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# Crystal structure of di-$\mu$-chlorido-bis{chlorido[(−)-5,6-pinenebipyridine]cobalt(II)} aquadichlorido-[(−)-5,6-pinenebipyridine]cobalt(II)

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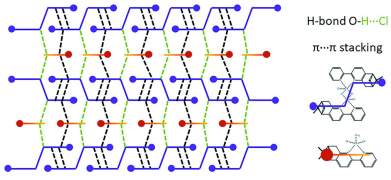
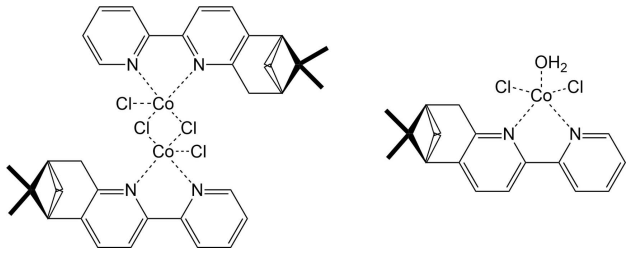
The crystal structure of $[Co_2Cl_4(C_{17}H_{18}N_2)_2][CoCl_2(C_{17}H_{18}N_2)(H_2O)]$ or $[Co(L)Cl(\mu\text{-}Cl)]_2[Co(L)(Cl)_2(OH_2)]$, where $L$ is the enantiopure bidentate ligand (−)-5,6-pinenebipyridine ($C_{17}H_{18}N_2$), has been determined. Crystals suitable for X-ray structure analysis were obtained by slow evaporation of an ethanolic solution containing equimolar amounts of $L$ and $CoCl_2{\cdot}6H_2O$. The $Co^{II}$ cations all have a coordination number of five, and in each case the coordination polyhedron is a trigonal bipyramid. The Co—N bonds lengths range from 2.037 (7) to 2.195 (7) Å, and Co—Cl bonds lengths range from 2.284 (2) to 2.509 (2) Å. The asymmetric unit contains two discrete complexes, one dinuclear and the other mononuclear. Between the two molecules, two types of intermolecular interactions have been evidenced: $\pi$–$\pi$ stackings involving the bipyridine units, and O—H···Cl hydrogen bonds between the hydrogen atoms of the aqua ligand coordinating to the mononuclear complex and the non-bridging chlorido ligand coordinating to the dinuclear molecule. These interactions lead to a two-dimensional supramolecular arrangement parallel to the *ab* plane.

## 1. Chemical context

Single-molecule magnets (SMMs) are metal–organic compounds that are superparamagnetic below a blocking temperature. It is important to note that this type of magnetism has a molecular origin, instead of the more traditional bulk-originated magnetism (Zhu *et al.*, 2013). Below the blocking temperature, a SMM exhibits magnetic hysteresis. In order to obtain a coordination compound behaving as an SMM, a paramagnetic metal cation has to be used, for example $Co^{II}$ (Lang *et al.*, 2019). Moreover, the use of chiral ligands for these paramagnetic metal cations can lead to predetermination of their chirality and thus to the synthesis of magnetochiral materials (Liu *et al.*, 2018). The enantiomers of 5,6-pinene bipyridine ($C_{17}H_{18}N_2$; $L$) and their derivatives have the ability to predetermine the chirality of $d$ and $f$ metal cations (Lama *et al.*, 2008; Mamula & von Zelewsky, 2003).

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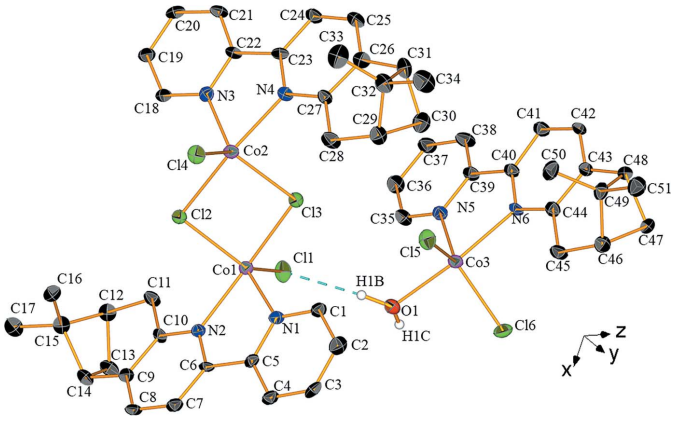


**Figure 1**
The molecular structures of the two complexes present in (**1**), with the O—H···Cl hydrogen bond shown as a dashed line. Displacement ellipsoids are set at the 30% probability level. Carbon-bound hydrogen atoms are omitted for clarity.

Within a current project we are investigating the metal complexes obtained with paramagnetic metal cations, *i.e.* $Co^{II}$, and report here the crystal structure of $[Co(L)Cl(\mu\text{-}Cl)]_2[Co(L)(Cl)_2(OH_2)]$ (**1**).

## 2. Structural commentary

The asymmetric unit of (**1**) comprises two discrete complexes (Fig. 1). The dinuclear complex possess two bidentate terminal (−)-5,6-pinenebipyridine ligands coordinated by two distinct $Co^{II}$ cations (Co1, Co2) *via* their nitrogen atoms. The two $Co^{II}$ cations are linked by two bridging chlorido ligands (Cl2, Cl3). Each coordination sphere is completed by two additional terminal chlorido ligands (Cl1, Cl4), leading to a coordination number of 5 in each case. The mononuclear complex (Co3) also features a $Co^{II}$ cation with a coordination number of 5. In this case, one bidentate (−)-5,6-pinenebipyridine, two terminal chlorido ligands (Cl5; Cl6) and an aqua ligand bind to the $Co^{II}$ cation. The two types of complexes interact *via* an O—H···Cl hydrogen bond (indicated with a dashed line in Fig. 1; Table 1) between one hydrogen atom belonging to the aqua ligand of the mononuclear complex and a terminal chlorido ligand belonging to the dinuclear complex. The other hydrogen atom of the water molecule forms another hydrogen bond with a dinuclear complex belonging to a neighbouring molecule (*vide infra*).

The geometric parameters for the trigonal–bipyramidal coordination environments are similar for the three $Co^{II}$ cations. In order to compare their coordination polyhedra, the values for the parameter $\tau$ were calculated. For a perfect trigonal–bipyramidal arrangement $\tau$ is 1, and for a perfect square-pyramidal arrangement $\tau$ is 0 (Addison *et al.*, 1984). The polyhedron around the cation in the mononuclear complex (Co3 in Fig. 2) is the closest to trigonal–bipyramidal ($\tau$ = 0.78). However, those of the cations of the dinuclear complex are not so different ($\tau$ = 0.69 for Co1, $\tau$ = 0.64 for Co2, see Fig. 2).

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

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—H1*A*···Cl1 | 0.84 (10) | 2.37 (10) | 3.194 (7) | 166 (9) |
| O1—H1*B*···Cl4$^{i}$ | 0.87 (10) | 2.43 (10) | 3.260 (7) | 161 (9) |

Symmetry code: (i) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$.

The Co—N bond lengths are between 2.037 (7) and 2.195 (7) Å, the Co—Cl bonds lengths are between 2.284 (2) and 2.509 (2) Å and the Co—O bond length is 2.160 (6) Å, which are all within the expected ranges (Bernhardt & Lawrance, 2003).

## 3. Supramolecular features

In the crystal, hydrogen-bonding interactions occur between the dinuclear and mononuclear complexes, leading to a supramolecular zigzag chain extending parallel to the *b* axis

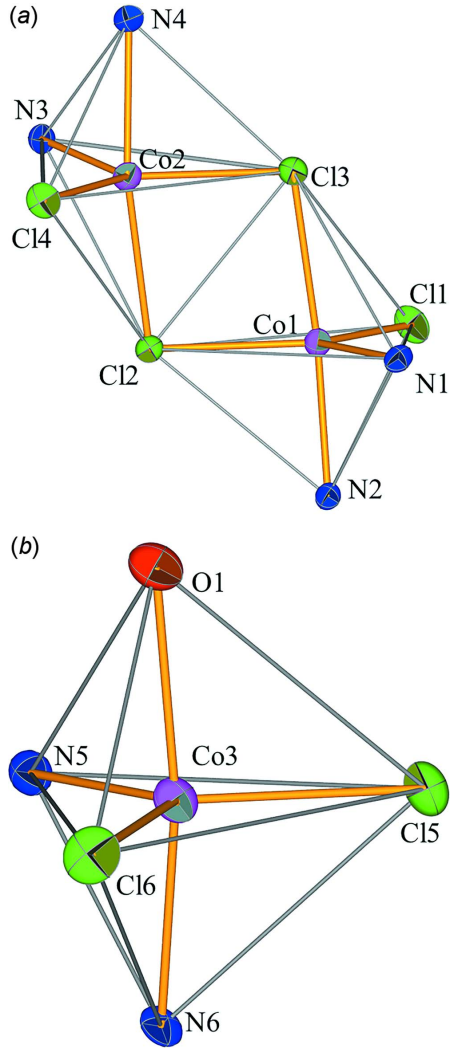


**Figure 2**
The trigonal–bipyramidal coordination spheres of the $Co^{II}$ cations in (*a*) the dinuclear complex and (*b*) the mononuclear complex. Non-coordinating atoms are omitted for clarity.

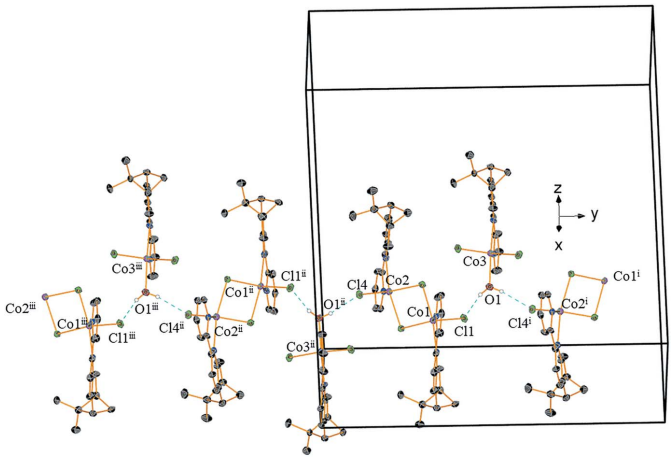


**Figure 3**
Hydrogen bonds (blue dotted lines) forming an infinite supramolecular chain. Carbon-bound hydrogen atoms are omitted for clarity. [Symmetry codes: (i) $1-x, \frac{1}{2}+y, \frac{1}{2}-z$; (ii) $1-x, -\frac{1}{2}+y, \frac{1}{2}-z$; (iii) $x, -1+y, z$.]

(Fig. 3). The hydrogen atoms of the aqua ligand of the mononuclear complex form hydrogen bonds with the terminal chlorido ligands belonging to the dinuclear complex. The bond lengths and angles (Table 1), are in the expected ranges for this type of interaction (Steiner, 2002).

This arrangement is stabilized by $\pi$–$\pi$ stacking interactions, which are responsible for the cohesion of the structure by forming layers of alternating dinuclear and mononuclear complexes extending parallel to the *ab* plane (Fig. 4). Neighbouring dinuclear complexes are connected *via* $\pi$–$\pi$ interactions between the bipyridine units whereby two $\pi$–$\pi$ interactions are established between the two pyridine rings annelated to the pinene moiety and the two 'free pyridines' (the pinene-free pyridine rings of the pinene-bipyridine ligands). The distances between the aromatic centroids are 3.793 (5) Å (slippage 0.987 Å) and 3.940 (5) Å (slippage 1.278 Å). The two pinene bipyridine ligands belonging to neighbouring dinuclear complexes are connected *via* their 'free' pyridine entity to the 'free' pyridine entities of the pinenebipyridine ligands of the mononuclear complexes. The distances [3.625 (5) Å with a slippage of 1.137 Å, and 3.718 (5) Å with a slippage of 1.503 Å] are typical for these kinds of interactions (Robin & Fromm, 2006).

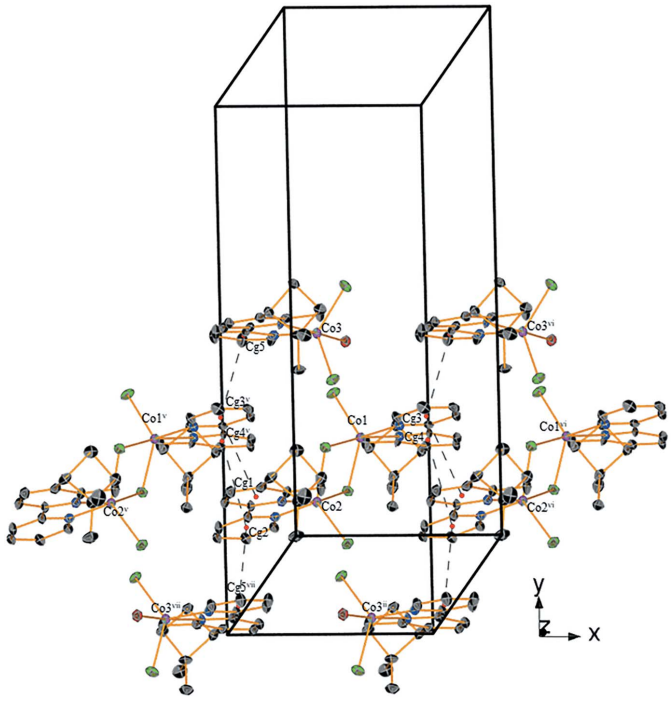


**Figure 4**
$\pi$–$\pi$ stacking interactions shown as dotted black lines. [Symmetry codes: (ii) $1-x, -\frac{1}{2}+y, \frac{1}{2}-z$; (v) $-1+x, y, z$; (vi) $1+x, y, z$; (vii) $-x, -\frac{1}{2}+y, \frac{1}{2}-z$.]

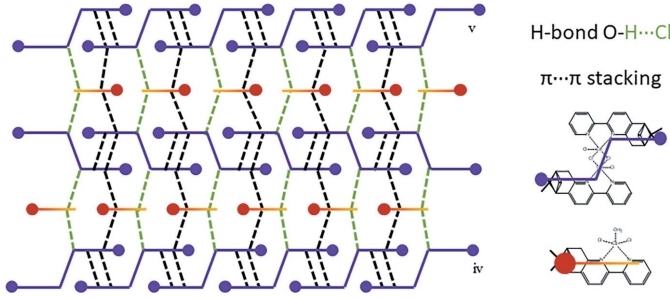


**Figure 5**
Schematic representation of the two-dimensional arrangement in the crystal structure of (**1**). [Symmetry codes: (iv) $-x, \frac{1}{2}+y, \frac{1}{2}-z$; (v) $-1+x, y, z$.]

Considering all the intermolecular interactions (hydrogen bonds and $\pi$–$\pi$ stackings), the two-dimensional supramolecular arrangement can be drawn schematically as shown in Fig. 5.

## 4. Database survey

A survey of the Cambridge Structural Database (Version 5.42, September 2021; Groom *et al.*, 2016) revealed no cobalt complexes containing the ligand (−) or (+)-5,6-pinenebipyridine (nor 4,5-pinenebipyridine). However, a few mononuclear complexes with ligands containing the 5,6-pinenebipyridine moiety in their skeleton have been reported. A tetrahedral $Co^{II}$ complex, UCUFAZ, containing a bidentate bipyridine ligand analogue to the ligand *L* but containing two pinene groups, has been characterized (Lötscher *et al.*, 2001). Two tridentate ligands, UKITOX and UKIVAL (Suhr *et al.*, 2002), composed of 2,2′:6′,2″ terpyridine containing two pinene groups annelated to the terminal pyridine rings, coordinated by a $Co^{II}$ cation together with two chloride anions to form a complex whose geometry is pseudo-trigonal–bipyramidal. Finally, Yeung *et al.* (2009) used terpyridine ligands from the same family as the ones of Suhr *et al.* and obtained similar structures (XUDHOU and XUDJEM).

## 5. Synthesis and crystallization

A pink solution of $CoCl_2{\cdot}6H_2O$ (238 mg, 1 mmol) in ethanol (4 ml) was added to a colourless solution containing *L*

**Table 2**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $[Co_2Cl_4(C_{17}H_{18}N_2)_2]$-$[CoCl_2(C_{17}H_{18}N_2)(H_2O)]$ |
| $M_r$ | 1158.50 |
| Crystal system, space group | Orthorhombic, $P2_12_12_1$ |
| Temperature (K) | 200 |
| $a, b, c$ (Å) | 8.5470 (4), 22.0971 (9), 26.9407 (12) |
| $V$ (Å$^3$) | 5088.1 (4) |
| $Z$ | 4 |
| Radiation type | Cu $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 10.82 |
| Crystal size (mm) | 0.21 × 0.11 × 0.05 |
| Data collection | |
| Diffractometer | Stoe IPDS 2T |
| Absorption correction | Integration (*X-RED32*; Stoe, 2016) |
| $T_{min}$, $T_{max}$ | 0.176, 0.523 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 40552, 8979, 7084 |
| $R_{int}$ | 0.129 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.602 |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.053, 0.138, 1.07 |
| No. of reflections | 8979 |
| No. of parameters | 617 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.63, −0.51 |
| Absolute structure | Flack $x$ determined using 2418 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013). |
| Absolute structure parameter | −0.042 (4) |

Computer programs: *X-AREA* and *X-RED32* (Stoe, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*), *OLEX2* (Dolomanov *et al.*, 2009), *PLATON* (Spek, 2020) and *publCIF* (Westrip, 2010).

(250 mg, 1 mmol) in ethanol (20 ml) and stirred for a few minutes. A fraction of the total volume of the resulting blue solution (about 3 ml) was transferred into a test tube and left to evaporate slowly under ambient conditions. Within a few days, violet single crystals were harvested.

## 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The C-bound H atoms were placed in geometrically idealized positions (C—H = 0.95–1.00 Å) while those attached to O were positioned from a difference-Fourier map, then refined for a few cycles to ensure that reasonable displacement parameters could be achieved. Their coordinates were adjusted to give O—H = 0.87 Å. All hydrogen atoms were refined using a riding model with isotropic displacement parameters 1.2–1.5 times those of the parent atoms.


## Acknowledgements

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## References


Addison, A. W., Rao, T. N., Reedijk, J., van Rijn, J. & Verschoor, G. C. (1984). *J. Chem. Soc. Dalton Trans.* pp. 1349–1356.

Bernhardt, P. V. & Lawrance, G. A. (2003). *Comprehensive Coordination Chemistry II*, vol. 6, ch. 6.1 *Cobalt.* Amsterdam: Elsevier Pergamon.

Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.

Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.

Lama, M., Mamula, O., Kottas, G. S., De Cola, L., Stoeckli-Evans, H. & Shova, S. (2008). *Inorg. Chem.* **47**, 8000–8015.

Lang, W.-J., Kurmoo, M. & Zeng, M.-H. (2019). *Inorg. Chem.* **58**, 7236–7242.

Liu, M.-J., Yuan, J., Wang, B.-L., Wu, S.-T., Zhang, Y.-Q., Liu, C.-M. & Kou, H.-Z. (2018). *Cryst. Growth Des.* **18**, 7611–7617.

Lötscher, D., Rupprecht, S., Collomb, P., Belser, P., Viebrock, H., von Zelewsky, A. & Burger, P. (2001). *Inorg. Chem.* **40**, 5675–5681.

Mamula, O. (2003). *Coord. Chem. Rev.* **242**, 87–95.

Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* B**69**, 249–259.

Robin, Y. A. & Fromm, K. M. (2006). *Coord. Chem. Rev.* **250**, 2127–2157.

Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.

Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.

Spek, A. L. (2020). *Acta Cryst.* E**76**, 1–11.

Steiner, T. (2002). *Angew. Chem. Int. Ed.* **41**, 48–76.

Stoe (2016). *X-AREA* and *X-RED32*. Stoe & Cie, Darmstadt, Germany.

Suhr, D., Lötscher, D., Stoeckli-Evans, H. & von Zelewsky, A. (2002). *Inorg. Chim. Acta*, **341**, 17–24.

Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

Yeung, C.-T., Sham, K.-C., Lee, W.-S., Wong, W.-T., Wong, W.-Y. & Kwong, H.-L. (2009). *Inorg. Chim. Acta*, **362**, 326–3273.

Zhu, Y.-Y., Cui, C., Zhang, Y.-Q., Jia, J.-H., Guo, X., Gao, C., Qian, K., Jiang, S.-D., Wang, B.-W., Wang, Z.-M. & Gao, S. (2013). *Chem. Sci.* **4**, 1802–1806.

# supporting information

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# Crystal structure of di-$\mu$-chlorido-bis{chlorido[(−)-5,6-pinenebipyridine]-cobalt(II)} aquadichlorido[(−)-5,6-pinenebipyridine]cobalt(II)

**Massimo Varisco, Aurelien Crochet and Olimpia Mamula Steiner**

## Computing details

Data collection: *X-AREA* (Stoe, 2016); cell refinement: *X-AREA* (Stoe, 2016); data reduction: *X-RED32* (Stoe, 2016); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015b); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *PLATON* (Spek, 2020) and *publCIF* (Westrip, 2010).

**Di-$\mu$-chlorido-bis{chlorido[(–)-5,6-pinenebipyridine]cobalt(II)} aquadichlorido[(–)-5,6-pinenebipyridine]cobalt(II)**

*Crystal data*

$[Co_2Cl_4(C_{17}H_{18}N_2)_2][CoCl_2(C_{17}H_{18}N_2)(H_2O)]$
$M_r$ = 1158.50
Orthorhombic, $P2_12_12_1$
$a$ = 8.5470 (4) Å
$b$ = 22.0971 (9) Å
$c$ = 26.9407 (12) Å
$V$ = 5088.1 (4) Å$^3$
$Z$ = 4
$F(000)$ = 2380
$D_x$ = 1.512 Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda$ = 1.54186 Å
Cell parameters from 32247 reflections
$\theta$ = 2.6–68.1°
$\mu$ = 10.82 mm$^{-1}$
$T$ = 200 K
Prism, violet
0.21 × 0.11 × 0.05 mm

*Data collection*

Stoe IPDS 2T diffractometer
Radiation source: Genix-Cu, 3D, microfocus
Multilayer optic monochromator
Detector resolution: 6.67 pixels mm$^{-1}$
rotation method, $\omega$ scans
Absorption correction: integration (*X-Red32*; Stoe, 2016)
$T_{min}$ = 0.176, $T_{max}$ = 0.523
40552 measured reflections
8979 independent reflections
7084 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.129
$\theta_{max}$ = 68.1°, $\theta_{min}$ = 2.6°
$h$ = −10→9
$k$ = −25→26
$l$ = −31→31

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.138
$S$ = 1.07
8979 reflections
617 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0437P)^2 + 12.2194P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.63 e Å$^{-3}$

$\Delta\rho_{min}$ = −0.51 e Å$^{-3}$
Extinction correction: SHELXL2017/1 (Sheldrick 2015b), Fc$^{*}$=kFc[1+0.001xFc$^{2}\lambda^{3}$/sin(2$\theta$)]$^{-1/4}$
Extinction coefficient: 0.00083 (12)

Absolute structure: Flack *x* determined using 2418 quotients [($I^{+}$)-($I^{-}$)]/[($I^{+}$)+($I^{-}$)] (Parsons *et al.*, 2013).
Absolute structure parameter: −0.042 (4)

*Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^{2}$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Co1 | 0.59450 (15) | 0.33142 (6) | 0.21719 (5) | 0.0261 (3) |
| Co2 | 0.36326 (15) | 0.20357 (6) | 0.25146 (5) | 0.0272 (3) |
| Co3 | 0.34642 (15) | 0.50356 (6) | 0.35583 (5) | 0.0276 (3) |
| H1A | 0.494 (12) | 0.473 (4) | 0.271 (4) | 0.041* |
| H1B | 0.518 (12) | 0.535 (5) | 0.281 (4) | 0.041* |
| Cl1 | 0.4747 (3) | 0.41998 (10) | 0.19496 (8) | 0.0416 (6) |
| Cl2 | 0.5387 (2) | 0.23709 (9) | 0.18204 (8) | 0.0335 (5) |
| Cl3 | 0.4013 (3) | 0.30157 (10) | 0.27947 (8) | 0.0361 (5) |
| Cl4 | 0.4998 (3) | 0.11977 (10) | 0.27565 (8) | 0.0373 (5) |
| Cl5 | 0.4083 (3) | 0.40985 (10) | 0.38590 (9) | 0.0416 (5) |
| Cl6 | 0.4811 (3) | 0.58759 (10) | 0.38148 (8) | 0.0398 (5) |
| O1 | 0.5059 (7) | 0.4990 (3) | 0.2938 (2) | 0.0365 (14) |
| N1 | 0.7658 (8) | 0.3446 (3) | 0.2702 (3) | 0.0269 (15) |
| N2 | 0.7995 (8) | 0.3417 (3) | 0.1717 (3) | 0.0263 (15) |
| N3 | 0.1742 (8) | 0.1839 (3) | 0.2083 (2) | 0.0279 (16) |
| N4 | 0.1745 (8) | 0.1953 (3) | 0.3063 (2) | 0.0272 (15) |
| N5 | 0.1534 (8) | 0.5088 (3) | 0.3085 (3) | 0.0286 (15) |
| N6 | 0.1529 (7) | 0.5121 (3) | 0.4079 (2) | 0.0247 (15) |
| C1 | 0.7377 (10) | 0.3510 (4) | 0.3188 (3) | 0.032 (2) |
| H1 | 0.633294 | 0.348141 | 0.330610 | 0.038* |
| C2 | 0.8577 (12) | 0.3618 (4) | 0.3522 (3) | 0.041 (2) |
| H2 | 0.835136 | 0.366735 | 0.386451 | 0.049* |
| C3 | 1.0110 (11) | 0.3653 (4) | 0.3355 (3) | 0.034 (2) |
| H3 | 1.094813 | 0.371669 | 0.358029 | 0.041* |
| C4 | 1.0386 (10) | 0.3593 (4) | 0.2854 (3) | 0.032 (2) |
| H4 | 1.142196 | 0.362691 | 0.272955 | 0.039* |
| C5 | 0.9144 (10) | 0.3483 (3) | 0.2528 (3) | 0.0258 (17) |
| C6 | 0.9352 (8) | 0.3422 (3) | 0.1989 (3) | 0.0223 (17) |
| C7 | 1.0783 (10) | 0.3370 (4) | 0.1769 (3) | 0.0303 (19) |
| H7 | 1.170105 | 0.335479 | 0.196705 | 0.036* |
| C8 | 1.0903 (10) | 0.3339 (4) | 0.1254 (3) | 0.0313 (19) |
| H8 | 1.189342 | 0.329634 | 0.109810 | 0.038* |
| C9 | 0.9546 (10) | 0.3371 (4) | 0.0976 (3) | 0.0292 (18) |
| C10 | 0.8095 (9) | 0.3408 (4) | 0.1224 (3) | 0.0251 (18) |

| | | | | |
|---|---|---|---|---|
| C11 | 0.6631 (10) | 0.3451 (4) | 0.0912 (3) | 0.032 (2) |
| H11A | 0.605315 | 0.382354 | 0.100103 | 0.038* |
| H11B | 0.594716 | 0.310019 | 0.098448 | 0.038* |
| C12 | 0.7014 (10) | 0.3461 (4) | 0.0360 (3) | 0.033 (2) |
| H12 | 0.609579 | 0.350497 | 0.013238 | 0.040* |
| C13 | 0.8383 (11) | 0.3905 (4) | 0.0265 (3) | 0.037 (2) |
| H13A | 0.840412 | 0.425698 | 0.049239 | 0.044* |
| H13B | 0.848812 | 0.403466 | −0.008566 | 0.044* |
| C14 | 0.9509 (10) | 0.3382 (4) | 0.0417 (3) | 0.033 (2) |
| H14 | 1.054115 | 0.336437 | 0.024254 | 0.040* |
| C15 | 0.8196 (11) | 0.2943 (4) | 0.0222 (3) | 0.034 (2) |
| C16 | 0.8006 (11) | 0.2322 (4) | 0.0473 (3) | 0.036 (2) |
| H16A | 0.708187 | 0.211769 | 0.033864 | 0.054* |
| H16B | 0.787762 | 0.237847 | 0.083197 | 0.054* |
| H16C | 0.893754 | 0.207563 | 0.040997 | 0.054* |
| C17 | 0.8308 (12) | 0.2847 (5) | −0.0338 (3) | 0.044 (2) |
| H17A | 0.918498 | 0.257568 | −0.041212 | 0.066* |
| H17B | 0.847684 | 0.323695 | −0.050272 | 0.066* |
| H17C | 0.733323 | 0.266590 | −0.045969 | 0.066* |
| C18 | 0.1847 (10) | 0.1732 (4) | 0.1592 (3) | 0.0292 (19) |
| H18 | 0.283740 | 0.176303 | 0.143423 | 0.035* |
| C19 | 0.0552 (10) | 0.1578 (4) | 0.1312 (3) | 0.034 (2) |
| H19 | 0.066389 | 0.149282 | 0.096842 | 0.041* |
| C20 | −0.0916 (10) | 0.1547 (4) | 0.1534 (3) | 0.033 (2) |
| H20 | −0.182621 | 0.145898 | 0.134516 | 0.040* |
| C21 | −0.1003 (10) | 0.1649 (4) | 0.2038 (3) | 0.0318 (19) |
| H21 | −0.197951 | 0.161697 | 0.220489 | 0.038* |
| C22 | 0.0331 (9) | 0.1797 (4) | 0.2302 (3) | 0.0264 (18) |
| C23 | 0.0307 (9) | 0.1907 (4) | 0.2846 (3) | 0.0274 (18) |
| C24 | −0.1050 (10) | 0.1930 (4) | 0.3122 (3) | 0.034 (2) |
| H24 | −0.203972 | 0.188426 | 0.296598 | 0.041* |
| C25 | −0.0950 (11) | 0.2022 (4) | 0.3632 (3) | 0.038 (2) |
| H25 | −0.187522 | 0.205081 | 0.382585 | 0.045* |
| C26 | 0.0491 (10) | 0.2071 (4) | 0.3854 (3) | 0.0314 (19) |
| C27 | 0.1835 (9) | 0.2034 (4) | 0.3552 (3) | 0.0273 (18) |
| C28 | 0.3411 (11) | 0.2064 (5) | 0.3802 (3) | 0.040 (2) |
| H28A | 0.401156 | 0.240988 | 0.366747 | 0.048* |
| H28B | 0.400354 | 0.168884 | 0.373056 | 0.048* |
| C29 | 0.3229 (12) | 0.2137 (4) | 0.4364 (3) | 0.040 (2) |
| H29 | 0.423458 | 0.217251 | 0.455186 | 0.048* |
| C30 | 0.2034 (12) | 0.2649 (5) | 0.4465 (4) | 0.045 (2) |
| H30A | 0.201003 | 0.296958 | 0.420834 | 0.054* |
| H30B | 0.209118 | 0.282349 | 0.480339 | 0.054* |
| C31 | 0.0764 (12) | 0.2152 (4) | 0.4400 (3) | 0.040 (2) |
| H31 | −0.019252 | 0.219355 | 0.461137 | 0.047* |
| C32 | 0.2033 (12) | 0.1683 (5) | 0.4587 (4) | 0.042 (2) |
| C33 | 0.1953 (14) | 0.1045 (5) | 0.4379 (4) | 0.053 (3) |
| H33A | 0.291683 | 0.082708 | 0.446264 | 0.079* |

| | | | | |
|---|---|---|---|---|
| H33B | 0.183686 | 0.106261 | 0.401750 | 0.079* |
| H33C | 0.105422 | 0.083267 | 0.452311 | 0.079* |
| C34 | 0.2119 (15) | 0.1650 (6) | 0.5151 (4) | 0.060 (3) |
| H34A | 0.122910 | 0.141598 | 0.527733 | 0.091* |
| H34B | 0.208541 | 0.205985 | 0.528978 | 0.091* |
| H34C | 0.309717 | 0.145238 | 0.525017 | 0.091* |
| C35 | 0.1575 (11) | 0.5104 (4) | 0.2590 (3) | 0.035 (2) |
| H35 | 0.256420 | 0.508368 | 0.243038 | 0.043* |
| C36 | 0.0247 (11) | 0.5150 (4) | 0.2297 (3) | 0.040 (2) |
| H36 | 0.032768 | 0.515822 | 0.194525 | 0.048* |
| C37 | −0.1175 (11) | 0.5183 (5) | 0.2523 (4) | 0.041 (2) |
| H37 | −0.210136 | 0.521652 | 0.233001 | 0.049* |
| C38 | −0.1261 (11) | 0.5167 (4) | 0.3032 (4) | 0.039 (2) |
| H38 | −0.224616 | 0.519085 | 0.319403 | 0.047* |
| C39 | 0.0098 (9) | 0.5117 (4) | 0.3307 (3) | 0.0284 (18) |
| C40 | 0.0108 (9) | 0.5097 (4) | 0.3862 (3) | 0.0278 (18) |
| C41 | −0.1264 (10) | 0.5042 (4) | 0.4130 (3) | 0.0321 (19) |
| H41 | −0.224508 | 0.501035 | 0.396664 | 0.039* |
| C42 | −0.1172 (10) | 0.5036 (4) | 0.4646 (3) | 0.0318 (19) |
| H42 | −0.209570 | 0.499709 | 0.483986 | 0.038* |
| C43 | 0.0268 (10) | 0.5086 (4) | 0.4875 (3) | 0.0291 (18) |
| C44 | 0.1613 (10) | 0.5124 (4) | 0.4574 (3) | 0.0291 (19) |
| C45 | 0.3188 (10) | 0.5155 (4) | 0.4826 (3) | 0.036 (2) |
| H45A | 0.373290 | 0.553019 | 0.472336 | 0.043* |
| H45B | 0.383375 | 0.480620 | 0.472063 | 0.043* |
| C46 | 0.3010 (11) | 0.5148 (4) | 0.5391 (3) | 0.036 (2) |
| H46 | 0.400691 | 0.518064 | 0.558356 | 0.043* |
| C47 | 0.1712 (11) | 0.5602 (4) | 0.5540 (3) | 0.035 (2) |
| H47A | 0.162442 | 0.595626 | 0.531732 | 0.042* |
| H47B | 0.174588 | 0.572696 | 0.589298 | 0.042* |
| C48 | 0.0517 (10) | 0.5089 (4) | 0.5428 (3) | 0.033 (2) |
| H48 | −0.044342 | 0.507588 | 0.564008 | 0.040* |
| C49 | 0.1861 (12) | 0.4632 (4) | 0.5563 (3) | 0.034 (2) |
| C50 | 0.1942 (13) | 0.4021 (4) | 0.5297 (4) | 0.045 (2) |
| H50A | 0.292556 | 0.381758 | 0.538188 | 0.067* |
| H50B | 0.189308 | 0.408512 | 0.493756 | 0.067* |
| H50C | 0.105855 | 0.376814 | 0.540143 | 0.067* |
| C51 | 0.1961 (13) | 0.4523 (5) | 0.6124 (4) | 0.048 (3) |
| H51A | 0.107222 | 0.427388 | 0.622993 | 0.072* |
| H51B | 0.193296 | 0.491166 | 0.629808 | 0.072* |
| H51C | 0.293984 | 0.431247 | 0.620174 | 0.072* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Co1 | 0.0219 (7) | 0.0243 (7) | 0.0321 (7) | 0.0015 (5) | −0.0018 (6) | −0.0016 (6) |
| Co2 | 0.0223 (7) | 0.0276 (7) | 0.0317 (7) | 0.0009 (6) | −0.0020 (6) | 0.0015 (6) |
| Co3 | 0.0202 (7) | 0.0290 (7) | 0.0335 (7) | 0.0008 (6) | −0.0011 (6) | −0.0008 (6) |

| | | | | | | |
|---|---|---|---|---|---|---|
| Cl1 | 0.0492 (14) | 0.0341 (11) | 0.0416 (12) | 0.0167 (10) | −0.0021 (11) | 0.0008 (9) |
| Cl2 | 0.0325 (11) | 0.0299 (10) | 0.0382 (11) | −0.0058 (9) | 0.0089 (9) | −0.0055 (9) |
| Cl3 | 0.0336 (11) | 0.0342 (11) | 0.0404 (11) | −0.0040 (9) | 0.0092 (10) | −0.0076 (9) |
| Cl4 | 0.0370 (12) | 0.0346 (11) | 0.0402 (12) | 0.0098 (9) | −0.0051 (10) | 0.0038 (9) |
| Cl5 | 0.0469 (13) | 0.0316 (11) | 0.0463 (13) | 0.0114 (10) | 0.0015 (11) | 0.0020 (10) |
| Cl6 | 0.0375 (12) | 0.0360 (11) | 0.0460 (13) | −0.0144 (10) | −0.0018 (10) | −0.0004 (10) |
| O1 | 0.027 (3) | 0.040 (4) | 0.042 (4) | 0.001 (3) | 0.004 (3) | −0.006 (3) |
| N1 | 0.022 (4) | 0.029 (4) | 0.029 (4) | −0.004 (3) | −0.001 (3) | 0.002 (3) |
| N2 | 0.024 (4) | 0.023 (4) | 0.032 (4) | −0.002 (3) | 0.000 (3) | −0.001 (3) |
| N3 | 0.031 (4) | 0.023 (3) | 0.030 (4) | 0.002 (3) | 0.001 (3) | 0.001 (3) |
| N4 | 0.024 (4) | 0.027 (4) | 0.031 (4) | 0.002 (3) | 0.001 (3) | 0.004 (3) |
| N5 | 0.027 (4) | 0.026 (4) | 0.033 (4) | 0.000 (3) | 0.003 (3) | 0.001 (3) |
| N6 | 0.012 (3) | 0.029 (4) | 0.033 (4) | 0.004 (3) | 0.003 (3) | 0.000 (3) |
| C1 | 0.031 (5) | 0.037 (5) | 0.028 (5) | −0.005 (4) | −0.003 (4) | −0.002 (4) |
| C2 | 0.052 (6) | 0.044 (5) | 0.027 (5) | −0.008 (5) | −0.004 (5) | 0.001 (4) |
| C3 | 0.037 (5) | 0.027 (4) | 0.037 (5) | −0.005 (4) | −0.017 (4) | −0.005 (4) |
| C4 | 0.023 (4) | 0.034 (5) | 0.039 (5) | −0.003 (4) | −0.010 (4) | −0.005 (4) |
| C5 | 0.027 (4) | 0.017 (4) | 0.033 (4) | −0.001 (3) | −0.003 (4) | −0.002 (3) |
| C6 | 0.010 (4) | 0.022 (4) | 0.035 (4) | −0.001 (3) | −0.002 (3) | 0.000 (3) |
| C7 | 0.020 (4) | 0.031 (4) | 0.039 (5) | −0.001 (4) | −0.006 (4) | −0.003 (4) |
| C8 | 0.016 (4) | 0.035 (5) | 0.043 (5) | −0.006 (4) | 0.001 (4) | −0.003 (4) |
| C9 | 0.025 (4) | 0.029 (4) | 0.034 (5) | −0.002 (4) | −0.001 (4) | −0.003 (4) |
| C10 | 0.017 (4) | 0.027 (4) | 0.031 (4) | 0.009 (3) | 0.000 (3) | −0.002 (4) |
| C11 | 0.024 (4) | 0.042 (5) | 0.029 (4) | 0.002 (4) | −0.001 (4) | 0.003 (4) |
| C12 | 0.030 (5) | 0.034 (5) | 0.034 (5) | 0.011 (4) | −0.009 (4) | 0.005 (4) |
| C13 | 0.034 (5) | 0.040 (5) | 0.036 (5) | 0.002 (4) | 0.005 (4) | 0.013 (4) |
| C14 | 0.025 (5) | 0.039 (5) | 0.035 (5) | 0.005 (4) | 0.006 (4) | 0.003 (4) |
| C15 | 0.034 (5) | 0.037 (5) | 0.030 (4) | 0.008 (4) | −0.007 (4) | 0.003 (4) |
| C16 | 0.038 (5) | 0.030 (5) | 0.039 (5) | −0.002 (4) | −0.004 (4) | −0.001 (4) |
| C17 | 0.039 (6) | 0.057 (7) | 0.036 (5) | 0.008 (5) | −0.003 (4) | 0.004 (5) |
| C18 | 0.025 (4) | 0.028 (4) | 0.034 (5) | −0.005 (4) | −0.001 (4) | 0.001 (4) |
| C19 | 0.030 (5) | 0.035 (5) | 0.037 (5) | −0.006 (4) | −0.002 (4) | −0.001 (4) |
| C20 | 0.026 (4) | 0.029 (5) | 0.045 (5) | −0.005 (4) | −0.011 (4) | 0.003 (4) |
| C21 | 0.017 (4) | 0.043 (5) | 0.036 (5) | −0.004 (4) | 0.000 (4) | 0.005 (4) |
| C22 | 0.018 (4) | 0.029 (4) | 0.032 (4) | −0.001 (3) | 0.003 (4) | 0.007 (3) |
| C23 | 0.017 (4) | 0.031 (4) | 0.035 (5) | −0.001 (3) | −0.005 (4) | 0.004 (4) |
| C24 | 0.021 (4) | 0.037 (5) | 0.044 (5) | 0.004 (4) | 0.005 (4) | 0.000 (4) |
| C25 | 0.029 (5) | 0.040 (5) | 0.044 (5) | 0.003 (4) | 0.009 (4) | 0.004 (4) |
| C26 | 0.031 (5) | 0.025 (4) | 0.039 (5) | 0.004 (4) | 0.000 (4) | 0.008 (4) |
| C27 | 0.022 (4) | 0.032 (4) | 0.028 (4) | 0.003 (4) | 0.002 (4) | 0.001 (4) |
| C28 | 0.026 (5) | 0.057 (6) | 0.035 (5) | 0.000 (4) | −0.006 (4) | 0.002 (5) |
| C29 | 0.041 (6) | 0.047 (6) | 0.031 (5) | 0.004 (5) | −0.002 (4) | 0.000 (4) |
| C30 | 0.055 (7) | 0.042 (6) | 0.040 (6) | 0.000 (5) | −0.001 (5) | 0.000 (5) |
| C31 | 0.045 (6) | 0.038 (5) | 0.036 (5) | 0.012 (4) | 0.005 (4) | −0.002 (4) |
| C32 | 0.045 (6) | 0.044 (6) | 0.037 (5) | 0.005 (5) | 0.004 (5) | 0.007 (5) |
| C33 | 0.067 (8) | 0.038 (6) | 0.052 (6) | 0.013 (5) | −0.010 (6) | 0.011 (5) |
| C34 | 0.063 (8) | 0.078 (8) | 0.041 (6) | 0.004 (7) | 0.000 (6) | 0.015 (6) |
| C35 | 0.027 (5) | 0.046 (6) | 0.033 (5) | 0.004 (4) | 0.004 (4) | 0.003 (4) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C36 | 0.037 (5) | 0.053 (6) | 0.029 (5) | 0.003 (5) | −0.005 (4) | 0.001 (4) |
| C37 | 0.026 (5) | 0.060 (6) | 0.037 (5) | −0.003 (4) | −0.004 (4) | 0.000 (5) |
| C38 | 0.021 (5) | 0.053 (6) | 0.044 (5) | 0.000 (4) | −0.002 (4) | 0.005 (4) |
| C39 | 0.018 (4) | 0.029 (4) | 0.038 (5) | 0.005 (3) | −0.001 (4) | 0.003 (4) |
| C40 | 0.020 (4) | 0.025 (4) | 0.038 (5) | 0.004 (3) | 0.002 (4) | 0.000 (4) |
| C41 | 0.023 (4) | 0.032 (5) | 0.042 (5) | 0.002 (4) | 0.002 (4) | 0.000 (4) |
| C42 | 0.024 (4) | 0.028 (4) | 0.043 (5) | −0.004 (4) | 0.014 (4) | −0.004 (4) |
| C43 | 0.028 (4) | 0.024 (4) | 0.036 (5) | −0.003 (4) | 0.009 (4) | −0.002 (4) |
| C44 | 0.027 (4) | 0.027 (4) | 0.033 (5) | 0.003 (4) | 0.002 (4) | 0.001 (4) |
| C45 | 0.024 (5) | 0.045 (5) | 0.038 (5) | −0.003 (4) | −0.001 (4) | −0.001 (4) |
| C46 | 0.040 (5) | 0.035 (5) | 0.033 (5) | −0.003 (4) | 0.001 (4) | 0.000 (4) |
| C47 | 0.035 (5) | 0.029 (4) | 0.040 (5) | −0.006 (4) | 0.003 (4) | −0.010 (4) |
| C48 | 0.027 (5) | 0.035 (5) | 0.037 (5) | −0.007 (4) | 0.005 (4) | −0.007 (4) |
| C49 | 0.044 (6) | 0.024 (4) | 0.033 (5) | −0.001 (4) | 0.002 (4) | −0.002 (4) |
| C50 | 0.055 (7) | 0.027 (5) | 0.052 (6) | 0.004 (4) | −0.002 (5) | −0.008 (4) |
| C51 | 0.060 (7) | 0.047 (6) | 0.037 (5) | −0.005 (5) | 0.000 (5) | −0.001 (5) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Co1—Cl1 | 2.288 (2) | C20—H20 | 0.9500 |
| Co1—Cl2 | 2.339 (2) | C20—C21 | 1.379 (12) |
| Co1—Cl3 | 2.445 (2) | C21—H21 | 0.9500 |
| Co1—N1 | 2.066 (7) | C21—C22 | 1.382 (11) |
| Co1—N2 | 2.151 (7) | C22—C23 | 1.486 (11) |
| Co2—Cl2 | 2.509 (2) | C23—C24 | 1.379 (11) |
| Co2—Cl3 | 2.316 (2) | C24—H24 | 0.9500 |
| Co2—Cl4 | 2.284 (2) | C24—C25 | 1.392 (12) |
| Co2—N3 | 2.037 (7) | C25—H25 | 0.9500 |
| Co2—N4 | 2.195 (7) | C25—C26 | 1.374 (12) |
| Co3—Cl5 | 2.286 (3) | C26—C27 | 1.411 (11) |
| Co3—Cl6 | 2.291 (2) | C26—C31 | 1.501 (12) |
| Co3—O1 | 2.160 (6) | C27—C28 | 1.508 (12) |
| Co3—N5 | 2.089 (7) | C28—H28A | 0.9900 |
| Co3—N6 | 2.178 (6) | C28—H28B | 0.9900 |
| O1—H1A | 0.84 (10) | C28—C29 | 1.531 (12) |
| O1—H1B | 0.87 (10) | C29—H29 | 1.0000 |
| N1—C1 | 1.338 (11) | C29—C30 | 1.549 (14) |
| N1—C5 | 1.356 (10) | C29—C32 | 1.553 (14) |
| N2—C6 | 1.373 (10) | C30—H30A | 0.9900 |
| N2—C10 | 1.332 (10) | C30—H30B | 0.9900 |
| N3—C18 | 1.347 (10) | C30—C31 | 1.555 (14) |
| N3—C22 | 1.345 (10) | C31—H31 | 1.0000 |
| N4—C23 | 1.365 (10) | C31—C32 | 1.581 (13) |
| N4—C27 | 1.330 (10) | C32—C33 | 1.519 (14) |
| N5—C35 | 1.334 (10) | C32—C34 | 1.525 (13) |
| N5—C39 | 1.366 (10) | C33—H33A | 0.9800 |
| N6—C40 | 1.350 (10) | C33—H33B | 0.9800 |
| N6—C44 | 1.333 (10) | C33—H33C | 0.9800 |

| | | | |
|---|---|---|---|
| C1—H1 | 0.9500 | C34—H34A | 0.9800 |
| C1—C2 | 1.385 (13) | C34—H34B | 0.9800 |
| C2—H2 | 0.9500 | C34—H34C | 0.9800 |
| C2—C3 | 1.387 (14) | C35—H35 | 0.9500 |
| C3—H3 | 0.9500 | C35—C36 | 1.386 (13) |
| C3—C4 | 1.375 (12) | C36—H36 | 0.9500 |
| C4—H4 | 0.9500 | C36—C37 | 1.361 (13) |
| C4—C5 | 1.399 (12) | C37—H37 | 0.9500 |
| C5—C6 | 1.469 (11) | C37—C38 | 1.374 (13) |
| C6—C7 | 1.364 (11) | C38—H38 | 0.9500 |
| C7—H7 | 0.9500 | C38—C39 | 1.381 (12) |
| C7—C8 | 1.391 (12) | C39—C40 | 1.496 (11) |
| C8—H8 | 0.9500 | C40—C41 | 1.383 (11) |
| C8—C9 | 1.383 (12) | C41—H41 | 0.9500 |
| C9—C10 | 1.411 (11) | C41—C42 | 1.392 (12) |
| C9—C14 | 1.506 (12) | C42—H42 | 0.9500 |
| C10—C11 | 1.509 (11) | C42—C43 | 1.381 (12) |
| C11—H11A | 0.9900 | C43—C44 | 1.410 (11) |
| C11—H11B | 0.9900 | C43—C48 | 1.507 (12) |
| C11—C12 | 1.524 (12) | C44—C45 | 1.509 (12) |
| C12—H12 | 1.0000 | C45—H45A | 0.9900 |
| C12—C13 | 1.549 (13) | C45—H45B | 0.9900 |
| C12—C15 | 1.571 (12) | C45—C46 | 1.531 (12) |
| C13—H13A | 0.9900 | C46—H46 | 1.0000 |
| C13—H13B | 0.9900 | C46—C47 | 1.548 (13) |
| C13—C14 | 1.560 (12) | C46—C49 | 1.575 (13) |
| C14—H14 | 1.0000 | C47—H47A | 0.9900 |
| C14—C15 | 1.573 (13) | C47—H47B | 0.9900 |
| C15—C16 | 1.538 (12) | C47—C48 | 1.556 (11) |
| C15—C17 | 1.527 (12) | C48—H48 | 1.0000 |
| C16—H16A | 0.9800 | C48—C49 | 1.573 (13) |
| C16—H16B | 0.9800 | C49—C50 | 1.530 (12) |
| C16—H16C | 0.9800 | C49—C51 | 1.531 (13) |
| C17—H17A | 0.9800 | C50—H50A | 0.9800 |
| C17—H17B | 0.9800 | C50—H50B | 0.9800 |
| C17—H17C | 0.9800 | C50—H50C | 0.9800 |
| C18—H18 | 0.9500 | C51—H51A | 0.9800 |
| C18—C19 | 1.382 (12) | C51—H51B | 0.9800 |
| C19—H19 | 0.9500 | C51—H51C | 0.9800 |
| C19—C20 | 1.392 (12) | | |

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| Cl1—Co1—Cl2 | 124.40 (10) | C20—C21—C22 | 120.0 (8) |
| Cl1—Co1—Cl3 | 96.21 (9) | C22—C21—H21 | 120.0 |
| Cl2—Co1—Cl3 | 84.24 (8) | N3—C22—C21 | 122.1 (8) |
| N1—Co1—Cl1 | 112.2 (2) | N3—C22—C23 | 115.6 (7) |
| N1—Co1—Cl2 | 123.4 (2) | C21—C22—C23 | 122.3 (7) |
| N1—Co1—Cl3 | 92.4 (2) | N4—C23—C22 | 115.0 (7) |
| N1—Co1—N2 | 78.5 (3) | N4—C23—C24 | 121.6 (7) |

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| N2—Co1—Cl1 | 97.2 (2) | C24—C23—C22 | 123.3 (7) |
| N2—Co1—Cl2 | 91.72 (19) | C23—C24—H24 | 120.4 |
| N2—Co1—Cl3 | 166.0 (2) | C23—C24—C25 | 119.1 (8) |
| Cl3—Co2—Cl2 | 83.28 (8) | C25—C24—H24 | 120.4 |
| Cl4—Co2—Cl2 | 98.44 (9) | C24—C25—H25 | 120.1 |
| Cl4—Co2—Cl3 | 126.39 (10) | C26—C25—C24 | 119.8 (8) |
| N3—Co2—Cl2 | 96.4 (2) | C26—C25—H25 | 120.1 |
| N3—Co2—Cl3 | 119.8 (2) | C25—C26—C27 | 118.3 (8) |
| N3—Co2—Cl4 | 113.3 (2) | C25—C26—C31 | 125.2 (8) |
| N3—Co2—N4 | 77.5 (3) | C27—C26—C31 | 116.5 (8) |
| N4—Co2—Cl2 | 164.91 (19) | N4—C27—C26 | 122.1 (7) |
| N4—Co2—Cl3 | 87.81 (19) | N4—C27—C28 | 120.0 (7) |
| N4—Co2—Cl4 | 96.65 (19) | C26—C27—C28 | 117.8 (7) |
| Cl5—Co3—Cl6 | 120.73 (10) | C27—C28—H28A | 109.5 |
| O1—Co3—Cl5 | 94.9 (2) | C27—C28—H28B | 109.5 |
| O1—Co3—Cl6 | 87.4 (2) | C27—C28—C29 | 110.9 (8) |
| O1—Co3—N6 | 169.3 (3) | H28A—C28—H28B | 108.0 |
| N5—Co3—Cl5 | 116.7 (2) | C29—C28—H28A | 109.5 |
| N5—Co3—Cl6 | 122.4 (2) | C29—C28—H28B | 109.5 |
| N5—Co3—O1 | 91.6 (3) | C28—C29—H29 | 114.9 |
| N5—Co3—N6 | 77.8 (3) | C28—C29—C30 | 108.6 (8) |
| N6—Co3—Cl5 | 91.49 (19) | C28—C29—C32 | 112.4 (8) |
| N6—Co3—Cl6 | 96.71 (19) | C30—C29—H29 | 114.9 |
| Co1—Cl2—Co2 | 94.77 (8) | C30—C29—C32 | 88.3 (8) |
| Co2—Cl3—Co1 | 97.09 (9) | C32—C29—H29 | 114.9 |
| Co3—O1—H1A | 121 (7) | C29—C30—H30A | 114.4 |
| Co3—O1—H1B | 109 (7) | C29—C30—H30B | 114.4 |
| H1A—O1—H1B | 111 (9) | C29—C30—C31 | 85.6 (7) |
| C1—N1—Co1 | 124.3 (6) | H30A—C30—H30B | 111.5 |
| C1—N1—C5 | 119.9 (7) | C31—C30—H30A | 114.4 |
| C5—N1—Co1 | 115.7 (5) | C31—C30—H30B | 114.4 |
| C6—N2—Co1 | 112.6 (5) | C26—C31—C30 | 107.6 (8) |
| C10—N2—Co1 | 128.3 (5) | C26—C31—H31 | 116.2 |
| C10—N2—C6 | 118.7 (7) | C26—C31—C32 | 109.9 (7) |
| C18—N3—Co2 | 123.0 (6) | C30—C31—H31 | 116.2 |
| C22—N3—Co2 | 118.5 (5) | C30—C31—C32 | 87.1 (7) |
| C22—N3—C18 | 118.5 (7) | C32—C31—H31 | 116.2 |
| C23—N4—Co2 | 112.3 (5) | C29—C32—C31 | 84.6 (7) |
| C27—N4—Co2 | 127.7 (5) | C33—C32—C29 | 119.2 (9) |
| C27—N4—C23 | 119.1 (7) | C33—C32—C31 | 117.4 (9) |
| C35—N5—Co3 | 126.2 (6) | C33—C32—C34 | 109.0 (9) |
| C35—N5—C39 | 117.4 (7) | C34—C32—C29 | 112.6 (9) |
| C39—N5—Co3 | 116.4 (5) | C34—C32—C31 | 112.4 (8) |
| C40—N6—Co3 | 113.6 (5) | C32—C33—H33A | 109.5 |
| C44—N6—Co3 | 127.1 (6) | C32—C33—H33B | 109.5 |
| C44—N6—C40 | 118.9 (7) | C32—C33—H33C | 109.5 |
| N1—C1—H1 | 119.3 | H33A—C33—H33B | 109.5 |
| N1—C1—C2 | 121.4 (8) | H33A—C33—H33C | 109.5 |

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| C2—C1—H1 | 119.3 | H33B—C33—H33C | 109.5 |
| C1—C2—H2 | 120.1 | C32—C34—H34A | 109.5 |
| C1—C2—C3 | 119.9 (9) | C32—C34—H34B | 109.5 |
| C3—C2—H2 | 120.1 | C32—C34—H34C | 109.5 |
| C2—C3—H3 | 120.8 | H34A—C34—H34B | 109.5 |
| C4—C3—C2 | 118.3 (8) | H34A—C34—H34C | 109.5 |
| C4—C3—H3 | 120.8 | H34B—C34—H34C | 109.5 |
| C3—C4—H4 | 119.9 | N5—C35—H35 | 118.3 |
| C3—C4—C5 | 120.1 (8) | N5—C35—C36 | 123.4 (8) |
| C5—C4—H4 | 119.9 | C36—C35—H35 | 118.3 |
| N1—C5—C4 | 120.3 (8) | C35—C36—H36 | 120.7 |
| N1—C5—C6 | 116.7 (7) | C37—C36—C35 | 118.7 (8) |
| C4—C5—C6 | 123.0 (8) | C37—C36—H36 | 120.7 |
| N2—C6—C5 | 115.2 (7) | C36—C37—H37 | 120.2 |
| C7—C6—N2 | 121.6 (7) | C36—C37—C38 | 119.5 (9) |
| C7—C6—C5 | 123.2 (7) | C38—C37—H37 | 120.2 |
| C6—C7—H7 | 119.9 | C37—C38—H38 | 120.3 |
| C6—C7—C8 | 120.3 (8) | C37—C38—C39 | 119.5 (8) |
| C8—C7—H7 | 119.9 | C39—C38—H38 | 120.3 |
| C7—C8—H8 | 120.8 | N5—C39—C38 | 121.6 (8) |
| C9—C8—C7 | 118.5 (8) | N5—C39—C40 | 115.5 (7) |
| C9—C8—H8 | 120.8 | C38—C39—C40 | 122.8 (8) |
| C8—C9—C10 | 118.9 (8) | N6—C40—C39 | 116.0 (7) |
| C8—C9—C14 | 124.1 (8) | N6—C40—C41 | 122.6 (8) |
| C10—C9—C14 | 117.0 (7) | C41—C40—C39 | 121.4 (8) |
| N2—C10—C9 | 122.0 (7) | C40—C41—H41 | 120.8 |
| N2—C10—C11 | 120.1 (7) | C40—C41—C42 | 118.4 (8) |
| C9—C10—C11 | 118.0 (7) | C42—C41—H41 | 120.8 |
| C10—C11—H11A | 109.3 | C41—C42—H42 | 120.2 |
| C10—C11—H11B | 109.3 | C43—C42—C41 | 119.6 (8) |
| C10—C11—C12 | 111.5 (7) | C43—C42—H42 | 120.2 |
| H11A—C11—H11B | 108.0 | C42—C43—C44 | 118.4 (8) |
| C12—C11—H11A | 109.3 | C42—C43—C48 | 124.6 (7) |
| C12—C11—H11B | 109.3 | C44—C43—C48 | 117.0 (8) |
| C11—C12—H12 | 115.5 | N6—C44—C43 | 122.0 (8) |
| C11—C12—C13 | 109.5 (7) | N6—C44—C45 | 119.9 (7) |
| C11—C12—C15 | 110.9 (7) | C43—C44—C45 | 118.1 (7) |
| C13—C12—H12 | 115.5 | C44—C45—H45A | 109.4 |
| C13—C12—C15 | 86.4 (7) | C44—C45—H45B | 109.4 |
| C15—C12—H12 | 115.5 | C44—C45—C46 | 111.0 (7) |
| C12—C13—H13A | 114.1 | H45A—C45—H45B | 108.0 |
| C12—C13—H13B | 114.1 | C46—C45—H45A | 109.4 |
| C12—C13—C14 | 87.3 (6) | C46—C45—H45B | 109.4 |
| H13A—C13—H13B | 111.3 | C45—C46—H46 | 115.5 |
| C14—C13—H13A | 114.1 | C45—C46—C47 | 108.8 (8) |
| C14—C13—H13B | 114.1 | C45—C46—C49 | 111.2 (7) |
| C9—C14—C13 | 106.7 (7) | C47—C46—H46 | 115.5 |
| C9—C14—H14 | 116.7 | C47—C46—C49 | 86.9 (7) |

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| C9—C14—C15 | 109.8 (7) | C49—C46—H46 | 115.5 |
| C13—C14—H14 | 116.7 | C46—C47—H47A | 114.1 |
| C13—C14—C15 | 86.0 (7) | C46—C47—H47B | 114.1 |
| C15—C14—H14 | 116.7 | C46—C47—C48 | 87.1 (7) |
| C12—C15—C14 | 86.0 (7) | H47A—C47—H47B | 111.3 |
| C16—C15—C12 | 118.5 (8) | C48—C47—H47A | 114.1 |
| C16—C15—C14 | 118.5 (7) | C48—C47—H47B | 114.1 |
| C17—C15—C12 | 112.0 (7) | C43—C48—C47 | 106.8 (7) |
| C17—C15—C14 | 111.8 (8) | C43—C48—H48 | 116.7 |
| C17—C15—C16 | 108.5 (8) | C43—C48—C49 | 109.2 (7) |
| C15—C16—H16A | 109.5 | C47—C48—H48 | 116.7 |
| C15—C16—H16B | 109.5 | C47—C48—C49 | 86.7 (7) |
| C15—C16—H16C | 109.5 | C49—C48—H48 | 116.7 |
| H16A—C16—H16B | 109.5 | C48—C49—C46 | 85.5 (6) |
| H16A—C16—H16C | 109.5 | C50—C49—C46 | 118.3 (8) |
| H16B—C16—H16C | 109.5 | C50—C49—C48 | 119.4 (8) |
| C15—C17—H17A | 109.5 | C50—C49—C51 | 108.7 (8) |
| C15—C17—H17B | 109.5 | C51—C49—C46 | 111.7 (8) |
| C15—C17—H17C | 109.5 | C51—C49—C48 | 111.7 (8) |
| H17A—C17—H17B | 109.5 | C49—C50—H50A | 109.5 |
| H17A—C17—H17C | 109.5 | C49—C50—H50B | 109.5 |
| H17B—C17—H17C | 109.5 | C49—C50—H50C | 109.5 |
| N3—C18—H18 | 119.1 | H50A—C50—H50B | 109.5 |
| N3—C18—C19 | 121.7 (8) | H50A—C50—H50C | 109.5 |
| C19—C18—H18 | 119.1 | H50B—C50—H50C | 109.5 |
| C18—C19—H19 | 120.0 | C49—C51—H51A | 109.5 |
| C18—C19—C20 | 120.0 (8) | C49—C51—H51B | 109.5 |
| C20—C19—H19 | 120.0 | C49—C51—H51C | 109.5 |
| C19—C20—H20 | 121.2 | H51A—C51—H51B | 109.5 |
| C21—C20—C19 | 117.6 (8) | H51A—C51—H51C | 109.5 |
| C21—C20—H20 | 121.2 | H51B—C51—H51C | 109.5 |
| C20—C21—H21 | 120.0 | | |

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| Co1—N1—C1—C2 | −177.8 (7) | C20—C21—C22—N3 | −0.9 (13) |
| Co1—N1—C5—C4 | 177.8 (6) | C20—C21—C22—C23 | −179.6 (8) |
| Co1—N1—C5—C6 | −0.4 (9) | C21—C22—C23—N4 | 170.0 (8) |
| Co1—N2—C6—C5 | −12.1 (8) | C21—C22—C23—C24 | −6.6 (13) |
| Co1—N2—C6—C7 | 167.6 (6) | C22—N3—C18—C19 | −0.4 (12) |
| Co1—N2—C10—C9 | −168.1 (6) | C22—C23—C24—C25 | 178.5 (8) |
| Co1—N2—C10—C11 | 13.1 (11) | C23—N4—C27—C26 | 0.4 (12) |
| Co2—N3—C18—C19 | 177.6 (6) | C23—N4—C27—C28 | 177.9 (8) |
| Co2—N3—C22—C21 | −178.2 (6) | C23—C24—C25—C26 | −1.8 (14) |
| Co2—N3—C22—C23 | 0.6 (9) | C24—C25—C26—C27 | 0.9 (13) |
| Co2—N4—C23—C22 | 11.9 (9) | C24—C25—C26—C31 | −178.1 (8) |
| Co2—N4—C23—C24 | −171.5 (7) | C25—C26—C27—N4 | −0.1 (13) |
| Co2—N4—C27—C26 | 168.7 (6) | C25—C26—C27—C28 | −177.7 (8) |
| Co2—N4—C27—C28 | −13.8 (12) | C25—C26—C31—C30 | −135.5 (9) |
| Co3—N5—C35—C36 | −178.8 (7) | C25—C26—C31—C32 | 131.2 (9) |

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| Co3—N5—C39—C38 | 178.5 (7) | C26—C27—C28—C29 | −0.5 (12) |
| Co3—N5—C39—C40 | −1.0 (9) | C26—C31—C32—C29 | 79.7 (8) |
| Co3—N6—C40—C39 | −9.5 (9) | C26—C31—C32—C33 | −40.5 (12) |
| Co3—N6—C40—C41 | 169.1 (7) | C26—C31—C32—C34 | −168.0 (9) |
| Co3—N6—C44—C43 | −169.7 (6) | C27—N4—C23—C22 | −178.0 (7) |
| Co3—N6—C44—C45 | 9.0 (11) | C27—N4—C23—C24 | −1.4 (12) |
| N1—C1—C2—C3 | −0.8 (14) | C27—C26—C31—C30 | 45.5 (10) |
| N1—C5—C6—N2 | 8.6 (10) | C27—C26—C31—C32 | −47.8 (11) |
| N1—C5—C6—C7 | −171.1 (8) | C27—C28—C29—C30 | −48.1 (11) |
| N2—C6—C7—C8 | 3.0 (13) | C27—C28—C29—C32 | 47.9 (11) |
| N2—C10—C11—C12 | 177.4 (7) | C28—C29—C30—C31 | 84.5 (8) |
| N3—C18—C19—C20 | 1.9 (13) | C28—C29—C32—C31 | −81.3 (9) |
| N3—C22—C23—N4 | −8.8 (10) | C28—C29—C32—C33 | 37.2 (12) |
| N3—C22—C23—C24 | 174.6 (8) | C28—C29—C32—C34 | 166.6 (9) |
| N4—C23—C24—C25 | 2.1 (13) | C29—C30—C31—C26 | −81.9 (8) |
| N4—C27—C28—C29 | −178.1 (8) | C29—C30—C31—C32 | 28.0 (7) |
| N5—C35—C36—C37 | 0.3 (15) | C30—C29—C32—C31 | 28.1 (7) |
| N5—C39—C40—N6 | 7.2 (11) | C30—C29—C32—C33 | 146.5 (9) |
| N5—C39—C40—C41 | −171.4 (8) | C30—C29—C32—C34 | −84.0 (9) |
| N6—C40—C41—C42 | 2.5 (12) | C30—C31—C32—C29 | −28.0 (7) |
| N6—C44—C45—C46 | −179.6 (7) | C30—C31—C32—C33 | −148.1 (9) |
| C1—N1—C5—C4 | −0.5 (12) | C30—C31—C32—C34 | 84.3 (10) |
| C1—N1—C5—C6 | −178.6 (7) | C31—C26—C27—N4 | 178.9 (8) |
| C1—C2—C3—C4 | 1.5 (13) | C31—C26—C27—C28 | 1.4 (11) |
| C2—C3—C4—C5 | −1.7 (12) | C32—C29—C30—C31 | −28.6 (7) |
| C3—C4—C5—N1 | 1.3 (12) | C35—N5—C39—C38 | −0.6 (12) |
| C3—C4—C5—C6 | 179.3 (8) | C35—N5—C39—C40 | 179.9 (7) |
| C4—C5—C6—N2 | −169.4 (7) | C35—C36—C37—C38 | −0.3 (15) |
| C4—C5—C6—C7 | 10.8 (12) | C36—C37—C38—C39 | −0.1 (15) |
| C5—N1—C1—C2 | 0.3 (13) | C37—C38—C39—N5 | 0.6 (14) |
| C5—C6—C7—C8 | −177.3 (7) | C37—C38—C39—C40 | −180.0 (9) |
| C6—N2—C10—C9 | 3.4 (12) | C38—C39—C40—N6 | −172.3 (8) |
| C6—N2—C10—C11 | −175.4 (7) | C38—C39—C40—C41 | 9.2 (13) |
| C6—C7—C8—C9 | 0.9 (13) | C39—N5—C35—C36 | 0.1 (14) |
| C7—C8—C9—C10 | −2.6 (12) | C39—C40—C41—C42 | −179.0 (7) |
| C7—C8—C9—C14 | 176.0 (8) | C40—N6—C44—C43 | 1.8 (12) |
| C8—C9—C10—N2 | 0.4 (12) | C40—N6—C44—C45 | −179.6 (8) |
| C8—C9—C10—C11 | 179.2 (8) | C40—C41—C42—C43 | 0.3 (12) |
| C8—C9—C14—C13 | −132.5 (9) | C41—C42—C43—C44 | −1.9 (12) |
| C8—C9—C14—C15 | 135.7 (9) | C41—C42—C43—C48 | 179.5 (8) |
| C9—C10—C11—C12 | −1.4 (11) | C42—C43—C44—N6 | 0.9 (12) |
| C9—C14—C15—C12 | 78.5 (7) | C42—C43—C44—C45 | −177.8 (8) |
| C9—C14—C15—C16 | −42.0 (10) | C42—C43—C48—C47 | −135.9 (9) |
| C9—C14—C15—C17 | −169.3 (7) | C42—C43—C48—C49 | 131.6 (9) |
| C10—N2—C6—C5 | 175.1 (7) | C43—C44—C45—C46 | −0.9 (11) |
| C10—N2—C6—C7 | −5.1 (12) | C43—C48—C49—C46 | 79.3 (7) |
| C10—C9—C14—C13 | 46.1 (10) | C43—C48—C49—C50 | −40.7 (11) |
| C10—C9—C14—C15 | −45.6 (10) | C43—C48—C49—C51 | −169.1 (7) |

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| C10—C11—C12—C13 | −45.3 (10) | C44—N6—C40—C39 | 177.9 (7) |
| C10—C11—C12—C15 | 48.3 (10) | C44—N6—C40—C41 | −3.5 (12) |
| C11—C12—C13—C14 | 82.7 (8) | C44—C43—C48—C47 | 45.4 (10) |
| C11—C12—C15—C14 | −81.4 (8) | C44—C43—C48—C49 | −47.0 (10) |
| C11—C12—C15—C16 | 39.0 (11) | C44—C45—C46—C47 | −46.5 (10) |
| C11—C12—C15—C17 | 166.7 (8) | C44—C45—C46—C49 | 47.6 (10) |
| C12—C13—C14—C9 | −81.3 (8) | C45—C46—C47—C48 | 83.7 (8) |
| C12—C13—C14—C15 | 28.2 (6) | C45—C46—C49—C48 | −81.6 (8) |
| C13—C12—C15—C14 | 28.0 (6) | C45—C46—C49—C50 | 39.5 (12) |
| C13—C12—C15—C16 | 148.5 (8) | C45—C46—C49—C51 | 166.8 (8) |
| C13—C12—C15—C17 | −83.9 (8) | C46—C47—C48—C43 | −81.3 (8) |
| C13—C14—C15—C12 | −27.8 (6) | C46—C47—C48—C49 | 27.8 (6) |
| C13—C14—C15—C16 | −148.3 (8) | C47—C46—C49—C48 | 27.4 (6) |
| C13—C14—C15—C17 | 84.3 (8) | C47—C46—C49—C50 | 148.5 (9) |
| C14—C9—C10—N2 | −178.3 (8) | C47—C46—C49—C51 | −84.2 (8) |
| C14—C9—C10—C11 | 0.5 (11) | C47—C48—C49—C46 | −27.3 (6) |
| C15—C12—C13—C14 | −28.2 (6) | C47—C48—C49—C50 | −147.3 (8) |
| C18—N3—C22—C21 | −0.1 (12) | C47—C48—C49—C51 | 84.3 (8) |
| C18—N3—C22—C23 | 178.7 (7) | C48—C43—C44—N6 | 179.6 (7) |
| C18—C19—C20—C21 | −2.8 (13) | C48—C43—C44—C45 | 0.9 (11) |
| C19—C20—C21—C22 | 2.3 (13) | C49—C46—C47—C48 | −27.7 (6) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—H1*A*···Cl1 | 0.84 (10) | 2.37 (10) | 3.194 (7) | 166 (9) |
| O1—H1*B*···Cl4$^{i}$ | 0.87 (10) | 2.43 (10) | 3.260 (7) | 161 (9) |

Symmetry code: (i) −*x*+1, *y*+1/2, −*z*+1/2.