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Poly[aqua(μ_4-benzene-1,3-dicarboxylato-κ^4O:O':O'':O''')bis(imidazole-κN)palladium(II)]

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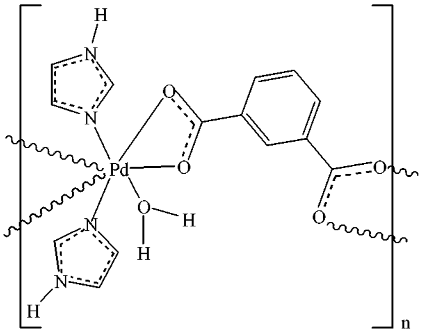
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.031; wR factor = 0.071; data-to-parameter ratio = 14.3.

In the title compound, $[Pd(C_8H_4O_4)(C_3H_4N_2)_2(H_2O)]_n$, two monodendate imidazole ligands are bonded to the Pd^{II} atom, and individual units are linked into chains by 1,3-benzenedicarboxylate anions. The Pd^{II} atom is seven-coordinated by two N atoms from two imidazole ligands, four O atoms from two independent 1,3-benzenedicarboxylate anions and one water molecule, exhibiting a distorted pentagonal–bipyramidal coordination environment. One of the carboxylate O atoms at the base of the pyramid is bonded only very loosely, with a Pd—O distance of 2.771 (2) Å [*cf.* 2.312 (2)–2.488 (3) Å for the other Pd—O distances]. N—H···O and O—H···O hydrogen-bonding interactions link parallel chains together.

Related literature

For related literature, see: Church & Halvorson (1959); Chung *et al.* (1971); Okabe & Oya (2000); Serre *et al.* (2005); Pocker & Fong (1980); Scapin *et al.* (1997).



Experimental

Crystal data

$[Pd(C_8H_4O_4)(C_3H_4N_2)_2(H_2O)]$
M_r = 424.69
Monoclinic, $P2_1/n$
a = 8.5814 (17) Å
b = 19.426 (4) Å
c = 10.118 (2) Å
β = 96.62 (3)°
V = 1675.5 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.14 mm^{-1}
T = 293 (2) K
0.43 × 0.28 × 0.22 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.640, T_{max} = 0.788
8842 measured reflections
3280 independent reflections
2523 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.071
S = 1.00
3280 reflections
229 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.63 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H22···O5^{i}	0.83 (3)	2.02 (3)	2.743 (4)	145 (5)
O1—H23···O2^{ii}	0.83 (4)	1.97 (4)	2.762 (3)	162 (5)
N2—H21···O3^{iii}	0.901 (19)	1.92 (2)	2.801 (4)	165 (4)
N4—H20···O2^{iv}	0.88 (2)	2.14 (2)	3.020 (5)	173 (5)

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2039).

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supplementary materials

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Poly[aqua(μ_4-benzene-1,3-dicarboxylato-κ^4*O*:*O*':*O*'':*O*''')bis(imidazole-κ*N*)palladium(II)]

L.-J. Hao and T.-L. Yu

Comment

In recent years, carboxylic acids have been widely used as polydentate ligands, which can coordinate to transition or rare earth ions yielding complexes with interesting properties that are useful in materials science (Church & Halvorson, 1959; Chung *et al.*, 1971) and in biological systems (Okabe & Oya, 2000; Serre *et al.*, 2005; Pocker & Fong, 1980; Scapin *et al.*, 1997). Herein, we report the synthesis and X-ray crystal structure analysis of the title compound, poly[aqua (benzene-1,3-dicarboxylato) bis(imidazole) palladium(II)].

The molecular structure of the title compound is shown in Fig. 1. Pd(II) is seven-coordianted with two N atoms from two imidazole ligands, four O atoms from two independent 1,3-benzene dicarboxylate, and one water molecule. The palladium ion exhibits a distorted pentagonal bipyramidal coordination environment with one of the imidazole ligands and the water molecule being located in the two apical positions, and the N and O atoms of the remaining imidazole and the carboxylate ions in the basal plane. One of the carboxylate ions is coordinated to the Pd(II) ion in an asymmetric fashion with the Pd(1)—O(3) bond being with 2.771 (2) Å much longer than the other Pd—O bonding distances (2.312 (2) to 2.488 (3) Å). The 1,3-benzene dicarboxylate ions bridge neigboring Pd(II) ion to gives rise to one-dimensional zigzag chains (Fig. 2). N—H···O and O—H···O hydrogen bonding interactions connect the parallel chains with each other stabilize the structure (see the hydrogen-bond geometry table for numerical values and Figure 3 for a packing diagram showing the H-bond interactions).

Experimental

A mixture of palladium acetate (0.5 mmol), imidazole (1.0 mmol), benzene-1,3-dicarboxylic acid (0.5 mmol), H_2O (8 ml) and ethanol (8 ml) in a 25 ml Teflon-lined stainless steel autoclave was kept at 413 K for three days. Colorless crystals were obtained after cooling to room temperature with a yield of 27%. Anal. Calc. for $C_{14}H_{14}N_4O_5Pd$: C 39.56, H 3.30, N 13.19%; Found: C 39.51, H 3.27, N 13.17%.

Refinement

O—H and N—H hydrogen atoms were located in difference density maps and were refined with distance restraints of d(O—H) = 0.82 (2) Å, d(N—H) = 0.88 (2) Å. All other H atoms were placed in calculated positions with a C—H bond distance of 0.93 Å. U_{iso}(H) for the water H atoms was set to $1.5U_{eq}$, all others to $1.2U_{eq}$ of the respective carrier atom.

Figures

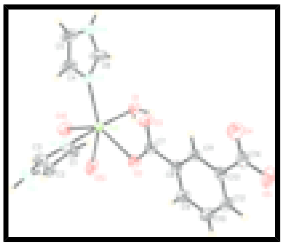

Fig. 1. A view of the structure of (I), showing 30% probability displacement ellipsoids.

Fig. 2. Chains of the title compound along the [010] direction.

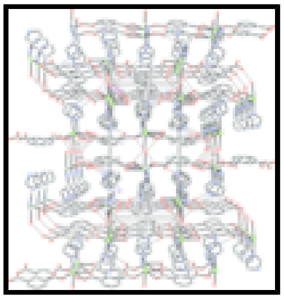

Fig. 3. Packing diagram, viewed along the [010] direction. Dashed lines indicate hydrogen bonds, hydrogen atoms themselves are omitted for clarity.

Poly[aqua(μ_4-benzene-1,3-dicarboxylato-κ^4O:O^{I}:O^{II}:O^{III}) bis(imidazole-κN)palladium(II)]

Crystal data

$[Pd(C_8H_4O_4)(C_3H_4N_2)_2(H_2O)]$

$M_r = 424.69$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 8.5814\ (17)$ Å

$b = 19.426\ (4)$ Å

$c = 10.118\ (2)$ Å

$\beta = 96.62\ (3)$°

$V = 1675.5\ (6)$ Å^3

$Z = 4$

$F_{000} = 848$

$D_x = 1.684$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Cell parameters from 3516 reflections

$\theta = 1.7$–27.0°

$\mu = 1.14$ mm^{-1}

$T = 293\ (2)$ K

Cube, colourless

0.43 × 0.28 × 0.22 mm

Data collection

Bruker APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293(2)$ K

φ and ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2001)

3280 independent reflections

2523 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.031$

$\theta_{\text{max}} = 26.0$°

$\theta_{\text{min}} = 2.3$°

$h = -10 \rightarrow 9$

T_{min} = 0.640, T_{max} = 0.788	$k = -23 \rightarrow 11$
8842 measured reflections	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.031$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.071$	$w = 1/[\sigma^2(F_o^2) + (0.0336P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{max} = 0.003$
3280 reflections	$\Delta\rho_{max} = 0.40$ e Å^{-3}
229 parameters	$\Delta\rho_{min} = -0.62$ e Å^{-3}
4 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Pd1	0.37425 (3)	0.126490 (13)	1.00522 (2)	0.03491 (10)
C1	0.4890 (4)	0.2744 (2)	0.8764 (4)	0.0612 (10)
H1	0.4473	0.2630	0.7902	0.073*
C2	0.5780 (5)	0.3305 (2)	0.9103 (5)	0.0653 (11)
H2	0.6097	0.3638	0.8530	0.078*
C3	0.5445 (4)	0.2718 (2)	1.0866 (4)	0.0641 (11)
H3	0.5504	0.2588	1.1755	0.077*
C4	0.0640 (5)	0.2230 (2)	0.9641 (5)	0.0741 (13)
H4	0.1256	0.2624	0.9630	0.089*
C5	−0.0908 (5)	0.2224 (3)	0.9456 (6)	0.1025 (19)
H5	−0.1565	0.2601	0.9272	0.123*
C6	−0.0034 (5)	0.1199 (2)	0.9826 (6)	0.0913 (17)
H6	−0.0009	0.0726	0.9959	0.110*
C7	0.4815 (4)	0.09640 (18)	0.7491 (3)	0.0413 (8)

C8	0.5730 (3)	0.08147 (17)	0.6351 (3)	0.0368 (7)
C9	0.7237 (4)	0.0550 (2)	0.6573 (3)	0.0533 (10)
H9	0.7684	0.0460	0.7438	0.064*
C10	0.8084 (4)	0.0420 (2)	0.5512 (4)	0.0663 (12)
H10	0.9091	0.0238	0.5665	0.080*
C11	0.5089 (4)	0.09436 (17)	0.5050 (3)	0.0388 (7)
H11	0.4072	0.1114	0.4889	0.047*
C12	0.7429 (4)	0.0560 (2)	0.4226 (3)	0.0555 (10)
H12	0.8002	0.0476	0.3516	0.067*
C13	0.5931 (4)	0.08233 (17)	0.3994 (3)	0.0397 (8)
C14	0.5215 (5)	0.09758 (19)	0.2596 (3)	0.0505 (9)
N1	0.4694 (3)	0.23673 (16)	0.9882 (3)	0.0532 (8)
N2	0.6116 (4)	0.32854 (18)	1.0435 (4)	0.0638 (9)
H21	0.682 (4)	0.3538 (18)	1.096 (3)	0.077*
N3	0.1206 (3)	0.15876 (16)	0.9847 (3)	0.0482 (7)
N4	−0.1346 (4)	0.1571 (3)	0.9587 (6)	0.1079 (16)
H20	−0.229 (3)	0.140 (3)	0.935 (6)	0.129*
O1	0.2779 (3)	0.01164 (13)	0.9913 (2)	0.0496 (6)
O2	0.5538 (3)	0.09052 (13)	0.8661 (2)	0.0489 (6)
O3	0.3421 (3)	0.11470 (14)	0.7305 (2)	0.0593 (7)
O4	0.3885 (3)	0.12419 (16)	0.2416 (2)	0.0722 (8)
O5	0.5971 (4)	0.08483 (16)	0.1641 (2)	0.0753 (9)
H22	0.296 (6)	−0.006 (2)	0.920 (3)	0.113*
H23	0.327 (5)	−0.013 (2)	1.048 (4)	0.113*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd1	0.02806 (14)	0.04265 (16)	0.03440 (14)	0.00380 (12)	0.00523 (9)	0.00091 (12)
C1	0.055 (2)	0.061 (3)	0.065 (3)	−0.003 (2)	−0.0070 (19)	0.006 (2)
C2	0.054 (2)	0.062 (3)	0.079 (3)	−0.008 (2)	0.004 (2)	0.011 (2)
C3	0.059 (2)	0.074 (3)	0.061 (2)	−0.016 (2)	0.012 (2)	−0.013 (2)
C4	0.046 (2)	0.051 (3)	0.126 (4)	0.010 (2)	0.011 (2)	−0.004 (3)
C5	0.046 (3)	0.066 (3)	0.193 (6)	0.021 (2)	0.003 (3)	−0.021 (4)
C6	0.034 (2)	0.057 (3)	0.181 (5)	0.002 (2)	0.008 (3)	−0.004 (3)
C7	0.044 (2)	0.047 (2)	0.0327 (17)	0.0011 (17)	0.0051 (14)	0.0022 (15)
C8	0.0366 (17)	0.0414 (19)	0.0331 (16)	−0.0007 (15)	0.0074 (13)	0.0008 (14)
C9	0.042 (2)	0.080 (3)	0.0361 (18)	0.010 (2)	−0.0005 (15)	0.0035 (18)
C10	0.044 (2)	0.109 (4)	0.048 (2)	0.018 (2)	0.0117 (17)	0.000 (2)
C11	0.0363 (17)	0.0436 (19)	0.0364 (17)	0.0022 (16)	0.0031 (14)	0.0011 (15)
C12	0.051 (2)	0.080 (3)	0.0386 (19)	0.004 (2)	0.0174 (16)	−0.0040 (19)
C13	0.0437 (19)	0.0415 (19)	0.0339 (17)	−0.0019 (16)	0.0050 (14)	−0.0011 (15)
C14	0.069 (3)	0.048 (2)	0.0343 (19)	−0.003 (2)	0.0053 (18)	−0.0017 (16)
N1	0.0498 (18)	0.0557 (19)	0.0534 (18)	−0.0094 (16)	0.0024 (14)	−0.0037 (16)
N2	0.0489 (19)	0.061 (2)	0.082 (3)	−0.0150 (17)	0.0069 (17)	−0.0165 (19)
N3	0.0309 (15)	0.0496 (18)	0.064 (2)	0.0047 (14)	0.0043 (13)	−0.0022 (15)
N4	0.036 (2)	0.076 (3)	0.210 (5)	0.001 (2)	0.011 (3)	−0.020 (3)
O1	0.0518 (15)	0.0479 (15)	0.0500 (15)	0.0053 (12)	0.0090 (12)	0.0011 (12)

O2	0.0502 (14)	0.0645 (16)	0.0324 (12)	0.0089 (13)	0.0065 (10)	0.0026 (11)
O3	0.0444 (14)	0.089 (2)	0.0459 (14)	0.0201 (14)	0.0101 (11)	0.0048 (13)
O4	0.0713 (19)	0.103 (2)	0.0404 (14)	0.0198 (18)	−0.0011 (13)	0.0079 (14)
O5	0.109 (2)	0.086 (2)	0.0328 (13)	0.0261 (19)	0.0157 (14)	−0.0034 (14)

Geometric parameters (Å, °)

Pd1—N3	2.252 (3)	C7—O3	1.242 (4)
Pd1—N1	2.305 (3)	C7—O2	1.276 (4)
Pd1—O2	2.311 (2)	C7—C8	1.497 (4)
Pd1—O1	2.378 (3)	C8—C9	1.386 (4)
Pd1—O4^{i}	2.382 (2)	C8—C11	1.390 (4)
Pd1—O5^{i}	2.487 (3)	C9—C10	1.387 (4)
C1—C2	1.353 (5)	C9—H9	0.9300
C1—N1	1.374 (5)	C10—C12	1.383 (5)
C1—H1	0.9300	C10—H10	0.9300
C2—N2	1.346 (5)	C11—C13	1.377 (4)
C2—H2	0.9300	C11—H11	0.9300
C3—N1	1.312 (4)	C12—C13	1.379 (4)
C3—N2	1.340 (5)	C12—H12	0.9300
C3—H3	0.9300	C13—C14	1.505 (4)
C4—C5	1.321 (6)	C14—O4	1.247 (4)
C4—N3	1.347 (5)	C14—O5	1.249 (4)
C4—H4	0.9300	N2—H21	0.901 (19)
C5—N4	1.335 (7)	N4—H20	0.88 (2)
C5—H5	0.9300	O1—H22	0.83 (3)
C6—N3	1.302 (5)	O1—H23	0.83 (4)
C6—N4	1.336 (5)	O4—Pd1^{ii}	2.382 (2)
C6—H6	0.9300	O5—Pd1^{ii}	2.487 (3)
N3—Pd1—N1	94.67 (11)	C11—C8—C7	120.6 (3)
N3—Pd1—O2	137.15 (9)	C8—C9—C10	120.3 (3)
N1—Pd1—O2	88.21 (10)	C8—C9—H9	119.9
N3—Pd1—O1	85.99 (10)	C10—C9—H9	119.9
N1—Pd1—O1	172.26 (9)	C12—C10—C9	120.0 (3)
O2—Pd1—O1	86.09 (9)	C12—C10—H10	120.0
N3—Pd1—O4^{i}	92.00 (10)	C9—C10—H10	120.0
N1—Pd1—O4^{i}	96.59 (11)	C13—C11—C8	121.2 (3)
O2—Pd1—O4^{i}	130.19 (9)	C13—C11—H11	119.4
O1—Pd1—O4^{i}	91.10 (9)	C8—C11—H11	119.4
N3—Pd1—O5^{i}	144.39 (9)	C13—C12—C10	120.2 (3)
N1—Pd1—O5^{i}	95.63 (11)	C13—C12—H12	119.9
O2—Pd1—O5^{i}	77.22 (8)	C10—C12—H12	119.9
O1—Pd1—O5^{i}	88.23 (10)	C11—C13—C12	119.6 (3)
O4^{i}—Pd1—O5^{i}	52.98 (9)	C11—C13—C14	120.1 (3)
C2—C1—N1	109.8 (4)	C12—C13—C14	120.3 (3)
C2—C1—H1	125.1	O4—C14—O5	121.1 (3)

N1—C1—H1	125.1	O4—C14—C13	119.1 (3)
N2—C2—C1	106.3 (4)	O5—C14—C13	119.7 (4)
N2—C2—H2	126.8	C3—N1—C1	104.6 (3)
C1—C2—H2	126.8	C3—N1—Pd1	124.9 (3)
N1—C3—N2	111.8 (4)	C1—N1—Pd1	129.3 (3)
N1—C3—H3	124.1	C3—N2—C2	107.4 (3)
N2—C3—H3	124.1	C3—N2—H21	123 (3)
C5—C4—N3	110.6 (4)	C2—N2—H21	129 (3)
C5—C4—H4	124.7	C6—N3—C4	104.8 (3)
N3—C4—H4	124.7	C6—N3—Pd1	128.3 (3)
C4—C5—N4	106.5 (4)	C4—N3—Pd1	126.8 (3)
C4—C5—H5	126.7	C5—N4—C6	106.9 (4)
N4—C5—H5	126.7	C5—N4—H20	126 (4)
N3—C6—N4	111.1 (4)	C6—N4—H20	125 (4)
N3—C6—H6	124.4	Pd1—O1—H22	110 (4)
N4—C6—H6	124.4	Pd1—O1—H23	111 (4)
O3—C7—O2	121.5 (3)	H22—O1—H23	103 (5)
O3—C7—C8	121.4 (3)	C7—O2—Pd1	104.5 (2)
O2—C7—C8	117.1 (3)	C14—O4—Pd1$^{\text{ii}}$	95.4 (2)
C9—C8—C11	118.7 (3)	C14—O5—Pd1$^{\text{ii}}$	90.4 (2)
C9—C8—C7	120.6 (3)		

Symmetry codes: (i) *x*, *y*, *z*+1; (ii) *x*, *y*, *z*−1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H22···O5$^{\text{iii}}$	0.83 (3)	2.02 (3)	2.743 (4)	145 (5)
O1—H23···O2$^{\text{iv}}$	0.83 (4)	1.97 (4)	2.762 (3)	162 (5)
N2—H21···O3$^{\text{v}}$	0.901 (19)	1.92 (2)	2.801 (4)	165 (4)
N4—H20···O2$^{\text{vi}}$	0.88 (2)	2.14 (2)	3.020 (5)	173 (5)

Symmetry codes: (iii) −*x*+1, −*y*, −*z*+1; (iv) −*x*+1, −*y*, −*z*+2; (v) *x*+1/2, −*y*+1/2, *z*+1/2; (vi) *x*−1, *y*, *z*.

Fig. 1

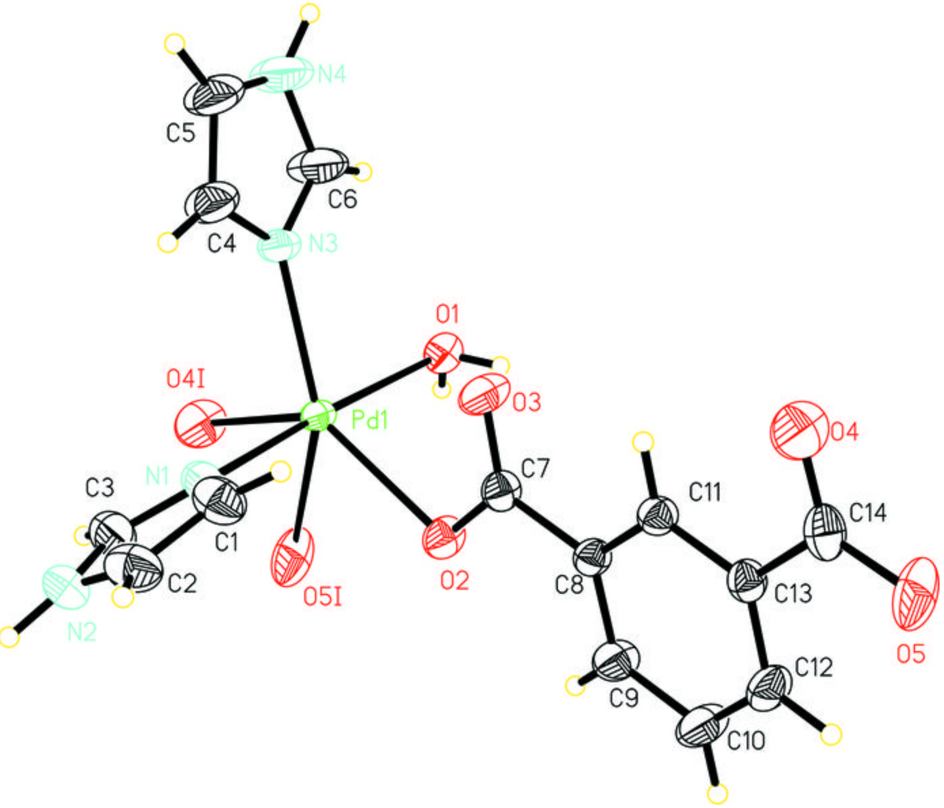
N4
C5
C6
C4
N3
O1
O4I
Pd1
O3
O4
N1
C3
C7
C11
C14
C1
O5I
O2
C2
C8
C13
N2
O5
C12
C9
C10

Fig. 2

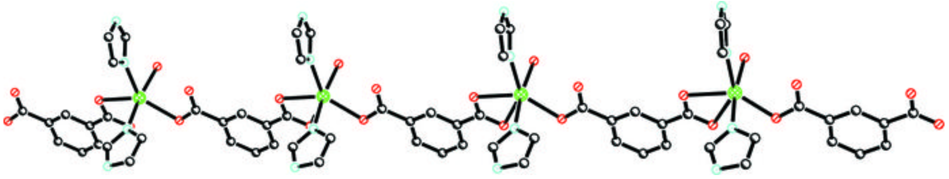

Fig. 3