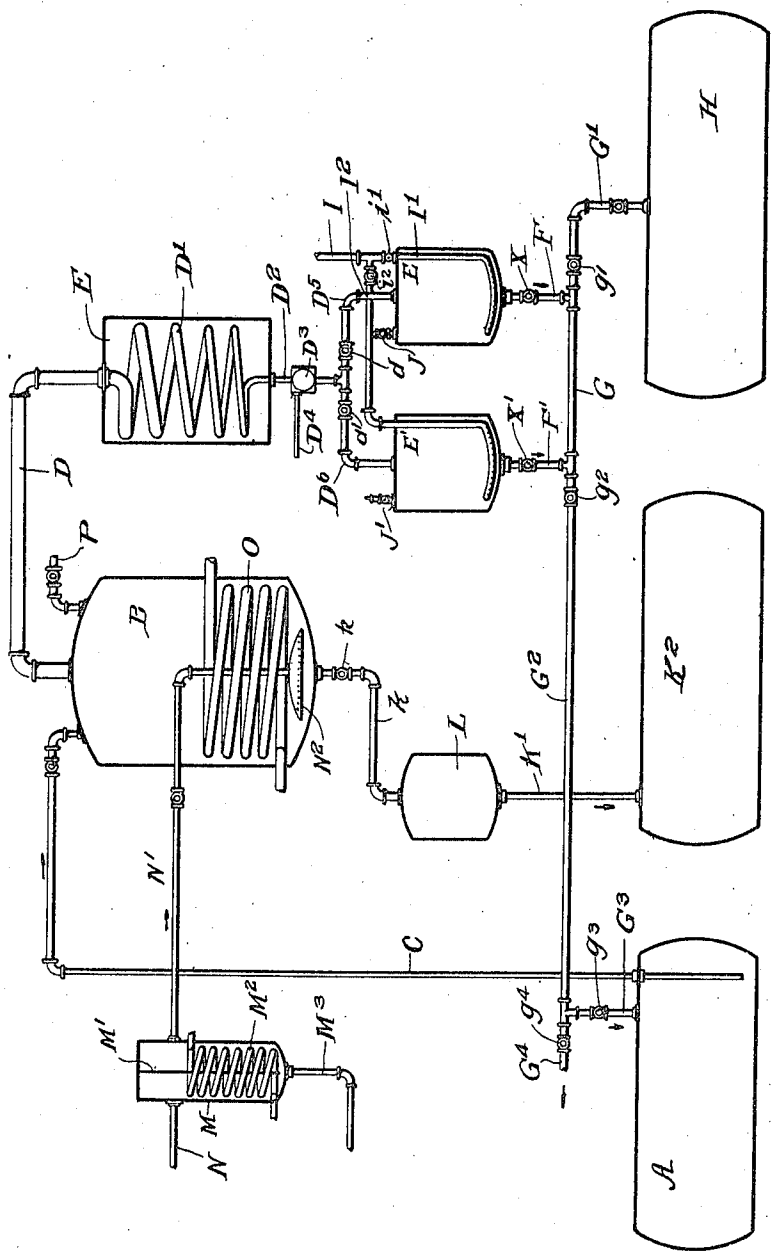
F. E. DODGE & F. H. RHODES.

APPARATUS FOR THE RECOVERY OF PYRIDINE BASES.

APPLICATION FILED JAN. 17, 1918.

1,275,000. Patented Aug. 6, 1918.

Fig.



Witness

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FRANK E. DODGE, OF NEW YORK, N. Y., AND FREDRICK H. RHODES, OF PHILADELPHIA, PENNSYLVANIA, ASSIGNORS TO THE BARRETT COMPANY, A CORPORATION OF NEW JERSEY.

APPARATUS FOR THE RECOVERY OF PYRIDINE BASES.

1,275,000. Specification of Letters Patent. Patented Aug. 6, 1918.

Application filed January 17, 1918. Serial No. 212,377.

To all whom it may concern:

Be it known that we, FRANK E. DODGE, of the city, county, and State of New York, and FREDRICK H. RHODES, of the city and county of Philadelphia, State of Pennsylvania, both citizens of the United States, have invented certain new and useful Improvements in Apparatus for the Recovery of Pyridine Bases, of which the following is a true and exact description, reference being had to the accompanying drawings, which form a part thereof.

Our invention relates to apparatus especially designed and intended for use in the recovery of pyridine bases from the bath liquor of the direct or semi-direct ammonium sulfate processes, and in accordance with the new method for the recovery of such pyridine bases which forms the subject matter of our co-pending application for Letters Patent filed January 17, 1918, Serial Number 212,375.

In the direct or semi-direct ammonium sulfate processes the gases from the tar extractors are freed from ammonia by passing them through a saturated solution of ammonium sulfate, which solution is kept acid with from ten to six per cent. of free sulfuric acid. The ammonia in the gas is absorbed by this acid solution, and crystallizes out of the bath liquor as ammonium sulfate. The pyridine vapor forming a portion of the gas treated is also absorbed by the solution in the saturater bath, but no compound of pyridine separates from the bath liquor until the concentration of pyridine in the bath liquor reaches a certain value.

In our new process for recovering the pyridine bases as set forth in our above-mentioned co-pending application, we remove the bath liquor from a saturater bath which has been in use, preferably before the saturation of the bath liquor with pyridine has reached the point where the ammonium sulfate is contaminated by pyridine sulfates. We then pass ammonia or ammonia vapor into the bath, or mix with it a strong aqueous solution of ammonia, with the result that the ammonia first reacts with the free acid present to form ammonium sulfate, and after most of the free acid is thus neutralized the ammonia begins to react with the pyridine sulfate in the solution, forming ammonium sulfate and setting free the pyridine bases.

While the pyridine bases can be recovered by agitating the bath liquor with an oil, as described in our co-pending application for Letters Patent filed January 17, 1918, Serial Number 212,376, our preferred method of recovering such bases is, as described in our application Serial Number 212,375, to distil off the pyridine bases from the neutralized bath liquor, condensing the distillate in suitable receptacles and effecting a gravital separation of the pyridine bases from the aqueous condensate by passing ammonia through such receptacles, and our present invention is of apparatus especially intended for use in this last-mentioned method of recovering pyridine bases.

The nature of our invention will be best understood as described in connection with the drawing in which it is illustrated, and which drawing is a diagrammatic view of our improved apparatus, in which—

A is a tank adapted to receive and contain the old bath liquor from the direct or semi-direct ammonium sulfate processes. B is a lead-lined neutralizing still into which portions of the old bath liquor are forced from time to time through the pipe C. The still as shown is provided with a lead steam coil O for the heating of the bath, when necessary, with an acid inlet indicated at P, and with a drainage pipe K, having a valve indicated at k, and leading to a filter L, from which a pipe K′ leads to a storage tank K^2. N is a pipe leading from an ammonia still or other source of ammonia supply into a heater and separator M, which is formed with a partition M′ and provided with a steam coil M^2, a drainage pipe M^3 being provided leading to the ammonia still or saturater not shown. From the heater and separator M a pipe N′ leads into the neutralizing still, terminating in a perforated head N^2. D is a conduit leading from the still and connecting with a condenser worm D′ in a condenser tank E, the worm in turn connecting through a pipe D^2, which may advantageously be provided with a sight glass as indicated at D^3 and a vent pipe D^4, to conduits D^5 and D^6, each provided with valves d and d', and connecting respectively with the receivers E and E′, from which drainage

pipes F and F′, provided with valves X and X′, lead to a conduit G, which, through a drainage pipe G′, connects to a pyridine storage tank H, and, through a drainage pipe G^2 with the connection G^3, leading to the storage tank A and the connection G^4, which may lead to a hot drain tank not shown. g', g^2, g^3 and g^4 are valves, by opening and closing which the contents of the receivers E and E′ can be directed to a desired receptacle. I is a pipe leading from a still or other source of ammonia, and connecting through pipes I′ and I^2 with the tanks E and E′, i' and i^2 being valves by which the course of the ammonia can be directed to either receptacle at will. J and J′ are vents for the ammonia, which of course will be connected to a proper receptacle not shown.

In operation, the old bath liquid is forced into the still B, and ammonia, preferably dried in the heater and separator M, is forced through the perforated head N^2 into the bottom of the still. The ammonia will first react with the free acid in the bath, and after it has been neutralized or nearly so, will begin to act upon the pyridine sulfates, forming ammonium sulfate and liberating the pyridine bases, which, by the heat of the reaction, aided if necessary by the steam coil O, are distilled off, together with aqueous vapor, through the pipe D, condensed in the worm D′ and delivered at will to the tanks E and E′. When either of said tanks is filled to the desired degree, ammonia is introduced through the pipes I′ or I^2, with the result that a good gravital separation is effected between the pyridine bases and the aqueous solution of ammonia formed from the water of condensation. When the separation has been effected, the aqueous solution is drawn off to the tank A or the hot drain tank, and the layer of pyridine bases is drawn off to the storage tank H.

The liquor in the neutralizing still B, when freed of the pyridine bases, is drawn off through the pipe K, preferably through a filter L, and delivered to the storage tank K^2.

Having now described our invention, what we claim as new and desire to secure by Letters Patent, is:

1. Apparatus for the recovery of pyridine bases from the saturated baths of the direct or semi-direct ammonium sulfate processes, consisting of a still to contain the bath liquor in combination with means for introducing ammonia into said still, a condenser connected to receive the distillate from the still, a receiver for condensates, means for introducing ammonia into said receiver, a receptacle for ammonia liquor and a receptacle for the pyridine bases, and a conduit system whereby the receiver can be connected with either of said receptacles at will.

2. Apparatus for the recovery of pyridine bases from the saturated baths of the direct or semi-direct ammonium sulfate processes, consisting of a tank for the storage of the saturated bath liquor to be treated, in combination with a still to contain portions of such bath liquor, means for introducing ammonia into said still, a condenser connected to receive the distillates from the still, a receiver for the condensed liquid from the still, means for introducing ammonia into said receiver, a storage tank for pyridine bases and a conduit system leading from the receiver to the bath storage tank and to the pyridine base tank.

3. Apparatus for the recovery of pyridine bases from the saturated baths of the direct or semi-direct ammonium sulfate processes, consisting of a tank for a storage of the saturated bath liquor to be treated, in combination with a still to contain portions of such bath liquor, means for introducing ammonia into said still, a condenser connected to receive the distillates from the still, a receiver for the condensed liquid from the still, means for introducing ammonia into said receiver, a storage tank for pyridine bases, a conduit system leading from the receiver to the bath storage tank and to the pyridine base tank, a tank for the neutralized bath from the still and a conduit from said still to said bath.

4. Apparatus for the recovery of pyridine bases from the saturated baths of the direct or semi-direct ammonium sulfate processes, consisting of a tank for the storage of the saturated bath liquor to be treated, in combination with a still to contain portions of such bath liquor, means for introducing ammonia into said still, a condenser connected to receive the distillates from the still, a receiver for the condensed liquid from the still, means for introducing ammonia into said receiver, a storage tank for pyridine bases, a conduit system leading from the receiver to the bath storage tank and to the pyridine base tank, a tank for the neutralized bath from the still, a filter and conduits leading from the still to the filter and from the filter to the tank.

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