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5-(4-Methoxybenzoyl)-6-(4-methoxyphenyl)-3-phenyl-3,4-dihydro-2*H*-1,3-oxazine-2,4-dione

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.005 Å
R factor = 0.055
wR factor = 0.154
Data-to-parameter ratio = 11.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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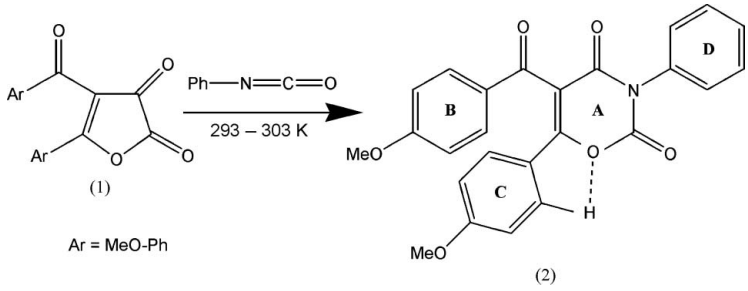
In the title compound, $C_{25}H_{19}NO_6$, the molecular structure is stabilized by intra- and intermolecular C—H···O hydrogen bonds. The intermolecular hydrogen bonds link the molecules into a herringbone-like dimer.

Comment

Oxazine derivatives have been shown to be antimicrobial agents (Bayomi *et al.*, 1985), fungicides (Player *et al.*, 1993), and also to exhibit some cytotoxic or antitumour activity (Eger & Frey, 1992; Mordarski *et al.*, 1970; Mordarski & Chylinska, 1971, 1972). In the light of this, we have synthesized and characterized the title compound, (2), and have determined its structure by X-ray analysis.



The molecular structure of (2) is illustrated in Fig. 1. The rings (*A*, *B*, *C* and *D*) are each essentially planar, with r.m.s. deviations of 0.031 (2), 0.019 (2), 0.017 (2) and 0.006 (2) Å, respectively. The dihedral angles between the rings are *A*/*B* = 64.31 (9)°, *A*/*C* = 19.49 (16)°, *A*/*D* = 82.31 (8)°, *B*/*C* = 62.22 (9)°, *B*/*D* = 61.61 (9)° and *C*/*D* = 63.73 (8)°. The bond lengths and angles are in agreement with reported literature values (Allen *et al.*, 1987).

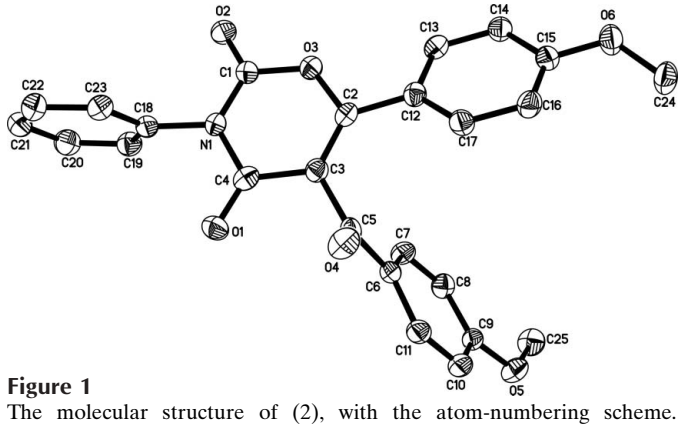


Figure 1
The molecular structure of (2), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted.

The structure is stabilized by intra- and intermolecular C—H···O hydrogen bonds (Table 1). In the crystal structure, the C—H···O intermolecular hydrogen bonds link the molecules into herringbone-like dimers which are stacked along the *b* axis (Fig. 2).

Experimental

Compound (1) was prepared from the cyclocondensation reaction that occurs between p,p′-dimethoxydibenzoylketene and oxalyl chloride (Hökelek *et al.*, 2002). Compound (2) was obtained from 1.0 g (2.96 mmol) (1) and 0.35 g (2.96 mmol) phenyl isocyanate in a 25 ml round-bottomed flask equipped with a calcium chloride tube. The mixture was heated at 393 K for 1 h. The cooled reaction mixture was triturated with dry diethyl ether and then recrystallized from *n*-butanol (yield 0.83 g, 65%, m.p. 480 K). IR (KBr, cm^{-1}): ν 1774 (C5—O4), 1690 (C4—O1), 1646 (C1—O2). ^{1}H NMR ($CDCl_3$): δ 7.95–6.81 (*m*, 13H, Ar—H), 3.84, 3.79 (*s*, 6H, CH3O); ^{13}C NMR ($CDCl_3$): δ 190.80 (C5—O4), 166.60 (C4—O1), 165.04 (C1—O2), 162.36–113.47 (C═C, aromatic and aliphatic), 57.52, 57.44 (CH_3O). Analysis calculated for $C_{40}H_{35}N_3O_5$: C 69.93, H 4.42, N 3.26%; found: C 69.80, H 4.51, N 3.14%.

Crystal data

$C_{25}H_{19}NO_6$
M_r = 429.41
Monoclinic, $P2_1/c$
a = 10.950 (2) Å
b = 5.8163 (12) Å
c = 30.968 (6) Å
β = 91.010 (4)°
V = 1972.0 (7) Å^3
Z = 4
D_x = 1.446 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1626 reflections
θ = 4.5–50.4°
μ = 0.10 mm^{-1}
T = 150 (2) K
Needle, colourless
0.32 × 0.12 × 0.12 mm

Data collection

Bruker SMART1000 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.967, T_{max} = 0.988
13592 measured reflections
3456 independent reflections
2069 reflections with $I > 2\sigma(I)$
R_{int} = 0.089
θ_{max} = 25.0°
$h = -13 \rightarrow 13$
$k = -6 \rightarrow 6$
$l = -36 \rightarrow 36$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.154
S = 0.98
3456 reflections
291 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0743P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C13—H13···O3	0.95	2.33	2.665 (4)	100
C14—H14···O2^{i}	0.95	2.45	3.384 (4)	168
C19—H19···O1^{ii}	0.95	2.47	3.231 (4)	137
C24—H24*A*···O2^{iii}	0.98	2.52	3.225 (4)	128

Symmetry codes: (i) $-x+2, -y+1, -z$; (ii) $x, y-1, z$; (iii) $-x+2, -y+2, -z$.

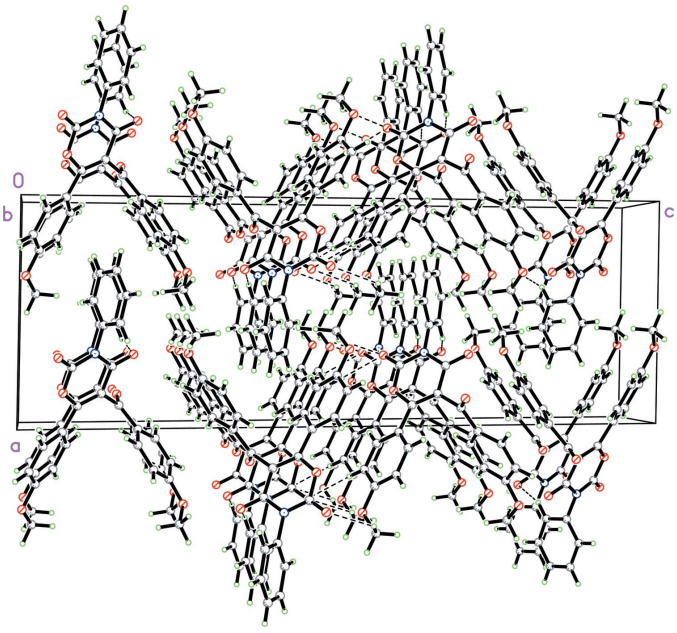


Figure 2
Packing diagram of (2); C—H···O hydrogen bonds are indicated by dashed lines.

H atoms were positioned geometrically [C—H = 0.95 (CH) and 0.98 Å (CH_3)] and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2 (1.5 for methyl) times U_{eq}(C).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SHELXTL* (Bruker, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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