Journal of Molecular Structure 1018 (2012) 185–189

Contents lists available at SciVerse ScienceDirect

Journal of Molecular Structure

journal homepage: www.elsevier.com/locate/molstruc

# Synthesis, characterization and properties of copper(I) complexes with bis(diphenylphosphino)-ferrocene ancillary ligand

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ARTICLE INFO

*Article history:*
Received 27 January 2012
Received in revised form 3 March 2012
Accepted 7 March 2012
Available online 16 March 2012

*Keywords:*
Acetanilide
Acetate-bridged
Bis(diphenylphosphino)-ferrocene
Copper(I) amidate complex

ABSTRACT

Three copper(I) complexes (**2–4**) containing dppf ancillary ligand (dppf = bis(diphenylphosphino)-ferrocene) were synthesized when chloride-bridged copper(I) complex **1** reacted with acetanilide and characterized by IR, element analysis and NMR spectrum. And the crystal structures of complexes **2** and **4** have been determined by X-ray diffraction method. Complex **2**, an acetate-bridged copper(I) complex, was obtained under $N_2$ atmosphere in un-dried solvent; the acetate ion came from the hydrolysis reaction of acetanilide due to residual water in solvent. Acetanilide was deprotonated and coordinated with the copper(I) centre to form a copper(I) amidate complex **3** when reacted in pre-dried solvent. In addition, a known complex **4**, the oxidation product of dppf, was isolated from the same reaction system when reacted in air atmosphere. CV and TG experiments were carried out to check the electron transfer properties and thermal stabilities of complexes **2–3.** Finally, the arylation reaction of complex **3** with iodobenzene was performed to study the reaction mechanism of copper(I) catalyzed Goldberg reaction.

## 1. Introduction

Much efforts have been devoted to the design and synthesis of copper(I) complexes because the raw materials are easily available and environmentally friendly compared to expensive precious metals [1–4]. Most importantly, copper(I) salts and their complexes are widely used as catalysts for various organic reactions such as Ullmann reaction, Goldberg reaction and Sandmeyer reaction [5–9]. Furthermore, some well-defined copper(I) complexes have been isolated as reaction intermediates to study the reaction mechanisms of copper(I) catalyzed organic reactions [10–12]. For example, copper(I) amidate complexes have been determined as intermediates of Goldberg reaction by kinetic and spectroscopic studies, theoretical calculation and synthetic works (Scheme 1) [13,11,6,14,15]. However, the preparation and isolation of copper(I) complexes with a terminal amidate ligand is rare and only three examples are reported [13,11,6]. This is partly because strong base is needed to deprotonate the amidate ligand while the resulting copper(I) amidate complex is easily to break down under extreme alkaline conditions. Furthermore, among few reported arylation reactions between copper amidate complexes and aryl halides, the ancillary ligands and amides are limited to chelating *N*,*N*-donor ligands, sometimes mono-phosphine ligand, and cycloamides. More general acyclic amides are rarely reported.

On the other hand, copper(I) is a soft Lewis acid and preferably coordinates with "soft" atoms such as sulphur and phosphorus. The bidentate ligand, bis(diphenylphosphino)-ferrocene (abbr. dppf), has been reported to be a good ancillary ligand for copper(I) ion, since its "soft" phosphorus atom and chelating ability can provide additional stability to the resulting copper(I) complexes [16–18]. And the chloride-bridged copper(I) complex from the reaction of CuCl and dppf ligand has been repeatedly used as reaction precursor due to suitable stability under alkaline conditions and good solubility in organic solvent.

In this paper, the reaction between chloride-bridged copper(I) precursor and acetanilide was studied to obtain the desired copper(I) amidate complex, and to determine the reaction mechanism of copper(I) catalyzed Goldberg reaction via the arylation reaction between the copper(I) amidate intermediate and iodobenzene. Interestingly, different products (**2**–**4**) were prepared under different conditions (Scheme 2). Firstly, a acetate-bridged copper(I) complex **2** was obtained due to the hydrolysis reaction of acetanilide in the presence of residual water in un-dried solvent. Secondly, a copper(I) amidate complex **3** was prepared when reaction solvent was pre-dried, where acetanilide was deprotonated and coordinated with the copper(I) centre. Finally, a known complex **4**, the oxidation product of dppf was isolated from the same reaction system in air atmosphere. The versatile products show that it is very important to control the reaction conditions of organometallic reactions; minor differences even a little water or air may result in wide divergence. Herein, the detailed synthesis and characterization of

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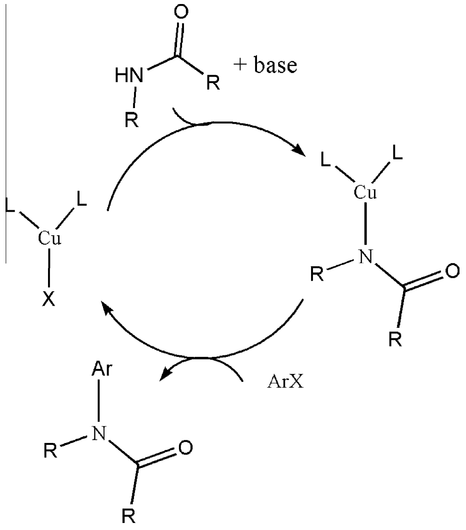
http://dx.doi.org/10.1016/j.molstruc.2012.03.020



**Scheme 1.** The copper catalyzed Goldberg reaction.

complexes **2–4** have been reported. CV and TG experiments were performed to check the electron transfer properties and thermal stabilities of complexes **2–3**. Finally, the arylation reaction of complex **3** with iodobenzene was studied to prove the catalytic cycle of Goldberg reaction shown in Scheme 1.

## 2. Experimental section

### 2.1. Materials and methods

CuCl, acetanilide, dppf and other chemicals were obtained from commercial sources and used without further purification. Dichloromethane was used directly without dried or pre-dried by common method and freshly distilled prior to use.

IR spectrum was recorded on a FTLA2000 spectrometer using KBr pellets. NMR spectrum was collected on a Bruker ACF-400 spectrometer. $^1$H NMR and $^{13}$C chemical shifts are recorded with deuterated chloroform as solvent and tetramethylsilane as internal standard. $^{31}$P NMR chemical shifts are recorded with deuterated chloroform as solvent and 85% $H_3PO_4$ as external standard. GC–MS analysis was performed on a Finnigan TRACE GC–MS equipped with a PEG 20,000 column (0.5 mm i.d. × 25 m). C, H, N analyses were done using a Perkin–Elmer 2400 Series II CHNS/O analyzer. TG curve was obtained with an Exstar 6000 analyzer in nitrogen at a heating rate of 15 °C/min from 25 to 600 °C. Cyclic voltammetry was performed on an IM6e electrochemical workstation (ZAHNER elektrik, Germany) at room temperature with a conventional three-electrode system consisting of a Pt-disc working electrode, a Pt-wire counter electrode and an Ag/AgCl reference electrode in $CH_3CN$ solution ($10^{-4}$ mol/L) containing 0.100 mol/L $[Bu_4N][PF_6]$ as the supporting electrolyte. X-ray data for complex **2** were collected at 223 K using a Bruker SMART APEX II CCD area detector diffractometer (Mo Ka, $\lambda = 0.71073$ Å). The crystal structures were solved using the SHELXTL program [19] and refined using full matrix least squares [20]. The hydrogen atom positions were calculated theoretically and included in the final cycles of refinement in a riding model along with attached carbon atoms. The crystal data and structural determination summary for complex **2** was shown in Table 1.

**Table 1**
Crystal data and structural determination summary for complex **2**.

| | Complex **2** |
|---|---|
| Formula | $C_{72}H_{62}C_{l0}Cu_2Fe_2O_4P_4$ |
| Formula weight (g/mol) | 1353.88 |
| Crystal system | Triclinic |
| Space group | p-1 |
| $a$ (Å) | 10.197(2) |
| $b$ (Å) | 11.587(2) |
| $c$ (Å) | 13.815(3) |
| $\alpha$ (°) | 102.28(3) |
| $\beta$ (°) | 99.69(3) |
| $\gamma$ (°) | 106.41(3) |
| $V$ (Å$^3$) | 1483.8(5) |
| $Z$ | 1 |
| Dc (g/cm$^3$) | 1.515 |
| $\mu$ (mm$^{-1}$) | 1.346 |
| $F$(000) | 696 |
| Reflections collected | 14,922 |
| Independent reflections | 6666 |
| Rint | 0.0572 |
| Parameters | 380 |
| GOF | 1.114 |
| $R_1$ ($I > 2\sigma(I)$) | 0.0776 |
| $wR_2$ (all data) | 0.1714 |
| $(\Delta\rho)_{max}$, $(\Delta\rho)_{min}$ (e/Å$^3$) | 1.336, −0.684 |

### 2.2. Synthesis of complexes 2–4

The chloride-bridged complex $[Cu_2(\mu\text{-}Cl)_2(dppf)_2]$ **1** was obtained by reaction of dppf with CuCl in tetrahydrofuran according to the published procedure (Scheme 2) [17].

#### 2.2.1. Complex 2

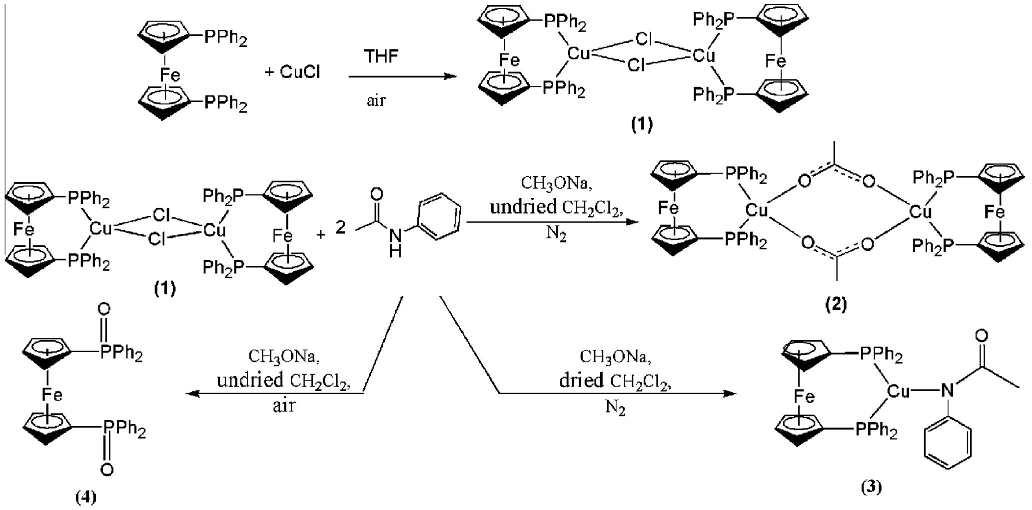
Complex **1** (0.131 g, 0.100 mmol), slightly excess of acetanilide (0.0340 g, 0.250 mmol) and excessive sodium methoxide (0.0540 g, 1.00 mmol) were added in a Schlenk flask, then dichlormethane (20.0 mL) was added under nitrogen atmosphere. The mixture was stirred at room temperature for 24 h. The solution was filtered over Celite to remove resulting sodium chloride and the solvent was removed by rotary evaporator at room temperature. The yellow residue was dissolved easily in diethyl ether and block crystals were obtained after 10 h in 92.0% yield. Anal. calcd. for $C_{36}H_{31}CuFeO_2P_2$: C, 63.87; H, 4.62. Found: C, 63.65; H, 4.65%. **IR (υ, cm$^{-1}$)**: 1731(w), 1679(w), 1594(vs), 1568(s), 1434(s), 1405(m), 1164(m), 1097(m), 1028(m), 813(w), 743(s), 697(vs) (Fig. S1). **$^1$H NMR (400 MHz, $CDCl_3$)**: $\delta$ 7.70–7.65 (m, 7H, Ph), 7.65 (s, 1H), 7.39–7.33 (m, 12H, Ph), 4.31 (s, 4H, Cp), 4.19 (s, 4H, Cp), 2.12 (s, 3H, $CH_3$) (Fig. S2). **$^{13}$C NMR (101 MHz, $CDCl_3$)**: $\delta$ 179.54(C=O), 134.04–128.62 (Ph), 73.82–65.85 (Cp), 23.18($CH_3$) (Fig. S3). **$^{31}$P NMR (162 MHz, $CDCl_3$)**: $\delta$ −17.34 (Fig. S4).

#### 2.2.2. Complex 3

The synthesis of complex **3** was generally the same with that of complex **2**, except that dichlormethane was pre-dried prior to use. Complex **3** was crystallized from diethyl ether as yellow block crystals in 82.0% yield. Anal. calcd. for $C_{42}H_{36}CuFeONP_2$: C, 67.07; H, 4.82; N, 1.86. Found: C, 66.82; H, 4.86; N, 1.89%. **IR (υ, cm$^{-1}$)**: 1686(m), 1599(m), 1553(m), 1499(m), 1481(m), 1435(s),1368(m), 1315(m), 1262(w), 1165(m), 1096(m), 1024(m), 838(w), 746(s), 695(s) (Fig. S5). **$^1$H NMR (400 MHz, $CDCl_3$)**: $\delta$ 7.86–7.30 (m, 10H, Ph), 7.14 (s, 10H, Ph), 7.00–6.87 (m, 5H, Ph), 4.37–4.35 (m, 4H, Cp), 4.11 (s, 4H, Cp), 2.06 (s, 3H, $CH_3$) (Fig. S6). **$^{31}$P NMR (162 MHz, $CDCl_3$)**: $\delta$ −12.18 (Fig. S7). ESI MS (+): 617 $[Cu(dppf)]^+$ (Fig. S8).

#### 2.2.3. Complex 4

Complex **1** (0.131 g, 0.100 mmol), acetanilide (0.0340 g, 0.250 mmol), sodium methylate (0.0540 g, 1.00 mmol) and 20.0 mL dichlormethane were added in a 100 mL round-bottomed flask; the mixture was stirred at room temperature in air for 24 h.



**Scheme 2.** The versatile reaction products of chloride-bridged complex 1 with acetanilide under different conditions.

After the same post-treatment with that of complex **2**, complex **4** was crystallized from diethyl ether in 78.0% yield. The characterization of elementary analysis and X-ray crystallography showed it to be $Ph_2P(O)C_5H_4FeC_5H_4P(O)PPh_2$ ($dppfO_2$). Similar oxidation reactions of dppf to $dppfO_2$ catalyzed by $H_2O_2$ or palladium have been reported earlier [21–23].

## 3. Results and discussion

### 3.1. Synthesis and characterization

Treatment of halide-bridged complex **1** with 2.0 equiv acetanilide in dichloromethane in the presence of sodium methoxide at room temperature afforded complexes **2–4** when the reaction conditions were consciously or unconsciously different.

### 3.1.1. Complex 2

Complex **2** was formed in nitrogen atmosphere when dichloromethane was not pre-dried prior to use. We presume that the acetate bridges come from the hydrolysis reaction of acetanilide in the presence of water introduced by solvent molecule. Our speculation was proven to be correct by GC–MS because the presence of by-product aniline was determined in the reaction system and shown in Fig. S9 (the retention time is 9.98 min and the corresponding m/z is 93.1). The IR spectrum of complex **2** exhibits a strong broad band at 1594 $cm^{-1}$, which is attributed to the stretch vibration of carboxyl and is comparable with that found at 1567 or 1561 $cm^{-1}$ for the reported acetate-bridged complexes [24]. The NMR spectrums of **2** in $CDCl_3$ show the signals of the $CH_3COO$ protons at $\delta = 2.12$ ppm (Fig. S2) and signals at $\delta = 179.54$ and 23.18 ppm of the COO and $CH_3COO$ carbons (Fig. S3) in the $^1H$ and $^{13}C$ NMR spectrum, respectively.

### 3.1.2. Complex 3

Weak acidic acetanilide was deprotonated by strong base in pre-dried dichloromethane and coordinated with copper(I) centre to form complex **3**, which was crystallized from diethyl ether as yellow block crystals. Unfortunately, crystal **3** was easily efflorescent in air in less than 1 min, so the crystal structure of complex **3** was difficult to be determined by X-ray diffraction method. The IR spectrum of **3** exhibits a strong broad band at 1594 $cm^{-1}$, which is attributed to the stretch vibration of carbonyl group. The $^1H$ NMR spectrum of **3** in $CDCl_3$ shows the signal of methyl protons at $\delta = 2.06$ ppm (Fig. S6). The proton position of methyl group and the total proton numbers were different from those in complex **2**. Furthermore, Complex **3** was identified by mass spectrometry by comparison of its m/z value (ESI MS (+): 617, Fig. S8) with that of the complex containing $[Cu(dppf)]^+$ fragment [25].

### 3.1.3. Complex 4

When complex **1** reacted with acetanilide in air atmosphere, it broke down and dppf ligand was oxidized to its pentavalent phosphine compound. Complex **4** was also crystallized from diethyl ether. However, the crystal structure has been reported by previous researchers [26].

### 3.2. Crystal structure of complex 2

The crystal structure of complex **2** was determined by X-ray diffraction and illustrated in Fig. 1; selected bond distances and angles were displayed in Table 2. In Fig. 1, two Cu(I) centres are doubly bridged by two acetate ions giving a dinuclear unit in which the Cu–Cu distance is 3.904(2) Å. The dimer is symmetrical with the same coordination environments for the two Cu(I) ions; each Cu(I) ion is coordinated by two phosphorus atoms (P1, P2) from

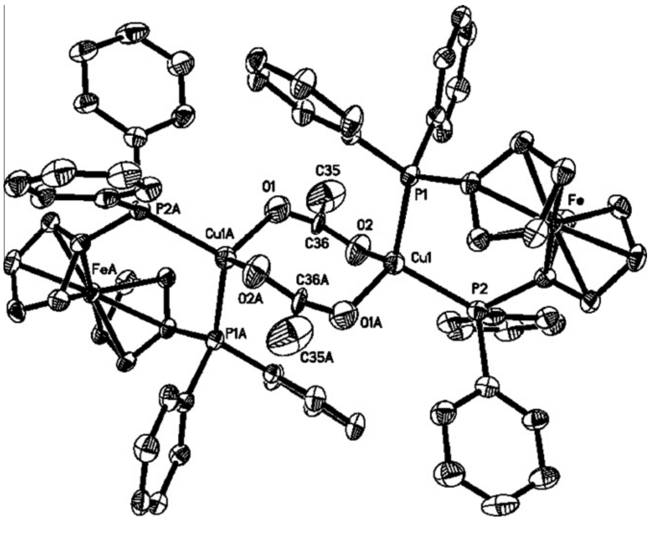


**Fig. 1.** ORTEP representation of the structure of complex **2**. Thermal ellipsoids are drawn at the 30% probability level. Hydrogen atoms are omitted for clarity.

**Table 2**
Selected bond lengths (Å) and bond angles (°) of complex **2**.

| Bond distances | (Å) | Bond angles | ° |
|---|---|---|---|
| Cu(1)–(O1A) | 2.068(5) | O(2)–Cu(1)–O(1A) | 114.7(2) |
| Cu(1)–(O2) | 2.054(4) | O(2)–Cu(1)–P(1) | 111.2(2) |
| Cu(1)–(P1) | 2.263(2) | O(1A)–Cu(1)–P(1) | 106.1(2) |
| Cu(1)–(P2) | 2.274(2) | O(2)–Cu(1)–P(2) | 104.8(1) |
| C(36)–O(1A) | 1.153(7) | O(1A)–Cu(1)–P(2) | 108.0(2) |
| C(36)–O(2) | 1.174(7) | P(1)–Cu(1)–P(2) | 112.1(7) |

Symmetry codes for **2**: *A* = −*x* + 1, −*y* + 1, −*z* + 1.

one dppf ligand and two oxygen atoms (O1A, O2) from two carboxyl bridges in a distorted tetrahedron arrangement. The Cu–P bond lengths are 2.263(2) and 2.274(2) Å, which are comparable to the reported copper(I) complexes with dppf, dppb, dtpb and other bidentate phosphine ligands [27,28]. The bond distances of Cu1–O bonds are 2.054(4) and 2.068(5) Å, which are within the range of those found in acetate-bridged copper complexes [29].

Nee and coworkers [30] have reported a formate-bridged copper(I) complex (**4**) with dppf ligand. The structures of **2** and **4** are almost identical except the bridged carboxyl groups. However, the Cu–Cu distance of **4** (4.561(1) Å) is much longer than that of **2** (3.904(2) Å). The phenomenon may be related to the partial double bond feature of C36–O from the electron resonance between C36–O1A and C36–O2 bonds. The electron-donating methyl group increases the number of electrons of the delocalized π bond, and thus the bond distance of C36–O in **2** (1.153(8), 1.174(9) Å) are shorter than those of complex **4** (1.211(7), 1.233(7) Å). The coordination of the O atoms in **2** with the copper(I) centres are also enhanced due to the electron-rich delocalized π bond. All these results are consistent with the short Cu–Cu distance in complex **2** compared with that of the formate-bridged complex **4**.

There are multiple aromatic rings in the dppf ligand, so weak C–H···π interactions are an important factor in the crystal packing of complex **2** (Fig. S10). The dinuclear copper(I) units are linked into 1D wavy double chains by C30–H30···C4 (i, 3.646(2) Å, blue dotted line) in the *b* direction, which are linked by C8–H8···C4 (ii, 3.189(9) Å, pink dotted line) in the *c* direction into a 2D layer structure. The 2D planes in neighbouring layers are further linked by C35–H35B···C25 (iii, 2.997(9) Å, orange dotted line) in the *a* direction into the 3D supramolecular framework. According to the classification of C–H···π interactions by Suezawa [31], the third interaction (iii, ($D_{\text{pln}}$ = 2.997(9) Å < $D_{\max}$, $\theta$ = 36.98(6)° < 60°) is located in "region 1" where H is above the aromatic ring; the first two interactions are located in "region 2" ($D_{\text{lin}}$ = 2.883(2) Å < $D_{\max}$, $\theta$ = 35.62(3)° < 60°, $\omega$ = 120.7(2)° < 130° for i; $D_{\text{lin}}$ = 2.878(1) Å < $D_{\max}$, $\theta$ = 34.91(3)° < 60°, $\omega$ = 93.69(2)° < 130° for ii) where H is out of region **1** but may interact with π-orbital.

### 3.3. Thermal property

In order to determine the thermal stabilities of complexes **2** and **3**, TG experiments were carried out and the thermal curves were shown in Fig. 2. Complex **2** shows a weight loss of 8.21% (calcd. 8.72%) in the range of 76–256 °C, which is attributed to release of the acetate bridges. Complex **2** continues to decompose up to 549 °C and the remainders are copper oxide and ferric oxide, who account for 23.34% (calcd. 23.54%). In contrast, complex **3** is thermally stable from room temperature to 240 °C, and then complex **3** gradually decomposes and finally decomposes to copper oxide and ferric oxide (21.79%, calcd. 21.19%) at 550 °C. In conclusion, complex **3** has better thermal stability than the acetate-bridged complex **2**.

### 3.4. Electrochemical properties

In order to study the electron transfer properties of complexes **2–3** and dppf ligand, CV experiments were carried out and shown in Fig. 3. The CV curves of dppf, complexes **2** and **3** similarly display one major oxidation and reduction peaks (assigned as $I^{ox}$, $I^{red}$; $I^{ox2}$, $I^{red2}$ and $I^{ox3}$, $I^{red3}$ in Fig. 3) with the Em-values of −0.80, −0.87 and −0.82 V. The oxidation process is assigned to the oxidation of the ferrocene centre from Fe$^{II}$ to Fe$^{III}$, by analogy with data for other Cu(I) complex with dppf ligand [17]. Compared to the free ligand, the Em-values of complexes **2–3** are shifted to more negative potentials. This shift is due to the back-donation effect between copper and phosphorus atom resulting in the increase in the electron density of the ligand [17]. Unfortunately, the redox process of the copper centres are not observed, which is proved by previous literature because the oxidation of the Cu(I) centres usually lies outside the potential range of the solvent window [16]. As described in previous literature, the CV experiments show the oxidation of copper centres is posterior to the ferrocene units [16], so the copper centres are hard to be oxidized and complexes **2–3** are stable in air even for months.

### 3.5. Reactivity of complex 3

Arylation reaction of complex **3** with iodobenzene (see Scheme 3) was performed in DMF under nitrogen atmosphere for 24 h to determine the reaction mechanism of copper(I) catalyzed Goldberg reaction. *N*,*N*-diphenylacetamide was obtained in 75% yield and determined by $^1$H NMR spectrum {$^1$H NMR (400 MHz, DMSO):

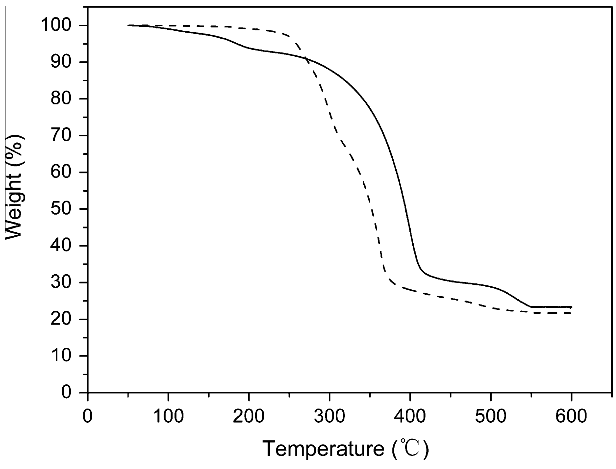


**Fig. 2.** TG curves of complexes **2** (solid line) and **3** (dotted line).

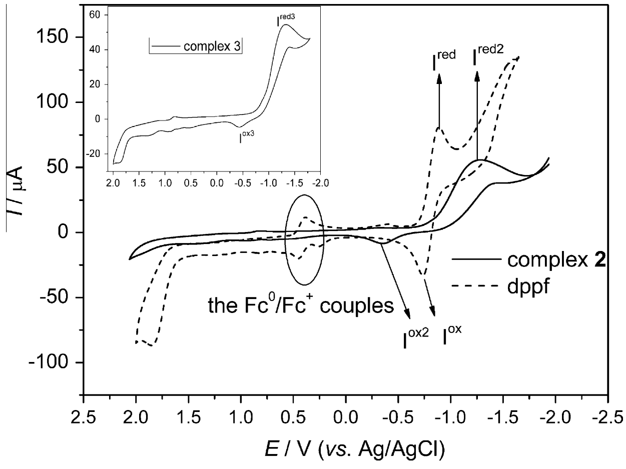
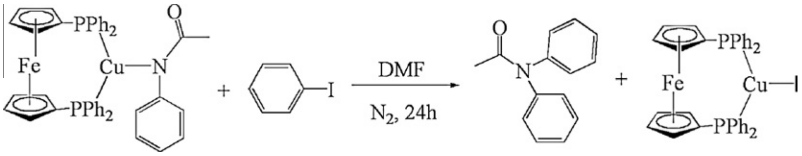


**Fig. 3.** CV curves of complexes **2–3** and dppf ligand.



**Scheme 3.** *N*-arylation reaction between complex **3** and iodobenzene.

$\delta$ = 2.41(s, 3H), 7.00–7.35(m, 6H), 7.75(d, J = 7.2 Hz, 4H)}. Meanwhile a copper(I) complex Cu(dppf)I was formed and determined by LC–MS analysis {ESI MS (+): 617 [Cu(dppf)]$^+$}. The reactivity study of complex **3** and iodobenzene proved the catalytic cycle of Goldberg reaction shown in Scheme 1.

## 4. Conclusions

In summary, different products (complexes **2–4**) were obtained by reacting of chloride-bridged complex **1** with acetanilide under different conditions. The acetate-bridged complex **2** was the reasonable product of hydrolysis reaction of acetanilide due to residual water in un-dried solvent. The copper(I) amidate complex **3** indicated that weak acidic acetanilide was deprotonated by strong base in pre-dried solvent and coordinated with the copper(I) centre. When complex **1** reacted with acetanilide in air, the oxidation product of dppf was obtained in good yield. TG experiments showed that complex **3** was thermally stable up to 240 °C, so it was more stable than complex **2** and was thermally competent for the high-temperature Goldberg reaction. CV experiment showed that oxidation of the copper centre was posterior to the ferrocene unit, so the copper centre was hard to be oxidized and complexes **2–3** were stable in air for months. Finally, the reaction mechanism with complex **3** as reaction intermediate was proven by the arylation reaction.

## 5. Supporting information

CCDC-863900 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html [or from the Cambridge Crystallographic Data Centre (CCDC), 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 (0)1223 336033; email: deposit@ccdc.cam.ac.uk]. The IR, $^1$H NMR of complexes **2–3**, $^{13}$C NMR spectrum of complex **2** and GC–MS spectrum of the reaction system after isolation of complex **2** are available from the supporting information.

## Acknowledgements

(The authors are grateful for the financial support from the National Natural Science Foundation of China (No. 20971058), Graduate Innovation and Research Foundation of Jiangsu Province (No. CXZZ11_0468) and the Fundamental Research Funds for the Central Universities (No. JUDCF11004)).

## Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.molstruc.2012.03.020.

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