April 3, 1934. E. W. GARD ET AL 1,953,345

PROCESS FOR THE MANUFACTURE OF ASPHALTIC PRODUCTS

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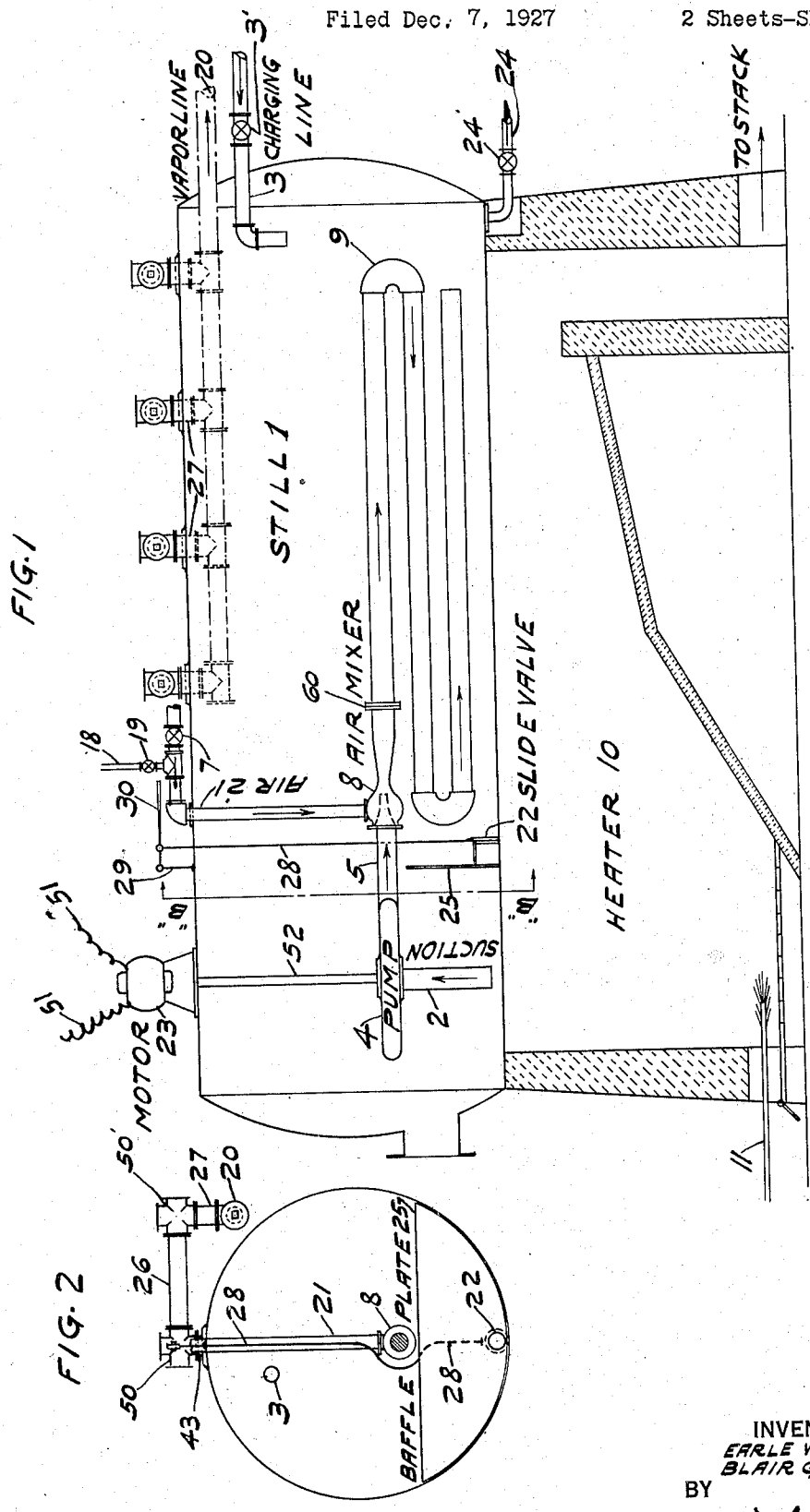


FIG. 1

FIG. 2

INVENTORS
EARLE W. GARD &
BLAIR G. ALDRIDGE.

BY
Marvin L. Chappell
ATTORNEY

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FIG. 4

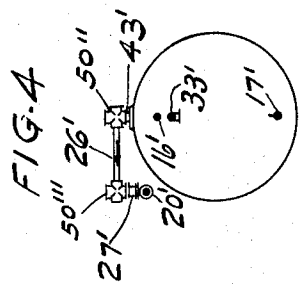


FIG. 3

INVENTORS
EARLE W. GARD &
BLAIR G. ALDRIDGE.
BY
Marvin L. Chappell
ATTORNEY

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PROCESS FOR THE MANUFACTURE OF ASPHALTIC PRODUCTS

Earle W. Gard, Palos Verdes Estates, and Blair G. Aldridge, Watson, Calif.

Application December 7, 1927, Serial No. 238,427

25 Claims. (Cl. 196—74)

This invention relates to a process for treating petroleum oils and has particular reference to a method of producing high grade asphalt products suitable for any of the various uses in the art.

An object of the invention is to provide a simple, efficient and economical process by which petroleum oils may be converted into asphaltic products, which will have a relatively high melting point, flash and penetration, and at the same time will be of a suitable ductility.

Another object of the invention is to provide a simple and efficient process for manufacturing asphaltic products, in which the various characteristics of ductility, high melting point, flash and penetration required by the different branches of the individual art may be controlled, separately or in combination.

Another object of the invention is to provide a method by which petroleum oil may be uniformly oxidized to the required degree at a minimum consumption of time and with a minimum consumption of the oxidizing agent.

Heretofore, difficulty has been experienced in providing an asphaltic cement entirely suitable for the manufacture of roofing compounds, battery sealing products and the like, in which relatively high flash and high melting point materials are required and one which will have suitable ductility at 77 degrees F. We have discovered that this is due to producing products which are not uniformly oxidized, in which some part of the asphaltic cement has been overoxidized, while other parts have not been sufficiently oxidized to give the required melting point, flash, ductility and penetration.

We have discovered that these difficulties may be overcome by employing a smaller quantity of air and a more uniform and intimate contact between the asphaltic oil and the air, in which case the necessary oxidizing action is uniformly effected in a much shorter time and without the formation of brittle carbonaceous material.

In the conventional method of oxidizing asphaltic oils for the production of oxidized asphalt, air is introduced into a cylindrical still containing the asphaltic oil through a spray pipe and at the same time the asphaltic oil contained in the still is heated to a temperature sufficient to obtain the necessary oxidizing action, approximately 450 to 550 degrees F. By such methods the air is not in intimate contact with the asphaltic oil as by our invention, passing upward in large globules or bubbles through the depth of the oil in the still, whereby only a small percentage of the air comes into intimate contact with the oil. On account of not producing an intimate contact of the air with the oil, excessive quantities of air must be employed to obtain the desired results and a uniform product cannot be obtained. Also a uniform temperature is difficult to maintain since the oxidizing action is exothermic and the added heat may overheat the asphaltic oil to a point of cracking or other decomposition reaction, thereby rendering the product more brittle or less ductile than it would be if a uniform oxidation could have been effected. By our invention the rate of oxidation is controlled by the introduction of the air whereby a uniform temperature may be maintained without overheating the oil.

We have also discovered that when excessive quantities of air, such as are employed by the conventional cylindrical batch still method, a higher percentage of distillation products is produced, which by our process are conserved and oxidized to desirable asphaltic products.

For example, by the conventional cylindrical batch still method the time required to convert a batch of asphaltic oil to an oxidized product suitable for the manufacture of roofing material requires approximately 24 hours or more, while by our invention the reaction is accomplished in about one half the time and the total quantity of air employed is approximately only 50 per cent of the air required by the conventional batch still method.

In the manufacture of oxidized asphalt suitable for roofing material and the like by our invention, we may obtain a product having a flash up to 260 degrees F. or higher, and at the same time maintain a ductility of 1 cm. or higher. The flash of such a product may range from 430 to 460 degrees F. or a little higher and will have an approximate penetration at 77° F. of 15 to 18.

We have found it advantageous to circulate the oil at a relatively high speed through a jet mixer or other types of mixers known in the art for commingling a liquid and a gas into which a regulated quantity of air is admitted, by which procedure we are enabled to produce a superior asphaltic cement, since the oxidation is not as severe, with the result that less carbonaceous material is produced and a greater ductility and penetration is obtained at a given flash and melting point.

With the foregoing preliminary explanation, the preferred embodiment of our invention will now be more fully explained by reference to the accompanying drawings which are diagrammatical representations of the preferred apparatuses

for carrying out the operations of the present invention.

In Figure 1, 1 represents generally a still or container for holding the asphaltic oil to be oxidized, which is heated by means of furnace 10 directly underneath the still 1. The heater 10 is provided with a burner 11 which leads to a source of fuel not shown. The pipe coil 9 inside of the still 1 is connected to an air mixer 8 by the flange 60. The air pipe 21 controlled by the valve 7 connects the air mixer 8 to a source of air not shown. A steam pipe 18 controlled by the valve 19 is connected to the air pipe 21 and leads to a source of steam not shown. A pipe 5 connects the air mixer 8 to a pump 4. A pipe 2 is connected to the suction of the pump 4. A drive shaft 52 connects the pump 4 to the motor 23. The motor 23 is operated by an electric current coming through the wires 51 which lead to a source of electric power not shown. A pipe 24 controlled by the valve 24' is connected to the bottom of the still 1 and leads to a storage not shown. A charging line 3 controlled by the valve 3' is connected to the still 1 near the top and leads to a source of asphaltic oil not shown. A vapor pipe 20 is connected to the still 1 at the top by the pipe fittings 27 and leads to a condenser not shown. 25 designates a baffle plate attached to the shell inside of the still 1 with a slide valve 22 which is operated by the lever 30 through the extension rod 28 and the fulcrum 29.

Figure 2 is a cross-section of the still 1 through that portion designated by the line "B"—"B". 25 is a baffle plate and 22 is a slide valve in the baffle plate which can be opened or closed by means of the extension rod 28. A pipe 21 connects the air mixer 8 to a source of air not shown. The pipe 26 connects the cross fitting 50 to the cross fitting 50'. The cross fitting 50' is connected to the vapor line 20 by the pipe 27. The cross fitting 50 is connected to the still 1 by a pipe 43.

In Figure 3, 1' represents generally a still or chamber for holding a bulk supply of oil, which may be heated by means of a furnace directly underneath the still 1', or the heat may be entirely supplied to the oil on its passage through the heating coil 9'. A pipe 2' connects the still 1' at the bottom to the inlet side of the pump 4' controlled by the valve 40'. A pipe 3'' controlled by the valve 41' is connected to the pipe 2' and leads to a source of asphaltic oil not shown. A pipe 5' controlled by the valve 6' connects the discharge side of the pump 4' to the air mixer 8', a pipe 21' controlled by the valve 7' is connected to the air mixer 8'. The pipe 21' leads to a source of air not shown. a pipe 32' connects the air mixer 8' to heating coil 9'. The pipe 18' controlled by the valve 19' is connected to the pipe 32' and leads to a source of steam not shown. The heating coil 9' is interposed in the furnace 10' and is provided with a burner 11' which leads to a source of fuel not shown. A pipe 33' controlled by the valve 33'' connects the heating coil 9' at the bottom to the still 1', passing through the shell of the still 1' near the top. A pipe 12' controlled by the valve 31' connects the pipe 5' to a storage tank 13'. A pipe 16' controlled by the valve 15' connects the steam pipe 14' to the still 1' near the top. The pipe 14' controlled by the valve 34' is connected to the spray pipe 17' which is in the still 1' near the bottom. A pipe 35' controlled by the valve 36' is connected to the steam pipe 14'. Steam pipe 14' leads to a source of steam not shown. A vapor pipe 20' is connected to the still 1' at the top by the pipe fittings 27'.

In Figure 4, which is a cross-section of the still 1' showing the connection of the vapor pipe 20' to the still 1' through the line "A"—"A", 50'' is a cross-fitting connected to the top of the still by means of a pipe 43'. The pipe 26' connects the cross fitting 50'' to the cross-fitting 50'''. The pipe 27' connects the cross-fitting 50''' to the vapor pipe 20' by means of a T. 16' is a steam line which enters the still 1' at the top and 33' is the pipe coming from the heater coil 9'. 17' is the steam spray pipe which runs along near the bottom of the still 1'. The pipes 33', 32', 2', 5', and air mixer 8' are preferably covered with a suitable insulation material to prevent any substantial loss of heat, or they may be steam jacketed.

The preferred process as carried out in the apparatus described in Figure 1 is as follows:

Petroleum oil, which may be any crude petroleum oil, or preferably a residuum derived from the distillation of an asphalt or mixed base crude petroleum oil, and preferably ranging in gravity from 10 or below to 16 degrees Bé., is charged into still 1 to a predetermined level coming through the charging line 3 regulated by the valve 3'. The charging line 3 leads to a source of asphaltic oil not shown. When still 1 contains a sufficient charge of the asphaltic oil stock, valve 3' is closed, heat is applied to the furnace 10 by means of the burner 11 and regulated so that the oil will attain a temperature of approximately 450 to 550 degrees F., or whatever temperature is desired for that particular run. During the heating of the oil to the desired oxidizing temperature in the still 1, the oil is circulated through pipe coil 9 by operating pump 4 which takes suction through the pipe 2 and discharges the oil through the pipe 5 and into the air mixer 8 from where it passes through the pipe coil 9 and then out into the still at the opposite end. The still 1 is provided with a baffle plate 25 so as to prevent the circulating oil from returning along the bottom of the still 1 to the suction pipe 2 of the pump 4. When the circulating oil has attained a suitable temperature a regulated stream of air is introduced through the air pipe 21 by opening valve 7, the pipe 21 leading to a source of compressed air not shown. The air passes from the pipe 21 into the air mixer 8 where it is intimately commingled with the oil to be oxidized. The commingled air and oil pass through the heater coil 9 and then into the still 1. The commingled oil and air mixture after being discharged into the still 1 flows in a generally upward direction and the air separates from the oil passing out through the vapor line 20 along with the products of distillation and the oil flows back over the top of the baffle plate 25 to the suction end of the pump 4 where it is again passed through the pipe coil 9 along with commingled air, in like manner, as before.

The circulation of the oil at an elevated oxidizing temperature and the introduction of air is maintained for a period of time necessary to obtain the desired product, such time depending upon the quality of the oil employed, the product desired, and the amount of air introduced.

In producing certain varieties of oxidized asphalt it may be found advantageous to introduce steam along with the air which is carried out by opening valve 19 sufficiently to admit the desired amount of steam from pipe 18. The introduction of steam decreases the time of reaction and reduces the tendency of the oil to crack, thereby yielding a product of superior quality.

During the oxidation of the asphalt oil with air,

or steam and air, the air in conjunction with the products of distillation passes from the still 1 into the vapor line 20. The vapor line 20 leads to a condenser not shown where the oil products are condensed and separated from the residual air.

When the petroleum oil residuum has been oxidized to the required degree it is discharged from the still 1 through the pipe 24 to a storage not shown by operating the valve 24.

In some cases where the oxidized asphalt is not at a sufficiently high flash after the oxidizing reaction, the flash may be further raised by steaming the charge for a period of time. This is accomplished by introducing steam into the still 1 through the pipe 18 by the proper regulation of valve 19 and closing the valve 7 so as to permit the introduction of steam only into the mixer 8. Also in certain cases it is found advantageous to raise the flash of the asphaltic oil before the oxidizing operations.

The operation of this invention for the production of an oxidized asphalt with the desired flash, melting point, ductility and penetration may be conducted in various ways, for example, during the oxidation process both steam and air may be admitted into the mixer 8, or steam and air may be intermittently introduced into the mixer, the regulation of which depends upon the stock under treatment and the product desired.

In the Figures 3 and 4 there is also shown a complete apparatus for carrying out the objects of the present invention in which the air, steam and air, or steam alone, may be intimately mixed in any desired amounts with the oil during the oxidizing process by means of a jet mixer and a pipe coil stationed outside of the still. The operation of this apparatus is similar to the operation of the apparatus described, except that the commingled air and oil, steam and oil, or steam, air and oil may be heated during the passage through the pipe coil.

While the process herein described is well adapted for carrying out the objects of the present invention, it is to be understood that various modifications and changes may be made without departing from the spirit of the invention and the invention includes all such modifications and changes as come within the scope of the appended claims.

What we claim is:

1. A process of forming oxidized asphalt comprising intially heating a bulk supply of asphaltic oil to an oxidizing temperature by the application of heat to the bulk supply maintained in a still and maintaining the heated bulk supply of asphaltic oil at said oxidizing temperature, circulating the oil commingled with air through a pipe coil within the still and back into the bulk supply, and continuously permitting the residual air and products of distillation to separate from the oil and pass out of the system.

2. A process of forming oxidized asphalt comprising maintaining a buk supply of asphaltic oil is a still, maintaining a substantial constant elevated oxidizing temperature by the application of heat to the bottom of said still, causing the oil to circulate through a pipe coil in the still and back to the bulk supply, continuously introducing air into the circulating body of oil in the pipe coil, and continuously separating the residual aid and products of distillation from the oil.

3. A process of forming oxidized asphalt comprising maintaining a bulk supply of asphaltic oil in a still at a substantial elevated oxidizing temperature, substantially below the cracking temperature by the application of heat to the bottom of said still, causing the oil to circulate through a pipe coil in the said still and back to the bulk supply, continuously introducing and commingling in a mixer a regulated quantity of air with the circulating oil in the pipe coil and continuously separating the residual air with the products of distillation from the oil, continuing said circulation, heating and introduction of air until the oil has been oxidized to the required degree.

4. A process of forming oxidized asphalt comprising maintaining a bulk supply of asphaltic oil in a still at a temperature of approximately 450 to 550 degrees F. by the application of heat to the bottom of the still, causing the oil to commingle in a mixer with regulated quantities of air and circulate through a pipe coil in the still and back to the bulk supply in the still, continuously separating and withdrawing the residual air and products of distillation from the still, continuing said circulating, heating, introduction of air, withdrawal of residual air and products of distillation from the still until the asphaltc oil has been oxidized to the required degree.

5. A process of forming oxidized asphalt comprising maintaining a bulk supply of asphaltc oil in a container, maintaining an elevated oxidizing temperature of the oil by the application of heat to the bottom of the container, causing the oil to continuously commingle in a mixer with regulated quantities of air and circulate through a pipe coil inside of the container and back to the bulk supply in the container, continuously separating the air and products of distillation from the oil, continuing the circulation and oxidation of the oil until an oxidized asphalt of the required specification has been obtained.

6. A process of forming oxidized asphalt comprising maintaining a bulk supply of oil in a container, maintaining an elevated oxidizing temperature by the application of heat to the bottom of the container, circulating the oil commingled with air and steam through a pipe coil in the said container and back to the bulk supply, continuing the introduction of regulated quantities of steam and air into the pipe coil and continuously withdrawing the residual air, steam and products of distillation from the container until a product of the desired melting point, flash, ductility and penetration has been obtained.

7. A method of producing oxidized asphalt which comprises maintaining a body of oil in a still circulating a stream of said oil through an elongated conduit back to the body of oil in the still introducing air into the circulating stream of oil and withdrawing vapors and gases from the still and removing oxidized asphalt from said still.

8. A method of producing oxidized asphalt which comprises maintaining a body of oil in a still circulating a stream of said oil through an elongated conduit back to the body of oil in the still introducing air into the oil circulating in the elongated conduit and withdrawing vapors and gases from said still and removing oxidized asphalt from said still.

9. A method of producing oxidized asphalt which comprises maintaining a body of oil in a still circulating a stream of said oil through an elongated conduit back to the body of oil in the still introducing air into the circulating stream of oil and heating said oil in said elongated conduit and withdrawing vapors and gases from said still and removing oxidized asphalt from said still.

10. A method of producing oxidized asphalt which comprises maintaining a body of oil in a

still circulating a stream of said oil through an elongated conduit back to the body of oil in the still introducing air into the oil circulating in the elongated conduit and heating said oil in said conduit and withdrawing vapors and gases from said still and removing oxidized asphalt from said still.

11. A process of oxidizing asphaltic oil which comprises maintaining a bulk supply of asphaltic oil at an elevated oxidizing temperature in a still, commingling the oil in a jet mixer within the still with air, causing the commingled oil and air to pass through a pipe coil within the still and regulating the supply of air and maintaining the oil in the still at an elevated oxidizing temperature sufficient to oxidize said oil to form asphalt.

12. A process of forming oxidized asphalt comprising maintaining a heated bulk supply of asphaltic oils in a chamber at an elevated oxidizing temperature, continuously circulating the oil from the bulk supply to and through a heating coil and back to the bulk supply, continuously introducing a regulated stream of air into the circulating oil before the oil enters the heating coil, commingling the air with the oil during its passage through the heating coil and continuously permitting the residual air to separate from the oil in the chamber and pass out of the system.

13. A process of forming oxidized asphalt comprising maintaining a bulk supply of asphaltic oil in a chamber, maintaining a substantially constant elevated oxidizing temperature therein by continuously circulating the oil from the bulk supply to and through a heating coil and back to the bulk supply, continuously introducing a regulated stream of air into the circulating oil before the oil enters the heating coil, commingling the air with the oil during its passage through the heating coil and then separating the residual air with the products of distillation from the oil.

14. A process of forming oxidized asphalt comprising maintaining a bulk supply of asphaltic oils in a chamber at a substantially uniform oxidizing temperature, substantially below a cracking temperature, by continuously circulating the oil from the bulk supply to and through a heating coil and back to the bulk supply, continuously introducing a regulated stream of air into the oil before the oil enters the heating coil, commingling the air with the oil during its passage through the heating coil and continuously separating and withdrawing the residual air and products of distillation from the chamber, and continuing said circulation, heating, and introduction of air until the asphaltic oil has been oxidized to the required degree.

15. A process of forming oxidized asphalt comprising maintaining a bulk supply of asphaltic oil in a chamber at a temperature of approximately 450 to 550° F., by causing the oil to continuously circulate from the bulk supply to and through a heating coil and back to the bulk supply, continuously introducing into the oil a regulated stream of air before the oil enters the heating coil, commingling the air with the oil during its passage through the heating coil continuously separating and withdrawing the residual air and products of distillation from the chamber, and continuing said circulation, heating, introduction of air, withdrawal of the residual air and products of distillation, from the chamber, until the asphaltic oil has been oxidized to the required degree.

16. A process of forming oxidized asphalt comprising maintaining a bulk supply of asphaltic oils in a chamber, continuously circulating the oil from the bulk supply to and through a heating coil heating the oil therein to an oxidizing temperature, continuously introducing a regulated stream of air into the oil before the oil enters the heating coil, commingling the air with the oil during its passage through the heating coil continuously returning the oil and air leaving the coil back to the bulk supply and then separating the residual air and products of distillation from the oil, and continuing the circulation and oxidation of the oil until an oxidized asphalt of the required melting point has been obtained.

17. A process of forming oxidized asphalt comprising maintaining a bulk supply of asphaltic oil in a container, continuously circulating the oil from the bulk supply to and through a heating coil and back to the bulk supply, heating the oil in its passage through the heating coil to an oxidizing temperature, substantially below a cracking temperature, continuously introducing a regulated stream of air into the oil before entering the heating coil, commingling the air with the oil during its passage through the heating coil continuously withdrawing the residual air and products of distillation from said container, and continuing the circulation and oxidation of the oil until the desired degree of oxidation has been obtained.

18. A process of forming oxidized asphalt comprising maintaining a bulk supply of asphaltic oil in a container, continuously circulating the oil from the bulk supply to and through a heating coil and back to the bulk supply, heating the oil in its passage through the heating coil to an oxidizing temperature substantially below the cracking temperature, continuously introducing a regulated stream of air into the circulating oil before the oil enters the heating coil commingling the air with the oil during its passage through the heating coil and continuously withdrawing the residual air and products of distillation from said container, continuing the circulation and oxidation of the oil until the desired degree of oxidation has been obtained, and then discontinuing the introduction of air and introducing steam into the container until the desired melting point, flash and ductility has been obtained.

19. A process of forming oxidized asphalt comprising maintaining a bulk supply of oil in a container, continuously circulating the oil from the bulk supply to and through a heating coil and back to the bulk supply, heating the oil in its passage through the heating coil to an oxidizing temperature, continuously introducing regulated streams of air and steam into the circulating oil before the oil enters the heating coil, commingling the air and steam with the oil during its passage through the heating coil, continuously withdrawing the residual air, steam and products of distillation from said container, and continuing the circulation and introduction of air and steam until a product of the desired melting point, flash, ductility and penetration has been obtained.

20. A process of forming oxidized asphalt comprising maintaining a bulk supply of asphaltic oil in a container, continuously circulating the oil from the bulk supply to and through a heating coil and back to the bulk supply, heating the oil in its passage through the heating coil to an oxidizing temperature, intermittently introducing regulated streams of air and steam into the circulating oil before the oil enters the heating coil, intermittently commingling the air and steam with the oil during its passage through the heat-

ing coil, continuously withdrawing the steam, air and products of distillation from said container, and continuing the circulation and itermittent introduction of air and steam into the heating coil until a product of the desired melting point, flash and ductility has been obtained.

21. A method of producing oxidized asphalt which comprises maintaining a body of oil in a still and circulating the oil from said still, through an elongated conduit back into the body of oil in the still, commingling said circulating oil with air so that oil commingled with air passes through said elongated conduit to oxidize said oil and to produce asphalt, withdrawing vapors and gases from the still and removing the oxidized asphalt from the still.

22. A method of producing oxidized asphalt which comprises maintaining a body of oil in a still circulating a stream of oil withdrawn from said body through an elongated conduit, commingling said circulating oil with air so that oil commingled with air passes through said elongated conduit and maintaining said oil in said elongated conduit at a controlled elevated temperature and introducing said stream into the body of the oil in the still, withdrawing vapors and gases from said still, continuing said circulation and air introduction at the elevated temperature until asphalt of the desired consistency is produced and withdrawing said asphalt from said still.

23. A method of producing oxidized asphalt which comprises maintaining a body of oil in a still at an elevated asphalt forming temperature, pumping said oil from said bulk supply back into said bulk supply in a restricted circulating stream, intimately commingling air with said restricted circulating stream of oil, separating vapors and gases from said oil continuing said circulation to produce oxidized asphalt and removing oxidized asphalt from said still.

24. A method of producing oxidized asphalt which comprises maintaining a bulk supply of asphaltic oil at an elevated asphalt forming temperature in a still, circulating the oil from said bulk supply through a jet mixer positioned within the still, commingling the air and oil in said jet mixer and returning the circulating oil to the bulk supply, separating the vapors and gases from the still, continuing said circulation to produce oxidized asphalt and removing oxidized asphalt from the still.

25. A method of producing oxidized asphalt which comprises maintaining a bulk supply, inducing a circulating stream of oil within the bulk supply by pumping said oil from said bulk supply by a pump positioned within the still back to the bulk supply and introducing air into said circulating stream of oil, maintaining said oil at an elevated asphalt forming temperature, separating vapors and gases from said oil, continuing said circulation to produce oxidized asphalt and removing oxidized asphalt from said still.

EARLE W. GARD.
BLAIR G. ALDRIDGE.