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Ethyl 2-(*N*-cyclohexyl-2-nitrophenylsulfonamido)-2-oxoacetate

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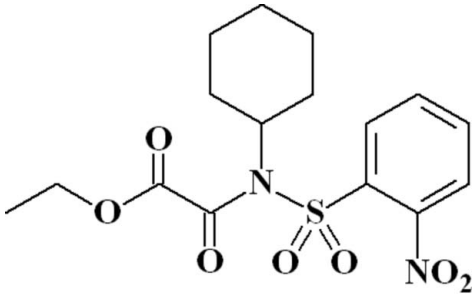
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.005 Å; R factor = 0.047; wR factor = 0.128; data-to-parameter ratio = 13.7.

In the title compound, $C_{16}H_{20}N_2O_7S$, the cyclohexane ring has a chair conformation and the two C=O groups are in a *trans* arrangement, the O=C−C=O torsion angle being −114.0 (5)°. The dihedral angle between the tertiary amine plane and the ethoxycarbonyl group O−C=O is 75.1 (2)°, while the dihedral angle between the amine plane and the benzene ring is 74.0 (2)°.

Related literature

For related reports, see: Schulz *et al.* (1988); Aulabaugh & Schloss (1990); Lee *et al.* (2005); Wang *et al.* (2005).



Experimental

Crystal data

$C_{16}H_{20}N_2O_7S$
M_r = 384.40
Monoclinic, $P2_1/n$
a = 10.962 (7) Å
b = 12.352 (7) Å
c = 13.879 (8) Å
β = 101.813 (11)°
V = 1839.5 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 294 (2) K
0.24 × 0.22 × 0.20 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.896, T_{max} = 0.960
9177 measured reflections
3240 independent reflections
1813 reflections with $I > 2\sigma(I)$
R_{int} = 0.074

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.129
S = 1.01
3240 reflections
237 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2108).

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supplementary materials

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Ethyl 2-(*N*-cyclohexyl-2-nitrophenylsulfonamido)-2-oxoacetate

B.-L. Wang, F.-Q. He, Z.-M. Li and H.-B. Song

Comment

Inhibitors of KARI (ketol-acid reductoisomerase) have been synthesized and tested as herbicides. 2-Dimethylphosphinoyl-2-hydroxy acetic acid (Hoe 704, Schulz *et al.*, 1988), *N*-hydroxy-*N*-isopropyloxamate (IpOHA, Aulabaugh *et al.*, 1990) and cyclopropane-1,1-dicarboxylate (CPD, Lee *et al.*, 2005) are potent inhibitors of the enzyme *in vitro* but their activity as herbicides are weak. There are few reports about the design, synthesis and biological activity of new KARI inhibitors. Nevertheless, inhibitors of KARI remain a potential source for finding novel herbicidal compounds (Wang *et al.*, 2005). With this in mind, a series of ethyl-2-(*N*-substituted-arylsulfonamido)-2-oxoacetate compounds has been designed and synthesized, based on the structure of KARI inhibitor IpOHA. The X-ray crystal structure determination of the title compound, (I), was undertaken in order to investigate the relationship between structure and herbicidal activity.

The X-ray analysis reveals that in the title compound, the cyclohexane ring is in a chair conformation and is bonded to the amine N atom (Fig. 1). Two C═O groups are *trans* arranged, with the torsion angle O5—C13—C14—O6 being −114.0 (5)°. The dihedral angle between the tertiary amine plane (S1/C7/C13/N2) and ethoxylcarbonyl O—C═O group (O6/O7/O14) is 75.1 (2)°. The dihedral angle between the amine plane and benzene ring C1···C6 is 74.0 (2)°.

Experimental

To a well stirred solution of *N*-cyclohexyl-2-nitrobenzenesulfonamide (5.68 g, 0.02 mol) in 20 ml of dry THF was added 50% NaH (1.44 g, 0.03 mol) slowly. After 1 h under stirring, a solution of ethyl oxalyl chloride (2.73 g, 0.02 mol) in 5 ml of dry THF was dropwise added and the mixture further stirred at 313–323 K for 3 h. After removing the solvent, water was added (20 ml), and the mixture was extracted with ethyl acetate and dried over magnesium sulfate. The ester was removed and the residue was purified by chromatography over silica gel with petroleum ether/ethyl acetate as eluent (9:1), affording (I). Yield: 87%; m.p. 391–392 K. ^{1}H NMR ($CDCl_3$) δ: 8.34–7.75 (m, 4H, Ph—H), 4.36 (q, *J*=7.20 Hz, 2H, CH_2), 3.88–3.78 (m, 1H, cyclohexyl-CH), 1.37 (t, *J*=7.20 Hz, 3H, CH_3), 2.23–1.11 (m, 10H, cyclohexyl-CH_2). Anal. Calcd. for $C_{16}H_{20}N_2O_7S$: C 49.99, H 5.24, N 7.29%. Found: C 50.13, H 5.31, N 7.61%. Colourless single crystals suitable for X-ray diffraction analysis were obtained by recrystallization from petroleum ether and ethyl acetate (1:1).

Refinement

H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C—H distances of 0.93 (aromatic CH), 0.96 (methyl CH_3), 0.97 (methylene CH_2) or 0.98 Å (methine CH), and with U_{iso}(H) = 1.5U_{eq}(carrier C atom) for methyl group and U_{iso}(H) = 1.2U_{eq}(carrier C atom) for other H atoms.

Figures

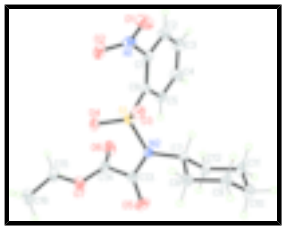


Fig. 1. The molecular structure of (I). Displacement ellipsoids are drawn at the 20% probability level.

Ethyl 2-(*N*-cyclohexyl-2-nitrophenylsulfonamido)-2-oxoacetate

Crystal data

$C_{16}H_{20}N_2O_7S$	F_{000} = 808
M_r = 384.40	D_x = 1.388 Mg m^{-3}
Monoclinic, $P2_1/n$	Melting point: 391-392 K
Hall symbol: -P 2yn	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 10.962 (7) Å	Cell parameters from 1731 reflections
b = 12.352 (7) Å	θ = 2.2–21.7º
c = 13.879 (8) Å	μ = 0.22 mm^{-1}
β = 101.813 (11)º	T = 294 (2) K
V = 1839.5 (19) Å^3	Block, colourless
Z = 4	0.24 × 0.22 × 0.20 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	3240 independent reflections
Radiation source: fine-focus sealed tube	1813 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.074
T = 294(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.2º
Absorption correction: multi-scan (SADABS; Bruker, 1998)	h = −13→11
T_{min} = 0.896, T_{max} = 0.960	k = −14→12
9177 measured reflections	l = −16→16

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.047	$w = 1/[\sigma^2(F_o^2) + (0.0538P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.129	$(\Delta/\sigma)_{max}$ = 0.001

S = 1.01	$\Delta\rho_{max}$ = 0.23 e Å^{-3}
3240 reflections	$\Delta\rho_{min}$ = −0.26 e Å^{-3}
237 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.029 (2)
Secondary atom site location: difference Fourier map	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.30775 (8)	0.54366 (6)	0.13647 (6)	0.0434 (3)
O1	−0.0287 (3)	0.6633 (2)	0.0056 (2)	0.0895 (10)
O2	0.1287 (3)	0.7293 (2)	0.10732 (18)	0.0711 (8)
O3	0.2700 (2)	0.56089 (16)	0.03315 (15)	0.0525 (6)
O4	0.3794 (2)	0.62380 (16)	0.19704 (17)	0.0578 (7)
O5	0.5814 (3)	0.3564 (2)	0.2134 (2)	0.1016 (11)
O6	0.4471 (3)	0.4822 (2)	0.36867 (19)	0.0739 (8)
O7	0.6185 (2)	0.55085 (19)	0.32483 (16)	0.0622 (7)
N1	0.0524 (3)	0.6590 (2)	0.0797 (2)	0.0554 (8)
N2	0.3898 (2)	0.42994 (18)	0.15192 (18)	0.0429 (7)
C1	0.0573 (3)	0.5618 (2)	0.1426 (2)	0.0444 (8)
C2	−0.0531 (3)	0.5281 (3)	0.1653 (3)	0.0630 (10)
H2	−0.1273	0.5634	0.1389	0.076*
C3	−0.0524 (4)	0.4413 (3)	0.2278 (3)	0.0704 (11)
H3	−0.1263	0.4173	0.2438	0.085*
C4	0.0586 (4)	0.3903 (3)	0.2665 (3)	0.0645 (11)
H4	0.0592	0.3324	0.3094	0.077*
C5	0.1687 (3)	0.4233 (2)	0.2427 (2)	0.0494 (9)
H5	0.2425	0.3874	0.2694	0.059*
C6	0.1703 (3)	0.5101 (2)	0.1792 (2)	0.0396 (8)
C7	0.3473 (3)	0.3418 (2)	0.0781 (2)	0.0423 (8)
H7	0.2593	0.3564	0.0502	0.051*
C8	0.4136 (3)	0.3436 (3)	−0.0075 (2)	0.0556 (10)
H8A	0.5012	0.3272	0.0158	0.067*
H8B	0.4072	0.4152	−0.0368	0.067*
C9	0.3552 (3)	0.2602 (3)	−0.0844 (2)	0.0618 (11)
H9A	0.2698	0.2809	−0.1121	0.074*
H9B	0.4008	0.2591	−0.1374	0.074*
C10	0.3567 (4)	0.1474 (3)	−0.0398 (3)	0.0631 (11)
H10A	0.3144	0.0972	−0.0892	0.076*
H10B	0.4422	0.1232	−0.0186	0.076*
C11	0.2935 (4)	0.1475 (3)	0.0467 (3)	0.0668 (11)
H11A	0.2993	0.0757	0.0757	0.080*
H11B	0.2059	0.1644	0.0241	0.080*
C12	0.3517 (4)	0.2296 (3)	0.1251 (2)	0.0613 (10)
H12A	0.4374	0.2097	0.1524	0.074*
H12B	0.3061	0.2302	0.1780	0.074*

C13	0.5018 (4)	0.4223 (3)	0.2206 (3)	0.0598 (10)
C14	0.5169 (4)	0.4910 (3)	0.3133 (3)	0.0539 (9)
C15	0.6404 (4)	0.6210 (3)	0.4111 (3)	0.0705 (11)
H15A	0.6423	0.5787	0.4702	0.085*
H15B	0.5742	0.6743	0.4057	0.085*
C16	0.7611 (4)	0.6753 (4)	0.4156 (4)	0.1046 (16)
H16A	0.8246	0.6219	0.4152	0.157*
H16B	0.7816	0.7173	0.4749	0.157*
H16C	0.7557	0.7221	0.3597	0.157*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0479 (5)	0.0345 (5)	0.0484 (5)	−0.0014 (4)	0.0111 (4)	−0.0009 (4)
O1	0.068 (2)	0.105 (2)	0.083 (2)	0.0049 (16)	−0.0142 (17)	0.0407 (17)
O2	0.099 (2)	0.0392 (15)	0.0726 (18)	−0.0055 (15)	0.0123 (16)	0.0008 (13)
O3	0.0659 (16)	0.0518 (14)	0.0419 (13)	0.0043 (11)	0.0156 (11)	0.0094 (11)
O4	0.0561 (16)	0.0374 (13)	0.0761 (16)	−0.0097 (11)	0.0047 (13)	−0.0102 (12)
O5	0.070 (2)	0.104 (2)	0.112 (2)	0.0413 (18)	−0.0251 (17)	−0.0567 (19)
O6	0.082 (2)	0.0768 (19)	0.0632 (17)	−0.0175 (15)	0.0156 (16)	−0.0083 (14)
O7	0.0566 (16)	0.0690 (17)	0.0578 (15)	−0.0069 (13)	0.0042 (12)	−0.0229 (13)
N1	0.056 (2)	0.050 (2)	0.059 (2)	0.0125 (16)	0.0109 (17)	0.0091 (16)
N2	0.0451 (17)	0.0363 (15)	0.0455 (16)	0.0027 (12)	0.0050 (13)	−0.0092 (12)
C1	0.050 (2)	0.042 (2)	0.0414 (19)	0.0009 (17)	0.0101 (16)	0.0018 (15)
C2	0.047 (2)	0.070 (3)	0.073 (3)	0.006 (2)	0.017 (2)	0.003 (2)
C3	0.062 (3)	0.072 (3)	0.084 (3)	−0.008 (2)	0.032 (2)	0.012 (2)
C4	0.075 (3)	0.056 (2)	0.070 (3)	−0.006 (2)	0.032 (2)	0.014 (2)
C5	0.056 (2)	0.039 (2)	0.054 (2)	0.0025 (16)	0.0137 (18)	0.0072 (16)
C6	0.046 (2)	0.0351 (18)	0.0382 (18)	−0.0046 (15)	0.0088 (15)	−0.0032 (14)
C7	0.045 (2)	0.0376 (19)	0.0441 (19)	−0.0018 (15)	0.0078 (16)	−0.0091 (15)
C8	0.059 (2)	0.051 (2)	0.062 (2)	−0.0047 (17)	0.026 (2)	−0.0094 (18)
C9	0.076 (3)	0.061 (2)	0.054 (2)	−0.002 (2)	0.025 (2)	−0.0127 (19)
C10	0.078 (3)	0.051 (2)	0.061 (2)	−0.0043 (19)	0.016 (2)	−0.0168 (19)
C11	0.095 (3)	0.041 (2)	0.067 (3)	−0.007 (2)	0.022 (2)	−0.0048 (19)
C12	0.088 (3)	0.046 (2)	0.050 (2)	0.000 (2)	0.014 (2)	−0.0057 (18)
C13	0.054 (2)	0.054 (2)	0.066 (2)	0.0071 (19)	−0.002 (2)	−0.0185 (19)
C14	0.051 (2)	0.050 (2)	0.055 (2)	0.0026 (19)	−0.005 (2)	−0.0090 (18)
C15	0.072 (3)	0.066 (3)	0.069 (3)	−0.012 (2)	0.003 (2)	−0.027 (2)
C16	0.094 (4)	0.094 (3)	0.125 (4)	−0.035 (3)	0.019 (3)	−0.045 (3)

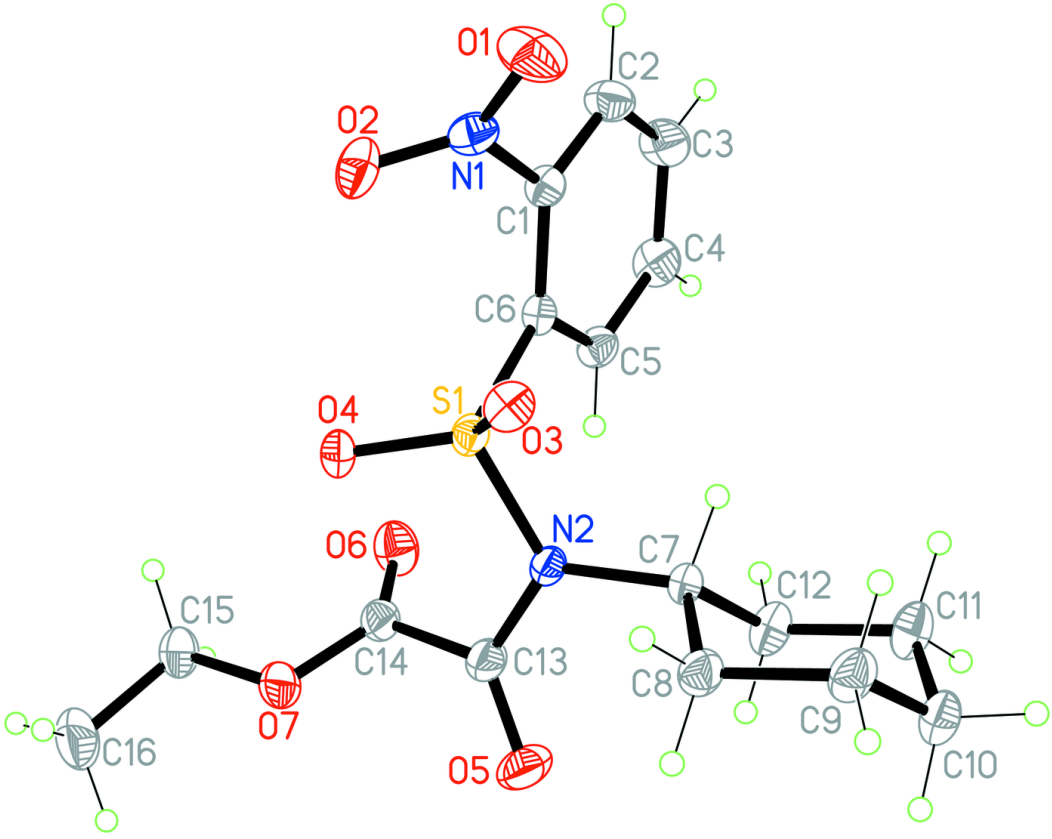
Geometric parameters (Å, °)

S1—O3	1.425 (2)	C7—C12	1.528 (4)
S1—O4	1.426 (2)	C7—H7	0.9800
S1—N2	1.658 (3)	C8—C9	1.527 (4)
O5—C13	1.212 (4)	C8—H8A	0.9700
N2—C13	1.395 (4)	C8—H8B	0.9700
N2—C7	1.502 (4)	C9—C10	1.524 (4)
S1—C6	1.778 (3)	C9—H9A	0.9700

O1—N1	1.216 (4)	C9—H9B	0.9700
O2—N1	1.212 (3)	C10—C11	1.504 (5)
O6—C14	1.195 (4)	C10—H10A	0.9700
O7—C14	1.319 (4)	C10—H10B	0.9700
O7—C15	1.458 (4)	C11—C12	1.528 (4)
N1—C1	1.478 (4)	C11—H11A	0.9700
C1—C2	1.377 (5)	C11—H11B	0.9700
C1—C6	1.395 (4)	C12—H12A	0.9700
C2—C3	1.377 (5)	C12—H12B	0.9700
C2—H2	0.9300	C13—C14	1.522 (5)
C3—C4	1.378 (5)	C15—C16	1.473 (5)
C3—H3	0.9300	C15—H15A	0.9700
C4—C5	1.376 (5)	C15—H15B	0.9700
C4—H4	0.9300	C16—H16A	0.9600
C5—C6	1.390 (4)	C16—H16B	0.9600
C5—H5	0.9300	C16—H16C	0.9600
C7—C8	1.514 (4)		
O3—S1—C6	106.24 (15)	C10—C9—C8	111.4 (3)
O3—S1—O4	120.07 (14)	C10—C9—H9A	109.4
O3—S1—N2	107.14 (13)	C8—C9—H9A	109.4
O4—S1—N2	107.06 (14)	C10—C9—H9B	109.4
O4—S1—C6	111.55 (14)	C8—C9—H9B	109.4
N2—S1—C6	103.50 (14)	H9A—C9—H9B	108.0
C7—N2—S1	116.2 (2)	C11—C10—C9	110.8 (3)
C13—N2—C7	121.6 (2)	C11—C10—H10A	109.5
C13—N2—S1	121.6 (2)	C9—C10—H10A	109.5
C14—O7—C15	115.2 (3)	C11—C10—H10B	109.5
O2—N1—O1	125.0 (3)	C9—C10—H10B	109.5
O2—N1—C1	117.1 (3)	H10A—C10—H10B	108.1
O1—N1—C1	117.8 (3)	C10—C11—C12	112.0 (3)
C2—C1—C6	122.3 (3)	C10—C11—H11A	109.2
C2—C1—N1	117.0 (3)	C12—C11—H11A	109.2
C6—C1—N1	120.6 (3)	C10—C11—H11B	109.2
C1—C2—C3	119.2 (3)	C12—C11—H11B	109.2
C1—C2—H2	120.4	H11A—C11—H11B	107.9
C3—C2—H2	120.4	C11—C12—C7	109.1 (3)
C2—C3—C4	119.4 (4)	C11—C12—H12A	109.9
C2—C3—H3	120.3	C7—C12—H12A	109.9
C4—C3—H3	120.3	C11—C12—H12B	109.9
C5—C4—C3	121.3 (3)	C7—C12—H12B	109.9
C5—C4—H4	119.4	H12A—C12—H12B	108.3
C3—C4—H4	119.4	O5—C13—N2	122.5 (3)
C4—C5—C6	120.4 (3)	O5—C13—C14	119.3 (3)
C4—C5—H5	119.8	N2—C13—C14	117.9 (3)
C6—C5—H5	119.8	O6—C14—O7	127.5 (3)
C5—C6—C1	117.3 (3)	O6—C14—C13	121.3 (3)
C5—C6—S1	120.9 (2)	O7—C14—C13	111.0 (4)
C1—C6—S1	121.4 (2)	O7—C15—C16	107.5 (3)
N2—C7—C8	113.1 (2)	O7—C15—H15A	110.2

N2—C7—C12	112.9 (2)	C16—C15—H15A	110.2
C8—C7—C12	111.7 (3)	O7—C15—H15B	110.2
N2—C7—H7	106.2	C16—C15—H15B	110.2
C8—C7—H7	106.2	H15A—C15—H15B	108.5
C12—C7—H7	106.2	C15—C16—H16A	109.5
C7—C8—C9	109.8 (3)	C15—C16—H16B	109.5
C7—C8—H8A	109.7	H16A—C16—H16B	109.5
C9—C8—H8A	109.7	C15—C16—H16C	109.5
C7—C8—H8B	109.7	H16A—C16—H16C	109.5
C9—C8—H8B	109.7	H16B—C16—H16C	109.5
H8A—C8—H8B	108.2		
O3—S1—N2—C13	135.1 (3)	O4—S1—C6—C1	93.4 (3)
O4—S1—N2—C13	5.1 (3)	N2—S1—C6—C1	−151.9 (2)
C6—S1—N2—C13	−112.9 (3)	C13—N2—C7—C8	−77.8 (4)
O3—S1—N2—C7	−35.9 (2)	S1—N2—C7—C8	93.3 (3)
O4—S1—N2—C7	−166.0 (2)	C13—N2—C7—C12	50.3 (4)
C6—S1—N2—C7	76.1 (2)	S1—N2—C7—C12	−138.7 (2)
O2—N1—C1—C2	130.9 (3)	N2—C7—C8—C9	−173.6 (3)
O1—N1—C1—C2	−47.8 (4)	C12—C7—C8—C9	57.7 (4)
O2—N1—C1—C6	−46.9 (4)	C7—C8—C9—C10	−56.1 (4)
O1—N1—C1—C6	134.4 (3)	C8—C9—C10—C11	55.5 (4)
C6—C1—C2—C3	1.1 (5)	C9—C10—C11—C12	−56.1 (4)
N1—C1—C2—C3	−176.7 (3)	C10—C11—C12—C7	56.6 (4)
C1—C2—C3—C4	0.2 (6)	N2—C7—C12—C11	173.7 (3)
C2—C3—C4—C5	−1.0 (6)	C8—C7—C12—C11	−57.5 (4)
C3—C4—C5—C6	0.4 (5)	C7—N2—C13—O5	14.3 (6)
C4—C5—C6—C1	0.8 (5)	S1—N2—C13—O5	−156.2 (3)
C4—C5—C6—S1	−172.5 (3)	C7—N2—C13—C14	−158.5 (3)
C2—C1—C6—C5	−1.6 (5)	S1—N2—C13—C14	30.9 (4)
N1—C1—C6—C5	176.1 (3)	C15—O7—C14—O6	−6.1 (5)
C2—C1—C6—S1	171.7 (3)	C15—O7—C14—C13	179.1 (3)
N1—C1—C6—S1	−10.6 (4)	O5—C13—C14—O6	−114.0 (5)
O3—S1—C6—C5	133.9 (2)	N2—C13—C14—O6	59.1 (5)
O4—S1—C6—C5	−93.6 (3)	O5—C13—C14—O7	61.2 (5)
N2—S1—C6—C5	21.2 (3)	N2—C13—C14—O7	−125.7 (3)
O3—S1—C6—C1	−39.2 (3)	C14—O7—C15—C16	176.2 (3)

Fig. 1


O1
C2
O2
C3
N1
C1
C4
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O4
S1
O3
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C7
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C11
C15
C14
C13
C8
O7
C9
C16
O5
C10