

***trans*-Bis{2-[3-(cyclohexylamino)propyl-
iminomethyl]phenolato- κ^3 O,N,N'}cobalt(III)
perchlorate**Zhong-Lu You,^{a,b} Yang Qu,^{a,b}
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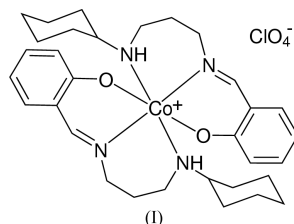
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Key indicatorsSingle-crystal X-ray study
 $T = 298\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.007\text{ \AA}$
Disorder in solvent or counterion
 R factor = 0.066
 wR factor = 0.149
Data-to-parameter ratio = 13.7For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The title compound, $[\text{Co}(\text{C}_{16}\text{H}_{23}\text{N}_2\text{O})_2]\text{ClO}_4$, has been synthesized and characterized. The crystal structure was determined by X-ray crystallography from single-crystal data. It is a mononuclear compound, and the central Co^{III} atom, lying on an inversion center, is coordinated by four N atoms and two O atoms from two Schiff bases, giving an approximately octahedral coordination environment.

Comment

Transition metal compounds containing Schiff base ligands have been of great interest for many years (Yamada, 1999; Chang *et al.*, 1998; Chaturvedi, 1977; Archer & Wang, 1990). These compounds play an important role in the development of coordination chemistry related to catalysis and enzymatic reactions, magnetism and molecular architectures (Costa-magna *et al.*, 1992; Bhatia *et al.*, 1981). Since cobalt compounds are present in the active sites of several important classes of metalloproteins, their study is of great interest in various aspects of chemistry (Downing & Urbach, 1969; Ganeshpure *et al.*, 1996; Bosnich, 1968; Costes *et al.*, 1995; Jacobsen *et al.*, 1997; Ferrari *et al.*, 1976). The catalytic potential of cobalt–Schiff base compounds, such as Co(salen), has been widely studied (Boca *et al.*, 1998). A mononuclear cobalt(III) compound is reported here.



The title compound, (I), is the perchlorate salt of a discrete centrosymmetric mononuclear cobalt(III) complex cation (Fig. 1). The central Co^{III} atom is in an octahedral geometry and is coordinated by two Schiff base ligands. The Schiff base acts as a tridentate ligand through the phenolate O and two N atoms. The three *trans* angles are all exactly 180° , as a result of centrosymmetry, and all other angles around Co1 are close to 90° , ranging from $86.94(13)$ to $93.06(13)^\circ$, indicating a slightly distorted octahedral geometry. The $\text{Co1}-\text{O1}$ (phenolate O atom) bond length of $1.876(3)\text{ \AA}$ is comparable to the value of $1.884(2)\text{ \AA}$ observed in a Schiff base–cobalt(III) complex we reported recently (Zhu *et al.*, 2003). The $\text{Co1}-\text{N1}$ (imine) bond distance is $1.936(4)\text{ \AA}$ and the $\text{Co1}-\text{N2}$ (amine) length is $2.062(3)\text{ \AA}$, both of which are slightly longer than the corresponding bonds in the same cobalt(III) complex described above.

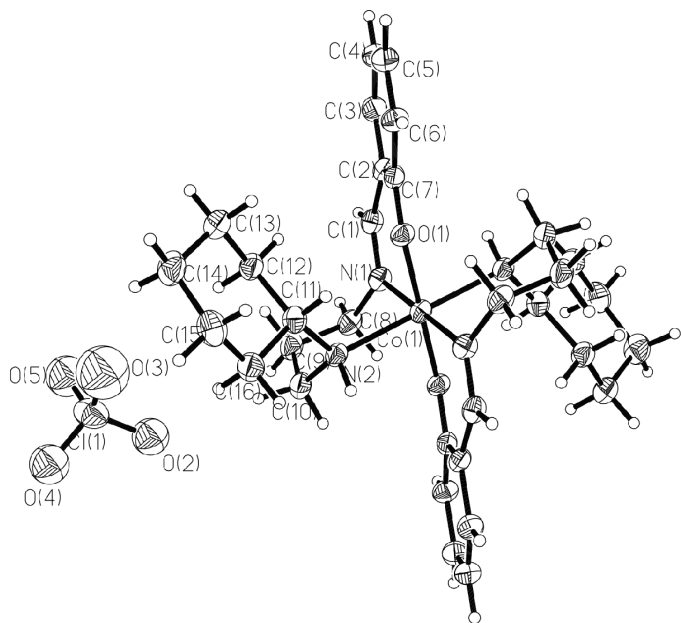


Figure 1
The structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme.

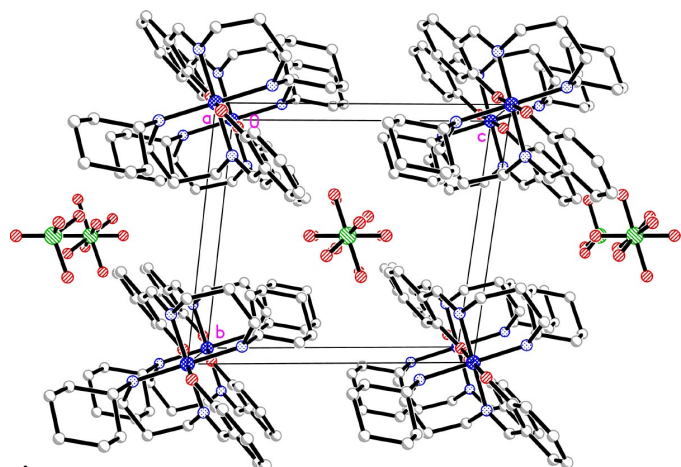


Figure 2
The crystal packing of (I), viewed along the *a* axis, including both disorder components of the anions.

The crystal structure consists of $[\text{Co}(\text{C}_{16}\text{H}_{23}\text{N}_2\text{O})_2]^+$ cations and ClO_4^- anions. As expected, the cyclohexyl groups in the complex adopt chair conformations to minimize steric effects. The perchlorate anion is disordered over an inversion center.

Experimental

N-Cyclohexyl-1,3-diaminopropane and salicylaldehyde were available commercially and were used without further purification. *N*-Cyclohexyl-1,3-diaminopropane (2.0 mmol, 312 mg) and salicylaldehyde (2.0 mmol, 244 mg) were dissolved in methanol (10 ml). The mixture was stirred for 1 h to give a clear orange solution of *L* (2.0 mmol), where *L* is 2-[(3-cyclohexylaminopropylimino)methyl]-phenol. To the solution of *L* was added a solution of $\text{Co}(\text{ClO}_4)_3 \cdot 9\text{H}_2\text{O}$ (1.0 mmol, 519 mg) in methanol (10 ml), with stirring. After keeping the resulting solution in air for 10 d, brown block-shaped crystals were formed at the bottom of the vessel on slow evaporation of the

solvent. The crystals were isolated, washed three times with methanol, and dried in a vacuum desiccator using P_4O_{10} (yield 77.8%). Analysis found: C 56.72, H 6.97, N 8.12%; calculated for $\text{C}_{32}\text{ClCoH}_{46}\text{N}_4\text{O}_6$: C 56.76, H 6.85, N 8.27%. IR data: 3446.9 (*m*); 3237.0 (*m*); 2924.6 (*s*); 2847.8 (*m*); 1613.7 (*s*); 1542.0 (*m*); 1450.0 (*s*); 1342.3 (*m*); 1311.5 (*s*); 1214.2 (*w*); 1096.4 (*s*); 901.8 (*m*); 773.8 (*m*); 620.2 (*w*) cm^{-1} .

Crystal data

$[\text{Co}(\text{C}_{16}\text{H}_{23}\text{N}_2\text{O})_2]\text{ClO}_4$
 $M_r = 677.11$
Triclinic, $P\bar{1}$
 $a = 8.547$ (5) Å
 $b = 9.431$ (6) Å
 $c = 10.222$ (6) Å
 $\alpha = 96.143$ (10)°
 $\beta = 94.232$ (9)°
 $\gamma = 103.217$ (9)°
 $V = 793.4$ (8) Å³

$Z = 1$
 $D_x = 1.417$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 822 reflections
 $\theta = 2.5$ – 22.1 °
 $\mu = 0.68$ mm⁻¹
 $T = 298$ (2) K
Block, brown
 $0.22 \times 0.15 \times 0.13$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.866$, $T_{\max} = 0.917$
4017 measured reflections

2732 independent reflections
1653 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.045$
 $\theta_{\max} = 25.0$ °
 $h = -10 \rightarrow 9$
 $k = -7 \rightarrow 11$
 $l = -11 \rightarrow 12$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.066$
 $wR(F^2) = 0.149$
 $S = 0.90$
2732 reflections
200 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0674P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.47$ e Å⁻³
 $\Delta\rho_{\min} = -0.34$ e Å⁻³

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 and 0.96 Å, respectively, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C/N})$.

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

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