SHORT COMMUNICATIONS

Preparation of ethyldisilane

Introduction

It has been reported that ethyllithium reacts with silane to form ethylsilanes[1]. Although a number of alkyldisilanes containing a silicon–hydrogen bond have been prepared[2–4], no reaction between an alkyllithium compound and a disilane have been reported. In this note, we describe the synthesis of ethyldisilane from the reaction between ethyllithium and disilane. A minor unisolated product from the reaction between ethylene and silane at 450–510° was tentatively identified as ethyldisilane[5]. Our work confirms that this unisolated product was ethyldisilane.

Experimental

A standard high-vacuum system was used for this investigation. The known volatile products were identified by their infrared spectra and vapor pressures as reported in the literature. Disilane was prepared by the reduction of hexachlorodisilane[6].

Benzene and 1,2-dimethoxyethane were dried with molten potassium. The ethyllithium was obtained from the Lithium Corporation of America as a 2 *M* slurry in benzene. Transfer of ethyllithium was carried out in a dry bag with a nitrogen atmosphere.

Infrared spectra were obtained in 10 cm gas phase cells on a Perkin-Elmer Spectrometer Model 137. The proton NMR spectrum of ethyldisilane was obtained in benzene with tetramethylsilane as an external standard on a Varian A-60 NMR spectrometer.

Preparation of ethyldisilane

(*1*) *Reaction time 15 minutes.* Disilane (2.46 mmoles) and benzene (2 ml) were condensed into a 100 ml reaction vessel containing 2 mmoles of ethyllithium in 1 ml of benzene. The reaction mixture was stirred at room temperature for 15 minutes. The volatile materials were distilled from the reactor and separated by fractional condensation. The products were silane (0.16 mmole) and ethyldisilane (0.74 mmole); 1.3 mmole of unreacted disilane was recovered.

(*2*) *Reaction time 1 hour.* The reaction between disilane (2.42 mmoles) and ethyllithium (1 mmole) was repeated as described above. After a reaction time of 1 h, the volatile products were silane (0.72 mmole) and ethyldisilane (0.21 mmole); 0.98 mmole of unreacted disilane was recovered.

(*3*) *Reaction time 15 hours.* When the reaction described above was carried out for 15 hours with disilane (1.03 mmole) and ethyllithium (1 mmole), the volatile products were silane (1.19 mmole) and ethylsilane (0.09 mmole). All of the disilane had been consumed.

Reaction of ethyldisilane with potassium hydride

Ethyldisilane (0.47 mmole) and 1,2-dimethoxyethane (2 ml) were condensed onto potassium hydride (1 mmole) in a 50-ml reactor fitted with a Teflon needle valve and a standard taper joint. The reaction was carried out at room temperature for 10 min. The volatile products were silane (0.25 mmole) and ethylsilane (0.23 mmole) while 0.02 mmole of ethyldisilane was recovered unreacted. All of the volatile materials were removed from the reactor and then the solid products were treated with excess hydrogen chloride. The products from this reaction were ethylsilane (0.045 mmole) and about 0.005 mmole each of silane, disilane and ethyldisilane.

Identification of ethyldisilane

The identification of ethyldisilane is based on the following results. (*1*) The ethylsilane obtained from the reaction with potassium hydride establishes that a silicon–ethyl bond had been present. (*2*) The infrared spectrum (Table 1) shows the presence of a silicon–hydrogen bond. (*3*) A molecular weight (by vapor density) of 90.05 (theoretical 90.28) was obtained. (*4*) The only two compounds containing silicon–ethyl bonds and silicon–hydrogen bonds with a molecular weight near 90 are ethyldisilane and diethylsilane; the infrared spectrum of our sample rules out diethylsilane[7]. (*5*) The proton NMR spectrum (Table 2) is consistent with ethyldisilane and is reasonably analogous to that reported for methyldisilane[8].

TABLE 1

INFRARED SPECTRUM OF $C_2H_5Si_2H_5$

cm^{-1}	*Relative strength*	*Assignment*[a]
2920	strong	C–H stretch
2120	very strong	Si–H stretch
1460	medium	C_2H_5 deform.
1292	weak	C_2H_5 deform.
1030	medium	
940	strong	Si_2H_5 deform.
893–883	very strong	Si_2H_5 deform.
793	very strong	C_2H_5 deform.
743	medium	Si–C stretch coupled
676	medium	with Si_2H_5 deform.

[a] The assignments are based on results and conclusions reported in the literature[2,7]. The last four assignments must be considered tentative.

TABLE 2

PROTON NMR SPECTRUM OF $C_2H_5Si_2H_5$ (Me_4Si AT 0.00)

Absorption[a]	*Relative integrated intensity*	*Assignment*
poorly defined quartet at −2.98 ppm	2.00	SiH_2
triplet at −2.55 ppm	2.92	SiH_3
complex system −0.4 to +0.5 ppm	5.02	C_2H_5

[a] Chemical shifts with a minus sign are downfield from tetramethylsilane.

Vapor pressure

The vapor pressure of ethyldisilane obeys the following equation from −63 to 25°:

$$\log P_{mm} = \frac{-1364.2}{T} + 6.9918$$

Results and discussion

Ethyldisilane was prepared by the reaction between ethyllithium and disilane.

$$LiC_2H_5 + Si_2H_6 \xrightarrow{C_6H_6} C_2H_5Si_2H_5 + LiH$$

The yield of ethyldisilane is dependent on the reaction time as the product is converted into silane if allowed to remain in contact with the reaction mixture. A yield of 65% was obtained for a reaction time of 15 minutes.

Ethyldisilane reacts with potassium hydride producing ethylsilane and silane as the volatile products. When the solid products were treated with hydrogen chloride, ethylsilane was produced along with much smaller quantities of silane, disilane and ethyldisilane. These results suggest that the ethylsilyl, silyl, disilanyl and ethyldisilanyl salts of potassium[8,9] were also products from the reaction between potassium hydride and ethyldisilane. Since the anion fraction did not account for all of the silicon present in the solid, polymeric silanes were probably also produced.

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Department of Chemistry, San Diego State College, W. J. Boldūc
San Diego, California 92115 (U.S.A.) M. A. Ring

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