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DIARYL CONTAINING WAX-LIKE SOLID

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This invention relates to a new diaryl-containing wax-like solid.

It has been known that when benzol is passed through a red-hot tube, diphenyl is formed. The composition of the product produced in this way varies with the temperature of the hot tube, the filling material used in the tube, the velocity of the vapors and the presence of such gases as hydrogen or nitrogen mixed with the benzol. We have discovered that further variation in the composition of the product is obtained when such compounds as toluene and xylene are present in the benzol treated in this manner, and that highly condensed complex hydrocarbons are formed.

It has recently been discovered that when benzol is passed through a bath of molten lead, heated to a temperature about 700° C., diphenyl and hydrogen are formed. However, it has not heretofore been known so far as we are aware, that the complex products mentioned above are produced together with diphenyl in the lead bath method of treating benzol, nor how the composition of the product could be varied at will. It is one of the objects of this invention to provide a new diaryl-containing wax-like solid of high boiling point.

A further object of this invention is to provide a means whereby the complex character of the product as determined by the boiling points of its various fractions can be varied at will. A still further object of this invention is to provide a product which is useful in producing a chlorinated diaryl which does not become crystalline at the higher chlorine content.

These objects are accomplished in the following manner: We have discovered that when benzol is passed into and through a bath of molten lead heated to between 700° and 800° C., considerable amounts of diphenyl together with a mixture of more complex hydrocarbons are formed. We have further found that the amount of the more complex hydrocarbon can be materially increased by increasing the temperature of the lead bath, and also by changing the composition of the benzol used as a starting material. Further, we have found that the material thus produced is of great value, since when chlorinated its tendency to crystallize is greatly minimized.

The particular manner in which our invention may be carried out and the new product produced may be illustrated by the following examples:

Example I

Commercially pure liquid benzol which distils within a range of 2° C. is pumped through a coil of pipe which is immersed in a bath of a molten substance inert with respect to the benzol, such as lead or aluminum, and which is maintained at the maximum temperature at which substantially no diphenyl is formed. This coil acts as a preheater and serves to vaporize the liquid benzol and preheat the benzol vapor to a point in the neighborhood of the diphenyl formation temperature.

The coil which is at the bottom of the bath is perforated, and the vapor streams out and in rising through the molten substance, in physical contact therewith, agitates the bath in such a manner as to maintain a practically uniform temperature throughout the bath. By employing a large mass in the bath, we are enabled to prevent over-heating of the vapor due to the heat capacity of the bath, and by means of a thermocouple immersed therein to observe temperature changes which may occur and change the heating so as to supply additional heat, or lessen the rate of heating, as desired. We prefer to operate the pre-heater bath at a temperature of from 600° to 650° C., and have found from experience that

this is the optimum temperature range for continuous operation.

After the preheated vapor emerges from the preheater bath, it is immediately passed into a converter bath which is of similar design and arrangement as the preheater. The vapor passes to the bottom of the bath and streams out through holes in a distributor, and then bubbles up through the bath in a manner similar to that described for the preheater. The converter bath is kept at a temperature range of from 750° to 850°, preferably around the latter temperature. When the vapor passes from the converter bath it is passed, at a high velocity, directly into a water-cooled condenser, so constructed that the vapor is quickly cooled and the unconverted benzene and the diphenyl are condensed and collected, while the hydrogen which is liberated when diphenyl is formed is vented through a water seal.

We have found that when operating in this manner with commercially pure benzole, the percentage of crude diphenyl (i. e., the percent of the fraction boiling between 200° and 270° C.) with 750° C. converter temperature is approximately 4.5% of the total condensate, while the fraction boiling above 270° C. is approximately 0.4% of the total.

If now the converter temperature be raised to 800° C., the crude diphenyl fraction (200°–270° C.) increases to approximately 8% of the total condensate, while the high-boiling fraction (above 270° C.) increases approximately to 1% of the total. By further increasing the temperature to 850° C., the crude diphenyl fraction increases to approximately 15.5% of the total condensate, while the high-boiling fraction increases to approximately 5% of the total.

Example II

Instead of the commercially pure benzol used in Example I, pass "commercial 90% benzol" through the converter units described in example I, and exactly in the same manner. Commercial 90% benzol contains certain percentages of other aromatic hydrocarbons, especially toluene and xylene. It is identified by its distillation range. For example, it starts distillation at 76.2° C.; 95% distils up to 100° C.; and all distils below 120° C. The new product formed in this case is somewhat different in composition from that formed in Example I.

In the following table, these products are compared. These results were obtained by fractionally distilling the new product obtained as heretofore outlined (boiling above 200° C.) into two fractions, one distilling between 200° C. and 270° C. and the second distilling above 270° C. The latter fraction (above 270° C.) is termed high boiling compound while the fraction boiling between 200° and 270° C. is termed mainly diphenyl.

Example III

Bath temperature	High-boiling compound (above 270° C.) in new product (above 200° C.)	
	Commercial benzol	Commercial 90 per cent benzol
750	7.6	12.7
800	11.5	20.0
850	25.0	30.5

It will be apparent that the composition of my product may be further varied by using mixtures of commercial benzol and commercial 90% benzol, in various proportions. Further variations may be accomplished by means of temperature changes, as indicated in the above table.

The product containing varying amounts of high boiling compounds may find a variety of applications in the art. It may be used as a wood preservative, impregnating material for electrical purposes, or as a heat transfer medium where pure diphenyl itself is not suitable or is too expensive. It may also be chlorinated and a non-crystalline solid obtained which is fire-retarding and has excellent properties making it valuable for electrical insulating purposes.

The advantages of this invention are that a new product containing varying amounts of high-boiling substances may be cheaply produced. The new product may have a varying composition which is obtained at will by varying the starting materials or the temperature of the reactions. The value of the new product lies in the fact that the physical properties of the diphenyl-containing product may be varied at will within the range above set out.

The new product distills above 200° C. and is comprised of two classes of compound, namely, crude diphenyl boiling between 200° and 270° and high boiling compound boiling above 270° and below the decomposition temperature of the compound which is in the neighborhood of 350° to 400° C.

What we claim is:

1. The process for producing a diphenyl containing wax-like solid, comprising vaporizing benzol, contacting said vapor with a fluid mass inert with respect to said vapors, and maintained at a temperature at which substantial quantities of diphenyl are formed, condensing the resulting condensible vapors and heating to 200° C. to remove low boiling constituents.

2. The process for producing a diphenyl containing wax-like solid, comprising vaporizing benzol, contacting said vapor with a fluid mass inert with respect to said vapor and maintained at a temperature of between

700° C. and 800° C., condensing the resulting vapors and heating said condensate to 200° C. to remove low boiling constituents.

3. The process for producing a diphenyl containing wax-like solid, comprising volatilizing benzol admixed with substantial quantities of toluol and xylol, contacting said vapor with a fluid mass maintained at diphenyl forming temperatures, condensing the resulting vapors and heating said condensate to 200° C. to remove low-boiling constituents.

4. The process for producing a diphenyl containing wax-like solid, comprising volatilizing "commercial 90% benzol", contacting the thus formed vapors with an inert liquid mass maintained at diphenyl forming temperatures, condensing the resulting vapors and heating said condensate to remove hydrocarbons boiling below 200° C.

5. The process for producing a diphenyl containing wax-like solid, comprising volatilizing commercial 90% benzol, contacting the thus formed vapors with molten lead maintained at diphenyl forming temperatures, condensing the resulting vapors and heating said condensate to remove hydrocarbons boiling below 200° C.

6. In a process of producing diphenyl by thermal synthesis from benzol by contacting benzol with a heated inert molten substance, the step of controlling the percentage of complex hydrocarbons boiling above 270° C. by controlling the temperature of said substance.

7. In a process of producing diphenyl by thermal synthesis from benzol by contacting benzol with a heated inert molten substance, the step of controlling the percentage of complex hydrocarbons boiling above 270° C. by maintaining the temperature of said substance at above 800° C.

8. In a process of producing diphenyl by thermal synthesis from benzol by contacting benzol with a heated inert molten substance comprising vaporizing commercial 90% benzol, and subjecting the vapors by contact with said molten substance to a temperature of from 800° to 850° C. to increase the production of complex hydrocarbons boiling above 270° C.

9. In a process of producing diphenyl by thermal synthesis from benzol by contacting benzol with a heated inert molten substance comprising, vaporizing a mixture of commercial benzol and commercial 90% benzol, and subjecting the vapors by contact with said molten substance to a temperature of from 800° to 850° C. to increase the production of complex hydrocarbons boiling above 270° C.

In testimony whereof we affix our signatures.

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