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1,619,663

APPARATUS FOR HEATING AND FOR CONTROLLING REACTIONS AT HIGH TEMPERATURES

Original Filed March 4, 1920

Fig. 1.

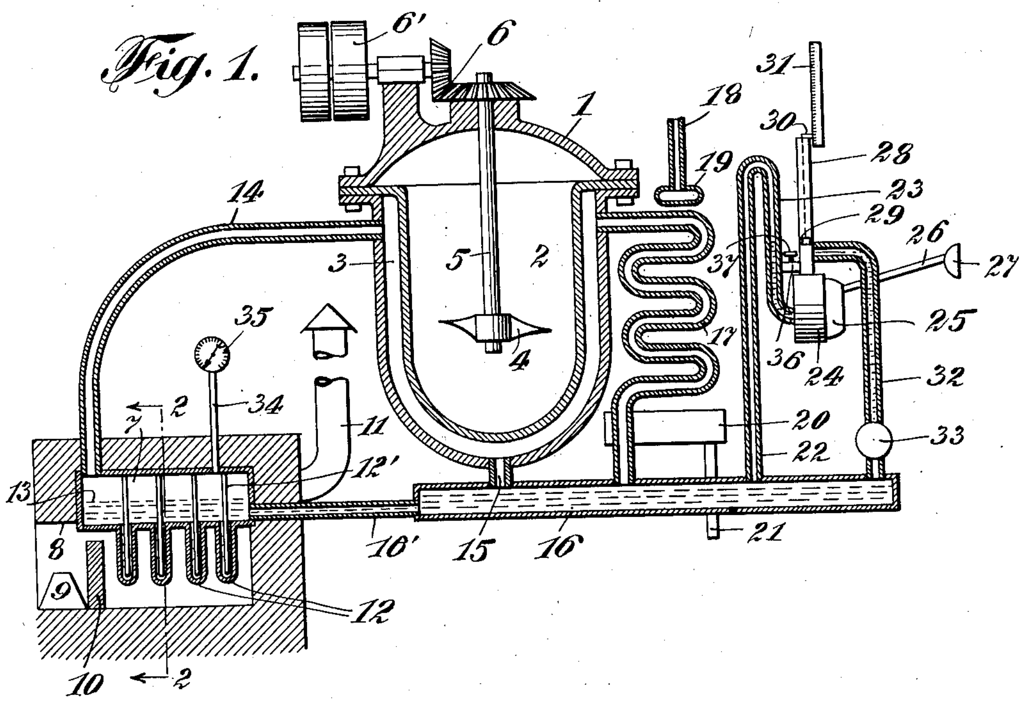


Fig. 2.

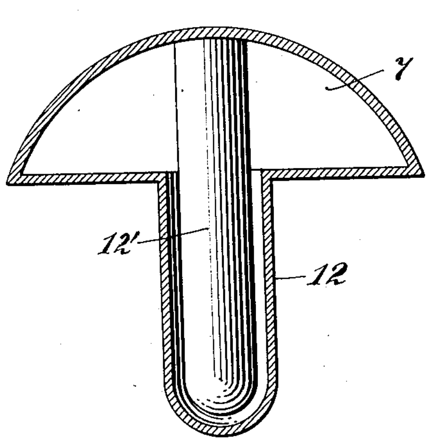
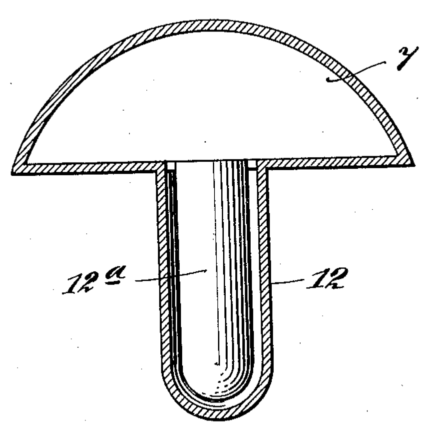


Fig. 3.



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UNITED STATES PATENT OFFICE.

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APPARATUS FOR HEATING AND FOR CONTROLLING REACTIONS AT HIGH TEMPERATURES.

Original application filed March 4, 1920, Serial No. 363,129. Divided and this application filed July 15, 1922. Serial No. 575,365.

This invention relates to an improved method and apparatus for controlling chemical reactions at high temperatures. The method is claimed in my application Serial No. 363,129, filed March 4, 1920, of which this case is a division; and this case includes apparatus claims only. Generically considered this invention contemplates the provision of means whereby certain organic or hydrocarbon materials which require a temperature greater than the boiling point of water, may be sulphonated, nitrated, distilled, fused, or subjected to various other chemical reactions.

In all chemical reactions the control of the temperature at which the reaction occurs is necessary in order to insure a maximum yield and the best quality of the product. Under comparatively low temperatures, which may be readily obtained by means of hot water or steam, such control is well-known in the art When, however, high temperature control is attempted, the results so far obtained have not been satisfactory. Some difficulties, which it is the purpose of my invention to obviate, are mentioned in my earlier co-pending application, now Patent No. 1,403,471.

By the term "chemical reaction" in the present disclosure is meant the combination of chemicals well-known in the art, and in addition thereto the disassociation of chemicals by means of heat and the concentration of chemicals in their various solutions, and further, the change of chemicals from one state or atomic structure to another, as by distillation, sublimation, evaporation, concentration, precipitaton, etc., and those other operations in industrial chemical processes by which chemical substances are transformed and obtained in the condition requisite to their subsequent use in the arts.

Although this delicate control of temperature is necessary in all chemical reactions, it is especially desirable and necessary in reactions dealing with organic materials, and in particular dye stuffs, and intermediates which have been derived from coal tar and in connection with which my present invention has been used with pronounced success.

The invention comprehends an improved apparatus whereby very delicate temperature control a low pressure may be obtained with a correspondingly large transfer of heat per surface unit. As a result of my improved method of temperature control, the required apparatus is relatively simple, may be readily installed, occupies a minimum of space and requires the attention of the operative only at infrequent intervals.

I have employed the improved method with marked success in sulphonation and distillation processes as heretofore used in the art in the production of beta naphthol, alpha naphthol, paranitraniline, anthracene, anthraquinone, acetanilid, naphthalene, phenanthrene, and believe that my invention is applicable to the entire family of organic materials, of which there are several thousand compounds known in the art.

In the use of mercury and its vapor as a heat transfer medium for these organic compounds, the mercury itself does not come into actual contact with the substance being treated, but transfers its heat through another medium, such, for example, as steel. This method may be successfully employed in the sulphonation of naphthalene in the production of beta disulphonic acid.

The advantages of the use of mercury or mercury vapor for these chemical reactions include the following. Mercury vapor at atmospheric pressure has a boiling point of 357° C. Hence, a comparatively high temperature can be obtained with little or no pressure, and such pressure as may exist is preferably from the atmosphere to the interior of the boiler or conduit containing the mercury vapor which is more or less desirable, since it will prevent the leakage of poisonous mercury fumes. Mercury is insoluble in either hot or cold water and also in hydrochloric acid, although it is soluble in nitric and concentrated sulphuric acids. Furthermore, it has a melting point of about 39° below zero C., and consequently will always remain liquid under normal operating conditions. Mercury readily forms amalgams with metals which are easily obtainable. Therefore, by inserting in parts of the apparatus where pressure might be created, metal with which the mercury will amalgamate, there will be no danger of the mercury fumes escaping to the atmosphere and dangerously affecting persons in the vicinity. Mercury will not form an amalgam with iron or steel, nor does it wet the surface of these metals. Consequently, when it is used in connection with my improved method which I shall now describe, a very large heat transfer per surface unit will be noted.

In these drawings wherein similar reference characters designate corresponding parts throughout the several views,

Fig. 1 is a diagrammatic sectional view showing one form of apparatus in which the transfer of heat from the vaporized mercury is effected entirely by conduction and convection through the wall of the vessel or container.

Fig. 2 is a transverse section through the mercury boiler taken on the line 2—2, Fig. 1.

Fig. 3 is a similar view showing a slightly modified form of the boiler.

Referring in detail to the drawings, 1 designates a closed kettle or vessel having a chamber 2 to receive the substance to be treated and provided with a jacket 3 for the wall of said chamber. A rotary agitator 4 is fixed upon the lower end of a shaft 5 extending into the chamber 2 and is driven through the medium of the gears 6 and the belt pulley 6′, or any other convenient source of power. The mercury is heated and vaporized in a boiler 7 arranged within a suitably constructed furnace 8. 9 indicates the source of heat which may be obtained from gas, oil, coal or other combustible fuel. The gases pass over a baffle wall 10 and between spaced tubes 12 depending from the boiler 7 and out through the stack 11 which is connected to the furnace chamber. In order to obtain a quick heating of the mercury vertically disposed rods 12′ are arranged in the boiler and extend downwardly into the tubes 12, so that only a relatively small quantity of the mercury will flow into said tubes and around the rods. These rods may extend above the level 13 of the mercury in the boiler 7 and be fixed to the top wall of the boiler as seen in Fig. 2, or the comparatively short rods 12^a may be used and wholly submerged within the mercury. It will be understood that the mercury is heated to the boiling point and converted into vapor, such vapor collecting in the boiler above the mercury level 13 and passing therefrom through the pipe 14 into the jacket 3 of the vessel or container. Here the heat of the mercury vapor is given up and transferred through the wall of the vessel 1 to the substance contained therein and thereby effects the desired chemical reaction. Of course, as the vapor gives up its heat, it is partially condensed and falls into the lower end of the jacket 3 and finds an outlet through the connection 15 with the catchall 16 which is connected by a pipe or conduit 16′ to the boiler 7. Such vapor as is not condensed in the jacket 3 passes into the condenser 17 which is connected to the upper end of the jacket. This condenser is in the form of a pipe coil and on the exterior surfaces of the coils water is delivered from a perforated pipe 19 connected to the end of the water supply pipe 18. The water from the pipe coils is collected in a pan or trough 20 from which it flows through the drain 21. The lower end of the condenser 17 is connected to the catchall 16, so that the condensed mercury is returned with the condensation from the jacket 3 to the boiler.

An adjustably determinable, uniform pressure, preferably below atmospheric, is maintained upon the entire system and to this end a pipe 22 is connected to the catchall 16 and at its upper end has a return bend or leg 23 which is connected to the pump 24. This pump is preferably a vacuum pump of the hydraulic centrifugal type using mercury instead of water. This pump is driven by a motor 25 receiving current from the controller 27 through a lead 26.

In practice, the leg 23 of the pipe 22 should be at least 30 inches long to afford a barometric column so that the mercury which fills it can never be pulled back through the pipe 22 when the pump is shut down even though there may be a very high vacuum in the boiler and circulating system. The speed of the pump, regulated by means of the controller 27, determines the degree of vacuum in the pipe 22, 23, and in the catchall 16 and consequently in the jacket 3 and the boiler 7. The faster the pump is operated the greater will be the quantity of liquid mercury which is drawn down the leg 23. This mercury is forced up into the pipe 28 connected to the pump. Thus functionally considered, the downflow intake pipe, 23, with the upflow outlet pipe, 28, constitutes a barometric U-tube, the two legs of which are connected through the pump 24. There is some mercury vapor which is not completely condensed in the condenser 17 and this vapor is drawn into pipe 22, 23 where it then condenses and raises the level of the mercury in the leg 23. To give more complete control, I provide a by-pass 36 between the pipe leg 23 and the pipe 28 which is provided with a valve 37. By adjusting this valve some of the mercury may be recirculated and in this manner the degree of vacuum on the system may be more precisely controlled.

The pump 24 eventually forces all of the liquid mercury in the pipe leg 23, including the condensed vapor, up into pipe 28. An overflow pipe 32 is connected to the pipe 28 and has its lower end connected to the catchall 16. A normally closed valve 33 in the pipe 32 prevents the flow of the mercury from said pipe into the catchall. Hence the liquid mercury may continue to collect in pipes 32 and 28 until they are entirely filled, the pipe 28 being open to the atmosphere at its upper end. In the pipe 28 a float 29 (diagrammatically illustrated) is arranged and a pointer 30 is connected to the upper end of said float. This pointer moves on a scale 31 and indicates the filling of the pipe 28. Through the medium of this indicator, either automatically or by hand, at the proper time, the valve 33 may be opened so as to permit the mercury to drain into the catchall 16 until the pointer 30 falls to zero on the scale 31. It will be understood that when the pump 24 is not working and the circulatory system is at exactly atmospheric pressure, the mercury in the downflow intake pipe, 23, will stand at the same height as the mercury in pipe 28. When the pump is running, the mercury will be higher in 28 than in 23 and if under such conditions the pump be stopped, the situation will immediately be reversed, the mercury rising in 23 to exceed the height in 28 by the same amount that the height in 28 previously exceeded that in 23.

The pipes 23 and 28 are so proportioned that the volume of liquid mercury in the pipe 28 up to the lower side of the pipe connection 32, plus the mercury in pump 24 which is free to return to the pipe leg 23 when the operation of the pump is stopped, will fill the pipe leg 23 up to the level of thirty inches and thereby prevent the ingress of air into the system. At any desired point a gauge 35 may be connected by means of a pipe 34 to indicate the internal pressure.

From the foregoing, the operation of the apparatus for carrying out my improved method will be readily understood. By the maintenance of a uniform pressure, preferably below atmosphere, throughout the system, it will be apparent that the heat of the mercury vapor in the jacket 3 will be absorbed by the wall of the vessel 1 at substantially the initial vaporization temperature of the mercury in the boiler 7. Owing to the absence of pressure, there will be no appreciable decrease in the temperature of the vapor during its passage through the pipe connection 14 and the substance contained in the vessel 1 will therefore be quickly heated to the requisite degree in order to cause the desired chemical reaction. The return of the condensed mercury vapor to the boiler from the catchall 16 maintains a substantially uniform level of the mercury at all times in the catchall and the boiler and, as rapidly as the liquid mercury is converted into vapor, the supply of mercury in the boiler is replenished from the catchall 16. It will thus be apparent that not only have I succeeded in eliminating mercury fumes, but the consumption of the mercury is exceedingly slow, thus rendering my improved method very economical. The necessary apparatus is also very simple in its construction as well as compact in arrangement, so that it may be very readily installed in comparatively small space.

It will be evident that some of the apparatus claims herein are broad enough to cover the apparatus disclosed in my method Patent No. 1,403,471, the application for which was co-pending with current application Serial No. 363,129 filed March 4, 1920, of which this later case is a division.

My present apparatus omits the superheater which is shown in said patent and also the valve in the pipe between the boiler and the container which valve is shown in my said patent and also in my companion divisional application of even date herewith. While these expedients might be employed in conduit 14 of my present case, there is special advantage in making said conduit as shown; i. e., a short, large pipe leading by smooth curves from the vapor space in the top of the boiler, to the container, as directly as possible, so as to afford very low resistance to the mercury vapor flow. Thus the vacuum imposed on the system by the pump is practically the same in the boiler as it is in the condenser, thereby insuring practically the same temperature at the condensing point as at the boiling point, notwithstanding the mercury seal at the point where the condensed mercury flows back into the boiler.

I claim:

1. A mercury boiler, a container including condensation surfaces in heat transferring relations to the material to be treated therein, an intermediate supply conduit affording a direct low resistance path for conducting vapor from the boiler to the condensation surfaces, drainage passages arranged in parallel for drainage of the primary condensate and for condensation and drainage of the surplus vapor back to the boiler, in combination with pressure controlling means operating on the system from a point beyond the container whereby the pressure and temperature of the vaporization are approximately the same as those of the condensation.

2. A mercury boiler, a container including condensation surfaces in heat transferring relation to the material to be treated therein, an intermediate supply conduit affording a direct low resistance path for conducting vapor from the boiler to the condensation surfaces, drainage passages arranged in parallel for drainage of the primary condensate and for condensation and drainage of the surplus vapor to the boiler, in combination with pressure controlling means operating on the system from a point beyond the container, said means for controlling the pressure including a

barometric U tube in which a standing column of liquid mercury is maintained of sufficient height, to seal the system against reverse flow of air.

3. A mercury boiler, a container including condensation surfaces in heat transferring relation to the material to be treated therein, an intermediate supply conduit affording a direct, low resistance path for flow of vapor from the vapor space in the boiler to the condensation surfaces, means for condensing surplus vapor, and a return conduit for the condensate, opening beneath the level of the mercury in the boiler, in combination with pressure controlling means operating on the circulating system, between the container and the return conduit.

4. A mercury boiler, a container including condensation surfaces in heat transferring relation to the material to be treated therein, an intermediate supply conduit affording a direct, low resistance path for flow of vapor from the vapor space in the boiler to the condensation surfaces, means for condensing surplus vapor, and a return conduit for the condensate, opening beneath the level of the mercury in the boiler, in combination with pressure controlling means operating on the circulating system, between the container and the return conduit, said pressure controlling means including a pump and a barometric U tube, one leg of which communicates with the pump inlet and the other leg with the pump outlet, said legs and the pump containing sufficient mercury to prevent reverse flow of air into the system when the pump is not running.

5. A mercury boiler, a container including condensation surfaces in heat transferring relation to the material to be treated therein, an intermediate supply conduit affording a direct, low resistance path for flow of vapor from the vapor space in the boiler to the condensation surfaces, means for condensing surplus vapor, and a return conduit for the condensate, opening beneath the level of the mercury in the boiler, in combination with pressure controlling means operating on the circulating system, between the container and the return conduit, said pressure controlling means including a vacuum pump.

6. A relatively shallow mercury boiler, a container for material to be treated, a conduit for conducting vapor from the boiler to condensation surfaces in heat transmitting relation to the material in the container, an outlet conduit for gravity flow of condensed mercury, a parallel outlet through a condenser and conduit for escape of the condensate, and a low level catchall for the condensate, associated with a common return for the liquid mercury opening beneath the level of the mercury in the boiler, so that the catchall operates as a reservoir to minimize variations of level of the mercury in the boiler.

7. A mercury boiler, a container for material to be treated, a conduit for conducting vapor from the boiler to condensation surfaces in heat transmitting relation to the material in the container, an outlet conduit for gravity flow of condensed mercury, a parallel outlet through a condenser and conduit for escape of the condensate, and a common return for the liquid mercury opening beneath the level of the mercury in the boiler, in combination with a conduit for withdrawing fluid from the circulation and a barometric U-tube in which liquid mercury is maintained to operate as a gas check but permitting escape of liquid mercury.

8. A mercury boiler, a container for material to be treated, a conduit for conducting vapor from the boiler to condensation surfaces in heat transmitting relation to the material in the container, an outlet conduit for gravity flow of condensed mercury, a parallel outlet through a condenser and conduit for escape of the condensate, in combination with means for withdrawing fluid from the system including a mercury vacuum pump and a barometric U-tube in which liquid mercury is maintained to permit escape of liquid mercury and permanent gases while sealing the system against reverse inflow of air.

Signed at New York city in the county of New York and State of New York, this 14th day of July, 1922.

CROSBY FIELD.