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# (Adipato-$\kappa^2O,O'$)diaqua[bis(pyridin-2-yl-$\kappa N$)amine]cobalt(II) trihydrate

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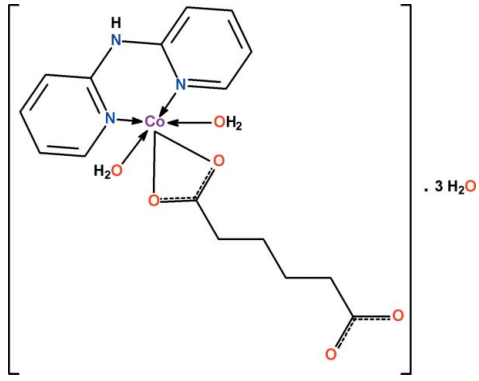
Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.030; $wR$ factor = 0.097; data-to-parameter ratio = 26.8.

In the monomeric title complex, $[Co(C_6H_8O_4)(C_{10}H_9N_3)(H_2O)_2]\cdot 3H_2O$, the distorted octahedral $CoN_2O_4$ coordination environment comprises two N-atom donors from the bidentate dipyridyldiamine ligand, two O-atom donors from one of the carboxylate groups of the bidentate chelating adipate ligand and two water molecules. In addition, there are three solvent water molecules which are involved in both intra- and inter-unit O—H···O hydrogen-bonding interactions, which together with an amine–water N—H···O hydrogen bond produce a three-dimensional framework.

## Related literature

For the background to metal-dicarboxylate complexes, see: Rao *et al.* (2004); Setifi *et al.* (2006, 2007); Wen *et al.* (2010).




## Experimental

### Crystal data

$[Co(C_6H_8O_4)(C_{10}H_9N_3)(H_2O)_2]\cdot 3H_2O$
$M_r$ = 464.34
Triclinic, $P\overline{1}$
$a$ = 9.9587 (3) Å
$b$ = 10.5458 (3) Å
$c$ = 11.0885 (3) Å
$\alpha$ = 100.887 (1)°
$\beta$ = 105.891 (1)°
$\gamma$ = 107.889 (1)°
$V$ = 1017.38 (5) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.90 mm$^{-1}$
$T$ = 296 K
0.26 × 0.21 × 0.18 mm

### Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
$T_{min}$ = 0.800, $T_{max}$ = 0.855
28998 measured reflections
8197 independent reflections
6984 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.024

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.097
$S$ = 0.99
8197 reflections
306 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.37 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.34 e Å$^{-3}$

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

| | | | |
|---|---|---|---|
| Co1—O1 | 2.0680 (10) | Co1—O6 | 2.1336 (9) |
| Co1—O2 | 2.3079 (9) | Co1—N1 | 2.0781 (9) |
| Co1—O5 | 2.0877 (12) | Co1—N2 | 2.0596 (10) |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N3—H3···O9$^{i}$ | 0.78 (2) | 2.05 (2) | 2.8228 (17) | 172 (2) |
| O5—H51···O7$^{ii}$ | 0.775 (19) | 1.923 (19) | 2.6933 (15) | 172.7 (19) |
| O5—H52···O3$^{iii}$ | 0.80 (2) | 1.97 (2) | 2.7706 (17) | 173 (2) |
| O6—H61···O2$^{iv}$ | 0.83 (2) | 2.00 (2) | 2.8278 (12) | 178 (4) |
| O6—H62···O8 | 0.77 (2) | 1.94 (2) | 2.7056 (16) | 170 (2) |
| O7—H71···O3$^{v}$ | 0.74 (3) | 2.56 (3) | 3.2596 (18) | 160 (3) |
| O7—H72···O1 | 0.89 (2) | 1.86 (2) | 2.7543 (16) | 175 (2) |
| O8—H81···O4$^{v}$ | 0.88 (3) | 1.97 (3) | 2.8221 (19) | 163 (2) |
| O8—H82···O4$^{vi}$ | 0.79 (3) | 2.05 (3) | 2.832 (2) | 175 (3) |
| O9—H91···O3$^{vii}$ | 0.77 (2) | 2.12 (2) | 2.8670 (17) | 164 (2) |
| O9—H92···O3$^{v}$ | 0.79 (3) | 1.99 (3) | 2.7452 (17) | 160 (3) |

Symmetry codes: (i) $-x+1, -y+1, -z$; (ii) $-x+2, -y+1, -z$; (iii) $-x+3, -y+1, -z+1$; (iv) $-x+2, -y+1, -z+1$; (v) $-x+3, -y+2, -z+1$; (vi) $x-1, y, z$; (vii) $x-1, y, z-1$.

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON* and *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ5322).

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# supplementary materials

*Acta Cryst.* (2013). E**69**, m335–m336 [doi:10.1107/S1600536813012981]

## (Adipato-$\kappa^2O,O'$)diaqua[bis(pyridin-2-yl-$\kappa N$)amine]cobalt(II) trihydrate

**Zouaoui Setifi, Fatima Setifi, Graham Smith, Malika El-Ghozzi, Djamil-Azzeddine Rouag, Daniel Avignant and Hocine Merazig**

### Comment

Dicarboxylates have been widely used as ligands in metal coordination chemistry because they possess interesting features, such as: (i) the presence of two carboxylato groups capable of bidentate and monodentate linking modes, (ii) the possibility of obtaining mono- or dianionic forms, (iii) the probability of triply coordinated oxygen atoms and (iv) the possibility of forming secondary building blocks (Rao *et al.*, 2004). Their use as bridging ligands has generated metal-organic coordination polymers with diverse and interesting structural features (Setifi *et al.*, 2006, 2007; Wen *et al.*, 2010). Given the rich coordination chemistry and the flexibility of these anionic ligands, we are interested in using them in combination with other chelating co-ligands to explore their structural features and properties in the large field of molecular materials. This led us to the synthesis of the parent coordination compound, the title complex [Co(dpa)(adip)$(H_2O)_2$]. $3H_2O$, (where dpa is 2,2′-dipyridylamine and adip is the adipate dianion), and the structure is described herein.

In this monomeric complex, the distorted octahedral $MN_2O_4$ coordination sphere comprises two N-donors from the bidentate chelate dpa ligand, two carboxyl O-donors (O1, O2) from one of the carboxyl groups of the adipate ligand and two water molecules (O5, O6). In addition, there are three water molecules of solvation (O7–O9) (Fig. 1). The (N,*N*′) interaction is essentially symmetric [Co—N, 2.0596 (10), 2.0781 (9) Å] (Table 1) but the (O,*O*′) interaction is asymmetric [Co—O, 2.0680 (10), 2.3079 (9) Å], with a 'bite′ angle of 59.68 (3)°. The second adipate carboxyl group is not involved in coordination.

In the crystal, both the coordinated water molecules and the solvent water molecules are involved in both intra- and inter-unit O—H···O hydrogen-bonding interactions. The amine N-atom of the dpa ligand is also hydrogen-bonded to a water molecule (O9) (Table 2), giving an overall three-dimensional framework structure (Fig. 2).

### Experimental

All reagents were purchased from commercial sources and used as received. Under aerobic conditions, an ethanolic solution of 2,2′-dipyridylamine (0.017 g, 5 ml) was added, with stirring at room temperature to an ethanolic solution of $CoCl_2.6H_2O$ (0.024 g, 5 ml), resulting in a pink suspension. Adipic acid was dissolved in water (0.015 g, 10 ml) and added quickly to the mixture. The final solution was filtered and the filtrate allowed to evaporate in air for two weeks, giving brown crystals of the title compound suitable for X-ray diffraction analysis.

### Refinement

All H-atoms potentially involved in hydrogen-bonding were located from a difference-Fourier and both positional and isotropic displacement parameters were refined. Other H-atoms were placed in calculated positions with C—H(aromatic) = 0.93 Å or *C*—*H*(methylene) = 0.97 Å and with $U_{iso}$(H) = 1.2$U_{eq}$(C). Several reflections (8), considered to be affected by beam stop interference were omitted from the refinement.

### Computing details

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT* (Bruker, 2008); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

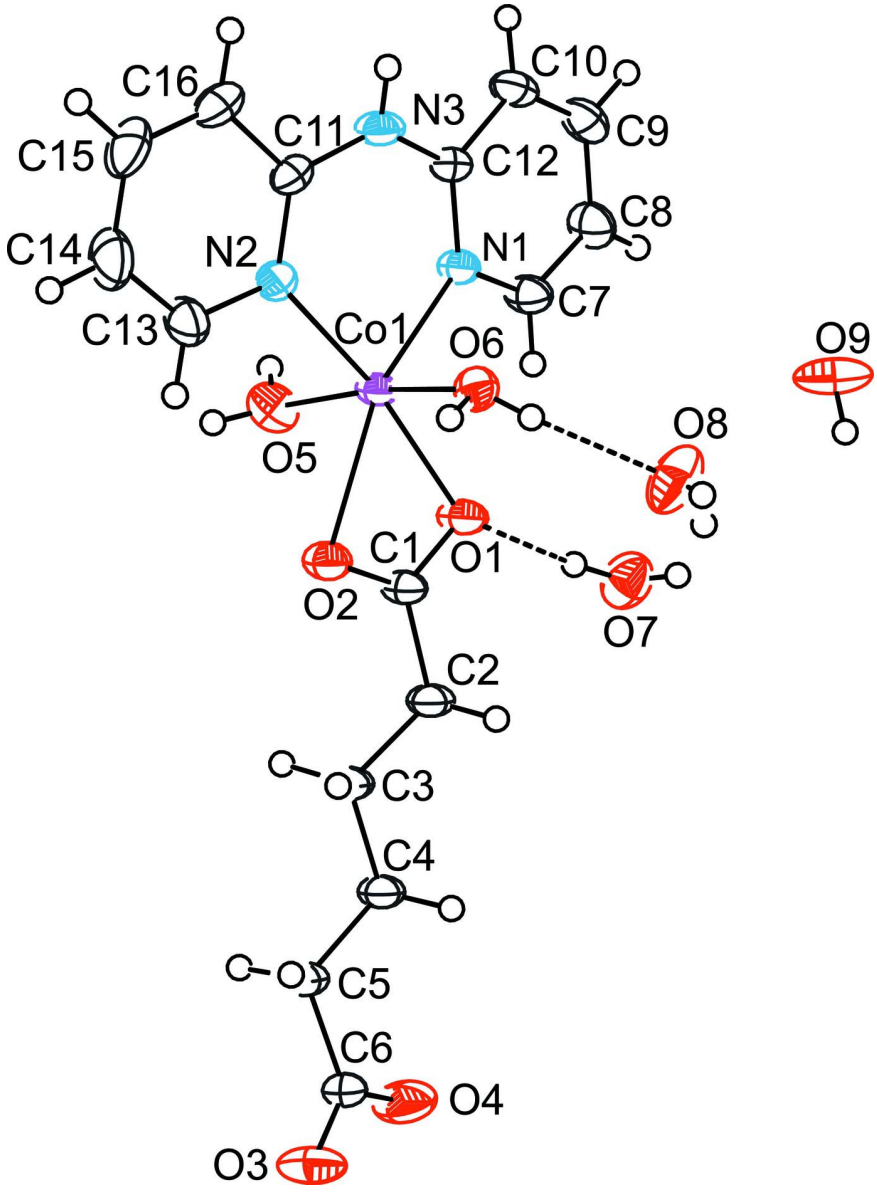


**Figure 1**

A view of the title compound with the atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

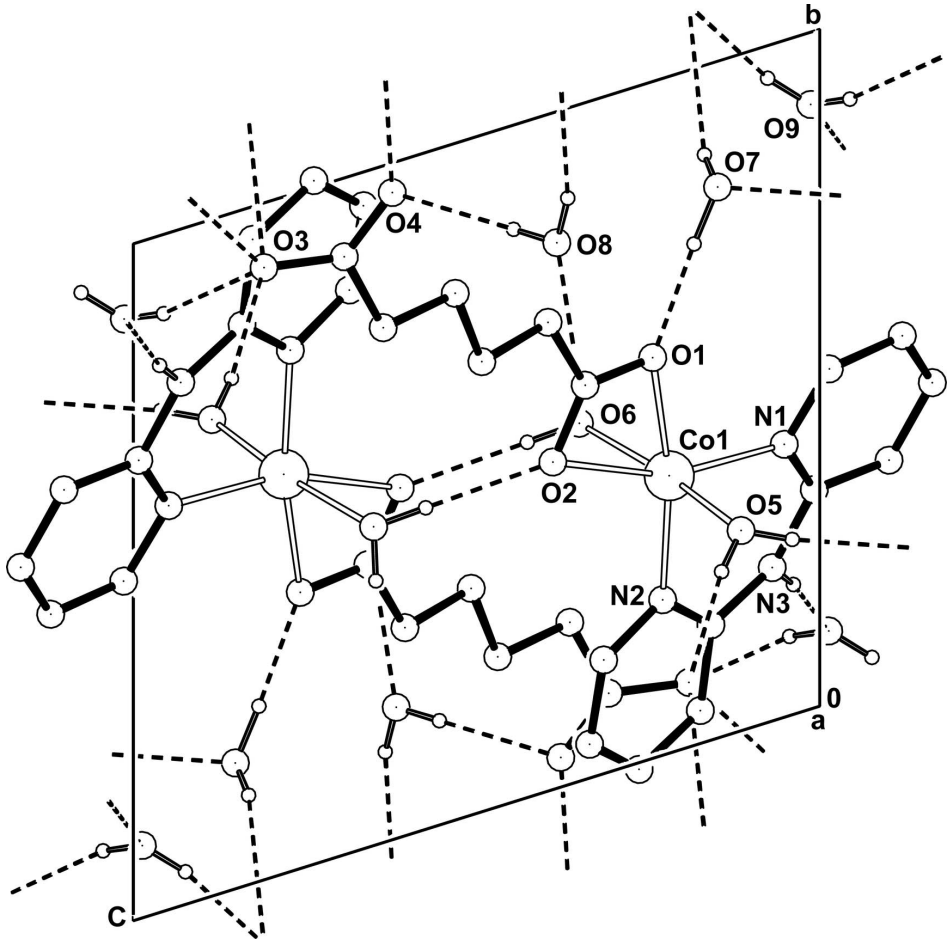


**Figure 2**

The crystal packing of the title compound in the unit cell viewed down a, showing hydrogen bonds as dashed lines. Non-associative H-atoms are omitted.

### (Adipato-$\kappa^2O,O'$)diaqua[bis(pyridin-2-yl-$\kappa N$)amine]cobalt(II) trihydrate

*Crystal data*

$[Co(C_6H_8O_4)(C_{10}H_9N_3)(H_2O)_2]\cdot 3H_2O$
$M_r = 464.34$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 9.9587$ (3) Å
$b = 10.5458$ (3) Å
$c = 11.0885$ (3) Å
$\alpha = 100.887$ (1)°
$\beta = 105.891$ (1)°
$\gamma = 107.889$ (1)°
$V = 1017.38$ (5) Å$^3$
$Z = 2$
$F(000) = 486$
$D_x = 1.516$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9938 reflections
$\theta = 2.5$–$33.9$°
$\mu = 0.90$ mm$^{-1}$
$T = 296$ K
Prism, brown
0.26 × 0.21 × 0.18 mm

*Data collection*

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans

Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
$T_{min}$ = 0.800, $T_{max}$ = 0.855
28998 measured reflections
8197 independent reflections
6984 reflections with $I > 2\sigma(I)$

$R_{int}$ = 0.024
$\theta_{max}$ = 33.9°, $\theta_{min}$ = 3.3°
$h$ = −15→15
$k$ = −16→16
$l$ = −17→17

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.097
$S$ = 0.99
8197 reflections
306 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0677P)^2 + 0.048P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.37 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.34 e Å$^{-3}$

*Special details*

**Geometry**. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

**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 $F^2 > 2\sigma(F^2)$ 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 | 0.87882 (1) | 0.40987 (1) | 0.21923 (1) | 0.0256 (1) |
| O1 | 1.05309 (9) | 0.59199 (9) | 0.24329 (8) | 0.0346 (2) |
| O2 | 1.10712 (9) | 0.48396 (9) | 0.38874 (8) | 0.0334 (2) |
| O3 | 1.92943 (11) | 0.90473 (12) | 0.80972 (9) | 0.0509 (3) |
| O4 | 1.88720 (12) | 0.95610 (12) | 0.62266 (12) | 0.0579 (3) |
| O5 | 0.97049 (11) | 0.29574 (11) | 0.11448 (10) | 0.0401 (3) |
| O6 | 0.80952 (9) | 0.52897 (9) | 0.35009 (8) | 0.0318 (2) |
| N1 | 0.71591 (9) | 0.40327 (9) | 0.05172 (8) | 0.0259 (2) |
| N2 | 0.73045 (10) | 0.22973 (9) | 0.22883 (9) | 0.0289 (2) |
| N3 | 0.51381 (10) | 0.22666 (11) | 0.06928 (10) | 0.0338 (2) |
| C1 | 1.14696 (10) | 0.58243 (11) | 0.34175 (9) | 0.0274 (2) |
| C2 | 1.30419 (11) | 0.69271 (12) | 0.39566 (11) | 0.0329 (3) |
| C3 | 1.42382 (11) | 0.66497 (12) | 0.49212 (11) | 0.0347 (3) |
| C4 | 1.57881 (11) | 0.77823 (11) | 0.53102 (11) | 0.0315 (3) |
| C5 | 1.69945 (12) | 0.76076 (12) | 0.63591 (12) | 0.0365 (3) |
| C6 | 1.84988 (11) | 0.88359 (11) | 0.69272 (11) | 0.0305 (2) |
| C7 | 0.76159 (12) | 0.49385 (12) | −0.01408 (11) | 0.0332 (3) |
| C8 | 0.66587 (14) | 0.50906 (13) | −0.12028 (11) | 0.0374 (3) |
| C9 | 0.51360 (13) | 0.42605 (14) | −0.16405 (10) | 0.0367 (3) |
| C10 | 0.46425 (12) | 0.33147 (12) | −0.10088 (10) | 0.0329 (3) |

| | | | | |
|---|---|---|---|---|
| C11 | 0.58275 (11) | 0.17043 (10) | 0.15844 (10) | 0.0274 (2) |
| C12 | 0.56943 (10) | 0.32248 (10) | 0.00812 (9) | 0.0255 (2) |
| C13 | 0.78977 (14) | 0.16797 (14) | 0.31479 (14) | 0.0401 (3) |
| C14 | 0.70567 (18) | 0.05002 (16) | 0.33525 (17) | 0.0495 (5) |
| C15 | 0.55197 (17) | −0.01009 (13) | 0.26316 (16) | 0.0469 (4) |
| C16 | 0.48925 (14) | 0.04862 (12) | 0.17371 (13) | 0.0387 (3) |
| O7 | 1.14682 (14) | 0.81433 (12) | 0.14879 (11) | 0.0492 (3) |
| O8 | 0.88888 (16) | 0.80611 (12) | 0.38293 (15) | 0.0613 (4) |
| O9 | 0.80392 (11) | 0.89324 (14) | 0.01315 (13) | 0.0604 (4) |
| H2A | 1.29990 | 0.77980 | 0.43830 | 0.0390* |
| H2B | 1.33620 | 0.70600 | 0.32230 | 0.0390* |
| H3 | 0.426 (2) | 0.1928 (19) | 0.0392 (18) | 0.050 (5)* |
| H3A | 1.42590 | 0.57550 | 0.45290 | 0.0420* |
| H3B | 1.39850 | 0.66010 | 0.57000 | 0.0420* |
| H4A | 1.60690 | 0.77760 | 0.45390 | 0.0380* |
| H4B | 1.57380 | 0.86830 | 0.56270 | 0.0380* |
| H5A | 1.71530 | 0.67790 | 0.59900 | 0.0440* |
| H5B | 1.66320 | 0.74550 | 0.70680 | 0.0440* |
| H7 | 0.86380 | 0.54890 | 0.01450 | 0.0400* |
| H8 | 0.70200 | 0.57340 | −0.16210 | 0.0450* |
| H9 | 0.44560 | 0.43450 | −0.23560 | 0.0440* |
| H10 | 0.36270 | 0.27410 | −0.12970 | 0.0400* |
| H13 | 0.89300 | 0.20800 | 0.36220 | 0.0480* |
| H14 | 0.75020 | 0.01120 | 0.39570 | 0.0590* |
| H15 | 0.49160 | −0.08990 | 0.27540 | 0.0560* |
| H16 | 0.38660 | 0.00850 | 0.12400 | 0.0460* |
| H51 | 0.932 (2) | 0.2581 (19) | 0.0397 (19) | 0.046 (5)* |
| H52 | 1.002 (2) | 0.242 (2) | 0.143 (2) | 0.065 (6)* |
| H61 | 0.836 (3) | 0.526 (3) | 0.427 (2) | 0.083 (7)* |
| H62 | 0.838 (2) | 0.607 (2) | 0.3537 (19) | 0.057 (5)* |
| H71 | 1.117 (3) | 0.868 (3) | 0.168 (3) | 0.107 (10)* |
| H72 | 1.122 (2) | 0.745 (2) | 0.184 (2) | 0.063 (6)* |
| H81 | 0.958 (3) | 0.868 (3) | 0.367 (2) | 0.075 (6)* |
| H82 | 0.884 (3) | 0.849 (3) | 0.447 (3) | 0.085 (8)* |
| H91 | 0.833 (2) | 0.880 (2) | −0.044 (2) | 0.067 (6)* |
| H92 | 0.870 (3) | 0.949 (3) | 0.077 (3) | 0.085 (8)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Co1 | 0.0168 (1) | 0.0288 (1) | 0.0261 (1) | 0.0034 (1) | 0.0039 (1) | 0.0117 (1) |
| O1 | 0.0216 (3) | 0.0398 (4) | 0.0325 (4) | 0.0018 (3) | 0.0020 (3) | 0.0170 (3) |
| O2 | 0.0243 (3) | 0.0370 (4) | 0.0328 (3) | 0.0047 (3) | 0.0054 (3) | 0.0156 (3) |
| O3 | 0.0306 (4) | 0.0617 (6) | 0.0328 (4) | −0.0007 (4) | −0.0034 (3) | 0.0056 (4) |
| O4 | 0.0370 (5) | 0.0520 (6) | 0.0595 (6) | −0.0090 (4) | 0.0017 (4) | 0.0276 (5) |
| O5 | 0.0393 (5) | 0.0504 (5) | 0.0350 (4) | 0.0211 (4) | 0.0143 (4) | 0.0141 (4) |
| O6 | 0.0328 (4) | 0.0307 (4) | 0.0303 (4) | 0.0101 (3) | 0.0098 (3) | 0.0113 (3) |
| N1 | 0.0198 (3) | 0.0288 (4) | 0.0253 (3) | 0.0054 (3) | 0.0050 (3) | 0.0103 (3) |
| N2 | 0.0245 (4) | 0.0270 (4) | 0.0352 (4) | 0.0075 (3) | 0.0106 (3) | 0.0133 (3) |
| N3 | 0.0188 (4) | 0.0360 (4) | 0.0377 (4) | 0.0013 (3) | 0.0047 (3) | 0.0144 (4) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C1 | 0.0182 (4) | 0.0318 (4) | 0.0265 (4) | 0.0041 (3) | 0.0052 (3) | 0.0090 (3) |
| C2 | 0.0187 (4) | 0.0328 (5) | 0.0356 (5) | 0.0015 (3) | 0.0020 (3) | 0.0100 (4) |
| C3 | 0.0201 (4) | 0.0371 (5) | 0.0347 (5) | 0.0003 (4) | 0.0015 (4) | 0.0138 (4) |
| C4 | 0.0194 (4) | 0.0324 (5) | 0.0322 (4) | 0.0017 (3) | 0.0021 (3) | 0.0109 (4) |
| C5 | 0.0228 (4) | 0.0311 (5) | 0.0409 (5) | 0.0000 (4) | −0.0019 (4) | 0.0148 (4) |
| C6 | 0.0198 (4) | 0.0281 (4) | 0.0342 (4) | 0.0045 (3) | 0.0035 (3) | 0.0052 (4) |
| C7 | 0.0251 (4) | 0.0388 (5) | 0.0314 (4) | 0.0058 (4) | 0.0064 (4) | 0.0171 (4) |
| C8 | 0.0370 (5) | 0.0444 (6) | 0.0326 (5) | 0.0150 (5) | 0.0096 (4) | 0.0205 (4) |
| C9 | 0.0350 (5) | 0.0473 (6) | 0.0264 (4) | 0.0204 (5) | 0.0035 (4) | 0.0111 (4) |
| C10 | 0.0223 (4) | 0.0386 (5) | 0.0288 (4) | 0.0097 (4) | 0.0010 (3) | 0.0051 (4) |
| C11 | 0.0257 (4) | 0.0227 (4) | 0.0309 (4) | 0.0045 (3) | 0.0117 (3) | 0.0068 (3) |
| C12 | 0.0198 (4) | 0.0265 (4) | 0.0247 (4) | 0.0059 (3) | 0.0046 (3) | 0.0050 (3) |
| C13 | 0.0338 (5) | 0.0419 (6) | 0.0522 (7) | 0.0166 (5) | 0.0152 (5) | 0.0269 (5) |
| C14 | 0.0541 (8) | 0.0447 (7) | 0.0679 (9) | 0.0250 (6) | 0.0285 (7) | 0.0362 (7) |
| C15 | 0.0548 (8) | 0.0299 (5) | 0.0661 (9) | 0.0130 (5) | 0.0331 (7) | 0.0243 (6) |
| C16 | 0.0340 (5) | 0.0280 (5) | 0.0469 (6) | 0.0008 (4) | 0.0168 (5) | 0.0103 (4) |
| O7 | 0.0603 (7) | 0.0403 (5) | 0.0440 (5) | 0.0140 (5) | 0.0175 (5) | 0.0159 (4) |
| O8 | 0.0727 (8) | 0.0345 (5) | 0.0813 (9) | 0.0112 (5) | 0.0427 (7) | 0.0201 (5) |
| O9 | 0.0245 (4) | 0.0761 (8) | 0.0492 (6) | −0.0076 (5) | 0.0069 (4) | 0.0033 (5) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Co1—O1 | 2.0680 (10) | C2—C3 | 1.5111 (17) |
| Co1—O2 | 2.3079 (9) | C3—C4 | 1.5177 (17) |
| Co1—O5 | 2.0877 (12) | C4—C5 | 1.5106 (17) |
| Co1—O6 | 2.1336 (9) | C5—C6 | 1.5172 (17) |
| Co1—N1 | 2.0781 (9) | C7—C8 | 1.3681 (17) |
| Co1—N2 | 2.0596 (10) | C8—C9 | 1.387 (2) |
| O1—C1 | 1.2715 (13) | C9—C10 | 1.3705 (18) |
| O2—C1 | 1.2537 (14) | C10—C12 | 1.4064 (15) |
| O3—C6 | 1.2569 (15) | C11—C16 | 1.4070 (17) |
| O4—C6 | 1.2387 (17) | C13—C14 | 1.368 (2) |
| O5—H51 | 0.775 (19) | C14—C15 | 1.388 (3) |
| O5—H52 | 0.80 (2) | C15—C16 | 1.374 (2) |
| O6—H62 | 0.77 (2) | C2—H2A | 0.9700 |
| O6—H61 | 0.83 (2) | C2—H2B | 0.9700 |
| O7—H71 | 0.74 (3) | C3—H3A | 0.9700 |
| O7—H72 | 0.89 (2) | C3—H3B | 0.9700 |
| O8—H81 | 0.88 (3) | C4—H4B | 0.9700 |
| O8—H82 | 0.79 (3) | C4—H4A | 0.9700 |
| O9—H92 | 0.79 (3) | C5—H5A | 0.9700 |
| O9—H91 | 0.77 (2) | C5—H5B | 0.9700 |
| N1—C12 | 1.3350 (14) | C7—H7 | 0.9300 |
| N1—C7 | 1.3527 (15) | C8—H8 | 0.9300 |
| N2—C11 | 1.3363 (15) | C9—H9 | 0.9300 |
| N2—C13 | 1.3580 (17) | C10—H10 | 0.9300 |
| N3—C12 | 1.3815 (15) | C13—H13 | 0.9300 |
| N3—C11 | 1.3785 (15) | C14—H14 | 0.9300 |
| N3—H3 | 0.78 (2) | C15—H15 | 0.9300 |
| C1—C2 | 1.5059 (16) | C16—H16 | 0.9300 |

| | | | |
|---|---|---|---|
| O1—Co1—O2 | 59.68 (3) | C9—C10—C12 | 119.02 (11) |
| O1—Co1—O5 | 89.56 (4) | N3—C11—C16 | 116.66 (11) |
| O1—Co1—O6 | 88.57 (4) | N2—C11—N3 | 121.87 (10) |
| O1—Co1—N1 | 100.35 (4) | N2—C11—C16 | 121.46 (10) |
| O1—Co1—N2 | 169.33 (4) | N1—C12—N3 | 121.29 (9) |
| O2—Co1—O5 | 84.94 (4) | N3—C12—C10 | 116.80 (10) |
| O2—Co1—O6 | 87.44 (3) | N1—C12—C10 | 121.92 (10) |
| O2—Co1—N1 | 159.98 (4) | N2—C13—C14 | 123.44 (14) |
| O2—Co1—N2 | 109.79 (4) | C13—C14—C15 | 118.04 (15) |
| O5—Co1—O6 | 172.04 (4) | C14—C15—C16 | 119.82 (14) |
| O5—Co1—N1 | 94.09 (4) | C11—C16—C15 | 118.94 (13) |
| O5—Co1—N2 | 91.18 (4) | C1—C2—H2B | 108.00 |
| O6—Co1—N1 | 93.86 (4) | H2A—C2—H2B | 107.00 |
| O6—Co1—N2 | 89.24 (4) | C3—C2—H2A | 108.00 |
| N1—Co1—N2 | 90.21 (4) | C3—C2—H2B | 108.00 |
| Co1—O1—C1 | 95.33 (7) | C1—C2—H2A | 108.00 |
| Co1—O2—C1 | 84.88 (6) | C4—C3—H3B | 109.00 |
| Co1—O5—H51 | 122.6 (16) | H3A—C3—H3B | 108.00 |
| Co1—O5—H52 | 120.3 (15) | C4—C3—H3A | 109.00 |
| H51—O5—H52 | 104 (2) | C2—C3—H3A | 109.00 |
| Co1—O6—H61 | 116 (2) | C2—C3—H3B | 109.00 |
| Co1—O6—H62 | 112.1 (15) | C3—C4—H4A | 109.00 |
| H61—O6—H62 | 107 (3) | C3—C4—H4B | 109.00 |
| H71—O7—H72 | 112 (3) | C5—C4—H4A | 109.00 |
| H81—O8—H82 | 103 (3) | C5—C4—H4B | 109.00 |
| H91—O9—H92 | 111 (3) | H4A—C4—H4B | 108.00 |
| C7—N1—C12 | 117.69 (9) | C6—C5—H5A | 109.00 |
| Co1—N1—C12 | 125.38 (7) | H5A—C5—H5B | 108.00 |
| Co1—N1—C7 | 116.84 (8) | C4—C5—H5B | 109.00 |
| Co1—N2—C13 | 116.29 (9) | C6—C5—H5B | 109.00 |
| C11—N2—C13 | 118.28 (10) | C4—C5—H5A | 109.00 |
| Co1—N2—C11 | 125.44 (7) | N1—C7—H7 | 118.00 |
| C11—N3—C12 | 132.59 (11) | C8—C7—H7 | 118.00 |
| C12—N3—H3 | 110.7 (14) | C9—C8—H8 | 121.00 |
| C11—N3—H3 | 116.2 (14) | C7—C8—H8 | 121.00 |
| O1—C1—C2 | 116.83 (10) | C10—C9—H9 | 120.00 |
| O2—C1—C2 | 123.13 (10) | C8—C9—H9 | 120.00 |
| O1—C1—O2 | 120.04 (10) | C9—C10—H10 | 120.00 |
| C1—C2—C3 | 116.75 (10) | C12—C10—H10 | 121.00 |
| C2—C3—C4 | 111.55 (10) | N2—C13—H13 | 118.00 |
| C3—C4—C5 | 113.16 (10) | C14—C13—H13 | 118.00 |
| C4—C5—C6 | 114.59 (10) | C15—C14—H14 | 121.00 |
| O4—C6—C5 | 119.03 (11) | C13—C14—H14 | 121.00 |
| O3—C6—O4 | 124.09 (12) | C14—C15—H15 | 120.00 |
| O3—C6—C5 | 116.88 (11) | C16—C15—H15 | 120.00 |
| N1—C7—C8 | 123.70 (12) | C11—C16—H16 | 120.00 |
| C7—C8—C9 | 118.30 (12) | C15—C16—H16 | 121.00 |
| C8—C9—C10 | 119.36 (11) | | |

| | | | |
|---|---|---|---|
| O2—Co1—O1—C1 | 1.54 (6) | C12—N1—C7—C8 | 1.49 (17) |
| O5—Co1—O1—C1 | −82.85 (7) | Co1—N1—C12—N3 | −4.11 (15) |
| O6—Co1—O1—C1 | 89.41 (7) | Co1—N1—C12—C10 | 175.23 (8) |
| N1—Co1—O1—C1 | −176.92 (7) | C7—N1—C12—N3 | 179.59 (10) |
| O1—Co1—O2—C1 | −1.56 (6) | C7—N1—C12—C10 | −1.07 (16) |
| O5—Co1—O2—C1 | 90.90 (7) | Co1—N2—C11—N3 | 0.16 (15) |
| O6—Co1—O2—C1 | −91.40 (7) | Co1—N2—C11—C16 | −178.58 (9) |
| N1—Co1—O2—C1 | 2.85 (14) | C13—N2—C11—N3 | 179.90 (11) |
| N2—Co1—O2—C1 | −179.67 (7) | C13—N2—C11—C16 | 1.16 (17) |
| O1—Co1—N1—C7 | 11.20 (9) | Co1—N2—C13—C14 | 178.23 (13) |
| O1—Co1—N1—C12 | −165.14 (9) | C11—N2—C13—C14 | −1.5 (2) |
| O2—Co1—N1—C7 | 7.33 (16) | C12—N3—C11—N2 | 17.69 (19) |
| O2—Co1—N1—C12 | −169.01 (9) | C12—N3—C11—C16 | −163.51 (12) |
| O5—Co1—N1—C7 | −79.11 (9) | C11—N3—C12—N1 | −15.42 (19) |
| O5—Co1—N1—C12 | 104.56 (9) | C11—N3—C12—C10 | 165.21 (12) |
| O6—Co1—N1—C7 | 100.44 (9) | O1—C1—C2—C3 | −167.09 (10) |
| O6—Co1—N1—C12 | −75.89 (9) | O2—C1—C2—C3 | 12.39 (16) |
| N2—Co1—N1—C7 | −170.31 (9) | C1—C2—C3—C4 | 175.72 (9) |
| N2—Co1—N1—C12 | 13.36 (9) | C2—C3—C4—C5 | 175.19 (10) |
| O2—Co1—N2—C11 | 169.49 (9) | C3—C4—C5—C6 | −170.72 (10) |
| O2—Co1—N2—C13 | −10.25 (10) | C4—C5—C6—O3 | 150.64 (12) |
| O5—Co1—N2—C11 | −105.46 (10) | C4—C5—C6—O4 | −29.52 (17) |
| O5—Co1—N2—C13 | 74.80 (10) | N1—C7—C8—C9 | −0.72 (19) |
| O6—Co1—N2—C11 | 82.49 (9) | C7—C8—C9—C10 | −0.50 (19) |
| O6—Co1—N2—C13 | −97.25 (9) | C8—C9—C10—C12 | 0.87 (18) |
| N1—Co1—N2—C11 | −11.37 (9) | C9—C10—C12—N1 | −0.08 (16) |
| N1—Co1—N2—C13 | 168.89 (9) | C9—C10—C12—N3 | 179.29 (11) |
| Co1—O1—C1—O2 | −2.83 (11) | N2—C11—C16—C15 | 0.00 (19) |
| Co1—O1—C1—C2 | 176.67 (8) | N3—C11—C16—C15 | −178.80 (13) |
| Co1—O2—C1—O1 | 2.54 (10) | N2—C13—C14—C15 | 0.7 (2) |
| Co1—O2—C1—C2 | −176.93 (10) | C13—C14—C15—C16 | 0.5 (2) |
| Co1—N1—C7—C8 | −175.13 (10) | C14—C15—C16—C11 | −0.9 (2) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N3—H3···O9$^{i}$ | 0.78 (2) | 2.05 (2) | 2.8228 (17) | 172 (2) |
| O5—H51···O7$^{ii}$ | 0.775 (19) | 1.923 (19) | 2.6933 (15) | 172.7 (19) |
| O5—H52···O3$^{iii}$ | 0.80 (2) | 1.97 (2) | 2.7706 (17) | 173 (2) |
| O6—H61···O2$^{iv}$ | 0.83 (2) | 2.00 (2) | 2.8278 (12) | 178 (4) |
| O6—H62···O8 | 0.77 (2) | 1.94 (2) | 2.7056 (16) | 170 (2) |
| O7—H71···O3$^{v}$ | 0.74 (3) | 2.56 (3) | 3.2596 (18) | 160 (3) |
| O7—H72···O1 | 0.89 (2) | 1.86 (2) | 2.7543 (16) | 175 (2) |
| O8—H81···O4$^{v}$ | 0.88 (3) | 1.97 (3) | 2.8221 (19) | 163 (2) |
| O8—H82···O4$^{vi}$ | 0.79 (3) | 2.05 (3) | 2.832 (2) | 175 (3) |

| | | | | |
|---|---|---|---|---|
| O9—H91···O3$^{vii}$ | 0.77 (2) | 2.12 (2) | 2.8670 (17) | 164 (2) |
| O9—H92···O3$^{v}$ | 0.79 (3) | 1.99 (3) | 2.7452 (17) | 160 (3) |

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*; (ii) −*x*+2, −*y*+1, −*z*; (iii) −*x*+3, −*y*+1, −*z*+1; (iv) −*x*+2, −*y*+1, −*z*+1; (v) −*x*+3, −*y*+2, −*z*+1; (vi) *x*−1, *y*, *z*; (vii) *x*−1, *y*, *z*−1.