March 14, 1933. L. C. KARRICK 1,901,169

DISTILLATION OF SOLID CARBONACEOUS MATERIAL

Filed May 7, 1924 2 Sheets-Sheet 1

Fig. 1.

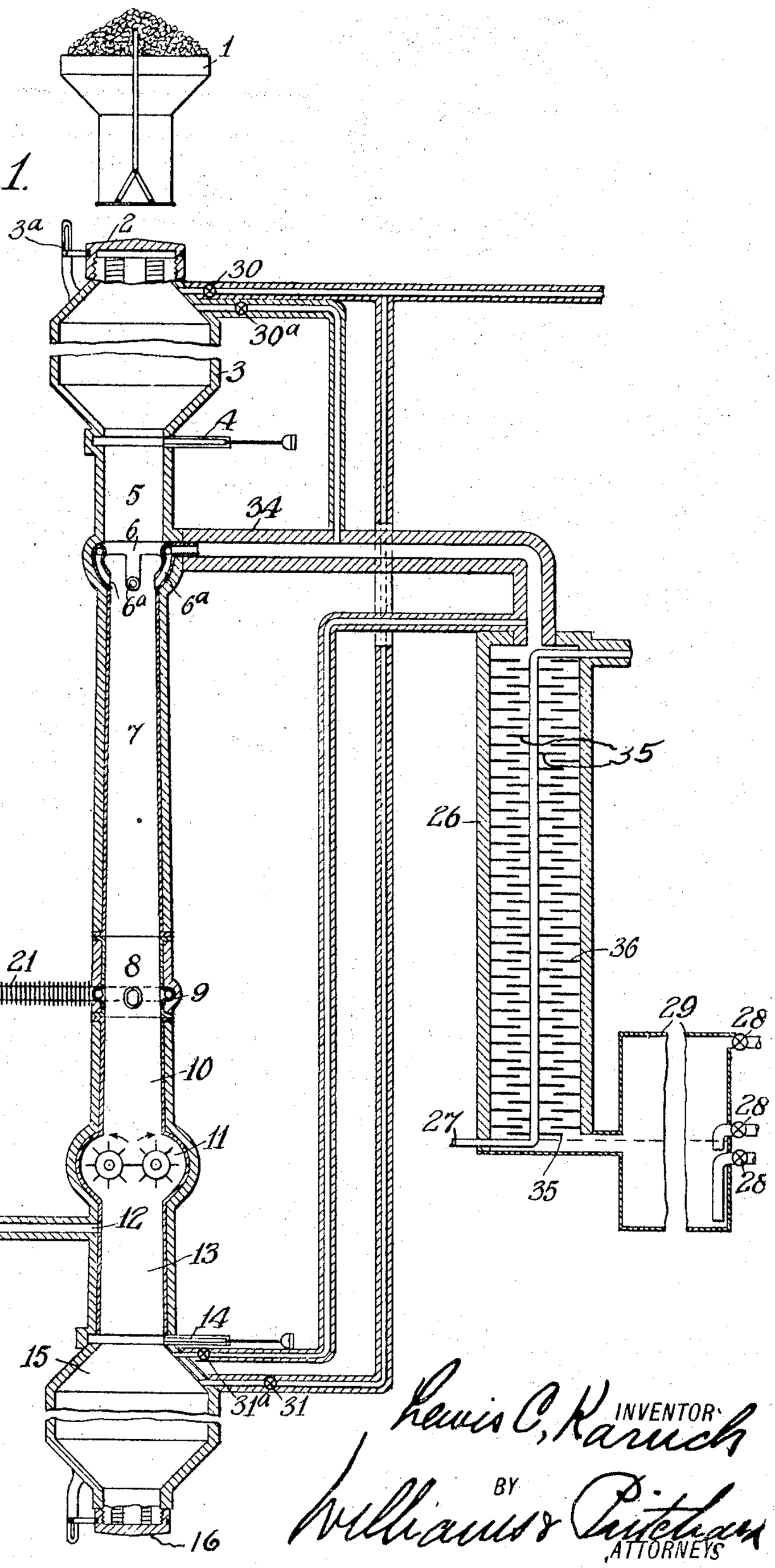

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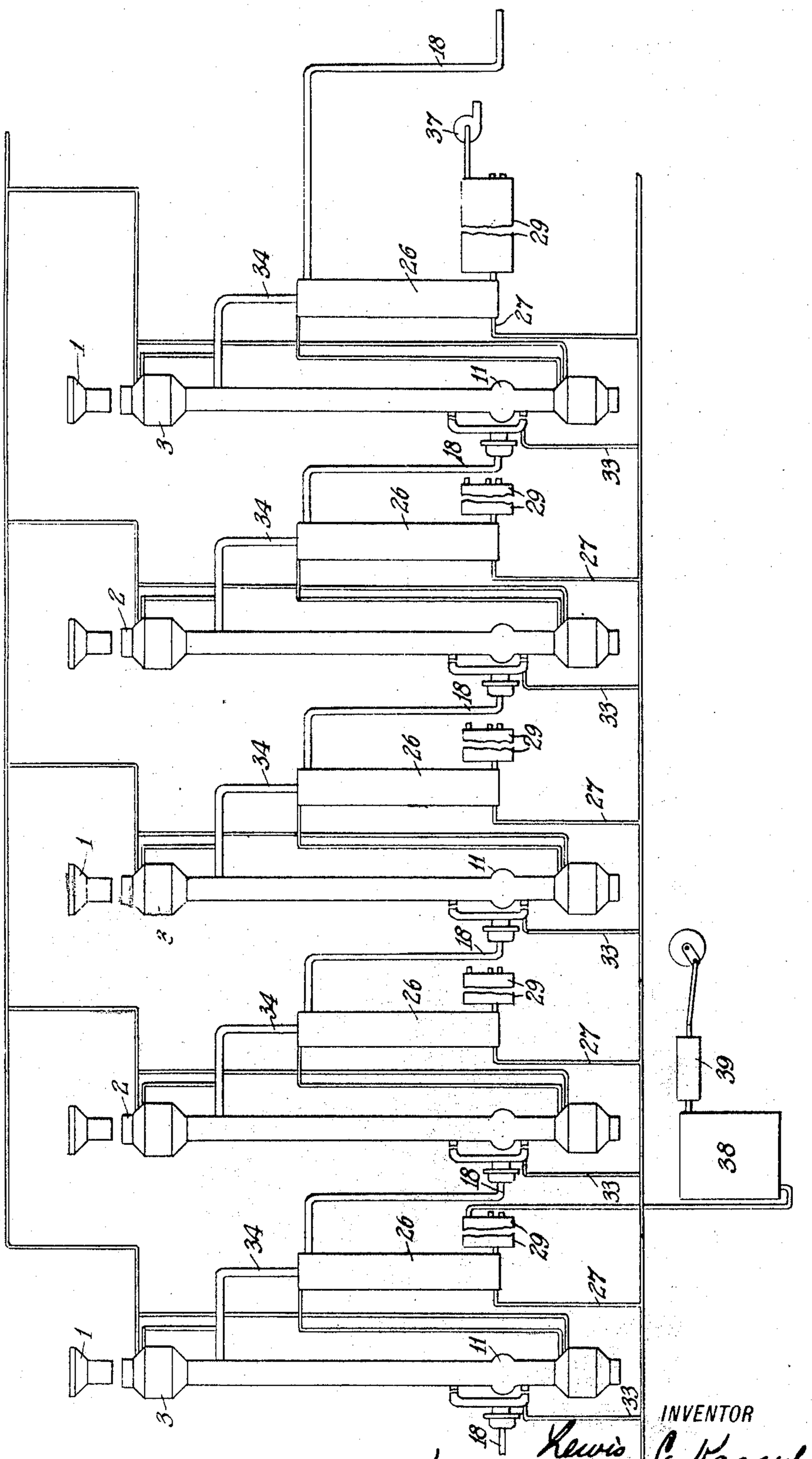
Fig. 2.



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UNITED STATES PATENT OFFICE

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DISTILLATION OF SOLID CARBONACEOUS MATERIAL

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This invention relates to a process for the treatment of solid carbonaceous substances for the purpose of removing all or any percentage of the total volatile constituents therefrom and, in the case of coals, leaving the coke either devoid of volatile ingredients or still containing any desired portion of the original volatile ingredients. The coke has desirable kindling and burning properties and will burn smokelessly. A large quantity of ammonia may be produced from the nitrogen containing ingredients of the material being treated. Liquid hydrocarbons, commonly called oils or low temperature tars are obtained from the volatized ingredients and also a residual combustible gas which may or may not carry in a vaporized condition "fixed-gas gasoline" which is similar in gravity and volatility to casinghead gasoline obtainable from wet natural gas.

The invention will be described in connection with the heat treatment of coal, but it is to be understood that it is also applicable to other materials such as oil shales, lignite, peat, wood or other solid substances classifiable as fuels broadly or substances which may yield fuels.

The raw material is heated in a retort which may be of any desired size to effect successful mechanical operation but the economic operation is enhanced by the use of large volumetric capacity. The material being treated or destructively distilled passes continuously through the distilling section of the retort, but is charged at the top and the coke is discharged at the bottom intermittently.

The material being distilled is sized by screening before delivery to the retorts, each size being preferably treated separately in different retorts to make possible greater plant capacity than would be the case if mixed sizes of material were fed to all retorts.

Superheated steam is used exclusively for the heat treatment of the raw material. The process heat that is utilized is the sensible heat of partially de-superheating the steam and the steam leaves the retort still superheated but at a lower temperature.

The superheated steam is supplied at a temperature high enough to distill such volatiles as may be desired and is preferably between 1200° F. and 1600° F. as it is within this range of temperatures that a large part of the organic nitrogen compounds in the presence of steam are converted to ammonia; this range of temperature is also suitable for carrying on the destructive distillation.

Since steam is the only source of heat and only a part of the heat of superheat of the steam is available for use in distilling, only a small percentage of the total heat of the steam is utilized in the retort.

A considerable part of the remaining total heat of steam discharged from the retort may be advantageously used to generate part of the fresh supply of steam required for the retort or preferably for a second retort operating at a lower pressure, and the sensible and latent heats of the coal volatiles may be used in the same way.

I may take advantage of the fact that vapors at different pressures condense at different temperatures. The retorts described in this specification may operate in batteries or units of two or more, for example, five retorts, and each retort may be maintained at a different pressure from the others, the first being held at the highest pressure and each one thereafter in the sequence or unit should be at a successively lower pressure. The pressure difference between the retorts should be such that difference between the condensing-temperature of the steam and coal volatiles and that of the freshly generated steam is preferably made equal or nearly so between each pair of retorts, the last retort operating at atmospheric pressure or preferably below atmospheric pressure in order that maximum advantage may be taken of the steam discharged from one retort in producing steam for the succeeding one. The vapors and gases from the first retort are conducted into a heat exchanger and allowed to condense, thereby giving up their heat to the water surrounding the tubes and generating fresh steam preferably at a lower pressure, and this new steam will be conducted through a second superheater and into the

next retort with an additional supply of steam if necessary.

My process also takes advantage of the sensible heat of the coke to provide part of the heat necessary to carry on the distillation. This may be accomplished by dividing the steam supplied to the retort before it reaches the superheater. Part of the steam goes through the superheater and into the retort at approximately its center, whereas the balance of the steam passes into the bottom section of the retort and by its counter current flow to the direction of flow of the moving coke, it abstracts the heat from the coke and the steam becomes superheated to approximately the same temperature as the balance of the superheated steam by the time it reaches the point of introduction of the latter steam. The steam used for this heat recovery purpose may be wet steam so as to insure ample quenching of the highly combustible coke formed. There will be a saving of fuel if the steam used for heat recovery is generated by water coming into contact with the hot coke in the bottom of the retort instead of being generated in the boiler or evaporator.

In the accompanying drawings:

Figure 1 is a diagrammatic sectional side view of a retort and its appurtenances.

Figure 2 is a similar view of a battery of retorts and some of their inter-connections.

Sized coal or material to be treated is delivered to the retorts from the carrier 1, which is cylindrical in shape, has a hopper top and a hinged bottom; the capacity is equal to the volume of the charging bin 3. The vapor-tight closure or cap 2 of the retort is released by giving it a sixty degree turn and swinging it back on a suitable hinge 3*a*. In order to relieve the pressure existing in the bin 3, a pressure release valve 30 is opened and the vapors are exhausted into one of the following evaporators about at atmospheric pressure, thereby not wasting the heat of the exhausted vapors. The carrier with its load is then lowered into the bin 3 until nearly resting on the bin bottom or slide 4 and then the carrier bottom is released and the carrier raised, thus permitting the charge to slide into the bin with the minimum amount of abrasion to the coal or material charged. It is assumed that the retort has been in continuous operation while the charging operation just described was accomplished, and therefore the gate or slide 4 at the bottom of the bin was closed and the retort proper 7, 8, was full up to the slide 4. During the charging operation the distillation has been in continuous progress and the charge-level has gradually settled from just below the slide 4 and is gradually approaching the level of the vapor off-take 6. As soon as the bin 3 is filled, which should not require more than one minute, the top 2 is closed, the valve 30*a* connected to a source of fairly high pressure, as the vapor line 34 or separator 29, is opened to partially balance the pressure against the lower side of the slide 4, and then the slide 4 is opened, thereby allowing the auxiliary bin 5 to fill up from 3. The bin 3 now remains closed at the top until the charge-level again sinks below the slide 4, whereupon the charging operations are repeated.

Since the flow of charge is continuous and uninterrupted in its passage through the retorting shaft 7, 8, it is proper that the flow of superheated steam from its source should be constant, and therefore, the supply of steam and its temperature are fixed when the plant is put into operation once a proper balance between supply of steam and rate of feed of the charge is determined.

Retorting shaft 7 is made, preferably, of steel plate, designed to suit the strength requirement of each retort, which obviously will be different, and may be of slightly increasing dimensions toward the bottom, as shown. The dimensions of the shaft 7 should be such that there will be ample time allowed for the largest lumps of coal composing the charge to completely distill while passing down through the retort 7, 8, and that the steam passing off from the retort will have part of its heat of superheat abstracted but leave it still superheated to avoid possibility of any appreciable condensation taking place in the retort.

The vapor off-take 6 is a manifold type with four or six branches 6*a* leading off from the retort at equidistant points around the circumference of the retort. The branches rise a few inches from their junction with the retort wall in order that no fine material of the charge will be blown out into the vapor line 34 by the current of the exhaust vapors and gases.

The retort shaft will be hottest at the point of introduction of the superheated steam and if the retort walls are properly insulated against radiation, the walls at all points will have the same temperature as the steam in contact with them. In order that the high temperatures necessary for the distillations will not make necessary frequent shutdowns for repairs to the retort, it is desirable that the portion of the retort walls that will be exposed to the highest temperatures should be made of a material which will give long service. The retort is therefore preferably built to embody a replaceable section 8 composed of cast nichrome or other refractory material. The section 8 need extend only a short distance above where the superheated steam enters.

The feed regulating mechanism or device 11 consists of two rolls or wheels having pockets around their circumferences. These turn on opposite directions and away from

each other at the top in order that the material passing them will not be crushed or disintegrated as would be the case if the rolls turned in the direction to cause the material to pass between them. The rolls turn at a uniform speed and may be provided with gears, not shown, so that if desired, the rate of feed can be changed.

The parts of the shaft below the inlet 9 for superheated steam and above the inlet 12 for saturated steam, including the section 10 above the feed regulating mechanism 11, and also including the space about the feed regulating mechanism 11, are cooling or heat recovery sections of the retorting device. The section 10 of the retort should be long and may be fully as long as the section 7, 8. The discharge from the feed regulating mechanism 11 falls directly through the gate or slide 14 which is just above the discharge bin 15, and falls into the discharge bin 15, until this bin is full, or full nearly, up to the slide 14. As above described saturated steam at the same pressure as the superheated steam, flows into the section 13 of the shaft through the port 12. Gas admitted through the valve 31 will have the same effect. As this saturated steam passes up counter-currently to the downcoming treated material, it first becomes dry steam and then becomes superheated. With a proper adjustment of the supply of saturated steam, the steam admitted at the port 12 will arrive opposite the ports 9 in approximately the same degree of superheat as the steam admitted from the superheater diagrammatically shown at 21. The coke will then be reduced in temperature from the maximum value at the level 8 down to the temperature of saturated steam, at the pressure held in the retort, and thereby almost all the residual sensible heat of the coke will have been abstracted and this same heat also will have been used to help distill the coal in the retort.

The discharge bin 15 is closed at its bottom by a cap 16 which may be of the same construction as the cap 2 at the top of the feed bin 3. The discharge bin 15 is also provided with a pressure-relief valve 31 connected to the same conduit as the pressure-relief valve 30 of the feed bin 3.

When the slide 4 has been closed, due to the feed bin 3 being emptied, the discharge bin 15 should be full of the treated material; therefore the slide 14 is closed. The discharge bin 15 is now opened by taking off the bottom cap 16, after momentarily opening its pressure-release valve 31, corresponding to the relief valve 30 of the bin 4 and the charge is allowed to run out. The cap 16 is quickly closed, the valve 31*a* corresponding to the relief valve 30*a* of the bin 4 opened momentarily again to restore the pressure in the discharge bin 15, and the slide 14 is opened, thus allowing the treated material which has collected above the slide 14 to drop into the discharge bin 15.

The flow-dividing valve 19 is quick acting and will vary the flow in any proportions between the two steam feeding ports. This valve or divider is especially desirable when placed following a main steam valve 18.

Water may be sprayed from the pipe 33 into the flowing steam going into the retort through the port 12 to be evaporated by the coke, but in such an amount that no free moisture will be left in the coke. It is preferable that saturated steam carrying atomized water should be used. The best results will be attained in the use of partially devolatilized coal as a smokeless fuel, if it is used in a physically dry state, and therefore too much water carried into the retort with the steam may be objectionable because it will leave the treated material wet.

The mixed vapors and gases are conducted from the retort by the vapor line 34 into the heat exchanger, waste-heat boiler, or evaporator 26. Water from the feedwater heater of the steam plant or any other advantageous source of hot water, is fed into the evaporator 26 by the pipe 27. This pipe is shown for convenience of illustration as provided with heat-conducting fins 35. Annular baffle plates 36 extend inward from the wall of the heat-exchanger 26 between the fins. By proper regulation of the flow of water practically all the vapors will be caused to condense and the gas cooled, and as a result, steam will be generated from the water and this steam will be used in the following retort. The generation of steam for each retort after the first one may be accomplished in this manner, and in order to effect the necessary heat transfer in generating the steam, it is necessary that there be a difference in temperature maintained in the evaporators 26 between the incoming mixed vapors and gases and the outgoing freshly produced steam, and this may be effected by maintaining correspondingly different pressures. This pressure regulation is controlled by automatic pressure-regulating throttle-valves 28.

In the example shown in the drawings (Fig. 2) with five retorts sucessively arranged the successive pressures may be lower than in the successive retorts from left to right. Should it be desirable in any instance to carry the pressures in some of the retorts to a point below atmospheric pressure then vacuum pumps 37 would be needed as shown in relation to the fifth retort. The vacuum pumps to draw off the fixed gases and deliver them against the higher external atmospheric or still higher pipe line pressure.

Each separator 29 receives the discharge of combined condensates and cooled gases under the pressure that exists in the corresponding evaporator. Sufficient volume is pro-

vided to permit ample time for the maximum cleanness of separation of gas, oil and water to be effected. The gas produced may be sent to a storage or scrubbing tank 38 and used for any desired purpose. The oil may be refined, and the condensed water treated for the recovery of the valuable material it carries, principally nitrogen compounds.

Utah coal from the Castle Gate coal district was heated by steam entering at a temperature of 1100 to 1500° F. in a continuously fed retort according to the procedure described above. A very large part of the heat from the heated product was recovered. At the temperature thus obtained little or no decomposition took place of the hydrocarbon or other volatile products. The coal yielded oil at the rate of 35 gallons oil per ton of coal. This oil was separated by fractional distillation into

	Per cent
Motor fuel distillate	22
Heavy motor engine distillate	22
Cracking stock	25

balance, lubricating oil, wax distillate, and pitch residue. From one lot of the crude oil there was separated 20% of tar acids suitable for froth flotation operations and for wood preservation. Two different runs of run-of-mine coal from two different collieries yielded similar results both when distilled by atmospheric pressures and at higher pressures. The coal yielded in addition 3500 cubic feet of 950 B. t. u. gas per ton of coal. The gas contained very little illuminants rich in paraffin hydrocarbons and hydrogen. The residual coal was as easily kindled as the raw coal, under favorable conditions capable of being lit with a match. By the process a similar easily kindled coal was made from anthracite coal commercially sold in the Pittsburgh market, the small amount of oil and gas produced being allowed to waste. Quenching the treated coal from the retort by dry steam arrested the tendency to spontaneous combustion that it would otherwise possess, although its property of being easily kindled was not destroyed.

Having thus described certain embodiments of my invention what I claim is:

1. The combination with a retort, of means for distilling the contents thereof by superheated steam, a second retort, means for heating the second retort by steam, a heat-exchange device between the retorts, means in said heat exchange device to supply water to be heated to make steam for said second retort, and connections to and from the heat-exchange device whereby the discharged steam and vapors and gases from the first retort enter the heat exchange device, and steam for the second retort is produced in the heat-exchange device.

2. The combination with a retort, of means for distilling the contents thereof by internally supplied superheated steam under high pressure, a second retort, means for distilling the contents thereof by internally supplied superheated steam under lower pressure, a heat-exchange device between the retorts and having on its respective sides the pressures of the corresponding retorts, means in said heat exchange device to supply water to be heated to make steam for said second retort, off-takes for liquid and gas connected to said exchange device for the products of the first retort, and connections for carrying steam from the heat-exchange device to the second retort.

3. The combination with a retort, of means for heating it internally by steam, a second retort, means for heating the second retort internally by steam, a heat-exchange device between the retorts, means in said heat exchange device to supply water to be heated to make steam for said second retort, and connections to and from the heat-exchange device whereby the discharge steam from the first retort enters the heat-exchange device and steam for the second retort is supplied from the heat-exchange device.

4. The combination with a retort, of means for heating it internally by steam, a second retort, means for heating the second retort by steam, a heat-exchange device between the retorts, a water supply for said heat exchange device, off-takes for liquid and gas connected to said exchange device for the products of the first retort, valves for said off-takes to control the pressure at their side of the exchange device, and connections for conveying steam to the second retort from the heat-exchange device.

5. In a retort the combination of a charging device at the top and a discharging device at the bottom, a vapor line for leading away vapors materially below the charging device, a steam supply, means for dividing the steam supply, means for superheating one part and delivering it to the retort materially below the vapor line, and means for adding water to the other part of the steam supply and delivering it to the retort at a zone still further down in the retort, whereby the steam at the lower point of delivery receives heat from the charge as it rises to the point at which the superheated steam is admitted.

6. The process of distilling solid carbonaceous material which consists in feeding the material through a retort where it is exposed to superheated steam, distilling and discharging volatile products, condensing the volatile products and steam in a heat-exchange device at substantially retort pressure, boiling water by said device to produce another supply of steam at lower pressure, superheating part of the other supply, and heating carbonaceous material in a second retort from said other supply at said lower pressure.

7. The process of distilling solid carbonaceous material, which consists in feeding carbonaceous material simultaneously through a plurality of parallel retorts at different pressures, heating a first retort by superheated steam, passing the resulting vapors through a heat-exchange device that is provided with a supply of water thereby generating another supply of steam at lower pressure, and heating a second retort from said other supply.

8. The process of operating a plurality of retorts for distilling solid carbonaceous material, which consists in heating a first retort by superheated steam at a relatively high pressure to distill off volatile material, passing the resulting vapors through a heat-exchange device supplied with water to furnish another supply of steam at a lower pressure to heat a second retort, to distill off another lot of volatile material, and passing said other lot of distilled vapors through a second heat-exchange device supplied with water to furnish another supply of steam at a still lower pressure to supply heat to a third retort.

9. The process of operating a plurality of parallel retorts to recover volatile material from solid carbonaceous material, which consists in heating a first retort with superheated steam, condensing the steam and resulting volatile material in a heat-exchange device supplied with water to produce another supply of steam at lower pressure from the latent heat so released, and delivering this steam to a second retort to heat carbonaceous material therein.

10. The process of operating a plurality of parallel retorts to recover volatile material from solid carbonaceous material, which consists in heating a retort with superheated steam, condensing the steam and resulting volatile material in a heat-exchange device supplied with water to produce another supply of steam, conducting said steam through a second retort to which additional heat is also supplied, and condensing the volatile material in the second retort.

11. The process of distilling coal, which comprises passing lumps of coal through a chamber and heating it by means of superheated steam, one part of which is introduced into direct contact therewith intermediate the ends of said chamber and another part of which is obtained by saturated steam after contacting with the hot solid residue, the temperatures of the portions of steam being substantially equal to each other where they contact with the coal that is undergoing treatment.

12. The process of distilling coal, which comprises passing lumps of coal through a chamber and heating it by means of superheated steam, one part of which is introduced intermediate the ends of said chamber and another part of which is obtained by saturated steam after contacting with the hot residue, the amount of the saturated steam being such that the superheated steam therefrom will have approximately the same temperature as the other part of the steam when it reaches the place where the other part of the steam is introduced.

13. The process of distilling solid carbonaceous material, which comprises separating crushed material into sizes, feeding different sizes to different retorts, distilling the contents of each retort by superheated steam in contact with the material, maintaining different pressures in the retorts, using the waste heat of vapors and gases from one retort to produce steam for a succeeding retort at a lower pressure, and using the coarser material in the retort of lower pressure.

14. The process of operating a plurality of retorts for distilling solid carbonaceous material, which consists in heating a first retort by superheated steam under high pressure at a relatively high temperature to distill off oil and gases, passing the resulting vapors and gases under pressure into a heat-exchange device supplied with water to furnish another supply of steam at a lower pressure to heat a second retort, to distill off another lot of oil and gases, and passing said other lot of distilled vapors and gases under pressure into a second heat-exchange device supplied with water to furnish another supply of steam at a still lower pressure to supply heat to a third retort, and withdrawing the incondensible gases under pressure from said heat-exchange devices.

In testimony whereof, I have affixed my signature to this specification.

LEWIS C. KARRICK.