

UNITED STATES PATENT OFFICE.

OTTO GRAUL, OF LUDWIGSHAFEN-ON-THE-RHINE, GERMANY, ASSIGNOR TO BADISCHE ANILIN & SODA FABRIK, OF LUDWIGSHAFEN-ON-THE-RHINE, GERMANY, A CORPORATION.

PROCESS OF MAKING CHLORINATED HYDROCARBONS.

1,045,139.

Specification of Letters Patent.

Patented Nov. 26, 1912.

No Drawing.

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To all whom it may concern:

Be it known that I, OTTO GRAUL, subject of the Duke of Anhalt, residing at Ludwigshafen-on-the-Rhine, Germany, have invented new and useful Improvements in Processes of Making Chlorinated Hydrocarbons, of which the following is a specification.

When olefinic hydrocarbons, such as the olefins themselves C_nH_{2n} , are treated with a halogen, there is a difference in the result, as is well known, according to whether chlorine or bromine is used. The reaction with bromine results in the smooth production of the compounds containing two additional bromine atoms, whereas the addition of chlorine has hitherto always been accompanied by the formation of considerable quantities of substitution products.

My present invention consists in a process of producing the chlorinated hydrocarbons by breaking the double bond of the unsaturated hydrocarbons and adding chlorine, while avoiding any considerable formation of substitution products. The said process consists in treating the unsaturated hydrocarbons with sulfuryl chloride. The action of the sulfuryl chloride may be very violent and in such cases it should be moderated by cooling. The optimum temperature for the reaction varies for different hydrocarbons, but can readily be ascertained. The sulfurous acid which is set free during the reaction remains dissolved in the reaction product and is driven off and can be used again for the manufacture of sulfuryl chloride.

The following example will serve to illustrate further the nature of my invention, which, however, is not confined to this example. The parts are by weight. Thoroughly cool about eighty parts of trimethylethylene (amylene) and add to it (slowly and while stirring well) about one hundred and thirty-five parts of cold sulfuryl chloride, regulating the temperature so that

the liquid remains at a temperature of between minus two degrees and zero centigrade. Continue stirring for some time after the addition is effected. Distill off the sulfurous acid and the excess of trimethylethylene and purify the residue, if desired, by distilling *in vacuo*. The dichloropentane thus obtained boils at from forty-eight to fifty-two degrees centigrade at forty-eight millimeters pressure. This example can be varied in many respects, for instance if it is desired to produce the dichloride continuously, the reagents can be separately cooled and run together in a worm in which they gradually assume the temperature at which the reaction takes place. My invention can also be similarly applied to the chlorination of the other olefinic hydrocarbons.

Now what I claim is:—

1. The process of making chlorinated hydrocarbons by treating olefinic hydrocarbons with sulfuryl chloride.

2. The process of making chlorinated hydrocarbons by treating an excess of olefinic hydrocarbon with sulfuryl chloride, distilling off the sulfur dioxide and excess of olefinic hydrocarbon and purifying the resulting chlorinated hydrocarbon by distillation.

3. The process of making chlorinated hydrocarbons by treating amylene with sulfuryl chloride.

4. The process of making chlorinated hydrocarbons by treating an excess of amylene with sulfuryl chloride, distilling off the sulfur dioxide and excess of olefinic hydrocarbon and purifying the resulting chlorinated hydrocarbon by distillation.

In testimony whereof I have hereunto set my hand in the presence of two subscribing witnesses.

OTTO GRAUL.

Witnesses:

J. ALEC. LLOYD,
JOSEPH PEIFFER.