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BIS-BRIDGED DIRHODIUM OR DIIRIDIUM COMPLEXES DERIVED FROM DIAMINE- OR DIHYDROXY-NAPHTHALENES

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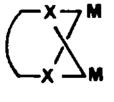
Summary

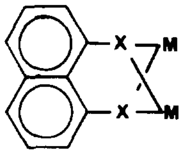
Homo- and hetero-dinuclear bis(di-μ-amide or -oxo) rhodium(I) and iridium(I) complexes of the formulae $MM'(\mu\text{-}L)(COD)_2$ ($M = M' =$ Rh, Ir; L = 1,8-$(NH)_2$naphth (naphth = naphthalene), 1,8-$(O)_2$naphth; $M = M' =$ Rh; L = 2,3-$(NH)_2$naphth, 2,3-$(O)_2$naphth; M = Rh, M′ = Ir, L = 1,8-$(NH)_2$naphth, 1,8-$(O)_2$naphth) are described. The related carbonyl derivatives $Rh_2(\mu\text{-}L)(CO)_4$ (L = 1,8-$(NH)_2$naphth, 2,3-$(O)_2$naphth), $Rh_2(\mu\text{-}1,8\text{-}(NH)_2\text{naphth})(CO)_2(PR_3)_2$ (R = Ph, OEt, *o*-tolyl) have been obtained by carbonylation. Some mononuclear ionic complexes of formulae $[Rh(COD)(LH_2)]ClO_4$ (LH_2 = 1,8-$(NH_2)_2$naphth, 2,3-$(OH)_2$naphth), $NBu_4[Rh(2,3\text{-}(O)_2\text{naphth})(COD)]$ and $[Rh(CO)_2(1,8\text{-}(NH_2)_2\text{naphth})]ClO_4$ have been also obtained.

Introduction

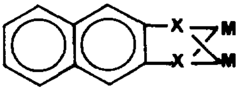
Recently the ability of a number of difunctional ligands to act as bridges connecting two metal atoms has received considerable interest. In particular a variety of bridging P–P (e.g. bis(diphenylphosphino)methane [1]) or N–N (e.g. 1,8-naphthyridine [2]) ligands have been used. Carboxylates can also form dinuclear complexes [3,4], but there are few known complexes in which the two donor centres are bound to the transition metals in both a bridging and chelating manner as in structure I [5,6]. In a preliminary report [7] we commented recently on the ability of



(I)



(IIa, X = NH;
IIb, X = O)



(IIIa, X = NH;
IIIb, X = O)

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TABLE 1

ANALYTICAL AND PHYSICAL DATA FOR THE DINUCLEAR COMPLEXES

Compound	Analyses (Found (calcd.)(%))			Colour	M.W. [a] (Found (calcd.))	Yield (%)	Selected IR bands (cm^{-1})
	N	C	H				
$Rh_2(\mu$-1,8-$(NH)_2$naphth)$(COD)_2$	4.52 (4.84)	54.03 (53.97)	5.93 (5.53)	orange	597 (578)	71	3285 [d], 1290 [e]
$Ir_2(\mu$-1,8-$(NH)_2$naphth)$(COD)_2$	3.91 (3.70)	41.11 (41.26)	4.28 (4.26)	deep-red	[b]	75	3270 [d], 1280 [e]
$Rh_2(\mu$-2,3-$(NH)_2$naphth)$(COD)_2$	4.79 (4.84)	54.27 (53.97)	5.46 (5.53)	orange	548 (578)	71	3330 [d], 1230 [e]
$Rh_2(\mu$-1,8-$(O)_2$naphth)$(COD)_2$	–	54.38 (53.81)	5.31 (5.21)	yellow	621 (580)	68	1260 [f]
$Ir_2(\mu$-1,8-$(O)_2$naphth)$(COD)_2$	–	40.31 (41.15)	3.65 (3.98)	yellow	[b]	62	1270 [f]
$Rh_2(\mu$-2,3-$(O)_2$naphth)$(COD)_2$	–	53.63 (53.81)	5.32 (5.21)	orange	609 (580)	75	1315 [f]
RhIr(μ-1,8-$(O)_2$naphth)$(COD)_2$	–	46.30 (46.64)	4.83 (4.52)	yellow	[b]	60	1270 [f]
RhIr(μ-1,8-$(NH)_2$naphth)$(COD)_2$	3.79 (4.20)	47.62 (46.75)	4.48 (4.80)	deep-red	[b]	85	3285 [d], 1290 [e]
$Rh_2(\mu$-1,8-$(NH)_2$naphth)$(CO)_4$	5.68 (5.90)	35.73 (35.44)	2.14 (1.68)	yellow	478 (474)	75	3370 [d], 1290 [e] 2070, 2050, 1995 [g]
$Rh_2(\mu$-2,3-$(O)_2$naphth)$(CO)_4$	–	34.80 (35.29)	1.31 (1.26)	gold-green	[c]	73	1250 [f] 2080, 2050, 1995 [g]
$Rh_2(\mu$-1,8-$(NH)_2$naphth)$(CO)_2$-$(PPh_3)_2$	2.54 (2.97)	60.33 (61.16)	4.79 (4.06)	yellow	991 (942)	83	3340 [d] 1950 [g]
$Rh_2(\mu$-1,8-$(NH)_2$naphth)$(CO)_2$-$(P(OEt)_3)_2$	3.77 (3.73)	37.70 (38.42)	4.95 (5.10)	yellow	723 (750)	62	3360 [d] 1965 [g]
$Rh_2(\mu$-1,8-$(NH)_2$naphth)$(CO)_2$-(P(o-tolyl)$_3)_2$	2.65 (2.73)	62.53 (63.17)	5.31 (4.91)	yellow	1031 (1026)	68	3340 [d] 1950 [g]

[a] Ca. 2×10^{-3} *M* chloroform solutions. [b] Decomposes. [c] Insoluble. [d] ν(NH). [e] ν(C–N). [f] ν(C–O) (Nujol mulls. [g] ν(C≡O) (dichloromethane solution).

the deprotonated 1,8-diaminenaphthalene (IIa) to act as di(μ-amide) bridging ligand, and we now describe a simple and general procedure which allows the preparation of new dirhodium or diiridium complexes containing deprotonated 1,8-diaminenaphthalene (IIa), 1,8-dihydroxynaphthalene (IIb), 2,3-diaminenaphthalene (IIIa) and 2,3-dihydroxynaphthalene (IIIb) as bis-dihapto-bridging ligands, along with some related mononuclear ionic compounds.

Results and discussion

Dinuclear cyclooctadiene complexes

The reaction of $[M(\mu\text{-OMe})(COD)]_2$ (COD = 1,5-cyclooctadiene) complexes with stoichiometric amounts of diamine- or dihydroxy-naphthalene ligands (LH_2) causes deprotonation of the ligands and formation of dinuclear complexes according to eq. 1:

$$[M(\mu\text{-OMe})(COD)]_2 + LH_2 \rightarrow M_2(\mu\text{-L})(COD)_2 + 2\ MeOH \qquad (1)$$

LH_2 = 1,8-$(NH_2)_2$naphth (M = Rh,Ir); 1,8-$(OH)_2$naphth (M = Rh,Ir); 2,3-$(NH_2)_2$naphth (M = Rh); 2,3-$(OH)_2$naphth (M = Rh).

Formation of the related iridium complexes with ligands IIIa or IIIb has been observed, but the low stability of the red or orange complexes prepared "in situ" has prevented their characterization.

IR spectra (Table 1) show the presence of ν(N–H) bands from the deprotonated diaminenaphthalene ligands and ν(C–O) bands corresponding to the deprotonated dihydroxynaphthalene ligands. Molecular weights in solution correspond to dinuclear species in which the ligands are probably acting as di(μ-amide) or di(μ-oxo) bridging ligands (Structure II and III). The ^1H NMR data for the complexes $Rh_2(\mu\text{-1,8-(NH)}_2\text{naphth})(COD)_2$ or $Rh_2(\mu\text{-2,3-(O)}_2\text{naphth})(COD)_2$ support the proposed formulation (Table 2). An alternative route to $M_2(\mu\text{-1,8-(NH)}_2\text{naphth})(COD)_2$ complexes involves the reaction of M(acac)(COD) (M = Rh or Ir) with the ligand according to eq. 2:

$$2\ M(acac)(COD) + 1,8\text{-}(NH_2)_2\text{naphth} \rightarrow M_2(\mu\text{-1,8-(NH)}_2\text{naphth})(COD)_2 + 2\ H(acac) \qquad (2)$$

Nutton and Maitlis have previously reported μ-amide dirhodium or iridium(III) complexes of formula $[M_2(C_5Me_5)_2(\mu\text{-NHPh})(\mu\text{-OH})_2]^+$ [8]. Amide-bridged compounds are relatively rare [9], and as far as we know no related di(μ-amide) or di(μ-oxo)-bridged dirhodium or iridium complexes have been previously described.

The complex $Rh_2(\mu\text{-2,3-(O)}_2\text{naphth})(COD)_2$ reacts with excess of 2,3-$(OH)_2$naphth to give yellow solids of variable composition. Probably molecules of the ligand are linked by hydrogen bonds to the oxygens of the $[2,3\text{-(O)}_2\text{naphth}]^{2-}$ (or $[2,3\text{-O,(OH)naphth}]^-$) anion coordinated to rhodium; in this connection it is noteworthy that the crystal structure of $Rh(C_5Me_5)(C_6H_4O_2)$ has shown the presence of catechol molecules of solvation linked to the oxygens of the catecholatorhodium moiety [10].

The heterodinuclear $RhIr(\mu\text{-1,8-(O)}_2\text{naphth})(COD)_2$ complex can be prepared by reaction of equimolecular amounts of $Rh_2(\mu\text{-1,8-(O)}_2\text{naphth})(COD)_2$ and $Ir_2(\mu\text{-1,8-(O)}_2\text{naphth})(COD)_2$. However, such a redistribution reaction is not observed for the related $M_2(\mu\text{-1,8-(NH)}_2\text{naphth})(COD)_2$ compouds, but the heterodinuclear complex

TABLE 2

^{1}H NMR CHEMICAL SHIFTS

Complex	$\delta(CDCl_3)$ (ppm from TMS)		
	naphth	COD	NH
$Rh_2(\mu\text{-}1,8\text{-}(NH)_2naphth)(COD)_2$	7.23(d,2)	3.83(br,4)	3.01(s,2)
	7.02(t,2)	3.63(br,4)	
	6.54(d,2)	2.58(br,4)	
		2.21(m,4)	
		1.75–1.96(m,8)	
$Rh_2(\mu\text{-}2,3\text{-}(O)_2naphth)(COD)_2$	7.21(m,2)	4.15(br,8)	
	7.17(m,2)	2.44(br,4)	
	5.51(s,2)	2.19(br,4)	
		1.73–1.96(m,8)	

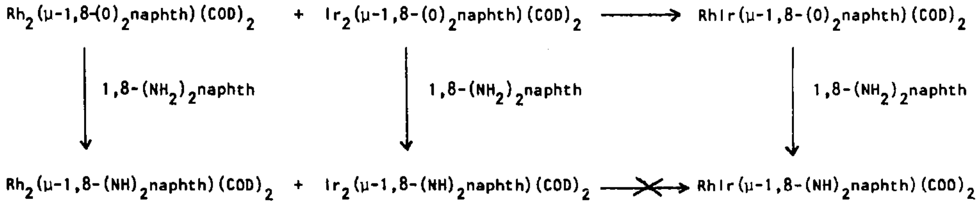
$RhIr(\mu\text{-}1,8\text{-}(NH)_2naphth)(COD)_2$ can be prepared by addition of $1,8\text{-}(NH_2)_2naphth$ to the complex $RhIr(\mu\text{-}1,8\text{-}(O)_2naphth)(COD)_2$. Displacement of the coordinated IIa ligand by reaction of $M_2(\mu\text{-}1,8\text{-}(O)_2naphth)(COD)_2$ complexes with 1,8-diaminonaphthalene is general (see Scheme 1).

An alternative route for the synthesis of heterodinuclear $RhIr(\mu\text{-}L)(COD)_2$ (L = IIa, IIb or IIIb) complexes involves the addition of the appropriate ligand to equimolecular mixtures of $[Rh(\mu\text{-}OMe)(COD)]_2$ and $[Ir(\mu\text{-}OMe)(COD)]_2$. The homo- or heterodinuclear formulation of the above-mentioned complexes is supported by infrared studies on carbonylation reactions. Thus, the IR spectrum of the solution obtained by carbonylation of $RhIr(\mu\text{-}1,8\text{-}(NH)_2naphth)(COD)_2$ shows four ν(CO) bands, at 2075, 2050, 2005 and 1985 cm^{-1}, not coincident with those expected for the corresponding homodinuclear $Rh_2(\mu\text{-}1,8\text{-}(NH)_2naphth)(CO)_4$ derivative (see below).

Mononuclear cyclooctadiene complexes

The addition of 1,8-diaminenaphthalene or 2,3-dihydroxynaphthalene to $[Rh(COD)(Me_2CO)_x]ClO_4$ (prepared "in situ" by treating $[Rh(\mu\text{-}Cl)(COD)]_2$ with $AgClO_4$) results in immediate formation of $[Rh(COD)(1,8\text{-}(NH_2)_2naphth)]ClO_4$ or $[Rh(COD)(2,3\text{-}(OH)_2naphth)]ClO_4$. These complexes behave as completely dissociated 1/1 electrolytes in acetone (Table 3), but in the solid state the absorptions expected for the perchlorate anion (T_d) [11] are split, probably due to some $XH\ldots OClO_3$ (X = NH or O) interaction in the solid state. A similar $NH\ldots OClO_3$ interaction has been recently reported for $[Rh(H_2bbzim)L_2]ClO_4$ complexes (H_2-

SCHEME 1



bbzim = 2,2′-bibenzimidazole; L_2 = COD or L = CO) [12]. IR bands in the 3400–3100 cm^{-1} region due to ν(N–H) or ν(OH) are observed for these complexes.

Interestingly treatment of a methanol solution of [Rh(COD)(2,3-$(OH)_2$naphth)]ClO_4 with potassium hydroxide and tetrabutylammonium chloride gives NBu_4[Rh(2,3-$(O)_2$naphth)(COD)]. This reaction can be reversed by addition of perchloric acid, according to eq. 3:

$$[Rh(COD)(2,3\text{-}(OH)_2naphth)]ClO_4 \underset{HClO_4}{\overset{KOH/MeOH;\ NBu_4Cl}{\rightleftharpoons}} NBu_4[Rh(2,3\text{-}(O)_2naphth)(COD)] \quad (3)$$

An alternative and more direct route for the synthesis of NBu_4[Rh(2,3-$(O)_2$naphth)(COD)] is the reaction of $[Rh(\mu\text{-}Cl)(COD)]_2$ with potassium hydroxide in methanol (where $[Rh(\mu\text{-}OMe)(COD)]_2$ must be present) and subsequent addition of 2,3-dihydroxynaphthalene. However attempts to prepare the related complex NBu_4[Rh(1,8-$(NH)_2$naphth)(COD)] by this method were unsuccessful because of formation of the dinuclear complex $Rh_2(\mu\text{-}1,8\text{-}(NH)_2naphth)(COD)_2$.

Balch and coworkers have reported related anionic dioxolene complexes of formula $[Rh(1,2\text{-}O_2C_6Cl_4)]^-$ along with some related mono- or dinuclear derivatives [13,14].

Carbonylation reactions

Bubbling of carbon monoxide at atmospheric pressure through solutions of representative dinuclear or mononuclear diolefin rhodium complexes results in displacement of the diolefin and formation of the corresponding carbonyl derivatives, according to eqs. 4–6:

$$[Rh(COD)(1,8\text{-}(NH_2)_2naphth)]ClO_4 + 2\ CO \rightarrow [Rh(CO)_2(1,8\text{-}(NH_2)_2naphth)]ClO_4 + COD \quad (4)$$

$$NBu_4[Rh(2,3\text{-}(O)_2naphth)(COD)] + 2\ CO \rightarrow NBu_4[Rh(2,3\text{-}(O)_2naphth)(CO)_2] + COD \quad (5)$$

$$Rh_2(\mu\text{-}L)(COD)_2 + 4\ CO \rightarrow Rh_2(\mu\text{-}L)(CO)_4 + 2\ COD \quad (6)$$

(L = IIa or IIIb)

The $Rh_2(\mu\text{-}1,8\text{-}(NH)_2naphth)(CO)_4$ complex was also prepared by treating $Rh(acac)(CO)_2$ with 1,8-diaminenaphthalene.

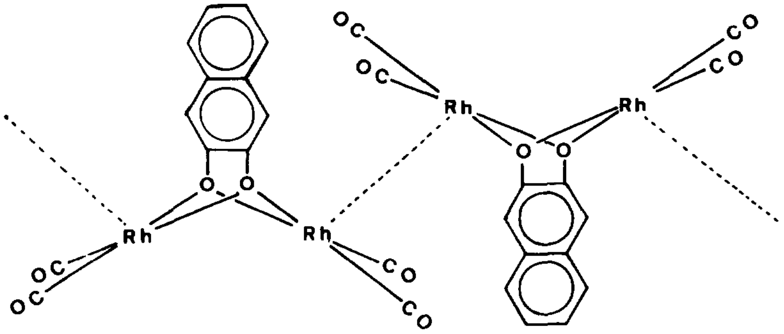
These dinuclear complexes show three ν(CO) absorptions in solution, consistent with C_{2v} or approximately C_{2v} symmetry (Table 1). The complex with IIIb separates spontaneously as a gold-green material. The presence of a rhodium–rhodium interaction in the solid state is strongly suggested by the dark colour, the metallic lustre, and the more complex IR spectrum in Nujol mull. Extended metal–metal chain interactions have been reported for several $[M(\mu\text{-}Cl)L]_2$ complexes (e.g. M = Ir, L = PF_3 [15]; M = Rh, L = CO [16]) in which zig-zag chain structures are present. We suspect that a similar situation is probably present in $Rh_2(\mu\text{-}2,3\text{-}(O)_2naphth)(CO)_4$.

TABLE 3

ANALYTICAL AND PHYSICAL DATA FOR THE MONONUCLEAR COMPLEXES

Compound	Analyses (Found (calcd.) (%))			Colour	Λ_M [a] (ohm^{-1} cm^2 mol^{-1})	Yield (%)	Selected IR bands (cm^{-1})
	N	C	H				
[Rh(COD)(1,8-$(NH_2)_2$naphth)]ClO_4	5.59 (5.97)	46.35 (46.10)	4.73 (4.69)	yellow	111	79	3265–3130 [b] 1270 [c]
[Rh(COD)(2,3-$(OH)_2$naphth)]ClO_4	–	45.48 (45.90)	4.37 (4.25)	yellow	141	80	3400–3165 [d] 1285 [e]
[Rh$(CO)_2$(1,8-$(NH_2)_2$naphth)]ClO_4	6.57 (6.72)	34.12 (34.57)	2.26 (2.40)	yellow	117	72	3300–3160 [b] 2085, 2020 [f]
NBu_4[Rh(2,3-$(O)_2$naphth)(COD)]	2.19 (2.29)	66.45 (66.77)	9.26 (8.83)	yellow	98	87	1275 [e] 2020, 1975 [f]

[a] Ca. 2×10^{-3} *M* acetone or nitromethane solutions. [b] ν(N–H). [c] ν(C–N). [d] ν(O–H). [e] ν(C–O) (Nujol mulls). [f] ν(C≡O) (dichloromethane solution).



Furthermore the $Rh_2(\mu\text{-}1,8\text{-}(NH)_2naphth)(CO)_4$ complex reacts with P-donor ligands (Rh/P-donor = 1/1) with formation of *trans*-$(R_3P)(CO)Rh(\mu\text{-}1,8\text{-}(NH)_2naphth)Rh(CO)(PR_3)$ (R = Ph, OEt, *o*-tolyl) as suggested by the presence of only one $\nu(CO)$ band in these complexes. Finally, we have recently reported the ability of $(PPh_3)(CO)Rh(\mu\text{-}1,8\text{-}(NH)_2naphth)Rh(CO)(PPh_3)$ to react with molecular iodine. The X-ray structure of the resulting complex shows that a two centre oxidative addition with concurrent metal–metal bond formation has occurred [7]. Further work in this area is in progress.

Experimental

C, H and N analyses were carried out with a Perkin–Elmer 240-B microanalyzer. IR spectra were recorded on a Perkin–Elmer 599 spectrophotometer over the range 4000–200 cm^{-1}, using Nujol mulls between polyethylene sheets or dichloromethane solutions between sodium chloride windows and calibrated with polystyrene. Conductivities were measured at 20°C with ca. 4×10^{-4} *M* acetone solution using a Philips PW 9509 conductimeter. Molecular weights were measured in ca. 10^{-3} *M* chloroform solutions with a Perkin–Elmer 115 osmometer. ^1H NMR spectra were recorded in $CDCl_3$ solutions at room temperature on a Varian XL200 spectrometer.

Preparation of $M_2(\mu\text{-}L)(COD)_2$ complexes

The $M_2(\mu\text{-}L)(COD)_2$ (M = Rh, Ir; L = 1,8-$(NH)_2$naphth, 1,8-$(O)_2$naphth; M = Rh, L = 2,3-$(NH)_2$naphth, 2,3-$(O)_2$naphth) compounds were prepared by the addition of $[Rh(\mu\text{-}OMe)(COD)]_2$ to a solution of LH_2 in diethyl ether.

In a typical procedure, $[Rh(\mu\text{-}OMe)(COD)]_2$ (100 mg, 0.2 mmol) was added to a solution of 1,8-$(NH_2)_2$naphth (33 mg, 0.2 mmol) in degassed diethyl ether. The solution immediately changed colour and an orange precipitate was formed. The suspension was stirred for 30 min, then the solid was filtered off, washed with diethyl ether, and air dried; yield 83 mg (70%).

Preparation of $RhIr(\mu\text{-}1,8\text{-}(O)_2naphth)(COD)_2$

$[Rh(\mu\text{-}OMe)(COD)]_2$ (50 mg, 0.103 mmol) and $[Ir(\mu\text{-}OMe)(COD)]_2$ (68.4 mg, 0.103 mmol) were added to a solution of 1,8-$(OH)_2$naphth (33 mg, 0.206 mmol) in degassed dichloromethane. The solution immediately turned orange, which was allowed to stand for 30 min. After concentration of the solution, addition of diethyl ether gave yellow crystals which were filtered off, washed with diethyl ether and air-dried. Yield 82 mg (60%).

Preparation of RhIr(μ-1,8-(NH)$_2$naphth)(COD)$_2$

1,8-(NH$_2$)$_2$naphth (12 mg, 0.075 mmol) was added to a solution of RhIr(μ-1,8-(O)$_2$naphth)(COD)$_2$ (50 mg, 0.075 mmol) in degassed dichloromethane. The solution immediately turned deep red and was allowed to stand for 20 min. The solution was evaporated to dryness and the deep red solid formed was collected by addition of diethyl ether, filtered off, washed with diethyl ether and air-dried. Yield 41 mg (82%).

Preparation of [Rh(COD)(LH$_2$)]ClO$_4$ complexes

The complexes [Rh(COD)(LH$_2$)]ClO$_4$ (LH$_2$ = 1,8-(NH$_2$)$_2$naphth, 2,3-(OH)$_2$naphth) were prepared by similar methods. As a typical example, a mixture of [Rh(μ-Cl)(COD)]$_2$ (100 mg, 0.202 mmol) and AgClO$_4$ (84 mg, 0.404 mmol) in acetone was stirred for 30 min. The AgCl was filtered off and 1,8-(NH$_2$)$_2$naphth (64 mg, 0.405 mmol) was added to the filtrate. The yellow solution obtained was stirred for 10 min. After concentration of the solution, the addition of diethyl ether gave a yellow precipitate, which was filtered off, washed with diethyl ether, and air-dried. Yield 150 mg (80%).

Preparation of NBu$_4$[Rh(2,3-(O)$_2$naphth)(COD)]

[Rh(μ-Cl)(COD)]$_2$ (200 mg, 0.405 mmol) and 2,3-(OH)$_2$naphth (130 mg, 0.810 mmol) were added to a solution of KOH in methanol. The mixture was stirred for 15 min and NBu$_4$Cl (225 mg, 0.810 mmol) was added. The volume of the solution was reduced and water was slowly added to produced a yellow precipitate, which was filtered off, washed with water and diethyl-ether, and air-dried. Yield 430 mg (87%).

Preparation of [Rh(CO)$_2$(1,8-(NH$_2$)$_2$naphth)]ClO$_4$

Carbon monoxide was bubbled through a solution of [Rh(COD)(1,8-(NH$_2$)$_2$naphth)]ClO$_4$ (50 mg, 0.106 mmol) in dichloromethane for 30 min. The yellow solution was concentrated and diethyl ether was added to yield a yellow solid, which was filtered off, washed with diethyl ether, and air-dried. Yield 32 mg (72%).

Preparation of Rh$_2$(μ-2,3-(O)$_2$naphth)(CO)$_4$

Carbon monoxide was bubbled through a solution of Rh$_2$(μ-2,3-(O)$_2$naphth)(COD)$_2$ (50 mg, 0.086 mmol) in dichloromethane for 40 min. The gold-green crystals formed in the reaction were filtered off, washed with dichloromethane, and air dried. Yield 30 mg (73%).

Preparation of Rh$_2$(μ-1,8-(NH)$_2$naphth)(CO)$_4$

Carbon monoxide was bubbled through a solution of Rh$_2$(μ-1,8-(NH)$_2$naphth)(COD)$_2$ (50 mg, 0.086 mmol) in dichloromethane for 20 min. The solution turned yellow. After concentration of the solution, addition of hexane gave yellow crystals, which were filtered off, washed with hexane, and air-dried. Yield 31 mg (75%).

Preparation of Rh$_2$(μ-1,8-(NH)$_2$naphth)(CO)$_2$(PR$_3$)$_2$ complexes

The complexes Rh$_2$(μ-1,8-(NH)$_2$naphth)(CO)$_2$(PR$_3$)$_2$ (R = Ph, OEt, *o*-tolyl) were prepared by similar methods. As a typical example, PPh$_3$ (272 mg, 1.037 mmol) was added to a solution of Rh$_2$(μ-1,8-(NH)$_2$naphth)(CO)$_4$ prepared "in situ" from

$Rh_2(\mu\text{-}1,8\text{-}(NH)_2naphth)(COD)_2$ (300 mg, 0.518 mmol) by the above method. The orange solution was stirred for 30 min, then concentrated, and addition of n-pentane gave a yellow precipitate, which was filtered off, washed with n-pentane, and air-dried. Yield 390 mg (80%).

Acknowledgement

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