organic compounds

2,5-Dioxopyrrolidin-1-yl 3-(furan-2-yl)-acrylate

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The title compound, $C_{11}H_9NO_5$, was prepared by the reaction of 2-furanacrylic acid and *N*-hydroxysuccinimide. The molecule consists of two approximately planar moieties, *viz.* a succinimide group and the rest of the molecule [the largest deviations from the least-squares planes are 0.120 (1) and 0.210 (1) Å, respectively]. The dihedral angle between these fragments is 63.70 (5)°. In the crystal, molecules are linked by C—H···O hydrogen bonds into two-dimensional nets.

Related literature

For derivatives of *N*-hydroxysuccinimide, see: Anderson *et al.* (1964); Blumberg & Vallee (1975); Brown *et al.* (2005); Cheng *et al.* (2007); Jones (2003).
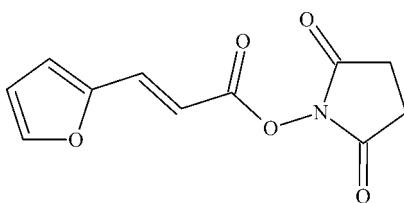
Experimental

Crystal data

$C_{11}H_9NO_5$	a = 10.3054 (13) Å
M_r = 235.19	b = 9.2376 (12) Å
Orthorhombic, *Pbca*	c = 21.892 (3) Å

V = 2084.0 (5) Å^3	μ = 0.12 mm^{-1}
Z = 8	T = 296 K
Mo $K\alpha$ radiation	0.30 × 0.30 × 0.10 mm

Data collection

Bruker APEXII CCD area-detector diffractometer	16939 measured reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2005) T_{min} = 0.977, T_{max} = 0.977	2399 independent reflections 1900 reflections with $I > 2\sigma(I)$ R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035	154 parameters
$wR(F^2)$ = 0.082	H-atom parameters constrained
S = 1.02	$\Delta\rho_{max}$ = 0.20 e Å^{-3}
2399 reflections	$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12A···O1^i	0.93	2.48	3.3533 (17)	156
C14—H14A···O5^{ii}	0.93	2.45	3.3672 (18)	169

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; 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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: YK2020).

References

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supplementary materials

2,5-Dioxopyrrolidin-1-yl 3-(furan-2-yl)acrylate

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Comment

N-Hydroxysuccinimide is frequently used in organic chemistry as an activating reagent, which readily form crystalline adducts with amines or acids (Jones, 2003). *N*-Hydroxysuccinimide esters are widely used as leaving groups to activate carboxylic acids (Cheng *et al.*, 2007). The title compound *N*-(2-furanacryloyl)-succinimide ester is an intermediate of FAP-GG which is the substrate of diagnostic reagent. We have used a simple procedure to synthesize the title compound in reasonable yields (Blumberg & Vallee, 1975; Brown *et al.*, 2005).

The molecular structure of the title compound (I) is shown in Fig.1. In the molecule, the dihedral angle between the furan and succinimide rings is 56.26 (43)°. In the crystal structure, molecules are linked by the C12—H12···O1^i hydrogen bonds to form chains along *a* and C14—H14···O5^{ii} hydrogen bonds to form chains along *b* directions.

Experimental

2-Furanacrylic acid (13.81 g, 0.10 mol), *N*-hydroxysuccinimide (11.51 g, 0.10 mol) and dicyclohexylcarbodiimide (20.63 g, 0.10 mol) were added to 200 ml dioxane in a round flask. This mixture was stirred at 4°C for 14 h before the dicyclohexylurea was removed by filtration. Then the resulting dark brown filtrate was evaporated in vacuum to give the dark brown residue. Slight brown crystals were obtained by recrystallization from 2-propanol (15.29 g, 65%). 1H NMR(400 MHz, CDCl3): \d 7.62 (d, J=16 Hz, 1H), 7.56 (d, J=1.6 Hz, 1H), 6.77 (d, J=3.6 Hz, 1H), 6.53–6.52 (dd, J=3.6 Hz, J=1.6 Hz, 1H), 6.47 (d, J=16 Hz, 1H), 2.87 (s, 4H).

Figures
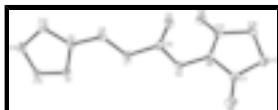
Fig. 1. The molecular structure of (I) showing displacement ellipsoids at the 30% probability level.

2,5-Dioxopyrrolidin-1-yl 3-(furan-2-yl)acrylate

Crystal data

$C_{11}H_9NO_5$	$F(000) = 976$
$M_r = 235.19$	$D_x = 1.499$ Mg m^{-3}
Orthorhombic, *Pbca*	Mo *Kα* radiation, $\lambda = 0.71073$ Å
Hall symbol: -p 2ac 2ab	Cell parameters from 3779 reflections
$a = 10.3054$ (13) Å	$\theta = 2.7–27.4°$
$b = 9.2376$ (12) Å	$\mu = 0.12$ mm^{-1}
$c = 21.892$ (3) Å	$T = 296$ K

supplementary materials

$V = 2084.0\ (5)\ \text{Å}^3$

$Z = 8$

Needle, brown

$0.30 \times 0.30 \times 0.10$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer	2399 independent reflections
Radiation source: fine-focus sealed tube	1900 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.041$
Detector resolution: 0 pixels mm^{-1}	$\theta_{max} = 27.6°$, $\theta_{min} = 1.9°$
φ and ω scans	$h = -13 \rightarrow 13$
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	$k = -12 \rightarrow 12$
$T_{min} = 0.977$, $T_{max} = 0.977$	$l = -28 \rightarrow 28$
16939 measured reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.035$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.082$	H-atom parameters constrained
$S = 1.02$	$w = 1/[\sigma^2(F_o^2) + (0.0285P)^2 + 0.9232P]$ where $P = (F_o^2 + 2F_c^2)/3$
2399 reflections	$(\Delta/\sigma)_{max} < 0.001$
154 parameters	$\Delta\rho_{max} = 0.20$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.24$ e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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, convention-al *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	0.80658 (9)	0.22692 (10)	0.28201 (4)	0.0261 (2)
O2	0.89760 (9)	0.55049 (10)	0.10826 (4)	0.0273 (2)
O3	0.67881 (9)	0.58836 (11)	0.11454 (5)	0.0314 (2)

O4	0.81461 (10)	0.44760 (11)	−0.00431 (5)	0.0356 (3)
O5	1.01727 (10)	0.81990 (11)	0.09170 (5)	0.0349 (3)
C6	0.68207 (13)	0.36659 (14)	0.21024 (6)	0.0237 (3)
H6A	0.6008	0.3944	0.1960	0.028*
C7	0.78634 (13)	0.42777 (14)	0.18443 (6)	0.0244 (3)
H7A	0.8686	0.4048	0.1989	0.029*
N8	0.89355 (11)	0.63213 (12)	0.05495 (5)	0.0246 (3)
C9	0.68679 (12)	0.26114 (14)	0.25831 (6)	0.0220 (3)
C10	0.77299 (13)	0.52965 (14)	0.13407 (6)	0.0233 (3)
C11	0.96337 (13)	0.76125 (14)	0.04991 (6)	0.0243 (3)
C12	0.59420 (14)	0.18035 (14)	0.28687 (6)	0.0255 (3)
H12A	0.5054	0.1829	0.2793	0.031*
C13	0.85503 (13)	0.56972 (15)	0.00020 (6)	0.0250 (3)
C14	0.65847 (14)	0.09163 (15)	0.33025 (6)	0.0270 (3)
H14A	0.6205	0.0249	0.3566	0.032*
C15	0.78534 (14)	0.12362 (15)	0.32556 (6)	0.0279 (3)
H15A	0.8503	0.0810	0.3489	0.034*
C16	0.87575 (15)	0.68378 (15)	−0.04765 (6)	0.0289 (3)
H16A	0.7934	0.7229	−0.0614	0.035*
H16B	0.9214	0.6439	−0.0826	0.035*
C17	0.95729 (14)	0.80146 (15)	−0.01663 (6)	0.0286 (3)
H17A	1.0438	0.8046	−0.0341	0.034*
H17B	0.9170	0.8956	−0.0217	0.034*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0231 (5)	0.0287 (5)	0.0264 (5)	−0.0003 (4)	−0.0014 (4)	0.0072 (4)
O2	0.0248 (5)	0.0329 (5)	0.0242 (5)	−0.0006 (4)	−0.0008 (4)	0.0097 (4)
O3	0.0272 (5)	0.0337 (5)	0.0334 (5)	0.0032 (4)	−0.0018 (4)	0.0105 (4)
O4	0.0398 (6)	0.0254 (5)	0.0416 (6)	−0.0037 (5)	−0.0043 (5)	−0.0042 (5)
O5	0.0351 (6)	0.0322 (5)	0.0376 (6)	−0.0055 (5)	−0.0022 (5)	−0.0085 (5)
C6	0.0264 (7)	0.0222 (6)	0.0226 (6)	0.0026 (5)	−0.0019 (5)	−0.0008 (5)
C7	0.0253 (7)	0.0243 (7)	0.0235 (7)	0.0016 (6)	−0.0028 (5)	0.0022 (5)
N8	0.0284 (6)	0.0253 (6)	0.0199 (5)	−0.0037 (5)	−0.0009 (5)	0.0052 (5)
C9	0.0222 (6)	0.0232 (6)	0.0208 (6)	0.0025 (5)	−0.0012 (5)	−0.0009 (5)
C10	0.0245 (7)	0.0215 (6)	0.0238 (6)	−0.0014 (6)	−0.0009 (5)	0.0000 (5)
C11	0.0211 (6)	0.0211 (6)	0.0309 (7)	0.0008 (5)	0.0032 (6)	−0.0016 (6)
C12	0.0245 (7)	0.0251 (7)	0.0268 (7)	−0.0017 (6)	0.0018 (5)	−0.0016 (5)
C13	0.0239 (7)	0.0243 (7)	0.0269 (7)	0.0037 (6)	−0.0016 (6)	−0.0025 (5)
C14	0.0363 (8)	0.0227 (6)	0.0220 (6)	−0.0047 (6)	0.0031 (6)	0.0010 (5)
C15	0.0359 (8)	0.0253 (7)	0.0226 (6)	0.0014 (6)	−0.0030 (6)	0.0065 (6)
C16	0.0355 (8)	0.0283 (7)	0.0229 (7)	0.0055 (6)	0.0013 (6)	0.0007 (6)
C17	0.0280 (7)	0.0246 (7)	0.0333 (8)	0.0012 (6)	0.0048 (6)	0.0068 (6)

Geometric parameters (Å, °)

O1—C15	1.3664 (16)	C9—C12	1.3632 (18)
O1—C9	1.3759 (15)	C11—C17	1.5044 (19)

O2—N8	1.3900 (13)	C12—C14	1.4185 (19)
O2—C10	1.4162 (16)	C12—H12A	0.9300
O3—C10	1.1912 (16)	C13—C16	1.5011 (19)
O4—C13	1.2066 (16)	C14—C15	1.344 (2)
O5—C11	1.1996 (16)	C14—H14A	0.9300
C6—C7	1.3391 (18)	C15—H15A	0.9300
C6—C9	1.4348 (18)	C16—C17	1.533 (2)
C6—H6A	0.9300	C16—H16A	0.9700
C7—C10	1.4561 (18)	C16—H16B	0.9700
C7—H7A	0.9300	C17—H17A	0.9700
N8—C13	1.3880 (17)	C17—H17B	0.9700
N8—C11	1.3974 (17)		
C15—O1—C9	106.25 (10)	C14—C12—H12A	126.4
N8—O2—C10	112.43 (9)	O4—C13—N8	123.89 (13)
C7—C6—C9	124.66 (12)	O4—C13—C16	130.41 (12)
C7—C6—H6A	117.7	N8—C13—C16	105.69 (11)
C9—C6—H6A	117.7	C15—C14—C12	106.01 (12)
C6—C7—C10	121.09 (12)	C15—C14—H14A	127.0
C6—C7—H7A	119.5	C12—C14—H14A	127.0
C10—C7—H7A	119.5	C14—C15—O1	111.26 (12)
C13—N8—O2	120.55 (11)	C14—C15—H15A	124.4
C13—N8—C11	115.69 (11)	O1—C15—H15A	124.4
O2—N8—C11	120.93 (11)	C13—C16—C17	105.46 (11)
C12—C9—O1	109.22 (11)	C13—C16—H16A	110.6
C12—C9—C6	133.19 (13)	C17—C16—H16A	110.6
O1—C9—C6	117.58 (11)	C13—C16—H16B	110.6
O3—C10—O2	122.25 (12)	C17—C16—H16B	110.6
O3—C10—C7	130.02 (13)	H16A—C16—H16B	108.8
O2—C10—C7	107.72 (11)	C11—C17—C16	106.07 (11)
O5—C11—N8	124.31 (13)	C11—C17—H17A	110.5
O5—C11—C17	130.25 (13)	C16—C17—H17A	110.5
N8—C11—C17	105.42 (11)	C11—C17—H17B	110.5
C9—C12—C14	107.25 (12)	C16—C17—H17B	110.5
C9—C12—H12A	126.4	H17A—C17—H17B	108.7

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12A···O1[i]	0.93	2.48	3.3533 (17)	156.
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Fig. 1