Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Redetermination of *trans*-diaquatetramethanolcobalt(**II**) bis(*rac*-1,1′-binaphthalene-2,2′-diylphosphate) methanol disolvate monohydrate: a two-dimensional supramolecular hydrogen-bonded network

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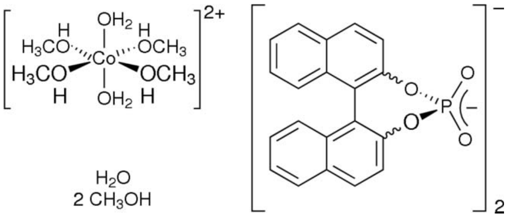
Received 9 May 2007; accepted 16 May 2007

Key indicators: single-crystal X-ray study; *T* = 203 K; mean σ(C–C) = 0.006 Å; *R* factor = 0.052; *wR* factor = 0.147; data-to-parameter ratio = 14.2.

In the title compound, *trans*-$[Co(CH_3OH)_4(H_2O)_2]$-$(C_{20}H_{12}PO_4)_2\cdot 2CH_3OH\cdot H_2O$, the crystal packing shows a separation of the hydrophobic naphthyl ring systems from the hydrophilic part of the structure, *viz*. the $(RO)_2PO_2^-$ phosphate anion, the cobalt complex cation and the solvent molecules. The binaphthyl tail-to-tail packing in the hydrophobic layer is governed by weak C—H···π interactions. The present study performed at 203 K confirms the previous room-temperature study [McCann, Murphy, Cardin & Convery (1991), *Polyhedron*, **10**, 2771–2777], but with improved precision. The centrosymmetric cobalt complex has very similar Co—O bond lengths and is isostructural with the *trans*-$[Cu(H_2O)_2(CH_3OH)_4]^{2+}$ cation (which features a tetragonally compressed instead of the typical Jahn–Teller distorted elongated copper octahedron) in the isotypic copper(II) compound. The high degree of similarity in the Co and Cu structures shows the dominating effect of the hydrogen-bonding network on the metal coordination polyhedra. All H atoms of the Co and Cu aqua and methanol ligands are engaged in typical strong hydrogen-bonding interactions.

Related literature

For isotypic compounds and closely related structures, see: Deeth & Hearnshaw (2006); Dorn *et al.* (2006); Janiak (2000); McCann *et al.* (1991); Nishio (2004); Wisser & Janiak (2007).



Experimental

Crystal data

$[Co(CH_4O)_4(H_2O)_2](C_{20}H_{12}PO_4)_2\cdot 2CH_4O\cdot H_2O$
M_r = 999.76
Monoclinic, $C2/c$
a = 41.795 (9) Å
b = 8.6674 (19) Å
c = 13.161 (3) Å
β = 105.548 (4)°
V = 4593.2 (18) Å^3
Z = 4
Mo *Kα* radiation
μ = 0.52 mm^{-1}
T = 203 (2) K
0.49 × 0.26 × 0.02 mm

Data collection

Bruker APEX II CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.786, T_{max} = 0.988
17662 measured reflections
4506 independent reflections
2576 reflections with $I > 2\sigma(I)$
R_{int} = 0.085

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.147
S = 0.99
4506 reflections
317 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.57 e Å^{-3}

Table 1
Selected bond lengths (Å).

Co—O3	2.038 (3)	P1—O6	1.481 (3)
Co—O1	2.089 (3)	P1—O4	1.607 (3)
Co—O2	2.104 (3)	P1—O5	1.613 (3)
P1—O7	1.479 (3)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*D*···O6^i	0.90 (3)	1.80 (4)	2.677 (4)	164 (4)
O2—H2*D*···O9	0.80 (5)	2.02 (5)	2.777 (7)	158 (5)
O3—H3*A*···O8	0.88 (5)	1.82 (5)	2.692 (4)	170 (5)
O3—H3*B*···O6^{ii}	0.85 (5)	1.83 (5)	2.679 (4)	172 (5)
O8—H8*B*···O7^{iii}	0.81 (5)	1.87 (5)	2.673 (4)	168 (5)
O9—H9*B*···O7^{iv}	0.99	1.80	2.781 (9)	172.1

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Crystal Impact, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2007).

doi:10.1107/S1600536807024166

Support through grant No. Ja466/14-1 from DFG (Deutsche Forschungsgemeinschaft) is acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2111).

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supplementary materials

Acta Cryst. (2007). E**63**, m1732-m1733 [**doi:10.1107/S1600536807024166**]

Redetermination of *trans*-diaquatetramethanolcobalt(II) bis(*rac*-1,1'-binaphthalene-2,2'-diylphosphate) methanol disolvate monohydrate: a two-dimensional supramolecular hydrogen-bonded network

B. Wisser and **C. Janiak**

Comment

In the structure of the title compound (I) one inversion-symmetrical *trans*-$[Co(H_2O)_2(CH_3OH)_4]^{2+}$ cation is combined with two binaphthyl phosphate counterions, and one water and two methanol solvate molecules (McCann *et al.* 1991; Dorn *et al.*, 2006). The packing of the title compound can be rationalized by a separation of the hydrophobic binaphthyl backbone from the hydrophilic $(RO)_2PO_2^{-}$ phosphate groups, the cobalt complex cation and the solvent molecules into an inverse bilayer structure, as seen before (Wisser & Janiak, 2007; Dorn *et al.*, 2006). The structure of (I) is isotypic to that of the copper(II) analogue where the expected normal Jahn-Teller distortion of an elongated octahedron is absent. Instead, a tetragonal compressed octahedron, indicative of a dynamic Jahn-Teller effect, is observed (Dorn *et al.*, 2006). The M—O (*M* = Co and Cu) bonds lengths and their variations in the analogous structures are highly similar with M–O(H_2O) = 2.038 (3) and 1.937 (4) Å, and M–O(CH_3OH) = 2.089 (3)/2.104 (3) and 2.112 (4)/2.167 (4) Å for *M* = Co and Cu. The close similarity between the Co and Cu structures and the metal coordination polyhedra indicates a structure directing effect of the hydrogen-bonding interactions. For the Cu structure the two elongated Jahn-Teller-distorted states along the two *trans*-CH_3OH–Cu–CH_3OH bonds are of identical low energy and both occupied. There is no differentiation from any intermolecular interactions between these two states. The average of two tetragonally elongated octahedra then looks like a compressed octahedron for Cu (Deeth & Hearnshaw, 2006).

Fig. 1 shows a projection of the unit cell crystal packing to illustrate the layer-like arrangement of the hydrophobic and hydrophilic regions. The latter are also highlighted by the hydrogen-bonding network as red dashes (see Table for bond distances and angles). The interaction between the binaphthyl phosphate and the octahedrally coordinated cobalt(II) cation is visualized in Fig. 2. The binaphthyl tail-to-tail packing in the hydrophobic layer is governed by C–H$\cdots\pi$ interactions (Dorn *et al.*, 2006; Janiak, 2000; Nishio, 2004).

Experimental

A solution of racemic 1,1'-binaphthalene-2,2'-diyl phosphoric acid (139.2 mg, 0.20 mmol) (Dorn *et al.*, 2006) in 12 ml of methanol was added to a solution of $CoCl_2\cdot6H_2O$ (47.6 mg, 0.2 mmol) in 4 ml of distilled water. The solvent was slowly allowed to evaporate. After two days pink plates had formed which were separated by filtration. Crystal yield 96 mg, 48%. Analysis calculated for $C_{46}H_{54}CoO_{17}P_2$ (999.76): C 55.26, H 5.44; found: C 55.30, H 4.98. IR (KBr, ν cm^{-1}): 3209, 1653, 1617, 1587, 1506, 1464, 1430, 1328, 1236, 1208, 1093, 1068, 1022, 991, 960, 944, 868, 852, 816, 747, 719, 657, 580, 565, 532, 479, 415.

Refinement

The previous room-temperature study (McCann *et al.* 1991; Refcode:KUPYID) converged with *R*(F) = 0.13 for 1577 unique reflections with I>2σ(I) that were collected up to 20°/θ. Cell parameters were a = 41.93 (3), b = 8.683 (2), c = 13.21 (1) Å, β = 105.41 (4)°. The low precision of the previous data was attributed to the smallness of the largest available crystals. No bond lengths or angles were given, neither in the original publication nor in the entry of the Cambridge Crystallographic Data base (message: No three-dimensional coordinates available). Our low-temperature structure redetermination gave improved cell parameters by a factor of 3–10. Data was collected to over $\theta = 26°$, so that 2576 unique reflections with I>2σ(I) were available and the *R* factors improved considerably. H atoms bonded to C were refined with riding models and U_{eq}(H) = 1.2 U_{eq}(C_aromatic) or 1.5 U_{eq}(C_methyl), respectively. H atoms bonded to O atoms (H_2O, CH_3OH) were found from difference Fourier maps and their positions were freely refined with U_{eq}(H) = 1.2 U_{eq}(O_CH_3OH) and 1.5 U_{eq}(O_H_2O), respectively.

Figures

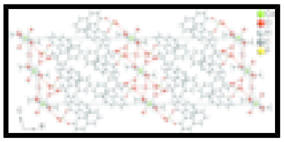

Fig. 1. : Projection of the crystal packing in compound (I) onto the (010) plane. Hydrogen bonds are indicated with red dashed lines. Displacement ellipsoids are drawn at the 50% probability level and H atoms are given as spheres of arbitrary radius.

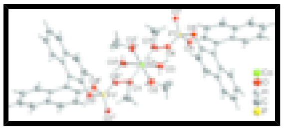

Fig. 2. : Interaction between the 1,1'-binaphthalene-2,2'-diyl phosphate anion and the *trans*-diaqua-tetramethanol-cobalt(II) cation in compound (I). The cobalt cation is located on an inversion center. Hydrogen bonds are indicated with red dashed lines. Displacement ellipsoids are drawn at the 50% probability level and H atoms are given as spheres of arbitrary radius. Symmetry code: i =– *x*, 1 – *y*, – *z*.

***trans*-Diaquatetramethanolcobalt(II) bis(rac-1,1'-binaphthalene-2,2'-diylphosphate) methanol disolvate monohydrate**

Crystal data

$[Co(CH_4O)_4(H_2O)_2](C_{20}H_{12}PO_4)_2·2CH_4O·H_2O$	F_{000} = 2092
M_r = 999.76	D_x = 1.446 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
a = 41.795 (9) Å	Cell parameters from 1024 reflections
b = 8.6674 (19) Å	θ = 2.4–20.0°
c = 13.161 (3) Å	μ = 0.52 mm^{-1}
β = 105.548 (4)°	*T* = 203 (2) K
V = 4593.2 (18) Å^3	Plate, pink
Z = 4	0.49 × 0.26 × 0.02 mm

Data collection

Bruker APEX II CCD area-detector diffractometer	4506 independent reflections

Radiation source: fine-focus sealed tube	2576 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\mathrm{int}} = 0.085$
$T = 203(2)$ K	$\theta_{\mathrm{max}} = 26.0^\circ$
\w scans	$\theta_{\mathrm{min}} = 2.0^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -51 \rightarrow 51$
$T_{\mathrm{min}} = 0.786$, $T_{\mathrm{max}} = 0.988$	$k = -10 \rightarrow 10$
17662 measured reflections	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.052$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.147$	$w = 1/[\sigma^2(F_o^2) + (0.0631P)^2 + 8.4598P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.99$	$(\Delta/\sigma)_{\mathrm{max}} < 0.001$
4506 reflections	$\Delta\rho_{\mathrm{max}} = 0.53$ e Å^{-3}
317 parameters	$\Delta\rho_{\mathrm{min}} = -0.57$ e Å^{-3}
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2^ against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2^, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2^. The threshold expression of F^2^ > 2sigma(F^2^) is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2^ are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Co	0.0000	0.5000	0.5000	0.0259 (2)
O1	−0.02130 (8)	0.3144 (3)	0.5593 (2)	0.0382 (7)
H1D	−0.0282 (11)	0.320 (5)	0.618 (3)	0.046*
O2	0.02480 (8)	0.3480 (4)	0.4222 (3)	0.0421 (8)
H2D	0.0147 (12)	0.313 (6)	0.366 (4)	0.050*
O3	0.03873 (8)	0.5095 (4)	0.6325 (2)	0.0400 (8)
H3A	0.0530 (12)	0.587 (6)	0.645 (4)	0.060*
H3B	0.0410 (12)	0.444 (6)	0.682 (4)	0.060*
C1	−0.01939 (13)	0.1553 (5)	0.5351 (4)	0.0532 (14)

H1A	−0.0185	0.1445	0.4625	0.080*
H1B	−0.0388	0.1021	0.5446	0.080*
H1C	0.0005	0.1109	0.5817	0.080*
C2	0.05817 (12)	0.3240 (7)	0.4397 (4)	0.0681 (17)
H2A	0.0662	0.3815	0.3883	0.102*
H2B	0.0623	0.2149	0.4330	0.102*
H2C	0.0696	0.3585	0.5102	0.102*
P1	0.07186 (3)	0.84064 (12)	0.27597 (8)	0.0261 (3)
O4	0.09320 (6)	0.7848 (3)	0.1977 (2)	0.0278 (6)
O5	0.10030 (6)	0.8622 (3)	0.3853 (2)	0.0279 (6)
O6	0.04962 (7)	0.7097 (3)	0.2814 (2)	0.0308 (7)
O7	0.05750 (6)	0.9960 (3)	0.2485 (2)	0.0333 (7)
C3	0.15083 (9)	0.8126 (4)	0.2862 (3)	0.0229 (8)
C4	0.12404 (9)	0.8498 (4)	0.2035 (3)	0.0264 (9)
C5	0.12665 (10)	0.9437 (5)	0.1196 (3)	0.0311 (9)
H5	0.1079	0.9645	0.0633	0.037*
C6	0.15689 (10)	1.0049 (5)	0.1208 (3)	0.0347 (10)
H6	0.1590	1.0657	0.0638	0.042*
C7	0.18505 (10)	0.9783 (4)	0.2061 (3)	0.0308 (9)
C8	0.21623 (11)	1.0472 (5)	0.2093 (4)	0.0381 (11)
H8A	0.2184	1.1081	0.1525	0.046*
C9	0.24276 (11)	1.0259 (5)	0.2937 (4)	0.0432 (12)
H9A	0.2633	1.0715	0.2949	0.052*
C10	0.23970 (11)	0.9358 (5)	0.3792 (4)	0.0401 (11)
H10A	0.2581	0.9223	0.4379	0.048*
C11	0.21021 (10)	0.8677 (5)	0.3778 (3)	0.0322 (10)
H11	0.2086	0.8086	0.4361	0.039*
C12	0.18207 (9)	0.8835 (4)	0.2913 (3)	0.0256 (9)
C13	0.14619 (9)	0.7076 (4)	0.3716 (3)	0.0233 (8)
C14	0.12153 (10)	0.7362 (4)	0.4189 (3)	0.0258 (9)
C15	0.11689 (10)	0.6481 (5)	0.5036 (3)	0.0325 (10)
H15	0.0999	0.6733	0.5352	0.039*
C16	0.13723 (11)	0.5265 (5)	0.5390 (3)	0.0362 (10)
H16	0.1351	0.4710	0.5982	0.043*
C17	0.16142 (10)	0.4822 (4)	0.4885 (3)	0.0312 (9)
C18	0.18039 (11)	0.3463 (5)	0.5177 (4)	0.0397 (11)
H18	0.1781	0.2889	0.5760	0.048*
C19	0.20178 (12)	0.2974 (5)	0.4635 (4)	0.0465 (13)
H19	0.2138	0.2057	0.4836	0.056*
C20	0.20605 (11)	0.3825 (5)	0.3777 (4)	0.0409 (11)
H20	0.2209	0.3479	0.3401	0.049*
C21	0.18852 (10)	0.5168 (4)	0.3486 (3)	0.0320 (9)
H21	0.1916	0.5734	0.2911	0.038*
C22	0.16599 (9)	0.5716 (4)	0.4030 (3)	0.0255 (9)
O8	0.08231 (7)	0.7421 (3)	0.6938 (3)	0.0385 (8)
H8B	0.0747 (11)	0.826 (5)	0.702 (4)	0.046*
C23	0.11277 (12)	0.7125 (6)	0.7674 (4)	0.0559 (14)
H23A	0.1307	0.7326	0.7354	0.084*
H23B	0.1136	0.6054	0.7894	0.084*

H23C	0.1151	0.7789	0.8282	0.084*
O9	0.0000 (2)	0.1564 (4)	0.2500 (8)	0.0335 (9)
H9B	0.0193	0.0954	0.2435	0.050*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co	0.0305 (4)	0.0208 (4)	0.0277 (4)	−0.0006 (3)	0.0104 (3)	−0.0001 (3)
O1	0.058 (2)	0.0227 (15)	0.0414 (18)	−0.0052 (14)	0.0267 (16)	0.0016 (14)
O2	0.0330 (18)	0.049 (2)	0.045 (2)	−0.0008 (15)	0.0105 (15)	−0.0194 (16)
O3	0.050 (2)	0.0264 (17)	0.0384 (18)	−0.0094 (15)	0.0023 (15)	0.0069 (14)
C1	0.077 (4)	0.030 (3)	0.062 (3)	−0.001 (2)	0.035 (3)	−0.002 (2)
C2	0.043 (3)	0.096 (5)	0.063 (4)	0.017 (3)	0.010 (3)	−0.030 (3)
P1	0.0264 (6)	0.0216 (5)	0.0317 (6)	−0.0004 (4)	0.0102 (5)	−0.0011 (4)
O4	0.0237 (14)	0.0331 (15)	0.0264 (15)	−0.0022 (12)	0.0065 (12)	−0.0028 (12)
O5	0.0322 (15)	0.0231 (14)	0.0281 (15)	0.0023 (12)	0.0075 (12)	−0.0038 (12)
O6	0.0321 (16)	0.0269 (15)	0.0356 (16)	−0.0092 (12)	0.0131 (13)	−0.0031 (12)
O7	0.0267 (15)	0.0246 (15)	0.0483 (18)	0.0058 (12)	0.0095 (13)	0.0076 (14)
C3	0.026 (2)	0.0181 (19)	0.025 (2)	0.0021 (16)	0.0088 (17)	−0.0019 (16)
C4	0.026 (2)	0.025 (2)	0.031 (2)	0.0027 (17)	0.0129 (18)	−0.0021 (17)
C5	0.031 (2)	0.030 (2)	0.033 (2)	0.0070 (18)	0.0102 (19)	0.0022 (18)
C6	0.041 (3)	0.029 (2)	0.039 (2)	0.007 (2)	0.020 (2)	0.007 (2)
C7	0.034 (2)	0.026 (2)	0.037 (2)	0.0030 (18)	0.0168 (19)	−0.0012 (18)
C8	0.038 (3)	0.035 (2)	0.048 (3)	−0.007 (2)	0.023 (2)	−0.003 (2)
C9	0.031 (2)	0.043 (3)	0.058 (3)	−0.011 (2)	0.017 (2)	−0.013 (2)
C10	0.030 (2)	0.037 (3)	0.052 (3)	−0.002 (2)	0.008 (2)	−0.009 (2)
C11	0.034 (2)	0.026 (2)	0.035 (2)	−0.0004 (18)	0.0076 (19)	−0.0025 (18)
C12	0.025 (2)	0.0181 (19)	0.034 (2)	0.0017 (16)	0.0088 (18)	−0.0029 (17)
C13	0.027 (2)	0.0177 (19)	0.023 (2)	−0.0013 (16)	0.0031 (17)	−0.0012 (15)
C14	0.030 (2)	0.021 (2)	0.026 (2)	0.0000 (17)	0.0053 (18)	0.0007 (16)
C15	0.036 (2)	0.038 (2)	0.026 (2)	−0.004 (2)	0.0128 (19)	0.0024 (19)
C16	0.045 (3)	0.035 (3)	0.028 (2)	−0.009 (2)	0.010 (2)	0.0058 (19)
C17	0.031 (2)	0.024 (2)	0.033 (2)	−0.0059 (18)	−0.0002 (18)	0.0045 (18)
C18	0.040 (3)	0.026 (2)	0.047 (3)	−0.004 (2)	0.000 (2)	0.013 (2)
C19	0.043 (3)	0.022 (2)	0.066 (3)	0.005 (2)	0.000 (3)	0.005 (2)
C20	0.037 (3)	0.025 (2)	0.058 (3)	0.0079 (19)	0.006 (2)	−0.005 (2)
C21	0.030 (2)	0.023 (2)	0.041 (2)	−0.0015 (18)	0.0060 (19)	0.0003 (19)
C22	0.022 (2)	0.0192 (19)	0.032 (2)	−0.0039 (16)	0.0027 (18)	−0.0001 (17)
O8	0.0364 (18)	0.0285 (17)	0.0480 (19)	0.0082 (13)	0.0069 (15)	0.0007 (15)
C23	0.044 (3)	0.071 (4)	0.051 (3)	0.017 (3)	0.008 (3)	0.012 (3)
O9	0.028 (2)	0.027 (2)	0.047 (3)	0.000	0.0125 (19)	0.000

Geometric parameters (Å, °)

Co—O3	2.038 (3)	C7—C8	1.424 (6)
Co—O3^{i}	2.038 (3)	C8—C9	1.355 (6)
Co—O1	2.089 (3)	C8—H8A	0.9400
Co—O1^{i}	2.089 (3)	C9—C10	1.404 (6)

Co—O2	2.104 (3)	C9—H9A	0.9400
Co—O2^{i}	2.104 (3)	C10—C11	1.362 (6)
O1—C1	1.422 (5)	C10—H10A	0.9400
O1—H1D	0.90 (3)	C11—C12	1.407 (5)
O2—C2	1.367 (5)	C11—H11	0.9400
O2—H2D	0.80 (5)	C13—C14	1.362 (5)
O3—H3A	0.88 (5)	C13—C22	1.435 (5)
O3—H3B	0.85 (5)	C14—C15	1.407 (5)
C1—H1A	0.9700	C15—C16	1.356 (6)
C1—H1B	0.9700	C15—H15	0.9400
C1—H1C	0.9700	C16—C17	1.404 (6)
C2—H2A	0.9700	C16—H16	0.9400
C2—H2B	0.9700	C17—C18	1.414 (6)
C2—H2C	0.9700	C17—C22	1.420 (5)
P1—O7	1.479 (3)	C18—C19	1.353 (7)
P1—O6	1.481 (3)	C18—H18	0.9400
P1—O4	1.607 (3)	C19—C20	1.400 (6)
P1—O5	1.613 (3)	C19—H19	0.9400
O4—C4	1.389 (4)	C20—C21	1.374 (5)
O5—C14	1.402 (4)	C20—H20	0.9400
C3—C4	1.375 (5)	C21—C22	1.410 (5)
C3—C12	1.429 (5)	C21—H21	0.9400
C3—C13	1.498 (5)	O8—C23	1.401 (5)
C4—C5	1.400 (5)	O8—H8B	0.81 (5)
C5—C6	1.367 (6)	C23—H23A	0.9700
C5—H5	0.9400	C23—H23B	0.9700
C6—C7	1.411 (6)	C23—H23C	0.9700
C6—H6	0.9400	O9—H9B	0.9881
C7—C12	1.422 (5)		

O3—Co—O3^{i}	180.00 (16)	C6—C7—C12	119.4 (4)
O3—Co—O1	91.81 (13)	C6—C7—C8	121.1 (4)
O3^{i}—Co—O1	88.19 (13)	C12—C7—C8	119.5 (4)
O3—Co—O1^{i}	88.19 (13)	C9—C8—C7	120.5 (4)
O3^{i}—Co—O1^{i}	91.81 (13)	C9—C8—H8A	119.7
O1—Co—O1^{i}	180.00 (17)	C7—C8—H8A	119.7
O3—Co—O2	93.42 (13)	C8—C9—C10	120.2 (4)
O3^{i}—Co—O2	86.58 (13)	C8—C9—H9A	119.9
O1—Co—O2	90.83 (12)	C10—C9—H9A	119.9
O1^{i}—Co—O2	89.17 (12)	C11—C10—C9	120.4 (4)
O3—Co—O2^{i}	86.58 (13)	C11—C10—H10A	119.8
O3^{i}—Co—O2^{i}	93.42 (13)	C9—C10—H10A	119.8
O1—Co—O2^{i}	89.17 (12)	C10—C11—C12	121.8 (4)
O1^{i}—Co—O2^{i}	90.83 (12)	C10—C11—H11	119.1
O2—Co—O2^{i}	180.00 (16)	C12—C11—H11	119.1
C1—O1—Co	127.4 (3)	C11—C12—C7	117.6 (4)
C1—O1—H1D	107 (3)	C11—C12—C3	123.5 (4)

Co—O1—H1D	123 (3)	C7—C12—C3	118.9 (4)
C2—O2—Co	128.8 (3)	C14—C13—C22	117.8 (3)
C2—O2—H2D	111 (4)	C14—C13—C3	119.8 (3)
Co—O2—H2D	118 (4)	C22—C13—C3	122.3 (3)
Co—O3—H3A	122 (3)	C13—C14—O5	119.4 (3)
Co—O3—H3B	122 (3)	C13—C14—C15	123.0 (4)
H3A—O3—H3B	116 (5)	O5—C14—C15	117.5 (3)
O1—C1—H1A	109.5	C16—C15—C14	119.2 (4)
O1—C1—H1B	109.5	C16—C15—H15	120.4
H1A—C1—H1B	109.5	C14—C15—H15	120.4
O1—C1—H1C	109.5	C15—C16—C17	121.0 (4)
H1A—C1—H1C	109.5	C15—C16—H16	119.5
H1B—C1—H1C	109.5	C17—C16—H16	119.5
O2—C2—H2A	109.5	C16—C17—C18	121.4 (4)
O2—C2—H2B	109.5	C16—C17—C22	119.5 (4)
H2A—C2—H2B	109.5	C18—C17—C22	119.0 (4)
O2—C2—H2C	109.5	C19—C18—C17	121.3 (4)
H2A—C2—H2C	109.5	C19—C18—H18	119.4
H2B—C2—H2C	109.5	C17—C18—H18	119.4
O7—P1—O6	119.39 (16)	C18—C19—C20	120.3 (4)
O7—P1—O4	112.08 (16)	C18—C19—H19	119.8
O6—P1—O4	105.30 (15)	C20—C19—H19	119.8
O7—P1—O5	105.46 (15)	C21—C20—C19	119.8 (4)
O6—P1—O5	111.56 (15)	C21—C20—H20	120.1
O4—P1—O5	101.75 (14)	C19—C20—H20	120.1
C4—O4—P1	120.6 (2)	C20—C21—C22	121.5 (4)
C14—O5—P1	116.4 (2)	C20—C21—H21	119.2
C4—C3—C12	118.5 (3)	C22—C21—H21	119.2
C4—C3—C13	119.6 (3)	C21—C22—C17	117.9 (4)
C12—C3—C13	121.8 (3)	C21—C22—C13	122.8 (4)
C3—C4—O4	119.7 (3)	C17—C22—C13	119.1 (4)
C3—C4—C5	122.8 (4)	C23—O8—H8B	113 (3)
O4—C4—C5	117.4 (4)	O8—C23—H23A	109.5
C6—C5—C4	118.9 (4)	O8—C23—H23B	109.5
C6—C5—H5	120.5	H23A—C23—H23B	109.5
C4—C5—H5	120.5	O8—C23—H23C	109.5
C5—C6—C7	121.1 (4)	H23A—C23—H23C	109.5
C5—C6—H6	119.4	H23B—C23—H23C	109.5
C7—C6—H6	119.4		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1D···O6^{i}	0.90 (3)	1.80 (4)	2.677 (4)	164 (4)
O2—H2D···O9	0.80 (5)	2.02 (5)	2.777 (7)	158 (5)
O3—H3A···O8	0.88 (5)	1.82 (5)	2.692 (4)	170 (5)
O3—H3B···O6^{ii}	0.85 (5)	1.83 (5)	2.679 (4)	172 (5)

O8—H8B···O7^{iii}	0.81 (5)	1.87 (5)	2.673 (4)	168 (5)
O9—H9B···O7^{iv}	0.99	1.80	2.781 (9)	172.1

Symmetry codes: (i) −*x*, −*y*+1, −*z*+1; (ii) *x*, −*y*+1, *z*+1/2; (iii) *x*, −*y*+2, *z*+1/2; (iv) *x*, *y*−1, *z*.

Fig. 1

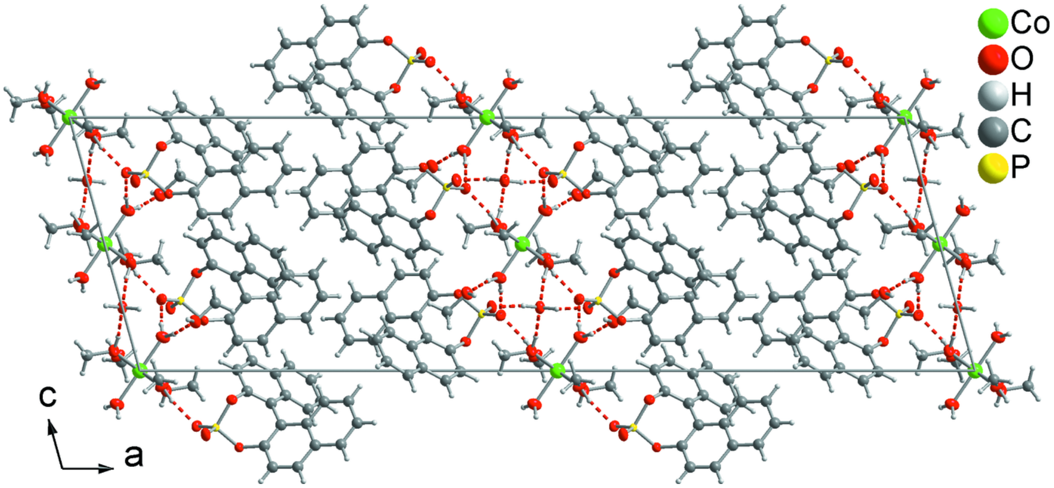

Fig. 2