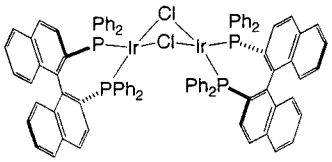
ture was solved by direct methods (maXus) and refined with all data by full-matrix least squares on *F*. All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were added in idealized positions and included in the refinement. Crystallographic data (excluding structure factors) for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Center as supplementary publication no. CCDC-102228. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ, UK (fax: (+44) 1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

[11] It was proposed that the boron enolates of α-unsubstituted ketones favor the more stable U form over the W form by 1–2 kcal mol^{-1}. Similarly, it is conceivable that the bulky phenoxy group of the enolate of **4a** renders the U form most likely, and gives a twist-boat transition structure: a) C. Gennari, R. Todeschini, M. G. Beretta, G. Favini, C. Scolastico, *J. Org. Chem.* **1986**, *51*, 612; b) R. W. Hoffmann, K. Ditrich, S. Froech, *Tetrahedron* **1985**, *41*, 5517; see also reference [2b].

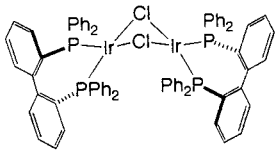
Facile Oxidative Addition of O–H Bonds of Methanol and Water to Ir^{I} Complexes Having Peraryldiphosphane Ligands**

Kazuhide Tani,* Aika Iseki, and Tsuneaki Yamagata

Studies on the oxidative addition of alcohol and water to transition metal complexes leading to the formation of hydrido(hydroxo) and hydrido(alkoxo) complexes, respectively, have received much attention for the past decade due to their potential relevance to catalysis.[1] In particular hydrido(alkoxo) complexes of late transition metals have often been postulated as intermediates or transition states in the catalytic hydrogenation of carbonyl compounds[1a] and catalytic hydrogen transfer reactions.[2] Isolation or detection of such complexes, however, is rare, and their chemistry remains relatively unexplored. About a decade ago we isolated and characterized a hydrido(alkoxo) complex of iridium as a model complex of the reaction intermediates for the rhodium-catalyzed hydrogenation of carbonyl compounds.[3] Milstein and his co-workers reported their efforts on the oxidative addition of hydrogen–heteroatom bonds to rhodium and iridium leading to hydrido(alkoxo) and hydrido(hydroxo) complexes, among others.[4] In addition the isolation of a few hydrido(alkoxo) complexes[5] and hydrido(hydroxo) complexes[1d, 5d] of late transition metals has also been reported. Almost all of these complexes, however, bear basic peralkylphosphane ligands or an electron-donating Cp* ligand (Cp* = C_5Me_5). We have found that dinuclear Ir^{I} complexes [IrCl(diphosphane)]$_2$ carrying even peraryldiphosphanes that are not so strongly electron donating (diphosphane = 2,2′-bis(diphenylphosphanyl)-1,1′-binaphthyl (binap), see **1a**, or 2,2′-bis(diphenylphosphanyl)-1,1′-biphenyl (bpbp), see **1b**)[6, 7] can easily activate protic molecules (MeOH or H_2O) at ambient temperature.

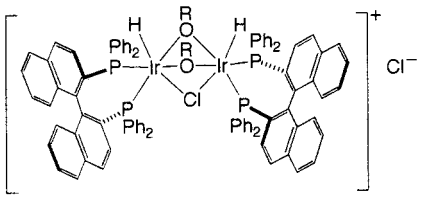


1a



1b

Upon treatment of a solution of the extremely air-sensitive complex **1a** in toluene with a large excess of methanol, the color changed immediately from deep red to pale yellow. After the reaction mixture had been stirred at room temperature for several hours, the solvents were removed in vacuo to give quantitatively the almost pure hydrido(methoxo) complex **2a** as air-stable and thermally stable pale yellow microcrystals. The formulation was supported by the elemental analyses (see Table 1 and the supporting information).



2a: R = Me
3a: R = H

The ^{31}P NMR spectrum of **2a** exhibits two doublets at $\delta = -15.2$ and -1.8 (^2J(P,P) = 19 Hz) in a 1:1 ratio, indicating the presence of two nonequivalent phosphorus atoms. The presence of terminal hydrido ligands was suggested by the IR ($\tilde{\nu}$(Ir–H) = 2270 cm^{-1}) and ^{1}H NMR spectra ($\delta = -23.10$ (dd, ^2J(H,P) = 18, 23 Hz)). The methoxy methyl protons appear at $\delta = 2.63$ as a triplet (^4J(H,P) = 3.4 Hz). The signal is more than 1 ppm upfield of the analogous signals of the mononuclear iridium complexes *mer-cis*-[Ir(Cl)(H)(OMe)(PEt$_3$)$_3$] ($\delta = 4.01$)[4d] and *cis*-[Ir(H)(OMe)(PMe$_3$)$_4$]$^+$PF$_6^-$ ($\delta = 3.637$)[4a] and appears at 0.55 ppm higher field than even that of the Ir^{I}-methoxo complex [Ir$_2$(μ-OMe)$_2$(cod)$_2$] ($\delta = 3.28$; cod = 1,5-cyclooctadiene).[8] This may be due to the diamagnetic shielding by a benzene ring of the diphenylphosphanyl moiety of the binap ligands (vide infra). Complex **2a** is much more thermally stable than these monomeric complexes. The ^{1}H NMR spectrum indicates the presence of a molecule of solvated methanol: a doublet at $\delta = 3.38$ ($J = 5.6$ Hz) for the methyl protons and a quartet at $\delta = 1.62$ for the OH proton. This may indicate strong hydrogen bonding between the chloro bridge and the solvated methanol even in solution (vide infra).[9] Exchange between the methoxo and the

[*] Prof. Dr. K. Tani, A. Iseki, Dr. T. Yamagata
Department of Chemistry, Graduate School of Engineering Science
Osaka University
Toyonaka, Osaka, 560–8531 (Japan)
Fax: (+81) 6-6850-6245
E-mail: tani@chem.es.osaka-u.ac.jp

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Supporting information for this article is available on the WWW under http://www.wiley-vch.de/home/angewandte/ or from the author.

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Table 1. Selected data for complexes **2a**, **b** and **3a**, **b**.

2a: M.p. > 108 °C (decomp in a capillary under vacuum); ^{1}H NMR (270 MHz, 35 °C, CD_2Cl_2): $\delta = -23.10$ (dd, ^2J(H,P) = 18, 23 Hz, 2H; Ir–H), 1.62 (q, J = 5.6 Hz, 1H; CH_3OH), 2.63 (t, ^4J(H,P) = 3.4 Hz, 6H; Ir–OCH_3), 3.38 (d, J = 5.6 Hz, 3H; CH_3OH), 6.14 – 8.02 (m, 64H; Ph + Naph); ^{31}P{^{1}H} NMR (109.25 MHz, 35 °C, CD_2Cl_2): $\delta = -1.8$ (d, ^2J(P,P) = 19 Hz), −15.2 (d, ^2J(P,P) = 19 Hz); IR (film): $\tilde{\nu} = 2270\ cm^{-1}$ (br m; Ir–H); $\Lambda_0 = 80.4\ S\,cm^2\,mol^{-1}$ (in dry MeOH, at 25 °C); the elemental analysis of **2a** obtained after the sample had been thoroughly dried in vacuo agreed well with the chemical formula without the solvate methanol; elemental analysis calcd for $C_{90}H_{72}Cl_2Ir_2O_2P_4$: C 61.25, H 4.11; found: C 61.05, H 4.27

2b: M.p. > 130 °C (decomp in capillary under vacuum); ^{1}H NMR (270 MHz, 35 °C, $CDCl_3$): $\delta = -23.25$ (dd, ^2J(H,P) = 18, 23 Hz, 2H; Ir–H), 2.77 (t, ^3J(H,P) = 3 Hz, 6H; Ir–OCH_3), 6.22 – 7.77 (m, 56H; ArH); ^{31}P{^{1}H} NMR (109.25 MHz, 35 °C, $CDCl_3$): $\delta = -3.3$ (d, ^2J(P,P) = 20 Hz), −13.3 (d, ^2J(P,P) = 20 Hz); IR (film): $\tilde{\nu} = 2261\ cm^{-1}$ (br m; Ir–H); elemental analysis calcd for $C_{74}H_{64}Cl_2Ir_2O_2P_4$: C 56.81, H 4.12; found: C 56.56, H 4.43

3a: M.p. > 110 °C (decomp in a capillary under vacuum); ^{1}H NMR (270 MHz, 35 °C, $CDCl_3$) $\delta = -22.14$ (dd, ^2J(H,P) = 16, 22 Hz, 2H; Ir–H), 1.49 (t, ^3J(H,P) = 3 Hz, 2H; Ir–OH), 6.43 – 7.86 (m, 64H; Ph + Naph); ^{31}P{^{1}H} NMR (109.25 MHz, 35 °C, $CDCl_3$): $\delta = -0.9$ (d, ^2J(P,P) = 20 Hz), −2.1 (d, ^2J(P,P) = 20 Hz); IR (film): $\tilde{\nu} = 3525$ (m, (O–H)), 2267 cm^{-1} (br m; Ir–H); $\Lambda_0 = 69.3\ S\,cm^2\,mol^{-1}$ (in THF/H_2O (1/1 v/v), at 25 °C); elemental analysis calcd for $C_{88}H_{68}Cl_2Ir_2O_2P_4$: C 60.86, H 3.95; found: C 60.96, H 4.35

3b: M.p. > 120 °C (decomp in a capillary under vacuum); ^{1}H NMR (270 MHz, 35 °C, $CDCl_3$): $\delta = -23.26$ (dd, ^2J(P,P) = 18, 22 Hz, 2H; Ir–H), 1.80 (br, 2H; Ir–OH), 6.10 – 7.82 (m, 56H; ArH); ^{31}P{^{1}H} NMR (109.25 MHz, 35 °C, $CDCl_3$): $\delta = -2.5$ (d, ^2J(P,P) = 20 Hz), −6.6 (d, ^2J(P,P) = 20 Hz); IR (film): $\tilde{\nu} = 3560$ (m; O–H), 2258 cm^{-1} (br m; Ir–H); elemental analysis calcd for $C_{72}H_{60}Cl_2Ir_2O_2P_4$: C 56.28, H 3.94; found: C 55.71, H 4.17

hydrido ligands with the solvated methanol was not detected below 40 °C in CD_2Cl_2 in ^{1}H NMR experiments.

The structure was confirmed by X-ray analysis. Single crystals of **2a** containing one molecule of solvated methanol suitable for X-ray analysis were grown from absolute methanol.[10] The molecular view in Figure 1 reveals the presence of a C_2 axis. The cationic part of **2a** has a dinuclear iridium structure. The coordination environment of each iridium atom can be described as a highly distorted octahedron with two phosphorus atoms, two bridging methoxo ligands, a chloro ligand, and a terminal hydrido ligand. The chloride counteranion is located near the solvate methanol, which is situated on a C_2 axis, as two disordered peaks and the hydrogen bond between the chloride and the methanol should be present (O2 ··· Cl2 2.62(2) Å). A terminal hydrido ligand should be present at the position *trans* to the μ-chloro ligand and *cis* to the methoxy groups. The methoxy methyl groups are situated just above the benzene rings of the diphenylphosphanyl moieties of the binap ligands. The complex has a bent $Ir_2(\mu\text{-}OMe)_2$ core with a hinge angle of 141.4(1)°, which deviates seriously from the expected value (109.5°) made by two fused face-sharing octahedrons. The dihedral angle between the pair of naphthyl planes is 81.4(1)°, the largest angle among binap complexes characterized to date.[6, 14] The Ir–Cl bond length (2.534(2) Å) is the longest of the Ir^{III}–μ-Cl distances reported so far.[15] These structural features agree well with the spectroscopic characteristics.

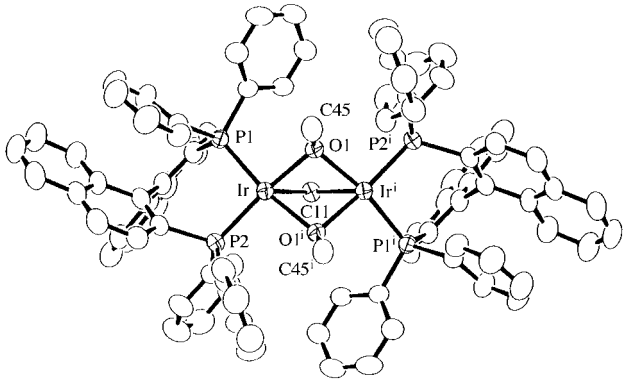
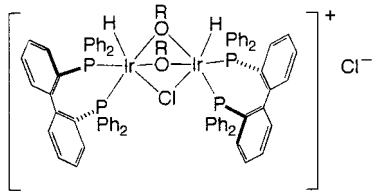


Figure 1. X-ray crystral structure showing the cationic part of **2a** (ORTEP view, ellipsoids at 50% probability). Selected interatomic distances [Å] and angles [°]: Ir–O1 2.106(4), Ir–O1^i 2.138(4), Ir–P1 2.2477(13), Ir–P2 2.2624(13), Ir–Cl1 2.534(2), Ir ··· Ir^i 3.1461(7); O1-Ir-O1^i 76.5(2), O1-Ir-P1 93.57(10), O1^i-Ir-P1 169.69(10), O1-Ir-P2 172.09(11), O1^i-Ir-P2 96.96(10), P1-Ir-P2 93.15(5), O1-Ir-Cl1 75.38(10), O1^i-Ir-Cl1 74.87(10), P1-Ir-Cl1 100.18(4), P2-Ir-Cl1 107.50(4), Ir-Cl1-Ir^i 76.75(5), Ir-O1-Ir^i 95.67(14), C45-O1-Ir 115.5(4), C45-O1-Ir^i 125.2(4). i: ($-x$, y, $-z$).

The analogous reaction of **1b** with methanol gave the corresponding hydrido(methoxo) complex **2b** as pale yellow crystals (see Table 1 and the supporting information). Complexes **1a** and **1b** can also activate water. Thus, the reaction of



2b: R = Me
3b: R = H

1a and **1b** with excess water in THF at ambient temperature gave hydrido(hydroxo) complexes **3a** and **3b**, respectively, as air-stable, pale yellow powders in almost quantitative yields (see Table 1). Based on the analogies in the ^{1}H NMR spectra of **2a** and **3a** [for the latter, Ir–H signal at $\delta = -22.14$ (dd, ^2J(H,P) = 16, 22 Hz), Ir–OH signal at $\delta = 1.49$ (t, ^3J(H,P) = 3 Hz)] and in the ^{31}P{^{1}H} NMR spectra [$\delta = -0.9$ (d, ^2J(P,P) = 20 Hz), −2.1 (d, ^2J(P,P) = 20 Hz)], complexes **3** are also considered to have similar triply bridged dinuclear structures with two μ_2-OH groups and a μ_2-Cl atom (vide supra). The ionic structure of **3a** has been confirmed by its limiting equivalent conductance, $\Lambda_0 = 69.3\ S\,cm^2\,mol^{-1}$ measured in THF/H_2O (1/1 v/v) at 25 °C.[16]

We have demonstrated that iridium – diphosphane complexes **1a** and **1b**, which have peraryldiphosphane ligands, easily activate methanol and water to give hydrido(methoxo) complexes **2** as well as hydrido(hydroxo) complexes **3** in good yield. The structures of these new complexes were characterized spectroscopically and by X-ray crystallographic analysis of **2a**. We are continuing to explore the catalytic application of these complexes.

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[10] X-ray crystal structure determination of **2a**: $C_{90}H_{72}Cl_2Ir_2O_2P_4 \cdot CH_3OH$, $M_r = 1796.70$; crystal dimensions $0.43 \times 0.23 \times 0.13$ mm^3; monoclinic, space group $C2$, $a = 17.722(3)$, $b = 16.201(4)$, $c = 16.424(3)$ Å; $\beta = 113.604(12)^\circ$, $V = 4321.0(16)$ Å^3, $Z = 2$, $\rho_{calcd} = 1.381$ g cm^{-3}, $\mu = 3.258$ mm^{-1}. Single crystals suitable for X-ray measurements were obtained by repeated recrystallization of **2a** from anhydrous methanol under argon. Because the surface of the pale yellow crystals became rough immediately when isolated from the mother liquor, presumably as a consequence of loss of the solvate methanol, the crystals were coated with silicon grease and sealed in glass capillaries containing methanol. Cell constants and an orientation matrix for data collection were determined by the least-squares refinement, using the setting angles of 25 reflections in the range $23^\circ < 2\theta < 25^\circ$ on a Rigaku diffractometer (AFC-7R) equipped with a Rotaflex rotating anode X-ray generator (50 kV, 200 mA) with monochromated $Mo_{K\alpha}$ radiation. For the data collection, reflections in the range 0 to $+h$, 0 to $+k$, $-l$ to $+l$, and the corresponding Friedel pairs (matrix: $(-1\ 0\ 0)$, $(0\ -1\ 0)$, $(0\ 0\ -1)$) were measured by the $\omega - 2\theta$ scan technique (scan speed 16.0° min^{-1}, scan width $(1.57 + 0.30\tan\theta)^\circ$). Three standard reflections were monitored every 200 reflections and showed a 7.19% intensity decrease over the collection period. Intensity data were corrected for Lorentz and polarization effects. The absorption corrections ($T_{min} = 0.4874$, $T_{max} = 0.6545$) based on psi-scan data and a linear correction factor were applied for the raw data. A total of 10642 reflections ($3.0^\circ < 2\theta < 55.12^\circ$) were collected at 293(2) K, of which 9955 unique reflections ($R_{int} = 0.0171$) were used for the structure solution and refinement. The structure was solved by direct methods (SHELXS-86)[11] and weighted Fourier techniques[12] and refined on F^2 by full-matrix least-squares methods using SHELXL-93.[13] Non-hydrogen atoms of the cationic part were refined anisotropically. The chlorine atom of the anionic part and non-hydrogen atoms of methanol were refined isotropically. Hydrogen atoms of aromatic and methoxy groups were included in the refinement on calculated positions riding on their carrier atoms ($C{-}H_{sp^2} = 0.93$ Å, $C{-}H_{sp^3} = 0.96$ Å, $U_{iso}(H) = 1.2\,U_{eq}(C)$). The hydrogen atoms of the methyl group of the solvate methanol were located on a difference-Fourier map and fixed in the refinements. The OH hydrogen atoms of the methanol molecule were not included in the calculations. The function minimized was $[\Sigma w(F_o^2 - F_c^2)^2]$ ($w = 1/[\sigma^2(F_o^2) + (0.0534P)^2 + 1.6777P]$), where $P = (\max(F_o^2,0) + 2F_c^2)/3$ with $\sigma^2(F_o^2)$ from counting statistics. The functions $R1$ and $wR2$ were $(\Sigma\,||F_o| - |F_c||)/\Sigma\,|F_o|$ and $[\Sigma w(F_o^2 - F_c^2)^2/\Sigma(wF_o^4)]^{1/2}$, respectively; $R1 = 0.0290$ and $wR2 = 0.0838$ for 9226 reflections with $I > 2.0\sigma(I)$. Final R factors: $R1 = 0.0351$ and $wR2 = 0.0866$ for 9955 reflections (all data). The number of parameters is 455. Flack χ parameter shows $-0.018(7)$, and the absolute configuration of the binap ligand is R. GOF values are 1.153 for $I > 2.0\sigma(I)$ and 1.146 for all data. The maximum shift to esd ratio in the last full-matrix least-squares cycle was 0.000. The final difference Fourier map revealed a number of areas of residual electron density, the greatest of which corresponds to 1.109 e Å^{-3}. The minimum hole of the final difference Fourier map is -1.270 e Å^{-3}. For more details refer to the supporting information. Crystallographic data (excluding structure factors) for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Center as supplementary publication no. CCDC-101027. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: (+44) 1223-336-033; e-mail: deposit@ccdc.cam.ac.uk). All calculations were carried out on Silicon Graphics SGI Power Challenge workstation at the Research Center for Protein Engineering, Institute for Protein Research, Osaka University.

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Micro-Scale Frontal Affinity Chromatography with Mass Spectrometric Detection: A New Method for the Screening of Compound Libraries

David C. Schriemer, David R. Bundle, Liang Li, and Ole Hindsgaul*

The application of combinatorial chemistry to accelerated drug discovery has generated new problems in screening for potential therapeutics. Classical approaches of a one-compound, one-assay variety are often untenable because of time and resource constraints since the goal of combinatorial chemistry is to produce large libraries of compounds. These libraries are either generated on a solid phase (beads) or in solution.[1] A binding assay must be performed against a target at some point, and this usually means assaying either a large

[*] Prof. O. Hindsgaul, Dr. D. Schriemer, Prof. D. Bundle, Prof. L. Li
Department of Chemistry, University of Alberta
Edmonton, T6G 2G2 (Canada)
Fax: (+1) 403-492-7705
E-mail: ole.hindsgaul@ualberta.ca

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