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2-(2,7-Dimethoxy-1-naphthoyl)benzoic acid

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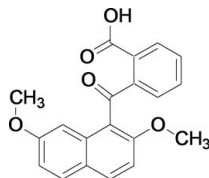
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 Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.032; wR factor = 0.095; data-to-parameter ratio = 13.0.

In the title compound, $\text{C}_{20}\text{H}_{16}\text{O}_5$, the dihedral angle between the naphthalene ring system and the benzene ring is 67.43 (5)°. The bridging carbonyl $\text{C}-\text{C}(=\text{O})-\text{C}$ plane makes dihedral angles of 82.64 (6) and 41.79 (7)°, respectively, with the naphthalene ring system and the benzene ring. The dihedral angle between the carboxy $\text{O}-\text{C}(=\text{O})-\text{C}$ plane and the benzene ring is 36.38 (7)° and that between the bridging carbonyl $\text{C}-\text{C}(=\text{O})-\text{C}$ plane and the carboxy $\text{O}-\text{C}(=\text{O})-\text{C}$ plane is 51.88 (8)°. The crystal structure is stabilized by intermolecular $\text{O}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen-bonding interactions. An intramolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bond occurs between a naphthalene H atom and the carbonyl O atom of the carboxy group.

Related literature

For electrophilic aromatic substitution of naphthalene derivatives, see: Okamoto & Yonezawa (2009). For related structures, see: Mitsui, Nakaema *et al.* (2008); Mitsui, Noguchi *et al.* (2009); Watanabe, Nakaema, Muto *et al.* (2010); Watanabe, Nakaema, Nishijima *et al.* (2010); Hijikata *et al.* (2010).



Experimental

Crystal data

$\text{C}_{20}\text{H}_{16}\text{O}_5$
 $M_r = 336.33$
 Monoclinic, $P2_1/c$
 $a = 10.8311$ (2) Å
 $b = 10.61451$ (19) Å

$c = 15.4492$ (3) Å
 $\beta = 111.728$ (1)°
 $V = 1649.95$ (5) Å³
 $Z = 4$
 Cu $K\alpha$ radiation

$\mu = 0.81$ mm⁻¹
 $T = 296$ K

$0.60 \times 0.60 \times 0.30$ mm

Data collection

Rigaku R-AXIS- APID
 diffractometer
 Absorption correction: multi-scan
 (NUMABS; Higashi, 1999)
 $T_{\min} = 0.711$, $T_{\max} = 0.785$

29727 measured reflections
 3023 independent reflections
 2710 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.028$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.032$
 $wR(F^2) = 0.095$
 $S = 1.04$
 3023 reflections
 233 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.16$ e Å⁻³
 $\Delta\rho_{\min} = -0.12$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O3}-\text{H1}\cdots\text{O1}^{\text{i}}$	0.91 (2)	1.83 (2)	2.7320 (15)	173.4 (19)
$\text{C7}-\text{H7}\cdots\text{O3}^{\text{ii}}$	0.93	2.49	3.3215 (19)	150
$\text{C15}-\text{H15}\cdots\text{O2}^{\text{iii}}$	0.93	2.48	3.3152 (16)	149
$\text{C17}-\text{H17}\cdots\text{O5}^{\text{iv}}$	0.93	2.55	3.2821 (18)	136
$\text{C9}-\text{H9}\cdots\text{O2}$	0.93	2.47	3.3850 (16)	169

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

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 III* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: OM2323).

References

- 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). *ORTEP III*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
- Higashi, T. (1999). *NUMABS*. Rigaku Corporation, Tokyo, Japan.
- Hijikata, D., Nakaema, K., Watanabe, S., Okamoto, A. & Yonezawa, N. (2010). *Acta Cryst.* **E66**, o554.
- Mitsui, R., Nakaema, K., Noguchi, K., Okamoto, A. & Yonezawa, N. (2008). *Acta Cryst.* **E64**, o1278.
- Mitsui, R., Noguchi, K. & Yonezawa, N. (2009). *Acta Cryst.* **E65**, o543.
- 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., Muto, T., Okamoto, A. & Yonezawa, N. (2010). *Acta Cryst.* **E66**, o403.
- Watanabe, S., Nakaema, K., Nishijima, T., Okamoto, A. & Yonezawa, N. (2010). *Acta Cryst.* **E66**, o615.

supporting information

Acta Cryst. (2010). E66, o713 [doi:10.1107/S1600536810006847]

2-(2,7-Dimethoxy-1-naphthoyl)benzoic acid

Daichi Hijikata, Kosuke Nakaema, Shoji Watanabe, 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 the aid of suitable acidic mediators (Okamoto & Yonezawa, 2009). Recently, we reported the crystal structures of several 1,8-diaroylated naphthalene homologues exemplified by bis(4-bromobenzoyl)(2,7-dimethoxynaphthalene-1,8-diyl)dimethanone (Watanabe, Nakaema, Muto *et al.*, 2010). The aryl groups at the 1,8-positions of the naphthalene rings in these compounds are twisted almost perpendicularly but the benzene ring moieties of the aryl groups tilt slightly toward the *exo* sides of the naphthalene rings. Moreover, the X-ray crystal structural analysis of 1-(4-substituted benzoyl)naphthalenes, i.e., 1-(4-chlorobenzoyl)-2,7-dimethoxynaphthalene (Mitsui *et al.*, 2008), (4-chlorobenzoyl)(2-ethoxy-7-methoxynaphthalen-1-yl)methanone (Mitsui *et al.*, 2009), 1-(4-nitrobenzoyl)-2,7-dimethoxynaphthalene (Watanabe, Nakaema, Nishijima *et al.*, 2010) and methyl 4-(2,7-dimethoxy-1-naphthoyl)benzoate (Hijikata *et al.*, 2010), has also revealed essentially the same non-coplanar structure as the 1,8-diaroylated naphthalenes.

As a part of our continuing study on the molecular structures of these homologous molecules, the crystal structure of title compound, 1-monoarylnaphthalene bearing carboxy group, is discussed in this report.

In the molecule of the title compound, shown in Fig. 1, the dihedral angle between the naphthalene ring (C1—C10) and the benzene ring (C12—C17) is 67.43 (5)°. The bridging carbonyl C1—C11(=O1)—C12 plane makes dihedral angles of 82.64 (6)° [C2—C1—C11—O1 torsion angle = -77.60 (16)°] and 41.79 (7)° [O1—C11—C12—C13 torsion angle = -37.07 (17)°], respectively, with the naphthalene ring system and the benzene ring. The dihedral angle between the carbonyl O3—C18(=O2)—C13 plane and the benzene ring is 36.38 (7)° [C12—C13—C18—O2 torsion angle = -35.33 (19)°]. The dihedral angle between the bridging carbonyl C1—C11(=O1)—C12 plane and the carboxy O3—C18(=O2)—C13 plane is 51.88 (8)°. The torsion angle between one methoxy group and the naphthalene ring plane is relatively large [C29—O4—C2—C3 = -15.3 (2)°] and that between the other methoxy group and the naphthalene ring plane is rather small [C20—O5—C8—C7 = 3.8 (2)°]. The crystal structure is stabilized by intermolecular O3—H1ⁱ [symmetry code: (i) -x+1, y-1/2, -z+1/2] and C—Hⁱ⋯O hydrogen-bonding interactions with one intramolecular C9—H9ⁱ⋯O2 hydrogen bonding (Table 1, Fig 1, 2).

S2. Experimental

The title compound was prepared by AlCl₃-mediated regioselective electrophilic aromatic arylation of 2,7-dimethoxynaphthalene with acetic anhydride. Single crystals suitable for X-ray diffraction were obtained by recrystallization from diethyl ether.

Spectroscopic Data: ¹H NMR (300 MHz, CDCl₃) δ 3.65 (3H, s), 3.78 (3H, s), 7.00 (1H, dd, *J* = 2.4, 9.0 Hz), 7.43-7.58 (4H, m), 7.66 (1H, d, *J* = 9.0 Hz), 7.84-7.88 (2H, m); ¹³C NMR (75.0 MHz, CDCl₃) δ 55.3, 56.5, 102.7, 110.5, 117.4, 124.4, 129.4, 129.5, 129.9, 130.9, 131.4, 133.8, 142.1, 157.1, 159.6, 198.0; IR (KBr): 1698, 1627, 1512; HRMS (*m/z*):

$[M+H]^+$ Calcd for $C_{20}H_{17}O_5$, 337.1076; Found, 337.1057.

S3. Refinement

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

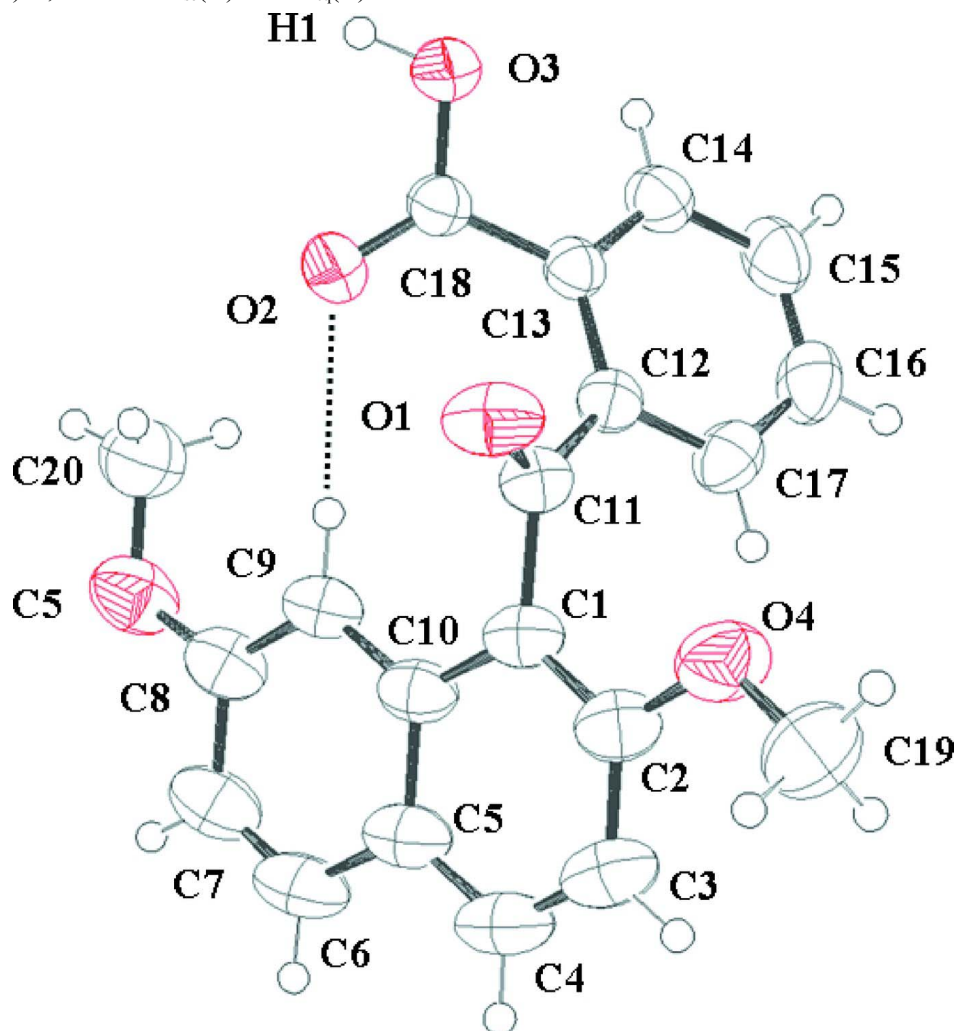


Figure 1

The molecular structure, showing the atom-labeling scheme and 50% probability displacement ellipsoids. The intramolecular C—H \cdots O interaction is shown as a dashed line.

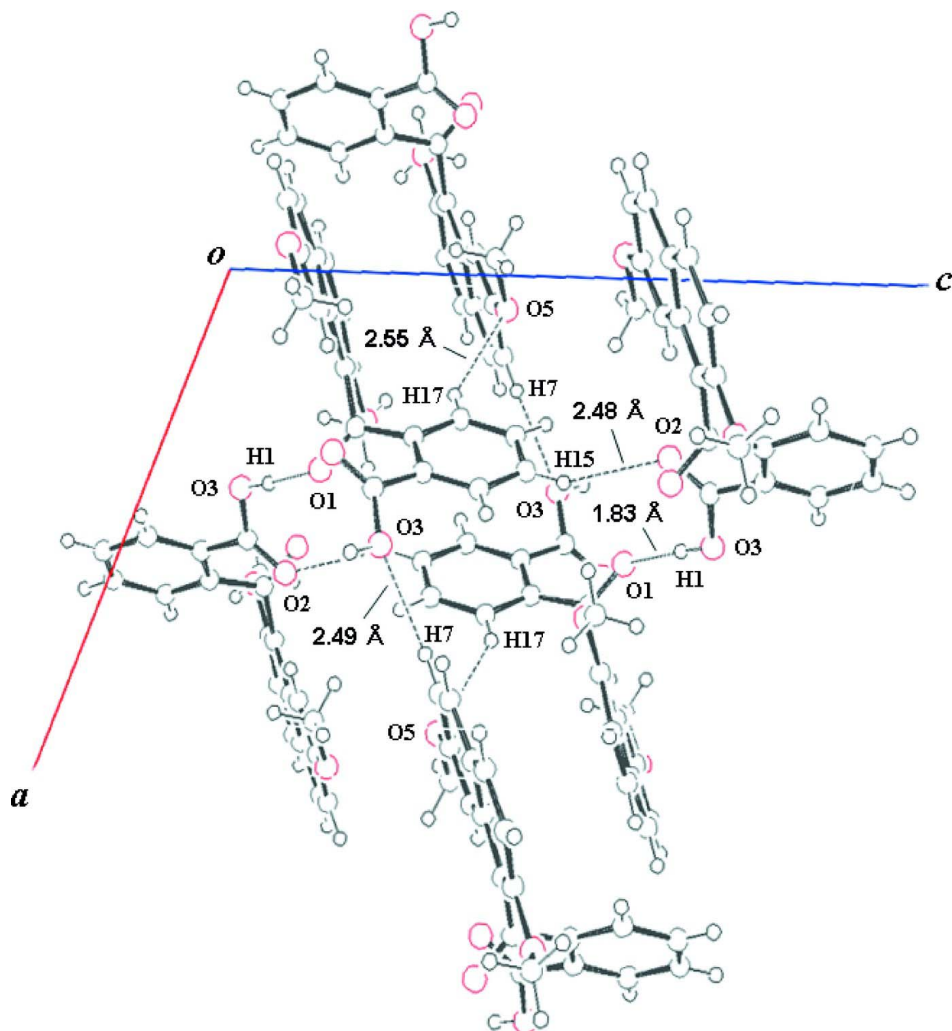


Figure 2

A partial crystal packing diagram viewed down *b*. The intermolecular O—H...O and C—H...O interactions are shown as dashed lines.

2-(2,7-Dimethoxy-1-naphthoyl)benzoic acid

Crystal data

$C_{20}H_{16}O_5$

$M_r = 336.33$

Monoclinic, $P2_1/c$

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

$a = 10.8311(2)\ \text{\AA}$

$b = 10.61451(19)\ \text{\AA}$

$c = 15.4492(3)\ \text{\AA}$

$\beta = 111.728(1)^\circ$

$V = 1649.95(5)\ \text{\AA}^3$

$Z = 4$

$F(000) = 704$

$D_x = 1.354\ \text{Mg m}^{-3}$

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

Cell parameters from 26822 reflections

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

$\mu = 0.81\ \text{mm}^{-1}$

$T = 296\ \text{K}$

Block, colorless

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

*Data collection*Rigaku R-AXIS- APID
diffractometer

Radiation source: rotating anode

Graphite monochromator

Detector resolution: 10.00 pixels mm⁻¹ ω scansAbsorption correction: multi-scan
(NUMABS; Higashi, 1999) $T_{\min} = 0.711$, $T_{\max} = 0.785$

29727 measured reflections

3023 independent reflections

2710 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.028$ $\theta_{\max} = 68.2^\circ$, $\theta_{\min} = 4.4^\circ$ $h = -13 \rightarrow 13$ $k = -12 \rightarrow 12$ $l = -18 \rightarrow 18$ *Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.032$ $wR(F^2) = 0.095$ $S = 1.04$

3023 reflections

233 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
map

Hydrogen site location: difference Fourier map

H atoms treated by a mixture of independent
and constrained refinement $w = 1/[\sigma^2(F_o^2) + (0.0481P)^2 + 0.2862P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.16 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.12 \text{ e } \text{\AA}^{-3}$ Extinction correction: *SHELXL97* (Sheldrick,
2008), $F_c^* = kFc[1 + 0.001x\text{Fc}^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0021 (3)

Special details

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.40529 (9)	0.47494 (10)	0.24166 (7)	0.0630 (3)
O2	0.36166 (9)	0.19907 (10)	0.24736 (6)	0.0647 (3)
O3	0.54599 (9)	0.15634 (11)	0.36707 (7)	0.0709 (3)
H1	0.5589 (19)	0.100 (2)	0.3272 (14)	0.104 (6)*
O4	0.30544 (10)	0.73160 (9)	0.28563 (8)	0.0726 (3)
O5	-0.05580 (10)	0.17144 (10)	0.06622 (8)	0.0725 (3)
C1	0.18889 (11)	0.54939 (12)	0.21895 (8)	0.0487 (3)
C2	0.18877 (13)	0.67861 (13)	0.22803 (10)	0.0580 (3)
C3	0.07232 (16)	0.74830 (15)	0.18184 (12)	0.0706 (4)
H3	0.0716	0.8349	0.1906	0.085*
C4	-0.03897 (15)	0.68890 (15)	0.12452 (11)	0.0707 (4)
H4	-0.1149	0.7362	0.0937	0.085*
C5	-0.04262 (13)	0.55796 (14)	0.11051 (9)	0.0590 (3)
C6	-0.15566 (13)	0.49476 (17)	0.04818 (11)	0.0711 (4)

H6	-0.2311	0.5412	0.0148	0.085*
C7	-0.15665 (14)	0.36879 (17)	0.03601 (11)	0.0710 (4)
H7	-0.2324	0.3292	-0.0048	0.085*
C8	-0.04226 (12)	0.29720 (14)	0.08549 (9)	0.0576 (3)
C9	0.06985 (11)	0.35343 (12)	0.14630 (9)	0.0505 (3)
H9	0.1442	0.3050	0.1787	0.061*
C10	0.07285 (11)	0.48552 (12)	0.16004 (8)	0.0495 (3)
C11	0.31457 (11)	0.47747 (11)	0.27012 (8)	0.0447 (3)
C12	0.33101 (10)	0.41688 (11)	0.36061 (7)	0.0409 (3)
C13	0.40131 (10)	0.30406 (11)	0.39141 (7)	0.0402 (3)
C14	0.43259 (12)	0.26655 (13)	0.48298 (8)	0.0511 (3)
H14	0.4795	0.1921	0.5037	0.061*
C15	0.39508 (14)	0.33816 (15)	0.54409 (9)	0.0605 (4)
H15	0.4171	0.3121	0.6055	0.073*
C16	0.32540 (13)	0.44760 (14)	0.51365 (9)	0.0607 (4)
H16	0.3004	0.4960	0.5546	0.073*
C17	0.29207 (12)	0.48650 (12)	0.42223 (9)	0.0523 (3)
H17	0.2431	0.5600	0.4019	0.063*
C18	0.43300 (11)	0.21726 (11)	0.32640 (7)	0.0424 (3)
C19	0.32145 (18)	0.86387 (15)	0.28282 (13)	0.0792 (5)
H19A	0.4114	0.8861	0.3201	0.095*
H19B	0.2620	0.9052	0.3069	0.095*
H19C	0.3017	0.8900	0.2196	0.095*
C20	0.05113 (17)	0.09279 (16)	0.11674 (13)	0.0798 (5)
H20A	0.0300	0.0072	0.0966	0.096*
H20B	0.0671	0.0990	0.1820	0.096*
H20C	0.1293	0.1185	0.1061	0.096*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0532 (5)	0.0731 (6)	0.0744 (6)	0.0134 (4)	0.0372 (5)	0.0261 (5)
O2	0.0577 (5)	0.0842 (7)	0.0423 (5)	0.0184 (5)	0.0068 (4)	-0.0127 (4)
O3	0.0549 (5)	0.0910 (8)	0.0539 (5)	0.0303 (5)	0.0051 (4)	-0.0173 (5)
O4	0.0693 (6)	0.0470 (5)	0.0925 (8)	-0.0002 (5)	0.0194 (6)	0.0074 (5)
O5	0.0589 (6)	0.0676 (6)	0.0757 (7)	-0.0136 (5)	0.0073 (5)	0.0077 (5)
C1	0.0445 (6)	0.0497 (7)	0.0544 (7)	0.0074 (5)	0.0212 (5)	0.0128 (5)
C2	0.0566 (8)	0.0524 (7)	0.0659 (8)	0.0054 (6)	0.0238 (6)	0.0107 (6)
C3	0.0750 (10)	0.0527 (8)	0.0863 (10)	0.0201 (7)	0.0326 (8)	0.0185 (7)
C4	0.0591 (8)	0.0723 (10)	0.0796 (10)	0.0264 (7)	0.0245 (8)	0.0246 (8)
C5	0.0472 (7)	0.0696 (9)	0.0616 (8)	0.0142 (6)	0.0216 (6)	0.0198 (6)
C6	0.0434 (7)	0.0907 (12)	0.0713 (9)	0.0153 (7)	0.0121 (6)	0.0229 (8)
C7	0.0430 (7)	0.0918 (12)	0.0670 (9)	-0.0025 (7)	0.0073 (6)	0.0146 (8)
C8	0.0459 (7)	0.0673 (8)	0.0574 (7)	-0.0049 (6)	0.0165 (6)	0.0115 (6)
C9	0.0398 (6)	0.0567 (7)	0.0531 (7)	0.0037 (5)	0.0151 (5)	0.0141 (5)
C10	0.0411 (6)	0.0580 (7)	0.0518 (6)	0.0067 (5)	0.0200 (5)	0.0138 (5)
C11	0.0402 (6)	0.0435 (6)	0.0520 (6)	-0.0009 (5)	0.0190 (5)	0.0036 (5)
C12	0.0340 (5)	0.0447 (6)	0.0449 (6)	-0.0026 (4)	0.0154 (4)	-0.0013 (5)

C13	0.0345 (5)	0.0464 (6)	0.0388 (5)	-0.0007 (4)	0.0125 (4)	-0.0013 (4)
C14	0.0502 (7)	0.0599 (7)	0.0410 (6)	0.0052 (6)	0.0143 (5)	0.0036 (5)
C15	0.0610 (8)	0.0820 (10)	0.0397 (6)	0.0000 (7)	0.0200 (6)	-0.0030 (6)
C16	0.0622 (8)	0.0724 (9)	0.0560 (7)	-0.0029 (7)	0.0319 (6)	-0.0168 (6)
C17	0.0493 (7)	0.0511 (7)	0.0623 (7)	0.0009 (5)	0.0274 (6)	-0.0056 (6)
C18	0.0383 (5)	0.0476 (6)	0.0398 (6)	0.0030 (5)	0.0128 (5)	0.0003 (5)
C19	0.0928 (12)	0.0515 (8)	0.0992 (12)	-0.0041 (8)	0.0423 (10)	0.0056 (8)
C20	0.0753 (10)	0.0634 (9)	0.0870 (11)	-0.0022 (8)	0.0142 (8)	-0.0008 (8)

Geometric parameters (Å, °)

O1—C11	1.2163 (13)	C8—C9	1.3670 (18)
O2—C18	1.1945 (13)	C9—C10	1.4166 (18)
O3—C18	1.3195 (14)	C9—H9	0.9300
O3—H1	0.91 (2)	C11—C12	1.4885 (16)
O4—C2	1.3684 (17)	C12—C17	1.3884 (16)
O4—C19	1.4173 (18)	C12—C13	1.4042 (16)
O5—C8	1.3637 (18)	C13—C14	1.3856 (16)
O5—C20	1.4065 (19)	C13—C18	1.4935 (15)
C1—C2	1.3788 (19)	C14—C15	1.3849 (18)
C1—C10	1.4215 (17)	C14—H14	0.9300
C1—C11	1.5046 (15)	C15—C16	1.370 (2)
C2—C3	1.4074 (19)	C15—H15	0.9300
C3—C4	1.358 (2)	C16—C17	1.3850 (19)
C3—H3	0.9300	C16—H16	0.9300
C4—C5	1.405 (2)	C17—H17	0.9300
C4—H4	0.9300	C19—H19A	0.9600
C5—C6	1.415 (2)	C19—H19B	0.9600
C5—C10	1.4256 (16)	C19—H19C	0.9600
C6—C7	1.350 (2)	C20—H20A	0.9600
C6—H6	0.9300	C20—H20B	0.9600
C7—C8	1.4126 (19)	C20—H20C	0.9600
C7—H7	0.9300		
C18—O3—H1	110.5 (12)	C12—C11—C1	118.89 (9)
C2—O4—C19	118.76 (12)	C17—C12—C13	119.07 (10)
C8—O5—C20	117.39 (11)	C17—C12—C11	116.93 (11)
C2—C1—C10	120.37 (11)	C13—C12—C11	123.23 (10)
C2—C1—C11	119.08 (11)	C14—C13—C12	119.21 (10)
C10—C1—C11	120.53 (11)	C14—C13—C18	118.63 (10)
O4—C2—C1	116.08 (12)	C12—C13—C18	121.94 (9)
O4—C2—C3	123.52 (13)	C15—C14—C13	121.04 (12)
C1—C2—C3	120.37 (14)	C15—C14—H14	119.5
C4—C3—C2	119.94 (14)	C13—C14—H14	119.5
C4—C3—H3	120.0	C16—C15—C14	119.65 (12)
C2—C3—H3	120.0	C16—C15—H15	120.2
C3—C4—C5	121.82 (13)	C14—C15—H15	120.2
C3—C4—H4	119.1	C15—C16—C17	120.35 (12)

C5—C4—H4	119.1	C15—C16—H16	119.8
C4—C5—C6	122.79 (13)	C17—C16—H16	119.8
C4—C5—C10	118.80 (13)	C16—C17—C12	120.65 (12)
C6—C5—C10	118.40 (13)	C16—C17—H17	119.7
C7—C6—C5	121.64 (13)	C12—C17—H17	119.7
C7—C6—H6	119.2	O2—C18—O3	122.85 (11)
C5—C6—H6	119.2	O2—C18—C13	124.36 (10)
C6—C7—C8	119.83 (14)	O3—C18—C13	112.69 (9)
C6—C7—H7	120.1	O4—C19—H19A	109.5
C8—C7—H7	120.1	O4—C19—H19B	109.5
O5—C8—C9	125.01 (12)	H19A—C19—H19B	109.5
O5—C8—C7	113.99 (13)	O4—C19—H19C	109.5
C9—C8—C7	121.00 (14)	H19A—C19—H19C	109.5
C8—C9—C10	120.03 (11)	H19B—C19—H19C	109.5
C8—C9—H9	120.0	O5—C20—H20A	109.5
C10—C9—H9	120.0	O5—C20—H20B	109.5
C9—C10—C1	122.26 (10)	H20A—C20—H20B	109.5
C9—C10—C5	119.11 (12)	O5—C20—H20C	109.5
C1—C10—C5	118.59 (12)	H20A—C20—H20C	109.5
O1—C11—C12	119.88 (10)	H20B—C20—H20C	109.5
O1—C11—C1	121.07 (10)		
C19—O4—C2—C1	166.64 (13)	C6—C5—C10—C9	-0.91 (18)
C19—O4—C2—C3	-15.3 (2)	C4—C5—C10—C1	-2.41 (18)
C10—C1—C2—O4	-179.44 (11)	C6—C5—C10—C1	176.71 (12)
C11—C1—C2—O4	-1.13 (18)	C2—C1—C11—O1	-77.60 (16)
C10—C1—C2—C3	2.4 (2)	C10—C1—C11—O1	100.70 (14)
C11—C1—C2—C3	-179.30 (12)	C2—C1—C11—C12	97.80 (14)
O4—C2—C3—C4	178.89 (14)	C10—C1—C11—C12	-83.90 (14)
C1—C2—C3—C4	-3.1 (2)	O1—C11—C12—C17	132.79 (12)
C2—C3—C4—C5	1.0 (2)	C1—C11—C12—C17	-42.67 (15)
C3—C4—C5—C6	-177.30 (14)	O1—C11—C12—C13	-37.07 (17)
C3—C4—C5—C10	1.8 (2)	C1—C11—C12—C13	147.48 (11)
C4—C5—C6—C7	-179.97 (15)	C17—C12—C13—C14	-1.43 (16)
C10—C5—C6—C7	0.9 (2)	C11—C12—C13—C14	168.22 (11)
C5—C6—C7—C8	-0.7 (2)	C17—C12—C13—C18	173.01 (10)
C20—O5—C8—C9	3.8 (2)	C11—C12—C13—C18	-17.34 (16)
C20—O5—C8—C7	-176.41 (14)	C12—C13—C14—C15	0.31 (18)
C6—C7—C8—O5	-179.36 (14)	C18—C13—C14—C15	-174.32 (11)
C6—C7—C8—C9	0.4 (2)	C13—C14—C15—C16	0.3 (2)
O5—C8—C9—C10	179.34 (12)	C14—C15—C16—C17	0.2 (2)
C7—C8—C9—C10	-0.4 (2)	C15—C16—C17—C12	-1.4 (2)
C8—C9—C10—C1	-176.87 (11)	C13—C12—C17—C16	1.96 (17)
C8—C9—C10—C5	0.67 (18)	C11—C12—C17—C16	-168.33 (11)
C2—C1—C10—C9	177.90 (12)	C14—C13—C18—O2	139.14 (13)
C11—C1—C10—C9	-0.38 (17)	C12—C13—C18—O2	-35.33 (18)
C2—C1—C10—C5	0.35 (17)	C14—C13—C18—O3	-37.23 (15)
C11—C1—C10—C5	-177.93 (11)	C12—C13—C18—O3	148.30 (11)

C4—C5—C10—C9

179.96 (12)

Hydrogen-bond geometry (Å, °)

<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>
O3—H1···O1 ⁱ	0.91 (2)	1.83 (2)	2.7320 (15)	173.4 (19)
C7—H7···O3 ⁱⁱ	0.93	2.49	3.3215 (19)	150
C15—H15···O2 ⁱⁱⁱ	0.93	2.48	3.3152 (16)	149
C17—H17···O5 ^{iv}	0.93	2.55	3.2821 (18)	136
C9—H9···O2	0.93	2.47	3.3850 (16)	169

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