

(*N*-*tert*-Butylpyrazine-2-carboxamide- κ N⁴)chloridobis(dimethylglyoximate- κ^2 N,N')cobalt(III) hemihydrate

 S. Martin,^a C. Revathi,^a Babu Varghese^b and A. Dayalan^{a*}
^aDepartment of Chemistry, Loyola College (Autonomous), Chennai 600 034, Tamil Nadu, India, and ^bSophisticated Analytical Instruments Facility, Indian Institute of Technology Madras, Chennai 600 036, India

Correspondence e-mail: dayalansgp@gmail.com

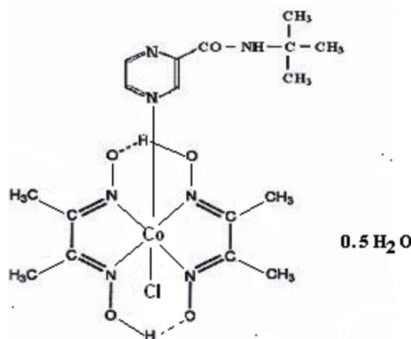
Received 17 September 2007; accepted 29 September 2007

 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; H-atom completeness 97%; disorder in solvent or counterion; R factor = 0.039; wR factor = 0.121; data-to-parameter ratio = 14.0.

The title compound, $[\text{Co}(\text{C}_4\text{H}_7\text{N}_2\text{O}_2)_2\text{Cl}(\text{C}_9\text{H}_{13}\text{N}_3\text{O})] \cdot 0.5\text{H}_2\text{O}$, *viz.* *trans*- $[\text{Co}(\text{dmgH})_2\text{Cl}(\textit{t}\text{-BuPzAM})] \cdot 0.5\text{H}_2\text{O}$, has two molecules in the asymmetric unit with some differences in the conformation and positions of the H atoms with regard to the hydroxy groups of the glyoximate groups. The water molecule is disordered equally over two sites.

Related literature

For related literature, see: Dayalan & Vijayaraghavan (2001); Gupta *et al.* (2000, 2001, 2004); Ohkubo & Fukuzumi (2005); Phor *et al.* (1985); Razavelt *et al.* (2005); Trommel *et al.* (2001); Vijayaraghavan & Dayalan (1992).



Experimental

Crystal data

 $[\text{Co}(\text{C}_4\text{H}_7\text{N}_2\text{O}_2)_2\text{Cl}(\text{C}_9\text{H}_{13}\text{N}_3\text{O})] \cdot 0.5\text{H}_2\text{O}$
 $M_r = 521.84$

 Monoclinic, $P2_1/c$
 $a = 11.4320$ (2) Å

 $b = 15.8134$ (4) Å

 $c = 26.2781$ (6) Å

 $\beta = 90.06$ (2) $^\circ$
 $V = 4750.52$ (18) Å³
 $Z = 8$

 Mo $K\alpha$ radiation

 $\mu = 0.88$ mm⁻¹
 $T = 293$ (2) K

 $0.30 \times 0.30 \times 0.20$ mm

Data collection

Bruker Kappa-APEXII CCD diffractometer

Absorption correction: multi-scan (SADABS; Bruker, 1999)

 $T_{\min} = 0.779$, $T_{\max} = 0.844$

92600 measured reflections

8366 independent reflections

 7136 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.028$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.121$
 $S = 1.17$

8366 reflections

599 parameters

2 restraints

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.62$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.48$ e Å⁻³
Table 1

 Hydrogen-bond geometry (Å, $^\circ$).

| $D-H \cdots A$ | $D-H$ | $H \cdots A$ | $D \cdots A$ | $D-H \cdots A$ |
|--------------------|-------|--------------|--------------|----------------|
| O1—H1 \cdots O3 | 0.82 | 1.69 | 2.476 (4) | 161 |
| O2—H2 \cdots O4 | 0.82 | 1.69 | 2.483 (4) | 162 |
| O6—H6 \cdots O8 | 0.82 | 1.72 | 2.507 (4) | 161 |
| O9—H9A \cdots O7 | 0.82 | 1.68 | 2.483 (4) | 165 |

Data collection: APEX2 (Bruker–Nonius, 2004); cell refinement: APEX2 and SAINT (Bruker–Nonius, 2004); data reduction: SAINT and XPREP (Bruker–Nonius, 2004); program(s) used to solve structure: SIR92 (Altomare *et al.*, 1993); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-32 (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

The authors are thankful to Rev. Fr. A. Albert Muthumali, S. J., Principal, Loyola College (Autonomous), Chennai, India, for providing the necessary facilities, the Head, SAIF, CDRI, Lucknow, India, for supplying elemental data and the Head, SAIF, IIT Madras, Chennai, India, for recording NMR spectra and for the X-ray data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2106).

References

- Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. (1993). *J. Appl. Cryst.* **26**, 343–350.
- Bruker (1999). SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker–Nonius (2004). APEXII and SAINT and XPREP. Bruker AXS Inc., Madison, Wisconsin, USA.
- Dayalan, A. & Vijayaraghavan, V. R. (2001). *Indian J. Chem. Sect. A*, **40**, 959–964.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Gupta, B. D., Singh, V., Quanango, K., Vijay Kanth, V., Yamuna, R., Barclay, T. & Cordes, W. (2000). *J. Organomet. Chem.* **602**, 1–4.
- Gupta, B. D., Tiwari, U., Barley, T. & Cordes, W. (2001). *J. Organomet. Chem.* **629**, 83–92.
- Gupta, B. D., Vijayaikanth, V. & Sing, V. (2004). *Organometallics*, **23**, 2067–2079.
- Ohkubo, K. & Fukuzumi, S. (2005). *J. Phys. Chem.* **109**, 1105–1113.
- Phor, N. B., Forcolin, M., Toscano, P. J., Summers, M. F., Randaccio, L. & Marzilli, L. G. (1985). *Coord. Chem. Rev.* **63**, 1–125.
- Razavelt, M., Artero, V. & Fentcave, M. (2005). *Inorg. Chem.* **44**, 4786–4795.
- Sheldrick, G. M. (1997). SHELXL97. University of Göttingen, Germany.
- Trommel, J. S., Warncke, K. & Marzilli, L. G. (2001). *J. Am. Chem. Soc.* **123**, 3358–3366.
- Vijayaraghavan, V. R. & Dayalan, A. (1992). *J. Indian Chem. Soc.* **69**, 383–384.

supplementary materials

Acta Cryst. (2007). E63, m2670 [doi:10.1107/S1600536807047903]

(*N*-*tert*-Butylpyrazine-2-carboxamide- κ N⁴)chloridobis(dimethylglyoximato- κ ²N,N')cobalt(III) hemihydrate

S. Martin, C. Revathi, B. Varghese and A. Dayalan

Comment

Dimethylglyoximato cobalt(III) complexes, generally known as cobaloximes, have been studied extensively as model compounds for vitamin-B₁₂ (Trommel, *et al.*, 2001; Ohkubo & Fukuzumi, 2005). Most of the work on cobaloximes include electron-transfer reactions (Dayalan & Vijayaraghavan, 2001) and catalytic activity (Razavelt, *et al.*, 2005) in solution. There are few literature evidences (Gupta, *et al.*, 2000; Gupta, *et al.*, 2001; Gupta, *et al.*, 2004) relating the structural aspects of cobaloximes. We report here the synthesis, physical characterization and the crystal structure of a typical cobaloxime *viz.*, pyrazine-2-*tert*-butylcarboxamidochlorocobaloxime. Figure 1 shows the *ORTEP* representation of the two independent molecules in the asymmetric unit, with 50% anisotropic ellipsoids. Figure 2 presents a packing diagram of the structure depicting the hydrogen bonding interactions. The geometry around the metal atom is octahedral; the bonds to chlorines and pyrazine N atoms are almost perpendicular to the mean glyoximate planes. The difference Fourier clearly shows one O—H group in each one of the glyoximate moieties attached to Co2, while in those attached to Co1, both hydrogen atoms are bound to only one of the glyoximate moieties. The planar glyoximate groups subtend dihedral angles of 7.76 (13)° (Co1) and 0.95 (13)° (Co2). There are strong intra molecular O—H \cdots O hydrogen bonds linking the glyoximate moieties (Figure 2). There is one crystal water molecule per asymmetric unit (disordered into two positions O11, O11') hydrogen bonded to the chlorine atoms Cl2, attached to Co2. The corresponding water H atoms could not be located in the difference map and were not included in the model.

Experimental

The green colored Trans-Hydrogen dichlorobis(dimethylglyoximato) cobaltate(III), H[Co(dmgh)₂Cl₂] was prepared by the method reported in the literature (Phor, *et al.*, 1985). Using this complex, as the starting material, the cobaloxime: pyrazine-2-*tert*-butylcarboxamidopyrazinechlorocobaloxime *viz.*, [Co(dmgh)₂(t-BuPzAM)Cl] was prepared by adopting the method reported earlier (Vijayaraghavan & Dayalan, 1992). Hydrogen dichlorobis (dimethylglyoximato)cobaltate(III) (0.01 mole) and 0.01 mole of Pyrazine-2-*tert*-butylcarboxamide were taken in about 60 ml of absolute alcohol and stirred for 1 hr, with warming over hot water bath, until the green colour due to dichlorocobaloxime was discharged to get the required brown colored complex. The resulting complex was filtered, washed with alcohol and dried in a vacuum desiccator. Crystals of the complex were grown in ethanol by slow evaporation.

Refinement

Even though the unit cell satisfies orthorhombic metric conditions within the limits of s.u., R_{int} pointed clearly to a monoclinic symmetry ($R_{\text{int}}^{\text{ortho}}=0.59$, $R_{\text{int}}^{\text{mono}}=0.027$). The structure was solved and refined in $P2_1/c$. All the H atoms were located in a difference Fourier map. H atoms linked to carbon and oxygen atoms were relocated at idealized positions and allowed to ride with C—H(aromatic): 0.93Å%, and 1.2 U_{eq} C; C—H(tertiary): 0.96Å%, and 1.5 U_{eq} C; O—H: 0.82Å%, and

supplementary materials

1.5 U_{eq} O. N—H hydrogen distances were restrained (N—H:0.83 (1)) and refined isotropically. The disordered crystal water molecule is split into two equally populated sites (O11 and O11', with 50% occupancy each).

Figures

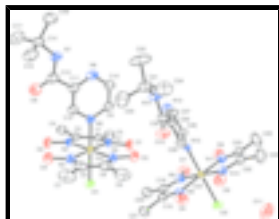


Fig. 1. The *ORTEP* representation of the asymmetric unit with 35% probability anisotropic ellipsoid. Hydrogen atoms and disordered crystal water molecule omitted, for clarity.

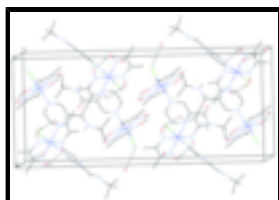


Fig. 2. Packing diagram of the molecules in the unit cell. Hydrogen bonds shown in dotted lines.

(*N*-*tert*-Butylpyrazine-2-carboxamide- κN^4)chloridobis(dimethylglyoximate- $\kappa^2 N, N'$)cobalt(III) hemihydrate

Crystal data

[Co(C₄H₇N₂O₂)₂Cl(C₉H₁₃N₃O)]·0.5H₂O
 $M_r = 521.84$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 11.4320$ (2) Å

$b = 15.8134$ (4) Å

$c = 26.2781$ (6) Å

$\beta = 90.06$ (2)°

$V = 4750.52$ (18) Å³

$Z = 8$

$F_{000} = 2136$

$D_x = 1.434$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 5042 reflections

$\theta = 2.2$ – 24.0 °

$\mu = 0.88$ mm⁻¹

$T = 293$ (2) K

Plate, brown

$0.30 \times 0.30 \times 0.20$ mm

Data collection

Bruker Kappa-APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293$ (2) K

ω and φ scans

Absorption correction: multi-scan
(SADABS; Bruker, 1999)

$T_{\min} = 0.779$, $T_{\max} = 0.844$

92600 measured reflections

8366 independent reflections

7136 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.028$

$\theta_{\text{max}} = 25.0$ °

$\theta_{\text{min}} = 1.5$ °

$h = -13 \rightarrow 13$

$k = -18 \rightarrow 18$

$l = -31 \rightarrow 31$

Refinement

| | |
|--|--|
| Refinement on F^2 | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.039$ | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2) = 0.121$ | $w = 1/[\sigma^2(F_o^2) + (0.0498P)^2 + 4.9702P]$ |
| $S = 1.17$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 8366 reflections | $(\Delta/\sigma)_{\max} < 0.001$ |
| 599 parameters | $\Delta\rho_{\max} = 0.62 \text{ e } \text{\AA}^{-3}$ |
| 2 restraints | $\Delta\rho_{\min} = -0.48 \text{ e } \text{\AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Extinction correction: none |

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 | 0.19069 (3) | 0.40860 (3) | 0.881531 (15) | 0.04025 (12) | |
| Co2 | 0.68130 (3) | 0.16125 (2) | 0.932065 (14) | 0.03599 (12) | |
| Cl1 | 0.29360 (7) | 0.49466 (5) | 0.93160 (3) | 0.0526 (2) | |
| Cl2 | 0.84549 (7) | 0.18912 (6) | 0.97384 (4) | 0.0594 (2) | |
| O1 | 0.2782 (2) | 0.27258 (15) | 0.94263 (10) | 0.0616 (6) | |
| H1 | 0.3243 | 0.2754 | 0.9188 | 0.092* | |
| O2 | -0.0168 (2) | 0.50395 (15) | 0.89776 (9) | 0.0575 (6) | |
| H2 | 0.0129 | 0.5249 | 0.8723 | 0.086* | |
| O3 | 0.3940 (2) | 0.31032 (17) | 0.86636 (11) | 0.0678 (7) | |
| O4 | 0.1109 (2) | 0.55231 (16) | 0.82713 (10) | 0.0636 (7) | |
| O5 | -0.1258 (3) | 0.4237 (2) | 0.72781 (11) | 0.0833 (9) | |
| O6 | 0.7355 (2) | 0.29998 (16) | 0.86563 (10) | 0.0632 (7) | |
| H6 | 0.7663 | 0.2588 | 0.8522 | 0.095* | |
| O7 | 0.5523 (2) | 0.16829 (17) | 1.02569 (8) | 0.0594 (6) | |
| O8 | 0.8124 (2) | 0.15686 (18) | 0.84180 (10) | 0.0649 (7) | |
| O9 | 0.6362 (2) | 0.02904 (16) | 1.00172 (9) | 0.0615 (6) | |

supplementary materials

| | | | | | |
|------|--------------|--------------|--------------|-------------|------|
| H9A | 0.6060 | 0.0710 | 1.0145 | 0.092* | |
| O10 | 0.4877 (2) | 0.20804 (16) | 0.74627 (8) | 0.0578 (6) | |
| O11 | 0.0668 (6) | 0.0909 (6) | 0.9244 (4) | 0.131 (3) | 0.50 |
| O11' | 0.0274 (10) | -0.0106 (12) | 0.9790 (4) | 0.248 (9) | 0.50 |
| N1 | 0.1903 (2) | 0.32829 (16) | 0.93528 (10) | 0.0439 (6) | |
| N2 | 0.0507 (2) | 0.44081 (16) | 0.91462 (10) | 0.0422 (6) | |
| N3 | 0.1944 (3) | 0.49340 (18) | 0.83087 (10) | 0.0513 (7) | |
| N4 | 0.3315 (2) | 0.37604 (19) | 0.84952 (11) | 0.0532 (7) | |
| N5 | 0.1012 (2) | 0.33275 (16) | 0.83651 (9) | 0.0419 (6) | |
| N6 | -0.0132 (2) | 0.22650 (19) | 0.76889 (10) | 0.0529 (7) | |
| N7 | -0.1513 (3) | 0.2913 (2) | 0.69531 (11) | 0.0591 (8) | |
| N8 | 0.5929 (2) | 0.21253 (17) | 0.98524 (9) | 0.0436 (6) | |
| N9 | 0.6790 (2) | 0.27532 (16) | 0.90817 (10) | 0.0436 (6) | |
| N10 | 0.7705 (2) | 0.11158 (18) | 0.87964 (10) | 0.0476 (6) | |
| N11 | 0.6847 (2) | 0.04996 (16) | 0.95777 (10) | 0.0455 (6) | |
| N12 | 0.53778 (19) | 0.13526 (14) | 0.89466 (8) | 0.0318 (5) | |
| N13 | 0.3360 (2) | 0.09520 (17) | 0.84150 (10) | 0.0446 (6) | |
| N14 | 0.3224 (3) | 0.1300 (2) | 0.74051 (10) | 0.0519 (7) | |
| C1 | 0.1072 (3) | 0.3342 (2) | 0.96801 (12) | 0.0441 (7) | |
| C2 | 0.0209 (3) | 0.3986 (2) | 0.95452 (12) | 0.0438 (7) | |
| C3 | 0.1040 (4) | 0.2855 (3) | 1.01639 (15) | 0.0698 (11) | |
| H3A | 0.1820 | 0.2693 | 1.0257 | 0.105* | |
| H3B | 0.0569 | 0.2358 | 1.0119 | 0.105* | |
| H3C | 0.0709 | 0.3200 | 1.0428 | 0.105* | |
| C4 | -0.0885 (3) | 0.4132 (2) | 0.98398 (15) | 0.0599 (9) | |
| H4A | -0.1238 | 0.4654 | 0.9732 | 0.090* | |
| H4B | -0.0703 | 0.4164 | 1.0196 | 0.090* | |
| H4C | -0.1419 | 0.3674 | 0.9781 | 0.090* | |
| C5 | 0.2836 (4) | 0.4923 (2) | 0.80097 (13) | 0.0609 (10) | |
| C6 | 0.3647 (3) | 0.4230 (3) | 0.81171 (15) | 0.0628 (10) | |
| C7 | 0.3023 (5) | 0.5578 (3) | 0.76089 (17) | 0.0937 (16) | |
| H7A | 0.2283 | 0.5815 | 0.7511 | 0.141* | |
| H7B | 0.3386 | 0.5323 | 0.7318 | 0.141* | |
| H7C | 0.3519 | 0.6017 | 0.7740 | 0.141* | |
| C8 | 0.4763 (4) | 0.4079 (3) | 0.7832 (2) | 0.0947 (17) | |
| H8A | 0.5262 | 0.3714 | 0.8028 | 0.142* | |
| H8B | 0.5151 | 0.4608 | 0.7774 | 0.142* | |
| H8C | 0.4589 | 0.3817 | 0.7511 | 0.142* | |
| C9 | 0.1165 (3) | 0.2493 (2) | 0.83801 (12) | 0.0484 (7) | |
| H9 | 0.1671 | 0.2261 | 0.8619 | 0.058* | |
| C10 | 0.0583 (3) | 0.1969 (2) | 0.80457 (13) | 0.0526 (8) | |
| H10 | 0.0694 | 0.1388 | 0.8071 | 0.063* | |
| C11 | -0.0292 (3) | 0.3095 (2) | 0.76843 (12) | 0.0476 (7) | |
| C12 | 0.0257 (3) | 0.3632 (2) | 0.80229 (12) | 0.0488 (8) | |
| H12 | 0.0101 | 0.4209 | 0.8013 | 0.059* | |
| C13 | -0.1088 (3) | 0.3476 (3) | 0.72809 (13) | 0.0569 (9) | |
| C14 | -0.2259 (3) | 0.3111 (2) | 0.65050 (13) | 0.0567 (9) | |
| C15 | -0.1623 (4) | 0.3724 (3) | 0.61602 (15) | 0.0743 (12) | |
| H15A | -0.1484 | 0.4243 | 0.6339 | 0.111* | |

| | | | | |
|------|-------------|--------------|--------------|-------------|
| H15B | -0.2092 | 0.3834 | 0.5864 | 0.111* |
| H15C | -0.0889 | 0.3482 | 0.6058 | 0.111* |
| C16 | -0.2478 (5) | 0.2279 (3) | 0.62361 (19) | 0.0955 (17) |
| H16A | -0.1743 | 0.2019 | 0.6153 | 0.143* |
| H16B | -0.2913 | 0.2379 | 0.5929 | 0.143* |
| H16C | -0.2916 | 0.1910 | 0.6455 | 0.143* |
| C17 | -0.3399 (4) | 0.3488 (3) | 0.66804 (18) | 0.0834 (13) |
| H17A | -0.3253 | 0.4018 | 0.6847 | 0.125* |
| H17B | -0.3772 | 0.3108 | 0.6914 | 0.125* |
| H17C | -0.3901 | 0.3578 | 0.6392 | 0.125* |
| C18 | 0.5706 (3) | 0.2921 (2) | 0.98100 (12) | 0.0470 (7) |
| C19 | 0.6235 (3) | 0.32953 (19) | 0.93559 (13) | 0.0474 (8) |
| C20 | 0.5008 (4) | 0.3412 (3) | 1.01852 (15) | 0.0691 (11) |
| H20A | 0.4574 | 0.3030 | 1.0397 | 0.104* |
| H20B | 0.4476 | 0.3779 | 1.0008 | 0.104* |
| H20C | 0.5524 | 0.3745 | 1.0393 | 0.104* |
| C21 | 0.6167 (4) | 0.4217 (2) | 0.92368 (18) | 0.0730 (12) |
| H21A | 0.6423 | 0.4537 | 0.9527 | 0.110* |
| H21B | 0.5374 | 0.4365 | 0.9156 | 0.110* |
| H21C | 0.6661 | 0.4341 | 0.8951 | 0.110* |
| C22 | 0.7898 (3) | 0.0309 (2) | 0.88436 (15) | 0.0578 (9) |
| C23 | 0.7377 (3) | -0.0052 (2) | 0.92970 (15) | 0.0570 (9) |
| C24 | 0.8640 (4) | -0.0181 (3) | 0.84738 (19) | 0.0903 (15) |
| H24A | 0.8934 | 0.0195 | 0.8217 | 0.136* |
| H24B | 0.8176 | -0.0615 | 0.8317 | 0.136* |
| H24C | 0.9284 | -0.0436 | 0.8652 | 0.136* |
| C25 | 0.7458 (5) | -0.0962 (3) | 0.9446 (2) | 0.0982 (17) |
| H25A | 0.7551 | -0.1005 | 0.9808 | 0.147* |
| H25B | 0.8119 | -0.1216 | 0.9280 | 0.147* |
| H25C | 0.6757 | -0.1251 | 0.9344 | 0.147* |
| C26 | 0.4487 (3) | 0.09584 (19) | 0.91725 (11) | 0.0417 (7) |
| H26 | 0.4541 | 0.0815 | 0.9515 | 0.050* |
| C27 | 0.3490 (3) | 0.0762 (2) | 0.89058 (12) | 0.0487 (8) |
| H27 | 0.2885 | 0.0485 | 0.9074 | 0.058* |
| C28 | 0.4248 (2) | 0.13517 (18) | 0.81967 (10) | 0.0353 (6) |
| C29 | 0.5259 (2) | 0.15585 (18) | 0.84576 (10) | 0.0361 (6) |
| H29 | 0.5859 | 0.1843 | 0.8291 | 0.043* |
| C30 | 0.4141 (3) | 0.1617 (2) | 0.76477 (11) | 0.0416 (7) |
| C31 | 0.2904 (3) | 0.1486 (3) | 0.68706 (12) | 0.0613 (10) |
| C32 | 0.1821 (5) | 0.0962 (4) | 0.67658 (17) | 0.115 (2) |
| H32A | 0.2000 | 0.0374 | 0.6810 | 0.173* |
| H32B | 0.1563 | 0.1059 | 0.6423 | 0.173* |
| H32C | 0.1212 | 0.1122 | 0.6998 | 0.173* |
| C33 | 0.2628 (5) | 0.2417 (3) | 0.68204 (19) | 0.0954 (16) |
| H33A | 0.3333 | 0.2740 | 0.6864 | 0.143* |
| H33B | 0.2072 | 0.2577 | 0.7076 | 0.143* |
| H33C | 0.2306 | 0.2526 | 0.6489 | 0.143* |
| C34 | 0.3904 (5) | 0.1230 (3) | 0.65208 (15) | 0.0874 (14) |
| H34A | 0.4561 | 0.1596 | 0.6578 | 0.131* |

supplementary materials

| | | | | |
|------|------------|-------------|-------------|-------------|
| H34B | 0.3656 | 0.1275 | 0.6173 | 0.131* |
| H34C | 0.4127 | 0.0656 | 0.6591 | 0.131* |
| H7 | -0.133 (3) | 0.2419 (10) | 0.6996 (15) | 0.064 (13)* |
| H14 | 0.278 (3) | 0.101 (2) | 0.7576 (13) | 0.061 (12)* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|------|-------------|-------------|-------------|---------------|---------------|---------------|
| Co1 | 0.0387 (2) | 0.0422 (2) | 0.0398 (2) | 0.00500 (17) | -0.00959 (16) | -0.00484 (17) |
| Co2 | 0.0374 (2) | 0.0339 (2) | 0.0367 (2) | -0.00246 (16) | -0.00596 (16) | 0.00130 (16) |
| Cl1 | 0.0478 (4) | 0.0535 (5) | 0.0566 (5) | 0.0013 (3) | -0.0129 (3) | -0.0159 (4) |
| Cl2 | 0.0508 (5) | 0.0577 (5) | 0.0696 (6) | -0.0058 (4) | -0.0235 (4) | -0.0023 (4) |
| O1 | 0.0552 (14) | 0.0551 (14) | 0.0744 (16) | 0.0222 (11) | -0.0217 (12) | -0.0014 (12) |
| O2 | 0.0526 (13) | 0.0568 (14) | 0.0630 (14) | 0.0199 (11) | -0.0130 (11) | 0.0055 (12) |
| O3 | 0.0477 (13) | 0.0603 (16) | 0.095 (2) | 0.0139 (12) | -0.0015 (13) | -0.0215 (14) |
| O4 | 0.0710 (16) | 0.0537 (14) | 0.0661 (16) | 0.0037 (13) | -0.0198 (13) | 0.0123 (12) |
| O5 | 0.111 (2) | 0.070 (2) | 0.0687 (18) | 0.0054 (17) | -0.0454 (17) | -0.0072 (15) |
| O6 | 0.0688 (16) | 0.0582 (15) | 0.0627 (15) | -0.0142 (12) | 0.0061 (12) | 0.0176 (12) |
| O7 | 0.0679 (15) | 0.0744 (17) | 0.0360 (12) | -0.0099 (13) | 0.0003 (11) | 0.0040 (11) |
| O8 | 0.0557 (14) | 0.0785 (18) | 0.0604 (15) | -0.0041 (13) | 0.0187 (12) | 0.0028 (13) |
| O9 | 0.0762 (17) | 0.0552 (15) | 0.0531 (14) | -0.0011 (12) | -0.0038 (12) | 0.0200 (12) |
| O10 | 0.0665 (15) | 0.0694 (16) | 0.0374 (11) | -0.0237 (13) | -0.0042 (10) | 0.0077 (11) |
| O11 | 0.066 (4) | 0.161 (8) | 0.166 (8) | 0.018 (5) | 0.012 (5) | -0.038 (7) |
| O11' | 0.166 (10) | 0.44 (2) | 0.134 (9) | 0.200 (13) | -0.060 (8) | -0.139 (12) |
| N1 | 0.0445 (14) | 0.0398 (14) | 0.0472 (15) | 0.0094 (11) | -0.0192 (12) | -0.0065 (11) |
| N2 | 0.0405 (13) | 0.0406 (14) | 0.0455 (14) | 0.0095 (11) | -0.0139 (11) | -0.0058 (12) |
| N3 | 0.0568 (16) | 0.0513 (16) | 0.0457 (15) | -0.0060 (13) | -0.0103 (13) | -0.0003 (13) |
| N4 | 0.0427 (15) | 0.0569 (17) | 0.0600 (18) | -0.0013 (13) | -0.0028 (13) | -0.0215 (14) |
| N5 | 0.0407 (13) | 0.0473 (15) | 0.0375 (13) | 0.0019 (11) | -0.0045 (10) | -0.0027 (11) |
| N6 | 0.0542 (16) | 0.0636 (19) | 0.0410 (14) | -0.0138 (14) | -0.0043 (12) | -0.0023 (13) |
| N7 | 0.0677 (19) | 0.063 (2) | 0.0460 (16) | -0.0100 (17) | -0.0225 (14) | 0.0035 (15) |
| N8 | 0.0456 (14) | 0.0493 (16) | 0.0359 (13) | -0.0056 (12) | -0.0085 (11) | -0.0029 (11) |
| N9 | 0.0436 (14) | 0.0422 (14) | 0.0450 (14) | -0.0098 (11) | -0.0097 (11) | 0.0080 (12) |
| N10 | 0.0363 (13) | 0.0548 (17) | 0.0518 (16) | 0.0003 (12) | 0.0005 (11) | -0.0042 (13) |
| N11 | 0.0458 (14) | 0.0397 (14) | 0.0510 (15) | -0.0024 (12) | -0.0124 (12) | 0.0074 (12) |
| N12 | 0.0373 (12) | 0.0282 (11) | 0.0300 (11) | 0.0003 (9) | 0.0012 (9) | -0.0014 (9) |
| N13 | 0.0433 (14) | 0.0490 (15) | 0.0416 (14) | -0.0100 (12) | -0.0047 (11) | 0.0013 (12) |
| N14 | 0.0562 (17) | 0.0667 (19) | 0.0327 (14) | -0.0186 (15) | -0.0074 (12) | 0.0015 (13) |
| C1 | 0.0478 (17) | 0.0433 (17) | 0.0412 (16) | -0.0025 (14) | -0.0136 (14) | -0.0022 (13) |
| C2 | 0.0418 (16) | 0.0423 (17) | 0.0473 (18) | -0.0019 (13) | -0.0070 (13) | -0.0116 (14) |
| C3 | 0.074 (3) | 0.072 (3) | 0.063 (2) | -0.008 (2) | -0.0149 (19) | 0.019 (2) |
| C4 | 0.051 (2) | 0.056 (2) | 0.072 (2) | -0.0057 (16) | 0.0080 (17) | -0.0145 (18) |
| C5 | 0.076 (2) | 0.063 (2) | 0.0438 (18) | -0.021 (2) | -0.0018 (18) | -0.0100 (17) |
| C6 | 0.063 (2) | 0.069 (2) | 0.057 (2) | -0.0193 (19) | 0.0118 (18) | -0.0249 (19) |
| C7 | 0.128 (4) | 0.092 (3) | 0.061 (3) | -0.040 (3) | 0.009 (3) | 0.007 (2) |
| C8 | 0.086 (3) | 0.094 (3) | 0.104 (4) | -0.027 (3) | 0.043 (3) | -0.038 (3) |
| C9 | 0.0442 (17) | 0.0531 (19) | 0.0479 (18) | 0.0004 (14) | -0.0064 (14) | -0.0025 (15) |
| C10 | 0.057 (2) | 0.0503 (19) | 0.0504 (19) | -0.0090 (16) | -0.0063 (15) | -0.0021 (15) |

| | | | | | | |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C11 | 0.0481 (17) | 0.059 (2) | 0.0356 (16) | -0.0074 (15) | -0.0034 (13) | -0.0009 (14) |
| C12 | 0.0498 (18) | 0.0533 (19) | 0.0431 (17) | 0.0009 (15) | -0.0093 (14) | -0.0014 (14) |
| C13 | 0.060 (2) | 0.069 (3) | 0.0424 (18) | -0.0054 (18) | -0.0146 (15) | -0.0027 (17) |
| C14 | 0.061 (2) | 0.066 (2) | 0.0433 (18) | -0.0154 (18) | -0.0194 (16) | 0.0015 (16) |
| C15 | 0.077 (3) | 0.096 (3) | 0.050 (2) | -0.018 (2) | -0.0136 (19) | 0.012 (2) |
| C16 | 0.129 (4) | 0.073 (3) | 0.084 (3) | -0.009 (3) | -0.062 (3) | -0.004 (2) |
| C17 | 0.063 (3) | 0.113 (4) | 0.074 (3) | -0.010 (2) | -0.013 (2) | 0.016 (3) |
| C18 | 0.0469 (17) | 0.0482 (19) | 0.0460 (17) | 0.0005 (14) | -0.0146 (14) | -0.0132 (14) |
| C19 | 0.0480 (18) | 0.0355 (16) | 0.059 (2) | -0.0018 (13) | -0.0195 (15) | -0.0050 (14) |
| C20 | 0.068 (2) | 0.071 (3) | 0.068 (2) | 0.008 (2) | -0.0103 (19) | -0.030 (2) |
| C21 | 0.077 (3) | 0.040 (2) | 0.102 (3) | -0.0010 (18) | -0.031 (2) | 0.003 (2) |
| C22 | 0.0475 (19) | 0.056 (2) | 0.070 (2) | 0.0124 (16) | -0.0085 (16) | -0.0154 (18) |
| C23 | 0.055 (2) | 0.0414 (18) | 0.074 (2) | 0.0075 (15) | -0.0159 (18) | 0.0001 (17) |
| C24 | 0.081 (3) | 0.092 (3) | 0.098 (3) | 0.033 (3) | 0.005 (3) | -0.028 (3) |
| C25 | 0.125 (4) | 0.046 (2) | 0.123 (4) | 0.022 (3) | -0.014 (3) | 0.009 (3) |
| C26 | 0.0469 (17) | 0.0436 (17) | 0.0345 (15) | -0.0086 (13) | -0.0014 (12) | 0.0052 (13) |
| C27 | 0.0462 (17) | 0.057 (2) | 0.0426 (17) | -0.0170 (15) | -0.0008 (13) | 0.0082 (15) |
| C28 | 0.0402 (15) | 0.0344 (14) | 0.0314 (14) | -0.0006 (12) | -0.0001 (11) | -0.0036 (11) |
| C29 | 0.0375 (14) | 0.0396 (15) | 0.0311 (14) | -0.0031 (12) | 0.0015 (11) | -0.0021 (12) |
| C30 | 0.0464 (17) | 0.0452 (17) | 0.0331 (15) | -0.0014 (14) | -0.0022 (13) | -0.0045 (13) |
| C31 | 0.068 (2) | 0.083 (3) | 0.0332 (16) | -0.020 (2) | -0.0126 (15) | 0.0017 (17) |
| C32 | 0.118 (4) | 0.170 (6) | 0.056 (3) | -0.081 (4) | -0.040 (3) | 0.016 (3) |
| C33 | 0.110 (4) | 0.100 (4) | 0.077 (3) | 0.010 (3) | -0.027 (3) | 0.029 (3) |
| C34 | 0.108 (4) | 0.115 (4) | 0.039 (2) | -0.018 (3) | 0.002 (2) | -0.012 (2) |

Geometric parameters (Å, °)

| | | | |
|---------|------------|----------|-----------|
| Co1—N4 | 1.888 (3) | C7—H7A | 0.9600 |
| Co1—N3 | 1.890 (3) | C7—H7B | 0.9600 |
| Co1—N2 | 1.892 (3) | C7—H7C | 0.9600 |
| Co1—N1 | 1.899 (3) | C8—H8A | 0.9600 |
| Co1—N5 | 1.970 (2) | C8—H8B | 0.9600 |
| Co1—Cl1 | 2.2282 (9) | C8—H8C | 0.9600 |
| Co2—N11 | 1.885 (3) | C9—C10 | 1.379 (5) |
| Co2—N10 | 1.886 (3) | C9—H9 | 0.9300 |
| Co2—N8 | 1.906 (3) | C10—H10 | 0.9300 |
| Co2—N9 | 1.910 (3) | C11—C12 | 1.380 (5) |
| Co2—N12 | 1.955 (2) | C11—C13 | 1.521 (5) |
| Co2—Cl2 | 2.2177 (9) | C12—H12 | 0.9300 |
| O1—N1 | 1.350 (3) | C14—C17 | 1.507 (6) |
| O1—H1 | 0.8200 | C14—C15 | 1.513 (5) |
| O2—N2 | 1.337 (3) | C14—C16 | 1.515 (6) |
| O2—H2 | 0.8200 | C15—H15A | 0.9600 |
| O3—N4 | 1.337 (4) | C15—H15B | 0.9600 |
| O4—N3 | 1.337 (4) | C15—H15C | 0.9600 |
| O5—C13 | 1.218 (5) | C16—H16A | 0.9600 |
| O6—N9 | 1.349 (3) | C16—H16B | 0.9600 |
| O6—H6 | 0.8200 | C16—H16C | 0.9600 |
| O7—N8 | 1.355 (3) | C17—H17A | 0.9600 |

supplementary materials

| | | | |
|-------------------------------------|-------------|-------------|-----------|
| O8—N10 | 1.316 (4) | C17—H17B | 0.9600 |
| O9—N11 | 1.323 (4) | C17—H17C | 0.9600 |
| O9—H9A | 0.8200 | C18—C19 | 1.463 (5) |
| O10—C30 | 1.218 (4) | C18—C20 | 1.488 (5) |
| O11 ⁱ —O11 ⁱⁱ | 1.314 (19) | C19—C21 | 1.493 (5) |
| N1—C1 | 1.286 (4) | C20—H20A | 0.9600 |
| N2—C2 | 1.288 (4) | C20—H20B | 0.9600 |
| N3—C5 | 1.288 (5) | C20—H20C | 0.9600 |
| N4—C6 | 1.298 (5) | C21—H21A | 0.9600 |
| N5—C9 | 1.331 (4) | C21—H21B | 0.9600 |
| N5—C12 | 1.335 (4) | C21—H21C | 0.9600 |
| N6—C11 | 1.326 (5) | C22—C23 | 1.449 (6) |
| N6—C10 | 1.329 (4) | C22—C24 | 1.505 (5) |
| N7—C13 | 1.330 (5) | C23—C25 | 1.495 (5) |
| N7—C14 | 1.486 (4) | C24—H24A | 0.9600 |
| N7—H7 | 0.82 (3) | C24—H24B | 0.9600 |
| N8—C18 | 1.288 (4) | C24—H24C | 0.9600 |
| N9—C19 | 1.287 (4) | C25—H25A | 0.9600 |
| N10—C22 | 1.301 (5) | C25—H25B | 0.9600 |
| N11—C23 | 1.293 (5) | C25—H25C | 0.9600 |
| N12—C29 | 1.333 (3) | C26—C27 | 1.372 (4) |
| N12—C26 | 1.334 (4) | C26—H26 | 0.9300 |
| N13—C28 | 1.327 (4) | C27—H27 | 0.9300 |
| N13—C27 | 1.333 (4) | C28—C29 | 1.382 (4) |
| N14—C30 | 1.325 (4) | C28—C30 | 1.507 (4) |
| N14—C31 | 1.480 (4) | C29—H29 | 0.9300 |
| N14—H14 | 0.82 (3) | C31—C33 | 1.512 (6) |
| C1—C2 | 1.461 (4) | C31—C32 | 1.514 (5) |
| C1—C3 | 1.487 (5) | C31—C34 | 1.523 (6) |
| C2—C4 | 1.490 (5) | C32—H32A | 0.9600 |
| C3—H3A | 0.9600 | C32—H32B | 0.9600 |
| C3—H3B | 0.9600 | C32—H32C | 0.9600 |
| C3—H3C | 0.9600 | C33—H33A | 0.9600 |
| C4—H4A | 0.9600 | C33—H33B | 0.9600 |
| C4—H4B | 0.9600 | C33—H33C | 0.9600 |
| C4—H4C | 0.9600 | C34—H34A | 0.9600 |
| C5—C6 | 1.463 (6) | C34—H34B | 0.9600 |
| C5—C7 | 1.492 (6) | C34—H34C | 0.9600 |
| C6—C8 | 1.499 (6) | | |
| N4—Co1—N3 | 81.95 (14) | N6—C10—C9 | 122.2 (3) |
| N4—Co1—N2 | 179.10 (12) | N6—C10—H10 | 118.9 |
| N3—Co1—N2 | 98.75 (12) | C9—C10—H10 | 118.9 |
| N4—Co1—N1 | 98.70 (13) | N6—C11—C12 | 122.7 (3) |
| N3—Co1—N1 | 176.55 (11) | N6—C11—C13 | 118.8 (3) |
| N2—Co1—N1 | 80.55 (11) | C12—C11—C13 | 118.5 (3) |
| N4—Co1—N5 | 90.50 (11) | N5—C12—C11 | 120.4 (3) |
| N3—Co1—N5 | 91.20 (11) | N5—C12—H12 | 119.8 |
| N2—Co1—N5 | 90.05 (10) | C11—C12—H12 | 119.8 |

| | | | |
|-------------|-------------|---------------|-----------|
| N1—Co1—N5 | 92.18 (11) | O5—C13—N7 | 126.8 (3) |
| N4—Co1—C11 | 88.85 (8) | O5—C13—C11 | 119.4 (3) |
| N3—Co1—C11 | 88.31 (9) | N7—C13—C11 | 113.8 (3) |
| N2—Co1—C11 | 90.61 (8) | N7—C14—C17 | 109.7 (3) |
| N1—Co1—C11 | 88.33 (8) | N7—C14—C15 | 109.5 (3) |
| N5—Co1—C11 | 179.23 (8) | C17—C14—C15 | 110.3 (4) |
| N11—Co2—N10 | 82.07 (12) | N7—C14—C16 | 106.3 (3) |
| N11—Co2—N8 | 98.34 (12) | C17—C14—C16 | 110.1 (4) |
| N10—Co2—N8 | 179.22 (12) | C15—C14—C16 | 110.9 (4) |
| N11—Co2—N9 | 178.16 (11) | C14—C15—H15A | 109.5 |
| N10—Co2—N9 | 99.23 (12) | C14—C15—H15B | 109.5 |
| N8—Co2—N9 | 80.34 (11) | H15A—C15—H15B | 109.5 |
| N11—Co2—N12 | 90.04 (10) | C14—C15—H15C | 109.5 |
| N10—Co2—N12 | 89.95 (10) | H15A—C15—H15C | 109.5 |
| N8—Co2—N12 | 90.72 (10) | H15B—C15—H15C | 109.5 |
| N9—Co2—N12 | 91.27 (10) | C14—C16—H16A | 109.5 |
| N11—Co2—C12 | 89.49 (8) | C14—C16—H16B | 109.5 |
| N10—Co2—C12 | 89.23 (8) | H16A—C16—H16B | 109.5 |
| N8—Co2—C12 | 90.11 (8) | C14—C16—H16C | 109.5 |
| N9—Co2—C12 | 89.22 (8) | H16A—C16—H16C | 109.5 |
| N12—Co2—C12 | 179.11 (7) | H16B—C16—H16C | 109.5 |
| N1—O1—H1 | 109.5 | C14—C17—H17A | 109.5 |
| N2—O2—H2 | 109.5 | C14—C17—H17B | 109.5 |
| N9—O6—H6 | 109.5 | H17A—C17—H17B | 109.5 |
| N11—O9—H9A | 109.5 | C14—C17—H17C | 109.5 |
| C1—N1—O1 | 120.1 (3) | H17A—C17—H17C | 109.5 |
| C1—N1—Co1 | 116.8 (2) | H17B—C17—H17C | 109.5 |
| O1—N1—Co1 | 122.7 (2) | N8—C18—C19 | 112.6 (3) |
| C2—N2—O2 | 120.3 (3) | N8—C18—C20 | 123.9 (3) |
| C2—N2—Co1 | 117.3 (2) | C19—C18—C20 | 123.5 (3) |
| O2—N2—Co1 | 122.5 (2) | N9—C19—C18 | 113.0 (3) |
| C5—N3—O4 | 122.0 (3) | N9—C19—C21 | 123.9 (4) |
| C5—N3—Co1 | 116.0 (3) | C18—C19—C21 | 123.0 (3) |
| O4—N3—Co1 | 122.0 (2) | C18—C20—H20A | 109.5 |
| C6—N4—O3 | 122.8 (3) | C18—C20—H20B | 109.5 |
| C6—N4—Co1 | 115.8 (3) | H20A—C20—H20B | 109.5 |
| O3—N4—Co1 | 121.4 (2) | C18—C20—H20C | 109.5 |
| C9—N5—C12 | 117.5 (3) | H20A—C20—H20C | 109.5 |
| C9—N5—Co1 | 121.2 (2) | H20B—C20—H20C | 109.5 |
| C12—N5—Co1 | 121.3 (2) | C19—C21—H21A | 109.5 |
| C11—N6—C10 | 116.1 (3) | C19—C21—H21B | 109.5 |
| C13—N7—C14 | 125.5 (3) | H21A—C21—H21B | 109.5 |
| C13—N7—H7 | 117 (3) | C19—C21—H21C | 109.5 |
| C14—N7—H7 | 117 (3) | H21A—C21—H21C | 109.5 |
| C18—N8—O7 | 120.3 (3) | H21B—C21—H21C | 109.5 |
| C18—N8—Co2 | 117.2 (2) | N10—C22—C23 | 113.2 (3) |
| O7—N8—Co2 | 122.5 (2) | N10—C22—C24 | 122.6 (4) |
| C19—N9—O6 | 120.5 (3) | C23—C22—C24 | 124.1 (4) |
| C19—N9—Co2 | 116.8 (2) | N11—C23—C22 | 113.4 (3) |

supplementary materials

| | | | |
|-------------|-------------|---------------|-----------|
| O6—N9—Co2 | 122.6 (2) | N11—C23—C25 | 122.0 (4) |
| C22—N10—O8 | 122.9 (3) | C22—C23—C25 | 124.6 (4) |
| C22—N10—Co2 | 115.5 (3) | C22—C24—H24A | 109.5 |
| O8—N10—Co2 | 121.5 (2) | C22—C24—H24B | 109.5 |
| C23—N11—O9 | 121.7 (3) | H24A—C24—H24B | 109.5 |
| C23—N11—Co2 | 115.7 (2) | C22—C24—H24C | 109.5 |
| O9—N11—Co2 | 122.5 (2) | H24A—C24—H24C | 109.5 |
| C29—N12—C26 | 117.8 (2) | H24B—C24—H24C | 109.5 |
| C29—N12—Co2 | 121.22 (19) | C23—C25—H25A | 109.5 |
| C26—N12—Co2 | 121.03 (19) | C23—C25—H25B | 109.5 |
| C28—N13—C27 | 116.2 (3) | H25A—C25—H25B | 109.5 |
| C30—N14—C31 | 125.2 (3) | C23—C25—H25C | 109.5 |
| C30—N14—H14 | 116 (3) | H25A—C25—H25C | 109.5 |
| C31—N14—H14 | 118 (3) | H25B—C25—H25C | 109.5 |
| N1—C1—C2 | 112.8 (3) | N12—C26—C27 | 120.8 (3) |
| N1—C1—C3 | 123.6 (3) | N12—C26—H26 | 119.6 |
| C2—C1—C3 | 123.5 (3) | C27—C26—H26 | 119.6 |
| N2—C2—C1 | 112.3 (3) | N13—C27—C26 | 122.3 (3) |
| N2—C2—C4 | 124.4 (3) | N13—C27—H27 | 118.8 |
| C1—C2—C4 | 123.3 (3) | C26—C27—H27 | 118.8 |
| C1—C3—H3A | 109.5 | N13—C28—C29 | 122.5 (3) |
| C1—C3—H3B | 109.5 | N13—C28—C30 | 119.0 (3) |
| H3A—C3—H3B | 109.5 | C29—C28—C30 | 118.5 (3) |
| C1—C3—H3C | 109.5 | N12—C29—C28 | 120.4 (3) |
| H3A—C3—H3C | 109.5 | N12—C29—H29 | 119.8 |
| H3B—C3—H3C | 109.5 | C28—C29—H29 | 119.8 |
| C2—C4—H4A | 109.5 | O10—C30—N14 | 125.6 (3) |
| C2—C4—H4B | 109.5 | O10—C30—C28 | 119.6 (3) |
| H4A—C4—H4B | 109.5 | N14—C30—C28 | 114.8 (3) |
| C2—C4—H4C | 109.5 | N14—C31—C33 | 109.1 (3) |
| H4A—C4—H4C | 109.5 | N14—C31—C32 | 105.4 (3) |
| H4B—C4—H4C | 109.5 | C33—C31—C32 | 110.3 (4) |
| N3—C5—C6 | 113.3 (3) | N14—C31—C34 | 109.6 (3) |
| N3—C5—C7 | 122.3 (4) | C33—C31—C34 | 111.3 (4) |
| C6—C5—C7 | 124.4 (4) | C32—C31—C34 | 111.1 (4) |
| N4—C6—C5 | 113.0 (3) | C31—C32—H32A | 109.5 |
| N4—C6—C8 | 122.8 (4) | C31—C32—H32B | 109.5 |
| C5—C6—C8 | 124.2 (4) | H32A—C32—H32B | 109.5 |
| C5—C7—H7A | 109.5 | C31—C32—H32C | 109.5 |
| C5—C7—H7B | 109.5 | H32A—C32—H32C | 109.5 |
| H7A—C7—H7B | 109.5 | H32B—C32—H32C | 109.5 |
| C5—C7—H7C | 109.5 | C31—C33—H33A | 109.5 |
| H7A—C7—H7C | 109.5 | C31—C33—H33B | 109.5 |
| H7B—C7—H7C | 109.5 | H33A—C33—H33B | 109.5 |
| C6—C8—H8A | 109.5 | C31—C33—H33C | 109.5 |
| C6—C8—H8B | 109.5 | H33A—C33—H33C | 109.5 |
| H8A—C8—H8B | 109.5 | H33B—C33—H33C | 109.5 |
| C6—C8—H8C | 109.5 | C31—C34—H34A | 109.5 |
| H8A—C8—H8C | 109.5 | C31—C34—H34B | 109.5 |

| | | | |
|----------------|------------|----------------|------------|
| H8B—C8—H8C | 109.5 | H34A—C34—H34B | 109.5 |
| N5—C9—C10 | 120.9 (3) | C31—C34—H34C | 109.5 |
| N5—C9—H9 | 119.5 | H34A—C34—H34C | 109.5 |
| C10—C9—H9 | 119.5 | H34B—C34—H34C | 109.5 |
| N4—Co1—N1—C1 | 176.3 (2) | Co1—N1—C1—C3 | -170.2 (3) |
| N2—Co1—N1—C1 | -3.2 (2) | O2—N2—C2—C1 | -178.1 (2) |
| N5—Co1—N1—C1 | -92.9 (2) | Co1—N2—C2—C1 | 2.5 (3) |
| Cl1—Co1—N1—C1 | 87.7 (2) | O2—N2—C2—C4 | 1.8 (4) |
| N4—Co1—N1—O1 | 2.8 (2) | Co1—N2—C2—C4 | -177.6 (2) |
| N2—Co1—N1—O1 | -176.6 (2) | N1—C1—C2—N2 | -5.0 (4) |
| N5—Co1—N1—O1 | 93.7 (2) | C3—C1—C2—N2 | 170.5 (3) |
| Cl1—Co1—N1—O1 | -85.7 (2) | N1—C1—C2—C4 | 175.1 (3) |
| N3—Co1—N2—C2 | -176.5 (2) | C3—C1—C2—C4 | -9.4 (5) |
| N1—Co1—N2—C2 | 0.1 (2) | O4—N3—C5—C6 | 179.6 (3) |
| N5—Co1—N2—C2 | 92.3 (2) | Co1—N3—C5—C6 | 1.3 (4) |
| Cl1—Co1—N2—C2 | -88.1 (2) | O4—N3—C5—C7 | 1.7 (5) |
| N3—Co1—N2—O2 | 4.2 (2) | Co1—N3—C5—C7 | -176.7 (3) |
| N1—Co1—N2—O2 | -179.2 (2) | O3—N4—C6—C5 | -178.6 (3) |
| N5—Co1—N2—O2 | -87.0 (2) | Co1—N4—C6—C5 | -0.9 (4) |
| Cl1—Co1—N2—O2 | 92.6 (2) | O3—N4—C6—C8 | 0.2 (5) |
| N4—Co1—N3—C5 | -1.4 (2) | Co1—N4—C6—C8 | 177.9 (3) |
| N2—Co1—N3—C5 | 178.0 (2) | N3—C5—C6—N4 | -0.3 (4) |
| N5—Co1—N3—C5 | -91.7 (3) | C7—C5—C6—N4 | 177.6 (3) |
| Cl1—Co1—N3—C5 | 87.7 (2) | N3—C5—C6—C8 | -179.1 (4) |
| N4—Co1—N3—O4 | -179.7 (2) | C7—C5—C6—C8 | -1.1 (6) |
| N2—Co1—N3—O4 | -0.3 (2) | C12—N5—C9—C10 | 1.4 (5) |
| N5—Co1—N3—O4 | 89.9 (2) | Co1—N5—C9—C10 | -176.5 (2) |
| Cl1—Co1—N3—O4 | -90.7 (2) | C11—N6—C10—C9 | -2.5 (5) |
| N3—Co1—N4—C6 | 1.2 (2) | N5—C9—C10—N6 | 1.5 (5) |
| N1—Co1—N4—C6 | -175.3 (2) | C10—N6—C11—C12 | 0.9 (5) |
| N5—Co1—N4—C6 | 92.4 (3) | C10—N6—C11—C13 | 179.2 (3) |
| Cl1—Co1—N4—C6 | -87.2 (2) | C9—N5—C12—C11 | -3.0 (5) |
| N3—Co1—N4—O3 | 179.0 (2) | Co1—N5—C12—C11 | 174.9 (2) |
| N1—Co1—N4—O3 | 2.4 (2) | N6—C11—C12—N5 | 2.0 (5) |
| N5—Co1—N4—O3 | -89.9 (2) | C13—C11—C12—N5 | -176.4 (3) |
| Cl1—Co1—N4—O3 | 90.5 (2) | C14—N7—C13—O5 | 2.0 (7) |
| N4—Co1—N5—C9 | 64.1 (3) | C14—N7—C13—C11 | -176.5 (3) |
| N3—Co1—N5—C9 | 146.1 (3) | N6—C11—C13—O5 | 178.7 (4) |
| N2—Co1—N5—C9 | -115.2 (3) | C12—C11—C13—O5 | -2.9 (5) |
| N1—Co1—N5—C9 | -34.6 (3) | N6—C11—C13—N7 | -2.6 (5) |
| N4—Co1—N5—C12 | -113.8 (3) | C12—C11—C13—N7 | 175.8 (3) |
| N3—Co1—N5—C12 | -31.8 (3) | C13—N7—C14—C17 | -63.4 (5) |
| N2—Co1—N5—C12 | 67.0 (3) | C13—N7—C14—C15 | 57.7 (5) |
| N1—Co1—N5—C12 | 147.5 (3) | C13—N7—C14—C16 | 177.6 (4) |
| N11—Co2—N8—C18 | -179.6 (2) | O7—N8—C18—C19 | 178.8 (2) |
| N9—Co2—N8—C18 | 1.8 (2) | Co2—N8—C18—C19 | -2.4 (3) |
| N12—Co2—N8—C18 | -89.4 (2) | O7—N8—C18—C20 | -0.2 (4) |
| Cl2—Co2—N8—C18 | 90.9 (2) | Co2—N8—C18—C20 | 178.7 (2) |
| N11—Co2—N8—O7 | -0.7 (2) | O6—N9—C19—C18 | -178.2 (2) |

supplementary materials

| | | | |
|-----------------|------------|-----------------|------------|
| N9—Co2—N8—O7 | -179.4 (2) | Co2—N9—C19—C18 | -0.4 (3) |
| N12—Co2—N8—O7 | 89.4 (2) | O6—N9—C19—C21 | -0.3 (5) |
| Cl2—Co2—N8—O7 | -90.2 (2) | Co2—N9—C19—C21 | 177.6 (3) |
| N10—Co2—N9—C19 | 180.0 (2) | N8—C18—C19—N9 | 1.8 (4) |
| N8—Co2—N9—C19 | -0.7 (2) | C20—C18—C19—N9 | -179.3 (3) |
| N12—Co2—N9—C19 | 89.8 (2) | N8—C18—C19—C21 | -176.2 (3) |
| Cl2—Co2—N9—C19 | -90.9 (2) | C20—C18—C19—C21 | 2.7 (5) |
| N10—Co2—N9—O6 | -2.2 (2) | O8—N10—C22—C23 | 178.4 (3) |
| N8—Co2—N9—O6 | 177.1 (2) | Co2—N10—C22—C23 | 0.2 (4) |
| N12—Co2—N9—O6 | -92.4 (2) | O8—N10—C22—C24 | 1.1 (5) |
| Cl2—Co2—N9—O6 | 86.9 (2) | Co2—N10—C22—C24 | -177.1 (3) |
| N11—Co2—N10—C22 | 1.0 (2) | O9—N11—C23—C22 | -177.1 (3) |
| N9—Co2—N10—C22 | 179.7 (2) | Co2—N11—C23—C22 | 2.8 (4) |
| N12—Co2—N10—C22 | -89.0 (2) | O9—N11—C23—C25 | 1.1 (5) |
| Cl2—Co2—N10—C22 | 90.6 (2) | Co2—N11—C23—C25 | -178.9 (3) |
| N11—Co2—N10—O8 | -177.2 (2) | N10—C22—C23—N11 | -2.0 (4) |
| N9—Co2—N10—O8 | 1.5 (2) | C24—C22—C23—N11 | 175.2 (4) |
| N12—Co2—N10—O8 | 92.8 (2) | N10—C22—C23—C25 | 179.9 (4) |
| Cl2—Co2—N10—O8 | -87.6 (2) | C24—C22—C23—C25 | -2.9 (6) |
| N10—Co2—N11—C23 | -2.2 (2) | C29—N12—C26—C27 | 1.0 (4) |
| N8—Co2—N11—C23 | 178.5 (2) | Co2—N12—C26—C27 | -178.6 (2) |
| N12—Co2—N11—C23 | 87.7 (2) | C28—N13—C27—C26 | -0.5 (5) |
| Cl2—Co2—N11—C23 | -91.5 (2) | N12—C26—C27—N13 | -0.2 (5) |
| N10—Co2—N11—O9 | 177.8 (2) | C27—N13—C28—C29 | 0.3 (4) |
| N8—Co2—N11—O9 | -1.6 (2) | C27—N13—C28—C30 | -177.6 (3) |
| N12—Co2—N11—O9 | -92.3 (2) | C26—N12—C29—C28 | -1.2 (4) |
| Cl2—Co2—N11—O9 | 88.5 (2) | Co2—N12—C29—C28 | 178.5 (2) |
| N11—Co2—N12—C29 | -132.3 (2) | N13—C28—C29—N12 | 0.5 (4) |
| N10—Co2—N12—C29 | -50.2 (2) | C30—C28—C29—N12 | 178.5 (3) |
| N8—Co2—N12—C29 | 129.4 (2) | C31—N14—C30—O10 | -2.8 (6) |
| N9—Co2—N12—C29 | 49.0 (2) | C31—N14—C30—C28 | 178.0 (3) |
| N11—Co2—N12—C26 | 47.4 (2) | N13—C28—C30—O10 | 170.2 (3) |
| N10—Co2—N12—C26 | 129.4 (2) | C29—C28—C30—O10 | -7.9 (4) |
| N8—Co2—N12—C26 | -51.0 (2) | N13—C28—C30—N14 | -10.6 (4) |
| N9—Co2—N12—C26 | -131.3 (2) | C29—C28—C30—N14 | 171.4 (3) |
| O1—N1—C1—C2 | 178.9 (2) | C30—N14—C31—C33 | -63.1 (5) |
| Co1—N1—C1—C2 | 5.3 (3) | C30—N14—C31—C32 | 178.6 (4) |
| O1—N1—C1—C3 | 3.4 (5) | C30—N14—C31—C34 | 59.0 (5) |

Symmetry codes: (i) $-x, -y, -z+2$.

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

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--------------------|-------|-------------|-------------|---------------|
| O1—H1 \cdots O3 | 0.82 | 1.69 | 2.476 (4) | 161 |
| O2—H2 \cdots O4 | 0.82 | 1.69 | 2.483 (4) | 162 |
| O6—H6 \cdots O8 | 0.82 | 1.72 | 2.507 (4) | 161 |
| O9—H9A \cdots O7 | 0.82 | 1.68 | 2.483 (4) | 165 |

Fig. 1

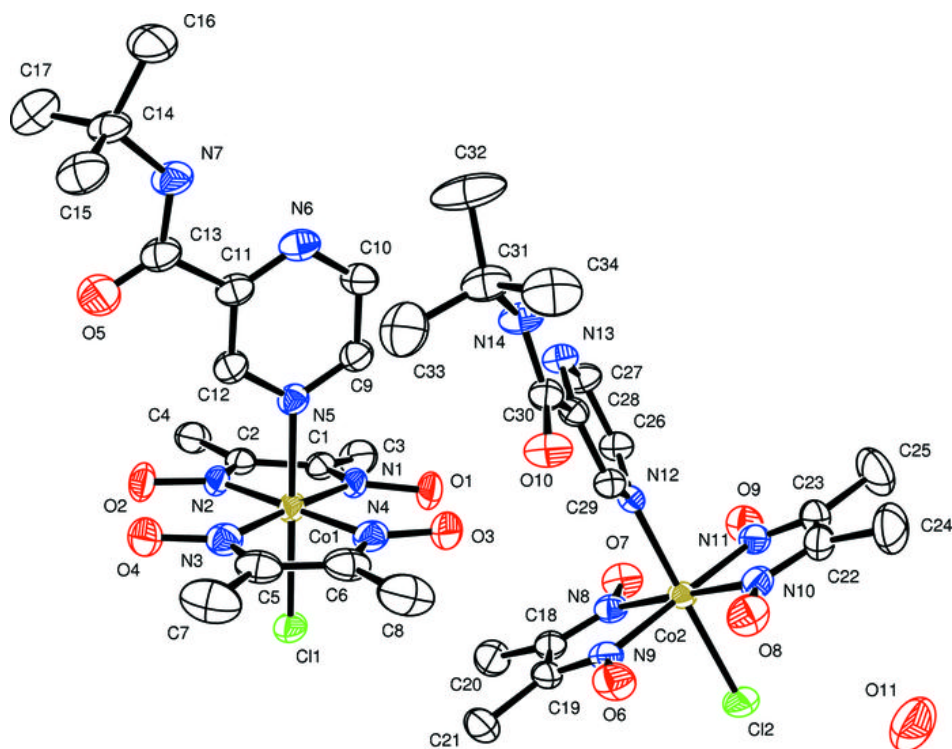


Fig. 2

