# Bis(2,4,6-triamino-1,3,5-triazin-1-ium) hexaaquacobalt(II) bis[bis(pyridine-2,6-dicarboxylato)cobaltate(II)] tetrahydrate

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The title compound, $(C_3H_7N_6)_2[Co(H_2O)_6][Co(C_7H_3NO_4)_2]_2 \cdot 4H_2O$, or $(tataH)_2[Co(H_2O)_6][Co(pydc)_2]_2 \cdot 4H_2O$ (where tata is 2,4,6-triamino-1,3,5-triazine and pydc is pyridine-2,6-dicarboxylic acid), was obtained by reaction of $Co(NO_3)_2 \cdot 6H_2O$ with the proton-transfer compound $(tataH)_2(pydc)$ in aqueous solution. The $[Co(pydc)_2]^{2-}$ anion is a six-coordinate $Co^{II}$ complex with a distorted octahedral coordination geometry. The structure also contains hexaaquacobalt(II) cations (site symmetry $\bar{1}$), $(tataH)^+$ cations and uncoordinated water molecules. The two $(pydc)^{2-}$ ligands in each $[Co(pydc)_2]^{2-}$ anion are almost perpendicular to each other [dihedral angle between their mean planes = 82.3 (1)°]. There is extensive O—H⋯O, N—H⋯N, O—H⋯N and C—H⋯O hydrogen bonding in the structure, as well as $\pi$–$\pi$ stacking between $(pydc)^{2-}$ ligands with an interplanar distance of 3.484 (15) Å.

## Related literature

For related literature, see: Aghabozorg, Attar Gharamaleki *et al.* (2007); Aghabozorg, Daneshvar *et al.* (2007); Sheshmani *et al.* (2006).
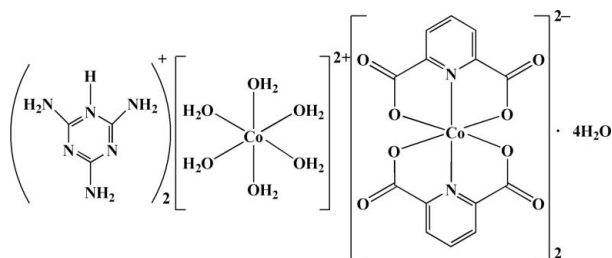


## Experimental

### Crystal data

| | |
|---|---|
| $(C_3H_7N_6)_2[Co(H_2O)_6]$- $[Co(C_7H_3NO_4)_2]_2 \cdot 4H_2O$ | $\beta$ = 106.017 (5)° |
| $M_r$ = 1271.66 | $\gamma$ = 107.133 (5)° |
| Triclinic, $P\bar{1}$ | $V$ = 1185.73 (14) Å³ |
| $a$ = 8.4003 (6) Å | $Z$ = 1 |
| $b$ = 11.3014 (7) Å | Mo $K\alpha$ radiation |
| $c$ = 13.8794 (10) Å | $\mu$ = 1.15 mm⁻¹ |
| $\alpha$ = 95.901 (6)° | $T$ = 120 (2) K |
| | 0.50 × 0.50 × 0.45 mm |

### Data collection

| | |
|---|---|
| Stoe IPDSII diffractometer | 14236 measured reflections |
| Absorption correction: numerical ($X$-$SHAPE$; Stoe & Cie, 2004) | 6289 independent reflections |
| $T_{min}$ = 0.570, $T_{max}$ = 0.595 | 6111 reflections with $I > 2\sigma(I)$ |
| | $R_{int}$ = 0.021 |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)]$ = 0.027 | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2)$ = 0.071 | |
| $S$ = 1.06 | |
| 6289 reflections | $\Delta\rho_{max}$ = 0.48 e Å⁻³ |
| 426 parameters | $\Delta\rho_{min}$ = −0.90 e Å⁻³ |

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H⋯$A$ | $D$—H | H⋯$A$ | $D$⋯$A$ | $D$—H⋯$A$ |
|---|---|---|---|---|
| N3—H3$B$⋯O13ⁱ | 0.83 (2) | 1.97 (2) | 2.7982 (18) | 178 (3) |
| N3—H3$C$⋯O3 | 0.84 (2) | 2.21 (2) | 3.0041 (17) | 159 (2) |
| N5—H5$B$⋯O3ⁱⁱ | 0.83 (2) | 2.35 (2) | 2.9713 (16) | 133 (2) |
| N5—H5$C$⋯N4ⁱⁱ | 0.82 (2) | 2.16 (2) | 2.9765 (17) | 178 (2) |
| N7—H7$A$⋯O10ⁱⁱ | 0.82 (2) | 2.22 (2) | 3.0291 (16) | 169 (2) |
| N7—H7$B$⋯O7ⁱ | 0.80 (2) | 2.26 (2) | 3.0560 (17) | 172 (2) |
| N8—H8⋯O8ⁱ | 0.85 (2) | 1.89 (2) | 2.7404 (17) | 174 (2) |
| O9—H9$A$⋯N6ⁱⁱ | 0.79 (3) | 1.94 (3) | 2.7298 (16) | 179 (4) |
| O9—H9$B$⋯O2ⁱⁱⁱ | 0.87 (3) | 1.89 (3) | 2.7263 (16) | 162 (3) |
| O10—H10$B$⋯O12 | 0.81 (2) | 1.88 (3) | 2.6759 (17) | 167 (3) |
| O10—H10$C$⋯O4ⁱᵛ | 0.81 (2) | 1.97 (2) | 2.7675 (15) | 171 (3) |
| O11—H11$B$⋯O5 | 0.80 (3) | 1.97 (2) | 2.7313 (14) | 157 (2) |
| O11—H11$C$⋯O2ᵛ | 0.83 (2) | 1.90 (2) | 2.7101 (16) | 165 (2) |
| O12—H12$B$⋯O4ᵛⁱ | 0.79 (3) | 2.06 (3) | 2.8389 (17) | 174 (2) |
| O12—H12$C$⋯O6 | 0.84 (3) | 1.98 (3) | 2.8196 (16) | 171 (2) |
| O13—H13$A$⋯O1ᵛⁱⁱ | 0.77 (3) | 2.01 (3) | 2.7642 (16) | 171 (3) |
| O13—H13$B$⋯O8 | 0.82 (3) | 1.87 (3) | 2.6642 (14) | 163 (2) |
| C4—H4⋯O13ᵛⁱⁱⁱ | 0.93 | 2.43 | 3.164 (2) | 136 |
| C10—H10$A$⋯O6ⁱˣ | 0.93 | 2.45 | 3.344 (2) | 162 |

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

Data collection: *X-AREA* (Stoe & Cie, 2005); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BI2263).

# metal-organic compounds

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# supporting information

## Bis(2,4,6-triamino-1,3,5-triazin-1-ium) hexaaquacobalt(II) bis[bis(pyridine-2,6-dicarboxylato)cobaltate(II)] tetrahydrate

**Hossein Aghabozorg, Jafar Attar Gharamaleki, Shirin Daneshvar, Mohammad Ghadermazi and Hamid Reza Khavasi**

### S1. Comment

Recently, we have reported the reactions between $Co(NO_3)_2.6H_2O$ and two proton-transfer compounds: $(GH)_2(pydc)$ (where G is guanidine) and $(pipzH_2)(pydc)$ (where pipz is piperazine) in a 1:2 molar ratio. These reactions lead to the formation of the complexes $(GH)_2[Co(H_2O)_6][Co(pydc)_2]_2$ (Sheshmani *et al.*, 2006) and $(pipzH_2)[Co(H_2O)_6]$ $[Co(pydc)_2]_2.8H_2O$ (Aghabozorg, Attar Gharamaleki *et al.*, 2007), respectively.

Here, we report the synthesis and X-ray crystal structure of the title compound (Fig. 1). The compound contains $[Co(pydc)_2]^{2-}$ anions, $[Co(H_2O)_6]^{2+}$ cations (site symmetry $\bar{1}$), and $(tataH)^+$ cations. In the $[Co(pydc)_2]^{2-}$ anions, the $Co^{II}$ atom is hexacoordinated by two N atoms (N1 and N2) and four O atoms (O1, O3, O5 and O7) from the carboxylate groups of two $(pydc)^{2-}$ groups that act as tridentate ligands. The coordination geometry is distorted octahedral, with atoms N1 and N2 occupying axial positions and the O atoms forming the equatorial plane. The N1—Co2—N2 angle deviates *ca* 7.7° from linearity. The mean Co—N and Co—O bond lengths for Co1 are 2.0214 (11) and 2.1658 (10) Å, respectively, consistent with similar complexes in the literature. The dihedral angle between the mean planes of the two $(pydc)^{2-}$ groups is 82.3 (1)°.

There is extensive O—H⋯O, N—H⋯O, N—H⋯N and C—H⋯O hydrogen bonding in the structure, as well as $\pi$–$\pi$ stacking between $(pydc)^{2-}$ ligands with an interplanar distance of 3.484 (15) Å (symmetry operator: -*x*, -*y* + 1, -*z* + 1). Atom O8 of the C14?O8 carboxyl group lies above the N2/C9–C13 ring with an O⋯centroid distance of 3.240 (1) Å (symmetry operator: *x*, -*y* + 1, -*z*).

### S2. Experimental

The proton-transfer compound, $(tataH)_2(pydc)$, was prepared by the reaction of pyridine-2,6-dicarboxylic acid $(pydcH_2)$ with 2,4,6-triamino-1,3,5-triazine (tata). The reaction between $Co(NO_3)_2.6H_2O$ (115 mg, 0.5 mmol) in water (20 ml) and $(tataH)_2(pydc)$ (420 mg, 1.0 mmol) in water (20 ml), in a 1:2 molar ratio gave a violet crystalline compound after slow evaporation of the solvent at room temperature.

### S3. Refinement

The N-bound and O-bound H atoms were located in difference Fourier maps and their positions were freely refined. Other H atoms were placed in calculated positions (C—H = 0.93 Å) and refined as riding with $U_{iso}(H) = 1.2U_{eq}(C)$.
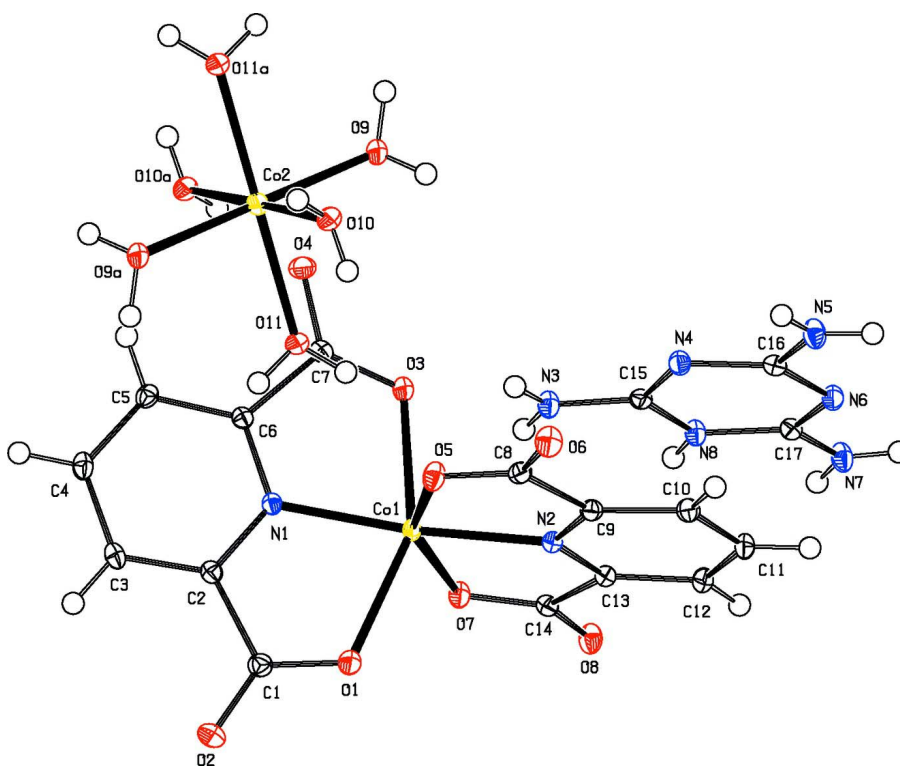
**Figure 1**

The molecular structure with displacement ellipsoids drawn at the 50% probability level for non-H atoms. Water molecules are omitted. The suffix a denotes atoms generated by the symmetry operator -*x*, -*y*, -*z* + 1.
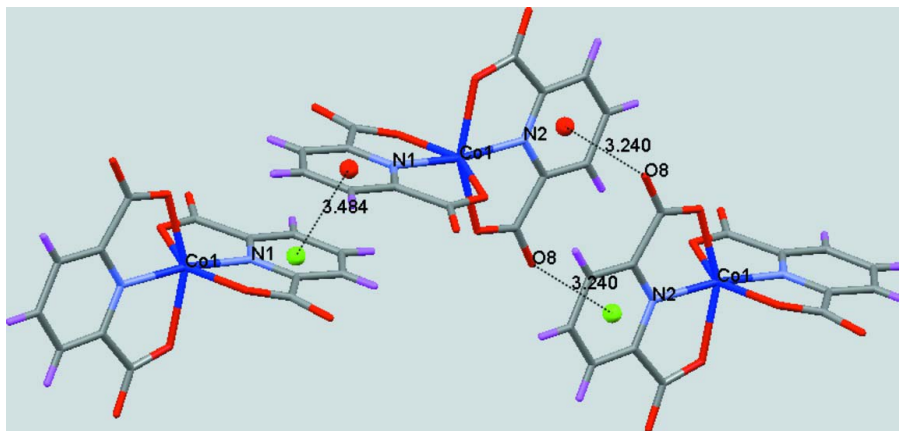


**Figure 2**

$\pi\cdots\pi$ Stacking interaction between two aromatic rings of (pydc)$^{2-}$ units, with interplanar distance of 3.484 (15) Å [-*x*, -*y* + 1, -*z* + 1]; C—O$\cdots\pi$ stacking interactions between CO groups of carboxylate fragments with aromatic rings of (pydc)$^{2-}$ with distances of 3.240 (1) Å for C14–O8$\cdots$*Cg*1 (*x*, -*y* + 1, -*z*) [*Cg*1 is the centroid for N2/C9–C13 ring].
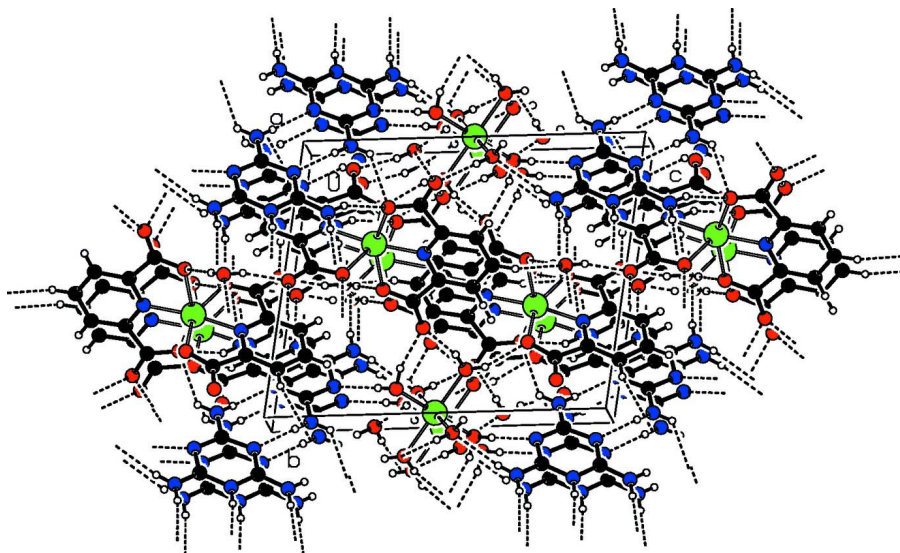
**Figure 3**

Unit cell packing. Hydrogen bonds are shown as dashed lines.

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*Crystal data*

| | |
|---|---|
| $(C_3H_7N_6)_2[Co(H_2O)_6][Co(C_7H_3NO_4)_2]_2 \cdot 4H_2O$ | $Z = 1$ |
| $M_r = 1271.66$ | $F(000) = 651$ |
| Triclinic, $P\bar{1}$ | $D_x = 1.781$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $a = 8.4003$ (6) Å | Cell parameters from 2500 reflections |
| $b = 11.3014$ (7) Å | $\theta = 1.9$–29.2° |
| $c = 13.8794$ (10) Å | $\mu = 1.15$ mm$^{-1}$ |
| $\alpha = 95.901$ (6)° | $T = 120$ K |
| $\beta = 106.017$ (5)° | Block, violet |
| $\gamma = 107.133$ (5)° | $0.50 \times 0.50 \times 0.45$ mm |
| $V = 1185.73$ (14) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Stoe IPDSII diffractometer | 14236 measured reflections |
| Radiation source: fine-focus sealed tube | 6289 independent reflections |
| Graphite monochromator | 6111 reflections with $I > 2\sigma(I)$ |
| $\varphi$ scans | $R_{int} = 0.021$ |
| Absorption correction: numerical (*X-SHAPE*; Stoe & Cie, 2004) | $\theta_{max} = 29.2°$, $\theta_{min} = 1.9°$ |
| $T_{min} = 0.570$, $T_{max} = 0.595$ | $h = -11 \rightarrow 11$ |
| | $k = -15 \rightarrow 15$ |
| | $l = -18 \rightarrow 18$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | 426 parameters |
| Least-squares matrix: full | 0 restraints |
| $R[F^2 > 2\sigma(F^2)] = 0.027$ | Primary atom site location: structure-invariant direct methods |
| $wR(F^2) = 0.071$ | |
| $S = 1.06$ | Secondary atom site location: difference Fourier map |
| 6289 reflections | |

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0323P)^2 + 1.0178P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.019$
$\Delta\rho_{max} = 0.48$ e Å$^{-3}$
$\Delta\rho_{min} = -0.90$ 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 (Å²)*

|      | *x*          | *y*          | *z*           | $U_{iso}*/U_{eq}$ |
|------|--------------|--------------|---------------|-------------------|
| C1   | 0.09807 (17) | 0.59767 (12) | 0.39201 (10)  | 0.0120 (2)        |
| C2   | 0.19794 (16) | 0.52881 (12) | 0.46046 (9)   | 0.0100 (2)        |
| C3   | 0.26752 (17) | 0.56041 (12) | 0.56675 (10)  | 0.0120 (2)        |
| H3A  | 0.2520       | 0.6273       | 0.6035        | 0.014*            |
| C4   | 0.36138 (17) | 0.48851 (12) | 0.61643 (10)  | 0.0123 (2)        |
| H4   | 0.4087       | 0.5068       | 0.6874        | 0.015*            |
| C5   | 0.38413 (17) | 0.38926 (12) | 0.55950 (10)  | 0.0110 (2)        |
| H5A  | 0.4481       | 0.3416       | 0.5917        | 0.013*            |
| C6   | 0.30917 (16) | 0.36313 (11) | 0.45369 (9)   | 0.0092 (2)        |
| C7   | 0.32416 (16) | 0.26121 (11) | 0.37973 (10)  | 0.0099 (2)        |
| C8   | −0.23329 (17)| 0.17773 (12) | 0.16471 (10)  | 0.0110 (2)        |
| C9   | −0.17660 (16)| 0.21879 (12) | 0.07477 (10)  | 0.0102 (2)        |
| C10  | −0.26571 (17)| 0.16132 (12) | −0.02722 (10) | 0.0118 (2)        |
| H10A | −0.3694      | 0.0926       | −0.0460       | 0.014*            |
| C11  | −0.19517 (17)| 0.20971 (12) | −0.10097 (10) | 0.0125 (2)        |
| H11A | −0.2524      | 0.1736       | −0.1700       | 0.015*            |
| C12  | −0.03839 (17)| 0.31260 (12) | −0.07062 (10) | 0.0118 (2)        |
| H12A | 0.0096       | 0.3464       | −0.1188       | 0.014*            |
| C13  | 0.04411 (16) | 0.36325 (11) | 0.03343 (9)   | 0.0096 (2)        |
| C14  | 0.21779 (16) | 0.47113 (12) | 0.08186 (10)  | 0.0102 (2)        |
| C15  | 0.34004 (16) | 0.22483 (12) | 0.01662 (10)  | 0.0102 (2)        |
| C16  | 0.12106 (17) | 0.07415 (12) | −0.11206 (10) | 0.0112 (2)        |
| C17  | 0.33515 (17) | 0.21334 (12) | −0.15514 (10) | 0.0111 (2)        |
| N1   | 0.21838 (14) | 0.43202 (10) | 0.40734 (8)   | 0.00895 (19)      |
| N2   | −0.02564 (14)| 0.31650 (10) | 0.10290 (8)   | 0.00952 (19)      |
| N3   | 0.41635 (16) | 0.28384 (11) | 0.11324 (9)   | 0.0126 (2)        |
| H3B  | 0.504 (3)    | 0.349 (2)    | 0.1304 (17)   | 0.027 (5)*        |
| H3C  | 0.370 (3)    | 0.258 (2)    | 0.1568 (17)   | 0.021 (5)*        |
| N4   | 0.19481 (15) | 0.12353 (10) | −0.01054 (8)  | 0.0114 (2)        |
| N5   | −0.02803 (16)| −0.02294 (11)| −0.14102 (9)  | 0.0145 (2)        |

| | | | | |
|------|------------|------------|-------------|-------------|
| H5B | −0.079 (3) | −0.057 (2) | −0.2023 (19) | 0.029 (6)* |
| H5C | −0.073 (3) | −0.049 (2) | −0.0984 (18) | 0.025 (5)* |
| N6 | 0.18535 (15) | 0.11571 (11) | −0.18643 (8) | 0.0118 (2) |
| N7 | 0.41327 (17) | 0.25597 (11) | −0.22155 (9) | 0.0142 (2) |
| H7A | 0.369 (3) | 0.218 (2) | −0.2810 (17) | 0.022 (5)* |
| H7B | 0.497 (3) | 0.320 (2) | −0.2040 (17) | 0.022 (5)* |
| N8 | 0.41245 (15) | 0.27160 (11) | −0.05467 (8) | 0.0113 (2) |
| H8 | 0.504 (3) | 0.337 (2) | −0.0403 (19) | 0.034 (6)* |
| O1 | 0.04288 (13) | 0.54960 (9) | 0.29686 (7) | 0.01419 (18) |
| O2 | 0.07887 (16) | 0.69381 (10) | 0.43161 (8) | 0.0199 (2) |
| O3 | 0.26322 (13) | 0.26350 (9) | 0.28504 (7) | 0.01189 (17) |
| O4 | 0.39502 (13) | 0.18586 (9) | 0.41537 (8) | 0.01399 (18) |
| O5 | −0.12884 (13) | 0.24112 (9) | 0.25325 (7) | 0.01391 (18) |
| O6 | −0.37131 (13) | 0.08811 (10) | 0.14719 (8) | 0.01572 (19) |
| O7 | 0.27238 (12) | 0.50218 (9) | 0.17825 (7) | 0.01198 (17) |
| O8 | 0.29441 (13) | 0.52186 (9) | 0.02292 (7) | 0.01449 (18) |
| O9 | 0.04230 (14) | −0.08822 (10) | 0.37722 (8) | 0.01480 (19) |
| H9A | −0.024 (4) | −0.097 (3) | 0.322 (2) | 0.043 (7)* |
| H9B | 0.053 (3) | −0.162 (3) | 0.381 (2) | 0.043 (7)* |
| O10 | −0.27658 (13) | −0.08879 (9) | 0.43229 (8) | 0.01282 (18) |
| H10B | −0.331 (3) | −0.047 (2) | 0.404 (2) | 0.035 (6)* |
| H10C | −0.321 (3) | −0.116 (2) | 0.4736 (19) | 0.032 (6)* |
| O11 | −0.02330 (13) | 0.15266 (9) | 0.42733 (8) | 0.01306 (18) |
| H11B | −0.071 (3) | 0.158 (2) | 0.370 (2) | 0.033 (6)* |
| H11C | −0.044 (3) | 0.207 (2) | 0.4624 (18) | 0.028 (6)* |
| O12 | −0.47942 (15) | 0.01920 (11) | 0.31460 (9) | 0.0196 (2) |
| H12B | −0.512 (3) | 0.069 (2) | 0.340 (2) | 0.036 (6)* |
| H12C | −0.443 (3) | 0.048 (2) | 0.268 (2) | 0.033 (6)* |
| O13 | 0.29453 (14) | 0.49240 (11) | −0.16999 (8) | 0.0180 (2) |
| H13A | 0.205 (4) | 0.480 (2) | −0.210 (2) | 0.039 (7)* |
| H13B | 0.277 (3) | 0.488 (2) | −0.115 (2) | 0.037 (6)* |
| Co1 | 0.10510 (2) | 0.385862 (16) | 0.253625 (12) | 0.00886 (5) |
| Co2 | 0.0000 | 0.0000 | 0.5000 | 0.01165 (6) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|-----|-----------|-----------|-----------|-----------|-----------|-----------|
| C1 | 0.0127 (5) | 0.0121 (5) | 0.0124 (6) | 0.0050 (4) | 0.0048 (4) | 0.0029 (4) |
| C2 | 0.0104 (5) | 0.0095 (5) | 0.0101 (5) | 0.0027 (4) | 0.0042 (4) | 0.0016 (4) |
| C3 | 0.0132 (5) | 0.0119 (5) | 0.0100 (5) | 0.0032 (4) | 0.0047 (4) | −0.0005 (4) |
| C4 | 0.0133 (5) | 0.0131 (5) | 0.0086 (5) | 0.0016 (4) | 0.0039 (4) | 0.0011 (4) |
| C5 | 0.0112 (5) | 0.0108 (5) | 0.0101 (5) | 0.0022 (4) | 0.0032 (4) | 0.0037 (4) |
| C6 | 0.0094 (5) | 0.0083 (5) | 0.0096 (5) | 0.0019 (4) | 0.0039 (4) | 0.0018 (4) |
| C7 | 0.0100 (5) | 0.0087 (5) | 0.0111 (5) | 0.0019 (4) | 0.0049 (4) | 0.0017 (4) |
| C8 | 0.0118 (5) | 0.0109 (5) | 0.0111 (5) | 0.0045 (4) | 0.0037 (4) | 0.0034 (4) |
| C9 | 0.0107 (5) | 0.0094 (5) | 0.0108 (5) | 0.0036 (4) | 0.0034 (4) | 0.0023 (4) |
| C10 | 0.0110 (5) | 0.0107 (5) | 0.0120 (6) | 0.0029 (4) | 0.0019 (4) | 0.0015 (4) |
| C11 | 0.0137 (6) | 0.0137 (6) | 0.0082 (5) | 0.0046 (5) | 0.0012 (4) | 0.0001 (4) |

| | | | | | |
|---|---|---|---|---|---|
| C12 | 0.0133 (6) | 0.0131 (6) | 0.0089 (5) | 0.0044 (5) | 0.0035 (4) | 0.0022 (4) |
| C13 | 0.0104 (5) | 0.0092 (5) | 0.0092 (5) | 0.0034 (4) | 0.0029 (4) | 0.0018 (4) |
| C14 | 0.0108 (5) | 0.0087 (5) | 0.0108 (5) | 0.0032 (4) | 0.0036 (4) | 0.0015 (4) |
| C15 | 0.0116 (5) | 0.0102 (5) | 0.0104 (5) | 0.0055 (4) | 0.0040 (4) | 0.0024 (4) |
| C16 | 0.0129 (5) | 0.0092 (5) | 0.0118 (6) | 0.0045 (4) | 0.0036 (4) | 0.0020 (4) |
| C17 | 0.0132 (5) | 0.0107 (5) | 0.0103 (5) | 0.0054 (4) | 0.0039 (4) | 0.0018 (4) |
| N1 | 0.0094 (4) | 0.0086 (4) | 0.0083 (4) | 0.0019 (4) | 0.0032 (4) | 0.0015 (4) |
| N2 | 0.0105 (5) | 0.0094 (4) | 0.0084 (4) | 0.0034 (4) | 0.0026 (4) | 0.0017 (4) |
| N3 | 0.0137 (5) | 0.0129 (5) | 0.0095 (5) | 0.0021 (4) | 0.0039 (4) | 0.0012 (4) |
| N4 | 0.0123 (5) | 0.0107 (5) | 0.0098 (5) | 0.0024 (4) | 0.0032 (4) | 0.0015 (4) |
| N5 | 0.0147 (5) | 0.0131 (5) | 0.0115 (5) | −0.0002 (4) | 0.0039 (4) | 0.0000 (4) |
| N6 | 0.0131 (5) | 0.0113 (5) | 0.0094 (5) | 0.0025 (4) | 0.0032 (4) | 0.0014 (4) |
| N7 | 0.0169 (5) | 0.0126 (5) | 0.0103 (5) | 0.0003 (4) | 0.0059 (4) | 0.0005 (4) |
| N8 | 0.0115 (5) | 0.0113 (5) | 0.0092 (5) | 0.0013 (4) | 0.0038 (4) | 0.0008 (4) |
| O1 | 0.0176 (5) | 0.0155 (4) | 0.0107 (4) | 0.0089 (4) | 0.0033 (4) | 0.0020 (3) |
| O2 | 0.0300 (6) | 0.0169 (5) | 0.0167 (5) | 0.0149 (4) | 0.0067 (4) | 0.0016 (4) |
| O3 | 0.0142 (4) | 0.0128 (4) | 0.0088 (4) | 0.0053 (3) | 0.0035 (3) | 0.0011 (3) |
| O4 | 0.0184 (5) | 0.0137 (4) | 0.0146 (4) | 0.0089 (4) | 0.0078 (4) | 0.0057 (3) |
| O5 | 0.0141 (4) | 0.0150 (4) | 0.0096 (4) | 0.0009 (4) | 0.0034 (3) | 0.0028 (3) |
| O6 | 0.0131 (4) | 0.0149 (4) | 0.0155 (4) | −0.0006 (4) | 0.0049 (4) | 0.0029 (4) |
| O7 | 0.0126 (4) | 0.0122 (4) | 0.0092 (4) | 0.0018 (3) | 0.0033 (3) | 0.0015 (3) |
| O8 | 0.0157 (4) | 0.0147 (4) | 0.0110 (4) | 0.0005 (4) | 0.0061 (4) | 0.0026 (3) |
| O9 | 0.0201 (5) | 0.0161 (5) | 0.0092 (4) | 0.0093 (4) | 0.0034 (4) | 0.0011 (4) |
| O10 | 0.0156 (4) | 0.0126 (4) | 0.0115 (4) | 0.0053 (4) | 0.0050 (4) | 0.0044 (3) |
| O11 | 0.0183 (5) | 0.0114 (4) | 0.0108 (4) | 0.0066 (4) | 0.0045 (4) | 0.0036 (3) |
| O12 | 0.0271 (6) | 0.0227 (5) | 0.0190 (5) | 0.0157 (5) | 0.0131 (4) | 0.0095 (4) |
| O13 | 0.0139 (5) | 0.0264 (5) | 0.0110 (4) | 0.0030 (4) | 0.0041 (4) | 0.0031 (4) |
| Co1 | 0.01010 (9) | 0.00890 (8) | 0.00667 (8) | 0.00262 (6) | 0.00218 (6) | 0.00084 (6) |
| Co2 | 0.01542 (12) | 0.01014 (11) | 0.00968 (11) | 0.00476 (9) | 0.00401 (9) | 0.00194 (8) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—O2 | 1.2401 (16) | C16—N4 | 1.3541 (16) |
| C1—O1 | 1.2729 (16) | C16—N6 | 1.3548 (16) |
| C1—C2 | 1.5195 (17) | C17—N7 | 1.3232 (17) |
| C2—N1 | 1.3358 (16) | C17—N6 | 1.3310 (17) |
| C2—C3 | 1.3922 (17) | C17—N8 | 1.3636 (16) |
| C3—C4 | 1.3965 (18) | N1—Co1 | 2.0218 (11) |
| C3—H3A | 0.930 | N2—Co1 | 2.0211 (11) |
| C4—C5 | 1.3952 (18) | N3—H3B | 0.83 (2) |
| C4—H4 | 0.930 | N3—H3C | 0.84 (2) |
| C5—C6 | 1.3898 (17) | N5—H5B | 0.83 (2) |
| C5—H5A | 0.930 | N5—H5C | 0.82 (2) |
| C6—N1 | 1.3356 (16) | N7—H7A | 0.82 (2) |
| C6—C7 | 1.5199 (17) | N7—H7B | 0.81 (2) |
| C7—O4 | 1.2438 (16) | N8—H8 | 0.86 (3) |
| C7—O3 | 1.2781 (15) | O1—Co1 | 2.1383 (10) |
| C8—O6 | 1.2373 (16) | O3—Co1 | 2.1802 (10) |

| | | | |
|---|---|---|---|
| C8—O5 | 1.2833 (16) | O5—Co1 | 2.1532 (10) |
| C8—C9 | 1.5223 (17) | O7—Co1 | 2.1916 (9) |
| C9—N2 | 1.3391 (16) | O9—Co2 | 2.0538 (10) |
| C9—C10 | 1.3877 (17) | O9—H9A | 0.79 (3) |
| C10—C11 | 1.3989 (18) | O9—H9B | 0.87 (3) |
| C10—H10A | 0.930 | O10—Co2 | 2.1250 (10) |
| C11—C12 | 1.3982 (18) | O10—H10B | 0.81 (3) |
| C11—H11A | 0.930 | O10—H10C | 0.81 (3) |
| C12—C13 | 1.3920 (17) | O11—Co2 | 2.1166 (10) |
| C12—H12A | 0.930 | O11—H11B | 0.81 (3) |
| C13—N2 | 1.3364 (16) | O11—H11C | 0.83 (3) |
| C13—C14 | 1.5196 (17) | O12—H12B | 0.79 (3) |
| C14—O8 | 1.2576 (15) | O12—H12C | 0.85 (3) |
| C14—O7 | 1.2624 (15) | O13—H13A | 0.77 (3) |
| C15—N3 | 1.3205 (17) | O13—H13B | 0.83 (3) |
| C15—N4 | 1.3324 (16) | Co2—O9$^{i}$ | 2.0538 (10) |
| C15—N8 | 1.3706 (16) | Co2—O11$^{i}$ | 2.1166 (10) |
| C16—N5 | 1.3251 (17) | Co2—O10$^{i}$ | 2.1250 (10) |
| | | | |
| O2—C1—O1 | 126.05 (12) | C15—N3—H3C | 118.9 (14) |
| O2—C1—C2 | 118.92 (12) | H3B—N3—H3C | 120 (2) |
| O1—C1—C2 | 115.03 (11) | C15—N4—C16 | 115.97 (11) |
| N1—C2—C3 | 121.29 (12) | C16—N5—H5B | 121.4 (16) |
| N1—C2—C1 | 112.54 (11) | C16—N5—H5C | 120.0 (16) |
| C3—C2—C1 | 126.15 (11) | H5B—N5—H5C | 119 (2) |
| C2—C3—C4 | 117.93 (12) | C17—N6—C16 | 115.98 (11) |
| C2—C3—H3A | 121.0 | C17—N7—H7A | 117.6 (15) |
| C4—C3—H3A | 121.0 | C17—N7—H7B | 120.4 (15) |
| C5—C4—C3 | 119.94 (12) | H7A—N7—H7B | 122 (2) |
| C5—C4—H4 | 120.0 | C17—N8—C15 | 119.54 (11) |
| C3—C4—H4 | 120.0 | C17—N8—H8 | 116.7 (16) |
| C6—C5—C4 | 118.55 (12) | C15—N8—H8 | 123.8 (16) |
| C6—C5—H5A | 120.7 | C1—O1—Co1 | 116.52 (8) |
| C4—C5—H5A | 120.7 | C7—O3—Co1 | 114.43 (8) |
| N1—C6—C5 | 120.80 (11) | C8—O5—Co1 | 115.52 (8) |
| N1—C6—C7 | 113.45 (11) | C14—O7—Co1 | 114.48 (8) |
| C5—C6—C7 | 125.72 (11) | Co2—O9—H9A | 119 (2) |
| O4—C7—O3 | 126.28 (12) | Co2—O9—H9B | 116.3 (18) |
| O4—C7—C6 | 118.49 (11) | H9A—O9—H9B | 105 (3) |
| O3—C7—C6 | 115.22 (11) | Co2—O10—H10B | 117.2 (18) |
| O6—C8—O5 | 126.18 (12) | Co2—O10—H10C | 111.2 (17) |
| O6—C8—C9 | 118.67 (11) | H10B—O10—H10C | 107 (2) |
| O5—C8—C9 | 115.14 (11) | Co2—O11—H11B | 134.2 (18) |
| N2—C9—C10 | 121.47 (12) | Co2—O11—H11C | 113.0 (16) |
| N2—C9—C8 | 113.40 (11) | H11B—O11—H11C | 102 (2) |
| C10—C9—C8 | 125.11 (11) | H12B—O12—H12C | 108 (2) |
| C9—C10—C11 | 118.14 (12) | H13A—O13—H13B | 107 (3) |
| C9—C10—H10A | 120.9 | N2—Co1—N1 | 172.24 (4) |

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|---|---|---|---|
| C11—C10—H10A | 120.9 | N2—Co1—O1 | 107.89 (4) |
| C12—C11—C10 | 119.79 (12) | N1—Co1—O1 | 76.33 (4) |
| C12—C11—H11A | 120.1 | N2—Co1—O5 | 76.99 (4) |
| C10—C11—H11A | 120.1 | N1—Co1—O5 | 96.02 (4) |
| C13—C12—C11 | 118.36 (12) | O1—Co1—O5 | 99.47 (4) |
| C13—C12—H12A | 120.8 | N2—Co1—O3 | 100.34 (4) |
| C11—C12—H12A | 120.8 | N1—Co1—O3 | 76.35 (4) |
| N2—C13—C12 | 121.12 (11) | O1—Co1—O3 | 151.15 (4) |
| N2—C13—C14 | 112.50 (11) | O5—Co1—O3 | 92.44 (4) |
| C12—C13—C14 | 126.37 (11) | N2—Co1—O7 | 76.19 (4) |
| O8—C14—O7 | 125.66 (12) | N1—Co1—O7 | 110.86 (4) |
| O8—C14—C13 | 117.54 (11) | O1—Co1—O7 | 87.05 (4) |
| O7—C14—C13 | 116.80 (11) | O5—Co1—O7 | 153.11 (4) |
| N3—C15—N4 | 120.51 (12) | O3—Co1—O7 | 94.11 (4) |
| N3—C15—N8 | 118.32 (12) | O9—Co2—O9<sup>i</sup> | 180.000 (1) |
| N4—C15—N8 | 121.16 (11) | O9—Co2—O11<sup>i</sup> | 88.64 (4) |
| N5—C16—N4 | 116.86 (12) | O9<sup>i</sup>—Co2—O11<sup>i</sup> | 91.36 (4) |
| N5—C16—N6 | 117.28 (12) | O9—Co2—O11 | 91.36 (4) |
| N4—C16—N6 | 125.86 (12) | O9<sup>i</sup>—Co2—O11 | 88.64 (4) |
| N7—C17—N6 | 120.34 (12) | O11<sup>i</sup>—Co2—O11 | 180.0 |
| N7—C17—N8 | 118.30 (12) | O9—Co2—O10<sup>i</sup> | 89.04 (4) |
| N6—C17—N8 | 121.36 (12) | O9<sup>i</sup>—Co2—O10<sup>i</sup> | 90.96 (4) |
| C6—N1—C2 | 121.47 (11) | O11<sup>i</sup>—Co2—O10<sup>i</sup> | 87.97 (4) |
| C6—N1—Co1 | 119.06 (8) | O11—Co2—O10<sup>i</sup> | 92.03 (4) |
| C2—N1—Co1 | 119.45 (9) | O9—Co2—O10 | 90.96 (4) |
| C13—N2—C9 | 121.11 (11) | O9<sup>i</sup>—Co2—O10 | 89.04 (4) |
| C13—N2—Co1 | 119.93 (9) | O11<sup>i</sup>—Co2—O10 | 92.03 (4) |
| C9—N2—Co1 | 118.84 (9) | O11—Co2—O10 | 87.97 (4) |
| C15—N3—H3B | 121.1 (16) | O10<sup>i</sup>—Co2—O10 | 180.00 (6) |
| | | | |
| O2—C1—C2—N1 | −175.99 (12) | N5—C16—N6—C17 | 179.97 (12) |
| O1—C1—C2—N1 | 3.28 (16) | N4—C16—N6—C17 | 0.49 (19) |
| O2—C1—C2—C3 | 2.4 (2) | N7—C17—N8—C15 | −176.18 (12) |
| O1—C1—C2—C3 | −178.33 (12) | N6—C17—N8—C15 | 3.55 (19) |
| N1—C2—C3—C4 | 0.67 (19) | N3—C15—N8—C17 | −179.25 (12) |
| C1—C2—C3—C4 | −177.60 (12) | N4—C15—N8—C17 | −0.25 (19) |
| C2—C3—C4—C5 | 0.53 (19) | O2—C1—O1—Co1 | 178.25 (11) |
| C3—C4—C5—C6 | −1.09 (18) | C2—C1—O1—Co1 | −0.95 (14) |
| C4—C5—C6—N1 | 0.48 (18) | O4—C7—O3—Co1 | 168.69 (10) |
| C4—C5—C6—C7 | 178.42 (11) | C6—C7—O3—Co1 | −12.44 (13) |
| N1—C6—C7—O4 | −174.80 (11) | O6—C8—O5—Co1 | 177.49 (11) |
| C5—C6—C7—O4 | 7.12 (19) | C9—C8—O5—Co1 | −1.93 (14) |
| N1—C6—C7—O3 | 6.23 (15) | O8—C14—O7—Co1 | −177.89 (10) |
| C5—C6—C7—O3 | −171.84 (12) | C13—C14—O7—Co1 | 2.28 (14) |
| O6—C8—C9—N2 | 179.96 (12) | C13—N2—Co1—O1 | 84.95 (10) |
| O5—C8—C9—N2 | −0.58 (16) | C9—N2—Co1—O1 | −99.00 (10) |
| O6—C8—C9—C10 | −1.74 (19) | C13—N2—Co1—O5 | −179.15 (10) |
| O5—C8—C9—C10 | 177.73 (12) | C9—N2—Co1—O5 | −3.10 (9) |

| | | | |
|---|---|---|---|
| N2—C9—C10—C11 | −1.12 (19) | C13—N2—Co1—O3 | −89.02 (10) |
| C8—C9—C10—C11 | −179.30 (12) | C9—N2—Co1—O3 | 87.03 (10) |
| C9—C10—C11—C12 | 0.45 (19) | C13—N2—Co1—O7 | 2.71 (9) |
| C10—C11—C12—C13 | 0.63 (19) | C9—N2—Co1—O7 | 178.76 (10) |
| C11—C12—C13—N2 | −1.12 (19) | C6—N1—Co1—O1 | −178.41 (10) |
| C11—C12—C13—C14 | 177.53 (12) | C2—N1—Co1—O1 | 2.95 (9) |
| N2—C13—C14—O8 | −179.98 (11) | C6—N1—Co1—O5 | 83.28 (9) |
| C12—C13—C14—O8 | 1.28 (19) | C2—N1—Co1—O5 | −95.36 (9) |
| N2—C13—C14—O7 | −0.13 (16) | C6—N1—Co1—O3 | −7.78 (9) |
| C12—C13—C14—O7 | −178.88 (12) | C2—N1—Co1—O3 | 173.58 (10) |
| C5—C6—N1—C2 | 0.73 (18) | C6—N1—Co1—O7 | −97.00 (9) |
| C7—C6—N1—C2 | −177.45 (11) | C2—N1—Co1—O7 | 84.36 (10) |
| C5—C6—N1—Co1 | −177.89 (9) | C1—O1—Co1—N2 | 172.32 (9) |
| C7—C6—N1—Co1 | 3.94 (14) | C1—O1—Co1—N1 | −0.91 (9) |
| C3—C2—N1—C6 | −1.32 (18) | C1—O1—Co1—O5 | 93.04 (10) |
| C1—C2—N1—C6 | 177.17 (11) | C1—O1—Co1—O3 | −20.05 (14) |
| C3—C2—N1—Co1 | 177.29 (9) | C1—O1—Co1—O7 | −113.22 (10) |
| C1—C2—N1—Co1 | −4.23 (14) | C8—O5—Co1—N2 | 2.70 (9) |
| C12—C13—N2—C9 | 0.48 (19) | C8—O5—Co1—N1 | −173.89 (9) |
| C14—C13—N2—C9 | −178.34 (11) | C8—O5—Co1—O1 | 109.02 (9) |
| C12—C13—N2—Co1 | 176.44 (9) | C8—O5—Co1—O3 | −97.36 (9) |
| C14—C13—N2—Co1 | −2.38 (14) | C8—O5—Co1—O7 | 6.70 (15) |
| C10—C9—N2—C13 | 0.67 (19) | C7—O3—Co1—N2 | −161.61 (9) |
| C8—C9—N2—C13 | 179.04 (11) | C7—O3—Co1—N1 | 11.20 (9) |
| C10—C9—N2—Co1 | −175.33 (9) | C7—O3—Co1—O1 | 30.34 (13) |
| C8—C9—N2—Co1 | 3.04 (14) | C7—O3—Co1—O5 | −84.40 (9) |
| N3—C15—N4—C16 | 176.38 (12) | C7—O3—Co1—O7 | 121.69 (9) |
| N8—C15—N4—C16 | −2.61 (18) | C14—O7—Co1—N2 | −2.67 (9) |
| N5—C16—N4—C15 | −176.90 (12) | C14—O7—Co1—N1 | 173.94 (9) |
| N6—C16—N4—C15 | 2.58 (19) | C14—O7—Co1—O1 | −111.90 (9) |
| N7—C17—N6—C16 | 176.15 (12) | C14—O7—Co1—O5 | −6.68 (14) |
| N8—C17—N6—C16 | −3.57 (18) | C14—O7—Co1—O3 | 96.99 (9) |

Symmetry code: (i) −x, −y, −z+1.

*Hydrogen-bond geometry (Å, °)*

| D—H···A | D—H | H···A | D···A | D—H···A |
|---|---|---|---|---|
| N3—H3*B*···O13ii | 0.83 (2) | 1.97 (2) | 2.7982 (18) | 178 (3) |
| N3—H3*C*···O3 | 0.84 (2) | 2.21 (2) | 3.0041 (17) | 159 (2) |
| N5—H5*B*···O3iii | 0.83 (2) | 2.35 (2) | 2.9713 (16) | 133 (2) |
| N5—H5*C*···N4iii | 0.82 (2) | 2.16 (2) | 2.9765 (17) | 178 (3) |
| N7—H7*A*···O10iii | 0.82 (2) | 2.22 (2) | 3.0291 (16) | 169 (2) |
| N7—H7*B*···O7ii | 0.80 (2) | 2.26 (2) | 3.0560 (17) | 172 (2) |
| N8—H8···O8ii | 0.85 (2) | 1.89 (2) | 2.7404 (17) | 174 (2) |
| O9—H9*A*···N6iii | 0.79 (3) | 1.94 (3) | 2.7298 (16) | 179 (4) |
| O9—H9*B*···O2iv | 0.87 (3) | 1.89 (3) | 2.7263 (16) | 162 (3) |
| O10—H10*B*···O12 | 0.81 (2) | 1.88 (3) | 2.6759 (17) | 167 (3) |

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|---|---|---|---|---|
| O10—H10$C$···O4[i] | 0.81 (2) | 1.97 (2) | 2.7675 (15) | 171 (3) |
| O11—H11$B$···O5 | 0.80 (3) | 1.97 (2) | 2.7313 (14) | 157 (2) |
| O11—H11$C$···O2[v] | 0.83 (2) | 1.90 (2) | 2.7101 (16) | 165 (2) |
| O12—H12$B$···O4[vi] | 0.79 (3) | 2.06 (3) | 2.8389 (17) | 174 (2) |
| O12—H12$C$···O6 | 0.84 (3) | 1.98 (3) | 2.8196 (16) | 171 (2) |
| O13—H13$A$···O1[vii] | 0.77 (3) | 2.01 (3) | 2.7642 (16) | 171 (3) |
| O13—H13$B$···O8 | 0.82 (3) | 1.87 (3) | 2.6642 (14) | 163 (2) |
| C4—H4···O13[viii] | 0.93 | 2.43 | 3.164 (2) | 136 |
| C10—H10$A$···O6[ix] | 0.93 | 2.45 | 3.344 (2) | 162 |

Symmetry codes: (i) −$x$, −$y$, −$z$+1; (ii) −$x$+1, −$y$+1, −$z$; (iii) −$x$, −$y$, −$z$; (iv) $x$, $y$−1, $z$; (v) −$x$, −$y$+1, −$z$+1; (vi) $x$−1, $y$, $z$; (vii) −$x$, −$y$+1, −$z$; (viii) $x$, $y$, $z$+1; (ix) −$x$−1, −$y$, −$z$.