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(1′*S*,4′*S*)-5-(2,5-Dimethylphenyl)-4′-methoxy-6-oxa-3-azaspiro[bicyclo-[3.1.0]hexane-2,1′-cyclohexan]-4-one

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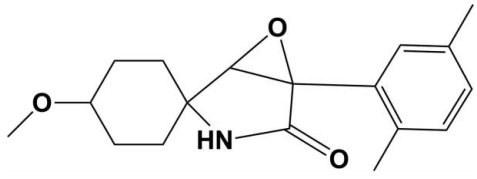
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.045; wR factor = 0.122; data-to-parameter ratio = 17.9.

In the title compound, $C_{18}H_{23}NO_3$, the cyclohexane ring has a chair conformation. The oxirane plane (OCC) makes a dihedral angle of 76.15 (13)° with that of the pyrrolidine ring to which it is fused. The mean plane of the cyclohexane ring and the benzene ring are almost normal to the pyrrolidine ring, with dihedral angles of 88.47 (8) and 77.85 (8)°, respectively. In the crystal, molecules are linked *via* pairs of N—H···O hydrogen bonds, forming inversion dimers. These dimers are linked *via* pairs of C—H···O hydrogen bonds, forming chains along the *a*-axis direction.

Related literature

For the pesticide spirotetramat, the central unit of the title compound, see: Fischer & Weiss (2008); Maus (2008). For structures of spirotetramat derivatives, see: Fischer *et al.* (2010). For the metabolic transformation of spirotetramat, see: Bruck *et al.* (2009)



Experimental

Crystal data

$C_{18}H_{23}NO_3$ M_r = 301.37
Monoclinic, $P2_1/n$
a = 9.1932 (4) Å
b = 9.8139 (4) Å
c = 17.6979 (7) Å
β = 91.198 (1)°
V = 1596.38 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 296 K
0.53 × 0.38 × 0.36 mm

Data collection

Rigaku R-AXIS RAPID/ZJUG diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.946, T_{max} = 0.970
15333 measured reflections
3629 independent reflections
2407 reflections with $I > 2\sigma(I)$
R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.122
S = 1.00
3629 reflections
203 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3^{i}	0.86	2.25	3.0760 (17)	160
C9—H9A···O2^{ii}	0.97	2.56	3.413 (2)	147

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

Data collection: *PROCESS-AUTO* (Rigaku, 2006); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku,2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2559).

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supporting information

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(1′*S*,4′*S*)-5-(2,5-Dimethylphenyl)-4′-methoxy-6-oxa-3-azaspiro[bicyclo-[3.1.0]hexane-2,1′-cyclohexan]-4-one

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S1. Comment

Spirotetramat is a new systemic insecticide which belongs chemically to the class of spirocyclic tetramic acid derivatives and be developed by Bayer CropScience AG (Fischer *et al.*, 2008; Maus, 2008). Recently, it has been found that when spirotetramat was introduced into plants or animals, it was hydrolysed to its enol form and as a weak acid this metabolite can move acropetally and basipetally with small log *P* (Bruck *et al.*, 2009). That is, the excellent bioactivities of spirotetramat maybe caused by this metabolite, which stimulated our interest in the synthesis of some novel analogues of this metabolite. We have designed a new simple route to synthesized the title compound and report herein on its crystal structure.

The molecular structure of the title molecule is shown in Fig. 1. The cyclohexane ring adopts a chair conformation; atoms C5, C6, C8 and C9 lie in a plane with atoms C4 and C7 deviating by 0.676 (4) and -0.664 (0) Å, respectively. The oxirane plane (O2/C2/C3) makes a dihedral angle of 76.15 (13)° with the pyrrolidine ring (N1/C1-C4) to which it is fused. The mean plane of the cyclohexane ring (C4-C9) and the benzene ring (C11-C16) are almost normal to the pyrrolidine ring with dihedral angles of 88.47 (8) and 77.85 (8)°, respectively.

In the crystal, molecules are linked via a pair of N-H···O hydrogen bonds forming inversion dimers (Table 1). These dimers are linked via a pair of C-H···O hydrogen bonds forming chains along the a axis direction (Table 1).

S2. Experimental

The synthesis of the title compound is described in Fig. 2. A solution of sulfuryl chloride (0.80 g, 6mmoL) in anhydrous chloroform (10 ml) was added drop wise to a solution of compound 2 (0.90 g, 3mmoL) in anhydrous chloroform (20 ml) at 0 degree and stirred for 10 min. The reaction mixture was allowed to warm to room temperature and stirred at room temperature for 1 h. The reaction mixture was then washed with water (15 ml), saturated sodium bicarbonate (15 ml) and saturated sodium chloride solution and dried over anhydrous Na_2SO_4. The solvent was evaporated, and the residual solid was crystallized from ethanol to afford 0.95 g compound 3 as a white solid: yield 94.8%. To a solution of compound 3 (100 mg, 0.30 mmoL) in 2-propanol (10 ml) was added $NaBH_4$ (13.6 mg, 0.36 mmoL) at 0 degree. Then the reaction mixture was allowed to room temperature and stirred for 2 h. After removal of the solvent *in vacuo*, 1 N HCl (10 ml) was added to the residue and the whole mixture was extracted with CH_2Cl_2 (8 ml *τ*imes 3). The organic layer was washed successively with 3% Na_2CO_3 (8 ml) and water (8 ml) and dried over Na_2SO_4. Evaporation of the solvent gave a residue, which was purified by flash chromatography on silica gel using a mixture of petroleum ether (boiling point range 60–90 degree) and ethyl acetate (1:1 by volume) as the eluent to afford 32 mg compound 4 as a white solid [yield 35.6%; ESI-MS: 336 (*M*+H)+ (100%)]. Spectroscopic data for the title compound is available in the archived CIF.

S3. Refinement

The H atoms were included in calculated positions (C–H = 0.93–0.98 Å) and refined as riding, with U_{iso}(H) = 1.2U_{eq}(C).

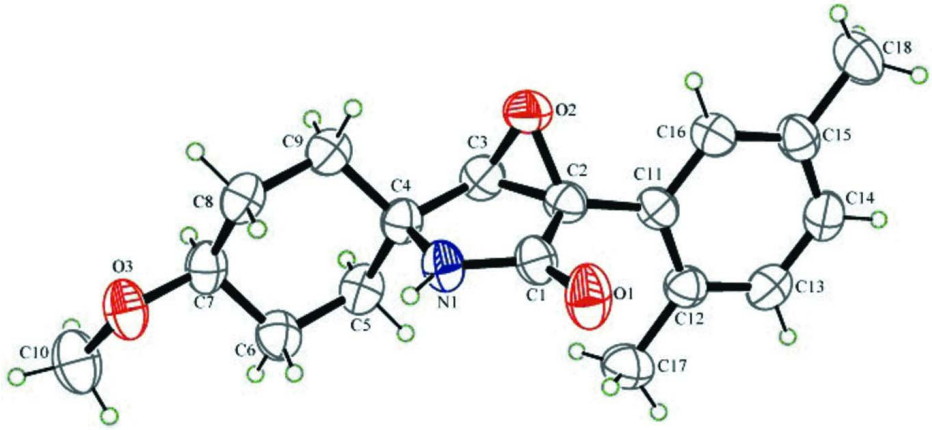

Figure 1

The molecular structure of the title molecule, with atom labelling. Displacement ellipsoids are drawn at the 40% probability level.

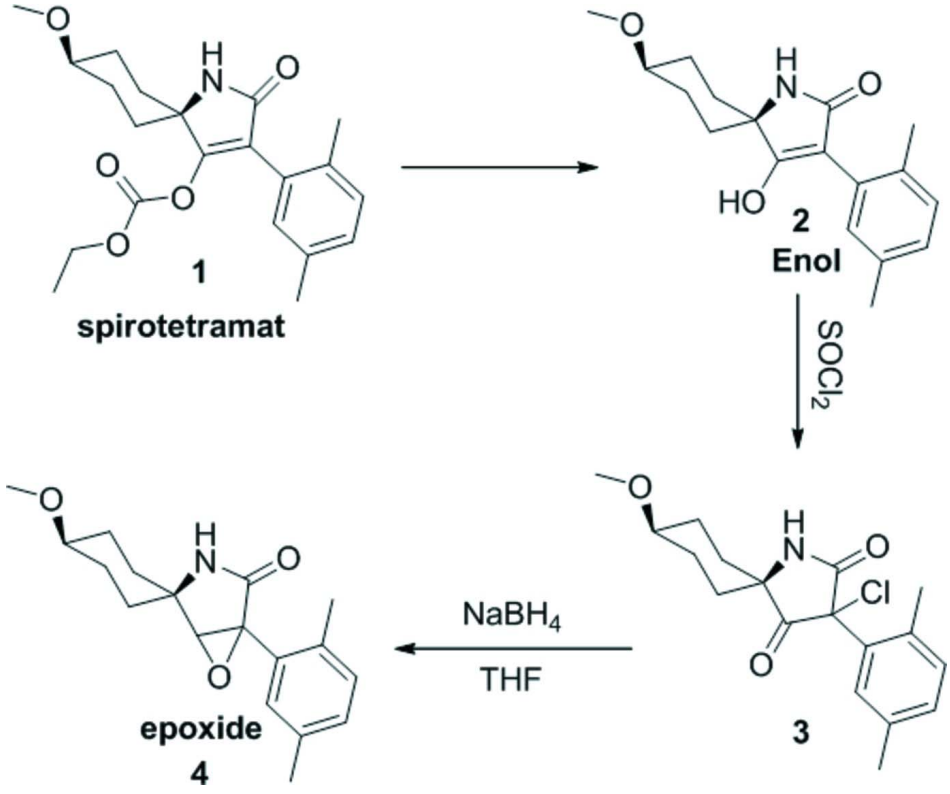

Figure 2

Reaction scheme.

(1′*S*,4′*S*)-5-(2,5-Dimethylphenyl)-4′-methoxy-6-oxa-3-azaspiro[bicyclo[3.1.0]hexane-2,1′-cyclohexan]-4-one

Crystal data

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b = 9.8139 (4) Å
c = 17.6979 (7) Å
β = 91.198 (1)°
V = 1596.38 (11) Å^3
Z = 4
$F(000)$ = 648
D_x = 1.254 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9900 reflections
θ = 3.0–27.4°
μ = 0.09 mm^{-1}
T = 296 K
Block, colourless
0.53 × 0.38 × 0.36 mm

Data collection

Rigaku R-AXIS RAPID/ZJUG diffractometer
Radiation source: rotating anode
Graphite monochromator
Detector resolution: 10.00 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.946, T_{max} = 0.970
15333 measured reflections
3629 independent reflections
2407 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 27.5°, θ_{min} = 3.0°
h = −11→11
k = −12→12
l = −22→22

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.122
S = 1.00
3629 reflections
203 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
$w = 1/[\sigma^2(F_o^2) + (0.0501P)^2 + 0.4587P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.049 (3)

Special details

Experimental. Spectroscopic data for the title compound: ^{1}H NMR (500 MHz, $CDCl_3$): 7.28 (s, 1H, Ph—H), 7.21 (s, 1H, Ph—H), 7.11 (s, 1H, Ph—H), 5.57 (s, 1H, –NH–), 3.80 (d, J = 2.65 Hz, 1H, –CH—O—C–), 3.40–3.39 (m, 1H, –CH—O–), 3.37 (s, 3H, –OCH3), 2.35 (s, 3H, Ph—Me), 2.33 (s, 3H, Ph—Me), 2.04–2.00 (m, 1H, Cyclohexane-H1), 1.94–1.82 (m, 4H, Cyclohexane-H4), 1.75–1.62 (m, 3H, Cyclohexane-H3); ^{13}C NMR (125 MHz, $CDCl_3$): 171.4, 135.4, 133.7, 130.1, 129.8, 128.8, 128.5, 75.3, 64.9, 62.8, 57.0, 55.7, 31.7, 28.2, 27.1, 26.4, 20.8, 19.3.

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

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}
O1	0.60742 (14)	0.85489 (13)	0.31242 (7)	0.0625 (4)
O2	0.42952 (12)	0.79911 (13)	0.47491 (7)	0.0565 (4)
O3	1.09024 (13)	0.91600 (12)	0.60804 (7)	0.0563 (4)
N1	0.71200 (14)	0.84251 (13)	0.43139 (7)	0.0448 (4)
C1	0.60987 (17)	0.81496 (16)	0.37776 (9)	0.0436 (5)
C2	0.49630 (16)	0.72456 (16)	0.41369 (9)	0.0423 (5)
C3	0.54729 (17)	0.70679 (17)	0.49249 (9)	0.0468 (5)
C4	0.69248 (17)	0.77653 (16)	0.50507 (8)	0.0418 (5)
C5	0.81229 (19)	0.67175 (16)	0.52153 (10)	0.0494 (5)
C6	0.96044 (18)	0.73875 (18)	0.53488 (10)	0.0520 (6)
C7	0.95550 (18)	0.84352 (17)	0.59826 (9)	0.0482 (5)
C8	0.83812 (18)	0.94787 (17)	0.58211 (9)	0.0491 (5)
C9	0.68976 (18)	0.88081 (18)	0.56940 (9)	0.0499 (5)
C10	1.2063 (2)	0.8335 (2)	0.63718 (12)	0.0688 (7)
C11	0.39938 (16)	0.63409 (15)	0.36728 (9)	0.0406 (5)
C12	0.45709 (17)	0.52733 (16)	0.32516 (9)	0.0444 (5)
C13	0.3604 (2)	0.44859 (18)	0.28230 (9)	0.0524 (6)
C14	0.21297 (19)	0.47469 (18)	0.27957 (9)	0.0515 (5)
C15	0.15504 (17)	0.58089 (18)	0.32083 (9)	0.0479 (5)
C16	0.25058 (17)	0.65911 (16)	0.36478 (9)	0.0455 (5)
C17	0.61810 (19)	0.4977 (2)	0.32605 (11)	0.0622 (7)
C18	−0.00622 (19)	0.6083 (2)	0.31909 (12)	0.0702 (8)
H1	0.78430	0.89560	0.42300	0.0540*
H3	0.52710	0.62120	0.51880	0.0560*
H5A	0.78730	0.61940	0.56590	0.0590*
H5B	0.81810	0.60930	0.47920	0.0590*
H6A	1.03190	0.66930	0.54780	0.0620*
H6B	0.99050	0.78300	0.48870	0.0620*
H7	0.93360	0.79660	0.64560	0.0580*
H8A	0.86290	0.99970	0.53750	0.0590*
H8B	0.83310	1.01070	0.62430	0.0590*
H9A	0.61800	0.95040	0.55750	0.0600*
H9B	0.66110	0.83580	0.61550	0.0600*
H10A	1.17290	0.78150	0.67940	0.1030*
H10B	1.28570	0.89070	0.65330	0.1030*
H10C	1.23840	0.77260	0.59840	0.1030*
H13	0.39640	0.37600	0.25460	0.0630*
H14	0.15190	0.42050	0.24970	0.0620*
H16	0.21370	0.73030	0.39330	0.0550*
H17A	0.63690	0.42140	0.29380	0.0930*
H17B	0.65020	0.47680	0.37670	0.0930*
H17C	0.66960	0.57610	0.30820	0.0930*
H18A	−0.04210	0.60580	0.26780	0.1050*
H18B	−0.02450	0.69660	0.34030	0.1050*
H18C	−0.05480	0.54010	0.34810	0.1050*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0669 (8)	0.0706 (8)	0.0493 (7)	−0.0175 (6)	−0.0146 (6)	0.0190 (6)
O2	0.0454 (7)	0.0587 (7)	0.0656 (8)	0.0023 (5)	0.0056 (6)	−0.0174 (6)
O3	0.0560 (7)	0.0530 (7)	0.0590 (7)	−0.0094 (6)	−0.0168 (6)	0.0124 (6)
N1	0.0435 (7)	0.0462 (7)	0.0445 (7)	−0.0098 (6)	−0.0046 (6)	0.0091 (6)
C1	0.0428 (8)	0.0416 (8)	0.0460 (8)	−0.0011 (7)	−0.0061 (7)	0.0053 (7)
C2	0.0392 (8)	0.0400 (8)	0.0477 (8)	0.0023 (6)	0.0018 (7)	−0.0008 (7)
C3	0.0500 (9)	0.0447 (8)	0.0457 (9)	−0.0055 (7)	0.0038 (7)	0.0000 (7)
C4	0.0460 (9)	0.0418 (8)	0.0376 (8)	−0.0044 (7)	−0.0015 (6)	0.0031 (6)
C5	0.0621 (11)	0.0392 (8)	0.0467 (9)	0.0000 (7)	−0.0062 (8)	0.0012 (7)
C6	0.0525 (10)	0.0466 (9)	0.0565 (10)	0.0056 (8)	−0.0084 (8)	−0.0006 (8)
C7	0.0546 (10)	0.0473 (9)	0.0422 (8)	−0.0091 (8)	−0.0077 (7)	0.0068 (7)
C8	0.0597 (10)	0.0428 (8)	0.0447 (8)	−0.0027 (8)	−0.0033 (7)	−0.0049 (7)
C9	0.0516 (10)	0.0507 (9)	0.0473 (9)	−0.0008 (8)	0.0013 (7)	−0.0048 (7)
C10	0.0639 (12)	0.0686 (12)	0.0728 (13)	−0.0008 (10)	−0.0226 (10)	0.0132 (10)
C11	0.0376 (8)	0.0394 (8)	0.0447 (8)	−0.0024 (6)	0.0008 (6)	0.0038 (7)
C12	0.0436 (9)	0.0453 (9)	0.0443 (8)	0.0033 (7)	0.0020 (7)	0.0015 (7)
C13	0.0612 (11)	0.0516 (9)	0.0444 (9)	0.0018 (8)	0.0022 (8)	−0.0059 (8)
C14	0.0548 (10)	0.0572 (10)	0.0421 (8)	−0.0102 (8)	−0.0050 (7)	0.0001 (8)
C15	0.0407 (8)	0.0556 (10)	0.0474 (9)	−0.0055 (7)	−0.0013 (7)	0.0081 (8)
C16	0.0419 (9)	0.0428 (8)	0.0519 (9)	0.0001 (7)	0.0028 (7)	0.0002 (7)
C17	0.0528 (11)	0.0624 (11)	0.0715 (12)	0.0123 (9)	0.0046 (9)	−0.0057 (10)
C18	0.0423 (10)	0.0888 (15)	0.0793 (14)	−0.0067 (10)	−0.0036 (9)	−0.0021 (12)

Geometric parameters (Å, º)

O1—C1	1.221 (2)	C15—C16	1.392 (2)
O2—C2	1.454 (2)	C15—C18	1.506 (2)
O2—C3	1.441 (2)	C3—H3	0.9800
O3—C7	1.436 (2)	C5—H5A	0.9700
O3—C10	1.427 (2)	C5—H5B	0.9700
N1—C1	1.348 (2)	C6—H6A	0.9700
N1—C4	1.4703 (19)	C6—H6B	0.9700
N1—H1	0.8600	C7—H7	0.9800
C1—C2	1.520 (2)	C8—H8A	0.9700
C2—C3	1.472 (2)	C8—H8B	0.9700
C2—C11	1.492 (2)	C9—H9A	0.9700
C3—C4	1.512 (2)	C9—H9B	0.9700
C4—C9	1.532 (2)	C10—H10A	0.9600
C4—C5	1.530 (2)	C10—H10B	0.9600
C5—C6	1.526 (2)	C10—H10C	0.9600
C6—C7	1.523 (2)	C13—H13	0.9300
C7—C8	1.511 (2)	C14—H14	0.9300
C8—C9	1.527 (2)	C16—H16	0.9300
C11—C12	1.397 (2)	C17—H17A	0.9600
C11—C16	1.390 (2)	C17—H17B	0.9600

C12—C17	1.508 (2)	C17—H17C	0.9600
C12—C13	1.391 (2)	C18—H18A	0.9600
C13—C14	1.379 (3)	C18—H18B	0.9600
C14—C15	1.385 (2)	C18—H18C	0.9600

C2—O2—C3	61.14 (10)	C4—C5—H5B	109.00
C7—O3—C10	113.52 (13)	C6—C5—H5A	109.00
C1—N1—C4	116.10 (13)	C6—C5—H5B	109.00
C4—N1—H1	122.00	H5A—C5—H5B	108.00
C1—N1—H1	122.00	C5—C6—H6A	109.00
O1—C1—C2	125.77 (15)	C5—C6—H6B	109.00
N1—C1—C2	107.27 (13)	C7—C6—H6A	109.00
O1—C1—N1	126.96 (15)	C7—C6—H6B	109.00
O2—C2—C11	116.94 (12)	H6A—C6—H6B	108.00
O2—C2—C1	108.82 (12)	O3—C7—H7	109.00
O2—C2—C3	58.99 (10)	C6—C7—H7	109.00
C3—C2—C11	128.73 (14)	C8—C7—H7	109.00
C1—C2—C11	121.65 (14)	C7—C8—H8A	109.00
C1—C2—C3	104.93 (13)	C7—C8—H8B	109.00
O2—C3—C4	113.84 (13)	C9—C8—H8A	109.00
C2—C3—C4	110.39 (13)	C9—C8—H8B	109.00
O2—C3—C2	59.87 (10)	H8A—C8—H8B	108.00
N1—C4—C9	111.67 (13)	C4—C9—H9A	109.00
N1—C4—C3	101.10 (12)	C4—C9—H9B	109.00
N1—C4—C5	111.35 (13)	C8—C9—H9A	109.00
C5—C4—C9	109.29 (13)	C8—C9—H9B	109.00
C3—C4—C5	110.70 (13)	H9A—C9—H9B	108.00
C3—C4—C9	112.57 (13)	O3—C10—H10A	109.00
C4—C5—C6	112.11 (13)	O3—C10—H10B	110.00
C5—C6—C7	111.38 (14)	O3—C10—H10C	109.00
O3—C7—C8	107.34 (13)	H10A—C10—H10B	109.00
O3—C7—C6	112.60 (13)	H10A—C10—H10C	109.00
C6—C7—C8	110.52 (13)	H10B—C10—H10C	109.00
C7—C8—C9	111.60 (14)	C12—C13—H13	119.00
C4—C9—C8	111.66 (13)	C14—C13—H13	119.00
C2—C11—C16	119.24 (14)	C13—C14—H14	120.00
C2—C11—C12	120.73 (13)	C15—C14—H14	120.00
C12—C11—C16	120.01 (14)	C11—C16—H16	119.00
C11—C12—C17	121.51 (14)	C15—C16—H16	119.00
C11—C12—C13	117.53 (15)	C12—C17—H17A	109.00
C13—C12—C17	120.96 (15)	C12—C17—H17B	109.00
C12—C13—C14	122.16 (16)	C12—C17—H17C	109.00
C13—C14—C15	120.64 (16)	H17A—C17—H17B	109.00
C14—C15—C16	117.67 (15)	H17A—C17—H17C	109.00
C14—C15—C18	120.83 (15)	H17B—C17—H17C	110.00
C16—C15—C18	121.48 (15)	C15—C18—H18A	109.00
C11—C16—C15	121.98 (15)	C15—C18—H18B	109.00
O2—C3—H3	120.00	C15—C18—H18C	109.00

C2—C3—H3	119.00	H18A—C18—H18B	109.00
C4—C3—H3	120.00	H18A—C18—H18C	109.00
C4—C5—H5A	109.00	H18B—C18—H18C	109.00
C2—O2—C3—C4	100.60 (14)	O2—C3—C4—N1	−60.56 (15)
C3—O2—C2—C1	−96.47 (14)	O2—C3—C4—C5	−178.64 (13)
C3—O2—C2—C11	120.88 (16)	C2—C3—C4—C5	−113.55 (15)
C10—O3—C7—C8	169.18 (14)	C2—C3—C4—N1	4.53 (16)
C10—O3—C7—C6	−68.97 (18)	C3—C4—C5—C6	−179.75 (13)
C4—N1—C1—C2	2.84 (18)	N1—C4—C5—C6	68.63 (17)
C4—N1—C1—O1	−177.41 (15)	C3—C4—C9—C8	178.88 (13)
C1—N1—C4—C5	113.01 (15)	C5—C4—C9—C8	55.45 (17)
C1—N1—C4—C9	−124.52 (15)	C9—C4—C5—C6	−55.21 (18)
C1—N1—C4—C3	−4.60 (17)	N1—C4—C9—C8	−68.20 (17)
N1—C1—C2—O2	62.13 (16)	C4—C5—C6—C7	55.95 (18)
O1—C1—C2—C11	22.9 (2)	C5—C6—C7—C8	−55.29 (18)
O1—C1—C2—O2	−117.63 (17)	C5—C6—C7—O3	−175.32 (13)
O1—C1—C2—C3	−179.43 (16)	O3—C7—C8—C9	179.04 (12)
N1—C1—C2—C11	−157.31 (14)	C6—C7—C8—C9	55.90 (17)
N1—C1—C2—C3	0.32 (17)	C7—C8—C9—C4	−56.98 (17)
O2—C2—C11—C16	22.8 (2)	C2—C11—C12—C13	−178.99 (15)
C1—C2—C11—C12	63.6 (2)	C2—C11—C12—C17	1.2 (2)
C1—C2—C3—C4	−3.17 (17)	C16—C11—C12—C13	−0.6 (2)
C3—C2—C11—C12	−88.3 (2)	C16—C11—C12—C17	179.58 (15)
C3—C2—C11—C16	93.3 (2)	C2—C11—C16—C15	178.10 (15)
C1—C2—C3—O2	103.25 (13)	C12—C11—C16—C15	−0.3 (2)
C11—C2—C3—C4	152.30 (15)	C11—C12—C13—C14	1.2 (2)
O2—C2—C11—C12	−158.82 (14)	C17—C12—C13—C14	−179.01 (16)
C1—C2—C11—C16	−114.81 (17)	C12—C13—C14—C15	−0.8 (3)
O2—C2—C3—C4	−106.43 (14)	C13—C14—C15—C16	−0.1 (2)
C11—C2—C3—O2	−101.28 (17)	C13—C14—C15—C18	−178.94 (16)
O2—C3—C4—C9	58.71 (17)	C14—C15—C16—C11	0.7 (2)
C2—C3—C4—C9	123.80 (14)	C18—C15—C16—C11	179.49 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O3^{i}	0.86	2.25	3.0760 (17)	160
C9—H9*A*···O2^{ii}	0.97	2.56	3.413 (2)	147

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