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2-(1-Hydroxy-2-oxo-1,2-dihydroacenaphthylen-1-yl)prop-2-enitrile

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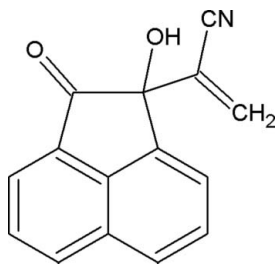
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.052; wR factor = 0.169; data-to-parameter ratio = 25.1.

In the title compound, $\text{C}_{15}\text{H}_9\text{NO}_2$, which is a Baylis–Hillman product, the five-membered ring adopts an envelope conformation. The crystal structure is stabilized by $\text{O}-\text{H}\cdots\text{O}$ intermolecular hydrogen bonds, linking the molecules into pairs around a center of symmetry.

Related literature

For related literature, see: Naveen *et al.* (2006); Ambrosi *et al.* (1994); Bernstein *et al.* (1995); Carta *et al.* (2002); Cremer & Pople (1975); Dawood *et al.* (1999); Nardelli (1983); Ohsumi *et al.* (1998); Sanna *et al.* (2000); Shi *et al.* (2002); Sonar *et al.* (2005).



Experimental

Crystal data

$\text{C}_{15}\text{H}_9\text{NO}_2$
 $M_r = 235.23$
Monoclinic, $P2_1/c$
 $a = 9.6980$ (3) Å
 $b = 10.1923$ (3) Å

$c = 12.1248$ (4) Å
 $\beta = 108.570$ (1)°
 $V = 1136.08$ (6) Å³
 $Z = 4$
Mo $K\alpha$ radiation

$\mu = 0.09$ mm⁻¹
 $T = 293$ (2) K

$0.25 \times 0.22 \times 0.19$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: none
16220 measured reflections
4109 independent reflections
2803 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.022$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.052$
 $wR(F^2) = 0.169$
 $S = 1.03$
4109 reflections
164 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.34$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.23$ e Å⁻³

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1}\cdots\text{O2}^i$	0.82	2.02	2.765 (1)	150

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

Data collection: APEX2 (Bruker, 2004); cell refinement: APEX2 and SAINT (Bruker, 2004); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: SHELXL97 and PLATON (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2178).

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supplementary materials

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2-(1-Hydroxy-2-oxo-1,2-dihydroacenaphthylen-1-yl)prop-2-enenitrile

M. Nizam Mohideen, P. S. Kannan, A. Subbiah Pandi, E. Ramesh and R. Raghunathan

Comment

Baylis-Hillman adducts are well known in organic synthesis because of their biological relevance (Shi *et al.*, 2002). Acrylonitriles represent an interesting class of biologically active compounds. Many derivatives of acrylonitriles have been shown to possess antitumor (Ohsumi *et al.*, 1998), antitubercular (Sanna *et al.*, 2000) and antiproliferative activities (Carta *et al.*, 2002). It is well known that acrylonitriles are useful intermediates in organic synthesis and are capable of undergoing many useful organic transformations (Ambrosi *et al.*, 1994), for example, into pyrazole, isoxazole and pyrimidine derivatives (Dawood *et al.*, 1999). Recently, the crystal structures of some bioactive heteroarylacrylonitriles have been reported (Sonar *et al.*, 2005; Naveen *et al.*, 2006). Against this background and in order to obtain detailed information on its molecular conformation, the structural determination of the title compound (I) has been carried out and the results are presented here.

In (I) (Fig. 1), the five-membered ring (atoms C1, C2, C3, C11, C12) of the acenaphthylene group adopts an envelope conformation with C1 being the out of plane atom. The puckering parameters are $q_2=0.086$ (2) Å and $\varphi=184.5$ (5)° (Cremer & Pople, 1975), and the lowest displacement asymmetry parameters $\Delta_S(C1)$ is 0.58 (1)° (Nardelli, 1983), with atoms O1 and O2 deviating by 1.021 (1) and 0.206 (1) Å from the least squares plane of the ring.

The value of the C14—C15 [1.439 (2) Å] bond length is slightly shorter than the value for C—C single bond because of conjugation effects. The O1—C1—C14—C15, C14—C1—C2=O2, and O1—C1—C2=O2 torsion angles [−51.3 (2), 73.2 (2), and −51.4 (1)°, respectively] describe the relationship of the two oxygen atoms to each other and to the nitrile moiety.

The crystal is stabilized by O—H···O intermolecular hydrogen bonds (Table 1) that generate centrosymmetric hydrogen-bonded dimers with a cyclic $R^2_2(10)$ ring system (Bernstein, *et al.*, 1995).

Experimental

A mixture of acenaphthenequinone, (5 mmol), acrylonitrile (10 mmol, 0.66 ml) and DABCO (0.1 mmol) was kept at room temperature for 12 h. This reaction mixture was diluted with ether (20 ml), washed with 2 N HCl and water. The organic layer was dried over anhydrous Na₂SO₄. Ether solvent was evaporated and the residue thus obtained, was purified by silica gel column chromatography to provide the corresponding Baylis-Hillman adduct. Single crystals of the title compound suitable for X-ray diffraction were obtained by slow evaporation of a solution in ethylacetate.

Refinement

All H atoms were fixed geometrically and allowed to ride on their parent C atoms, with O—H distance of 0.82 Å and $u_{iso}(H) = 1.5U_{eq}(O)$, and C—H distances of 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

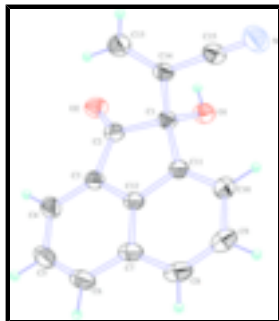


Fig. 1. The molecular configuration and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 50% probability level.

2-(1-Hydroxy-2-oxo-1,2-dihydroacenaphthylen-1-yl)prop-2-enitrile

Crystal data

$C_{15}H_9NO_2$	$Z = 4$
$M_r = 235.23$	$F_{000} = 488$
Monoclinic, $P2_1/c$	$D_x = 1.375 \text{ Mg m}^{-3}$
Hall symbol: $-P 2ybc$	Mo $K\alpha$ radiation
$a = 9.6980 (3) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 10.1923 (3) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$c = 12.1248 (4) \text{ \AA}$	$T = 293 (2) \text{ K}$
$\beta = 108.570 (1)^\circ$	Block, colourless
$V = 1136.08 (6) \text{ \AA}^3$	$0.25 \times 0.22 \times 0.19 \text{ mm}$

Data collection

'Bruker APEXII CCD area-detector diffractometer'	2803 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{\text{int}} = 0.022$
Monochromator: graphite	$\theta_{\text{max}} = 32.5^\circ$
$T = 293(2) \text{ K}$	$\theta_{\text{min}} = 2.7^\circ$
ω and π scan	$h = -14 \rightarrow 14$
Absorption correction: none	$k = -12 \rightarrow 15$
16220 measured reflections	$l = -11 \rightarrow 18$
4109 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.052$	$w = 1/[\sigma^2(F_o^2) + (0.0952P)^2 + 0.1059P]$
$wR(F^2) = 0.169$	where $P = (F_o^2 + 2F_c^2)/3$
	$(\Delta/\sigma)_{\text{max}} < 0.001$

$S = 1.03$ $\Delta\rho_{\max} = 0.34 \text{ e } \text{\AA}^{-3}$
 4109 reflections $\Delta\rho_{\min} = -0.23 \text{ e } \text{\AA}^{-3}$
 164 parameters Extinction correction: SHELXL97 (Sheldrick, 1997),
 $F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
 Primary atom site location: structure-invariant direct methods Extinction coefficient: 0.001
 Secondary atom site location: difference Fourier map

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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.16425 (10)	0.58159 (9)	0.12796 (9)	0.0528 (2)
H1	0.0786	0.5774	0.0876	0.079*
O2	0.13500 (10)	0.40059 (11)	-0.06878 (8)	0.0566 (3)
N	0.10202 (17)	0.43772 (17)	0.37898 (13)	0.0737 (4)
C1	0.22679 (11)	0.45533 (11)	0.13915 (10)	0.0401 (2)
C2	0.23713 (12)	0.40259 (11)	0.02072 (10)	0.0428 (3)
C3	0.38837 (12)	0.36483 (11)	0.03838 (11)	0.0429 (3)
C4	0.45780 (16)	0.31235 (13)	-0.03435 (13)	0.0542 (3)
H4	0.4062	0.2876	-0.1101	0.065*
C5	0.61016 (17)	0.29756 (14)	0.01034 (16)	0.0628 (4)
H5	0.6588	0.2605	-0.0369	0.075*
C6	0.68889 (15)	0.33555 (14)	0.12038 (16)	0.0603 (4)
H6	0.7892	0.3238	0.1459	0.072*
C7	0.62122 (13)	0.39236 (12)	0.19640 (13)	0.0493 (3)
C8	0.68822 (14)	0.44653 (15)	0.30866 (14)	0.0590 (4)
H8	0.7885	0.4409	0.3430	0.071*
C9	0.60619 (16)	0.50646 (16)	0.36602 (13)	0.0606 (4)
H9	0.6526	0.5431	0.4386	0.073*
C10	0.45315 (14)	0.51554 (14)	0.31996 (12)	0.0521 (3)
H10	0.3999	0.5569	0.3615	0.062*
C11	0.38503 (12)	0.46227 (11)	0.21295 (10)	0.0412 (2)
C12	0.46947 (11)	0.40296 (11)	0.15199 (11)	0.0409 (2)
C13	0.09463 (17)	0.24185 (14)	0.14739 (14)	0.0569 (3)
H13A	0.0459	0.1891	0.1855	0.068*
H13B	0.1089	0.2127	0.0792	0.068*

supplementary materials

C14	0.14353 (12)	0.35824 (12)	0.19015 (10)	0.0421 (3)
C15	0.12052 (13)	0.40191 (13)	0.29585 (12)	0.0503 (3)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0440 (5)	0.0402 (4)	0.0729 (6)	0.0072 (3)	0.0169 (4)	0.0031 (4)
O2	0.0430 (5)	0.0725 (7)	0.0493 (5)	-0.0024 (4)	0.0075 (4)	0.0010 (4)
N	0.0696 (9)	0.0951 (11)	0.0627 (8)	0.0041 (8)	0.0298 (7)	-0.0049 (7)
C1	0.0331 (5)	0.0387 (5)	0.0491 (6)	0.0025 (4)	0.0141 (4)	0.0006 (4)
C2	0.0377 (5)	0.0423 (6)	0.0486 (6)	-0.0022 (4)	0.0138 (5)	0.0027 (4)
C3	0.0401 (5)	0.0387 (5)	0.0530 (6)	-0.0011 (4)	0.0189 (5)	0.0008 (4)
C4	0.0609 (8)	0.0451 (6)	0.0672 (8)	-0.0044 (5)	0.0353 (6)	-0.0038 (6)
C5	0.0637 (8)	0.0455 (7)	0.0974 (11)	0.0035 (6)	0.0514 (9)	0.0006 (7)
C6	0.0409 (6)	0.0469 (7)	0.1014 (12)	0.0050 (5)	0.0345 (7)	0.0088 (7)
C7	0.0346 (5)	0.0411 (6)	0.0722 (8)	0.0005 (4)	0.0169 (5)	0.0104 (5)
C8	0.0352 (6)	0.0570 (7)	0.0756 (9)	-0.0049 (5)	0.0046 (6)	0.0110 (7)
C9	0.0514 (7)	0.0597 (8)	0.0600 (8)	-0.0119 (6)	0.0025 (6)	-0.0017 (6)
C10	0.0483 (7)	0.0514 (7)	0.0549 (7)	-0.0049 (5)	0.0142 (6)	-0.0061 (5)
C11	0.0349 (5)	0.0389 (5)	0.0496 (6)	-0.0010 (4)	0.0135 (4)	0.0007 (4)
C12	0.0325 (5)	0.0363 (5)	0.0548 (6)	-0.0001 (4)	0.0151 (4)	0.0041 (4)
C13	0.0596 (8)	0.0479 (7)	0.0703 (8)	-0.0060 (6)	0.0305 (7)	-0.0010 (6)
C14	0.0325 (5)	0.0450 (6)	0.0502 (6)	0.0038 (4)	0.0149 (4)	0.0023 (4)
C15	0.0402 (6)	0.0577 (7)	0.0548 (7)	0.0026 (5)	0.0180 (5)	0.0023 (5)

Geometric parameters (\AA , $^\circ$)

O1—C1	1.4107 (14)	C6—H6	0.9300
O1—H1	0.8200	C7—C12	1.4009 (16)
O2—C2	1.2143 (15)	C7—C8	1.420 (2)
N—C15	1.1387 (19)	C8—C9	1.357 (2)
C1—C11	1.5120 (15)	C8—H8	0.9300
C1—C14	1.5270 (16)	C9—C10	1.4125 (19)
C1—C2	1.5660 (17)	C9—H9	0.9300
C2—C3	1.4646 (16)	C10—C11	1.3669 (18)
C3—C4	1.3767 (17)	C10—H10	0.9300
C3—C12	1.4060 (17)	C11—C12	1.4029 (16)
C4—C5	1.411 (2)	C13—C14	1.3208 (19)
C4—H4	0.9300	C13—H13A	0.9300
C5—C6	1.365 (2)	C13—H13B	0.9300
C5—H5	0.9300	C14—C15	1.4395 (18)
C6—C7	1.415 (2)		
C1—O1—H1	109.5	C6—C7—C8	128.18 (12)
O1—C1—C11	109.78 (9)	C9—C8—C7	120.14 (12)
O1—C1—C14	111.32 (9)	C9—C8—H8	119.9
C11—C1—C14	111.01 (9)	C7—C8—H8	119.9
O1—C1—C2	112.09 (9)	C8—C9—C10	122.71 (13)
C11—C1—C2	102.09 (8)	C8—C9—H9	118.6

C14—C1—C2	110.21 (9)	C10—C9—H9	118.6
O2—C2—C3	128.32 (12)	C11—C10—C9	118.60 (13)
O2—C2—C1	123.58 (11)	C11—C10—H10	120.7
C3—C2—C1	108.04 (10)	C9—C10—H10	120.7
C4—C3—C12	119.98 (11)	C10—C11—C12	118.98 (11)
C4—C3—C2	132.82 (12)	C10—C11—C1	132.30 (11)
C12—C3—C2	107.00 (10)	C12—C11—C1	108.71 (10)
C3—C4—C5	117.49 (14)	C7—C12—C11	123.28 (12)
C3—C4—H4	121.3	C7—C12—C3	123.14 (11)
C5—C4—H4	121.3	C11—C12—C3	113.39 (10)
C6—C5—C4	122.44 (13)	C14—C13—H13A	120.0
C6—C5—H5	118.8	C14—C13—H13B	120.0
C4—C5—H5	118.8	H13A—C13—H13B	120.0
C5—C6—C7	121.45 (12)	C13—C14—C15	119.28 (12)
C5—C6—H6	119.3	C13—C14—C1	126.38 (11)
C7—C6—H6	119.3	C15—C14—C1	114.33 (10)
C12—C7—C6	115.45 (13)	N—C15—C14	179.29 (16)
C12—C7—C8	116.26 (12)		
O1—C1—C2—O2	-51.37 (15)	C2—C1—C11—C10	170.29 (13)
C11—C1—C2—O2	-168.79 (11)	O1—C1—C11—C12	-127.22 (10)
C14—C1—C2—O2	73.20 (14)	C14—C1—C11—C12	109.27 (11)
O1—C1—C2—C3	125.95 (10)	C2—C1—C11—C12	-8.16 (11)
C11—C1—C2—C3	8.53 (11)	C6—C7—C12—C11	-176.91 (11)
C14—C1—C2—C3	-109.48 (10)	C8—C7—C12—C11	-0.44 (17)
O2—C2—C3—C4	-3.5 (2)	C6—C7—C12—C3	-2.27 (17)
C1—C2—C3—C4	179.33 (13)	C8—C7—C12—C3	174.20 (11)
O2—C2—C3—C12	171.23 (12)	C10—C11—C12—C7	1.61 (18)
C1—C2—C3—C12	-5.93 (12)	C1—C11—C12—C7	-179.69 (10)
C12—C3—C4—C5	0.83 (18)	C10—C11—C12—C3	-173.50 (11)
C2—C3—C4—C5	175.02 (12)	C1—C11—C12—C3	5.19 (13)
C3—C4—C5—C6	-1.4 (2)	C4—C3—C12—C7	1.03 (18)
C5—C6—C7—C12	1.73 (19)	C2—C3—C12—C7	-174.52 (10)
C5—C6—C7—C8	-174.25 (14)	C4—C3—C12—C11	176.15 (11)
C12—C7—C8—C9	-1.10 (19)	C2—C3—C12—C11	0.60 (13)
C6—C7—C8—C9	174.85 (13)	O1—C1—C14—C13	129.43 (14)
C7—C8—C9—C10	1.5 (2)	C11—C1—C14—C13	-107.95 (14)
C8—C9—C10—C11	-0.3 (2)	C2—C1—C14—C13	4.42 (17)
C9—C10—C11—C12	-1.22 (19)	O1—C1—C14—C15	-51.36 (13)
C9—C10—C11—C1	-179.54 (12)	C11—C1—C14—C15	71.26 (12)
O1—C1—C11—C10	51.23 (17)	C2—C1—C14—C15	-176.38 (10)
C14—C1—C11—C10	-72.27 (16)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1 \cdots O2 ⁱ	0.82	2.02	2.765 (1)	150

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

Fig. 1

