Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1716 measured reflections
1683 independent reflections
824 observed reflections [$F > 4\sigma(F)$]

$\theta_{\max} = 23°$
$h = 0 \rightarrow 16$
$k = 0 \rightarrow 6$
$l = -13 \rightarrow 13$
3 standard reflections
frequency: 120 min
intensity decay: <1%

Refinement

Refinement on F
$R = 0.0664$
$wR = 0.0633$
$S = 2.7130$
824 reflections
154 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F) + 0.000116F^2]$
$(\Delta/\sigma)_{\max} = 0.026$
$\Delta\rho_{\max} = 0.287$ e Å^{-3}
$\Delta\rho_{\min} = -0.229$ e Å^{-3}
Atomic scattering factors from *SHELX76* (Sheldrick, 1976)

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	$U_{\rm eq}$
O	0.4275 (3)	−0.8174 (10)	0.8124 (3)	0.137 (2)
N	0.4157 (3)	0.1207 (7)	0.1614 (3)	0.076 (2)
C(1)	0.4083 (5)	0.2737 (12)	0.0758 (5)	0.088 (3)
C(2)	0.3437 (5)	0.4575 (13)	0.0592 (6)	0.095 (3)
C(3)	0.2829 (5)	0.4870 (12)	0.1341 (5)	0.093 (3)
C(4)	0.2883 (4)	0.3320 (11)	0.2226 (5)	0.079 (2)
C(5)	0.3549 (3)	0.1529 (9)	0.2347 (4)	0.067 (2)
C(6)	0.3629 (3)	−0.0157 (9)	0.3262 (4)	0.071 (2)
C(7)	0.3678 (3)	−0.1591 (10)	0.3991 (4)	0.075 (2)
C(8)	0.3744 (3)	−0.3337 (11)	0.4796 (4)	0.088 (2)
C(9)	0.3793 (3)	−0.4890 (13)	0.5448 (5)	0.106 (3)
C(10)	0.3873 (6)	−0.7073 (16)	0.6253 (6)	0.116 (4)
C(11)	0.4294 (6)	−0.6120 (17)	0.7308 (6)	0.123 (4)

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

N—C(1)	1.327 (6)	C(6)—C(7)	1.177 (6)
N—C(5)	1.345 (5)	C(7)—C(8)	1.359 (8)
C(1)—C(2)	1.371 (7)	C(8)—C(9)	1.154 (7)
C(2)—C(3)	1.357 (7)	C(9)—C(10)	1.533 (9)
C(3)—C(4)	1.363 (7)	C(10)—C(11)	1.454 (8)
C(4)—C(5)	1.370 (6)	O—C(11)	1.495 (8)
C(5)—C(6)	1.436 (6)		
C(1)—N—C(5)	116.3 (5)	C(4)—C(5)—C(6)	121.7 (5)
N—C(1)—C(2)	124.1 (6)	C(5)—C(6)—C(7)	177.9 (5)
C(1)—C(2)—C(3)	118.7 (6)	C(6)—C(7)—C(8)	177.1 (5)
C(2)—C(3)—C(4)	118.7 (7)	C(7)—C(8)—C(9)	177.3 (6)
C(3)—C(4)—C(5)	119.7 (6)	C(8)—C(9)—C(10)	176.1 (7)
N—C(5)—C(4)	122.6 (5)	C(9)—C(10)—C(11)	106.5 (7)
N—C(5)—C(6)	115.7 (5)	O—C(11)—C(10)	106.5 (7)

D—H···A	D—H	H···A	D···A	D—H···A
O—H(O)···N$^{\rm i}$	0.90 (8)	1.95 (8)	2.807 (6)	157 (8)

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

Preliminary photographic studies on the crystal using rotation, Weissenberg and precession cameras revealed the approximate unit-cell dimensions and the reflection conditions $h0l$ for $h = 2n$ and $h00$ for $h = 2n$, which indicated a uniquely defined centrosymmetric monoclinic space group $P2_1/a$ (C_{2h}^5). The independent reflections were collected using an ω-scan width of $(0.8 + 0.35\tan\theta)°$. During the data collection, one orientation reflection ($80\bar{1}$) was monitored every 200 reflections and the three standard reflections, ($\bar{7}2\bar{3}$), ($80\bar{2}$) and ($\bar{4}3\bar{4}$), varied by less than 1%. Intensity data were corrected for Lp factors with the program *SDP* (Enraf–Nonius, 1985). An asymmetric unit (Suh *et al.*, 1993) of X-ray intensity data were collected. *SDP* was used for data collection, cell refinement and data reduction. The structure was solved by direct methods using *SHELXS86* (Sheldrick, 1985). The *SHELX76* program (Sheldrick, 1976) was used for full-matrix least-squares refinement with anisotropic displacement factors for all the non-H atoms. All H-atom positions was found from difference Fourier syntheses and refined isotropically. Geometric calculations were carried out using the *GEOM* program (Gilmore, 1979).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates, together with extra unit-cell packing diagrams, have been deposited with the IUCr (Reference: KH1007). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Barrow, M. J., Milburn, G. H. W. & Zeng, Z. (1994). *Acta Cryst.* C**50**, 650–652.

Baughman, R. H. (1974). *J. Polym. Sci. Polym. Phys. Ed.* **12**, 1511–1535.

Enraf–Nonius (1985). *Structure Determination Package.* Enraf–Nonius, Delft, The Netherlands.

Gilmore, C. J. (1979). *GEOM. Program for the Calculation of Molecular and Crystal Geometry.* Univ. of Glasgow, Scotland.

Johnson, C. K. (1976). *ORTEPII.* Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination.* Univ. of Cambridge, England.

Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures.* Univ. of Göttingen, Germany.

Suh, I. H., Kim, K. J., Choo, G. H., Lee, J. H., Choh, S. H. & Kim, M. J. (1993). *Acta Cryst.* A**49**, 369–371.

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α-Naphthoflavone†

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Abstract

The crystal structure of the title compound, 2-phenyl-4*H*-naphtho[1,2-*b*]pyran-4-one, $C_{19}H_{12}O_2$, is stabilized by both intra- and intermolecular C—H···O hydrogen bonds.

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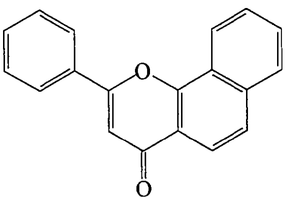
Comment

The crystal and molecular structure of the title compound (1) was first reported by Rossi & Glusker (1979) without atomic coordinates. The present work describes the structure determination and crystal packing in detail.



(1)

Bond distances and angles within the molecule are quite regular with average C—C distances in the phenyl and naphthyl rings of 1.384 (3) and 1.393 (3) Å, respectively. The phenyl and benzopyrone rings are planar with maximum deviations of 0.007 and 0.025 Å, respectively. The interplanar angle between the phenyl ring (C1–C6) and the benzopyrone ring (O1, C7–C13, C18–C19) is 24.55 (9)°. The conformation of the molecule is similar to those of some related flavone structures described in the literature (Shoja, 1994; Neuman *et al.*, 1989). The O atom O1 is involved in two intramolecular hydrogen bonds: one with atom H6 [C6···O1 2.737 (2), H6···O1 2.43 (2) Å, C6—H6···O1 98 (1)°] and the second with atom H17 [C17···O1 2.778 (2), H17···O1 2.47 (2) Å, C17—H17···O1 99 (1)°]. Also, the carbonyl atom O2 participates in one intramolecular [C11···O2 2.873 (3), H11···O2 2.57 (2) Å, C11—H11···O2 98 (1)°] and two intermolecular hydrogen bonds [C3···O2($x-\frac{1}{2}$, y, $\frac{3}{2}-z$) 3.308 (3), H3···O2 2.51 (3) Å, C3—H3···O2 139 (2)°; C16···O2($x-\frac{1}{2}$, $\frac{1}{2}-y$, $1-z$) 3.556 (3), H16···O2 2.60 (2) Å, C16—H16···O2 163 (2)°] (Desiraju, 1991).

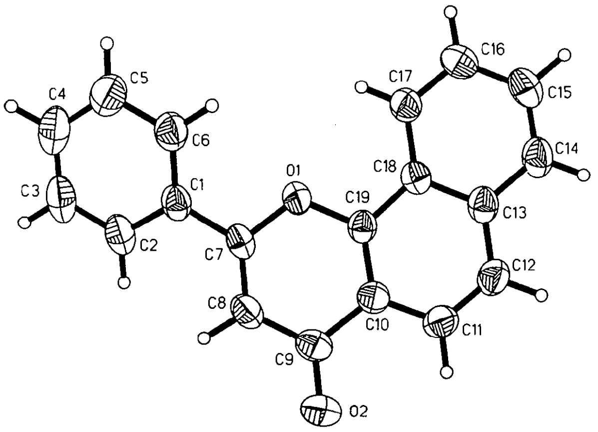


Fig. 1. The molecular structure of the title compound with the atom labelling and 50% probability displacement ellipsoids shown.

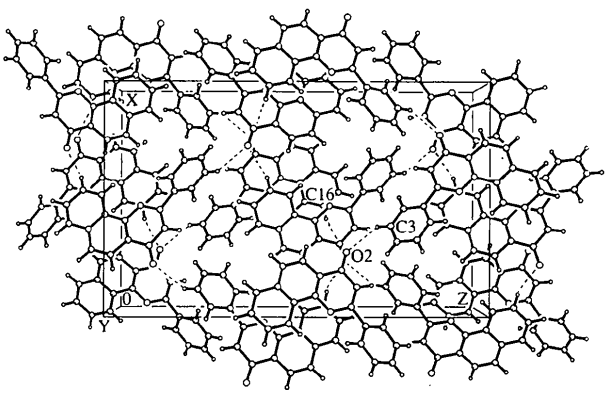


Fig. 2. A perspective drawing of the packing arrangement.

Experimental

Crystals of the title compound were obtained from a benzene solution of the commercially available material by slow evaporation of the solvent.

Crystal data

$C_{19}H_{12}O_2$
M_r = 272.29
Orthorhombic
Pbca
a = 14.788 (4) Å
b = 7.470 (2) Å
c = 24.188 (4) Å
V = 2672.0 (11) Å^3
Z = 8
D_x = 1.354 Mg m^{-3}

Cu $K\alpha$ radiation
λ = 1.54178 Å
Cell parameters from 25 reflections
θ = 15–45°
μ = 0.696 mm^{-1}
T = 293 (2) K
Needle
0.3 × 0.15 × 0.1 mm
Pale yellow

Data collection

*P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
2382 measured reflections
1769 independent reflections
1487 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int}$ = 0.0267

$\theta_{\rm max}$ = 56.60°
$h = -1 \rightarrow 16$
$k = -1 \rightarrow 8$
$l = -26 \rightarrow 1$
3 standard reflections monitored every 100 reflections
intensity decay: 2%

Refinement

Refinement on F^2
$R(F)$ = 0.0399
$wR(F^2)$ = 0.1034
S = 1.112
1769 reflections
238 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0462P)^2 + 0.5380P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max}$ = 0.001
$\Delta\rho_{\rm max}$ = 0.123 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.185 e Å^{-3}
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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}
C1	−0.03036 (13)	0.1485 (3)	0.67626 (7)	0.0426 (5)
C2	−0.0407 (2)	0.0483 (3)	0.72444 (8)	0.0552 (6)
C3	−0.1144 (2)	0.0748 (4)	0.75841 (10)	0.0645 (7)
C4	−0.1780 (2)	0.2022 (4)	0.74550 (10)	0.0662 (7)
C5	−0.1689 (2)	0.3031 (4)	0.69802 (10)	0.0624 (7)
C6	−0.09588 (14)	0.2745 (3)	0.66298 (9)	0.0518 (6)
C7	0.04832 (13)	0.1191 (3)	0.64003 (7)	0.0402 (5)
C8	0.12805 (13)	0.0491 (3)	0.65484 (8)	0.0462 (5)
C9	0.19999 (14)	0.0180 (3)	0.61580 (8)	0.0446 (5)
C10	0.17788 (12)	0.0676 (3)	0.55896 (7)	0.0402 (5)
C11	0.23877 (14)	0.0361 (3)	0.51489 (8)	0.0486 (6)
C12	0.21800 (14)	0.0840 (3)	0.46266 (9)	0.0515 (6)
C13	0.13430 (13)	0.1676 (3)	0.45022 (8)	0.0436 (5)
C14	0.1110 (2)	0.2213 (3)	0.39597 (8)	0.0516 (6)
C15	0.0293 (2)	0.2977 (3)	0.38491 (9)	0.0544 (6)
C16	−0.0333 (2)	0.3254 (3)	0.42700 (8)	0.0503 (6)
C17	−0.01359 (14)	0.2764 (3)	0.48018 (8)	0.0431 (5)
C18	0.07033 (12)	0.1979 (2)	0.49290 (7)	0.0369 (5)
C19	0.09558 (12)	0.1444 (2)	0.54715 (7)	0.0360 (5)
O1	0.03107 (8)	0.1720 (2)	0.58699 (5)	0.0420 (4)
O2	0.27408 (9)	−0.0452 (2)	0.62897 (6)	0.0613 (5)

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

C1—C6	1.388 (3)	C10—C19	1.375 (3)
C1—C2	1.393 (3)	C10—C11	1.415 (3)
C1—C7	1.473 (3)	C11—C12	1.348 (3)
C2—C3	1.380 (3)	C12—C13	1.419 (3)
C3—C4	1.374 (4)	C13—C14	1.415 (3)
C4—C5	1.380 (4)	C13—C18	1.418 (3)
C5—C6	1.389 (3)	C14—C15	1.363 (3)
C7—C8	1.338 (3)	C15—C16	1.391 (3)
C7—O1	1.367 (2)	C16—C17	1.368 (3)
C8—C9	1.441 (3)	C17—C18	1.407 (3)
C9—O2	1.235 (2)	C18—C19	1.422 (3)
C9—C10	1.461 (3)	C19—O1	1.371 (2)
C6—C1—C2	118.8 (2)	C11—C10—C9	121.6 (2)
C6—C1—C7	121.0 (2)	C12—C11—C10	121.1 (2)
C2—C1—C7	120.2 (2)	C11—C12—C13	120.9 (2)
C3—C2—C1	120.5 (3)	C14—C13—C18	117.9 (2)
C4—C3—C2	120.3 (2)	C14—C13—C12	122.3 (2)
C3—C4—C5	120.0 (2)	C18—C13—C12	119.8 (2)
C4—C5—C6	120.0 (3)	C15—C14—C13	121.1 (2)
C1—C6—C5	120.4 (2)	C14—C15—C16	120.6 (2)
C8—C7—O1	121.9 (2)	C17—C16—C15	120.4 (2)
C8—C7—C1	126.5 (2)	C16—C17—C18	120.3 (2)
O1—C7—C1	111.6 (2)	C17—C18—C13	119.7 (2)
C7—C8—C9	122.5 (2)	C17—C18—C19	123.4 (2)
O2—C9—C8	123.2 (2)	C13—C18—C19	116.9 (2)
O2—C9—C10	122.6 (2)	O1—C19—C10	122.2 (2)
C8—C9—C10	114.2 (2)	O1—C19—C18	115.0 (2)
C19—C10—C11	118.4 (2)	C10—C19—C18	122.8 (2)
C19—C10—C9	119.9 (2)	C7—O1—C19	119.1 (1)

Data collection: *P*4 diffractometer software. Cell refinement: *XSCANS* (Siemens, 1991). Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, including H-atom geometry, have been deposited with the IUCr (Reference: KA1111). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Desiraju, G. R. (1991). *Acc. Chem. Res.* **24**, 290–296.

Neuman, P. A., Becquart, J., Gillier, B. H., Leroux, Y., Queval, P. & Moretti, J. L. (1989). *Acta Cryst.* C**45**, 1966–1970.

Rossi, M. & Glusker, J. (1979). *Am. Crystallogr. Assoc. Ser.* 2, **6**, p. 69.

Sheldrick, G. M. (1985). *SHELXS*86. *Crystallographic Computing* 3, edited by G. M. Sheldrick, C. Krüger & R. Goddard, pp. 175–189. Oxford Univ. Press.

Sheldrick, G. M. (1990). *SHELXTL-Plus Structure Determination Software Programs.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures.* Univ. of Göttingen, Germany.

Shoja, M. (1994). *Acta Cryst.* C**50**, 771–773.

Siemens (1991). *XSCANS Users Manual.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

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Pair Stacking of 10-(4-Nitrophenyl)-5(10*H*)-phenazinyl Radicals

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Abstract

The title radicals, $C_{18}H_{12}N_3O_2.0.5C_6H_6$, are arranged in radical pairs along the *a* axis with specific overlap of the π systems, leading to short intermolecular contacts between atoms with large spin populations.

Comment

Like Wurster's blue radical cation salts (Hausser, 1956; Yamauchi & Fujita, 1990), 10-ethyl-5(10*H*)-phenazinyl, (1) (McIlwain, 1937) shows an anomalous temperature (T) dependence of the static magnetic susceptibility (χ) in the solid state, with χT falling off with decreasing temperature (Hausser 1956; Serafimov & Zimmermann, 1972). Therefore, crystal structures of 5(10*H*)-phenazinyl radicals, which can provide some insight into this phenomenon, are of particular interest. Recently, we succeeded in determining the crystal