Phenylsulfonyl Ene-Allenenes as Efficient Precursors to Bicyclic Systems *via* Intramolecular [2+2]-Cycloaddition Reactions

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Supporting Information Available: ORTEP drawing and X-ray data for structure **25b**. The authors have deposited atomic coordinates for these structures with the Cambridge Crystallographic Data Centre. This material is available free of charge *via* the Internet at <http://pubs.acs.org>.

General Experimental Section Paragraph

Melting points are uncorrected. Mass spectra were determined at an ionizing voltage of 70eV. Unless otherwise noted, all reactions were performed in flame dried glassware under an atmosphere of dry argon. Solutions were evaporated under reduced pressure with a rotary evaporator and the residue was chromatographed on a silica gel column using a 5% ethyl acetate/hexane mixture as the eluent unless specified otherwise. All solids were recrystallized from 3% ethyl acetate/hexane for analytical data.

Table of Contents for Supporting Information

S2	Ortep Drawing for Compound 25b
S3	Table of Summary Data for X-ray of 25b
S4	Atom Coordinates for 25b
S5	Temperature Factors for 25b
S6	Hydrogen Atom Coordinates for 25b
S7	Bond Lengths and angles for 25b

Ortep of 2-Phenylsulfonylmethylene-7*a*-methyl-2,2*a*,7,7*a*-tetrahydro-1*H*-cyclobuta[*a*]indene (**25b**).

Table Summary of Crystal Data, Intensity Collection, and Structure Refinement for **25b**.

formula	C ₁₉ H ₁₈ O ₂ S
mol. wt.	310.39
<i>a</i> , Å	160.33(3)
<i>b</i> , Å	9.020(2)
<i>c</i> , Å	23.092(5)
α , deg	90.00(0)
β , deg	102.95(3)
γ , deg	90.00(0)
vol, Å ³	32092(12)
crystal system	Monoclinic
space group	C2/c
d_{calcd} , g/cm ³	1.267
<i>Z</i>	8
Temp, °C	25
diffractometer	Siemens P4
radiation	Mo K α (10.71073Å)
abs. coeff., μ , mm ⁻¹	0.203
crystal size, mm	0.31 x 0.28 x 0.17
data collected	-1 $\leq h \leq 13$, -1 $\leq k \leq 9$, -22 $\leq l \leq 24$, (2° $\leq 2\theta \leq 35^\circ$)
structure solution and refinement	SHELXS-86 and SHELXL-93 (Sheldrick, 1993)
reflections collected	1632
total data (<i>R</i> _{int} %)	1105 (0.0382)
unique data (<i>I</i> > 2 <i>s</i> (<i>I</i>))	1105
<i>F</i> (000)	1312
no. of variables	199
goodness of fit	1.086
Final <i>R</i> indices [<i>I</i> > 2 <i>s</i> (<i>I</i>)]	<i>R</i> 1 = 0.0477, <i>wR</i> 2 = 0.0926
<i>R</i> indices (all data)	<i>R</i> 1 = 0.0887, <i>wR</i> 2 = 0.1099
Extinction coefficient	0.0052(7)
max and min difference peaks, e/Å ³	0.160 and -0.178

Table 1. Atom Coordinates ($\times 10^4$) for **25b**.

atom	x	y	z	U _{equiv}
S(1)	6597(1)	919(2)	6112(1)	51(1)
O(1)	6979(3)	2149(5)	5860(2)	62(2)
O(2)	5917(3)	1230(5)	6410(2)	64(2)
C(1)	4990(6)	2469(11)	3404(3)	60(2)
C(2)	4671(5)	3675(11)	3656(5)	68(2)
C(3)	4997(5)	4031(9)	4248(5)	68(2)
C(4)	5633(5)	3163(10)	4589(3)	55(2)
C(5)	5944(4)	1962(9)	4337(4)	40(2)
C(6)	5635(5)	1618(9)	3747(4)	48(2)
C(7)	6092(4)	296(8)	3563(3)	56(2)
C(8)	6609(4)	-330(8)	4147(3)	47(2)
C(9)	6624(4)	886(8)	4632(3)	38(2)
C(10)	6294(4)	-263(7)	5008(3)	40(2)
C(11)	6097(4)	-1348(7)	4493(3)	49(2)
C(12)	7482(4)	-929(8)	4114(3)	73(2)
C(13)	6241(4)	-411(7)	5568(3)	43(2)
C(14)	7403(5)	-11(8)	6623(3)	44(2)
C(15)	8221(7)	-140(9)	6514(3)	65(2)
C(16)	8828(5)	-931(11)	6902(5)	83(3)
C(17)	8658(6)	-1587(9)	7388(4)	74(3)
C(18)	7856(8)	-1448(10)	7493(3)	76(3)
C(19)	7216(5)	-688(9)	7112(4)	64(2)

* Equivalent isotropic U defined as one third of the trace of the orthogonalised U_{ij} tensor.

Table 2. Temperature factors ($\text{\AA}^2 \times 10^3$) for **25b**.

atom	U11	U22	U33	U23	U13	U12
S(1)	56(1)	52(1)	41(1)	-5(1)	5(1)	-2(1)
O(1)	80(4)	46(3)	55(3)	11(3)	6(3)	-14(3)
O(2)	54(3)	85(4)	56(3)	-18(3)	18(3)	7(3)
C(1)	61(6)	66(6)	55(6)	6(6)	15(6)	-1(6)
C(2)	63(6)	75(8)	71(7)	33(6)	26(6)	20(6)
C(3)	81(7)	50(6)	82(7)	14(6)	35(6)	28(6)
C(4)	66(6)	47(6)	55(6)	10(5)	21(5)	11(5)
C(5)	37(5)	39(6)	49(7)	5(5)	20(5)	12(5)
C(6)	53(6)	56(6)	35(6)	9(5)	10(5)	-1(6)
C(7)	68(5)	55(6)	50(5)	-7(5)	22(5)	9(5)
C(8)	51(5)	50(5)	37(5)	-5(4)	6(4)	17(5)
C(9)	40(5)	40(5)	39(4)	0(5)	17(4)	-2(5)
C(10)	41(5)	38(5)	39(5)	-12(5)	7(4)	2(4)
C(11)	58(5)	44(5)	48(5)	12(5)	15(4)	-7(4)
C(12)	78(6)	63(6)	80(6)	-14(5)	23(5)	12(5)
C(13)	44(5)	44(5)	37(4)	-5(4)	4(4)	-10(4)
C(14)	43(6)	49(5)	36(5)	4(4)	1(5)	-8(4)
C(15)	61(7)	70(6)	58(6)	26(5)	3(6)	-12(6)
C(16)	51(6)	96(7)	95(7)	49(6)	1(6)	0(6)
C(17)	75(8)	58(6)	75(8)	15(6)	-13(6)	-2(6)
C(18)	116(8)	76(8)	36(6)	21(5)	14(7)	14(7)
C(19)	75(7)	72(6)	46(5)	5(5)	18(6)	2(6)

The anisotropic temperature factor exponent takes the form:

$$-2p^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2hka^{*}b^{*}U_{12} + 2hla^{*}c^{*}U_{13} + 2klb^{*}c^{*}U_{23}).$$

Table 3 Hydrogen Atom Coordinates ($\times 10^4$) and temperature factors ($\text{\AA}^2 \times 10^3$) for **25b**.

atom	x	y	z	U ₁₁
H(1B)	4770(6)	2239(11)	2992(3)	80
H(2B)	4214(5)	4252(11)	3422(5)	80
H(3A)	4787(5)	4891(9)	4416(5)	80
H(4A)	5848(5)	3387(10)	5002(3)	80
H(7A)	6457(4)	589(8)	3305(3)	80
H(7B)	5690(4)	-428(8)	3364(3)	80
H(9A)	7172(4)	1305(8)	4809(3)	80
H(11A)	6340(4)	-2314(7)	4588(3)	80
H(11B)	5499(4)	-1412(7)	4306(3)	80
H(12A)	7755(4)	-234(8)	3902(3)	80
H(12B)	7828(4)	-1065(8)	4508(3)	80
H(12C)	7415(4)	-1862(8)	3909(3)	80
H(13A)	5989(4)	-1303(7)	5679(3)	80
H(15A)	8349(7)	348(9)	6175(3)	80
H(16A)	9387(5)	-1045(11)	6821(5)	80
H(17A)	9092(6)	-2139(9)	7656(4)	80
H(18A)	7741(8)	-1880(10)	7848(3)	80
H(19A)	6645(5)	-651(9)	7176(4)	80

Table 4. Bond lengths (Å) and angles (deg) for **25b**.

S(1)-O(2)	1.441(4)	O(1)-S(1)-C(14)	108.8(4)
S(1)-O(1)	1.451(4)	C(13)-S(1)-C(14)	103.3(3)
S(1)-C(13)	1.738(6)	C(2)-C(1)-C(6)	119.5(7)
S(1)-C(14)	1.756(7)	C(1)-C(2)-C(3)	120.2(8)
C(1)-C(2)	1.385(9)	C(4)-C(3)-C(2)	119.7(7)
C(1)-C(6)	1.387(9)	C(5)-C(4)-C(3)	119.8(7)
C(2)-C(3)	1.388(9)	C(4)-C(5)-C(6)	120.8(7)
C(3)-C(4)	1.383(9)	C(4)-C(5)-C(9)	127.7(8)
C(4)-C(5)	1.374(8)	C(6)-C(5)-C(9)	111.5(7)
C(5)-C(6)	1.376(8)	C(5)-C(6)-C(1)	119.9(7)
C(5)-C(9)	1.504(8)	C(5)-C(6)-C(7)	111.6(8)
C(6)-C(7)	1.510(9)	C(1)-C(6)-C(7)	128.5(8)
C(7)-C(8)	1.525(7)	C(6)-C(7)-C(8)	104.1(6)
C(8)-C(12)	1.518(8)	C(12)-C(8)-C(7)	114.3(6)
C(8)-C(9)	1.564(8)	C(12)-C(8)-C(9)	114.8(6)
C(8)-C(11)	1.566(8)	C(7)-C(8)-C(9)	107.0(5)
C(9)-C(10)	1.521(8)	C(12)-C(8)-C(11)	114.3(6)
C(10)-C(13)	1.322(7)	C(7)-C(8)-C(11)	115.2(5)
C(10)-C(11)	1.518(8)	C(9)-C(8)-C(11)	88.4(4)
C(14)-C(19)	1.373(8)	C(5)-C(9)-C(10)	112.9(5)
C(14)-C(15)	1.396(8)	C(5)-C(9)-C(8)	103.6(5)
C(15)-C(16)	1.366(9)	C(10)-C(9)-C(8)	88.8(5)
C(16)-C(17)	1.349(9)	C(13)-C(10)-C(11)	130.8(6)
C(17)-C(18)	1.368(10)	C(13)-C(10)-C(9)	137.3(6)
C(18)-C(19)	1.375(9)	C(11)-C(10)-C(9)	91.7(5)
O(2)-S(1)-O(1)	118.4(3)	C(10)-C(11)-C(8)	88.9(5)
O(2)-S(1)-C(13)	108.5(3)	C(10)-C(13)-S(1)	123.7(5)
O(1)-S(1)-C(13)	109.8(3)	C(19)-C(14)-C(15)	119.8(7)
O(2)-S(1)-C(14)	107.0(4)	C(19)-C(14)-S(1)	120.2(8)
		C(15)-C(14)-S(1)	119.9(7)
		C(16)-C(15)-C(14)	119.1(7)
		C(17)-C(16)-C(15)	121.8(8)

C(16)-C(17)-C(18)	118.8(8)
C(17)-C(18)-C(19)	121.8(8)
C(14)-C(19)-C(18)	118.7(7)