with $C_2H_5^{\bullet}$. The reactivity ratio is probably even higher than the upper limit suggests. For example, Fe^{2+}, V^{2+}, Cr^{2+}, and $Co(CN)_5^{3-}$ have k_{SCN}/k_{NCS} ratios of $>4 \times 10^4$, 10^2, 10^4, and 10^{2-3}, respectively.[34,35]

The reactivity of $Co(NH_3)_5N_3^{2+}$ ($k = 3.7 \times 10^7$ L mol^{-1} s^{-1}) is somewhat higher than that of $Co(NH_3)_5SCN^{2+}$, whereas the opposite is true for the metal donors.[34,35] The product from the azido reaction was not established, and the higher reactivity toward ethyl radicals might possibly result from attack of $^{\bullet}C_2H_5$ on the N=N bonds of coordinated azide ions.

The complexes $Ru(NH_3)_5X^{2+}$ (X = Cl, Br) are more reactive than their cobalt counterparts by factors of 300 (Cl) and 10 (Br). The greater reactivity of ruthenium was also observed in their reductions by α-hydroxyalkyl radicals.[36]

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Contribution from the Laboratoire de Chimie de Coordination CNRS, UP No. 8241 Liée par Conventions
à l'Université Paul Sabatier et à l'Institut National Polytechnique, 205 Route de Narbonne, Toulouse Cedex, France,
and the Department of Chemistry and Molecular Structure Center, Indiana University, Bloomington, Indiana 47405

Modification of the Reactivity of Polyhydrides with Lewis Acids. Synthesis and Reactivity of $\{IrH_5[P(^iC_3H_7)_3]_2\}_2Cu(CF_3SO_3)$. X-ray Crystal Structure of $\{IrH_2(MeCN)_2[P(^iC_3H_7)_3]_2\}(BF_4)$

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The reaction of $IrH_5(P^iPr_3)_2$ (**1**) with $[Cu(MeCN)_4]BF_4$ (2:1) in acetone yields $[IrH_2(MeCN)_2(P^iPr_3)_2]BF_4$ (**2**), characterized by spectroscopic methods and X-ray diffraction, in 46% yield. A similar reaction with $CuCF_3SO_3$ in CH_2Cl_2 yields $[IrH_5(P^iPr_3)_2]_2CuCF_3SO_3$ (**3**) in 91% yield. Compound **3** reacts rapidly with MeCN, D_2, and cyclopentadiene at 25 °C in CH_2Cl_2 to give respectively a 1:1 mixture of **1** and **2**, deuterated **3**, and $[CpIrH(P^iPr_3)_2]SO_3CF_3$ and shows a reduced relaxation time T_1 for the hydrides, compared to **1**.

Introduction

Since the first demonstration by Kubas et al. of the coordination of dihydrogen to a transition metal,[1] the chemistry of polyhydride derivatives has been dominated by the dilemma "classical or nonclassical"; i.e., are the so-called "polyhydrides" real hydrido derivatives or do they contain coordinated H_2?[2] The situation is probably not so clear, since in simple cases like $W(CO)_3(PR_3)_2H_2$[1] and $CpRuL_2H_2^{+}$[3] the coexistence of dihydride and dihydrogen isomers has been demonstrated. We have reported recently a dynamic equilibrium between a dihydride and a dihydrogen complex.[4] Furthermore it is anticipated that slight modifications in the electron density on the metal can change the mode of bonding of the hydrogen ligands. The modification can be made in the phosphine substituents.[5] This behavior is also revealed by the protonation reaction[6]

$$IrH_5(PCy_3)_2 + H^+ \rightarrow [IrH_2(H_2)_2(PCy_3)_2]^+ \qquad (1)$$

In this case, not only one hydrogen molecule is formed by protonation of a hydride ligand but a second one is formed by reductive coupling of two hydride ligands.

After the demonstration by one of us[7] and by Venanzi[8] that Lewis acid fragments can give adducts with polyhydrides, we have attempted to manipulate the free energy difference between hydrides and coordinated H_2 in complexes containing both type of ligands.[9] Although this goal has not yet been achieved, we succeeded there in observing exchange coupling between terminal and bridged hydride ligands. Nevertheless, coordination of a Lewis acid to a classical polyhydride is expected to reduce the electron density on the metal. A change in the structure and/or an increase in the reactivity of the polyhydride should normally result from this reaction. As a comparison to eq 1, we report in this paper the reaction of $IrH_5(PR_3)_2$ with copper salts and the chemical reactivity of one such adduct.

Experimental Section

All experiments were conducted under dry argon by using conventional Schlenk tube techniques. Infrared spectra were recorded as KBr disks or Nujol mulls with a Perkin-Elmer 983 grating spectrometer. ^1H and ^{31}P NMR spectra were recorded by using Bruker WH90, AC200, or WM250 NMR spectrometers. NMR integrations were measured either on one scan in concentrated solution or with a repetition delay of 1 min, to avoid incomplete and differential relaxation. Both methods gave results that agreed to within 10%. Microanalyses were performed by the "Centre de Microanalyse du CNRS" or in-house.

$[IrH_2(CH_3CN)_2(P^iPr_3)_2]BF_4$ (2). To 500 mg, 0.97 mmol, of $IrH_5(P^iPr_3)_2$[10] was added an acetone solution (20 mL) containing 161 mg, 0.48 mmol, of $[Cu(CH_3CN)_4]BF_4$. With stirring, $IrH_5(P^iPr_3)_2$ dissolved and a colorless solution was obtained in 5 min. After being stirred for 1 h, the resulting solution was concentrated to a small volume under reduced pressure and treated with 15 mL of hexane to precipitate white microcrystals, which were washed with hexane (3 × 5 mL). The product $[IrH_2(CH_3CN)_2(P^iPr_3)_2]BF_4$ was obtained in 46.5% yield (255 mg) based on Ir. Anal. Calcd for $IrP_2N_2BF_4C_{22}H_{50}$: C, 38.65; H, 7.37; N, 4.10. Found: C, 38.26; H, 7.77; N, 4.33. $IrH_5(P^iPr_3)_2$ (50% of initial quantity) may be recovered from the resulting filtrate.

Crystals suitable for X-ray diffraction studies may be obtained by cooling a saturated acetone solution to −20 °C.

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Table I. Crystallographic Data for $[Ir(H)_2(NCMe)_2(P^iPr_3)_2]BF_4$

chem formula	$C_{22}H_{50}IrN_2P_2^+BF_4^-$	space group	$P2_1/a$
a, Å	15.870 (3)	T, °C	−155
b, Å	16.002 (3)	λ, Å	0.71069
c, Å	11.496 (2)	ρ_{calcd}, g cm^{-3}	1.555
β, deg	90.03 (1)	μ(Mo Kα), cm^{-1}	47.0
V, Å^3	2919.63	R	0.0396
Z	4	R_w	0.0385
fw	683.62		

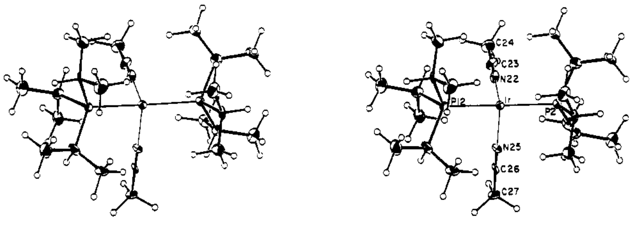


Figure 1. Stereo ORTEP view of $[Ir(H)_2(NCMe)_2(P^iPr_3)_2]^+$, showing selected atom labeling. Hydride hydrogens were not located.

$[IrH_5(P^iPr_3)_2]_2CuCF_3SO_3 \cdot 2CH_2Cl_2$ (3). To 56 mg, 0.22 mmol, of $CuCF_3SO_3 \cdot 0.5C_6H_6$ (purchased from Aldrich) was added a dichloromethane solution of 230 mg, 0.44 mmol, of $IrH_5(P^iPr_3)_2$. $CuCF_3SO_3 \cdot 0.5C_6H_6$ dissolved, and a pale yellow solution was obtained after stirring this mixture for a 0.5 h. After the solution was stirred for another 1 h, the solvent was removed under reduced pressure. The resulting residue was washed with ether (2 × 5 mL). A pale yellow powder $[IrH_5(P^iPr_3)_2]_2CuCF_3SO_3 \cdot 2CH_2Cl_2$ was obtained in 91% yield (249 mg). Anal. Calcd for $Ir_2CuP_4SO_3F_3Cl_4C_{39}H_{98}$: C, 33.04; H, 6.97. Found: C, 33.19; H, 6.97. The CH_2Cl_2 in the crystal lattice was independently quantitated by ^{1}H NMR spectroscopy.

Recrystallization from CH_2Cl_2/Et_2O at −20 °C yielded $[IrH_5(P^iPr_3)_2]_2CuCF_3SO_3 \cdot xCH_2Cl_2$ ($x > 2$) in the form of white monocrystals, which lost solvent rapidly and transformed into a yellow powder.

Crystal Structure Determination of $[Ir(H)_2(NCMe)_2(P^iPr_3)_2]BF_4$. A very small crystal, grown from acetone, was selected from the bulk sample by using inert-atmosphere handling techniques. The crystal was transferred to the goniostat where it was cooled to −155 °C for characterization and data collection.[11] A systematic manual search of a limited hemisphere of reciprocal space yielded a set of reflections that exhibited at least monoclinic ($2/m$) symmetry. The reflections were indexed by using a monoclinic lattice; however, the angles were all equal to 90° within experimental error. Closer inspection of the reflections showed that the lattice was indeed monoclinic but pseudoorthorhombic. The initial indexing was as a monoclinic lattice with c unique; the data were then reindexed to the normal setting of b unique. The systematic extinctions of $h0l$ for $h = 2n + 1$ and of $0k0$ for $k = 2n + 1$ identified the space group as $P2_1/a$. It should be noted that in general all reflections having $h + k = 2n$ were much stronger than those having $h + k = 2n + 1$; for the $0kl$ zone there was a pseudoextinction for $k + l = 2n + 1$. The choice of the space group $P2_1/a$ was confirmed by the successful solution and refinement of the structure.

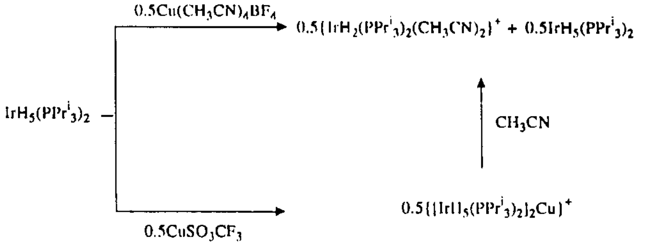
Data collection (−155 °C; 6° < 2θ < 45°) yielded a total of 8396 reflections (including standards and space group extinctions). Parameters of the crystal and data set appear in Table I. Following the usual data reduction and averaging of redundant data, a unique set of 3837 reflections were obtained. The R for the averaging was 0.047 for 3703 reflections observed more than once. It is interesting to note that when an incorrect b axis was chosen, the data set averaged to 0.15, showing how dominant the pseudosymmetry is. A plot of the standard reflections showed no systematic trends; no absorption correction was performed.

The structure was determined with the usual combination of direct methods and Fourier techniques. All non-hydrogen atoms were located without difficulty, and almost all of the carbon-bound hydrogen atoms were located in a later difference Fourier following initial refinement. The full-matrix least-squares refinement was completed by using anisotropic thermal parameters on all non-hydrogen atoms and using fixed idealized hydrogen atoms. The two hydride hydrogen atoms were not detectable. The final difference map contained three peaks of about 1 e/Å^3 in the immediate vicinity of the Ir atom; the next higher peaks were located near BF_4^-. The results of the structural study are shown in Table II and Figure 1. Further details are available as supplementary material. The bond angles about phosphorus are unexceptional: ∠Ir–P–C varies from 112.5 (3) to 116.1 (3)° and ∠C–P–C varies from 103.4 (5) to 106.2 (5)°.

Table II. Selected Bond Distances (Å) and Angles (deg) for $[Ir(H)_2(NCMe)_2(P^iPr_3)_2]BF_4$

Ir(1)–P(2)	2.3263 (27)	F(30)–B(32)	1.393 (15)
Ir(1)–P(12)	2.3251 (26)	F(31)–B(32)	1.378 (15)
Ir(1)–N(22)	2.147 (9)	N(22)–C(23)	1.122 (13)
Ir(1)–N(25)	2.128 (9)	N(25)–C(26)	1.126 (12)
F(28)–B(32)	1.390 (14)	C(23)–C(24)	1.447 (15)
F(29)–B(32)	1.365 (16)	C(26)–C(27)	1.469 (15)
P(2)–Ir(1)–P(12)	167.39 (9)	N(22)–C(23)–C(24)	179.3 (11)
P(2)–Ir(1)–N(22)	94.75 (22)	N(25)–C(26)–C(27)	179.5 (10)
P(2)–Ir(1)–N(25)	94.78 (22)	F(28)–B(32)–F(29)	110.4 (11)
P(12)–Ie(1)–N(22)	95.46 (22)	F(28)–B(32)–F(30)	108.1 (10)
P(12)–Ir(1)–N(25)	93.17 (22)	F(28)–B(32)–F(31)	108.3 (10)
N(22)–Ir(1)–N(25)	86.7 (3)	F(29)–B(32)–F(30)	110.5 (10)
Ir(1)–N(22)–C(23)	176.1 (9)	F(29)–B(32)–F(31)	110.6 (11)
Ir(1)–N(25)–C(26)	170.8 (8)	F(30)–B(32)–F(31)	108.9 (11)

Scheme I



Exchange of 3 with D_2. Deuterium was slowly bubbled into a dichloromethane solution (10 mL) of 100 mg (0.08 mmol) of **3** for 15 min at 25 °C. The resulting solution was evaporated to dryness and analyzed by ^{1}H NMR spectroscopy.

$[(\eta^5\text{-}C_5H_5)IrH(P^iPr_3)_2]SO_3CF_3$ (4). (a) To a solution of $IrH_5(P^iPr_3)_2$ (126 mg, 0.24 mmol) in 20 mL of CH_2Cl_2 was added 20 μL (0.24 mmol) of C_5H_6 and 61 mg (0.24 mmol) of $CuSO_3CF_3$. After the solution was stirred for 15 h at 25 °C, the solvent was removed in vacuum and the residue extracted with CH_2Cl_2. The resulting solution was concentrated in vacuo and treated with 20 mL of Et_2O to precipitate the product. Yield: 110 mg (63%).

(b) To a solution of $IrH_5(P^iPr_3)_2$ (130 mg, 0.25 mmol) in 25 mL of CH_2Cl_2 was added $HBF_4 \cdot OEt_2$ (34 μL, 0.25 mmol) and C_5H_6 (21 μL, 0.25 mmol). The solution was refluxed for 4 h. The resulting solution was concentrated to 5 mL in vacuo, and Et_2O was slowly added until the colorless solid $[CpIrH(P^iPr_3)_2]BF_4$ had precipitated. Yield: 135 mg (80%). Anal. Calcd for $C_{23}H_{48}BF_4IrP_2$: C, 41.50; H, 7.27. Found: C, 41.21; H, 7.20.

Results

$IrH_5(P^iPr_3)_2$[10] (**1**) reacts rapidly with 0.5 equiv of $CuOSO_2CF_3$ in dichloromethane without detectable gas evoluation to give (Scheme I) an off-white solution from which off-white crystals analyzing for $[IrH_5(P^iPr_3)_2]_2Cu(OSO_2CF_3)$ (**3**) are obtained upon recrystallization in CH_2Cl_2/Et_2O. Compound **3** shows a high-field triplet at δ −10.0 (J_{PH} = 9.8 Hz) in the ^{1}H NMR spectrum, which is close to the position of the hydride ligands of **1** (δ −11, J_{PH} = 12 Hz). Compound **3** is insoluble in nonpolar solvents, and a variable-temperature NMR study in CD_2Cl_2 shows that the triplet signal is not modified down to 193 K. The methyl and methine protons of the phosphine appear at 1.90 (m) and 1.20 (dd) ppm. The integration ratio between the hydride and the methine hydrogens (see Experimental Section) was measured as 4.3:6, a value in satisfactory agreement with the 5:6 required by formula **3**. The number of hydrides was independently established by the selectively *coupled* ^{31}P NMR spectrum. The sharp $^{31}P\{^1H\}$ NMR singlet at 43.6 ppm becomes a sextet when only the isopropyl hydrogens are decoupled.

The infrared spectrum of **3** shows three broad Ir–H stretches at 1945, 1780, and 1689 cm^{-1} (compare 1944 cm^{-1} in compound **1**). Bridging hydrides yield intense absorptions in related Ru/Cu compounds.[9]

In order to try to ascertain the character of the hydrogen ligands in **3**, their relaxation time T_1 was determined at variable tem-

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(11) General crystallographic methods: Huffman, J. C.; Lewis, L. N.; Caulton, K. G. *Inorg. Chem.* **1980**, *19*, 2755.

perature. The minimum value observed is 76 ± 5 ms at 193 K and 250 MHz. Although this value is significantly shorter than that found for $IrH_5(PCy_3)_2$[6] (780 ms at 193 K and 500 MHz), it is not conclusive, since, for example, low T_1 values were observed for ReH_7L_2[2] whereas several compounds of this kind were shown by neutron diffraction to adopt a classical structure in the solid state.[12]

Because crystals of this Cu/Ir adduct were unsuitable for structure determination by X-ray crystallography, we examined a copper reagent with a different anion.

The polyhydride complex $IrH_5(P^iPr_3)_2$[10] reacts rapidly at room temperature with 0.5 equiv of $[Cu(MeCN)_4]BF_4$ in acetone to give an off-white solution from which off-white crystals of $[IrH_2(MeCN)_2(P^iPr_3)_2]BF_4$ (**2**) are obtained upon addition of ether and cooling (Scheme I). NMR monitoring of the reaction shows the products to be a 1:1 mixture of **1** and **2**. Addition of excess $[Cu(MeCN)_4]BF_4$ leads to the almost complete transformation of **1** into **2**. This compound is a new example from the series of complexes $[IrH_2S_2L_2]^+$ studied by Crabtree.[13] Compound **2** shows a high-field triplet at δ −22.18 (J_{PH} = 15.2 Hz, intensity 2) in the ^1H NMR (compare[14] −22.2 ppm and 14.8 Hz for *cis*-$[IrH_2(MeCN)_2(PCy_3)_2]PF_6$) together with the methyl groups of the triisopropylphosphine at δ 1.35–1.42 (intensity 36), the unique isopropyl protons at δ 2.46, and the methyl groups of acetonitrile at δ 2.37 (intensity 6).

The solid-state structure of **2** is composed of noninteracting BF_4^- and *cis,cis,trans*-$[IrH_2(MeCN)_2(P^iPr_3)_2]^+$ ions. The pseudooctahedral coordination geometry of the cation shows only modest distortions. The bulky phosphines bend slightly away from the nitriles and toward the hydrides (P–Ir–P = 167.39 (9)°), and the nitriles actually bend toward each other (N–Ir–N = 86.7 (3)°). As a result, P–Ir–N angles (93.17 (22)–95.46 (22)°) are all greater than 90°. The hydride ligands were not detectable in the X-ray data. The nitrile ligands are essentially linear at nitrogen (170.8 (8) and 176.1 (9)°). The Ir–P distances in this cation (2.321 Å) are the same as those to triphenylphosphine in $[Ir(H)_2(PPh_3)_2L_2]^+$, where L_2 = $(OCMe_2)_2$,[13b] *o*-$I_2C_6H_4$,[15] methyl quinoline,[16] and $(MeI)_2$.[17] The P–Ir–P angles in these molecules are 171.6 (1), 166.5 (2), 165.0 (1), and 156.6 (1)°. The Ir–P distances are only slightly longer than the 2.309 (1)-Å distance in $IrH_5(P^iPr_3)_2$.[10] The Ir–N distances (average value 2.14 Å) are slightly longer than those (average value 2.09 Å) in *fac*-$[Os(NCMe)_3(PMe_2Ph)_3]^{2+}$.[18]

This reaction is clearly different in both product identity and reaction stoichiometry from that using CuO_3SCF_3. There is obviously active participation by acetonitrile coordinated initially to the copper reagent. The balanced reaction (eq 2) leaves unidentified the product X, which could either be some soluble aggregate of "CuH" or Cu^0 and $^1/_2$ H_2 (yet no copper metal was detected).

$$IrH_5(P^iPr_3)_2 + [Cu(NCMe)_4]^+ \rightarrow [IrH_2(NCMe)_2(P^iPr_3)_2]^+ + H_2 + 2MeCN + X \quad (2)$$

Reactivity of Compound 3. The formation of **2** in the presence of acetonitrile prompted us to study more generally the reactivity of **3**. While **1** does not react at 25 °C with acetonitrile, deuterium, or cyclopentadiene, **3** reacts with each of these.

Compound **3** is stable in CH_2Cl_2 but reacts rapidly with MeCN to give a 1:1 mixture of **1** and **2** (^1H NMR evidence). The production of **1** here provides additional evidence for the retention of the 5:1 H:Ir stoichiometry in **3**. Moreover, if D_2 (1 atm) is passed through a CH_2Cl_2 solution of **3** for 15 min at 25 °C, ^1H NMR spectra showed the characteristic isotopic modification of chemical shift and integration showed replacement of 70% of hydride by deuteride (integration IrH:CH:CH_3 ratio of 1.25:6:36; see Experimental Section). In contrast, $IrH_5(P^iPr_3)_2$ does not exchange with D_2 under these conditions.

Finally, the reaction between **3** and cyclopentadiene (1 mol/mol of Ir) yields a mixture of both **1** and the new complex **4**. High yields of **4** were obtained by reacting **1** with cyclopentadiene in the presence of 1 mol of either $CuSO_3CF_3$ or HSO_3CF_3. Compound **4** was identified as $[CpIrH(P^iPr_3)_2]SO_3CF_3$ and is analogous to $CpIrH(PPh_3)_2^+$ prepared earlier.[19] The ^1H NMR spectrum shows a hydride triplet at −16.83 ppm (intensity 1, J_{PH} = 30 Hz) and a Cp resonance at 5.95 ppm (intensity 5); a $^{31}P\{^1H\}$ singlet appears at 27.8 ppm. In contrast to the reaction of **3** with cyclopentadiene at 25 °C, $[Ir(H)_2(H_2)_2(P^iPr_3)_2]^+$ (prepared from **1** and $HBF_4 \cdot OEt_2$) requires reflux temperature to react with C_5H_6.

Discussion

In all of the above examples, Cu^+ is the reagent responsible for "activating" the classical polyhydride $IrH_5(P^iPr_3)_2$. The enhanced reactivity can be H_2 displacement (by MeCN), exchange with D_2, or even H_2 displacement with C/H bond activation.

In conclusion, this study shows that a classical polyhydride, $IrH_5(P^iPr_3)_2$, can be activated toward reaction with a nucleophilic ligand like acetonitrile by interaction with a Lewis acid: "Cu^+". The observation of Ir–H/D_2 exchange together with the fact that the analogous but nonclassical polyhydride $RuH_4(PPh_3)_3$ reacts with acetonitrile to give $RuH_2(MeCN)(PPh_3)_3$[20] suggests that, if the ground state of **3** is in fact a classical polyhydride, at least in an equilibrium, one Ir^V moiety is reduced to a reactive Ir^{III} dihydrogen derivative in **3**. In other words, **3** has a thermally accessible dihydrogen form through which it reacts. This activating effect of a Lewis acid is a controllable tool that warrants further study.

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Supplementary Material Available: Tables of full crystallographic data and positional and thermal parameters (3 pages); a listing of observed and calculated structure factors (8 pages). Ordering information is given on any current masthead page.

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