A mixed-valence [Co$^{II}_{4}$Co$^{III}_{2}$] cluster with defect disk-shaped topology

Hua Yang,*a Yu-Pei Fu,a Yuan Huang,b Xiao-Li Chen,a Dan Qiaoa and Hua-Li Cuia

*aSchool of Chemistry and Chemical Engineering, Shaanxi Key Laboratory of Chemical Reaction Engineering, Laboratory of New Energy and New Function Materials, Yan’an University, Yan’an 716000, People’s Republic of China, and bCollege of Chemistry, Chemical Engineering and Materials Science, Soochow University, Suzhou 215123, People’s Republic of China. *Correspondence e-mail: yanghua_08@163.com

The employment of the new Schiff base ligand 2-[(4-chloro-2-hydroxybenzylideneamino)methyl]phenol (H$_2$L) bearing O$_2$N donors for the preparation of a novel Co$_6$ cluster is reported. The hexanuclear cobalt complex, namely, di-/C$_2$2-acetatotetrakis{/C$_2$2-2-[(4-chloro-2-oxidobenzylideneamino)methyl]phenolato}tetra-/C$_2$2-3-methanolato-tetracobalt(II)dicobalt(III), [Co$^{II}_{4}$Co$^{III}_{2}$(C$_{14}$H$_{10}$ClNO$_2$)$_4$(CH$_3$COO)$_2$(CH$_3$O)$_4$], was obtained using Co(CH$_3$COO)$_2$·4H$_2$O and H$_2$L as starting materials in MeOH under solvothermal conditions. The six metal ions are linked together by the μ$_3$-O atoms of four deprotonated MeOH molecules, two CH$_3$COO units and six phenolate O atoms of four L$_2$ ligands to form a defect disk-shaped topology. DC magnetic susceptibility investigations revealed the existence of antiferromagnetic interactions in the Co$_6$ cluster.

1. Introduction

Polynuclear coordination compounds of 3$d$ transition metals have attracted continued attention for several decades due to their structural novelty, interesting catalytic (Dastidar & Chattopadhyay, 2022; Shul’pin & Shulpina, 2021; Nesterov & Nesterova, 2018; Jing et al., 2020) and biological properties (Hazari et al., 2017; Amtul et al., 2002; Azizian et al., 2012; Tanaka et al., 2003), and their potential as single-molecule magnets (SMMs) (Radu et al., 2017; Pattacini et al., 2011). Among numerous polynuclear 3$d$ complexes, cobalt clusters have received particular interest because of their pleasing topological aesthetics (Brechin et al., 1997; Cao et al., 2013), their relevance to dioxygen reduction (Monte-Pérez et al., 2017) and their fascinating magnetic properties (Liu et al., 2020; Sarto et al., 2018; Li et al., 2020; Ma et al., 2012).

Several synthetic methodologies towards polynuclear cobalt clusters were established and one of the most efficient approaches involves the employment of hydroxy-containing Schiff base ligands. Schiff base ligands are easy to synthesize and their steric properties can be tuned by varying the size of the amine or carbonyl substituents (Qin et al., 2017, 2018; Ge et al., 2018; Li et al., 2021). More importantly, the hydroxy moieties of the Schiff base ligands can combine many metal ions with μ-O bridges, resulting in the formation of large polynuclear clusters.

In the present work, we utilized the hydroxy-containing Schiff base 2-[(4-chloro-2-hydroxybenzylideneamino)methyl]phenol (H$_2$L) (Huang et al., 2019) as a ligand to assemble a polynuclear cobalt cluster. The hexanuclear cobalt compound [Co$^{II}_{4}$Co$^{III}_{2}$(L)$_4$(CH$_3$COO)$_2$(MeO)$_4$] (1) was obtained successfully and we report its structural diversity and discuss its magnetic properties.
2. Experimental

2.1. Materials and physical measurements

All chemicals were of reagent grade, purchased from commercial suppliers and used without further purification. All manipulations were conducted under aerobic and solvothermal conditions. H$_2$L was synthesized following the literature procedure of Huang et al. (2019). Elemental analyses for C, H and N were performed with a Carlo-Erba EA1110 CHNO-S analyser. The FT–IR spectrum was determined on a Nicolet Magna-IR 500 spectrometer using KBr pellets in the range 400–4000 cm$^{-1}$. DC magnetic susceptibilities were measured in the temperature range 2–300 K in a field of 1000 Oe using a Quantum Design MPMS-7 SQUID magnetometer.

2.2. Synthesis and crystallization

To a Pyrex tube (10 ml) was added a mixture of H$_2$L (0.0291 g, 0.02 mmol), Co(CH$_3$COO)$_2$·4H$_2$O (0.0249 g, 0.1 mmol), Et$_3$N (0.0202 g, 0.2 mmol) and MeOH (1.5 ml). The tube was sealed and heated at 80°C for 48 h under autogenous pressure. It was then cooled to room temperature and dark-red needle-like crystals were obtained. The crystals were collected, washed with MeOH (2 ml) and dried in air (yield: 0.020 g; 48% based on cobalt). Analysis calculated (%) for C$_{64}$H$_{58}$Cl$_4$Co$_6$N$_4$O$_{16}$: C 46.18, H 4.048, N 3.280. There exists an approximate 2 symmetry in the molecule. The vibrations of the C-H bands. The signals of the carboxyl $v_{as}$(CO$_2$) and $v_s$(CO$_2$) vibrations were found in the 1637–1419 cm$^{-1}$ range. The vibrations of the C=O bond appear at 1268–1185 cm$^{-1}$ range were assigned to the vibrations of the aromatic rings. The sharp signals in the 979–766 cm$^{-1}$ range were ascribed to the vibrations of C–H bonds.

2.3. Structure determination

Crystal data, collection and structure refinement details are summarized in Table 1. The crystal structure contained disordered solvent that could not be satisfactorily refined. The SQUEEZE (Spek, 2015) routine of PLATON (Spek, 2020) was used in the treatment of the crystallographic data. All H atoms were placed in geometrically idealized positions, with C–H = 0.95–0.99 Å. The H atoms of the CH$_2$ and amide groups were constrained to ride on their parent atoms, with $U_{iso}$(H) = $1.2U_{eq}$(C). The H atoms of CH$_3$ groups were refined as rotating groups, with $U_{iso}$(H) = $1.5U_{eq}$(C).

3. Results and discussion

3.1. Synthesis of complex 1 and IR spectral analysis

The reaction of H$_2$L with Co(CH$_3$COO)$_2$·4H$_2$O in MeOH under solvothermal conditions led to the isolation of 1 in moderate yield. Co(CH$_3$COO)$_2$·4H$_2$O is a good starting material because it not only serves as a convenient metal source, but also provides CH$_3$COO$^-$ bridging ligands. In the solid state, complex 1 is stable in air and its elemental analysis is consistent with the given molecular formula.

The vibrational bands in the IR spectrum agree well with the formulation of complex 1 (see Fig. S1 in the supporting information). The signals of the carboxyl $v_{as}$(CO$_2$) and $v_s$(CO$_2$) vibrations were found in the 1637–1419 cm$^{-1}$ range. The vibrations of the C=O bond appear at 1268–1185 cm$^{-1}$ range were assigned to the vibrations of the aromatic rings. The sharp signals in the 979–766 cm$^{-1}$ range were ascribed to the vibrations of C–H bonds.

3.2. Structure description of 1

Single crystals of 1 were obtained from MeOH under solvothermal conditions. Complex 1 crystallized in the orthorhombic space group P2$_1$2$_1$2$_1$. The structure is shown in Fig. 1. The structure analysis shows that complex 1 is composed of six cobalt ions, four 2-[4-chloro-2-oxidobenzylideneamino)methyl]phenolate (L$^{2-}$) ligands, two acetate ligands and four methanol-solvent-derived MeO$^-$ ligands. There exists an approximate $C_2$ symmetry in the molecule. The imine N atom and both phenolate O-atom donors of each $L^{2-}$ ligand coordinate each cobalt centre. Bond valence calculations (Brese & O’Keeffe, 1991; Brown & Altermatt, 1985) gave valence parameters of 1.90, 2.32, 3.60, 2.12, 3.64 and 2.31 for the Co1–Co6 ions, respectively, indicating that the Co3 and Co5 ions are in $3+$ valence states, and that the Co1, Co2, Co4

---

Table 1

| Crystal data | Chemical formula |
|--------------|------------------|
| $M_r$ | $[Co_6(C_4H_6CINO_2)_{12}^+ (CH_3O)_{12}]^{2+}$ |
| Crystal system, space group | Orthorhombic, P2$_1$2$_1$2$_1$ |
| Temperature (K) | 120 |
| $a$, $b$, $c$ (Å) | 15.4873 (10), 16.2116 (11), 28.1099 (19) |
| $V$ (Å$^3$) | 7057.7 (8) |
| $Z$ | 4 |
| Radiation type | Mo Kα |
| $\mu$ (mm$^{-1}$) | 1.60 |
| Crystal size (mm) | 0.4 $\times$ 0.2 $\times$ 0.2 |

---

**Table 1**

Experimental details.

| Chemical formula | $[Co_6(C_4H_6CINO_2)_{12}^+ (CH_3O)_{12}]^{2+}$ |
|------------------|--------------------------------------------------|
| Crystal data     | $[Co_6(C_4H_6CINO_2)_{12}^+ (CH_3O)_{12}]^{2+}$ |
| $M_r$            | Orthorhombic, P2$_1$2$_1$2$_1$                    |
| Crystal system   | Orthorhombic, P2$_1$2$_1$2$_1$                    |
| space group      | Orthorhombic, P2$_1$2$_1$2$_1$                    |
| Temperature (K)  | 120                                               |
| $a$, $b$, $c$ (Å)| 15.4873 (10), 16.2116 (11), 28.1099 (19)         |
| $V$ (Å$^3$)      | 7057.7 (8)                                       |
| $Z$              | 4                                                |
| Radiation type   | Mo Kα                                            |
| $\mu$ (mm$^{-1}$)| 1.60                                             |
| Crystal size (mm)| 0.4 $\times$ 0.2 $\times$ 0.2                    |

---

**Computer programs:** SAINT (Bruker, 2016), APEX2 (Bruker, 2016), olex2.solve (Bourhis et al., 2015), SHELXL (Sheldrick, 2015), OLEX2 (Dolomanov et al., 2009) and PLATON (Spek, 2020).
and Co6 ions are in 2+ oxidation states. The formation of four fused defect cubes confirms the involvement of four methanol-solvent-derived \( \mu_3\text{-O}^{-} \) groups, giving the \( \text{Co}_6\text{O}_{10} \) structure. Thus, the molecular structure of 1 displays a defect disk-shaped topology [Fig. 1(b)]. Of the six cobalt centres, the Co1, Co3, Co4 and Co5 ions are six-coordinated, and the Co2 and Co6 ions are five-coordinated. The coordination environments of the Co2 and Co6 ions, and the Co3 and Co5 ions are individually identical. The Co1 centre is present in a distorted octahedral \( \text{O}_6 \) coordination environment, among which two O atoms are from two \( \mu_2\text{-k}^{\text{O}}:\text{O},\text{O}^{'},\text{N} \ L^2- \) ligands and four O atoms are from four \( \mu_3\text{-O}^{-} \) MeO\(^{-}\) ligands. The Co2 centre is enclosed by the N and O atoms of one \( \mu_2\text{-k}^{\text{O}}:\text{O},\text{O}^{'},\text{N} \ L^2- \) ligand, one O atom of a \( \mu_3\text{-k}^{\text{O}}:\text{O},\text{N},\text{O}^{'},\text{O} \ L^2- \) ligand and one O atom of one \( \mu_3\text{-O}^{-} \) MeO\(^{-}\) ligand. The six-coordinate NO\(_3\) environment around the Co3 ion is accomplished by two \( \mu_3\text{-O}^{-} \) MeO\(^{-}\) groups, one O atom from one acetate bridge and the N and O atoms of one \( \mu_3\text{-k}^{\text{O}}:\text{O},\text{N},\text{O}^{'},\text{O} \ L^2- \) ligand. The six O-donor atoms around the Co6 centre originate from bridging acetate ligands, two \( \mu_3\text{-O}^{-} \) MeO\(^{-}\) groups and two \( \mu_3\text{-k}^{\text{O}}:\text{O},\text{N},\text{O}^{'},\text{O} \ L^2- \) ligands. The \( \text{H}_2\text{L} \) ligand exhibits two types of coordination mode.

The geometries of the five-coordinated Co2 and Co6 atoms were analyzed with the program SHAPE (Version 2.0; Pinsky & Avnir, 1998). The calculated values revealed trigonal bipyramid\((D_{3h})\) geometry for both atoms, with a minimum \( \text{CShM} \) (continuous shape measure) value of 1.065 for Co2 and 1.172 for Co6.

Complex 1 joins a small family of Co\(_6\) clusters. Hexanuclear cobalt complexes mainly exhibit wheel, cage and ring topologies (Shi et al., 2021; Zou et al., 2014; Wang et al., 2013; Guo et al., 2013; Lazzarini et al., 2012; Chen et al., 2010; Malassa et al., 2010; Tudor et al., 2010; Colacio et al., 2009; Jones et al., 2009; Shiga & Oshio, 2007; Alley et al., 2006; Murrie et al., 2003; Kumagai et al., 2003; Gutschke et al., 1999). Complex 1 is a rare example that displays a defect disk-shaped structure.

### 3.3. Magnetic properties of 1

Magnetic susceptibility data as a function of temperature for complex 1 are shown in Fig. 2. The room temperature \( \chi_M T \) value is 10.96 cm\(^3\) mol\(^{-1}\) K, which is greater than the value of 7.50 cm\(^3\) mol\(^{-1}\) K for four uncoupled \( S = \frac{3}{2} \) Co\(^{II}\) centres, possibly owing to the orbital contributions of the metal ions.
Upon lowering the temperature, the $\chi_M T$ value drops slightly to a minimum value of 3.49 cm$^3$ mol$^{-1}$ K at 2 K, which suggests possible antiferromagnetic couplings between the unpaired spins. The data of $1/\chi_M$ at 2 K, which suggests possible antiferromagnetic couplings between the unpaired spins. The data of $1/\chi_M$ in the temperature range 2–300 K were fitted by the Curie–Weiss Law of $1/\chi_M = (T - \theta)/C$. The Curie constant $C = 12.09$ cm$^3$ mol$^{-1}$ K and the Weiss constant $\theta = -37.24$ K were obtained. The negative $\theta$ value proves the antiferromagnetic interactions.

The magnetic dynamic behaviour of 1 was also explored. The ac magnetic susceptibilities for 1 at 1000 Hz under a zero-dc field in the temperature range 2–25 K were shown in Fig. S2 (see supporting information). The $\chi''$ susceptibilities at 1000 Hz did not increase upon lowering the temperature and no peaks were determined. These phenomena revealed that complex 1 is not a single-molecule magnet.

4. Conclusion

A hexanuclear cobalt complex of composition [Co$_2^{III}$Co$_4^{II}$-(L)$_2$(CH$_3$COO)$_2$(MeOH)$_4$] (1), based on the hydroxy-containing Schiff base ligand 2-[4-chloro-2-hydroxybenzylideneamino)methyl]phenol (H$_2$L) was prepared and characterized. Complex 1 exhibits a defect disk-shaped topology. Four cobalt ions are six-coordinated and two cobalt ions are five-coordinated. An investigation of the magnetic properties revealed that there exist antiferromagnetic interactions between the Co$^{II}$ ions.

Funding information

Funding for this research was provided by: Natural Science Foundation of Shaanxi Province (grant Nos. 2022JZ-49 and 2022NY-071); Innovation and Entrepreneurship Training Program of College Students of Shaanxi Province (grant No. S202210719111); Natural Science Foundation of Yulin (grant No. CXY-2020-065).

References

Alley, K. G., Bircher, R., Waldmann, O., Ochsenbein, S. T., Güdel, H. U., Moubarakia, B., Murray, K. S., Fernandez-Alonso, F., Abrahams, B. F. & Boskovic, C. (2006). Inorg. Chem. 45, 8950–8957.

Amlut, Z., Atta-ur-Rahman, B. S. P., Siddiqui, R. A. & Choudhary, M. I. (2002). Curr. Med. Chem. 9, 1323–1348.

Azizian, H., Nabati, F., Sharifi, A., Siavoshi, F., Mahdavi, M. & Amanlou, M. (2012). J. Mol. Model. 18, 2917–2927.

Bourhis, L. J., Dolomanov, O. V., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2015). Acta Cryst. A71, 59–75.

Brechin, E. K., Harris, S. G., Harrison, A., Parsons, S., Gavin Whittaker, A. & Winpenny, R. E. P. (1997). Chem. Commun. pp. 653–654.

Brese, N. E. & O’Keefe, M. (1991). Acta Cryst. B47, 192–197.

Brown, I. D. & Altermatt, D. (1985). Acta Cryst. B41, 244–247.

Bruker (2016). APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.

Cao, Y. Y., Chen, Y. M., Li, L., Gao, D. D., Liu, W., Hu, H. L., Li, W. & Li, Y. H. (2013). Dalton Trans. 42, 10912–10918.

Chen, Q., Zeng, M. H., Wei, L. Q. & Kurmoo, M. (2010). Chem. Mater. 22, 4328–4334.

Colacio, E., Aouryaghah, H., Mota, A. J., Cano, J., Sillanpää, R. & Rodríguez-Diéquez, A. (2009). CrystEngComm 11, 2054–2064.

Dastidar, T. G. & Chattopadhyay, S. (2022). Polyhedron, 211, 115511–115536.

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

Ge, Y., Qin, Y. R., Cui, Y. F., Pan, Y. D., Huang, Y., Li, Y. H., Liu, W. & Zhang, Y. Q. (2018). Chem. Asian J. 13, 3753–3761.

Guo, J. Y., Zhang, D., Chen, L., Song, Y., Zhu, D. R. & Xu, Y. (2013). Dalton Trans. 42, 8454–8459.

Gutscheke, S. O. H., Price, D. I., Powell, A. K. & Wood, P. T. (1999). Angew. Chem. Int. Ed. 38, 1088–1090.

Hazari, A., Das, A., Mahapatra, P. & Ghosh, A. (2017). Polyhedron, 134, 99–106.

Huang, Y., Qin, Y. R., Ge, Y., Cui, Y. F., Zhang, X. M., Li, Y. H. & Yao, J. L. (2019). New J. Chem. 43, 19344–19354.

Jing, Y., Zhang, X. M., Cui, Y. F., Li, D. W., Sun, H., Ge, Y. & Li, Y. H. (2020). Chin. J. Struct. Chem. 39, 1057–1062.

Jones, L. F., Kilner, C. A. & Halcrow, M. A. (2009). Angew. Chem. Int. Ed. 48, 4667–4675.

Kumagai, H., Oka, Y., Kawata, S., Ohba, M., Inoue, K., Kurmoo, M. & Okawa, H. (2003). Polyhedron, 22, 1917–1920.

Lazzarini, I. C., Carrella, L., Rentschler, E. & Alborés, P. (2012). Polyhedron, 31, 779–788.
Li, D. W., Ding, M. M., Huang, Y., Tello Yepes, D. F., Li, H. Y., Li, Y. H., Zhang, Y. Q. & Yao, J. L. (2021). *Dalton Trans.* 50, 217–228.

Li, X., Guo, Y., Xu, T., Fang, M., Xu, Q., Zhang, F., Wu, Z., Li, C. & Zhu, W. (2020). *J. Chin. Chem. Soc.* 67, 1070–1077.

Liu, Y. N., Hou, J. L., Wang, Z., Gupta, R. K., Jaglicˇic´, Z., Jagodicˇ, M., Wang, W. G., Tung, C. H. & Sun, D. (2020). *Inorg. Chem.* 59, 5683–5693.

Ma, Y. S., Xue, F. F., Tang, X. Y., Chen, B. & Yuan, R. X. (2012). *Inorg. Chem. Commun.* 15, 285–287.

Malassa, A., Agthe, C., Görls, H., Podewitz, M., Yu, L., Herrmann, C., Reiher, M. & Westerhausen, M. (2010). *Eur. J. Inorg. Chem.* 2010, 1777–1790.

Monte-Pérez, I., Kundu, S., Chandra, A., Craigo, K. E., Chernev, P., Kuhlmann, U., Dau, H., Hildebrandt, P., Greco, C., Van Stappen, C., Lehner, N. & Ray, K. (2017). *J. Am. Chem. Soc.* 139, 15033–15042.

Murrie, M., Teat, S. J., Stoeckli-Evans, H. & Güdel, H. U. (2003). *Angew. Chem. Int. Ed.* 42, 4653–4656.

Nesterov, D. S. & Nesterova, O. V. (2018). *Catalysts*, 8, 602–623.

Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* B69, 249–259.

Pattacini, R., Teo, P., Zhang, J., Lan, Y. H., Powell, A. K., Nehrkorn, J., Waldmann, O., Hor, T. S. A. & Braunstein, P. (2011). *Dalton Trans.* 40, 10526–10534.

Pinsky, M. & Avnir, D. (1998). *Inorg. Chem.* 37, 5575–5582.

Qin, Y., Zhang, H. F., Sun, H., Pan, Y. D., Ge, Y., Li, Y. H. & Zhang, Y. Q. (2017). *Chem. Asian J.* 12, 2834–2844.

Qin, Y. R., Jing, Y., Ge, Y., Liu, W., Li, Y. H. & Zhang, Y. Q. (2018). *Dalton Trans.* 47, 15197–15205.

Radu, I., Kravtsov, V. Ch., Ostrovsky, S. M., Reu, O. S., Krämer, K., Decurtins, S., Liu, S. X., Klokishner, S. I. & Baca, S. G. (2017). *Inorg. Chem.* 56, 2662–2676.

Sarto, C., Rouzières, M., Liu, J. L., Bamberger, H., van Slageren, J., Clérac, R. & Alborés, P. (2018). *Dalton Trans.* 47, 17055–17066.

Sheldrick, G. M. (2015). *Acta Cryst.* C71, 3–8.

Shi, Z. H., Huang, Y., Wu, Y. Z., Chen, X. L. & Yang, H. (2021). *Chin. J. Struct. Chem.* 40, 495–500.

Shiga, T. & Oshio, H. (2007). *Polyhedron*, 26, 1881–1884.

Shul’pin, G. B. & Shul’pina, L. S. (2021). *Catalysts*, 11, 186–223.

Spek, A. L. (2020). *Acta Cryst.* E76, 1–11.

Spek, A. L. (2015). *Acta Cryst.* C71, 9–18.

Tanaka, T., Kawase, M. & Tani, S. (2003). *Life Sci.* 73, 2985–2990.

Tudor, V., Madalan, A., Lupu, V., Lloret, F., Julve, M. & Andruh, M. (2010). *Inorg. Chim. Acta.* 363, 823–826.

Wang, J., Jian, F. F., Huang, B. X. & Bai, Z. S. (2013). *J. Solid State Chem.* 204, 272–277.

Zou, J. Y., Shi, W., Gao, H. L., Cui, J. Z. & Cheng, P. (2014). *Wuji Huaxue Xuebao*, 30, 149–154.
A mixed-valence \([\text{Co}^{II}_4\text{Co}^{III}_2]\) cluster with defect disk-shaped topology

Hua Yang, Yu-Pei Fu, Yuan Huang, Xiao-Li Chen, Dan Qiao and Hua-Li Cui

Computing details

Data collection: SAINT (Bruker, 2016); cell refinement: APEX2 (Bruker, 2016); data reduction: SAINT (Bruker, 2016); program(s) used to solve structure: olex2.solve (Bourhis et al., 2015); program(s) used to refine structure: SHELXL (Sheldrick, 2015); molecular graphics: OLEX2 (Dolomanov et al., 2009); software used to prepare material for publication: OLEX2 (Dolomanov et al., 2009) and PLATON (Spek, 2020).

Di-\(\mu_2\)-acetato-tetrakis(\(\mu_2\)-2-[4-chloro-2-oxidobenzylideneamino)methyl]phenolato)tetr-\(\mu_3\)-methanolato-tetracobalt(II)dicobalt(III)

Crystal data

\([\text{Co}_6(\text{C}_4\text{H}_8\text{Cl}\text{NO}_2)_4(\text{C}_2\text{H}_3\text{O}_2)_2(\text{CH}_3\text{O})_4]\)

\(D_\lambda = 1.538 \text{ Mg m}^{-3}\)

Mo \(K\alpha\) radiation, \(\lambda = 0.71073\) Å

Cell parameters from 9807 reflections

\(\theta = 2.3-25.0^\circ\)

\(\mu = 1.60 \text{ mm}^{-1}\)

\(T = 120 \text{ K}\)

Block, red

0.4 × 0.2 × 0.2 mm

Data collection

Bruker SMART APEXII diffractometer

Graphite monochromator

phi and \(\omega\) scans

Absorption correction: multi-scan (SADABS; Bruker, 2016)

\(T_{\text{min}} = 0.612, T_{\text{max}} = 0.746\)

78040 measured reflections

Refinement

Refinement on \(F^2\)

Least-squares matrix: full

\(R[F^2 > 2\sigma(F^2)] = 0.057\)

\(wR(F^2) = 0.174\)

\(S = 1.04\)

16170 reflections

853 parameters

12 restraints

Primary atom site location: iterative

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

\(w = 1/[\sigma(F^2) + (0.0906P)^2 + 7.497P]\)

where \(P = (F^2 + 2F_c^2)/3\)

\((\Delta\sigma)_{\text{max}} = 0.001\)

\(\Delta\rho_{\text{max}} = 0.65 \text{ e} \AA^{-3}\)

\(\Delta\rho_{\text{min}} = -0.78 \text{ e} \AA^{-3}\)

Absolute structure: Flack \(x\) determined using 4303 quotients \([(I+)-(I-)]/[(I+)+(I-)]\) (Parsons et al., 2013)

Absolute structure parameter: 0.011 (8)
**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.** SQUEEZE, constraints & restraints, applied to the data, 17 reflections OMITted

Data for 1 was collected on a Bruker SMART APEXII diffractometer equipped with a graphite monochromator utilizing Mo Kα radiation (λ = 0.71073). The crystal structure of 1 was solved with the OLEX2 program (Dolomanov et al., 2009) and refined by SHELXL package (Sheldrick, 2015). The crystal structure of 1 contained disordered solvent that could not be satisfactorily refined. The SQUEEZE routine of PLATON was used in the treatment of the crystallographic data.

CCDC-1991979 (1) contains the supplementary crystallographic data for this paper. The data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Centre, 12 Union Road, Cambridge CB21EZ, UK; Fax: (+44) 1223–336-033; or deposit@ccdc.cam.ac.uk). The crystallographic data was shown in Table 1. Selected bond lengths and bond angles of 1 are listed in Table 2.

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

|   | x     | y     | z     | Ueq  |
|---|-------|-------|-------|------|
| Co1 | 0.76532 (7) | 0.48030 (7) | 0.63042 (4) | 0.0272 (3) |
| Co2 | 0.75457 (8) | 0.37642 (7) | 0.53446 (4) | 0.0310 (3) |
| Co3 | 0.64711 (8) | 0.53340 (8) | 0.54249 (4) | 0.0308 (3) |
| Co4 | 0.60975 (9) | 0.60947 (8) | 0.63671 (4) | 0.0339 (3) |
| Co5 | 0.71075 (8) | 0.57569 (8) | 0.72480 (4) | 0.0309 (3) |
| Co6 | 0.88399 (8) | 0.48935 (8) | 0.72294 (4) | 0.0311 (3) |
| Cl1 | 0.27968 (19) | 0.4953 (3) | 0.70412 (13) | 0.0880 (13) |
| Cl2 | 0.6728 (4) | 0.94857 (19) | 0.58576 (13) | 0.0949 (15) |
| Cl3 | 1.0115 (2) | 0.2814 (2) | 0.34231 (10) | 0.0618 (8) |
| Cl4 | 1.0081 (2) | 0.2435 (2) | 0.91102 (11) | 0.0657 (9) |
| O1 | 0.8938 (4) | 0.4073 (4) | 0.7727 (2) | 0.0380 (15) |
| O2 | 0.8905 (4) | 0.4868 (4) | 0.6523 (2) | 0.0312 (13) |
| O3 | 0.8270 (4) | 0.5965 (4) | 0.7389 (2) | 0.0360 (15) |
| O4 | 0.5975 (4) | 0.5510 (4) | 0.7035 (2) | 0.0328 (14) |
| O5 | 0.8388 (5) | 0.3739 (4) | 0.4831 (2) | 0.0409 (16) |
| O6 | 0.7686 (4) | 0.3639 (4) | 0.6037 (2) | 0.0326 (14) |
| O7 | 0.6375 (4) | 0.4225 (4) | 0.5223 (2) | 0.0352 (15) |
| O8 | 0.6589 (4) | 0.6401 (4) | 0.5695 (2) | 0.0344 (14) |
| O9 | 0.5259 (4) | 0.5511 (4) | 0.5349 (2) | 0.0422 (16) |
| O10 | 0.4897 (5) | 0.6094 (5) | 0.6053 (2) | 0.0453 (17) |
| O11 | 0.5940 (6) | 0.7169 (4) | 0.6734 (2) | 0.052 (2) |
| O12 | 0.6775 (5) | 0.6864 (4) | 0.7377 (2) | 0.0432 (17) |
| O13 | 0.6353 (4) | 0.4952 (4) | 0.6070 (2) | 0.0298 (13) |
| O14 | 0.7492 (4) | 0.4674 (4) | 0.70863 (19) | 0.0289 (13) |
| O15 | 0.7654 (4) | 0.5070 (4) | 0.5537 (2) | 0.0288 (13) |
| O16 | 0.7373 (4) | 0.5983 (4) | 0.6593 (2) | 0.0301 (13) |
| N1 | 1.0097 (5) | 0.5200 (5) | 0.7269 (3) | 0.0342 (17) |
| N2 | 0.6850 (5) | 0.5540 (5) | 0.7906 (3) | 0.0364 (19) |
| N3 | 0.7266 (5) | 0.2548 (5) | 0.5280 (3) | 0.0352 (18) |
| N4 | 0.6619 (5) | 0.5716 (5) | 0.4782 (3) | 0.0364 (18) |
| C1 | 0.4468 (6) | 0.5296 (7) | 0.7069 (4) | 0.045 (3) |
| Atom | x   | y   | z    | Ueq  |
|------|-----|-----|------|------|
|  C1  | 0.4423 | 0.5343 | 0.6733 | 0.054* |
|  C2  | 0.3754 (7) | 0.5145 (9) | 0.7339 (4) | 0.060 (3) |
|  C3  | 0.3781 (7) | 0.5108 (8) | 0.7839 (4) | 0.053 (3) |
|  C4  | 0.3270 | 0.5027 | 0.8020 | 0.064* |
|  C5  | 0.4559 (7) | 0.5190 (7) | 0.8052 (4) | 0.049 (3) |
|  C6  | 0.5230 (6) | 0.5333 (6) | 0.7801 (4) | 0.039 (2) |
|  C7  | 0.6097 (7) | 0.5405 (6) | 0.8072 (3) | 0.038 (2) |
|  C8  | 0.6048 | 0.5348 | 0.8408 | 0.045* |
|  C9  | 0.7583 (7) | 0.5597 (6) | 0.8253 (3) | 0.036 (2) |
|  H8A | 0.7351 | 0.5577 | 0.8581 | 0.043* |
|  C10 | 0.7970 | 0.5117 | 0.8209 | 0.043* |
|  C11 | 0.8089 (7) | 0.6371 (6) | 0.8189 (3) | 0.038 (2) |
|  C12 | 0.8217 (8) | 0.6933 (7) | 0.8549 (4) | 0.048 (3) |
|  C13 | 0.7953 | 0.6842 | 0.8850 | 0.058* |
|  C14 | 0.8722 (8) | 0.7628 (7) | 0.8482 (4) | 0.052 (3) |
|  H12 | 0.8968 | 0.7219 (6) | 0.7656 (4) | 0.062* |
|  C15 | 0.9468 | 0.8234 | 0.7980 | 0.062* |
|  H13 | 0.9209 | 0.7312 | 0.7350 | 0.054* |
|  C16 | 0.8449 (7) | 0.6517 (6) | 0.7750 (3) | 0.039 (2) |
|  C17 | 0.9556 (7) | 0.3433 (7) | 0.8366 (4) | 0.044 (2) |
|  H15 | 0.9028 | 0.3052 | 0.8403 | 0.052* |
|  C18 | 0.9615 (6) | 0.3927 (6) | 0.8002 (3) | 0.035 (2) |
|  C19 | 1.0425 (6) | 0.4339 (6) | 0.7952 (3) | 0.035 (2) |
|  H18 | 1.0999 (7) | 0.3583 (8) | 0.8636 (4) | 0.051 (3) |
|  C20 | 1.1452 | 0.3475 | 0.8855 | 0.062* |
|  C21 | 1.1099 (7) | 0.4154 (7) | 0.8276 (4) | 0.047 (3) |
|  H19 | 1.1636 | 0.4432 | 0.8244 | 0.057* |
|  C22 | 1.0212 (7) | 0.3171 (7) | 0.8668 (4) | 0.044 (2) |
|  C23 | 1.0635 (6) | 0.4918 (6) | 0.7579 (3) | 0.035 (2) |
|  H21 | 1.1215 | 0.5106 | 0.7561 | 0.043* |
|  C24 | 1.0409 (7) | 0.5708 (7) | 0.6875 (3) | 0.040 (2) |
|  C25 | 1.1000 | 0.5907 | 0.6944 | 0.048* |
|  C26 | 1.0029 | 0.6194 | 0.6834 | 0.048* |
|  C27 | 1.0413 (6) | 0.5210 (7) | 0.6432 (3) | 0.039 (2) |
|  C28 | 1.1158 (7) | 0.5121 (6) | 0.6156 (4) | 0.042 (2) |
|  C29 | 1.1665 | 0.5412 | 0.6248 | 0.050* |
|  C30 | 1.1186 (7) | 0.4631 (7) | 0.5759 (3) | 0.044 (2) |
|  C31 | 1.1704 | 0.4593 | 0.5579 | 0.052* |
|  C32 | 1.0474 (7) | 0.4197 (7) | 0.5621 (4) | 0.043 (2) |
|  C33 | 1.0499 | 0.3850 | 0.5349 | 0.051* |
|  C34 | 0.9702 (6) | 0.4266 (6) | 0.5882 (3) | 0.036 (2) |
|  C35 | 0.9207 | 0.3960 | 0.5788 | 0.043* |
|  C36 | 0.9662 (6) | 0.4782 (6) | 0.6278 (3) | 0.034 (2) |
|  C37 | 0.8365 (6) | 0.2853 (6) | 0.6654 (3) | 0.037 (2) |
| Atom  | x      | y      | z      | Ueq  |
|-------|--------|--------|--------|------|
| H29   | 0.8709 | 0.3318 | 0.6734 | 0.044*|
| C30   | 0.8427 (7) | 0.2144 (6) | 0.6922 (4) | 0.043 (2) |
| H30   | 0.8797 | 0.2121 | 0.7192 | 0.052*|
| C31   | 0.7947 (7) | 0.1476 (7) | 0.6794 (4) | 0.048 (3) |
| H31   | 0.7971 | 0.0989 | 0.6981 | 0.057*|
| C32   | 0.7422 (7) | 0.1498 (6) | 0.6391 (4) | 0.045 (2) |
| H32   | 0.7105 | 0.1019 | 0.6304 | 0.054*|
| C33   | 0.7353 (6) | 0.2198 (6) | 0.6117 (3) | 0.033 (2) |
| C34   | 0.7802 (6) | 0.2900 (5) | 0.6267 (3) | 0.032 (2) |
| C35   | 0.6792 (6) | 0.2187 (6) | 0.5677 (3) | 0.037 (2) |
| H35A  | 0.6624 | 0.1614 | 0.5599 | 0.044*|
| H35B  | 0.6260 | 0.2512 | 0.5733 | 0.044*|
| C36   | 0.7550 (6) | 0.2086 (6) | 0.4935 (3) | 0.036 (2) |
| H36   | 0.7351 | 0.1532 | 0.4929 | 0.044*|
| C37   | 0.8119 (6) | 0.2338 (6) | 0.4560 (3) | 0.036 (2) |
| C38   | 0.8302 (7) | 0.1754 (7) | 0.4205 (3) | 0.043 (2) |
| H38   | 0.8014 | 0.1236 | 0.4217 | 0.052*|
| C39   | 0.8873 (7) | 0.1895 (7) | 0.3846 (3) | 0.047 (3) |
| H39   | 0.8966 | 0.1497 | 0.3603 | 0.056*|
| C40   | 0.9318 (7) | 0.2643 (8) | 0.3847 (3) | 0.047 (3) |
| C41   | 0.9149 (7) | 0.3239 (7) | 0.4177 (4) | 0.042 (2) |
| H41   | 0.9455 | 0.3745 | 0.4162 | 0.050*|
| C42   | 0.8532 (6) | 0.3123 (6) | 0.4540 (3) | 0.035 (2) |
| C43   | 0.5196 (6) | 0.3442 (7) | 0.4902 (4) | 0.042 (2) |
| H43   | 0.5135 | 0.3150 | 0.5194 | 0.051*|
| C44   | 0.5833 (6) | 0.4050 (6) | 0.4854 (3) | 0.035 (2) |
| C45   | 0.4645 (7) | 0.3265 (8) | 0.4515 (4) | 0.055 (3) |
| H45   | 0.4230 | 0.2835 | 0.4535 | 0.066*|
| C46   | 0.4723 (7) | 0.3731 (9) | 0.4106 (4) | 0.056 (3) |
| H46   | 0.4342 | 0.3632 | 0.3847 | 0.068*|
| C47   | 0.5346 (8) | 0.4342 (8) | 0.4064 (4) | 0.053 (3) |
| H47   | 0.5384 | 0.4660 | 0.3781 | 0.064*|
| C48   | 0.5921 (7) | 0.4488 (6) | 0.4441 (3) | 0.040 (2) |
| C49   | 0.6648 (7) | 0.5090 (6) | 0.4405 (3) | 0.041 (2) |
| H49A  | 0.7203 | 0.4788 | 0.4425 | 0.049*|
| H49B  | 0.6624 | 0.5367 | 0.4092 | 0.049*|
| C50   | 0.6674 (6) | 0.6489 (6) | 0.4657 (3) | 0.036 (2) |
| H50   | 0.6716 | 0.6603 | 0.4327 | 0.043*|
| C51   | 0.6675 (6) | 0.7168 (6) | 0.4969 (3) | 0.036 (2) |
| C52   | 0.6742 (7) | 0.7961 (6) | 0.4756 (4) | 0.045 (3) |
| H52   | 0.6781 | 0.7998 | 0.4420 | 0.054*|
| C53   | 0.6754 (8) | 0.8669 (7) | 0.5016 (4) | 0.054 (3) |
| H53   | 0.6802 | 0.9192 | 0.4866 | 0.064*|
| C54   | 0.6692 (8) | 0.8603 (7) | 0.5515 (4) | 0.051 (3) |
| C55   | 0.6638 (8) | 0.7861 (6) | 0.5741 (4) | 0.045 (3) |
| H55   | 0.6612 | 0.7836 | 0.6078 | 0.055*|
| C56   | 0.6622 (6) | 0.7123 (6) | 0.5466 (3) | 0.036 (2) |
| C57   | 0.4733 (7) | 0.5833 (8) | 0.5639 (4) | 0.053 (3) |
Atomic displacement parameters (Å²)

|     | $U^{11}$  | $U^{22}$  | $U^{33}$  | $U^{12}$  | $U^{13}$  | $U^{23}$  |
|-----|-----------|-----------|-----------|-----------|-----------|-----------|
| Co1 | 0.0306 (6) | 0.0269 (6) | 0.0242 (6) | 0.0002 (5) | −0.0015 (5) | −0.0026 (5) |
| Co2 | 0.0346 (6) | 0.0300 (6) | 0.0283 (6) | 0.0024 (5) | −0.0027 (5) | −0.0032 (5) |
| Co3 | 0.0349 (6) | 0.0329 (7) | 0.0247 (6) | 0.0038 (5) | −0.0031 (5) | −0.0006 (5) |
| Co4 | 0.0406 (7) | 0.0336 (6) | 0.0275 (6) | 0.0086 (6) | 0.0001 (5)  | −0.0009 (5) |
| Co5 | 0.0384 (7) | 0.0304 (6) | 0.0240 (6) | 0.0010 (5) | 0.0017 (5)  | −0.0028 (5) |
| Co6 | 0.0315 (6) | 0.0358 (6) | 0.0261 (6) | −0.0023 (5) | −0.0015 (5) | −0.0027 (5) |
| Cl1 | 0.0342 (14)| 0.163 (4)  | 0.067 (2)  | 0.013 (2)  | 0.0036 (13)| 0.009 (2)  |
| Cl2 | 0.189 (5)  | 0.0361 (16)| 0.060 (2)  | −0.007 (2) | 0.005 (2)  | −0.0041 (14)|
|   | 0.045 (6) | 0.051 (6) | 0.033 (5) | -0.001 (5) | 0.004 (4) | -0.006 (5) |
|---|-----------|-----------|-----------|------------|-----------|------------|
|   | 0.055 (7) | 0.056 (7) | 0.029 (5) | 0.013 (6)  | -0.004 (5) | -0.006 (5) |
|   | 0.049 (6) | 0.067 (8) | 0.024 (5) | 0.019 (6)  | 0.002 (4)  | 0.011 (5)  |
|   | 0.044 (6) | 0.040 (6) | 0.041 (6) | 0.002 (5)  | 0.007 (5)  | 0.009 (5)  |
|   | 0.036 (5) | 0.040 (5) | 0.031 (5) | 0.011 (4)  | 0.002 (4)  | 0.000 (4)  |
|   | 0.037 (5) | 0.049 (6) | 0.041 (6) | -0.009 (5) | -0.010 (4) | -0.004 (5) |
|   | 0.031 (5) | 0.041 (5) | 0.031 (5) | 0.003 (4)  | -0.005 (4) | -0.010 (4) |
|   | 0.038 (6) | 0.074 (8) | 0.055 (7) | -0.007 (6) | -0.007 (5) | -0.021 (6) |
|   | 0.046 (6) | 0.094 (10)| 0.029 (5) | -0.016 (7) | -0.014 (5) | -0.016 (6) |
|   | 0.059 (7) | 0.072 (8) | 0.028 (5) | 0.015 (6)  | -0.010 (5) | -0.010 (5) |
|   | 0.048 (6) | 0.047 (6) | 0.024 (4) | 0.004 (5)  | -0.001 (4) | -0.007 (4) |
|   | 0.063 (6) | 0.038 (5) | 0.021 (4) | 0.006 (5)  | -0.002 (4) | 0.000 (4)  |
|   | 0.033 (5) | 0.041 (5) | 0.034 (5) | 0.003 (4)  | 0.001 (4)  | 0.004 (4)  |
|   | 0.037 (5) | 0.044 (6) | 0.027 (5) | 0.004 (4)  | -0.002 (4) | 0.006 (4)  |
|   | 0.056 (6) | 0.042 (6) | 0.037 (5) | -0.010 (5) | -0.011 (5) | 0.012 (5)  |
|   | 0.075 (8) | 0.036 (6) | 0.049 (7) | -0.010 (6) | -0.003 (6) | 0.011 (5)  |
|   | 0.072 (8) | 0.041 (6) | 0.041 (6) | -0.003 (6) | -0.005 (5) | 0.004 (5)  |
|   | 0.071 (7) | 0.030 (5) | 0.035 (5) | 0.000 (5)  | -0.009 (5) | 0.004 (4)  |
|   | 0.040 (5) | 0.033 (5) | 0.034 (5) | 0.013 (4)  | -0.002 (4) | 0.010 (4)  |
|   | 0.047 (6) | 0.071 (8) | 0.040 (6) | 0.028 (6)  | 0.000 (5)  | 0.003 (6)  |
|   | 0.039 (7) | 0.162 (15)| 0.087 (10)| 0.037 (9)  | -0.015 (7) | -0.024 (11)|
|   | 0.121 (12)| 0.038 (6) | 0.027 (5) | 0.021 (7)  | 0.005 (6)  | -0.002 (4) |
|   | 0.098 (5) | 0.091 (5) | 0.092 (5) | 0.004 (3)  | -0.001 (3) | -0.001 (3) |
|   | 0.032 (5) | 0.040 (5) | 0.038 (5) | -0.007 (4) | -0.002 (4) | 0.001 (4)  |
|   | 0.033 (5) | 0.034 (5) | 0.041 (5) | -0.004 (4) | 0.005 (4)  | 0.003 (4)  |
|   | 0.050 (6) | 0.026 (5) | 0.035 (5) | -0.005 (4) | -0.003 (4) | 0.001 (4)  |
|   | 0.043 (5) | 0.040 (5) | 0.038 (5) | -0.011 (4) | -0.010 (4) | 0.011 (4)  |

**Geometric parameters (Å, °)**

|   | 2.036 (6) | C16—C17 | 1.428 (14) |
|---|-----------|----------|------------|
|   | 2.031 (6) | C17—C19 | 1.416 (14) |
|   | 2.132 (6) | C17—C21 | 1.444 (13) |
|   | 2.222 (5) | C18—H18 | 0.9500     |
|   | 2.201 (6) | C18—C19 | 1.381 (15) |
|   | 2.123 (6) | C18—C20 | 1.393 (16) |
|   | 1.945 (7) | C19—H19 | 0.9500     |
|   | 1.969 (6) | C21—H21 | 0.9500     |
|   | 1.991 (6) | C22—H22A| 0.9900     |
|   | 2.191 (6) | C22—H22B| 0.9900     |
|   | 2.021 (8) | C22—C23 | 1.482 (14) |
|   | 1.890 (7) | C23—C24 | 1.399 (14) |
|   | 1.898 (6) | C23—C28 | 1.422 (13) |
|   | 1.910 (7) | C24—H24 | 0.9500     |
|   | 1.925 (6) | C24—C25 | 1.372 (14) |
|   | 1.907 (6) | C25—H25 | 0.9500     |
|   | 1.923 (8) | C25—C26 | 1.365 (15) |
|   | 2.111 (6) | C26—H26 | 0.9500     |
| Bond               | Length (Å) | Bond               | Length (Å) |
|--------------------|------------|--------------------|------------|
| Co4—O8             | 2.097 (6)  | C26—C27            | 1.408 (13) |
| Co4—O10            | 2.057 (7)  | C27—H27            | 0.9500     |
| Co4—O11            | 2.039 (7)  | C27—C28            | 1.396 (13) |
| Co4—O13            | 2.070 (6)  | C29—H29            | 0.9500     |
| Co4—O16            | 2.083 (6)  | C29—C30            | 1.378 (14) |
| Co5—O3             | 1.874 (7)  | C29—C34            | 1.397 (13) |
| Co5—O4             | 1.897 (6)  | C30—H30            | 0.9500     |
| Co5—O12            | 1.902 (7)  | C30—C31            | 1.362 (15) |
| Co5—O14            | 1.909 (6)  | C31—H31            | 0.9500     |
| Co5—O16            | 1.921 (6)  | C31—C32            | 1.394 (15) |
| Co5—N2             | 1.924 (8)  | C32—H32            | 0.9500     |
| Co6—O1             | 1.937 (6)  | C32—C33            | 1.376 (13) |
| Co6—O2             | 1.989 (6)  | C33—C34            | 1.399 (13) |
| Co6—O3             | 2.000 (7)  | C33—C35            | 1.511 (13) |
| Co6—O14            | 2.156 (6)  | C35—H35A           | 0.9900     |
| Co6—N1             | 2.012 (8)  | C35—H35B           | 0.9900     |
| Cl1—C2             | 1.731 (12) | C36—H36            | 0.9500     |
| Cl2—C54            | 1.727 (11) | C36—C37            | 1.433 (14) |
| Cl3—C40            | 1.738 (11) | C37—C38            | 1.404 (13) |
| Cl4—C20            | 1.734 (11) | C37—C42            | 1.425 (14) |
| O1—C16             | 1.324 (11) | C38—H38            | 0.9500     |
| O2—C28             | 1.367 (10) | C38—C39            | 1.362 (15) |
| O3—C14             | 1.380 (11) | C39—H39            | 0.9500     |
| O4—C6              | 1.322 (11) | C39—C40            | 1.394 (16) |
| O5—C42             | 1.311 (11) | C40—C41            | 1.366 (15) |
| O6—C34             | 1.373 (10) | C41—H41            | 0.9500     |
| O7—C44             | 1.364 (10) | C41—C42            | 1.410 (13) |
| O8—C56             | 1.336 (11) | C43—H43            | 0.9500     |
| O9—C57             | 1.265 (12) | C43—C44            | 1.400 (14) |
| O10—C57            | 1.264 (13) | C43—C45            | 1.414 (14) |
| O11—C59            | 1.268 (13) | C44—C48            | 1.369 (13) |
| O12—C59            | 1.265 (14) | C45—H45            | 0.9500     |
| O13—C61            | 1.417 (11) | C45—C46            | 1.381 (17) |
| O14—C62            | 1.432 (10) | C46—H46            | 0.9500     |
| O15—C64            | 1.447 (11) | C46—C47            | 1.387 (17) |
| O16—C63            | 1.428 (11) | C47—H47            | 0.9500     |
| N1—C21             | 1.290 (12) | C47—C48            | 1.405 (14) |
| N1—C22             | 1.462 (12) | C48—C49            | 1.493 (15) |
| N2—C7              | 1.274 (12) | C49—H49A           | 0.9900     |
| N2—C8              | 1.500 (12) | C49—H49B           | 0.9900     |
| N3—C35             | 1.473 (12) | C50—H50            | 0.9500     |
| N3—C36             | 1.294 (11) | C50—C51            | 1.407 (14) |
| N4—C49             | 1.469 (12) | C51—C52            | 1.420 (14) |
| N4—C50             | 1.304 (13) | C51—C56            | 1.403 (13) |
| C1—H1              | 0.9500     | C52—H52            | 0.9500     |
| C1—C2              | 1.363 (15) | C52—C53            | 1.361 (15) |
| C1—C6              | 1.426 (14) | C53—H53            | 0.9500     |
| C2—C3              | 1.407 (16) | C53—C54            | 1.408 (15) |
| Bond     | Length (Å) | Bond     | Length (Å) |
|----------|------------|----------|------------|
| C3—H3    | 0.9500     | C54—C55  | 1.364 (14) |
| C3—C4    | 1.352 (16) | C55—H55  | 0.9500     |
| C4—H4    | 0.9500     | C55—C56  | 1.424 (14) |
| C4—C5    | 1.393 (14) | C57—C58  | 1.509 (16) |
| C5—C6    | 1.411 (14) | C58—H58A | 0.9800     |
| C5—C7    | 1.431 (14) | C58—H58B | 0.9800     |
| C7—H7    | 0.9500     | C58—H58C | 0.9800     |
| C8—H8A   | 0.9900     | C59—C60  | 1.507 (19) |
| C8—H8B   | 0.9900     | C60—H60A | 0.9800     |
| C8—C9    | 1.490 (14) | C60—H60B | 0.9800     |
| C9—C10   | 1.375 (14) | C60—H60C | 0.9800     |
| C9—C14   | 1.377 (13) | C61—H61A | 0.9800     |
| C10—H10  | 0.9500     | C61—H61B | 0.9800     |
| C10—C11  | 1.385 (16) | C61—H61C | 0.9800     |
| C11—H11  | 0.9500     | C62—H62A | 0.9800     |
| C11—C12  | 1.428 (16) | C62—H62B | 0.9800     |
| C12—H12  | 0.9500     | C62—H62C | 0.9800     |
| C12—C13  | 1.388 (14) | C63—H63A | 0.9800     |
| C13—H13  | 0.9500     | C63—H63B | 0.9800     |
| C13—C14  | 1.417 (14) | C63—H63C | 0.9800     |
| C15—H15  | 0.9500     | C64—H64A | 0.9800     |
| C15—C16  | 1.396 (14) | C64—H64B | 0.9800     |
| C15—C20  | 1.354 (14) | C64—H64C | 0.9800     |

| Bond     | Angle (°)  |
|----------|------------|
| O2—Col1—O13 | 170.5 (2) |
| O2—Col1—O14 | 79.2 (2)  |
| O2—Col1—O15 | 106.6 (2) |
| O2—Col1—O16 | 91.9 (2)  |
| O6—Col1—O2  | 97.8 (3)  |
| O6—Col1—O13 | 90.8 (2)  |
| O6—Col1—O14 | 106.3 (2) |
| O6—Col1—O15 | 79.6 (2)  |
| O6—Col1—O16 | 169.5 (3) |
| O13—Col1—O14 | 102.1 (2) |
| O13—Col1—O15 | 71.1 (2)  |
| O15—Col1—O14 | 171.3 (2) |
| O16—Col1—O13 | 79.8 (2)  |
| O16—Col1—O14 | 71.5 (2)  |
| O16—Col1—O15 | 101.4 (2) |
| O5—Col2—O6  | 131.1 (3) |
| O5—Col2—O7  | 119.4 (3) |
| O5—Col2—O15 | 98.7 (3)  |
| O5—Col2—N3  | 92.7 (3)  |
| O6—Col2—O7  | 108.0 (3) |
| O6—Col2—O15 | 81.2 (2)  |
| O6—Col2—N3  | 90.6 (3)  |
| O7—Col2—O15 | 75.5 (2)  |
| O7—Col2—N3  | 99.8 (3)  |

*Acta Cryst.* (2022). *C78*, 488-492
| Bond          | Bond Angle (°) |
|--------------|---------------|
| N3—Co2—O15  | 168.6 (3)     |
| O7—Co3—O8   | 173.8 (3)     |
| O7—Co3—O9   | 91.8 (3)      |
| O7—Co3—O13  | 88.2 (3)      |
| O7—Co3—O15  | 84.9 (3)      |
| O7—Co3—N4   | 91.9 (3)      |
| O8—Co3—O9   | 90.1 (3)      |
| O8—Co3—O13  | 85.7 (3)      |
| O8—Co3—O15  | 92.7 (3)      |
| O8—Co3—N4   | 94.0 (3)      |
| O9—Co3—O13  | 93.4 (3)      |
| O9—Co3—N4   | 88.0 (3)      |
| O15—Co3—O9  | 174.6 (3)     |
| O15—Co3—O13 | 82.2 (2)      |
| N4—Co3—O13  | 178.6 (3)     |
| O8—Co4—O4   | 160.1 (2)     |
| O10—Co4—O4  | 107.4 (3)     |
| O10—Co4—O8  | 86.6 (3)      |
| O10—Co4—O13 | 89.9 (3)      |
| O10—Co4—O16 | 170.9 (3)     |
| O11—Co4—O4  | 85.6 (3)      |
| O11—Co4—O8  | 107.3 (3)     |
| O11—Co4—O10 | 96.3 (3)      |
| O11—Co4—O13 | 172.5 (3)     |
| O11—Co4—O16 | 91.9 (3)      |
| O13—Co4—O4  | 88.5 (2)      |
| O13—Co4—O8  | 77.2 (2)      |
| O13—Co4—O16 | 82.2 (2)      |
| O16—Co4—O4  | 77.0 (2)      |
| O16—Co4—O8  | 87.2 (3)      |
| O3—Co5—O4   | 173.4 (3)     |
| O3—Co5—O12  | 92.8 (3)      |
| O3—Co5—O14  | 85.2 (3)      |
| O3—Co5—O16  | 87.9 (3)      |
| O3—Co5—N2   | 91.6 (3)      |
| O4—Co5—O12  | 90.5 (3)      |
| O4—Co5—O14  | 91.1 (3)      |
| O4—Co5—O16  | 86.3 (3)      |
| O4—Co5—N2   | 94.2 (3)      |
| O12—Co5—O14 | 176.1 (3)     |
| O12—Co5—O16 | 93.4 (3)      |
| O12—Co5—N2  | 86.2 (3)      |
| O14—Co5—O16 | 83.1 (2)      |
| O14—Co5—N2  | 97.2 (3)      |
| O16—Co5—N2  | 179.4 (3)     |
| O1—Co6—O2   | 134.7 (3)     |
| O1—Co6—O3   | 118.0 (3)     |
| Bond/Angle | Degrees/Adjs |
|------------|--------------|
| O1—Co6—O14 | 95.6 (2) C39—C38—C37 | 122.9 (11) |
| O1—Co6—N1  | 93.1 (3) C39—C38—H36 | 117.2 (9)  |
| O2—Co6—O3  | 105.3 (3) N3—C36—C37 | 125.5 (9)  |
| O2—Co6—O14 | 81.9 (2) C37—C36—H36 | 117.2 (9)  |
| O2—Co6—N1  | 90.6 (3) C38—C37—C36 | 116.9 (9)  |
| O3—Co6—O14 | 76.0 (2) C38—C37—C42 | 119.0 (9)  |
| O3—Co6—N1  | 101.5 (3) C42—C37—C36 | 124.0 (8)  |
| N1—Co6—O14 | 171.1 (3) C37—C38—H38 | 118.5 (9)  |
| C16—O1—Co6 | 127.4 (6) C39—C38—C37 | 117.8 (10) |
| Co6—O2—Co1 | 104.8 (3) C39—C38—H36 | 117.8 (10) |
| C28—O2—Co1 | 131.3 (5) C38—C39—H39 | 121.1 (9)  |
| C28—O2—Co6 | 123.2 (5) C38—C39—C40 | 117.8 (10) |
| C34—O3—Co6 | 102.7 (3) C40—C39—C41 | 121.4 (9)  |
| C14—O3—Co5 | 117.8 (6) C39—C40—C13 | 119.3 (9)  |
| C14—O3—Co6 | 129.8 (6) C41—C40—C13 | 119.3 (9)  |
| Co5—O4—Co4 | 95.9 (3) C41—C40—C39 | 121.4 (9)  |
| C6—O4—Co4  | 130.2 (6) C40—C41—H41 | 119.1 (9)  |
| C6—O4—Co5  | 127.2 (6) C40—C41—C42 | 121.9 (9)  |
| C42—O5—Co2 | 126.4 (6) C42—C41—H41 | 119.1 (9)  |
| Co2—O6—Co1 | 105.5 (3) O5—C42—C37 | 125.5 (9)  |
| C34—O6—Co1 | 129.8 (5) O5—C42—C41 | 117.8 (9)  |
| C34—O6—Co2 | 124.7 (5) C41—C42—C37 | 116.8 (9)  |
| Co3—O7—Co2 | 103.5 (3) C44—C43—H43 | 120.2 (9)  |
| C44—O7—Co2 | 127.8 (6) C44—C43—C45 | 119.5 (10) |
| C44—O7—Co3 | 118.3 (6) C45—C43—H43 | 120.2 (9)  |
| Co3—O8—Co4 | 96.3 (3) O7—C44—C43 | 120.5 (9)  |
| C56—O8—Co3 | 127.6 (6) O7—C44—C48 | 118.4 (9)  |
| C56—O8—Co4 | 130.9 (6) C48—C44—C43 | 121.2 (9)  |
| C57—O9—Co3 | 128.6 (7) C43—C45—H45 | 120.7 (9)  |
| C57—O10—Co4 | 125.2 (7) C46—C45—C43 | 118.6 (11) |
| C59—O11—Co4 | 124.4 (7) C46—C45—H45 | 120.7 (9)  |
| C59—O12—Co5 | 127.7 (7) C45—C46—H46 | 119.3 (9)  |
| Co3—O13—Co1 | 103.7 (3) C45—C46—C47 | 121.5 (10) |
| Co3—O13—Co4 | 96.3 (3) C47—C46—H46 | 119.3 (9)  |
| Co4—O13—Co1 | 99.1 (2) C46—C47—H47 | 120.1 (9)  |
| C61—O13—Co1 | 117.1 (5) C46—C47—C48 | 119.8 (11) |
| C61—O13—Co3 | 119.2 (5) C48—C47—H47 | 120.1 (9)  |
| C61—O13—Co4 | 117.6 (5) C44—C48—C47 | 119.3 (10) |
| Co5—O14—Co1 | 100.6 (2) C44—C48—C49 | 118.1 (8)  |
| Co5—O14—Co6 | 96.1 (2) C47—C48—C49 | 122.5 (10) |
| Co6—O14—Co1 | 93.5 (2) N4—C49—C48 | 112.3 (8)  |
| C62—O14—Co1 | 123.3 (5) N4—C49—H49A | 109.1 (9)  |
| C62—O14—Co5 | 117.1 (5) N4—C49—H49B | 109.1 (9)  |
| C62—O14—Co6 | 120.7 (5) C48—C49—H49A | 109.1 (9)  |
| Co2—O15—Co1 | 93.0 (2) C48—C49—H49B | 109.1 (9)  |
| Co3—O15—Co1 | 101.8 (3) H49A—C49—H49B | 107.9 (9)  |
| Co3—O15—Co2 | 95.9 (2) N4—C50—H50 | 117.1 (9)  |
| C64—O15—Co1 | 121.5 (5) N4—C50—C51 | 125.8 (9)  |
| Bond                  | Angle (°) (ESD) | Bond                  | Angle (°) (ESD) | Bond                  | Angle (°) (ESD) |
|----------------------|----------------|----------------------|----------------|----------------------|----------------|
| C64—O15—Co2         | 120.3 (6)      | C51—C50—H50         | 117.1          |                     |                |
| C64—O15—Co3         | 118.6 (5)      | C50—C51—C52         | 116.6 (8)      |                     |                |
| Co4—O16—Co1         | 99.0 (2)       | C56—C51—C50         | 125.4 (9)      |                     |                |
| Co5—O16—Co1         | 103.8 (3)      | C56—C51—C52         | 118.1 (9)      |                     |                |
| Co5—O16—Co4         | 96.1 (3)       | C51—C52—H52         | 118.7          |                     |                |
| C63—O16—Co1         | 117.3 (5)      | C53—C52—C51         | 122.6 (10)     |                     |                |
| C63—O16—Co4         | 118.0 (5)      | C53—C52—H52         | 118.7          |                     |                |
| C63—O16—Co5         | 118.9 (5)      | C52—C53—H53         | 121.0          |                     |                |
| C21—N1—Co6          | 125.1 (7)      | C52—C53—C54         | 117.9 (10)     |                     |                |
| C21—N1—C22          | 119.9 (8)      | C54—C53—H53         | 121.0          |                     |                |
| C22—N1—Co6          | 114.6 (6)      | C53—C54—C12         | 119.3 (9)      |                     |                |
| C7—N2—Co5           | 125.1 (7)      | C55—C54—C12         | 118.2 (8)      |                     |                |
| C7—N2—C8            | 117.6 (8)      | C55—C54—C53         | 122.4 (10)     |                     |                |
| C8—N2—Co5           | 117.1 (6)      | C54—C55—H55         | 120.3          |                     |                |
| C35—N3—Co2          | 115.0 (6)      | C54—C55—C56         | 119.3 (9)      |                     |                |
| C36—N3—Co2          | 124.7 (7)      | C56—C55—H55         | 120.3          |                     |                |
| C36—N3—C35          | 120.2 (8)      | O8—C56—C51          | 121.9 (9)      |                     |                |
| C49—N4—Co3          | 117.3 (6)      | O8—C56—C55          | 118.4 (8)      |                     |                |
| C50—N4—Co3          | 124.8 (7)      | C51—C56—C55         | 119.7 (9)      |                     |                |
| C50—N4—C49          | 117.9 (8)      | O9—C57—C58          | 115.4 (11)     |                     |                |
| C2—C1—H1            | 120.6          | O10—C57—O9          | 126.9 (10)     |                     |                |
| C2—C1—C6            | 118.7 (9)      | O10—C57—C58         | 117.7 (10)     |                     |                |
| C6—C1—H1            | 120.6          | C57—C58—H58A        | 109.5          |                     |                |
| C1—C2—C11           | 117.2 (9)      | C57—C58—H58B        | 109.5          |                     |                |
| C1—C2—C3            | 122.8 (11)     | C57—C58—H58C        | 109.5          |                     |                |
| C3—C2—C11           | 120.0 (8)      | H58A—C58—H58B       | 109.5          |                     |                |
| C2—C3—H3            | 121.2          | H58A—C58—H58C       | 109.5          |                     |                |
| C4—C3—C2            | 117.6 (10)     | H58B—C58—H58C       | 109.5          |                     |                |
| C4—C3—H3            | 121.2          | O11—C59—C60         | 116.9 (12)     |                     |                |
| C3—C4—H4            | 118.5          | O12—C59—O11         | 127.9 (10)     |                     |                |
| C3—C4—C5            | 123.0 (10)     | O12—C59—C60         | 115.1 (11)     |                     |                |
| C5—C4—H4            | 118.5          | C59—C60—H60A        | 109.5          |                     |                |
| C4—C5—C6            | 118.7 (9)      | C59—C60—H60B        | 109.5          |                     |                |
| C4—C5—C7            | 117.1 (9)      | C59—C60—H60C        | 109.5          |                     |                |
| C6—C5—C7            | 124.2 (9)      | H60A—C60—H60B       | 109.5          |                     |                |
| O4—C6—C1            | 118.4 (9)      | H60A—C60—H60C       | 109.5          |                     |                |
| O4—C6—C5            | 122.6 (9)      | H60B—C60—H60C       | 109.5          |                     |                |
| C5—C6—C1            | 119.0 (8)      | O13—C61—H61A        | 109.5          |                     |                |
| N2—C7—C5            | 126.0 (9)      | O13—C61—H61B        | 109.5          |                     |                |
| N2—C7—H7            | 117.0          | O13—C61—H61C        | 109.5          |                     |                |
| C5—C7—H7            | 117.0          | H61A—C61—H61B       | 109.5          |                     |                |
| N2—C8—H8A           | 109.2          | H61A—C61—H61C       | 109.5          |                     |                |
| N2—C8—H8B           | 109.2          | H61B—C61—H61C       | 109.5          |                     |                |
| H8A—C8—H8B          | 107.9          | O14—C62—H62A        | 109.5          |                     |                |
| C9—C8—N2            | 111.9 (8)      | O14—C62—H62B        | 109.5          |                     |                |
| C9—C8—H8A           | 109.2          | O14—C62—H62C        | 109.5          |                     |                |
| C9—C8—H8B           | 109.2          | H62A—C62—H62B       | 109.5          |                     |                |
| C10—C9—C8           | 123.0 (9)      | H62A—C62—H62C       | 109.5          |                     |                |
C10—C9—C14 119.2 (10) H62B—C62—H62C 109.5
C14—C9—C8 117.7 (8) O16—C63—H63A 109.5
C9—C10—H10 119.4 O16—C63—H63B 109.5
C9—C10—C11 121.3 (10) O16—C63—H63C 109.5
C10—C11—H11 120.4 H63B—C63—H63C 109.5
C10—C11—C12 119.2 (10) H63A—C63—H63C 109.5
C12—C11—H12 119.8 O15—C64—H64B 109.5
C13—C12—C11 120.4 (11) O15—C64—H64C 109.5
C13—C12—H12 119.8 H64A—C64—H64B 109.5
C12—C13—H13 121.2 H64A—C64—H64C 109.5
C12—C13—C14 117.6 (10) H64B—C64—H64C 109.5

Co1—O2—C28—C23 150.5 (7) C4—C5—C6—C1 1.2 (15)
Co1—O2—C28—C27 −30.5 (12) C4—C5—C7—N2 1.8 (17)
Co1—O6—C34—C29 −36.0 (12) O15—C64—H64A 178.7 (9)
Co1—O6—C34—C33 143.4 (7) C4—C5—C7—N2 −179.6 (10)
Co2—O5—C42—C37 −2.7 (13) C6—C1—C2—C3 3.1 (19)
Co2—O5—C42—C41 177.4 (7) C6—C1—C2—Cl1 1.5 (16)
Co2—O6—C34—C29 142.0 (7) C6—C1—C2—C3 179.8 (10)
Co2—O6—C34—C33 −38.6 (11) C6—C1—C2—C3 174.2 (9)
Co2—O7—C44—C43 94.2 (10) C6—C1—C2—C3 1.8 (17)
Co2—O7—C44—C48 −86.5 (10) C6—C1—C2—C3 1.8 (17)
Co2—O7—C44—C47 −67.2 (9) C6—C1—C2—C3 179.8 (10)
Co2—N3—C35—C33 4.8 (14) C6—C1—C2—C3 179.8 (10)
Co3—O7—C44—C43 −126.8 (8) C6—C1—C2—C3 179.8 (10)
Co3—O7—C44—C48 52.4 (10) C6—C1—C2—C3 179.8 (10)
Co3—O8—C56—C55 −7.8 (13) C6—C1—C2—C3 179.8 (10)
Co3—O8—C56—C51 174.7 (7) C6—C1—C2—C3 179.8 (10)
Co3—O9—C57—O10 −1.9 (19) C6—C1—C2—C3 179.8 (10)
Co3—O9—C57—C58 178.5 (11) C6—C1—C2—C3 179.8 (10)
Co3—N4—C49—C48 49.1 (10) C6—C1—C2—C3 179.8 (10)
Co3—N4—C50—C51 3.2 (14) C6—C1—C2—C3 179.8 (10)
Co4—O4—C6—C1 28.7 (13) C6—C1—C2—C3 179.8 (10)
Co4—O4—C6—C5 −150.4 (8) C6—C1—C2—C3 179.8 (10)
Co4—O8—C56—C51 −156.0 (7) C6—C1—C2—C3 179.8 (10)
Co4—O8—C56—C55 26.5 (13) C6—C1—C2—C3 179.8 (10)
Co4—O10—C57—O9 2.0 (19) C6—C1—C2—C3 179.8 (10)
Co4—O10—C57—C58 −178.4 (11) C6—C1—C2—C3 179.8 (10)
Co4—O11—C59—O12 7.2 (2) C6—C1—C2—C3 179.8 (10)
Co4—O11—C59—C60 −177.1 (10) C6—C1—C2—C3 179.8 (10)
Co5—O3—C14—C9 55.1 (11) C6—C1—C2—C3 179.8 (10)
Co5—O3—C14—C13 −123.0 (9) C6—C1—C2—C3 179.8 (10)
Co5—O4—C6—C1 172.9 (7) C6—C1—C2—C3 179.8 (10)
Co5—O4—C6—C5 −6.2 (13) C6—C1—C2—C3 179.8 (10)
Co5—O12—C59—O11 −12 (2) C6—C1—C2—C3 179.8 (10)
Co5—O12—C59—C60 172.4 (10) C6—C1—C2—C3 179.8 (10)
Co5—N2—C7—C5        4.8 (15)        C22—N1—C21—C17     −172.7 (9)
Co5—N2—C8—C9        48.4 (10)       C22—C23—C24—C25     176.7 (9)
Co6—O1—C16—C15     175.9 (7)       C22—C23—C28—O2       4.2 (14)
Co6—O1—C16—C17     −4.0 (13)        C22—C23—C28—C27     −174.8 (9)
Co6—O2—C28—C23     −41.1 (11)       C23—C24—C25—C26     −0.8 (15)
Co6—O2—C28—C27     137.9 (7)        C24—C23—C28—O2       −177.7 (8)
Co6—O3—C14—C9     −84.4 (11)       C24—C23—C28—C27     3.3 (14)
Co6—O3—C14—C13    97.5 (10)         C24—C25—C26—C27     1.2 (15)
Co6—N1—C21—C17     −0.4 (14)        C25—C26—C27—C28     0.7 (15)
Co6—N1—C22—C23     −68.5 (9)        C26—C27—C28—O2       178.0 (8)
C11—C2—C3—C4       174.3 (10)      C26—C27—C28—C23     −3.0 (14)
C12—C54—C55—C56    −179.0 (9)       C28—C23—C24—C25     −1.5 (15)
C13—C40—C41—C42    −176.7 (8)       C29—C30—C31—C32     1.9 (17)
O1—C16—C17—C19     178.0 (9)        C30—C29—C34—O6       173.2 (9)
O1—C16—C17—C21     −4.6 (15)        C30—C29—C34—C33     −6.2 (14)
O7—C44—C48—C47     −177.3 (9)       C30—C31—C32—C33     −1.6 (17)
O7—C44—C48—C49     4.4 (13)         C31—C32—C33—C34     −2.6 (15)
O9—Co3—O7—Co2      −172.2 (3)       C31—C32—C33—C35     178.6 (10)
O9—Co3—O7—C44      40.1 (7)          C32—C33—C34—O6      −173.0 (9)
O9—Co3—O8—Co4      77.2 (3)         C32—C33—C34—C29     6.4 (14)
O9—Co3—O8—C56      −79.2 (8)        C32—C33—C35—N3      −132.3 (9)
O12—Co5—O3—Co6    −175.3 (3)       C34—C29—C30—C31     2.0 (15)
O12—Co5—O3—C14    35.4 (7)          C34—C33—C35—N3      49.0 (12)
O12—Co5—O4—Co4    76.5 (3)          C35—N3—C36—C37     −172.1 (9)
O12—Co5—O4—C6     −76.8 (7)        C35—C33—C34—O6      5.7 (14)
O13—Co3—O7—Co2    −16.3 (3)        C35—C33—C34—C29     −174.9 (9)
O13—Co3—O7—C44    133.4 (6)        C35—N3—C35—C33     109.9 (10)
O13—Co3—O8—C56    −172.6 (8)       C36—C37—C38—C39     −175.8 (10)
O14—Co5—O3—Co6     1.3 (3)           C36—C37—C42—O5     −7.0 (15)
O14—Co5—O3—C14   −148.0 (7)       C36—C37—C42—C41     172.9 (9)
O14—Co5—O4—Co4    −99.9 (2)        C37—C38—C39—C40     2.8 (16)
O14—Co5—O4—C6     106.8 (7)        C38—C37—C42—O5     176.1 (9)
O15—Co3—O7—Co2    3.5 (3)           C38—C37—C42—C41     −4.0 (13)
O15—Co3—O7—C44   −144.3 (7)       C38—C39—C40—C41     174.8 (8)
O15—Co3—O8—Co4   −98.2 (3)        C38—C39—C40—C41     −4.2 (15)
O15—Co3—O8—C56   105.4 (8)         C39—C40—C41—C42     1.4 (16)
O16—Co5—O3—Co6   −82.0 (3)        C40—C41—C42—C37     2.7 (14)
O16—Co5—O3—C14  128.8 (6)         C40—C41—C42—C37     1.4 (15)
O16—Co5—O4—Co4   −16.9 (2)        C41—C44—C48—C47     1.9 (15)
O16—Co5—O4—C6   −170.2 (7)       C41—C44—C48—C49     −176.3 (9)
N1—C22—C23—C24  −125.0 (10)      C43—C44—C48—C47     2.4 (18)
N1—C22—C23—C28   53.1 (12)        C43—C44—C48—C49     3.4 (17)
N2—Co5—O3—Co6   98.4 (3)          C44—C43—C45—C46     −5.7 (12)
N2—Co5—O3—C14  −50.9 (7)        C45—C43—C44—O7      −179.5 (10)
N2—Co5—O4—Co4  162.8 (3)         C45—C43—C44—C48     1.2 (15)
N2—Co5—O4—C6   9.5 (8)            C45—C46—C47—C48     0.8 (18)
N2—C8—C9—C10  123.2 (10)        C46—C47—C48—C44     −3.0 (16)
| Bond                        | Angle   | | Bond                        | Angle   |
|-----------------------------|---------| |-----------------------------|---------|
| N2—C8—C9—C14               | −57.7 (12) | | C46—C47—C48—C49            | 175.2 (10) |
| N3—C36—C37—C38             | −177.4 (10) | | C47—C48—C49—N4             | 124.2 (10) |
| N3—C36—C37—C42             | 5.6 (16)  | | C49—N4—C50—C51            | −178.7 (9) |
| N4—Co3—O7—Co2              | 99.8 (3)  | | C50—N4—C49—C48            | −129.2 (9) |
| N4—Co3—O7—C44              | −48.0 (7)  | | C50—C51—C52—C53           | −179.8 (11) |
| N4—Co3—O8—Co4              | 165.1 (3)  | | C50—C51—C56—O8            | 1.9 (15)  |
| N4—Co3—O8—C56              | 8.8 (8)   | | C50—C51—C56—C55           | 179.4 (10) |
| N4—C50—C51—C52             | 179.8 (10) | | C51—C52—C53—C54           | −0.3 (18)  |
| N4—C50—C51—C56             | 0.4 (16)  | | C52—C51—C56—O8            | −177.5 (9) |
| C1—C2—C3—C4                | −3 (2)    | | C52—C51—C56—C55           | −0.1 (15)  |
| C2—C1—C6—O4                | 178.7 (11) | | C52—C53—C54—Cl2           | 178.6 (10) |
| C2—C1—C6—C5                | −2.1 (16) | | C52—C53—C54—C55           | 1.2 (19)   |
| C2—C3—C4—C5                | 1.9 (19)  | | C53—C54—C55—C56           | −1.6 (19)  |
| C3—C4—C5—C6                | −1.1 (17) | | C54—C55—C56—O8            | 178.5 (10) |
| C3—C4—C5—C7                | −179.8 (11)| | C54—C55—C56—C51           | 1.0 (16)   |
| C4—C5—C6—O4                | −179.7 (9)| | C56—C51—C52—C53           | −0.3 (16)  |