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{2,6-Bis[(dimethylamino-κ*N*)methyl]-4-{[(2,5-dioxo-1-pyrrolidinyl)oxy]carbonyl}phenyl-κC^1}(diiodine)iodidoplatinum(II) dichloromethane hemisolvate

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.006 Å
Disorder in main residue
R factor = 0.024
wR factor = 0.054
Data-to-parameter ratio = 22.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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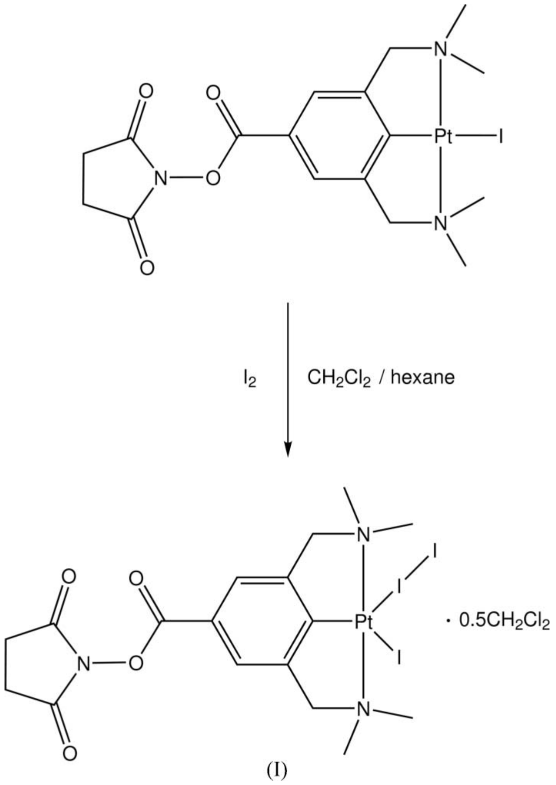
In the title compound $[Pt(C_{17}H_{22}N_3O_4)I(I_2)]\cdot 0.5CH_2Cl_2$, the coordination environment of the Pt^{II} centre is distorted square-pyramidal and is defined by two neutral N atoms and an anionic C atom from the mono-anionic ligand, an iodide anion, and an additional η^1-coordinated I_2 molecule. Intermolecular C—H⋯O contacts result in a dimeric structure. The asymmetric unit of (I) consists of two independent $[PtI(C_{17}H_{22}N_3O_4)(I_2)]$ molecules and a CH_2Cl_2 solvent molecule

Comment

The study of the interaction of simple diatomic molecules with d^8 transition metals is crucial for a better understanding of the mechanism of oxidative addition and reductive elimination reactions. Therefore, we report here the crystal structure of the title compound, (I), as a product of the addition of I_2 to the pincer Pt^{II} complex 3,5-bis-[(dimethylamino)methyl]-4-[iodidoplatino(II)]benzoic acid *N*-hydroxysuccinimide ester.



(I)

The asymmetric unit of (I) consists of two independent $[PtI(C_{17}H_{22}N_3O_4)(I_2)]$ molecules and a CH_2Cl_2 solvent molecule (Fig. 1). One molecule (containing Pt2) shows disorder

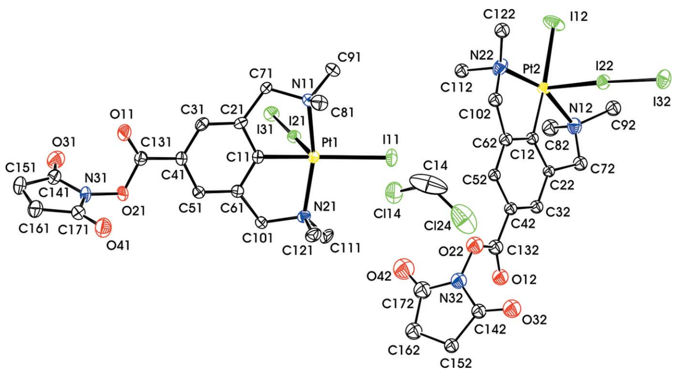


Figure 1
The asymmetric unit of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity. Only one of the two disordered components is shown for molecule 2.

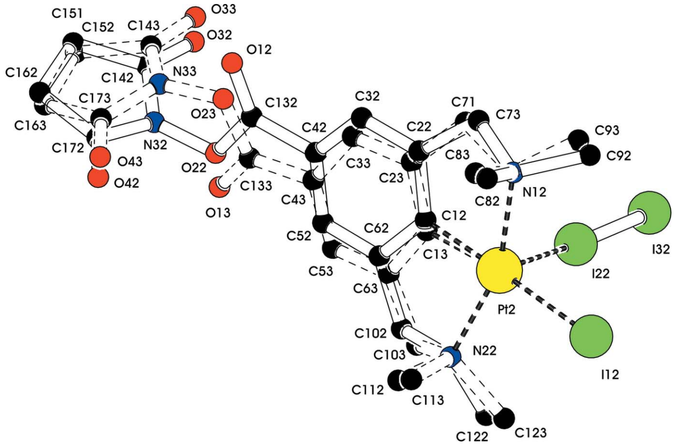


Figure 2
The disorder in the structure of the second molecule (Pt2). H atoms have been omitted for clarity. The minor disorder component is drawn with dashed bonds.

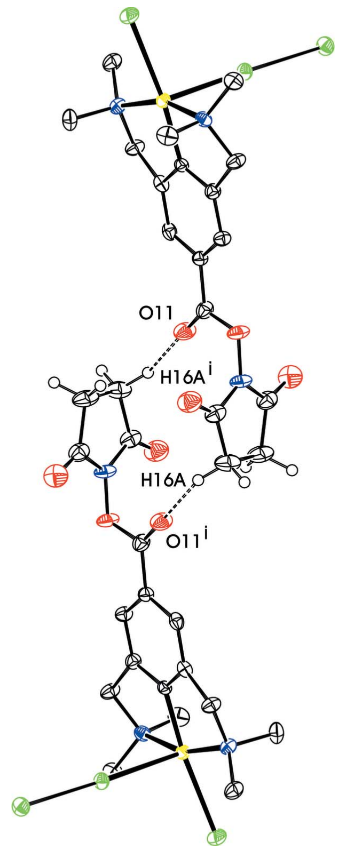


Figure 3
Hydrogen-bond interactions in (I). The C—H···O contacts are shown as dashed lines. [Symmetry code: (i) 1 − x, −y, 1 − z.]

about the molecular axis over all atomic positions, except for the heavy atoms and the N atoms bonded to Pt2 (Fig. 2). In the following, we discuss only molecule 1, containing Pt1. Geometric parameters for the Pt2 coordination environment are very similar to those for Pt1 and are given in Table 1.

The coordination environment of the Pt^{II} centre is defined by the mono-anionic ligand 2,6-bis[(dimethylamino)-methyl]-4-{[(2,5-dioxo-1-pyrrolidinyl)oxy]carbonyl}phenyl ($C_{17}H_{22}N_3O_4$), an iodide anion and an I_2 molecule. The ligand is coordinated in a tridentate manner to the metal *via* the anionic C atom [Pt1—C11 = 1.942 (4) Å] and the two neutral amine N atoms [Pt1—N11 = 2.104 (4) Å and Pt1—N12 = 2.103 (4) Å]. The iodide anion is coordinated *trans* to atom C11 [C11—Pt1—I11 = 173.70 (12)°], with a Pt1—I11 distance of 2.72985 (19) Å. The I_2 molecule is end-on η^1-coordinated to the Pt^{II} centre [Pt1—I21 = 2.8260 (3) Å] and is part of a linear Pt1—I21—I23 arrangement [177.248 (14)°], with I21—I31 = 2.8379 (4) Å. These distances compare well with those observed in similar Pt^{II} complexes, such as [PtI(NCN)(η^1-I_2) {NCN = 2,6-bis[(dimethylamino)methyl]phenyl, $C_{12}H_{19}N_2$} reported by Gossage *et al.* (1999).

The small bite angles of the chelate ligand [C11—Pt1—N11 = 82.09 (15) and C11—Pt1—N21 = 82.15 (15)°] result in a distorted square-pyramidal geometry of the central Pt^{II} atom. This can be quantified as 91.8% on the pathway from trigonal bipyramid to square pyramid (Holmes, 1984). The sum of the *cis* angles is 360°, although they deviate by up to 8° (for N21—Pt1—I11) from the ideal value of 90°. The largest deviation for the *trans* angles is 19° (N21—Pt1—N11) from the ideal value of 180°. A conformational analysis of ring puckering results in coefficients of 98.3° for the sine form of the PtC$_3$N11 chelate ring, and of 87.6° for the sine form of the PtC$_3$N21 chelate ring (Evans & Boeyens, 1989). Therefore, the two five-membered PtC$_3$N chelate rings are best described as twist conformations and are puckered in the same direction, with the N atoms mutually *cis.* The torsion angles Pt1—N11—C71—C21 = 26.1 (4)° and Pt1—N21—C101—C61 = −28.6 (4)° result in a local non-crystallographic C_s symmetry.

The *N*-oxosuccinimide groups connect two molecules into a dimer structure *via* an intermolecular C—H···O contact, with H16A···O11^i = 2.46Å [symmetry code: (i) 1 − x, −y, 1 − z] (Table 2 and Fig. 3).

Experimental

The reaction of 3,5-bis-[(dimethylamino)methyl]-4-[iodidoplatino(II)]benzoic acid, prepared as reported by Suijkerbuijk *et al.* (2002), with I_2 in CH_2Cl_2–hexane (1:1) results in the title compound,

m1224 von Chrzanowski *et al.* • [Pt($C_{17}H_{22}N_3O_4$)I(I_2)]·0.5CH_2Cl_2 *Acta Cryst.* (2007). E**63**, m1223–m1225

(I). Dark-red crystals suitable for X-ray data collection were obtained after recrystallization from CH_2Cl_2–hexane (1:1).

Crystal data

$[Pt(C_{17}H_{22}N_3O_4)I(I_2)]\cdot 0.5CH_2Cl_2$
$M_r = 950.63$
Triclinic, $P\overline{1}$
$a = 8.7012$ (1) Å
$b = 15.1862$ (2) Å
$c = 19.8391$ (4) Å
$\alpha = 99.2137$ (14)°
$\beta = 90.7250$ (15)°
$\gamma = 103.7589$ (13)°
$V = 2509.90$ (7) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 9.41$ mm^{-1}
$T = 150$ (2) K
0.42 × 0.09 × 0.06 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: analytical (de Meulenaer & Tompa, 1965)
$T_{min} = 0.14$, $T_{max} = 0.63$
35716 measured reflections
11455 independent reflections
8859 reflections with $I > 2\sigma(I)$
$R_{int} = 0.036$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.024$
$wR(F^2) = 0.054$
$S = 0.94$
11455 reflections
511 parameters
162 restraints
H-atom parameters constrained
$\Delta\rho_{max} = 3.03$ e Å^{-3}
$\Delta\rho_{min} = -3.35$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Pt1—C11	1.942 (4)	Pt2—C12	1.966 (8)
Pt1—N21	2.103 (4)	Pt2—N22	2.098 (4)
Pt1—N11	2.104 (4)	Pt2—N12	2.103 (4)
Pt1—I11	2.6862 (3)	Pt2—I12	2.6827 (4)
Pt1—I21	2.8260 (3)	Pt2—I22	2.8198 (3)
I21—I31	2.8379 (4)	I22—I32	2.8472 (4)
Pt2—C13	1.908 (9)		
C11—Pt1—N21	82.15 (15)	C13—Pt2—N12	85.7 (3)
C11—Pt1—N11	82.09 (15)	C12—Pt2—N12	79.3 (3)
N21—Pt1—N11	161.06 (14)	N22—Pt2—N12	160.14 (16)
C11—Pt1—I11	173.70 (12)	C13—Pt2—I12	173.3 (4)
N21—Pt1—I11	98.26 (10)	C12—Pt2—I12	172.6 (4)
N11—Pt1—I11	96.26 (10)	N22—Pt2—I12	97.20 (12)
C11—Pt1—I21	84.54 (12)	N12—Pt2—I12	97.75 (11)
N21—Pt1—I21	94.22 (10)	C13—Pt2—I22	83.7 (5)
N11—Pt1—I21	94.68 (10)	C12—Pt2—I22	85.4 (4)
I11—Pt1—I21	101.675 (11)	N22—Pt2—I22	94.64 (11)
Pt1—I21—I31	177.248 (14)	N12—Pt2—I22	95.10 (10)
C13—Pt2—C12	6.5 (4)	I12—Pt2—I22	101.569 (11)
C13—Pt2—N22	78.2 (3)	Pt2—I22—I32	177.533 (15)
C12—Pt2—N22	84.3 (3)		
Pt1—N11—C71—C21	26.1 (4)	Pt2—N22—C102—C62	−28.4 (7)
Pt1—N21—C101—C61	−28.6 (4)	Pt2—N12—C73—C23	20.5 (11)
Pt2—N12—C72—C22	25.9 (9)	Pt2—N22—C103—C63	−35.1 (9)

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

D—H···A	D—H	H···A	D···A	D—H···A
C161—H16A···O11^{i}	0.99	2.46	3.296 (7)	142

Symmetry code: (i) $1 - x, -y, 1 - z$.

All H atoms were introduced in geometrically idealized positions, with C—H = 0.95–0.99 Å and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C) for CH and CH_2 H atoms, or 1.5U_{eq}(C) for methyl H atoms. Disordered C, N and O atoms were refined isotropically. The highest peak and deepest hole in the residual electron density are both 0.8 Å from atom I12. All C and O atoms and atom N32 in the second molecule (Pt2) are disordered about the long axis of the molecule. The occupancy factors for these positions were refined to 0.566 (6) and 0.434 (6). Atoms C41/C131/O11/O21/N31, C42/C132/O12/O22/N32 and C42/C133/O13/O23/N33 were restrained to lie on respective common planes.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *DIRAX* (Duisenberg, 1992); data reduction: *EVALCCD* (Duisenberg *et al.*, 2003) and *SORTAV* (Blessing, 1987); program(s) used to solve structure: *DIRDIF97* (Beurskens *et al.*, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: manual editing of the *SHELXL97* output.

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