Supporting Information

Bismuth-catalyzed Intermolecular Hydroamination of 1,3-Dienes with Carbamates, Sulfonamides, and Carboxamides

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Experimental Section

General: Infrared (IR) spectra were recorded on a JASCO FT/IR 410 Fourier transform infrared spectrophotometer. NMR spectra were recorded on a JEOL JNM-LA500 spectrometer, operating at 500 MHz for ^1H NMR and 125.65 MHz for ^{13}C NMR. Chemical shifts in CDCl_3 were reported in the scale relative to CHCl_3 (7.24 ppm for ^1H NMR) and CDCl_3 (77.0 ppm for ^{13}C NMR) as an internal reference. ESI mass spectra (for LRMS) were measured on a Waters ZQ4000 spectrometer. FAB mass spectra (for HRMS) were measured on a JEOL JMS-700 spectrometer. Column chromatography was performed with silica gel Merck 60 (230-400 mesh ASTM). Reactions were carried out in anhydrous 1,4-dioxane under an argon atmosphere, unless otherwise stated. Anhydrous 1,4-dioxane was purchased from Kanto chemicals and used as received. $\text{Bi}(\text{OTf})_3$, $\text{Cu}(\text{CH}_3\text{CN})_4\text{PF}_6$, and KPF_6 were purchased from Aldrich Co., Ltd. and used as received. Commercially available carbamates, sulfonamides, and amides were purchased and used without purification.

General procedure:

$\text{Cu}(\text{CH}_3\text{CN})_4\text{PF}_6$ as additive

1-(4-toluenesulfonylamino)-2-cyclohexene (3af): To a 1,4-dioxane (1 mL) solution of Bi(OTf)₃ (19.6 mg, 0.03 mmol), Cu(CH₃CN)₄PF₆ (11.2 mg, 0.03 mmol), dppe (4.8 mg, 0.012 mmol) and *p*-toluenesulfonamide (**2f**) (51.4 mg, 0.30 mmol) in a test tube at 25 °C was added 1,3-cyclohexadiene (**1a**) (0.057 mL, 0.60 mmol, 2 equiv). The reaction mixture was stirred at 50°C. After 3 h, the mixture was diluted with diethyl ether (5 mL) and silica gel (ca.1 g) was added to the mixture. After filtration and washing with diethyl ether, the ether solution was evaporated under reduced pressure. The residue was purified by silica gel column chromatography (hexane/ethyl acetate = 5:1) to give **3af** (63 mg, 84%) as a colorless solid.

KPF₆ as additive

To a 1,4-dioxane (1 mL) solution of Bi(OTf)₃ (19.6 mg, 0.03 mmol), KPF₆ (5.5 mg, 0.03 mmol), and *p*-toluenesulfonamide (**2f**) (102.6 mg, 0.60 mmol) in a test tube at 25 °C was added 1,3-cyclohexadiene (**1a**) (0.069 mL, 0.72 mmol, 1.2 equiv). The reaction mixture was stirred at 25 °C. After 3 h, the mixture was diluted with diethyl ether (5 mL) and silica gel (ca.1 g) was added to the mixture. After filtration and washing with diethyl ether, the ether solution was evaporated under reduced pressure. The residue was purified by silica gel column chromatography (hexane/ethyl acetate = 5:1) to give **3af** (125 mg, 83%) as a colorless solid.

Compound **3aa**, **3ab**, **3ae**, **3af**, **3ai**, and **3aj** are known. For the known compound, the registry number is shown for each compound.

Benzyl cyclohex-2-enylcarbamate (3aa):

registry No. 91230-17-0 (known compound)

^1H NMR (CDCl_3) δ 1.52-1.57 (m, 1H), 1.62-1.65 (m, 2H), 1.89-1.92 (m, 1H), 1.98 (m, 2H), 4.23 (brs, 1H), 4.79 (brs, 1H), 5.08 (d, $J = 12.2$ Hz, 1H), 5.09 (d, $J = 12.2$ Hz, 1H), 5.58-5.70 (m, 1H), 5.80-5.85 (m, 1H), 7.28-7.41 (m, 5H); ^{13}C NMR (CDCl_3) δ 19.6, 24.7, 29.7, 46.3, 66.6, 127.7, 128.1, 128.1, 128.5, 130.8, 136.6, 155.6; LRMS (ESI, methanol) m/z 254 $[\text{M}+\text{Na}^+]$;

Methyl cyclohex-2-enylcarbamate (3ab):

registry No. 86766-64-5 (known compound)

^1H NMR (CDCl_3) δ 1.50-1.56 (m, 1H), 1.61-1.65 (m, 2H), 1.86-1.92 (m, 1H), 1.98-2.02 (m, 2H), 3.66 (s, 3H), 4.20 (brs, 1H), 4.66 (brs, 1H), 5.58-5.63 (m, 1H), 5.82-5.86 (m, 1H); ^{13}C NMR (CDCl_3) δ 19.6, 24.7, 29.7, 46.3, 51.9, 127.8, 130.7, 156.3.

Butyl cyclohex-2-enylcarbamate (3ac):

colorless oil; IR (neat) ν 726, 779, 1070, 1239, 1309, 1530, 1695, 2934, 3025, 3325 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.93 (t, $J = 7.3$ Hz, 3H), 1.34-1.41 (m, 2H), 1.50-1.69 (m, 5H), 1.85-2.04 (m, 3H), 4.05 (t, $J = 5.7$ Hz, 2H), 4.20 (brs, 1H), 4.65 (brs, 1H), 5.61 (brd, $J = 9.5$ Hz, 1H), 5.80-5.86 (m, 1H); ^{13}C NMR (CDCl_3) δ 13.7, 19.1, 19.6, 24.8, 29.8, 31.1, 46.1, 64.6, 127.9, 130.7, 156.0; LRMS (ESI, methanol) m/z 220 $[\text{M}+\text{Na}^+]$; HRMS (FAB) m/z 198.1496 $[\text{M}+\text{H}^+]$ calcd. for $\text{C}_{11}\text{H}_{20}\text{NO}_2 = 198.1489$.

3-(Cyclohex-2-enyl)oxazolidin-2-one (3ad):

colorless oil; ^1H NMR (CDCl_3) δ 1.57-2.03 (m, 6H), 3.47-3.53 (m, 2H), 4.30-4.33 (m, 2H), 4.47-4.50 (m, 1H), 5.49 (brd, $J = 10.0$ Hz, 1H), 5.93-5.96 (m, 1H); ^{13}C NMR (CDCl_3) δ 20.8, 24.4, 26.4, 41.0, 49.3, 62.0,

126.5, 132.2, 158.1; LRMS (ESI, methanol) m/z 190 $[M+Na^+]$; HRMS (FAB) m/z 168.1022 $[M+H^+]$ calcd. for $C_9H_{14}NO_2$ = 168.1019.

***N*-(Cyclohex-2-enyl)benzenesulfonamide (3ae):**

registry No. 108223-53-6 (known compound)

1H NMR ($CDCl_3$) δ 1.53-1.61 (m, 3H), 1.75-1.79 (m, 1H), 1.90-1.97 (m, 2H), 3.84-3.85 (m, 1H), 4.53 (brd, J = 8.2 Hz, 1H), 5.33-5.36 (m, 1H), 5.76-5.79 (m, 1H), 7.50-7.53 (m, 2H), 7.56-7.59 (m, 1H), 7.89-7.91 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 19.2, 24.4, 30.2, 49.0, 126.9, 126.9, 129.0, 131.5, 132.4, 141.3.

***N*-(Cyclohex-2-enyl)-4-methylbenzenesulfonamide (3af):**

registry No. 57981-18-7 (known compound)

1H NMR ($CDCl_3$) δ 1.52-1.62 (m, 3H), 1.74-1.79 (m, 1H), 1.86-2.01 (m, 2H), 2.43 (s, 3H), 3.79-3.84 (m, 1H), 4.54 (d, J = 7.3 Hz, 1H), 5.33-5.37 (m, 1H), 5.74-5.79 (m, 1H), 7.30 (d, J = 8.2 Hz, 2H), 7.77 (d, J = 8.2 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 19.2, 21.4, 24.4, 30.1, 48.9, 126.9, 127.0, 129.6, 131.3, 138.3, 143.1; LRMS (ESI, methanol) m/z 274 $[M+Na^+]$.

***N*-(Cyclohex-2-enyl)-4-methoxybenzenesulfonamide (3ag):**

colorless solid; IR (KBr) ν 582, 1077, 1156, 1262, 1327, 1499, 1596, 2931, 3280 cm^{-1} ; 1H NMR ($CDCl_3$) δ 1.50-1.57 (m, 2H), 1.60-1.64 (m, 1H), 1.72-1.78 (m, 1H), 1.90-1.98 (m, 2H), 3.75-3.82 (m, 1H), 3.87 (s, 3H), 4.87 (d, J = 8.5 Hz, 1H), 5.34-5.38 (m, 1H), 5.73-5.77 (m, 1H), 6.96-6.99 (m, 2H), 7.82-7.85 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 19.3, 24.4, 30.1, 48.9, 55.5, 114.1, 127.0, 129.0, 131.3, 132.9, 162.7; LRMS (ESI, methanol) m/z 290 $[M+Na^+]$; HRMS (FAB) m/z 268.1003 $[M+H^+]$ calcd. for $C_{13}H_{18}NO_3S$ = 268.1002.

***N*-(Cyclohex-2-enyl)-4-(trifluoromethyl)benzenesulfonamide (3ah):**

colorless solid; IR (KBr) ν 430, 613, 714, 1064, 1130, 1164, 1326, 3289 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.53-1.65 (m, 3H), 1.75-1.80 (m, 1H), 1.89-2.01 (m, 2H), 3.84-3.90 (m, 1H), 5.07 (d, $J = 8.6$ Hz, 1H), 5.34-5.38 (m, 1H), 5.77-5.81 (m, 1H), 7.78 (d, $J = 8.2$ Hz, 2H), 8.04 (d, $J = 8.2$ Hz, 2H); ^{13}C NMR (CDCl_3) δ 19.2, 24.4, 30.2, 49.3, 123.3 (q, $J_{\text{(C-F)}} = 272$ Hz), 126.2, 126.5, 127.4, 132.0, 134.2 (q, $J_{\text{(C-F)}} = 33.0$ Hz), 145.03; LRMS (ESI, methanol) m/z 328 $[\text{M}+\text{Na}^+]$; HRMS (FAB) m/z 306.0762 $[\text{M}+\text{H}^+]$ calcd. for $\text{C}_{13}\text{H}_{15}\text{F}_3\text{NO}_2\text{S} = 306.0770$.

***N*-(Cyclohex-2-en-1-yl)-2-nitrobenzenesulfonamide (3ai):**

registry No. 647011-33-4 (known compound)

^1H NMR (CDCl_3) δ 1.54-1.69 (m, 3H), 1.76-1.84 (m, 1H), 1.90-2.03 (m, 2H), 4.01-4.08 (m, 1H), 5.32 (d, $J = 8.2$ Hz, 1H), 5.39-5.44 (m, 1H), 5.79-5.85 (m, 1H), 7.73-7.76 (m, 2H), 7.87-7.89 (m, 1H), 8.16-8.20 (m, 1H); ^{13}C NMR (CDCl_3) δ 19.2, 24.4, 30.1, 50.1, 125.4, 126.3, 130.6, 132.2, 132.9, 133.4, 135.2, 148.2.

***N*-(Cyclohex-2-en-1-yl)benzamide (3aj)**

registry No. 4654-36-8 (known compound)

^1H NMR (CDCl_3) δ 1.61-1.72 (m, 3H), 1.96-2.07 (m, 3H), 4.66-4.73 (m, 1H), 5.65-5.71 (m, 1H), 5.87-5.93 (m, 1H), 6.22 (brs, 1H), 7.39-7.43 (m, 2H), 7.45-7.50 (m, 1H), 7.76-7.79 (m, 2H); ^{13}C NMR (CDCl_3) δ 19.7, 24.8, 29.4, 45.1, 126.9, 127.6, 128.4, 131.1, 131.2, 134.7, 166.7.

***N*-(Cyclohex-2-en-1-yl)-4-(trifluoromethyl)benzamide (3ak):**

colorless solid; IR (KBr) ν 420, 692, 856, 1017, 1065, 1122, 1166, 1327, 1542, 1634, 2937, 3301 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.64-1.72 (m, 3H), 2.01-2.17 (m, 3H), 4.70 (m, 1H), 5.66-5.71 (m, 1H), 5.92-5.97 (m, 1H), 6.12 (brs, 1H), 7.69 (d, $J = 8.0$ Hz, 2H), 7.87 (d, $J = 8.0$ Hz, 2H); ^{13}C NMR (CDCl_3) δ 19.7, 24.7, 29.3, 45.4, 123.6 (q, $J_{\text{(C-F)}} = 272$ Hz), 125.4 (q, $J_{\text{(C-F)}} = 4.1$ Hz), 127.1, 127.4, 131.5, 133.0 (q, $J_{\text{(C-F)}} = 32.8$ Hz), 138.0, 165.5; LRMS (ESI,

methanol) m/z 292 $[M+Na^+]$; HRMS (FAB) m/z 270.1099 $[M+H^+]$ calcd. for $C_{14}H_{15}F_3NO=$ 270.1100.

(E)-3-(Pent-3-en-2-yl)oxazolidin-2-one (3bd):

colorless oil; IR (neat) ν 764, 972, 1013, 1047, 1252, 1379, 1422, 1484, 1747, 2976 cm^{-1} ; 1H NMR ($CDCl_3$) δ 1.26 (d, $J = 7.5$ Hz, 3H), 1.70-1.72 (m, 3H), 3.47 (t, $J = 8.0$ Hz, 2H), 4.31 (t, $J = 8.0$ Hz, 2H), 4.47-4.50 (m, 1H), 5.42-5.48 (m, 1H), 5.62-5.71 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 17.0, 17.7, 40.1, 49.7, 61.9, 127.8, 129.5, 157.8; LRMS (ESI, methanol) m/z 178 $[M+Na^+]$; HRMS (FAB) m/z 156.1018 $[M+H^+]$ calcd. for $C_8H_{14}NO_2 = 156.1019$.

3-(3-Methylbut-2-enyl)oxazolidin-2-one (3cd):

colorless oil; IR (neat) ν 763, 1038, 1256, 1426, 1486, 1748, 2918 cm^{-1} ; 1H NMR ($CDCl_3$) δ 1.70 (s, 3H), 1.76 (s, 3H), 3.50 (m, 2H), 3.85 (d, $J = 7.3$ Hz, 2H), 4.30 (m, 2H), 5.15-5.22 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 17.8, 25.7, 41.7, 43.9, 61.7, 117.9, 137.9, 158.3; LRMS (ESI, methanol) m/z 178 $[M+Na^+]$; HRMS (FAB) m/z 156.1024 $[M+H^+]$ calcd. for $C_8H_{14}NO_2 = 156.1019$.

(E)-3-(4-Phenylbut-3-en-2-yl)oxazolidin-2-one (3dd):

colorless oil; IR (neat) ν 695, 751, 969, 1050, 1252, 1421, 1483, 1746, 2976 cm^{-1} ; 1H NMR ($CDCl_3$) δ 1.39 (d, $J = 7.0$ Hz, 3H), 3.51 (m, 2H), 4.32 (m, 2H), 4.70 (m, 1H), 6.18 (dd, $J = 5.8, 16.2$ Hz, 1H), 6.54 (d, $J = 16.2$ Hz, 1H), 7.23-7.26 (m, 1H), 7.30-7.33 (m, 2H), 7.36-7.38 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 17.0, 40.2, 49.9, 62.0, 126.4, 127.8, 127.9, 128.5, 131.7, 136.2, 157.8; LRMS (ESI, methanol) m/z 240 $[M+Na^+]$; HRMS (FAB) m/z 218.1177 calcd. for $C_{15}H_{20}NO_2 = 218.1176$.

3ed isomers were separated by silica gel column chromatography (eluent: hexane/ethyl acetate = 4/1).

(E)-3-(Dodec-3-en-2-yl)oxazolidin-2-one (3ed-major):

colorless oil; IR (neat) ν 1068, 1241, 1523, 1698, 2935, 3325 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.88 (t, $J = 7.0$ Hz, 3H), 1.24-1.37 (m, 15H), 2.03 (m, 2H), 3.46 (m, 2H), 4.30 (m, 2H), 4.49 (m, 1H), 5.41 (dd, $J = 5.8, 15.6$ Hz, 1H), 5.64 (dt, $J = 6.7, 15.6$ Hz, 1H); ^{13}C NMR (CDCl_3) δ 14.0, 17.1, 22.6, 29.1, 29.1, 29.2, 29.2, 31.8, 32.2, 40.1, 49.7, 61.9, 128.1, 133.3, 157.8; LRMS (ESI, methanol) m/z 276 $[\text{M}+\text{Na}^+]$; HRMS (FAB) m/z 254.2119 $[\text{M}+\text{H}^+]$ calcd. for $\text{C}_{15}\text{H}_{28}\text{NO}_2 = 254.2115$.

(E)-3-(Dodec-2-en-4-yl)oxazolidin-2-one (3ed-minor):

colorless oil; IR (neat) ν 701, 762, 1040, 1251, 1420, 1747, 2921 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.88 (t, $J = 7.0$ Hz, 3H), 1.26-1.31 (m, 12H), 1.53-1.57 (m, 2H), 1.70 (d, $J = 6.4$ Hz, 3H), 3.45 (m, 2H), 4.25 (brdd, $J = 7.3, 7.3$ Hz, 1H), 4.31 (m, 2H), 5.37-5.42 (dd, $J = 6.3, 15.5$ Hz, 1H), 5.65-5.70 (dq, $J = 6.4, 15.5$ Hz, 1H); ^{13}C NMR (CDCl_3) δ 14.1, 17.8, 22.6, 26.1, 29.2, 29.3, 29.4, 31.8, 31.8, 40.2, 54.7, 61.9, 128.3, 128.7, 158.1; LRMS (ESI, methanol) m/z 276 $[\text{M}+\text{Na}^+]$; HRMS (FAB) m/z 254.2111 $[\text{M}+\text{H}^+]$ calcd. for $\text{C}_{15}\text{H}_{28}\text{NO}_2 = 254.2115$

3fd isomers were separated by silica gel column chromatography (eluent: hexane/ethyl acetate = 4/1).

(E)-3-(6-Phenylhex-3-en-2-yl)oxazolidin-2-one (3fd-major):

colorless oil, IR (neat) ν 1050, 1252, 1422, 1746, 2924 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.22 (d, $J = 7.4$ Hz, 3H), 2.34-2.40 (m, 2H), 2.65-2.75 (m, 2H), 3.15-3.20 (m, 1H), 3.32-3.37 (m, 1H), 4.18-4.27 (m, 2H), 4.45 (m, 1H), 5.37 (dd, $J = 5.7, 15.5$ Hz, 1H), 5.61-5.67 (m, 1H), 7.13-7.19 (m, 3H), 7.24-7.29 (m, 2H); ^{13}C NMR (CDCl_3) δ 16.9, 33.9, 35.3, 39.9, 49.6, 61.9, 125.8, 128.2, 128.5, 129.2, 131.9, 141.4, 151.7; LRMS (ESI, methanol) m/z 268 $[\text{M}+\text{Na}^+]$; HRMS (FAB) m/z 246.1485 $[\text{M}+\text{H}^+]$ calcd. for $\text{C}_{15}\text{H}_{20}\text{NO}_2 = 246.1489$.

(E)-3-(6-Phenylhex-2-en-4-yl)oxazolidin-2-one (3fd-minor):

colorless oil; IR (neat) ν 761, 973, 1044, 1250, 1378, 1420, 1484, 1747, 2856, 2925 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.71 (d, $J = 6.4$ Hz, 3H),

1.87-1.95 (m, 2H), 2.55-2.72 (m, 2H), 3.33-3.49 (m, 2H), 4.17-4.24 (m, 1H) 4.25-4.35 (m, 2H), 5.40 (dd, $J = 5.2, 15.3$ Hz, 1H), 5.70 (dq, $J = 6.4, 15.3$ Hz, 1H), 7.16-7.21 (m, 3H), 7.26-7.31 (m, 2H); ^{13}C NMR (CDCl_3) δ 17.8, 33.6, 33.5, 40.4, 54.8, 61.9, 126.0, 128.0, 128.3, 128.4, 129.3, 141.3, 157.5; LRMS (ESI, methanol) m/z 268 [$\text{M}+\text{Na}^+$]; HRMS (FAB) m/z 246.1489 [$\text{M}+\text{H}^+$] calcd. for $\text{C}_{15}\text{H}_{20}\text{NO}_2 = 246.1489$

3gd isomers were inseparable by silica gel column chromatography.

(*E*)-3-(Hex-2-en-4-yl)oxazolidin-2-one (3gd-minor): 1,2 adduct

colorless oil; IR (neat) ν 1050, 1253, 1421, 1747, 2966 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.91 (t, $J = 7.6$ Hz, 3H), 1.54-1.69 (m, 2H), 1.70-1.72 (m, 3H), 3.43-3.49 (m, 2H), 4.17 (brdd, $J = 7.6, 7.6$ Hz, 1H), 4.30-4.33 (m, 2H), 5.39-5.44 (m, 1H), 5.65-5.72 (m, 1H);

(*E*)-3-(Hex-3-en-2-yl)oxazolidin-2-one (3gd-major):1,4 adduct δ

0.99 (t, $J = 7.4$ Hz, 3H), 1.26 (d, $J = 7.0$ Hz, 3H), 2.02-2.11 (m, 2H), 3.43-3.49 (m, 2H), 4.30-4.33 (m, 2H), 4.49 (m, 1H), 5.39-5.44 (m, 1H), 5.65-5.72 (m, 1H); ^{13}C NMR (CDCl_3 : mixture of 1,2- and 1,4-isomers) δ 10.6, 13.4,

17.1, 17.8, 24.8, 25.2, 40.1, 40.2, 49.7, 56.3, 61.9, 127.2, 128.1, 128.9, 134.7, 157.7, 158.2; LRMS (ESI, methanol) m/z 192 [$\text{M}+\text{Na}^+$]; HRMS (FAB) m/z 170.1174 [$\text{M}+\text{H}^+$] calcd. for $\text{C}_9\text{H}_{16}\text{NO}_2 = 170.1176$.

[Mechanistic Studies]

To gain insight into the role of $\text{Bi}(\text{OTf})_3$, $\text{Cu}(\text{CH}_3\text{CN})_4\text{PF}_6$ **4**, and KPF_6 **6**, the reaction profiles (conc. of diene **1a**) under conditions A-F (see, below) were monitored by GC. Under conditions D, E and F, desired 1:1 adduct **3af** was isolated at 3 h to evaluate the ratio of the desired 1:1 reaction and undesired polymerizations.

Experimental Procedure for GC Analysis:

GC: SHIMADZU Co. Ltd. GC-14A

Conditions: Column: Restek Co. Inc. RT-BDEXm 30 m; Detector: FID; Flowing pressure: N_2 4 kPa; Column temperature: 60°C; Injector temperature: 100 °C; Detector temperature: 200 °C

Procedure:

To a 1,4-dioxane (1 mL) solution of $\text{Bi}(\text{OTf})_3$ (x mmol), $\text{Cu}(\text{CH}_3\text{CN})_4\text{PF}_6$ (**4**) (y mmol) {for conditions F: KPF_6 (w mmol)}, *p*-toluenesulfonamide (**2f**) (z mmol), and hexane (0.07 mL, internal standard) in a test tube was added 1,3-cyclohexadiene (**1a**) (0.069 mL, 0.72 mmol) via syringe. The reaction mixture was stirred at 25°C, and samples were taken via syringe (ca. 0.1 mL) after 0.5, 1, 2, 3, 4, and 6 h. The sampled solution was treated with saturated NaHCO_3 solution and the organic material was extracted with diethyl ether. The ether solution was injected into GC to analyze the relative amount of remaining **1a**.

GC chart:

Experimental conditions A-F:

without amide 2f:

conditions A: $x = 0.03$, $y = 0$, $z = 0$, $w = 0$ (**1a** and Bi(OTf)₃)

conditions B: $x = 0$, $y = 0.03$, $z = 0$, $w = 0$ (**1a** and Cu(CH₃CN)₄PF₆ **4**)

conditions C: $x = 0.03$, $y = 0.03$, $z = 0$, $w = 0$ (**1a**, Bi(OTf)₃, and Cu(CH₃CN)₄PF₆ **4**)

with amide 2f:

conditions D: $x = 0.03$, $y = 0$, $z = 0.60$, $w = 0$ (**1a**, **2f** and Bi(OTf)₃)

conditions E: $x = 0.03$, $y = 0.03$, $z = 0.60$, $w = 0$ (**1a**, **2f**, Bi(OTf)₃, and Cu(CH₃CN)₄PF₆ **4**)

conditions F: $x = 0.03$, $y = 0$, $z = 0.60$, $w = 0.03$ (**1a**, **2f**, Bi(OTf)₃, and KPF₆ **6**)

Results:

Conditions A-C (without amide)

Polymerization of **1a** was observed under conditions A, while no polymerization occurred under conditions B. These results suggest that Bi activates diene **1a** to generate allyl cationic species. On the other hand, the polymerization rate increased under conditions C, indicating that more active bismuth species are generated in the presence of Cu(CH₃CN)₄PF₆ **4**.

Conditions D-F (with amide)

Under conditions D, the profile of [**1a**] was similar to that observed under conditions B.

Under conditions D, 0.20 mmol of **1a** was consumed after 3 h and only 0.108 mmol of the desired 1:1 adduct **3af** was isolated at that period. Thus, the desired 1:1 addition and undesired polymerization competed under conditions D. The rate of the polymerization was almost same as that of the desired reaction. Under conditions E and F, the best reaction rate was observed among conditions A-F. Similar reaction rate in conditions E and F would suggest the generation of similar active species. Under conditions E, 0.50 mmol of **1a** was consumed after 3 h. 0.438 mmol (conditions E) and 0.498 mmol (conditions F) of the desired 1:1 adduct **3af** was isolated after 3 h, suggesting that the desired reaction was the major pathway in conditions E and F. Thus, PF_6^- clearly accelerated the desired 1:1 addition over undesired polymerization.

Ability of Lewis acidic bismuth to interact with carbonyl group of amide **2j** was suggested by the following React IR and ^{13}C -NMR analyses.

Experimental Procedure for React IR

Apparatus: React IRTM 4000 from METTLER Co. Ltd.

Solvent: CH_2Cl_2

Temperature; 25 °C

Experiment RI-A:

Benzamide (**2j**) (45.3 mg, 0.30 mmol), CH_2Cl_2 (1 mL)

[background spectrum: CH_2Cl_2 (1 mL)]

Experiment RI-B:

$\text{Bi}(\text{OTf})_3$ (197 mg, 0.30 mmol), Benzamide (**2j**) (45.3 mg, 0.30 mmol), CH_2Cl_2 (1 mL)

[background spectrum: $\text{Bi}(\text{OTf})_3$ (197 mg, 0.30 mmol), CH_2Cl_2 (1 mL)]

Experiment RI-A

Experiment RI-B

The peak corresponding to carbonyl of **2j** (1733 cm^{-1}) shifted to 1653 cm^{-1} in the presence of $\text{Bi}(\text{OTf})_3$ (without adding diene), supporting the interaction of Bi with carbonyl group of amide **2j**.

Experimental conditions for NMR study

Apparatus: JEOL ECX-500

Solvent: 1,4-dioxane (with D₂O sealed rod)

temp: 27 °C

Experiment NMR-A:

Benzamide (36.3 mg, 0.30 mmol) in 1,4-dioxane (1 mL)

Experiment NMR-B:

Benzamide (36.3 mg, 0.30 mmol) and Bi(OTf)₃ (197 mg, 0.30 mmol) in 1,4-dioxane (1 mL)

Results:

Experiment NMR-A

Experiment NMR-B

Low field shift of the carbonyl peak in the presence of $\text{Bi}(\text{OTf})_3$ (A: 167.9 ppm vs B: 173.5 ppm) also supported the interaction of Bi with carbonyl group of amide **2j**.

The results in IR and NMR experiments implied that bismuth can act as a Lewis acid for carbonyl groups, while GC analyses of reaction profiles suggested the ability of bismuth to activate dienes. On the basis of these results, postulated catalytic cycle is shown below. We assume that $\text{Bi}(\text{OTf})_3$ activates diene to generate an allyl cationic species, which is trapped with amides (desired) or with diene (polymerization). Counter anion exchange with PF_6^- would then generate intermediate (I). With PF_6^- as the counter anion, the coordination site of Bi would be available. Therefore, the amide can interact with the Bi center and be positioned close to the reaction site (II), accelerating the desired 1:1 addition. Protonation of (III) regenerates catalyst (IV).

Postulated catalytic cycle:

Date : Fri Nov 5 02:01:18 2004

FileName : LoadingFID.nmdata
 Comment : MeO2C-NH-C6-diene
 SliceHistory :
 EXMODE : bcm

POINT : 32768 points
 SAMP0 : 32768 points
 FREQ0 : 33898.3 Hz
 FILTR : 16950 Hz
 DELAY : 11.8 usec
 DEADT : 10.0 usec
 INIYL : 29.5 usec
 TIMES : 1600 times
 DUMMY : 1 times
 PD : 2.0333 sec
 ACQTM : 966.6560 msec
 PREDL : 0.01000 msec
 INIWT : 1000.0000 msec
 RESOL : 1.03 Hz
 PW1 : 6.25 usec
 13C : 125.65 MHz
 OBNUC : 127958.00 Hz
 OBFRQ : 18
 1H : 500.00 MHz
 OBFRQ : 162160.00 Hz
 IRSET : 50.0 usec
 IRPW : 0
 IRFNS : 0

SCANS : 224 times
 SLVNT : CDCl3
 SPINNING : 11 Hz
 TEMP : 26.6 C

3ab

DFILE C:\My Documents\pgousei\G
 COMNT ny200511082
 DATIM 08-11-2005 17:54:09
 OBNUC 1H
 EXMOD single_pulse.ex2
 OBFRO 495.13 MHz
 OBSET 4.38 KHz
 OBFIN 9.6 Hz
 POINT 16384
 FREQU 9286.8 Hz
 SCANS 8
 ACQTM 1.764 sec
 PD 5.000 sec
 PW1 6.2 us
 IRNUC 1H
 CTEMP 25.0 c
 SLVNT CDCL3
 EXREF 7.24 ppm
 BF 0.00 Hz
 RGAIN 46

DFILE C:\My Documents\qin\1704\bucarc6\13
 COMNT 1h Line
 DATIM Mon Apr 11 14:48:26 2005
 OBNUC 13C
 EXMOD bcm
 OBFRQ 125.65 MHz
 OBSEF 120.00 KHz
 OBFIN 7958.0 Hz
 POINT 32768
 FREQU 33898.3 Hz
 SCANS 250
 ACQTM 0.967 sec
 PD 2.033 sec
 PW1 6.2 us
 IRNUC 1H
 CTEMP 24.4 C
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 31

DFILE C:\My Documents\PGousei
 COMNT hy200511091
 DATIM 09-11-2005 01:07:29
 OBNUC 1H
 EXMOD single pulse.ex2
 OBFRQ 495.13 MHz
 OBSET 4.38 KHz
 OBFIN 9.6 Hz
 POINT 16384
 FREQU 9286.8 Hz
 SCANS 8
 ACQTM 1.764 sec
 PD 5.000 sec
 PW1 6.2 us
 IRNUC 1H
 CTEMP 20.7 c
 SLVNT CDCL3
 EXREF 7.24 ppm
 BF 0.00 Hz
 RGAIN 40

DF11E C:\My Documents\qin\1704\oxazolidio
 COMNT 1H Line
 DATIM Fri Apr 8 21:50:49 2005
 OBNUC 13C
 EXMOD bcm
 OBFRO 125.65 MHz
 OBSET 120.00 KHz
 OBFIN 7958.0 Hz
 POINT 32768
 FREOU 33898.3 Hz
 SCANS 131
 ACQTM 0.967 sec
 PD 2.033 sec
 PW1 6.2 us
 IRRUC 23.0 c
 CTEMP
 SIVMT
 EXREF
 BF 77.00 ppm
 0.12 Hz
 RGAIN 30

3ad

DEPT
CPDPR
NY200511051
INSTR
05-11-2005 22:57:17
NAME
1H
PULSE
495.13 Hz
OBSRV
4.38 Hz
OBSRV
9.6 Hz
POINT
16384
FREQ
9286.8 Hz
SCANS
8
ACQIN
1.764 sec
PD
5.000 sec
PWL
6.2 us
IRREL
1H
CHD13
21.0 c
SILVER
7.24 PPM
EXPER
0.12 Hz
RF
44
NAME

3ad

DFILE D:\qinmr-2\lambdabda from 0504\170705
COMNT 1H Line
DATIM Mon Jul 4 22:48:27 2005
OENUC 13C
EXMOD bcm
OFRQ 125.65 MHz
OBSET 120.00 KHz
OBFIN 7956.0 Hz
POINT 32768
FREOU 33898.3 Hz
SCANS 150
ACQTM 0.967 sec
PD 2.033 sec
PWL 6.2 us
IRNUC 1H
CTEMP 28.5 C
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 30

DATE: Mon Jul 4 22:37:48 2005
 FILE: C:\My Documents\qin\qhs02phadd\qhs02phadd.mdata
 1H NMR
 EXPT: 500.00 MHz
 OBSFQ: 160.00 KHz
 OBSF1: 2160.0 Hz
 POINT: 32768
 FREQ0: 10000.0 Hz
 SCANS: 8
 ACQTM: 3.277 sec
 PD: 3.723 sec
 PUL: 6.0 us
 TRATE: 511
 C12M: 27.5 c
 C12C13: 0.00 ppm
 EXREF: 0.12 Hz
 RGAIN: 20

3ae

DFILE C:\My Documents\qin\data0712\data13
 COMNT 1H Line
 DATIM Mon Jul 11 18:06:55 2005
 ENMOD 13C
 EXMOD bcm
 OFFRQ 125.65 MHz
 OBSET 120.00 KHz
 OFFTN 7958.0 Hz
 POINT 32768
 FREQU 33898.3 Hz
 SCANS 221
 ACQTM 0.967 sec
 PD 2.033 sec
 PW1 6.2 us
 IRNUC 1H
 CTEMP 26.8 C
 SIVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 30

C:\My Documents\qin\data0712\data1HTSNHC6d1eneadd\data1HTSNHC6d1eneadd.mdata
1H Line

DFILE C:\My Documents\qin\data0712\data1H
COMNT 1H Line
DATIM Mon Jul 11 23:09:49 2005
OBNUC 1H
EXMOD non
OBRQ 500.00 MHz
OBSET 160.00 KHz
OBFIN 2160.0 Hz
POINT 32768
FREQU 10000.0 Hz
SCANS 13
ACQTM 3.277 sec
PD 3.723 sec
PWL 6.0 us
IRNUC 1H
CTEMP 24.8 C
SIVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 19

DFILE C:\My Documents\qin\13Cdata\MeOPhSo2
 COMNT 1H Line
 DATIM Fri Nov 5 02:01:18 2004
 EXMOD 13C
 EXMOD bcm
 OBFRO 125.65 MHz
 OBFRO 120.00 KHz
 OBFRO 7958.0 Hz
 POINT 32768
 FREQU 33898.3 Hz
 SCANS 93
 ACQTM 0.967 sec
 PD 2.033 sec
 PW1 6.2 us
 IRNUC 1H
 CTMP 26.9 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 31

DFILE C:\My Documents\qin\1hdata\MeOPhSO2N
 COMMENT 1H Line
 DATE Mon Jul 18 00:57:33 2005
 EXMOD 1H
 EXREF 14
 FREQ 500.00 MHz
 OBSET 160.00 KHz
 OBFIN 2160.0 Hz
 POINT 32768
 FREQ 10000.0 Hz
 SCANS 13
 ACQTM 3.277 sec
 PD 3.723 sec
 PW 6.0 us
 IRNUC 26.4 C
 CTMP CDCL3
 SLVNT 0.00 ppm
 EXREF 0.12 Hz
 BF 14
 RGAIN

30g

FILE C:\My Documents\qin\13CdataC6dienec
 COMNT 1H Line
 DATIM Mon Jul 18 00:46:27 2005
 OENUC 13C
 EXMOD bcm
 OBRQ 125.65 MHz
 OBSE 120.00 KHz
 OBRIN 7958.0 Hz
 POINT 32768
 FREQ 33898.3 Hz
 SCANS 266
 ACQTM 0.967 sec
 PD 2.033 sec
 PUL 6.2 us
 IRNUC 1H
 CTEMP 26.8 C
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 31

FILE C:\My Documents\qin\data1HC6dieneCF
COMNT 1H Line
DATIM Mon Jul 18 00:32:25 2005
OBNUC 1H
EXMOD non
OBFRO 500.00 MHz
OBSET 160.00 KHz
OBFIN 2160.0 Hz
POINT 32768
FREQU 10000.0 Hz
SCANS 9
ACQTM 3.277 sec
PD 3.723 sec
PWL 6.0 us
IRNUC 1H
CTEMP 25.8 C
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 14

Nscyclohexdieneadd

Date : Fri Nov 5 02:01:18 2004

FileName : LoadingFID.rmd
Comment : Nscyclohexdieneadd
Slicehistory :
EXMODE : bcm

POINT 32768 points
SAMPD 32768 points
FREQU 33898.3 Hz
FILTR 16950 Hz
DELAY 11.8 usec
DEADT 10.0 usec
INTVL 29.5 usec
TIMES 1600 times
DUMMY 1 times
PD 2.0333 sec
ACQTM 956.6560 msec
PREDL 0.01000 msec
IN1WT 1000.0000 msec
RESOL 1.03 Hz
PWI 6.25 usec
13C 125.55 MHz
OBFRQ 127958.00 Hz
ORSET 16
RGAIN 16
IRNUC 500.00 MHz
IRFAG 162160.00 Hz
IRSET 50.0 usec
IRRPW 0
IRNS 0
SCANS 123 times
SLVNT CDCL3
SPINNING 8 Hz
TEMP 25.9 C

S-34

Date : Mon Jul 18 00:24:40 2005

Filename : LoadingID.nmdata
 Comment : dataC6d1eneNHCOPh
 SliceHistory :
 EXMODE : bcm

POINT	32768 points
SAMPD	32768 points
FREQU	33898.3 Hz
FILTR	16950 Hz
DELAY	11.8 usec
DEADT	10.0 usec
INVL	29.5 usec
TIMES	1500 times
DUMMY	1 times
PD	2.0333 sec
ACQTM	966.6560 msec
PREDL	0.01000 msec
ININT	1000.0000 msec
RESOL	1.03 Hz
PW1	6.25 usec
OBNUC	¹³ C
OBFRQ	125.65 MHz
OBSET	127958.00 Hz
RGAIN	31
IRNUC	¹ H
IRFRQ	500.00 MHz
IRSET	162160.00 Hz
IRPW	50.0 usec
IRNS	0
SCANS	201 times
SLVNT	CDCl ₃
SPINNING	13 Hz
TEMP	26.4 C

3aJ

1H Line

FILE C:\My Documents\qin\1HdataC6dienenH
 COMNT 1H Line
 DATIM Mon Jul 18 00:27:15 2005
 OBNUC 1H
 EXMOD non
 OBFO 500.00 MHz
 OBSET 160.00 KHz
 OBFIN 2160.0 Hz
 POINT 32768
 FREOU 10000.0 Hz
 SCANS 11
 ACQTM 3.277 sec
 PD 3.723 sec
 PW1 6.0 us
 IRNUC 1H
 CTEMP 26.0 C
 SIVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 15

3a

5-37

CF3benzamide

Date : Sat Jul 30 15:01:56 2005

FileName : LoadingFID.nmdata
 Comment : CF3benzamide
 SliceHistory :
 EXMODE : bcm

POINT	32768 points
SAMPD	32768 points
FREQU	33898.3 Hz
FILTR	16950 Hz
DELAY	11.8 usec
DEADT	15.1 usec
INTVL	29.5 usec
TIMES	1600 times
DUMMY	1 times
PD	2.0333 sec
ACQTM	966.6560 msec
PREDL	0.01000 msec
INVT	1000.0000 msec
RESOL	1.03 Hz
PM1	5.25 usec
OBNUC	13C
OBFRQ	125.40 MHz
OBSET	143041.00 Hz
RGAIN	28
IRNUC	1H
IRFRQ	499.10 MHz
IRSET	128250.00 Hz
IRRPW	65.0 usec
IRRNS	0
SCANS	430 times
SLVNT	CDCL3
SPINNING	11 Hz
TEMP	25.7 C

D:\qinmr-2\HA-data\addition-Nov-05\13Cpiperyleoxadone-200\13Cpiperyleoxadone-200.als
 1H Line
 125.65 MHz
 120.00 KHz
 7958.0 Hz
 32768
 33898.3 Hz
 209
 0.967 sec
 2.033 sec
 6.2 us
 28.1 c
 77.00 ppm
 0.12 Hz
 20

3bd

DFILE C:\My Documents\PGousei\qin
COMNT single_pulse
DATIM 04-06-2005 22:03:10
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 495.13 MHz
OBSET 4.38 KHz
OBFIN 9.6 Hz
POINT 13107
FREQU 7429.3 Hz
SCANS 8
AQTM 1.764 sec
PD 5.000 sec
PW1 5.6 us
IRNUC 1H
CTEMP 20.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 50

36d

S-41

DATIM Fri Nov 11 20:15:21 2005
 DEFILE C:\My Documents\qin\13C1sopreneoxadone-2nd\13C
 13C
 bcm
 125.65 MHz
 120.00 KHz
 7958.0 Hz
 32768
 33898.3 Hz
 305
 0.567 sec
 2.033 sec
 6.2 us
 511
 27.9 °
 77.00 ppm
 0.12 Hz
 30

309

DFILE C:\My Documents\PGousei\gin
 COMNT single pulse decoupled gate
 DATIM 27-07-2005 17:17:27
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFRQ 124.51 MHz
 OBSET 3.45 KHz
 OBFIN 6.0 Hz
 POINT 26213
 FREQU 31249.5 Hz
 SCANS 170
 ACQTM 0.839 sec
 PD 2.000 sec
 PW1 3.6 us
 IRNUC 1H
 CTEMP 22.4 C
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 56

3dd

5-44

C:\My Documents\qin\1Hdata\chchchchchchone\1Hdata\chchchchchchone.rmdata
1H Line

309

S-45

Date : Mon Aug 1 19:54:58 2005

FileName : LoadingFileID.nmdata
 Comment : C914acc
 SliceHistory :
 EXMODE : bcm

POINT : 32768 points
 SAMPD : 32768 points
 FREQU : 33898.3 Hz
 FILTR : 16950 Hz
 DELAY : 11.8 usec
 DEADT : 10.0 usec
 INIYL : 29.5 usec
 TIMES : 1600 times
 DUMMY : 1 times
 PD : 2.0333 sec
 ACQTM : 966.6560 msec
 PREDL : 0.01000 msec
 INIWT : 1000.0000 msec
 RESOL : 1.03 Hz
 PW1 : 6.25 usec
 OBNUC : ¹³C
 OBFRQ : 125.65 MHz
 OBSET : 127958.00 Hz
 RGAIN : 31
 IRNUC : ¹H
 IFRQ : 500.00 MHz
 IRSET : 162160.00 Hz
 TRAPW : 50.0 usec
 IPRNS : 0

SCANS : 317 times
 SLVNT : CDCl₃
 SPINNING : 11 Hz
 TEMP : 28.0 C

FILE C:\My Documents\qin\1hdatac9dieneox
 COMNT 1H line
 DATIM Mon Aug 1 19:37:28 2005
 OBNUC 1H
 EXMOD non
 OBRQ 500.00 MHz
 OBSE 160.00 KHz
 OBRIN 2160.0 Hz
 POINT 32768
 FREQU 10000.0 Hz
 SCANS 16
 ACQTM 3.277 sec
 PD 3.723 sec
 PWT 6.0 us
 IRNUC 1H
 CTEMP 26.8 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 16

S-42

DFILE C:\My Documents\qin\1HC9diene
 COMNT 1H Line
 DATIM Sat Nov 12 18:31:04 2005
 OBNUC 1H
 EXMOD non
 OBFRQ 500.00 MHz
 OBSET 160.00 KHz
 OBFIN 2160.0 Hz
 POINT 32768
 FREOU 10000.0 Hz
 SCANS 16
 ACQTM 3.277 sec
 PD 3.723 sec
 PW1 6.1 us
 IRNUC 1H
 CTEMP 27.7 C
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 19

3ed power

Date : Fri Nov 5 02:01:18 2004

Filename : LoadingFID.nmdata
 Comment : PhEtOxal14adduct
 SliceHistory :
 EXMODE : bcm

POINT 32768 points
 SAMPD 32768 points
 FREQ 33898.3 Hz
 FILTR 16950 Hz
 DELAY 11.8 usec
 DEADT 10.0 usec
 INTVL 29.5 usec
 TIMES 1600 times
 DUMMY 1 times
 PD 2.0333 sec
 ACQTM 966.6560 msec
 PREDL 0.01000 msec
 INIWT 1000.0000 msec
 RESOL 1.03 Hz
 PW1 6.25 usec
 13C 125.65 MHz
 OBSFQ 127958.00 Hz
 RGAIN 19
 1H 500.00 MHz
 IFRQ 162160.00 Hz
 IRSET 50.0 usec
 IRRPW 0
 IRRNS 0

SCANS 286 times

SLVNT CDCl3
 SPINNING 0 Hz
 TEMP 27.2 C

DFILE C:\My Documents\PGousei
 COMNT 1H Line
 DATIM
 OBNUC 13C
 EXMOD bcm
 OBFRO 125.40 MHZ
 OBSET 140.00 KHz
 OBFIN 3041.0 Hz
 POINT 32768
 FREQU 33898.3 Hz
 SCANS 301
 ACQTM 0.967 sec
 PD 2.033 sec
 PW1 5.8 us
 IRNUC 1H
 CTEMP 25.4 C
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 28

3fa isomer

FILE C:\My Documents\qin\1HdataPh
COMNT 1H Line
DATIM Sat Nov 12 23:11:03 2005
OBNDC 1H
EXMOD non
OBFRO 500.00 MHz
OBFET 160.00 KHz
OBFIN 2160.0 Hz
POINT 32768
FREOU 10000.0 Hz
SCANS 24
ACQTM 3.277 sec
PD 3.723 sec
PM1 6.0 us
IRNUC 1H
CTEMP 26.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 25

3rd isomer

Date : Fri Nov 5 02:01:16 2004

Filename : LoadingFID.mdata
 Comment : 24hexdienemix13C
 SliceHistory :
 EXMODE : bcm

POINT : 32768 points
 SAMPD : 32768 points
 FREQ : 33898.3 Hz
 FILTR : 15950 Hz
 DELAY : 11.8 usec
 DEADT : 10.0 usec
 INTVL : 29.5 usec
 TIMES : 1500 times
 DUMMY : 1 times
 PD : 2.0333 sec
 ACQTM : 966.5560 msec
 PREDL : 0.01000 msec
 INIWT : 1000.0000 msec
 RESOL : 1.03 Hz
 PW1 : 6.25 usec
 OBNUC : 13C
 OBFREQ : 125.65 MHz
 OBSET : 127958.00 Hz
 RGAIN : 16
 IRNUC : 1H
 IREFREQ : 500.00 MHz
 IRSET : 162160.00 Hz
 IRRPW : 50.0 usec
 IRRNS : 0

SCANS : 227 times

SLVNT : CDCL3
 SPINNING : 13 Hz
 TEMP : 27.8 C

D:\qinmr-2\lambdambda from 0504\170705\1Hdatahexadienedone12add-mix\1Hdatahexadienedone12add.rmda ta
 1H line