Metallacarboranes and triple-decker complexes with the (C₄Me₄)Pt fragment

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ABSTRACT

Complex Cb^*PtCl_2 ($Cb^* = \eta\text{-}C_4Me_4$) reacts with the carborane anions $[7,8\text{-}C_2B_9H_{11}]^{2-}$ and $[9\text{-}SMe_2\text{-}7,8\text{-}C_2B_9H_{10}]^{-}$ giving platinacarboranes $Cb^*Pt(\eta\text{-}7,8\text{-}C_2B_9H_{11})$ (1) and $[Cb^*Pt(\eta\text{-}9\text{-}SMe_2\text{-}7,8\text{-}C_2B_9H_{10})]^{+}$ (2), respectively. Reactions of the $[Cb^*Pt]^{2+}$ fragment (as a labile nitromethane solvate) with the sandwich compounds $Cp^*Fe(\eta\text{-}C_5H_3Me_2BMe)$ and $Cp^*Rh(\eta^5\text{-}C_4H_4BPh)$ afford the triple-decker cations $[Cb^*Pt(\mu\text{-}\eta:\eta\text{-}C_5H_3Me_2BMe)FeCp^*]^{2+}$ (3) and $[Cb^*Pt(\mu\text{-}\eta^5:\eta^5\text{-}C_4H_4BPh)RhCp^*]^{2+}$ (4) with bridging boratabenzene and borole ligands. The structures of 1 and $3(CF_3SO_3)_2$ were determined by X-ray diffraction.

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1. Introduction

The fragments $[Cp^*M]^{2+}$ (M = Rh, Ir) are widely used in organometallic synthesis [1]. In particular, using these fragments a number of metallacarboranes [2–5] and triple-decker complexes [6–8] have been prepared. In contrast, chemistry of the related fragment $[Cb^*Pt]^{2+}$ ($Cb^* = \eta\text{-}C_4Me_4$) is notably less developed. In 1975, Canziani and Malatesta have described a two-step procedure for the preparation of the chloride complex Cb^*PtCl_2 from $(CO)_2PtCl_2$ and 2-butyne in 47% yield [9]. Moreto and Maitlis have synthesized this compound in 60% yield using more available acetonitrile complex $(MeCN)_2PtCl_2$ instead of $(CO)_2PtCl_2$ [10]. The chloride Cb^*PtCl_2 has been proven to be a useful synthon of the $[Cb^*Pt]^{2+}$ fragment [11]. In particular, Herberich et al. have synthesized the sandwich cations $[Cb^*PtCp]^{+}$ and $[Cb^*Pt(\eta\text{-}C_6Me_6)]^{2+}$ via the intermediate solvate complex $[Cb^*Pt(Me_2CO)_3]^{2+}$ [12]. Herein we report the synthesis and structures of the first Cb^*Pt-containing metallacarborane and triple-decker complexes.

2. Results and discussion

2.1. Synthesis

We found that reactions of Cb^*PtCl_2 with the carborane anions $[7,8\text{-}C_2B_9H_{11}]^{2-}$ and $[9\text{-}SMe_2\text{-}7,8\text{-}C_2B_9H_{10}]^{-}$ afford complexes 1

(71%) and 2 (17%) (Scheme 1).[1] The low yield of 2 may be caused by a side demethylation of the SMe_2 group [13].
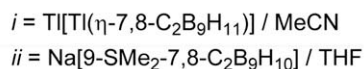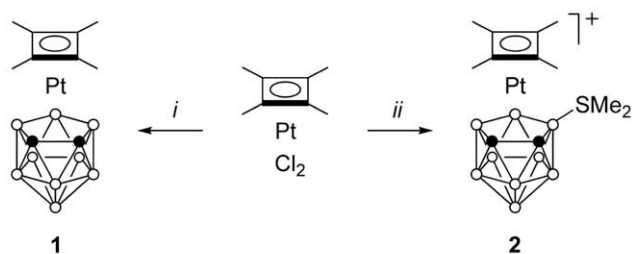
Electrophilic stacking of sandwich compounds with $[(\text{ring})M]^{n+}$ fragments is an effective method for the preparation of cationic triple-decker complexes [14]. In particular, complexes with bifacially-bonded boratabenzene [6,15] and borole [8,16] ligands have been prepared by this way. Using the stacking reaction of $Cp^*Fe(\eta\text{-}C_5H_3Me_2BMe)$ with the labile solvate $[Cb^*Pt(MeNO_2)_3]^{2+}$ we synthesized the μ-boratabenzene triple-decker complex 3 (17%) (Scheme 2). Similar reaction of $Cp^*Rh(\eta^5\text{-}C_4H_4BPh)$ affords the μ-borole cation 4 (64%). The low yield of 3 may be explained by easy oxidation of $Cp^*Fe(\eta\text{-}C_5H_3Me_2BMe)$ in contrast to $Cp^*Rh(\eta^5\text{-}C_4H_4BPh)$. The solvate complex $[Cb^*Pt(MeNO_2)_3]^{2+}$ was generated by treatment of Cb^*PtCl_2 with Ag^+ in nitromethane. It should be emphasized, that cations 3 and 4 are the first examples of stable Cb^*Pt-containing triple-decker complexes. Herberich et al. have described earlier the formation of the thermally unstable μ-cyclopentadienyl cation $[Cb^*Pt(\mu\text{-}\eta:\eta\text{-}Cp)FeCp^*]^{2+}$ which was detected only by ¹H NMR spectroscopy at 0 °C [14b]. Greater stability of triple-decker complexes with bridging boron heterocycles is a general tendency associated with favorable balance of donor and acceptor properties of these ligands.
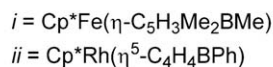
2.2. X-ray diffraction study
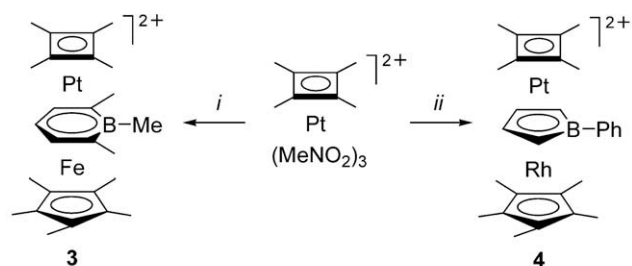
The structures of the metallacarborane complex 1 and the triple-decker cation 3 are shown in Figs. 1 and 2. Selected bond

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[1] All the cationic complexes described here were isolated as salts with the PF_6, BF_4, or CF_3SO_3 anions.

Scheme 1.

i = Tl[Tl(η-7,8-$C_2B_9H_{11}$)] / MeCN
ii = Na[9-SMe_2-7,8-$C_2B_9H_{10}$] / THF

Scheme 2.

i = Cp*Fe(η-$C_5H_3Me_2BMe$)
ii = Cp*Rh(η^5-C_4H_4BPh)

Fig. 2. Structure of cation **3**. Atoms are represented by 50% thermal ellipsoids.

lengths and angles are given in Tables. 1 and 2. The dihedral angle between the C_4 and C_2B_3 planes in **1** is equal to 4.7°. In the case of **3** the crystallographic cell contains two independent molecules; data for only one of them will be used in further discussion. Cation **3** is formed by three cyclic frames (C_4, C_5B, and C_2B_3); the dihedral angle C_4/C_5B (6.3°) is somewhat larger than C_5B/C_5 (2.7°). We have shown earlier, that the metal-to-ring Co$\cdots$$C_4$ distance in Cb*Co complexes is strongly dependent on donor–acceptor ability of other ligands at the Co atom [17]. In contrast, the Pt$\cdots$$C_4$ distance

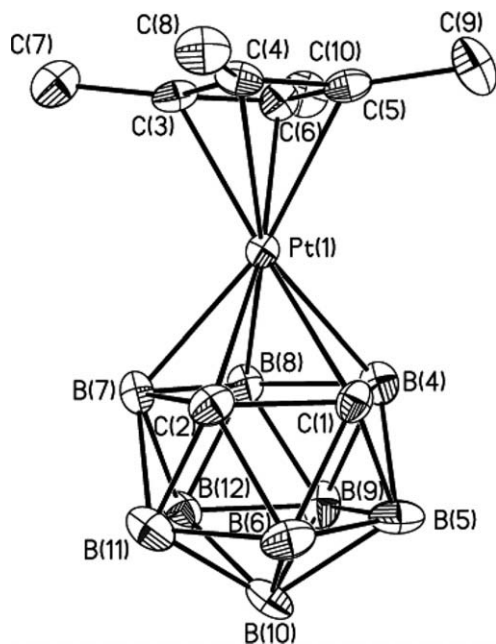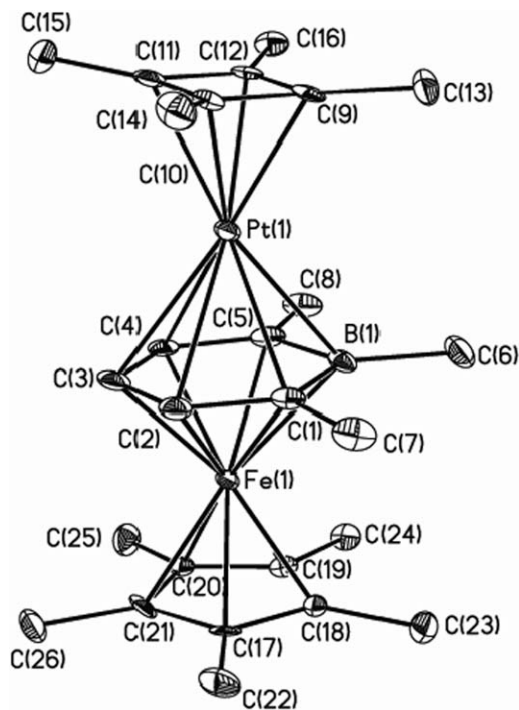**Fig. 1.** Structure of complex **1**. Atoms are represented by 50% thermal ellipsoids.

Table 1
Selected bond lengths (Å) and angles (°) for compound **1**

Pt(1)–C(1)	2.246(6)	C(3)–C(4)	1.457(9)
Pt(1)–C(2)	2.247(7)	C(4)–C(5)	1.471(10)
Pt(1)–C(3)	2.108(6)	C(5)–C(6)	1.466(9)
Pt(1)–C(4)	2.188(6)	C(3)–C(6)	1.467(10)
Pt(1)–C(5)	2.160(7)	C(1)–C(2)	1.622(10)
Pt(1)–C(6)	2.099(7)	C(1)–B(4)	1.736(10)
Pt(1)–B(4)	2.219(8)	C(2)–B(7)	1.737(10)
Pt(1)–B(7)	2.240(8)	B(4)–B(8)	1.827(12)
Pt(1)–B(8)	2.226(7)	B(7)–B(8)	1.844(10)
C(3)–C(4)–C(5)	89.8(5)	C(2)–B(7)–B(8)	104.7(5)
C(4)–C(5)–C(6)	90.0(6)	B(7)–B(8)–B(4)	106.1(5)
C(5)–C(6)–C(3)	89.6(5)	B(8)–B(4)–C(1)	104.7(5)
C(6)–C(3)–C(4)	90.6(6)	B(4)–C(1)–C(2)	112.6(6)
C(1)–C(2)–B(7)	111.8(5)		

Table 2
Selected bond lengths (Å) and angles (°) for cation **3**

Pt(1)–C(1)	2.364(4), 2.392(4)	Fe(1)–C(17)	2.058(4), 2.073(4)
Pt(1)–C(2)	2.457(4), 2.475(4)	Fe(1)–C(18)	2.073(4), 2.071(4)
Pt(1)–C(3)	2.489(5), 2.483(5)	Fe(1)–C(19)	2.071(4), 2.062(4)
Pt(1)–C(4)	2.462(4), 2.418(4)	Fe(1)–C(20)	2.068(4), 2.068(4)
Pt(1)–C(5)	2.373(4), 2.326(4)	Fe(1)–C(21)	2.068(4), 2.074(5)
Pt(1)–B(1)	2.376(5), 2.373(5)	C(1)–C(2)	1.436(6), 1.450(6)
Pt(1)–C(9)	2.116(4), 2.109(4)	C(2)–C(3)	1.454(6), 1.443(6)
Pt(1)–C(10)	2.147(4), 2.130(4)	C(3)–C(4)	1.447(6), 1.452(6)
Pt(1)–C(11)	2.179(5), 2.176(5)	C(4)–C(5)	1.445(6), 1.428(6)
Pt(1)–C(12)	2.134(4), 2.125(4)	B(1)–C(1)	1.557(6), 1.547(7)
Fe(1)–C(1)	2.136(4), 2.146(4)	B(1)–C(5)	1.564(7), 1.573(6)
Fe(1)–C(2)	2.079(4), 2.088(4)	C(9)–C(10)	1.459(6), 1.462(6)
Fe(1)–C(3)	2.059(5), 2.060(5)	C(10)–C(11)	1.460(6), 1.456(6)
Fe(1)–C(4)	2.070(5), 2.079(4)	C(11)–C(12)	1.459(6), 1.443(6)
Fe(1)–C(5)	2.140(4), 2.139(4)	C(9)–C(12)	1.480(6), 1.460(6)
Fe(1)–B(1)	2.218(5), 2.219(5)		
C(1)–C(2)–C(3)	120.9(4), 121.0(4)	B(1)–C(1)–C(2)	122.5(4), 122.1(4)
C(2)–C(3)–C(4)	120.8(4), 120.9(4)	C(9)–C(10)–C(11)	90.9(4), 89.6(4)
C(3)–C(4)–C(5)	121.5(4), 121.2(4)	C(10)–C(11)–C(12)	89.9(4), 90.5(4)
C(4)–C(5)–B(1)	121.5(4), 122.0(4)	C(11)–C(12)–C(9)	90.1(3), 90.2(4)
C(1)–B(1)–C(5)	112.8(4), 112.8(4)	C(12)–C(9)–C(10)	89.1(4), 89.6(4)

Table 3
Crystallographic data and structure refinement parameters for **1** and **3**(CF$_3$SO$_3$)$_2$

Compound	**1**	**3**(CF$_3$SO$_3$)$_2$
Empirical formula	C$_{10}$H$_{23}$B$_9$Pt	C$_{28}$H$_{36}$BF$_6$FeO$_6$PtS$_2$
Molecular weight	435.66	908.44
Crystal colour, habit	White prism	Yellow prism
Crystal size (mm)	0.40 × 0.25 × 0.20	0.45 × 0.03 × 0.03
Crystal system	Monoclinic	Triclinic
Space group	$P2_1/n$	$P\bar{1}$
a (Å)	7.0157(15)	8.5609(10)
b (Å)	17.601(4)	17.541(2)
c (Å)	13.152(3)	21.572(3)
α (°)	90	97.264(2)
β (°)	101.594(18)	90.184(2)
γ (°)	90	91.228(2)
V (Å^3)	1590.9(6)	3212.6(7)
Z	4	4
D_{calc} (g cm^{-3})	1.819	1.878
T (K)	163	100
θ Range (°)	3.18–26.00	0.95–29.00
μ (cm^{-1})	87.97	50.06
Absorption correction		Multi-scan
T_{max}/T_{min}		0.8643/0.2116
Collected reflections	6317	60184
Independent reflections (R_{int})	2978 (0.0945)	17059 (0.0775)
Observed reflections ($I > 2\sigma(I)$)	2486	12220
Parameters	181	835
R_1 (on F for observed reflections)	0.0420	0.0352
wR_2 (on F^2 for all reflections)	0.1075	0.0662
Weighting scheme	$w^1 = \sigma^2(F_o^2) + (aP)^2$, where $P = 1/3(F_o^2 + 2F_c^2)$	
a	0.0652	0.0105
$F(000)$	824	1788
Goodness-of-fit	0.988	0.952
Largest difference in peak and hole (e Å^{-3})	2.154 and −3.820	1.389 and −1.185

is only weakly dependent on auxiliary ligands. For example, these distances in **1** (1.868 Å) and **3** (1.875 Å) are very close to those in Cb*PtCl$_2$ (1.869 Å [18]) and [Cb*Pt(η-C$_6$Me$_6$)]$^{2+}$ (1.873 Å [12]).

Usually, M—B bonds are considerably longer than M—C bonds [15b] in accordance with larger covalent radius of boron versus carbon. However, in the triple-decker cation **3** the Pt—B(1) bond (2.376 Å) is shorter than Pt—C(3) (2.489 Å). In the metallacarborane complex **1** the Pt—B bonds (2.219–2.240 Å, av. 2.23 Å) are also shorter than the Pt—C bonds (2.246–2.247 Å, av. 2.25 Å). Similar pattern has been observed earlier for other dicarbollide complexes of platinum, e.g. (PEt$_3$)$_2$Pt(η-7,8-C$_2$B$_9$H$_{11}$) and (cod)Pt(η-7,8-C$_2$B$_9$H$_{11}$) [19,20].

3. Conclusion

The chloride Cb*PtCl$_2$ was shown to be very useful for the synthesis of complexes with boron-containing ligands. Its reactions with carborane anions afford (cyclobutadiene)platinacarboranes. Electrophilic stacking of sandwich compounds with [Cb*Pt(Me-NO$_2$)$_3$]$^{2+}$ (generated by treatment of Cb*PtCl$_2$ with Ag$^+$) gives platinum-containing triple-decker complexes.

4. Experimental

4.1. General

All reactions were carried out under an inert atmosphere in dry solvents. The isolation of products was conducted in air. The starting materials Cb*PtCl$_2$ [10], Tl[Tl(η-7,8-C$_2$B$_9$H$_{11}$)] [21], Na[9-SMe$_2$-7,8-C$_2$B$_9$H$_{10}$] [22], Cp*Fe(η-C$_5$H$_3$Me$_2$BMe) [6,23], Cp*Rh(η^5-C$_4$H$_4$BPh) [16e] and AgBF$_4$ · 3dioxane [24] were prepared as described in the literature. ^{1}H and ^{11}B{^{1}H} NMR spectra (δ in ppm) were recorded on a Bruker Avance-400 spectrometer (^{1}H 400.13;

^{11}B 128.38 MHz) relative to residual protons of the solvents (^{1}H) or BF$_3$ · Et$_2$O (^{11}B, external standard).

4.2. Synthesis of Cb*Pt(η-7,8-C$_2$B$_9$H$_{11}$) (**1**)

Acetonitrile (1 ml) was added to a mixture of complex Cb*PtCl$_2$ (60 mg, 0.160 mmol) and Tl[Tl(η-7,8-C$_2$B$_9$H$_{11}$)] (104 mg, 0.192 mmol). The reaction mixture was stirred for 24 h and eluted through a layer of Al$_2$O$_3$ (4 cm) by acetone. The solvent was removed in vacuo and the residue was washed by petroleum ether giving complex **1** as a white solid (50 mg, 71%). Anal. Calc. for C$_{10}$H$_{23}$B$_9$Pt: C, 27.57; H, 5.32; B, 22.33. Found: C, 27.35; H, 5.22; B, 22.43%. ^{1}H NMR (acetone-d_6): δ = 2.44 (s, 12 H, Cb*, J(PtH) 12), 3.49 (s, 2H, CH-cage, J(PtH) 22). ^{11}B{^{1}H} NMR (acetone-d_6): δ = −24.1 (s, 1B), −20.9 (s, 2B), −12.6 (s, 2B, J(PtB) 101), −10.9 (s, 2B), −9.7 (s, 1B), −2.1 (s, 1B, J(PtB) 148).

4.3. Synthesis of [Cb*Pt(η-9-SMe$_2$-7,8-C$_2$B$_9$H$_{10}$)]PF$_6$ (**2**PF$_6$)

A solution of Na[9-SMe$_2$-7,8-C$_2$B$_9$H$_{10}$] in THF (2 ml of a 0.085 M solution, 0.170 mmol) was added to a suspension of complex Cb*PtCl$_2$ (52 mg, 0.139 mmol) in THF (4 ml). The reaction mixture was stirred for 24 h. The solvent was removed in vacuo and the solid residue was extracted with water. Then an excess of an aqueous NH$_4$PF$_6$ solution was added. The white precipitate was filtered off, washed with water, and dried in vacuo. Complex **2**PF$_6$ was obtained as a white solid (15 mg, 17%). Anal. Calc. for C$_{12}$H$_{28}$B$_9$F$_6$PPtS: C, 22.46; H, 4.40; B, 15.16. Found: C, 22.44; H, 4.37; B, 15.11%. ^{1}H NMR (acetone-d_6): δ = 2.57 (s, 12 H, Cb*, J(PtH) 12), 2.73 (s, 3H, SMe$_2$), 2.75 (s, 3H, SMe$_2$), 3.84 (s, 1H, CH-cage, J(PtH) 22), 4.59 (s, 1H, CH-cage, J(PtH) 19). ^{11}B{^{1}H} NMR (acetone-d_6): δ = −23.9 (s, 1B), −21.0 (s, 1B), −20.3 (s, 1B), −13.2 (s, 1B), −11.7 (s, 1B), −10.9 (s, 1B), −9.8 (s, 2B, J(PtB) 106), −4.1 (s, 1B, J(PtB) 125).

4.4. Synthesis of [Cb*Pt(μ-η:η-C$_5$H$_3$Me$_2$BMe)FeCp*](CF$_3$SO$_3$)$_2$ (**3**(CF$_3$SO$_3$)$_2$)

MeNO$_2$ (1 ml) was added to Cb*PtCl$_2$ (48 mg, 0.128 mmol) and AgCF$_3$SO$_3$ (66 mg, 0.257 mmol). The reaction mixture was stirred for ca. 0.5 h and the precipitate of AgCl was centrifuged off. The solution obtained was added to complex Cp*Fe(η-C$_5$H$_3$Me$_2$BMe) (40 mg, 0.129 mmol) and the mixture was stirred for 2 h. Ether was added to precipitate brown solid. It was washed by acetone and reprecipitated by ether from MeNO$_2$ to give complex **3**(CF$_3$SO$_3$)$_2$ (20 mg, 17%) as a red solid. Anal. Calc. for C$_{28}$H$_{39}$BF$_6$FeO$_6$PtS$_2$: C, 36.90; H, 4.31. Found: C, 36.74; H, 4.31%. ^{1}H NMR (nitromethane-d_3): δ = 1.13 (s, 3H, BMe), 1.80 (s, 15H, Cp*), 2.00 (s, 12H, Cb*), 2.39 (s, 6H, Me), 6.13 (d, 2H, 3-/5-H, J(HH) 6), 6.31 (t, 1H, 4-H). ^{11}B{^{1}H} NMR (nitromethane-d_3): δ = 11.8 (bs, 1B).

4.5. Synthesis of [Cb*Pt(μ-η^5:η^5-C$_4$H$_4$BPh)RhCp*](BF$_4$)$_2$ (**4**(BF$_4$)$_2$)

MeNO$_2$ (1 ml) was added to Cb*PtCl$_2$ (25 mg, 0.067 mmol) and AgBF$_4$ · 3dioxane (62 mg, 0.135 mmol). The reaction mixture was stirred for ca. 0.5 h and the precipitate of AgCl was centrifuged off. The solution obtained was added to complex Cp*Rh(η^5-C$_4$H$_4$BPh) (25 mg, 0.066 mmol) and the mixture was stirred for 1 h. Ether was added to precipitate complex **4**(BF$_4$)$_2$ which was reprecipitated twice from CH$_2$Cl$_2$ by ether. Yield 36 mg (64%) as a red solid. Anal. Calc. for C$_{28}$H$_{36}$B$_3$F$_8$PtRh·0.5CH$_2$Cl$_2$: C, 38.14; H, 4.16. Found: C, 37.97; H, 4.02%. ^{1}H NMR (nitromethane-d_3): δ = 1.87 (s, 15 H, Cp*), 2.03 (s, 12 H, Cb*), 4.87 (m, 2H, 2-/5-H), 5.98 (m, 2H, 3-/4-H), 7.41 (m, 1H, p-Ph), 7.48 (m, 2H, m-Ph), 7.63 (m, 2H, o-Ph). ^{11}B{^{1}H} NMR (acetone-d_6): δ = −0.9 (s, 2B, BF$_4$), 9.6 (br s, 1B, C$_4$H$_4$B).

4.6. X-ray crystallography

Crystals were grown up by slow diffusion in a two-layer system (petroleum ether and a solution of complex in CH_2Cl_2 for **1**; ether/petroleum ether mixture and a solution of complex in CH_2Cl_2/$MeNO_2$ for **3**$(CF_3SO_3)_2$).

The principal crystallographic data, procedures for collecting data, and characteristics of structure refinement are listed in Table 3. Single-crystal X-ray diffraction experiment for **1** was carried out with a rebuild Syntex P2$_1$/Siemens P3 four-circle diffractometer, using graphite monochromated Mo Kα radiation ($\lambda = 0.71073$ Å, $\theta/2\theta$-scans). Analogous experiment for **3**$(CF_3SO_3)_2$ was carried out with a Bruker SMART APEX2 CCD area detector, using graphite monochromated Mo Kα radiation ($\lambda = 0.71073$ Å, φ and ω scans). The structures were solved by direct method and refined by the full-matrix least-squares against F^2 in anisotropic (for non-hydrogen atoms). The hydrogen atoms of the BH groups were found in the difference Fourier synthesis. The positions of other hydrogen atoms were calculated from the geometrical point of view and were included in the final refinement using a rigid model. All calculations were performed using the SHELXTL software [25]. In the case of **1** there is a high positive residual density of 2.15 e Å^{-3} near the Pt(1) center (distance 0.82 Å) due to considerable absorption effects which could not be corrected.

Acknowledgements

Appendix A. Supplementary material

CCDC 686613 and 686614 contain the supplementary crystallographic data for **1** and **3**$(CF_3SO_3)_2$. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2008.09.029.

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