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## Structure Reports

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

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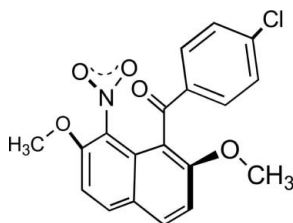
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Key indicators: single-crystal X-ray study;  $T = 193$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.033;  $wR$  factor = 0.093; data-to-parameter ratio = 12.8.

In the title compound,  $\text{C}_{19}\text{H}_{14}\text{ClNO}_5$ , the aryl group is attached to the naphthalene ring system with a non-coplanar configuration. The dihedral angle between naphthalene ring system and benzene ring is  $70.62$  ( $6$ )°. The nitro group is oriented in parallel with the adjacent carbonyl plane. The torsion angle of the carbonyl group and naphthalene ring is  $54.68$  ( $19$ )° ( $\text{C}-\text{C}-\text{C}-\text{O}$ ), and that of nitro group and naphthalene ring is  $54.26$  ( $18$ )° ( $\text{O}-\text{N}-\text{C}-\text{C}$ ). 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

$\text{C}_{19}\text{H}_{14}\text{ClNO}_5$   
 $M_r = 371.76$   
 Monoclinic,  $P2_1/c$   
 $a = 8.57511$  ( $16$ ) Å  
 $b = 14.0424$  ( $3$ ) Å

$c = 14.0842$  ( $3$ ) Å  
 $\beta = 100.206$  ( $1$ )°  
 $V = 1669.12$  ( $5$ ) Å<sup>3</sup>  
 $Z = 4$   
 Cu  $K\alpha$  radiation

$\mu = 2.31$  mm<sup>-1</sup>  
 $T = 193$  K

0.60 × 0.40 × 0.30 mm

## Data collection

Rigaku R-Axis RAPID  
 diffractometer  
 Absorption correction: numerical  
 (NUMABS; Higashi, 1999)  
 $T_{\min} = 0.294$ ,  $T_{\max} = 0.544$

29408 measured reflections  
 3058 independent reflections  
 2793 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.028$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$   
 $wR(F^2) = 0.093$   
 $S = 1.03$   
 3058 reflections

238 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.21$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.19$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C13}-\text{H13}\cdots\text{O5}^i$	0.93	2.44	3.137 (2)	132
$\text{C18}-\text{H18C}\cdots\text{O3}^i$	0.96	2.51	3.449 (2)	167
$\text{C19}-\text{H19B}\cdots\text{Cg1}^{ii}$	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*.

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 a Sasakawa Scientific Research Grant from the Japan Science Society.

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

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**supplementary materials**

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

T. Nishijima, A. Nagasawa, T. Takada, A. Okamoto and N. Yonezawa

### 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-monoaroylnaphthalene compounds having a substituent, other than aryl group, at the 8-position such as (8-bromo-2,7-dimethoxy-1-naphthyl)(4-chlorophenyl)methanone (Mitsui, Nagasawa, Watanabe *et al.* 2010). The aryl 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... $\pi$  interaction and  $\pi$ - $\pi$  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).

## 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  $\text{CHCl}_3$  (15 ml  $\times$  3). The combined extracts were washed with water followed by washing with brine. The organic layers thus obtained were dried over anhydrous  $\text{MgSO}_4$ . The solvent was removed under reduced pressure to give cake. The crude product was purified by reprecipitation (good solvent:  $\text{CHCl}_3$ , poor solvent: hexane) and column chromatography (silica gel,  $\text{CHCl}_3$ ) (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:**  $^1\text{H}$  NMR  $\delta$  (300 MHz,  $\text{CDCl}_3$ ); 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).  $^{13}\text{C}$  NMR  $\delta$  (75 MHz,  $\text{CDCl}_3$ ); 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 ( $\text{NO}_2$ ), 1275 (O—Me)  $\text{cm}^{-1}$ . HRMS ( $m/z$ );  $[M + \text{H}]^+$  Calcd for  $\text{C}_{19}\text{H}_{15}\text{ClNO}_5$ , 272.0639; found, 272.0601. m.p. = 483.1–486.4 K.

## 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  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

## Figures

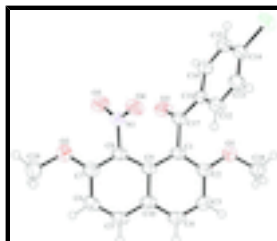


Fig. 1. Molecular structure of the title compound with displacement ellipsoids at the 50% probability level.



Fig. 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.

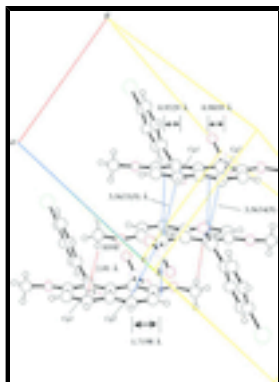


Fig. 3. Side view of the C—H... $\pi$  interactions (red dashed lines) and the  $\pi$ — $\pi$  interactions (blue dashed lines).

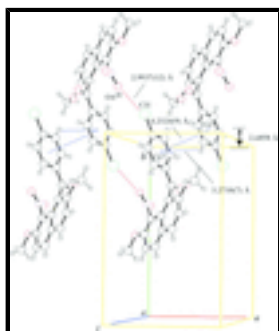


Fig. 4. Side view of the O...Cl interactions (red dashed lines) and the  $\pi$ — $\pi$  interactions (blue dashed lines).

#### (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 2ybc

$a = 8.57511$  (16) Å

$b = 14.0424$  (3) Å

$c = 14.0842$  (3) Å

$\beta = 100.206$  (1)°

$V = 1669.12$  (5) Å<sup>3</sup>

$Z = 4$

$F(000) = 768$

$D_x = 1.479$  Mg m<sup>-3</sup>

Melting point = 486.4–483.1 K

Cu  $K\alpha$  radiation,  $\lambda = 1.54187$  Å

Cell parameters from 22820 reflections

$\theta = 3.1$ – $68.2$ °

$\mu = 2.31$  mm<sup>-1</sup>

$T = 193$  K

Platelet, yellow

$0.60 \times 0.40 \times 0.30$  mm

##### Data collection

Rigaku R-Axis RAPID  
diffractometer

Radiation source: fine-focus sealed tube  
graphite

Detector resolution: 10.00 pixels mm<sup>-1</sup>

$\omega$  scans

Absorption correction: numerical  
(NUMABS; Higashi, 1999)

$T_{\min} = 0.294$ ,  $T_{\max} = 0.544$

3058 independent reflections

2793 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.028$

$\theta_{\max} = 68.2$ °,  $\theta_{\min} = 4.5$ °

$h = -10 \rightarrow 10$

$k = -16 \rightarrow 16$

$l = -16 \rightarrow 16$

# supplementary materials

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29408 measured reflections

## Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.033$	H-atom parameters constrained
$wR(F^2) = 0.093$	$w = 1/[\sigma^2(F_o^2) + (0.0492P)^2 + 0.5094P]$
$S = 1.03$	where $P = (F_o^2 + 2F_c^2)/3$
3058 reflections	$(\Delta/\sigma)_{\max} = 0.001$
238 parameters	$\Delta\rho_{\max} = 0.21 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta\rho_{\min} = -0.19 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: <i>SHELXL97</i> (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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
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 ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
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)

## supplementary materials

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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





## supplementary materials

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C13—H13...O5 <sup>i</sup>	0.93	2.44	3.137 (2)	132
C18—H18C...O3 <sup>i</sup>	0.96	2.51	3.449 (2)	167
C19—H19B...Cg1 <sup>ii</sup>	0.96	2.81	3.5845 (19)	139

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

Fig. 1

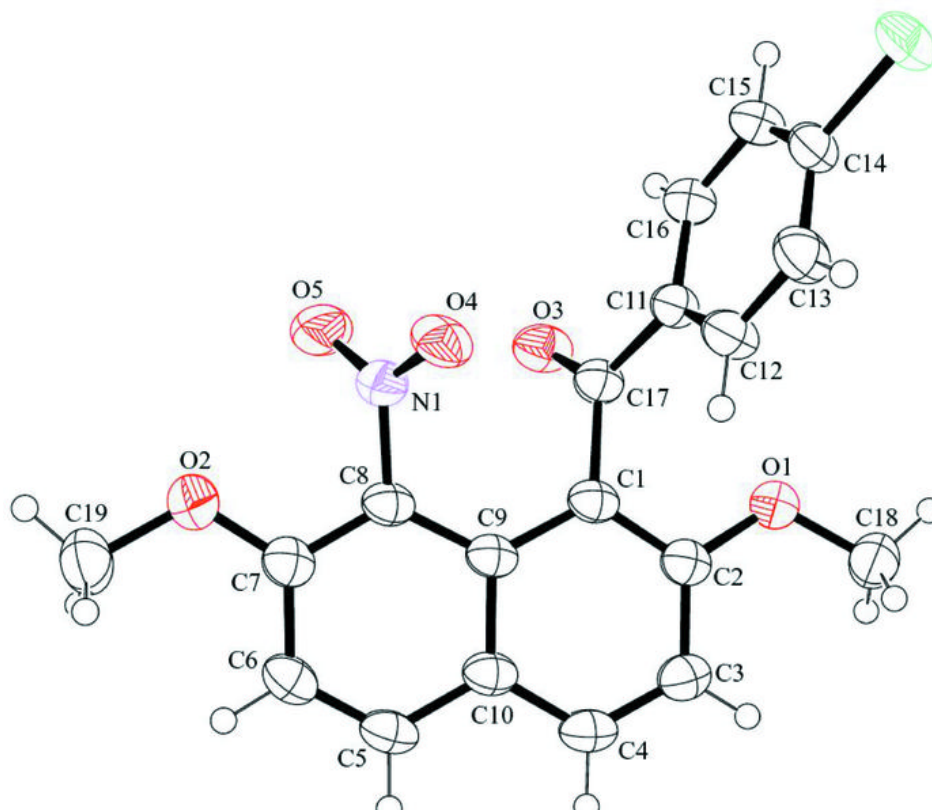


Fig. 2

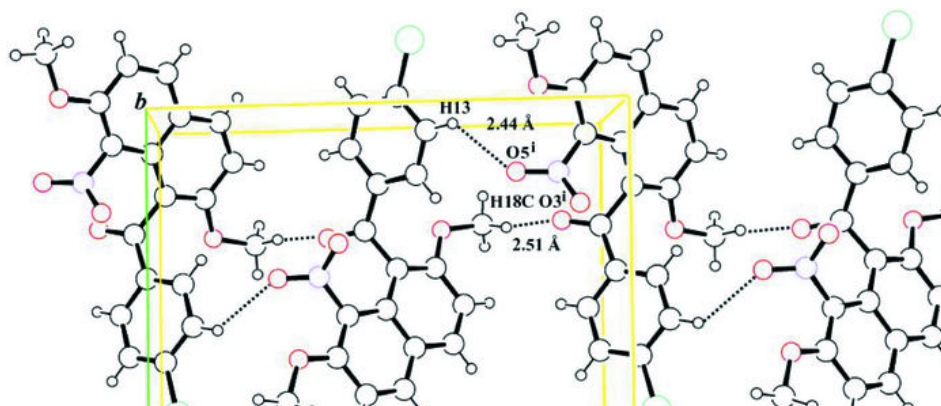


Fig. 3

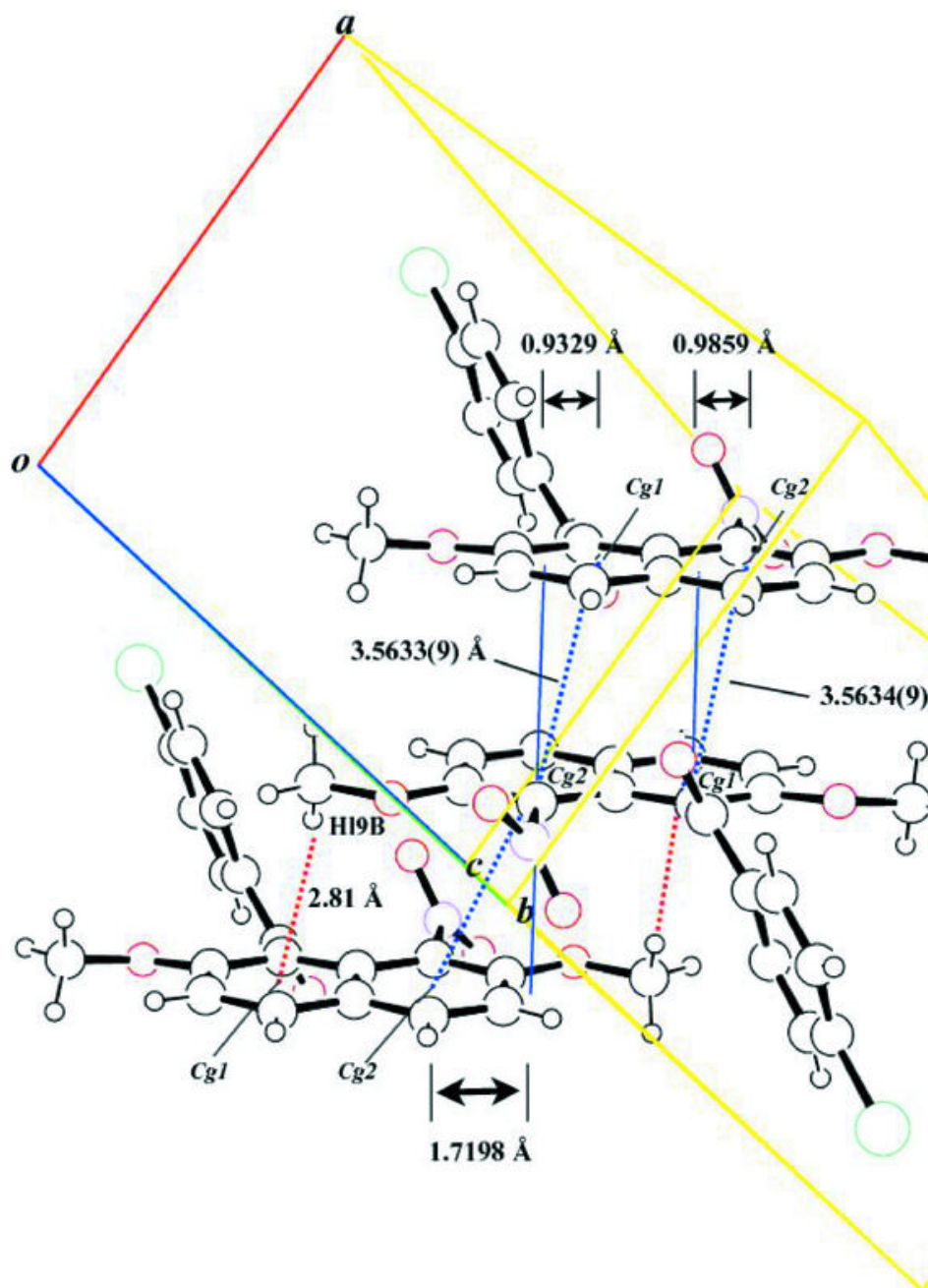


Fig. 4

