

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

(5,5'-Dimethyl-2,2'-bipyridine)iodido-trimethylplatinum(IV)Fredrik Lundvall,^{a*} David Stephen Wragg^b and Mats Tilset^c

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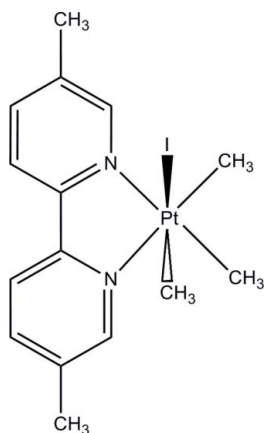
Received 17 March 2011; accepted 14 April 2011

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.008$ Å; R factor = 0.031; wR factor = 0.078; data-to-parameter ratio = 23.8.

In the title compound, $[\text{Pt}(\text{CH}_3)_3\text{I}(\text{C}_{12}\text{H}_{12}\text{N}_2)]$, the Pt^{IV} atom is six-coordinated in a slightly distorted octahedral configuration with one CH_3 group and the I atom forming a near perpendicular axis relative to the square plane formed by the bipyridine ligand and the two remaining CH_3 groups. The CH_3 group *trans* to the I atom has a slightly elongated bond to Pt compared to the other CH_3 groups, indicating a difference in *trans* influence between iodine and the bipyridine ligand.

Related literature

For synthetic background to related complexes containing $\text{Pt}(\text{CH}_3)_3$, see: Clegg *et al.* (1972); Vetter *et al.* (2006). For structural information on complexes exhibiting a similar geometrical configuration around the Pt^{IV} atom, see: Hambley (1986); Hojjat Kashani *et al.* (2008); Vetter, Bruhn & Steinborn (2010); Vetter, Wagner & Steinborn (2010). For examples of bimetallic metal-organic frameworks (MOFs), see: Bloch *et al.* (2010); Szeto *et al.* (2006, 2008).



Experimental

Crystal data

$[\text{Pt}(\text{CH}_3)_3\text{I}(\text{C}_{12}\text{H}_{12}\text{N}_2)]$	$V = 1655.8$ (6) Å ³
$M_r = 551.32$	$Z = 4$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 15.354$ (3) Å	$\mu = 10.33$ mm ⁻¹
$b = 12.394$ (2) Å	$T = 293$ K
$c = 9.0627$ (18) Å	$0.4 \times 0.4 \times 0.1$ mm
$\beta = 106.222$ (2)°	

Data collection

Bruker APEXII CCD area-detector diffractometer	18721 measured reflections
Absorption correction: numerical (SADABS; Bruker, 2005)	4094 independent reflections
$T_{\min} = 0.022$, $T_{\max} = 0.356$	3477 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.048$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.031$	172 parameters
$wR(F^2) = 0.078$	H-atom parameters constrained
$S = 1.01$	$\Delta\rho_{\text{max}} = 0.82$ e Å ⁻³
4094 reflections	$\Delta\rho_{\text{min}} = -2.14$ e Å ⁻³

Table 1

Selected geometric parameters (Å, °).

Pt1—C14	2.045 (5)	Pt1—N2	2.160 (4)
Pt1—C13	2.055 (5)	Pt1—N1	2.175 (4)
Pt1—C15	2.092 (5)	Pt1—I1	2.7755 (5)
C14—Pt1—C13	85.8 (2)	N2—Pt1—N1	76.52 (16)
C13—Pt1—N1	98.62 (18)	C15—Pt1—I1	179.94 (17)
C15—Pt1—N1	90.43 (18)		

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); data reduction: SAINT; program(s) used to solve structure: SIR92 (Altomare *et al.*, 1993); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: DIAMOND (Brandenburg, 2004); software used to prepare material for publication: publCIF (Westrip, 2010).

We acknowledge support from the Norwegian Research Council and inGAP

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LR2005).

References

- Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. (1993). *J. Appl. Cryst.* **26**, 343–350.
- Bloch, E. D., Britt, D., Lee, C., Doonan, C. J., Uribe-Rome, F. J., Furukawa, H., Long, J. R. & Yaghi, O. M. (2010). *J. Am. Chem. Soc.* **132**, 14382–14384.
- Brandenburg, K. (2004). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
- Bruker (2005). *APEX2*, *SAINTE* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Clegg, D. E., Hall, J. R. & Swile, G. A. (1972). *J. Organomet. Chem.* **38**, 403–420.
- Hambley, T. W. (1986). *Acta Cryst.* **C42**, 49–51.
- Hojjat Kashani, L., Amani, V., Yousefi, M. & Khavasi, H. R. (2008). *Acta Cryst.* **E64**, m905–m906.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Szeto, K. C., Kongshaug, K. O., Jakobsen, S., Tilset, M. & Lillerud, K. P. (2008). *Dalton Trans.* pp. 2054–2060.
- Szeto, K. C., Lillerud, K. P., Tilset, M., Bjørger, M., Prestipino, C., Zecchina, A., Lambertini, C. & Bordiga, S. (2006). *J. Phys. Chem. B*, **110**, 21509–21520.

metal-organic compounds

Vetter, C., Bruhn, C. & Steinborn, D. (2010). *Acta Cryst.* **E66**, m941–m942.
Vetter, C., Wagner, C., Schmidt, J. & Steinborn, D. (2006). *Inorg. Chim. Acta*, **359**, 4326–4334.

Vetter, C., Wagner, C. & Steinborn, D. (2010). *Acta Cryst.* **E66**, m286.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supplementary materials

Acta Cryst. (2011). E67, m617-m618 [doi:10.1107/S1600536811014085]

(5,5'-Dimethyl-2,2'-bipyridine)iodidotrimethylplatinum(IV)

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Comment

As a part of a larger project, the title compound and several other compounds based on substituted bipyridines and Pt^{IV} salts were synthesized in an NMR-screening for new Pt^{IV} complexes for potential application in bimetallic MOFs (Metal-Organic Frameworks). Bimetallic MOFs containing bipyridine, Pt(II) (Szeto *et al.*, 2006, 2008) and Pd(II) (Bloch *et al.*, 2010) have previously been reported, but so far no thermally stable bimetallic MOF containing Pt^{IV} is reported in the literature.

The title compound has previously been reported (Clegg *et al.*, 1972) as a part of an extensive NMR-studies to find evidence for *cis* and *trans* influences in trimethylplatinum(IV) compounds. The NMR spectra of the compound is in good accordance with what was reported, and the crystal structure supports the finds with regards to *trans* influences.

Experimental

The title compound was synthesized by a modified version of the method used by Clegg *et al.* (1972). 5,5'-dimethyl-2,2'-bipyridine (1.2 mg, 0.006 mmol) and Pt^{IV}(CH₃)₃I (2.0 mg, 0.005 mmol) was weighed out in an NMR-tube, and 0.5 ml of deuterated benzene (C₆D₆) was added. The resulting mixture was heated to 75 °C for 3 days without stirring. A number of NMR-spectra were recorded during the synthesis to monitor the formation of the complex. After the NMR-experiments were finished, the NMR-tube was left at ambient temperature for 7 days, during which crystals of the complex formed.

Refinement

H-atoms were positioned geometrically at distances of 0.93(Å) (CH) and 0.96(Å) (CH₃) and refined using a riding model with U_{iso}(H)=1.2 U_{eq}(C) and U_{iso}(H)=1.5 U_{eq}(C_{methyl})

Figures

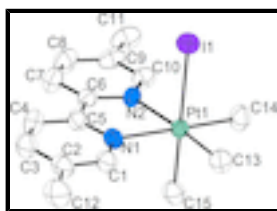


Fig. 1. [The asymmetric unit of the title compound with atom labels and 50% probability displacement ellipsoids. Hydrogen atoms are omitted for clarity.]

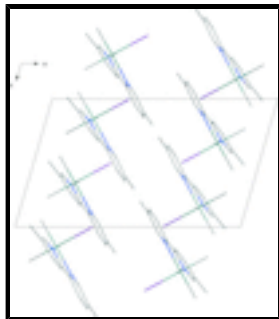


Fig. 2. [Packing diagram of the title compound viewed along the *b* axis. Hydrogen atoms are omitted for clarity.]

(5,5'-Dimethyl-2,2'-bipyridine)iodidotrimethylplatinum(IV)

Crystal data

[Pt(CH₃)₃I(C₁₂H₁₂N₂)]

M_r = 551.32

Monoclinic, *P*2₁/*c*

Hall symbol: -*P* 2ybc

a = 15.354 (3) Å

b = 12.394 (2) Å

c = 9.0627 (18) Å

β = 106.222 (2)°

V = 1655.8 (6) Å³

Z = 4

F(000) = 1024

D_x = 2.211 Mg m⁻³

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 7297 reflections

θ = 2.8–27.6°

μ = 10.33 mm⁻¹

T = 293 K

Plate, orange

0.4 × 0.4 × 0.1 mm

Data collection

Bruker APEXII CCD area-detector diffractometer

Radiation source: sealed tube graphite

φ and ω scans

Absorption correction: numerical (*SADABS*; Bruker, 2005)

T_{min} = 0.022, *T_{max}* = 0.356

18721 measured reflections

4094 independent reflections

3477 reflections with *I* > 2σ(*I*)

R_{int} = 0.048

θ_{max} = 29.0°, θ_{min} = 2.2°

h = -19→19

k = -16→16

l = -11→12

Refinement

Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2σ(*F*²)] = 0.031

wR(*F*²) = 0.078

S = 1.01

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

w = 1/[σ²(*F_o*²) + (0.045*P*)²]

where *P* = (*F_o*² + 2*F_c*²)/3

4094 reflections	$(\Delta/\sigma)_{\max} = 0.001$
172 parameters	$\Delta\rho_{\max} = 0.82 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta\rho_{\min} = -2.14 \text{ e } \text{\AA}^{-3}$

Special details

Experimental. Synthesis of the complex was performed in deuterated solvent.

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Pt1	0.208561 (12)	0.071224 (14)	0.65323 (2)	0.03239 (8)
I1	0.34139 (2)	0.03855 (3)	0.50370 (4)	0.04465 (10)
N2	0.3088 (3)	0.0375 (3)	0.8682 (4)	0.0354 (9)
C1	0.1397 (3)	-0.1655 (4)	0.5835 (6)	0.0412 (11)
H1	0.0981	-0.1344	0.4995	0.049*
C3	0.1999 (4)	-0.3208 (4)	0.7271 (7)	0.0540 (14)
H3	0.2019	-0.3949	0.7433	0.065*
C9	0.4219 (4)	0.0869 (5)	1.0999 (6)	0.0459 (13)
C8	0.4289 (4)	-0.0211 (5)	1.1424 (6)	0.0512 (14)
H8	0.4691	-0.0412	1.2355	0.061*
C7	0.3777 (4)	-0.0985 (5)	1.0502 (7)	0.0520 (14)
H7	0.3831	-0.1707	1.0796	0.062*
N1	0.1984 (3)	-0.1024 (3)	0.6788 (5)	0.0369 (9)
C13	0.1095 (4)	0.0898 (4)	0.4489 (6)	0.0440 (12)
H13A	0.0845	0.0206	0.4125	0.066*
H13B	0.1353	0.1220	0.3743	0.066*
H13C	0.0623	0.1356	0.4643	0.066*
C2	0.1371 (4)	-0.2776 (4)	0.6031 (7)	0.0474 (13)
C15	0.1083 (4)	0.0958 (4)	0.7657 (6)	0.0451 (12)
H15A	0.1350	0.0914	0.8748	0.068*
H15B	0.0623	0.0414	0.7342	0.068*
H15C	0.0817	0.1658	0.7397	0.068*
C4	0.2599 (4)	-0.2542 (4)	0.8277 (6)	0.0507 (13)
H4	0.3021	-0.2835	0.9125	0.061*
C14	0.2236 (4)	0.2350 (4)	0.6478 (6)	0.0487 (13)
H14A	0.2700	0.2578	0.7372	0.073*
H14B	0.1673	0.2694	0.6465	0.073*
H14C	0.2404	0.2548	0.5571	0.073*

supplementary materials

C11	0.4762 (4)	0.1730 (5)	1.1990 (7)	0.0587 (16)
H11A	0.5147	0.1412	1.2907	0.088*
H11B	0.4360	0.2239	1.2259	0.088*
H11C	0.5127	0.2093	1.1441	0.088*
C12	0.0671 (5)	-0.3445 (5)	0.4897 (8)	0.0661 (17)
H12A	0.0305	-0.2984	0.4115	0.099*
H12B	0.0291	-0.3806	0.5422	0.099*
H12C	0.0971	-0.3970	0.4433	0.099*
C5	0.2577 (3)	-0.1439 (4)	0.8034 (6)	0.0381 (10)
C6	0.3172 (3)	-0.0669 (4)	0.9108 (6)	0.0358 (10)
C10	0.3605 (3)	0.1121 (4)	0.9595 (5)	0.0395 (10)
H10	0.3550	0.1837	0.9276	0.047*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pt1	0.02979 (11)	0.02980 (11)	0.03496 (12)	-0.00090 (6)	0.00471 (8)	0.00234 (6)
I1	0.04027 (19)	0.0491 (2)	0.04544 (19)	-0.00213 (15)	0.01335 (15)	-0.00193 (15)
N2	0.035 (2)	0.036 (2)	0.034 (2)	0.0054 (17)	0.0069 (17)	0.0060 (16)
C1	0.043 (3)	0.035 (3)	0.048 (3)	0.000 (2)	0.018 (2)	-0.001 (2)
C3	0.063 (4)	0.032 (3)	0.072 (4)	0.002 (3)	0.027 (3)	0.006 (3)
C9	0.030 (3)	0.061 (3)	0.042 (3)	0.004 (2)	0.002 (2)	-0.009 (2)
C8	0.046 (3)	0.063 (4)	0.038 (3)	0.009 (3)	0.000 (2)	0.005 (3)
C7	0.051 (3)	0.049 (3)	0.052 (3)	0.014 (3)	0.008 (3)	0.015 (3)
N1	0.040 (2)	0.033 (2)	0.040 (2)	-0.0036 (18)	0.0136 (19)	0.0008 (17)
C13	0.041 (3)	0.044 (3)	0.041 (3)	-0.007 (2)	0.002 (2)	0.000 (2)
C2	0.052 (3)	0.031 (2)	0.068 (4)	-0.005 (2)	0.031 (3)	-0.007 (2)
C15	0.048 (3)	0.037 (3)	0.049 (3)	-0.008 (2)	0.012 (2)	-0.002 (2)
C4	0.059 (3)	0.036 (3)	0.055 (3)	0.010 (3)	0.013 (3)	0.008 (2)
C14	0.053 (3)	0.030 (3)	0.057 (3)	-0.003 (2)	0.006 (3)	0.002 (2)
C11	0.044 (3)	0.075 (4)	0.048 (3)	0.002 (3)	-0.001 (3)	-0.014 (3)
C12	0.069 (4)	0.046 (3)	0.084 (5)	-0.012 (3)	0.023 (4)	-0.012 (3)
C5	0.043 (3)	0.034 (2)	0.041 (2)	0.004 (2)	0.018 (2)	0.003 (2)
C6	0.032 (2)	0.043 (3)	0.033 (2)	0.0062 (19)	0.009 (2)	0.0047 (19)
C10	0.036 (3)	0.042 (3)	0.037 (2)	0.000 (2)	0.006 (2)	-0.003 (2)

Geometric parameters (\AA , $^\circ$)

Pt1—C14	2.045 (5)	N1—C5	1.339 (6)
Pt1—C13	2.055 (5)	C13—H13A	0.9600
Pt1—C15	2.092 (5)	C13—H13B	0.9600
Pt1—N2	2.160 (4)	C13—H13C	0.9600
Pt1—N1	2.175 (4)	C2—C12	1.511 (8)
Pt1—I1	2.7755 (5)	C15—H15A	0.9600
N2—C10	1.342 (6)	C15—H15B	0.9600
N2—C6	1.346 (6)	C15—H15C	0.9600
C1—N1	1.317 (6)	C4—C5	1.384 (7)
C1—C2	1.402 (7)	C4—H4	0.9300
C1—H1	0.9300	C14—H14A	0.9600

C3—C2	1.369 (8)	C14—H14B	0.9600
C3—C4	1.375 (8)	C14—H14C	0.9600
C3—H3	0.9300	C11—H11A	0.9600
C9—C8	1.388 (8)	C11—H11B	0.9600
C9—C10	1.392 (7)	C11—H11C	0.9600
C9—C11	1.490 (8)	C12—H12A	0.9600
C8—C7	1.367 (9)	C12—H12B	0.9600
C8—H8	0.9300	C12—H12C	0.9600
C7—C6	1.400 (7)	C5—C6	1.482 (7)
C7—H7	0.9300	C10—H10	0.9300
C14—Pt1—C13	85.8 (2)	H13B—C13—H13C	109.5
C14—Pt1—C15	88.3 (2)	C3—C2—C1	116.9 (5)
C13—Pt1—C15	87.9 (2)	C3—C2—C12	123.2 (5)
C14—Pt1—N2	99.02 (19)	C1—C2—C12	119.8 (5)
C13—Pt1—N2	175.08 (17)	Pt1—C15—H15A	109.5
C15—Pt1—N2	91.41 (19)	Pt1—C15—H15B	109.5
C14—Pt1—N1	175.34 (19)	H15A—C15—H15B	109.5
C13—Pt1—N1	98.62 (18)	Pt1—C15—H15C	109.5
C15—Pt1—N1	90.43 (18)	H15A—C15—H15C	109.5
N2—Pt1—N1	76.52 (16)	H15B—C15—H15C	109.5
C14—Pt1—I1	91.72 (16)	C3—C4—C5	120.3 (5)
C13—Pt1—I1	92.07 (16)	C3—C4—H4	119.8
C15—Pt1—I1	179.94 (17)	C5—C4—H4	119.8
N2—Pt1—I1	88.65 (10)	Pt1—C14—H14A	109.5
N1—Pt1—I1	89.55 (11)	Pt1—C14—H14B	109.5
C10—N2—C6	119.5 (4)	H14A—C14—H14B	109.5
C10—N2—Pt1	124.9 (3)	Pt1—C14—H14C	109.5
C6—N2—Pt1	115.6 (3)	H14A—C14—H14C	109.5
N1—C1—C2	122.9 (5)	H14B—C14—H14C	109.5
N1—C1—H1	118.5	C9—C11—H11A	109.5
C2—C1—H1	118.5	C9—C11—H11B	109.5
C2—C3—C4	119.8 (5)	H11A—C11—H11B	109.5
C2—C3—H3	120.1	C9—C11—H11C	109.5
C4—C3—H3	120.1	H11A—C11—H11C	109.5
C8—C9—C10	116.7 (5)	H11B—C11—H11C	109.5
C8—C9—C11	122.5 (5)	C2—C12—H12A	109.5
C10—C9—C11	120.8 (5)	C2—C12—H12B	109.5
C7—C8—C9	121.4 (5)	H12A—C12—H12B	109.5
C7—C8—H8	119.3	C2—C12—H12C	109.5
C9—C8—H8	119.3	H12A—C12—H12C	109.5
C8—C7—C6	118.6 (5)	H12B—C12—H12C	109.5
C8—C7—H7	120.7	N1—C5—C4	119.6 (5)
C6—C7—H7	120.7	N1—C5—C6	117.1 (4)
C1—N1—C5	120.3 (4)	C4—C5—C6	123.3 (5)
C1—N1—Pt1	125.0 (3)	N2—C6—C7	121.0 (5)
C5—N1—Pt1	114.7 (3)	N2—C6—C5	116.0 (4)
Pt1—C13—H13A	109.5	C7—C6—C5	123.0 (5)
Pt1—C13—H13B	109.5	N2—C10—C9	122.8 (5)
H13A—C13—H13B	109.5	N2—C10—H10	118.6

supplementary materials

Pt1—C13—H13C	109.5	C9—C10—H10	118.6
H13A—C13—H13C	109.5		
C14—Pt1—N2—C10	0.3 (4)	N1—C1—C2—C3	-0.7 (8)
C15—Pt1—N2—C10	-88.2 (4)	N1—C1—C2—C12	179.4 (5)
N1—Pt1—N2—C10	-178.3 (4)	C2—C3—C4—C5	-0.7 (8)
I1—Pt1—N2—C10	91.9 (4)	C1—N1—C5—C4	3.0 (7)
C14—Pt1—N2—C6	179.4 (3)	Pt1—N1—C5—C4	-177.3 (4)
C15—Pt1—N2—C6	90.9 (3)	C1—N1—C5—C6	-175.8 (4)
N1—Pt1—N2—C6	0.8 (3)	Pt1—N1—C5—C6	3.9 (5)
I1—Pt1—N2—C6	-89.1 (3)	C3—C4—C5—N1	-1.8 (8)
C10—C9—C8—C7	0.1 (8)	C3—C4—C5—C6	177.0 (5)
C11—C9—C8—C7	179.4 (6)	C10—N2—C6—C7	1.3 (7)
C9—C8—C7—C6	-0.2 (9)	Pt1—N2—C6—C7	-177.9 (4)
C2—C1—N1—C5	-1.8 (8)	C10—N2—C6—C5	-180.0 (4)
C2—C1—N1—Pt1	178.6 (4)	Pt1—N2—C6—C5	0.9 (5)
C13—Pt1—N1—C1	-2.1 (4)	C8—C7—C6—N2	-0.4 (8)
C15—Pt1—N1—C1	85.8 (4)	C8—C7—C6—C5	-179.1 (5)
N2—Pt1—N1—C1	177.1 (4)	N1—C5—C6—N2	-3.2 (6)
I1—Pt1—N1—C1	-94.1 (4)	C4—C5—C6—N2	178.0 (5)
C13—Pt1—N1—C5	178.2 (4)	N1—C5—C6—C7	175.5 (5)
C15—Pt1—N1—C5	-93.9 (4)	C4—C5—C6—C7	-3.3 (8)
N2—Pt1—N1—C5	-2.5 (3)	C6—N2—C10—C9	-1.4 (7)
I1—Pt1—N1—C5	86.2 (3)	Pt1—N2—C10—C9	177.6 (4)
C4—C3—C2—C1	1.9 (8)	C8—C9—C10—N2	0.8 (8)
C4—C3—C2—C12	-178.2 (5)	C11—C9—C10—N2	-178.5 (5)

Fig. 1

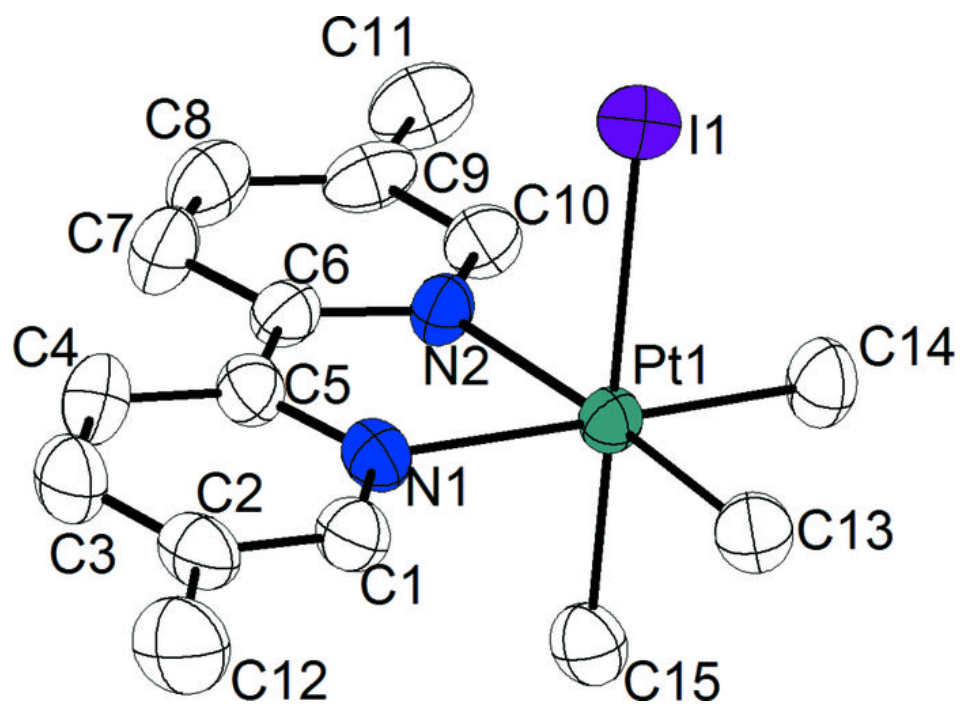


Fig. 2

