[CONTRIBUTION FROM THE CHEMICAL LABORATORY, IOWA STATE UNIVERSITY]

Novel Reduction of Ketones by Diphenylsilane

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Received May 17, 1961

The reduction of certain diaryl-type ketones to the corresponding hydrocarbon derivatives using diphenylsilane at relatively high temperatures is reported. A possible intermediate in the reduction route of benzophenone was established as benzhydryloxydiphenylsilane. The scope and limitations of the use of silicon hydrides as reducing agents are discussed.

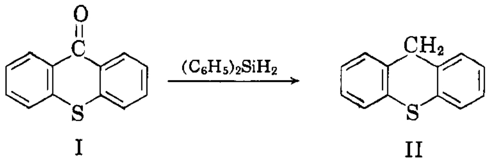
In a preliminary communication,[1] we reported that it was possible to reduce certain ketones to their corresponding hydrocarbons using diphenylsilane at relatively high temperatures. We are now presenting details, scope, and limitations of the reaction.

Although the reducing properties of the Si-H system was initially recognized nearly seventy-five years ago, only limited use has been made with this information. The first observation of the reducing abilities of the silicon hydrides was that of Buchner,[2] who reported that silane (SiH_4) was capable of reducing an aqueous solution of silver nitrate to metallic silver. Other investigators studying the preparation and properties of silane and silicochloroform (trichlorosilane) also noted that the Si-H system was a possible reducing agent.[3-6]

More recently, triarylsilanes[7] and trialkylsilanes[8-10] have been recognized as having reducing properties. Anderson studied the reducing power of trialkylhydrides of the Group IV B elements. In a series of papers he reported the use of halides of certain transition elements and elements in regular groups for the replacement of hydrogen attached to germanium,[11] to tin,[12] and to silicon.[13] For example, triethylsilane reduced certain halides or sulfates of at least ten transition elements and five elements in regular groups, either to a lower oxidation state or to the free element.[13] Other correlations concerning the relative reactivities of these hydrides have been reported.[14]

Triphenylsilane was found to add to the olefinic linkage in the presence of benzoyl peroxide to give fair yields of alkyltriphenylsilanes.[15] Gadsby[16] synthesized several carboxyalkylsilanes by the addition of a trialkylsilane to double bonds using benzoyl peroxide as a catalyst. A comparison of the behavior of triphenylsilane, triphenylgermane, and triphenyltin hydride in the presence of olefins has been reported.[17] All the hydrides added to the double bond when peroxide initiation was employed; however, triphenylsilane failed to react with olefins under ultraviolet irradiation, indicating that triphenylsilane apparently is not so reactive as the other hydrides.[17] Nitzsche and Wick[18] found that 1,3-dimethyldisiloxane, in the presence of 2–5% dibutyltin dilaurate, was an active reducing agent even at room temperature. It reduced carbonyl compounds to alcohols and nitro compounds to amines; however, olefinic double bonds were not attacked. This reaction generally had a brief induction period which could be shortened by the addition of trace amounts of benzoyl peroxide.

In the course of investigating the preparation of new types of organosilicon compounds, we had occasion to treat diphenylsilane with 10-thiaxanthenone. The reaction of diphenylsilane with sulfur-containing heterocycles is a recognized method for preparing cyclic-silicon compounds in which the sulfur atom is replaced by the diphenylsilylene grouping.[19,20] The interaction of diphenylsilane with 10-thiaxanthenone (I) at 230° for two hours gave a 92.4% recovery of starting materials; however, at reflux temperature for twenty hours a 38.4% yield of the reduced product, thiaxanthene (II), was realized:



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[1] J. W. Diehl and H. Gilman, *Chem. & Ind.* (*London*), 1095 (1959).

[2] G. Buchner, *Ber.*, **18**, 317R (1885).

[3] O. Ruff and C. Albert, *Ber.*, **38**, 53 (1905).

[4] O. Ruff and C. Albert, *Ber.*, **38**, 2222 (1905).

[5] A. Besson and L. Fournier, *Compt. rend.*, **148**, 1192 (1909).

[6] A. Stock and C. Somieski, *Ber.*, **54**, 524 (1921).

[7] J. W. Jenkins and H. W. Post, *J. Org. Chem.*, **15**, 556 (1950).

[8] F. P. Price, *J. Am. Chem. Soc.*, **69**, 2600 (1947).

[9] C. Eaborn, *J. Chem. Soc.*, 3077 (1950).

[10] C. Eaborn, *J. Chem. Soc.*, 2517 (1955).

[11] H. H. Anderson, *J. Am. Chem. Soc.*, **79**, 326 (1957).

[12] H. H. Anderson, *J. Am. Chem. Soc.*, **79**, 4913 (1957).

[13] H. H. Anderson, *J. Am. Chem. Soc.*, **80**, 5083 (1958).

[14] H. H. Anderson and A. Hendifar, *J. Am. Chem. Soc.*, **81**, 1027 (1959).

[15] See C. Eaborn, *Organosilicon Compounds* for a splendid coverage of such addition reactions; H. Merten and H. Gilman, *J. Am. Chem. Soc.*, **76**, 5798 (1954).

[16] G. N. Gadsby, *Research* (*London*), **3**, 338 (1950).

[17] R. Fuchs and H. Gilman, *J. Org. Chem.*, **22**, 1009 (1957).

[18] R. Nitzsche and M. H. Wick, *Angew. Chem.*, **69**, 96 (1957).

[19] H. Gilman and D. Wittenberg, *J. Am. Chem. Soc.*, **79**, 6339 (1957).

[20] D. Wittenberg, H. A. McNinch, and H. Gilman, *J. Am. Chem. Soc.*, **80**, 5418 (1958).

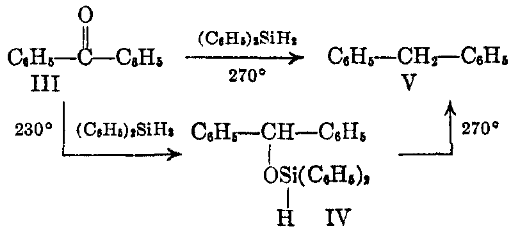
The reduction was carried out successfully with a number of diaryl ketones. In a typical experiment, equimolar quantities of 10-thiaxanthenone and diphenylsilane were heated, in the absence of any catalyst or activating agent, to reflux (temperature about 260°). Within a few minutes, an exothermic reaction took place with the temperature rising rapidly as high as 310°. After a short interval, the temperature spontaneously dropped to 260°, where it was maintained for twenty hours. There was no hydrogen sulfide given off during the reaction.[19,20] The cooled red reaction mixture was chromatographed on alumina and eluted with petroleum ether (b.p. 60–70°) to give a 38.4% yield of thiaxanthene. Several additional experiments were conducted at reflux for various lengths of time and it was observed that yields as high as 63.6% of reduced product could be realized if the reduction temperature were maintained for twelve hours. A reduction employing an inert atmosphere did not appear to increase the yield of reduced product. The ketones which were successfully reduced to the corresponding hydrocarbons utilizing an analogous procedure to that described above are listed in Table I.

TABLE I

KETONES REDUCED BY DIPHENYLSILANE

Ketone	Product	Yield, %
Anthraquinone	Anthracene	44.8
Benzophenone	Diphenylmethane	36.9
p-Chlorobenzophenone	(*p*-Chlorophenyl)phenylmethane	68.0
9-Fluorenone	Fluorene	37.3
4-Methylbenzophenone	Phenyl-*p*-tolylmethane	38.5
10-Thiaxanthenone	Thiaxanthene	63.6
9-Xanthenone	Xanthene	27.5

Phenyl-, diphenyl-, and triphenylsilane have been reported to add to benzophenone at temperatures around 230° to form addition products in which the silicon atom is bonded to the oxygen atom.[21] Silicochloroform and triphenylsilane reacted with acetone and pentanone-3, each catalyzed by ultraviolet radiation, to give the "abnormal" addition products.[22] However, under these conditions acetophenone and benzophenone failed to react.[22] Diphenylsilane adds to benzophenone (III) at 220–230° to form benzhydryloxydiphenylsilane (IV).[21] Since diphenylsilane reduced benzophenone to diphenylmethane (V) in a 36.9% yield at reflux temperature, it was believed that benzhydryloxydiphenylsilane (IV) might be an intermediate in this reduction route. Heating benzhydryloxydiphenylsilane at 270° for twelve hours afforded a 44.2% yield of diphenylmethane (V).



A small quantity (5%) of benzhydryl ether and resinous oils was also obtained by the heating of benzhydryloxydiphenylsilane. Attempted reduction of benzhydrol with diphenylsilane resulted in a high recovery of diphenylsilane and a 60% yield of benzhydryl ether.

Anthraquinone afforded a 45% yield of anthracene under the general conditions of the reduction. Phenanthraquinone underwent a vigorous reaction with diphenylsilane but phenanthrene could not be isolated.

The reaction of triphenylsilane with benzophenone at reflux gave the same product as that obtained at the lower temperatures, namely, benzhydryloxytriphenylsilane. Diphenylmethane was not isolated from this reaction. Triphenylsilane reacted with anthraquinone at reflux to give a 25% yield of a compound which is thought to be 10-triphenylsilyloxyanthrone. The analysis and infrared spectrum of this product indicate that triphenylsilane added "abnormally" across only one carbonyl grouping. Probably because the reflux temperature of phenylsilane is low, in the neighborhood of 120°, there was no evidence of reduction in the reaction of phenylsilane with 10-thiaxanthenone; a 95.4% recovery of starting materials was realized.

The fate of the silicon moiety has not been definitely established; however, it is thought that the resin-like material obtained in each reaction is a mixture of cyclosiloxanes. These resins show the characteristic Si—O—Si absorption band. Some (7%) hexaphenylcyclotrisiloxane was isolated along with a 68% yield of (*p*-chlorophenyl)phenylmethane from the reaction of diphenylsilane with 4-chlorobenzophenone. In a related reduction, a trace of hexaphenylcyclotrisiloxane was isolated in addition to a 76% yield of 10-ethylphenothiazine from the reaction of diphenylsilane with 10-ethylphenothiazine 5-oxide at temperatures of 200–250°.[20]

There was no reduction product isolated from the interaction of diphenylsilane with quinoline, phenanthridine, or acetophenone, and only starting materials were recovered.

Kuivila and Beumel[23] in attempting the addition of diphenyltin dihydride to methyl vinyl ketone, isolated methylvinylcarbinol in a good yield. These authors[23] believed that, since an hydrolysis step was not necessary, there was a transfer of two hydrogens in a noncatalyzed manner from the tin to the carbonyl grouping. They then found

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[21] H. Gilman and D. Wittenberg, *J. Org. Chem.*, **23**, 501 (1958).

[22] R. Calas and N. Duffaut, *Compt. rend.*, **245**, 906 (1957).

[23] H. G. Kuivila and O. F. Beumel, *J. Am. Chem. Soc.*, **83**, 1246 (1961).

that it was possible selectively to reduce several α,β-unsaturated aldehydes and ketones to the corresponding alcohols using diphenyltin dihydride. Organotin hydrides, however, are capable of undergoing a noncatalyzed addition to olefinic double bonds at moderate temperatures.[24] Two recent communications[25,26] have indicated that triphenyltin hydride offers promise as a reducing agent. Refluxing triphenyltin hydride with bromobenzene gave 60–75% yields of the hydrogenolysis product, benzene.[25] Triphenyltin hydride also reduced carbonyl groups to the corresponding alcohols.[26] We have found that when triphenylsilane was refluxed with bromobenzene only starting materials and a trace of hexaphenyldisiloxane are obtained. This result is in keeping with other correlations on the relative reactivities of triarylhydrides of the Group IV B elements.[17]

EXPERIMENTAL[27]

Reaction of diphenylsilane with 10-thiaxanthenone. (a) *At 230°.* Nine and two-tenths grams (0.05 mole) of diphenylsilane[28] and 10.6 g. (0.05 mole) of 10-thiaxanthenone were heated to 220–230° over a 1-hr. period and held at this temperature for 2 hr. Upon cooling to room temperature, a yellow solid crystallized which was filtered to give 9.8 g. (92.4%) of 10-thiaxanthenone, m.p. 212–214°, identified by a mixed melting point with an authentic sample.

(b) *At reflux.* Twenty-one and three-tenths grams (0.1 mole) of 10-thiaxanthenone and 18.4 g. (0.1 mole) of diphenylsilane were heated to reflux over a 1-hr. period. Boiling started at 260°, then the temperature rose rapidly to about 310°. The yellow color changed to red and some white solid sublimed. The temperature slowly dropped to 260° where it was maintained for 20 hr. Upon cooling to room temperature, a red solid formed which was dissolved in hot petroleum ether (b.p. 60–70°). Chromatography of the petroleum ether (b.p. 60–70°) solution on alumina gave a pink-red solid, m.p. 124–128°. Recrystallization from ethanol (Norit) gave 7.6 g. (38.4%) of thiaxanthene, m.p. 128–130°, identified by a mixed melting point and a comparison of the infrared spectrum with an authentic specimen. No other solid was isolated from the column by further elution with various solvents.

(c) *At reflux for various lengths of time*

Reaction of diphenylsilane with benzophenone. (a) *At 220–230°.* Nine and two-tenths grams (0.05 mole) of diphenylsilane and 9.1 g. (0.05 mole) of benzophenone were heated for 2 hr. at 220–230°. Distillation gave an 86% recovery of starting materials.

(b) *At 230–235°.* This experiment was a repetition of the experiment described above except that the temperature was held at 230–235° for 4 hr. Distillation gave a 5.5% recovery of starting materials, identified by their infrared spectra. The distillation residue was taken up in petroleum ether, filtered, and concentrated to give 13.8 g. (75.4%) of benzhydryloxydiphenylsilane, m.p. 78–80°. Recrystallization from ethanol raised the m.p. to 80–81.5°. A mixed melting point with an authentic sample[21] showed no depression.

(c) *At reflux.* This experiment was a repeat of part (a) except that the temperature was permitted to reach reflux. At approximately 265°, the temperature rose quickly to 340° then reverted to 265° where it was held for 12 hr. Distillation of the viscous oil gave 3.1 g. (36.9%) of diphenylmethane, b.p. 68–70° (0.025 mm.), n_D^{18} 1.5650, m.p. 23.5–25°. An infrared spectrum of this material was identical with that of an authentic specimen (n_D^{16} 1.5696, m.p. 24–26°). The tetranitro derivative melted at 173–174°, lit.[30] m.p. 172°. Chromatography of the waxy distillation residue gave only oils, an infrared spectra of which showed the silicon-oxygen absorption band.

Time	Yield of Thiaxanthene, %
3 Days	35.4
20 Hr.	38.4
12 Hr.	63.6[a,b]
4 Hr.	37.4[a]

[a] Approximately 2.0 g. of pink solid melting over the range 315–320° was isolated in these experiments. This compound was not soluble in the common solvents and did not contain silicon. The infrared spectrum was inconclusive; however, the solid might possibly be dithioxanthyl, lit.[29] m.p. 325°.
[b] An experiment using a nitrogen atmosphere gave a 61% yield of the reduced product.

Heating of benzhydryloxydiphenylsilane. Twelve and eight-tenths grams (0.035 mole) of benzhydryloxydiphenylsilane was heated, without added solvent or catalyst, at 270° for 12 hr. Distillation gave 2.6 g. (44.2%) of diphenylmethane, b.p. 93–95° (2 mm.), $n_D^{17.5}$ 1.5793, m.p. 25–26°. The infrared spectrum was superimposable with that of an authentic sample. Chromatography of the distillation residue on alumina gave 0.3 g. (4.9%) of benzhydryl ether, m.p. 108–109.5°. Further elution failed to give any crystalline materials. The oils indicated silicon-phenyl and silicon-oxygen absorption bands in the infrared.

Reaction of diphenylsilane. (a) *With 9-fluorenone.* Nine and two-tenths grams (0.05 mole) of diphenylsilane and 9.0 g. (0.05 mole) of 9-fluorenone were heated to reflux over a 1-hr period. At 270°, a violent exothermic reaction took place and the temperature rose rapidly to 330°. The color changed from yellow to red and a white solid sublimed. The temperature declined to 270° where it was held for 12 hr. The brown cake was dissolved in petroleum ether (b.p. 60–70°) and chromatographed on alumina, giving 5.0 g. (60.3%) of a white solid, m.p. 101–108°. Two recrystallizations from ethanol gave 3.1 g. (37.3%) of fluorene, m.p. 112–115°; a mixed melting point was undepressed. Further elution of the column gave only oils which could not be crystallized.

(*b*) *With 9-xanthenone.* Nine and two-tenths grams (0.05 mole) of diphenylsilane and 9.8 g. (0.05 mole) of 9-xanthenone were heated to reflux over a 1-hr. period. Boiling started at 260°; then the temperature rose rapidly to 320°. It slowly dropped back to 260° where it was held for 12 hr. Dissolution of the brown cake in petroleum ether (b.p. 60–70°) and chromatography on alumina gave 2.5 g. (27.5%) of xanthene, m.p. 101–103° (mixed melting point). Further elution of the column failed to give any crystalline materials.

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(24) G. J. M. van der Kerk, J. G. Noltes, and J. G. A. Luijten, *J. Appl. Chem. (London)*, **7**, 356 (1957).

(25) L. A. Rothman and E. I. Becker, *J. Org. Chem.*, **25**, 2203 (1960).

(26) J. G. Noltes and G. J. M. van der Kerk, *Chem. & Ind. (London)*, 294 (1959).

(27) All melting and boiling points are uncorrected.

(28) The diphenylsilane was prepared by the reduction of diphenyldichlorosilane (Dow Corning) with lithium aluminum hydride according to the procedure of R. A. Benkeser, H. Landesman, and D. J. Foster, *J. Am. Chem. Soc.*, **74**, 648 (1952) except that the solvent employed was tetrahydrofuran instead of ether. With the application of this slightly modified procedure the yield of product was raised from 76% reported earlier to 89% based on the chlorosilane, b.p. 65–66° (0.05 mm.), n_D^{20} 1.5783.

(29) A. Schönberg and A. Mustafa, *J. Chem. Soc.*, 657 (1945).

(30) R. L. Shriner and R. C. Fuson, *The Systematic Identification of Organic Compounds*, 3rd ed., J. Wiley and Sons, Inc., New York, 1948, p. 259.

(c) *With anthraquinone.* Ten and four-tenths grams (0.05 mole) of anthraquinone and 18.4 g. (0.1 mole) of diphenylsilane were heated to reflux (about 275°). The temperature rose rapidly to a maximum of 360° then slowly dropped to 275° and was maintained there for 12 hr. The greenish brown reaction mass was dissolved in benzene, filtered, and concentrated to give a bright yellow solid, m.p. 205–216°. Two recrystallizations from ethanol gave 4.0 g. (44.8%) of anthracene, m.p. 216–217.5°. A mixed melting point with an authentic sample was not depressed.

(d) *With phenanthraquinone.* Ten and four-tenths grams (0.05 mole) of phenanthraquinone and 18.4 g. (0.1 mole) of diphenylsilane were heated to reflux (about 270°) and maintained for 12 hr. There was no exothermic reaction, and the reaction mass gradually assumed a deep red appearance. Distillation gave a 5% recovery of diphenylsilane. Chromatography of the distillation residue on alumina with benzene afforded a 31.8% recovery of phenanthraquinone, m.p. 204–207°. Further elution gave only tars and gums which resisted crystallization.

(e) *With benzhydrol.* Nine and two-tenths grams (0.05 mole) of benzhydrol and 9.2 g. (0.05 mole) of diphenylsilane were heated to reflux. The temperature was about 170°, where it was held for 12 hr. The viscous clear oil was distilled to give 9.0 g. (97.8%) of diphenylsilane, b.p. 104–105° (3.5 mm.) $n^{18.5}_D$ 1.5783. The infrared spectrum showed the presence of silicon-hydrogen absorption band. The distillation residue gave a 60% yield of benzhydryl ether which after recrystallization from ethanol melted at 109–111° (mixed melting point).

(f) *With acetophenone.* Six grams (0.05 mole) of acetophenone and 9.2 g. (0.05 mole) of diphenylsilane were heated to reflux (about 220°) and held at this temperature for 12 hr. Distillation of the viscous oil gave a 40% recovery of acetophenone, b.p. 60–62° (0.5 mm.), n^{20}_D 1.5349; and a 70% yield of recovered diphenylsilane, b.p. 78–80° (0.5 mm.), n^{20}_D 1.5841. The infrared spectrum of each material was identical with that of an authentic sample.

(g) *With 4-chlorobenzophenone.* Nine and two-tenths grams (0.05 mole) of diphenylsilane and 10.8 g. (0.05 mole) of 4-chlorobenzophenone were heated to reflux (about 270°). An exothermic reaction took place with the temperature reaching a maximum of 330° before it slowly dropped to 270°.

Distillation gave 6.9 g. (68%) of colorless 4-chlorodiphenylmethane, b.p. 122–123.5° (2.2 mm.), n^{20}_D 1.5854, lit.[31] b.p. 298° (742.5 mm.). The infrared spectrum indicated the absence of a carbonyl absorption band and the spectrum was, in general, quite similar to that of diphenylmethane. Chromatography of the distillation residue on alumina and elution with benzene gave 0.7 g. (7.1%) of crude white solid, m.p. 170–175°. Two recrystallizations from a mixture of benzene and petroleum ether (b.p. 60–70°) gave 0.5 g. (5.2%) of hexaphenylcyclotrisiloxane, m.p. 187–189°. A mixture melting point with an authentic sample[20] was not depressed.

(h) *With 4-methylbenzophenone.* Nine and two-tenths grams (0.05 mole) of diphenylsilane and 9.8 g. (0.05 mole) of 4-methylbenzophenone were heated to reflux (about 275°) over a 1-hr. period. The temperature rose rapidly to 330°, and the colorless oil became yellow. The temperature slowly dropped to 275° where it was maintained for 12 hr. Distillation afforded 3.5 g. (38.5%) of phenyl-*p*-tolylmethane, b.p. 87–89° (0.35 mm.), n^{20}_D 1.5683. The infrared spectrum of the liquid was superimposable with that of an authentic sample (n^{20}_D 1.5682). Chromatography of the distillation residue failed to yield any crystalline products.

(i) *With quinoline.* Nine and two-tenths grams (0.05 mole) of diphenylsilane was mixed with 6.5 g. (0.05 mole) of freshly distilled quinoline in a nitrogen atmosphere. The clear reaction mass was heated to reflux, about 260°, and held at this temperature for 12 hr. There was no sudden exothermic reaction. Distillation of the light yellow oil gave 4.9 g. (75.4%) of recovered quinoline, b.p. 60° (0.01 mm.), n^{20}_D 1.6144, and 8.3 g. (90%) of recovered diphenylsilane, b.p. 68° (0.01 mm.), n^{20}_D 1.5827. The infrared spectrum of each material was identical with that of an authentic sample.

(j) *With phenanthridine.* Nine and two-tenths grams (0.05 mole) of diphenylsilane and 8.95 g. (0.05 mole) of phenanthridine were heated to reflux (about 250°) and held at this temperature for 12 hr. There was no exothermic reaction noted. Distillation of the viscous, yellow oil gave a 20% recovery of diphenylsilane and a 31.3% recovery of phenanthridine, b.p. 130–135° (0.01 mm.), m.p. 106–108°. Chromatography of the waxy, brown distillation residue on alumina gave oil which could not be crystallized or further purified.

Reaction of phenylsilane with 10-thiaxanthenone. Phenylsilane, 5.4 g. (0.05 mole) and 10.6 g. (0.05 mole) of 10-thiaxanthenone were heated slowly to reflux temperature for 8 hr. (about 120°). The reaction mass was then permitted to cool to room temperature. Petroleum ether (b.p. 60–70°) was added and the resulting slurry filtered to give 10.1 g. (95.4%) of recovered 10-thiaxanthenone, m.p. 213–215° (mixed melting point).

Reaction of triphenylsilane. (a) *With benzophenone.* Thirteen grams (0.05 mole) of triphenylsilane[32] and 9.1 g. (0.05 mole) of benzophenone were heated to reflux (about 280°) for 12 hr. Dissolution of the reaction mass in petroleum ether (b.p. 60–70°), filtering, and concentrating gave 10.5 g. (47.6%) of benzhydryloxytriphenylsilane, m.p. 81–83°. A mixed melting point with an authentic sample[21] was undepressed.

(b) *With bromobenzene.* Fifty grams (0.318 mole) of bromobenzene was added to 91 g. (0.350 mole) of triphenylsilane and the reaction mass heated to reflux for 15 hr. There was no color change during the reaction. Distillation gave a 79% recovery of bromobenzene, n^{17}_D 1.5613. Chromatography of the distillation residue afforded an 85% recovery of triphenylsilane, m.p. 46–48°, and a trace of hexaphenyldisiloxane, m.p. 225–228°. There was no triphenylbromosilane isolated, nor any benzene, the expected hydrogenolysis product of bromobenzene.

(c) *With anthraquinone.* Thirteen grams (0.05 mole) of triphenylsilane and 5.2 g. (0.025 mole) of anthraquinone were heated to reflux (about 235–240°) and held there for 12 hr. Upon cooling to room temperature, a yellow gum remained. Several recrystallizations from ethanol gave 3.0 g. (25.6%) of a pale yellow solid, m.p. 120–122°. An infrared spectrum of this compound showed the silicon-phenyl, silicon-oxygen, and carbonyl bands.

Anal. Calcd. for $C_{32}H_{24}O_2Si$: Si, 5.98. Found: Si, 5.82, 6.02. This compound may be 10-triphenylsilyloxyanthrone.

Acknowledgment. Infrared analyses were obtained through the courtesy of the Institute for Atomic Research, Iowa State University, and special acknowledgment is made to Dr. V. A. Fassel and Mr. R. Kniseley for the spectra.

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(31) P. J. Montagne, *Rec. trav. chim.*, **26**, 263 (1907).

(32) H. Gilman and H. W. Melvin, Jr., *J. Am. Chem. Soc.*, **71**, 4050 (1949).