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(5*R*,6*S*)-6-Methyl-5-(phenylsulfonylacetyl)-bicyclo[2.2.1]hept-2-ene

Wen Pei,[a]* Yongjiang Wang,[a,b] Mengzhan Sun,[a] Xiaobo Xiong[a] and Changquan Yu[a]

[a]College of Chemical Engineering and Materials Science, Zhejiang University of Technology, Hangzhou, 310014, People's Republic of China, and [b]College of Biological and Chemical Engineering, Zhejiang University of Science and Technology, Hangzhou, 310023, People's Republic of China

Correspondence e-mail:
pei_wen58@hotmail.com

Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.004 Å
R factor = 0.045
wR factor = 0.138
Data-to-parameter ratio = 16.8

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

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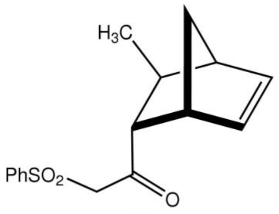
The title compound, $C_{16}H_{18}O_3S$, an *endo*-cycloadduct isomer, was obtained from the Diels–Alder reaction of (*E*)-1-(phenylsulfonyl)pent-3-en-2-one with cyclopentadiene catalysed by a chiral titanium reagent. The crystal structure confirms the absolute streochemistry.

Comment

Cycloadducts of asymmetric Diels–Alder reactions have attracted attention owing to their utility in the synthesis of natural compounds (Corey, 2002). Some sulfonyl-functionalized chelating enones have demonstrated effective stereoselectivity as prochiral electrophilic substrates in catalysed asymmetric-carbon-bond formations. Hence, 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 molecular structure of (I) confirms the absolute stereochemistry.



(I)

Experimental

The title compound was prepared according to the procedure of Pei (1998). Under the protection of nitrogen, a 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 25ml flask with stirring. $TiCl_2(O^iPr)_2$ (0.1 ml, 1.118 mol l^{-1}) was then quickly added dropwise to this solution with stirring. After 1h, (*E*)-1-(phenylsulfonyl)pent-3-en-2-one (0.112 g, 0.5 mmol) dissolved in CH_2Cl_2 (2 ml) was added to the solution and 5 min later, cyclopentadiene (0.5 ml, 5 mmol) was added. After stirring for 24 h at room temperature, the reaction was halted by the addition of H_2O. Extraction, drying, filtration, concentration and column chromography gave the title product in 92% yield. Diffraction-quality crystals were obtained by the slow evaporation of an ethanol solution at room temperature.

Crystal data

$C_{16}H_{18}O_3S$
M_r = 290.38
Monoclinic, $P2_1$
a = 11.2342 (3) Å
b = 5.8141 (1) Å
c = 12.3584 (3) Å
β = 115.5233 (8)°
V = 728.44 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 296 (1) K
0.28 × 0.10 × 0.04 mm

Data collection

Rigaku RAXIS-RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{\min} = 0.917$, $T_{\max} = 0.991$
7013 measured reflections
3074 independent reflections
2761 reflections with $F^2 > 2.0\sigma(F^2)$
$R_{\rm int} = 0.046$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.138$
$S = 1.00$
3074 reflections
183 parameters
H-atom parameters constrained
$\Delta\rho_{\max} = 0.27$ e Å^{-3}
$\Delta\rho_{\min} = -0.21$ e Å^{-3}
Absolute structure: Flack (1983), 1251 Friedel Pairs
Flack parameter: −0.067 (2)

All H atoms were placed in calculated positions with C—H(aromatic, ethylenic) = 0.93 Å, C—H(methine) = 0.97 Å, C—H(methenyl) = 0.98 Å and C—H(methyl) = 0.96 Å. All H atoms were included in the final cycles of refinement in the riding model, with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(carrier atom).

Data collection: *PROCESS-AUTO* (Rigaku,1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al*., 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

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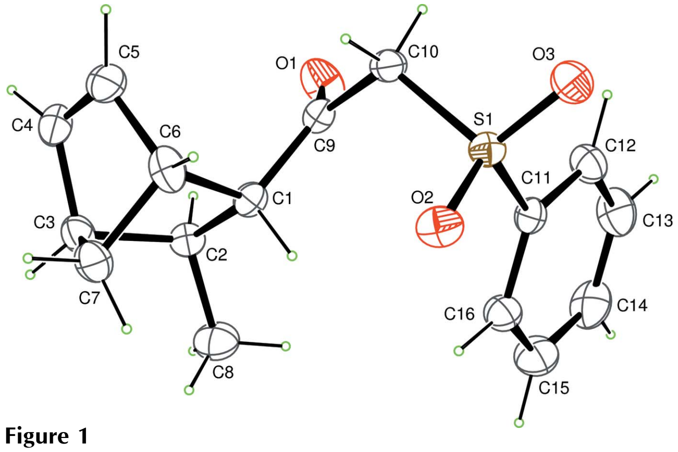

Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

References

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.

Corey, E. J. (2002). *Angew. Chem. Int. Ed.* **41**, 1650–1667.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.

Pei, W. (1998). *Chem. J. Chin. Univ.* **19**, 402–404.

Rigaku (1998). *PROCESS-AUTO*. Rigaku Corporation, Tokyo, Japan.

Rigaku/MSC (2004). *CrystalStructure*. Version 3.7.0. Rigaku/MSC, The Woodlands. Texas, USA.

Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.

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