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Comparison of two polymeric transition metal complexes with 1,4-benzenedicarboxylate ions as bridging ligands, $[Co(C_8H_4O_4)(C_{12}H_8N_2)(H_2O)]$ and $[Cu(C_8H_4O_4)(C_{10}H_9N_3)]\cdot H_2O$

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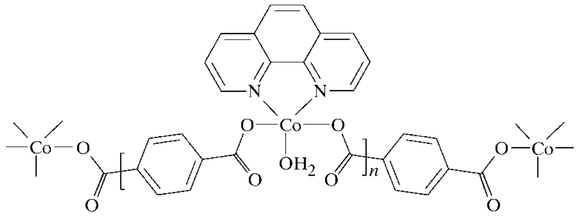
The title complexes, *catena*-poly[[aqua(1,10-phenanthroline-κ^2N,N')cobalt(II)]-μ-benzene-1,4-dicarboxylato-κ^2O^1:O^4], $[Co(C_8H_4O_4)(C_{12}H_8N_2)(H_2O)]$, (I), and *catena*-poly[[[(di-2-pyridyl-κN-amine)copper(II)]-μ-benzene-1,4-dicarboxylato-$\kappa^4O^1,O^{1'}$:$O^4,O^{4'}$] hydrate], $[Cu(C_8H_4O_4)(C_{10}H_9N_3)]\cdot H_2O$, (II), take the form of zigzag chains, with the 1,4-benzenedicarboxylate ion acting as an amphimonodentate ligand in (I) and a bis-bidentate ligand in (II). The Co^{II} ion in (I) is five-coordinate and has a distorted trigonal–bipyramidal geometry. The Cu^{II} ion in (II) is in a very distorted octahedral 4+2 environment, with the octahedron elongated along the *trans* O—Cu—O bonds and with a *trans* O—Cu—O angle of only 137.22 (8)°.

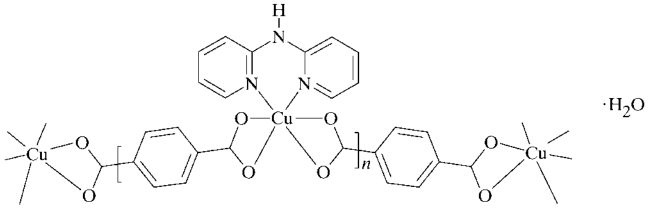
Comment

The dianion of 1,4-benzenedicarboxylic acid (or terephthalic acid, H_2tpht), is a potential bis-bidentate and bridging ligand. Although an example of a copper(II) compound containing uncoordinated tpht has been described recently by Huaqiang *et al.* (1997), in the case of first-row transition metal complexes, tpht ions typically act as bis-bidentate (Verdaguer *et al.*, 1984; Deng *et al.*, 1992; Cano *et al.*, 1994; Sun *et al.*, 2001), tridentate (Bakalbassis *et al.*, 1992) or bidentate (Verdaguer *et al.*, 1984; Chaudhuri *et al.*, 1988; Bakalbassis *et al.*, 1991; Cueto *et al.*, 1991; Xanthopoulos *et al.*, 1993; Rogan *et al.*, 2000; Kim *et al.*, 2001; Sun *et al.*, 2001) ligands.

With the exception of rare discrete complexes (Rogan *et al.*, 2000) where tpht is coordinated by only one of its COO groups, tpht complexes are binuclear or polymeric in nature. Interest in such complexes is related to molecular magnetism, a continually growing field of research in modern inorganic chemistry and materials science. It is well documented that two paramagnetic centres could interact through extended bridging ligands. In the case of Cu–tpht complexes, this was initially investigated by Verdaguer *et al.* (1984), but the magnetic interactions were weak. Very shortly afterwards, some binuclear Cu^{II} complexes with unexpectedly strong antiferromagnetic interactions were described (Bakalbassis *et al.*, 1985; Chaudhuri *et al.*, 1988). Hence, magnetic properties, together with an orbital interpretation of the magnetic exchange mechanism, were also discussed in many of the papers cited above. Here, two ternary tpht complexes, $[Co(H_2O)(phen)(tpht)]$, (I), and $[Cu(dipya)(tpht)]\cdot H_2O$, (II), containing Co^{II} or Cu^{II} ions and 1,10-phenanthroline (phen) or 2,2′-dipyridylamine (dipya), are presented.



(I)



(II)

The structure of complex (I) has recently been solved using low-temperature data collected at 193.2 K (Sun *et al.*, 2001). However, an unusually high *S* value (2.52), some duplicated H atoms detected in the corresponding CIF and an inability to locate the water H atoms led us to prepare the same compound by a different procedure and to collect a set of data at room temperature. In addition, for reasons of comparison, it was necessary to study both the structures described here at the same temperature.

The two reported structures of (I) are found to be essentially identical. The change of temperature caused very small variations of the unit-cell parameters (0.11% for *a*, −0.74% for *b*, −0.69% for *c* and −1.28% for the unit-cell volume). As expected, the atomic displacement parameters have lower values and more isotropic character in the low-temperature data set.

Due to the bridging role of the tpht ions, both (I) and (II) are polymeric and can be described as zigzag chains, with

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neighbouring diamine ligands *trans* to each other (Figs. 1 and 2). Very similar chains are found in [Zn(H_2O)(phen)(tpht)], which is isostructural with (I), and in [Cu(phen)(tpht)] (Sun *et al.*, 2001), as well as in [Cu(en)(H_2O)(tpht)] (en is ethylenediamine; Bakalbassis *et al.*, 1988). In all these complexes, the metal centres are bridged by tpht ions coordinated in an amphimonodentate fashion.

Although the orientation of the chains in (I) and (II) is quite different, being parallel to [211] in (I) and to [$1\bar{1}1$] in (II), the unit-cell volumes (see *Crystal data*) are very similar; the volume of (I) is slightly larger, due to the bulkier phen ligand. Intrachain Co···Co distances in (I) are alternately 11.063 and 11.289 Å, while the shortest interchain Co···Co distance is 5.908 Å. The corresponding Cu···Cu distances in (II) are 10.706, 10.898 and 5.153 Å, respectively. These values are quite normal for tpht-bridged complexes, and therefore both intra- and interchain magnetic interactions might be expected. The shortest intrachain metal–metal distances in [Co(H_2O)(phen)(tpht)], [Zn(H_2O)(phen)(tpht)] and [Cu(phen)(tpht)] are 11.040, 10.853 and 11.094 Å, respectively (Sun *et al.*, 2001).

In both complexes, two crystallographically different but chemically identical tpht ions exist. Since the crystallographic inversion centres coincide with the centres of the aromatic rings, only half of each tpht ion belongs to the asymmetric unit. The dihedral angles between the two tpht aromatic rings are almost identical for both compounds, with a value of 79.3 (3)° in (I) and 79.0 (3)° in (II).

In (I), the tpht ions do not deviate very much from planarity, with an acute dihedral angle between the C14–C16 aromatic ring and the adjacent COO group of 6.8 (2)°. The corresponding angle for the C18–C20 ring is 7.3 (3)°. In (II), the analogous angles differ more; that for C12–C14 is 17.3 (2)° and that for C16–C18 is only 4.7 (3)°. In two recently published series of tpht complexes (Rogan *et al.*, 2000; Sun *et al.*, 2001), the corresponding angles are relatively small, ranging between 3.5 and 22.3°. Nevertheless, angles up to 51.9° have been found in some Cu complexes containing additional triamine ligands (Verdaguer *et al.*, 1984; Bakalbassis *et al.*, 1991). Besides the presence of the four O atoms as potential donor sites, it seems that easy rotation around the $C_{\rm aromatic}$—$C_{\rm carboxylate}$ bonds has a great influence on the coordination of tpht ions and on the resulting structures, which cover a wide range from discrete mononuclear complex entities, through binuclear units and chains, up to three-dimensional network structures. According to Kaduk (Kaduk, 2000; Kaduk & Golab, 1999), the completely planar conformation of tpht ions has a minimum energy. However, an increase in the angle of (both) COO groups up to 30° requires an energy increase of less than 20 kJ mol^{-1}. This could easily be compensated for by more favourable coordination geometries and crystal packing.

The main difference between (I) and (II) is the denticity of the tpht ligands, which are bis-bidentate in (II) and only amphimonodentate in (I). As a result, the coordination polyhedra are also very different. In (I), the Co atom is surrounded by five atoms in a deformed trigonal–bipyramidal arrangement, with atoms O1, N1 and O*W*1 in the equatorial plane (Table 1). The large O1—Co—O*W*1 angle could be a consequence of the hydrogen bond mentioned below, with the participation of atoms O2 and O*W*1. A long Co···O2 contact of 2.653 (3) Å, which is not usually regarded as a Co—O bond but which is significantly shorter than the sum of van der Waals radii, should also be noted. The dihedral angle between

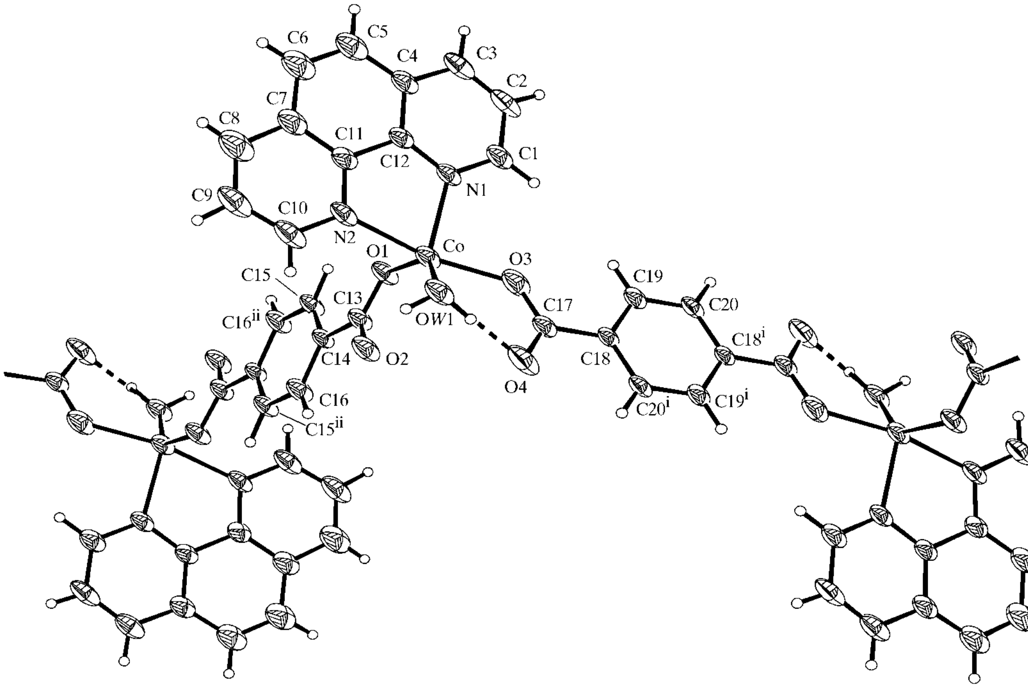

Figure 1
Part of the polymeric chain of complex (I) with the atom-numbering scheme. The intramolecular hydrogen bond is represented by a dashed line. Displacement ellipsoids are plotted at the 30% probability level and H atoms are shown as small spheres of arbitrary radii [symmetry codes: (i) $-x$, $2-y$, $1-z$; (ii) $2-x$, $3-y$, $2-z$].

COO groups coordinated to the same Co atom is 80.2 (3)°. No significant variations are observed for the Co—O and Co—N bond distances in (I) and in the previously reported structure (Sun *et al.*, 2001).

In (II), the Cu atoms are in a 4+2 environment (Table 3), which, under usual circumstances, should be close to an elongated octahedron. However, to our knowledge, this is the first example of a tpht complex with two COO groups chelating to the same central atom. Due to the constraints imposed by such coordination and the formation of two four-membered rings with O—Cu—O angles less than 60° (Table 3), the coordination polyhedron is highly deformed. For example, the O2—Cu—O4 angle is only 137.22 (8)° (the octahedron is elongated along O2—Cu—O4). In addition, the maximum displacement from the equatorial Cu/N1/N2/O1/O3 plane is 0.611 (2) Å (for atom N2) and the distribution of ligating atoms in the N1/N2/O1/O3 plane is strongly puckered, with an average displacement of 0.426 (2) Å. The coordinated COO groups are almost perpendicular to each other, with a dihedral angle of 88.9 (3)°.

The bond distances and angles within the ligands in (I) and (II) are similar to the values found in the free compounds (H_2tpht: Bailey & Brown, 1967; phen: Nishigaki *et al.*, 1978; dipya: Johnson & Jacobson, 1973) and will not be discussed in detail, although there are two points worthy of note. First, the N1/C1–C4 segment of the phen ligand in (I), where, for example, atom C2 deviates from the plane of the ligand by 0.056 (4) Å (*ca* 13σ), should be mentioned. This could be accounted for by some thermal motion or slight disorder, but this part of the ligand is sandwiched between two other aromatic rings from neighbouring chains, and so van der Waals and/or π interactions cannot be excluded. Secondly, the dihedral angle between the two pyridine rings of dipya in (II) is 14.5 (1)°, which is less than in dipya alone (23°; Johnson & Jacobson, 1973), but is within the range of values already found for copper(II) complexes (Poleti *et al.*, 1990).

There are two hydrogen bonds in complex (I) and three in complex (II) (Tables 2 and 4). In (I), the coordinated water molecule is a double hydrogen-bond donor. One of the hydrogen bonds is intramolecular (Fig. 1 and Table 2), while

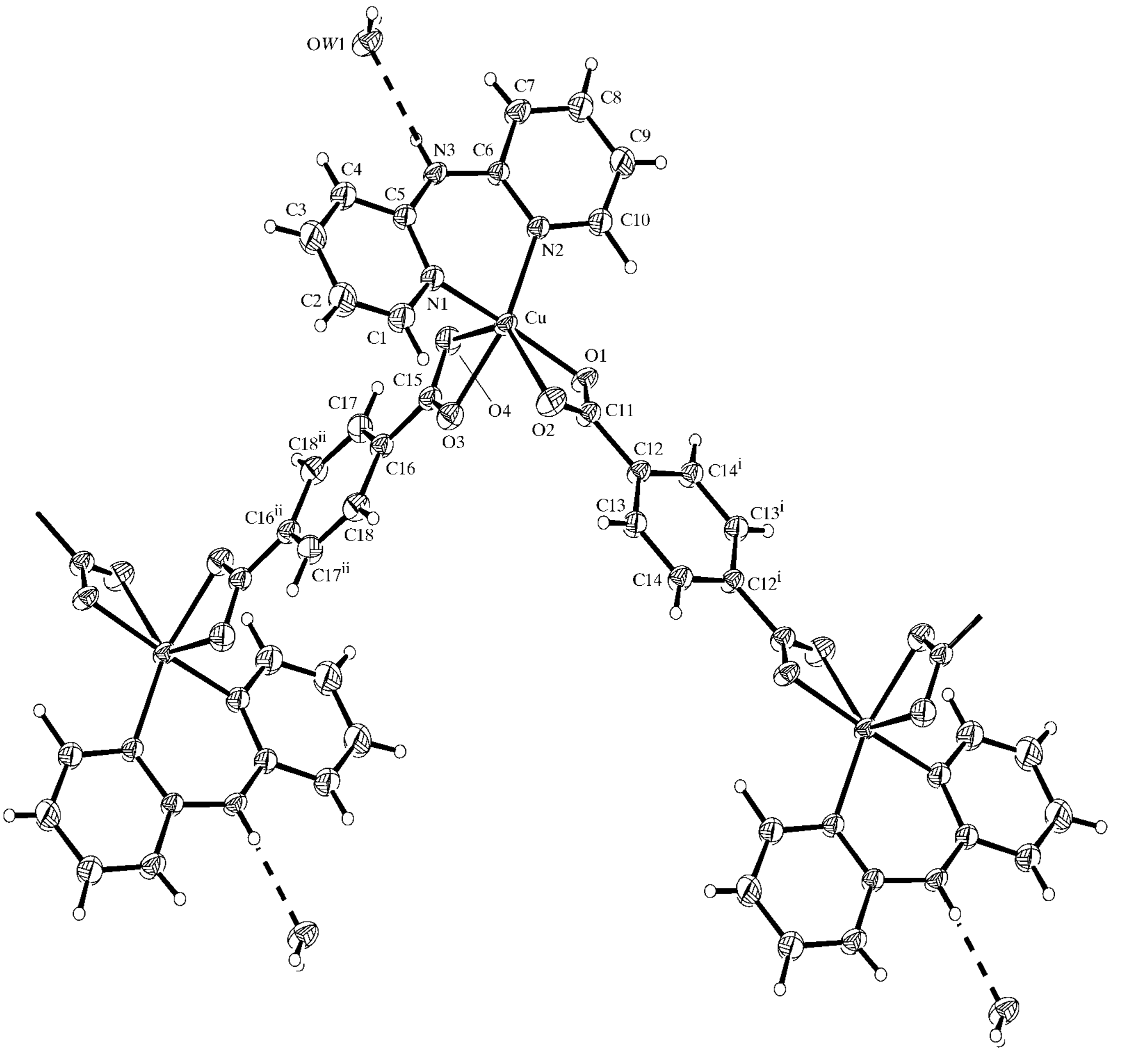

Figure 2
Part of the polymeric chain of complex (II) with the atom-numbering scheme. The N3—H···*O*W1 hydrogen bond is represented by a dashed line. Displacement ellipsoids are plotted at the 30% probability level and H atoms are shown as small spheres of arbitrary radii [symmetry codes: (i) $-x$, $2-y$, $1-z$; (ii) $1-x$, $1-y$, $2-z$].

the other connects adjacent chains. In (II), the water of crystallization acts as a double hydrogen-bond donor to the carboxylate O atoms, and as a hydrogen-bond acceptor from the amine H atom of dipya (Fig. 2 and Table 4).

Experimental

Because the title complexes are insoluble in all common solvents, single crystals were prepared by a modification of the slow diffusion method. Typically, a dilute dimethyl sulfoxide (DMSO) solution (~0.02 mol dm^{-3}), containing equimolar quantities of cobalt(II) or copper(II) nitrate, aromatic amine and H_2tpht, was prepared in a small test tube. A dilute solution of Na_2tpht in H_2O was then layered carefully and very slowly in order to minimize mixing of the solutions. After approximately one week, single crystals of suitable size had formed near the solution boundary. In the case of the copper(II) system, two kinds of crystals, one dark-blue and one green, were obtained. The dark-blue crystals transformed slowly to the green phase and, according to their IR spectra, very probably contained DMSO as an additional constituent. Due to their instability, these crystals were not characterized further.

Compound (I)

Crystal data

$[Co(C_8H_4O_4)(C_{12}H_8N_2)(H_2O)]$
$M_r = 421.26$
Triclinic, $P\overline{1}$
$a = 9.2688$ (17) Å
$b = 10.4550$ (18) Å
$c = 11.349$ (2) Å
$\alpha = 112.462$ (3)°
$\beta = 94.924$ (2)°
$\gamma = 113.908$ (2)°
$V = 890.6$ (3) Å^3
$Z = 2$
$D_x = 1.571$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 954 reflections
$\theta = 3.5$–24.2°
$\mu = 1.00$ mm^{-1}
$T = 298$ (1) K
Irregular, brown–purple
0.27 × 0.22 × 0.18 mm

Data collection

Bruker CCD area-detector diffractometer
φ and ω scans
Absorption correction: empirical (*XPREP* in *SHELXTL*; Bruker, 1997)
$T_{\min} = 0.736$, $T_{\max} = 0.864$
4652 measured reflections
3128 independent reflections
2012 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.032$
$\theta_{\max} = 25.5°$
$h = -11 \rightarrow 10$
$k = -10 \rightarrow 12$
$l = -10 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.060$
$S = 0.88$
3128 reflections
309 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0171P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.22$ e Å^{-3}
$\Delta\rho_{\min} = -0.25$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °) for (I).

Co—O3	2.013 (2)	Co—N1	2.102 (2)
Co—O1	2.044 (2)	Co—N2	2.142 (2)
Co—O*W*1	2.061 (2)		
O3—Co—O1	99.03 (8)	O*W*1—Co—N1	124.44 (8)
O3—Co—O*W*1	89.2 (1)	O3—Co—N2	167.33 (8)
O1—Co—O*W*1	140.21 (8)	O1—Co—N2	90.04 (8)
O3—Co—N1	92.54 (9)	O*W*1—Co—N2	89.5 (1)
O1—Co—N1	94.22 (8)	N1—Co—N2	77.89 (9)

Table 2
Hydrogen-bonding geometry (Å, °) for (I).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O*W*1—H*W*1···O4	0.93 (4)	1.70 (4)	2.608 (4)	164 (4)
O*W*1—H*W*2···O2$^{\rm i}$	0.88 (5)	1.82 (5)	2.696 (3)	172 (4)

Symmetry code: (i) $1 - x, 2 - y, 2 - z$.

Compound (II)

Crystal data

$[Cu(C_8H_4O_4)(C_{10}H_9N_3)]\cdot H_2O$
$M_r = 416.87$
Triclinic, $P\overline{1}$
$a = 9.009$ (4) Å
$b = 9.289$ (3) Å
$c = 11.171$ (6) Å
$\alpha = 99.11$ (4)°
$\beta = 106.64$ (4)°
$\gamma = 92.39$ (3)°
$V = 880.6$ (7) Å^3
$Z = 2$
$D_x = 1.572$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 23 reflections
$\theta = 13.2$–16.2°
$\mu = 1.28$ mm^{-1}
$T = 293$ (2) K
Prism, green
0.31 × 0.18 × 0.08 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
4069 measured reflections
3746 independent reflections
2814 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.012$
$\theta_{\max} = 27°$
$h = 0 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = -14 \rightarrow 13$
2 standard reflections frequency: 1 min intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.100$
$S = 1.01$
3746 reflections
296 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0512P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.37$ e Å^{-3}
$\Delta\rho_{\min} = -0.51$ e Å^{-3}

Table 3
Selected geometric parameters (Å, °) for (II).

Cu—N1	1.951 (2)	Cu—O3	2.070 (2)
Cu—O1	1.955 (2)	Cu—O2	2.412 (2)
Cu—N2	1.991 (2)	Cu—O4	2.440 (2)
N1—Cu—O1	162.91 (9)	N2—Cu—O2	127.75 (9)
N1—Cu—N2	91.8 (1)	O3—Cu—O2	83.35 (9)
O1—Cu—N2	96.59 (9)	N1—Cu—O4	93.3 (1)
N1—Cu—O3	89.80 (9)	O1—Cu—O4	101.57 (9)
O1—Cu—O3	91.12 (9)	N2—Cu—O4	89.67 (9)
N2—Cu—O3	147.15 (9)	O3—Cu—O4	57.48 (8)
N1—Cu—O2	103.9 (1)	O2—Cu—O4	137.22 (8)
O1—Cu—O2	59.29 (8)		

Table 4
Hydrogen-bonding geometry (Å, °) for (II).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—HN3···O*W*1	0.76 (3)	2.06 (3)	2.812 (4)	171 (4)
O*W*1—H*W*1···O4$^{\rm i}$	0.85	1.95	2.743 (4)	155
O*W*1—H*W*2···O1$^{\rm ii}$	0.85	2.07	2.920 (4)	174

Symmetry codes: (i) $2 - x, 2 - y, 2 - z$; (ii) $1 + x, y, z$.

For both compounds, all H atoms were found in difference Fourier maps and were refined isotropically with no constraints. For compound (II), however, the final geometry of the water molecule was not satisfactory. Because of this, the positions of the water H atoms, H*W*1 and H*W*2, were recalculated using the program *HYDROGEN* (Nardelli, 1999) after the final cycle of refinement.

For compound (I), data collection: *SMART-NT* (Bruker, 1998); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SHELXTL* (Bruker, 1997); molecular graphics: *ORTEX*8a (McArdle, 1995; Burnett & Johnson, 1996). For compound (II), data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: local modification of *MolEN* (Fair, 1990); molecular graphics: *ORTEX*7e (McArdle, 1995; Burnett & Johnson, 1996). For both compounds, program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1983, 1995).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1551). Services for accessing these data are described at the back of the journal.

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