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A New Organosilicon Synthon, 2,3,5,6-Tetrakis(trimethylsilyl)-1,3-cyclohexadiene. Versatile Polyfunctionalization of *o*-Dichlorobenzene†

Jacques Dunoguès,* Denis N'Gabe, Michel Laguerre, Norbert Duffaut, and Raymond Calas

Laboratoire de Chimie Organique du Silicium et de l'Etain associé au CNRS (L.A. 35),
Université de Bordeaux I, 351, Cours de la Libération, 33405 Talence Cêdex, France

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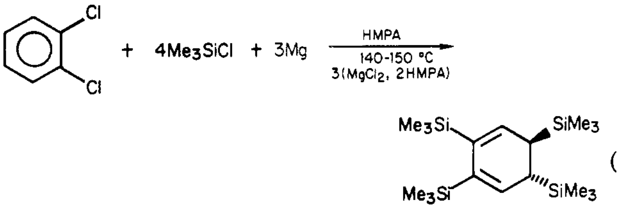
Direct tetrasilylation of *o*-dichlorobenzene using the Me_3SiCl/Mg/hexamethylphosphoramide (HMPA) reagent at 140–150 °C affords a new organosilicon intermediate, 2,3,5,6-tetrakis(trimethylsilyl)-1,3-cyclohexadiene, I, in 90% yield. This product is reactive in cycloaddition reactions with maleic anhydride and dimethyl acetylenedicarboxylate. In the latter case, *trans*-1,2-bis(trimethylsilyl)ethylene and dimethyl 4,5-bis(trimethylsilyl)-*o*-phthalate, a precursor of 4,5-diodo-*o*-phthalic acid, were formed in quantitative yield. Hydrolysis of I provides 3,6-bis(trimethylsilyl)-1,4-cyclohexadiene. Thus *o*-dichlorobenzene can be converted selectively to 1,2-, 1,3-, or 1,4-bis-,1,2,4-tris-, or 1,2,4,5-tetrakis(trimethylsilyl)benzene, intermediates which serve in the regiospecific synthesis of 1,2-, 1,3-, 1,4-, 1,2,4-, and 1,2,4,5-functional benzenes, respectively.

As a part of our studies of the synthesis of silylated polyunsaturated hydrocarbons from aromatic precursors as starting materials[1] we have focused our interest on the use of polyhaloaromatic hydrocarbons and we have investigated the reactivity of *o*-dichlorobenzene. Complete[2,3] or partial[4] silylation of this compound had been studied previously, and (*o*-(trimethylsilyl)phenyl)- or (*o*-chlorophenyl)trimethylsilanes were obtained. We have reinvestigated the silylation reaction with use of more drastic conditions.

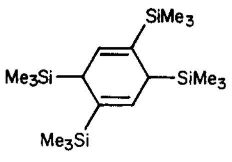
Results and Discussion

In the presence of magnesium and with HMPA as the solvent, trimethylchlorosilane reacted with *o*-dichlorobenzene at 140–150 °C (eq 1). In order to identify the product, we compared its ^1H NMR spectrum with those of the tetrasilylated derivatives II–IV. These had been obtained in high (for II) or low (for III and IV) yields by Brennan and Gilman[5] by silylation of *p*-dichloro-, *p*-dibromo-, or *p*-bis(trimethylsilyl)benzene with an excess of the Me_3SiCl/Li/THF reagent.

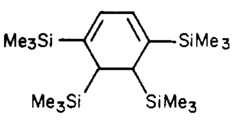


I (90%)

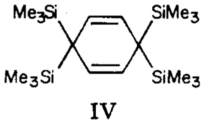
(1)



II



III

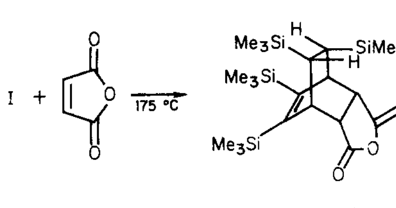


IV

^1H NMR data (δ): I (in CCl_4; $HCCl_3$ at δ 7.27 as the internal standard), two signals at 6.15 and 6.03 (two olefinic H), two signals at 1.74 and 1.62 (two allylic CHSi); II,[5] two signals centered at 5.91 (two olefinic H), two signals centered at 2.67 (two biallylic CHSi); III,[5] two signals centered at 5.88 (two olefinic H), two signals centered at 2.42 (two allylic CHSi); IV,[5] one signal at 5.10 (four olefinic H), no allylic CHSi.

Although NMR data of II–IV were not completely given, one can easily note that the product we obtained is different from II and IV which had been identified with certainty by Brennan and Gilman. We synthesized II from *p*-bis(trimethylsilyl)benzene according to a route described by us previously in the case of benzene (65% yield) and found it unreactive in a Diels–Alder reaction with dimethyl acetylenedicarboxylate. The structure of III was suggested as such by the authors but was not proven. Consequently, it was necessary to continue our study; the chemical properties of I confirmed both the structure and the stereochemistry proposed.

The Diels–Alder cycloaddition of I with maleic anhydride afforded solid adduct V (eq 2). The NMR spectrum of V showed the absence of ethylenic protons and the presence of two Me_3Si groups bonded to sp^2-carbon atoms



V (60%)

(2)

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† With technical collaboration of Françoise Pisciotti.

(1) (a) R. Calas and J. Dunoguès, *J. Organomet. Chem. Libr.*, **2**, 277 (1976) and references therein; (b) M. Laguerre, J. Dunogues, R. Calas, and N. Duffaut, *J. Organomet. Chem.*, **112**, 49 (1976); (c) M. Laguerre, G. Félix, J. Dunoguès, and R. Calas, *J. Org. Chem.*, **44**, 4275 (1979); (d) *Tetrahedron Lett.*, **22**, 1227 (1981).

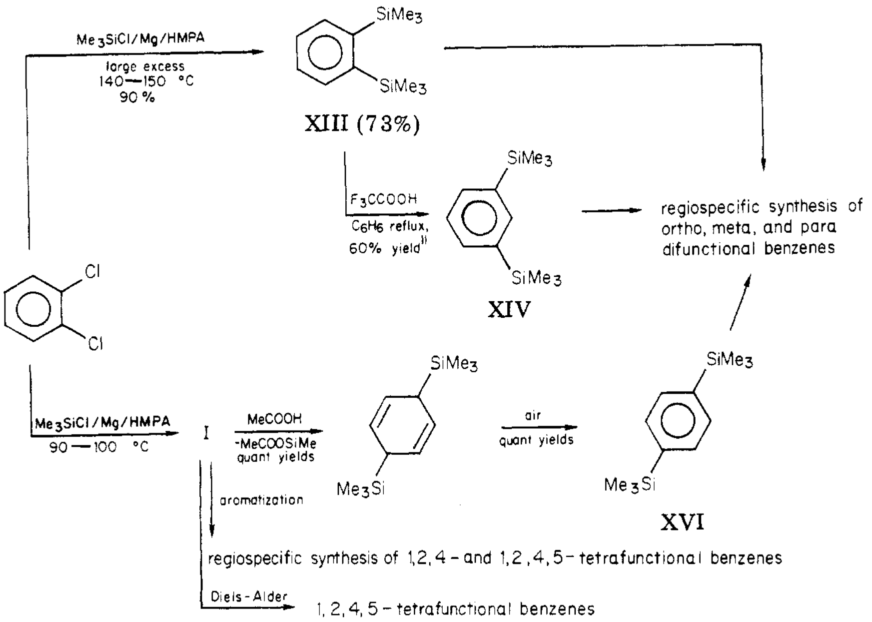
(2) (a) H. Clark, A. F. Gordon, C. W. Young, and M. J. Hunter, *J. Am. Chem. Soc.*, **73**, 3798 (1951); (b) C. Eaborn, K. L. Laura, and D. R. M. Walton, *J. Chem. Soc.* 1198 (1964); (c) C. Eaborn, and D. R. M. Walton, *J. Chem. Soc. B* 15 (1969).

(3) P. Bourgeois and R. Calas, *J. Organomet. Chem.*, **84**, 16 (1975).

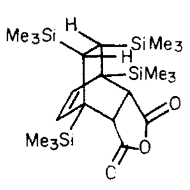
(4) J. Dunoguès, E. Jousseaume, and R. Calas, *J. Organomet. Chem.*, **71**, 377 (1974).

(5) T. Brennan and H. Gilman, *J. Organomet. Chem.*, **12**, 291 (1968).

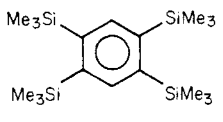
Scheme I



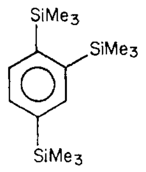
and two Me_3Si groups bonded to sp^3-carbon atoms (^1H and ^{29}Si NMR spectroscopy) (cf. Experimental Section) and is in agreement with structure V as shown and therefore with I and is not compatible with the alternate structure Va which should result form the reaction of III.



Va

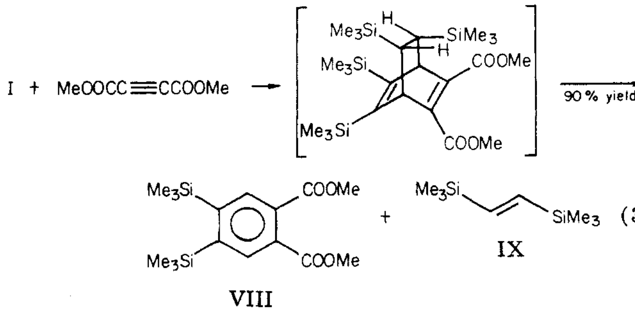


VI



VII

Aromatization of I by refluxing in benzene in the presence of chloranil provided a mixture of VI (6–12%) and VII (~60%). These are precursors for tetra- or trifunctional benzenes, respectively. This result confirmed the structure of I. I underwent a Diels–Alder reaction with dimethyl acetylenedicarboxylate, but the expected adduct was not isolated. Dimethyl 4,5-bis(trimethylsilyl)-*o*-phthalate, VIII, and *trans*-1,2-bis(trimethylsilyl)ethylene, IX, were formed quantitatively according to eq 3.

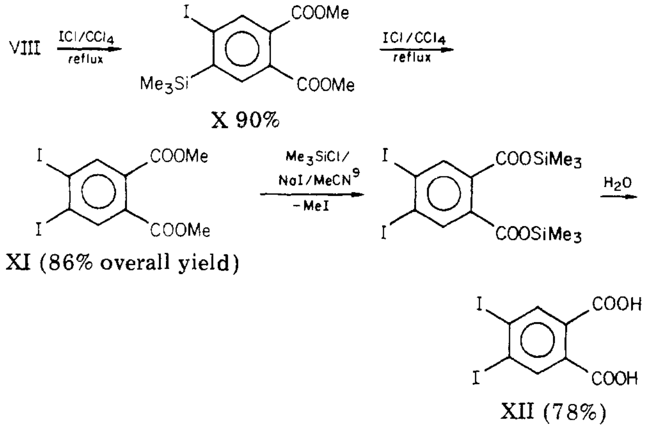


(3)

VIII IX

Since the difficult to prepare[6,7] cis isomer of IX is not appreciably isomerized when heated to 150 °C,[6,7] we conclude that I has a trans structure. (Such Diels–Alder reactions do not involve epimerization.)

From a synthetic point of view, the Diels–Alder reaction of I with dimethyl acetylenedicarboxylate has a double advantage: (i) the simple and rapid synthesis of IX, a useful precursor of 1,2 bifunctional ethylenes,[8] and (ii) the possibility of access to 1,2,4,5 tetrafunctional benzenes from either I or VIII. As an example, we studied iodination of VIII, a regiospecific precursor of 4,5-diodo-*o*-phthalic acid, which is a possible tracer for biological fluids.



X 90%

XI (86% overall yield)

XII (78%)

During the course of the hydrolysis of XI the diiodo monoacid ester XIa was identified (but not isolated) by NMR spectroscopy. Me_3SiI prepared in situ[9] was used for the hydrolysis of the ester functions to avoid the possible deiodination during the course of a normal saponification. XII previously was very difficult to prepare since it was isolated in only ~8% yield during the iodination of *o*-phthalic acid under drastic conditions (long heating at 76 °C and rapid heating at 200 °C, in 50% fuming sulfuric acid) together with a complex mixture of products.[10]

Hydrolysis of I in the presence of acetic acid led to 3,6-bis(trimethylsilyl)-1,4-cyclohexadiene, XV, which was aromatized into the *p*-bis(trimethylsilyl)benzene, XVI, in the presence of air. Since the Seyferth method[11] permits

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(6) V: reference given in V. Bažant, V. Chvalovský, and J. Rathouský, Academic Press, New York and London. Concerning the *cis*-bis(trimethylsilyl)ethylene; see also the 2nd and 3rd ed. of this dictionary.

(7) H. Bock and H. Seidl, *J. Organomet. Chem.*, **13**, 87 (1968).

(8) (a) J.-P. Pillot, J. Dunoguès, and R. Calas, Bull. Soc. Chim. Fr., 2143 (1975); (b) Synth. Commun., 395 (1979).

(9) G. A. Olah, S. C. Narang, B. G. B. Gupta, and R. Malhotra, *J. Org. Chem.*, **44**, 1247 (1979).

(10) D. S. Pratt and G. A. Perkins, *J. Am. Chem. Soc.*, **40**, 221 (1918).

conversion of *o*-bis(trimethylsilyl)benzene to the corresponding meta isomer, it is now possible to use *o*-dichlorobenzene for a regiospecific synthesis of ortho, meta, and para difunctional benzenes. These observations are summarized in Scheme I.

Experimental Section

Generally ^{1}H NMR spectra were recorded on a Varian A 60 or Hitachi Perkin-Elmer R-24 B (60-MHz) instrument. Unless otherwise specified, CCl_4 with $HCCl_3$ (δ 7.27) as the standard was used as the solvent for all NMR spectra, and results are given as δ values.

Trimethylchlorosilane and HMPA were used after distillation. HMPA should be handled with great care since it has been found to cause cancer in laboratory animals[13] (handle in a cooker hood until the acidic hydrolysis and neutralization are carried out).

Preparation of 2,3,5,6-Tetrakis(trimethylsilyl)-1,3-cyclohexadiene. HMPA (200 mL), magnesium powder (Carlo-Erba, Co d.459065; 10 g, 0.32 mol), and trimethylchlorosilane (80 g, 0,73 mol) were charged into a four-necked round-bottomed flask equipped with a mechanical stirrer, a thermometer, a dropping funnel, and a reflux condenser topped with a calcium chloride tube. After the soltion was stirred 0.5 h in a oil bath heated to about 150 °C, *o*-dichlorobenzene (14.7 g, 0.1 mol) in HMPA (100 mL) was added dropwise while the temperature was maintained as indicated until the magnesium had been consumed (24–48 h). During the course of the reaction, crystals of $MgCl_2 \cdot 2HMPA$ appeared in the cold part of the vessel and precipitated during the cooling. The mixture then was poured onto crushed ice in the presence of an excess of HCl solution and extracted with ether, and the ethereal layer was washed several times. The low boiling products were removed under reduced pressure (25 torr). Then I was isolated by distillation: bp 105–110 °C (0.1 torr); 33.1 g (90%); IR 3030, 2970, 2910, 2860, 1540, 1415, 1265, 1255, 1185, 1105, 1035, 980, 880, 840, 760, 715, 705, 695, 640 cm^{-1}; ^{1}H NMR (part not given in the Results) δ 0.20 (s, 9 H, $SiMe_3$), 0.33 (s, 9 H, $SiMe_3$) (the resolution of the AA′XX′ spectrum due to the hydrogen atoms ofthe ring did not improve at 270 MHz); ^{29}Si NMR (17.87 MHz) (C_6D_6, lock $CDCl_3$) 2.33 (allylic Si), −5.24 (vinylic Si); the molecular peak of I (M, *m/e* 368) was observed in mass spectroscopy.

Anal. Calcd for $C_{18}H_{40}Si_4$: C, 58.7; H, 10.9, Si, 30,4. Found: C, 58.5; H, 10.7; Si, 30,2.

Diels–Alder Reaction of I with Maleic Anhydride. I (5 g, 136 mmol) was warmed for 6 h at 175 °C with maleic anhydride (3 g, 304 mmol). After the mixture was cooled, the adduct V was purified by chromatography (silica column, methylene chloride eluent; 3.8 g (60%). V was identified by its mass spectrum peaks, namely, at *m/e* 466 (molecular peak), 451 (M − 15) 438 (M − 28), and 393 (M − 73). ^{1}H NMR δ 0.20 (s, 9 H, $SiMe_3$), 0.34 (s, 9 H, $SiMe_3$), 0.40 (s, 18 H, 2siMe$_3$, 2 s at 90 MHz) 0.81 (m, 2 H, 2CHSi), 3.14 (m, 2 H), 3.60 (m, 2 H) (CHC=O and CHC=C not assigned); ^{29}Si NMR δ −8.16 and −7.75 (2SiC=C), +4.28 and +4.49 (2SiCC) (SPI assignation).

Anal. Calcd for $C_{22}H_{42}O_3Si_4$: C, 56.65; H, 9.0. Found: C, 56.3; H, 8.9.

Aromatization of I. A mixture of I (2.56 g, 7 mmol) dissolved in benzene (30 mL) and chloranil (2.1 g, 8.5 mmol) was heated at reflux for 24 h under an argon atmosphere. After the mixture was cooled, pentane (100 mL) was added. The solution was filtered and the pentane removed. By chromatography on Al_2O_3 VI (0.15–0.3 g (6–12 %); mp 170–171 °C) and VII (≃3 g (≃60 %); mp 45 °C) were isolated and identified by comparison with authentic samples prepared according to previously reported routes.[3,5]

Synthesis of the 1,3,4,6-Tetrakis(trimethylsilyl)-1,4-cyclohexadiene (II). Using the route previously described for the silylation of benzene,[1a] we synthesized II from *p*-bis(trimethylsilyl)benzene in 65% yield.

Diels–Alder Reaction of I with Dimethyl Acetylenedicarboxylate. I (3.68 g, 10 mmol) and dimethyl acetylenedicarboxylate (1.42 g, 10 mmol) were heated for 48 h at 175 °C. After the mixture was cooled, a ^{1}H NMR spectrum of the reaction mixture indicated the quantitative formation of *trans*-1,2-bis(trimethylsilyl)ethylene, IX (^{1}H NMR δ 0.05 (s, 18 H, 2SiMe$_3$), 6.56 (s, 2 H, CH=CH[7,14])), and 4,5-bis(trimethylsilyl)-*o*-phthalate, VIII (^{1}H NMR δ 0.52 (s, 18 H, 2SiMe$_3$), 4.23 (s, 6 H, 2COOMe), 7.90 (s, 2 aromatic H)).

Anal. Calcd for $C_{16}H_{26}O_4Si_2$: C, 56.8; H, 7.7. Found: C, 56.9; H, 7.5.

IX and VIII were separated on a silica gel column by using first pentane and then pentane/methylene chloride (1/1 vol) as the eluent (90% yield).

IX also was identified by comparison with an authentic sample synthesized according to ref 7. The structure of VIII was confirmed by mass spectrometry (molecular ions at *m/e* 338, 323 (M − 15)$^+$, 307 (M − 31), 279 (M − 59), etc.) and by its chemcial behavior (see below).

Attempts to effect a Diels–Alder reaction between II and dimethyl acetylenedicarboxylate under similar conditions were unsuccessful.

Iodination of VIII. ICl (6 g, 37 mmol) was added dropwise to a stirred solution of VIII (3.4 g, ~10 mmol) in CCl_4 (30 mL). The mixture was heated at reflux for 24 h, cooled, diluted with ether (100 mL), and washed with an aqueous solution of sodium thiosulfate and then several times with water. The ethereal layer was dried over sodium sulfate, and the low boiling point products were removed. Remaining 4,5-diodo-*o*-phthalate, XI, was recrystallized from ethanol; mp 117 °C; 86% yield; ^{1}H NMR δ 3.82 (s, 6 H, 2COOMe), 8.05 (s, 2 aromatic H); mass spectrum, *m/e* 446 (molecular peak) 415 (M − 31), 384 (M − 62), 356 (M − 62 − 28), 127 (I).

By reaction of ICl (1.63 g, 10 mmol), VIII (3.4 g, 10 mmol) and CCl_4 (30 mL), 4-iodo-5-(trimethylsilyl)-*o*-phthalate, X, was formed: yield 90%; ^{1}H NMR δ 0.50 (s, 9 H, $SiMe_3$), 3.83 (s, 6 H, 2COOMe), 7.63 (s, 1 aromatic H), 8.07 (s, 1 aromatic H).

Hydrolysis of XI into the 4,5-Diodo-*o*-phthalic Acid (XII). XII (~1 g, 78% yield) was obtained from XI (1.5 g, 30 mmol), NaI (7.5 g, large excess), Me_3SiCl (5.5 g, 50 mmol, large excess), and anhydrous MeCN (50 mL according to the procedure reported by Olah et al.,[9] with only the reaction time longer, 80 h). XII was recrystallized from boiling acetic acid: mp 218–220 °C (uncorrected) (lit.[10] mp 221–222 °C); identified by its mass spectrum (molecular peak M, *m/e* 418, very weak, regarding the main peaks at *m/e* 400 (M − 18), 356 (M − 18 − 44), 229 (M − 18 − 44 − 127), 201 (M − 217), 127 (corresponding to the presence of iodine), 74 (corresponding to C_6H_2)); IR (COOH)ν(OH) between 3700 and 3000 cm^{-1}; ^{1}H NMR disappearance of the COOMe groups. The corresponding anhydride (mp 217 °C) aslo was obtained according to ref 10.

Before the reaction was complete the monoacid ester was observed by ^{1}H NMR spectroscopy (presence of 1 H (COOH exchange with D_2O), 1CH$_3$(COOMe), and two singlets (1 H) in the region of the aromatic H).

Hydrolysis of I in the Presence of Acetic Acid. A mixture of I (5.62 g, 15 mmol) and acetic acid (3.4 g, 57 mmol) was refluxed for 16 h, cooled, diluted with CCl_4, treated with aqueous Na_2CO_3 for 3 h, then washed several times with water, and dried over $MgSO_4$. The volatiles were removed under reduced pressure (25 torr) to give a residue of 3,6-bis(trimethylsilyl)-1,4-cyclohexadiene, XV. In the presence of air XV underwent aromatization affording XVI.[1b] XV and XVI were identified by comparison of their properties with those of samples synthesized according to ref 1b.

Synthesis of *o*-Bis(trimethylsilyl)benzene (XIII). We used our previously reported route,[3,4] and XIII was obtained in 73% yield.

Synthesis of *m*-Bis(trimethylsilyl)benzene (XIV). Isomerization of XVIII (22.2 g, 0.1 mol) according to the method

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(11) D. Seyferth and D. A. White, *J. Am. Chem. Soc.*, **94**, 3132 (1972). High yields are observed from a few millimoles of XIII as reported by these authors but from 0.1 mol of XIII the *meta* derivative XIV was formed in 55–60% yields.

(12) (a) G. Félix, F. Pisciotti, J. Dunoguès, and R. Calas, *Angew. Chem.*, **89**, 502 (1977); *Angew. Chem., Int. Ed. Engl.* **16**, 488 (1977); (b) *ibid.*, **18**, 402 (1979). For previous studies, see the review of C. Eaborn, *J. Organomet. Chem.*, **100**, 43 (1975).

(13) J. A. Zapp, Jr., *Science* (*Washington, D.C.*), 190 (1975).

(14) J. Dunoguès, J.-P. Pillot, and R. Calas, *C. R. Hebd. Seances Acad. Sci., Ser. C*, **278**, 467(1974).

reported by Seyferth and White[11] afforded XIV in 50–60% yield. XIV also was identified by comparison with an authentic sample.

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Registry No. I, 82679-24-1; II, 17229-87-7; III, 18921-01-2; IV, 17156-62-6; V, 82679-25-2; VI, 17156-61-5; VII, 17864-15-2; VIII, 82679-26-3; IX, 18178-59-1; X, 82679-27-4; XI, 67193-32-2; XII, 82679-28-5; XIII, 17151-09-6; XIV, 2060-89-1; XV, 18090-43-2; XVI, 13183-70-5; *o*-dichlorobenzene, 95-50-1.

Thermolysis Mechanism of Symmetrical and Unsymmetrical Dialkylplatinum(II) Complexes with Tertiary Phosphine Ligands. Controlling Factors for β Elimination

Sanshiro Komiya, Yoshiyuki Morimoto, Akio Yamamoto,* and Takakazu Yamamoto*

Research Laboratory of Resources Utilization, Tokyo Institute of Technology, 4259 Nagatsuta, Midori-ku, Yokohama 227, Japan

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A series of symmetrical and unsymmetrical *cis*-dialkylplatinum(II) complexes with tertiary phosphine ligands *cis*-$\mathrm{Pt(R_a)(R_b)L_2}$ ($\mathrm{R_a}$, $\mathrm{R_b}$ = Me, Et, *n*-Pr, *i*-Pr, *n*-Pr, *i*-Bu, *n*-pentyl, $\mathrm{C_2H_4Ph}$, $\mathrm{C_2H_4C_6H_4}$-*p*-OMe; $\mathrm{L_2} = \mathrm{(PPh_3)_2}$, $\mathrm{(PPhMe_2)_2}$, dpe) has been prepared by reactions of $\mathrm{Pt(R_a)ClL_2}$ with alkylating reagents such as $\mathrm{R_bLi}$ and $\mathrm{R_bMgX}$. These complexes are characterized by elemental analyses and IR and NMR (^{1}H and ^{31}P{^{1}H}) spectroscopy as well as by chemical reactions. Thermolysis of the dialkylplatinum complexes gives only the disproportionation products of the two alkyl groups via β elimination. A kinetic study of the thermolysis reveals the existence of the nondissociative path in addition to the dissociative one. In the presence of an excess of $\mathrm{PPh_3}$, thermolysis proceeds mainly via a nondissociative path in which the liberation of olefin after facile reversible β elimination is probably rate determining. When the thermolysis of the unsymmetrical *cis*-$\mathrm{Pt(R_a)(R_b)L_2}$ type complex is carried out in the absence of excess $\mathrm{PPh_3}$, the relative rate of β elimination involving the $\mathrm{R_a}$ group to that involving the $\mathrm{R_b}$ group is proportional to the ratio of the number of β-hydrogen atoms in the $\mathrm{R_a}$ group to that in the $\mathrm{R_b}$ group.

Introduction

Transition-metal complexes having metal-to-carbon bonds play crucial roles in various organic reactions catalyzed by transition metals. Understanding the behavior of these transition-metal alkyl complexes is of fundamental importance in the elucidation of the metal-promoted catalytic processes. Recent studies on the thermal decomposition of isolated transition-metal alkyl complexes have shed some light for understanding the stability and reactivity of transition metal-to-carbon bond.[1] For transition-metal dialkyl complexes several thermolysis pathways are possible: (1) homolytic cleavage of the M–C bond (radical pathway); (2) reductive elimination (coupling reaction); (3) β elimination (disproportionation of two alkyl groups); (4) other pathways (α or γ elimination).

Among them, the β-elimination process is the most frequently observed thermolysis pathway. Clarification of the factors controlling the thermolysis pathway through β elimination is essential in understanding the mechanisms of the catalytic processes involving olefins and for design of organic syntheses utilizing transition-metal catalysts.

Dialkylplatinum(II) complexes, among other transition-metal alkyls, serve as excellent models for studying the β-elimination process since thermal decomposition of $\mathrm{PtR_2L_2}$ results in the clean disproportionation liberating alkane, RH, and alkene, R(–H), in a 1:1 ratio.

$$\mathrm{PtR_2L_2} \xrightarrow{\Delta} \mathrm{RH} + \mathrm{R(-H)} + \mathrm{Pt(0)}\ \text{complexes}$$

Whitesides proposed a dissociative mechanism for the decomposition of $\mathrm{Pt(}n\mathrm{\text{-}Bu)_2(PPh_3)_2}$ and pointed out the importance of blocking the site for β elimination by the ligand in increasing the stability of the complex.[2] Reger also showed that an open coordination site on iron, formed by ligand dissociation, is required for β elimination from $\mathrm{Fe(Cp)(CO)(PPh_3)R}$ to occur.[3] Recently, the steric influence of the ligands in *trans*-$\mathrm{PdEt_2L_2}$ in the disproportionation reaction of the ethyl groups has been reported.[4] In this case, a thermolysis process involving ligand dissociation does not constitute the main decomposition pathway and the presence of bulky tertiary phosphine ligands in the undissociated *trans*-$\mathrm{PdEt_2L_2}$ was found to facilitate the β-elimination process rather than to block it in contrast to the behavior of platinum dialkyls.

In order to obtain further insight into the β-elimination process, we have initiated a project to prepare and thermolyze a series of unsymmetrical dialkylplatinum(II) complexes, since the examination of products of the thermal decomposition was expected to afford direct information about the relative ease of β-hydrogen elimination from platinum alkyls. A part of the results have been

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