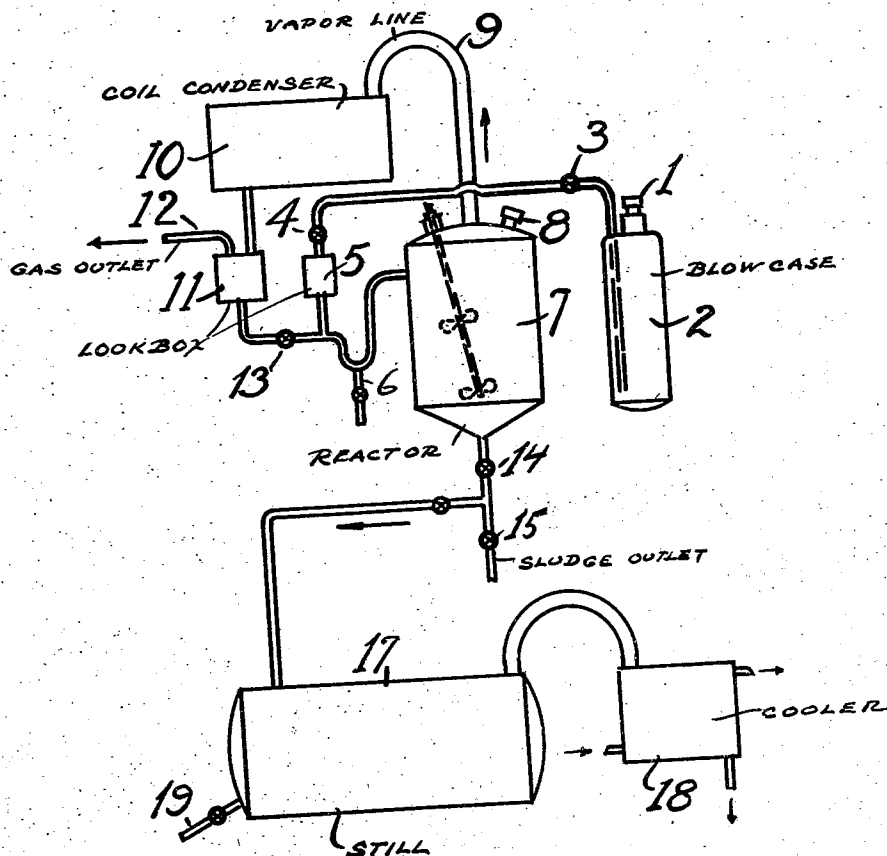


Dec. 14, 1943.

E. LIEBER ET AL
LUBRICATING OILS
Filed Oct. 4, 1939

2,336,620



Eugene Lieber Inventors
Aloysius F. Cashman
By *R. P. Young* Attorney

UNITED STATES PATENT OFFICE

2,336,620

LUBRICATING OIL

Eugene Lieber, Staten Island, N. Y., and Aloysius F. Cashman, Bayonne, N. J., assignors to Standard Oil Development Company, a corporation of Delaware

Application October 4, 1939, Serial No. 297,882

16 Claims. (Cl. 252-59)

This invention relates to the production of lubricating oil compositions of low pour point and of desirable color characteristics in both transmitted and reflected light and to the compounds incorporated therein which effect such desirable features in lubricating compositions otherwise deficient in these attributes.

The pour point, color and fluorescent characteristics of an untreated petroleum distillate vary with the type of crude from which it is obtained and also with the boiling range of the distillate. The pour point of an oil is determined by the nature and amount of waxy hydrocarbon content. The quality of the fluorescence, or cast, of a refined stock depends greatly upon the refining treatment; distillates tend to be bluish, while residuals tend to be green; and acid treatment tends to eliminate the green cast and cause the development of a brownish-blue cast. The lubricating oil stocks from Pennsylvania crudes set the standard of color and cast in the early days of the use of petroleum lubricants and today the green cast, or "bloom," as it is commonly termed, of these oils is considered to be more advantageous, especially from the commercial sales viewpoint, than the blue or other cast appearance of lubricating oil stocks derived from the Coastal or highly naphthenic type crudes.

In addition to the more pleasing appearance of these oils, a feature of utility of the green bloom is that it conveys to the oil so characterized, a better visibility against a dark or light background than a blue or other color cast. The sales preference for lubricating oils possessed of low pour point, the acceptable olive or golden green cast and the reddish color by transmitted light, has established the need for the manufacture of lubricating oil addition agents which can suitably impart to composites these desired characteristics without adversely affecting other desirable properties of the lubricating oil composition. The present invention relates to this field.

An object therefore of the invention is the preparation of a composition of matter which when admixed in small amounts imparts to lubricating oil stocks of poor cast and color characteristics, usually associated with oils derived from coastal and naphthenic type crudes, a desirable olive or golden green cast and reddish color by transmitted light.

Another object of the invention is the preparation of a composition of matter which depresses the pour points of waxy lubricating oil stocks when admixed therewith in small proportions.

Another object of the invention is the prepara-

tion of lubricating oil compositions of low pour point and good color characteristics in both transmitted and reflected lights by incorporating in lubricating oil stocks deficient in these respects, small amounts of a compound the preparation of which is hereinafter disclosed.

In the preparation of the lubricating oil addition agent of this invention, according to one method an organic compound of cyclic structure, such as the aromatic hydrocarbons and their substitution products, a low boiling alkyl polyhalide and a condensing agent are heated as a mixture to a pre-determined temperature and a low boiling alkyl mono-halide added, and heating continued for a sufficient time to bring about the formation of a condensation product which separates when the reaction mixture is suitably hydrolyzed. A superior product is prepared by continuing the condensation one step further. Thus, to the heated reaction mixture a small quantity of halogenated high molecular weight aliphatic compound, such as chlor-wax, is added and heating continued until a second condensation takes place.

The cyclic hydrocarbon may be of aryl, alkaryl or heterocyclic structure, but preferably of condensed ring type such as an aromatic. The aromatic hydrocarbons which give superior products are benzene, naphthalene, anthracene and phenanthrene, and of these, naphthalene serves particularly well. Derivatives of these hydrocarbons such as halogeno (e. g., mono-chloro-), hydroxy, alkoxy, amino, alkamino substitution products of aromatic hydrocarbons, which react similarly to the hydrocarbons themselves in the condensation reaction, may also be employed. The low boiling alkyl polyhalides which have been found to be suitable are the dihalides of short chain aliphatic hydrocarbons, having less than 10 carbon atoms in the molecule, but preferably containing from 1 to 5 carbon atoms and in which the chlorine atoms are in adjacent positions. Very suitable compounds of this class are dichlor-methane, dichlor-ethane and dibromo-propane. In general, compounds which can be suitably employed instead of the low molecular weight poly-halides, are any of the halogenated derivatives of the hydrocarbons containing up to six carbon atoms and not more than two halogen atoms per molecule. The low boiling aliphatic mono-halide may be any mono-halide derived from a lower saturated hydrocarbon such as propane, butane and pentane. Instead of the aliphatic halides, the corresponding olefins may be used. Instead of the halogenated high molec-

ular weight aliphatic compounds, the corresponding unsaturated derivatives may be used. Suitable condensing agents are those of the Friedel-Crafts type, namely aluminum chloride, zinc chloride, boron fluoride, and the like, but acid catalysts such as sulfuric and phosphoric acids, and known equivalents may also be used.

As an illustration of this invention, the process is carried out by employing approximately equimolecular quantities of the cyclic hydrocarbon or the corresponding halogen derivative, and the low molecular weight aliphatic poly-halide, and about 20% of condensing agent with or without the use of an inert solvent, such as naphtha and inert highly chlorinated hydrocarbons such as tetrachlorethane, di- and tri- chlor-benzol, etc. The mixture is mechanically stirred and heated to about 85° F. The aliphatic mono-halide is then gradually added to the mixture over a period of about 20 minutes while maintaining the reaction temperature at about 85° F. After the addition of the alkyl mono-halide, the reaction mixture is stirred at about 85° F. for five hours in order to complete the initial part of the condensation. The temperature is then raised to the reflux point and maintained there for from 2 to 12 hours in order to complete the condensation. The product so obtained may be hydrolyzed by treating with a dilute hydrochloric acid solution, that is, a solution containing about 5% hydrochloric acid.

Another method of preparing the dye material, and one which gives a product of improved cast and potency, is to treat the condensation product previously prepared with chlorinated paraffin wax in an amount of 25% by weight of the reaction mixture by adding gradually over a 15 minute period while the temperature is held at about 230° F. The product so obtained may be hydrolyzed according to the details given for the first process, or the reaction mixture may be diluted with ethylene dichloride, or some other suitable solvent, cooled to about 150° F., neutralized with an isopropyl alcohol-aqueous caustic solution, and after settling, the ethylene dichloride solution is distilled with fire and steam to 600° F. to recover the dye product.

This method of preparing the lubricating oil addition agent bears some similarity to the process disclosed in the co-pending application of Eugene Lieber, Serial No. 245,673 now Patent No. 2,270,062. In that application, pour point depressants are prepared by the condensation of an alkylated cyclic compound with a di-halo organic compound. When the cyclic radical is of condensed structure such as in anthracene, phenanthrene and naphthalene, the condensation products obtained exhibited dye properties. The yields obtained in such cases are low and the preparation of dye products by that process is uneconomic.

In the present invention, the order of adding the reagents differs from that disclosed in the co-pending application, Serial No. 245,673 now Patent No. 2,270,062, and in the alternative method of preparing the dye product, the condensation is carried one step further than is disclosed in the co-pending application. The products obtained by the present process are more satisfactorily soluble in all types of lubricating oil stocks, and the potency and the yields are considerably greater than those obtainable by the process disclosed in the co-pending application No. 245,673 now Patent No. 2,270,062.

The products prepared according to this in-

vention are evaluated by standard tests for pour point and color characteristics of oil compositions. The pour point of an oil is the temperature at which the oil fails to flow in the test jar under standard conditions. The potency of the dye product is estimated in terms of actual color units which are determined by dissolving the material in an oil of standard color, and evaluating the color change in the Robinson colorimeter. The determination of the cast or "bloom" imparting qualities depends largely upon the judgment or experience of the observer. The cast may be evaluated by means of a tint photometer and results expressed in the form of a color absorption graph, but this method is today very seldom employed. It is more usual to evaluate the cast by arranging a series of Pennsylvania oils and dyed pale oils in order of "pleasing effect on the eye," and rating the dyed oils with regard to the appearance of the Pennsylvania stocks.

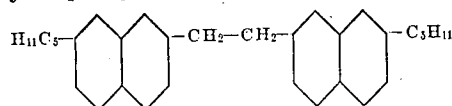
The amount of the product employed for purposes of lowering the pour point and of dyeing lube oils deficient in color and cast, depends greatly upon the extent of the deficiency and on the potency of the product. Usually, however, it is added to an oil in proportions varying from 0.5% to 4% of the oil, depending on the nature of the oil and the potency of the material. The preferable range is an amount varying from about 1% to 2% by volume of the oil.

The following examples will serve to illustrate the invention:

EXAMPLE 1

Into a 3-liter, 4-necked, round bottom flask, equipped with reflux condenser, dropping funnel, thermometer and provision for heating, was placed 280 grams of naphthalene, 220 grams of ethylene dichloride and 44 grams of aluminum chloride. The stirrer was started and the amyl chloride ("mixed amyl chlorides" of commerce) was added to the reaction mixture at such a rate that the reaction temperature did not exceed 90° F. This required from 30 to 40 minutes. After the addition of the amyl chloride, the reaction mixture was allowed to agitate for two hours at 90° F. and then the temperature was increased until the ethylene dichloride was vigorously refluxing (210-230° F.) and maintained thereat for two hours. The reaction mixture was diluted with ethylene dichloride (200-400 cc.), cooled to 150° F. and hydrolyzed with a mixture of alcohol and water. After settling, the ethylene dichloride extract was distilled with fire and steam to 600° F. in order to remove the solvent and low boiling products. A bottoms residue comprising 306 grams (64% yield) of the dye-condensation product was obtained.

The distillate obtained up to 600° F. is settled and the water condensate removed and discarded. The remainder is distilled to recover ethylene dichloride and about 15% of a low molecular weight condensation product. This low molecular weight condensation product comprised chiefly di-(amyl-naphthyl)-ethane



and it was found that this material could be converted to the same valuable dye product by merely refluxing with ethylene dichloride in the

presence of aluminum chloride, or that it may be recycled back to the process as described above. The 64% yield previously given was calculated in this manner.

When 0.15% of this dye product was added to a petroleum distillate lubricating "pale oil" of 11 Robinson color, the blend had a 4¼ Robinson color and a brilliant red color by transmitted light and a pleasing green fluorescence. In addition a very good "match" of the glass on the colorimeter machine was obtained. To those skilled in the art, the significance of that term is clear, i. e., the dyed pale oil approaches very closely to the appearance of a Pennsylvania oil in color and cast. The addition of 0.23% of the condensation product to the same pale oil gave a blend of a 1¼ Robinson color with a much more intense brilliant red color and green bloom. On the other hand, in order to obtain the same color drops with a commercial cracking coil tar dye now on the market, it was necessary to add from 0.45% and 1.00%, respectively, of that material. Further, there is obtained only a "poor match of the glass" on the colorimeter machine and at the same time it is deficient in green bloom, a fact which sets it aside immediately from the color of Pennsylvania type oils.

EXAMPLE 2

The procedure and proportion of reagents of Example 1 was repeated exactly except that the time of reflux or condensation period was increased from 2 to 4 hours. The dye-condensation product was recovered as previously described. A yield of 66% was obtained. However, from the point of view of its use as a dye-stuff, the yield has practically doubled over Example 1, as subsequently indicated.

The dye-condensation products of Examples 1 and 2 and cracking coil tar dye were added to a pale oil of Robinson color 11 until 2 and 4 Robinson colors were respectively obtained.

The following table shows the comparative results obtained:

Dye	Per cent of dye	Robinson color	Match of glass
Cracking coil.....	None	11	
Tar dye.....	1.00	2	Off shade.
From Example 1.....	0.50	4	Do.
From Example 2.....	0.45	2	Very good match.
	0.225	4	Do.
	0.225	2	Do.
	0.1125	4	Do.

It will be observed that the dye product of Example 2 was twice as potent in tinctorial power as the dye product of Example 1. In comparison with cracking coil tar dye, the tinctorial power of the dye products of Examples 1 and 2 were from 2 to 4 times as powerful.

EXAMPLE 3

280 grams of naphthalene, 220 grams of ethylene dichloride and 44 grams of aluminum chloride, were charged to a 3-liter, 4-neck round bottom flask fitted with mechanical stirrer, thermometer, reflux condenser and dropping funnel. The stirrer was started and 236 grams of amyl chloride was added to the reaction mixture drop by drop over a period of 20 minutes while maintaining a reaction temperature of 75-85° F. After the addition of the amyl chloride, the reaction mixture was stirred at 75-85° F. for 5 hours. The temperature was increased to 230-235° F. and maintained thereat for 5 to 6 hours,

at which time a noticeable decrease was observable in the reflux of ethylene dichloride. 100 grams of chlorinated paraffin wax (121° F. M. P., chlorinated to 34.5° A. P. I. gravity) was then added to the reaction mixture drop by drop over a period of 15 minutes while maintaining a reaction temperature of 230-235° F. Reaction of the condensation-polymer with the chloro-wax was extremely vigorous under the conditions used which makes it unnecessary to prolong the reaction much beyond the total addition of the chloro-wax. After the addition of all of the chloro-wax, the reaction was continued for 15 minutes at 230-235° F., then diluted with 1000 grams of ethylene dichloride and cooled to 125-150° F. and neutralized with an isopropyl alcoholic caustic solution. After settling, the ethylene dichloride extract was distilled with fire and steam to 600° F. to recover the dye product.

A total yield of 413 grams of dry dye was obtained which corresponds to a 72.1 per cent yield based on all of the reagents including the chloro-wax. Examination of the dyeing potency showed 7.8×10^5 actual color units.

EXAMPLE 4

The agitator comprised a 3-liter nickel reaction vessel, similar in construction to the glass reactor described in Example 1.

The following proportions of reagents were used:

	Grams
Naphthalene	280
Ethylene dichloride	220
Aluminum chloride	44
Mixed amyl chlorides	236
Chlorinated wax (11.8% Cl)	100

The procedure used for carrying out the dye synthesis was the same as described in Example 3. The dye-product was recovered as before by cooling the reaction mixture to about 100-125° F., diluting with ethylene dichloride and decomposing the aluminum chloride with an isopropyl alcoholic aqueous caustic solution. After settling, the ethylene dichloride extract is distilled with fire and steam to 600° F. to recover the dye product.

A total yield of 361 grams of dry dye was obtained which corresponds to a 63.1% yield based on all of the reagents including the chloro-wax. Examination of the dyeing potency showed 6.1×10^5 actual color units.

The dye prepared as described above was added to various oils, whose original inspections are indicated in the table given below, for color deepening and cast improvement.

The following table summarizes the results obtained:

		Oil		#1		#2	
Mg. dry dye/100 ml. oil.....				0	100	0	100
Inspection:							
65	Flash °F.....	385	395	375	370		
	Viscosity @ 100 °F.....	153.5	154.2	518	514		
	Viscosity @ 210 °F.....	43.8	44.1	54.8	54.7		
	Viscosity index.....	106	111	28	28		
	Robinson color.....	12	4.7	12.7	4.7		
	Color hold (16 hrs. @ 210 ° F.).....	10.5	4.5	10.2	4.2		
	Daylight stability (10 days).....	11.8	4.6	14.4	4.7		
70	Conradson carbon.....	0.01	0.03	0.04	0.07		
	Ramsbottom coke "A".....	0.11	0.13	0.16	0.17		
	Ramsbottom coke "B".....	1.11	0.68	1.19	1.24		
	Slight oxidation test.....	3	8	48	49		
	Oxygen absorption test—						
	@ 24 hours.....	18	4	18	20		
	@ 48 hours.....	95	48	96	90		
75	@ 72 hours.....	128	80	170	157		

It will be observed from an inspection of the table that the dye had no deteriorating effect upon the quality of the oil.

Attention is directed to the stability of the dye product under severe conditions of test. Samples of dyed oils #1 and #2 containing 100 mg. of the dry dye (see table) were placed in a thermostatically controlled oven at 210° F. for 16 hours. It will be observed from the table that the dyed samples underwent practically no change, while the original oils suffered a change in color. The same relationship is observed in the daylight stability test.

EXAMPLE 5

The following example illustrates the use of the lower olefins in place of the low boiling aliphatic mono-halides.

The following reagents were used in the proportions indicated:

	Grams
Mixed amylenes	35
Naphthalene	65
Ethylene dichloride	50
Chlorinated wax (12% Cl)	25
Aluminum chloride	10

The procedure used for carrying out the reaction and for recovery of product was the same as described for Example 3.

A total yield of 72 grams of dry dye was obtained which corresponds to a 58% yield based on all the reagents. Examination of the dyeing potency showed 5.8×10^5 actual color units. The dyed oils showed a good cast and an excellent through red color.

When 2% of the dry dye, obtained as described above, was added to a waxy-oil, the pour point of which was +30° F., the pour point was found to be -10° F.

EXAMPLE 6

The following example illustrates the pour depressant potency of the products of this invention.

The dye product tested was that obtained in Example 3. This was blended in various concentrations in a waxy-oil and the pour point determined by the A. S. T. M. procedure. The following results were obtained:

	Pour point, °F.
Original waxy-oil	+30
Original waxy-oil+1% dry dye (Ex. #3)	-10
Original waxy-oil+2% dry dye (Ex. #3)	-20

EXAMPLE 7

The following example is offered as a distinction of the present invention from the process outlined in co-pending application Serial No. 245,673.

The following reagents:

	Grams
Mono-amyl naphthalene	200
Ethylene dichloride	220
Aluminum chloride	22

were reacted by the procedure of co-pending application Serial No. 245,673, now Patent No. 2,270,062, i. e., by heating to the reflux temperature of the ethylene dichloride and maintaining thereat for 3 hours, at which time the reaction mixture was cooled, diluted with ethylene dichloride and the aluminum chloride decomposed by the addition of alcohol and water. The ethylene dichloride extract, after settling, was dis-

tilled with fire and steam to 600° F., in order to remove the solvent and to recover the dry dye.

A total yield of 58 grams of dry dye was obtained. This corresponds to a 22% yield based on all reagents. A determination of the dyeing ability showed 2.8×10^5 actual color units. Further, the green cast imparted to the oil is of an inferior quality compared to the dye made by the present process.

EXAMPLE 8

The following is a comparison of the cast or bloom of dyed pale oils (viscosity of 200 Saybolt Universal seconds at 100° F.) and correspondingly representative Pennsylvania oils.

Procedure

The following samples matched to give 2 Robinson color were placed in 4 oz. bottles and compared:

Filtered Pennsylvania Bright stocks	3
Unfiltered Pennsylvania Bright stocks	2
Samples of pale oil blended to 2R color respectively with 0.17% of product of Exp. 1, 0.24% of product of Exp. 3, and 1% Commercial cracking coil tar dye product	3

Results

The oils were examined and placed in order of cast from golden green to bluish green.

Order of cast	Remarks
1 to 3 filtered Penn. Bright stocks	Golden green bloom showing little distinction.
4 pale oil+dye product of Exp. 3	Golden green bloom slightly less marked than above.
5 pale oil+dye product of Exp. 1	Golden green bloom but green more pronounced.
6 to 7 unfiltered Penn. Bright stocks	Grass green with no pronounced golden color.
8 Pale oil+Com. cracking coil tar dye	

EXAMPLE 9

In effecting the invention upon a semi-plant scale, equi-molecular proportions of naphthalene, ethylene dichloride and mixed amyl chlorides were used together with 20% by weight anhydrous aluminum chloride based on the ethylene dichloride and 90% by weight of chlor-wax (12% Cl) based on the ethylene dichloride. The apparatus employed is diagrammatically represented in the accompanying drawing to which relate the numerals used in the following description.

Naphthalene (138 lbs.) was added through a removable cap 8 to a nickel lined reactor 7, connected by vapor line 9 to a condenser 10 fitted with gas outlet 12 through look box 11. Ethylene dichloride (110 lbs.) was poured in through the same opening. Aluminum chloride (20 lbs.) was added through this opening and then the cap was replaced and the mixer started. Mixed amyl chlorides (120½ lbs.) were then placed in the blowcase¹ 2 through the removable cap 1 in the top. The case was sealed and heated by means of a steam coil until 4 lbs. of pressure was built up. The valves 3 and 4 between the blowcase and the reactor were opened and valves 5 and 13 closed, and the amyl chloride blown through look box 5 into the reactor over a period of 1¾ hours. Valves 3 and 4 were closed and valve 13 opened. Temperature of the reactor was maintained at 75° F.

¹ The blowcase 2 could be replaced with a gravity feed tank for the addition of liquids to the reactor.

Agitation was continued for 2 hours, the temperature of the reactor being maintained at 80° F. Chlor-wax (12% Cl) (100 lbs.) was then placed in the blowcase and was blown over into the reactor over a period of 2 hours, the temperature of the reactor being maintained at 90° F. The reactor was then maintained at 85° F. for 16 hours, vigorous agitation being maintained throughout. It was then heated by means of a steam coil to 185° F. over a period of five hours. At 185° F. refluxing started. The temperature was then allowed to rise naturally as the refluxing permitted. Maximum temperature was 226° F. The refluxing was continued for 7 hours, then the reactor was cooled, diluted with 30 gallons of ethylene dichloride, and neutralized by blowing into the reactor through the blowcase 45 gallons of water, 10 gallons of 38° Baumé caustic soda, and 8 gallons of 91% isopropyl alcohol. Agitation was then stopped and the mixture allowed to settle overnight. The sludge was then separated by opening valves 14 and 15. The valves were closed and then 20 gallons of kerosene and 20 gallons (153 lbs.) of very heavy cable oil were added to the contents of reactor 7. This mixture was charged to the still 17 by opening valves 14 and 16.

The still was fired and the ethylene dichloride, kerosene, and unreacted low boiling materials taken overhead by a fire and steam distillation to 600° F., and cooled in condenser 18. The residue after cooling was removed through valve 19. This residue was a very viscous oil and weighed 313 lbs.

Subtracting the 153 lbs. of cable oil which does not distill under these conditions, there was in the still bottoms, 160 lbs. of dye concentrate. This corresponds to a per cent weight yield of 48.5%.

The following inspections were made on the still bottoms:

S. U. viscosity @ 210° F.	seconds	199
A. P. I. gravity @ 60° F.	degrees	16.7
O. C. flash point	°F.	465
O. C. fire point	°F.	525
Cl.	per cent.	0.18
Dye value	a. c. u.	2.64×10^5
Neut. No.		0.05
Saponification No.		0.60

A small portion of the ethylene dichloride solution was reduced separately with no cable oil in the still. The concentrate is a dark brown resin which can be powdered. It is soluble in all proportions in petroleum lubricating oils. The melting point of the resin varies with the amount of steam stripping done. The powdered material is dark brownish red in color.

It is to be understood that the scope of the invention is not to be limited by or to the examples given, which are presented merely for the purpose of illustration and that modifications and variations may be made therein without departing substantially from the spirit of the invention.

We claim:

1. A condensation product soluble in lubricating oil and having the properties of depressing the pour point of waxy lubricating oils, and of imparting to lubricating oils of poor color and cast characteristics a deep reddish color by transmitted light and a golden green color by reflected light, comprising a condensation product prepared by adding a low boiling aliphatic compound containing not substantially more than 5 carbon

atoms of the class consisting of saturated mono-halides and the corresponding unsaturated compounds to an unreacted mixture of an aromatic compound and an aliphatic poly-halide having less than 10 carbon atoms and not more than 2 halogen atoms and a condensing agent and heating to a reaction temperature.

2. A condensation product soluble in lubricating oil and having the properties of depressing the pour point of waxy lubricating oils and of imparting to lubricating oils of poor color and cast characteristics a deep reddish color by transmitted light and a golden green color by reflected light, comprising a condensation product prepared by adding a low boiling alkyl mono-halide containing not more than 5 carbon atoms to an unreacted mixture of an aromatic hydrocarbon, an aliphatic poly-halide having less than 10 carbon atoms and not more than 2 halogen atoms and a condensing agent and heating to a reaction temperature.

3. A condensation product soluble in lubricating oil having the properties of depressing the pour point of waxy lubricating oils and of imparting to lubricating oils of poor color and cast characteristics a deep reddish color by transmitted light and a golden green color by reflected light, comprising a condensation product prepared by adding amyl chloride to an unreacted mixture of naphthalene and ethylene dichloride and a condensation agent of the Friedel-Crafts type and heating to a reaction temperature.

4. An improved condensation product soluble in lubricating oil and having the properties of depressing the pour point of waxy lubricating oils and of imparting to lubricating oils deficient in color and cast characteristics a deep reddish color by transmitted light and a golden green color by reflected light, comprising a condensation product prepared by the reaction of an aromatic compound and an aliphatic di-halide having less than 10 carbon atoms with a low boiling aliphatic compound containing not substantially more than 5 carbon atoms of the class consisting of saturated mono-halides and a corresponding unsaturated compound and a condensing agent and the subsequent condensation of said product with a halogenated high molecular weight aliphatic compound.

5. An improved condensation product soluble in lubricating oils and having the properties of depressing the pour point of waxy lubricating oils and of imparting to lubricating oils deficient in color and cast characteristics a deep reddish color by transmitted light and a golden green color by reflected light, comprising a condensation product prepared by the reaction of an aromatic hydrocarbon, an aliphatic di-halide having less than 6 carbon atoms, an alkyl mono-halide having 3 to 5 carbon atoms and a condensing agent of the Friedel-Crafts type, and the subsequent condensation of said product with a halogenated high molecular weight aliphatic compound.

6. An improved condensation product soluble in lubricating oils and having the properties of depressing the pour point of waxy lubricating oils and of imparting to lubricating oils deficient in color and cast characteristics a deep reddish color by transmitted light and a golden green color by reflected light, comprising a condensation product of the reactions of naphthalene, ethylene dichloride, amyl chloride and aluminum chloride at a temperature between about 85° F. and reflux temperature, and the subsequent condensation of

said product with chlor-wax in the presence of aluminum chloride.

7. The process for producing lubricating oil addition agents having the properties of being soluble in lubricating oils of depressing the pour point of waxy lubricating oils and of imparting to lubricating oils deficient in color and cast characteristics a deep reddish color by transmitted light and a golden green color by reflected light, comprising adding a low boiling aliphatic compound containing not substantially more than 5 carbon atoms of the class consisting of saturated mono-halide and the corresponding unsaturated compounds to an unreacted mixture of an aromatic compound and an aliphatic poly-halide having less than 10 carbon atoms and not more than 2 halogen atoms and a condensing agent and heating to a reaction temperature.

8. An improved process for preparing lubricating oil addition agents having the properties of being soluble in lubricating oils of depressing the pour point of waxy lubricating oils and of imparting to lubricating oils deficient in color and cast characteristics a deep reddish color by transmitted light and a golden green color by reflected light, comprising adding a low boiling aliphatic compound having 3 to 5 carbon atoms of the class consisting of saturated mono-halides and the corresponding unsaturated compounds to an unreacted mixture of an aromatic hydrocarbon compound, an aliphatic di-halide having less than 6 carbon atoms and a Friedel-Crafts condensing agent, heating the mixture to a reaction temperature, and subsequently condensing product of said reaction with a halogenated high molecular weight aliphatic compound.

9. An improved lubricating oil of low pour point and of deep red color by transmitted light and a golden green color by reflected light, comprising a lubricating oil and a product of claim 1.

10. An improved lubricating oil of low pour point and of deep reddish color by transmitted light and a golden green color by reflected light, comprising a lubricating oil and a product of claim 4.

11. An improved lubricating oil of low pour point comprising a waxy lubricating oil having normally a high pour point and a small amount, effective in reducing the pour point of said oil, of the product described in claim 1.

12. An improved lubricating oil having desirable characteristics of deep red color by transmitted light and golden green color by reflected

light, comprising a lubricating oil of undesirable color characteristics and a small amount, effective in improving the color characteristics of said oil, of the product described in claim 1.

13. An improved lubricating oil having the desirable characteristics of deep red color by transmitted light and a golden green color by reflected light comprising a lubricating oil of undesirable color characteristics and a small amount, effective in improving the color characteristics of said oil, of the product described in claim 4.

14. An improved lubricating oil having the desirable characteristics of deep red color by transmitted light and a golden green color by reflected light comprising a lubricating oil of undesirable color characteristics and a small amount, effective in improving the color characteristics of said oil, of the product described in claim 6.

15. An improved process for preparing lubricating oil addition agents having the properties of being soluble in lubricating oils and of imparting to lubricating oils deficient in color and cast characteristics a deep reddish color by transmitted light and a golden green color by reflected light comprising adding a low boiling aliphatic compound having 3 to 5 carbon atoms of the class consisting of saturated monohalides and the corresponding unsaturated compounds to an unreacted mixture of naphthalene, an aliphatic dichloride having less than 6 carbon atoms and a Friedel-Crafts condensing agent, heating the mixture to a reaction temperature between the approximate limits of 85° F. and reflux temperature, and subsequently condensing the product of said reaction with a halogenated high molecular weight aliphatic compound.

16. The process which comprises mixing approximately equimolecular quantities of naphthalene and ethylene dichloride, adding about 20% by weight of aluminum chloride, based on the weight of ethylene dichloride used, stirring and heating the mixture to about 85° F. without causing any substantial reaction, gradually adding thereto an approximately equimolecular amount of mixed amyl chloride, maintaining the resultant mixture at about 85° F. to complete the initial part of the condensation, then raising the temperature to reflux temperature and maintaining it thereat for about 2 to 12 hours to complete the condensation, and hydrolyzing and removing the catalyst.

EUGENE LIEBER.

ALOYSIUS F. CASHMAN.