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(*E*)-3-(Oxolan-2-ylidene)-1-phenylpyrrolidine-2,5-dione

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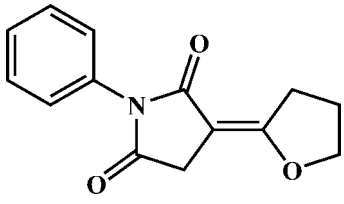
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.005 Å; R factor = 0.075; wR factor = 0.161; data-to-parameter ratio = 13.4.

In the title compound, $C_{14}H_{13}NO_3$, the dihedral angles between the central pyrrolidine ring and the pendant tetrahydrofuran and phenyl rings are 5.34 (18) and 58.99 (17)°, respectively. The tetrahydrofuran ring is almost planar (r.m.s. deviation = 0.008 Å). In the crystal, molecules are linked by C—H···O interactions, generating a three-dimensional network.

Related literature

For synthetic background, see: Han *et al.* (2013); Sodhi *et al.* (2012).



Experimental

Crystal data

$C_{14}H_{13}NO_3$
M_r = 243.25
Monoclinic, $P2_1/n$
a = 8.144 (2) Å
b = 13.729 (4) Å
c = 11.160 (3) Å
β = 105.177 (8)°
V = 1204.2 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 296 K
0.30 × 0.28 × 0.25 mm

Data collection

Rigaku Mercury CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2000)
T_{min} = 0.736, T_{max} = 0.977
11234 measured reflections
2201 independent reflections
1893 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.075
$wR(F^2)$ = 0.161
S = 1.08
2201 reflections
164 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 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
C6—H6···O2^{i}	0.93	2.59	3.487 (4)	161
C9—H9A···O1^{ii}	0.97	2.50	3.403 (3)	154
C14—H14A···O2^{iii}	0.97	2.50	3.376 (4)	150
C14—H14B···O2^{iv}	0.97	2.51	3.384 (4)	149

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

Data collection: *CrystalClear* (Rigaku, 2000); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku, 2000); 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: *SHELXTL*.

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7212).

References

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Rigaku (2000). *CrystalClear* and *CrystalStructure*. Rigaku Corporation, Tokyo, Japan.
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Sodhi, R. K., Paul, S. & Clark, J. H. (2012). *Green Chem.* **14**, 1649–1656.

supporting information

Acta Cryst. (2014). E**70**, o521 [doi:10.1107/S1600536814007193]

(*E*)-3-(Oxolan-2-ylidene)-1-phenylpyrrolidine-2,5-dione

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S1. Comment

The area of allylic and benzylic oxidation with *tert*-butyl hydroperoxide (TBHP) in the presence of $Co(acac)_2$ has been attracted more and more attention (Han, *et al.*, 2013; Sodhi, *et al.*, 2012). The title compound, $C_{14}H_{13}NO_3$, was synthesized by $Co(acac)_2$ catalyzed cross-dehydrogenative-coupling (CDC) between 1-phenyl-1*H*-pyrrole-2,5-dione and tetrahydrofuran in the presence of *t*-BuOOH as a oxidant in the air. In the molecule of the title compound (Fig. 1), the compound adopts an E conformation. All the non-H atoms of the pyrrolidine-2,5-dione and the tetrahydrofuran fragment, linked by carbon—carbon double bond, are nearly coplanar, with a maximum deviation of 0.056 (1) Å. While the dihedral angle between the benzene ring and the pyrrolidine-2,5-dione ring is 59.9 Å. In the crystal, C—H···O interactions link the molecules (Table 1).

S2. Experimental

1-Phenyl-1*H*-pyrrole-2,5-dione(86.6 mg, 0.5 mmol), THF (0.5 ml, 7.0 mmol), cobalt(II) acetylacetonate (12.9 mg), 1,4-diazabicyclo[2.2.2]octane (70.1 mg, 0.6 mmol), TBHP (2.0 equiv, 70% aqueous solution 140 uL), 1.0 ml acetonitrile, 1.0 ml 1,4-dioxane were added to a tube under air. The reaction mixture was stirred at 60 °C for 4 h. Then the reaction mixture was quenched with saturated Na_2SO_3 solution, extracted repeatedly with ethyl acetate, and dried over Na_2SO_4. It was then removal of the organic solvent in vacuum and followed by flash silica gel column chromatographic purification afforded product with petroleum/ ethyl acetate mixtures. Yield 40%. Colourless crystals were obtained by slow evaporation of ethyl acetate and CH_2Cl_2 mixed solvent.

S3. Refinement

All the H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances of 0.93–0.97 Å, and with $U_{iso}(H) = 1.2U_{eq}(C)$.

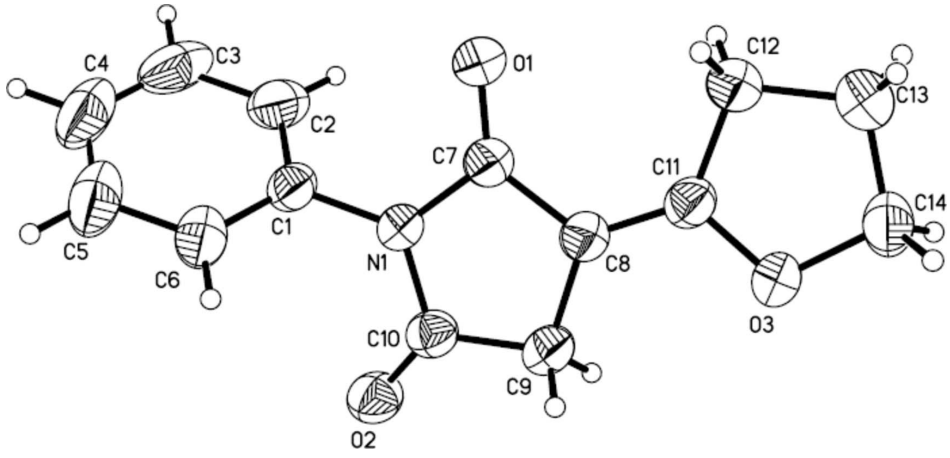

Figure 1

View of the title compound, showing 50% probability ellipsoids.

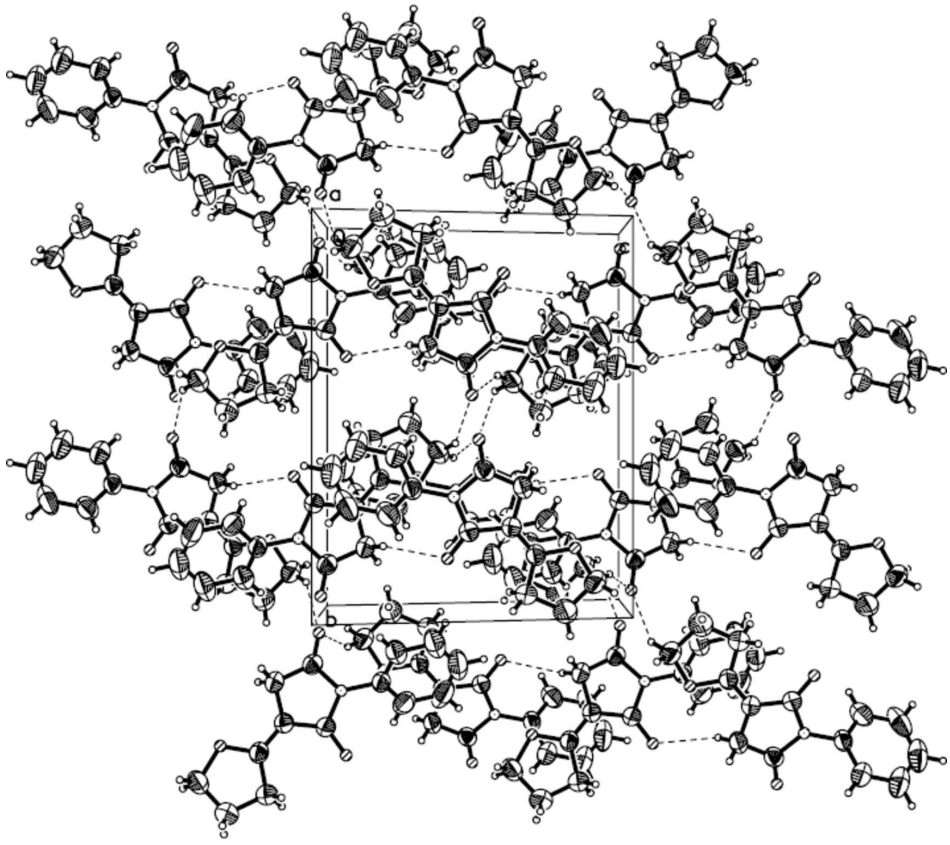

Figure 2

Perspective view of the packing of the title compound along a direction.

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Crystal data

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c = 11.160 (3) Å
β = 105.177 (8)°
V = 1204.2 (6) Å^3
Z = 4
F(000) = 512
D_x = 1.342 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71070 Å
Cell parameters from 4321 reflections
θ = 3.2–25.3°
μ = 0.10 mm^{-1}
T = 296 K
Block, colorless
0.30 × 0.28 × 0.25 mm

Data collection

Rigaku Mercury CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 7.31 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2000)
T_{min} = 0.736, T_{max} = 0.977

11234 measured reflections
2201 independent reflections
1893 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 25.3°, θ_{min} = 3.2°
h = −9→9
k = −16→15
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.075
$wR(F^2)$ = 0.161
S = 1.08
2201 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
$w = 1/[\sigma^2(F_o^2) + (0.0555P)^2 + 0.8273P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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	0.5949 (3)	0.34863 (14)	0.08693 (17)	0.0660 (6)
O2	0.3330 (3)	0.05454 (15)	−0.01104 (17)	0.0632 (6)
O3	0.4751 (3)	0.32363 (14)	−0.31330 (16)	0.0612 (6)
N1	0.4759 (3)	0.19466 (15)	0.06629 (18)	0.0505 (6)
C1	0.5003 (3)	0.1742 (2)	0.1954 (2)	0.0526 (7)
C2	0.4297 (4)	0.2340 (2)	0.2671 (3)	0.0720 (9)
H2	0.3637	0.2873	0.2323	0.086*
C3	0.4596 (6)	0.2129 (3)	0.3938 (3)	0.0938 (13)

H3	0.4153	0.2531	0.4447	0.113*
C4	0.5547 (6)	0.1324 (4)	0.4428 (3)	0.0986 (14)
H4	0.5739	0.1185	0.5269	0.118*
C5	0.6209 (5)	0.0727 (3)	0.3697 (3)	0.0866 (11)
H5	0.6835	0.0180	0.4035	0.104*
C6	0.5947 (4)	0.0937 (2)	0.2458 (3)	0.0639 (8)
H6	0.6406	0.0536	0.1957	0.077*
C7	0.5310 (3)	0.28121 (19)	0.0200 (2)	0.0514 (6)
C8	0.4917 (3)	0.27047 (19)	−0.1134 (2)	0.0507 (6)
C9	0.4145 (4)	0.1720 (2)	−0.1479 (2)	0.0571 (7)
H9A	0.3038	0.1775	−0.2069	0.069*
H9B	0.4875	0.1319	−0.1838	0.069*
C10	0.3991 (3)	0.1303 (2)	−0.0279 (2)	0.0503 (6)
C11	0.5168 (3)	0.34047 (19)	−0.1899 (2)	0.0508 (7)
C12	0.5857 (4)	0.4404 (2)	−0.1610 (3)	0.0603 (7)
H12A	0.7015	0.4385	−0.1089	0.072*
H12B	0.5161	0.4776	−0.1189	0.072*
C13	0.5797 (6)	0.4839 (3)	−0.2852 (3)	0.0956 (13)
H13A	0.5089	0.5418	−0.2989	0.115*
H13B	0.6932	0.5019	−0.2895	0.115*
C14	0.5080 (4)	0.4095 (2)	−0.3792 (3)	0.0679 (8)
H14A	0.4032	0.4329	−0.4351	0.081*
H14B	0.5878	0.3943	−0.4276	0.081*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0912 (15)	0.0522 (11)	0.0478 (11)	−0.0092 (11)	0.0059 (10)	−0.0045 (9)
O2	0.0689 (13)	0.0623 (12)	0.0580 (11)	−0.0180 (10)	0.0161 (9)	−0.0065 (10)
O3	0.0833 (14)	0.0570 (11)	0.0427 (10)	−0.0044 (10)	0.0154 (9)	0.0014 (9)
N1	0.0628 (14)	0.0465 (12)	0.0390 (11)	−0.0043 (10)	0.0080 (9)	−0.0018 (9)
C1	0.0590 (16)	0.0550 (16)	0.0420 (14)	−0.0120 (13)	0.0103 (12)	−0.0030 (12)
C2	0.086 (2)	0.068 (2)	0.0651 (19)	−0.0158 (17)	0.0259 (17)	−0.0166 (16)
C3	0.120 (3)	0.108 (3)	0.066 (2)	−0.048 (3)	0.046 (2)	−0.038 (2)
C4	0.114 (3)	0.128 (4)	0.048 (2)	−0.053 (3)	0.010 (2)	0.008 (2)
C5	0.084 (2)	0.112 (3)	0.056 (2)	−0.020 (2)	0.0052 (17)	0.022 (2)
C6	0.0620 (18)	0.073 (2)	0.0542 (17)	−0.0030 (15)	0.0099 (13)	0.0091 (14)
C7	0.0576 (16)	0.0468 (15)	0.0464 (14)	0.0016 (12)	0.0077 (12)	−0.0012 (12)
C8	0.0559 (16)	0.0484 (14)	0.0445 (14)	0.0023 (12)	0.0074 (11)	−0.0001 (12)
C9	0.0667 (18)	0.0587 (17)	0.0431 (14)	−0.0069 (14)	0.0092 (12)	−0.0059 (12)
C10	0.0478 (15)	0.0512 (15)	0.0496 (15)	0.0000 (12)	0.0085 (11)	−0.0050 (12)
C11	0.0532 (15)	0.0532 (15)	0.0435 (14)	0.0046 (12)	0.0080 (11)	−0.0025 (12)
C12	0.0725 (19)	0.0493 (15)	0.0572 (16)	0.0014 (14)	0.0136 (14)	−0.0015 (13)
C13	0.153 (4)	0.067 (2)	0.069 (2)	−0.030 (2)	0.032 (2)	0.0000 (18)
C14	0.086 (2)	0.0648 (19)	0.0566 (17)	0.0054 (16)	0.0264 (16)	0.0122 (15)

Geometric parameters (Å, º)

O1—C7	1.217 (3)	C6—H6	0.9300
O2—C10	1.208 (3)	C7—C8	1.447 (4)
O3—C11	1.349 (3)	C8—C11	1.336 (4)
O3—C14	1.451 (3)	C8—C9	1.498 (4)
N1—C10	1.390 (3)	C9—C10	1.492 (4)
N1—C7	1.414 (3)	C9—H9A	0.9700
N1—C1	1.430 (3)	C9—H9B	0.9700
C1—C2	1.372 (4)	C11—C12	1.486 (4)
C1—C6	1.379 (4)	C12—C13	1.498 (4)
C2—C3	1.402 (5)	C12—H12A	0.9700
C2—H2	0.9300	C12—H12B	0.9700
C3—C4	1.378 (6)	C13—C14	1.472 (4)
C3—H3	0.9300	C13—H13A	0.9700
C4—C5	1.364 (6)	C13—H13B	0.9700
C4—H4	0.9300	C14—H14A	0.9700
C5—C6	1.374 (4)	C14—H14B	0.9700
C5—H5	0.9300		
C11—O3—C14	110.3 (2)	C10—C9—H9A	110.9
C10—N1—C7	112.4 (2)	C8—C9—H9A	110.9
C10—N1—C1	123.6 (2)	C10—C9—H9B	110.9
C7—N1—C1	124.0 (2)	C8—C9—H9B	110.9
C2—C1—C6	121.0 (3)	H9A—C9—H9B	108.9
C2—C1—N1	120.0 (3)	O2—C10—N1	124.1 (2)
C6—C1—N1	118.9 (3)	O2—C10—C9	128.0 (2)
C1—C2—C3	118.4 (4)	N1—C10—C9	107.9 (2)
C1—C2—H2	120.8	C8—C11—O3	119.3 (2)
C3—C2—H2	120.8	C8—C11—C12	129.6 (2)
C4—C3—C2	119.8 (4)	O3—C11—C12	111.1 (2)
C4—C3—H3	120.1	C11—C12—C13	104.4 (2)
C2—C3—H3	120.1	C11—C12—H12A	110.9
C5—C4—C3	121.0 (3)	C13—C12—H12A	110.9
C5—C4—H4	119.5	C11—C12—H12B	110.9
C3—C4—H4	119.5	C13—C12—H12B	110.9
C4—C5—C6	119.6 (4)	H12A—C12—H12B	108.9
C4—C5—H5	120.2	C14—C13—C12	107.1 (3)
C6—C5—H5	120.2	C14—C13—H13A	110.3
C5—C6—C1	120.1 (3)	C12—C13—H13A	110.3
C5—C6—H6	119.9	C14—C13—H13B	110.3
C1—C6—H6	119.9	C12—C13—H13B	110.3
O1—C7—N1	122.7 (2)	H13A—C13—H13B	108.6
O1—C7—C8	130.7 (3)	O3—C14—C13	107.1 (2)
N1—C7—C8	106.5 (2)	O3—C14—H14A	110.3
C11—C8—C7	123.7 (2)	C13—C14—H14A	110.3
C11—C8—C9	127.5 (2)	O3—C14—H14B	110.3
C7—C8—C9	108.7 (2)	C13—C14—H14B	110.3

C10—C9—C8	104.1 (2)	H14A—C14—H14B	108.6
C10—N1—C1—C2	−120.8 (3)	C11—C8—C9—C10	−173.3 (3)
C7—N1—C1—C2	60.9 (4)	C7—C8—C9—C10	4.4 (3)
C10—N1—C1—C6	58.9 (4)	C7—N1—C10—O2	−175.2 (3)
C7—N1—C1—C6	−119.3 (3)	C1—N1—C10—O2	6.4 (4)
C6—C1—C2—C3	1.7 (4)	C7—N1—C10—C9	4.8 (3)
N1—C1—C2—C3	−178.6 (3)	C1—N1—C10—C9	−173.6 (2)
C1—C2—C3—C4	−1.4 (5)	C8—C9—C10—O2	174.5 (3)
C2—C3—C4—C5	0.1 (6)	C8—C9—C10—N1	−5.5 (3)
C3—C4—C5—C6	0.9 (6)	C7—C8—C11—O3	−179.6 (2)
C4—C5—C6—C1	−0.7 (5)	C9—C8—C11—O3	−2.2 (4)
C2—C1—C6—C5	−0.6 (4)	C7—C8—C11—C12	−0.3 (5)
N1—C1—C6—C5	179.6 (3)	C9—C8—C11—C12	177.1 (3)
C10—N1—C7—O1	176.9 (3)	C14—O3—C11—C8	178.4 (3)
C1—N1—C7—O1	−4.7 (4)	C14—O3—C11—C12	−1.0 (3)
C10—N1—C7—C8	−1.9 (3)	C8—C11—C12—C13	−179.0 (3)
C1—N1—C7—C8	176.5 (2)	O3—C11—C12—C13	0.3 (4)
O1—C7—C8—C11	−2.6 (5)	C11—C12—C13—C14	0.5 (4)
N1—C7—C8—C11	176.1 (2)	C11—O3—C14—C13	1.3 (4)
O1—C7—C8—C9	179.6 (3)	C12—C13—C14—O3	−1.0 (4)
N1—C7—C8—C9	−1.7 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O2^{i}	0.93	2.59	3.487 (4)	161
C9—H9*A*···O1^{ii}	0.97	2.50	3.403 (3)	154
C14—H14*A*···O2^{iii}	0.97	2.50	3.376 (4)	150
C14—H14*B*···O2^{iv}	0.97	2.51	3.384 (4)	149

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