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Arene complexes $[(\eta\text{-}C_5H_5)M(\eta\text{-}C_6R_6)]^{2+}$ (M = Rh, Ir)

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The dicationic arene complexes $[CpM(arene)](BF_4)_2$ (arene = C_6H_6, 1,3,5-$C_6H_3Me_3$, or C_6Me_6) were synthesized by the reactions of the solvated complexes $[CpM(MeNO_2)_3](BF_4)_2$ (M = Rh, Ir) with benzene and its derivatives. The solvated complexes were generated *in situ* by abstraction of I^- from $[CpMI_2]_2$ with $AgBF_4$. A procedure was developed for the synthesis of the iodide $[CpRhI_2]_2$ based on the reaction of the cyclooctadiene derivative $CpRh(1,5\text{-}C_8H_{12})$ with I_2. The structure of the $[CpRh(C_6Me_6)](BF_4)_2$ complex was established by X-ray diffraction analysis.

Key words: sandwich compounds, arene complexes, rhodium, iridium.

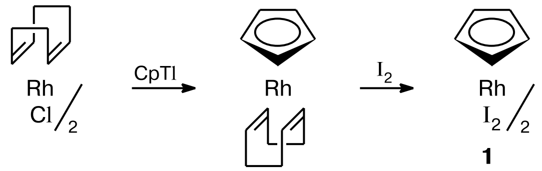
The dicationic rhodium and iridium arene complexes $[Cp^*M(arene)]^{2+}$ ($Cp^* = C_5Me_5$) are known for more than 30 years.[1] These complexes have attracted attention because of high activity of the arene ligand toward nucleophilic reagents. In particular, it was suggested[2] to use these compounds as catalysts for the nucleophilic substitution of the fluorine atom in the benzene ring and as reagents in a closed cycle of reduction of benzene to cyclohexene.[3] The main procedure for the synthesis of the $[Cp^*M(arene)]^{2+}$ cations (M = Rh, Ir) involves the reaction of arenes with the solvated complexes $[Cp^*M(Solv)_3]^{2+}$ (generally, Solv = Me_2CO), which are prepared by the reactions of silver salts with the chlorides $[Cp^*MCl_2]_2$.[4—6] This method was used for the synthesis of rhodium and iridium arene complexes with aryl-substituted amino acids[7] and iridium complexes with corannulenes.[8] We have demonstrated[9—11] that the $[Cp^*M(arene)]^{2+}$ cations can be synthesized without the use of silver salts by the reactions of chloride complexes with arenes in the presence of protic acids. This method has successfully been used for the preparation of compounds containing a hydrocarbon bridge between cyclic ligands.[12—14]

Unlike the $[Cp^*M(arene)]^{2+}$ cations, their analogs containing the unsubstituted cyclopentadienyl ring, *viz.*, $[CpM(arene)]^{2+}$ ($Cp = C_5H_5$), remain insufficiently studied. In the present study, we developed a procedure for the synthesis of such complexes and established the structure of the hexamethylbenzene derivative of rhodium by X-ray diffraction analysis.

Results and Discussion

The main reason for the fact that the arene complexes $[CpM(arene)]^{2+}$ (M = Rh, Ir) are poorly studied is the lack of convenient procedures for the synthesis of the starting halide derivatives $[CpMX_2]_2$. The rhodium chloride complex $[CpRhCl_2]_2$ has been prepared earlier[15] by the reaction of $RhCl_3 \cdot 3H_2O$ with cyclopentadiene (55—60%) or by the reaction of the allyl complex $[(\eta^3\text{-}C_3H_5)_2RhCl]_2$ with CpTl followed by treatment of the $CpRh(\eta^3\text{-}C_3H_5)(\eta^1\text{-}C_3H_5)$ complex that formed with hydrochloric acid (63%).[16] The $[CpRhBr_2]_2$ and $[CpRhI_2]_2$ compounds (**1**) were synthesized by the reactions of $CpRh(\eta^4\text{-}C_5H_5Ph)$ with halogens.[17] Instead of the phenylcyclopentadiene complex, the cyclooctadiene analog $CpRh(1,5\text{-}C_8H_{12})$ was successfully used in the synthesis of iodide **1** (Scheme 1). This analog was prepared by the reaction of $[(1,5\text{-}C_8H_{12})RhCl]_2$ with CpNa [18] or CpTl and can be used in the reaction with I_2 without isolation in the individual state. Using CpTl, compound **1** was prepared in a total yield of 97%. Taking into account the fact that the starting $[(1,5\text{-}C_8H_{12})RhCl]_2$ complex was prepared by the reaction of $RhCl_3 \cdot 3H_2O$ with cyclooctadiene in 94% yield,[19] compound **1** becomes readily accessible. Recently,[20] we have also described a convenient procedure for the synthesis of the iridium analog $[CpIrI_2]_2$ (**2**).

Scheme 1



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The reaction of the solvated complexes $[CpM(MeNO_2)_3]^{2+}$ (M = Rh, Ir) with benzene and its

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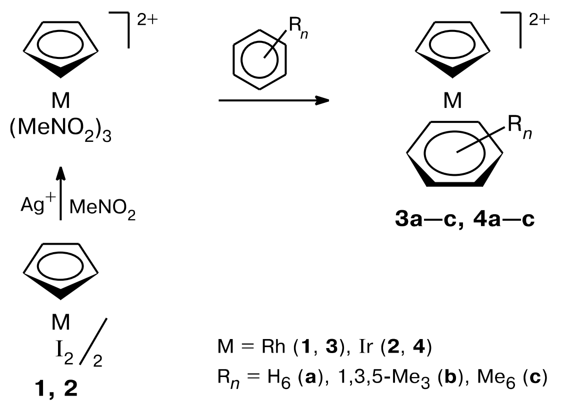
derivatives afforded dicationic arene complexes **3a—c** and **4a—c**, which were isolated as tetrafluoroborates in 50—70% yields (Scheme 2). The solvated complexes were generated *in situ* by abstraction of I^- from **1** and **2** using $AgBF_4$ in nitromethane. In the case of rhodium, the chloride complex $[CpRhCl_2]_2$ was equally successfully used instead of compound **1**. Cations **3a** and **3c** have been characterized earlier. The benzene complex **3a** has been synthesized by elimination of H^- from the cyclohexadiene derivative $CpRh(\eta^4\text{-}C_6H_8)$ under the action of $[Ph_3C]^+$.[21] Hexamethylbenzene analog **3c** has been prepared by the exchange of arene in **3a**.[22]

Scheme 2



It is known that the rhodium complexes $[Cp^*Rh(arene)]^{2+}$, unlike their iridium analogs, readily exchange the arene ligand for the solvent molecules (Me_2CO, MeCN, or Me_2SO).[1,4] The compounds synthesized in the present study behave analogously. In the ^1H NMR spectrum of complex **3a** in acetone-d_6, the signal of the coordinated benzene completely disappeared after 2 h, which indicates that the benzene is replaced with acetone molecules. An analogous reaction with acetonitrile was completed in 15 min to give the acetonitrile complex $[CpRh(MeCN)_3]^{2+}$, which was identified by ^1H NMR spectroscopy.* By contrast, iridium analog **4a** is stable in acetonitrile for at least one week.

In the ^1H NMR spectra of complexes **3a—c** and **4a—c**, as in the spectra of the C_5Me_5 analogs,[4] the signals for the protons of the arene ligand are shifted downfield relative to the free arene (Table 1). This is a consequence of a high positive charge, whose effect overrides the opposite effect of coordination to the transition metal atom.

Table 1. Parameters of the ^1H NMR spectra of cations **3a—c** and **4a—c** in CD_3NO_2

Cation	δ		
	Cp	$H_{b.r}$	Me
3a	6.88 (5 H)	7.71 (6 H)	—
3b	6.72 (5 H)	7.48 (3 H)	2.71 (9 H)
3c	6.50 (5 H)	—	2.78 (18 H)
4a	6.84 (5 H)	7.69 (6 H)	—
4b	6.71 (5 H)	7.55 (3 H)	2.86 (9 H)
4c	6.53 (5 H)	—	2.83 (18 H)

Note. All signals are singlets; $H_{b.r}$ are the protons of the benzene ring.

Table 2. Selected bond lengths (*d*) in cation **3c**

Bond	*d*/Å	Bond	*d*/Å
Rh(1)—C(1c)	2.116(6)	C(1c)—C(2c)	1.373(6)
Rh(1)—C(2c)	2.089(6)	C(2c)—C(3c)	1.373(6)
Rh(1)—C(3c)	2.095(7)	C(3c)—C(4c)	1.373(6)
Rh(1)—C(4c)	2.126(7)	C(4c)—C(5c)	1.373(6)
Rh(1)—C(5c)	2.139(6)	C(1c)—C(5c)	1.373(6)
Rh(1)—C(1)	2.204(4)	C(1)—C(2)	1.432(6)
Rh(1)—C(2)	2.214(5)	C(2)—C(3)	1.436(5)
Rh(1)—C(3)	2.217(5)	C(3)—C(4)	1.420(6)
Rh(1)—C(4)	2.208(4)	C(4)—C(5)	1.424(5)
Rh(1)—C(5)	2.210(4)	C(5)—C(6)	1.427(5)
Rh(1)—C(6)	2.207(5)	C(1)—C(6)	1.415(6)

Structure of cation 3c. The structure of the cationic complex $\mathbf{3c}(BF_4)_2$ is composed of isolated cations **3c** and the BF_4^- anions, one of two BF_4^- anions being disordered (two fluorine atoms each occupy two positions with occupancies of 0.5). The structure of the cation is shown in Fig. 1. The main bond lengths are given in Table 2. The planes of the cyclic ligands are coplanar (Cp/C_6Me_6 dihedral angle is 0.4°). The $Rh—C(C_6Me_6)$ bonds (2.204(4)—2.217(5) Å; aver., 2.210 Å) are substantially longer than the Rh—C(Cp) bonds (2.089(6)—2.139(6) Å;

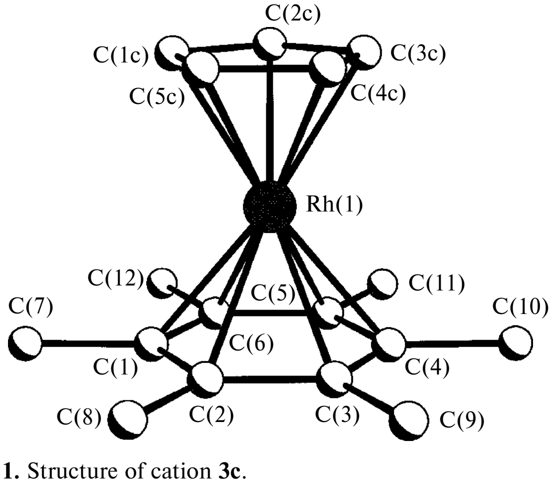


Fig. 1. Structure of cation **3c**.

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* ^1H NMR (CD_3NO_2), δ: 2.54 (s, 9 H, MeCN); 6.42 (s, 5 H, Cp) (*cf.* lit. data[4] for $[Cp^*Rh(MeCN)_3]^{2+}$).

Table 3. Distances from the rhodium atom to the planes of the cyclic ligands (Δ/Å) in cation **3c** and related complexes

Cation	Refcode*	Δ(Rh...Cp)	Δ(Rh...arene)
3c		1.760(4)	1.689(2)
$[Cp^*Rh(C_6H_5Me)]^{2+}$ [23]	MIGMIY	1.791	1.766
$[Cp^*Rh(C_6H_5NH_2)]^{2+}$ [24]	TAPWIQ	1.789	1.758
$[Cp^*Rh(C_6H_4(OH)CH_2CHRR')]^{2+}$ (R = C(O)OEt, R′ = NHC(O)Me)[7]	VEDQEA	1.809	1.776
$[Cp^*Rh(C_6H_4(OH)CH_2CHRR')]^{2+}$ (R = C(O)OH, R′ = NHC(O)Me)[7]	VEDQIE	1.799	1.782
$Cp^*Rh(pcp)]^{2+}$ (pcp is [2.2]paracyclophane)[25]	BAHKUR	1.785	1.805
$[Cp^*Rh(C_6H_5R)]^{2+}$ (R = $(BC_4H_4)RhCp$)**	—	1.789	1.760
$[Cp_2Rh]^+$ [26]	KIWZEV	1.806, 1.810	—
$[Cp^*{}_2Rh]^+$ [27]	FOQBAO	1.814	—
$[(C_5H_4R)_2Rh]^+$ (R = $C_2H_4NMe_2$)[28]	RAGGIP	1.800, 1.803	—
$[(C_5H_4R)_2Rh]^+$ (R = $CPh_2COOCPh_2$)[29]	BOBZIB	1.797, 1.792	—
$[(C_5H_2R_3)_2Rh]^+$ (R = 2,3,5-C(O)OMe)[30]	BASXAU	1.800, 1.799	—
$[(C_5HPh_4)_2Rh]^+$ [31]	NUZSUW	1.851, 1855	—

* The reference code in the Cambridge Structural Database.

** D. A. Loginov, D. V. Muratov, P. V. Petrovskii, Z. A. Starikova, and A. R. Kudinov, unpublished data.

aver., 2.113 Å), which is, apparently, indicative of a weaker bonding between the rhodium atom and the arene ligand compared to the bonding with the cyclopentadienyl ligand. This is consistent with the fact that the arene ligand in the $[CpRh(arene)]^{2+}$ complexes is easily displaced under the action of Me_2CO or MeCN.

To reveal the general structural features of such complexes, we analyzed the structures of the related sandwich Rh^{III} complexes available in the Cambridge Structural Database* (Table 3). In most complexes, the Rh atom is located above/below the centroids of the rings and, consequently, the distances from the metal atom to the planes of the rings, *viz*., Δ(Rh...Cp) and Δ(Rh...arene), are consistent with the Rh...centroid distances and correlate with the Rh—C(Cp) and Rh—C(arene) bond lengths. Hence, the Δ(Rh...Cp) and Δ(Rh...arene) distances can be considered as reliable characteristics of the strength of bonding between the Rh atom and the rings. It appeared that the Δ(Rh...Cp) distance in **3c** (1.760 Å) is shorter than the analogous distances in the $[Cp_2Rh]^+$ complex (1.806 and 1.810 Å). An analogous situation is observed for the compounds containing the C_5Me_5 ligand: Δ(Rh...Cp*) in the $[Cp^*Rh(arene)]^{2+}$ complexes are shorter than those in $[Cp^*{}_2Rh]^+$ (see Table 3). Apparently, this is associated with a nonuniform electron density distribution in the $[(C_5R_5)Rh(arene)]^{2+}$ complexes due to the different nature of the ligands. Interestingly, the Δ(Rh...C_5R_5) distance in a series of the bis(cyclopentadienyl) derivatives $[(C_5R_5)_2Rh]^+$ varies relatively little (1.797—1.814 Å). This distance is substantially longer (1.856 Å) only in the sterically hindered $[(C_5HPh_4)_2Rh]^+$ compound. The Δ(Rh...arene) distance in the $[Cp^*Rh(arene)]^{2+}$ complexes (see Table 3) varies over a wider range (1.758—1.805 Å) and, apparently, depends to a larger extent on the donor-acceptor properties of the substituents. In cation **3c**, this distance is substantially shortened (1.689(2) Å).

Experimental

The reactions were carried out under argon with the use of anhydrous solvents, which were purified according to standard procedures. Operations associated with isolation of the reaction products were performed in air. The starting $[(1,5\text{-}C_8H_{12})RhCl]_2$,[19] $[CpIrI_2]_2$,[20] and $AgBF_4 \cdot 3dioxane$[32] compounds were synthesized according to known procedures. The ^1H NMR spectra were recorded on a Bruker AMX-400 instrument relative to residual protons of the solvent.

Bis[diiodo(η-cyclopentadienyl)rhodium] $[CpRhI_2]_2$ (1). Tetrahydrofuran (7 mL) was added to a mixture of the $[(1,5\text{-}C_8H_{12})RhCl]_2$ complex (150 mg, 0.304 mmol) and CpTl (172 mg, 0.638 mmol). The reaction mixture was stirred for 2.5 h. The solvent was removed *in vacuo* and the solid residue was extracted with light petroleum. After removal of the

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* November 2002 release.

solvent, the CpRh(1,5-C_8H_{12}) complex was obtained as a yellow solid compound. The product was dissolved in CH_2Cl_2 (5 mL). Then a solution of I_2 (154 mg, 0.607 mmol) in the same solvent (5 mL) was added and the reaction mixture was stirred for 2 h. The precipitate that formed was filtered off and washed with acetone to give a brown solid compound in a yield of 250 mg (97%). Found (%): C, 14.28; H, 1.21. $C_{10}H_{10}I_4Rh_2$. Calculated (%): C, 14.24; H, 1.19. ^{1}H NMR (DMSO-d_6), δ: 6.09 (s, Cp).

Synthesis of the [CpM(arene)]$(BF_4)_2$ complexes (M = Rh, Ir) (general procedure). Nitromethane (1 mL) was added to a mixture of the $[CpMI_2]_2$ complex (0.073 mmol) and $AgBF_4 \cdot 3$ dioxane (0.294 mmol). The reaction mixture was stirred for ~1 h. The precipitate of AgI that formed was removed by centrifugation and an excess of the arene (0.7 mL of C_6H_6 or 1,3,5-$C_6H_3Me_3$, or 120 mg of C_6Me_6) was added to the solution. The reaction mixture was stirred for ~12 h and concentrated *in vacuo*. The residue was washed with acetone (3×1 mL) and reprecipitated from nitromethane with diethyl ether. White or cream-colored solid compounds were isolated.

[(η-Cyclopentadienyl)(η-benzene)rhodium]bis(tetrafluoroborate) (3a), M = Rh, arene = C_6H_6, the yield was 58%. Found (%): C, 30.90; H, 2.46. $C_{11}H_{11}B_2F_8Rh$. Calculated (%): C, 31.48; H, 2.64.

[(η-Cyclopentadienyl)(η-mesitylene)rhodium]bis(tetrafluoroborate) (3b), M = Rh, arene = 1,3,5-$C_6H_3Me_3$, the yield was 66%. Found (%): C, 35.44; H, 3.58. $C_{14}H_{17}B_2F_8Rh$. Calculated (%): C, 36.41; H, 3.71.

[(η-Cyclopentadienyl)(η-hexamethylbenzene)rhodium]bis(tetrafluoroborate) (3c), M = Rh, arene = C_6Me_6, the yield was 63%. Found (%): C, 40.36; H, 4.41. $C_{17}H_{23}B_2F_8Rh$. Calculated (%): C, 40.52; H, 4.60.

[(η-Cyclopentadienyl)(η-benzene)iridium]bis(tetrafluoroborate) (4a), M = Ir, arene = C_6H_6, the yield was 76%. Found (%): C, 25.81; H, 2.29. $C_{11}H_{11}B_2F_8Ir$. Calculated (%): C, 25.95; H, 2.18.

[(η-Cyclopentadienyl)(η-mesitylene)iridium]bis(tetrafluoroborate) (4b), M = Ir, arene = 1,3,5-$C_6H_3Me_3$, the yield was 49%. Found (%): C, 30.45; H, 3.09. $C_{14}H_{17}B_2F_8Ir$. Calculated (%): C, 30.51; H, 3.11.

[(η-Cyclopentadienyl)(η-hexamethylbenzene)iridium]bis(tetrafluoroborate) (4c), M = Ir, arene = C_6Me_6, the yield was 53%. Found (%): C, 34.27; H, 3.81. $C_{17}H_{23}B_2F_8Ir$. Calculated (%): C, 34.42; H, 3.91.

X-ray diffraction study of the [3c]$(BF_4)_2$ complex. Yellow prismatic crystals of composition $C_{17}H_{23}B_2F_8Rh$ were grown up by crystallization from $MeNO_2/Et_2O$. The crystals belong to the monoclinic system. The unit cell parameters: a = 9.234(2) Å, b = 12.913(3) Å, c = 9.237(2) Å, β = 120.00(3)°, V = 953.8(3) Å^3, space group $P2_1$, Z = 4, d_{calc} = 1.754 g cm^{-3}. A total of 7885 reflections were collected on a Bruker SMART 1000 CCD diffractometer at 100 K (Mo-K_α radiation, $2\theta_{max}$ = 63.84°) from a single crystal of dimensions 0.5×0.3×0.3 mm. Merging of the equivalent reflections gave 4068 independent reflections (R_{int} = 0.0350), which were used in the structure solution and refinement. The absorption correction (μ = 0.970 mm^{-1}) was applied using the SADABS program (T_{max} and T_{min} are 0.928 and 0.442, respectively). The structure was solved by direct methods. All nonhydrogen atoms were revealed from difference electron density maps and refined anisotropically against F^2_{hkl}. All hydrogen atoms were placed in geometrically calculated positions and refined using the riding model with $U(H) = n \cdot U(C)$, where U(C) is the equivalent thermal parameters of the corresponding pivot carbon atoms, n = 1.2 and 1.5 for the atoms of the Cp ring and Me groups, respectively. The final reliability factors were R_1 = 0.0505 (calculated based on F_{hkl} for 3330 reflections with $I > 2\sigma(I)$), wR_2 = 0.1110 (calculated based on F^2_{hkl} for a total of 4068 reflections), 265 parameters were refined, GOOF = 0.925. All calculations were carried out using the SHELXTL PLUS 5 program package.[33] The atomic coordinates and thermal parameters were deposited with the Cambridge Structural Database.

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References

1. C. White and P. M. Maitlis, *J. Chem. Soc., A*, 1971, 3322.
2. R. P. Houghton and M. Voyle, *J. Chem. Soc., Chem. Commun.*, 1980, 884.
3. S. L. Grundy, A. J. Smith, H. Adams, and P. M. Maitlis, *J. Chem. Soc., Dalton Trans.*, 1984, 1747.
4. C. White, S. J. Thompson, and P. M. Maitlis, *J. Chem. Soc., Dalton Trans.*, 1977, 1654.
5. P. Espinet, P. M. Bailey, R. F. Downey, and P. M. Maitlis, *J. Chem. Soc., Dalton Trans.*, 1980, 1048.
6. S. L. Grundy and P. M. Maitlis, *J. Organomet. Chem.*, 1984, **272**, 265.
7. D. A. Herebian, C. S. Schmidt, W. S. Sheldrick, and C. van Wüllen, *Eur. J. Inorg. Chem.*, 1998, 1991.
8. C. M. Alvarez, R. J. Angelici, A. Sygula, R. Sygula, and P. W. Rabideau, *Organometallics*, 2003, **22**, 624.
9. M. I. Rybinskaya, A. R. Kudinov, and V. S. Kaganovich, *J. Organomet. Chem.*, 1983, **246**, 279.
10. M. I. Rybinskaya, V. S. Kaganovich, and A. R. Kudinov, in *Advances in Organometallic Chemistry*, Ed. O. A. Reutov, MIR Publishers, Moscow, 1984, p. 117.
11. V. S. Kaganovich, A. R. Kudinov, and M. I. Rybinskaya, *Metalloorg. Khim.*, 1988, **1**, 294 [*Organomet. Chem. USSR*, 1988, **1**, 162 (Engl. Transl.)].
12. V. S. Kaganovich, A. R. Kudinov, and M. I. Rybinskaya, *J. Organomet. Chem.*, 1987, **323**, 111.
13. P. O. Lumme, U. Turpeinen, V. S. Kaganovich, A. R. Kudinov, and M. I. Rybinskaya, *J. Organomet. Chem.*, 1988, **348**, 255.
14. P. O. Lumme, U. Turpeinen, V. S. Kaganovich, A. R. Kudinov, and M. I. Rybinskaya, *Acta Crystallogr.*, 1989, **C45**, 559.
15. J. W. Kang, K. Moseley, and P. M. Maitlis, *J. Am. Chem. Soc.*, 1969, **91**, 5970.
16. J. Powell and B. L. Shaw, *J. Chem. Soc., A*, 1968, 583.
17 R. J. Angelici and E. O. Fischer, *J. Am. Chem. Soc.*, 1963, **85**, 3733.
18 J. Chatt and L. M. Venanzi, *J. Chem. Soc.*, 1957, 4735.
19 G. Giordano and R. H. Crabtree, *Inorg. Synth.*, 1979, **19**, 218.

20. A. R. Kudinov, D. A. Loginov, Z. A. Starikova, and P. V. Petrovskii, *J. Organomet. Chem.*, 2002, **649**, 136.
21. B. F. G. Johnson, J. Lewis, and D. J. Yarrow, *J. Chem. Soc., Dalton Trans.*, 1972, 2084.
22. W. J. Bowyer, J. W. Merkert, and W. E. Geiger, *Organometallics*, 1989, **8**, 191.
23. M. Maekawa, N. Hashimoto, T. Kuroda-Sowa, Y. Suenaga, and M. Munakata, *Anal. Sci.*, 2001, **17**, 1361.
24. S. Ogo, H. Chen, M. M. Olmstead, and R. H. Fish, *Organometallics*, 1996, **15**, 2009.
25. M. Maekawa, N. Hashimoto, T. Kuroda-Sowa, Y. Suenaga, and M. Munakata, *Inorg. Chim. Acta*, 2002, **328**, 254.
26. B. Klingert and G. Rihs, *J. Inclus. Phenom. Mol.*, 1991, **10**, 255.
27. D. Buchholz, L. Zsolnai, G. Huttner, and D. Astruc, *J. Organomet. Chem.*, 2000, **593—594**, 494.
28. A. I. Philippopoulos, N. Hadjiliadis, C. E. Hart, B. Donnadieu, P. C. Mc Gowa, and R. Poilblanc, *Inorg. Chem.*, 1997, **36**, 1842.
29. J. Jeffery, R. J. Mawby, M. B. Hursthouse, and N. P. C. Walker, *J. Chem. Soc., Chem. Commun.*, 1982, 1411.
30. M. I. Bruce, J. R. Rodgers, and J. K. Walton, *J. Chem. Soc., Chem. Commun.*, 1981, 1253.
31. I. A. Guzei, A. L. Rheingold, J. E. Collins, and M. P. Castellani, *Z. Krist.-New Cryst. St.*, 1998, **213**, 431.
32. M. E. Woodhouse, F. D. Lewis, and T. J. Marks, *J. Am. Chem. Soc.*, 1982, **104**, 5586.
33. G. M. Sheldrick, *SHELXTL Ver. 5.0*, *Software Reference Manual*, Siemens Industrial Automation, Inc., Madison, 1994.

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