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## 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.

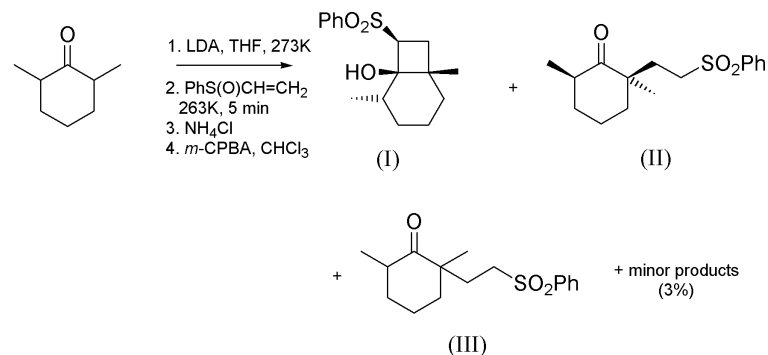
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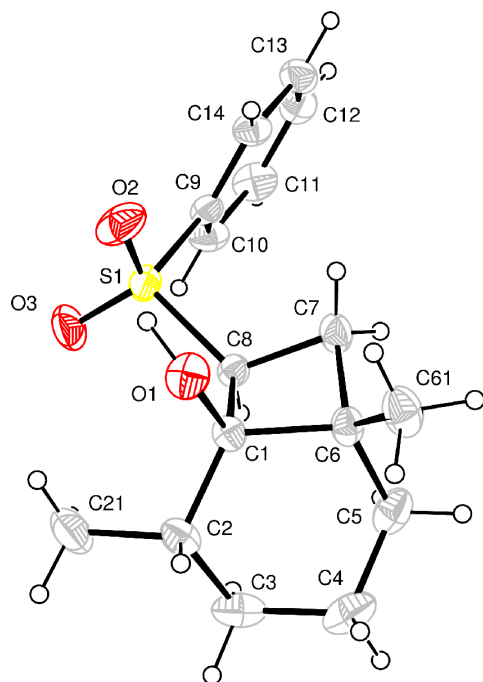
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## Comment

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]alkan-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*.

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