to a considerable extent by alcohol but not dissolved.

The light color of all of the above samples indicates that they were made in the absence of air, probably in vacuo. When those of low acid value were heated to 475° F., they

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*Thickening of enamel paint may often be due to the use of varnishes of high acid value. Varnishes of low acid value but having the incorrect degree of colloidal viscosity may be so adsorbed by the pigments as to cause similar phenomena.

became very dark. All of those of high acid value are readily affected by alcohol. Those of low acid value are practically unaffected by alcohol with the exception of No. 6. Lime was found present in some of those of low acid value. When fragments of the samples were placed on a table and subjected to the pressure of the thumb, most of them broke down to a very fine, fluffy powder that was markedly less crystalline than rosin treated in a similar manner.

Experimental.—The fact that in the presence of tung oil a combination of glycerin and rosin could be satisfactorily effected in an open kettle without special apparatus, has for some time been known by the writer and his assistants.* A series of experiments just completed has indicated that when a mixture of rosin and glycerin is heated together in the presence of tung oil, possible combination may occur either of the glycerin with the rosin or of the glycerin with the tung oil. The character of the resulting product also indicates whether the glycerin first combines with the rosin or with the tung oil, or whether a combination of these two is simultaneously effected. The experiments also indicated that the presence of lime or lime soaps greatly accelerates the reactions. The more important of the experiments, together with some tests indicating the effect of lime upon rosin, are presented below. The experiments showing the methods of producing the low acid value tung esters are followed by experiments showing how the reactions involved may be applied to the production of three different classes of varnish.

In the experiments reported, the losses during heating were determined by weighing the material used and reweighing the material after cooking. The difference was considered as fume loss. In Experiments 12 and 14 this loss was calculated only on the amount of rosin used. When lime was present in any of the experiments, a deduction from the total loss, in amount equal to the hy-

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*Acknowledgment is made to the contributing work of Mr. Max Y. Seaton.

droxyl group in the lime, was made. When glycerin was used in any of the tests, a similar deduction for the hydroxyl group in the glycerin was made from the total loss. When lime and glycerin were used in any series, similar deductions were made.

EXPERIMENT NO. 12.

Test made to determine the loss in weight when Rosin is raised to certain temperatures and whether the acid value of the remaining product is higher or lower than the original Rosin.

The Rosin (W. G. grade) was heated in 15 minutes to 300° F. and held at that temperature for 15 minutes. It was then raised in 20 minutes to 475° F. and held at that temperature for 1 hour.

Resulting product dark brown.

Fume loss 21%.

Acid value (in alcohol) of original rosin	*158.5*
Acid value (in alcohol) of resulting heated rosin	*155.6*

The results indicate that the volatile portion was of about the same acidity as the non-volatile residue.

EXPERIMENT NO. 14.

Test made to determine the loss in weight on heating Rosin and the acid value of resulting product.

The Rosin was heated in 9 minutes to 350° F. and held at that temperature for 15 minutes. It was then raised in 11 minutes to 525° F. It was then allowed to drop in 5 minutes to 475° F. and held at that temperature for 45 minutes.

Resulting product dark brown.
Fume loss 19%.

Acid value (in alcohol) of original rosin	*158.5*
Acid value (in alcohol) of resulting heated rosin	*156.5*

The results indicate that the acid value of the rosin was not materially decreased by this heating.

EXPERIMENT NO. 18.

Test made to determine effect of Lime on the fume loss and acid value of Rosin heated to certain temperatures.

The Rosin was heated in 5 minutes to 350° F. 6% of lime was added and the mass raised and held at 500° F. for 15 minutes.

Resulting product brown in color. Fairly hard.
Fume loss (less water from lime) 7%.

Acid value (in alcohol) of original rosin	*158.5*
Acid value (in alcohol-benzol) of resulting heated-limed rosin	*78.5*

The results indicate lower fume loss and lower acid value when rosin is limed. The results check closely with factory practice on a large scale.

EXPERIMENT NO. 22.

Test made to determine if Glycerin will combine with lime hardened rosin in an open kettle. The limed Rosin produced in Experiment No. 18 was used.

The limed Rosin was heated in 20 minutes to 350° F. 12% of Glycerin was added. The mass was then raised in 20 minutes to 440° F. at which temperature it became clear. It was then raised in 10 minutes to 500° F.

Resulting product dark brown.
Fume loss (less water in glycerin) 5.6%.

Acid value (in alcohol-benzol) of original limed rosin	*78.5*
Acid value (in alcohol-benzol) of resulting product	*61.*

The results indicate that some combination occurred, the acid value being decreased considerably.

EXPERIMENT NO. 24.

Test made to determine whether Rosin and Glycerin combine under certain conditions in the absence of other ingredients.

The rosin was heated in 20 minutes to 350° F. when it became clear.

15% of glycerin was added and the mass held at that temperature for 10 minutes.

The mass was then raised in 30 minutes to 430°, when it became clear.

The mass was then raised in 5 minutes to 475° F. and held at that temperature for 45 minutes.

Resulting product dark brown.
Fume loss (rosin and glycerin) 27%.

Acid value (in alcohol) of original Rosin	*158.5*
Acid value (in alcohol) of resulting product	*141.6*

The results indicate that some combination was effected.

Experiment No. 25.

Test made to determine the influence of a small amount of Tung Oil in accelerating the combination of the Rosin and Glycerin.

The Rosin with 5% of Tung Oil was heated in 10 minutes to 300° F. 15% of glycerin was added. The mass was then held at 300° F. for 15 minutes.

The mass was raised in 15 minutes to 460° F. It became clear.

The mass was raised in 10 minutes to 475° F. and held at that temperature for 45 minutes.

Resulting product dark brown.
Fume loss (Rosin and Glycerin) 22.5%.

Acid value (in alcohol) of original Rosin	*158.5*
Acid value (in alcohol) of resulting product	*123.4*

Comparison with Experiment 24 shows that the presence of Tung Oil in Experiment 25 stimulated the formation of the ester.

Experiment No. 26.

Test made to determine the influence of a small amount of Tung Oil in accelerating the combination of lime hardened Rosin and Gylcerin.

The rosin was heated in 10 minutes to 350° F.

6% of Lime was added.

The mass was raised in 10 minutes to 500° F.

10% of Tung Oil was added.

The mass was cooled to 350° F. and 12% of Glycerin was added.

It was raised to 475° F. and held at that temperature for 30 minutes. Only slight fume was noted.

Resulting product brown.
Fume loss 12.6%.

Acid value (in alcohol) of original rosin	*158.5*
Acid value (in alcohol-benzol) of resulting product	*52.1*

It is apparent that the tung oil has to some extent effected ester formation.

EXPERIMENT No. 27.

Test to determine whether further heating of the product made in Experiment 26 would lower the acid value.

A portion of the resulting product produced in Experiment 26 was heated to 500° F. for 3 hours.

Color dark brown.	
Fume loss 11%	
Acid value (in alcohol-benzol) of product from Experiment No. 26	*52.1*
Acid value (in alcohol-benzol) of resulting product	*18.9*

Apparently some free resin acids are volatilized and indications are that further ester formation occurred, lowering the acid value.

EXPERIMENT No. 44.

Test made to determine the effect of a larger amount of Tung Oil on the esterification of Rosin and Glycerin.

The rosin with approximately 40% Tung Oil was heated in 10 minutes to 500° F.

The mass was cooled in 10 minutes to 350° F. and 15% of Glycerin was added.

The mass was then heated to 430° F. when it became clear.

The mass was raised in 7 minutes to 475° F. and held at that temperature for 45 minutes.

Color brown.	
Fume loss 15.1%	
Acid value (in alcohol-benzol) of rosin and tung oil before addition of glycerin	*82.9*
Acid value (in alcohol-benzol) of rosin and tung oil and glycerin mixture	*77.9*
Acid value (in alcohol-benzol) of final product after cooking	*70.4*

The product was hard and very tough.

EXPERIMENT NO 45.

(Tunga Resin.)

Test made to determine the effect of adding lime during the process of combining Rosin and Glycerin in the presence of a moderate amount of Tung Oil, and maintaining a high temperature for a considerable period.

The Rosin was heated with 15% of Tung Oil in 10 minutes to 350° F.

6% of Lime was added and the mass heated rapidly to 500° F. It was then cooled in 15 minutes to 350° F.

20% of Glycerin was added.

The mass was slowly raised in 1 hour to 500° F. and held at that temperature for 2 hours.

Fume loss 20.4%.

Color dark brown.

Acid value (in alcohol) of resulting product	*7.8*
Acid value (in alcohol-benzol) of resulting product	*16.*

The resulting product was very hard.

EXPERIMENT NO. 46.

(Tunga Resin.)

Test made to determine the effect of adding Lime during the process of combining Rosin and Glycerin in the presence of a considerable amount of Tung Oil and maintaining a high temperature for a limited period.

The rosin with 40% tung oil was heated in 20 minutes to 350° F.

6% of lime was added.

The mass was raised in 12 minutes to 500° F. and held at that temperature for 7 minutes.

The mass was cooled to 350° F. and 15% of glycerin was added.

The mass was then heated in 20 minutes to 430° F. when it became clear.

The mass was raised in 10 minutes to 475° F. and held at that temperature for 45 minutes.

Brown color.

Fume loss 15%.

Acid value (in alcohol) of resulting product	*20.4*
Acid value (in alcohol-benzol) of resulting product	*24.1*

The above test indicates that glycerin combines more readily with rosin in the presence of Tung Oil if Lime is added and soaps then formed. Lime Tungates and Lime Resinates formed stimulated the ester formation

Part of the resulting product from Experiment 46 was heated to 500° F. for 1 hour. It became lighter in color. It had an acid value (in alcohol) of 19 and (in alcohol-benzol) of 19.6

EXPERIMENT No. *48.*

(Tunga Resin.)

Test made to determine the effect of adding previously made Lime Soaps such as Calcium Tungate, upon the combination of Rosin and Glycerin in the presence of Tung Oil.

Heated 80 parts of Tung Oil in 10 minutes to 350° F.

Added 12 parts of Lime and held at 350° for 20 minutes.

Flocculent Lime Tungate was formed and rose to the surface, later subsiding.

Added 200 parts of Rosin.

Raised temperature in 30 minutes to 425° F. The soaps were dissolved.

Dropped temperature to 350°.

Added 30 parts of Glycerin.

Raised temperature in 20 minutes to 450° F. when it became clear.

Raised temperature in 7 minutes to 500° F.

Cooled mass to 475° and held at that temperature for 45 minutes.

Color brown.

Fume loss 8.3%.

Acid value (in alcohol) of resulting product	*21.8*
Acid value (in alcohol-benzol) of resulting product	*24.4*

Since the lime present in the above test was fully combined with the tung oil, it is probable that it could not have had any neutralizing effect upon the rosin. The low acid value of the product must therefore be attributed to glycerin ester formation with the rosin. The presence of lime tungate undoubtedly stimulated this esterification.

The resin produced in this experiment was hard and superior in many of its properties to most commercial esters.

Comparison of Commercial Esters and Prepared Tung Oil Rosin Esters (Tunga Resin.)

Color.—The resulting products made by the methods described above were considerably darker in color than the esters described on page 218 (Table 85). It should be noted, however, that the commercial esters, although initially light in color, became dark brown when heated to 450° F.

Powdering.—The low acid number esters produced (Tunga Resin) powdered in a manner similar to commercial esters, but showed greater toughness.

Clarity.—The prepared esters (Tunga Resin) were very clear and did not have the cloudy appearance of some commercial esters. When the commercial esters of cloudy appearance were heated, they became clearer and remained so on cooling. This would indicate that in the commercial esters there is some uncombined glycerin or perhaps occluded water.

Effect of Alcohol.—The effect of alcohol on the prepared esters (Tunga Resin) was similar to that on the commercial esters. Those having a high acid value were affected, while those having a low acid value were practically unaffected. One sample remained in alcohol for one month without showing apparent effect.

Observations Made During Tests.

In the various experiments outlined above, the glycerin was usually added at about 350° F. and the temperature then gradually raised. When the glycerin was added to the melted rosin or rosin and tung oil, the mixture became milky and opaque when cooled on glass. This indicated an emulsion. The condition referred to persisted until the mass was heated for a considerable period and preferably to 430° F. or over, when it cleared up. This point indicated that the glycerin was then combined. In the experiments in which but little combination or esterification was effected, the change from a cloudy to a clear condition was rapid. The mixtures containing tung oil, especially in the presence of lime, cleared gradually and progressively, the final end point being sharp. The mixtures containing tung oil, especially in the presence of lime, cleared gradually and progressively, the final end point being sharp.

Very small amounts of fume were given off at temperatures below 410° F. Between 410° and 430° white fumes were given off in most instances. In one case a portion of the fumes was collected and found to contain glycerin. In those experiments in which esterification was slight, as shown by the high acid values of the resulting products, a larger amount of fume was given off. In those experiments where esterification was marked, only a relatively small amount of fume was observed. As the percentage of tung oil was increased, the amount of fume was apparently decreased and the color of the product better maintained.

In the experiments containing tung oil, especially those having up to 40% of the amount of rosin present, a head or froth formed over the top during the heating process. This phenomenon, which would minimize the effect of air oxidation, is no doubt responsible to some extent for the low loss and somewhat light color of the product.

It will be noted that the acid value of the products showing high acidity was usually determined by the alcohol method. This method is perhaps best with such products since a large amount of alkali is required and the water present would make an emulsion if benzol were added. A sharp end point might thus be prevented.

Results Indicated by Tests.

(1). When rosin is heated with glycerin and tung oil, the combination effected is either one between the glycerin and the rosin to form a rosin ester or between the glycerin and the tung oil to form a corresponding amount of di- or mono-glycerides of tung oil.

(2). Theoretically rosin requires approximately 10% of glycerin to form a neutral tri-glyceride, assuming that there is no loss of glycerin during the heating process and that no glyceride other than the tri-glyceride is formed. It is probable, however, that during the process of heating, the mono- and di-glycerides are formed together with the tri-glycerides. Moreover, it is apparent that some loss of glycerin invariably occurs, and it is therefore advisable to have present an excess of glycerin in order to form a neutral ester.

(3). The combination is greatly stimulated by the presence of lime soaps.

(4). The effect of the lime when present as a resinate or as a tungate, or a mixture of these, is similar but apparently much more rapid when present as a tungate. In the absence of lime soaps, the combination of the glycerin tends to be initially with the tung oil rather than with the rosin.

(5). In the presence of lime soaps the glycerin tends to combine largely with the rosin rather than with the tung oil.

(6). The activity of the Tung Oil in causing combination between rosin and glycerin may be due to the emulsifying properties of polymerized Tung Oil. A well made emulsion would show a greater dispersion of the glycerin and cause more rapid combination than when no emulsion was present.

(7). It is apparent that the process as evolved (experiments 45-46-48) can be practically applied to the manufacture of a low acid rosin ester product (Tunga Resin). Ordinary open varnish kettles may be employed without provision for pressure, vacuum or similar arrangements. The best temperature to employ is not less than 475° F., and maintenance of that temperature for from 30 to 60 minutes.

(8). With increase in tung oil content, decrease in fume loss is observed. The color of the resulting product is also better maintained.

(9). The speed of reaction is greatly affected by the quality of lime used and the care with which it is incorporated. Some types of lime are not reactive. Freshly made lime that may easily be dissolved by melted rosin should be selected. It should be finely divided and must be sprinkled in slowly and thoroughly stirred.

(10). Esters produced by carrying on the process in vacuo with special apparatus for the removal of water, or in the presence of inert gases, may be light in color. These products, however, when heated up to the temperatures used in the manufacture of varnish, may darken immediately. This would indicate that for cooked varnishes, esters produced without the special precautions and apparatus noted above would be quite as suitable.

(11). During the process of ester formation, various materials present undoubtedly act as catalyzers. The lime soaps of rosin, or of tung oil, as well as uncombined tung oil have been found by the writer to be excellent catalysts. They also enter into reactions that produce products of great toughness not possessed by ordinary ester gums.

Application of the Process to the Manufacture of Varnishes.

The production of waterproof varnishes in accordance with three procedures suggested by the above tests are briefly outlined below. They are merely given as representative procedure.

It is apparent that when using the esterified rosin product referred to above in the manufacture of varnishes, the best results will be obtained when there is used a fairly large percentage of tung oil during the processing. It should be borne in mind, however, that the temperature and length of time of heating required to effect thorough esterification is higher and of longer duration than that usually used for treating tung oil varnishes. This would suggest that the preferred procedure would be to add simply enough tung oil to effect combination during the early stages of cooking. The balance and major portion of the tung oil may be added at the end of the cooking. With this procedure and utilizing the information developed from the above recorded experiments, three different varnishes were prepared. The materials used in these varnishes and the methods of manufacture are presented below. It will be noted that some turpentine in combination with mineral spirits is specified as a thinner. During the thinning process the turpentine should first be added in order to effect a solution of the base in the turpentine and diffuse it and maintain the colloidal viscosity. The mineral spirits should then subsequently be added as usual.

Waterproof Spar Varnish.

Materials (Parts by Weight).

Rosin	100	parts
Tung Oil	240	"
Lime	6	"
Glycerin	20	"
Lead Tungate Paste Drier	8	"
Manganese Tungate Paste Drier	14	"
Turpentine	66	"
Mineral Spirits	300	"

Method.

Melt the rosin with 25 parts of tung oil at 350° F. Add the lime and stir thoroughly. Raise the temperature to 500° F. to take up the lime and to partially polymerize the Tung Oil. Drop heat to 350° F. and add glycerin. Raise heat slowly to 500° F. and hold one hour. Add balance of Tung Oil slowly, keeping temperature of mass 450° or above, during addition. When adding the balance of the tung oil, care should be taken that it is not added too quickly and the temperature of the mass allowed to drop very much below 450° F. as the ester already formed is liable to be thrown out of solution and is only brought back again with difficulty. When all the tung oil is added raise temperature to 450° F. Add driers. Hold at 450° F. for proper body. This may require from 30 minutes to one hour. The pill should be stiff but not gelatinous or stringy. Cool and thin to 350° F. to 400° F. If cooked too heavy, more thinner than specified above will be required. The turpentine might be largely replaced by an additional amount of mineral spirits.

Properties of Varnish.

Acid value (in alcohol-benzol) 5.37.

Dries hard in 5 hours.

Coated panel in boiling water for 15 minutes shows no whitening or softening of film.

Flexible.

Long Oil Varnish.

Materials (Parts by Weight).

Rosin	100	parts
Tung Oil	400	"
Lime	8	"
Glycerin	25	"
Manganese Tungate Drier	20	"
Lead Tungate Drier	12	"
Turpentine	125	"
Mineral Spirits	450	"

Add lime to cold tung oil. Heat mixture to 350° F. Hold until soaps form and then settle to bottom of kettle. Oil layer will be clear. Add rosin and raise temperature to about 400° F. Soaps will dissolve. Drop to 350° F. Add glycerin. Raise to 475° F. and hold for 45 minutes. Drop to 450° F. and add driers. Hold at 450° F. until proper body is obtained. This will usually require from 15 to 30 minutes.

Properties of Varnish.

Acid value (in alcohol-benzol) 5.74.

Dries hard in 10 hours.

Coated panel in boiling water for 15 minutes shows only very slight whitening and no softening of film.

Flexible.

Short Oil Varnish.

Materials (Parts by Weight).

Rosin	100	parts
Tung Oil	160	"
Glycerin	55	"
Manganese Tungate Drier	10	"
Lead Tungate Drier	6	"
Turpentine	50	"
Mineral Spirits	220	"

Melt rosin and tung oil together at 350° F. Add glycerin. Raise temperature to 475° F. and hold for 45 minutes. Drop to 450° F. Add driers and hold at 450° F. until proper body is obtained. This will usually require 45 minutes.

Properties of Varnish

Acid value (in alcohol-benzol) 16.8.

Dries hard in 10 hours.

Coated panel in boiling water for 15 minutes shows only very slight whitening and no softening of film.

Flexible.

As outlined above, the esterification of rosin with glycerin may be carried out as a process in itself, resulting in the formation of the so-called Tunga Varnish Resin, or the esterification may be brought about during the actual varnish-making process, in which case the product is the

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Note.—The acid values of a number of commercial waterproof spar varnishes recently examined at the Bureau of Standards ranged from 5 to 26.

finished varnish. It is the purpose in the following paragraphs to discuss the resin, its physical characteristics and chemical properties, and the quality of the varnishes prepared directly from it.

In the various batches of Tunga Resin recently made, analytical values differed slightly, probably due to slight differences in manipulation, temperature relations, etc. The greatest variation occurs in the acid value, but even here it varies between rather narrow limits (considering the acid value of rosin), usually being less than 20. Upon examination of an average sample the following results were obtained:

Color: Dark reddish brown.
Specific Gravity: 1.132.
Melting Point (capillary tube): 105° C.—125° C.
Acid Value (in alcohol-benzol): 16.2.
Saponification Number: 71.59.
Solubility in Alcohol: Unaffected in cold. Slightly softened by boiling.
“ “ Benzol: Slightly soluble in cold. Entirely soluble on boiling.
Solubility in Turpentine: Very slightly in cold. Entirely soluble on boiling.
“ “ Amyl Alcohol: Insoluble in cold. Slowly breaks down on boiling to a light suspension but does not dissolve.
“ “ Acetone: Slowly broken down in cold to a light suspension. On boiling, the same condition is produced more rapidly.
“ “ Benzine: Very slightly softened in cold. Broken down to a suspension on heating but does not dissolve.

In employing the resin as a varnish gum, various combinations with oils were tried out with the idea of determining its applicability to the making of various types of varnishes. As indicated in the first part of this chapter, long oil, short oil, and spar varnishes can be made by carrying out the esterification during the varnish-making process. The same types can be prepared by combinations of the made resin with oils. For example, a waterproof varnish corresponding closely to the spar varnish described on page — was made according to the following formula:

	Parts by weight
Tunga Resin	100
Tung Oil	225
Manganese Tungate Paste Drier	20
Turpentine	80
Mineral Spirits	300

The Tunga Resin and tung oil were gradually heated in one hour to 500° F. The temperature was dropped to 450° F. and the drier added. The mass was then again brought to 500° F. and held at that temperature for 20 minutes to bring to the desired body. It was allowed to cool to 350° F. and then thinned, the turpentine being added first.

Physical Properties of the Varnish.

Brushes and levels well.
Becomes tacky in 20 minutes.
Sets to touch in 3½ hours.
Dries tack-free in 5 hours.
Dries hard in 7 hours.

When a coated panel is placed in boiling water for 15 minutes, it shows no whitening or softening of film. Film is flexible.

Satisfactory varnishes can be prepared, using linseed oil in place of tung oil. The heating must, however, be continued at least three times as long. A smaller amount of thinner is required than when tung oil is used, and the varnish dries much more slowly. Varnishes made from linseed oil and Tunga Resin are tough and flexible and fairly resistant to water.

When mixtures of tung and linseed are used, a greater quantity of turpentine is required on account of the increased collodial viscosity produced by tung oil. The time of cooking decreases with increase in proportion of tung oil. The varnishes become more resistant to water as the quantity of tung oil is increased.

In connection with an investigation of the properties of perilla oil being carried on at the present time, a number of varnishes have been made, using Tunga Resin. While it has not as yet been possible to make varnishes from this oil as easily as with tung and linseed oil, still the results thus far indicate that with proper manipulation, products with certain desirable characteristics can be prepared.

In the formulas described below, 20 parts manganese tungate paste drier was used in each case and the method of making was the same as for the tung oil varnish given above:

TABLE 86.

	Tunga Resin	Oil		Thinners		Final Heating for body
			Parts		Parts	
No. 1	100 parts	Perilla	225	Turpentine Min'l Spts.	40 110	2 hours
No. 2	200 "	"	225	Turpentine Min'l Spts.	40 180	1¾ "
No. 3	100 "	Tung Perilla	112½ 112½	Turpentine Min'l Spts.	40 260	1½ "
No. 4	100 "	Linseed Perilla	112½ 112½	Turpentine Min'l Spts.	40 150	1¾ "

Physical Characteristics of Perilla Varnishes.

Despite the high iodine number of perilla oil, all these varnishes with the exception of No. 3 dried rather slowly. It would appear that perilla oil prevents to some extent the polymerization and bodying of other oils and lengthens the time of cooking. Its presence markedly increases the gloss of the varnish and causes it to dry to a heavier film. Its rather slow drying and high lustre might make it useful as a finishing varnish for certain classes of automobile work. Although these varnishes remain quite thin and do not congeal or gelatinize on cooling, considerable quantities of thinner are required, considering their viscosity. Usually, the higher the percentage of perilla used, the lower the quantity of thinner that is required.

CHAPTER XVI.

Bulking Values and Yields of Pigments and Liquids Used in Paint and Enamel Manufacture

Scope of Work.—The bulk or gallonage of paint that may be produced from any pigment, is inversely proportional to the specific gravity of the pigment as determined in an air-free condition. With specific gravity figures available, it is possible for the paint grinder to rapidly calculate the yield of any specified formula.* The object of this paper, therefore, is to place such data before the grinders. The work was undertaken at the request of the Cost Accounting Committee of the Paint Manufacturers' Association of the United States, as will be noted from the following extract of the report of that Committee, made at the Chicago meeting in October, 1919:

> "As a preliminary to the proper determination of a paint yield it is necessary that two charts be formulated. First, a chart which will give the paint manufacturer the correct bulk in gallons per hundred pounds of each of his pigments, and, second, a chart showing the percentage of loss in manufacturing a batch of paint, this loss being computed on a basis, first, of the size of the batch, and, second, of the percentage of volatile material present.
>
> "Whenever in this connection the bulk of a pigment is mentioned it refers to the bulk occupied by the pigment after it has been made into a paint which is naturally considerably less than the dry bulk. The easiest method for determining the bulk of a pigment is from its specific gravity.
>
> "The paint manufacturer can secure the specific gravity of a pigment in one of three ways. First, by requesting it from the manufacturer of the pigment; second, by having the determination made in his own laboratory (showing a commonly used method); third, if his own laboratory is inadequate or not equipped with

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*See Circular 53. Educational Bureau P. M. A.

> the proper apparatus the gravity may be secured by sending a sample to a good commercial chemist.
>
> "After the specific gravity has been determined it is not difficult to compute the bulk per hundred pounds.
>
> "It is a known fact that the bulking properties of pigments sold by various manufacturers under the same commercial name vary. It is also true that pigments turned out by any one manufacturer will vary in their bulking properties from time to time, though not to an appreciable extent; therefore, your committee suggests the compiling of a bulking table which can be the basis for the figuring of all formulas by paint manufacturers.
>
> "We recommend that the Scientific Section of the Educational Bureau undertake a complete study of the bulking properties of all pigments on the market and then publish the results of such information in pamphlet form for the benefit of the members of the Association.
>
> "Some manufacturers might be under the impression that the ability to figure a theoretical yield correctly is not highly important inasmuch as in the regular routine of their manufacturing departments they are naturally able to obtain information as to yields from the actual results secured.
>
> "To such manufacturers we would say that the ability to figure theoretical yields is highly important, first of all when it comes to making samples which are submitted to prospective buyers, accompanied by a selling price; secondly, in figuring on specification paints, and, thirdly, in checking yields reported by the factory."

It should also be pointed out that the specific gravity of liquids is of as much importance to the grinder as the specific gravity of pigments. All yield calculations, for instance, should be based on the specific gravity of the liquids used. The writer has often found manufacturers still using the antediluvian method of calculating the yield and cost of paint on the basis of 7½ lbs. per gallon for linsed oil. This practice should be discontinued at once if accuracy is desired.

Organization of Work.—As a first step in the work, an attempt was made to secure all pigments produced in America or consumed by the American paint-grinding industry. Accordingly a letter was sent out to producers, jobbers, importers, and consumers of pigments and colors, describing the object of the work and requesting pigment samples. A suggestion was also made that producers submit such information regarding the chemical composition or methods of production as they might care to make public. The response was very encouraging. Certain important pigments submitted by producers were also sent in by paint grinders, so that duplicate samples were received in many instances. After the pigments were obtained they were divided into two portions and placed in carefully labelled screw-cap glass jars. One set of the jars was delivered to the paint laboratory of the Bureau of Standards; the other set being reserved in the writer's laboratory for check determinations. The work was then started, the determinations being carefully checked and under the supervision of representatives of the writer's laboratory and representatives of the Bureau of Standards.

Some Older Methods.—Heretofore the specific gravity* of a pigment has usually been determined by weighing the pigment in air and again in benzine or similar liquid, to determine the weight of material displaced. From the resulting figures the specific gravity was calculated. In some methods that were published, shaking with benzine was recommended to remove the air. Unfortunately, however, mere shaking is insufficient to accomplish this; only a very small amount of the occluded air being removed from certain pigments by this procedure. The result would be a figure that might be incorrect to such a degree that paint yields and cost figures calculated therefrom would often be unreliable.

The specific gravity figures now in use at some paint factories were often determined by the existing methods referred to above and generally upon pigments which had not been

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***Specific Gravity.**—The ratio of the weight of a unit volume of a substance to the weight of an equal volume of water at defined temperatures.

Density.—The use of this term should be avoided if possible.

Density is a scientific term meaning the mass of a unit volume. Its numerical expression will vary with the units selected, and there is no occasion for using it when the term "specific gravity" is defined. Confusion may be avoided by not using the word "density" in specifications. A. S. T. M. 1918 Year Book.

dried. Some of these pigments, such as blues, may have contained as high as 6% to 8% of moisture. Others such as white lead may have contained from 0.5% to 1% of moisture. **Since all commercial pigments contain varying amounts of moisture, it can readily be seen that specific gravity figures obtained on undried pigments would give high bulking values.** The same pigments, when kept in a dry, warm store room, would lose moisture and give lower bulking factors when made into paint. Specific gravity results on one pigment could thus be obtained, that would vary widely according to the amount of moisture present in the pigment at the time of determination. For this reason it is the writer's belief that all specific gravity determinations should be made on dried pigments so that uniform and checkable results will be obtained. It is better to under-estimate than to over-estimate the bulking factor of a pigment.

Secure Factors from Color Makers.—It is suggested that **when grinders do not have facilities for making careful determinations of specific gravity, they request every pigment manufacturer with whom they deal to furnish the specific gravity of each pigment supplied.** From these figures the paint grinder can at once determine bulking factors by referring to the charts on pages 258 to 264 in this book. It is also suggested that grinders obtain from the color makers the composition of each color (percentage of true color and percentage and character of inert base) purchased, so that they may properly formulate specification paints. Such data would also be of value in figuring the approximate specific gravity of a combination pigment.* For instance, if a para red pigment consists of 10% para red and 90% calcium carbonate, the following method of calculation could be used to determine the specific gravity of the pigment:

Average specific gravity of pure Para Red Toner (1.51) x 10 = 15.10
" " " " Calcium Carbonate (2.71) x 90 = 243.90

259.00

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*This calculation might not quite compensate for the greater bulking value resulting from precipitation rather than mechanical mixing.

$\frac{259}{100}$ = 2.59 approximate specific gravity of composite pigment.

The approximate specific gravity of any other color of known composition might in the same manner be calculated, provided there be available the specific gravity factor of each component.

Research Work on Method.—With these difficulties in view, a study was immediately made at the outset of the investigation to devise a means of entirely eliminating the occluded air from a pigment when admixed with the wetting liquid chosen for the determination. It was at first found that when a pigment and liquid of even high wetting value were placed in a gravity bottle, only a portion of the entrained air could be removed by shaking. It was further found that when the same bottle was subsequently placed in a glass desiccator, attached to a vacuum water pump of the laboratory type, a steady efflux of air bubbles coming from the pigment-oil mixture could be observed for a period of from 5 to 10 minutes. It was at first assumed that the occluded air was at this period entirely removed from the mass. Determination of the specific gravity at this point, however, was found to give figures which could not be closely checked in repeat determinations. This result suggested that more occluded air was present around the pigment particles, every angle of which had apparently not been entirely wetted by the liquid. The next step in the investigation, therefore, was to arrange for a higher vacuum than could be obtained by the ordinary type of laboratory water pump. Accordingly, a high vacuum laboratory oil pump was secured. This was attached to the desiccator containing the specific gravity bottle, subsequent to the action of the water pump which had been applied until no more bubbles of entrained air appeared. As soon as the oil pump was attached, a stream of air bubbles immediately appeared and continued intermittently with certain pigments for periods of up to an hour. With a standardized procedure adopted from this experimental work, it was found possible to secure very close check determinations with nearly all pigments.

Shrinkage of Pigments Upon Grinding.—The majority of pigments consist of finely divided particles which are agglomerated together as small lumps. When these are mixed with oil a granular paste results. By passing this product through a heavy plate or roller mill, a smooth paste is obtained. What the mill really accomplishes is a breaking down of the pigment agglomerates and a wetting of each pigment particle with the oil. This action is accompanied by the elimination of the entrained or occluded air in the pigment. Upon standing for a short time the mass of finely ground paint produced as above, may subsequently show a slight shrinkage in volume, due to the elimination of a further small amount of occluded air or gas that is driven out as each pigment particle becomes more thoroughly wetted. The writer has found that the process of grinding can be eliminated if vacuum is applied to a mixture of oil and pigment. The surtace tension effects are thus overcome and a smooth paint is produced. It is probable that in the near future a commercial application of this principle could be made in the industry.

Subsequently a series of tests were made to determine the best type of specific gravity bottle to use. Bottles having a capacity of only 25 cc. invariably gave low and non-checkable results. Those having a wide-hole short capillary tube were found faulty because of the possibility of removing too large a drop of liquid from the capillary tube after filling. Flasks of the LeChatelier type, such as are used for specific gravity determinations on Portland cement, were experimented with. The amount of pigment required in these flasks was too great to secure entire removal of the air, and the results obtained were correct only to within two points in the first decimal place. The results of the investigation finally narrowed down the choice of bottles that were found satisfactory and accurate to the two types which are illustrated below. These are of 50 cc. capacity.

A further investigation was then made to determine what type of liquid was most satisfactory. Ordinarily a liquid would be required that could be used with all pigments, so that the standardized method of examination could be followed without modification. Water was found unsatisfactory because of its failure to wet carbon pigments and organic lakes. Benzine was found unsatisfactory because of its rapid evaporative value, which caused losses at the capillary tube and seepage at the joints of the bottle necks. Alcohol and benzol were subject to the same difficulties and had the further defect of being solvents for certain lakes derived from coal tar intermediates. Solvent naptha (boiling point 160° C.) was found of high wetting value but had a somewhat too rapid evaporative value. Furthermore it acted rapidly as a solvent for certain lakes. Carbon tetrachloride was found too volatile. Linseed and similar fatty oils were too immobile and did not show sufficiently high wetting value for quick determinations. The investigation finally showed that water-white kerosene was the best medium. Accordingly several gallons were obtained and allowed to settle to throw down any small amounts of water present. The kerosene was subsequently filtered and kept in five-gallon bottles.

The method finally developed is given below. It should be pointed out that this method gives slightly lower bulking figures than methods in which the pigments are not dried or in which air is not removed.

METHOD DEVELOPED AND STANDARDIZED

1. STANDARDIZATION OF PYKNOMETERS.—*Fill the pyknometers with freshly boiled distilled water and bring to temperature of 15.6° C. Dry and weigh as outlined in paragraph 6. Clean, weigh and dry the pyknometers. Fill them with the kerosene to be used and bring to temperature of 15.6° C. Dry and weigh in the same manner. Calculate the specific gravity of the kerosene by dividing the weight of water into the weight of kerosene.*

2. DRYING.—*Dry the pigment in an oven, preferably electric, at 105° C. for 2 hours.*

3. WEIGHING.—*Weigh a sample of the pigment, by difference, in the weighing bottle. For blacks, blues and lakes of light specific gravity, use about 1 gram; for inert crystalline pigments about 4 grams; for opaque white pigments 7 to 10 grams; for red lead 15-20 grams. Due to the hygroscopic nature of some of the pigments it is necessary to use a weighing bottle fitted with a cork stopper.*

4. TRANSFERRING TO PYKNOMETER.—*Pour sufficient kerosene into the pyknometer to form a quarter of an inch layer in the bottom and add a quantity of pigment from the weighing bottle reaching approximately three-quarters of the distance to the kerosene level. The kerosene should always cover the pigment. Stir the sample with a polished round bottom glass rod*

until completely covered by the kerosene, if necessary adding more kerosene from the wash bottle. Wash rod with kerosene.

5. REMOVAL OF OCCLUDED AIR.—*Place the pyknometers in the desiccator which should then be closed and attached to the water pump until the greater part of the air is expelled from the system. This takes from 5 to 10 minutes. Close the system with a pinchcock and attach the desiccator to an oil pump for the removal of the small amounts of air given off at the low pressures obtainable with the oil pump. The manometer is used to indicate whether the oil pump is giving the proper vacuum. When the manometer indicates that the vacuum, which should be not greater than 3 millimeters, is constant, the oil pump may be cut off for short periods, precaution being taken that the vacuum does not change materially due to leakage. It will be noticed that bubbles of air come from the pigments very rapidly at first and that this action gradually decreases and finally stops altogether. The time required for complete removal of air varies from one-half hour to two hours, depending upon the nature of the pigment. When no more bubbles can be seen it is assumed that all the occluded air has been given off and the pigment thoroughly wet with kerosene. Air can then be slowly admitted to the desiccator by means of the pinchcock.*

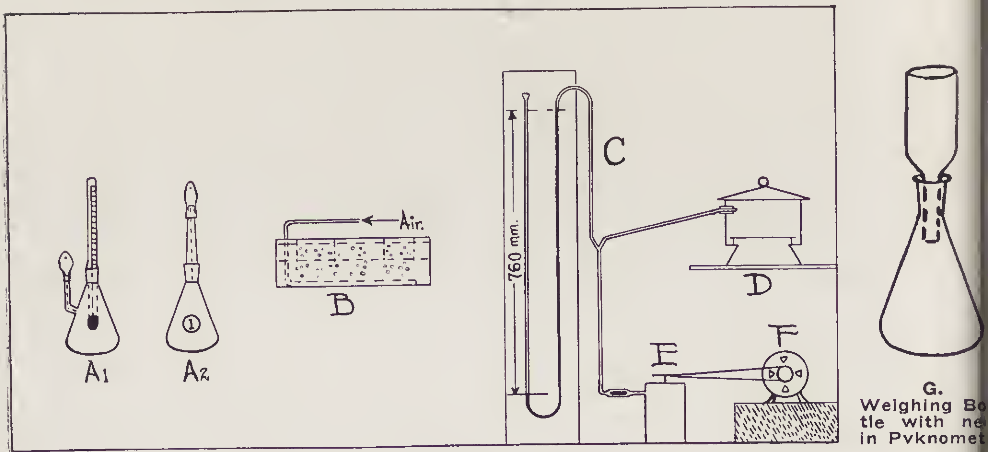

Fig. 29
APPARATUS USED IN WORK.

6. FILLING AND BRINGING TO TEMPERATURE.—*Take the pyknometer from the desiccator and fill with kerosene, care being taken to add sufficient to prevent the formation of air bubbles when placement of the thermometer is made. Place the thermometer in the water bath. Cool the bath with ice to between 10 and 13° C. Place the pyknometer in the bath and allow it to come to constant temperature. Insert the pyknometer thermometer or capillary tube. Add enough warm water to the bath to raise the temperature suddenly to about 14.5° C. in order to expand the kerosene and prevent it from creeping down the capillary and admitting a small amount of air. Allow the bath to come to a temperature of 15.6° C. Wipe the capillary tube with filter paper and put on the cap. Read all temperatures on the thermometer in the bath and not on the thermometer in the pyknometer. Remove the pyknometer from the bath and dry. Allow it to stand for one-half hour to enable it to come to room temperature. Weigh. It is advisable to allow pyknometer to stand approximately the same time before each weighing so as to compensate for slight errors due to evaporation at the joints.*

7. PRECAUTIONS.—*Before a new desiccator is first used it should be wrapped in a towel and tested under the vacuum to be used. Great care should be exercised in handling the desiccator when the vacuum is on, as any sudden jar may cause it to collapse.*

8. NUMBER OF SAMPLES.—*It has been found convenient to run six samples at one time, the desiccator specified being of the proper size to accommodate the above number.*

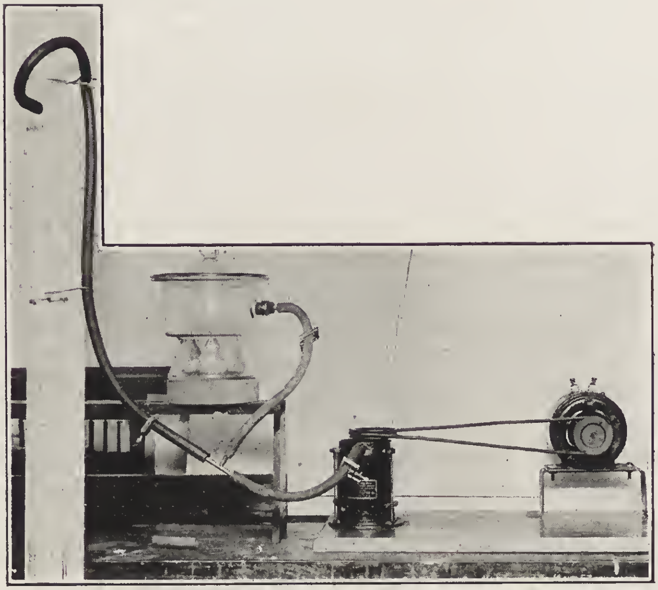

Fig. 30.

PHOTO OF APPARATUS.

BULKING VALUES AND YIELDS OF PIGMENTS AND LIQUIDS

DESCRIPTION OF APPARATUS USED IN METHOD

A. Pyknometer.	Capacity 50 CC.
Type A 1.	Thermometer at neck, and capillary tube at side. A. H. Thomas Co. No. 46688. Central Scientific Co. No. 1730.
Type A 2.	Long, thin capillary tube at neck. Specially made by Eimer and Amend.
B. Water Bath.	Vessel filled with sufficient water to permit of only a very gradual rise in temperature. Equipped with a stirring device, preferably air blown as the pyknometers are easily tipped over.
C. Manometer.	Open tube Manometer to show vacuum obtained in desiccator. Made of 6 mm diameter glass tubing filled with mercury to approximately 86 cm, fitted with pressure tubing attached to a Y tube leading to desiccator and pump.
D. Desiccator.	Heavy Wall Glass Desiccator constructed for vacuum work, with hole at side. To withstand vacuum of one atmosphere. A. H. Thomas Co. No. 25834. Scientific Materials Co. No. 1919. Central Scientific Co. No. 3776.
Water Pump.	Laboratory water vacuum pump necessary to expel the greater portion of air in the desiccator. Scientific Materials Co. No. 2554, No. 2556. Central Scientific Co. No. 5476.
E. High Vacuum Pump.	Small vacuum pump to give vacuum of not more than 3 mm. The Nelson Oil Pump is satisfactory. This pump has a displacement of approximately 200 cubic inches per minute or 7 cubic feet per hour at a speed of 900 R. P. M. It will evacuate a flask of one liter capacity to a vacuum of 1 mm. in two minutes. Scientific Materials Co. No. 143.
F. Electric Motor.	To drive Vacuum Oil Pump. A constant speed type of motor is most satisfactory. If for D. C., a shunt wound motor, or, if for A. C. an induction motor. The most efficient speed is between 800 and 900 R. P. M. At this speed it gives as good results as when driven faster, and develops less heat.
G. Weighing Bottle.	Weighing Bottle with cork. Neck to be small enough to fit inside the neck of the pyknometer. This is essential, as small quantities of pigment easily adhere to the ground glass joint of the pyknometer. 25 cc Babcock Milk Test Bottles are satisfactory after cutting off the neck to approximately 1½ inch. A. H. Thomas Co. No. 33964. Scientific Materials Co. No. 4176. Central Scientific Co. No. 9104.
Thermometer.	Range 0° to 60° C. graduated in 1/10ths of a degree.
Wash Bottle.	Ordinary water bottle type for kerosene.

Note: The difference in levels of the mercury in the manometer when the system is in operation, subtracted from the barometer reading taken at the same time, gives the vacuum of the system in millimeters of mercury. The difference between the Barometer and the Manometer should not exceed 3 MM.

EXAMPLE OF CALCULATIONS

Standardization of Pyknometer

Weight of bottle and water = 88.2777
Weight of bottle = 33.3527

Weight of water at 15.5° C. = 54.9250

Weight of bottle and kerosene = 77.9420
Weight of bottle = 33.3527

Wt. of kerosene at 15.5° C. = 44.5893

$$\text{Specific gravity of Kerosene } 15.5^\circ \text{ C.} = \frac{\text{Weight Kerosene}}{\text{Weight Water}} = \frac{44.5893}{54.9250} = .8118$$

METHOD OF CALCULATING RESULTS

Let K = Weight of bottle filled with kerosene only
Let P = Weight of pigment used.
Let F = Final weight of bottle with pigment and kerosene.
Let S = Specific gravity of the kerosene used.

$$\text{Then Specific Gravity of Pigment} = \frac{P \times S}{(P + K) - F}$$

Thus:

Red Lead

K = Weight of bottle filled with kerosene only = 77.9420
P = 16.0000
F = Final weight of bottle with pigment and kerosene = 92.4826
S = .8118

$$\text{Sp. Gr. of sample Red Lead} = \frac{16.0000 \times .8118}{(16.0000 + 77.9420) - 92.4826} = 8.90$$

Results of Investigation.—The results of the investigation are presented in the preceding charts which give the commercial name of each pigment, the producer or selling agent, information voluntarily submitted regarding composition, the specific gravity, weight per solid gallon, and the fraction of a gallon bulked by one pound. While these figures may in most instances be taken as representative of the pigments named as they are produced today, it should be pointed out that different batches of pigments may occasionally vary slightly in specific gravity. It should also be suggested that some of the manufacturers named, may, if requested, be able to produce pigments of higher or lower specific gravity as desired.

METHOD FOR DETERMINING SPECIFIC GRAVITY OF PAINT LIQUIDS

The specific gravity of a liquid may be determined with sufficient accuracy with a Westphal balance or a standardized hydrometer and thermometer in the customary manner, making any necessary corrections for temperature. For viscous blown or bodied oils or for mixed paints, satisfactory results may be obtained, when a quantity is available, by weighing a liter of the oil or paint in a graduated tared liter flask. When only small amounts are available or when great accuracy is desired the method below* should be used:

The specific gravity of an oil is expressed as the ratio of the weight of a given volume at 25° C. to that of an equal volume of water at the same temperature. The determination of specific gravity, in the case of heavy bodied oils especially, is best made in a straight walled glass tube pyknometer approximately 70 mm. long and 22 mm. in diameter, carefully ground to receive an accurately fitting glass stopper with a hole of 1.5 to 1.7 mm. bore in place of the usual capillary opening. The stoppered tube should have a capacity of about 24 cc., and when empty should weigh not over 35 grams.

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*This method is adapted from the proposed tentative standard test for the specific gravity of road oils, road tars, asphalt cements, and soft tar pitches. See Proc. Amer. Soc. Test. Mater., Vol. XX, 1920.

The type of bottle described is illustrated below.

Fig. 31.

PYKNOMETER OR WEIGHING BOTTLE

The pyknometer with stopper is first calibrated by weighing it, clean and dry, upon an analytical balance. This weight is called A. It is then filled with distilled water at a temperature of 25° C., the stopper firmly inserted, all surplus moisture wiped from the surface with a clean, dry cloth, and again weighed. This weight is called B.

For the determination of the specific gravity of an ordinary oil or paint liquid, the material shall be brought to a temperature of 25° C. and poured into the pyknometer until it is full, using care to prevent the inclusion of air bubbles. The stopper is then inserted and the excess of liquid forced through the opening is carefully removed with a clean, dry cloth. The pyknometer and contents are then weighed, and this weight is called C. The specific gravity of the material may then be calculated from the following formula:

$$\text{Specific gravity} = \frac{C - A}{B - A}$$

When the specific gravity of extremely heavy bodied oils or blown oils, or certain paste products is to be determined, the viscosity of these materials may make the previously described method inapplicable. Such materials should first be gently warmed, using care to prevent loss by evaporation. When fluid, enough is poured into the clean, dry pyknometer to about half fill it. Precautions shall be taken to keep the material from touching the sides of the tube above the final level and to prevent the inclusion of air bubbles. It is advisable to slightly warm the tube before filling. The pyknometer and contents are then cooled to room temperature and weighed with the stopper. This weight is called C. The pyknometer is next removed from the balance, filled with distilled water, and the stopper firmly inserted. It is then completely immersed for not less than 30 minutes in a beaker of distilled water maintained at 25° C., after which it is removed and all surplus water wiped off with a clean cloth. It is immediately weighed. This weight is called D. The specific gravity of the material may be calculated from the following formula:

$$\text{Specific gravity} = \frac{C - A}{(B\text{-}A)\ (D\text{-}C)}$$

EXAMPLES OF FIGURING BULKING VALUES OF PAINT FORMULAS

Example I

Pounds	Spec. Grav. of material	One Pound bulks gallons	Poundage used bulks gallons	Mat'l Cost	Invoice per lb.	Material Cost	Invoice Pounds used
Pigment 64%							
19 Basic Carbonate White Lead	6.81	.01763	.334				
19 Basic Sulphate White Lead	6.41	.01873	.355				
19 Zinc Oxide	5.66	.02121	.402				
7 Barium Sulphate	4.45	.02698	.188				
Liquid 36%							
30 Linseed Oil	.932	.1288	3.864				
3 Mineral Spirits	.775	.1549	.464				
3 Drier	.802	.1497	.449				
100 lbs			6.056 Gals.				

Gallons bulked 6.056
Weight per gallon .... 16.5 lbs.
Raw Material cost per gallon ....

From the above figures Deduction for Volatile and Grinding Loss should be made.
To the above figures Cost of Cans and Cases should be added.
To the above figures Manufacturing Costs should be added.
To the above figures Selling Price over Factory Cost should be added.
To the above figures Net Profit on Sales should be added.

} See Report of Cost Accounting Committee.

Example II

Pounds	Spec. Grav. of material	One Pound bulks gallons	Poundage used bulks gallons	Mat'l Cost	Invoice per lb.	Material Cost	Invoice Pounds used
275 lbs. Basic Carbonate White Lead	6.81	.01763	4.85				
275 lbs. Basic Sulphate White Lead	6.41	.01873	5.15				
300 lbs. Zinc Oxide	5.66	.02121	6.36				
100 lbs. Silica	2.65	.04531	4.53				
25 lbs. Whiting	2.71	.04431	1.11				
442 lbs. Raw Linseed Oil	9.32	.1288	56.93				
40 lbs. Bodied Linseed Oil	9.86	.1225	4.90				
15 lbs. Drier	8.02	.1497	2.25				
12 lbs. Mineral Spirits	7.75	.1549	1.86				
15 lbs. Water Solution	1.00	.1200	1.80				
1499 Lbs.			89.74 Gals.				

Gallons bulked 89.74
Weight per gallon .... 16.7 lbs.
Raw material Cost per gallon ....

From the above figures Deduction for Volatile and Grinding Loss should be made.
To the above figures Cost of Cans and Cases should be added.
To the above figures Manufacturing Costs should be added.
To the above figures Selling Price over Factory Cost should be added.
To the above figures Net Profit on Sales should be added.

See Report of Cost Accounting Committee.

Example II is similar to a formula recently sent out to various grinders for bulk estimations, except that the liquid components vary slightly. For grinders who still base their batches on gallonage rather than poundage of liquids, the poundage can be obtained from the weight per gallon.

Average Specific Gravity Data.—For the convenience of manufacturers who do not have facilities for making specific gravity determinations on various types of pigments and who desire a chart showing the average specific gravity of many of the commonly used pigments, a special table has been prepared. It should be pointed out, however, that the specific gravity of even such colors as are termed chemically pure, such for instance as chrome green, may vary to a great extent. This is due to the fact that these greens may contain a small or a large percentage of blue, and in some instances varying amounts of lead sulphate. This variance in composition, therefore, of even chemically pure colors, is sufficient to warn the paint grinder that serious mistakes may occur in calculations made on average specific gravity figures of certain colored pigments. A chart is also presented showing the specific gravity and bulking values of many liquids that are used in paint and enamel manufacture.

Calculation Charts.—For the further convenience of manufacturers, there is presented a series of calculation charts in which the specific gravity, weight per solid gallon and gallons bulked per pound of every liquid and pigment substance used is presented. These tables start at .70 which is lower than the density of alcohol, benzol, or benzine, the light liquids used in paints. They run up to 9.70, which is higher than the density of the heaviest pigment used. With these charts the yield of a batch of paint may be determined rapidly, as will be indicated by examples on page 254.

TABLE 87

TABLE OF BULKING AVERAGES

Specific Gravity of Paint Liquids

	Specific Gravity	Weight per solid gallon	One pound bulks gallons.
Raw Linseed Oil	.932	7.764	.1288
Boiled Linseed Oil	.942	7.847	.1274
Heavy Bodied Linseed Oil	.98	8.163	.1225
Raw Soya Bean Oil	.929	7.739	.1292
Raw Tung Oil	.940	7.830	.1277
**Liquid Paint Drier	.85	6.681	.1497
**Mixing Varnish	.905	7.539	.1326
Turpentine	.867	7.222	.1385
**Mineral Spirits	.775	6.456	.1549
Benzol (90%)	.882	7.347	.1361
Benzine (62°)	.745	6.206	.1611
Solvent Naphtha (160°)	.902	7.514	.1331
Grain Alcohol	.785	6.539	.1529
Wood Alcohol	.791	6.589	.1518
Acetone	.797	6.639	.1506
Carbon Tetrachloride	1.60	13.328	.07502
Amyl Acetate	.874	7.280	.1374
Ethyl Acetate	.902	7.514	.1331

****THESE LIQUIDS WILL VARY GREATLY IN SPECIFIC GRAVITY. GRINDERS SHOULD DETERMINE THE FIGURE FOR THE PRODUCTS THEY USE.**

TABLE 88

TABLE OF BULKING AVERAGES

Specific Gravity of Dry Pigments

	Specific Gravity	Weight per solid gallon	One pound bulks gallons
Basic Carbonate White Lead	6.81	56.73	.01763
Basic Sulphate White Lead	6.41	53.40	.01873
Zinc Oxide	5.66	47.15	.02121
Zinc Oxide, Leaded 35%	5.95	49.56	.02018
Lithopone	4.30	35.82	.02792
Titanox	4.30	35.82	.02792
Asbestine	2.85	23.74	.04212
Barytes	4.45	37.07	.02698
China Clay	2.62	21.82	.04583
Silica	2.65	22.07	.04531
Talc	2.84	23.66	.04227
Whiting	2.71	22.57	.04431
*Venetian Red, (20% Fe_2O_3)	3.05	25.41	.03935
Red Oxide, (40% Fe_2O_3)	3.45	28.74	.03479
Red Oxide, (95% Fe_2O_3)	4.95	41.23	.02425
Indian Red, (90% Fe_2O_3)	4.92	40.98	.02440
Ferric Oxide, (98% Fe_2O_3)	5.15	42.90	.02331
Tuscan Red	3.95	32.90	.03040
Ochre	2.80	23.32	.04288
Sienna, Raw	3.27	27.24	.03671
Sienna, Burnt	3.95	32.90	.03040
Umber, Raw	2.68	22.32	.04480
Umber, Burnt	3.80	31.65	.03160
Brown Oxide, (50% Fe_2O_3)	3.35	27.91	.03583
Mineral Brown, (45% Fe_2O_3)	3.34	27.82	.03595
Chromium Oxide	4.95	41.23	.02425
Litharge	9.40	78.30	.01277
Orange Mineral	8.80	73.30	.01364
Red Lead	8.80	73.30	.01364
Pure Para Toner	1.50	12.50	.0800
Para Red 10% (on Lime and Barium Base)	2.65	22.07	.04531
Pure Toluidine Red Toner	1.49	12.41	.08058
*Chrome Green, C. P.	*3.90 *5.08		
Green Earth	2.75	22.91	.04365
Limeproof Green	2.80	23.32	.04288
American Blue	1.85	15.41	.06489
Ultramarine Blue	2.35	19.58	.05107
*Chrome Yellow, C. P.	*6.00		
Lampblack	1.78	14.83	.06743
Carbon Black	1.81	15.08	.06631
Drop Black	2.64	21.99	.04548
Graphite	2.36	19.66	.05086
Keystone Filler	2.71	22.57	.04431
Zinc Dust	7.06	58.81	.01700
Aluminum Dust	2.64	21.99	.04548
Lead Dust	11.09	92.38	.01082

*THESE WILL VARY WIDELY ACCORDING TO COMPOSITION REQUIRED FOR SHADE OR TONE AND CHARACTER OF BASE.

TABLE 89

Liquid and Pigment Yield Chart

Specific Gravity	Weight Per Solid Gallon	[illegible] Bulks [illegible]	Specific Gravity	Weight Per Solid Gallon	One Pound Bulks Gallons	Specific Gravity	Weight Per Solid Gallon	One Pound Bulks Gallons
.70	5.831	.1715	.98	8.163	.1225	1.40	11.66	.08576
.71	5.914	.1691	.99	8.247	.1213	1.41	11.75	.08511
.72	5.998	.1667	1.00	8.330	.1200	1.42	11.83	.08453
.73	6.081	.1644	1.01	8.413	.1189	1.43	11.91	.08396
.74	6.164	.1622	1.02	8.497	.1177	1.44	12.00	.08333
.75	6.248	.1601	1.03	8.580	.1166	1.45	12.08	.08278
.76	6.331	.1580	1.04	8.663	.1154	1.46	12.16	.08224
.77	6.414	.1559	1.05	8.747	.1143	1.47	12.25	.08163
.78	6.497	.1539	1.06	8.830	.1133	1.48	12.33	.08110
.79	6.581	.1520	1.07	8.913	.1122	1.49	12.41	.08058
.80	6.664	.1501	1.08	8.996	.1112	1.50	12.50	.08000
.81	6.747	.1482	1.09	9.080	.1101	1.51	12.58	.07949
.82	6.831	.1464	1.10	9.163	.1091	1.52	12.66	.07899
.83	6.914	.1446	1.11	9.246	.1082	1.53	12.75	.07843
.84	6.997	.1429	1.12	9.330	.1072	1.54	12.83	.07794
.85	7.081	.1412	1.13	9.413	.1062	1.55	12.91	.07746
.86	7.164	.1396	1.14	9.496	.1053	1.56	12.99	.07698
.87	7.247	.1380	1.15	9.580	.1044	1.57	13.08	.07645
.88	7.330	.1364	1.16	9.663	.1035	1.58	13.16	.07599
.89	7.414	.1349	1.17	9.746	.1026	1.59	13.24	.07553
.90	7.497	.1334	1.18	9.829	.1017	1.60	13.33	.07502
.91	7.580	.1319	1.19	9.913	.1009	1.61	13.41	.07457
.92	7.664	.1305	1.20	9.996	.1000	1.62	13.49	.07413
.930	7.747	.1291	1.21	10.08	.09921	1.63	13.58	.07364
.931	7.755	.1289	1.22	10.16	.09843	1.64	13.66	.07321
.932	7.764	.1288	1.23	10.25	.09756	1.65	13.74	.07278
.933	7.772	.1287	1.24	10.33	.09681	1.66	13.83	.07231
.934	7.780	.1285	1.25	10.41	.09606	1.67	13.91	.07189
.935	7.789	.1284	1.26	10.50	.09524	1.68	13.99	.07148
.936	7.797	.1283	1.27	10.58	.09452	1.69	14.08	.07102
.937	7.805	.1281	1.28	10.66	.09381	1.70	14.16	.07062
.938	7.814	.1280	1.29	10.75	.09302	1.71	14.24	.07022
.939	7.822	.1278	1.30	10.83	.09234	1.72	14.33	.06978
.940	7.830	.1277	1.31	10.91	.09166	1.73	14.41	.06940
.941	7.839	.1276	1.32	11.00	.09091	1.74	14.49	.06901
.942	7.847	.1274	1.33	11.08	.09025	1.75	14.58	.06859
.943	7.855	.1273	1.34	11.16	.08961	1.76	14.66	.06821
.944	7.864	.1272	1.35	11.25	.08889	1.77	14.74	.06784
.945	7.872	.1270	1.36	11.33	.08826	1.78	14.83	.06743
.95	7.914	.1264	1.37	11.41	.08764	1.79	14.91	.06707
.96	7.997	.1250	1.38	11.50	.08696	1.80	14.99	.06671
.97	8.080	.1238	1.39	11.58	.08636	1.81	15.08	.06631

TABLE 89 (Continued)

Specific Gravity	Weight Per Solid Gallon	One Pound Bulks Gallons	Specific Gravity	Weight Per Solid Gallon	One Pound Bulks Gallons	Specific Gravity	Weight Per Solid Gallon	One Pound Bulks Gallons
1.82	15.16	.06596	2.26	18.83	.05311	2.70	22.49	.04446
1.83	15.24	.06562	2.27	18.91	.05288	2.71	22.57	.04431
1.84	15.33	.06523	2.28	18.99	.05266	2.72	22.66	.04413
1.85	15.41	.06489	2.29	19.08	.05241	2.73	22.74	.04398
1.86	15.49	.06456	2.30	19.16	.05219	2.74	22.82	.04382
1.87	15.58	.06418	2.31	19.24	.05197	2.75	22.91	.04365
1.88	15.66	.06386	2.32	19.33	.05173	2.76	22.99	.04350
1.89	15.74	.06353	2.33	19.41	.05152	2.77	23.07	.04335
1.90	15.83	.06317	2.34	19.49	.05131	2.78	23.16	.04318
1.91	15.91	.06285	2.35	19.58	.05107	2.79	23.24	.04303
1.92	15.99	.06254	2.36	19.66	.05086	2.80	23.32	.04288
1.93	16.08	.06219	2.37	19.74	.05065	2.81	23.41	.04272
1.94	16.16	.06188	2.38	19.83	.05043	2.82	23.49	.04257
1.95	16.24	.06158	2.39	19.91	.05023	2.83	23.57	.04243
1.96	16.33	.06124	2.40	19.99	.05003	2.84	23.66	.04227
1.97	16.41	.06094	2.41	20.08	.04980	2.85	23.74	.04212
1.98	16.49	.06064	2.42	20.16	.04960	2.86	23.82	.04198
1.99	16.58	.06031	2.43	20.24	.04941	2.87	23.91	.04182
2.00	16.66	.06002	2.44	20.33	.04919	2.88	23.99	.04168
2.01	16.74	.05974	2.45	20.41	.04900	2.89	24.07	.04155
2.02	16.83	.05942	2.46	20.49	.04880	2.90	24.16	.04139
2.03	16.91	.05914	2.47	20.58	.04859	2.91	24.24	.04125
2.04	16.99	.05886	2.48	20.66	.04840	2.92	24.32	.04112
2.05	17.08	.05855	2.49	20.74	.04822	2.93	24.41	.04097
2.06	17.16	.05828	2.50	20.83	.04801	2.94	24.49	.04083
2.07	17.24	.05800	2.51	20.91	.04782	2.95	24.57	.04070
2.08	17.33	.05770	2.52	20.99	.04764	2.96	24.66	.04055
2.09	17.41	.05744	2.53	21.07	.04746	2.97	24.74	.04042
2.10	17.49	.05718	2.54	21.16	.04726	2.98	24.82	.04029
2.11	17.58	.05688	2.55	21.24	.04708	2.99	24.91	.04014
2.12	17.66	.05663	2.56	21.32	.04690	3.00	24.99	.04002
2.13	17.74	.05637	2.57	21.41	.04671	3.01	25.07	.03989
2.14	17.83	.05609	2.58	21.49	.04653	3.02	25.16	.03975
2.15	17.91	.05583	2.59	21.57	.04636	3.03	25.24	.03962
2.16	17.99	.05559	2.60	21.66	.04617	3.04	26.32	.03949
2.17	18.08	.05531	2.61	21.74	.04600	3.05	25.41	.03935
2.18	18.16	.05507	2.62	21.82	.04583	3.06	25.49	.03923
2.19	18.24	.05482	2.63	21.91	.04564	3.07	25.57	.03911
2.20	18.33	.05456	2.64	21.99	.04548	3.08	25.66	.03897
2.21	18.41	.05432	2.65	22.07	.04531	3.09	25.74	.03885
2.22	18.49	.05408	2.66	22.16	.04513	3.10	25.82	.03873
2.23	18.58	.05382	2.67	22.24	.04496	3.11	25.91	.03860
2.24	18.66	.05359	2.68	22.32	.04480	3.12	25.99	.03848
2.25	18.74	.05336	2.69	22.41	.04462	3.13	26.07	.03836

TABLE 89 (Continued)

Specific Gravity	Weight Per Solid Gallon	One Pound Bulks Gallons	Specific Gravity	Weight Per Solid Gallon	One Pound Bulks Gallons	Specific Gravity	Weight Per Solid Gallon	One Pound Bulks Gallons
3.14	26.16	.03823	3.58	29.82	.03353	4.02	33.49	.02986
3.15	26.24	.03811	3.59	29.90	.03344	4.03	33.57	.02979
3.16	26.32	.03799	3.60	29.99	.03334	4.04	33.65	.02972
3.17	26.41	.03786	3.61	30.07	.03326	4.05	33.74	.02964
3.18	26.49	.03775	3.62	30.15	.03317	4.06	33.82	.02957
3.19	26.57	.03764	3.63	30.24	.03307	4.07	33.90	.02950
3.20	26.66	.03751	3.64	30.32	.03298	4.08	33.99	.02942
3.21	26.74	.03740	3.65	30.40	.03289	4.09	34.07	.02935
3.22	26.82	.03729	3.66	30.49	.03280	4.10	34.15	.02928
3.23	26.91	.03716	3.67	30.57	.03271	4.11	34.24	.02921
3.24	26.99	.03705	3.68	30.65	.03263	4.12	34.32	.02914
3.25	27.07	.03694	3.69	30.74	.03253	4.13	34.40	.02907
3.26	27.16	.03682	3.70	30.82	.03245	4.14	34.49	.02899
3.27	27.24	.03671	3.71	30.90	.03236	4.15	34.57	.02893
3.28	27.32	.03660	3.72	30.99	.03227	4.16	34.65	.02886
3.29	27.41	.03648	3.73	31.07	.03219	4.17	34.74	.02879
3.30	27.49	.03638	3.74	31.15	.03210	4.18	34.82	.02872
3.31	27.57	.03627	3.75	31.24	.03201	4.19	34.90	.02865
3.32	27.66	.03615	3.76	31.32	.03193	4.20	34.99	.02858
3.33	27.74	.03605	3.77	31.40	.03185	4.21	35.07	.02851
3.34	27.82	.03595	3.78	31.49	.03176	4.22	35.15	.02845
3.35	27.91	.03583	3.79	31.57	.03168	4.23	35.24	.02838
3.36	27.99	.03573	3.80	31.65	.03160	4.24	35.32	.02831
3.37	28.07	.03563	3.81	31.74	.03151	4.25	35.40	.02825
3.38	28.16	.03551	3.82	31.82	.03143	4.26	35.49	.02818
3.39	28.24	.03541	3.83	31.90	.03135	4.27	35.57	.02811
3.40	28.32	.03531	3.84	31.99	.03126	4.28	35.65	.02805
3.41	28.41	.03520	3.85	32.07	.03118	4.29	35.74	.02798
3.42	28.49	.03510	3.86	32.15	.03110	4.30	35.82	.02792
3.43	28.57	.03500	3.87	32.24	.03102	4.31	35.90	.02786
3.44	28.66	.03489	3.88	32.32	.03094	4.32	35.99	.02779
3.45	28.74	.03479	3.89	32.40	.03086	4.33	36.07	.02772
3.46	28.82	.03470	3.90	32.49	.03078	4.34	36.15	.02766
3.47	28.91	.03459	3.91	32.57	.03070	4.35	36.24	.02759
3.48	28.99	.03449	3.92	32.65	.03062	4.36	36.32	.02753
3.49	29.07	.03440	3.93	32.74	.03054	4.37	36.40	.02747
3.50	29.16	.03429	3.94	32.82	030.47	4.38	36.49	.02740
3.51	29.24	.03420	3.95	32.90	.03040	4.39	36.57	.02734
3.52	29.32	.03411	3.96	32.99	.03031	4.40	36.65	.02729
3.53	29.40	.03401	3.97	33.07	.03024	4.41	36.74	.02722
3.54	29.49	.03391	3.98	33.15	.03017	4.42	36.82	.02716
3.55	29.57	.03382	3.99	33.24	.03008	4.43	36.90	.02710
3.56	29.65	.03373	4.00	33.32	.03001	4.44	36.99	.02703
3.57	29.74	.03362	4.01	33.40	.02994	4.45	37.07	.02698

TABLE 89 (Continued)

Specific Gravity	Weight Per Solid Gallon	One Pound Bulks Gallons	Specific Gravity	Weight Per Solid Gallon	One Pound Bulks Gallons	Specific Gravity	Weight Per Solid Gallon	One Pound Bulks Gallons
4.46	37.15	.02692	4.90	40.82	.02450	5.34	44.48	.02248
4.47	37.24	.02685	4.91	40.90	.02445	5.35	44.57	.02244
4.48	37.32	.02680	4.92	40.98	.02440	5.36	44.65	.02240
4.49	37.40	.02674	4.93	41.07	.02435	5.37	44.73	.02236
4.50	37.49	.02667	4.94	41.15	.02430	5.38	44.82	.02231
4.51	37.57	.02662	4.95	41.23	.02425	5.39	44.90	.02227
4.52	37.65	.02656	4.96	41.32	.02420	5.40	44.98	.02223
4.53	37.73	.02650	4.97	41.40	.02415	5.41	45.07	.02219
4.54	37.82	.02644	4.98	41.48	.02411	5.42	45.15	.02215
4.55	37.90	.02639	4.99	41.57	.02406	5.43	45.23	.02211
4.56	37.98	.02633	5.00	41.65	.02401	5.44	45.32	.02207
4.57	38.07	.02627	5.01	41.73	.02396	5.45	45.40	.02203
4.58	38.15	.02621	5.02	41.82	.02391	5.46	45.48	.02199
4.59	38.23	.02616	5.03	41.90	.02386	5.47	45.57	.02194
4.60	38.32	.02610	5.04	41.98	.02382	5.48	45.65	.02191
4.61	38.40	.02604	5.05	42.07	.02377	5.49	45.73	.02187
4.62	38.48	.02599	5.06	42.15	.02373	5.50	45.82	.02182
4.63	38.57	.02593	5.07	42.23	.02368	5.51	45.90	.02179
4.64	38.65	.02587	5.08	42.32	.02363	5.52	45.98	.02175
4.65	38.73	.02582	5.09	42.40	.02358	5.53	46.06	.02171
4.66	38.82	.02576	5.10	42.48	.02354	5.54	46.15	.02167
4.67	38.90	.02571	5.11	42.57	.02349	5.55	46.23	.02163
4.68	38.98	.02565	5.12	42.65	.02345	5.56	46.31	.02159
4.69	39.07	.02560	5.13	42.73	.02340	5.57	46.40	.02155
4.70	39.15	.02554	5.14	42.82	.02335	5.58	46.48	.02151
4.71	39.23	.02549	5.15	42.90	.02331	5.59	46.56	.02148
4.72	39.32	.02543	5.16	42.98	.02327	5.60	46.65	.02144
4.73	39.40	.02538	5.17	43.07	.02322	5.61	46.73	.02140
4.74	39.48	.02533	5.18	43.15	.02317	5.62	46.81	.02136
4.75	39.57	.02527	5.19	43.23	.02313	5.63	46.90	.02132
4.76	39.65	.02522	5.20	43.32	.02308	5.64	46.98	.02129
4.77	39.73	.02517	5.21	43.40	.02304	5.65	47.06	.02125
4.78	39.82	.02511	5.22	43.48	.02200	5.66	47.15	.02121
4.79	39.90	.02506	5.23	43.57	.02295	5.67	47.23	.02117
4.80	39.98	.02501	5.24	43.65	.02291	5.68	47.31	.02114
4.81	40.07	.02496	5.25	43.73	.02287	5.69	47.40	.02110
4.82	40.15	.02491	5.26	43.82	.02282	5.70	47.48	.02106
4.83	40.23	.02486	5.27	43.90	.02278	5.71	47.56	.02103
4.84	40.32	.02480	5.28	43.98	.02274	5.72	47.65	.02099
4.85	40.40	.02475	5.29	44.07	.02269	5.73	47.73	.02095
4.86	40.48	.02470	5.30	44.15	.02265	5.74	47.81	.02092
4.87	40.57	.02465	5.31	44.23	.02261	5.75	47.90	.02088
4.88	40.65	.02460	5.32	44.32	.02256	5.76	47.98	.02084
4.89	40.73	.02455	5.33	44.40	.02252	5.77	48.06	.02081

TABLE 89 (Continued)

Specific Gravity	Weight Per Solid Gallon	One Pound Bulks Gallons	Specific Gravity	Weight Per Solid Gallon	One Pound Gallons	Specific Gravity	Weight Per Solid Gallon	One Pound Bulks Gallons
5.78	48.15	.02077	6.22	51.81	.01930	6.66	55.48	.01802
5.79	48.23	.02073	6.23	51.90	.01927	6.67	55.56	.01800
5.80	48.31	.02070	6.24	51.98	.01924	6.68	55.64	.01797
5.81	48.40	.02066	6.25	52.06	.01921	6.69	55.73	.01794
5.82	48.48	.02063	6.26	52.15	.01918	6.70	55.81	.01792
5.83	48.56	.02059	6.27	52.23	.01915	6.71	55.89	.01789
5.84	48.65	.02056	6.28	52.31	.01912	6.72	55.98	.01786
5.85	48.73	.02052	6.29	52.40	.01908	6.73	56.06	.01784
5.86	48.81	.02049	6.30	52.48	.01905	6.74	56.14	.01781
5.87	48.90	.02045	6.31	52.56	.01903	6.75	56.23	.01778
5.88	48.98	.02042	6.32	52.65	.01899	6.76	56.31	.01776
5.89	49.06	.02038	6.33	52.73	.01896	6.77	56.39	.01773
5.90	49.15	.02034	6.34	52.81	.01894	6.78	56.48	.01771
5.91	49.23	.02031	6.35	52.90	.01890	6.79	56.56	.01768
5.92	49.31	.02028	6.36	52.98	.01888	6.80	56.64	.01766
5.93	49.40	.02024	5.37	53.06	.01885	6.81	56.73	.01763
5.94	49.48	.02021	6.38	53.15	.01881	6.82	56.81	.01760
5.95	49.56	.02018	6.39	53.23	.01879	6.83	56.89	.01758
5.96	49.65	.02014	6.40	53.31	.01876	6.84	56.98	.01755
5.97	49.73	.02011	6.41	53.40	.01873	6.85	57.06	.01753
5.98	49.81	.02008	6.42	53.48	.01870	6.86	57.14	.01750
5.99	49.90	.02004	6.43	53.56	.01867	6.87	57.23	.01747
6.00	49.98	.02001	6.44	53.65	.01864	6.88	57.31	.01745
6.01	50.06	.01998	6.45	53.73	.01861	6.89	57.39	.01742
6.02	50.15	.01994	6.46	53.81	.01858	6.90	57.48	.01740
6.03	50.23	.01991	6.47	53.90	.01855	6.91	57.56	.01737
6.04	50.31	.01988	6.48	53.98	.01853	6.92	57.64	.01735
6.05	50.40	.01984	6.49	54.06	.01850	6.93	57.73	.01732
6.06	50.48	.01981	6.50	54.15	.01847	6.94	57.81	.01730
6.07	50.56	.01978	6.51	54.23	.01844	6.95	57.89	.01727
6.08	50.65	.01974	6.52	54.31	.01841	6.96	57.98	.01725
6.09	50.73	.01971	6.53	54.39	.01839	6.97	58.06	.01722
6.10	50.81	.01968	6.54	54.48	.01836	6.98	58.14	.01720
6.11	50.90	.01965	6.55	54.56	.01833	6.99	58.23	.01717
6.12	50.98	.01962	6.56	54.64	.01830	7.00	58.31	.01715
6.13	51.06	.01958	6.57	54.73	.01827	7.01	58.39	.01713
6.14	51.15	.01955	6.58	54.81	.01824	7.02	58.48	.01710
6.15	51.23	.01952	6.59	54.89	.01822	7.03	58.56	.01708
6.16	51.31	.01949	6.60	54.98	.01819	7.04	58.64	.01705
6.17	51.40	.01946	6.61	55.06	.01816	7.05	58.73	.01703
6.18	51.48	.01943	6.62	55.14	.01814	7.06	58.81	.01700
6.19	51.56	.01939	6.63	55.23	.01811	7.07	58.89	.01698
6.20	51.65	.01936	6.64	55.31	.01808	7.08	58.98	.01695
6.21	51.73	.01933	6.65	55.39	.01805	7.09	59.06	.01693

TABLE 89 (Continued)

Specific Gravity	Weight Per Solid Gallon	One Pound Gallons	Specific Gravity	Weight Per Solid Gallon	One Pound Bulks Gallons	Specific Gravity	Weight Per Solid Gallon	One Pound. Bulks Gallons
7.10	59.14	.01691	7.54	62.81	.01592	7.98	66.47	.01504
7.11	59.23	.01688	7.55	62.89	.01590	7.99	66.56	.01502
7.12	59.31	.01686	7.56	62.97	.01588	8.00	66.64	.01501
7.13	59.39	.01684	7.57	63.06	.01586	8.01	66.72	.01499
7.14	59.48	.01681	7.58	63.14	.01584	8.02	66.81	.01497
7.15	59.56	.01679	7.59	63.22	.01582	8.03	66.89	.01495
7.16	59.64	.01677	7.60	63.31	.01580	8.04	66.97	.01493
7.17	59.73	.01674	7.61	63.39	.01578	8.05	67.06	.01491
7.18	59.81	.01672	7.62	63.47	.01576	8.06	67.14	.01489
7.19	59.89	.01670	7.63	63.56	.01573	8.07	67.22	.01488
7.20	59.98	.01667	7.64	63.64	.01571	8.08	67.31	.01486
7.21	60.06	.01665	7.65	63.72	.01569	8.09	67.39	.01484
7.22	60.14	.01663	7.66	63.81	.01567	8.10	67.47	.01482
7.23	60.23	.01660	7.67	63.89	.01565	8.11	67.56	.01480
7.24	60.31	.01658	7.68	63.97	.01563	8.12	67.64	.01478
7.25	60.39	.01656	7.69	64.06	.01561	8.13	67.72	.01477
7.26	60.48	.01653	7.70	64.14	.01559	8.14	67.81	.01475
7.27	60.56	.01651	7.71	64.22	.01557	8.15	67.89	.01473
7.28	60.64	.01649	7.72	64.31	.01555	8.16	67.97	.01471
7.29	60.73	.01647	7.73	64.39	.01553	8.17	68.06	.01469
7.30	60.81	.01644	7.74	64.47	.01551	8.18	68.14	.01468
7.31	60.89	.01642	7.75	64.56	.01549	8.19	68.22	.01466
7.32	60.98	.01640	7.76	64.64	.01547	8.20	68.31	.01464
7.33	61.06	.01638	7.77	64.72	.01545	8.21	68.39	.01462
7.34	61.14	.01636	7.78	64.81	.01543	8.22	68.47	.01460
7.35	61.23	.01633	7.79	64.89	.01541	8.23	68.56	.01459
7.36	61.31	.01631	7.80	64.97	.01539	8.24	68.64	.01457
7.37	61.39	.01629	7.81	65.06	.01537	8.25	68.72	.01455
7.38	61.48	.01627	7.82	65.14	.01535	8.26	68.81	.01453
7.39	61.56	.01624	7.83	65.22	.01533	8.27	68.89	.01452
7.40	61.64	.01622	7.84	65.31	.01531	8.28	68.97	.01450
7.41	61.73	.01620	7.85	65.39	.01529	8.29	69.06	.01448
7.42	61.81	.01618	7.86	65.47	.01527	8.30	69.14	.01446
7.43	61.89	.01616	7.87	65.56	.01525	8.31	69.22	.01445
7.44	61.98	.01613	7.88	65.64	.01523	8.32	69.31	.01443
7.45	62.06	.01611	7.89	65.72	.01522	8.33	69.39	.01441
7.46	62.14	.01609	7.90	65.81	.01520	8.34	69.47	.01439
7.47	62.23	.01607	7.91	65.89	.01518	8.35	69.56	.01438
7.48	62.31	.01605	7.92	65.97	.01516	8.36	69.64	.01436
7.49	62.39	.01603	7.93	66.06	.01514	8.37	69.72	.01434
7.50	62.48	.01601	7.94	66.14	.01512	8.38	69.81	.01432
7.51	62 56	.01598	7.95	66.22	.01510	8.39	69.89	.01431
7.52	62.64	.01596	7.96	66.31	.01508	8.40	69.97	.01429
7.53	62.72	.01594	7.97	66.39	.01506	8.41	70.06	.01427

TABLE 89 (Continued)

Specific Gravity	Weight Per Solid Gallon	One Pound Bulks Gallons	Specific Gravity	Weight Per Solid Gallon	One Pound Bulks Gallons	Specific Gravity	Weight Per Solid Gallon	One Pound Bulks Gallons
8.42	70.14	.01426	8.86	73.80	.01355	9.30	77.47	.01291
8.43	70.22	.01424	8.87	73.89	.01353	9.31	77.55	.01289
8.44	70.31	.01422	8.88	73.97	.01352	9.32	77.64	.01288
8.45	70.39	.01421	8.89	74.05	.01350	9.33	77.72	.01287
8.46	70.47	.01419	8.90	74.14	.01349	9.34	77.80	.01285
8.47	70.56	.01417	8.91	74.22	.01347	9.35	77.89	.01284
8.48	70.64	.01416	8.92	74.30	.01346	9.36	77.97	.01283
8.49	70.72	.01414	8.93	74.39	.01344	9.37	78.05	.01281
8.50	70.81	.01412	8.94	74.47	.01343	9.38	78.14	.01280
8.51	70.89	.01411	8.95	74.55	.01341	9.39	78.22	.01278
8.52	70.97	.01409	8.96	74.64	.01340	9.40	78.30	.01277
8.53	71.05	.01407	8.97	74.72	.01338	9.41	78.39	.01276
8.54	71.14	.01406	8.98	74.80	.01337	9.42	78.47	.01274
8.55	71.22	.01404	8.99	74.89	.01335	9.43	78.55	.01273
8.56	71.30	.01403	9.00	74.97	.01334	9.44	78.64	.01272
8.57	71.39	.01401	9.01	75.05	.01332	9.45	78.72	.01270
8.58	71.47	.01399	9.02	75.14	.01331	9.46	78.80	.01269
8.59	71.55	.01398	9.03	75.22	.01329	9.47	78.89	.01268
8.60	71.64	.01396	9.04	75.30	.01328	9.48	78.97	.01266
8.61	71.72	.01394	9.05	75.39	.01326	9.49	79.05	.01265
8.62	71.80	.01393	9.06	75.47	.01325	9.50	79.14	.01264
8.63	71.89	.01391	9.07	75.55	.01324	9.51	79.22	.01262
8.64	71.97	.01389	9.08	75.64	.01322	9.52	79.30	.01261
8.65	72.05	.01388	9.09	75.72	.01321	9.53	79.38	.01260
8.66	72.14	.01386	9.10	75.80	.01319	9.54	79.47	.01259
8.67	72.22	.01385	9.11	75.89	.01318	9.55	79.55	.01257
8.68	72.30	.01383	9.12	75.97	.01316	9.56	79.63	.01256
8.69	72.39	.01381	9.13	76.05	.01315	9.57	79.72	.01254
8.70	72.47	.01380	9.14	76.14	.01313	9.58	79.80	.01253
8.71	72.55	.01378	9.15	76.22	.01312	9.59	79.88	.01252
8.72	72.64	.01377	9.16	76.30	.01311	9.60	79.97	.01250
8.73	72.72	.01375	9.17	76.39	.01309	9.61	80.05	.01249
8.74	72.80	.01374	9.18	76.47	.01308	9.62	80.13	.01248
8.75	72.89	.01372	9.19	76.55	.01306	9.63	80.22	.01247
8.76	72.97	.01370	9.20	76.64	.01305	9.64	80.30	.01245
8.77	73.05	.01369	9.21	76.72	.01303	9.65	80.38	.01244
8.78	73.14	.01367	9.22	76.80	.01302	9.66	80.47	.01243
8.79	73.22	.01366	9.23	76.89	.01301	9.67	80.55	.01241
8.80	73.30	.01364	9.24	76.97	.01299	9.68	80.63	.01240
8.81	73.39	.01363	9.25	77.05	.01298	9.69	80.72	.01239
8.82	73.47	.01361	9.26	77.14	.01296	9.70	80.80	.01238
8.83	73.55	.01360	9.27	77.22	.01295			
8.84	73.64	.01358	9.28	77.30	.01294			
8.85	73.72	.01356	9.29	77.39	.01292			

TABLE 90

WHITE LEAD

Commercial Name of Pigment	Furnished by	Information Voluntarily Submitted	Specific Gravity	Weight per solid Gallon *	One pound Bulks Gallons **
Basic Carbonate White Lead	Eagle Picher Lead Co.		6.81	56.73	.01763
Sublimed White Lead	Eagle Picher Lead Co.	Lead Sulfate 81.56%, Lead Oxide 12.05%, Zinc Oxide 6.09%	6.39	53.23	.01879
Basic Sulfate White Lead	National Lead Co.		6.44	53.65	.01864
Matheson White Lead	National Lead Co.		6.72	55.98	.01786
Old Dutch White Lead	National Lead Co.		6.81	56.73	.01763
Carter White Lead	National Lead Co.		6.78	56.48	.01771
White Lead	E. I. DuPont de Nemours & Co.		6.70	55.81	.01792
Anaconda White Lead	Anaconda Lead Products Co.	Lead Carbonate 70.2% Lead Hydrate 29.7%	6.79	56.56	.01768

*Weight per solid gallon is obtained by multiplying the specific gravity of a pigment by 8.33 (apparent weight in air of 1 gallon of water at 60° F.)

**Divide 100 by weight per solid gallon to obtain fraction of gallon bulked by 1 lb. of pigment. Move decimal point one place to right for 10 pounds—two places to right for 100 lbs., etc.

TABLE 91

ZINC OXIDES

Commercial Name of Pigment	Furnished by	Information Voluntarily Submitted	Specific Gravity	Weight per solid Gallon	One pound Bulks Gallons
Florence Green Seal Zinc Oxide	New Jersey Zinc Co.	Florence French Process. Zinc Oxide 99.3% Minimum. Impurities & Moisture .7% Maximum.	5.70	47.48	.02106
Florence White Seal Zinc Oxide	New Jersey Zinc Co.	Florence French Process. Zinc Oxide 99.3% Minimum. Impurities & Moisture .7% Maximum.	5.66	47.15	.02121
Florence Red Seal Zinc Oxide	New Jersey Zinc Co.	Florence French Process. Zinc Oxide 99.3% Minimum; Impurities & Moisture .7% Maximum.	5.72	47.65	.02099
U. S. P. Zinc Oxide	New Jersey Zinc Co.		5.70	47.48	.02106
White Seal Imported French Zinc Oxide	Reichard-Coulston, Inc.	Bureau of Standards analysis shows 95.3% Zinc calculated as Zn0 also $C0_2$, Mg0, etc. Pigment probably contains some carbonate.	5.32	44.32	.02256
ZZZ Leaded Zinc Oxide	American Zinc Lead and Smelt. Co.		5.59	46.56	.02148

TABLE 92

LEADED ZINC OXIDES

Commercial Name of Pigment	Furnished by	Information Voluntarily Submitted	Specific Gravity	Weight per solid Gallon	One pound Bulks Gallons
Lehigh Leaded Zinc Oxide	New Jersey Zinc Co.	Zinc Oxide. 60% Minimum Lead as Lead Sulfate 33-37% Impurities and Moisture 1.5% Maximum.	5.95	49.56	.02018
Sterling Leaded Zinc Oxide	New Jersey Zinc Co.	Zinc Oxide. 86.5% Minimum Lead as Lead Sulfate 8-12% Impurities and Moisture .5% Maximum.	5.75	47.90	.02088
Standard Leaded Zinc Oxide	New Jersey Zinc Co.	Zinc Oxide 93% Minimum Lead as Lead Sulfate 3-5.5% Impurities and Moisture 1.5% Maximum.	5.62	46.81	.02136
Superior Leaded Zinc Oxide	New Jersey Zinc Co.	Zinc Oxide 77.5% Minimum Lead as Lead Sulfate 18.5-21% Impurities and Moisture 1.5% Maximum.	5.92	49.31	.02028
Lehigh Leaded Zinc Oxide	Mineral Point Zinc Co.		5.88	48.98	.02042
Sterling Leaded Zinc Oxide	Mineral Point Zinc Co.		5.73	47.73	.02095
Standard Leaded Zinc Oxide	Mineral Point Zinc Co.		5.58	46.48	.02151
Superior Leaded Zinc Oxide	Mineral Point Zinc Co.		5.77	48.06	.02081
Ozlo Leaded Zinc Oxide	Sherwin-Williams Co.	Zinc Oxide 65% Lead Sulfate 35%	5.94	49.48	.02021
Z Leaded Zinc Oxide	American Zinc Lead and Smelt. Co.		5.66	47.15	.02121
ZZ Leaded Zinc Oxide	American Zinc Lead and Smelt. Co.		5.61	46.73	.02140

TABLE 92 (Continued)

LEADED ZINC OXIDES (Continued)

Commercial Name of Pigment	Furnished by	Information Voluntarily Submitted		Specific Gravity	Weight per solid Gallon	One pound Bulks Gallons
Zenith Leaded Zinc Oxide Arctic Brand	American Metals Co.	Zinc Oxide	65.26	5.85	48.73	.02052
		Lead Sulfate	24.09			
		Lead Oxide	9.09%			
		Chlorine	0.08			
		Zinc Sulfate	0.48			
		Sulfur Dioxide	0.02			
		Water	0.22			
Zenith Leaded Zinc Oxide Polar Brand	American Metal Co.	Zinc Oxide	95.5	5.60	46.65	.02144
		Lead Sulfate	2.9			
		Chlorine	0.08			
		Zinc Sulfate	0.83			
		Sulfur Dioxide	0.01			
		Water	0.14			

TABLE 93

LITHOPONE

Commercial Name of Pigment	Furnished by	Information Voluntarily Submitted	Specific Gravity	Weight per solid Gallon	One pound Bulks Gallons
Sterling Lithopone	Midland Chem. Co.		4.31	35.90	.02786
Standard AA W Lithopone	New Jersey Zinc Co.	Zinc Sulfide 27% Minimum; Barium Sulfate 70.5% Maximum; Impurities and Moisture 1.5% Maximum	4.31	35.90	.02786
Green Label Lithopone	New Jersey Zinc Co.	Zinc Sulfide 27% Minimum; Barium Sulfate 71% Maximum; Impurities and Moisture 1.5% Maximum	4.29	35.74	.02798
Green Seal Beckton White Lithopone	E. I. DuPont de Nemours and Co.		4.30	35.82	.02792
Ponolith AXI Lithopone	Krebs Pigment and Chem. Co.	Zinc Sulfide 27.06%; Barium Sulfate 71.86%; Total Zinc 19.68%; Zinc Oxide 1.90%	4.31	35.90	.02786
Zincolith Lithopone	Chemical Pigments Corp.	Barium Sulfate 70.65%; Zinc Sulfide 27.68%; Zinc Oxide 1.29%; Moisture .05%	4.29	35.74	.02798
Lithopone	Sherwin Williams Co.	Zinc Sulfide 30% Barium Sulfate 70%	4.28	35.65	.02805
Marbon White Lithopone	Minerals Ref. and Chem. Corp.	Zinc Sulfide 28% Barium Sulfate 70% Zinc Oxide 2%	4 27	35.57	.02811

TABLE 94

MISCELLANEOUS WHITE PIGMENTS

Commercial Name of Pigment	Furnished by	Information Voluntarily Submitted	Specific Gravity	Weight per solid Gallon	One pound Bulks Gallons
B X Titanox	Titanium Pigment Co., Inc. Niagara Falls	Titanium Oxide 16% Barium Sulfate 84% 100%	4.31	35.90	.02786
B X X Titanox	Titanium Pigment Co., Inc. Niagara Falls	Titanium Oxide 25% Barium Sulfate 75% 100%	4.28	35.65	.02805
Asbestine	International Pulp Co.		2.85	23.74	.04212
Gypsum	(H.B. Hughes)		2.36	19.66	.05086
"Dunkru" Magnesium Silicate	Dunn & Kruse		2.72	22.65	.04413
A XXX Metro Nite	S. C. Johnson & Son	Magnesium-Aluminum Silicate. Finely divided and white.	2.94	24.49	.04083

Name in brackets indicates makers name as furnished by grinder.

TABLE 95

BARIUM SULFATE

Commercial Name of Pigment	Furnished by	Information Voluntarily Submitted	Specific Gravity	Weight per solid Gallon	One pound Bulks Gallons
Barytes "A"	Point Milling and Mfg. Co.	Barium Sulfate 99.52%; Moisture .05%; Silica .42%; Sesqui-Oxide of Iron .005%; Alumina, Lime and Carbonic acid—None.	4.44	36.99	.02703
Barytes "B"	Point Milling and Mfg. Co.	Barium Sulfate 99.52%; Moisture .05%; Silica .42%; Sesqui-Oxide of Iron .005%; Alumina, Lime and Carbonic acid—None.	4.45	37.07	.02698
Barytes "C"	Point Milling and Mfg. Co.	Barium Sulfate 99.52%; Moisture .05%; Silica .42%; Sesqui-Oxide of Iron .005%; Alumina, Lime and Carbonic acid—None.	4.45	37.07	.02698
Foam "A" Barytes	J. C. Finck Mineral Milling Co.		4.46	37.15	.02692
Cream Floated Lead Bloom Barytes	Nulsen Corp.		4.46	37.15	.02692
Blanc Fixe Standard Quality	Rollin Chem. Co.	Barium Sulfate 98.08%; Barium Carbonate .76%; Alkali .04%; Loss on Ignition 1.06%; Moisture .06%; Direct Precipitation.	4.28	35.65	.02805

TABLE 96

WHITE CLAY, CHINA CLAY

(Aluminum Silicate)

Commercial Name of Pigment	Furnished by	Information Voluntarily Submitted	Specific Gravity	Weight per solid Gallon	One pound Bulks Gallons
China Clay	Tamms Silica Co.		2.62	21.82	.04583
Alumicla China Clay	Keystone Minerals Co.	Silica 45.94%; Alumina 36.82%; Titanium Oxide 1.25%; Calcium Oxide .31%; Iron Oxide 1.05%; Sodium Oxide .30%; Magnesium Oxide .10%; Potassium Oxide .10%	2.62	21.82	.04583
Pure White Clay	American China Clay Co. American Kaolin Co., Agents		2.60	21.66	.04617
A A Lump Clay	(Phila. Clay Co.)		2.65	22.07	.04531

TABLE 97

TALC

(Magnesium Silicate)

Commercial Name of Pigment	Furnished by	Information Voluntarily Submitted	Specific Gravity	Weight per solid Gallon	One pound Bulks Gallons
(A) Talc.	Magnesia Talc Co.	200 Mesh and Finer. Silica 60%; Alumina 4%; Pyrite 3.5%; Magnesia 28%; Water 4.5%	2.80	23.32	.04288
(D. C.) Talc.	Magnesia Talc Co.	300 Mesh and Finer. Silica 60%; Alumina 4%; Pyrite 3.5%; Magnesia 28%; Water 4.5%	2.78	23.16	04318
No. 110 Blue Ridge Talc.	Franklin Soapstone Products Corp.	Silica .43.24%; Alumina .9.22%; Ferrous Oxide 5.47%; Ferric Oxide 6.72%; Titanic Acid .48%; Manganese Oxide .61%; Lime 1.66%; Magnesia 25.62%; Sodium Oxide .34%; Carbon Dioxide .79%; Sulphur .09%	2.88	23.99	.04168
13-G Talc.	American Minerals Co.	Magnesia 26.3%; Alumina 15.9%; Silica 33.9%; Iron 2.9%; Loss on Ignition 21.0%	2.88	23.99	04168

TABLE 98

WHITING

Commercial Name of Pigment	Furnished by	Information Voluntarily Submitted	Specific Gravity	Weight per solid Gallon	One pound Bulks Gallons
Gilders Bolted Whiting	Heath and Milligan		2.72	22.66	.04413
White Mineral Primer	Heath and Milligan		2.72	22.66	.04413
G-5946 Whiting	E. M. & F. Waldo		2.71	22.57	.04431
G 5947 Whiting	E. M. & F. Waldo		2.71	22.57	.04431
T. C. Floated Whiting	Tamms Silica Co.	Calcium Carbonate 99.06%; Magnesium Carbonate .13%; Alumina .22%; Loss on Ignition .08%; Silica .51%; Alkali .056%	2.74	22.82	.04382
Reliance Whiting	Tamms Silica Co.	Calcium Carbonate 95.91%; Magnesium Carbonate .88%; Alumina 1.41%; Silica 1.39%; Iron .41%; Alkali .077%	2.72	22.66	.04413
Danish Gilders Whiting	Tamms Silica Co.	Calcium Carbonate 98.83%; Magnesium Carbonate .65%; Iron and Alumina .18%; Silica .34%; Alkali .049%	2.71	22.57	.04431
Calcite White Primer	Tamms Silica Co.	Calcium Carbonate 99.06%; Magnesium Carbonate .13%; Alumina .22%; Silica .51%; Loss on Ignition .08%	2.73	22.74	.04398
Extra Gilders Whiting	Nulsen Corp.		2.71	22.57	.04431

TABLE 98 (Continued)

WHITING (Continued)

Commercial Name of Pigment	Furnished by	Information Voluntarily Submitted	Specific Gravity	Weight per solid Gallon	One pound Bulks Gallons
White Mineral Primer	Nulsen Corp.		2.71	22.57	.04431
Gilders Whiting	(B. Moore & Co.)		2.71	22.57	.04431
Nulsen Gilders Whiting	(Nulsen Corp.)		2.71	22.57	.04431
Bolted Gilders Whiting	H. F. Taintor Mfg. Co.		2.71	22.57	.04431

TABLE 99

SILICA

Commercial Name of Pigment	Furnished by	Information Voluntarily Submitted	Specific Gravity	Weight per solid Gallon	One pound Bulks Gallons
No. 68 Crystalline Silica	Tamms Silica Co.	Silica 99.8%	2.65	22.07	.04531
Velveteen Silica*	Tamms Silica Co.	200 Mesh Silica 99%; Alumina .2%; Magnesium Oxide .4%; Undetermined and Moisture .4%	2.64	21.99	.04548
Gold Bond Silica*	Tamms Silica Co.	450 Mesh Silica 99%; Alumina .2%; Magnesium Oxide .4%; Undetermined and Moisture .4%	2.65	22.07	.04531
No. 10 Snow White Crystalline Silica	Tamms Silica Co.	Silica 99.8%	2.65	22.07	.04531
Silver Bond Silica*	Tamms Silica Co.	350 Mesh Silica 99%; Alumina .2%; Magnesium Oxide .4%; Undetermined and Moisture .4%	2.64	21.99	.04548
No. 1 Silica	International Silica Co.	Silica 99.4% Alumina .22% Iron Oxide .14% Calcium and Magnesium Oxides None; Amorphous Decomposed	2.65	22.07	.04531
No. 160 Soft Silica	(Innis-Speiden Co.)		2.64	21.99	.04548
Pure Carrara Silica	Isco-Bautz Co.	Silica 99.5%; 99%-350 Mesh All 300 Mesh; Amorphous Decomposed Quartz ground in water Floatation Precipitation.	2.65	22.07	.04531
No. 400 Mesh Pure Silica	(Eureka Flint & Spar Co.)		2.66	22.16	.04513

*Amorphhous Decomposed Quartz—Water Ground and Floated.

TABLE 99 (Continued)

SILICA (Continued)

Commercial Name of Pigment	Furnished by	Information Voluntarily Submitted		Specific Gravity	Weight per solid Gallon	One pound Bulks Gallons
Lithowhite Silex	Lithowhite Silex Co. Still River, Conn.	Pure Quartz Silica		2.66	22.16	.04513
No. 200 Kallite Silica Infusorial Earth.	Tamms Silica Co.	S_1O_2 Fe_2O_3 MgO Loss on Ignition	88.39 6.31 .38 4.92 100.00	1.95	16.24	.06158

TABLE 100

MINERAL FILLERS

Commercial Name of Pigment	Furnished by	Information Voluntarily Submitted	Specific Gravity	Weight per solid Gallon	One pound Bulks Gallons
Keystone Filler	Heath and Milligan		2.72	22.66	.04413
Keystone Black Filler No. 1	Keystone Minerals Co.	98.5%-300 Mesh, nearly all 200 Mesh. Silica 59.24% Alumina 17.10%; Iron 7.26%; Carbon 14.30%; Volatile and Water 2.00%	2.71	22.57	.04431
Keystone Filler No. 1	(Keystone Min. Co.)		2.71	22.57	.04431
XXX Keystone Filler	(Keystone Pt. & Filler Co.)		2.69	22.41	.04462

TABLE 101

RED LEAD, LITHARGE, BLUE LEAD

Commercial Name of Pigment	Furnished by	Information Voluntarily Submitted	Specific Gravity	Weight per solid Gallon	One pound bulks Gallons
AAA Orange Mineral	Eagle Picher Co.	Pb_3O_4 93%	8.91	74.22	.01347
Sublimed Blue Lead	Eagle Picher Co.	Lead Sulfate 49.75% Lead Sulfide 6.05% Zinc Oxide 1.00% Lead Oxide 37.68% Lead Sulfite 2.75% Undetermined 2.77%	6.67	55.56	.01800
Sublimed Litharge	Eagle Picher Co.	17 grams to cubic inch	9.53	79.38	.01260
Paint Grinders Red Lead	Eagle Picher Co.	Pb_3O_4 88.8%	8.90	74.14	.01349
96% Red Lead	National Lead Co.	Pb_3O_4 96%	8.70	72.47	.01380
87% Red Lead	National Lead Co.	Pb_3O_4 87%	8.98	74.80	.01337
Litharge	E. I. DuPont de Nemours & Co.		9.31	77.55	.01289
Red Lead	E. I. DuPont de Nemours & Co.		8.85	73.72	.01356
No. 4 Orange Mineral	E. I. DuPont de Nemours & Co.		8.35	69.56	.01438
Tours Orange Mineral	Reichard Coulston, Inc.		8.78	73.14	.01367
Lead Oxide	Metals Disintegrating Co.	Manufactured from Standard Brands Pig Lead and Contains same impurities.	9.62	80.13	.01248

TABLE 102

LAKES AND TONERS

Commercial Name of Pigment	Furnished by	Information Voluntarily Submitted	Specific Gravity	Weight per solid Gallon	One pound Bulks Gallons
Toluidine Lake	Sherwin-Williams Co.	Toluidine Toner.	2.79	23.24	.04303
Brilliant Orange Lake	Sherwin-Williams Co.		2.64	21.99	.04548
Chrome Scarlet	Sherwin-Williams Co.	Scarlet Lead Chromate American Vermilion.	6.54	54.48	.01836
Permanent Geranium Lake	Sherwin-Williams Co.	Production discontinued by manufacturers.	2.47	20.58	.04859
Burgundy Lake	Sherwin-Williams Co.	Color modified by producer since sample was submitted.	2.80	23.32	.04288
Para Toner L	Sherwin-Williams Co.	Light Shade of Para. 3-5% Base Material Probably Lime Carbonate	1.50	12.50	.08000
Para Toner Dark	Sherwin-Williams Co.	Pure Para Toner Deep Shade.	1.62	13.49	.07413
Maroon Toner	Sherwin-Williams Co.		1.74	14.49	.06901
Para Lake D Single Strength	Sherwin-Williams Co.	7% Para Toner precipitated on inert base chiefly Calcium Carbonate.	2.73	22.74	.04398
Oriental Red L	Sherwin-Williams Co.	10% Lt. Shade Para Toner precipitated on 90% inert base composed of 60% Carbonate Lime and 40% Barium Sulfate.	2.65	22.07	.04531
Azoline "O" Para Red	(Kohnstamm & Co.)		3.74	31.15	.03210
Nopal Maroon 10163	Devoe Reynolds	Lake Color Organic Dye Blanc Fixe Base.	3.55	29.57	.03382
Nopal Maroon 10201	Devoe Reynolds	Lake Color Organic Dye Blanc Fixe Base.	3.65	30.40	.03289
Chatemuc Lake 2729	Devoe Reynolds	Lake Color, Natural Dye. Calcium Carbonate Base	2.12	17.66	.05663
Rose Pink 2727	Devoe Reynolds	Lake Color Natural Dye. Calcium Carbonate Base	2.43	20.24	.04941

TABLE 102 (Continued)

LAKES AND TONERS (Continued)

Commercial Name of Pigment	Furnished by	Information Voluntarily Submitted	Specific Gravity	Weight per solid Gallon	One pound Bulks Gallons
Coach Red D	G. Siegle Corp.	6% Pure Para Red on 94% Calcium Carbonate	2.56	21.32	.04690
Mode Red D	G. Siegle Corp.	6% Pure Para Red on 94% Barium Sulfate	4.01	33.40	.02994
Maroon Lake	(A. B. Ansbacker & Co.)		3.78	31.49	.03176
Perfectona Extra Light	F. O. Pierce Co.	100% Pure Para Red	1.51	12.58	.07949
Rubeina Uzatona Light	F. O. Pierce Co.	100% Pure Para Red	1.51	12.58	.07949
Uzatona Dark	F. O. Pierce Co.	100% Pure Para Red	1.47	12.25	.08163
Uzatona Toluidin Extra Light	F. O. Pierce Co.	100% Pure Toluidin Red	1.47	12.25	.08163
Scarlet Lake No. 116		Barium Sulfate 70.% Scarlet Dye 22. Aluminum Hydrate 8.	3.25	27.91	.03583
Lithia Vermilion Dark No. 129		Barium Sulfate 57. % Orange Mineral 38. Para Toner 3. Precipitated Eosin 1.5	5.09	42.40	.02358
Para Red 8062 B	Devoe Reynolds	100% pure Para Red	1.51	12.58	07949
Para Red 10169 B	Devoe Reynolds	20% Para Red on Barium Carbonate, Aluminum Hydrate and Blanc Fixe Base	2.99	24.91	.04014
Red 467C	Devoe Reynolds	Lake Color Organic Dye. Blanc Fixe Base	3.43	28.57	.03500
Quaker Red 9479	Devoe Reynolds	16% Para Red on Barytes	3.21	26.74	.03740

TABLE 103

TUSCAN RED

Commercial Name of Pigment	Furnished by	Information Voluntarily Submitted	Specific Gravity	Weight per solid Gallon	One pound Bulks Gallons
No. 1 Tuscan Red	Reichard-Coulston, Inc.	Indian Red 31.5-35.0% Barium Sulfate 56.0-58.0% Zinc 3.0-10.0%; Dye 9.1%	3.97	33.07	.03024
No. 60 Tuscan Red	Reichard-Coulston, Inc.	Iron Oxide 7.3%; Dye 12.4% Barium Sulfate 80.3%	3.93	32.74	.03054
No. 776 Tuscan Red	Reichard-Coulston, Inc.	Barium Sulfate 63.0-65.0% Lead Oxide 1.75%; Dye 9.95% Lime Carb. 26.0-28.0%	3.51	29.24	.03420

TABLE 104

RED AND BROWN OXIDES

Commercial Name of Pigment	Furnished by	Information Voluntarily Submitted	Specific Gravity	Weight per solid Gallon	One pound Bulks Gallons
G 4007 Red Oxide	E. M. & F. Waldo	Iron Oxide 26.12% Balance Silica and Insoluble	3.02	25.16	.03974
G 4835 Red Oxide	E. M. & F. Waldo	Aluminum Silicate 66.85% Iron Oxide 19.26% Alumina 9.12%; Magnesia .17% Loss on Ignition 4.6%	2.85	23.74	.04212
G 5435 Red Oxide	E. M. & F. Waldo	Iron Oxide 65%; Other Metallic Oxides, 25%	3.90	32.49	.03078
G 5183 Brown Oxide	E. M. & F. Waldo	Iron Oxide 54.72%; Alumina 6.15%; Manganese Oxide .25%; Calcium Oxide .34%; Magnesium Oxide .16%; Insoluble Silicates 31.10%. Loss on Ignition 4.67%; Moisture 2.61%	3.42	28.49	.03510
G 5895 Brown Oxide	E. M. & F. Waldo	Iron Oxide 48.00%; Silica 28.43%; Alumina 10.80%; Sulfur None; Manganese Oxide 1.86%; Calcium Oxide .96%; Loss on Ignition 8.92%	3.27	27.24	.03671
Mineral Brown	Prince Mfg. Co.	Sesquioxide Iron 47.67% Alumina 9.46%; Silica 35.75%; Lime 2.44%; Magnesia 1.39%; Manganese Oxide 1.39%; Loss on Ignition 1.60%; Undetermined .29%	3.34	27.82	.03595
No. 44 Venetian Red	Prince Mfg. Co.	Iron Oxide 20%; Calcium Sulfate 80%	3.05	25.41	.03935
No. 66 Red Oxide	Prince Mfg. Co.	Iron Oxide 30%; Gypsum 60%; Lime Carbonate 5-10%	3.32	27.66	.03615
No. 88 Red Oxide	Prince Mfg. Co.	Iron Oxide 40% Gypsum 60%	3.47	28.91	.03459
Princes Metallic	(P. M. P. Co.) Devoe Reynolds		3.39	28.24	.03541

TABLE 104 (Continued)

RED AND BROWN OXIDES (Continued)

Commercial Name of Pigment	Furnished by	Information Voluntarily Submitted	Specific Gravity	Weight per solid Gallon	One pound Bulks Gallons
No. 58 Red Oxide	Reichard-Coulston, Inc.	Ferric Oxide 93.6% Free Acid .15%	4.93	41.07	.02435
No. 686 Red Oxide	Reichard-Coulston, Inc.	Ferric Oxide 81.4%; Loss on Ignition 3.8%; Calcium Oxide 1.4%; Alumina 9.2%; Silica 3.8%	4.50	37.49	.02667
E 6 Oxide	(G. S. Mepham)		4.42	36.82	.02716
33% Oxide	(G. S. Mepham)		3.33	27.74	.03605
G 2806 Spanish Oxide	(E. M. & F. Waldo)	88-90% Fe_2O_3 6% S_1O_2 Balance $CaCO_3$ and Clay	4.54	37.82	.02644
Red Oxide	Frazer Paint Co.	Amorphous Clay From Decomposed Mica Base 14.6% Fe_20_3	2.71	22.57	.04431
Yellow Ferric Oxide	(Chicago Color Works)		3.88	32.32	.03094
12 L Venetian Red	(C. K. W. Co.) Devoe Reynolds	Iron Oxide 20.0% Calcium Sulphate 75.0% Calcium Carbonate 5.0%	3.03	25.24	.03962
1212 Venetian Red	(C. K. W. Co.) Devoe Reynolds	Iron Oxide 40.0% Calcium Sulphate 60.0 (Fully dehydrated)	3.21	26.74	.03740
1195 B Venetian Red	(C. K. W. Co.) Devoe Reynolds	Iron Oxide 98.0%	5.13	42.73	.02340

BULKING VALUES AND YIELDS OF PIGMENTS AND LIQUIDS

TABLE 105

INDIAN RED

Commercial Name of Pigment	Furnished by	Information Voluntarily Submitted	Specific Gravity	Weight per solid Gallon	One pound Bulks Gallons
No. 20 Indian Red	Reichard-Coulston, Inc.	Ferric Oxide 92.1% Free Acid None. Lime .3% Magnesia .04%; Silica 2.0%	4.93	41.07	.02435
No. 25 Indian Red	Reichard-Coulston, Inc.	Ferric Oxide 80.00% Free Acid None.	4.59	38.23	.02616
No. 43 Indian Red	Reichard-Coulston, Inc.	Ferric Oxide 70.00% Free Acid None.	4.44	36.99	.02703
Indian Red L	(G. S. Mepham)		5.15	42.90	.02331
Indian Red M	(G. S. Mepham)		5.14	42.82	.02335
Indian Red D	(G. S. Mepham)		5.07	42.23	.02368
No. 59 Indian Red	(L. N. Co.) Devoe Reynolds		5.02	41.82	.02391
No. 65 Indian Red	(L. N. Co.) Devoe Reynolds		5.11	42.57	.02349

TABLE 106

OCHRES

Commercial Name of Pigment	Furnished by	Information Voluntarily Submitted	Specific Gravity	Weight per solid Gallon	One pound Bulks Gallons
No. 9 Golden Ochre	Reichard-Coulston, Inc.	Calcium Sulfate 56.7%; Lead Chromate 7.58%; Silica 15.1%; Alumina 1.3%; Ferric Oxide 2.5%; Loss on Ignition 17.5%	2.76	22.99	.04350
FAR No. 1 French Ochre	Reichard-Coulston, Inc.	Silica 48.1%; Alumina 19.1% Ferric Oxide 22.3%; Loss on Ignition 9.8%; Calcium Oxide .4%	2.89	24.07	.04155
R. C. No. 47 French Ochre	Reichard-Coulston, Inc.	Silica 48.6%; Alumina 18.8%; Ferric Oxide 21.2%; Calcium Oxide .4%; Loss on Ignition 10.8%	2.88	23.99	.04168
Golden Yellow Ochre	Tamms Silica Co.	Silica 65.48%; Alumina 19.54%; Iron Oxide 14.98%; Magnesia and Lime Trace.	2.77	23.07	.04335
Natural Grey Ochre	Tamms Silica Co.	Silica 69%; Alumina 24% Ferric Oxide 7%	2.75	22.91	.04365
G 5927 Yellow Ochre	E. M. & F. Waldo	Iron Oxide .... 18.23 Silica .... 54.67 Alumina .... 15.21 Lime .... .52 Magnesia .... .36 Moisture @ 100° C. .... 1.58 Loss on Ignition 8.22	2.83	23.57	.04243
California Ochre	West Coast Kalsomine Co.	Yellow Ferric Oxide.	3.66	30.49	.03280
Ferrite Yellow Lemon	(N. F. Co.) Devoe Reynolds		4.20	34.99	.02858
Ferrite Yellow Orange	(N. F. Co.) Devoe Reynolds		3.80	31.65	.03160

TABLE 107

UMBERS

Commercial Name of Pigment	Furnished by	Information Voluntarily Submitted	Specific Gravity	Weight per solid Gallon	One pound Bulks Gallons
Turkey No. 14 Burnt Umber	Reichard-Coulston, Inc.	Ferric Oxide 55.61%; Alumina 6.34%; Silica 15.40%; Calcium Oxide 3.12%; Manganese Dioxide 12.85%; Loss on Ignition 6.43%	3.68	30.65	.03263
No. 15 Burnt Turkey Umber	Reichard-Coulston, Inc.	Ferric Oxide 56.54%; Alumina 4.02%; Silica 16.23%; Calcium Oxide 3.78%; Manganese Dioxide 11.15%; Loss on Ignition 7.81%	3.83	31.90	.03135
No. 17 Burnt Turkey Umber	Reichard-Coulston, Inc.	Ferric Oxide 40.03%; Alumina 7.11%; Silica 19 20%; Calcium Oxide 3.14%; Manganese Dioxide 13.89%; Loss on Ignition 16.14%	3.52	29.32	.03411
G. Raw Umber	(J. Lee Smith and Co.)		2.60	21.66	.04617
G. D. Raw Umber	(J. Lee Smith and Co.)		2.70	22.49	.04446
M Burnt Umber	(J. Lee Smith and Co.)		3.77	31.40	.03185
M. Raw Turkey Umber	(J. L. S. Co.) Devoe Reynolds		3.49	29.07	.03440

TABLE 108

SIENNAS

Commercial Name of Pigment	Furnished by	Information Voluntarily Submitted	Specific Gravity	Weight per solid Gallon	One pound Bulks Gallons
G. Raw Sienna	(J. Lee Smith and Co.)		3.27	27.24	.03671
O. H. S. Burnt Sienna	(J. Lee Smith and Co.)		3.96	32.99	.03031
Frazer Sienna	Frazer Paint Co.	Raw Sienna 77.7% Fe_2O_3	3.87	32.24	.03102

TABLE 109

YELLOWS

Commercial Name of Pigment	Furnished by	Information Voluntarily Submitted	Specific Gravity	Weight per solid Gallon	One pound Bulks Gallons
C. P. Yellow Light	(F. L. Lavanburg)		6.00	49.98	.02001
C. P. Yellow Med.	(F. L. Lavanburg)		5.90	49.15	.02034
C. P. Yellow Lt. Orange	(F. L. Lavanburg)		6.70	55.81	.01792
C. P. Yellow Dk. Orange	(F. L. Lavanburg)		6.91	57.56	.01737
C. P. Chrome Yellow Lt.	G. Siegle Corp.	Precipitate with considerable Lead Sulfate to obtain shade.	6.08	50.65	.01974
C. P. Chrome Yellow Med.	G. Siegle Corp.	Precipitated with small amount of Lead Sulfate to obtain shade.	6.13	51.06	.01958
C. P. Yellow Light	Sherwin-Williams Co.	Acetate type Lemon Shade Chrome Yellow.	6.01	50.06	.01998
C. P. Yellow M.	Sherwin-Williams Co.	Acetate type Medium Shade Chrome Yellow.	6.05	50.40	.01984
C. P. Yellow O.	Sherwin-Williams Co.	Acetate type Orange Shade Chrome Yellow.	6.30	52.48	.01905
C. P. Yellow D. O.	Sherwin-Williams Co.	Acetate type Deep Orange Shade Chrome Yellow.	5.58	46.48	.02151
Primrose Yellow	Sherwin-Williams Co.	Extra Light Shade Chrome Yellow.	6.03	50.23	.01991
Dutch Pink	(F. L. Lavanburg)		2.48	20.66	.04840
C. P. Chrome Yellow Lt. 4347	Devoe Reynolds	Acetate Type Pure	5.79	48.23	.02073
C. P. Chrome Yellow Med. 4511C	Devoe Reynolds	Acetate Type Pure	5.58	46.48	.02151
C. P. Chrome Yellow Dp. 2285	Devoe Reynolds	Acetate Type Pure	6.57	54.73	.01827
C. P. Chrome Yellow Orange 7470	Devoe Reynolds	Acetate Type Pure	6.89	57.39	.01742

TABLE 110

BLUES

Commercial Name of Pigment	Furnished by	Information Voluntarily Submitted	Specific Gravity	Weight per solid Gallon	One pound Bulks Gallons
Sp. Chinese Blue	Sherwin-Williams Co.	Ferric Ferro Cyanide Pure	1.88	15.66	.06386
Sp. Bronze Blue	Sherwin-Williams Co.	Ferric Ferro Cyanide Pure Pure Prussian Blue without inert paste.	1.82	15.16	.06596
Sp. Sol. Prussian Blue	Sherwin-Williams Co.	Ferric Ferro Cyanide Pure.	1.87	15.58	.06418
C. P. Milori Blue	G. Siegle Corp.	Made only from Prussiate of Soda and Iron Salts. No Inert Base.	1.84	15.33	.06523
Chinese Blue	(F. L. Lavanburg)		1.80	14.99	.06671
C. U. Ultramarine Blue	(Ultramarine Company)		2.33	19.41	05152
S. 4 Ultramarine Blue	(Ultramarine Company)		2.89	24.07	.04155
H. M. Ultramarine Blue	(Ultramarine Company)		2.36	19.66	.05086
L. L. Ultramarine Blue	(Standard Ultramarine Co.)		2.32	19.33	.05173
L. L. Ultramarine Blue	Heath and Milligan	Made by Standard Ultra Marine Co.	2.35	19.58	.05107

TABLE 111

GREENS

Commercial Name of Pigment	Furnished by	Information Voluntarily Submitted	Specific Gravity	Weight per solid Gallon	One pound Bulks Gallons
Peerless Green L	(F. L. Lavanburg)		3.92	32.65	.03062
Peerless Green M	(F. L. Lavanburg)		3.88	32.32	.03094
Peerless Green D	(F. L. Lavanburg)		3.87	32.24	.03102
C. P. Chrome Green L*	G. Siegle Corp.		5.08	42.32	.02363
C. P. Chrome Green D*	G. Siegle Corp.		3.57	29.74	.03362
C. P. Grinders Green 2900 L*	G. Siegle Corp.		4.56	37.98	.02633
C. P. Grinders Green 2900 M*	G. Siegle Corp.		4.29	35.74	.02798
C. P. Grinders Green 2900 D*	G. Siegle Corp.		4.19	34.90	.02865
French Crown Green D	Sherwin-Williams Co.	Inert Base Barium Sulfate Chrome Green 25% Pure.	4.05	33.74	.02964
Chromium Oxide	(G. Siegle Corp.)		4.97	41.40	.02415
Paris Green	Devoe Reynolds		3.22	26.82	.03729

*All C. P. Greens contain different amounts of Chrome Yellow and Blue to produce desired shade. Lighter shades contain about 10% of Blue and 90% of Yellow. Darker shades contain about half and half of each color.

TABLE 111 (Continued)

GREENS (Continued)

Commercial Name of Pigment	Furnished by	Information Voluntarily Submitted	Specific Gravity	Weight per solid Gallon	One pound Bulks Gallons
Yellow Tone C. P. Green Shade L.	Sherwin-Williams Co.	Light Shade Acetate Green.	5.38	44.82	.02231
Yellow Tone C. P. Green Shade M.	Sherwin-Williams Co.	Med. Shade Acetate Green.	4.18	34.82	.02872
Yellow Tone C. P. Green Shade Dk.	Sherwin-Williams Co.	Dark Shade Acetate Green.	3.71	30.90	.03236
Blue Tone C. P. Green Shade M.	Sherwin-Williams Co.	Med. Shade C. P. Greens.	4.69	39.07	.02560
Blue Tone C. P. Green Shade Dk.	Sherwin-Williams Co.	Dark Shade C. P. Greens.	4.00	33.32	.03001
Green Earth	Tamms Silica Co.	Silica 69%; Alumina 24% Iron Oxide 7%	2.76	22.99	.04350
Green Earth	Heath and Milligan	Imported from Germany. Not used now.	2.74	22.82	.04382
Limeproof Green G	Heath and Milligan	10% Malachite Green and Auramine Precipitated on 90% Aluminum Silicate	2.75	22.91	04365
Limeproof Green B	Heath and Milligan	10% Emerald Green Precipitated on 90% Aluminum Silicate	2.85	23.74	04212
C. P. Chrome Green Lt. 2303 A	Devoe Reynolds	Acetate Type Pure	5.07	42.23	.02368
C. P. Chrome Green Med. 2303 B	Devoe Reynolds	Acetate Type Pure	4.86	40.48	.02470
C. P. Chrome Green Dp. 2303 C.	Devoe Reynolds	Acetate Type Pure	4.58	38.15	.02621
Saxon Green 6320	Devoe Reynolds	Nitrate Type Pure	4.63	38.57	.02593

TABLE 112

BLACKS

Commercial Name of Pigment	Furnished by	Information Voluntarily Submitted	Specific Gravity	Weight per solid Gallon	One pound Bulks Gallons
Special Grinders No. 8 Lampblack	Wilckes-Martin Wilckes Co.	Highest and Strongest Grade. Carbon, 96.81%; gaseous, .38%; Oil, 1.56%; Moisture, .97%; Ash, .21%.	1.78	14.83	.06743
Blue Tone No. 12 Lampblack	Wilckes-Martin Wilckes Co.	Medium Grade Carbon, 97.21%; Gaseous, .32%; Oil, 1.43%; Moisture, .81%; Ash, .14%.	1.78	14.83	.06743
Bear Velvet No. 10 Lampblack	Wilckes-Martin Wilckes Co.	Regular Run. Carbon 97.48%; Gaseous, .35%; Oil, 1.28%; Moisture .67%; Ash, .09%.	1.78	14.83	.06743
Trimount Germantown Lampblack	Samuel Cabot	Carbon 97.9% Oil 1.5 Moisture .5 Ash .1 100.0%	1.77	14.74	.06784
Old Abe Lampblack	Bihn & Wolff		1.77	14.74	.06784
No. 1 Grinders Lampblack	Bihn & Wolff		1.75	14.58	.06859
Charlton Velvet Lampblack	Bihn & Wolff		1.78	14.83	.06743
Carbon Black	Binney and Smith	Carbon 98.6%; Moisture 1.3%; Iron Trace.	1.80	14.99	.06671
Kalista Carbon Black	Godfrey L. Cabot	High grade Roller Process Black chiefly used for half tone printing Inks. Pure Black with possibly only 80% carbon.	1.87	15.58	.06418
Auk Carbon Black	Godfrey L. Cabot	Medium grade black of good color.	1.81	15.08	.06631

TABLE 112 (Continued)

BLACKS (Continued)

Commercial Name of Pigment	Furnished by	Information Voluntarily Submitted	Specific Gravity	Weight per solid Gallon	One pound Bulks Gallons
Sunset Elf Carbon Black	Godfrey L. Cabot	Extra Fluid Black chiefly useful for news inks not as good color as Auk but more fluid in ink than any Black other than Roller Process Black.	1.74	14.49	.06901
No. 1 Lampblack	L. Martin Co.	Ash 4-5%	1.51	12.58	.07949
No. 2 Lampblack	L. Martin Co.	Ash Trace. Generally less than ½ or ¼ or 1% ash.	1.79	14.91	.06707
No. 3 Lampblack	L. Martin Co.	Ash Trace. Generally less than ½ or ¼ or 1% ash.	1.80	14.99	.06671
No. 4 Lampblack	L. Martin Co.	Ash, Trace. Generally less than ½ or ¼ or 1% ash.	1.76	14.66	.06821
No. 5 Lampblack	L. Martin Co.	Ash, Trace. Generally less than ½ or ¼ or 1% ash.	1.78	14.83	.06743
No. 6 Lampblack	L. Martin Co.	Ash Trace. Generally less than ½ or ¼ or 1% ash. Slight Oil Content.	1.83	15.24	.06562
Imperial A Drop Black	(Wm. Browne)		2.64	21.99	.04548
No. 1 Air Float Graphite	(Cummins-Moore Co.)		2.28	18.99	.05266
Graphite Dust	Superior Flake Graphite Co.		2.44	20.33	.04919
Formastat	Point Milling & Mfg. Co.	Micaceous Iron Oxide 94.01% Granite (Gangue) 5.94 Iron Pyrites .05	4.90	40.82	.02450
Black Oxide Iron	Laboratory Sample	Pure Precipitated Black Magnetic Oxide Marked Magnetic Pigment Co. Baltimore, Md.	4.81	40.07	.02496

TABLE 113

MISCELLANEOUS METAL PIGMENTS

Commercial Name of Pigment	Furnished by	Information Voluntarily Submitted	Specific Gravity	Weight per solid Gallon	One pound Bulks Gallons
Aluminum Dust	Institute of Industrial Research, Laboratory Sample		2.64	21.99	.04548
Magnesium Powder	Institute of Industrial Research, Laboratory Sample		1.77	14.74	.06784
Copper Scale Black	Bureau of Standards, Laboratory Sample,	Commercial Cuprous Oxide. Ferric Oxide 6.8% Insoluble 1.7%; 71.2% passes 150 Mesh.	5.76	47.98	.02084
Copper Bronze	Bureau of Standards, Laboratory Sample,		8.53	71.05	.01407
Finest Copper Bronze	Bureau of Standards, Laboratory Sample		7.83	65.22	.01533
F. F. Copper Bronze	Bureau of Standards, Laboratory Sample		8.28	68.97	.01450
Zinc Dust	New Jersey Zinc Co.	94% Metallic Zinc 90% will pass 350 mesh.	7.09	59.06	.01693
Zinc Dust	Metals Disintegrating Co.	97-98% Metallic Zinc	7.04	58.64	.01705
Lead Dust	Metals Disintegrating Co.	Manufactured from Standard Brands Pig Lead and Contains some impurities.	11.09	92.38	.01082

CHAPTER XVII

Oil Absorption of Pigments

(An Important Physical Test)

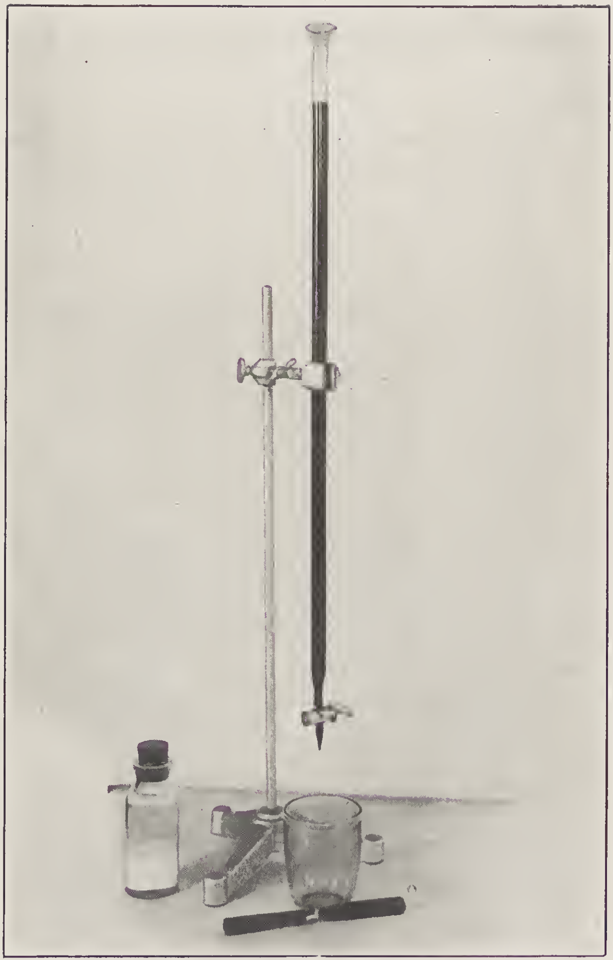

FIG. 32

Apparatus Required for Determining Oil Absorption of Pigments.

In mixing a pigment and a liquid a point is reached where independent of such mechanical forces as could be developed by actual grinding, the surface of each pigment particle is thoroughly wet by the liquid and the pigment mass becomes thoroughly saturated. This point represents the oil absorption property of the pigment, and is expressed by a factor termed the oil absorption factor. This factor is therefore a measure of the quantity of a given oil or liquid, required to thoroughly wet all the absolute particle surface of the pigment mass. The factor is ascertained by determining the number of cubic centimeters of liquid required to saturate 20 grams of pigment. It is then expressed as the amount required for 100 grams of pigment.

The oil absorption of a pigment is particularly important to the manufacturer who wishes to maintain a product of uniform quality and to the user who desires such a product. For instance a user of Lithopone might require for a certain paint, a pigment of a certain well defined oil absorption, in order that his product would remain uniform. If a shipment of markedly higher or lower oil absorption should be sent to him, he could discover this fact by the oil absorption test, previous to storing or using the pigment.

In designing a paint the oil absorption factor is especially valuable for determining the amount of oil required to grind each pigment to paste form or the quantity of each pigment required to produce a certain desired consistency. In improving the properties of an existing paint, a consideration of the oil absorption factor is also important. Thus if it is desired to increase the consistency of a paint without increasing the percentage of pigment, high oil absorbing pigments may be used. The opposite may be accomplished by the use of low oil absorbing pigments. In using high oil absorbing opaque pigments, inert pigments of low oil absorption may be added in order to lessen the amount of oil required to suspend the other pigments. When it is desired to secure maximum opacity, regardless of other qualities, a low oil absorption opaque pigment may be used. If a glossy, enamel-like finish is desired, a pigment of high oil absorption is indicated.

The amount of oil required for pigment saturation or wetting is directly proportional to the specific surface * of the pigment mass, existing at the point of saturation. As the specific surface of the mass is relative to its degree of particle sub-division or fineness, it also measures to a great extent the fineness of the pigment. The oil absorption factor being relative to the surface conditions of the pigment is independent of its chemical composition or Specific Gravity. The practical advantages of knowing the oil absorption factor lies in the information it gives concerning the following physical conditions of the pigment.

(*a*) Relative amount of surface in the pigment mass.

(*b*) State of sub-division of the pigment particles.

(*c*) Comparative specific surface of various pigments.

(*d*) Variation in fineness or particle sub-division of various lots of the same pigment.

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*It is believed that the oil absorption of a pigment may be reduced by compacting. The small particles would then form aggregates and the specific surface would be reduced, thus reducing the oil absorption. Similarly, by effecting electrical changes in the particles to cause a coalescence or by effecting a change in the surface tension of the particles, a reduction in oil absorption might be accomplished.

TABLE 114

OIL ABSORPTION FACTORS OF SOME PIGMENTS

Pigment	Low-Oil Absorption Type	High-Oil Absorption Type	Average Type
Basic Carbonate White Lead	15	22.5	
Basic Sulfate White Lead	26	32	30
Zinc Oxide	47.6	54.1	52
35% Leaded Zinc	31.3	36.5	32
Lithopone	22.75	38.5	33
Asbestine	32	50	50
Barytes	13	15	13.5
Blanc Fixe	23	36	30
China Clay	41.5	53	51
Gypsum	26	35	33.5
Silica (Crystalline)	20	28	23
Silica (Amorphous)	30	38	32
Talc	40	65	60
Whiting	28	35	32

The property of absorbing* a given quantity of oil is relative to the specific surface of the pigment mass. Thus the difference in oil absorption and body imparting property shown by various pigments is due to the difference in specific surface or particle sub-division. From this it follows that the variation or difference in pigments in this respect exists up to the saturation point only. Beyond this point they all become uniform in the amount of oil required to bring the mixtures to a certain consistency. This fact is important in the comparative testing

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*DISTINCTION BETWEEN ABSORPTION AND ADSORPTION.

It may be appropriate to attempt a distinction between Absorption and Adsorption to show the application of each to paint chemistry. There is first given the definition of each, followed by a discussion and summary.

"**Absorption.** To swallow up; to engulf; to imbibe; to cause to disof pigments in liquid paints. The following is illustrative of its application.

If a series of pigments are ground into pastes on the basis of the individual oil absorptions of each, and the same amount of thinning mixture is then added to the same weight of each dry pigment in its paste form, (pigment plus oil required to saturate) the resulting mixed paints, barring thickening due to any chemical action, will be of practically the same consistency.

Considerable information is also obtained by studying the condition of the pastes at the oil absorption point. A

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appear as if by swallowing up. To take up by cohesion, chemical or any molecular action." (Webster.)

"**Adsorption.** To suck in (compare with absorption). A common phenomenon consisting in the adhesion of the molecules of gases or dissolved substances to the surfaces of solid bodies resulting in a relatively high concentration of the gas or solution at the place of contact." (Webster.)

Discussion. Absorption. It will be seen from the definition above that the term "Absorption" in its broadest sense includes any imbibing or taking up action. It does not, however, imply any particular form or method of this imbibing action, or a necessary change in the chemical composition or the physical structure of the absorbent or absorbed material. It may be purely mechanical—such as the soaking up of water by a sponge; ink by a blotter; water by sand and similar instances. It may also effect a physical change of the absorbent such as the taking up of water by salt, sugar, or calcium chloride, in which case the absorbent is dissolved by the absorbed material. It may be molecular absorption, such as takes place in the chemical union of two substances, or it may be a type which is accompanied by a peculiar and definite physical change. Adsorption is an example of this latter type.

Adsorption. This condition while essentially a type or manner of absorption implies certain definite and peculiar physical conditions which are characteristic of it. As indicated by the definition above, this characteristic is the concentration of portions of the "absorbed" material at certain points in the "absorbent." In its most general sense, adsorption implies the unequal distribution of a substance at the boundary between two heterogeneous phases. As this reaction is definite and always occurs to the same degree and at the same rate with certain given conditions, it must be considered as an absolute term, and only applied when the condition above described exists.

Summary. From the above it is evident that the term ADSORPTION should only be applied when there are indications that the condition stated exists, and when the concentration and unequal distribution of substances as described is implied. In applying the term adsorption to the reaction the extent to which it exists in the mass must of course be sufficient to be of importance.

As absorption indicates the condition of taking up or imbibing without implying in any way the manner in which this is accomplished or the nature of the result of this imbibition, this term should be applied in all instances when it is desired to describe an absorbing condition without giving consideration to the type of absorption occurring.

From the above it is apparent that in describing the taking up of the oil by the pigment mass, the term "Absorption" is the proper one to use. While no doubt adsorption does occur in some instances, particularly with colloidal oils and varnishes, there is no evidence to show that it does in all cases.

very considerable difference will be noted in the characteristics of the pastes produced by various pigments. Some pastes will be short and tough. Others will be long, stringy and soft. Some will have a high gloss. Others will present a dull appearance. Some of the pastes will be dense, requiring heavy pressure to flatten them out. Others will be soft and easily spread. The amount of oil absorbed does not entirely control these factors. In other words, two pigments of practically the same oil absorption may yield entirely different types of pastes. For example, one may be dense and the other soft. These physical characteristics all indicate certain pigment qualities.

The oil absorption point varies with different pigments and is influenced by certain conditions. A pigment in its dry state consists of individual particles and agglomerates of particles. The particle surface and the mass particle surface combine to make the absolute surface existing in a given volume of the pigment. As the amount of oil required to saturate a certain amount of pigment is the amount required to wet the absolute particle surface, any influence which tends to change the surface content (by changing the specific surface) varies the oil absorption point. Two common influences accomplishing such change are found in the dispersion effect of certain vehicles, and the effect of mixing and grinding pressure. While these influences are always present to some degree in all paint making procedure, the basis of the oil absorption factor should be the condition of the pigment in its dry form with the elimination of these influences to as great an extent as may be possible. The oil absorption factor of all pigments is changed to the same relative degree by the introduction of these influences to an equal extent.

Certain oils and liquids, due to surface tension phenomena or to such colloidal conditions as effect adsorption or the development of great surface, tend to cause either a coalesence of the pigment particles into particle agglomerates or by their dispersion effect, to break down the existing agglomerates and disperse the particles into an extremely fine state of sub-division. Either of these conditions change the specific surface of the mass and accordingly change the oil absorption point. This variation in oil absorption with different oils is illustrated by the following table, which gives the oil absorption factor of several pigments in Linseed Oil, in Soya Bean Oil, and in a liquid having a relatively high colloidal viscosity but with an apparent viscosity approximately the same as the other two.

TABLE 115

Pigment	Raw Linseed Oil		Soya Bean Oil		Colloidal Condition Oil	
	Oil absorption factor	Character of paste	Oil absorption factor	Character of paste	Oil absorption factor	Character of paste
Zinc Oxide	54.	Smooth Glossy Quite long.	53.	Same as with Linseed Oil.	56.5	Glossy Smeary Quite rubbery.
Leaded Zinc Oxide (35%	36.8	Soft	35.5	Same as with Linseed Oil.	39.	Quite glossy. Smeary. Rubbery. Slightly granular.
Lithopone (High Oil Absorption)	37.5	Short. Chalky. Smooth.	37.	Same as with Linseed Oil.	38.	Fairly glossy. Long but not tough. Slightly granular in appearance.
Lithopone (Low Oil Absorption)	25.5	Soft and Long. Fair gloss. Contains large lumps.	23.5	Same as with Linseed Oil.	26.	Very soft. Runny and smeary. Good gloss. Lumpy.

Selection of Test Liquid.—In view of the results shown in the above chart it is important that the oil used for determining the oil absorption factor be one that will have the minimum effect in causing either a dispersion or coalesence of the pigment particles. In such a vehicle the pigment tends to form a mechanical suspension or mixture only. Thus the degree of particle sub-division or agglomeration existing in the dry pigment is retained to the greatest extent. Of the common and available oils, Raw Linseed Oil is the best suited for the purpose. With most pigments it has but little dispersing or coalescing effect. It is therefore used as the standard vehicle for determining the oil absorbing property of a pigment. If, however, it is desired to determine the oil absorption factor with certain other liquids, the results will be relative to the dispersing effect of the liquid used. In following the standard procedure a well settled Raw Linseed Oil, clear and free from foots and having an acid value of from one to three should be used. An oil of this kind is readily obtainable and easily kept. A variation in the acid value of the oil affects the oil absorption point; the amount of oil required increasing with an increase of the acid number. This difference is due either to a greater particle dispersion or to a thickening caused by the formation of soaps. It is important, therefore, that in making determinations where very accurate results or close checks are desired, that the oil used be always of the same acid value.

Effect of Moisture on Test.—The moisture contained in the pigment, between certain limits, seems to play no important part in the determination, since check results may be obtained on portions of the same sample of pigment whether air dried, oven dried or even slightly moist. Therefore, the ordinary air dried sample of commerce is in a satisfactory condition if it does not contain sufficient moisture to cause it to mat together or cake.

The temperature of the relatively small amount of oil used during the operation is not important, because the oil is immediately cooled by coming in contact with the much larger mass of pigment. This statement is made with the assumption that the oil is of the temperature prevailing in the ordinary laboratory. When, however, the pigment is quite warm, the oil absorption is slightly decreased. This is due to the fact that the viscosity of the oil is decreased by rise in temperature and consequently the thickness of the film with which it wets the particles would be decreased. However, the difference between 70° and 100° Fahrenheit, which is the average range of a laboratory, is very slight so that the temperature need not be taken into consideration except during extremes in either way.

Principles to be Observed in Test.—The principle of this test is that of effecting the wetting of all the "particle surface" or "particle agglomerate surface" of the pigment mass without the application of mixing or grinding pressure. This is accomplished by certain oil addition and stirring procedure as described herewith. In all stirring operations, care must be taken not to cause mixing of the pigment and oil by applying pressure, as this factor will tend to vary the results. All stirring and mixing should be done very carefully and lightly. A finger and wrist motion is all that should be used, allowing the forearm to rest quietly.

When the oil first comes in contact with the pigment, a corresponding amount of particle surface is wetted. The particles thus wetted then coalesce or cling together and form agglomerates or lumps of paste consisting of oil and pigment. These small lumps of paste should be kept distributed through the mass by stirring. As the absorption of oil by the mass increases these lumps will mat together, forming larger lumps or balls of paste. With further absorption of oil by the mass, these larger lumps or agglomerates of oil and pigment paste will coalesce and with most pigments will form one large ball. (With a certain few pigments this large ball is not formed; the separate smaller lumps remaining separated.) After this point is reached a small amount of dry pigment may still be left around the bottom and lower parts of the container. This pigment is wet by bringing it in contact with the free oil existing on the surface of the large ball or lumps of paste. The end point of the determination is reached when all of this dry pigment has been taken up and wet. This point is indicated by the following condition. While any dry unsaturated pigment still remains, the paste will not smear on the glass. At the point, however, where all of the pigment particles are wet, the paste becomes soft and will smear on the sides and bottom of the container.* The balls of paste may then lose their rigidity and tend to flatten out or lose their spherical form. This point, which is very sharp, occurs within narrow limits and is the end point.

APPARATUS REQUIRED.

Burette: A standardized burette should preferably be used. If not standardized, the determinations should always be made by starting with the same burette filled to the zero point. In this way possible differences due to variations in the burette graduation are largely eliminated.

Container for Mixing Pigment and Oil.—For this purpose a smooth, round bottom jelly glass is very well suited. Those having dimensions of approximately 2½ inches diameter at top, and 3½ inches deep are easily obtainable. Flat bottom glasses or those having fluted sides or bottom should not be used, as the pigment and paste tend to collect in the crevices, making accurate determinations difficult.

Spatula.—A blunt end, 4 inch, stiff blade spatula should be used. If the blade is too limber, excessive stirring is required and the results are liable to vary.

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*See Fig. 33.

METHOD OF PROCEDURE.

Preparation of Sample.—The sample to be tested should first be placed in a suitable container (a small wide-mouth bottle is adaptable) and well shaken so as to assure the elimination of any packed particles or lumps. This is especially important if the pigment has been packed in barrels or otherwise kept in a manner that is liable to cause it to pack together. If excessively moist, the sample should then be air dried at room temperature.

Procedure.—Twenty grams of the pigment are placed in the glass. The oil is then run in from the burette, either drop by drop or with successive small additions. Either of these methods will give check results. If the drop method is used the rate of flow at the start should be about one drop per second. If the small addition method is used, quantities of ½ cc. should be added. As the absorption of the oil increases, the rate of flow or quantity of the additions is decreased. This is fully explained later. The oil is run in so as to strike the dry pigment in the center. As the oil comes in contact with the pigment, the dry pigment that has not been wet should be lifted from the outer edge and placed over the oil so as to bring all the oil surface in contact with the pigment. This is best accomplished either by lifting it up on the spatula and dumping it off on the oil, or very lightly throwing it over the oil.

When the pigment particles become wet with the oil they tend to coalesce and form small lumps of paste. These lumps should be kept distributed throughout the mass. This is done by lightly stirring the mixture of these paste lumps and dry pigment in the manner of stirring described above, being careful not to use pressure in the mixing. If the drop method of oil addition is used this stirring should be carried on continuously with the operation of bringing the dry pigment in contact with the oil surface. If the successive addition method is used this stirring should be done after each addition. As the absorption of oil progresses these lumps of paste, by taking up more pigment and matting together, form larger lumps which when stirred around form balls. When this point is reached the rate and quantity of the oil addition should be very much decreased and only a few drops added at a time. In adding the oil at this point it should be allowed to strike on these lumps and not on the remaining dry pigment. After each oil addition these lumps are lightly stirred around so as to bring the oily surface into contact with the remaining dry pigment.

With further oil addition and stirring, these balls will with most pigments join together and form one large lump, with but little dry pigment remaining. With certain pigments, however, the lumps or balls of paste do not join together but remain separate and continue to collect more pigment. At this point, which is close to the end point, the oil is added very carefully, one or two drops at a time. With many pigments one drop is all that is necessary to establish the end point. At this stage, the oil should be allowed to strike on the surface of the lump or lumps. They should then be worked around so as to pick up more of the pigment. When all of the remaining dry pigment has been picked up and wet, the end point is reached. This is indicated as above explained by the paste lump becoming much softer and easily spread with the spatula. When stirred around it smears on the sides and bottom of the glass. This end point is very sharp and occurs within narrow limits. With most pigments only one or two drops of oil is required to change the relatively hard dry lumps which do not smear the walls of the container into a fairly soft paste which will smear when stirred around. If, when the end point is reached, the paste lumps are broken down or spread with the spatula, it will be noted that the oil is uniformly distributed throughout the mass and no dry pigment remains.

Different pigments have certain individual characteristics in absorbing oil. These must be given consideration, and it is sometimes necessary to slightly modify the procedure in order to conform with these peculiarities. For example, some pigments absorb the oil readily and in a uniform manner, while others take up the oil slowly. The paste lumps of some are smooth and free from hard agglomerates and are easily kept uniformly mixed. Others tend to form hard lumps. With such it is difficult to keep the mass uniform. When the end point is reached with certain pigments, the paste lumps will be quite firm. Other pigments will be firm up to the end point and then suddenly become very soft; in some instances of semi-paste consistency. These differences are largely due to certain surface tension relations of the pigment and oil, a discussion of which is outside the scope of this paper. A little experience with the various pigments will enable the operator to effect such modifications as will be necessary to conform with these conditions.

Close checks can be obtained on the test made in accordance with the procedure explained above. Exper-

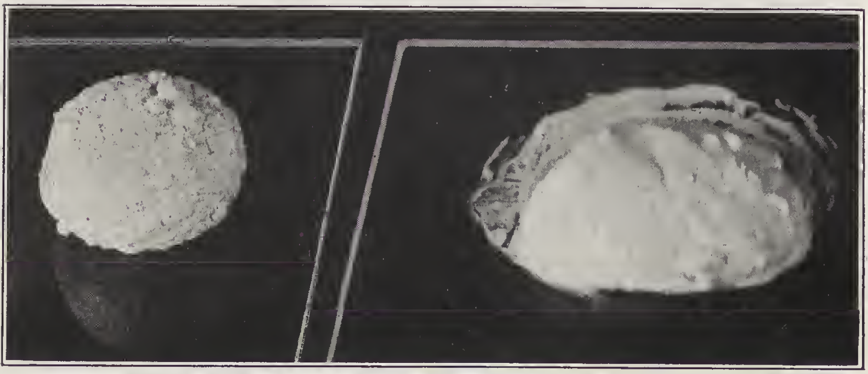

FIG. 33

Pigment Mass Just Before Point of Saturation. Mass is Spherical and Does Not Mark Glass.

Pigment Mass at Point of Saturation. Mass Loses Its Spherical Form and Smears Glass.

ienced operators rarely vary more than 0.1 cc in check tests or in tests conducted by different operators on the same sample. For ordinary purposes a variation of 0.2 cc is sufficiently close. A variation of more than this amount on check tests can only be considered as an error in the operation. In testing new pigments or where a close determination is desired, two or more check tests should be made. A little experience and a consideration of the following previously stated precautions is sufficient to insure accurate results:

(*a*) The pigment must not be moist.

(*b*) It should be well shaken before making the test.

(*c*) It should not be of a temperature lower than 70° or higher than 100° F.

(*d*) The oil should be well settled and of a standard acid value.

(*e*) The oil should not be added too fast, nor should it be added slower than is necessary.

(*f*) The mixture should *only* be stirred enough to keep the mass uniform and bring the pigment in contact with the oil.

(*g*) Excessive stirring tends to lower the result.

(*h*) The stirring should be done lightly and care taken not to use pressure or endeavor to mix the pigment and oil by this means.

(*i*) When nearing the end point, the oil should be added slowly and in small amounts (one or two drops at a time).

CHAPTER XVIII

Fineness and Texture of Pigments

(A New Method of Examination.)

Finely divided pigments grind easily, with low power consumption, and produce smooth paints. Coarse pigments grind with difficulty. They require much power and often produce coarse paints. Some grinders have heretofore purchased certain pigments on specifications which require a certain degree of fineness as determined with a 200 mesh silk bolting cloth or with 200 and 325 mesh metal screens, using water or kerosene as the carrier for the pigments. Tests made in the above manner simply indicate that a certain percentage of the pigment is coarse. While these tests are of value, they do not disclose the true physical nature of a pigment. It is believed, however, that the new method of examination described below as used by D. W. Dinwiddie of the Edison

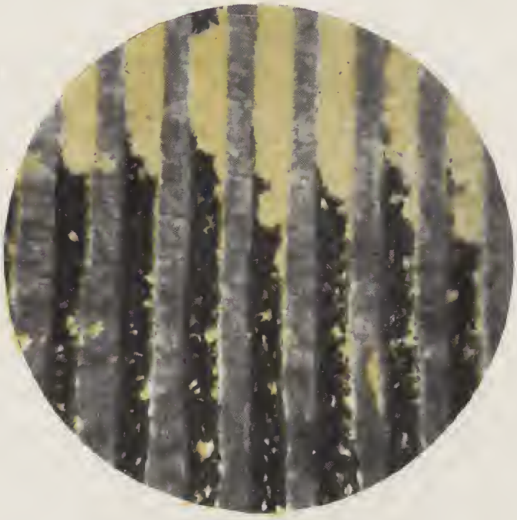

FIG. 34

Photomicrograph of yellow pigment placed on talking machine record

Laboratories indicates many of the physical properties of a finely divided substance and can be of great value to the grinder in acquainting him with the characteristics of all pigments and of different batches of the same pigment.

The writer has adapted this method to photomicrographic work, and presents herewith some views of a few pigments that are commonly used in the industry.

FIG. 35

Particles of a very coarse red pigment.

Method of Test: The test is made by rubbing with the finger a portion of the pigment across the grooves of an Edison disc phonograph record and then observing the surface with a microscope. The disc has a spiral groove with 150 convolutions per inch of radius. The lines are 1/150 of an inch from centre to centre. The radius of curvature of the sound grooves is 4/1000 (.004) of an inch. The average depth of the grooves is 1/1000 inch.

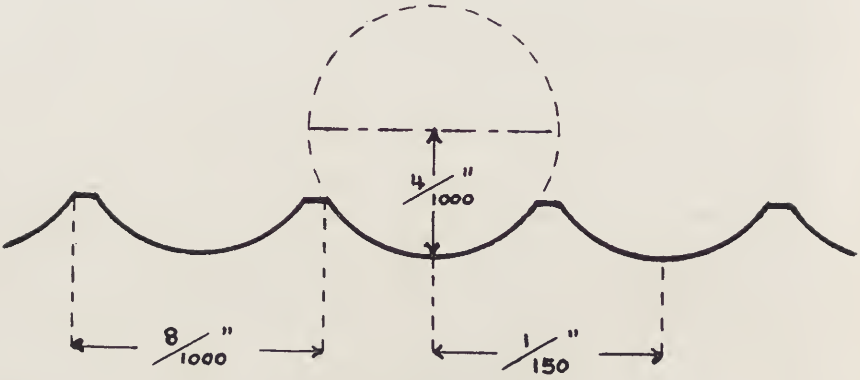

FIG. 36

One record may be used for several pigments which may be numbered and the records kept in a cabinet for comparison as standards for various subsequent batches of pigments produced or received in the factory. During the procedure of rubbing the pigments into the grooves, characteristic differences are at once noted. Some pigments will feel coarse and gritty. Others will have a soapy or unctuous feeling. For instance, two samples of china clay or barytes which appear to be of equally fine texture when rubbed between the fingers, may upon rubbing into the record show entirely different characteristics. One pigment may feel soft or "silky" in texture, while the other may feel granular and "gritty." Microscopic examination of the surface will then immediately disclose the reason for this phenomenon.

FIG. 37

Record with various pigments mounted for microscopic test.

Most pigments are made up of particles of varying size. The large particles are often covered with fine ones. When such pigments are placed on a record, the rubbing action forces the large crystals between the grooves and arranges them with their facets parallel to the plane of the record. The fine particles are at the same time wiped from the larger crystals, so that the form of the latter is disclosed. With fume pigments, the very fine particles as well as the agglomerates may be shown. The relative degree of color, whiteness and hiding power of the pigments are to some extent also indicated.

For the microscopic examination it is advisable to use a 32 mm objective and a 12.5 × eye-piece. Greater depth of focus is thus obtained and the magnification is sufficient. It is usual to look at the disc obliquely in order to get the specular reflection of the light.

For a photographic record of the appearance of the pigments, great care should be taken that a strong light is provided and that a water cell is interposed for absorbing the heat rays of the light. Otherwise the light, impinging upon the record at one point, will cause a melting down of the grooves. Photography then becomes impracticable.

FIG. 38

Particles of a very finely divided white pigment. Agglomerates shown.

Counting Particles: The writer has conducted other experiments with the object of actually counting the number of particles in a given weight of pigment. These experiments indicate that this may be accomplished with the Haemacytometer such as is used for counting blood corpuscles. For this purpose, the writer has found it advisable to use an extremely small amount of pigment and to suspend it in water containing a small amount of emulsification reagent to keep the pigment in suspension. One gram of pigment

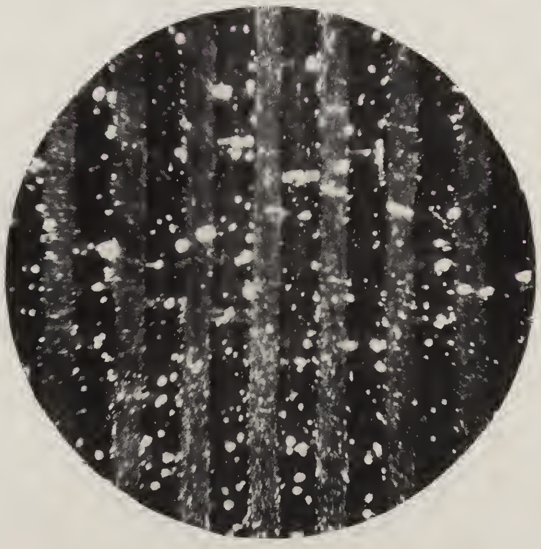

FIG. 39

Particles of a coarse fibrous mineral pigment. Note rod-like structure.

to 1000 grams of water containing a very small amount of gelatin has been found fairly satisfactory. After the emulsion has been thoroughly shaken, a few drops are withdrawn and placed in the counting chamber of the object slide. This slide is divided into squares occupying an area of 1/400 of a square millimeter. By counting the number of particles disclosed in the squares and by proper multiplication, an estimate of the number of particles in a known weight of the pigment may apparently be made.

CHAPTER XIX

Metal Powders as Pigments

Zinc—Aluminum—Lead—Copper—Iron

Zinc.—Finely divided metal powders have specific uses as pigments. Zinc powder (zinc dust) for instance, has been employed to some extent as a pigment, and for many years has formed a substantial part of the antifouling and anticorrosive ships bottom paints used by the Navy Department. It would seem that a much more extensive application of this powder in paints could be made. For instance, a paint made of finely divided zinc powder suspended in a heavy bodied drying oil, a spar varnish, or a lacquer could be used as a "liquid galvanizing." When applied to a bare iron surface or to a galvanized surface from which the galvanizing had worn off, there would result a waterproof film containing a metal that would protect the iron from corrosion. Exposures made by the writer of what might be called "liquid galvanizing" made along the above lines have given excellent results after a period of two years. Zinc powder of the following grade will be found quite satisfactory for use in paints. Great fineness is the chief requisite for successful application in this field.

> It shall consist of zinc dust that contains at least 90% metallic zinc. It shall be of such fineness that 96% shall pass through a 300-mesh screen.

Aluminum.—Aluminum powder has been used to some minor extent; in aluminum bronze paints, being suspended in a liquid usually composed of a quick-drying varnish. The temporary character of the varnish that has usually been selected for this purpose may be responsible for the existiig belief that such paints are not satisfactory for exterior work on metal. As a matter of fact, however, aluminum powder when used with a good grade of exterior spar varnish will give good results and it would appear that the value of this type of paint has been overlooked by the designers of protective coatings. Because of their low weight they might be preferred for certain specific purposes, such as the waterproofing of speed boat hulls. Another property possessed by this type of paint is that of high solar radiation; surfaces coated with it remaining very much cooler when exposed to the sun than those coated with dark colored paints. A similar type of coating might also be used with satisfactory results as a protective for rubber automobile tires. The high reflection obtained would largely prevent the destructive action of the sun, which is responsible for the rapid decay of rubber. Aluminum powder of the following grade is quite satisfactory for use in paints.

> It shall consist of powdered aluminum containing at least 98% metallic aluminum and not over .5% copper. It shall be of such fineness that it will all pass through a 200-mesh screen. When such paints are used over rubber surfaces, the amount of copper present in the aluminum powder should preferably be less than 0.1%.

Lead.—Finely divided metallic lead has also been used in metal paints with successful results. For instance, bituminous compositions for under water use, containing finely divided lead, have been used to a conisderable extent.

Copper.—There has been but little industrial application of finely divided metallic copper as a pigment, but it would appear that it could be used with great success in certain types of marine work. Where copper oxide paint is now employed, copper might be used if prepared with a sufficient degree of subdivision. A more recent method of protecting ships' hulls in order to give a copper surface in place of copper sheathing, is to apply a coat of quick-drying spar varnish and then dust or blow on finely divided copper. The copper adheres to the varnished surface and produces a metal-like sheathing that is toxic to marine growths.

Iron.—Finely divided iron in the form of iron filings or precipitated iron has been used to a slight extent in paints and also as an ingredient in surface coatings for floors, being admixed with the cement or other material to form a very hard surface. Other metals in powdered form may also find special application as surface coatings and become listed in the index to the many hundreds of raw materials used in the manufacture of paint.

Although both zinc and aluminum have a high protective action on iron surfaces, it would appear to the writer that the efficiency of either as a pigment in metal primers would depend to some extent upon the amount of pigment actually present, since nearly every test of metal primers has shown that increase of pigment up to a certain maximum adds greatly to the durability.

When metal powders are employed in primer paints, those metals should preferably be used that have a higher solution tension than the iron itself. This same general principle should apply, for instance, in the plating of metal. For example, when iron is plated with either copper or nickel, a very dangerous condition will result and active corrosion may ensue at an early period. In order to better understand this conidtion, a brief outline of the causes of corrosion is given below.

If two metals are placed in contact in the presence of water, a primary battery is formed, and galvanic action ensues. The electropositive metal will go into solution and the electronegative metal will be unacted upon. This effect may be illustrated by placing a strip of iron in a solution of copper, when it will be found that the iron (electropositive metal in this couple) will go into solution and the copper (electronegative) will plate out on the iron. If a strip of iron is placed in a solution of zinc, no such phenomenon will occur, zinc being electropositive to the iron. If copper plated steel or iron becomes scratched or abraded, moisture and air will be admitted. The iron, being the electropositive metal in the galvanic couple that is formed, will rapidly pass into solution. The oxygen of the air will then oxidize this solution and iron rust will be precipitated out, piling up into a spongy mass which will retain moisture that will serve to continue the corrosion.

There are shown below a number of the commonly used metals, in the order of their solution tendencies:

Aluminum
Zinc
Iron
Nickel
Lead
Copper
Tin
Antimony

Any metal shown above, when placed in contact in the presence of water with another metal lower in the series, will tend to protect the latter metal from corrosion. A study of the above series will indicate that the only commercially available metal of moderate price and applicable as a coating, that is higher in the series than iron, is zinc. This metal is therefore theoretically ideal to use as a protective coating on iron surfaces when applied in the form of galvanizing. The film of zinc produced, even when abraded, will have a marked protective influence upon the adjacent areas of bare steel. For paint uses, both zinc and aluminum powders may be used in primers, with due observance of the principles outlined above.

CHAPTER XX

Primers for Aluminum and Galvanized Iron

FIG. 40

SPOT TEST ON ALUMINUM.

Etched surface of Aluminum. Caused by Basic Pigment.

Unaffected surface of Aluminum. Neutral Pigment used.

Aluminum Corrosion as Affected by Basic Pigments.—Metallic aluminum is popularly supposed to be non-corrodible since it does not evidence marked discoloration under ordinary conditions of use. This is due to the fact that its oxygen compound is white and less readily discernible than the oxides formed when other metals, such as iron, corrode. Under severe conditions of exposure, aluminum shows very rapid corrosion because of its high solution tension. The white oxide that forms presents a rough surface to which soot will adhere tenaciously, causing a mottled appearance. Aluminum metal should therefore be given surface protection whenever practicable, but great care should be exercised to select the proper type of coatings.

As a result of some recent experiments, the writer has found that aluminum is rapidly attacked by pigments of a basic (alkaline) nature, such as are considered necessary for the protection of iron and steel. It is affected only to a slight extent by some pigments which stimulate the corrosion of iron, and practically unaffected by inert, crystalline pigments. For instance, red lead, white lead, zinc oxide, and other basic pigments, when applied to aluminum, etch the surface rapidly whereas silicious earth and other neutral crystalline pigments have no etching action. This phenomenon may be demonstrated by grinding a pigment in water to paste form and placing a small amount of the paste on a sheet of aluminum for a period of 24 hours. The result shown in the above illustration will be duplicated.

Automobile bodies, household devices, and similar products are often constructed of aluminum on account of the lightness and other unique physical properties of this metal. When these articles are "primed" or "filled" with paint, it is customary to sand or otherwise roughen the metal in order to prevent peeling. The solid part of the priming paint might for this special application be made up of a substantial amount of basic pigments admixed with silicious earth pigments. Under favorable

FIG. 41

SPOT TEST ON GALVANIZED IRON
Showing Etching Effect of Basic Pigments

conditions, the surface of the metal would be slightly etched by the basic pigments and a firm foundation from which subsequent coats would not peel would then be provided. The moisture shedding effect of the many outer coats should be sufficient to prevent any substantial progressive corrosion.

For other forms of aluminum, which may be exposed to atmospheric conditions, the use of a high grade spar varnish is recommended. The bright color of the metal would not then be obscured.

Pigments for Galvanized Iron Primers.—The smooth, spangled surface exhibited by galvanized iron treated by the hot dip galvanizing process is in some instances the cause of the peeling of subsequently applied paint coatings. As the result of some tests just made by the writer, it would appear that much of this trouble could be obviated by the use of special priming paints made up with basic pigments. This would be indicated by spot tests which show that basic pigments in the presence of moisture rapidly etch the surface of galvanized iron. Results such as are shown in the above photograph may be obtained by grinding a pigment in water to paste form and placing a small amount of the paste on a sheet of galvanized iron for a period of 24 hours. The pigment should then be washed off and the etching effect observed.

A primer paint made up with substantial amounts of basic pigments, such as red lead and zinc oxide, ground in an oleaginous liquid containing sufficient water to make reactive the basic properties of the pigments gave initially satisfactory results. Although more extended exposure tests of such a material are required upon which to base a conclusion as to its value, it is believed that the principle involved in its use is sound.

CHAPTER XXI

Rare Elements as Paint Pigments

Many of the rarer elements that heretofore have had but little industrial application, received much consideration during the war-time period. It is probably safe to state that many of them will be used as the basis of preparations destined to become of great importance in the arts. Their possibilities as pigment bases, for instance, has for several years engaged the writer's interest. Among the more obscure or not commonly known metals that he has experimented with are molybdenum, cerium, lanthanum, selenium, thorium, titanium, tungsten, uranium, vanadium, zirconium, beryllium, and radium. Nearly all of the above metals, in the form of their respective insoluble compounds, have a high refractive index and consequently produce, when ground with oil, very opaque mixtures having a hiding power comparable with lead or zinc pigments.

From the standpoint of war-time usage, radium pigments have been of the greatest interest; substantial quantities having been used and with most successful results on the dials of altimeters, air speed meters, and similar aircraft instruments, and on other military paraphernalia used in modern warfare. The radium may be applied as a salt to an active or sympathetic base having real pigment properties, such as zinc sulphide. This activated pigment is mixed with a thin, clear varnish that acts as a binder. Only small amounts of radium salts are required in such paints to produce energetic vibrations that send out active radiations in the form of a soft greenish glow. The light produced is sufficient for the result intended. The radium salts used are manufactured largely from carnotite ore mined in Colorado. These paints have almost entirely replaced the old luminous calcium sulphide paints which, although comparatively low in price, depend upon absorption of energy during the day time when exposed to sunlight. They are of but short life and not intensely phosphorescent. The only substitute for radium is mesothorium, an element found in monazite sand that is mined in certain of our Southern States. This sand is the product from which thoria is extracted for use as the glow base for incandescent gas mantles; as high as 5% of thoria being present in some American sands and 9% in some of the East Indian varieties. The meso-thorium present in the sand is, like radium, capable of sending out active radiations and has been used as the basis of some luminous paints. Its longevity is not as great as that of radium.

Selenium, an element analogous in some of its properties to sulphur, which is obtained during the smelting and refining of copper, has had but little industrial application. The writer has prepared several most interesting pigments from it (the selenites of lead and barium, for instance) which are very white and of fine grain. Their exceptionally high refractive index gives to them intense opacity. Other pigments of a closely related nature may be produced from beryllium, an element found as a silicate or aluminate in several Eastern States. The oxide and similar products of zirconium have a very high degree of whiteness, opacity, and permanence. From titanium, an element which is widely distributed, many most interesting compounds have been produced, and it is highly probable that they will some day be used to a great extent as pigments where great opacity, permanence, and other closely related physical properties are desired.

Among the more common elements from which white pigments may be made are arsenic, antimony, bismuth, and tin. The color compounds of these metals produce black, yellow and orange pigments that have had but little use. When their salts are precipitated as oxides, oxychlorides or other basic salts, white pigments of high opacity are obtained. The toxic nature of some of these products and their high price as compared to white lead or zinc pigments has limited or prevented their use.

CHAPTER XXII

Waterproof Glues

In the paint industry, animal glues (gelatine) and vegetable glues (processed starch) have been used as adhesives for cold water paints and calcimines, and for labeling gums. These forms of glue possess the undesirable property of becoming weak when exposed to moisture or subjected to the action of water. As a result of war time endeavors to develop satisfactory glues for seaplane plywood, laminated gun stocks, and other products, there became available glues produced with casein or blood albumen as a base. These glues have extraordinary water resisting properties. Plywood made of 3-ply veneer 1/16 inch thick glued with casein glue may be boiled in water for eight hours and still show an average breaking load of not less than 90 pounds per square inch. One-inch test blocks may be placed together with this glue and show a shearing strength of 2,200 pounds per square inch.

Casein glue is cheaper than blood albumen glue, and is probably more practical for most commercial purposes where application is made by hand rather than by machine or hot press. It is possible that casein glue might be found useful as a base for label pastes. Its slightly alkaline reaction would etch the surface of tin with the possibility of greater bonding power than some other adhesives. Because of its high waterproofing properties, it might also be useful as an adhesive in calcimine and cold water paints. The writer has experimented with such glue as a part of the liquid portion of exterior paints, and secured interesting results. If desired, casein glue may be prepared direct from casein by the addition of moderate and well regulated amounts of slaked lime, caustic soda, or sodium silicate. As a rule, however, it is advisable to purchase the glue in prepared form, since carefully selected and finely divided casein, having a very low acid, fat, ash, and moisture content, and high nitrogen content, is used for this purpose and skillfully mixed to produce a successful product.

It is of great importance when any type of casein glue is used that it be properly mixed. Otherwise unsatisfactory results might follow. One of the best mixers is of the change can, bladed type, such as is used for mixing paint previous to grinding. The dry powder is placed in the mixer and from one to two parts of water added, according to the degree of fluidity desired. It is preferable that the container for the material should be of the enameled type, as copper, aluminum, and brass vessels are slightly attacked by the glue. After the glue has been mixed, it is usual to allow it to stand for a period of one-half hour before use, in order to develop its full strength. Glue thus made, as a rule, will remain fluid for a period of five hours, although some types may remain fluid for a much longer period. After standing in aqueous admixture for 24 hours, most casein glues set up to a hard, unusable mass, and it is then necessary to make up fresh batches. The glue dries to a very waterproof film of great strength.

CHAPTER XXIII

Quick Drying Lacquer Coatings

Coatings of a very rapid drying nature were required during the war period for many types of manufactured products. Tung oil varnishes, cut with a high percentage of volatile spirits, were used for the base of many of these coatings. It was found, however, that where greater rapidity of drying was required, spirit varnishes of the shellac type were necessary. Later on, speed of production on some products demanded the use of finishes that would dry within a few minutes, and cellulose lacquers or "dopes" were adopted with successful results. Such "dopes" were already in use for making moisture resistant and rendering taut the fabric wing surfaces of aircraft. They were composed of either cellulose acetate or cellulose nitrate dissolved in volatile solvents. For aircraft work their composition resembled those shown below:

Cellulose Acetate Dope.

Methyl acetate	60%
Methyl ketone	10%
Benzol	15%
Acetone	10%
Diacetone alcohol	5%

Each gallon of dope made with the above liquids contained approximately 7 to 9 oz. of cellulose acetate, 1 oz. triphenylphosphate, and very small quantities of chemicals such as benzyl acetate, benzyl benzoate, and urea.

The various stabilizers and other solid ingredients in the dopes were used for specific purposes (such as to increase fire resistance and flexibility, or to prevent the development of free acid or "blushing"), but are not usually required where the "dopes" are to be used only as protective coatings.

Cellulose Nitrate Dope.

Butyl acetate	20%
Ethyl acetate	50%
Benzol	30%

Each gallon of dope made with the above liquids contained from 6 to 8 oz. of cellulose nitrate.

Cellulose nitrate "dope" is greatly improved as a protective coating by the addition of from 5% to 7% of castor oil or treated tung oil. Greater elasticity of film, and slower evaporation result.

It is quite possible that cellulose nitrate lacquers made on the above basis will find application in peace times for certain specific purposes where quick drying, hard, and elastic films are required. They may be admixed with pigments to produce colored coatings which dry to a flat, washable surface. When mixed with aluminum powder or zinc powder, quick drying, hard primers for metal are formed. These may be used satisfactorily as the base for many metal finishes. As substitutes for shellac on some types of work they should also prove of value.

For coating shells and similar metal objects, these paints should prove efficient, as they may be applied by spray, brush, or dipping, drying almost immediately to a moisture resisting, flexible film. Baking at a low temperature is permissible.

The writer has found clear or colored cellulose nitrate lacquers to act as excellent primers for certain types of cement floors that are to be painted. Through their use, waterproofing greases or other materials in the cement are insulated from action on subsequently applied enamels.

It should be pointed out that cellulose lacquers or enamels are not as durable as those made with oil or varnish. They are, however useful for certain purposes where the longevity may be partly sacrified to obtain rapid drying.

CHAPTER XXIV

Notes on the Standardization of Mineral Spirits

Properly refined mineral spirits have come into extended use in the last fifteen years for thinning paints and varnishes. During the present abnormal situation in the turpentine industry their use has been greatly extended. The type most desired is that which most closely resembles turpentine products in evaporative value. Grades for instance, which have given excellent results show upon distillation 97% between 150° and 215°C.; others of good quality show 97% between 160° and 230°C. Grades showing a high end point may contain sufficient mineral oil to slow down the drying of certain paints and enamels. The higher boiling grades are, however, useful for thinning flat wall paints where a fairly long drying period is desired in order to allow the paints an opportunity to flow out to smooth films. Ordinarily, however, the lighter boiling grades are preferred.

Probably the most important point to consider in the selection of mineral spirits is that of solvent properties. The solvent properties depend very largely upon the base from which the distillates are derived. For instance, those produced from an asphaltum base petroleum are usually more unsaturated and contain more cyclic hydrocarbons. They often have, therefore, higher solvent properties than those produced from a paraffin base petroleum. A series of comparative tests are under way to determine the solvent properties of each grade of mineral spirits when used for thinning varnishes, boiled oils and blown oils. This investigation will also include information regarding their effect on glossing, flatting and other properties.

A series of analytical determinations upon a number of samples of mineral spirits recently obtained by the writer from the producers and users are shown herewith.

TABLE 116

Trade Initial	Appearance	Color	Flash point Tag. Closed cup	Spot test after 30 minutes	Sulphur test with white lead	Distillation			Reaction of residue	Gravity by hydrometer
						Initial boiling point	Final boiling point at 97%	Percentage of distillate		
S. O. Co.	Clear. Free from suspended matter and water	Practically water white	39°C.	None	Not darkened	154°C.	207°C.	97	Neutral	0.775
A. R. P. T. Co.	Clear. Free from suspended matter and water	Practically water white	30.5°C.	None	Not darkened	141°C.	224°C.	97	Neutral	0.763
No. 180. T. W. O. W.	Clear. Free from suspended matter and water	Practically water white	30.5°C.	None	Not darkened	136°C.	214°C.	97	Neutral	0.763
No. 185 T. W. O. W.	Clear. Free from suspended matter and water	Practically water white	79°C.	Slight spot left	Not darkened	210°C.	235°C.	97	Neutral	0.784
T. S. P. T. C.	Clear. Free from suspended matter and water	Practically water white	39.5°C.	None	Not darkened	155°C.	215°C.	97	Neutral	0.782
No. 18 S. P. T. S. Co.	Clear. Free from suspended matter and water	Practically water white	30.8°C.	Slight spot left	Not darkened	129°C.	235°C.	97	Neutral	0.813
S. S. P. A. R. C.	Clear. Free from suspended matter and water	Practically water white	34°C.	None	Not darkened	146°C.	207°C.	97	Neutral	0.776
No. 18 T. S. Co.	Clear. Free from suspended matter and water	Practically water white	28.5°C.	Slight spot left	Not darkened	130°C.	239°C.	97	Neutral	0.813
No. 19 T. S. Co.	Clear. Free from suspended matter and water	Practically water white	54°C.	Slight spot left	Not darkened	162°C.	249°C.	97	Neutral	0.838
TE. No. 1 T. B. Co.	Clear. Free from suspended matter and water	Practically water white	47.2°C.	None	Not darkened	165°C.	230°C.	97	Neutral	0.771
TE. No. 2 T. B. Co.	Clear. Free from suspended matter and water	Practically water white	41.1°C.	None	Not darkened	147°C.	224°C.	97	Neutral	0.773
T. S. T. C.	Clear. Free from suspended matter and water	Practically water white	36.1°C.	None	Not darkened	155°C.	217°C.	97	Neutral	0.785

NOTES ON STANDARDIZATION OF MINERAL SPIRITS

The results of these investigations have been used as a basis for drawing up standard specifications for mineral spirits by the Interdepartmental Committee on Paint Specifications Standardization. The following specifications were submitted for their consideration:

APPEARANCE:—It shall be clear and free from suspended matter and water.
COLOR:—It shall be water white.
SPOT TEST:—It shall evaporate completely from filter paper without leaving a greasy mark.
FLASH POINT:—It shall be not lower than 30°C (85°F) when tested in a Tag closed tester.
SULPHUR:—Sulphur shall be absent as determined by the white lead test.
Distillate below 130°C. (266°F) shall not exceed 5 percent.
FOR TYPE 1:—Distillate below 215°C. shall be not less than 97 per cent.
FOR TYPE 2:—Distillate below 235°C. shall be not less than 97 per cent.
REACTION:—It shall be neutral.

CHAPTER XXV

An Emollient for Skin Protection in the Dope and Varnish Using Industries

During the war period workers upon airplane wing fabrics found it difficult to avoid occasional splashing of compositions containing large quantities of volatile solvents such as benzol, acetone, and alcohol. Their hands occasionally became encrusted with dried dope, which could only be removed through the use of strong solvents such as acetone and methyl acetate. As a result, cases of skin trouble were observed. The hands would become swollen and covered with an eruption. Similar cases of skin trouble have been observed among workers who constantly have their hands in contact with benzine, and like solvents or in contact with lubricating and cutting oils which may carry dirt into skin cracks and cause infections.

The writer, during the war period recommended for dope workmen a hand salve made on the following formula:

Formula:

Lanolin (Adeps Lanae Hydrosus)	25	parts	by	weight
Petrolatum	25	"	"	"
Stearine	25	"	"	"
Glycerin	25	"	"	"
Total	100	"	"	"

This material was rubbed into the hands before starting work. It is not rapidly attacked by solvents. After washing up at night the workmen may rub in a little more of the salve. It keeps the skin in good condition. Its use apparently avoided all cases of skin trouble. During the last year it has been used successfully in a large oilcloth and artificial leather factory where in the machine and drying rooms several hundred gallons of coating solvents are daily evaporated. Such use might appropriately be extended to other industries where there are used coating compositions containing large amounts of volatile organic solvents or thinners.

CHAPTER XXVI

Illumination From Paint

Lighting with Paint.—People are just beginning to appreciate the value of paint from a lighting standpoint. For instance, the now almost universal practice of painting the interior walls and ceilings of factories in order to increase the illumination has developed within a few years. The tremendous savings in electricity or other illuminants, and the increased efficiency of the workers resulting from such practice, have given mill owners an appreciation of paint for this purpose. A further and even greatly extended use of paint as an illuminant reflector will soon be observed.

Lighting Terminals and Carriers.—In practically every old-style urban railroad terminal there are immense train sheds which present a neglected and gloomy appearance. Where paint was used, a very dark color was generally selected, as paint was looked upon simply as a preservative and black was the prevailing color of metal preservative paints. In some instances, however, no paint was applied. If such structures should now be painted so as to provide a finishing coat of white or of light tint, the amount of light inside the sheds would be tremendously increased. Accidents would be prevented and many glaring lights might be done away with. Moreover, the arriving guest would not feel depressed or secure a wrong impression of the community. While it is appreciated that the smoke from the locomotives would stain white paint, its surface would not become as dark as the uncoated structure. In any event, the lower parts might be washed occasionally. Similarly, in warehouses, freight sheds, wharf houses, interiors of freight cars, vessel holds, etc., the application of light paints to the present uncoated interiors would immensely improve the working conditions.

Lighting Machinery.—Very little has so far been done with paint for lighting machinery. For instance, many of the surfaces of the machines in large factories are uncoated. They gradually assume a dark color that gives shadows that may be responsible for many accidents. If the working surfaces of such machines should be painted white a much greater amount of light would be obtained. Accidents would thus be minimized and production efficiency increased.

Lighting Streets and Country Highways.—While the use of paint for marking asphalt streets for traffic regulation purposes has increased in some cities to quite an extent and has effectually aided in bringing about a more general observance of the rules of trafic, it can readily be seen how much greater should be the extension of this principle. For instance, all stone curbing on dark or poorly lighted streets might be painted white. A decrease in motor accidents would probably result. Along the open highways and boulevards, especially at dangerous corners and along embankments, marking stones and the trunks of trees at well-defined distances on either side, if painted white, would reflect the illumination afforded by headlights and make night driving much safer.

Painting Motor Tires.—The (solar) reflecting value of white and very light tints has also been shown recently to be a factor of great importance. Certain exposed structures, such as gasoline tanks, can be kept much cooler if the finishing coats of paint are of light color. This principle should be applied to many other products that are often uncoated. For instance, all automobile tires are not white, although many manufacturers, as a result of the demand for a more attractive tire and one of great durability, are resuming the production of at least white side walls. Those that are black, red or other dark colors absorb heat (solar) rays which are tremendously harmful to rubber. For this reason the owner might appropriately paint all dark colored automobile tires with specially designed white tire paints. The high solar radiation shown by such paints will have a protective influence of great importance.

Painting Telegraph Poles.—It would be difficult to state how many million telegraph poles line the highways and railroads of America. It is not difficult, however, to show that these poles, under the climatic conditions existing in many sections, do not last indefinitely. Impregnation of the underground part is a practice that is generally adhered to in order to provide protection from underground decay. The upper part, exposed to the air, is seldom surfaced, and the ravages caused by exposure to the air soon become apparent. It is quite fair to state that overground protection, to be had through paint application, is also necessary, especially on all that are squared or otherwise dressed. Such treatment would extend the replacement period and help to conserve forests.

Painting Railroad Ties.—The millions of wood ties used on railroads, even though they are in many instances impregnated with zinc chloride, creosote or other preservatives, would probably last an even longer period than at present if they were subsequently given surface protection with the proper type of paint. These ties should at least be painted on trestle bridges where hot coals dropped from locomotive fire-boxes might cause destruction.

Painting Rails and Track Equipment.—The above observation may lead some people to inquire why even railroad tracks are not painted. While it might be of some advantage to preserve the flange surfaces from corrosion by painting, the bearing parts really require no surface protection, as they are kept bright and free from corrosion by the constant friction of the wheels. While the surface of new rails may show a film of rust, used rails seldom rust, even during a period of long inactivity. This is due to the physical condition of the much stressed and hammered surface, which apparently becomes passive to electrolytic corrosion. Disregarding, however, the use of paint from the protective standpoint, the flange parts of steel rails at cross-overs, switches and other places might with advantage be painted white for safety purposes. Grade crossings thus treated would be better indicated at night. Railroads now average about one fangate to the mile. These should always be painted white and with sufficient frequency to maintain a surface of high reflection.

The following research conducted by the writer presents clearly the scientific data on which the foregoing principles are based.

Some Principles Involved in the Manufacture of Interior Wall Coatings Having High Reflection Factors.—In the paper entitled, "Light Reflection Values of White and Colored Paints",* the writer presented some measurements showing the reflection factors of various tinted wall colors, which ranged from 11 to 66 per cent, according to the shade, tint or intensity of color. The results presented in that paper have been of considerable value to decorators in selecting colors for dwellings, hospitals and other public buildings where specific amounts of light are desired. In the same paper, the subject of factory illumination was discussed and the savings in coal or electricity resulting from proper painting were pointed out. Since the interiors of factories are almost invariably painted white, further data has apparently become necessary for the guidance of factory engineers in order that intelligent selections of wall and ceiling colors may be made. Accordingly, more extended tests have just been made in which were included a number of commercial paints obtainable upon the market that have been widely used for increasing the light in factories.

The test pieces selected were of sheet tin, approximately six inches square. They were first primed with a flat white lithopone paint and then given three coats of

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*Reprinted in the author's book, "Paint Researches and Their Practical Applications."

the various paints made up for test, allowing two days for drying between each coat. The reflection factor of each specimen was determined in an integrating sphere in which a beam of light was thrown upon the samples at an angle of 15 degrees to the normal. The surfaces were compared with a freshly surfaced block of magnesium carbonate having a reflection factor of 88. The results appear in chart form below.

It would appear that the instruments now used for determining the reflection factors of painted surfaces do not give as close results as might be desired. For instance, in previous tests* white paints gave only slightly higher reflection factors than white paints that had been tinted to a very faint cream color. It is the writer's opinion, however, that paints which have been tinted to a cream color, or paints which have become soiled or darkened due to exposure, actually give very much less light reflection in a room than paints of a high degree of whiteness. It should therefore be of interest to have tests conducted on a large scale in factories where paints of both types are in service on large wall areas. It is possible that the Sharp portable photometer might be of service for such work.

TABLE 117

Series I.—Reflection Factors on Commercial Paints.

Composition.	Reflection Factor
Aluminum Enamel	52
White Paste Paint that has become slightly yellow with age and dusty	68
Mill White Gloss A	70
Mill White Gloss B	70
White Enamel	73
Mill White Flat	74
Flat Interior Wall Paint	76

There has been considerable discussion as to whether the gloss type of factory paint is more to be desired than the flat type. Because of the glossy surface produced

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*Ibid.

by the former type, it has been assumed that such paints have a higher light reflecting value. This apparently is not so. The tests charted above show that the gloss paints give slightly less illumination than the flat paints. The difference in the reflecting power is due to the higher amounts of pigment contained in the flat wall paints; such paints having approximately 70 per cent of pigment whereas gloss paints usually contain less than 60 per cent of pigment. The percentage and opacity of the pigment present has a direct bearing on the light reflecting value of the paint; the higher the pigment content the greater the reflection. It should, however, be borne in mind that gloss paints are less receptive to dust retention than flat paints; the latter type invariably presenting a rougher surface to which dust will adhere more tenaciously than upon well-hardened gloss surfaces. For this reason some factories may prefer the gloss surface to the flat, whereas other factories in which there is little dust may prefer the flat to the gloss because of the slightly greater illuminating value of the former. Resistance to washing is another factor that deserves consideration. Gloss paints are as a rule more easily washed and stand washing better than most flat paints.

Attention is also called to the results obtained on aluminum enamel, a product that heretofore has been considered as possessed of high reflecting value. It is shown by these tests that aluminum enamel is very much lower in actual illuminating power than white paint.

In pursuing the investigation it was thought desirable to obtain actual light reflection values of some pigments used in the paint industry and for this purpose there was prepared a series of paints in which each pigment was used in the same amount by weight in a flat wall liquid such as is in common use for the manufacture of flat wall paints. This liquid was reduced with a considerable amount of thinner, as the formula will indicate. The results obtained, which are of a tentative nature, are charted below:

TABLE 118

Series II.—Reflection Factors on Various White Pigments, Each Made Up on a Weight Basis with the Following Formula:

Pigment	66.6%
Flatting Oil	10.7%
Turpentine	22.7%

Pigment.	Reflection Factor.
Leaded Zinc (35% Lead)	78
Lithopone	80
Leaded Zinc (5% Lead)	80
Zinc Oxide (American Process)	81
Zinc Oxide (French Process)	81
Magnesium Carbonate dry was taken as the standard at	88

When used in the same formula as given in Series II, titanium pigment (16% TiO_2) gave a reflection factor of 80. Titanium pigment (25% TiO_2) gave a reflection factor of 81 in Series II.

Pigments of low specific gravity when ground in oil have a high light reflecting value whereas many heavy pigments in oil have comparatively low reflecting values. This is due to the marked difference in oil absorption of the two types. The low gravity pigment paints were bulky and white; the high gravity pigment paints were less viscous and of a lesser degree of whiteness due to the larger amount of vehicle present. Had the paints been made up by using the same amount of pigment by volume rather than by weight, different results might have been obtained. However, a formula was selected which allowed for the manufacture of paints, all of which could be applied by brush, and which approximately corresponds to the formula in use in the manufacture of many interior wall paints—66 2/3 pigment, 33 1/3 liquid.

Another series of tests were then made to determine the light-reflecting properties of several common pigments when made into paints on the volume basis rather than on the weight basis. These paints were tested in the same manner as those in Series II. Their composition and reflection factors are given in the attached chart. It will be noted that they were all ground in refined linseed oil, the varnish mixture used in Series II being impractical for the volume formula used, due to the stiffness of some paints made with pigments of light gravity.

Special attention is called to the results shown with

TABLE 119

Series III.—Reflection Factors of Pigments Made Into Paints on the Volume Basis.

Number	Pigment	Specific Gravity of Pigment	Pounds Pigment per solid gallon	Grams Pigment to 65 grams oil (Refined Linseed)	Percentage weight of Pigment in Paint	Reflection Factor
1	Lithopone	4.25	35.4	106	61.9	73
2	Zinc Oxide (French Process)	5.60	46.6	140	68.2	77.5
3	Zinc Oxide (American Process)	5.60	46.6	140	68.2	77.5
4	Zinc Oxide (5% Leaded)	5.66	47.1	141	68.4	73
5	Zinc Oxide (35% Leaded)	5.89	49.0	147	69.3	71
6	White Lead	6.62	55.1	165	71.7	65
7	White Lead*	6.62	55.1	165*	78.5	76*

Magnesium Carbonate dry was taken as the standard at 88.

*In this test only 45 grams of oil were used with 165 grams of white lead pigment, in order to show the effect of increasing the pigment percentage.

Titanium pigment (16% TiO_2) and also titanium pigment (25% TiO_2) each gave a reflection factor of 80 in Series III when used in the same volume basis formula.

white lead having a reflection factor of 65. This pigment, when added in sufficient quantity to constitute 78.5% of the paint, then gave a reflection factor of 76. This result gives further confirmation of the findings that the light-reflecting value of a paint increases with pigment concentration.

It is of the greatest interest to note the extraordinarily high reflecting value of most of the paints used in Series II. They were ground in a minimum amount of liquid, more than two-thirds of which was of a volatile nature. These paints when dry contained more than 84 per cent of pigment and less than 16 per cent of liquid binder. The previous tests* made by the writer were on paints of normal composition, averaging from 65 to 70 per cent of pigment and showing reflection values of 62 to 66. In this connection it should be pointed out that dry white magnesium carbonate, which is taken as a standard for reflection measurements, has a value of only 88, which value was very nearly approached by many of the paints on which results are charted above. This result is in further confirmation of the writer's findings that increase in pigment means increase in light reflecting value and explains why flat white paints of high pigment content give greater reflection than white paints of low pigment content. Moreover the results point to the possibility of producing paints of extraordinary whiteness and exceedingly high light reflecting values. In the writer's opinion a flat wall paint requires a relatively small amount of solid binder to give permanent results. Through the use of heavy bodied oils, cut down with volatile thinners, paints of this character may be obtained which will show reflecting values from 20 to 25 per cent greater than many paints of this character now on the market. It is, of course, necessary to have present sufficient binder to prevent settling of the pigments in the container and to produce a film that is washable and

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*Ibid.

resistant to decay. Similarly, gloss paints of greater hiding power may be produced by increasing the pigment content through the use of less viscous liquids.

Experiments were also made to determine whether a practically colorless liquid that has not heretofore been used in wall paints, would increase the light-reflecting properties of a pigment. The liquid referred to is a plasticized nitrocellulose solution. This was mixed with lithopone and applied in three-coat work. The rapidity of drying, due to rapid evaporation of solvents, prevented the paint from flowing as well as desired. It was found, however, that each coat would dry hard in less than 20 minutes and could then be re-coated. This feature would make the use of such a product of interest where rapid work was considered necessary. The finish was very white, flat and washable. It is probable that such a liquid could be used successfully in mill whites, and it is believed that the absence of drying oils would prevent the yellowing that is often observed with some oil paints.

TABLE 120
Series IV.

Formula		Reflection factor
No. 1.		
Lithopone	50	
Nitrocellulose solution	50	72
	100	
No. 2.		
Lithopone	55	
Nitrocellulose solution	35	76
Added volatile solvent	10	
	100	

It will be noted that increase in pigment concentration and use of a more fluid liquid gave higher reflection values to Formula No. 2.

As bearing on the subject of the illuminating power of paints some quotations are made from an article entitled, "The Lighting of Buildings," which appeared on page 227 of the May, 1919, issue of the Journal of the Institute of American Architects:

* * * "This question of painting is obviously one that should receive the most careful attention of every architect when specifying the interior finishes in buildings used for commercial purposes, and where the cost of operation is a material factor. In one very large office building, the suggested change from light buff-tinted ceilings and dark buff-tinted walls to the kind of treatment suggested above would have saved in energy consumption for lighting about $14,000 a year. Under these conditions it would obviously pay to use a paint that can be thoroughly cleaned without injury and to pay the cost of the cleaning at least once every six months." * * *

* * * "Since all but a comparatively small portion of the light emitted by lamps is initially received on the ceiling, any reduction of the reflection factor of the ceiling means a corresponding increase in the number of lumens that the lamps must emit. A change in this reflection factor from 50 per cent, which is the reflection factor of a light buff lead and oil, to 70 per cent, which is the reflection factor of fresh white lead, means an increase in illumination on the working surface in the room of nearly 50 per cent, if the treatment of the wall-surface shows a reflection factor not less than 50 per cent. It is not too much to say that the selection of the wall and ceiling coatings is more important than the selection of any particular type of reflector equipment on the lamp." * * *

* * * "It has been found that lead-and-oil coatings lose their reflection factor quite rapidly—about 15 per cent a year, independently of dirt accumulations—due entirely to chemical changes in the paint. In the case of kalsomines, and similar paints, the loss in reflection factor is about the same as with lead and oil, but due to the absorption of dirt into the porous paint." * * *

* * * "Where tinting is considered necessary, it should be remembered that lamp-black has an exceedingly low reflection factor, so that even a small portion of it mixed into the paint will reduce the reflection factor of the paint materially. If tinting is essential it should be obtained in other ways than by the use of lamp-black. For instance, a series of greys can be obtained by mixing various red and blue pigments and thinning them out with white. Such greys have a lively appearance and lack the dead quality of the grey obtained by the use of lamp-black. They also have a considerably higher reflection factor than similar tints obtained with lamp-black." * * *

CHAPTER XXVII

Dark Wall Colors for Operating Rooms

An advanced idea in the decoration of operating rooms has recently been presented by a San Francisco surgeon in an interesting article which is reprinted below by joint permission of Dr. H. M. Sherman and the editor of the *California State Journal of Medicine.* The eye strain, produced by certain brilliant high gloss colors, is discussed and recommendations made for the use of dark shades. It is probable that at least modifications of Dr. Sherman's suggestions will ultimately be adopted in hospitals throughout the country. Unfortunately, no mention is made of the results that might be obtained through the use of flat wall colors of the washable type, since the non-reflecting surfaces presented by such paints might be of advantage in such rooms, although, as a rule, gloss paints are used on account of their resistance to frequent washings with antiseptic solutions. The attention of hospital authorities should, however, be directed to this feature of interior painting by the master painter having such work in charge.

In a private communication to the writer Dr. Sherman states that a postscript should be added regarding the gowns, sheets, table covers, and towels, which he uses. Black was found to be rather somber, and, while it was theoretically the best, optically it did not prove to be absolutely essential; therefore a dark blue cotton fabric was selected for this purpose.

THE GREEN OPERATING ROOM AT ST. LUKE'S HOSPITAL

By HARRY M. SHERMAN, A. M., M. D., San Francisco Polyclinic

The difficulty I have had in seeing into the mouth of a small child to properly trim and suture the soft palate and uvula, in cases of cleft palate, led me, some ten years ago, to use black cloths around the mouth instead of the ordinary white toweling. With the white environment, the hollow of the mouth is in the deepest shadow and cannot be comfortably seen, unless it be specially and over illuminated, as by the wearing of a headlight by the operator. With the black environment, however, the mouth is in the high light, not in the shadow, the pupils of the operator are not dominated by the light reflected from the white towels, and ordinarily bright daylight is ample illumination for the work in the back of the mouth.

The discomfort I have had in the present-day white operating rooms led me to suggest that we have dark floors and wainscots in these rooms, so that the operator who looks up from a wound shall not encounter a glare of light and find his eyes useless for a moment, as he looks back into the less well illuminated wound. The color scheme, it seemed to me, should start from the red of the blood and of the tissues, therefore I advised that green, the complementary color to red, should be chosen for the color of the floor and wainscot. The particular shade of green to be selected was that which was complementary to hemoglobin, and it was found to be the green of the spinach leaf. Incidentally, it may be said that the iron in the chlorophyl of spinach is said to be the same chemical combination as is the iron in hemoglobin, but I know nothing of the value of this, in making spinach green complementary to hemoglobin red.

A room painted in this way, the floor and the walls for six feet from the floor, a bright spinach green, and all above a glazzed white, was matched for use against a room painted a glazed white—floor, walls, and ceilings—in the little operating pavilion built at St. Luke's Hospital just after the fire. No one who could get into the green room to do an operation ever went into the white room, and after some months of this experience the point was accepted as settled sufficiently to warrant the innovation of a room similarly colored in the operating suite in the new hospital. Here, however, we could not get in tiles as close an approximation to spinach green as we could in paint; the tile for the floor had to be duller and darker and that for the wall darker, but the two shades harmonize and answer the purpose perfectly of preventing the bright daylight from being reflected upward from walls and floors into the eyes.

Above the level of the six-foot green wainscot is white encaustic tiling to the ceiling, and the ceiling itself is a bright buff. This arrangement imitates fairly well the optical environment out in the fields or among low bushes, where the ground of the surroundings, to above the level of the eyes, is green, and the sky overhead is full of white daylight. This, again, is the optical condition for which the eye was originally adapted by natural selection, and it seems only right to reproduce this condition for the eye when it is to be relied upon for quick and accurate work; while the condition of the greatest eye strain—the dead white and glare of snow on ground and bushes—is the optical condition reproduced by the white rooms and the white furnishings.

The green room is lighted by a window which gives us a northern light, and it reaches from about three feet above the floor up to the high ceiling. At first this window was glazed with ground glass, but it was at once seen that the room would bear much more light than the ground glass permitted, for the excess of light, the useless light which fell upon the floor and the lower part of the walls and could not be reflected thence on to the operating field, but could be reflected from a white floor and white wainscot in the eyes of the operator, was all absorbed by the green floor and wainscot. Therefore, the ground glass was taken out and transparent glass put in, giving a distinctly brighter illumination of the operating field. In working with this it was soon noticed that the light reflected into the operator's eyes by the white sheets and towelings was as dazzling and as interfering as was that reflected from the floor, and so the same color scheme was followed out, and green toweling and green sheets of galatea were provided. The color in these however, did not stand the superheated steam in the sterilization. They became a dingy gray. I then decided to surround the whole operation field with black, as giving a surface from which no light whatever could be reflected, using it around all wounds, as I had used it around the mouth in cleft-palate operations and around the vagina in operations there. I not only had sheets and towelings of black, but I had black gowns made, and the coverings for the instrument tables were all of black, and I found that they were exceedingly satisfactory. The only objection I have heard urged against them was the superstitious fear that people coming up for operation would see this somber accoutrement and consider it a color of bad omen. This has proved to be a groundless fear, for patients who have had work done upon them under local anethesia have expressed no objection to the black dress of the operator, nurses, and room.

In the new hospital, as in the old operating pavilion, there is a very decided preference among operators for this green room, as against the other operating rooms, which are finished in the conventional encaustic tiling; and those operators who have taken advantage of the whole equipment, and have used the black table coverings, towelings and sheets, and the black gowns, have appreciated the improved optical conditions which they gave, for the eye was not compelled all the while to receive light rays from every direction when the only rays it wished to receive or needed were those that came from the wound itself. Under ordinary conditions, the eye might be considered as trying to keep out, by pupil contraction, all the extraneous light, and at the same time trying to let in, by pupil dilation, all it really wished to get from the wound; all of which was a definite over strain of the accommodative and visual capacities of the eye, which would conduce certainly to nerve fatigue and consequently to general fatigue. Indeed, it has not been uncommon in this and other hospitals to hear nurses who had to spend a good part of the day at their duties in the operating room, complain of the effects of the brilliant white environment.

I think we should have long ago learned this lesson in optics from our co-workers in the laboratories, for no one ever saw in a laboratory where the microscope was to be used, white tables, benches, and shelving. On the contrary, these are stained a dead black; and in this connection it may be mentioned that some years ago Dr. George M. Gould, then of Philadelphia, suggested that newspapers should be printed in white letters on a black ground, for in the present printed page, with black letters on a white ground, we see the white ground but not the black letters, and we read really from the shadows of these letters, cast on the retina. On a black page with white letters we would see the letters and not the page, and while the effect, so far as understanding was concerned, would not be different, in the one we would be conserving eye energy, whereas in the other we would be and are overworking the eye unnecessarily. The eye is a long-suffering organ, and we are conventional people, resistant to innovations, but whoever takes the trouble to study, either theoretically or practically, fatigue in its relation to efficiency, will learn that the conservation of energy, even in such matters as saving eyework, where that is possible, is a valuable consideration, and those of us who have long and difficult operative procedures to do will find that this conservation may easily be a deciding factor in success or failure of fine manipulations.

We have tried to test light efficiency in the two kinds of rooms by a test card, such as is used by oculists, put into the bottom of a pasteboard box, which was lined with black. We found that it could be read, down to the smallest type, in either room, but it was plain that it was much more comfortable to read it in the green room, and that probably is the measure of the room's value—that you can work in it with much greater comfort than you could in a room where the optical conditions were more trying. The acme of discomfort would be, I should imagine, what I once saw in an eastern hospital, where the operator was working in a brilliantly lighted, dead white room, and had to wear an electric headlight to over illuminate his field, in order to see in spite of the glare. In the same room I provided myself with yellow spectacles to put on to protect myself from the discomfort of the light.

CHAPTER XXVIII

Notes on the Effect of Colored Light Upon Plant Growth and Pigmentation

With a Suggestion Regarding the Precipitation of Paint Pigments Under Colored Light

Noticing the pale appearance of the leaves of a plant kept in a room painted and decorated with various shades of blue, the writer assumed that the wall color may have had some effect upon the development of the chlorophyl (coloring matter)* in the plant, since the production and continuance of chlorophyl is ordinarily dependent upon the action of sunlight (white light). This observation suggested an experiment to determine the effect of various other colors upon the growth of plant life. Accordingly a number of potted belladonna seedlings were selected because they were easily obtained of the same relative size and in vigorous condition. Potted plants were placed under each of a number of large porous paper cones of sufficient height and width to prevent interfer-

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*A brief research has given the following references which may be of value to those who care to study the subject of plant etiolation:

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FIG. 42
Belladonna Exposed to Influence of (Left) Blue Paint, (Right) Orange Paint.

ence with the growth of the plants. The interior surfaces of each cone were painted with relatively pure colors in distemper form, including white, red, orange, yellow, green, and a purple tone of solid blue. One set of the tests was maintained in the laboratory in a position to receive indirect sunlight, and another set was placed on the roof of the laboratory, in direct sunlight. Control specimens of the potted plants were also maintained without the cones. The plants were watered daily.

Within three days it was noted that some of the leaves of the plants under the purple tone blue color had bleached out to a light yellow. One yellow leaf had broken off at the stem. There was marked evidence of what might be called plant fatigue. When the plants were returned to sunlight, they were restored to their normal, vigorous condition, the yellow leaves rapidly becoming green again.

Under the green cones, some yellowing of the leaves was shown, but the plants were apparently in good physical condition. The red, yellow and orange colors apparently had no marked effect either upon the pigmentation or growth of the specimens. The plants under the white cone were vigorous and of normal color. Some of those under the black cones had grown much more rapidly than the others and had fully maintained the original green color of the leaves. This latter result, in so far as the growth was concerned, was at first attributed to the fact that the earth might remain damper for a longer period in the pots under the dark cones; the greater resulting humidity causing a more continued growth. However, this test indicated that illumination is not always necessary for the continuance of plant life, and that sunlight is not always required to produce or at least maintain the green coloring matter.

Another series of tests were then made in three small chambers constructed of wood and provided with means for admitting air. The interior of the chambers was painted in red, yellow and purple blue respectively. The roof of each chamber, where the light was admitted, was covered with colored glass to correspond with the color

FIG. 43

Belladonna Exposed to Influence of (Left) Blue Paint and Glass (Right) Yellow Paint and Glass.

of the walls. Belladonna plants in one test and tomato plants in another were placed in the chambers for one week. The results corresponded to those obtained under the paper cones, except that the etiolation was more rapid in the case of the purple blue. One plant showing yellow leaves was restored to normal condition by removing it from the box and placing it near a quartz tube mercury light sending forth strongly actinic rays.

While the experiments outlined above are but of a preliminary character and cannot be relief upon to indicate what might happen with other types of plants under similar conditions, or even the same plants under longer

FIG. 44

Tomato Plant Exposed to Influence of (Left) Blue (Right) Red Paint and Glass.

periods of exposure, they are nevertheless of some interest as indicating that the color of the interior sash of greenhouses might for some plant life preferably be of a dark shade instead of white. Possibly a glaze color on the glass, or the use of colored glass might also be found useful in certain instances. A further study of the subject by competent botanists might show that plants having active medicinal, alkaloidal, or aromatic principles could be stimulated to produce larger yields. It is possible, moreover, that plants yielding oil seeds might be so grown as to prevent the development of chlorophyl which causes the yellow-green color in drying oils. A water white oil might thereby be obtained.

The above tests also suggested to the writer that color environment might influence to some extent the shade or tone of pigments precipitated in vessels under colored light. Accordingly, triplicate samples of blue and of an organic red lake were precipitated in colored chambers roofed with colored glass, and left therein for a period of 10 hours before removing to observe the color. While the results obtained were not startling, a slight difference in the tone of some pigments precipitated under different colored lights was indicated. It is believed, however, that a more extended study of this principle might yield information of value to color makers. To the lithopone manufacturer there is suggested the possibility of lithopone precipitation not only under the influence of colored lights but of actinic rays from mercury lamps.

CHAPTER XXIX

Metric Equivalents

The majority of countries use the metric system of measurements. Although this system is now practically standard in American science, it has not yet come into wide commercial application in this country. In order, however, to understand the requirements of South American and European countries, and to harmonize with their standards when required for export trade, it is suggested that the following equivalents be studied. There is first presented the relative measure of the United States and Imperial liquid gallons in comparison with what might be tentatively termed a metric gallon equivalent. These figures are followed by a table of United States and metric systems of weights and measures.

U. S. liquid gallon	231	cubic inches
Metric measure 4 liters	244.16	" "
British Imperial liquid gallon	277.27	" "

Equivalents in Metric and U. S. Systems of Measure.

Cubic centimeter	=	.061	cubic inch
Cubic inch	=	16.39	cubic centimeters
Milliliter	=	.0338	U. S. liquid ounce
U. S. Liquid ounce	=	29.57	milliliters
Liter	=	1.057	U. S. liquid quart
U. S. liquid quart	=	.946	liters
Liter	=	.2642	U. S. liquid gallon
U. S. liquid gallon	=	3.785	liters
Gram	=	.0353	avoirdupois ounce
Avoirdupois ounce	=	28.35	grams
Gram	=	.002205	avoirdupois pound
Avoirdupois pound	=	453.6	grams
Kilo	=	2.205	avoirdupois pounds
Avoirdupois pound	=	.4536	kilo
Metric ton	=	1.102	short ton (2,000 lbs.)
Short ton	=	.907	metric ton

METRIC EQUIVALENTS

For a fuller understanding of the metric system the following table is given:

TABLE 121

Length

10 millimeters .... mm. = 1 centimeter .... cm.
10 centimeters .... = 1 decimeter .... dm.
10 decimeters .... = 1 meter (about 40 inches) .... m.
10 meters .... = 1 dekameter .... dkm.
10 dekameters .... = 1 hectometer .... hm.
10 hectometers .... = 1 kilometer (about 5/8 mile) km.

Mass.

10 milligrams .... mg. = 1 centigram .... cg.
10 centigrams .... = 1 decigram .... dg.
10 decigrams .... = 1 gram (about 15 grains) .... g.
10 grams .... = 1 dekagram .... dkg
10 dekagrams .... = 1 hectogram .... hg.
10 hectograms .... = 1 kilogram (about 2 pounds) ... kg.

Capacity.

10 milliliters .... ml. = 1 centiliter .... cl.
10 centiliters .... = 1 deciliter .... dl.
10 deciliters .... = 1 liter (about 1 quart) .... l.
10 liters .... = 1 dekaliter .... dkl.
10 dekaliters .... = 1 hectoliter (about a barrel) ..... hl.
10 hectoliters .... = 1 kiloliter .... kl.

CHAPTER XXX

Spreading Rates of Prepared Paint Products

In making estimates on the cost of finishing various structural surfaces, data should be available as to the approximate quantity of paint materials required for the work. Since the cost of application is usually several times that of the material, the economy of high-grade products is apparent, especially when such products show as high or even greater spreading values than those of inferior quality.

In the table presented below, there are shown the spreading rates of twenty high-grade painting products. These products were obtained in prepared form and applied under practical conditions to large areas of surface—on wood, wall board, metal, cement, plaster, or brick. In most instances the determinations were made for one-, two,- and three-coat work.

Discussion.—The spreading rate of a paint, enamel, or similar coating may be either low or high, according to the plasticity (body) and other physical properties shown by the product. By extra brushing effort, a paint of good opacity may be spread in such a thin film that even three coat work will not thoroughly hide a surface. The spreading rate should, therefore, be the number of square feet covered by one gallon of the paint so spread that three coats will fully hide the surface. In the tests charted above, normal brushing conditions were followed, and such results obtained.

It is of interest to record here some observations regarding the thickness of paint films as measured microscopically and also as computed theoretically. A paint that spreads approximately 900 sq. ft. per gallon will produce a film having a thickness of .0016 inches (sixteen ten-thousandths of an inch), or, more graphically, 1/600 of an inch. A paint that spreads approximately 650 sq. ft. per gallon will produce a film having a thick-

TABLE 122

Spreading Rates of Prepared Paint Products

(Square Feet per Gallon.)

No.	Product	Surface	1st Coat	2nd Coat	3rd Coat	Av.
1	Prepared Paint (Outside White)	Cedar	841	920	905	888
		Yellow Pine	1015	1024	902	980
		Cypress	876	980	920	925
2	Flat Wall Finish (Interior White	Plaster	321	586	735	547
		Wall Board	695	837	944	825
3	Mill White (Flat)	Yellow Pine	780	821	999	779
		Plaster	450	672	814	645
4	Mill White (Gloss)	Yellow Pine	862	904	920	895
		Plaster	558	1060	1120	913
5	White Enamel (Gum Base)	Cedar with 3 coats of Primer	747	....	....	747
6	White Enamel (Oil Base)	Cedar with 3 coats of Primer	1083			1083
7	Concrete Coating (White)	Concrete	247	466	566	426
8	Brick Coater	Brick	462	620	880	654
9	Metal Paint (Red Oxide Base)	Black Iron	1247	1072	1170	1163
10	Red Lead (Prepared Form)	Black Iron	1095	1055	1116	1088
11	Aluminum Enamel	Black Iron	1288	870	962	1040
12	Auto Finish (Black)	Black Iron with 2 coats of Primer	874	....	....	874
13	Exterior Spar Varnish	Oak	847	914	1092	951
14	Interior Finish Varnish	Oak	632	774	791	732
15	White Shellac	Oak	632	725	....	678
16	Orange Shellac	Oak	803	791	....	797
17	Varnish Stain (Oak Finish)	Oak	681	....	....	681
18	Oil Stain (Oak Finish)	Oak	626	....	....	626
19	Floor Varnish	Oak	834	1053	1032	973
		Linoleum	1184	....	....	1184
20	Floor Paint (Gray)	Yellow Pine	882	1236	1308	1142

ness of .0025 inches (twenty-five ten-thousandths of an inch), or 1/400 of an inch.

The character of surface has a vital effect in determining the spreading rate of a painting product. For instance, the first and second coats on plaster, brick, or cement are usually low on account of the porosity of these materials. After the porous surface is well filled, however, additional coats are of the normal spreading rate shown on many other surfaces. On wood, the spreading rate will vary with the density of grain and resinous character of the species. Woods presenting a hard, resinous surface absorb but little paint, while those that are soft and relatively free from resins or oils show high absorption. Very high-spreading rates are obtaining with the first coat of paint applied to the dense, smooth surfaces of iron and steel sheets. The second coat usually shows a slightly lower figure than the first. This is due to the fact that primer paint films may not be as smooth as the bare metal. The second coat being built up, however, affords a smooth area for the third coat which usually shows a high spreading rate.

The percentage and character of solids or pigments in a paint constitute additional factors which have a determining influence on the spreading rate of the product. Fine fume pigments (carbon black, zinc oxide, basic sulfate white lead, leaded zinc) give thin films, chemically manufactured or precipitated pigments (basic carbonate white lead and lithopone) present films of medium thickness; crystalline and earth pigments (barytes, silica china clay, ochre) show relatively thick films. Paints containing a large amount of pigment cover a limited area. Those containing a medium amount spread over a much greater area. The latter type are therefore more economical, provided they are sufficiently opaque. For the above reason, paints that are very heavy are not to be considered as surperior to those of lower weight per gallon, provided they do not hide a surface in two or three coats any better than the lower weight paint, and further provided that the durability on exposure of the two is the same. Economy in weight means economy in freight and effort of application.

It is of special interest to contrast the spreading rates shown by the modern type of white enamel made with heat-treated oils (1083 square feet per gallon) to that of the old type enamel made on a gum base (747 square feet per gallon). The much greater spreading power of the former, its superior degree of whiteness, and its excellent flowing and brushing qualities are features which have brought it into universal use.

It is of further interest to note that paints made on a varnish base, such as mill whites, floor paints, etc., are now compounded so that they may be spread over practically the same areas as those made with raw oil. This is an accomplishment of the paint industry that deserves special attention, since varnish paints are being more generally adopted for certain special purposes where heretofore oil paints have been in use.

CHAPTER XXXI

A Study of the Practicability of Spray Painting

The war placed such great demands upon the painting industry that it was found necessary to utilize every means to accomplish the huge painting program that developed. The shortage of men capable of wielding brushes soon became apparent and the great speed demanded in production developed as a vital factor. As a result, the steel hulls of many vessels, the rough siding of many temporary buildings, and the surfaces of thousands of guns, tractors, and other military equipment were coated by the spray machine. Because of the speed obtained through the use of this device for preserving or camou-

FIG. 45
Spray Painting the Brick Walls of a Factory

flaging materials of warfare, attempts have been made to develop it for peace time painting purposes. To many observers the question has come as to whether the machine is of sufficient practical value to merit a permanent place in the art of painting and whether it will to any extent replace the old-time hand paint brush.

Some observers have stated that hand-brush manufacturers cannot produce sufficient brushes to apply the paint for which such great demands exist. While the apparent shortage of bristles might to some extent be held responsible for such a situation, it has been suggested that even though bristles were as plentiful as at any time previous to the war, it would be difficult for manufacturers to produce an over-abundance of brushes for coming

FIG. 46
Factory Ceiling

needs, and that the brush industry could not therefore be injured through the development of the spray-painting machine.

Similarly it has been advanced that the occupation of the journeyman painter could not in any way be injured by the adoption of spray painting for certain special classes of work, since it is often impossible to obtain sufficient labor to apply the paint for which such enormously increased demands exist. Furthermore, it has been claimed that unless some means is provided for at least partially relieving the situation, millions of dollars of loss may result from the surface decay that will take place on unpainted structures. It is undoubtedly true that painting was neglected to such an extent during the war period that immediate action must now be taken to preserve the property that will otherwise be damaged if longer left unpainted. This means that during coming years an even greater demand will develop for paint and varnish products. Any legitimate and satisfactory means for the application of these products should therefore be welcomed not only by the master painter but by the journeyman painter and the public. The use, for instance, of the spray machine (if found practicable) will not only be of service to the property owner but will actually stimulate the employment of and demand for painting labor. To make this more clear, it has been suggested that a comparison of the situation be drawn with the effect of the sewing machine upon the tailoring industry. The journeyman tailor undoubtedly at first looked askance at the development of such a machine. It was soon found, however, that this machine created an almost entirely new industry in the production of ready-made clothing. The availability of these products at once effected a great demand and increased usage. As a result, thousands of operators were required where but hundreds were employed before.

It is believed, therefore, that any device that creates

FIG. 47
Cement Stucco

new business in new fields is to be given the hearty support of all, if found to be of a practical nature. Whether or not the machine will prove useful will depend upon the results obtained by the painter during the coming period of great activity in his trade. He may, for instance, find it well suited for certain classes of work and unsatisfactory for other kinds.

Some observations made by the writer on tests with the spray painting machine may be of interest in judging of its possible usefulness. The tests described below were made on large surfaces with both hand brushes and spray machines. After a sufficient period of exposure, data will be available as to the comparative durability of each type of work.

FIG. 48
Steel Girders of Elevated Railway

Spray Paint Tests at Washington.

The tests referred to were made in Washington on government buildings by a competent painter. The machine used in the experiments consisted of a 4 H. P. motor, a large air tank, and a 5-gallon paint tank. It required 220-volt direct current. For the roof work the paint tank was hoisted to the roof and two hose leaders were carried from the air tank located on the ground. Two operators could work at the same time with the paint tank which was fitted with two spray guns.

For the exterior work an experienced spray brush operator started the work on one side of the building, and two journeymen with 4½-inch hand brushes started the

FIG. 49
Ball Park Pavilion

work on the other side, which was a duplicate in size, shape and construction of the side selected for the spray test. After the work had been started a journeyman painter entirely unfamiliar with the use of the spray gun was shown how to operate it. He completed the tests, including the walls and roof area. It was apparent that a very short period of time was required to instruct a man to use a spray gun. The tests were of good size and included on the side walls an area of over 8,000 feet and on the roof an area of nearly 9,000 feet.

The paint used for the exterior wall work was a lead paint tinted with ochre, weighing 17.6 lbs. per gallon for the first coat and 20 lbs. per gallon for the second coat. Both paints were easily handled by the spray gun. The paint used on the roof was an oxide of iron paint weighing about 14 lbs. per gallon. The paint used for the interior work was a modern sanitary flat wall paint of the lithopone type, weighing 14 lbs. per gallon. It was apparent that the spray gun would successfully handle paint of practically any weight per gallon.

In doing the first coat on the exterior brick walls, all cornices and trim were cut in with the spray on the side of the building where the spray test was made. On the second coat, however, the cornices were cut in by hand with a brush, in order to assure a neat job. The time of the brush work was counted in as spray-gun time.

It has previously been assumed that the average journeyman painter, working on wall surfaces and using a hand paint-brush would do about 200 square feet an hour, or about 250 square feet an hour on roof work. It will be noted in the attached tables that apparently much greater speed was attained in the hand-brush work. It is assumed that this was due to the great interest of the painters in the test.

FIG. 50
Metal Roof

Observation of the completed work showed that practically no difference in the appearance of the spray and the hand-brush work existed, with the exception that the spray work was slightly more opaque. The painters in applying the paint by hand with 4½-inch brushes used drop cloths at the base of their work, whereas no drop cloths were used by the spray workmen. There was apparently little paint falling to the ground, the only loss being in the form of a fine mist. On a damp day this mist, of course, would be greatly intensified due to the presence of the volatile constituents of the thinner. This mist would lead an observer to believe that considerable paint was being lost, whereas, as a matter of fact, only a very little quantity of paint was being dissipated as and the effect was largely optical. On the interior work,

FIG. 51
Shingle Roof

mist. The mist was of a somewhat colloidal character however, a noticeable difference was shown. The mist in the room where the paint was being applied by spray guns was very noticeable. Drop cloths were required on the floors in order to prevent staining. Painters employed for continuous periods on interior spray work might to advantage wear a simple form of respirator. The roof work was, of course, subjected to strong currents of air, but there was apparently no very large loss of paint. It was observed, however, that the overalls of the painters using the spray gun became somewhat more soiled than where hand-brush work was being done.

On the interior tests, one room was done by two paint-

FIG. 52
Frame Dwelling

ers with hand brushes and two rooms with the spray gun by one operator. The rooms faced a courtyard in which the machine was placed with hose leaders running up to the work. The ceilings of the rooms were arched, four arches meeting in the centre of each. This made the painting rather difficult by hand but very much easier for spray work. The side walls had four projecting columns, one at each corner, and between the tops of these columns and the arched ceiling there was a heavy scroll cornice. Each room also had a fireplace and chimney breast and large recessed combination windows. The hand work was somewhat marred by streaks and the covering was poor. The spray work was greatly superior. A very much heavier coating of paint was ap-

FIG. 53
Farm Building

parently applied. It was necessary to put on two coats of paint by the hand brush in some instances in order to get satisfactory covering.

Information gathered from some of the journeymen painters indicate that they are not averse to the use of the spray gun after they become acquainted with it. In fact, the painters showed less fatigue at night than when using hand brushes. Some were therefore enthusiastic about its use.

TABLE 123

EXTERIOR WORK

Previously Painted Metal Roof

	Area of Surface Square Feet	Paint Used Gallons	Time 1 Man Hours
Spraying	578	1.49	.5 (½)
Brushing	578	1.35	1.5 (1½)

Results Calculated to 10,000 Sq. Ft.

Spraying	10,000	25.8	8.6
Brushing	10,000	23.3	25.9

Comparative Cost of 10,000 Square Feet of Work

Spraying	25.8 gallons Paint at $4.00		$103.20
	8.6 hours Labor at .90		7.74
			$110.94
Brushing	23.3 gallons Paint at $4.00		$93.20
	25.9 hours Labor at .90		23.31
			$116.51

SPRAYING REQUIRES APPROXIMATELY 10% MORE PAINT THAN BRUSHING.

BRUSHING REQUIRES APPROXIMATELY 200% MORE LABOR THAN SPRAYING.

TABLE 124

EXTERIOR WORK

Previously Painted Brick Walls and Stone Cornice

	Area of Surface Square Feet	Paint Used Gallons	Time 1 Man Hours
Spraying	8,364	10.8	20.
Brushing	8,188	9.87	41.

Results Calculated to 10,000 Sq. Ft.

Spraying	10,000	12.90	23.9
Brushing	10,000	12.05	50.

Comparative Cost of 10,000 Square Feet of Work

Spraying	12.90	gallons Paint	at	$4.00	$51.60
	23.9	hours Labor	at	.90	21.51
					$73.11
Brushing	12.05	gallons Paint	at	$4.00	$48.20
	50.	hours Labor	at	.90	45.00
					$93.20

SPRAYING REQUIRED APPROXIMATELY 7% MORE PAINT THAN BRUSHING.

BRUSHING REQUIRED APPROXIMATELY 109% MORE LABOR THAN SPRAYING.

TABLE 125

INTERIOR WORK

Combined Ceilings and Walls of Plaster

	Area of Surface Square Feet	Paint Used Gallons	Time 1 Man Hours
Spraying	2,600	6.39	5.33 (5 1/3)
Brushing	1,000	1.75	5.33 (5 1/3)

Results Calculated to 10,000 Sq. Ft.

Spraying	10,000	24.5	20.5
Brushing	10,000	17.5	53.3

Comparative Cost for 10,000 Square Feet of Work

Spraying	24.5 gallons Paint at $4.00	$98.00
	20.5 hours Labor at .90	18.45
		$116.45
Brushing	17.5 gallons Paint at $4.00	$70.00
	53.3 hours Labor at .90	47.97
		$117.97

SPRAYING REQUIRED APPROXIMATELY 40% MORE PAINT THAN BRUSHING BUT GAVE QUITE GOOD HIDING IN ONE COAT.

BRUSHING REQUIRED APPROXIMATELY 160% MORE LABOR THAN SPRAYING AND GAVE POOR HIDING IN ONE COAT.

OTHER TESTS

There are given herewith the results of some carefully conducted tests made by a manufacturer of a prominent spray machine. The tests were made on several different types of surfaces, most of which were new, by two operators versed in the use of the apparatus. The time given on all interior work, with one exception noted, included time employed in mixing paint, filling tanks, caring for engine, placing scaffolding and cleaning up. No helpers were employed.

Interior Walls and Ceiling (2 Coats)

Character of Surface	Rough brick walls. Beamed ceiling. Unpainted.
Area (Square Feet)	Ceilings 18,876. Sidewalls and posts 5,764, or total of 49,280 square feet of paint applied in the two coats.
Type of Paint	Blank R Undercoat — then Blank R Mill White.
Gallons Required	175
Spreading Rate per Gal. (Sq. Ft.)	281 average each coat.
Time Required (Hours)	63
Square Feet Coated per Hour	780 each coat.

Ceiling (1 Coat)

Character of Surface	Rough beamed ceiling—unpainted.
Area (Square Feet)	12,858
Type of Paint	Blank D 1 coat made of 2/3 undercoat and 1/3 enamel.
Gallons Required	63
Spreading Rate per Gal. (Sq. Ft.)	204
Time Required (Hours)	44
Square Feet Coated per Hour	292

Stock Room (1 Coat)

Character of Surface	Rough beamed ceiling and joists—unpainted.
Area (Square Feet)	6,390
Type of Paint	Blank D 1 coat made of 2/3 undercoat and 1/3 enamel.
Gallons Required	23
Spreading Rate per Gal. (Sq. Ft.)	278
Time Required (Hours)	10
Square Feet Coated per Hour	639

Stock Room (1 Coat).

Character of Surface	Rough brick sidewalls—unpainted.
Area (Square Feet)	4,470
Type of Paint	Blank A—1 coat made of ½ undercoat and ½ enamel.
Gallons Required	25
Spreading Rate per Gal. (Sq. Ft.)	179
Time Required (Hours)	9
Square Feet Coated per Hour	496

Shipping Room (1 Coat)

Character of Surface	Smooth board ceiling — unpainted.
Area (Square Feet)	1,272
Type of Paint	Blank R-A 1 coat made of 2/3 undercoat and 1/3 enamel.
Gallons Required	4
Spreading Rate per Gal. (Sq. Ft.)	318
Time Required (Hours)	3
Square Feet Coated per Hour	424

Shipping Room (1 Coat)

Character of Surface	Rough brick sidewalls—unpainted.
Area (Square Feet)	2,772
Type of Paint	Blank R-A 1 coat made of 2/3 undercoat and 1/3 enamel.
Gallons Required	12
Spreading Rate per Gal. (Sq. Ft.)	231
Time Required (Hours)	6
Square Feet Coated per Hour	462

Interior Plaster Wall

Character of Surface	New plaster on wall and ceiling of house. Wall first sized with glue.
Area (Square Feet)	Not given.
Type of Paint	Blank L Sanitary Finish Oil Type.
Gallons Required	
Spreading Rate per Gal. (Sq. Ft.)	1,000 (over sized wall.)
Time Required (Hours)	Not given.
Square Feet Coated per Hour	1,030 each coat, not including mixing or filling paint.

Exterior (1 Coat)

Character of Surface	Exterior rough brick wall—unpainted.
Area (Square Feet)	9,120
Type of Paint	Blank P Cement Coater
Gallons Required	41
Spreading Rate per Gal. (Sq. Ft.)	222
Time Required (Hours)	29
Square Feet Coated per Hour	314 (including time of moving scaffolding and mixing paint)

Exterior (1 Coat)

Character of Surface	Old rough brick walls—unpainted.
Area (Square Feet)	12,000
Type of Paint	Blank Q water mixed kalsomine type coating.
Gallons Required	550 *pounds* dry kalsomine type coating.
Time Required (Hours)	22
Square Feet Coated per Hour	545

New Lumber Shed (1 Coat)

Character of Surface	Box siding, rough wood (new).
Area (Square Feet)	6,000
Type of Paint	Blank L. O.
Gallons Required	39
Spreading Rate per Gal. (Sq. Ft.)	154
Time Required (Hours)	8
Square feet Coated per Hour	750

Wood House (Old) 2 Coats

Character of Surface	Very poor surface. Previously painted. Cleaning necessary.
Area (Square Feet)	6,526, including trim and shingles.
Type of Paint	Blank L. O.
Gallons Required	25
Spreading Rate per Gal. (Sq. Ft.)	522 average each coat.
Time Required (Hours)	95

New Shingle Roof on House

Character of Surface	New shingles.
Area (Square Feet)	1,300
Type of Paint	Blank X shingle stain.
Gallons Required	8
Spreading Rate per Gal. (Sq. Ft.)	162
Time Required (Hours)	3
Square feet Coated per Hour	433

Aeroplane Hangar

Character of Surface	New wood—unpainted. No windows.
Area (Square Feet)	6,500
Type of Paint	Blank U. S. Hangar Paint.
Gallons Required	Not given.
Spreading Rate per Gal. (Sq. Ft.)	(Not obtained.)
Time Required (Hours)	9
Square Feet Coated per Hour	722, with one laborer as helper.

CHAPTER XXXII

The Preservative Function of Paint and Varnish*

The decay of structural materials is rapid. The microscope will record the initial start of such decay even after 24 hours' exposure of some building products. The naked eye is sufficiently strong to perceive the erosion often evidenced in a few weeks. Those materials that show relatively slow decay, almost invariably present at first, or later assume, a gloomy color that is objectionable from several standpoints. It is therefore apparent that surface protection through paint or varnish application is quite as important as sound engineering or design.

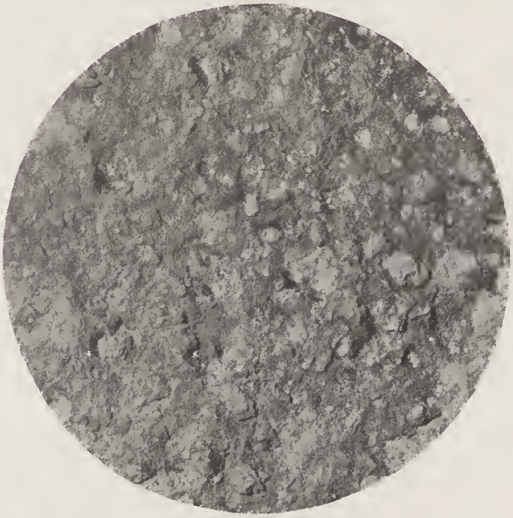

FIG. 54

Photograph of Uncoated Steel Showing Corrosion

The photographic reproductions presented herein illustrate clearly the necessity of providing protection from the elements or from surface wear. Some of the newer uses for paint and varnish are also discussed. It is believed that not only the master painter, but all people who have to do with the upkeep of public or private property, may find something of interest in the discussion.

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*The cost of a coat of paint or varnish may average a few pennies per square foot. The annual decay loss per square foot of surface on many unpainted surfaces may reach several times the cost of painting.

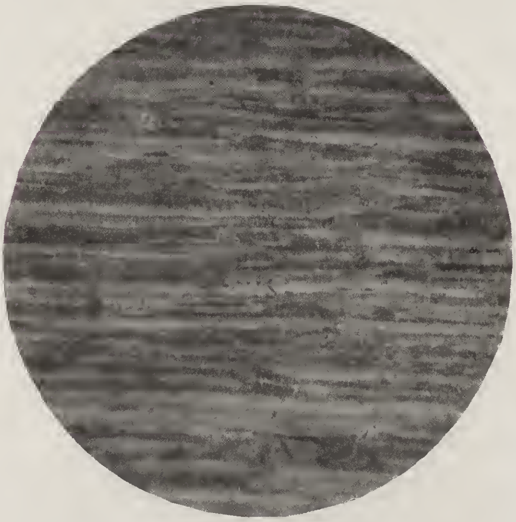

FIG. 55
Photograph of Uncoated Wood (Magnified)

FIG. 56
Photograph of Varnished Wood (Magnified)

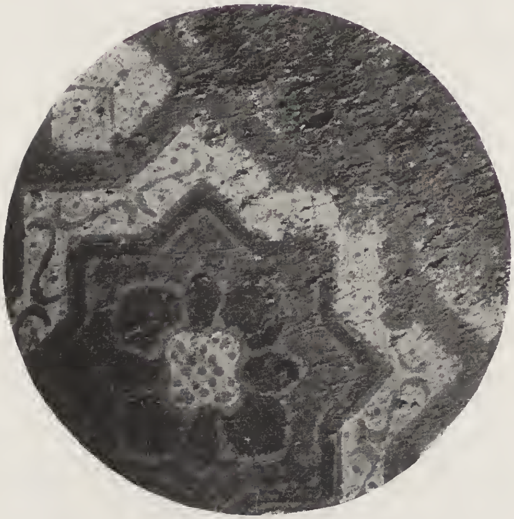

FIG. 57
Photograph of Floor Covering Showing Abrasion of Printed Design

FIG. 58
Photograph of Linoleum (Magnified)

A microscopic view of even the dressed lumber that is used for interior trim in buildings, for musical instruments and cabinet work, for automobile or yacht construction, will show the deep channel like grain of the wood fibre. When varnish is applied, the pores are filled up and a smooth water repelling, wear resisting surface results. The beauty of the grain and coloring of the wood is at the same time greatly enhanced.

The printed designs on some types of floor coverings wear off as a result of foot abrasion. The unsightly dark colored base may then be exposed. Occasional coats of varnish prevent this defect. Even high grade inlaid linoleum is improved and given greater longevity when varnished. The surfaces of all types of floors or floor coverings may be beautified and made dirt and water resisting with floor varnishes.

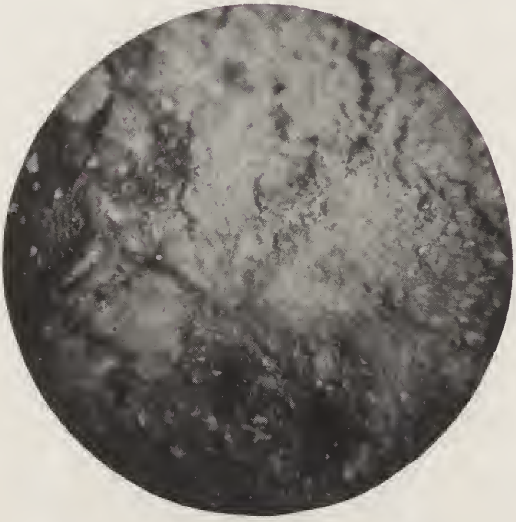

FIG. 59
Photograph of Brick Surface (Magnified)

FIG. 60
Photograph of Brick Section (Magnified)

Building brick may absorb from 10 to 15% of water. If a brick building should be subjected to constant rain

FIG. 61
Photograph of Uncoated Steel (Magnified)

for several days it is conceivable that many tons of water would be absorbed. Dampness might then prevail within the building and in cold weather the tonnage of coal required to heat the interior would probably be greatly increased. The application of paint to brick, fills up the pores and produces a water resisting film. The use of white or light tinted paints improves the appearance of the structure and gives sufficient light reflection to the airways to repay for the expense of painting.

The almost universal custom of painting the interior brick walls of factories is now being extended to cover the exterior walls as well. Many gloomy appearing brick residences are also being transformed by exterior painting.

Metal surfaces may appear smooth to the naked eye but under the miscroscope they often evidence a rough, porous condition. On many metals, this condition is responsible for moisture retention and subsequent corrosion. Such metals as iron and steel demand immediate protection to prevent rust accumulation. The loss suf-

FIG. 62
Photograph of Painted Steel (Magnified)

fered yearly through neglect to keep metal well painted amounts to many millions of dollars.

FIG. 63
Photograph of Uncoated Cement Structure. After Exposure

FIG. 64

Photograph of Painted Cement Structure. After Exposure

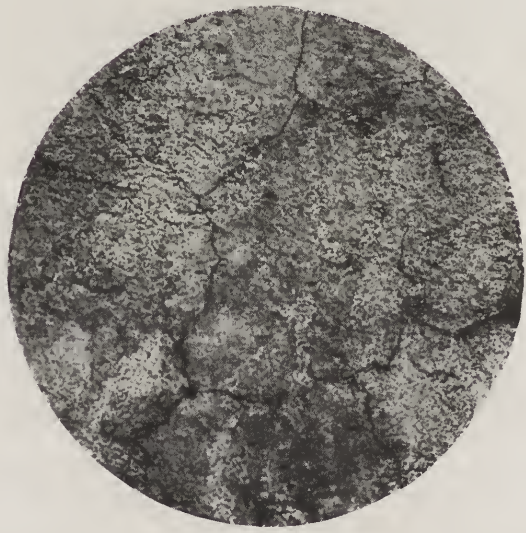

FIG. 65

Photograph of Uncoated Cement Stucco After Exposure

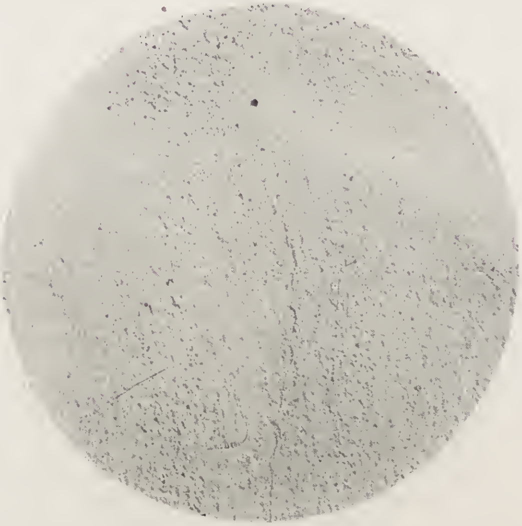

FIG. 66.
Photograph of Painted Cement Stucco After Exposure

FIG. 67
Photograph of Cross-section of Wood, Showing Fibers and Cells
(Magnified)

Cement or stucco invariably present a rough surface. Water absorption is rapid. Dust and soot accumulation is marked, especially in industrial communities. All these defects may be prevented by paint application. It is safe to state that the value of any cement building may be increased from 10 to 20% by the use of a pore filling, water resisting paint. Dust adherence then becomes minimized. Cement structures are almost invariably painted when appearance is a consideration.

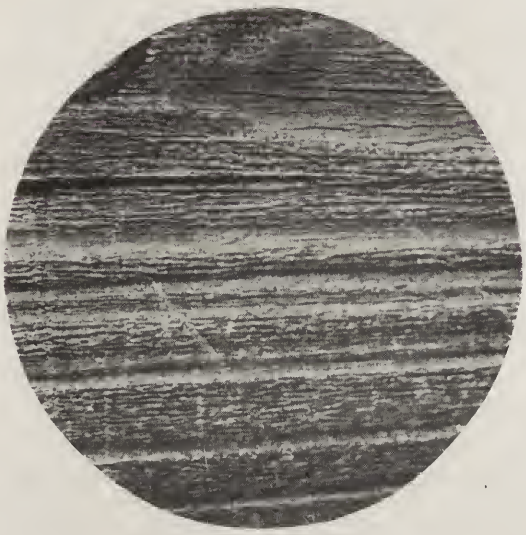

FIG. 68

Photograph of Uncoated Wood. Taken After Exposure to Weather

A wood panel one-half of an inch thick, when exposed to moisture, may gain 10 ounces per square yard in a week. When exposed to water it may gain 50 ounces. Moisture absorption, checking, fungus growth and fibre abrasion may take place on uncoated wood. When coated with paint, the moisture absorption is reduced to a negligible quantity and the surface is preserved from attack. Wood lasts indefinitely if kept well painted.

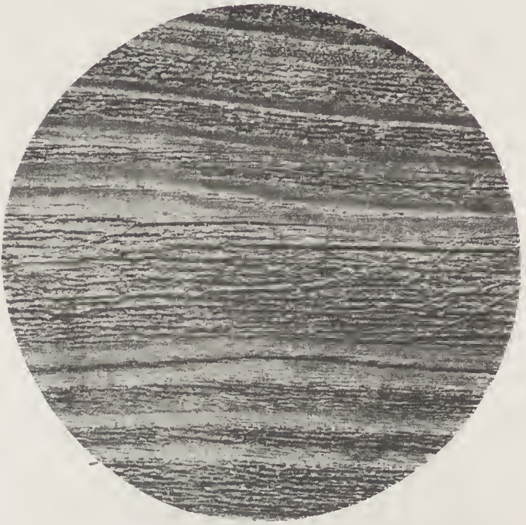

FIG. 69
Photograph of Uncoated Wood. Taken After Exposure to Weather

FIG. 70
Photograph of Painted Wood. Taken After Exposure to Weather

FIG. 71

Photograph of Wall Paper (Magnified)

Wall paper is fibrous and absorbent. It may retain moisture and dust. Paints are therefore generally preferred for wall decoration. On existing papered walls, paints may be applied direct to the paper if desired. A smooth, washable surface of any color may thus be produced.

CHAPTER XXXIII

Paints for the Home

The following information regarding paints for the home is presented in a popular fashion in order to acquaint the average home-owner with some of the general principles involved in painting and varnishing:

An inquiry made a few years ago in the Middle West States as to the value of paint upon farm buildings developed the information that bankers would lend from 5 to 50 per cent more on land where farm buildings were well painted and kept in good condition than on land where they were not. It was maintained that the painted buildings were an indication of thrift, and that a thrifty farmer was a good client to whom money could be safely loaned; whereas the farmer who allowed his buildings to remain in a bad state of repair, through the lack of paint, showed indication that he was slow pay and of poor rating.

It developed that in practically every instance where the buildings on a farm had been repaired when necessary and given proper paint protection, the valuation of the farm immediately increased at least 20 per cent, whereas the cost of making repairs and painting in many instances was probably not over 5 to 8 per cent.

Selection and Wearing Properties of Paint

The serviceability of any paint should be judged by the surface which it leaves for repainting after a period of from four to five years, as well as the durability of the repainting job. It has been found, for instance, that inferior and cheap paints will crack, check and scale to a rough surface. Such defects show through successively applied paint coatings, seriously affecting their strength and detracting from the beauty of the job.

On the other hand, high-grade paints weather gradually without showing marked defects, and when repainting time comes they present a uniform surface which will absorb and amalgamate with the freshly applied paint. Such paints show under the widest range of conditions good hiding power, adhesiveness, freedom from internal strains, permanency of color, water resistance and elasticity.

White lead, the oldest and perhaps the most widely used of all white pigments, is admirably adapted as a painting material, presenting a highly opaque surface when ground in oil, in which form it is the base of paste paints that may be used by the painter. Another pigment that has proved of great value is zinc oxide, the use of which may be said to have almost revolutionized the paint industry. This pigment is of great whiteness, fineness and possessed of excellent hiding power.

In the manufacture of the highest-grade prepared paints the above referred to metallic pigments, in a finely divided form, are thoroughly ground with linseed oil and mixed with a moderate amount of drier and thinner, subsequently being colored with the necessary color pigments to give the desired tint or shade. These paints usually contain from about 62 to 65 per cent of pigment, the balance being liquids. The liquid is usually 90 per cent of linseed oil and 10 per cent of turpentine or similar thinner and drier.

For the darker shades and solid colors, many earth pigments are used in large quantity, such as red oxide of iron, ocher, sienna, umber, and so forth. Linseed-oil paints made with these pigments have also proved very durable upon exposure. The latter class of paints, however, is used most widely upon barns and outbuildings and for metal work.

Effect of Different Climates

A careful study has been made of the effect of various climates upon paints. Immense test structures have been erected for this purpose in many parts of the United States where the prevailing weather conditions were of a markedly different nature. In general these tests have demonstrated the fact that the summer months cause more rapid wearing of paints than the winter months, and that the side of a dwelling facing the sun and the strong prevailing winds is more rapidly affected than the other sides.

Most careful studies of hundreds of different pigments and oil mixtures have been made, and while it is true that some may show a very slight superiority in some regions, the general conclusion from all the tests shows that a high-grade paint will give satisfactory results under all conditions of climate, whether in the coastal region, in the mountains, or on the plains.

What Color to Use and How Many Coats

Property owners should bear in mind the fact that more durable results are obtained with tinted paints than with white paints. Permanent coloring materials ground into a white-paint base have the effect of preventing chalking and checking, and thus increase the longevity of the paint.

One of the many lessons that have been learned from extended tests is the superior painting results obtained with three coats rather than with two coats of paint. Two coats are insufficient to give proper protection to new wood, and on the best types of work four coats are applied. For repainting work, while two coats are usually sufficient, some people believe that the cost of an additional coat of paint is not an expense but a saving in the end, for the repainting period is thereby considerably postponed.

When to Paint

Springtime means clean-up and naturally paint-up time, and it is probable that most painting is done in the early months of the year. However, the dry period during the fall months is said to be the best time for painting, as the house is given protection for the trying period of the ensuing winter. As a matter of fact, however, the time to paint is just before the surface requires painting, regardless of season, provided the weather is clear. Paint applied during rainy weather does not give the service to be expected when it is applied during dry weather.

Kind and Treatment of Lumber

Many inquiries have come to the writer respecting the durability of various types of wood for building purposes, and answer has generally been made to the effect that lumber used for dwellings will last indefinitely if kept well painted.

Striking illustrations of the truth of this statement are afforded by the excellent condition of those century-old Colonial dwellings which have been properly cared for. Such structures may be found in every one of the states comprising the thirteen original colonies, but are most common throughout New England, to whose shores in the sixteenth century came many of the most skilled woodworkers, decorators and other artisans of the mother country. Since many kinds of lumber are represented in these structures, the durability of painted wood is well known by observing their condition.

Many claims are now made for the durability of particular types of wood, such as cypress and California redwood. Some enthusiastic producers have even stated that their woods need no paint. While it is universally recognized that there are many examples of the longevity of these and other species of American woods, people would look askance at the dwelling that was left unpainted.

The modern desire for community betterment demands æsthetic surroundings and properly decorated homes. As a matter of fact, however, any type of wood that is left unpainted will soon become eroded by the weather, spotted with fungus growth and made heavy by the large quantities of moisture that are absorbed. Any species of unpainted wood will darken, warp and become fuzzy and damp.

Paint acts as a wood preservative because it closes the openings in the wood, preventing the entrance of decay-producing organisms. Thoroughly seasoned wood will last indefinitely if kept well painted.

The particular locality in which structures are built often has a bearing upon the type of lumber that is selected, and while it is true that the painting of each type of wood demands the special consideration of the painter, the writer has found that a high-grade paint is well suited for the preservation of every kind of wood, provided the paint is properly treated in the hands of an intelligent workman who can produce lasting results on almost every type and in every climate.

Exterior siding of some grades of wood shows the effect of rosin, contained in the knots, which during hot weather bleeds through the paint, disrupting the film and causing an unsightly condition. This defect is not prevented by the application of shellac to the knots previous to painting, but by proper manipulation of the priming coat of paint, which is referred to below.

One of the chief requisites to a successful job of painting is well-seasoned lumber. Such lumber is not only stronger, but in a much more receptive condition for paint. Paints which are defective in elacticity are affected by the volume changes taking place in partly seasoned lumber, and cracking may occur.

General Rules for Exterior Work

Some general rules on painting the exterior of dwellings are given here:

No paint or other finishing material should be applied in damp weather.

The interior plaster work of a new structure should be allowed to dry thoroughly before applying paint to the exterior of a building, as the water drawn out through the wood might cause blistering.

The surface of the wood must be free from moisture.

Weathering of some types of wood previous to the application of the second and third coats of paint is sometimes advisable in order to allow thorough seasoning and drying out.

If the wood has been previously painted, all old, loose paint that may sometimes be observed should be removed with a wire brush.

If the wood has not been previously painted, all knots and sap streaks should be brush-coated with turpentine not more than one hour previous to the application of the first coat of paint. Never use shellac for coating knots.

Priming Coat.—Never use ocher as a priming coat. Serious results are bound to follow. A high-grade prepared lead-and-zinc paint made by a reputable manufacturer should be selected. To one gallon of paint there should be added from two to three pints of turpentine or benzol. The thoroughly stirred mixture should then be applied and allowed to dry to a hard undercoating which is the foundation of painting success. A drying period of a week should be allowed if possible.

The paint will penetrate deeply into the wood and provide a permanent foundation for the subsequent coats.

Second and Third Coats.—When the priming coat is thoroughly dry, close all nail holes and other imperfections in the wood with a good grade of putty. This should be followed by the application of the second coat of paint, which may be used as it comes from the can. If the paint is thought to be too heavy, a pint of turpentine to the gallon of paint may be used for thinning. After a suitable drying period the third coat may be applied, using the paint as it comes from the can without reduction.

Paints for Shingle Roofs

When mineral paints are applied to shingle roofs, they dry to a durable, water-proof film that has the effect of smoothing the rough, fuzzy surface of the wood, thus eliminating at once an important fire hazard. A shingled dwelling upon which paint has been used is rain-proof. It is, moreover, made very attractive in appearance.

Another important function is performed by the paint in preventing the warping of the shingles at the edges, thus doing away with the formation of pockets in which hot cinders might lodge and burn.

Another characteristic is the resistance of the paint to fire. Although the oil content is more or less inflammable, there is present in the dried film only a minor portion of oil, the major portion consisting of metallic pigments which are unaffected by fire. A hot cinder or spark falling upon a shingled roof properly treated with paint would in most instances roll from the roof to the ground, as there would be no pockets in which to lodge and burn.

The best results are obtained by brush-coating or dipping new shingles. After drying, they are laid and should then be given an additional brush-coating of paint. Some shingle manufacturers are putting the priming coat on at the mill, which process is much to be preferred.

Shingle roofs treated as above outlined are really more fire-resistant than many other types. Because of their low weight and heat-reflecting properties, painted shingles are often preferred to heavier types of roofing. Old shingle roofs may also be painted or stained after they have become thoroughly dry. The most suitable paints for shingles are those especially made for the purpose and containing asbestine and other mineral pigments that are not reducible to metals, ground in linseed oil, thinner and drier.

Shingle stains are also widely used for their preservative qualities and decorative effects. They are generally prepared of high-grade creosote admixed with tinting colors ground in oil. Soft and pleasing finishes are obtained with such stains, especially upon the shingle sides of bungalows.

Paints for Concrete and Stucco

The scarcity of other building materials in some communities has led to the use of Portland cement in enormous quantities. Its general availability, combined with its adaptability to the principle of reenforcement, has developed for this material many new applications in the building world. Throughout the country its use is seen in the construction of dwellings, factories, silos, culverts, tanks, posts, barns, creameries, and so forth. For creameries it has become most valuable to the producer of certified milk products, on account of its cleanliness and resistance to attack from vermin.

In the suburbs of the larger cities there is seen an ever-increasing use of Portland-cement stucco either upon brick or upon metal lath over frame buildings. In the urban communities its use is observed in the construction of factories. schools, hospitals and pavilions. With this ever-increasing use has come a desire for appropriate surface treatment, and such treatment will lead to a much wider use of this material.

If left unpainted, the dull, drab appearance of the cement is unpleasing, and its rough surface will absorb considerable quantities of moisture, attracting dust particles and soot from the air, which will give a most unpleasing and unsightly condition. For these reasons there is a growing tendency among home owners to paint all cement structures.

It should be remembered, however, that the alkaline lime present in freshly laid cement becomes reactive in the presence of moisture, and under such conditions might prove destructive to freshly applied paints. For this reason it is customary to prime the surfaces of all freshly laid cement work with a solution of zinc sulphate crystals dissolved in water in the proportion of three pounds to a gallon. This colorless solution is brushed on and a chemical reaction ensues, which transforms the surface lime into a neutral compound, calcium sulphate.

After drying for a day, ordinary paints such as are used upon frame buildings may be applied, or high-grade prepared cement coatings which are especially designed for the purpose. Both types of paint dry upon cement surfaces usually to a semiflat film which is very pleasing in effect. Their value lies in the fact that they render the cement damp-proof and also give to it a smooth surface which will resist the accumulation of dust and dirt.

Paints for Metal

The need of paint protection for the metal work about our homes and other buildings seems to be generally understood, but there is a lack of understanding of the proper kinds of paint to use for this purpose. Steel structural members, such as columns and girders, iron railings, fences and grills, tin roofs, galvanized-iron rain gutters, spouts and flashing and all other corrodible metal exposed to the air and moisture require paint protection. If the paint used for this purpose is a cheap one, it probably contains pigments which excite the formation of rust. Under such conditions, repainting will be necessary within a short time.

Practical tests have shown that ultimate economy is effected by using only the highest grades of paint for metal protection. For bare iron and steel, a first coat of paint containing a basic pigment such as red lead should be used, over which may be applied a high-grade moisture-resisting paint of any color desired. On tin roofs a good grade of red oxide of iron paint is generally applied, after making sure that all oil and grease are removed from the surface of the roof by rubbing with a cloth saturated with benzine.

Galvanized iron presents a special problem to the painter, as will be understood by anyone who has observed paint peeling from the surface of an improperly treated galvanized-iron structure. To secure the best results, a new galvanized-iron surface should be brush-coated with a solution of copper sulphate, chloride or acetate, made by dissolving four ounces of any one of these salts in one gallon of water. This solution slightly roughens the galvanized coating, but does not damage it. After an hour or so the surface may be dusted off and then painted with a high-grade prepared metal paint. Well-painted galvanized iron should last for many years, as it has the protection afforded by the galvanizing as well as the paint.

Paints for the Interior

Of whatever material the exterior of a home is made, the interior trim is invariably of woods of different types, and it is the decoration of this interior trim that makes the home livable and lovable. The selection of decorative schemes which harmonize with the furniture and draperies to produce various effects desired in the different rooms is most important.

As a rule, in all modern houses every bit of the trim, from the kitchen to the bathroom, is finished in white or ivory zinc oxide enamel, which gives to the structure an immaculate appearance and a contrast to the different color effects used on the walls.

One property of interior white paint is its so-called dirt-exposing character. Insanitary conditions are impossible where finishes of this character are used and kept clean. Moreover, they form a most pleasing contrast with the cretonne hangings, mahogany furniture, and bric-a-brac. White interior finishes are made either with enamels or with interior oil paints, the latter being similar to those used for exterior work, but having a clearer tone.

Interior oil paints require less effort to apply than enamels and as a rule are easier to remove should a change to some other type of finish be desired. These oil paints, however, are as a rule used on the less expensive types of dwellings. For higher-grade work the enamel finishes are preferred. These enamel finishes are really varnish paints which require careful undercoating and rubbing to produce the best results; from three to four coats of opaque white paint being applied on the woodwork before the application of the enamel. The enamels dry generally to a very high gloss surface which may be rubbed if desired to a dull surface. Either finish will remain in excellent condition for many years if properly cared for.

For high-grade interior trim that is to be treated with white paint or enamel, soft woods that are fairly free from rosin are as a rule selected because of their absorptive and nonstaining properties.

Where varnished or stained work is desired for interior work, hardwoods are generally used because of the beautiful grain and finish which may be produced thereon. Varnishes for interior work are usually of two types, although many producers make a grade that is fairly well suited for all types of work.

For floors there is generally used a varnish which dries rapidly to a very hard, abrasion-resisting surface. Such a varnish is usually made with a large content of fossil resins dissolved by heat in linseed and Chinese wood oils and thinned with turpentine or similar thinners. On floors such varnishes give vastly superior results to those often referred to as spirit varnishes, which are made of shellac dissolved in alcohol.

Even though a floor is to be finished with wax, it is desirable first to preserve the wood with varnish, and the above outlined procedure should first be carried out, subsequently applying the floor wax and polishing it with a waxing brush.

Refinishing Old Floors

For old floors it is often advisable to apply a modern floor stain to hide imperfections, and then to apply a varnish, or, if desired, the so-called varnish stains may be applied in two-coat work. On rough, splintery floors that could not be made to present a good appearance through the use of varnish, it is customary to apply so-called floor paint which contains color pigments of various kinds and dries to a very hard, durable, wear-resisting surface.

Another method of finishing floors is to scrape them thoroughly so that a smooth surface may be offered. After lightly sandpapering, a coat of liquid wood filler is applied, and then two coats of varnish, allowing at least a day between coats. Many painters omit the filler and apply three coats of varnish, with excellnet results.

Sanitary Value of Interior Finishes

For the interior trim, paneling, cabinet work, and so forth, so-called interior finishing varnishes are used, which usually are made on a Chinese wood oil base with a lower content of resin. Chinese wood oil, which has become one of the prime requisites of the varnish industry, is an oil expressed from the nuts of a tree grown in Manchuria. It has remarkable varnish-making properties, being used as the base of most of the water-resisting varnishes now made. It is with this type of varnish that tables are usually finished, where resistance to hot water and other destructive agents is required.

The decoration of the interior of dwellings has become of even greater importance in one sense than the painting of the exteriors. The custom in many modern dwellings is to use paint on the walls, in order to obtain the various color effects desired, and in this fashion maintain the most sanitary conditions possible.

From the standpoint of sanitation and hygiene, properly painted walls are superior to papered walls upon which bacteria may in some instances be harbored, where, for example, it has been the practice to repaper without taking off the old paper which, having been glued with organic pastes, acts as a culture medium. Usually both tenant and landlord are oblivious to conditions which cannot readily be seen or detected, and these conditions have therefore not been given very much thought.

From many tests that have been made, the writer is convinced that one of the most efficient means of destroying bacteria in quarters previously occupied by persons suffering from contagious disease is to be had by painting the rooms with an oil-pigment paint. The vapors from such paint develop formaldehyde, an antiseptic which is fatal to bacilli. In hospitals where it is necessary to maintain sanitary conditions, the walls are invariably painted with paints that can be frequently washed. It is just as important that similar precautions should be taken in the home.

Sanitary Wall Paints of the Oil Type

For wall work there are now used paints which are referred to as prepared, sanitary, flat wall paints of the oil type. Such paints when applied produce waterproof films which may be frequently washed without disturbing their color or durability. They dry to a flat or matte, velvet-like surface which has no glaring effect upon the eye and gives highly pleasing color effects. These paints can be made in practically any tint desired.

On the borders of the walls so decorated attractive stencil designs bring out in relief the color combinations that have been used; and in recent years methods have been developed for producing so-called Tiffany effects, consisting of blended colors which give the appearance of tapestry wall papers. The ease with which these materials may be applied, their rapidity of drying, their soft color effects and their durability have brought them into almost universal use for interior wall decoration.

On old and dry walls these paints may be applied as they come from the container, without reduction. If desired, a varnish size containing a little of the color may first be applied to reduce the suction, and then be followed by one or two coats of the paint. On newly plastered walls the paint should be reduced with a little oil and drier and then applied in two-coat work. Drying-out of the plaster and the development of free lime may in some instances cause slight fading out of the colors, especially if they are of light tints. This, however, may be expected on new work, and the application of a third coat of paint several months after the first two coats will produce a very good finish.

The Light-Reflecting Power of Paint

In addition to the antiseptic value of interior painting, the subject should be considered from the standpoint of illumination and psychological effect. Little thought has been given to the light-reflecting power of the interior surfaces of buildings. These surfaces, however, constitute a more important factor than the type of illuminant used. This is shown by the fact that the illumination of an interior space lighted by a tungsten lamp can be made to vary from 12 to 67 per cent by applying different wall colors, ranging from white to dark green. Some tests made a few years ago by the writer showed the following coefficients of reflection of wall surfaces painted in various colors:

TABLE 126

Light Reflection of Colored Paints

Paints made on a mixed white pigment base, tinted with chrome yellow, chrome green, Prussian blue, Para red, ocher, sienna, carbon black, and so forth.

	Coefficient of reflection
Light cream	66
Light pink	60
Light yellow	58
Light blue	55
Light greenish yellow	54
Light buff	52
Light green	42
Light terra cotta	41
Medium terra cotta	39
Light greenish blue	36
Medium blue	32
Warm green	19
Medium green	14
Red	12
Blue, dark	12
Green	11

The illumination of factories, railroad terminals, department stores, hospitals and office buildings can very easily be increased by the application of white paints or light-tinted paints. In fact, it has been shown in practical demonstrations that the rays from powerful lights falling upon dark walls give less light to a room than the rays from weak illuminants falling upon walls that have been painted in light colors with dust-resisting, washable paints. From the standpoint of economy, the cost of illumination can be greatly reduced by proper wall treatment.

Care Should Be Used in Color Selection

In schoolrooms and hospitals, and even in the home, very careful selection should be made of the colors that are used for the walls and ceilings, otherwise lessened efficiency and physical fatigue may result. Dark colors, such as brilliant reds, dark browns, dull grays and similar dark colors, may be the active cause of lessened efficiency, nervousness, and so forth,. whereas light tints of blue, green and yellow stimulate to activity and are conducive to happiness and amiability.

Large radiators in rooms of the home do not present a very attractive appearance unless they are properly decorated. For this purpose a paint should be used which will harmonize with the wall colorings, and one of the best paints obtainable for this purpose is the ordinary sanitary, flat wall paint that is used upon walls. It has been found that these paints dry with a flat surface having a high heat-transmission factor. For heating efficiency as well as for decoration, these paints are therefore to be recommended.

CHAPTER XXXIV

Paint and Varnish in the Great War

When the exploits and accomplishments of industry in the Great War are recorded on the pages of history a statement may appear to the effect that the conflict was prolonged by paint and that this material had a somewhat direct bearing upon the ultimate outcome. Such a statement will seem less startling to the person who will look back over the exciting incidents that filled the four years of strife, to the closing days of 1914, when the Huns, sweeping irresistibly through Belgium, were finally checked at the Marne and there arrested while they made preparations for the continuation of their drive. Another week would perhaps have marked the end of the war had it not been for the ability of the Allies at this crisis to provide more men, ammunition and equipment. It was at this point that paint played its role, since practically every instrument and material used in modern warfare has a useful life of something less than a week unless protected by paint or varnish. The rains and fog of September might over night have rendered useless much of the equipment of the resisting forces had it not been for the preserving effects of paint. To present this more clearly there is given below a description of a few of the materials of warfare, the successful use of which rests upon paint protection.

Aircraft.—Since the stability of aircraft depends upon the maintenance of wing and control surfaces that are light and of a rigid form, it is certain that the airplane could not have been used in warfare had it not been for the availability of airplane dope and protective coatings. These materials are applied to the linen or cotton cloth that is used for covering the immense wing surfaces (some planes requiring 400 square yards of cloth for this purpose). It is impossible to make this cloth sufficiently taut by stretching. Moreover, it is subject to rapid absorption of moisture and may become flabby upon short exposure. To overcome this condition, there is applied to the cloth-covered surface, four or five coats of airplane dope which consists of cellulose acetate or nitrate (the latter being a form of gun cotton) dissolved in rapidly evaporating solvents such as acetone or methyl and ethyl acetates. Some of these dopes are made plastic and fire resisting with such materials as triphenylphosphate. When they are applied, they rapidly dry, producing films that shrink the cloth to a drum-tight condition on the wings. Even when this is accomplished it is necessary to varnish or paint the treated cloth to further protect it from water and keep it in a permanently rigid form. The use of paint is preferable as its pigment content shuts out the actinic rays of light which would penetrate a transparent film and rapidly reduce the strength of the cloth. Suitable anti-actinic paints or enamels shut out this light to such an extent that even

FIG. 72

FIG. 73

after several months of service the cloth will show practically its original strength.

It should also be pointed out that the spruce strength members of airplanes (spars and beams), might warp out of shape and throw the surfaces out of alignment after a few days' exposure to moist conditions if they were left bare. Moreover, practically the entire body or fuselage of the airplane is constructed of extremely thin plywood (veneer) consisting usually of three laminations of 1/8-inch stock glued together. Without varnish or paint protection the life of such parts would be brief, especially when exposed to conditions such as obtain along the seacoast. The metal fittings, the stay wires and the control cables used on aircraft also require protection with paint and varnish in order to prevent rapid corrosion, and consequent diminution of strength, the lowering of which would add greatly to the risk of flying.

With all of the above and many other applications it is safe to state that the airplane requires more paint protection than any other modern instrument of warfare of the same size; as high as 300 gallons of dope, varnish and enamel being used on some of the more modern seaplanes having fairly large hull surfaces.

Shells.—Modern high explosive shells are filled with picric acid or with tri-nitro-toluol mixtures which have the property of acting upon the iron surface of the shell to form iron compounds (iron picrate for example) which are extremely unstable and subject to premature explosion. In order to prevent the formation of such products the interior surfaces of all shells are first coated with a varnish that will act as an insulating film between the iron and the explosive mixture. These are usually spirit varnishes (shellac, collodion or bitumen) and they dry by evaporation of their light solvent content. The better grades are made of linseed and tung oils and require baking after application to the shell in order to develop their full degree of toughness and elasticity. This process of baking is referred to as "stoving," and automatic spraying machinery for applying the varnishes and ovens for baking them are installed in many of the shell-filling plants. Due to the necessity of great speed in shell making, the use of baking varnishes has not been general, as the slow cooling down of the shells after baking would interfere with rapid production. It is also interesting to record the fact that all shell varnishes must be free of the usual lead drier; even a few hundredths of one per cent being considered dangerous because of the avidity of lead compounds to form the highly unstable lead picrates.

The exterior surfaces of all shells are painted. If the shells were left bare and stacked in the field for long periods of time, as is the custom, they would show rapid corrosion which might possibly interfere with their loading into the guns. Another important reason for painting is to afford means of identification of each type of shell. For this purpose a series of colors are used which are bright and readily recognizable. These colors at once differentiate the high explosive, shrapnel, illuminating, anti-aircraft, gas, and other shells. The requirements of shell paints are extremely rigid. They must hide the metal in one coat and dry hard in one hour to a flat, tough surface that will not flake or chip upon exposure. Since ordinary oil paints require 24 hours for drying it is evident that great skill is necessary to produce a good shell paint.

Cartridges.—There was produced in this country during the period of active hostilities over twenty million cartridges per day and every one of these required varnish protection. The interior of the neck of the cartridge shell must be coated with a varnish in order that a tight seal will be formed where the cupro-nickel bullet is fixed and thus prevent the admission of moisture to the powder contained within the shell. The primer cap on the end of each cartridge is also sealed with the same varnish in order to maintain the sensitive primer mixture in a dry and functionable condition. It is rather interesting to note that when unvarnished cartridges reach the temperature of a hot summer's day, the air contained within the shell expands and if the cartridge is then cooled off by placing it in water, a vacuum is immediately formed; water is then sucked in at the neck or around the primer, and the powder may be rendered inactive. With the modern varnished bullet such danger does not attend and it would be safe for the soldier campaigning in the Sahara Desert to place his cartridges in a tank of water over night (if water were available) without fear of rendering them defective. In fact, tests are now made at the cartridge factories requiring specimens of every run of varnished cartridges to be warmed up and placed in a tank of water for 24 hours with subsequent successful use in the ballistic tests. The varnish used on such bullets is of an extremely rapid drying nature and is applied by a machine which varnishes them and inserts the primers. Within a few minutes the cartridges are filled with powder and the bullet inserted by another machine. It can be readily seen that a varnish for this work must have extraordinary characteristics. It must become dry previous to the introduction of the powder in order to prevent the adherence of powder granules to the varnished surface and it must remain elastic to withstand the abrasion caused by the inserted bullet. It must also form a waterproof seal.

Ship Paints.—Ships of war are ships of steel but this metal is serviceable only as long as it is protected from corrosion. Paint protection is therefore a necessity to the successful operation of a battle fleet. To the uninitiated it might seem rather surprising to learn that the coating of one large ship may require over 100 tons of paint and that the paint is renewed practically every six months in order to maintain permanent protection and appearance. The steel, wood, and concrete vessels of the merchant marine, the giant transports, the submarine chasers, and every other type of craft used in warfare require the same type of protection. While much of the paint applied was for the purpose of camouflage, which was found successful in rendering craft optically elusive to the prowling submarine, the main object was for protection against the elements.

Camouflage and Color.—At no previous time in the world's history has color played so important a rôle as the present. While the use of paint in camouflage should really be considered as secondary to its use as a preservative, some very important applications were made which rendered it of great value. This art of protective coloration was developed practically over night and became one of the important considerations of the war, receiving the attention and application of some of the world's most talented marine artists. In fact, the camouflage squad of the Army and the Navy became as well recognized as a necessary complement as any other organization. The lives of thousands of wounded troops confined in hospitals were often saved through the skill of the camoufleur in concealing by clever coloration the roofs of buildings from the view of hostile aviators. The successful voyages of vessels and troopships with their much-needed cargoes may also be attributed in large measure to the modern developments in surface craft coloration. The hiding or making elusive by optical distortion, of guns, aircraft, and nearly every conceivable type of instrument of warfare depended to a great extent upon the use of camouflage paint. Highly important discoveries were made in the optics of paint pigments and many applications of scientific principles were called upon in working out special formulas, the history and importance of which it is probably early to disclose.

Mobile Equipment.—The auto truck, tractor and general mobile equipment used for transporting ammunition and men would have been ineffective unless protected with paint. It may be imagined how long the spokes of an artillery wheel would retain their form when subjected to hard field usage if left in an unvarnished or unpainted condition. It is not difficult to visualize the amount of warping and rotting of wood parts or corrosion of bare metal parts that would take place. The same applies to railroad equipment and it may be incidentally mentioned that the one hundred thousand odd freight cars and locomotives that were constructed in this country for use abroad were all painted according to standard practice before shipment or use.

General.—It is of importance to state that the American paint industry was one of the very first at the outbreak of the war to offer the free services of its leaders and experts to the various departments of the Government. The history of the unceasing efforts of the War Service Committee of the Paint Industry forms an interesting contribution to this subject. This committee worked out, under most intense pressure, new paints of many types that were immediately required for war use and which involved great skill in preparation.

In chapter XXXV are given a number of these specifications which were drawn up for the War Department.

CHAPTER XXXV

Standard Paint Specifications of the War Department

The General Specifications which follow are a part of all specifications for paints and varnish for the War Department. They apply to orders that are placed with manufacturers of army equipment where such manufacturers buy the paints and varnish for application to such equipment, and to orders that are placed direct by the War Department.

GENERAL SPECIFICATIONS

1. Unless otherwise specified, all paints shall be furnished in prepared form suitable for finish coat work and tinted to match the standard shades shown herein*

2. Unless otherwise requested by the Department there shall be submitted with bids two one-pint samples of the paint or varnish which it is proposed to furnish and which must conform to the specifications herein.

Delivered lots must conform to submitted samples.

3. When required by purchaser, the manufacturer shall furnish samples of raw materials used in these paints. When required invoice must be accompanied by an affidavit to the effect that the paint or varnish shipped has been made conformable to the specifications herein.

4. The paint or varnish furnished on these specifications will be submitted to chemical and physical tests, and if found deficient in either will be rejected.

They may be submitted to time exposure tests. In cases where the award of a contract cannot be delayed for the exposure tests, the award will be made on the basis of the other requirements, including the chemical and physical requirements referred to in the specifications. Any prod-

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*The standard color samples referred to are omitted in the following reprint of specifications.

uct, however, which does not satisfactorily withstand exposure tests will be omitted from future consideration.

5. All paint and varnish shall be delivered in packages conformable to the specifications and marked in accordance with regulations which will be furnished to successful bidders.

Unless otherwise specified, where tin containers are used they shall be such as are in ordinary use by paint and varnish manufacturers and shall be cased in boxes of the standard type now in use by manufacturers and conforming to the regulations of the Interstate Commerce Commission.

6. All percentages of materials given in the following specifications are percentages by weight, unless otherwise stated.

7. The Linseed Oil, Turpentine, Mineral Spirits, Drier and Varnish contained in all paints shall conform to the standards prescribed herein.

8. Paints must conform to the following physical requirements: They must not harden or cake in the package; they must be satisfactory in every respect, including color, consistency, fineness, working properties, spreading, hiding power, flowing, drying, finish and durability.

9. Special requirements peculiar to the paints in question are stated under individual specifications.

10. A variation of 2 per cent either way when definite amounts are specified will be allowed. When limits are set the numbers stated will be adhered to.

11. In paint composed of Zinc Oxide and Basic Carbonate or Basic Sulphate White Lead, Leaded Zinc may be used, provided the paint composition specified is maintained.

12. Paints and Varnishes will be purchased f. o. b. bidder's plant or warehouse, the location of which shall be stated.

13. All containers must be plainly marked with the actual net volume of the paint or varnish therein.

Where paint is purchased in paste form, the containers must be plainly marked with the actual net weight of the paint therein.

14. The inspection of Paints and Varnish may be made at the place of manufacture, or elsewhere, as the Inspection Department may direct.

The Inspector shall have full and free access to all parts of the manufacturer's plant and the manufacturer shall, at all times, afford him every reasonable facility for examination and inspection of all materials used or to be used in the manufacture of any product hereunder.

15. Color cards are attached to nearly all specifications except black and white. These cards show the color required, but the type of finish (flat or gloss) must comply with the particular requirements of the specifications.

RAW OR REFINED LINSEED OIL

U. S. Army Standard

W. D. 1

Specification.

It shall be absolutely pure, well-settled oil of best quality. It shall be perfectly clear at a temperature of 60° F. and shall not show a loss of over 0.2 per cent when heated to 100° C. It shall conform to the following requirements:

Specific gravity at 15.5°/15.5° C.	0.936	0.932
Or specific gravity at 25°/25° C.	0.931	0.927
Acid number	6	
Saponification number	195	189
Unsaponifiable matter, per cent	1.50	....
Refractive index at 25° C.	1.4805	1.4790
Iodine number (Hanus)	....	170

BOILED LINSEED OIL

U. S. Army Standard

W. D. 2

Specification.

It shall be absolutely pure, well-settled linseed oil boiled with oxides of manganese and lead. It shall conform to the following requirements:

	Maximum.	Minimum.
Specific gravity at 15.5°/15.5° C	0.945	0.937
Acid number	8	....
Saponification number	195	189
Unsaponifiable matter, per cent	1.50	....
Refractive index at 25° C	1.484	1.479
Iodine number (Hanus)	....	168
Ash, per cent	0.7	0.2
Manganese, per cent	....	0.03
Lead, per cent	....	0.1

TURPENTINE

U. S. Army Standard

W. D. 3

Specification.

This specification applies both to the turpentine that is distilled from pine oleoresins, and commonly known as gum turpentine or spirits turpentine, and to the turpentine commonly known as wood turpentine that is obtained from resinous wood, whether by extraction with volatile solvents, by steam, or by destructive distillation. The bidder should state whether gum spirits or wood turpentine is furnished.

The turpentine shall be clear and free from suspended matter and water. The color shall be water white. The specific gravity shall not be less than 0.860 or more than 0.875 at 15.5° C. The refractive index shall not be less than 1.468 or more than 1.478 at 15.5° C. The initial boiling point shall be not less than 150° C. nor more than 160° C. Ninety per cent of the turpentine shall distill below 170° C. The polymerization residue shall not exceed 2 per cent and its refractive index at 15.5° C. shall not be less than 1.500.

VOLATILE MINERAL SPIRITS

U. S. Army Standard

W. D. 4

Specification.

This shall be a hydrocarbon distillate, water white, neutral, clear and free from water. It shall have no darkening effect when mixed with basic carbonate white lead.

When 10 c. c. are put in a glass crystallizing dish 2½ in. in diameter, and placed on a steam bath for 2½ hours, the residue must not exceed .03 gms.

When tested according to the standard tests for Paint Thinners Other than Turpentine, prescribed by the American Society for Testing Materials, the flash point shall be not less than 29° C. (85° F.), in a closed tester, and in the distillation test, the first drop must issue from the condenser at a temperature not below 129° C. (265° F.), and 97 per cent must distill below 243° C. (470° F.).

DRIER

U. S. Army Standard

W. D. 5

Specification.

It shall be composed of lead, manganese, or cobalt compounds, or a mixture of any of these, combined with pure linseed oil and thinner. The thinner shall consist of approximately 75 per cent volatile mineral spirits and 25 per cent turpentine. It shall be free from adulterants, sediment, and suspended matter. Resins or "gums" other than rosin may be used at the option of the manufacturer (but the presence of rosin in any form shall be cause for rejection). The drier shall not flash below 35° C. (95° F.) in an open tester. When the drier is thoroughly mixed with linseed oil that will not break under 315° C. (600° F.) in the proportion of 5 volumes of drier to 95 volumes of oil, no curdling shall result within 2 hours after mixing, and the mixture when flowed on a clean glass plate held in a vertical position, shall dry in not more than 18 hours at a temperature between 15° and 35° C.

VARNISH*

U. S. Army Standard

W. D. 6

Specification.

The material shall be the best long oil varnish. It must

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*This specification does not apply to interior coating of projectiles.

be resistant to air, light and water. The manufacturer is given wide latitude in the selection of raw materials and processes of manufacture, so that he may produce a varnish of the highest quailty. Preference will be given to rapid drying varnishes.

The varnish shall be clear and transparent.

The color of the varnish shall be no darker than that of a standard color solution, made by dissolving six (6) grams of pure powdered potassium bichromate in 100 cc. of pure concentrated sulphuric acid (specific gravity 1.84). Gentle heat may be used if necessary to perfect the solution of the bichromate. The standard color solution and a sample of varnish shall be placed in clear thin-walled glass tubes of the same diameter. The color comparison shall be made by placing the tubes close together and looking through them. The tubes used for this test shall be ⅝ to 13/16 inch in diameter, and shall be filled with the liquids to a depth of at least one (1) inch.

When tested in an open tester, the varnish shall not flash below 35° C. (95° F.).

This test shall be made on a film of varnish flowed on one side of a panel of bright tin, which is 0.0125 to 0.0158 inch thick and about four (4) inches wide by six (6) inches long. The panel shall be thoroughly cleaned with benzol before applying the varnish. When the panel is placed in a vertical position and maintained at a temperature of 21° to 32° C. (70° to 90° F.), the varnish must set to touch at points within one (1) inch of the side and top edges of the film, in not more than five (5) hours and must dry hard in not more than twenty-four (24) hours, to a clear hard glossy film.

The panel shall then be allowed to dry for a further period of not less than five (5) days, and then brought to a temperature of from 70° to 75° F. The panel, with the varnished side on the outside, shall then be rapidly bent double over a rod ⅛ inch in diameter. The varnish film must neither crack nor flake at the point of bending.

A basswood panel shall be filled with a coat of drop black in oil, thinned with turpentinue and drier, and allowed to dry for at least ten (10) days. The filler must have suitable body to give proper brushing, flowing, and covering properties. A coat of varnish shall then be applied, allowed to dry for forty-eight (48) hours, and lightly sandpapered. A second coat of varnish shall then be applied and allowed to dry for seventy-two (72) hours. The panel shall then be inclined at an angle of forty-five (45) degrees, and a gentle stream of cold tap water allowed to flow for eighteen (18) hours down the middle of the panel. After wiping off, with a chamois skin, any deposits due to tap water, the varnish must show no appreciable whitening, dulling, or other defects. A small stream of boiling distilled water will then be allowed to flow down another portion of the panel for twenty (20) minutes. The water will be siphoned through a small glass tube directly from a container in which it is boiling, onto the surface of the panel, in such a manner that there will be no appreciable lowering of the temperature of the water before it touches the varnish film. The siphon delivery tube will be in a plane nearly paralled to the plane of the panel, so that the impact of the water will not tend to break the firm. The varnish shall show no appreciable whitening and no more than a very slight dulling, or other indications of marked deterioration, either when observed immediately after removing from the water, or after drying for two (2) hours.

The varnish shall be applied, in three (3) coats, to two (2) unfilled panels of maple wood. The panels must not be less than 5½ inches wide, by 18 inches long by ¾ inch thick. The first coat of varnish, after drying indoors for three (3) days shall be lightly sandpapered with No. 00 sandpaper before the application of the second coat. The second and third coats of varnish shall each be allowed to dry three (3) days indoors, but neither coat shall be sandpapered or rubbed. A protective coat of varnish (not for test purposes) shall be applied to the back and edges of the panels. The duplicate panels shall then be placed outdoors at an angle of 45 degrees to the vertical and facing the south.

The durability of the varnish on the test panel must be equivalent to that of an approved varnish exposed under the same conditions.

ASPHALTUM VARNISH

U. S. Army Standard

W. D. 7

Specification.

This liquid shall be made on a high-grade asphaltum base, compounded with linseed oil and drier.

It shall contain not less than 10 gallons of pure linseed oil to 100 gallons of the finished product.

It shall be reducible with Naptha and free from sediment and dirt.

It shall set to touch in 90 minutes and shall dry hard to a black, lustrous film in less than 15 hours. When dry and hard, it shall not rub up by friction under the fingers.

It shall not flash under 85° F. in a closed Tester.

BLACK ENAMEL

U. S. Army Standard

W. D. 8

Specification.

Pigment (not more than) 5%.
The balance shall be liquid.
The pigment portion shall be—
Carbon Black.*

The liquid portion shall be—
Varnish.

Special Requirements: The color shall be a deep black, and when flowed on to bright tin held in a vertical position, it shall dry rapidly with a high lustre, to a tough

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*A product of natural gas containing not more than 1.25% ash.

elastic film, within 12 hours, and cover solidly with one coat.

BLACK PAINT FOR GENERAL PURPOSES

U. S. Army Standard
W. D. 9

Specification.

Pigment	30%
Liquid	70%

The pigment portion shall consist of—

Carbon Black*	20%
Red Lead	5%

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*A product of natural gas containing not more than 1.25% ash.

The balance shall be shale black, aluminum silicate, magnesium silicate, or a mixture thereof.

The liquid portion shall consist of—

Pure Raw Linseed Oil (not less than)	80%

The balance shall be combined drier and thinner. The thinner shall consist of approximately equal parts of turpentine and volatile mineral spirits.

Special Requirements: The paint shall weigh not less than 9 pounds per gallon and shall dry within 18 hours to a full oil gloss.

It is intended for general use on metal or wood surfaces and shall be deep black in color.

BLUE PAINT

U. S. Army Standard
W. D. 10

Specification.

Pigment	50%
Liquid	50%

The pigment portion shall consist of—

Lithopone (containing not less than 26% Zinc Sulphide)	75%
Aluminum Silicate, Magnesium Silicate, or a mixture thereof, combined with the necessary amount of Pure Chinese Blue to produce the desired shade (a total of)	25%

The liquid portion shall consist of—

Pure Raw Linseed Oil (not less than) 65%

The balance to be combined drier, turpentine and volatile mineral spirits.

Special Requirement: The paint shall dry rapidly to a hard, dull surface.

BRIGHT RED OXIDE PAINT

U. S. Army Standard

W. D. 11

Specification.

Pigment 55%
Liquid 45%

The pigment portion shall consist of—

Not less than 30% Ferric Oxide (Fe_2O_3). It shall also contain not less than 5% red lead.

The balance to be silica or silicious earth pigments.

The liquid portion shall consist of—

Pure Raw Linseed Oil (not less than) 75%

The balance to be combined drier and volatile mineral spirits.

Special Requirements: This paint shall weigh not less than 13 pounds per gallon and shall dry within 18 hours to a full oil gloss.

BRONZE GREEN PAINT

U. S. Army Standard

W. D. 12

Specification.

Pigment 58%
Liquid 42%

The pigment portion shall consist of—

Chrome green which must carry not less than 20% chemically pure chrome green precipitated on an inert base which may consist of barium sulphate, aluminum silicate, magnesium silicate, silica, or a mixture thereof. To this green may be added the necessary pure tinting colors to produce the desired shade.

The liquid portion shall consist of—

Pure Raw Linseed Oil (not less than)	85%

The balance to be combined drier and thinner. The thinner shall be turpentine, or volatile mineral spirits, or a mixture thereof.

Special Requirements: The paint shall weigh not less than 13 pounds per gallon and shall dry within 18 hours to a full oil gloss.

BROWN ENAMEL PRIMER

U. S. Army Standard

W. D. 13

Specification.

Pigment	60%
Liquid	40%

The pigment portion shall consist of—

Not less than 35% ferric oxide (Fe_2O_3); the balance to be silica or silicious earth pigments.

The liquid portions shall consist of—

Varnish	70%
Combined Drier and Thinner	30%

The thinner shall consist of approximately equal parts of turpentine and volatile mineral spirits.

There shall be present not less than 30% of non-volatile in the liquid portion of the paint.

Special Requirements: This paint shall weigh not less than 13½ pounds per gallon and shall dry rapidly to a flat surface.

CAMOUFLAGE COLORS

U. S. Army Standard

Special Requirements: All of the following camouflage paints, Nos. 1, 2, 3 and 4, shall dry within 6 hours to a flat, non-reflecting surface, and shall be of proper consistency to apply with a stencil brush.

W. D. 14

No. 1, Yellow.
No. 2, Green.
No. 3, Cream.

Specification.

Pigment	70%
Liquid	30%

The pigment portion shall consist of—

White Lead (Basic Carbonate, Basic Sulphate, or a mixture thereof)	40%
Zinc Oxide	20%
Magnesium Silicate, combined with the necessary pure tinting colors to produce the desired shades (a total of)	40%

The liquid portion shall consist of treated oils or varnish, and volatiles. The liquid portion must contain not less than 42% of non-volatiles, the balance to be volatiles. Of the non-volatiles, not less than 80% must be pure linseed oil and pure Chinese wood oil (heat treated). The volatile portion shall consist of turpentine or volatile mineral spirits, or a mixture thereof.

These paints shall weigh not less than 16 pounds per gallon and shall conform to the special requirements above.

W. D. 15

No. 4, Black

Specification.

Pigment	56%
Liquid	44%

The pigment portion shall consist of—

White Lead (Basic Carbonate, Basic Sulphate, or a mixture thereof)	40%
Zinc Oxide	20%
Combined Magnesium Silicate and Carbon Black*: a total of	40%

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*A product of natural gas containing not more than 1.25% ash.

The liquid portion shall consist of treated oils or varnish and volatiles. The liquid portion must contain not less than 42% of non-volatiles, the balance to be volatiles. Of the non-volatiles, not less than 80% must be pure linseed oil and pure Chinese wood oil (heat treated). The volatile portion shall consist of turpentine or volatile mineral spirits, or a mixture thereof.

This paint must be deep black in color and weigh not less than 13 pounds per gallon.

COPPER PAINT

U. S. Army Standard
W. D. 16

Specification.

Pigment	44%
Liquid	56%

The pigment portion shall consist of finely ground natural iron oxide containing at least 40% Fe_2O_3. It shall also contain copper oxide in amount sufficient to insure not less than 12.5% of copper (Cu) in the completed paint.

The liquid portion shall consist of pine tar, drier, and suitable thinners.

On heating 5 grams of the paint in a flat-bottom dish 3 inches in diameter for 2 hours at a temperature of 105° to 110° C., the residue remaining shall be not less than 69% of the paint.

Special Requirements: This paint shall dry satisfactorily to a tough, salt-water resisting film. It shall be suitable for use on wood bottoms and have good antifouling and protective properties.

DARK RED OXIDE PAINT

U. S. Army Standard
W. D. 17

Specification.

Pigment	55%
Liquid	45%

The pigment portion shall consist of—

Ferric Oxide (Fe_2O_3) (not less than) 30%

The balance shall be silica or silicous earth pigments.

The liquid portion shall consist of—

Pure Raw Linseed Oil (not less than) 75%

The balance to be combined drier and volatile mineral spirits.

Special Requirements: The paint shall weigh not less than 12½ pounds per gallon and shall dry within 18 hours to a full oil gloss.

DECK AND FLOOR PAINT

U. S. Army Standard

W. D. 18

Specification.

Pigment	50%
Liquid	50%

The pigment portion shall consist of—

Lithopone (containing not less than 26% Zinc Sulphide)	56%
White Lead (Basic Carbonate, Basic Sulphate, or a mixture thereof), and Zinc Oxide	24%
Aluminum Silicate, Magnesium Silicate, or a mixture thereof, combined with the necessary pure Tinting Colors to produce the desired shade (a total of)	20%

The liquid portion shall consist of—

Varnish, linseed oil, drier and thinner, and shall contain not less than 30% non-volatile matter.

Special Requirements: The paint must weigh not less than 11¼ pounds per gallon and must dry within 6 hours.

EXTERIOR CANTONMENT PAINT (GRAY)

U. S. Army Standard

W. D. 19

Specification.

This paint also to be furnished in white when required.

Pigment	64%
Liquid	36%

The pigment portion shall consist of—

White Lead (Basic Carbonate, Basic Sulphate, or a mixture thereof)	42%
Zinc Oxide	33%
Aluminum Silicate, Magnesium Silicate, or a mixture thereof, combined with the necessary pure tinting colors to produce the desired shade (a total of)	25%

The liquid portion shall consist of—

Pure Raw Linseed Oil	20%
Menhaden Oil,* Soya Bean Oil,** or a mixture thereof	40%

The balance to be combined drier and volatile mineral spirits.

Special Requirements: This paint shall weigh not less than 15 pounds per gallon, and shall dry within 12 hours, and leave a surface suitable to recoat in 36 hours.

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*The Menhaden Oil used in this paint shall be pure, refined, light in color, and not of objectionable odor. It shall have the following chemical constants:

Specific gravity	.92-.93
Iodine number, not less than	166
Saponification number, not less than	186
Acid number, not more than	8

**The Soya Bean Oil used in this paint shall be pure, light in color, and shall have the following chemical constants:

Specific gravity	.92-.93
Iodine number, not less than	130
Saponification number, not less than	180
Acid number, not more than	4

EXTERIOR CANTONMENT PAINT (GREEN)

U. S. Army Standard

W. D. 20

Specification.

Pigment	58%
Liquid	42%

The pigment portion shall consist of—

Chrome green which must carry not less than 20 % chemically pure chrome green precipitated on an inert base which may consist of barium sulphate, aluminum silicate, magnesium silicate, silica, or a mixture thereof. To this green may be added the necessary pure tinting colors to produce the desired shade.

The liquid portion shall consist of—

Pure Raw Linseed Oil	20%
Menhaden Oil, Soya Bean Oil, or a mixture thereof	40%

The balance to be combined drier and volatile mineral spirits.

Special Requirements: This paint shall dry within 12 hours and leave a surface suitable to repeat in 36 hours.

The Menhaden and Soya Bean Oils used herein shall conform to the requirements of those oils as given for Formula W. D. 19.

FIRE RETARDANT PAINT

U. S. Army Standard

W. D. 21

In any Tint desired.

For exterior use on lumber construction wherever fire resistance is desired.

White Paint and Tinted Paints Made on a White Base.

Pigment	60%
Liquid	40%

The pigment portion shall consist of—

Basic Sulphate White Lead	28%
Zinc Oxide	22%
Magnesium Silicate, combined with the necessary pure tinting colors to produce the desired shade (a total of)	50%

The liquid portion shall consist of—

Pure Raw Linseed Oil (not less than)	55%

The balance to consist of equal parts of drier, turpentine and volatile mineral spirits.

Special Requirement: This paint shall weigh not less than 13 pounds per gallon. When applied to new lumber constructions it shall penetrate and dry rapidly to a durable film. It shall successfully withstand the standard firebrand test when applied to two-coat work.

INTERIOR CANTONMENT PAINT

Flat Finish Interior Paint.

U. S. Army Standard

W. D. 22

In White or any Light Tint desired.

Specification.

White and Light Tints Made on a White Base.

Pigment	70%
Liquid	30%

The pigment portion shall consist of—

Lithopone (containing not less than 26% zinc sulphide) (not less than)	80%
Zinc Oxide (not less than)	10%

The balance to be White Mineral Pigments and Tinting Colors.

The liquid portion shall consist of—

Treated oils and volatiles, or varnishes which produce a satisfactory washable flat finish.

The liquid portion must contain:

Non-Volatiles (not less than)	20%
Volatiles (not more than)	80%

The volatiles shall be turpentine, or volatile mineral spirits, or a mixture thereof.

Of the total volatiles, not more than 2½% may be water.

Special Requirements: The physical characteristics particularly required are fineness, flowing and flatting. The paint shall weigh not less than 14½ pounds per gallon.

GRAPHITE PAINT

U. S. Army Standard

W. D. 23

Specification.

Pigment	40%
Liquid	60%

The pigment portion shall consist of—

Either the natural or artificial form of graphite. It shall contain at least 60% graphite carbon and the balance shall consist of iron oxide, silica, or silicious earth pigments.

The liquid portion shall consist of—

Pure raw linseed oil (not less than) 80%, the balance to be combined drier and thinner. The thinner shall consist of turpentine, volatile mineral spirits, or a mixture thereof.

Special Requirements: This paint shall weigh not less than 10 pounds per gallon and shall dry within 18 hours to a full oil gloss.

GRAY PAINT

U. S. Army Standard

W. D. 24

Specification.

Pigment	64%
Liquid	36%

The pigment portion shall consist of—

White Lead (Basic Carbonate, Basic Sulphate, or a mixture thereof)	45%
Zinc Oxide	35%
Aluminum Silicate, Magnesium Silicate, or a mixture thereof, combined with the necessary amount of Carbon Black* to produce the desired shade (a total of)	20%

The liquid portion shall consist of—

Pure raw linseed oil (not less than) 85%, the balance to be combined drier and thinner. The thinner shall consist of approximately equal parts turpentine and volatile mineral spirits.

Special Requirements: The paint is intended for general use on metal or wood surfaces. It shall weigh not less than 15 pounds per gallon and shall dry within 18 hours to a full oil gloss.

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*A product of natural gas containing not more than 1.25% ash.

GRAY PAINT—SPECIAL QUICK DRYING

U. S. Army Standard

W. D. 25

Specification.

Pigment	52%
Liquid	48%

The pigment portion shall consist of—

White Lead (Basic Carbonate, Basic Sulphate, or a mixture thereof) (not less than)	28%
Zinc Oxide (not less than)	22%

The balance to be magnesium silicate, combined with the necessary amount of carbon black* to produce the desired shade.

The liquid portion shall consist of treated oils or varnish, and volatiles. The liquid portion must contain not less than 42% of non-volatiles, the balance to be volatiles. Of the non-volatiles, not less than 80% must be pure linseed oil and pure Chinese wood oil (heat treated). The volatile portion shall consist of turpentine or volatile mineral spirits, or a mixture thereof.

Special Requirements: The paint shall weigh not less than 11½ pounds per gallon and shall dry within 6 hours to a dull finish.

LAMP BLACK IN OIL

(Paste)

U. S. Army Standard

W. D. 26

Specification.

Pigment	25%
Liquid	75%

The pigment portion shall consist of—

Lampblack (not less than)	85%
Barium Sulphate (not more than)	15%

The combined pigment used in making the paste, when ashed shall show not more than 16% of total ash. The

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*A product of natural gas containing not more than 1.25% ash.

lampblack used shall be the perfectly calcined product of oils only.

The liquid shall be—

Pure raw linseed oil.

Special Requirement: The pigment shall be finely ground to a medium stiff paste which shall break up readily on thinning.

One pound sample shall be submitted with bid.

LEADED ZINC

U. S. Army Standard

W. D. 27

Specification

The dry pigment shall be pure leaded zinc, free from all adulterants.

It shall contain not more than 40% basic lead sulphate, the balance to be zinc oxide.

Moisture shall not exceed	0.5 %
Water soluble salts shall not exceed	1.5 %
Sulphur dioxide shall not exceed	.75%

The pigment, when required to be ground to a paste in oil, shall be of the same quality as the dry pigment, and shall be ground in at least 12% of pure bleached or refined linseed oil.

OLIVE DRAB PAINT

U. S. Army Standard

W. D. 28

Specification.

Pigment	64%
Liquid	36%

The pigment portion shall consist of—

White Lead (Basic Carbonate, Basic Sulphate, or a mixture thereof) (not less than)	42%
Zinc Oxide (not less than)	33%

The balance to consist of aluminum silicate, magnesium silicate, or a mixture thereof, combined with the necessary pure tinting colors to produce the desired shade.

The liquid portion shall consist of—

Pure raw linseed oil (not less than) 68%, the balance to be combined drier and thinner. The thinner shall consist of turpentine, or volatile mineral spirits, or a mixture thereof.

Special Requirements: The paint shall weigh not less than 15 pounds per gallon, and shall dry within 18 hours to a full oil gloss.

OLIVE DRAB PAINT—SPECIAL QUICK DRYING

U. S. Army Standard
W. D. 29

Color identical with Olive Drab Paint, U. S. Army Standard.
W. D. 28.

Specification.

Pigment	60%
Liquid	40%

The pigment portion shall consist of—

White Lead (Basic Carbonate, Basic Sulphate, or a mixture thereof) (not less than)	42%
Zinc Oxide (not less than)	33%

The balance to consist of aluminum silicate, magnesium silicate, or a mixture thereof, combined with the necessary pure tinting colors to produce the desired shade.

The liquid portion shall consist of treated oils or varnish, and volatiles. The liquid portion must contain not less than 42% of non-volatiles, the balance to be volatiles. Of the non-volatiles, not less than 80% must be pure linseed oil and pure Chinese wood oil (heat treated). The volatile portion shall consist of turpentine or volatile mineral spirits, or a mixture thereof.

Special Requirements: This paint shall weigh not less than 13½ pounds per gallon and shall dry within 6 hours to a dull finish.

OLIVE GREEN PAINT

U. S. Army Standard

W. D. 30

Specification.

Pigment	64%
Liquid	36%

The pigment portion shall consist of—

White Lead (Basic Carbonate, Basic Sulphate, or a mixture thereof) (not less than)	42%
Zinc Oxide (not less than)	33%

The balance to consist of aluminum silicate, magnesium silicate, or a mixture thereof, combined with the necessary pure tinting colors to produce the desired shade.

The liquid portion shall consist of—

Pure raw linseed oil (not less than) 68%, the balance to be combined drier and thinner. The thinner shall consist of turpentine or volatile mineral spirits, or a mixture thereof.

Special Requirements: The paint shall weigh not less than 15 pounds per gallon and shall dry within 10 hours to a dull finish.

ORANGE SHELLAC VARNISH

W. D 31

Specification

This material shall consist of 4½ pounds of Shellac cut in one gallon of clear neutral denatured alcohol.*

The shellac used shall be a high grade orange shellac which, when treated with hot 95% alcohol, will not show a residue of insoluble matter exceeding 1.75%.

The shellac shall be free from rosin and other adulterants.

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*The alcohol used shall be No. 1 Internal Revenue Dept. Standard consisting of 100 gallons of grain alcohol and 5 gallons approved wood alcohol.

PAINT AND VARNISH REMOVER

U. S. Army Standard

W. D. 32

Specification.

This shall be composed of suitable ingredients so combined as to be of the highest efficiency.

It shall be free from mineral matter, alkalies or acids.

It shall be light in color and satisfactory in working qualities, and efficient as a paint remover.

These qualities will be determined by practical tests and comparisons.

PROJECTILE PAINTS

U. S. Army Standard

For Exterior Use.

General Requirements of All Projectile Paints.

All projectile plants shall cover bright steel with one coat. They shall dry sufficiently hard to handle within one hour after application. They shall dry very hard within three hours after application, with a flat or semi-flat finish, to a tough, elastic film which will not flake, scale or chip from the metal surface. They shall weigh between 13 and 15½ lbs. per gallon, with the exception of black, which shall weigh between 7½ and 8½ lbs. per gallon.

W. D. 33

White Projectile Paint

Specification.

Pigment	60%
Liquid	40%

The pigment portion shall consist of—

Lithopone (containing not less than 26% Zinc Sulphide)	90%
Zinc Oxide	10%

The liquid portion shall consist of treated oils or varnish, and volatiles. The liquid portion must contain not less than 33% of non-volatiles, the balance to be volatiles. Of the non-volatiles, not less than 80% must be pure linseed oil and pure Chinese wood oil (heat treated). The volatile portion shall consist of turpentine or volatile mineral spirits, or a mixture thereof.

(See General Requirements above.)

Light Olive Green Projectile Paint, Blue Gray Projectile Paint, Warm Gray Projectile Paint.

These colors shall be identical in composition with the above white, except that they shall be tinted with pure tinting colors to the desired shades.

U. S. Army Standard
W. D. 34
Blue Projectile Paint

Specification

Pigment	50%
Liquid	50%

The pigment portion shall consist of—

Lithopone (containing not less than 26% Zinc Sulphide)	75%
Neutral Blanc Fixe, combined with the necessary amount of Pure Chinese Blue to produce the desired shade (a total of)	25%

The composition of the liquid portion shall be identical with that used in the above white. W. D. 33.

(See General Requirements.)

W. D. 35
Green Projectile Paint

Specification

Pigment	64%
Liquid	36%

The pigment portion shall consist of—

Lithopone (containing not less than 26% Zinc Sulphide)	50%
Chrome Green	50%

The chrome green must carry not less than 20% chemically pure chrome green, precipitated on an inert base. The base may consist of barium sulphate, aluminum silicate, magnesium silicate, silica or a mixture thereof.

The composition of the liquid portion shall be identical with that used in the above white.

(See General Requirements.)

W. D. 36

Vermilion Projectile Paint

Specification.

Pigment	60%
Liquid	40%

The pigment portion shall consist of—

Non-fading, organic para red, precipitated upon a white base, which shall consist of aluminum silicate, magnesium silicate, barium sulphate or other suitable inert materials.

It shall contain pure organic coloring matter (nt less than)...... 10%

The composition of the liquid portion shall be identical with that used in the above white. W. D. 33.

(See General Requirements.)

U. S. Army Standard

W. D. 37

Yellow Projectile Paint

Specification.

Pigment	55%
Liquid	45%

The pigment portion shall consist of—

A mixture of yellow ochre, pure chrome yellow and iron oxide, in the proper proportions to produce the desired shade.

The composition of the liquid portion shall be identical with that used in the above white. W. D. 33.

(See General Requirements.)

W. D. 38

Black Projectile Paint

Specification

Pigment	8%
Liquid	92%

The pigment portion shall consist of—

Pure carbon black (a product of natural gas, containing not more than 1.25% ash).

The composition of the liquid portion shall be identical with that used in the above white. W. D. 33.

(See General Requirements.)

PUTTY

U. S. Army Standard
W. D. 39

Specification

85 % Whiting.
15% Pure Raw Linseed Oil.

The Whiting shall be of the best quality, practically free from grit or alkali, and shall not contain more than 1% of water.

One pound sample shall be submitted with bid.

REINFORCED RED LEAD PAINT

U. S. Army Standard
W. D. 40

Specification

Pigment	64%
Liquid	36%

The pigment portion shall consist of—

Red lead (not less than) 60%, the balance to be silicious matter, such as aluminum silicate, magnesium silicate, silica or a mixture thereof.

The red lead used shall contain not less than 85% Pb_3O_4, the balance to be PbO.

The liquid portion shall consist of—

Pure raw linseed oil (not less than) 90%, the balance to be combined drier and thinner. The thinner shall be turpentine.

Special Requirements: The paint must weigh not less than 16 pounds per gallon. It is intended to be used for a priming coat on steel and when applied to a smooth iron surface, it shall dry in 12 hours without running, streaking or sagging.

SEAL BROWN PAINT

U. S. Army Standard

W. D. 41

Specification

Pigment	55%
Liquid	45%

The pigment portion shall consist of—

Natural iron oxides, tinted to match the standard color. It shall contain at least 20% ferric oxide.

The liquid portion shall consist of—

Pure raw linseed oil (not less than) 75%, the balance to be combined drier and volatile mineral spirits.

Special Requirements: The paint must weigh not less than 12¼ pounds per gallon and must dry within 18 hours to a full oil gloss.

SLATE PAINT

U. S. Army Standard

W. D. 42

Specification

Pigment	64%
Liquid	36%

The pigment portion shall consist of—

White Lead (Basic Carbonate, Basic Sulphate, or a mixture thereof) (not less than)	42%
Zinc Oxide (not less than)	33%

The balance to consist of aluminum silicate, magnesium silicate, or a mixture thereof, combined with the necessary pure tinting colors to produce the desired shade.

The liquid portion shall consist of—

Pure raw linseed oil (not less than) 68%, the balance to be combined drier and thinner. The thinner shall consist of turpentine, or volatile mineral spirits, or a mixture thereof.

Special Requirements: This paint shall weigh not less than 15 pounds per gallon and shall dry within 10 hours to a dull finish.

STENCIL BLACK

U. S. Army Standard
W. D. 43

Specification

Pigment	50%
Liquid	50%

The pigment portion shall consist of—

Drop Black	50%
Calcium Carbonate	50%

The liquid portion shall consist of—

Varnish	70%
Combined Drier and Thinner	30%

The thinner shall consist of turpentine, or volatile mineral spirits, or a mixture thereof.

Special Requirement: The paint shall dry rapidly to a flat black surface that hides well.

STENCIL WHITE

U. S. Army Standard
W. D. 44

Specification

Pigment	55%
Liquid	45%

The pigment portion shall consist of—

Basic Sulphate White Lead and Zinc Oxide (a total of not less than)	25%
Lithopone (containing not less than 26% Zinc Sulphide) (not less than)	25%

The balance to be calcium carbonate.

The liquid portion shall consist of—

Varnish	70%
Combined Drier and Thinner	30%

The thinner shall be turpentine, or volatile mineral spirits, or a mixture thereof.

Special requirement: The paint shall dry rapidly to a flat surface.

VERMILION PAINT

(Para Red)
Liquid Form
U. S. Army Standard
W. D. 45

Specification

Pigment	50%
Liquid	50%

The pigment portion shall consist of—

Non-fading organic para red, precipitated upon a white base, and shall contain at least 10% of pure organic coloring matter. The balance shall consist of approximately 10% of magnesium silicate and 80% of barium sulphate.

The liquid portion shall consist of—

Pure raw linseed oil (not less than) 85%, the balance to be combined drier and thinner. The thinner shall consist of turpentine, or volatile mineral spirits, or a mixture thereof.

Special Requirements: This paint shall weigh not less than 12 pounds per gallon and shall dry within 10 hours to a full oil gloss.

VERMILION

(Para Red)
Paste Form
U. S. Army Standard
W. D. 46

Color identical with Vermilion in liquid form, U. S. Army Standard.

Specification

Pigment	80%
Liquid	20%

The pigment portion shall consist of—

Non-fading organic para red precipitated upon a white base, and shall contain at least 10% of pure organic coloring matter. The balance shall consist of approximately 10% of magnesium silicate and 80% of barium sulphate.

The liquid portion shall be pure raw linseed oil.

Special Requirements: This paint shall be ground to a stiff paste which shall break up readily on thinning. It shall be resistant to excessive bleeding.

One pound sample shall be submitted with bid.

WHITE ENAMEL
U. S. Army Standard
W. D. 47

Specification

The pigment shall consist wholly of—

Pure French process zinc oxide.

The liquid shall consist of—

High grade varnish resins or treated oils, with such volatiles as are necessary. It must not contain more than 1% of moisture in any form.

Special Requirements: This enamel shall be clear white and shall dry with a high lustre to a tough, elastic film, within 12 hours. When brushed on a panel of clean bright tin and allowed to dry for 48 hours, it shall withstand bending over a rod (1/8 inch in diameter) without showing cracking or flaking at the point of bending.

WHITE LEAD—BASIC CARBONATE
U. S. Army Standard
W. D. 48

Specification

The dry pigment shall be of the best quality, amorphous in structure, and of great opacity.

It shall not contain more than 0.5% moisture.

Unless otherwise specified, this basic carbonate white lead shall be delivered in paste form, finely ground in pure, clear raw or refined linseed oil in the proportion of:

92 pounds Pigment.
8 pounds Oil.

WHITE LEAD—BASIC SULPHATE

U. S. Army Standard
W. D. 49

Specification

The dry pigment shall be of the best quality, amorphous in structure, and of great opacity.

It shall contain:

Not more than	0.5 %	Moisture
Not more than	8.5 %	Zinc Oxide (ZnO)
Not more than	.075%	Sulphur Dioxide (SO_2), and
Not less than	12. %	Lead Oxide (PbO).

Unless otherwise specified, this basic sulphate-white lead shall be delivered in paste form, finely ground in pure, clear raw or refined linseed oil in the proportion of:

90 pounds	Pigment.
10 pounds	Oil.

WHITE PAINT AND TINTED PAINTS MADE ON A WHITE BASE

U. S. Army Standard
W. D. 50

Specification

Pigment	64%
Liquid	36%

The pigment portion shall consist of—

White Lead (Basic Carbonate, Basic Sulphate, or a mixture thereof) (not less than)	45%
Zinc Oxide (not less than)	35%

The balance to consist of aluminum silicate, magnesium silicate, or a mixture thereof, combined with the necessary pure tinting colors to produce the desired shade.

The liquid portion shall consist of—

Pure raw linseed oil (not less than) 85%, the balance to be combined drier and thinner. The thinner shall consist of approximately equal parts of turpentine and volatile mineral spirits.

Special Requirements: These paints shall weigh not less than 15 pounds per gallon and shall dry within 18 hours to a full oil gloss.

WHITE TARGET PAINT

U. S. Army Standard
W. D. 51

Specification

Pigment	55%
Liquid	45%

The pigment portion shall consist of—

Zinc Oxide (not less than)	50%

(The zinc oxide used in this paint may be leaded zinc containing not over 5% of lead sulphate.)

The balance to consist of magnesium silicate.

The liquid portion shall consist of—

Pure Refined Linseed Oil	50%
Varnish	25%
Volatile Mineral Spirits	20%
Turpentine	5%
The non-volatile portion of the liquid shall be not less than	60%

Special Requirements: The paint shall weigh not less than 12 pounds per gallon and shall dry rapidly to a semi-flat surface.

WOOD FILLER

(Liquid Form)
U. S. Army Standard
W. D. 52

Specification

Pigment	17%
Liquid	83%

The pigment portion shall consist of—

Finely divided silica that will pass through a 200 mesh screen.

The liquid portion shall consist of—

Varnish	77%

The balance to be turpentine or volatile mineral spirits, or a mixture thereof.

Special Requirements: When applied to wood, it shall dry in not more than 5 hours. It shall be of the proper consistency for either brushing or dipping.

ZINC WHITE PAINT

U. S. Army Standard

W. D. 53

Specification

Pigment	55%
Liquid	45%

The pigment portion shall be pure French process zinc oxide.

The liquid portion shall consist of—

Pure refined linseed oil (not less than) 70%, the balance to be combined drier and thinner. The thinner shall be turpentine, or volatile mineral spirits, or a mixture thereof.

Special Requirements: The paint shall weigh not less than 13 pounds per gallon and shall dry rapidly to a semi-flat surface.

CHAPTER XXXVI

Interdepartmental Specifications

The United States Interdepartmental Committee on Paint Specification Standardization was appointed at the suggestion of the Secretary of Commerce. The Committee consists of representatives of the War, Navy, Agriculture, Interior, Post Office, Treasury, and Commerce Departments, the Railroad Administration, the Panama Canal, and the Educational Bureau of the Paint Manufacturers' Association of the United States. The present writer is the member representing the Educational Bureau above referred to. The Committee has met in Washington for a period of over a year, and has drawn up a series of specifications for linseed oil, turpentine, mineral spirits, basic carbonate-white lead, basic sulphate-white lead, zinc oxide, leaded zinc, red lead, ochre, black paint, red paint, green paint, spar varnish, drier, composite paint oil, and similar materials.

The published specifications contain the standards together with methods of sampling, and complete methods of analysis. It would obviously be impossible to reprint all of these specifications in this volume. It is suggested, therefore, to those who may be interested in obtaining a complete set that they write to the Superintendent of Documents, Government Printing Office, Washington, D. C. Each specification may be obtained for 5 cents per copy. Three of the most important specifications are presented herewith, namely, linseed oil, turpentine, and white paint. These three were selected because of the general importance of the analytical data required for the determination of the quality of such widely used paint products.

RECOMMENDED SPECIFICATION FOR LINSEED OIL—RAW, REFINED, AND BOILED.

1. GENERAL

Linseed oil, raw, refined, or boiled, as specified in contract, shall be pure and shall conform to the following requirements:

RAW LINSEED OIL

	Maximum	Minimum
Loss on heating at 105 to 110° C (per cent)	0.2	....
Foots by volume (per cent)	2.0	....
Specific gravity 15.5/15.5° C	.936	0.932
Acid number	6.0	....
Saponification number	195.0	189.0
Unsaponsifiable matter (per cent)	1.5	....
Iodine number (Hanus)[a]	....	170.0
Color	Not darker than a freshly prepared solution of 1.0 g potassium bichromate in 100 cc pure strong (1.84 specific gravity) sulphuric acid.	

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[a] When raw linseed oil from North American seed is specified by the purchaser, the iodine number must be not less than 180 and the oil shall conform to all the other requirements as above.

REFINED LINSEED OIL

Contract shall state whether acid refined or alkali refined is desired.

	Maximum	Minimum
Loss on heating at 105 to 110° C (per cent)	0.2	
Foots by volume (per cent)	.2	
Specific gravity at 15.5/15.5° C	.936	0.932
Acid number (acid refined oil)	9.0	3.0
Acid number (alkali refined oil)	3.0	
Saponification number	195.0	189.0
Unsaponifiable matter (per cent)	1.5	
Iodine number (Hanus) *a*		170.0
Color	Not darker than a freshly prepared solution of 0.1 g potassium bichromate in 100 cc pure strong (1.84 specific gravity) sulphuric acid.	

a When refined linseed oil from North American seed is specified by the purchaser, the iodine number must be not less than 180 and the oil shall conform to all the other requirements as above.

BOILED LINSEED OIL

Boiled oil shall be pure, well-settled linseed oil that has been boiled with oxides of manganese and lead. It shall conform to the following requirements:

	Maximum	Minimum
Loss on heating at 105 to 110° C (per cent)	0.2	
Specific gravity at 15.5/15.5° C	.945	0.937
Acid number	8.0	
Saponification number	195.0	189.0
Unsaponifiable matter (per cent)	1.50	
Iodine number (Hanus) *a*		168.0
Ash (per cent)	.7	.2
Manganese (per cent)		.03
Lead (per cent)		.1
Time of drying on glass (hours)	20.0	

a When boiled linseed oil from North American seed is specified by the purchaser, the iodine number must be not less than 178 and the oil shall conform to all the other requirements as above.

2. SAMPLING

The method of sampling given under (*a*) below should be used whenever it is feasible to apply it. To meet conditions when (*a*) is not applicable, method (*b*), (*c*), or (*d*) is to be used, according to the special conditions that obtain.

(*a*) *During Loading of Tank Cars or Filling of Containers for Shipment at the Factory.*—The purchaser's inspector shall draw a sample at the discharge pipe where it enters the receiving vessel or vessels. The total sample shall be not less than 5 gallons and shall be a composite of small samples of not more than 1 pint each taken at regular intervals during the entire period of loading or filling.

The sample thus obtained shall be thoroughly mixed, and from this composite sample three portions of not less than 1 quart each shall be placed in clean dry glass bottles or tin cans which must be filled with the sample and securely stoppered with new clean corks or well-fitting metal covers or caps. These shall be sealed and labeled distinctly by the inspector, and one delivered to the buyer, one to the seller, and the third held for check in case of dispute.

(*b*) *From Loaded Tank Cars or Other Large Vessels.*—The total sample shall be not less than 5 gallons and shall be a composite of numerous small samples of not more than 1 pint each taken from the top, bottom, and intermediate points by means of a glass or metal container with removable stopper or top. This device attached to a suitable pole is lowered to the various desired depths when the stopper or top is removed and the container allowed to fill. The sample thus obtained is handled as in (*a*).

(*c*) *Barrels and Drums.*—Not less than 5 per cent of the packages in any shipment or delivery of barrels and drums shall be sampled. The packages shall be shaken, rolled, and stirred to thoroughly mix the contents. The samples from the individual containers shall be taken through the bung hole or holes not less than 1¼ inch in diameter bored in the head or side for the purpose. The apparatus for drawing the sample shall consist of a glass tube about 1 inch in diameter and somewhat longer than the length or diameter of the oil container, a conical stopper that will fit the glass tube and is not more than ½ inch long fastened to a stiff metal rod not more than ¼ inch in diameter and not less than 4 inches longer than the glass tube. The stopper is lowered by the rod until it rests on the bottom of the cask, the tube slipped down slowly over the rod, and finally pressed on the stopper. By holding tube and rod the column of oil can then be removed. This process is repeated until the required amount of sample is obtained, which must be not less than 2 gallons. This is mixed and handled as in (*a*).

(*d*) *Small Containers, Cans, etc., of 10 Gallons or Less.*—Small containers, cans, etc., of 10 gallons or less should be sampled while filling by method (*a*) whenever possible. When method (*a*) is not applicable, it is mutually agreed that: In all cases the total sample taken shall not be less than 3 quarts. This shall be obtained by taking at least one package from each lot of not more than 300 packages. The sample thus taken shall be thoroughly mixed and subdivided as in (*a*).

3. LABORATORY EXAMINATION

Samples shall, in general, be tested by the following methods, but the purchaser reserves the right to apply any additional tests such as specific tests for foreign oils, rosin, etc., or use any available information to ascertain whether the material meets the specification. The laboratory sample shall be thoroughly mixed by shaking, stirring, or pouring from one vessel to another and the samples for the individual tests taken from this thoroughly mixed sample.

(*a*) *Loss on Heating at 105 to 110° C.*—Place 10 g. of the oil in an accurately weighed 200 cc. Erlenmeyer flask; weigh. Heat in an oven at a temperature between 105 and 110° C. for 30 minutes; cool and weigh. Calculate the percentage loss. This determination shall be made in a current of dry carbon dioxide gas.

(*b*) *Foots.*—With all materials at a temperature between 20 and 27° C. mix, by shaking in a stoppered flask for exactly one minute, 25 cc. of the well-shaken sample of oil, 25 cc. of acetone (see 4(*a*)) and 10 cc. of the acid calcium chloride solution (see 4(*b*)). Transfer the mixture to a burette where settling can take place for 24 hours. The temperature during this period should be between 20 and 27° C.

The volume of the stratum lying between the clear calcium chloride solution and the clear acetone and oil mixture is read in tenths of a cubic centimeter or a fraction thereof. This reading multiplied by four expresses the amount of foots present as percentage by volume of the oil taken.

(*c*) *Specific Gravity.*—Use a pyknometer accurately standardized and having a capacity of at least 25 cc. or any other equally accurate method, making the test at 15.5° C., water being unity at 15.5° C.

(*d*) *Acid Number.*—Weigh from 5 to 10 g. of the oil. Transfer to a 350 cc. Erlenmeyer flask. Add 50 cc. of neutral 95 per cent ethyl alcohol. Put a condenser loop inside the neck of the flask. Heat on a steam bath for 30 minutes. Cool and add phenolphthalein indicator. Titrate to a faint permanent pink color with the standard sodium hydroxide solution. Calculate the acid number (milligrams KOH per gram of oil).

(*e*) *Saponification Number.*—Weigh about 2 g. of the oil in a 350 cc. Erlenmeyer flask. Add 25 cc. alcoholic sodium hydroxide solution. Put a condenser loop inside the neck of the flask and heat on the steam bath for one hour. Cool, add phenolphthalein as indicator, and titrate with half normal sulphuric acid. Run two blanks with the alcoholic sodium hydroxide solution. (See 4(*h*).) These should check within 0.1 cc. N/2 H_2SO_4. From the difference between the number of cubic centimeters of

N/2 H_2SO_4 required for the blank and for the determination, calculate the saponification number (milligrams KOH required for 1 g. of oil).

(*f*) *Unsaponifiable Matter.*—Weigh 8 to 10 g. of the oil. Transfer to a 250 cc., long-neck flask. Add 5 cc. of strong solution of sodium hydroxide (equal weights of NaOH and H_2O), and 50 cc. 95 per cent ethyl alcohol. Put a condenser loop inside the neck of the flask and boil for two hours. Occasionally agitate the flask to break up the liquid but do not project the liquid onto the sides of the flask. At the end of two hours remove the condenser and allow the liquid to boil down to about 25 cc.

Transfer to a 500 cc. glass-stoppered separatory funnel, rinsing with water. Dilute with water to 250 cc., add 100 cc. redistilled ether. Stopper and shake for one minute. Let stand until the two layers separate sharp and clear. Draw all but one or two drops of the aqueous layer into a second 500 cc. separatory funnel and repeat the process using 60 cc. of ether. After thorough separation draw off the aqueous solution into a 400 cc. beaker, then the ether solution into the first separatory funnel, rinsing down with a little water. Return the aqueous solution to the second separatory funnel and shake out again with 60 cc. of ether in a similar manner, finally drawing the aqueous solution into the beaker and rinsing the ether into the first separatory funnel.

Shake the combined ether solution with the accumulated water rinsings and let the layers separate sharp and clear. Draw off the water and add it to the main aqueous solution. Shake the ether solution with two portions of water (about 25 cc. each). Add these to the main water solution.

Swirl the separatory funnel so as to bring the last drops of water down to the stopcock, and draw off until the ether solution just fills the bore of the stopcock. Wipe out the stem of the separatory funnel with a bit of cotton on a wire. Draw the ether solution (portionwise if necessary) into a 250 cc. flask and distill off. While still hot, drain the flask into a small weighed beaker, rinsing with a little ether. Evaporate this ether, cool and weigh. (*The unsaponifiable oil from adulterated drying oils is volatile and will evaporate on long heating. Therefore heat the beaker on a warm plate, occasionally blowing out with a current of dry air. Discontinue heating as soon as the odor of ether is gone.*)

(*g*) *Iodine Number.*—Place a small quantity of the sample in a small weighing burette or beaker. Weigh accurately. Transfer by dropping about 0.15 g. (0.10 to 0.20 g.) to a 500 cc. bottle having a well-ground glass stopper, or an Erlenmeyer flask having a specially flanged neck for the iodine test. Reweigh the burette or beaker and determine the amount of sample used. Add 10 cc. of chloroform. Whirl the bottle to dissolve the sample. Add 10 cc. of chloroform to two empty bottles like that used for the sample. Add to each bottle 25 cc. of the Hanus solution (see 4 (*g*)) and let stand with occasional shaking for one-half hour. Add 10 cc. of the 15 per cent potassium iodide solution and 100 cc. of water, and titrate with standard sodium thiosulphate using starch as indicator. The titrations on the two blank tests should agree within 0.1 cc. From the difference between the average of the blank titration and the titration on the samples and the iodine value of the thiosulphate solution, calculate the iodine number of the samples tested. (Iodine number is centigrams of iodine to 1 g. of sample.)

(*h*) *Ash.*—Tare a porcelain crucible or dish. Add 10 to 25 cc. of oil, carefully weighing the amount added. Place on a stone slab on the floor of a hood. Ignite by playing the flame of a burner on the surface of the oil and allow to burn quietly until most of the oil is burned off; then transfer to a muffle or over a flame and continue heating at a very low temperature (not over a dull red) until all carbonaceous matter is consumed. Cool, weigh, and calculate the percentage of ash. Moisten the ash with a few drops of water and test with litmus paper. Record whether neutral or alkaline. Wash any ash adhering to the test paper back into the crucible. Dissolve the ash in dilute nitric acid to which a little hydrogen peroxide has been added. After solution is complete make up the volume to about 50 cc. with nitric acid and water so that the final volume will contain about 1 volume of concentrated nitric acid and 3 volumes of water. Boil to remove excess of hydrogen peroxide. Determine manganese by the bismuthate method as described in Treadwell-Hall, Analytical Chemistry, third edition, volume 2, page 617.

Ash another portion of the oil and dissolve the ash as above in nitric acid and hydogen peroxide. Transfer to a 250 cc. beaker and dilute to about 200 cc. This volume of solution should contain 15 to 20 cc. of concentrated nitric acid. Electrolyze this solution using platinum electrodes (the anode being previously weighed) with a current density of about 0.5 ampere and 2 to 2.5 volts. It is best to pass the current overnight (about 15 hours). On removing the anode, it is carefully washed in clear water, dried in a steam oven, transferred to an oven where it is heated to 180° C., cooled and weighed. The increase in weight of the anode multiplied by 0.86 gives the weight of lead in the sample. Calculate to percentage. If desired, the lead may be determined by the sulphate or any other accurate method in place of the electrolytic method given above.

(i) *Time of Drying on Glass.*—Flow the oil over a perfectly clean glass plate and allow to drain in a vertical position in a well-ventilated room at a temperature between 15 and 39°. After about 2 hours the film is tested at intervals with the finger at points not less than 2½ cm. from the edges. The film will be considered dry when it adheres no longer to the finger and does not rub up appreciably when the finger is rubbed lightly across the surface. With boiled linseed oil this usually occurs in from 5 to 18 hours.

(j) *Color.*—Prepare a fresh solution of pure potassium bichromate in pure strong (1.84 specific gravity) colorless sulphuric acid. For raw oil, this solution should be in the proportion of 1.0 g. potassium bichromate to 100 cc. (184.0 g.) sulphuric acid. For refined oil, the solution should be in the proportion of 0.1 g. potassium bichromate to 100 cc. sulphuric acid. Place the oil and colored solution in separate thin-walled, clear glass tubes of the same diameter (1 to 2 cm.) to a depth of not less than 2.5 cm. and compare the depths of color by looking transversely through the columns of liquid by transmitted light.

4. REAGENTS FOR TESTING

The following reagents will be required:

(*a*) *Acetone* that will pass the specification of the United States Pharmacopoeia.

(*b*) *Acid Calcium Chloride Solution.*—Saturate with calcium chloride a mixture of 90 parts water and 10 parts concentrated hydrochloric acid (specific gravity 1.2).

(*c*) *Standard 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 1000 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.

(*d*) *Starch Solution.*—Stir up 2 to 3 g. of potato starch or 5 g. soluble starch with 100 cc. of 1 per cent salicylic acid solution, add 300 to 400 cc. boiling water, and boil the mixture until the starch is practically dissolved. Dilute to 1 liter.

(*e*) *Standard Iodine Solution.*—Dissolve 13 g. of resublimed iodine and 18 g. of pure potassium iodide (free from iodates) in 50 cc. of distilled water, and dilute to 1000 cc. Determine its exact value by titrating with the standard sodium thiosulphate solution.

(*f*) *Potassium Iodide Solution.*—Dissolve 150 g. of potassium iodide free from iodate in distilled water and dilute to 1000 cc.

(*g*) *Hanus Solution.*—Dissolve 13.2 g. of iodine in 1000 cc. of glacial acetic acid (99.5 per cent) that will not reduce chromic acid. Add enough bromine to double the halogen content, determined by titration (3 cc. of bromine is about the proper amount). The iodine may be dissolved by the aid of heat, but the solution should be cold when the bromine is added.

(*h*) *Standard Sodium Hydroxide Solution.*—Prepare a stock concentrated solution of sodium hydroxide by dissolving sodium hydroxide in water in the proportion of 200 g. NaOH to 200 cc. water. Allow this solution to cool and settle in a stoppered bottle for several days. Decant the clear liquid from the precipitate of sodium carbonate into another clean bottle. Add clear barium hydroxide solution until no further precipitate forms. Again allow to settle until clear. Draw off about 175 cc. and dilute to 10 liters with freshly boiled distilled water. Preserve in a stock bottle provided with a large guard tube filled with soda lime. Determine the exact strength by titrating against pure benzoic acid (C_6H_5COOH) using phenolpthalein as indicator. (See Bureau of Standards Scientific Paper 183.) This solution will be approximately one-fourth normal, but do not attempt to adjust it to any exact value. Determine its exact strength and make proper corrections in using it.

(*i*) *Alcoholic Sodium Hydroxide Solution.*—Dissolve pure sodium hydroxide in 95 per cent ethyl alcohol in the proportion of about 22 g. per 1000 cc. Let stand in a stoppered bottle. Decant the clear liquid into another bottle, and keep well stoppered. This solution should be colorless or only slightly yellow when used; it will keep colorless longer if the alcohol is previously treated with NaOH (about 80 g. to 1000 cc.), kept at about 50° C. for 15 days, and then distilled.

(*j*) *Half Normal Sulphuric Acid Solution.*—Add about 15 cc. sulphuric acid (1.84 specific gravity) to distilled water, cool and dilute to 1000 cc. Determine the exact strength by titrating against freshly standardized sodium hydroxide or by any other accurate method. Either adjust to exactly half normal strength or leave as originally made, applying appropriate correction.

5. BASIS OF PURCHASE

Material is to be purchased by weight or volume as specified in the contract. When purchased by weight, the price shall be quoted per pound or per hundred pounds. When purchased by volume, a gallon of oil shall mean 231 cubic inches, at 15.5° C.

RECOMMENDED SPECIFICATIONS FOR TURPENTINE

(Gum Spirits and Wood Turpentine)

I. GENERAL.

These specifications apply both to the turpentine which is distilled from pine oleoresins, commonly known as gum spirits or spirits of turpentine, and to turpentine commonly known as wood turpentine, which is obtained from resinous wood, whether by steam or by destructive distillation. When ordering under these specifications, the purchaser shall specify whether (*a*) gum spirits or (*b*) wood turpentine is desired. When wood turpentine is specified, it may be stated whether steam or destructively distilled wood turpentine shall be furnished.

Turpentine shall be pure and conform to the following requirements:

APPEARANCE.—Shall be clear and free from suspended matter and water.

COLOR.—Shall be "standard" or better.

ODOR.—Shall be characteristic of the variety of turpentine specified and, if desired, shall conform to the odor of the sample agreed upon.

	Maximum	Minimum
Specific gravity, 15.5/15.5° C	0.875	0.862
Refractive index at 20° C	1.478	1.468
Residue after polymerization with 38*N* H_2SO_4:		
Gum spirits—		
Volume (per cent)	2.0	
Refractive index at 20° C		1.500
Wood turpentine—		
Volume (per cent)	2.5	
Refractive index at 20° C		1.495
Initial boiling point at 760 mm pressure	160° C	150° C
Distilling below 170° C at 760 mm pressure (per cent)		90

2. DETECTION AND REMOVAL OF SEPARATED WATER.

Draw a portion by means of a glass or metal container with a removable stopper or top, or with a "thief," from the lowest part of the container, or by opening the bottom valve of the perfectly level tank car. If water is found to be present, draw it all out, record the quantity, and deduct it from the total volume of liquid delivered.

3. SAMPLING.

The method of sampling given under (*a*) should be used whenever feasible. When method (*a*) is not applicable, method (*b*), (*c*), or (*d*) is to be used according to the special conditions that obtain.

(*a*) *While Loading Tank Car or While Filling Containers for Shipment.*—Samples shall be drawn by the purchaser's inspector at the discharge pipe where it enters the receiving vessel or vessels. The composite sample shall be not less than five gallons and shall consist of small portions of not more than one quart each taken at regular intervals during the entire period of loading or filling.

The composite sample thus obtained shall be thoroughly mixed and from it three samples of not less than one quart each shall be placed in clean, dry, glass bottles or tin cans, which must be nearly filled with the sample and securely stoppered with new, clean corks or well-fitting covers or caps. These shall be sealed and distinctly labeled by the inspector; one shall be delivered to the buyer, one to the seller, and the third held for check in case of dispute.

(*b*) *From Loaded Tank Car or Other Large Vessel.*—The composite sample taken shall be not less than five gallons and shall consist of numerous small samples of not more than one quart each taken from the top, bottom, and intermediate points by means of a metal or glass container with removable stopper or top. This device, attached to a suitable pole, is lowered to the various desired depths, when the stopper or top is removed and the container allowed to fill. The sample thus obtained is handled as in (*a*).

(*c*) *Barrels and Drums.*—Barrels and drums shall be sampled after gauging contents. Five per cent of the packages in any shipment or delivery shall be represented in the sample. Thoroughly mix the contents of each barrel to be sampled by stirring with a clean rod and withdraw a portion from about the center by means of a "thief" or other sampling device. The composite sample thus obtained shall be not less than three quarts, shall consist of equal portions of not less than one-half pint from each package sampled, and shall be handled as in (*a*). Should the inspector suspect adulteration, he shall draw the samples from the suspected packages.

(*d*) *Small Containers, Cans, etc., of 10 Gallons or Less.*—These should be sampled, while filling, by method (*a*) whenever possible; but in case this is impossible the composite sample taken shall be not less than three quarts. This shall be drawn from at least five packages (from all when fewer), and in no case from less than 2 per cent of the packages. The composite sample thus taken shall be thoroughly mixed and subdivided as in (*a*).

4. 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 specifications:

(*a*) *Appearance.*—Examine to determine compliance with the specifications.

(*b*) *Color.*—Fill a 200 mm. perfectly flat-bottomed colorimeter tube, graduated in millimeters, to a depth of from 40 to 50 mm. with the turpentine to be examined. Place the tube in a colorimeter and place on or under it a No. 2 yellow Lovibond glass. Over or under a second graduated tube in the colorimeter, place a No. 1 yellow Lovibond glass and run in the same turpentine until the color matches as nearly as possible the color in the first tube. Read the difference in depth of the turpentine in the two tubes. If this difference is 50 mm. or more, the turpentine is "standard" or better.

(*c*) *Odor.*—Determine by comparison with several samples of known purity, which have been kept in the dark in completely filled, well-stoppered bottles and are free from separated water.

(*d*) *Specific Gravity.*—Determine at 15.5/15.5° C., in a pycnometer accurately standardized and having a capacity of at least 25 cc., or by any other equally accurate method.

(*e*) *Refractive Index.*—Determine refractive index at 20° C. with an accurate instrument. When the refractive index is determined at any other temperature, the readings obtained shall be corrected to 20° C. by adding to or by subtracting from the actual reading 0.00045 for each degree centigrade that the temperature at which the determination was made is, respectively, above or below 20° C.

(*f*) *Distillation.*—*Apparatus.*—*Condenser.*—The type of apparatus (see Fig. 74) adopted by the American Society for Testing Materials for the distillation of paint thinners other than turpentine, substituting for the thermometer there described* an immersed thermometer such as is described below, is preferred. In case the A. S. T. M. distillation apparatus is not available, use an ordinary straight glass-tube condenser, about 22 inches long, with 16 inches in contact with the cooling water. The end of the condenser tube should be fitted with an adapter or should be bent down to a nearly vertical position, and the tip should be cut off or ground down at an acute angle. The tip should extend a short distance into the receiving cylinder.

Flask.—Comparable results can be obtained only by using flasks of the same dimensions. The distilling flask used shall be the standard Engler flask, as used for petroleum distillation, having the following dimensions: Diameter of bulb, 6.5 cm.; cylindrical neck, 15 cm. long, 1.6 cm. internal diameter; side or vapor tube, 10 cm. long, 0.6 cm. external diameter, attached to neck at an angle of 75°, so that when the flask contains its charge of 100 cc. of oil the surface of the liquid shall be 9 cm. below the bottom of the junction of the side tube and neck.

Support for Flask.—Support the flask on a plate of asbestos 20 cm. in diameter, having an opening 4 cm. in diameter in its center, and heat with an open flame. Surround the flask and burner with a shield to prevent fluctuation in the temperature of the neck of the flask. Or, support the flask in a metal cup, 15 to 20 cm. in diameter, containing high-boiling mineral oil or glycerin and fitted with a concave cover having in the center a circular opening 5½ to 6 cm. in diameter. In all cases take the necessary precautions to prevent fluctuation in temperature in the neck of the flask.

Thermometer.—The thermometer used for turpentine distillation shall conform to the following specifications:

It shall be graduated from 145° to at least 200° C. in

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*A. S. T. M. Standards, p. 607; 1918.

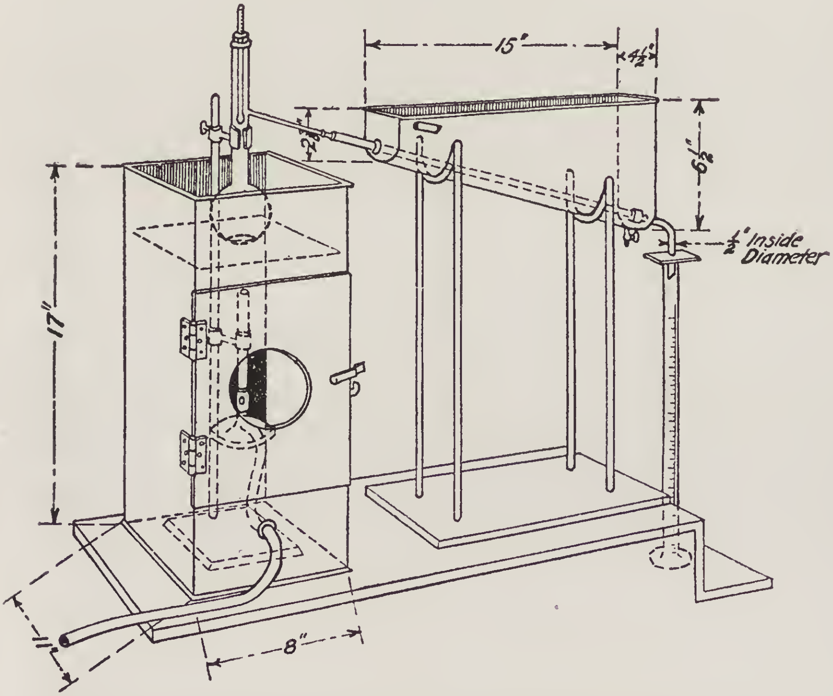
0.2° intervals. Thermometers graduated above 200° C. may be used, provided they also comply with the following requirements: Length, bottom of thermometer to 175° mark, not more than 8 nor less than 6.5 cm. Length, top of bulb to 145° mark, not less than 1.5 cm. Length, 145 to 175° mark, not more than 6 cm.

The thermometer shall be made of suitable thermometric glass and thoroughly annealed, so that the scale errors will not increase after continued heating.

The thermometer shall be filled above the mercury with an inert gas, with sufficient pressure above the mercury column to prevent breaking of the column. It shall have a reservoir at the top, so that the pressure will not become excessive at the highest temperature.

Fig. 74

DISTILLATION APPARATUS.



Every fifth graduation shall be longer than the intermediate ones, and the marks shall be numbered at each interval of 5°. The graduation marks shall be clear-cut and fine and the numbering clear-cut and distinct.

The error at any point on the scale shall not exceed ± 0.5° C. when tested for total immersion of the mercury column.

Receiving Cylinder.—Collect the distillate in an accurately graduated 50 or 100 cc. cylinder. The so-called normal or precision cylinder of 50 cc. capacity, having an internal diameter of 1.5 cm. and graduated in 0.2 cc., is preferred. If a cylinder with larger inside diameter is used, a pasteboard cover should be placed over the top and surround the condenser tube.

Operation.—Place 100 cc. of the turpentine and several small pieces of pumice (or glass) in the distilling flask, fit the thermometer so that the top of the mercury bulb is level with the bottom of the side tube, and the 175° C. (347° F.) mark is below the cork. Place the flask in position on the asbestos board or oil bath and connect with the condenser. Apply the heat cautiously at first, and, when distillation begins, regulate the heat so that the turpentine distills at the rate of not less than 4 nor more than 5 cc. per minute (approximately two drops per second). The initial boiling point is the thermometer reading at the instant when the first drop falls from the end of the condenser. Discontinue distillation when the temperature reaches 170.0° C. (338°F.), or an equivalent thereof, depending on the atmospheric pressure, as outlined below; let the condenser drain and read the percentage distilled.

The percentage distilled below successive selected temperatures and the temperature at which each successive 10 cc. distills may also be determined, if desired, making the necessary correction of the temperature for variations in atmospheric pressure.

Correction for Variation in Atmospheric Pressure.—Since distillation results are comparable only when obtained under exactly the same pressure conditions, turpentine shall be distilled at that pressure which, at room temperature, is equivalent to a pressure of 760 mm. of mercury at 0° C. Whenever the atmospheric pressure after correcting to 0° C. is other than 760 mm., a correction must be made. Since alteration of the pressure in the distilling system requires rather complicated apparatus, it is simpler to alter the temperature observation points to correspond to the prevailing pressure.

To determine what the atmospheric pressure at the prevailing room temperature, or at the temperature of the barometer, would be at 0° C., read the barometer and thermometer alongside when about to begin distillation. Under the column nearest the observed pressure reading, and on the line nearest the observed temperature of the barometer will be found the correction which must be subtracted from the observed pressure reading to obtain the equivalent, or true, reading at 0° C.

The distilling temperature of turpentine is affected plus (+) or minus (—) 0.057° C. for each millimeter variation of the barometer above or below the normal 760 mm. at 0° C*. If the barometer reading, after correcting to 0° C., is below 760 mm., the turpentine will distill at a slightly lower temperature than under normal pressure. Therefore, the temperature recorded at the beginning of distillation (and any others observed during the course of the distillation) must be corrected to get its equivalent at normal pressure. The final temperature observation point (170° C. of the specifications) must be altered accordingly to get its equivalent at the pressure (corrected to —° C.) at which distillation is made.

For example, if the barometer reading, after correcting to 0° C., is 750 mm., the correction of the observed initial distilling temperature will be 0.057 × 10 mm. ≐

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*Landolt-Bornstein Physikalisch-Chemische Tabellen, Ed. 4, Table 127, p. 435.

0.6° C. approximately. If the reading of the thermometer when the turpentine begins to distill is 155.6° C., the corrected initial distilling temperature will be 155.6° + 0.6° = 156.2° C. Furthermore, the temperature observation point at end of distillation (170.0° C. at 760 mm.) must be altered to the same extent. Since the turpentine is distilling 0.6° C. below what it would at normal pressure, distillation must be discontinued at 0.6° C. below the specified limit of 170.0° C. to determine the percentage distilling below 170.0° C.

If the barometer reading corrected to 0° C. is above 760 mm., subtract the temperature correction from the observed thermometer reading to determine the initial distilling point, and continue distillation to 170.0° C. plus the correction to determine the percentage distilling below 170.0° C.

(*g*) *Polymerization.*—Place 20 cc. of 38 *N* (equivalent to 100.92 per cent H_2SO_4) sulphuric acid in a graduated, narrow-necked Babcock flask, stopper, and place in ice water to cool. Add slowly, from a pipette, 5 cc. of the turpentine to be examined. Gradually mix the contents, keeping warm, but being very careful that the temperature does not rise above 60° C. When the mixture no longer warms up on shaking, agitate thoroughly and place the flask in a water bath and heat at 60 to 65° C. for not less than 10 minutes, keeping the contents of the flask thoroughly mixed by vigorous shaking for one-half minute each time, six times during the period. Do not stopper the flask after the turpentine has been added, as it may explode. Cool to room temperature, fill the flask with concentrated sulphuric acid until the unpolymerized oil rises into the graduated neck and centrifuge from four to five minutes at not less than 1200 r. p. m., or for 15 minutes at 900 r. p. m., or allow to stand, lightly stoppered, for 12 hours. Calculate the percentage, note the consistency and color, and determine the refractive index (at 20° C.) of the unpolymerized residue. The consistency should be viscous and the color straw or darker.

Reagent for Testing.—In a weighed glass-stoppered bottle (the regular 2½-liter acid bottle is of a convenient size) mix ordinary concentrated sulphuric acid (sp. gr. 1.84) with fuming sulphuric acid. If the fuming acid used contains 50 per cent excess SO_3, the ratio of one part, by weight, of the former to three-fourths of a part, by weight, of the latter will give a mixture slightly stronger than the required strength. To determine the exact strength of this mixture in terms of H_2SO_4, weigh ex-

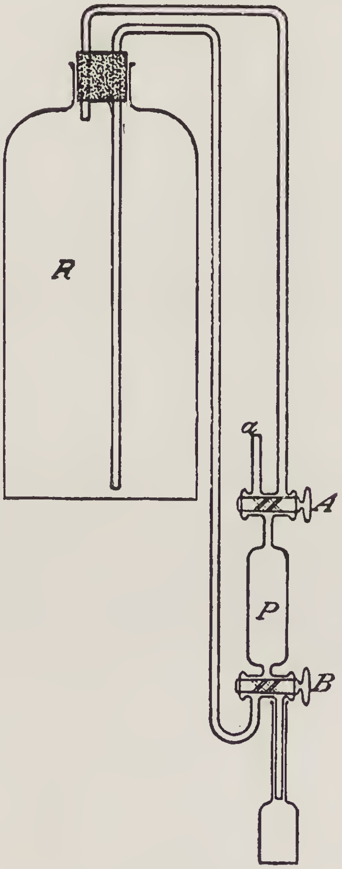

Fig. 75
Acid Bottle and Pipette

actly, in a weighing pipette of about 10 cc. capacity, approximately 20 g. of the acid. Allow it to flow down the sides of the neck into a 1000-cc. volumetric flask containing about 200 cc. of distilled water. When the pipette has drained, wash all traces of the acid remaining in the pipette into the flask, taking precautions to prevent loss of SO_3, and make up to the mark. Titrate 20-cc. portions, drawn from a burette, against half normal alkali. Calculate the concentration in terms of the percentage of H_2SO_4 in the sample taken.

In the same way determine the percentage of H_2SO_4 in the stock of ordinary concentrated acid (sp. gr. 1.84). From these data calculate the quantity of the latter which must be added to the quantity of mixed acid in the weighed bottle to bring it to a concentration, in terms of H_2SO_4, of 100.92 per cent.

After adjusting the concentration by the addition of the ordinary sulphuric acid, thoroughly shake the bottle of mixed acid and again determine its concentration. The allowable variation is $\pm$ 0.05 per cent H_2SO_4. Finally as a check run a polymerization test on gum turpentine known to be pure. The residue should fall below 2 per cent.

Special precautions must be taken to prevent dilution of this acid by the absorption of atmospheric moisture. The arrangement shown in Fig. 75 is most suitable for storing and delivering measured quantities of this reagent.

With the three-way stopcocks *A* and *B* in the position shown, acid is siphoned into the pipettte *P*, the displaced air passing into *R*. To empty the pipette, *A* and *B* are turned to the position shown by the broken lines, air passing in at *a*. The acid adhering to the walls of the pipette dries this air so that when it passes into *R* on again filling the pipette there is no accumulation of moisture in the acid remaining in the reservoir. If such arrangement is not to be had, the acid should be kept in well-fitting glass-stoppered bottles of not more than one-half liter capacity.

RECOMMENDED SPECIFICATIONS FOR WHITE PAINT AND TINTED PAINTS MADE ON A WHITE BASE—SEMIPASTE AND READY MIXED[1]

1. GENERAL

White paint and tinted paints made on a white base may be ordered either in the form of semipaste pigment ground in linseed oil, or of ready-mixed paint.

The semipaste shall be purchased by net weight, the ready-mixed paint either by weight or volume (231 cubic inches to the gallon).

(*a*) *Pigment.*—The pigment shall be composed of:

	Maximum	Minimum
	Per cent	Per cent
White lead (basic carbonate, basic sulphate, or a mixture thereof)	70	45
Zinc oxide (ZnO)	55	30
Silica, magnesium silicate, aluminum silicate, barium sulphate, pure tinting colors, or any mixture thereof	15	0

In no case shall the sum of the basic lead carbonate, basic lead sulphate, and zinc oxide be less than 85 per cent. The lead and zinc pigments may be introduced in the form of any mixture preferred of basic carbonate white lead, basic sulphate white lead, zinc oxide, or leaded zinc, provided the above requirements as to composition are met.

(*b*) *Liquid.*—The liquid in semipaste paint shall be entirely pure raw or refined linseed oil; in ready-mixed paint

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[1] It is believed that this specification admits practically all high-grade prepared paints generally available in the United States, and which are therefore obtainable without requiring manufacturers to make up special lots. On large contracts for which paint will be specially made, the purchaser may require the bidder to submit the formula of the paint he proposes to furnish as conforming to the specifications.

it shall contain not less than 90 per cent pure raw linseed oil, the balance to be combined drier and thinner. The thinner shall be turpentine, volatile mineral spirits, or a mixture thereof.

(*c*) *Semipaste.*—Semipaste shall be made by thoroughly grinding the pigment with pure raw or refined linseed oil.

The semipaste as received and three months thereafter shall be not caked in the container and shall break up readily in linseed oil to form a smooth paint of brushing consistency. It shall mix readily with linseed oil, turpentine, or volatile mineral spirits, or any combination of these substances, in all proportions without curdling. The color and hiding power when specified shall be equal to that of a sample mutually agreed upon by buyer and seller. The weight per gallon shall be not less than 19.0 pounds. The paste shall consist of:

	Maximum	Minimum
	Per cent	Per cent
Pigment	77	73
Linseed oil	27	23
Moisture and other volatile matter	0.7	
Coarse particles and "skins" (total residue retained on No. 200 screen based on pigment)[1]	0.5	

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[1] The No. 200 screen is the same as the screen formerly known as 200-mesh.

(*d*) *Ready-Mixed Paint.*—Ready-mixed paints shall be well ground, shall not settle badly or cake in the container, shall be readily broken up with a paddle to a smooth, uniform paint of good brushing consistency, and shall dry within 18 hours to a full oil gloss, without streaking, running or sagging. The color and hiding power when specified shall be equal to those of a sample mutually agreed upon by buyer and seller. The weight per gallon shall be not less than 15¾ pounds. The paint shall consist of:

	Maximum	Minimum
	Per cent	Per cent
Pigment	66	62
Liquid (containing at least 90 per cent linseed oil)	38	34
Water	0.5	
Coarse particles and "skins" (total residue retained on No. 200 screen based on pigment)	0.5	

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, and when this is for any reason not done, the inspector shall determine by thoroughly 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 of the thoroughly mixed paint, place it in a clean, dry metal or glass container, which must be filled with the sample, closed with a tight cover, sealed, marked, and sent to the laboratory for test with the inspector's report on caking in container.

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.

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.

3. LABORATORY EXAMINATION—SEMIPASTE

(*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 paste must be no more difficult to break up than a normal good grade of semipaste paint. The semipaste 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 thoroughly mixed semipaste shall be placed in a clean container and the 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.*—From the weight of a known volume of the paste calculate the specific gravity, which multiplied by 8.34 gives the weight in pounds per gallon. Any suitable container of known volume may be used for the purpose, but a short cylinder of heavy glass with rounded bottom about 75 mm. high and having a capacity of from 125 to 175 cc. (a glass cap to keep dust from reagent bottle stopper) is a convenient apparatus for the purpose. The capacity of this vessel is determined to within 1 cc. The paste is packed into it until completely full, the top leveled off smooth with a spatula, and weighed to ± 0.5 g. Subtract the weight of the empty container and divide the remainder by the number of cubic centimeters representing the capacity of the container. The quotient is the specific gravity, which can be thus determined within ± 2 in the second decimal place.

(*d*) *Mixing with Linseed Oil.*—One hundred grams of the paste shall be placed in a cup, 18 cc. linseed oil added slowly with careful stirring and mixing with a spatula or paddle. The resulting mixture must be smooth and of good brushing consistency.

(*e*) *Moisture and Other Volatile Matter.*—Weigh accurately from 3 to 5 g. of the paste into a tared flat-bottomed dish about 5 cm. in diameter, spreading the paste over the bottom. Heat at 105 to 110° C. for one hour, cool and weigh. Calculate loss in weight as percentage of moisture and volatile matter.

(*f*) *Percentage of Pigment.*—Weigh accurately about 15 g. of the paste into a weighed centrifuge tube. Add 20 to 30 cc. of "extraction mixture" (see reagents), mix thoroughly with a 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.

(*g*) *Preparation of Fatty Acids.*—To about 25 g. of the paste in a porcelain casserole, add 15 cc. of aqueous sodium hydroxide (see reagents) and 75 cc. of ethyl alcohol, mix and heat uncovered on a steam bath until saponification is complete (about one hour). Add 100 cc. of water, boil, add an excess of sulphuric acid of specific gravity 1.2 (8 to 10 cc. will usually suffice), boil, stir, and transfer to a separatory funnel to which some water has been previously added. Draw off as much as possible of the acid aqueous layer and lead sulphate precipitate, wash once with water, then add 50 cc. of water and 50 cc. of ether. Shake very gently with a whirling motion to dissolve the fatty acids in the ether, but not so violently as to form an emulsion. Draw off the aqueous layer and wash the ether layer with one 15 cc. portion of water and then with 5 cc. portions of water until free from sulphuric acid. Then draw off the water layer completely. Transfer the ether solution to a dry flask and add 25 to 50 g. anhydrous sodium sulphate. Stopper the flask and let stand with occasional shaking at a temperature below 25° C until the water is completely removed from the ether solution, which will be shown by the solution becoming perfectly clear above the solid sodium sulphate. Decant this clear solution, if necessary, through a dry filter paper, into a dry 100 cc. Erlenmeyer flask. Pass a rapid current of dry air (pass through a $CaCl_2$ tower) into the mouth of the Erlenmeyer flask and heat to a temperature below 75° C. on a dry hot plate until the ether is entirely driven off.

The fatty acids prepared as above should be kept in a stoppered flask and examined at once.

(*h*) *Test for Mineral Oil and Other Unsaponifiable Matter.*—Place 10 drops of the fatty acid (*g*) in a 50 cc. test tube, add 5 cc. of alcoholic soda (see reagents), boil vigorously for five minutes, add 40 cc. of water, and mix; a clear solution indicates that not more than traces of unsaponifiable matter are present. If the solution is not clear, the oil is not pure linseed oil.

(*i*) *Iodine Number of Fatty Acids.*—Place a small quantity of the fatty acids (*g*) in a small weighing burette or beaker. Weigh accurately. Transfer by dropping about 0.15 g. (0.10 to 0.20 g.) into a 500 cc. bottle having a well-ground glass stopper, or an Erlenmeyer flask having a specially flanged neck for the iodine test. Reweigh the burette or beaker and determine amount of to dissolve the sample. Add 10 cc. of chloroform to two

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Note.—It is important to follow all of the details since ether generally contains alcohol and after washing with water always contains water. It is very difficult to remove water and alcohol by evaporation from fatty acids, but the washing of the ether solution and subsequent drying with anhydrous sodium sulphate removes both water and alcohol. Ether, in the absence of water and alcohol, is easily removed from fatty acids by gentle heat.

empty bottles like that used for the sample. Add to each bottle 25 cc. of the Hanus solution (see reagents) and let stand with occasional shaking for one-half hour. Add 10 cc. of the 15 per cent potassium iodide solution and 100 cc. of water, and titrate with standard sodium thiosulphate, using starch as indicator. The titrations on the two blank tests should agree within 0.1 cc. From the difference between the average of the blank titrations and the titration on the sample and the iodine value of the thiosulphate solution, calculate the iodine number of the sample tested. (Iodine number is centigrams of iodine to 1 g. of sample.) If the iodine number is less than 170, the oil does not meet the specification.

(*j*) *Coarse Particles and Skins.*—Dry in an oven at 105 to 110° C. a No. 200 sieve, cool, and weigh accurately. Weigh an amount of semipaste containing 100 g. of pigment (see 3 (*f*)), 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. The total residue left on the sieve should not be more than 0.50 g.

4. ANALYSIS OF PIGMENT

(*a*) *Qualitative Analysis.* — A complete qualitative analysis following the well-established methods is always advisable, but the work may be usually very much shortened by adding acetic acid slowly to the pigment until all carbonate is decomposed (noting whether any hydrogen sulphide is evolved), then adding a large excess of acid ammonium acetate, boiling, filtering, and testing the filtrate for metals other than lead and zinc (especially calcium and barium). Material meeting the specification should show absence of metals other than lead and zinc in this solution. Test another portion of pigment with hydrochloric acid (1:1). No odor of hydrogen sulphide should develop.

(b) Extending Pigments.—Weigh accurately about 1 g. of the pigment, transfer to a 250 cc. beaker, moisten with a few drops of alcohol, add slowly dilute (about 20 per cent) acetic acid until all carbonate is decomposed (no further effervescence), then add 50 cc. of acid ammonium acetate solution (see reagents), and boil for two minutes. Decant through a weighed Gooch crucible, leaving any undecomposed matter in the beaker. Wash the Gooch crucible with a small amount (about 20 cc) of hot water. To the residue in the beaker add 50 cc. of the acid ammonium acetate solution and boil two minutes. Filter through the same Gooch crucible, transferring the insoluble matter to the crucible, and wash thoroughly with hot water. Dry the crucible at 105 to 120° C., cool, and weigh. Calculate the percentage of extending and tinting pigments.

(c) Total Lead.—Unite the filtrates from (*b*), heat to boiling, and add dropwise with stirring a light excess of dichromate solution (see reagents). Heat until the precipitate assumes an orange color, let settle, filter on a weighed Gooch crucible, wash by decantation with hot water until the washings are colorless, finally transferring all of the precipitate. Then wash with 10 cc. of 95 per cent alcohol and finally with 10 cc. of ethyl ether. Dry at 110 to 120° C. Cool and weigh the lead chromate. Calculate to PbO by multiplying by the factor 0.69.

(*d*) *Zinc Oxide.*—Weigh accurately about 1 g. of the pigment, transfer to a 400 cc. beaker, add 30 cc. of hydrochloric acid (1:2), boil for two or 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 hydro-

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Note.—If preferred, lead may be determined as sulphate.

chloric acid, heat nearly to boiling, and titrate with standard ferrocyanide as in standardizing that solution (see reagents). Calculate total zinc as zinc oxide.

(e) Lead Sulphate.—Treat 1 g. of the pigment in a 400 cc. beaker with a few drops of alcohol, add 10 cc. of bromine water, 10 cc. of hydrochloric acid (1:1), and 3 g. of ammonium chloride. Cover with a watch glass and heat on a steam bath for five minutes, add hot water to give a total volume of about 200 cc., boil for five minutes, filter, and wash thoroughly with hot water. Neutralize the clear filtrate in a covered beaker with dry sodium carbonate, add 1 g. more of dry sodium carbonate and boil for 10 to 15 minutes; wash off cover, let settle, filter, and wash with hot water. Redissolve the precipitate in hydrochloric acid (1:1), reprecipitate with sodium carbonate as above, filter, and wash thoroughly with hot water. Acidify the united filtrates with hydrochloric acid, adding about 1 cc. in excess. Boil to expel bromine, and to the clear boiling solution add slowly with stirring 15 cc. of barium chloride solution (see reagents). Let stand on a steam bath for about one hour, filter on a weighed Gooch crucible, wash thoroughly with boiling water, dry, ignite, cool, and weigh as barium sulphate. Calculate to lead sulphate, using the factor 1.3.

(*f*) *Lead Carbonate.*—Determine carbon dioxide by evolution with dilute acid and absorption in soda lime or potassium hydroxide solution. Calculate to basic lead carbonate ($2PbCO_3.Pb(OH)_2$), using the factor 8.81.

(*g*) *Calculations.*—From the percentage of lead sulphate (see 4 (e)), calculate the PbO equivalent, using the factor 0.736. From the percentage of $2PbCO_3.Pb(OH)_2$ (see 4 (*f*)), calculate the PbO equivalent, using the factor 0.863; add these two PbO equivalents and subtract from the total lead as PbO (see 4 (*c*)), add the remainder to the lead sulphate found (see 4 (*e*)) and report the sum as basic sulphate. Extending and tinting pigments (see 4 (*b*)), zinc oxide (see 4 (*d*)), and basic lead carbonate (see 4 (*f*)), are reported as already calculated.

5. LABORATORY EXAMINATION—MIXED PAINT

(*a*) *Caking in Container.*—Follow the procedure outlined in 3 (*a*), noting that the paint should be no more difficult to break up than a good grade of mixed paint.

(*b*) *Color.*—Follow the procedure outlined in 3 (*b*).

(*c*) *Weight per Gallon.*—Weigh a clean, dry, 100 cc. graduated flask. Fill to the mark with the thoroughhly mixed paint and weigh again. The increase in weight expressed in grams, divided by 100, gives the specific gravity, which multiplied by 8.34 gives the weight in pounds per gallon.

(*d*) *Brushing Properties and Time of Drying.*—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 stand for 18 hours. The paint should be dry and free from streaks.

(*e*) *Water.*—Mix 100 g. of the paint in a 300 cc. flask with 75 cc. of toluol. Connect with a condenser and distill 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. Material complying with the specification should yield less than 0.5 cc.

(*f*) *Volatile Thinner.*—Follow the procedure outlined in 3(*e*). Correct the result for any water found (see 5(*e*)) and report the remainder as volatile thinner.

(*g*) *Percentage of Pigment.*—Follow the procedure outlined in 3(*f*).

(*h*) *Testing Nonvolatile Vehicle.*—Follow the procedure outlined in 3(*g*), 3(*h*), and 3(*i*), except that in the preparation of the fatty acids the mixture of paint and alkali is heated on the steam bath until all volatile thinner is driven off.

(*i*) *Coarse Particles and Skins.*—Follow the procedure outlined in 3(*j*).

(*j*) *Testing Pigment.*—Follow the procedure outlined in 4(*a*) to 4(*g*) inclusive.

6. REAGENTS

(*a*) *Acid Ammonium Acetate Solution.*—Mix 150 cc. of 80 per cent acetic acid, 100 cc. of water, and 95 cc. of strong ammonia (specific gravity, 0.90).

(*b*) *Dichromate Solution.*—Dissolve 100 g. of sodium dichromate ($Na_2Cr_2O_{72}H_2O$) or potassium dichromate ($K_2Cr_2O_7$) in water and dilute to 1000 cc.

(*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 1000 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 amounts used in standardization and in titration of the sample. The standardization must be made under the same conditions of temperature, volume, and acidity as obtained when the sample is titrated.

(*e*) *Barium Chloride Solution.*—Dissolve 100 g. of pure crystallized barium chloride in water and dilute to 1000 cc.

(*f*) *Standard Sodium Thiosulphate Solution.*—Dissolve pure sodium thiosulphate in distilled water (that has been well boiled to free it from carbon dioxide) in the proportion of 24.83 g. crystallized sodium thiosulphate to 1000 cc. of the solution. It is best to let this solution stand for about two weeks before standardizing. Standardize with pure resublimed iodine. (See Treadwell-Hall, Analytical Chemistry, vol. 2, 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. Preserve in a stock bottle provided with a guard tube filled with soda lime.

(*g*) *Starch Solution.*—Stir up 2 to 3 g. of potato starch or 5 g. of soluble starch with 100 cc. of 1 per cent salicylic acid solution, add 300 to 400 cc. boiling water, and boil the mixture until the starch is practically dissolved, then dilute to 1 liter.

(*h*) *Extraction Mixture.*—

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

(*i*) *Aqueous Sodium Hydroxide.*—Dissolve 100 g. of sodium hydroxide in distilled water and dilute to 300 cc.

(*j*) *Potassium Iodide Solution.*—Dissolve 150 g. of potassium iodide free from iodate in distilled water and dilute to 1000 cc.

(*k*) *Hanus Solution.*—Dissolve 13.2 g. of iodine in 1000 cc. of 99.5 per cent glacial acetic acid, which will not reduce chromic acid. Add enough bromine to double the halogen content, determined by titration (3 cc. of bromine is about the proper amount). The iodine may be dissolved by the aid of heat, but the solution should be cold when the bromide is added.

(*l*) *Alcoholic Sodium Hydroxide Solution.*—Dissolve pure sodium hydroxide in 95 per cent ethyl alcohol in the proportion of about 22 g. per 1000 cc. Let stand in a stoppered bottle. Decant the clear liquid into another bottle and keep well stoppered. The solution should be colorless or only slightly yellow when used, and it will keep colorless longer if the alcohol is prviously treated with sodium hydroxide (about 80 g. to 1000 cc.), kept at about 50° C., for 15 days, and then distilled.

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