A Novel Synthesis of (β-Organochalcogenyl)allyl Phenyl Sulfoxides via Regioselective Hydrochalcogenation Reaction of 1,2-Allenyl Sulfoxides

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Abstract: (β-Organochalcogenyl)allyl phenyl sulfoxides **2** were prepared by treatment of allenyl sulfoxides **1** with sodium organyl chalcogenolates in good yields. The reaction was regioselective, giving exclusively one isomer in all cases. The applications of (β-phenyltelluro)allyl phenyl sulfoxide **2d** and (β-organosulfanyl)allyl phenyl sulfoxide **2m** in organic synthesis were also described.

Key words: allenyl sulfoxide, hydrochalcogenation, regioselectivity

Allyl sulfoxides are versatile and valuable building blocks in organic synthesis, as shown by their remarkable utility in the synthesis of a variety of functionalized compounds[1] and natural products.[2] β-Functionalized allyl sulfoxides[3] are also class of compounds with significant synthetic potential. Hence, efficient and convenient methods for the synthesis of such compounds are still in demand and their goal is a challenge.

In the last few decades, there has been remarkable interest in the synthesis of vinylic chalcogenides and their synthetic applications.[4] Considering the well-known chemical reactivities of vinylic chalcogenides, β-organochalcogenyl substituted allyl sulfoxides, which combine the unit of vinylic chalcogenides and allyl sulfoxides into one molecule, will be intermediates of great synthetic potential. Recently, we[5] and others[6–8] have reported the regio- and stereoselective hydrochalcogenation reaction of electron-deficient carbon–carbon triple bond to prepare several functionalized vinylic chalcogenides. Another class of compounds that attracted our interest is the electron-deficient 1,2-dienes, which have been found to have high and unique reactivity.[9,10] Usually, one of the two carbon–carbon double bonds can be selectively reacted to give vinylic products via delicately tuning the steric and electronic factors. Thus, hydrochalcogenation of electron-deficient allene would be one of the most efficient ways to prepare β-organochalcogenyl substituted allyl compounds. As a part of ongoing study of this area, in this paper we wish to disclose our recent results on the corresponding hydrochalcogenation reaction of allenyl sulfoxides.

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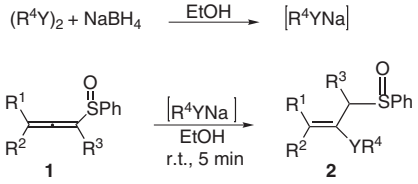
We started our investigations with the hydrotelluration reaction of 1,2-allenyl sulfoxides **1a**. When allenyl sulfoxide **1a** was added to an ethanol solution of sodium phenyltellurolate, prepared by reduction of diphenyl ditelluride with sodium borohydride at room temperature, to our delight, the (β-phenyltelluro)allyl phenyl sulfoxide **2a** was formed in 80% yield with total regioselectivity (Scheme 1, Table 1, entry 1). Then, a series of allenyl sulfoxides[11] were prepared to study such a reaction. As shown in Table 1, the substrates can be mono- (entry 1), di- (entry 2) and trisubstituted (entries 3, 4) allenyl sulfoxides. With these results in mind, the similar procedure was used for the synthesis of (β-organoselanyl) and (β-organosulfanyl)allyl phenyl sulfoxides (Scheme 1).



Scheme 1

The results are summarized in Table 1 (entries 5–16). These reactions showed exclusive regioselectivity, and only one regioisomer (**2e**–**p**) was obtained. For the mono-, di-, or trisubstituted allenyl sulfoxides, yields of (β-organoselanyl) (entries 5–12) and (β-organosulfanyl)allyl (entries 13–16) phenyl sulfoxides were very high.

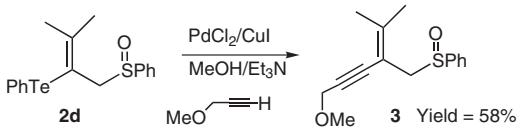
The mechanism for the current regioselective hydrochalcogenation reaction is believed to be a nucleophilic conjugate addition of RY^- to allenyl sulfoxides at the β-position to afford an allyl carbanion stabilized by the sulfoxide group.

(β-Organochalcogenyl)allyl phenyl sulfoxides **2** are useful intermediates and undergo many transformations in organic chemistry. For example, when compound **2d** was treated with alkyne using $PdCl_2$/CuI as the catalyst, the β-alkynyl allyl sulfoxide **3** was obtained in moderate yields (Scheme 2). And compound **2m** can be easily oxidized to 2,3-bis(phenylsulfonyl)prop-1-ene (**4**)[12] by MCPBA in CH_2Cl_2 at room temperature (Scheme 3). Compound **3** has many valuable transformation in organic synthesis, such as α-alkylation reaction,[1b,13] 1,3-rearrangement,[14] 1,3-dipolar cycloaddition,[15] etc.[16]

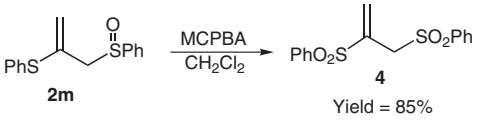
Table 1 (β-Organochalcogenyl)allyl Phenyl Sulfoxides Prepared

Entry	R^1	R^2	R^3	R^4Y	Yields (%)[a]	Products
1	H	H	H	C_6H_5Te	80	**2a**
2	H	H	C_6H_5	C_6H_5Te	79	**2b**
3	-$(CH_2)_5$-		H	C_6H_5Te	81	**2c**
4	CH_3	CH_3	H	C_6H_5Te	75	**2d**
5	H	H	H	C_6H_5Se	83	**2e**
6	H	H	H	*n*-C_4H_9Se	82	**2f**
7	H	H	C_6H_5	C_6H_5Se	81	**2g**
8	H	H	C_6H_5	*n*-C_4H_9Se	80	**2h**
9	-$(CH_2)_5$-		H	C_6H_5Se	84	**2i**
10	-$(CH_2)_5$-		H	*n*-C_4H_9Se	82	**2j**
11	CH_3	CH_3	H	C_6H_5Se	83	**2k**
12	CH_3	CH_3	H	*n*-C_4H_9Se	83	**2l**
13	H	H	H	C_6H_5S	84	**2m**
14	H	H	C_6H_5	C_6H_5S	79	**2n**
15	-$(CH_2)_5$-		H	C_6H_5S	80	**2o**
16	CH_3	CH_3	H	C_6H_5S	81	**2p**

[a] Isolated yields based on the allenyl sulfoxides.



Scheme 2



Scheme 3

In summary, we have disclosed here the high efficient regioselective hydrohalcogenation of allenyl sulfoxides to afford synthetically important (β-organochalcogenyl)allyl phenyl sulfoxides. The application of (β-phenyltelluro)allyl phenyl sulfoxide **2d** and (β-organosulfanyl)allyl phenyl sulfoxide **2m** in organic synthesis has also been described.

All ^1H and ^{13}C NMR spectra were measured in $CDCl_3$ and recorded on Bruker Avance-400 (400 MHz) spectrometer with TMS as the internal standard. Chemical shifts are expressed in ppm and *J* values are given in Hz. IR spectra were run on a Bruker vector 22 spectrometer. EIMS were determined with a HP5989B mass spectrometer. Elemental analyses were performed on an EA-1110 instrument.

(β-Organochalcogenyl)allyl Phenyl Sulfoxides 2a–p; General Procedure

A solution of allenyl sulfoxide **1** (0.5 mmol) in EtOH (2 mL) was added dropwise to a solution of RYNa, prepared in situ by reduction of $(R^4Y)_2$ (0.25 mmol) with $NaBH_4$ (0.6 mmol) in EtOH (2 mL) at r.t. under N_2. After completion of the reaction (5 min), the mixture was diluted with brine (10 mL) and extracted with EtOAc (3 × 15 mL). The combined organic layers were dried ($MgSO_4$) and filtered. Removal of the solvent in vacuo, and purification of the residue by silica gel chromatography with *n*-hexane–EtOAc (5:1) as eluent gave the product **2**.

2a

Yellow oil.

IR (neat): 1610, 1040 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 7.86–7.82 (m, 2 H), 7.54–7.48 (m, 6 H), 7.37–7.24 (m, 2 H), 6.06 (s, 1 H), 5.71 (s, 1 H), 3.82 (d, *J* = 13.2 Hz, 1 H), 3.49 (d, *J* = 8.8 Hz, 1 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 142.9, 139.9, 131.6, 131.2, 129.7, 129.4, 129.0, 124.3, 115.1, 113.2, 69.6.

MS (EI): *m*/*z* = 373 [M^+ + 1].

Anal. Calcd for $C_{15}H_{14}OSTe$: C, 48.70; H, 3.81. Found: C, 48.64; H, 3.93.

2b

Yellow oil.

IR (neat): 1625, 1040 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 7.82–7.80 (m, 2 H), 7.39–7.31 (m, 10 H), 7.28–7.20 (m, 3 H), 4.2 (s, 2 H), 2.1 (s, 1 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 143.2, 141.8, 140.0, 138.7, 135.2, 129.4, 128.5, 128.4, 128.2, 127.7, 124.1, 111.4, 70.0.

MS (EI): *m*/*z* = 449 [M^+ + 1].

Anal. Calcd for $C_{21}H_{18}OSTe$: C, 56.55; H, 4.07. Found: C, 56.63; H, 3.98.

2c
Yellow oil.

IR (neat): 1620, 1034 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 7.72–7.70 (m, 2 H), 7.43–7.40 (m, 5 H), 7.30–7.29 (m, 1 H), 7.24–7.20 (m, 2 H), 4.25 (d, *J* = 12.8 Hz, 1 H), 3.79 (d, *J* = 13.2 Hz, 1 H), 2.44–240 (m, 2 H), 2.39–2.36 (m, 1 H), 2.15–2.10 (m, 1 H), 1.51–1.49 (m, 5 H), 1.46–1.33 (m, 1 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 156.4, 143.5, 138.7, 130.9, 129.5, 128.9, 128.1, 124.4, 114.3, 98.8, 66.0, 41.6, 31.5, 28.1, 28.0, 26.2.

MS (EI): *m*/*z* = 441 [M^+ + 1].

Anal. Calcd for $C_{20}H_{22}OSTe$: C, 54.84; H, 5.06. Found: C, 54.76; H, 5.18.

2d
Yellow oil.

IR (neat): 1615, 1036 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 7.75–7.73 (m, 2 H), 7.41–7.39 (m, 5 H), 7.32–7.30 (m, 1 H), 7.25–7.21 (m, 2 H), 4.26 (d, *J* = 13.2 Hz, 1 H), 3.71 (d, *J* = 13.2 Hz, 1 H), 2.0 (s, 3 H), 1.70 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 148.2, 143.6, 138.9, 130.9, 129.6, 128.9, 128.2, 124.2, 114.0, 101.6, 66.6, 30.4, 20.9.

MS (EI): *m*/*z* = 400 [M^+ + 1].

Anal. Calcd for $C_{17}H_{18}OSTe$: C, 51.30; H, 4.56. Found: C, 51.45; H, 4.48.

2e
Oil.

IR (neat): 1628, 1040 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 7.55–7.53 (m, 4 H), 7.51–7.45 (m, 3 H), 7.32–7.29 (m, 3 H), 5.6 (s, 1 H), 5.42 (s, 1 H), 3.72 (d, *J* = 13.2 Hz, 1 H), 3.51 (d, *J* = 12.8 Hz, 1 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 143.3, 135.0, 131.2, 130.6, 129.5, 129.0, 128.5, 127.7, 124.2, 123.7, 66.7.

MS (EI): *m*/*z* = 323 [M^+ + 1].

Anal. Calcd for $C_{15}H_{14}OSSe$: C, 56.07; H, 4.39. Found: C, 56.14; H, 4.30.

2f
Oil.

IR (neat): 1625, 1043 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 7.53–7.52 (m, 2 H), 7.38–7.36 (m, 3 H), 5.39 (s, 1 H), 5.11 (s, 1 H), 3.67 (d, *J* = 12.8 Hz, 1 H), 3.44 (d, *J* = 12.8 Hz, 1 H), 2.64 (t, *J* = 7.2 Hz, 3 H), 1.58–1.51 (m, 2 H), 1.31 (m, 2 H), 0.79 (t, *J* = 7.2 Hz, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 143.4, 131.2, 129.6, 128.9, 124.3, 119.5, 67.9, 31.2, 26.3, 22.9, 13.5.

MS (EI): *m*/*z* = 303 [M^+ + 1].

Anal. Calcd for $C_{13}H_{18}OSSe$: C, 51.82; H, 6.02. Found: C, 51.91; H, 5.97.

2g
Oil.

IR (neat): 1637, 1032 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 7.48–7.46 (m, 3 H), 7.41–7.35 (m, 3 H), 7.34–7.29 (m, 4 H), 7.25–7.21 (m, 5 H), 4.19 (s, 2 H), 1.77 (s, 1 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 145.3, 141.5, 140.2, 138.4, 135.0, 129.1, 128.5, 127.9, 127.4, 127.0, 124.1, 112.4, 69.5.

MS (EI): *m*/*z* = 399 [M^+ + 1].

Anal. Calcd for $C_{21}H_{18}OSSe$: C, 63.47; H, 4.57. Found: C, 63.35; H, 4.64.

2h
Oil.

IR (neat): 1645, 1038 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 7.51–7.49 (m, 2 H), 7.38 (m, 5 H), 7.26–7.20 (m, 3 H), 4.37 (s, 2 H), 2.75 (t, *J* = 7.2 Hz, 2 H), 2.04 (s, 1 H), 1.60 (m, 2 H), 1.32 (m, 2 H), 0.83 (t, *J* = 7.2 Hz, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 143.5, 135.2, 132.5, 130.2, 129.6, 129.0, 128.3, 126.5, 124.3, 112.6, 68.3, 30.2, 27.8, 23.6, 13.4.

MS (EI): *m*/*z* = 379 [M^+ + 1].

Anal. Calcd for $C_{19}H_{22}OSSe$: C, 60.47; H, 5.88. Found: C, 60.38; H, 5.80.

2i
Oil.

IR (neat): 1630, 1034 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 7.52–7.51 (m, 2 H), 7.50–7.38 (m, 5 H), 7.25–7.20 (m, 3 H), 4.11 (d, *J* = 13.2 Hz, 1 H), 3.63 (d, *J* = 12.8 Hz, 1 H), 2.63–2.60 (m, 1 H), 2.52–2.49 (m, 1 H), 2.30–2.25 (m, 1 H), 2.06–2.03 (m, 1 H), 1.53–1.47 (m, 5 H), 1.36–1.34 (m, 1 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 154.9, 143.7, 132.1, 130.9, 130.5, 129.3, 128.9, 127.0, 124.3, 111.3, 64.2, 36.2, 31.9, 28.1, 28.0, 26.1.

MS (EI): *m*/*z* = 391 [M^+ + 1].

Anal. Calcd for $C_{20}H_{22}OSSe$: C, 61.69; H, 5.69. Found: C, 61.75; H, 5.60.

2j
Oil.

IR (neat): 1628, 1035 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 7.60–7.59 (m, 2 H), 7.44–7.43 (m, 3 H), 4.16 (d, *J* = 12.4 Hz, 1 H), 3.79 (d, *J* = 12.8 Hz, 1 H), 2.63 (t, *J* = 7.2 Hz, 2 H), 2.56–2.53 (m, 1 H), 2.42–2.39 (m, 1 H), 2.10–2.07 (m, 1 H), 1.86–1.85 (m, 1 H), 1.58–1.52 (m, 2 H), 1.46–1.40 (m, 3 H), 1.39–1.32 (m, 4 H), 1.18–1.16 (m, 1 H), 0.85 (t, *J* = 7.2 Hz, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 153.9, 144.6, 130.9, 128.9, 124.5, 109.9, 65.2, 36.0, 32.2, 31.5, 27.9, 27.8, 26.7, 26.1, 22.8, 13.5.

MS (EI): *m*/*z* = 371 [M^+ + 1].

Anal. Calcd for $C_{18}H_{26}OSSe$: C, 58.52; H, 7.09. Found: C, 58.60; H, 7.15.

2k
Oil.

IR (neat): 1645, 1045 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 7.52–7.50 (m, 2 H), 7.45–7.42 (m, 5 H), 7.28–7.26 (m, 3 H), 4.08 (d, *J* = 13.2 Hz, 1 H), 3.58 (d, *J* = 13.2 Hz, 1 H), 2.06 (s, 3 H), 1.70 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 146.7, 143.5, 131.0, 128.8, 124.2, 112.5, 65.4, 32.3, 26.3, 25.8, 22.9, 20.9, 13.6.

MS (EI): *m*/*z* = 351 [M$^+$ + 1].

Anal. Calcd for $C_{17}H_{18}OSSe$: C, 58.45; H, 5.19. Found: C, 58.39; H, 5.25.

2l
Oil.

IR (neat): 1640, 1048 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.68–7.66 (m, 2 H), 7.53–7.51 (m, 2 H), 4.19 (d, *J* = 12.4 Hz, 1 H), 3.81 (d, *J* = 12.8 Hz, 1 H), 2.74 (t, *J* = 7.2 Hz, 2 H), 2.0 (s, 3 H), 1.67–1.61 (m, 2 H), 1.53 (s, 3 H), 1.47–1.41 (m, 2 H), 0.94 (t, *J* = 7.2 Hz, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 145.7, 143.5, 131.0, 128.8, 124.2, 112.5, 65.4, 32.3, 26.3, 25.8, 22.9, 20.9, 13.6.

MS (EI): *m*/*z* = 331 [M$^+$ + 1].

Anal. Calcd for $C_{15}H_{22}OSSe$: C, 54.70; H, 6.73. Found: C, 54.81; H, 6.62.

2m
Oil.

IR (neat): 1618, 1050 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.67 (m, 5 H), 7.42–7.40 (m, 2 H), 7.37–7.28 (m, 3 H), 5.27 (s, 1 H), 5.18 (s, 1 H), 3.70 (d, *J* = 12.4 Hz, 1 H), 3.46 (d, *J* = 12.4 Hz, 1 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 143.5, 134.6, 133.5, 131.4, 129.4, 129.2, 129.0, 128.5, 124.2, 11.9, 63.7.

MS (EI): *m*/*z* = 275 [M$^+$ + 1].

Anal. Calcd for $C_{15}H_{14}OS_2$: C, 65.66; H, 5.14. Found: C, 65.54; H, 5.21.

2n
Oil.

IR (neat): 1608, 1049 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.51–7.49 (m, 2 H), 7.41–7.36 (m, 3 H), 7.34–7.30 (m, 6 H), 7.29–7.24 (m, 4 H), 4.20 (s, 2 H), 1.68 (s, 1 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 145.2, 140.5, 140.2, 139.4, 138.4, 135.1, 128.6, 127.9, 127.4, 127.0, 124.1, 113.4, 68.9.

MS (EI): *m*/*z* = 351 [M$^+$ + 1].

Anal. Calcd for $C_{21}H_{18}OS_2$: C, 71.96; H, 5.18. Found: C, 72.05; H, 5.06.

2o
Oil.

IR (neat): 1635, 1030 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.62–7.56 (m, 2 H), 7.55–7.50 (m, 3 H), 7.29–7.25 (m, 2 H), 7.20–7.15 (m, 3 H), 4.04 (d, *J* = 12.8 Hz, 1 H), 3.54 (d, *J* = 12.8 Hz, 1 H), 2.69–2.66 (m, 1 H), 2.57–2.54 (m, 1 H), 2.32–2.27 (m, 1 H), 2.11–2.07 (m, 1 H), 1.56–1.51 (m, 5 H), 1.39 (m, 1 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 156.7, 143.6, 135.3, 131.0, 129.1, 129.0, 128.8, 126.2, 124.3, 113.1, 62.6, 33.5, 32.1, 28.1, 28.0, 26.1.

MS (EI): *m*/*z* = 343 [M$^+$ + 1].

Anal. Calcd for $C_{20}H_{22}OS_2$: C, 70.13; H, 6.47. Found: C, 70.21; H, 6.35.

2p
Oil.

IR (neat): 1618, 1050 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.54–7.46 (m, 5 H), 7.29–7.17 (m, 5 H), 4.01 (d, *J* = 12.0 Hz, 1 H), 3.52 (d, *J* = 12.0 Hz, 1 H), 2.06 (s, 3 H), 1.78 (s, 1 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 148.7, 134.9, 131.0, 131.1, 129.1, 129.0, 128.9, 126.4, 124.2, 116.2, 65.2, 23.7, 21.8.

MS (EI): *m*/*z* = 303 [M$^+$ + 1].

Anal. Calcd for $C_{17}H_{18}OS_2$: C, 67.51; H, 6.00. Found: C, 67.60; H, 5.89.

5-Methoxy-2-(1-methylethylidene)pent-3-ynyl Phenyl Sulfoxide (3)
A mixture of **2d** (398 mg, 1 mmol), 3-methoxypropyne (140 mg, 2.0 mmol), $PdCl_2$ (10 mol%), CuI (10 mol%) and Et_3N (101 mg, 1.0 mmol) in MeOH (2 mL) was stirred at r.t. for 24 h under N_2. After completion of the reaction, the mixture was diluted with brine (10 mL) and extracted with EtOAc (3 × 15 mL). The organic layer was dried ($MgSO_4$). After filtration and removal of the solvent in vacuo, the residue was purified via chromatography on silica gel with *n*-hexane–EtOAc (3:1) as the eluent to give **3** as an oil; yield: 152 mg (58%).

IR (KBr): 2230, 1605, 1049 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.65–7.64 (m, 2 H), 7.50–7.48 (m, 3 H), 4.19 (s, 2 H), 3.89 (d, *J* = 12.0 Hz, 1 H), 3.55 (d, *J* = 12.4 Hz, 1 H), 3.38 (s, 3 H), 1.96 (s, 3 H), 1.50 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 149.4, 143.2, 131.2, 128.8, 124.4, 106.3, 88.3, 85.4, 61.4, 60.2, 57.5, 24.0, 20.2.

MS (EI): *m*/*z* = 263 [M$^+$ + 1].

Anal. Calcd for $C_{15}H_{18}O_2S$: C, 68.67; H, 6.92. Found: C, 68.76; H, 6.81.

2,3-Bis(phenylsulfonyl)prop-1-ene (4)
To a solution of **2m** (274 mg, 1 mmol) in CH_2Cl_2 (5 mL) was added MCPBA (690 mg, 5 mmol) at 0 °C, then the temperature was allowed to warm up to r.t. After completion of the reaction (TLC), the mixture was quenched with aq NH_4Cl (5 mL), and extracted with Et_2O (3 × 15 mL). The combined Et_2O extracts were dried (Mg_2SO_4) and filtered. Removal of the solvent in vacuo afforded the crude product, which was purified by flash chromatography (hexane–EtOAc, 3:1) to afford **4**; yield: 274 mg (85%); mp 128–129 °C (Lit[12] mp 128–129.5 °C).

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.54–7.40 (m, 2 H), 7.38–7.35 (m, 3 H), 7.32–7.24 (m, 5 H), 6.80 (s, 1 H), 5.21 (s, 1 H), 3.70 (s, 2 H).

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