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Note

Syntheses and characterization of luminescent dinuclear and polynuclear Cu(I) complexes containing 4,4′-bis(diphenylphosphino)biphenylene bridge

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Abstract

The dinuclear complex $[Cu_2(dpbp)_2(NCMe)_4][BF_4]_2$ (**1**) has been prepared by treating $[Cu(NCMe)_4][BF_4]$ with 4,4′-bis(diphenylphosphino)biphenylene (abbreviated as dpbp). Reactions of **1** with 2,2′-bipyridine and 1,1′-bis(diphenylphosphino)ferrocene (abbreviated as dppf) afford $[Cu_2(dpbp)_2(2,2'\text{-bipy})_2][BF_4]_2$ (**2**) and $[Cu_2(dpbp)(dppf)_2][BF_4]_2$ (**3**), respectively. In contrast, compound **1** reacts with tetra(2-pyridyl)ethyl-1,4-diaminobutane (abbreviated as tpyda) to produce the polymeric complex $\{[Cu_2(dpbp)(tpyda)][BF_4]_2\}_n$ (**4**). Compounds **1**–**4** are photoluminescent with the emission band (λ_{max}) in the range 510–554 nm. The crystal structures of **1** and **4** have been determined by an X-ray diffraction study.

Keywords: 4,4′-bis(diphenylphosphino)biphenylene; 1,1′-bis(diphenylphosphino)ferrocene; Tetra(2-pyridyl)ethyl-1,4-diaminobutane; Cu(I) complexes

1. Introduction

Assembly of metal complexes (or ions) with suitable multidentate ligands allows the preparation of well-defined inorganic metal clusters and supramolecular architectures in solution [1,2]. Recently, interest in the design and synthesis of copper dimeric and polymeric complexes was stimulated by their potential roles in catalysis, material science and drug design [3], and in biologically active molecules [4]. The 4,4′-bis(diphenylphosphino)biphenylene (abbreviated as dpbp) ligand [5], which contains two PPh_2 groups linked by a rigid aromatic spacer, may serve as a phosphorous σ-donor bridge in coordination and organometallic compounds [6]. Yet, only a few polymeric Ag(I) [7] and Au(I) [8] complexes of this ligand were known. We report herein four new dinuclear and polynuclear Cu(I) complexes containing dpbp bridge, with the reactions summarized in Scheme 1. Identity of these new compounds has been characterized by elemental analysis, mass and NMR spectroscopy, and by X-ray crystallography for **1** and **4**.

2. Results and discussion

Treatment of $[Cu(NCMe)_4][BF_4]$ with one equiv of dpbp in dichloromethane at room temperature under a dinitrogen atmosphere produces air-stable, colorless crystals of $[Cu_2(dpbp)_2(NCMe)_4][BF_4]_2$ (**1**) in 93% yield. The ^1H NMR spectrum shows a multiplet in the range δ 7.53–7.08 for the C_6H_5 and C_6H_4 groups and a singlet at δ 2.19 for the NCMe groups. The $^{31}P\{^1H\}$ NMR spectrum presents a broad signal at δ 0.24 for the coordinated dpbp groups, which shows a downfield shift in comparison with the free ligand at $\delta - 5.61$. The structure for the cation of **1**, depicted in Fig. 1, consists of two $[Cu(NCMe)_2]^+$ groups bridged by two dpbp ligands. The asymmetric unit contains half of the molecule, and the complete structure is

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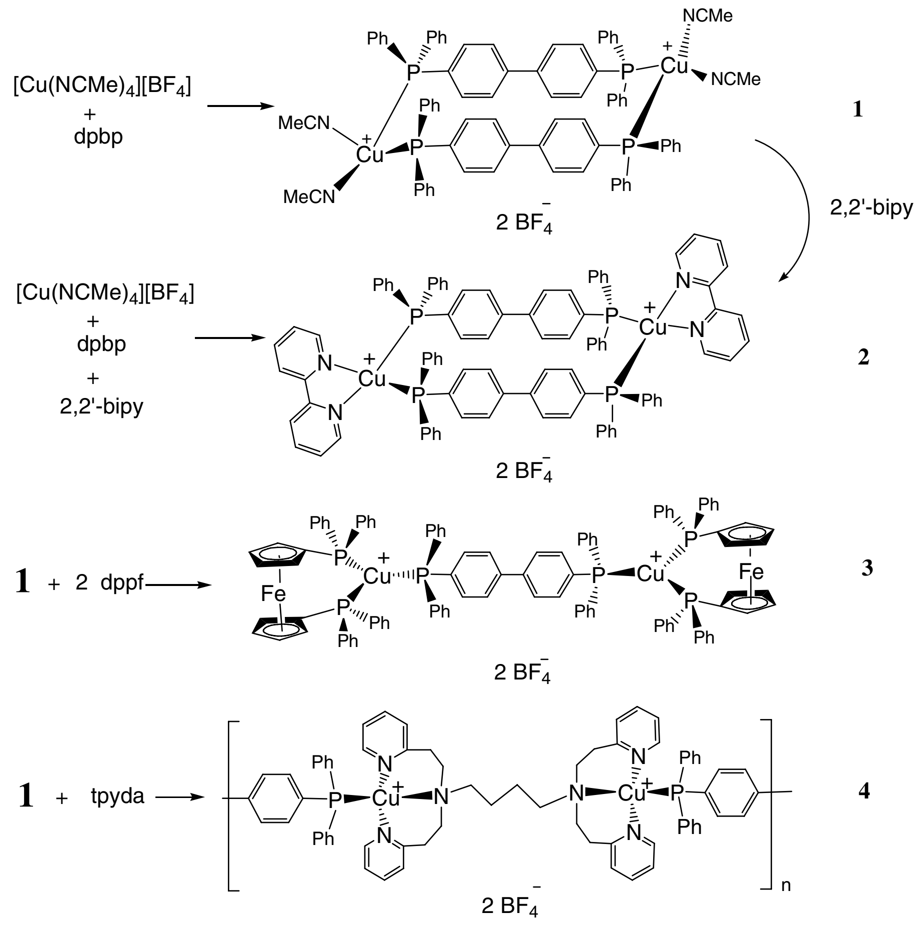

Scheme 1.

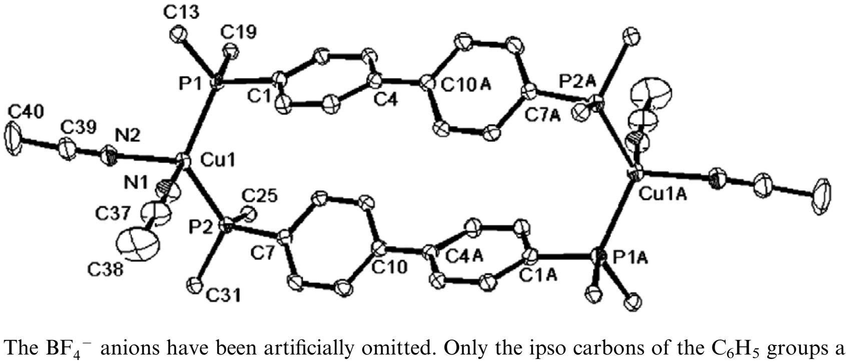

Fig. 1. ORTEP diagram of **1**. The BF_4^- anions have been artificially omitted. Only the ipso carbons of the C_6H_5 groups are shown for clarity. Selected bond distances (Å) and angles (°): Cu1–N1 2.083(3), Cu1–N2 2.033(3), Cu1–P1 2.2799(8), Cu1–P2 2.2765(8); P1–Cu1–P2 120.92(3), P1–Cu1–N1 109.2(1), P1–Cu1–N2 109.60(9), P2–Cu1–N1 102.9(1), P2–Cu1–N2 114.8(1), N2–Cu1–N1 96.0(1).

generated by a crystallographic inversion. The coordination about the Cu^+ ion can be described as an irregular tetrahedron, where the angles around the copper atom vary from 96.0(1)° for N1–Cu1–N2 to 120.92(3)° for P1–Cu1–P2. These values are significantly deviated from the ideal value of 109.5°, and may be attributed to the great steric effect of the bulky phosphine moieties. A similar case was found in $[Cu_2(dppm)_2(NCMe)_4]^{2+}$ [9]. The $[Cu_2(dpbp)_2]^{2+}$ unit forms a chair-like dimetallacycle. With the four phosphorous atoms making the basal plane, the two copper atoms are in the opposite sides and 0.91 Å from the plane, where the Cu···Cu distance is 12.2 Å. The biphenyl group is slightly bowed, such that the C1, C4, C10A and C7A atoms are 0.08–0.11 Å deviated from the P1–P2A line, and the two C_6H_4 rings are staggered by 34.9° in average. The Cu–P lengths are about equal, being 2.278 ± 0.003 Å, while the Cu1–N1 length of 2.083(3) Å is slightly longer than the Cu1–N2 length of 2.033(3) Å.

Compound **1** contains four labile acetonitrile ligands, which are readily replaced by 2,2′-bipyridine in dichloromethane at room temperature to produce $[Cu_2(dpbp)_2(2,2'\text{-}bipy)_2][BF_4]_2$ (**2**) in 95% yield. Alternatively, compound **2** can be obtained in 76% yield by self-assembling of equal quantity of $[Cu(NCMe)_4][BF_4]$, dpbp and 2,2′-bipyridine in acetonitrile/dichloromethane solvents. Compound **2** forms an air-stable, yellow crystalline solid. The ^{1}H NMR spectrum shows four 4H signals at δ 8.54, 8.32, 8.08 and 7.51 for the pyridine protons and a multiplet (56H) in the range δ 7.41–7.08 for the phenyl protons. The ^{31}P resonance for the phosphine groups is recorded at δ 1.99. The ESI mass spectrum shows the highest peak at m/z 741.5, corresponding to the $[Cu_2(dpbp)_2(bipy)_2]^{2+}$ species. Thus, compound **2** is likely to have a structure similar to **1**, except that the acetonitrile groups are replaced by a 2,2′-bipyridine chelate on each copper atom.

Treatment of **1** with 1,1′-bis(diphenylphosphino)ferrocene (abbreviated as dppf) in dichloromethane, however, results in the dissociation of one dpbp bridge to afford $[Cu_2(dpbp)(dppf)_2][BF_4]_2$ (**3**; 78% yield) as air-stable, orange crystals. The ^{1}H NMR spectrum displays a multiplet (68H) ranging from δ 7.48–7.14 for the phenyl protons, and two broad 8H signals at δ 4.51 and 4.24 for the Cp protons. The $^{31}P\{^1H\}$ NMR spectrum presents a triplet at δ 2.73 ($J_{P-P} = 95$ Hz) and a doublet at $\delta - 8.07$ in about a 1:2 ratio for the dpbp and dppf groups, respectively. On the basis of the spectral data, a structure with each copper ion chelated by a dppf ligand, and the two $[Cu(dppf)]^+$ units bridged by a dpbp ligand can be constructed for **3**. The importance of steric constrains in the trigonal $[Cu(PPh_2R)_3]^+$ complexes was well established [10]. Thus, it is plausible that each copper ion of **3** adopts a trigonal coordination with the three phosphorous atoms forming the basal plane, analogous to the structure determined for $[Cu_2(Ph_2PC{\equiv}CPPh_2)(dppf)_2][BF_4]_2$ [11].

Both 2,2′-bipyridine and dppf serve as chelating agents in **2** and **3**. It was thought that treating **1** with a multifunctional bridging ligand might lead to an oligomeric or polymeric species. Thus, compound **1** was reacted with tetra(2-pyridyl)ethyl-1,4-diaminobutane (abbreviated as tpyda) [12] in dichloromethane at room temperature to produce air-stable, pale-yellow crystals of $\{[Cu_2(dpbp)(tpyda)][BF_4]_2\}_n$ (**4**) in 50% yield, after crystallization from acetonitrile/toluene. Complex **4** is highly soluble in DMSO and moderately soluble in dichloromethane and acetonitrile. The ^{1}H NMR spectrum shows the phenyl and pyridyl proton resonances in the range δ 8.08–7.07 (44H) and the methylene proton resonances at δ 2.82 (8H), 2.62 (12H) and 2.14 (4H). The ^{31}P resonance for the dpbp bridge is recorded at $\delta - 0.55$. The crystal structure of **4** consists of a packing of chains of Cu^+–dpbp–Cu^+–tpyda and tetrafluoroborate ions (Fig. 2A), and the configuration about the dicopper unit is amplified in Fig. 2B. The polymer is arranged in a zigzag manner, where the Cu ions are bridged by dpbp and tpyda ligands alternatively, with the Cu···Cu distances of 13.2 Å and 9.2 Å. The coordination about the Cu^+ ion is a distorted tetrahedron, with the P1–Cu1–N angles in the range 111.8(3)–120.0(2)° and N–Cu1–N angles in the range 96.9(3)–105.3(3)°. The Cu1–P1 distance of 2.214(3) Å is ca. 0.06 Å shorter than that found in **1**, consistent with the weaker steric strains in **4**. The amine N1atom is coordinated to Cu1 with Cu1–N1 2.16(1) Å, and the two other pyridyl N2 and N3 atoms are at relatively shorter distances to copper, with Cu1–N2 2.070(8) Å and Cu1–N3 2.069(9) Å.

The electronic absorption spectra of compounds **1**–**4** in acetonitrile solvent display broad UV absorption bands at 258–300 nm. The free dpbp, dppf, 2,2′-bipyridine and tpyda ligands absorb similarly with maxima at 236–

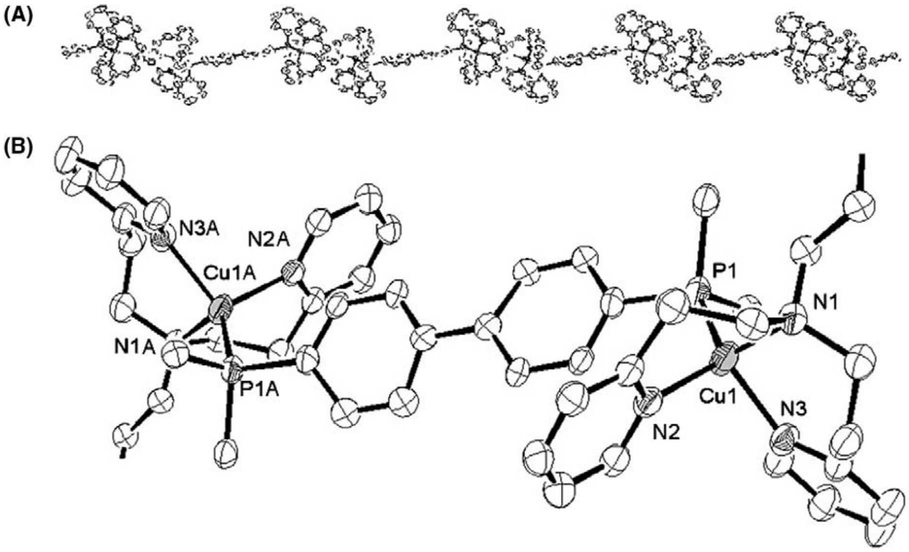

Fig. 2. ORTEP diagrams of **4** with (A) showing the polymeric chain structure, and (B) showing the coordination of the dicopper(I) unit. The BF_4^- anions have been artificially omitted. Only the ipso carbons of the C_6H_5 groups are shown for clarity. Selected bond distances (Å) and angles (°): Cu1–N1 2.16(1), Cu1–N2 2.070(8), Cu1–N3 2.069(9), Cu1–P1 2.214(3); P1–Cu1–N1 118.8(2), P1–Cu1–N2 120.0(2), P1–Cu1–N3 111.8(3), N1–Cu1–N2 96.9(3), N1–Cu1–N3 98.8(3), N2–Cu1–N3 105.3(3).

301 nm. The resemblance of the metal complex and free ligand absorptions suggests significant $\pi \rightarrow \pi^*$ (Ph, Py) character in the transitions [13]. Furthermore, upon the UV radiation at 350 nm, compounds **1**–**4** (in acetonitrile) exhibit photoluminescence with the emission band (λ_{max}) at 510 nm for **1**, 521 nm for **2**, 543 nm for **3**, and 554 nm for **4**. Since the dpbp ligand emits in the same region with λ_{max} 533 nm, emission of the complexes likely involve an excited state of metal perturbing intraligand origin [14].

In conclusion, the results presented here describe novel dinuclear and polynuclear Cu(I) complexes containing dpbp and tpyda bridges. There has been an increasing interest in inorganic polymers with the expectation that these polymers might exhibit unique properties difficult to achieve with the conventional organic polymers [15]. The formation of **4** suggests that compound **1** may serve as a pre-organized dicopper building block for constructing a 2D or 3D network by reaction with the appropriate linking ligands. Further investigation is progressive in our laboratory.

3. Experimental

3.1. General methods

All manipulations were carried out under an atmosphere of purified dinitrogen with standard Schlenk techniques. $[Cu(MeCN)_4][BF_4]$ [16], 4,4′-bis(diphenylphosphino)-biphenylene [5], 1,1′-bis(diphenylphosphino)ferrocene [17], and tetra(2-pyridyl)ethyl-1,4-diaminobutane [12] were prepared as described in the literature. Solvents were dried over appropriate reagents under a dinitrogen atmosphere and distilled immediately before use. Fast-atom-bombardment (FAB) and electrospray ionization (ESI) mass spectra were recorded on a VG Blotch-5022 mass spectrometer and a Bruker APEX II mass spectrometer, respectively. ^{1}H and ^{31}P NMR spectra were obtained on a Varian Unity INOVA-500 spectrometer. Elemental analyses were performed at the National Chen-Kung University, Tainan, Taiwan.

3.2. Preparation of **1**

$[Cu(CH_3CN)_4][BF_4]$ (100 mg, 0.32 mmol) and dpbp (167 mg, 0.32 mmol) were placed in an oven-dried 100 ml Schlenk flask, equipped with a magnetic stir bar and a rubber serum stopper. Freshly distilled dichloromethane (10 ml) was introduced into the flask via a syringe through the serum stopper. The mixture was stirred at room temperature for 24 h under a dinitrogen atmosphere. Toluene (20 ml) was then added to produce a white precipitate. The crude product was crystallized from acetonitrile/diethyl ether to afford air stable, colorless crystals of $[Cu_2(dpbp)_2(NCMe)_4][BF_4]_2$(**1**) (224 mg, 93% yield). *Anal.* Calc. for $C_{80}H_{68}B_2Cu_2F_8N_4P_4$: C, 63.63; H, 4.54; N, 3.71. Found: C, 63.62; H, 4,44; N, 3.30%. Mass (FAB): *m/z* 585 ($Cu_2dpbp_2{}^{2+}$, ^{63}Cu). ^{1}H NMR (CD_2Cl_2, 20 °C): δ 7.53–7.08 (m, 56H, Ph), 2.19 (s, 12H, MeCN). $^{31}P\{^1H\}$ NMR (CD_2Cl_2, 20 °C): δ 0.24 (br). UV–Vis (CH_3CN, 28 °C): 265 nm.

3.3. Preparation of **2**

Compound **1** (68 mg, 0.045 mmol), 2,2′-bibyridine (15 mg, 0.096 mmol) and dichloromethane (10 ml) were placed in an oven-dried 50 ml Schlenk flask. The mixture was stirred at room temperature for 6 h to result in a clear yellow solution. Toluene (20 ml) was then introduced to afford a yellow precipitate. The crude product was crystallized from dichloromethane/toluene at ambient temperature to give air-stable, bright yellow crystals of $[Cu_2(dpbp)_2(2,2'\text{-bipy})_2][BF_4]_2$ (**2**) (71 mg, 95% yield). *Anal.* Calc. for $C_{92}H_{72}B_2Cu_2F_8N_4P_4$: C, 66.64; H, 4.38; M, 3.38. Found: C, 66.61; H, 4.40; N, 3.32%. Mass (ESI): *m/z* 741.5 ($Cu_2(dpbp)_2(bipy)_2{}^{2+}$,^{63}Cu). ^{1}H NMR ($CD_2Cl_2$, 20 °C): δ 8.54 (d, $J = 5$ Hz, 4H), 8.32 (d, $J = 8$ Hz, 4H), 8.08 (dd, 4H), 7.51 (dd, 4H, py), 7.41–7.08 (m, 56H, Ph). $^{31}P\{^1H\}$ NMR (CD_2Cl_2, 20 °C): δ 1.99 (br). UV–Vis (CH_3CN, 28 °C): 276 nm.

3.4. Preparation of **3**

Compound **1** (30 mg, 0.020 mmol), 1,1′-bis(diphenylphosphino)ferrocene (23 mg, 0.042 mmol) and dichloromethane (5 ml) were placed in an oven-dried 50 ml Schlenk tube. The mixture was stirred at room temperature for 6 h to result in a clear orange solution. The solution was carefully layered with diethyl ether (15 ml) to afford air-stable, orange crystals of $[Cu_2(dpbp)(dppf)_2][BF_4]_2$ (**3**) (30 mg, 78% yield). *Anal.* Calc. for $C_{104}H_{84}B_2Cu_2F_8Fe_2P_6$: C, 64.65; H, 4.38. Found: C, 64.24; H, 4.40%. Mass (ESI): *m/z* 1139 ($Cu(dpbp)(dppf)^+$, ^{63}Cu). ^{1}H NMR (CD_2Cl_2, 20 °C): δ 7.48–7.14 (m, 68H, Ph), 4.51 (s, 8H), 4.24 (s, 8H, Cp). $^{31}P\{^1H\}$ NMR (CD_2Cl_2, 20 °C): δ 2.73 (t, $J_{P–P} = 95$ Hz, dpbp), −8.07 (d, dppf). UV–Vis (CH_3CN, 28 °C): 258 nm.

3.5. Preparation of **4**

Compound **1** (50 mg, 0.033 mmol), tetra(2-pyridyl)ethyl-1,4-diaminobutane (17 mg, 0.033 mmol) and dichloromethane (10 ml) were placed in an oven-dried 50 ml Schlenk tube. The mixture was stirred at room temperature for 8 h. Toluene (20 ml) was then introduced to afford a bright yellow precipitate. The crude product was crystallized from acetonitrile/toluene to give air-stable, pale-yellow crystals of $\{[Cu_2(dpbp)(tpyda)][BF_4]_2\}_n$ (**4**) (22 mg, 50% yield). *Anal.* Calc. for $\{C_{68}H_{68}B_2Cu_2F_8N_6P_2\}_n$: C, 61.32; H, 5.15; N, 6.31. Found: C, 61.28; H, 5.42; N, 5.93%. Mass (ESI): *m/z* 571 ($Cu(tpyda)^+$, ^{63}Cu). ^{1}H NMR (DMSO-d_6, 20 °C): δ 8.08–7.07 (44H, py, ph), 2.82 (br, 8H), 2.62 (br, 12H), 2.14 (br, 4H, CH_2). $^{31}P\{^1H\}$ NMR (CD_2Cl_2, 20 °C): $\delta - 0.55$ (br). UV–Vis (CH_3CN, 28 °C): 243, 261, 300 nm.

Table 1
Crystallographic data for **1** and **4**

	1	**4**
Formula	$C_{88}H_{88}B_2Cu_2F_8N_4O_2P_4$	$C_{76}H_{86}B_2Cu_2F_8N_{10}O_2P_2$
Formula weight	1658.20	1534.2
Crystal system	triclinic	monoclinic
Space group	$P\bar{1}$	$P2_1/n$
a (Å)	12.8191(7)	16.3114(9)
b (Å)	12.8777(7)	11.3801(7)
c (Å)	13.4393(7)	20.8164(11)
α (°)	96.264(1)	90
β (°)	90.147(1)	98.719(3)
γ (°)	114.140(1)	90
V (Å^3)	2009.7(2)	3819.4(4)
T (K)	150(1)	150(1)
Z	1	2
μ (mm^{-1})	0.678	0.670
Final R_1/wR_2	0.0609/0.1647	0.1354/0.3758

3.6. Structure determination for *1* and *4*

A crystal of **1** (ca. 0.27 × 0.25 × 0.25 mm) and **4** (ca. 0.20 × 0.15 × 0.15 mm) was each mounted in a thin-walled glass capillary and aligned on the Bruker SMART Apex CCD (for **1**) or the Nonius Kappa CCD (for **4**) diffractometer with graphite-monochromated Mo Kα radiation ($\lambda = 0.71073$ Å). The θ range for data collection is 1.74–27.50° for **1** and 2.05–25.00° for **4**. Of the 26722 and 22109 reflections collected for **1** and **4**, 9206 and 6717 reflections were independent, respectively. All data were corrected for Lorentz and polarization effects and for the effects of absorption. The structure was solved by the direct method and refined by full-matrix least-squares on F^2. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included but not refined. All calculations were performed using the SHELXTL-97 package [18]. The data collection and refinement parameters are presented in Table 1.

Acknowledgements

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Appendix A. Supplementary data

Crystallographic data for **1** and **4** have been deposited with the Cambridge Crystallographic Data Centre, deposition numbers CCDC 294172 and 294173. Copies of these data may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.ica.2006.05.007.

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