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Reactions of oligosilyl potassium compounds with Group 4 metallocene dichlorides

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Abstract

By the reaction of silyl anions with Group 4 metallocene dichlorides, a number of new Group 4 oligosilyl compounds have been synthesized. The silyl residues employed include the popular tris(trimethylsilyl)silyl group and sterically more encumbered derivatives. For the cases of the tris(trimethylsilyl)silyl group both the mono and the disilylated products were obtained.

Keywords: Silylanions; Zirconocene; Hafnocene; Silyl transition metal compounds

1. Introduction

The chemistry of silyl transition metal compounds has received increased attention in recent times. While most of these studies are concerned with late metal chemistry, relatively few reports dealt with early metal silyl derivatives [1]. Among these Tilley and coworkers have explored the frontiers of the chemistry of Group 4 oligosilyl compounds [2,3]. In particular they proved the utility of these compounds in mechanistic studies [2] and examined a number of insertion reactions into the metal silicon bond [3].

Most of the compounds studied so far contain either the trimethylsilyl or the tris(trimethylsilyl)silyl group. The reason for this somewhat restricted concentration on these groups is the limited availability of different silylanions, which are the most convenient precursor molecules.

2. Results and discussion

Recently we have reported the synthesis of a number of oligosilyl potassium compounds by treatment of suitable precursors with potassium *tert*-butylate in THF [4]. As part of our ongoing studies of the reactivity of these compounds we are investigating their reactions with Group 4 halides [5]. As has been demonstrated already Group 4 metallocene halides react quite smoothly with silyl anions. Tilley et al. have shown that for the reactions of Cp_2ZrCl_2 and $Cp_2Zr(Cl)SiMe_3$ with $(Me_3Si)_3SiLi$ [6]. Therefore, we expected similar reactivity for the conversion of $(Me_3Si)_3SiK$ [4a] with Group 4 metallocene halides. A main interest of our study was to find out whether we could attach bulkier oligosilyl groups to the metallocenes or if even double silylation could be achieved.

Reactions of one equivalent of $(Me_3Si)_3SiK$ with Cp_2MCl_2 ($M = Zr$, Hf) indeed proceed completely clean and give the expected monosilylated compounds (**1**, **2**). The same was found true for the conversions of the bulkier silylanions: bis(trimethylsilyl)(*tert*-butyldimethylsilyl)silyl potassium [4b], bis(trimethylsilyl)(thexyldimethylsilyl)silyl potassium [4b] and {[tris(trimethylsilyl)silyl]dimethylsilyl}bis(trimethylsilyl)silyl potassium [4b] with zirconocene dichloride (Scheme 1). However, reaction with pentakis(trimethylsilyl)disilanyl potassium [4a] an even more sterically overcrowded anion was not possible. Although the typically red color of the silyl zirconium compounds was observed, the NMR spectroscopic analysis did not indicate a clean

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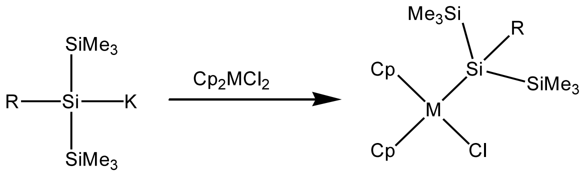


R = $SiMe_3$ M=Zr (**1**), M=Hf (**2**),
$SiMe_2CMe_3$ M=Zr (**3**)
$SiMe_2CMe_2CHMe_2$ M=Zr (**4**)
$SiMe_2Si(SiMe_3)_3$ M=Zr (**5**)

Scheme 1. Reactions of bulky oligosilanes with Group 4 metallocene dichlorides.

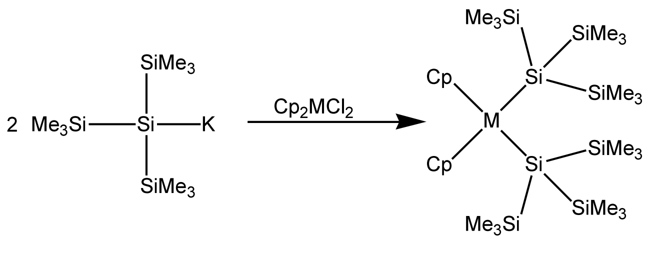
reaction but rather the formation of several different compounds including the respective hydrosilane.

Recent synthesis of zircono and hafnocenacyclopentasilanes by reaction of 1,4-dipotassiotetrasilanes with the respective Group 4 metallocene dichlorides [7] indicated that there should be no sterical reasons prohibiting double silylation. Therefore, in a second series of experiments we were studying reactions with two equivalents of tris(trimethylsilyl)silyl potassium. In a surprisingly smooth reaction we observed formation of double silylated compounds **6** and **7** (Scheme 2). Running the reactions in pentane instead of THF proved to be advantageous. Probably THF coordinates to the metal atoms and makes them less accessible for the second attack of the silylanion.

Compounds **4**, **6** and **7** were subjected to X-ray crystal structure analyses (Table 1, Figs. 1–3). Compared with the three known structures of the type $Cp_2Zr(Cl)SiR_3$ [R = $SiPh_3$ (Si–Zr = 2.814 Å) [8], R = $Si(SnMe_3)_3$ (Si–Zr = 2.765 and 2.773 Å) [9], R = $Si(SiMe_3)_3$ (Si–Zr = 2.833 Å) [9] the enhanced sterical demand of the $-Si(SiMe_3)_2(SiMe_2Thex)$ group in **4** is reflected by the longest Zr–Si bond length (Si–Zr = 2.853 Å) in the series.

Comparison of compound **6** with 2,2,5,5-tetrakis(trimethylsilyl)-3,3,4,4-tetramethylzirconocenacyclopentasilane [7] (Si–Zr = 2.8264 and 2.8497 Å) shows that the silicon metal bond length is elongated to a value of 2.8771 Å. To our knowledge this is the longest known Si–Zr distance for a neutral compound. Only the recently found ate-complex $Li[(Me_2N)_3Zr(SiPh_2^tBu)_2]$ [10] exhibits a longer bond (Si–Zr = 2.933 Å). The Si–Zr–Si angle of **6** (119.43°) is much larger than in the zirconocenacyclopentasilane (Si–Zr–Si = 97.70°), where the silyl ligands are connected within the framework of the five-membered ring. The rather large dihedral angle reflects the higher steric demand of the tris(trimethylsilyl)silyl group compared with the cyclopentadienyl ligand.

For the case of hafnium compound **7** the situation is quite similar. Again the silicon metal bond is rather long (Si–Hf = 2.850 Å). It is markedly longer than in 2,2,5,5-tetrakis(trimethylsilyl)-3,3,4,4-tetramethylhafnocenacyclopentasilane [7] (Si–Hf = 2.791 and 2.823 Å). Only the large Cp* group in $CpCp^*Hf(Cl)Si(SiMe_3)_3$ [11] causes an even longer bond (Si–Hf = 2.888 Å) which was attributed to the interaction between the Cp* ligand and the bulky silyl group.



M = Zr (**6**), Hf (**7**)

Scheme 2. Double silylation of zircono and hafnocene dichlorides.

3. Experimental

All reactions were carried out under an atmosphere of dry argon or nitrogen using either Schlenck techniques or a glovebox. Solvents were dried over sodium–potassium alloy and distilled freshly before use. Tetrakis(trimethylsilyl)silane [12], tris(trimethylsilyl)*tert*-butyldimethylsilylsilane [4a], tris(trimethylsilyl)thexyldimethylsilylsilane [4a], bis[tris(trimethylsilyl)silyl]dimethylsilane [4a] and the respective potassium compounds [4a] were prepared following published procedures. All other chemicals were used as received from chemical suppliers.

^1H- (300 MHz), ^{13}C- (75.4 MHz), and ^{29}Si-NMR (59.3 MHz) spectra were recorded either on a Bruker MSL 300 or on a Varian INOVA 300. Samples for ^{29}Si spectra were either dissolved in C_6D_6 or in cases of reaction samples measured with a D_2O capillary in order to provide an external lock frequency signal. To compensate for the low isotopic abundance of ^{29}Si the INEPT pulse sequence was used for the amplification of the signal. The completeness of the reactions were controlled by NMR. Mass spectra were obtained on a Kratos Profile instrument. Elemental analyses of were carried out using a Heraeus elementar vario EL apparatus. For X-ray structure analysis the crystals were mounted onto the tip of a glass fiber, and the data collection was performed with a Bruker-AXS SMART APEX CCD diffractometer. The data were reduced to F_o^2 and corrected for absorption effects with SAINT [13] and SADABS [14], respectively. The structures were solved by direct methods and refined by full-matrix least-squares method (SHELXL97) [15]. All non hydrogen atoms were refined with anisotropic displacement parameters. All hydrogens were located in calculated positions to correspond to standard bond lengths and angles.

Table 1
Crystallographic data

	4	**6**	**7**
Empirical formula	$ZrClSi_4C_{24}H_{47}$	$ZrSi_8C_{28}H_{64}$	$HfSi_8C_{28}H_{64}$
M_w	574.65	716.73	804.00
Temperature (K)	293(2)	223(2)	223(2)
Wavelength (Å)	0.71073	0.71073	0.71073
Size (mm)	0.60 × 0.34 × 0.19	0.24 × 0.18 × 0.06	0.31 × 0.24 × 0.01
Crystal system	Monoclinic	Trigonal	Trigonal
Space group	$P2_1/c$	$P3_221$	$P3_121$
a (Å)	19.177(4)	9.7435(14)	9.7579(14)
b (Å)	10.419(2)	9.7435(14)	9.7579(14)
c (Å)	16.696(3)	38.439(8)	38.270(8)
α (°)	90	90	90
β (°)	114.83(3)	90	90
γ (°)	90	120	120
V (Å^3)	3027.6(10)	3160.3(9)	3155.7(9)
Z	4	3	3
ρ_{calc} (g cm^{-3})	1.261	1.130	1.269
Absorption coefficient (mm^{-1})	0.620	0.504	2.723
F(000)	1216	1152	1248
θ Range (°)	$2.28 < \theta < 24.71$	$1.59 < \theta < 23.26$	$1.60 < \theta < 24.73$
Reflections collected/unique	13 169/5002	13 362/3020	18 887/3347
Completeness to θ (%)	96.9	99.9	91.6
Absorption correction	Empirical	Empirical	Empirical
Data/restraints/parameters	5002/0/286	3020/0/178	3347/0/178
Goodness-of-fit on F^2	1.091	1.142	0.876
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0830$, $wR_2 = 0.2294$	$R_1 = 0.0325$, $wR_2 = 0.0742$	$R_1 = 0.0573$, $wR_2 = 0.1178$
R indices (all data)	$R_1 = 0.0864$, $wR_2 = 0.2308$	$R_1 = 0.0342$, $wR_2 = 0.0748$	$R_1 = 0.0737$, $wR_2 = 0.1213$
Extinction coefficient	None	0.0000(3)	0.0014(3)
Largest difference peak and hole (e Å^{-3})	1.158/−0.971	0.304/−0.423	1.008/−1.673

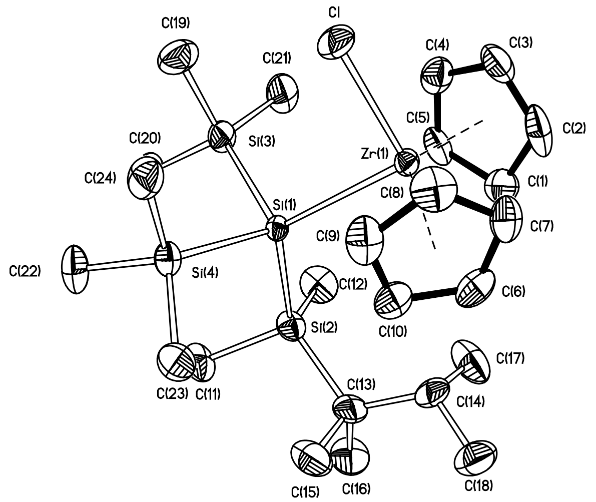


Fig. 1. The molecular structure and numbering of **4** with 30% probability thermal ellipsoids; all hydrogens have been removed for clarity; selected bond lengths (Å) and bond angels (°) with estimated standard deviations: Zr(1)–Cl, 2.405(3); Zr(1)–Si(1), 2.853(2); Si(1)–Si(2), 2.405(3); Si(1)–Si(3), 2.385(3); Si(1)–Si(4), 2.392(3); Cl–Zr(1)–Si(1), 95.93(9); Si(2)–Si(1)–Zr(1), 123.36(11); Si(3)–Si(1)–Zr(1), 110.75(11); Si(4)–Si(1)–Zr(1), 110.18(10); Si(3)–Si(1)–Si(4), 99.74(13); Si(3)–Si(1)–Si(2), 101.43(13); Si(4)–Si(1)–Si(2), 108.57(13).

3.1. Synthesis of tris(trimethylsilyl)silyl metallocenes **1** and **2**

At 0 °C a tris(trimethylsilyl)silyl potassium solution in pentane is added to a suspension of the metallocene dichloride (3 mmol an excess of 5%) in pentane. The reaction mixture was allowed to warm to room temperature (r.t.) and stirring was continued for an hour. Then the solvent was removed in vacuum, the residue dissolved in pentane, filtered, and the filtrate cooled to −30 °C to obtain crystals of **1** or **2**.

3.1.1. [Tris(timethylsilyl)silyl] zirconocenechloride (**1**)

Deep red crystals were obtained in 85% yield. ^{29}Si-NMR (pentane, D_2O, δ ppm): −6.1 (*Si*Me$_3$); −85.5 (*Si*Zr). ^{13}C (C_6D_6, δ ppm): 111.2 (*Cp*); 5.1 (Si*Me*$_3$). ^{1}H (C_6D_6, δ ppm): 5.97 (s, 10H); 0.40 (s, 27H).

3.1.2. [Tris(timethylsilyl)silyl] hafnocenechloride (**2**)

Deep red crystals were obtained in 83% yield. ^{29}Si-NMR (pentane, D_2O, δ ppm): −5.3 (*Si*Me$_3$); −79.7 (*Si*Hf). ^{13}C (C_6D_6, δ ppm): 110.4 (*Cp*); 5.4 (Si*Me*$_3$). ^{1}H (C_6D_6, δ ppm): 5.89 (s, 10H); 0.41 (s, 27H). MS: *m*/*z* (%): 592 (6) [M$^+$]; 577 (2) [M$^+$ −Me]; 345, (100) [M$^+$ −Si(SiMe$_3$)$_3$]; 247 (35) [M$^+$ −(Cp)$_2$HfCl)]; 73 (81) [SiMe$_3$]; 58 (16) [SiMe$_2$].

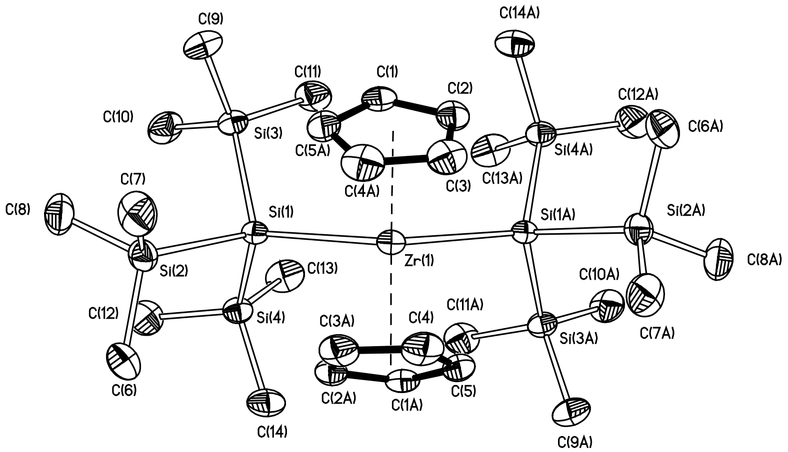


Fig. 2. The molecular structure and numbering of **6** with 30% probability thermal ellipsoids; all hydrogens have been removed for clarity; selected bond lengths (Å) and bond angels (°) with estimated standard deviations: Zr(1)–Si(1), 2.8771(10); Si(1)–Si(2), 2.3877(13); Si(1)–Si(3), 2.3780(14); Si(1)–Si(4), 2.3816(13); Si(1)–Zr(1)–Si(1A), 119.43(4); Si(3)–Si(1)–Si(4), 101.21(5); Si(3)–Si(1)–Si(2), 104.21(5); Si(4)–Si(1)–Si(2), 101.41(5); Si(3)–Si(1)–Zr(1), 116.30(4); Si(4)–Si(1)–Zr(1), 120.05(4); Si(2)–Si(1)–Zr(1), 111.50(4).

*3.2. Syntheses of silyl zirconocenes **3**, **4** and **5***

Zirconocene dichloride (3.0 mmol) and the respective silyl potassium compound (2.3 mmol) were dissolved in 5 ml toluene. The deep red solution was stirred at r.t. for 12 h. Then the solvent was removed in vacuum, the residue dissolved in pentane, filtered, and the filtrate cooled to −30 °C to obtain crystals.

*3.2.1. {(tert-Butyldimethylsilyl)[bis(trimethylsilyl)]-silyl} zirconocenechloride (**3**)*

Compound **3** was obtained starting from [(bis(trimethylsilyl)-*tert*-butyldimethylsilyl)silyl] potassium as deep red crystals in 60% yield. ^{29}Si-NMR (pentane, D$_2$O, δ ppm): 10.4 (SiMe$_2^t$Bu); −7.1 (SiMe$_3$); −85.6 (SiZr). ^{13}C (C$_6$D$_6$, δ ppm): 111.3 (Cp); 28.9 (CMe_3); 20.2 (CMe$_3$); 6.0 (SiMe_3); 1.8 (SiMe_2). ^{1}H (C$_6$D$_6$, δ ppm): 6.02 (s, 10H); 0.98 (s, 9H); 0.46 (s, 18H); 0.39 (s, 3H); 0.25 (s, 3H). Anal. Calc. for C$_{22}$H$_{43}$ClSi$_4$Zr: C, 48.34; H, 7.93. Found: C, 47.87; H, 7.78%.

*3.2.2. {[Bis(trimethylsilyl)](thexyldimethylsilyl)silyl} zirconocenechloride (**4**)*

Compound **4** was obtained starting from [(bis(trimethylsilyl)thexyldimethylsilyl)silyl] potassium as deep red crystals in 72% yield. ^{29}Si-NMR (pentane, D$_2$O, δ ppm): 13.8 (SiMe$_2$Thex); −6.9 (SiMe$_3$); −83.7(SiZr). ^{13}C (C$_6$D$_6$, δ ppm): 111.4 (Cp); 34.7 (CHMe$_2$); 26.6 (CMe$_2$); 22.2 (CMe_2); 19.1 (CHMe_2); 6.7 (SiMe_3); 4.5 (SiMe_2). ^{1}H (C$_6$D$_6$, δ ppm): 6.07 (s, 10H); 1.85 (m, 1H); 0.89 (d, 6H, $J = 6.6$ Hz); 0.51 (s, 18H); 0.44 (s, 6H); 0.28 (s, 3H); 0.27 (s, 3H). Anal. Calc. for C$_{24}$H$_{47}$ClSi$_4$Zr: C, 50.16; H, 8.24. Found: C, 50.19; H, 8.29%.

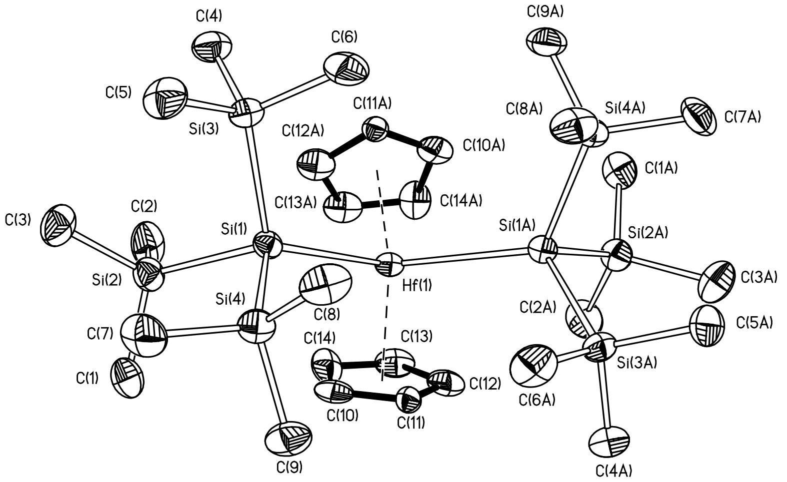


Fig. 3. The molecular structure and numbering of **7** with 30% probability thermal ellipsoids; all hydrogens have been removed for clarity; selected bond lengths (Å) and bond angels (°) with estimated standard deviations: Hf(1)–Si(1), 2.850(4); Si(1)–Si(2), 2.399(5); Si(1)–Si(3), 2.382(5); Si(1)–Si(4), 2.387(5); Si(1)–Hf(1)–Si(1A), 117.79(14); Si(3)–Si(1)–Si(4), 100.84(19); Si(3)–Si(1)–Si(2), 103.57(18); Si(4)–Si(1)–Si(2), 100.95(18);Si(2)–Si(1)–Hf(1), 111.04(16); Si(3)–Si(1)–Hf(1), 117.24(16); Si(4)–Si(1)–Hf(1), 120.73(16).

*3.2.3. 1,1,1,3,3-Pentakis(trimethylsilyl-2,2-dimethyltrisilanyl zirconocenechloride (**5**)*

Compound **5** was obtained starting from 1,1,1,3,3-pentakis(trimethylsilyl)-2,2-trimethyltrisilyl potassium as deep red crystals in 79% yield. ^{29}Si-NMR (pentane, D_2O, δ ppm): -6.9 (ZrSi(*Si*Me$_3$)$_2$); -9.0 (Si(*Si*Me$_3$)$_3$); -17.8 (ZrSi*Si*Me$_2$); -63.0 (*Si*Zr); -114.1 (*Si*(SiMe$_3$)$_3$). ^{13}C (C_6D_6, δ ppm): 111.8 (*Cp*); 9.0 (Si*Me*$_2$); 6.9 (Si(*Me*$_3$)$_2$); 5.3 (Si(*Me*$_3$)$_3$). ^{1}H (C_6D_6, δ ppm): 6.09 (s, 10H); 0.73 (s, 6H); 0.51 (s, 18H), 0.43 (s, 27H). Anal. Calc. for $C_{27}H_{61}ClSi_8Zr$: C, 43.99; H, 8.34. Found: C, 43.47; H, 8.16%.

*3.3. Synthesis of the disilyl metallocenes **6** and **7***

The respective metallocene dichloride and tris(trimethylsilyl)silyl potassium (two equivalents) were mixed and pentane was added. The deep red solution was stirred at r.t. for 12 h. Then the solvent was concentrated, the residue dissolved in toluene, filtered, and the filtrate cooled to -30 °C to obtain crystals.

*3.3.1. Bis[tris(trimethylsilyl)silyl] zirconocene (**6**)*

Deep red crystals were obtained in 85% yield. ^{29}Si-NMR (C_6D_6, δ ppm): -6.7 (*Si*Me$_3$); -83.9 (*Si*Zr). ^{13}C (C_6D_6, δ ppm): 109.1 (*Cp*); 6.6 (Si*Me*$_3$). ^{1}H (C_6D_6, δ ppm): 6.35 (s, 10H); 0.38 (s, 54H). Anal. Calc. for $C_{28}H_{64}Si_8Zr$: C, 46.92; H, 9.00. Found: C, 46.38; H, 9.04%.

*3.3.2. Bis[tris(trimethylsilyl)silyl] hafnocene (**7**)*

Red crystals were obtained in 81% yield. ^{29}Si-NMR (C_6D_6, δ ppm): -8.1 (*Si*Me$_3$); -82.4 (*Si*Hf). ^{13}C (C_6D_6, δ ppm): 109.2 (*Cp*); 6.5 (Si*Me*$_3$). ^{1}H (C_6D_6, δ ppm): 6.25 (s, 10H); 0.38 (s, 54H). Anal. Calc. for $C_{28}H_{64}Si_8Hf$: C, 41.83; H, 8.02. Found: C, 41.53; H, 8.03%.

*3.4. Synthesis of the potassium reagents (**8**)*

The required precursor compound and potassium *tert*-butylat (1.05 equivalents) were dissolved in THF. After 12 h stirring at r.t. the solvent was removed under reduced pressure. Usually, the residue was dissolved in pentane or toluene and used as such for the reactions with the metallocene dihalides.

4. Supplementary material

Crystallographic data (excluding structure factors) for the structure of **4**, **6** and **7** reported in this paper have been deposited with the Cambridge Crystallographic Data Center as supplementary publication number CCDC 192911 for **4**, CCDC 192913 for **6** and CCDC 192912 for **7**. Copies of the data can be obtained free of charge on application to The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK [Fax: (internat.) +44-1223-336033; e-mail: deposit@chemcrys.cam.ac.uk].

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