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Methyl (*E*)-2-({2-[(*E*)-(hydroxyimino)-methyl]phenoxy}methyl)-3-(4-methylphenyl)acrylate

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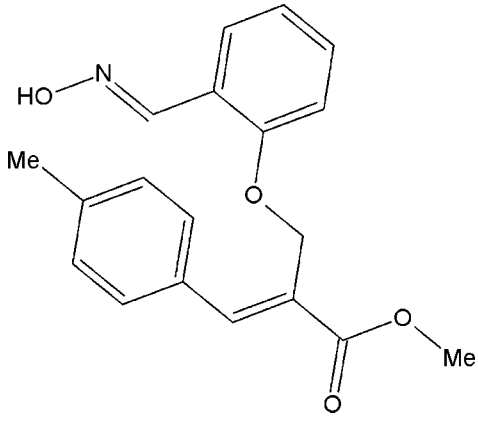
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.045; wR factor = 0.132; data-to-parameter ratio = 20.7.

In the title compound, $C_{19}H_{19}NO_4$, the dihedral angle between the mean planes through the benzene rings is 82.18 (7)°. The C=N double bond is *trans*-configured. The molecules are linked into centrosymmetric dimers *via* pairs of O—H···N hydrogen bonds with the motif $R_2^2(6)$. The crystal packing also features C—H···O interactions. The methyl group attached to one of the aromatic rings is disordered over two almost equally occupied positions [occpancy ratio = 0.51 (4):0.49 (4)].

Related literature

For information on oximes, see: Chaudhuri (2003). For a related structure, see: SakthiMurugesan *et al.* (2011).



Experimental

Crystal data

$C_{19}H_{19}NO_4$
M_r = 325.35
Triclinic, $P\overline{1}$
a = 8.8683 (2) Å
b = 9.3246 (2) Å
c = 11.9259 (3) Å
α = 75.200 (2)°
β = 76.453 (2)°
γ = 65.142 (1)°
V = 856.04 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 K
0.35 × 0.30 × 0.25 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.970, T_{max} = 0.978
20292 measured reflections
4580 independent reflections
3493 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.132
S = 1.04
4580 reflections
221 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···N1^{i}	0.82	2.11	2.8211 (15)	145
C15—H15···O3^{ii}	0.93	2.40	3.247 (2)	151

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97* and *PLATON*.

The authors thank Dr Babu Varghese, SAIF, IIT, Chennai, India for the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5884).

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supplementary materials

Acta Cryst. (2012). E**68**, o1617 [doi:10.1107/S1600536812019046]

Methyl (*E*)-2-({2-[(*E*)-(hydroxyimino)methyl]phenoxy}methyl)-3-(4-methylphenyl)acrylate

G. Ganesh, J. Srinivasan, E. Govindan, M. Bakthadoss and A. SubbiahPandi

Comment

Oximes are a classical type of chelating ligands which are widely used in coordination and analytical chemistry (Chaudhuri, 2003). Against this background, and in order to obtain detailed information on molecular conformations in the solid state, an X-ray study of the title compound was carried out.

X-Ray analysis confirms the molecular structure and atom connectivity as illustrated in Fig. 1. The bond lengths and angles in (Fig. 1) agree with those observed in other acrylate derivatives (SakthiMurugesan *et al.*, 2011). the whole molecule is not planar as the dihedral angle between the two phenyl rings is 82.18 (7)°, it shows that both the rings are almost perpendicular to each other. The methoxybutene group connects the two phenyl rings, results in twisting the rings and placed those rings in perpendicular direction. The oxime group having the C═N forming an E configuration. The atom C19 is deviated by -0.037 (2) Å from the least squares plane of the C13—C18 ring. The hydroxyethanimine group is essentially coplanar with the benzene ring, the largest deviation from the mean plane being -0.008 (1) Å for the C2 atom.

The enoate group assumes an extended conformation as can be seen from torsion angles C8—C9—C10—O3 [169.97 (15)°] and C9—C10—O4—C11 [179.55 (14)°]. The hydroxyethanimine group in the molecules are linked into cyclic centrosymmetric dimers *via* O—H···N hydrogen bonds with the motif $R_2^2(6)$. In addition to van der Waals interactions the crystal packing is stabilized by C–H..O and O–H···N interactions.

Experimental

To a stirred solution of (*E*)-methyl 2-((2-formylphenoxy)methyl)-3 - *p*-tolyacrylate (4 mmol) in 10 ml of EtOH/H_2O mixture(1:1) was added $NH_2OH.HCl$(6 mmol) in the presence of 50% NaOH at room temperature. Then the reaction mixture was allowed to stir at room temperature For 1.5 h. After completion of the reaction, solvent was removed and crude mass was diluted with water (15 ml) and extracted with ethyl acetate (3x15ml). The combined organic layer was washed with brine (2x10ml) and dried over anhydrous Na_2SO_4 and then evaporated under reduced pressure to obtain(*E*)-methyl 2-((2-((*E*)-(hydroxyimino) methyl)phenoxy) methyl)-3-*p*-tolylacrylate as a colourless solid. Single crystals suitable for X-ray diffraction were obtained by slow evaporation of a solution of the title compound in acetone at room temperature.

Refinement

All H atoms were fixed geometrically and allowed to ride on their parent atoms, with C—H distances in the range 0.93–0.97 Å and an O—H distance of 0.82 Å and with U_{iso}(H) = 1.5U_{eq}(C_{methyl}, O) or 1.2U_{eq}(C) for other H atoms.

Computing details

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

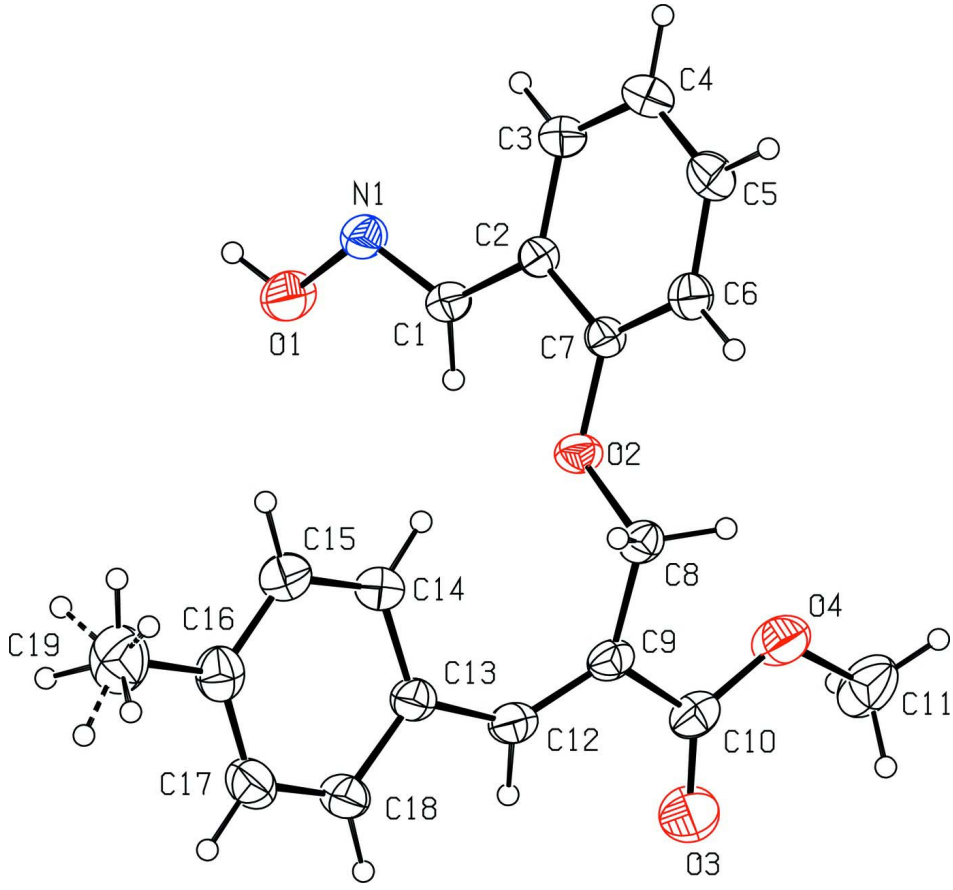

Figure 1

View of the title molecule with the atom labelling scheme. The displacement ellipsoids are drawn at the 30% probability level while the H atoms are shown as small spheres of arbitrary radii.

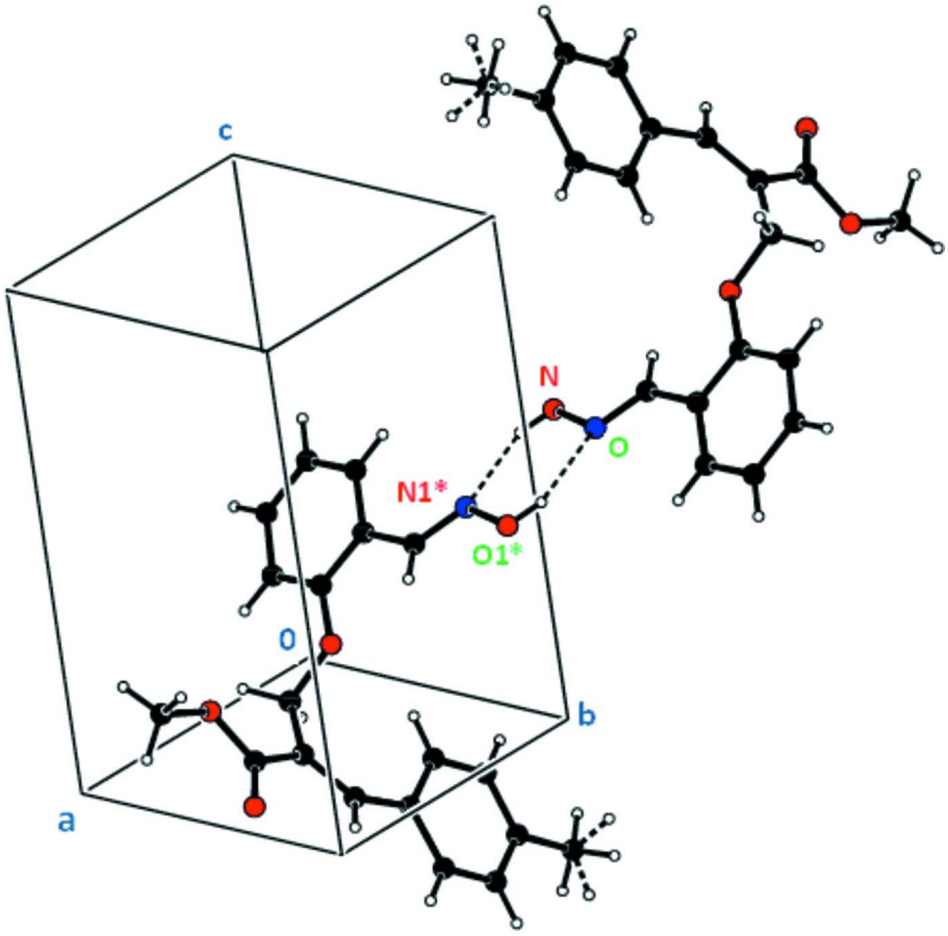

Figure 2

The crystal structure showing the centrosymmetric hydrogen bond motif $R_2^2(6)$. For the sake of clarity, the H atoms not involved in the motif have been omitted. The atoms marked with an asterisk (*) are at the symmetry position (2 - *x*, -*y*, 1 - *z*). The dashed lines indicate the hydrogen bonds.

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Crystal data

$C_{19}H_{19}NO_4$
M_r = 325.35
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Hall symbol: -P 1
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c = 11.9259 (3) Å
α = 75.200 (2)°
β = 76.453 (2)°
γ = 65.142 (1)°
V = 856.04 (3) Å^3
Z = 2
F(000) = 344
D_x = 1.262 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6056 reflections
θ = 2.5–32.5°
μ = 0.09 mm^{-1}
T = 293 K
Block, white crystalline
0.35 × 0.30 × 0.25 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.970, T_{max} = 0.978
20292 measured reflections
4580 independent reflections
3493 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 29.2°, θ_{min} = 2.5°
h = −12→12
k = −12→12
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.132
S = 1.03
4580 reflections
221 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0596P)^2 + 0.1563P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.30137 (15)	0.78709 (15)	0.40342 (11)	0.0446 (3)	
H1	0.3839	0.8244	0.3607	0.053*	
C2	0.32860 (14)	0.61892 (14)	0.41323 (10)	0.0397 (3)	
C3	0.24555 (17)	0.54149 (16)	0.50493 (12)	0.0521 (3)	
H3	0.1701	0.5977	0.5623	0.063*	
C4	0.2729 (2)	0.38274 (18)	0.51246 (13)	0.0608 (4)	
H4	0.2161	0.3325	0.5743	0.073*	
C5	0.38495 (19)	0.29887 (16)	0.42785 (13)	0.0569 (3)	
H5	0.4027	0.1920	0.4326	0.068*	
C6	0.47131 (16)	0.37155 (15)	0.33607 (12)	0.0476 (3)	
H6	0.5470	0.3139	0.2795	0.057*	
C7	0.44431 (14)	0.53102 (13)	0.32886 (10)	0.0385 (2)	
C8	0.64247 (16)	0.53394 (14)	0.15207 (11)	0.0450 (3)	
H8A	0.5849	0.5135	0.1017	0.054*	
H8B	0.7225	0.4320	0.1859	0.054*	
C9	0.73103 (15)	0.64103 (15)	0.08311 (11)	0.0441 (3)	
C10	0.89857 (17)	0.60977 (17)	0.11066 (13)	0.0532 (3)	

C11	1.0978 (2)	0.4600 (3)	0.24022 (17)	0.0852 (6)	
H11A	1.1801	0.4565	0.1713	0.128*	
H11B	1.1307	0.3571	0.2908	0.128*	
H11C	1.0899	0.5402	0.2807	0.128*	
C12	0.67354 (16)	0.76491 (15)	−0.00294 (11)	0.0475 (3)	
H12	0.7450	0.8193	−0.0359	0.057*	
C13	0.51907 (16)	0.82972 (14)	−0.05413 (11)	0.0456 (3)	
C14	0.37187 (17)	0.80613 (17)	−0.00162 (12)	0.0546 (3)	
H14	0.3663	0.7467	0.0737	0.065*	
C15	0.23412 (19)	0.86949 (18)	−0.05950 (14)	0.0594 (4)	
H15	0.1378	0.8506	−0.0228	0.071*	
C16	0.23571 (19)	0.96063 (17)	−0.17098 (13)	0.0578 (4)	
C17	0.3798 (2)	0.98842 (18)	−0.22200 (13)	0.0619 (4)	
H17	0.3833	1.0512	−0.2962	0.074*	
C18	0.51779 (19)	0.92530 (16)	−0.16541 (12)	0.0558 (3)	
H18	0.6129	0.9466	−0.2020	0.067*	
C19	0.0852 (3)	1.0257 (3)	−0.23393 (18)	0.0884 (6)	
H19A	0.0030	0.9840	−0.1886	0.133*	0.51 (4)
H19B	0.1192	0.9937	−0.3093	0.133*	0.51 (4)
H19C	0.0376	1.1408	−0.2440	0.133*	0.51 (4)
H19D	0.0618	0.9382	−0.2433	0.133*	0.49 (4)
H19F	0.1082	1.0858	−0.3096	0.133*	0.49 (4)
H19E	−0.0102	1.0945	−0.1890	0.133*	0.49 (4)
N1	0.16707 (14)	0.88269 (13)	0.45217 (10)	0.0493 (3)	
O1	0.16721 (14)	1.03758 (12)	0.43411 (11)	0.0699 (3)	
H1A	0.0766	1.0977	0.4635	0.105*	
O2	0.52349 (11)	0.61418 (10)	0.24323 (7)	0.0467 (2)	
O3	0.99150 (15)	0.67203 (16)	0.05342 (13)	0.0872 (4)	
O4	0.93678 (13)	0.49931 (16)	0.20693 (9)	0.0720 (3)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0417 (6)	0.0459 (6)	0.0413 (6)	−0.0165 (5)	0.0028 (5)	−0.0081 (5)
C2	0.0363 (5)	0.0416 (6)	0.0369 (5)	−0.0131 (4)	−0.0052 (4)	−0.0034 (4)
C3	0.0498 (7)	0.0526 (7)	0.0433 (6)	−0.0177 (6)	0.0034 (5)	−0.0034 (5)
C4	0.0628 (9)	0.0551 (8)	0.0556 (8)	−0.0281 (7)	0.0009 (7)	0.0061 (6)
C5	0.0620 (8)	0.0428 (6)	0.0639 (8)	−0.0235 (6)	−0.0082 (7)	−0.0009 (6)
C6	0.0480 (7)	0.0427 (6)	0.0502 (7)	−0.0160 (5)	−0.0055 (5)	−0.0095 (5)
C7	0.0366 (5)	0.0407 (6)	0.0362 (5)	−0.0144 (4)	−0.0063 (4)	−0.0032 (4)
C8	0.0453 (6)	0.0428 (6)	0.0431 (6)	−0.0146 (5)	0.0033 (5)	−0.0141 (5)
C9	0.0415 (6)	0.0467 (6)	0.0424 (6)	−0.0162 (5)	0.0055 (5)	−0.0172 (5)
C10	0.0449 (7)	0.0574 (7)	0.0560 (8)	−0.0177 (6)	0.0030 (6)	−0.0205 (6)
C11	0.0524 (9)	0.1224 (16)	0.0710 (11)	−0.0173 (10)	−0.0144 (8)	−0.0242 (11)
C12	0.0462 (7)	0.0483 (6)	0.0483 (7)	−0.0221 (5)	0.0060 (5)	−0.0136 (5)
C13	0.0499 (7)	0.0404 (6)	0.0446 (6)	−0.0181 (5)	0.0019 (5)	−0.0110 (5)
C14	0.0515 (7)	0.0568 (7)	0.0484 (7)	−0.0215 (6)	−0.0001 (6)	−0.0027 (6)
C15	0.0504 (8)	0.0629 (8)	0.0635 (9)	−0.0243 (7)	−0.0030 (6)	−0.0091 (7)
C16	0.0623 (9)	0.0521 (7)	0.0576 (8)	−0.0157 (6)	−0.0123 (7)	−0.0148 (6)
C17	0.0745 (10)	0.0535 (8)	0.0487 (7)	−0.0211 (7)	−0.0075 (7)	−0.0017 (6)

C18	0.0600 (8)	0.0490 (7)	0.0534 (8)	−0.0246 (6)	0.0010 (6)	−0.0029 (6)
C19	0.0806 (13)	0.1011 (15)	0.0808 (12)	−0.0241 (11)	−0.0337 (10)	−0.0105 (11)
N1	0.0485 (6)	0.0423 (5)	0.0524 (6)	−0.0168 (4)	0.0037 (5)	−0.0118 (5)
O1	0.0685 (7)	0.0460 (5)	0.0879 (8)	−0.0241 (5)	0.0180 (6)	−0.0223 (5)
O2	0.0513 (5)	0.0418 (4)	0.0421 (4)	−0.0193 (4)	0.0095 (4)	−0.0118 (3)
O3	0.0586 (7)	0.0883 (9)	0.1159 (11)	−0.0418 (6)	−0.0146 (7)	0.0059 (8)
O4	0.0507 (6)	0.1054 (9)	0.0517 (6)	−0.0264 (6)	−0.0068 (5)	−0.0074 (6)

Geometric parameters (Å, º)

C1—N1	1.2644 (16)	C11—H11B	0.9600
C1—C2	1.4602 (17)	C11—H11C	0.9600
C1—H1	0.9300	C12—C13	1.4566 (19)
C2—C3	1.3879 (17)	C12—H12	0.9300
C2—C7	1.4027 (16)	C13—C14	1.3902 (18)
C3—C4	1.378 (2)	C13—C18	1.3964 (18)
C3—H3	0.9300	C14—C15	1.378 (2)
C4—C5	1.377 (2)	C14—H14	0.9300
C4—H4	0.9300	C15—C16	1.383 (2)
C5—C6	1.3815 (19)	C15—H15	0.9300
C5—H5	0.9300	C16—C17	1.380 (2)
C6—C7	1.3860 (17)	C16—C19	1.506 (2)
C6—H6	0.9300	C17—C18	1.370 (2)
C7—O2	1.3618 (14)	C17—H17	0.9300
C8—O2	1.4386 (14)	C18—H18	0.9300
C8—C9	1.4925 (17)	C19—H19A	0.9600
C8—H8A	0.9700	C19—H19B	0.9600
C8—H8B	0.9700	C19—H19C	0.9600
C9—C12	1.3386 (18)	C19—H19D	0.9600
C9—C10	1.4886 (19)	C19—H19F	0.9600
C10—O3	1.1910 (17)	C19—H19E	0.9600
C10—O4	1.3336 (18)	N1—O1	1.4052 (14)
C11—O4	1.443 (2)	O1—H1A	0.8200
C11—H11A	0.9600		
N1—C1—C2	120.89 (11)	H11A—C11—H11C	109.5
N1—C1—H1	119.6	H11B—C11—H11C	109.5
C2—C1—H1	119.6	C9—C12—C13	131.50 (12)
C3—C2—C7	118.50 (11)	C9—C12—H12	114.2
C3—C2—C1	122.09 (11)	C13—C12—H12	114.2
C7—C2—C1	119.40 (10)	C14—C13—C18	116.93 (13)
C4—C3—C2	121.16 (13)	C14—C13—C12	125.81 (12)
C4—C3—H3	119.4	C18—C13—C12	117.26 (12)
C2—C3—H3	119.4	C15—C14—C13	121.00 (13)
C5—C4—C3	119.61 (13)	C15—C14—H14	119.5
C5—C4—H4	120.2	C13—C14—H14	119.5
C3—C4—H4	120.2	C14—C15—C16	121.55 (14)
C4—C5—C6	120.81 (13)	C14—C15—H15	119.2
C4—C5—H5	119.6	C16—C15—H15	119.2
C6—C5—H5	119.6	C17—C16—C15	117.63 (14)

C5—C6—C7	119.55 (12)	C17—C16—C19	121.61 (15)
C5—C6—H6	120.2	C15—C16—C19	120.76 (16)
C7—C6—H6	120.2	C18—C17—C16	121.27 (14)
O2—C7—C6	124.60 (11)	C18—C17—H17	119.4
O2—C7—C2	115.05 (10)	C16—C17—H17	119.4
C6—C7—C2	120.35 (11)	C17—C18—C13	121.57 (14)
O2—C8—C9	107.57 (9)	C17—C18—H18	119.2
O2—C8—H8A	110.2	C13—C18—H18	119.2
C9—C8—H8A	110.2	C16—C19—H19A	109.5
O2—C8—H8B	110.2	C16—C19—H19B	109.5
C9—C8—H8B	110.2	C16—C19—H19C	109.5
H8A—C8—H8B	108.5	C16—C19—H19D	109.5
C12—C9—C10	115.91 (12)	C16—C19—H19F	109.5
C12—C9—C8	125.82 (12)	H19D—C19—H19F	109.5
C10—C9—C8	118.27 (11)	C16—C19—H19E	109.5
O3—C10—O4	122.75 (14)	H19D—C19—H19E	109.5
O3—C10—C9	125.04 (14)	H19F—C19—H19E	109.5
O4—C10—C9	112.19 (12)	C1—N1—O1	111.94 (11)
O4—C11—H11A	109.5	N1—O1—H1A	109.5
O4—C11—H11B	109.5	C7—O2—C8	118.77 (9)
H11A—C11—H11B	109.5	C10—O4—C11	116.22 (14)
O4—C11—H11C	109.5		

N1—C1—C2—C3	−23.53 (19)	C8—C9—C12—C13	−0.6 (2)
N1—C1—C2—C7	157.62 (12)	C9—C12—C13—C14	20.6 (2)
C7—C2—C3—C4	−1.2 (2)	C9—C12—C13—C18	−159.99 (14)
C1—C2—C3—C4	179.93 (13)	C18—C13—C14—C15	2.5 (2)
C2—C3—C4—C5	0.2 (2)	C12—C13—C14—C15	−178.12 (13)
C3—C4—C5—C6	0.6 (2)	C13—C14—C15—C16	−0.9 (2)
C4—C5—C6—C7	−0.3 (2)	C14—C15—C16—C17	−1.0 (2)
C5—C6—C7—O2	179.27 (12)	C14—C15—C16—C19	178.59 (15)
C5—C6—C7—C2	−0.80 (19)	C15—C16—C17—C18	1.3 (2)
C3—C2—C7—O2	−178.54 (11)	C19—C16—C17—C18	−178.27 (15)
C1—C2—C7—O2	0.35 (16)	C16—C17—C18—C13	0.3 (2)
C3—C2—C7—C6	1.53 (17)	C14—C13—C18—C17	−2.2 (2)
C1—C2—C7—C6	−179.59 (11)	C12—C13—C18—C17	178.37 (13)
O2—C8—C9—C12	−82.19 (15)	C2—C1—N1—O1	178.81 (11)
O2—C8—C9—C10	97.89 (12)	C6—C7—O2—C8	0.84 (17)
C12—C9—C10—O3	−10.0 (2)	C2—C7—O2—C8	−179.09 (10)
C8—C9—C10—O3	169.98 (14)	C9—C8—O2—C7	−170.37 (10)
C12—C9—C10—O4	171.55 (12)	O3—C10—O4—C11	1.0 (2)
C8—C9—C10—O4	−8.52 (16)	C9—C10—O4—C11	179.55 (13)
C10—C9—C12—C13	179.32 (12)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···N1^{i}	0.82	2.11	2.8211 (15)	145

C15—H15···O3^{ii}	0.93	2.40	3.247 (2)	151

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