

July 29, 1958

M. H. EDSON
ASPHALT COMPOSITION

2,845,359

Filed Dec. 29, 1953

2 Sheets-Sheet 1

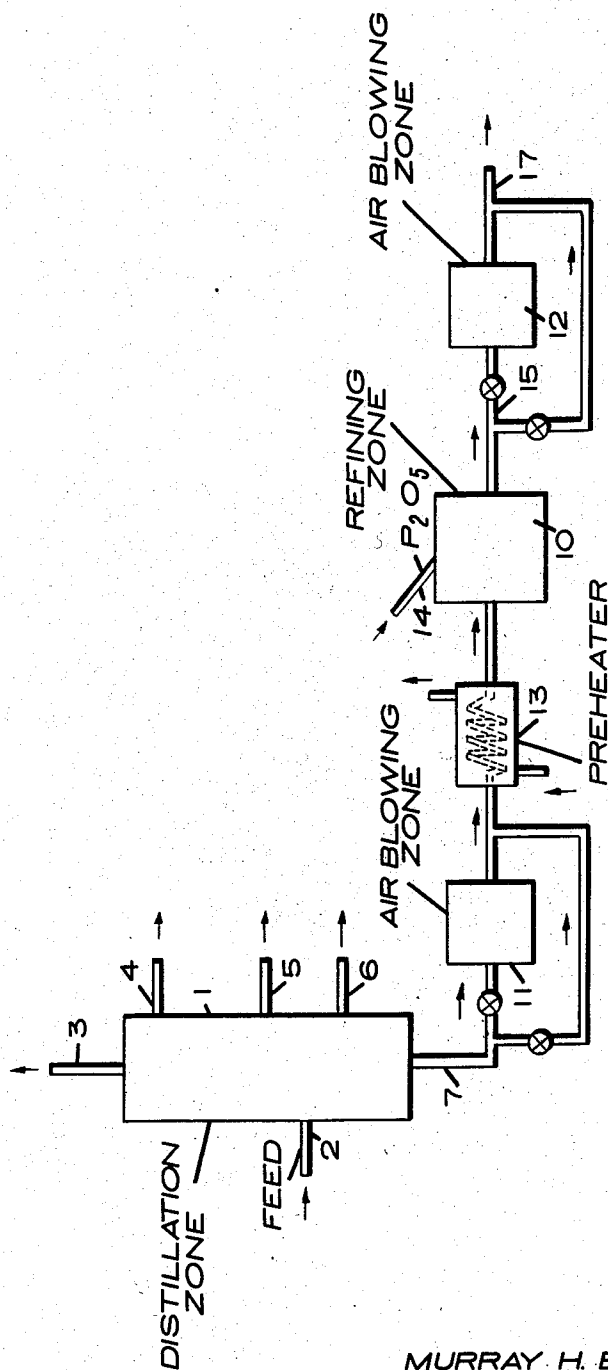


FIG. 1

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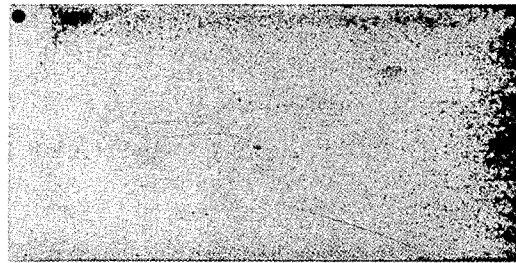
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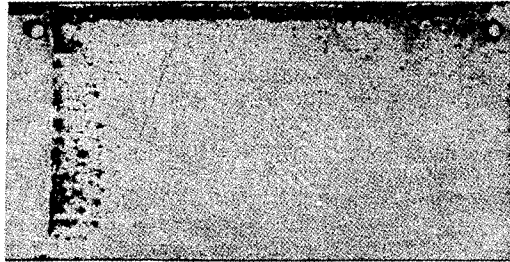
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2 Sheets-Sheet 2

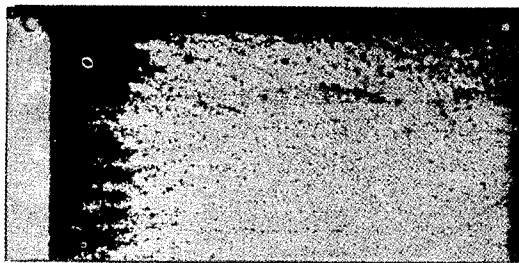
FIG. - 2
THIN FILM RUST PREVENTIVES
50 DAY ACCELERATED WEATHERING TEST



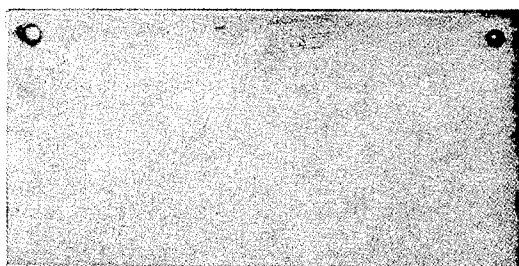
160/180



180/200



220/235



ASPHALT P_2O_5 TREATED
SOFTENING POINT 220/235

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Inventor

By *L. F. Dray*

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2,845,359

ASPHALT COMPOSITION

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Application December 29, 1953, Serial No. 400,964

4 Claims. (Cl. 106—14)

This invention concerns compositions, including asphalts, made by a novel process for treating residual oils and hydrocarbon residues to obtain asphalts of desirable properties. The asphalts have special utility in cutback compositions. The process employed depends upon a heat treatment of the residual oil in the presence of P_2O_5 . This process can in many cases be advantageously varied by air blowing the residual oil prior to contact with P_2O_5 , or by air blowing the asphalt obtained by treatment of residual oil with P_2O_5 . While as indicated, the process is particularly adapted for the treatment of residual oils to recover asphalts therefrom; the process may also be employed for the upgrading of asphalts obtained by conventional processing steps.

This application is a continuation-in-part of copending application, Serial No. 228,499, filed on May 26, 1951, now U. S. Patent No. 2,676,910.

The present invention concerns the recovery of asphalts and the refining and use of asphalts obtained from petroleum oils. The principal objective of the process herein disclosed is to provide asphalts having unusual properties; in particular, asphalts are obtained which have unusually high penetration and ductility properties for a given softening point, although, as will be brought out, other desirable properties may also be realized. These include non-gelling and excellent rust prevention characteristics when used in cutback asphalt compositions. This unusual combination of characteristics is demanded in a number of applications, examples of which include the lining of irrigation canals, thin film rust preventives, etc. Asphalts employed to line irrigation canals ideally should have properties to enable the asphalt lining to retain its shape in hot weather, to be non-brittle in cold weather, and to conform to the contour of the surface of the canal without tearing. Asphalts having these properties must have a softening point of about 175° to 200° F., and must have high ductility and high values of penetration. It has been found that asphalts obtained by conventional processing methods do not have the necessary combination of properties. More precisely, the asphalts obtained by conventional processing techniques, in the range of softening points identified, have too low a ductility and too low a penetration to meet the desired characteristics.

It is therefore the principal object of this invention to provide an asphalt recovery and treating process which will vary the usual combination of softening point, penetration, and ductility properties. To bring out the manner in which these asphalt properties are affected, it is helpful to consider the results of subjecting crude residua to an oxidation treatment as achieved by conventional air blowing. Oxidation treatments of this character are well known to the art and it has been appreciated that the low temperature susceptibility of asphalts may to some extent be improved by oxidation. However, when

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air blowing is applied and continued, while the softening point of an asphalt is raised, the penetration is decreased and the ductility is lowered. Consequently, contra to this change in asphalt properties, the process of this invention provides an increase in penetration properties and an increase in ductility concomitant with the increase in softening point of the asphalt.

The process by which these desirable changes in asphalt properties are obtained depends upon the catalytic treatment of residual oils or asphalts with P_2O_5 . The precise mechanism of the asphalt change which occurs is not understood, although the principal effect appears to depend upon polymerization and condensation reactions. It is important to note that the treatment is in no respect an oxidation treatment, and in fact oxidation or the presence of air during the process is to be avoided. Thus, it has been determined that for the purposes of this invention, degradation in asphalt quality results from the presence of air during treatment with the P_2O_5 .

The quantity of P_2O_5 to be employed ranges from about 0.1 to 5 percent by weight. The precise amount of P_2O_5 to be used depends principally upon the properties desired and the starting materials. In all cases treatment with P_2O_5 is preferably continued until equilibrium has been obtained. As the P_2O_5 treatment of asphalt proceeds readily, the time required is reasonably short and does not ordinarily exceed one hour. The temperature of treatment with P_2O_5 is chosen from the range of 300° to 550° F. Again, the precise temperature to be used depends to some extent upon the asphalt characteristics desired and the starting materials.

This treatment of a residual oil or an asphalt with P_2O_5 at 300° to 550° F. in the absence of air or oxygen produces a material change in the properties of the asphalt. In particular, the softening point of the asphalt is raised and the temperature susceptibility of the asphalt is improved to values otherwise unobtainable.

While as indicated, the critical treatment with P_2O_5 depends upon the exclusion of any oxidation effects, it has been found valuable in certain applications to employ an oxidation treatment either before or after, but not simultaneously with the P_2O_5 treatment. One adaptation of the process of this invention therefore depends upon oxidizing an asphalt followed with P_2O_5 treatment in the absence of air; another adaptation of the process utilizes P_2O_5 treatment in the absence of air followed by an oxidation treatment. Both of these adaptations cause a variation in asphalt properties different from those which occur without the oxidation treatment or when the oxidation is applied simultaneously with the P_2O_5 treatment. Furthermore, the oxidation treatment, when applied before or after the P_2O_5 treatment, decreases the amount of P_2O_5 required to attain a particular softening point.

In the figures:

Fig. 1 is a flow plan of a preferred mode for conducting the process; and

Fig. 2 presents photographs of steel panels after rust tests using cutback asphalts of the present invention and of conventional types.

To fully disclose the nature of this invention, reference will be made to the accompanying Fig. 1. The process illustrated in the drawing concerns the preparation of asphalt from a residual oil obtained by distillation of a crude petroleum oil, although it is to be understood that the process illustrated may be applied to asphalts which may be obtained by other methods. The term "residual oils" by definition is employed to identify the liquid or semi-solid residues obtained from the destructive distillation of non-asphaltic petroleum, from the distillation of

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semi-asphaltic and asphaltic petroleum, from the distillation of pressure tar, or from the fluxing of harder residual asphalts with heavy distillates. The term "flux" or flux oil" may be used synonymously with the term "residual oil."

By way of example, in the process illustrated in the drawing, a residual oil is obtained in distillation zone 1 by treatment of a semi-asphaltic or asphaltic petroleum oil introduced into still 1 through line 2. Normally gaseous hydrocarbons may be withdrawn from distillation zone 1 through line 3, while side stream products consisting of gasoline, kerosene, gas oil, lubricating oil, etc., may be withdrawn through lines 4, 5, 6, etc. The residual oil may be removed as the bottoms product from the distillation zone through line 7 and will include substantially all of the asphaltic constituents originally present in the crude oil distilled. The specific gravity of the residual oil at 77° F. will range from about 0.85 to 1.07, and the fusing point of the oil as determined by the ring and ball method will generally fall within the range of 32° to 120° F. This residual oil may be converted to an asphalt of desirable properties by the processing sequence illustrated in the drawing.

As will be brought out, the primary and essential treatment of the residual oil depends upon a catalytic treatment with P_2O_5 in refining zone 10. Use of the two air blowing zones 11 and 12 is optional and for simplicity it may be assumed that zones 11 and 12 are bypassed by the by-pass lines provided. In this case the residual oil withdrawn from distillation zone 1 through line 7 will preferably be passed through a pre-heater 13 and will then be introduced directly into refining zone 10. Sufficient P_2O_5 is introduced to zone 10 through line 14 to provide a weight percent of about 0.1 to 5%. Mechanical agitation or agitation by inert gases such as nitrogen is employed in zone 10 to thoroughly mix the residual oil and P_2O_5 . By suitably preheating the residual oil in preheater 13 and/or by utilizing heating jackets, steam coils, etc., in zone 10, mixing in zone 10 is carried out at a temperature in the range of 300° to 550° F. Care is taken to exclude air or oxygen from zone 10. As a result, with this treatment of P_2O_5 , the softening point of the asphalt is increased and other properties of the asphalt are changed. The ultimate properties obtainable are dependent upon the particular temperature of treatment, the percentage of P_2O_5 employed, and the initial flux oil treated. Prior to reaching equilibrium properties, the time of treatment will also affect the asphalt properties. However, it is important to continue the mixing until no further change in properties occurs. This can be readily determined by periodically determining the softening point, the penetration or other inspections of the asphalt during treatment. In general, about 30 to 90 minutes are to be employed for the P_2O_5 treatment, depending upon starting material, temperature, quantity of change, amount of P_2O_5 , and degree of agitation. The final asphalt product may then be removed from zone 10 through line 15.

As will be demonstrated, in some cases it is preferred to air blow the residual oil or asphalt prior to the P_2O_5 treatment. In this case the residual oil of line 7 is introduced into zone 11 for contact with an oxidation agent such as air. The oxidation treatment may be conducted in any desired manner; for example, by blowing about 30 to 50 cu. ft. of air per minute per ton of asphalt through the residual oil at about 300° to 550° F. temperature. Thereafter the air blown oil may be withdrawn from zone 11 and passed through preheater 13 for treatment with P_2O_5 in zone 10 as described. The final asphalt product is then obtained by withdrawing the asphalt directly from zone 10 through line 15.

Rather than applying the oxidation treatment prior to P_2O_5 treatment, it is sometimes preferred to apply the oxidation treatment after the P_2O_5 treatment. In this case the residual oil of line 7 will by-pass zone 11 and

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will be subjected to P_2O_5 treatment in zone 10. Thereafter the asphalt removed from zone 10 through line 15 will be passed to oxidation zone 12 wherein a treatment similar to that applied in oxidation zone 11 will be employed.

In this case the final asphalt product will be withdrawn from line 17.

To exemplify the operability and utility of the process described, the following examples are presented, showing the properties of asphalts obtainable.

EXAMPLE I

As typical of the asphalts obtained by the conventional reduction of residual oils a Venezuelan crude oil was subjected to vacuum reduction providing an asphalt of the following properties:

Table I.—Vacuum reduced asphalt

Flash point (Cleveland Open Cup).....	° F.	485
Softening point (ring and ball).....	° F.	90
Penetration at 77° F., 100 grams, 5 sec.	$\frac{\text{mm.}}{10}$	300+
Penetration at 32° F., 200 grams, 60 sec.	$\frac{\text{mm.}}{10}$	95
Penetration at 115° F., 50 grams, 5 sec.	$\frac{\text{mm.}}{10}$	300+
Ductility at 77° F., cm.....		125
Furol viscosity at 210° F., seconds.....		345

After an oxidation treatment as provided by air blowing, an asphalt of the character identified in Table I has the following typical properties provided by different degrees of oxidation A, B and C.

Table II.—Oxidized reduced asphalt

	A	B	C
Flash Point (Cleveland Open Cup), ° F.....	530	510	585
Softening Point (Ring and Ball), ° F.....	195	225	291
Penetration at 77° F./100 gms./5 sec.....	29	16	5
Penetration at 32° F./200 gms./60 sec.....	18	11	2
Penetration at 115° F./50 gms./5 sec.....	45	31	6
Ductility at 77° F., cm.....	3.5	2.5	0
P. T. S. $\times 100^*$	1.22	1.14	1.13

*Penetration Temperature Susceptibility = $\frac{\text{Log } 800 - \text{Log Pen. @ } 77^\circ \text{ F.}}{\text{Softening Point } (^\circ \text{ F.}) - 77}$

As indicated by this example, asphalts obtained by the oxidation of residual oils are characterized by high temperature susceptibility. The asphalts are relatively brittle, possess poor ductility and are otherwise unsuitable for such applications as the lining of irrigation canals.

EXAMPLE II

The residual oil identified in Table I was subjected to P_2O_5 treatment in the absence of air at a temperature of 450° F. Various quantities of P_2O_5 , reported in weight percent, were employed. The residual oil and P_2O_5 were stirred in a closed vessel, while passing a flow of nitrogen through the mixture to completely exclude the presence of air. The resulting asphalt possessed the following properties:

Table III.—Residual oil P_2O_5 treated in absence of oxygen

Weight Percent P_2O_5 Treat	2%	3%	4.5%
Flash Point (Cleveland Open Cup), ° F.....	550	535	525
Softening Point (Ring and Ball), ° F.....	192	225	281
Penetration at 77° F./100 gms./5 sec.....	60	55	31
Penetration at 32° F./200 gms./60 sec.....	42	36	27
Penetration at 115° F./50 gms./5 sec.....	110	99	47
Ductility at 77° F., cm.....	6	4	2.5
Penetration Temperature Susceptibility $\times 100$	.978	.787	.673

In considering these data, it is significant that the P_2O_5 treatment in the absence of oxygen resulted in an increase in the softening point of the asphalt in proportion to the amount of P_2O_5 employed. To this extent, the treatment has a similar effect to oxidation as illustrated by Table II. However, unlike oxidation, the P_2O_5 treatment provides asphalts of lower temperature susceptibility and greater ductility at comparable softening points. This is well demonstrated by a comparison of Table II and Table III, showing the properties of three asphalts of approximately the same softening point as obtained by oxidation and P_2O_5 treatment respectively.

To further illustrate the effect of reacting P_2O_5 in the absence of air other data employing asphalts and blends of asphalts from other crude sources were obtained:

Table IV

	Vacuum Reduced Talco Crude 110° F. S. P. 1156 Furol Vis. Sec. at 210° F.	Talco Blend of 21/30 Pen. Straight Reduced Asphalt with Phenol Ex- tract 1079 Furol Vis. Sec. at 210° F.	Talco Blend of 21/30 Pen. Straight Reduced Asphalt with Phenol Ex- tract 836 Furol Vis. Sec. at 210° F.
Percent P_2O_5	1.2	2½	2½
Flash (Cleveland Open Cup), °F.....		525	530
S. P. (Ring and Ball), °F.....	174	187	205
Pen. 77° F./100 gms./5 sec.....	47	57	53
Pen. 32° F./200 gms./60 sec.....	29	32	31
Pen. 115° F./50 gms./5 sec.....	85	101	80
Duct. 77° F., cm.....	5¼	5½	3½
P. T. S. × 100.....	1.27	1.04	.92

In the data shown, the blended asphalts were prepared by utilizing the phenol extract of lubricating oil to provide asphalts of the indicated properties. These data reveal that crude source, composition, viscosity, etc., may influence, but do not prevent the reaction of P_2O_5 in the absence of air with the asphalt. The properties shown above indicate that the catalytically prepared asphalts have lower temperature susceptibility, are more ductile and are better than those that may be produced by blending or vacuum reduction.

EXAMPLE III

As indicated, the treatment of a residual oil or an asphalt with P_2O_5 in accordance with this invention provides asphalts having properties not obtainable by an oxidation treatment. To obtain the full benefits of this invention, therefore, P_2O_5 treatment must be conducted so as to exclude air to eliminate oxidation effects occurring simultaneously with the P_2O_5 treatment. The deleterious effects caused by the presence of oxygen while treating with P_2O_5 are shown by the following data:

Table V

[Talco flux — 110° F. S. P. 1156 Furol vis. at 210° F.]

Percent P_2O_5	No Air Present		Air Present	
	0.7	1.94	0.7	1.96
S. P. (Ring and Ball), °F.....	130	164	127	164
Pen. 77° F./100 gms./5 sec.....	80	60	85	54
Pen. 32° F./200 gms./60 sec.....	28	30	34	27
Duct. 77° F., cm.....	67	10	60	5.4
P. T. S. × 100.....	1.89	1.29	1.95	1.35

As demonstrated by these data, at the same P_2O_5 level and the same S. P. level, when reacting with P_2O_5 in the presence of air the ductility of final product is lower than when air is excluded. Also, the low temperature properties of the final product are improved when air is excluded.

EXAMPLE IV

As demonstrated, oxidation during P_2O_5 treatment is to be avoided to prevent degradation in the properties of the asphalt obtained. However, carefully controlled and relatively mild oxidation may in some cases advantageously be employed prior to the P_2O_5 treatment. This permits a reduction in the amount of P_2O_5 required to obtain an asphalt of a particular softening point, while still providing an asphalt of substantially the same quality. The asphalt obtained is superior to asphalt produced by oxidation alone or produced by P_2O_5 treatment conducted in the presence of air. This process is particularly adapted for improving the properties of an asphalt which has been oxidized.

These principles are illustrated in Table VI below, showing the effect of P_2O_5 treating an asphalt, in the absence of oxygen, after submitting the asphalt to a prior oxidation treatment conducted so as to only partially raise the softening point toward the desired value. The air blowing or oxidation treatment employed consisted of passing 30.5 cu. ft. of air/minute/ton through the asphalt for two hours at 450° F. The subsequent P_2O_5 treatment employed 1½ weight percent P_2O_5 .

Table VI.—Oxidation followed by P_2O_5 treatment

	Venezue- lan Flux	Air Blown	P_2O_5 Treat- ment of Air Blown Flux
Flash Point (Cleveland Open Cup), °F.....	485	540	540
Softening Point (Ring and Ball), °F.....	90	105	198
Pen. 77° F./100 gms./5 sec.....	300+	173	53
Pen. 32° F./200 gms./60 sec.....	95	51	31
Pen. 115° F./50 gms./5 sec.....	300+	300+	91
Duct. 77° F., cm.....		4.5	3.5

EXAMPLE V

It is sometimes desirable to utilize an oxidation treatment as a finishing step after a prior P_2O_5 treatment conducted in the absence of oxygen. This permits an increase in the softening point of the P_2O_5 treated asphalt without causing any substantial degradation in the other properties of the asphalt. Again this process permits obtaining an asphalt of a desired softening point and other properties while minimizing the quantity of P_2O_5 required.

Table VII shows the properties of an asphalt which was subjected to oxidation treatment after a P_2O_5 treat in the absence of oxygen. The initial feed material was a Venezuelan flux which was raised to a softening point of 232° F. by treatment with P_2O_5 in the absence of oxygen. Thereafter this asphalt was oxidized to a final softening point of 250° F. The inspections of this asphalt were as follows:

Table VII.—Oxidation of P_2O_5 treated asphalt

Flash (Cleveland Open Cup).....°F.....	540
Softening point (ring and ball).....°F.....	250
Penetration at 77° F./100 gms./5 sec.....	42
Penetration at 32° F./200 gms./60 sec.....	29
Penetration at 115° F./50 gms./5 sec.....	60
Ductility at 77° F., cm.....	2
P. T. S. × 100.....	0.74

The relatively high ductility and low temperature susceptibility of this high softening point asphalt are significant.

EXAMPLE VI

As brought out by the preceding examples, asphalts of desired properties are obtained by a P_2O_5 treatment in the absence of air. In particular such asphalts are noted

by high softening point, low temperature susceptibility, and high ductility. However, other properties of these asphalts are notable. For example, it has been found that cut-back asphalts prepared by dilution of an asphalt with an agent such as naphtha are resistant to gelation. "Gelling" of an asphalt is notable on storage, causing a substantial increase in the viscosity of the asphalt, making use of the asphalt difficult or impossible. The gelling properties of an asphalt prepared by air blowing a Venezuelan stock and by P_2O_5 treating this stock are shown in the following table:

Table VIII

CUTBACK ASPHALT—VISCOSITY INCREASE
[45% asphalt by wt. 55% naphtha* by wt.]

Storage Time	220/235 Venezuelan Stock Prepared by Air Blowing, Pen. at 32° F.=11	210 S. P. Venezuelan Stock Prepared by P_2O_5 Treatment of Venezuelan Flux, Pen. at 32° F.=39
1 day-----	60,000+ cps. @ 25° C. gelled immediately.	31 cps. @ 25° C.
4 days-----	-----	34 cps. @ 25° C.
11 days-----	-----	37 cps. @ 25° C.
13 days-----	-----	41 cps. @ 25° C.
19 days-----	-----	44 cps. @ 25° C.

*Naphtha A.—Paraffinic-type naphtha containing 3 vol. percent aromatics and having a boiling range of about 240° to 300° F., a refractive index at 20° C. of 1.40894 and a specific gravity at 20° C. of 0.737.

The P_2O_5 treated asphalt was prepared by treating the identified flux with 2.2 wt. percent of P_2O_5 with agitation in the absence of air for 5 hours at 450° F. As shown by these data, the P_2O_5 treated asphalt was not subject to gelation.

It has also been found that the non-gelling asphalt cutback formulations are excellent thin film rust preventives, and may be used to prevent metals from corroding under a wide variety of conditions including direct exposure to the atmosphere and other corrosive elements. Rust preventives formulated from common grades of asphalts having softening points greater than 170° F. in suitable solvents such as various hydrocarbon naphthas do not have the properties which enable the rust preventives to meet the salt spray and weathering requirements of government specifications for this type of compound. Commercial grades of asphalts with softening points less than 170° F. may meet the above requirements but they do not comply with the high temperature performance requirements or other tests normally specified for these formulations.

Products formulated from suitable solvents and asphalts produced by reacting flux stocks with P_2O_5 in the absence of air, meet the salt spray, accelerated weathering and low temperature adhesion tests mentioned above.

The asphalt used in the following tests was prepared by reacting Venezuelan flux asphalt (200–250 SSF @ 210° F.) with P_2O_5 in the absence of air at 450° F. Incremental amounts of P_2O_5 were added until a softening point of 226° F. was attained. At this level, 3.75 weight percent P_2O_5 was contained in the asphalt. This product had the following properties. The data are compared with commercial oxidized products.

	P_2O_5 -Treated Material	Oxidized Asphalts		
		220/235	180/200	160/180
Softening Point (R & B), °F.-----	226	229	183	171
Penetration 77/100 g./5"-----	45	21	27	32
Penetration 32/200 g./60"-----	28	13	23	21
Ductility @ 77° F., cm.-----	2.8	2.3	3.6	3.6

The above P_2O_5 -treated asphalt and the commercial oxidized asphalts were cut back with a Naphtha B having a boiling range of 320° to 400° F., an aromatic content of about 17 volume percent, a specific gravity at 20° C. of 0.785 and a refractive index at 20° C. of 1.43911. The gelling characteristics of the cutback compositions are shown below:

Asphalt in Naphtha-----	P_2O_5 -Treated Material	Oxidized Asphalt	
		220/235	180/200
15 Asphalt in Cutback, wt. percent-----	60	60	50
Viscosity at 25° C. cps:-----			
After 1 day-----	279	Gelled in 14 days.	270
After 5 days-----	-----		510
After 7 days-----	372		510
After 11 days-----	-----		610
After 14 days-----	392	-----	-----

The data show that when the P_2O_5 -treated asphalt is cut back with 40% solvent, the product did not gel and the viscosity level and increase in viscosity were acceptable. With conventionally produced asphalts having a 220/235° F. softening point, the cutback containing 40% solvent gelled in 14 days. With the 180/200° F. softening point asphalt, the viscosity level was too high and the gelling tendency too pronounced when using 40% solvent; therefore, the viscosity level was lowered by adding more solvent (50%) as shown. Even with 50% solvent, this cutback asphalt increased in viscosity more rapidly than the P_2O_5 -treated asphalt containing 40% solvent, even though the two compositions had about the same initial viscosity.

It is known that the gelling tendency of asphalt solvent cutbacks can be reduced by increasing the aromatic content of the solvent and/or reducing the asphalt concentration in the cutback. Also it is known that the performance of solvent deposited films depends on the coating thickness, which, in turn, is dependent upon the viscosity of the cutback and evaporating characteristics of the solvent. Therefore, since conventionally produced asphalts gel excessively and result in products having relatively high viscosities, in order to maintain uniform film thickness for performance evaluation, it is necessary to increase the aromaticity of the solvent and reduce the concentration of asphalt in the cutback.

In another example, the P_2O_5 -treated asphalt described above was cut back with Naphtha B boiling in the range of 320–400° F. and having an aromatic content of about 17 volume percent. The conventional asphalts were cut back with Naphtha C boiling in about the same range (310–365° F.) but having an aromatic content of 98 volume percent (determined by the Philadelphia Club method). This was necessary in order to obtain the required film thickness. The actual asphalt concentrations used, film thicknesses obtained, and solvent characteristics are shown below.

Asphalt Grade Used in Cutback-----	220/235 Asphalt Produced by P_2O_5 Treatment	Oxidized Asphalt		
		220/235	180/200	160/180
65 Asphalt Concentration in Cutback, Wt. Percent-----	60	43.4	57.3	62
Actual Film Thickness, mils-----	2.5	2.5	2.9	3.0
70 Naphtha Boiling Range, °F.-----	320–400	310–365	310–365	310–365
Refractive Index of Naphtha, 20/0-----	1.439	1.495	1.495	1.495
Aromatic Content of Naphtha, Vol. Percent-----	Appx. 17	Appx. 98	Appx. 98	Appx. 98

These cutbacks were then tested as shown in the following table:

Asphalt Grade	Softening Point (°F.)	Performance of Solvent Deposited Coatings ¹		
		Salt Spray Test ²	50 Day Weatherometer Test ³	Low Temperature Adhesion ⁴
160/180 oxidized.....	171	Pass.....	Fail.....	Pass.
180/200 oxidized.....	183	Pass.....	Fail.....	Pass.
220/235 oxidized.....	229	Borderline..	Fail.....	Pass.
220/235 produced by P ₂ O ₅ Treatment.	226	Pass.....	Pass.....	Pass.

¹ According to Specification MIL-C-16173; all coatings of 3 mil maximum thickness. "Pass" for salt spray and weathering tests represents no rusting of coated steel test panels.

² Specification VV-L-791, method 400.1; 20% sodium chloride solution sprayed on coated steel specimens for 18 days at 95° F. and 95-100% relative humidity.

³ Specification TT-P-141, method 615.1; coated steel panels subjected to ultraviolet radiation with and without direct water spray for 50 days.

⁴ Specifications AXS-673, MIL-C-6708, MIL-C-972 and MIL-C-16173; measure of flaking of coating @ 0°F.

The outstanding superiority of the P₂O₅-treated material in weathering properties is shown in that:

(1) The P₂O₅-treated asphalt film is superior to the film produced when the asphalt used in formulating the cutback is normal air-blown asphalt having the same softening point.

(2) The P₂O₅-treated asphalt film is superior to the films produced from cutbacks made from normal air-blown asphalts of varying softening point. As the softening point is increased, the weathering resistance of the air-blown materials is decreased.

However, even at a softening point of 226° F., the film deposited by the P₂O₅-produced asphalt cutback provided a protective coating which resisted the effects of the 50 day accelerated weathering test (test (3) above), as shown in the detailed data below:

Asphalt Coating	Results of 50 Day Weatherometer Tests		
	Minimum Coating Life (Days to External Appearance of Rust)	Appearance of Coating	Condition of Coated Steel Panel After 50 Days
160/180 Oxidized.....	28.....	Many cracks.	Covered with rust spots.
180/200 Oxidized.....	24.....	do.....	Do.
220/235 Oxidized.....	22.....	do.....	Heavy rust and spots.
220/235 Produced by P ₂ O ₅ Treatment.	No rust @ 50 days.	No cracks...	No rust.

Reproductions of the photographs of steel panels from these tests are shown in Fig. 2.

The solvent in which the asphalt of the present invention is dissolved may be any suitable light hydrocarbon oils, such as volatile mineral oils. These solvents include kerosene, toluene, petroleum naphthas, such as aromatic solvent fractions and paraffin solvent fractions, benzene, petrolene, and the like. The amount of asphalt in the compositions may vary over a rather wide range such as about 15 to 80% by weight based on the total composition. Generally, in the range of about 20 to 65% of asphalt is preferred. The cutback compositions may be used as paints, coating compositions, and the like on metal

surfaces, etc., where improved resistance to corrosion and weathering is desired.

The weathering and rust preventive compositions of the present invention may also be further improved by the addition thereto of relatively small amounts of high molecular weight hydrocarbon polymeric substances, such as polymers and copolymers of olefins and diolefins. Among the polymers and copolymers that may be used are copolymers of styrene and isobutylene as described in U. S. 2,274,749, copolymers of butadiene and styrene and copolymers of isobutylene and butadiene or isoprene. Various other olefins useful in polymerization and copolymerization include the mono-olefins, particularly the iso-olefins, having 4 to 8 carbon atoms such as isobutylene, isopentene and the higher diolefins. Polyisobutylene is particularly useful. Various other cyclic olefins include indene, alphasethyl styrene, etc.

These polymers are usually formed at low temperatures in the presence of a Friedel-Crafts or peroxide catalyst and a solvent. The preferred polymeric materials will have average molecular weights above about 6,000, i. e., in the range of about 10,000 to 300,000. Such materials are well known to the art.

The asphalt cutback compositions may contain in the range of about 0.1 to 15% by weight of the polymeric material, although amounts such as 0.2 to 3% by weight will generally give the desired improvement.

It is often desirable to incorporate in the asphalt along with the polymeric material small amounts, such as from 1 to 20 wt. percent, of mineral oil which boils above 800° F., for example, bright stock, solvent extracts of high boiling lubricating oils, etc., in order to improve further the ductility and low temperature susceptibility of the asphalt composition.

What is claimed is:

1. A non-gelling composition consisting essentially of a volatile hydrocarbon oil and in the range of about 15 to 80% by weight, based on the total composition, of an asphalt prepared by mixing a residual oil and about 0.1 to 5 weight percent P₂O₅ in the absence of free oxygen at a temperature in the range of about 300° to 550° F.

2. A composition as in claim 1 wherein said composition includes from about 0.1 percent to about 15 percent by weight of hydrocarbon polymeric compounds having an average molecular weight above about 6000.

3. A non-gelling rust preventive composition consisting essentially of a naphtha and in the range of about 20 to 65% by weight of an asphalt prepared by agitating an asphalt and about 0.1 to 5 weight percent P₂O₅ in the absence of free oxygen at a temperature in the range of about 300° to 550° F.

4. A composition as in claim 2 wherein said hydrocarbon polymeric compounds are selected from the group consisting of polymers and copolymers of iso-olefins having 4 to 8 carbon atoms.

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