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Tetraaquadiazidocobalt(II) 4,4'-dicarboxylato-1,1'-ethylenedi- pyridinium dihydrate

Kun Wang, Yan-Qin Wang, Jian-Yong Zhang and En-Qing Gao*

Shanghai Key Laboratory of Green Chemistry and Chemical Processes, Department of Chemistry, East China Normal University, Shanghai 200062, People's Republic of China

Correspondence e-mail: eqgao@chem.ecnu.edu.cn

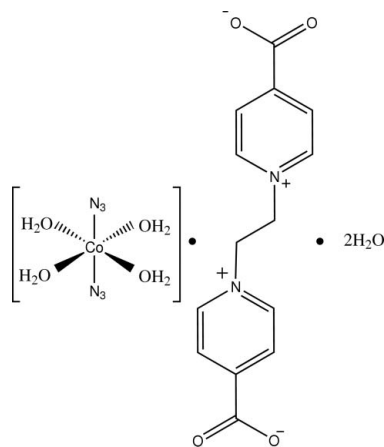
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; disorder in solvent or counterion; R factor = 0.027; wR factor = 0.089; data-to-parameter ratio = 11.1.

In the title compound, $[\text{Co}(\text{N}_3)_2(\text{H}_2\text{O})_4] \cdot \text{C}_{14}\text{H}_{12}\text{N}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$, the metal complex molecule is centrosymmetric, the Co(II) ion being six-coordinated by two azide N atoms and four aqua O atoms with a *trans*-octahedral geometry. The zwitterionic organic molecule is also centrosymmetric. In the crystal, the components are associated into a two-dimensional network through $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds. Further $\text{O}-\text{H} \cdots \text{O}$ and $\text{O}-\text{H} \cdots \text{N}$ interactions give a three-dimensional structure. The free water molecule is disordered over two positions in a 0.787 (5):0.213 (5) ratio.

Related literature

For background information on hydrogen bonds in crystal engineering, see: Baures *et al.* (2006); Braga & Grepioni (2000); Maly *et al.* (2006). For the ligand synthesis, see: Loeb *et al.* (2006). For hydrogen-bond motifs, see: Bernstein *et al.* (1995); Etter (1990).



Experimental

Crystal data

$[\text{Co}(\text{N}_3)_2(\text{H}_2\text{O})_4] \cdot \text{C}_{14}\text{H}_{12}\text{N}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$
 $M_r = 523.34$
 Triclinic, $P\bar{1}$
 $a = 7.1951$ (5) Å
 $b = 9.0354$ (7) Å
 $c = 9.0915$ (5) Å
 $\alpha = 71.402$ (3)°
 $\beta = 85.568$ (2)°
 $\gamma = 69.752$ (2)°
 $V = 525.20$ (6) Å³
 $Z = 1$
 Mo $K\alpha$ radiation
 $\mu = 0.89$ mm⁻¹
 $T = 296$ K
 $0.08 \times 0.08 \times 0.02$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2007)
 $T_{\min} = 0.932$, $T_{\max} = 0.982$
 6498 measured reflections
 2029 independent reflections
 2016 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.027$
 $wR(F^2) = 0.089$
 $S = 1.18$
 2029 reflections
 183 parameters
 13 restraints
 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.34$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.35$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{O}3-\text{H}31 \cdots \text{O}5^{\text{i}}$	0.826 (16)	1.908 (18)	2.727 (2)	170 (2)
$\text{O}3-\text{H}31 \cdots \text{O}5^{\text{ii}}$	0.826 (16)	1.96 (2)	2.664 (8)	143 (3)
$\text{O}4-\text{H}41 \cdots \text{O}2^{\text{ii}}$	0.812 (16)	2.072 (18)	2.870 (2)	167 (3)
$\text{O}5-\text{H}51 \cdots \text{O}2^{\text{iii}}$	0.802 (17)	2.110 (18)	2.877 (3)	160 (4)
$\text{O}3-\text{H}32 \cdots \text{O}1$	0.847 (16)	1.847 (17)	2.694 (2)	178 (3)
$\text{O}4-\text{H}42 \cdots \text{O}2$	0.846 (16)	1.847 (17)	2.687 (2)	172 (2)
$\text{O}5-\text{H}5 \cdots \text{N}4$	0.824 (17)	2.19 (2)	2.864 (3)	138.6 (19)

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

Data collection: APEX2 (Bruker, 2007); cell refinement: SAINT (Bruker, 2007); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2309).

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supplementary materials

Acta Cryst. (2009). E65, m1682 [doi:10.1107/S1600536809049848]

Tetraaquadiazidocobalt(II) 4,4'-dicarboxylato-1,1'-ethylenedipyridinium dihydrate

K. Wang, Y.-Q. Wang, J.-Y. Zhang and E.-Q. Gao

Comment

The D—H···A hydrogen bonds, ranging from the strong ones involving O—H and N—H to the weak ones involving C—H, have been widely used as a putative tool for engineering organic and metal-organic solids (Braga & Grepioni, 2000; Baures *et al.*, 2006; Maly *et al.*, 2006). In this paper, we report the hydrogen-bonded structure of the title compound, (I), which contains a neutral metal complex molecule, [Co(N₃)₂(H₂O)₄], and a zwitterionic dicarboxylate, 1,2-bis(4-carboxylatopyridinium)ethane (Loeb *et al.*, 2006).

The molecular structure is shown in Fig. 1. The metal complex molecule is centrosymmetric, with the Co(II) ion being six-coordinated by two azides and four aquas with a *trans*-octahedral geometry. The axial Co—N distances are slightly shorter than the equatorial Co—O ones. The zwitterionic molecule is also centrosymmetric. As shown in Fig. 2, the inorganic complex molecules and the organic molecules are associated into a two-dimensional sheet along the [101] direction through O—H···O hydrogen bonds involving the coordinated aqua ligands (O3 and O4) and the carboxylate oxygen atoms (O1 and O2). Two O4 aqua ligands from different complex molecules and two O2 atoms from different organic molecules, form a hydrogen-bonded ring which can be denoted by the graph set $R_4^2(8)$ (Bernstein *et al.*, 1995; Etter, 1990), and the carboxylate group forms a $R_2^2(8)$ hydrogen-bonded ring with two aqua ligands from the same complex molecule. The three-dimensional structure is formed *via* the hydrogen bonds between the disordered free water molecules (O5 and O5') and the terminal azide nitrogen (N4), the carboxylate oxygen (O2) or the coordinated water molecule (O3) (Fig. 3).

Experimental

The crystals were synthesized using the hydrothermal method in a 23 ml Teflon-lined Parr bomb. CoCl₂·6H₂O (0.0238 g, 0.1 mmol), 1,2-bis(4-carboxylatopyridinium)ethane (0.0434 g, 0.1 mmol), NaN₃ (0.052 g, 0.8 mmol) and distilled water (3 ml) were placed into the bomb and sealed. The bomb was then heated in a 70°C oven for 3 d and allowed to cool to room temperature. The clear colorless solution was decanted to give sheet orange crystals. Yield: 71.7%. Elemental analysis: calculated for C₁₄H₂₄CoN₈O₁₀: C 32.13, H 4.62, N 21.41%; found: C 32.28, H 4.79, N 21.73%. IR (KBr, ν/cm⁻¹): 2086, 1607, 1561, 1457, 1372, 1193, 1138, 1110, 1043, 782, 686.

Refinement

All hydrogen atoms attached to carbon atoms were placed at calculated positions and refined with the riding model using AFIX 43 and AFIX 23 instructions for aromatic C—H and secondary CH₂. The water hydrogen atoms were initially located from difference Fourier maps and refined isotropically with restraints on O—H distance (0.85 Å) and H—O—H angle, and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$. The free water molecule is disordered over two positions with the occupancies being refined to be 0.79 (O5) and 0.21 (O5').

Figures

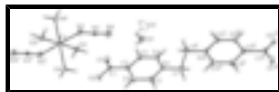


Fig. 1. Molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

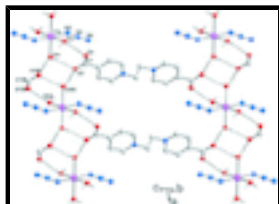


Fig. 2. Part of the crystal structure of (I), showing the formation of the two-dimensional network by O—H \cdots O hydrogen bonds which are shown in dashed lines. Hydrogen atoms not involved in the hydrogen bonds have been omitted for clarity.

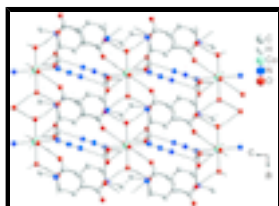


Fig. 3. The three-dimensional structure of (I) formed via O—H \cdots O and O—H \cdots N hydrogen-bonds which are shown as dashed lines.

Tetraaquadiazidocobalt(II) 4,4'-dicarboxylato-1,1'-ethylenedipyridinium dihydrate

Crystal data

[Co(N₃)₂(H₂O)₄] \cdot C₁₄H₁₂N₂O₄ \cdot 2H₂O

$M_r = 523.34$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 7.1951$ (5) Å

$b = 9.0354$ (7) Å

$c = 9.0915$ (5) Å

$\alpha = 71.402$ (3)°

$\beta = 85.568$ (2)°

$\gamma = 69.752$ (2)°

$V = 525.20$ (6) Å³

$Z = 1$

$F(000) = 271$

$D_x = 1.655$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 7436 reflections

$\theta = 2.5$ – 27.6 °

$\mu = 0.89$ mm⁻¹

$T = 296$ K

Sheet, orange

$0.08 \times 0.08 \times 0.02$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube graphite

phi and ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2007)

$T_{\min} = 0.932$, $T_{\max} = 0.982$

6498 measured reflections

2029 independent reflections

2016 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.020$

$\theta_{\text{max}} = 26.0$ °, $\theta_{\text{min}} = 2.4$ °

$h = -7 \rightarrow 8$

$k = -11 \rightarrow 11$

$l = -11 \rightarrow 11$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.027$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.089$	$w = 1/[\sigma^2(F_o^2) + (0.0517P)^2 + 0.2305P]$
$S = 1.18$	where $P = (F_o^2 + 2F_c^2)/3$
2029 reflections	$(\Delta/\sigma)_{\max} < 0.001$
183 parameters	$\Delta\rho_{\max} = 0.34 \text{ e } \text{\AA}^{-3}$
13 restraints	$\Delta\rho_{\min} = -0.35 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: <i>SHELXL97</i> (Sheldrick, 2008), $F_c^* = kF_c[1+0.001xF_c^2\lambda^3/\sin(2\theta)]^{-1/4}$
	Extinction coefficient: 0.072 (7)

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Co1	0.5000	0.5000	0.0000	0.02233 (16)	
N1	-0.0182 (2)	0.06625 (19)	0.78289 (17)	0.0240 (3)	
N2	0.5369 (3)	0.4349 (3)	0.2408 (2)	0.0458 (5)	
N3	0.4823 (2)	0.3822 (2)	0.36258 (18)	0.0283 (4)	
O3	0.4820 (2)	0.26543 (17)	0.02090 (16)	0.0300 (3)	
H31	0.425 (4)	0.272 (3)	-0.058 (2)	0.045*	
H32	0.420 (4)	0.229 (3)	0.099 (2)	0.045*	
O4	0.18626 (19)	0.59340 (17)	0.00759 (16)	0.0298 (3)	
H42	0.146 (4)	0.535 (3)	0.087 (2)	0.045*	
H41	0.135 (4)	0.600 (3)	-0.072 (2)	0.045*	
C1	0.1394 (3)	0.2551 (3)	0.3161 (2)	0.0316 (4)	
C2	0.0846 (3)	0.1895 (2)	0.4838 (2)	0.0267 (4)	
C3	0.1985 (3)	0.0345 (2)	0.5774 (2)	0.0284 (4)	
H3A	0.3109	-0.0288	0.5392	0.034*	
C4	0.1448 (3)	-0.0255 (2)	0.7271 (2)	0.0279 (4)	

supplementary materials

H4A	0.2211	-0.1298	0.7903	0.033*	
C5	-0.1306 (3)	0.2167 (2)	0.6939 (2)	0.0322 (4)	
H5A	-0.2426	0.2779	0.7342	0.039*	
C6	-0.0818 (3)	0.2812 (3)	0.5435 (2)	0.0335 (4)	
H6A	-0.1602	0.3859	0.4823	0.040*	
C7	-0.0719 (3)	-0.0022 (2)	0.9445 (2)	0.0283 (4)	
H7A	-0.0663	-0.1156	0.9636	0.034*	
H7B	-0.2061	0.0631	0.9607	0.034*	
N4	0.4358 (4)	0.3278 (3)	0.4876 (2)	0.0546 (6)	
O1	0.2747 (3)	0.1567 (2)	0.26605 (18)	0.0470 (4)	
O2	0.0391 (2)	0.4020 (2)	0.24191 (17)	0.0447 (4)	
O5	0.2585 (3)	0.2801 (3)	0.7844 (2)	0.0415 (7)	0.787 (5)
H5	0.344 (3)	0.244 (3)	0.727 (3)	0.062*	
H51	0.193 (5)	0.377 (3)	0.760 (4)	0.062*	0.787 (5)
O5'	0.4261 (18)	0.1544 (12)	0.7953 (9)	0.059 (3)	0.213 (5)
H52	0.506 (13)	0.097 (6)	0.744 (6)	0.089*	0.213 (5)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.0217 (2)	0.0262 (2)	0.0185 (2)	-0.00944 (14)	0.00190 (12)	-0.00503 (13)
N1	0.0264 (7)	0.0293 (8)	0.0161 (7)	-0.0115 (6)	0.0029 (5)	-0.0050 (6)
N2	0.0542 (12)	0.0691 (13)	0.0221 (9)	-0.0363 (11)	0.0023 (8)	-0.0080 (8)
N3	0.0273 (8)	0.0341 (8)	0.0252 (9)	-0.0110 (7)	-0.0001 (6)	-0.0105 (7)
O3	0.0309 (7)	0.0337 (7)	0.0279 (7)	-0.0147 (6)	0.0038 (5)	-0.0096 (6)
O4	0.0256 (6)	0.0341 (7)	0.0267 (7)	-0.0118 (5)	0.0019 (5)	-0.0041 (6)
C1	0.0371 (10)	0.0469 (11)	0.0172 (8)	-0.0266 (9)	-0.0001 (7)	-0.0048 (8)
C2	0.0309 (9)	0.0378 (10)	0.0169 (8)	-0.0208 (8)	-0.0002 (7)	-0.0058 (7)
C3	0.0308 (9)	0.0330 (9)	0.0230 (9)	-0.0120 (8)	0.0068 (7)	-0.0108 (7)
C4	0.0299 (9)	0.0262 (8)	0.0231 (8)	-0.0073 (7)	0.0028 (7)	-0.0048 (7)
C5	0.0291 (9)	0.0329 (10)	0.0260 (9)	-0.0043 (8)	0.0027 (7)	-0.0052 (8)
C6	0.0327 (10)	0.0333 (10)	0.0248 (9)	-0.0077 (8)	-0.0026 (7)	0.0008 (8)
C7	0.0322 (9)	0.0345 (10)	0.0163 (8)	-0.0142 (8)	0.0062 (7)	-0.0037 (7)
N4	0.0637 (14)	0.0823 (16)	0.0274 (10)	-0.0407 (13)	0.0125 (9)	-0.0150 (10)
O1	0.0690 (11)	0.0510 (9)	0.0277 (7)	-0.0306 (9)	0.0198 (7)	-0.0143 (7)
O2	0.0378 (8)	0.0585 (10)	0.0247 (7)	-0.0191 (7)	0.0001 (6)	0.0074 (7)
O5	0.0513 (14)	0.0524 (13)	0.0298 (10)	-0.0267 (11)	0.0064 (8)	-0.0162 (9)
O5'	0.107 (9)	0.048 (5)	0.029 (4)	-0.031 (5)	-0.014 (4)	-0.011 (3)

Geometric parameters (\AA , $^\circ$)

Co1—N2	2.0903 (18)	C1—C2	1.524 (2)
Co1—N2 ⁱ	2.0903 (18)	C2—C3	1.382 (3)
Co1—O3 ⁱ	2.1152 (14)	C2—C6	1.384 (3)
Co1—O3	2.1152 (14)	C3—C4	1.375 (3)
Co1—O4 ⁱ	2.1230 (13)	C3—H3A	0.9300
Co1—O4	2.1230 (13)	C4—H4A	0.9300
N1—C5	1.340 (2)	C5—C6	1.376 (3)

N1—C4	1.350 (2)	C5—H5A	0.9300
N1—C7	1.479 (2)	C6—H6A	0.9300
N2—N3	1.154 (2)	C7—C7 ⁱⁱ	1.519 (4)
N3—N4	1.159 (3)	C7—H7A	0.9700
O3—H31	0.826 (16)	C7—H7B	0.9700
O3—H32	0.847 (16)	O5—H5	0.824 (17)
O4—H42	0.846 (16)	O5—H51	0.802 (17)
O4—H41	0.812 (16)	O5'—H5	0.894 (17)
C1—O1	1.239 (3)	O5'—H52	0.85 (2)
C1—O2	1.256 (3)		
N2—Co1—N2 ⁱ	180.0	O1—C1—O2	126.83 (18)
N2—Co1—O3 ⁱ	89.06 (7)	O1—C1—C2	116.63 (18)
N2 ⁱ —Co1—O3 ⁱ	90.94 (7)	O2—C1—C2	116.51 (18)
N2—Co1—O3	90.94 (7)	C3—C2—C6	118.98 (16)
N2 ⁱ —Co1—O3	89.06 (7)	C3—C2—C1	120.08 (18)
O3 ⁱ —Co1—O3	180.00 (8)	C6—C2—C1	120.92 (18)
N2—Co1—O4 ⁱ	87.27 (7)	C4—C3—C2	119.67 (17)
N2 ⁱ —Co1—O4 ⁱ	92.73 (7)	C4—C3—H3A	120.2
O3 ⁱ —Co1—O4 ⁱ	88.65 (5)	C2—C3—H3A	120.2
O3—Co1—O4 ⁱ	91.35 (5)	N1—C4—C3	120.32 (17)
N2—Co1—O4	92.73 (7)	N1—C4—H4A	119.8
N2 ⁱ —Co1—O4	87.27 (7)	C3—C4—H4A	119.8
O3 ⁱ —Co1—O4	91.35 (5)	N1—C5—C6	120.57 (18)
O3—Co1—O4	88.65 (5)	N1—C5—H5A	119.7
O4 ⁱ —Co1—O4	180.00 (3)	C6—C5—H5A	119.7
C5—N1—C4	120.90 (15)	C5—C6—C2	119.56 (18)
C5—N1—C7	120.17 (15)	C5—C6—H6A	120.2
C4—N1—C7	118.94 (15)	C2—C6—H6A	120.2
N3—N2—Co1	148.27 (17)	N1—C7—C7 ⁱⁱ	109.17 (18)
N2—N3—N4	177.0 (2)	N1—C7—H7A	109.8
Co1—O3—H31	109.2 (19)	C7 ⁱⁱ —C7—H7A	109.8
Co1—O3—H32	113.4 (18)	N1—C7—H7B	109.8
H31—O3—H32	108 (2)	C7 ⁱⁱ —C7—H7B	109.8
Co1—O4—H42	111.1 (18)	H7A—C7—H7B	108.3
Co1—O4—H41	110.5 (19)	H5—O5—H51	121 (3)
H42—O4—H41	112 (2)	H5—O5'—H52	107 (3)
O3 ⁱ —Co1—N2—N3	129.7 (4)	C5—N1—C4—C3	0.1 (3)
O3—Co1—N2—N3	-50.3 (4)	C7—N1—C4—C3	179.88 (17)
O4 ⁱ —Co1—N2—N3	-141.6 (4)	C2—C3—C4—N1	-0.1 (3)
O4—Co1—N2—N3	38.4 (4)	C4—N1—C5—C6	-0.2 (3)
O1—C1—C2—C3	7.4 (3)	C7—N1—C5—C6	-179.94 (18)
O2—C1—C2—C3	-174.40 (18)	N1—C5—C6—C2	0.2 (3)
O1—C1—C2—C6	-170.78 (19)	C3—C2—C6—C5	-0.1 (3)
O2—C1—C2—C6	7.4 (3)	C1—C2—C6—C5	178.15 (18)
C6—C2—C3—C4	0.0 (3)	C5—N1—C7—C7 ⁱⁱ	-107.1 (2)

supplementary materials

C1—C2—C3—C4 -178.22 (17) C4—N1—C7—C7ⁱⁱ 73.1 (3)
Symmetry codes: (i) $-x+1, -y+1, -z$; (ii) $-x, -y, -z+2$.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O3—H31 \cdots O5 ⁱⁱⁱ	0.83 (2)	1.91 (2)	2.727 (2)	170 (2)
O3—H31 \cdots O5 ⁱⁱⁱ	0.83 (2)	1.96 (2)	2.664 (8)	143 (3)
O4—H41 \cdots O2 ^{iv}	0.81 (2)	2.07 (2)	2.870 (2)	167 (3)
O5—H51 \cdots O2 ^v	0.80 (2)	2.11 (2)	2.877 (3)	160 (4)
O3—H32 \cdots O1	0.85 (2)	1.85 (2)	2.694 (2)	178 (3)
O4—H42 \cdots O2	0.85 (2)	1.85 (2)	2.687 (2)	172 (2)
O5—H5 \cdots N4	0.82 (2)	2.19 (2)	2.864 (3)	139.(2)

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

Fig. 1

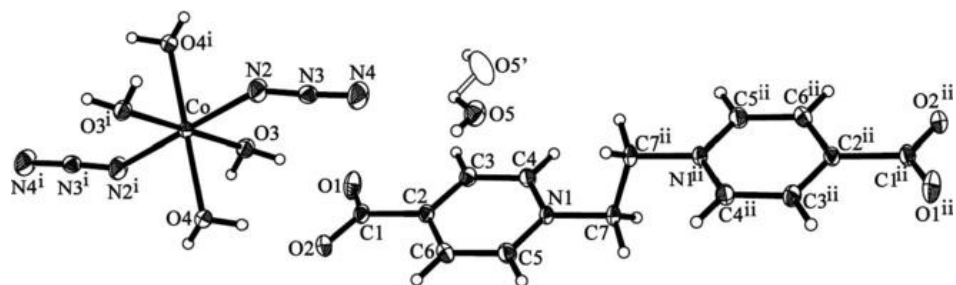


Fig. 2

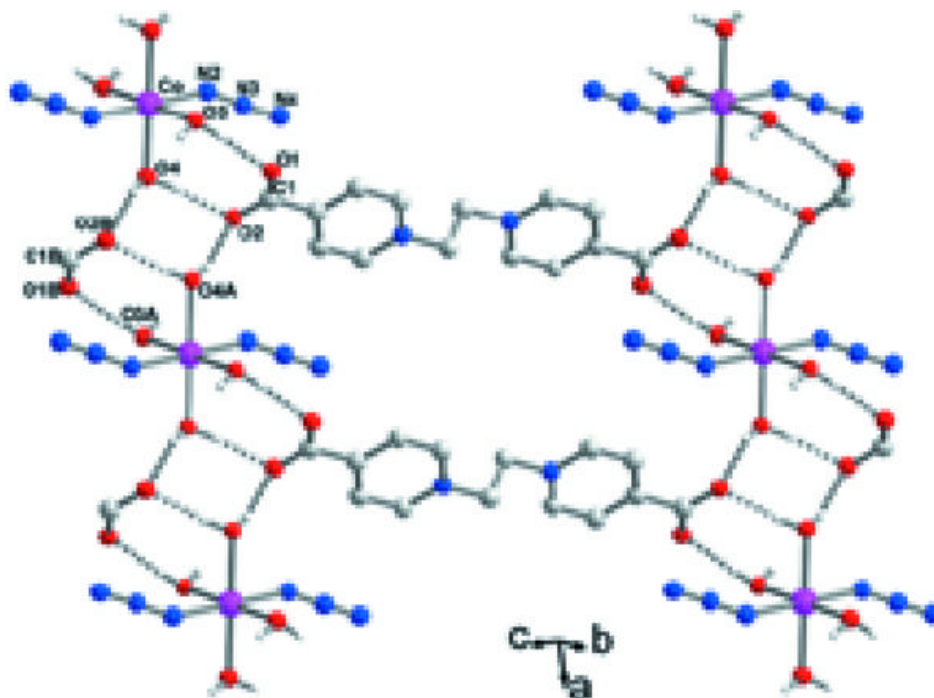


Fig. 3

