Supporting Information

1,2-Thio Group Migration in Rh(II) Carbene Reactions

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Experimental Section

Caution: Diazo compounds are potentially explosive. They should be handled with care in a well-ventilated fumehood.

General Information. All reactions with air- and moisture-sensitive components were performed under a nitrogen atmosphere in a flame-dried reaction flask. All solvents were distilled prior to use. CH₂Cl₂ was freshly distilled from CaH₂ and THF was freshly distilled from Na before use. For chromatography, 200-300 mesh silica gel was employed.

Ethyl 2-Diazo-3-(benzylthio)pentanoate (1b): 3 h, 81 %. TLC petroleum ether : ethyl acetate = 30 : 1 R_f = 0.41; IR (film) 2086, 1686, 1285, 1110, 1070, 701 cm⁻¹; ¹H NMR (200 MHz, CDCl₃) δ 0.95 (t, J = 7.6 Hz, 3H), 1.25 (t, J = 7.2 Hz, 3H), 1.60-1.74 (m, 2H), 3.65 (dd, J = 6.6, 8.2 Hz, 1H), 3.74 (s, 2H), 4.20 (q, J = 7.2 Hz, 2H), 7.24-7.32 (m, 5H); ¹³C NMR (50 MHz, CDCl₃) δ 11.7, 14.39, 26.9, 35.8, 41.6, 60.8, 126.9, 128.4, 128.6, 138.1, 166.3; EI-MS (m/z , relative intensity) 250 [(M-28)⁺, 3.90], 204 (14), 159 (8), 91 (100), 83 (37), 69 (42), 57 (83). Anal. calcd for C₁₄H₁₈N₂O₂S: C, 60.41; H, 6.52; N, 10.06. Found: C, 60.54; H, 6.54; N, 10.25.

Ethyl 2-Diazo-3-(phenylthio)nonanoate (1c): 8 h, 55 %. TLC petroleum ether : ethyl acetate = 30 : 1 R_f = 0.51; IR (film) 2085, 1692, 1290, 1121, 1095, 743, 692 cm⁻¹; ¹H NMR (200 MHz, CDCl₃) δ 0.87 (t, J = 6.3 Hz, 3H), 1.11-1.45 (m, 11H), 1.64-1.79 (m, 2H), 4.05-4.14 (m, 3H), 7.22-7.47 (m, 5H); ¹³C NMR (50 MHz, CDCl₃) δ 13.96, 14.36, 22.47, 27.20, 28.65, 31.52, 33.22, 43.79, 60.84, 127.83, 128.85, 133.17, 133.41; EI-MS (m/z , relative intensity) 292 [(M-28)⁺, 25], 246 (41), 211 (17), 189 (36), 155 (38), 109 (100), 95 (29), 67 (53). Anal. calcd for C₁₇H₂₄N₂O₂S: C, 63.72; H, 7.55; N, 8.74. Found: C, 64.07; H, 7.45; N, 8.46.

Ethyl 2-Diazo-3-(benzylthio)nonanoate (1d): 2 h, 65 %. TLC petroleum ether : ethyl acetate = 30 : 1 R_f = 0.56; IR (film) 2086, 1689, 1288, 1095, 701 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 0.85 (t, J = 6.6 Hz, 3H), 1.23-1.34 (m, 11H), 1.55-1.68 (m, 2H), 3.69-3.74 (m, 3H), 4.19 (q, J = 7.2 Hz, 2H), 7.24-7.34 (m, 5H); ¹³C NMR (50 MHz, CDCl₃) δ 13.9, 14.3, 22.4, 26.9, 28.5, 31.4, 33.4, 35.8, 39.9, 60.2, 126.8, 128.3, 128.6, 138.1, 166.3; EI-MS (m/z , relative intensity) 306 [(M-28)⁺, 3], 260 (11), 169 (12), 109 (14), 91 (100), 65 (10), 41 (14). Anal. calcd for C₁₈H₂₈N₂O₂S: C, 64.64; H, 7.84; N, 8.38. Found: C, 64.92; H, 7.73; N, 8.28.

2-Diazo-3-(phenylthio)-2-hexanone (1e): 4 h, 87 %. TLC petroleum ether : ethyl acetate = 30 : 1 R_f = 0.11; IR (film) 2071, 1641, 1323, 1287, 745, 692 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 1.03 (t, J = 7.2 Hz, 3H), 1.65-1.89 (m, 2H), 4.29 (s, 1H), 7.28-7.41 (m, 5H); ^{13}C NMR (75 MHz, CDCl_3) δ 11.7, 25.2, 26.2, 43.1, 127.5, 128.8, 132.3, 133.1, 189.2; EI-MS (m/z , relative intensity): 234 (M^+ , 0.49), 206 $[(\text{M}-28)^+$, 3], 163 (1), 135 (3), 125 (12), 110 (25), 97 (12), 77 (4), 65 (9), 43 (100). Anal. calcd for $\text{C}_{12}\text{H}_{14}\text{N}_2\text{OS}$: C, 61.51; H, 6.02; N, 11.96. Found: C, 65.02; H, 5.78; N, 8.95.

2-Diazo-3-(benzylthio)-2-hexanone (1f): 2 h, 82 %. TLC petroleum ether : ethyl acetate = 30 : 1 R_f = 0.12; IR (film) 2072, 1637, 1366, 1287, 703 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 0.93 (t, J = 7.2 Hz, 3H), 1.53-1.73 (m, 2H), 2.04 (s, 3H), 3.70 (dd, J = 13.7, 18.6 Hz, 2H), 3.90 (s, 1H), 7.24-7.31 (m, 5H); ^{13}C NMR (75 MHz, CDCl_3) δ 11.5, 25.3, 26.4, 36.0, 40.7, 126.8, 128.2, 128.3, 138.1, 189.2; EI-MS (m/z , relative intensity) 220 $[(\text{M}-28)^+$, 5], 177 (3), 129 (17), 97 (6), 91 (100). Anal. calcd for $\text{C}_{13}\text{H}_{16}\text{N}_2\text{OS}$: C, 62.87; H, 6.49; N, 11.28. Found: C, 62.82; H, 6.60; N, 11.10.

2-Diazo-3-(phenylthio)-1-phenyl-2-pentanone (1g): 4 h, 68 %. TLC petroleum ether : ethyl acetate = 30 : 1 R_f = 0.18; IR (film) 2073, 1607, 1348, 1327, 755, 697 cm^{-1} ; ^1H NMR (200 MHz, CDCl_3) δ 1.11 (t, J = 7.4 Hz, 3H), 1.72-1.97 (m, 2H), 4.49 (t, J = 7.3 Hz, 1H), 7.23-7.48 (m, 10H); ^{13}C NMR (50 MHz, CDCl_3) δ 11.8, 26.2, 44.5, 126.7, 127.7, 128.4, 128.9, 131.3, 132.8, 133.0, 137.3; EI-MS (m/z , relative intensity) 268 $[(\text{M}-28)^+$, 22], 159 (82), 131 (87), 105 (100), 91 (42), 77 (61). Anal. calcd for $\text{C}_{17}\text{H}_{16}\text{N}_2\text{OS}$: C, 68.89; H, 5.44; N, 9.45. Found: C, 69.19; H, 5.58; N, 9.13.

2-Diazo-3-(benzylthio)-1-phenyl-2-pentanone (1h): 4.5 h, 65 %. TLC petroleum ether : ethyl acetate = 30 : 1 R_f = 0.26; IR (film) 2071, 1604, 1334, 713, 697 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 1.01 (t, J = 7.5 Hz, 3H), 1.63-1.82 (m, 2H), 3.78 (dd, J = 13.8, 20.1 Hz, 2H), 4.12 (t, J = 7.2 Hz, 1H), 7.18-7.51 (m, 10H); ^{13}C NMR (50 MHz, CDCl_3) δ 11.7, 26.5, 36.1, 41.8, 126.9, 127.6, 128.4, 128.5, 129.0, 131.5, 137.4, 138.1, 187.8; EI-MS (m/z , relative intensity) 282 $[(\text{M}-28)^+$, 20], 253 (6), 191 (75), 147 (19), 131 (59), 105 (78), 91 (100), 77 (55). Anal. calcd for $\text{C}_{18}\text{H}_{18}\text{N}_2\text{OS}$: C, 69.65; H, 5.84; N, 9.02. Found: C, 69.77; H, 5.76; N, 9.01.

Ethyl 2-Diazo-3-(naphthylthio)pentanoate (1i): 6 h, 57 %. TLC petroleum ether : ethyl

acetate = 30 : 1 R_f = 0.46; IR (film) 2086, 1690, 1286, 1116, 813, 744 cm^{-1} ; ^1H NMR (200 MHz, CDCl_3) δ 1.03-1.12 (m, 6H), 1.72-1.90 (m, 2H), 4.00-4.18 (m, 3H), 7.43-7.53 (m, 3H), 7.73-7.91 (m, 4H); ^{13}C NMR (50 MHz, CDCl_3) δ 11.9, 14.2, 26.6, 45.1, 60.8, 126.2, 126.4, 127.4, 127.5, 128.3, 129.9, 130.6, 131.9, 132.4, 133.5, 166.0; EI-MS (m/z , relative intensity) 286 $[(\text{M}-28)^+$, 6], 257 (4), 240 (14), 179 (15), 160 (98), 159 (32), 155 (49), 127 (36), 115 (100), 99 (65). Anal. calcd for $\text{C}_{17}\text{H}_{18}\text{N}_2\text{O}_2\text{S}$: C, 64.94; H, 5.77; N, 8.91. Found: C, 65.02; H, 5.78; N, 8.95.

Ethyl 2-Diazo-3-phenyl-3-(benzylthio)propionate (1k): 68 %. TLC petroleum ether : ethyl acetate = 30 : 1 R_f = 0.36; IR (film) 2087, 1688, 1243, 1098, 704 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 1.24 (t, J = 7.2 Hz, 3H), 3.72 (dd, J = 13.5, 18.3 Hz, 2H), 4.20 (q, J = 7.2 Hz, 2H), 4.86 (s, 1H), 7.25-7.36 (m, 10H); ^{13}C NMR (75 MHz, CDCl_3) δ 14.3, 36.7, 42.9, 61.0, 127.0, 127.1, 127.9, 128.4, 128.7, 128.7, 137.3, 138.3, 165.5; EI-MS (m/z , relative intensity) 298 $[(\text{M}-28)^+$, 14], 203 (10), 175 (8), 147 (15), 135 (17), 103 (13), 91 (100), 77 (7), 65 (11), 45 (9), 29 (30). Anal. calcd for $\text{C}_{18}\text{H}_{18}\text{N}_2\text{O}_2\text{S}$: C, 66.23; H, 5.56; N, 8.58. Found: C, 66.34; H, 5.71; N, 8.44.

Ethyl 2-Diazo-3-(*p*-phenyl)phenyl-3-(phenylthio) propionate (1l): 40 %. TLC petroleum ether : ethyl acetate = 30 : 1 R_f = 0.33; IR (film) 2087, 1689, 1291, 1240, 1099, 746, 691 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 1.18 (t, J = 7.2 Hz, 3H), 4.10-4.18 (m, 2H), 5.42 (s, 1H), 7.24-7.61 (m, 14H); ^{13}C NMR (75 MHz, CDCl_3) δ 14.3, 46.7, 61.0, 126.9, 127.4, 127.5, 127.6, 127.7, 128.7, 128.9, 132.1, 133.5, 137.0, 140.2, 141.0, 165.4; EI-MS (m/z , relative intensity) 360 $[(\text{M}-28)^+$, 100], 315 (8), 287 (43), 251 (27), 223 (30), 211 (33), 197 (12), 178 (54), 165 (19), 152 (16), 125 (11), 110 (13), 69 (14), 51 (7), 29 (26). Anal. calcd for $\text{C}_{23}\text{H}_{20}\text{N}_2\text{O}_2\text{S}$: C, 71.11; H, 5.19; N, 7.21. Found: C, 71.28; H, 5.20; N, 6.94.

Ethyl 2-Diazo-3-(*p*-methoxy)phenyl-3-(phenylthio)propionate (1m): 47 %. TLC petroleum ether : ethyl acetate = 30 : 1 R_f = 0.19; IR (film) 2087, 1693, 1508, 1250, 1173, 1032, 830, 744, 690 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 1.18 (t, J = 7.2 Hz, 3H), 3.79 (s, 3H), 4.08-4.16 (m, 2H), 5.34 (s, 1H), 6.87-6.90 (m, 2H), 7.26-7.46 (m, 7H); ^{13}C NMR (75 MHz, CDCl_3) δ 14.3, 46.4, 55.2, 61.0, 114.2, 127.7, 128.4, 128.9, 130.0, 132.1, 133.8, 159.3; EI-MS (m/z , relative intensity) 314 $[(\text{M}-28)^+$, 100], 269 (10), 241 (38), 205 (52), 177 (73), 165 (78), 149 (18), 135 (56), 110 (45). Anal. calcd for $\text{C}_{18}\text{H}_{18}\text{N}_2\text{O}_3\text{S}$: C, 63.14; H, 5.30; N, 8.18. Found: C, 63.15; H, 5.49; N, 7.95.

(Z)-Ethyl 2-(Benzylthio)-2-pentenoate (2Zb): 12 h, 41 %. TLC petroether : ethyl acetate = 30 : 1 R_f = 0.47; IR (film) 1712, 1260, 1233, 1133, 1044, 753, 699 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 0.80 (t, J = 7.5 Hz, 3H), 1.35 (t, J = 7.2 Hz, 3H), 2.11 (m, 2H), 3.92 (s, 2H), 4.27 (q, J = 7.2 Hz, 2H), 7.14-7.29 (m, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 12.4, 14.2, 23.9, 38.1, 61.2, 125.6, 126.8, 128.2, 128.8, 138.1, 154.3, 165.4; EI-MS (m/z , relative intensity) 250 (M^+ , 7), 204 (23), 159 (7), 131 (11), 91 (100). Anal. calcd for $\text{C}_{14}\text{H}_{18}\text{O}_2\text{S}$: C, 67.16; H, 7.25. Found: C, 66.77; H, 7.25.

(Z)-Ethyl 2-(Phenylthio)-2-nonenoate (2Zc): 10 h, 83 %. TLC petroleum ether : ethyl acetate = 30 : 1 R_f = 0.51; IR (film) 2927, 1713, 1478, 1241, 1044, 739, 690 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 0.87 (t, J = 6.7 Hz, 3H), 1.07 (t, J = 7.2 Hz, 3H), 1.27-1.36 (m, 6H), 1.47-1.52 (m, 2H), 2.49-2.56 (m, 2H), 4.12 (q, J = 7.2 Hz, 2H), 7.15-7.27 (m, 5H), 7.40 (t, J = 7.8 Hz, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ 13.8, 13.9, 22.4, 28.3, 28.9, 30.7, 31.5, 61.3, 125.8, 126.5, 128.0, 128.8, 136.0, 153.2, 165.3; EI-MS (m/z , relative intensity) 292 (M^+ , 83), 246 (100), 218 (9), 189 (79), 175 (37), 147 (69), 135 (54), 109 (71), 91 (22), 67 (55). Anal. calcd for $\text{C}_{17}\text{H}_{24}\text{O}_2\text{S}$: C, 69.82; H, 8.27. Found: C, 69.85; H, 8.13.

(Z)-Ethyl 2-(Benzylthio)-2-nonenoate (2Zd): 19 h, 49 %. TLC petroleum ether : ethyl acetate = 30 : 1 R_f = 0.53; IR (film) 2927, 1713, 1240, 1045, 690 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 0.86 (t, J = 7.1 Hz, 3H), 1.17-1.27 (m, 8H), 1.34 (t, J = 7.1 Hz, 3H), 2.15-2.24 (m, 2H), 2.22 (s, 2H), 4.19 (q, J = 7.1 Hz, 2H), 7.14-7.28 (m, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 13.9, 14.2, 22.4, 28.0, 28.8, 30.5, 31.4, 38.1, 61.2, 126.1, 126.8, 128.2, 128.8, 138.1, 153.2, 165.4; EI-MS (m/z , relative intensity) 306 (M^+ , 55), 260 (83), 215 (59), 169 (84), 141 (63), 107 (22), 91 (100), 71 (46), 65 (45), 29 (37). Anal. calcd for $\text{C}_{18}\text{H}_{26}\text{O}_2\text{S}$: C, 70.54; H, 8.55. Found: C, 70.76; H, 8.63.

(Z)-3-(Phenylthio)-3-ene-2-hexanone (2Ze): 2 h, 72 %. TLC petroleum ether : ethyl acetate = 30 : 1 R_f = 0.35; IR (film) 1687, 1592, 1478, 1356, 1209, 1024, 740, 690 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 1.11 (t, J = 7.5 Hz, 3H), 2.29 (s, 3H), 2.47-2.57 (m, 2H), 7.15-7.29 (m, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 12.7, 20.0, 27.3, 125.8, 127.3, 129.0, 133.6, 135.6, 153.1, 197.5; EI-MS (m/z , relative intensity) 206 (M^+ , 99), 135 (59), 129 (72), 121 (89), 109 (40), 91 (35), 85 (71), 43 (100). Anal. calcd for $\text{C}_{12}\text{H}_{14}\text{O}_2\text{S}$: C, 69.86; H, 6.84. Found: C, 69.62; H, 6.76.

(Z)-3-(Benzylthio)-3-ene-2-hexanone (2Zf): 11 h, 54 %. TLC petroether : ethyl acetate = 30 : 1 R_f = 0.26; IR (film) 1677, 1454, 1356, 1227, 1210, 767, 700 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 0.85 (t, J = 7.5 Hz, 3H), 2.24-2.34 (m, 5H), 3.85 (s, 2H), 6.95 (t, J = 7.1 Hz, 1H), 7.15-7.28 (m, 5H); ^{13}C NMR (75 MHz, CDCl_3) δ 12.5, 24.2, 26.6, 37.6, 126.8, 128.1, 128.8, 135.2, 138.1, 153.7, 196.7; EI-MS (m/z , relative intensity) 220 (M^+ , 16), 129 (8), 91 (100), 85 (4), 65 (10), 43 (18). Anal. calcd for $\text{C}_{13}\text{H}_{16}\text{O}_2\text{S}$: C, 70.87; H, 7.32. Found: C, 70.43; H, 7.29.

(Z)-Ethyl 2-(Naphthylthio)-2-pentenoate (2Zi): 3.5 h, 52 %. TLC petroleum ether : ethyl acetate = 30 : 1 R_f = 0.59; IR (film) 1712, 1236, 1133, 1043, 813, 745 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 1.04 (t, J = 7.1 Hz, 3H), 1.11 (t, J = 7.5 Hz, 3H), 2.52-2.62 (m, 2H), 4.10 (q, J = 7.2 Hz, 2H), 7.31-7.51 (m, 4H), 7.65-7.78 (m, 4H); ^{13}C NMR (75 MHz, CDCl_3) δ 12.8, 13.9, 24.2, 61.4, 125.5, 126.0, 126.1, 126.1, 126.4, 127.0, 127.6, 128.4, 131.6, 133.3, 133.6, 154.8, 165.4; EI-MS (m/z , relative intensity) 286 (M^+ , 65), 257 (3), 240 (44), 211 (20), 155 (100), 128 (53), 115 (51). Anal. calcd for $\text{C}_{17}\text{H}_{18}\text{O}_2\text{S}$: C, 71.30; H, 6.34. Found: C, 71.31; H, 6.44.

Ethyl 3-(*p*-Phenyl)phenyl-2-(phenylthio)propionate (8k): 95 %. TLC petroleum ether : ethyl acetate = 30 : 1 R_f = 0.33; IR (film) 1730, 1486, 1257, 1151, 1025, 822, 747, 693 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 1.05 (t, J = 7.2 Hz, 3H), 3.06-3.28 (AB part of ABX, J_{AB} = 13.8 Hz, 2H), 3.91-3.96 (X part of ABX, J_{AX} = 6.3 Hz, J_{BX} = 9.3 Hz, 1H), 3.99-4.08 (m, 2H), 7.25-7.57 (m, 14H); ^{13}C NMR (75 MHz, CDCl_3) δ 13.8, 37.4, 52.0, 61.0, 126.8, 127.0, 127.1, 127.9, 128.6, 128.8, 129.3, 133.0, 136.6, 139.6, 140.6, 171.4; EI-MS (m/z , relative intensity) 362 (M^+ , 8), 289 (2), 252 (56), 224 (5), 207 (27), 180 (24), 167 (100), 152 (15), 135 (10), 126 (11), 110 (9), 89 (7), 78 (21), 69 (13), 43(16), 29 (13). Anal. calcd for $\text{C}_{23}\text{H}_{22}\text{O}_2\text{S}$: C, 76.21; H, 6.12. Found: C, 76.22; H, 6.20.

Ethyl 3-(Phenyl)-2-(benzylthio)propionate (8l): 87 %. TLC petroleum ether : ethyl acetate = 30 : 1 R_f = 0.42; IR (film) 1727, 1494, 1454, 1259, 1149, 1030, 747, 698 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 1.17 (t, J = 7.2 Hz, 3H), 2.85-3.19 (m, AB part of ABX, J_{AB} = 13.8 Hz, 2H), 3.39-3.44 (X part of ABX, J_{AX} = 6.6 Hz, J_{BX} = 9.0 Hz, 1H), 3.74-3.85 (m, 2H), 7.07-7.10 (m, 2H), 7.18-7.35 (m, 8H); ^{13}C NMR (75 MHz, CDCl_3) δ 14.0, 36.0, 37.4, 47.2, 60.9, 126.5, 127.0, 128.2, 128.3, 128.9, 137.2, 137.8, 171.8; EI-MS (m/z , relative intensity) 300 (M^+ , 4), 227 (2), 193 (2), 178 (51), 135 (9), 123 (23), 104 (21), 91 (100), 78 (9), 65 (10), 51 (2), 45 (6), 29 (7). Anal. calcd for $\text{C}_{18}\text{H}_{20}\text{O}_2\text{S}$: C, 71.96; H, 6.71. Found: C, 71.90; H, 6.73.

Ethyl 3-(*p*-nitro)phenyl-3-(phenylthio)propionate (7): 67 %. TLC petroleum ether : ethyl acetate = 30 : 1 R_f = 0.15; IR (film) 1732, 1520, 1345, 1152, 856, 748, 693 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 1.18 (t, J = 7.3 Hz, 3H), 2.88-3.07 (AB part of ABX, J_{AB} = 15.9 Hz, 2H), 4.04-4.12 (m, 2H), 4.65-4.71 (X part of ABX, J_{AX} = 6.9 Hz, J_{BX} = 8.7 Hz, 1H), 7.24-7.29 (m, 7H), 8.09-8.13 (m, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 14.0, 40.1, 48.6, 61.0, 123.6, 124.1, 128.5, 128.5, 129.0, 132.1, 133.9, 148.3, 170.0; EI-MS (m/z , relative intensity) 331 (M^+ , 13), 221 (26), 193 (51), 176 (100), 149 (47), 130 (49), 110 (35), 102 (36), 29 (54). Anal. calcd for $\text{C}_{17}\text{H}_{17}\text{NO}_4\text{S}$: C, 61.61; H, 5.17; N, 4.23. Found: C, 61.41; H, 5.21; N 4.37.