organic papers

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.002 Å
R factor = 0.036
wR factor = 0.095
Data-to-parameter ratio = 13.7

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

3-(4-Methoxyanilino)isobenzofuran-1(3H)-one

Crystals of the title compound, $C_{15}H_{13}NO_3$, are stabilized by N—H···O intermolecular hydrogen bonds and two C—H···π intermolecular interactions. The N—H···O hydrogen bonds generate an $R_2^2(12)$ ring motif and the phthalide part of the molecule is planar. The dihedral angle between the phthalide group and the benzene ring is 71.18 (5)°.

Comment

Phthalides (isobenzofuranones) are five-membered lactones found in plants and are known to show diverse biological activities as hormones, pheromones and antibiotics (Aoki *et al.*, 1973; Lacova, 1973; Elderfield, 1951; Tsi & Tan, 1997). Considering the potential interest of such phthalide-3-phosphonates in synthetic organic chemistry, and as agrochemical and pharmaceutical agents, we decided to investigate the solid state structures of 3-substituted phthalides. The structure of the title compound, (I), is reported here (Fig. 1 and Table 1).

(I)
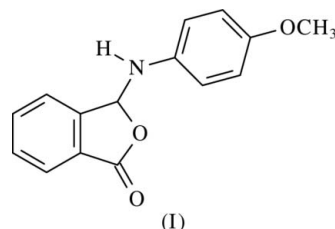
The dihedral angle between the five-membered ring and the fused six-membered ring is 0.89 (7)°, whereas that between the benzene ring and the mean plane of the overall phthalide group is 71.18 (5)°. The geometry of the molecule does not show any significant differences from the average geometry found for 3-(4-hydroxyanilino)isobenzofuran-1(3H)-one (Odabaşoğlu & Büyükgüngör, 2006).

Two inversion-related molecules of (I) are linked by N1—H1···O1 hydrogen bonds (Fig. 2 and Table 2), which generate an $R_2^2(12)$ ring motif (Etter, 1990). There are no direction-specific aromatic π–π interactions between adjacent rings in (I), but two C—H···π interactions link parallel phthalide rings (Table 2).

Experimental

The title compound was prepared as described by Odabaşoğlu & Büyükgüngör (2006), using phthalaldehydic acid and *p*-methoxy-aniline as starting materials (yield 95%; m.p. 416–417 K). Crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an ethanol (95%) solution at room temperature.

Crystal data

$C_{15}H_{13}NO_3$
$M_r = 255.26$
Monoclinic, $P2_1/c$
$a = 10.5814$ (8) Å
$b = 8.1613$ (4) Å
$c = 15.2896$ (11) Å
$\beta = 111.656$ (5)°
$V = 1227.18$ (14) Å^3

$Z = 4$
$D_x = 1.382$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.10$ mm^{-1}
$T = 296$ (2) K
Prism, brown
$0.61 \times 0.53 \times 0.38$ mm

Data collection

Stoe IPDS-2 diffractometer
ω scans
Absorption correction: integration
  (X-$RED32$; Stoe & Cie, 2002)
  $T_{min} = 0.940$, $T_{max} = 0.969$

16415 measured reflections
2406 independent reflections
1968 reflections with $I > 2\sigma(I)$
$R_{int} = 0.059$
$\theta_{max} = 26.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.096$
$S = 1.05$
2406 reflections
176 parameters
H atoms treated by a mixture of
  independent and constrained
  refinement

$w = 1/[\sigma^2(F_o^2) + (0.0553P)^2 + 0.0742P]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.17$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—O1	1.2056 (15)	C8—N1	1.4022 (15)
C1—O2	1.3505 (14)	C8—O2	1.4902 (14)
C2—C7	1.3771 (17)	C9—N1	1.4085 (15)
C7—C8	1.4974 (16)		
O1—C1—O2	121.86 (11)	N1—C8—O2	112.65 (9)
O1—C1—C2	129.60 (12)	N1—C8—C7	113.32 (10)

Table 2
Hydrogen-bond geometry (Å, °).

$Cg1$ and $Cg2$ are the centroids of the C2–C7 and C9–C14 rings, respectively.

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^i	0.891 (15)	2.160 (15)	3.0141 (14)	160.4 (12)
C8—H8···$Cg1^{ii}$	0.98	2.66	3.534 (1)	148
C10—H10···$Cg2^{iii}$	0.93	3.18	3.812 (1)	127

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

All C-bound H atoms were refined using the riding model approximation with C—H = 0.93 Å for aromatic, 0.98 Å for methine and 0.96 Å for methyl H atoms [U_{iso}(H) = $1.2U_{eq}$(parent atom)]. The N-bound H atom was located in a difference Fourier map and refined freely with an isotropic displacement parameter.

Data collection: X-$AREA$ (Stoe & Cie, 2002); cell refinement: X-$AREA$; data reduction: X-$RED32$ (Stoe & Cie, 2002); program(s) used to solve structure: $SHELXS97$ (Sheldrick, 1990); program(s) used to refine structure: $SHELXL97$ (Sheldrick, 1997); molecular
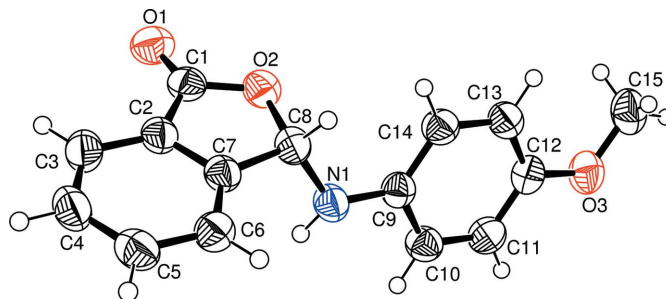

Figure 1
A view of (I), showing the atomic numbering scheme, with displacement ellipsoids drawn at the 50% probability level.
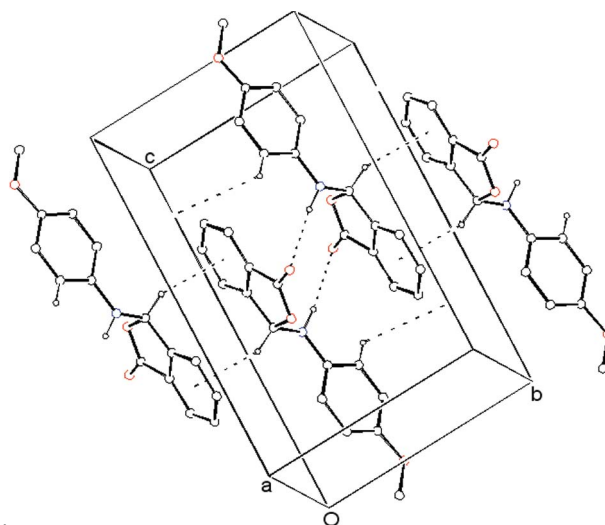

Figure 2
Packing diagram of (I). Hydrogen bonds and C—H···π stacking interactions are drawn as dashed lines.

graphics: $ORTEP$-3 for Windows (Farrugia, 1997); software used to prepare material for publication: $WinGX$ (Farrugia, 1999).

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