

***trans*-Bis(1-cyclohexylpyrrolidin-2-one)-dinitratopalladium(II)**

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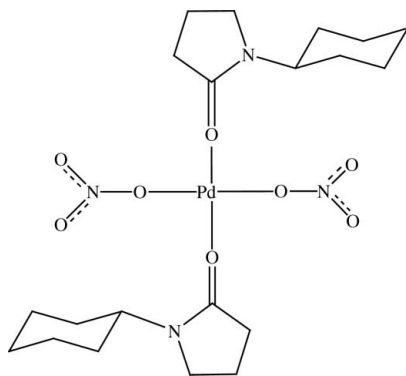
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Key indicators: single-crystal X-ray study;  $T = 173$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.020;  $wR$  factor = 0.052; data-to-parameter ratio = 17.7.

In the title compound,  $[\text{Pd}(\text{NO}_3)_2(\text{C}_{10}\text{H}_{17}\text{NO})_2]$ , the  $\text{Pd}^{\text{II}}$  centre is located on an inversion center and is coordinated in a square-planar geometry by two O atoms of the monodentate nitrate groups and two carbonyl O atoms of the 1-cyclohexylpyrrolidin-2-one ligands.

**Related literature**

For general background to ambidentate ligands, see: Fairlie & Taube (1985); Rack *et al.* (2003); Sigel & Martin (1982). For amide complexes of metal ions, see: Anget *et al.* (1990); Curtis *et al.* (1983). Pankratov *et al.* (2004); Wayland & Schramm (1969); Rheingold & Staley (1988). For the structures of ambidentate ligand complexes of  $\text{Pd}^{\text{II}}$ , see: Johnson *et al.* (1981); Johansson *et al.* (2001); Langs *et al.* (1967). For the structures of nitrate complexes of  $\text{Pd}^{\text{II}}$ , see: Bennett *et al.* (1967); Adrian *et al.* (2006); Rath *et al.* (1999); Bray *et al.* (2005); Cerdà *et al.* (2006); Gromilov *et al.* (2008); Khranenko *et al.* (2007); Laligant *et al.* (1991). For a discussion on the relationship between bond lengths and ligand donorities, see: Gutmann (1967, 1968); Koshino *et al.* (2005).

**Experimental***Crystal data*

$[\text{Pd}(\text{NO}_3)_2(\text{C}_{10}\text{H}_{17}\text{NO})_2]$   
 $M_r = 564.91$   
Triclinic,  $P\bar{1}$   
 $a = 7.6431$  (5) Å  
 $b = 9.8892$  (8) Å  
 $c = 10.1118$  (7) Å  
 $\alpha = 60.8650$  (19)°  
 $\beta = 66.057$  (2)°  
 $\gamma = 68.845$  (2)°  
 $V = 597.24$  (7) Å<sup>3</sup>  
 $Z = 1$   
Mo  $K\alpha$  radiation  
 $\mu = 0.83$  mm<sup>-1</sup>  
 $T = 173$  K  
 $0.78 \times 0.41 \times 0.07$  mm

*Data collection*

Rigaku R-Axis RAPID diffractometer  
Absorption correction: numerical (ABSCOR; Higashi, 1999)  
 $T_{\text{min}} = 0.754$ ,  $T_{\text{max}} = 0.943$   
5836 measured reflections  
2696 independent reflections  
2662 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.021$

*Refinement*

$R[F^2 > 2\sigma(F^2)] = 0.020$   
 $wR(F^2) = 0.052$   
 $S = 1.07$   
2696 reflections  
152 parameters  
H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.32$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.93$  e Å<sup>-3</sup>

**Table 1**

Selected bond lengths (Å).

Pd(1)—O(1)	2.0092 (11)	Pd(1)—O(2)	2.0112 (15)
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Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 2006); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994) and *DIRDIF99* (Beurskens *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

The authors would like to thank Takeshi Kawasaki for his useful comments.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BR2124).

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**supplementary materials**

*Acta Cryst.* (2009). E65, m1533-m1534 [ doi:10.1107/S1600536809045930 ]

## ***trans*-Bis(1-cyclohexylpyrrolidin-2-one)dinitratopalladium(II)**

**Y. Takahashi and Y. Ikeda**

### **Comment**

Ambidentate ligands are known as ligands with two different coordination sites, such as thiocyanate ion (N and S), cyanate ion (N and O), dimethyl sulfoxide (DMSO, O and S), and *N,N*-dimethylformamide (DMF, N and O) (Fairlie *et al.*, 1985; Rack *et al.*, 2003; Sigel *et al.*, 1982). For amide complexes of metal ions classified as hard Lewis acids, such as  $[M(\text{NH}_3)_5(\text{amide})]^{3+}$  ( $M = \text{Co}, \text{Cr}$ ) (Anget *et al.*, 1990; Curtis *et al.*, 1983), the O-bonded form is thermodynamically and kinetically more favored than the N-bonded form. On the other hand,  $\text{Pd}^{\text{II}}$  classified as a soft Lewis acid usually exhibits a weak affinity to O-donor ligands. Hence, amide compounds should coordinate to  $\text{Pd}^{\text{II}}$  through a nitrogen atom more preferably. In fact, it has been known that the  $\text{Pd}^{\text{II}}$  complex with 2-pyrrolidone is N-bonded form, *i.e.*, *cis*- $\text{PdCl}_2(\text{pyrrolidine-2-ol})_2$  (Pankratov *et al.*, 2004). However,  $\text{Pd}^{\text{II}}$  complexes with O-bonded amides have been also reported, *e.g.*,  $\text{PdCl}_2(L)_2$ ,  $\text{Pd}(L)_4(\text{ClO}_4)_2$  ( $L = \text{DMF}, N,N\text{-dimethylacetamide}, N\text{-methylacetamide}, \text{and } N\text{-methylformamide}$ ) (Wayland *et al.*, 1969), and  $\text{Pd}(\text{DMF})_2(o\text{-}(N\text{-methyliminomethyl)phenyl})\cdot\text{BF}_4$  (Rheingold *et al.*, 1988). In a similar manner to amides,  $\text{Pd}^{\text{II}}$  complexes with S- and O-bonded DMSO have been reported, such as *trans*- $\text{PdCl}_2(\text{DMSO})_2$  with two S-bonded DMSO, and  $\text{Pd}(\text{DMSO})_4(\text{BF}_4)_2\cdot\text{DMSO}$  with two S- and O-bonded DMSO and a solvated DMSO (Johansson *et al.*, 1981; Johansson *et al.*, 2001; Langs *et al.*, 1967). In  $\text{Pd}^{\text{II}}$  nitrate complexes, some crystal structures with S-, P-, N-, or O-donor ligand have been reported, *e.g.*, *cis*- $\text{Pd}(\text{NO}_3)_2(\text{DMSO})_2$ , (Bennett *et al.*, 1967)  $\text{Pd}(\text{NO}_3)_2(\text{dppm})\cdot 3\text{CDCl}_3$  (dppm = bis(diphenylphosphino)methane), (Adrian *et al.*, 2006; Rath *et al.*, 1999)  $\text{enPd}(\text{NO}_3)_2$  (en = ethylenediamine), (Bray *et al.*, 2005; Cerdà *et al.*, 2006) and *trans*- $\text{Pd}(\text{NO}_3)_2(\text{H}_2\text{O})_2$  (Gromilov *et al.*, 2008; Khramenko *et al.*, 2007; Laligant *et al.*, 1991). In all of these complexes, nitrate coordinates to  $\text{Pd}^{\text{II}}$  as the oxygen donor unidentate ligand. So far as we know, *trans*- $\text{Pd}(\text{NO}_3)_2(L)_2$  ( $L$ : oxygen donor unidentate ligand) is only *trans*- $\text{Pd}(\text{NO}_3)_2(\text{H}_2\text{O})_2$  and  $\text{Pd}(\text{NO}_3)_2(\text{O-bonded amide})_2$  has not been reported. We prepared  $\text{Pd}^{\text{II}}$  nitrate complex with the O-bonded amide, *trans*- $\text{Pd}(\text{NO}_3)_2(\text{NCP})_2$  (NCP = *N*-cyclohexyl-2-pyrrolidone), and analyzed its crystal structure using the single-crystal X-ray analytical method. An ORTEP view of *trans*- $\text{Pd}(\text{NO}_3)_2(\text{NCP})_2$  is shown in Fig. 1. In this complex, the configuration around Pd atom is square planar. The nitrate and NCP coordinate to  $\text{Pd}^{\text{II}}$  through their oxygen atoms. The cyclohexyl group of NCP is torsional to pyrrolidone ring. Fig. 2 shows the configuration of coordinated nitrate in *trans*- $\text{Pd}(\text{NO}_3)_2(\text{NCP})_2$ . From this figure, it is found that the nitrate is planar with O—N—O angles close to 120°, and that the distance of Pd···O(3) is longer than Pd···O(2), and almost same as that of *trans*- $\text{Pd}(\text{NO}_3)_2(\text{H}_2\text{O})_2$  (2.926 Å; Khramenko *et al.* (2007)). This reflects the fact that in the *trans*- $\text{Pd}(\text{NO}_3)_2(\text{NCP})_2$  complex nitrate coordinates to  $\text{Pd}^{\text{II}}$  as the unidentate ligand. As mentioned above, the skeletal structure of *trans*- $\text{Pd}(\text{NO}_3)_2(\text{NCP})_2$  is almost same as that of *trans*- $\text{Pd}(\text{NO}_3)_2(\text{H}_2\text{O})_2$ . However, the Pd—O(NO<sub>3</sub>) distance in *trans*- $\text{Pd}(\text{NO}_3)_2(\text{NCP})_2$  is slightly longer than that in *trans*- $\text{Pd}(\text{NO}_3)_2(\text{H}_2\text{O})_2$  (1.999 (5) Å) (Khramenko *et al.* (2007)), and the Pd—O(NCP) distance is 0.02 Å shorter than the Pd—O(water) distance (2.030 (5) Å). The differences in Pd—O( $L$ ) ( $L = \text{H}_2\text{O}$  or NCP) distances are considered to be due to those in electron donicity of  $L$ , that is, the donor number (28.6) of NCP is larger than that (18.0) of water (Gutmann, 1967, Gutmann, 1968, Koshino *et al.*, 2005). Thus, the NCP molecules should more strongly coordinate

## supplementary materials

to the Pd(NO<sub>3</sub>)<sub>2</sub> moiety than water. This may result in a slightly longer distance of Pd—O(NO<sub>3</sub>) in *trans*-Pd(NO<sub>3</sub>)<sub>2</sub>(NCP)<sub>2</sub> than in *trans*-Pd(NO<sub>3</sub>)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>. Infrared spectrum of *trans*-Pd(NO<sub>3</sub>)<sub>2</sub>(NCP)<sub>2</sub> in the solid state was measured as a CaF<sub>2</sub> pellet by Shimadzu FT—IR-8400S spectrophotometer. The carbonyl stretching band of NCP was observed at 1593 cm<sup>-1</sup>, which is lower frequency than that (1670 cm<sup>-1</sup>) of free NCP. The lower shift value ( $\Delta \nu = 77 \text{ cm}^{-1}$ ) is comparable to those (68–107 cm<sup>-1</sup>) for other Pd<sup>II</sup> amide complexes (Wayland *et al.*, 1969). This supports the result of single-crystal analysis that NCP coordinates to the Pd(NO<sub>3</sub>)<sub>2</sub> moiety through carbonyl oxygen atom. <sup>1</sup>H and <sup>13</sup>C NMR spectra of solution prepared by dissolving *trans*-Pd(NO<sub>3</sub>)<sub>2</sub>(NCP)<sub>2</sub> and (CH<sub>3</sub>)<sub>4</sub>Si into CDCl<sub>3</sub> were also measured using Jeol ECX-400 NMR spectrometer (<sup>1</sup>H: 399.8 MHz). The <sup>1</sup>H and <sup>13</sup>C NMR signals corresponding to free NCP were not observed. Most of <sup>1</sup>H and <sup>13</sup>C NMR signals due to coordinated NCP were found to be shifted to lower field compared with those of free NCP. In <sup>1</sup>H NMR spectrum, the signals of methyne (CH) proton in cyclohexyl group and the methylene protons (N—CH<sub>2</sub>) in pyrrolidone ring were observed as a broad multiplet at 3.78 p.p.m. (0.14 p.p.m. high field shift compared with that of free NCP) and triplet at 3.58 and 3.50 p.p.m. (0.02 and 0.10 p.p.m. low field shift compared with those of free NCP), respectively. In the <sup>13</sup>C NMR spectrum, carbonyl carbon and methylene carbon (N—CH<sub>2</sub>) in pyrrolidone ring were observed at 180.49 p.p.m. (6.44 p.p.m. low field shift compared with that of free NCP) and 46.09 p.p.m. (3.22 p.p.m. low field shift compared with that of free NCP). These results suggest that even in CDCl<sub>3</sub> solution two NCP molecules coordinate to Pd<sup>II</sup>. It is worth noting that in spite of the soft Lewis acid all coordination sites of Pd<sup>II</sup> are occupied by oxygen donor ligands. The present result should be first example for the crystal analysis of *trans*-Pd(NO<sub>3</sub>)<sub>2</sub>(L)<sub>2</sub> complex, in which L is the ambidentate ligand with O- and N-bonding sites.

### Experimental

The crystal of *trans*-Pd(NO<sub>3</sub>)<sub>2</sub>(NCP)<sub>2</sub> was prepared by adding Pd(NO<sub>3</sub>)<sub>2</sub>·2H<sub>2</sub>O (0.8218 g, 3.084 mmol, Kojima Chemicals Co., Inc., 38.85wt% in Pd) to CH<sub>2</sub>Cl<sub>2</sub> solution of NCP (1.035 g, 6.186 mmol, Aldrich, 99%). The mixture was refluxed for 30 min with stirring and filtered off any undissolved Pd<sup>II</sup> nitrate. The resulting solution was concentrated to approximately 5 ml, and then diethyl ether was added to form bilayer and to precipitate the complexes. Brown crystals were formed (yield 1.065 g, 59%). Elemental analyses were carried out by LECO CHNS-932 elemental analyzer. Calcd. for H<sub>34</sub>C<sub>20</sub>N<sub>4</sub>O<sub>8</sub>Pd: C, 42.52; H, 6.07; N, 9.92. Found: C, 42.25; H, 5.80; N, 9.88%.

### Refinement

The H atoms of methylene and methyne were placed in calculated positions with C—H = 0.99 and 1.00, respectively. All H atoms were refined as riding on their parent atoms with  $U_{iso}(\text{H}) = 1.2U_{eq}(\text{C})$

### Figures

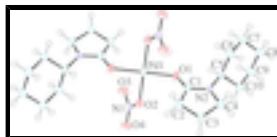


Fig. 1. The ORTEP view of *trans*-Pd(NO<sub>3</sub>)<sub>2</sub>(NCP)<sub>2</sub> complex with the atomic numbering. The thermal ellipsoids are drawn at 50% probability.

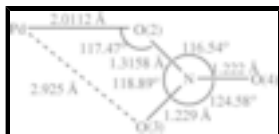


Fig. 2. The configuration of coordinated nitrate in *trans*-Pd(NO<sub>3</sub>)<sub>2</sub>(NCP)<sub>2</sub>.

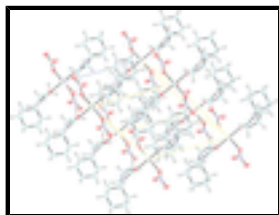


Fig. 3. The packing view of *trans*-Pd(NO<sub>3</sub>)<sub>2</sub>(NCP)<sub>2</sub> complex. The thermal ellipsoids are drawn at 50% probability.

### *trans*-Bis(1-cyclohexylpyrrolidin-2-one)dinitratopalladium(II)

#### Crystal data

[Pd(NO<sub>3</sub>)<sub>2</sub>(C<sub>10</sub>H<sub>17</sub>NO)<sub>2</sub>]

$M_r = 564.91$

Triclinic,  $P\bar{1}$

Hall symbol: -P 1

$a = 7.6431$  (5) Å

$b = 9.8892$  (8) Å

$c = 10.1118$  (7) Å

$\alpha = 60.8650$  (19)°

$\beta = 66.057$  (2)°

$\gamma = 68.845$  (2)°

$V = 597.24$  (7) Å<sup>3</sup>

$Z = 1$

$F_{000} = 292.00$

$D_x = 1.571$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71075$  Å

Cell parameters from 5845 reflections

$\theta = 3.3$ – $27.5$ °

$\mu = 0.83$  mm<sup>-1</sup>

$T = 173$  K

Platelet, brown

$0.78 \times 0.41 \times 0.07$  mm

#### Data collection

Rigaku R-Axis RAPID  
diffractometer

Detector resolution: 10.00 pixels mm<sup>-1</sup>

$T = 173$  K

$\omega$  scans

Absorption correction: numerical  
(ABSCOR; Higashi, 1999)

$T_{\min} = 0.754$ ,  $T_{\max} = 0.943$

5836 measured reflections

2696 independent reflections

2662 reflections with  $F^2 > 2\sigma(F^2)$

$R_{\text{int}} = 0.021$

$\theta_{\text{max}} = 27.5$ °

$h = -9 \rightarrow 9$

$k = -12 \rightarrow 12$

$l = -11 \rightarrow 13$

#### Refinement

Refinement on  $F^2$

$R[F^2 > 2\sigma(F^2)] = 0.020$

$wR(F^2) = 0.052$

$S = 1.07$

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0294P)^2 + 0.1351P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.001$

## supplementary materials

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2696 reflections

$$\Delta\rho_{\max} = 0.32 \text{ e } \text{\AA}^{-3}$$

152 parameters

$$\Delta\rho_{\min} = -0.93 \text{ e } \text{\AA}^{-3}$$

Primary atom site location: structure-invariant direct methods

Extinction correction: none

Secondary atom site location: difference Fourier map

### *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Pd(1)	1.0000	1.0000	0.0000	0.02155 (5)
O(1)	0.92669 (15)	0.93354 (13)	0.23446 (12)	0.0268 (2)
O(2)	1.18410 (17)	1.12278 (13)	-0.03171 (13)	0.0308 (2)
O(3)	0.98979 (19)	1.33981 (14)	-0.14453 (18)	0.0454 (3)
O(4)	1.25913 (19)	1.34980 (16)	-0.12849 (17)	0.0447 (3)
N(1)	1.14168 (19)	1.27768 (16)	-0.10479 (15)	0.0289 (2)
N(2)	0.99744 (17)	0.78446 (13)	0.46810 (13)	0.0201 (2)
C(1)	1.04947 (19)	0.84244 (15)	0.31243 (15)	0.0200 (2)
C(2)	1.2637 (2)	0.78476 (17)	0.24741 (16)	0.0246 (2)
C(3)	1.3405 (2)	0.70029 (19)	0.39282 (17)	0.0283 (3)
C(4)	1.1591 (2)	0.6721 (2)	0.53523 (17)	0.0308 (3)
C(5)	0.79357 (19)	0.80910 (15)	0.56480 (15)	0.0197 (2)
C(6)	0.7016 (2)	0.67022 (18)	0.61976 (19)	0.0284 (3)
C(7)	0.4882 (2)	0.6965 (2)	0.7192 (2)	0.0320 (3)
C(8)	0.4713 (2)	0.7307 (2)	0.85611 (18)	0.0331 (3)
C(9)	0.5632 (2)	0.8697 (2)	0.79808 (19)	0.0329 (3)
C(10)	0.7782 (2)	0.8389 (2)	0.70401 (18)	0.0288 (3)
H(1)	1.4309	0.5987	0.3943	0.034*
H(2)	1.4106	0.7671	0.3930	0.034*
H(3)	0.7185	0.9056	0.4966	0.024*
H(4)	1.1427	0.5617	0.5814	0.037*
H(5)	1.1670	0.6947	0.6176	0.037*
H(6)	0.8353	0.9314	0.6650	0.035*
H(7)	0.8527	0.7458	0.7733	0.035*
H(8)	0.5543	0.8869	0.8894	0.040*
H(9)	0.4907	0.9666	0.7304	0.040*
H(10)	0.3316	0.7544	0.9134	0.040*
H(11)	0.5371	0.6359	0.9307	0.040*
H(12)	0.4091	0.7865	0.6515	0.038*
H(13)	0.4353	0.6011	0.7612	0.038*
H(14)	1.3286	0.8742	0.1665	0.030*
H(15)	1.2847	0.7108	0.2003	0.030*
H(16)	0.7772	0.5718	0.6833	0.034*
H(17)	0.7068	0.6578	0.5268	0.034*

### *Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Pd(1)	0.02369 (9)	0.02268 (9)	0.01332 (8)	-0.00108 (5)	-0.00923 (5)	-0.00290 (6)

O(1)	0.0252 (5)	0.0317 (5)	0.0153 (4)	0.0013 (4)	-0.0097 (3)	-0.0052 (4)
O(2)	0.0337 (5)	0.0282 (5)	0.0285 (5)	-0.0043 (4)	-0.0169 (4)	-0.0047 (4)
O(3)	0.0352 (6)	0.0284 (5)	0.0613 (8)	-0.0068 (4)	-0.0266 (6)	0.0010 (5)
O(4)	0.0404 (7)	0.0437 (7)	0.0463 (7)	-0.0220 (5)	-0.0156 (5)	-0.0031 (6)
N(1)	0.0270 (6)	0.0299 (6)	0.0209 (5)	-0.0104 (4)	-0.0063 (4)	-0.0008 (4)
N(2)	0.0224 (5)	0.0203 (5)	0.0152 (5)	-0.0035 (4)	-0.0084 (4)	-0.0035 (4)
C(1)	0.0242 (6)	0.0195 (5)	0.0162 (5)	-0.0047 (4)	-0.0083 (4)	-0.0049 (4)
C(2)	0.0229 (6)	0.0275 (6)	0.0181 (6)	-0.0022 (5)	-0.0075 (5)	-0.0059 (5)
C(3)	0.0241 (7)	0.0327 (7)	0.0221 (6)	-0.0021 (5)	-0.0110 (5)	-0.0055 (5)
C(4)	0.0262 (7)	0.0367 (7)	0.0188 (6)	-0.0018 (5)	-0.0123 (5)	-0.0017 (5)
C(5)	0.0228 (6)	0.0197 (5)	0.0151 (5)	-0.0050 (4)	-0.0067 (4)	-0.0042 (4)
C(6)	0.0278 (7)	0.0286 (7)	0.0356 (8)	-0.0083 (5)	-0.0092 (5)	-0.0160 (6)
C(7)	0.0262 (7)	0.0341 (7)	0.0388 (8)	-0.0127 (5)	-0.0082 (6)	-0.0132 (6)
C(8)	0.0292 (7)	0.0403 (8)	0.0232 (7)	-0.0150 (6)	-0.0022 (5)	-0.0064 (6)
C(9)	0.0331 (8)	0.0436 (8)	0.0268 (7)	-0.0137 (6)	-0.0004 (6)	-0.0199 (7)
C(10)	0.0311 (7)	0.0397 (8)	0.0242 (7)	-0.0169 (6)	-0.0014 (5)	-0.0174 (6)

*Geometric parameters (Å, °)*

Pd(1)—O(1)	2.0092 (11)	C(9)—C(10)	1.533 (2)
Pd(1)—O(1) <sup>i</sup>	2.0092 (11)	C(2)—H(14)	0.990
Pd(1)—O(2)	2.0112 (15)	C(2)—H(15)	0.990
Pd(1)—O(2) <sup>i</sup>	2.0112 (15)	C(3)—H(1)	0.990
O(1)—C(1)	1.2699 (17)	C(3)—H(2)	0.990
O(2)—N(1)	1.3158 (16)	C(4)—H(4)	0.990
O(3)—N(1)	1.229 (2)	C(4)—H(5)	0.990
O(4)—N(1)	1.222 (2)	C(5)—H(3)	1.000
N(2)—C(1)	1.3200 (17)	C(6)—H(16)	0.990
N(2)—C(4)	1.4754 (19)	C(6)—H(17)	0.990
N(2)—C(5)	1.4713 (15)	C(7)—H(12)	0.990
C(1)—C(2)	1.5020 (17)	C(7)—H(13)	0.990
C(2)—C(3)	1.535 (2)	C(8)—H(10)	0.990
C(3)—C(4)	1.5304 (18)	C(8)—H(11)	0.990
C(5)—C(6)	1.525 (2)	C(9)—H(8)	0.990
C(5)—C(10)	1.525 (2)	C(9)—H(9)	0.990
C(6)—C(7)	1.5351 (19)	C(10)—H(6)	0.990
C(7)—C(8)	1.525 (3)	C(10)—H(7)	0.990
C(8)—C(9)	1.518 (3)		
O(1)—Pd(1)—O(1) <sup>i</sup>	180.00 (7)	C(4)—C(3)—H(2)	110.7
O(1)—Pd(1)—O(2)	89.93 (5)	H(1)—C(3)—H(2)	108.8
O(1)—Pd(1)—O(2) <sup>i</sup>	90.07 (5)	N(2)—C(4)—H(4)	111.1
O(1) <sup>i</sup> —Pd(1)—O(2)	90.07 (5)	N(2)—C(4)—H(5)	111.1
O(1) <sup>i</sup> —Pd(1)—O(2) <sup>i</sup>	89.93 (5)	C(3)—C(4)—H(4)	111.1
O(2)—Pd(1)—O(2) <sup>i</sup>	180.00 (6)	C(3)—C(4)—H(5)	111.1
Pd(1)—O(1)—C(1)	121.33 (8)	H(4)—C(4)—H(5)	109.0
Pd(1)—O(2)—N(1)	117.47 (11)	N(2)—C(5)—H(3)	107.6
O(2)—N(1)—O(3)	118.89 (17)	C(6)—C(5)—H(3)	107.7



## supplementary materials

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O(2)—N(1)—O(4)	116.54 (14)	C(10)—C(5)—H(3)	107.6
O(3)—N(1)—O(4)	124.58 (13)	C(5)—C(6)—H(16)	109.5
C(1)—N(2)—C(4)	112.87 (10)	C(5)—C(6)—H(17)	109.5
C(1)—N(2)—C(5)	123.33 (12)	C(7)—C(6)—H(16)	109.5
C(4)—N(2)—C(5)	123.11 (10)	C(7)—C(6)—H(17)	109.5
O(1)—C(1)—N(2)	121.72 (11)	H(16)—C(6)—H(17)	108.1
O(1)—C(1)—C(2)	127.06 (11)	C(6)—C(7)—H(12)	109.4
N(2)—C(1)—C(2)	111.21 (11)	C(6)—C(7)—H(13)	109.4
C(1)—C(2)—C(3)	103.45 (11)	C(8)—C(7)—H(12)	109.4
C(2)—C(3)—C(4)	105.44 (12)	C(8)—C(7)—H(13)	109.4
N(2)—C(4)—C(3)	103.45 (10)	H(12)—C(7)—H(13)	108.0
N(2)—C(5)—C(6)	110.78 (12)	C(7)—C(8)—H(10)	109.4
N(2)—C(5)—C(10)	111.59 (14)	C(7)—C(8)—H(11)	109.4
C(6)—C(5)—C(10)	111.32 (11)	C(9)—C(8)—H(10)	109.4
C(5)—C(6)—C(7)	110.86 (14)	C(9)—C(8)—H(11)	109.4
C(6)—C(7)—C(8)	111.34 (17)	H(10)—C(8)—H(11)	108.0
C(7)—C(8)—C(9)	111.23 (12)	C(8)—C(9)—H(8)	109.5
C(8)—C(9)—C(10)	110.64 (16)	C(8)—C(9)—H(9)	109.5
C(5)—C(10)—C(9)	109.88 (17)	C(10)—C(9)—H(8)	109.5
C(1)—C(2)—H(14)	111.1	C(10)—C(9)—H(9)	109.5
C(1)—C(2)—H(15)	111.1	H(8)—C(9)—H(9)	108.1
C(3)—C(2)—H(14)	111.1	C(5)—C(10)—H(6)	109.7
C(3)—C(2)—H(15)	111.1	C(5)—C(10)—H(7)	109.7
H(14)—C(2)—H(15)	109.0	C(9)—C(10)—H(6)	109.7
C(2)—C(3)—H(1)	110.7	C(9)—C(10)—H(7)	109.7
C(2)—C(3)—H(2)	110.7	H(6)—C(10)—H(7)	108.2
C(4)—C(3)—H(1)	110.7		
O(1)—Pd(1)—O(2)—N(1)	-113.91 (10)	C(5)—N(2)—C(1)—O(1)	4.9 (2)
O(2)—Pd(1)—O(1)—C(1)	-66.76 (14)	C(5)—N(2)—C(1)—C(2)	-174.43 (15)
O(1)—Pd(1)—O(2) <sup>i</sup> —N(1) <sup>i</sup>	-66.09 (10)	C(4)—N(2)—C(5)—C(6)	-74.5 (2)
O(2) <sup>i</sup> —Pd(1)—O(1)—C(1)	113.24 (14)	C(4)—N(2)—C(5)—C(10)	50.2 (2)
O(1) <sup>i</sup> —Pd(1)—O(2)—N(1)	66.09 (10)	C(5)—N(2)—C(4)—C(3)	-174.89 (17)
O(2)—Pd(1)—O(1) <sup>i</sup> —C(1) <sup>i</sup>	-113.24 (14)	O(1)—C(1)—C(2)—C(3)	172.09 (18)
O(1) <sup>i</sup> —Pd(1)—O(2) <sup>i</sup> —N(1) <sup>i</sup>	113.91 (10)	N(2)—C(1)—C(2)—C(3)	-8.6 (2)
O(2) <sup>i</sup> —Pd(1)—O(1) <sup>i</sup> —C(1) <sup>i</sup>	66.76 (14)	C(1)—C(2)—C(3)—C(4)	16.71 (19)
Pd(1)—O(1)—C(1)—N(2)	-172.54 (13)	C(2)—C(3)—C(4)—N(2)	-18.6 (2)
Pd(1)—O(1)—C(1)—C(2)	6.7 (2)	N(2)—C(5)—C(6)—C(7)	-179.40 (13)
Pd(1)—O(2)—N(1)—O(3)	2.66 (19)	N(2)—C(5)—C(10)—C(9)	178.01 (11)
Pd(1)—O(2)—N(1)—O(4)	-177.21 (12)	C(6)—C(5)—C(10)—C(9)	-57.65 (14)
C(1)—N(2)—C(4)—C(3)	14.4 (2)	C(10)—C(5)—C(6)—C(7)	55.81 (17)
C(4)—N(2)—C(1)—O(1)	175.65 (17)	C(5)—C(6)—C(7)—C(8)	-54.16 (16)
C(4)—N(2)—C(1)—C(2)	-3.7 (2)	C(6)—C(7)—C(8)—C(9)	55.18 (16)
C(1)—N(2)—C(5)—C(6)	95.31 (17)	C(7)—C(8)—C(9)—C(10)	-57.20 (18)
C(1)—N(2)—C(5)—C(10)	-140.05 (16)	C(8)—C(9)—C(10)—C(5)	58.08 (16)

Symmetry codes: (i)  $-x+2, -y+2, -z$ .

Fig. 1

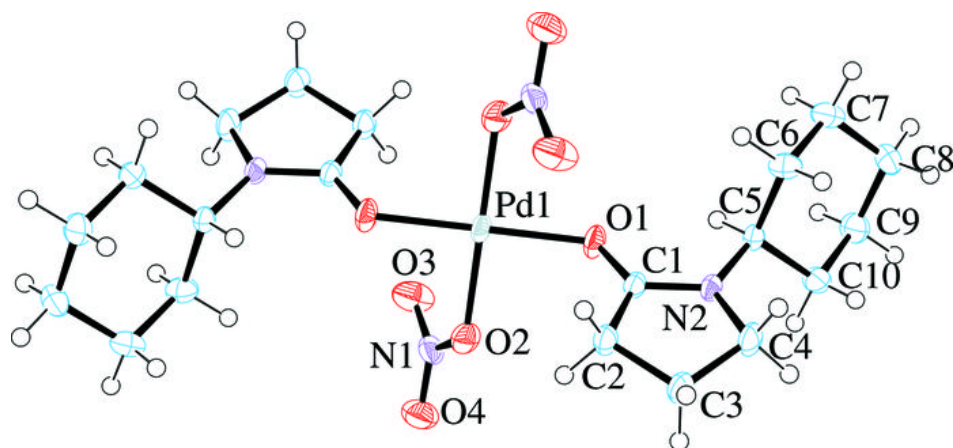


Fig. 2

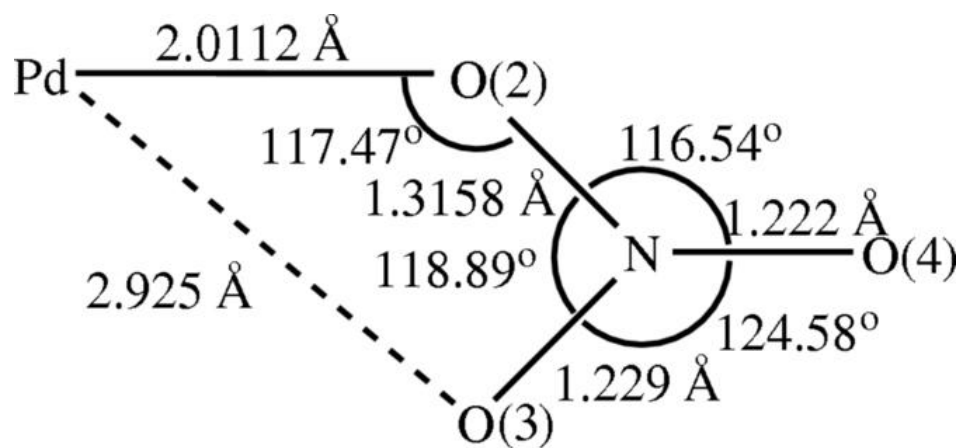


Fig. 3

