

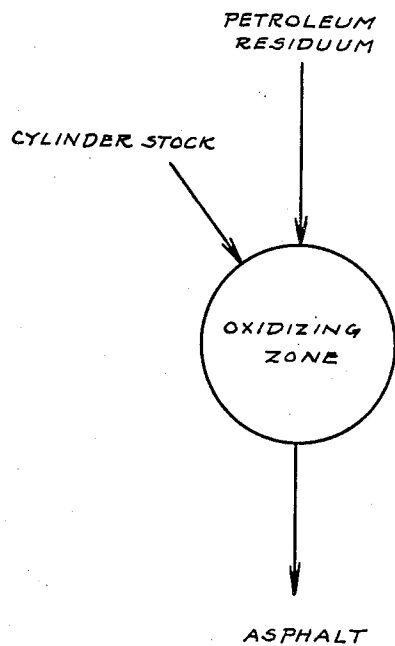
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PROCESS OF MAKING ASPHALT

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PROCESS OF MAKING ASPHALT

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The manufacture of asphalt has been based on Mexican or like purely asphalt-type petroleum, and it has been generally supposed that roofing asphalt and the like could not be made where there were oily constituents present, and that such a material as Mid-Continent petroleum was too oily to be operable for asphalt. In such manufacture moreover, the ratio of penetration and melting point has been circumscribed to the inherent limitations determined by the source, and a desirable freedom of control of these factors has been lacking. In accordance with the present invention however, it now becomes possible to regulate penetration and melting point as desired. Also to make various grades of asphalt from Mid-Continent or other semi-asphaltic oils.

To the accomplishment of the foregoing and related ends, the invention, then, comprises the features hereinafter fully described, and particularly pointed out in the claims, the following description setting forth in detail certain illustrative embodiments of the invention, these being indicative however of but a few of the various ways in which the principle of the invention may be employed.

In the annexed drawing:

The sole figure is a schematic showing on the order of a flow sheet illustrating a mode of carrying out the invention.

In its general aspects, the invention contemplates oxidation treating of cylinder oil stock with a distillation residuum, and selecting the proportions in view of the penetration and melting point to be obtained. By "cylinder stock" is meant those petroleum portions which are a viscous cut distilled from the crude petroleum and which contain little or no asphaltenes. Such a stock is generally used for making lubricating oil. The petroleum residuum or bottoms left from distillation, for instance from a Mid-Continent stock, may be that produced in a vacuum or other type of still, or most usually, a vacuum pipe still. In general, the amount of cylinder stock employed with the distillation residuum may be from 15 to 85 per cent, depending upon the particular character of the product wanted.

The mixture of cylinder stock and distillation residuum is subjected to oxidation treatment, most conveniently for example by blowing air therethrough, while maintaining the temperature in a range of about 375–550° F., and sampling, and shutting down at the desired melting point. The lower amounts of cylinder stock yield an asphalt having a relatively low melting point for a given penetration. This melting point is higher however than it would be for a straight residuum oxidized. For instance, if it is desired to produce an asphalt having a penetration of

50–60 at 77° F. and a melting point of 140°–150° F., a mixture of 20 per cent cylinder stock and 80 per cent residuum is oxidized. If a high melting point is desired, the proportion of cylinder stock is increased. Thus an asphalt of 50–60 penetration and 150–160° melting point can be had from 30 per cent cylinder stock and 70 per cent residuum. By contrast, the processing of the straight 180–200 penetration (at 77° F.) residuum when oxidized to 50–60 penetration (at 77° F.) would yield an asphalt having a 130–140° melting point. For instance also, a 50–60 penetration asphalt having a melting point of about 214° F. may be obtained from a mixture containing 45–50 per cent of cylinder stock. And in general, the ratio of melting point to penetration may thus be controlled closely in accordance with the desired properties of products in view. The susceptibility to temperature change is thus also closely regulable, and whereas for example, roofing asphalts formerly made had an unfortunate tendency to flow under sun-exposure on a hot day, this can now be readily obviated by employment of a suitable amount of cylinder stock, and the product may be gauged to a satisfactory melting point and satisfactory penetration at 32°, 77°, and 115°. An asphalt particularly desirable from the standpoint of flexibility is readily obtainable.

Blends of cylinder stock and distillation residuum or bottoms and cracking coil tar or like cracking residue fluxes may be made up, and be oxidation-treated to the desired melting point, with advantage. Cracking coil tar or the like, as well known, is the residue left from the cracking or conversion of petroleum, most usually gas oil, in the lower boiling products, gasoline, etc. The amount of such residuum from a cracking-operation added to the material to be oxidized may be 10–40 per cent.

Some illustrative examples are:—

I. About 20 per cent of cylinder stock, in the form of the heaviest cut taken from the side of the tower of a vacuum pipe still and having a gravity of 19° to 21° A. P. I., and a flash of 550° F. is mixed with about 80 per cent pipe still bottoms and is air-blown in a suitable still or the like, with maintenance of the temperature at around 450° to 550° for about 48 hours, depending upon the air-rate and temperature. A product is attained having a melting point of 170° F., a penetration of 31 at 77° F., and ductility of 5 at 77° F., and a flash point of 560° F. By oxidation treating similarly, other combinations of melting point and penetration may be realized, as for instance melting point 185° F., penetration 27 at 77° F. and ductility 4; melting point 202° F., penetration 20 at 77° F. and ductility 2½; melting point 227° F., penetration 12 at 77° F., and duc-

tility 2½. Such asphalt is particularly desirable for usages as roofing and the like.

II. In similar manner, by oxidizing a mixture of 25 per cent cylinder stock and 75 per cent pipe still bottoms, products having a melting point of 172° F., penetration 30 at 77° F., and ductility 3½ at 77° F. may be had. Or by carrying the oxidation treatment further, a melting point for instance of 186° F., penetration 25 at 77° F., and ductility 3½; or a melting point of 228° F., penetration 14 at 77° F., and ductility 2½.

III. Similarly, with a mixture of 25 per cent cylinder stock and 75 per cent distillation residuum, as from a pipe still, an asphalt may be made up having a melting point 183° F., penetration 28 at 77° F., ductility 3½ at 77° F., a flow test of 1½ inches at 175° F., and a plus bending test. (The flow test is performed by molding a test-piece of the asphalt ¾ inch in diameter and ¾ inch long, and this is placed on a plate inclined at an angle of 45° in an oven for two hours at a temperature of 175° F. The bending test is performed by molding a test-piece in a block-strip 1" x 1" x 12" long, and immersing the test-piece in a water bath at 50° for 1½ hours, removing and bending the piece quickly in an arc so that the two ends meet without breaking the bar.) If it is desired to further reduce the tendency to flow, an additional 5 to 10 per cent of cylinder stock may be added to the oxidized asphalt, which is then re-oxidized to 180-200 melting point, tests being taken on the oxidizing tank or still to determine when the oxidation shall be terminated to produce the desired melting point and penetration characteristics. The asphalt thus produced will have a greater penetration for a given melting point than the asphalt produced by oxidizing the original mixture and will be less susceptible to change in consistency with temperature change, and will have less tendency to flow. Similar results may be attained if the proportion of cylinder stock in the original mixture is increased. It will be seen however, that by the present invention, the range of properties can be controlled to a nicety, even after oxidation has been carried on.

IV. A mixture of 85 per cent cylinder stock and 15 per cent distillation residuum, similarly, may be oxidation treated to produce an asphalt having a melting point of 189° F. and a penetration of 111 at 77° F.

The mixtures outlined above are based on a residuum of 180-200 penetration at 77° F., and cylinder stock of 190-210 viscosity Saybolt at 210° F. If the residuum is softer in penetration a smaller amount of cylinder stock will be required, due to the fact that the residuum is made softer by the presence of a larger percentage of non-bituminous oils. If a harder residuum is used, a correspondingly larger proportion of cylinder stock will be required.

V. A mixture of 20 per cent cylinder stock and 80 per cent distillation residuum is oxidation-treated to a melting point of 143½° F. and a penetration 51 (at 77° F.), 10 per cent of reduced cracking coil tar (prepared by distilling cracking coil tar down about 50 per cent to 460° F. flash) is added, and the material further oxidation-treated to attain a product having a melting point 142½° F., penetration 50 at 77° F., 20 at 32° F., 127 at 115° F., and ductility 10 at 77° F., and specific gravity 1.006 at 77° F.

VI. A mixture of 20 per cent cylinder stock, 65 per cent distillation residuum and 15 per cent reduced cracking coil tar is oxidation-treated to

attain a product having a melting point 142° F., penetration 53 at 77° F., 23 at 32° F., 153 at 115° F., ductility 14, and specific gravity 1.003 at 77°.

A mixture of 20 per cent cylinder stock and 80 per cent distillation residuum as in Example I, may be oxidized to yield products ranging from 105 melting point and 300 penetration (at 77° F.) to 325 m. p. and 2 penetration (at 77° F.). Similarly, a mixture of 25 per cent of cylinder stock and 75 per cent distillation residuum may be made to yield products ranging from 112 m. p. and 300 penetration (at 77° F.) to 325 m. p. and 3 penetration (at 77° F.), and mixtures for instance of 85 per cent cylinder stock and 15 per cent distillation residuum may be made to yield products ranging from 140 m. p. and 300 penetration (at 77° F.) to 325 m. p. and 36 penetration (at 77° F.).

By prolonging the oxidation, products may be obtained having melting points from 350 to 375° F. The flash point of the final product can also be controlled by the cylinder stock. For instance, by employing a cylinder stock of flash point of 550° F. with the distillation residuum, products of final flash point at 560° F. may be obtained. Again, with a cylinder stock for instance of flash point of 570° F. with the distillation residuum, products of final flash point 580° F. may be had. Thus in general, flash points of products can be determined by the cylinder stock employed and this aspect of the invention makes it possible to control the flash point of products from even such residuum as Mexican or like heavy asphalt residuum, by admixing cylinder stock of desired controlling flash point, and then oxidizing. In this manner, products of much higher flash point than have heretofore been obtainable on such asphaltic petroleum, now become possible.

Other modes of applying the principle of the invention may be employed, change being made as regards the details described, provided the features stated in any of the following claims, or the equivalent of such, be employed.

I therefore particularly point out and distinctly claim as my invention:

1. A process of making asphalt, which comprises mixing a distillation residuum substantially freed from lubricating hydrocarbons and an oily distillate cylinder stock having a viscosity of at least 190 at 210° F. Saybolt, and oxidizing the mixture.

2. A process of making asphalt, which comprises mixing a distillation residuum substantially freed from lubricating hydrocarbons and a high viscosity oily distillate cylinder stock, oxidizing the mixture, adding an additional portion of a high viscosity cylinder stock to control the relationship of melting point to penetration, and again oxidizing.

3. A process of making asphalt, which comprises blending an oily distillate cylinder stock, having a flash point not less than 550° F., non-cracking distillation residuum having a flash point of approximately 650° F., and residuum from a cracking operation, and oxidizing.

4. A process of making asphalt, which comprises mixing with a non-cracking distillation residuum, which is substantially free from lubricating fractions, selected proportions of lubricating oil distillate stock and residuum from a cracking operation, and oxidizing.

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