

Hydrogen-bonded layers directed by the $[3\text{-O}_3\text{S-C}_6\text{H}_4\text{-PO}_3\text{H}]^{2-}$ dianion: *catena*-poly[[silver(I)- μ -4,4'-bipyridine- $\kappa^2\text{N:N}'$] 3-[hydroxy(oxido)phosphinoyl]benzenesulfonate trihydrate] and *catena*-poly[[[tetraaquacobalt(II)]- μ -4,4'-bipyridine- $\kappa^2\text{N:N}'$] 3-[hydroxy(oxido)phosphinoyl]benzenesulfonate]

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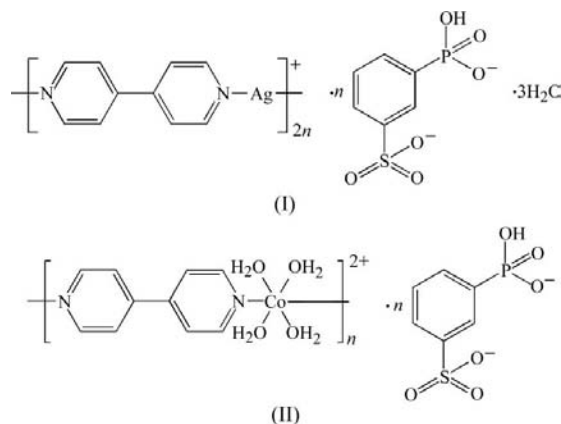
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Both title compounds, $\{[\text{Ag}_2(\text{C}_{10}\text{H}_8\text{N}_2)_2](\text{C}_6\text{H}_5\text{O}_6\text{PS})\cdot 3\text{H}_2\text{O}\}_n$ (I), and $\{[\text{Co}(\text{C}_{10}\text{H}_8\text{N}_2)_2(\text{H}_2\text{O})_4](\text{C}_6\text{H}_5\text{O}_6\text{PS})\}_n$ (II), respectively, contain similar novel symmetric dimeric $[\text{O}_3\text{S-C}_6\text{H}_4\text{-PO}_3\text{H}]^{2-}$ anions formed *via* two shared (O...H...O) H atoms on twofold positions between two $-\text{PO}_3\text{H}^-$ groups. The two-coordinate Ag^{I} structure features one-dimensional polymeric chains of $\{[\text{Ag}(4,4'\text{-bipy})]_2^+\}_n$ (4,4'-bipy is 4,4'-bipyridine), weakly linked by π - π interactions, separated by the anionic dimers which are stabilized into layers by hydrogen bonding to three water molecules. In (II), a twofold crystallographic axis runs through the one-dimensional $\{[\text{Co}(4,4'\text{-bipy})(\text{H}_2\text{O})_4]^{2+}\}_n$ chains containing six-coordinate Co^{II} ; three-dimensional packing is provided by hydrogen bonding using all sulfonate and $-\text{PO}_3\text{H}^-$ O atoms as acceptors and the Co-bound water H atoms as donors. In this latter case, the benzenesulfonate aromatic rings are also constrained to a mirror plane. This report illustrates how a previously unreported dianion can affect the crystallization of polymeric metal complex cations.

Comment

Over the past few years, interest in the design, synthesis, characterization and functions of hybrid inorganic-organic supramolecular compounds has been growing (Lehn, 1995). Usually, hydrogen-bonding and π - π stacking interactions are important in solid-state assembly. In the case of ionic supramolecular compounds, the counter-ions play an important role

in the stabilization of their structures through electrostatic interaction (Bianchi *et al.*, 1997; Min & Suh, 2000; Noro *et al.*, 2002). In general, small anions, such as NO_3^- , BF_4^- , ClO_4^- , PF_6^- , *etc.*, are used as counter-ions for cationic metal components. Reports on the use of phosphonic acids as a building block for supramolecular complexes are rare, although they have been shown to form extremely strong hydrogen bonds in their solid-state structures (Clearfield *et al.*, 2001; Sharma & Clearfield, 2001). 3-Sulfophenylphosphonic acid, $\text{HO}_3\text{S-C}_6\text{H}_4\text{-PO}_3\text{H}_2$, the first sulfonate-phosphonate ligand to be investigated, has recently been employed as a metal-organic coordination fragment due to its diverse coordination modes (Du *et al.*, 2006*a,b*; Du, Li *et al.*, 2007; Du, Prosvirin & Mao, 2007; Du, Xie & Wen, 2007; Du, Xu *et al.*, 2007). When the deprotonated 3-sulfophenylphosphonic acid is not coordinated, it can also be employed as an organic counter-ion for constructing hybrid inorganic-organic supramolecular arrays. We report here the two title compounds, (I) and (II), which illustrate two novel lattice architectures resulting from the association of metal-organic units with the $[\text{O}_3\text{S-C}_6\text{H}_4\text{-PO}_3\text{H}]^{2-}$ anion and water molecules.



The structure of compound (I) features one-dimensional chains of $\{[\text{Ag}(4,4'\text{-bipy})]_2^+\}_n$ (4,4'-bipy is 4,4'-bipyridine) with $[\text{O}_3\text{S-C}_6\text{H}_4\text{-PO}_3\text{H}]^{2-}$ anions as counter-ions. There are two independent Ag^{I} ions in the asymmetric unit of (I) (Fig. 1). Both Ag^{I} ions are two-coordinated by two N atoms from two 4,4'-bipy ligands in a linear geometry (Table 1). The interconnection of the Ag^{I} ions by the bidentate bridging 4,4'-bipy ligands results in the formation of one-dimensional chains along the *c* axis. These chains are further assembled into a two-dimensional supramolecular layered architecture *via* weak π - π packing interactions (Fig. 2). One aryl ring in the 4,4'-bipy ligand is disordered, displaying two ring orientations (*C7A/C8A/C9/C15A/C16A/N1* and *C7B/C8B/C9/C15B/C16B/N1*) (Fig. 1).

The $[\text{O}_3\text{S-C}_6\text{H}_4\text{-PO}_3\text{H}]^{2-}$ anion in (I) forms a dimer through two short hydrogen bonds between two pairs of twofold symmetry-related $-\text{PO}_3\text{H}^-$ O atoms, with the bonded H atoms (H4 and H5) located on a twofold axis (Fig. 3*a* and Table 2). This is a unique arrangement, with the most similar example being seen in diprotonated $-\text{PO}_3\text{H}^-$ groups (see, for example, Clarke *et al.*, 2005). It is noted that symmetrical hydrogen bonds and hydrogen-bonded dimers with similar

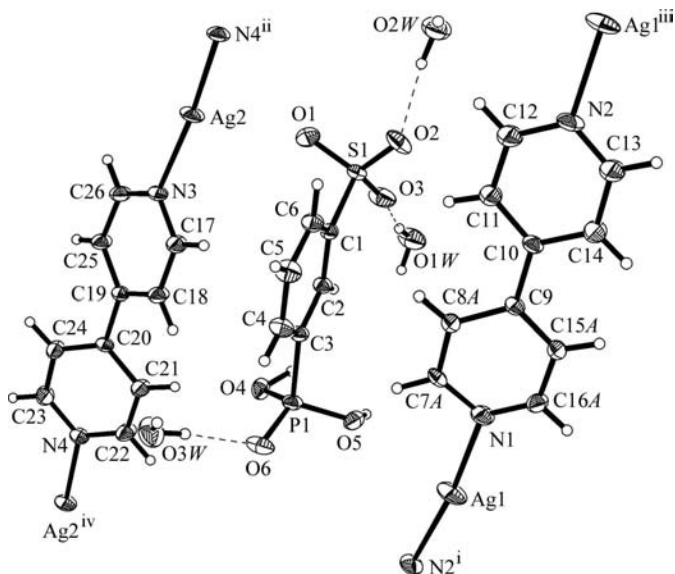


Figure 1
The structure of compound (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii. For the sake of clarity, only one orientation is shown for the disordered aryl ring (see *Comment*). Hydrogen bonds are represented by dashed lines. [Symmetry codes: (i) $x, 1 - y, -\frac{1}{2} + z$; (ii) $x, -y, \frac{1}{2} + z$; (iii) $x, 1 - y, \frac{1}{2} + z$; (iv) $x, -y, -\frac{1}{2} + z$.]

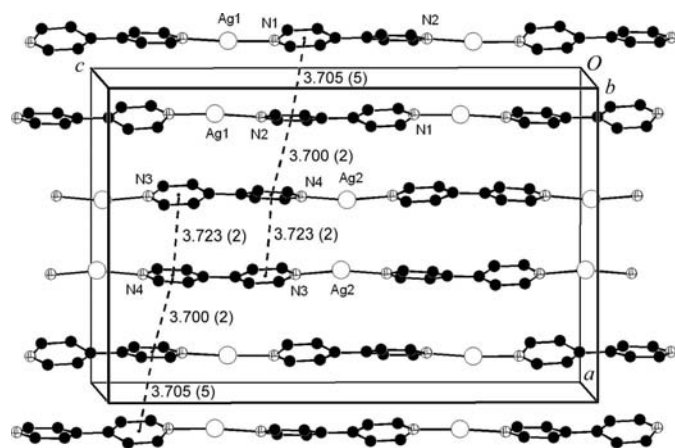


Figure 2
A view of the two-dimensional supramolecular layered architecture of $\{[Ag(C_{10}H_8N_2)]_2^+\}_n$ in (I) down the b axis, showing the weak π - π interactions (dashed lines; distances in Å). For clarity, generalized atom labels without symmetry codes are used. Ag, N and C atoms are drawn as white, octant and black spheres, respectively.

dimensions of other phosphonic acids have been reported previously (Sharma & Clearfield, 2000*a,b*; Bowes *et al.*, 2003; Man *et al.*, 2006; Courtney *et al.*, 2006; Latham *et al.*, 2007). The dimer is further stabilized by extensive hydrogen bonding with solvent water molecules, using one $-PO_3H^-$ and two sulfonate O atoms as acceptors (Table 2). Coupled with this is a cyclic water tetramer formed by hydrogen-bonding associations between two O2W atoms and two O3W atoms (Fig. 4 and Table 1); the four O atoms are not coplanar, with the torsion angle $O2W^{ii}-O3W^{iv}-O2W^{iii}-O3W$ being *ca* 42.6°

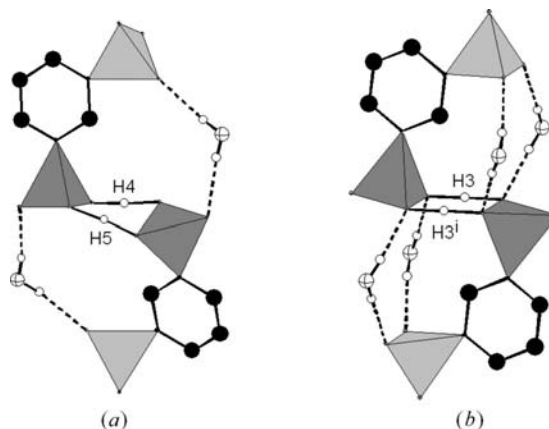


Figure 3
The dimer of $[O_3S-C_6H_4-PO_3H]^{2-}$ anions in (a) compound (I) and (b) compound (II). H atoms attached to aryl C atoms have been omitted for clarity. The CPO_3 and CSO_3 groups are shaded in medium and light grey, respectively. O, C and H atoms are drawn as crossed, black and white spheres, respectively. Hydrogen bonds between $[O_3S-C_6H_4-PO_3H]^{2-}$ anions and water molecules are represented by dashed lines. [Symmetry code: (i) $-x, 1 - y, -z$.]

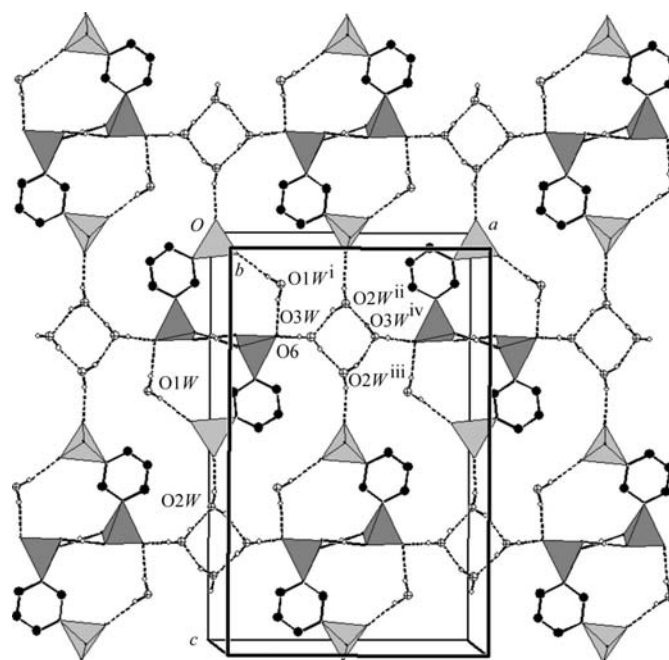


Figure 4
A view of the two-dimensional supramolecular layered architecture of $[(O_3S-C_6H_4-PO_3H)^{2-} \cdot 3H_2O]_n$ in (I) down the b axis. For display details, see the caption for Fig. 3. [Symmetry codes: (i) $-x, y, \frac{1}{2} - z$; (ii) $\frac{1}{2} - x, \frac{1}{2} - y, -\frac{1}{2} + z$; (iii) $\frac{1}{2} + x, \frac{1}{2} - y, 1 - z$; (iv) $1 - x, y, \frac{1}{2} - z$.]

[symmetry codes: (ii) $\frac{1}{2} - x, \frac{1}{2} - y, -\frac{1}{2} + z$; (iii) $\frac{1}{2} + x, \frac{1}{2} - y, 1 - z$; (iv) $1 - x, y, \frac{1}{2} - z$]. All water molecules act as a hydrogen-bond acceptor and two hydrogen-bond donors.

The hydrogen-bond interactions of these $[O_3S-C_6H_4-PO_3H]^{2-}$ dimers and water tetramers in (I) lead to the formation of a novel two-dimensional supramolecular layer aggregate (Fig. 4). The layer features two kinds of rings, discussed here according to graph-set analysis nomenclature

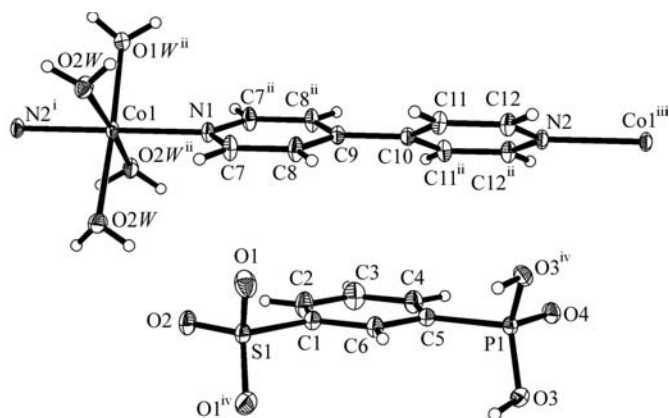


Figure 5

The structure of compound (II), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii. [Symmetry codes: (i) $x, -\frac{1}{2} + y, \frac{1}{2} - z$; (ii) $\frac{1}{2} - x, y, \frac{1}{2} - z$; (iii) $x, \frac{1}{2} + y, \frac{1}{2} - z$; (iv) $-x, y, z$.]

(Bernstein *et al.*, 1995). One ring, including one solvent water molecule, one $[\text{O}_3\text{S}-\text{C}_6\text{H}_4-\text{PO}_3\text{H}]^{2-}$ anion and one $-\text{PO}_3\text{H}^-$ group from another $[\text{O}_3\text{S}-\text{C}_6\text{H}_4-\text{PO}_3\text{H}]^{2-}$ anion, can be specified as having an $R_3^4(14)$ pattern, whereas the other ring, including five solvent water molecules, one $[\text{O}_3\text{S}-\text{C}_6\text{H}_4-\text{PO}_3\text{H}]^{2-}$ anion, and one $-\text{PO}_3\text{H}^-$ group and one sulfonate group from two other $[\text{O}_3\text{S}-\text{C}_6\text{H}_4-\text{PO}_3\text{H}]^{2-}$ anions, can be specified as having an $R_8^5(24)$ pattern. The whole packing scheme of (I) thus presents a layer structure following the alternating array pattern of $\dots ABAB\dots$, where *A* is an $[\text{Ag}(4,4'\text{-bipy})_2]$ layer and *B* is an $[(\text{O}_3\text{S}-\text{C}_6\text{H}_4-\text{PO}_3\text{H})\cdot 3\text{H}_2\text{O}]$ layer.

The structure of compound (II) features one-dimensional $\{[\text{Co}(4,4'\text{-bipy})(\text{H}_2\text{O})_4]^{2+}\}_n$ cationic chains with similar dimer $[\text{O}_3\text{S}-\text{C}_6\text{H}_4-\text{PO}_3\text{H}]^{2-}$ anions as in (I). The cationic unit possesses twofold crystallographic symmetry, while the $[\text{O}_3\text{S}-\text{C}_6\text{H}_4-\text{PO}_3\text{H}]^{2-}$ anion is constrained to a mirror plane. The Co^{II} ion is octahedrally coordinated by two N atoms from two symmetry-related 4,4'-bipy ligands and by two pairs of symmetry-related water molecules (Fig. 5 and Table 3). Although $\{[\text{Co}(4,4'\text{-bipy})(\text{H}_2\text{O})_4]^{2+}\}_n$ cationic chains are commonly observed (see, for example, Wang *et al.*, 2006), their supramolecular arrays are diverse and strongly affected by the different counter-ions being employed.

The dimer anions in (II) are similar to those in (I) through the common twofold symmetry relationship between each hydrogen-bonded $[\text{O}_3\text{S}-\text{C}_6\text{H}_4-\text{PO}_3\text{H}]^{2-}$ dimer centred on the bonded H atom (H3) (Table 4 and Fig. 3*b*). In addition, the benzene rings are all ordered and constrained to have mirror plane symmetry, so the dihedral angle between the two benzene rings in the dimer anion is 0° , whereas that in (I) is $24.5(1)^\circ$ for the ordered molecule. The dimer is stabilized by hydrogen bonding to two pairs of O2W water molecules, utilizing two pairs of sulfonate and two pairs of $-\text{PO}_3\text{H}^-$ O atoms as receptors. This dimer further utilizes its remaining one pair of sulfonate and one pair of $-\text{PO}_3\text{H}^-$ O atoms as receptors and is connected to four neighbouring dimers *via* the hydrogen-bonding associations of four pairs of O1W water

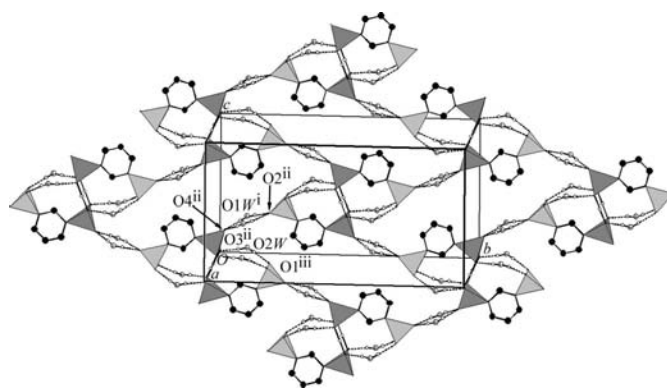


Figure 6

Hydrogen-bond interactions between the $[\text{O}_3\text{S}-\text{C}_6\text{H}_4-\text{PO}_3\text{H}]^{2-}$ anions and aqua molecules in compound (II). For display details, see the caption for Fig. 3. [Symmetry codes: (i) $\frac{1}{2} - x, y, \frac{1}{2} - z$; (ii) $\frac{1}{2} + x, -\frac{1}{2} + y, z$; (iii) $\frac{1}{2} - x, \frac{1}{2} - y, -z$.]

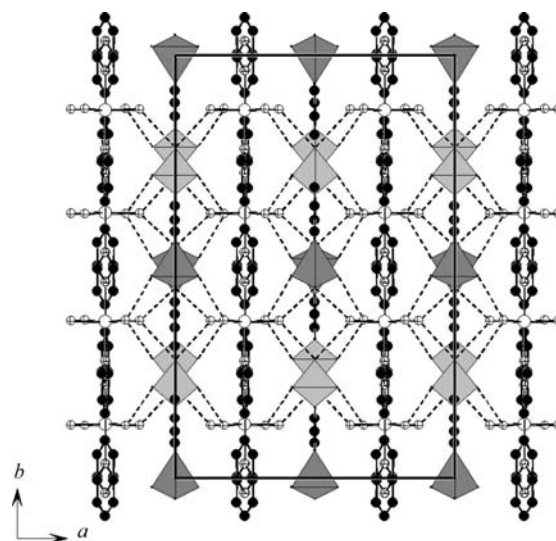


Figure 7

View of the structure of compound (II) down the *c* axis. H atoms have been omitted for clarity. Co atoms are drawn as white spheres; for other display details, see the caption for Fig. 3.

molecules, leading to the formation of a novel two-dimensional supramolecular layer aggregate (Table 4 and Fig. 6). This layer contains graph-set rings such as $R_2^4(8)$ and $R_4^4(12)$, *etc.*, which are completely different to the supramolecular layer structure in (I). It is noted that all aqua molecules here act as hydrogen-bond donors and each is hydrogen bonded to one $-\text{PO}_3\text{H}^-$ and one sulfonate O atom.

The interconnection of the Co^{II} ions of (II) by the bidentate bridging 4,4'-bipy ligands results in the formation of one-dimensional chains along the *b* axis, which are further assembled into an extensive three-dimensional supramolecular architecture *via* the hydrogen-bond interactions described above (Fig. 7 and Table 4).

In summary, two novel hydrogen-bonded layers directed by $[\text{O}_3\text{S}-\text{C}_6\text{H}_4-\text{PO}_3\text{H}]^{2-}$ anions have been observed in compounds (I) and (II). The $[\text{O}_3\text{S}-\text{C}_6\text{H}_4-\text{PO}_3\text{H}]^{2-}$ anions in both compounds form unique dimers *via* two short hydrogen

bonds between two pairs of $-\text{PO}_3\text{H}^-$ O atoms, and are further hydrogen bonded to solvent water or aqua molecules.

Experimental

For the preparation of (I), a mixture of AgNO_3 (56 mg, 0.33 mmol), 3-sulfophenylphosphonic acid (67 mg, 0.28 mmol) and 4,4'-bipy (52 mg, 0.33 mmol) in distilled water (10 ml) was placed in a Parr Teflon-lined autoclave (23 ml) and heated at 423 K for 4 d. Colourless column-shaped crystals were collected in a ca 72% yield based on Ag. Analysis calculated for $\text{C}_{26}\text{H}_{27}\text{Ag}_2\text{N}_4\text{O}_9\text{PS}$: C 38.16, H 3.33, N 6.85%; found: C 38.10, H 3.40, N 6.82%. IR data (KBr, ν , cm^{-1}): 3429 (s), 3039 (m), 1599 (s), 1528 (m), 1486 (m), 1406 (m), 1217 (vs), 1190 (vs), 1107 (s), 1071 (m), 1037 (s), 995 (m), 904 (m), 851 (m), 803 (s), 732 (m), 689 (m), 618 (m), 564 (m), 531 (m). For the preparation of (II), a mixture of CoCO_3 (36 mg, 0.30 mmol), 3-sulfophenylphosphonic acid (83 mg, 0.35 mmol) and 4,4'-bipy (47 mg, 0.30 mmol) in distilled water (10 ml) was placed in a Parr Teflon-lined autoclave (23 ml) and heated at 413 K for 4 d. Orange brick-shaped crystals were collected in a ca 55% yield based on Co. Analysis calculated for $\text{C}_{16}\text{H}_{21}\text{CoN}_2\text{O}_{10}\text{PS}$: C 36.72, H 4.04, N 5.35%; found: C 36.68, H 4.11, N 5.31%. IR data (KBr, ν , cm^{-1}): 3401 (s), 3071 (m), 1607 (s), 1534 (m), 1490 (m), 1414 (m), 1219 (m), 1171 (s), 1100 (vs), 1067 (s), 1030 (vs), 997 (s), 862 (m), 810 (m), 732 (m), 688 (m), 618 (m), 559 (m).

Compound (I)

Crystal data

$[\text{Ag}_2(\text{C}_{10}\text{H}_8\text{N}_2)_2](\text{C}_6\text{H}_5\text{O}_6\text{PS})\cdot 3\text{H}_2\text{O}$ $V = 5747(2) \text{ \AA}^3$
 $M_r = 818.29$ $Z = 8$
 Orthorhombic, *Pbcn* $\text{Mo K}\alpha$ radiation
 $a = 14.539(3) \text{ \AA}$ $\mu = 1.55 \text{ mm}^{-1}$
 $b = 17.472(4) \text{ \AA}$ $T = 293(2) \text{ K}$
 $c = 22.623(4) \text{ \AA}$ $0.50 \times 0.12 \times 0.12 \text{ mm}$

Data collection

Rigaku Mercury70 diffractometer 42765 measured reflections
 Absorption correction: multi-scan 6577 independent reflections
 (*CrystalClear*; Molecular 6186 reflections with $I > 2\sigma(I)$
 Structure Corporation & $R_{\text{int}} = 0.023$
 Rigaku, 2000)
 $T_{\text{min}} = 0.796$, $T_{\text{max}} = 0.834$

Table 1

Selected geometric parameters (\AA , $^\circ$) for (I).

Ag1—N2 ⁱ	2.139 (2)	Ag2—N3	2.137 (2)
Ag1—N1	2.145 (2)	Ag2—Ag2 ⁱⁱⁱ	3.2610 (8)
Ag2—N4 ⁱⁱ	2.131 (2)		
N2 ⁱ —Ag1—N1	172.38 (10)	N4 ⁱⁱ —Ag2—N3	170.82 (9)

Symmetry codes: (i) $x, -y + 1, z - \frac{1}{2}$; (ii) $x, -y, z + \frac{1}{2}$; (iii) $-x, -y, -z + 1$.

Table 2

Hydrogen-bond geometry (\AA , $^\circ$) for (I).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1W—H11 \cdots O3	0.87 (5)	2.06 (5)	2.913 (4)	167 (5)
O1W—H12 \cdots O6 ^{iv}	0.74 (5)	2.19 (5)	2.916 (3)	166 (6)
O2W—H22 \cdots O3W ^v	0.78 (5)	2.20 (5)	2.898 (4)	149 (5)
O2W—H21 \cdots O2	0.82 (5)	2.01 (5)	2.796 (3)	161 (5)
O3W—H32 \cdots O6	0.88 (5)	1.84 (5)	2.717 (4)	174 (5)
O3W—H31 \cdots O2W ^{vi}	0.71 (5)	2.09 (5)	2.795 (4)	171 (6)
O5—H5 \cdots O5 ^{iv}	1.273 (6)	1.273 (6)	2.528 (4)	166 (5)

Symmetry codes: (iv) $-x, y, -z + \frac{1}{2}$; (v) $-x + \frac{1}{2}, -y + \frac{1}{2}, z + \frac{1}{2}$; (vi) $x + \frac{1}{2}, -y + \frac{1}{2}, -z + 1$.

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.078$
 $S = 1.04$
 6577 reflections
 434 parameters
 3 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.55 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.44 \text{ e \AA}^{-3}$

Compound (II)

Crystal data

$[\text{Co}(\text{C}_{10}\text{H}_8\text{N}_2)_2(\text{H}_2\text{O})_4](\text{C}_6\text{H}_5\text{O}_6\text{PS})$ $V = 4106.8(4) \text{ \AA}^3$
 $M_r = 523.31$ $Z = 8$
 Orthorhombic, *Cmca* $\text{Mo K}\alpha$ radiation
 $a = 14.9569(8) \text{ \AA}$ $\mu = 1.07 \text{ mm}^{-1}$
 $b = 22.5625(11) \text{ \AA}$ $T = 293(2) \text{ K}$
 $c = 12.1697(7) \text{ \AA}$ $0.43 \times 0.20 \times 0.20 \text{ mm}$

Data collection

Rigaku Mercury70 diffractometer 15395 measured reflections
 Absorption correction: multi-scan 2446 independent reflections
CrystalClear (Molecular 2337 reflections with $I > 2\sigma(I)$
 Structure Corporation & $R_{\text{int}} = 0.030$
 Rigaku, 2000)
 $T_{\text{min}} = 0.771$, $T_{\text{max}} = 0.811$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.092$
 $S = 1.06$
 2446 reflections
 171 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.51 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.42 \text{ e \AA}^{-3}$

Table 3

Selected bond lengths (\AA) for (II).

Co1—N1	2.098 (2)	Co1—N2 ⁱ	2.120 (2)
Co1—O2W	2.0976 (17)	Co1—O1W	2.1384 (18)

Symmetry code: (i) $x, y - \frac{1}{2}, -z + \frac{1}{2}$.

Table 4

Hydrogen-bond geometry (\AA , $^\circ$) for (II).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1W—H11 \cdots O2	0.76 (3)	2.13 (3)	2.880 (2)	166 (3)
O1W—H12 \cdots O4 ⁱ	0.82 (3)	1.93 (3)	2.745 (2)	175 (3)
O2W—H21 \cdots O1 ⁱⁱ	0.79 (3)	1.95 (3)	2.730 (2)	170 (3)
O2W—H22 \cdots O3 ⁱⁱⁱ	0.78 (3)	1.95 (3)	2.731 (2)	174 (3)
O3—H3 \cdots O3 ^{iv}	1.2638 (16)	1.2638 (16)	2.528 (3)	180 (4)

Symmetry codes: (i) $x, y - \frac{1}{2}, -z + \frac{1}{2}$; (ii) $-x + \frac{1}{2}, -y + \frac{1}{2}, -z$; (iii) $x + \frac{1}{2}, y - \frac{1}{2}, z$; (iv) $x, -y + 1, -z$.

H atoms bonded to C atoms were positioned geometrically ($C-H = 0.93 \text{ \AA}$) and included in the refinement in the riding-model approximation, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. Water H atoms and H atoms of $-\text{PO}_3\text{H}^-$ groups were located in a difference map and refined with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$. Atoms C7, C8, C15 and C16 of the same benzene ring in (I) were found to be disordered and were modelled over two sets of positions (*A* and *B*) giving refined occupancies of 0.531 (6) and 0.469 (4), respectively. The anisotropic displacement parameters of atoms C15*B* and C16*B* were restrained due to their larger value.

For both compounds, data collection: *CrystalClear* (Molecular Structure Corporation & Rigaku, 2000); cell refinement: *CrystalClear*;

data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXTL*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: GA3106). Services for accessing these data are described at the back of the journal.

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