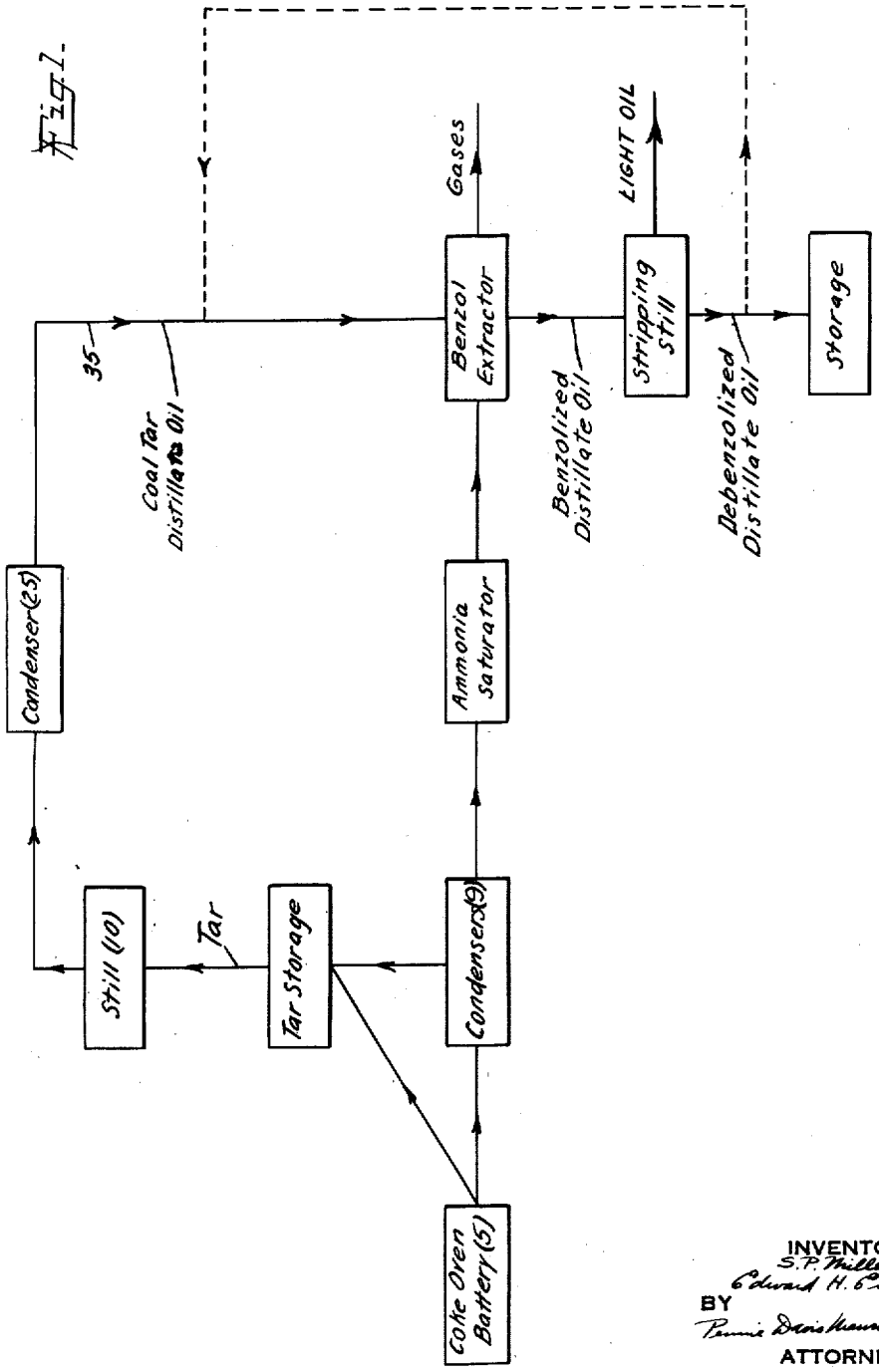
June 28, 1932. S. P. MILLER ET AL 1,864,749

RECOVERY OF LIGHT OILS

Filed Feb. 18, 1930 3 Sheets-Sheet 1



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Filed Feb. 18, 1930 3 Sheets-Sheet 2

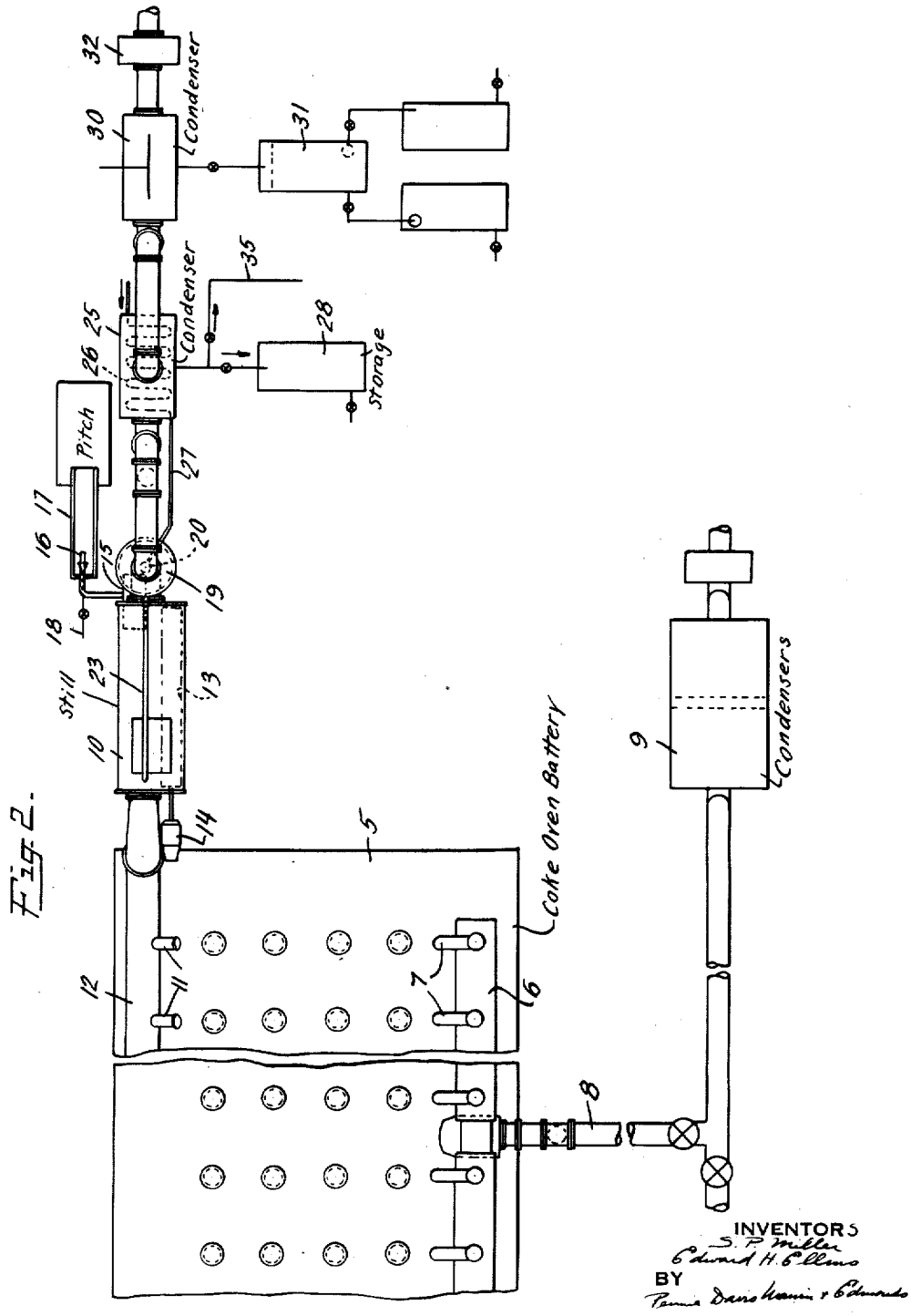

Fig 2.

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RECOVERY OF LIGHT OILS

Filed Feb. 18, 1930 3 Sheets-Sheet 3

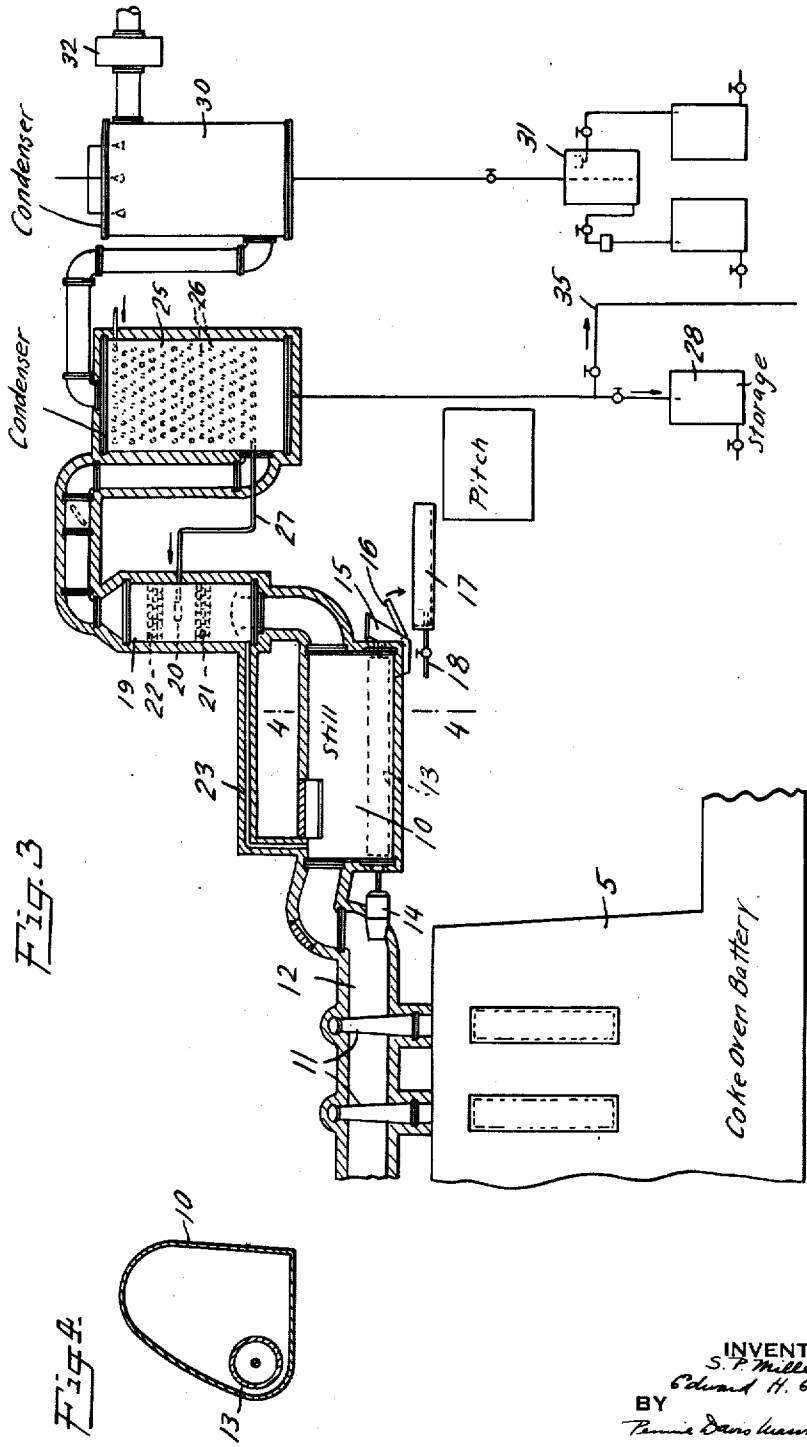

Fig. 3

Fig. 4.

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UNITED STATES PATENT OFFICE

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RECOVERY OF LIGHT OILS

Application filed February 18, 1930. Serial No. 429,304.

This invention relates to the recovery of light oil from coal distillation gases, such as coke oven gases and gas retort gases. The invention relates more particularly to the use of a high boiling coal tar oil as a wash oil for absorbing light oil from coal distillation gases with recovery of the light oil from the wash oil and the use of the resulting high boiling coal tar oil as creosote oil, etc.

Coal distillation gases such as the gases from a coke oven plant or a gas retort plant are ordinarily cooled to separate condensable constituents and then after passing the gases through an ammonia absorber the gases are ordinarily washed with a high boiling oil to remove the vapors of oils of low boiling range which, alone, are condensable, but which are present in the coal distillation gases at such dilution that they are not condensable from the gas mixture at temperatures practically attainable in plant operation. The oils of low boiling range are known as "light oil." They consist chiefly of aromatic hydrocarbons with a boiling range from about 65° C. to 180° C., although the boiling range may vary depending upon the operation of the coal distillation plant. The light oil may contain some unsaturated compounds and also paraffin hydrocarbons, depending upon the coal distilling operation.

Straw oil, a high boiling, limpid petroleum product, is often employed as a wash oil for absorbing the light oil from the coal distillation gases. After the gases leave the ammonia absorber they are passed through one or more absorption towers through which straw oil is allowed to trickle. The absorption towers expose a large surface of the wash oil to the gases, and the light oil present in the gases is absorbed by the oil. The operation is a countercurrent operation so that the fresh oil is brought into direct contact with the gases from which most of the light oil has been absorbed. The straw oil which has been used to absorb the light oil from the gases, which is referred to as "benzolized wash oil," is then heated to distill off the light oil. It is then referred to as "debenzolized wash oil." The light oil distillate may be treated for the production of desired light oil constituents, such as motor fuel, benzene, toluene, xylenes, solvent naphtha, etc. The debenzolized straw oil is then used again for the absorption of further light oil from the gases. Instead of straw oil other oils may be employed which have a sufficiently high boiling point to permit recovery of the light oil by distillation from the wash oil.

In the past difficulties have arisen in operating light oil recovery systems; owing to the fact that the wash oil removes from the gases higher boiling constituents such as naphthalene, coumarone, indene, etc. which are not completely volatilized at the relatively low temperature employed for separating the light oil from the wash oil, the wash oil eventually becomes contaminated with such substances and their decomposition and polymerization products, which result from heating the wash oil to distill off the light oil. Straw oil or other wash oil which has been in use for some little time becomes contaminated with such foreign substances, and it is necessary to purify the wash oil at intervals. This necessitates keeping a considerable stock of the wash oil on hand, and involves distillation costs, etc. which we now find are unnecessary.

According to this invention, we employ as a wash oil a coal tar distillate of relatively high boiling range, for example 250–300° C. and upwards. This is employed in the way that straw oil or any other wash oil would be employed to remove light oil from the gases. It is a more effective absorbent light oil than the absorbing oils generally employed. Coal tar distillate which has been used for absorbing light oil, generally referred to as "benzolized wash oil", i. e. benzolized distillate is then heated to distill off the light oil. The resulting debenzolized creosote oil may then be disposed of as creosote oil directly, or it may be employed in the preparation of coal tar solutions directly, or by recirculating it through the light oil absorbing towers it may be reused several times and then be disposed of as above, or otherwise as a coal tar product. In any case according

to my invention the necessity is eliminated of subjecting the absorbing oil to purification or refining to remove undesired substances absorbed or derived from the gases. Such undesired substances if accumulated in straw oil, for example, would eventually prevent further use of the straw oil as a wash oil unless such substances were first removed as by distillation.

According to a preferred method of carrying out the invention, tar recovered from the coal distillation gases or a portion of the tar recovered from the coal distillation gases is distilled at the coal distillation plant. The high boiling oil fraction, which may be the fraction with a boiling range from 250–300° C. and upward, for example, as it is produced, or a portion of the distillate of this boiling range, may be used directly in the light oil absorption process, and after being debenzolized it may be run to storage. It may be blended with other distillate of this boiling range or distillate of another range or it may be blended with tar to produce coal tar solution. According to this invention the debenzolized oil is sold directly as such, or it is blended as such, without being treated for the removal of naphthalene or condensation and polymerization products, etc. which may result from the light oil absorption and subsequent distillation to volatilize the light oil.

The coal tar distillate under special conditions may be used as wash oil only once; ordinarily it will advantageously be recirculated several times through the absorbing towers and then through the light oil still before being run to storage. One batch of distillate may be employed as wash oil for a whole day, for example, before being run to storage.

When straw oil or a wash oil other than coal tar distillate is used for absorbing light oil from coal distillation gases any distillation of the wash oil which may occur during the absorbing operation will cause the presence of constituents of the straw oil or other wash oil in the coal distillation gases as a foreign substance. Similarly, any of the straw oil or other wash oil which may distill over in the stripping still in which the light oil is distilled from the wash oil will be present in the light oil distillate as a foreign substance largely of a paraffine-like nature. When a coal tar oil is used, however, any distillate from the coal tar oil which goes over with the gases or with the light oil is not a foreign substance since the coal tar distillate is derived originally from coal distillation gases; a purer and more valuable light oil is therefore obtained when the wash oil is coal tar distillate.

Further, the tendency of the coal tar distillate to distill in the absorbing towers is negligible since the gases are already saturated with constituents of the coal tar distillate. By removal of light oil from the gases, the volume of the gases is reduced and the amount of higher boiling constituents necessary to saturate the gases is likewise reduced. Instead of distilling any of the wash oil into the gases therefore, the tendency is for the volume of the wash oil to be increased somewhat, owing to condensation of higher boiling constituents from the gases. When coal tar distillate is employed as the wash oil, such increase does not introduce foreign substances into the wash oil. Any increase which is brought about in this manner increases the amount of coal tar distillate.

Ordinary coal distillation gases as they come from the ammonia absorbers contain fine suspended particles of tar, i. e. tar fog. Wash oil employed for scrubbing such ordinary gases removes at least a portion of the suspended tar particles from the gases and the tar contaminates the wash oil. Where coal tar distillate is employed for washing the gases to remove light oil, the distillate will become contaminated with traces of tar. Distillate which has been so used and which is contaminated with a small amount of tar may be employed directly for the preparation of coal tar solution by blending with coal tar in the proper proportions after first being debenzolized.

If the coal tar distillate used for recovering light oil from the gases is to be disposed of as a clean oil product, for example clean creosote oil, it may be desirable to clean the gases in an electrical precipitator before they are scrubbed with the coal tar oil. When the light oil absorbers are located beyond the ammonia saturator, the precipitator may be located between the ammonia saturator and the light oil absorber, or it may be located ahead of the ammonia saturator. The precipitator removes all entrained particles from the gases so that coal tar distillate used in the light oil absorbers will remain free from tar particles, etc., and can be disposed of as a clean coal tar product.

An electrical precipitator may be located beyond the light oil absorber to remove from the gases traces of oil which may be carried from the by-product recovery system as a suspension of small drops.

The apparatus for carrying out the invention may be a by-product recovery system of any usual type, including a light oil absorber and a stripping still for removing the light oil from the wash oil employed in the absorber.

The still employed for the distillation of tar may be any tar still, but according to a preferred method of carrying out the invention, a still in which the tar is distilled by direct contact with the hot coal distillation gases is employed.

The invention will be further described in

connection with the accompanying drawings.

Fig. 1 is a flow sheet illustrating the application of the invention to a coke oven battery;

Fig. 2 is a plan view showing a coke oven battery and one type of tar still which may be employed for distilling tar by direct contact with the coke oven gases;

Fig. 3 is an elevation of the apparatus shown in Fig. 2; and

Fig. 4 is a section on the line 4—4 of Fig. 3.

The numbers indicated in parentheses on the flow sheet correspond with the parts of the apparatus shown in Figs. 2 and 3.

The invention may be carried out in the usual by-product recovery apparatus with such additional apparatus as may be necessary for producing and handling the coal tar distillate. As indicated in Fig. 1, the coal distillation gases which in the drawings are represented as the gases from a coke oven battery are cooled to separate tar. The cooling may be effected in the ordinary collector main and condensers. The tar distilled may be all or a part of the total tar combined by mixing the tar from several sources, such as the collector main and condensers, or all or a part of any tar fraction may be distilled. The gases are ordinarily passed through an ammonia saturator or absorber in which water or sulfuric acid is used for extracting the ammonia before the gases pass to a benzol extractor and the apparatus shown provides for such removal of ammonia.

Any tar still suitable for producing distillate of the desired high boiling range may be used. The distillate or a portion of the distillate is employed for extracting the light oil in the so-called benzol extractor. From the benzol extractor the gases pass to a storage tank or to burners. The gases may when required be treated for the removal of sulfur compounds, etc., in the usual way.

The wash oil leaving the benzol extractor is commonly referred to as benzolized wash oil. It contains benzol and other light oil. It is introduced into a stripping still which may be any usual type of stripping still. Ordinarily a steam heated column still is employed in which live steam is also introduced to assist in volatilizing the light oil. Heat interchangers, etc. may be employed in the usual manner to recover heat from the vapors and from the non-volatilized wash oil, which in this case is creosote distillate with the addition of any naphthalene, or other high boiling impurities which are added to the distillate during the light oil extraction. This coal tar oil may then be run directly to storage to be disposed of as creosote oil or to be used for blending for the production of coal tar solution, etc. It may be blended with other coal tar distillate of high boiling range, which has not been used as wash oil, or all or a part of the distillate may be again passed through the benzol extractor and used for the recovery of further light oil.

The wash oil from which light oil has been removed in the stripping still is referred to as debenzolized wash oil. Instead of passing the debenzolized wash oil directly to storage, all or a portion of the oil may be recirculated as shown in dotted lines for reuse in the benzol extractor. Fresh wash oil, i. e. fresh distillate, is added to the cycle to replace any debenzolized oil which is drawn off to storage.

Figs. 2 and 3 show a preferred form of distilling apparatus. The coke oven battery is indicated at 5. It is equipped in the usual way with a collector main 6 which is connected with the individual ovens of the battery through uptake pipes 7. A cross-over main 8 connects the collector main with condensers 9. From the condensers the gases pass to the ammonia saturator and benzol extractor or wash oil absorber (not shown in Figs. 2 and 3). This apparatus may be of the usual type.

The tar still 10 is located on the opposite side of the coke oven block from the collector main. Selected ovens of the battery are provided with uptake pipes 11. A hot gas header 12 connects these uptake pipes with the still 10. By proper manipulation of valves in the uptake pipes 7 and 11 the gases from selected ovens are passed into the still instead of into the collector main. In the still is a roll 13 which is driven at a high speed, for example 900–1200 R. P. M., by the motor 14. This sprays tar or pitch from the bottom of the still up into the hot coke oven gases passing through the still in the form of a fine intense spray which scrubs and detars the gases. Tar is distilled to pitch which is drawn off through the coke trap 15 and the levelling arm 16. The position of this levelling arm determines the depth of the tar and pitch maintained within the still and thus determines the nature of the tar spray produced by the roll 13. The pitch is drawn off into the trough 17 where it is chilled and granulated by a jet of cold water from the pipe 18.

The gases and vapors from the still 10 pass into the saturator 19. Tar is sprayed into the saturator thru the nozzle 20. Baffles 21 and 22 are provided to remove particles of tar entrained in the gases and to provide a large surface of contact between the tar sprayed in thru the nozzle 20 and the hot gases and vapors which pass up thru the saturator. The tar is distilled to semi-pitch in this saturator. The semi-pitch is drawn off from the bottom of the saturator thru the main 23 and introduced into the still at the end at which the hot gases enter from the header 12. This partially distilled tar or semi-pitch is further distilled in the still

and a pitch of high melting point may readily be produced.

The gases and vapors from the saturator pass into the heat interchanger 25. The tar to be distilled, or a portion of this tar, is passed thru the heat interchanger thru the coil 26 where it is preheated, and it is conveyed by the pipe 27 to the nozzle 20. The hot gases and vapors are partially cooled in this heat interchanger and oil is condensed. The cooling in the heat interchanger is so regulated that the oil fraction obtained and drawn off into the tank 28 is a coal tar distillate of high boiling range, for example 250–275° C., or higher.

The partially cooled gases and vapors which leave the heat interchanger 25 are further cooled in the condenser 30. This may be any suitable type of condenser, either a direct condenser or indirect condenser. Distillate of low boiling range which is condensed in the condenser 30 is drawn off into the tank 31. From this condenser the gases pass thru an exhauster 32 to storage or to burners. The storage tank may be the same storage tank in which the gases from the collector main 6 are stored.

All or a portion of the heavy creosote oil is drawn off thru the line 35 to the benzol extractor employed for removing light oil from the gases which have passed thru the condensers 9. The resulting benzolized oil passes to a stripping still where the light oil is removed. The resulting debenzolized oil may then be drawn off to storage. If only a part of the distillate condensed in the condenser 25 is employed as wash oil, the debenzolized wash oil may be blended with the rest of the distillate in the tank 28. A separate storage tank may be provided for the debenzolized wash oil, or it may be run directly to a blending tank for blending with coal tar, etc., to produce coal tar solution, etc.

In the claims the reference to drawing off the debenzolized oil to storage is to be interpreted to mean collecting the debenzolized wash oil for disposal as creosote oil, for blending with other coal tar distillate to be disposed of as creosote oil, etc., or for blending with other materials to produce coal tar products, such as coal tar solution.

We claim:

1. The method of recovering light oil from coal distillation gases, which comprises cooling the coal distillation gases to separate a coal tar, distilling the coal tar so as to produce a coal tar distillate of high boiling range, bringing this distillate into direct contact with the coal distillation gases after cooling, and after the removal of ammonia, to absorb light oil therefrom, stripping the resulting solution of light oil in coal tar distillate to remove light oil, and running the resulting oil to storage.

2. The method of recovering light oil from coal distillation gases, which comprises cooling the coal distillation gases to separate a coal tar, distilling the coal tar so as to produce a coal tar distillate of high boiling range, bringing this distillate into direct contact with the coal distillation gases after cooling and after the removal of ammonia to absorb light oil therefrom, stripping the resulting solution of light oil in coal tar distillate to remove light oil, reusing at least a portion of the resulting oil for further extraction of light oil and stripping it to remove light oil, and running the resulting oil to storage.

3. The method of recovering light oil from coal distillation gases, which comprises bringing a high boiling coal tar distillate into direct and intimate contact with the gases to absorb light oil therefrom, separating the light oil from the high boiling distillate by distillation, and reusing at least a portion of the resulting oil for further extraction of light oil from coal distillation gases, and repeating the operation and running the resulting oil from which light oil has been distilled to storage before it becomes contaminated with undesirable constituents to such an extent that it is no longer useful for creosoting purposes.

4. The method of recovering light oil from coal distillation gases which have been cleaned in an electrical precipitator, which comprises bringing high boiling coal tar distillate into direct and intimate contact with the gases to absorb light oil therefrom, separating the light oil by distillation and reusing at least a portion of the resulting oil for further extraction of light oil from coal distillation gases, and repeating the operation and running the oil from which light oil has been distilled to storage before it becomes contaminated with undesirable constituents to such an extent that it is no longer useful as coal tar distillate.

5. The method of recovering light oil from coal distillation gases, which comprises bringing cooled coal distillation gases into direct contact with high boiling coal tar distillate so as to dissolve light oil therein, heating the distillate to distill off the light oil, reusing at least a portion of the resulting oil for further extraction of light oil from the gases, and, finally, after removing light oil from the distillate, blending the resulting debenzolized oil with tar to produce coal tar solution.

6. The method of recovering light oil from coal distillation gases, which comprises continuously circulating coal tar distillate through a benzol extractor to bring the distillate into direct contact with the coal distillation gases and absorb light oil from these gases and thus produce a benzolized oil, continuously heating the benzolized oil to distill off the light oil and produce a debenzolized oil, continually drawing off a portion of the

debenzolized oil to storage, adding fresh coal tar distillate to the remainder of the debenzolized oil and again circulating it through the benzol extractor.

7. In combination with a coal distillation plant and by-product recovery system, a still, means for supplying tar recovered from the coal distillation gases to the still, a condenser for condensing from the resulting distillate vapors a creosote oil of high boiling range, means for introducing the creosote oil as a wash oil into the benzol extractor of the by-product recovery system, a stripping still connected with the extractor for the removal of benzol from the wash oil and means for running the debenzolized wash oil from the stripping still of the by-product recovery system to storage.

8. In combination with a coke oven battery, coolers, an ammonia saturator and a benzol extractor for treating the coal distillation gases, a still, means for passing coal distillation gases from a portion of the plant through the still, means for introducing tar recovered from the gases into the still and distilling it therein, a condenser for coal tar distillate of high boiling range, means for introducing this distillate into the benzol extractor, a stripping still, means for introducing benzolized wash oil from the benzol extractor into the stripping still, and means for running benzolized wash oil from the stripping still to storage.

9. In combination with a coke oven battery, coolers, an ammonia saturator and a benzol extractor for treating the coal distillation gases, a still, means for passing coal distillation gases from a portion of the plant through the still, means for introducing tar recovered from the gases into the still and distilling it therein, a condenser for coal tar distillate of high boiling range, means for introducing tar recovered from the gases into the still, and distilling it therein, a condenser for coal tar distillate of high boiling range, means for introducing this distillate into the benzol extractor, a stripping still, means for recirculating the benzolized wash oil through the benzol extractor, and means for running debenzolized wash oil to storage.

10. In combination with the by-product recovery system of a coal distillation plant comprising a benzol extractor and a stripping still, a still for distilling tar recovered from the coal distillation gases, a condenser for recovering coal tar distillate of high boiling range, a storage tank, means for running a portion of this coal tar distillate of high boiling range to the storage tank, means for introducing the balance into the benzol extractor, and means for introducing debenzolized wash oil from the stripping still into the storage tank.

11. The method of producing coal tar oil at a coal distillation plant and recovering light oil from the coal distillation gases produced at the plant, which comprises producing from the coal distillation gases a coal tar oil fraction substantially free from lower boiling oils, using at least a portion of this fraction for the recovery of light oil from the coal distillation gases by scrubbing the gases therewith after cooling them, separating light oil from the fraction by distillation and running the resulting oil to storage.

12. The method of producing coal tar oil at a coal distillation plant and recovering light oil from the coal distillation gases, which comprises producing from the gases, pitch and coal tar oil including a coal tar oil fraction substantially free from lower boiling oils, dividing the fraction into at least two parts, using one part of the fraction for removing light oil from the gases after cooling the gases, heating the resulting solution of the light oil in the fraction to distill off light oil and adding the resulting oil to another part of the fraction.

13. The method of producing coal tar oils at a coal distillation plant and recovering light oil from the coal distillation gases, which comprises producing from the coal distillation gases pitch and coal tar oil, dividing the oil into at least two parts, using one part of the oil for removing light oil from the gases after cooling the gases, distilling light oil from the resulting solution of light oil in coal tar oil and adding the residual oil to another part of the oil.

14. The method of recovering light oil from coal distillation gases, which comprises dividing a stream of coal tar oil into two parts, using one part of the oil for removing light oil from the coal distillation gases, distilling light oil from the resulting solution and adding the residual oil to the stream.

15. The method of recovering light oil from coal distillation gases at a coal distillation plant, which comprises producing from the coal distillation gases a substantially clean coal tar oil substantially free from lower boiling oils, passing a part of the oil directly to storage, using another part of the oil for dissolving light oil from the coal distillation gases after cooling them, distilling light oil from the resulting solution and then passing the oil remaining after such distillation to the same storage.

16. The method of producing coal tar oil at a coal distillation plant and recovering light oil from the coal distillation gases, which comprises producing from the coal distillation gases pitch and coal tar oil, using a part of the coal tar oil for the removal of light oil from the gases, distilling light oil from the resulting solution and blending the resulting oil with other coal tar oil which has not been used in light oil recovery.

17. The method of producing coal tar oil at a coal distillation plant and recovering

light oil from the coal distillation gases which comprises producing from the coal distillation gases pitch and substantially clean coal tar oil, using a coal tar oil fraction substantially free from lower boiling constituents for recovering light oil from the gases by a cyclic process in which the oil is used for extracting the light oil from the gases, light oil is distilled from the resulting solution and the resulting residual oil is at least in part recycled for light oil extraction, adding to the cycle fresh coal tar oil produced at the plant and blending residual oil from the cycle with coal tar oil produced at the plant and which is not used for light oil extraction.

18. In combination with a coal distillation plant, means for separating the constituents of coal distillation gases into pitch and clean oil, benzol extraction apparatus with means for passing coal distillation gases from the plant therethrough, a benzol stripping still connected with the benzol extraction apparatus, oil storage means connected with the means for producing clean oil and means for passing a part of the clean oil from the clean oil producing means through the benzol extraction apparatus and returning it from the stripping still to the storage means.

In testimony whereof we affix our signatures.

S. P. MILLER.
EDWARD H. ELLMS.

CERTIFICATE OF CORRECTION.

Patent No. 1,864,749. **June 28, 1932.**

STUART PARMELEE MILLER ET AL.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 1, line 87, before the word "light" insert the word for; 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 27th day of September, A. D. 1932.

(Seal)

M. J. Moore,
Acting Commissioner of Patents.

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18. In combination with a coal distillation plant, means for separating the constituents of coal distillation gases into pitch and clean oil, benzol extraction apparatus with means for passing coal distillation gases from the plant therethrough, a benzol stripping still connected with the benzol extraction apparatus, oil storage means connected with the means for producing clean oil and means for passing a part of the clean oil from the clean oil producing means through the benzol extraction apparatus and returning it from the stripping still to the storage means.

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