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Diazidobis(1,10-phenathroline)cobalt(III) nitrate

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Key indicators

Single-crystal X-ray study
T = 223 K
Mean σ(C–C) = 0.003 Å
R factor = 0.033
wR factor = 0.082
Data-to-parameter ratio = 18.0

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

The Co atom and nitrate anion in the title compound, $[Co(C_{12}H_8N_2)_2(N_3)_2]NO_3$, are each disposed about a twofold axis of symmetry. The Co atom exists in a distorted octahedral geometry, with the azide ligands *cis* to each other.

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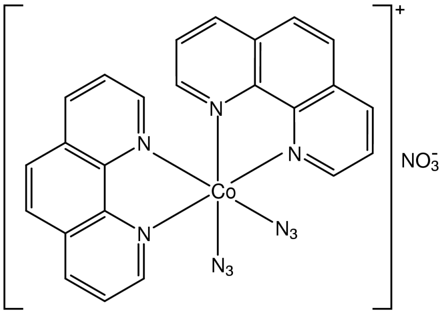
Comment

The asymmetric unit in $[Co(phen)_2(N_3)_2]NO_3$, (I), comprises half each of a cation and anion, as each is located on a crystallographic twofold axis. In the cation (Fig. 1), the Co atom is hexacoordinate, existing in a distorted octahedral N_6 geometry with the maximum deviation from the ideal being no more than 6.5°. The azide ligands occupy *cis*-positions. The Co–N distances *trans* to the azide ligands [Co—N5 = 1.9518(16) Å] are longer than the Co—N4 distances [1.9358 (12) Å] that are not (Table 1). A similar coordination geometry and pattern of Co—N bond distances was reported recently for, arguably, the most closely related structure in the literature, *viz.* $\{Co[(NC_5H_4)NH(C_5H_4N)]_2(N_3)_2\}ClO_4$ (Du *et al.*, 2001). The crystal structure features columns comprised of cations, interspersed by columns of anions, all running parallel to the *c* direction. Links between cations of neighbouring columns are afforded by C12—H$\cdots$N3^{i} interactions involving the terminal N atoms of the azide ligands; H$\cdots$N3^{i} = 2.55 Å, C12$\cdots$N^{i} 3.445 (3) Å, angle at H = 158° [symmetry code: (i) $\frac{1}{2}+x, \frac{1}{2}+y, z-\frac{1}{2}$]. Links between anions and cations are of the type C—H$\cdots$O, the shortest of these being C9$\cdots$O2^{ii} is 3.142 (3) Å [symmetry code: (ii) $-\frac{1}{2}-x, \frac{1}{2}+y, z$].



(I)

Experimental

Single crystals of $[Co(phen)_2(N_3)_2]NO_3$ were grown by slow diffusion, using an H-double-tube glass vessel with an aqueous solution of $Co(phen)(NO_3)_2$ (0.01 M) in one arm and NaN_3 (0.02 M) in the other. After two months, dark-brown crystals precipitated.

Crystal data

$[Co(C_{12}H_8N_2)_2(N_3)_2]NO_3$
$M_r = 565.41$
Orthorhombic, *Iba*2
$a = 15.4858$ (6) Å
$b = 9.7359$ (4) Å
$c = 14.7769$ (8) Å
$V = 2227.89$ (17) Å^3
$Z = 4$
$D_x = 1.686$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 3743 reflections
$\theta = 2.5$–$29.0°$
$\mu = 0.83$ mm^{-1}
$T = 223$ (2) K
Block, brown
0.23 × 0.23 × 0.05 mm

Data collection

Bruker AXS SMART CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*, Bruker, 2000; Blessing, 1995)
$T_{min} = 0.739$, $T_{max} = 0.960$
8877 measured reflections

3200 independent reflections
2868 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 30.0°$
$h = -16 \rightarrow 21$
$k = -13 \rightarrow 11$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.082$
$S = 1.00$
3200 reflections
178 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0514P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.56$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}
Absolute structure: Flack (1983), 1511 Friedel pairs
Flack parameter = −0.033 (14)

Table 1
Selected geometric parameters (Å, °).

Co—N1	1.9320 (18)	O2—N6	1.240 (3)
Co—N4	1.9358 (12)	N1—N2	1.198 (2)
Co—N5	1.9518 (16)	N2—N3	1.152 (2)
O1—N6	1.233 (6)		
N1—Co—N4	90.16 (7)	N4—Co—N4^i	179.79 (12)
N1—Co—N5	173.57 (8)	N4—Co—N5^i	96.06 (7)
N1—Co—N1^i	93.80 (12)	N5—Co—N5^i	88.68 (9)
N1—Co—N4^i	89.69 (7)	Co—N1—N2	120.19 (15)
N1—Co—N5^i	89.06 (6)	N1—N2—N3	175.6 (2)
N4—Co—N5	84.09 (7)		

Symmetry code: (i) $-x, -y, z$.

The H atoms were included in the riding-model approximation, with C—H distances of 0.94 Å and $U_{iso}(H) = 1.2U_{eq}$(parent atom).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SHELXTL* (Bruker, 2000); program(s) used to solve structure: *PATTY* in *DIRDIF*92 (Beurskens *et al.*, 1992); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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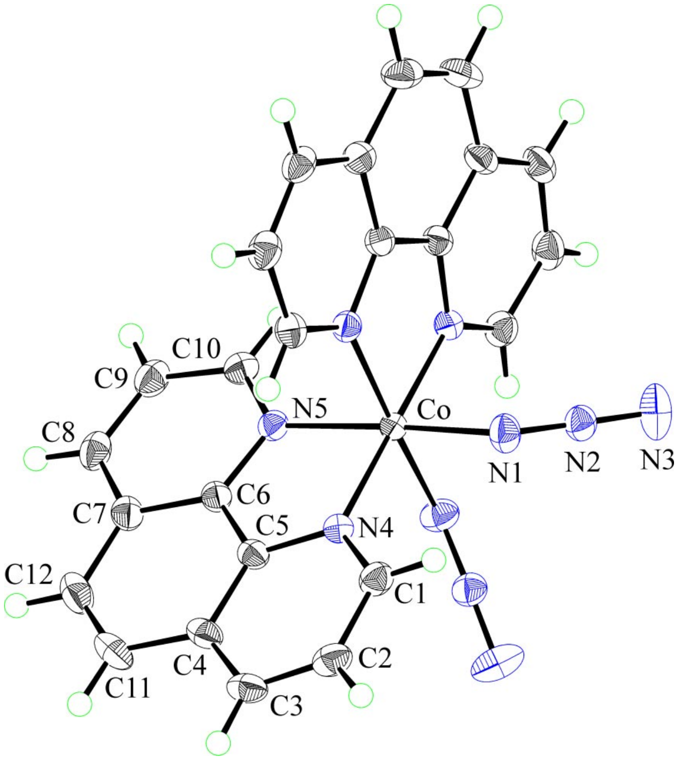

Figure 1
The molecular structure and crystallographic numbering scheme for the cation in $[Co(phen)_2(N_3)_2]NO_3$. The unlabelled half of the molecule is generated by the symmetry operation ($-x$, $-y$, z). Displacement ellipsoids are drawn at the 50% probability level (Johnson, 1976).

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