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Bis[bis(2-pyridyl)amine-*N*,*N*′](nitrato-*O*,*O*′)cobalt(**II**) nitrate

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Key indicators

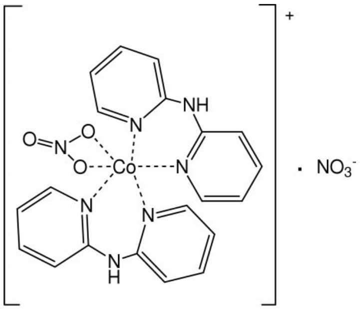
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.034
wR factor = 0.079
Data-to-parameter ratio = 12.5

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

The crystal structure of the title compound, $[Co(NO_3)(C_{10}H_9N_3)_2]NO_3$, consists of mononuclear cations and uncoordinated nitrate anions held together by means of an extensive three-dimensional network of N—H···O and C—H···O hydrogen bonds. The Co^{II} centre has a distorted octahedral geometry, coordinated with two O atoms of a bidentate nitrate ligand and with four pyridine N atoms of two bis(2-pyridyl)amine ligands in a *trans–trans* mode.

Comment

During our studies of first-row transition metal complexes with N-donor aromatic ligands (Castillo *et al.*, 2001; Castillo *et al.*, 2000), we have obtained the compound bis[bis(2-pyridyl)amine-*N*,*N*′](nitrato-*O*,*O*′)cobalt(II) nitrate, (I).



(I)

Ligands containing aromatic nitrogen heterocycles play an important role in the molecular self-assembling process that leads to supramolecular architectures (Jones, 1998). The crystal structure of (I) comprises discrete $[Co(NO_3)(C_{10}H_9N_3)_2]^+$ cations and NO_3^- anions. The Co^{II} centre is six-coordinated by four pyridine N atoms of two bis(2-pyridyl)amine moieties and by two O atoms of a bidentate nitrate anion, as shown in Fig. 1. The bidentate bis(2-pyridyl)amine ligands chelate the Co^{II} atom to form two six-membered coordination rings. The Co—N bond distances range from 1.935 (2) to 1.947 (2) Å. The pyridine rings in the same ligand are in the normal *trans–trans* mode (Du *et al.*, 2001) and the dihedral angles are 27.7 (1) and 37.4 (1)°, respectively. The nitrate ligand is coordinated with two essentially equivalent Co—O bond distances, 1.907 (2) and 1.909 (2) Å. The main distortion of the octahedral geometry is due to the small O—Co—O bite angle [69.15 (9)°] of the bidentate nitrate anion. The N—O bond distance involving the uncoordinated O atom of the nitrato ligand is significantly shorter than those for the coordinated O atoms, but is similar to those of the nitrate anion.

In the crystal structure of (I), the cations and nitrate anions are held together by means of an extensive network of N—H···O and C—H···O hydrogen bonds (Fig. 2).

Experimental

An aqueous solution (30 ml) of bis(2-pyridyl)amine (0.340 g, 2.0 mmol) was added slowly to an aqueous solution (30 ml) of $Co(NO_3)_2.6H_2O$ (0.290 g, 1.0 mmol) with vigorous stirring. The resulting solution was filtered off to remove impurities and then allowed to stand at room temperature. After two weeks, red crystals were harvested by slow evaporation of the solvent. Crystals were found to be stable to air and X-ray exposure.

Crystal data

$[Co(NO_3)(C_{10}H_9N_3)_2]NO_3$
$M_r = 525.35$
Monoclinic, $P2_1/n$
$a = 17.191$ (3) Å
$b = 7.308$ (1) Å
$c = 17.843$ (5) Å
$\beta = 104.94$ (3)°
$V = 2165.9$ (8) Å^3
$Z = 4$
$D_x = 1.611$ Mg m^{-3}
$D_m = 1.62$ (1) Mg m^{-3}
D_m measured by flotation in a mixture of carbon tetrachloride and bromoform
Mo $K\alpha$ radiation
Cell parameters from 14 084 reflections
$\theta = 2.5$–25.8°
$\mu = 0.85$ mm^{-1}
$T = 293$ (2) K
Irregular, red
0.42 × 0.20 × 0.08 mm

Data collection

Stoe IPDS diffractometer
Area-detector (φ) scans
Absorption correction: numerical (Stoe & Cie, 1998)
$T_{min} = 0.815$, $T_{max} = 0.934$
14 084 measured reflections
4037 independent reflections
2598 reflections with $I > 2\sigma(I)$
$R_{int} = 0.048$
$\theta_{max} = 25.8$°
$h = -21 \rightarrow 20$
$k = -8 \rightarrow 8$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.079$
$S = 1.04$
4037 reflections
324 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0787P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.37$ e Å^{-3}
$\Delta\rho_{min} = -0.52$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Co1—O1	1.909 (2)	O1—N1	1.335 (4)
Co1—O2	1.907 (2)	O2—N1	1.337 (4)
Co1—N11	1.935 (2)	O3—N1	1.216 (4)
Co1—N19	1.947 (2)	O4—N2	1.246 (4)
Co1—N21	1.935 (2)	O5—N2	1.215 (4)
Co1—N29	1.935 (2)	O6—N2	1.213 (4)
O1—Co1—O2	69.15 (9)	N11—Co1—N29	174.66 (9)
O1—Co1—N11	88.41 (9)	N19—Co1—N21	94.67 (9)
O1—Co1—N19	165.50 (9)	N19—Co1—N29	94.94 (9)
O1—Co1—N21	99.81 (9)	N21—Co1—N29	88.51 (9)
O1—Co1—N29	86.31 (9)	O1—N1—O2	108.3 (3)
O2—Co1—N11	88.49 (9)	O1—N1—O3	126.1 (3)
O2—Co1—N19	96.38 (9)	O2—N1—O3	125.6 (3)
O2—Co1—N21	168.95 (9)	O4—N2—O6	120.7 (3)
O2—Co1—N29	90.37 (9)	O4—N2—O5	119.7 (3)
N11—Co1—N19	90.37 (9)	O5—N2—O6	119.6 (3)
N11—Co1—N21	91.61 (9)		

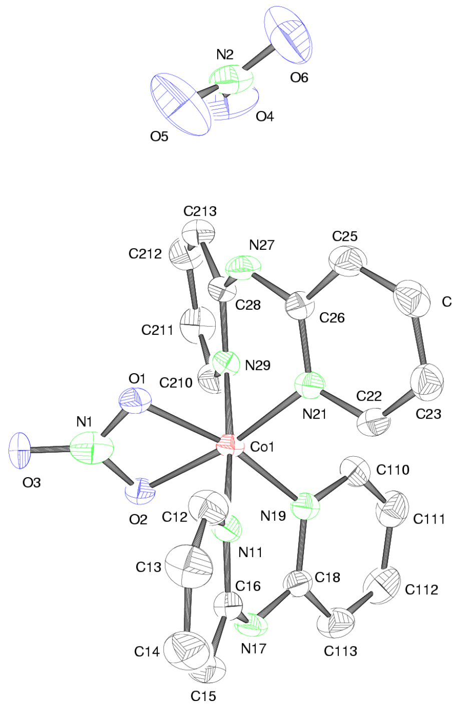


Figure 1
The asymmetric unit of (I) with 50% probability displacements ellipsoids. H atoms have been omitted for clarity.

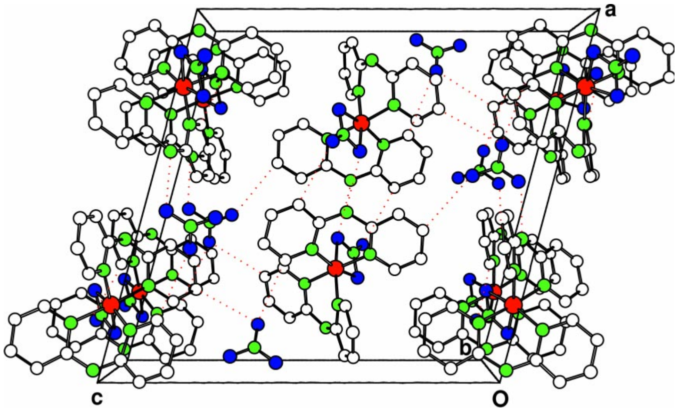


Figure 2
Packing diagram of (I). Dotted lines show hydrogen bonds. H atoms have been omitted for clarity.

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

D—H···A	D—H	H···A	D···A	D—H···A
N17—H17···O2^{i}	0.78 (3)	2.15 (3)	2.884 (3)	157 (3)
N27—H27···O4^{ii}	0.84 (3)	2.00 (3)	2.842 (4)	179 (3)
C13—H13···O1^{iii}	0.93	2.57	3.400 (4)	149
C14—H14···O5^{iv}	0.93	2.57	3.404 (5)	148
C22—H22···O3^{v}	0.93	2.35	3.224 (4)	157
C110—H110···O6^{vi}	0.93	2.51	3.418 (4)	167
C113—H113···O3^{i}	0.93	2.44	3.343 (4)	163
C211—H211···O6^{vii}	0.93	2.40	3.324 (4)	171

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

H atoms bonded to C atoms were kept fixed at calculated positions with U_{iso} 20% larger than U_{eq} for the atoms to which they are

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attached. H atoms bonded to N atoms were located in difference Fourier maps and refined isotropically.

Data collection: *IPDS Software* (Stoe & Cie, 1998); cell refinement: *IPDS Software*; data reduction: *IPDS Software*; program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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