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Synthesis, characterization and crystal structures of a new 2-ferrocenylnaphthoxazole and its mercurated derivatives

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Abstract

A new ferrocenylnaphthoxazole $[(\eta^5\text{-}C_5H_5)Fe\{(\eta^5\text{-}C_5H_4)C(O)=N(C_{10}H_6)\}]$ (**1**) was synthesized under mild conditions. Two mercurated derivatives: *ortho*-mercurated product $[HgCl\{(\eta^5\text{-}C_5H_5)Fe[(\eta^5\text{-}C_5H_3)C(O)=N(C_{10}H_6)]\}]$ (**2**) and the product mercurated on the unsubstituted Cp ring $[HgCl\{(\eta^5\text{-}C_5H_4)Fe[(\eta^5\text{-}C_5H_4)C(O)=N(C_{10}H_6)]\}]$ (**3**) were obtained by the reaction of **1** with mercuric acetate. All the new compounds **1**, **2** and **3** were characterized by elemental analyses, IR, NMR, MS spectra and X-ray crystal structure analysis. The crystal structure of **1** extended into a 2D supramolecular network through the intermolecular π–π stacking interaction between the Cp ring and naphthoxazole ring. In the crystal of **2,** there exist bridged Cl–Hg bonds, CH(Cp) ⋯ Cl and CH⋯ Hg hydrogen bonds, π–π stacking interactions, which facilitate construction of this complex into a 3D supramolecular structure.

Keywords: Ferrocenylnaphthoxazole; Mercury (II) complexes; Crystal structures; π – π Stacking; Hydrogen bonds

1. Introduction

2-Substituted naphthoxazoles are an important subunit of some natural products [1], biologically active compounds [2], fluorescent probes [3], or intermediates for azo dyes [4]. General method for the synthesis of 2-substituted naphthoxazoles is the coupling of 1-amino-2-naphthol hydrochloride with aromatic aldehyde in the presence of piperidine or morpholine, as a catalyst [4,5]. But this method gave low yields due to the formation of byproduct – piperidylquinone. In this paper, a new 2-ferrocenylnaphthoxazole $[(\eta^5\text{-}C_5H_5)Fe\{(\eta^5\text{-}C_5H_4)C(O)=N(C_{10}H_6)\}]$ **1** was synthesized in moderate yield by condensation of formylferrocene with 1-amino-2-naphthol hydrochloride in the presence of triethylamine in refluxing ethanol. Although there is considerable interest in the chemistry of ferrocenyloxazoline [6], only few studies about the combination of ferrocene and oxazole have been investigated [7]. Considering the wide applications of ferrocene and naphthoxazoles, we studied the complexation properties of **1** with mercury(II) giving both the *ortho*-mercurated product $[HgCl\{(\eta^5\text{-}C_5H_5)Fe[(\eta^5\text{-}C_5H_3)C(O)=N(C_{10}H_6)]\}]$ **2** and the product mercurated on the unsubstituted Cp ring $[HgCl\{(\eta^5\text{-}C_5H_4)Fe[(\eta^5\text{-}C_5H_4)C(O)=N(C_{10}H_6)]\}]$ **3** (Scheme 1). In addition, the crystal structures of **1** and **2** were determined.

2. Results and discussion

2.1. Synthesis, IR and NMR spectroscopy

By condensation of formylferrocene with 1-amino-2-naphthol hydrochloride in the presence of triethylamine in refluxing ethanol, we obtained the product **1** (Scheme 1). The $\upsilon_{C=N}$ at 1640 cm^{-1} in the IR spectrum of **1** is lower than that of ferrocenyloxazoline [8], which may be attributed to the big π–π conjugated system of ferrocene and naphthoxazole. The signal at δ 165.3 ppm in ^{13}C NMR

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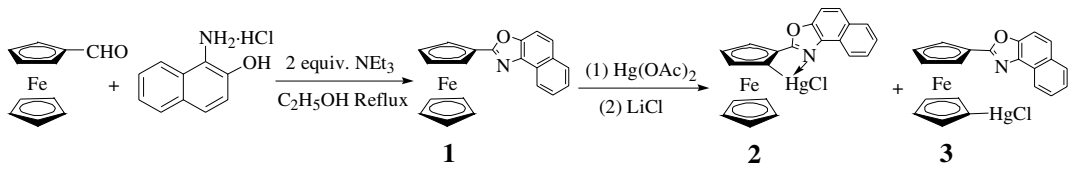

Scheme 1.

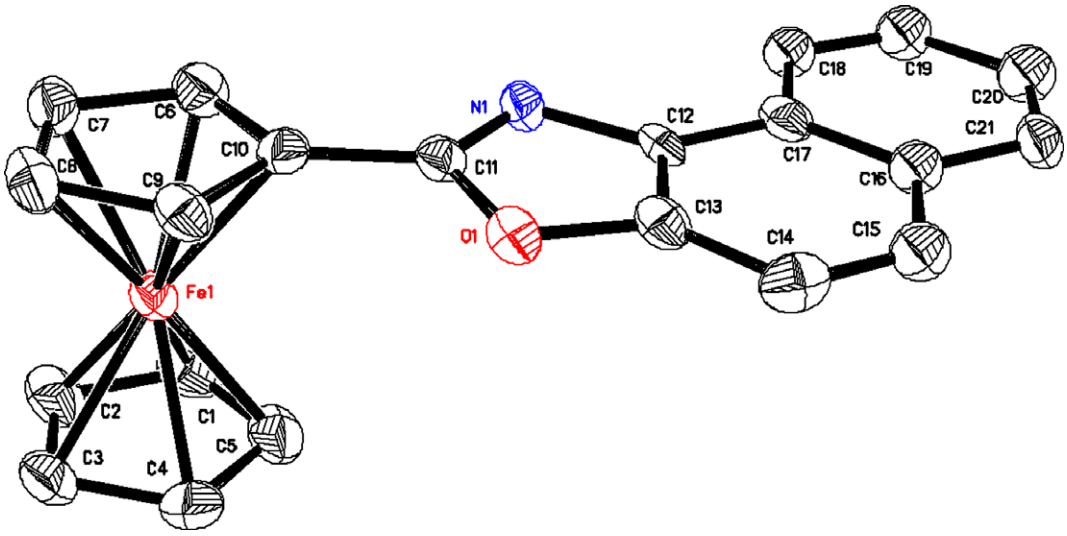

Fig. 1. Molecular structure of complex **1**.

spectrum of **1** further confirmed the formation of imine moiety. Finally, the structure of **1** was unequivocally confirmed by X-ray diffraction analysis (Fig. 1).

Then, we studied the reaction of **1** with $Hg(OAc)_2$ followed by treatment with LiCl according to our previously reported procedure for cyclomercuration of ferrocenylimines [9]. Two red mercurated derivatives: **2** (Yield 16%) and **3** (Yield 17%) were obtained after purification by preparative TLC. Although statistical weights of the positions open to mercuration are different as 2:5, mercuration into *ortho*-position occurs easier than into the unsubstituted Cp-ring due to precoordination N–Hg. So the yields of the mercurated derivatives are almost equal. The strong absorption at 1100 cm^{-1} in IR spectrum of **2** indicated the presence of an unsubstituted Cp-ring [10]. Compared to **1**, the C=N absorption (1633 cm^{-1}) of **2** shifted to lower energy owing to the intramolecular coordination of nitrogen to mercury. This result is well in agreement with those reported on related cyclomercurated mono-ferrocenylimines [11]. ^1H NMR spectrum of **2** exhibited three signals at δ 4.47 ppm, 4.70 ppm and 5.23 ppm for the substituted Cp ring and one signal at δ 4.20 ppm for the unsubstituted Cp ring with the proton ratio of 1:1:1:5, clearly showing that **2** was *ortho*-mercurated product. Moreover, the signal at δ 86.1 ppm in ^{13}C NMR spectrum of **2** shifted to lower field than that of **1** (δ 68.4 ppm) for the formation of carbon–mercury bond [12]. In addition, the structure of the complex **2** has been confirmed by X-ray diffraction study (Fig. 3). In compound **3**, the C=N absorption (1637 cm^{-1}) shifted slightly to higher energy in comparison with **2** (1633 cm^{-1}) because of the absence of the intramo-

Table 1
Crystal and structure refinement data for **1** and **2**

Compound	1	2
Empirical formula	$C_{21}H_{15}FeNO$	$C_{21}H_{14}Cl$ Fe Hg N O
Formula weight	353.19	588.22
Crystal system	Orthorhombic	Monoclinic
Crystal size (mm)	$0.20 \times 0.18 \times 0.17$	$0.20 \times 0.18 \times 0.17$
Space group	$P212121$	$P21c$
a (Å)	9.2889(19)	15.656(3)
b (Å)	11.174(2)	18.055(4)
c (Å)	14.816(3)	14.045(3)
α (°)	90	90
β (°)	90	112.99(3)
γ (°)	90	90
V (Å^3)	1537.8(5)	3654.8(13)
Z	4	8
D_{Calc} (g cm^{-3})	1.526	2.138
μ (MoKα) (mm^{-1})	0.987	9.340
θ Range (°)	2.28–25.50	1.41–25.00
Data/restraints/ parameters	2649/0/193	5914/0/470
Goodness-of-fit on F^2	1.097	1.015
R (F) [$I > 2\sigma$ (I)]	$R_1 = 0.0509$, $wR_2 = 0.1334$	$R_1 = 0.0511$, $wR_2 = 0.1181$
R indices (all data)	$R_1 = 0.0659$, $wR_2 = 0.1423$	$R_1 = 0.0900$, $wR_2 = 0.1399$

Table 2
Selected bond lengths (Å) and angles (°) for **1** and **2**

1			
C(10)–C(11)	1.452(6)	O(1)–C(11)–C(10)	116.6(4)
N(1)–C(11)	1.289(6)	N(1)–C(11)–C(10)	128.4(4)
O(1)–C(11)	1.386(5)	N(1)–C(11)–O(1)	115.0(4)
N(1)–C(12)	1.421(6)	C(11)–N(1)–C(12)	103.7(4)
O(1)–C(13)	1.394(6)	C(11)–O(1)–C(13)	103.7(4)
2			
Hg(1)–Cl(1)	2.313(4)	C(6)–Hg(1)–Cl(1)	175.6(3)
Hg(1′)–Cl(1′)	2.309(4)	C(6′)–Hg(1′)–Cl(1′)	175.9(3)
Hg(1)–C(6)	2.063(12)	C(10)–C(6)–Hg(1)	120.1(9)
Hg(1′)–C(6′)	2.014(12)	C(10′)–C(6′)–Hg(1′)	123.8(9)
C(10)–C(11)	1.404(16)	N(1)–C(11)–C(10)	126.5(10)
C(10′)–C(11′)	1.454(15)	N(1′)–C(11′)–C(10′)	127.0(10)
O(1)–C(11)	1.372(12)	O(1)–C(11)–C(10)	120.9(11)
O(1′)–C(11′)	1.385(12)	O(1′)–C(11′)–C(10′)	117.7(8)
N(1)–C(11)	1.316(15)	N(1)–C(11)–O(1)	112.6(11)
N(1′)–C(11′)	1.269(12)	N(1′)–C(11′)–O(1′)	115.3(10)
N(1)–C(12)	1.405(14)	C(11)–N(1)–C(12)	106.1(10)
N(1′)–C(12′)	1.393(14)	C(11′)–N(1′)–C(12′)	105.5(9)
O(1)–C(13)	1.384(14)	C(11)–O(1)–C(13)	104.9(10)
O(1′)–C(13′)	1.358(15)	C(13′)–O(1′)–C(11′)	102.9(8)

lecular coordination of nitrogen to mercury. In particular, the ^{1}H NMR spectrum is informative for the elucidation of compound **3**. The four signals at δ 4.16 ppm, 4.56 ppm, 4.65 ppm and 5.28 ppm with the proton ratio of 2:2:2:2, are in good consistent with A_2B_2 systems typical of mono-substituted cyclopentadiene rings. The signal at δ 86.9 ppm in ^{13}C NMR spectrum of **3** shifted to lower field than that of **1** (δ 69.9 ppm) due to the formation of carbon–mercury bond, too [12].

2.2. Crystal structures of *1* and *2*

The crystal structures of **1** and **2** were determined, the crystal data and refinement details for both structures are in Table 1. Fig. 1 shows molecular structure of **1** together with atom-labeling scheme. Selected bond lengths and angles are listed in Table 2. The naphthoxazole ring and the Cp ring are approximately coplanar with a dihedral angel of 2.1°. The N1–C11 (1.289(6) Å) and O1–C11 (1.386(5) Å) bond lengths in **1** are a little longer than those

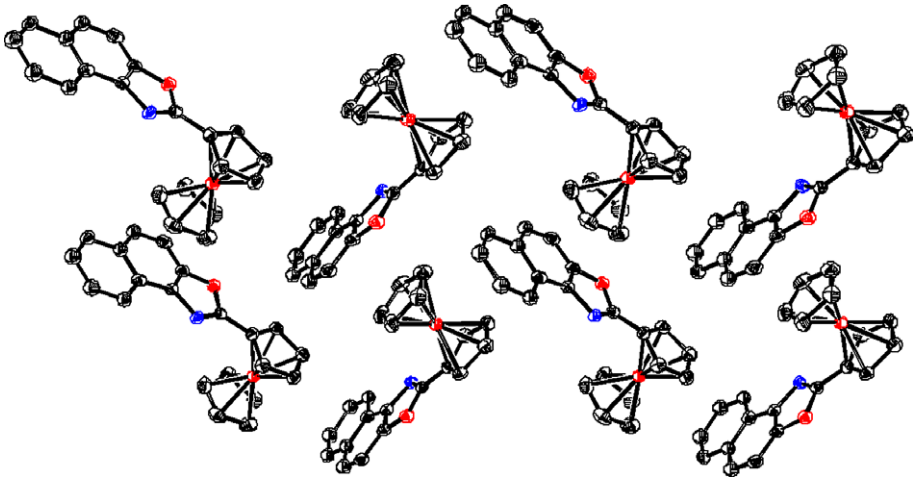

Fig. 2. The π–π interactions in complex **1**.

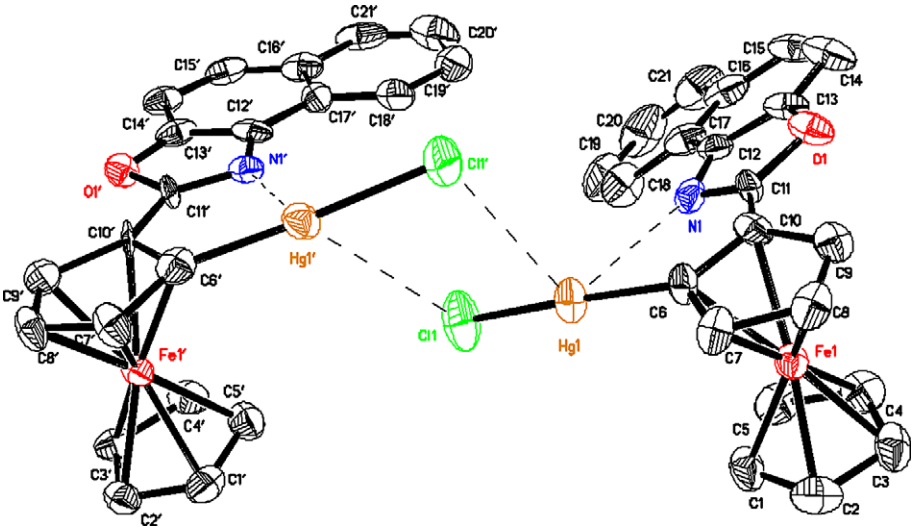

Fig. 3. Molecular structure of complex **2**.

found in the complex with no conjugated naphthoxazole group [N1–C11 = 1.237(5) Å and O1–C11 = 1.354(5) Å respectively] [13]. It can be seen from Fig. 2 that the Cp ring and naphthoxazole ring of neighboring molecules are almost parallel with each other with mean interplanar distance of 3.5363 Å (dihedral angle is 2.7°), indicating strong intermolecular π–π stacking interaction between neighboring molecules.

The crystal structure of **2** reveals two molecules in the asymmetric unit with somewhat difference in geometry (Fig. 3). The N1···Hg1 and N1′···Hg1′ distance are 2.922 Å and 2.939 Å, shorter than the sum of van der Waals radii of Hg, 2.00 Å [14], and N, 1.55 Å, indicating the presence of an intramolecular coordination in the compound. The chelation of mercury(II) with nitrogen atom forms a nearly planar five-membered ring with max-

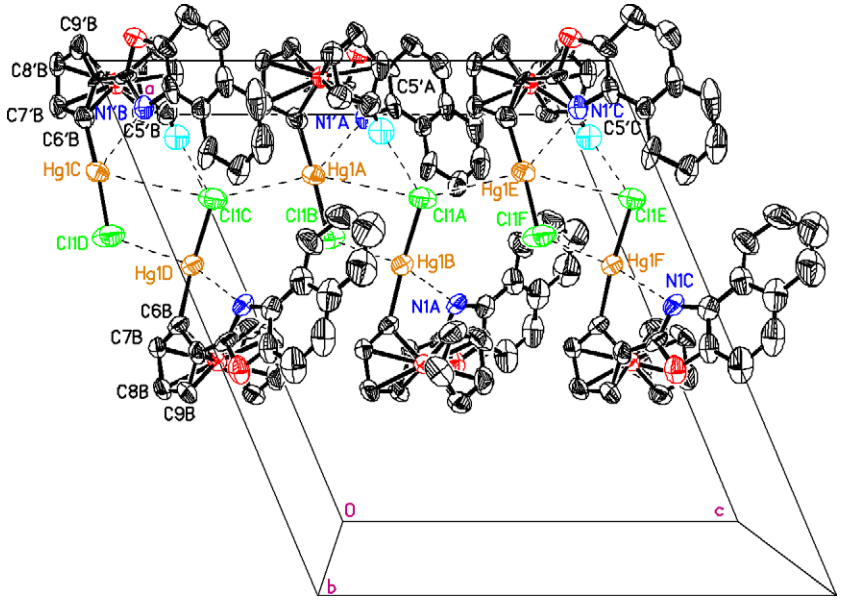

Fig. 4. 1D chain structure of the complex **2** along **c** axis.

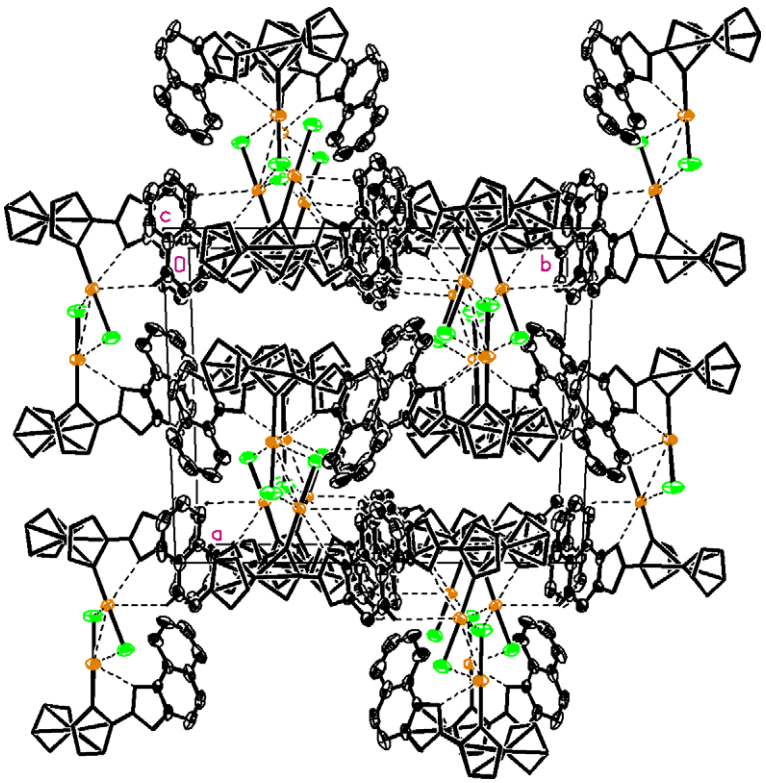

Fig. 5. The 3D network packed from the 1D chain structure through π–π interactions and C–H···Hg hydrogen bonds in complex **2**.

imum deviation of 0.130 Å for C8 atom. There are weak interactions between Hg1 (Hg1′) and Cl1 (Cl1′) atoms of adjacent molecules. Thus, complex **2** can be seen as a dimer bridged by two chlorine atoms (Cl1–Hg1 = 2.313 Å, Cl1–Hg1′ = 3.939 Å, Cl1′–Hg1′ = 2.309 Å, Cl1′–Hg1 = 3.245 Å). In compound **2** the dihedral angle between two Cp rings are 2.4°(Fe) and 2.8°(Fe′), while the dihedral angle between naphthoxazole ring and the Cp ring are 21.7°(Fe) and 8.1°(Fe′).

Fig. 4 shows the one dimensional chain structure of complex **2** along *c* axis, which results from the fact that the dimer is connected to each other through the weak Hg–Cl interactions (Hg1′–Cl1A = 3.522 Å). In the crystal there is a intermolecular hydrogen bond between the chlorine atom and the adjacent C–H group of Cp ring (Cl1–H5′ = 2.821 Å). The naphthoxazole rings in adjacent one dimensional columns are parallel with each other and partially overlapped (dihedral angle is 0°), indicating strong intermolecular π–π stacking interactions with the interplane distances of 3.5342 Å and 3.3169 Å respectively. Furthermore, there is an interesting C–H···M hydrogen bond (Hg1′···H15′A = 3.167 Å) [15] in the crystal structure of **2**. Under such circumstance, the π–π stacking interactions of naphthoxazole rings and directional C–H···M hydrogen bonds between adjacent 1D chains facilitated the assembly of these columns into a three dimensional structure. This structure feature is more evident in Fig. 5, extending of 1D chanis in **ab** plane by means of π–π stacking interactions and Van der Waals force constitutes infinite 3D supermolecular network.

3. Conclusion

In the presence of triethylamine, we have prepared successfully a new ferrocenylnaphthoxazole **1** and its cyclomercury(II) derivatives **2** and **3**. The crystal structure of **1** was stabilized by the intermolecular π–π stacking interactions between the Cp ring and naphthoxazole ring. In the crystal structure of **2**, there exist Cl–Hg bonds, CH(Cp)···Cl and CH···Hg hydrogen bonds, π–π stacking interactions, which are attributed to the construction of 3D network structure of complex **2.**

4. Experimental

4.1. General techniques

Melting points were measured with the use of a WC-1 microscopic apparatus. Elemental analyses were determined with a Carlo Erba 1160 elemental analyzer. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker DPX-400 spectrometer in $CDCl_3$ with TMS as an internal standard. IR spectra were recorded on a Bruker VECTOR 22 spectrophotometer. Mass spectra were obtained on a Bruker Esquire 3000 ESI ion trap mass spectrometer. Preparative TLC was performed on dry silica gel plates developed with selected appropriate solvents as eluents. Solvents were purified and dried by standard techniques, and distilled before use. Formylferrence [16] and 1-amino-2-naphthol hydrochloride [17] were prepared according to previously reported procedures. All other starting materials were received from commercial sources.

4.2. Synthesis and characteristic data of 2-ferrocenylnaphthoxazole (*1*)

A mixture of 1-amino-2-naphthol hydrochloride (980 mg, 5 mmol), formylferrence (107 mg, 5 mmol), and triethylamine (1.5 mL, 10 mmol) in 20 mL of ethanol was refluxed until the carbonyl absorption of formylferrence disappeared according to IR spectrum. After cooling to room temperature, the solution was concentrated *in vacuo*, and the residue was purified by column chromatography eluting with ethyl acetate/petroleum ether(1:20) to yield 1320 mg (75%) of the oxazole **1** as a red solid. m.p. 229–230 °C. Anal. Calc. for $C_{21}H_{15}FeNO$: C, 71.41, H 4.28, N 3.97; Found: C, 71.27, H 4.16, N 3.73%. IR (KBr): υ_{max} 1640 (C=N), 1594, 1437, 1372, 1272, 1237, 1103, 1002, 927, 807, 754, 489 cm^{-1}. ^{1}H NMR(400 MHz, $CDCl_3$): δ 4.20(s, 5H, Fc-unsubst.), 4.53(bs, 2H, Fc), 5.16(bs, 2H, Fc), 7.51–7.55(m, 1H, Ar–H), 7.63–7.67(m, 1H, Ar–H), 7.69(d, $J = 8.8$ Hz, 1H, Ar–H), 7.77(d, $J = 8.8$ Hz, 1H, Ar–H), 7.96(d, $J = 8.2$ Hz, 1H, Ar–H), 8.53(d, $J = 8.2$ Hz, 1H, Ar–H) ppm. ^{13}C NMR(100 MHz, $CDCl_3$): δ 68.4(CH), 69.9(CH, Fc-unsubst.), 70.5(C), 78.8(CH), 110.7(CH), 122.4(CH), 124.9(CH), 125.2(CH), 126.1(C), 126.7(CH), 128.5(CH), 131.2(C), 137.7(C), 147.6(C), 165.3(C=N) ppm. MS (ESI): $m/z = 353.6$ $[M+H]^+$.

4.3. Synthesis and characteristic data of mercury (II) complexes *2* and *3*

Compound **1** (127 mg, 0.4 mmol) was dissolved in 5 mL of dichloromethane, to which $Hg(OAc)_2$ (141 mg, 0.4 mmol) in 10 mL methanol was added dropwise over a period of 30 min at room temperature. The resulting mixture was stirred for 4 h. Then 0.8 mmol of LiCl in 5 mL methanol was added dropwise, and stirred for another 30 min. The reaction was quenched with water and extracted with dichloromethane. The organic layer was washed with brine, dried over $MgSO_4$, filtered and concentrated under reduced pressure. The crude product was purified by preparative TLC eluting with ethyl acetate/petroleum ether (1:8) to give two red solids: **2** (36 mg, 16%) and **3** (40 mg, 17%). The characteristic data of **2**: m.p. 227–228 °C. Anal. Calc. for $C_{21}H_{14}ClFeHgNO$: C, 42.88; H, 2.40; N, 2.38; Found: C, 43.25; H, 2.37; N, 2.25%. IR (KBr): υ_{max} 1633 (C=N), 1583, 1412, 1375, 1273, 1240, 1129, 1101, 1001, 950, 799, 734, 492 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.19(s, 5 H, Fc-unsubst.) 4.45(bs, 1H, Fc), 4.69(bs, 1H, Fc), 5.21(bs, 1H, Fc), 7.51–7.54(m, 1H, Ar–H), 7.63(d, $J = 8.9$ Hz, 1H, Ar–H), 7.66–7.69(m, 1H, Ar–H), 7.74(d, $J = 8.9$ Hz, 1H, Ar–H), 7.92(d, $J = 8.2$ Hz, 1H, Ar–H), 8.50(d, $J = 8.2$ Hz, 1H,

Ar–H) ppm. ^{13}C NMR(100 MHz, $CDCl_3$): δ 69.3(CH), 70.7(CH, Fc-unsubst.), 73.8(CH), 74.6(C), 76.4(CH), 86.1(C–HgCl), 111.0(CH), 123.2(CH), 125.6(CH), 126.0(CH), 126.2(C), 127.6(CH), 128.8(CH), 131.7(C), 137.3(C), 148.6(C), 167.2(C = N) ppm. MS (ESI): m/z = 589.6 $[M+H]^+$.

The characteristic data of **3**: m.p. 209–211 °C. Anal. Calc. for $C_{21}H_{14}ClFeHgNO$: C, 42.88; H, 2.40; N, 2.38; Found: C, 43.26; H, 2.38; N, 2.30%. IR (KBr): υ_{max} 1637(C = N), 1595, 1440, 1376, 1281, 1242, 1108, 1007, 933, 803, 735, 487 cm^{-1}. ^{1}H NMR(400 MHz, $CDCl_3$): δ 4.07(bs, 2H, Fc), 4.47(bs, 2H, Fc), 4.56(bs, 2H, Fc), 5.19(bs, 2H, Fc), 7.50–7.54(m, 1H, Ar–H), 7.64–7.68(m, 1H, Ar–H), 7.71(d, J = 8.8 Hz, 1H, Ar–H), 7.78(d, J = 8.8 Hz, 1H, Ar–H), 7.94(d, J = 8.2 Hz, 1H, Ar–H), 8.64(d, J = 8.2 Hz, 1H, Ar–H) ppm. ^{13}C NMR(100 MHz, $CDCl_3$): δ 69.0(CH), 70.9(C), 71.1(CH), 71.3(CH), 75.4(CH), 86.9(C–HgCl), 110.7(CH), 122.8 (CH), 125.3(CH), 125.3(CH), 126.2(C), 126.7(CH), 128.3(CH), 131.3(C), 137.7(C), 147.5(C), 164.5(C = N) ppm. MS (ESI): m/z = 589.5 $[M+H]^+$.

4.4. X-ray structure determination

Intensity data of the complexes **1** and **2** were measured on a Rigaku-Raxis-IV X-ray diffractometer using graphite-monochromated MoKα radiation (λ = 0.71073 Å) at 291(2) K. The data were corrected for Lorebtz and polarization factors. The structure was solved by direct methods [18] and expanded using Fourier techniques and refined on $F^2$by full-matrix least-squares methods using the SHELXTL-97 program package. All calculations were performed using the teXan[19] crystallographic software package of Molecular Structure corporation. Non-hydrogen atoms were refined anisotropically, the hydrogen atoms were refined isotropically using a riding model. Crystallographic data and details about data collection and refinement are listed in Table 1.

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Appendix A. Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 288931 for **1** and No. 294685 for **2**. These data can be obtained free of charge via www.ccdc.cam. ac.uk/conts/retreiving.html (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; Fax: + 44 1223336033; or deposit@ccdc.cam.ac.uk). Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2006.10.070.

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