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Synthesis of copper(I) β -diketone complexes

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Abstract

At room temperature, the reaction of metallic copper powder with 2-thenoyltrifluoroacetone (tfac) and selected phosphine ligands gives the new copper(I) complexes [Cu(PPh₃)₂(tfac)] (1), [Cu(dppm)–(tfac)]₂ (2), [Cu(dppe)(tfac)]₂ (3), [Cu(dppp)(tfac)] (4), [Cu(dppb)–(tfac)]₂ (5), Ph₂P(CH₂)_nPPh₂ [n = 1 (dppm), 2 (dppe), 3 (dppp), 4 (dppb)]. These complexes have been characterized by physicochemical and spectroscopic methods. X-ray structure analysis of 1 and 3 shows that tfac behaves as a chelating ligand and dppe coordinates as a bridging bidentate ligand to Cu(I) atoms in the Cu(I) complexes. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Copper; Complexes; Diketone; Crystal structure

1. Introduction

Copper(I) displays wide diversity in its structural chemistry, the copper coordination number ranging from two to six. Procedures to synthesize Cu(I) complexes are of great interest because of the variety of products resulting from almost the same methodology.

Binuclear Cu(I) complexes containing diphosphine ligands have been the focus of much investigation in the last few years. In light of this growing interest [1], many examples of complexes containing diphosphine ligands are known with a variety of metals in differing oxidation states and stereochemistry [2-4]. In general, bridging diphosphine ligands give an M₂P₄ core structure with the two metal atoms held in close proximity to each other (regardless of whether a metal-metal bond is present or not). This feature is presumably one of the chief reasons for the unusual bonding, reactivity, and catalytic properties of M_2 (diphosphine)₂ compounds. These compounds require additional ligands, since each metal ion in the M_2P_2 framework is coordinatively unsaturated. There are few reports concerning investigations of the structure and properties of the $Cu_2(dppe)_2$ framework [5]. In the present paper we describe an easy way to synthesize Cu(I) β -diketone(2thenoyltrifluoroacetone) (Cu(I) β -diketone(tfac)) complexes using phosphine ligands (PPh₃, dppm, dppe, dppp, dppb) and metallic copper powder. Structural results allowed us to interpret some properties of the complexes, such as molecular weight and conductivity measurements.

2. Experimental

2.1. General considerations

The compounds (dppm, dppe, dppp, and dppb) were synthesized according to the literature [3]. All other chemicals were of reagent grade quality obtained from commercial sources and used without further purification. All solvents were dried by standard methods. Elemental analyses were performed on an ERBA-1106 instrument (Italy). Cu and P contents were determined using a JA96-970 spectrometer. Molecular weight determinations were made on CHCl₃ solutions at 25°C using a CORONA-117 analyzer (American instrument). IR spectra were recorded on a Nicolet 170SX IR spectrophotometer. TG-DTA spectra were recorded on a PE-TGS-2 instrument. Conductivity measurements were carried out in acetone solutions thermostated at

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25°C using a Shanghai DDS-11A conductometer and DJS-1 type platinum black electrode. Melting points were determined on an electrothermal apparatus and are uncorrected. Room temperature (r.t.) ³¹P NMR spectra were taken on a DPX-400 NMR spectrometer, and measured in CDCl₃-d with 85% H₃PO₄ as external reference. XPS spectra were recorded on a VG ES-CALAB MK II instrument with Al K α radiation. Voltage, electric current and pressure were set at 12.5 kV, 20 mA and 6×10^{-6} Pa, respectively.

2.2. Synthesis of complexes 1–5

Dppe (2 mmol, 0.796 g) was added to a mixture of metallic copper powder (2 mmol, 0.127 g) and tfac (2 mmol, 0.444 g) in acetone. The mixture was stirred at r.t. for 48 h giving a yellow precipitate. The product was collected by filtration and washed with acetone. Yellow crystals were obtained by recrystallization of the product from CHCl₃ and THF. The crystals are stable in air. The complex $[Cu(dppb)(tfac)]_2$ (5) was prepared by a similar method. Using an acetone and THF solution, the complexes 1, 2, and 4 can be obtained by the above-described method. ¹H NMR (1, ppm): 6.449 (s, CH, tfac); 7.191–7.829 (m, ph, C₄H₃S). ¹H NMR (2, ppm): 3.185 (s, CH₂, dppm); 6.442 (s, CH, tfac); 6.937–7.728 (m, ph, C₄H₃S). ¹H

Table 1

Crystal data and structure refinement parameters

Empirical formula Color Crystal system Space group Unit cell dimensions	$C_{44}H_{33}CuF_3O_2P_2S$ yellow monoclinic $P2_1/n$	$\begin{array}{l} {\rm Cu_2C_{68}H_{56}P_4O_4S_2F_6}\\ {\rm yellow}\\ {\rm monoclinic}\\ {C_2/c} \end{array}$
$a (\mathring{A})$ $b (\mathring{A})$ $c (\mathring{A})$ $\alpha (\degree)$ $\beta (\degree)$ $\gamma (\degree)$ $\gamma (\clubsuit)$	11.012(2) 21.282(4) 17.241(3) 90 101.68(3) 90 2056 0(14)	25.215(6) 15.304(9) 18.851(6) 90 107.20(3) 90 (040.0(2))
V (A ²) Z D_{calc} (g cm ⁻³) Absorption coefficient (cm ⁻¹)	4 1.357 7.36	4 1.375 8.196
Crystal size (mm) 2θ Range (°) Index ranges	$\begin{array}{l} 0.5 \times 0.4 \times 0.2 \\ 3.08 - 46 \\ 0 < h < 12, \\ -23 < k < 23, \\ -18 < l < 18 \end{array}$	$\begin{array}{l} 0.1 \times 0.1 \times 0.15 \\ 2-49 \\ 0 < h < 27, \ 0 < k < 18, \\ -22 < l < 22 \end{array}$
Independent reflections Solution Final <i>R</i> indices	5484 direct methods R = 0.0568, $R_w = 0.1437$ $[I > 2\sigma(I)]$	6301 direct methods R = 0.06682, $R_w = 0.07326$ $[I > 3\sigma(I)]$
Largest difference peak and hole (e $Å^{-3}$)	0.439	0.842

NMR (**3**, ppm): 1.750–2.597 (m, CH₂, dppe); 6.444 (s, CH, tfac); 7.206–7.829 (m, ph, C_4H_3S). ¹H NMR (**4**, ppm): 2.456–2.546 (m, CH₂, dppp); 6.445 (s, CH, tfac); 7.198–7.802 (m, ph, C_4H_3S). ¹H NMR (**5**, ppm): 1.720–2.307 (m, CH₂, dppb); 6.447 (s, CH, tfac); 7.184–7.748 (m, ph, C_4H_3S).

2.3. X-ray data collection and structure determination

A single crystal of each complex (1 and 3) was selected and mounted on an automatic Enraf-Nonius CAD-4 four-circle diffractometer equipped with a graphite monochromator and Mo K α radiation (λ = 0.71073 Å). Unit cell dimensions and intensity data were measured at r.t. Reflections were collected using $\omega - 2\theta$ scan (scan width $1.00 + 0.35 \tan \theta$). No significant change was detected in the intensity of three standard reflections. Lorentz, polarization, and absorption corrections (empirical method, Ψ -scan) were applied to the intensity data. The structure was solved by direct methods and Fourier synthesis. The structure was refined by full-matrix least-squares method. Computations were performed using the SDP program on a PDP11/44 computer. Details of the crystal data and intensity collection are summarized in Table 1. Selected bond lengths and angles are given in Table 2.

3. Results and discussion

3.1. Preparation of the complexes

According to the color changes that accompany the preparation of complexes 1-5, metallic copper powder has reacted with the phosphine ligand and tfac to form Cu(I) complexes. The direct use of metallic copper powder and tfac is characteristic of the present synthetic method. Molecular weights were measured in CHCl₃ solution, the complexes being soluble enough for measurements. The results are quite reproducible and seem to be scarcely influenced by concentration changes in the accessible range. The molecular weight determinations strongly suggest that the complexes 2, 3, and 5 are binuclear. The Cu(I) complexes (1-5) can be obtained with high yields. The elemental analyses of the five complexes agreed well with their formulae (Table 3). Conductance data show that the complexes are non-electrolytes. At room temperature, the title complexes are crystalline, diamagnetic compounds, are fairly stable in air in the solid state, are soluble in organic solvents such as CH₂Cl₂, DMF, and CHCl₃, and are sparingly soluble in benzene, THF, and hexane.

Table 2										
Selected	bond	lengths	(Å)	and	angles	(°)	for	1	and	3

Complex 1					
Bond lengths					
Cu–O(1)	2.059(5)	Cu–O(2)	2.118(5)	Cu–P(1)	2.2243(19)
Cu–P(2)	2.254(2)	O(2)–C(2)	1.261(10)	O(1)-C(4)	1.232(9)
Bond angles					
O(1)-Cu-O(2)	89.0(2)	O(1)–Cu–P(1)	114.37(16)	O(2)–Cu–P(1)	107.71(16)
O(1)-Cu-P(2)	106.20(17)	O(2)–Cu–P(2)	112.94(16)	P(1)-Cu-P(2)	121.87(8)
C(4)-O(1)-Cu	125.6(6)	C(2)–O(2)–Cu	120.0(5)		
Complex 3					
Bond lengths					
Cu(1) - P(1)	2.236(2)	Cu(1)–P(2)	2.244(3)	Cu(1)–O(1)	2.096(6)
Cu(1)–O(2)	2.064(6)	O(1)–C(3)	1.255(10)	O(2)–C(5)	1.259(9)
Bond angles					
P(1)-Cu(1)-P(2)	118.49(9)	P(1)-Cu(1)-O(1)	106.9(2)	P(1)-Cu(1)-O(2)	115.9(2)
P(2)-Cu(1)-O(1)	106.3(2)	P(2)-Cu(1)-O(2)	115.0(2)	O(1)–Cu(1)–O(2)	88.7(2)
Cu(1)-P(1)-C(1)	112.6(3)	Cu(1)-P(1)-C(11)	117.7(3)	Cu(1)–P(1)–C(21)	115.5(3)
Cu(1)-O(1)-C(3)	126.8(6)	Cu(1)-O(2)-C(5)	124.4(6)	Cu(1)-P(2)-C(2)	110.9(3)
Cu(1)-P(2)-C(31)	116.5(3)	Cu(1)-P(2)-C(41)	118.1(3)	Cu(1)-O(1)-C(3)	126.8(6)

Table 3

Analytical data and physical properties of the complexes

Complex	С	H (calc.%)	Р	Cu	Yield (%)	m.p. (°C)	$\Lambda_{\rm M}$ (S cm ² mol ⁻¹)	M (calc.)	Color
1	65.44 (65.31)	3.98 (4.20)	7.8 (7.7)	8.1 (7.8)	76	116	5.2	807 (808.5)	yellow
2	59.48 (59.24)	4.01 (3.89)	9.6 (9.3)	9.3 (9.5)	71	225	1.2	1339 (1337)	orange
3	59.63 (59.78)	4.27 (4.10)	8.8 (9.1)	9.0 (9.3)	79	248	4.9	1367 (1365)	yellow
4	60.15 (60.30)	4.58 (4.31)	9.1 (8.9)	8.9 (9.1)	66	110	5.6	695 (696.5)	yellow
5	60.77 (60.80)	4.35 (4.50)	8.9 (8.7)	9.1 (8.9)	81	234	3.8	1419 (1421)	orange

Table 4

Absorption maxima and IR of the complexes

Complex	$\lambda_{\rm max}$ (nm)	³¹ P ^a (ppm)	$v_{c=0} \ (cm^{-1})$	$v_{c=c} \ (cm^{-1})$	$v_{\rm M-O}~({\rm cm}^{-1})$	$v_{M-P} (cm^{-1})$	$\lambda_{\rm em}$ (nm)
1	326, 310, 213		1605	1545	300	195	540, 610
2	327, 256, 243	-13.6	1610	1550	290	196	525
3	320, 261, 240	-6.2	1608	1552	298	198	525
4	325, 258, 239	-3.2	1610	1550	300	200	
5	323, 260, 242	-7.7	1610	1535	300	198	530

^a 85% H₃PO₄ as an external reference.

3.2. IR data and ³¹P NMR spectra

The characteristic $v_{c=0}(1650 \text{ cm}^{-1})$ of free tfac shifts to lower frequency $(1605-1610 \text{ cm}^{-1})$ in the Cu(I) complexes **1–5** (Table 4). β -Diketone(tfac) in the complex is in the enol form and is bonded to the metal in a chelate mode. The C=O and C=C stretching vibration of the enol ring were assigned to absorptions at 1605– 1610 and 1535–1550 cm⁻¹, respectively. The vibrational absorption peak shape and intensity change were obviously in the range 1000–1400 cm⁻¹. The P–Ph absorption, at about 1100 cm⁻¹, shows an increase in frequency and intensity, which is characteristic of P–metal coordination. In ³¹P NMR spectra of the complexes (Table 4), the corresponding phosphorus resonance is shifted to higher field compared to that of the free ligand (dppm: -22.9; dppe: -12.6; dppp: -13.0; dppb: -11.9 ppm). ³¹P NMR spectra at room temperature only display a single resonance showing that all phosphorus atoms in each molecule are chemically equivalent [3].

3.3. TG-DTA analysis and XPS spectra

The results of the thermogravimetric analyses of the complexes, compared with those of free ligands, indicate that the thermal stability of the phosphine compounds increases upon complexation. The TG-DTA thermogram of 1 reveals that the pyrolytic decomposition takes place in one step. This process in the 200– 500 range corresponds to a pronounced weight loss of 89% and is probably due to the decomposition of the organic moiety (loss of weight calc. 90.2%), giving CuO as final residue (exp.: 11.0%; calc.: 9.8%). The TG-DTA pattern of complexes 2-4 is similar to that of complex 1.

The XPS data of the complexes give information concerning copper-ligand binding. The binding energy of the complexes **1**, **2**, and **3** are at C_{1s}: 288.65 (**1**), 288.63 (**2**), 288.61 (**3**); O_{1s}: 536.25 (**1**), 536.23 (**2**), 536.21 (**3**); S_{2p}: 168.20 (**1**), 168.18 (**2**), 168.19 (**3**); P_{2p}: 135.00 (**1**), 134.71 (**2**), 133.52 (**3**); and Cu_{2p}: 936.85 (**1**), 936.30 (**2**), and 936.12 (**3**) eV, respectively. The binding energy P_{2p} of the phosphine ligand in the complexes **1**-**3** is larger than the relative value of the free ligand (PPh₃: 133.9 eV; dppm: 132.5 eV; dppe: 132.4 eV). This is attributed to electronic density reduction of the P atom because of the formation of a P→Cu bond in the complexes.

3.4. Luminescence properties

The electronic absorption spectrum of the title complexes in CH₂Cl₂ exhibit low-energy bands in the 310– 330 nm region. Excitation of solid samples of the complexes at $\lambda = 355$ nm at room temperature produces long-lived luminescence at $\lambda = 525-540$ nm. Luminescence spectra of **1** are shown in Fig. 1. The room temperature solid-state emission spectra of the complexes **2**–**5** showed very similar patterns. There is extensive literature on the emission of Cu(I) complexes [6–8]. In the trinuclear Cu(I) complex [Cu₃(dpmp)₂-(MeCN)₂Cl₂]⁺, where dpmp is bis(diphenylphosphinomethyl)methyl, an emission at 560 nm was assigned

as a metal-centered $3d^94s^1 \rightarrow 3d^{10}$ transition [6]. In the binuclear Cu(I) complex [(PPh₃)Cu₂Cl₂(py)], a similar type of assignment has been made [7]. Based on a literature assignment of analogous compounds and our experimental observation, the low-energy and long lifetime emission (525-540 nm) is most likely associated with the metal-centered excited state 3d⁹4s¹ of Cu(I) since the low-energy emission occurs at similar energies for complexes 1-5, irrespective of the nature of the phosphine ligand. Complex 1 also exhibits another emission band maximum at ~ 610 nm. The Cu(I) center, a d¹⁰ system, is very electron rich in nature and can be stabilized by ligands having π -acid character, viz. PPh₃, bpy, phen, CO, CN, etc. Complexes having the general formula $[Cu(PPh_3)_2(NN)]^+$ are known to exhibit emission properties both at low and room temperature. There are emissions at 608 nm for $[Cu(PPh_3)(phen)]^+$ and at 620 nm for $[Cu(PPh_3)(bpy)]^+$ [9]. These emissions were tentatively assigned as metalto-ligand charge transfers (MLCT). Thus, the emission band at ~ 610 nm in 1 has also been assigned as MLCT.

3.5. Crystal structures of complexes 1 and 3

The molecular structures of **1** and **3** are depicted in Figs. 2 and 3, respectively. In complex **1** the copper ion is four coordinate with a tetrahedral sphere of two phosphorus atoms from PPh₃ and two oxygen atoms from tfac. The Cu–P distances in $[Cu(PPh_3)_2(tfac)]$ vary from 2.2243(19) to 2.254(2) Å, with a mean value of 2.239(2) Å, in good agreement with the average value of 2.2312(8) Å found in $[Cu(PPh_3)_2(NO_3)]$ [10]. The thiophene ring in **1** may rotate around the C(4) atom.

The solid-state **3** consists of a neutral dimeric molecular unit with the two copper atoms bridged by a pair



Fig. 1. Luminescence spectrum of complex 1; excitation was performed at $\lambda = 355$ nm.



Fig. 2. Molecular structure of complex 1.



Fig. 3. Molecular structure of complex 3.

of dppe ligands. In addition, each copper atom is bound by tfac in a chelating fashion. The two copper atoms are doubly bridged by two dppe ligands to form a ten-membered $Cu_2P_4C_4$ ring. Each copper atom is four coordinate, having a P_2O_2 donor set. The slightly distorted tetrahedral coordination around copper is completed by two phosphorus atoms from the dppe ligand and two oxygen atoms from tfac. It has already been observed that Cu–P bond lengths in similar compounds are mainly influenced by the number of phosphorus atoms bonded to the same copper atom. In the present compound, the Cu(I) atom is coordinated by two phosphorus atoms. The Cu–P distances in **3** of 2.236(2) and 2.244(3) Å are in good agreement with the values of 2.240(6) and 2.249(6) Å found in [Cu(S₂CC₆- $H_4Me-O(dppm)]_2$ [11]. The Cu-Cu separation of 3.652(10) Å is significantly longer than the range of Cu-Cu bond lengths [2.494(5)-2.674(5) Å] found in $H_6Cu_6(PPh_3)_6$ DMF [12], suggesting that the copper atoms in the present compound are not involved in metal-metal bonding interactions. The C-O and C-C bond lengths in tfac are in the region expected for localized symmetrical bonding of the β -diketone ligand (tfac). The bond lengths of C(3)-C(4), C(4)-C(5), C(3)-C(6), and C(5)-C(10) are 1.410(14), 1.381(4), 1.492(3), and 1.521(14) Å, respectively. The bond lengths of C(3)–C(4) and C(4)–C(5) are shorter than those of C(3)–C(6) and C(5)–C(10) because C(3), C(4), and C(5) form a conjugated system. The bond C(3)-C(6), linking to the thiophene ring, falls within the limits for single and double bond lengths. This certifies that there is a conjugated system formed between the chelating ring and the thiophene ring.

In each copper moiety, all of the distances between the copper and donor atoms are different. The observed Cu-O distances of 2.096(6) and 2.064(6) Å are comparable to the Cu–O values [1.954(11) Å] found for the complex [Cu(2,2'-bipy)(tfac)] [13]. Structural studies involving binuclear phosphine species of other transition metals (Mn [14], Mo [15]) are less common but, in general, reveal linear P-M-P fragments and planar M₂P₄ skeletal units. For d¹⁰ complexes, however, the P-M-P units are distinctly non-linear [16,17]. X-ray structural characterization of the complex [Cu-(dppe)(tfac)]₂, reported here, clearly reveals that the P-M-P angles in the complex are not restricted by steric crowding of dppe phenyl rings. The P-Cu-P angles in 3, in particular, are much more bent (118.49°) than the corresponding P-M-P values (131.8°) found in $[Cu(dppm)(NO_3)]_2$ [3], but similar to the corresponding P(1)-Cu-P(2) angle of 119.9(1)° found in the related binuclear (dppm)₂ complex [Cu(dppm)(MeCN)₂]₂- $(ClO_4)_2$ [18]. The two bis(diphenylphosphine)ethane ligands in 3 are located on opposite sides of the complex to minimize repulsion between the ligands.

In summary, we have prepared the following Cu(I) β -diketone complexes [Cu(PPh_3)₂(tfac)], [Cu-(dppp)(tfac)], and [Cu(tfac)L]₂ (L = dppm, dppe, dppb). The doubly bridged bis(diphenylphosphine)ethane complex [Cu(dppe)(tfac)]₂ has been structurally characterized by single-crystal X-ray diffraction and found to contain a folded Cu₂P₄ core. In addition, the M₂(dppe)₂ core in **3** is apparently quite flexible, exhibiting varying degrees of folding along the M···M axis in the solid state. The coordinative unsaturation of the dimer is particularly intriguing in that this should facilitate the binding of other species [19,20].

4. Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC Nos. 147876 and 147877 for compounds 1 and 3, respectively. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (fax: +44-1223-336033; e-mail: deposit@ccdc.cam. ac.uk or www: http://www.ccdc.cam.ac.uk).

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