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{2,2′-Bis[(3-aminopropyl)iminomethyl]-oxalanilide}cobalt(III) perchlorate

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The title compound, $[Co(C_{22}H_{26}N_6O_2)]ClO_4$, was prepared from diformyloxanilide, cobalt(II) perchlorate and 1,3-propanediamine. The crystal structure contains a complex cobalt(III) cation and a perchlorate anion. Cobalt is coordinated by six N atoms from the anilide ligand in chelating mode, forming a distorted octahedral geometry. Inter-cation hydrogen bonds are present in the crystal structure.

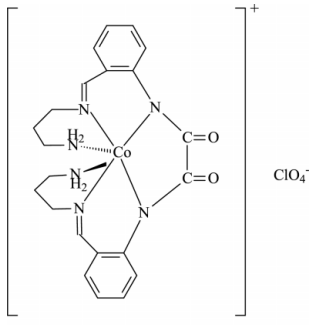
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.054
wR factor = 0.118
Data-to-parameter ratio = 15.0

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

Comment

The coordination properties *N,N*-bis-oxamides have been thoroughly investigated, both in aqueous solution and in the solid state (Ruiz *et al.*, 1999). In the presence of metal ions and when the oxamide has another coordinating group at a position which can form five- or six-membered chelate rings, the amide group deprotonates and coordinates simultaneously in low pH range. Recently, an increasing interest has been shown in the design of mononuclear oxamidate-bridged complexes. However, most of the studies were focused on the mononuclear oxamidate-bridged copper complex (Tang *et al.*, 2002); by comparison, the oxamidate-bridged cobalt(III) complex has hardly been investigated.



(I)

The crystal structure of the title complex, (I), is composed of a {2,2′-bis[(3-aminopropyl)iminomethyl]oxalanilide}-cobalt(III) cation (Fig. 1) and a perchlorate anion. The ligand coordinates to the cobalt(III) ion in chelating mode, leading to a distorted octahedron. The N—Co—N angles involving adjacent vertices of the octahedron are in the range 82.9 (1)–96.5 (1)°, while the range of those involving opposite vertices is 173.5 (1)–178.0 (1)°, and Co—N bonds lengths are 1.908 (3)–2.016 (3) Å. The packing diagram (Fig. 2) shows the N—H···O hydrogen bonds between neighboring cations, giving chains.

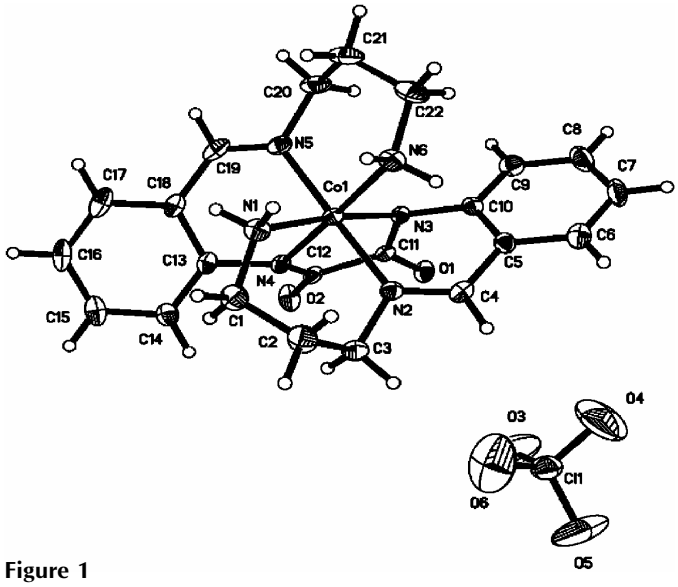


Figure 1
View of the title compound, with displacement ellipsoids shown at the 50% probability level.

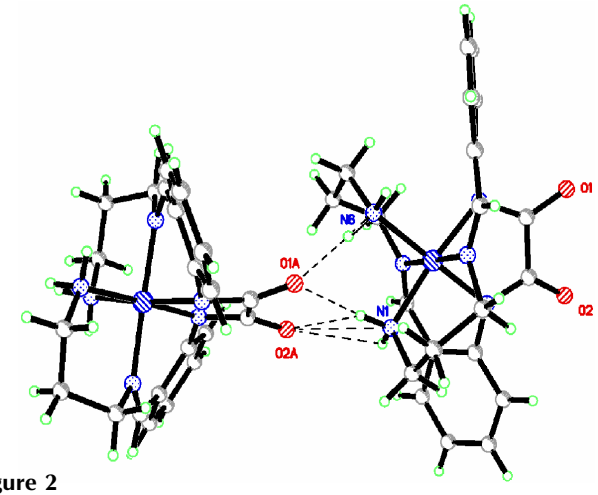


Figure 2
View of the hydrogen-bond interactions (shown as dashed lines).

Experimental

The title compound, (I), was prepared by refluxing and stirring diformyloxanilide (2.49 g, 0.01 mol), 1,3-propanediamine (1.6 ml, 0.02 mol) and cobalt(II) perchlorate for 3.5 h in 50 ml of MeOH in the presence of four drops of 2 *M* NaOH. After the mixture was cooled and filtered, the resulting precipitate was washed with water, methanol and diethyl ether successively, and dried under vacuum. The resulting deep-red filtrate, kept at room temperature for several days, produced red prismatic crystals suitable for X-ray analysis.

Crystal data

$[Co(C_{22}H_{26}N_6O_2)]ClO_4$
M_r = 564.87
Monoclinic, $P2_1/c$
a = 9.728 (3) Å
b = 17.232 (5) Å
c = 14.953 (5) Å
β = 108.076 (6)°
V = 2382.8 (13) Å^3
Z = 4
D_x = 1.575 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 897 reflections
θ = 2.2–23.3°
μ = 0.89 mm^{-1}
T = 293 (2) K
Prism, red
0.32 × 0.24 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.743, T_{max} = 0.838
11 184 measured reflections
4873 independent reflections
2884 reflections with $I > 2\sigma(I)$
R_{int} = 0.064
θ_{max} = 26.4°
$h = -12 \rightarrow 10$
$k = -21 \rightarrow 15$
$l = -6 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.118
S = 0.99
4873 reflections
325 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0383P)^2 + 1.7668P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1C···O1^{i}	0.90	2.17	3.057 (5)	168
N1—H1C···O2^{i}	0.90	2.37	2.876 (4)	115
N6—H6B···O1^{i}	0.90	2.05	2.929 (4)	166
N1—H1D···O2^{i}	0.90	2.52	2.876 (4)	104
C1—H1B···O6^{ii}	0.97	2.45	3.294 (8)	145
C14—H14A···O4^{iii}	0.93	2.55	3.182 (8)	126
C16—H16A···O1^{iv}	0.93	2.43	3.314 (5)	158

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

H atoms were positioned geometrically (N—H = 0.90 Å, C—H = 0.93–0.97 Å) and refined using a riding model, with U_{iso} = 1.2U_{eq}(C,N).

Data collection: *SMART* (Bruker, 1999); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1999); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL* and *WinGX* (Farrugia, 1999).

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