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(*SP*-4-2)-(4,4′-Di-*tert*-butyl-2,2′-bipyridine-κ^2N,N')diiododipalladium(II)

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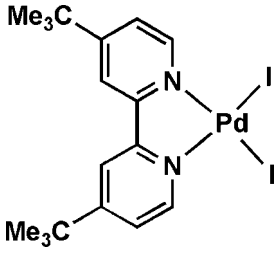
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.008 Å; R factor = 0.043; wR factor = 0.121; data-to-parameter ratio = 24.0.

In the title compound, $[PdI_2(C_{18}H_{24}N_2)]$, the coordination at the Pd atom is distorted square planar; the ligand bite is 79.19 (17)°. The compound is isotypic with the dichlorido analogue. The Pd—N bond lengths of 2.047 (4) and 2.062 (4)Å are *ca* 0.03 Å longer than those of the chloride derivative.

Related literature

For related literature, see: MacLean *et al.* (2002); Qin *et al.* (2002).



Experimental

Crystal data

$[PdI_2(C_{18}H_{24}N_2)]$
M_r = 628.59
Monoclinic, $P2_1/c$
a = 7.7201 (6) Å
b = 19.4695 (16) Å
c = 13.4120 (11) Å
β = 100.290 (4)°
V = 1983.5 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 4.05 mm^{-1}
T = 100 (2) K
0.15 × 0.07 × 0.07 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.643, T_{max} = 0.765
65072 measured reflections
5132 independent reflections
4403 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.121
S = 1.07
5132 reflections
214 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 2.95 e Å^{-3}
$\Delta\rho_{min}$ = −2.64 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Pd—N1	2.047 (4)	Pd—I2	2.5403 (6)
Pd—N11	2.062 (4)	Pd—I1	2.5596 (6)
N1—Pd—N11	79.19 (17)	N1—Pd—I1	96.79 (12)
N1—Pd—I2	174.30 (13)	N11—Pd—I1	171.98 (13)
N11—Pd—I2	98.14 (12)	I2—Pd—I1	86.459 (19)

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL97*.

We thank Professor J. Vicente, University of Murcia, for his continuing support and encouragement.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2547).

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supplementary materials

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(*SP*-4-2)-(4,4'-Di-*tert*-butyl-2,2'-bipyridine-κ^2N,N')diiodidopalladium(II)

P. G. Jones, M. J. Fernández-Rodríguez and A. J. Martínez-Martínez

Comment

Crystals of the title compound were unintentionally obtained by the liquid diffusion method from solutions of [C_6{PdI(^{t}Bubpy)}$_3$-1,3,5-(CH_2OH)3–2,4,6] in $CDCl_3$ layered with n-hexane (this sample was used for structure determination) or [PdI{$C(O)C_6H_4${NHC(Me)═CHC(O)Me}-2}(^{t}Bubpy)] in CH_2Cl_2 layered with diethyl ether (cell determination only).

The molecular structure is shown in Fig. 1. The coordination at palladium is square planar (Table 1), but slightly distorted by the narrow bite of the chelating ligand. The least-squares plane though Pd and the four donor atoms has an r.m.s. deviation of 0.102 Å, whereby the donor atoms deviate from the plane alternately by *ca* ±0.1 Å. The planes of the bipyridine ligand subtend an interplanar angle of 9.8 (2)°.

The crystal structure of the homologous chloro complex [$PdCl_2$(^{t}Bubpy)] has been determined twice (Qin *et al.*, 2002; MacLean *et al.*, 2002). The chloro and iodo complexes appear to be isotypic, although the beta angle of the chloro (96.6°) is significantly narrower than that of the iodo complex. The Pd—N bonds in the diiodo complex [2.047 (4), 2.062 (4) Å] are slightly longer than in the dichloro complex [2.028 (6), 2.029 (6) or 2.015 (3), 2.022 (3) Å], reflecting the greater *trans* influence of iodo ligands.

Experimental

The pure compound was prepared in 87% yield from $PdCl_2$, ^{t}Bubpy and NaI (1:1:4, in acetone, 2 h at room temperature). The complex was extracted into dichloromethane and precipitated with diethyl ether. *M*.p. > 320 °C. ^{1}H-NMR: (600 MHz, $CDCl_3$): δ 1.45 (s, 18 H, ^{t}Bu), 7.50 (dd, 2 H, H5, $^3J_{HH}$ = 6 Hz, $^4J_{HH}$ = 2 Hz), 7.95 (d, 2 H, H3, $^4J_{HH}$ = 2 Hz), 9.81 (d, 2H, H6, $^3J_{HH}$ = 6 Hz). ^{13}C{^{1}H-NMR (151 MHz, $CDCl_3$): δ 30.5 (Me), 35.9 (*C*Me_3), 119.5 (CH_3), 124.6 (C5), 153.8 (C6), 156.5 (C2), 164.2 (C4). Analysis: calcd for $C_{18}H_{24}I_2N_2Pd$: C, 34.39; H, 3.85; N, 4.46; Found: C, 34.50; H, 3.77; N, 4.62%.

Refinement

Methyl hydrogen atoms were located in a difference synthesis; the methyl groups were idealized and refined as rigid groups allowed to rotate but not tip, with C—H 0.98 Å, H—C—H 109.5°. Other hydrogen atoms were included using a riding model with C—H 0.95 Å; U(H) values were fixed at $n \times U_{eq}$(C) of the parent C atom, with n = 1.5 for methyl and 1.2 for other H atoms.

The two largest difference peaks lie 0.4 Å from the Pd atom.

Figures

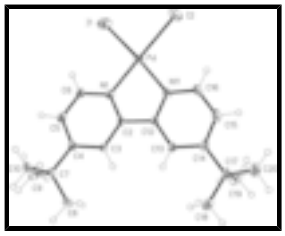


Fig. 1. The formula unit of the title compound in the crystal. Ellipsoids represent 50% probability levels.

(SP-4–2)-(4,4'-Di-*tert*-butyl-2,2'-bipyridine- κ*N*,κ*N*')diiodidopalladium(II)

Crystal data

$[PdI_2(C_{18}H_{24}N_2)]$	F_{000} = 1192
M_r = 628.59	D_x = 2.105 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 7.7201 (6) Å	Cell parameters from 9885 reflections
b = 19.4695 (16) Å	θ = 2.6–28.8º
c = 13.4120 (11) Å	μ = 4.05 mm^{-1}
β = 100.290 (4)º	T = 100 (2) K
V = 1983.5 (3) Å^3	Prism, brown
Z = 4	0.15 × 0.07 × 0.07 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	5132 independent reflections
Radiation source: fine-focus sealed tube	4403 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.038
T = 100(2) K	θ_{max} = 28.7º
φ and ω scans	θ_{min} = 1.9º
Absorption correction: multi-scan (SADABS; Bruker, 1998)	h = −10→10
T_{min} = 0.643, T_{max} = 0.765	k = −26→26
65072 measured reflections	l = −18→18

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.043	H-atom parameters constrained
$wR(F^2)$ = 0.121	$w = 1/[\sigma^2(F_o^2) + (0.0506P)^2 + 20.9904P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.07	$(\Delta/\sigma)_{max}$ = 0.001

5132 reflections	$\Delta\rho_{max}$ = 2.95 e Å^{-3}
214 parameters	$\Delta\rho_{min}$ = −2.64 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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.

Least-squares planes (*x*,*y*,*z* in crystal coordinates) and deviations from them (* indicates atom used to define plane)

6.7811 (0.0033) *x* + 5.5841 (0.0167) *y* + 2.9416 (0.0118) *z* = 6.0043 (0.0102)

* 0.0153 (0.0014) Pd * 0.1254 (0.0021) N1 * −0.1325 (0.0021) N11 * −0.1004 (0.0016) I1 * 0.0922 (0.0016) I2

Rms deviation of fitted atoms = 0.1021

7.4041 (0.0047) *x* + 2.7758 (0.0447) *y* + 0.9312 (0.0274) *z* = 3.5963 (0.0293)

Angle to previous plane (with approximate e.s.d.) = 12.35 (0.16)

* 0.0039 (0.0036) N1 * 0.0070 (0.0036) C2 * −0.0129 (0.0037) C3 * 0.0083 (0.0037) C4 * 0.0022 (0.0040) C5 * −0.0085 (0.0041) C6

Rms deviation of fitted atoms = 0.0079

7.0350 (0.0076) *x* + 1.8324 (0.0427) *y* + 3.1076 (0.0311) *z* = 4.2574 (0.0208)

Angle to previous plane (with approximate e.s.d.) = 9.78 (1/5)

* −0.0132 (0.0035) N11 * 0.0068 (0.0036) C12 * 0.0052 (0.0038) C13 * −0.0110 (0.0040) C14 * 0.0050 (0.0043) C15 * 0.0071 (0.0042) C16

Rms deviation of fitted atoms = 0.0086

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}
Pd	0.24845 (5)	0.57498 (2)	0.38217 (3)	0.01519 (10)
I1	0.14298 (7)	0.69901 (2)	0.35049 (3)	0.03758 (14)
I2	0.33171 (6)	0.57969 (2)	0.20742 (3)	0.03264 (13)
N1	0.2067 (6)	0.5684 (2)	0.5283 (3)	0.0151 (8)
C2	0.2257 (7)	0.5047 (2)	0.5707 (3)	0.0124 (9)
C3	0.2144 (7)	0.4936 (2)	0.6717 (4)	0.0135 (9)

H3	0.2243	0.4483	0.6983	0.016*
C4	0.1887 (7)	0.5487 (3)	0.7348 (4)	0.0145 (9)
C5	0.1693 (8)	0.6136 (3)	0.6890 (4)	0.0197 (11)
H5	0.1502	0.6527	0.7281	0.024*
C6	0.1777 (8)	0.6212 (3)	0.5882 (4)	0.0201 (11)
H6	0.1623	0.6658	0.5593	0.024*
C7	0.1949 (7)	0.5410 (3)	0.8480 (4)	0.0168 (10)
C8	0.1975 (8)	0.4657 (3)	0.8812 (4)	0.0202 (11)
H8A	0.0926	0.4424	0.8448	0.030*
H8B	0.1984	0.4634	0.9542	0.030*
H8C	0.3033	0.4433	0.8657	0.030*
C9	0.0338 (9)	0.5766 (3)	0.8799 (5)	0.0264 (12)
H9A	0.0284	0.6246	0.8573	0.040*
H9B	0.0453	0.5751	0.9538	0.040*
H9C	−0.0741	0.5528	0.8488	0.040*
C10	0.3628 (9)	0.5758 (3)	0.9012 (4)	0.0250 (12)
H10A	0.4645	0.5555	0.8777	0.037*
H10B	0.3749	0.5694	0.9746	0.037*
H10C	0.3572	0.6250	0.8855	0.037*
N11	0.2985 (6)	0.4722 (2)	0.4115 (3)	0.0157 (8)
C12	0.2671 (7)	0.4498 (3)	0.5022 (3)	0.0134 (9)
C13	0.2737 (7)	0.3806 (3)	0.5276 (4)	0.0173 (10)
H13	0.2504	0.3667	0.5918	0.021*
C14	0.3140 (8)	0.3312 (3)	0.4602 (4)	0.0197 (11)
C15	0.3502 (8)	0.3563 (3)	0.3687 (4)	0.0254 (12)
H15	0.3811	0.3250	0.3204	0.030*
C16	0.3421 (8)	0.4250 (3)	0.3473 (4)	0.0219 (11)
H16	0.3683	0.4400	0.2843	0.026*
C17	0.3111 (9)	0.2538 (3)	0.4818 (5)	0.0272 (13)
C18	0.3405 (9)	0.2390 (3)	0.5955 (5)	0.0285 (13)
H18A	0.4543	0.2580	0.6279	0.043*
H18B	0.3398	0.1893	0.6066	0.043*
H18C	0.2462	0.2604	0.6249	0.043*
C19	0.1298 (11)	0.2262 (3)	0.4317 (6)	0.0421 (19)
H19A	0.0383	0.2470	0.4640	0.063*
H19B	0.1274	0.1762	0.4397	0.063*
H19C	0.1081	0.2377	0.3594	0.063*
C20	0.4554 (11)	0.2171 (4)	0.4360 (5)	0.0376 (17)
H20A	0.4321	0.2230	0.3622	0.056*
H20B	0.4555	0.1681	0.4524	0.056*
H20C	0.5704	0.2370	0.4641	0.056*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd	0.0179 (2)	0.01681 (19)	0.00966 (17)	−0.00128 (15)	−0.00068 (13)	0.00382 (13)
I1	0.0497 (3)	0.0287 (2)	0.0323 (2)	0.00421 (19)	0.00187 (19)	0.01204 (17)
I2	0.0357 (2)	0.0439 (3)	0.02018 (19)	−0.00452 (19)	0.00987 (16)	0.00242 (16)

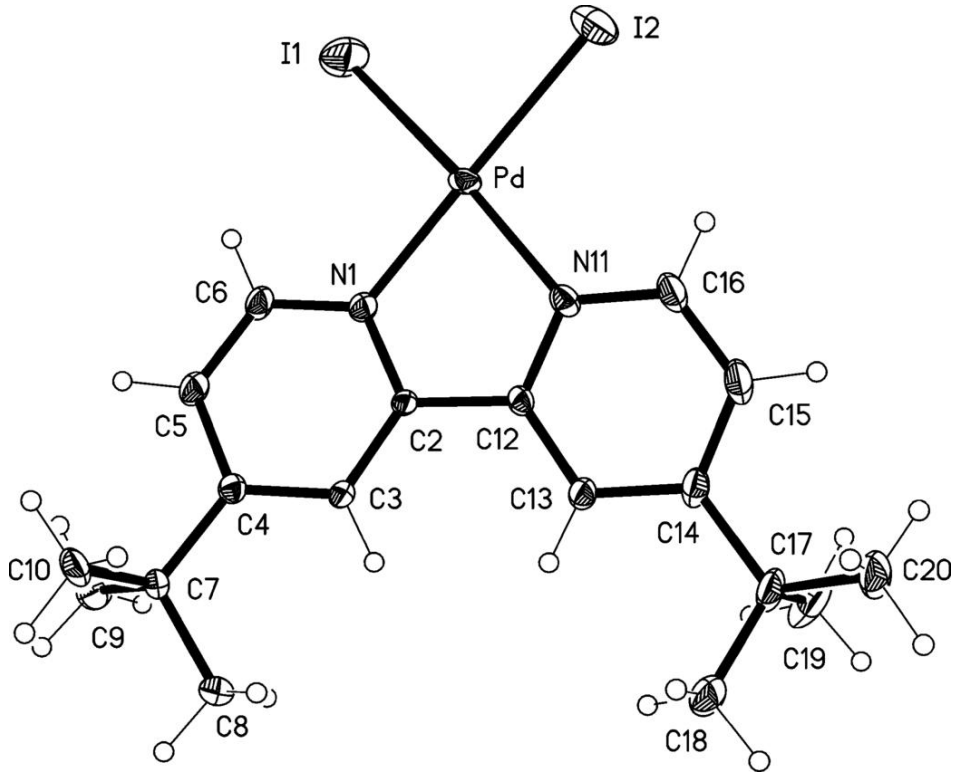
N1	0.019 (2)	0.012 (2)	0.0141 (19)	0.0023 (16)	0.0016 (16)	0.0007 (15)
C2	0.016 (2)	0.010 (2)	0.010 (2)	−0.0001 (17)	−0.0005 (17)	0.0007 (16)
C3	0.018 (2)	0.010 (2)	0.012 (2)	−0.0015 (18)	0.0014 (17)	−0.0005 (17)
C4	0.016 (2)	0.013 (2)	0.013 (2)	−0.0011 (19)	0.0001 (17)	−0.0007 (18)
C5	0.027 (3)	0.012 (2)	0.019 (2)	0.004 (2)	0.002 (2)	−0.0009 (19)
C6	0.032 (3)	0.010 (2)	0.016 (2)	0.005 (2)	−0.002 (2)	0.0000 (18)
C7	0.025 (3)	0.014 (2)	0.012 (2)	−0.004 (2)	0.0046 (19)	−0.0028 (17)
C8	0.027 (3)	0.018 (3)	0.015 (2)	−0.003 (2)	0.006 (2)	0.0025 (19)
C9	0.033 (3)	0.023 (3)	0.028 (3)	0.003 (2)	0.017 (2)	−0.003 (2)
C10	0.035 (3)	0.028 (3)	0.011 (2)	−0.012 (3)	0.002 (2)	−0.005 (2)
N11	0.017 (2)	0.019 (2)	0.0108 (18)	0.0027 (17)	0.0013 (15)	0.0001 (16)
C12	0.016 (2)	0.014 (2)	0.009 (2)	0.0004 (18)	−0.0004 (17)	−0.0013 (17)
C13	0.025 (3)	0.013 (2)	0.013 (2)	0.004 (2)	−0.0004 (19)	−0.0007 (18)
C14	0.025 (3)	0.016 (2)	0.015 (2)	0.008 (2)	−0.004 (2)	−0.0040 (19)
C15	0.035 (3)	0.025 (3)	0.015 (2)	0.011 (2)	0.002 (2)	−0.006 (2)
C16	0.028 (3)	0.025 (3)	0.013 (2)	0.004 (2)	0.005 (2)	−0.004 (2)
C17	0.042 (4)	0.015 (3)	0.022 (3)	0.012 (2)	−0.002 (2)	−0.004 (2)
C18	0.041 (4)	0.018 (3)	0.025 (3)	0.011 (3)	0.001 (2)	0.001 (2)
C19	0.058 (5)	0.015 (3)	0.044 (4)	0.002 (3)	−0.016 (3)	−0.006 (3)
C20	0.064 (5)	0.026 (3)	0.021 (3)	0.026 (3)	0.005 (3)	−0.001 (2)

Geometric parameters (Å, °)

Pd—N1	2.047 (4)	C3—H3	0.9500
Pd—N11	2.062 (4)	C5—H5	0.9500
Pd—I2	2.5403 (6)	C6—H6	0.9500
Pd—I1	2.5596 (6)	C8—H8A	0.9800
N1—C6	1.348 (7)	C8—H8B	0.9800
N1—C2	1.361 (6)	C8—H8C	0.9800
C2—C3	1.389 (7)	C9—H9A	0.9800
C2—C12	1.481 (7)	C9—H9B	0.9800
C3—C4	1.402 (7)	C9—H9C	0.9800
C4—C5	1.402 (7)	C10—H10A	0.9800
C4—C7	1.519 (7)	C10—H10B	0.9800
C5—C6	1.373 (7)	C10—H10C	0.9800
C7—C10	1.523 (8)	C13—H13	0.9500
C7—C8	1.530 (7)	C15—H15	0.9500
C7—C9	1.549 (8)	C16—H16	0.9500
N11—C16	1.343 (7)	C18—H18A	0.9800
N11—C12	1.354 (6)	C18—H18B	0.9800
C12—C13	1.389 (7)	C18—H18C	0.9800
C13—C14	1.392 (7)	C19—H19A	0.9800
C14—C15	1.394 (8)	C19—H19B	0.9800
C14—C17	1.536 (8)	C19—H19C	0.9800
C15—C16	1.368 (8)	C20—H20A	0.9800
C17—C18	1.529 (8)	C20—H20B	0.9800
C17—C19	1.538 (10)	C20—H20C	0.9800
C17—C20	1.540 (9)		
N1—Pd—N11	79.19 (17)	C4—C5—H5	119.7

N1—Pd—I2	174.30 (13)	N1—C6—H6	118.4
N11—Pd—I2	98.14 (12)	C5—C6—H6	118.4
N1—Pd—I1	96.79 (12)	C7—C8—H8A	109.5
N11—Pd—I1	171.98 (13)	C7—C8—H8B	109.5
I2—Pd—I1	86.459 (19)	H8A—C8—H8B	109.5
C6—N1—C2	117.6 (4)	C7—C8—H8C	109.5
C6—N1—Pd	126.5 (4)	H8A—C8—H8C	109.5
C2—N1—Pd	115.7 (3)	H8B—C8—H8C	109.5
N1—C2—C3	121.9 (4)	C7—C9—H9A	109.5
N1—C2—C12	114.6 (4)	C7—C9—H9B	109.5
C3—C2—C12	123.4 (4)	H9A—C9—H9B	109.5
C2—C3—C4	120.6 (5)	C7—C9—H9C	109.5
C3—C4—C5	116.2 (5)	H9A—C9—H9C	109.5
C3—C4—C7	123.1 (5)	H9B—C9—H9C	109.5
C5—C4—C7	120.6 (5)	C7—C10—H10A	109.5
C6—C5—C4	120.6 (5)	C7—C10—H10B	109.5
N1—C6—C5	123.1 (5)	H10A—C10—H10B	109.5
C4—C7—C10	107.2 (4)	C7—C10—H10C	109.5
C4—C7—C8	112.4 (4)	H10A—C10—H10C	109.5
C10—C7—C8	108.9 (5)	H10B—C10—H10C	109.5
C4—C7—C9	110.4 (5)	C12—C13—H13	119.5
C10—C7—C9	109.2 (5)	C14—C13—H13	119.5
C8—C7—C9	108.6 (4)	C16—C15—H15	119.4
C16—N11—C12	117.5 (5)	C14—C15—H15	119.4
C16—N11—Pd	127.0 (4)	N11—C16—H16	118.6
C12—N11—Pd	115.2 (3)	C15—C16—H16	118.6
N11—C12—C13	121.8 (5)	C17—C18—H18A	109.5
N11—C12—C2	114.7 (4)	C17—C18—H18B	109.5
C13—C12—C2	123.4 (4)	H18A—C18—H18B	109.5
C12—C13—C14	120.9 (5)	C17—C18—H18C	109.5
C13—C14—C15	115.7 (5)	H18A—C18—H18C	109.5
C13—C14—C17	122.8 (5)	H18B—C18—H18C	109.5
C15—C14—C17	121.4 (5)	C17—C19—H19A	109.5
C16—C15—C14	121.2 (5)	C17—C19—H19B	109.5
N11—C16—C15	122.8 (5)	H19A—C19—H19B	109.5
C18—C17—C14	111.6 (5)	C17—C19—H19C	109.5
C18—C17—C19	109.4 (6)	H19A—C19—H19C	109.5
C14—C17—C19	107.7 (5)	H19B—C19—H19C	109.5
C18—C17—C20	108.8 (5)	C17—C20—H20A	109.5
C14—C17—C20	110.1 (6)	C17—C20—H20B	109.5
C19—C17—C20	109.3 (6)	H20A—C20—H20B	109.5
C2—C3—H3	119.7	C17—C20—H20C	109.5
C4—C3—H3	119.7	H20A—C20—H20C	109.5
C6—C5—H5	119.7	H20B—C20—H20C	109.5

Fig. 1


I1
I2
Pd
N1
N11
C6
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C15
C2
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C10
C7
C20
C9
C19
C8
C18