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Crystal structures of cycloheteropentasilanes $(\eta^5\text{-Cp})_2\text{Ti}(\text{SiPh}_2)_5$ and $\text{O}(\text{SiPh}_2)_5$

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Abstract

An X-ray diffraction study of two heterocyclic polysilanes $(\eta^5\text{-Cp})_2\text{Ti}(\text{SiPh}_2)_5$ and $\text{O}(\text{SiPh}_2)_5$ has been carried out. In the titanasilane cycle the relevant bond lengths and angles are as follows: Ti–Si 2.755(7) and 2.765(8) Å, Si–Si 2.40–2.45(3) Å, SiTiSi 99.1(2)°, TiSiSi and SiSiSi 114.4–118.3(3)°; the cycle has a chair conformation and torsion angles of 43–59°. The geometric parameters of the oxasilane cycle are: Si–O 1.643 and 1.644(3) Å, Si–Si 2.369–2.388(2) Å, SiOSi 145.7(2)°, OSiSi and SiSiSi 106.5–109.3(1)°; the cycle has a boat conformation.

Introduction

At present cyclic organopolysilanes are being extensively studied which, in particular, is because their unusual electronic structure differing greatly from that of other saturated organic or organoelement compounds [1,2]. As a result of the one-electron reduction some cyclosilanes form stable anion-radicals with an unpaired electron delocalized in the cycle at low temperatures [1,3]. In addition to homocyclic compounds, heterocyclic polysilanes $\text{X}_m(\text{SiR}_2)_n$ involving non-metals $\text{X} = \text{O}, \text{S}, \text{N}, \text{P}, \text{B}$ [1,4,5] or metals $\text{X} = \text{Ge}, \text{Sn}, \text{Pt}, \text{Ti}$ [6–8] have also been prepared. Recently it was shown that in contrast to other heterocycles, such as oxygen-containing ones, heterocyclosilanes involving the metal atoms with variable valence (e.g. Ti, or Zr) can be readily reduced by sodium in THF to give extremely stable anion-radicals [9]. In these species the metal atom also takes part in the delocalization of an unpaired electron. Thus the interaction of Si atoms with this M atom may be somewhat different from the usual M–Si interaction which in turn is reflected in some unique features of the heterocycle geometry. We intend to study the structure of heterocyclosilane anion-radicals, so the present paper is the first step towards this and presents the results of an X-ray diffraction study of $(\eta^5\text{-Cp})_2\text{Ti}(\text{SiPh}_2)_5$ (I) and

$O(\text{SiPh}_2)_5$ (II) * carried out to elucidate the geometrical peculiarities of these 6-membered polysilane heterocycles.

Discussion

The structures I and II are depicted in Figs. 1 and 2 respectively, bond lengths and bond angles in the heterocycles are given in Figs. 3 and 4.

Although the oxidation state of the Ti atom in the previously studied complex $(\eta^5\text{-Cp})_2\text{Ti}(\text{SiMe}_3)\text{Cl}$ [11] is the same as in I, the Si–Ti distance 2.67(1) Å in the former is significantly shorter than the corresponding distances 2.755(7) and 2.765(8) Å found in I. Elongation of the Si–M bonds on going to di- and polysilane ligands has been also observed in iron silanes: an increase in the length (from 2 to 7 Si atoms) of the silane chain attached to the iron-containing moiety is accompanied by elongation of the Si–Fe bond from 2.328(1) to 2.363(1) Å [12,13]. Interestingly, in the complex with two $\eta^5\text{-Cp}(\text{CO})_2\text{Fe}$ groups [12] the Si–Fe bond which links the Fe atom directly with the pentasilane cycle is longer than the other Si–Fe bonds (2.363(1) and 2.350(2) Å). The role of homoatomic bonds in the above-mentioned complexes should be emphasized; substitution of the terminal Si atom in $(\eta^5\text{-$

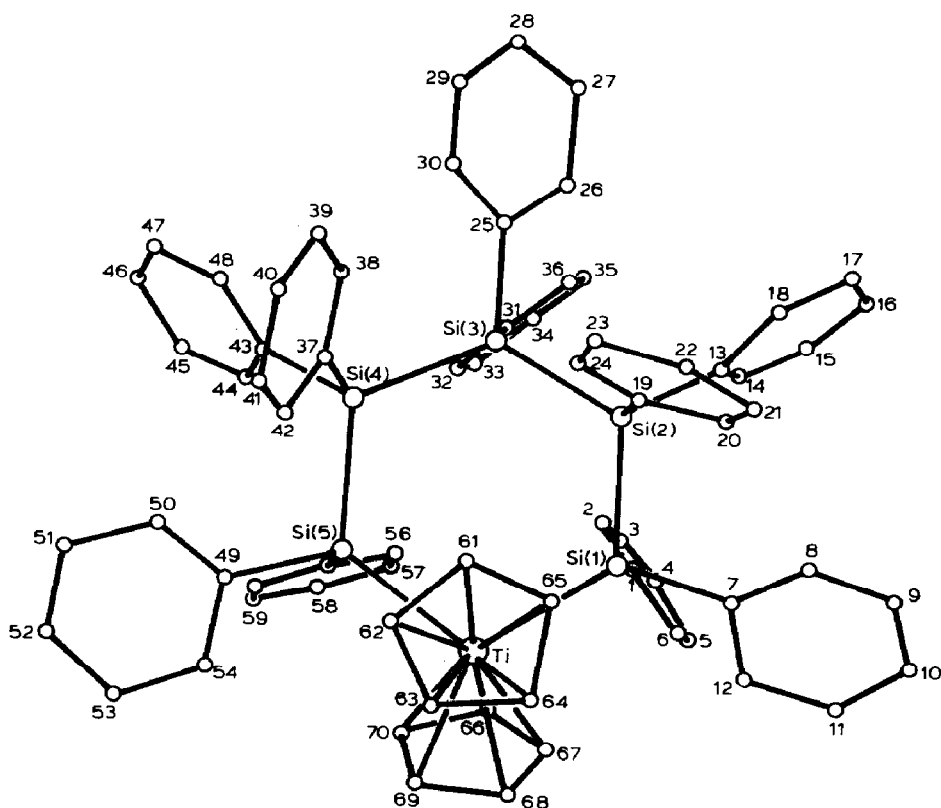


Fig. 1. Structure of I. H atoms are omitted.

* The structure of II has been studied earlier [10], however our results are more accurate.

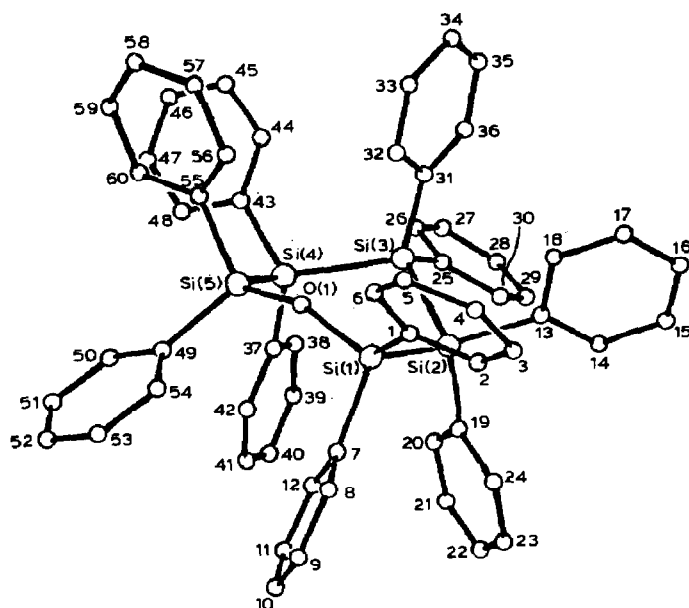


Fig. 2. Structure of II. H atoms are omitted.

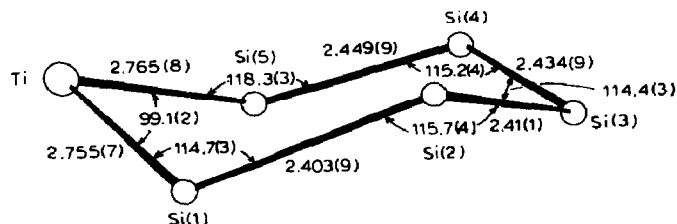


Fig. 3. Heterocycle I with the bond lengths and bond angles.

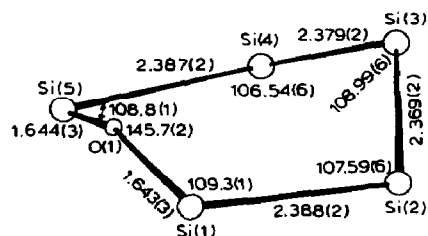


Fig. 4. Heterocycle II with the bond lengths and bond angles.

$\text{Cp}(\text{CO})_2\text{FeSiMe}_2\text{SiPh}_3$ by the Ge atom results in shortening of the Si-Fe bond by 0.02 Å [13].

The similar influence of the metal atom on the Si-Si bonds in a silanyl ligand should also be noted; these bonds are elongated though to a lesser extent. However, in this respect homoatomic bonds in the ligands are not exceptional; attachment of germysilanyl ligand to the Fe atom led to an elongation of the Si-Ge bond similar to that observed for Si-Si bonds (viz. by 0.02 Å) [13]. The average Si-Si bond length of 2.424 Å in the structure I exceeds the generally accepted value of 2.40 Å [14,15] in the perphenylated cyclopenta- and cyclohexasilanes by the same amount,

viz., ca. 0.02 Å (individual Si–Si distance in I vary considerably, which is probably because of the low accuracy of the structure).

The question of whether or not elongation of the M–Si and Si–Si bonds in the metallapolysilanes as compared with the isolated bonds of these types are characteristic features of these particular compounds is answered by comparison of the geometrical parameters of various heterocycles with a similar “chemical topology”. The following bond lengths are found in the heterocycle of the sulfide analogue of I, $(\eta^5\text{-Cp})_2\text{TiS}_5$ (III): Ti–S 2.422(1) and 2.428(1) Å, S–S 2.056–2.067(1) Å [16]. In the structure of $(\eta^5\text{-Cp})_2\text{Ti}(\text{SMe})_2$ [17] the isolated Ti–S bond length is equal to 2.403(1) Å and the usual value of the isolated S–S bond length is 2.02–2.03 Å [18,19]. Thus bond elongation is also observed in the heterocycle III, although for Ti–S in this case it is not so pronounced as it is for Ti–Si in I. It is noteworthy that the Ti–S bonds in the eight-membered cycle Ti_2S_6 [20] have approximately the same length as those in III. Other geometrical parameters of the heterocycles I and III are also similar: the SiSiSi, TiSiSi, SSS and TiSS bond angles are somewhat larger than the usual values (taking into account the high variability of the SiSiSi angles, the average value of 113.8° [15] in $(\text{SiPh}_2)_6$ should be regarded as standard), the Cp^*TiCp^* and SiTiSi (STiS) angles are 130, 99 and 134, 95° in I and III respectively, both cycles TiSi_5 and TiS_5 have the chair conformation.

The perphenylpentasilane chain of the six-membered heterocycle II (Fig. 4) is the same as in I, however, in contrast to I it has a non-metallic oxygen atom. The typical values of the Si–O bond lengths and the SiOSi bond angles in the siloxane fragments of unstrained cycles are 1.60–1.62 Å and 145–155°, respectively [21]. Hence the Si–O bond length in II exceeds the usual value by ca. 0.03 Å. The influence of the O heteroatom on the Si–Si bonds is opposite to that by the metal atom; the average Si–Si distance in II (2.381 Å) is shorter than that in $(\text{SiPh}_2)_5$ and $(\text{SiPh}_2)_6$ by 0.02 Å [14,15], which is probably due to the high electronegativity of the O atom. It is of little value to compare the overall conformations of the heterocycles I and II because (i) the large differences between the two bond lengths involving the heteroatoms in these cycles and (ii) the differences between the volumes of the substituents bring about the large variations in the intramolecular non-bonded interactions controlling these conformations. The SiSiSiSi torsion angles in cycles I and II are 40–50°; the average values are close to the observed torsion angle of 47.5° in the highly symmetrical $(\text{SiPh}_2)_6$ cycle [15].

Thus comparison of the structure of complex I with its various analogues suggests that the structural features observed in the case of interaction of the metal atom with the polysilane chain do not qualitatively differ from the effects of the interaction of this metal atom with other homoatomic chains or of other heteroatoms with the same polysilane grouping $(\text{SiPh}_2)_n$. However significant elongation of the Ti–Si bond in I may reflect, in principle, the unique features of the electronic structure of the titanasilane heterocycle.

Experimental

Crystal data for I. Dark-green crystals, monoclinic, at -120°C a 13.91(2), b 15.42(2), c 17.51(1) Å, β 111.04(7)°, V 3505(6) Å³, d_{calc} 1.26 g·cm⁻³, $Z = 2$ ($\text{C}_{70}\text{H}_{69}\text{TiSi}_5 \cdot 3\text{C}_6\text{H}_6$), space group $P2_1$.

* Cp^* is the centroid of the Cp ring.

Table 1

Atomic coordinates ($\times 10^4$) for I

Atom	x	y	z
Ti	3709(3)	1500	4240(2)
Si(1)	3495(5)	798(5)	2741(4)
Si(2)	1864(5)	1076(5)	1675(4)
Si(3)	372(5)	559(5)	1929(4)
Si(4)	316(5)	977(5)	3253(4)
Si(5)	1949(5)	836(5)	4408(3)
C(1)	3710(15)	-392(14)	2692(12)
C(2)	3051(16)	-1036(15)	2539(13)
C(3)	3226(16)	-1907(16)	2502(13)
C(4)	4129(17)	-2174(16)	2644(14)
C(5)	4976(17)	-1635(17)	2861(14)
C(6)	4769(18)	-761(17)	2870(14)
C(7)	4443(16)	1271(15)	2294(12)
C(8)	4498(16)	959(17)	1562(13)
C(9)	5268(16)	1170(16)	1263(13)
C(10)	5968(17)	1761(16)	1656(13)
C(11)	5959(17)	2098(16)	2375(14)
C(12)	5263(16)	1865(15)	2733(13)
C(13)	1855(15)	555(14)	686(11)
C(14)	2049(18)	-281(17)	569(14)
C(15)	2092(18)	-646(17)	-130(14)
C(16)	1877(20)	-132(19)	-789(16)
C(17)	1528(18)	705(18)	-767(15)
C(18)	1514(17)	1080(18)	-39(14)
C(19)	1745(16)	2278(14)	1493(12)
C(20)	2385(16)	2738(15)	1144(13)
C(21)	2352(17)	3568(17)	948(14)
C(22)	1634(15)	4048(14)	1110(12)
C(23)	959(17)	3713(16)	1432(13)
C(24)	1080(17)	2837(16)	1635(13)
C(25)	-849(16)	908(17)	1077(13)
C(26)	-879(18)	1517(19)	476(14)
C(27)	-1836(17)	1709(18)	-240(14)
C(28)	-2700(16)	1275(16)	-227(14)
C(29)	-2649(17)	748(17)	371(13)
C(30)	-1805(16)	521(15)	982(13)
C(31)	246(16)	-583(16)	1754(12)
C(32)	444(17)	-1258(17)	2395(14)
C(33)	406(17)	-2116(17)	2242(13)
C(34)	168(18)	-2423(17)	1426(14)
C(35)	21(16)	-1825(16)	820(13)
C(36)	59(15)	-975(15)	985(12)
C(37)	-217(16)	2129(15)	3125(12)
C(38)	-980(16)	2425(15)	2452(13)
C(39)	-1389(17)	3227(16)	2354(13)
C(40)	-960(17)	3842(16)	2937(13)
C(41)	-172(19)	3590(18)	3614(15)
C(42)	227(16)	2763(16)	3712(13)
C(43)	-735(16)	319(14)	3494(12)
C(44)	-565(15)	-462(14)	3865(12)
C(45)	-1306(17)	-922(17)	4104(14)
C(46)	-2311(17)	-540(16)	3887(13)
C(47)	-2475(17)	230(17)	3509(14)
C(48)	-1708(17)	663(16)	3298(14)

Table 1 (continued)

Atom	x	y	z
C(49)	1664(17)	1342(16)	5298(13)
C(50)	719(16)	1334(16)	5349(13)
C(51)	530(17)	1637(19)	6053(14)
C(52)	1369(19)	1928(18)	6709(15)
C(53)	2365(17)	1959(16)	6705(13)
C(54)	2531(17)	1655(17)	5986(13)
C(55)	2102(14)	-312(14)	4699(11)
C(45)	2299(17)	-930(16)	4198(13)
C(57)	2461(19)	-1813(19)	4380(15)
C(58)	2340(20)	-2155(19)	5103(16)
C(59)	2119(18)	-1541(18)	5595(14)
C(60)	1984(18)	-692(18)	5411(15)
C(61)	2607(17)	2671(15)	3597(13)
C(62)	2888(17)	2775(16)	4443(13)
C(63)	3928(19)	2949(18)	4749(15)
C(64)	4338(18)	2864(17)	4111(14)
C(65)	3431(17)	2729(16)	3365(13)
C(66)	4573(17)	121(17)	4684(14)
C(67)	5240(16)	646(16)	4509(13)
C(68)	5453(16)	1360(17)	5026(14)
C(69)	4870(15)	1236(14)	5539(11)
C(70)	4319(17)	459(16)	5349(13)
C(71) ^a	2771(19)	3548(18)	8433(16)
C(72)	3675(19)	3881(20)	8443(16)
C(73)	4562(21)	3597(21)	8969(17)
C(74)	4549(24)	2866(23)	9476(19)
C(75)	3692(23)	2641(21)	9522(19)
C(76)	2723(21)	2913(20)	8933(17)
C(77)	5099(41)	-325(41)	7616(34)
C(78)	4873(42)	-623(44)	7900(35)
C(79)	4782(33)	-745(34)	8648(27)
C(80)	4313(30)	-42(29)	8897(24)
C(81)	4227(42)	662(42)	8395(33)
C(82)	4435(43)	385(43)	7750(37)
C(83)	1373(40)	5606(42)	2897(34)
C(84)	1393(34)	5437(34)	3671(28)
C(85)	2368(33)	5113(34)	3918(27)
C(86)	3029(35)	4950(35)	3487(28)
C(87)	2849(30)	5442(29)	2775(24)
C(88)	1849(30)	5670(31)	2505(25)

^a The C(71) ... C(88) atoms belong to the benzene molecules.

The unit cell parameters and intensities of 4196 independent reflections were measured with an automated Syntex $P2_1$ diffractometer at -120°C ($\lambda(\text{Mo-K}_\alpha)$, graphite monochromator, $\theta/2\theta$ scan, $\theta_{\text{max}} 22^\circ$). Because of its instability in air the crystal was sealed in a glass capillary. The structure was solved by direct methods and refined by a full-matrix least-squares technique anisotropically for the Ti and Si atoms and isotropically for the C atoms. The positions of the H atoms were calculated but were not refined. Final discrepancy factors are $R = 0.102$ ($R_w = 0.092$) for 2219 reflections with $I > 2\sigma(I)$. The atomic coordinates are listed in Table 1.

Table 2

Atomic coordinates ($\times 10^5$ for Si, $\times 10^4$ for O and C, $\times 10^3$ for H) for II

Atom	x	y	z
Si(1)	73947(7)	5483(9)	46375(6)
Si(2)	69242(6)	-9031(9)	40580(6)
Si(3)	74137(6)	-10766(9)	31139(6)
Si(4)	74503(6)	4762(9)	26078(6)
Si(5)	79548(7)	15735(9)	34992(6)
O(1)	7905(2)	1085(2)	4220(1)
C(1)	7933(2)	335(3)	5514(2)
C(2)	7681(3)	-247(4)	5946(2)
C(3)	8036(3)	-362(4)	6622(3)
C(4)	8646(3)	125(5)	6866(2)
C(5)	8907(3)	705(5)	6448(3)
C(6)	8554(3)	807(4)	5771(2)
C(7)	6682(2)	1418(3)	4702(2)
C(8)	6701(3)	1942(4)	5299(2)
C(9)	6201(3)	2640(4)	5326(3)
C(10)	5683(3)	2853(4)	4772(3)
C(11)	5644(3)	2348(4)	4189(3)
C(12)	6133(3)	1635(4)	4151(2)
C(13)	7150(2)	-2049(3)	4577(2)
C(14)	6657(2)	-2767(3)	4567(2)
C(15)	6827(3)	-3631(3)	4931(2)
C(16)	7501(3)	-3784(3)	5301(2)
C(17)	7998(3)	-3084(4)	5318(2)
C(18)	7832(2)	-2225(3)	4960(2)
C(19)	5937(2)	-812(3)	3824(2)
C(20)	5536(2)	-739(3)	3173(2)
C(21)	4810(2)	-713(4)	3041(2)
C(22)	4494(2)	-765(4)	3569(3)
C(23)	4875(3)	-805(4)	4217(3)
C(24)	5590(2)	-833(4)	4349(2)
C(25)	6925(2)	-1990(3)	2487(2)
C(26)	7062(2)	-2063(3)	1846(2)
C(27)	6715(3)	-2724(3)	1385(2)
C(28)	6225(3)	-3341(3)	1536(2)
C(29)	6088(3)	-3296(3)	2161(2)
C(30)	6431(2)	-2628(3)	2636(2)
C(31)	8326(2)	-1566(3)	3439(2)
C(32)	8835(2)	-1032(4)	3878(2)
C(33)	9516(3)	-1366(4)	4098(3)
C(34)	9699(3)	-2236(4)	3883(3)
C(35)	9214(3)	-2781(4)	3455(3)
C(36)	8525(3)	-2464(4)	3229(2)
C(37)	6520(2)	756(3)	2135(2)
C(38)	6194(3)	178(3)	1586(2)
C(39)	5506(3)	293(4)	1263(3)
C(40)	5122(3)	1008(4)	1463(3)
C(41)	5418(3)	1605(4)	1983(3)
C(42)	6108(2)	1486(3)	2320(2)
C(43)	8909(2)	1716(3)	3528(2)
C(44)	9442(2)	1386(4)	4048(2)
C(45)	10138(3)	1505(4)	4036(3)
C(46)	10303(3)	1962(5)	3495(3)
C(47)	9790(3)	2314(5)	2974(3)
C(48)	9094(3)	2197(4)	2984(2)

Table 2 (continued)

Atom	x	y	z
C(49)	7581(2)	2837(3)	3446(2)
C(50)	7396(3)	3378(4)	2860(2)
C(51)	7184(3)	4344(4)	2871(3)
C(52)	7151(4)	4764(4)	3462(3)
C(53)	7323(4)	4247(4)	4037(3)
C(54)	7540(3)	3290(4)	4034(3)
C(55)	8027(2)	503(3)	1990(2)
C(56)	8623(2)	-60(4)	2072(2)
C(57)	9070(3)	35(4)	1645(3)
C(58)	8925(3)	696(4)	1136(3)
C(59)	8345(3)	1257(4)	1041(2)
C(60)	7888(3)	1168(3)	1463(2)
C(61) ^a	242(4)	4147(6)	4913(4)
C(62) ^a	326(4)	5678(5)	5444(3)
C(63) ^a	-661(4)	5282(7)	4607(4)
H(2)	724(2)	-64(3)	576(2)
H(3)	787(2)	-80(3)	692(2)
H(4)	893(2)	11(3)	736(2)
H(5)	934(2)	105(3)	662(2)
H(6)	876(2)	127(3)	545(2)
H(8)	707(2)	171(3)	572(2)
H(9)	621(2)	301(3)	571(2)
H(10)	535(2)	335(3)	481(2)
H(11)	533(2)	251(3)	376(2)
H(12)	609(2)	127(3)	373(2)
H(14)	617(2)	-267(2)	431(2)
H(15)	644(2)	-406(3)	488(2)
H(16)	758(2)	-435(3)	553(2)
H(17)	849(2)	-318(2)	557(2)
H(18)	819(2)	-169(3)	499(2)
H(20)	574(2)	-72(2)	277(2)
H(21)	458(2)	-68(3)	261(2)
H(22)	404(2)	-77(3)	347(2)
H(23)	468(2)	-87(3)	464(2)
H(24)	584(2)	-91(3)	482(2)
H(26)	742(2)	-159(2)	174(2)
H(27)	675(2)	-275(3)	99(2)
H(28)	595(2)	-380(3)	116(2)
H(29)	573(2)	-375(3)	228(2)
H(30)	629(2)	-260(3)	307(2)
H(32)	871(2)	-38(3)	409(2)
H(33)	988(2)	-97(3)	438(2)
H(34)	1017(2)	-249(3)	404(2)
H(35)	932(2)	-337(3)	329(2)
H(36)	819(2)	-289(3)	290(2)
H(38)	646(2)	-31(2)	140(2)
H(39)	530(2)	-11(3)	88(2)
H(40)	462(2)	109(3)	122(2)
H(41)	521(2)	215(3)	213(2)
H(42)	629(2)	192(3)	268(2)
H(44)	932(2)	109(3)	444(2)
H(45)	1047(2)	123(3)	438(2)
H(46)	1074(2)	204(3)	349(2)
H(47)	993(2)	267(3)	260(2)
H(48)	868(2)	247(3)	259(2)

Table 2 (continued)

Atom	x	y	z
H(50)	739(2)	308(3)	244(2)
H(51)	708(2)	463(3)	249(2)
H(52)	697(2)	540(3)	348(2)
H(53)	732(2)	457(3)	440(2)
H(54)	770(2)	290(3)	444(2)
H(56)	877(2)	-55(3)	241(2)
H(57)	951(2)	-35(3)	173(2)
H(58)	923(2)	79(3)	87(2)
H(59)	825(2)	172(3)	71(2)
H(60)	741(2)	163(3)	138(2)
H(61) ^a	63(3)	378(4)	518(2)
H(62) ^a	-82(2)	433(3)	415(2)
H(63) ^a	-118(2)	548(3)	435(2)

^a The atoms of the benzene molecule.

Crystal data for II. Crystals are monoclinic, at 20 °C a 19.706(1), b 13.730(1), c 20.442(2) Å, β 104.036(8)°, V 5365.7(9) Å³, d_{calc} 1.216 g · cm⁻³, $Z = 4$ (C₆₀H₅₀OSi₅ · 0.5C₆H₆), space group $P2_1/c$.

Unit cell parameters and intensities of 8478 independent reflections were measured with an automated Hilger & Watts diffractometer at 20 °C (λ (Mo- K_{α}), graphite monochromator, $\theta/2\theta$ scan, θ_{max} 28°). The structure was solved by direct methods and refined anisotropically by a block-diagonal least-squares technique for non-hydrogen atoms. Hydrogen atoms were located in a difference Fourier synthesis and refined isotropically. Final discrepancy factors are $R = 0.058$ ($R_w = 0.050$) for 6119 reflections with $I > 2\sigma(I)$. Atomic coordinates are listed in Table 2.

All calculations were carried out with an "Eclipse S/200" computer using INEXTL program package [22].

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