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# Spectroscopic Properties and Molecular Structure of a Luminescent Cyclometalated Mercury(II) Complex of 2,9-Diphenyl-1,10-phenanthroline

**Chin-Wing Chan,[†] Shie-Ming Peng,[‡] and Chi-Ming Che[*,†]**

Departments of Chemistry, The University of Hong Kong, Pokfulam Road, Hong Kong, and National Taiwan University, Taipei, Taiwan

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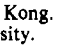
The reaction of mercuric acetate with 2,9-diphenyl-1,10-phenanthroline (dpp) in refluxing ethanol and in the presence of lithium chloride gave the cyclometalated complex $[Hg(C\wedge N\text{-dpp})Cl]_2$. X-ray crystal analysis revealed a dimeric molecular structure: $[HgC_{24}H_{15}N_2Cl]_2$, fw = 567.48 × 2, $P2_1/n$, $a = 11.79(1)$ Å, $b = 17.80(1)$ Å, $c = 18.84(1)$ Å, $\beta = 99.23(6)°$, $V = 3904(5)$ Å$^3$, $Z = 4$. The long Hg–N distances (2.60–3.85 Å) and proton chemical shifts of the metalated dpp suggest that the 1,10-phenanthroline ring is weakly coordinated to the Hg–phenyl moiety. The complex is luminescent at both 77 and 298 K. Photosensitivity of $[Hg(C\wedge N\text{-dpp})Cl]_2$ at room temperature is evidenced by its facile transformation into the protonated salt of dpp ($Hdpp^+$) in dichloromethane. The low-temperature emission spectrum of $[Hg(C\wedge N\text{-dpp})Cl]_2$ is almost identical to that of dpp. The photophysical and emission properties of $[Hg(C\wedge N\text{-dpp})Cl]_2$ are compared with those of the related cyclometalated Pt(II) and Au(III) complexes, viz. $[Pt(C\wedge N\wedge N\text{-dpp})(NCCH_3)]^+$ and $[Au(C\wedge N\wedge N\text{-dpp})Cl]^+$.

## Introduction

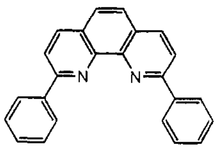
Cyclometalated transition metal complexes[1] have been of considerable importance in the study of inorganic photochemistry.[2] Although there are numerous reports on the photophysical and photochemical properties of cyclometalated palladium(II) and platinum(II) complexes,[2,3] related studies on the gold(III)[4] and mercury(II)[5] systems are still sparse. The propensity of mercury (II) toward electrophilic substitution on aromatic rings provides a useful synthetic method for cyclomercurated complexes.[6] In spite of the synthetic utilities of mercury(II) compounds in both inorganic and organic chemistry, little attention has been given to their emission and photochemical properties.[5,7] Vogler had reported the first luminescent cyclomercurated complex, [Hg(L)Cl]$_4$ (HL= 2-phenylpyridine), the emission of which was attributed to come from a metal-center/LMCT excited state.[5] Herein is described the synthesis and spectroscopic properties of a cyclometalated mercury(II) complex of 2,9-diphenyl-1,10-phenanthroline.



HL

## Experimental Section

**Materials.** The ligand 2,9-diphenyl-1,10-phenanthroline (dpp) was prepared as a pale yellow solid by the reported procedure.[8] All other reagents were used as received. Dichloromethane was freshly distilled from $P_2O_5$.



dpp

**Synthesis.** A mixture of mercuric acetate (0.318 g, 1 mmol) and dpp (0.33 g, 1 mmol) was refluxed in absolute ethanol (20 mL) for 6 h. Anhydrous lithium chloride (0.1 g, 2.4 mmol) in methanol (20 mL) was added, and the resulting mixture was further refluxed for 30 min. The solution was filtered through Celite when hot. The cream-white precipitate separated from solution upon cooling to room temperature was filtered out and washed with ethyl acetate to remove any unreacted dpp. The yield was 20%. The $^1H$ NMR spectrum was measured in $CDCl_3$: $\delta$ 8.41 (m, H16,20), 8.38 (d, H8), 8.32 (d, H13), 8.16 (d, H9), 8.14 (d, H12), 8.03 (dd, H5), 7.83 (dd, H10,11), 7.66 (dd, H2), 7.53 (m, H17,18,19), 7.53 (M, H3,4). IR (cm$^{-1}$): 3045 w, 1582 m, 1497 m, 1480 m, 635 w, 438 w. The UV–vis spectrum was recorded in $CH_2Cl_2$: ($\lambda_{max} = 269$ nm, $10^{-3}\epsilon = 77.1$), (310, 51.6), (327, 35.4), (340, 25), (358, 8). The structure of the complex has also been estalished by X-ray crystal analysis.

**Physical Measurements.** Infrared spectra were obtained in KBr pellets on a Nicolet 20 SXC FT-IR spectrometer. UV–visible spectra were obtained on a Milton Roy Spectronic 3000 spectrophotometer. Proton NMR spectra were obtained on a Jeol GSX 270 FT-NMR spectrometer with TMS as an internal standard.

Steady-state emission spectra were recorded on a Spex 1681 Fluorolog-2 Series F111AI spectrometer. Steady-state photolysis was also taken on the same instrument at fixed wavelength. Solutions for photophysical measurements were degassed through no fewer than three freeze–pump–thaw cycles.

**Crystal Data Collection and Analysis.** A light yellow prism of $[Hg(C\wedge N\text{-dpp})Cl]_2$ was obtained by slow diffusion of diethyl ether into the acetone solution of the complex. Diffraction measurements were carried out on a Nonius CAD-4 fully automated four-circle diffractometer at National Taiwan University. The unit cell was determined and refined using setting angles of 25 reflections, with 2Θ angles in the range from

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[*] To whom correspondence should be addressed.

[†] The University of Hong Kong.

[‡] National Taiwan University.

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**Table 1.** Crystal Data and Refinement Parameters for [Hg(C^N-dpp)Cl]$_2$

| | | | |
|---|---|---|---|
| formula | $[HgC_{24}H_{15}N_2Cl]_2$ | *c*, Å | 18.84(1) |
| fw | 567.48 × 2 | *β*, deg | 99.23(6) |
| cryst system | monoclinic | *V*, Å$^3$ | 3904(5) |
| space group | $P2_1/n$ | *Z* | 4 |
| *a*, Å | 11.79(1) | *ρ*(calcd), g·cm$^{-3}$ | 1.931 |
| *b*, Å | 17.80(1) | | |

**Table 2.** Atomic Coordinates and $B_{eq}$ Values (Å$^2$) of Non-Hydrogen Atoms for [Hg(C^N-dpp)Cl]$_2$, where Esd's Refer to the Last Digit Printed

| | *x* | *y* | *z* | $B_{eq}$ |
|---|---|---|---|---|
| Hg(A) | 0.58214(11) | 0.60132(6) | 0.02244(8) | 4.62(7) |
| Cl(A) | 0.6068(8) | 0.5149(5) | −0.0658(5) | 6.4(5) |
| N(1A) | 0.7929(23) | 0.6481(12) | 0.1005(12) | 4.1(14) |
| N(2A) | 0.8635(19) | 0.5011(12) | 0.1021(12) | 3.3(12) |
| C(1A) | 0.547(3) | 0.6871(14) | 0.0932(14) | 3.7(17) |
| C(2A) | 0.434(3) | 0.6994(16) | 0.1062(17) | 4.3(17) |
| C(3A) | 0.417(3) | 0.7533(17) | 0.1527(20) | 5.1(19) |
| C(4A) | 0.508(3) | 0.7944(15) | 0.1872(16) | 4.3(18) |
| C(5A) | 0.614(3) | 0.7838(16) | 0.1719(15) | 4.1(16) |
| C(6A) | 0.645(3) | 0.7298(14) | 0.1228(14) | 3.1(15) |
| C(7A) | 0.7559(22) | 0.7185(14) | 0.1064(15) | 3.2(15) |
| C(8A) | 0.839(3) | 0.7786(15) | 0.1078(18) | 4.7(19) |
| C(9A) | 0.937(3) | 0.7643(16) | 0.0921(18) | 4.8(18) |
| C(10A) | 0.983(3) | 0.6906(16) | 0.0864(15) | 3.9(15) |
| C(11A) | 1.0906(22) | 0.6712(16) | 0.0741(16) | 3.5(16) |
| C(12A) | 1.1239(23) | 0.5997(19) | 0.0715(18) | 4.9(19) |
| C(13A) | 1.0517(24) | 0.5412(16) | 0.0846(15) | 3.7(15) |
| C(14A) | 1.0875(22) | 0.4648(17) | 0.0876(17) | 4.3(17) |
| C(15A) | 1.002(3) | 0.4136(15) | 0.0973(18) | 5.6(21) |
| C(16A) | 0.898(3) | 0.4330(16) | 0.1048(16) | 3.4(15) |
| C(17A) | 0.8993(25) | 0.6331(15) | 0.0919(16) | 3.7(16) |
| C(18A) | 0.9339(24) | 0.5550(13) | 0.0919(14) | 3.0(14) |
| C(19A) | 0.816(3) | 0.3734(15) | 0.1197(16) | 4.0(16) |
| C(20A) | 0.840(3) | 0.2982(16) | 0.1138(17) | 4.8(19) |
| C(21A) | 0.760(3) | 0.2458(16) | 0.1331(19) | 5.6(20) |
| C(22A) | 0.665(3) | 0.2662(18) | 0.1618(18) | 6.0(20) |
| C(23A) | 0.637(3) | 0.3429(19) | 0.1704(18) | 5.8(20) |
| C(24A) | 0.715(3) | 0.3935(15) | 0.1467(18) | 5.1(19) |
| Hg(B) | 0.15672(11) | 1.04084(6) | 1.03089(7) | 4.37(7) |
| Cl(B) | 0.1041(7) | 0.9184(4) | 0.9939(5) | 6.1(5) |
| N(1B) | 0.3219(16) | 1.0879(12) | 0.9660(12) | 2.6(11) |
| N(2B) | 0.1993(24) | 1.0158(11) | 0.8532(13) | 3.6(14) |
| C(1B) | 0.229(3) | 1.1400(16) | 1.0794(16) | 4.5(7) |
| C(2B) | 0.1503(24) | 1.1808(19) | 1.1188(17) | 4.7(17) |
| C(3B) | 0.192(3) | 1.2478(20) | 1.1511(17) | 5.5(19) |
| C(4B) | 0.294(4) | 1.2761(17) | 1.1377(19) | 6.0(20) |
| C(5B) | 0.3521(24) | 1.2398(18) | 1.0921(18) | 4.1(18) |
| C(6B) | 0.3135(22) | 1.1703(15) | 1.0623(15) | 2.9(14) |
| C(7B) | 0.3788(24) | 1.1324(15) | 1.0154(18) | 3.7(16) |
| C(8B) | 0.503(3) | 1.1408(15) | 1.0200(17) | 4.8(17) |
| C(9B) | 0.567(3) | 1.1021(18) | 0.9723(19) | 4.6(18) |
| C(10B) | 0.503(3) | 1.0561(17) | 0.9203(17) | 3.8(16) |
| C(11B) | 0.553(3) | 1.0114(21) | 0.8700(23) | 6.1(22) |
| C(12B) | 0.494(4) | 0.9692(20) | 0.8214(20) | 5.9(22) |
| C(13B) | 0.374(4) | 0.9687(17) | 0.8178(19) | 5.2(21) |
| C(14B) | 0.301(5) | 0.9284(19) | 0.7609(22) | 6.7(26) |
| C(15B) | 0.190(4) | 0.9312(18) | 0.7549(20) | 6.3(24) |
| C(16B) | 0.134(4) | 0.9789(16) | 0.8001(19) | 4.9(21) |
| C(17B) | 0.382(3) | 1.0531(15) | 0.9170(14) | 3.4(15) |
| C(18B) | 0.314(3) | 1.0118(14) | 0.8618(16) | 3.3(16) |
| C(19B) | 0.012(4) | 0.9912(22) | 0.7906(19) | 5.8(23) |
| C(20B) | −0.069(4) | 0.9434(23) | 0.7512(23) | 8.5(26) |
| C(21B) | −0.175(5) | 0.955(3) | 0.738(3) | 11.1(37) |
| C(22B) | −0.221(4) | 1.017(3) | 0.759(3) | 8.3(31) |
| C(23B) | −0.151(4) | 1.0682(23) | 0.8015(24) | 8.5(27) |
| C(24B) | −0.035(3) | 1.0536(22) | 0.8144(21) | 6.3(22) |

23.06 to 24.60°. All data reduction and structure refinement were performed using the NRCC-SDP-VAX packges. The structure was solved by the Patterson method and refined by least squares; all non-hydrogen atoms were refined with anisotropic thermal parameters. Regression analysis was carried out on 3222 unique data and 501 parameters. The residual extrema in the final difference map ranged from 2.430 to −2.410 e Å$^{-3}$. The final agreement factors *R*, $R_w$, and *S* were 0.075, 0.073, and 2.95, respectively. The crystal data and refinement parameters are summarized in Table 1. Table 2 lists the atomic coordinates of non-hydrogen atoms. Selected bond distances and bond angles are given in Table 3.

**Table 3.** Selected Bond Distances and Bond Angles of [Hg(C^N-dpp)Cl]$_2$[a,b]

| | | | |
|---|---|---|---|
| | Bond Distances (Å) | | |
| Hg–Hg′ | 4.117(3) | Hg–N(1) | 2.80(3) |
| Hg–Cl | 2.317(8) | Hg–N(2) | 3.85(2) |
| Hg–Cl′ | 3.240(8) | Hg–C(1) | 2.11(3) |
| Cl–Cl′ | 3.843(2) | | |
| | Bond Angles (deg) | | |
| Cl–Hg–C(1) | 173.2(8) | Hg–C(1)–C(6) | 113.9(2) |
| N(1)–Hg–C(1) | 72.62(2) | C(1)–C(6)–C(7) | 124(3) |
| N(1)–Hg–Cl | 111.26(2) | N(1)–C(7)–C(6) | 118.7(2) |
| Hg–C(1)–C(2) | 120.6(2) | | |

[a] There are two distinct structural units in the crystal lattice. [b] The atoms corresponding to the other half of the dimer are denoted by an apostrophe.

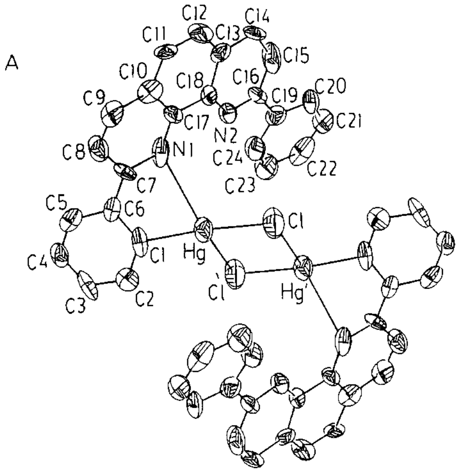

**Figure 1.** Perspective view of dimeric [Hg(C∧N-dpp)Cl]$_2$ (conformer A).

## Results and Discussion

Mercuration on a benzene ring with the electrophilic mercury(II) acetate has been an important reaction in the development of organomercury(II) chemistry.[9] Examples of cyclomercuration were previously observed in cases of 2-phenylpyridine[6a,b] and 6-phenyl-2,2′-bipyridine.[6c] However, molecular structural data are available for the former case only.[6b]

In this work, X-ray crystal analysis revealed two structural units of [Hg(C∧N-dpp)Cl]$_2$ in the crystal lattice. The two types of structural units A and B are distinguished by their different conformations around the interannular bonds between the phenyl group and phenanthroline ring. Figure 1 shows the structure of conformer A of [Hg(C∧N-dpp)Cl]$_2$ with an intramolecular metal–metal separation of 4.117(3) Å. Unlike that of (2-pyridylphenyl)mercury(II) chloride which has a tetrameric structure,[6b] the degree of aggregation for the mercurated dpp is apparently prevented by the steric bulkiness of ligand. The disposition of atoms (C(1), Cl, Cl′) around Hg(II) approximates an elongated tetrahedron with one of the two Hg–Cl distances (Hg–Cl′ = 3.240(8) Å) substantially longer than the other (Hg–Cl = 2.317(8) Å).

The Hg–C distances (2.11(3) Å) are slightly longer than those observed in the other aryl–mercury(II) complexes such as phenylmercury(II) acetate (Hg–C = 1.92 Å).[10] In both types of conformations A and B, the Hg(II) ions are almost coplanar with the metalated phenyl ring (maximum deviation less than 0.09(5) Å). The measured Hg–N(1) and Hg–N(2) distances

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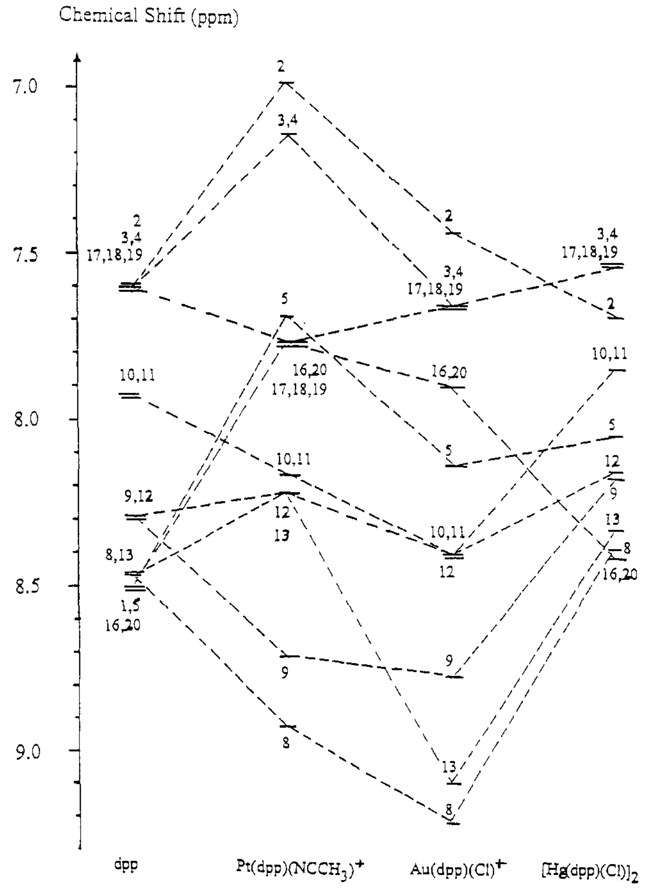


**Figure 2.** Correlation diagram of chemical shifts for the $^1$H-NMR of dpp, $[Pt(C\wedge N\wedge N\text{-dpp})(NCCH_3)]^+$, $[Au(C\wedge N\wedge N\text{-dpp})Cl]^+$, and $[Hg(C\wedge N\text{-dpp})Cl]_2$.

are 2.80(3) and 3.85(2) Å, respectively, suggesting that the phenanthroline ring is only weakly coordinated to the Hg$^{II}$–phenyl moiety.[11] In the monomeric unit of $[Hg(C\wedge N\text{-dpp})Cl]_2$, the non-metalated phenyl ring forms larger dihedral angles with phenanthroline (type A = 28.6°, type B = 38.6°) than those of metalated one (type A = 23°, type B = 16.2°).

**Mass Spectrometric and 2D-NMR Characterization.** The ratio of $M/M + 2$ for the largest molecular fragment ($m/z = 568$) of $[Hg(C\wedge N\text{-dpp})Cl]_2$ is about 3/1. This indicates dissociation of the complex into its monomer counterpart $[Hg(C\wedge N\text{-dpp})Cl]$. Mass fragments at $m/z = 533$ and 331 revealed fragmentation of the complex at the Hg–Cl and Hg–C bonds, respectively. The mass fragment at $m/z = 366$ ($M/M + 2 = 3/1$) is indicative of chloro-substituted dpp probably formed by reductive elimination of chloride and cyclometalated dpp.

Though the abundance of $^{199}$Hg ($I = {}^1/_2$) is 16.86%, no spin satellite arising from $^1$H–$^{199}$Hg coupling[12] has been observed in the $^1$H NMR spectrum recorded at room temperature (Figure S1 of supplementary material). The chemical shifts are analyzed and compared with those of free dpp, $[Pt^{II}(C\wedge C\wedge dpp)(NCCH2)]^+$, and $[Au^{III}(C\wedge N\wedge N\text{-dpp})Cl]^+$, and the results are shown in Figure 2. The magnitudes of chemical shift splitting for protons on the phenanthroline ring are not as large as those in the corresponding cyclometalated platinum(II) and gold(III) complexes. This is not unexpected given the weak interaction between mercury(II) and nitrogen observed in the molecular structure. Figure 2 compares the chemical shifts for structurally related cyclometalated complexes of dpp along with the free ligand. The apparent deshielding effect on phenyl and phenanthroline

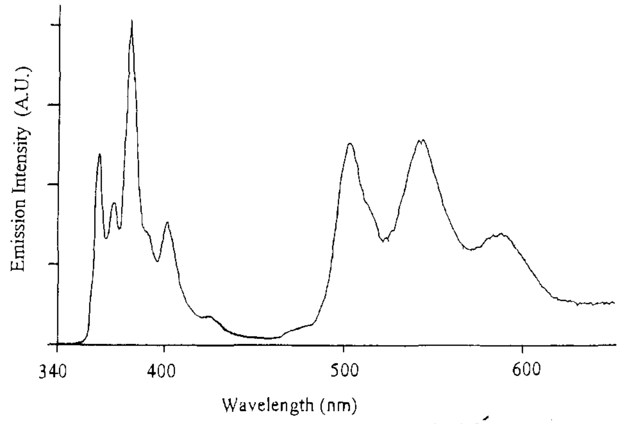
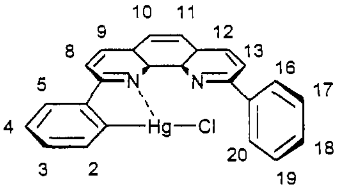


**Figure 3.** Emission spectrum of $[Hg(C\wedge N\text{-dpp})Cl]_2$ in 77 K *n*-butanenitrile glass excited at 330 nm.

protons increases upon metalation with d$^8$ Pt(II) and Au(III). Shielding of the protons adjacent to the metalated carbon in $[Pt(C\wedge N\wedge N\text{-dpp})(NCCH_3)]^+$ and $[Au(C\wedge N\wedge N\text{-dpp})Cl]^+$ has previously been ascribed to the almost perpendicular orientation of the metalated and non-metalated phenyl rings.[2d,4] The proton chemical shifts of $[Hg(C\wedge N\text{-dpp})Cl]_2$



numbering scheme for monomeric unit of mercury(II) compound

are almost the same as those of free ligand with deviations less than 0.2 ppm except for that of H(5), which experiences an upfield shift of 0.45 ppm upon mercuration. The small perturbation reflects little magnetic influence generated by the Hg(II) ion which is consistent with the closed-shell d$^{10}$ configuration of mercury(II) and the weak covalent interaction between Hg(II) and ($C\wedge N$-dpp).

**Spectroscopic and Photochemical Properties.** The UV–vis absorption spectrum of $[Hg(C\wedge N\text{-dpp})Cl]_2$ measured in dichloromethane (see also spectral trace at time = 0 in Figure 4) is virtually identical to those of dpp measured under the same conditions.[13] Apart from the $\pi$–$\pi^*$ transitions from 269 to 340 nm, there seems no indication of low-energy bands attributable to either LMCT or MLCT transition. Presumably, these transitions may occur at high-UV region and cannot be located under our conditions.

Photoluminescence has been observed from the $[Hg(C\wedge N\text{-dpp})Cl]_2$ complex at both 77 and 298 K. In an *n*-butanenitrile glass at 77 K, the emission bands are more intense and better resolved. The emission spectrum shown in Figure 3 (77 K *n*-butanenitrile glass, excitation at 330 nm) is almost identical to that of the free ligand taken under the same conditions, except with a small increase in the intensity of the low-energy emission band. As shown in Figure 3, a high-energy vibronic emission band at 350–410 nm and a lower energy one at 520–600 nm were recorded. Lifetimes of these emission bands cannot be measured accurately because of the rapid decomposition of the mercury complex upon flashing the solution with a laser pulse at 355 nm. Nevertheless, the emission at 520–600 nm was found to have a lifetime in the microsecond time domain in degassed dichloromethane at room temperature. Since the vibrational progression of the emission is characteristic of the skeletal vibration frequency

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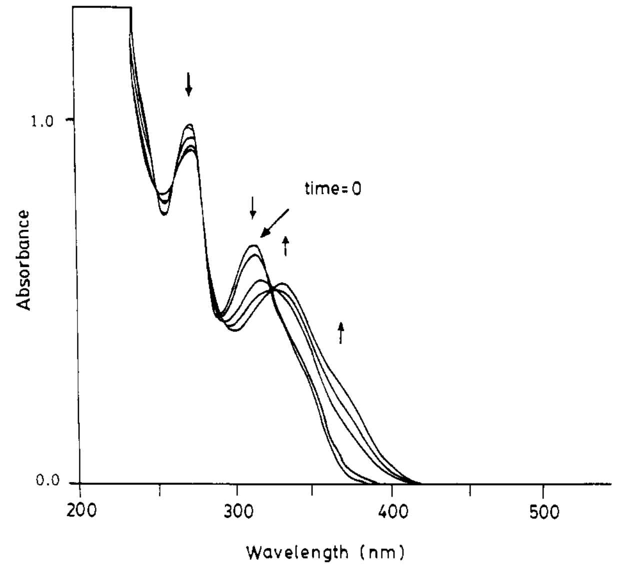

**Figure 4.** UV–vis spectrum of [Hg(C∧N-dpp)Cl]$_2$ in dichloromethane (time = 0 min) and sequential scans at time intervals of 10 min during steady-state irradiation at 324 nm. (Isosbestic points: 324, 283, 275, 260, and 245 nm.)

of the aromatic ring (1400 cm$^{-1}$), an intraligand excited state is suggested for the observed emission. We tentatively assign the emission bands at 350–410 nm and 520–600 nm in Figure 3 to the fluorescence and phosphorescence of the cyclometalated ligand, although some participation of the Hg(II) ion in the excited states cannot be excluded.

Measurement of the emission spectrum of a degassed dichloromethane solution of [Hg(C∧N-dpp)Cl]$_2$ is complicated by the instability of the complex toward photodecomposition. The high-energy emission band was replaced by a featureless low-energy band (500 nm) when a solution of [Hg(C∧N-dpp)Cl]$_2$ was exposed to UV–vis light for more than 1 h. A degassed dichloromethane solution of [Hg(C∧N-dpp)Cl]$_2$ is particularly photosensitive. Steady-state irradiation at 324 nm caused a progressive change of the UV–vis absorption spectrum, where isosbestic points at 324, 283, 275, 260, and 245 nm were recorded (Figure 4). The final UV–vis absorption and emission spectra at the end of irradiation resemble that of protonated dpp (Hdpp$^+$).[13] Addition of a base such as triethylamine to the solution after photolysis gave a UV–visible absorption spectrum essentially identical to that of free dpp.

**General Comments.** Observation of photoluminescence from cyclometalated Hg(II) complexes is not unprecedented in the literature. For the [Hg(L)Cl]$_4$ complex in a 77 K ethanol and toluene glass (HL = 2-phenylpyridine), a strong featureless emission at ~500 nm was observed by Vogler and co-workers.[5] This emission was attributed to come from mixed 5d–6s/LMCT excited state. The conclusion was based on the low $n\mathrm{d}^{10} \rightarrow n\mathrm{d}^9(n+1)\mathrm{s}^1$ promotion energy of mercury and on the absence of phosphorescence from 2-phenylpyridine.[5] However, as described in the previous section, the emission of [Hg(C∧N-dpp)Cl]$_2$ is essentially intraligand in nature. One possible reason for such a difference could be that the $\pi$–$\pi^*$ excited state of dpp is of lower energy than that of 2-phenylpyridine. Examination of the structural data of [Hg(L)Cl]$_4$ also revealed that there are both chloride bridges and a semi-Hg–C–Hg bridge in the tetranuclear complex.[6b] The Hg–C distances are 3.45(1) and 2.07(1) Å. This kind of bridging by metalated carbon does not exist in the dimeric structure of [Hg(C∧N-dpp)Cl]$_2$ and may perhaps account for the lower energy of the 5d–6s/LMCT excited state of [Hg(L)Cl]$_4$.

Previously, we have investigated the luminescent properties of the cyclometalated complexes [Pt(C∧N∧N-ddp)(NCCH$_3$)]$^+$ [2d] and [Au$^{\mathrm{III}}$(C∧N∧N-dpp)Cl]$^+$,[4] which also display vibronic structured emission at 500–600 nm. The emission of the gold(III) complex has been ascribed to come from a metal-perturbed intraligand excited state[4] whereas that of the platinum(II) complex is metal-to-ligand charge transfer in nature.[24] The structural similarities between these two cyclometalated species with that of [Hg(C∧N-dpp)Cl]$_2$ allow comparison of their emission properties and hence the nature of their excited states. In this context, the $E_{00}$ energies (first maxima) for [Hg(C∧N-dpp)Cl]$_2$, [Au$^{\mathrm{III}}$(C∧N∧N-dpp)Cl]$^+$, and [Pt(C∧N∧N-dpp)(NCCH$_3$)]$^+$ are $20.3 \times 10^3$, $18.9 \times 10^3$, and $18.1 \times 10^3$ cm$^{-1}$, respectively. This order in emission energies, Hg(II) > Au(III) > Pt(II), may be correlated with the increasing amount of MLCT character in the lowest excited state. While previous excited-state assignments for cyclometalated complexes are based on comparison of emission spectra recorded on both complex and free ligand, the cyclometalated Hg(II) complex is obviously a better reference point when minimum disturbance of the electronic structure of the cyclometalated ligand is of primary concern.

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**Supplementary Material Available:** Crystal data collection and refinement parameters (Table S1), hydrogen atom coordinates (Table S2), and Figure S1 (2D-NMR of [Hg(C∧N-dpp)Cl]$_2$ in CDCl$_3$) (3 pages). Ordering information is given on any current masthead page.