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cis-(Cyclobutane-1,1-dicarboxylato)-bis(2-methylpyridine)platinum(II)

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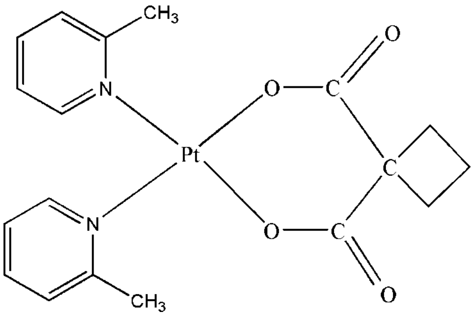
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; disorder in main residue; R factor = 0.017; wR factor = 0.044; data-to-parameter ratio = 18.2.

The asymmetric unit in the title compound, $[Pt(C_6H_6O_4)(C_6H_7N)_2]$, is composed of one-half of a molecule. The complex lies on a mirror plane which contains the Pt atom and three C atoms of the cyclobutane group, the fourth C atom being disordered with respect to the mirror plane. The Pt^{II} ion is tetracoordinated in a square-planar environment.

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

$[Pt(C_6H_6O_4)(C_6H_7N)_2]$
M_r = 523.45
Orthorhombic, *Pnma*
a = 12.7339 (7) Å
b = 14.5313 (8) Å
c = 9.7716 (6) Å
V = 1808.14 (18) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 7.79 mm^{-1}
T = 298 (2) K
0.26 × 0.22 × 0.12 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*APEX2*; Bruker, 2004)
T_{min} = 0.237, T_{max} = 0.455
14744 measured reflections
2278 independent reflections
2031 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.017
$wR(F^2)$ = 0.044
S = 1.04
2278 reflections
125 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.78 e Å^{-3}
$\Delta\rho_{min}$ = −0.66 e Å^{-3}

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: *ORTEPIII* (Burnett & Johnson, 1996) and *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: DN2231).

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supplementary materials

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cis-(Cyclobutane-1,1-dicarboxylato)bis(2-methylpyridine)platinum(II)

M.-J. Xie, Y. Yu, W.-P. Liu, S.-Q. Hou and Q.-S. Ye

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 asymmetric unit in the title compound, $C_{18}H_{20}N_2O_4Pt$,is composed of half a molecule (Fig. 1). Indeed the complex is distributed around a mirror plane which contains the Platinum and the C8, C9 and C11 atoms of the cyclobutane group, the fourth one, C10, is disordered with respect to the mirror plane. The Pt atom is coordinated in a square-pyramidal enironment (Fig. 1). The 1,1-cyclobutanedicarboxylate 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 symetry related 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. After standing in the dark for 30 min at room temperature, 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 1,1-cyclobutanedicarboxylate (1.07 g, 2.99 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 analysis.

Refinement

All H atoms were initially located in a difference Fourier map but were positioned with idealized geometry and treated as riding on their parent atoms with C—H = 0.93 Å (aromatic), 0.96 Å (methyl) and 0.97 Å (methylene) and with $U_{iso}(H)$ = $1.2U_{eq}$(aromatic, methylene) and 1.5 for methyl H atoms) or $U_{iso}(H) = 1.5U_{eq}$(methyl).

The C10 atom is statistically distributed with respect to the crystallographic mirror plane. It was then refined using the PART −1 instruction within *SHELXL97* (Sheldrick, 1997) and C—C restraints.

Figures

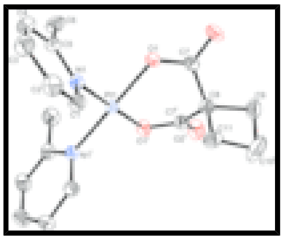

Fig. 1. Molecular view of the complex, with the atomic labeling scheme. Displacement ellipsoids are drawn at the 30% probability level. Only one component of the disordered moiety is represented. H atoms have been omitted for clarity. [Symmetry code: (i) *x*, –Y+3/2, *z*].

cis-(Cyclobutane-1,1-dicarboxylato)bis(2-methylpyridine)platinum(II)

Crystal data

$[Pt(C_6H_6O_4)(C_6H_7N)_2]$

$M_r = 523.45$

Orthorhombic, *Pnma*

Hall symbol: -P2ac2n

$a = 12.7339$ (7) Å

$b = 14.5313$ (8) Å

$c = 9.7716$ (6) Å

$V = 1808.14$ (18) Å^3

$Z = 4$

$F_{000} = 1008$

$D_x = 1.923$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Cell parameters from 2278 reflections

$\theta = 2.5$–28.3°

$\mu = 7.79$ mm^{-1}

$T = 298$ (2) K

Block, colourless

0.26 × 0.22 × 0.12 mm

Data collection

Bruker APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298(2)$ K

φ and ω scans

Absorption correction: numerical (APEX2; Bruker, 2004)

$T_{min} = 0.237$, $T_{max} = 0.455$

14744 measured reflections

2278 independent reflections

2031 reflections with $I > 2\sigma(I)$

$R_{int} = 0.028$

$\theta_{max} = 28.3$°

$\theta_{min} = 2.5$°

$h = -16\rightarrow16$

$k = -19\rightarrow19$

$l = -12\rightarrow12$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.017$

$wR(F^2) = 0.044$

$S = 1.04$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.027P)^2 + 0.1067P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.003$

2278 reflections	$\Delta\rho_{max} = 0.78$ e Å^{-3}
125 parameters	$\Delta\rho_{min} = -0.66$ e Å^{-3}
2 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 $F^2 > 2\text{sigma}(F^2)$ 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}	Occ. (<1)
Pt1	0.615174 (10)	0.7500	0.091373 (11)	0.02943 (6)	
N1	0.66661 (15)	0.64874 (13)	−0.03283 (19)	0.0326 (4)	
O1	0.56091 (14)	0.65080 (11)	0.21436 (17)	0.0401 (4)	
O2	0.50259 (19)	0.60774 (15)	0.41509 (18)	0.0605 (6)	
C1	0.76953 (19)	0.62816 (18)	−0.0318 (3)	0.0401 (6)	
H1	0.8138	0.6634	0.0227	0.048*	
C2	0.8127 (3)	0.5579 (2)	−0.1070 (3)	0.0484 (7)	
H2	0.8839	0.5442	−0.1011	0.058*	
C3	0.7478 (2)	0.50885 (18)	−0.1906 (3)	0.0547 (8)	
H3	0.7752	0.4629	−0.2464	0.066*	
C4	0.6415 (2)	0.5272 (2)	−0.1925 (3)	0.0524 (7)	
H4	0.5971	0.4929	−0.2483	0.063*	
C5	0.6010 (2)	0.5970 (2)	−0.1110 (3)	0.0401 (6)	
C6	0.4857 (2)	0.6157 (2)	−0.1055 (3)	0.0571 (8)	
H6A	0.4658	0.6300	−0.0132	0.086*	
H6B	0.4480	0.5624	−0.1360	0.086*	
H6C	0.4694	0.6669	−0.1639	0.086*	
C7	0.54870 (18)	0.66383 (16)	0.3446 (3)	0.0377 (5)	
C8	0.5967 (3)	0.7500	0.4092 (3)	0.0397 (9)	
C9	0.5995 (4)	0.7500	0.5655 (4)	0.0626 (14)	
H9A	0.5886	0.8102	0.6059	0.075*	0.50
H9B	0.5538	0.7045	0.6070	0.075*	0.50
C11	0.7208 (4)	0.7500	0.4086 (4)	0.0517 (11)	
H11A	0.7519	0.7040	0.3492	0.062*	0.50
H11B	0.7520	0.8101	0.3941	0.062*	0.50
C10	0.7137 (6)	0.7211 (7)	0.5593 (7)	0.097 (5)	0.50
H10A	0.7242	0.6557	0.5735	0.117*	0.50

H10B	0.7589	0.7567	0.6191	0.117*	0.50

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pt1	0.02763 (8)	0.03190 (8)	0.02875 (8)	0.000	0.00074 (4)	0.000
N1	0.0328 (11)	0.0361 (10)	0.0290 (10)	−0.0017 (8)	0.0011 (8)	−0.0006 (8)
O1	0.0460 (10)	0.0357 (9)	0.0385 (9)	−0.0090 (7)	0.0058 (7)	0.0003 (7)
O2	0.0720 (16)	0.0569 (13)	0.0527 (13)	−0.0192 (11)	0.0175 (10)	0.0103 (9)
C1	0.0373 (14)	0.0443 (14)	0.0389 (14)	0.0011 (10)	−0.0007 (10)	−0.0031 (11)
C2	0.0447 (16)	0.0495 (16)	0.0510 (16)	0.0106 (13)	0.0063 (12)	−0.0044 (12)
C3	0.067 (2)	0.0439 (16)	0.0532 (17)	0.0066 (14)	0.0063 (14)	−0.0108 (12)
C4	0.0647 (19)	0.0453 (16)	0.0472 (16)	−0.0095 (13)	−0.0023 (13)	−0.0129 (13)
C5	0.0444 (16)	0.0387 (15)	0.0372 (14)	−0.0077 (11)	−0.0014 (10)	−0.0014 (11)
C6	0.0415 (17)	0.068 (2)	0.062 (2)	−0.0114 (15)	−0.0108 (13)	−0.0126 (14)
C7	0.0335 (13)	0.0376 (13)	0.0420 (14)	0.0011 (10)	0.0043 (10)	0.0044 (11)
C8	0.036 (2)	0.046 (2)	0.036 (2)	0.000	0.0050 (13)	0.000
C9	0.083 (4)	0.069 (3)	0.035 (2)	0.000	−0.003 (2)	0.000
C11	0.037 (2)	0.052 (2)	0.066 (3)	0.000	−0.0118 (17)	0.000
C10	0.089 (6)	0.140 (14)	0.062 (4)	0.003 (5)	−0.032 (4)	0.023 (5)

Geometric parameters (Å, °)

Pt1—O1^{i}	1.9999 (15)	C6—H6A	0.9600
Pt1—O1	1.9999 (15)	C6—H6B	0.9600
Pt1—N1	2.0167 (19)	C6—H6C	0.9600
Pt1—N1^{i}	2.0167 (19)	C7—C8	1.530 (3)
N1—C1	1.344 (3)	C8—C9	1.528 (5)
N1—C5	1.359 (3)	C8—C7^{i}	1.530 (3)
O1—C7	1.296 (3)	C8—C11	1.580 (6)
O2—C7	1.218 (3)	C9—C10^{i}	1.514 (8)
C1—C2	1.373 (4)	C9—C10	1.514 (8)
C1—H1	0.9300	C9—H9A	0.9700
C2—C3	1.363 (4)	C9—H9B	0.9700
C2—H2	0.9300	C11—C10^{i}	1.533 (7)
C3—C4	1.380 (4)	C11—C10	1.533 (7)
C3—H3	0.9300	C11—H11A	0.9700
C4—C5	1.389 (4)	C11—H11B	0.9700
C4—H4	0.9300	C10—H10A	0.9700
C5—C6	1.493 (4)	C10—H10B	0.9700
O1^{i}—Pt1—O1	92.24 (9)	O1—C7—C8	118.5 (2)
O1^{i}—Pt1—N1	178.71 (7)	C9—C8—C7	114.97 (19)
O1—Pt1—N1	87.02 (8)	C9—C8—C7^{i}	114.97 (19)
O1^{i}—Pt1—N1^{i}	87.02 (8)	C7—C8—C7^{i}	109.8 (3)
O1—Pt1—N1^{i}	178.71 (7)	C9—C8—C11	88.9 (3)
N1—Pt1—N1^{i}	93.71 (11)	C7—C8—C11	113.47 (19)

C1—N1—C5	118.7 (2)	C7^{i}—C8—C11	113.47 (19)
C1—N1—Pt1	118.33 (16)	C10^{i}—C9—C10	32.2 (8)
C5—N1—Pt1	122.83 (17)	C10^{i}—C9—C8	89.0 (4)
C7—O1—Pt1	121.76 (15)	C10—C9—C8	89.0 (4)
N1—C1—C2	123.5 (3)	C10^{i}—C9—H9A	84.5
N1—C1—H1	118.2	C10—C9—H9A	113.8
C2—C1—H1	118.2	C8—C9—H9A	113.8
C3—C2—C1	117.8 (3)	C10^{i}—C9—H9B	141.4
C3—C2—H2	121.1	C10—C9—H9B	113.8
C1—C2—H2	121.1	C8—C9—H9B	113.8
C2—C3—C4	120.2 (3)	H9A—C9—H9B	111.0
C2—C3—H3	119.9	C10^{i}—C11—C10	31.8 (8)
C4—C3—H3	119.9	C10^{i}—C11—C8	86.4 (4)
C3—C4—C5	119.8 (3)	C10—C11—C8	86.4 (4)
C3—C4—H4	120.1	C10^{i}—C11—H11A	142.0
C5—C4—H4	120.1	C10—C11—H11A	114.2
N1—C5—C4	119.9 (3)	C8—C11—H11A	114.2
N1—C5—C6	118.9 (2)	C10^{i}—C11—H11B	85.3
C4—C5—C6	121.3 (3)	C10—C11—H11B	114.2
C5—C6—H6A	109.5	C8—C11—H11B	114.2
C5—C6—H6B	109.5	H11A—C11—H11B	111.4
H6A—C6—H6B	109.5	C9—C10—C11	91.1 (4)
C5—C6—H6C	109.5	C9—C10—H10A	113.4
H6A—C6—H6C	109.5	C11—C10—H10A	113.4
H6B—C6—H6C	109.5	C9—C10—H10B	113.4
O2—C7—O1	121.0 (2)	C11—C10—H10B	113.4
O2—C7—C8	120.4 (2)	H10A—C10—H10B	110.7

Symmetry codes: (i) x, $-y+3/2$, z.

Fig. 1