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DIHYDROIRIDIUM DIOLEFIN WITH SULFUR LIGANDS AND RELATED COMPLEXES

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Summary

The reactions with H_2 of the Ir^I cationic complexes $[Ir(COD)L_2]ClO_4$, $[Ir_2\{\mu\text{-}(L-L)\}_2(COD)_2](ClO_4)_2$, $[Ir(COD)L_2L']ClO_4$ COD = 1,5-cyclooctadiene, L and L–L = sulfur mono and bidentate donor ligand, respectively, L′ = Group Vb ligand and related complexes have been studied. Dihydroiridium cationic complexes containing mono and bidentate sulfur ligands have been isolated; these are of two types namely those containing only sulfur ligands and those containing both sulfur and Group Vb ligands. One dihydride carbonyl compound has also been isolated.

Introduction

Oxidative addition of H_2 to d^8 square-planar complexes is an important step in homogeneous catalyses of hydrogenation and hydroformylation [5]. This reaction has been extensively studied for Ir^I complexes, especially for Vaska's complex, $IrCl(CO)(PPh_3)_2$ [6–9], and for cationic diolefin bis(phosphine)iridium(I) complexes [10–14]. The $[Ir(COD)(PR_3)_2]^+$ complexes (COD = 1,5-cyclooctadiene, $R_3 = Pr^i$, Ph_3, or $MePh_2$) are active catalyst precursors for the hydrogenation and isomerisation of alkenes in dichloromethane solutions [6–8], and the dihydro diolefin complexes have been described and studied as intermediates in the homogeneous hydrogenation of olefins [8,9]. The mechanism of the H_2 addition to Ir^I complexes and the influence of electronic and steric factors on the reaction are matters of growing interest [15–19].

We report here the preparation and some spectroscopic properties of some cationic dihydridoiridium(III) complexes mono and dinuclear containing sulfur ligands namely $[IrH_2(COD)L_2]ClO_4$, $[IrH_2(COD)(L-L)]_2(ClO_4)_2$, $[IrH_2(COD)LL']ClO_4$, $[Ir_2H_4(COD)_2(L-L)(PPh_3)_2](ClO_4)_2$ and $[IrH_2(CO)_2(L-L)]_2(ClO_4)_2$ where L and L–L are mono or bidentate sulphur donor ligands respectively and L′ is PPh_3, $AsPh_3$ or $SbPh_3$.

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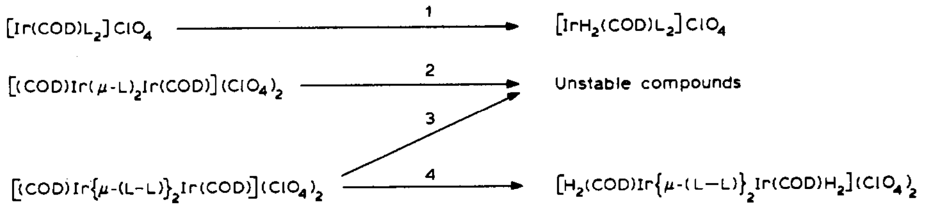


SCHEME 1. Syntheses of dihydridoiridium(III) complexes with sulfur donor ligands. Reaction with H_2: 1. L = tht, tms; 2. L = dms, des; 3. L–L = dmds; 4. L–L = tmdto, dt. Reaction time 3 min.

Results and discussion

When H_2 is bubbled for 3 min at room temperature through dichloromethane solutions of the iridium complexes $[Ir(COD)L_2]ClO_4$ (COD = 1,5-cyclooctadiene, L = tetrahydrothiophene (tht) or trimethylene sulfide (tms), $L_2 = (CH_3S)_2(CH_2)_3$ (dth) and $[Ir(COD)(L-L)]_2(ClO_4)_2$ (L–L) = $(t\text{-}BuS)_2(CH_2)_2$ (tmdto) or 1,4-dithiacyclohexane (dt)) either previously isolated or used as prepared "in situ" [1], the solutions become lighter in colour and the corresponding dihydridoiridium(III) complexes can be isolated by addition of diethyl ether (Scheme 1).

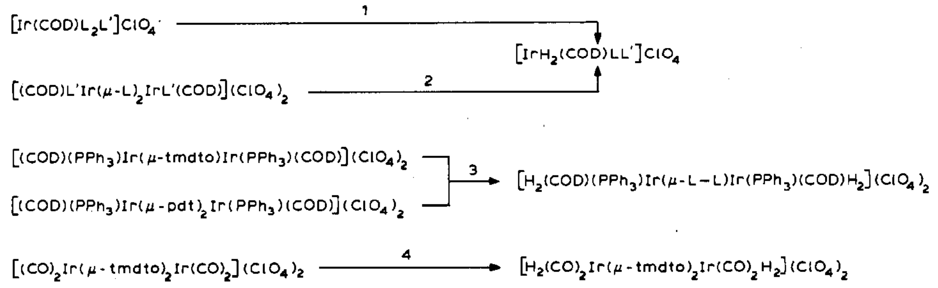
The products from the reactions of complexes with L = SMe_2 (dms), SEt_2 (des) and L–L = $(CH_3S)_2$ (dmds) [1] are unstable (Scheme 1).

Bubbling of H_2 through dichloromethane solutions of the $[Ir(COD)L_2L'](ClO_4)$ (L = tht or tms, L′ = PPh_3, $AsPh_3$ or $SbPh_3$) $[Ir(COD)LL']_2(ClO_4)_2$ (L = dms or des L′ = PPh_3, $AsPh_3$ or $SbPh_3$) $[Ir_2(COD)_2(tmdto)(PPh_3)_2](ClO_4)_2$ and $[Ir(COD)(dt)(PPh_3)]_2(ClO_4)_2$ [1,2] similarly gives the corresponding dihydridoiridium(III) complexes (Scheme 2). One hydrido carbonyl compound was isolated $[IrH_2(CO)_2(tmdto)]_2(ClO_4)_2$ by this procedure (Scheme 2). The starting carbonyl complex used was reported in [3].

The complexes can also be obtained by stirring the dichloromethane solutions under hydrogen at room temperature for a few minutes.

The C and H analyses, conductivity data, melting points and yields of the isolated complexes are listed in Table 1 and Table 2.

The solids are fairly air-stable, except for XX, and are soluble in the usual organic solvents except for II and IV, which are almost insoluble.



SCHEME 2. Syntheses of the mixed dihydridoiridium(III) complexes with sulfur donor ligands. Reaction with H_2: 1. L = tht, tms; L′ = PPh_3, $AsPh_3$, $SbPh_3$; 2. L = dms, des; L′ = PPh_3, $AsPh_3$, $SbPh_3$; 3. L–L = tmdto. Reaction time 3 min.

TABLE 1

ANALYTICAL RESULTS, CONDUCTIVITIES DATA, MELTING POINTS AND YIELDS OF THE DIHYDRIDOIRIDIUM(III) COMPLEXES WITH SULFUR DONOR LIGANDS

Complex	Found(%) (calcd.)(%)		Conductivity data		Yield (%)	M.p. [a] (°C)
	C	H	A^{b}	$A_M{}^{c}$		
$[IrH_2(COD)(tht)_2]ClO_4$ (I)	32.74 (33.20)	4.90 (5.19)	-	123	86	90–95
$[IrH_2(COD)(tms)_2]ClO_4$ (II)	30.88 (30.55)	4.49 (4.73)	-	114	82	160–165
$[H_2(COD)Ir(\mu\text{-tmdto})_2Ir(COD)H_2](ClO_4)_2$ (III)	34.65 (35.53)	5.66 (5.92)	812	225	86	132–137
$[H_2(COD)Ir(\mu\text{-pdt})_2Ir(COD)H_2](ClO_4)_2$ (IV)	26.88 (27.59)	4.01 (4.21)	499 [d]	123 [d]	87	150–160
$[IrH_2(COD)dth]ClO_4$ (V)	28.56 (29.00)	4.67 (4.85)	-	120	78	80–85

[a] Decomposes. [b] Slope found for Onsager's equation $\Lambda_e = \Lambda_0 - A\sqrt{c}$. [c] Measured in acetone solution (ohm^{-1} cm^2 mol^{-1}). [d] Measured in nitromethane solution (ohm^{-1} cm^2 mol^{-1}).

The conductivities of the products were determined in acetone or nitromethane solutions of various concentrations. The values of A in Onsager's equation ($\Lambda_e = \Lambda_0 - A\sqrt{c}$) measured in the concentration range ca. 10^{-3}–10^{-5} M confirmed the binuclear nature of the complexes [20,21].

IR spectra

The Ir spectra of all the isolated complexes show bands due to the coordinated sulfur ligands together with those due to the uncoordinated perchlorate anion and to the Ir–H stretching modes [22,23] (Table 3).

The spectra (Nujol mull) of $[IrH_2(COD)L_2]ClO_4$ (L = tht, tms) show two sharp bands, suggesting that the two hydrogens occupy *cis* positions [13,24], and in the case of $[IrH_2(COD)(tmdto)]_2(ClO_4)_2$ the spectrum also shows two bands consistent with a *cis* disposition of the hydrogens and with similar environments for the two iridium(I) atoms. In contrast, for $[IrH_2(COD)(dth)]ClO_4$, the spectrum shows only one sharp band at 2020s cm^{-1}, implying a *trans* arrangement of the hydrogens.

The spectra of the $[IrH_2(COD)(dt)]_2(ClO_4)_2$, $[IrH_2(COD)LL']ClO_4$ and $[Ir_2H_4(COD)_2(L\text{–}L)(PPh_3)_2](ClO_4)_2$ complexes initially show two ν(Ir–H) bands (Table 3), characteristic of a *cis* disposition of the hydrogens, but there is rapid isomerization to a *trans*-isomer, and after a few minutes only a single sharp ν(Ir–H) band is present, at 2020vs cm^{-1} (Fig. 1).

It is noteworthy that in the initially *cis* complexes $[IrH_2(COD)LL']ClO_4$ (L = sulfur ligand, L′ = PPh_3, $AsPh_3$ or $SbPh_3$) the ν(Ir–H) bands are always shifted to higher frequencies in the mixed complexes compared to those for $[IrH_2(COD)L_2]ClO_4$, and $\nu(Ir\text{–}H)_{PPh_3} > \nu(Ir\text{–}H)_{AsPh_3} > \nu(Ir\text{–}H)_{SbPh_3}$.

The IR spectrum of $[IrH_2(CO)_2(\mu\text{-tmdto})]_2(ClO_4)_2$ shows two Ir–H frequencies, together with the ν(CO) bands at 2105s, 2070s and 1980s cm^{-1}. This compound loses H_2 progressively to regenerate $[Ir(CO)_2(tmdto)]_2(ClO_4)_2$. The stretching fre-

TABLE 2

ANALYTICAL RESULTS, CONDUCTIVITIES DATA, MELTING POINTS AND YIELDS OF THE DIHYDROIRIDIUM(III) MIXED COMPLEXES WITH SULFUR DONOR LIGANDS

Complex	Found (calcd.) (%)		Conductivity data		Yield (%)	M.p. [a] (°C)
	C	H	Λ [b]	Λ_M [c]		
$[IrH_2(COD)(tht)PPh_3]ClO_4$ (VI)	47.12	4.72	-	112	83	102–108
	(47.86)	(4.92)				
$[IrH_2(COD)(tht)AsPh_3]ClO_4$ (VII)	44.36	4.51	-	119	86	105–108
	(45.21)	(4.65)				
$[IrH_2(COD)(tht)SbPh_3]ClO_4$ (VIII)	42.26	4.26	-	127	79	94–97
	(42.69)	(4.39)				
$[IrH_2(COD)(tms)PPh_3]ClO_4$ (IX)	46.10	4.74	-	128	77	120–125
	(47.15)	(4.74)				
$[IrH_2(COD)(tms)AsPh_3]ClO_4$ (X)	43.64	4.61	-	122	92	108–112
	(44.50)	(4.48)				
$[IrH_2(COD)(tms)SbPh_3]ClO_4$ (XI)	40.90	4.02	-	128	86	130–134
	(41.98)	(4.22)				
$[IrH_2(COD)(dms)PPh_3]ClO_4$ (XII)	45.05	5.00	-	124	76	90–95
	(46.28)	(4.82)				
$[IrH_2(COD)(dms)AsPh_3]ClO_4$ (XIII)	42.15	4.36	-	130	84	80–85
	(43.64)	(4.55)				
$[IrH_2(COD)(dms)SbPh_3]ClO_4$ (XIV)	40.80	4.30	-	124	92	96–100
	(41.13)	(4.28)				
$[IrH_2(COD)(des)PPh_3]ClO_4$ (XV)	46.31	4.99	-	122	80	86–90
	(47.74)	(5.17)				
$[IrH_2(COD)(des)AsPh_3]ClO_4$ (XVI)	44.12	4.74	-	128	91	90–94
	(45.11)	(4.89)				
$[IrH_2(COD)(des)SbPh_3]ClO_4$ (XVII)	42.00	4.39	-	125	85	93–97
	(42.60)	(4.62)				
$[H_2(COD)(PPh_3)Ir(\mu\text{-tmdto})Ir(PPh_3)(COD)H_2](ClO_4)_2$ (XVIII)	47.30	5.50	406 [d]	117 [d]	98	145–150
	(48.51)	(5.20)				
$[H_2(COD)(PPh_3)Ir(\mu\text{-pdt})Ir(PPh_3)(COD)H_2](ClO_4)_2$ (XIX)	46.08	4.65	454 [d]	123 [d]	85	140–145
	(46.41)	(4.56)				
$[H_2(CO)_2Ir(\mu\text{-tmdto})_2Ir(CO)_2H_2](ClO_4)_2$ (XX)	26.97	4.51	736	232	89	90–95
	(25.91)	(4.32)				

[a] Decomposes. [b] Slope found for Onsager's equation $\Lambda_e = \Lambda_0 - A\sqrt{c}$. [c] Measured in acetone solution ($ohm^{-1}\ cm^2\ mol^{-1}$). [d] Measured in nitromethane solution ($ohm^{-1}\ cm^2\ mol^{-1}$).

quencies of the coordinated carbon monoxide ligands are shifted to higher frequencies in the dihydride complex [3] (Fig. 2).

^{1}H NMR data

Chatt et al., and Wilkinson et al. [25,26] observed that for dihydridoiridium(III) complexes the NMR chemical shift of the hydride depends on the ligands *trans* to hydride.

The ^{1}H NMR spectra of $CDCl_3$ solutions of the new complexes at 20°C were recorded and show Ir–H resonances [22,23]. In the ^{1}H NMR spectrum of $[IrH_2(COD)(tht)_2]ClO_4$ two close metal–hydride signals can be distinguished, one at

TABLE 3

INFRARED DATA FOR THE DIHYDROIRIDIUM(III) COMPLEXES

Complex	ν(Ir–H)
$[IrH_2(COD)(tht)_2]ClO_4$	2125m, 2180m
$[IrH_2(COD)(tms)_2]ClO_4$	2095m, 2180m
$[H_2(COD)Ir(\mu\text{-tmdto})_2Ir(COD)H_2](ClO_4)_2$	2115s, 2190m
$[H_2(COD)Ir(\mu\text{-pdt})_2Ir(COD)H_2](ClO_4)_2$	2120m, 2155m
$[IrH_2(COD)dth]ClO_4$	2120s
$[IrH_2(COD)(tht)PPh_3]ClO_4$	2155m, 2220m
$[IrH_2(COD)(tht)AsPh_3]ClO_4$	2140m, 2200m
$[IrH_2(COD)(tht)SbPh_3]ClO_4$	2090m, 2145m
$[IrH_2(COD)(tms)PPh_3]ClO_4$	2150m, 2220m
$[IrH_2(COD)(tms)AsPh_3]ClO_4$	2135m, 2200m
$[IrH_2(COD)(tms)SbPh_3]ClO_4$	2085m, 2150m
$[IrH_2(COD)(dms)PPh_3]ClO_4$	2145m, 2210m
$[IrH_2(COD)(dms)AsPh_3]ClO_4$	2140m, 2180m
$[IrH_2(COD)(dms)SbPh_3]ClO_4$	2130m, 2090m
$[IrH_2(COD)(des)PPh_3]ClO_4$	2150m, 2210m
$[IrH_2(COD)(des)AsPh_3]ClO_4$	2145m, 2180m
$[IrH_2(COD)(des)SbPh_3]ClO_4$	2090m, 2135m
$[H_2(COD)PPh_3Ir(\mu\text{-tmdto})IrPPh_3(COD)H_2](ClO_4)_2$	2195s, 2225m
$[H_2(COD)PPh_3Ir(\mu\text{-pdt})IrPPh_3(COD)H_2](ClO_4)_2$	2130m, 2180m
$[H_2(CO)_2Ir(\mu\text{-tmdto})_2Ir(CO)_2H_2](ClO_4)_2$	2140m, 2175m

δ −13.07 and the other at δ 13.56 ppm. However only one set of signals from COD or tht are observed (δ 1.85–2.30 ppm CH_2–C(COD); δ 2.10 ppm CH_2–C(tht)); δ 3.17 ppm CH_2–S(tht) and δ 4.11 ppm CH–C(COD)). These data together with the IR data suggest a structure in which the two *cis*-hydride ligands are practically equivalent.

In the case of $[IrH_2(COD)(tmdto)]_2(ClO_4)_2$ the ^1H NMR spectrum shows two singlets for the hydride ligands, at δ −12.30 and δ −14.32 ppm, suggesting that two different atoms *trans* to the hydride ligands are present [25]. Splitting of the ^1H NMR signals from the COD and tmdto ligands confirm this (δ 1.55 2.22 ppm CH_2–C (COD); δ 2.84, 3.15 CH_2–S (tmdto); 1.40, 1.47 CH_3–C (tmdto) and 4.02, 4.81 ppm CH–C(COD)).

The $CDCl_3$ solutions of the dihydroiridium mixed complexes at 20°C are unstable, but Ir–H resonances can be distinguished (Table 4). The $[IrH_2(COD)L(PPh_3)]ClO_4$ (L = tht, tms) and $[Ir_2H_4(COD)_2(tmdto)(PPh_3)_2](ClO_4)_2$ complexes show a triplet hydride resonance, probably due to coupling with *cis* phosphorus nuclei, ^2J(P–H) 20 Hz [13], suggesting that redistributions take place in solution.

The spectra of the mixed complexes $[IrH_2(COD)LL'](ClO_4)$ (L = tht, L′ = $AsPh_3$; L = tms, dms, L′ = $SbPh_3$) each show only one singlet for Ir–H, consistent with equivalent *trans* hydride ligands. In the case of the complex $[IrH_2(COD)(tms)(AsPh_3)]ClO_4$, three resonances are observed, and this could be attributed to equilibration between *cis* and *trans* isomers in solution. However, in view of the instability of the solutions and of the ^1H NMR data for the complexes containing PPh_3, redistribution reactions in the solutions of mixed complexes cannot be excluded.

The $[IrH_2(CO)_2(tmdto)]_2(ClO_4)_2$ complex show two singlets for the hydride

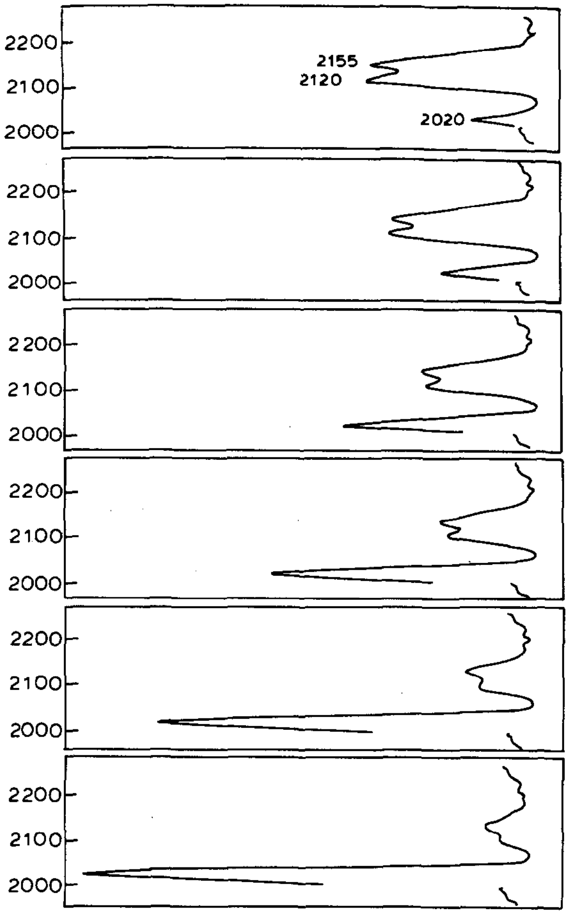


Fig. 1. Variation of IR spectrum in 2200–2000 cm^{-1} region for $[H_2(COD)Ir(\mu\text{-pdt})_2Ir(COD)H_2](ClO_4)_2$. Spectra recording every 30 min.

ligands, (Table 4), one at δ −10.14 and the other at −13.08 ppm. As expected the signals from the tmdto ligand are split (δ 3.27, 3.57 CH_2–S; 1.62, 1.68 ppm CH_3–C). The signal at δ −10.14 ppm may be tentatively assigned to a hydride *trans* to a carbonyl group [26].

Experimental

The previously reported Ir^I cationic complexes used as starting materials were prepared by standard Schlenk tube techniques under nitrogen [1–3]. Hydrogenations were carried out at room temperature by bubbling H_2 through dichloromethane solutions. All the solvents were previously deoxygenated.

The elemental analyses were carried out with a Perkin–Elmer 240B microanalyzer. The IR spectra were recorded on a Beckman IR 4260 spectrophotometer using Nujol mulls between polyethylene sheets. Conductivities were measured in acetone solutions in the concentration range ca. 1×10^{-4}–5×10^{-4} with a Red. Copenh. CDM3 Radiometer conductimeter. Values of Λ were determined from

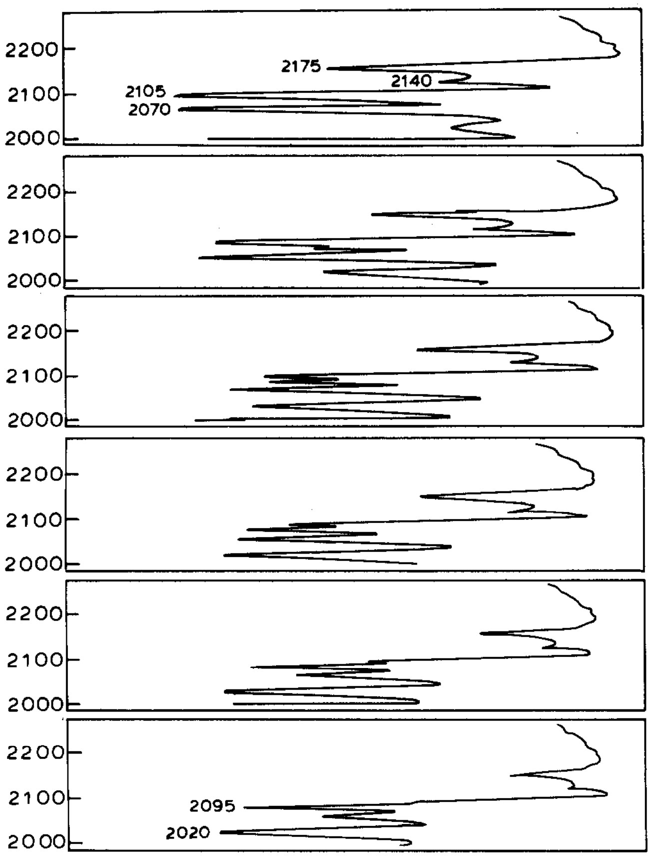


Fig. 2. Variation of IR spectrum in 2200–2000 cm^{-1} region for $[H_2(CO)_2Ir(\mu\text{-tmdto})_2Ir(CO)_2H_2](ClO_4)_2$. Spectra recording every 10 min. The $\nu(CO)$ in the last spectrum (2095, 2020 cm^{-1}) correspond to those of $[(CO)_2Ir(\mu\text{-tmdto})_2Ir(CO)_2](ClO_4)_2$.

Onsager's equation $\Lambda_e = \Lambda_0 - A\sqrt{c}$ using several concentrations in acetone or nitromethane solutions in the 10^{-3}–10^{-5} *M* range. The ^1H NMR spectra were measured on a XL-200 Varian spectrometer using $CDCl_3$ as the solvent and $SiMe_4$

TABLE 4

^1H NMR DATA FOR MIXED DIHYDROIRIDIUM COMPLEXES

Complex	δ(Ir–H) (ppm)
$[IrH_2(COD)(tht)PPh_3]ClO_4$	−17.10
$[IrH_2(COD)(tht)AsPh_3]ClO_4$	−14.00
$[IrH_2(COD)(tms)PPh_3]ClO_4$	−16.20
$[IrH_2(COD)(tms)AsPh_3]ClO_4$	−14.2, −13.7, −13.3
$[IrH_2(COD)(tms)SbPh_3]ClO_4$	−14.70
$[IrH_2(COD)(dms)PPh_3]ClO_4$	−17.30
$[IrH_2(COD)(dms)SbPh_3]ClO_4$	−14.70
$[H_2(COD)(PPh_3)Ir(\mu\text{-tmdto})Ir(PPh_3)(COD)H_2](ClO_4)_2$	−16.00
$[H_2(CO)_2Ir(\mu\text{-tmdto})_2Ir(CO)_2H_2](ClO_4)_2$	−13.08, −10.14

as the internal reference at 20°C. Melting points were determinated with a Buchi 510 Melting Point apparatus.

Preparation of $[IrH_2(COD)L_2]ClO_4$ *complexes (L = tht or tms;* L_2 *= dth)*

H_2 was bubbled for 3 min, at room temperature through solutions of $[Ir(COD)L_2]ClO_4$ (L = tht, tms; L_2 = dth) (0.1 mmol) prepared "in situ" in dichloromethane (1–2 ml) [1]. The solutions became lighter in colour and addition of diethyl ether gave the corresponding dihydridoiridium(III) complexes. The solids, pale yellow, pink, and orange respectively, were filtered off, washed with ether, and vacuum dried.

Preparation of $[IrH_2(COD)(L\text{–}L)]_2(ClO_4)_2$ *complexes (L–L = tmdto or dt)*

H_2 was bubbled through dichloromethane solutions of $[Ir(COD)(L\text{–}L)]_2(ClO_4)_2$ (L–L = tmdto or dt) (0.05 mmol) (prepared "in situ", [1]) for 3 min, at room temperature. Diethyl ether was then added and the dihydridoiridium solids, pale yellow and white, respectively, were filtered off, washed with ether, and vacuum dried.

Preparation of $[IrH_2(COD)LL'](ClO_4)$ *complexes (L = tht, tms, dms or des; L' =* PPh_3*,* $AsPh_3$ *or* $SbPh_3$*)*

Bubbling of H_2 through dichloromethane solutions of $[Ir(COD)L_2L']ClO_4$ (L = tht or tms) (0.1 mmol) or $[Ir(COD)LL']_2(ClO_4)_2$ (L = dms or des) (0.05 mmol) (prepared as [2] for 3 min. caused decoloration of the solutions. The iridium(III) complexes, pale yellow, pale orange or pink, were precipitated by addition of diethyl ether, filtered off, washed with ether, and vacuum dried.

Preparation of $[Ir_2H_4(COD)_2(L\text{–}L)(PPh_3)_2](ClO_4)_2$ *complexes (L–L = tmdto or dt)*

H_2 was bubbled for 3 min at room temperature through solutions of $[Ir_2(COD)_2(tmdto)(PPh_3)_2](ClO_4)_2$ and $[Ir(COD)(dt)PPh_3)]_2(ClO_4)_2$ (0.05 mmol.), (prepared as in [2]) in dichloromethane. The solutions became lighter in colour and addition of diethyl ether gave the corresponding $[Ir_2H_4(COD)_2(L\text{–}L)(PPh_3)_2](ClO_4)_2$ (L–L = tmdto or dt). The solids, pale yellow and pink, respectively, were filtered off, washed with ether, and vacuum dried.

Preparation of $[IrH_2(CO)_2(tmdto)]_2(ClO_4)_2$ *complex*

H_2 was bubbled for 3 min through a dichloromethane solution of $[Ir(CO)_2(tmdto)]_2(ClO_4)_2$ (0.05 mmol) (prepared as in [2]) at room temperature. The solution became lighter in colour. The yellow product was precipitated by addition of diethyl ether, filtered off, washed with ether, and vacuum dried.

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