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2-[(3*S*)-5-Oxooxolan-3-yl]isoindoline-1,3-dione

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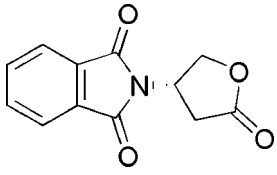
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.028; wR factor = 0.073; data-to-parameter ratio = 7.0.

The oxolan-2-one ring in the title compound, $C_{12}H_9NO_4$, has an envelope conformation with the atom linking the two five-membered rings being the flap atom.

Related literature

For the synthesis of the title compound, see: Temperini *et al.* (2010). For the structure of the closely related compound, 2-(2,5-dioxotetrahydrofuran-3-yl)isoindoline-1,3-dione, see: Qian (2008).



Experimental

Crystal data

$C_{12}H_9NO_4$ M_r = 231.20
Orthorhombic, $P2_12_12_1$
a = 5.7224 (3) Å
b = 10.5839 (5) Å
c = 16.8532 (10) Å
V = 1020.72 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 296 K
0.20 × 0.20 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.977, T_{max} = 0.984
4468 measured reflections
1077 independent reflections
1002 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.073
S = 1.20
1077 reflections
154 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.10 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5369).

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supporting information

Acta Cryst. (2012). E**68**, o24 [doi:10.1107/S160053681105135X]

2-[(3*S*)-5-Oxooxolan-3-yl]isoindoline-1,3-dione

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S1. Comment

The title compound is a key intermediate in our organic synthesis work. It was originally synthesized by Temperini *et al.* (2010). We report herein its crystal structure.

The molecular structure of the title compound (I) is shown in Fig. 1. The structure of the closely related compound, 2-(2,5-Dioxotetrahydrofuran-3-yl)isoindoline-1,3-dione, has already been published (Qian, 2008). In (I) The five-membered ring is in an envelope conformation.

S2. Experimental

The title compound was prepared from L-Aspartic acid as starting material. First, the amino acid was converted into the Cbz-protected lactone. Then, the Cbz-protected lactone (2.34 g, 10 mmoL) was hydrogenated at 1 atm with phthalic anhydride (1.78 g, 12 mmoL) in 30 mL of DMF and 0.53 g of 10% Pd/C. Single crystals suitable for X-ray diffraction were obtained by evaporation of the a DMF solution of the title compound.

S3. Refinement

H atoms were placed in calculated positions and treated as riding atoms with C-H= 0.93 - 0.98 Å, with $U_{iso}(H)=1.2U_{eq}(C)$. In the absense of significant anomalous dispersion effects the Friedel pairs were merged. The absolute configuration was based on that of the starting material.

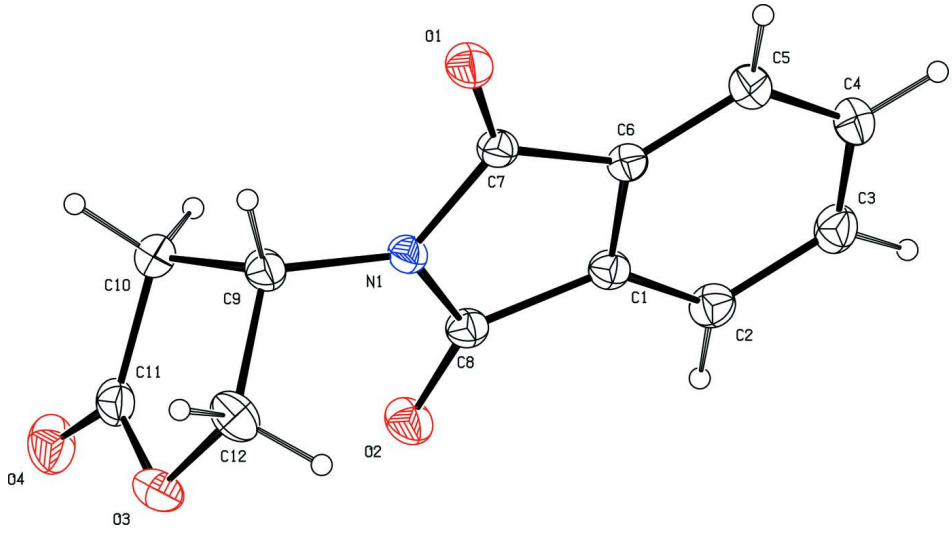

Figure 1

The molecular structure of with displacement ellipsoids drawn at the 20% probability level.

2-[(3*S*)-5-Oxooxolan-3-yl]isoindoline-1,3-dione

Crystal data

$C_{12}H_9NO_4$
$M_r = 231.20$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 5.7224$ (3) Å
$b = 10.5839$ (5) Å
$c = 16.8532$ (10) Å
$V = 1020.72$ (9) Å^3
$Z = 4$
$F(000) = 480$
$D_x = 1.505$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2599 reflections
$\theta = 2.3$–$27.2°$
$\mu = 0.12$ mm^{-1}
$T = 296$ K
Block, colourless
0.20 × 0.20 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.977$, $T_{max} = 0.984$
4468 measured reflections
1077 independent reflections
1002 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.3°$
$h = -6 \rightarrow 6$
$k = -12 \rightarrow 12$
$l = -19 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.073$
$S = 1.20$
1077 reflections
154 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
$w = 1/[\sigma^2(F_o^2) + (0.0366P)^2 + 0.0827P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.10$ e Å^{-3}
$\Delta\rho_{min} = -0.13$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2$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}
C1	0.8388 (3)	0.49330 (17)	0.93941 (11)	0.0362 (4)
C2	0.6577 (4)	0.5765 (2)	0.95245 (13)	0.0445 (5)
H2	0.5424	0.5900	0.9145	0.053*
C3	0.6548 (4)	0.6395 (2)	1.02494 (13)	0.0493 (5)
H3	0.5347	0.6960	1.0360	0.059*

C4	0.8280 (4)	0.6193 (2)	1.08081 (13)	0.0501 (5)
H4	0.8220	0.6627	1.1288	0.060*
C5	1.0100 (4)	0.5361 (2)	1.06693 (13)	0.0470 (5)
H5	1.1268	0.5232	1.1044	0.056*
C6	1.0115 (3)	0.47294 (17)	0.99517 (11)	0.0365 (4)
C7	1.1748 (3)	0.37695 (18)	0.96411 (11)	0.0366 (4)
C8	0.8880 (3)	0.41079 (18)	0.87031 (11)	0.0371 (4)
C9	1.2036 (4)	0.2499 (2)	0.83890 (11)	0.0422 (5)
H9	1.3392	0.2152	0.8669	0.051*
C10	1.0378 (4)	0.1429 (2)	0.81692 (13)	0.0500 (6)
H10A	1.1173	0.0621	0.8192	0.060*
H10B	0.9051	0.1408	0.8527	0.060*
C11	0.9611 (4)	0.1714 (2)	0.73468 (13)	0.0449 (5)
C12	1.2804 (4)	0.2984 (2)	0.75729 (11)	0.0524 (6)
H12A	1.2911	0.3898	0.7575	0.063*
H12B	1.4318	0.2639	0.7432	0.063*
N1	1.0948 (3)	0.34698 (16)	0.88763 (9)	0.0351 (4)
O1	1.3448 (3)	0.33039 (14)	0.99497 (8)	0.0510 (4)
O2	0.7776 (3)	0.39782 (15)	0.80953 (9)	0.0510 (4)
O3	1.1040 (3)	0.25678 (15)	0.70162 (8)	0.0522 (4)
O4	0.8005 (3)	0.12687 (16)	0.69809 (11)	0.0653 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0359 (10)	0.0372 (10)	0.0356 (10)	−0.0026 (9)	0.0013 (9)	0.0020 (8)
C2	0.0405 (11)	0.0454 (11)	0.0476 (12)	0.0047 (9)	0.0003 (10)	0.0038 (10)
C3	0.0514 (12)	0.0423 (11)	0.0543 (13)	0.0063 (11)	0.0120 (11)	−0.0014 (10)
C4	0.0613 (13)	0.0461 (11)	0.0429 (12)	0.0036 (11)	0.0050 (11)	−0.0071 (10)
C5	0.0543 (12)	0.0460 (12)	0.0406 (12)	0.0020 (10)	−0.0040 (10)	−0.0038 (9)
C6	0.0408 (9)	0.0344 (10)	0.0342 (10)	−0.0010 (8)	0.0003 (9)	0.0027 (8)
C7	0.0396 (10)	0.0371 (10)	0.0332 (10)	−0.0022 (9)	−0.0040 (9)	0.0023 (8)
C8	0.0360 (9)	0.0384 (10)	0.0369 (10)	−0.0005 (8)	−0.0034 (9)	0.0024 (8)
C9	0.0442 (10)	0.0453 (11)	0.0371 (11)	0.0112 (10)	−0.0067 (9)	−0.0038 (9)
C10	0.0698 (14)	0.0366 (11)	0.0436 (12)	0.0036 (11)	−0.0023 (11)	−0.0004 (9)
C11	0.0484 (13)	0.0404 (11)	0.0460 (12)	0.0097 (10)	−0.0052 (10)	−0.0094 (10)
C12	0.0452 (12)	0.0686 (16)	0.0435 (12)	−0.0027 (12)	0.0070 (10)	−0.0075 (11)
N1	0.0370 (8)	0.0379 (9)	0.0306 (8)	0.0039 (7)	−0.0028 (7)	−0.0008 (7)
O1	0.0515 (8)	0.0563 (9)	0.0452 (8)	0.0123 (8)	−0.0160 (8)	−0.0030 (7)
O2	0.0479 (8)	0.0621 (10)	0.0430 (8)	0.0088 (8)	−0.0144 (7)	−0.0063 (7)
O3	0.0560 (8)	0.0674 (10)	0.0331 (8)	0.0037 (9)	−0.0010 (7)	0.0007 (8)
O4	0.0620 (9)	0.0601 (10)	0.0737 (11)	0.0063 (9)	−0.0234 (10)	−0.0173 (9)

Geometric parameters (Å, º)

C1—C2	1.378 (3)	C8—O2	1.211 (2)
C1—C6	1.381 (3)	C8—N1	1.394 (2)
C1—C8	1.482 (3)	C9—N1	1.455 (2)

C2—C3	1.392 (3)	C9—C10	1.523 (3)
C2—H2	0.9300	C9—C12	1.532 (3)
C3—C4	1.383 (3)	C9—H9	0.9800
C3—H3	0.9300	C10—C11	1.484 (3)
C4—C5	1.384 (3)	C10—H10A	0.9700
C4—H4	0.9300	C10—H10B	0.9700
C5—C6	1.382 (3)	C11—O4	1.203 (2)
C5—H5	0.9300	C11—O3	1.340 (3)
C6—C7	1.476 (3)	C12—O3	1.446 (3)
C7—O1	1.208 (2)	C12—H12A	0.9700
C7—N1	1.404 (2)	C12—H12B	0.9700

C2—C1—C6	121.98 (18)	N1—C9—C12	113.13 (18)
C2—C1—C8	130.15 (18)	C10—C9—C12	102.07 (17)
C6—C1—C8	107.87 (16)	N1—C9—H9	109.4
C1—C2—C3	117.1 (2)	C10—C9—H9	109.4
C1—C2—H2	121.5	C12—C9—H9	109.4
C3—C2—H2	121.5	C11—C10—C9	105.11 (18)
C4—C3—C2	121.0 (2)	C11—C10—H10A	110.7
C4—C3—H3	119.5	C9—C10—H10A	110.7
C2—C3—H3	119.5	C11—C10—H10B	110.7
C3—C4—C5	121.5 (2)	C9—C10—H10B	110.7
C3—C4—H4	119.3	H10A—C10—H10B	108.8
C5—C4—H4	119.3	O4—C11—O3	121.1 (2)
C6—C5—C4	117.4 (2)	O4—C11—C10	128.7 (2)
C6—C5—H5	121.3	O3—C11—C10	110.14 (18)
C4—C5—H5	121.3	O3—C12—C9	106.29 (18)
C1—C6—C5	121.05 (19)	O3—C12—H12A	110.5
C1—C6—C7	108.61 (16)	C9—C12—H12A	110.5
C5—C6—C7	130.32 (19)	O3—C12—H12B	110.5
O1—C7—N1	124.45 (19)	C9—C12—H12B	110.5
O1—C7—C6	129.62 (19)	H12A—C12—H12B	108.7
N1—C7—C6	105.93 (15)	C8—N1—C7	111.08 (16)
O2—C8—N1	124.41 (18)	C8—N1—C9	125.97 (16)
O2—C8—C1	129.20 (18)	C7—N1—C9	122.54 (16)
N1—C8—C1	106.39 (16)	C11—O3—C12	111.18 (16)
N1—C9—C10	113.28 (16)		

C6—C1—C2—C3	−0.3 (3)	C12—C9—C10—C11	−21.9 (2)
C8—C1—C2—C3	178.4 (2)	C9—C10—C11—O4	−165.3 (2)
C1—C2—C3—C4	0.4 (3)	C9—C10—C11—O3	16.2 (2)
C2—C3—C4—C5	0.0 (3)	N1—C9—C12—O3	−101.25 (19)
C3—C4—C5—C6	−0.4 (3)	C10—C9—C12—O3	20.8 (2)
C2—C1—C6—C5	−0.2 (3)	O2—C8—N1—C7	−176.92 (19)
C8—C1—C6—C5	−179.17 (18)	C1—C8—N1—C7	3.3 (2)
C2—C1—C6—C7	178.63 (17)	O2—C8—N1—C9	−4.1 (3)
C8—C1—C6—C7	−0.3 (2)	C1—C8—N1—C9	176.10 (18)
C4—C5—C6—C1	0.6 (3)	O1—C7—N1—C8	176.68 (18)

C4—C5—C6—C7	−177.97 (19)	C6—C7—N1—C8	−3.45 (19)
C1—C6—C7—O1	−177.9 (2)	O1—C7—N1—C9	3.6 (3)
C5—C6—C7—O1	0.8 (4)	C6—C7—N1—C9	−176.58 (16)
C1—C6—C7—N1	2.3 (2)	C10—C9—N1—C8	−52.5 (3)
C5—C6—C7—N1	−179.1 (2)	C12—C9—N1—C8	63.0 (2)
C2—C1—C8—O2	−0.4 (4)	C10—C9—N1—C7	119.54 (18)
C6—C1—C8—O2	178.5 (2)	C12—C9—N1—C7	−124.9 (2)
C2—C1—C8—N1	179.41 (19)	O4—C11—O3—C12	178.82 (19)
C6—C1—C8—N1	−1.7 (2)	C10—C11—O3—C12	−2.6 (2)
N1—C9—C10—C11	100.02 (19)	C9—C12—O3—C11	−12.1 (2)