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Triaquachlorido(1,10-phenanthroline- κ^2N,N')cobalt(II) chloride monohydrate

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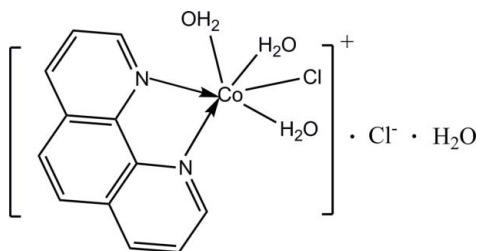
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Key indicators: single-crystal X-ray study; $T = 273$ K; mean $\sigma(C-C) = 0.013$ Å; R factor = 0.071; wR factor = 0.177; data-to-parameter ratio = 13.8.

In the title compound, $[CoCl(C_{12}H_8N_2)(H_2O)_3]Cl \cdot H_2O$, the Co^{II} ion is coordinated by two N atoms from the 1,10-phenanthroline ligand [$Co-N = 2.125(6)$ and $2.146(6)$ Å], one chloride ligand [$Co-Cl = 2.459(2)$ Å] and three water molecules [$Co-O = 2.070(5)$ – $2.105(5)$ Å] in a distorted octahedral geometry. Intermolecular $O-H \cdots Cl$ and $O-H \cdots O$ hydrogen bonds form an extensive three-dimensional hydrogen-bonding network, which consolidates the crystal packing.

Related literature

For related crystal structures, see: Liu *et al.* (2006); Zhang *et al.* (1999).



Experimental

Crystal data

$[CoCl(C_{12}H_8N_2)(H_2O)_3]Cl \cdot H_2O$

$M_r = 382.10$

Monoclinic, $P2_1/c$
 $a = 7.646(4)$ Å
 $b = 12.426(6)$ Å
 $c = 16.987(8)$ Å
 $\beta = 103.54(2)^\circ$
 $V = 1569.1(13)$ Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 1.45$ mm⁻¹
 $T = 273(2)$ K
 $0.37 \times 0.25 \times 0.19$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{min} = 0.616$, $T_{max} = 0.770$

6533 measured reflections
 2733 independent reflections
 1564 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.080$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.071$
 $wR(F^2) = 0.177$
 $S = 1.04$
 2733 reflections
 198 parameters

13 restraints
 H-atom parameters constrained
 $\Delta\rho_{max} = 0.67$ e Å⁻³
 $\Delta\rho_{min} = -0.56$ e Å⁻³

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1–H1A \cdots O4	0.85	1.93	2.693 (9)	149
O1–H1B \cdots Cl2	0.85	2.55	3.258 (6)	141
O2–H2A \cdots Cl1 ⁱ	0.85	2.31	3.139 (6)	166
O2–H2B \cdots Cl2 ⁱ	0.85	2.29	3.119 (6)	164
O3–H3A \cdots Cl2 ⁱⁱ	0.85	2.33	3.114 (6)	153
O3–H3B \cdots Cl1 ⁱⁱ	0.85	2.30	3.128 (6)	166
O4–H4A \cdots Cl2 ⁱⁱⁱ	0.85	2.34	3.185 (8)	170
O4–H4B \cdots Cl1 ⁱⁱ	0.85	2.58	3.278 (8)	141

Symmetry codes: (i) $-x + 1, -y + 1, -z + 2$; (ii) $-x + 2, -y + 1, -z + 2$; (iii) $-x + 2, y + \frac{1}{2}, -z + \frac{5}{2}$.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); 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: CV2481).

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supporting information

Acta Cryst. (2009). E65, m114 [doi:10.1107/S1600536808042591]

Triaquachlorido(1,10-phenanthroline- κ^2N,N')cobalt(II) chloride monohydrate**Yaru Liu, Yutian Pan, Junshan Sun and Chuan Zhang****S1. Comment**

The structures of some tetraaqua(1,10-phenanthroline) metal ionic complexes with different anions have been reported (Liu *et al.*, 2006; Zhang *et al.*, 1999). In our study in this field, we select 1,10-phenanthroline as the co-ligand to continue our exploration of the Co complexes. Herein we report the structure of the title compound (I), which was characterized by elemental analyses and X-ray crystallography diffraction.

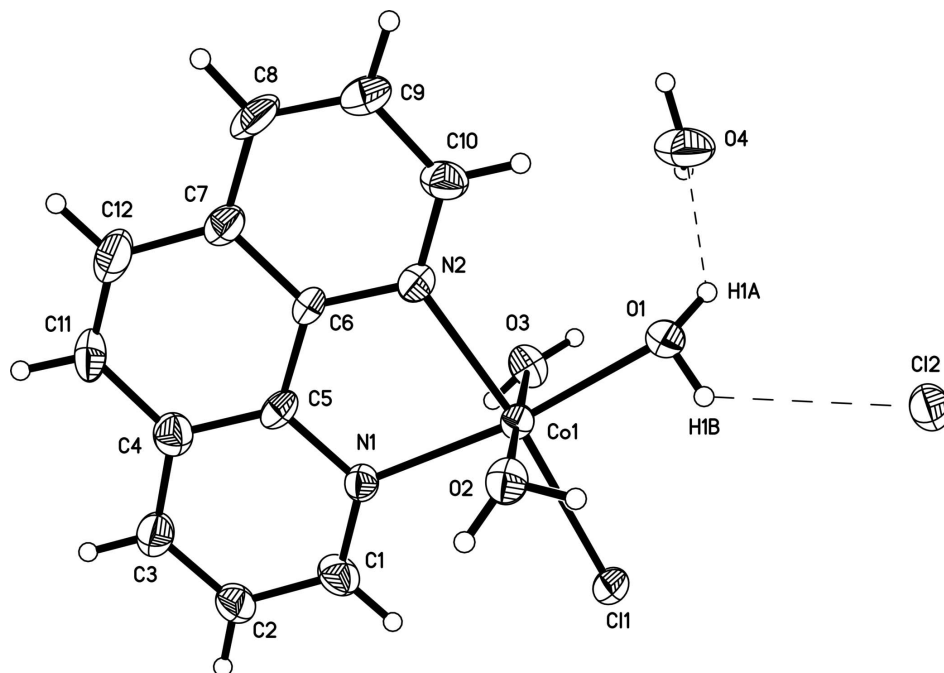
In (I) (Fig. 1), each Co^{II} ion is coordinated by two N atoms from 1,10-phenanthroline ligand [Co—N 2.125 (6), 2.146 (6) Å], one chlorine anion [Co—Cl 2.459 (2) Å] and three water molecules [Co—O 2.070 (5)–2.105 (5) Å] in a distorted octahedral geometry. Two aqua O atoms and two N atoms from 1,10-phenanthroline ligand define the special position on a mirror plane. One aqua group and chlorine anion fill the axial apical positions. The intermolecular O—H \cdots Cl and O—H \cdots O hydrogen bonds (Table 1) form an extensive three-dimensional hydrogen-bonding network, which consolidate the crystal packing.

S2. Experimental

A mixture of Cu(Cl)₂·3H₂O (168 mg, 1 mmol) and 1,10-phenanthroline (185 mg, 1 mmol) in methanol (30 ml) was placed in a Teflon-lined stainless steel Parr bomb that was heated at 423 K for 72 h. The bomb was then cooled down to the room temperature, the solution was filtered. The solvent was removed from the filtrate under vacuum, and the solid residue was recrystallized from diethyl ether; blue crystals suitable for X-Ray diffraction study were obtained. Yield, 0.724 g, 81%. m.p. 566 K. Analysis, calculated for C₁₂H₁₆Cl₂CoN₂O₄: C 37.72, H 4.22, N 7.33; found: C 37.53, H 4.68, N 7.17%. The elemental analyses were performed with a Perkin Elmer PE2400II instrument.

S3. Refinement

Water H atoms were located in a difference map and were isotropically refined with an O—H strict distance restraint of 0.85/Å. H atoms bound to C atoms were placed in calculated positions (C—H = 0.93/Å) and refined in the riding-model approximation, $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

**Figure 1**

The structure of the title complex, showing 30% probability displacement ellipsoids and the atom-numbering scheme. Dashed lines denote hydrogen bonds.

Triaquachlorido(1,10-phenanthroline- κ^2 N,N')cobalt(II) chloride monohydrate

Crystal data

$[\text{CoCl}(\text{C}_{12}\text{H}_8\text{N}_2)(\text{H}_2\text{O})_3]\text{Cl}\cdot\text{H}_2\text{O}$

$M_r = 382.10$

Monoclinic, $P2_1/c$

$a = 7.646$ (4) Å

$b = 12.426$ (6) Å

$c = 16.987$ (8) Å

$\beta = 103.54$ (2)°

$V = 1569.1$ (13) Å³

$Z = 4$

$F(000) = 780$

$D_x = 1.617$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 580 reflections

$\theta = 3.0\text{--}20.1^\circ$

$\mu = 1.45$ mm⁻¹

$T = 273$ K

Block, blue

$0.37 \times 0.25 \times 0.19$ mm

Data collection

Bruker SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.616$, $T_{\max} = 0.770$

6533 measured reflections

2733 independent reflections

1564 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.080$

$\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 2.1^\circ$

$h = -9 \rightarrow 5$

$k = -10 \rightarrow 14$

$l = -17 \rightarrow 20$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.071$
 $wR(F^2) = 0.177$
 $S = 1.04$
 2733 reflections
 198 parameters
 13 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0517P)^2 + 4.0479P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.67 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.56 \text{ e } \text{Å}^{-3}$

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	$U_{\text{iso}}^*/U_{\text{eq}}$
Co1	0.75359 (13)	0.66886 (9)	0.99070 (6)	0.0342 (3)
Cl1	0.6997 (3)	0.49910 (16)	0.91423 (11)	0.0431 (5)
Cl2	0.8145 (3)	0.34056 (18)	1.16347 (12)	0.0536 (6)
N1	0.6918 (8)	0.7697 (5)	0.8867 (4)	0.0360 (15)
N2	0.7887 (8)	0.8255 (5)	1.0452 (4)	0.0405 (16)
O1	0.8190 (8)	0.5892 (4)	1.1032 (3)	0.0478 (15)
H1A	0.9003	0.5920	1.1469	0.15 (6)*
H1B	0.7797	0.5251	1.0958	0.10 (4)*
O2	0.4845 (7)	0.6729 (4)	0.9965 (3)	0.0459 (13)
H2A	0.4527	0.6214	1.0231	0.06 (3)*
H2B	0.4185	0.6755	0.9488	0.05 (3)*
O3	1.0230 (7)	0.6657 (4)	0.9883 (3)	0.0456 (14)
H3A	1.0372	0.6788	0.9411	0.05 (3)*
H3B	1.1121	0.6289	1.0137	0.09 (4)*
O4	1.1261 (10)	0.6517 (6)	1.2062 (4)	0.075 (2)
H4A	1.1566	0.7013	1.2412	0.06 (3)*
H4B	1.2171	0.6296	1.1901	0.16 (7)*
C1	0.6476 (11)	0.7393 (8)	0.8103 (5)	0.049 (2)
H1	0.6394	0.6664	0.7979	0.059*
C2	0.6123 (11)	0.8160 (7)	0.7468 (5)	0.050 (2)
H2	0.5810	0.7933	0.6931	0.060*
C3	0.6243 (11)	0.9222 (7)	0.7645 (5)	0.052 (2)
H3	0.6017	0.9726	0.7228	0.062*
C4	0.6706 (11)	0.9573 (7)	0.8456 (5)	0.046 (2)
C5	0.7032 (10)	0.8787 (6)	0.9056 (5)	0.037 (2)

C6	0.7525 (9)	0.9047 (6)	0.9885 (5)	0.0338 (17)
C7	0.7636 (10)	1.0150 (7)	1.0107 (5)	0.044 (2)
C8	0.8142 (12)	1.0398 (8)	1.0931 (6)	0.062 (3)
H8	0.8232	1.1111	1.1102	0.075*
C9	0.8501 (13)	0.9583 (8)	1.1481 (6)	0.062 (3)
H9	0.8841	0.9740	1.2031	0.074*
C10	0.8363 (10)	0.8527 (7)	1.1225 (5)	0.049 (2)
H10	0.8616	0.7985	1.1612	0.058*
C11	0.6849 (12)	1.0667 (7)	0.8682 (6)	0.058 (3)
H11	0.6645	1.1199	0.8285	0.070*
C12	0.7284 (13)	1.0944 (7)	0.9475 (6)	0.063 (3)
H12	0.7357	1.1669	0.9614	0.075*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.0399 (6)	0.0288 (6)	0.0335 (6)	-0.0003 (5)	0.0080 (4)	0.0012 (5)
Cl1	0.0576 (13)	0.0299 (11)	0.0394 (12)	-0.0009 (9)	0.0068 (9)	-0.0034 (10)
Cl2	0.0673 (14)	0.0495 (14)	0.0443 (12)	0.0020 (11)	0.0133 (10)	0.0060 (12)
N1	0.040 (4)	0.033 (4)	0.036 (4)	0.000 (3)	0.011 (3)	0.002 (3)
N2	0.043 (4)	0.034 (4)	0.045 (4)	-0.001 (3)	0.009 (3)	-0.003 (4)
O1	0.068 (4)	0.040 (4)	0.034 (3)	-0.006 (3)	0.009 (3)	0.002 (3)
O2	0.050 (3)	0.042 (4)	0.048 (3)	0.000 (3)	0.016 (3)	0.004 (3)
O3	0.043 (3)	0.055 (4)	0.042 (3)	0.006 (3)	0.015 (2)	0.007 (3)
O4	0.080 (5)	0.080 (5)	0.058 (4)	0.008 (4)	0.001 (4)	-0.028 (4)
C1	0.058 (6)	0.059 (6)	0.030 (5)	0.005 (4)	0.010 (4)	0.004 (4)
C2	0.061 (6)	0.055 (6)	0.032 (4)	0.003 (4)	0.008 (4)	0.004 (4)
C3	0.062 (6)	0.040 (6)	0.052 (6)	0.003 (4)	0.012 (4)	0.005 (5)
C4	0.046 (5)	0.044 (6)	0.047 (5)	0.005 (4)	0.009 (4)	0.006 (4)
C5	0.036 (4)	0.025 (4)	0.055 (6)	0.000 (3)	0.018 (4)	-0.009 (4)
C6	0.038 (4)	0.022 (4)	0.042 (5)	-0.001 (3)	0.010 (3)	-0.001 (4)
C7	0.043 (5)	0.032 (5)	0.057 (6)	0.001 (4)	0.013 (4)	-0.005 (4)
C8	0.078 (7)	0.034 (6)	0.074 (7)	-0.002 (5)	0.014 (5)	-0.023 (5)
C9	0.080 (7)	0.045 (6)	0.059 (6)	-0.001 (5)	0.016 (5)	-0.021 (5)
C10	0.053 (5)	0.054 (6)	0.038 (5)	0.004 (4)	0.009 (4)	-0.004 (5)
C11	0.072 (7)	0.038 (6)	0.062 (7)	0.009 (5)	0.012 (5)	0.020 (5)
C12	0.075 (7)	0.030 (5)	0.082 (7)	0.002 (5)	0.013 (5)	0.004 (6)

Geometric parameters (Å, °)

Co1—O3	2.071 (5)	C1—H1	0.9300
Co1—O2	2.084 (5)	C2—C3	1.35 (1)
Co1—O1	2.106 (5)	C2—H2	0.9301
Co1—N1	2.127 (6)	C3—C4	1.41 (1)
Co1—N2	2.145 (7)	C3—H3	0.9300
Co1—Cl1	2.461 (2)	C4—C5	1.39 (1)
N1—C1	1.317 (9)	C4—Cl1	1.41 (1)
N1—C5	1.390 (9)	C5—C6	1.41 (1)

N2—C10	1.321 (9)	C6—C7	1.42 (1)
N2—C6	1.361 (9)	C7—C8	1.40 (1)
O1—H1A	0.8500	C7—C12	1.44 (1)
O1—H1B	0.8501	C8—C9	1.36 (1)
O2—H2A	0.8500	C8—H8	0.9301
O2—H2B	0.8498	C9—C10	1.38 (1)
O3—H3A	0.8500	C9—H9	0.9301
O3—H3B	0.8500	C10—H10	0.9300
O4—H4A	0.8500	C11—C12	1.36 (1)
O4—H4B	0.8500	C11—H11	0.9300
C1—C2	1.42 (1)	C12—H12	0.9300
O3—Co1—O2	178.4 (2)	C1—C2—H2	120.2
O3—Co1—O1	89.0 (2)	C2—C3—C4	120.5 (8)
O2—Co1—O1	89.7 (2)	C2—C3—H3	119.7
O3—Co1—N1	91.3 (2)	C4—C3—H3	119.7
O2—Co1—N1	89.8 (2)	C5—C4—C3	117.4 (8)
O1—Co1—N1	171.9 (2)	C5—C4—C11	119.3 (8)
O3—Co1—N2	90.1 (2)	C3—C4—C11	123.3 (8)
O2—Co1—N2	89.0 (2)	N1—C5—C4	121.6 (8)
O1—Co1—N2	93.2 (2)	N1—C5—C6	116.3 (7)
N1—Co1—N2	78.8 (2)	C4—C5—C6	122.0 (7)
C1—N1—C5	119.6 (7)	N2—C6—C5	120.3 (7)
C1—N1—Co1	127.3 (6)	N2—C6—C7	121.4 (7)
C5—N1—Co1	113.1 (5)	C5—C6—C7	118.3 (7)
C10—N2—C6	118.8 (7)	C8—C7—C6	117.7 (8)
C10—N2—Co1	129.7 (6)	C8—C7—C12	123.8 (8)
C6—N2—Co1	111.5 (5)	C6—C7—C12	118.4 (8)
Co1—O1—H1A	138.0	C9—C8—C7	119.2 (8)
Co1—O1—H1B	107.8	C9—C8—H8	120.4
H1A—O1—H1B	109.2	C7—C8—H8	120.4
Co1—O2—H2A	114.5	C8—C9—C10	120.2 (9)
Co1—O2—H2B	109.1	C8—C9—H9	119.9
H2A—O2—H2B	110.8	C10—C9—H9	119.9
Co1—O3—H3A	111.3	N2—C10—C9	122.7 (9)
Co1—O3—H3B	133.1	N2—C10—H10	118.7
H3A—O3—H3B	108.6	C9—C10—H10	118.7
H4A—O4—H4B	110.4	C12—C11—C4	120.0 (8)
N1—C1—C2	121.2 (8)	C12—C11—H11	120.0
N1—C1—H1	119.4	C4—C11—H11	120.0
C2—C1—H1	119.4	C11—C12—C7	121.9 (9)
C3—C2—C1	119.7 (8)	C11—C12—H12	119.1
C3—C2—H2	120.2	C7—C12—H12	119.1

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O1—H1A \cdots O4	0.85	1.93	2.693 (9)	149

O1—H1B···Cl2	0.85	2.55	3.258 (6)	141
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O4—H4B···Cl1 ⁱⁱ	0.85	2.58	3.278 (8)	141

Symmetry codes: (i) $-x+1, -y+1, -z+2$; (ii) $-x+2, -y+1, -z+2$; (iii) $-x+2, y+1/2, -z+5/2$.