Supporting Information

Nickel-Catalyzed [3+1+1] Cycloaddition Reactions of Alkenyl Fischer Carbene Complexes with Methylenecyclopropanes

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General Procedure for nickel catalyzed [3+1+1] cycloaddition reactions of alkenyl Fischer carbene complexes with methylenecyclopropanes

To a solution of α,β -unsaturated Fischer carbene complex **3** (or **6**) (0.10 mmol) and MCP (0.10 mmol) in DMF (2.0 mL), Ni(cod)₂ (0.010 mmol) in DMF (1.0 mL) was added to the solution by cannula at 0 °C under argon. The resulting solution was stirred for 1.5 h at 0 °C. The solution was extracted with ether, and organic layer was washed with brine, dried over MgSO₄, filtrate and evaporated under reduced pressure. The residue was purified by silica gel to give the cycloaddition product.

Compound 4

¹H NMR (500 MHz, CDCl₃) δ 2.39-2.46 (1H, m), 2.59-2.66 (1H, m), 2.93-3.01 (1H, m), 3.07-3.14 (1H, m), 3.34 (3H, s), 6.18 (1H, d, J = 16.0 Hz), 6.65 (1H, d, J = 16.0 Hz), 6.72 (1H, t, J = 2.2 Hz), 7.23- 7.27 (1H, m), 7.29-7.32 (3H, m), 7.38-7.44 (6H, m); ¹³C NMR (126 MHz, CDCl₃) δ 24.5, 35.5, 52.9, 87.2, 127.0, 127.3, 127.6, 128.3, 128.4, 128.6, 128.7, 129.0, 134.1, 136.0, 136.5, 138.1, 214.2; IR (CHCl₃) 2937, 1747, 1634, 1449 cm⁻¹; MS (relative intensity) m/z 304 (M⁺, 64), 276 (99), 245 (100), 215 (68), 185 (75) ; HRMS calcd for C₂₁H₂₀O₂ 304.1463. Found: 304.1461.

Compound 5

^1H NMR (500 MHz, CDCl_3) δ 2.34-2.39 (1H, m), 2.46-2.52 (1H, m), 2.78-2.85 (1H, m), 2.92-2.98 (1H, m), 3.34 (3H, s), 6.36 (1H, d, $J = 16.0$ Hz), 6.44 (1H, t, $J = 2.5$ Hz), 6.70 (1H, d, $J = 16.0$ Hz), 7.20-7.23 (1H, m), 7.25-7.34 (7H, m), 7.41-7.43 (2H, m); ^{13}C NMR (126 MHz, CDCl_3) δ 25.8, 29.6, 51.5, 85.0, 123.1, 123.2, 126.7, 127.7, 128.0, 128.5, 128.6, 130.3, 136.8, 137.0, 141.6, 144.3; IR (CHCl_3) 2927, 2852, 1491, 1448, 1418 cm^{-1} ; MS (relative intensity) m/z 276 (M^+ , 69), 245 (63), 215 (32), 185 (42), 115 (95), 91 (100); HRMS calcd for $\text{C}_{20}\text{H}_{20}\text{O}$ 276.1514. Found: 276.1514.

Complex 7a

^1H NMR (400 MHz, CDCl_3) δ 1.75 (3H, dd, $J = 1.4, 6.5$ Hz), 2.30-2.37 (1H, m), 2.53-2.60 (1H, m), 2.83-2.91 (1H, m), 3.00-3.07 (1H, m), 3.28 (3H, s), 5.48 (1H, dd, $J = 1.4, 15.4$ Hz), 5.80 (1H, dq, $J = 6.5, 15.4$ Hz), 6.61 (1H, t, $J = 2.3$ Hz), 7.26-7.30 (1H, m), 7.37-7.42 (4H, m); ^{13}C NMR (100 MHz, CDCl_3) δ 18.1, 24.2, 35.2, 52.6, 87.2, 127.3, 127.5, 128.5, 128.8, 129.5, 131.4, 136.5, 138.2, 214.7; IR (CHCl_3) 2990, 2932, 1750, 1560, 1464 cm^{-1} ; MS (relative intensity) m/z 242 (M^+ , 22), 214 (26), 199 (100); HRMS calcd for $\text{C}_{16}\text{H}_{18}\text{O}_2$ 242.1307. Found: 242.1307.

Complex 7b

^1H NMR (400 MHz, CDCl_3) δ 2.37-2.46 (1H, m), 2.58-2.67 (1H, m), 2.94-3.00 (1H, m), 3.07-3.17 (1H, m), 3.35 (3H, s), 4.11 (5H, s), 4.25 (2H, s), 4.35-4.37 (2H, m), 5.78 (1H, d, $J = 15.8$ Hz), 6.43 (1H, d, $J = 15.8$ Hz), 6.71 (1H, s), 7.31-7.34 (1H, m), 7.41-7.47 (4H, m); ^{13}C NMR (100 MHz, CDCl_3) δ 24.5, 35.3, 52.8, 67.3, 67.5, 69.3, 69.4, 81.4, 87.2, 124.2, 127.5, 127.8, 128.6, 129.0, 133.2, 136.6, 138.5, 214.3; IR (CHCl_3) 3155, 2928, 2850, 1748, 1635, 1601, 1464 cm^{-1} ; MS (relative intensity) m/z 412 (M^+ , 13), 380 (11), 279 (6), 256 (6), 149 (43), 69 (100) ; HRMS calcd for $\text{C}_{25}\text{H}_{24}\text{O}_2\text{Fe}$ 412.1125. Found: 412.1125.

Complex 7c

^1H NMR (500 MHz, CDCl_3) δ 2.43-2.50 (1H, m), 2.59-2.66 (1H, m), 2.96-3.03 (1H, m), 3.05-3.13 (1H, m), 3.31 (3H, s), 5.24 (1H, t, $J = 6.4$ Hz), 5.34 (1H, d, $J = 6.4$ Hz), 5.41 (3H, m), 6.08 (1H, d, $J = 6.1$ Hz), 6.23 (1H, d, $J = 6.1$ Hz), 6.72 (1H, t, $J = 2.5$ Hz), 7.29-7.33 (1H, m), 7.38-7.45 (4H, m); ^{13}C NMR (126 MHz, CDCl_3) δ 24.46, 35.50, 52.81, 86.50, 89.75, 90.80, 91.08, 93.26, 172.72, 128.56, 128.99, 129.25, 129.51, 130.45, 136.06, 137.34, 213.58, 232.66; IR (CHCl_3) 3033, 3004, 2957, 1973, 1902, 1632, 1605, 1452, 1452, 1072 cm^{-1} ; MS (relative intensity) m/z 440 (M^+ , 19), 356 (84), 326 (94), 324 (57), 215 (45), 52 (100) ; HRMS calcd for $\text{C}_{24}\text{H}_{20}\text{O}_5\text{Cr}$ 440.0716. Found: 440.0716.

Compound 9a

^1H NMR (400 MHz, CDCl_3) δ 2.40 (3H, s), 2.40-2.49 (1H, m), 2.59-2.68 (1H, m), 2.94-3.02 (1H, m), 3.07-3.16 (1H, m), 3.35 (3H, s), 6.20 (1H, d, $J = 16.1$ Hz), 6.67 (1H, d, $J = 16.1$ Hz), 6.70 (1H, t, $J = 2.5$ Hz), 7.22-7.42 (9H, m); ^{13}C NMR (100 MHz,

CDCl₃) δ 21.2, 24.4, 35.4, 52.7, 87.1, 126.9, 127.3, 128.2, 128.3, 128.6, 128.9, 129.2, 133.6, 133.9, 135.9, 136.9, 137.4, 214.3; IR (CHCl₃) 3365, 3002, 2957, 1746, 1602, 1573, 1450 cm⁻¹; MS (relative intensity) m/z 318 (M⁺, 62), 290 (34), 275 (50), 259 (35), 119 (100) ; HRMS calcd for C₂₂H₂₂O₂ 318.1620. Found: 318.1617.

Compound 9b

¹H NMR (400 MHz, CDCl₃) δ 2.40-2.49 (1H, m), 2.58-2.67 (1H, m), 2.91-3.00 (1H, m), 3.05-3.13 (1H, m), 3.34 (3H, s), 3.85 (3H, s), 6.19 (1H, d, J = 16.1 Hz), 6.65 (1H, d, J = 16.1 Hz), 6.66 (1H, t, J = 2.5 Hz), 6.95 (2H, d, J = 8.8 Hz), 7.26-7.33 (3H, m), 7.37-7.41 (4H, m); IR (CHCl₃), 3005, 2958, 2935, 1746, 1606, 1152, 251 cm⁻¹; MS (relative intensity) m/z 334 (M⁺, 91), 306 (65), 275 (100); HRMS calcd for C₂₂H₂₂O₃ 334.1569. Found: 334.1569.

Compound 9c

¹H NMR (400 MHz, CDCl₃) δ 2.31-2.40 (1H, m), 2.56-2.65 (1H, m), 2.82-2.97 (2H, m), 2.93 (6H, s), 3.04 (3H, s), 6.23 (1H, d, J = 16.1 Hz), 6.61 (2H, d, J = 8.6 Hz), 6.77 (1H, d, J = 16.1 Hz), 6.81 (1H, s), 7.19-7.30 (3H, m), 7.35-7.37 (2H, m), 7.49 (2H, d, J = 8.6 Hz); ¹³C NMR (100 MHz, CDCl₃) δ 28.94, 35.53, 40.15, 87.53, 111.42, 123.28, 125.93, 126.86, 128.05, 128.42, 129.69, 131.02, 131.35, 133.35, 136.13, 149.52, 215.39; IR (CHCl₃) 2997, 2958, 2927, 2858, 1744, 1605 cm⁻¹; MS (relative intensity) m/z 347 (M⁺, 100), 333 (17), 316 (53), 304 (23), 288 (52) ; HRMS calcd for C₂₃H₂₅NO₂ 347.1885. Found: 347.1881.

Compound 9d

^1H NMR (400 MHz, CDCl_3) δ 2.40-2.49 (1H, m), 2.59-2.68 (1H, m), 2.89-2.99 (1H, m), 3.02-3.10 (1H, m), 3.35 (3H, s), 6.17 (1H, d, $J = 16.1$ Hz), 6.64 (1H, d, $J = 16.1$ Hz), 6.68 (1H, t, $J = 2.3$ Hz), 7.26-7.41 (9H, m); ^{13}C NMR (100 MHz, CDCl_3) δ 24.3, 35.3, 52.9, 87.0, 126.9, 127.0, 128.5, 128.6, 128.7, 130.1, 131.0, 133.3, 134.1, 134.8, 135.8, 139.0, 213.7; IR (CHCl_3) 2994, 2954, 2928, 1731, 1599, 1044, 905 cm^{-1} ; MS (relative intensity) m/z 338 (M^+ , 73), 310 (76), 279 (99), 215 (68), 91 (100); HRMS calcd for $\text{C}_{21}\text{H}_{19}\text{O}_2\text{Cl}$ 338.1073. Found: 338.1075.

Compound 9e

^1H NMR (400 MHz, CDCl_3) δ 2.41-2.50 (1H, m), 2.61-2.70 (1H, m), 2.94-3.02 (1H, m), 3.05-3.14 (1H, m), 3.38 (3H, s), 6.18 (1H, d, $J = 16.0$ Hz), 6.65 (1H, d, $J = 16.0$ Hz), 6.76 (1H, t, $J = 2.9$ Hz), 7.27- 7.34 (3H, m), 7.39- 7.42 (2H, m), 7.53 (2H, d, $J = 8.6$ Hz), 7.67 (2H, d, $J = 8.6$ Hz); ^{13}C NMR (100 MHz, CDCl_3) δ 24.3, 35.4, 53.0, 86.9, 125.4, 125.5, 126.7, 126.8, 126.9, 128.5, 128.6, 128.7, 129.0, 134.2, 135.7, 141.3, 213.4; IR (CHCl_3) 3029, 3007, 2933, 1748, 1325, 1130 cm^{-1} ; MS (relative intensity) m/z 372 (M^+ , 30), 344 (33), 313 (46), 275 (20), 81 (59), 69 (100) ; HRMS calcd for $\text{C}_{22}\text{H}_{19}\text{O}_2\text{F}_3$ 372.1337. Found: 372.1329.

Compound 9f

^1H NMR (400 MHz, CDCl_3) δ 1.72 (9H, s), 2.46-2.56 (1H, m), 2.65-2.74 (1H, m), 2.91-3.00 (1H, m), 3.03-3.11 (1H, m), 3.37 (3H, s), 6.25 (1H, d, $J = 16.2$ Hz), 6.66 (1H, d, $J = 16.2$ Hz), 6.90 (1H, s), 7.26-7.41 (7H, m), 7.68 (1H, d, $J = 7.6$ Hz), 7.74 (1H, s), 8.15 (1H, d, $J = 7.8$ Hz); ^{13}C NMR (100 MHz, CDCl_3) δ 25.1, 28.1, 34.8, 52.7, 84.2, 87.1, 115.1, 116.7, 117.6, 118.9, 122.9, 123.5, 124.9, 126.8, 127.4, 128.3, 128.5, 130.0, 133.9, 134.6, 135.8, 137.7, 149.7, 214.1; IR (CHCl_3) 3689, 2992, 2959, 2929, 1734, 1601, 1556, 1454, 1371 cm^{-1} ; MS (relative intensity) m/z 443 (M^+ , 22), 414 (22), 387 (33), 370 (23), 329 (28), 57 (100); HRMS calcd for $\text{C}_{28}\text{H}_{29}\text{NO}_4$ 443.2097. Found: 443.2098.

Synthesis of bis cyclopentanpyridazine derivative **18**

A solution of **4** (30 mg, 0.10 mmol), *p*-toluenesulfonylhydrazine (22 mg, 0.12 mmol) in methanol (5.0 mL) was heated at 70 °C and stirred for 5h. Colorless crystals **18** were precipitated and filtrate. Mother liquor was concentrated and purified by silica gel to give the second crop of **18** (total amount 30 mg, 0.034 mmol, 68%).

Compound **18**

Mp 189 °C; ^1H NMR (400 MHz, CDCl_3) δ 1.98-2.06 (2H, m), 2.33 (6H, s), 2.46-2.53 (2H, m), 2.68-2.75 (2H, m), 2.83-2.90 (2H, m), 3.19 (2H, s), 3.92 (2H, s), 6.32 (2H, s), 6.53 (4H, d, $J = 7.0$ Hz), 6.86-6.89 (4H, m), 6.93-6.97 (8H, m), 7.02-7.04 (8H, m), 7.74 (4H, d, $J = 8.3$ Hz); ^{13}C NMR (100 MHz, CDCl_3) δ 21.5, 27.6, 31.3, 46.2, 54.6, 54.7, 126.7, 126.8, 127.2, 127.3, 128.0, 128.3, 128.9, 131.1, 135.7, 137.6, 143.0, 162.3, 163.3; IR (CHCl_3) 2999, 2958, 2928, 1600, 1574, 1557, 1093 cm^{-1} . Anal. Calcd for $\text{C}_{54}\text{H}_{48}\text{N}_4\text{O}_4\text{S}_2$: C, 73.1; H, 5.49; N, 6.36. Found: C, 73.28; H, 5.62; N, 6.47.

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13C

BCM

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5.50 usec

1H

22.1 C

CDCL3

0.00 ppm

0.12 Hz

23

DFILE COMNT
DATIM
OBNUC
EXMOD
OBFRO
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

9d

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

C:\WINNMR98\COMMON_DEFAULT
Thu Jan 26 19:41:04 2006
1H
NON
399.65 MHz
124.00 KHz
10500.00 Hz
32768
7992.01 Hz
16
4.1001 sec
2.9010 sec
5.80 usec
1H
20.0 c
CDCL3
0.00 ppm
0.12 Hz
18

DFILE COMNT
DATIM Thu Jan 26 15:00:21 2006
OBNUC 13C
EXMOD BCM
OBFRO 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27210.88 Hz
SCANS 1000
ACQTM 1.2042 sec
PD 1.7940 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 20.7 C
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 24

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRO
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

C:\WINNMR98\COMMON\DEFAULT
Tue Feb 07 20:13:51 2006
1H
NON
399.65 MHz
124.00 KHz
10500.00 Hz
32768
7992.01 Hz
8
4.1001 sec
2.9010 sec
5.80 usec
1H
CDCL3
0.00 ppm
0.12 Hz
13

C:\WINNMR98\COMMON_DEFAULT

DFILE
COMNT
DATIM Tue Feb 07 20:34:31 2006
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27210.88 Hz
SCANS 400
ACQTM 1.2042 sec
PD 1.7940 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 20.5 C
SLVNT CDCL3
EXREF 76.90 ppm
BF 0.12 Hz
RGAIN 23

Fri Oct 14 15:40:29 2005

1H

NON

399.65 MHz

124.00 KHz

10500.00 Hz

32768

7992.01 Hz

8

4.1001 sec

2.9010 sec

5.80 usec

1H

CDCL3

21.4 c

0.00 ppm

0.12 Hz

19

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFREQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

S27

Thu Feb 16 17:33:04 2006

DFILE COMNT
DATIM
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27210.88 Hz
SCANS 400
ACQTM 1.2042 sec
PD 1.7940 sec
FW1 5.50 usec
IRNUC 1H
CTEMP 21.1 C
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 22

S28