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4,6-Dimethyl-2-(4-nitrobenzylidene)-3(2*H*)-benzofuranone

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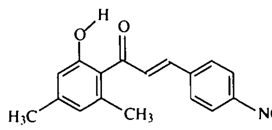
Abstract

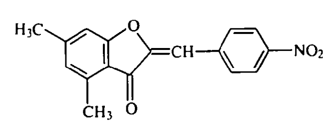
The crystal structure of the title compound, $C_{17}H_{13}NO_4$, is stabilized by both intra- and intermolecular C—H···O hydrogen bonds, and also by $\pi\cdots\pi$ interactions.

Comment

We have found that some 2′-hydroxy-4′,6′-dimethylchalcones undergo solid-state Michael reactions to yield the corresponding flavanones. In connection with this, we attempted to synthesize various chalcone derivatives to examine their solid-state reactivities (Goud, Panneerselvam, Zacharias & Desiraju, 1995). The literature procedure for the synthesis of these chalcones involves treatment of 2-hydroxy-3,5-dimethylacetophenone with the corresponding substituted benzaldehydes in a basic medium. However, when this procedure was attempted with 4-nitrobenzaldehyde [using the recommended $Ba(OH)_2$ rather than NaOH; Matsuoka & Fujise, 1957], the product was not the expected 2′-hydroxy-4′,6′-dimethyl-4-nitrochalcone, (1), but instead the oxidation product aurone, (2). In general, aurones can serve as convenient precursors to flavonoid-type natural products (Adam, Hadjiarapoglou & Levai, 1992); therefore, we solved the crystal structure of (2) to examine the structural aspects.



(1)



(2)

Fig. 1 shows aurone (2) with the atomic numbering scheme. There are two molecules in the asymmetric unit. The double bond of the aurone has a *Z* configuration. Both molecules in the asymmetric unit are planar and this planar geometry is possibly stabilized by intramolecular C—H···O hydrogen bonding (C5···O3, H5···O3 and C5—H5···O3 are 2.93, 2.28 Å and 126°, respectively, in molecule *A*, and 2.98, 2.28 Å and 127°, respectively, in molecule *B*). The crystal structure of (2) is stabilized additionally by extensive intermolecular C—H···O hydrogen bonds, $\pi\cdots\pi$

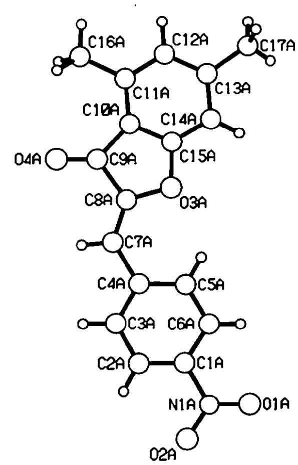


Fig. 1. Atomic numbering scheme for molecule *A* of aurone (2). The numbering for molecule *B* is identical.

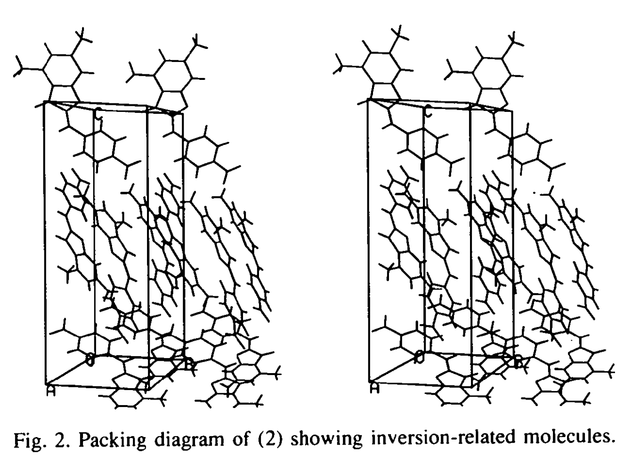


Fig. 2. Packing diagram of (2) showing inversion-related molecules.

and herringbone interactions. There are around 20 intermolecular C—H···O hydrogen bonds with C···O distances in the range 3.37–3.82 Å and C—H···O angles in the range 118–163°. The shortest C—H···O hydrogen bond is C3*B*—H3*B*···O2*A* and the longest is C17*B*—H172*B*···O1*B*. These ranges are typical of those found for C—H···O hydrogen-bonded compounds (Desiraju, 1991). The dimethylphenyl moieties of (2) stack with the nitrophenyl moieties, presumably to improve the $\pi\cdots\pi$ interactions. These stacked molecules are inversion related. The packing diagram of (2) is given in Fig. 2.

Experimental

Crystal data

$C_{17}H_{13}NO_4$
$M_r = 295.28$
Triclinic
$P\bar{1}$
$a = 8.1610$ (10) Å
$b = 8.2220$ (10) Å
$c = 23.113$ (2) Å
$\alpha = 93.46$ (2)°
$\beta = 96.88$ (2)°
$\gamma = 113.43$ (2)°
$V = 1402.8$ (3) Å^3
$Z = 4$
$D_x = 1.398$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 20 reflections
$\theta = 10$–20°
$\mu = 0.101$ mm^{-1}
$T = 293$ (2) K
Needle
0.2 × 0.15 × 0.1 mm
Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
6135 measured reflections
6135 independent reflections
5006 observed reflections [$I > 2\sigma(I)$]

$\theta_{max} = 27.47°$
$h = 0 \rightarrow 10$
$k = -10 \rightarrow 9$
$l = -29 \rightarrow 29$
3 standard reflections monitored every 200 reflections
intensity decay: 2.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0576$
$wR(F^2) = 0.1494$
$S = 1.092$
6135 reflections
501 parameters
H atoms isotropic
$w = 1/[\sigma^2(F_o^2) + (0.0641P)^2 + 0.2542P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = -0.477$
$\Delta\rho_{max} = 0.150$ e Å^{-3}
$\Delta\rho_{min} = -0.249$ e Å^{-3}
Extinction correction: none
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*A*	−0.1246 (2)	0.6071 (3)	−0.18484 (8)	0.0428 (4)
C2*A*	−0.1726 (3)	0.4342 (3)	−0.20835 (8)	0.0490 (5)
C3*A*	−0.1143 (3)	0.3257 (3)	−0.17585 (9)	0.0478 (4)
C4*A*	−0.0081 (2)	0.3902 (3)	−0.12047 (8)	0.0422 (4)
C5*A*	0.0399 (3)	0.5681 (3)	−0.09862 (8)	0.0452 (4)
C6*A*	−0.0175 (3)	0.6783 (3)	−0.13061 (8)	0.0454 (4)
C7*A*	0.0484 (3)	0.2678 (3)	−0.08903 (8)	0.0442 (4)
C8*A*	0.1578 (2)	0.3002 (3)	−0.03776 (8)	0.0421 (4)
C9*A*	0.2212 (3)	0.1710 (3)	−0.00981 (8)	0.0438 (4)
C10*A*	0.3398 (2)	0.2757 (2)	0.04380 (8)	0.0402 (4)
C11*A*	0.4428 (3)	0.2374 (3)	0.08990 (8)	0.0440 (4)
C12*A*	0.5384 (3)	0.3739 (3)	0.13482 (8)	0.0472 (4)
C13*A*	0.5374 (3)	0.5434 (3)	0.13519 (8)	0.0460 (4)
C14*A*	0.4370 (3)	0.5810 (3)	0.08861 (8)	0.0453 (4)
C15*A*	0.3412 (2)	0.4444 (3)	0.04496 (7)	0.0399 (4)
C16*A*	0.4500 (4)	0.0581 (3)	0.09021 (12)	0.0620 (6)
C17*A*	0.6420 (4)	0.6862 (4)	0.18535 (11)	0.0637 (6)
N1*A*	−0.1931 (2)	0.7212 (3)	−0.21728 (7)	0.0516 (4)
O1*A*	−0.1571 (3)	0.8723 (2)	−0.19518 (7)	0.0676 (4)
O2*A*	−0.2876 (3)	0.6565 (3)	−0.26524 (7)	0.0783 (5)
O3*A*	0.2342 (2)	0.4627 (2)	−0.00340 (5)	0.0448 (3)
O4*A*	0.1822 (2)	0.0182 (2)	−0.02998 (7)	0.0615 (4)
C1*B*	0.6165 (3)	0.9938 (3)	0.31312 (8)	0.0434 (4)
C2*B*	0.4717 (3)	1.0309 (3)	0.29125 (8)	0.0473 (4)
C3*B*	0.3303 (3)	0.9921 (3)	0.32235 (8)	0.0470 (4)
C4*B*	0.3318 (3)	0.9181 (2)	0.37540 (8)	0.0433 (4)
C5*B*	0.4803 (3)	0.8804 (3)	0.39568 (9)	0.0484 (5)
C6*B*	0.6217 (3)	0.9174 (3)	0.36470 (9)	0.0478 (5)
C7*B*	0.1792 (3)	0.8865 (3)	0.40664 (8)	0.0459 (4)
C8*B*	0.1483 (3)	0.8220 (3)	0.45776 (8)	0.0441 (4)
C9*B*	−0.0191 (3)	0.7845 (3)	0.48448 (8)	0.0444 (4)
C10*B*	0.0117 (2)	0.7074 (2)	0.53795 (8)	0.0410 (4)
C11*B*	−0.0930 (3)	0.6403 (3)	0.58150 (8)	0.0450 (4)
C12*B*	−0.0169 (3)	0.5748 (3)	0.62624 (9)	0.0488 (4)
C13*B*	0.1535 (3)	0.5705 (3)	0.62777 (8)	0.0485 (5)
C14*B*	0.2550 (3)	0.6364 (3)	0.58373 (8)	0.0471 (4)
C15*B*	0.1801 (3)	0.7036 (2)	0.54015 (8)	0.0424 (4)
C16*B*	−0.2793 (3)	0.6343 (4)	0.57922 (12)	0.0597 (6)
C17*B*	0.2243 (4)	0.4922 (4)	0.67680 (12)	0.0648 (7)
N1*B*	0.7724 (2)	1.0423 (2)	0.28230 (7)	0.0521 (4)
O1*B*	0.8930 (2)	0.9957 (3)	0.29931 (8)	0.0721 (5)
O2*B*	0.7767 (3)	1.1294 (3)	0.24088 (8)	0.0727 (5)
O3*B*	0.2651 (2)	0.7714 (2)	0.49296 (6)	0.0475 (3)
O4*B*	−0.1491 (2)	0.8119 (2)	0.46372 (6)	0.0579 (4)

Data collection: Enraf–Nonius CAD-4 diffractometer software. Cell refinement: Enraf–Nonius CAD-4 diffractometer software. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *PLUTO* (Motherwell & Clegg, 1978). 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 have been deposited with the IUCr (Reference: KA1091). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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9(10*H*)-Acridone

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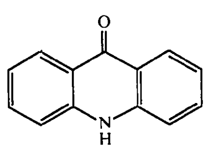
Abstract

The molecular packing arrangement in $C_{13}H_9NO$ is characterized by two major interaction types. The first comprises N—H···O hydrogen bonds between glide-related molecules, with an N···O distance of 2.782 (3) Å, such that each molecule is hydrogen bonded to two adjacent molecules. The second consists of π–π interactions between molecules stacked along the short crystal axis.

Comment

The structure determination of the title compound, (I), was undertaken as part of an investigation into the importance of specific intermolecular interactions in relation to the observed packing arrangement adopted by planar aromatic systems.



(I)

The molecule is planar with no atoms deviating by more than 0.02 Å from the molecular plane defined by the non-H ring atoms and the O atom; all torsion angles lie within ±1.5° of 0 or 180°. The molecules adopt a herringbone packing arrangement very similar to that found in anthraquinone (Lenstra & van Loock, 1984), indigo (Suesse, Steins & Kupcik, 1988) and quinacridone (Potts, Jones, Bullock, Andrews & Maginn, in preparation). Hydrogen bonding is maximized in such structures.

Overall hydrogen bonding and π–π interactions are the dominant factors controlling the crystal arrangement (Fig. 2).

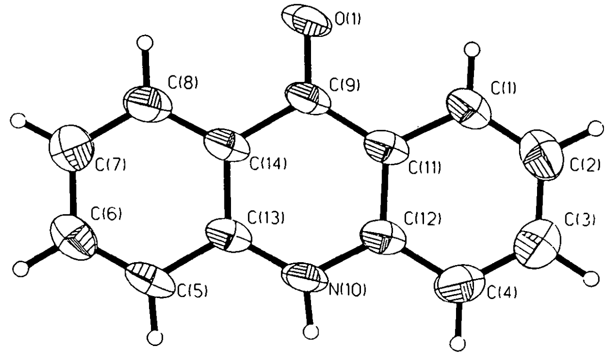


Fig. 1. *SHELXTL-Plus* (Sheldrick, 1990*b*) view of the molecule with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

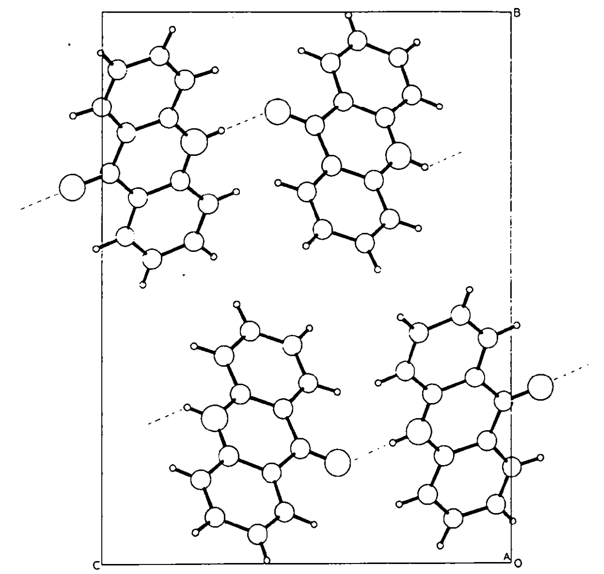


Fig. 2. Crystal structure viewed down the *a* axis with the hydrogen bonds shown.

Experimental

Crystal data

$C_{13}H_9NO$	Mo $K\alpha$ radiation
$M_r = 195.21$	$\lambda = 0.71073$ Å
Monoclinic	Cell parameters from 25 reflections
$P2_1/n$	$\theta = 8–15°$
$a = 4.5330$ (10) Å	$\mu = 0.088$ mm^{-1}
$b = 16.537$ (3) Å	$T = 296 (2)$ K
$c = 12.687$ (3) Å	Plate
$\beta = 97.22$ (3)°	