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Dicationic triple-decker complexes with a bridging boratabenzene ligand

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Abstract

New dicationic triple-decker complexes with a bridging boratabenzene ligand [Cp*Fe(μ-η:η-C_5H_5BMe)ML]X_2 (ML = CoCp*, **6**(CF_3SO_3)$_2$; RhCp, **7**(BF_4)$_2$; IrCp, **8**(CF_3SO_3)$_2$; Ru(η-C_6H_6), **9**(CF_3SO_3)$_2$; Ru(η-$C_6H_3Me_3$-1,3,5), **10**(CF_3SO_3)$_2$; Ru(η-C_6Me_6), **11**(CF_3SO_3)$_2$) were synthesized by stacking reactions of Cp*Fe(η-C_5H_5BMe) (**2**) with the corresponding half-sandwich fragments $[ML]^{2+}$. The structure of **10**(CF_3SO_3)$_2$ was determined by X-ray diffraction study. © 2002 Published by Elsevier Science B.V.

Keywords: Boron; Cobalt; Iridium; Iron; Rhodium; Ruthenium; Boratabenzene; Sandwich compounds; Triple-decker complexes; X-ray structure

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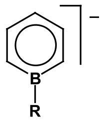
1. Introduction

Boron-containing heterocycles are known to possess high propensity to the formation of triple-decker complexes. Many examples of such complexes were prepared using five-membered heterocycles containing one, two or three boron atoms [1–3]. Boratabenzene anion **1** is the simplest six-membered boron-containing heterocycle. Although chemistry of its metal derivatives is rather well studied [4], only several examples of μ-boratabenzene triple-decker complexes have been reported [5].



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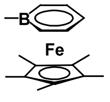
Herein we describe the synthesis of new dicationic triple-decker complexes with a bridging boratabenzene ligand as well as the results of X-ray diffraction study of one of them.

2. Results and discussion

2.1. Synthesis of triple-decker complexes [Cp*Fe(μ-η:η-C_5H_5BMe)ML]X_2

Electrophilic stacking of sandwich compounds with cationic half-sandwich fragments is an effective method for the preparation of cationic triple-decker complexes [6]. In particular, using this method the complexes with bifacially bonded cyclopentadienyl [6a,7], phospholyl [8], pentaphospholyl [9], and borole [10] ligands have been prepared. Three μ-boratabenzene dicationic complexes **3**–**5** have been also prepared with the use of the same method, viz. by reaction of the fragments $[Cp^*M']^{2+}$ with boratabenzene complexes Cp*Fe(η-C_5H_5BMe) (**2**) or Cp*Ru(η-C_5H_5BMe) [5].



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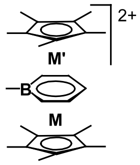

3: M = Fe, M' = Rh
4: M = Fe, M' = Ir
5: M = Ru, M' = Rh

Using stacking reactions of **2** with dicationic fragments $[LM]^{2+}$ we were able to prepare new dicationic 30-electron triple-decker complexes **6**–**11** with a bridging boratabenzene ligand (Scheme 1, the anions are omitted for clarity). The fragments $[LM]^{2+}$ were generated in form of labile solvates by reactions of the halide

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Table 1
^{1}H- and ^{11}B-NMR data for complexes **2** [5a] and **6**–**11**

	^{1}H-NMR							^{11}B-NMR
	4-H, 3-/5-H	2-/6-H	$^3J_{23}$	$^3J_{34}$	ML	FeCp*	BMe	
2	4.83 [a]	3.78 [a]	8.8 [a]			1.79 [a]	0.53 [a]	19.0 [b]
6 [a]	6.35, 6.25	4.95	8.8	5.2	1.82 (Cp*)	1.73	1.36	12.9
7 [a]	6.59	5.46	8.0		6.27 (Cp)	1.84	1.31	14.9, −1.4
8 [c]	6.83, 6.74	5.52	8.8	5.2	6.52 (Cp)	1.91	1.45	10.1
9 [c]	6.71	5.21	8.8		6.51 (C_6H_6)	1.86	1.32	12.7
10 [c]	6.53	4.99	8.0		6.44 ($C_6\mathit{H}_3Me_3$), 2.34 ($C_6H_3\mathit{Me}_3$)	1.86	1.33	12.0
11 [c]	6.44, 6.32	4.88	8.8	5.2	2.39 (C_6Me_6)	1.84	1.20	10.8

Chemical shifts in ppm, coupling constants in Hz.

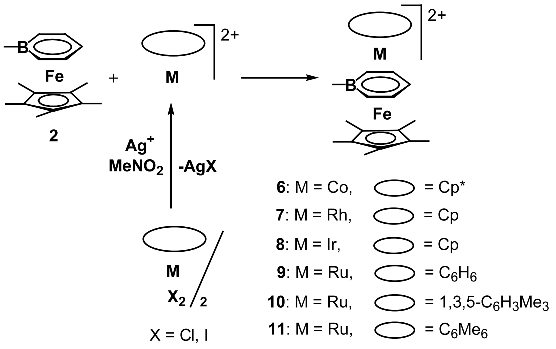
[a] In CD_3NO_2.

[b] In C_6D_6.

[c] In $(CD_3)_2CO$.

complexes $[LMX_2]_2$ (LM = Cp*Co, CpRh, (arene)Ru, X = Cl; LM = CpIr, X = I) with $AgCF_3SO_3$ or $AgBF_4$ in nitromethane[1]. Alternatively, the fragment $[CpRh]^{2+}$ was generated using $CpRhI_2(CO)$. It is noteworthy that the fragments $[Cp^*Co]^{2+}$, $[CpRh]^{2+}$, and $[CpIr]^{2+}$ were not previously used for the synthesis of triple-decker complexes.

Triflate or tetrafluoroborate salts of triple-decker cations **6**–**11**[2] were isolated as deep-coloured solids with the yields ca. 50–60%. They were characterized by ^{1}H- and ^{11}B-NMR spectroscopy (Table 1) and elemental analysis. Signals of boratabenzene ring protons are down-field shifted and the signals of boron atoms are up-field shifted as compared with the corresponding signals for sandwich compound **2**. The same pattern has been observed for compounds **3**–**5** [5].



Scheme 1.

The complexes **6**–**11** are moderately air-stable, complex **7** being the less stable among them. They are readily soluble in $MeNO_2$ with the formation of rather stable solutions. However, they undergo nucleophilic degradation in strongly coordinating solvents (e.g. MeCN), the rate being dependent on the fragment LM. Thus, CpRh complex **7** is immediately destroyed by MeCN (with colour change from violet to orange), while CpIr complex **8** reacts within 5 min. However, (C_6H_6)Ru complex **9** decomposes only within ca. 10 h. More rapid nucleophilic degradation of rhodium and iridium complexes as compared with the ruthenium analogue is apparently connected with higher oxidation state of Rh and Ir atoms. In accordance with ^{1}H- and ^{11}B-NMR data, complexes **7** and **8** decompose regioselectively with elimination of **2** via nucleophilic attack at Rh or Ir atom; in the case of **8** the formation of the acetonitrile complex $[CpIr(MeCN)_3]^{2+}$ was also detected. Pentamethylated analogues **3** and **4** have been shown to decompose in a similar way [5b]. In contrast, degradation of **9** proceeds by two pathways: (a) by nucleophilic attack at Ru atom to give **2** and $[(\eta\text{-}C_6H_6)Ru(MeCN)_3]^{2+}$; (b) by the attack at Fe atom to give $[(\eta\text{-}C_6H_6)Ru(C_5H_5BMe)]^+$ (cf. $[(C_6H_6)Ru(C_5H_5BPh)]^+$ [15]); the ratio of the products formed by ways (a) and (b) being ca. 2:1. Lower selectivity of nucleophilic degradation of **9** is apparently connected with comparable susceptibility to nucleophilic attack of both metal atoms.

2.2. Structure of $[Cp^*Fe(\mu\text{-}\eta{:}\eta\text{-}C_5H_5BMe)Ru(\eta\text{-}C_6H_3Me_3\text{-}1,3,5)](CF_3SO_3)_2$

The structure of triple-decker cation **10** is shown in Fig. 1 and selected bond lenghts and angles are given in

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[1] Complex $[CpIrI_2]_2$ [11], which was used for the generation of the fragment $[CpIr]^{2+}$, was prepared by a simple high-yield procedure based on the reaction of I_2 with $CpIr(C_2H_4)_2$. The latter was obtained by one-pot procedure [12] (based on the reaction of $[(C_8H_{14})_2IrCl]_2$ with C_2H_4 to give $[(C_2H_4)_2IrCl]_2$ with subsequent treatment with CpTl) and used without purification. Overall yield of $[CpIrI_2]_2$ from $[(C_8H_{14})_2IrCl]_2$ is 91 %. Direct reaction of $[(C_8H_{14})_2IrCl]_2$ with CpTl gives a mixture of $CpIr(C_8H_{14})_2$ [13] and $CpIr(C_5H_6)$ [14]; only the former reacts with I_2 to give $[CpIrI_2]_2$, while the latter gives $[Cp_2Ir]^+$.

[2] Usually $CF_3SO_3^-$ was used as a counter-ion with the exception of cation **7**, which was isolated as the tetrafluoroborate salt since we were unable to prepare its triflate salt in analytically pure form.

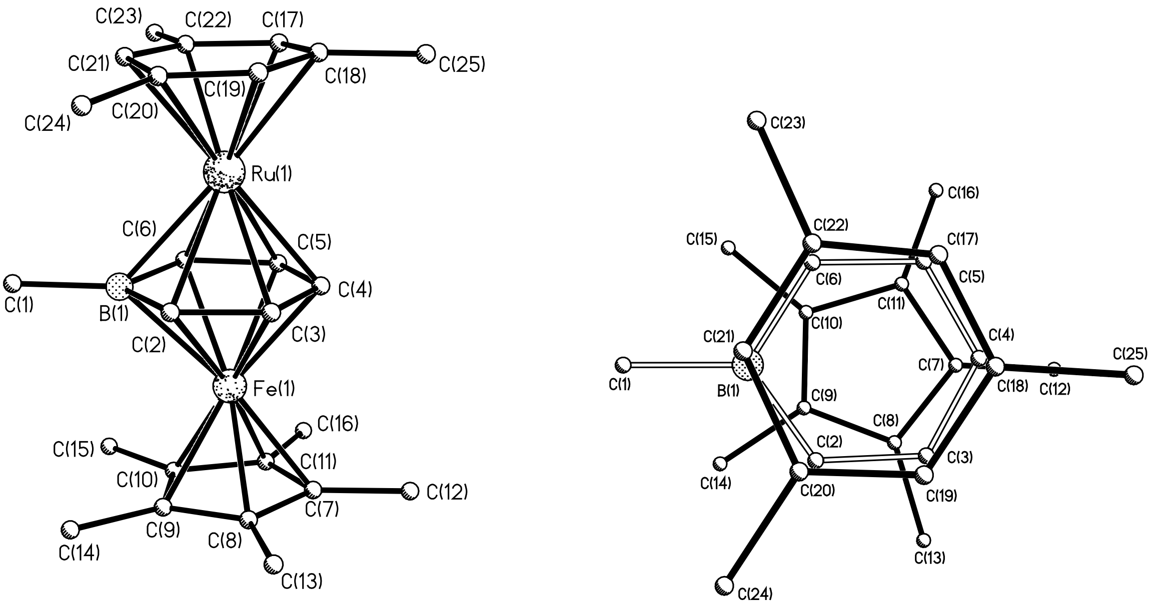

Fig. 1. Structure of cation **10**. Hydrogen atoms are omitted for clarity.

Table 2. The cation **10** consists of three cyclic frames ($C_6H_3Me_3$, C_5H_5BMe and Cp*), between which two metal atoms (Ru and Fe) are located. The planes of cyclic ligands are ca. parallel; the dihedral angles $C_6H_3Me_3/C_5H_5BMe$ and C_5H_5BMe/Cp* are equal to 2.0(4) and 1.4(5)°, respectively. The six-membered cycles are eclipsed. The metal-to-ring distances (Δ) are as follows: Ru···C_5H_5BMe 1.725(3), Fe···C_5H_5BMe 1.571(3) Å, Ru···$C_6H_3Me_3$ 1.679(3) Å and Fe···Cp* 1.657(4) Å. It is noteworthy that Δ(Ru···C_5H_5BMe) and Δ(Fe···C_5H_5BMe) values are close to the corresponding average statistic standard values for metal-(η-C_6R_6) bonding (Δ(Ru···C_6R_6) 1.745 Å, Δ(Fe···C_6R_6) 1.563 Å [16]). However, Δ(Ru···$C_6H_3Me_3$) (1.679 Å) and Δ(Fe···Cp*) (1.657 Å) values for **10** are shorter than the corresponding standard values (Δ(Ru···C_6R_6) 1.745 Å, Δ(Fe···C_5R_5) 1.739 Å). These data suggest that π-bonding of Ru and Fe atoms with boratabenzene ring is very similar to the bonding with C_6R_6, while bonding with terminal rings ($Me_3C_6H_3$ and Cp*) is stronger than that usually observed for these rings. The bonds Ru(1)–B(1) (2.340(8) Å) and Fe(1)–B(1) (2.242(9) Å) are considerably longer than the corresponding bonds Ru(1)–C(C_5H_5BMe) (2.221–2.278 Å, av. 2.248 Å) and Fe(1)–C(C_5H_5BMe) (2.094–2.167 Å, av. 2.130 Å). This fact is consistent with the larger covalent radius of boron versus carbon[3] and may also have an electronic origin. The boratabenzene intra-ring bonds B–C (1.51(1), 1.56(1) Å) are longer than the bonds C–C (1.42–1.44(1) Å). The difference between intra-ring bonds causes asymmetry of exocyclic valent angles (C(1)B(1)C(2) 125.4(7), C(1)B(1)C(6) 122.5(7)°). The ring is slightly folded along the line C(2)···C(6) away from the ruthenium atom, the folding angle being 1.5°.

3. Conclusion

Using stacking reactions of boratabenzene complex **2** with dicationic half-sandwich fragments a series of new dicationic triple-decker complexes with bifacially bonded boratabenzene ligand was synthesized thus illustrating that this ligand is very perspective for creation of new triple-decker complexes.

Table 2
Selected bond lengths [Å] and angles [°] for cation **10**

Bond lengths			
Ru(1)–C(17)	2.168(8)	Fe(1)–C(7)	2.062(9)
Ru(1)–C(20)	2.179(8)	Fe(1)–C(11)	2.064(8)
Ru(1)–C(21)	2.181(9)	Fe(1)–C(4)	2.094(8)
Ru(1)–C(19)	2.183(8)	Fe(1)–C(3)	2.115(7)
Ru(1)–C(22)	2.187(9)	Fe(1)–C(5)	2.115(7)
Ru(1)–C(18)	2.221(8)	Fe(1)–C(2)	2.159(7)
Ru(1)–C(4)	2.228(8)	Fe(1)–C(6)	2.167(8)
Ru(1)–C(3)	2.231(8)	Fe(1)–B(1)	2.242(9)
Ru(1)–C(6)	2.249(8)	B(1)–C(2)	1.511(11)
Ru(1)–C(2)	2.255(7)	B(1)–C(6)	1.558(11)
Ru(1)–C(5)	2.278(7)	B(1)–C(1)	1.605(12)
Ru(1)–B(1)	2.340(8)	C(2)–C(3)	1.430(10)
Fe(1)–C(10)	2.055(8)	C(3)–C(4)	1.424(11)
Fe(1)–C(8)	2.059(8)	C(4)–C(5)	1.436(11)
Fe(1)–C(9)	2.060(8)	C(5)–C(6)	1.441(11)
Bond angles			
C(3)–C(2)–B(1)	123.0(7)	C(5)–C(6)–B(1)	123.1(7)
C(4)–C(3)–C(2)	121.0(7)	C(2)–B(1)–C(6)	112.0(7)
C(3)–C(4)–C(5)	121.9(8)	C(2)–B(1)–C(1)	125.4(7)
C(4)–C(5)–C(6)	118.9(7)	C(6)–B(1)–C(1)	122.5(7)

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[3] Covalent radii: 0.82 Å (boron), 0.77 Å (carbon) [17].

4. Experimental

4.1. General

The reactions were carried out under an inert atmosphere in dry solvents, unless otherwise stated. The isolation of products was conducted in air. Starting materials **2** [5b], $[Cp^*CoCl_2]_2$ [18], $[CpRhCl_2]_2$ [19], $CpRhI_2(CO)$ [20], $[(C_8H_{14})_2IrCl]_2$ [21], $[(\eta\text{-}C_6H_6)RuCl_2]_2$ [22], $[(\eta\text{-}C_6H_3Me_3\text{-}1,3,5)RuCl_2]_2$ [22], and $[(\eta\text{-}C_6Me_6)RuCl_2]_2$ [23] were prepared as described in the literature. ^1H- and $^{11}B\{^1H\}$-NMR spectra were recorded on a Bruker AMX-400 spectrometer (^1H 400.13; ^{11}B 128.38 MHz) relative to residual protons of the solvents (^1H) or $BF_3\cdot Et_2O$ (^{11}B, external standard).

4.2. Synthesis of $[CpIrI_2]_2$

Ethylene was bubbled through a suspension of $[(C_8H_{14})_2IrCl]_2$ (447 mg, 1.00 mg equiv.) in THF (10 ml) for 0.5 h. CpTl (280 mg, 1.04 mmol) was added to the solution and the mixture was stirred for 1 h. The solvent was removed in vacuo and the residue was extracted with petroleum ether. After removal of the solvent in vacuo a white solid was dissolved in CH_2Cl_2 (5 ml) and a solution of I_2 (254 mg, 1 mmol) in the same solvent was added (an inert atmosphere is not necessary). The reaction mixture was stirred for 4 h. The brown precipitate formed was centrifuged off and washed by acetone until washings become colorless. After drying in vacuo the complex $[CpIrI_2]_2$ was obtained as a brown solid, yield 465 mg (91%). ^1H-NMR (DMSO-d^6, ppm): δ 6.00 (s, Cp).

4.3. Synthesis of $[Cp^*Fe(\mu\text{-}\eta:\eta\text{-}C_5H_5BMe)ML]X_2$ (**6–11**)

$MeNO_2$ (1 ml) was added to a mixture of $[LMX_2]_2$ (LM = Cp*Co, CpRh, (η-C_6H_6)Ru, (η-$C_6H_3Me_3$-1,3,5)Ru, (η-C_6Me_6)Ru, X = Cl; LM = CpIr, X = I) or $CpRhI_2(CO)$ (0.071 mg equiv.) and $AgCF_3SO_3$ or $AgBF_4\cdot$3dioxane (0.142 mmol). The reaction mixture was stirred for ca. 0.5 h. The precipitate of AgX was centrifuged off and the solution obtained was added to $Cp^*Fe(\eta\text{-}C_5H_5BMe)$ (20 mg, 0.071 mmol). The solution was stirred for 2 h. Ether (ca. 10 ml) was added to precipitate a bright-coloured solid, which was filtered off and reprecipitated twice from acetone by ether.

6$(CF_3SO_3)_2$, green, yield 28 mg (51%). Anal. Calc. for $C_{28}H_{38}BCoF_6FeO_6S_2$: C, 43.43; H, 4.95%. Found: C, 43.49; H, 4.92%.

7$(BF_4)_2$, violet, yield 32 mg (72%) from $[CpRhCl_2]_2$ or 23 mg (43%) from $CpRhI_2(CO)$. Anal. Calc. for $C_{21}H_{28}B_3F_8FeRh$: C, 40.44; H, 4.53%. Found: C, 39.79; H, 4.42%.

8$(CF_3SO_3)_2$, red, yield 39 mg (66%). Anal. Calc. for $C_{23}H_{28}BF_6FeIrO_6S_2$: C, 32.99; H, 3.37%. Found: C, 32.92; H, 3.30%.

9$(CF_3SO_3)_2$, red, yield 27 mg (50%). Anal. Calc. for $C_{24}H_{29}BF_6FeO_6S_2Ru$: C, 37.96; H, 3.85%. Found: C, 37.80; H, 3.84%.

10$(CF_3SO_3)_2$, red, yield 32 mg (56%). Anal. Calc. for $C_{27}H_{35}BF_6FeO_6S_2Ru$: C, 40.46; H, 4.40%. Found: C, 40.01; H, 4.33%.

11$(CF_3SO_3)_2$, red, yield 36 mg (60%). Anal. Calc. for $C_{30}H_{41}BF_6FeO_6S_2Ru$: C, 42.72; H, 4.90%. Found: C, 42.07; H, 4.93%.

4.4. X-ray crystallography

4.4.1. Crystal structure analysis of **10**

Crystals of **10**$(CF_3SO_3)_2$ were obtained by slow diffusion of petroleum ether and CH_2Cl_2 solution of the complex. Single-crystal X-ray diffraction experiments were carried out with a Bruker SMART 1000 CCD area detector, using graphite monochromated Mo–K_α radiation ($\lambda = 0.71073$ Å, ω-scans with a 0.3° step in ω and 10 s per frame exposure, $2\theta < 59.8°$) at 110 K. Low temperature of the crystals was maintained with a Cryostream (Oxford Cryosystems) open-flow N_2 gas cryostat. The structure was solved by direct method and refined by full-matrix least squares against F^2 in anisotropic approximation [24]. All hydrogen atoms were placed in the geometrically calculated positions and included in the refinement using the riding model approximation with the $U_{iso}(H) = 1.2\ U_{eq}(C)$ for CH and $U_{iso}(H) = 1.5\ U_{eq}(C)$ for CH_3 groups, where $U_{eq}(C)$ is the equivalent isotropic temperature factor of the carbon atom bonded to the corresponding H atom.

4.4.2. Crystal data

$C_{27}H_{35}BF_6FeO_6RuS_2$ ($M = 801.40$), monoclinic, space group $P2_1/c$, $a = 16.677(3)$, $b = 17.738(4)$, $c = 10.534(2)$ Å, $\beta = 90.604(3)$, $V = 3116(1)$ Å^3, $Z = 4$, $D_{calc} = 1.708$ g cm^{-3}, $\mu = 1.160$ mm^{-1}, $F(000) = 1624$, crystal size $0.35 \times 0.20 \times 0.15$ mm, T_{min}/T_{max} 0.353/0.802, No. of measured and independent reflections 27 198 and 8753, $R_{int} = 0.119$, No. of parameters used in refinement 397, $R_1 = 0.0733$ (from 2652 unique reflections with $I > 2\sigma(I)$) and $wR_2 = 0.1957$ (from all 8753 unique reflections).

5. Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Center, CCDC No. 172158 for **10**. Copies of this information may be obtained free of charge from: The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (Fax: +44-1223-336-033; e-mail: deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

Acknowledgements

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