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Bis[3-chloro-6-(3,5-dimethyl-1*H*-pyrazol-1-yl-κN^2)picolinato-$\kappa^2 N,O$]cobalt(II) 2.5-hydrate

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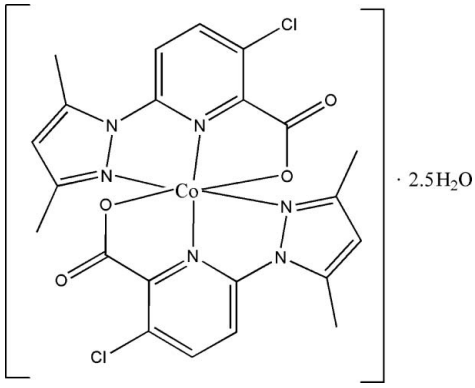
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; some non-H atoms missing; disorder in solvent or counterion; R factor = 0.041; wR factor = 0.130; data-to-parameter ratio = 13.2.

In the title compound, $[Co(C_{11}H_9ClN_3O_2)_2]\cdot 2.5H_2O$, the Co^{II} atom, which lies on a twofold rotation axis, is coordinated by four N atoms and two O atoms from two ligands in a distorted octahedral geometry. In the crystal structure, molecules are linked together by intermolecular O—H···O hydrogen bonds.

Related literature

For related literature, see: Bhatia *et al.* (1981); Costamagna *et al.* (1992).



Experimental

Crystal data

$[Co(C_{11}H_9ClN_3O_2)_2]\cdot 2.5H_2O$
M_r = 605.29
Monoclinic, $C2/c$
a = 20.207 (3) Å
b = 11.7918 (13) Å
c = 14.3531 (16) Å
β = 129.875 (2)°
V = 2624.6 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.91 mm^{-1}
T = 293 (2) K
0.52 × 0.39 × 0.37 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.649, T_{max} = 0.730
6384 measured reflections
2305 independent reflections
1847 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.130
S = 1.09
2305 reflections
175 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.71 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SG2193).

References

Bhatia, S. C., Bindlish, J. M., Saini, A. R. & Jain, P. C. (1981). *J. Chem. Soc. Dalton Trans.* pp. 1773–1779.
Costamagna, J., Vargas, J., Latorre, R. & Alvarado, A. (1992). *Coord. Chem. Rev.* **119**, 67–88.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*a*). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Systems, Inc., Madison, Wisconsin, USA.

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supplementary materials

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Bis[3-chloro-6-(3,5-dimethyl-1*H*-pyrazol-1-yl-κN^2)picolinato-$\kappa^2 N,O$]cobalt(II) 2.5-hydrate

K. Zhao, X.-H. Yin, Y. Feng and J. Zhu

Comment

In recent years, there has been an increasing interest in the coordination chemistry due to the increased recognition of it's role in catalysis enzymatic reactions, magnetism and molecular architectures (Costamagna *et al.*, 1992; Bhatia *et al.*, 1981). We report here the crystal structure of a new cobalt(II) complex with the ligand 6-(3-chloro- (3,5-dimethyl-1*H*-pyrazol-1-yl)) picolinic acid(CDPA)·(I) (Fig.1).

The title compound, (I), consists of a central mononuclear cobalt(II) complex together with two uncoordinated water molecules. The Co atom is coordinated by four N atoms and two O atoms from the two CDPA ligands. The Co^{II} atom is a slightly distorted octahedral environment. The Co—O bond length is 2.090 (2) Å, The Co—N distances range from 2.071 (2) to 2.129 (3) Å, *i.e.* normal values. The C1—C2 bond length is 1.529 (5) Å, being in the normal C—C ranges in cobalt carboxylate complexes·The angles around Co^{II} atom are from 74.49 (10) to 167.64 (15)°. The CDPA molecule acts as a bidentate ligand.

In the title compound, the oxygen atoms contribute to the formation of intermolecular hydrogen bonds involving water O3w atom, as well as carboxyl O2 atom and carboxyl O1 atom (Table 2).

Experimental

3-Chloro-6-(3,5-dimethyl-1*H*-pyrazol-1-yl)picolinic acid, and $CoCl_2$. $6H_2O$ were available commercially and were used without further purification. Equimolar 6-(3-chloro-(3,5-dimethyl-1*H*-pyrazol-1-yl))picolinic acid (1 mmol, 217 mg) was dissolved in anhydrous alcohol (15 ml). The mixture was stirred to give a clear solution, To this solution was added $CoCl_2·6H_2O$ (0.5 mmol, 119 mg) in anhydrous alcohol (10 ml). After keeping the resulting solution in air to evaporate about half of the solvents, dark red prisms of the title compound were formed. The crystals were isolated, washed with alcohol three times and dried in a vacuum desiccator using silica gel (Yield 75%). Elemental analysis: found: C, 53.708; H, 4.20; N, 17.04; O, 13.22; calc. for $C_{22}H_{20}Co_N6_O4$: C, 53.78; H, 4.10; N, 17.10; O, 13.02

Refinement

All the H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with N—H and C—H distances of 0.90 Å and 0.96 Å, respectively. They were treated as riding atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$, $U_{iso}(H) = 1.2U_{eq}(N)$ and $U_{iso}(H) = 1.5U_{eq}(O)$.

Figures

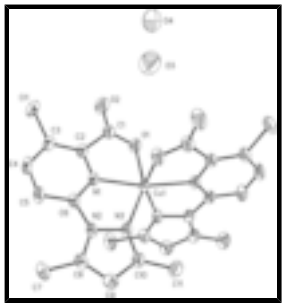

Fig. 1. The structure of the title compound (I) showing 50% probability displacement ellipsoids and the atom-numbering scheme.

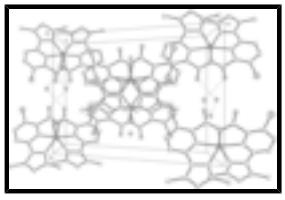

Fig. 2. Crystal packing of (I) showing the hydrogen bonded interactions as dashed lines.

Bis[3-chloro-6-(3,5-dimethyl-1*H*-pyrazol-1-yl-κN^2)picolinato- κ^2N,O]cobalt(II) 2.5-hydrate

Crystal data

$[Co(C_{11}H_9Cl_1N_3O_2)_2]\cdot 2.5H_2O_1$	F_{000} = 1240
M_r = 605.29	D_x = 1.532 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 3006 reflections
a = 20.207 (3) Å	θ = 2.2–28.0º
b = 11.7918 (13) Å	μ = 0.91 mm^{-1}
c = 14.3531 (16) Å	*T* = 293 (2) K
β = 129.875 (2)º	Block, red
V = 2624.6 (5) Å^3	0.52 × 0.39 × 0.37 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	2305 independent reflections
Radiation source: fine-focus sealed tube	1847 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.025
T = 293(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.2º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −24→23
T_{min} = 0.649, T_{max} = 0.730	*k* = −14→9
6384 measured reflections	*l* = −16→17

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.041$	H-atom parameters constrained
$wR(F^2) = 0.130$	$w = 1/[\sigma^2(F_o^2) + (0.0624P)^2 + 4.5881P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.09$	$(\Delta/\sigma)_{max} < 0.001$
2305 reflections	$\Delta\rho_{max} = 0.71$ e Å^{-3}
175 parameters	$\Delta\rho_{min} = -0.48$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Co1	0.0000	0.88230 (5)	0.2500	0.0405 (2)	
Cl1	0.25684 (7)	0.64296 (9)	0.28440 (10)	0.0700 (3)	
N1	0.12695 (15)	0.8634 (2)	0.3226 (2)	0.0400 (6)	
N2	0.15784 (15)	1.0115 (2)	0.4471 (2)	0.0435 (6)	
N3	0.06995 (15)	1.0112 (2)	0.3844 (2)	0.0450 (7)	
O1	−0.00805 (14)	0.7608 (2)	0.1370 (2)	0.0584 (7)	
O2	0.0689 (2)	0.6276 (3)	0.1374 (4)	0.0981 (12)	
O3	0.0520 (3)	0.4551 (4)	0.4323 (5)	0.160 (2)	
H3A	0.0156	0.5085	0.4053	0.192*	
H3B	0.0605	0.4261	0.4934	0.192*	
O4	0.0000	0.3204 (11)	0.2500	0.118 (4)	0.50
H4A	0.0167	0.3626	0.3096	0.142*	0.50
C1	0.0613 (2)	0.7152 (3)	0.1753 (3)	0.0539 (9)	
C2	0.1436 (2)	0.7766 (3)	0.2794 (3)	0.0430 (7)	
C3	0.2279 (2)	0.7541 (3)	0.3311 (3)	0.0489 (8)	
C4	0.2934 (2)	0.8222 (4)	0.4251 (3)	0.0614 (10)	
H4	0.3503	0.8078	0.4595	0.074*	
C5	0.2752 (2)	0.9103 (4)	0.4677 (3)	0.0578 (10)	

H5	0.3186	0.9570	0.5300	0.069*
C6	0.18908 (19)	0.9271 (3)	0.4139 (3)	0.0427 (7)
C7	0.2919 (3)	1.1245 (4)	0.6132 (4)	0.0785 (14)
H7A	0.3035	1.1900	0.6614	0.118*
H7B	0.3102	1.1387	0.5669	0.118*
H7C	0.3227	1.0604	0.6654	0.118*
C8	0.1967 (2)	1.1000 (3)	0.5284 (3)	0.0519 (9)
C9	0.1331 (2)	1.1555 (4)	0.5161 (3)	0.0591 (10)
H9	0.1394	1.2196	0.5589	0.071*
C10	0.0553 (2)	1.0985 (3)	0.4266 (3)	0.0503 (8)
C11	−0.0344 (3)	1.1244 (4)	0.3785 (4)	0.0714 (12)
H11A	−0.0708	1.0598	0.3348	0.107*
H11B	−0.0565	1.1885	0.3250	0.107*
H11C	−0.0333	1.1413	0.4448	0.107*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.0225 (3)	0.0521 (4)	0.0380 (4)	0.000	0.0152 (3)	0.000
Cl1	0.0582 (6)	0.0774 (7)	0.0825 (7)	0.0267 (5)	0.0489 (6)	0.0137 (5)
N1	0.0260 (12)	0.0485 (16)	0.0385 (14)	0.0019 (11)	0.0175 (11)	0.0032 (11)
N2	0.0247 (12)	0.0551 (17)	0.0372 (14)	−0.0030 (12)	0.0137 (11)	−0.0024 (12)
N3	0.0254 (13)	0.0600 (18)	0.0404 (14)	0.0003 (12)	0.0169 (11)	−0.0028 (13)
O1	0.0334 (12)	0.0703 (17)	0.0556 (15)	−0.0012 (11)	0.0212 (11)	−0.0165 (12)
O2	0.0618 (19)	0.095 (2)	0.125 (3)	−0.0092 (17)	0.054 (2)	−0.056 (2)
O3	0.135 (4)	0.127 (4)	0.243 (6)	0.037 (3)	0.133 (4)	0.064 (4)
O4	0.084 (7)	0.139 (10)	0.111 (8)	0.000	0.053 (7)	0.000
C1	0.042 (2)	0.059 (2)	0.056 (2)	−0.0016 (17)	0.0292 (17)	−0.0095 (17)
C2	0.0363 (16)	0.0491 (19)	0.0426 (17)	0.0051 (14)	0.0247 (15)	0.0072 (14)
C3	0.0405 (18)	0.058 (2)	0.0521 (19)	0.0134 (16)	0.0315 (16)	0.0133 (16)
C4	0.0261 (16)	0.083 (3)	0.063 (2)	0.0117 (18)	0.0236 (17)	0.011 (2)
C5	0.0263 (16)	0.079 (3)	0.052 (2)	−0.0006 (17)	0.0179 (16)	−0.0035 (19)
C6	0.0285 (15)	0.0522 (19)	0.0378 (16)	−0.0001 (14)	0.0168 (14)	0.0042 (14)
C7	0.039 (2)	0.084 (3)	0.081 (3)	−0.020 (2)	0.024 (2)	−0.026 (2)
C8	0.0371 (18)	0.057 (2)	0.0472 (19)	−0.0088 (16)	0.0204 (16)	−0.0071 (16)
C9	0.051 (2)	0.061 (2)	0.056 (2)	−0.0064 (18)	0.0305 (19)	−0.0133 (18)
C10	0.0389 (18)	0.060 (2)	0.0471 (19)	0.0011 (16)	0.0254 (16)	−0.0026 (16)
C11	0.048 (2)	0.085 (3)	0.073 (3)	0.004 (2)	0.035 (2)	−0.020 (2)

Geometric parameters (Å, °)

Co1—N1	2.071 (2)	C1—C2	1.529 (5)
Co1—N1^{i}	2.071 (2)	C2—C3	1.380 (4)
Co1—O1	2.090 (2)	C3—C4	1.388 (5)
Co1—O1^{i}	2.090 (2)	C4—C5	1.371 (6)
Co1—N3	2.129 (3)	C4—H4	0.9300
Co1—N3^{i}	2.129 (3)	C5—C6	1.393 (4)
Cl1—C3	1.735 (4)	C5—H5	0.9300

N1—C6	1.323 (4)	C7—C8	1.504 (5)
N1—C2	1.346 (4)	C7—H7A	0.9600
N2—C8	1.375 (4)	C7—H7B	0.9600
N2—N3	1.384 (3)	C7—H7C	0.9600
N2—C6	1.414 (4)	C8—C9	1.350 (5)
N3—C10	1.321 (4)	C9—C10	1.408 (5)
O1—C1	1.252 (4)	C9—H9	0.9300
O2—C1	1.221 (4)	C10—C11	1.499 (5)
O3—H3A	0.8500	C11—H11A	0.9600
O3—H3B	0.8502	C11—H11B	0.9600
O4—H4A	0.8500	C11—H11C	0.9600
N1—Co1—N1^{i}	167.64 (15)	C2—C3—Cl1	122.9 (3)
N1—Co1—O1	76.79 (9)	C4—C3—Cl1	117.7 (3)
N1^{i}—Co1—O1	94.64 (10)	C5—C4—C3	120.7 (3)
N1—Co1—O1^{i}	94.64 (10)	C5—C4—H4	119.6
N1^{i}—Co1—O1^{i}	76.79 (9)	C3—C4—H4	119.6
O1—Co1—O1^{i}	93.49 (16)	C4—C5—C6	117.2 (3)
N1—Co1—N3	74.49 (10)	C4—C5—H5	121.4
N1^{i}—Co1—N3	114.91 (10)	C6—C5—H5	121.4
O1—Co1—N3	150.26 (9)	N1—C6—C5	121.5 (3)
O1^{i}—Co1—N3	96.33 (11)	N1—C6—N2	112.9 (3)
N1—Co1—N3^{i}	114.91 (10)	C5—C6—N2	125.6 (3)
N1^{i}—Co1—N3^{i}	74.49 (10)	C8—C7—H7A	109.5
O1—Co1—N3^{i}	96.33 (11)	C8—C7—H7B	109.5
O1^{i}—Co1—N3^{i}	150.26 (9)	H7A—C7—H7B	109.5
N3—Co1—N3^{i}	88.88 (15)	C8—C7—H7C	109.5
C6—N1—C2	122.0 (3)	H7A—C7—H7C	109.5
C6—N1—Co1	121.3 (2)	H7B—C7—H7C	109.5
C2—N1—Co1	116.4 (2)	C9—C8—N2	106.2 (3)
C8—N2—N3	110.5 (3)	C9—C8—C7	129.0 (4)
C8—N2—C6	133.1 (3)	N2—C8—C7	124.8 (3)
N3—N2—C6	116.3 (2)	C8—C9—C10	107.5 (3)
C10—N3—N2	105.8 (3)	C8—C9—H9	126.3
C10—N3—Co1	139.3 (2)	C10—C9—H9	126.3
N2—N3—Co1	114.9 (2)	N3—C10—C9	110.0 (3)
C1—O1—Co1	116.5 (2)	N3—C10—C11	120.5 (3)
H3A—O3—H3B	108.2	C9—C10—C11	129.5 (3)
O2—C1—O1	126.4 (4)	C10—C11—H11A	109.5
O2—C1—C2	117.8 (3)	C10—C11—H11B	109.5
O1—C1—C2	115.8 (3)	H11A—C11—H11B	109.5
N1—C2—C3	119.0 (3)	C10—C11—H11C	109.5
N1—C2—C1	112.0 (3)	H11A—C11—H11C	109.5
C3—C2—C1	129.0 (3)	H11B—C11—H11C	109.5
C2—C3—C4	119.4 (3)		
N1^{i}—Co1—N1—C6	−137.3 (2)	Co1—N1—C2—C1	5.0 (3)
O1—Co1—N1—C6	175.8 (3)	O2—C1—C2—N1	−172.3 (4)

O1^{i}—Co1—N1—C6	−91.7 (3)	O1—C1—C2—N1	7.8 (5)
N3—Co1—N1—C6	3.6 (2)	O2—C1—C2—C3	7.4 (6)
N3^{i}—Co1—N1—C6	84.9 (3)	O1—C1—C2—C3	−172.4 (3)
N1^{i}—Co1—N1—C2	36.9 (2)	N1—C2—C3—C4	−0.9 (5)
O1—Co1—N1—C2	−10.1 (2)	C1—C2—C3—C4	179.4 (3)
O1^{i}—Co1—N1—C2	82.4 (2)	N1—C2—C3—Cl1	178.8 (2)
N3—Co1—N1—C2	177.7 (2)	C1—C2—C3—Cl1	−0.9 (5)
N3^{i}—Co1—N1—C2	−101.0 (2)	C2—C3—C4—C5	0.8 (6)
C8—N2—N3—C10	0.1 (4)	Cl1—C3—C4—C5	−179.0 (3)
C6—N2—N3—C10	−177.7 (3)	C3—C4—C5—C6	0.8 (6)
C8—N2—N3—Co1	177.8 (2)	C2—N1—C6—C5	2.4 (5)
C6—N2—N3—Co1	0.0 (3)	Co1—N1—C6—C5	176.2 (3)
N1—Co1—N3—C10	174.9 (4)	C2—N1—C6—N2	−178.4 (3)
N1^{i}—Co1—N3—C10	−13.7 (4)	Co1—N1—C6—N2	−4.6 (4)
O1—Co1—N3—C10	159.4 (3)	C4—C5—C6—N1	−2.4 (5)
O1^{i}—Co1—N3—C10	−92.1 (4)	C4—C5—C6—N2	178.5 (3)
N3^{i}—Co1—N3—C10	58.6 (3)	C8—N2—C6—N1	−174.4 (3)
N1—Co1—N3—N2	−1.7 (2)	N3—N2—C6—N1	2.8 (4)
N1^{i}—Co1—N3—N2	169.7 (2)	C8—N2—C6—C5	4.8 (6)
O1—Co1—N3—N2	−17.2 (4)	N3—N2—C6—C5	−178.0 (3)
O1^{i}—Co1—N3—N2	91.4 (2)	N3—N2—C8—C9	−0.3 (4)
N3^{i}—Co1—N3—N2	−118.0 (2)	C6—N2—C8—C9	177.0 (3)
N1—Co1—O1—C1	15.0 (3)	N3—N2—C8—C7	178.8 (4)
N1^{i}—Co1—O1—C1	−156.0 (3)	C6—N2—C8—C7	−3.9 (6)
O1^{i}—Co1—O1—C1	−79.0 (3)	N2—C8—C9—C10	0.4 (4)
N3—Co1—O1—C1	30.3 (4)	C7—C8—C9—C10	−178.7 (4)
N3^{i}—Co1—O1—C1	129.1 (3)	N2—N3—C10—C9	0.1 (4)
Co1—O1—C1—O2	163.4 (4)	Co1—N3—C10—C9	−176.7 (3)
Co1—O1—C1—C2	−16.8 (4)	N2—N3—C10—C11	−180.0 (3)
C6—N1—C2—C3	−0.7 (5)	Co1—N3—C10—C11	3.3 (6)
Co1—N1—C2—C3	−174.8 (2)	C8—C9—C10—N3	−0.3 (5)
C6—N1—C2—C1	179.1 (3)	C8—C9—C10—C11	179.8 (4)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3A···O2^{ii}	0.85	1.98	2.825 (8)	172
O3—H3B···O2^{iii}	0.85	2.06	2.907 (8)	172
O4—H4A···O3	0.85	1.79	2.636 (8)	178

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

Fig. 1

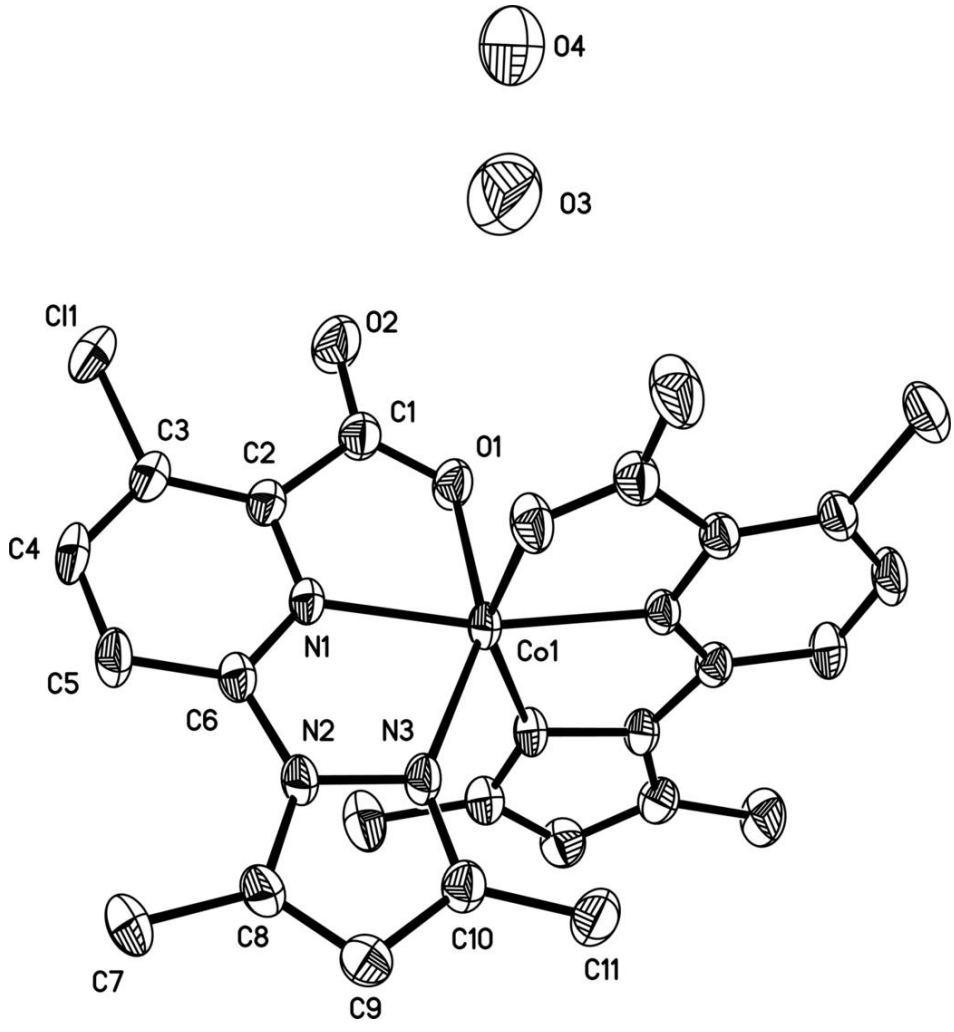

Fig. 2