Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(1*RS*,2*RS*,6*SR*,8*SR*)-2-Methyl-8-(phenylsulfonyl)bicyclo[4.2.0]octan-1-ol

Wendy A. Loughlin, Michelle A. McCleary and Peter C. Healy*

School of Science, Griffith University, Nathan, Brisbane 4111, Australia

Correspondence e-mail: p.healy@griffith.edu.au

Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.006 Å
R factor = 0.053
wR factor = 0.174
Data-to-parameter ratio = 20.3

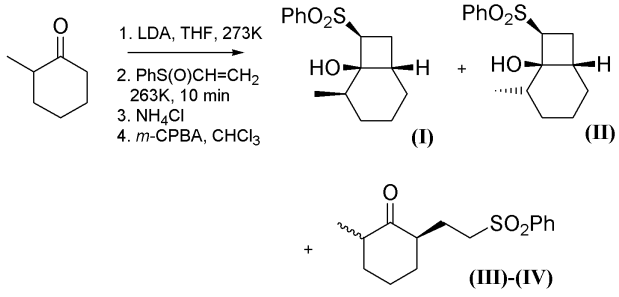
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{15}H_{20}O_3S$, shows a conformational arrangement of the phenyl ring that permits hydrogen-bonded dimers to be disposed about a centre of symmetry.

Received 3 June 2004
Accepted 7 June 2004
Online 12 June 2004

Comment

The preparation of fused carbocyclic ring systems bearing a bridgehead hydroxyl group has been achieved by the reaction between the lithium enolates of simple unfunctionalized ketones and phenyl vinyl sulfoxide (Loughlin *et al.*, 2002; Loughlin & McCleary, 2003; Healy *et al.*, 2002; Loughlin *et al.*, 2003). In the current study, reaction of the lithium enolate of 2-methylcyclohexan-1-one (obtained from lithium diisopropylamide, LDA) with phenyl vinyl sulfoxide and subsequent oxidation with *m*-chloroperoxybenzoic acid (*m*-CPBA) generated a novel methylbicyclo[4.2.0]octan-1-ol, compound (I).



Bicyclo[4.2.0]octan-1-ols with a methyl substituent proximal to the bridgehead have been observed as key intermediates in synthetic and mechanistic studies (Blanchard & Burnell, 2001; Syah & Ghisalberti, 1998; Jamart-Gregoire *et al.*, 1995). Under the present unoptimized reaction conditions, (I) was formed as the major bicyclo[4.2.0]octan-1-ol isomer in a 21:14:65 ratio of (I):(II):(III) and (IV) from 2-methylcyclohexan-1-one and phenyl vinyl sulfoxide (see Scheme). Here we report the synthesis, isolation and solid state structural characterization of the novel bicyclo[4.2.0]octan-1-ol, (I) (Fig. 1). The S—O3 bond in conjunction with the phenyl group is directed away from the bicyclo[4.2.0]alkan-1-ol ring, whereas the S—O2 bond is oriented towards this ring. The structure of (I) is stabilized by three-centred bifurcated intra- and intermolecular hydrogen bonds between the hydroxyl H and sulfone O atoms, forming a dimeric structure disposed about a crystallographic centre of symmetry (Table 2, Fig. 2). This differs from the chain-like intermolecular hydrogen-bonding pattern previously observed in the analogues of (I) that lack the C2 methyl group (Healy *et al.*, 2002; Loughlin *et al.*, 2003).

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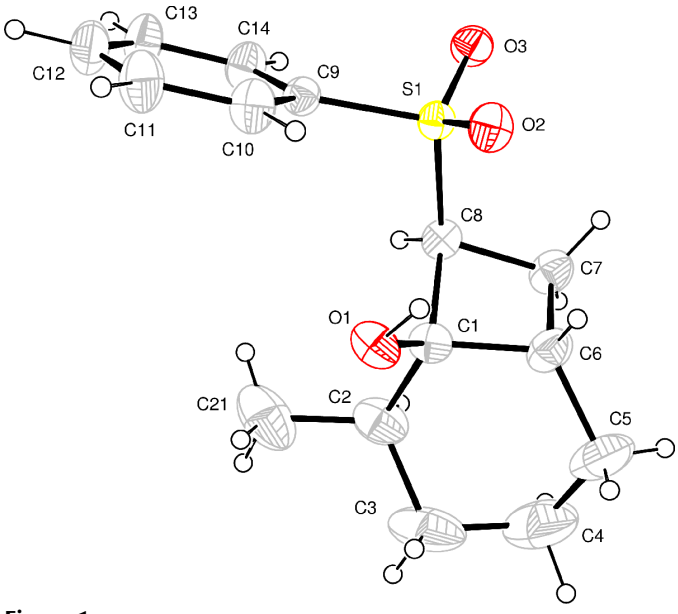

Figure 1
ORTEP-3 (Farrugia, 1997) plot, showing the atomic numbering scheme for the molecule of (I). Displacement ellipsoids for non-H atoms are drawn at the 30% probability level.

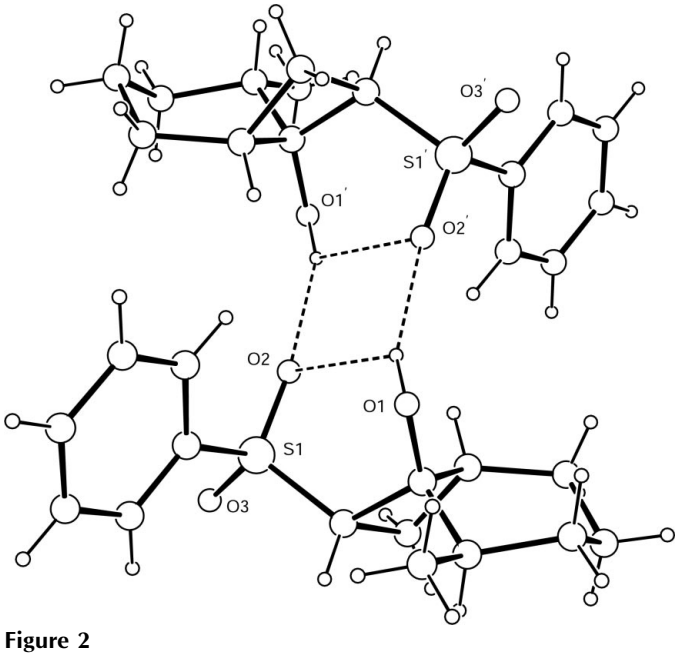

Figure 2
Hydrogen-bonded dimeric structure of (I). The primes correspond to symmetry code (i) in Table 2.

Experimental

2-Methylcyclohexanone (0.5 ml, 4.119 mmole) was reacted with lithium diisopropylamide (1.4 *M*, 2.95 ml, 4.119 mmol) in THF (44.5 ml) at 273 K under nitrogen over a period of 1 h. The reaction vessel was shielded from light. Rapid addition of phenyl vinyl sulfoxide (0.55 ml, 3.119 mmol) at 263 K with a 10 min reaction time and work-up as described elsewhere (Loughlin *et al.*, 2002) gave the crude sulfoxide mixture (1.074 g). This was subsequently oxidized with *m*-CPBA (1 equiv) in chloroform (50 ml). Work-up of the reaction mixture as described elsewhere (Loughlin *et al.*, 2002) was followed by silica chromatography (hexane:ethyl acetate, 60:40). A mixture of compounds (I)–(IV) (658 mg, 57%) was obtained. An analytically pure sample of compound (I) was obtained by semi-preparative HPLC (hexane–ethyl acetate, 80:20, retention time 11.8 min, 3 ml min^{-1}). Colourless crystals of compound (I) (m.p. 383.2–384.6 K) were isolated by slow evaporation of a hexane–ethyl acetate (80:20) solution of the compound. Analysis found: C 64.52, H 7.36, S 11.32%; calculated for $C_{15}H_{20}SO_3$: C 64.25, H 7.19, S 11.43%.

Crystal data

$C_{15}H_{20}O_3S$	D_x = 1.217 Mg m^{-3}
M_r = 280.38	Mo Kα radiation
Monoclinic, $P2_1/n$	Cell parameters from 24 reflections
a = 12.328 (6) Å	θ = 12.8–16.0°
b = 10.362 (4) Å	μ = 0.21 mm^{-1}
c = 12.149 (2) Å	T = 295 K
β = 99.60 (3)°	Prism, colourless
V = 1530.2 (10) Å^3	0.30 × 0.20 × 0.10 mm
Z = 4	

Data collection

Rigaku AFC-7*R* diffractometer	θ_{max} = 27.5°
ω–2θ scans	$h = -7 \rightarrow 16$
Absorption correction: none	$k = 0 \rightarrow 13$
3987 measured reflections	$l = -15 \rightarrow 15$
3516 independent reflections	3 standard reflections every 150 reflections
1717 reflections with $I > 2\sigma(I)$	intensity decay: none
R_{int} = 0.067	

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.053	$w = 1/[\sigma^2(F_o^2) + (0.0801P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.174	$(\Delta/\sigma)_{max}$ = 0.001
S = 0.98	$\Delta\rho_{max}$ = 0.29 e Å^{-3}
3516 reflections	$\Delta\rho_{min}$ = −0.35 e Å^{-3}
173 parameters	

Table 1
Selected geometric parameters (Å, °).

S1—O2	1.441 (2)	S1—C9	1.764 (4)
S1—O3	1.442 (2)	O1—C1	1.404 (4)
S1—C8	1.765 (4)		
O2—S1—O3	118.05 (14)	O1—C1—C6	117.1 (3)
O2—S1—C8	109.69 (15)	O1—C1—C8	119.4 (3)
O2—S1—C9	108.36 (15)	S1—C8—C1	117.3 (2)
O3—S1—C8	106.78 (15)	S1—C8—C7	112.1 (3)
O3—S1—C9	107.55 (15)	S1—C9—C10	120.2 (3)
C8—S1—C9	105.74 (18)	S1—C9—C14	119.2 (3)
O1—C1—C2	107.6 (3)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2	0.96	2.44	3.036 (4)	120
O1—H1···O2^{i}	0.96	2.02	2.918 (3)	154

Symmetry code: (i) $2-x, 1-y, 2-z$.

H atoms were positioned geometrically (C—H = 0.95 and O—H =0.96 Å) and refined as riding, with $U_{iso}(H) = 1.2U_{eq}$(parent atom).

Data collection: *MSC/AFC-7 Diffractometer Control Software for Windows* (Molecular Structure Corporation, 1999); cell refinement: *MSC/AFC-7 Diffractometer Control Software for Windows*; data reduction: *TEXSAN for Windows* (Molecular Structure Corporation, 1997–2001); program(s) used to solve structure: *TEXSAN* for

Windows; program(s) used to refine structure: *TEXSAN for Windows* and *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2001) and *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *TEXSAN* for Windows and *PLATON*.

The authors thank Griffith University and the Australian Research Council for financial assistance.

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