SHORT COMMUNICATION

The synthesis of *O*-silyl-substituted enols and related compounds

Satisfactory yields of *O*-silyl-substituted enols are obtained by reacting sodium enolates of carbonyl compounds with alkylhalosilanes as described in a paper by Krüger and Rochow[1].

We are now conducting large-scale investigations on the reactions of organometallic (Hg, Sn, Ge) compounds containing an ester or carbonyl group in the β-position with respect to the metal atom, with the hydrides or halides of some organometallic compounds. In particular, the reaction of halosilanes with α-trialkylstannylketones give excellent results in the synthesis of pure (α-alkylvinyloxy)silanes.

$$\equiv SiCl + R_3SnCH_2COR' \longrightarrow \equiv Si{-}O{-}C(R')=CH_2 + R_3SnCl$$

Thus, as a result of the action of trimethylchlorosilane on tripropylacetonyltin, 2-(trimethylsiloxy)propene, $CH_2=C(CH_3)OSi(CH_3)_3$, is obtained. The yield is 80 %; b.p. 92–94°/758 mm; n_D^{20} 1.3988; d_4^{20} 0.7873; MR_D 40.01, calcd. 39.83. (Found: C, 55.10; H, 10.95; Si, 20.88. $C_6H_{14}SiO$ calcd.: C, 55.33; H, 10.83; Si, 21.55 %.)

An intensive absorption band with a frequency of 1652 cm^{-1} was detected in the IR spectrum of the above compound, corresponding to the valency oscillations of a C=C double bond.

Krüger and Rochow[1] could not separate this compound in a pure state and reported that the boiling point was between 101 and 126°, which is much higher than we found.

Using ethyl acetate and trimethylchlorosilane they obtained the *O*- and *C*-derivatives with yields of 13.7 and 22.3 %, respectively; these isomers can be separated only by gas chromatography. We have already published[2] details of the synthesis of *O*- and *C*-derivatives with high yields by the interaction of triethyliodosilane with bis(carbomethoxymethyl)mercury. By adjusting the conditions of the reaction it is possible to obtain either of the derivatives in a pure state. The best results for the synthesis of *O*-derivatives were obtained by using alkyl (trialkylstannyl)acetates

$$(C_2H_5)_3SnCH_2COOCH_3 + (C_2H_5)_3SiI \longrightarrow CH_2=C(OCH_3)(OSi(C_2H_5)_3) + (C_2H_5)_3SnI$$

In this way, *O*-(triethylsilyl)-*O*-methylketeneacetal has been isolated with a yield of 70 %; b.p. 63–65°/7 mm; n_D^{20} 1.4360; d_4^{20} 0.8918; MR_D 55.20, calcd. 55.56. The literature values[2] are: b.p. 65–66.5°/7 mm; n_D^{20} 1.4355; d_4^{20} 0.8870.

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Received November 16th, 1964