A Novel (Tricobaltiomethoxy)silatrane: Synthesis and Structure of $[Co_3(CO)_9\{\mu_3\text{-}CO\overline{Si(OCH_2CH_2)_3N}\}]$[1]

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Introduction

Silatranes[2] of type $X\overline{Si(OCH_2CH_2)_3N}$ are cyclic silicon ethers containing a hypervalent silicon atom with a transannular bonding interaction[3] between the silicon and the bridgehead nitrogen atom. The nature of substituents X affects the properties of silatranes such as the biological activities[4] including antitumor properties[5] and the strength of the transannular N–Si bonding.[6]

The reactivity of silatranes, unlike the relatively wider scope of triazasilatranes, is limited in narrow range and largely depends on the type of X.[2,8–12] The exocyclic Lewis basicity of 1-alkoxysilatranes,[10] π-coordinating ability of 1-arylsilatranes,[11] and the formation of the Si–Pt bonding interaction via HCl elimination[12] are examples in point. In particular, the structural analysis[12] of the compound $[Pt\{Si(OCH_2CH_2)_3N\}Cl(PMe_2Ph)_2]$, which constitutes the only silatrane with a bonding interaction between Si and a transition metal, reveals that the formation of a Si–Pt bond occurs concomitantly with the disappearance of the transannular interaction in the silatrane moiety, raising at least two engaging questions as to the synthesis of new silatrane–platinum complexes with anticancer biological activity and the effect of the introduction of a Si–transition metal bonding interaction on the transannular Si–N interaction.

As part of our efforts in pursuing this matter, we have explored wide range of reactions including the oxidative addition reaction of 1-hydrosilatrane with $Co_2(CO)_8$. Reported herein are accounts of the synthesis and structural characterization of a hitherto unprecedented 1-((enneacarbonyltricobaltio)methoxy)silatrane $[Co_3(CO)_9\{\mu_3\text{-}CO\overline{Si(OCH_2CH_2)_3N}\}]$.

Experimental Section

Preparation of Compounds. All operations were carried out under a pure dinitrogen atmosphere. Organic solvents were carefully dried by distillation from suitable drying agents under an atmosphere of dinitrogen and degassed prior to use: THF, from sodium benzophenone ketyl; *n*-hexane, from sodium metal; CH_2Cl_2, from CaH_2 followed by storage over activated 3A molecular sieves. The compounds $Co_2(CO)_8$, $\overline{N(CH_2CH_2O)_3B}$ (Aldrich), and $HSi(OEt)_3$ (Petrach) were used as received. 1-Hydrosilatrane was prepared from boratrane according to the literature method.[2]

$[Co_3(CO)_9\{\mu_3\text{-}CO\overline{Si(OCH_2CH_2)_3N}\}]$ (1). $Co_2(CO)_8$(0.515 g, 1.5 mmol) and 1-hydrosilatrane (0.175 g, 1 mmol) were dissolved in ca. 30 mL of THF and the resulting yellowish brown solution was stirred for 12 h at room temperature, resulting in a dark red reaction mixture. Small amount of violet precipitate was removed by filtration, and the dark red solid obtained by removal of solvent of the filtrate was washed twice with 20 mL of *n*-hexane and then twice with 20 mL of H_2O and dried *in vacuo*. Double recrystallization of this solid from THF/*n*-hexane afforded analytically pure crystalline red solids **1** in a yield of 54% (0.340 g). Anal. Calcd for $C_{16}H_{12}Co_3NO_{13}Si$: C, 30.44; H, 1.90; N, 2.22. Found: C, 30.02; H, 2.08; N, 2.46. IR (KBr, cm^{-1}): 2980 (w), 2950 (w), 2901 (w), 2889 (w), 2093 (m), 2049 (sh, vs), 2030 (vs), 2024 (sh, vs), 1988 (s), 1488 (w), 1460 (w), 1343 (w), 1271 (w), 1122 (s), 1090 (s), 1043 (w), 1022 (w), 942 (w), 918 (w), 811 (m), 766 (w), 709 (m), 653 (w), 588 (m), 538 (w), 510 (m), 477 (w), and 438 (w). ^1H-NMR (CD_3CN): δ 3.04 (t, J = 6 Hz, N–CH_2) and 3.86 (t, J = 6 Hz, –O–CH_2). $^{13}C\{^1H\}$-NMR (CD_3CN): δ 51.70 (N–CH_2, $\Delta_{1/2}$ = 0.8Hz, $^1J_{C-H}$ = 140 Hz), 58.22 (O–CH_2, $\Delta_{1/2}$ = 0.8 Hz, $^1J_{C-H}$ = 148 Hz), 203.10 (terminal CO, $\Delta_{1/2}$ = 14 Hz). ^{29}Si MAS NMR: δ –104.9.

X-ray Crystal Structure Determination. A dark red cubic crystal of $[Co_3(CO)_9\{\mu_3\text{-}CO\overline{Si(OCH_2CH_2)_3N}\}]$ (1), grown from CH_2Cl_2/*n*-hexane solution, was mounted in a sealed capillary tube on an Enraf-Nonius CAD4 diffractometer with a Mo X-ray source equipped with a graphite monochromator. Unit cell parameters were determined from a least-squares fit of 25 accurately centered reflections having $12° \le 2\theta \le 20°$. The diffraction data were collected at room temperature in the ω–2θ scan mode to a maximum 2θ value of 45°. Three standard reflections, monitored every 60 min, showed no signs of decay in intensities. Details of the data collection and the crystal parameters are summarized in Table I. The intensity data were corrected for Lorentz and polarization effects. The structure was solved with use of Patterson (SHELXS 86) and difference Fourier methods and refined by blocked-matrix least-squares procedures (SHELX 76) on the CRAY-2S/4-128 supercomputer. An initial attempt at anisotropic refinement of all non-hydrogen atoms of two independent molecules in the asymmetric unit resulted in the observation of nonpositive definite behavior of four oxygen (12a, 21a, 23a, and 32a) and four carbon (5a, 21a, 31a, and 33a) atoms in one of two molecules. Therefore, all non-hydrogen atoms except those eight atoms were refined anisotropically. Hydrogen atoms were included in the two molecules at 1.08 Å from the bonded carbon atom with fixed U values of 0.07 Å^2. Final R factors and other data are given in Table I. Positional and equivalent isotropic thermal parameters for non-hydrogen atoms of **1** are listed in Table II.

Other Physical Measurements. All NMR spectra were recorded at ambient temperature. The ^1H (300.13 MHz) and $^{13}C\{^1H\}$ (75.47 MHz) solution FT NMR spectra were obtained with a Bruker AM-300 spectrometer equipped with a deuterium lock. The ^{29}Si solid-state FT NMR was also recorded on the same spectrometer operating at 59.63 MHz with the MAS technique. For measurement of solid-state NMR, a ground crystalline powder sample and the spinning rate of 4 kHz were used. Chemical shifts were referenced to external TMS. Infrared spectra were recorded on a BOMEM MB-100 FT IR spectrometer operating between 4000 and 400 cm^{-1} with a resolution of four reciprocal centimeters. Microanalyses were carried out by Han Yang Chemical Cooperation Research Center, Taejon, Korea.

Results and Discussion

The 2:3 molar reaction of $H\overline{Si(OCH_2CH_2)_3N}$ and $Co_2(CO)_8$ in THF produces in moderate yield $[Co_3(CO)_9\{\mu_3\text{-}CO\overline{Si(OCH_2CH_2)_3N}\}]$ (**1**) containing a rather ubiquitous triangular Co_3 unit

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(1) Dedicated to Professor Richard H. Holm on the occasion of his 60th birthday.

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Table I. Crystallographic Data for **1**

formula	$C_{16}H_{12}Co_3NO_{13}Si$	λ(Mo *K*α), Å	0.710 73
fw	631.15	transm coeff	0.999–0.642
temp, K	293	μ, cm^{-1}	22.4
cryst size, mm	0.50 × 0.50 × 0.40	scan method	ω–2θ
space group	$P\bar{1}$	2θ max, deg	45.0
a, Å	13.035(1)	no. of unique reflns	5849
b, Å	13.392(2)		
c, Å	15.563(1)	no. of obsd reflns ($I > 3\sigma(I)$)	4669
α, deg	66.40(1)		
β, deg	66.73(1)	no. of params refined	307 + 267 (2 blocks)
γ, deg	89.58(1)		
V, Å^3	2250.08(3)	R^a	0.064
Z	4	$R_w{}^b$	0.076
d_{calcd}, g cm^{-3}	1.863		

[a] $R = \sum||F_o| - |F_c||/\sum|F_o|$. [b] $R_w = [\sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2]^{1/2}$; $w = [\sigma^2(F) + 0.00967F^2]^{-1}$.

linked to an unprecedented (silatranyloxy)methylidyne group.[13] It is interesting to note that the observed major formation of $Et_3SiCo(CO)_4$ in the reaction system of $Et_3SiH/Co_2(CO)_8$ and $Et_3SiH/Co_2(CO)_8$/pyridine involves initial homoleptic cleavage of the Co–Co bond followed by oxidative addition of hydrosilane into the mononuclear cobalt radical species.[14] In the case of the latter reaction system with the longer reaction time, the *in situ* formation of $[Co_3(CO)_9COSiEt_3]$, a cluster closely related to **1**, has been detected, suggesting that the reaction route to **1** is likely to include oxidative addition of 1-hydrosilatrane.[15]

The new silatrane derivative **1** was characterized by elemental analysis, various spectroscopic means, and single-crystal X-ray diffraction study. The ν_{CO} spectrum (2093 m, 2049 vs, sh, 2024 vs, sh, 1988 s cm^{-1}) of **1** is structurally informative in a sense that its similarity to those of $[Co_3(CO)_9COSiEt_3]$[14] and $[Co_3(CO)_9COSiEt_2Co(CO)_4]$[16] indicates the presence of an enneacarbonyltricobalt unit in **1**. The ^1H and $^{13}C\{^1H\}$ NMR spectra show the feature of silatranyl methylene groups, and the solid-state ^{29}Si NMR[17] shift at −104.9 ppm implies the presence of strong transannular interaction between Si and the bridgehead atom N.[10] These structural implications and the bulk composition of **1** established by elemental analysis are consistent with the results of the X-ray diffraction study on **1**.

The crystal structure consists of discrete molecules, and depicted in Figure 1 is the molecular structure of one of the two independent molecules in the asymmetric unit. The two independent molecules adopt similar geometrical parameters but show a slight difference in spatial configuration of the silatranyl moiety with respect to the Co_3 plane as illustrated in Figure 2. Selected bond distances and angles are given in Table III. The structure of **1** contains a (silatranyloxy)methylidyne group triply bridging a $Co_3(CO)_9$ fragment. The Si–N distance of 2.010(5) Å in **1** constitutes the second shortest ever observed for silatranes, the shortest being that (1.965 Å) in $[Me_2O\overline{Si(OCH_2CH_2)_3N}](BF_4)$ (**2**(BF_4)).[10] These values can be informatively compared with the calculated Si–N value of 1.885 Å in the silatranyl cation $[\overline{Si(OCH_2CH_2)_3N}]^+$, where Si–N interaction is expected to be the strongest,[18] leading to an interesting conjecture that the silatranyl cation is trapped in the compound **1** via the interaction with $[Co_3CO(CO)_9]^-$, a well-defined anion.[13] The geometry around silicon approaches TBP, and the silicon atom situates 0.109 Å from the plane of the equatorial oxygens toward the axial oxygen, resulting in average O_{eq}–Si–O_{eq}, O_{ax}–Si–O_{eq}, and N–Si–O_{eq} anglesof 119.6, 93.8, and 86.2°, respectively. It is also worthy of noting that all Si–O_{eq} distances of 1.639, 1.644, and 1.661 Å

Table II. Positional and Equivalent Isotropic Thermal Parameters (Å^2) for Non-Hydrogen Atoms of **1**

atom	x/a	y/b	z/c	$\langle U^2\rangle$
		Molecule 1		
Co1	0.55485(8)	0.35604(7)	0.18233(7)	0.0352
Co2	0.68185(8)	0.53250(7)	0.11860(7)	0.0358
Co3	0.76179(8)	0.36276(7)	0.12637(7)	0.0343
Si1	0.54292(16)	0.31333(16)	0.44951(14)	0.0329
O1	0.63170(42)	0.32984(44)	0.49555(35)	0.0286
O2	0.51512(46)	0.19265(41)	0.45148(37)	0.0284
O3	0.46247(41)	0.40802(40)	0.42556(38)	0.0271
C1	0.59551(70)	0.28515(76)	0.60560(57)	0.0560
C2	0.42364(72)	0.10727(62)	0.53967(58)	0.0494
C3	0.36176(76)	0.40896(67)	0.50415(69)	0.0624
C4	0.47266(78)	0.28320(75)	0.65428(62)	0.0640
C5	0.40769(76)	0.12597(64)	0.63308(63)	0.0526
C6	0.31892(72)	0.29268(75)	0.59323(69)	0.0613
N1	0.42555(52)	0.24970(47)	0.59764(42)	0.0627
Cb	0.65011(55)	0.39208(56)	0.23609(54)	0.0372
Ob	0.64751(41)	0.36822(44)	0.32574(34)	0.0255
C11	0.55971(61)	0.37852(58)	0.05525(63)	0.0423
O11	0.56241(55)	0.39335(53)	−0.02043(46)	0.0515
C12	0.42067(69)	0.38815(67)	0.24874(63)	0.0524
O12	0.33287(56)	0.40307(63)	0.29228(56)	0.0610
C13	0.51015(69)	0.20648(70)	0.25423(60)	0.0529
O13	0.47768(62)	0.11380(50)	0.29901(51)	0.0525
C21	0.81014(65)	0.37611(66)	−0.00902(70)	0.0543
O21	0.83990(58)	0.38757(57)	−0.08960(48)	0.0559
C22	0.89051(74)	0.40950(62)	0.12456(53)	0.0450
O22	0.97228(54)	0.43820(56)	0.12442(50)	0.0513
C23	0.75055(67)	0.21711(83)	0.20127(64)	0.0579
O23	0.74122(63)	0.12597(51)	0.25114(60)	0.0602
C31	0.72116(73)	0.59002(63)	−0.02103(67)	0.0532
O31	0.74652(74)	0.62785(57)	−0.10709(51)	0.0639
C32	0.78209(85)	0.60102(69)	0.13702(77)	0.0685
O32	0.83809(74)	0.64334(67)	0.15851(78)	0.1037
C33	0.56688(74)	0.60227(65)	0.16041(58)	0.0518
O33	0.49387(63)	0.64583(57)	0.18805(57)	0.0722
		Molecule 2		
Co1a	0.93613(8)	1.13028(7)	−0.18971(7)	0.0362
Co2a	0.79358(8)	0.95677(7)	−0.09165(7)	0.0329
Co3a	0.73174(8)	1.13925(8)	−0.13626(7)	0.0370
Si1a	0.89509(17)	1.15494(15)	−0.43721(14)	0.0362
O1a	0.87951(49)	1.27097(41)	−0.42748(39)	0.0348
O2a	1.01651(49)	1.11012(47)	−0.45314(40)	0.0375
O3a	0.79925(44)	1.09434(42)	−0.45427(37)	0.0320
C1a	0.91221(76)	1.37479(66)	−0.51766(66)	0.0559
C2a	0.10974(73)	1.15828(81)	−0.55046(69)	0.0648
C3a	0.80812(83)	1.12228(77)	−0.55518(73)	0.0770
C4a	0.99831(116)	1.35935(80)	−0.60847(71)	0.0888
C5a	1.07316(101)	1.19913(98)	−0.63192(89)	0.0320
C6a	0.88189(89)	1.23387(97)	−0.62899(70)	0.0831
N1a	0.96768(52)	1.24743(50)	−0.59397(42)	0.0616
Cba	0.82771(58)	1.07904(53)	−0.22528(50)	0.0344
Oba	0.83096(48)	1.07137(40)	−0.30560(36)	0.0304
C11a	0.94804(66)	1.13386(66)	−0.07558(72)	0.0537
O11a	0.95414(65)	1.13383(69)	−0.00696(58)	0.0775
C12a	1.05073(66)	1.07369(63)	−0.24623(68)	0.0532
O12a	1.12635(62)	1.04044(57)	−0.29202(53)	0.0197
C13a	0.99584(74)	1.27002(75)	−0.28495(66)	0.0197
O13a	1.03720(59)	1.35671(57)	−0.36405(64)	0.0629
C21a	0.77275(66)	0.92063(64)	0.04246(70)	0.0516
O21a	0.76117(61)	0.89584(57)	0.12476(43)	0.0540
C22a	0.67379(68)	0.88512(68)	−0.08067(71)	0.0613
O22a	0.59403(69)	0.83754(66)	−0.07381(60)	0.0247
C23a	0.88945(66)	0.86913(60)	−0.12474(58)	0.0471
O23a	0.94702(58)	0.81163(56)	−0.14317(50)	0.0182
C31a	0.68791(66)	1.13607(63)	−0.00607(61)	0.0161
O31a	0.66081(56)	1.13165(56)	0.07187(45)	0.0573
C32a	0.75689(65)	1.28503(67)	−0.21981(59)	0.0159
O32a	0.77246(56)	1.37613(47)	−0.27147(51)	0.0495
C33a	0.60416(68)	1.10534(62)	−0.13907(59)	0.0496
O33a	0.52101(58)	1.08476(54)	−0.14434(49)	0.0175

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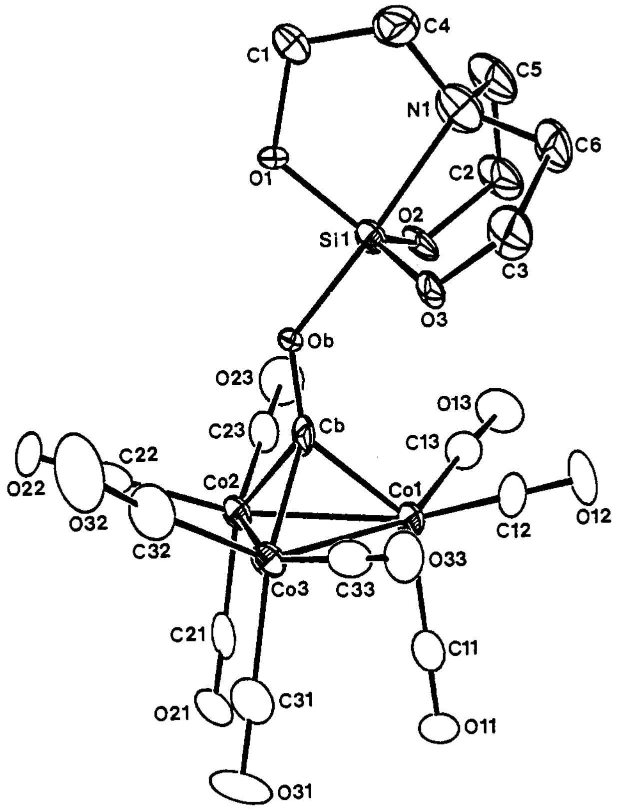

Figure 1. Structure of one (molecule 1) of the two independent $[Co_3(CO)_9\{\mu_3\text{-}COSi(OCH_2CH_2)_3N\}]$ (**1**) molecules, showing the atom-numbering scheme and 30% probability ellipsoids.

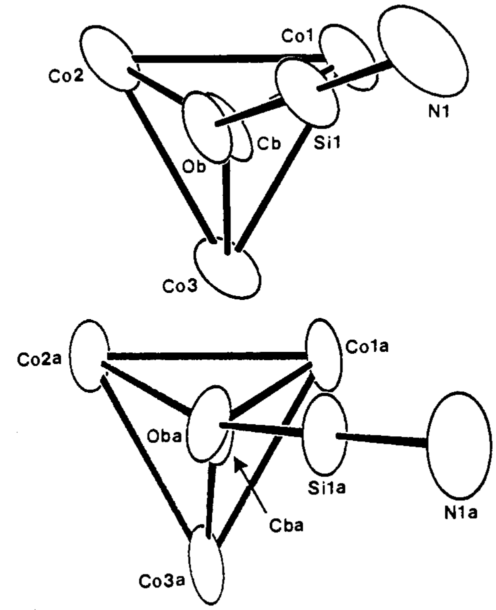

Figure 2. Projections of (silatranyloxy)methylidyne frameworks on Co_3 planes for two independent molecules of **1**. The atom-numbering scheme for molecule 2 is the same as that of molecule 1 (see Figure 1) except it is affixed with a letter "a".

in the silatranyl unit are shorter than the sum (1.83 Å) of their single-bond covalent radii[19] and are quite comparable with those observed for **2**(BF_4) (1.642 Å), 1-ethoxysilatrane (1.648 Å), and 1-ethoxysilatrane–trifluoroacetic acid adduct, $CF_3C(O)O\text{-}HEtOSi(OCH_2CH_2)_3N$ (**3**), (1.652 Å).[10] The distance between silicon and axial oxygen in **1** (Si–O_b = 1.715 Å) is similar to that (1.710 Å) in **3**. The μ_3-C_b–O_b distance of 1.287 Å in **1** is longer than μ_3-C–O (1.190 Å) in $LiCo_3(CO)_{10}\cdot i\text{-}Pr_2O$.[20]

Table III. Selected Bond Distances (Å) and Angles (deg) for One (Molecule 1) of the Two Independent $[Co_3(CO)_9\{\mu_3\text{-}COSi(OCH_2CH_2)_3N\}]$ Molecules

Si1–N1	2.010(5)	Co1–Co2	2.475(1)
Co2–Co3	2.472(1)	Co3–Co1	2.488(1)
Si1–Ob	1.707(4)	Si1–O1	1.639(7)
Si1–O2	1.644(7)	Si1–O3	1.661(6)
Cb–Ob	1.287(10)	Cb–Co1	1.905(10)
Cb–Co2	1.937(8)	Cb–Co3	1.927(6)
Co1–C11	1.852(10)	Co1–C12	1.797(8)
Co1–C13	1.819(8)	Co2–C21	1.876(11)
Co2–C22	1.781(10)	Co2–C23	1.798(10)
Co3–C31	1.834(10)	Co3–C32	1.776(13)
Co3–C33	1.799(9)		
N1–Si1–Ob	177.2(4)	Si1–Ob–Cb	134.4(5)
Si1–N1–C4	106.8(4)	Si1–N1–C5	105.2(5)
Si1–N1–C6	105.4(5)	N1–Si1–O1	85.4(3)
N1–Si1–O2	87.0(3)	N1–Si1–O3	86.1(3)
O1–Si1–O2	119.3(3)	O2–Si1–O3	120.2(4)
O3–Si1–O1	119.2(3)	Ob–Si1–O1	91.8(3)
Ob–Si1–O2	94.3(3)	Ob–Si1–O3	95.3(3)
Ob–Cb–Co1	137.4(4)	Ob–Cb–Co2	126.6(6)
Ob–Cb–Co3	131.1(6)	Co2–Co1–Co3	59.7(0)
Co3–Co2–Co1	60.4(0)	Co1–Co3–Co2	59.9(0)

In contrast to the structure of $LiCo_3(CO)_{10}\cdot i\text{-}Pr_2O$, which contains one μ_3-CO, three μ_2-CO, and six terminal CO groups,[20] the $Co_3(CO)_{10}$ fragment in **1** consists of one μ_3-CO and nine terminal CO groups coordinated to an almost regular Co_3 triangle. The geometric values for this fragment are comparable with those observed for other (oxymethylidyne)tricobalt clusters.[21] Among nine terminal CO groups, three axial groups have weaker bonding interaction with metal centers than six equatorial groups, as indicated by the average Co–CO(axial) and Co–CO(equatorial) distances of 1.854 and 1.795 Å, respectively. The structural entity of **1** may not be rigid in solution phase at ambient temperature since the $^{13}C\{^1H\}$-NMR spectrum in CD_3CN reveals only one signal at 203.1 ppm in the region of terminal CO ligands.[22]

In summary, a novel (tricobaltiomethoxy)silatrane has been prepared via oxidative addition of 1-hydrosilatrane to $Co_2(CO)_8$ and characterized, and the X-ray structure of $[Co_3(CO)_9\{\mu_3\text{-}COSi(OCH_2CH_2)_3N\}]$ has been determined. The possible use of relatively labile axial CO groups of the foregoing silatrane in preparing new derivatives is under search in our laboratory.

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Supplementary Material Available: Complete listings of thermal parameters, atomic parameters of the H atoms, and bond distances and angles for **1** (8 pages). Ordering information is given on any current masthead page.

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(17) The detection of a solution ^{29}Si NMR signal was hampered by a broad hump around −104 ppm caused by the probe and NMR tube glassware.

(18) The silatranyl cation is an unknown species, but its possible involvement or presence in reaction courses has been observed in our laboratory. Semiempirical calculations (MNDO and PM3) on this cation support its existance: Kim, J.; Shin, H. C.; Lee, Y. S.; Do, Y.; Knobler, C. B.; Hawthorne, M. F. Manuscript in preparation.

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(22) The ^{13}C NMR signal for μ_3-CO of **1** was not observed within the region of +498 to −11 ppm. The following parameters were adopted for the measurement of the spectrum: pulse repetition rate, 0.21 Hz; spectral bandwidth, 38 462 Hz; data words, 32 668; transmitter offset, 14 288 Hz.