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

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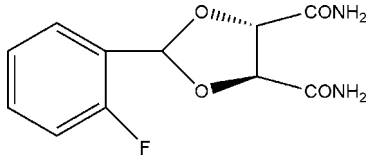
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.009 Å; R factor = 0.060; wR factor = 0.125; data-to-parameter ratio = 7.9.

In the molecule of the title compound, $C_{11}H_{11}FN_2O_4$, the five-membered ring adopts an envelope conformation. An intramolecular N—H···F hydrogen bond occurs. In the crystal structure, intermolecular N—H···O hydrogen bonds link the molecules.

Related literature

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



Experimental

Crystal data

$C_{11}H_{11}FN_2O_4$
M_r = 254.22
Orthorhombic, $P2_12_12_1$
a = 4.8760 (5) Å
b = 9.1290 (7) Å
c = 24.8160 (9) Å
V = 1104.63 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 294 (2) K
0.40 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.978, T_{max} = 0.987
2157 measured reflections
1301 independent reflections
898 reflections with $I > 2\sigma(I)$
R_{int} = 0.072
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.125
S = 1.00
1301 reflections
164 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O4^{i}	0.86	2.32	3.089 (4)	149
N1—H1B···O3^{ii}	0.86	2.37	3.164 (4)	153
N2—H2A···O4^{iii}	0.86	2.09	2.944 (5)	172
N2—H2B···F1	0.86	2.31	3.130 (4)	160

Symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{1}{2}, -z+1$; (ii) $x-1, y, z$; (iii) $x-\frac{1}{2}, -y+\frac{3}{2}, -z+1$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software*; 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: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2584).

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Xin-Hua Li, De-Cai Wang, Bo-Nian Liu and Wei Xu

S1. Comment

Antitumor platinum drug is one kind of the most effective anticancer agents currently available. (2*S*,3S)-Diethyl 2,3-*O*-alkyltartrate 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 report herein the crystal structure of the title compound.

In the molecule of the title compound (Fig. 1) the bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. The five-membered ring adopts envelope conformation with C7 atom displaced by 0.557 (3) Å from the plane of the other ring atoms. The intramolecular N-H···F hydrogen bond (Table 1) results in the formation of a nine-membered ring: (F1/C1/C6/C7/O2/C9/C11/N2/H2B) having twisted conformation.

In the crystal structure, intermolecular N-H···O hydrogen bonds (Table 1) link the molecules, in which they may be effective in the stabilization of the structure.

S2. Experimental

For the preparatrion of the title compound, a mixture of 2-fluorbenzaldehyde (302 mg, 2.43 mmol), (2*S*,3S)-diethyl-tartrate (500 mg, 2.43 mmol), anhydrous copper sulfate (776 mg, 2.86 mmol) and one drop of methanesulfonic acid in anhydrous toluen (8 ml) was stirred at room temperature for 12 h. Anhydrous potassium carbonate (40 mg) was added to the reaction mixture, which was then stirred for a further 20 min. The resulting colorless precipitate was obtained by evaporation and dried in vacuo (yield; 87%). The obtained colorless product (10 mmol) was dissolved in anhydrous ethanol (50 ml), then a current of dry ammonia, dried with calcium chloride passed into the reaction mixture at room temperature for about 6 h. The reaction mixture was evaporated to dryness. Pure compound was obtained by crystallization from dichloromethane. Crystals suitable for X-ray analysis were obatined by slow evaporation of an ethanol solution after one week.

S3. Refinement

H atoms were positioned geometrically, with N-H = 0.86 Å (for NH_2) and C-H = 0.93 and 0.98 Å for aromatic and methine H, respectively, and constrained to ride on their parent atoms with $U_{iso}(H) = 1.2U_{eq}(C,N)$.

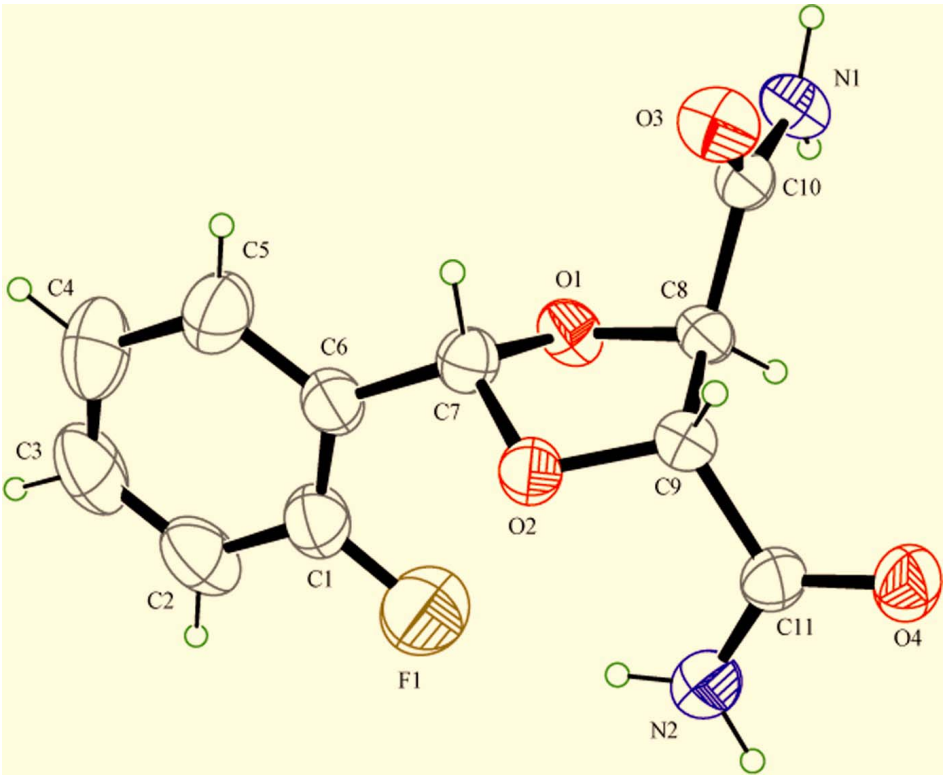

Figure 1

The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

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

Crystal data

$C_{11}H_{11}FN_2O_4$
$M_r = 254.22$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 4.8760\ (5)$ Å
$b = 9.1290\ (7)$ Å
$c = 24.8160\ (9)$ Å
$V = 1104.63\ (15)$ Å^3
$Z = 4$
$F(000) = 528$
$D_x = 1.529$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9$–12°
$\mu = 0.13$ mm^{-1}
$T = 294$ K
Block, colorless
0.40 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.978$, $T_{max} = 0.987$
2157 measured reflections
1301 independent reflections
898 reflections with $I > 2\sigma(I)$
$R_{int} = 0.072$
$\theta_{max} = 26.0°$, $\theta_{min} = 1.6°$
$h = -5\rightarrow5$
$k = 0\rightarrow11$
$l = 0\rightarrow30$
3 standard reflections every 120 min
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.125$
$S = 1.00$
1301 reflections
164 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
$w = 1/[\sigma^2(F_o^2) + (0.002P)^2 + 1.775P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.023 (4)

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}
O1	0.6898 (8)	0.3384 (4)	0.36430 (13)	0.0497 (10)
O2	0.9373 (9)	0.5438 (3)	0.37339 (12)	0.0489 (10)
O3	1.1580 (8)	0.2201 (5)	0.44108 (17)	0.0576 (11)
O4	0.7161 (10)	0.6057 (4)	0.50750 (13)	0.0599 (12)
N1	0.7627 (11)	0.0954 (4)	0.44684 (15)	0.0513 (12)
H1A	0.8450	0.0144	0.4540	0.062*
H1B	0.5866	0.0979	0.4449	0.062*
N2	0.5605 (12)	0.6946 (5)	0.42850 (18)	0.0681 (16)
H2A	0.4529	0.7561	0.4441	0.082*
H2B	0.5674	0.6905	0.3939	0.082*
F1	0.4814 (11)	0.6150 (5)	0.30682 (15)	0.1054 (16)
C1	0.6335 (15)	0.5470 (7)	0.2681 (2)	0.0633 (18)
C2	0.572 (2)	0.5797 (8)	0.2158 (3)	0.083 (2)
H2	0.4353	0.6472	0.2076	0.099*
C3	0.715 (2)	0.5109 (8)	0.1758 (3)	0.088 (3)
H3	0.6700	0.5274	0.1399	0.105*
C4	0.9265 (19)	0.4170 (9)	0.1887 (2)	0.095 (3)
H4	1.0286	0.3731	0.1615	0.114*
C5	0.9875 (19)	0.3878 (8)	0.2426 (2)	0.076 (2)
H5	1.1281	0.3230	0.2511	0.092*
C6	0.8410 (14)	0.4542 (6)	0.2832 (2)	0.0539 (16)
C7	0.9126 (13)	0.4196 (6)	0.34082 (18)	0.0466 (13)
H7	1.0818	0.3617	0.3423	0.056*

C8	0.7392 (14)	0.3487 (5)	0.42137 (18)	0.0461 (14)
H8	0.5644	0.3507	0.4409	0.055*
C9	0.8871 (13)	0.4968 (5)	0.42764 (19)	0.0470 (14)
H9	1.0617	0.4826	0.4465	0.056*
C10	0.9067 (11)	0.2163 (6)	0.4389 (2)	0.0407 (12)
C11	0.7142 (14)	0.6068 (5)	0.4576 (2)	0.0492 (14)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.056 (2)	0.0459 (19)	0.0467 (19)	−0.007 (2)	−0.009 (2)	0.0041 (16)
O2	0.059 (2)	0.0420 (18)	0.0451 (19)	−0.007 (2)	0.006 (2)	−0.0011 (15)
O3	0.038 (2)	0.059 (2)	0.075 (3)	−0.006 (2)	−0.003 (2)	0.007 (2)
O4	0.079 (3)	0.054 (2)	0.046 (2)	−0.016 (3)	0.003 (2)	−0.0063 (17)
N1	0.055 (3)	0.044 (2)	0.055 (2)	−0.001 (3)	−0.009 (3)	0.008 (2)
N2	0.093 (4)	0.060 (3)	0.052 (3)	0.020 (4)	0.000 (3)	−0.004 (2)
F1	0.106 (4)	0.128 (4)	0.082 (3)	0.045 (4)	−0.016 (3)	0.000 (3)
C1	0.071 (5)	0.066 (4)	0.054 (3)	0.003 (4)	−0.003 (3)	0.005 (3)
C2	0.106 (6)	0.072 (4)	0.070 (4)	−0.004 (5)	−0.022 (5)	0.017 (4)
C3	0.118 (7)	0.091 (5)	0.053 (4)	−0.021 (6)	−0.021 (5)	0.021 (4)
C4	0.108 (6)	0.134 (7)	0.043 (3)	−0.003 (7)	0.000 (4)	−0.005 (4)
C5	0.088 (5)	0.087 (5)	0.054 (4)	0.010 (5)	0.009 (4)	−0.010 (3)
C6	0.068 (4)	0.048 (3)	0.046 (3)	−0.003 (3)	0.000 (3)	0.005 (2)
C7	0.048 (3)	0.047 (3)	0.045 (3)	0.002 (3)	0.001 (3)	0.000 (2)
C8	0.063 (4)	0.037 (2)	0.038 (3)	0.003 (3)	−0.002 (3)	0.002 (2)
C9	0.056 (4)	0.042 (3)	0.043 (3)	0.001 (3)	0.002 (3)	0.003 (2)
C10	0.045 (3)	0.039 (3)	0.038 (3)	0.001 (3)	0.001 (3)	−0.002 (2)
C11	0.062 (4)	0.039 (3)	0.047 (3)	−0.009 (3)	0.002 (3)	−0.006 (2)

Geometric parameters (Å, º)

O1—C7	1.438 (6)	C3—C4	1.378 (11)
O1—C8	1.440 (5)	C3—H3	0.9300
O2—C7	1.398 (5)	C4—H4	0.9300
O2—C9	1.434 (5)	C5—C4	1.396 (8)
O3—C10	1.227 (6)	C5—H5	0.9300
O4—C11	1.240 (5)	C6—C1	1.372 (8)
N1—C10	1.323 (6)	C6—C5	1.376 (8)
N1—H1A	0.8600	C7—C6	1.505 (7)
N1—H1B	0.8600	C7—H7	0.9800
N2—H2A	0.8600	C8—H8	0.9800
N2—H2B	0.8600	C9—C8	1.540 (7)
F1—C1	1.363 (7)	C9—H9	0.9800
C2—C1	1.366 (7)	C10—C8	1.522 (7)
C2—H2	0.9300	C11—N2	1.313 (7)
C3—C2	1.367 (10)	C11—C9	1.507 (7)
C7—O1—C8	103.8 (4)	C5—C6—C7	118.9 (6)

C7—O2—C9	106.6 (3)	O1—C7—C6	108.5 (5)
C10—N1—H1A	120.0	O1—C7—H7	110.1
C10—N1—H1B	120.0	O2—C7—O1	104.4 (4)
H1A—N1—H1B	120.0	O2—C7—C6	113.5 (4)
C11—N2—H2A	120.0	O2—C7—H7	110.1
C11—N2—H2B	120.0	C6—C7—H7	110.1
H2A—N2—H2B	120.0	O1—C8—C10	108.6 (4)
F1—C1—C2	116.8 (7)	O1—C8—C9	103.6 (4)
F1—C1—C6	119.4 (5)	O1—C8—H8	109.9
C2—C1—C6	123.9 (7)	C9—C8—H8	109.9
C1—C2—C3	118.5 (7)	C10—C8—C9	114.6 (5)
C1—C2—H2	120.8	C10—C8—H8	109.9
C3—C2—H2	120.8	O2—C9—C8	104.3 (4)
C2—C3—C4	119.9 (6)	O2—C9—C11	111.0 (4)
C2—C3—H3	120.0	O2—C9—H9	109.8
C4—C3—H3	120.0	C8—C9—H9	109.8
C3—C4—C5	120.1 (7)	C11—C9—C8	111.9 (5)
C3—C4—H4	120.0	C11—C9—H9	109.8
C5—C4—H4	120.0	O3—C10—N1	123.2 (6)
C6—C5—C4	120.5 (7)	O3—C10—C8	121.8 (5)
C6—C5—H5	119.8	O4—C11—N2	123.9 (6)
C4—C5—H5	119.8	O4—C11—C9	118.9 (5)
C1—C6—C5	117.1 (6)	N1—C10—C8	114.8 (4)
C1—C6—C7	124.1 (5)	N2—C11—C9	117.1 (5)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O4^{i}	0.86	2.32	3.089 (4)	149
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