Refinement

Refinement on F^2
$R(F) = 0.0335$
$wR(F^2) = 0.0916$
$S = 1.031$
1551 reflections
152 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0542P)^2 + 0.9685P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = -0.034$

$\Delta\rho_{max} = 0.169$ e Å^{-3}
$\Delta\rho_{min} = -0.178$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.038 (3)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
O1	0.60988 (9)	0.03455 (12)	0.64819 (9)	0.0466 (4)
N1	0.65007 (10)	0.23416 (13)	0.19492 (10)	0.0402 (4)
N2	0.65184 (11)	0.33591 (14)	0.12573 (11)	0.0462 (4)
N3	0.64398 (11)	0.45117 (14)	0.16673 (11)	0.0470 (4)
N4	0.63653 (10)	0.42559 (13)	0.26429 (10)	0.0398 (4)
C1	0.63337 (10)	0.22477 (14)	0.37749 (11)	0.0311 (3)
C2	0.60061 (11)	0.2934 (2)	0.44894 (13)	0.0380 (4)
C3	0.59357 (12)	0.2288 (2)	0.53898 (13)	0.0394 (4)
C4	0.61844 (10)	0.09348 (15)	0.55846 (11)	0.0339 (4)
C5	0.65219 (11)	0.0242 (2)	0.48882 (12)	0.0365 (4)
C6	0.66007 (11)	0.08935 (15)	0.39946 (12)	0.0360 (4)
C7	0.64051 (10)	0.29221 (14)	0.28151 (11)	0.0325 (3)
O*W*1	1/2	0.1661 (2)	3/4	0.0446 (4)
O*W*2	0.37968 (13)	0.36321 (14)	0.61262 (12)	0.0699 (5)

Table 2. *Selected geometric parameters (Å, °)*

O1—C4	1.358 (2)	N3—N4	1.344 (2)
N1—C7	1.325 (2)	N4—C7	1.338 (2)
N1—N2	1.360 (2)	C1—C7	1.459 (2)
N2—N3	1.286 (2)		
C7—N1—N2	106.3 (1)	O1—C4—C5	122.4 (1)
N3—N2—N1	110.7 (1)	O1—C4—C3	117.6 (1)
N2—N3—N4	106.4 (1)	N1—C7—N4	107.4 (1)
C7—N4—N3	109.3 (1)	N1—C7—C1	127.0 (1)
C2—C1—C7	121.1 (1)	N4—C7—C1	125.6 (1)
C6—C1—C7	120.2 (1)		

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—HO1···N1^{i}	0.85 (2)	1.91 (2)	2.743 (2)	166 (2)
N4—HN4···O*W*2^{ii}	0.96 (2)	1.75 (2)	2.706 (2)	178 (2)
O*W*1—H1*W*1···O1^{iii}	0.88 (2)	1.94 (2)	2.805 (1)	167 (2)
O*W*2—H1*W*2···N2^{iv}	0.79 (2)	2.18 (2)	2.962 (2)	172 (2)
O*W*2—H2*W*2···O*W*1	0.85 (2)	1.92 (2)	2.764 (2)	169 (2)

Symmetry codes: (i) $x, -y, \frac{1}{2}+z$; (ii) $1-x, 1-y, 1-z$; (iii) $1-x, y, \frac{3}{2}-z$; (iv) $1-x, y, \frac{1}{2}-z$.

One water molecule was placed in a special position. All H atoms were located from $\Delta\rho$ maps and refined isotropically with a common displacement factor.

Data collection: *CAD*-4 *Express* (Enraf–Nonius, 1992). Cell refinement: *MolEN* (Fair, 1990). Data reduction: *MolEN*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93. The calculations were performed on a DEC 3000 AXP and PC/486 computer.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1405). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1997). **C53**, 144–146

1,2-Dihydronaphtho[3,4-*b*]cyclopenteno-[1,2-*e*]-4*H*-pyran-4-one

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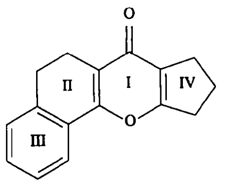
Abstract

In the title compound, 2,3,5,6-tetrahydrocyclopenta[1,2-*e*]naphtho[1,2-*b*]pyran-4(1*H*)-one, $C_{16}H_{14}O_2$, the planar pyrone ring makes dihedral angles of less than 10° with the best planes of each of the other three rings. The maximum deviation for each of the planes through the non-H atoms is less than 0.225 (4) Å. Molecules in the crystal are packed in columns. Molecular-dynamics cal-

culations of the interaction energy between neighbouring molecules within the columns gave a rather large value of *ca* 25 kJ mol^{-1}. Only weak interactions were found between the columns.

Comment

We attempted to synthesize a different compound by the reaction of adipyl chloride with tetralone pyrrolidine enamine (Hûnig & Lendle, 1960). However, we obtained mainly the title compound, (1), whose molecular structure was finally confirmed by this single-crystal determination after conventional UV, IR, NMR, MS and elemental analyses.



(1)

There are no significant differences between the geometries of the molecules in the asymmetric unit. Bond lengths conform to expected values and do not suggest that pyrone ring (I) is conjugated with aromatic ring (III). Rings (I) and (III) are planar, while there are small deviations from planarity in rings (II) and (IV) consistent with their chemistry. The best planes of rings (II), (III) and (IV) make angles of less than 10° with that of pyrone ring (I).

The molecules are stacked in columns along the *a* axis, with neighbouring molecules antiparallel. There are some quite short contacts between molecules in neighbouring columns (see Table 2) involving C—H groups and suggesting C—H···O hydrogen bonding (Taylor & Kennard, 1982; Berkovitch-Yellin & Leíserowitz, 1984; Sharma & Desiraju, 1994). The interaction energies between a molecule in the stack and the ones above and below it, calculated with *OPEC* (Gavezzotti, 1983), are 25.2 and 24.8 kJ mol^{-1}. Since there can be no great π–π interactions, this must be the result of a number of favourable van der Waals contacts.

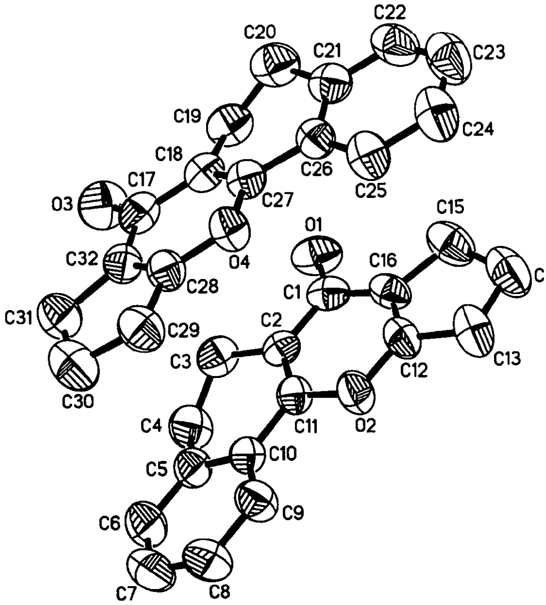


Fig. 1. The molecular structure of (1) showing 50% probability displacement ellipsoids.

Experimental

To a stirred solution containing chloroform (60 ml), triethylamine (2 ml) and tetralone pyrrolidine enamine (1.5 g, 0.007 mol), a solution of adipyl chloride (1.54 g, 0.084 mol) and chloroform (14 ml) were added dropwise at room temperature over a period of 1 h. The mixture was refluxed for 12 h, cooled and hydrolyzed with hydrochloric acid. The organic layer was separated, dried over sodium sulfate and distilled under reduced pressure. The crude product was purified by column chromatography, then recrystallized from acetone–petroleum ether.

Crystal data

$C_{16}H_{14}O_2$
M_r = 238.27
Monoclinic
$P2_1/n$
a = 8.2300 (10) Å
b = 18.205 (2) Å
c = 16.384 (2) Å
β = 99.000 (10)°
V = 2424.5 (5) Å^3
Z = 8
D_x = 1.306 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 26 reflections
θ = 1.68–25.00°
μ = 0.85 mm^{-1}
T = 288 (2) K
Tabular
0.60 × 0.60 × 0.36 mm
Colourless

Data collection

Siemens *P*4 diffractometer
ω scans
Absorption correction: none
4023 measured reflections
3590 independent reflections
2098 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int}$ = 0.0179

$\theta_{\max}$ = 23.50°
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 20$
$l = -18 \rightarrow 18$
3 standard reflections monitored every 200 reflections intensity decay: 0.5%

Refinement

Refinement on F^2
$R(F)$ = 0.0377
$wR(F^2)$ = 0.1093
S = 0.850
3589 reflections
438 parameters
H atoms refined isotropically
$w = 1/[\sigma^2(F_o^2) + (0.05P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max}$ = −0.101

$\Delta\rho_{\max}$ = 0.173 e Å^{-3}
$\Delta\rho_{\min}$ = −0.111 e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.0200 (10)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U^{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
O1	0.4364 (2)	0.73943 (8)	0.21498 (10)	0.0788 (5)
O2	0.6081 (2)	0.92946 (7)	0.12654 (8)	0.0589 (4)
O3	0.1708 (2)	0.85553 (9)	0.39600 (10)	0.0845 (5)
O4	0.1563 (2)	0.96869 (7)	0.17986 (8)	0.0550 (4)
C1	0.4874 (2)	0.79699 (12)	0.18767 (15)	0.0568 (6)
C2	0.5726 (2)	0.85353 (11)	0.24156 (12)	0.0489 (5)
C3	0.5976 (3)	0.84393 (14)	0.33344 (14)	0.0644 (7)
C4	0.7410 (4)	0.8864 (2)	0.3783 (2)	0.0775 (8)
C5	0.7585 (3)	0.96239 (12)	0.34378 (13)	0.0567 (6)
C6	0.8342 (3)	1.0199 (2)	0.3915 (2)	0.0759 (8)
C7	0.8631 (3)	1.0864 (2)	0.3577 (2)	0.0784 (8)
C8	0.8185 (3)	1.09725 (15)	0.2748 (2)	0.0721 (7)
C9	0.7420 (3)	1.04213 (12)	0.22619 (14)	0.0608 (6)
C10	0.7108 (2)	0.97487 (10)	0.25988 (12)	0.0470 (5)
C11	0.6272 (2)	0.91542 (11)	0.21007 (12)	0.0477 (5)
C12	0.5320 (3)	0.87687 (12)	0.07562 (13)	0.0570 (6)
C13	0.5092 (4)	0.8881 (2)	−0.01503 (15)	0.0765 (8)
C14	0.4374 (4)	0.8136 (2)	−0.0468 (2)	0.0889 (9)
C15	0.3982 (4)	0.76975 (15)	0.0274 (2)	0.0777 (8)
C16	0.4735 (2)	0.81390 (11)	0.10109 (14)	0.0563 (6)
C17	0.1686 (3)	0.88998 (13)	0.33063 (13)	0.0589 (6)
C18	0.1062 (2)	0.85825 (10)	0.25028 (13)	0.0515 (5)
C19	0.0524 (4)	0.77859 (13)	0.2427 (2)	0.0701 (7)
C20	−0.0739 (4)	0.7638 (2)	0.1675 (2)	0.0774 (8)
C21	−0.0406 (3)	0.80133 (12)	0.09117 (15)	0.0604 (6)
C22	−0.0980 (3)	0.7739 (2)	0.0125 (2)	0.0754 (8)
C23	−0.0772 (3)	0.8120 (2)	−0.0575 (2)	0.0824 (9)
C24	0.0017 (3)	0.8788 (2)	−0.0510 (2)	0.0771 (8)
C25	0.0614 (3)	0.90731 (13)	0.02640 (14)	0.0635 (7)
C26	0.0408 (2)	0.86913 (11)	0.09761 (13)	0.0519 (6)
C27	0.1009 (2)	0.89716 (10)	0.18027 (12)	0.0487 (5)
C28	0.2194 (3)	0.99836 (11)	0.25389 (13)	0.0523 (5)
C29	0.2825 (4)	1.07509 (13)	0.2588 (2)	0.0665 (7)
C30	0.3543 (4)	1.0820 (2)	0.3498 (2)	0.0834 (9)
C31	0.3028 (4)	1.0141 (2)	0.3941 (2)	0.0786 (8)
C32	0.2279 (2)	0.96411 (11)	0.32595 (12)	0.0541 (6)

Table 2. *Contact distances (Å)*

O1···C7$^{\rm i}$	3.538 (5)	O3···C22$^{\rm iv}$	3.418 (4)
O1···C8$^{\rm i}$	3.270 (4)	C25···C25$^{\rm v}$	3.590 (5)
O1···C29$^{\rm ii}$	3.553 (5)	O1···C19	3.339 (5)
O3···C4$^{\rm iii}$	3.548 (4)		

Symmetry codes: (i) $\frac{3}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (ii) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (iii) $x - 1, y, z$; (iv) $\frac{1}{2} + x, \frac{3}{2} - y, \frac{1}{2} + z$; (v) $-x, 2 - y, -z$.

Data collection: *XSCANS* (Siemens, 1992). Cell refinement: *XSCANS*. Data reduction: *SHELXTL-Plus* (Sheldrick, 1991). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXTL-Plus*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HA1171). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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