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trans-Bis[5-(4-fluorophenyl)tetrazolato]bis(triphenylphosphine)platinum(II)

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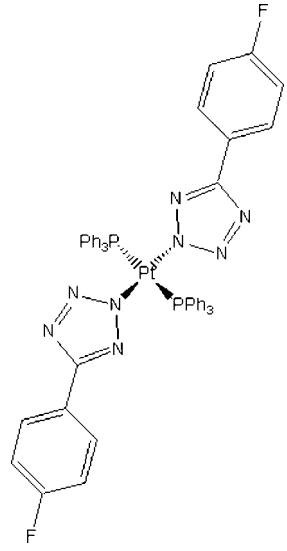
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.003 Å; R factor = 0.020; wR factor = 0.047; data-to-parameter ratio = 18.6.

The diazide platinum(II) complex, $[Pt(N_3)_2(PPh_3)_2]$, reacts with 4-FC_6H_4CN under microwave irradiation to give the corresponding neutral title bis(tetrazolate) complex *trans*-$[Pt(N_4CR)_2(PPh_3)_2]$ or $[Pt(C_7H_4FN_4)_2(C_{18}H_{15}P)_2]$. The Pt^{II} atom lies on a crystallographic inversion centre and is coordinated by two N atoms and two P atoms in a slightly distorted square-planar environment. The unique tetrazolate ring is essentially planar and the corresponding C⋯N and N⋯N bond distances lie in the range 1.317 (3)–1.349 (3) Å.

Related literature

For related literature, see: Allen *et al.* (1987); Fehlhammer *et al.* (1983); Himo *et al.* (2003); Huisgen (1968); Kim *et al.* (2002); Kukushkin & Pombeiro (2002); Le Page (1987); Mukhopadhyay *et al.* (2007); Orpen *et al.* (1989).



Experimental

Crystal data

$[Pt(C_7H_4FN_4)_2(C_{18}H_{15}P)_2]$
M_r = 1045.91
Monoclinic, $P2_1/n$
a = 11.9747 (10) Å
b = 9.5514 (8) Å
c = 18.8830 (18) Å
β = 98.125 (5)°
V = 2138.1 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 3.41 mm^{-1}
T = 150 (2) K
0.1 × 0.07 × 0.05 mm

Data collection

Bruker Kappa-APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.727, T_{max} = 0.848
60325 measured reflections
5330 independent reflections
4253 reflections with $I > 2\sigma(I)$
R_{int} = 0.064

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.047
S = 1.01
5330 reflections
286 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.59 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *WinGX* (Farrugia, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2513).

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supplementary materials

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trans-Bis[5-(4-fluorophenyl)tetrazolato]bis(triphenylphosphine)platinum(II)

S. Mukhopadhyay, J. Lasri, M. F. C. Guedes da Silva, M. A. J. Charmier and A. J. L. Pombeiro

Comment

Among heterocyles tetrazoles are an important species as they find use in *e.g.* material science, pharmaceutical chemistry and as precursors for various types of nitrogen containing heterocyles (Himo *et al.*, 2003). Cycloaddition between an organic azide (RN_3) and an organic nitrile (RCN) or isonitrile (RNC) provides tetrazoles, though in harsh conditions (Huisgen, 1968). However, the formation of substituted tetrazoles has been achieved by using a transition metal coordinated azide and free organonitriles (Kukushkin & Pombeiro, 2002) or isonitriles (Kim *et al.*, 2002). Among several transition metals, platinum(II) has been used (Fehlhammer *et al.* 1983). The existence of several possible isomers for cycloadded compounds in solution was reported by them but the crystal structures were not determined. Herein we report the crystal structure of the tetrazolato complex *trans*-[Pt(5-*p*-fluorophenyltetrazolato)$_2$(PPh_3)$_2$] (I).

Complex (I) (Fig. 1) is square-planar with the platinum(II) ion in a special position and coordinated to two N^2 atoms of the tetrazol moieties and two phosphorus of the phosphines. The tetrazolato ring is nearly planar with bond lengths in the range 1.317 (3) − 1.349 (3) Å, which is similar to those of complexes *trans*-[Pt(N_4CR)$_2$(PPh_3)$_2$] (*R* = Ph or 4-ClC_6H_4), *cis*-[Pt(N_4CPh)$_2$(2,2-bipy)] and *trans*-[Pt(C≡ *N*)(ethyltetrazolato) (PPh_3)$_2$)] (Mukhopadhyay *et al.*, 2007). All other bond lengths are normal (Allen *et al.*, 1987; Orpen *et al.*, 1989).

Experimental

The title complex was prepared by dissolving *cis*-[Pt(N_3)$_2$(PPh_3)$_2$] (0.080 g, 0.10 mmol) and 4-fluorobenzonitrile (0.137 g, 1.00 mmol) in 5 ml of DMF and irradiating the solution with focused microwave for 1 h at 373 K. The product precipitated as a white solid which was washed several times with diethyl ether and dried *in vacuo*. Single crystals were grown by keeping the mother liquid at 277 K for several days. IR (KBr pellet): 1637 ν(C═C). Anal. Calc. for $Pt_{50}H_{38}N_8P_2F_2$:*C*, 57.41; H, 3.63; N, 10.71%. Found: C,57.12; H 3.67; N10.59%. Characterization by NMR spectroscopy was not possible due to the very poor solubility of the complex in common solvents.

Refinement

All H atoms were placed in calculated positions with C—H = 0.95Å and were included in the riding-motion approximation with U_{iso}(H) = 1.2U_{eq}(C).

Figures

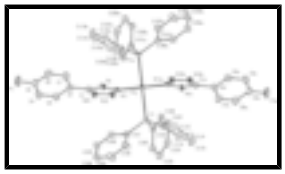


Fig. 1. The molecular structure of the title compound (50% probability displacement ellipsoids)

trans-Bis[5-(4-fluorophenyl)tetrazolato]bis(triphenylphosphine)platinum(II)

Crystal data

$[Pt(C_7H_4FN_4)_2(C_{18}H_{15}P)_2]$	F_{000} = 1040
M_r = 1045.91	D_x = 1.625 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71069 Å
a = 11.9747 (10) Å	Cell parameters from 9539 reflections
b = 9.5514 (8) Å	θ = 2.4–28.2º
c = 18.8830 (18) Å	μ = 3.41 mm^{-1}
β = 98.125 (5)º	T = 150 (2) K
V = 2138.1 (3) Å^3	Prism, colourless
Z = 2	0.1 × 0.07 × 0.05 mm

Data collection

Bruker Kappa-APEXII diffractometer	R_{int} = 0.064
φ and ω scans	θ_{max} = 28.4º
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	θ_{min} = 2.4º
T_{min} = 0.727, T_{max} = 0.848	h = −15→15
60325 measured reflections	k = −12→12
5330 independent reflections	l = −25→25
4253 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0197P)^2 + 0.8432P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.020	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.047	$\Delta\rho_{max}$ = 0.59 e Å^{-3}
S = 1.01	$\Delta\rho_{min}$ = −0.46 e Å^{-3}
5330 reflections	Extinction correction: none
286 parameters	

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.25933 (19)	0.8162 (2)	0.12471 (12)	0.0212 (5)
C2	0.35904 (19)	0.7248 (2)	0.13425 (13)	0.0236 (5)
C3	0.3966 (2)	0.6622 (2)	0.20023 (13)	0.0267 (5)
H3	0.3579	0.6794	0.2399	0.032*
C4	0.4899 (2)	0.5751 (3)	0.20797 (14)	0.0316 (6)
H4	0.5161	0.5322	0.2526	0.038*
C5	0.5436 (2)	0.5523 (3)	0.14959 (16)	0.0313 (6)
C6	0.5094 (2)	0.6118 (3)	0.08396 (15)	0.0334 (6)
H6	0.5489	0.5942	0.0446	0.04*
C7	0.4163 (2)	0.6976 (3)	0.07671 (14)	0.0308 (6)
H7	0.3907	0.7389	0.0316	0.037*
C101	−0.09454 (18)	0.6805 (2)	0.07073 (12)	0.0191 (4)
C102	−0.1385 (2)	0.5590 (2)	0.03659 (14)	0.0300 (5)
H102	−0.1903	0.5651	−0.0063	0.036*
C103	−0.1061 (2)	0.4285 (2)	0.06565 (15)	0.0364 (6)
H103	−0.1356	0.3457	0.0421	0.044*
C104	−0.0329 (2)	0.4187 (2)	0.12727 (14)	0.0306 (6)
H104	−0.0131	0.3292	0.1472	0.037*
C105	0.0133 (2)	0.5388 (3)	0.16155 (15)	0.0297 (6)
H105	0.0664	0.5315	0.2038	0.036*
C106	−0.01898 (19)	0.6690 (2)	0.13330 (13)	0.0247 (5)
H106	0.011	0.7512	0.1571	0.03*
C111	−0.25426 (18)	0.8254 (2)	−0.03436 (11)	0.0182 (4)
C112	−0.36433 (19)	0.8649 (2)	−0.02846 (13)	0.0268 (5)
H112	−0.3807	0.9086	0.0141	0.032*
C113	−0.4500 (2)	0.8407 (3)	−0.08437 (14)	0.0327 (6)
H113	−0.525	0.8686	−0.0803	0.039*
C114	−0.4269 (2)	0.7762 (3)	−0.14607 (13)	0.0305 (6)
H114	−0.4861	0.7588	−0.1841	0.037*
C115	−0.3176 (2)	0.7367 (3)	−0.15279 (14)	0.0306 (6)
H115	−0.3017	0.6919	−0.1952	0.037*
C116	−0.2319 (2)	0.7629 (2)	−0.09746 (13)	0.0245 (5)
H116	−0.1566	0.738	−0.1025	0.029*
C121	−0.20093 (17)	0.9337 (2)	0.10834 (11)	0.0172 (4)
C122	−0.19352 (19)	1.0780 (2)	0.11778 (12)	0.0223 (5)
H122	−0.1505	1.1321	0.0892	0.027*
C123	−0.2485 (2)	1.1434 (3)	0.16862 (13)	0.0279 (5)
H123	−0.2429	1.242	0.1748	0.034*

C124	−0.3111 (2)	1.0653 (3)	0.21010 (13)	0.0303 (6)
H124	−0.3479	1.1101	0.2453	0.036*
C125	−0.3206 (2)	0.9212 (3)	0.20067 (13)	0.0296 (6)
H125	−0.365	0.8679	0.2287	0.036*
C126	−0.26518 (19)	0.8557 (2)	0.15023 (12)	0.0235 (5)
H126	−0.271	0.7571	0.1441	0.028*
N1	0.19737 (15)	0.83754 (19)	0.06116 (10)	0.0203 (4)
N11	0.11799 (15)	0.92373 (18)	0.07663 (9)	0.0176 (4)
N12	0.12859 (17)	0.9569 (2)	0.14495 (11)	0.0240 (4)
N13	0.21954 (16)	0.8883 (2)	0.17724 (10)	0.0252 (4)
F1	0.63453 (14)	0.46572 (17)	0.15627 (11)	0.0472 (5)
P1	−0.13779 (5)	0.85416 (5)	0.03686 (3)	0.01532 (11)
Pt1	0	1	0	0.01374 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0218 (11)	0.0226 (11)	0.0183 (11)	−0.0007 (9)	−0.0006 (9)	0.0035 (9)
C2	0.0208 (12)	0.0250 (11)	0.0236 (12)	−0.0002 (9)	−0.0022 (10)	0.0038 (9)
C3	0.0259 (12)	0.0290 (12)	0.0237 (13)	−0.0007 (10)	−0.0015 (10)	0.0031 (10)
C4	0.0300 (13)	0.0305 (14)	0.0301 (14)	0.0017 (11)	−0.0096 (11)	0.0063 (11)
C5	0.0200 (12)	0.0243 (11)	0.0468 (17)	0.0027 (10)	−0.0050 (12)	0.0020 (12)
C6	0.0273 (13)	0.0386 (15)	0.0348 (15)	0.0038 (11)	0.0064 (11)	−0.0008 (12)
C7	0.0253 (13)	0.0407 (14)	0.0256 (13)	0.0055 (11)	0.0013 (10)	0.0050 (11)
C101	0.0201 (11)	0.0170 (10)	0.0216 (11)	0.0006 (8)	0.0079 (9)	0.0024 (8)
C102	0.0405 (15)	0.0191 (11)	0.0295 (14)	−0.0023 (11)	0.0018 (11)	0.0002 (11)
C103	0.0542 (18)	0.0150 (12)	0.0407 (16)	−0.0018 (11)	0.0096 (14)	0.0001 (11)
C104	0.0385 (15)	0.0185 (12)	0.0381 (15)	0.0070 (10)	0.0168 (12)	0.0058 (10)
C105	0.0276 (13)	0.0306 (12)	0.0315 (14)	0.0041 (10)	0.0064 (11)	0.0099 (10)
C106	0.0258 (12)	0.0204 (11)	0.0285 (13)	−0.0017 (9)	0.0054 (10)	0.0038 (9)
C111	0.0199 (11)	0.0170 (10)	0.0176 (11)	−0.0038 (8)	0.0015 (9)	0.0020 (8)
C112	0.0234 (12)	0.0337 (12)	0.0232 (13)	0.0016 (10)	0.0028 (10)	−0.0030 (10)
C113	0.0220 (12)	0.0446 (15)	0.0299 (14)	0.0033 (11)	−0.0016 (11)	−0.0009 (12)
C114	0.0303 (14)	0.0347 (13)	0.0237 (13)	−0.0065 (11)	−0.0058 (11)	0.0001 (10)
C115	0.0378 (15)	0.0323 (13)	0.0221 (13)	−0.0071 (11)	0.0056 (11)	−0.0067 (10)
C116	0.0222 (12)	0.0288 (12)	0.0231 (12)	−0.0023 (9)	0.0056 (10)	−0.0028 (10)
C121	0.0155 (10)	0.0204 (11)	0.0152 (11)	−0.0009 (8)	0.0008 (8)	0.0001 (8)
C122	0.0252 (12)	0.0196 (11)	0.0222 (12)	0.0003 (9)	0.0042 (10)	0.0003 (9)
C123	0.0298 (13)	0.0270 (12)	0.0263 (13)	0.0060 (10)	0.0011 (11)	−0.0089 (10)
C124	0.0260 (13)	0.0456 (15)	0.0197 (13)	0.0134 (11)	0.0046 (10)	−0.0041 (11)
C125	0.0260 (13)	0.0402 (15)	0.0240 (13)	0.0044 (11)	0.0080 (11)	0.0077 (11)
C126	0.0227 (11)	0.0256 (11)	0.0230 (12)	−0.0004 (9)	0.0057 (10)	0.0017 (9)
N1	0.0202 (9)	0.0221 (9)	0.0185 (10)	0.0035 (7)	0.0022 (8)	0.0037 (7)
N11	0.0186 (9)	0.0172 (9)	0.0174 (9)	−0.0025 (7)	0.0039 (7)	0.0026 (7)
N12	0.0254 (11)	0.0264 (9)	0.0200 (10)	0.0024 (8)	0.0023 (9)	0.0024 (8)
N13	0.0257 (10)	0.0294 (10)	0.0196 (10)	0.0024 (8)	0.0000 (8)	0.0035 (8)
F1	0.0299 (9)	0.0437 (9)	0.0650 (12)	0.0160 (7)	−0.0039 (8)	0.0021 (8)
P1	0.0159 (3)	0.0147 (2)	0.0155 (3)	−0.0013 (2)	0.0032 (2)	0.0006 (2)

Pt1	0.01427 (6)	0.01345 (5)	0.01367 (6)	−0.00017 (5)	0.00250 (4)	0.00131 (5)

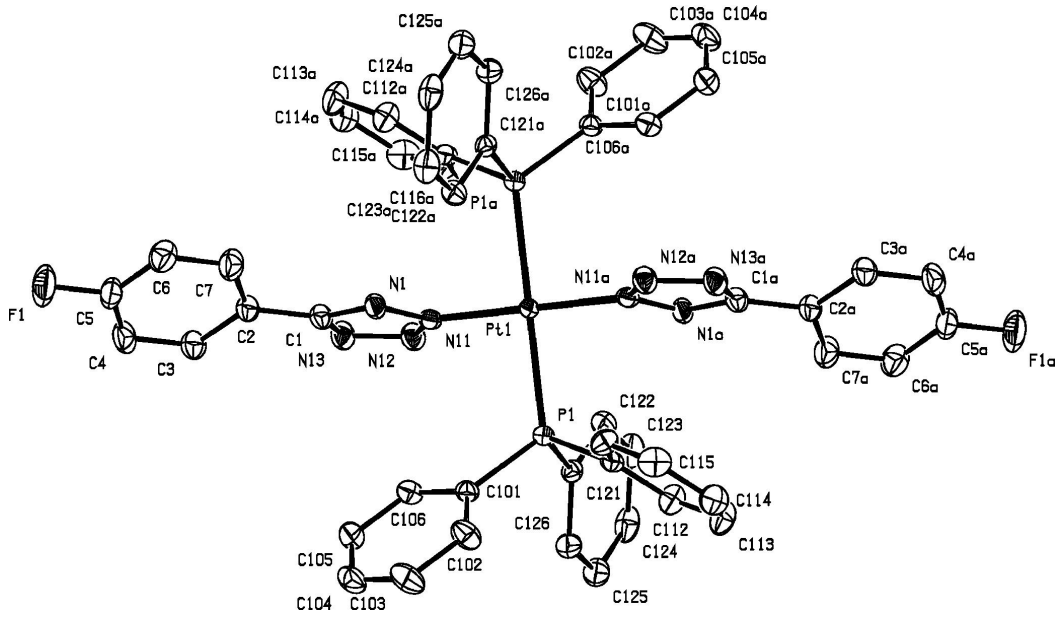
Geometric parameters (Å, °)

C1—N1	1.334 (3)	C112—C113	1.384 (3)
C1—N13	1.349 (3)	C112—H112	0.95
C1—C2	1.469 (3)	C113—C114	1.380 (3)
C2—C7	1.389 (3)	C113—H113	0.95
C2—C3	1.398 (3)	C114—C115	1.385 (4)
C3—C4	1.384 (3)	C114—H114	0.95
C3—H3	0.95	C115—C116	1.380 (3)
C4—C5	1.370 (4)	C115—H115	0.95
C4—H4	0.95	C116—H116	0.95
C5—F1	1.359 (3)	C121—C122	1.391 (3)
C5—C6	1.372 (4)	C121—C126	1.395 (3)
C6—C7	1.374 (3)	C121—P1	1.805 (2)
C6—H6	0.95	C122—C123	1.387 (3)
C7—H7	0.95	C122—H122	0.95
C101—C106	1.387 (3)	C123—C124	1.378 (4)
C101—C102	1.394 (3)	C123—H123	0.95
C101—P1	1.826 (2)	C124—C125	1.391 (4)
C102—C103	1.395 (3)	C124—H124	0.95
C102—H102	0.95	C125—C126	1.385 (3)
C103—C104	1.357 (4)	C125—H125	0.95
C103—H103	0.95	C126—H126	0.95
C104—C105	1.393 (4)	N1—N11	1.321 (2)
C104—H104	0.95	N11—N12	1.317 (3)
C105—C106	1.386 (3)	N11—Pt1	2.0106 (18)
C105—H105	0.95	N12—N13	1.341 (3)
C106—H106	0.95	P1—Pt1	2.3392 (5)
C111—C112	1.391 (3)	Pt1—N11^{i}	2.0106 (18)
C111—C116	1.392 (3)	Pt1—P1^{i}	2.3392 (5)
C111—P1	1.816 (2)		
N1—C1—N13	111.95 (19)	C112—C113—H113	119.9
N1—C1—C2	122.7 (2)	C113—C114—C115	120.3 (2)
N13—C1—C2	125.4 (2)	C113—C114—H114	119.9
C7—C2—C3	119.0 (2)	C115—C114—H114	119.9
C7—C2—C1	119.9 (2)	C116—C115—C114	119.5 (2)
C3—C2—C1	121.1 (2)	C116—C115—H115	120.3
C4—C3—C2	120.3 (2)	C114—C115—H115	120.3
C4—C3—H3	119.8	C115—C116—C111	120.9 (2)
C2—C3—H3	119.8	C115—C116—H116	119.5
C5—C4—C3	118.3 (2)	C111—C116—H116	119.5
C5—C4—H4	120.8	C122—C121—C126	119.1 (2)
C3—C4—H4	120.8	C122—C121—P1	119.13 (16)
F1—C5—C4	118.9 (2)	C126—C121—P1	121.49 (17)
F1—C5—C6	118.0 (3)	C123—C122—C121	120.5 (2)
C4—C5—C6	123.1 (2)	C123—C122—H122	119.8

C5—C6—C7	118.1 (2)	C121—C122—H122	119.8
C5—C6—H6	120.9	C124—C123—C122	120.0 (2)
C7—C6—H6	120.9	C124—C123—H123	120
C6—C7—C2	121.1 (2)	C122—C123—H123	120
C6—C7—H7	119.4	C123—C124—C125	120.3 (2)
C2—C7—H7	119.4	C123—C124—H124	119.9
C106—C101—C102	119.1 (2)	C125—C124—H124	119.9
C106—C101—P1	119.28 (17)	C126—C125—C124	119.8 (2)
C102—C101—P1	121.63 (18)	C126—C125—H125	120.1
C101—C102—C103	119.8 (2)	C124—C125—H125	120.1
C101—C102—H102	120.1	C125—C126—C121	120.3 (2)
C103—C102—H102	120.1	C125—C126—H126	119.8
C104—C103—C102	120.6 (2)	C121—C126—H126	119.8
C104—C103—H103	119.7	N11—N1—C1	102.86 (18)
C102—C103—H103	119.7	N12—N11—N1	113.18 (18)
C103—C104—C105	120.5 (2)	N12—N11—Pt1	125.35 (14)
C103—C104—H104	119.7	N1—N11—Pt1	121.41 (14)
C105—C104—H104	119.7	N11—N12—N13	106.71 (18)
C106—C105—C104	119.3 (3)	N12—N13—C1	105.29 (18)
C106—C105—H105	120.4	C121—P1—C111	104.97 (10)
C104—C105—H105	120.4	C121—P1—C101	104.38 (10)
C105—C106—C101	120.8 (2)	C111—P1—C101	105.78 (10)
C105—C106—H106	119.6	C121—P1—Pt1	111.07 (7)
C101—C106—H106	119.6	C111—P1—Pt1	111.71 (7)
C112—C111—C116	118.9 (2)	C101—P1—Pt1	117.91 (7)
C112—C111—P1	122.48 (17)	N11—Pt1—N11^{i}	180
C116—C111—P1	118.58 (17)	N11—Pt1—P1	91.35 (5)
C113—C112—C111	120.2 (2)	N11^{i}—Pt1—P1	88.65 (5)
C113—C112—H112	119.9	N11—Pt1—P1^{i}	88.65 (5)
C111—C112—H112	119.9	N11^{i}—Pt1—P1^{i}	91.35 (5)
C114—C113—C112	120.2 (2)	P1—Pt1—P1^{i}	180.00 (3)
C114—C113—H113	119.9		

Symmetry codes: (i) −*x*, −*y*+2, −*z*.

Fig. 1


C125a
C103a C104a
C113a
C124a
C102a
C105a
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C126a
C101a
C114a
C121a
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C115a
C116a
C123a C122a
P1a
C3a
C4a
N12a
N13a
N1
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C6
C7
F1
C5
C2
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C4
C3
N13
N12
N11
C5a
F1a
C7a
C6a
C122
P1
C123
C115
C101
C121
C114
C106
C126
C112
C113
C105
C102
C124
C104
C103
C125