

Xiao-Hong Ren,<sup>a</sup> Li-Li Sun,<sup>b\*</sup>  
Yu-Ping Guo,<sup>b</sup> Fei Wang<sup>b</sup> and  
Zuo-Jun Qi<sup>b</sup><sup>a</sup>College of Pharmaceuticals & Biotechnology,  
Tianjin University, Tianjin 300072, People's  
Republic of China, and <sup>b</sup>Department of  
Chemistry, Tianjin University, Tianjin 300072,  
People's Republic of ChinaCorrespondence e-mail:  
sunlilichemistry@hotmail.com

## Key indicators

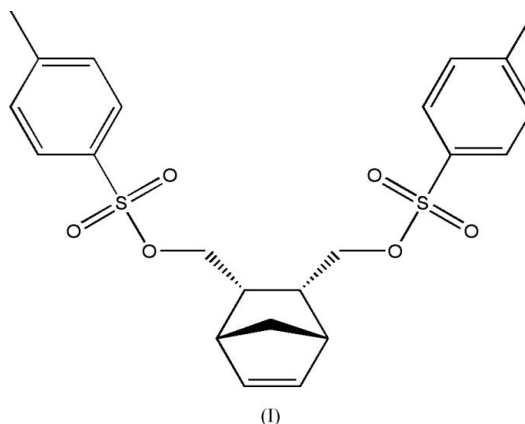
Single-crystal X-ray study  
 $T = 294\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$   
 $R$  factor = 0.045  
 $wR$  factor = 0.130  
Data-to-parameter ratio = 16.6For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.*endo*-Bicyclo[2.2.1]hept-2-ene-5,6-dimethylene  
bis(*p*-toluenesulfonate)

The structure of the title compound,  $\text{C}_{23}\text{H}_{26}\text{O}_6\text{S}_2$ , contains three rings in a bicyclo[2.2.1] system (two five-membered rings and one six-membered ring). There are four  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds (three intramolecular hydrogen bonds and one intermolecular hydrogen bond) in the structure.

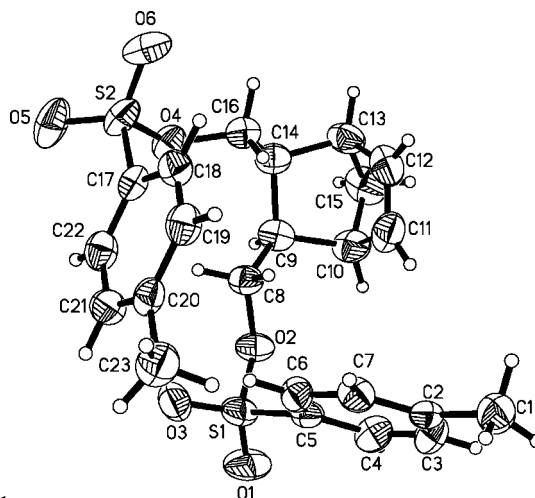
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## Comment

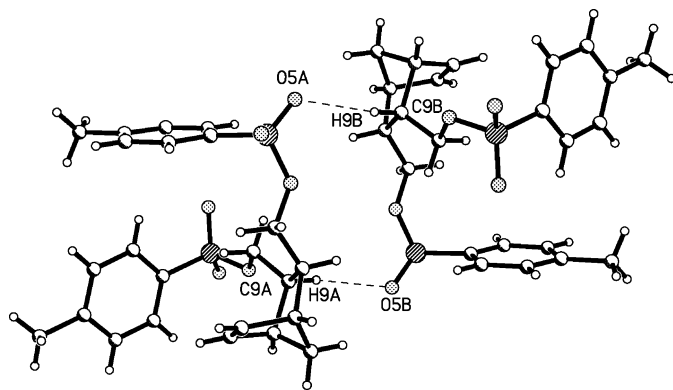
The structure determination of the title compound, (I), showed that, in the bicyclo[2.2.1] ring system, both five-membered rings have envelope conformations (Table 1), atom C15 being the out-of-plane atom in both rings (C10/C11/C12/C13/C15 and C10/C9/C14/C13/C15). The six-membered ring



(C9–C14) displays a boat conformation. There are four  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds in (I), one of them being intermolecular (Table 2).



**Figure 1**  
The molecular structure of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 35% probability level.



**Figure 2**  
Intermolecular hydrogen bonds in (I), shown as dashed lines.

## Experimental

5,6-Bis(*endo*-hydroxymethyl)bicyclo[2.2.1]hept-2-ene (2 g, 13 mmol) was added to NaH (1.78 g, 74 mmol) in 100 ml of dry tetrahydrofuran. The mixture was stirred at 273 K for 10 min. *p*-Toluenesulfonyl chloride (5.45 g, 28.6 mmol) was then added. The mixture was stirred at 273 K for another 6 h. The reaction mixture was poured into ice and extracted with dichloromethane. The extract was washed with water, dried over anhydrous sodium sulfate and evaporated *in vacuo* to give a solid. Colourless prisms of (I) were obtained after recrystallization from a methanol solution of the solid (yield: 3 g, 50 wt%; m.p. 359 K). IR (CHCl<sub>3</sub>, cm<sup>-1</sup>): 1600, 1361, 1173.

### Crystal data

C <sub>23</sub> H <sub>26</sub> O <sub>6</sub> S <sub>2</sub>	Z = 4
M <sub>r</sub> = 462.56	D <sub>x</sub> = 1.339 Mg m <sup>-3</sup>
Monoclinic, P2 <sub>1</sub> /c	Mo Kα radiation
a = 14.139 (2) Å	μ = 0.27 mm <sup>-1</sup>
b = 13.186 (2) Å	T = 294 (2) K
c = 13.626 (2) Å	Prism, colourless
β = 115.424 (3)°	0.24 × 0.20 × 0.14 mm
V = 2294.4 (6) Å <sup>3</sup>	

### Data collection

Siemens SMART CCD diffractometer	12673 measured reflections
φ and ω scans	4679 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	2574 reflections with I > 2σ(I)
T <sub>min</sub> = 0.927, T <sub>max</sub> = 0.963	R <sub>int</sub> = 0.040
	θ <sub>max</sub> = 26.4°

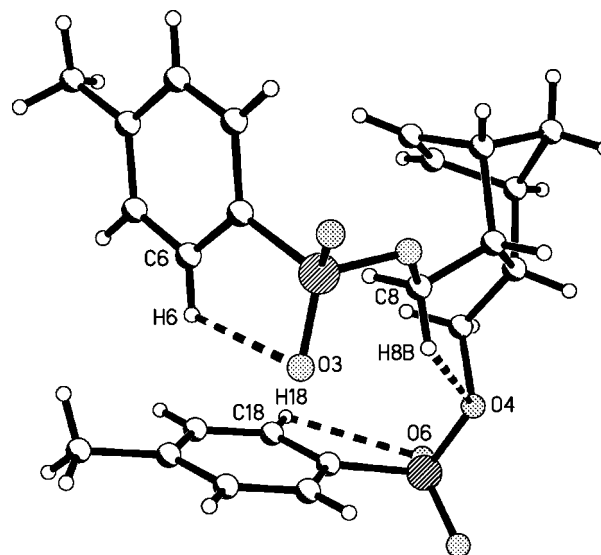
### Refinement

Refinement on F <sup>2</sup>	w = 1/[σ <sup>2</sup> (F <sub>o</sub> <sup>2</sup> ) + (0.0547P) <sup>2</sup> + 0.5608P]
R[F <sup>2</sup> > 2σ(F <sup>2</sup> )] = 0.045	where P = (F <sub>o</sub> <sup>2</sup> + 2F <sub>c</sub> <sup>2</sup> )/3
wR(F <sup>2</sup> ) = 0.130	(Δ/σ) <sub>max</sub> = 0.002
S = 0.99	Δρ <sub>max</sub> = 0.20 e Å <sup>-3</sup>
4679 reflections	Δρ <sub>min</sub> = -0.32 e Å <sup>-3</sup>
282 parameters	
H-atom parameters constrained	

**Table 1**

Selected torsion angles (°).

C10—C11—C12—C13	1.6 (3)	C10—C9—C14—C13	2.4 (3)
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**Figure 3**  
The intramolecular hydrogen bonds of (I), shown as dashed lines.

**Table 2**

Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O3	0.93	2.54	2.915 (3)	104
C8—H8B···O4	0.97	2.57	3.113 (3)	115
C9—H9···O5 <sup>i</sup>	0.98	2.56	3.529 (3)	169
C18—H18···O6	0.93	2.56	2.930 (3)	104

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

All H atoms were observable in a difference Fourier map, although the methyl H atoms were the least clear. Nevertheless, all H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 Å and U<sub>iso</sub>(H) = 1.2U<sub>eq</sub>(C) for aryl H atoms, C—H = 0.98 Å and U<sub>iso</sub>(H) = 1.2U<sub>eq</sub>(C) for tertiary H atoms, C—H = 0.97 Å and U<sub>iso</sub>(H) = 1.2U<sub>eq</sub>(C) for methylene H atoms, and C—H = 0.96 Å and U<sub>iso</sub>(H) = 1.5U<sub>eq</sub>(C) for methyl H atoms.

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); 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, 1997); software used to prepare material for publication: SHELXTL.

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## References

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