# metal-organic compounds

## Tetraaquadiazidocobalt(II) 3,3′-dicarboxylato-1,1′-ethylenedipyridinium

### Yan-Qing Wen, Chun-Yan Tian and En-Qing Gao*

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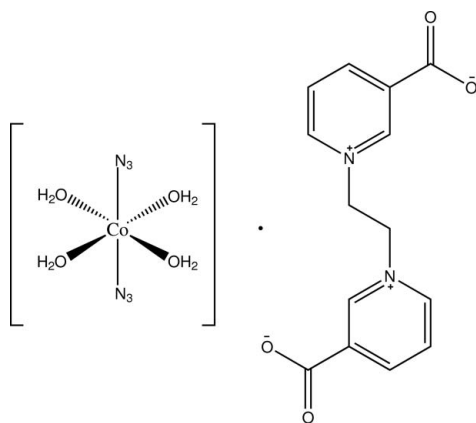Correspondence e-mail: eqgao@chem.ecnu.edu.cn

The asymmetric unit of the title compound, $[Co(N_3)_2(H_2O)_4]\cdot\cdot C_{14}H_{12}N_2O_4$, comprises half of the cobalt(II) complex molecule and a half of the 3,3′-dicarboxylato-1,1′-ethylenedipyridinium molecule. The $Co^{II}$ atom is located on an inversion centre and hence the complex molecule adopts a centrosymmetric *trans*-octahedral geometry. The zwitterionic organic molecule is also centrosymmetric with the centre of the C—C bond of the ethylene moiety coinciding with an inversion centre. The adduct of metal complex and organic molecule is associated into a three-dimenional network through O—H···O hydrogen bonds.

## Related literature

For background to hydrogen bonds, see: Braga & Grepioni (2000); Fabbiani *et al.* (2010); Salitros *et al.* (2010); Schultheis *et al.* (2010). For the synthesis of the ligand, see: Loeb *et al.* (2006). For hydrogen-bond motifs, see: Bernstein *et al.* (1995); Etter (1990).



## Experimental

### Crystal data

| | |
|---|---|
| $[Co(N_3)_2(H_2O)_4]\cdot C_{14}H_{12}N_2O_4$ | $\gamma$ = 95.011 (2)° |
| $M_r$ = 487.31 | $V$ = 488.71 (7) Å³ |
| Triclinic, $P\bar{1}$ | $Z$ = 1 |
| $a$ = 7.4309 (6) Å | Mo $K\alpha$ radiation |
| $b$ = 7.7507 (7) Å | $\mu$ = 0.94 mm⁻¹ |
| $c$ = 8.5582 (7) Å | $T$ = 296 K |
| $\alpha$ = 95.463 (2)° | 0.25 × 0.20 × 0.15 mm |
| $\beta$ = 90.586 (2)° | |

### Data collection

| | |
|---|---|
| Bruker APEXII CCD area-detector diffractometer | 6091 measured reflections |
| | 1907 independent reflections |
| Absorption correction: multi-scan (*SADABS*; Bruker, 2008) | 1889 reflections with $I > 2\sigma(I)$ |
| $T_{min}$ = 0.799, $T_{max}$ = 0.872 | $R_{int}$ = 0.015 |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)]$ = 0.024 | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2)$ = 0.074 | |
| $S$ = 1.12 | |
| 1907 reflections | $\Delta\rho_{max}$ = 0.28 e Å⁻³ |
| 154 parameters | $\Delta\rho_{min}$ = −0.28 e Å⁻³ |
| 9 restraints | |

**Table 1**
Selected bond lengths (Å).

| | | | |
|---|---|---|---|
| Co1—O4 | 2.0780 (12) | Co1—O3 | 2.1431 (12) |
| Co1—N2 | 2.0958 (15) | | |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O3—H3$B$···O1$^i$ | 0.84 (2) | 2.01 (2) | 2.8180 (18) | 163 (2) |
| O3—H3$C$···O1$^{ii}$ | 0.84 (2) | 1.91 (2) | 2.7395 (17) | 172 (2) |
| O4—H4$C$···O2$^{iii}$ | 0.86 (2) | 1.84 (2) | 2.6901 (18) | 173 (3) |
| O4—H4$B$···O2 | 0.81 (2) | 2.03 (2) | 2.8028 (18) | 159 (2) |

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

**References**

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# supporting information

## Tetraaquadiazidocobalt(II) 3,3′-dicarboxylato-1,1′-ethylenedipyridinium

**Yan-Qing Wen, Chun-Yan Tian and En-Qing Gao**

### S1. Comment

Hydrogen bonds play a key role in biological systems and materials, and they have been widely used as a putative supramolecular tool for engineering organic and metal-organic solids (Fabbiani *et al.*, 2010; Salitros *et al.*, 2010; Schultheis *et al.*, 2010; Braga & Grepioni, 2000). In this paper, we report the structure of the title compound, (I), which contains a neutral metal complex molecule, $[Co(N_3)_2(H_2O)_4]$, and a zwitterionic dicarboxylate, 1,2-bis(3-carboxyl-atopyridinium)ethane (Fig. 1). The metal complex molecule is centrosymmetric, with the octahedral-coordinated $Co^{II}$ by two azide anions and four water molecules in a *trans* arrangement (Fig. 1, Table 1). Two opposite Co—O distances are longer than the Co—N and other Co—O ones, defining an axially elongated geometry. The zwitterionic molecule is also centrosymmetric (Fig.1 ). The inorganic complex molecules and the carboxylate groups are associated into a sheet through O—H⋯O hydrogen bonds involving the coordinated aqua ligands (O3 and O4) and the carboxylate oxygen atoms (O1 and O2) (Fig. 2, Table 2). The two O4 aqua ligands from symmetry related complex molecules and two O2 atoms from symmetry related 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). Similar hydrogen-bonded rings are formed by O1 and O3. The carboxylate group forms a $R_2^2(8)$ hydrogen-bonded ring with two aqua ligands from the same complex molecule. Besides, a large hydrogen-bonded ring $[R_4^4(16)]$ is formed by two carboxylate groups and four aqua ligands from two complex molecules. The organic ligands interlink the hydrogen-bonded sheets of the metal complexes into the three-dimensional structure (Fig. 3).

### S2. Experimental

The zwitterionic ligand ($[H_2L^1]Br_2$) was synthesised from 1,2-dibromoethane and ethyl nicotinate according to the published procedure (Loeb *et al.*, 2006). An aqueous solution (4 mL) of $[H_2L^1]Br_2$ (0.1 mmol) and $NaN_3$ (1 mmol) was added to a DMF solution (1.5 mL) of $Co(ClO_4)_2.6H_2O$ (0.2 mmol) with stirring. The resulting solution was allowed to evaporate slowly at room temperature, yielding light-red block crystals of (I) in three days. Yield: 75%. Anal. calcd (found) (%) for $CoC_{14}H_{20}N_8O_8$: C, 34.79 (34.51); H, 4.39 (4.14); N, 22.87 (23.00). Main IR bands (KBr, *v*/cm$^{-1}$): 3427*m*, 3097w, 2042 s, 1637 s, 1606 s, 1392*m*, 765*m*, 688*m*.

### S3. 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 $CH_2$. 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 $U_{iso}(H) = 1.5U_{eq}(O)$. The 'rigid-bond′ restraint was applied on the azide moiety (N2—N3—N4) using the *SHELXL* DELU instruction.
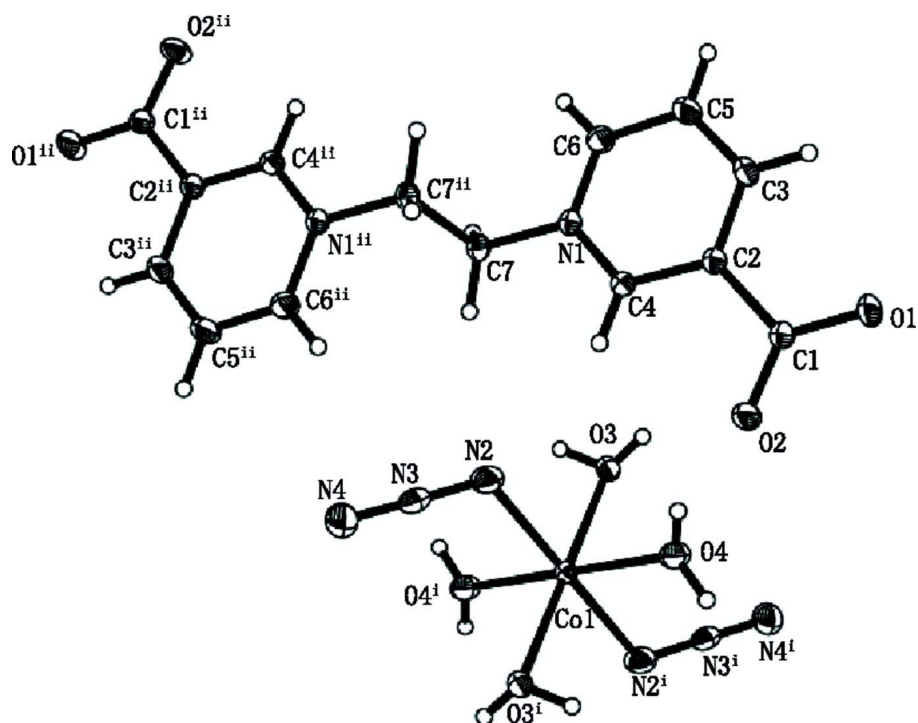
**Figure 1**

The molecular structure of the title compound showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. [Symmetry code: (i) -*x*, -*y*, 1 - *z*; (ii) 1 - *x*, -*y*, -*z*].
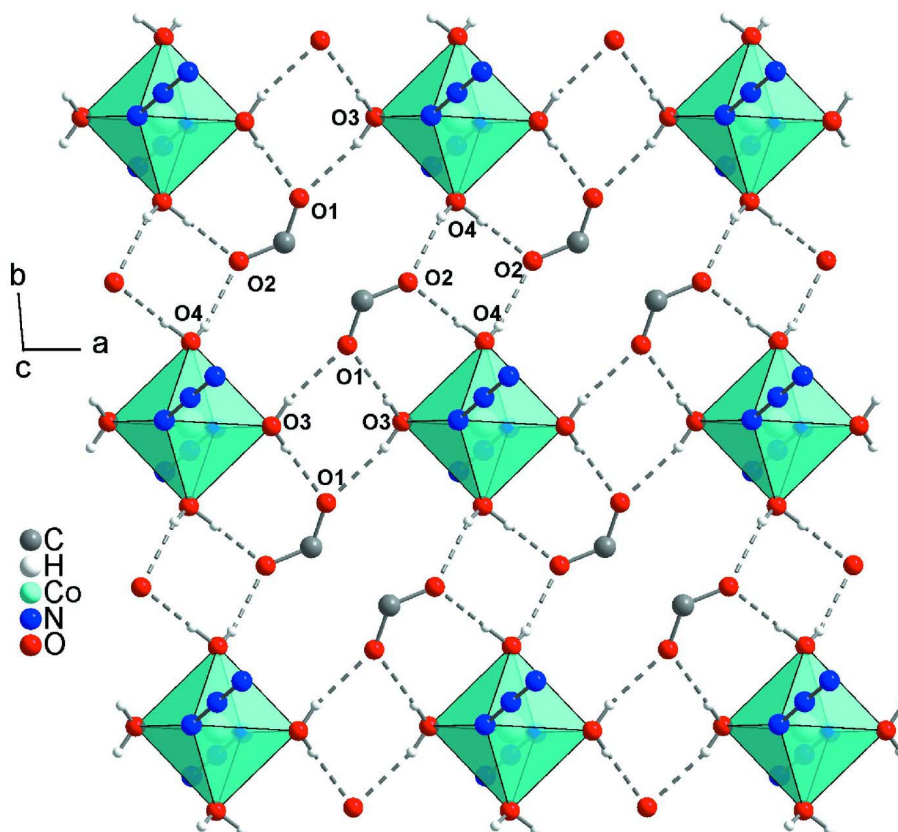
**Figure 2**

Two-dimensional layer structure connected through intermolecula O—H···O hydrogen bonds. [Symmetry code: (i) -*x* + 1, -*y* + 1, -*z* + 1; (ii) *x*, *y* - 1, *z*; (iii)-*x*, -*y* + 1, -*z* + 1].
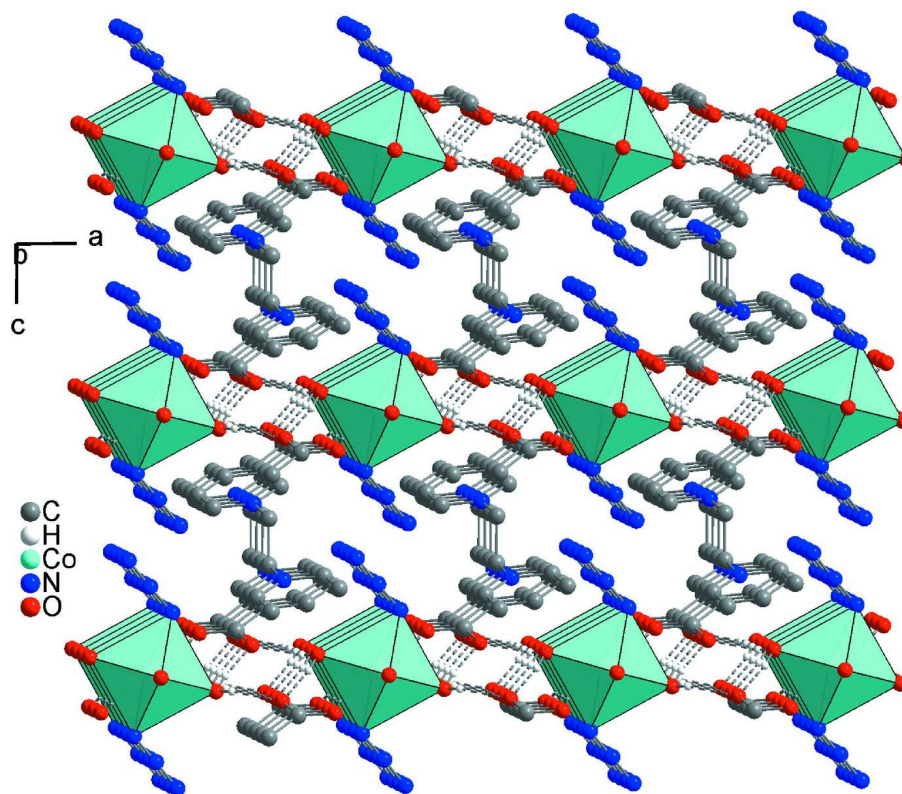
**Figure 3**

Three dimensional structure connected by the organic ligands interlinking the hydrogen-bonded sheets.

**Tetraaquadiazidocobalt(II) 3,3′-dicarboxylato-1,1′-ethylenedipyridinium**

*Crystal data*

| | |
|---|---|
| $[Co(N_3)_2(H_2O)_4]·C_{14}H_{12}N_2O_4$ | $Z = 1$ |
| $M_r = 487.31$ | $F(000) = 251$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.656$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $a = 7.4309$ (6) Å | Cell parameters from 15377 reflections |
| $b = 7.7507$ (7) Å | $\theta = 3.4–27.5°$ |
| $c = 8.5582$ (7) Å | $\mu = 0.94$ mm$^{-1}$ |
| $\alpha = 95.463$ (2)° | $T = 296$ K |
| $\beta = 90.586$ (2)° | Block, red |
| $\gamma = 95.011$ (2)° | $0.25 \times 0.20 \times 0.15$ mm |
| $V = 488.71$ (7) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Bruker APEXII CCD area-detector diffractometer | 6091 measured reflections |
| Radiation source: fine-focus sealed tube | 1907 independent reflections |
| Graphite monochromator | 1889 reflections with $I > 2\sigma(I)$ |
| phi and $\omega$ scans | $R_{int} = 0.015$ |
| Absorption correction: multi-scan (*SADABS*; Bruker, 2008) | $\theta_{max} = 26.1°$, $\theta_{min} = 2.4°$ |
| | $h = -9 \rightarrow 9$ |
| | $k = -9 \rightarrow 8$ |
| $T_{min} = 0.799$, $T_{max} = 0.872$ | $l = -10 \rightarrow 10$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier |
| Least-squares matrix: full | map |
| $R[F^2 > 2\sigma(F^2)] = 0.024$ | Hydrogen site location: inferred from |
| $wR(F^2) = 0.074$ | neighbouring sites |
| $S = 1.12$ | H atoms treated by a mixture of independent |
| 1907 reflections | and constrained refinement |
| 154 parameters | $w = 1/[\sigma^2(F_o^2) + (0.0411P)^2 + 0.2239P]$ |
| 9 restraints | where $P = (F_o^2 + 2F_c^2)/3$ |
| Primary atom site location: structure-invariant | $(\Delta/\sigma)_{max} < 0.001$ |
| direct methods | $\Delta\rho_{max} = 0.28$ e Å$^{-3}$ |
| | $\Delta\rho_{min} = -0.28$ e Å$^{-3}$ |

*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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Co1 | 0.0000 | 0.0000 | 0.5000 | 0.02282 (12) |
| C1 | 0.3729 (2) | 0.5896 (2) | 0.3420 (2) | 0.0283 (3) |
| C2 | 0.5025 (2) | 0.4708 (2) | 0.26023 (19) | 0.0258 (3) |
| C3 | 0.6822 (2) | 0.5278 (2) | 0.2423 (2) | 0.0335 (4) |
| H3A | 0.7234 | 0.6424 | 0.2751 | 0.040* |
| C4 | 0.4437 (2) | 0.3011 (2) | 0.20731 (19) | 0.0255 (3) |
| H4A | 0.3228 | 0.2613 | 0.2153 | 0.031* |
| C5 | 0.8009 (2) | 0.4148 (3) | 0.1756 (3) | 0.0392 (4) |
| H5A | 0.9215 | 0.4530 | 0.1625 | 0.047* |
| C6 | 0.7382 (2) | 0.2466 (2) | 0.1293 (2) | 0.0339 (4) |
| H6A | 0.8173 | 0.1685 | 0.0875 | 0.041* |
| C7 | 0.5003 (2) | 0.0111 (2) | 0.08913 (19) | 0.0286 (3) |
| H7A | 0.3795 | −0.0177 | 0.1265 | 0.034* |
| H7B | 0.5803 | −0.0667 | 0.1303 | 0.034* |
| N1 | 0.56206 (18) | 0.19334 (17) | 0.14405 (16) | 0.0257 (3) |
| N2 | 0.0817 (2) | −0.0222 (2) | 0.26583 (18) | 0.0399 (4) |
| N3 | −0.0146 (2) | −0.1006 (2) | 0.16704 (17) | 0.0320 (3) |
| N4 | −0.1073 (3) | −0.1740 (2) | 0.0672 (2) | 0.0461 (4) |
| O1 | 0.43867 (18) | 0.73681 (16) | 0.39597 (19) | 0.0435 (4) |
| O2 | 0.21281 (17) | 0.52889 (16) | 0.35044 (18) | 0.0405 (3) |
| O3 | 0.27781 (16) | −0.00456 (16) | 0.56790 (15) | 0.0319 (3) |
| H3B | 0.347 (3) | 0.086 (2) | 0.569 (3) | 0.048* |
| H3C | 0.321 (3) | −0.082 (2) | 0.508 (3) | 0.048* |
| O4 | 0.02796 (18) | 0.26995 (16) | 0.50991 (18) | 0.0379 (3) |

| | | | |
|---|---|---|---|
| H4B | 0.090 (3) | 0.323 (3) | 0.449 (3) | 0.057* |
| H4C | −0.053 (3) | 0.334 (3) | 0.547 (3) | 0.057* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Co1 | 0.02008 (17) | 0.02004 (17) | 0.02747 (17) | −0.00003 (11) | 0.00302 (11) | −0.00082 (11) |
| C1 | 0.0273 (8) | 0.0202 (7) | 0.0363 (8) | 0.0007 (6) | 0.0062 (7) | −0.0031 (6) |
| C2 | 0.0247 (8) | 0.0210 (7) | 0.0305 (8) | 0.0009 (6) | 0.0035 (6) | −0.0024 (6) |
| C3 | 0.0282 (9) | 0.0247 (8) | 0.0445 (10) | −0.0047 (7) | 0.0040 (7) | −0.0067 (7) |
| C4 | 0.0220 (7) | 0.0230 (8) | 0.0301 (8) | 0.0002 (6) | 0.0040 (6) | −0.0026 (6) |
| C5 | 0.0231 (8) | 0.0348 (9) | 0.0565 (12) | −0.0035 (7) | 0.0081 (8) | −0.0077 (8) |
| C6 | 0.0247 (8) | 0.0311 (9) | 0.0447 (10) | 0.0045 (7) | 0.0068 (7) | −0.0056 (7) |
| C7 | 0.0331 (8) | 0.0179 (7) | 0.0336 (9) | 0.0000 (6) | 0.0054 (7) | −0.0030 (6) |
| N1 | 0.0256 (7) | 0.0203 (6) | 0.0297 (7) | 0.0006 (5) | 0.0035 (5) | −0.0043 (5) |
| N2 | 0.0335 (8) | 0.0555 (10) | 0.0297 (8) | −0.0006 (7) | 0.0064 (6) | 0.0028 (7) |
| N3 | 0.0314 (8) | 0.0343 (8) | 0.0318 (8) | 0.0069 (6) | 0.0117 (7) | 0.0059 (6) |
| N4 | 0.0475 (10) | 0.0485 (10) | 0.0398 (9) | −0.0053 (8) | 0.0032 (8) | −0.0011 (8) |
| O1 | 0.0330 (7) | 0.0226 (6) | 0.0701 (10) | −0.0026 (5) | 0.0105 (6) | −0.0159 (6) |
| O2 | 0.0282 (6) | 0.0241 (6) | 0.0665 (9) | −0.0017 (5) | 0.0160 (6) | −0.0082 (6) |
| O3 | 0.0230 (6) | 0.0274 (6) | 0.0434 (7) | 0.0016 (5) | 0.0014 (5) | −0.0067 (5) |
| O4 | 0.0356 (7) | 0.0210 (6) | 0.0575 (8) | 0.0026 (5) | 0.0178 (6) | 0.0028 (5) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Co1—O4 | 2.0780 (12) | C5—C6 | 1.366 (3) |
| Co1—O4$^i$ | 2.0780 (12) | C5—H5A | 0.9300 |
| Co1—N2 | 2.0958 (15) | C6—N1 | 1.349 (2) |
| Co1—N2$^i$ | 2.0958 (15) | C6—H6A | 0.9300 |
| Co1—O3$^i$ | 2.1431 (12) | C7—N1 | 1.478 (2) |
| Co1—O3 | 2.1431 (12) | C7—C7$^{ii}$ | 1.519 (3) |
| C1—O1 | 1.245 (2) | C7—H7A | 0.9700 |
| C1—O2 | 1.246 (2) | C7—H7B | 0.9700 |
| C1—C2 | 1.521 (2) | N2—N3 | 1.188 (2) |
| C2—C4 | 1.381 (2) | N3—N4 | 1.164 (2) |
| C2—C3 | 1.384 (2) | O3—H3B | 0.836 (15) |
| C3—C5 | 1.386 (3) | O3—H3C | 0.839 (15) |
| C3—H3A | 0.9300 | O4—H4B | 0.813 (16) |
| C4—N1 | 1.348 (2) | O4—H4C | 0.856 (16) |
| C4—H4A | 0.9300 | | |
| | | | |
| O4—Co1—O4$^i$ | 180.0 | N1—C4—H4A | 120.1 |
| O4—Co1—N2 | 91.41 (6) | C2—C4—H4A | 120.1 |
| O4$^i$—Co1—N2 | 88.59 (6) | C6—C5—C3 | 119.02 (16) |
| O4—Co1—N2$^i$ | 88.59 (6) | C6—C5—H5A | 120.5 |
| O4$^i$—Co1—N2$^i$ | 91.41 (6) | C3—C5—H5A | 120.5 |
| N2—Co1—N2$^i$ | 180.0 | N1—C6—C5 | 120.23 (16) |
| O4—Co1—O3$^i$ | 88.83 (5) | N1—C6—H6A | 119.9 |

| | | | |
|---|---|---|---|
| O4<sup>i</sup>—Co1—O3<sup>i</sup> | 91.17 (5) | C5—C6—H6A | 119.9 |
| N2—Co1—O3<sup>i</sup> | 92.17 (6) | N1—C7—C7<sup>ii</sup> | 109.09 (16) |
| N2<sup>i</sup>—Co1—O3<sup>i</sup> | 87.83 (6) | N1—C7—H7A | 109.9 |
| O4—Co1—O3 | 91.17 (5) | C7<sup>ii</sup>—C7—H7A | 109.9 |
| O4<sup>i</sup>—Co1—O3 | 88.83 (5) | N1—C7—H7B | 109.9 |
| N2—Co1—O3 | 87.83 (6) | C7<sup>ii</sup>—C7—H7B | 109.9 |
| N2<sup>i</sup>—Co1—O3 | 92.17 (6) | H7A—C7—H7B | 108.3 |
| O3<sup>i</sup>—Co1—O3 | 180.0 | C4—N1—C6 | 121.82 (14) |
| O1—C1—O2 | 126.73 (15) | C4—N1—C7 | 120.05 (13) |
| O1—C1—C2 | 116.50 (14) | C6—N1—C7 | 118.13 (14) |
| O2—C1—C2 | 116.75 (14) | N3—N2—Co1 | 120.05 (12) |
| C4—C2—C3 | 118.77 (14) | N4—N3—N2 | 178.01 (19) |
| C4—C2—C1 | 120.22 (14) | Co1—O3—H3B | 119.4 (17) |
| C3—C2—C1 | 120.97 (14) | Co1—O3—H3C | 107.1 (17) |
| C2—C3—C5 | 120.26 (16) | H3B—O3—H3C | 108.0 (19) |
| C2—C3—H3A | 119.9 | Co1—O4—H4B | 122.9 (18) |
| C5—C3—H3A | 119.9 | Co1—O4—H4C | 123.1 (17) |
| N1—C4—C2 | 119.85 (14) | H4B—O4—H4C | 109 (2) |
| | | | |
| O1—C1—C2—C4 | 175.22 (17) | C2—C4—N1—C6 | −0.5 (3) |
| O2—C1—C2—C4 | −3.6 (2) | C2—C4—N1—C7 | 179.21 (14) |
| O1—C1—C2—C3 | −2.5 (3) | C5—C6—N1—C4 | −1.6 (3) |
| O2—C1—C2—C3 | 178.67 (17) | C5—C6—N1—C7 | 178.70 (17) |
| C4—C2—C3—C5 | −1.5 (3) | C7<sup>ii</sup>—C7—N1—C4 | 108.4 (2) |
| C1—C2—C3—C5 | 176.28 (18) | C7<sup>ii</sup>—C7—N1—C6 | −71.9 (2) |
| C3—C2—C4—N1 | 2.0 (2) | O4—Co1—N2—N3 | −122.40 (16) |
| C1—C2—C4—N1 | −175.75 (15) | O4<sup>i</sup>—Co1—N2—N3 | 57.60 (16) |
| C2—C3—C5—C6 | −0.6 (3) | O3<sup>i</sup>—Co1—N2—N3 | −33.51 (16) |
| C3—C5—C6—N1 | 2.1 (3) | O3—Co1—N2—N3 | 146.49 (16) |

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

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O3—H3*B*···O1<sup>iii</sup> | 0.84 (2) | 2.01 (2) | 2.8180 (18) | 163 (2) |
| O3—H3*C*···O1<sup>iv</sup> | 0.84 (2) | 1.91 (2) | 2.7395 (17) | 172 (2) |
| O4—H4*C*···O2<sup>v</sup> | 0.86 (2) | 1.84 (2) | 2.6901 (18) | 173 (3) |
| O4—H4*B*···O2 | 0.81 (2) | 2.03 (2) | 2.8028 (18) | 159 (2) |

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