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(4*S*,5*S*)-2-(4-Chlorophenyl)-1,3-dioxolane-4,5-dicarboxamide

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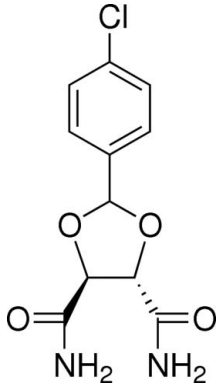
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.048; wR factor = 0.122; data-to-parameter ratio = 11.8.

The title compound, $C_{11}H_{11}ClN_2O_4$, is an important intermediate for the preparation of platinum anticancer drugs. The dioxolane ring adopts a twist conformation with an equatorially attached chlorophenyl substituent. In the crystal structure, molecules are linked into a two-dimensional network parallel to (001) by N—H···O and C—H···O hydrogen bonds.

Related literature

For bond-length data, see: Allen *et al.* (1987). For general background to platinum anticancer drugs, see: Kim *et al.* (1994); Pandey *et al.* (1997).



Experimental

Crystal data

$C_{11}H_{11}ClN_2O_4$
M_r = 270.67
Monoclinic, $P2_1$
a = 9.2780 (19) Å
b = 4.7600 (10) Å
c = 13.245 (3) Å
β = 93.15 (3)°
V = 584.1 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.34 mm^{-1}
T = 293 K
0.20 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.936, T_{max} = 0.967
2248 measured reflections
2113 independent reflections
1694 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.122
S = 1.07
2113 reflections
179 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Absolute structure: Flack (1983), 916 Friedel pairs
Flack parameter: 0.07 (14)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O3^{i}	0.79 (4)	2.20 (4)	2.979 (5)	166 (4)
N2—H2A···O3^{ii}	0.87 (4)	2.42 (3)	3.173 (5)	145 (3)
N2—H2B···O4^{iii}	0.81 (6)	2.26 (6)	3.012 (4)	155 (4)
C9—H9···O4^{iii}	0.98	2.31	3.075 (4)	135

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

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1989); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); 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: CI2828).

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supplementary materials

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(4*S*,5*S*)-2-(4-Chlorophenyl)-1,3-dioxolane-4,5-dicarboxamide

D.-C. Wang, J. Bai, W. Xu, T. Gai and H.-Q. Liu

Comment

Platinum antitumor drug is one kind of the most effective anticancer agents currently available. (2*S*,3S)-Diethyl 2,3-*O*-alkyl-tartrate analogues are starting materials for the syntheses of platinum complexes with antitumor activity (Kim *et al.*,1994), and are also important intermediates in organic syntheses (Pandey *et al.*, 1997). As part of our studies on the syntheses and characterizations of these compounds, we have synthesized the title compound and reported herein its crystal structure.

In the molecule of the title compound (Fig 1), the bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. The dioxolane ring adopts a twist conformation and the chlorophenyl unit is equatorially attached.

In the crystal structure, N—H···O and C—H···O intermolecular hydrogen bonds (Table 1) link the molecules to form a two-dimensional network (Fig. 2) parallel to the (001).

Experimental

4-Chlorobenzaldehyde (278 mg, 1.98 mmol), (2*S*,3S)-diethyltartrate (378 mg,1.84 mmol) and cyclohexane (10 ml) were placed in a round-bottomed flask, and 4-methylbenzenesulfonic acid (30 mg) was added. The flask was fitted with a water-distributor. The mixture was heated under reflux for 3 h. The reaction mixture was cooled to room temperature, and then transfered into a separatory funnel, washed with water (200 ml) and extracted with acetate (200 ml). The organic phase was distilled under pressure, and the residual was dissolved in anhydrous ethanol (50 ml). Then, a current of dry ammonia was passed through the reaction mixture at room temperature for about 4 h. The reaction mixture was then added dropwise to a vigorously stirred water (600 ml). The resulting colourless precipitate was obtained by filtration and dried *in vacuo* (Kim *et al.*, 1994). Single crystals suitable for X-ray analysis were obtained by slow evaporation of a methanol solution after two weeks.

Refinement

H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and included in the refinement in riding motion approximation, with U_{iso}(H) = 1.2 or 1.5U_{eq} of the carrier atom.

Figures

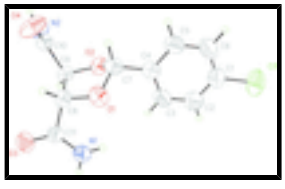

Fig. 1. The molecular structure of the title compound, showing the atom-numbering scheme and displacement ellipsoids at the 30% probability level.

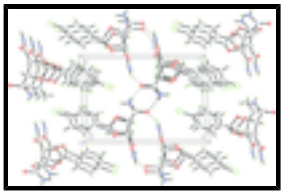

Fig. 2. A packing diagram of the title compound. Hydrogen bonds are shown as dashed lines.

(4*S*,5*S*)-2-(4-Chlorophenyl)-1,3-dioxolane-4,5-dicarboxamide

Crystal data

$C_{11}H_{11}ClN_2O_4$	$F_{000} = 280$
$M_r = 270.67$	$D_x = 1.539$ Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: P 2yb	Cell parameters from 25 reflections
$a = 9.2780$ (19) Å	$\theta = 9$–13º
$b = 4.7600$ (10) Å	$\mu = 0.34$ mm^{-1}
$c = 13.245$ (3) Å	$T = 293$ K
$\beta = 93.15$ (3)º	Block, colourless
$V = 584.1$ (2) Å^3	0.20 × 0.20 × 0.10 mm
$Z = 2$	

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{int} = 0.027$
Radiation source: fine-focus sealed tube	$\theta_{max} = 25.3$º
Monochromator: graphite	$\theta_{min} = 1.5$º
$T = 293$ K	$h = 0 \rightarrow 11$
$\omega/2\theta$ scans	$k = -5 \rightarrow 5$
Absorption correction: ψ scan (North *et al.*, 1968)	$l = -15 \rightarrow 15$
$T_{min} = 0.936$, $T_{max} = 0.967$	3 standard reflections
2248 measured reflections	every 200 reflections
2113 independent reflections	intensity decay: 1%
1694 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.048$	$w = 1/[\sigma^2(F_o^2) + (0.0601P)^2 + 0.0558P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.122$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.07$	$\Delta\rho_{max} = 0.20$ e Å^{-3}
2113 reflections	$\Delta\rho_{min} = -0.26$ e Å^{-3}

179 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack (1983), 916 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.07 (14)
Secondary atom site location: difference Fourier map	

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}
Cl1	0.35368 (14)	−0.0236 (4)	−0.21514 (8)	0.0925 (5)
O1	0.2854 (2)	0.2859 (5)	0.26692 (18)	0.0475 (6)
O2	0.0917 (2)	0.0071 (5)	0.24181 (16)	0.0461 (6)
O3	0.3515 (3)	0.0218 (6)	0.51614 (17)	0.0568 (7)
O4	−0.0323 (3)	0.4727 (5)	0.3827 (2)	0.0657 (8)
N1	0.4522 (4)	−0.0804 (9)	0.3700 (3)	0.0597 (10)
H1A	0.516 (4)	−0.168 (8)	0.397 (3)	0.038 (10)*
H1B	0.451 (4)	−0.069 (10)	0.305 (3)	0.064 (13)*
N2	−0.1472 (4)	0.0634 (7)	0.3925 (3)	0.0458 (7)
H2A	−0.230 (4)	0.139 (7)	0.403 (2)	0.034 (9)*
H2B	−0.140 (5)	−0.105 (12)	0.399 (3)	0.071 (15)*
C1	0.2948 (4)	0.0470 (10)	−0.0955 (3)	0.0573 (10)
C2	0.3508 (5)	−0.1037 (10)	−0.0134 (3)	0.0675 (12)
H2	0.4179	−0.2455	−0.0224	0.081*
C3	0.3072 (4)	−0.0438 (10)	0.0813 (3)	0.0618 (10)
H3	0.3440	−0.1476	0.1363	0.074*
C4	0.2085 (4)	0.1701 (7)	0.0964 (3)	0.0467 (9)
C5	0.1527 (4)	0.3126 (9)	0.0133 (3)	0.0614 (11)
H5	0.0852	0.4541	0.0216	0.074*
C6	0.1949 (5)	0.2500 (11)	−0.0830 (3)	0.0701 (12)
H6	0.1549	0.3468	−0.1387	0.084*
C7	0.1632 (4)	0.2384 (7)	0.1998 (3)	0.0471 (8)
H7	0.1002	0.4038	0.1976	0.057*
C8	0.2436 (3)	0.2201 (7)	0.3666 (2)	0.0392 (7)
H8	0.2264	0.3935	0.4039	0.047*
C9	0.1005 (3)	0.0548 (7)	0.3490 (2)	0.0378 (7)
H9	0.1064	−0.1248	0.3852	0.045*
C10	−0.0327 (3)	0.2166 (7)	0.3777 (3)	0.0395 (8)

C11	0.3560 (3)	0.0449 (8)	0.4236 (2)	0.0423 (8)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.1056 (9)	0.1139 (10)	0.0597 (6)	−0.0231 (10)	0.0197 (6)	−0.0196 (7)
O1	0.0522 (13)	0.0425 (14)	0.0473 (13)	−0.0157 (11)	−0.0015 (11)	0.0055 (11)
O2	0.0474 (12)	0.0375 (13)	0.0539 (13)	−0.0122 (12)	0.0066 (10)	−0.0116 (12)
O3	0.0565 (14)	0.0719 (19)	0.0423 (13)	−0.0098 (15)	0.0051 (11)	0.0030 (14)
O4	0.0582 (15)	0.0206 (12)	0.120 (2)	0.0031 (12)	0.0207 (14)	−0.0055 (15)
N1	0.055 (2)	0.074 (3)	0.050 (2)	0.0199 (19)	−0.0040 (17)	−0.0013 (18)
N2	0.0402 (17)	0.0289 (17)	0.069 (2)	0.0016 (14)	0.0126 (14)	0.0046 (14)
C1	0.057 (2)	0.064 (3)	0.051 (2)	−0.019 (2)	0.0029 (17)	−0.008 (2)
C2	0.071 (3)	0.065 (3)	0.066 (3)	0.011 (2)	−0.003 (2)	−0.019 (2)
C3	0.078 (3)	0.058 (2)	0.048 (2)	0.012 (2)	−0.0093 (18)	−0.003 (2)
C4	0.049 (2)	0.0377 (19)	0.053 (2)	−0.0036 (16)	−0.0039 (17)	−0.0016 (16)
C5	0.057 (2)	0.061 (3)	0.066 (3)	0.009 (2)	0.0015 (19)	0.012 (2)
C6	0.073 (3)	0.082 (3)	0.055 (2)	−0.005 (3)	−0.006 (2)	0.016 (2)
C7	0.049 (2)	0.0352 (18)	0.057 (2)	0.0005 (17)	−0.0044 (17)	0.0031 (16)
C8	0.0428 (18)	0.0303 (17)	0.0450 (18)	−0.0061 (15)	0.0073 (15)	−0.0031 (14)
C9	0.0388 (16)	0.0245 (15)	0.0500 (19)	−0.0048 (14)	0.0021 (14)	−0.0002 (13)
C10	0.0395 (17)	0.0312 (19)	0.0479 (19)	−0.0016 (15)	0.0023 (15)	0.0005 (15)
C11	0.0343 (16)	0.045 (2)	0.0475 (19)	−0.0100 (16)	0.0032 (14)	−0.0074 (17)

Geometric parameters (Å, °)

Cl1—C1	1.738 (4)	C2—C3	1.370 (5)
O1—C7	1.420 (4)	C2—H2	0.93
O1—C8	1.431 (4)	C3—C4	1.391 (5)
O2—C7	1.415 (4)	C3—H3	0.93
O2—C9	1.436 (4)	C4—C5	1.370 (5)
O3—C11	1.233 (4)	C4—C7	1.490 (5)
O4—C10	1.221 (4)	C5—C6	1.387 (6)
N1—C11	1.313 (5)	C5—H5	0.93
N1—H1A	0.79 (4)	C6—H6	0.93
N1—H1B	0.86 (4)	C7—H7	0.98
N2—C10	1.312 (5)	C8—C11	1.506 (5)
N2—H2A	0.86 (3)	C8—C9	1.550 (4)
N2—H2B	0.81 (6)	C8—H8	0.98
C1—C6	1.355 (6)	C9—C10	1.522 (4)
C1—C2	1.379 (6)	C9—H9	0.98
C7—O1—C8	107.2 (2)	C5—C6—H6	120.2
C7—O2—C9	105.3 (2)	O2—C7—O1	104.7 (3)
C11—N1—H1A	120 (3)	O2—C7—C4	110.7 (3)
C11—N1—H1B	123 (3)	O1—C7—C4	110.8 (3)
H1A—N1—H1B	117 (4)	O2—C7—H7	110.2
C10—N2—H2A	122 (2)	O1—C7—H7	110.2
C10—N2—H2B	121 (3)	C4—C7—H7	110.2

H2A—N2—H2B	117 (4)	O1—C8—C11	111.6 (3)
C6—C1—C2	120.4 (4)	O1—C8—C9	104.2 (2)
C6—C1—Cl1	120.0 (3)	C11—C8—C9	111.0 (3)
C2—C1—Cl1	119.6 (3)	O1—C8—H8	109.9
C3—C2—C1	119.7 (4)	C11—C8—H8	109.9
C3—C2—H2	120.1	C9—C8—H8	109.9
C1—C2—H2	120.1	O2—C9—C10	108.9 (2)
C2—C3—C4	121.0 (4)	O2—C9—C8	103.3 (2)
C2—C3—H3	119.5	C10—C9—C8	114.0 (3)
C4—C3—H3	119.5	O2—C9—H9	110.1
C5—C4—C3	118.0 (4)	C10—C9—H9	110.1
C5—C4—C7	121.2 (3)	C8—C9—H9	110.1
C3—C4—C7	120.9 (3)	O4—C10—N2	123.2 (3)
C4—C5—C6	121.3 (4)	O4—C10—C9	121.2 (3)
C4—C5—H5	119.4	N2—C10—C9	115.5 (3)
C6—C5—H5	119.4	O3—C11—N1	123.9 (3)
C1—C6—C5	119.7 (4)	O3—C11—C8	119.2 (3)
C1—C6—H6	120.2	N1—C11—C8	116.9 (3)
C6—C1—C2—C3	−1.3 (6)	C3—C4—C7—O1	52.9 (5)
Cl1—C1—C2—C3	178.4 (3)	C7—O1—C8—C11	134.7 (3)
C1—C2—C3—C4	−0.9 (7)	C7—O1—C8—C9	14.8 (3)
C2—C3—C4—C5	2.1 (6)	C7—O2—C9—C10	91.5 (3)
C2—C3—C4—C7	−178.9 (4)	C7—O2—C9—C8	−30.0 (3)
C3—C4—C5—C6	−1.2 (6)	O1—C8—C9—O2	9.3 (3)
C7—C4—C5—C6	179.8 (4)	C11—C8—C9—O2	−111.0 (3)
C2—C1—C6—C5	2.2 (6)	O1—C8—C9—C10	−108.7 (3)
Cl1—C1—C6—C5	−177.5 (3)	C11—C8—C9—C10	130.9 (3)
C4—C5—C6—C1	−1.0 (7)	O2—C9—C10—O4	−93.1 (4)
C9—O2—C7—O1	40.4 (3)	C8—C9—C10—O4	21.7 (5)
C9—O2—C7—C4	159.7 (3)	O2—C9—C10—N2	84.4 (4)
C8—O1—C7—O2	−34.2 (3)	C8—C9—C10—N2	−160.8 (3)
C8—O1—C7—C4	−153.5 (3)	O1—C8—C11—O3	164.5 (3)
C5—C4—C7—O2	116.2 (4)	C9—C8—C11—O3	−79.7 (4)
C3—C4—C7—O2	−62.8 (4)	O1—C8—C11—N1	−17.0 (4)
C5—C4—C7—O1	−128.1 (4)	C9—C8—C11—N1	98.8 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···O3^{i}	0.79 (4)	2.20 (4)	2.979 (5)	166 (4)
N2—H2A···O3^{ii}	0.87 (4)	2.42 (3)	3.173 (5)	145 (3)
N2—H2B···O4^{iii}	0.81 (6)	2.26 (6)	3.012 (4)	155 (4)
C9—H9···O4^{iii}	0.98	2.31	3.075 (4)	135

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

Fig. 1

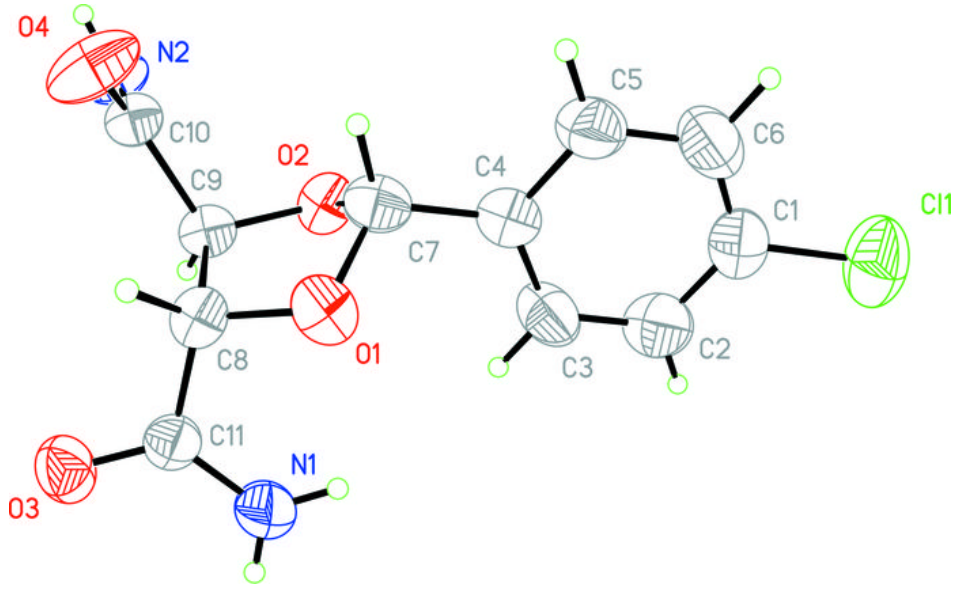

Fig. 2