LETTER

Diallylsilanediol, $(CH_2\text{-}CH\text{-}CH_2)_2Si(OH)_2$

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Organosilanediol is an interesting compound in the study of silicones. While saturated silanediols have been described in various papers, unsaturated silanediols have not been previously reported. Recently Frish, Goodwin and Scott[1] prepared a series of organosilanediols which have one unsaturated and one saturated hydrocarbon radical on the same silicon atom. But the one which has two unsaturated hydrocarbon radicals on the same silicon atom has not been prepared. We wish now to report on diallylsilanediol which has been prepared directly from diallyldichlorosilane with a similar principle as that of Lucas and Martin.[2]

In a three-necked 2 l.-flask, an efficient stirrer and two dropping funnels were inserted in each neck. As a hydrolyzing mixture, saturated sodium chloride solution (270 g.) with an excess of solid sodium chloride and ethyl ether (400 cc.) were used. Redistilled (30 g.) diallyldichlorosilane (*Anal.* Cl: Calcd. 39.2,

(1) K. C. Frisch, P. A. Goodwin and R. E. Scott, *J. Am. Chem. Soc.*, **74**, 4584 (1952).

(2) G. R. Lucas and R. W. Martin, *J. Am. Chem. Soc.*, **74**, 5225 (1952).

Found 39.3) prepared by the direct method was diluted with ethyl ether (200 cc.) and 1 N-sodium hydroxide solution were placed in each dropping funnel. By using methyl orange as indicator the two solutions were added differentially to the hydrolyzing mixture with vigorous stirring. During the course of addition the temperature of the mixture was kept below 4°C. and the color change of methyl orange was carefully controlled between slight red and orange, because *p*H of the mixture has a great influence on the yield. Sodium chloride was added at a time when the solid disappeared from the hydrolyzing mixture.

After the addition, the ether layer was separated and completely dehydrated on sodium sulfate. Then ether was distilled off on a water-bath below 40°C., or better aspirated off. The product, oily matter and crystalline solid were washed with a little quantity of petroleum ether (yield 21 g., 87% of the theory). A sample for analysis was recrystallized from ethyl ether, m. p. 81°C., d^{10} 1.105. *Anal.* Calcd. for $C_6H_{12}O_2Si$ (144.2): C, 50.09; H, 8.57; Si, 19.38; hydroxyl, 23.9%. Found: C, 49.96; H, 8.39; Si, 19.44; mol. wt. 147 (in dioxane), 144.9 (from X-ray analysis); hydroxyl (Karl Fischer) 22.5%. Iodine value I_0: Calcd. 352, Found 355.*

Details on the preparation and properties of this compound will be reported later.

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* The high iodine values (I_e) proportional to the excessa mount (e) of Wijs reagent were observed. The value I_0 was obtained from the several I_e values by extrapolating graphically when $e \to 0$.