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Dichloridobis(pyridine-2-carboxylato-κ^2N,O)platinum(IV) acetonitrile solvate

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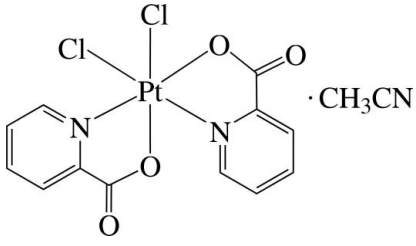
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.008 Å; R factor = 0.023; wR factor = 0.052; data-to-parameter ratio = 15.8.

The asymmetric unit of the title compound, $[PtCl_2(C_6H_4NO_2)_2]\cdot CH_3CN$, contains a neutral Pt^{IV} complex and an acetonitrile solvent molecule. In the complex, the Pt^{4+} atom is six-coordinated in a distorted octahedral environment by two N atoms and two O atoms from two pyridinecarboxylate (pic) ligands and two Cl atoms. The Cl atoms are *cis* with respect to each other. The compound displays inter- and intramolecular C—H···O and C—H···Cl hydrogen bonding.

Related literature

For the synthesis and structure of the Pt(IV)-pic complex, $[PtCl_4(pic)]^-$, see: Griffith *et al.* (2005). For a related Pt(II)-dipicolinate complex, see: Goodgame *et al.* (1995).



Experimental

Crystal data

$[PtCl_2(C_6H_4NO_2)_2]\cdot C_2H_3N$
M_r = 551.25
Monoclinic, $P2_1/c$
a = 6.103 (3) Å
b = 27.988 (12) Å
c = 9.823 (4) Å
β = 91.076 (7)°
V = 1677.7 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 8.71 mm^{-1}
T = 293 K
0.20 × 0.15 × 0.15 mm

Data collection

Bruker SMART 1000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.203, T_{max} = 0.271
9732 measured reflections
3437 independent reflections
3051 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.052
S = 1.11
3437 reflections
218 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.04 e Å^{-3}
$\Delta\rho_{min}$ = −0.58 e Å^{-3}

Table 1
Selected bond lengths (Å).

Pt1—O1	1.999 (3)	Pt1—N1	2.025 (4)
Pt1—N2	2.013 (3)	Pt1—Cl2	2.2910 (14)
Pt1—O3	2.022 (3)	Pt1—Cl1	2.3003 (13)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O2^{i}	0.93	2.45	3.207 (7)	139
C7—H7···Cl1^{ii}	0.93	2.75	3.583 (5)	150
C7—H7···Cl2	0.93	2.76	3.334 (5)	121
C10—H10···O4^{iii}	0.93	2.42	3.223 (6)	145
C13—H13A···O2^{iv}	0.96	2.43	3.256 (8)	144
C13—H13B···Cl1^{v}	0.96	2.84	3.625 (7)	140

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2009); 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: BT2950).

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supporting information

Acta Cryst. (2009). E**65**, m667 [doi:10.1107/S1600536809017966]

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S1. Comment

The asymmetric unit of the title compound, $[PtCl_2(C_6H_4NO_2)_2].CH_3CN$, contains a neutral Pt^{IV} complex and a CH_3CN solvent molecule (Fig. 1). In the complex, the Pt^{4+} ion is six-coordinated in a distorted octahedral environment by two N atoms and two O atoms from two pyridinecarboxylate (pic) anion ligands and two Cl atoms. The Cl atoms are disposed in the *cis* position. The main contributions to the distortion are the tight O—Pt—N chelate angles (82.32 (14)° and 82.16 (13)°), which result in non-linear *trans* axes (<Cl1—Pt1—N1 = 175.68 (10)°, <Cl2—Pt1—O3 = 178.67 (10)° and <O1—Pt1—N2 = 173.39 (14)°). The different *trans* effects of the Cl, O and N atoms are not distinct, because the Pt1—Cl, Pt1—O and Pt1—N bond lengths are almost equal (Pt1—Cl: 2.3003 (13) and 2.2910 (14) Å; Pt1—O 1.999 (3) and 2.022 (3) Å; Pt1—N 2.025 (4) and 2.013 (3) Å), respectively (Table 1). The compound displays inter- and intramolecular C—H···O and C—H···Cl hydrogen bonding (Table 2 and Fig. 2). There may also be weak intermolecular π-π interactions between adjacent pyridine rings, with a shortest centroid-centroid distance of 5.223 (4) Å.

S2. Experimental

A suspension of K_2PtCl_6 (0.2148 g, 0.442 mmol) and pyridine-2-carboxylic acid (0.2000 g, 1.459 mmol) in H_2O (10 ml) was refluxed for 5 h. The formed precipitate was separated by filtration and washed with water (20 ml) and dried under vacuum, to give a pale green powder (0.2304 g). Colorless crystals suitable for X-ray analysis were obtained by slow evaporation from a CH_3CN solution.

S3. Refinement

H atoms were positioned geometrically and allowed to ride on their respective parent atoms [C—H = 0.93 (aromatic) or 0.96 Å (CH_3) and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C)].

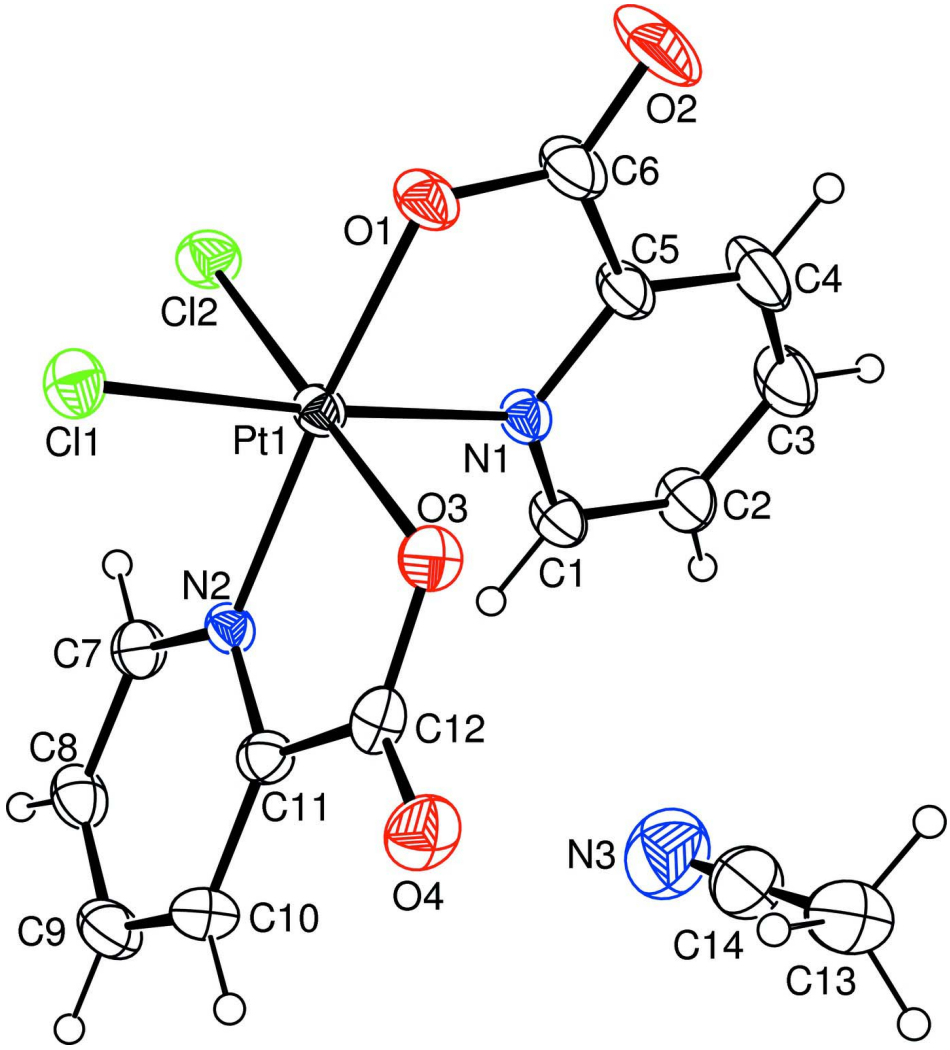

Figure 1

The structure of the title compound, with displacement ellipsoids drawn at the 30% probability level for non-H atoms.

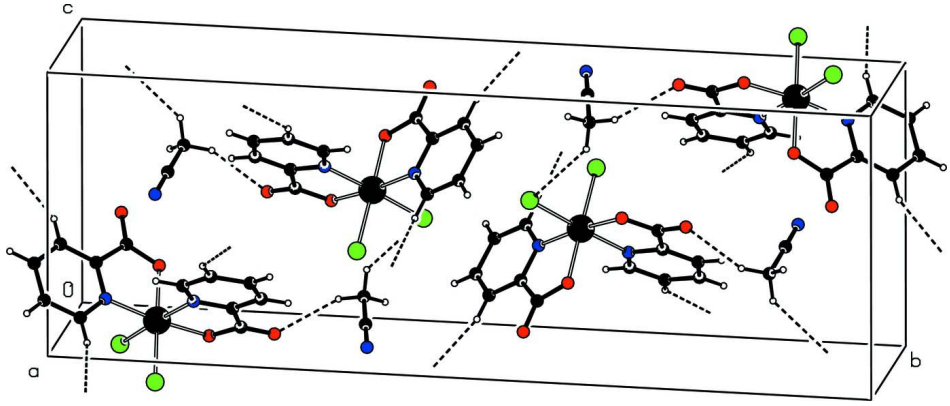

Figure 2

View of the unit-cell contents of the title compound. Hydrogen-bond interactions are drawn with dashed lines.

Dichloridobis(pyridine-2-carboxylato-κ^2N,O)platinum(IV) acetonitrile solvate

Crystal data

$[PtCl_2(C_6H_4NO_2)_2]\cdot C_2H_3N$
$M_r = 551.25$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 6.103$ (3) Å
$b = 27.988$ (12) Å
$c = 9.823$ (4) Å
$\beta = 91.076$ (7)°
$V = 1677.7$ (12) Å^3
$Z = 4$
$F(000) = 1040$
$D_x = 2.182$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 979 reflections
$\theta = 2.5$–$25.9°$
$\mu = 8.71$ mm^{-1}
$T = 293$ K
Stick, colorless
0.20 × 0.15 × 0.15 mm

Data collection

Bruker SMART 1000 CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.203$, $T_{max} = 0.271$
9732 measured reflections
3437 independent reflections
3051 reflections with $I > 2\sigma(I)$
$R_{int} = 0.025$
$\theta_{max} = 26.4°$, $\theta_{min} = 1.5°$
$h = -6\rightarrow7$
$k = -35\rightarrow33$
$l = -12\rightarrow12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.023$
$wR(F^2) = 0.052$
$S = 1.11$
3437 reflections
218 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0126P)^2 + 3.1343P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.04$ e Å^{-3}
$\Delta\rho_{min} = -0.58$ e Å^{-3}

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}
Pt1	0.67421 (3)	0.119200 (6)	0.082756 (17)	0.03061 (6)
Cl1	0.47415 (19)	0.06563 (4)	−0.04645 (12)	0.0421 (3)
Cl2	0.9275 (2)	0.12741 (4)	−0.08481 (13)	0.0454 (3)
O1	0.5138 (5)	0.17577 (11)	0.0066 (3)	0.0444 (8)

O2	0.4983 (9)	0.25418 (14)	0.0228 (6)	0.0971 (19)
O3	0.4557 (5)	0.11261 (11)	0.2338 (3)	0.0389 (7)
O4	0.3574 (6)	0.06264 (14)	0.3949 (4)	0.0533 (10)
N1	0.8376 (6)	0.17036 (13)	0.1894 (4)	0.0336 (8)
N2	0.8101 (6)	0.06220 (12)	0.1765 (4)	0.0297 (8)
C1	1.0064 (8)	0.16447 (16)	0.2751 (5)	0.0410 (11)
H1	1.0535	0.1337	0.2961	0.049*
C2	1.1128 (9)	0.20275 (18)	0.3335 (5)	0.0504 (14)
H2	1.2320	0.1980	0.3923	0.060*
C3	1.0415 (10)	0.2478 (2)	0.3040 (6)	0.0610 (16)
H3	1.1108	0.2743	0.3428	0.073*
C4	0.8651 (11)	0.25356 (19)	0.2158 (7)	0.0680 (19)
H4	0.8137	0.2841	0.1957	0.082*
C5	0.7654 (9)	0.21476 (17)	0.1578 (5)	0.0452 (12)
C6	0.5793 (10)	0.21694 (19)	0.0564 (6)	0.0550 (15)
C7	0.9907 (8)	0.03922 (17)	0.1407 (5)	0.0380 (11)
H7	1.0735	0.0509	0.0695	0.046*
C8	1.0567 (9)	−0.00183 (17)	0.2081 (5)	0.0451 (12)
H8	1.1841	−0.0176	0.1832	0.054*
C9	0.9325 (10)	−0.01915 (18)	0.3122 (6)	0.0520 (14)
H9	0.9745	−0.0468	0.3581	0.062*
C10	0.7448 (9)	0.00483 (18)	0.3481 (5)	0.0476 (13)
H10	0.6595	−0.0064	0.4187	0.057*
C11	0.6848 (7)	0.04566 (16)	0.2781 (4)	0.0341 (10)
C12	0.4840 (8)	0.07409 (17)	0.3081 (5)	0.0386 (11)
N3	0.7810 (12)	0.1214 (2)	0.5485 (7)	0.091 (2)
C13	0.4113 (12)	0.1411 (2)	0.6554 (7)	0.0761 (19)
H13A	0.3723	0.1734	0.6331	0.114*
H13B	0.4226	0.1376	0.7525	0.114*
H13C	0.3007	0.1198	0.6201	0.114*
C14	0.6175 (14)	0.1297 (2)	0.5963 (7)	0.0677 (18)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pt1	0.02912 (10)	0.02809 (10)	0.03449 (10)	0.00214 (7)	−0.00261 (7)	0.00113 (7)
Cl1	0.0381 (6)	0.0414 (6)	0.0465 (7)	−0.0055 (5)	−0.0040 (5)	−0.0048 (5)
Cl2	0.0479 (7)	0.0448 (7)	0.0439 (7)	−0.0037 (5)	0.0083 (5)	0.0066 (5)
O1	0.044 (2)	0.0332 (18)	0.055 (2)	0.0085 (15)	−0.0173 (17)	0.0024 (15)
O2	0.122 (4)	0.035 (2)	0.131 (4)	0.026 (2)	−0.079 (4)	−0.002 (2)
O3	0.0307 (17)	0.0435 (18)	0.0428 (19)	0.0064 (14)	0.0072 (14)	−0.0001 (15)
O4	0.048 (2)	0.063 (2)	0.049 (2)	−0.0049 (18)	0.0169 (18)	−0.0001 (18)
N1	0.033 (2)	0.030 (2)	0.037 (2)	0.0027 (16)	−0.0055 (17)	−0.0021 (16)
N2	0.031 (2)	0.0259 (18)	0.0325 (19)	0.0037 (15)	−0.0014 (16)	−0.0002 (15)
C1	0.040 (3)	0.031 (2)	0.052 (3)	0.002 (2)	−0.009 (2)	0.001 (2)
C2	0.051 (3)	0.044 (3)	0.055 (3)	−0.002 (2)	−0.019 (3)	−0.006 (2)
C3	0.069 (4)	0.039 (3)	0.074 (4)	−0.005 (3)	−0.023 (3)	−0.010 (3)
C4	0.084 (5)	0.027 (3)	0.092 (5)	0.009 (3)	−0.031 (4)	−0.009 (3)

C5	0.052 (3)	0.030 (3)	0.053 (3)	0.010 (2)	−0.013 (3)	0.000 (2)
C6	0.064 (4)	0.036 (3)	0.064 (4)	0.013 (3)	−0.024 (3)	0.001 (3)
C7	0.036 (3)	0.038 (3)	0.040 (3)	0.003 (2)	0.003 (2)	−0.002 (2)
C8	0.044 (3)	0.037 (3)	0.055 (3)	0.010 (2)	−0.007 (2)	−0.004 (2)
C9	0.065 (4)	0.034 (3)	0.057 (3)	0.004 (3)	−0.011 (3)	0.006 (2)
C10	0.060 (4)	0.042 (3)	0.041 (3)	−0.002 (3)	0.002 (3)	0.012 (2)
C11	0.036 (3)	0.035 (2)	0.032 (2)	−0.005 (2)	−0.001 (2)	0.0020 (19)
C12	0.038 (3)	0.043 (3)	0.035 (3)	−0.003 (2)	0.002 (2)	−0.009 (2)
N3	0.097 (5)	0.099 (5)	0.078 (4)	0.003 (4)	−0.014 (4)	−0.009 (4)
C13	0.097 (6)	0.065 (4)	0.067 (4)	−0.004 (4)	0.001 (4)	0.012 (3)
C14	0.092 (6)	0.057 (4)	0.053 (4)	−0.002 (4)	−0.017 (4)	−0.002 (3)

Geometric parameters (Å, º)

Pt1—O1	1.999 (3)	C3—H3	0.9300
Pt1—N2	2.013 (3)	C4—C5	1.364 (7)
Pt1—O3	2.022 (3)	C4—H4	0.9300
Pt1—N1	2.025 (4)	C5—C6	1.498 (7)
Pt1—Cl2	2.2910 (14)	C7—C8	1.382 (6)
Pt1—Cl1	2.3003 (13)	C7—H7	0.9300
O1—C6	1.311 (6)	C8—C9	1.373 (7)
O2—C6	1.197 (6)	C8—H8	0.9300
O3—C12	1.312 (6)	C9—C10	1.379 (7)
O4—C12	1.205 (6)	C9—H9	0.9300
N1—C1	1.328 (6)	C10—C11	1.379 (6)
N1—C5	1.352 (6)	C10—H10	0.9300
N2—C7	1.329 (6)	C11—C12	1.495 (6)
N2—C11	1.350 (5)	N3—C14	1.134 (10)
C1—C2	1.373 (6)	C13—C14	1.432 (10)
C1—H1	0.9300	C13—H13A	0.9600
C2—C3	1.364 (7)	C13—H13B	0.9600
C2—H2	0.9300	C13—H13C	0.9600
C3—C4	1.379 (8)		
O1—Pt1—N2	173.39 (14)	C5—C4—H4	119.8
O1—Pt1—O3	91.24 (14)	C3—C4—H4	119.8
N2—Pt1—O3	82.16 (13)	N1—C5—C4	119.7 (5)
O1—Pt1—N1	82.32 (14)	N1—C5—C6	115.4 (4)
N2—Pt1—N1	97.41 (15)	C4—C5—C6	124.9 (5)
O3—Pt1—N1	90.57 (14)	O2—C6—O1	122.7 (5)
O1—Pt1—Cl2	89.09 (11)	O2—C6—C5	121.5 (5)
N2—Pt1—Cl2	97.51 (11)	O1—C6—C5	115.7 (4)
O3—Pt1—Cl2	178.67 (10)	N2—C7—C8	120.7 (5)
N1—Pt1—Cl2	88.19 (11)	N2—C7—H7	119.6
O1—Pt1—Cl1	93.37 (10)	C8—C7—H7	119.6
N2—Pt1—Cl1	86.89 (11)	C9—C8—C7	119.4 (5)
O3—Pt1—Cl1	89.70 (10)	C9—C8—H8	120.3
N1—Pt1—Cl1	175.68 (10)	C7—C8—H8	120.3

Cl2—Pt1—Cl1	91.57 (5)	C8—C9—C10	119.4 (5)
C6—O1—Pt1	114.4 (3)	C8—C9—H9	120.3
C12—O3—Pt1	113.7 (3)	C10—C9—H9	120.3
C1—N1—C5	120.3 (4)	C9—C10—C11	119.3 (5)
C1—N1—Pt1	127.5 (3)	C9—C10—H10	120.3
C5—N1—Pt1	112.1 (3)	C11—C10—H10	120.3
C7—N2—C11	120.9 (4)	N2—C11—C10	120.2 (4)
C7—N2—Pt1	126.8 (3)	N2—C11—C12	116.2 (4)
C11—N2—Pt1	112.1 (3)	C10—C11—C12	123.6 (4)
N1—C1—C2	121.5 (4)	O4—C12—O3	122.2 (5)
N1—C1—H1	119.2	O4—C12—C11	122.5 (5)
C2—C1—H1	119.2	O3—C12—C11	115.3 (4)
C3—C2—C1	119.2 (5)	C14—C13—H13A	109.5
C3—C2—H2	120.4	C14—C13—H13B	109.5
C1—C2—H2	120.4	H13A—C13—H13B	109.5
C2—C3—C4	118.9 (5)	C14—C13—H13C	109.5
C2—C3—H3	120.6	H13A—C13—H13C	109.5
C4—C3—H3	120.6	H13B—C13—H13C	109.5
C5—C4—C3	120.5 (5)	N3—C14—C13	178.8 (8)

O3—Pt1—O1—C6	89.9 (4)	C1—N1—C5—C4	−0.8 (8)
N1—Pt1—O1—C6	−0.5 (4)	Pt1—N1—C5—C4	−176.9 (5)
Cl2—Pt1—O1—C6	−88.8 (4)	C1—N1—C5—C6	177.9 (5)
Cl1—Pt1—O1—C6	179.7 (4)	Pt1—N1—C5—C6	1.8 (6)
O1—Pt1—O3—C12	173.1 (3)	C3—C4—C5—N1	1.3 (10)
N2—Pt1—O3—C12	−7.2 (3)	C3—C4—C5—C6	−177.3 (6)
N1—Pt1—O3—C12	−104.6 (3)	Pt1—O1—C6—O2	−179.1 (6)
Cl1—Pt1—O3—C12	79.7 (3)	Pt1—O1—C6—C5	1.6 (7)
O1—Pt1—N1—C1	−176.5 (4)	N1—C5—C6—O2	178.3 (6)
N2—Pt1—N1—C1	10.1 (4)	C4—C5—C6—O2	−3.0 (11)
O3—Pt1—N1—C1	92.3 (4)	N1—C5—C6—O1	−2.4 (8)
Cl2—Pt1—N1—C1	−87.2 (4)	C4—C5—C6—O1	176.3 (6)
O1—Pt1—N1—C5	−0.8 (3)	C11—N2—C7—C8	−1.0 (7)
N2—Pt1—N1—C5	−174.1 (3)	Pt1—N2—C7—C8	−174.9 (3)
O3—Pt1—N1—C5	−92.0 (4)	N2—C7—C8—C9	0.7 (7)
Cl2—Pt1—N1—C5	88.5 (3)	C7—C8—C9—C10	−0.3 (8)
O3—Pt1—N2—C7	−179.5 (4)	C8—C9—C10—C11	0.3 (8)
N1—Pt1—N2—C7	−89.9 (4)	C7—N2—C11—C10	1.0 (7)
Cl2—Pt1—N2—C7	−0.8 (4)	Pt1—N2—C11—C10	175.7 (4)
Cl1—Pt1—N2—C7	90.4 (4)	C7—N2—C11—C12	−179.2 (4)
O3—Pt1—N2—C11	6.2 (3)	Pt1—N2—C11—C12	−4.5 (5)
N1—Pt1—N2—C11	95.7 (3)	C9—C10—C11—N2	−0.7 (7)
Cl2—Pt1—N2—C11	−175.1 (3)	C9—C10—C11—C12	179.6 (5)
Cl1—Pt1—N2—C11	−84.0 (3)	Pt1—O3—C12—O4	−173.6 (4)
C5—N1—C1—C2	−0.3 (8)	Pt1—O3—C12—C11	6.7 (5)
Pt1—N1—C1—C2	175.1 (4)	N2—C11—C12—O4	178.8 (4)
N1—C1—C2—C3	0.9 (8)	C10—C11—C12—O4	−1.4 (7)
C1—C2—C3—C4	−0.3 (9)	N2—C11—C12—O3	−1.5 (6)

C2—C3—C4—C5	−0.8 (10)	C10—C11—C12—O3	178.3 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O2^{i}	0.93	2.45	3.207 (7)	139
C7—H7···Cl1^{ii}	0.93	2.75	3.583 (5)	150
C7—H7···Cl2	0.93	2.76	3.334 (5)	121
C10—H10···O4^{iii}	0.93	2.42	3.223 (6)	145
C13—H13*A*···O2^{iv}	0.96	2.43	3.256 (8)	144
C13—H13*B*···Cl1^{v}	0.96	2.84	3.625 (7)	140

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