

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  
 $T = 295\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$   
 $R$  factor = 0.047  
 $wR$  factor = 0.131  
Data-to-parameter ratio = 19.3For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.(1*RS*,2*SR*,6*SR*,8*SR*)-2,6-Dimethyl-8-(phenylsulfonyl)bicyclo[4.2.0]octan-1-ol

The title compound,  $\text{C}_{16}\text{H}_{22}\text{O}_3\text{S}$ , exhibits a conformational arrangement that permits intramolecular  $\text{O}-\text{H}\cdots\text{O}-\text{S}$  hydrogen bonds. Intermolecular bifurcated hydrogen bonding between the hydroxyl H and the sulfone O atoms, observed in the unmethylated derivative, is disrupted by the presence of the methyl group at the 6-position.

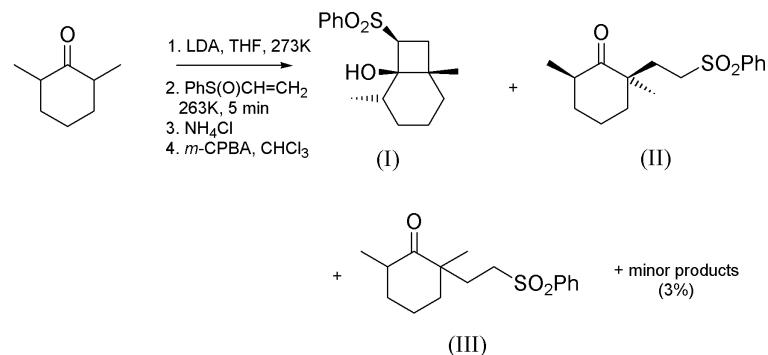
Received 4 June 2004

Accepted 7 June 2004

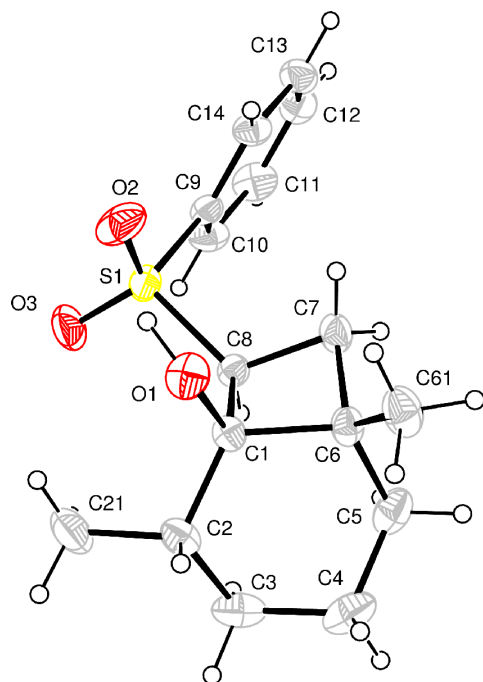
Online 12 June 2004

## Comment

We have recently shown that a novel cyclization reaction between the lithium enolates of simple unfunctionalized ketones and phenyl vinyl sulfoxide provides a simple and convenient route to the preparation of fused carbocyclic ring systems bearing a bridgehead hydroxyl group (Loughlin & McCleary, 2003; Loughlin, Rowen & Healy, 2002). In the current study, reaction of the lithium enolate of 2,6-dimethylcyclohexan-1-one (obtained from lithium diisopropylamide, LDA) with phenyl vinyl sulfoxide and subsequent oxidation with *m*-chloroperoxybenzoic acid (*m*-CPBA) generated a novel dimethylbicyclo[4.2.0]octan-1-ol, (I).



A bicyclo[4.2.0]octan-1-ol with a methyl bridgehead substituent can be perceived as a key structural component of, for example, natural products such as melleolide K (Momose *et al.*, 2000) and cyclobutatusin (Zelnik *et al.*, 1977; Wang *et al.*, 1974) and as a synthetic intermediate in natural product synthesis (Morisaki *et al.*, 1985, Ayer *et al.*, 1981). Under the present unoptimized reaction conditions, (I) was formed as the major bicyclo[4.2.0]octan-1-ol isomer in a 22:78 ratio of (I):(II) (Loughlin, McCleary & Healy, 2002) and (III) from 2,6-dimethylcyclohexan-1-one and phenyl vinyl sulfoxide with less than 5 percent of other products observed (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—O2 and S—O3 bonds are directed away from the bicyclo[4.2.0]alken-1-ol ring, whereas the phenyl group is oriented towards this ring. Intramolecular hydrogen-bonding



**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.

interactions are observed between the hydroxyl H atom and the sulfone O atom O2, with an O1...O2 distance of 2.765 (3) Å and an estimated O—H...O angle of 153°. The six-membered ring displays a pseudo-chair conformation. In the previously reported 8-(phenylsulfonyl)bicyclo[4.2.0]octan-1-ol, which lacks the 2,6-dimethyl groups of compound (I), the orientation of the six-membered ring allows intermolecular bifurcated hydrogen bonding between the hydroxyl H atom and the sulfone O atoms (Healy *et al.*, 2002). However, in compound (I) the presence of the methyl group in the C6 position now disrupts this and intermolecular hydrogen bonding is not observed in the solid-state structure of (I).

## Experimental

2,6-Dimethylcyclohexanone (0.55 ml, 3.96 mmole), lithium diisopropylamide (1.55 M, 2.56 ml, 3.96 mmole) in THF, and phenyl vinyl sulfoxide (0.55 ml, 3.96 mmol) were reacted at 263 K with a 5 min reaction time, worked up and subsequently oxidized with *m*-CPBA (1 equiv) in chloroform (50 ml) as described elsewhere (Loughlin, Rowen & Healy, 2002). After silica chromatography (hexane:ethyl acetate, 80:20), compound (I) was obtained (197 mg, 17%) in conjunction with monoalkylated 2,6-dimethylcyclohexanone, compounds (II) and (III) (577 mg, 53%) and other minor products (3%). An analytically pure sample of compound (I) was obtained by semi-preparative HPLC (hexane–ethyl acetate, 90:10, retention time 17.4 min, 3 ml min<sup>-1</sup>). Colourless crystals of compound (I) (m.p. 381.5–382.1 K) were isolated by slow evaporation of a hexane:ethyl acetate (90:10) solution of (I). Analysis found: C 65.34, H 7.64, O 16.36%; calculated for C<sub>16</sub>H<sub>22</sub>O<sub>3</sub>: C 65.27, H 7.53, O 16.30%.

## Crystal data

C<sub>16</sub>H<sub>22</sub>O<sub>3</sub>S  
*M<sub>r</sub>* = 294.41  
 Monoclinic, *P*2<sub>1</sub>/*c*  
*a* = 11.340 (3) Å  
*b* = 18.026 (6) Å  
*c* = 7.8442 (16) Å  
 $\beta$  = 106.788 (18)°  
*V* = 1535.1 (7) Å<sup>3</sup>  
*Z* = 4

*D<sub>x</sub>* = 1.274 Mg m<sup>-3</sup>  
 Mo K $\alpha$  radiation  
 Cell parameters from 25 reflections  
 $\theta$  = 12.6–17.4°  
 $\mu$  = 0.22 mm<sup>-1</sup>  
*T* = 295 K  
 Prism, colourless  
 0.40 × 0.35 × 0.20 mm

## Data collection

Rigaku AFC-7R diffractometer  
 $\omega$ -2 $\theta$  scans  
 Absorption correction: none  
 4336 measured reflections  
 3518 independent reflections  
 1904 reflections with *I* > 2 $\sigma$ (*I*)  
*R*<sub>int</sub> = 0.053

$\theta_{\max}$  = 27.5°  
*h* = -6 → 14  
*k* = -23 → 10  
*l* = -10 → 9  
 3 standard reflections  
 every 150 reflections  
 intensity decay: 0.8%

## Refinement

Refinement on *F*<sup>2</sup>  
*R* [*F*<sup>2</sup> > 2 $\sigma$ (*F*<sup>2</sup>)] = 0.047  
*wR*(*F*<sup>2</sup>) = 0.131  
*S* = 1.00  
 3518 reflections  
 182 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0382P)^2 + 0.7618P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.23 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.27 \text{ e \AA}^{-3}$

**Table 1**

Selected geometric parameters (Å, °).

S1—O2	1.445 (2)	S1—C9	1.768 (3)
S1—O3	1.432 (3)	O1—C1	1.425 (3)
S1—C8	1.770 (2)		
O2—S1—O3	118.11 (14)	O1—C1—C8	112.01 (18)
O2—S1—C8	108.50 (12)	O1—C1—C2	108.1 (2)
O2—S1—C9	107.31 (12)	S1—C8—C1	121.69 (16)
O3—S1—C8	110.16 (13)	S1—C8—C7	117.36 (18)
O3—S1—C9	108.45 (13)	S1—C9—C10	119.7 (2)
C8—S1—C9	103.25 (11)	S1—C9—C14	118.67 (19)
O1—C1—C6	109.7 (2)		

H atoms were positioned geometrically (C—H = 0.95, O—H = 0.96 Å) and refined as riding, with *U*<sub>iso</sub>(H) = 1.2*U*<sub>eq</sub>(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 *SHELXL97* (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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