Polyhedron 28 (2009) 4072–4076

Contents lists available at ScienceDirect

Polyhedron

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

Mixed-ligand complexes of copper(I) with diimines and phosphines: Effective catalysts for the coupling of phenylacetylene with halobenzene

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ARTICLE INFO

Article history:
Received 25 July 2009
Accepted 16 September 2009
Available online 29 September 2009

Keywords:
Copper(I) complexes
Crystal structures
Catalysis
Fluorescence

ABSTRACT

New copper(I) mixed-ligand complexes **1–4** of the formula Cu(N–N)PR_3X, where N–N = 1,10-phenanthroline (phen), 2,2′-bipyridine (bpy), 5,5′-dimethyl-2,2′-bipyridine (5,5′dimbpy) and PR_3 = tricyclohexylphosphine, tris(2-cyanoethyl)phosphine and isopropyldiphenylphosphine, have been synthesized. The complexes were characterized by EA, IR, NMR and single crystal X-ray diffraction. The solution fluorescence emission spectra were measured. The single crystal X-ray analysis showed that the copper(I) ion is four-coordinate with a distorted tetrahedral geometry. The complexes catalyze the formation of diphenylacetylene from the coupling of halobenzene with phenylacetylene. The complex Cu(5,5′-dimethylbpy)P(cyhexyl)$_3$I showed the highest catalytic activity. At room temperature all four complexes exhibit, in dichloromethane, emission maxima in the 329–344 nm range, corresponding to intra-ligand excited states.

1. Introduction

Cross-coupling reactions to form C–C and C-heteroatom bonds are of major importance in organic synthesis. While palladium-catalyzed reactions have seen important developments [1–5], copper-catalyzed reactions remain more economically attractive. The classical copper-based Ullman cross-coupling chemistry [6,7] has many disadvantages such as the drastic experimental conditions, requiring stoichiometric copper systems and affording rather moderate yields, in addition to the sensitivity to the substrate substituents [8,9]. Mechanistic studies have revealed that soluble cuprous species are most likely the active ones [10]. Recently, an increase of interest in copper-based cross-coupling chemistry has taken place [11–16] and versatile Cu(I)-based systems have been reported for C–C and C–N bond formation to produce arylacetylenes and triarylamines [17,18]. Most of the catalytic systems reported so far are not well defined as they are generated in situ, based on copper salts, ligands and additives. They require relatively prolonged reaction times and convert effectively, among aryl halides, only the most reactive aryl iodide substrates. Therefore there is a need to develop new well defined and versatile soluble catalysts that are active under milder conditions and less sensitive to the functional groups on the substrates.

Polypyridine metal complexes have been widely investigated for their photophysical properties, with various potential applications such as electroluminescent devices and solar energy conversion [19–21]. Complexes with higher quantum yields and emission shifts towards the visible range are being targeted [22,23]. Ru(II) complexes represent the most extensively investigated family [19]. Despite the fact that copper-based complexes are more attractive, both from a cost and a toxicity point of view, they have been less studied than their ruthenium counterparts [24]. Due to the diversity of their excited states, mixed-ligand copper(I) complexes based on polypyridine and phosphine ligands appear to have significant potential for photocatalytic applications. However, only few reports exist in the literature, including photochemical isomerization of norbornadiene to quadricyclane [25], reduction of cobalt (III) complexes [26] and reduction of methyl viologen [27]. As part of our interest in copper(I) mixed-ligand complexes [28], we describe herein the synthesis, spectroscopic characterization, X-ray structure and the results of the catalytic activity of four new mixed-ligand copper(I) complexes as versatile catalysts in the formation of aryl-acetylene bonds. Results of solution emission studies are also presented.

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doi:10.1016/j.poly.2009.09.021

2. Experimental section

2.1. General information

The experiments were carried out under an atmosphere of dried nitrogen using standard Schlenk techniques. Solvents were purified by conventional procedures and freshly distilled before use. All chemicals were purchased form Aldrich Chemicals. The compounds halobenzene, phenylacetylene, CuX (X = Br, I), the diimines and phosphines were used as received without further purification. The melting points were recorded using a Büchi 510 melting point apparatus and are uncorrected. ^{1}H NMR, ^{13}C NMR and ^{31}P NMR were recorded on a JEOL LAMBDA 500 spectrometer using tetramethylsilane and 85% H_3PO_4 as internal and external references respectively. Mass spectra were recorded on GC–MS Varian Saturn 2000 spectrometer. IR spectra were recorded using a Perkin Elmer FTIR 16FPC spectrometer. Gas chromatograms were obtained using an HP 6890 Plus instrument. Elemental analyses were carried out using a Perkin Elmer 2400 CHNS analyzer. UV–Vis spectra were obtained using a Perkin Elmer Lambda EZ 210 spectrometer. Solution photoluminescence measurements were carried out using a Shimadzu PF-5301 PC spectrofluorometer in dichloromethane with concentrations of 2×10^{-4} M for **1**, **3**, 4 and 2.5×10^{-4} M for **2**. Excitations were at 290 nm for each of the compounds **1**, 3 and **4** while compound **2** was excited at 300 nm.

2.2. X-ray structure determination

Data were collected at 120 K on a Bruker-Axs Smart Apex diffractometer using graphite monochromatized Mo Kα radiation (λ = 0.71073 Å). An empirical absorption correction was applied using the SADABS program [29]. All structures were solved by direct methods and subsequent Fourier difference techniques and refined anisotropically for all non-hydrogen atoms by full-matrix least-squares calculations on F^2 using the SHELXTL package [30]. All hydrogen atoms, except for the hydration water molecule in **4**, were included at calculated positions using a riding model. Crystal data and details of data collection and structure refinements are given in Table 1. Further details are included in the Supporting Information.

2.3. General procedure for the synthesis of complexes **1–4**

The reaction mixture of the diimine (1.0 mmol), CuX (1.0 mmol) and the phosphine (1.0 mmol) in 20.0 mL of dimethylformamide was stirred at 60 °C for 6 h. The mixture was filtered and the filtrate left to evaporate for 3–4 weeks to afford yellow or orange crystalline solids.

2.3.1. Cu(Phen)P[$(CH_2)_2$CN]$_3$I

Complex **1**: yellow, yield. 68%, M.p. (°C): 235–236. *Anal.* Calc. for $C_{21}H_{20}CuIN_5P$: C, 44.73; H, 3.58; N, 12.42. Found: C, 44.14; H, 3.56; N, 11.62%. IR (KBr, cm^{-1}): 3043s, 2911s, 2240s ($\nu_{C\equiv N}$), 1921w, 1586m, 1500m, 1416s, 1206m, 1050s, 949m, 839m, 753s, 509m, 458m. UV–Vis (DCM) λ, nm (ε, M^{-1} cm^{-1}): 269.0 (8360), 290.5 sh (3390), 315.0 sh (1020), 383.0 (769). ^{1}H NMR (r.t., DMSO-d_6, δ in ppm): 9.25 (br. s, $H^{2,9}_{phen}$, 2H), 8.76 (br. s, $H^{4,7}_{phen}$, 2H), 8.21 (br. s, $H^{5,6}_{phen}$, 2H), 8.05 (br. s, $H^{3,8}_{phen}$, 2H), 2.75–2.80 (m, PCH_2, 6H), 2.13–2.17 (m, CH_2CN, 6H). ^{31}P NMR (r.t., DMSO-d_6, 202.35 MHz): −12.09.

2.3.2. Cu(bpy)P[$(Ph)_2$(i-Pr)]Br

Complex **2**: orange, yield. 60%, M.p. (°C): 176–177. *Anal.* Calc. for $C_{25}H_{25}CuBrN_2P$: C, 56.87; H, 4.78; N, 5.31. Found: C, 56.65; H, 4.78; N, 5.74%. IR (KBr, cm^{-1}): 3773m, 3550s, 3004s, 2876s, 2760m, 2706m, 1975m, 1896m, 1818w, 1667w, 1577s, 1423s, 1298s, 1086s, 1001s, 753s, 661s, 470s. UV–Vis (DCM) λ, nm (ε, M^{-1} cm^{-1}): 288.5 (10 600), 387.5 (976). ^{1}H NMR (r.t., DMSO-d_6, δ in ppm): 8.70 (br. s, $H^{6,6'}_{bipy}$, 2H), 8.56 (br. s, $H^{3,3'}_{bipy}$, 2H), 8.14 (br. s, $H^{4,4'}_{bipy}$, 2H), 7.63 (br. s, $H^{5,5'}_{bipy}$, 2H), 7.40 (m, Ph, 10H), 2.77 (br. s, i-Pr, 1H), 0.81–0.86 (dd, i-Pr, 6H). ^{31}P NMR (r.t., DMSO-d_6, 202.35 MHz): 5.87.

2.3.3. Cu(5,5′-dimethylbpy)P[$(cyhexyl)_3$]I

Complex **3**: orange, yield. 62%, M.p. (°C): 180–181. *Anal.* Calc. for $C_{30}H_{45}CuIN_2P$: C, 54.99; H, 6.94; N, 4.28. Found: C, 55.50; H, 7.24; N, 4.19%. IR (KBr, cm^{-1}): 3869m, 3784m, 3708m, 3494m, 3426m, 3341m, 3155m, 3023m, 2920s, 2849s, 2651m, 2372m, 1966w, 1664m, 1573m, 1446s, 1161m, 1001m, 840m, 730m, 474m. UV–Vis (DCM) λ, nm (ε, M^{-1} cm^{-1}): 251.0 (9720), 303.0 (5540), 312.0 sh (5330), 429.5 (1870). ^{1}H NMR (r.t., DMSO-d_6, δ in ppm): 8.56 (s, $H^{6,6'}_{Dimbipy}$, 2H), 8.41 (s, $H^{3,3'}_{Dimbipy}$, 2H), 7.97 (s, $H^{4,4'}_{Dimbipy}$,

Table 1
Crystal and structure refinement data for **1**–**4**.

Complex	**1**	**2**	**3**	**4**
Chemical formula	$C_{21}H_{20}CuIN_5P$	$C_{25}H_{25}BrCuN_2P$	$C_{30}H_{45}CuIN_2P$	$C_{27}H_{29}BrCuN_2P.0.5(H_2O)$
Formula weight	563.83	527.89	655.09	564.95
Space group (No.)	C2/c (15)	P2(1)/n (14)	$P\bar{1}$ (2)	C2/c (15)
Temperature (K)	120	120	120	120
Radiation		Mo Kα (λ = 0.71073 Å)		
ρ_{calc} (g cm^{-3})	1.725	1.515	1.457	1.422
a (Å)	24.844(2)	12.082(2)	9.9791(7)	21.126(3)
b (Å)	7.9571(7)	13.512(2)	18.486(1)	16.557(2)
c (Å)	22.833(2)	14.201(2)	18.536(1)	17.851(3)
α (°)	90	90	117.671(1)	90
β (°)	105.831(1)	93.566(2)	96.773(1)	122.293(2)
γ (°)	90	90	92.717(1)	90
V (Å^3)	4342.6(7)	2313.9(5)	2986.8(4)	5278.2(13)
Z	8	4	4	8
μ (cm^{-1})	2.519	2.754	1.839	2.421
Reflections collected	18 504	19 955	26 522	22 822
Reflections unique (*R*(int))	5214 (0.0273)	5505 (0.0218)	13755 (0.0208)	6289 (0.0497)
Reflections observed ($I > 2\sigma(I)$)	4590	4940	11884	5129
R_1[a]	0.0258	0.0283	0.0339	0.0409
wR_2[b]	0.0653	0.0723	0.0775	0.0949

[a] $R_1 = \Sigma(|F_o| - |F_c|)/\Sigma|F_o|$ for ($I > 2\sigma(I)$).

[b] $wR_2 = \{\Sigma[w(F_0^2 - F_c^2)^2]/\Sigma[w(F_0^2)^2]\}^{1/2}$ for all data.

2H), 2.43 (s, CH_3, 6H), 1.26–1.84 (m, c-hexyl, 33H). ^{31}P NMR (DMSO-d_6, 202.35 MHz): −10.68.

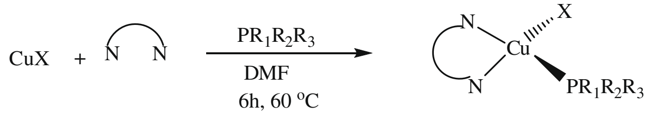
2.3.4. *Cu(5,5′-dimethylbpy)P[(Ph)$_2$(i-Pr)]Br. 0.5 H_2O*

Complex **4**: yellow, yield. 66%, M.p. (°C): 170–171. *Anal.* Calc. for $C_{27}H_{30}CuBrN_2O_{0.5}P$: C, 57.39; H, 5.36; N, 4.96. Found: C, 55.61; H, 5.13; N, 5.10%. IR (KBr, cm^{-1}): 3938m, 3771m, 3583m, 3419m, 3306m, 3233m, 3089m, 2813s, 2655s, 2531s, 2450s, 2240m, 1973m, 1838m, 1675m, 1572s, 1447s, 1239s, 1090m, 1031s, 835m, 694s, 513s. UV–Vis (DCM) λ, nm (ε, M^{-1} cm^{-1}): 253.0 (15 600), 267.5 sh (13 300), 300.5 (10 900), 382.5 (1140). ^{1}H NMR (r.t., DMSO-d_6, δ in ppm): 8.41 (br. s, $H^{6,6',3,3'}{}_{\text{Dimbipy}}$, 4H), 7.90 (br. s, $H^{4,4'}{}_{\text{Dimbipy}}$, 2H), 7.40 (s, Ph, 10H), 2.75 (br. s, i-Pr, 1H), 2.33 (s, Dimbipy CH_3, 6H), 0.79–0.84 (dd, i-Pr CH_3, 6H). ^{31}P NMR (DMSO-d_6, = 202.35 MHz): 6.09.

2.4. *General procedure for the catalytic coupling of phenylacetylene with halobenzene using catalysts **1–4***

In a nitrogen-filled glove box, a Schlenk tube equipped with a Teflon stirring bar, was charged with potassium carbonate (2.00 mmol) and the catalyst (10.0 mol% with respect to phenylacetylene), and was sealed with a rubber septum. The sealed tube was taken out of the glove box and phenylacetylene (2.50 mmol), iodobenzene (2.00 mmol) and toluene (15.0 mL) were injected into the tube through the septum. The mixture was then stirred at 115 °C for 24 h. The reaction mixture was then cooled to room temperature and filtered to remove any insoluble materials. The solvent was removed and the dry residue was recrystallized from pentane giving the product as a white solid.

M.p.: 62–63 °C. *Anal.* Calc. for $C_{14}H_{10}$: C, 94.33; H, 5.67. Found: C, 93.80; H, 6.02%. ^{1}H NMR (500 MHz, DMSO-d_6, δ in ppm): 7.41–7.43 (m, 4H), 7.54–7.56 (m, 6H). ^{13}C{^{1}H} NMR (125.65 MHz, DMSO-d_6, δ in ppm): 132.12, 130.81, 128.70, 123.20, 88.98.



Scheme 1. Synthesis of complexes **1–4**.

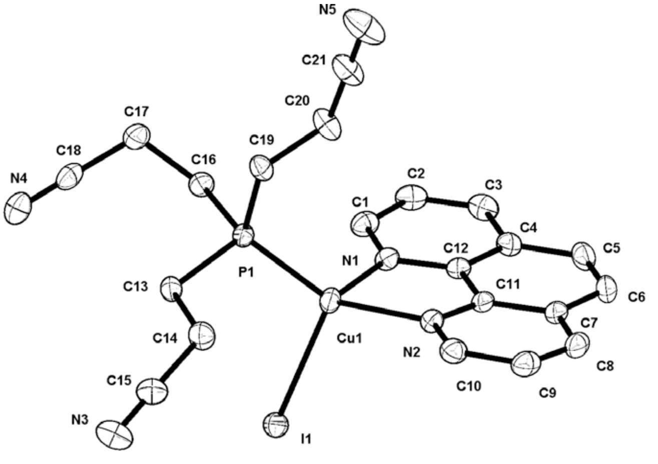


Fig. 1. Molecular structure of complex **1**.

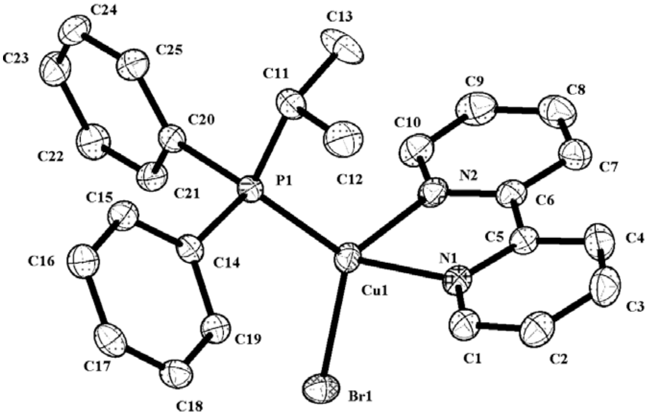


Fig. 2. Molecular structure of complex **2**.

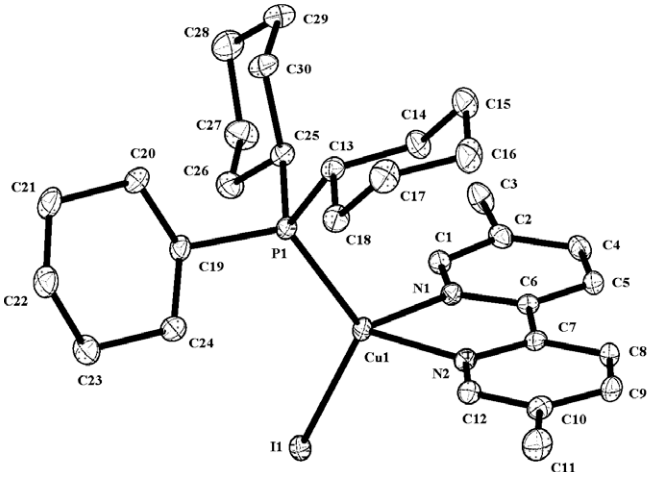


Fig. 3. Molecular structure of complex **3**.

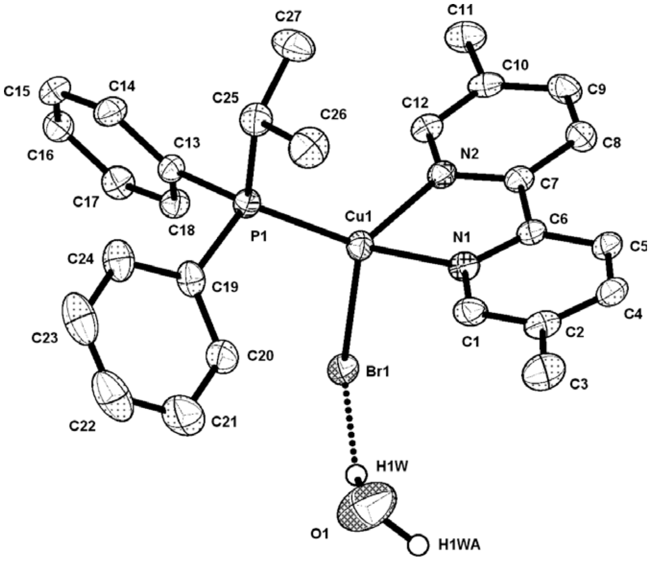


Fig. 4. Molecular structure of complex **4**.

Table 2
Selected bond lengths and bond angles for **1**–**4**.

Complex **1**		Complex **2**		Complex **3**		Complex **4**	
Cu1–N1	2.083(2)	Cu1–N1	2.086(2)	Cu1–N1	2.075(2)	Cu1–N1	2.064(2)
Cu1–N2	2.051(2)	Cu1–N2	2.095(2)	Cu1–N2	2.076(2)	Cu1–N2	2.069(2)
Cu1–P1	2.1817(6)	Cu1–P1	2.1967(5)	Cu1–P1	2.2033(8)	Cu1–P1	2.1792(8)
Cu1–I1	2.6427(4)	Cu1–Br1	2.4395(4)	Cu1–I1	2.5999(4)	Cu1–Br1	2.4933(5)
N2–Cu1–N1	81.17(7)	N1–Cu1–N2	78.52(6)	N1–Cu1–N2	78.99(9)	N1–Cu1–N2	79.44(9)
N2–Cu1–P1	132.71(5)	N1–Cu1–P1	117.93(5)	N1–Cu1–P1	112.84(6)	N1–Cu1–P1	123.36(6)
N1–Cu1–P1	117.14(5)	N2–Cu1–P1	118.70(5)	N2–Cu1–P1	120.22(6)	N2–Cu1–P1	125.41(6)
N2–Cu1–I1	106.28(5)	N1–Cu1–Br1	102.95(5)	N1–Cu1–I1	99.63(6)	N1–Cu1–Br1	104.89(6)
N1–Cu1–I1	108.31(5)	N2–Cu1–Br1	107.66(5)	N2–Cu1–I1	113.76(6)	N2–Cu1–Br1	104.74(6)
P1–Cu1–I1	107.66(2)	P1–Cu1–Br1	122.16(2)	P1–Cu1–I1	120.57(2)	P1–Cu1–Br1	113.34(2)

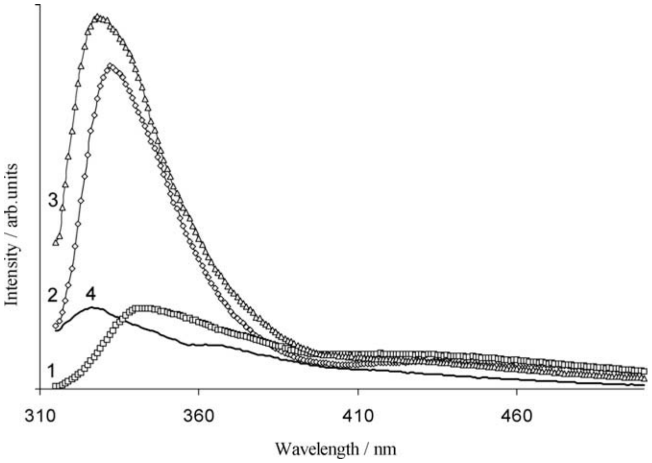


Fig. 5. Emission spectra of **1**–**4** at room temperature in dichloromethane.

3. Results and discussion

3.1. Synthesis of the complexes

Complexes **1**–**4** were synthesized as shown in Scheme 1. The reaction of stoichiometric amounts of the diimine and the phosphine with CuX in anhydrous dimethylformamide under reflux afforded mononuclear complexes in 60–68% yields. Complexes **1**, **2** and **4** are air stable while complex **3** decomposes after few weeks in air. All the compounds were characterized by elemental analysis, FT-IR, UV–Vis, ^{1}H and ^{31}P NMR spectroscopy. The ^{31}P NMR resonance shows a downfield shift in the range of 5.45–12.46 ppm relative to the free ligands, supporting the fact that the phosphorus atoms coordinate to the metal ion.

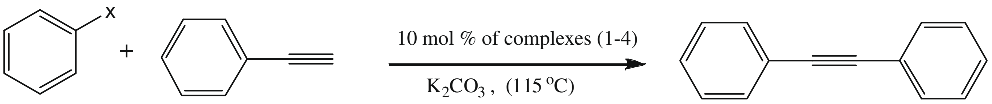
3.2. Molecular structures of complexes **1**–**4**

The molecular structures of complexes **1**–**4** were further confirmed by single crystal X-ray analysis (Figs. 1–4). In the four complexes, the copper(I) ion is four-coordinate with a distorted tetrahedral geometry (Table 2). The bond lengths and bond angles are in the same range as those reported for similar compounds [28]. In complex **1**, two of the three cyanoethyl groups adopt a conformation close to the expected energetically favorable anti conformation. The corresponding torsion angles are P1–C13–C14–C15 = −167.44° and P1–C19–C20–C21 = 178.52°. The third one has a less favorable synclinal conformation (P1–C16–C17–C18 = −75.69°). Due to the rigidity of 1,10-phenanthroline, the chelate bite angle is larger in **1** (81.17°) than for 2,2′-bipyridine and its derivative in compounds **2**–**4** (78.52–79.44°). In compounds **2** and **4**, the isopropyl group is anti to the bromo ligand with torsion angles C11–P1–Cu1–Br1 of 167.89° and C25–P1–Cu1–Br1 of 172.96°, respectively. For these two compounds, the electronic effect of the methyl substituent on the bipyridine ligand is reflected on the Cu–N bond distances, which are shorter in the 5,5′-dimethylbipyridine-based complex and associated with a longer Cu–Br bond distance. In compound **3**, the cyclohexyl groups adopt a chair conformation. In this case, the large cone angle of the phosphine and its conformation induces a distortion that affects more significantly the bond angles P1–Cu1–I1 and P1–Cu1–N2, 120.57° and 120.22°, respectively, in one of the molecules of the asymmetric unit. Compound **4** crystallizes as a hydrate and the bromo ligand is engaged in hydrogen bonding with the water molecule.

3.3. Catalysis

The four complexes were examined as catalysts for the coupling of aryl halides with phenyl acetylene to give diphenylacetylene using K_2CO_3 as a base in two different solvents, namely toluene

Table 3
Isolated yields of the reaction of aryl-halides with phenylacetylene in the presence of 10 mol% of **1**–**4**.



X	Catalyst							
	1		**2**		**3**		**4**	
I	69[a]	45[b]	66	55	95	75	73	59
Br	7	25	6	28	23	43	16	39
Cl	4	14	3	5	6	28	6	22

[a] In toluene.
[b] In DMF.

and dimethylformamide (Table 3). Good yields were obtained in the case of iodobenzene. Complex **3** showed the highest activity. Lower yields were obtained for bromo and chlorobenzene. Compound **3** appears to be more active for the formation of diphenylacetylene than the previously reported Cu(phen)(PPh$_3$)Br compound [17]. For iodobezene, the use of toluene as a solvent gives higher yields than dimethylformamide, however the opposite is observed for bromo and chlorobenzene.

3.4. Absorption and emission studies in solution

The absorption and emission spectra were recored in dichloromethane at room temparature. By comparison to the diimine free ligand absorption spectra, the following changes are observed upon coordination. For the metal complexes, the weakly structured and intense UV absorption bands (λ_{max} = 250–315 nm), attributed to intra-ligand transitions, are shifted to lower energies compared to the free ligands. This shift is more pronounced in the 5,5′-dimethylbipyridine based complexes **3** and **4**. A new broad band in the 320–500 nm range, attributed to a metal to ligand charge transfer (MLCT), appears.

Only excitations in the high energy region, corresponding to intra-ligand transitions, resulted in measurable fluoresence. The emission spectra of the four compounds are presented in Fig. 5. Complexes **1** and **4** have emissions with weak maxima at 344 nm and 329 nm, respectively, while compounds **2** and **3** have intense maxima at 333 and 331 nm, respectively.

4. Conclusion

New copper(I) mixed-ligand complexes based on diimines and phosphines have been synthesized and characterized. In the four compounds, the copper(I) ion is four-coordinate with a distorted tetrahedral geometry. The complexes catalyze the coupling of halobenzene with phenylacetylene to form diphenylacetylene. Good yields were obtained in the case of iodobenzene, while lower yields were obtained for bromo and chlorobenzene. The complex Cu(5,5′-dimethylbpy)P(cyhexyl)$_3$I showed the highest catalytic activity. It is also more active than the previously reported Cu(neocup)(PPh$_3$)Br and Cu(Phen)(PPh$_3$)Br catalysts. The photocatalytic studies of these complexes are underway.

5. Supplementary data

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic data Center with CCDC Nos. 731100, 731102, 731101 and 731099 for **1**, **2**, **3** and **4**, respectively. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

Acknowledgments

We gratefully acknowledge the King Fahd University of Petroleum and Minerals, Dhahran, Saudi Arabia for financial support.

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