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#### Key indicators

Single-crystal X-ray study

$T = 298$  K

Mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å

$R$  factor = 0.025

$wR$  factor = 0.068

Data-to-parameter ratio = 15.4

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

# [Co(H<sub>2</sub>O)<sub>6</sub>][[Co(C<sub>4</sub>H<sub>4</sub>N<sub>2</sub>)(H<sub>2</sub>O)<sub>2</sub>][V<sub>2</sub>O<sub>2</sub>(pmida)<sub>2</sub>]]·2H<sub>2</sub>O [H<sub>4</sub>pmida is *N*-(phosphonomethyl)iminodiacetic acid]: the first two-dimensional hybrid framework containing [V<sub>2</sub>O<sub>2</sub>(pmida)<sub>2</sub>]<sup>4-</sup> building blocks

The crystal structure of the title compound, polymeric hexaaquacobalt(II) diaquadioxodi- $\mu_2$ -pyrazine-bis[ $\mu_3$ -*N*-(phosphonomethyl)iminodiacetato]cobalt(II)divanadate(IV) dihydrate, {[Co(H<sub>2</sub>O)<sub>6</sub>][CoV<sub>2</sub>(C<sub>3</sub>H<sub>4</sub>O<sub>8</sub>P)<sub>2</sub>(C<sub>4</sub>H<sub>4</sub>N<sub>2</sub>)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>]}·2H<sub>2</sub>O]<sub>*n*</sub>, is the first example of a two-dimensional hybrid framework containing centrosymmetric dimeric [V<sub>2</sub>O<sub>2</sub>(pmida)<sub>2</sub>]<sup>4-</sup> anionic units [H<sub>4</sub>pmida is *N*-(phosphonomethyl)iminodiacetic acid]. The structure contains two crystallographically unique cobalt(II) centres, both located at inversion centres and exhibiting Jahn–Teller-distorted octahedral coordination geometries. One Co<sup>2+</sup> cation establishes physical links between adjacent [V<sub>2</sub>O<sub>2</sub>(pmida)<sub>2</sub>]<sup>4-</sup> anionic units, forming one-dimensional anionic ribbons which run along the [010] direction, {[Co(H<sub>2</sub>O)<sub>2</sub>][V<sub>2</sub>O<sub>2</sub>(pmida)<sub>2</sub>]}<sub>*n*</sub><sup>2*n*-</sup>. Pyrazine ligands, with their centroids located at inversion centres, bridge the above-mentioned Co<sup>2+</sup> centres, forming {[Co(H<sub>2</sub>O)<sub>2</sub>][V<sub>2</sub>O<sub>2</sub>(pmida)<sub>2</sub>]}<sub>*n*</sub><sup>2*n*-</sup> anionic layers which alternate along the [001] direction with [Co(H<sub>2</sub>O)<sub>6</sub>]<sup>2+</sup> cations and the water molecules of crystallization. An extensive and highly directional hydrogen-bonded network interconnects the structural components.

#### Comment

The design of coordination-based materials in which the topology is extended from zero-dimensional (*i.e.* discrete complexes) into one-, two- or three-dimensional, is a topical and interesting field of research. Since the seminal paper by Hoskins & Robson (1990), where diamondoid-type hybrid frameworks were engineered based upon the simple structures of cadmium and zinc cyanides, the field has expanded rapidly. This was motivated by the unusual architectures and the potential applications of such frameworks (Batten & Robson, 1998; Janiak, 2003; Mori *et al.*, 2004; Moulton & Zaworotko, 2001; Rowsell & Yaghi, 2004). In order to control, at least partially, the occurrence of ‘supramolecular isomerism’ (Moulton & Zaworotko, 2001), these hybrid crystalline materials are now being constructed using a typical modular approach: rigid and highly robust coordination-based cores are used as secondary building units (SBUs) for the construction of multidimensional metal–organic frameworks (MOFs) (Yaghi *et al.*, 1998).

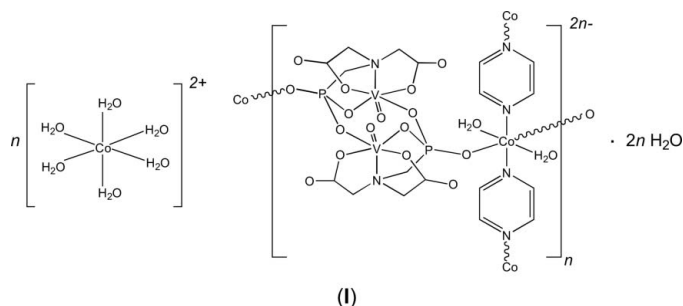
During the course of our work on crystalline organic–inorganic hybrid materials (Paz *et al.*, 2002, 2002*a,b,c*; Paz & Klinowski, 2003, 2004*a,b,c*; Paz, Shi *et al.*, 2004), we came across *N*-(phosphonomethyl)iminodiacetic acid (H<sub>4</sub>pmida), an organic molecule which, despite possessing several types of chelating functional groups, is relatively unexplored in the construction of MOFs (Fan *et al.*, 2004; Gutschke *et al.*, 1999; Mao *et al.*, 2002; Pei *et al.*, 2004; Song *et al.*, 2004), as confirmed

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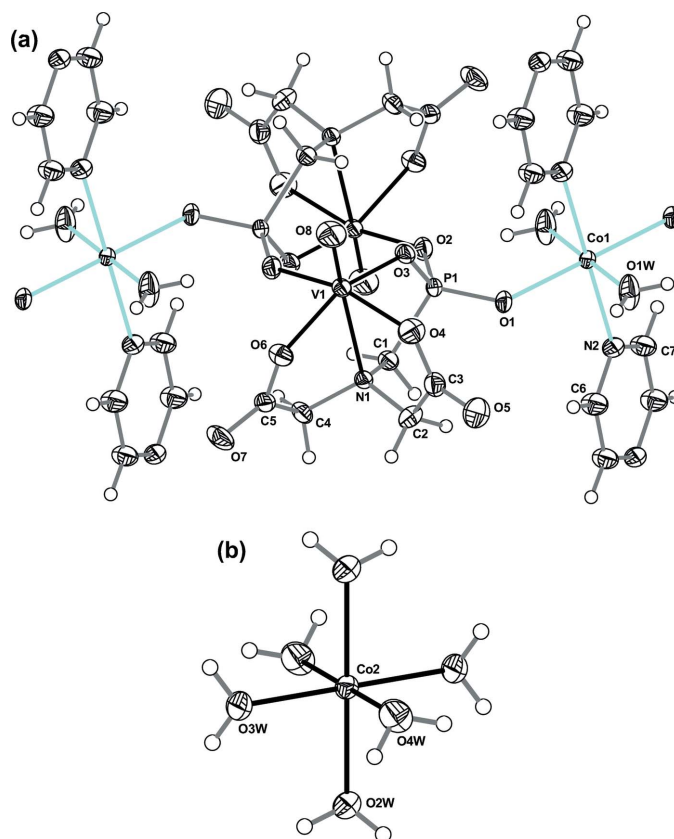
by a search in the Cambridge Structural Database (Version 5.25 of November 2003; Allen, 2002; Allen & Motherwell, 2002). This molecule forms with  $V^{4+}$  centres centrosymmetric dimeric  $[V_2O_2(pmida)_2]^{4-}$  anionic units, as originally reported by Crans *et al.* (1998); this anionic dimer is a rather convenient SBU owing, on the one hand, to its predictable self-assembly in aqueous media (particularly when hydrothermal synthetic approaches are employed) and, on the other, to the several terminal O atoms which can bound to a large variety of metal centres through various coordination modes. We recently reported the first three-dimensional frameworks containing  $[V_2O_2(pmida)_2]^{4-}$  anionic units, through their combination with transition metal centres (namely,  $Cd^{2+}$  and  $Co^{2+}$ ) and the bridging 4,4'-bipyridine organic ligand (Paz, Shi *et al.*, 2004). We report here the first two-dimensional framework containing these anionic units:  $[Co(H_2O)_6][Co(pyr)(H_2O)_2][V_2O_2(pmida)_2] \cdot 2(H_2O)$ , (I) (where pyr is pyrazine).



The title compound, (I), crystallizes in the triclinic space group  $P\bar{1}$ , with all its primary building blocks having inversion symmetry: the  $[V_2O_2(pmida)_2]^{4-}$  anionic unit has its centroid located at the inversion centre  $(\frac{1}{2}, 0, 0)$ ; both Co1 and Co2 are themselves located at inversion centres, with the former positioned in the middle of the *ab* plane, at  $(\frac{1}{2}, \frac{1}{2}, 0)$ , and the latter in the centre of the unit cell, at  $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$ ; the pyrazine bridging organic ligand, as for the  $[V_2O_2(pmida)_2]^{4-}$  anionic unit, is also centrosymmetric, with its centroid located at  $(0, \frac{1}{2}, 0)$ .

The geometry of the  $[V_2O_2(pmida)_2]^{4-}$  anionic unit is very similar to that previously reported for related compounds (Crans *et al.*, 1998; Paz, Shi *et al.*, 2004), with the  $pmida^{4-}$  anionic ligands encapsulating the  $V^{4+}$  centres inside three five-membered chelate rings (average bite angle of *ca*  $77^\circ$ , Table 1). The coordination polyhedron of these metal centres resembles a highly distorted octahedron,  $\{VNO_5\}$ , with the oxo group  $[V=O = 1.593(2) \text{ \AA}]$  markedly showing its *trans* influence in the rather long  $V-N$  distance  $[2.354(2) \text{ \AA}]$ . The phosphonate groups establish the physical bridges between adjacent  $V^{4+}$  centres (Fig. 1a), leading to a  $V1 \cdots V1^v$  distance of  $5.149(2) \text{ \AA}$  [symmetry code: (v)  $1 - x, -y, 1 - z$ ].

Both cobalt(II) centres adopt Jahn–Teller-distorted octahedral coordination geometries, as depicted in Fig. 1 and Table 1. For Co1, the equatorial plane is formed by two water molecules plus two O-donor atoms from phosphonate groups

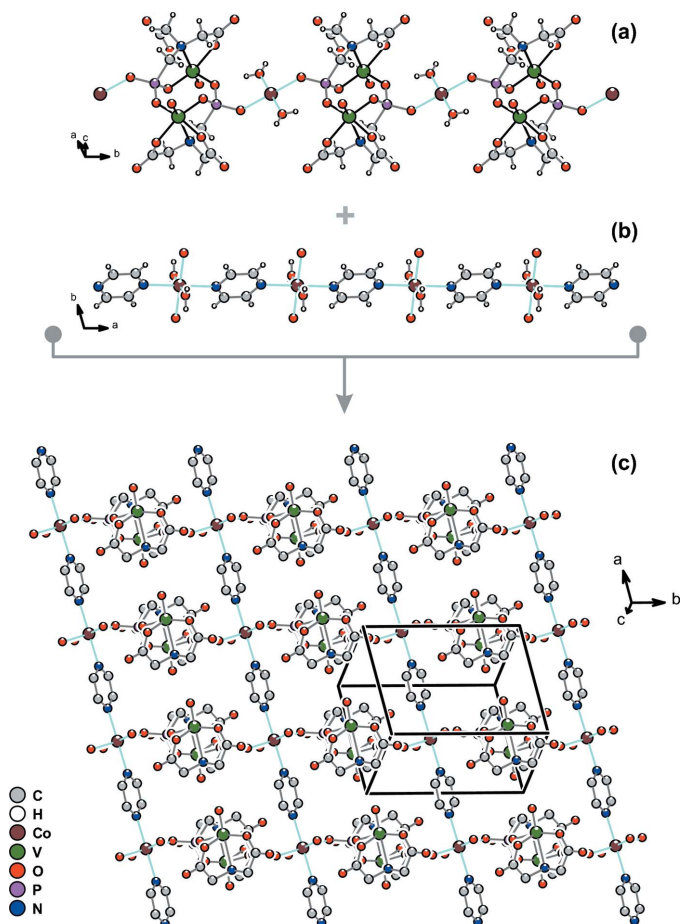


**Figure 1**

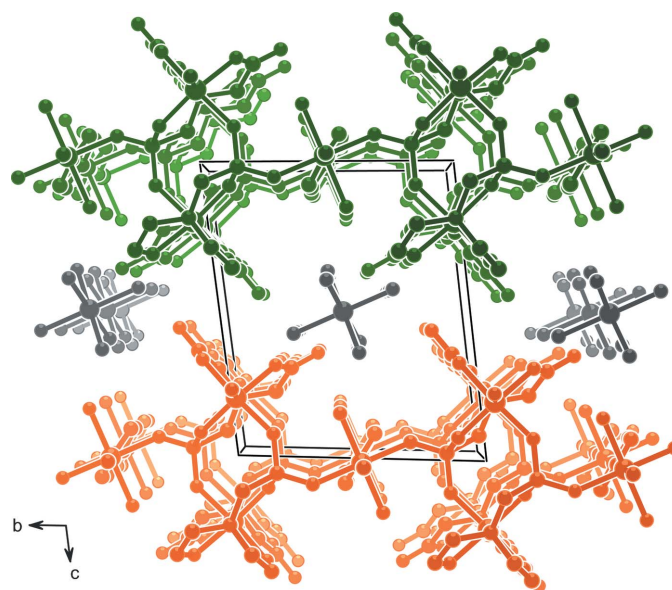
Schematic representation of (a) a portion of the anionic two-dimensional  $\{[Co(pyr)(H_2O)_2][V_2O_2(pmida)_2]\}_n^{2n-}$  layer and (b) the interlayer  $[Co(H_2O)_6]^{2+}$  cations, showing the labelling scheme for selected atoms in the asymmetric unit. Displacement ellipsoids are drawn at the 50% probability level.

(Fig. 1a), with an average  $Co-O$  distance of *ca*  $2.07 \text{ \AA}$ ; pyr ligands, axially coordinated, complete the coordination, with  $Co-N$  bond lengths of  $2.218(2) \text{ \AA}$  (Table 1). Co2 is coordinated only by water molecules, with O3W and O4W forming the equatorial plane (the average  $Co-O$  bond length is  $2.06 \text{ \AA}$ ) and O2W located  $2.144(2) \text{ \AA}$  from the metal centre. The *cis* octahedral angles are within the ranges  $88.04(6)$ – $91.96(6)$  and  $87.84(6)$ – $92.16(6)^\circ$  for Co1 and Co2, respectively (Table 1).

The centrosymmetric  $[V_2O_2(pmida)_2]^{4-}$  anionic unit is bound to two adjacent Co1 metal centres *via* the *trans*-uncoordinated  $P-O$  bonds, imposing a  $Co1 \cdots Co1^{viii}$  separation of  $10.140(2) \text{ \AA}$  [symmetry code: (viii)  $x, -1 + y, z$ ] (Fig. 1a). The repetition of this bridging motif leads to the formation of a one-dimensional anionic ribbon (running along the  $[010]$  direction),  $\{[Co(H_2O)_2][V_2O_2(pmida)_2]\}_n^{2n-}$ , as depicted in Fig. 2(a). Centrosymmetric bridging pyr ligands are axially coordinated to these Co1 centres (Fig. 2b), establishing physical connections between adjacent ribbons and further imposing a  $Co1 \cdots Co1^{vii}$  separation of  $7.220(1) \text{ \AA}$  [symmetry code: (vii)  $1 + x, y, z$ ]. Such an arrangement leads to the formation of a decorated two-dimensional  $\{[Co(pyr)(H_2O)_2][V_2O_2(pmida)_2]\}_n^{2n-}$  anionic layer with distances between Co1 atoms defined by the *a*- and *b*-axis lengths. The charge of this

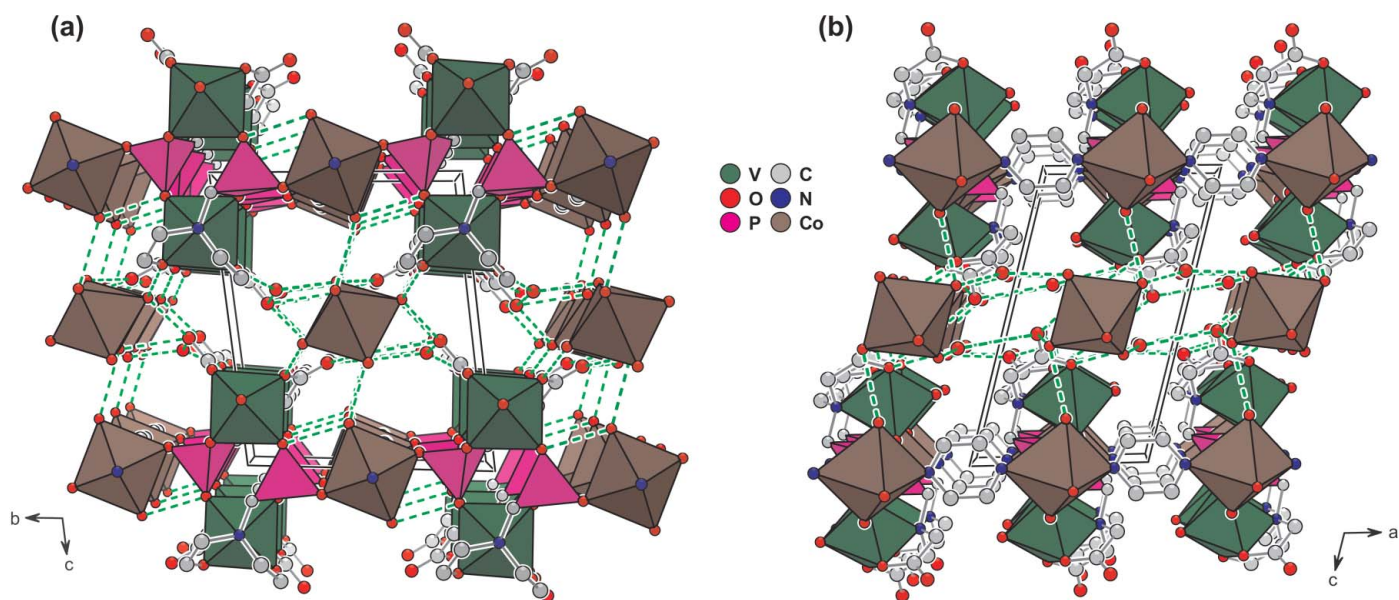


**Figure 2**  
Schematic representation of the construction of the anionic two-dimensional  $[[\text{Co}(\text{pyr})(\text{H}_2\text{O})_2][\text{V}_2\text{O}_2(\text{pmida})_2]]_n^{2n-}$  layers, which lie in the  $ab$  plane of the unit cell.



**Figure 3**  
Alternation along the  $[001]$  direction of anionic two-dimensional  $[[\text{Co}(\text{pyr})(\text{H}_2\text{O})_2][\text{V}_2\text{O}_2(\text{pmida})_2]]_n^{2n-}$  layers (in green and orange), which are intercalated by  $[\text{Co}(\text{H}_2\text{O})_6]^{2+}$  cations (in grey). H atoms have been omitted for clarity.

layer is compensated by the presence of hexaaquacobalt(II) cations,  $[\text{Co}(\text{H}_2\text{O})_6]^{2+}$  (Fig. 1*b*), which also act as space-filling groups, located in the available spaces resulting from the parallel packing, along the  $[001]$  direction, of individual anionic layers (Fig. 3). Each  $[[\text{Co}(\text{H}_2\text{O})_2][\text{V}_2\text{O}_2(\text{pmida})_2]]_n^{2n-}$  layer is further interconnected to the  $[\text{Co}(\text{H}_2\text{O})_6]^{2+}$  cations via a series of very strong and highly directional hydrogen-bond interactions, also involving the water molecules of crystallization (Fig. 4 and Table 2).



**Figure 4**  
Perspective view of the crystal packing of the title compound, viewed along the (a)  $[100]$  and (b)  $[010]$  directions of the unit cell. Hydrogen bonds are represented as green dashed bonds and H atoms have been omitted for clarity.



## Experimental

Chemicals were readily available from commercial sources and were used as received without further purification: *N*-(phosphonomethyl)iminodiacetic acid hydrate (H<sub>4</sub>pmida, 97% Fluka), pyrazine (98% Fluka), vanadium(IV) oxide sulfate pentahydrate (99% Sigma-Aldrich) and cobalt acetate tetrahydrate (99.0% Fluka). Syntheses were typically carried out in PTFE-lined stainless steel reaction vessels (*ca* 40 cm<sup>3</sup>), under autogenous pressure and static conditions in a preheated oven at 373 K. Reactions took place over a period of 4 d, after which the vessels were removed from the oven and left to cool to ambient temperature before opening. The title compound proved to be air- and light-stable, and insoluble in water and common organic solvents such as methanol, ethanol, acetone, dichloromethane, toluene, dimethyl sulfoxide and chloroform. The title compound was synthesized from a mixture containing VOSO<sub>4</sub>·5H<sub>2</sub>O (0.40 g), CoC<sub>4</sub>H<sub>6</sub>O<sub>4</sub>·4H<sub>2</sub>O (0.34 g), H<sub>4</sub>pmida (0.27 g), pyrazine (0.10 g) and NaOH (0.20 g) in distilled water (*ca* 10 g). The mixture was stirred at ambient temperature for 30 min, yielding a suspension with a molar composition of 2.1:1.2:1.0:1.1:4.2:467, which was transferred to the reaction vessel. After the reaction, a large quantity of light-blue single crystals of the title compound were isolated by vacuum filtration, washed with copious amounts of distilled water (*ca* 3 × 50 ml), and then air-dried at ambient temperature.

### Crystal data

C<sub>14</sub>H<sub>20</sub>CoN<sub>4</sub>O<sub>18</sub>P<sub>2</sub>V<sub>2</sub>·CoH<sub>12</sub>O<sub>6</sub>·  
2H<sub>2</sub>O  
M<sub>r</sub> = 958.15  
Triclinic, *P* $\bar{1}$   
*a* = 7.2200 (14) Å  
*b* = 10.140 (2) Å  
*c* = 12.080 (2) Å  
 $\alpha$  = 93.79 (3)°  
 $\beta$  = 103.21 (3)°  
 $\gamma$  = 104.21 (3)°  
*V* = 827.6 (3) Å<sup>3</sup>

*Z* = 1  
*D<sub>x</sub>* = 1.922 Mg m<sup>-3</sup>  
Mo *K*α radiation  
Cell parameters from 5000  
reflections  
 $\theta$  = 2–22.5°  
 $\mu$  = 1.73 mm<sup>-1</sup>  
*T* = 298 (2) K  
Prism, blue  
0.44 × 0.33 × 0.14 mm

### Data collection

Kuma KM-4 CCD diffractometer  
 $\omega$  scans  
Absorption correction: numerical  
(*SADABS*; Sheldrick, 1996)  
*T*<sub>min</sub> = 0.354, *T*<sub>max</sub> = 0.724  
14552 measured reflections  
3994 independent reflections

3510 reflections with *I* > 2σ(*I*)  
*R*<sub>int</sub> = 0.033  
 $\theta$ <sub>max</sub> = 28.8°  
*h* = -9 → 9  
*k* = -13 → 13  
*l* = -16 → 16

### Refinement

Refinement on *F*<sup>2</sup>  
*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.025  
*wR*(*F*<sup>2</sup>) = 0.068  
*S* = 1.08  
3994 reflections  
260 parameters  
H atoms treated by a mixture of  
independent and constrained  
refinement

$w = 1/[\sigma^2(F_o^2) + (0.0369P)^2 + 0.2906P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
( $\Delta\sigma$ )<sub>max</sub> = 0.001  
 $\Delta\rho$ <sub>max</sub> = 0.62 e Å<sup>-3</sup>  
 $\Delta\rho$ <sub>min</sub> = -0.46 e Å<sup>-3</sup>  
Extinction correction: *SHELXL97*  
Extinction coefficient: 0.0134 (13)

**Table 1**

Selected geometric parameters (Å, °).

Co1—O1 <sup>i</sup>	2.044 (2)	V1—O6	2.030 (2)
Co1—O1W	2.093 (2)	V1—O8	1.593 (2)
Co1—N2	2.218 (2)	V1—N1	2.354 (2)
Co2—O2W	2.144 (2)	O1—P1	1.508 (2)
Co2—O3W	2.063 (2)	O2—P1	1.534 (2)
Co2—O4W	2.061 (2)	O3—P1	1.529 (2)
V1—O2 <sup>ii</sup>	1.996 (2)	O1—Co1	2.044 (2)
V1—O3	1.957 (2)	P1—C1	1.829 (2)
V1—O4	2.041 (2)		
O1—Co1—O1W <sup>i</sup>	89.40 (6)	O3—V1—N1	80.43 (6)
O1—Co1—O1W	90.60 (6)	O4—V1—N1	76.45 (6)
O1 <sup>i</sup> —Co1—N2	91.96 (6)	O6—V1—O4	87.87 (6)
O1—Co1—N2	88.04 (6)	O6—V1—N1	74.10 (5)
O1W <sup>i</sup> —Co1—N2	88.11 (7)	O8—V1—O2 <sup>iii</sup>	100.68 (8)
O1W—Co1—N2	91.89 (7)	O8—V1—O3	105.45 (7)
O3W—Co2—O2W	88.56 (6)	O8—V1—O4	93.29 (8)
O3W—Co2—O2W <sup>iii</sup>	91.44 (6)	O8—V1—O6	100.33 (7)
O4W—Co2—O2W <sup>iii</sup>	87.84 (6)	O8—V1—N1	168.34 (7)
O4W—Co2—O2W	92.16 (6)	O1—P1—O3	112.77 (8)
O4W—Co2—O3W <sup>iii</sup>	91.43 (7)	O1—P1—O2	111.06 (7)
O4W—Co2—O3W	88.57 (7)	O3—P1—O2	110.17 (7)
O2 <sup>ii</sup> —V1—O4	165.00 (5)	O1—P1—C1	110.75 (8)
O2 <sup>ii</sup> —V1—O6	84.32 (6)	O3—P1—C1	104.04 (7)
O2 <sup>ii</sup> —V1—N1	89.08 (6)	O2—P1—C1	107.73 (8)
O3—V1—O2 <sup>ii</sup>	91.38 (6)	P1—O1—Co1	136.12 (7)
O3—V1—O4	90.11 (6)	P1—O3—V1	126.89 (7)
O3—V1—O6	154.21 (5)		

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

**Table 2**

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O1W—H1C...O2 <sup>i</sup>	0.84 (2)	1.98 (1)	2.7489 (19)	154 (2)
O1W—H1D...O2W	0.84 (2)	2.09 (1)	2.878 (2)	160 (3)
O2W—H2C...O5	0.84 (2)	2.00 (1)	2.798 (2)	159 (2)
O2W—H2D...O7 <sup>iv</sup>	0.84 (2)	1.93 (1)	2.736 (2)	166 (2)
O3W—H3A...O5	0.84 (2)	1.87 (1)	2.672 (2)	159 (2)
O3W—H3B...O6 <sup>v</sup>	0.84 (2)	1.95 (2)	2.7801 (18)	173 (2)
O4W—H4C...O5W <sup>iii</sup>	0.84 (2)	1.86 (1)	2.698 (2)	175 (3)
O4W—H4D...O7 <sup>vi</sup>	0.84 (2)	1.86 (1)	2.689 (2)	167 (2)
O5W—H5B...O4	0.84 (2)	2.14 (2)	2.893 (2)	151 (3)
O5W—H5A...O5 <sup>vii</sup>	0.84 (2)	2.54 (2)	3.311 (3)	157 (3)

Symmetry codes: (i)  $-x + 1, -y + 1, -z + 2$ ; (iii)  $-x + 1, -y + 1, -z + 1$ ; (iv)  $x, y + 1, z$ ; (v)  $-x + 1, -y, -z + 1$ ; (vi)  $-x, -y, -z + 1$ ; (vii)  $x + 1, y, z$ .

Non-H atoms were located from difference Fourier maps calculated in successive least-squares refinement cycles. The C-bound H atoms were placed in idealized positions and refined as riding with C—H = 0.93–0.97 Å and *U*<sub>iso</sub> = 1.2*U*<sub>eq</sub>(C). H atoms of water molecules were located in difference Fourier maps, and refined with the O—H and H...H distances restrained to 0.84 (1) and 1.37 (1) Å, respectively, to ensure chemically reasonable geometry, with *U*<sub>iso</sub> fixed at 1.5*U*<sub>eq</sub>(O).

Data collection: *CrysAlis CCD* (Kuma, 1999); cell refinement: *CrysAlis RED* (Kuma, 1999); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXTL* (Bruker, 2001); molecular graphics: *DIAMOND* (Brandenburg, 2001); software used to prepare material for publication: *SHELXTL*.

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