F. RASCHIG.

PROCESS AND APPARATUS FOR CONTINUOUS DISTILLATION.

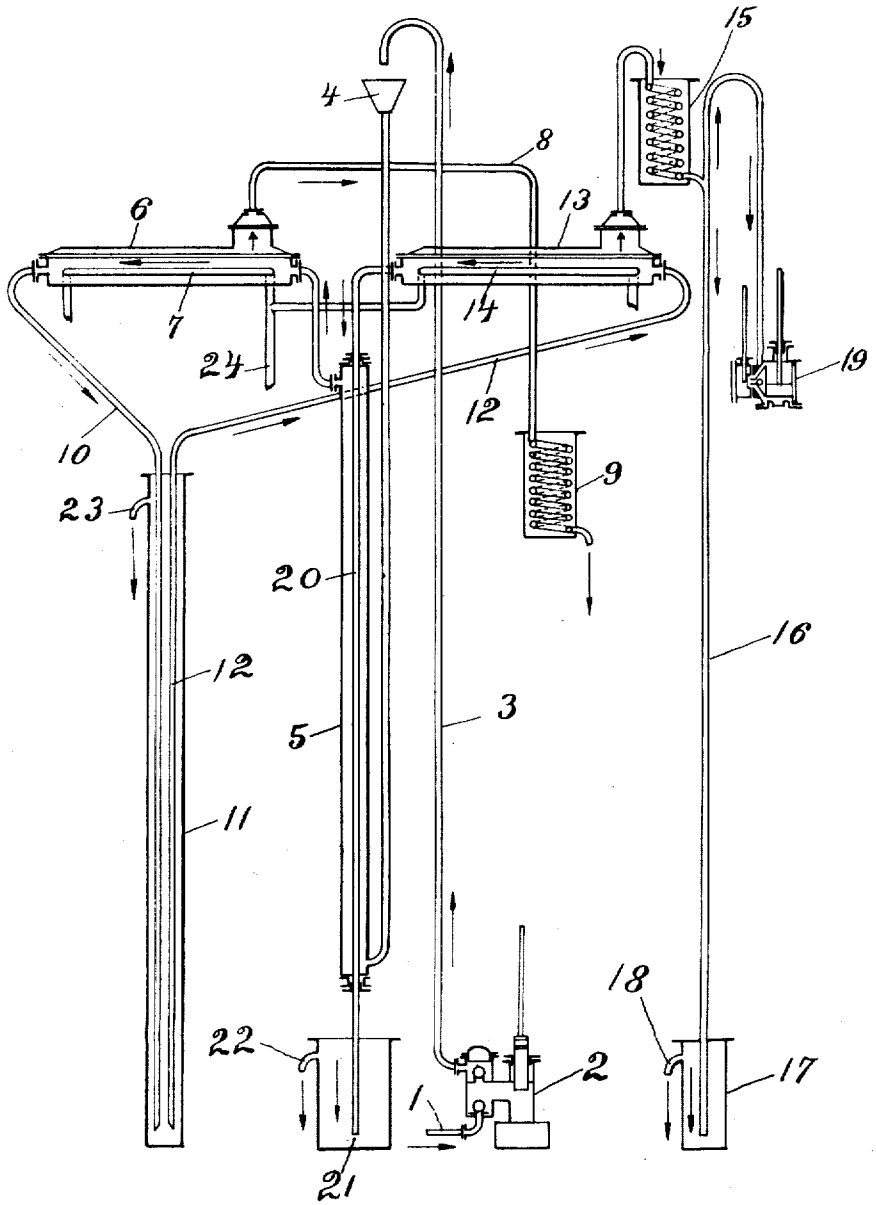
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2 SHEETS—SHEET 1.

Fig. 1.



WITNESSES

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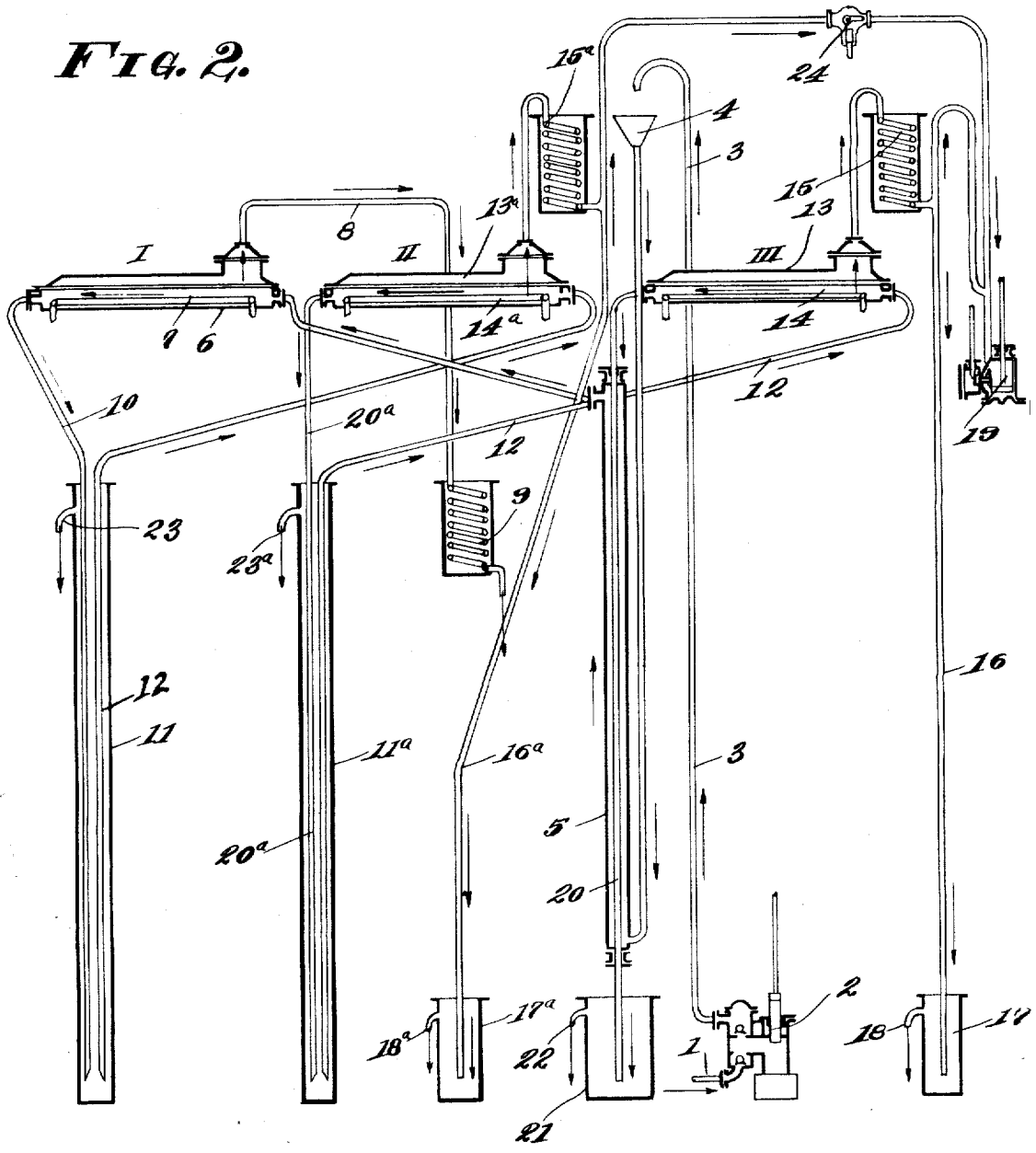
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2 SHEETS—SHEET 2.

FIG. 2.



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PROCESS AND APPARATUS FOR CONTINUOUS DISTILLATION.

1,141,265. Specification of Letters Patent. Patented June 1, 1915.

Application filed January 8, 1913. Serial No. 740,804.

To all whom it may concern:

Be it known that I, FRITZ RASCHIG, doctor of philosophy, chemist, a citizen of the German Empire, residing at Ludwigshafen-on-the-Rhine, Germany, have invented a new and useful Improvement in Processes and Apparatus for Continuous Distillation, of which the following is a specification.

This invention relates to a novel process and apparatus for distillation of tar, petroleum, etc., in a continuous manner; and it comprises, as an apparatus, a plurality of distillation retorts, each successive one of which is maintained under a higher vacuum than the preceding retort, each retort being provided with heating means and with means for removing in a continuous manner the distillate therefrom, and which retorts have between them open vessels by means of which the access of liquid into the one retort is so regulated that it remains equal to that running off from the preceding retort; and, as a process, it comprises a continuous process of distillation of such liquids as tar, petroleum, etc., in which the liquid is passed successively through a series of two or more retorts heated advantageously to the same temperature, but, maintained under a progressively increasing vacuum, and from each of which retorts that portion of the distillate resulting at the particular pressure and temperature is continuously removed. The inflow into each retort is maintained equal to the outflow from the preceding retort during the operation, all as more fully hereinafter set forth and as claimed.

In the distillation of tar, petroleum, etc., by allowing the tar to flow in an even stream through a series of retorts heated successively to higher temperatures, each succeeding retort affords a higher fraction than the preceding one; but this process has the disadvantage that the heating of the retorts must be very carefully watched and regulated since any variations in temperature cause great irregularities in the quantity, composition and boiling point of the distillates, and also of the outflowing residual oil. This process has the further disadvantage that automatic heating by high pressure steam is precluded, because, first, a different temperature would be necessary for each apparatus, and, also because the temperatures necessary are higher than the highest available for such purposes,—those corresponding to pressures around 20 atmospheres.

Where distillation is effected by allowing the tar to flow only through a single apparatus and all of the volatile constituents are driven off and the vapors fractionally condensed in several successive condensers arranged in series, it is difficult to control the temperature and amount of cooling water through the various condensers and thus maintain the desired composition of the resulting fractional condensates. This process, furthermore, presents difficulties in the regulation and maintenance of the successively increasing temperatures, similar to those referred to in the preceding paragraph.

According to the present invention, the distillation is carried out throughout in a continuous manner, but without the necessity of variable temperature control. The distillation retorts are arranged in series, and generally are all maintained at the same temperature. The continuation of the process is secured by operating each succeeding apparatus under a higher vacuum than the preceding one. Between each two consecutive retorts an open vessel is arranged from which the flow is effected into the next retort by the vacuum therein. The arrangement is such that in the last retort, the maximum possible vacuum can be maintained, and for this purpose a head of liquid is maintained in the inlet from the open vessel immediately preceding equal to or greater than that corresponding to a perfect vacuum. The inflow into each apparatus is thus automatically controlled and can never be greater than the outflow from the preceding retort into the intermediate vessel. The first retort is usually maintained at atmospheric pressure, this being the easiest pressure to obtain, and being sufficiently constant for practical purposes. The successive retorts are maintained under progressively increasing vacua, obtained by maintaining a suitable differential pressure or head of liquid between each retort and the next succeeding retort, or by independent vacuum-producing appliances.

In the accompanying drawings, Figure 1 shows an apparatus illustrative of the present invention; and Fig. 2 shows a modified form of apparatus.

The simplest form of the apparatus of the present invention is that consisting of only

two retorts, one maintained at atmospheric pressure and the other at as high a vacuum as can possibly be obtained by vacuum producing means and maintained by the differential head of liquid in the inlet to the retort above that in the intermediate vessel. Such an apparatus and arrangement of retorts is illustrated diagrammatically in Fig. 1 of the accompanying drawings.

The tar is admitted through pipe 1, and is forced in a steady stream by pump 2 up through pipe 3 into funnel 4, from which it flows—up through pipe 5, where it becomes preheated by the hot outflowing contents of pipe 20—into the first retort 6, heated by a high pressure steam coil 7. Here the lower boiling constituents of the tar are distilled off, *e. g.*: ammonia, benzol, toluol, water, etc., and escape through pipe 8 to condenser 9. The distillation in this retort is effected at atmospheric pressure, the flow through the retort being maintained by the height of the inlet 4 over that of the retort outlet. From the retort the remaining tar flows by gravity through pipe 10 into the intermediate vessel 11, open to the atmosphere, and of a depth of about 10 meters. From this intermediate vessel 11 the second apparatus, which is constantly maintained under a vacuum by means of the air pump 19, draws the tar automatically through the long pipe 12 which extends to the bottom of vessel 11 and is at least 11 meters long, into the second retort 13, heated by the high pressure steam coil 14. Here the tar, under the influence of the vacuum, loses its higher boiling constituents, such as naphthalene, carbolic oil and creosote oil, which escape through the condenser 15 where they are liquefied and flow out freely through long pipe 16, of a length of 11 meters into the receptacle 17, provided with outlet 18. The refuse tar runs off from the retort through pipe 20, also 11 meters long, and preheats counter-currently the crude tar entering through pipe 5, being itself cooled; and then runs off into receptacle 21 provided with outlet 22.

The length of the three pipes 12, 20 and 16 is about 11 meters or more, and corresponds to a column of liquid greater than would be balanced by a perfect vacuum (76 cm. of mercury). Accordingly the highest vacuum practicable in ordinary operation will be balanced by the heads of liquids in these three pipes, these heads varying somewhat with the degree of vacuum, but it never being posible to draw back liquid from vessel 17 through pipe 16, or from vessel 21 through pipe 20.

The operation of the second retort is as follows: When the vacuum pump is working the vacuum corresponds to the difference in levels of the liquid in the retort 13 (that of the retort outlet) and that in the intermediate receptacle 11, and is measured by the column of tar in pipe 12 between these levels. As the liquid level rises in the intermediate receptacle 11, due to the inflow from retort 6, this difference in level tends to decrease, and tar is accordingly drawn into the second retort 13 by the vacuum. As long as the tar pump 2 and the vacuum pump are running regularly the inflow and outflow of the first retort will be regular and the outflow will be flowing continuously into the intermediate receptacle. From this receptacle the tar will also be regularly drawn into the second retort 13, and the liquid residue and distillate regularly withdrawn. The flow through the second retort is thus maintained by the difference between the liquid level of the receptacle 11 and that of the receptacles 17 and 21, combined with the selective action of the vacuum which can draw the tar from the receptacle 11 when the level in this receptacle is not too low, but cannot draw it from the receptacles 17 and 21.

It will thus be seen that the apparatus is entirely automatic, as long as the tar pump and vacuum pump are running regularly, and that hardly any supervision is required. Should the vacuum pump stop for any reason, the second retort would gradually fill with air, and the liquid level in pipes 12, 20 and 16 would gradually fall to that of the receptacles 11, 21 and 17 respectively. Distillation in the second retort would stop, since tar could no longer be sucked in. Thereupon the liquid level in receptacle 11 would rise as tar flowed into it from retort 6 until it reached outlet 23, whence it would flow back to the tar pit. In such case the first retort would continue in operation, and the tar would be continually freed only from its more volatile constituents. Should at any time tar pump 2 stop, distillation in both retorts would stop, but would begin again automatically as soon as the trouble were removed and the pump again started.

In order that the temperature of the retorts may be kept constant, high pressure steam is supplied to the steam coils 7 and 14, and this steam may advantageously be of the same temperature and derived from the same source, through pipe 24. Temperature difficulties are thus avoided, and there can be obtained from each retort only the desired and expected distillate. The process is thus easily regulable, and once started can be allowed to continue automatically as long as the pumps are working and the steam heat is turned on. The operation of the process and the arrangement of the different retorts is the same when a plurality of retorts are used, that is, two or more than two. The arrangement is such that each successive retort is maintained under a progressively higher vacuum, and gives a distillate of cor-

respondingly higher boiling point at the same distillation temperature.

In Fig. 2 is shown an apparatus with three stills instead of 2, the intermediate still 13^a being similar in its construction and operation, to still 13, and being provided with a heating coil 14^a for steam at the same constant temperature as that used in the other stills. The condenser 15^a, outlet pipe 16^a, receptacle 17^a, still outlet pipe 20^a, and receptacle 11^a, without overflow outlet 23^a, are similar to the corresponding parts of the other stills. The stills are maintained under a progressively increasing vacuum by means of the suction pump 19, the reducing valve 24 enabling the difference in degree of vacuum in stills 13 and 13^a to be regulated as desired, the still 6 being maintained at atmospheric pressure the same as in the apparatus of Fig. 1.

The arrangement of the present invention is also such that the tar running off from the last apparatus of the series is used to heat the tar being fed to the first one. Thus in the specific embodiments of the invention shown, the tar flowing from retort 13, through pipe 20 heats counter-currently the fresh tar being fed into retort 6 by pipe 5. The hot tar residue is thus cooled before being discharged, and the cold tar thus preheated before reaching the retort.

The present apparatus and process are adapted for the treatment not only of tar from wood, lignite and coal, but also of petroleum, from which benzin and illuminating oils, or other fractions are easily separated, etc. This apparatus has the further advantage of minimizing and preventing any danger from fire.

Variations and modifications can be made from the foregoing description without departing from the spirit and scope of the invention, as set forth in the accompanying claims.

I claim:

1. The continuous process of fractional distillation at constant temperatures which comprises passing the liquid to be fractionally distilled successively and continuously through a series of retorts heated to constant temperatures and maintained under progressively increasing vacua, and continuously removing from each retort the distillate formed therein and the undistilled liquid.

2. The continuous process of fractional distillation at constant temperatures which comprises supplying the liquid to be distilled to a distillation retort heated to such temperature and maintained at about atmospheric pressure, continuously removing the distillate from such retort, removing the undistilling liquid in a continuous manner from said retort to another retort maintained at such temperature and at a decreased pressure and continuously removing the distillate and undistilled liquid for said last mentioned retort.

3. The continuous process of fractional distillation at constant temperatures which comprises supplying the liquid to be distilled to a distillation retort heated to such temperature and maintained at about atmospheric pressure, continuously removing the distillate from such retort, removing the undistilled liquid in a continuous manner from said retort and supplying the same through a suitable conduit to another retort maintained at such temperature and under a high vacuum, maintaining a head of liquid in the supply conduit corresponding approximately to the vacuum employed, and continuously removing the distillate and the undistilled liquid from said last mentioned retort.

4. The continuous process of fractional distillation at constant temperatures which comprises passing the liquid to be fractionally distilled successively and continuously through a series of retorts maintained under progressively increasing vacua, continuously removing from each retort the distillate formed therein, continuously removing from each retort the undistilled liquid, and supplying the liquid to each retort and removing the liquid therefrom through conduits containing heads of liquids corresponding to the vacuum in such retorts.

5. An apparatus for the continuous distillation of oils, comprising two or more retorts arranged in series, means for heating said retorts, means for continuously feeding oil to said retorts and for continuously removing the distillate and the residue from each of said retorts separately, and means for maintaining each successive retort under a lower pressure than the preceding retort.

6. An apparatus for the continuous distillation of oils comprising two or more retorts arranged in series, means for heating said retorts, means for maintaining each successive retort at a lower pressure than the preceding retort, an open vessel between each two successive retorts arranged to receive the oil flowing from the preceding retort, and means for conveying said liquid at said rate of outflow into the succeeding retort by the lower pressure therein.

7. An apparatus for the continuous distillation of oils, comprising two retorts arranged in series, heating means for said retorts, one of said retorts being maintained at atmosphereic pressure, and the other of said retorts under a vacuum, means for feeding oil regularly to said first mentioned retort and for removing therefrom the distillate and remaining oil, an open receptacle arranged to receive the outflowing oil, and a pipe about 11 meters long leading from

the bottom of said receptacle and adapted to convey oil therefrom under the influence of the vacuum to said vacuum retort, and means for removing from said vacuum retort the distillate and residual oil.

8. An apparatus for the continuous distillation of oils comprising two or more distillation retorts arranged in series, heating means for heating said retorts, means for maintaining each succeeding retort under a lower pressure than the next preceding retort, and means for utilizing the oil running off from the last apparatus of the series to preheat the oil being fed to the first apparatus.

9. An apparatus for the continuous distillation of oils comprising two stills arranged in series, heating coils for heating said stills by high pressure steam, means for feeding oil regularly to the first of said retorts at atmospheric pressure, means for removing from said retort the distillate and residual oil, a vacuum pump connected to the second of said retorts for maintaining a vacuum therein, and for removing therefrom the distillate, a pipe about 11 meters long, for removing from said vacuum retort the residual oil, a receptacle about 10 meters high, intermediate retorts arranged to receive the oil flowing from said first retort, and a pipe extending from near the bottom of said receptacle through a vertical distance of about 11 meters to said vacuum retort and adapted to convey the oil to said vacuum retort under the influence of the vacuum pump.

In testimony whereof, I have hereunto set my hand in the presence of two subscribing witnesses.

FRITZ RASCHIG.

Witnesses:
ROBERT GERLACH,
JOSEPH FEIFFER.

Correction in Letters Patent No. 1,141,265.

It is hereby certified that in Letters Patent No. 1,141,265, granted June 1, 1915, upon the application of Fritz Raschig, of Ludwigshafen-on-the-Rhine, Germany, for an improvement in "Processes and Apparatus for Continuous Distillation," an error appears in the printed specification requiring correction as follows: Page 3, line 67, for the word "for" read *from;* and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 29th day of June, A. D., 1915.

[SEAL.]

J. T. NEWTON,

Acting Commissioner of Patents.