PATENT SPECIFICATION

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PROVISIONAL SPECIFICATION.

Improvements in the Production of \( \beta \) \( \beta \)-dichlorehyl Sulphide.

We, WILLIAM JACKSON POPE, of "Holmestead," Brooklands Avenue, Cambridge, Chemist, CHARLES STANLEY GIBSON, The Chemical Laboratory, Pembroke Street, Cambridge, Chemist, and Major General HENRY FLEETWOOD THUILLIER, Controlling Chemical Warfare Department, Avenue House, 21, Northumberland Avenue, London, W.C. 2, do hereby declare the nature of this invention to be as follows:—

This invention relates to the production of organic compounds.

The invention consists in producing \( \beta \) \( \beta \)-dichlorehyl sulphide (CH\( _2 \)\( _3 \)\( _2 \)Cl\( _2 \))\( _2 \)S by causing ethylene and sulphur monochloride, S\( _2 \)Cl\( _2 \), to react together.

The production of the compound referred to may be effected by bringing the ethylene and sulphur monochloride together under suitable temperature conditions. The production may, for instance, be effected in the cold or at slightly elevated temperatures, but it may be pointed out that the yield is influenced by the temperature conditions under which the reaction is carried out.

We have found, for instance, that while a 10 per cent. yield only may be obtained in the cold, a yield of upwards of 90 per cent. of the amount theoretically obtainable may be obtained when the reaction is effected at temperatures between 50° and 70° C.

If desired, the reaction may be carried out in the presence of suitable solvents, or under pressure. Catalytically acting materials may be employed for facilitating or accelerating the reaction.

The reaction taking place between the ethylene and sulphur monochloride may be represented by the following equation:—

\[
S\text{Cl}_2 + 2C\text{H}_4 \rightarrow \text{Cl(\text{CH}_2\text{CH}_2\text{S} + S.}
\]

The process of producing \( \beta \) \( \beta \)-dichlorehyl sulphide may be carried out, in accordance with the invention, by passing dry ethylene into cold or warm sulphur monochloride, until the reaction ceases. A mixture of sulphur with \( \beta \) \( \beta \)-dichlorehyl sulphide or a solution of sulphur in this body will thus be obtained from which the \( \beta \) \( \beta \)-dichlorehyl sulphide may be separated in any suitable manner.

Dated this 3rd day of April, 1918.

MARKS & CLERK.
COMPLETE SPECIFICATION.

Improved in the Production of $\beta$ $\beta$-dichlorethyl Sulphide.

We, WILLIAM JACKSON POPE, of "Holmestead," Brooklands Avenue, Cambridge, in the County of Cambridge, Chemist, CHARLES STANLEY GIBSON, of The Chemical Laboratory, Pembroke Street, Cambridge, in the County of Cambridge, Chemist, and HENRY FLEETWOOD THILLIER, Major-General, Controller, Chemical Warfare Department, Avenue House, 21, Northumberland Avenue, London, W.C. 2, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:

This invention relates to the production of organic compounds.

The invention consists in producing $\beta$ $\beta$-dichlor-ethyl sulphide ($\text{CH}_2\text{CH}_2\text{Cl}_2\text{S}$) by causing ethylene and sulphur monochloride, $\text{S}_2\text{Cl}_2$, to react together.

The production of the compound referred to may be effected by bringing the ethylene and sulphur monochloride together under suitable temperature conditions. The production may, for instance, be effected in the cold or at slightly elevated temperatures, but it may be pointed out that the yield is influenced by the temperature conditions under which the reaction is carried out.

We have found, for instance, that while a 10 per cent. yield only may be obtained in the cold, a yield of upwards of 90 per cent. of the amount theoretically obtainable may be obtained when the reaction is effected at temperatures between 50° and 70° C.

If desired, the reaction may be carried out in the presence of suitable solvents, or under pressure. Catalytically acting materials may be employed for facilitating or accelerating the reaction.

The reaction taking place between the ethylene and sulphur monochloride may be represented by the following equation:

$$\text{S}_2\text{Cl}_2 + 2\text{C}_2\text{H}_4 \rightarrow \text{ClCH}_2\text{CH}_2\text{S} + \text{S}.$$

The process of producing $\beta$ $\beta$-dichlorethyl sulphide may be carried out, in accordance with the invention, by passing dry ethylene into cold or warm sulphur monochloride, until the reaction ceases. A mixture of sulphur with $\beta$ $\beta$-dichlor-ethyl sulphide or a solution of sulphur in this body will thus be obtained from which the $\beta$ $\beta$-dichlorethyl sulphide may be separated in any suitable manner.

The following particulars are given by way of example to illustrate methods of carrying the invention into effect:

1. A current of well-dried ethylene gas is passed into sulphur monochloride previously heated to 60° C.; the absorption of the gas is facilitated by vigorous agitation of the liquid and by the previous addition of a small proportion of $\beta$ $\beta$-dichlorethyl sulphide. Care should be taken to keep the temperature at or about 60° C. and, as the reaction is highly exothermic, this may necessitate the provision of special cooling arrangements if the quantity of material dealt with is large. Absorption proceeds at a fairly uniform rate and the liquid becomes turbid at a certain stage in the reaction. When absorption is at an end owing to the theoretical quantity of ethylene having been taken up, it is convenient, but not essential to heat the product to about 100° C. and then allow it to cool to the ordinary temperature; the greater part of the sulphur liberated in the reaction then crystallises out and the $\beta$ $\beta$-dichlorethyl sulphide may be poured off. In a properly conducted operation an almost theoretical
yield of \( \beta \) \( \beta \)-dichlorethyl sulphide containing little in solution besides about 5 per cent. of sulphur may be obtained.

2. A current of dried ethylene gas, as free as possible from alcohol vapour, is brought into intimate contact with sulphur monochloride maintained at 30° C. The absorption is much less rapid than in example 1 and the liquid does not become turbid or deposit sulphur on cooling. After absorption ceases pure \( \beta \) \( \beta \)-dichlorethyl sulphide may be separated from the product by distillation or other suitable treatment.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:

1. The process of producing \( \beta \) \( \beta \)-dichlorethyl sulphide which comprises causing ethylene and sulphur monochloride to react together.
2. The process of producing \( \beta \) \( \beta \)-dichlorethyl sulphide, substantially as here-

Dated this 2nd day of October, 1918.

MARKS & CLERK.

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