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Silver(I) complexes of bis[2-(diphenylphosphino)phenyl] ether

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Abstract

The reaction of AgOTf in dichloromethane with bis(2-(diphenylphosphino)phenyl) ether (DPEphos) in an equimolar ratio afforded a dinuclear complex [$Ag_2(\kappa^2$-*P*,*P*′-DPEphos$)_2(\mu$-OTf$)_2$] (**1**), whereas the similar reaction in a 1:2 molar ratio resulted in the formation of a bis-chelating complex [Ag(κ^2-*P*,*P*′-DPEphos$)_2$][OTf] (**2**). The silver(I) complex **1** was obtained as a dimer, in which two silver atoms are bridged by two triflate groups to form three adjacent eight-membered spirocyclic rings. The mixed-ligand complex [Ag(κ^2-*P*,*P*′-DPEphos)(2,2′-bpy)][OTf] (**3**) was obtained in the reaction of **1** in dichloromethane with 2,2′-bipyridine. The crystal structures of complexes **1**–**3** were determined by single crystal X-ray analyses.

Keywords: Mixed-ligand silver complexes; DPEphos; 2,2′-Bipyridine; Chelating complexes; Crystal structures

1. Introduction

The coordination chemistry of bis(2-(diphenylphosphino)phenyl) ether (DPEphos) is interesting due to its versatile coordination behavior and its catalytic utility in a variety of organic transformations. The tridentate nature of this ligand produces interesting coordination architectures, often the ether-O donor being hemilabile in nature. van Leeuwan and co-workers [1] and others [2] have extensively studied the transition metal chemistry and catalytic utility of DPEphos [3]. We recently reported the synthesis of Ru^{II} [4] and Cu^{I} [5] complexes of DPEphos and their application in catalytic hydrogenation of styrenes. As a part of our interest [6] herein we report for the first time, the synthesis, reactivity and crystal structures of Ag^{I} complexes containing DPEphos.

2. Results and discussion

Treatment of AgOTf with DPEphos in 1:1 and 1:2 molar ratios at room temperature afford colorless crystalline solids [$Ag_2(\kappa^2$-*P*,*P*′-DPEphos$)_2(\mu$-OTf$)_2$] (**1**) and [Ag(κ^2-*P*,*P*′-DPEphos$)_2$][OTf] (**2**), respectively, in good yield. Complex **2** can be prepared by treating complex **1** with DPEphos in 0.5:1 stoichiometry as shown in Scheme 1.

The $^{31}P\{^1H\}$ NMR spectrum of complex **1** shows a broad doublet at −10.0 ppm with J_{AgP} coupling of 486.8 Hz at room temperature, whereas at −50 °C, the peaks resolve into two doublets centered at −10.4 ppm with J^{109}_{AgP} and J^{107}_{AgP} couplings of 526.8 and 456.7 Hz, respectively. This clearly indicates that both the phosphorus atoms in the DPEphos ligand are attached to silver(I) in a chelating fashion and are equivalent. The coordination chemical shift is 7.0 ppm. The $^{31}P\{^1H\}$ NMR spectrum of complex **2** consists of two doublets centered at −10.5 ppm with J^{109}_{AgP} and J^{107}_{AgP} couplings of 269.7 and 233.8 Hz, respectively. The J_{AgP} values are comparable with those reported in the literature for analogous complexes [7,8]. The EI mass spectrum of complex **2** shows a

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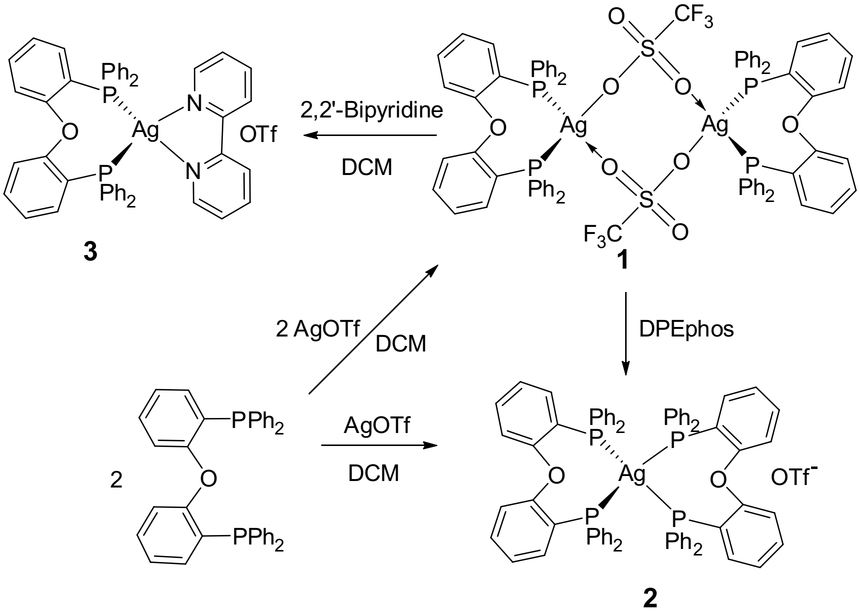

Scheme 1.

molecular ion peak corresponding to the cationic complex part at m/z 1185.46 $[M]^+$ with the appropriate isotopic pattern. This evidences that the Ag^I metal center is bis-chelated by DPEphos ligands to form a spirocyclic compound (Fig. 2). The microanalysis supports the structural composition of complex **2**, which is further confirmed by single crystal X-ray data.

The solid state structure of complex **1** shows the presence of a Ag^I complex as a dimer, in which DPEphos binds in a chelating mode whereas the two triflate ions are bridging the two silver atoms *via* O=S–O^- units, thus making a three eight-membered spirocyclic ring structure as shown in Fig. 1. The counter ion plays an important role in coordination with silver atoms, providing stability to complex **1**. Similarly, counter ions such as nitrate, acetate and perchlorate are also known to bridge silver atoms [9–11]. Although the triflate units anchor the silver(I) centers to stabilize complex **1**, they are labile and can be easily substituted with P- and N-donors. The mixed-ligand complex [Ag(κ^2-P,P'-DPEphos)(2,2′-bpy)][OTf] (**3**) was synthesized in good yield by the reaction of **1** with 2,2′-bipyridine in a 1:2 molar ratio at room temperature. The $^{31}P\{^1H\}$ NMR spectrum of complex **3** at room temperature exhibits a pair of doublets centered at −8.8 ppm with J^{109}_{AgP} and J^{107}_{AgP} values of 426 and 374 Hz, respectively. The ^1H NMR spectrum confirms the presence of 2,2′-bipyridine, which presents peaks at 8.04 and 8.35 ppm. The J_{AgP} values are in the range 370–430 Hz, which is in agreement with those values reported for analogous complexes [10,12].

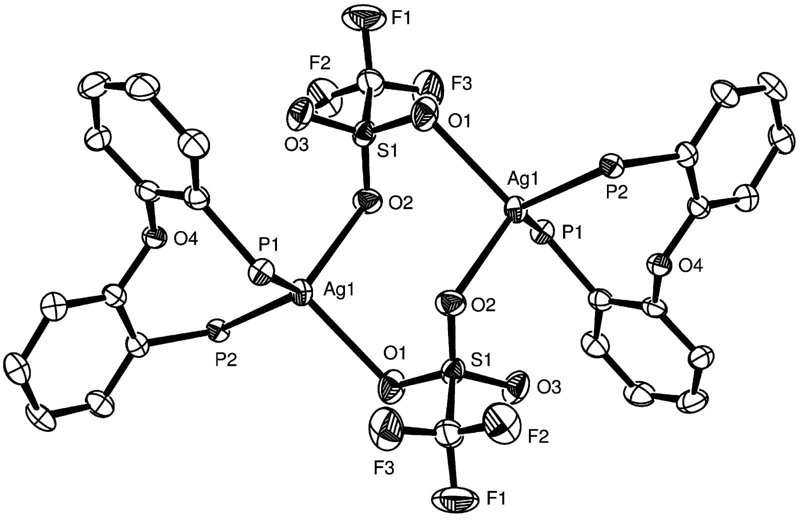

Fig. 1. Molecular structure of complex **1**. Thermal ellipsoids are drawn at the 50% probability level. Hydrogen atoms, phenyl rings attached to phosphorus and uncoordinated solvent molecules have been omitted for clarity.

*2.1. Crystal structures of complexes **1–3***

Complexes **1** and **2** were recrystallized from a mixture of CH_2Cl_2/Et_2O, and complex **3** was recrystallized from a $CHCl_3/Et_2O$ mixture. The molecular structures of complexes **1–3** are shown in Figs. 1–3, respectively, and selected bond parameters are displayed in Tables 1 and 2. The crystallographic information is given in Table 3.

Complex **1** crystallizes in the monoclinic crystal system with a center of symmetry and it is dimeric in nature. The Ag^I center possesses a distorted tetrahedral geometry, whose edges are shared by two phosphorus atoms of a DPEphos ligand and two oxygen atoms of triflate ions. Each silver atom is anchored to a chelating DPEphos and bridged by triflate units. The Ag1–O1, Ag1–O2, Ag1–P1 and Ag1–P2 bond distances are 2.553(2),

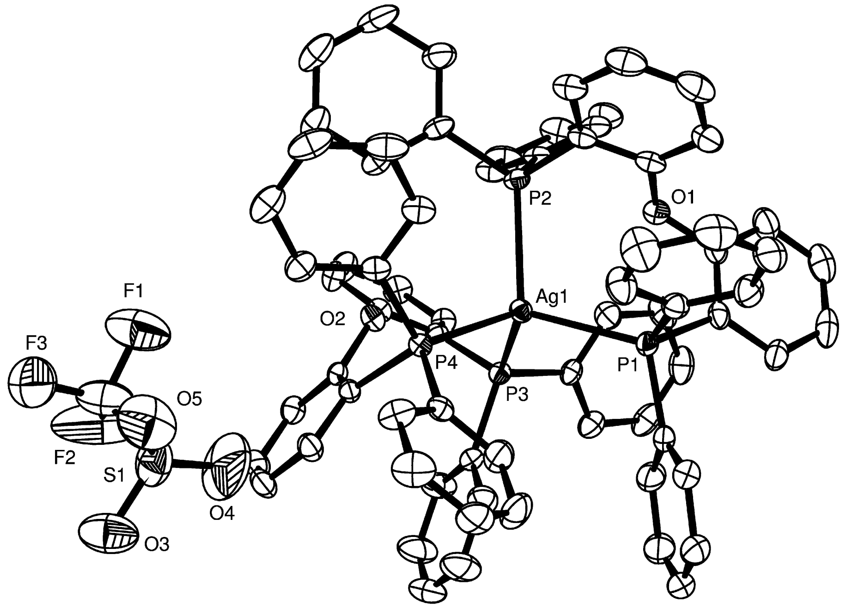

Fig. 2. Molecular structure of complex **2**. Thermal ellipsoids are drawn at the 30% probability level. Hydrogen atoms have been omitted for clarity.

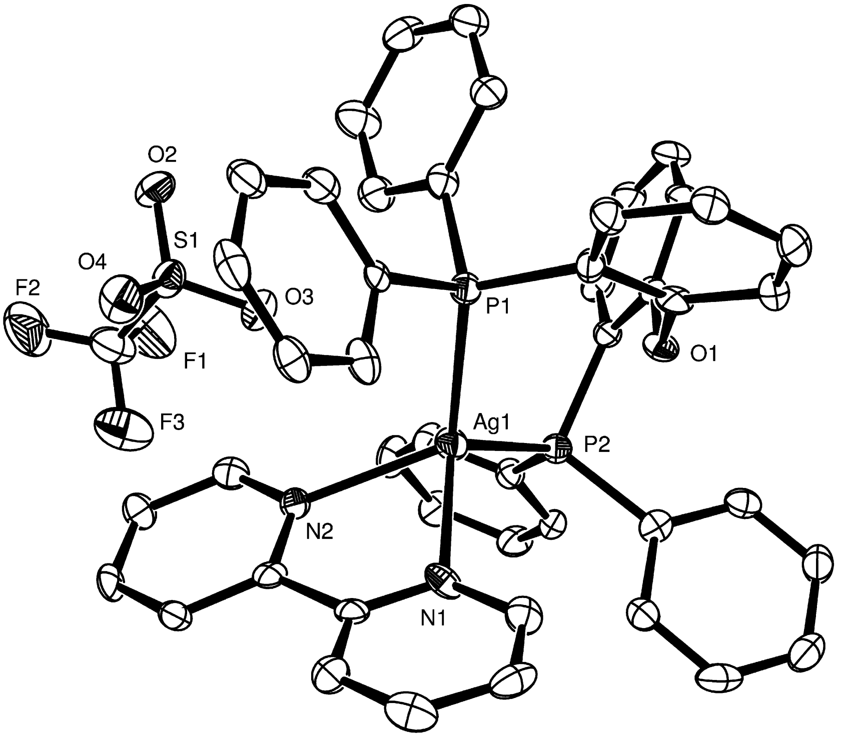

Fig. 3. Molecular structure of complex **3**. Thermal ellipsoids are drawn at the 30% probability level. Hydrogen atoms and uncoordinated solvent molecules have been omitted for clarity.

Table 1
Selected bond distances and bond angles for complexes **1** and **2**

Complex **1**				Complex **2**			
Bond distances (Å)		Bond angles (°)		Bond distances (Å)		Bond angles (°)	
Ag1–O2	2.411(2)	O2–Ag1–P2	111.52(6)	Ag1–P3	2.5385(7)	P3–Ag1–P4	106.37(2)
Ag1–P2	2.4383(9)	O2–Ag1–P1	113.49(6)	Ag1–P4	2.5461(7)	P3–Ag1–P1	107.47(2)
Ag1–P1	2.4521(8)	P2–Ag1–P1	117.40(3)	Ag1–P1	2.6119(7)	P4–Ag1–P1	115.38(2)
Ag1–O1	2.553(2)	O2–Ag1–O1	99.12(8)	Ag1–P2	2.6232(7)	P3–Ag1–P2	107.77(2)
S1–O3	1.426(2)	P2–Ag1–O1	104.07(6)	S1–O3	1.450(3)	P4–Ag1–P2	110.56(2)
S1–O1	1.436(3)	P1–Ag1–O1	109.13(6)	S1–C73	1.786(5)	P1–Ag1–P2	108.96(2)
S1–C1	1.815(3)	O3–S1–O1	115.41(15)	F1–C73	1.338(5)		
F1–C1	1.327(4)	S1–O1–Ag1	112.58(13)				

Table 2
Selected bond distances and bond angles for complex **3**

Complex **3**			
Bond distances (Å)		Bond angles (°)	
Ag1–N2	2.305(6)	N2–Ag1–N1	70.4(2)
Ag1–N1	2.355(6)	N2–Ag1–P2	126.98(16)
Ag1–P2	2.405(2)	N1–Ag1–P2	119.60(17)
Ag1–P1	2.488(2)	N2–Ag1–P1	110.69(15)
S1–O2	1.439(6)	N1–Ag1–P1	107.77(15)
F3–C47	1.317(11)	P2–Ag1–P1	113.51(7)
S1–C49	1.853(11)		

2.411(2), 2.453(3) and 2.438(2) Å, respectively. The Ag1–O2 bond distances of 0.142(2) Å is shorter than Ag1–O1, which indicates that the former one is an ionic bond and the latter one is a covalent bond. The bite angle of the DPEphos ligand is 117.40(3)°, which is 15.4° larger than its natural bite angle (β_n). Complex **2** crystallizes in the monoclinic crystal system and Ag^{I} exists in a distorted tetrahedral geometry. The silver(I) atom is attached to four phosphorus atoms of two DPEphos ligands. The Ag–P bond distances are 2.5385(7), 2.5460(7), 2.6118(7) and 2.6232(7) Å and are approximately 0.1–0.19 Å longer than the same distances in complex **1**. The bite angles of the two DPEphos ligands are 106.37(2)° and 108.96(2)°, which are 10.73° and 8.44° less when compared with the same angles in complex **1**.

In complex **3**, the Ag^{I} atom possesses a distorted tetrahedral geometry containing two phosphorus and two nitrogen atoms. The pyridyl units of the 2,2′-bipyridine ligand are not in a plane and are twisted by an angle of 14.50°. The Ag1–P1 bond (2.488(2) Å) is considerably longer than the Ag1–P2 bond (2.405(2) Å). The Ag1–N1 and Ag1–N2 bond distances are 2.305(6) and 2.355(6) Å, respectively. One set of diagonally opposite Ag–N and Ag–P bonds are longer than the other set of Ag–N and Ag–P bonds.

Table 3
Crystallographic data for complexes **1**–**3**

	1	**2**	**3**
Formula	$C_{38}H_{30}AgCl_2F_3O_4P_2S$	$C_{73}H_{56}AgF_3O_5P_4S$	$C_{48}H_{37}AgCl_3F_3N_2O_4P_2S$
F_w	880.39	1333.99	1071.02
Crystal system	monoclinic	monoclinic	triclinic
Space group	$P2_1/a$	$P2_1/c$	$P\bar{1}$
a (Å)	12.2506(15)	14.04760(10)	9.547(5)
b (Å)	26.477(5)	19.0664(2)	13.800(5)
c (Å)	12.5255(6)	23.1718	19.221(5)
α (°)	90	90	107.804(5)
β (°)	115.035(8)	91.6160(10)	94.406(5)
γ (°)	90	90	92.280(5)
V (Å^3)	3681.1(8)	6203.80(10)	2398.6(17)
Z	4	4	2
ρ_{calc} (g cm^{-3})	1.589	1.428	1.483
μ(Mo Kα) (mm^{-1})	0.892	0.524	0.754
F(000)	1776	2736	1084
Crystal size (mm)	0.33 × 0.26 × 0.21	0.26 × 0.21 × 0.18	0.08 × 0.17 × 0.23
T (K)	100(2)	150(2)	150(2)
2θ Range (°)	2.92–25.00	2.90–25.00	3.05–30.00
Total number reflections	20732	60904	8354
Number of independent reflections [R_{int}]	6460 [0.027]	10906 [0.0447]	3817 [0.089]
Goodness-of-fit (F^2)	1.074	1.051	0.842
R_1^{a}	0.0332	0.0358	0.0678
wR_2^{b}	0.0800	0.0850	0.1705

[a] $R = \sum||F_o| - |F_c||/\sum|F_o|$.

[b] $R_w = \{[\sum w(F_o^2 - F_c^2)/\sum w(F_o^2)^2]\}^{1/2}$ $w = 1/[\sigma^2(F_o^2) + (xP)^2]$ where $P = (F_o^2 + 2F_c^2)/3$.

A similar trend was observed in the analogous copper complex [Cu(κ^2-*P*,*P*′-DPEphos)(μ-4,4′-bpy)]$_n$[BF$_4$]$_n$, but to a lesser extent [5].

3. Conclusion

The silver(I) complexes [Ag$_2$(κ^2-*P*,*P*′-DPEphos)$_2$(μ-OTf)$_2$] (**1**), [Ag(κ^2-*P*,*P*′-DPEphos)$_2$][OTf] (**2**) and [Ag(κ^2-*P*,*P*′-DPEphos)(2,2′-bpy)][OTf] (**3**) have been synthesized and structurally characterized. In all the complexes, the silver(I) atoms exist in a distorted tetrahedral environment and the DPEphos ligand binds in a chelating fashion. The solid state structure of complex **1** evidences the presence of three adjacent eight-membered spirocyclic rings. The triflate ions in complex **1** act as bridging units between the two silver atoms to form a dinuclear complex.

4. Experimental

4.1. General methods

All manipulations were performed under an atmosphere of dry nitrogen using standard Schlenk techniques. All the solvents were purified by conventional procedures and distilled under nitrogen prior to use [13]. The ligand DPEphos was prepared according to the published procedure [14].

4.2. Instrumentation

The ^{1}H and ^{31}P{^{1}H} NMR (δ in ppm) spectra were recorded using a Varian 400 Mercury Plus spectrometer operating at the appropriate frequencies using TMS and 85% H_3PO_4 as internal and external references, respectively. Microanalyses were performed on a Carlo Erba Model 1112 elemental analyzer. Electro-spray ionization (EI) mass spectrometry experiments were carried out using a Waters Q-Tof micro-YA-105.

4.2.1. Synthesis of [Ag$_2$(κ^2-P,P′-DPEphos)$_2$(μ-OTf)$_2$] (1)

A mixture of DPEphos (0.252 g, 0.467 mmol) and AgOTf (0.120 g, 0.467 mmol) in CH_2Cl_2 (20 mL) was stirred for 4 h at room temperature. All the solvents were dried under vacuum to obtain a white solid, which was recrystallized from CH_2Cl_2/Et_2O as colorless crystals of **1**. Yield: 83% (0.309 g), m.p.: 256–258 °C. ^{1}H NMR (400 MHz, $CDCl_3$): 6.75–7.46 ppm (m, 56H, Ph). ^{31}P NMR (162 MHz, $CDCl_3$): −10.0 ppm (d, $J_{AgP} = 486.8$ Hz). ^{31}P NMR (162 MHz, 223 K, $CDCl_3$): −10.4 ppm (2d, $J^{109}_{AgP} = 526.8$, $J^{107}_{AgP} = 456.7$). *Anal.* Calc. for $C_{74}H_{56}O_8P_4Ag_2F_6S_2$: C, 55.86; H, 3.55; S, 4.03. Found: C, 55.90; H, 3.56; S, 3.92%.

4.2.2. Synthesis of [Ag(κ^2-P,P′-DPEphos)$_2$][OTf] (2)

The procedure was similar to that of **1**, but using AgOTf (0.025 g, 0.097 mmol) and DPEphos (0.105 g, 0.194 mmol). Yield: 98% (0.127 g), m.p.: >260 °C. ^{1}H NMR (400 MHz, $CDCl_3$): 6.68–7.21 (m, 56H, Ph). ^{31}P NMR (162 MHz, $CDCl_3$): −10.5 ppm (2d, $J^{109}_{AgP} = 269.7$, $J^{107}_{AgP} = 233.8$). MS (EI): *m*/*z* 1185.46 [M−OTf]$^+$. *Anal.* Calc. for $C_{73}H_{56}O_5P_4AgF_3S$: C, 65.72; H, 4.23; S, 2.40. Found: C, 65.74; H, 4.20; S, 2.44%.

4.2.3. Synthesis of [Ag(κ^2-P,P′-DPEphos)(2,2′-bpy)][OTf] (3)

A solution of 2,2′-bipyridine (0.010 g, 0.063 mmol) in CH_2Cl_2 (5 mL) was added dropwise to a stirring solution of **1** (0.050 g, 0.031 mmol) also in CH_2Cl_2 (10 mL) at room temperature. After 4 h all the solvents were removed under vacuum and the remaining white solid was recrystallized from a $CHCl_3$/Et_2O mixture as colorless crystals of **3**. Yield: 88% (0.053 g), m.p.: >260 °C. ^{1}H NMR (400 MHz, $CDCl_3$): 6.79–7.38 (m, 28H, Ph), 7.00 (t, 2H, bpy), 8.04 (dt, 2H, bpy), 8.35 ppm (m, 4H, bpy). ^{31}P NMR (162 MHz, $CDCl_3$): −8.8 ppm (2d, $J^{109}_{AgP} = 425.6$, $J^{107}_{AgP} = 374.3$). *Anal.* Calc. for $C_{47}H_{36}O_4P_2AgF_3SN_2$: C, 59.32; H, 3.81; N, 2.94; S, 3.37. Found: C, 59.25; H, 3.85; N, 2.86; S, 3.32%.

4.3. X-ray crystallography

A single crystal of each compound **1**–**3** suitable for X-ray crystal analysis was mounted on a glass fiber with epoxy resin. Unit cell determination and data collection of all the compounds were collected on an Oxford Diffraction XCALIBUR-S CCD system using Mo Kα radiation (λ = 0.71073 Å). The structures were solved and refined by full-matrix least-squares techniques on F^2 using SHELX-97 (SHELXL program package) [15]. The absorption corrections were done by multi-scan and all the data were corrected for Lorentz and polarization effects. The non-hydrogen atoms were refined with anisotropic thermal parameters. All the hydrogen atoms were geometrically fixed and allowed to refine using a riding model.

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Appendix A. Supplementary material

CCDC 643885, 643886 and 643887 contain the supplementary crystallographic data for **1**, **2** and **3**. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK, fax: (+44) 1223-336-033, or e-mail: deposit@ccdc.cam.ac.uk. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.poly.2007.11.019.

References

[1] (a) M. Kranenburg, Y.E.M. van der Burgt, P.C.J. Kamer, P.W.N.M. van Leeuwen, K. Goubitz, J. Fraanje, Organometallics 14 (1995) 3081;
(b) R.J. van Haaren, E. Zuidema, J. Fraanje, K. Goubitz, P.C.J. Kamer, P.W.N.M. van Leeuwen, G.P.F. van Strijdonck, Comptes Rendus Chimie 5 (2002) 431;
(c) M.A. Zuideveld, B.H.G. Swennenhuis, M.D.K. Boele, Y. Guari, G.P.F. van Strijdonck, J.N.H. Reek, P.C.J. Kamer, K. Goubitz, J. Fraanje, M. Lutz, A.L. Spek, P.W.N.M. van Leeuwen, J. Chem. Soc., Dalton Trans. (2002) 2308.

[2] (a) D. Kim, B.-M. Park, J. Yun, Chem. Commun. (2005) 1755;
(b) S.-M. Kuang, D.G. Cuttell, D.R. McMillin, P.E. Fanwick, R.A. Walton, Inorg. Chem. 41 (2002) 3313;
(c) S.-M. Kuang, P.E. Fanwick, R.A. Walton, Inorg. Chem. 41 (2002) 405;
(d) J. Wilting, C. Muller, A.C. Hewat, D.D. Ellis, D.M. Tooke, A.L. Spek, D. Vogt, Organometallics 24 (2005) 13;
(e) A. Pintado-Alba, H. de la Riva, M. Nieuwhuyzen, D. Bautista, P.R. Raithby, H.A. Sparkes, S.J. Teat, J.M. López-de-Luzuriagae, M.C. Lagunas, J. Chem. Soc., Dalton Trans. (2004) 3459.

[3] (a) P.W.N.M. van Leeuwen, P.C.J. Kamer, J.N.H. Reek, P. Dierkes, Chem. Rev. 100 (2000) 2741;
(b) M. Ahmed, A.M. Seayad, R. Jackstell, M. Beller, J. Am. Chem. Soc. 125 (2003) 10311;
(c) P.-E. Broutin, I. Cerna, M. Campaniello, F. Leroux, F. Colobert, Org. Lett. 6 (2004) 4419;
(d) Y. Chen, X.P. Zhang, J. Org. Chem. 68 (2003) 4432;
(e) R. Csuk, A. Barthel, C. Raschke, Tetrahedron 60 (2004) 5737;
(f) G.-Y. Gao, A.J. Colvin, Y. Chen, X.P. Zhang, Org. Lett. 5 (2003) 3261;
(g) Y. Kozawa, M. Mori, J. Org. Chem. 68 (2003) 3064;
(h) R. Kuwano, Y. Kondo, Y. Matsuyama, J. Am. Chem. Soc. 125 (2003) 12104;
(i) M. Ogasawara, H. Ikeda, T. Nagano, T. Hayashi, Org. Lett. 3 (2001) 2615;
(j) C.H. Oh, V.R. Reddy, Tetrahedron Lett. 45 (2004) 5221;
(k) M.C. Willis, D. Taylor, A.T. Gillmore, Org. Lett. 6 (2004) 4755;
(l) J.P. Wolfe, M.A. Rossi, J. Am. Chem. Soc. 126 (2004) 1620.

[4] R. Venkateswaran, J.T. Mague, M.S. Balakrishna, Inorg. Chem. 46 (2007) 809.

[5] R. Venkateswaran, M.S. Balakrishna, S.M. Mobin, H.M. Tuononen, Inorg. Chem. 46 (2007) 6335.

[6] (a) M.S. Balakrishna, R. Panda, J.T. Mague, J. Chem. Soc., Dalton Trans. (2002) 4617;
(b) M.S. Balakrishna, R. Panda, J.T. Mague, Polyhedron 22 (2003) 587;
(c) S. Priya, M.S. Balakrishna, S.M. Mobin, R. McDonald, J. Organomet. Chem. 688 (2003) 227;
(d) P. Chandrasekaran, J.T. Mague, M.S. Balakrishna, Inorg. Chem. 44 (2005) 7925;
(e) P. Chandrasekaran, J.T. Mague, M.S. Balakrishna, Organometallics 24 (2005) 3780;
(f) P. Chandrasekaran, J.T. Mague, M.S. Balakrishna, Inorg. Chem. 45 (2006) 5893;
(g) P. Chandrasekaran, J.T. Mague, M.S. Balakrishna, Inorg. Chem. 45 (2006) 6678;
(h) B. Punji, J.T. Mague, M.S. Balakrishna, J. Chem. Soc., Dalton Trans. (2006) 1322;
(i) B. Punji, C. Ganesamoorthy, M.S. Balakrishna, J. Mol. Catal. A: Chem. 259 (2006) 78;
(j) B. Punji, J.T. Mague, M.S. Balakrishna, J. Organomet. Chem. 691 (2006) 4265;
(k) R. Venkateswaran, M.S. Balakrishna, S.M. Mobin, Eur. J. Inorg. Chem. (2007) 1930.

[7] M. Bardaji, O. Crespo, A. Laguna, A.K. Fischer, Inorg. Chim. Acta 304 (2000) 7.

[8] E.J. Sekabunga, M.L. Smith, T.R. Webb, W.E. Hill, Inorg. Chem. 41 (2002) 1205.

[9] P.G. Jones, Acta Crystallogr., Sect. C 49 (1993) 1148.

[10] M.C. Gimeno, P.G. Jones, A. Laguna, C. Sarroca, J. Chem. Soc., Dalton Trans. (1995) 1473.

[11] Effendy, C.D. Nicola, M. Fianchini, C. Pettinari, B.W. Skelton, N. Somers, A.H. White, Inorg. Chim. Acta 358 (2005) 763.

[12] A. Cingolani, Effendy, F. Marchetti, C. Pettinari, R. Pettinari, B.W. Skelton, A.H. White, Inorg. Chim. Acta 329 (2002) 100.

[13] W.L.F. Armarego, D.D. Perrin, Purification of Laboratory Chemicals, IVth ed., Butterworth-Heinemann Linacre House, Jordan Hill, Oxford, UK, 1996.

[14] M. Kranenburg, Y.E.M. van der Burgt, P.C.J. Kamer, P.W.N.M. van Leeuwen, K. Goubitz, J. Fraanje, Organometallics 14 (1995) 3081.

[15] G.M. Sheldrick, SHELXS97 and SHELXL97, University of Gottingen, Gottingen, Germany, 1997.