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Synthesis and structures of the triple-decker type complexes $CpCo(\mu\text{-}1,3\text{-}C_3B_2Me_5)MX_2L$ (M = Rh and Ir; X = Cl and Br; L = Me_2SO and PPh_3)

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ABSTRACT

The dimeric triple-decker type halides $[CpCo(\mu\text{-}1,3\text{-}C_3B_2Me_5)MX_2]_2$ (M = Rh, X = Cl, **1**; M = Ir, X = Cl, **2**; M = Rh, X = Br, **3**; M = Ir, X = Br, **4**) react with Me_2SO and PPh_3 giving adducts $CpCo(\mu\text{-}1,3\text{-}C_3B_2Me_5)M(L)X_2$ (**5**–**12**). The Me_2SO ligand is S-bonded to the metal atom in the solid state, while in acetone it is O-bonded. Structures of **5**–**12** were confirmed by X-ray diffraction.

1. Introduction

The (pentamethylcyclopentadienyl)halide complexes $[Cp^*MX_2]_2$ (M = Rh, Ir; X = Cl, Br, I) are widely used as synthons of the Cp*M fragment [1]. Since Maitlis et al. opened up the rich chemistry of these dimers and monomeric derivatives Cp^*MX_2L, their catalytic properties were extensively studied for different processes: homogeneous hydrogenation [2], oxidation of primary and secondary alcohols [3], asymmetric transfer hydrogenation of aromatic ketones [4], N-alkylation of carbamates and amides [5], intermolecular hydroarylation of alkynes [6], arylation of imines *via* C–H bond functionalization [7], Rh-catalized indole synthesis [8], Grignard-type arylation of aldehydes *via* a Rh-catalyzed C–H activation [9], oxidative carbonylation of benzamides for phthalimide synthesis *via* C–H activation [10].

Analogous halide complexes [(1,2,3,4,7-pentamethylindenyl)$MX_2]_2$ [11], $[(9\text{-}SMe_2\text{-}7,8\text{-}C_2B_9H_{10})MX_2]_2$ [12], $[CpCo(\mu\text{-}1,3\text{-}C_3B_2Me_5)MX_2]_2$ [13] were synthesized in our laboratory and proved to be synthetically useful. For instance, the metallacarborane halides $[(9\text{-}SMe_2\text{-}7,8\text{-}C_2B_9H_{10})MX_2]_2$ react with PPh_3 and CO giving mononuclear compounds $(9\text{-}SMe_2\text{-}7,8\text{-}C_2B_9H_{10})M(L)X_2$ (L = PPh_3, CO) [14]. Herein, we describe similar reactions of the triple-decker type halides $[CpCo(\mu\text{-}1,3\text{-}C_3B_2Me_5)MX_2]_2$ (M = Rh and Ir) with dimethylsulfoxide and triphenylphosphine. The structures of the adducts obtained are also discussed.

2. Results and discussion

2.1. Synthesis

Compounds $[CpCo(\mu\text{-}1,3\text{-}C_3B_2Me_5)MX_2]_2$ (M = Rh, X = Cl, **1**; M = Ir, X = Cl, **2**; M = Rh, X = Br, **3**; M = Ir, X = Br, **4**) [13] are insoluble in low coordinating solvents such as CH_2Cl_2 and acetone, but soluble in strongly coordinating MeCN and Me_2SO. We found that the reaction of halides **1**–**4** with 2-electron donor ligands Me_2SO and PPh_3 leads to the formation of the monomeric complexes **5**–**12** in high yields (Scheme 1).

Adducts **5**–**12** are deep-colored solids, stable in air for long periods of time. All complexes were characterized by elemental analysis, NMR and IR spectroscopy. The IR spectra of solid complexes **5**–**8** indicate that the Me_2SO ligand is S-bonded to the metal atom (ν(S=O) 1019 cm^{-1}); it was further supported by X-ray diffraction (*vide infra*). However, in acetone solution only the

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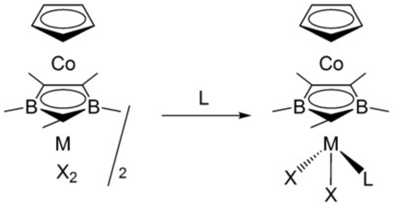

	M	X	L	%
5	Rh	Cl	Me_2SO	91
6	Ir	Cl	Me_2SO	90
7	Rh	Br	Me_2SO	97
8	Ir	Br	Me_2SO	96
9	Rh	Cl	PPh_3	83
10	Ir	Cl	PPh_3	91
11	Rh	Br	PPh_3	96
12	Ir	Br	PPh_3	82

	M	X
1	Rh	Cl
2	Ir	Cl
3	Rh	Br
4	Ir	Br

Scheme 1. Synthesis of the adducts.

stretching vibration of S=O group at 902 cm^{-1} is observed suggesting that the Me_2SO ligand becomes O-bonded to the metal atom [15]. Similar tautomeric behavior was previously reported for $[Cp^*M(Me_2SO)_3](PF_6)_2$ complexes [16].

The signals of the Me groups of $C_3B_2Me_5$ ligand in ^1H NMR spectra of complexes **5**–**12** are down-field shifted in comparison with the triple-decker complexes $CpCo(\mu\text{-}1,3\text{-}C_3B_2Me_5)MC_5R_5$ (M(ring) = RuCp, RuCp*, $Co(C_4Me_4)$) [17]. All signals of Me groups in ^1H NMR spectrum of the chloride $CpCo(\mu\text{-}1,3\text{-}C_3B_2Me_5)RhCl_2PPh_3$ (**9**) appear as singlets whereas for the related bromide **11** doublets are observed for 4,5-CMe and 1,3-BMe groups. In the $^{31}P\{^1H\}$ NMR spectra the doublets are observed for both these complexes.

2.2. X-ray diffraction studies

Structures of the complexes **5**–**12** were confirmed by X-ray diffraction (the representative structures for **5** and **9** are shown in Figs. 1 and 2). Selected structural parameters for these compounds are given in Table S1 and S2 (see Supporting Information). Crystals of compounds **5**–**8**[1] are isomorphous. In addition, adducts **9**/**10** and **11**/**12** are isomorphous pairwise. The Cp and $C_3B_2Me_5$ ring ligands are almost parallel (the dihedral angles 0.0–2.6°), and the metal atoms are placed nearly over the centroids of the rings. The mutual orientation of the rings in compounds **9**–**12** is staggered, while for complexes **5**–**8** it is nearly eclipsed for independent molecule A, and staggered for independent molecule B. All Me groups of the $C_3B_2Me_5$ ligand are displaced from the C_3B_2 ring plane in the direction towards the Co atom with deviation about 0.02(1)–0.21(1) Å for all complexes. The C_3B_2 ring in complexes **5**–**12** is almost planar with a small folding along the B···B line (*ca* 1.4°).

The M centers (M = Rh and Ir) in complexes **5**–**12** possess pseudo-octahedral (three-legged piano stool) geometry where the $[CpCo(\mu\text{-}1,3\text{-}C_3B_2Me_5)]$ group occupies three positions and three ligands (Br/Cl and Me_2SO/PPh_3) complete the pseudo-octahedral coordination. In complexes **5**–**8** the Me_2SO ligand is S-bonded and M–S bond length lies within the range of 2.313(1)–2.319(1) and 2.284(1)–2.290(2) Å for Rh and Ir complexes, respectively. The M–P bonds in complexes **9**–**12** are almost equidistant (2.311(1)–2.330(1) Å). The M–Cl and M–Br bond lengths do not deviate significantly in complexes **5**–**12** and lie within the ranges of 2.387(1)–2.408(1) and 2.497(1)–2.534(1) Å, respectively. These data are comparable with those for the analogous compounds $Cp^*MCl_2PR_3$ (M = Rh and Ir, R = Ph, C_6F_5, OEt and other) [18].

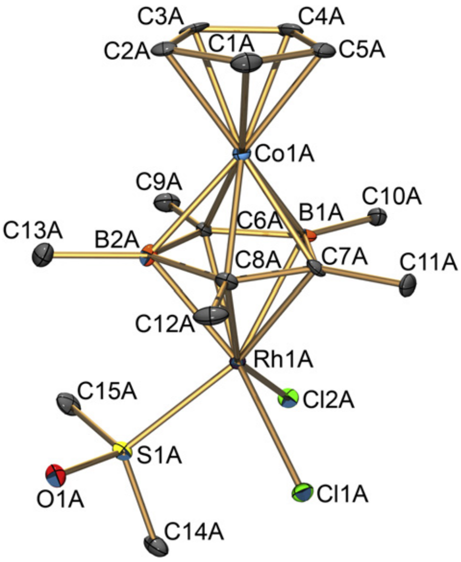

Fig. 1. Molecular structure of independent molecule A for compound **5**. Ellipsoids are shown at the 50% level.

Noteworthy, for complexes **5**–**12** the angles X1–M–X2, X1–M–P(S), and X2–M–P(S) (M = Rh and Ir; X = Cl and Br) reflect the pseudo-octahedral coordination geometry around the metal center. The angles increase in the order X(2)–M–P(S) < X(1)–M–P(S) < X1–M–X2 irrespectively to the value of M–P or M–S bond length. Such angles order can be explained by the repulsion of negatively charged halogen atoms.

Interestingly, the halogen atoms X (X = Cl or Br) in complexes **5**–**12** are always oriented in *trans*-position to the C6 and C8 atoms of the C_3B_2 ring, while ligand L (L = Me_2SO or PPh_3) is oriented in *cis*-position to B2 atom and *trans*-position to C7–B1 bond. This type of coordination causes the elongation of M–B1 bond in comparison with M–B2 by 0.030(2)–0.090(2) Å and shortening of M–C6 and M–C8 bonds in comparison with M–C7 by 0.010(2)–0.040(2) Å for all complexes. One may conclude that in adducts **5**–**12** Me_2SO and

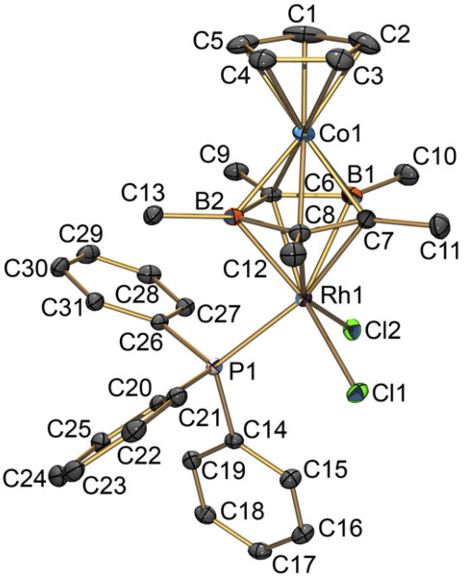

Fig. 2. Molecular structure of compound **9**. Ellipsoids are shown at the 50% level.

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[1] The unit cells of Me_2SO-adducts **5**–**8** contain two independent molecules; averaged values will be used in further discussion.

PPh_3 possess moderate structural *trans*-effect *vs.* halide anions in accordance with general tendency [19].

3. Conclusion

The reactions of the halide complexes **1**–**4** with 2-electron donor ligands Me_2SO and PPh_3 allowed to prepare the triple-decker type adducts $CpCo(\mu\text{-}1,3\text{-}C_3B_2Me_5)MX_2L$ (L = Me_2SO and PPh_3). The IR spectroscopy revealed the tautomeric behavior for dimethylsulfoxide ligand (S or O coordination) in adducts **5**–**8**. The X-ray diffraction study of adducts **5**–**12** showed that the Me_2SO and PPh_3 ligands possess moderate structural *trans*-effect, whereas halogen atoms show weak *trans*-effect in the presence of $[CpCo(\mu\text{-}1,3\text{-}C_3B_2Me_5)]$ sandwich unit.

4. Experimental

4.1. General

All reactions were carried out under argon in anhydrous solvents which were purified and dried using standard procedures. The isolation of products was conducted in air. Starting materials were prepared as described in the literature: $CpCo(1,3\text{-}C_3B_2Me_5H)$ [20], $[(cod)RhCl]_2$ [21], $[(cod)IrCl]_2$ [22], $[CpCo(\mu\text{-}1,3\text{-}C_3B_2Me_5)MBr_2]_2$ [13], and $[CpCo(\mu\text{-}1,3\text{-}C_3B_2Me_5)MCl_2]_2$ analogously to the former bromide complexes. The ^1H, $^{11}B\{^1H\}$, and $^{31}P\{^1H\}$ NMR spectra were recorded with a Bruker AMX 400 spectrometer operating at 400.13, 128.38, and 161.98 MHz, respectively. The IR spectra of the compounds were measured by Infralum FT-801 (Lumex) FTIR spectrometer over the range 400–4000 cm^{-1}.

4.2. Synthesis of

4.2.1. $CpCo(\mu\text{-}1,3\text{-}C_3B_2Me_5)MX_2(Me_2SO)$

(**5**) M = Rh; X = Cl. The Me_2SO/CH_2Cl_2 mixture (20 ml; 1/20) was added to the solid complex **1** (0.060 g, 0.07 mmol) and stirred for 24 h. The green solution was slowly evaporated to form a black crystalline precipitate. The product was washed with 3 ml of cooled CH_2Cl_2 (0 °C) from excess of Me_2SO and dried in vacuum. Yield 0.064 g (91%). Black solid. $C_{15}H_{26}B_2Cl_2CoORhS$ (508.80): calcd. C 35.41, H 5.15, B 4.25; found C 35.30, H 5.10, B 4.13. ^1H NMR (CD_2Cl_2): $\delta = 4.88$ (s, 5H, CoCp), 2.93 (s, 6H, Me_2SO), 2.32 (s, 6H, 4,5-Me), 1.53 (s, 6H, 1,3-BMe), 1.51 (s, 3H, 2-Me). $^{11}B\{^1H\}$ NMR (CD_2Cl_2): $\delta = 22.29$ (bs). IR (ν, solid sample, KBr, cm^{-1}): 1019 (SO). IR (ν, acetone solution, KBr, cm^{-1}): 902 (SO).

(**6**) M = Ir; X = Cl. Complex **6** was prepared similarly to **5** from complex **2** (0.045 g, 0.04 mmol). Yield 0.046 g (90%). Black solid. $C_{15}H_{26}B_2Cl_2CoIrOS$ (598.11): calcd. C 30.12, H 4.38, B 3.61; found C 29.73, H 4.36, B 3.45. ^1H NMR (CD_2Cl_2): $\delta = 4.88$ (s, 5H, CoCp), 3.11 (s, 6H, Me_2SO), 2.45 (s, 6H, 4,5-Me), 1.56 (s, 6H, 1,3-BMe), 1.53 (s, 3H, 2-Me). $^{11}B\{^1H\}$ NMR (CD_2Cl_2): $\delta = 16.09$ (bs). IR (ν, solid sample, KBr, cm^{-1}): 1018 (SO). IR (ν, acetone solution, KBr, cm^{-1}): 902 (SO).

(**7**) M = Rh; X = Br. Complex **7** was prepared similarly to **5** from complex **3** (0.062 g, 0.06 mmol). Yield 0.067 g (94%). Black solid. $C_{15}H_{26}B_2Br_2CoORhS$ (597.70): calcd. C 30.14, H 4.38, B 3.62; found C 30.20, H 4.45, B 3.58. ^1H NMR (CD_2Cl_2): $\delta = 4.87$ (s, 5H, CoCp), 3.07 (s, 6H, Me_2SO), 2.42 (s, 6H, 4,5-Me), 1.60 (s, 6H, 1,3-BMe), 1.57 (s, 3H, 2-Me). $^{11}B\{^1H\}$ NMR (CD_2Cl_2): $\delta = 22.22$ (bs). IR (ν, solid sample, KBr, cm^{-1}): 1026 (SO). IR (ν, acetone solution, KBr, cm^{-1}): 902 (SO).

(**8**) M = Ir; X = Br. Complex **8** was prepared similarly to **5** from complex **4** (0.06 g, 0.05 mmol). Yield 0.063 g (93%). Black solid. $C_{15}H_{26}B_2Br_2CoIrOS$ (687.01): calcd. C 26.22, H 3.81, B 3.15; found C 26.35, H 3.97, B 3.12. ^1H NMR (CD_2Cl_2): $\delta = 4.88$ (s, 5H, CoCp), 3.32 (s, 6H, Me_2SO), 2.53 (s, 6H, 4,5-Me), 1.63 (s, 6H, 1,3-BMe), 1.53 (s, 3H, 2-Me). $^{11}B\{^1H\}$ NMR (CD_2Cl_2): $\delta = 16.05$ (bs). IR (ν, solid sample, KBr, cm^{-1}): 1027 (SO). IR (ν, acetone solution, KBr, cm^{-1}): 902 (SO).

4.2.2. $CpCo(\mu\text{-}1,3\text{-}C_3B_2Me_5)MX_2PPh_3$

(**9**) M = Rh; X = Cl. The CH_2Cl_2 (2 ml) was added to a mixture of complex **1** (0.043 g, 0.05 mmol) and PPh_3 (0.026 g, 0.1 mmol). The reaction mixture was stirred for 12 h and then filtered. The product was precipitated by addition of Et_2O (20 ml), centrifuged, washed with Et_2O and dried in vacuo. Yield 0.057 g (83%). Dark-green solid. $C_{31}H_{35}B_2Cl_2CoRhP$ (692.96): calcd. C 53.73, H 5.09, B 3.12; found C 53.90, H 5.22, B 3.04. ^1H NMR (CD_2Cl_2): $\delta = 7.62$–7.68 (m, 6H, PPh_3), 7.37–7.47 (m, 6H, PPh_3), 7.05–7.20 (m, 3H, PPh_3), 4.68 (s, 5H, CpCo), 2.35 (s, 6H, 4,5-Me), 1.09 (s, 6H, 1,3-BMe), 0.58 (s, 3H, 2-Me). $^{11}B\{^1H\}$ NMR (CD_2Cl_2): $\delta = 21.27$ (s). $^{31}P\{^1H\}$ NMR (CD_2Cl_2): $\delta = 29.84$ (d, $^1J_{RhP} = 161,0$ Hz).

(**10**) M = Ir; X = Cl. Complex **10** was prepared similarly to **9** from complex **2** (0.062 g, 0.06 mmol) and PPh_3 (0.032 g, 0.12 mmol). Yield 0.085 g (91%). Dark-brown solid. $C_{31}H_{35}B_2Cl_2CoIrP$ (782.27): calcd. C 47.60, H 4.51, B 2.76; found C 47.51, H 4.45, B 2.86. ^1H NMR (CD_2Cl_2): $\delta = 7.56$–7.63 (m, 6H, PPh_3), 7.47–7.39 (m, 9H, PPh_3), 4.70 (s, 5H, Cp), 2.54 (s, 6H, 4,5-Me), 1.14 (s, 6H, 1,3-BMe), 0.85 (s, 3H, 2-Me). $^{11}B\{^1H\}$ NMR (CD_2Cl_2): $\delta = 15.81$ (s). $^{31}P\{^1H\}$ NMR (CD_2Cl_2): $\delta = 0.41$ (s).

(**11**) M = Rh; X = Br. Complex **11** was prepared similarly to **9** from complex **3** (0.062 g, 0.06 mmol) and PPh_3 (0.032 g, 0.06 mmol). Yield 0.086 g (92%). Dark-green solid. $C_{31}H_{35}B_2Br_2CoRhP$ (781.86): calcd. C 47.62, H 4.51, B 2.77; found C 47.71, H 4.65, B 2.86. ^1H NMR ($CDCl_3$): $\delta = 7.58$–7.68 (m, 6H, PPh_3), 7.37–7.43 (m, 6H, PPh_3), 7.05–7.15 (m, 3H, PPh_3), 4.61 (s, 5H, CpCo), 2.47 (d, 6H, $^3J_{RhH} = 0.64$ Hz, 4,5-Me), 1.20 (d, 6H, $^3J_{RhH} = 0.32$ Hz, 1,3-BMe), 0.58 (c, 3H, 2-Me). $^{11}B\{^1H\}$ NMR ($CDCl_3$): $\delta = 21.16$ (s). $^{31}P\{^1H\}$ NMR ($CDCl_3$): $\delta = 35.04$ (d, $^1J_{RhP} = 121.6$ Hz).

(**12**) M = Ir; X = Br. Complex **12** was prepared similarly to **9** from complex **4** (0.061 g, 0.05 mmol) and PPh_3 (0.026 g, 0.1 mmol). Yield 0.075 g (86%). Dark-brown solid. $C_{31}H_{35}B_2Br_2CoIrP$ (871.17): calcd. C 42.74, H 4.05, B 2.48; found C 42.27, H 3.91, B 2.75. ^1H NMR ($CDCl_3$): $\delta = 7.60$–7.70 (m, 6H, PPh_3), 7.47–7.39 (m, 9H, PPh_3), 7.14–7.04 (m, 3H, PPh_3), 4.65 (s, 5H, Cp), 2.65 (s, 6H, 4,5-Me), 1.24 (s, 6H, 1,3-BMe), 0.94 (s, 3H, 2-Me). $^{11}B\{^1H\}$ NMR ($CDCl_3$): $\delta = 16.34$ (s). $^{31}P\{^1H\}$ NMR ($CDCl_3$): $\delta = -1.03$ (s).

4.3. X-ray crystallography of **5**–**12**

The crystals suitable for X-ray study were obtained by slow evaporation of CH_2Cl_2 from Me_2SO/CH_2Cl_2 solutions (1/20) of complexes **5**–**8**, slow evaporation of acetone/CH_2Cl_2 solutions (1/1) of complexes **9**–**12** in NMR tube. The principal crystallographic data and refinement parameters are listed in Tables 1 and 2. X-ray diffraction experiment was carried out with a Bruker Apex II CCD area detector, using graphite monochromated Mo K_α radiation ($\lambda = 0.71073$ Å) at 100 K. Absorption correction was applied semi-empirically using APEX2 program [23]. The structures were solved by direct methods and refined by the full-matrix least-squares against F^2 in an anisotropic (for non-hydrogen atoms) approximation. The compounds **5**–**8** crystallize with two independent molecules in the unit cell which are differ from each other by orientation of the cyclic ligands. The compounds **9**–**12** crystallize with one CH_2Cl_2 solvate molecule. All hydrogen atom positions were refined in isotropic approximation in "riding" model with the $U_{iso}(H)$ parameters equal to 1.2 $U_{eq}(C_i)$, for methyl groups equal to 1.5 $U_{eq}(C_{ii})$, where $U(C_i)$ and $U(C_{ii})$ are respectively the equivalent thermal parameters of the carbon atoms to which the corresponding H atoms are bonded. All calculations were performed using the SHELXTL software [24].

Table 1
Summary of crystallographic data and structure refinement for complexes **5**–**8**.

	5	**6**	**7**	**8**
Empirical formula	$C_{15}H_{26}B_2Cl_2CoORhS$	$C_{15}H_{26}B_2Cl_2CoIrOS$	$C_{15}H_{26}B_2Br_2CoORhS$	$C_{15}H_{26}B_2Br_2CoIrOS$
Molecular weight	508.78	598.07	597.70	686.99
Crystal system	Orthorhombic	Orthorhombic	Orthorhombic	Orthorhombic
Space group	*Pca2₁*	*Pca2₁*	*Pca2₁*	*Pca2₁*
Crystal colour, habit	pyramid/black	pyramid/black	pyramid/black	pyramid/black
Crystal size (mm)	0.25 × 0.19 × 0.11	0.19 × 0.17 × 0.08	0.45 × 0.40 × 0.30	0.32 × 0.20 × 0.16
a (Å)	28.2721(17)	28.3157(8)	28.5751(12)	28.5980(9)
b (Å)	9.4959(6)	9.5197(3)	9.7398(4)	9.7591(3)
c (Å)	14.3224(8)	14.3176(4)	14.3874(6)	14.3633(4)
α (°)	90	90	90	90
β (°)	90	90	90	90
γ (°)	90	90	90	90
V (Å^3)	3845.1(4)	3859.4(2)	4004.2(3)	4008.7(2)
Z	8	8	8	8
D_{calc} (g cm^{-3})	1.758	2.059	1.983	2.277
$2\theta_{\max}$ (°)	28.50	28.00	27.00	28.00
Abs. coeff., μ(Mo-*K*α) (cm^{-1})	2.106	8.132	5.747	11.550
Absorption correction	Sadabs			
$T_{\max}/T_{\min}$	0.8014/0.6210	0.5623/0.3072	0.2775/0.1819	0.2594/0.1194
Number of collected reflections	44040	127333	39980	112191
Number of independent reflections	9749	9313	8629	4920
Number of observed reflections ($I > 2\sigma(I)$)	7646	8778	8387	4857
R_{int}	0.0942	0.0498	0.0247	0.0513
Number of parameters	430	429	429	430
R_1 (on F for observed reflexions)[a]	0.0405	0.0159	0.0168	0.0175
wR_2 (on F^2 for all reflexions)[b]	0.0766	0.0358	0.0404	0.0415
Weighting scheme	$w^{-1} = \sigma^2(F_o^2) + (aP)^2 + bP$, where $P = 1/3(F_o^2 + 2F_c^2)$			
A	0.02	0.01	0.009	0.014
B	0.0	1.5	0.44	15.99
F(000)	2048	2304	2336	2592
Goodness-of-fit	1.003	1.034	1.006	1.002
$\Delta\rho_{\max}/\Delta\rho_{\max}$ (e Å^{-3})	0.705/-0.986	1.442/-1.083	1.528/-0.462	3.810/-1.122

Table 2
Summary of crystallographic data and structure refinement for complexes **9**–**12**.

	9·CH_2Cl_2	**10**·CH_2Cl_2	**11**·CH_2Cl_2	**12**·CH_2Cl_2
Empirical formula	$C_{31}H_{35}B_2Cl_2CoPRh \cdot CH_2Cl_2$	$C_{31}H_{35}B_2Cl_2CoIrP \cdot CH_2Cl_2$	$C_{31}H_{35}B_2Br_2CoPRh \cdot CH_2Cl_2$	$C_{31}H_{35}B_2Br_2CoIrP \cdot CH_2Cl_2$
Molecular weight	777.85	867.14	866.77	956.06
Crystal system	Monoclinic	Monoclinic	Monoclinic	Monoclinic
Space group	*P*2₁/*c*	*P*2₁/*c*	*P*2₁/*n*	*P*2₁/*n*
Crystal colour, habit	plate/light brown	plate/light brown	plate/light brown	plate/light brown
Crystal size (mm)	0.59 × 0.45 × 0.18	0.58 × 0.45 × 0.32	0.33 × 0.21 × 0.09	0.41 × 0.32 × 0.14
a (Å)	12.8082(17)	12.8536(16)	9.7742(17)	9.779(2)
b (Å)	12.8350(17)	12.8219(16)	17.788(3)	17.809(4)
c (Å)	20.068(3)	20.091(3)	19.419(3)	19.360(4)
α (°)	90	90	90	90
β (°)	91.410(2)	92.108(2)	102.608(2)	102.774(3)
γ (°)	90	90	90	90
V (Å^3)	3298.1(8)	3309.0(8)	3294.8(9)	3288.3(13)
Z	4	4	4	4
D_{calc} (g cm^{-3})	1.567	1.741	3294.8(9)	1.931
$2\theta_{\max}$ (°)	28.00	28.00	30.50	27.10
Abs. coeff., μ(Mo-*K*α) (cm^{-1})	1.398	4.912	3.663	7.211
Absorption correction	Sadabs			
$T_{\max}/T_{\min}$	0.7870/0.4926	0.2024/0.0830	0.734/0.377	0.431/0.156
Number of collected reflections	43216	43241	50866	40390
Number of independent reflections	7948	7967	9976	7169
Number of observed reflections ($I > 2\sigma(I)$)	7053	7089	8447	5805
R_{int}	0.0285	0.0378	0.0387	0.0595
Number of parameters	375	373	370	370
R_1 (on F for observed reflexions)[a]	0.0230	0.0274	0.0228	0.0253
wR_2 (on F^2 for all reflexions)[b]	0.0567	0.0727	0.0538	0.0566
Weighting scheme	$w^{-1} = \sigma^2(F_o^2) + (aP)^2 + bP$, where $P = 1/3(F_o^2 + 2F_c^2)$			
A	0.023	0.043	0.023	0.018
B	3.03	5.32	1.380	6.69
F(000)	1576	1704	1720	1848
Goodness-of-fit	1.000	0.957	1.025	0.966
$\Delta\rho_{\max}/\Delta\rho_{\max}$ (e Å^{-3})	0.489/-0.436	1.968/-0.969	0.521/-0.532	0.958/-0.638

[a] $R_1 = \sum|F_o| - |F_c||/\sum(F_o)$

[b] $wR_2 = \{\sum[w(F_o^2 - F_c^2)^2]/\sum w(F_o^2)^2\}^{1/2}$.

Appendix A. Supplementary material

Supplementary data related to this article can be found online at http://dx.doi.org/10.1016/j.jorganchem.2012.06.003.

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