Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-(2,5-Dioxotetrahydrofuran-3-yl)isoindoline-1,3-dione

Shao-Song Qian

School of Life Sciences, Shandong University of Technology, Zibo 255049, People's Republic of China
Correspondence e-mail: njuqss@yahoo.com.cn

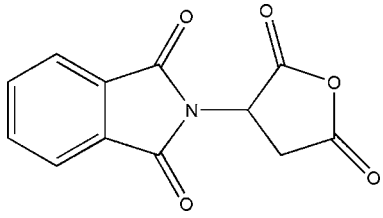
Received 25 July 2008; accepted 29 July 2008

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.044; wR factor = 0.134; data-to-parameter ratio = 11.4.

In the title compound, $C_{12}H_7NO_5$, the dihedral angle between the isoindole-1,3-dione plane and the least-squares plane of the furan ring is 89.2 (2)°. In the crystal structure, molecules are linked through intermolecular C—H···O hydrogen bonds, forming centrosymmetric dimers.

Related literature

For related literature, see: Abdel & Atef (2004); Allen *et al.* (1987); King & Kidd (1951); Qian *et al.* (2006).



Experimental

Crystal data

$C_{12}H_7NO_5$
M_r = 245.19
Monoclinic, $P2_1/n$
a = 12.129 (2) Å
b = 5.1385 (10) Å
c = 16.818 (3) Å
β = 100.21 (3)°
V = 1031.6 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 293 (2) K
0.30 × 0.30 × 0.05 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.963, T_{max} = 0.994
1963 measured reflections
1870 independent reflections
1492 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.133
S = 1.07
1870 reflections
164 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9A···O1	0.98	2.54	2.915 (3)	103 (4)
C12—H12B···O5^{i}	0.97	2.58	3.476 (3)	153 (4)

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); 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: *XP* in *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

This project was sponsored by the Doctoral Research Foundation (Shandong University of Technology, People's Republic of China).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2757).

References

Abdel, H. & Atef, A. M. (2004). *Arch. Pharm. Res.* **27**, 495–501.
Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Enraf–Nonius (1989). *CAD-4 Software*. Enraf–Nonius, Delft, The Netherlands.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
King, F. E. & Kidd, D. A. A. (1951). *J. Chem. Soc.* pp. 2976–2978.
Qian, S.-S., Cui, H.-Y. & Zhao, B.-S. (2006). *Acta Cryst.* E**62**, o2276–o2277.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2008). E**64**, o1663 [doi:10.1107/S1600536808024094]

2-(2,5-Dioxotetrahydrofuran-3-yl)isoindoline-1,3-dione

Shao-Song Qian

S1. Comment

The title compound has attracted attention for its anticonvulsant activity (Abdel & Atef, 2004). In addition, it was an intermediate for the synthesis of aspartic acid (King & Kidd, 1951). Here, we report its crystal structure.

The dihedral angle between the isoindole-1,3-dione plane and the plane of cyclopentane-1,3-dione is 90.0 (2)°. All the bond lengths are within normal ranges (Allen *et al.*, 1987) and comparable to the values observed in other similar compounds (Qian *et al.*, 2006). In the crystal structure, the molecules are linked through intermolecular C–H···O hydrogen bonds, forming centrosymmetric dimers.

S2. Experimental

The title compound was synthesized according to a literature method (Qian *et al.*, 2006). *L*-aspartic acid (13.3 g, 0.1 mol) reacted with *N*-carboethoxy phthalimide (21.9 g, 0.1 mol) in 200 ml of water and 23.3 g (0.21 mol) of sodium carbonate. As a result, 21.3 g of the N-phthaloyl-*L*-aspartic acid was obtained (yield, 81%). 10.8 g of the title compound was obtained through heating of N-phthaloyl-*L*-aspartic acid (13.2 g, 0.05 mol) in 30 ml of acetic anhydride under reflux for 20 minutes. Subsequently, 0.1 g of the title compound was dissolved in acetic acid (20 ml). Single crystals suitable for X-ray diffraction were obtained by spontaneous evaporation of the solvent.

S3. Refinement

All H atoms were geometrically positioned and constrained to ride on their parent atoms with C—H distance in the range 0.93–0.98 Å, with U_{iso}(H) = 1.2U_{eq}(C).

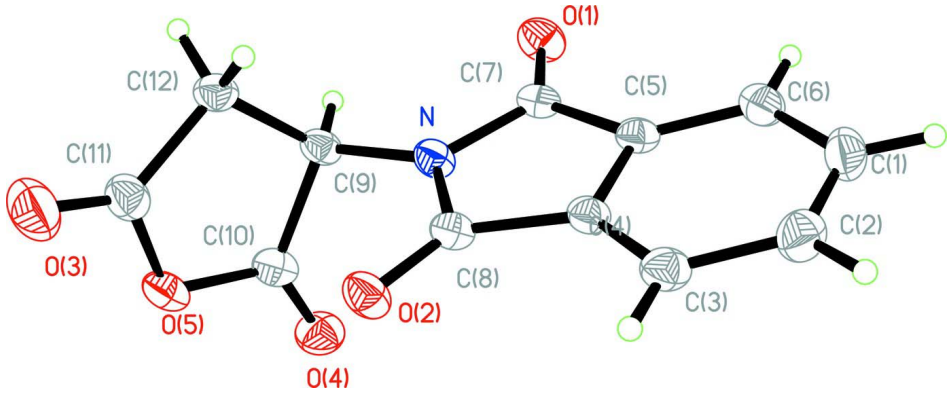


Figure 1

The structure of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 35% probability level.

2-(2,5-Dioxotetrahydrofuran-3-yl)isoindoline-1,3-dione

Crystal data

$C_{12}H_7NO_5$
$M_r = 245.19$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 12.129$ (2) Å
$b = 5.1385$ (10) Å
$c = 16.818$ (3) Å
$\beta = 100.21$ (3)°
$V = 1031.6$ (4) Å^3
$Z = 4$
$F(000) = 504$
$D_x = 1.579$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10$–13°
$\mu = 0.13$ mm^{-1}
$T = 293$ K
Prism, colorless
0.30 × 0.30 × 0.05 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.963$, $T_{max} = 0.994$
1963 measured reflections
1870 independent reflections
1492 reflections with $I > 2\sigma(I)$
$R_{int} = 0.040$
$\theta_{max} = 25.3°$, $\theta_{min} = 1.9°$
$h = 0 \rightarrow 14$
$k = 0 \rightarrow 6$
$l = -20 \rightarrow 19$
3 standard reflections every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.133$
$S = 1.07$
1870 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
$w = 1/[\sigma^2(F_o^2) + (0.0585P)^2 + 0.7913P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.055 (5)

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}
N	0.76293 (16)	−0.0485 (4)	0.04496 (11)	0.0347 (5)
O1	0.88949 (15)	−0.3867 (4)	0.04821 (11)	0.0466 (5)

C1	0.9239 (2)	0.0386 (6)	−0.18134 (16)	0.0487 (7)
H1A	0.9710	0.0066	−0.2183	0.058*
O2	0.65372 (15)	0.3125 (3)	0.00935 (10)	0.0430 (5)
C2	0.8492 (2)	0.2458 (6)	−0.19425 (15)	0.0481 (7)
H2A	0.8462	0.3474	−0.2404	0.058*
C3	0.7791 (2)	0.3047 (5)	−0.14002 (14)	0.0409 (6)
H3A	0.7292	0.4436	−0.1485	0.049*
O3	0.44844 (18)	0.1302 (5)	0.13241 (15)	0.0729 (7)
O4	0.81433 (16)	0.2349 (4)	0.20434 (11)	0.0523 (6)
C4	0.78694 (19)	0.1471 (5)	−0.07286 (13)	0.0332 (6)
O5	0.62761 (15)	0.2101 (3)	0.18451 (10)	0.0424 (5)
C5	0.85913 (19)	−0.0636 (5)	−0.06052 (13)	0.0341 (6)
C6	0.9295 (2)	−0.1217 (5)	−0.11412 (15)	0.0413 (6)
H6A	0.9786	−0.2621	−0.1057	0.050*
C7	0.84523 (19)	−0.1944 (5)	0.01570 (14)	0.0339 (6)
C8	0.7244 (2)	0.1603 (5)	−0.00526 (13)	0.0338 (6)
C9	0.7191 (2)	−0.1091 (5)	0.11716 (13)	0.0339 (6)
H9A	0.7604	−0.2566	0.1449	0.041*
C10	0.7312 (2)	0.1266 (5)	0.17350 (14)	0.0388 (6)
C11	0.5426 (2)	0.0628 (6)	0.13833 (15)	0.0445 (7)
C12	0.5937 (2)	−0.1667 (5)	0.10354 (15)	0.0388 (6)
H12A	0.5639	−0.1847	0.0464	0.047*
H12B	0.5791	−0.3258	0.1309	0.047*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N	0.0446 (11)	0.0297 (10)	0.0303 (10)	0.0057 (9)	0.0077 (8)	0.0006 (8)
O1	0.0528 (11)	0.0376 (10)	0.0481 (11)	0.0136 (9)	0.0056 (8)	0.0040 (8)
C1	0.0487 (15)	0.0611 (18)	0.0394 (14)	−0.0068 (14)	0.0165 (12)	−0.0077 (14)
O2	0.0536 (11)	0.0369 (10)	0.0398 (10)	0.0133 (9)	0.0117 (8)	0.0027 (8)
C2	0.0594 (17)	0.0513 (17)	0.0336 (13)	−0.0100 (14)	0.0082 (12)	0.0012 (12)
C3	0.0535 (15)	0.0341 (13)	0.0339 (13)	−0.0014 (12)	0.0045 (11)	−0.0007 (11)
O3	0.0529 (13)	0.0827 (17)	0.0840 (16)	0.0099 (13)	0.0147 (11)	−0.0248 (14)
O4	0.0595 (12)	0.0508 (12)	0.0444 (11)	−0.0101 (10)	0.0036 (9)	−0.0093 (9)
C4	0.0392 (12)	0.0296 (12)	0.0302 (12)	−0.0021 (11)	0.0046 (10)	−0.0041 (10)
O5	0.0583 (11)	0.0337 (10)	0.0353 (9)	0.0070 (8)	0.0090 (8)	−0.0050 (7)
C5	0.0388 (12)	0.0292 (12)	0.0328 (12)	−0.0040 (10)	0.0023 (10)	−0.0057 (10)
C6	0.0406 (13)	0.0396 (14)	0.0432 (14)	0.0004 (12)	0.0061 (11)	−0.0071 (12)
C7	0.0367 (12)	0.0276 (12)	0.0351 (12)	0.0009 (11)	0.0001 (10)	−0.0051 (10)
C8	0.0411 (13)	0.0285 (12)	0.0309 (12)	0.0018 (11)	0.0036 (10)	−0.0012 (10)
C9	0.0468 (14)	0.0266 (12)	0.0287 (11)	0.0047 (11)	0.0073 (10)	0.0014 (10)
C10	0.0555 (16)	0.0324 (13)	0.0277 (12)	0.0030 (12)	0.0048 (11)	0.0021 (10)
C11	0.0523 (16)	0.0446 (16)	0.0386 (14)	0.0034 (13)	0.0135 (12)	−0.0014 (12)
C12	0.0505 (15)	0.0286 (13)	0.0386 (13)	−0.0008 (11)	0.0110 (11)	0.0002 (10)

Geometric parameters (Å, º)

N—C8	1.394 (3)	C4—C5	1.385 (3)
N—C7	1.405 (3)	C4—C8	1.476 (3)
N—C9	1.443 (3)	O5—C10	1.370 (3)
O1—C7	1.208 (3)	O5—C11	1.398 (3)
C1—C6	1.391 (4)	C5—C6	1.380 (3)
C1—C2	1.390 (4)	C5—C7	1.483 (3)
C1—H1A	0.9300	C6—H6A	0.9300
O2—C8	1.217 (3)	C9—C12	1.526 (3)
C2—C3	1.387 (4)	C9—C10	1.529 (3)
C2—H2A	0.9300	C9—H9A	0.9800
C3—C4	1.379 (3)	C11—C12	1.499 (4)
C3—H3A	0.9300	C12—H12A	0.9700
O3—C11	1.180 (3)	C12—H12B	0.9700
O4—C10	1.188 (3)		
C8—N—C7	112.40 (19)	O1—C7—C5	130.7 (2)
C8—N—C9	122.82 (19)	N—C7—C5	104.9 (2)
C7—N—C9	124.76 (19)	O2—C8—N	123.0 (2)
C6—C1—C2	121.1 (2)	O2—C8—C4	131.4 (2)
C6—C1—H1A	119.4	N—C8—C4	105.6 (2)
C2—C1—H1A	119.4	N—C9—C12	114.92 (19)
C3—C2—C1	121.6 (2)	N—C9—C10	109.97 (19)
C3—C2—H2A	119.2	C12—C9—C10	103.30 (19)
C1—C2—H2A	119.2	N—C9—H9A	109.5
C4—C3—C2	116.7 (2)	C12—C9—H9A	109.5
C4—C3—H3A	121.7	C10—C9—H9A	109.5
C2—C3—H3A	121.7	O4—C10—O5	121.5 (2)
C3—C4—C5	122.1 (2)	O4—C10—C9	128.5 (2)
C3—C4—C8	129.3 (2)	O5—C10—C9	110.0 (2)
C5—C4—C8	108.6 (2)	O3—C11—O5	119.7 (3)
C10—O5—C11	111.05 (19)	O3—C11—C12	131.2 (3)
C6—C5—C4	121.3 (2)	O5—C11—C12	109.1 (2)
C6—C5—C7	130.2 (2)	C11—C12—C9	105.0 (2)
C4—C5—C7	108.5 (2)	C11—C12—H12A	110.7
C5—C6—C1	117.2 (2)	C9—C12—H12A	110.7
C5—C6—H6A	121.4	C11—C12—H12B	110.7
C1—C6—H6A	121.4	C9—C12—H12B	110.7
O1—C7—N	124.3 (2)	H12A—C12—H12B	108.8
C6—C1—C2—C3	1.4 (4)	C9—N—C8—C4	−177.8 (2)
C1—C2—C3—C4	−0.2 (4)	C3—C4—C8—O2	−1.9 (4)
C2—C3—C4—C5	−1.5 (4)	C5—C4—C8—O2	179.3 (3)
C2—C3—C4—C8	179.9 (2)	C3—C4—C8—N	178.5 (2)
C3—C4—C5—C6	1.9 (4)	C5—C4—C8—N	−0.3 (3)
C8—C4—C5—C6	−179.2 (2)	C8—N—C9—C12	59.4 (3)
C3—C4—C5—C7	−178.7 (2)	C7—N—C9—C12	−118.4 (2)

C8—C4—C5—C7	0.2 (3)	C8—N—C9—C10	−56.6 (3)
C4—C5—C6—C1	−0.7 (4)	C7—N—C9—C10	125.6 (2)
C7—C5—C6—C1	−179.9 (2)	C11—O5—C10—O4	176.2 (2)
C2—C1—C6—C5	−1.0 (4)	C11—O5—C10—C9	−2.7 (3)
C8—N—C7—O1	−178.7 (2)	N—C9—C10—O4	−61.0 (3)
C9—N—C7—O1	−0.7 (4)	C12—C9—C10—O4	175.9 (3)
C8—N—C7—C5	−0.2 (3)	N—C9—C10—O5	117.8 (2)
C9—N—C7—C5	177.9 (2)	C12—C9—C10—O5	−5.3 (2)
C6—C5—C7—O1	−2.3 (4)	C10—O5—C11—O3	−170.0 (3)
C4—C5—C7—O1	178.4 (2)	C10—O5—C11—C12	9.9 (3)
C6—C5—C7—N	179.3 (2)	O3—C11—C12—C9	167.1 (3)
C4—C5—C7—N	0.0 (2)	O5—C11—C12—C9	−12.7 (3)
C7—N—C8—O2	−179.4 (2)	N—C9—C12—C11	−109.3 (2)
C9—N—C8—O2	2.6 (4)	C10—C9—C12—C11	10.5 (2)
C7—N—C8—C4	0.3 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9*A*···O1	0.98	2.54	2.915 (3)	103 (4)
C12—H12*B*···O5^{i}	0.97	2.58	3.476 (3)	153 (4)

Symmetry code: (i) *x*, *y*−1, *z*.