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cis-(Malonato-κ^2O,O')bis(2-methylpyridine-κN)platinum(II)

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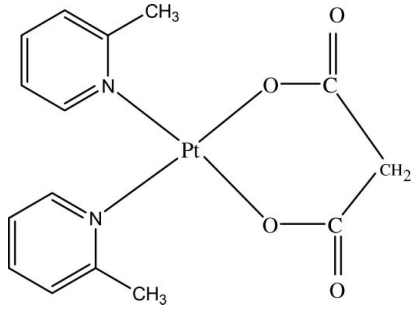
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.006 Å; R factor = 0.021; wR factor = 0.046; data-to-parameter ratio = 17.6.

In the crystal structure of the title compound, $[Pt(C_3H_2O_4)(C_6H_7N)_2]$, the Pt^{II} ion is tetracoordinated in a square-planar coordination geometry. There are intermolecular C—H···O hydrogen bonds.

Related literature

For related literature, see: Ali *et al.* (2002); Jakuper *et al.* (2003); Tu *et al.* (2004); Zhang *et al.* (2002).



Experimental

Crystal data

$[Pt(C_3H_2O_4)(C_6H_7N)_2]$
M_r = 483.39
Monoclinic, $P2_1/n$
a = 8.5790 (6) Å
b = 15.8079 (11) Å
c = 11.5595 (8) Å
β = 107.748 (1)°
V = 1493.04 (18) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 9.42 mm^{-1}
T = 298 (2) K
0.24 × 0.19 × 0.16 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*APEX2*; Bruker, 2004)
T_{min} = 0.141, T_{max} = 0.235
12687 measured reflections
3532 independent reflections
3007 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.021
$wR(F^2)$ = 0.046
S = 0.95
3532 reflections
201 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.76 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10···O1^{i}	0.93	2.53	3.455 (5)	173
C6—H6B···O3^{ii}	0.96	2.55	3.422 (5)	151
C6—H6A···O1	0.96	2.55	3.254 (5)	130

Symmetry codes: (i) $-x+1, -y, -z$; (ii) $x+1, y, z$.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2000); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2058).

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supplementary materials

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cis-(Malonato-κ^2O,O')bis(2-methylpyridine-κN)platinum(II)

M.-J. Xie, Y. Yu, W.-P. Liu, S.-Q. Hou and X.-Z. Chen

Comment

cis-diammine(1,1-cyclobutanedicarboxylato) platinum(II) (Carboplatin) is commonly used for the treatment of testicular and overian cancer as well as cervical, bladder and head and neck tumors. It has proven to be the only second-generation platinum complex commercially available worldwide at present (Jakuper *et al.*, 2003). But the application of carboplatin in therapy is limited by the dose-dependent nephrotoxicity and other side effects. Therefore, the search for the new potent platinum complexes possessing high antitumor activity and lack of cross-resistance is needed. The title compound is a new soluble carboplatin analogue containing an asymmetric chelating malonate anion as its carrier and anticancer tests are presently being carried out.

The title complex consists of discrete monomeric complexes, in which the Pt(II) is coordinated by two crystallographically independent 2-methylpyridine ligands and one malonate anions within an square planar geometry. The coordination behaviour of the tetradentate ligand displays similar features to those described in the literature (Tu *et al.*, 2004; Zhang *et al.*, 2002; Ali *et al.*, 2002). The six-membered chelate ring built up of the Pt(II) atom and the malonate anion adopts a boat conformation and the two 2-methylpyridine liagnds are oriented perpendicular to each other.

Experimental

Potassium tetrachloroplatinate(II) (5 g, 12 mmol) was dissolved in water (50 ml) and KI (12 g, 72 mmol) was added. The mixture were stored in the dark for 30 min at room temperature and afterwards a solution of 2-methylpyridine (1.08 g, 12 mmol in 50 ml water) was added dropwise. The mixture was stirred for 4 h and the yellow precipitate of di(2-methylpyridine)PtI_2 was filtered off. Afterwards 2.5 g (0.044 mmol) of di(2-methylpyridine)PtI_2, 75 ml of water and disilver malonate (1.36 g, 3.65 mmol) were stirred at 50°C for 72 h. The precipitate of AgI was filtered off and the filtrate was concentrated at 40°C under reduced pressure to about 5 ml until a white crystalline solid of the title compound precipitate. The compound was recrystallized from water to obtain crystals suitable for X-ray crystallography.

Refinement

All H atoms were initially located in a difference Fourier map but were positioned with idealized geometry and refined isotropic with U_{iso}(H) = 1.2U_{eq}(C) (1.5 for methyl H atoms) using a riding model with C—H = 0.93 (aromatic), 0.97 (methylen) and 0.96 Å) (methyl).

Figures

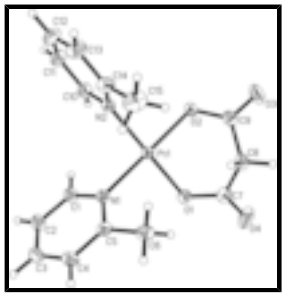


Fig. 1. Molecular view of the complex, with the atomic labeling scheme. Displacement ellipsoids are drawn at the 30% probability level.

cis-(Malonato-κ^2*O*,*O*')bis(2-methylpyridine-κ*N*)platinum(II)

Crystal data

$[Pt(C_3H_2O_4)(C_6H_7N)_2]$	F_{000} = 920
M_r = 483.39	D_x = 2.150 Mg m^{-3}
Monoclinic, *P*21/*n*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 3532 reflections
a = 8.5790 (6) Å	θ = 2.3–28.3º
b = 15.8079 (11) Å	μ = 9.42 mm^{-1}
c = 11.5595 (8) Å	*T* = 298 (2) K
β = 107.7480 (10)º	Block, colourless
V = 1493.04 (18) Å^3	0.24 × 0.19 × 0.16 mm
Z = 4	

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	3532 independent reflections
Radiation source: fine-focus sealed tube	3007 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.029
T = 298(2) K	θ_{max} = 28.3º
φ and ω scans	θ_{min} = 2.3º
Absorption correction: numerical (APEX2; Bruker, 2004)	*h* = −11→11
T_{min} = 0.141, T_{max} = 0.235	*k* = −20→20
12687 measured reflections	*l* = −15→15

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.021	H-atom parameters constrained
$wR(F^2)$ = 0.046	$w = 1/[\sigma^2(F_o^2) + (0.0222P)^2 + 0.6402P]$

	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.95$	$(\Delta/\sigma)_{max} = 0.002$
3532 reflections	$\Delta\rho_{max} = 0.53$ e Å^{-3}
201 parameters	$\Delta\rho_{min} = -0.76$ 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.

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	0.636843 (15)	0.055740 (8)	0.171724 (11)	0.02722 (5)
N1	0.8270 (3)	−0.00984 (17)	0.1478 (2)	0.0287 (6)
N2	0.6310 (3)	−0.01854 (18)	0.3124 (3)	0.0306 (6)
O1	0.6260 (3)	0.12457 (15)	0.0230 (2)	0.0361 (6)
O2	0.4457 (3)	0.11978 (15)	0.1953 (2)	0.0384 (6)
O3	0.3179 (4)	0.24193 (18)	0.1819 (3)	0.0663 (9)
O4	0.5635 (5)	0.24575 (19)	−0.0733 (3)	0.0718 (10)
C1	0.8018 (4)	−0.0905 (2)	0.1111 (3)	0.0374 (8)
H1	0.6975	−0.1132	0.0956	0.045*
C2	0.9251 (5)	−0.1411 (2)	0.0956 (4)	0.0436 (9)
H2	0.9039	−0.1969	0.0700	0.052*
C3	1.0802 (5)	−0.1078 (3)	0.1186 (3)	0.0443 (9)
H3	1.1662	−0.1411	0.1117	0.053*
C4	1.1050 (4)	−0.0241 (3)	0.1521 (3)	0.0410 (9)
H4	1.2080	−0.0001	0.1656	0.049*
C5	0.9767 (4)	0.0250 (2)	0.1660 (3)	0.0318 (7)
C6	1.0016 (5)	0.1165 (2)	0.2008 (4)	0.0428 (9)
H6A	0.9258	0.1504	0.1402	0.064*
H6B	1.1115	0.1328	0.2066	0.064*
H6C	0.9832	0.1250	0.2779	0.064*
C7	0.5863 (4)	0.2049 (2)	0.0186 (3)	0.0400 (9)
C8	0.5686 (5)	0.2435 (2)	0.1339 (3)	0.0401 (9)
H8A	0.5444	0.3033	0.1208	0.048*
H8B	0.6714	0.2380	0.1984	0.048*
C9	0.4338 (4)	0.2013 (2)	0.1732 (3)	0.0392 (9)
C10	0.5415 (4)	−0.0904 (2)	0.2854 (4)	0.0395 (8)

H10	0.4901	−0.1035	0.2042	0.047*
C11	0.5237 (5)	−0.1446 (2)	0.3729 (4)	0.0520 (11)
H11	0.4602	−0.1931	0.3512	0.062*
C12	0.6005 (6)	−0.1267 (3)	0.4929 (4)	0.0576 (12)
H12	0.5932	−0.1635	0.5538	0.069*
C13	0.6891 (6)	−0.0524 (3)	0.5207 (4)	0.0547 (11)
H13	0.7399	−0.0384	0.6017	0.066*
C14	0.7036 (5)	0.0014 (3)	0.4304 (3)	0.0415 (9)
C15	0.7993 (6)	0.0816 (3)	0.4616 (4)	0.0636 (13)
H15A	0.9039	0.0744	0.4487	0.095*
H15B	0.8148	0.0954	0.5452	0.095*
H15C	0.7407	0.1265	0.4108	0.095*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pt1	0.02567 (7)	0.02699 (8)	0.02939 (8)	0.00007 (5)	0.00899 (5)	−0.00007 (5)
N1	0.0244 (14)	0.0320 (15)	0.0288 (14)	0.0016 (12)	0.0070 (11)	0.0013 (12)
N2	0.0252 (14)	0.0321 (15)	0.0342 (15)	0.0014 (12)	0.0084 (12)	0.0009 (13)
O1	0.0420 (14)	0.0362 (14)	0.0310 (13)	0.0048 (11)	0.0125 (11)	0.0050 (11)
O2	0.0345 (14)	0.0330 (14)	0.0530 (16)	0.0052 (11)	0.0212 (12)	0.0032 (12)
O3	0.0483 (18)	0.0439 (17)	0.115 (3)	0.0094 (14)	0.0381 (19)	−0.0085 (18)
O4	0.120 (3)	0.0455 (18)	0.0496 (19)	0.0080 (18)	0.0254 (19)	0.0192 (15)
C1	0.0315 (19)	0.039 (2)	0.042 (2)	0.0010 (16)	0.0105 (16)	−0.0006 (17)
C2	0.052 (2)	0.033 (2)	0.047 (2)	0.0064 (18)	0.0173 (19)	−0.0038 (17)
C3	0.043 (2)	0.053 (3)	0.040 (2)	0.0174 (19)	0.0182 (18)	0.0073 (19)
C4	0.0287 (19)	0.057 (2)	0.038 (2)	0.0051 (17)	0.0111 (16)	0.0029 (18)
C5	0.0314 (18)	0.0384 (19)	0.0242 (17)	0.0017 (15)	0.0062 (14)	0.0038 (15)
C6	0.035 (2)	0.047 (2)	0.047 (2)	−0.0091 (17)	0.0132 (17)	−0.0061 (18)
C7	0.037 (2)	0.039 (2)	0.041 (2)	−0.0022 (16)	0.0071 (17)	0.0033 (17)
C8	0.042 (2)	0.0283 (19)	0.049 (2)	0.0016 (16)	0.0124 (18)	−0.0034 (17)
C9	0.0321 (19)	0.040 (2)	0.045 (2)	0.0001 (16)	0.0101 (17)	−0.0067 (17)
C10	0.037 (2)	0.035 (2)	0.047 (2)	−0.0030 (16)	0.0134 (17)	−0.0001 (17)
C11	0.053 (3)	0.034 (2)	0.074 (3)	−0.0059 (19)	0.027 (2)	0.010 (2)
C12	0.064 (3)	0.058 (3)	0.057 (3)	0.003 (2)	0.027 (2)	0.024 (2)
C13	0.056 (3)	0.070 (3)	0.036 (2)	0.000 (2)	0.010 (2)	0.011 (2)
C14	0.036 (2)	0.050 (2)	0.037 (2)	−0.0046 (18)	0.0102 (16)	−0.0012 (18)
C15	0.071 (3)	0.068 (3)	0.048 (3)	−0.028 (3)	0.013 (2)	−0.015 (2)

Geometric parameters (Å, °)

Pt1—O1	2.013 (2)	C5—C6	1.499 (5)
Pt1—O2	2.015 (2)	C6—H6A	0.9600
Pt1—N2	2.019 (3)	C6—H6B	0.9600
Pt1—N1	2.022 (3)	C6—H6C	0.9600
N1—C1	1.340 (5)	C7—C8	1.515 (5)
N1—C5	1.354 (4)	C8—C9	1.519 (5)
N2—C14	1.353 (4)	C8—H8A	0.9700
N2—C10	1.353 (5)	C8—H8B	0.9700

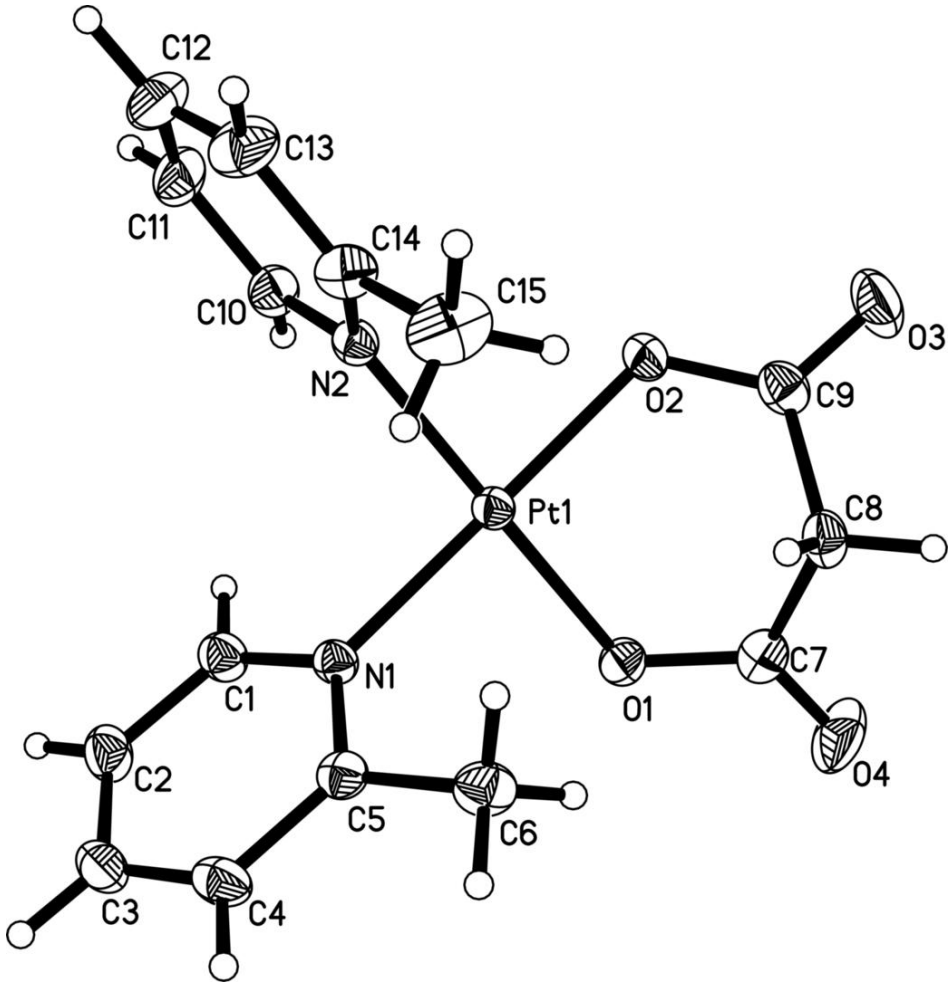
O1—C7	1.312 (4)	C10—C11	1.369 (5)
O2—C9	1.311 (4)	C10—H10	0.9300
O3—C9	1.213 (4)	C11—C12	1.371 (6)
O4—C7	1.207 (4)	C11—H11	0.9300
C1—C2	1.381 (5)	C12—C13	1.383 (6)
C1—H1	0.9300	C12—H12	0.9300
C2—C3	1.380 (5)	C13—C14	1.382 (5)
C2—H2	0.9300	C13—H13	0.9300
C3—C4	1.376 (6)	C14—C15	1.493 (6)
C3—H3	0.9300	C15—H15A	0.9600
C4—C5	1.395 (5)	C15—H15B	0.9600
C4—H4	0.9300	C15—H15C	0.9600
O1—Pt1—O2	90.94 (10)	H6B—C6—H6C	109.5
O1—Pt1—N2	175.20 (10)	O4—C7—O1	121.4 (4)
O2—Pt1—N2	87.82 (10)	O4—C7—C8	121.8 (3)
O1—Pt1—N1	89.39 (10)	O1—C7—C8	116.7 (3)
O2—Pt1—N1	179.30 (10)	C7—C8—C9	112.1 (3)
N2—Pt1—N1	91.80 (11)	C7—C8—H8A	109.2
C1—N1—C5	119.3 (3)	C9—C8—H8A	109.2
C1—N1—Pt1	118.2 (2)	C7—C8—H8B	109.2
C5—N1—Pt1	122.5 (2)	C9—C8—H8B	109.2
C14—N2—C10	118.9 (3)	H8A—C8—H8B	107.9
C14—N2—Pt1	124.0 (2)	O3—C9—O2	121.4 (3)
C10—N2—Pt1	117.0 (2)	O3—C9—C8	120.7 (3)
C7—O1—Pt1	120.0 (2)	O2—C9—C8	117.9 (3)
C9—O2—Pt1	119.0 (2)	N2—C10—C11	122.5 (4)
N1—C1—C2	122.4 (3)	N2—C10—H10	118.7
N1—C1—H1	118.8	C11—C10—H10	118.7
C2—C1—H1	118.8	C10—C11—C12	119.4 (4)
C3—C2—C1	119.0 (4)	C10—C11—H11	120.3
C3—C2—H2	120.5	C12—C11—H11	120.3
C1—C2—H2	120.5	C11—C12—C13	118.1 (4)
C4—C3—C2	118.6 (3)	C11—C12—H12	120.9
C4—C3—H3	120.7	C13—C12—H12	120.9
C2—C3—H3	120.7	C14—C13—C12	121.1 (4)
C3—C4—C5	120.5 (3)	C14—C13—H13	119.4
C3—C4—H4	119.8	C12—C13—H13	119.4
C5—C4—H4	119.8	N2—C14—C13	119.9 (4)
N1—C5—C4	120.0 (3)	N2—C14—C15	119.5 (3)
N1—C5—C6	119.0 (3)	C13—C14—C15	120.7 (4)
C4—C5—C6	121.0 (3)	C14—C15—H15A	109.5
C5—C6—H6A	109.5	C14—C15—H15B	109.5
C5—C6—H6B	109.5	H15A—C15—H15B	109.5
H6A—C6—H6B	109.5	C14—C15—H15C	109.5
C5—C6—H6C	109.5	H15A—C15—H15C	109.5
H6A—C6—H6C	109.5	H15B—C15—H15C	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10···O1^{i}	0.93	2.53	3.455 (5)	173
C6—H6B···O3^{ii}	0.96	2.55	3.422 (5)	151
C6—H6A···O1	0.96	2.55	3.254 (5)	130

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

Fig. 1


C12
C13
C11
C14
C15
C10
N2
O2
C9
O3
Pt1
C8
C1
N1
O1
C7
O4
C2
C5
C6
C3
C4