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Key indicators

Single-crystal X-ray study
T = 293 K
Mean $\sigma(\text{C}-\text{C}) = 0.009 \text{ \AA}$
Disorder in solvent or counterion
R factor = 0.105
wR factor = 0.231
Data-to-parameter ratio = 14.9

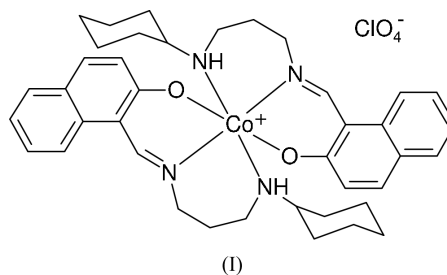
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

trans-Bis{1-[3-(cyclohexylamino)propylimino-methyl]-2-naphtholato- $\kappa^3\text{O},\text{N},\text{N}'$ }cobalt(III) perchlorate

In the title compound, $[\text{Co}(\text{C}_{20}\text{H}_{25}\text{N}_2\text{O})_2]\text{ClO}_4$, there are two independent centrosymmetric molecules of the cation, in which the central Co^{III} atoms are six-coordinated by four N atoms and two O atoms from two Schiff bases, giving approximately octahedral coordination environments.

Comment

Recently, we have reported some structures of Schiff base complexes (You, Lin *et al.*, 2003; You, Xiong *et al.*, 2004; You, Zhu & Liu, 2004; You, Chen *et al.*, 2004). As an extension of our work on the structural characterization of Schiff base complexes, a mononuclear cobalt(III) complex, (I), is reported here. It is similar to $[\text{Co}(\text{C}_{16}\text{H}_{23}\text{N}_2\text{O})_2]\text{ClO}_4$, which we reported recently (You, Qu *et al.*, 2003).



The title complex, (I), is the perchlorate salt of discrete centrosymmetric mononuclear cobalt(III) complex cations (Fig. 1). The structure is similar to the complex cited above, with the ligand 2-[(3-cyclohexylaminopropylimino)methyl]phenolate replaced by 3-[(3-cyclohexylaminopropylimino)methyl]naphthalen-2-olate. The structure contains two independent centrosymmetric molecules of the cation. Both Co^{III} atoms in the complex are in a slightly distorted octahedral geometry and are six-coordinated by two O atoms and four N atoms from two Schiff base ligands. The bond lengths and angles involving the Co atoms are the same (within experimental error) in each molecule (Table 1) and are comparable with those of the related complex (You, Qu *et al.*, 2003). The conformation of each of the six-membered rings containing the Co, azomethine N, amine N and three C atoms is a twisted chair. As expected, the cyclohexyl groups adopt chair conformations to minimize steric effects, as observed in the related complex. The unique ClO_4^- anion is disordered over two sites with occupancies 0.50:0.50.

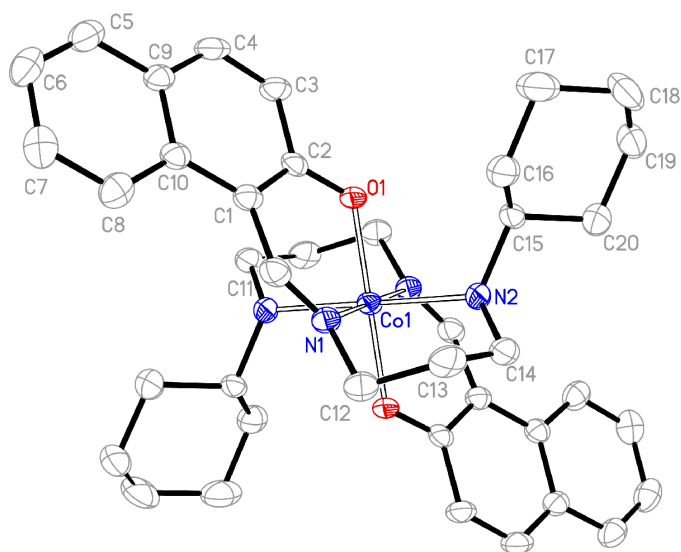
Experimental

N-Cyclohexyl-1,3-diaminopropane and 2-hydroxy-1-naphthaldehyde were obtained commercially and were used without further purification. *N*-Cyclohexyl-1,3-diaminopropane (0.2 mmol, 31.2 mg) and

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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. The disordered perchlorate anions have been omitted. Unlabelled atoms are related by the symmetry code $(2 - x, 2 - y, -z)$.

2-hydroxy-1-naphthaldehyde (0.2 mmol, 17.2 mg) were dissolved in ethanol (10 ml). The mixture was stirred for 1 h to give a clear yellow solution of *L* (0.2 mmol), where *L* is 3-[(3-cyclohexylaminopropyl-imino)methyl]naphthalen-2-ol. To the solution of *L* was added an ethanol solution (10 ml) of $\text{Cu}(\text{ClO}_4)_2 \cdot 7\text{H}_2\text{O}$ (0.1 mmol, 38.9 mg) with stirring. After allowing the resulting solution to stand in air for 11 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 ethanol and dried in a vacuum desiccator using anhydrous CaCl_2 (yield 71.3%). Analysis found: C 61.5, H 6.6, N 7.3%; calculated for $\text{C}_{40}\text{H}_{50}\text{ClCoN}_4\text{O}_6$: C 61.8, H 6.5, N 7.2%.

Crystal data

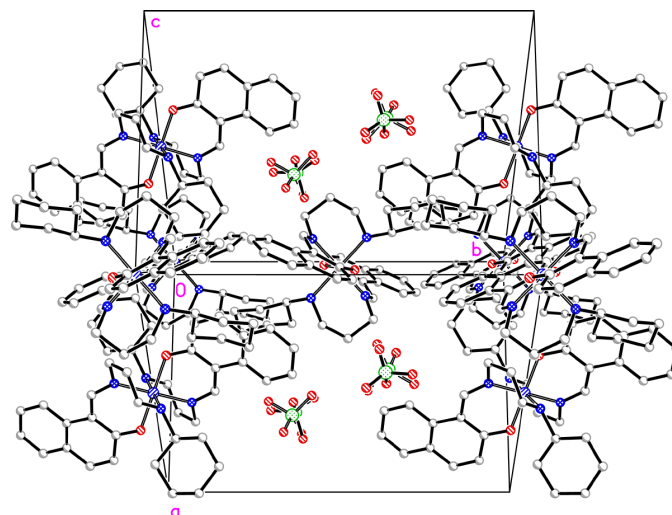
| | |
|---|---|
| $[\text{Co}(\text{C}_{20}\text{H}_{25}\text{N}_2\text{O})_2]\text{ClO}_4$ | $D_x = 1.354 \text{ Mg m}^{-3}$ |
| $M_r = 777.22$ | Mo $K\alpha$ radiation |
| Monoclinic, $P2_1/n$ | Cell parameters from 3603 reflections |
| $a = 12.167(2) \text{ \AA}$ | $\theta = 2.2\text{--}18.5^\circ$ |
| $b = 17.195(2) \text{ \AA}$ | $\mu = 0.57 \text{ mm}^{-1}$ |
| $c = 19.128(2) \text{ \AA}$ | $T = 293(2) \text{ K}$ |
| $\beta = 107.640(2)^\circ$ | Block, brown |
| $V = 3813.6(9) \text{ \AA}^3$ | $0.28 \times 0.22 \times 0.17 \text{ mm}$ |
| $Z = 4$ | |

Data collection

| | |
|---|--|
| Bruker SMART CCD diffractometer | 7707 independent reflections |
| ω scans | 5356 reflections with $I > 2\sigma(I)$ |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1996) | $R_{\text{int}} = 0.088$ |
| $T_{\text{min}} = 0.856, T_{\text{max}} = 0.909$ | $\theta_{\text{max}} = 26.5^\circ$ |
| 38 653 measured reflections | $h = -15 \rightarrow 15$ |
| | $k = -21 \rightarrow 21$ |
| | $l = -24 \rightarrow 23$ |

Refinement

| | |
|---------------------------------|--|
| Refinement on F^2 | $w = 1/[\sigma^2(F_o^2) + (0.0877P)^2 + 4.2908P]$ |
| $R[F^2 > 2\sigma(F^2)] = 0.105$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2) = 0.231$ | $(\Delta/\sigma)_{\text{max}} < 0.001$ |
| $S = 1.16$ | $\Delta\rho_{\text{max}} = 1.24 \text{ e \AA}^{-3}$ |
| 7707 reflections | $\Delta\rho_{\text{min}} = -0.34 \text{ e \AA}^{-3}$ |
| 517 parameters | |
| H-atom parameters constrained | |


Figure 2

The crystal packing of (I), showing cations and disordered perchlorate molecules. All H atoms have been omitted.

Table 1

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

| | | | |
|-------------------------|------------|--------------------------|------------|
| Co1—O1 | 1.892 (3) | Co2—O2 | 1.890 (4) |
| Co1—N1 | 1.933 (4) | Co2—N3 | 1.922 (4) |
| Co1—N2 | 2.026 (4) | Co2—N4 | 2.019 (5) |
| O1—Co1—O1 ⁱ | 180 | O2 ⁱⁱ —Co2—O2 | 180 |
| O1—Co1—N1 ⁱ | 89.65 (16) | O2—Co2—N3 ⁱⁱ | 89.98 (18) |
| O1—Co1—N1 | 90.35 (16) | O2—Co2—N3 | 90.02 (18) |
| N1 ⁱ —Co1—N1 | 180 | N3 ⁱⁱ —Co2—N3 | 180 |
| O1—Co1—N2 | 92.75 (15) | O2—Co2—N4 | 88.94 (19) |
| N1—Co1—N2 | 90.75 (17) | N3—Co2—N4 | 88.8 (2) |
| O1—Co1—N2 ⁱ | 87.25 (15) | O2—Co2—N4 ⁱⁱ | 91.06 (19) |
| N1—Co1—N2 ⁱ | 89.25 (17) | N3—Co2—N4 ⁱⁱ | 91.2 (2) |
| N2—Co1—N2 ⁱ | 180 | N4—Co2—N4 ⁱⁱ | 180 |

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

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H and N—H distances of 0.93–0.98 \AA and 0.91 \AA , respectively, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$. The largest peak in the final difference Fourier (1.24 e \AA^{-3}) is 2.40 \AA from H33A and the next highest peak is 0.55 e \AA^{-3} . The value of the *R* factor is higher than normal (0.105) and is probably due to the poor diffraction quality of the crystal and the effect of the disordered anion. This lowers the precision of the structure.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT; 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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References

Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.

- Sheldrick, G. M. (1997a). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997b). *SHELXTL*. Version 5.1 Software. Bruker AXS Inc., Madison, Wisconsin, USA.
- Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- You, Z.-L., Chen, B., Zhu, H.-L. & Liu, W.-S. (2004). *Acta Cryst.* **E60**, m884–m886.
- You, Z.-L., Lin, Y.-S., Liu, W.-S., Tan, M.-Y. & Zhu, H.-L. (2003). *Acta Cryst.* **E59**, m1025–m1027.
- You, Z.-L., Qu, Y., Liu, W.-S., Tan, M.-Y. & Zhu, H.-L. (2003). *Acta Cryst.* **E59**, m1038–m1040.
- You, Z.-L., Xiong, Z.-D., Liu, W.-S., Tan, M.-Y. & Zhu, H.-L. (2004). *Acta Cryst.* **E60**, m79–m81.
- You, Z.-L., Zhu, H.-L. & Liu, W.-S. (2004). *Acta Cryst.* **E60**, m560–m562.