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Review

Charge transfer excitation of organometallic compounds Spectroscopy and photochemistry

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Contents

Abstract

Excited state properties of organometallic compounds have been studied for many years. Owing to strong covalent bonding, molecular orbitals of organometallics are frequently delocalized. Accordingly, the usual classification of electronic transitions in coordination compounds (e.g. metal-centered and charge transfer) may not always be appropriate. Nevertheless, in suitable cases various important organic ligands can function as CT donors (e.g. cyclopentadienyl anions, allyl anions, carbenes) and CT acceptors (e.g. acetylenes, carbenes, arenes, cyclohexadienyl cations, cycloheptatrienyl cations). Accordingly, organometallic complexes with these ligands are frequently characterized by CT absorptions. CT excitation can lead to an emission or photoreaction. This behavior is illustrated by selected examples which have been recently studied in our laboratory.

Keywords: Absorption spectra; Emission spectra; Charge transfer; Photochemistry

1. Introduction

The diversity of organometallics makes it difficult to classify these compounds in a systematic manner. Frequently, organometallic complexes are arranged according to the nature of the organic ligand and how many carbon atoms are involved in the metal–ligand interaction. While this classification can be certainly applied to organometallic photochemistry and photophysics [1], other categories may be also appropriate in this case. Generally, it is useful to distinguish different types of excited states. Metal complexes are characterized by metal-centered (MC), ligand-to-metal charge transfer (LMCT), metal-to-ligand charge transfer (MLCT), ligand-to-ligand charge transfer (LLCT), metal-to-metal charge transfer (MMCT), intraligand (IL) and intraligand charge transfer (ILCT) excited states [2–5]. Ligands can thus function as CT donors and/or CT acceptors. In the following discussion some important ligands in organometallic chemistry are classified according to their ability to donate or accept electrons in low-energy CT transitions. These assignments are, however, only limiting descriptions since strong covalent interactions prevail in most organometallics. Frequently, CT donors are anions (e.g. cyclopentadienyl, allyl) while many CT acceptors are

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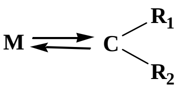
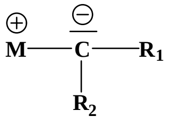
cations (e.g. cyclohexadienyl, cycloheptatrienyl). Neutral ligands such as carbenes, acetylenes, olefins and arenes can act as CT donors or acceptors depending on the metal and its oxidation state. Our short review does not cover this topic comprehensively. CT excited states which are associated with these ligands are illustrated by selected examples. This account is essentially based on recent observations in our laboratory.

2. Carbene complexes

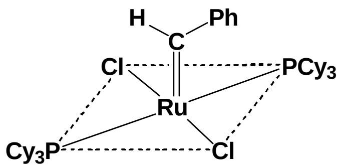
A variety of metal–carbene complexes has been prepared and characterized. Generally, the metal–carbene interaction leads to the formation of a metal–carbene double bond which consists of a σ-donor and a π-acceptor bond.



In most cases the free carbene ligand does not exist as a stable species but is stabilized by coordination to a transition metal in a low oxidation state. The presence of a reducing metal and an empty p-orbital at the free carbene yields a MLCT transition at relatively low energies. Numerous complexes which contain a carbene as CT acceptor are known [6]. For example, the complex $C_5H_5(CO)_2Mn^I(CPh_2)$ shows this MLCT absorption at $\lambda_{max} = 380$ nm [7]. In a simplified picture the MLCT excited state of a carbene complex can be described by the following valence formula which only reflects a charge shift.



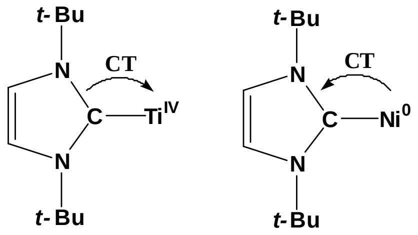
The photoreactivity is frequently based on this electron distribution [6,8–10]. For example, metal–carbene complexes can undergo a light-induced rotation around the metal–carbon bond. As a further possibility electrophiles may add to the carbon atom. Unfortunately, the identification of a reactive MLCT state is occasionally hampered by the presence of other excited states in the same energy region. This complication applies to Grubb's catalyst, $Ru^{II}(CHPh)(PCy_3)_2Cl_2$ with Cy, cyclohexyl [11] (Structure 1).



Structure 1

The broad longest-wavelength absorption of this complex at $\lambda_{max} = 525$ nm seems to consist of Ru → carbene MLCT and ligand field (LF) bands. The photolysis which leads to the release of a phosphine ligand is apparently based on a reactive LF state.

Quite recently *N*-heterocyclic carbenes (NHCs) have attracted much interest [12–14]. They exist as stable species but function also as ligands in organometallic compounds. In distinction to classical carbenes, NHCs are strong σ-donors but only weak π-acceptors. They are also able to coordinate to metals in high as well as in low oxidations states. Accordingly, NHCs could serve as CT donors and acceptors. Both functions have been recently confirmed. Suitable metals are Ti(IV) and Ni(0) [15] (Structure 2).



Structure 2

Since Ti(IV) has an empty valence shell (d^0) only MLCT transitions can occur at low energies. Indeed, the complex $TiCl_4$(dbiy) with dbiy = 1,3-di-*t*-butylimidazol-2-ylidene shows a long-wavelength absorption at $\lambda_{max} = 450$ nm which has been assigned to a (dbiy → Ti^{IV}) LMCT transition. Interestingly, $TiCl_4(PPh_3)_n$ displays a (PPh_3 → Ti^{IV}) LMCT absorption at comparable energies. On the other hand, Ni(0) is reducing and has a filled valence shell (d^{10}). It follows that $Ni(CO)_2(dbiy)_2$ should be characterized by a low-energy (Ni^0 → dbiy) MLCT transition. The corresponding absorption appears at rather short wavelength ($\lambda_{max} = 365$ nm). This observation is consistent with the assumption that the π* (or p) orbital at the coordinating carbon atom of the carbene is located at relatively high energies. $Ni(CO)_2(dbiy)_2$ exhibits an emission (Fig. 1) which originates from the (Ni^0 → dbiy) MLCT triplet.

3. Acetylene complexes

In contrast to acetylide complexes [16–18] which contain deprotonated acetylenes ($^{\ominus}|C{\equiv}C{-}R$) as ligands, very little is known about excited state properties of acetylene (or alkyne) complexes [1]. Generally, acetylenes might be CT donors and acceptors since they have available occupied π and empty π* orbitals at favorable energies. However, acetylene complexes are only stabilized by metals in low oxidation states. Accordingly, acetylene complexes are expected to display (M → acetylene) MLCT absorptions at long wavelength. This is exemplified by Cu^I(BTA)(hfac)

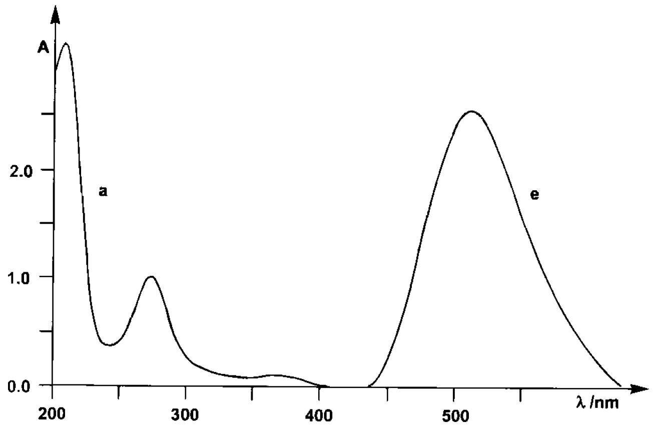
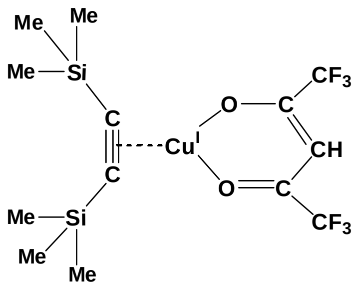


Fig. 1. Electronic absorption (a) and emission (e) spectrum of 1.19×10^{-4} M $Ni(CO)_2(dbiy)_2$ in CH_3CN under argon at rt, 1-cm cell. Emission: solid state, $\lambda_{exc} = 365$ nm, intensity in arbitrary units.

with BTA = bis(trimethylsilyl)acetylene and hfac = 1,1,1,5,5,5-hexafluoro-acetylacetonate [19] (Structure 3).



Structure 3

This complex show a ($Cu^I \rightarrow$ BTA) MLCT band at $\lambda_{max} = 324$ nm (Fig. 2). Moreover, the solid compound is emissive ($\lambda_{max} = 601$ nm). It is suggested that this luminescence originates from the lowest-energy ($Cu^I \rightarrow$ BTA) MLCT triplet. In solution the complex does not emit but undergoes a photodissociation with $\phi \sim 10^{-3}$ at $\lambda_{irr} = 333$ nm.

$$2Cu^I(BTA)(hfac) \rightarrow Cu^I_2(BTA)(hfac)_2 + BTA \qquad (1)$$

MLCT excitation leads to a weakening of the copper–acetylene bond and finally to the release of the BTA ligand.

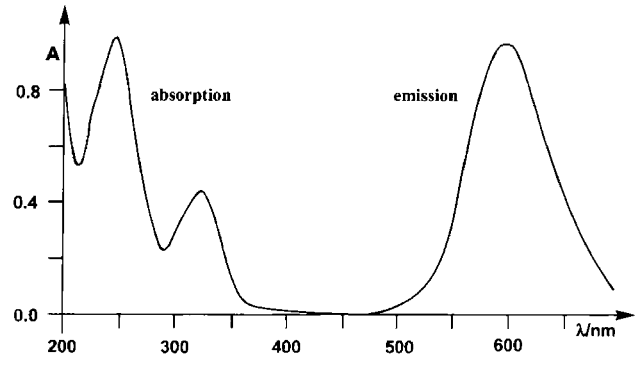


Fig. 2. Electronic absorption and emission spectrum of Cu^I(BTA)(hfac) under argon at rt absorption: 8.19×10^{-5} M in *n*-hexane, 1-cm cell. Emission: solid state, $\lambda_{exc} = 350$ nm, intensity in arbitrary units.

4. Olefin complexes

Since the spectroscopic and photochemical properties of olefin complexes [1] have been reviewed quite recently [20] only a few general aspects of this subject are presented here. By analogy with acetylenes, olefins can function as CT donors and acceptors owing to the presence of filled π and empty π^* orbitals, respectively [1,20]. Usually, π-bonding is important in olefin complexes. The metal occurs then in low oxidation states and is thus reducing. Such complexes are characterized by low-energy (M → olefin) MLCT transitions. A variety of olefin complexes containing donor metals including W(0), Mo(0), Fe(0), Rh(I), Ir(I) Pt(0) and Pt(II) have been shown to display MLCT absorptions as longest-wavelength band. Frequently, those MLCT excited states are reactive. Olefin isomerizations, substitutions as well as oxidative additions have been observed to result from MLCT excitation [1,20]. A very recent study deals with the photoreactivity of Cu(COD)(hfac) with COD = 1,5-cyclooctadiene [21]. Many copper(I) olefin complexes are well known to be light sensitive [1,22]. Unfortunately, in most cases the nature of the reactive excited state is not clear since metal complexes as well as free olefins were present as light-absorbing species. Accordingly, the role of copper(I) is not well defined. Cu(COD)(hfac) is reasonably stable in hexane. Its absorption spectrum contains a ($Cu^I \rightarrow$ COD) MLCT band at $\lambda_{max} = 241$ nm and an hfac IL band at 308 nm. The photolysis leads apparently to the release of the COD ligand. Cu(I)(hfac) as primary photoproduct is not stable, but disproportionates:

$$Cu^I(COD)(hfac) \rightarrow Cu^I(hfac) + COD \qquad (2)$$

$$2Cu^I(hfac) \rightarrow Cu^0 + Cu^{II}(hfac)_2 \qquad (3)$$

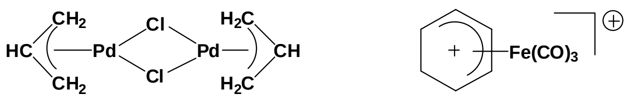
It is assumed that the photolysis originates from the ($Cu^I \rightarrow$ COD) MLCT state which can be populated directly or from the hfac IL state by an internal conversion. The dissociative loss of ligands induced by MLCT excitation is a general feature of many complexes with π-bonding ligands [23].

Although the spectroscopy and photochemistry of most olefin complexes seem to be determined by MLCT excited states, olefins can also act as CT donors [20]. This applies to complexes which contain oxidizing metal centers such as Ag(I) and Pd(II). Accordingly, (olefin → M) LMCT absorptions occur in the spectra of their olefin complexes. LMCT excitation of $Pd^{II}(COD)Cl_2$ leads to the reduction of Pd(II) to Pd(I) and oxidation of COD to its cation radical in the primary photochemical step [24]. Product formation occurs in subsequent reactions. In ethanol as solvent Pd(I) is finally reduced to the metal.

5. Allyl and enyl complexes

While neutral allyl or enyl groups are olefin derivatives with an odd number of electrons they can be viewed as anionic or cationic ligands with an even number of electrons depending on the specific complex. This picture is certainly an oversimplification since the metal–allyl or metal–enyl bonding is rather covalent with a considerable electron delocalization. Nevertheless, according to a simple MO analysis a formal distinction can be made. As a consequence, anions have an excess electron and are electron donors, while cations have an electron hole and are thus electron acceptors. Both cases are exemplified by the complexes $[\text{Pd(allyl)Cl}]_2$ [25] and $[\text{Fe}(\text{C}_6\text{H}_7)(\text{CO})_3]^+$ [26] (Structure 4).

Structure 4



According to this formalism the binuclear compound $[(\text{allyl})\text{PdCl}]_2$ contains Pd(II) and the allyl ligand as an anion [25]. The longest-wavelength absorption at $\lambda_{\max} = 318\,\text{nm}$ is then assigned to a $(\text{allyl}^- \rightarrow \text{Pd}^{\text{II}})$ LMCT transition although the actual CT contribution to this transition may be rather small (Fig. 3). LMCT excitation leads to a photoredox reaction according to the following mechanism:

$$[(\text{allyl})\text{Pd}^{\text{II}}\text{Cl}]_2 \xrightarrow{h\nu/\text{CH}_3\text{CN}} \text{allyl radical} + \text{Pd}^{\text{I}}\text{Cl} + \tfrac{1}{2}[(\text{allyl})\text{Pd}^{\text{II}}\text{Cl}]_2 \tag{4}$$

$$2\text{Pd}^{\text{I}}\text{Cl} + 2\text{CH}_3\text{CN} \rightarrow \text{Pd}^0 + \text{Pd}^{\text{II}}(\text{CH}_3\text{CN})_2\text{Cl}_2 \tag{5}$$

$$2\,\text{allyl radicals} \rightarrow \text{hexa-1, 5-diene} \tag{6}$$

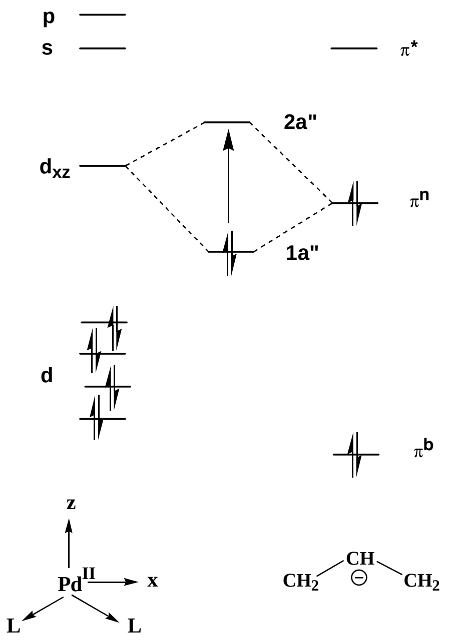


Fig. 3. Qualitative MO scheme for (π-allyl)$\text{Pd}^{\text{II}}\text{L}_2$ complexes (C_s symmetry).

The complex $[(\text{C}_6\text{H}_7)\text{Fe}(\text{CO})_3]^+$ can be considered to contain Fe(0) and a cyclohexadienyl cation [26]. The longest-wavelength absorption of the complex at $\lambda_{\max} = 281\,\text{nm}$ is then assigned to a $(\text{Fe}(0) \rightarrow \text{C}_6\text{H}_7^+)$ MLCT transition. The MLCT excitation is followed by a relaxation which is finally associated with a two-electron transfer:

$$[(\text{C}_6\text{H}_7{}^+)\text{Fe}^0(\text{CO})_3]^+ \xrightarrow{\text{MLCT}} [(\text{C}_6\text{H}_7{}^-)\text{Fe}^{\text{II}}(\text{CO})_3]^+ \tag{7}$$

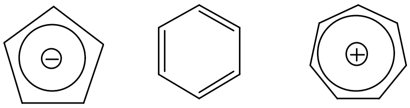
As a consequence of this “umpolung” the complex becomes susceptible to an electrophilic addition at the cyclohexadienyl ligand. In aqueous solution a proton is added:

$$[(\text{C}_6\text{H}_7{}^-)\text{Fe}^{\text{II}}(\text{CO})_3]^+ + \text{H}^+ \xrightarrow{h\nu} [(\text{C}_6\text{H}_8)\text{Fe}^{\text{II}}(\text{CO})_3]^{2+} \tag{8}$$

The resulting complex is not stable but decomposes to 1,3-cyclohexadiene and Fe^{2+} ions which are hydrated in aqueous solution.

6. Complexes with aromatic 6π-electron ligands

Aromatic 6π-electron donors constitute an important family of ligands in organometallic chemistry. The most significant ligands of this type are the cyclopentadienyl (Cp^-) anion, benzene and the cycloheptatrienyl cation (Structure 5).

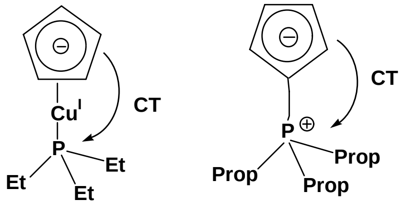


Structure 5

Since the π* orbitals of the Cp^- ligand are located at rather high energies it is essentially a CT donor, but hardly a CT acceptor [27]. On the contrary, $\text{C}_7\text{H}_7{}^+$ has its π* orbitals at relatively low energies [28]. Accordingly, $\text{C}_7\text{H}_7{}^+$ is a strong CT acceptor. Benzene is an intermediate case [29]. It is a CT donor and acceptor of moderate strength.

6.1. Cyclopentadienyl complexes

Since Cp^- is a CT donor, low-energy LMCT transitions occur if Cp^- coordinates to oxidizing metals [1,2,27]. This applies, for example, to the ferrocenium cation $[\text{Fe}^{\text{III}}\text{Cp}_2]^+$ and the neutral complex $\text{CpRe}^{\text{VII}}\text{O}_3$. If Cp complexes contain also acceptor ligands, LLCT absorptions may be observed. The complex $\text{CpCu}^{\text{I}}(\text{PEt}_3)$ is a recent example of this type of CT interaction [30] (Structure 6).

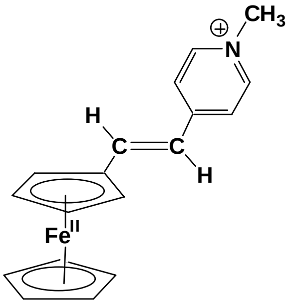


Structure 6

The $(\text{Cp}^- \rightarrow \text{PEt}_3)$ LLCT absorption of $\text{CpCu}(\text{PEt}_3)$ appears at $\lambda_{\max} = 350\,\text{nm}$. The complex is also luminescent

($\lambda_{max} = 525$ nm, $\phi = 0.003$ at $\lambda_{exc} = 320$ nm). This emission is suggested to originate from the LLCT triplet. Interestingly, a ($Cp^- \rightarrow PEt_3$) CT transition is also observed in the spectrum of the ylide Cp–P(Prop)$_3$ [31]. In the ylide the CT donor and acceptor are directly connected by a C–P bond without an intervening metal.

The Cp ligand cannot only participate as CT donor in LMCT and LLCT but also in ILCT transitions. In this case the coordinated Cp ligand carries a CT acceptor as substituent. The complex cation (E)-1-ferrocenyl-2-(1-methyl-4-pyridiniumyl)ethylene (E-fmpe$^+$) contains a pyridinium substituent as CT acceptor [32,33] (Structure 7).



Structure 7

This complex shows an absorption near 370 nm which was assigned to a ($Cp^- \rightarrow$ pyridinium$^+$) ILCT transition. An additional ($Fe^{II} \rightarrow$ pyridinium$^+$) MLCT band appears at longer wavelength. MLCT as well as ILCT excitation leads to a *trans*/*cis*-isomerization at the olefinic double bond [32]. The complex is photochromic since the photoisomerization is thermally reversed.

6.2. Benzene complexes

As mentioned above benzene is a CT donor and acceptor [29]. In combination with oxidizing metals (π^b benzene $\rightarrow$ metal) LMCT transitions occur while the coordination to reducing metals leads to (metal $\rightarrow$ π^* benzene) MLCT transitions at low energies [1,29].

Various complexes with long-wavelength (benzene $\rightarrow$ metal) LMCT absorptions have been identified, but photoreactivity originating from such LMCT states has been rarely observed [29,34,35]. A recent example is BiCl$_3$(benzene) which is assumed to be formed when BiCl$_3$ is dissolved in benzene. Irradiation of BiCl$_3$(benzene) initially leads to the population of a metal-centered sp excited state which is deactivated to a reactive (benzene $\rightarrow$ Bi^{III}) LMCT state [36]. Product formation can be rationalized by the following scheme:

$$Bi^{III}Cl_3(\text{benzene}) \xrightarrow{h\nu} C_6H_6{}^+[Bi^{II}Cl_3]^- \quad (9)$$

$$C_6H_6{}^+[Bi^{II}Cl_3]^- \rightarrow C_6H_5 + BiCl_2 + HCl \quad (10)$$

$$2C_6H_5 \rightarrow C_6H_5 - C_6H_5 \quad (11)$$

$$C_6H_5 + BiCl_3 \rightarrow C_6H_5Cl + BiCl_2 \quad (12)$$

$$3BiCl_2 \rightarrow Bi + 2BiCl_3 \quad (13)$$

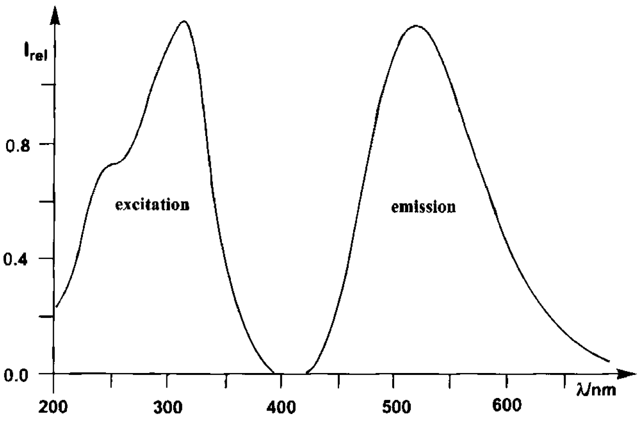


Fig. 4. Electronic excitation (λ_{em} = 518 nm) and emission ($\lambda_{exc} = 320$ nm) spectrum of solid Cu$_2$(CF$_3$SO$_3$)$_2$(C$_6$H$_6$) under argon at rt, intensity in arbitrary units.

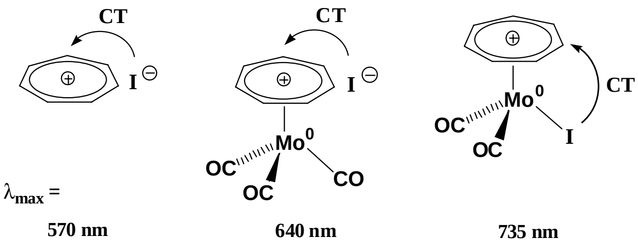
Benzene (or arene) complexes of reducing metals (e.g. Mo^0(arene)(CO)$_3$ [1] or [Os^{II}(C$_6$H$_6$)$_2$]$^{2+}$ [37]) have certainly available (M $\rightarrow$ arene) MLCT states at intermediate energies. Unfortunately, there is not much known about the excited state properties of these MLCT states because they are not emissive but are rapidly deactivated to LF states at lower energies. In their LF excited states these complexes simply undergo a photosubstitution, but not a photoredox reaction as expected for a CT state. How can this interference by LF states be avoided? Copper(I) is a suitable metal since it is reducing. Owing to its filled d-shell (d^{10}) it does not provide LF states at all. Indeed, the complex Cu$_2$(CF$_3$SO$_3$)$_2$(C$_6$H$_6$) is characterized by a lowest-energy (Cu^I $\rightarrow$ benzene) MLCT excited state [38]. This MLCT state has been observed to emit (Fig. 4). Electronic excitation of solid Cu$_2$(CF$_3$SO$_3$)$_2$(C$_6$H$_6$) leads to a luminescence ($\lambda_{max} = 518$ nm, $\phi \sim 10^{-2}$, $\tau = 0.5$ μs) which is assumed to come from a MLCT triplet.

6.3. Cycloheptatrienyl complexes

As discussed above, complexes such as Mo^0(arene)(CO)$_3$ have (Mo^0 $\rightarrow$ arene) MLCT states available, but they are located above the lowest-energy LF states [1]. An inversion may be achieved if the π^* orbitals of the arene are pushed to lower energies. This goal can be accomplished if ordinary arenes are replaced by the cycloheptatrienyl cation. In distinction to other (arene)Mo^0(CO)$_3$ complexes, the cation [C$_7$H$_7$Mo^0(CO)$_3$]$^+$ shows an emission (λ_{max} = 578 nm) which originates from the lowest-energy (Mo^0 $\rightarrow$ C$_7$H$_7{}^+$) triplet [28].

The C$_7$H$_7{}^+$ cation does not only serve as CT acceptor for reducing metals, but also for reducing anions such as iodide. It has been known for many years that the ion pair C$_7$H$_7{}^+$I$^-$ displays an ($I^- \rightarrow C_7H_7{}^+$) outer sphere CT absorption [39–41]. This CT transition occurs also in the ion pair [(η^7-C$_7$H$_7$)Mo^0(CO)$_3$]$^+$I$^-$ [42]. Finally, the (I$^-$ $\rightarrow$ C$_7$H$_7{}^+$) CT transition appears as a LLCT absorption in the spectrum of the complex $C_7H_7Mo^0(CO)_2I$ [42] (Structure 8).

Structure 8



7. Conclusion

Although organometallic compounds are generally characterized by extensive covalent bonding, it is often possible to identify metals and ligands as CT donors or accceptors. A variety of organic species such as carbenes, an acetylene, olefins, the allyl anion, the cyclohexadienyl cation, the cyclopentadienyl anion, benzene and the cycloheptatrienyl cation have been shown to function as ligands which participate in low-energy CT transitions. Frequently, such CT states are emissive and/or reactive.

Acknowledgements

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