Supporting Information

Enantioselective Construction of Quaternary Carbon Centers by Catalytic [2+2+2] Cycloaddition of 1,6-Enynes and Alkynes

Takanori Shibata,* Yoshikazu Arai, and Yu-ki Tahara

Department of Chemistry, School of Science and Engineering, Waseda University, Shinjuku, Tokyo, 169-8555, Japan

General. Optical rotation was measured using Jasco DIP-370 polarimeter. IR spectra were recorded with Horiba FT210 spectrophotometer. NMR spectra were measured with JASCO DIP-1000 or Varian VXR-300S spectrometer using tetramethylsilane as an internal standard and CDCl₃ was used as solvent. Mass spectra were measured with JEOL JMS-SX102A and elemental analyses with Perkin Elmer PE2400II.

Synthesis of 1,6-Enynes 1a-g

Enynes **1a**,¹ **1b**¹ and **1d**² were prepared by the alkylation of *N*-(butyn-2-yl)-, *N*-(3-phenylpropyn-2-yl)-, *N*-(propyn-2-yl)tosylamine, respectively with 1-bromo-2-methylprop-2-en using NaH as a base. Enynes **1c**¹ and **1e**³ were prepared from *N*-(butyn-2-yl)-, *N*-(propyn-2-yl)tosylamine, respectively and 2-phenylprop-2-en-1-ol under Mitsunobu reaction conditions. Enyne **1f**⁴ was prepared by the alkylation of Dimethyl propargylmalonate with 1-bromo-2-methylprop-2-en using NaH as a base. Ether **1g**⁵ was prepared by etherification of propargyl alcohol with 1-bromo-2-methylprop-2-en in aq. KOH.

3,4-Bis(methoxymethyl)-2,6-dimethyl-8-tosyl-8-azabicyclo[4.3.0]nona-1,3-diene (2a). Colorless oil. IR (neat) 2925, 2821, 2360, 2339, 1344, 1159, 1093 cm⁻¹, ¹H NMR δ= 1.00 (s, 3H), 1.68 (s, 3H), 2.06 (d, *J*= 16.4 Hz, 1H), 2.27 (d, *J*= 16.4 Hz, 1H), 2.43 (s, 3H), 2.76 (d, *J*= 8.8 Hz, 1H), 3.28 (s, 3H), 3.29 (s, 3H), 3.53 (d, *J*= 8.8 Hz, 1H), 3.78 (d, *J*= 14.8 Hz, 1H), 3.91-4.15 (m, 5H), 7.33 (d, *J*= 8.0 Hz, 2H), 7.72 (d, *J*= 8.0 Hz, 2H); ¹³C NMR δ= 15.0, 21.5, 21.6, 37.4, 40.8, 49.7, 57.8, 57.9, 62.4, 66.9, 71.4, 123.4, 127.6, 129.5, 130.8, 132.1, 132.9, 136.7, 143.4; HRMS (FAB⁺) for M-1 found *m/z* 390.1746, calcd for C₂₁H₂₈NO₄S: 390.1738. [α]_D²⁸= 32.0 (*c* 0.68, CHCl₃, 97% ee). Ee was determined by HPLC analysis using a chiral column (Daicel Chiralpak AD-H: 4 x 250mm, 254nm UV detector, rt, eluent: 10% 2-propanol in hexane, flow rate: 1.0 mL/min, retention time: 12 min for minor isomer and 14 min for major isomer).

3,4-Bis(methoxymethyl)-6-methyl-2-phenyl-8-tosyl-8-azabicyclo[4.3.0]nona-1,3-diene (2b). Colorless oil. IR (neat) 2925, 2819, 2359, 2333, 1342, 1159, 1092 cm⁻¹, ¹H NMR δ= 1.12 (s, 3H), 2.20 (d, *J*= 16.4 Hz, 1H), 2.38 (d, *J*= 16.4 Hz, 1H), 2.41 (s, 3H), 2.86 (d, *J*= 9.0 Hz, 1H), 2.97 (s, 3H), 3.32 (s, 3H), 3.47 (d,

$J = 15.6$ Hz, 1H), 3.56 (d, $J = 9.0$ Hz, 1H), 3.69 (s, 2H), 4.02 (d, $J = 12.2$ Hz, 1H), 4.13 (d, $J = 12.2$ Hz, 1H), 4.19 (d, $J = 15.6$ Hz, 1H), 7.05 (br, 2H), 7.27-7.32 (m, 5H), 7.63 (d, $J = 8.4$ Hz, 2H); ^{13}C NMR $\delta = 21.5, 21.6, 36.8, 41.5, 49.9, 57.7, 58.0, 60.4, 61.9, 67.4, 71.4, 127.1, 127.4, 128.0, 129.5, 130.3, 131.2, 133.3, 133.4, 137.7, 138.8, 143.3$; HRMS (FAB⁺) for M-1 found m/z 452.1904, calcd for $\text{C}_{26}\text{H}_{30}\text{NO}_4\text{S}$: 452.1895. $[\alpha]_{\text{D}}^{26} = 15.4$ (c 1.35, CHCl_3 , 92% ee). Ee was determined by HPLC analysis using a chiral column (Daicel Chiralpak AD-H: 4 x 250mm, 254nm UV detector, rt, eluent: 10% 2-propanol in hexane, flow rate: 1.0 mL/min, retention time: 8 min for major isomer and 10 min for minor isomer).

3,4-Bis(methoxymethyl)-2-methyl-6-phenyl-8-tosyl-8-azabicyclo[4.3.0]nona-1,3-diene (2c). White solid. mp 149°C, IR (neat) 2627, 2814, 2359, 2333, 1348, 1165, 1093, cm^{-1} , ^1H NMR $\delta = 1.88$ (s, 3H), 2.36 (d, $J = 16.0$ Hz, 1H), 2.38 (s, 3H), 2.73 (s, 3H), 2.80 (d, $J = 16.0$ Hz, 1H), 3.14 (s, 3H), 3.25 (d, $J = 9.6$ Hz, 1H), 3.78 (d, $J = 12.4$ Hz, 1H), 3.83 (d, $J = 12.4$ Hz, 1H), 3.90 (d, $J = 11.6$ Hz, 1H), 4.01-4.07 (m, 3H), 4.12 (d, $J = 14.8$ Hz, 1H), 7.14-7.16 (m, 7H), 7.50 (d, $J = 8.0$ Hz, 2H); ^{13}C NMR $\delta = 15.1, 21.6, 38.7, 49.1, 50.1, 56.8, 57.5, 64.0, 66.5, 70.4, 126.4, 126.6, 127.3, 127.5, 128.0, 129.3, 132.1, 132.7, 133.2, 134.0, 142.1, 143.1$; HRMS (FAB⁺) for M-1 found m/z 452.1891, calcd for $\text{C}_{26}\text{H}_{30}\text{NO}_4\text{S}$: 452.1895. $[\alpha]_{\text{D}}^{25} = 56.0$ (c 0.53, CHCl_3 , 91% ee). Ee was determined by HPLC analysis using a chiral column (Daicel Chiralpak AS-H: 4 x 250mm, 254nm UV detector, rt, eluent: 20% 2-propanol in hexane, flow rate: 1.0 mL/min, retention time: 15 min for minor isomer and 18 min for major isomer).

3,4-Bis(methoxymethyl)-6-methyl-8-tosyl-8-azabicyclo[4.3.0]nona-1,3-diene (2d). Colorless oil. IR (neat) 2927, 2823, 2360, 2339, 1340, 1161, 1091 cm^{-1} , ^1H NMR $\delta = 1.00$ (s, 3H), 2.13 (d, $J = 16.6$ Hz, 1H), 2.30 (d, $J = 16.6$ Hz, 1H), 2.43 (s, 3H), 2.82 (d, $J = 9.2$ Hz, 1H), 3.27 (s, 3H x 2), 3.41 (d, $J = 9.2$ Hz, 1H), 3.79 (d, $J = 15.2$ Hz, 1H), 3.91 (d, $J = 12.0$ Hz, 1H), 3.97-4.0 (m, 2H), 4.03 (d, $J = 12.0$ Hz, 1H), 4.20 (d, $J = 15.2$ Hz, 1H), 5.76 (s, 1H), 7.32 (d, $J = 8.2$ Hz, 2H), 7.71 (d, $J = 8.2$ Hz, 2H); ^{13}C NMR $\delta = 21.6, 21.7, 37.3, 41.2, 50.0, 57.8, 57.9, 62.0, 69.7, 70.7, 116.8, 127.5, 129.1, 129.4, 129.5, 133.0, 142.3, 143.4$; HRMS (FAB⁺) for M-1 found m/z 376.1566, calcd for $\text{C}_{20}\text{H}_{26}\text{NO}_4\text{S}$: 376.1582. $[\alpha]_{\text{D}}^{26} = 11.6$ (c 0.82, CHCl_3 , 98% ee). Ee was determined by HPLC analysis using a chiral column (Daicel Chiralpak AD-H: 4 x 250mm, 254nm UV detector, rt, eluent: 10% 2-propanol in hexane, flow rate: 1.0 mL/min, retention time: 18 min for minor isomer and 23 min for major isomer).

3,4-Bis(methoxymethyl)-6-phenyl-8-tosyl-8-azabicyclo[4.3.0]nona-1,3-diene (2e). White solid. mp 123°C, IR (neat) 2929, 2821, 2360, 2343, 1338, 1159, 1084 cm^{-1} , ^1H NMR $\delta = 2.37$ (s, 3H), 2.45 (d, $J = 16.4$ Hz, 1H), 2.75 (s, 3H), 2.79 (d, $J = 16.4$ Hz, 1H), 3.19 (s, 3H), 3.31 (d, $J = 9.6$ Hz, 1H), 3.75 (d, $J = 12.4$ Hz, 1H), 3.81 (d, $J = 12.4$ Hz, 1H), 3.89 (d, $J = 11.6$ Hz, 1H), 3.96-4.03 (m, 2H), 4.11 (d, $J = 9.6$ Hz, 1H), 4.17 (d, $J = 15.2$ Hz, 1H), 6.18 (s, 1H), 7.12-7.17 (m, 7H), 7.49 (d, $J = 8.00$ Hz, 2H); ^{13}C NMR $\delta = 21.5, 39.0, 49.4, 50.6, 56.8, 57.7, 63.7, 69.3, 69.9, 120.6, 126.2, 126.7, 127.2, 128.2, 129.3, 130.2, 130.3, 133.2, 139.2, 141.4, 143.1$; HRMS (FAB⁺) for M-1 found m/z 438.1742, calcd for $\text{C}_{25}\text{H}_{28}\text{NO}_4\text{S}$: 438.1738. $[\alpha]_{\text{D}}^{26} = 38.1$ (c 0.23, CHCl_3 , 95% ee). Ee was determined by HPLC analysis using a chiral column (Daicel Chiralpak AD-H: 4 x 250mm, 254nm UV detector, rt, eluent: 10% 2-propanol in hexane, flow

rate: 1.0 mL/min, retention time: 26 min for minor isomer and 29 min for major isomer).

Dimethyl 3,4-bis(methoxymethyl)-6-methylbicyclo[4.3.0]nona-1,3-diene-8,8-dicarboxylate (2f).

Colorless oil. IR (neat) 2954, 2823, 1378 cm^{-1} ; ^1H NMR δ = 0.97 (s, 3H), 2.30 (s, 2H), 2.31 (d, J = 12.8 Hz, 1H), 2.39 (d, J = 12.8 Hz, 1H), 2.93 (d, J = 18.8 Hz, 1H), 3.29 (s, 3H x 2), 3.35 (d, J = 18.8 Hz, 1H), 3.71 (s, 3H), 3.75 (s, 3H), 3.91 (d, J = 11.6 Hz, 1H), 3.99 (d, J = 11.2 Hz, 1H), 4.03 (d, J = 11.2 Hz, 1H), 4.07 (d, J = 11.6 Hz, 1H), 5.77 (s, 1H); ^{13}C NMR δ = 23.2, 38.4, 40.3, 41.0, 47.9, 52.9, 52.9, 57.7, 57.8, 59.5, 69.9, 70.9, 116.9, 129.5, 129.5, 147.4, 172.3, 172.5; HRMS (FAB) for M-1 found m/e 337.1652, calcd for $\text{C}_{18}\text{H}_{25}\text{O}_6$: 337.1651. $[\alpha]_{\text{D}}^{22}$ = 25.9 (c 1.05, CHCl_3 , 92% ee). Ee was determined by HPLC analysis using a chiral column (Daicel Chiralpak AD-H: 4 x 250mm, 254nm UV detector, rt, eluent: 5% 2-propanol in hexane, flow rate: 0.5 mL/min, retention time: 15 min for minor isomer and 18 min for major isomer).

3,4-Bis(methoxymethyl)-6-methyl-8-oxabicyclo[4.3.0]nona-1,3-diene (2g). Colorless oil. IR (neat) 2924, 2819, 1093 cm^{-1} ; ^1H NMR δ = 1.10 (s, 3H), 2.25 (d, J = 16.6 Hz 1H), 2.32 (d, J = 16.6 Hz, 1H), 3.31 (s, 3H x 2), 3.52 (d, J = 8.0 Hz, 1H), 3.91 (d, J = 8.0 Hz, 1H), 3.98 (d, J = 12.0 Hz, 1H), 4.05 (s, 2H), 4.10 (d, J = 12.0 Hz, 1H), 4.37 (d, J = 14.4 Hz, 1H), 4.61 (d, J = 14.4 Hz, 1H), 5.81 (s, 1H); ^{13}C NMR δ = 21.0, 36.1, 41.8, 57.7, 57.8, 68.9, 69.9, 71.0, 81.5, 114.3, 129.1, 129.8, 147.2; HRMS (FAB) for M-1 found m/e 223.1328, calcd for $\text{C}_{13}\text{H}_{19}\text{O}_3$: 223.1334. $[\alpha]_{\text{D}}^{24}$ = 23.5 (c 0.42, CHCl_3 , 92% ee). Ee was determined by HPLC analysis using a chiral column (Daicel Chiralpak AD-H: 4 x 250mm, 254nm UV detector, rt, eluent: 1%, 2-propanol in hexane, flow rate: 0.5 mL/min, retention time: 28 min for minor isomer and 30 min for major isomer).

3,4-Bis(hydroxymethyl)-2,6-dimethyl-8-tosyl-8-azabicyclo[4.3.0]nona-1,3-diene (3a). White solid. mp 120 $^{\circ}\text{C}$, IR (neat) 3413, 2924, 1342, 1159, 1047 cm^{-1} ; ^1H NMR δ = 0.88 (s, 3H), 1.25 (s, 1H x 2), 1.72 (s, 3H), 2.19 (d, J = 16.8 Hz, 1H), 2.24 (d, J = 16.8 Hz, 1H), 2.44 (s, 3H), 2.76 (d, J = 8.8 Hz, 1H), 3.54 (d, J = 8.8 Hz, 1H), 3.76 (d, J = 15.0 Hz, 1H), 4.14 (d, J = 15.0 Hz, 1H), 4.12-4.31 (m, 2H), 4.24 (d, J = 11.2 Hz, 1H), 4.30 (d, J = 11.2 Hz, 1H), 7.34 (d, J = 8.4 Hz, 2H), 7.73 (d, J = 8.4 Hz, 2H); ^{13}C NMR δ = 13.9, 21.2, 21.6, 38.2, 40.9, 49.6, 57.4, 62.2, 62.7, 123.3, 127.7, 129.6, 132.8, 133.4, 133.6, 136.9, 143.5; HRMS (FAB) for M-1 found m/e 362.1430, calcd for $\text{C}_{19}\text{H}_{24}\text{O}_4\text{NS}$: 362.1426. $[\alpha]_{\text{D}}^{24}$ = 41.5 (c 0.92, CHCl_3 , 98% ee). Ee was determined by HPLC analysis using a chiral column (Daicel Chiralpak doubly-arrayed AD-H: 4 x 250mm, 254nm UV detector, rt, eluent: 20%, 2-propanol in hexane, flow rate: 1.0 mL/min, retention time: 24 min for minor isomer and 27 min for major isomer).

2,6-Dimethyl-8-tosyl-8-azabicyclo[4.3.0]nona-1,3-diene (4a). Colorless oil. IR (neat); ^1H NMR δ = 1.02 (s, 3H), 1.59 (s, 3H), 2.02-2.14 (m, 2H), 2.44 (s, 3H), 2.77 (d, J = 8.8 Hz, 1H), 3.52 (d, J = 8.8 Hz, 1H), 3.74 (d, J = 14.6 Hz, 1H), 4.11 (d, J = 14.6 Hz, 1H), 5.69-5.70 (m, 1H), 5.70-5.72 (m, 1H), 7.33 (d, J = 7.8 Hz, 2H), 7.73 (d, J = 7.8 Hz, 2H); ^{13}C NMR δ = 17.5, 21.5, 34.2, 40.9, 49.0, 62.3, 122.2, 123.2, 127.7, 127.8, 129.6, 134.5, 143.5; HRMS (FAB) for M+1 found m/e 304.1371, calcd for $\text{C}_{17}\text{H}_{22}\text{O}_2\text{NS}$: 304.1371. $[\alpha]_{\text{D}}^{27}$ = 50.7 (c 1.025, CHCl_3 , 90.9% ee). Ee was determined by HPLC analysis using a chiral column (Daicel Chiralpak doubly-arrayed AD-H: 4 x 250mm, 254nm UV detector, rt, eluent: 5%, 2-propanol in

hexane, flow rate: 0.5 mL/min, retention time: 34 min for minor isomer and 35 min for major isomer).

Mixture (4/1) of 3-methoxymethyl-2,6-dimethyl-8-tosyl-8-azabicyclo[4.3.0]nona-1,3-diene (5aa) and 4-methoxymethyl-2,6-dimethyl-8-tosyl-8-azabicyclo[4.3.0]nona-1,3-diene (5ab). Colorless oil. IR (neat); ^1H NMR δ = 1.01 (s, 3H x 0.2), 1.03 (s, 3H x 0.8), 1.59 (s, 3H), 2.04-2.16 (m, 2H), 2.44 (s, 3H), 2.77 (d, J = 8.6 Hz, 1H x 0.8), 2.79 (d, J = 8.8 Hz, 1H x 0.2), 3.27 (s, 3H x 0.8), 3.28 (s, 3H x 0.2), 3.51 (d, J = 8.6 Hz, 1H x 0.8), 3.53 (d, J = 8.8 Hz, 1H x 0.2), 3.75-3.86 (m, 2H), 4.02 (d, J = 13.4 Hz, 1H), 4.12 (d, J = 13.4 Hz, 1H), 5.62-5.63 (br, 1H x 0.8), 5.69 (s, 1H x 0.2), 7.33 (d, J = 8.2 Hz, 2H), 7.73 (d, J = 8.2 Hz, 2H); ^{13}C NMR δ = 14.4, 17.5, 21.5, 21.5, 21.9, 34.1, 35.6, 41.0, 41.8, 48.9, 49.3, 57.5, 57.7, 62.0, 62.3, 73.3, 75.5, 121.9, 122.8, 123.0, 124.6, 127.7, 127.7, 129.6, 132.8, 133.0, 133.2, 134.0, 134.5, 136.9, 143.5, 143.5; HRMS (FAB) for M-1 found m/e 346.1461, calcd for $\text{C}_{19}\text{H}_{24}\text{O}_3\text{NS}$: 346.1477. Ee was determined by HPLC analysis using a chiral column (Daicel Chiralpak AS-H: 4 x 250mm, 254nm UV detector, rt, eluent: 5%, 2-propanol in hexane, flow rate: 0.5 mL/min, retention time: **5aa**: 31 min for minor isomer and 33 min for major isomer. **5ab**: 26 min for minor isomer and 35 min for major isomer).

Mixture (7/1) of 3-hydroxymethyl-2,6-dimethyl-8-tosyl-8-azabicyclo[4.3.0]nona-1,3-diene (6aa) and 4-hydroxymethyl-2,6-dimethyl-8-tosyl-8-azabicyclo[4.3.0]nona-1,3-diene (6ab). Colorless oil. IR (neat) 3543, 2924, 2360, 2341, 1342, 1165, 1093 cm^{-1} , ^1H NMR δ = 1.00 (s, 3H x 0.875), 1.03 (s, 3H, 0.125), 1.29 (bs, 1H), 1.52 (s, 3H x 0.125), 1.68 (s, 3H x 0.875), 2.06-2.16 (m, 2H), 2.44 (s, 3H), 2.76 (d, J = 8.8 Hz, 1H), 3.52 (d, J = 8.8 Hz, 1H), 3.76 (d, J = 15.2 Hz, 1H), 4.09-4.21 (m, 3H), 5.63-5.65 (br, 1H x 0.875), 5.70 (bs, 1H x 0.125), 7.34 (d, J = 8.2 Hz, 2H), 7.73 (d, J = 8.2 Hz, 2H); ^{13}C NMR (**6aa**) δ = 14.5, 21.5, 21.7, 34.0, 41.1, 49.3, 62.3, 63.7, 121.2, 122.3, 127.6, 129.5, 132.9, 136.7, 137.1, 143.4; HRMS (FAB^+) for M+1 found m/z 334.1478, calcd for $\text{C}_{18}\text{H}_{24}\text{NO}_3\text{S}$: 334.1478. Ee was determined by HPLC analysis using a chiral column (Daicel Chiralpak AS-H: 4 x 250mm, 254nm UV detector, rt, eluent: 20% 2-propanol in hexane, flow rate: 1.0 mL/min, retention time: **6aa**: 17 min for minor isomer and 19 min for minor isomer. **6ab**: 24min for major isomer and 30 min for major isomer).

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DFILE	A:\yun.Me.Ph2_default
COMNT	
DATIM	Thu Jul 14 18:13:52
OBNUC	¹ H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.0 Hz
POINT	32768
FREQU	8000.0 Hz
SCANS	16
ACQTM	4.096 sec
PD	2.904 sec
PW1	6.7 us
IRNUC	¹ H
CTEMP	29.2 C
SLVNT	CDCL3
EXREF	0.00 ppm
BF	1.00 Hz
RGAIN	21

A:\yun.Me.Ph.Carbon_default.als
nmu050713c-140Griecco

143.065
142.123
133.958
133.199
132.723
132.074
129.340
128.023
127.520
127.274
126.551
126.423

77.320
77.000
76.680
70.407
66.530
63.961
57.468
56.838
50.117
49.147

38.668

21.578

15.114

2c

¹³C NMR

DFILE	A:\yun.Me.Ph.Carbon_
COMNT	nmu050713c-140Griecco
DATIM	Thu Jul 14 09:05:00
OBNUC	13C
EXMOD	BCM
OBFRQ	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.0 Hz
POINT	32768
FREQU	30120.4 Hz
SCANS	130
ACQTM	1.088 sec
PD	1.794 sec
PW1	4.8 us
IRNUC	1H
CTEMP	29.1 C
SLVNT	CDCL3
EXREF	77.00 ppm
Bf	0.12 Hz
RGAIN	25

DFILE	A:\yun.H.Me2_default
COMNT	
DATIM	Tue Jul 12 16:29:14
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.0 Hz
POINT	32768
FREQU	8000.0 Hz
SCANS	16
ACQTM	4.096 sec
PD	2.904 sec
PW1	6.7 us
IRNUC	1H
CTEMP	29.7 C
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	21

143.422
142.288
133.025
129.523
129.413
129.102
127.530
116.776

77.311
77.000
76.680
70.746
69.685
61.995
57.944
57.816
49.970
41.210
37.260

21.688
21.615

¹³C NMR

DFILE	A:\yun.H,Me.Carbon_d
COMNT	
DATIM	Tue Jul 12 19:38:28
OBNUC	13C
EXMOD	BCM
OBFRQ	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.0 Hz
POINT	32768
FREQU	30120.4 Hz
SCANS	130
ACQTM	1.088 sec
PD	1.794 sec
PW1	4.8 us
IRNUC	1H
CTEMP	29.7 C
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	25

```

DFILE      A:\yun.H.Ph_default.
COMNT
DATIM      Fri Jul 15 13:21:23
OBNUC      1H
EXMOD      NON
OBFRO      399.65 MHz
OBSET      124.00 KHz
OBFIN      10500.0 Hz
POINT      32768
FREQU      8000.0 Hz
SCANS      16
ACQTM      4.096 sec
PD          2.904 sec
PW1        6.7 us
IRNUC      1H
CTEMP      30.4 C
SLVNT      CDCL3
EXREF      0.00 ppm
BF          1.00 Hz
RGAIN      22

```

143.074
141.447
139.170
133.226
130.319
130.163
129.331
128.206
127.191
126.661
126.158
120.553

77.311
77.000
76.680
69.859
69.264
63.668
57.715
56.783
50.620
49.385

38.988

21.542

DFILE	A:\yun.H.Ph.Carbon_d
COMNT	
DATIM	Sat Jul 16 08:47:30
OBNUC	13C
EXMOD	BCM
OBFRQ	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.0 Hz
POINT	32768
FREQU	30120.4 Hz
SCANS	130
ACQTM	1.088 sec
PD	1.794 sec
PW1	4.8 us
IRNUC	1H
CTEMP	30.8 C
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.00 Hz
RGAIN	25

7.266
5.772
4.085
4.056
4.043
4.015
4.004
3.989
3.976
3.929
3.900
3.747
3.745
3.715
3.712
3.376
3.325
3.292
3.289
2.951
2.908
2.411
2.377
2.325
2.295
1.617
0.970
0.000

DFILE	C:\Documents and Set
COMNT	
DATIM	Tue Jun 28 18:06:12
OBNUC	1H
EXMOD	NON
OBFRO	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.0 Hz
POINT	32768
FREQU	16000.0 Hz
SCANS	16
ACQTM	2.048 sec
PD	2.904 sec
PW1	6.7 us
IRNUC	1H
CTEMP	32.0 C
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	17

DFILE A:\Ex259dataac_default.
 COMNT Tue Jun 28 18:19:43 20
 DATIM 13C
 OBNUC 13C
 EXMOD BCM
 OBFRQ 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.0 Hz
 POINT 32768
 FREQU 27173.9 Hz
 SCANS 242
 ACQTM 1.206 sec
 PD 1.794 sec
 PW1 4.8 us
 IRNUC 1H
 CTEMP 32.1 C
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 25

DFILE	E:\DataOHMeOMe_defau
COMNT	Sat Jul 30 15:57:12
DATIM	1H
OBNUC	NON
EXMOD	
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.0 Hz
POINT	32768
FREQU	8000.0 Hz
SCANS	16
ACQTM	4.096 sec
PD	2.904 sec
PWL	6.7 us
IRNUC	1H
CTEMP	27.7 C
SLVNT	CDCL3
EXREF	0.00 ppm
BF	1.20 Hz
RGAIN	13

E:\ZÅ"CEr\QV"ä\4ara1OMeHC\4ara1OMeHC.nm.fid

FILE	E:\ZÅ"CEr\QV"ä\4ara
COMNT	
DATIM	Mon Aug 1 15:44:15
OBNUC	13C
EXMOD	bcm
OBFRO	125.65 MHz
OBFSE	120.00 KHz
OBFIN	7259.7 Hz
POINT	32768
FREQU	26385.2 Hz
SCANS	252
ACQTM	1.242 sec
PD	1.750 sec
PW1	5.9 us
IRNUC	1H
CTEMP	27.1 C
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.00 Hz
RGAIN	28

C:\Documents and Settings\Z\u\Documents\120\fffx\Nfgfbfv\araiData\DataNd1014_default.als

DFILE	COMNT	OBFRQ	OBSET	OBFIN	POINT	FREQU	SCANS	ACQTM	PD	PM1	IRNUC	CTEMP	SLVNT	EXREF	BF	RGAIN
	DATIM	EXMOD	OBNUC													
	Fri Jul 29 17:50:54	NON														
		399.65 MHz	124.00 KHz	10500.0 Hz	32768	8000.0 Hz	16	4.096 sec	2.904 sec	6.7 us	27.5 C					
												0.00 ppm				
												0.12 Hz				23

143.549
136.924
133.641
133.360
132.783
129.615
127.652
123.255

77.322
77.000
76.687

62.745
62.200
57.399

49.628

40.933
38.236

21.605
21.242
14.947

DFILE E:\DataCndiol_default
COMNT Tue Jul 26 18:35:26
DATIM 13C
OBNUC BCM
EXMOD
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 27173.9 Hz
SCANS 401
ACQTM 1.206 sec
PD 1.794 sec
PWL 4.8 us
IRNUC 1H
CTEMP 26.3 C
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 24

7.738
7.718
7.343
7.324
7.265

5.723
5.716
5.699
5.693
5.622
5.607

4.132
4.096
3.758
3.721
3.526
3.504
2.780
2.758
2.435
2.399
2.140
2.126
2.098
2.084
2.063
2.044
2.021
1.619
1.587
1.020
0.984

0.000

¹H NMR

1.0000
1.0190

0.4897
0.5020

0.5625
0.5216
0.5139

0.5014

1.5879

1.0817

2.1692

1.5128

FILE	C:\Documents and Set
COMNT	Sat Jul 30 13:59:43
DATIM	1H
OBNUC	NON
EXMOD	399.65 MHz
OBFRQ	124.00 KHz
OBSET	10500.0 Hz
OBFIN	32768
POINT	8000.0 Hz
FREQU	16
SCANS	4.096 sec
ACQTM	2.904 sec
PD	6.7 us
PW1	28.0 C
IRNUC	0.00 ppm
CTEMP	1.20 Hz
SLVNT	16
EXREF	
BF	
RGAIN

DFILE	C:\Documents and Set
COMNT	Mon Aug 1 15:21:38
DATIM	13C
OBNUC	bcm
EXMOD	125.65 MHz
OBFRQ	120.00 KHz
OBSET	7259.7 Hz
OBFIN	32768
POINT	26385.2 Hz
FREQU	252
SCANS	1.242 sec
ACQTM	1.750 sec
PD	5.9 us
PW1	1H
IRNUC	26.6 c
CTEMP	CDCL3
SLVNT	77.00 ppm
EXREF	0.10 Hz
BF	28
RGAIN

DFILE	C:\Documents and Set
COMNT	Sat Jul 30 19:24:17
DATIM	1H
OBNUC	NON
EXMOD	399.65 MHz
OBFRQ	124.00 KHz
OBSET	10500.0 Hz
OBFIN	32768
POINT	8000.0 Hz
FREQU	16
SCANS	4.096 sec
ACQTM	2.904 sec
PD	6.7 us
PW1	27.9 C
IRNUC	1H
CTEMP	0.00 ppm
SLVNT	0.12 Hz
EXREF	20
BF	
RGAIN

+ ¹H NMR

143.515
143.484
136.912
134.522
134.016
133.164
133.032
132.800
129.645
127.713
127.686
124.559
123.001
122.782
121.926

77.212
77.000
76.791
75.456
73.252
62.257
62.015
57.731
57.451
49.311
48.930
41.771
41.033
35.606
34.073
21.928
21.531
21.497
17.466
14.422
-0.024

DEFILE F:\208.als
COMNT
DATIM
OBNUC 13C
EXMOD
OBFRO 150.91 MHz
OBSET 7.90 KHz
OBFIN 5.5 Hz
POINT 65536
FREQU 33333.3 Hz
SCANS 8
ACQTM 0.000 sec
PD 0.000 sec
PWL 10.0 us
IRNUC
CTEMP 0.0 C
SLVNT
EXREF 77.00 ppm
BF 10.00 Hz
RGAIN 0

FILE	C:\Documents and Set
COMNT	Tue Aug 02 07:43:35
DATIM	1H
OBNUC	NON
EXMOD	399.65 MHz
OBFRQ	124.00 KHz
OBSET	10500.0 Hz
OBFIN	32768
POINT	8000.0 Hz
FREQU	16
SCANS	4.096 sec
ACQTM	2.904 sec
PD	6.7 us
PWL	26.8 c
IRNUC	1H
CTEMP	0.00 ppm
SLVNT	0.12 Hz
EXREF	19
BF	
RGAIN

A:\propalgy1.carbon.256_default.als

143.394
137.103
136.683
132.925
129.541
127.603
122.318
121.193

77.320
77.000
76.680

63.714
62.260

49.349

41.146

34.042

21.652
21.496

14.547

DFILE	A:\propalgy1.carbon.
COMNT	
DATIM	Tue Aug 02 07:59:31
OBNUC	13C
EXMOD	BCM
OBFRQ	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.0 Hz
POINT	32768
FREQU	30120.4 Hz
SCANS	257
ACQTM	1.088 sec
PD	1.794 sec
PW1	4.8 us
IRNUC	1H
CTEMP	26.9 C
SLVNT	CDCL3
EXREF	77.00 ppm
B ^F	1.00 Hz
RGAIN	24