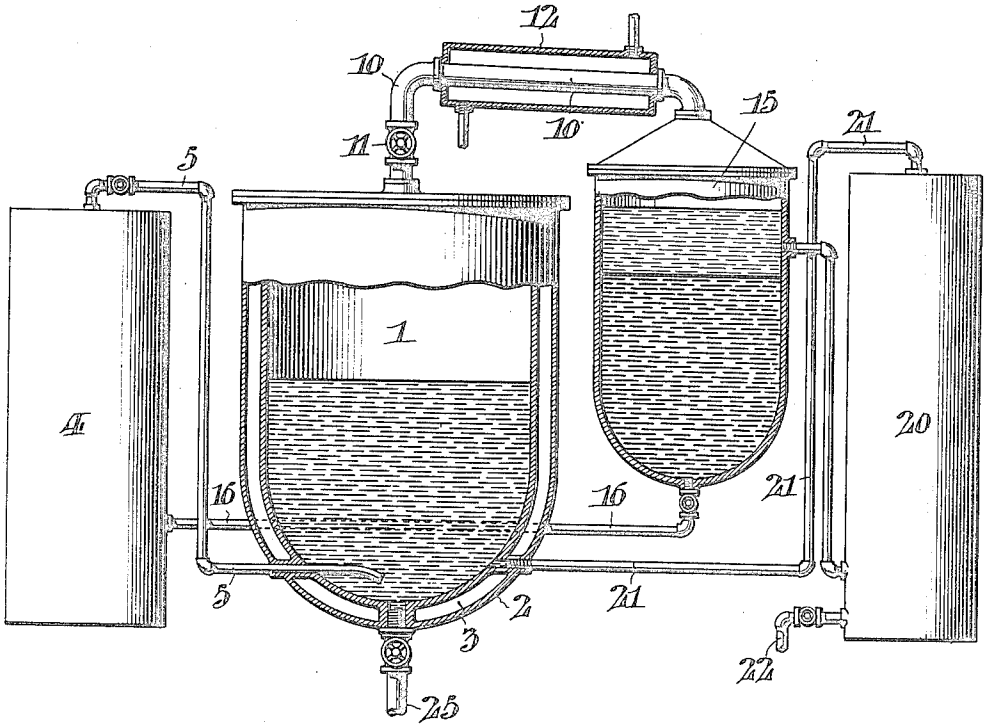
A. I. APPELBAUM.

PROCESS OF MANUFACTURING BENZALDEHYDE.

APPLICATION FILED MAY 17, 1918.

1,302,273. Patented Apr. 29, 1919.



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WITNESSES:

UNITED STATES PATENT OFFICE.

ALAN I. APPELBAUM, OF TRENTON, NEW JERSEY, ASSIGNOR TO THE IMPORT AND BY PRODUCTS COMPANY, INCORPORATED, OF TRENTON, NEW JERSEY, A CORPORATION OF DELAWARE.

PROCESS OF MANUFACTURING BENZALDEHYDE.

1,302,273. Specification of Letters Patent. Patented Apr. 29, 1919.

Application filed May 17, 1918. Serial No. 235,073.

To all whom it may concern:

Be it known that I, ALAN I. APPELBAUM, a citizen of the United States, residing in the city of Trenton, State of New Jersey, have invented certain new and useful Improvements in Processes of Manufacturing Benzaldehyde, of which the following is a specification.

In its broader aspects, my invention relates to the process of directly oxidizing hydro-carbons or compounds containing the CH_3 group to aldehydes, but in the description of the same, I shall refer specifically to the manufacture of benzaldehyde from toluol.

As above indicated, the object of my invention is to provide an improved process wherein and whereby a hydro-carbon compound containing the CH_3 group may be oxidized to an aldehyde. In the present application, I shall describe a process wherein and whereby benzaldehyde may be produced from toluol, an aromatic hydro-carbon by the use of manganese dioxid as an oxidizing agent in connection with a catalytic material. Any other suitable oxidizing agent or agents may be employed.

A further object of my invention is to cheapen and simplify the process of oxidizing or dehydrogenating the hydrocarbon compounds of the character mentioned, particularly toluol, for the production of benzaldehyde.

A further object of my invention is to provide a process which shall be continuous in its operation.

In the carrying out of my process, toluol, finely ground manganese dioxid, sulfuric acid mixed with three times its volume of water and a catalytic material or agent consisting either of ferric, copper or cerium sulfate are charged or inserted into a digester or reaction chamber which should be of acid-proof material or at least its inner surface should be acid-proof. This mixture should include approximately ninety-two parts of toluol, one hundred parts of manganese dioxid, one hundred and fifty parts of sulfuric acid, and a quantity of the catalytic agent equal approximately to five per cent. of the manganese dioxid employed in the mixture. The proportion of manganese dioxid as stated is employed when it has an oxidizing power of approximately eighty-five per cent., but as the percentage of oxidizing power varies the quantity of manganese dioxid to be used likewise varies. Upon being heated to boiling in the reaction chamber as hereinafter described, the ingredients become thoroughly mixed together.

After the ingredients have been inserted into the digester the temperature of the latter is raised to the boiling point which results, by an oxidation process, in the formation of benzaldehyde together with an insignificant quantity or proportion of benzoic acid.

Steam is caused to pass through the boiling mixture which effects separation therefrom and the passage from the reaction chamber of the benzaldehyde together with the unconverted toluol. The benzaldehyde, toluol, steam and any small proportion or trace of benzoic acid which may be present are passed through a condenser and are condensed. The condensate is collected in a suitable container and the benzaldehyde and toluol, being lighter than the water, are separated by gravity from the latter. The mixture of benzaldehyde and toluol is drawn off into a fractionator, while the water is returned to the boiler.

By reason of their different boiling points, the benzaldehyde and toluol may be separated from each other in the fractionator. This is due to the fact that toluol boils at 110° centigrade, while the benzaldehyde boils at 179° centigrade. It is found that by keeping the temperature of the fractionator at approximately 120° centigrade the toluol will boil off, leaving the benzaldehyde behind. The benzaldehyde may be purified by distillation in any known manner.

The foregoing is a description generally of the process without reference to apparatus of any kind or character whatsoever. It will be understood, however, that some form of apparatus is necessary to the employment of the process commercially, and I have indicated in the drawing accompanying this application, schematically and without any effort to show details of construction, one form of apparatus which may be employed. The said apparatus is shown partly in elevation and partly in section.

In the said drawing, I have shown a digester or reaction chamber 1, surrounded by an outer wall 2 forming an intermediate

chamber 3 for the reception of steam, by means of which the temperature of the interior of the digester or reaction chamber 1 may be kept at the boiling point. Into this digester or reaction chamber, toluol, manganese dioxid, sulfuric acid, mixed with three times its volume of water, and a catalytic material in the proportions above mentioned, are inserted or charged. After the insertion or charging of these substances into the digester or reaction chamber, the temperature of the latter is raised to the boiling point, whereupon steam is admitted from the boiler 4 through a pipe 5 to the bottom of the digester as indicated. The passage of steam through the mixture of materials in the digester causes the removal of the benzaldehyde which has been formed, together with unconverted toluol and such small negligible portions of benzoic acid as may have been formed. The mixture of toluol, benzaldehyde, steam and traces or small portions of benzoic acid pass outwardly through a pipe 10, controlled by the valve at 11. This vaporous or gaseous mixture is condensed by means of a condenser indicated at 12. The condensate formed is discharged into the separator 15. By reason of the fact that the water is heavier than the mixture of benzaldehyde and toluol, the latter two and the water separate from each other under the influence of gravity, the water going to the bottom of the separator 15 while the mixture of benzaldehyde and toluol floats upon the top of the water, as is indicated. From the separator 15, the water is conveyed through a valve-controlled pipe 16 to the boiler 4. The mixture of benzaldehyde and tuluol is conveyed from the separator to the lower end of a fractionator 20, wherein the toluol and benzaldehyde are separated by distillation. The mixture of benzaldehyde and toluol in the bottom of the fractionator is maintained at a temperature of approximately 120° centigrade, at which temperature distillation of the toluol takes place. The toluol travels upwardly through the fractionator and escapes therefrom through the pipe 21 by means of which it is conveyed back to the digester 1. The benzaldehyde which is left in the fractionator is either withdrawn through a valve-controlled pipe 22 and subsequently purified by distillation; or, by properly controlling the fractionator, the said benzaldehyde may be purified by further distillation in the said fractionator.

This process, as above described, is continuous as long as the charge which has been placed in the digester remains unexhausted; that is, as long as there remains free toluol to be oxidized and manganese dioxid which has not been reduced to sulfate of manganese.

By carrying on the process as above described, the benzaldehyde which is formed is withdrawn before sufficient oxidation has taken place to produce benzoic acid.

The spent or exhausted residue may be withdrawn from the digester through the valve controlled exhaust pipe 25.

I claim:

1. The process of producing benzaldehyde, which consists in mixing together toluol and an oxidizing agent and a catalytic material in the presence of acid, applying heat to raise the said mixture to a boiling temperature to cause reactions to take place to form the benzaldehyde and passing steam through the said mixture to separate the benzaldehyde therefrom.

2. The process of producing benzaldehyde, which consists in mixing together toluol and an oxidizing agent and a catalytic material in the presence of sulfuric acid, applying heat to raise the said mixture to boiling temperature to cause reactions to take place to form the benzaldehyde and thereafter separating the benzaldehyde therefrom.

3. The process of producing aldehydes from the aromatic hydro-carbon series, which consists in treating a hydro-carbon belonging to said series with an oxidizing agent and a catalytic in the presence of an acid and thereafter separating from the mass the aldehyde produced.

4. The process of producing benzaldehyde, which consists in mixing together toluol, finely ground manganese dioxid, a small percentage of a catalytic material, and sulfuric acid, heating the mixture to a temperature sufficient to cause reactions to take place to form benzaldehyde, separating the benzaldehyde from the mixture and purifying the same by distillation.

5. The process of producing benzaldehyde, which consists in mixing together toluol, finely ground manganese dioxid, a small percentage of a catalytic material, and sulfuric acid in a reaction chamber, heating the mixture to a temperature sufficiently high to cause reactions to take place to form benzaldehyde and passing steam through the said mixture to separate the benzaldehyde from the said mixture.

6. The process of producing benzaldehyde, which consists in mixing together toluol, manganese dioxid, sulfuric acid and a small percentage of a catalytic material and water, heating the said mixture to a temperature sufficiently high to cause reactions to take place to form the said benzaldehyde, separating the latter from the said mixture and then purifying the same by distillation.

7. The process of producing benzaldehyde, which consists in mixing together toluol, an oxidizing agent and a small percentage of a catalytic agent in the presence

of sulfuric acid, heating the mixture to a temperature sufficiently high to cause reactions to take place to form benzaldehyde, passing live steam through the said heated mixture to remove the benzaldehyde therefrom and thereafter purifying the benzaldehyde by distillation.

8. The process of producing benzaldehyde, which consists in mixing together toluol, finely ground manganese dioxid, sulfuric acid and a small quantity of a catalytic agent, heating the mixture to a boiling temperature, passing steam through the said mixture to separate the benzaldehyde and toluol from the said mixture, condensing the steam, benzaldehyde and toluol and collecting the same in a chamber, separating the benzaldehyde and toluol from the water of the condensate and thereafter separating the toluol from the benzaldehyde by distillation.

9. The process of producing benzaldehyde, which consists in mixing together toluol, finely ground manganese dioxid, sulfuric acid mixed with three times its volume of water, and a small percentage of a catalytic agent, heating the mixture to a boiling temperature, passing steam through the said mixture to separate the benzaldehyde and some toluol from the said mixture, condensing the steam, benzaldehyde and toluol and collecting the same in a chamber, separating the benzaldehyde and toluol from the water of the condensate by gravity and thereafter separating the toluol from the benzaldehyde by distillation.

10. The process of producing benzaldehyde, which consists in mixing together approximately ninety-two parts of toluol, approximately one hundred parts of finely ground eighty-five per cent manganese dioxid and approximately one hundred and fifty parts sulfuric acid mixed with approximately three times its volume of water and a small percentage of a catalytic agent, heating the said mixture to a boiling temperature to cause reactions to take place to form benzaldehyde, admitting steam to the bottom of said mixture and permitting it to pass through the same to separate the benzaldehyde and some toluol from the said mixture and thereafter separating the toluol and the benzaldehyde substantially as described.

In testimony that I claim the foregoing as my invention, I have hereunto signed my name this 16th day of May, A. D. 1918.

ALAN I. APPELBAUM.