

Wen Pei,* Yueshui Shao and Li Sun

College of Chemical Engineering, Zhejiang University of Technology, Hangzhou 310014, People's Republic of China

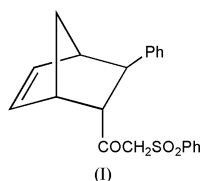
Correspondence e-mail:
pei_wen58@hotmail.com

Key indicators

Single-crystal X-ray study
 $T = 297\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$
 R factor = 0.039
 wR factor = 0.103
Data-to-parameter ratio = 10.9For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.(5*SR*,6*RS*)-6-Phenyl-5-(phenylsulfonylacetyl)-bicyclo[2.2.1]hept-2-eneThe title compound, $\text{C}_{21}\text{H}_{20}\text{O}_3\text{S}$, was obtained from the Diels–Alder reaction of 4-phenyl-1-phenylsulfonyl-3-buten-2-one and cyclopentadiene, catalyzed by a titanium reagent. This molecule is an *endo*-cycloadduct isomer.

Comment

As some sulfonyl-functionalized chelating enones have demonstrated effective diastereoselectivity as prochiral electrophilic substrates in catalyzed asymmetric carbon-bond formation, a series of new cycloadducts has been synthesized in our laboratory in order to investigate the mechanism of the asymmetric Diels–Alder reaction (Pei, 1998). The structure of the title compound, (I), is reported here as an early result in our study of this new series of compounds.



The molecular structure of (I) is shown in Fig. 1. It has been revealed that the product obtained by the Diels–Alder reaction is racemic, although a chiral butanediol derivative was used to form a chiral titanium catalyst.

Experimental

Under the protection of a nitrogen atmosphere, 4A molecular sieve (200 mg) and (2*R*,3*R*)-(–)-1,1,4,4-tetra-(1-naphthyl)-2,3-acetone-1,4-butanediol (73 mg, 0.11 mmol) were added to a 25 ml flask with stirring. $\text{TiCl}_2(\text{O}^i\text{Pr})_2$ (1.118 *M*, 0.1 ml) was then quickly added dropwise with stirring. After 1 h, 4-phenyl-1-phenylsulfonyl-3-buten-2-one (0.143 g) was dissolved in CH_2Cl_2 (2 ml) and added dropwise to the solution, and 5 min later cyclopentadiene (5 mmol, 0.5 ml) was added. The mixture was stirred for 24 h at room temperature and the reaction was then quenched by adding H_2O dropwise. Subsequent extraction, drying, filtration, concentration and column chromatography gave the title product (yield 82%). Crystals of (I) suitable for X-ray analysis were obtained from an *n*-hexane–ethyl acetate (2:1 *v/v*) solution by slow evaporation.

Crystal data

 $\text{C}_{21}\text{H}_{20}\text{O}_3\text{S}$
 $M_r = 352.45$
Triclinic, $P\bar{1}$
 $a = 8.6363(3)\text{ \AA}$
 $b = 9.6144(3)\text{ \AA}$
 $c = 12.0887(4)\text{ \AA}$
 $\alpha = 107.319(1)^\circ$
 $\beta = 104.509(1)^\circ$
 $\gamma = 104.8535(9)^\circ$
 $V = 866.19(5)\text{ \AA}^3$ $Z = 2$
 $D_x = 1.351\text{ Mg m}^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 3777 reflections
 $\theta = 3.4\text{--}27.5^\circ$
 $\mu = 0.20\text{ mm}^{-1}$
 $T = 297.1\text{ K}$
Chunk, colourless
 $0.40 \times 0.35 \times 0.28\text{ mm}$

Data collection

Rigaku RAXIS-RAPID
diffractometer
 ω scans
Absorption correction: multi-scan
(*ABSCOR*; Higashi, 1995)
 $T_{\min} = 0.857$, $T_{\max} = 0.945$
16 666 measured reflections

3939 independent reflections
2686 reflections with $F^2 > 2\sigma(F^2)$
 $R_{\text{int}} = 0.014$
 $\theta_{\max} = 27.5^\circ$
 $h = -11 \rightarrow 11$
 $k = -12 \rightarrow 12$
 $l = -15 \rightarrow 13$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.103$
 $S = 1.00$
2689 reflections
246 parameters

H-atom parameters constrained
 $w = (4F_o^2)/[0.0016F_o^2 + 3\sigma(F_o^2)]$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.22 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.47 \text{ e } \text{\AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

| | | | |
|---------------|------------|-------------|------------|
| S1—O2 | 1.435 (2) | C3—C9 | 1.560 (3) |
| S1—O3 | 1.426 (1) | C4—C5 | 1.505 (3) |
| S1—C1 | 1.781 (2) | C4—C8 | 1.537 (2) |
| S1—C16 | 1.761 (2) | C5—C6 | 1.320 (3) |
| O1—C2 | 1.203 (2) | C6—C7 | 1.506 (3) |
| C1—C2 | 1.526 (3) | C7—C8 | 1.533 (3) |
| C2—C3 | 1.501 (2) | C7—C9 | 1.555 (2) |
| C3—C4 | 1.572 (2) | C9—C10 | 1.516 (3) |
| C16—S1—C1—C2 | 65.7 (1) | O1—C2—C3—C4 | 120.2 (2) |
| C1—S1—C16—C17 | −81.7 (1) | O1—C2—C3—C9 | 3.0 (3) |
| C1—S1—C16—C21 | 100.7 (1) | C1—C2—C3—C4 | −57.0 (2) |
| S1—C1—C2—O1 | 67.4 (2) | C1—C2—C3—C9 | −174.2 (2) |
| S1—C1—C2—C3 | −115.3 (2) | | |

H atoms were treated using a riding model, with C—H distances of 0.93 \AA and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 2003); program(s) used to solve structure: *SIR92* (Altomare *et al.*,

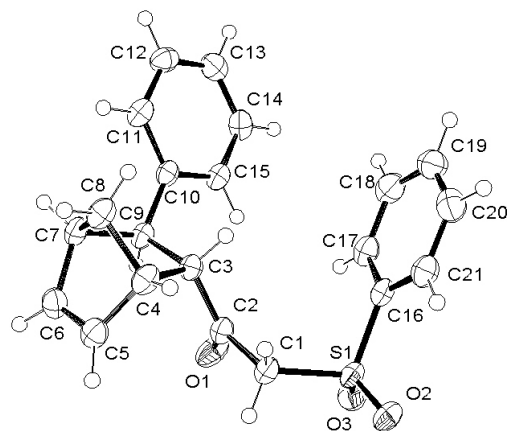


Figure 1

The molecular structure of (I), with 50% probability displacement ellipsoids and the atom-numbering scheme.

1994); program(s) used to refine structure: *CRYSTALS* (Watkin *et al.*, 1996); molecular graphics: *ORTEP-3* (Version 1.06; Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

References

- Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Pei, W. (1998). *Chem. J. Chin. Univ.* **19**, 402–405.
Rigaku (1998). *RAPID-AUTO*. Version 1.06. Rigaku Corporation, 3-9-12 Akishima, Tokyo 196-8666, Japan.
Rigaku/MS (2003). *CrystalStructure*. Version 3.5.1. Rigaku/MS, 9009 New Trails Drive, The Woodlands, TX 77381-520, USA.
Watkin, D. J., Prout, C. K., Carruthers, J. R. & Betteridge, P. W. (1996). *CRYSTALS*. Issue 10. Chemical Crystallography Laboratory, University of Oxford, England.