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(4-Chlorophenyl)(2,7-dimethoxy-8-nitro-naphthalen-1-yl)methanone

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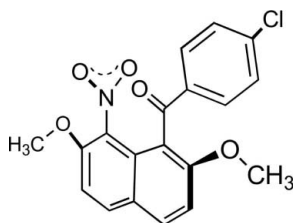
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 Key indicators: single-crystal X-ray study; $T = 193$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.033; wR factor = 0.093; data-to-parameter ratio = 12.8.

In the title compound, $\text{C}_{19}\text{H}_{14}\text{ClNO}_5$, the aroyl group is attached to the naphthalene ring system with a non-coplanar configuration. The dihedral angle between naphthalene ring system and benzene ring is $70.62(6)^\circ$. The nitro group is oriented in parallel with the adjacent carbonyl plane. The torsion angle of the carbonyl group and naphthalene ring is $54.68(19)^\circ$ ($\text{C}-\text{C}-\text{C}-\text{O}$), and that of nitro group and naphthalene ring is $54.26(18)^\circ$ ($\text{O}-\text{N}-\text{C}-\text{C}$). In the crystal, $\pi-\pi$ interactions between naphthalene systems [centroid-centroid distances = $3.5633(9)$, $3.5634(9)$, and $3.9758(9)$ Å], $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, intermolecular $\text{N}-\text{O}\cdots\text{Cl}$ interactions [$2.9937(12)$ Å] and $\text{C}-\text{H}\cdots\pi$ contacts are observed.

Related literature

For electrophilic aromatic substitution of naphthalene derivatives giving aryl naphthyl ketone compounds, see: Okamoto & Yonezawa (2009). For related structures, see: Kato *et al.* (2010); Mitsui *et al.* (2008, 2010); Nishijima *et al.* (2010); Watanabe *et al.* (2010).



Experimental

Crystal data

$\text{C}_{19}\text{H}_{14}\text{ClNO}_5$	$c = 14.0842(3)$ Å
$M_r = 371.76$	$\beta = 100.206(1)^\circ$
Monoclinic, $P2_1/c$	$V = 1669.12(5)$ Å ³
$a = 8.57511(16)$ Å	$Z = 4$
$b = 14.0424(3)$ Å	Cu $K\alpha$ radiation

$\mu = 2.31$ mm⁻¹
 $T = 193$ K

$0.60 \times 0.40 \times 0.30$ mm

Data collection

Rigaku R-Axis RAPID diffractometer	29408 measured reflections
Absorption correction: numerical (NUMABS; Higashi, 1999)	3058 independent reflections
$T_{\min} = 0.294$, $T_{\max} = 0.544$	2793 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.028$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$	238 parameters
$wR(F^2) = 0.093$	H-atom parameters constrained
$S = 1.03$	$\Delta\rho_{\max} = 0.21$ e Å ⁻³
3058 reflections	$\Delta\rho_{\min} = -0.19$ e Å ⁻³

Table 1

Hydrogen-bond geometry (Å, °).

Cg1 is the centroid of the C1–C4/C9/C10 ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C13–H13 \cdots O5 ⁱ	0.93	2.44	3.137 (2)	132
C18–H18C \cdots O3 ⁱ	0.96	2.51	3.449 (2)	167
C19–H19B \cdots Cg1 ⁱⁱ	0.96	2.81	3.5845 (19)	139

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

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

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: OM2387).

References

- Burla, M. C., Caliendo, 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). *ORTEP III*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
- Higashi, T. (1999). *NUMABS*. Rigaku Corporation, Tokyo, Japan.
- Kato, Y., Nagasawa, A., Hijikata, D., Okamoto, A. & Yonezawa, N. (2010). *Acta Cryst.* **E66**, o2659.
- Mitsui, R., Nagasawa, A., Watanabe, S., Okamoto, A. & Yonezawa, N. (2010). *Acta Cryst.* **E66**, o873.
- Mitsui, R., Nakaema, K., Noguchi, K., Okamoto, A. & Yonezawa, N. (2008). *Acta Cryst.* **E64**, o1278.
- Nishijima, T., Kataoka, K., Nagasawa, A., Okamoto, A. & Yonezawa, N. (2010). *Acta Cryst.* **E66**, o2904–o2905.
- Okamoto, A. & Yonezawa, N. (2009). *Chem. Lett.* **38**, 914–915.
- Rigaku (1998). *PROCESS-AUTO*. Rigaku Corporation, Tokyo, Japan.
- Rigaku/MS (2004). *CrystalStructure*. Rigaku/MS, The Woodlands, Texas, USA.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Watanabe, S., Nakaema, K., Nishijima, T., Okamoto, A. & Yonezawa, N. (2010). *Acta Cryst.* **E66**, o615.

supporting information

Acta Cryst. (2011). E67, o155 [https://doi.org/10.1107/S1600536810051998]

(4-Chlorophenyl)(2,7-dimethoxy-8-nitronaphthalen-1-yl)methanone

Takahiro Nishijima, Atsushi Nagasawa, Teruhisa Takada, Akiko Okamoto and Noriyuki Yonezawa

S1. Comment

In the course of our study on electrophilic aromatic arylation of 2,7-dimethoxynaphthalene, *peri*-arylnaphthalene compounds have proven to be formed regioselectively with aid of suitable acidic mediators (Okamoto & Yonezawa, 2009). Recently, we have reported the crystal structures of several 1,8-diaroylated naphthalene homologues exemplified by 1,8-bis(4-aminobenzoyl)-2,7-dimethoxynaphthalene (Nishijima *et al.*, 2010). The aromatic rings in these molecules are arranged in a non-coplanar alignment to each other. Furthermore, we have also clarified the crystal structures of 1-monoaroylated naphthalene compounds. They have essentially the same non-coplanar structure with the 1,8-diaroylated naphthalene, *e.g.*, 1-(4-chlorobenzoyl)-2,7-dimethoxynaphthalene (Mitsui *et al.*, 2008), 2,7-dimethoxy-1-(4-nitrobenzoyl)naphthalene (Watanabe *et al.*, 2010) and (2,7-dimethoxynaphthalen-1-yl)(phenyl)methanone (Kato, *et al.*, 2010). In the course of this work, we have revealed the crystal structure of 1-monoarylnaphthalene compounds having a substituent, other than aroyl group, at the 8-position such as (8-bromo-2,7-dimethoxy-1-naphthyl)(4-chlorophenyl)methanone (Mitsui, Nagasawa, Watanabe *et al.* 2010). The aroyl group and naphthalene ring in these molecules have similar configuration to 1,8-diaroylated naphthalene. As a part of our continuous study on the molecular structures of these kinds of homologous molecules, the crystal structure of title compound, a 1-chlorobenzoylated naphthalene bearing nitro group at the 8-position, is discussed in this report.

An *ORTEP* (Burnett & Johnson, 1996) plot of the title compound is displayed in Fig. 1. In the molecule, the dihedral angle between the benzene ring (C11—C16) and the naphthalene ring (C1—C10) is 70.62 (6)°. The nitro group are also twisted away from the naphthalene ring system, then the nitro group and the carbonyl group are arranged almost in parallel. The dihedral angle between the ketonic C=O plane (O3/C1/C11/C17) and naphthalene ring (C1—C10) is 60.33 (7)° [C9—C1—C17—O3 torsion angle is 54.68 (19)°] and between the nitro plane (O4/O5/N1/C8) and naphthalene ring (C1—C10) is 57.34 (8)° [O4—N1—C8—O9 torsion angle is 54.26 (18)°]. On the other hand, the carbonyl group and the 4-chlorophenyl one have almost coplanar configuration [C16—C11—C17—O3 torsion angle is 10.5 (2)°], but the torsion angle is larger than those of another homologous compounds, *i.e.*, the torsion angles of 1-(4-chlorobenzoyl)-2,7-dimethoxynaphthalene and (8-bromo-2,7-dimethoxy-1-naphthyl)(4-chlorophenyl)methanone are -4.4 (2)° and -3.6 (4)°, respectively.

The molecular packing of the title compound is mainly stabilized by weak intermolecular hydrogen bonds and van der Waals interactions. The 4-chlorophenyl groups interact with the nitro groups [C13—H13⋯O5 is 2.44 Å; (i) *x*, 3/2 - *y*, 1/2 + *z*] of adjacent molecules along the *c* axis (Fig. 2). Interaction between the methoxy groups and the carbonyl groups [C18—H18C⋯O3 = 2.51 Å; (i) *x*, 3/2 - *y*, 1/2 + *z*] form naphthalene ring systems into zigzag arrangement (Fig. 2). On the other hand, C—H⋯ π interaction and π - π interactions deposit layer upon layer of naphthalene rings [C1/C2/C3/C4/C9/C10 ring with centroid Cg1 and C5-C10 ring with centroid Cg2 (Fig. 3). Furthermore, O4 and C11

interact with each other [O4...C11 = 2.9937 (12) Å; (iii) 2 - x, -y, 1-z] (Fig. 4).

S2. Experimental

To a solution of 1-(4-chlorobenzoyl)-2,7-dimethoxynaphthalene (653.4 mg, 2.0 mmol) in methylene chloride (10 ml), aqueous 61% nitric acid (2.0 ml) was dropped by portions at 273 K. After the reaction mixture was stirred at 273 K for 2 h, it was poured into ice-cold water (25 ml). The aqueous solution was extracted with CHCl₃ (15 ml × 3). The combined extracts were washed with water followed by washing with brine. The organic layers thus obtained were dried over anhydrous MgSO₄. The solvent was removed under reduced pressure to give cake. The crude product was purified by reprecipitation (good solvent:CHCl₃, poor solvent:hexane) and column chromatography (silica gel, CHCl₃) (isolated yield 448.8 mg, 60%). Single crystals suitable for X-ray diffraction analysis were obtained by crystallization from acetone as yellow plates.

Spectral data: ¹H NMR δ (300 MHz, CDCl₃); 3.73 (3H, s), 3.97 (3H, s), 7.22 (1H, d, *J* = 9.3 Hz), 7.24 (1H, d, *J* = 8.6 Hz), 7.38 (2H, d, *J* = 8.9 Hz), 7.82 (2H, d, *J* = 8.6 Hz), 7.95 (1H, d, *J* = 9.3 Hz), 7.96 (1H, d, *J* = 8.9 Hz). ¹³C NMR δ (75 MHz, CDCl₃); 56.48, 57.22, 111.26, 111.86, 118.02, 124.16, 124.59, 128.64, 130.51, 132.59, 133.24, 134.39, 136.79, 139.17, 151.61, 157.76, 193.23. IR (KBr); 1655 (C=O), 1623 (Ar), 1527 (Ar), 1372 (NO₂), 1275 (O—Me) cm⁻¹. HRMS (*m/z*); [*M* + H]⁺ Calcd for C₁₉H₁₅ClNO₅, 272.0639; found, 272.0601. m.p. = 483.1–486.4 K.

S3. Refinement

All H atoms were found in a difference map and were subsequently refined as riding atoms, with C—H = 0.93 (aromatic) Å and C—H = 0.96 (methyl) Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

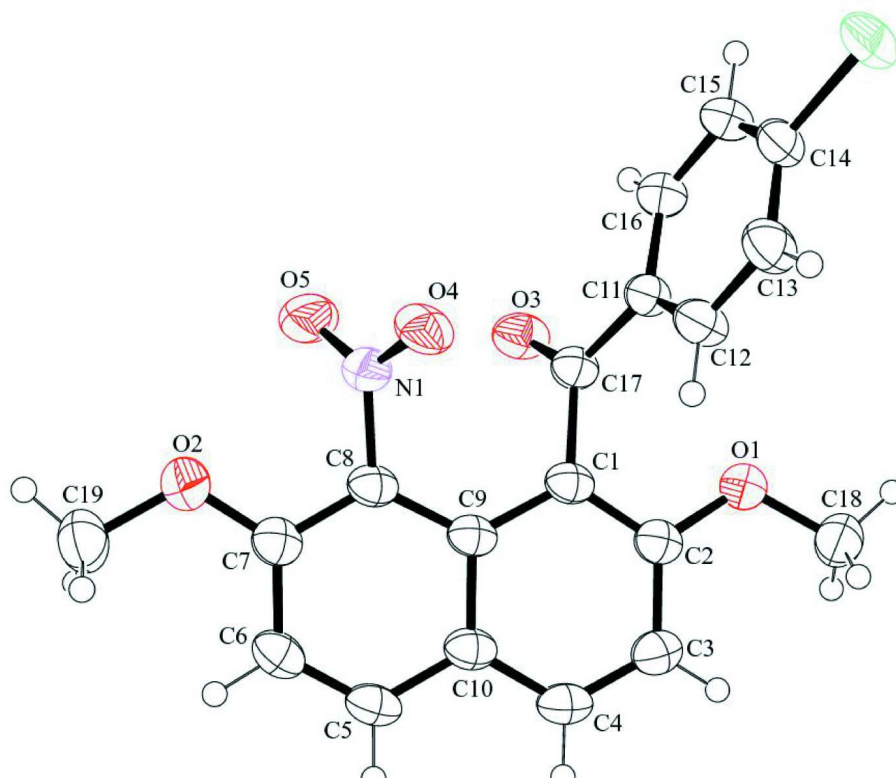


Figure 1

Molecular structure of the title compound with displacement ellipsoids at the 50% probability level.

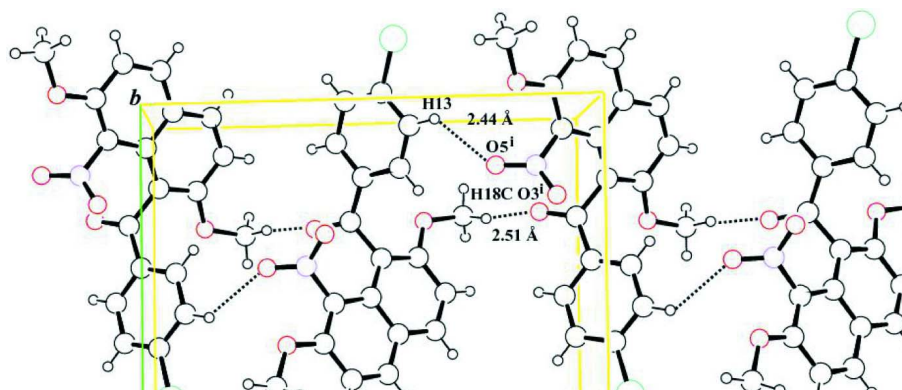


Figure 2

A partial crystal packing diagram of the title compound, viewed down the *b* axis. The intermolecular C—H...O hydrogen bonds are shown as dashed lines.

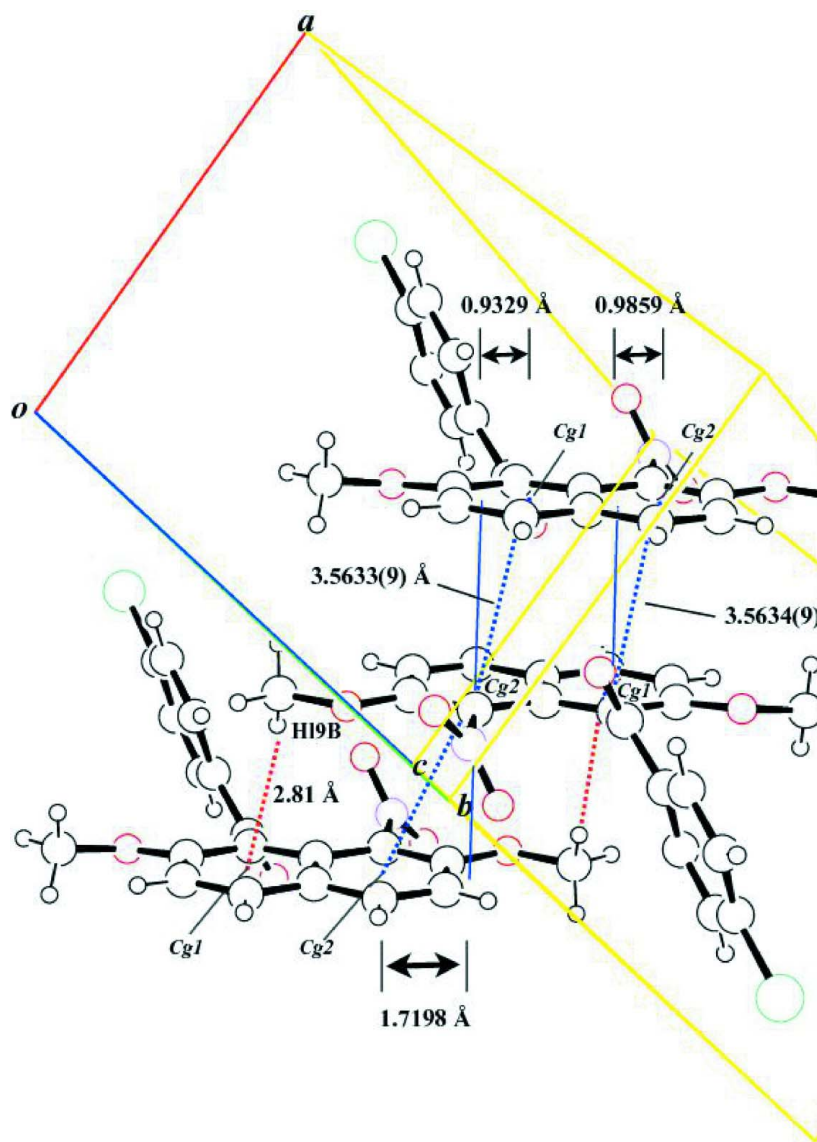


Figure 3

Side view of the C—H... π interactions (red dashed lines) and the π — π interactions (blue dashed lines).

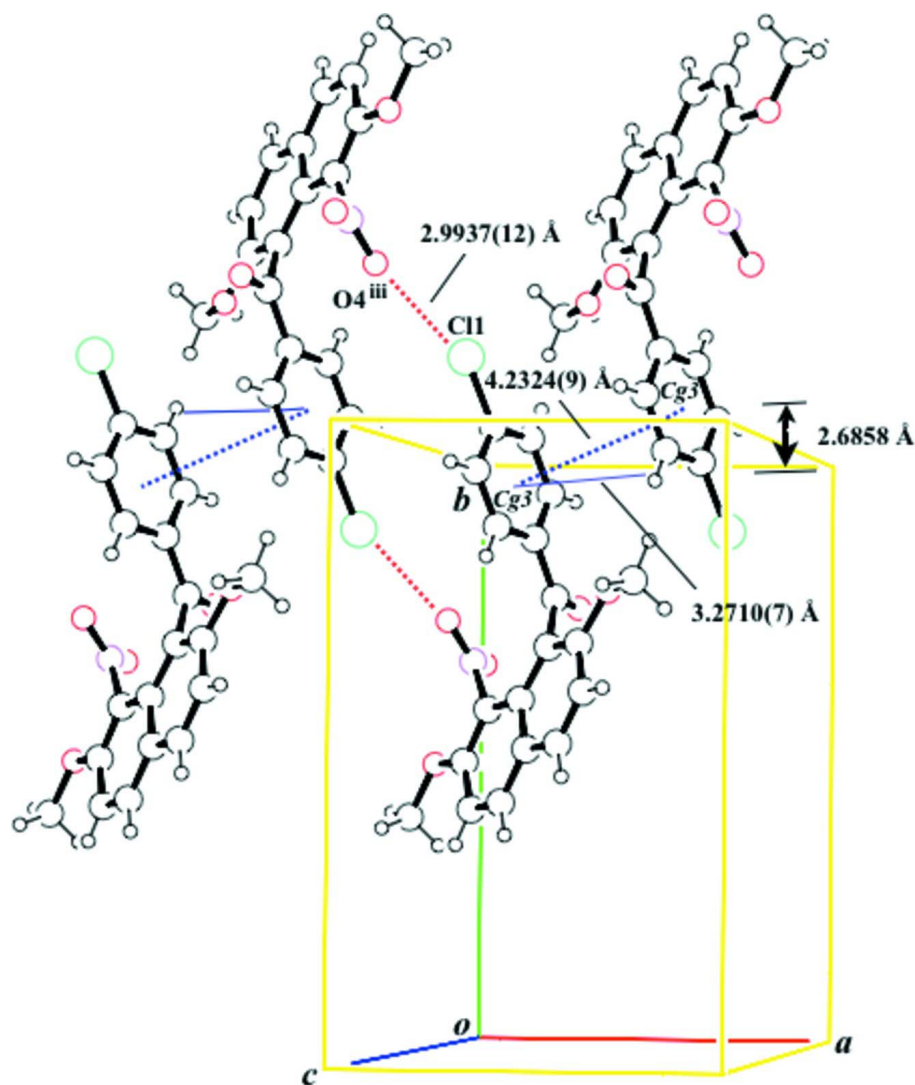


Figure 4

Side view of the O...Cl interactions (red dashed lines) and the π — π interactions (blue dashed lines).

(4-Chlorophenyl)(2,7-dimethoxy-8-nitronaphthalen-1-yl)methanone

Crystal data

$C_{19}H_{14}ClNO_5$

$M_r = 371.76$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/c$

$a = 8.57511(16)\ \text{\AA}$

$b = 14.0424(3)\ \text{\AA}$

$c = 14.0842(3)\ \text{\AA}$

$\beta = 100.206(1)^\circ$

$V = 1669.12(5)\ \text{\AA}^3$

$Z = 4$

$F(000) = 768$

$D_x = 1.479\ \text{Mg m}^{-3}$

Melting point = 486.4–483.1 K

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

Cell parameters from 22820 reflections

$\theta = 3.1$ – 68.2°

$\mu = 2.31\ \text{mm}^{-1}$

$T = 193\ \text{K}$

Platelet, yellow

$0.60 \times 0.40 \times 0.30\ \text{mm}$

*Data collection*Rigaku R-AXIS RAPID
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 10.00 pixels mm⁻¹ ω scansAbsorption correction: numerical
(NUMABS; Higashi, 1999) $T_{\min} = 0.294$, $T_{\max} = 0.544$

29408 measured reflections

3058 independent reflections

2793 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.028$ $\theta_{\max} = 68.2^\circ$, $\theta_{\min} = 4.5^\circ$ $h = -10 \rightarrow 10$ $k = -16 \rightarrow 16$ $l = -16 \rightarrow 16$ *Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.033$ $wR(F^2) = 0.093$ $S = 1.03$

3058 reflections

238 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0492P)^2 + 0.5094P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.001$ $\Delta\rho_{\max} = 0.21 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.19 \text{ e } \text{\AA}^{-3}$ Extinction correction: *SHELXL97* (Sheldrick,
2008), $F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0049 (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	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.16621 (6)	1.13841 (3)	0.55672 (4)	0.06727 (18)
O1	0.57537 (12)	0.75406 (7)	0.62980 (7)	0.0434 (3)
O2	-0.00458 (14)	0.47410 (8)	0.31568 (8)	0.0511 (3)
O3	0.41091 (13)	0.73567 (8)	0.38109 (7)	0.0468 (3)
O4	0.06202 (13)	0.71291 (7)	0.40079 (8)	0.0485 (3)
O5	0.11487 (19)	0.64395 (9)	0.27365 (8)	0.0676 (4)
N1	0.11528 (15)	0.64617 (8)	0.35979 (9)	0.0412 (3)
C1	0.38260 (16)	0.66673 (10)	0.52900 (9)	0.0338 (3)
C2	0.49357 (17)	0.67065 (10)	0.61336 (10)	0.0360 (3)
C3	0.52689 (18)	0.59082 (11)	0.67462 (10)	0.0405 (3)
H3	0.5989	0.5956	0.7322	0.049*
C4	0.45238 (18)	0.50699 (11)	0.64837 (11)	0.0415 (3)
H4	0.4776	0.4538	0.6873	0.050*
C5	0.26174 (18)	0.41072 (10)	0.53798 (11)	0.0408 (3)

H5	0.2885	0.3585	0.5781	0.049*
C6	0.15099 (18)	0.39956 (10)	0.45694 (11)	0.0410 (3)
H6	0.1042	0.3404	0.4419	0.049*
C7	0.10756 (17)	0.47805 (10)	0.39587 (10)	0.0377 (3)
C8	0.17971 (16)	0.56535 (9)	0.42014 (9)	0.0347 (3)
C9	0.29884 (16)	0.57978 (9)	0.50291 (9)	0.0329 (3)
C10	0.33774 (17)	0.49855 (10)	0.56356 (10)	0.0366 (3)
C11	0.32856 (16)	0.84584 (9)	0.48861 (10)	0.0350 (3)
C12	0.25859 (19)	0.86117 (10)	0.56900 (11)	0.0427 (3)
H12	0.2450	0.8103	0.6090	0.051*
C13	0.2088 (2)	0.95114 (11)	0.59030 (12)	0.0470 (4)
H13	0.1618	0.9612	0.6441	0.056*
C14	0.23033 (19)	1.02564 (10)	0.53021 (12)	0.0457 (4)
C15	0.3010 (2)	1.01289 (11)	0.45082 (12)	0.0501 (4)
H15	0.3156	1.0642	0.4116	0.060*
C16	0.35004 (18)	0.92290 (11)	0.43016 (11)	0.0423 (3)
H16	0.3980	0.9136	0.3766	0.051*
C17	0.37585 (16)	0.74943 (10)	0.46024 (10)	0.0350 (3)
C18	0.6731 (2)	0.76752 (13)	0.72157 (11)	0.0501 (4)
H18A	0.7157	0.8309	0.7256	0.060*
H18B	0.7584	0.7223	0.7296	0.060*
H18C	0.6113	0.7584	0.7714	0.060*
C19	-0.0836 (2)	0.38502 (13)	0.29095 (13)	0.0560 (4)
H19A	-0.1582	0.3921	0.2319	0.067*
H19B	-0.1386	0.3661	0.3416	0.067*
H19C	-0.0069	0.3373	0.2827	0.067*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0844 (3)	0.0300 (2)	0.0792 (3)	0.01301 (19)	-0.0079 (2)	-0.01089 (18)
O1	0.0501 (6)	0.0388 (6)	0.0398 (5)	-0.0041 (4)	0.0037 (4)	0.0022 (4)
O2	0.0594 (7)	0.0403 (6)	0.0494 (6)	-0.0012 (5)	-0.0019 (5)	-0.0042 (5)
O3	0.0609 (7)	0.0426 (6)	0.0418 (6)	0.0080 (5)	0.0226 (5)	0.0057 (4)
O4	0.0506 (6)	0.0319 (5)	0.0630 (7)	0.0123 (4)	0.0101 (5)	-0.0006 (5)
O5	0.1160 (12)	0.0474 (7)	0.0355 (6)	0.0052 (7)	0.0027 (6)	0.0034 (5)
N1	0.0494 (7)	0.0325 (6)	0.0400 (7)	0.0050 (5)	0.0031 (5)	0.0014 (5)
C1	0.0396 (7)	0.0292 (7)	0.0349 (7)	0.0073 (5)	0.0134 (5)	0.0021 (5)
C2	0.0401 (7)	0.0335 (7)	0.0366 (7)	0.0037 (6)	0.0128 (6)	0.0006 (5)
C3	0.0434 (8)	0.0412 (8)	0.0368 (7)	0.0069 (6)	0.0072 (6)	0.0046 (6)
C4	0.0472 (8)	0.0360 (8)	0.0421 (8)	0.0097 (6)	0.0106 (6)	0.0102 (6)
C5	0.0505 (8)	0.0282 (7)	0.0463 (8)	0.0070 (6)	0.0157 (7)	0.0067 (6)
C6	0.0488 (8)	0.0283 (7)	0.0488 (8)	0.0015 (6)	0.0164 (7)	-0.0014 (6)
C7	0.0415 (8)	0.0342 (7)	0.0393 (7)	0.0055 (6)	0.0126 (6)	-0.0030 (6)
C8	0.0420 (7)	0.0287 (7)	0.0356 (7)	0.0086 (5)	0.0128 (6)	0.0017 (5)
C9	0.0387 (7)	0.0289 (7)	0.0341 (7)	0.0079 (5)	0.0144 (5)	0.0017 (5)
C10	0.0427 (8)	0.0313 (7)	0.0382 (7)	0.0086 (6)	0.0140 (6)	0.0047 (5)
C11	0.0370 (7)	0.0296 (7)	0.0375 (7)	0.0001 (5)	0.0046 (5)	0.0030 (5)

C12	0.0536 (9)	0.0304 (7)	0.0467 (8)	0.0022 (6)	0.0154 (7)	0.0029 (6)
C13	0.0555 (9)	0.0356 (8)	0.0515 (9)	0.0040 (7)	0.0138 (7)	-0.0053 (7)
C14	0.0490 (9)	0.0272 (7)	0.0552 (9)	0.0027 (6)	-0.0061 (7)	-0.0038 (6)
C15	0.0652 (10)	0.0301 (8)	0.0511 (9)	-0.0034 (7)	-0.0002 (8)	0.0095 (6)
C16	0.0498 (8)	0.0359 (8)	0.0406 (7)	-0.0023 (6)	0.0063 (6)	0.0056 (6)
C17	0.0375 (7)	0.0330 (7)	0.0360 (7)	0.0013 (6)	0.0102 (6)	0.0028 (5)
C18	0.0544 (9)	0.0530 (9)	0.0413 (8)	-0.0073 (8)	0.0043 (7)	-0.0016 (7)
C19	0.0566 (10)	0.0471 (9)	0.0616 (10)	-0.0032 (8)	0.0026 (8)	-0.0134 (8)

Geometric parameters (Å, °)

C11—C14	1.7384 (15)	C6—H6	0.9300
O1—C2	1.3639 (17)	C7—C8	1.388 (2)
O1—C18	1.4230 (18)	C8—C9	1.422 (2)
O2—C7	1.3485 (18)	C9—C10	1.4287 (18)
O2—C19	1.436 (2)	C11—C12	1.389 (2)
O3—C17	1.2202 (16)	C11—C16	1.3914 (19)
O4—N1	1.2303 (15)	C11—C17	1.4882 (19)
O5—N1	1.2130 (16)	C12—C13	1.383 (2)
N1—C8	1.4661 (17)	C12—H12	0.9300
C1—C2	1.385 (2)	C13—C14	1.378 (2)
C1—C9	1.4311 (19)	C13—H13	0.9300
C1—C17	1.5066 (18)	C14—C15	1.375 (2)
C2—C3	1.413 (2)	C15—C16	1.379 (2)
C3—C4	1.359 (2)	C15—H15	0.9300
C3—H3	0.9300	C16—H16	0.9300
C4—C10	1.411 (2)	C18—H18A	0.9600
C4—H4	0.9300	C18—H18B	0.9600
C5—C6	1.358 (2)	C18—H18C	0.9600
C5—C10	1.412 (2)	C19—H19A	0.9600
C5—H5	0.9300	C19—H19B	0.9600
C6—C7	1.407 (2)	C19—H19C	0.9600
C2—O1—C18	118.08 (11)	C4—C10—C9	119.53 (13)
C7—O2—C19	118.35 (12)	C12—C11—C16	118.90 (13)
O5—N1—O4	123.61 (12)	C12—C11—C17	122.51 (12)
O5—N1—C8	119.60 (12)	C16—C11—C17	118.54 (12)
O4—N1—C8	116.79 (11)	C13—C12—C11	120.84 (14)
C2—C1—C9	119.42 (12)	C13—C12—H12	119.6
C2—C1—C17	117.58 (13)	C11—C12—H12	119.6
C9—C1—C17	122.15 (12)	C14—C13—C12	118.70 (15)
O1—C2—C1	115.73 (12)	C14—C13—H13	120.7
O1—C2—C3	122.38 (13)	C12—C13—H13	120.7
C1—C2—C3	121.75 (14)	C15—C14—C13	121.78 (14)
C4—C3—C2	119.12 (14)	C15—C14—C11	119.68 (12)
C4—C3—H3	120.4	C13—C14—C11	118.54 (13)
C2—C3—H3	120.4	C14—C15—C16	119.06 (14)
C3—C4—C10	121.77 (13)	C14—C15—H15	120.5

C3—C4—H4	119.1	C16—C15—H15	120.5
C10—C4—H4	119.1	C15—C16—C11	120.70 (14)
C6—C5—C10	122.53 (13)	C15—C16—H16	119.6
C6—C5—H5	118.7	C11—C16—H16	119.6
C10—C5—H5	118.7	O3—C17—C11	120.81 (12)
C5—C6—C7	119.55 (14)	O3—C17—C1	118.53 (12)
C5—C6—H6	120.2	C11—C17—C1	120.66 (11)
C7—C6—H6	120.2	O1—C18—H18A	109.5
O2—C7—C8	117.58 (12)	O1—C18—H18B	109.5
O2—C7—C6	123.50 (13)	H18A—C18—H18B	109.5
C8—C7—C6	118.88 (14)	O1—C18—H18C	109.5
C7—C8—C9	123.49 (12)	H18A—C18—H18C	109.5
C7—C8—N1	115.79 (12)	H18B—C18—H18C	109.5
C9—C8—N1	120.47 (12)	O2—C19—H19A	109.5
C8—C9—C10	115.73 (12)	O2—C19—H19B	109.5
C8—C9—C1	125.95 (12)	H19A—C19—H19B	109.5
C10—C9—C1	118.31 (13)	O2—C19—H19C	109.5
C5—C10—C4	120.69 (13)	H19A—C19—H19C	109.5
C5—C10—C9	119.78 (13)	H19B—C19—H19C	109.5
C18—O1—C2—C1	170.71 (12)	C2—C1—C9—C10	-2.42 (18)
C18—O1—C2—C3	-13.54 (19)	C17—C1—C9—C10	166.79 (12)
C9—C1—C2—O1	175.92 (11)	C6—C5—C10—C4	-179.90 (13)
C17—C1—C2—O1	6.22 (17)	C6—C5—C10—C9	-0.1 (2)
C9—C1—C2—C3	0.14 (19)	C3—C4—C10—C5	-179.85 (13)
C17—C1—C2—C3	-169.56 (12)	C3—C4—C10—C9	0.4 (2)
O1—C2—C3—C4	-173.05 (12)	C8—C9—C10—C5	1.55 (18)
C1—C2—C3—C4	2.4 (2)	C1—C9—C10—C5	-177.58 (12)
C2—C3—C4—C10	-2.7 (2)	C8—C9—C10—C4	-178.68 (12)
C10—C5—C6—C7	-0.7 (2)	C1—C9—C10—C4	2.19 (19)
C19—O2—C7—C8	-178.28 (13)	C16—C11—C12—C13	0.9 (2)
C19—O2—C7—C6	-0.3 (2)	C17—C11—C12—C13	-176.66 (14)
C5—C6—C7—O2	-177.91 (13)	C11—C12—C13—C14	-0.1 (2)
C5—C6—C7—C8	0.1 (2)	C12—C13—C14—C15	-0.7 (2)
O2—C7—C8—C9	179.59 (12)	C12—C13—C14—C11	179.50 (13)
C6—C7—C8—C9	1.5 (2)	C13—C14—C15—C16	0.8 (2)
O2—C7—C8—N1	5.20 (18)	C11—C14—C15—C16	-179.43 (12)
C6—C7—C8—N1	-172.92 (12)	C14—C15—C16—C11	0.0 (2)
O5—N1—C8—C7	-58.76 (19)	C12—C11—C16—C15	-0.8 (2)
O4—N1—C8—C7	120.32 (14)	C17—C11—C16—C15	176.83 (14)
O5—N1—C8—C9	126.67 (15)	C12—C11—C17—O3	167.06 (14)
O4—N1—C8—C9	-54.26 (18)	C16—C11—C17—O3	-10.5 (2)
C7—C8—C9—C10	-2.25 (19)	C12—C11—C17—C1	-13.4 (2)
N1—C8—C9—C10	171.89 (12)	C16—C11—C17—C1	169.08 (13)
C7—C8—C9—C1	176.80 (12)	C2—C1—C17—O3	114.72 (15)
N1—C8—C9—C1	-9.1 (2)	C9—C1—C17—O3	-54.68 (18)
C2—C1—C9—C8	178.55 (12)	C2—C1—C17—C11	-64.85 (17)
C17—C1—C9—C8	-12.2 (2)	C9—C1—C17—C11	125.75 (14)

Hydrogen-bond geometry (Å, °)

Cg1 is the centroid of the C1–C4/C9/C10 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>
C13—H13···O5 ⁱ	0.93	2.44	3.137 (2)	132
C18—H18C···O3 ⁱ	0.96	2.51	3.449 (2)	167
C19—H19B···Cg1 ⁱⁱ	0.96	2.81	3.5845 (19)	139

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