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ISSN 2414-3146

# Chloridobis(ethane-1,2-diamine)(4-fluoroaniline)-cobalt(III) dichloride monohydrate

A. SubbiahPandi,[a]* Y. AaminaNaaz,[a] C. Maharaja Mahalakshmi[b] and K. Anbalagan[c]

[a]Department of Physics, Presidency College (Autonomous), Chennai 600 005, India, [b]Department of Chemistry, Chellammal Womens College, Chennai 600 032, India, and [c]Department of Chemistry, Pondicherry University, Pondicherry 605 014, India. *Correspondence e-mail: aspandian59@gmail.com

Received 23 January 2019
Accepted 7 March 2019

Edited by M. Weil, Vienna University of Technology, Austria

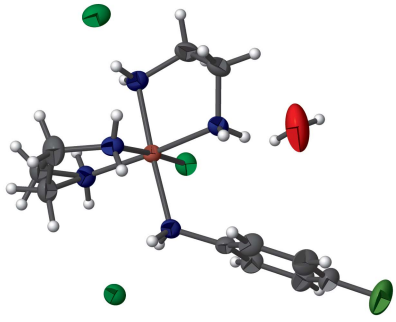
**Keywords:** crystal structure; ethane-1,2-diamine; fluoroaniline ligand; cobalt(III) complex; hydrogen bonding; C—H···Cl interactions.

**CCDC reference**: 785047

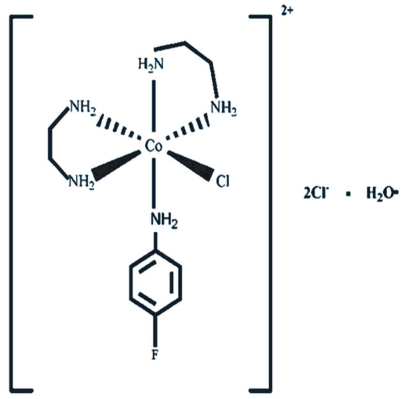
**Structural data**: full structural data are available from iucrdata.iucr.org

The hydrated title salt, $[CoCl(C_6H_6FN)(C_2H_8N_2)_2]Cl_2\cdot H_2O$, comprises of one chloridobis(ethane-1,2-diamine)(4-fluoroaniline)cobalt(III) cation, two chloride counter-anions and a water molecule of crystallization. The $Co^{III}$ ion has a distorted octahedral environment and is surrounded by four N atoms in the equatorial plane, with a fifth N atom and one $Cl^-$ ligand occupying the axial positions. One of the methylene C groups in one of the ethane-1,2-diamine ligands is disordered over two set of sites in a 0.832 (10):0.168 (10) ratio. In the crystal, the complex cation, the two counter-anions and the water molecule of crystallization are linked *via* N—H···Cl, O—H···Cl and C—H···Cl hydrogen bonds, generating rings with $R_4^2(8)$, $R_2^1(6)$, $R_4^2(10)$ and $R_2^2(6)$ graph-set motifs within a three-dimensional network.

**3D view**



**Chemical scheme**




## Structure description

As a result of the excellent coordination ability of ligands with N-donating groups, such as simple amines (Mitzi, 1996; Deeth *et al.*, 1984), cyanides (Wu *et al.*, 2003; Shores *et al.*, 2002), or N-heterocyclic rings (Hagrman *et al.*, 1999; Willett *et al.*, 2001), their respective transition-metal complexes have always been an active area in coordination chemistry. Ethylenediamine (en) has been used in innumerable coordination compounds as a ligand (Cullen & Lingafelter, 1970; Daniels *et al.*, 1995; Jameson *et al.*, 1982), because it not only chelates metal cations by two nitrogen atoms, but also donates hydrogen atoms to form N—H···*X* hydrogen bonds. In the vast majority of cases, en coordinates to a central metal ion as a bidentate ligand *via* the two N atoms, forming a five-membered chelate ring. This ligand has been widely used to prepare a number of cobalt(III) complexes (Bailar & Clapp, 1945; Bailar & Rollinson, 1946). Interestingly, mixed-ligand cobalt(III) complexes find potential applications in the fields of antitumor, antibacterial, anti-

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1$W$—H1$W$···Cl2$^{i}$ | 0.83 (1) | 2.36 (1) | 3.158 (2) | 163 (3) |
| O1$W$—H2$W$···Cl1 | 0.83 (1) | 2.47 (2) | 3.175 (2) | 144 (3) |
| N1—H1$C$···Cl3$^{ii}$ | 0.83 (2) | 2.76 (2) | 3.5027 (16) | 148.7 (17) |
| N1—H1$D$···Cl2 | 0.82 (2) | 2.47 (2) | 3.2421 (16) | 156.7 (19) |
| N2—H2$E$···Cl3 | 0.84 (2) | 2.60 (2) | 3.4080 (16) | 160.7 (18) |
| N2—H2$F$···Cl3$^{iii}$ | 0.91 (2) | 2.70 (2) | 3.5887 (16) | 166 (2) |
| N3—H3$C$···O1$W$ | 0.88 (2) | 2.18 (2) | 2.989 (2) | 152.2 (19) |
| N3—H3$D$···Cl2$^{iv}$ | 0.85 (2) | 2.50 (2) | 3.2791 (16) | 154.0 (17) |
| N4—H4$D$···Cl2 | 0.87 (2) | 2.61 (2) | 3.4007 (17) | 151.6 (18) |
| N4—H4$C$···Cl3$^{iii}$ | 0.84 (2) | 2.56 (2) | 3.3815 (16) | 168.7 (17) |
| N5—H5$A$···Cl3 | 0.83 (2) | 2.42 (2) | 3.2345 (15) | 168.6 (17) |
| N5—H5$B$···Cl3$^{ii}$ | 0.90 (2) | 2.38 (2) | 3.2778 (15) | 173.0 (17) |
| C4—H4$A$···Cl2$^{v}$ | 0.97 | 2.81 | 3.5148 (18) | 130 |

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

microbial, radiosenzitation and cytotoxicity activities (Sayed *et al.*, 1992; Teicher *et al.*, 1990; Arslan *et al.*, 2009; Delehanty *et al.*, 2008). It is well documented that cobalt(III)–chelate complexes can also function as efficient electron-transfer mediators in solar energy conversion schemes (Sapp *et al.*, 2002). Complexes of cobalt are also useful for nutritional supplementation to provide cobalt in a form that effectively increases the bioavailability, for instance, vitamin B12 by microorganisms present in the gut. The structure determination of the title compound has been carried out against this background to ascertain the molecular conformation, binding modes and hydrogen-bonding interactions in the crystal structure.

The structural entities of the title salt are displayed in Fig. 1. The coordination environment around the $Co^{III}$ atom is approximately octahedral and defined by one N-bound fluoroaniline ligand, one chloride ion and two ethylenediamine ligands. The angles subtended by the chelating en ligands deviate the most from 90° [N1—Co1—N2 = 85.23 (6)° and N3—Co1—N4 = 84.99 (6)°]. The N atoms N2, N3, N4 and N5 define the equatorial plane, and N1 and Cl1 the axial ligands. The Co—N bond lengths range from 1.9598 (14) to

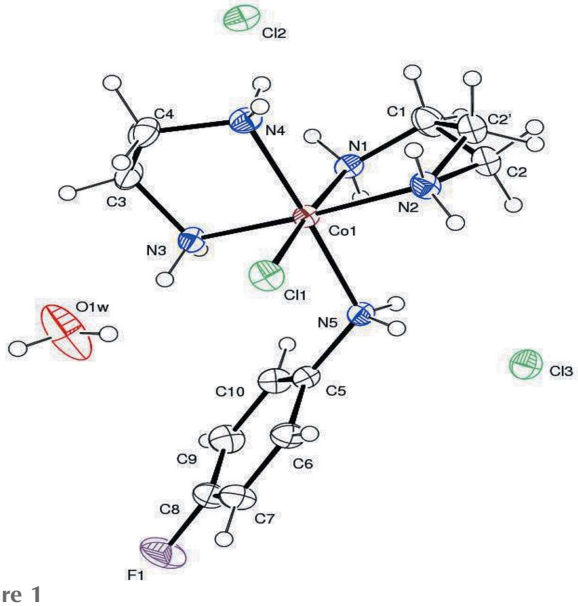



**Figure 1**
The structural entities of the title complex, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

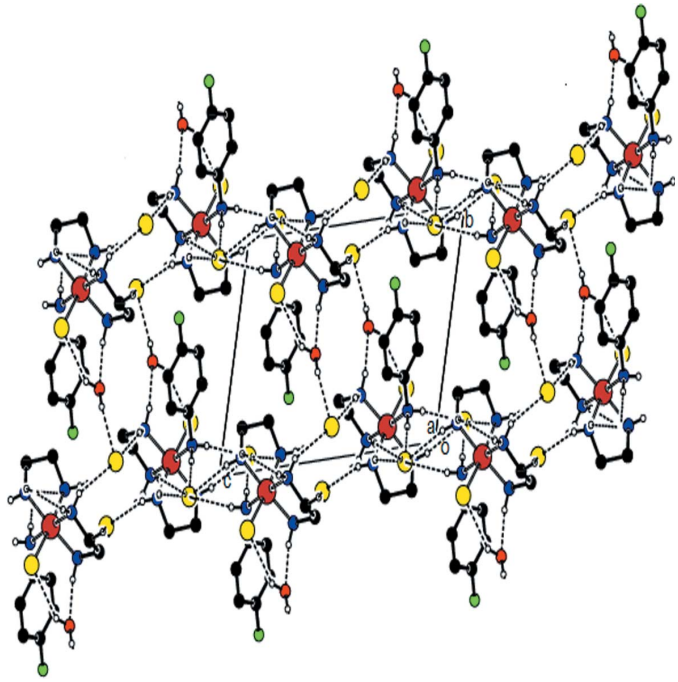



**Figure 2**
View of the three-dimensional hydrogen-bonded network of $[Co^{III}(en)_2(p\text{-}FC_6H_4NH_2)Cl]Cl_2\cdot H_2O$, with hydrogen bonds indicated by dashed lines

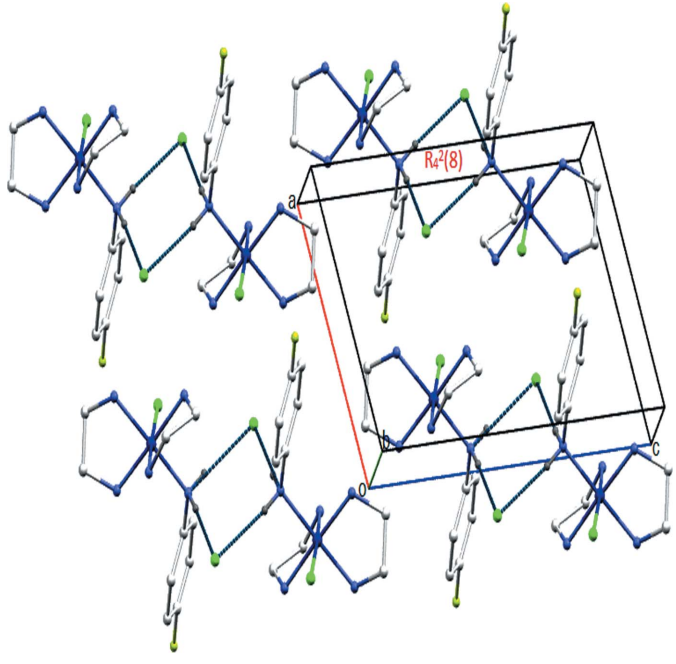



**Figure 3**
Representative of all other hydrogen-bonding interactions, N—H···Cl hydrogen bonds are shown (dotted lines), generating an $R_4^2(8)$ ring motif.

2.0077 (13) Å, with the longest (Co1—N5) being the bond to the monodentate 4-fluoroaniline ligand. The methylene C2 atom in one of the five-membered en ligands is disordered over two sets of sites, with a refined occupancy ratio of 0.831 (10):0.168 (10). The chelate ring (Co1/N1/C1/C2/N2) adopts a twisted conformation on the C1—C2 bond with puckering parameters $q_2$ = 0.4012 (18) Å, and $\varphi_2$ = 92.2 (16)°. The chelate ring Co1/N1/C1/C2′/N2 with the minor contribution to the disorder at C2′ likewise exhibits a twisted conformation with puckering parameters $q_2$ = 0.149 (5) Å, and $\varphi_2$ = 19 (3)°. The chelate ring Co1/N3/C3/C4/N4 has puckering parameters $q_2$ = 0.4275 (15) Å, and $\varphi_2$ = 282.72 (15)°. The latter value indicates a conformation between a twisted and an envelope form.

The packing of the crystal structure is dominated by N—H···Cl, O—H···Cl and C—H···Cl hydrogen-bonding interactions (Table 1) between the complex cation, the two counter-anions and the water molecule of crystallization, thereby generating rings with $R_4^2(8)$, $R_2^1(6)$, $R_4^2(10)$ and $R_2^2(6)$ graph-set motifs. Within the three-dimensional network (Figs. 2 and 3), no $\pi$–$\pi$ stacking interactions are observed.

## Synthesis and crystallization

The complex was synthesized using dichloridobis(1,2-diaminoethane)cobalt(III) chloride according to a reported method (Bailar & Clapp, 1945). 2 g of *trans*-[$Co^{III}(en)_2Cl_2$]Cl were suspended in 3–4 drops of deionized water. 3 ml of 4-fluoroaniline were added dropwise over 20 min, and the final mixture was ground well for 30 min. Grinding was continued for half an hour, and a colour change was observed for every addition of amine; the colour was found to change from dull green to rosey red. The reaction mixture was set aside until no further colour change was observed. The product was allowed to stand overnight. Finally, the solid was washed 3–4 times with ethanol. The final complex was dissolved in 5–10 ml of deionized water and the solution heated to 343 K. The cobalt(III) complex was recrystallized from hot water by addition of a few drops of conc. HCl and cooling. The crystals were filtered, washed with ethanol and dried under vacuum.

## Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The methylene group at C2 is disordered over two sets of sites and was refined with a 0.832 (10):0.168 (10) ratio.

**Table 2**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $[CoCl(C_6H_6FN)(C_2H_8N_2)_2]Cl_2\cdot H_2O$ |
| $M_r$ | 414.62 |
| Crystal system, space group | Triclinic, $P\overline{1}$ |
| Temperature (K) | 293 |
| $a, b, c$ (Å) | 8.1712 (6), 9.5435 (8), 11.9771 (10) |
| $\alpha, \beta, \gamma$ (°) | 104.231 (4), 99.490 (4), 100.705 (4) |
| $V$ (Å$^3$) | 867.57 (12) |
| $Z$ | 2 |
| Radiation type | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 1.47 |
| Crystal size (mm) | 0.25 × 0.20 × 0.15 |
| Data collection | |
| Diffractometer | Oxford Diffraction Xcalibur diffractometer with Eos detector |
| Absorption correction | Multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2009) |
| $T_{min}$, $T_{max}$ | 0.711, 0.810 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 15546, 3053, 2875 |
| $R_{int}$ | 0.022 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.595 |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.019, 0.058, 1.12 |
| No. of reflections | 3053 |
| No. of parameters | 243 |
| No. of restraints | 5 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.47, −0.23 |

Computer programs: *CrysAlis CCD* and *CrysAlis RED* (Oxford Diffraction, 2009), *SHELXS97* and *SHELXL2014* (Sheldrick, 2008, 2015) and *PLATON* (Spek, 2009).

## Acknowledgements

YA and ASP thank Dr Babu Varghese, SAIF, IIT, Chennai, India, for the data collection.

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# full crystallographic data

*IUCrData* (2019). **4**, x190327 [https://doi.org/10.1107/S2414314619003274]

## Chloridobis(ethane-1,2-diamine)(4-fluoroaniline)cobalt(III) dichloride monohydrate

**A. SubbiahPandi, Y. AaminaNaaz, C. Maharaja Mahalakshmi and K. Anbalagan**

### Chloridobis(ethane-1,2-diamine)(4-fluoroaniline)cobalt(III) dichloride monohydrate

*Crystal data*

$[CoCl(C_6H_6FN)(C_2H_8N_2)_2]Cl_2·H_2O$
$M_r$ = 414.62
Triclinic, $P\overline{1}$
$a$ = 8.1712 (6) Å
$b$ = 9.5435 (8) Å
$c$ = 11.9771 (10) Å
$\alpha$ = 104.231 (4)°
$\beta$ = 99.490 (4)°
$\gamma$ = 100.705 (4)°
$V$ = 867.57 (12) Å$^3$
$Z$ = 2
$F(000)$ = 428
$D_x$ = 1.587 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 6556 reflections
$\theta$ = 1.8–25.0°
$\mu$ = 1.47 mm$^{-1}$
$T$ = 293 K
Prism, dark-red
0.25 × 0.20 × 0.15 mm

*Data collection*

Oxford Diffraction Xcalibur diffractometer with Eos detector
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ and $\varphi$ scan
Absorption correction: multi-scan (CrysAlis PRO; Oxford Diffraction, 2009)
$T_{min}$ = 0.711, $T_{max}$ = 0.810
15546 measured reflections
3053 independent reflections
2875 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.022
$\theta_{max}$ = 25.0°, $\theta_{min}$ = 1.8°
$h$ = −9→9
$k$ = −11→11
$l$ = −14→14

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.019
$wR(F^2)$ = 0.058
$S$ = 1.12
3053 reflections
243 parameters
5 restraints
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0314P)^2 + 0.2678P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.040
$\Delta\rho_{max}$ = 0.47 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.23 e Å$^{-3}$

*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.

**Refinement**. H atoms bonded to N and O atoms were freely refined. Other H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and allowed to ride on their parent atoms, with $1.5U_{eq}$(C) for methyl H and $1.2U_{eq}$(C) for other H atoms.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| C1 | 0.8028 (3) | 0.2984 (2) | 0.70155 (17) | 0.0433 (4) | |
| H1A | 0.7069 | 0.2596 | 0.7323 | 0.052* | |
| H1B | 0.8789 | 0.2314 | 0.6994 | 0.052* | |
| C2 | 0.7412 (5) | 0.3087 (3) | 0.5810 (2) | 0.0366 (7) | 0.831 (10) |
| H2A | 0.8363 | 0.3268 | 0.5434 | 0.044* | 0.831 (10) |
| H2B | 0.6615 | 0.2167 | 0.5331 | 0.044* | 0.831 (10) |
| C2′ | 0.677 (2) | 0.2884 (16) | 0.5948 (9) | 0.0366 (7) | 0.168 (10) |
| H2C | 0.7164 | 0.2443 | 0.5251 | 0.044* | 0.168 (10) |
| H2D | 0.5688 | 0.2252 | 0.5932 | 0.044* | 0.168 (10) |
| C3 | 0.8170 (2) | 0.7410 (2) | 0.97814 (14) | 0.0355 (4) | |
| H3A | 0.8400 | 0.8379 | 1.0353 | 0.043* | |
| H3B | 0.8582 | 0.6730 | 1.0177 | 0.043* | |
| C4 | 0.6297 (2) | 0.6855 (2) | 0.92613 (16) | 0.0380 (4) | |
| H4A | 0.5701 | 0.6584 | 0.9842 | 0.046* | |
| H4B | 0.5839 | 0.7618 | 0.9003 | 0.046* | |
| C5 | 1.07502 (19) | 0.79981 (17) | 0.68416 (13) | 0.0260 (3) | |
| C6 | 1.0343 (2) | 0.91023 (19) | 0.63617 (15) | 0.0336 (4) | |
| H6 | 0.9318 | 0.8913 | 0.5816 | 0.040* | |
| C7 | 1.1462 (2) | 1.0489 (2) | 0.66945 (17) | 0.0398 (4) | |
| H7 | 1.1207 | 1.1239 | 0.6376 | 0.048* | |
| C8 | 1.2952 (2) | 1.0728 (2) | 0.75043 (17) | 0.0393 (4) | |
| C9 | 1.3379 (2) | 0.9665 (2) | 0.80007 (17) | 0.0404 (4) | |
| H9 | 1.4395 | 0.9871 | 0.8558 | 0.049* | |
| C10 | 1.2267 (2) | 0.82760 (19) | 0.76570 (15) | 0.0330 (4) | |
| H10 | 1.2539 | 0.7530 | 0.7974 | 0.040* | |
| N1 | 0.89406 (19) | 0.44838 (15) | 0.77835 (13) | 0.0274 (3) | |
| N2 | 0.65522 (19) | 0.43488 (16) | 0.59361 (13) | 0.0302 (3) | |
| N3 | 0.90333 (18) | 0.75159 (16) | 0.87969 (12) | 0.0270 (3) | |
| N4 | 0.60849 (19) | 0.55338 (17) | 0.82415 (13) | 0.0300 (3) | |
| N5 | 0.96160 (18) | 0.65309 (15) | 0.64670 (12) | 0.0264 (3) | |
| O1W | 0.8073 (3) | 1.0395 (2) | 0.8821 (2) | 0.0898 (7) | |
| F1 | 1.40512 (17) | 1.20859 (13) | 0.78300 (13) | 0.0650 (4) | |
| Cl1 | 0.63686 (5) | 0.75092 (4) | 0.66669 (4) | 0.03357 (11) | |
| Cl2 | 0.77375 (5) | 0.36339 (5) | 1.00298 (4) | 0.03664 (11) | |
| Cl3 | 0.77258 (5) | 0.56575 (4) | 0.37113 (3) | 0.03353 (11) | |
| Co1 | 0.78188 (2) | 0.59758 (2) | 0.73432 (2) | 0.02174 (8) | |
| H5A | 0.907 (2) | 0.641 (2) | 0.5793 (18) | 0.028 (5)* | |
| H5B | 1.027 (3) | 0.586 (2) | 0.6397 (17) | 0.040 (5)* | |
| H2E | 0.658 (3) | 0.459 (2) | 0.5306 (19) | 0.036 (5)* | |
| H3C | 0.907 (3) | 0.841 (2) | 0.8705 (18) | 0.043 (6)* | |
| H3D | 1.004 (3) | 0.744 (2) | 0.9011 (17) | 0.033 (5)* | |

| | | | | |
|---|---|---|---|---|
| H4C | 0.510 (3) | 0.535 (2) | 0.7820 (18) | 0.035 (5)* |
| H4D | 0.622 (3) | 0.478 (2) | 0.8502 (18) | 0.040 (5)* |
| H2F | 0.544 (3) | 0.417 (2) | 0.5982 (19) | 0.051 (6)* |
| H1D | 0.891 (3) | 0.446 (2) | 0.846 (2) | 0.040 (6)* |
| H1C | 0.995 (3) | 0.465 (2) | 0.7716 (17) | 0.034 (5)* |
| H2W | 0.747 (3) | 0.996 (3) | 0.8157 (14) | 0.087 (10)* |
| H1W | 0.778 (4) | 1.118 (2) | 0.903 (3) | 0.089 (10)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0486 (11) | 0.0300 (9) | 0.0484 (11) | 0.0114 (8) | 0.0022 (9) | 0.0098 (8) |
| C2 | 0.0345 (17) | 0.0298 (11) | 0.0397 (11) | 0.0034 (11) | 0.0061 (11) | 0.0039 (8) |
| C2′ | 0.0345 (17) | 0.0298 (11) | 0.0397 (11) | 0.0034 (11) | 0.0061 (11) | 0.0039 (8) |
| C3 | 0.0345 (9) | 0.0432 (10) | 0.0253 (8) | 0.0069 (7) | 0.0098 (7) | 0.0032 (7) |
| C4 | 0.0317 (9) | 0.0466 (10) | 0.0364 (9) | 0.0109 (8) | 0.0153 (7) | 0.0069 (8) |
| C5 | 0.0250 (8) | 0.0295 (8) | 0.0247 (8) | 0.0055 (6) | 0.0088 (6) | 0.0084 (6) |
| C6 | 0.0323 (9) | 0.0372 (9) | 0.0325 (9) | 0.0077 (7) | 0.0035 (7) | 0.0147 (7) |
| C7 | 0.0443 (11) | 0.0340 (9) | 0.0450 (10) | 0.0078 (8) | 0.0102 (8) | 0.0190 (8) |
| C8 | 0.0371 (10) | 0.0327 (9) | 0.0433 (10) | −0.0024 (7) | 0.0119 (8) | 0.0079 (8) |
| C9 | 0.0259 (9) | 0.0485 (11) | 0.0409 (10) | 0.0023 (8) | 0.0007 (7) | 0.0110 (8) |
| C10 | 0.0279 (9) | 0.0378 (9) | 0.0363 (9) | 0.0094 (7) | 0.0056 (7) | 0.0156 (7) |
| N1 | 0.0255 (8) | 0.0334 (7) | 0.0262 (8) | 0.0088 (6) | 0.0064 (6) | 0.0119 (6) |
| N2 | 0.0284 (8) | 0.0317 (7) | 0.0270 (7) | 0.0041 (6) | 0.0000 (6) | 0.0082 (6) |
| N3 | 0.0217 (7) | 0.0317 (8) | 0.0252 (7) | 0.0055 (6) | 0.0041 (5) | 0.0053 (6) |
| N4 | 0.0221 (7) | 0.0367 (8) | 0.0312 (7) | 0.0053 (6) | 0.0045 (6) | 0.0117 (6) |
| N5 | 0.0266 (7) | 0.0289 (7) | 0.0234 (7) | 0.0066 (6) | 0.0048 (6) | 0.0075 (6) |
| O1W | 0.0933 (15) | 0.0445 (10) | 0.0991 (16) | 0.0212 (10) | −0.0361 (12) | −0.0042 (10) |
| F1 | 0.0572 (8) | 0.0422 (7) | 0.0782 (9) | −0.0157 (6) | 0.0012 (7) | 0.0149 (6) |
| Cl1 | 0.0306 (2) | 0.0346 (2) | 0.0362 (2) | 0.01223 (17) | 0.00199 (17) | 0.01167 (17) |
| Cl2 | 0.0293 (2) | 0.0498 (3) | 0.0349 (2) | 0.00972 (18) | 0.00575 (17) | 0.02020 (19) |
| Cl3 | 0.0331 (2) | 0.0377 (2) | 0.0293 (2) | 0.00967 (17) | 0.00385 (16) | 0.00964 (16) |
| Co1 | 0.01859 (12) | 0.02502 (12) | 0.02089 (12) | 0.00481 (8) | 0.00245 (8) | 0.00680 (9) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—C2′ | 1.472 (12) | C7—H7 | 0.9300 |
| C1—N1 | 1.477 (2) | C8—F1 | 1.357 (2) |
| C1—C2 | 1.482 (3) | C8—C9 | 1.365 (3) |
| C1—H1A | 0.9700 | C9—C10 | 1.383 (3) |
| C1—H1B | 0.9700 | C9—H9 | 0.9300 |
| C2—N2 | 1.492 (3) | C10—H10 | 0.9300 |
| C2—H2A | 0.9700 | N1—Co1 | 1.9598 (14) |
| C2—H2B | 0.9700 | N1—H1D | 0.82 (2) |
| C2′—N2 | 1.445 (12) | N1—H1C | 0.83 (2) |
| C2′—H2C | 0.9700 | N2—Co1 | 1.9655 (14) |
| C2′—H2D | 0.9700 | N2—H2E | 0.84 (2) |
| C3—N3 | 1.485 (2) | N2—H2F | 0.91 (2) |

| | | | |
|---|---|---|---|
| C3—C4 | 1.494 (2) | N3—Co1 | 1.9512 (14) |
| C3—H3A | 0.9700 | N3—H3C | 0.88 (2) |
| C3—H3B | 0.9700 | N3—H3D | 0.85 (2) |
| C4—N4 | 1.484 (2) | N4—Co1 | 1.9613 (14) |
| C4—H4A | 0.9700 | N4—H4C | 0.84 (2) |
| C4—H4B | 0.9700 | N4—H4D | 0.87 (2) |
| C5—C10 | 1.383 (2) | N5—Co1 | 2.0077 (13) |
| C5—C6 | 1.384 (2) | N5—H5A | 0.83 (2) |
| C5—N5 | 1.447 (2) | N5—H5B | 0.90 (2) |
| C6—C7 | 1.384 (3) | O1W—H2W | 0.825 (10) |
| C6—H6 | 0.9300 | O1W—H1W | 0.825 (10) |
| C7—C8 | 1.369 (3) | Cl1—Co1 | 2.2610 (4) |

| | | | |
|---|---|---|---|
| C2′—C1—N1 | 117.6 (6) | C1—N1—Co1 | 109.86 (11) |
| N1—C1—C2 | 108.71 (17) | C1—N1—H1D | 105.6 (14) |
| N1—C1—H1A | 109.9 | Co1—N1—H1D | 111.8 (14) |
| C2—C1—H1A | 109.9 | C1—N1—H1C | 109.1 (13) |
| N1—C1—H1B | 109.9 | Co1—N1—H1C | 110.6 (13) |
| C2—C1—H1B | 109.9 | H1D—N1—H1C | 110 (2) |
| H1A—C1—H1B | 108.3 | C2′—N2—Co1 | 115.5 (5) |
| N2—C2—C1 | 107.1 (2) | C2—N2—Co1 | 109.53 (13) |
| N2—C2—H2A | 110.3 | C2′—N2—H2E | 117.5 (14) |
| C1—C2—H2A | 110.3 | C2—N2—H2E | 103.6 (13) |
| N2—C2—H2B | 110.3 | Co1—N2—H2E | 112.5 (14) |
| C1—C2—H2B | 110.3 | C2′—N2—H2F | 95.8 (11) |
| H2A—C2—H2B | 108.6 | C2—N2—H2F | 118.2 (11) |
| N2—C2′—C1 | 110.1 (9) | Co1—N2—H2F | 107.2 (13) |
| N2—C2′—H2C | 109.6 | H2E—N2—H2F | 106 (2) |
| C1—C2′—H2C | 109.7 | C3—N3—Co1 | 111.09 (10) |
| N2—C2′—H2D | 109.6 | C3—N3—H3C | 107.5 (14) |
| C1—C2′—H2D | 109.6 | Co1—N3—H3C | 110.8 (14) |
| H2C—C2′—H2D | 108.1 | C3—N3—H3D | 107.2 (13) |
| N3—C3—C4 | 107.41 (14) | Co1—N3—H3D | 111.6 (13) |
| N3—C3—H3A | 110.2 | H3C—N3—H3D | 108.5 (19) |
| C4—C3—H3A | 110.2 | C4—N4—Co1 | 108.62 (10) |
| N3—C3—H3B | 110.2 | C4—N4—H4C | 108.8 (13) |
| C4—C3—H3B | 110.2 | Co1—N4—H4C | 110.7 (13) |
| H3A—C3—H3B | 108.5 | C4—N4—H4D | 109.1 (13) |
| N4—C4—C3 | 106.80 (14) | Co1—N4—H4D | 109.6 (13) |
| N4—C4—H4A | 110.4 | H4C—N4—H4D | 110.0 (19) |
| C3—C4—H4A | 110.4 | C5—N5—Co1 | 121.40 (10) |
| N4—C4—H4B | 110.4 | C5—N5—H5A | 107.3 (13) |
| C3—C4—H4B | 110.4 | Co1—N5—H5A | 103.8 (13) |
| H4A—C4—H4B | 108.6 | C5—N5—H5B | 107.7 (13) |
| C10—C5—C6 | 120.28 (15) | Co1—N5—H5B | 109.6 (12) |
| C10—C5—N5 | 119.46 (14) | H5A—N5—H5B | 106.0 (18) |
| C6—C5—N5 | 120.23 (15) | H2W—O1W—H1W | 105 (3) |
| C5—C6—C7 | 119.91 (16) | N3—Co1—N1 | 92.75 (6) |

| | | | |
|---|---|---|---|
| C5—C6—H6 | 120.0 | N3—Co1—N4 | 84.99 (6) |
| C7—C6—H6 | 120.0 | N1—Co1—N4 | 90.62 (7) |
| C8—C7—C6 | 118.35 (16) | N3—Co1—N2 | 176.62 (6) |
| C8—C7—H7 | 120.8 | N1—Co1—N2 | 85.23 (6) |
| C6—C7—H7 | 120.8 | N4—Co1—N2 | 92.31 (6) |
| F1—C8—C9 | 118.59 (17) | N3—Co1—N5 | 92.74 (6) |
| F1—C8—C7 | 118.41 (17) | N1—Co1—N5 | 91.00 (6) |
| C9—C8—C7 | 123.00 (16) | N4—Co1—N5 | 177.27 (6) |
| C8—C9—C10 | 118.52 (16) | N2—Co1—N5 | 90.01 (6) |
| C8—C9—H9 | 120.7 | N3—Co1—Cl1 | 93.15 (5) |
| C10—C9—H9 | 120.7 | N1—Co1—Cl1 | 174.09 (4) |
| C5—C10—C9 | 119.93 (15) | N4—Co1—Cl1 | 89.58 (5) |
| C5—C10—H10 | 120.0 | N2—Co1—Cl1 | 88.86 (4) |
| C9—C10—H10 | 120.0 | N5—Co1—Cl1 | 89.03 (4) |
| | | | |
| N1—C1—C2—N2 | −48.3 (3) | N5—C5—C10—C9 | 178.51 (15) |
| N1—C1—C2′—N2 | −6.3 (13) | C8—C9—C10—C5 | −1.0 (3) |
| N3—C3—C4—N4 | 48.91 (19) | C2′—C1—N1—Co1 | 13.3 (8) |
| C10—C5—C6—C7 | 0.3 (2) | C2—C1—N1—Co1 | 36.3 (2) |
| N5—C5—C6—C7 | −177.80 (15) | C1—C2′—N2—Co1 | −3.8 (13) |
| C5—C6—C7—C8 | −0.4 (3) | C1—C2—N2—Co1 | 38.2 (3) |
| C6—C7—C8—F1 | 179.91 (17) | C4—C3—N3—Co1 | −32.55 (17) |
| C6—C7—C8—C9 | −0.3 (3) | C3—C4—N4—Co1 | −43.33 (17) |
| F1—C8—C9—C10 | −179.23 (16) | C10—C5—N5—Co1 | 88.59 (17) |
| C7—C8—C9—C10 | 1.0 (3) | C6—C5—N5—Co1 | −93.27 (16) |
| C6—C5—C10—C9 | 0.4 (2) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1*W*—H1*W*···Cl2$^{i}$ | 0.83 (1) | 2.36 (1) | 3.158 (2) | 163 (3) |
| O1*W*—H2*W*···Cl1 | 0.83 (1) | 2.47 (2) | 3.175 (2) | 144 (3) |
| N1—H1*C*···Cl3$^{ii}$ | 0.83 (2) | 2.76 (2) | 3.5027 (16) | 148.7 (17) |
| N1—H1*D*···Cl2 | 0.82 (2) | 2.47 (2) | 3.2421 (16) | 156.7 (19) |
| N2—H2*E*···Cl3 | 0.84 (2) | 2.60 (2) | 3.4080 (16) | 160.7 (18) |
| N2—H2*F*···Cl3$^{iii}$ | 0.91 (2) | 2.70 (2) | 3.5887 (16) | 166 (2) |
| N3—H3*C*···O1*W* | 0.88 (2) | 2.18 (2) | 2.989 (2) | 152.2 (19) |
| N3—H3*D*···Cl2$^{iv}$ | 0.85 (2) | 2.50 (2) | 3.2791 (16) | 154.0 (17) |
| N4—H4*D*···Cl2 | 0.87 (2) | 2.61 (2) | 3.4007 (17) | 151.6 (18) |
| N4—H4*C*···Cl3$^{iii}$ | 0.84 (2) | 2.56 (2) | 3.3815 (16) | 168.7 (17) |
| N5—H5*A*···Cl3 | 0.83 (2) | 2.42 (2) | 3.2345 (15) | 168.6 (17) |
| N5—H5*B*···Cl3$^{ii}$ | 0.90 (2) | 2.38 (2) | 3.2778 (15) | 173.0 (17) |
| C4—H4*A*···Cl2$^{v}$ | 0.97 | 2.81 | 3.5148 (18) | 130 |

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