

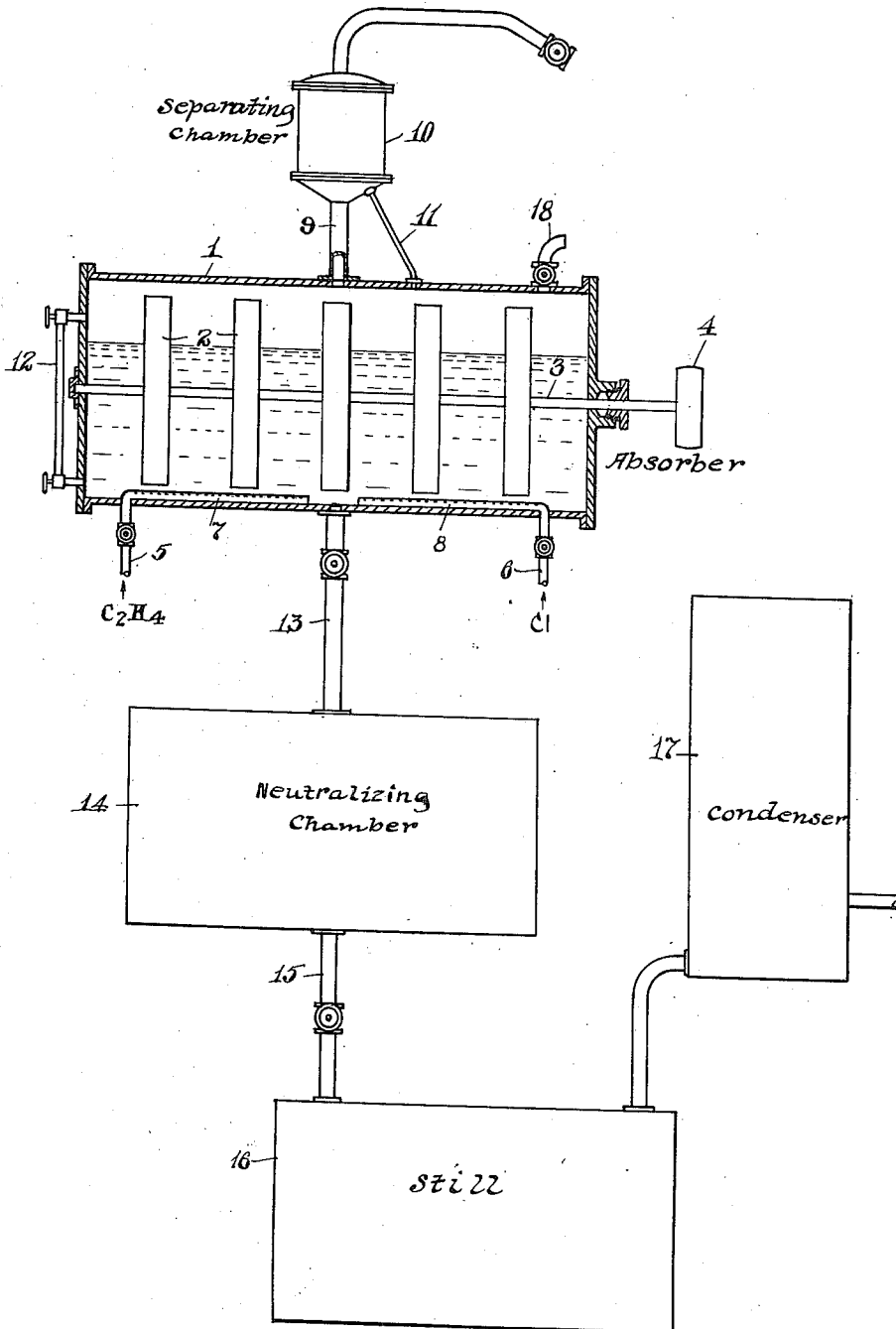
Jan. 29, 1924.

W. E. KIRST

1,482,306

APPARATUS FOR MAKING HALOGENATED PRODUCTS

Filed Oct. 17, 1919



INVENTOR
William E. Kirst
By Day, Oberlin & Day
ATTORNEYS.

UNITED STATES PATENT OFFICE.

WILLIAM E. KIRST, OF SAGINAW, MICHIGAN, ASSIGNOR TO THE DOW CHEMICAL COMPANY, OF MIDLAND, MICHIGAN, A CORPORATION OF MICHIGAN.

APPARATUS FOR MAKING HALOGENATED PRODUCTS.

Application filed October 17, 1919. Serial No. 331,446.

To all whom it may concern:

Be it known that I, WILLIAM E. KIRST, a citizen of the United States, and a resident of Saginaw, county of Saginaw, and State of Michigan, have invented a new and useful Improvement in Apparatus for Making Halogenated Products, of which the following is a specification, the principle of the invention being herein explained and the best mode in which I have contemplated applying that principle, so as to distinguish it from other inventions.

The present improvements, relating, as indicated, to the manufacture of halogenated products, have regard more particularly to the manufacture of chlorinated (or brominated) derivatives of ethylene, e. g. glycol chlorhydrin (C_2H_4OH or $CH_2Cl.CH_2OH$). Compounds of this character are sometimes referred to as halogen hydrins by way of general designation, it being understood that other halogen derivatives, as well as chlorhydrins, may be made by the same method or process, and employing the same or similar apparatus as that hereinafter described.

Approved methods for obtaining chlorhydrin, as described in the texts (e. g. see Bernthsen's Organic Chemistry), include passing hydrogen chloride into warm glycol, and the addition of hypochlorous acid to ethylene. The object of the present invention is the production of such chlorhydrin or related halogen derivative as directly as possible from the halogen and ethylene, and while a hypohalogenous acid may be formed as an intermediate, the reactions occur practically continuously, and it is not required to handle the acid as such.

To the accomplishment of the foregoing and related ends, the invention, then, consists of the means hereinafter fully described and particularly pointed out in the claims, the annexed drawing and the following description setting forth in detail one type of apparatus embodying the invention, such disclosure, however, constituting but one of various ways in which the principle of the invention may be used.

In said annexed drawing:—

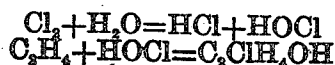
The single figure there appearing illustrates, in a more or less diagrammatic fashion, an arrangement of apparatus embodying my present improvements.

As illustrated in the drawing in question,

the principal element of the apparatus is a suitably lined horizontal cylindrical stationary absorber 1 equipped with an agitator in the form of a series of paddles 2 carried by an axial shaft 3 provided with a driving pulley 4 exterior of one end of the absorber. Suitable pipes 5 and 6, having perforated extensions 7 and 8 respectively disposed within the chamber along its bottom wall, provide means for introducing ethylene and chlorine as indicated. Connected with the upper wall of the absorber by means of a suitable pipe 9 is a separating chamber 10, through which gas may be drawn off without interrupting operation of the absorber, while any liquor that is carried along with such gas is allowed to run back through another connection 11. A sight glass 12 at one end of the absorber chamber serves to indicate the liquid level within the latter.

Leading from the bottom of the absorber chamber is a valve-controlled discharge pipe 13, through which the liquid contents of the chamber may be conducted to a neutralizing chamber 14 of any suitable construction. From the latter in turn the liquid may be transferred through a valve-controlled pipe 15 to a suitable still 16, having connected therewith a condenser 17.

Having thus described the arrangement of apparatus, the operation thereof may be now set forth. The ethylene and chlorine are passed, as previously indicated, through pipes 5 and 6 into the closed absorber or reaction chamber. Here the two gases mutually dissolve and react according to the following equations:—



A concentration of chlorhydrin can thus be obtained as high as fifteen per cent by weight, this apparently being the maximum, as the reaction stops at this point. The supply of ethylene and chlorine is then shut off, and the charge is run to the neutralizing tank or chamber 14, where the hydrochloric acid in such charge is removed with lime. The transfer of the charge from the absorber to the neutralizer may be facilitated by admitting a suitable gas under pressure above the same through a valve-controlled pipe 18 provided for the purpose. The neutralized charge is next transferred to still 16 and distilled through column 17 which

readily permits the securing of a forty per cent chlorhydrin portion. Such portion can then be subjected to further purification or concentration, this, however, forming no part of the present invention.

It will be observed that an unusually high concentration of chlorhydrin is obtainable by the use of the foregoing process and apparatus. It is also unnecessary to have any base present in the reaction, the latter in effect occurring directly between the ethylene and the halogen.

While the process in hand has been described with specific reference to the making of glycol chlorhydrin, it will be understood that such process is not necessarily limited to the particular olefin, viz, ethylene, from which this compound is derived, but that other of the gaseous and volatile olefins, such as propylene, the butylenes, the amylenes, etc., hereinafter referred to generally as olefinic bodies, so far as they are capable of producing compounds of the class of chlorhydrin may be similarly utilized in the manner described. On the other hand, as previously indicated, such apparatus is equally adaptable for the manufacture of other halohydrins, and particularly of bromhydrins, since bromine in vapor form can of course be introduced into the absorber in the same fashion as chlorine gas and the remaining steps of the operation will follow in the same sequence.

Other modes of applying the principle of

my invention may be employed instead of the one explained, change being made as regards the mechanism herein disclosed, provided the means stated by any of the following claims or the equivalent of such stated means be employed.

I therefore particularly point out and distinctly claim as my invention:—

1. In an apparatus for making halogenated products, the combination of a horizontal cylindrical stationary absorbing chamber, an agitator comprising an axial shaft within said chamber and a series of paddles carried by said shaft, and separate pipes for supplying a halogen and an olefinic body to said chamber, said pipes having perforated extensions, respectively, disposed within said chamber along its lower wall.

2. In an apparatus for making halogenated products, the combination of a horizontal cylindrical stationary absorbing chamber, a separator connected with the upper wall of said chamber, an agitator comprising an axial shaft within said chamber and a series of paddles carried by said shaft, and separate pipes for supplying a halogen and an olefinic body to said chamber, said pipes having perforated extensions, respectively, disposed within said chamber along its lower wall.

Signed by me this 9th day of October, 1919.

WILLIAM E. KIRST.