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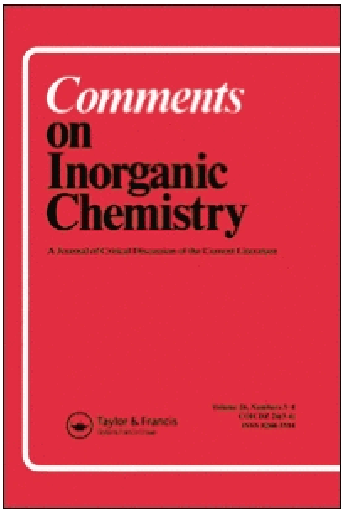

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Spectroscopic and Photochemical Properties of Olefin Complexes

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Spectroscopic and Photochemical Properties of Olefin Complexes

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Olefin complexes are characterized by a diversity of electronic transitions which involve a direct participation of the olefin ligand. Intraligand as well as charge transfer (LMCT, MLCT and LLCT) absorptions are observed in the electronic spectra of olefin complexes. Free olefins are also engaged in outer sphere CT interactions with metal complexes. The photochemistry of olefin complexes can be related to the electronic structure of the reactive excited states.

Keywords: *Olefin complexes, Charge transfer, Photochemistry*

1. INTRODUCTION

Olefin complexes play key role in organic and organometallic synthesis. Stoichiometric as well as catalytic transformations of olefins assisted by coordination compounds have found broad applications. Many of these reactions take also place as light-induced processes [1]. In contrast to the importance of this subject relatively little attention has been paid to

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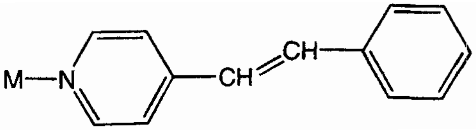
the nature of the reactive excited states. Only in a few cases electronic spectra of olefin complexes have been studied in some detail [1]. Since olefins have ambivalent properties a diversity of electronic transitions is feasible. We explored these possibilities during recent years and give here a first account of our observations on the spectroscopic and photochemical properties of selected olefin complexes. Some older studies are also discussed in this report in order to illustrate the considerable diversity of excited states and their photoreactivity.

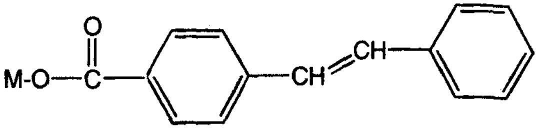
2. TYPES OF OLEFIN COMPLEXES AND ELECTRONIC TRANSITIONS

Olefins are characterized by ππ* transitions which are preserved but modified in the coordinated state. These intraligand (IL) excitations determine frequently the spectroscopy and photochemistry of coordinated olefins. Generally, olefin complexes are organometallic compounds in which the π and π* orbitals of the olefin are engaged in bonding interactions with the metal. Usually, π-back bonding is important. The metal occurs then in low oxidation states and is thus reducing. Accordingly, the electronic spectra should display absorptions which are attributed to metal-to-ligand charge transfer (MLCT) transitions terminating at π* orbitals of the olefin. If the metal is also oxidizing (e.g. Ag^I, Pd^{II}) long-wavelength ligand-to-metal charge transfer (LMCT) absorptions may be present, too. In mixed-ligand complexes which contain a strong donor ligand such as a carbanion (e.g. CH_3^-) in addition to an olefin it is also feasible that an electron is promoted from the donor ligand to the π* orbitals of the olefin. Such ligand-to-ligand charge transfer (LLCT) transitions are well known [2,3] but have not yet been observed for olefin complexes.

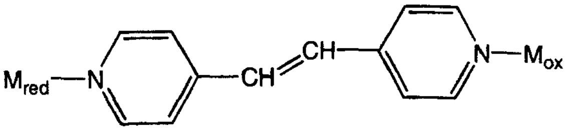
Olefins may be also incorporated in ligands which are not coordinated via the olefin carbon atoms. Stilbazol complexes are prototype compounds.



Of course, metals are not required to exist in low oxidations states since π back bonding is not necessary in this case. Nevertheless, stilbazole can still act as donor or acceptor for CT transitions. If the coordination takes place via functional groups which prevent a strong electronic coupling of olefin and metal the olefin appears as an isolated chromophore of the complex, e.g. in carboxylates:



In binuclear complexes a reducing and an oxidizing metal center may be electronically coupled by a bridging ligand. Olefins are well suited to mediate this interaction [4]:



As a result a metal-to-metal charge transfer (MMCT) transition may occur. Metal complexes with a partially filled d-shell have available ligand field (LF) excited states which are affected by all ligands including olefins. However, since olefins are not directly involved in MMCT and LF transitions they are omitted in the further discussion.

An olefin can undergo a CT interaction with a metal even if it is not bonded to the complex. An intimate contact may provide a sufficient electronic coupling for the observation of an outer sphere (OS) CT transition [5,6].

In summary, only these electronic transitions which involve a direct participation of the olefin are included in the following discussion:

IL
LMCT
MLCT
LLCT
OSCT

Our report deals with the spectroscopy and photochemistry of selected examples. For a comprehensive review of the photochemistry of olefin complexes until 1979 the reader is refered to Geoffroy's and Wrighton's book on Organometallic Photochemistry [1].

3. ELECTRONIC SPECTRA

3.1. IL Transitions

The ππ* absorptions of simple olefins appear at rather short wavelength (~ 200 nm). Coordinated olefins show their ππ* bands in the same spectral region. These IL absorptions are then frequently obscured by bands of different origin such as CT bands. However, the IL absorptions undergo a considerable red shift if the olefin carries suitable substituents. For example, stilbene and its derivatives including stilbazole (styrylpyridine) and stilbene-carboxylate display their ππ* bands in the near UV. As IL absorptions they appear well separated from other bands and are easily identified:

trans – 4 – styrylpyridine $\lambda_{max} = 308$ nm [7]

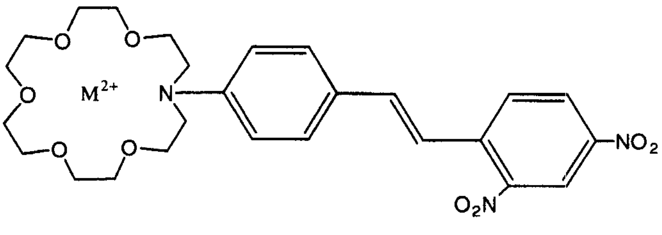
$Re^{I}(CO)_3Cl$(trans – 4 – styrylpyridine$)_2$ $\lambda_{max} = 328$ nm [7]

$Re^{VII}(CH)_3O_3$(trans–4–styrylpyridine) $\lambda_{max} = 342$ nm (Fig. 1) [8]

trans – 4 – stilbenecarboxylate $\lambda_{max} = 319$ nm [9, 10]

$[Co^{III}(NH_3)_5$(trans–4–stilbenecarboxylate$)]^{3+}$ $\lambda_{max} = 320$ nm [9, 10]

In certain cases the coordination has a more severe effect and changes the type of IL transition:



$M^{2+} = Ba^{2+}, Pb^{2+}$ $\lambda_{max} \sim 360$ nm [11]

The longest-wavelength absorption of the free ligand trans-2,4-dinitrostilbene-4′-monoaza-18-crown-6 at $\lambda_{max} = 476$ nm is attributed to a CT transition from the amine function to the nitro groups. On complexation the amine is blocked and the CT transition is replaced by a $\pi\pi^*$ transition of the stilbene moiety at higher energies.

3.2. LMCT Transitions

Since olefins are reducing owing to the presence of π electrons they are expected to serve as electron donors for CT transitions. In combination with oxidizing metals LMCT transitions should occur at relatively low energies. In this context it is of historical interest that Mulliken [12] and Orgel [13] suggested a long time ago that olefin complexes of Ag^+ are suitable candidates for the observation of olefin (π) to Ag^+ (5s) LMCT absorptions. Indeed, such a LMCT band was later identified:

$[Ag(cyclohexene)]^+$ $\lambda_{max} = 230$ nm [14]

Unfortunately, IL ($\pi\pi^*$) and LMCT absorptions of silver olefin complexes overlap strongly. This interference hampers further spectroscopic and photochemical studies.

Mercury(II) is oxidizing and isoelectronic (d^{10}) to Ag(I). The electronic spectra of Hg(II) complexes are characterized by LMCT absorptions [15]. Such CT bands appear also when olefins are added to Hg^{2+} in non-coordinating solvents, e.g.

$HgCl_2 \times cyclohexene$ $\lambda_{max} = 250$ nm [16]

Cu(I) is another d^{10} metal which forms olefin complexes. However, since Cu(I) is not only oxidizing but also reducing LMCT and MLCT transitions can occur in the same energy range. Definitive assignments have not yet been made [1,17].

While the previous examples involve d^{10} metal centers oxidizing transition metals with a partially filled d shell are also expected to show long-wavelength LMCT absorptions. Olefin complexes of Pd(II) are suitable examples, e.g.:

$Pd(1,5-cyclooctadiene)Cl_2$ $\lambda_{max} = 342$ nm (Fig. 2) [18]

In view of the large number of olefin complexes LMCT absorptions may be a general feature of their electronic spectra. However, their detection might be complicated by the presence of other bands of different origin. In particular, MLCT absorptions could appear in the same spectral region.

3.3. MLCT Transitions

Since olefin complexes are stabilized by π back bonding they require metals in relatively low oxidation states. Generally, such metals are reducing. Accordingly, MLCT transitions terminating at the olefin π* orbitals occur at relatively low energies, e.g.:

d^6 metals

cis-$[W^0(CO)_4(\text{trans-4-styrylpyridine})_2]$ $\lambda_{max} = 500$ nm [19]

$[Mo^0(CO)_4(\text{cycloheptatriene})]$ $\lambda_{max} = 486$ nm [20,21]

d^8 metals

$[Fe^0(CO)_4(\text{trans}-1,2-\text{dibromoethylene})]$ $\lambda_{max} = 288$ nm [1,22]

$[Fe^0(CO)_3(\text{duroquinone})]$ $\lambda_{max} = 350$ nm [23]

$[Rh^I(1,5-\text{cyclooctadiene})Cl]_2$ $\lambda_{max} = 380$ nm [24]

$[Ir^I(1,5-\text{cyclooctadiene})Cl]_2$ $\lambda_{max} = 455$ nm [24]

$[Pt^{II}Cl_3(C_2H_4)]^-$ $\lambda_{max} = 330$ nm [25]

In the complex [Fe(CO)4(duroquinone)] the quinone coordinates as a diolefin and not via its oxygen atoms.

d^{10} metals

$[Pt^0(PPh_3)_2(C_2H_4)]$ $\lambda_{max} = 285$ nm [26]

$[Pt^0(PPh_3)_2(TCNE)]$ $\lambda_{max} = 305$ nm [27]

$\{Pt^0[P(OPh)_3]_2C_{60}\}$ $\lambda_{max} = 770$ nm [28]

Although the MLCT absorption of $[Pt^0(PPh_3)_2(\text{olefin})]$ should be red-shifted if C_2H_4 is replaced by TCNE (tetracyanoethylene) the observed shift seems to be too small in view of the strong electron-accepting properties of TCNE. Fullerene is usually considered to be a three-dimensional aromatic molecule. However, since only one double bond of C_{60} is engaged in bonding to platinum we classify $\{Pt^0[P(OPh)_3]_2C_{60}\}$ as an olefin complex [28]. The very low energy of the MLCT absorption of the fullerene complex is in accord with the high electron affinity of C_{60}.

3.4. LLCT Transitions

Complexes of the type [olefin-M-carbanion] contain simultaneously an acceptor and a donor ligand. A CT interaction between both ligands mediated by the metal may lead to the appearance of a LLCT absorption. Recently, we have identified such $R^- \rightarrow$ olefin LLCT bands:

[Pt(1,5-cycloooctadiene)$(CH_3)_2$] $\lambda_{max} = 352$ and 318 nm(Fig.3) [29]

The longer wavelength absorption is assigned to a spin-forbidden transition.

3.5. OSCT Transitions

CT can also take place between species which are not connected by chemical bonds but are in close contact. A variety of metal complexes participate as donors and/or acceptors in such OSCT interactions [5,6]. Donor-acceptor pairs consisting of transition metal hydride complexes and olefins with electron-withdrawing substituents show such OSCT absorptions in the visible region [30]:

$[(C_5H_5)_2WH_2]$/maleic anhydride	$\lambda_{max} = 571$ nm
dimethyl maleate	$\lambda_{max} = 400$ nm
maleonitrile	$\lambda_{max} = 500$ nm

Although it was suggested that the d^2 metal W(IV) acts as donor it is also possible that these CT transitions originate from the hydride ligands.

4. PHOTOCHEMISTRY

Many photochemical studies of olefins in the presence of metal complexes have been carried out in order to utilize these photoreactions for applications such as hydrogenations, hydrosilations and valence isomerizations [1]. However, the photoreactive species and excited states were rarely identified. We describe here only complexes with reactive excited states which can be assigned with some confidence.

4.1. IL Excitation

Many olefins undergo trans/cis isomerization upon $\pi\pi^*$ excitation. This photoreaction may be simply explained by the fact that a rotation around the olefinic C-C bond occurs because the π-bond is not existent in the excited state. The same arguements should apply to coordinated olefins. Indeed, a variety of complexes undergo a trans/cis photoisomerization of the olefin ligand, e.g.

$$[\mathrm{Re^{I}(CO)_3Cl(trans-4-styrylpyridine)_2}]\ \phi=0.54 \text{ at } \lambda_{irr}=366 \text{ nm} \quad [7]$$

$$[\mathrm{Re^{VII}(CH_3)O_3(trans-4-styrylpyridine)}] \quad [8]$$

$$\left[\mathrm{Rh^{III}(NH_3)_5(trans-4-stilbenecarboxylate)}\right]^{2+}$$
$$\phi = 0.09 \text{ at } \lambda_{max} = 313 \text{ nm} \quad [31]$$

Generally, the photostationary trans/cis ratio and the quantum yields of the complexes are different from those of the free ligand.

As an alternative to isomerization the excited olefin may participate in electron transfer reactions, e.g.

$$[\mathrm{Co^{III}(NH_3)_5(trans-4-stilbenecarboxylate^*)}]^{2+}$$
$$\longrightarrow \mathrm{Co^{2+} + 5NH_3} + \text{oxidation products}$$
$$\phi = 0.16 \text{ at } \lambda_{irr} = 340-385 \text{ nm} \quad [9,10]$$

Although the stilbene moiety exists as an isolated chromophore it is apparently able to undergo an excited state electron transfer to the oxidizing metal.

The photochemistry of the free and coordinated ligands may be quite different if the complexation has a large influence on the electronic structure of the ligand. Trans-4-dinitrostilbene-4′-monoaza-18-crown-6 (see Electronic Spectra) undergoes a light-induced transformation to an isatogen. This photoreaction is induced by CT excitation in this donor/acceptor molecule [11]. Upon complexation of the crown ether by Ba^{2+}or Pb^{2+} a trans/cis photoisomerization of the stilbene moiety takes place because the coordination is associated with the disappearance of the CT interaction [11].

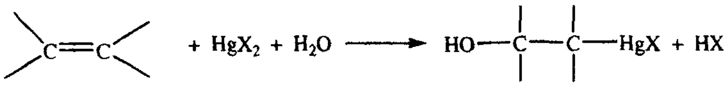
4.2. LMCT Excitation

Since Ag^+ is easily reduced to the metallic state it undergoes photoreductions initiated by LMCT excitation. A photoredox decomposition of a $[Ag(olefin)]^+$ complex was also observed [32]:

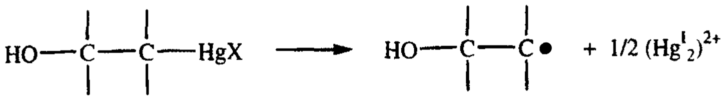
$$[\mathrm{Ag(norbornene)}]^+ \longrightarrow \mathrm{Ag}^0 + \bullet\,\mathrm{norbornene}^+$$

The norbornene radical cation reacts with the solvent (acetonitrile) in secondary processes. Unfortunately, the absorption spectrum of $[Ag(norbornene)]^+$ was not reported. While it is likely that this photoredox reaction is induced by LMCT excitation other reactive excited states cannot be ruled out presently.

A rather unconventional reduction of a metal and oxidation of an olefin takes place if aqueous Hg^{2+} is photolyzed in the presence of an olefin. In the first step the olefin reacts with Hg^{2+} and water thermally:



This hydroxomercuration [33–35] is followed by a photoredox reaction [36]:



$$\mathrm{HO{-}C{-}C{-}HgX} \longrightarrow \mathrm{HO{-}C{-}C}\bullet + 1/2\,(\mathrm{Hg^{I}}_2)^{2+}$$

The organic radical participates in secondary reactions including dimerization. While this photolyis is indeed initiated by LMCT excitation it is not a coordinated olefin but a carbanion which acts as donor ligand.

Palladium(II) is well known to undergo photoreduction upon LMCT excitation [37]. The primary photochemical step of a Pd(II) olefin complex is also induced by a LMCT transition [18]:

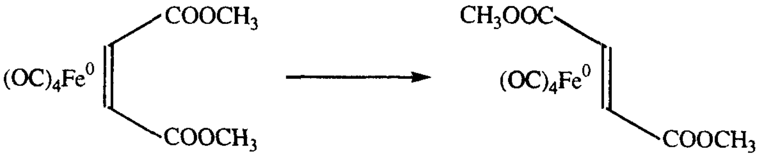
$$\left[\mathrm{Pd^{II}}(1,5-\mathrm{cyclooctadiene})\mathrm{Cl_2}\right] \longrightarrow \left[\mathrm{Pd^{I}Cl_2}\right]^{-} + 1,5-\mathrm{cyclooctadiene}^{\bullet +}$$

The cyclooctadiene radical cation isomerizes apparently. In acetonitrile a back electron transfer occurs which leads to the generation of 1,3-cyclooctadiene and a solvated Pd(II) complex. If acetonitrile is replaced by ethanol the cyclooctadiene radical cation and Pd(I) are reduced by the solvent. Metallic palladium is formed as a colloid.

4.3. MLCT Excitation

Since MLCT excitation of olefin complexes involves the population of an antibonding π orbital of the olefin the olefinic π bond is weakened in the excited state [38,39]. This should facilitate a rotation around the C-C bond. As a result a trans/cis isomerization may occur in analogy to IL-induced photoisomerizations. The complex $[\mathrm{Ru(bipy)_2(trans\text{-}4\text{-}styryl\text{-}pyridine)_2}]^{2+}$ undergoes a trans/cis photoisomerization of the styrylpyridine ligand [40]. It has been suggested that the photoreactivity originates from MLCT as well as IL states.

Such isomerizations following MLCT excitation are also observed for olefins which are coordinated side-on, e.g. [38,41]:



If π-back bonding contributes strongly to the stability, dπ→π*L MLCT transitions are associated with a considerable weakening of the M-L bond. Consequently, MLCT excitation might lead to the release of L. Indeed, several platinum complexes eject the olefin ligand upon MLCT excitation:

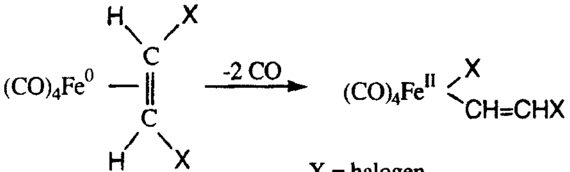
$$[\mathrm{Pt^0(PPh_3)_2(C_2H_4)}] \longrightarrow [\mathrm{Pt^0(PPh_3)_2}] + \mathrm{C_2H_4} \qquad [26]$$

$$\{\mathrm{Pt^0\,[P(OPh)_3]_2}\}\,\mathrm{C_{60}} \longrightarrow \{\mathrm{Pt^0\,[P(OPh)_3]_2}\} + \mathrm{C_{60}} \qquad [28]$$

$$[\mathrm{Pt^{II}Cl_3(C_2H_4)}]^- + \mathrm{H_2O} \longrightarrow [\mathrm{Pt^{II}Cl_3(H_2O)}]^- + \mathrm{C_2H_4} \qquad [42]$$

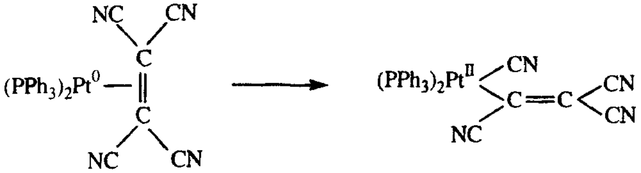
In the latter case (Zeise's salt) it is, however, not quite clear if the reactive state is of the MLCT [25] or LF type [42].

In favorable cases product formation can be related directly to the electron distribution in the excited state. MLCT transitions should then lead to the oxidation of the metal and reduction of the olefin. Oxidative additions are examples of such photoredox reactions:



X = halogen

[22]

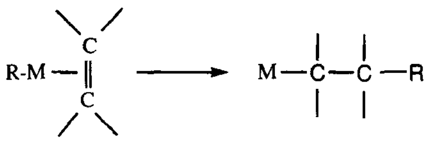


[27]

So far only olefins with electron-withdrawing substituents have been observed to undergo oxidative additions of this type. The Fe(II) complex is apparently formed in the primary photochemical step, but is converted to stable secondary products in subsequent thermal reactions.

4.4. LLCT Excitation

LLCT excitations of complexes of the type [olefin-M-R] with R = carbanions (e.g. CH_3^-) may result in an insertion:



This reaction is a key step in thermal olefin polymerization, but has not yet been observed as photochemical process. The complex $[Pt^{II}(1,5\text{-cyclooctadiene})(CH_3)_2]$. is indeed light sensitive upon $CH_3^- \rightarrow$cyclooctadiene LLCT excitation [29]. However, the photolysis leads only to a decomposition with the formation of elemental platinum. Organic products were not identified. In the presence of methyliodide an oxidative photoaddition takes place:

$$[Pt^{II}(1,5-\text{cyclooctadiene})(CH_3)_2] + CH_3I \longrightarrow 1/4\,[Pt^{IV}(CH_3)_3I]_4 + \text{cyclooctadiene}$$

While this reaction is quite interesting in its own right it does not seem to be typical for LLCT excitation.

4.5. OSCT Excitation

The photochemistry of the adducts $[(C_5H_5)_2WH_2 \times \text{olefin}]$ [30], which contain non-coordinated olefins with electron-withdrawing substituents such as CN has not yet been studied. It seems likely that outer sphere complex→olefin CT excitation leads simply to an one-electron transfer from the complex to the olefin in the primary photochemical step. This assumption is based on the observation that this redox reaction occurs thermally if the olefin is a rather strong oxidant such as TCNE [30]. Recently, we have studied a similar reaction [43]:

$$[(C_5H_5)_2Zr^{IV}ClH] + TCNE \longrightarrow [(C_5H_5)_2Zr^{IV}Cl]^+TCNE^- + 1/2H_2$$

In this case the hydride ligand serves as the electron donor since Zr(IV) is a d^0 metal.

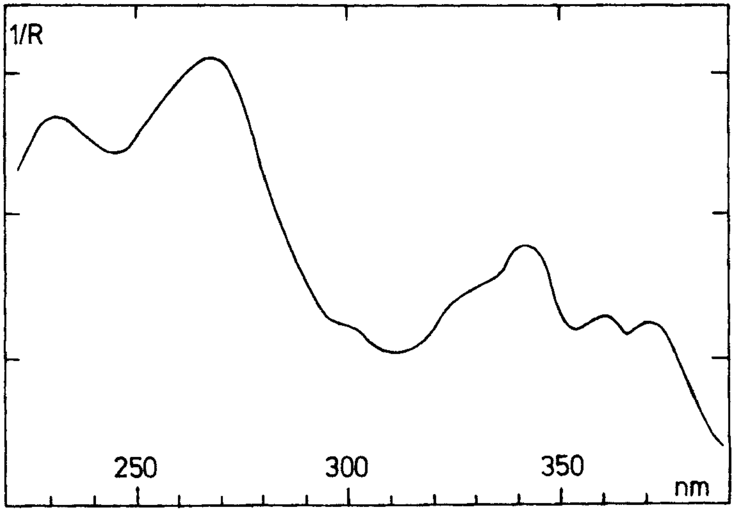


FIGURE 1 Reflectance spectrum of [MeReO$_3$(trans-4-styrylpyridine)]; 1/R (R = reflectance) in arbitrary units

5. CONCLUSION

While the importance of the photochemistry of olefin complexes has been recognized for many years a systematic characterization of the reactive excited states has begun only recently. This knowledge may extend the diversity of photoreactions of olefin complexes and lead to novel applications.

Acknowledgments

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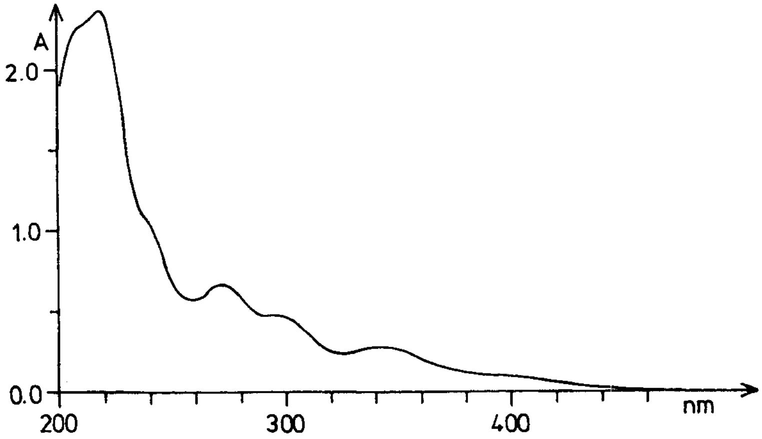

FIGURE 2 Electronic absorption spectrum of 1.46 × 10^{-4} M [Pd^{II}(1,5-COD)Cl_2] at r.t. in CH_3CN, 1 cm cell

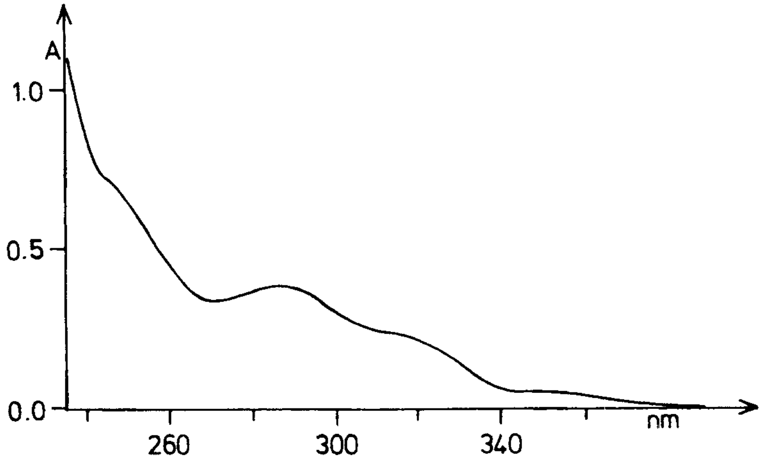

FIGURE 3 Electronic absorption spectrum of 2.88 × 10^{-4} M [Pt^{II}(1,5-COD)$(CH_3)_2$] at r.t. in CH_3CN, 1 cm cell

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