

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

1,8-Bis(4-aminobenzoyl)-2,7-dimethoxynaphthalene

Takahiro Nishijima, Kotaro Kataoka, Atsushi Nagasawa, Akiko Okamoto and Noriyuki Yonezawa*

Department of Organic and Polymer Materials Chemistry, Tokyo University of Agriculture & Technology, 2-24-16 Naka-machi, Koganei, Tokyo 184-8588, Japan
Correspondence e-mail: yonezawa@cc.tuat.ac.jp

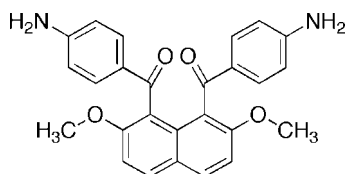
Received 2 October 2010; accepted 14 October 2010

Key indicators: single-crystal X-ray study; $T = 193$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å;
R factor = 0.043; wR factor = 0.114; data-to-parameter ratio = 10.6.

The title compound [systematic name: [8-(4-aminobenzoyl)-2,7-dimethoxynaphthalen-1-yl](4-aminophenyl)methanone], $\text{C}_{26}\text{H}_{22}\text{O}_4\text{N}_2$, possesses crystallographically imposed twofold symmetry, with two C atoms lying on the rotation axis. In the crystal, the molecules interact through intermolecular N—H \cdots O hydrogen bonds between the amino and methoxy groups on the naphthalene ring systems and N—H \cdots π interactions between the amino groups and the naphthalene rings. Furthermore, weak C—H \cdots O hydrogen bonds and π – π stacking interactions between the benzene rings are observed. The centroid–centroid and interplanar distances between the benzene rings of the aroyl group and the naphthalene ring systems of adjacent molecules are 3.6954 (8) and 3.2375 (5) Å, respectively. The dihedral angle between the mean planes of the benzene ring and the naphthalene ring system is 83.59 (5)°. The benzene ring and the carbonyl group in the benzoyl unit are almost coplanar [C—C—C—O torsion angle = 175.91 (10)°].

Related literature

For the formation reaction of aroylated naphthalene compounds *via* electrophilic aromatic aroylation of 2,7-dimethoxynaphthalene, see: Okamoto & Yonezawa (2009). For related structures, see: Muto *et al.* (2010); Nakaema *et al.* (2007, 2008); Watanabe *et al.* (2010*a,b*). For work-up procedure in the preparation of the title compound, see: Bellamy *et al.* (1984).



Experimental

Crystal data

$\text{C}_{26}\text{H}_{22}\text{N}_2\text{O}_4$
 $M_r = 426.46$
Monoclinic, $C2/c$
 $a = 14.2996$ (3) Å
 $b = 10.2811$ (2) Å
 $c = 15.4306$ (3) Å
 $\beta = 114.523$ (1)°
 $V = 2063.90$ (7) Å³
 $Z = 4$
Cu $K\alpha$ radiation
 $\mu = 0.76$ mm⁻¹
 $T = 193$ K
0.40 × 0.30 × 0.10 mm

Data collection

Rigaku R-Axis RAPID diffractometer
Absorption correction: numerical (NUMABS; Higashi, 1999)
 $T_{\min} = 0.751$, $T_{\max} = 0.928$
17453 measured reflections
1892 independent reflections
1690 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.056$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.114$
 $S = 1.12$
1892 reflections
178 parameters
H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.24$ e Å⁻³
 $\Delta\rho_{\min} = -0.37$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

Cg is the centroid of the C8–C13 ring.

D—H \cdots A	D—H	H \cdots A	D \cdots A	D—H \cdots A
N1—H4 \cdots O1 ⁱ	0.93 (2)	2.26 (2)	3.1708 (18)	169.1 (18)
C14—H14B \cdots O2 ⁱⁱ	0.96	2.57	3.5013 (17)	165
N1—H3 \cdots Cg ⁱⁱⁱ	0.92 (2)	2.50 (2)	3.3301 (13)	149.8 (18)

Symmetry codes: (i) $-x + \frac{3}{2}, -y + \frac{1}{2}, -z + 2$; (ii) $-x + \frac{3}{2}, y - \frac{1}{2}, -z + \frac{3}{2}$; (iii) $x, -y, z + \frac{1}{2}$.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSK, 2004); program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP3* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97*.

The authors express their gratitude to Professor Keiichi Noguchi, Instrumentation Analysis Center, Tokyo University of Agriculture & Technology, for technical advice. This work was partially supported by the Sasagawa Scientific Research Grant from the Japan Science Society.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2496).

References

- Bellamy, F. D. & Ou, K. (1984). *Tetrahedron Lett.* **25**, 839–842.
Burla, M. C., Caliandro, R., Camalli, M., Carrozzini, B., Cascarano, G. L., De Caro, L., Giacovazzo, C., Polidori, G. & Spagna, R. (2005). *J. Appl. Cryst.* **38**, 381–388.
Burnett, M. N. & Johnson, C. K. (1996). *ORTEP3*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
Higashi, T. (1999). *NUMABS*. Rigaku Corporation, Tokyo, Japan.
Muto, T., Kato, Y., Nagasawa, A., Okamoto, A. & Yonezawa, N. (2010). *Acta Cryst.* **E66**, o2752.
Nakaema, K., Okamoto, A., Noguchi, K. & Yonezawa, N. (2007). *Acta Cryst.* **E63**, o4120.

- Nakaema, K., Watanabe, S., Okamoto, A., Noguchi, K. & Yonezawa, N. (2008). *Acta Cryst.* **E64**, o807.
- Okamoto, A. & Yonezawa, N. (2009). *Chem. Lett.* **38**, 914–915.
- Rigaku (1998). *PROCESS-AUTO*. Rigaku Corporation, Tokyo, Japan.
- Rigaku/MSC (2004). *CrystalStructure*. Rigaku/MSC, The Woodlands, Texas, USA.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Watanabe, S., Nagasawa, A., Okamoto, A., Noguchi, K. & Yonezawa, N. (2010a). *Acta Cryst.* **E66**, o329.
- Watanabe, S., Nakaema, K., Muto, T., Okamoto, A. & Yonezawa, N. (2010b). *Acta Cryst.* **E66**, o403.

supporting information

Acta Cryst. (2010). E66, o2904–o2905 [https://doi.org/10.1107/S1600536810041346]

1,8-Bis(4-aminobenzoyl)-2,7-dimethoxynaphthalene

Takahiro Nishijima, Kotaro Kataoka, Atsushi Nagasawa, Akiko Okamoto and Noriyuki Yonezawa

S1. Comment

In the course of our study on selective electrophilic aromatic arylation of naphthalene core, *peri*-arylnaphthalene compounds have proved to be formed regioselectively by the aid of suitable acidic mediators (Okamoto & Yonezawa, 2009). The aryl groups at 1,8-positions of the naphthalene rings in these compounds are oriented in opposite direction. The aromatic rings in this molecule are clarified to be assembled with non-coplanar configuration resulting in partial disruption of π -conjugated ring systems. Recently, we reported the X-ray crystal structures of 1,8-diaroylated 2,7-dimethoxynaphthalenes such as 1,8-bis(4-chlorobenzoyl)-2,7-dimethoxynaphthalene (Nakaema *et al.*, 2007), 1,8-dibenzoyl-2,7-dimethoxynaphthalene (Nakaema *et al.*, 2008), bis(4-bromophenyl) (2,7-dimethoxynaphthalene-1,8-diyl)dimethanone (Watanabe *et al.*, 2010a), (2,7-dimethoxynaphthalene-1,8-diyl)bis(4-fluorophenyl)dimethanone (Watanabe *et al.*, 2010b) and 1,8-bis(4-methylbenzoyl)-2,7-dimethoxynaphthalene (Muto *et al.*, in press). As a part of our continuous study on the molecular structures of this kind of homologous molecules, the X-ray crystal structure of title compound, (I), a *peri*-arylnaphthalene bearing amino groups, is discussed in this article.

An *ORTEP* (Burnett & Johnson, 1996) plot of title compound is displayed in Fig. 1. The molecule of (I) lies across a crystallographic 2-fold axis so that the asymmetric unit contains one-half of the molecule. Thus, the two benzoyl groups are situated in *anti* orientation. The benzoyl groups are twisted away from the naphthalene moiety, and the dihedral angle is 83.59 (5)°. The torsion angle between the carbonyl group and the naphthalene ring is -89.52 (13)° [O2—C7—C8—C13], and that between the carbonyl group and the benzene ring is 175.91 (10)° [C5—C4—C7—O2].

The molecular packing of (I) is mainly stabilized by intermolecular hydrogen bond and van der Waals interaction (Table 1). The amino groups interact with the methoxy groups [N1—H4 \cdots O1ⁱ = 2.26 (2) Å; symmetry code: (i) $-x + 3/2, -y - 1/2, -z + 2$] of the adjacent molecules along the *b* axis (Fig. 2). Moreover, molecules are linked by N—H \cdots π interactions along the *c* axis (Fig. 3). Besides, relatively weak C—H \cdots O hydrogen bonding, C14—H14B \cdots O2ⁱⁱ [symmetry code: (ii) $-x + 3/2, y - 1/2, -z + 3/2$], and a π — π stacking interaction are observed. In the packing, the molecules form the column structure of stacked naphthalene rings by π — π interaction perpendicular to the *bc* plane with alignment by N—H \cdots π interaction and C—H \cdots O hydrogen bonding along the *c* axis of the unit cell (Fig. 4).

S2. Experimental

The title compound was prepared by reduction reaction of 1,8-bis(4-nitrobenzoyl)-2,7-dimethoxynaphthalene (486.4 mg, 1.0 mmol), which was obtained *via* electrophilic aromatic diarylation reaction of 2,7-dimethoxynaphthalene with 4-nitrobenzoyl chloride, with stannous chloride dihydrate (2.256 g, 10 mmol) in EtOH (8.0 ml) at 343 K for 2 h. The reaction mixture was worked up by reference to the previously outlined procedure (Bellamy *et al.*, 1984). Isolated yield 96%. Brown single crystals suitable for X-ray diffraction were obtained by recrystallization from methanol.

Spectral data: ¹H NMR (300 MHz, CDCl₃) δ 3.71 (6H, s), 4.02 (4H, s), 6.54 (4H, broad), 7.18 (2H, d, *J* = 8.7 Hz), 7.53 (4H, broad), 7.88 (2H, d, *J* = 9.6 Hz); ¹³C NMR (75 MHz, DMSO) δ 56.37, 111.59, 112.22, 122.11, 125.06, 127.41,

128.86, 131.15, 131.29, 153.09, 155.06, 192.49; IR (KBr): 3463.53 (N—H), 3374.82 (N—H), 1644.98 (C=O), 1595.81 (Ar), 1508.06 (Ar) cm^{-1} ; HRMS (m/z): $[M + H]^+$ Calcd for $\text{C}_{26}\text{H}_{23}\text{O}_4\text{N}_2$, 427.1658; found, 427.1633; m.p.= 580.5–583.0 K(decomp).

S3. Refinement

All the H-atoms could be located in difference Fourier maps. The amine and aromatic hydrogen atoms were freely refined. The hydrogen atom of methyl groups were subsequently refined as riding atoms with C—H = 0.96 Å and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

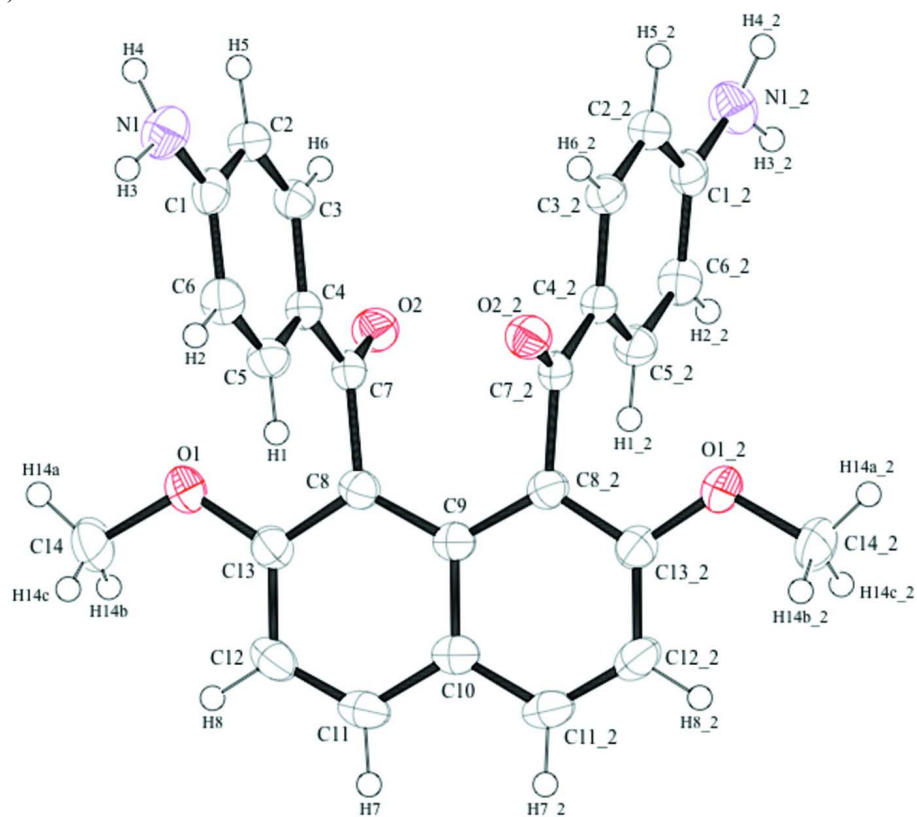
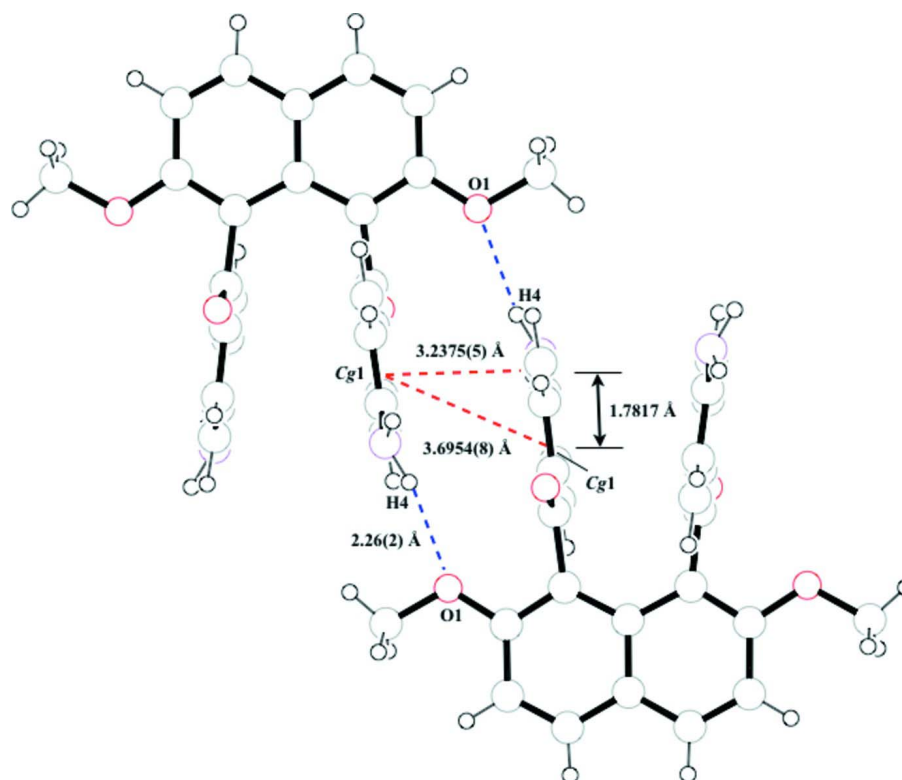
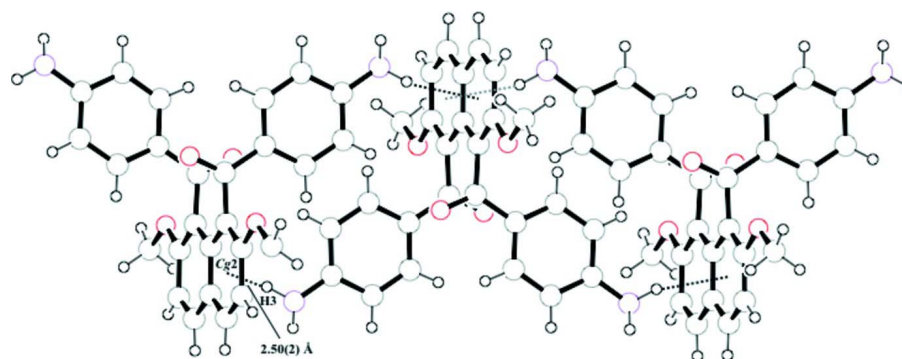


Figure 1

Molecular structure of title compound with displacement ellipsoids drawn at the 50% probability level. The symbol_2 refers to symmetry code: $-x + 1, y, -z + 3/2$.

**Figure 2**

Side view of the N—H···O hydrogen bond (blue dashed lines) and the π — π interaction (red dashed lines).

**Figure 3**

Side view of the N—H··· π interactions (dashed lines).

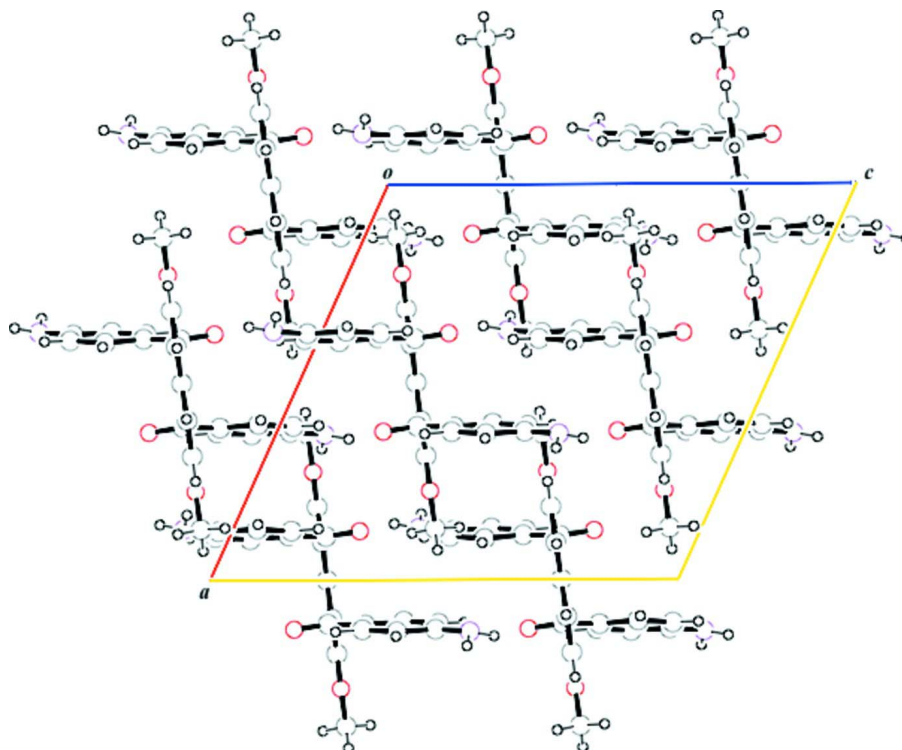


Figure 4

A partial packing diagram of the title compound, viewed down the *b* axis.

[8-(4-aminobenzoyl)-2,7-dimethoxynaphthalen-1-yl](4-aminophenyl)methanone

Crystal data

$C_{26}H_{22}N_2O_4$

$M_r = 426.46$

Monoclinic, $C2/c$

Hall symbol: $-C\ 2yc$

$a = 14.2996\ (3)\ \text{\AA}$

$b = 10.2811\ (2)\ \text{\AA}$

$c = 15.4306\ (3)\ \text{\AA}$

$\beta = 114.523\ (1)^\circ$

$V = 2063.90\ (7)\ \text{\AA}^3$

$Z = 4$

$F(000) = 896$

$D_x = 1.372\ \text{Mg m}^{-3}$

Melting point = 580.5–583.0 K

Cu $K\alpha$ radiation, $\lambda = 1.54187\ \text{\AA}$

Cell parameters from 11162 reflections

$\theta = 3.1\text{--}68.2^\circ$

$\mu = 0.76\ \text{mm}^{-1}$

$T = 193\ \text{K}$

Platelet, brown

$0.40 \times 0.30 \times 0.10\ \text{mm}$

Data collection

Rigaki R-AXIS RAPID
diffractometer

Radiation source: rotating anode

Graphite monochromator

Detector resolution: $10.00\ \text{pixels mm}^{-1}$

ω scans

Absorption correction: numerical
(*NUMABS*; Higashi, 1999)

$T_{\min} = 0.751$, $T_{\max} = 0.928$

17453 measured reflections

1892 independent reflections

1690 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.056$

$\theta_{\max} = 68.2^\circ$, $\theta_{\min} = 5.5^\circ$

$h = -17 \rightarrow 17$

$k = -12 \rightarrow 12$

$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.114$
 $S = 1.12$
 1892 reflections
 178 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0679P)^2 + 0.6125P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.24 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.37 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.77436 (7)	-0.11857 (9)	0.88398 (7)	0.0426 (3)
O2	0.62425 (7)	0.07379 (8)	0.72735 (6)	0.0341 (3)
N1	0.63099 (9)	0.33681 (13)	1.10437 (8)	0.0405 (3)
C1	0.62489 (9)	0.26028 (13)	1.02908 (8)	0.0307 (3)
C2	0.63071 (9)	0.31736 (12)	0.94876 (9)	0.0311 (3)
C3	0.62524 (9)	0.24184 (12)	0.87347 (8)	0.0293 (3)
C4	0.61301 (8)	0.10665 (11)	0.87440 (8)	0.0268 (3)
C5	0.60412 (9)	0.05115 (13)	0.95329 (9)	0.0311 (3)
C6	0.60986 (9)	0.12582 (13)	1.02970 (9)	0.0333 (3)
C7	0.61101 (8)	0.02784 (12)	0.79446 (8)	0.0266 (3)
C8	0.59675 (9)	-0.11834 (12)	0.79669 (8)	0.0278 (3)
C9	0.5000	-0.18296 (16)	0.7500	0.0267 (4)
C10	0.5000	-0.32223 (16)	0.7500	0.0302 (4)
C11	0.59352 (11)	-0.39011 (13)	0.79714 (9)	0.0355 (3)
C12	0.68471 (11)	-0.32756 (13)	0.84270 (9)	0.0366 (3)
C13	0.68596 (9)	-0.19016 (13)	0.84184 (9)	0.0328 (3)
C14	0.86976 (11)	-0.18638 (16)	0.93136 (11)	0.0495 (4)
H14A	0.9248	-0.1246	0.9574	0.059*
H14B	0.8815	-0.2415	0.8866	0.059*
H14C	0.8669	-0.2385	0.9818	0.059*
H1	0.5936 (11)	-0.0400 (16)	0.9542 (10)	0.038 (4)*
H2	0.6079 (12)	0.0862 (14)	1.0838 (11)	0.039 (4)*
H3	0.6425 (15)	0.2952 (19)	1.1606 (14)	0.068 (6)*
H4	0.6672 (15)	0.413 (2)	1.1123 (13)	0.065 (5)*

H5	0.6425 (11)	0.4089 (16)	0.9514 (10)	0.038 (4)*
H6	0.6305 (11)	0.2772 (14)	0.8178 (10)	0.034 (3)*
H7	0.5900 (12)	-0.4824 (17)	0.7944 (11)	0.047 (4)*
H8	0.7479 (13)	-0.3756 (16)	0.8739 (11)	0.048 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0267 (5)	0.0363 (6)	0.0529 (6)	0.0065 (4)	0.0048 (4)	0.0005 (4)
O2	0.0404 (5)	0.0317 (5)	0.0299 (5)	-0.0019 (4)	0.0141 (4)	0.0015 (4)
N1	0.0409 (7)	0.0464 (7)	0.0354 (6)	-0.0038 (6)	0.0170 (5)	-0.0089 (5)
C1	0.0217 (6)	0.0375 (7)	0.0311 (6)	0.0004 (5)	0.0091 (5)	-0.0033 (5)
C2	0.0309 (6)	0.0258 (7)	0.0348 (7)	-0.0006 (5)	0.0117 (5)	-0.0005 (5)
C3	0.0289 (6)	0.0275 (7)	0.0298 (6)	0.0001 (5)	0.0106 (5)	0.0034 (5)
C4	0.0221 (6)	0.0258 (6)	0.0291 (6)	0.0002 (4)	0.0074 (4)	0.0010 (4)
C5	0.0296 (6)	0.0279 (7)	0.0350 (7)	-0.0015 (5)	0.0126 (5)	0.0032 (5)
C6	0.0321 (6)	0.0383 (7)	0.0308 (6)	-0.0010 (5)	0.0144 (5)	0.0039 (5)
C7	0.0198 (5)	0.0271 (6)	0.0281 (6)	0.0002 (4)	0.0053 (4)	0.0016 (4)
C8	0.0305 (6)	0.0259 (7)	0.0271 (6)	0.0023 (5)	0.0120 (5)	0.0006 (4)
C9	0.0329 (9)	0.0249 (9)	0.0241 (8)	0.000	0.0136 (7)	0.000
C10	0.0407 (10)	0.0258 (9)	0.0278 (8)	0.000	0.0180 (7)	0.000
C11	0.0520 (8)	0.0235 (7)	0.0332 (7)	0.0058 (5)	0.0200 (6)	0.0030 (5)
C12	0.0408 (8)	0.0318 (7)	0.0350 (7)	0.0124 (6)	0.0135 (6)	0.0053 (5)
C13	0.0319 (7)	0.0319 (7)	0.0321 (6)	0.0033 (5)	0.0108 (5)	0.0004 (5)
C14	0.0308 (7)	0.0487 (9)	0.0550 (9)	0.0133 (6)	0.0040 (6)	-0.0041 (7)

Geometric parameters (Å, °)

O1—C13	1.3712 (15)	C6—H2	0.939 (15)
O1—C14	1.4331 (15)	C7—C8	1.5189 (17)
O2—C7	1.2223 (14)	C8—C13	1.3857 (17)
N1—C1	1.3756 (16)	C8—C9	1.4308 (14)
N1—H3	0.92 (2)	C9—C8 ⁱ	1.4308 (14)
N1—H4	0.92 (2)	C9—C10	1.432 (2)
C1—C6	1.3996 (19)	C10—C11 ⁱ	1.4132 (16)
C1—C2	1.4045 (17)	C10—C11	1.4132 (16)
C2—C3	1.3724 (17)	C11—C12	1.359 (2)
C2—H5	0.954 (16)	C11—H7	0.950 (18)
C3—C4	1.4017 (17)	C12—C13	1.4128 (19)
C3—H6	0.964 (14)	C12—H8	0.965 (17)
C4—C5	1.3972 (16)	C14—H14A	0.9600
C4—C7	1.4662 (16)	C14—H14B	0.9600
C5—C6	1.3806 (18)	C14—H14C	0.9600
C5—H1	0.950 (16)		
C13—O1—C14	118.41 (11)	C13—C8—C9	120.11 (12)
C1—N1—H3	117.1 (12)	C13—C8—C7	115.75 (10)
C1—N1—H4	115.9 (12)	C9—C8—C7	123.98 (11)

H3—N1—H4	113.7 (17)	C8—C9—C8 ⁱ	124.66 (15)
N1—C1—C6	121.01 (12)	C8—C9—C10	117.67 (8)
N1—C1—C2	120.00 (12)	C8 ⁱ —C9—C10	117.67 (8)
C6—C1—C2	118.97 (11)	C11 ⁱ —C10—C11	120.81 (17)
C3—C2—C1	120.48 (12)	C11 ⁱ —C10—C9	119.59 (8)
C3—C2—H5	122.7 (8)	C11—C10—C9	119.59 (8)
C1—C2—H5	116.7 (8)	C12—C11—C10	122.17 (13)
C2—C3—C4	121.05 (11)	C12—C11—H7	121.2 (9)
C2—C3—H6	122.9 (9)	C10—C11—H7	116.6 (10)
C4—C3—H6	116.1 (9)	C11—C12—C13	118.78 (12)
C5—C4—C3	118.09 (11)	C11—C12—H8	121.0 (10)
C5—C4—C7	122.03 (11)	C13—C12—H8	120.2 (10)
C3—C4—C7	119.88 (11)	O1—C13—C8	115.30 (11)
C6—C5—C4	121.49 (12)	O1—C13—C12	123.02 (11)
C6—C5—H1	119.3 (9)	C8—C13—C12	121.67 (12)
C4—C5—H1	119.2 (9)	O1—C14—H14A	109.5
C5—C6—C1	119.88 (11)	O1—C14—H14B	109.5
C5—C6—H2	120.3 (9)	H14A—C14—H14B	109.5
C1—C6—H2	119.7 (9)	O1—C14—H14C	109.5
O2—C7—C4	122.92 (11)	H14A—C14—H14C	109.5
O2—C7—C8	118.10 (10)	H14B—C14—H14C	109.5
C4—C7—C8	118.92 (10)		
N1—C1—C2—C3	179.66 (11)	C7—C8—C9—C8 ⁱ	-6.37 (8)
C6—C1—C2—C3	-2.20 (18)	C13—C8—C9—C10	-1.54 (11)
C1—C2—C3—C4	0.48 (18)	C7—C8—C9—C10	173.63 (8)
C2—C3—C4—C5	1.46 (17)	C8—C9—C10—C11 ⁱ	-178.56 (8)
C2—C3—C4—C7	-177.87 (10)	C8 ⁱ —C9—C10—C11 ⁱ	1.44 (8)
C3—C4—C5—C6	-1.70 (17)	C8—C9—C10—C11	1.44 (8)
C7—C4—C5—C6	177.61 (10)	C8 ⁱ —C9—C10—C11	-178.56 (8)
C4—C5—C6—C1	0.00 (18)	C11 ⁱ —C10—C11—C12	179.69 (13)
N1—C1—C6—C5	-179.93 (11)	C9—C10—C11—C12	-0.31 (13)
C2—C1—C6—C5	1.96 (18)	C10—C11—C12—C13	-0.74 (18)
C5—C4—C7—O2	-175.91 (10)	C14—O1—C13—C8	-179.48 (11)
C3—C4—C7—O2	3.40 (17)	C14—O1—C13—C12	-0.25 (18)
C5—C4—C7—C8	1.21 (16)	C9—C8—C13—O1	179.77 (9)
C3—C4—C7—C8	-179.48 (10)	C7—C8—C13—O1	4.22 (15)
O2—C7—C8—C13	89.52 (13)	C9—C8—C13—C12	0.53 (17)
C4—C7—C8—C13	-87.74 (13)	C7—C8—C13—C12	-175.03 (11)
O2—C7—C8—C9	-85.85 (13)	C11—C12—C13—O1	-178.55 (11)
C4—C7—C8—C9	96.90 (12)	C11—C12—C13—C8	0.64 (19)
C13—C8—C9—C8 ⁱ	178.46 (11)		

Symmetry code: (i) $-x+1, y, -z+3/2$.

Hydrogen-bond geometry (Å, °)

Cg is the centroid of the C8–C13 ring.

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H4···O1 ⁱⁱ	0.93 (2)	2.26 (2)	3.1708 (17)	169.1 (18)
C14—H14B···O2 ⁱⁱⁱ	0.96	2.57	3.5013 (18)	165
N1—H3···Cg ^{iv}	0.92 (2)	2.50 (2)	3.3301 (13)	149.8 (18)

Symmetry codes: (ii) $-x+3/2, -y+1/2, -z+2$; (iii) $-x+3/2, y-1/2, -z+3/2$; (iv) $x, -y, z+1/2$.