Enantioselective Additive Pummerer-Type Reaction in Chiral, Non-racemic Vinyl Sulfoxides Induced by *O*-Silylated Ketene Acetal

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Received September 29, 1995; accepted January 10, 1996

Treatment of chiral, non-racemic vinyl sulfoxides with *O*-silylated ketene acetal in the presence of a catalytic amount of zinc chloride in tetrahydrofuran resulted in an enantioselective Pummerer-type reaction, affording a considerable yield of the corresponding enantiomerically enriched methyl 4-siloxy-4-sulfenylbutyrate in 78—82% ee.

Key words *O*-methyl-*O*-*tert*-butyldimethylsilyl ketene acetal; additive Pummerer reaction; vinyl sulfoxide; enantioselective reaction; methyl 4-siloxy-4-sulfenylbutyrate

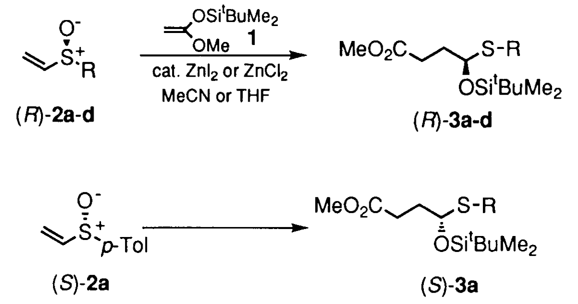
The Pummerer reaction of sulfoxides is a useful method for the synthesis of α-substituted sulfides[1)] and has attracted considerable attention from both synthetic and mechanistic points of view.[2)] In the last few years, we have reported a silicon-induced Pummerer-type reaction of sulfoxides with *O*-methyl-*O*-*tert*-butyldimethylsilyl ketene acetal (**1**),[3,4)] and its reaction mechanism.[5)]

While much attention has been focused on the additive Pummerer reaction of vinyl sulfoxides,[6)] there has been no report on the initial activation of the sulfinyl group of vinyl sulfoxides with *O*-silylated ketene acetal (**1**). Several years ago, we reported a silicon-induced additive Pummerer-type reaction with racemic vinyl sulfoxides.[7)] We now report the first enantioselective additive Pummerer-type reaction with chiral, non-racemic vinyl sulfoxides.

First we examined the reaction of the optically active vinyl sulfoxides (**2**) under our standard silicon-induced Pummerer-type reaction conditions.[3d,e)] Treatment of enantiomerically pure (*R*)-vinyl sulfoxide (**2a**) with **1** in the presence of a catalytic amount of zinc iodide (ZnI_2) in acetonitrile (MeCN) gave methyl (*R*)-4-siloxy-4-sulfenylbutyrate (**3a**) in 33% ee (66% chemical yield). The use of zinc chloride ($ZnCl_2$) instead of ZnI_2[3f,8)] in tetrahydrofuran (THF) was found to give the best result (78% ee, 78% chemical yield). Under the same conditions, other sulfoxides (*R*)-**2b**—**d** and (*S*)-**2a** reacted with **1** to give the corresponding additive Pummerer-type products (*R*)-**3b**—**d** and (*S*)-**3a** with high enantioselectivity (Table 1), although their chemical yields were lower than that of **2a**. The absolute stereochemistry of **3a**—**d** was tentatively assigned on the basis of the similarity of the shift patterns in the ^{1}H-NMR signals on addition of $Eu(hfc)_3$.[3d)]

Next, we examined the reaction of β-monosubstituted vinyl sulfoxides [(*R*)-**4***E* and (*R*)-**4***Z*] with **1**. Vinyl sulfoxides (*R*)-**4** [(*R*)-**4***E* and (*R*)-**4***Z*] were treated with **1** in the presence of a catalytic amount of $ZnCl_2$ in THF, but no product was obtained and the starting sulfoxides were recovered unchanged under the same reaction conditions as employed in the case of **2**. The use of ZnI_2 instead of $ZnCl_2$ in MeCN caused an additive Pummerer-

Table 1.



Sulfoxide	R	Conditions	Yield[a)] (%)	ee[b)] (%)
(*R*)-**2a**	*p*-Tol	ZnI_2, MeCN, 0 °C, 1 h	66	33
(*R*)-**2a**	*p*-Tol	$ZnCl_2$, MeCN, 0 °C, 2 h	69	60
(*R*)-**2a**	*p*-Tol	ZnI_2, THF, 0 °C, 10 min	71	33
(*R*)-**2a**	*p*-Tol	$ZnCl_2$, THF, R.T., 2 d	78	78
(*R*)-**2b**	Mesityl[c)]	$ZnCl_2$, THF, R.T., 2 d	16	78
(*R*)-**2c**	^{t}Bu	$ZnCl_2$, THF, 60 °C, 10 h	31	80
(*R*)-**2d**	iso-Pr	$ZnCl_2$, THF, R.T., 2 d	47	82
(*S*)-**2a**	*p*-Tol	$ZnCl_2$, THF, R.T., 1 d	78	80

^{t}Bu, *tert*-butyl. *a*) Isolated yield. *b*) ee values were determined by ^{1}H-NMR ($CDCl_3$) with $Eu(hfc)_3$. *c*) Mesityl is 2,4,6-trimethylphenyl.

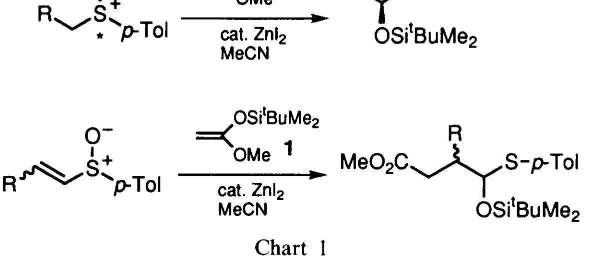


Chart 1

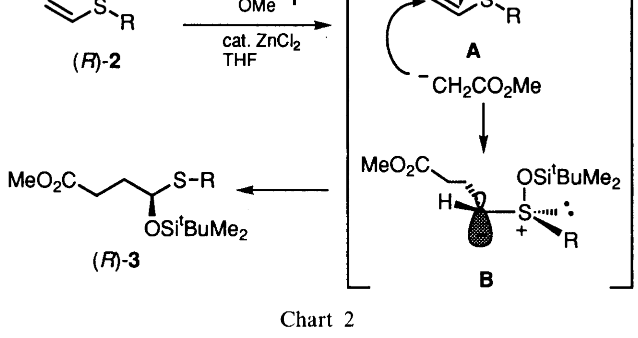


Chart 2

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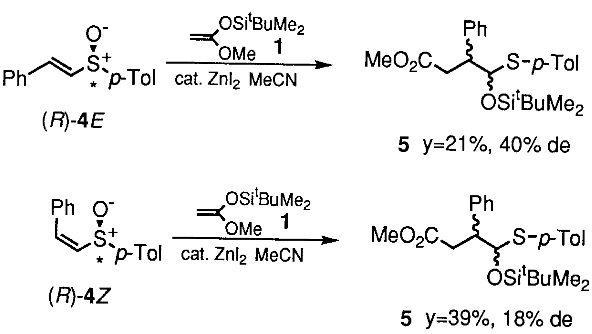

Chart 3

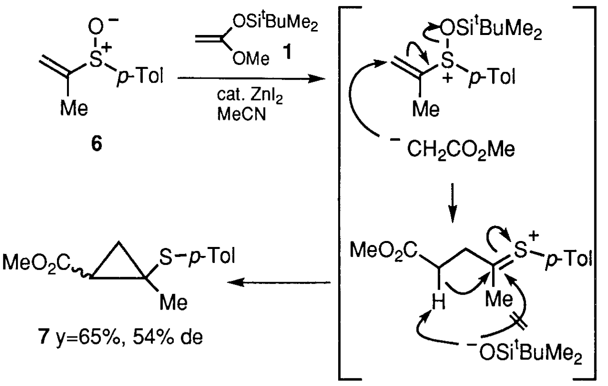

Chart 4

type reaction, which afforded the same adduct **5** in 21 and 39% yields (40 and 18% de), although the stereochemistry of the diastereomer has not been determined yet (Chart 3).

On the other hand, treatment of the α-substituted vinyl sulfoxide **6** and ZnI_2 with **1** in MeCN produced in 65% yield novel cyclopropanation reaction products **7** in a 10 : 3 ratio, which may reflect the steric hindrance at the α position to the sulfur atom (Chart 4).

The reaction mechanism and the asymmetric transformation in the present additive Pummerer-type reaction of the chiral, non-racemic vinyl sulfoxides **2** can be interpreted as follows: silylation of the sulfoxides with **1** affords an intermediate **A**, which may yield an anionic intermediate **B** by addition of a generated ester enolate at the β-position to the sulfur atom. The direction of the generated anion is *anti*-periplanar to the siloxy group. Then the siloxy group may be forced to migrate to the α-position on the same face of the sulfinyl oxygen as discussed in our preceding paper (Chart 2).[3d,e)]

Experimental

IR absorption spectra were recorded on a Shimadzu FTIR-8100 spectrophotometer with $CHCl_3$ as a solvent. ^{1}H-NMR spectra were measured on JEOL JNM-EX270 (270 MHz) and Hitachi R-250 HT (250 MHz) spectrometers with $CDCl_3$ as a solvent and tetramethylsilane as an internal standard, unless otherwise noted. Mass spectra (MS) and high-resolution MS were obtained with ESCO EMD-05A and JEOL JMS-D300 mass spectrometers. Optical rotations were measured in 1-dm cells of 1 ml capacity with a Perkin-Elmer 241 instrument. E. Merck precoated TLC plates and Silica gel F_{254} for preparative TLC were used. Organic layers were dried with anhydrous Na_2SO_4. The known vinyl sulfoxides (*R*)-**2a**,[9)] **2c**,[10)] **4*E*** and ***Z***[11)] were prepared by the reported methods, and other starting vinyl sulfoxides were prepared by the same procedure as reported.[9),10)]

(*R*)-2,4,6-Trimethylphenyl Vinyl Sulfoxide (*R*)-2b[10)] A 1.0 M solution of vinylmagnesium bromide in THF (4.4 ml, 4.40 mmol) was added dropwise to a solution of (2*S*)-1,1-diphenyl-1-hydroxypropyl 2-(2,4,6-trimethylphenyl)sulfinate[10)] (753 mg, 1.99 mmol) in THF (40 ml) at 0 °C. Under the same conditions, the reaction mixture was stirred for 20 min. Water was added and the whole was extracted with Et_2O three times. The organic layer was washed with saturated aqueous NaCl, dried and concentrated. The residue was purified by column chromatography with 20% AcOEt in hexane to gave (*R*)-**2b** (184.5 mg, 48%) as a colorless oil. $[\alpha]_D^{26}$ +498.2 (c=4.2, $CHCl_3$). IR ($CHCl_3$) cm^{-1}: 3005, 1603, 1456, 1065, 1042. ^{1}H-NMR δ: 2.26 (s, 3H), 2.48 (s, 6H), 5.93 (d, 1H, J=9.8 Hz), 6.12 (d, 1H, J=16.5 Hz), 6.62 (dd, 1H, J=9.8, 16.5 Hz), 6.84 (s, 2H). *Anal.* Calcd for $C_{11}H_{14}OS$: C, 68.00; H, 7.26. Found: C, 68.26; H, 7.35.

(*R*)-Isopropyl Vinyl Sulfoxide (*R*)-2d[10)] (2*S*)-1,1-Diphenyl-1-hydroxypropyl 2-isopropylsulfinate (460 mg, 1.45 mmol), a 1.0 M solution of vinylmagnesium bromide in THF (3.6 ml, 3.60 mmol), and THF (15 ml) afforded (*R*)-**2d** (51.0 mg, 30%) as a colorless oil. $[\alpha]_D^{27}$ +67.4 (c=0.1, $CHCl_3$). IR ($CHCl_3$) cm^{-1}: 1221, 1213. ^{1}H-NMR δ: 1.24, 1.30 (each d, each 3H, J=7.5 Hz), 2.74—2.92 (m, 1H), 6.02 (d, 1H, J=9.8 Hz), 6.09 (d, 1H, J=16.5 Hz), 6.56 (dd, 1H, J=9.8, 16.5 Hz).

(*S*)-4-Methylphenyl Vinyl Sulfoxide (*S*)-2a[9)] D-Menthyl (*R*)-(−)-4-methylphenylsulfinate (880 mg, 2.98 mmol), a 1.0 M solution of vinylmagnesium bromide in THF (4.5 ml, 4.50 mmol), and toluene (20 ml) afforded (*S*)-**2a** (397 mg, 83%) as a colorless oil. $[\alpha]_D^{23}$ −427.0 (c=0.5, EtOH). *Anal.* Calcd for $C_9H_{10}OS$: C, 65.02; H, 6.06; S, 19.29. Found: C, 65.13; H, 6.06; S, 19.03.

4-Methylphenyl 2-Propenyl Sulfoxide (6)[9)] L-Menthyl (*S*)-(+)-4-methylphenylsulfinate (1.13 g, 3.83 mmol), a 1.0 M solution of 2-propenylmagnesium bromide in THF (4.2 ml, 4.20 mmol), and toluene (25 ml) afforded **6** (672 mg, 97%) as a colorless oil. IR ($CHCl_3$) cm^{-1}: 3005, 1493, 1082, 1043. ^{1}H-NMR δ: 1.72 (s, 3H), 2.41 (s, 3H), 5.59, 5.99 (each s, each 1H), 7.27—7.51 (m, 4H). HRMS Calcd for $C_{10}H_{12}OS$ (M^+): 180.0609. Found: 180.0597.

General Procedure for the Pummerer-Type Reaction of *O*-Silylated Ketene Acetal (1) with Vinyl Sulfoxides (2 and 4) *O*-*tert*-Butyldimethylsilylketene acetal (**1**, 1.0 mmol) was added dropwise to a solution of sulfoxide (**2**, 0.2 mmol) and ZnI_2 or $ZnCl_2$ (0.02 mmol) in dry MeCN or THF at 0 °C. The reaction mixture was stirred under nitrogen under the conditions indicated in the Table 1. The mixture was poured into saturated aqueous sodium hydrogen carbonate and extracted with AcOEt three times. The organic layer was dried and concentrated. The residue was purified by preparative TLC to give the corresponding methyl 4-siloxy-4-sulfenylbutyrate (**3** and **5**) in yields between 16 and 78%.

Methyl (*R*)-4-*tert*-Butyldimethylsiloxy-4-(4-methylphenyl)sulfenylbutyrate (*R*)-3a (i) (*R*)-Vinyl sulfoxide (*R*)-**2a** (45.7 mg, 0.275 mmol), **1** (258.0 mg, 1.372 mmol), ZnI_2 (8.8 mg, 0.028 mmol) and dry MeCN (1 ml) afforded the methyl butyrate (*R*)-**3a** (64.1 mg, 66%, 33% ee).

(ii) (*R*)-Vinyl sulfoxide (*R*)-**2a** (14.3 mg, 0.086 mmol), **1** (80.9 mg, 0.430 mmol), $ZnCl_2$ (1.0 M solution in Et_2O; 0.008 ml, 0.008 mmol) and dry MeCN (1 ml) afforded the methyl butyrate (*R*)-**3a** (20.9 mg, 69%, 60% ee).

(iii) (*R*)-Vinyl sulfoxide (*R*)-**2a** (19.5 mg, 0.117 mmol), **1** (110.0 mg, 0.585 mmol), ZnI_2 (3.7 mg, 0.012 mmol) and dry THF (1 ml) afforded the methyl butyrate (*R*)-**3a** (29.5 mg, 71%, 33% ee).

(iv) (*R*)-Vinyl sulfoxide (*R*)-**2a** (21.8 mg, 0.131 mmol), **1** (123 mg, 0.654 mmol), $ZnCl_2$ (1.0 M solution in Et_2O; 0.013 ml, 0.013 mmol) and dry THF (1 ml) afforded the methyl butyrate (*R*)-**3a** (36.3 mg, 78%, 78% ee) as a colorless oil. $[\alpha]_D^{25}$ −29.0 (c=1.15, $CHCl_3$). IR ($CHCl_3$) cm^{-1}: 1732, 839. ^{1}H-NMR δ: 0.05, 0.06 (each s, each 3H), 0.89 (s, 9H), 2.04 (dt, 2H, J=6.3, 7.3 Hz), 2.33 (s, 3H), 2.48 (t, 1H, J=7.3 Hz), 2.49 (t, 1H, J=7.3 Hz), 3.66 (s, 3H), 5.07 (t, 1H, J=6.3 Hz), 7.11 (d, 2H, J=8.3 Hz), 7.38 (d, 2H, J=8.3 Hz). HRMS Calcd for $C_{18}H_{30}O_3SSi$ (M^+): 354.1682. Found: 354.1654.

Methyl (*R*)-4-*tert*-Butyldimethylsiloxy-4-(2,4,6-trimethylphenyl)sulfenylbutyrate (*R*)-3b (*R*)-Vinyl sulfoxide (*R*)-**2b** (18.1 mg, 0.093 mmol), **1** (87.6 mg, 0.466 mmol), $ZnCl_2$ (1.0 M solution in Et_2O; 0.009 ml, 0.009 mmol) and dry THF (1 ml) afforded the methyl butyrate (*R*)-**3b** (5.8 mg, 16%, 78% ee) as a colorless oil. $[\alpha]_D^{26}$ +7.2 (c=0.5, $CHCl_3$). IR ($CHCl_3$) cm^{-1}: 3020, 1734, 1223, 1211. ^{1}H-NMR δ: −0.26, −0.10 (each s, each 3H), 0.83 (s, 9H), 2.03 (dt, 2H, J=5.3, 7.6 Hz), 2.25 (s, 3H), 2.48 (s, 6H), 2.58 (t, 1H, J=7.6 Hz), 2.60 (t, 1H, J=7.6 Hz), 3.67 (s, 3H), 5.08 (t, 1H, J=5.3 Hz), 6.91 (s, 2H). HRMS Calcd for $C_{16}H_{25}O_3SSi$ ($M^+-{}^t$Bu): 325.1294. Found: 325.1300.

Methyl (*R*)-4-*tert*-Butyldimethylsiloxy-4-*tert*-butylsulfenylbutyrate (*R*)-3c (*R*)-Vinyl sulfoxide (*R*)-**2c** (26.7 mg, 0.202 mmol), **1** (190 mg, 1.01 mmol), $ZnCl_2$ (1.0 M solution in Et_2O; 0.020 ml, 0.020 mmol) and dry THF (1 ml) afforded the methyl butyrate (*R*)-**3c** (19.9 mg, 31%, 80% ee) as a colorless oil. $[\alpha]_D^{23}$ +13.6 (c=0.3, $CHCl_3$). IR

($CHCl_3$) cm^{-1}: 1732, 1221. ^{1}H-NMR δ: 0.14, 0.15 (each s, each 3H), 0.89 (s, 9H), 1.36 (s, 9H), 2.04—2.11 (m, 2H), 2.51 (t, 2H, $J=6.8$ Hz), 3.67 (s, 3H), 5.07 (t, 1H, $J=6.0$ Hz). HRMS Calcd for $C_{11}H_{23}O_3SSi$ ($M^+ - {}^t$Bu): 263.1135. Found: 263.1127. Anal. Calcd for $C_{15}H_{32}O_3SSi$: C, 56.20; H, 10.06. Found: C, 56.54; H, 9.97.

Methyl (*R*)-4-*tert*-Butyldimethylsiloxy-4-iso-propylsulfenylbutyrate (*R*)-3d (*R*)-Vinyl sulfoxide (*R*)-**2d** (6.2 mg, 0.053 mmol), **1** (49.4 mg, 0.263 mmol), $ZnCl_2$ (1.0 M solution in Et_2O; 0.005 ml, 0.005 mmol) and dry THF (1 ml) afforded the methyl butyrate (*R*)-**3d** (7.5 mg, 47%, 82% ee) as a colorless oil. $[\alpha]_D^{25}$ -6.7 ($c=0.2$, $CHCl_3$). IR ($CHCl_3$) cm^{-1}: 1732, 1217, 1093. ^{1}H-NMR δ: 0.14, 0.12 (each s, each 3H), 0.89 (s, 9H), 1.28 (t, 6H, $J=6.8$ Hz), 2.11 (dt, 2H, $J=6.8$, 7.3 Hz), 2.49 (t, 2H, $J=7.3$ Hz), 3.09 (quint., 1H, $J=6.8$ Hz), 3.67 (s, 3H), 4.95 (t, 1H, $J=6.3$ Hz). HRMS Calcd for $C_{10}H_{21}O_3SSi$ ($M^+ - {}^t$Bu): 249.0980. Found: 249.0966.

Methyl (*S*)-4-*tert*-Butyldimethylsiloxy-4-(4-methylphenyl)sulfenylbutyrate (*S*)-3a (*S*)-Vinyl sulfoxide (*S*)-**2a** (25.6 mg, 0.154 mmol), **1** (145 mg, 0.770 mmol), $ZnCl_2$ (1.0 M solution in Et_2O; 0.015 ml, 0.015 mmol) and dry THF (1 ml) afforded the methyl butyrate (*S*)-**3a** (42.7 mg, 78%, 80% ee) as a colorless oil. $[\alpha]_D^{26}$ $+29.1$ ($c=0.5$, $CHCl_3$). HRMS Calcd for $C_{14}H_{21}O_3SSi$ ($M^+ - {}^t$Bu): 297.0979. Found: 297.0979.

Methyl 4-*tert*-Butyldimethylsiloxy-4-(4-methylphenyl)sulfenyl-3-phenylbutyrate (5) (i) 4-Methylphenyl (*E*)-styryl sulfoxide$^{11)}$ (34.7 mg, 0.143 mmol), **1** (134.4 mg, 0.715 mmol), ZnI_2 (4.6 mg, 0.014 mmol) and dry MeCN (1 ml) afforded the methyl butyrate **5** (13.2 mg, 21%, 40% de) as a colorless oil. $[\alpha]_D^{28}$ $+7.9$ ($c=0.5$, $CHCl_3$). IR ($CHCl_3$) cm^{-1}: 1742, 1256. ^{1}H-NMR δ: -0.16, 0.03 (each s, each $70/100 \times 3$H), -0.15, -0.04 (each s, each $30/100 \times 3$H), 0.79 (s, $30/100 \times 9$H), 0.87 (s, $70/100 \times 9$H), 2.32 (s, $30/100 \times 3$H), 2.35 (s, $70/100 \times 3$H), 2.6—3.8 (m, 3H), 3.54 (s, $30/100 \times 3$H), 3.55 (s, $70/100 \times 3$H), 5.11 (d, $70/100 \times 1$H, $J=4.3$ Hz), 5.24 (d, $30/100 \times 1$H, $J=5.6$ Hz), 7.06—7.34 (m, 9H).

(ii) 4-Methylphenyl (*Z*)-styryl sulfoxide$^{11)}$ (18.9 mg, 0.078 mmol), **1** (73.3 mg, 0.390 mmol), ZnI_2 (2.5 mg, 0.008 mmol) and dry MeCN (1 ml) afforded the methyl butyrate **5** (13.1 mg, 39%, 18% de) as a colorless oil. $[\alpha]_D^{28}$ $+11.8$ ($c=1.3$, $CHCl_3$). IR ($CHCl_3$) cm^{-1}: 1740, 1254. ^{1}H-NMR δ: -0.16, 0.03 (each s, each $59/100 \times 3$H), -0.15, -0.04 (each s, each $41/100 \times 3$H), 0.80 (s, $41/100 \times 9$H), 0.88 (s, $59/100 \times 9$H), 2.32 (s, $41/100 \times 3$H), 2.35 (s, $59/100 \times 3$H), 2.6—3.8 (m, 3H), 3.54 (s, $41/100 \times 3$H), 3.55 (s, $59/100 \times 3$H), 5.11 (d, $59/100 \times 1$H, $J=4.3$ Hz), 5.25 (d, $41/100 \times 1$H, $J=5.6$ Hz), 7.06—7.34 (m, 9H).

Methyl 2-Methyl-2-(4-methylphenylsulfenyl)cyclopropanecarboxylate (7) Compound **1** (129 mg, 0.690 mmol) was added dropwise to a solution of **6** (24.8 mg, 0.138 mmol), ZnI_2 (4.4 mg, 0.014 mmol) in dry MeCN (1 ml) at 0 °C. The reaction mixture was stirred under nitrogen at 0 °C for 3.5 h, then poured into saturated aqueous sodium hydrogen carbonate and extracted with AcOEt three times. The organic layer was dried and evaporated. The residue was purified by preparative TLC to give diastereomers of **7** [major diastereomer (16.2 mg, 50%), minor diastereomer (4.9 mg, 15%)].

Major diastereomer; colorless oil. IR ($CHCl_3$) cm^{-1}: 1732, 1493, 1439, 1371, 1202, 1161. ^{1}H-NMR δ: 1.43 (d, 1H, $J=8.2$ Hz), 1.44 (d, 1H, $J=6.6$ Hz), 1.51 (s, 3H), 2.21 (dd, 1H, $J=6.6$, 8.2 Hz), 2.34 (s, 3H), 3.71 (s, 3H), 7.14, 7.32 (each d, each 2H, $J=8.0$ Hz). HRMS Calcd for $C_{13}H_{16}O_2S$ (M^+): 236.0871. Found: 236.0871.

Minor diastereomer; colorless oil. IR ($CHCl_3$) cm^{-1}: 1740, 1493, 1439, 1202, 1169. ^{1}H-NMR δ: 1.19 (dd, 1H, $J=5.3$, 7.9 Hz), 1.50 (s, 3H), 1.62 (t, 1H, $J=5.6$ Hz), 1.97 (t, 1H, $J=7.9$ Hz), 2.32 (s, 3H), 3.69 (s, 3H), 7.10, 7.28 (each d, each 2H, $J=7.3$ Hz). HRMS Calcd for $C_{13}H_{16}O_2S$ (M^+): 236.0871. Found: 236.0869.

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