

PATENT



SPECIFICATION

Application Date, Apr. 3, 1918. No. 5686/18.

Complete Left, Oct. 2, 1918.

Accepted, Nov. 5, 1918 (but withheld from Publication under Section 30 of Patents and Designs Acts, 1907 and 1919).

Authorised to be Published, Apr. 22, 1920. Date of Publication, June 10, 1920.

PROVISIONAL SPECIFICATION.

Improvements in the Production of β β -dichlorethyl Sulphide.

We, WILLIAM JACKSON POPE, of "Holmesdale," Brooklands Avenue, Cambridge, Chemist, CHARLES STANLEY GIBSON, The Chemical Laboratory, Pembroke Street, Cambridge, Chemist, and Major General HENRY FLEETWOOD THULLIER, Controller 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.

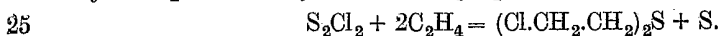
The invention consists in producing β β -dichlorethyl sulphide ($\text{CH}_2\text{CH}_2\text{S}$) by causing ethylene and sulphur monochloride, S_2Cl_2 , to react together.

10 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

15 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.

20 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:—



The process of producing β β -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 β β -dichlorethyl sulphide or a solution of sulphur in this body will thus

30 be obtained from which the β β -dichlorethyl sulphide may be separated in any suitable manner.

Dated this 3rd day of April, 1918.

MARKS & CLERK.

COMPLETE SPECIFICATION.

Improvements in the Production of β β -dichlorethyl Sulphide.

We, WILLIAM JACKSON POPE, of "Holmesdale," 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 THUILLIER, Major-General, 5
 Contoller, Chemical Warfare Department, Avenue House, 21, Northumber-
 land 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.

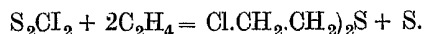
The invention consists in producing β β -dichlor-ethyl sulphide ($\text{CH}_2\text{CH}_2\text{S}$ 10
 $\text{CH}_2)_2\text{S}$ by causing ethylene and sulphur monochloride, S_2Cl_2 , to react together.

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 15
 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 theoretic- 20
 ally 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 25
 may be represented by the following equation :—



The process of producing β β -dichlorethyl sulphide may be carried out, in
 accordance with the invention, by passing dry ethylene into cold or warm 30
 sulphur monochloride, until the reaction ceases. A mixture of sulphur
 with β β -dichlor-ethyl sulphide or a solution of sulphur in this body will thus
 be obtained from which the β β -dichlor-ethyl 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 :— 35

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 β β -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 40
 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 45
 allow it to cool to the ordinary temperature; the greater part of the sulphur
 liberated in the reaction then crystallises out and the β β -dichlorethyl sulphide
 may be poured off. In a properly conducted operation an almost theoretical

yield of β β -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 β β -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 β β -dichlorethyl sulphide which comprises causing ethylene and sulphur monochloride to react together.

2. The process of producing β β -dichlorethyl sulphide, substantially as herebefore described.

Dated this 2nd day of October, 1918.

MARKS & CLERK.