Addition Reaction of Dialkyl Disulfides to Terminal Alkynes Catalyzed by a Rhodium Complex and Trifluoromethanesulfonic Acid

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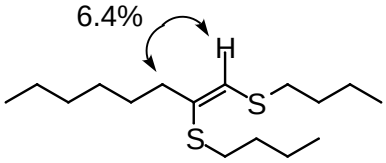
Supplementary Materials

^{1}H-NMR and ^{13}C-NMR spectra were obtained on a Varian Mercury (400 MHz). Chemical shift values are given in ppm relative to internal Me_4Si. IR spectra were recorded on a JASCO FT/IR-410. MS spectra were taken with a JEOL JMS-DX303 or a JEOL JMS-AX500.

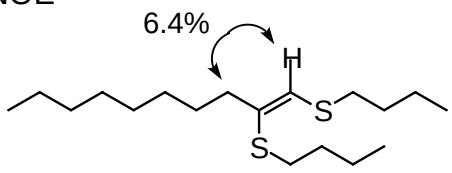
(*Z*)-1,2-Bis(butylthio)-1-octene. Colorless oil. ^{1}H-NMR (400MHz, $CDCl_3$) δ 0.89 (3H, t, J = 6.4 Hz), 0.91 (3H, t, J = 7.2 Hz), 0.92 (3H, t, J = 7.2 Hz), 1.25-1.33 (6H, m), 1.42 (4H, sixtet, J = 7.2 Hz), 1.47-1.65 (6H, m), 2.24 (2H, t, J = 7.2 Hz), 2.69 (4H, t, J = 7.2 Hz), 6.05 (1H, s). ^{13}C-NMR (100MHz, $CDCl_3$) δ 13.78, 13.81, 14.2, 21.9, 22.0, 22.7, 28.67, 28.74, 31.2, 31.7, 32.1, 32.5, 33.9, 37.0, 126.6, 132.0. IR (neat) 2956, 2927, 2857, 1572, 1464, 1378, 1270, 1222 cm^{-1}. MS (EI) m/z 288 (M^+, 100%). HRMS Calcd for $C_{16}H_{32}S_2$: 288.1946. Found: 288.1948.

NOE



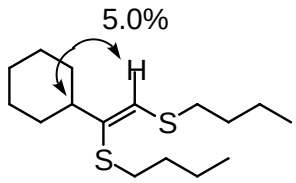
(*Z*)-1,2-Bis(butylthio)-1-decene. Colorless oil. ^{1}H-NMR (400MHz, $CDCl_3$) δ 0.88 (3H, t, J = 7.2 Hz), 0.91 (3H, t, J = 7.6 Hz), 0.92 (3H, t, J = 7.2 Hz), 1.22-1.30 (10H, m), 1.42 (4H, sixtet, J = 7.2 Hz), 1.49-1.55 (2H, m), 1.62 (4H, quintet, J = 7.2 Hz), 2.24 (2H, t, J = 7.6 Hz), 2.69 (4H, t, J = 7.2 Hz), 6.04 (1H, s). ^{13}C-NMR (100MHz, $CDCl_3$) δ 13.81, 13.83, 14.2, 21.9, 22.1, 22.8, 28.7, 29.1, 29.4, 29.5, 31.2, 32.0, 32.1, 32.6, 33.9, 37.1, 126.6, 132.1. IR (neat) 2956, 2926, 2855, 1464, 1378, 1272 cm^{-1}. MS (EI) m/z 316 (M^+, 100%), 57 (M^+-259, 41%). HRMS Calcd for $C_{18}H_{36}S_2$: 316.2258. Found: 316.2273.

NOE



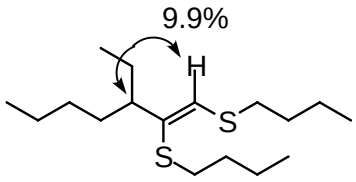
(*Z*)-1,2-Bis(butylthio)-1-cyclohexylethene. Colorless oil. ^{1}H-NMR (400MHz, $CDCl_3$) δ 0.91 (3H, t, J = 7.2 Hz), 0.92 (3H, t, J = 7.2 Hz), 1.13-1.33 (5H. m), 1.42 (4H, sixtet, J = 7.2 Hz), 1.52-1.69 (5H, m), 1.78 (2H, dd, J = 9.6, 3.2 Hz), 1.87 (2H, d, J = 10.4 Hz), 2.08 (1H, dt, J = 11.2, 2.4 Hz), 2.70 (4H, t, J = 7.2 Hz), 6.14 (1H, s). ^{13}C-NMR (100MHz, $CDCl_3$) δ 13.8, 13.9, 21.9, 22.1, 26.3, 26.7, 32.1, 32.4, 32.6, 32.9, 33.8, 45.9, 128.4, 136.4. IR (neat) 2956, 2927, 2852, 1567, 1449, 1221 cm^{-1}. MS (EI) m/z 286 (M^+, 78%), 41 (M^+-245, 100%). HRMS Calcd for $C_{16}H_{30}S_2$: 286.1989. Found: 286.1802.

NOE



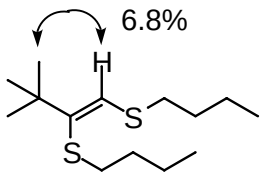
(*Z*)-1,2-Bis(butylthio)-3-ethyl-1-heptene. Colorless oil. ^{1}H-NMR (400MHz, $CDCl_3$) δ 0.81 (3H, t, J = 7.2 Hz), 0.88 (3H, t, J = 7.6 Hz), 0.91 (3H, t, J = 7.6 Hz), 0.92 (3H, t, J = 7.2 Hz), 1.16-1.21 (2H, m), 1.23-1.32 (2H, m), 1.37-1.49 (8H, m), 1.53-1.66 (4H, m), 2.07-2.13 (1H, m), 2.71 (2H, t, J = 7.6 Hz), 2.73 (2H, t, J = 7.6 Hz), 6.15 (1H, s). ^{13}C-NMR (100MHz, $CDCl_3$) δ 12.1, 13.80, 13.84, 14.2, 21.8, 22.1, 22.9, 26.8, 29.8, 32.2, 32.7, 32.8, 33.4, 33.8, 50.5, 131.2, 134.8. IR (neat) 2957, 2927, 2871, 1564, 1458, 1378, 1272 cm^{-1}. MS (EI) m/z 302 (M^+, 100%), 245 (M^+-57, 20%). HRMS Calcd for $C_{17}H_{34}S_2$: 302.2102. Found: 302.2100.

NOE



(*Z*)-1,2-Bis(butylthio)-3,3-dimethyl-1-butene. Colorless oil. ^{1}H-NMR (400MHz, $CDCl_3$) δ 0.92 (3H, t, J = 7.6 Hz), 0.93 (3H, t, J = 7.6 Hz), 1.15 (9H, s), 1.39-1.48 (4H, m), 1.55-1.67 (4H, m), 2.72 (2H, t, J = 7.6 Hz), 2.75 (2H, t, J = 7.2 Hz), 6.38 (1H, s). ^{13}C-NMR (100MHz, $CDCl_3$) δ 13.8, 13.9, 21.9, 22.2, 29.4, 32.0, 32.6, 33.6, 34.2, 39.6, 131.6, 140.4. IR (neat) 2957, 2870, 1463, 1357, 1236, 853 cm^{-1}. MS (EI) m/z 260 (M^+, 100%), 245 (M^+-15, 45%). HRMS Calcd for $C_{14}H_{28}S_2$: 260.1632. Found: 260.1624.

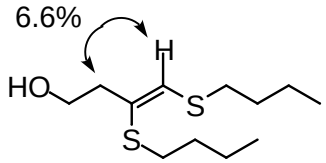
NOE



(*Z*)-1,2-Bis(butylthio)-1-buten-4-ol. Colorless oil. ^{1}H-NMR (400MHz, $CDCl_3$) δ 0.91 (3H, t,

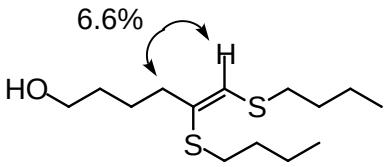
J = 7.2 Hz), 0.92 (3H, t, J = 7.2 Hz), 1.42 (2H, sixtet, J = 7.2 Hz), 1.43 (2H, sixtet, J = 7.2 Hz), 1.56 (2H, quintet, J = 7.6 Hz), 1.62 (2H, quintet, J = 7.2 Hz), 1.77 (1H, bs), 2.52 (2H, t, J = 5.6 Hz), 2.710 (2H, t, J = 7.2 HZ), 2.714 (2H, t, J = 7.2 Hz), 3.77 (2H, q, J = 5.6 Hz), 6.24 (1H, s). ^{13}C-NMR (100MHz, $CDCl_3$) δ 13.76, 13.79, 21.8, 22.0, 31.5, 32.0, 32.5, 33.9, 39.9, 61.3, 127.0, 131.1. IR (neat) 3370, 2956, 2928, 2871, 1570, 1458, 1268, 1220, 1047 cm^{-1}. MS (EI) m/z 248 (M^+, 100%), (M^+-87, 40%). HRMS Calcd for $C_{12}H_{24}S_2O_1$: 248.1269. Found: 248.1243.

NOE



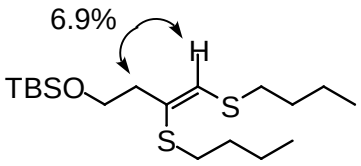
(*Z*)-1,2-Bis(butylthio)-1-hexen-6-ol. Colorless oil. ^{1}H-NMR (400MHz, $CDCl_3$) δ 0.91 (3H, t, J = 7.2 Hz), 0.92 (3H, t, J = 7.6 Hz), 1.35 (1H, bs), 1.38-1.47 (4H, m), 1.54-1.65 (8H, m), 2.29 (2H, t, J = 7.2 Hz), 2.688 (2H, t, J = 7.2 Hz), 2.692 (2H, t, J = 7.2 Hz), 3.66 (2H, t, J = 6.4 Hz), 6.09 (1H, s). ^{13}C-NMR (100MHz, $CDCl_3$) δ 13.79, 13.83, 21.9, 22.0, 24.8, 31.2, 32.1, 32.5, 33.9, 36.7, 62.8, 127.5, 131.2. IR (neat) 3349, 2955, 2929, 2870, 1458, 1270, 1222, 1062 cm^{-1}. MS (EI) m/z 276 (M^+, 100%), 129 (M^+-147, 61%). HRMS Calcd for $C_{14}H_{28}S_2O_1$: 276.1582. Found: 276.1575.

NOE



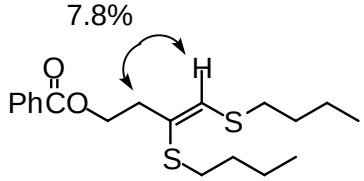
(*Z*)-1,2-Bis(butylthio)-4-(*t*-butyldimethyloxy)-1-butene. Colorless oil. ^{1}H-NMR (400MHz, $CDCl_3$) δ 0.06 (6H, s), 0.89 (9H, s), 0.91 (3H, t, J = 7.6 Hz), 0.92 (3H, t, J = 7.2 Hz), 1.418 (2H, sixtet, J = 7.2 Hz), 1.423 (2H, sixtet, J = 7.2 Hz), 1.56 (2H, quintet, J = 7.2 Hz), 1.62 (2H, quintet, J = 7.2 Hz), 2.46 (2H, t, J = 6.8 Hz), 2.69 (2H, t, J = 7.2 Hz), 3.74 (2H, t, J = 6.8 Hz), 6.17 (1H, s). ^{13}C-NMR (100MHz, $CDCl_3$) δ 5.1, 13.76, 13.82, 18.4, 21.8, 22.0, 26.1, 31.5, 32.1, 32.6, 33.9, 40.4, 62.0, 127.6, 130.0. IR (neat) 2956, 2928, 1464, 1254, 1099, 836, 776 cm^{-1}. MS (EI) m/z 362 (M^+, 27%), 305 (M^+-57, 100%). HRMS Calcd for $C_{18}H_{38}O_1S_2Si_1$: 362.2133. Found: 362.2124.

NOE



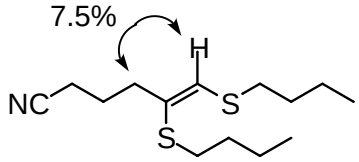
(*Z*)-1,2-Bis(butylthio)-4-benzoyloxy-1-butene. Colorless oil. ^{1}H-NMR (400MHz, $CDCl_3$) δ 0.88 (3H, t, J = 7.6 Hz), 0.91 (3H, t, J = 6.4 Hz), 1.34-1.48 (4H, m), 1.54-1.62 (4H, m), 2.68 (2H, t, J = 7.2 Hz), 2.71 (2H, t, J = 7.2 Hz), 2.75 (2H, t, J = 7.2 Hz), 4.48 (2H, t, J = 7.2 Hz), 6.28 (1H, s), 7.43 (2H, t, J = 7.2 Hz), 7.55 (1H, t, J = 7.6 Hz), 8.03 (2H, d, J = 8.0 Hz). ^{13}C-NMR (100MHz, $CDCl_3$) δ 13.7, 13.8, 21.8, 22.0, 31.6, 32.0, 32.6, 32.8, 36.5, 63.2, 126.3, 128.2, 129.4, 130.1, 131.5, 132.8, 166.2. IR (neat) 2957, 2871, 1719, 1452, 1271, 1116, 1070, 1025, 712 cm^{-1}. MS (EI) m/z 352 (M^+, 18%), 173 (M^+-179, 100%). HRMS Calcd for $C_{19}H_{28}S_2O_2$: 352.1531. Found: 351.0532.

NOE



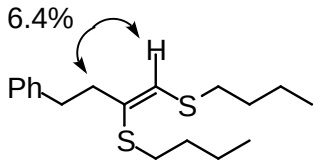
(*Z*)-1,2-Bis(butylthio)-5-pentenenitrile. Colorless oil. ^{1}H-NMR (400MHz, $CDCl_3$) δ 0.91 (3H, t, J = 7.2 Hz), 0.93 (3H, t, J = 7.2 Hz), 1.43 (4H, sixtet, J = 7.2 Hz), 1.56 (2H, quintet, J = 7.2 Hz), 1.62 (2H, quintet, J = 7.2 Hz), 1.89 (2H, quintet, J = 7.2 Hz), 2.34 (2H, t, J = 6.8 Hz), 2.43 (2H, t, J = 7.2 Hz), 2.67 (2H, t, J = 7.2 Hz), 2.71 (2H, t, J = 7.2 Hz), 6.24 (1H, s). ^{13}C-NMR (100MHz, $CDCl_3$) δ 13.77, 13.80, 16.1, 21.8, 22.0, 24.0, 31.3, 32.0, 32.6, 33.9, 35.3, 119.3, 128.0, 131.0. IR (neat) 2957, 2929, 2871, 2245, 1570, 1457, 1274, 1223 cm^{-1}. MS (EI) m/z 271 (M^+, 100%). HRMS Calcd for $C_{14}H_{25}S_2N_1$: 271.1428. Found: 271.1425.

NOE



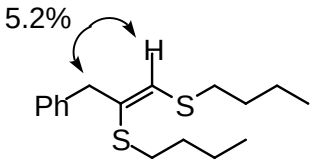
(*Z*)-1,2-Bis(butylthio)-4-phenyl-1-buten. Colorless oil. ^{1}H-NMR (400MHz, $CDCl_3$) δ 0.90 (3H, t, J = 7.2 Hz), 0.91 (3H, t, J = 7.2 Hz), 1.34-1.48 (4H, m), 1.51-1.61 (4H, m), 2.54 (2H, t, J = 7.2 Hz), 2.64 (2H, t, J = 7.2 Hz), 2.73 (2H, t, J = 7.6 Hz), 2.84 (2H, t, J = 7.6 Hz), 6.01 (1H, s), 7.18 (2H, d, J = 7.6 Hz), 7.19 (1H, t, J = 7.6 Hz), 7.28 (2H, t, J = 8.0 Hz). ^{13}C-NMR (100MHz, $CDCl_3$) δ 13.78, 13.84, 21.8, 22.1, 31.3, 32.1, 32.5, 33.8, 35.2, 39.1, 125.8, 128.2, 128.3, 128.4, 130.4, 141.2. IR (neat) 3025, 2956, 2927, 2870, 1570, 1495, 1454, 1272, 1223 cm^{-1}. MS (EI) m/z 308 (M^+, 100%), 161 (M^+-147, 94%). HRMS Calcd for $C_{18}H_{28}S_2$: 308.1632. Found: 308.1642.

NOE



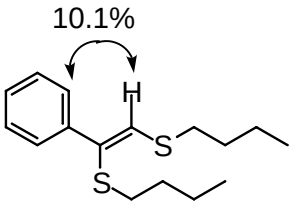
(*Z*)-1,2-Bis(butylthio)-3-phenyl-1-propene. Colorless oil. ^{1}H-NMR (400MHz, $CDCl_3$) δ 0.87 (3H, t, J = 7.2 Hz), 0.91 (3H, t, J = 8.0 Hz), 1.39 (4H, eightet, J = 6.8 Hz), 1.50 (2H, quintet, J = 8.0 Hz), 1.60 (2H, quintet, J = 7. Hz), 2.65 (2H, t, J = 7.2 Hz), 2.68 (2H, t, J = 7.2 Hz), 3.60 (2H, s), 6.06 (1H, s), 7.21 (2H, d, J = 8.0 Hz), 7.22 (1H, t, J = 7.2 Hz), 7.30 (2H, t, J = 7.2 Hz). ^{13}C-NMR (100MHz, $CDCl_3$) δ 13.8, 21.8, 22.0, 31.4, 32.0, 32.5, 33.9, 43.5, 126.3, 128.2, 128.7, 129.4, 130.2, 138.6. IR (neat) 3027, 2956, 2927, 2871, 1494, 1453, 1271, 1222 cm^{-1}. MS (EI) m/z 294 (M^+, 90%), 147 (M^+-147, 100%), 115 (M^+-179, 54%). HRMS Calcd for $C_{17}H_{26}S_2$: 294.1476. Found: 294.1465.

NOE



(*Z*)-1,2-Bis(butylthio)-1-phenylethene. Colorless oil. ^{1}H-NMR (400MHz, $CDCl_3$) δ 0.81 (3H, t, J = 7.6 Hz), 0.94 (3H, t, J = 7.2 Hz), 1.32 (2H, sixtet, J = 7.2 Hz), 1.46 (4H, sixtet, J = 7.6 Hz), 1.68 (2H, quintet, J = 7.6 Hz), 2.50 (2H, t, J = 7.2 Hz), 2.81 (2H, t, J = 7.6 Hz), 6.54 (1H, s), 7.25 (1H, t, J = 7.2 Hz), 7.32 (2H, t, J = 8.0 Hz), 7.48 (2H, d, J = 7.6 Hz). ^{13}C-NMR (100MHz, $CDCl_3$) δ 13.7, 13.8, 21.8, 21.9, 31.9, 32.4, 32.7, 34.1, 127.0, 127.1, 128.2, 130.7, 132.1, 139.2. IR (neat) 3095, 2956, 2927, 2871, 1545, 1274, 815, 754 cm^{-1}. MS (EI) m/z 280 (M^+, 100%), 134 (M^+-146, 52%). HRMS Calcd for $C_{16}H_{24}S_2$: 280.1320. Found: 280.1336.

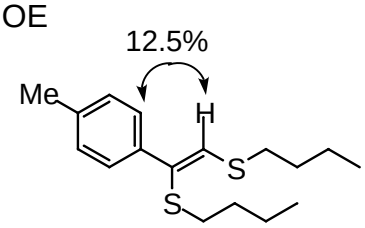
NOE



(*Z*)-1,2-Bis(butylthio)-1-(4-methylphenyl)ethene. Colorless oil. ^{1}H-NMR (400MHz, $CDCl_3$) δ 0.81 (3H, t, J = 7.2 Hz), 0.93 (3H, t, J = 7.2 Hz), 1.33 (2H, sixtet, J = 7.2 Hz), 1.45 (4H, sixtet, J = 7.2 Hz), 1.67 (2H, quintet, J = 7.2 Hz), 2.34 (3H, s), 2.49 (2H, t, J = 7.2 Hz), 2.80 (2H, t, J = 7.2 Hz), 6.48 (1H, s), 7.13 (2H, d, J = 8.4 Hz), 7.37 (2H, d, J = 8.0 Hz). ^{13}C-NMR (100MHz, $CDCl_3$) δ 13.77, 13.81, 21.2, 21.8, 21.9, 31.9, 32.4, 32.7, 34.1, 126.9, 128.9, 130.8, 131.0, 136.3,

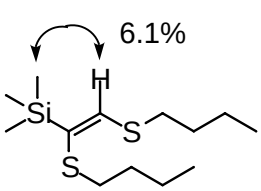
136.9. IR (neat) 2956, 2926, 2870, 1542, 1464, 1274, 1223, 806 cm^{-1}. MS (EI) m/z 294 (M^+, 100%), 148 (M^+-146, 52%). HRMS Calcd for $C_{17}H_{26}S_2$: 294.1476. Found: 294.1475.

NOE



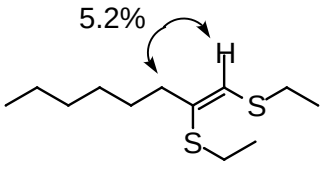
(*Z*)-1,2-Bis(butylthio)-1-(trimethylsilyl)ethene. Colorless oil. ^{1}H-NMR (400MHz, $CDCl_3$) δ 0.16 (9H, s), 0.91 (3H, t, J = 7.6 Hz), 0.93 (3H, t, J = 7.2 Hz), 1.43 (4H, septet, J = 7.2 Hz), 1.56 (2H, quintet, J = 7.6 Hz), 1.65 (2H, quintet, J = 7.2 Hz), 2.74 (2H, t, J = 7.2 Hz), 2.79 (2H, t, J = 7.2 Hz), 6.76 (1H, s). ^{13}C-NMR (100MHz, $CDCl_3$) δ 0.78, 13.8, 13.9, 21.8, 22.1, 31.0, 32.1, 32.6, 33.3, 33.9, 130.5, 144.4. IR (neat) 2957, 2929, 2872, 1511, 1463, 1244, 933, 835 cm^{-1}. MS (EI) m/z 276 (M^+, 56%), 73 (M^+-203, 100%). HRMS Calcd for $C_{13}H_{18}S_2Si_1$: 276.1402. Found: 276.1383.

NOE



(*Z*)-1,2-Bis(ethylthio)-1-octene. Colorless oil. ^{1}H-NMR (400MHz, $CDCl_3$) δ 0.89 (3H, t, J = 6.4 Hz), 1.25 (3H, t, J = 7.2 Hz), 1.26-1.32 (6H, m), 1.31 (3H, t, J = 7.2 Hz), 1.52 (2H, quintet, J = 7.2 Hz), 2.26 (2H, t, J = 8.0 Hz), 2.72 (2H, q, J = 7.2 Hz), 2.73 (2H, q, J = 7.2 Hz), 6.08 (1H, s). ^{13}C-NMR (100MHz, $CDCl_3$) δ 14.2, 15.1, 15.6, 22.7, 25.6, 28.1, 28.7, 28.8, 31.8, 37.1, 126.2, 132.2. IR (neat) 2959, 2926, 2856, 1453, 1257 cm^{-1}. MS (EI) m/z 232 (M^+, 100%), 102 (M^+-130, 54%). HRMS Calcd for $C_{12}H_{24}S_2$: 232.1319. Found: 232.1317.

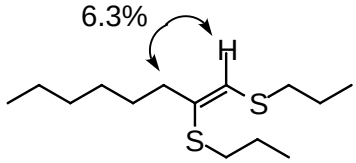
NOE



(*Z*)-1,2-Bis(propylthio)-1-octene. Colorless oil. ^{1}H-NMR (400MHz, $CDCl_3$) δ 0.89 (3H, t, J = 6.8 Hz), 0.998 (3H, t, J = 7.2 Hz), 1.00 (3H, t, J = 7.2 Hz), 1.26-1.33 (6H, m), 1.48-1.55 (2H, m), 1.60 (2H, sixtet, J = 7.6 Hz), 1.66 (2H, sixtet, J = 7.2 Hz), 2.24 (2H, t, J = 7.6 Hz), 2.67 (4H, t, J = 7.2 Hz), 6.05 (1H, s). ^{13}C-NMR (100MHz, $CDCl_3$) δ 13.4, 13.6, 14.2, 22.7, 23.4, 23.8, 28.65, 28.74, 31.7, 33.5, 36.2, 37.0, 126.6, 131.9. IR (neat) 2959, 2928, 2870, 2855, 1456, 1377, 1291,

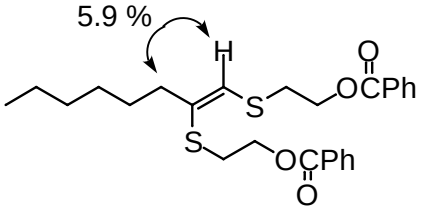
1233 cm^{-1}. MS (EI) m/z 260 (M^+, 100%), 43 (M^+-217, 56%). HRMS Calcd for $C_{14}H_{28}S_2$: 260.1632. Found: 260.1626.

NOE



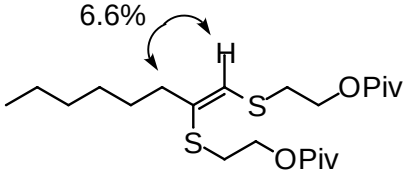
(*Z*)-1,2-Bis{2-(benzoyloxy)ethylthio}-1-octene. Colorless oil. ^{1}H-NMR (400MHz, $CDCl_3$) δ 0.88 (3H, t, J = 7.2 Hz), 1.25-1.29 (6H, m), 1.48-1.52 (2H, m), 2.30 (2H, t, J = 7.2 Hz), 3.04 (2H, t, J = 6.8 Hz), 3.05 (2H, t, J = 6.8 Hz), 4.42 (2H, t, J = 7.2 Hz), 4.48 (2H, t, J = 6.8 Hz), 6.24 (1H, s), 7.43 (4H, t, J = 7.6 Hz), 7.55 (2H, dt, J = 7.6, 2.0 Hz), 8.04 (4H, d, J = 8.0 Hz). ^{13}C-NMR (100MHz, $CDCl_3$) δ 14.2, 22.7, 28.6, 28.7, 30.0, 31.7, 32.4, 37.1, 63.9, 64.3, 128.19, 128.23, 128.4, 129.5, 129.7, 129.9, 131.7, 131.8, 132.9, 133.0, 166.02, 166.04. IR (neat) 3062, 2928, 2855, 1719, 1451, 1270, 1109, 1070, 1026 cm^{-1}. MS (EI) m/z 472 (M^+, 21%), 149 (M^+-323, 100%). HRMS Calcd for $C_{26}H_{32}S_2O_4$: 472.1742. Found: 472.1742.

NOE



(*Z*)-1,2-Bis{2-(pivaloyloxy)ethylthio}-1-octene. Colorless oil. ^{1}H-NMR (400MHz, $CDCl_3$) δ 0.89 (3H, t, J = 7.2 Hz), 1.20 (9H, s), 1.21 (9H, s), 1.26-1.28 (6H, m), 1.49-1.53 (2H, m), 2.28 (2H, t, J = 7.2 Hz), 2.90 (4H, dt, J = 6.8, 2.4 Hz), 4.15 (2H, t, J = 7.6 Hz), 4.22 (2H, t, J = 7.2 Hz), 6.18 (1H, s). ^{13}C-NMR (100MHz, $CDCl_3$) δ 14.2, 22.7, 27.3, 28.6, 28.7, 29.9, 31.7, 32.3, 37.1, 38.8, 63.3, 63.7, 128.3, 131.6, 178.0. IR (neat) 2959, 2931, 2871, 1731, 1480, 1281, 1148, 1032, 971 cm^{-1}. MS (EI) m/z 432 (M^+, 12%), 129 (M^+-303, 100%). HRMS Calcd for $C_{22}H_{40}S_2O_4$: 432.2368. Found: 432.2407.

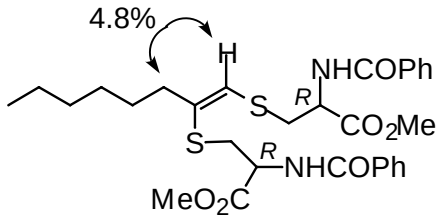
NOE



(*R*,*R*,*Z*)-1,2-Bis{(2-ethoxycarbonyl-2-benzoylamido)ethylthio}-1-octene. Colorless oil. ^{1}H-NMR (400MHz, $CDCl_3$) δ 0.85 (3H, t, J = 7.6 Hz), 1.13-1.19 (4H, m), 1.20-1.26 (2H, m), 1.27-1.34 (2H, m), 2.00-2.09 (2H, m), 3.23 (1H, dd, J = 14.8, 4.4 Hz), 3.27 (1H, dd, J = 12.4, 4.4

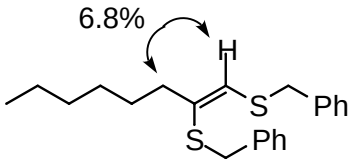
Hz), 3.30 (1H, dd, J = 13.2, 4.4 Hz), 3.36 (1H, dd, J = 14.0, 5.2 Hz), 3.63 (6H, s), 4.90-4.94 (1H, m), 4.99-5.03 (1H, m), 5.93 (1H, s), 7.38-7.43 (4H, m), 7.46-7.51 (1H, m), 7.61 (1H, d, J = 7.6 Hz), 7.86 (4H, t, J = 8.8 Hz). ^{13}C-NMR (100MHz, $CDCl_3$) δ 14.2, 22.6, 28.4, 28.6, 31.6, 32.6, 35.6, 35.9, 52.6, 52.7, 52.9, 53.3, 127.19, 127.24, 128.3, 129.5, 131.0, 131.6, 131.7, 133.3, 133.5, 166.9, 170.4, 170.7. IR (neat) 3328, 2929, 2855, 1746, 1651, 1538, 1488, 1214, 1093, 1027, 913, 802 cm^{-1}. MS (EI) m/z 586 (M^+, 15%), 206 (M^+-380, 72%), 105 (M^+-481, 100%). HRMS Calcd for $C_{30}H_{38}S_2O_6N_2$: 586.2171. Found: 586.2158. $[\alpha]_D^{23}$ 120 (c 1, $CHCl_3$).

NOE



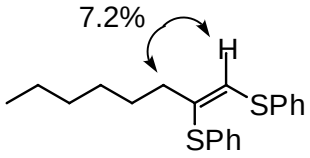
(*Z*)-1,2-Bis(benzylthio)-1-octene. Colorless oil. ^{1}H-NMR (400MHz, $CDCl_3$) δ 0.86 (3H, t, J = 7.2 Hz), 1.15-1.27 (6H, m), 1.38 (2H, quintet, J = 7.2 Hz), 2.09 (2H, t, J = 7.2 Hz), 3.886 (2H, s), 3.892 (2H, s), 6.07 (1H, s), 7.20-7.32 (10H, m). ^{13}C-NMR (100MHz, $CDCl_3$) δ 14.2, 22.7, 28.4, 28.6, 31.7, 36.2, 37.9, 38.2, 126.8, 127.0, 127.2, 128.2, 128.4, 128.72, 132.7, 137.9. IR (neat) 3028, 2926, 2854, 1494, 1453, 1235, 1070, 1029 cm^{-1}. MS (EI) m/z 356 (M^+, 100%). HRMS Calcd for $C_{22}H_{28}S_2$: 356.1632. Found: 356.1630.

NOE



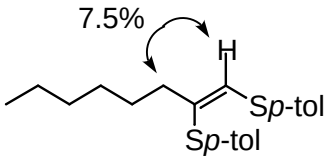
(*Z*)-1,2-Bis(phenylthio)-1-octene.[1] Colorless oil**.** ^{1}H-NMR (400MHz, $CDCl_3$) δ 0.85 (3H, t, J = 7.2 Hz), 1.19–1.28 (6H, m), 1.49 (2H, quintet, J = 7.6 Hz), 2.24 (2H, t, J = 7.6 Hz), 6.56 (1H, s), 7.20–7.34 (6H, m), 7.37 (2H, d, J = 7.6 Hz), 7.41 (2H, d, J = 7.6 Hz). ^{13}C-NMR (100MHz, $CDCl_3$) δ 14.2, 22.6, 28.55, 28.58, 31.58, 37.18, 126.56, 126.64, 128.76, 128.85, 128.90, 129.5, 130.3, 133.6, 134.3, 135.7. IR (neat) 3058, 2927, 2854, 1582, 1477, 1439, 1024, 837 cm^{-1}. MS (EI) m/z 328 (M^+, 100%), 167 (M^+-161, 48%), 147 (M^+-181, 76%). HRMS Calcd for $C_{20}H_{24}S_2$: 328.1320. Found: 328.1319.

NOE



(*Z*)-1,2-Bis(4-methylphenylthio)-1-octene.[2] Colorless oil. ^{1}H-NMR (400MHz, $CDCl_3$) δ 084 (3H, t, J = 7.2 Hz), 1.18-1.27 (6H, m), 1.46 (2H, quintet, J = 7.2 Hz), 2.19 (2H, t, J = 7.6 Hzz), 2.33 (3H, s), 2.34 (3H, s), 6.44 (1H, s), 7.11 (2H, d, J = 8.0 Hz), 7.13 (2H, d, J = 8.0 Hz), 7.28 (2H, d, J = 8.0 Hz), 7.32 (2H, d, J = 8.0 Hz). ^{13}C-NMR (100MHz, $CDCl_3$) δ 14.2, 21.17, 21.23, 22.7, 28.6, 31.6, 36.9, 128.3, 129.5, 129.7, 129.9, 130.0, 131.0, 132.3, 134.2, 136.7. IR (neat) 2925, 2855, 1491, 1456, 1091, 1017 cm^{-1}. MS (EI) m/z 356 (M^+, 100%), 161 (M^+-195, 58%). HRMS Calcd for $C_{22}H_{28}S_2$: 356.1632. Found: 356.1610.

NOE



[1] Kuniyasu, H.; Ogawa, A.; Miyazaki, S-I.; Ryu, I.; Kambe, N.; Sonoda, N. *J. Am. Chem. Soc.*, **1991**, *113*, 9796.

[2] Kuniyasu, H.; Ohtaka, A.; Nakazono, T.; Kinomoto, M.; Kurosawa, H. *J. Am. Chem. Soc.*, **2000**, *122*, 2375.