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BIDENTATE AMINES AS BRIDGES BETWEEN $M(CO)_2Cl$ (M = Rh OR Ir) UNITS

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Summary

The preparation and characterization by elemental analysis, electronic and infrared spectroscopy are reported for the monomeric complexes *cis*-(amine)-$M(CO)_2Cl$ (M = Ir or Rh, amine = 1,8-naphthyridine or pyridazine; M = Ir, amine = *o*-phenylenediamine) and the binuclear species (1,8-naphthyridine)-$Rh_2(CO)_4Cl_2$, (1,8-naphthyridine)$IrRh(CO)_4Cl_2$, (pyrazine)$Rh_2(CO)_4Cl_2$ and (1,3-di-4-pyridylpropane)$Rh_2(CO)_4Cl_2$.

Introduction

Recently the ability of a number of bifunctional ligands to act as bridges connecting two metal atoms has received considerable interest. Diphosphines with three to ten methylene groups separating the phosphorus atoms have been shown to form macrocyclic complexes (e.g. $[R_2P(CH_2)_nPR_2Rh(CO)Cl]_2$, R = t-Bu or Ph) with relatively large separations between the metal atoms [1—4]. In contrast, the diphosphine bis(diphenylphosphino)methane, which also forms complexes with two metals, can constrain the distance between the metals to fairly short distances and allow for some unusual reaction patterns (transannular oxidative addition [5], insertion into metal—metal bonds [6]). Carboxylates [7,8] and a number of other ligands including diisocyanides [9], triazenes [10] and bis(methylide) [11] are recognized for their abilities to form binuclear complexes.

This article describes a number of rhodium(I) and iridium(I) complexes involving the $M(CO)_2Cl$ unit with a single, potentially bridging, amine ligand. Other nitrogen donor ligands which have been found to bridge two rhodium centers include biimidazole [12,13], diimines [14], porphyrins [15], and other macrocycles [16].

Experimental

Documented procedures were used for the preparation of $Rh_2(CO)_4Cl_2$ [17], $(p\text{-}CH_3C_6H_4NH_2)Ir(CO)_2Cl$ [18], (pyridine)$Rh(CO)_2Cl$ [19,20], and 1,8-naphthyridine [21]. 1,3-Di-4-pyridylpropane from Reilly Tar and Chemical Corporation was recrystallized from diethyl ether before use. Other amines were commercial samples of highest purity and were used as received. All of the new complexes reported are sensitive to air both in solution and as solids and preparations should be done in oxygen free solvents under a nitrogen atmosphere.

The following three compounds were prepared in >80% yield by mixing solutions of $Rh_2(CO)_4Cl_2$ and the appropriate ligand in a 1/2 mole ratio in the solvent indicated at 25°C. If the product did not crystallize after mixing and cooling to 0°C, crystallization was induced by the addition of pentane.

(Ethylamine)Rh(CO)$_2$Cl

Green needles from pentane; m.p., 145—148°C (dec.) (Found: C, 20.05; H, 3.33. $C_4H_7ClNO_2Rh$ calcd.: C, 20.06, H, 2.95%).

(1,8-Naphthyridine)Rh(CO)$_2$Cl

Yellow needles from benzene/pentane; m.p., gradually decomposes above 100°C (Found: C, 36.84; H, 2.12, Cl, 10.95; N, 8.65. $C_{10}H_6ClN_2O_2Rh$: C, 37.01; H, 1.86; Cl, 10.92; N, 8.63%).

(Pyridazine)Rh(CO)$_2$)Cl

Greenish-bronze needles from benzene/pentane. m.p., decomposes gradually above 100°C (Found: C, 26,31; H, 1.63; N, 10.22. $C_6H_4ClN_2O_2Rh$ calcd.: C, 26.26; H, 1.47; N, 10.21%).

The following two compounds were prepared in >80% yield by mixing solutions of $(p\text{-}CH_3C_6H_4NH_2)Ir(CO)_2Cl$ and the appropriate ligand in ether in a 1/1 mole ratio. The products crystallized after cooling or evaporation under a stream of oxygen free nitrogen.

(1.8-Naphthyridine)Ir(CO)$_2$Cl

Red needles; m.p., 137—140°C (dec.) (Found: C, 29.58, H, 1.50; Cl, 8.33; N, 6.80. $C_{10}H_6ClIrN_2O_2$ calcd.: C, 29.02; H, 1.46, Cl, 8.57; N, 6.77%).

(Pyridazine)Ir(CO)$_2$Cl

Brown needles from diethyl ether; m.p., 139—140°C (Found: C, 19.56; H, 1.13; N, 7.53. $C_6H_4ClIrN_2O_2$ calcd.: C, 19.81; H, 1.11; N, 7.70%).

(Pyrazine)Rh$_2$(CO)$_4$Cl$_2$

A filtered solution of 86.4 mg (0.222 mol) of $Rh_2(CO)_4Cl_2$ in 10 ml of dichloromethane was added dropwise to a solution of 19.1 mg (0.239 mmol) of pyrazine in 5 ml of dichloromethane. The red microcrystalline solid which formed was collected by filtration, washed with dichloromethane and vacuum dried (yield: 77 mg, 74%); m.p., 129—131°C turns yellow but remains solid up to 230°C (Found: C, 19.57, H, 0.87; N, 6.16. $C_8H_4Cl_2N_2O_4Rh_2$ calcd.: C, 20.49; H, 0.86; N, 5.97%).

(1,8-Naphthyridine)$Rh_2(CO)_4Cl_2$

A solution of 66 mg (0.51 mmol) of 1,8-naphthyridine in 7 ml of benzene was added dropwise to a solution of 200 mg (0.51 mmol) of $Rh_2(CO)_4Cl_2$ in 7 ml of benzene. Fine green needles of the product formed rapidly. These were collected by filtration and washed with benzene. For purification the crystals were dissolved in a minimum volume of dichloromethane and after filtering the solution 5 ml of cyclohexane was added. The solution was evaporated under nitrogen to yield fine green needles (yield: 240 mg, 91%); m.p., 145—155°C, turns brown, 185—195°C dec. (Found: C, 27.40; H, 1.34; Cl, 14.19; N, 5.46. $C_{12}H_6Cl_2N_2O_4Rh_2$ calcd.: C, 27.78; H, 1.17; Cl, 13.66; N, 5.40%).

(1,8-Naphthyridine)$RhIr(CO)_4Cl_2$

A solution of 20 mg (0.10 mmol) of $Rh_2(CO)_4Cl_2$ in 5 ml of benzene was added dropwise to a solution of 46 mg (0.11 mmol) of (1,8-naphthyridine)$Ir(CO)_2Cl$ in 10 ml of benzene. A deep blue, crystalline precipitate formed rapidly. This was collected by filtration, washed with benzene and vacuum dried (yield: 56 mg, 88%); m.p., dec. above 190°C (Found: C, 23.80; H, 0.61; N, 4.46. $C_{12}H_6Cl_2IrN_2O_4Rh$ calcd.: C, 23.70; H, 0.99; N, 4.61%).

(o-Phenylenediamine)$Ir(CO)_2Cl$

A solution of 5.8 mg (0.54 mmol) of *o*-phenylenediamine in 3 ml of methanol was added to a solution of 308 mg (0.44 mmol) of $[Ph_4As][Ir(CO)_2Cl_2]$ in 5 ml of methanol. The solution was heated briefly under reflux. The orange microcrystalline precipitate which formed was collected by filtration, washed with methanol and vacuum dried. The complex has only slight solubility in methanol or dimethyl sulfoxide in which it rapidly turns blue; it is virtually insoluble in dichloromethane, acetone or nitromethane; m.p., dec. above 190°C (Found: C, 24.27; H, 1.96; Cl, 9.09; N, 7.56. $C_8H_8ClIrN_2O_2$ calcd.: C, 24.52; H, 2.06; Cl, 9.09; N, 7.56%).

(1,3-Di-4-pyridylpropane)$Rh_2(CO)_4Cl_2$

A filtered solution of 53 mg (0.27 mmol) of 1,3-di-4-pyridylpropane in 5 ml of benzene was added dropwise to a filtered solution of 100 mg (0.26 mmol) of $Rh_2(CO)_4Cl_2$ in 5 ml of benzene. Fine yellow needles immediately precipitated from solution. These were collected by filtration and washed with benzene and vacuum dried; m.p., 100—120°C (dec.) (Found: C, 34.50; H, 2.50. $C_{17}H_{14}Cl_2N_2O_4Rh_2$ calcd.: C, 34.78; H, 2.40%).

Infrared spectra were recorded on a Beckman IR-12 Spectrometer. Electronic spectra were obtained using a Cary 17 spectrophotometer. Conductivities were measured in nitromethane solution using an Industrial Instruments conductivity bridge. Proton magnetic resonance spectra were obtained using a JEOLCO PS-100 Digilab FTS operating at 100 MHz.

Results and discussion

Complex of the type *cis*-(amine)$Rh(CO)_2Cl$ have been prepared previously by the bridge splitting reactions of a variety of amines (including ammonia, methylamine, pyridine, and *p*-toluidine) with $Rh_2(CO)_4Cl_2$ [20]. Analogous iridium

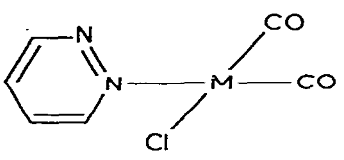
TABLE 1

SELECTED INFRARED SPECTRAL BANDS

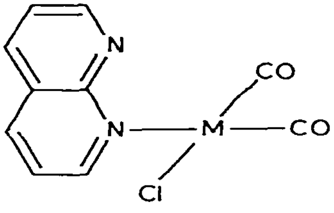
Compound	ν(CO) (cm^{-1})		ν(M—Cl) Solid (cm^{-1})
	Solid	Solution (CH_2Cl_2)	
(Ethylamine)$Rh(CO)_2Cl$	2083, 2057 2022, 1979	2084, 2005	309
(*p*-Toluidine)$Rh(CO)_2Cl$	2089, 2027	2086, 2011	300
(Pyridine)$Rh(CO)_2Cl$	2070, 2013		307
(Pyridazine)$Rh(CO)_2Cl$	2080, 2071	2087, 2016	305
(1,8-Naphthyridine)$Rh(CO)_2Cl$	2073, 2011	2080, 2007	303
(Pyrazine)$Rh_2(CO)_2Cl_2$	2106, 2075(sh) 2010, 1985(sh)		309
(1,8-Naphthyridine)$Rh_2(CO)_4Cl_2$	2107, 2070, 2034	2090, 2076, 2032(sh) 2019	300
(1,3-Di-4-pyridylpropane)$Rh_2(CO)_4Cl_2$	2091, 1997		318
(1,8-Naphthyridine)$IrRh(CO)_4Cl_2$	2101, 2059, 2031(sh) 2009(sh), 1995		310, 300
(Ethylamine)$Ir(CO)_2Cl$	2077, 2042 2012, 1966	2074, 1992	310
(*p*-Toluidine)$Ir(CO)_2Cl$	2085, 2047 2022, 1975	2075, 1996	305
(*o*-Phenylenediamine)$Ir(CO)_2Cl$	2073, 2014		312
(Pyridazine)$Ir(CO)_2Cl$	2066, 2049 1995, 1884	2077, 2000	306
(1,8-Naphthyridine)$Ir(CO)_2Cl$	2068, 2005	2072, 1993	312

complexes have been prepared from the reaction of $Ir(CO)_3Cl$ with amines or by amine exchange from *cis*-(*p*-toluidine)$Ir(CO)_2Cl$ [18]. In the present work the reactions of $Rh_2(CO)_4Cl_2$ or *cis*-(*p*-toluidine)$Ir(CO)_2Cl$ with a group of bifunctional amines have produced both mononuclear compounds, *cis*-(amine)-$M(CO)_2Cl$, and binuclear compounds (amine)$M_2(CO)_4Cl_2$.

Pyradizine and 1,8-naphthyridine form complexes *cis*-(pyradizine)$M(CO)_2Cl$, (I) and *cis*-(1,8-naphthyridine)$M(CO)_2Cl$ (II) with both rhodium and iridium.



(I)



(II)

These compounds have good solubility in dichloromethane, chloroform, acetone and nitromethane. In the latter solvent they are non-electrolytes. Selected infrared spectral features for these and other new complexes are reported in Table 1. Table 2 displays the electronic spectra of the new complexes. The

TABLE 2

ELECTRONIC SPECTRA

Compound	λ_{max}, (nm) (ϵ), dichloromethane solution
(1,8-Naphthyridine)$Rh(CO)_2Cl$	335(2500)(sh), 311(10200) 299(11600), 277(12400)
(Pyridazine)$Rh(CO)_2Cl$	355(2200), 268(8400)
(Pyridine)$Rh(CO)_2Cl$	341(2900), 259(9400)
(*p*-Toluidine)$Rh(CO)_2Cl$	337(3600), 270(sh) (7900), 253(8500)
(1,8-Naphthyridine)$Rh_2(CO)_4Cl_2$	422(1900), 311(13600), 303(13600) 265(14900)
(1,8-Naphthyridine)$IrRh(CO)_4Cl_2$	465(3000), 313(12000), 301(12000) 265(14000)
(1,3-Di-4-pyridylpropane)$Rh_2(CO)_4Cl_2$	343(5000) [a]
(Ethylamine)$Ir(CO)_2Cl$	393(442), 356(2450), 311(1820)
(1,8-Naphthyridine)$Ir(CO)_2Cl_2$	390(sh)(1000) 352(2400), 312(10800) 305(10800), 270(10500)
(Pyridazine)$Ir(CO)_2Cl$	420(sh) (700), 375(1800), 330(2800) 267(8300)
(*p*-Toluidine)$Ir(CO)_2Cl_2$	515(105), 440(—), [b] 392(700), 358(3100), 311(2700)

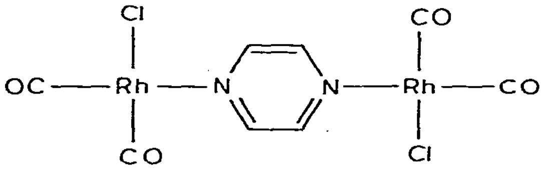
[a] Acetone solution. [b] Concentration dependent.

infrared spectra of solids I and II show distinct M—Cl stretching vibrations which confirm the presence of covalent M—Cl bonds that were suggested by the conductivity measurements. In solution each complex displays two carbonyl stretching frequencies so that the two carbonyl groups must be *cis*. The electronic spectra of these complexes show low-energy transitions which are characteristic of other *cis*-(amine)$M(CO)_2Cl$ complexes.

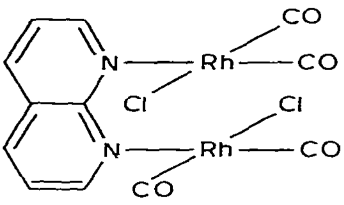
Because of the strong similarity of physical characteristics of I and II with other *cis*-(amine)$M(CO)_2Cl$ complexes these species are formulated as planar, four-coordinate complexes with one of the nitrogen atoms of each aromatic base not coordinated. Related structures have been proposed for complexes of the type *cis*-(amine)$Pt(PEt_3)_2Cl^+$ [22] and such a structure has been determined crystallographically for *cis*-(1,8-naphthyridine)$Pt(PEt_3)_2Cl^+$. In this complex the coordinated Pt—N distance is reported to be 2.08 Å while the Pt—N distance involving the uncoordinated out-of-plane nitrogen is 3.05 Å [22]. Similarly the gold complexes (1,8-naphthyridine)$Au(CH_3)_2X$ contain 1,8-naphthyridine coordinated to gold through a single nitrogen [22]. Both *cis*-(1,8-naphthyridine)Pt-$(PEt_3)_2Cl^+$ and (1,8-naphthyridine)$Au(CH_3)_2X$ are fluxional and I also shows signs of fluxional behavior. At room temperature in chloroform-*d* I (M = Ir) displays a proton magnetic resonance spectrum very much like that of free 1,8-naphthyridine [24]. The spectrum may be analysed as containing two equivalent rings and three chemically distinct proton resonances. The chemical shifts

(τ) are α, 0.65; β, 2.26; and γ, 1.52 ppm while the coupling constants are $\alpha\beta$, 4.2; $\alpha\gamma$, 1.3; and $\beta\gamma$, 8.3 Hz. On cooling to —65° C the resonance due to the β protons has broadened considerably and appears to be splitting apart in a fashion similar to that observed for *cis*-(1,8-naphthyridine)$Pt(PEt_3)_2Cl^+$ [22]. Complete resolution of this resonance into its low temperature limit was not possible within the temperature range available. The alternate possibility that *cis*-(1,8-naphthyridine)$Ir(CO)_2Cl$ possesses a static structure with a symmetrically bound chelating 1,8-naphthyridine ligand would require the complex to be square pyramidal with an axial chlorine. Such a structure appears unlikely for a complex which closely resembles *cis*-$(EtNH_2)Ir(CO)_2Cl$ in other physical properties.

Pyrazine and 1,8-naphthyridine react with $Rh_2(CO)_4Cl_2$ in a 1/1 molar ratio to produce the binuclear complexes III and IV respectively. The pyrazine complex III is isolated as fine metallic-red crystals which have very low solubility in noncoordinating solvents. The naphthyridine complex IV is soluble in acetone, dichloromethane and nitromethane (in which it is a non-electrolyte). In solution IV is gradually converted into a red microcrystalline substance whose infrared spectrum indicates the presence of 1,8-naphthyridine and bridging carbon monoxide (ν(CO), 1840, 1780 cm^{-1}). No solvent for this material has been found. Its formation from IV is hastened by heating and so recrystallization of IV is not advised. The two rhodium 1,8-naphthyridine complexes are readily interconverted via eq. 1 and 2. Similar reactions appear to occur with pyrazine except that the low solubility of III precludes the isolation of a monometallic species. Evidence of the formation of *cis*-(pyrazine)$Rh(CO)_2Cl$ comes from the fact that III will dissolve in benzene or dichloromethane containing an excess of pyrazine to give yellow solutions with ν(CO), 2090, 2016 cm^{-1}. These solutions produce only compound III, however, when they are concentrated and pentane is added.



(III)



(IV)

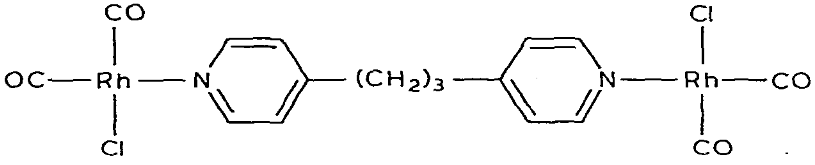
(1,8-naphthyridine)$Rh_2(CO)_4Cl_2$ + 1,8-naphthyridine →

cis-(1,8-naphthyridine)-$Rh(CO)_2Cl$ (1)

$Rh_2(CO)_4Cl_2$ + *cis*-(1,8-naphthyridine)$Rh(CO)_2Cl$ →

1,8-naphthyridine)-$Rh_2(CO)_4Cl_2$ (2)

1,3-Di-4-pyridylpropane reacts with $Rh_2(CO)_4Cl_2$ to yield the yellow binuclear complex V.

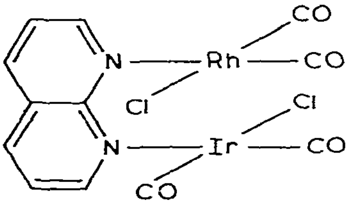


(V)

Although complexes are known in which pyridazine bridges two metal atoms [25—27], no bimetallic complex of the type (pyridazine)$M_2(CO)_4Cl_2$ has been detected. No binuclear iridium complexes have been found using these three heterocyclic bases. *o*-Phenylenediamine, which forms binuclear (*o*-phenylenediamine)$Rh_2(CO)_4Cl_2$ [28] when treated with $Rh_2(CO)_4Cl_2$, produces only mononuclear *cis*-(*o*-phenylenediamine)$Ir(CO)_2Cl$ from either *cis*-(*p*-toluidine)-$Ir(CO)_2Cl$ or *cis*-$Ir(CO)_2Cl_2^-$.

For the binuclear complexes III, IV and V the infrared spectra clearly indicate that the carbonyl group occupy *cis* coordination sites; however, the relative orientation of the chloride on the two metal centers cannot be determined from the available spectroscopic data.

One mixed metal compound has been obtained. Since reaction 2 readily forms the dirhodium species IV, an analogous reaction between $Rh_2(CO)_4Cl_2$ and I (M = Ir) was investigated. This reaction readily forms deep blue crystals of VI.



(VI)

The colors of the *cis*-(amine)$M(CO)_2Cl$ complexes as solids and their infrared spectra in the region of $\nu(CO)$ are correlated. Those complexes which form yellow solids and give yellow solutions show two carbonyl stretching modes in both the solid state and in solution. In these cases the solids must contain isolated monomeric metal complexes. On the other hand, those complexes which have metallic green, brown or blue colors as solids, (ethylamine)$Rh(CO)_2Cl$, (pyridazine)$Rh(CO)_2Cl$, (pyrazine)$Rh(CO)_2Cl$, (ethylamine)$Ir(CO)_2Cl$, (pyridazine)$Ir(CO)_2Cl$, and (*p*-toluidine)$Ir(CO)_2Cl$ have more complicated infrared spectra in the carbonyl stretching region although they also dissolve to give solutions with only two terminal carbonyl absorptions. In these cases the solids are suspected, as others have suggested [19,29], to contain metal—metal bonded aggregates of the monomeric units. In the case of IV, which is a model for a simple two unit aggregate, the ligand bridged complex retains its more complex infrared spectrum after it dissolves. Although examination of space filling models indicates that the two rhodium ions in V could approach one-another within 3.5 Å if the two phenyl rings were stacked directly over each other, neither the infrared nor the electronic spectra of this complex indicates that the rhodium ions closely approach each other.

The electronic spectra of ligand bridged, planar rhodium(I) complexes have been shown to be sensitive to the separation between the metal centers and the relative orientation of the coordination planes [4,5]. For complexes in the nearly paralleled orientation of these planes appreciable overlap of the out-of-plane orbitals, particularly the metal dz^2 orbitals and ligand orbitals, occurs when the metal centers are less than 3.5 Å apart. This overlap results in a shifting of the lowest energy metal-to-ligand ($dz^2 \rightarrow \pi^*$) charge transfer transition to lower energies as described previously [4,5]. For *cis*-(amine)Rh(CO)$_2$Cl the absorptions in the range 337—355 nm with extinction coefficients between 2200—3600 are assigned as the spin allowed $dz^2 \rightarrow \pi^*$, metal-to-ligand transitions that are characteristic of a number of rhodium(I) complexes containing π-acceptor ligands [30]. The spectra of the two 1,8-naphthyridine complexes II and IV are compared in Fig. 1. The strong absorptions seen in the 311—200 nm region are characteristic of the aromatic heterocycle itself. The shoulder seen at 335 nm in II is assigned as the metal-to-ligand charge transfer while in IV the band at 422 is believed to be a similar transition shifted to lower-energy by the close proximity of the two rhodium centers. The mixed metal complex

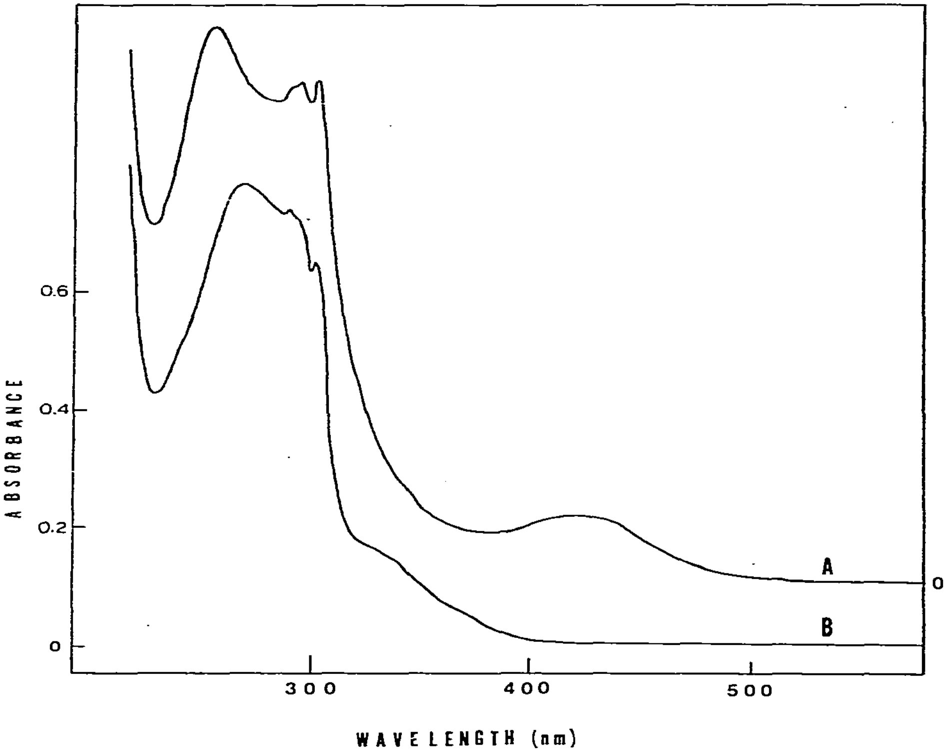


Fig. 1. Electronic spectra of (A) 0.63 mM (C$_8$H$_6$N$_2$)Rh$_2$(CO)$_4$Cl$_2$ and (B) 0.63 mM (C$_8$H$_6$N$_2$)Rh(CO)$_2$Cl in dichloromethane solution with cells of 1.0 mm path length.

V also shows a related low energy feature which is absent in II (M = Ir or Rh). For the iridium complexes the absorption bands in the range 390—420 nm with extinction coefficients of 400—1000 are assigned as spin forbidden $dz^2 \rightarrow$ ligand $p\pi^*$ transitions while the bands at 350—375 nm with extinction coefficients in the range 1800—3100 are assigned as spin allowed $dz^2 \rightarrow$ ligand $p\pi^*$ transition. With one exception the spectra of the *cis*-(amine)M(CO)$_2$Cl species obey Beer's law in solution thereby indicating that no significant self association is occurring. However, *cis*-(*p*-toluidine)Ir(CO)$_2$Cl does show a concentration dependent absorption at 440 nm. This band increases in intensity as the concentration of complex is increased. The effect is solvent dependent. It is most noticeable in benzene solution and is also observed in dichloromethane although the 440 absorption is absent in acetone solutions. This concentration dependent absorption may be due to the existence of metal—metal bonded aggregates in solution. However, the magnitude of the effect and the concentration range available for the complex do not make a quantitative study of this effect appear feasible. Since it was the observation of this departure from Beer's law which led to the attempt to form amine ligand bridged complexes containing Ir(CO)$_2$Cl and Rh(CO)$_2$Cl units, it is disappointing that no ligand bridged diiridium complexes could be formed.

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