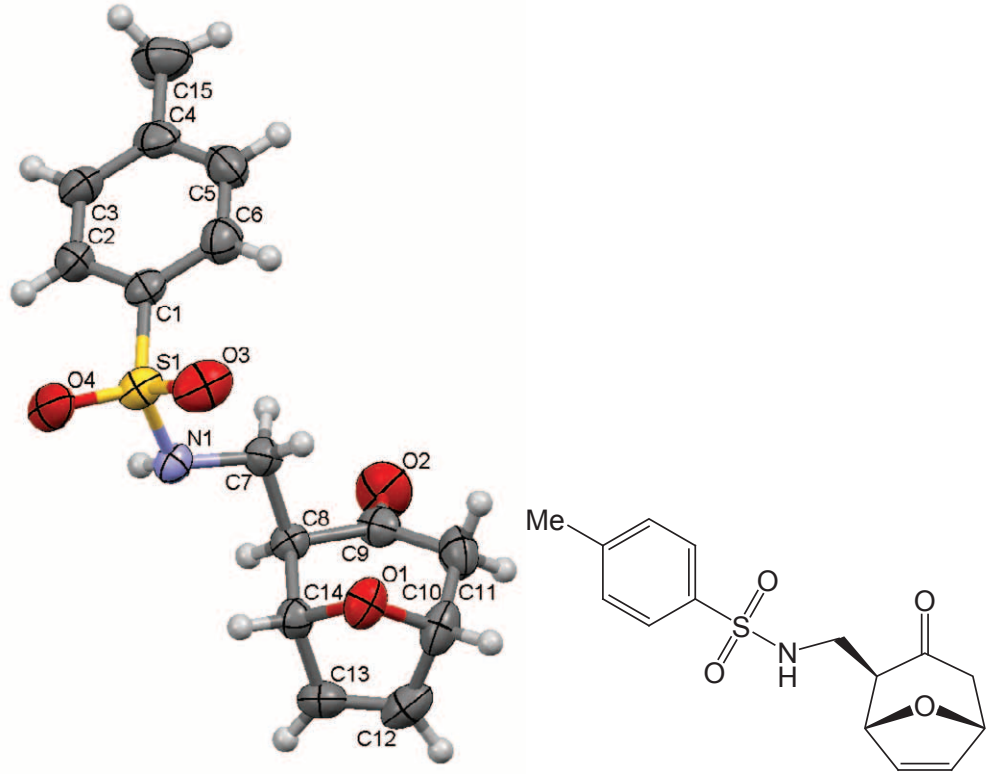
X-ray Crystal Structure of (–)-β-4aa

The following crystal structure has been deposited at the Cambridge Crystallographic Data Centre and allocated the deposition number CCDC 976137.



4-Methyl-N-(3-oxo-8-oxa-bicyclo[3.2.1]oct-6-en-2-yl methyl)-benzenesulfonamide

Comment

The compound, 4-Methyl-*N*-(3-oxo-8-oxa-bicyclo-[3.2.1]oct-6-en-2-yl-methyl)-benzene-sulfonamide, crystallizes in a primitive orthorhombic space group, $P\,2_12_12_1$ (#19). There are 4 asymmetric units in the unit cell. The compound is chiral. The absolute configuration is as shown, Flack parameter being 0.04 (8) based on 1054 Friedel pairs. The chiral atoms C8, C11 and C14 are in *R*-configurations.

The cyclohept-4-enone ring is in a flatened boat form, the tetrahydropyran-4-one ring in distorted chair form due to the ketone group. The five-membered ring, 2,5-dihydrofuran, is in the envelop form. All the bonding parameters are within the normal ranges.

There is intermolecular H-bond interaction, N1—H1···O3, present in the crystal lattice.

Experimental

A colourless plate crystal of $C_{15}H_{17}NO_4S$, having approximate dimensions of 0.16 *x* 0.18 *x* 0.36 mm was mounted on glass fiber. All measurements were made on a Bruker *Apex II* CCD detector with graphite monochromated Mo—Kα radiation. The crystal-to-detector distance was 50.00 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive orthorhombic cell with dimensions: a = 5.0949 (2) Å, b = 12.0655 (4) Å, c = 24.2090 (7) Å, V = 1488.19 (9) Å^3.

For $Z = 4$ and F.W. = 307.36, the calculated density is 1.372 g/cm^3. Based on a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be: $P\,2_12_12_1$ (#19)

The data were collected at a temperature of 23(1)°C to a maximum 2θ value of 54.9°. The exposure rate was 30.0 [sec./°]. The crystal-to-detector distance was 50.00 mm.

Of the 11344 reflections that were collected, 2616 reflections were unique. (R_{int} = 0.0336); equivalent reflections were merged.

Refinement

The structure was solved by direct methods (*SHELXS-97*) and expanded using Fourier techniques. All non-H atoms were refined anisotropically.
All of the C-bound H atoms were observable from difference Fourier map but were all placed at geometrical positions with C—H = 0.93, 0.96, 0.97 and 0.98Å for phenyl/vinyl, methyl, methylene and methine H-atoms respectively. All C-bound H-atoms were refined using riding model with U_{iso}(H) = 1.2U_{eq}(Carrier). The N-bound hydrogen atom was located from difference Fourier map and refined isotropically.
Highest peak is 0.17 at (0.0420, 0.2732, 0.0837) [0.89Å from H15C] Deepest hole is -0.21 at (0.1441, 0.9396, 0.0968) [0.68Å from S1]

Computing details

Data collection: *APEX* (Bruker AXS Inc, 2007); cell refinement: *SAINT* v7.34A (Bruker AXS Inc, 2007); data reduction: *CrystalStructure* (Rigaku/MSC and Rigaku Corporation, 2006); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: Mercury (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

Acknowledgements
The authors thanked Dr. Wesley T.K. Chan of the Hong Kong Polytechnic University for collecting the X-ray diffraction data.

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Crystal data

$C_{15}H_{17}NO_4S$	F(000) = 648
M_r = 307.36	D_x = 1.372 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation, l = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 11344 reflections
a = 5.0949(2)Å	θ = 1.7–25.0°
b = 12.0655(4)Å	μ = 0.23 mm^{-1}
c = 24.2090(7)Å	T = 296 K

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$V = 1488.19(9)Å^3$	Rod, colourless
$Z = 4$	0.36 × 0.18 × 0.16 mm

Data collection

Bruker APEX CCD diffractometer	2616 independent reflections
Radiation source: fine-focus sealed tube	2306 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.034$
ω & ϕ scans	$\theta_{max} = 25.0°$, $\theta_{min} = 1.7°$
Absorption correction: multi-scan *SADABS* (Sheldrick, 2008)	$h = -6 \rightarrow 6$
$T_{min} = 0.921$, $T_{max} = 0.964$	$k = -11 \rightarrow 14$
11344 measured reflections	$l = -28 \rightarrow 28$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.034$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.077$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.07$	$w = 1/[s^2(F_o^2) + (0.0361P)^2 + 0.199P]$ where $P = (F_o^2 + 2F_c^2)/3$
2616 reflections	$(\Delta/s)_{max} = 0.001$
196 parameters	$\Delta\rho_{max} = 0.17$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.21$ e Å^{-3}
Flack parameter: 0.04 (8)	Absolute structure: Flack (1983). 1054 Friedel pairs

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.87519 (11)	0.38532 (5)	0.40888 (2)	0.04290 (17)
O1	0.9715 (3)	0.55905 (13)	0.23178 (6)	0.0487 (4)
O2	0.4293 (4)	0.75312 (14)	0.29953 (8)	0.0729 (6)

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O3	1.1392 (3)	0.41462 (16)	0.39465 (7)	0.0636 (5)
O4	0.8054 (4)	0.27188 (12)	0.41694 (7)	0.0592 (5)
N1	0.6927 (4)	0.43064 (16)	0.35866 (8)	0.0383 (4)
C1	0.7853 (4)	0.46089 (18)	0.46825 (9)	0.0400 (5)
C2	0.5843 (5)	0.42179 (19)	0.50105 (9)	0.0465 (6)
H2	0.5081	0.3534	0.4936	0.056*
C3	0.4971 (6)	0.4847 (2)	0.54491 (9)	0.0519 (6)
H3	0.3636	0.4574	0.5673	0.062*
C4	0.6046 (5)	0.58763 (19)	0.55620 (9)	0.0488 (6)
C5	0.8073 (5)	0.6251 (2)	0.52321 (10)	0.0548 (6)
H5	0.8835	0.6936	0.5306	0.066*
C6	0.8986 (5)	0.56248 (19)	0.47944 (10)	0.0519 (6)
H6	1.0356	0.5886	0.4576	0.062*
C7	0.7175 (5)	0.54775 (17)	0.34314 (9)	0.0407 (5)
H7A	0.9018	0.5675	0.3409	0.049*
H7B	0.6367	0.5937	0.3713	0.049*
C8	0.5854 (4)	0.56933 (16)	0.28751 (8)	0.0348 (5)
H8	0.4022	0.5449	0.2896	0.042*
C9	0.5916 (5)	0.69510 (18)	0.27763 (9)	0.0433 (6)
C10	0.8088 (5)	0.74267 (19)	0.24330 (11)	0.0569 (7)
H10A	0.9515	0.7655	0.2673	0.068*
H10B	0.7451	0.8078	0.2240	0.068*
C11	0.9115 (5)	0.6590 (2)	0.20125 (10)	0.0546 (7)
H11	1.0632	0.6870	0.1807	0.066*
C12	0.6942 (5)	0.6215 (2)	0.16399 (10)	0.0581 (7)
H12	0.6473	0.6541	0.1306	0.070*
C13	0.5816 (5)	0.5350 (2)	0.18658 (9)	0.0499 (6)
H13	0.4400	0.4959	0.1721	0.060*
C14	0.7198 (4)	0.50975 (18)	0.23959 (9)	0.0381 (5)
H14	0.7328	0.4298	0.2461	0.046*
C15	0.5020 (7)	0.6561 (2)	0.60355 (10)	0.0725 (9)
H15A	0.5239	0.6160	0.6375	0.087*
H15B	0.3191	0.6715	0.5978	0.087*
H15C	0.5976	0.7246	0.6055	0.087*
H1	0.545 (5)	0.4161 (18)	0.3655 (9)	0.042 (7)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0417 (3)	0.0487 (3)	0.0383 (3)	0.0116 (3)	0.0021 (3)	0.0036 (3)
O1	0.0337 (8)	0.0571 (10)	0.0554 (10)	0.0069 (7)	0.0074 (8)	0.0117 (8)
O2	0.0854 (15)	0.0518 (10)	0.0814 (13)	0.0291 (11)	0.0122 (12)	-0.0066 (9)
O3	0.0374 (8)	0.0946 (14)	0.0589 (11)	0.0164 (9)	0.0026 (8)	0.0032 (10)
O4	0.0883 (13)	0.0385 (9)	0.0508 (10)	0.0169 (9)	0.0030 (10)	0.0049 (8)
N1	0.0360 (10)	0.0420 (11)	0.0370 (11)	-0.0007 (8)	0.0030 (8)	0.0045 (8)
C1	0.0413 (12)	0.0452 (12)	0.0335 (12)	0.0046 (10)	-0.0043 (10)	0.0055 (10)
C2	0.0593 (15)	0.0417 (12)	0.0386 (13)	-0.0042 (12)	0.0003 (11)	0.0015 (10)
C3	0.0637 (15)	0.0561 (15)	0.0359 (14)	-0.0021 (13)	0.0066 (12)	0.0044 (11)
C4	0.0639 (15)	0.0474 (14)	0.0349 (12)	0.0068 (13)	-0.0111 (12)	0.0005 (10)

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C5	0.0658 (16)	0.0415 (12)	0.0571 (16)	-0.0048 (13)	-0.0114 (14)	-0.0035 (12)
C6	0.0484 (13)	0.0555 (14)	0.0518 (15)	-0.0079 (13)	0.0010 (13)	0.0051 (12)
C7	0.0454 (12)	0.0391 (12)	0.0376 (12)	-0.0004 (10)	-0.0018 (10)	-0.0015 (10)
C8	0.0319 (11)	0.0375 (11)	0.0350 (11)	0.0025 (10)	-0.0006 (9)	0.0008 (9)
C9	0.0501 (13)	0.0367 (12)	0.0430 (13)	0.0099 (12)	-0.0101 (12)	-0.0011 (10)
C10	0.0623 (16)	0.0395 (13)	0.0687 (18)	-0.0053 (12)	-0.0047 (14)	0.0091 (12)
C11	0.0480 (15)	0.0568 (15)	0.0591 (15)	-0.0010 (12)	0.0090 (13)	0.0223 (12)
C12	0.0668 (16)	0.0692 (17)	0.0384 (14)	0.0158 (15)	0.0068 (12)	0.0116 (13)
C13	0.0558 (15)	0.0570 (15)	0.0369 (13)	0.0025 (13)	-0.0041 (12)	-0.0083 (11)
C14	0.0385 (11)	0.0351 (11)	0.0407 (13)	0.0009 (9)	0.0026 (10)	-0.0001 (10)
C15	0.096 (2)	0.0696 (19)	0.0520 (17)	0.0169 (17)	-0.0023 (17)	-0.0140 (13)

Geometric parameters (Å, º)

S1—O4	1.4275 (16)	C7—C8	1.528 (3)
S1—O3	1.4330 (17)	C7—H7A	0.9700
S1—N1	1.6252 (19)	C7—H7B	0.9700
S1—C1	1.763 (2)	C8—C14	1.527 (3)
O1—C14	1.426 (3)	C8—C9	1.537 (3)
O1—C11	1.447 (3)	C8—H8	0.9800
O2—C9	1.206 (3)	C9—C10	1.498 (3)
N1—C7	1.467 (3)	C10—C11	1.526 (3)
N1—H1	0.79 (2)	C10—H10A	0.9700
C1—C2	1.379 (3)	C10—H10B	0.9700
C1—C6	1.382 (3)	C11—C12	1.498 (4)
C2—C3	1.379 (3)	C11—H11	0.9800
C2—H2	0.9300	C12—C13	1.311 (3)
C3—C4	1.385 (3)	C12—H12	0.9300
C3—H3	0.9300	C13—C14	1.495 (3)
C4—C5	1.382 (3)	C13—H13	0.9300
C4—C15	1.507 (3)	C14—H14	0.9800
C5—C6	1.382 (3)	C15—H15A	0.9600
C5—H5	0.9300	C15—H15B	0.9600
C6—H6	0.9300	C15—H15C	0.9600
O4—S1—O3	120.21 (11)	C14—C8—H8	108.9
O4—S1—N1	106.40 (11)	C7—C8—H8	108.9
O3—S1—N1	105.91 (10)	C9—C8—H8	108.9
O4—S1—C1	108.65 (11)	O2—C9—C10	121.9 (2)
O3—S1—C1	108.21 (11)	O2—C9—C8	119.4 (2)
N1—S1—C1	106.69 (10)	C10—C9—C8	118.68 (19)
C14—O1—C11	103.02 (16)	C9—C10—C11	111.7 (2)
C7—N1—S1	117.79 (15)	C9—C10—H10A	109.3
C7—N1—H1	110.5 (16)	C11—C10—H10A	109.3
S1—N1—H1	108.2 (16)	C9—C10—H10B	109.3
C2—C1—C6	120.1 (2)	C11—C10—H10B	109.3
C2—C1—S1	119.05 (17)	H10A—C10—H10B	107.9
C6—C1—S1	120.66 (18)	O1—C11—C12	102.26 (19)
C3—C2—C1	119.6 (2)	O1—C11—C10	106.44 (19)

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C3—C2—H2	120.2	C12—C11—C10	110.4 (2)
C1—C2—H2	120.2	O1—C11—H11	112.4
C2—C3—C4	121.2 (2)	C12—C11—H11	112.4
C2—C3—H3	119.4	C10—C11—H11	112.4
C4—C3—H3	119.4	C13—C12—C11	108.2 (2)
C5—C4—C3	118.4 (2)	C13—C12—H12	125.9
C5—C4—C15	121.3 (2)	C11—C12—H12	125.9
C3—C4—C15	120.3 (3)	C12—C13—C14	108.3 (2)
C4—C5—C6	121.1 (2)	C12—C13—H13	125.8
C4—C5—H5	119.5	C14—C13—H13	125.8
C6—C5—H5	119.5	O1—C14—C13	102.99 (18)
C1—C6—C5	119.6 (2)	O1—C14—C8	107.90 (17)
C1—C6—H6	120.2	C13—C14—C8	110.20 (17)
C5—C6—H6	120.2	O1—C14—H14	111.8
N1—C7—C8	110.60 (17)	C13—C14—H14	111.8
N1—C7—H7A	109.5	C8—C14—H14	111.8
C8—C7—H7A	109.5	C4—C15—H15A	109.5
N1—C7—H7B	109.5	C4—C15—H15B	109.5
C8—C7—H7B	109.5	H15A—C15—H15B	109.5
H7A—C7—H7B	108.1	C4—C15—H15C	109.5
C14—C8—C7	113.08 (17)	H15A—C15—H15C	109.5
C14—C8—C9	109.71 (17)	H15B—C15—H15C	109.5
C7—C8—C9	107.25 (16)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O3^{i}	0.79 (2)	2.19 (2)	2.958 (3)	166 (2)

Symmetry code: (i) *x*-1, *y*, *z*.