Patented Jan. 19, 1954 2,666,776

UNITED STATES PATENT OFFICE

2,666,776

PRODUCTION OF ORGANIC SILICON COMPOUNDS

Siegfried Nitzsche, Burghausen, Upper Bavaria, Germany, assignor, by mesne assignments, to Wacker-Chemie G. m. b. H., Munich, Germany

No Drawing. Application April 19, 1950, Serial No. 156,947

Claims priority, application Germany May 5, 1949

7 Claims. (Cl. 260—448.2)

1

This invention relates to the production of organosilicon halides and it has for its object to provide a novel and improved process for this purpose.

Another object of the invention is to provide a simple and economical catalytic process for producing organosilicon halides, and particularly alkyl and aryl compounds thereof, with greater uniformity of conversion and higher yield than has heretofore been possible.

Various other objects and advantages will be apparent as the nature of the invention is more fully disclosed.

The present application is a continuation-in-part of application Serial No. 140,157, filed January 23, 1950.

It is possible to produce alkyl or aryl halides of silicon by reacting gaseous aryl or alkyl halides with silicon in the presence of copper, nickel, antimony, copper oxide or copper chloride. However, the various possible combinations of these materials yield fluctuating transformations and outputs, so that it is necessary for economic reasons to use catalysts which promote uniform transformation and high output.

I have found that these requirements are attained to a satisfactory degree, and that aryl or alkyl silicon halides, particularly dialkyl or diaryldihalogensilanes, are obtained if an alloy is used which contains besides silicon and copper also a metal of the 5th to 8th groups of the periodic table, particular cobalt, nickel, iron or phosphorus. These bodies can be used as powders, lumps, or as pressings. An additional increase in efficiency is attained if the catalyst is used in connection with an activator, for example a copper salt. For this purpose the catalyst is wetted or coated with solutions or pastes of copper salts. As such inorganic salts, particularly copper chloride or salt mixtures are preferred above all other. The activation may be performed before or during the conversion. It is also possible to bring about the formation of metal salts on the catalyst through corresponding transformations before or during the production of the organosilicon compound; thereby the desired oxidation stages may be adjusted or maintained before or during the transformation.

For the production of the catalyst the procedure is preferably such that an alloy of silicon, copper, and a metal of the 5th to 8th groups, for example cobalt, nickel, iron or phosphorous, is melted under slag or an inert gas. The finished alloy is reduced to pieces or ground

2

into a powder which is pressed into tablets. In order to activate this alloy the pieces may be wet with a copper chloride solution or paste, or the powdered alloy may be mixed with such solution and then compressed. The copper salt may also be formed by reaction of copper with an acid, such as hydrochloric acid, before or during the transformation. In the latter case the acid may be introduced into the reaction simultaneously with the halogen compounds. If the halide is conducted at an elevated temperature preferably between 250 and 380° C. over a catalyst such as that described above, the silicon is transformed uniformly and with a good output to the corresponding organic silicon compounds which contain chiefly dialkyl or aryldihalogensilanes.

Example I

An alloy consisting of silicon, copper and cobalt is melted and then reduced in the manner described above. Ethyl chloride is conducted in contact with this alloy at an elevated temperature as previously described. Ethylchlorosilanes are produced with a good output.

Example II

First an alloy of silicon, copper and nickel is melted up, and is ground to a powder after cooling. The powder is mixed with copper chloride paste and pressed into tablet form. Over this catalyst methyl chloride is passed for a period of 24 hours at a temperature of about 275° C. Of the conversion mixture formed in the quantity of 180 to 200 g. more than 60% consists of dimethyl dichlorsilane and other valuable alkylchlorsilanes When ethyl chloride is used as a starting material, one of the principal products is diethyl dichlorsilane.

Example III

An alloy of ferrosilicon (90% silicon) and copper (about 20–30%) is prepared. This is pulverized and the powder is treated with a solution of Cu-hydrochloric acid, which suitably contains some copper in bivalent form. After drying, the powder is converted with methyl chloride, preferably at 270–300° C. After 21 hours 6.690 kg. raw product was formed from about 2.1 kg. of treated silicon. The reaction product contained about 2,400 g. of pure dimethyldichlorsilane and 2,170 g. pure methyl trichlorsilane, up to 42° C. about 380 g. were transformed, the rest was distributed to intermediate fractions of 42.5–50° C., 50–60° C., 60–63° C., as well as a residue of 300 g. B. P. over 70° C.

(730 mm.). After the expiration of the above-mentioned 21 hours 5% of hydrochloric acid was added to the methyl chloride, and there was formed in the course of about the next succeeding 5 hours, at a somewhat higher temperature, a further kg. raw product, which consisted mainly of methylchlorosilanes. Usually when an iron containing silicon copper alloy of about 6 per cent iron is used, about 8 kg. of silanes are formed from each 10 kg. of methyl chloride.

Example IV

An alloy of silicon, copper and nickel which contains 1400 g. silicon is mixed with cuprous chloride. During a period of 26 hours methyl chloride is passed over this material at a temperature of about 275–280° C. 5.7 kg. product is obtained containing about 2.3 kg. pure dimethyldichlorosilane, 1.9 kg. pure methyltrichlorosilane and a remainder chiefly consisting out of $(CH_3)_3SiCl$ and $HSiCH_3Cl_2$ and interfractions. The total quantity of methyl chloride used is about 6.3 kg.

Although certain specific embodiments have been disclosed herein for purposes of illustration, it will be evident to those skilled in the art that the invention is capable of various modifications and adaptations within the scope of the appended claims.

The invention claimed it:

1. The process for producing organohalogenosilanes which comprises reacting a material selected from the group consisting of aryl and alkyl halides in vapor phase with an alloy consisting essentially of silicon, copper and a metal selected from the group consisting of iron, cobalt, and nickel, in the presence of a copper salt.

2. The process according to claim 1 in which the halide is methyl chloride.

3. The process according to claim 1 in which the halide is ethyl chloride.

4. The process according to claim 1 in which the reaction is conducted at a temperature of about 250 to 380° C.

5. The process according to claim 1 in which the copper salt is a copper choride.

6. The process according to claim 1 in which the halide employed contains also hydrogen halide.

7. A reaction mass for use in the production of organosilicon halides consisting essentially of an alloy of silicon, copper and a metal selected from the group consisting of iron, cabalt, and nickel, which reaction mass further contains a copper salt.

SIEGFRIED NITZSCHE.

References Cited in the file of this patent

UNITED STATES PATENTS

Number	Name	Date
1,777,174	Machin	Sept. 30, 1930
2,380,995	Rochow	Aug. 7, 1945
2,464,033	Gilliam	Mar. 8, 1949
2,488,487	Barry et al.	Nov. 15, 1949