

Synthesis and characterization of monomeric siloxo palladium(II) complexes: crystal structure of $[\text{Pd}(\text{tmeda})(\text{C}_6\text{F}_5)(\text{OSiPh}_3)]$

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Abstract

Mononuclear palladium-hydroxo complexes of the type $[\text{Pd}(\text{N-N})(\text{C}_6\text{F}_5)(\text{OH})]$ [(N-N) = 2,2'-bipyridine (bipy), 4,4'-dimethyl-2,2'-bipyridine (Me_2bipy), or *N,N,N',N'*-tetramethylethylenediamine (tmeda)] react with silanols HOSiR_3 in toluene giving the corresponding siloxo complexes $[\text{Pd}(\text{N-N})(\text{C}_6\text{F}_5)(\text{OSiR}_3)]$. The X-ray crystal structure of $[\text{Pd}(\text{tmeda})(\text{C}_6\text{F}_5)(\text{OSiPh}_3)]$ has been determined. In one of the two molecules in the asymmetric unit there is an intramolecular interaction by phenyl–pentafluorophenyl π -stacking.

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Keywords: Palladium complexes; Siloxo complexes; Crystal structure

1. Introduction

Molecular complexes incorporating M–O–Si bonds (M = transition metal) are good models for metal complexes immobilized on silica and/or silicated surfaces, which have been commonly used in catalysis [1–3]. While complexes of early transition elements containing siloxy ligands are well known and characterized, the data on the late transition metal–siloxide complexes in molecular form are scarce [3]. The very recently reported mono-siloxides of Pt(II) and Pd(II) of the type $[\text{M}(\text{COD})(\text{R})(\text{OSiPh}_3)]$ (COD = 1,5-cyclooctadiene; R = Me, Et, Ph; M = Pt, Pd) have been proved to be useful precursors of metallic nanoclusters [4]. In this paper, we report the synthesis and characterization of a new series of stable siloxo-palladium complexes of the type $[\text{Pd}(\text{N-N})(\text{C}_6\text{F}_5)(\text{OSiR}_3)]$, including the first crystal structure of a siloxo-palladium complex [3D Search using the Cambridge Structural Database, July 2003 release].

2. Experimental

2.1. Materials and physical measurements

C, H, and N analyses were performed with a Carlo Erba model EA 1108 microanalyzer. Decomposition temperatures were determined with a Mettler TG-50 thermobalance at a heating rate of $5\text{ }^\circ\text{C min}^{-1}$ and the solid samples under nitrogen flow (100 ml min^{-1}). The ^1H and ^{19}F NMR spectra were recorded on a Bruker AC 200E or Varian Unity 300 spectrometer, using SiMe_4 and CFCl_3 as the standard, respectively. Infrared spectra were recorded on a Perkin–Elmer 1430 spectrophotometer using Nujol mulls between polyethylene sheets.

The starting complexes $[\text{Pd}(\text{N-N})(\text{C}_6\text{F}_5)(\text{OH})]$ [(N-N) = 2,2'-bipyridine (bipy), 4,4'-dimethyl-2,2'-bipyridine (Me_2bipy), or *N,N,N',N'*-tetramethylethylenediamine (tmeda)] were prepared by procedures described elsewhere [5]. The commercially available chemicals were purchased from Aldrich Chemical Co. and were used without further purification. Solvents were dried by the usual methods.

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2.2. Preparation of $[Pd(N-N)(C_6F_5)(OSiR_3)]$ [$N-N = bipy$, $R_3 = Ph_3$ (1), Et_3 (2) or Me_2Bu^t (3); $N-N = Me_2bipy$, $R_3 = Ph_3$ (4), Et_3 (5) or Me_2Bu^t (6); $N-N = tmeda$, $R_3 = Ph_3$ (7), Et_3 (8) or Me_2Bu^t (9)]

To a solution or suspension of $[Pd(N-N)(C_6F_5)(OH)]$ (0.245 mmol) in toluene (12 cm³) was added the corresponding R_3SiOH (0.245 mmol) and the resulting solution was refluxed for 4 h. Then the solvent was partially evaporated under reduced pressure. On addition of hexane the yellow complexes precipitated and were filtered off and air-dried.

2.2.1. $[Pd(bipy)(C_6F_5)(OSiPh_3)]$ (1)

Yield: 111 mg, 64%. Anal. Found: C, 57.6; H, 3.2; N, 4.1. Calc. for $C_{34}H_{23}F_5N_2OPdSi$: C, 57.9; H, 3.3; N, 4.0%. m.p.: 199 dec. IR (Nujol, cm⁻¹): 794 (Pd–C₆F₅). ¹H NMR ((CD₃)₂CO): δ 9.06 (dd, 1H, H _{α} , $J(H_{\alpha}H_{\beta}) = 5.4$ Hz, $J(H_{\alpha}H_{\gamma}) = 1.3$ Hz), 8.54 (d, 2H, H _{δ} + H _{δ'} , $J(H_{\gamma}H_{\delta}) = J(H_{\gamma}H_{\delta'}) = 7.6$ Hz), 8.28 (m, 2H, H _{γ} + H _{γ'}), 7.97 (d, 1H, H _{α'} , $J(H_{\alpha'}H_{\beta'}) = 5.6$ Hz), 7.74 (m, 1H, H _{β}), 7.5 (m, 7H, H _{β'} + H _{m} of Ph), 7.2 (m, 9H, H _{o} + H _{p} of Ph). ¹⁹F NMR ((CD₃)₂CO): δ -117.3 (d, 2F _{o} , $J_{om} = 22.6$ Hz), -162.3 (t, 1F _{p} , $J_{pm} = 19.2$ Hz), -164.0 (m, 2F _{m}).

2.2.2. $[Pd(bipy)(C_6F_5)(OSiEt_3)]$ (2)

Yield: 95 mg, 69%. Anal. Found: C, 46.9; H, 4.0; N, 4.8. Calc. for $C_{22}H_{23}F_5N_2OPdSi$: C, 47.1; H, 4.1; N, 5.0%. m.p.: 246 dec. IR (Nujol, cm⁻¹): 792 (Pd–C₆F₅). ¹H NMR ((CD₃)₂CO): δ 9.07 (d, 1H, H _{α} , $J(H_{\alpha}H_{\beta}) = 5.2$ Hz), 8.54 (d, 2H, H _{δ} + H _{δ'} , $J(H_{\gamma}H_{\delta}) = J(H_{\gamma}H_{\delta'}) = 8.0$ Hz), 8.29 (m, 2H, H _{γ} + H _{γ'}), 8.06 (d, 1H, H _{α'} , $J(H_{\alpha'}H_{\beta'}) = 5.2$ Hz), 7.88 (m, 1H, H _{β}), 7.52 (m, 1H, H _{β'}), 0.84 (t, 9H, CH₃ of Et, $J(HH) = 7.9$ Hz), 0.19 (q, 6H, CH₂ of Et, $J(HH) = 7.9$ Hz). ¹⁹F NMR ((CD₃)₂CO): δ -115.7 (d, 2F _{o} , $J_{om} = 21.4$ Hz), -162.5 (t, 1F _{p} , $J_{pm} = 20.3$ Hz), -164.6 (m, 2F _{m}).

2.2.3. $[Pd(bipy)(C_6F_5)(OSiMe_2Bu^t)]$ (3)

Yield: 81 mg, 59%. Anal. Found: C, 46.9; H, 4.0; N, 4.6. Calc. for $C_{22}H_{23}F_5N_2OPdSi$: C, 47.1; H, 4.1; N, 5.0%. m.p.: 223 dec. IR (Nujol, cm⁻¹): 788 (Pd–C₆F₅). ¹H NMR ((CD₃)₂CO): δ 9.02 (dd, 1H, H _{α} , $J(H_{\alpha}H_{\beta}) = 5.4$ Hz, $J(H_{\alpha}H_{\gamma}) = 1.3$ Hz), 8.54 (d, 2H, H _{δ} + H _{δ'} , $J(H_{\gamma}H_{\delta}) = J(H_{\gamma}H_{\delta'}) = 8.1$ Hz), 8.34 (m, 1H, H _{γ}), 8.24 (m, 1H, H _{γ'}), 8.06 (d, 1H, H _{α'} , $J(H_{\alpha'}H_{\beta'}) = 5.7$ Hz), 7.84 (m, 1H, H _{β}), 7.52 (m, 1H, H _{β'}), 0.83 (s, 9H, CH₃ of Bu^t), -0.42 (s, 6H, CH₃Si). ¹⁹F NMR ((CD₃)₂CO): δ -115.8 (d, 2F _{o} , $J_{om} = 22.6$ Hz), -162.4 (t, 1F _{p} , $J_{pm} = 20.3$ Hz), -164.5 (m, 2F _{m}).

2.2.4. $[Pd(Me_2bipy)(C_6F_5)(OSiPh_3)]$ (4)

Yield: 142 mg, 79%. Anal. Found: C, 58.8; H, 3.5; N, 3.6. Calc. for $C_{36}H_{27}F_5N_2OPdSi$: C, 59.0; H, 3.7; N,

3.8%. m.p.: 199 dec. IR (Nujol, cm⁻¹): 792 (Pd–C₆F₅). ¹H NMR ((CD₃)₂CO): δ 8.87 (d, 1H, H _{α} , $J(H_{\alpha}H_{\beta}) = 5.5$ Hz), 8.38 (s, 2H, H _{δ} + H _{δ'}), 7.77 (d, 1H, H _{α'} , $J(H_{\alpha'}H_{\beta'}) = 5.8$ Hz), 7.5 (m, 6H, H _{m} of Ph), 7.2 (m, 11H, H _{β} + H _{β'} + H _{o} + H _{p} of Ph), 2.57 (s, 3H, CH₃), 2.51 (s, 3H, CH₃). ¹⁹F NMR ((CD₃)₂CO): δ -117.1 (d, 2F _{o} , $J_{om} = 22.6$ Hz), -162.5 (t, 1F _{p} , $J_{pm} = 19.2$ Hz), -164.2 (m, 2F _{m}).

2.2.5. $[Pd(Me_2bipy)(C_6F_5)(OSiEt_3)]$ (5)

Yield: 100 mg, 69%. Anal. Found: C, 48.6; H, 4.5; N, 4.6. Calc. for $C_{24}H_{27}F_5N_2OPdSi$: C, 48.9; H, 4.6; N, 4.8%. m.p.: 237 dec. IR (Nujol, cm⁻¹): 790 (Pd–C₆F₅). ¹H NMR ((CD₃)₂CO): δ 8.88 (d, 1H, H _{α} , $J(H_{\alpha}H_{\beta}) = 5.5$ Hz), 8.37 (s, 2H, H _{δ} + H _{δ'}), 7.85 (d, 1H, H _{α'} , $J(H_{\alpha'}H_{\beta'}) = 5.8$ Hz), 7.69 (d, 1H, H _{β} , $J(H_{\alpha}H_{\beta}) = 5.5$ Hz), 7.32 (d, 1H, H _{β'} , $J(H_{\alpha'}H_{\beta'}) = 5.8$ Hz), 2.60 (s, 3H, CH₃ of Me₂bipy), 2.51 (s, 3H, CH₃ of Me₂bipy), 0.87 (t, 9H, CH₃ of Et, $J(HH) = 7.9$ Hz), 0.19 (q, 6H, CH₂ of Et, $J(HH) = 7.9$ Hz). ¹⁹F NMR ((CD₃)₂CO): δ -115.6 (d, 2F _{o} , $J_{om} = 22.6$ Hz), -162.6 (t, 1F _{p} , $J_{pm} = 19.2$ Hz), -164.6 (m, 2F _{m}).

2.2.6. $[Pd(Me_2bipy)(C_6F_5)(OSiMe_2Bu^t)]$ (6)

Yield: 114 mg, 79%. Anal. Found: C, 48.6; H, 4.4; N, 4.5. Calc. for $C_{24}H_{27}F_5N_2OPdSi$: C, 48.9; H, 4.6; N, 4.8%. m.p.: 239 dec. IR (Nujol, cm⁻¹): 792 (Pd–C₆F₅). ¹H NMR ((CD₃)₂CO): δ 8.91 (d, 1H, H _{α} , $J(H_{\alpha}H_{\beta}) = 5.5$ Hz), 8.36 (s, 2H, H _{δ} + H _{δ'}), 7.83 (d, 1H, H _{α'} , $J(H_{\alpha'}H_{\beta'}) = 5.8$ Hz), 7.69 (d, 1H, H _{β} , $J(H_{\alpha}H_{\beta}) = 5.5$ Hz), 7.31 (d, 1H, H _{β'} , $J(H_{\alpha'}H_{\beta'}) = 5.8$ Hz), 2.60 (s, 3H, CH₃ of Me₂bipy), 2.51 (s, 3H, CH₃ of Me₂bipy), 0.85 (s, 9H, CH₃ of Bu^t), -0.43 (s, 6H, CH₃Si). ¹⁹F NMR ((CD₃)₂CO): δ -115.7 (d, 2F _{o} , $J_{om} = 23.4$ Hz), -162.6 (t, 1F _{p} , $J_{pm} = 19.5$ Hz), -164.7 (m, 2F _{m}).

2.2.7. $[Pd(tmeda)(C_6F_5)(OSiPh_3)]$ (7)

Yield: 111 mg, 68%. Anal. Found: C, 54.0; H, 4.5; N, 4.0. Calc. for $C_{30}H_{31}F_5N_2OPdSi$: C, 54.2; H, 4.7; N, 4.2%. m.p.: 183 dec. IR (Nujol, cm⁻¹): 790 (Pd–C₆F₅). ¹H NMR ((CD₃)₂CO): δ 7.5 (m, 6H, H _{m} of Ph), 7.18 (m, 9H, H _{o} + H _{p} of Ph), 2.87 (m, 4H, CH₂), 2.68 (s, 6H, CH₃), 2.54 (s, 6H, CH₃). ¹⁹F NMR ((CD₃)₂CO): δ -117.8 (d, 2F _{o} , $J_{om} = 22.8$ Hz), -163.0 (t, 1F _{p} , $J_{pm} = 20.3$ Hz), -164.7 (m, 2F _{m}).

2.2.8. $[Pd(tmeda)(C_6F_5)(OSiEt_3)]$ (8)

Yield: 94 mg, 74%. Anal. Found: C, 41.4; H, 6.0; N, 5.3. Calc. for $C_{18}H_{31}F_5N_2OPdSi$: C, 41.5; H, 6.0; N, 5.4%. m.p.: 217 dec. IR (Nujol, cm⁻¹): 786 (Pd–C₆F₅). ¹H NMR ((CD₃)₂CO): δ 2.85 (m, 4H, CH₂ of tmeda), 2.66 (s, 6H, CH₃ of tmeda), 2.52 (s, 6H, CH₃ of tmeda), 0.78 (t, 9H, CH₃ of Et, $J(HH) = 7.9$ Hz), 0.01 (q, 6H, CH₂ of Et, $J(HH) = 7.9$ Hz). ¹⁹F NMR ((CD₃)₂CO): δ -116.0 (d, 2F _{o} , $J_{om} = 23.7$ Hz), -163.3 (t, 1F _{p} , $J_{pm} = 20.3$ Hz), -165.2 (m, 2F _{m}).

2.2.9. $[Pd(tmeda)(C_6F_5)(OSiMe_2Bu^t)]$ (**9**)

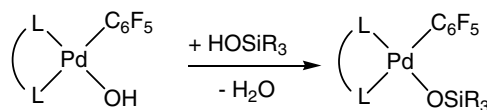
Yield: 82 mg, 64%. Anal. Found: C, 41.3; H, 5.7; N, 5.6. Calc. for $C_{18}H_{31}F_5N_2OPdSi$: C, 41.5; H, 6.0; N, 5.4%. m.p.: 169 dec. IR (Nujol, cm^{-1}): 788 (Pd– C_6F_5). 1H NMR ($(CD_3)_2CO$): δ 2.85 (m, 4H, CH_2 of tmeda), 2.67 (s, 6H, CH_3 of tmeda), 2.51 (s, 6H, CH_3 of tmeda), 0.70 (s, 9H, CH_3 of Bu^t), -0.58 (s, 6H, CH_3Si). ^{19}F NMR ($(CD_3)_2CO$): δ -116.2 (d, 2 F_o , $J_{om} = 23.4$ Hz), -163.3 (t, 1 F_p , $J_{pm} = 19.2$ Hz), -165.2 (m, 2 F_m).

2.3. Crystal structure determination of $[Pd(tmeda)(C_6F_5)(OSiPh_3)]$ (**7**)

Yellow prism of $0.72 \times 0.10 \times 0.10$ mm size grown by *n*-hexane/toluene diffusion, triclinic, space group $P\bar{1}$, $a = 14.9881(10)$ Å, $b = 15.6266(9)$ Å, $c = 16.4034(9)$ Å, $\alpha = 85.148(4)^\circ$, $\beta = 63.946(5)^\circ$, $\gamma = 61.553(4)^\circ$, $V = 2996.7(3)$ Å³, $Z = 4$, $2\theta_{max} = 50^\circ$, diffractometer Siemens P4, Mo K α ($\lambda = 0.71073$ Å), ω -scan, $T = 173(2)$ K, 15402 reflections collected of which 8399 were independent, $R_{int} = 0.0124$, direct primary solution and refinement on F^2 using SHELX-97 program [6], 771 refined parameters, the tmeda groups of both independent molecules are disordered over two sites [occupancy 82.8(7)/17.2(7)% and 76.9(9)/23.1(9)%], hydrogen atoms were refined using a riding model, $R_1 [I > 2\sigma(I)] = 0.0265$, wR_2 (all data) = 0.0707, max $\Delta/\sigma = 0.001$, max $\Delta\rho = 0.22$ e Å⁻³.

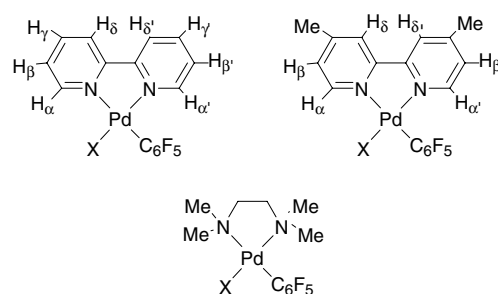
3. Results and discussion

The reaction of the monomeric hydroxo palladium complex $[Pd(N-N)(C_6F_5)(OH)]$ ($N-N =$ bipy, Me_2bipy , or tmeda) with silanol $HOSiR_3$ in toluene gives the corresponding siloxo complex $[Pd(N-N)(C_6F_5)(OSiR_3)]$ **1–9** (Scheme 1) in 59–79% yield. The acidic proton of the silanol is abstracted by the corresponding hydroxo-complex generating the anionic ligand R_3SiO^- with is subsequently trapped by the organometallic moiety $[Pd(N-N)(C_6F_5)]^+$ to form complexes **1–9** and the concomitant release of water. The structures were assigned on the basis of microanalytical, IR, and 1H and ^{19}F NMR data. Complexes **1–9** are all air-stable solids and the thermal analysis shows that they decompose above 169 °C in a dynamic N_2 atmosphere. The IR spectra show the characteristic absorptions of the C_6F_5 group [7] at 1630, 1490, 1450, 1050, 950 and a single band at ca. 800 cm^{-1} which is derived from the so-called X-sensitive mode [8] in C_6F_5 halogen molecules, which is characteristic of the presence of only one C_6F_5 group in the coordination sphere of the palladium atom and behaves like a $\nu(M-C)$ band [9–11]. The characteristic resonances of the neutral ligands [5,12–15] were observed in the 1H NMR spectra and the assignments presented in Section 2 are based on the atom numbering



	L ₂	R ₃
1	bipy	Ph ₃
2	bipy	Et ₃
3	bipy	Me ₂ Bu ^t
4	Me ₂ bipy	Ph ₃
5	Me ₂ bipy	Et ₃
6	Me ₂ bipy	Me ₂ Bu ^t
7	tmeda	Ph ₃
8	tmeda	Et ₃
9	tmeda	Me ₂ Bu ^t

Scheme 1.



Scheme 2.

given in Scheme 2. The low-field resonance (δ 8.8–9.1) in complexes **1–6** is assigned to the α -H atom [15]. Resonances of the methyl protons of the siloxy group in complexes **3**, **6** and **9** appears as a singlet at -0.42 , -0.43 and -0.58 , respectively [16]. The ^{19}F NMR spectra show the expected three resonances for 2 F_o :1 F_p :2 F_m of the C_6F_5 ligand.

The reactions of the triphenylsiloxo complex **7** with the unsaturated reagents CO, PhNCO and PhNCS were tried. When **7** was treated with CO, PhNCO and PhNCS at room temperature in toluene, the unchanged complex **7** was recovered, and the reaction of CO with **7** under refluxing toluene led to the formation of metallic palladium.

3.1. X-ray structure of $[Pd(tmeda)(C_6F_5)(OSiPh_3)]$ (**7**)

Fig. 1 shows the X-ray structure of complex **7**, with selected bond lengths and angles listed in Table 1. The crystal structure of compound **7** shows two independent molecules in the asymmetric unit with the palladium atom in a slightly distorted square planar geometry. The Pd–O bond length (1.986(3) Å) is in the typical range of Pd–O bonds reported for phenoxo palladium complexes [17,18] and slightly shorter than that found in

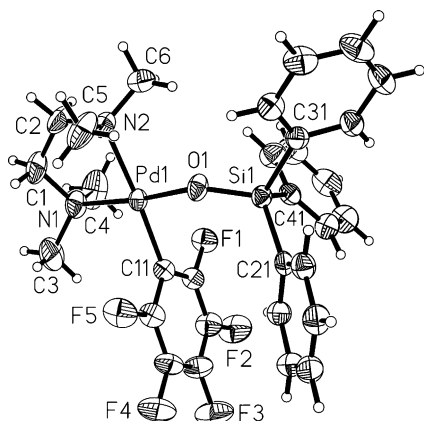


Fig. 1. Ellipsoid plot (30% probability) of compound [Pd(tmeda)-(C₆F₅)(OSiPh₃)] with the molecular labelling.

Table 1
Selected bond lengths (Å) and angles (°) for complex 7

Bond lengths		Bond angles	
<i>Molecule 1</i>			
Pd(1)–O(1)	1.986(3)	C(11)–Pd(1)–N(1)	93.2(2)
Pd(1)–C(11)	1.997(4)	C(11)–Pd(1)–N(2)	176.4(2)
Pd(1)–N(1)	2.055(4)	N(1)–Pd(1)–N(2)	84.5(2)
Pd(1)–N(2)	2.128(4)	O(1)–Pd(1)–N(2)	88.89(14)
Si(1)–O(1)	1.589(3)	O(1)–Pd(1)–N(1)	173.3(2)
		O(1)–Pd(1)–C(11)	93.3(2)
		Pd(1)–O(1)–Si(1)	137.9(2)
<i>Molecule 2</i>			
Pd(1A)–O(1A)	1.984(3)	C(11A)–Pd(1A)–N(1A)	94.1(2)
Pd(1A)–C(11A)	1.993(5)	C(11A)–Pd(1A)–N(2A)	172.9(2)
Pd(1A)–N(1A)	2.068(5)	N(1A)–Pd(1A)–N(2A)	84.9(2)
Pd(1A)–N(2A)	2.135(5)	O(1A)–Pd(1A)–N(2A)	89.6(2)
Si(1A)–O(1A)	1.586(4)	O(1A)–Pd(1A)–N(1A)	174.5(2)
		O(1A)–Pd(1A)–C(11A)	91.3(2)
		Pd(1A)–O(1)–Si(1A)	136.9(2)

[Pt(C₆F₅)₂(OC₆H₄NO₂-p)(CO)]⁻ (Pt–O = 2.070(4) Å) [19]. The Pd–O–Si angle of 137.9(2)° is in the range of those for siloxo complexes such as [Pt(COD)(R)(OSiPh₃)] (R = Ph, Et) [4]. The different Pd–N (Pd(1)–N(2), 2.128(4) Å; Pd(1)–N(1), 2.055(5) Å) distances are in agreement with the higher *trans* influence of the group C₆F₅ compared to OSiPh₃. The chelate angle N(1)–Pd(1)–N(2) (84.5(2)°) is similar to that found (83.9(2)°) in [Pd(tmeda)(C₆F₅)(CO₂Me)] [5]. The Pd–C₆F₅ bond length (1.997(4) Å) is in the range found in the literature for pentafluorophenyl–palladium complexes [15]. In one of the molecules in the asymmetric unit there is an intramolecular interaction by phenyl–pentafluorophenyl π -stacking [20,21]. The planes of both rings make an angle of 6.2°, with an interplanar distance (average of the two distances between the mean plane of one ring and the centroid of the other) of 3.51 Å, a centre-to-centre of 3.527 Å and a deviation of the centre–centre line of the perpendicular of the plane of 7.5° (pentafluorophenyl ring) and 11° (phenyl ring).

4. Supplementary material

Crystallographic data for the structural analysis of compound 7 have been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 230611. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or [www: http://www.ccdc.cam.ac.uk](http://www.ccdc.cam.ac.uk)).

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