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Bis(3,5-dicarboxybenzoato-κ^2O,O')(1,10-phenanthroline)cobalt(II)

Jin-Yu Han, Wen-Ying Wei, Xiao Dou and He-Ying Chang*

Key Laboratory for Green Chemical Technology of the State Education Ministry, School of Chemical Engineering and Technology, Tianjin University, Tianjin 300072, People's Republic of China

Correspondence e-mail: wwy7324@eyou.com

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.037
wR factor = 0.108
Data-to-parameter ratio = 11.2

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

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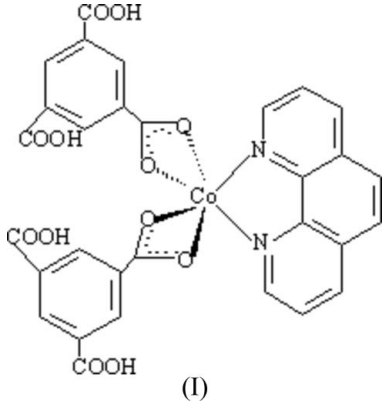
The title molecule, $[Co(C_9H_5O_6)_2(C_{12}H_8N_2)]$ or $[Co(H_2BTC)_2(phen)]$ (BTC = benzene-1,3,5-tricarboxylate and phen = 1,10-phenanthroline), has crystallographic twofold symmetry. The Co^{II} atom has a coordination geometry that is intermediate between octahedral and trigonal prismatic. In the crystal structure, molecules are connected *via* O—H···O hydrogen bonds [O···O = 2.621 (4)–2.684 (4) Å] to form a three-dimensional network.

Comment

The title compound is shown in Fig. 1 and selected bond lengths and angles are given in Table 1. The asymmetric unit contains one half of the cobalt complex, with the Co^{II} atom residing on a crystallographic twofold axis, and the complete molecule is generated by the action of the twofold symmetry. The coordination geometry of the four O atoms and two N atoms bonded to the Co^{II} atom is intermediate between octahedral and trigonal prismatic (see Fig. 2). This arrangement appears to be the effect of the small bite angles produced by the chelating ligands.



(I)

In the crystal structure, molecules are connected *via* O—H···O hydrogen bonds (Table 2) to form a three-dimensional network. The crystal structure of bis(dihydrogen benzene-1,3,5-tricarboxylato-O,O')-(1,10-phenanthroline)copper(II) has previously been determined (Hu *et al.*, 2004) and it is isostructural with (I).

Experimental

An aqueous solution (3 ml) of benzene-1,3,5-tricarboxylic acid (H_3BTC) (0.210 g) and 1,10-phenanthroline (phen) (0.198 g) was mixed with an aqueous solution (2 ml) of cobalt nitrate hexahydrate (0.146 g) and zinc nitrate hexahydrate (0.149 g) with continuous stirring. The mixture was sealed in a 25 ml Teflon-lined stainless steel

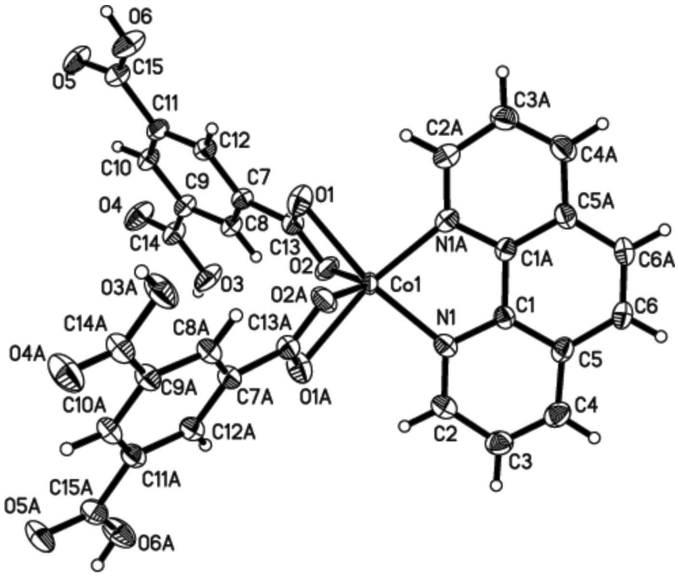


Figure 1
A view of the title complex, with 30% probability displacement ellipsoids (arbitrary spheres for H atoms). [Symmetry code: (A) $2-x, y, \frac{1}{2}-z$.]

vessel and heated at 373 K for 72 h under autogenous conditions. After cooling to room temperature, the resulting product was filtered off to obtain purple crystals of (I) (about 78.6% yield based on the Co source). Spectroscopic analysis: IR (KBr, ν, cm^{-1}): 3483, 3012, 1719, 1691, 1618, 1587, 1570, 1400, 1520, 1429, 1294, 937, 848, 727, 673, 528, 420. Elemental analysis, calculated for $C_{30}H_{18}CoN_2O_{12}$: C 54.76, H 2.74%; found: C 55.08, H 2.65%.

Crystal data

$[Co(C_9H_5O_6)_2(C_{12}H_8N_2)]$
M_r = 657.39
Monoclinic, $C2/c$
a = 9.912 (8) Å
b = 15.954 (13) Å
c = 16.685 (13) Å
β = 94.655 (11)°
V = 2630 (4) Å^3
Z = 4
D_x = 1.660 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1223 reflections
θ = 2.4–22.7°
μ = 0.73 mm^{-1}
T = 293 (2) K
Block, purple
0.22 × 0.18 × 0.12 mm

Data collection

Siemens SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.680, T_{max} = 0.920
6961 measured reflections
2321 independent reflections
1777 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 25.0°
$h = -11 \rightarrow 11$
$k = -18 \rightarrow 9$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.108
S = 1.04
2321 reflections
207 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0524P)^2 + 2.1449P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997*a*)
Extinction coefficient: 0.0025 (4)

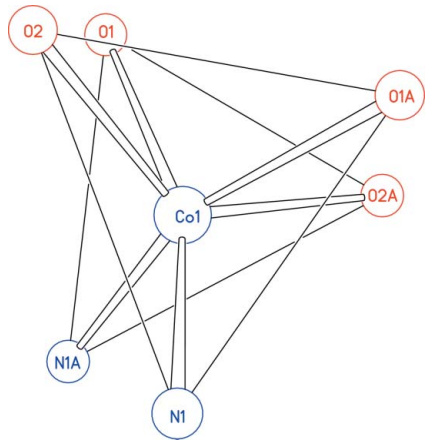


Figure 2
Diagram showing a projection of the six atoms coordinated to Co1 in the title complex. The two triangles shown are intermediate between the eclipsed and staggered arrangements for the respective ideal trigonal prismatic and octahedral coordination geometries [symmetry code: (A) $2-x, y, \frac{1}{2}-z$.]

Table 1
Selected geometric parameters (Å, °).

Co1—N1	2.068 (3)	Co1—O1	2.258 (3)
Co1—O2	2.121 (3)		
N1^{i}—Co1—N1	80.70 (15)	O2—Co1—O1^{i}	87.55 (10)
N1—Co1—O2	105.35 (9)	N1—Co1—O1	159.44 (9)
N1—Co1—O2^{i}	112.90 (9)	O2—Co1—O1	59.64 (9)
O2—Co1—O2^{i}	129.19 (14)	O1^{i}—Co1—O1	100.60 (14)
N1—Co1—O1^{i}	92.14 (11)		

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

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O2^{ii}	0.82	1.92	2.684 (4)	154
O6—H6···O5^{iii}	0.82	1.80	2.621 (4)	174

Symmetry codes: (ii) $-x+\frac{3}{2}, -y+\frac{1}{2}, -z+1$; (iii) $-x+3, -y, -z+1$.

All H atoms were placed in geometrically idealized positions (C—H = 93 Å and O—H = 0.82 Å) and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(O).

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

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