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Edwin C. Constable<sup>a</sup>; Catherine E. Housecroft<sup>a</sup>; Benson M. Kariuki<sup>b</sup>; Christopher B. Smith<sup>a</sup>

<sup>a</sup>Department of Chemistry, University of Basel, Basel, Switzerland <sup>b</sup>School of Chemistry, University of Birmingham, Edgbaston, Birmingham, UK

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# Switching on Hydrogen Bonding in Oligopyridine Ligands

EDWIN C. CONSTABLE<sup>a,\*</sup>, CATHERINE E. HOUSECROFT<sup>a</sup>, BENSON M. KARIUKI<sup>b</sup> and CHRISTOPHER B. SMITH<sup>a</sup>

<sup>a</sup>Department of Chemistry, University of Basel, Spitalstrasse 51, CH 4056 Basel, Switzerland; <sup>b</sup>School of Chemistry, University of Birmingham, Edgbaston, Birmingham BN5 2TT UK

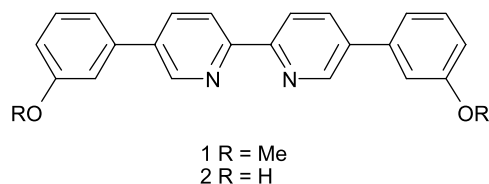
(Received 7 September 2005; Accepted 7 October 2005)

We have prepared two new 2,2'-bipyridine ligands bearing 3-aryl substituents at the 5 and 5'-positions using Suzuki methodology. The substituents differ only in the presence of a methoxy group or a phenolic hydroxy group. In the solid state a beautiful self-complementary hydrogen-bonded lattice incorporating one water molecule per ligand is formed with the phenolic compound.

**Keywords:** 2,2'-Bipyridine; Hydrogen-bonding; Crystal engineering; Crystal structure

## INTRODUCTION

In conventional coordination chemistry, ligands are designed to match the binding characteristics of the targeted metal ions and be complementary with the coordination number, coordination geometry and donor atom preferences of the metal [1–5]. Increasingly, in metallosupramolecular chemistry the tuning and optimisation of the metal-ligand interactions is sufficiently well understood that it is becoming secondary to designing and controlling specific intra- and inter-ligand interactions or molecular recognition events involving pendant functionality attached to the ligand skeleton. In this paper, we describe the consequences of replacing “innocent” methoxy substituents by hydroxy substituents in the free ligands **1** and **2** based upon 2,2'-bipyridine, one of the commonest metal-binding domains encountered in metallosupramolecular chemistry [6].



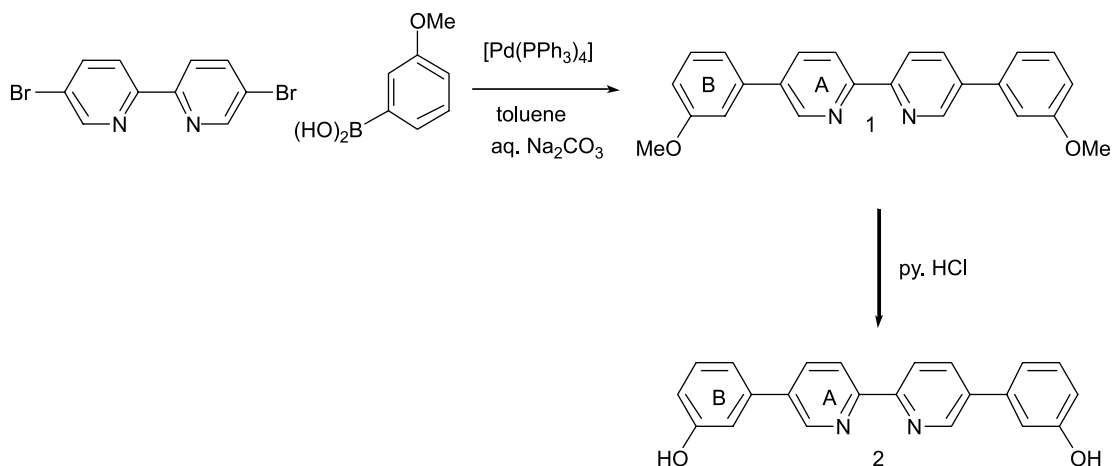
## RESULTS AND DISCUSSION

### Synthesis of the Ligands **1** and **2**

In the course of a synthetic programme designed to yield topologically novel knotted and catenated molecules assembled about a single metal centre, we required the functionalised 2,2'-bipyridines **1** and **2**. We recognised that these ligands would provide an ideal model system to investigate the effects of peripheral hydrogen bonding upon ligand and complex structure and conformation.

The bis(methoxy) compound **1** was prepared by Suzuki coupling of 5,5'-dibromo-2,2'-bipyridine [7–12] with 3-methoxyphenylboronic acid [13] in biphasic conditions in the presence of [Pd(PPh<sub>3</sub>)<sub>4</sub>] and was isolated in 86% yield as a white crystalline solid. Although Suzuki and Stille reactions of 3,8-dibromo-1,10-phenanthroline are well-established [14–22] and the coupling of 5,5'-dibromo-2,2'-bipyridine with alkynes has been demonstrated [23–30], only very few examples of arylation reactions involving 5,5'-dibromo-2,2'-bipyridine have been reported [12,31,32]. The compound exhibited the expected spectroscopic properties (see experimental section). Recrystallisation from ethyl acetate-toluene gave low quality crystals of **1** which were used for an X-ray structure determination. Conversion of the bis(methoxy) derivative **1** to the phenol **2** is conveniently achieved by heating the compound in molten pyridinium chloride at 210°C [33–35]. Despite the harsh reaction conditions, the phenol **2** was obtained in 88% yield. Recrystallisation of **2** from aqueous DMF gave X-ray quality crystals of 2.H<sub>2</sub>O. Synthetic transformations are summarised in Scheme 1.

\*Corresponding author. E-mail: edwin.constable@unibas.ch

SCHEME 1 Synthesis of **1** and **2** showing the labelling scheme adopted for the aromatic rings.

### Structural Characterisation of **1**

We have determined the solid state crystal structure of the methoxy compound **1** (Tables I and II). Although the crystals were poor, the structure is not of the highest quality, the contrasts with the hydroxy compound **2** are of interest. Fig. 1 shows the structure of the two independent molecules of **1** in the unit cell, together with the numbering scheme adopted. The two pyridine rings of **1** are coplanar and, as expected for a free ligand 2,2'-bipyridine [36–40], adopt a *trans* conformation. The 3-methoxyphenyl substituents are twisted with respect to the 2,2'-bipyridine with least squares planes angles of 32.2 and 32.4° in the two independent molecules; the methoxy-group of the aryl substituent lies towards

the same side of the molecule as the nitrogen of the pyridine ring to which it is attached. Bond lengths and angles within the molecule are typical and selected data are presented in Table II. Several types of aromatic-aromatic interactions [41,42] are present in the lattice; individual molecules form slightly offset face-to-face  $\pi$ -stacks with a direction of propagation along *b* with an interplanar distance of  $\approx 3.6$  Å (Fig. 2a). Individual stacks are then aligned beside each other, but with alternating directions of propagation to form ribbons decorated on the two outer surfaces with the methoxy groups, which form channels in the lattice (Fig. 2b). Within the ribbons there is a short contact of 2.55 Å between H24a and O1 (C24...O1, 3.39 Å;  $\angle$  C24–H24a–O1, 146.0°).

TABLE I Crystallographic data, data collection and refinement parameters for **1** and 2.H<sub>2</sub>O

	<b>1</b>		2.H <sub>2</sub> O
Empirical formula	C <sub>24</sub> H <sub>20</sub> N <sub>2</sub> O <sub>2</sub>		C <sub>22</sub> H <sub>18</sub> N <sub>2</sub> O <sub>3</sub>
Formula weight	368.42 g mol <sup>-1</sup>		358.38 g mol <sup>-1</sup>
Temperature	296(2)K		296(2)K
Wavelength	0.71069 Å		0.71069 Å
Crystal system	Monoclinic		Monoclinic
Space group	<i>P</i> 2 1/a		<i>P</i> 2 1/c
Unit cell dimensions			
<i>a</i> = 12.687(3) Å	$\alpha = 90^\circ$	<i>a</i> = 6.883(1) Å	$\alpha = 90^\circ$
<i>b</i> = 3.9673(7) Å	$\beta = 99.65(2)^\circ$	<i>b</i> = 13.568(2) Å	$\beta = 98.121(11)^\circ$
<i>c</i> = 37.275(6) Å	$\gamma = 90^\circ$	<i>c</i> = 19.129(4) Å	$\gamma = 90^\circ$
Volume, <i>Z</i>	1849.7(6) Å <sup>3</sup> , 4		1768.4(6) Å <sup>3</sup> , 4
Density (calculated)	1.323 mg m <sup>-3</sup>		1.346 mg m <sup>-3</sup>
Absorption coefficient	0.085 mm <sup>-1</sup>		0.091 mm <sup>-1</sup>
<i>F</i> (000)	776		752
Crystal size	0.50 × 0.10 × 0.10 mm		0.20 × 0.20 × 0.08 mm
$\theta$ range for data collection	0.55–22.06°		1.85–23.44°
Index ranges	$-13 \leq h \leq 13, -4 \leq k \leq 4, -39 \leq l \leq 38$		$-7 \leq h \leq 7, -15 \leq k \leq 13, -21 \leq l \leq 21$
Reflections collected	3987		6035
Independent reflections	1650		2274
Data/restraints/parameters	1650/0/254		2274/7/256
Goodness-of-fit on <i>F</i> <sup>2</sup> (all data)	1.174		1.077
Final <i>R</i> indices [ <i>I</i> > 2 $\sigma$ ]	<i>R</i> 1 = 0.1445, <i>wR</i> 2 = 0.3736		<i>R</i> 1 = 0.0770, <i>wR</i> 2 = 0.1343
<i>R</i> indices (all data)	<i>R</i> 1 = 0.1540, <i>wR</i> 2 = 0.3816		<i>R</i> 1 = 0.1737, <i>wR</i> 2 = 0.1769
Largest diff. peak and hole	0.647, -0.392 e Å <sup>-3</sup>		0.042, -0.178 e Å <sup>-3</sup>

TABLE II Selected bond distances (Å) and angles (°) in **1** and **2.H<sub>2</sub>O** with esds in parentheses

<b>1</b>		<b>2.H<sub>2</sub>O</b>	
O1–C1	1.379(14)	O1–C1	1.375(5)
O1–C12	1.422(15)	O2–C19	1.375(5)
O2–C13	1.379(13)	N1–C8	1.341(6)
O2–C24	1.463(14)	N1–C9	1.361(5)
N1–C8	1.336(14)	N2–C13	1.346(6)
N1–C9	1.365(14)	N2–C12	1.363(5)
N2–C20	1.346(14)		
N2–C21	1.363(14)		
C1–O1–C12	118.9(10)	C8–N1–C9	117.4(4)
C13–O2–C24	116.3(9)	C13–N2–C12	118.2(4)
C8–N1–C9	117.3(10)	C6–C1–O1	122.4(4)
C20–N2–C21	117.1(10)	O1–C1–C2	117.2(5)
C6–C1–O1	115.7(11)	N1–C8–C7	125.9(4)
C6–C1–C2	122.0(11)	N1–C9–C10	120.5(4)
O1–C1–C2	122.3(13)	N1–C9–C12	115.1(4)
N1–C8–C7	124.5(11)	N2–C12–C16	120.0(4)
C10–C9–N1	121.0(11)	N2–C12–C9	117.2(4)
N1–C9–C9a	117.7(12)	N2–C13–C14	126.3(4)
C7–C11–C10	118.9(12)	O2–C19–C18	117.7(5)
C14–C13–O2	126.1(11)	O2–C19–C20	121.9(4)
O2–C13–C18	113.3(10)		
N2–C20–C19	125.5(11)		
C22–C21–N2	120.4(10)		
N2–C21–C21a	116.8(11)		

### Structural Characterisation of **2.H<sub>2</sub>O**

Recrystallisation of **2** from aqueous DMF gave good quality crystals of the monohydrate, **2.H<sub>2</sub>O**. A view of the asymmetric unit in **2.H<sub>2</sub>O** is presented in Fig. 3.

Bond lengths and bond angles within the molecules are typical and selected values are presented in Table II. The bpy moiety exhibits, as expected [36–40], a *trans* conformation, but is not planar and the two pyridine rings make least squares planes angles of 24.73° with each other. In contrast, the 3-hydroxyphenyl substituents lie very close to the plane of the pyridine to which they are attached, exhibiting least squares planes angles of 4.62 and 12.54°. The phenolic hydroxy groups are directed toward the nitrogen of the pyridine ring to which the substituent is attached. The molecules of **2** are formed into ruffled sheets in the 011 plane.

The combination of one molecule of **2** with one water molecule leads to an elegant self-complementary hydrogen bonding system. Each molecule of **2** has two hydrogen bond acceptors (the pyridine nitrogen atoms) and two hydrogen bond donors (the two phenolic hydroxy groups); the water molecule can similarly present two acceptor and two donor sites. Water molecules link adjacent planes of molecules of **2** together in a lamellar [**2.H<sub>2</sub>O.2**]<sub>n</sub> arrangement. Each water molecule links two sheets of molecules of **2** by two donor interactions with one layer and two acceptor interactions with the other. The water is hydrogen bonded to the pyridine nitrogen of two adjacent molecules of **2** in the same layer (O...N 2.840, 2.810 Å, H...N 1.787, 1.821 Å, ∠N...H–O 167.3,

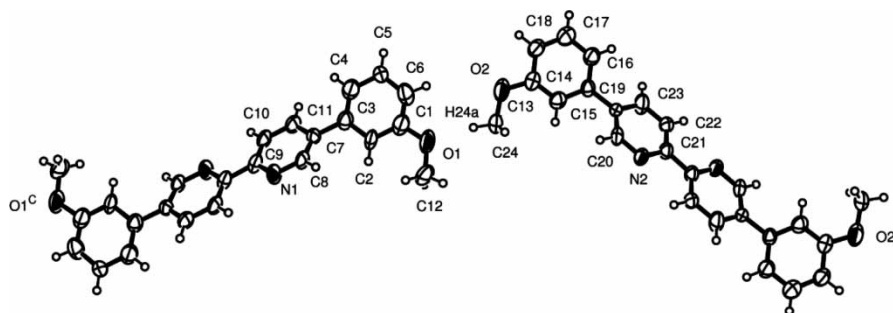


FIGURE 1 ORTEP representation of the two symmetry related molecules of **1** in the unit cell showing the numbering scheme adopted (50% probability thermal ellipsoids). Hydrogen atoms have been left unlabelled with the exception of H24a. The two oxygen atoms in the symmetry related half of each molecule have been labelled to indicate their symmetry: the symmetry label C relates to the symmetry operation  $-x, -y, -z$ .

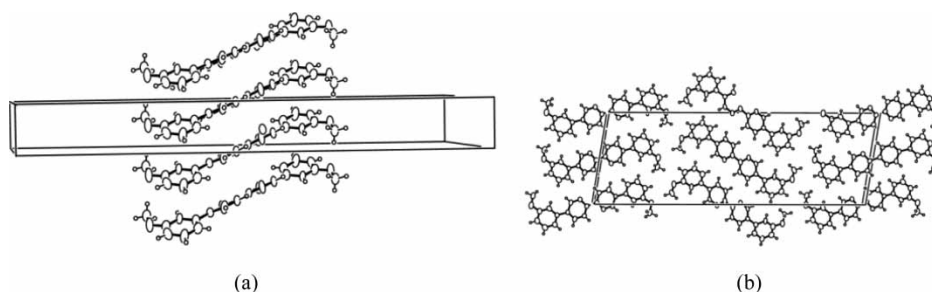


FIGURE 2 (a) Face-to-face stacking of molecules of **1** in the lattice; and (b) the channel structures formed by interdigitation of methoxy groups.

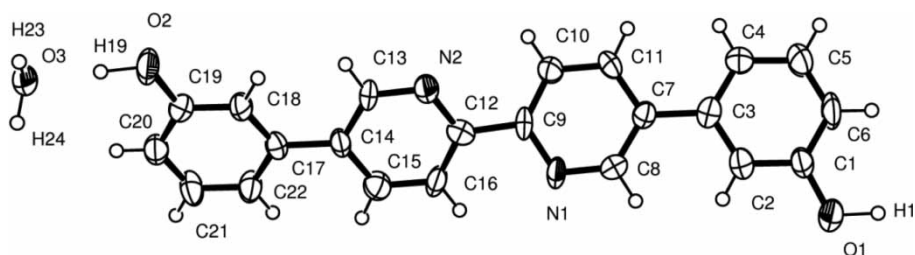


FIGURE 3 ORTEP representation of the asymmetric unit present in the unit cell of  $2 \cdot H_2O$  showing the numbering scheme adopted (50% probability thermal ellipsoids). Hydrogen atoms have been left unlabelled with the exception of H1, H19, H23 and H24.

$152.2^\circ$ ) and to two phenolic hydroxy groups of different molecules of **2** in the next layer (distances  $O \dots O$  2.791, 2.777 Å,  $O - H \dots O$  1.769, 1.721 Å,  $\angle O \dots H - O$  163.5,  $156.6^\circ$ ). The environment of the water molecule is presented in Fig. 4a and the packing of the  $[[2 \cdot H_2O \cdot 2] \{2 \cdot H_2O \cdot 2\} \dots]$  units in Fig. 4b. Hydrogen bonding data for  $2 \cdot H_2O$  are presented in Table III.

## CONCLUSIONS

The incorporation of aryl substituents at the 5- and 5'-positions of 2,2'-bipyridine using Suzuki coupling is facile. In the case of the compound 5,5'-bis(3'-hydroxyphenyl)-2,2'-bipyridine, a self-complementary hydrogen bonded monohydrate has been structurally characterised.

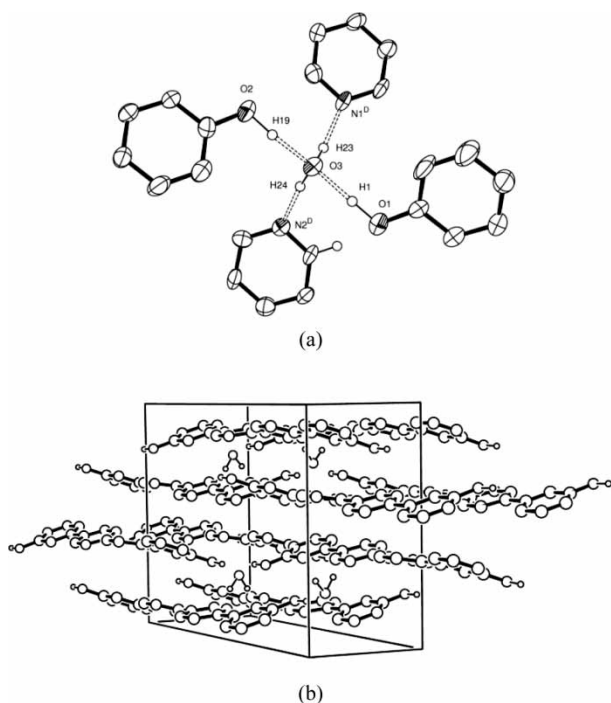


FIGURE 4 (a) ORTEP representation of the hydrogen bonding around the water molecule in  $2 \cdot H_2O$  (50% probability thermal ellipsoids). Atoms with the symmetry label D are generated by the symmetry operation  $x, -y - 1/2, z - 1/2$ ; (b) The arrangement of the lamellar  $[[2 \cdot H_2O \cdot 2] \{2 \cdot H_2O \cdot 2\} \dots]$  units in the lattice.

## EXPERIMENTAL

### 5,5'-Bis(3-methoxyphenyl)-2,2'-bipyridine **1**

3-Methoxyphenylboronic acid (1.44 g, 9.48 mmol) was added to 5,5'-dibromo-2,2'-bipyridine (1.19 g, 3.79 mmol) followed by toluene (80 mL) and aqueous sodium carbonate solution (2 M, 35 mL). The biphasic mixture was then degassed for 15 min by bubbling nitrogen through the solution after which time  $[Pd(PPh_3)_4]$  (6 mol% based on 5,5'-dibromo-2,2'-bipyridine, 0.26 g) was added, and the mixture heated to reflux. The reaction was monitored by TLC (silica, ethyl acetate/toluene 3:7), and judged to be complete after 18 h. After cooling to room temperature, the phases were separated and the aqueous layer extracted with  $CH_2Cl_2$  ( $3 \times 50$  mL). The organic extracts were combined, washed with water (100 mL), and then dried with  $MgSO_4$ . Removal of the solvent gave the crude product as an off-white powder, which was purified by flash chromatography on silica gel, eluting first with toluene to remove traces of high  $R_F$  impurities, followed by ethyl acetate/toluene (3:7) to give 5,5'-bis(3-methoxyphenyl)-2,2'-bipyridine **1** as white crystals (1.2 g, 86%);  $\delta_H$ /ppm (400 MHz,  $CDCl_3$ ,  $Me_4Si$ ) 8.94 (dd,  $J$  2.3, 0.6, 2H,  $H^{A6}$ ), 8.51 (dd,  $J$  8.2, 0.6, 2H,  $H^{A3}$ ), 8.04 (dd,  $J$  2.4, 8.3, 2H,  $H^{A4}$ ), 7.40 (t,  $J$  7.8, 2H,  $H^{B5}$ ), 7.26 (ddd,  $J$  7.5, 1.6, 1.0, 2H,  $H^{B6}$ ), 7.19 (d,  $J$  2.4, 1.6, 2H,  $H^{B2}$ ), 6.98 (ddd,  $J$  0.85, 2.5, 8.3, 2H,  $H^{B4}$ ), 3.90 (s, 6H); EI-MS  $m/z$  368( $M^+$ ). Found: C, 77.8; H, 5.5; N, 7.5.  $C_{24}H_{20}N_2O_2$  requires C, 78.2; H, 5.5; N, 7.6%.

### 5,5'-Bis(3-hydroxyphenyl)-2,2'-bipyridine **2**

Concentrated HCl (28.6 mL) was added slowly to pyridine (26 mL) with stirring to give a solution of pyridinium chloride. The mixture was heated to an internal temperature of  $210^\circ C$  and the water removed by distillation. The mixture was then cooled to  $140^\circ C$  and **1** (1.02 g, 2.77 mmol) was added. The yellow mixture was then heated to  $210^\circ C$  for 3 h after which time it was allowed to cool to  $120^\circ C$ , when water (50 mL) was added. This gave a yellow suspension of the crude product. After stirring overnight, the suspension was filtered, and the product washed with water (30–40 mL).



TABLE III Distances (Å) and angles (°) of hydrogen bond-type interactions in 2.H<sub>2</sub>O

D–H...A	Symmetry	Distance D...A	H...A	Angle D–H...A
O1–H1...O3	1 + x, y, -1 + z	2.791(5)	1.77(3)	157(5)
O2–H1...O3		2.777(5)	1.72(2)	164(4)
O3–H23...N1	-1 + x, 1/2 - y, 1/2 + z	2.811(5)	1.82(3)	152(2)
O3–H24...N2	x, 1/2 - y, 1/2 + z	2.840(5)	1.79(3)	167(3)

Recrystallisation of the solid from aqueous DMF gave 5,5'-bis(3''-hydroxyphenyl)-2,2'-bipyridine monohydrate 2.H<sub>2</sub>O. The anhydrous product **2** was obtained as pale brown crystals after drying the hydrate over P<sub>2</sub>O<sub>5</sub> under high vacuum (0.83 g, 88%);  $\delta_{\text{H}}$ /ppm (300 MHz, CD<sub>3</sub>SOCD<sub>3</sub>, Me<sub>4</sub>Si) 8.96 (dd, *J* 2.2, 0.7, 2H, H<sup>A6</sup>), 8.48 (d, *J* 8.1, 2H, H<sup>A3</sup>), 8.18 (dd, *J* 8.1, 2.2, 2H, H<sup>A4</sup>), 7.33 (t, *J* 8.1, 2H, H<sup>B5</sup>), 7.22 (dd, *J* 7.7, 0.7, 2H, H<sup>B6</sup>), 7.16 (dd, *J* 1.5, 2H, H<sup>B2</sup>), 6.85 (ddd, *J* 7.7, 2.2, 0.7, 2H, H<sup>B4</sup>). EI-MS *m/z* 340 (M<sup>+</sup>). Found C, 77.2; H, 4.6; N, 8.1. C<sub>22</sub>H<sub>16</sub>N<sub>2</sub>O<sub>2</sub> requires C, 77.6; H, 4.7; N, 8.2%.

### X-ray Crystallographic Data and Structure Determination

The intensity data were collected at ambient temperature (296 K) on a Rigaku R-axis IIC diffractometer using Mo K $\alpha$  radiation ( $\lambda$  0.71069 Å) and image plate scans. The structures were solved by direct methods using SHELXS-86 [43] and refined anisotropically (non-hydrogen atoms) by full-matrix least-squares on *F*<sup>2</sup> using SHELX-93 [44]. The H-atom positions were calculated geometrically and refined with a riding model. Crystallographic data and final refinement details are given in Table I. Crystallographic data (excluding structure factors) for the structures have been deposited with the Cambridge Crystallographic Data Centre (CCDC 281335 and 281334 for **1** and 2.H<sub>2</sub>O respectively). Copies of the data can be obtained free of charge at [www.ccdc.cam.ac.uk/products/csd/request/](http://www.ccdc.cam.ac.uk/products/csd/request/) or from the CCDC, 12 Union Road, Cambridge CB2 1EW, UK; fax: (+44) 1223-336-033; or [deposit@ccdc.cam.ac.uk](mailto:deposit@ccdc.cam.ac.uk).

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