Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-{4-[4-(3-Oxo-1,3-dihydroisobenzofuran-1-ylamino)benzyl]phenylamino}isobenzofuran-1(3*H*)-one dimethylformamide solvate

Mustafa Odabaşoğlu[a]* and Orhan Büyükgüngör[b]

[a]Department of Chemistry, Faculty of Arts & Science, Ondokuz Mayıs University, TR-55139 Kurupelit Samsun, Turkey, and [b]Department of Physics, Faculty of Arts & Science, Ondokuz Mayıs University, TR-55139 Kurupelit Samsun, Turkey

Correspondence e-mail: muodabas@omu.edu.tr

Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.006 Å
R factor = 0.072
wR factor = 0.203
Data-to-parameter ratio = 13.5

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

© 2006 International Union of Crystallography
All rights reserved

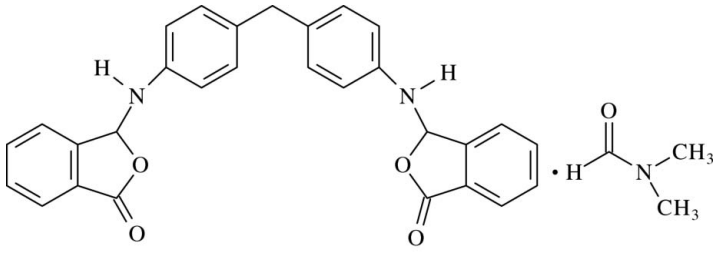
The crystal structure of the title compound, $C_{29}H_{22}N_2O_4\cdot C_3H_7NO$, is stabilized by two N—H···O and six C—H···O intermolecular hydrogen bonds and C—H···π and π–π interactions. The intermolecular hydrogen bonds generate $R_1^2(6)$ and $R_2^2(32)$ ring motifs.

Received 27 July 2006
Accepted 31 July 2006

3-Substituted phthalides, Part XVI

Comment

Benzolactones are found in plants and they show several pharmacological effects, such as fungicidal, bactericidal, herbicidal and analgesic activities (Aoki *et al.*, 1973; Lacova, 1973). The present work is a part of our systematic research on the crystal structure analysis of 3-substituted phthalides (3-substituted benzolactones) synthesized from reactions of aromatic amines and phthalaldehydic acid.



(I)

In (I), the molecules have a planar configuration at the N atoms. The molecular conformation (Fig. 1) and relative orientation of the solvent molecule are defined by ten dihedral angles [*A*/*B* = 58.35 (15)°, *A*/*C* = 45.04 (15)°, *A*/*D* = 71.49 (11)°, *A*/*E* = 36.9 (4)°, *B*/*C* = 78.50 (18)°, *B*/*D* = 62.89 (15)°, *B*/*E* = 37.3 (4)°, *C*/*D* = 54.82 (15)°, *C*/*E* = 73.6 (4)° and *D*/*E* = 72.2 (4)°], and these angles show that the molecules do not exhibit even approximate rotational symmetry but the orientations of *A*–*B* and *C*–*D* rings are similar for the two independent phenylphthalide units within the molecule.

The asymmetric unit (Fig. 1) contains one 3-{4-[4-(3-oxo-1,3-dihydroisobenzofuran-1-ylamino)benzyl]phenylamino}-isobenzofuran-1(3*H*)-one molecule and one dimethylformamide (DMF) molecule, which are linked by N2—H2···O5 and C34—H34···O5 hydrogen bonds (Table 2), forming a six-membered ring with graph-set notation $R_2^1(6)$ (Etter, 1990; Bernstein *et al.*, 1995). This motif further self-organizes through N—H···O hydrogen bonds (Fig. 2), generating an array of two hydrogen bonds, the ring having graph-set notation $R_2^2(32)$. These hydrogen-bonded rings are linked into a complex three-dimensional framework by a combination of C—H···O hydrogen bonds (Table 2 and Fig. 3). There are also C—H···π (Table 2) and π–π interactions. The π–π interaction occurs between the O2/C1/C2/

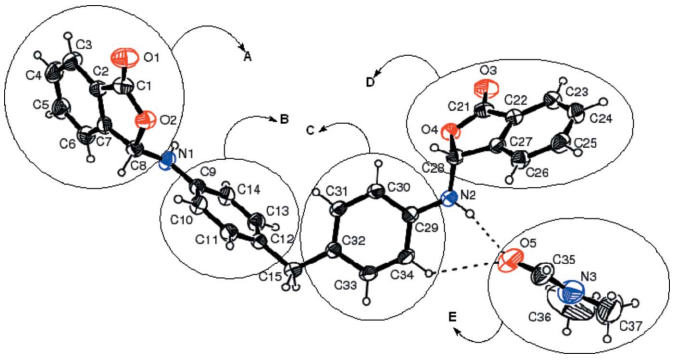


Figure 1
A view of (I), showing the atomic numbering scheme, with displacement ellipsoids drawn at the 30% probability level. The labels *A*, *B*, *C* and *D* denote the ring planes and *E* the DMF molecular plane excluding the H atom.

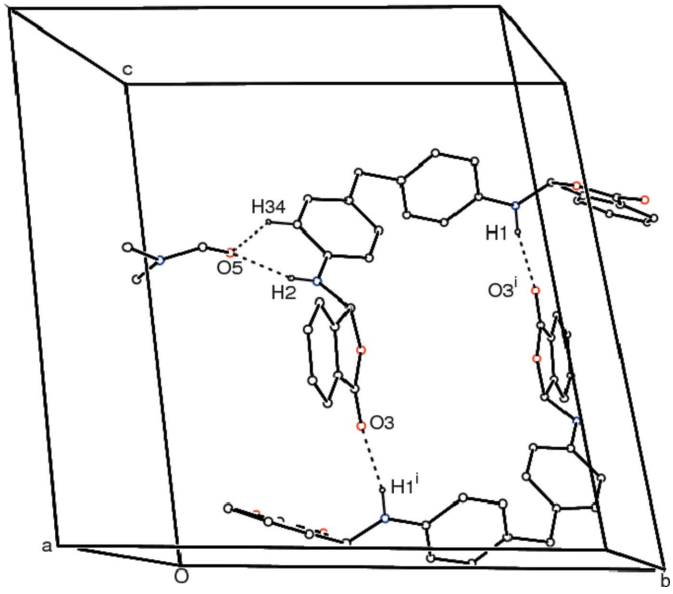


Figure 2
Part of the crystal structure of (I), showing the formation of two hydrogen-bonded ring motifs. For the sake of clarity, H atoms not involved in the motifs shown have been omitted. Atoms marked with an (i) are at the symmetry position $(x-1, y+\frac{1}{2}, z+\frac{1}{2})$.

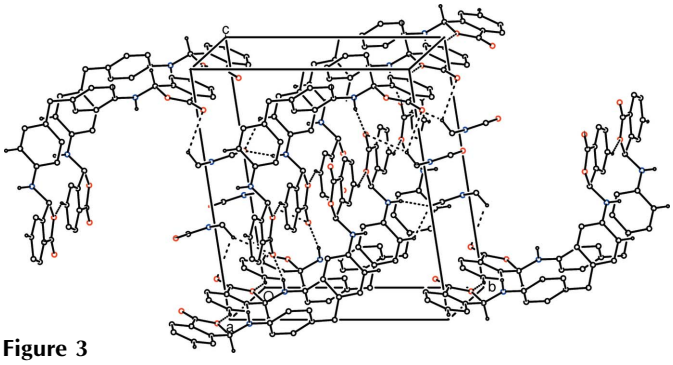


Figure 3
The packing of (I). H atoms have been omitted. The dashed lines indicate the intramolecular hydrogen bonds.

C7/C8 rings of the phthalide units at (x, y, z) and $(2-x, -y, -z)$, with a centroid-to-centroid distance of 3.506 (2) Å and a plane-to-plane separation of 3.411 Å.

Experimental

The title compound, (I), was prepared as described by Odabaşoğlu & Büyükgüngör (2006), using phthalaldehydic acid and 4-(4-aminobenzyl)benzenamine as starting materials (yield 88%; m.p. 512–513 K). Crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of a DMF solution at room temperature.

Crystal data

$C_{29}H_{22}N_2O_4\cdot C_3H_7NO$
$M_r = 535.58$
Triclinic, $P\overline{1}$
$a = 8.0267$ (7) Å
$b = 12.6709$ (11) Å
$c = 14.6529$ (13) Å
$\alpha = 98.911$ (7)°
$\beta = 100.893$ (7)°
$\gamma = 104.746$ (6)°
$V = 1382.5$ (2) Å^3
$Z = 2$
$D_x = 1.287$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.09$ mm^{-1}
$T = 296$ K
Long prismatic stick, light brown
0.71 × 0.34 × 0.11 mm

Data collection

Stoe IPDS-2 diffractometer
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
$T_{min} = 0.951$, $T_{max} = 0.988$
16524 measured reflections
5014 independent reflections
2712 reflections with $I > 2\sigma(I)$
$R_{int} = 0.101$
$\theta_{max} = 25.3°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.072$
$wR(F^2) = 0.203$
$S = 0.96$
5014 reflections
371 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1127P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.41$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—O1	1.204 (5)	C21—O4	1.338 (5)
C1—O2	1.357 (5)	C22—C27	1.376 (5)
C2—C7	1.375 (5)	C28—N2	1.394 (4)
C8—N1	1.383 (5)	C35—O5	1.207 (7)
C21—O3	1.204 (4)		
O1—C1—O2	121.6 (4)	O3—C21—O4	121.5 (3)
O1—C1—C2	129.3 (4)	O3—C21—C22	129.3 (4)
N1—C8—O2	113.6 (3)	N2—C28—O4	111.0 (3)

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

*Cg*1 is the centroid of the C29–C34 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O5	0.845 (18)	2.06 (2)	2.886 (5)	165 (4)
C34—H34···O5	0.93	2.81	3.505 (6)	133
N1—H1···O3^{i}	0.871 (19)	2.10 (2)	2.956 (4)	168 (4)
C5—H5···O2^{ii}	0.93	2.53	3.413 (6)	160
C8—H8···O1^{iii}	0.98	2.88	3.573 (5)	129
C25—H25···O4^{iv}	0.93	2.54	3.408 (4)	155
C36—H36b···O3^{v}	0.96	2.85	3.669 (13)	143
C37—H37c···O1^{vi}	0.96	2.60	3.208 (10)	121
C14—H14···*Cg*1^{iv}	0.93	3.00	3.735 (4)	137

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

All C-bound H atoms were refined using the riding-model approximation, with C—H = 0.93 Å for aromatic, 0.97 Å for methylene and 0.98 Å for methine H atoms [U_{iso}(H) = 1.2U_{eq}(C)] and C—H = 0.96 Å for methyl H atoms [U_{iso}(H) = 1.5U_{eq}(C)]. The amino atoms H1 and H2 were located in a Fourier difference map and refined with a distance restraint of 0.87 (2) Å.

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

The authors acknowledge the Faculty of Arts and Sciences, Ondokuz Mayıs University, Turkey, for the use of the Stoe IPDS-2 diffractometer (purchased under grant F.279 of the University Research Fund).

References

Aoki, T., Furusho, T., Kimura, T., Satake, S. & Funayama, S. (1973). JP Patent No. 7 324 724; *Chem. Abstr.* (1974), **80**, 129246.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Etter, M. C. (1990). *Acc. Chem. Res.* **23**, 120–126.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Lacova, M. (1973). *Chem. Zvesti*, **27**, 525–535; *Chem. Abstr.* (1974), **80**, 59757.
Odabaşoğlu, M. & Büyükgüngör, O. (2006). *Acta Cryst.* E**62**, o1879–o1881.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL97*, Univ. of Göttingen, Germany.
Stoe & Cie (2002). *X-AREA* (Version 1.18) and *X-RED32* (Version 1.04). Stoe & Cie, Darmstadt, Germany.