

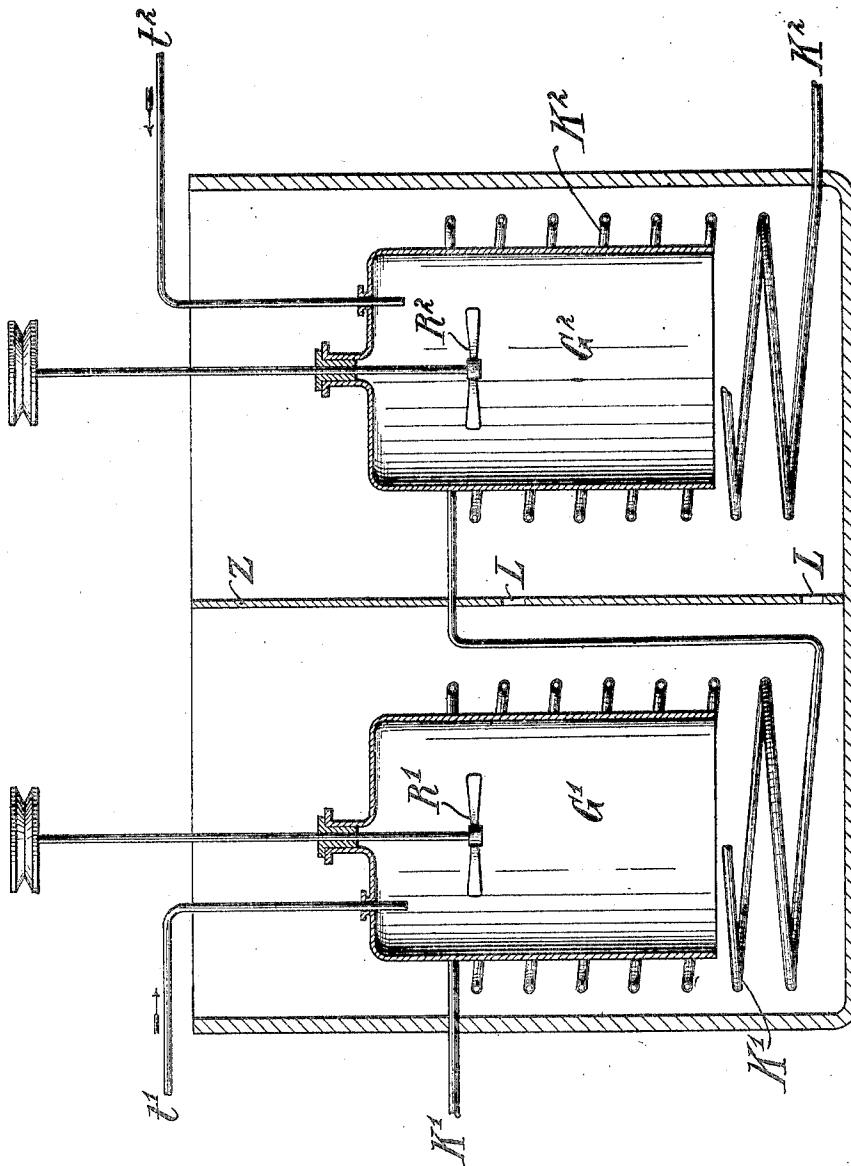
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P. ASKENASY & M. MUGDAN.

PROCESS OF MAKING ACETYLENE TETRACHLORID.

APPLICATION FILED NOV. 16, 1903.



Witnesses:

James M. Brown
Goldwin W. Underhill

Inventors:

Paul Askenasy
Martin Mugdan
by *Eustace W. Hopwood*
Att.

UNITED STATES PATENT OFFICE.

PAUL ASKENASY AND MARTIN MUGDAN, OF NUREMBERG, GERMANY.

PROCESS OF MAKING ACETYLENE TETRACHLORID.

No. 804,516.

Specification of Letters Patent.

Patented Nov. 14, 1905.

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To all whom it may concern:

Be it known that we, PAUL ASKENASY and MARTIN MUGDAN, subjects of the German Emperor, and residents of Nuremberg, Bavaria, German Empire, have invented a new and useful Process of and Apparatus for Producing Tetrachlorid of Acetylene, of which the following is a description.

As is known, the action of acetylene on pentachlorid of antimony produces compounds which are violently decomposable and from which compounds of acetylene and chlorin may be split off ("Acetylendi and Tetrachlorid," Berthelot and Jungfleisch, *Annales der Chemie und Pharm.*, VII. Suppl., 1870, page 252 and following pages.) A second method of producing tetrachlorid of acetylene is based on the action of chlorin on acetylene with the aid of chlorid of aluminium dissolved in acetylene chlorid or tetrachlorid of carbon, in connection with which process, however, it is absolutely necessary to exclude the very smallest quantity of air in order to avoid explosions. (Mouneyrat.) Many experiments have also been made with and without the aid of a cheap or indestructible intermediate substance to combine acetylene and chlorin without danger; but these experiments have hitherto had no technically exploitable result.

The process forming the object of the present invention enables the cheap and convenient production of acetylene tetrachlorid without danger and with a very slight loss of the intermediate substance.

Acetylene is fed into pentachlorid of antimony which should advantageously contain no free chlorin. The pentachlorid of antimony may be diluted with a suitable medium, such as tetrachlorid of acetylene. In the first place the compound described by Berthelot and Jungfleisch will be formed, which compound melts at about 50° centigrade. This compound, however, as we have found, in contradistinction to Berthelot's statement, will on further treatment with acetylene take up a second molecule of the gas. Berthelot and Jungfleisch heated the double compound of bichlorid of acetylene and pentachlorid of antimony with a surplus of the latter, (last cited page, 255, at the top.) We, however, feed chlorin into the product obtained from pentachlorid of antimony and acetylene, four atoms of the chlorin being bound by each molecule of acetylene present, tetrachlorid of acetylene being formed and pentachlorid of

antimony being re-formed. Acetylene is then fed into the same fluid, and then again chlorin, and so on alternately until, finally, if sufficient chlorin is fed, a mixture of pentachlorid of antimony, tetrachlorid of acetylene, and small quantities of the products of substitution of the latter body is obtained. If the pentachlorid of antimony is diluted to a very great extent by the tetrachlorid of acetylene produced, the reactions will still take place with sufficient speed for economical purposes.

In order to isolate the tetrachlorid of acetylene, the pentachlorid of antimony may be decomposed—for instance, by means of hydrochloric acid. It is, however, advantageous to subject the product of the reaction to a slow fractionated distillation, in which case the pentachlorid of antimony changes to trichlorid of antimony by splitting off the chlorin for the most part, partly as escaping gas, partly by chlorinating the tetrachlorid of acetylene to penta or hexa chlorethane. The trichlorid of antimony, which has a very high boiling-point, (220°,) may be easily separated from the tetrachlorid of acetylene, which boils at 146°.

The following are the details of a set of experiments: Two thousand nine hundred and seventy grams (—10 molecules) SbCl₅ absorbed consecutively four hundred and sixty-six grams (—18 molecules) C₂H₂ and two thousand five hundred grams (—71 atoms) Cl, then four hundred and fifty grams (—17.3 molecules) C₂H₂ and two thousand three hundred and ten grams (—65.5 atoms) Cl, then three hundred and seventy grams (—14.2 molecules) C₂H₂ and eighteen hundred and ninety grams (—53.5 atoms) Cl, then two hundred and eighty grams (—10.8 molecules) C₂H₂ and seventeen hundred and sixty grams (—49.8 atoms) Cl, then three hundred and twenty grams (—12.3 molecules) C₂H₂ and two thousand one hundred grams (—59.5 atoms) Cl. About half the liquid obtained (seven thousand six hundred and fifty grams) was subjected to fractionated distillation during which at first chlorin and hydrochloric acid escaped. At a temperature above 215° centigrade eight hundred and sixty grams—i. e., seventy-six per cent.—of the theoretical quantity of trichlorid of antimony distilled over. From the part which distilled below 148° centigrade five thousand nine hundred grams of crude tetrachlorid of acetylene could be obtained. The trichlorid of antimony after having been converted into pentachlorid was fit for use for a new operation.

The chlorin and acetylene may also be fed in simultaneously; but in this case they should enter the apparatus at different points. In this case the apparatus must be constructed in the first place so that the two gases do not come into contact with each other in order to avoid explosions, and, secondly, so that the absorbent liquid may circulate in order that the liquid treated with acetylene may come into contact with that treated with chlorin before the former is saturated, and vice versa.

In the accompanying drawing one form of apparatus is illustrated in the form of a diagram.

Chlorin flows into the bell G' through the pipe t' and acetylene flows into the bell G² through the pipe t'' . The liquid is intimately mixed with the gases by means of the stirrers R' and R². The heat generated by the reaction is taken off in the cooling-coils K' and K². The liquid circulates through the two orifices L in the partition-wall Z.

Acetylene tetrachlorid is particularly suited on account of its cheapness for extracting fat, dissolving resins, oils, and the like.

We claim as our invention—

1. A process for producing tetrachlorid of

acetylene, which consists in treating chlorid of antimony with acetylene and chlorin and then separating the tetrachlorid of acetylene from the resulting product by fractionation, substantially as described.

2. A process for producing tetrachlorid of acetylene, which consists in treating the product of the reaction of acetylene and chlorid of antimony with chlorin and acetylene and then separating the tetrachlorid of acetylene from the resulting product by fractionation, substantially as described.

3. A process for producing tetrachlorid of acetylene, which consists in treating the product of the reaction of acetylene and chlorid of antimony with chlorin and acetylene alternately and then separating the tetrachlorid of acetylene from the resulting product by fractionation substantially as described.

In witness whereof we have hereunto set our hands in presence of two witnesses.

PAUL ASKENASY.
MARTIN MUGDAN.

Witnesses:

LUDWIG SCUNINGER,
ALBERT BESOLD.