

# United States Patent

[11] 3,617,515

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[21] Appl. No. 827,603  
[22] Filed May 26, 1969  
[45] Patented Nov. 2, 1971  
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[54] PRODUCTION OF NEEDLE COKE FROM COAL  
FOR PITCH  
13 Claims, 2 Drawing Figs.

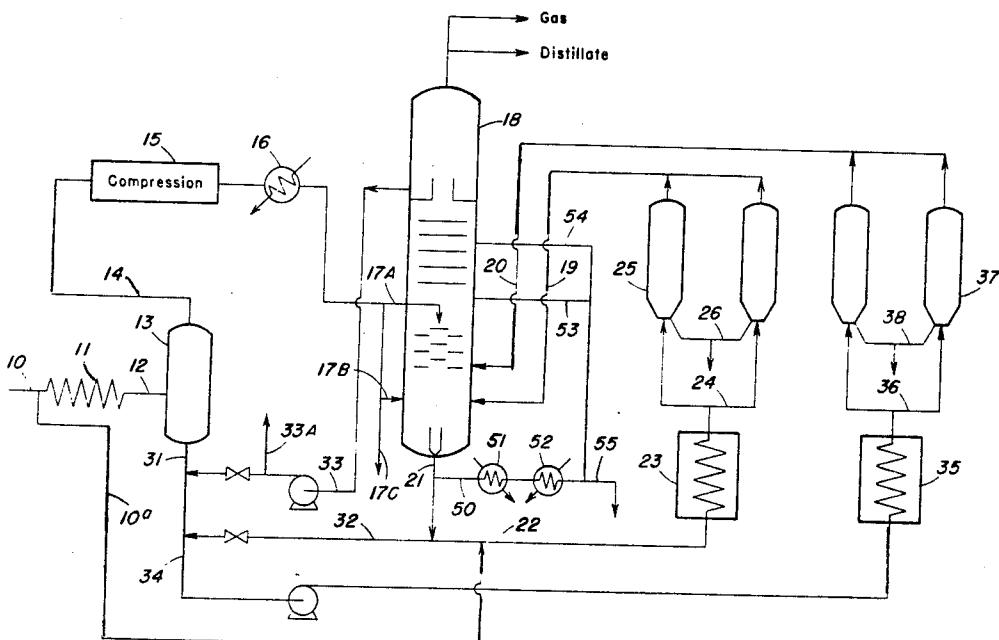
[52] U.S. Cl. 208/131,  
208/8, 208/80, 208/81, 208/82, 208/83, 208/93  
[51] Int. Cl. C10g 9/14  
[50] Field of Search 208/81-83,  
46, 106, 127, 131, 8, 44, 45; 201/25; 260/675

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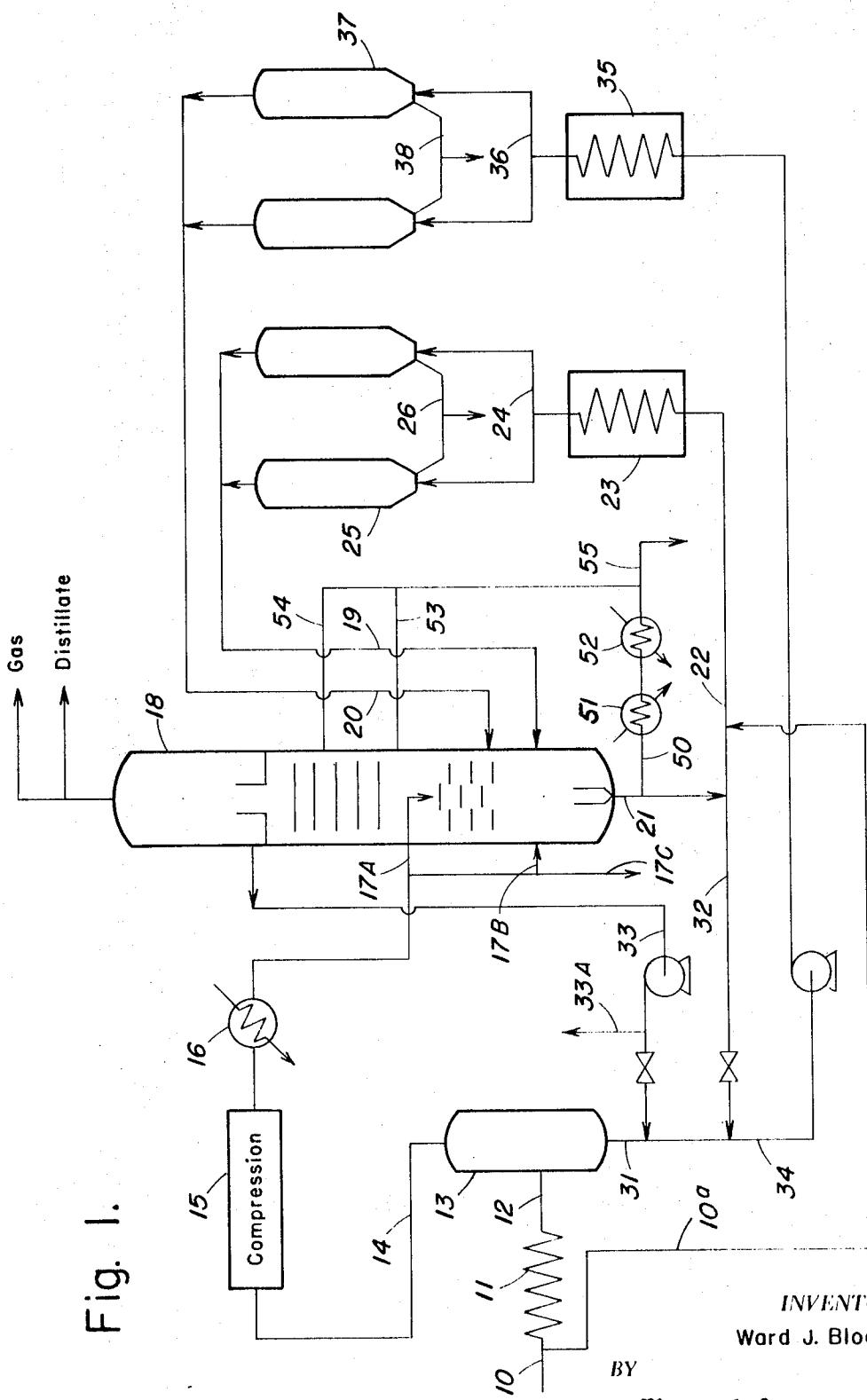
**ABSTRACT:** Process for producing graphitizable needle coke by delayed coking of a nonpetroleum fraction having a high content of condensed ring aromatic compounds and lower and upper cut points within the range from about 600° F. to about 1,200° F. In a preferred embodiment, the hereinabove noted fraction is separated from coal tar pitch having components boiling above 1,000° F. and the fraction boiling above 1,000° F. coked to carbon electrode grade coke.



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SHEET 1 OF 2



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SHEET 2 OF 2

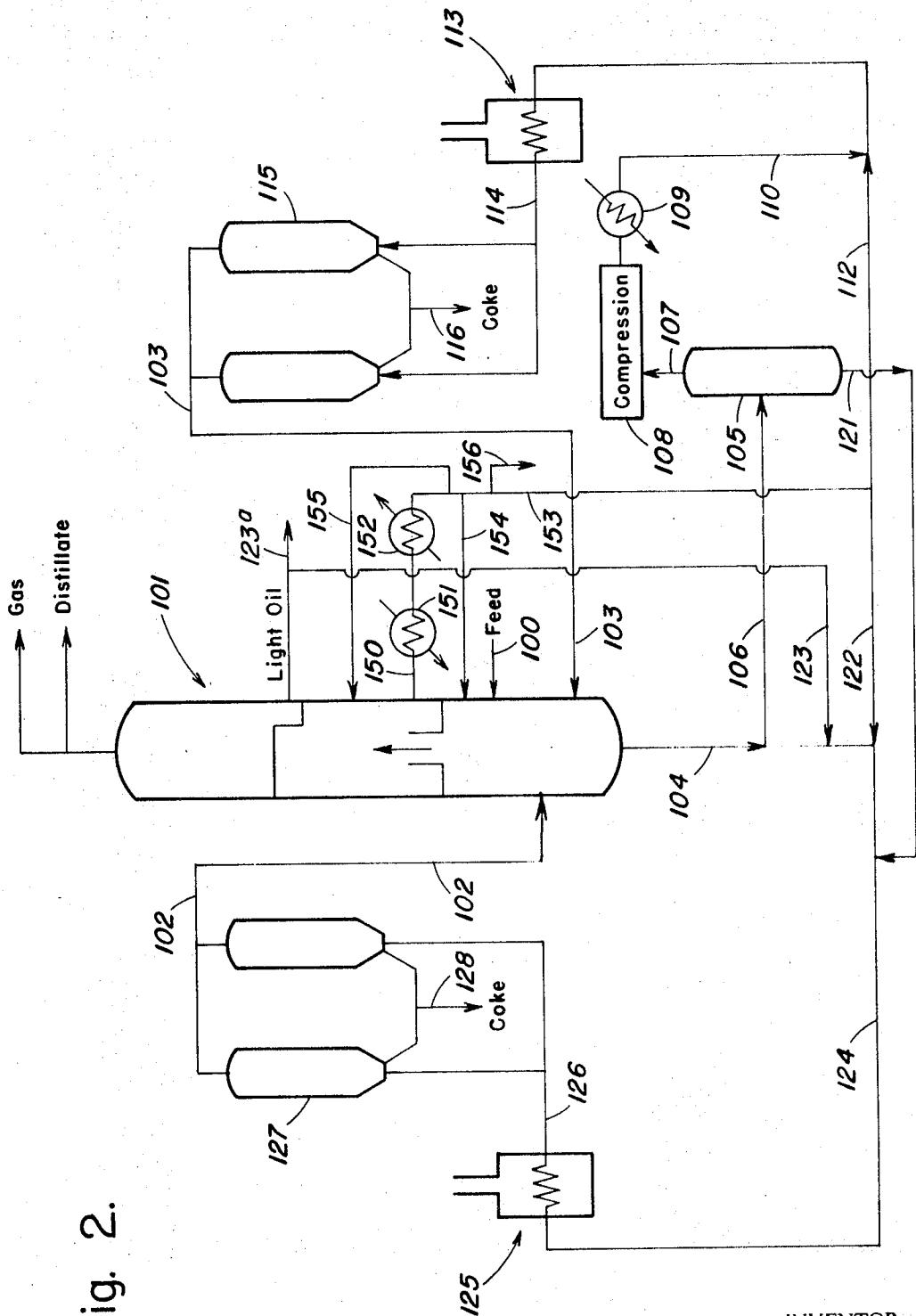


Fig. 2.

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## PRODUCTION OF NEEDLE COKE FROM COAL FOR PITCH

This invention relates to the production of graphitizable needle coke and more particularly to a process for the simultaneous production of such needle coke and carbon electrode grade coke.

Needle coke, after calcination and graphitization, is characterized by a low longitudinal coefficient of thermal expansion which is matched by a low electric resistivity and it is primarily used in producing high-quality synthetic graphite electrodes for electrosteel furnaces and for other electrothermal and chlor-alkali industries. Needle coke is a "premium" grade coke which is generally priced at \$50-\$100 per ton.

In copending application Ser. No. 746,706 filed of July 15, 1968 there is described a process for producing a carbon electrode grade coke from a particular feedstock having a high content of condensed aromatic compounds. Carbon electrode grade coke is generally priced at \$15-\$40 per ton and consequently the production of needle coke from such a feed is more desirable.

Accordingly, an object of this invention is to provide a new and improved process for producing graphitizable needle coke.

Another object of this invention is to provide a process for the simultaneous production of such needle coke and carbon electrode grade coke.

A further object of this invention is to provide a process for producing such needle coke by a delayed coking technique.

Still another object of this invention is to provide a process for producing such needle coke from a feed having a high content of condensed ring aromatic compounds.

These and other objects of the invention should be more readily apparent from the following detailed description thereof when read with reference to the accompanying drawing wherein:

FIG. 1 is a simplified schematic flow diagram of an embodiment of the invention; and

FIG. 2 is a simplified schematic flow diagram of another embodiment of the invention.

The objects of this invention are broadly accomplished by subjecting to coking conditions of temperature and pressure a fraction derived from a nonpetroleum source having a high content of condensed ring aromatic compounds and at least 80 percent of which boils within the range from about 600° F. to about 1,200° F., preferably from about 600° F. to about 1,000° F., to produce graphitizable needle coke. In accordance with a preferred embodiment of the invention, the fraction boiling within the range from about 600° F. to about 1,200° F. is derived from a feedstock which includes components boiling above 1,200° F. and the residue, after separation of the fraction used as a source of such needle coke, is employed for the production of a carbon electrode grade coke.

The feeds generally treated in accordance with the invention are derived from coal and contain a fraction having a high content; i.e., generally greater than about 70 percent, of condensed ring (polynuclear) aromatic compounds, both heterocyclic and isocyclic. The feed is preferably treated to recover a fraction having upper and lower cut points falling within the range from about 600° F. to about 1,200° F. and this fraction is coked to a high-grade needle coke, with the higher boiling residue; i.e., the fraction boiling above the upper cut point of the needle coke fraction, preferably being coked to carbon electrode grade coke. It is to be understood that the fraction employed for the production of needle coke may have components boiling throughout the 600° F. to 1,200° F. range or components boiling through only a portion of the range; i.e., 700°-900° F. It is further to be understood that components boiling below about 600° F. are only excluded for the reason that such components are not coke precursors and therefore would not be "coked" during the operation. Consequently, if desired, components boiling below about 600° F. may be included as a diluent, but in general such components are excluded in that they would diminish the overall capacity of the

equipment. A preferred feed is a coal tar pitch obtained by either the high-temperature or low-temperature carbonization of coal, as generally known in the art, the former feed particularly being generally characterized as completely comprised of condensed ring aromatic compounds (an estimated 5,000 of such compounds), with two-thirds of the aromatic compounds being isocyclic and the remaining third heterocyclic.

The feed to be converted to needle coke, as hereinabove noted, may also contain components which boil above about 1,200° F., but higher boiling components should not comprise more than about 20 percent of the feed to be coked to needle coke, preferably no greater than about 15 percent of the feed.

The coking of a feed containing components boiling above about 1,200° F. produces a grade of needle coke which is lower than the grade of needle coke produced from a feed free of such components, but this lower grade of needle coke has a coefficient of thermal expansion sufficiently low to meet various commercial specifications for a needle coke.

The invention will now be further described with respect to the accompanying drawings which illustrate embodiments for delayed coking of a feed containing coal tar pitch. The drawing has been simplified to facilitate the description thereof and therefore various processing expedients generally employed in

the art are not specifically shown therein. It is to be understood that the embodiment of the drawings are only illustrative of the invention and therefore the scope thereof is not to be limited thereby. Thus, for example, although the embodiments are particularly directed to the use of vacuum distillation equipment for recovering a 600°-1,000° F. fraction for the production of needle coke, it is to be understood that fractions having other boiling ranges or fractions obtained in a manner other than by vacuum distillation; e.g., solvent extraction are within the spirit and scope of the invention.

Referring now to FIG. 1, a feed, such as soft coal tar pitch derived from the higher temperature carbonization of coal, in line 10 is passed through a heater 11 to effect heating thereof to the operating temperature of a vacuum flash tower, as hereinafter described. The heated feed from heater 11 in line

12 is introduced into a vacuum flash tower 13, operating at a temperature and pressure designed to recover from the feed an overhead fraction containing components having boiling points up to about 1,000° F.; the tower 13 generally being

operated at a temperature from about 700° F. to about 850° F. and a pressure from about 0.25 p.s.i.a. to about 2.0 p.s.i.a.

An overhead is withdrawn from flash tower 13 through line 14, compressed to atmospheric pressure in a suitable compression device 15, preferably a multistage vacuum ejector

and passed through cooler 16 wherein the vapor is cooled to a temperature at which the vapor is condensed, generally a temperature from about 300° F. to about 500° F. The cooled liquid from cooler 16 in lines 17A and B is introduced above

and below, respectively, the vapor-liquid contact decks of a combination fractionator 18 operated under temperature and pressure conditions to produce a heavy oil bottoms having a lower cut point of about 600° F. and an upper cut point between about 900° F. and about 1,000° F.; a light oil, generally having cut points between about 400° F. and about

600° F.; and an overhead vapor comprised of gas and distillate, generally boiling up to about 400° F. The fractionator 18 is also provided with coke drum overhead vapors through lines 19 and 20 and heavy oil recycle through lines 53 and 54, as hereinafter described. The fractionator 18 is generally operated at an overhead temperature between about 300° F. and about 400° F., a bottoms temperature of between about 650° F. and about 850° F. a pressure between about 25 p.s.i.g. and about 90 p.s.i.g. and a volumetric recycle ratio of from

about 0.3:1 to about 2.0:1, preferably from about 0.5:1 to about 2.0:1, based upon equivalent feed to fractionator 18, with higher recycle ratios generally decreasing the overall capacity of the equipment. A portion of the liquid from cooler 16 may be passed through branch line 17C to storage and/or

further treatment; e.g., to produce carbon black.

A heavy oil bottoms having the hereinabove noted cut points is withdrawn from fractionator 18 through line 21 and a portion thereof is passed through line 22 to a coking heater 23 operated at an outlet temperature of between about 900° F. and about 1,000° F. and in a manner to prevent premature coking therein; i.e., the feed is maintained in turbulent motion or at a high velocity by providing temperature and pressure profiles in the heater that will produce partial vaporization of the feed, thereby preventing the coking problems caused by slow moving feed in the liquid state. In addition, controlled amounts of steam may be introduced into the coking heater 23 at appropriate places to obtain the required turbulence or high velocity.

The heated heavy oil is withdrawn from the coking heater 23 through line 24 and introduced into coke drums 25, of a type known in the art, wherein the heavy oil is converted to needle coke and lighter components. The coking drums are operated at a pressure of between about 15 p.s.i.g. and about 90 p.s.i.g., preferably between about 25 and about 90 p.s.i.g. and an overhead temperature of between about 840° F. and about 900° F., preferably between about 860° F. and about 900° F. The needle coke is withdrawn from the drums 25 through line 26.

An overhead is withdrawn from the coke drums 25 through line 19 and introduced into the fractionator 18 at a point below the introduction of the flashed overhead in line 17A. The cooled flashed overhead in line 17A aids in condensing heavy oil from the coke drum overhead vapor.

A bottoms recovered from the flash tower 13 in line 31, having an initial boiling point of greater than 900° F. to 1,000° F., is mixed with either heavy oil from the fractionator 18 in line 32, light oil from the fractionator in line 33 or both to dilute the high boiling bottoms. The light oil and/or heavy oil is mixed with the bottoms in an amount to provide a volumetric ratio of between about 0.2 and about 0.5 of light and/or heavy oil to bottoms. The remainder of the light oil is passed through line 33A to storage and/or further treatment.

The mixture is introduced into a coking heater 35, of a type known in the art. The coking heater is operated so as to produce an outlet temperature of between about 900° F. and about 960° F. The coking heater 35 is operated as generally known in the art to prevent premature coking therein, i.e., the feed is maintained in turbulent motion or at a high velocity by providing temperature and pressure profiles in the heater that will produce partial vaporization of the feed, thereby preventing the coking problems caused by slow moving feed in the liquid state. In addition, controlled amounts of steam may be introduced into the coking heater 35 at appropriate places to obtain the required turbulence or high velocity.

The heated mixture is withdrawn from the coking heater 35 through line 36 and introduced into coke drums 37, of a type known in the art, wherein the mixture is converted to carbon electrode grade coke and lighter components. The coking drums are operated at a pressure of between about 15 p.s.i.g. and about 90 p.s.i.g., preferably between about 25 and about 90 p.s.i.g. and an overhead temperature of between about 840° F. and about 900° F. preferably between about 860° F. and about 900° F. The coke is withdrawn from the drums 37 through line 38.

An overhead is withdrawn from the coke drums 37 through line 20 and introduced into the fractionator 18 at a point below the introduction of the flashed overhead in line 17A. The cooled flashed overhead in line 17A aids in condensing heavy oil from the coke drum overhead vapor. A portion of the heavy oil withdrawn from fractionator 18 through line 21 is passed through line 50, cooled in heat exchanger 51 by indirect heat transfer with a suitable coolant; e.g., the feed to the flash tower 13 in line 10, and further cooled by indirect heat transfer with a suitable coolant; e.g., boiler feed water, in heat exchanger 52 to a temperature suitable for inducing additional recycle in fractionator 18 to meet the hereinabove noted volumetric recycle requirements, generally a temperature from about 400° F. to about 700° F. A portion of the cooled heavy

oil from heat exchanger 52 is introduced into the fractionator 18 through line 53 to aid in providing the recycle requirements. Thus, the total recycle is comprised of: the heavy oil fraction returned through line 53 and the condensed portion of the coke drum overhead vapors introduced through lines 19 and 20, with the condensation of materials from the coke drum overhead vapors being induced by direct contact in fractionator 18 between the coke drum overhead vapors and both the cooled liquid introduced through line 17A and the cooled heavy oil recycle introduced through line 53. The remaining heavy oil from heat exchanger 52 is introduced into the fractionator 18 through line 54 to maintain desired operating conditions and passed through line 55 to storage and/or further treatment; e.g., the production of carbon black.

A further embodiment of the invention is illustrated in FIG. 2. Referring now to FIG. 2, a liquid feed, such as, soft coal tar pitch derived from the high- or low-temperature carbonization of coal, in line 100 generally at a temperature between about 400° F. and about 600° F. is introduced into a coker combination fractionator 101 operated under temperature and pressure conditions to produce a heavy oil having a lower cut point of about 600° F. and an upper cut point between about 700° F. and about 1,000° F.; light oil, generally having cut points between about 400° F. and about 600° F.; and an overhead vapor comprised of gas and distillate, generally boiling up to about 400° F. The fractionator 101 is generally operated at an overhead temperature of between about 300° F. and about 400° F., a pressure between about 25 p.s.i.g. and about 100 p.s.i.g., and a volumetric recycle ratio from about 0.3:1 to about 2.0:1, preferably from about 0.5:1 to about 2.0:1, based on equivalent feed, with higher recycle ratios generally decreasing the overall capacity of the equipment.

The liquid feed introduced into the fractionator 101 through line 100 is contacted with hot coke drum overhead vapors introduced through lines 102 and 103, obtained as hereinafter described, resulting in flashing of materials having boiling points up to about 900° F. to about 1,000° F. from the liquid feed. The unflashed portion of the liquid feed is withdrawn from the fractionator 101 through line 104 and if the heat input to the fractionator 101 is not sufficient to flash essentially all of the material boiling up to about 900° F. to about 1,000° F. from the feed, the portion of the liquid in line 104 is introduced into a flash tower 105 through line 106, operating at a temperature and pressure to provide an overhead containing components boiling up to about 1,000° F.; generally an operating temperature of between about 600° F. and about 800° F. and an operating pressure between about 0.25 p.s.i.a. and about 2.0 p.s.i.a. An overhead is withdrawn from flash tower 105 through line 107 compressed to atmospheric pressure in a suitable compression device 108, preferably a multistage vacuum ejector, and passed through cooler 109 wherein vapor is cooled to a temperature at which the vapor is condensed, generally a temperature from about 200° F. to about 400° F. The cooled liquid from cooler 109 in line 110 is mixed with heavy oil in line 112 withdrawn from the combination-fractionator 101, as hereinafter described and the mixture introduced into a coking heater 113, of a type known in the art. The coking heater 113 is operated at an outlet temperature of between about 900° F. and about 1,000° F. and in a manner to prevent premature coking therein; i.e., the feed is maintained in turbulent motion or at a high velocity by providing temperature and pressure profiles in the heater that will produce partial vaporization of the feed, thereby preventing the coking problems caused by slow moving feed in the liquid state. In addition, controlled amounts of steam may be introduced into the coking heater 113 at appropriate places to obtain the required turbulence or high velocity.

The heated heavy oil is withdrawn from the coking heater 113 through line 114 and introduced into coke drums 115, of a type known in the art, wherein the heavy oil is converted to graphitizable needle coke and lighter components. The coking drums are operated at a pressure of between about 15 p.s.i.g. and about 90 p.s.i.g., preferably between about 25 and about

90 p.s.i.g. and an overhead temperature of between about 840° F. and about 900° F., preferably between about 860° F. and about 900° F. The needle coke is withdrawn from the drums 115 through line 116.

An overhead is withdrawn from the coke drums 115 through line 103 and introduced into the fractionator 101 below the point of introduction of the feed in line 100 to recover various components from the overhead; the overhead also providing a portion of the heat requirements for flashing of the feed.

A bottoms recovered from the flash tower 105 in line 121 or the bottoms in line 104, having an initial boiling point of greater than 900° F. to 1,000° F., is mixed with either heavy oil from the fractionator 101 in line 122, light oil from the fractionator 101 in line 123 or both, to dilute the high boiling bottoms. The light oil and/or heavy oil is mixed with the bottoms in an amount to provide a volumetric ratio of between about 0.2 and about 1.0 of light and/or heavy oil to bottoms. The remainder of the light oil is passed through line 123A to storage and/or further treatment.

The mixture in line 124 is introduced into a coking heater 125, of a type known in the art. The coking heater is operated so as to produce an outlet temperature of between about 900° F. and about 960° F. The coking heater 125 is operated as generally known in the art to prevent premature coking therein; i.e., the feed is maintained in turbulent motion or at a high velocity by providing temperature and pressure profiles in the heater that will produce partial vaporization of the feed, thereby preventing the coking problems caused by slow moving feed in the liquid state. In addition, controlled amounts of steam may be introduced into the coking heater 125 at appropriate places to obtain the required turbulence or high velocity.

The heated mixture is withdrawn from the coking heater 125 through line 126 and introduced into coke drums 127, of a type known in the art, wherein the mixture is converted to carbon electrode grade coke and lighter components. The coke drums are operated at a pressure of between about 15 p.s.i.g. and about 90 p.s.i.g., preferably between about 25 and about 90 p.s.i.g. and an overhead temperature of between about 840° F. and about 900° F., preferably between about 860° F. and about 900° F. The coke is withdrawn from the drums 127 through line 128.

An overhead is withdrawn from the coke drums 128 through line 102 and introduced into the fractionator 101 below the point of introduction of the feed in line 100 to recover various components from the overhead; the overhead also providing a portion of the heat requirements for flashing of the feed.

A heavy oil fraction, having the hereinabove noted cut points, is withdrawn from fractionator 101 through line 150, cooled in heat exchanger 151 by indirect heat transfer with a suitable coolant; e.g., the feed in line 100, and further cooled by indirect heat transfer with a suitable coolant; e.g., boiler feed water, in heat exchanger 152 to a temperature suitable for inducing required recycle in fractionator 101, generally a temperature of from about 400° F. to about 700° F. A portion of the cooled heavy oil is passed through line 153 to provide the heavy oil for lines 112 and 122, as hereinabove described.

Another portion of the heavy oil from heat exchanger 152 is introduced into the fractionator 101 through line 154 at a rate to provide the hereinabove-described volumetric recycle ratio. Thus, the total recycle is comprised of the heavy oil fraction returned through line 154 and the condensed portion of the coke drum overhead vapors introduced through lines 102 and 103, the condensation being induced by direct contact with the cooled heavy oil fraction. The remaining portion of the heavy oil fraction from heat exchanger 152 is introduced into the fractionator 101 through line 155 to maintain desired operating conditions and passed through line 156 to storage and/or further treatment; e.g., production of high-grade carbon black as a result of its high BMCI, or recognized aromatic factor and its low sulfur content.

Numerous modifications of the hereinabove-described embodiments are possible within the scope of the invention. Thus, for example, needle coke may be produced from the 600° F. to 1,000° F. fraction without the simultaneous production of carbon electrode grade coke from the 1,000° F. + fraction. As another modification, a separate combination fractionator may be employed for each coking operation instead of the single fractionator as employed in the embodiments illustrated in FIGS. 1 and 2. The use of a second fractionator 10 may be advantageous in some operations in that the needle coke producing drums may then be operated at a higher pressure than the carbon electrode coke drums, without necessitating throttling of vapors.

As a further modification a portion of the coke drum overhead vapors may be used to assist the flashing in tower 13, such overhead vapors being passed to the fractionator through line 14 in admixture with the fraction flashed from the feed.

As yet a further modification, the embodiment of FIG. 2 may be operated in accordance with the process described in U.S. application Ser. No. 746,706, with the heavy oil recovered from the combination fractionator being employed for the production of graphitizable needle coke. This procedure is less preferred in that needle coke production is reduced in that the graphitizable needle coke precursors are not effectively recovered in the combination-fractionator.

As still another modification, the flash towers 13 and 105 of the embodiments of FIGS. 1 and 2, respectively, may be operated as conventional multijet induced vacuum flash towers with the overhead vapors being condensed for tower total reflux return and employed in lines 17 and 110, respectively, as hereinabove-described.

As yet a further modification, the embodiments of FIGS. 1 and 2 may be operated in a manner whereby the feed to the needle coke producing drums contains components boiling above about 1,200° F., the components boiling above about 1,200° F. comprising no more than about 20 percent of the feed to the needle coke drums, generally from about 5 to about 15 percent of the feed. Thus, for example, in accordance with the embodiment of FIG. 1, the heavy oil bottoms in line 22 may be mixed with a portion of coal tar pitch feed stock from line 10 in line 10a to provide components boiling above about 1,200° F. The coal tar pitch is employed in line 10a in an amount whereby the total feed introduced into heater 23, as hereinabove noted, contains no more than about 20 percent of components boiling above about 1,200° F. It is to be understood, however, that the coal tar pitch in line 10a may be mixed with the heavy oil in line 22 in proportions greater than 20 percent in that the coal tar pitch also contains components boiling below 1,200° F. and therefore such greater amounts will not provide more than 20 percent of components boiling above 1,200° F. Similarly, in accordance with the embodiment of FIG. 2, coal tar pitch may be added to the feed to the heater 113 in the manner hereinabove described.

These and other modifications should be apparent to those skilled in the art from the teachings herein.

The invention is further illustrated by the following examples but the scope of the invention is not to be limited thereby.

#### EXAMPLE I

A coal tar pitch is vacuum distilled into two fractions as described in table 1.

TABLE I

Inspections of Pitch	
specific gravity, 60°/60° F.	1.2308
Viscosity,	
SFS at 180° F.	214.0
SFS at 210° F.	57.5
Softening Pt., °F.	95
Conradson Carbon Residue, Wt. %	32.2
Sulfur, Wt. %	0.6

Ash, Wt. %  
Vacuum Distillation of Pitch

0.06

IBP, °F.	526
8.8 weight percent	612
17.9 weight percent	667
27.6 weight percent	714
37.2 weight percent	802
46.7 weight percent	856
61.6 weight percent	950
Properties of 950° F. + Residue	
Specific gravity at 100° F./100° F.	1.3223
Softening Pt., °F.	365
Conradson Carbon Residue, Wt. %	74.3
Sulfur, Wt. %	0.52
Weight Percent of Pitch	38.4

The distillate and residue were each coked in a single pass atmospheric operation at a temperature of 840°-870° F., with the coke produced from the distillate being a graphitizable needle coke having a low longitudinal coefficient of thermal expansion of not substantially above about  $6.0 \times 10^{-7}$  (°C.)<sup>11</sup> and the coke produced from the residue having a coefficient of thermal expansion in the range of  $12-20 \times 10^{-7}$  (°C.)<sup>11</sup>, being characterized as carbon electrode grade coke. The dual coking operation produced 20 parts of the needle coke and 36 parts of the carbon electrode grade coke, per 100 parts of whole coal tar pitch.

The coking of the pitch, without the dual coking of the invention, produces about 56 parts of carbon electrode grade coke per 100 parts of pitch.

## EXAMPLE II

A pitch having the properties of table 2 is delay coked as described in U.S. application Ser. No. 746,706, i.e., a heater outlet temperature of 890°-950° F., coke drum overhead temperature of 850° F., and pressure of 30 p.s.i.g. to produce the products of table 3.

TABLE 2

## Inspections of Pitch

Specific gravity, 100° F./60° F.	1.2234
Viscosity,	
SFS at 180° F.	238
SFS at 210° F.	61.8
Softening Pt., °F.	98.5
Conradson Carbon Residue, Wt. %	32.4
Ash, Wt. %	0.05
Sulfur, Wt. %	0.44

TABLE 3

Delayed Coking Product  
Yields on Pitch

On Coker Charge

Gas	2.5
Tar Light Oil	0.3
Carbolic Oil	0.2
Naphthalene Oil	1.2
Wash Oil	4.1
Heavy Oil	39.4
Coke, Carbon Electrode,	
Amorphous Coke	52.3
TOTAL	100.0

The heavy oil, is further distilled to an initial 650° F. cut point and this distilled fraction coked in a single pass atmospheric operation at a temperature of 840°-870° F. to produce the following products:

TABLE 4

Delayed Coking Yields of 650° F. Distillate	On Coker	
	Charge	On Pitch
Gas	1.8	0.6
Distillate	76.1	23.8
Coke, Graphite Electrode	22.1	6.9
Grade-Needle Coke	100.0	31.3
TOTAL		

The coke produced has the following properties:

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TABLE 5

15	Carbon electrode	Graphite electrode
	Grade-Amorphous	Grade-Needle
15	Carbon electrode	Graphite electrode
15	Grade-Amorphous	Grade-Needle
15	Coke	Coke
Yield, Wt. % on		
Coal Tar Pitch	52.3	6.9
Proximate Analysis		
Moisture, Wt. %	0.1	0.10
Volatile Matter, Wt. %	8.7	5.6
Fixed Carbon, Wt. %	91.3	94.2
Ash, Wt. %	nil	0.18
Sulfur, Wt. %	0.2	0.2
Bulk density, lb./cu. ft.	60.4	75.7

Thus, 100 parts of pitch produces 52.3 parts of carbon electrode grade coke and 6.9 parts of graphitizable needle coke and the yield of needle coke may be increased by operating the combination-fractionator to recover more such needle coke precursors from the initial coal tar pitch feed.

The overall process of the present invention is an improvement over the process of copending application Ser. No. 746,706 in that a feedstock having a high content of condensed ring aromatic compounds is upgraded to a coke commonly referred to as graphitizable needle coke, which after calcination and graphitization has a longitudinal coefficient of thermal expansion/°C. not substantially above about  $6.0 \times 10^{-7}$ , rather than solely to carbon electrode grade coke. In addition, the process of the invention incorporates the advantages inherent in an effective coking of a feedstock having a high content of condensed ring aromatic compounds, as noted in said copending application; i.e., high coke yields and the like.

Numerous modifications and variations of the invention are possible in light of the above teachings and therefore the invention may be practiced otherwise than as particularly described.

## What is claimed is:

1. A delayed coking process for producing graphitizable needle coke from a feed containing coal tar pitch, comprising: separating from a feed containing coal tar pitch a fraction having at least about 80 percent of the components thereof boiling somewhere within the range from about 600° F. to about 1,200° F. said fraction having a high content of a mixture of condensed ring aromatic compounds; rapidly heating said fraction to delayed coking temperature; and subjecting said fraction to delayed coking to produce graphitizable needle coke.
2. The process as defined in claim 1 wherein the upper and lower cut points of the fraction fall within the range from about 600° F. to about 1,200° F.
3. The process as defined in claim 1 wherein the upper and lower cut points of the fraction fall within the range from about 600° F. to about 1,000° F.
4. The process as defined in claim 1 wherein said coking is effected in a coke drum operating at an overhead temperature between about 840° F. and about 900° F.
5. The process as defined in claim 4 wherein said coke drum is operated at a pressure from about 15 to about 90 p.s.i.g.
6. A delayed coking process for producing graphitizable needle coke from a feed containing coal tar pitch, comprising:
  - a. introducing a feed containing coal tar pitch into a separation zone wherein a fraction having a boiling point of no greater than about 1,000° F. is separated from the feed,

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- said fraction having a high content of condensed ring aromatic compounds;
- b. introducing the fraction into a fractionation zone operated under conditions to produce a bottoms having upper and lower cut points within the range from about 600° F. to about 1,000° F.;
  - c. passing at least a portion of the bottoms through a heater to effect heating thereof to a temperature from about 900° F. to about 1,000° F.;
  - d. introducing the heated bottoms into a coking drum to effect delayed coking thereof at a temperature from about 840° F. to about 900° F. to graphitizable needle coke; and
  - e. passing overhead vapors from the coking drum to the fractionation zone.
7. The process as defined in claim 6 wherein the bottoms prior to step (c) is admixed with a coal tar pitch containing fraction in an amount to provide a mixture in which no more than about 20 percent of the components thereof boil above about 1200° F. 15
8. The process as defined in claim 6 wherein the fractionation zone is operated at an overhead temperature from about 300° F. to about 400° F., a bottoms temperature from about 650° F. to 850° F., a pressure from about 25 p.s.i.g. to about 90 p.s.i.g. and a volumetric recycle ratio, based on equivalent feed to the fractionation zone, from about 0.3:1 to about 2.0:1. 20
9. A delayed coking process for producing graphitizable needle coke comprising:
- a. introducing a feed containing coal tar pitch into a coker combination fractionation zone operating under conditions to provide a heavy oil fraction having upper and lower cut points within the range from about 600° F. to about 1,000° F., said fraction having a high content of condensed ring aromatic compounds, and to flash material boiling at a temperature no greater than about 1,000° F. from the feed; 35
  - b. recovering the heavy oil fraction from the combination fractionation zone;
  - c. passing at least a portion of the heavy oil fraction through a heating zone to effect heating thereof to a temperature from about 900° F. to about 1,000° F.;
  - d. introducing the heated heavy oil fraction into a coke drum operating at a temperature from about 840° F. to about 900° F. to effect delayed coking thereof; and
  - e. recovering graphitizable needle coke from said coke drum. 45
10. The process as defined in claim 9 wherein the heavy oil fraction prior to step (c) is admixed with a coal tar pitch containing fraction in an amount to provide a mixture in which no more than about 20 percent of the components thereof boil above about 1,200° F. 50
11. A delayed coking process for producing graphitizable needle coke and carbon grade coke from a feed containing coal tar pitch, comprising:
- separating a feed containing coal tar pitch into a first fraction having upper and lower cut points within the range from about 600° F. to about 1,000° F. and a remaining heavier second fraction, both having a high content of a mixture of condensed ring aromatic compounds; rapidly 60 heating the first fraction to delayed coking temperature; subjecting the first fraction to delayed coking to produce graphitizable needle coke; and subjecting the remaining heavier second fraction to delayed coking to produce carbon grade coke. 65
12. A delayed coking process for producing graphitizable needle coke and carbon grade coke from a feed containing

coal tar pitch, comprising:

- a. introducing a feed containing coal tar pitch into a separation zone to produce a first fraction having a boiling point of no greater than about 1000° F. and a second heavier remaining fraction, each having a high content of a mixture of condensed ring aromatic compounds;
  - b. introducing the first fraction into a fractionation zone operated under conditions to produce a bottoms having upper and lower cut points within the range from about 600° F. to about 1,000° F.;
  - c. passing at least a portion of the bottoms through a heating zone to effect heating thereof to a temperature from about 900° F. to about 1,000° F.;
  - d. introducing the heated bottoms into a first coking drum to effect delayed coking thereof at a temperature from about 840° F. to about 900° F. to produce graphitizable needle coke;
  - e. passing overhead vapors from the first coking drum to the fractionation zone;
  - f. introducing the remaining second fraction from the separation zone into a heating zone wherein the remaining second fraction is heated to a temperature from about 900° F. to about 960° F.;
  - g. introducing the heated remaining second fraction into a second coking drum, operating at a temperature from about 840° F. to about 900° F. to effect delayed coking thereof to carbon grade coke; and
  - h. passing overhead vapors from the second coking drum into said fractionation zone.
13. A delayed coking process for producing graphitizable needle coke and carbon grade coke from a feed containing coal tar pitch, comprising:
- a. introducing a feed containing coal tar pitch into a coker combination fractionation zone operating under conditions to provide a heavy oil fraction having upper and lower cut points within the range from about 600° F. to about 1,000° F., said fraction having a high content of condensed ring aromatic compounds, and to flash material boiling at a temperature no greater than about 1,000° F. from the feed;
  - b. recovering the heavy oil fraction from the combination fractionation zone;
  - c. passing at least a portion of the heavy oil fraction through a heating zone to effect heating thereof to a temperature from about 900° F. to about 1,000° F.;
  - d. introducing the heated heavy oil fraction into a first coke drum operating at a temperature from about 840° F. to about 900° F. to effect delayed coking thereof to produce graphitizable needle coke;
  - e. introducing overhead vapors from the first coking drum into the combination fractionation zone;
  - f. recovering the unflashed portion of the feed from the combination fractionation zone;
  - g. introducing said unflashed portion into a flash zone to flash any remaining material boiling at a temperature no greater than about 1,000° F. therefrom, said flashed material being combined with the portion of the recovered heavy oil fraction which is to be heated;
  - h. heating the remaining liquid fraction from the flash zone to a temperature from about 900° F. to about 960° F.;
  - i. introducing the heated remaining liquid fraction into a second coking drum operated at a temperature between about 840° F. and about 900° F. to effect delayed coking thereof to carbon grade coke; and
  - j. introducing overhead vapors from the second coking drum into the combination fractionation zone.

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