Hydrogen value: Calcd. for three Si—Si bonds per $C_{48}H_{40}Si_4O$: 90.4. Found: 94.7, 92 (ml./g.).

Hydrolysis of 1,4-dichlorooctaphenyltetrasilane (I). The same aqueous alcoholic aniline mixture described in the previous section was used to hydrolyze 0.55 g. (0.7 mmole) of 1,4-dichlorooctaphenyltetrasilane.[8-10] In this case refluxing was continued for 20 hours. After cooling, filtration gave 0.3 g. (58.3%) of a compound, m.p. 220–228°. Recrystallization from benzene–petroleum ether (b.p. 60–70°) raised the melting point to 226–228°. A mixed melting point with the product from the hydrolysis of the diiodo compound was not depressed and the infrared spectra were superimposable.

Hydrolysis of 1,4-dichlorooctaphenyltetrasilane (II). To a mixture of 50 ml. of *tert*-amyl alcohol, 40 ml. of toluene, and 140 ml. of water was added 8 g. (0.01 mole) of 1,4-dichlorooctaphenyltetrasilane. The mixture was stirred for 24 hours at room temperature. The layers were separated and the insoluble material was filtered off to yield 2.5 g. (33.6%), m.p. 215–220°. Several recrystallizations from benzene–petroleum ether (b.p. 60–70°) raised the melting point to 226–228°. The mixture melting point and the infrared spectrum identified this compound as the "monoxide."

Chromatography of 1,4-dichlorooctaphenyltetrasilane. Two grams of 1,4-dichlorooctaphenyltetrasilane were dissolved in benzene and chromatographed through an alumina column. Elution with petroleum ether (b.p. 60–70°) gave 0.9 g. (50%) of a product, m.p. 234–236°. This compound had an infrared spectrum identical with those of the above isolated samples.

Formation of 1,6-dihydroxydodecaphenylhexasilane. To a mixture of 25 ml. of *tert*-amyl alcohol, 20 ml. of toluene, and 70 ml. of water was added 4 g. (0.003 mole) of 1,6-dibromododecaphenylhexasilane.[15] The reaction mixture was stirred for 24 hours at room temperature. The layers were separated and the aqueous layer was extracted with ether. The organic extracts were combined and dried. Evaporation of the solvents gave 3.5 g. of a product melting over a range (100–130°). After several recrystallizations from benzene-petroleum ether (b.p. 60–70°) there was obtained 2 g. (56%) of a compound melting at 170–172°. This material showed strong Si—O—H bonding in the infrared. An identical product was obtained when 1,6-dibromododecaphenylhexasilane was hydrolyzed with 0.1*N* hydrochloric acid.

Anal. Calcd. for $C_{72}H_{62}Si_6O_2$: C, 76.80; H, 5.51; Si, 14.93. Found: C, 77.00, 77.03; H, 5.72, 5.63; Si, 14.86, 14.74. Hydrogen value: Calcd. for five Si—Si bonds per $C_{72}H_{62}Si_6O_2$: 99.7. Found: 103, 99.8.

Preparation of 2,2,3,3,5,5,6,6-octaphenyl-1,4-dioxa-2,3,5,6-tetrasilacyclohexane. A suspension of 2 g. of *sym*-tetraphenyldisilanediol[6] in 30 ml. of 88% formic acid was heated 30 min. to boiling. The solution was cooled and poured into 100 ml. of ice water. The white crystalline compound which was collected melted at 219–220° after one recrystallization from benzene–petroleum ether (b.p. 60–70°). The pure siloxane weighed 1.8 g. (95%).

Anal. Calcd. for $C_{48}H_{40}Si_4O_2$: C, 75.75; H, 5.30; Si, 14.75; O, 4.21. Found: C, 75.55, 75.75; H, 5.30, 5.36; Si, 14.60, 14.81. Hydrogen value: Calcd. for two Si—Si bonds per $C_{48}H_{40}Si_4O_2$: 59. Found: 59.5.

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[CONTRIBUTION FROM THE DEVELOPMENT DEPARTMENT, UNION CARBIDE CHEMICALS CO., DIVISION OF UNION CARBIDE CORP.]

Synthesis of 1,1'-Bis(tri-*n*-dodecylsilyl)ferrocene

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The previously unreported 1,1'-bis(tri-*n*-dodecylsilyl)ferrocene has been synthesized in a yield of about 54% from chlorotri-*n*-dodecylsilane via the intermediate tri-*n*-dodecylsilylcyclopentadiene. The procedure utilizes modifications of previously developed synthetic methods and should be applicable to the preparation of other disubstituted silylferrocenes. Some aspects of the preparation of the intermediate compounds and the ferrocene derivative are discussed.

During an investigation of potential high temperature lubricants and hydraulic fluids in this laboratory, the need arose for fairly large quantities of 1,1'-bis(tri-*n*-dodecylsilyl)ferrocene. At the time of our study, only two heteroannularly disubstituted trialkylsilylferrocenes had been reported in the literature, namely 1,1'-bis(trimethylsilyl)ferrocene[1] and 1,1'-bis(tri-*n*-hexylsilyl)ferrocene.[2] Each of these compounds had been prepared by two possible routes: (A) the metalation of ferrocene followed by reaction with the appropriate bromotrialkylsilane,[1,2] and (B) *via* coupling of the appropriate trialkylsilylcyclopentadiene with iron(II) chloride.[2] The former method also was used to prepare tri-*n*-dodecylsilylferrocene; however, in this instance isolation of 1,1'-bis(tri-*n*-dodecylsilyl)ferrocene from the resulting reaction mixture was not attempted.[2a]

In studies elsewhere the reaction of lithiated ferrocene with bromotrialkylsilanes led to low yields of a mixture of the mono- and disubstituted silylferrocenes contaminated with substantial amounts of the corresponding *n*-butyltrialkylsilanes (from the reaction of the bromosilanes with *n*-butyllithium used in the metalation reaction).[2a] The 1,1'-bis(trimethylsilyl)ferrocene was isolated in a yield of 27%,[1] while the yield of the *n*-hexyl derivative was 35%.[2b] The latter compound was

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(1) M. Rausch, M. Vogel, and H. Rosenberg, Wright Air Development Center Technical Report 57-62, Part I (1958).

(2) (a) M. Rausch, M. Vogel, H. Rosenberg, D. Mayo, and P. Shaw, Wright Air Development Center Technical Report 57-62, Part II (1958). (b) S. I. Goldberg, D. W. Mayo, M. Vogel, H. Rosenberg, and M. Rausch, *J. Org. Chem.*, 24, 824 (1959).

purified via chromatography on alumina. Coupling of trimethyl- and tri-n-hexylsilylcyclopentadiene with iron(II) chloride led to yields of the disubstituted ferrocenes of 16.5[2a,3] and 4%,[2b] respectively, based on the bromotrialkylsilanes. Again, it was necessary to chromatograph the reaction mixture on alumina in order to isolate the hexyl derivative.

Because of the variety of products obtained and the resulting complicated separation problems encountered when a halotrialkylsilane is treated with a metalated ferrocene, route B was chosen in our study as a means of preparing the desired 1,1′-bis(tridodecylsilyl)ferrocene. The sequence of reactions illustrated in equations I, II, and III was used to prepare this compound in a 54% yield, based on the chlorotrialkylsilane ($R = n\text{-}C_{12}H_{25}$—).[4]

$$R_3SiCl + C_5H_5Na \longrightarrow C_5H_5SiR_3 + NaCl \quad (I)$$

$$2C_5H_5SiR_3 + 2Na \longrightarrow 2NaC_5H_4SiR_3 + H_2 \quad (II)$$

$$2NaC_5H_4SiR_3 + FeCl_2 \longrightarrow Fe(C_5H_4SiR_3)_2 + 2NaCl \quad (III)$$

Attempts to isolate the trialkylsilylcyclopentadiene by distillation led to very low yields of liquid boiling over a wide range. The infrared spectrum of this material revealed Si—H bonds (absorption near 4.75 μ), and the elemental analyses did not agree with the values calculated for the silylcyclopentadiene. As a result, the substituted cyclopentadiene was not isolated, and reaction II was carried out on the crude mixtures recovered from step I.

In step I, an excess of cyclopentadienylsodium was treated with chlorotri-n-dodecylsilane and the excess of alkylsodium compound was destroyed by washing the reaction mixture with cold, 5% aqueous hydrochloric acid at temperatures ranging from 0 to 5°.

An excess of cyclopentadienylsodium was used in the first reaction in order to minimize the amount of unchanged chlorosilane present during the washing step. The chlorosilane hydrolyzes to high boiling siloxanes in the presence of water. The washing step not only removes the excess of cyclopentadienylsodium, which would lead to ferrocene and a mono-substituted ferrocene if left in the mixture, but also allows complete removal of the salts formed in step I without having to carry out a difficult filtration. In several exploratory runs in which equimolar amounts of cyclopentadienylsodium and the halosilane were used and the washing step was omitted, very low yields of the disubstituted ferrocene were obtained and the isolation was complicated by the presence of ferrocene and several other unidentified high boiling impurities.

The temperature was kept low during the washing step to minimize any attack of acid or water on the cyclopentadienylsilicon linkage.[5] Similarly, the metalation of the silylcyclopentadiene, Step II, was performed at low temperatures (0 to 5°) to prevent cleavage of the cyclopentadienyl ring from the silicon by the sodium.

The sandwich reaction, Step III, was performed in tetrahydrofuran using the method of Wilkinson.[6] Although this reaction was straightforward, a 40% molar excess of iron powder was used during the preparation of iron(II) chloride to assure complete reduction of the iron(III) chloride. Upon completion of the coupling reaction, replacement of tetrahydrofuran with petroleum ether was required to precipitate the co-product salts. These salts were partially soluble in the original solvent, and, if left in, considerably complicated the isolation of the silylferrocene.

Preparation of chlorotri-n-dodecylsilane was necessary since this compound was not readily available at the time of our study.[7] Several preparations of this compound were attempted using reaction IV.

$$n\text{-}C_{12}H_{25}SiCl_3 + 2n\text{-}C_{12}H_{25}MgBr \longrightarrow (n\text{-}C_{12}H_{25})_3SiCl + \text{Mg salts} \quad (IV)$$

Upon distillation of the products from reaction IV, some decomposition into lower boiling materials occurred. The infrared spectra of these materials indicated the presence of n-dodecene-1 (spectrum of one fraction was nearly identical with a spectrum of authentic n-dodecene-1), while absorption near 4.8 and 9.3 μ in the other fractions revealed the presence of Si—H and Si—O—Si bonds.[8] These findings are in agreement with the observations of Gilman and co-workers,[9] who isolated n-hexadecene-1 and a compound containing an Si—H bond during an attempt to prepare chlorotri-n-hexadecylsilane from trichloro-n-hexadecylsilane and n-hexadecylmagnesium bromide.

Subsequently, the desired chlorotri-n-dodecylsilane was prepared by the method recommended by Gilman and co-workers[9] and illustrated in equations V and VI ($R = n\text{-}C_{12}H_{25}$).

$$3RMgBr + HSiCl_3 \longrightarrow HSiR_3 + MgClBr \quad (V)$$

$$HSiR_3 + Cl_2 \longrightarrow ClSiR_3 + HCl \quad (VI)$$

Reaction V gave yields of tri-n-dodecylsilane ranging from 87.5 to 93%, while the chlorination,

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(3) The yield of the methyl derivative from trimethylsilylcyclopentadiene was 50%.

(4) Reaction I is a modification of the method of K. C. Frisch, *J. Am. Chem. Soc.*, **75**, 6050 (1953).

(5) See P. D. George, M. Prober, and J. R. Elliott, *Chem. Revs.*, **56**, 1087 (1956) for a discussion of the facile cleavage of allyl and indenyl groups from silicon by both electrophilic and nucleophilic reagents.

(6) G. Wilkinson, *Org. Syntheses*, **36**, 31 (1956).

(7) A small amount of technical grade chlorotri-n-dodecylsilane was obtained from Anderson Laboratories, Inc., Weston, Mich. This particular sample was not sufficiently pure for our purposes, and the material was not available in quantity.

(8) L. J. Bellamy, *The Infrared Spectra of Complex Molecules*, Wiley, New York, 1954, pp. 278–280.

(9) H. Gilman, D. H. Miles, L. O. Moore, and C. W. Gerow, *J. Org. Chem.*, **24**, 219 (1959).

performed in ethylene dichloride, led to yields of refined chlorotrialkylsilane from 89 to 94%, or overall yields based on trichlorosilane of 78.5 to 87.5%. For the best yields in step V, a molar ratio of Grignard reagent to trichlorosilane of 3.5 to 1 was used. When carbon tetrachloride was substituted for ethylene dichloride in step VI, the yield of chlorosilane dropped to around 83%.

The infrared spectrum of 1,1′-bis(tri-*n*-dodecylsilyl)ferrocene showed weak absorption in the region of 9 and 10 μ, apparently in contradiction to the 9–10 rule.[10] However, similar bands were present in the spectrum of the chlorotri-*n*-dodecylsilane, and the method of synthesis of the ferrocene derivative indicates that the bands near 9 and 10 μ are due to the substituent groups and not to any inconsistency of the 9–10 rule. Similar bands were noted in the spectrum of 1,1′-bis(tri-*n*-hexylsilyl)ferrocene.[2b]

EXPERIMENTAL[11]

Tri-n-dodecylsilane. In a 3-l. flask fitted with a Tru-Bore stirrer, a reflux condenser, a 125-ml. dropping funnel, and nitrogen inlet and outlet tubes was placed 2 moles of *n*-dodecylmagnesium bromide in 1150 ml. of ether. Trichlorosilane (74 g., 0.545 mole) was added dropwise over 1 hr. at ambient temperature. The resulting slurry was stirred overnight at room temperature, then hydrolyzed by adding a solution of 75 ml. of 37% hydrochloric acid diluted to 775 ml. with water. The resulting layers were separated, and the aqueous layer was extracted with two portions of ether. The combined extracts and organic layer were dried over anhydrous sodium sulfate, filtered, and the solvent was distilled *via* a 10-tray Oldershaw column. Distillation of the residual oil through a 30 × 200 mm. column packed with 1/8 in. burl saddles yielded 256 g. (87.5%) of tri-*n*-dodecylsilane, boiling at 220–223°/0.05 mm., n_D^{20} 1.4606, m.p. 16–16.5°.

Anal.[12] Calcd. for $C_{36}H_{76}Si$: C, 80.5; H, 14.3; Si, 5.2. Found: C, 79.6; H, 14.4; Si, 4.9.

Chlorotri-n-dodecylsilane. A. From tri-n-dodecylsilane. This preparation was carried out according to the procedure of Gilman and co-workers.[9] A solution of 306.5 g. (0.57 mole) of tri-*n*-dodecylsilane in 310 ml. of ethylene dichloride was allowed to react with 40 g. (0.565 mole) of chlorine gas at from 0 to 6°. The flow of chlorine was followed using a Rotameter, and after the specified amount had passed through the solution, nitrogen was passed through the reaction mixture until hydrogen chloride was no longer detectable in the off-gas. The solvent was distilled to leave 327 g. of a slightly yellow oil. Distillation of this oil gave 305 g. (0.534 mole, 93%) of product boiling at 228–238°/0.03 mm., n_D^{20} 1.4647, m.p. 4–5°.

Anal. Calcd. for $C_{36}H_{75}ClSi$: C, 75.7; H, 13.2; Cl, 6.2; Si, 4.9. Found: C, 75.5; H, 13.3; Cl, 6.73; Si, 4.85. An infrared spectrum indicated that no Si—H or Si—O—Si bonds were present in this material.

B. From trichloro-n-dodecylsilane. Several preparations were made using trichloro-*n*-dodecylsilane and *n*-dodecylmagnesium bromide. All of these reactions yielded mixtures of products and attempts to isolate chlorotri-*n*-dodecylsilane gave impure material. The best preparation is described.

n-Dodecylmagnesium bromide (2 moles) in 1050 ml. of ether was added to 320 g. (1.05 moles) of trichloro-*n*-dodecylsilane in 200 ml. of ether over a period of 1.5 hr. The mixture was heated to reflux during the addition and stirred at a temperature of 46–50° overnight. The reaction mixture was then cooled and centrifuged under nitrogen to remove the major portion of precipitated solids. The solids were washed with ether and the solvent was distilled to a flask temperature of 127°/2 mm., at which time additional solids precipitated. Attempted vacuum filtration through paper was very slow, hence the mixture was diluted with tetrahydrofuran and again centrifuged. Filtration of the centrifugate removed the remaining solids, and the solvents were distilled to a vapor temperature of 55°/2 mm. Further distillation gave 20.5 g. of oil boiling over the range 75–105°/0.7–1.3 mm. The infrared spectrum of this material indicated that it was an olefin, presumably 1-dodecene. At this point, more solids precipitated, and it was necessary to filter the concentrated liquid again. Continued distillation gave three fractions: 140 g., boiling at 195–202°/0.1 mm.; 32 g., boiling at 202–247°/0.1 mm., n_D^{20} 1.4615; and 99 g., boiling at 245–248°/0.1–0.2 mm. When the vapor temperature reached 248°, extensive decomposition occurred and the system pressure rose above 1 mm. Redistillation of the third fraction gave two fractions: 15 g., boiling at 196–242°/0.5–0.04 mm.; and 66 g., boiling at 236–242°/0.04 mm., n_D^{20} 1.4622, which was slightly impure product.

Anal. Calcd. for $C_{36}H_{75}ClSi$: C, 75.7; H, 13.2; Cl, 6.2; Si, 4.9. Found: C, 74.6; H, 13.1; Cl, 6.40; Si, 4.60.

A small amount of liquid which had an infrared spectrum almost identical with that of 1-dodecene was recovered from the traps of the second distillation. The infrared spectra of all fractions except the first 140 g. revealed the presence of Si—H and Si—O—Si components.

Tri-n-dodecylsilylcyclopentadiene (in situ). To a 12-l. flask fitted with a stirrer, a thermowell, an addition funnel and nitrogen inlet and outlet tubes was added 2,852 g. (5 moles) of chlorotri-*n*-dodecylsilane dissolved in 3400 ml. of ethyl ether. This solution was stirred and maintained at 0–5° while 5.5 moles of cyclopentadienylsodium[13] dissolved in tetrahydrofuran was added over a period of 3 hr. The resulting mixture was stirred an additional hour at 0–5° then overnight at ambient temperature. The solvent was removed at reduced pressure at a flask temperature of less than 20°, 5-l. of ether was added, and the suspension was cooled to −10°. A cold, 5% solution of aqueous hydrochloric acid was then added dropwise, with vigorous stirring, until the mixture was just acid to litmus (100 ml. required). Ice water (500 ml.) was then added to dissolve the remaining solids, and the mixture was transferred in portions to a 4-l. separatory funnel, where the layers were separated. The organic layer of each portion was washed with 200 ml. of ice water, and the combined organic layers were dried over anhydrous sodium sulfate. Ether was distilled at reduced pressure while maintaining the flask contents at a temperature below 20°. The resulting dark colored residual oil was utilized in the next reaction without further treatment.

1,1′-Bis(tri-n-dodecylsilyl)ferrocene. The crude tri-*n*-dodecylsilylcyclopentadiene, prepared as described above, was added over a 5-hr. period to a vigorously stirred mixture of

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[10] M. Rosenblum and R. B. Woodward, *J. Am. Chem. Soc.*, 80, 5443 (1958).

[11] All melting and boiling points are uncorrected. All operations were performed under a nitrogen atmosphere, and preparations utilizing reactive chlorosilanes or organometallic compounds were carried out under a dry nitrogen atmosphere.

[12] The elemental analyses were performed by Messrs. H. L. Thornburg and J. B. Kessler of these laboratories.

[13] Prepared by adding 330 g. (6.9 moles) of cyclopentadiene in an equal volume of tetrahydrofuran to 129 g. (5.6 g.-atoms) of sodium dispersed in 3200 ml. of anhydrous tetrahydrofuran.

119 g. (5.2 moles) of sodium dispersed in 5 l. of refined tetrahydrofuran. The reaction temperature was maintained at 0 to 5° during the addition and for 1 hr. thereafter. After further stirring at room temperature for 7 hr., the solution was cooled to 0° and 2.5 moles of freshly prepared iron(II) chloride[14] in 2 l. of refined tetrahydrofuran was added in portions with vigorous stirring. The temperature rose to 18° during this addition. The resulting suspension was stirred at ambient temperature for 1 hr., then at 55 to 60° for 12 hr. The solvent was then distilled at reduced pressure, and 5 l. of low boiling petroleum ether was added. The resulting solids were removed by centrifugation, and the petroleum ether and residual tetrahydrofuran were distilled to a flask temperature of 100°/10 mm. The residual concentrate was refined on a DPI molecular still to yield 1684 g. (1.34 moles, 53.5%) of an oil, vapor temperature 290–295°/0.004 mm. Fourteen separate fractions were collected n_D^{20} 1.4916 to 1.4931; the spectra of all 14 fractions were practically identical.

Composite, n_D^{20} 1.4922.

An infrared spectrum of this material indicated that it was free of Si—H and Si—O—Si bonds. There were weak bonds at 9 and 9.95 μ. These bonds also appeared in the spectrum of chlorotri-*n*-dodecylsilane.

Anal. Calcd. for $C_{82}H_{158}FeSi_2$: C, 78.41; H, 12.66; Fe, 4.48; Si, 4.45; mol. wt., 1256. Found: C, 78.32; H, 12.73; Fe, 4.49; Si, 4.15; mol. wt. (Menzies-Wright in benzene), 1234.

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[14] Prepared by the method of G. Wilkinson, *Org. Syntheses*, **36**, 31 (1956), using a 40 mole per cent excess of iron powder.

[CONTRIBUTION FROM THE NONMETALLIC MATERIALS LABORATORY, DIRECTORATE OF MATERIALS & PROCESSES, AERONAUTICAL SYSTEMS DIVISION]

Synthesis of Organolithium Compounds of Some Group IV and V Elements

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Triphenylgermyllithium, triphenyltinlithium, triphenylleadlithium and diphenylphosphinolithium have been prepared by the reaction of the various chlorides with lithium in tetrahydrofuran. Hexaphenyldigermane, hexaphenylditin, hexaphenyldilead, and tetraphenylbiphosphine have also been cleaved by lithium in tetrahydrofuran to yield the corresponding $(C_6H_5)_nM^{n+1}Li$ in excellent yields.

The direct method of preparing triphenylsilyllithium from chlorotriphenylsilane and lithium metal in tetrahydrofuran was first reported by Gilman.[1a] This report prompted our investigation to determine whether this was a convenient and general reaction applicable to other elements of Group IV and V of the periodic table to yield organolithium reagents of the general formula $(C_6H_5)_nM^{n+1}Li$. Wittig and later Tomboulian have prepared triphenylmethyllithium[2] from chlorotriphenylmethane and lithium in tetrahydrofuran, while Gilman[3] has reported the preparation of triphenylgermyllithium from bromotriphenylgermane and lithium in tetrahydrofuran.

In the past organolithium compounds have also been prepared by various other means: A. Butyllithium or phenyllithium reacts with $(C_6H_5)_3CH$,[4] $(C_6H_5)_2PH$,[5] and $(C_6H_5)_3GeH$[6] to yield $(C_6H_5)_3CLi$, $(C_6H_5)_2PLi$, and $(C_6H_5)_3GeLi$, respectively.

$$(C_6H_5)_nM^{n+1}H + C_4H_9Li \longrightarrow (C_6H_5)_nM^{n+1}Li + C_4H_{10} \quad (1)$$
$$M = C, P, Ge$$

B. Lithium in tetrahydrofuran has thus far successfully cleaved hexaphenyldisilane[7] and hexaphenyldigermane[8] to yield triphenylsilyllithium and triphenylgermyllithium. Attempts to cleave hexaphenylditin[9] have been reported to produce triphenyltinlithium in low yields.

$$(C_6H_5)_nM^{n+1}M^{n+1}(C_6H_5)_n + 2Li \longrightarrow 2(C_6H_5)_nM^{n+1}Li \quad (2)$$
$$M = Si, Ge, Sn$$

C. Lithium in tetrahydrofuran can also cleave a phenyl group from certain perphenylated elements[10]

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[1] University of Dayton, Research Institute, Dayton, Ohio.

[1a] H. Gilman, D. J. Peterson, and D. Wittenberg, *Chem. & Ind.*, 1479 (1958).

[2] G. Wittig, R. Mangold, and G. Felletschin, *Ann.*, **560**, 116 (1948); P. Tomboulian, *J. Org. Chem.*, **24**, 229 (1959).

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