metal-organic papers

(Ethane-1,2-diamine)dinitratopalladium(II)

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Key indicators

Single-crystal X-ray study
$T = 150$ K
Mean σ(C–C) = 0.002 Å
R factor = 0.016
wR factor = 0.043
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.

The title compound, $[Pd(NO_3)_2(C_2H_8N_2)]$, forms an infinite two-dimensional sheet-like motif, propagated by intermolecular hydrogen bonds between the amino groups of the ethane-1,2-diamine ligands and the nitrate O atoms. There are two complex molecules in the asymmetric unit.

Comment

Our group has long been interested in the use of metal complexes as components for the construction of large supramolecular architectures (Lindoy & Atkinson, 2000). In particular, we are interested in the construction and chemistry of metallocyclic systems (Clegg *et al.*, 2004, 2005). The title compound, (I), has found extensive use as a precursor in the preparation of cyclic metallo-supramolecular structures (Fujita *et al.*, 2005). Crystals suitable for this study were obtained in the course of our investigation into the interactions of N-donor ligand systems (Bray *et al.*, 2005) with (I).

(I)
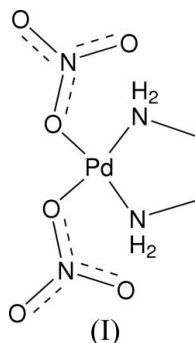
An *ORTEP* (Farrugia, 1997) representation of (I) is given in Fig. 1. As expected, each Pd[II] ion has a geometry close to an ideal square-plane (Table 1). The N donor atoms of the bidentate ethane-1,2-diamine ligand (en) occupy two coordination sites in a typical five-membered chelate arrangment. The remaining coordination sites are occupied by nitrate O atoms of two nitrate ligands.

The asymmetric unit contains two of these complexes, which pack *via* intermolecular hydrogen bonds between the NH$_2$ groups of the en ligands and the O atoms of the nitrate ligands. Hydrogen-bond details are provided in Table 2.

The intermolecular hydrogen bonds propagate in two dimensions, forming an infinite sheet-like motif that lies parallel to the *bc* plane (Fig. 2). Each of the N donor atoms forms hydrogen bonds to (at least) two O acceptor atoms, with only atoms O5 and O8 not involved in close interactions. The sheets stack along the *a* axis, as shown in the crystal packing diagram (Fig. 3).
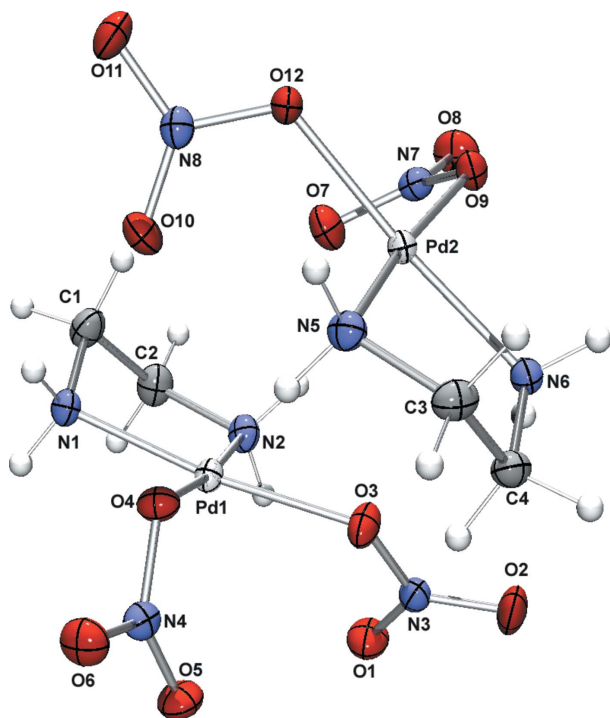
Figure 1
A representation of the asymmetric unit of (I), shown with 50% probability displacement ellipsoids.

Figure 3
A view of (I), along the *b* axis. Alternate two-dimensional sheets are shown in red and green. There are no hydrogen-bonding interactions connecting adjacent layers. There are no hydrogen-bonding interactions connecting adjacent layers (indicated by blue arrows).

Experimental

The title compound was prepared from *cis*-[Pd(en)Cl$_2$] and identified as the desired product by comparison with literature data (Fujita *et al.*, 1996; Tercero-Moreno *et al.*, 1996). Crystals of (I) suitable for the X-ray diffraction study were isolated from methanol after several days of slow evaporation. All reagents were purchased from Sigma–Aldrich.
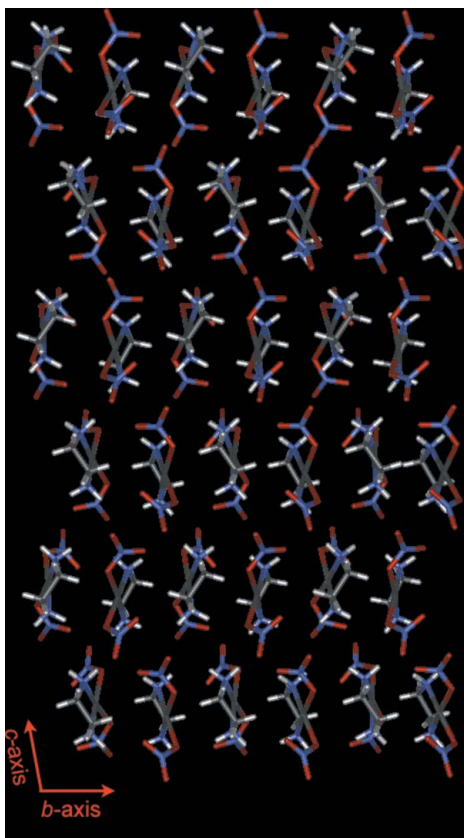

Figure 2
A view of part of one of the two-dimensional sheets formed by hydrogen bonding. The sheets extend infinitely in the *bc* plane and stack on top of each other along the *a* axis.
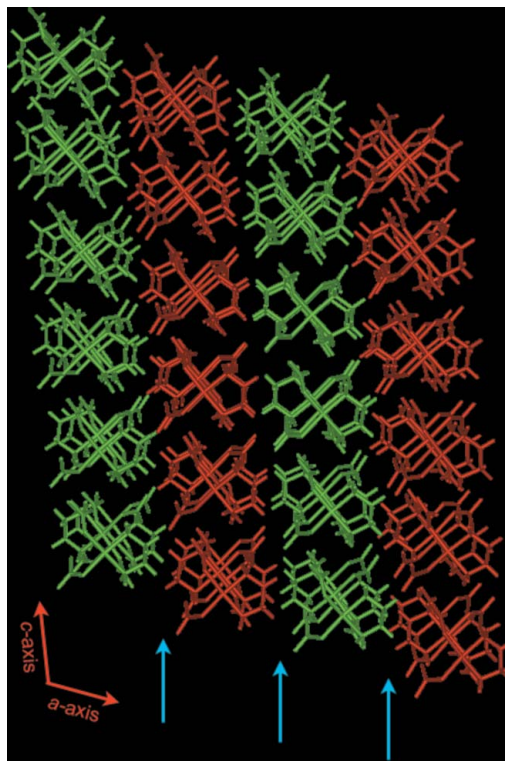
Crystal data

C$_2$H$_8$N$_4$O$_6$Pd	D_x = 2.396 Mg m^{-3}
M_r = 290.52	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 10524
a = 16.8478 (6) Å	reflections
b = 7.7746 (3) Å	θ = 2.4–28.3°
c = 13.0702 (5) Å	μ = 2.32 mm^{-1}
β = 109.816 (1)°	T = 150 (2) K
V = 1610.62 (10) Å^3	Block, colourless
Z = 8	0.51 × 0.30 × 0.16 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	3881 independent reflections
	3554 reflections with $I > 2\sigma(I)$
ω scans	R_{int} = 0.019
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1999)	θ_{max} = 28.3°
	$h = -22 \rightarrow 22$
T_{min} = 0.514, T_{max} = 0.690	$k = -10 \rightarrow 10$
15349 measured reflections	$l = -17 \rightarrow 16$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0236P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.016$	$+ 0.7251P]$
$wR(F^2) = 0.043$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} = 0.002$
3881 reflections	$\Delta\rho_{max} = 1.08$ e Å^{-3}
259 parameters	$\Delta\rho_{min} = -0.40$ e Å^{-3}
H atoms treated by a mixture of independent and constrained refinement	

Table 1
Selected geometric parameters (Å, °).

N1—Pd1	2.0032 (14)	O3—Pd1	2.0326 (12)
N2—Pd1	2.0231 (14)	O4—Pd1	2.0492 (11)
N5—Pd2	2.0102 (15)	O9—Pd2	2.0465 (12)
N6—Pd2	2.0150 (14)	O12—Pd2	2.0426 (11)
N1—Pd1—N2	83.84 (6)	N5—Pd2—N6	83.82 (6)
N1—Pd1—O3	172.80 (6)	N5—Pd2—O12	95.14 (5)
N2—Pd1—O3	94.62 (5)	N6—Pd2—O12	173.68 (5)
N1—Pd1—O4	90.81 (5)	N5—Pd2—O9	173.01 (5)
N2—Pd1—O4	173.19 (5)	N6—Pd2—O9	91.35 (5)
O3—Pd1—O4	90.16 (5)	O12—Pd2—O9	89.11 (5)

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

D—H$\cdots$$A$	D—H	H$\cdots$$A$	$D\cdots A$	D—H$\cdots$$A$
N2—H3$\cdots$O7	0.90 (2)	2.23 (2)	2.9167 (19)	134 (2)
N2—H3$\cdots$O11^i	0.90 (2)	2.45 (2)	3.0911 (19)	129 (2)
N2—H4$\cdots$O1	0.89 (2)	2.49 (2)	3.009 (2)	118 (2)
N2—H4$\cdots$O9^{ii}	0.89 (2)	2.58 (2)	3.415 (2)	158 (2)
N5—H6$\cdots$O4	0.88 (2)	2.45 (2)	3.2837 (19)	160 (2)
N5—H6$\cdots$O4	0.88 (2)	2.45 (2)	3.2837 (19)	160 (2)
N1—H2$\cdots$O11^{ii}	0.86 (2)	2.35 (2)	3.199 (2)	167 (2)
N1—H2$\cdots$O12^{ii}	0.86 (2)	2.46 (2)	3.1177 (19)	133 (2)
N1—H1$\cdots$O9^{iii}	0.88 (2)	2.24 (2)	3.0642 (18)	157 (2)
N6—H7$\cdots$O3	0.88 (2)	2.39 (2)	2.9550 (18)	122 (2)
N6—H7$\cdots$O10^i	0.88 (2)	2.50 (2)	3.1324 (19)	130 (2)
N6—H7$\cdots$O2	0.88 (2)	2.58 (2)	3.363 (2)	149 (2)
N6—H8$\cdots$O4^i	0.88 (2)	2.38 (2)	3.1695 (18)	150 (2)
N6—H8$\cdots$O6^i	0.88 (2)	2.40 (2)	3.1628 (19)	145 (2)

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

C-bound H atoms were included in idealized positions and refined using a riding-model approximation, with methylene C—H bond lengths fixed at 0.99 Å. N-bound H atoms were located in a difference Fourier map and refined with bond-length restraints of 0.90 (2) Å. U_{iso}(H) values were fixed at 1.2U_{eq}(C) and 1.5U_{eq}(N). The maximum residual electron-density peak is located 0.86 Å from atom Pd2.

Data collection: *SMART* (Bruker, 1995); cell refinement: *SAINT* (Bruker, 1995); data reduction: *SAINT* and *XPREP* (Bruker, 1995); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3* (Farrugia, 1997) and *WinGX32* (Farrugia, 1999); software used to prepare material for publication: *enCIFer* (Allen *et al.*, 2004).

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