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5-[(9*H*-Fluoren-9-ylidene)methyl]furan-2-carbonitrile

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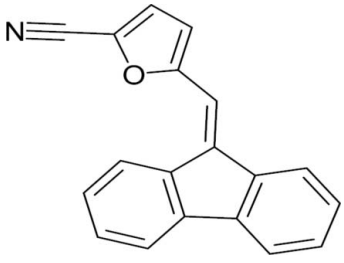
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.062; wR factor = 0.179; data-to-parameter ratio = 14.5.

The title compound, $C_{19}H_{11}NO$, is stabilized by one intramolecular C—H···O hydrogen bond. The compound can be synthesized in good yield (49%), by transformation of functional groups [starting with 5-(fluoren-9-ylidenemethyl)-furan-2-carbaldehyde]. The flourene and furan ring systems are nearly coplanar, with a dihedral angle of 6.36 (7)°.

Related literature

For a related structure, see: Britten *et al.* (2001). For related literature, see: Allen (2002); Leclerc (2001).



Experimental

Crystal data

$C_{19}H_{11}NO$
M_r = 269.29
Monoclinic, $P2_1/n$
a = 15.899 (3) Å
b = 5.6109 (11) Å
c = 15.664 (3) Å
β = 103.69 (3)°
V = 1357.6 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 293 K
0.32 × 0.07 × 0.05 mm

Data collection

Oxford Diffraction Gemini R CCD diffractometer
Absorption correction: analytical (Clark & Reid, 1995)
T_{min} = 0.921, T_{max} = 0.987
27628 measured reflections
2746 independent reflections
1524 reflections with $I > 2\sigma(I)$
R_{int} = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.179
S = 0.94
2746 reflections
190 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 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
C11—H11A···O1	0.93	2.27	3.034 (3)	140

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2007); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 1998); software used to prepare material for publication: *enCIFer* (Allen *et al.*, 2004).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2120).

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supporting information

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5-[(9*H*-Fluoren-9-ylidene)methyl]furan-2-carbonitrile

Lucia Perašínová, Anita Andicsová, Daniel Végh and Jozef Kožíšek

S1. Comment

Our synthetic research efforts have been focused to a set of multi-ring monomer systems based on furan and fluorene. Fluorene containing oligomeres are an important class of electroactive and photoactive materials. These compounds possesses exceptional electrooptical properties for applications in polymer light emitting diodes (PLEDs) and nanocomposite materials with advanced anticorrosive properties (Leclerc, 2001).

In the title compound the O1—C15 [1.359 (2) Å] and O1—C18 [1.381 (2) Å] bond lengths, are in a quite good agreement with similar furan compounds in the Cambridge Structural Database (CSD; Version 5.27, 2006 release; Allen, 2002)2-(1,2,3,5-Diselenadiazol-4-yl)-5-cyanofuran (Britten *et al.*, 2001; CSD refcode YIFHUQ) as representative example. The flourene moiety is almost planar with maximun deviation of 0.030 (2)Å for C13. The flourene and furan rings are nearly coplanar with a dihedral angle of 6.36 (7)°. In the crystal structure the molecular packing is stabilized by intramolecular hydrogen bond (Fig. 1).

S2. Experimental

A solution of 5-fluoren-9-ylidenemethyl-furan-2-carbaldehyde (0.0033 mol, 0.91 g), $NH_2OH.HCl$ (0.0039 mol, 0.3 g, 1.12 eq.) in *N*-Methyl-pyrrolidinone (5.5 ml) was heated at 110 - 115°C. Progress of the reaction was followed by TLC and after 8 h the mixture was poured into H_2O (100 ml) and extracted with EtOAc (2 *x* 50 ml). The combined layers EtOAc were dried (Na_2SO_4) and the solvent was evaporated *in vacuo*. Crude product could be purified by collumn chromatography using silikagel Merck 60 in toluene as eluent (40% yield) R_f = 0,51 (toluen). *M*.p.: 167–169°C.

^{1}H NMR (300 MHz, DMSO – d_6, p.p.m.): δ= 6.78 (d, 1H, J = 3.6 Hz), 7.15 (s, 1H), 7.31 - 7.29 (m, 1H), 7.45 - 7.33 (m, 4H), 7.72 - 7.66 (m, 3H), 8.51 (d, 1H, J = 7.65 Hz).

^{13}C-NMR (75 MHz, DMSO – d_6, p.p.m.) δ= 109.99, 111.77, 114.62, 119.80, 119.87, 120.33, 123.91, 125.74, 125.82, 127.20, 127.69, 129.69, 129.23, 129.88, 135.25, 138.06, 139.37, 139.62, 141.75, 156.19.

IR (KBr, cm^{-1}): 3136 (w), 3120 (vw), 3053(w), 2221(s, *v*C≡N), 1716(s, *v*(C=C)), 1633(*m*), 1611(*m*), 1600(m, *v*(C=C) aromatic), 1494(s, *v*(C=C) aromatic), 1469(w), 1448(*s*), 1354(*m*), 1297(*m*), 1290(*m*), 1274(m, ν_{as} (C—O—C)), 1263(*m*), 1198(w), 1180(*m*), 1152(*m*), 1138(w), 1111(*m*), 1098(w), 1029(s, ν_s (C—O—C)), 975(*m*), 966(*m*), 941(*m*), 917(*m*), 882(*m*), 871(*m*), 793(*v*s, γ(CCH)), 781(*v*s, γ(CCH)), 772(m, γ(CCH)), 737(m, γ(CCH)), 728(*v*s, γ(CCH)), 724(*v*s, γ(CCH)), 668(*m*), 645(*m*), 625(*m*), 580(w), 561(w), 524(w), 511(w), 473(*m*), 455(*m*), 442(vw), 432(vw), 401(*m*)

S3. Refinement

H atoms were placed in calculated positions and refined using a riding model, with C—H = 0.93 Å and U_{iso}(H) = 1.2 U_{eq}(C).

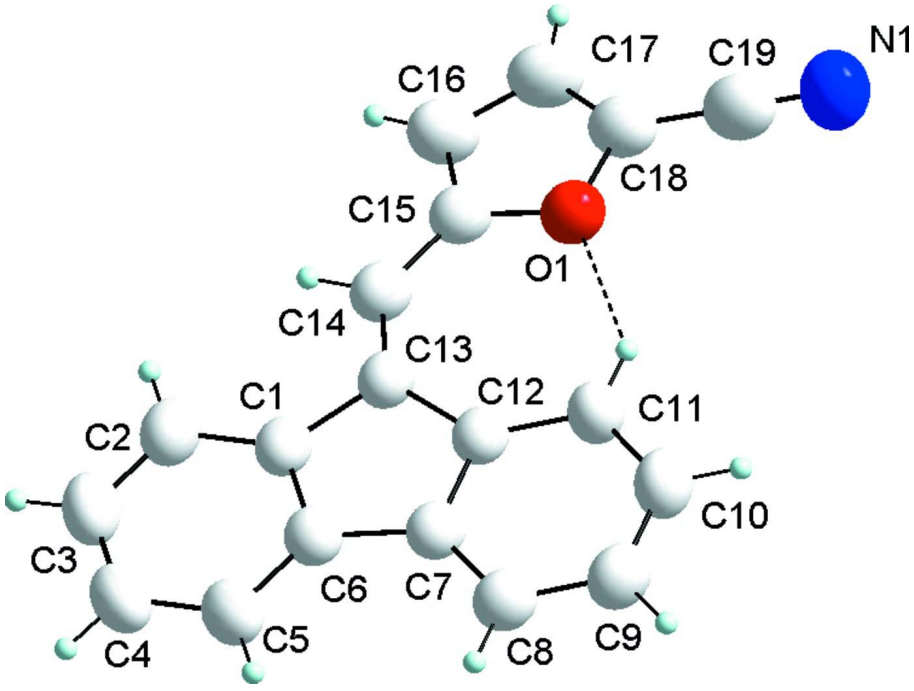

Figure 1

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Figure 2

The numbering scheme of 5-((9*H*-fluoren-9-ylidene) methyl) furan-2-carbonitrile. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen-bond interactions are indicated by dashed lines.

5-[(9*H*-Fluoren-9-ylidene)methyl]furan-2-carbonitrile

Crystal data

$C_{19}H_{11}NO$
$M_r = 269.29$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 15.899\ (3)$ Å
$b = 5.6109\ (11)$ Å
$c = 15.664\ (3)$ Å
$\beta = 103.69\ (3)°$
$V = 1357.6\ (5)$ Å^3
$Z = 4$
$F(000) = 560$
$D_x = 1.317$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7057 reflections
$\theta = 3.1–29.1°$
$\mu = 0.08$ mm^{-1}
$T = 293$ K
Block, yellow
0.32 × 0.07 × 0.05 mm

Data collection

Oxford Diffraction Gemini R CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Rotation method data acquisition using ω and phi scans
Absorption correction: analytical (Clark & Reid, 1995)
$T_{min} = 0.921$, $T_{max} = 0.987$
27628 measured reflections
2746 independent reflections
1524 reflections with $I > 2\sigma(I)$
$R_{int} = 0.043$
$\theta_{max} = 26.4°$, $\theta_{min} = 4.2°$
$h = -19\rightarrow19$
$k = -7\rightarrow7$
$l = -19\rightarrow19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.062$
$wR(F^2) = 0.179$
$S = 0.94$
2746 reflections
190 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
$w = 1/[\sigma^2(F_o^2) + (0.121P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

Special details

Experimental. face-indexed (*CrysAlis RED*; Oxford Diffraction, 2007)

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}
C1	0.17033 (14)	0.6172 (3)	0.88196 (13)	0.0544 (5)
C2	0.23065 (15)	0.6471 (4)	0.96102 (15)	0.0680 (6)
H2A	0.2259	0.7739	0.9978	0.082*
C3	0.29834 (16)	0.4852 (4)	0.98462 (15)	0.0737 (7)
H3A	0.3385	0.5046	1.0380	0.088*
C4	0.30765 (16)	0.2974 (4)	0.93141 (16)	0.0741 (7)
H4A	0.3537	0.1919	0.9484	0.089*
C5	0.24829 (15)	0.2672 (4)	0.85303 (15)	0.0651 (6)
H5A	0.2536	0.1400	0.8166	0.078*
C6	0.17991 (13)	0.4280 (3)	0.82809 (13)	0.0538 (5)
C7	0.10829 (13)	0.4398 (3)	0.75032 (13)	0.0521 (5)
C8	0.08974 (15)	0.2931 (4)	0.67850 (14)	0.0602 (6)
H8A	0.1248	0.1626	0.6747	0.072*
C9	0.01799 (15)	0.3426 (4)	0.61169 (14)	0.0660 (6)
H9A	0.0044	0.2436	0.5627	0.079*
C10	−0.03397 (15)	0.5382 (4)	0.61677 (14)	0.0686 (6)
H10A	−0.0818	0.5685	0.5709	0.082*
C11	−0.01619 (14)	0.6875 (4)	0.68787 (13)	0.0605 (6)
H11A	−0.0512	0.8188	0.6904	0.073*
C12	0.05522 (13)	0.6391 (3)	0.75621 (12)	0.0518 (5)
C13	0.09090 (13)	0.7573 (3)	0.84099 (13)	0.0532 (5)
C14	0.06414 (15)	0.9425 (3)	0.88210 (14)	0.0599 (6)
H14A	0.1013	0.9725	0.9365	0.072*
C15	−0.00743 (14)	1.1057 (3)	0.86312 (14)	0.0577 (6)

C16	−0.02863 (18)	1.2789 (4)	0.91496 (15)	0.0737 (7)
H16A	0.0017	1.3139	0.9720	0.088*
C17	−0.10323 (17)	1.3964 (4)	0.86887 (16)	0.0732 (7)
H17A	−0.1314	1.5227	0.8886	0.088*
C18	−0.12551 (15)	1.2896 (4)	0.79073 (16)	0.0647 (6)
C19	−0.19420 (18)	1.3303 (4)	0.7165 (2)	0.0743 (7)
N1	−0.25096 (16)	1.3660 (4)	0.65689 (17)	0.0953 (7)
O1	−0.06784 (9)	1.1091 (2)	0.78554 (9)	0.0624 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0575 (13)	0.0541 (11)	0.0489 (12)	−0.0072 (10)	0.0075 (10)	0.0027 (9)
C2	0.0730 (15)	0.0716 (13)	0.0545 (13)	−0.0084 (12)	0.0054 (12)	−0.0022 (11)
C3	0.0701 (15)	0.0820 (15)	0.0601 (14)	−0.0014 (13)	−0.0021 (12)	0.0087 (13)
C4	0.0673 (16)	0.0750 (15)	0.0735 (16)	0.0064 (12)	0.0036 (13)	0.0144 (13)
C5	0.0609 (14)	0.0672 (13)	0.0650 (14)	0.0063 (11)	0.0102 (12)	0.0039 (10)
C6	0.0540 (12)	0.0550 (11)	0.0524 (12)	−0.0026 (9)	0.0126 (10)	0.0015 (9)
C7	0.0565 (12)	0.0515 (11)	0.0487 (11)	−0.0040 (9)	0.0130 (10)	0.0011 (9)
C8	0.0649 (14)	0.0590 (12)	0.0572 (13)	−0.0001 (10)	0.0156 (11)	−0.0064 (10)
C9	0.0745 (15)	0.0676 (13)	0.0534 (13)	−0.0045 (12)	0.0101 (12)	−0.0116 (10)
C10	0.0710 (15)	0.0762 (14)	0.0521 (13)	0.0013 (12)	0.0015 (11)	−0.0044 (11)
C11	0.0613 (14)	0.0601 (12)	0.0542 (13)	0.0058 (10)	0.0020 (11)	−0.0042 (10)
C12	0.0540 (12)	0.0518 (11)	0.0485 (11)	−0.0052 (9)	0.0099 (10)	0.0009 (9)
C13	0.0578 (13)	0.0519 (10)	0.0478 (11)	−0.0054 (9)	0.0083 (10)	−0.0021 (9)
C14	0.0685 (14)	0.0564 (12)	0.0536 (12)	−0.0046 (10)	0.0119 (11)	−0.0030 (9)
C15	0.0656 (14)	0.0555 (11)	0.0526 (12)	−0.0046 (10)	0.0154 (11)	−0.0029 (9)
C16	0.0938 (19)	0.0651 (13)	0.0619 (14)	0.0079 (13)	0.0179 (13)	−0.0055 (11)
C17	0.0869 (18)	0.0621 (13)	0.0746 (16)	0.0074 (12)	0.0272 (14)	−0.0099 (11)
C18	0.0620 (14)	0.0586 (12)	0.0759 (16)	0.0016 (10)	0.0211 (13)	−0.0007 (11)
C19	0.0684 (17)	0.0674 (14)	0.0895 (19)	0.0048 (12)	0.0233 (15)	−0.0074 (13)
N1	0.0808 (16)	0.0981 (16)	0.0986 (18)	0.0118 (13)	0.0047 (15)	−0.0131 (13)
O1	0.0625 (10)	0.0616 (9)	0.0637 (10)	0.0017 (7)	0.0162 (8)	−0.0079 (7)

Geometric parameters (Å, º)

C1—C2	1.386 (3)	C10—C11	1.368 (3)
C1—C6	1.386 (3)	C10—H10A	0.9300
C1—C13	1.497 (3)	C11—C12	1.390 (3)
C2—C3	1.390 (3)	C11—H11A	0.9300
C2—H2A	0.9300	C12—C13	1.473 (3)
C3—C4	1.373 (3)	C13—C14	1.344 (3)
C3—H3A	0.9300	C14—C15	1.436 (3)
C4—C5	1.370 (3)	C14—H14A	0.9300
C4—H4A	0.9300	C15—O1	1.359 (2)
C5—C6	1.396 (3)	C15—C16	1.359 (3)
C5—H5A	0.9300	C16—C17	1.399 (3)
C6—C7	1.459 (3)	C16—H16A	0.9300

C7—C8	1.369 (3)	C17—C18	1.333 (3)
C7—C12	1.417 (3)	C17—H17A	0.9300
C8—C9	1.381 (3)	C18—O1	1.381 (2)
C8—H8A	0.9300	C18—C19	1.413 (4)
C9—C10	1.387 (3)	C19—N1	1.152 (3)
C9—H9A	0.9300		

C2—C1—C6	119.0 (2)	C11—C10—H10A	119.3
C2—C1—C13	130.90 (19)	C9—C10—H10A	119.3
C6—C1—C13	110.13 (18)	C10—C11—C12	118.7 (2)
C1—C2—C3	119.1 (2)	C10—C11—H11A	120.6
C1—C2—H2A	120.4	C12—C11—H11A	120.6
C3—C2—H2A	120.4	C11—C12—C7	119.62 (18)
C4—C3—C2	121.9 (2)	C11—C12—C13	132.57 (18)
C4—C3—H3A	119.1	C7—C12—C13	107.81 (17)
C2—C3—H3A	119.1	C14—C13—C12	133.0 (2)
C5—C4—C3	119.2 (2)	C14—C13—C1	122.10 (19)
C5—C4—H4A	120.4	C12—C13—C1	104.83 (17)
C3—C4—H4A	120.4	C13—C14—C15	136.0 (2)
C4—C5—C6	119.8 (2)	C13—C14—H14A	112.0
C4—C5—H5A	120.1	C15—C14—H14A	112.0
C6—C5—H5A	120.1	O1—C15—C16	107.54 (19)
C1—C6—C5	121.0 (2)	O1—C15—C14	123.50 (18)
C1—C6—C7	107.29 (17)	C16—C15—C14	129.0 (2)
C5—C6—C7	131.70 (19)	C15—C16—C17	109.2 (2)
C8—C7—C12	120.82 (19)	C15—C16—H16A	125.4
C8—C7—C6	129.27 (19)	C17—C16—H16A	125.4
C12—C7—C6	109.90 (17)	C18—C17—C16	105.7 (2)
C7—C8—C9	118.7 (2)	C18—C17—H17A	127.1
C7—C8—H8A	120.6	C16—C17—H17A	127.1
C9—C8—H8A	120.6	C17—C18—O1	110.3 (2)
C8—C9—C10	120.78 (19)	C17—C18—C19	132.0 (2)
C8—C9—H9A	119.6	O1—C18—C19	117.7 (2)
C10—C9—H9A	119.6	N1—C19—C18	178.7 (3)
C11—C10—C9	121.3 (2)	C15—O1—C18	107.23 (16)

C6—C1—C2—C3	−0.8 (3)	C8—C7—C12—C13	178.99 (18)
C13—C1—C2—C3	178.4 (2)	C6—C7—C12—C13	−1.8 (2)
C1—C2—C3—C4	0.6 (3)	C11—C12—C13—C14	3.9 (4)
C2—C3—C4—C5	−0.5 (4)	C7—C12—C13—C14	−175.5 (2)
C3—C4—C5—C6	0.5 (3)	C11—C12—C13—C1	−178.4 (2)
C2—C1—C6—C5	0.9 (3)	C7—C12—C13—C1	2.1 (2)
C13—C1—C6—C5	−178.45 (18)	C2—C1—C13—C14	−3.0 (3)
C2—C1—C6—C7	−179.96 (18)	C6—C1—C13—C14	176.26 (18)
C13—C1—C6—C7	0.7 (2)	C2—C1—C13—C12	179.0 (2)
C4—C5—C6—C1	−0.8 (3)	C6—C1—C13—C12	−1.7 (2)
C4—C5—C6—C7	−179.6 (2)	C12—C13—C14—C15	−1.0 (4)
C1—C6—C7—C8	179.8 (2)	C1—C13—C14—C15	−178.4 (2)

C5—C6—C7—C8	−1.2 (4)	C13—C14—C15—O1	−4.3 (4)
C1—C6—C7—C12	0.7 (2)	C13—C14—C15—C16	175.6 (2)
C5—C6—C7—C12	179.7 (2)	O1—C15—C16—C17	−0.8 (3)
C12—C7—C8—C9	−0.1 (3)	C14—C15—C16—C17	179.3 (2)
C6—C7—C8—C9	−179.15 (19)	C15—C16—C17—C18	0.5 (3)
C7—C8—C9—C10	0.5 (3)	C16—C17—C18—O1	0.0 (3)
C8—C9—C10—C11	−0.2 (3)	C16—C17—C18—C19	−179.5 (2)
C9—C10—C11—C12	−0.5 (3)	C16—C15—O1—C18	0.8 (2)
C10—C11—C12—C7	0.8 (3)	C14—C15—O1—C18	−179.31 (19)
C10—C11—C12—C13	−178.6 (2)	C17—C18—O1—C15	−0.5 (2)
C8—C7—C12—C11	−0.5 (3)	C19—C18—O1—C15	179.08 (19)
C6—C7—C12—C11	178.63 (17)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11*A*···O1	0.93	2.27	3.034 (3)	140