

F. SPARRE AND W. E. MASLAND.

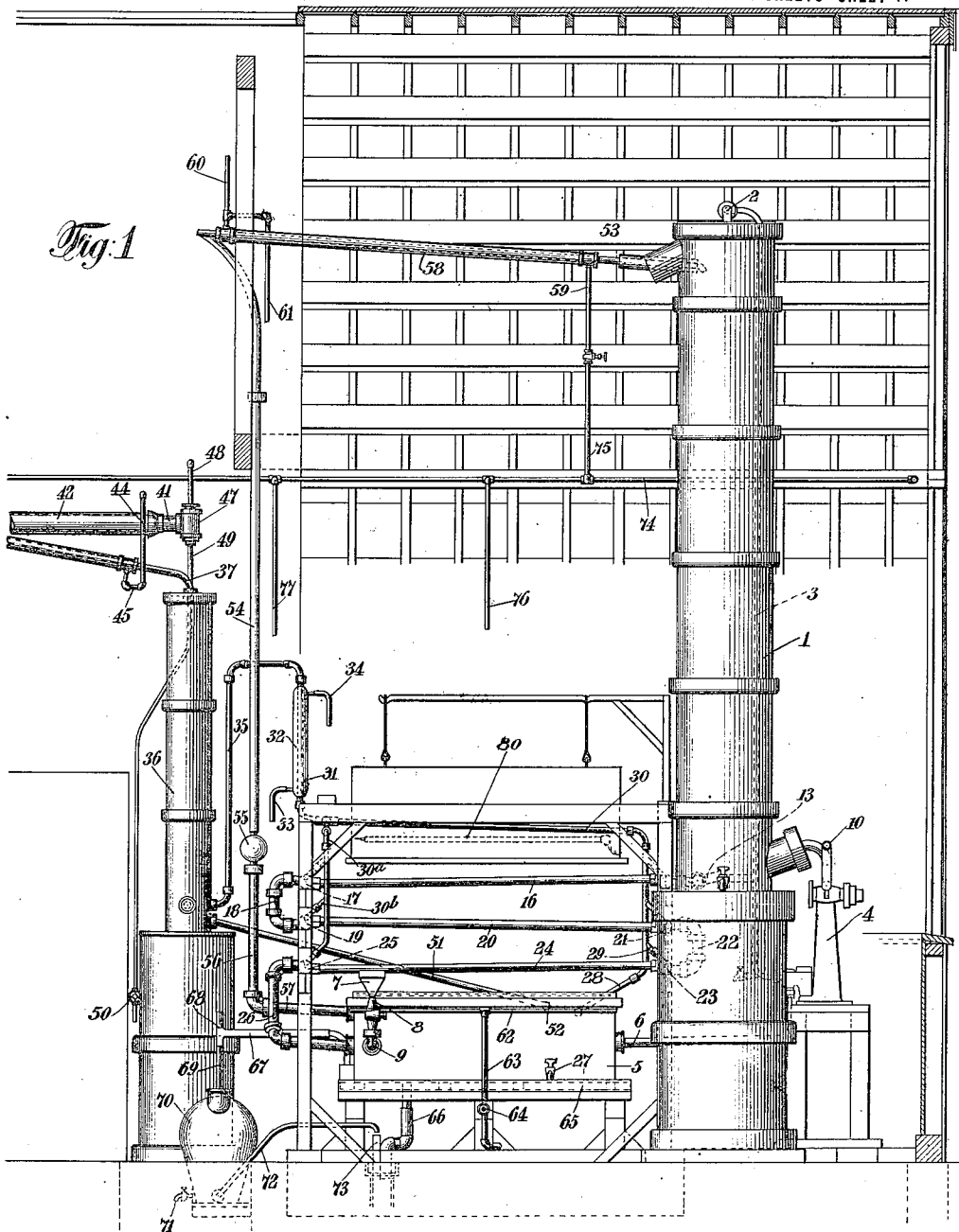
PROCESS OF CHLORINATION.

APPLICATION FILED OCT. 31, 1912. RENEWED MAR. 1, 1917.

1,379,367.

Patented May 24, 1921.

3 SHEETS—SHEET 1.



WITNESSES

J. Friendenoll
L. Broderick

INVENTOR

F. Sparre and
Walter E. Masland

BY

Princedbright
ATTORNEYS

F. SPARRE AND W. E. MASLAND.

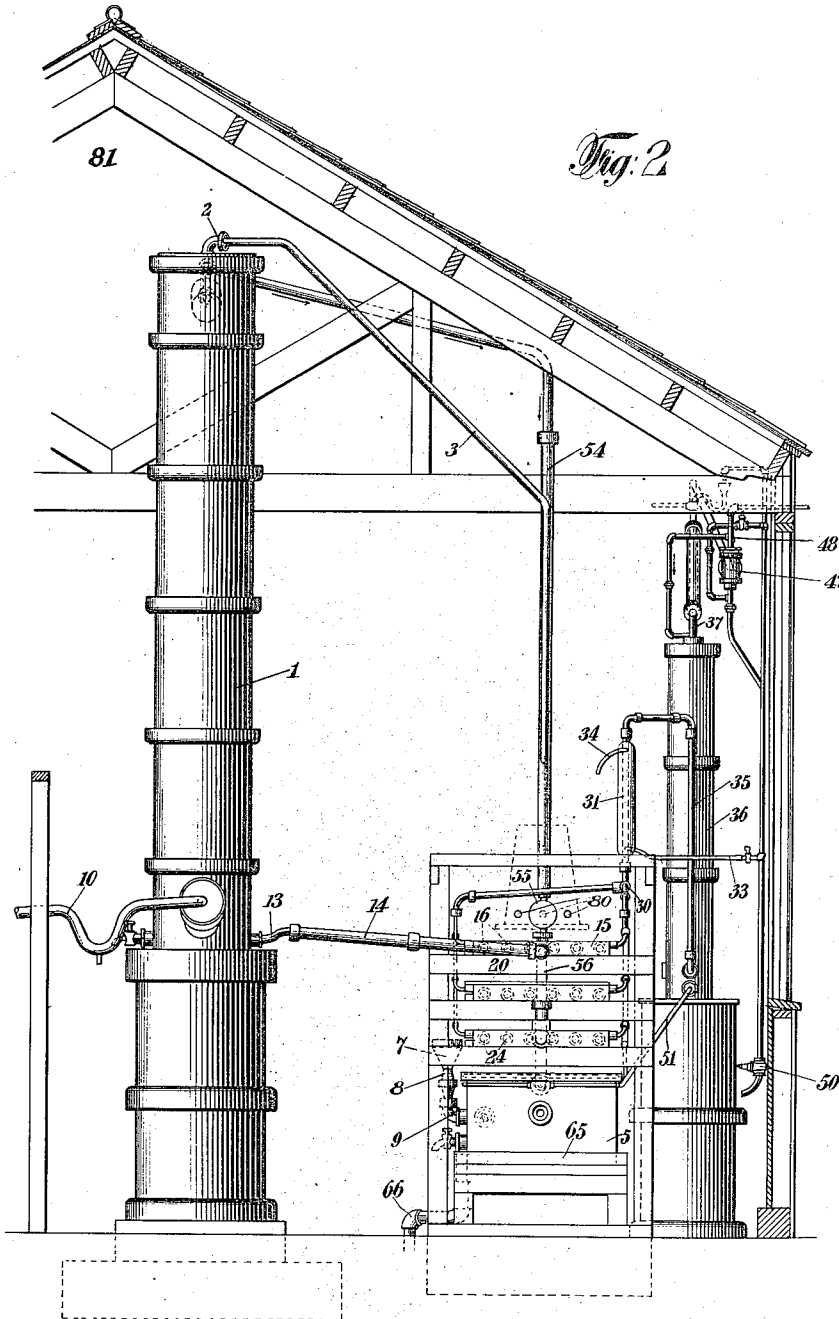
PROCESS OF CHLORINATION.

APPLICATION FILED OCT. 31, 1912. RENEWED MAR. 1, 1917.

1,379,367.

Patented May 24, 1921.

3 SHEETS—SHEET 2.



Witnesses:
J. Freundewoll
L. Broderick

Inventor
F. Sparre and Walter E. Masland
By their Attorneys
Orin Lee Wright

F. SPARRE AND W. E. MASLAND.

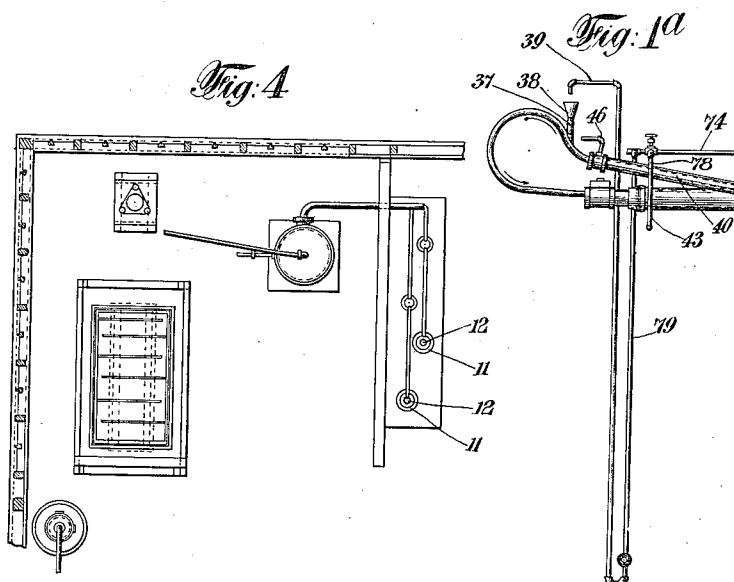
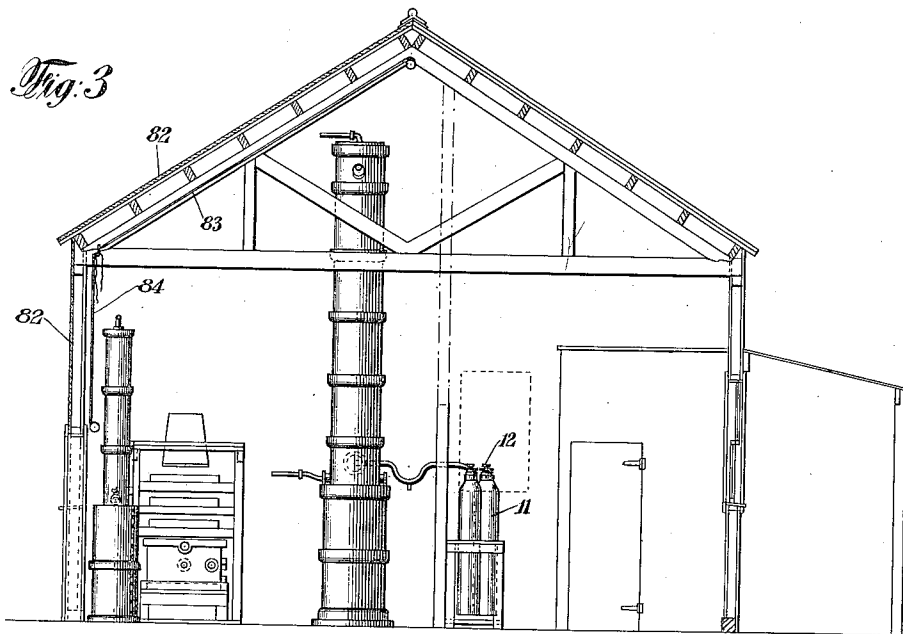
PROCESS OF CHLORINATION.

APPLICATION FILED OCT. 31, 1912. RENEWED MAR. 1, 1917.

1,379,367.

Patented May 24, 1921.

3 SHEETS—SHEET 3.



WITNESSES

J. Freundewoll
L. Broderick

INVENTOR

F. Sparre and
W. E. Masland
BY
Prindle, Wright
ATTORNEYS

UNITED STATES PATENT OFFICE.

FIN SPARRE AND WALTER E. MASLAND, OF WILMINGTON, DELAWARE, ASSIGNORS
TO E. I. DU PONT DE NEMOURS & COMPANY, OF WILMINGTON, DELAWARE, A COR-
PORATION OF DELAWARE.

PROCESS OF CHLORINATION.

1,379,367.

Specification of Letters Patent.

Patented May 24, 1921.

Application filed October 31, 1912, Serial No. 728,887. Renewed March 1, 1917. Serial No. 151,845.

To all whom it may concern:

Be it known that we, FIN SPARRE and WALTER E. MASLAND, of Wilmington, in the county of Newcastle and in the State of Delaware, have invented a certain new and useful Improvement in Processes of Chlorination, and do hereby declare that the following is a full, clear, and exact description thereof.

Our invention relates particularly to a process of chlorination or halogenation of saturated hydrocarbons, that is to say, producing a chlorin or halogen derivatives by treatment with chlorin or halogen respectively, although it is applicable also to the chlorination of many other compounds.

The object of our invention is to provide a process of the kind above referred to, in which the process of chlorination may be carried out continuously and with a great degree of rapidity, but with safety.

Hitherto, the process of chlorination has been ordinarily carried out by passing chlorin into a receptacle partly filled with the hydrocarbons, and subjecting the same to the action of chlorin carriers such as iodine, or to light, or both, in such a way that free spaces have been present over the body of liquid, thereby allowing the accumulation of gases over the surface of the liquid, or the reaction has been even conducted between the gases themselves. It has been found that when carried out in this manner the process frequently resulted in an explosion, owing to the fact that the reaction is effected at times very rapidly, and when a reaction takes place rapidly between two gaseous constituents, it is likely to result in an explosion.

Our invention comprises a process which will give better yields, a better control of operations, and avoid the possibility of such explosions. In accomplishing this result, we cause the chlorin to be absorbed in the hydrocarbons in a dark chamber. Inasmuch as the reaction between the chlorin and the saturated hydrocarbon takes place only in the presence of light, it will be seen that the absorption is effected under conditions which will avoid any reaction between the chlorin and the hydrocarbon. Under these circumstances, there cannot be any cause for an explosion during the absorption of the chlorin, as the explosions which have hither-

to taken place have been due to conditions which have permitted a violent reaction between the chlorin and the hydrocarbons. The chlorin having been absorbed by the hydrocarbons, the liquid is then conducted to a chamber in which it is subjected to the action of light. This chamber is so arranged as to avoid the presence of appreciable free spaces above the body of the liquid. So long as the reaction is effected within the body of the liquid, the danger of an explosion is eliminated, as the reaction could not, by any possibility, take place so violently or with local heating to the ignition point within the body of the liquid as to result in an explosion. The reaction within the chamber referred to is carried out with a great degree of rapidity as we use, in connection therewith, sunlight or a source of blue light which has been found to be the most active in effecting the chlorination. Furthermore, the reaction is carried out without the necessity of any additional re-agents and without the presence of any chlorin carriers such, for example, as iodine or sulfur. In fact, we have discovered that chlorin carriers are disadvantageous in effecting the reaction, owing to the possible danger in their action and difficulty in controlling the process. The process is, furthermore, carried out in such a manner that there are no mechanical losses, the hydrochloric acid produced in the reaction being recovered and any chlorin and hydrocarbon vapors that escape during the reaction being returned and utilized. The process is, furthermore, carried out in an apparatus in which it can be readily regulated and with a minimum amount of labor. In carrying out the process, it is merely necessary to control the amount of light admitted to the reaction chamber and to control the amount of chlorin admitted into the absorption chamber. These two manipulations can be readily taken care of by one attendant. In order to carry out the process, we have provided an apparatus which effects all of the results above referred to, and which has many new features which make it advantageous and valuable in the process of chlorination.

While we may make use of many different kinds of apparatus for carrying out the process, for the purposes of illustration and

as a preferred embodiment of the apparatus which may be used, we have shown only one form of said apparatus in the accompanying drawings, in which—

5 Figure 1 is a side elevation of a portion of an apparatus used for carrying out the process;

Fig. 1^a is a similar view of the remainder of said apparatus;

10 Fig. 2 is an end elevation of the apparatus shown in Figs. 1 and 1^a;

Fig. 3 is an end elevation of the entire plant containing said apparatus, showing the parts somewhat diagrammatically; and,

15 Fig. 4 is a plan view of a portion of the same.

In the drawings we have shown an opaque tower 1 having any suitable interior construction to effect the absorption of a gas in a liquid. This absorption tower has an inlet 2 at its top for liquid hydrocarbon, said inlet being connected to a discharge pipe 3 leading from a stoneware plunger pump 4 which withdraws the liquid from a tank 5 by means of a pipe 6. The tank 5 is charged with liquid through a lead funnel 7 connected to a stoneware stop-cock 8 supported in the outer end of an inlet pipe 9 leading into said tank. The stoneware pump 4 is operated in any suitable manner to force a continuous current of the liquid into the top of the absorption tower 1. Near the bottom of the tower there is provided an inlet pipe 10 for chlorin gas, leading from a series of chlorin bottles, 11, containing liquid chlorin. Stop-cocks 12 are provided on the tops of these bottles to permit the flow of chlorin gas to be readily regulated. The hydrocarbon liquid having absorbed the chlorin gas, it is conducted out of the tower 1 at its base by means of a glass tube 13 leading into a stoneware pipe 14. The pipe 14 conducts the liquid to a manifold 15, to which are attached a plurality of inclined glass tubes 16, in order to subject the liquid to the action of light. The liquid flowing through the tubes 16 is then conducted through a manifold 17 and by means of a pipe 18 to a manifold 19, another similar set of glass tubes 20, a manifold 21, a pipe 22 similar to pipe 18, a manifold 23, a similar set of glass tubes 24, and a manifold 25. As the liquid passes through the glass tubes 16, 20 and 24, the chlorin gas becomes combined with the hydrocarbons, giving off hydrochloric acid, and the process is so regulated that the yellow color of the chlorin gas will have disappeared when the liquid has passed out of the lowermost set of glass tubes 24. From the manifold 25, the liquid is conveyed by a pipe 26 to the tank 5, which may have a glass cover, if desired to increase the action of the light, and which is provided with a draw-off cock 27 to draw the liquid off when the reaction

carbons have been sufficiently chlorinated. The apparatus is usually operated in such a manner, however, as to circulate the liquid through the absorption tower and the glass tubes referred to until the required degree of chlorination has been effected. Any gas which accumulates in the tank 5, which is principally hydrochloric acid gas, is conducted away by means of a pipe 28. This pipe 28 has connected to it a series of branch pipes 29, one of which communicates with each of the manifolds 15, 21 and 23 in order to convey away from said manifolds any gases which have accumulated during the reaction. The gases which have been thus obtained from the tank 5 and the manifolds 15, 21 and 23 are then conveyed through the pipe 30, to which is connected a pipe 30^a, having a plurality of branches 30^b, one of which leads to each of the manifolds 17, 19 and 25. The gases which have thus been collected in the tank 5 and the various manifolds, are conveyed by means of the pipe 30 to a glass condenser 31 having provision for a water jacket 32 supplied with a current of cold water by inlet and outlet pipes 33 and 34. The purpose of the condenser 31 is to condense as much of the hydrocarbon vapor as possible and to return it to the main body of hydrocarbon liquid. From the condenser 31 the gases are conveyed by a pipe 35 to the bottom of an absorption tower 36, designed for absorbing the hydrochloric acid gas in water. The tower 36 has any suitable interior construction for effecting this purpose. The hydrochloric acid gas is conveyed upwardly through said tower and is subjected therein to a current of water which is conveyed downwardly through the same from an inlet pipe 37 having a funnel 38 to receive a stream of water from a water supply pipe 39. This pipe 37 also has a water jacket 40 to condense as much as possible of the condensable vapors which fail to be absorbed in the tower 36. The vapors which are not absorbed in this manner are conducted from the pipe 37 in the reverse direction into a vapor collector 41 having a water jacket 42 provided with a water inlet pipe 43 and an outlet pipe 44 for the waste water. The outlet pipe 44 from the water jacket 42 is connected to an inlet pipe 45 leading to the water jacket 40 upon the pipe 37, and from the water jacket 40 a water discharge pipe 46 leads to the sewer. The vapor collector 41 is provided with a body of oil to absorb any of the hydrocarbon vapors which fail to be condensed in the other portions of the apparatus. At its end, the vapor collector 41 has a T-joint 47 from the top of which a vent pipe 48 leads to the outer atmosphere, and from the bottom of the T-joint 47 a discharge pipe 49 leads downwardly to the outer part of the apparatus where it is provided with a hand

valve 50 to permit the contents of the vapor collector to be drawn off at any time when desired.

The water which flows downwardly through the tower 36 and which is to absorb the hydrochloric acid gas and any chlorin gas therein so as to thereby increase the concentration of the hydrocarbon vapors and condense the same under the cooling action of the water is conducted out of the said tower together with the condensed hydrocarbons by means of a pipe 51, and through a spray nozzle 52 into the tank 5. The liquid containing hydrochloric acid gas and chlorin gas is thus brought into intimate contact with the body of chlorinated hydrocarbons within the tank 5, and thereby any chlorin present in the water is abstracted by, and becomes absorbed in the hydrocarbons.

Any chlorin gas which fails to be absorbed by the hydrocarbons in the absorption tower 1 is conducted from the top thereof by means of a pipe 53 which is inclined upwardly from the top of the absorption tower to a pipe 54 which conveys the gas through a glass lantern 55, a pipe 56 and a pipe 57 to the tank 5, where the chlorin is, at least in part, absorbed by the hydrocarbons in said tank. The upwardly directed pipe 53 is provided with a water jacket 58 to condense any hydrocarbon vapors which may have escaped from the top of the absorption tower. This water jacket 58 has a water inlet pipe 59, a vent 60, leading to the open air, and a water overflow pipe 61 leading downwardly therefrom to the sewer.

In order to maintain the tank 5 as cool as possible, there is a water spraying pipe 62 located at each side thereof for spraying water on the sides of the tank. This water spraying pipe is supplied with a stream of water by means of a pipe 63 having a hand-operated valve 64. The water thus sprayed upon the sides of the tank is collected in a trough 65 surrounding the bottom of the same, from which an overflow pipe 66 leads to the sewer.

The tank 5 thus acts as an accumulator of all the liquid products of the apparatus, the hydrocarbons and the chlorinated hydrocarbons collecting in the top of the tank, and the hydrochloric acid and water collecting in the bottom thereof. The hydrochloric acid is conveyed from the bottom of said tank by an overflow tube 67 leading to a pipe 68, the top of which is open to the atmosphere and the bottom of which leads, by means of a pipe 69, to a stoneware reservoir 70 for receiving hydrochloric acid. The bottom of the reservoir 70 has a draw-off cock 71 and said reservoir is also supplied with an overflow pipe 72 leading upwardly from its bottom and discharging into a pipe 73, leading to the sewer.

The various water inlet pipes are provided with a supply of water from a common water inlet pipe 74 having a branch 75 leading to the pipe 59, a branch 76 leading to the pipe 63, a branch 77 leading to the pipe 33, a pipe 78 leading to the pipe 43, and a pipe 79 leading to the pipe 39. Over the glass tubes 16, 20 and 24, we have located a series of mercury vapor lights 80 which are adapted to throw a blue light upon said tubes. The mercury lights 80 are supported in such a manner that they may be readily moved away when it is desired to have sunlight thrown upon the tubes. The entire apparatus is inclosed in a building 81 having glass panes 82 at the top and sides thereof adjacent to the portion of the apparatus including the glass tubes referred to. In order to control the amount of light coming through the glass panes, we provide a curtain 83 beneath the panes in the roof, and a curtain 84 over the panes in the wall of the building. By a manipulation of the curtains 83 and 84 and the lights 80, the proper degree of light may be thrown upon the tubes in which the reaction is to be effected.

The operation of the process may be summarized as follows:

When a quantity of hydrocarbons has been placed in the tank 5, the pump 4 is started and a constant stream of the hydrocarbons is caused to pass downwardly through the absorption tower 1. The speed of this current may be regulated by the speed of operation of the pump 4. The current of chlorin is then admitted from the chlorin bottles 11 by properly manipulating the hand-valves thereon until a proper amount of chlorin is admitted to the absorption tower, the proper amount of chlorin being determined by the complete disappearance of the yellow color from the hydrocarbons as the same leave the last series of glass tubes 24. Inasmuch as the absorption of the chlorin is caused to take place in the hydrocarbons in a dark chamber, that is to say the tower 1, the absorption is effected before the reaction of chlorination begins. There is, consequently, no danger of an explosion during the absorption. This is very important, inasmuch as an explosion in the presence of vapors would be very destructive. It is for this reason that the applicant has devised a process and apparatus of such a character that the reaction will not take place in a chamber where there is free space for the collection of gases or vapors. The absorption having been effected, the hydrocarbons containing the absorbed chlorin pass downwardly through the glass tubes where the light, which may be either daylight or the blue light emanating from the mercury vapor lamps 80, causes the chlorination of the hydrocarbons to be effected. The

chlorinated hydrocarbons are then discharged into the tank 5. Any hydrochloric acid gas which escapes from the liquid during the reaction and after the liquid has been conducted into the tank 5, is conveyed by means of the pipe 30 through the condenser for the purpose of returning the vaporized hydrocarbons, and then into an absorption tower provided with a percolating stream of water to absorb the hydrochloric acid gas and to condense a further quantity of the hydrocarbons. The water containing the absorbed hydrochloric acid gas is then conducted to the tank 5 where it is sprayed through the body of hydrocarbons for the purpose of causing the hydrocarbons to take up from the same any absorbed chlorin gas and also to remove the hydrochloric acid still remaining in solution in the hydrocarbons and chlorhydrocarbons in the tank 5. It will be understood that the water containing hydrochloric acid may also contain chlorin gas owing to the escape thereof from the tank 5 or from the various manifolds of the glass tubes in which the reaction is effected. Some of the chlorin thus conducted into the absorption tower 36 may also have emanated from the top of the absorption tower 1 from which the unabsorbed chlorin gas is led by means of a series of pipes into the top of the tank 5. The liquid which is thus conveyed from the bottom of the tower 36 into the tank 5 may also contain any liquids condensed beyond the tower 36 in the condenser 40. Any gases which are not condensed in the condenser are led into the vapor collector where the uncondensed hydrocarbon vapors are absorbed in a body of kerosene or similar hydrocarbons which may be drawn off from time to time by the pipe 49. Any remaining gases or vapors are vented to the outer air by means of the vent 48. From the tank 5 the accumulated water containing hydrochloric acid is conveyed continuously to the storage receptacle 70 from which it flows out continuously from the bottom through the pipe 72. Any hydrocarbons or chlorhydrocarbons carried off accidentally into the receptacle 70 will remain in the upper part of the same and may be drawn off as desired through the cock 71. The height of the liquid in the tank 5 may be regulated as desired by raising or lowering the movable parts 67, 68 and 69. The hydrocarbons and chlorhydrocarbons which have collected in the tank 5 are circulated through the tower and the glass tubes in which the reaction takes place, until the proper degree of chlorination has been effected. In the first stages of the operation of the apparatus, a mixture of monochlor and dichlor substitution products is obtained, together with some unchanged hydrocarbons. As the treatment continues, some of the higher sub-

stitution products are formed in addition. If it is desirable to obtain only mono- and dichlor substitution products, the apparatus is operated until about one-half of the hydrocarbons have been chlorinated. The process is then stopped and the chlorinated hydrocarbons removed from the tank 5 by means of the draw-off cock 27.

While we have described our invention above in detail, we wish it to be understood that many changes may be made therein without departing from the spirit of our invention.

We claim:

1. The process which comprises chlorinating a body of liquid in such a manner as to avoid the maintenance of a body of gas over the same.
2. The process which comprises absorbing chlorin in a body of liquid in the absence of light, and then chlorinating the liquid in such a manner as to avoid the maintenance of a body of gas over the same.
3. The process which comprises chlorinating a body of hydrocarbon liquid in such a manner as to avoid the maintenance of a body of gas over the same.
4. The process which comprises absorbing chlorin in a body of hydrocarbon liquid in the absence of light, and then chlorinating the hydrocarbon liquid in such a manner as to avoid the maintenance of a body of gas over the same.
5. The process which comprises chlorinating a body of liquid in such a manner as to avoid the maintenance of a body of gas over the same in the presence of light.
6. The process which comprises absorbing chlorin in a body of liquid in the absence of light, and then chlorinating the liquid in such a manner as to avoid the maintenance of a body of gas over the same in the presence of light.
7. The process which comprises chlorinating a body of hydrocarbon liquid in such a manner as to avoid the maintenance of a body of gas over the same in the presence of light.
8. The process which comprises absorbing chlorin in a body of hydrocarbon liquid in the absence of light, and then chlorinating the hydrocarbon liquid in such a manner as to avoid the maintenance of a body of gas over the same in the presence of light.
9. The process which comprises chlorinating a body of liquid in such a manner as to avoid the maintenance of a body of gas over the same in the presence of a blue light.
10. The process which comprises chlorinating a body of hydrocarbon liquid in such a manner as to avoid the maintenance of a body of gas over the same in the presence of a blue light.
11. The process which comprises chlorinating a body of liquid in such a manner as

to avoid the maintenance of a body of gas over the same in the absence of chlorin carriers.

12. The process which comprises chlorinating a body of hydrocarbon liquid in such a manner as to avoid the maintenance of a body of gas over the same in the absence of chlorin carriers.

13. The process which comprises chlorinating a hydrocarbon liquid thereby causing the escape of a gas, absorbing escaping gas in water, and mixing the water with the chlorinated hydrocarbon.

14. The process which comprises chlorinating a hydrocarbon liquid thereby causing the escape of a gas, absorbing escaping gas in water, and mixing the water with the chlorinated hydrocarbon, while keeping the latter cool.

15. The process which comprises absorbing chlorin in a hydrocarbon, chlorinating the hydrocarbon liquid thereby causing the escape of chlorin, bringing unabsorbed chlorin gas into contact with water to absorb it, and mixing the water with the chlorinated hydrocarbon.

16. The process which comprises absorbing chlorin in a hydrocarbon, chlorinating the hydrocarbon liquid, thereby causing the escape of chlorin, bringing unabsorbed chlorin gas into contact with water to absorb it, and mixing the water with the chlorinated hydrocarbon while keeping the latter cool.

17. The process which comprises absorbing chlorin in a hydrocarbon, chlorinating the hydrocarbon liquid thereby causing the escape of chlorin gas and hydrochloric acid gas, absorbing escaping hydrochloric acid gas and unabsorbed chlorin gas in water, and mixing the water with the chlorinated hydrocarbon.

18. The process which comprises absorbing chlorin in a hydrocarbon, chlorinating the hydrocarbon liquid thereby causing the escape of chlorin gas and hydrochloric acid gas, absorbing escaping hydrochloric acid gas and unabsorbed chlorin gas in water, and mixing the water with the chlorinated hydrocarbon while keeping the latter cool.

19. The process which comprises chlorinating a hydrocarbon liquid thereby causing the escape of a gas, absorbing escaping gas in water, mixing the water with the chlorinated hydrocarbon, and preventing the escape of the unabsorbed gas.

20. The process which comprises chlorinating a hydrocarbon liquid thereby causing the escape of a gas, absorbing escaping gas in water, mixing the water with the chlorinated hydrocarbon, while keeping the latter cool, and preventing the escape of the unabsorbed gas.

21. The process which comprises chlorin-

ating a hydrocarbon liquid thereby causing the escape of a gas, absorbing escaping gas in water, condensing and preventing the escape of hydrocarbon vapors, and mixing the water with the chlorinated hydrocarbon.

22. The process which comprises chlorinating a hydrocarbon liquid, thereby causing the escape of a gas, absorbing escaping gas in water and condensing and preventing the escape of hydrocarbon vapors, and mixing the water with the chlorinated hydrocarbon, while keeping the latter cool.

23. The process which comprises absorbing chlorin in a hydrocarbon, chlorinating the hydrocarbon liquid, bringing unabsorbed chlorin gas into contact with water to absorb it, and thereby condensing and preventing the escape of hydrocarbon vapors, and mixing the water with the chlorinated hydrocarbon.

24. The process which comprises absorbing chlorin in a hydrocarbon, chlorinating the hydrocarbon liquid, bringing unabsorbed chlorin gas into contact with water to absorb it, and thereby condensing and preventing the escape of hydrocarbon vapors, and mixing the water with the chlorinated hydrocarbon while keeping the latter cool.

25. The process which comprises chlorinating a hydrocarbon liquid, thereby causing the escape of a gas, absorbing escaping gas in water, and thereby condensing and preventing the escape of hydrocarbon vapors, mixing the water with the chlorinated hydrocarbon, and recovering by subsequent condensation unabsorbed and uncondensed gas.

26. The process which comprises chlorinating a hydrocarbon liquid, thereby causing the escape of a gas, absorbing escaping gas in water, and thereby condensing and preventing the escape of hydrocarbon vapors, mixing the water with the chlorinated hydrocarbon, while keeping the latter cool, and recovering by subsequent condensation unabsorbed and uncondensed gas.

27. The process which comprises chlorinating a body of liquid in such a manner as to avoid the maintenance of a body of gas over the same in the presence of light containing blue rays.

28. The process which comprises chlorinating a body of hydrocarbon liquid in such a manner as to avoid the maintenance of a body of gas over the same in the presence of light containing blue rays.

29. The process which comprises halogenating a body of hydrocarbon liquid in such a manner as to avoid the maintenance of a body of gas over the same.

30. The process which comprises absorbing a halogen in a body of hydrocarbon liquid in the absence of light and then

halogenating the liquid in such a manner as to avoid the maintenance of a body of gas over the same.

31. The process which comprises halogenating a body of hydrocarbon in such a manner as to avoid the maintenance of a body of gas adjacent to the same.

32. The process which comprises halogenating a body of hydrocarbon in the presence of light, in such a manner as to avoid the maintenance of a body of gas adjacent to the same.

33. The process which comprises absorbing a halogen in a liquid hydrocarbon under such conditions as to avoid a reaction between the halogen and hydrocarbon, and conducting the liquid mixture into a reaction chamber having conditions established therein such as to bring about a reaction within the liquid between the hydrocarbon and the halogen.

34. The process which comprises absorbing a halogen in a liquid hydrocarbon in the absence of light to avoid a reaction between the halogen and hydrocarbon and conducting the liquid mixture into a position to be reacted upon by light under conditions to bring about a reaction within the liquid between the hydrocarbon and the halogen.

35. The process which comprises bringing together in a mixing chamber a halogen and a liquid hydrocarbon so as to mix the same under conditions such as to avoid reaction, and then transferring the mixed substances to a reaction chamber having conditions established therein such as to bring about a reaction, then returning the product to the mixing chamber, adding more of the halo-

gen and again transferring the mixture to the reaction chamber.

36. The process which comprises bringing together in an opaque mixing chamber a halogen and a liquid hydrocarbon so as to mix the same under conditions such as to avoid reaction, and then transferring the mixed substances to a transparent reaction chamber having conditions established therein such as to bring about a reaction, then returning the product to the mixing chamber, adding more of the halogen and again transferring the mixture to the reaction chamber.

37. The process of chlorinating hydrocarbons which comprises absorbing a halogen in a liquid comprising a hydrocarbon under such conditions as to substantially avoid a reaction between the halogen and the hydrocarbon, separating the resulting liquid from unabsorbed halogen, and then causing the hydrocarbon to react with the halogen carried thereby.

38. The process of chlorinating hydrocarbons which comprises saturating with chlorine a liquid comprising a hydrocarbon under such conditions as to substantially avoid a reaction between the chlorine and the hydrocarbon, separating the saturated liquid from unabsorbed chlorine, and then causing the hydrocarbon to react with the chlorine carried thereby.

In testimony that we claim the foregoing we have hereunto set our hands.

FIN SPARRE.

WALTER E. MASLAND.

Witnesses:

DONALD R. MORTON,
CHAS. E. WEBB, Jr.