

Chun-Lan Yuan

Department of Chemistry and Chemical Engineering, Baoji College of Arts and Sciences, Baoji 721007, People's Republic of China

Correspondence e-mail:
chunlanyuan@126.com

Key indicators

Single-crystal X-ray study
 $T = 298$ K
 Mean $\sigma(\text{C}-\text{C}) = 0.007$ Å
 Disorder in solvent or counterion
 R factor = 0.073
 wR factor = 0.160
 Data-to-parameter ratio = 17.2

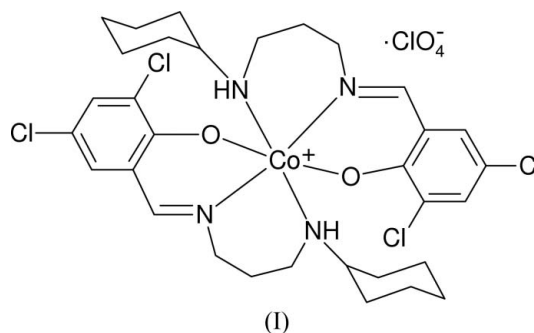
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

trans-Bis{2,4-dichloro-6-[(3-cyclohexylamino)propylimino)methyl]phenolato}cobalt(III) perchlorate

The title compound, $[\text{Co}(\text{C}_{16}\text{H}_{21}\text{Cl}_2\text{N}_2\text{O})_2]\text{ClO}_4$, possesses two independent half-molecules of the cation in the asymmetric unit. The symmetry of both cations is C_i . The Co^{III} atoms are six-coordinated by four N and two O atoms from two Schiff bases, forming slightly distorted octahedral coordination environments.

Comment

Schiff base complexes are very important in coordination chemistry because of their easy preparation, interesting structures and excellent properties (Chang *et al.*, 1998; Chaturvedl, 1977; Archer & Wang, 1990; Costamagna *et al.*, 1992; Bhatia *et al.*, 1981). In an investigation of crystal structures of such complexes, a new cobalt(III) complex, (I), is reported here.



Compound (I) is a perchlorate salt of discrete mononuclear cobalt(III) complex cations (Fig. 1). The structure is similar to that recently reported for the cobalt(III) complex containing the 3-[(3-cyclohexylaminopropylimino)methyl]naphthalen-2-olate ligand (Li *et al.*, 2004). There are two independent half-molecules of the cation in the asymmetric unit. The symmetry of both cations is C_i . Both Co^{III} atoms are in a slightly distorted octahedral geometry formed by two O and four N atoms from two Schiff bases. The bond lengths and angles involving the Co atoms are the same (within experimental error) in each cation (Table 1) and are comparable to those of the complex previously cited. As expected, the cyclohexyl groups adopt chair conformations to minimize the steric effects. The unique ClO_4^- anion is disordered over two sites with occupancies 0.581 (2):0.419 (2). The crystal packing is shown in Fig. 2.

Experimental

3,5-Dichlorosalicylaldehyde (0.1 mmol, 19.1 mg) and *N*-cyclohexyl-1,3-diaminopropane (0.1 mmol, 15.6 mg) were dissolved in MeOH

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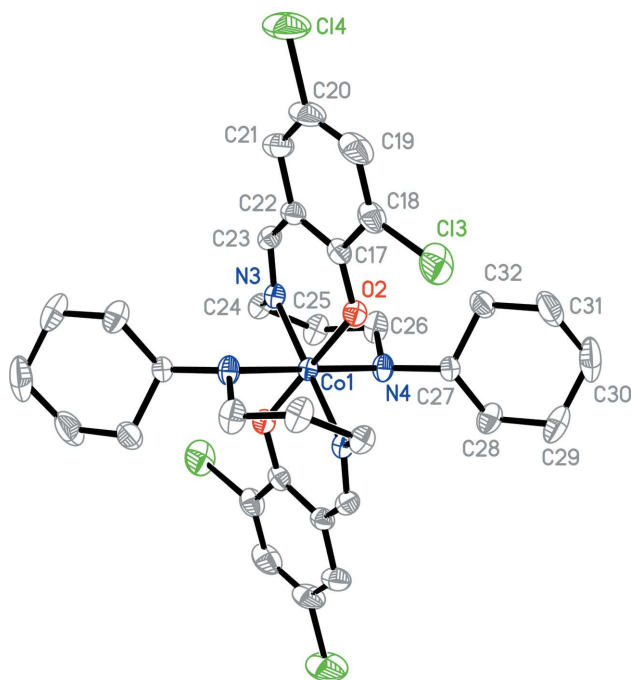


Figure 1

The structure of one of the independent cations of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are not shown. Unlabeled atoms are related to labeled atoms by the symmetry code (1 - *x*, 1 - *y*, 1 - *z*). The second cation has essentially the same appearance.

(10 ml). The mixture was stirred for 15 min to give a clear yellow solution, then a MeOH solution of $\text{Co}(\text{ClO}_4)_3 \cdot 7\text{H}_2\text{O}$ (0.1 mmol, 48.3 mg) was added. The mixture was stirred at room temperature for 15 min and filtered. The filtrate was kept in air for 3 d, and brown block-shaped crystals were formed at the bottom of the vessel on slow evaporation of the solvent.

Crystal data

$[\text{Co}(\text{C}_{16}\text{H}_{21}\text{Cl}_2\text{N}_2\text{O}_2)_2]\text{ClO}_4$	$D_x = 1.501 \text{ Mg m}^{-3}$
$M_r = 814.88$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 6581 reflections
$a = 16.136(2) \text{ \AA}$	$\theta = 2.2\text{--}25.2^\circ$
$b = 12.752(2) \text{ \AA}$	$\mu = 0.90 \text{ mm}^{-1}$
$c = 17.771(2) \text{ \AA}$	$T = 298(2) \text{ K}$
$\beta = 99.486(1)^\circ$	Block, brown
$V = 3606.7(8) \text{ \AA}^3$	$0.35 \times 0.17 \times 0.13 \text{ mm}$
$Z = 4$	

Data collection

Bruker SMART CCD area-detector diffractometer	8227 independent reflections
ω scan	6373 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.038$
$T_{\text{min}} = 0.745$, $T_{\text{max}} = 0.893$	$\theta_{\text{max}} = 27.5^\circ$
30380 measured reflections	$h = -20 \rightarrow 20$
	$k = -16 \rightarrow 16$
	$l = -23 \rightarrow 23$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0524P)^2 + 4.3537P]$
$R[F^2 > 2\sigma(F^2)] = 0.073$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.160$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.15$	$\Delta\rho_{\text{max}} = 0.56 \text{ e \AA}^{-3}$
8227 reflections	$\Delta\rho_{\text{min}} = -0.38 \text{ e \AA}^{-3}$
479 parameters	
H atoms treated by a mixture of independent and constrained refinement	

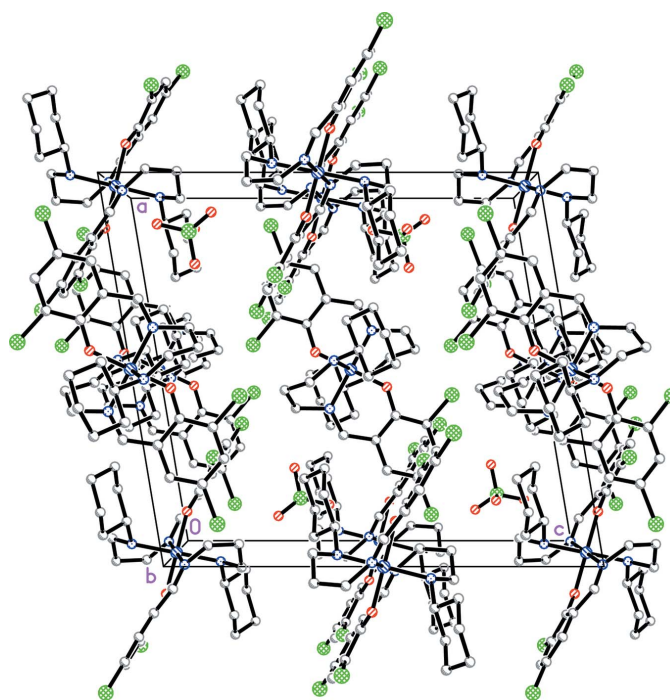


Figure 2

The crystal packing of (I), viewed along the *b* axis. The H atoms and minor anions of the disordered perchlorate anions have been omitted.

Table 1

Selected geometric parameters (\AA , $^\circ$).

Co1—O2	1.883 (2)	Co2—O1	1.886 (2)
Co1—N3	1.926 (3)	Co2—N1	1.924 (3)
Co1—N4	2.007 (3)	Co2—N2	2.010 (3)
O2 ⁱ —Co1—O2	180	O1—Co2—O1 ⁱⁱ	180
O2 ⁱ —Co1—N3	88.74 (12)	O1—Co2—N1	91.10 (12)
O2—Co1—N3	91.26 (12)	O1 ⁱⁱ —Co2—N1	88.90 (12)
N3 ⁱ —Co1—N3	180	N1—Co2—N1 ⁱⁱ	180
O2 ⁱ —Co1—N4	90.07 (12)	O1—Co2—N2	89.69 (12)
O2—Co1—N4	89.93 (12)	O1 ⁱⁱ —Co2—N2	90.31 (12)
N3 ⁱ —Co1—N4	91.42 (13)	N1—Co2—N2	89.27 (13)
N3—Co1—N4	88.58 (13)	N1 ⁱⁱ —Co2—N2	90.73 (13)
N4 ⁱ —Co1—N4	180	N2—Co2—N2 ⁱⁱ	180

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

Table 2

Hydrogen-bond geometry (\AA , $^\circ$).

$D\text{—H}\cdots A$	$D\text{—H}$	$\text{H}\cdots A$	$D\cdots A$	$D\text{—H}\cdots A$
N2—H2 \cdots O4 ⁱⁱⁱ	0.90 (4)	2.55 (4)	3.223 (8)	132 (4)

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

Atoms H2 and H4 were located in a difference Fourier map and refined isotropically, with N—H distances restrained to 0.90 (1) \AA and $U_{\text{iso}} = 0.08 \text{ \AA}^2$. All other H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.98 \AA , and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The O atoms of the perchlorate anion are disordered over two distinct sites with occupancies of 0.581 (2) and 0.419 (2). The Cl—O and

O··O distances in both disordered components were restrained to be equal.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINTE* (Bruker, 1998); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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