Supporting Information

Iridium Complex-Catalyzed Reaction of Enynes: Cycloaddition with Monoynes and Cycloisomerization

Satoko Kezuka, Toshiaki Okado, Eri Niou, and Ryo Takeuchi

*Department of Chemistry and Biological Science, Aoyama Gakuin University,
5-10-1, Fuchinobe, Sagamihara 229-8558, Japan*

Table of Contents

General	S2
Materials	S2
A typical procedure and data: Cycloaddition of enyne with monoyne	S2
A typical procedure and data: Cycloisomerization of enyne	S4
Diastereoselective cycloaddition of enyne 13 with monoyne	S6
References	S6
NMR data of (Z)-4a, (Z)-4b, and (Z)-4c (NOESY)	S7-9
NMR data (¹³C, ¹H)	S10-17
NMR data of 15b	S18-20

General. The ^1H NMR spectra and ^{13}C NMR spectra were measured using a JEOL JNM-ECP 500A spectrometer with CDCl_3 as the solvent and tetramethylsilane (Me_4Si) as the internal standard. GC analyses were performed on a Shimadzu GC-14B using 3.2 mm x 2 m glass columns packed with 5% OV-17 on 60/80 mesh Chromosorb WAW-DMCS. The products were purified by column chromatography on 70 - 230 mesh silica gel (Merck; Silica Gel 60). Elemental analyses were carried out on a Yanaco MT-5 CHN analyzer. High-resolution mass spectra were obtained with a JEOL Mstation JMS-700.

Materials. All reagents and the solvents were dried and purified before use by the usual procedures. 3-Hexyne (**2a**), 4-octyne (**2b**), and 5-decyne (**2c**) were purchased. 1,4-Dimethoxy-2-butyne (**2d**) was prepared by the reaction of disodio-2-butyne-1,4-diol with iodomethane. Enynes **1a-e** were prepared by the reaction of diethyl sodioallylmalonate with 1-bromo-2-alkynes.¹ 1-Bromo-2-alkynes were prepared by bromination of 2-alkyn-1-ols.² Enyne **13** was prepared by the reaction of diethyl sodio(1-phenylallyl)malonate³ with 1-bromo-2-butyne. $[\text{Ir}(\text{cod})\text{Cl}]_2$ was prepared by previously published methods.⁴ Bis(diphenylphosphino)methane, 1,2-bis(diphenylphosphino)ethane, 1,3-bis(diphenylphosphino)propane, 1,4-bis(diphenylphosphino)butane, 1,2-bis(diphenylphosphino)benzene, *cis*-1,2-bis(diphenylphosphino)ethylene, 1,1'-bis(diphenylphosphino)ferrocene, triphenylphosphine, methyldiphenylphosphine, 1,6-bis(diphenylphosphino)hexane, 2,2'-bis(diphenylphosphino)-1,1'-biphenyl, and 1,2-bis(dipentafluorophenylphosphino)ethane were purchased.

Cycloaddition of enyne with monoyne. A typical procedure (Table 1, entry 2). A flask was charged with $[\text{Ir}(\text{cod})\text{Cl}]_2$ (13.6 mg, 0.021 mmol) and dppe (15.6 mg, 0.040 mmol). The flask was evacuated and filled with argon. To the flask were added 5 mL of toluene and 3-hexyne (251.3 mg, 3.1 mmol) with stirring. Enyne **1a** (246.6 mg, 0.98 mmol) was then added. The reaction mixture was stirred under reflux for 3 h. The progress of the reaction was monitored by GLC. The solution was evaporated *in vacuo*. Column chromatography of the residue gave **3a** and **4a** as colorless oils (*n*-hexane/AcOEt=99/1; **3a** 245.2 mg, 75% yield; **4a** 25.1 mg, 10% yield).

Diethyl 5,6-diethyl-7-methyl-3a,4-dihydroindan-2,2-dicarboxylate (3a): ^1H NMR (500 MHz, CDCl_3) δ 0.93 (t, $J = 7.3$ Hz, 3H), 0.99 (t, $J = 7.3$ Hz, 3H), 1.25 (t, $J = 7.3$ Hz, 3H), 1.26 (t, $J = 7.3$ Hz, 3H), 1.73 (dt, $J = 2.8, 1.4$ Hz, 3H), 1.76-1.85 (m, 2H), 1.96-2.26 (m, 4H), 2.09 (dd, $J = 15.6, 6.9$ Hz, 1H), 2.42-2.55 (m, 1H), 2.65 (ddd, $J = 12.4, 6.9, 1.4$ Hz, 1H), 2.94 (d, $J = 17.9$ Hz, 1H), 3.09 (d, $J = 17.9$ Hz, 1H), 4.18 (q, $J = 7.3$ Hz, 2H), 4.20 (q, $J = 7.3$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 13.8, 14.0 (2C), 15.0, 15.1, 20.6, 26.3, 34.0, 37.3, 38.5, 41.5, 59.5, 61.4 (2C), 125.2, 132.4, 133.0,

134.6, 172.0 (2C). Anal. Calcd for C₂₀H₃₀O₄: C, 71.82; H, 9.04. Found: C, 72.00; H, 9.09.

Diethyl 7-methyl-5,6-di-*n*-propyl-3a,4-dihydroindan-2,2-dicarboxylate (3b): ¹H NMR (500 MHz, CDCl₃) δ 0.83 (t, *J* = 7.3 Hz, 3H), 0.84 (t, *J* = 7.3 Hz, 3H), 1.17 (t, *J* = 7.3 Hz, 3H), 1.18 (t, *J* = 7.3 Hz, 3H), 1.22-1.40 (m, 4H), 1.65 (dt, *J* = 2.3, 1.4 Hz, 3H), 1.66-1.75 (m, 2H), 1.85-2.18 (m, 4H), 2.01 (dd, *J* = 15.1, 6.9 Hz, 1H), 2.35-2.50 (m, 1H), 2.56 (ddd, *J* = 12.6, 7.3, 1.4 Hz, 1H), 2.87 (d, *J* = 18.3 Hz, 1H), 3.09 (d, *J* = 18.3 Hz, 1H), 4.11 (q, *J* = 7.3 Hz, 2H), 4.12 (q, *J* = 7.3 Hz, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 13.97, 14.02, 14.19, 14.20, 15.3, 22.1, 23.5, 29.9, 34.6, 35.6, 37.4, 38.3, 41.5, 59.6, 61.3, 61.4, 125.5, 131.1, 132.3, 134.7, 172.0, 172.1. HRMS: Calcd for C₂₂H₃₄O₄ (M⁺), 362.2457. Found: *m/z* 362.2464.

Diethyl 5,6-di-*n*-butyl-7-methyl-3a,4-dihydroindan-2,2-dicarboxylate (3c): ¹H NMR (500 MHz, CDCl₃) δ 0.91 (t, *J* = 7.3 Hz, 6H), 1.24 (t, *J* = 7.3 Hz, 3H), 1.25 (t, *J* = 7.3 Hz, 3H), 1.19-1.50 (m, 8H), 1.72 (dt, *J* = 2.3, 1.8 Hz, 3H), 1.73-1.83 (m, 2H), 1.95-2.22 (m, 4H), 2.08 (dd, *J* = 15.2, 6.9 Hz, 1H), 2.42-2.53 (m, 1H), 2.64 (ddd, *J* = 12.4, 6.9, 1.4 Hz, 1H), 2.94 (d, *J* = 17.9 Hz, 1H), 3.08 (d, *J* = 17.9 Hz, 1H), 4.18 (q, *J* = 7.3 Hz, 2H), 4.20 (q, *J* = 7.3 Hz, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 13.99, 14.01, 14.04, 14.1, 15.3, 22.9, 23.0, 27.6, 31.3, 32.7, 33.3, 34.6, 37.4, 38.4, 41.5, 59.6, 61.3, 61.4, 125.5, 131.2, 132.2, 134.6, 172.0, 172.1. HRMS: Calcd for C₂₄H₃₈O₄ (M⁺), 390.2770. Found: *m/z* 390.2748.

Diethyl 5,6-dimethoxymethyl-7-methyl-3a,4-dihydroindan-2,2-dicarboxylate (3d): ¹H NMR (500 MHz, CDCl₃) δ 1.24 (t, *J* = 7.3 Hz, 3H), 1.26 (t, *J* = 7.3 Hz, 3H), 1.73-1.90 (m, 2H), 1.80 (dt, *J* = 2.8, 1.4 Hz, 3H), 2.44 (dd, *J* = 15.5, 7.3 Hz, 1H), 2.55-2.64 (m, 1H), 2.69 (ddd, *J* = 12.2, 6.9, 1.4 Hz, 1H), 3.08 (d, *J* = 18.3 Hz, 1H), 3.11 (d, *J* = 18.3 Hz, 1H), 3.32 (s, 3H), 3.33 (s, 3H), 3.95 (d, *J* = 11.5 Hz, 1H), 4.03 (d, *J* = 11.0 Hz, 1H), 4.06 (dd, *J* = 11.0, 1.4 Hz, 1H), 4.15-4.23 (m, 2H), 4.17 (d, *J* = 11.5 Hz, 1H), 4.20 (q, *J* = 7.3 Hz, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 14.0 (2C), 14.7, 32.2, 37.3, 38.0, 41.3, 57.6, 57.8, 59.4, 61.4 (2C), 67.2, 71.3, 124.3, 132.9, 133.0, 137.7, 171.7, 171.8. Anal. Calcd for C₂₀H₃₀O₆: C, 65.55; H, 8.25. Found: C, 65.77; H, 8.25.

Diethyl 5,6,7-triethyl-3a,4-dihydroindan-2,2-dicarboxylate (3e): ¹H NMR (500 MHz, CDCl₃) δ 0.93 (t, *J* = 7.3 Hz, 3H), 0.94 (t, *J* = 7.3 Hz, 3H), 1.00 (t, *J* = 7.3 Hz, 3H), 1.24 (t, *J* = 6.9 Hz, 3H), 1.26 (t, *J* = 6.9 Hz, 3H), 1.74-1.83 (m, 2H), 1.95-2.30 (m, 6H), 2.07 (dd, *J* = 14.7, 6.9 Hz, 1H), 2.39-2.53 (m, 1H), 2.63 (ddd, *J* = 12.6, 7.3, 1.4 Hz, 1H), 2.93 (dd, *J* = 17.9, 3.2 Hz, 1H), 3.11 (d, *J* = 17.9 Hz, 1H), 4.18 (q,

$J = 6.9$ Hz, 2H), 4.20 (q, $J = 6.9$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 13.86, 13.89, 13.99, 14.03, 15.4, 20.3, 22.4, 26.4, 34.1, 36.8, 38.3, 41.2, 59.6, 61.3, 61.4, 131.8, 132.6, 133.2, 134.3, 172.0, 172.1. HRMS: Calcd for $\text{C}_{21}\text{H}_{32}\text{O}_4$ (M^+), 348.2301. Found: m/z 348.2287.

Diethyl 7-*n*-butyl-5,6-diethyl-3a,4-dihydroindan-2,2-dicarboxylate (3f): ^1H NMR (500 MHz,

CDCl_3) δ 0.90 (t, $J = 7.8$ Hz, 3H), 0.91 (t, $J = 6.9$ Hz, 3H), 0.99 (t, $J = 7.3$ Hz, 3H), 1.22-1.34 (m, 4H), 1.24 (t, $J = 7.3$ Hz, 3H), 1.26 (t, $J = 7.3$ Hz, 3H), 1.72-1.83 (m, 2H), 1.94-2.31 (m, 6H), 2.06 (dd, $J = 15.1, 6.9$ Hz, 1H), 2.39-2.53 (m, 1H), 2.62 (dd, $J = 12.4, 6.9$ Hz, 1H), 2.92 (dd, $J = 17.9, 3.2$ Hz, 1H), 3.12 (d, $J = 17.9$ Hz, 1H), 4.18 (q, $J = 7.3$ Hz, 2H), 4.20 (q, $J = 7.3$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 13.9, 14.00, 14.04, 14.05, 15.4, 20.4, 22.9, 26.4, 29.4, 31.7, 34.1, 37.1, 38.5, 41.2, 59.6, 61.3, 61.4, 130.4, 132.9, 133.2, 134.8, 172.0, 172.1. HRMS: Calcd for $\text{C}_{23}\text{H}_{36}\text{O}_4$ (M^+), 376.2614. Found: m/z 376.2618.

Cycloisomerization of enyne. A typical procedure (Table 3, entry 11). A flask was charged with $[\text{Ir}(\text{cod})\text{Cl}]_2$ (14.7 mg, 0.022 mmol) and dppf (25.3 mg, 0.046 mmol). The flask was evacuated and filled with argon. To the flask were added 5 mL of benzene and enyne **1a** (252.3 mg, 1.0 mmol) with stirring. The reaction mixture was stirred under reflux for 2 h. The progress of the reaction was monitored by GLC. The solution was evaporated *in vacuo* and column chromatography of the residue gave **4a** as a colorless oil (*n*-hexane/AcOEt=98/2; **4a** 230.9 mg, 92% yield).

Diethyl (Z)-3-ethylidene-4-methylenecyclopentane-1,1-dicarboxylate ((Z)-4a): ^1H NMR (400 MHz, CDCl_3) δ 1.24 (t, $J = 6.4$ Hz, 6H), 1.83 (d, $J = 7.3$ Hz, 3H), 2.98 (s, 2H), 3.05 (s, 2H), 4.18 (q, $J = 6.4$ Hz, 4H), 5.16 (s, 1H), 5.23 (s, 1H), 5.65 (q, $J = 7.3$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 14.0 (2C), 15.1, 42.5, 42.9, 57.5, 61.4 (2C), 110.6, 121.4, 136.0, 144.5, 171.3 (2C). Anal. Calcd for $\text{C}_{14}\text{H}_{20}\text{O}_4$: C, 66.65; H, 7.99. Found: C, 66.41; H, 8.07.

Stereochemistry of (Z)-**4a** were determined by 2D NOESY.

Diethyl (E)-3-ethylidene-4-methylenecyclopentane-1,1-dicarboxylate ((E)-4a): ^1H NMR (400 MHz, CDCl_3) δ 1.25 (t, $J = 7.3$ Hz, 6H), 1.72 (d, $J = 7.3$ Hz, 3H), 2.97 (s, 2H), 3.01 (t, $J = 2.1$ Hz, 2H), 4.19 (q, $J = 7.3$ Hz, 4H), 4.81 (s, 1H), 5.23 (s, 1H), 5.94 (qt, $J = 7.3, 2.8$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 14.0 (2C), 14.8, 37.3, 41.4, 57.6, 61.5 (2C), 102.4, 116.6, 137.0, 145.3, 171.4 (2C).

Compound **5a** could not be isolated in pure form. Partial ^1H NMR spectra was obtained from the

mixture of (Z)-**4a**, (E)-**4a**, and **5a**. ^1H NMR (400 MHz, CDCl_3) 5.05 (d, $J = 17.0$ Hz, 1H), 5.06 (d, $J = 11.0$ Hz, 1H), 5.56 (dd, $J = 17.0, 11.0$ Hz, 1H).

Diethyl (Z)-4-methylene-3-propylidenecyclopentane-1,1-dicarboxylate ((Z)-4b): ^1H NMR (400 MHz, CDCl_3) δ 1.03 (t, $J = 7.3$ Hz, 3H), 1.24 (t, $J = 7.3$ Hz, 6H), 2.27 (qd, $J = 7.3, 6.6$ Hz, 2H), 2.98 (s, 2H), 3.04 (s, 2H), 4.18 (q, $J = 7.3$ Hz, 4H), 5.13 (s, 1H), 5.18 (s, 1H), 5.49 (t, $J = 6.6$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 13.9 (2C), 14.1, 22.5, 42.4, 42.8, 57.5, 61.4 (2C), 110.3, 129.6, 134.4, 144.3, 171.3 (2C). Anal. Calcd for $\text{C}_{15}\text{H}_{22}\text{O}_4$: C, 67.64; H, 8.33. Found: C, 67.60; H, 8.43.

Compound (E)-**4b** could not be isolated in pure form. Partial ^1H NMR spectra was obtained from the mixture of (E)-**4b** and (Z)-**4b**. ^1H NMR (400 MHz, CDCl_3) 4.82 (s, 1H), 5.25 (s, 1H), 5.83-5.90 (m, 1H).

Stereochemistry of (Z)-**4b** were determined by 2D NOESY.

Diethyl (Z)-4-methylene-3-pentylidenecyclopentane-1,1-dicarboxylate ((Z)-4c): ^1H NMR (400 MHz, CDCl_3) δ 0.82 (t, $J = 7.3$ Hz, 3H), 1.16 (t, $J = 7.3$ Hz, 6H), 1.26 (sext, $J = 7.3$ Hz, 2H), 1.33 (quint, $J = 7.3$ Hz, 2H), 2.18 (td, $J = 7.3, 6.9$ Hz, 2H), 2.91 (s, 2H), 2.96 (s, 2H), 4.10 (q, $J = 7.3$ Hz, 4H), 5.05 (s, 1H), 5.12 (s, 1H), 5.43 (t, $J = 6.9$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 13.8, 13.9 (2C), 22.4, 28.9, 31.7, 42.5, 42.8, 57.5, 61.3 (2C), 110.2, 128.1, 134.7, 144.3, 171.3 (2C). Anal. Calcd for $\text{C}_{17}\text{H}_{26}\text{O}_4$: C, 69.36; H, 8.90. Found: C, 69.15; H, 8.86.

Compound (E)-**4c** and **5c** could not be isolated in pure form. Partial ^1H NMR spectra was obtained from the mixture of (E)-**4c**, (Z)-**4c** and **5c**. (Z)-**4c**: ^1H NMR (400 MHz, CDCl_3) δ 4.81 (d, $J = 1.4$ Hz, 1H), 5.24 (d, $J = 1.8$ Hz, 1H), 5.80-5.90 (m, 1H). **5c**: ^1H NMR (400 MHz, CDCl_3) δ 5.54 (qt, $J = 15.6, 6.9$ Hz, 1H), 6.23 (d, $J = 15.6$ Hz, 1H).

Stereochemistry of the products were determined by 2D NOESY.

Diethyl (Z)-3-benzylidene-4-methylenecyclopentane-1,1-dicarboxylate ((Z)-4d): ^1H NMR (400 MHz, CDCl_3) δ 1.26 (t, $J = 7.3$ Hz, 6H), 3.06 (t, $J = 2.3$ Hz, 2H), 3.15 (d, $J = 1.8$ Hz, 2H), 4.21 (q, $J = 7.3$ Hz, 4H), 4.91 (t, $J = 2.3$ Hz, 1H), 5.13 (t, $J = 2.3$ Hz, 1H), 6.49 (t, $J = 1.8$ Hz, 1H), 7.19 (t, $J = 7.3$ Hz, 1H), 7.28 (dd, $J = 7.8, 7.3$ Hz, 2H), 7.34 (d, $J = 7.8$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 14.0 (2C), 42.8, 43.0, 57.1, 61.5 (2C), 110.9, 124.9, 126.8, 128.2 (2C), 128.4 (2C), 137.0, 137.5, 142.5, 171.2 (2C).

Compound (E)-**4d** could not be isolated in pure form. Partial ^1H NMR spectra was obtained from the mixture of (E)-**4d** and (Z)-**4d**. ^1H NMR (400 MHz, CDCl_3) δ 4.99 (s, 1H), 5.49 (t, $J = 2.1$ Hz, 1H), 6.87 (t, $J = 2.5$ Hz, 1H).

Stereochemistry of (Z)-**4d** were determined by 2D NOESY.

Diethyl (E)-4-methylene-3-(1-trimethylsilylethylidene)cyclopentane-1,1-dicarboxylate ((E)-4e)

¹H NMR (400 MHz, CDCl₃) δ 0.11 (s, 9H), 1.23 (t, *J* = 7.3 Hz, 6H), 2.69 (d, *J* = 1.8 Hz, 2H), 2.85 (t, *J* = 1.4 Hz, 2H), 4.15 (q, *J* = 7.3 Hz, 2H), 4.17 (q, *J* = 7.3 Hz, 2H), 4.94 (m, 1H), 4.96 (m, 1H), 6.35 (t, *J* = 1.8 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ -2.6 (3C), 14.0 (2C), 32.5, 35.6, 54.1, 61.3 (2C), 113.8, 135.6, 139.6, 139.7, 171.1 (2C).

Stereochemistry of (E)-**4e** were determined by 2D NOESY.

Diethyl 5,6-diethyl-7-methyl-3-phenyl-3a,4-dihydroindan-2,2-dicarboxylate (15b)

¹H NMR (500 MHz, CDCl₃) δ 0.74 (t, *J* = 7.3 Hz, 3H), 0.84 (t, *J* = 7.3 Hz, 3H), 0.89 (t, *J* = 7.3 Hz, 3H), 1.14 (t, *J* = 7.3 Hz, 3H), 1.70 (d, *J* = 1.8 Hz, 3H), 1.65-1.96 (m, 3H), 2.00-2.25 (m, 3H), 2.70 (d, *J* = 8.3 Hz, 1H), 2.92-3.09 (m, 1H), 3.24 (d, *J* = 8.3 Hz, 1H), 3.53 (dq, *J* = 10.5, 7.3 Hz, 1H), 3.57 (d, *J* = 12.4 Hz, 1H), 3.84 (dq, *J* = 10.5, 7.3 Hz, 1H), 4.08 (dq, *J* = 10.5, 7.3 Hz, 1H), 4.15 (dq, *J* = 10.5, 7.3 Hz, 1H), 7.14 (t, *J* = 7.3 Hz, 1H), 7.19 (t, *J* = 7.3 Hz, 2H), 7.26 (d, *J* = 7.3 Hz, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 13.4, 13.6, 14.0, 14.8, 15.1, 20.7, 26.3, 33.0, 38.8, 42.7, 57.6, 60.8, 61.1, 64.2, 125.5, 126.9, 127.9 (2C), 128.8 (2C), 132.0, 132.7, 133.1, 138.7, 170.9, 171.9. Anal. Calcd for C₂₆H₃₄O₄: C, 76.06; H, 8.35. Found: C, 76.01; H, 8.53.

Stereochemistry of **15b** were determined by 2D NOESY.

References

- 1 Hicks, F. A.; Kablaoui, N. M.; Buchwald, S. L. *J. Am. Chem. Soc.* **1996**, *118*, 9450.
- 2 Schweizer, E. E.; Creasy, W. S.; Light, K. K.; Shaffer, F. T. *J. Org. Chem.* **1969**, *34*, 212.
- 3 Takeuchi, R.; Kashio, M. *Angew. Chem., Int. Ed. Engl.* **1997**, *36*, 263; Takeuchi, R.; Kashio, M. *J. Am. Chem. Soc.* **1998**, *120*, 8647.
- 4 Crabtree, R. H.; Quirk, J. M.; Felkin, H.; Fillebeen-Khan, T. *Synth. React. Inorg. Met.-Org. Chem.* **1982**, *12*, 407.
- 5 Chatani, N.; Inoue, H.; Morimoto, T.; Muto, T.; Murai, S. *J. Org. Chem.* **2001**, *66*, 4433.
- 6 Oh, C. H.; Lim, Y. M. *Tetrahedron Lett.* **2003**, *44*, 267.
- 7 Grigg, R.; Loganathan, V.; Sridharan, V.; Stevenson, P.; Sukirthalingam, S.; Worakun, T. *Tetrahedron* **1996**, *52*, 11479.

A 2D NMR spectrum, likely a COSY or NOESY experiment, showing correlations between protons. The horizontal and vertical axes are both labeled in PPM, with major ticks at 0.0, 2.5, and 5.0. The 1D ¹H NMR spectrum is projected along both axes. The 2D plot contains several cross-peaks, with three specific correlations highlighted by colored arrows: a red arrow points from a peak at approximately (4.5, 3.5) to (3.5, 4.5); a green arrow points from a peak at approximately (3.5, 3.5) to (3.5, 5.5); and a blue arrow points from a peak at approximately (5.5, 3.5) to (5.5, 5.5). Each cross-peak is accompanied by a small molecular structure diagram showing the spatial relationship between the correlated protons.

OBFRQ	500.00	MHz
OBSET	162.00	KHz
OBFIN	416.02	Hz
POINT	512	
FREQU	7507.51	Hz
CLPNT	512	
TODAT	256	
CLFRQ	7507.51	Hz
SCANS	16	
ACQTM	0.0682	sec
PD	1.5000	sec
PW1	12.50	usec
PW2	12.50	usec
PW3	12.50	usec
PI1	1.0000	msec
PI2	1.0000	msec
PI3	1.0000	msec
IRNUC	1H	
CTEMP	30.0	C
SLVNT	CDCL3	
EXREF	0.00	ppm
CLEXR	0.00	
RGAIN	15

(Z) -4b-noesy

DFILE C:\Documents and Settings\user\My Documents\NMR\13-FEB-2004 03:17:11
COMNT (Z) -4b-noesy
DATIM 13-FEB-2004 03:17:11
EXMOD noesy_phase_sens
OBNUC 1H
OBFRQ 500.00 MHz
OBSET 162.00 KHz
OBFIN 416.02 Hz
POINT 512
FREQU 7507.51 Hz
CLPNT 512
TODAT 256
CLFRQ 7507.51 Hz
SCANS 16
ACQTM 0.0682 sec
PD 1.5000 sec
PW1 12.50 usec
PW2 12.50 usec
PW3 12.50 usec
PI1 1.0000 msec
PI2 1.0000 msec
PI3 1.0000 msec
IRNUC 1H
CTEMP 23.1 C
SLVNT CDCL3
EXREF 0.00 ppm
CLEXR 0.00
RGAIN 15

DFILE C:\Documents and S€
 COMNT (Z) -4c-noesy
 DATIM 31-JAN-2004 04:32:4
 EXMOD noesy_phase_sens
 OBNUC 1H

OBFRO 500.00 MHz
 OBSET 162.00 KHz
 OBFIN 416.02 Hz
 POINT 512
 FREQU 7507.51 Hz
 CLPNT 512
 TODAT 256
 CLFRQ 7507.51 Hz
 SCANS 16
 ACQTM 0.0682 sec
 PD 1.5000 sec
 PW1 12.50 usec
 PW2 12.50 usec
 PW3 12.50 usec
 PI1 1.0000 msec
 PI2 1.0000 msec
 PI3 1.0000 msec
 IRNUC 1H
 CTEMP 23.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 CLEXR 0.00
 RGAIN 15

3b-C

DFILE C:\Documents and
 COMNT 3b-C
 DATIM 17-JAN-2004 07:47
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFRQ 125.00 MHz
 OBSET 777.00 KHz
 OBFIN 875.47 Hz
 POINT 65536
 FREQU 31446.54 Hz
 SCANS 256
 ACQTM 2.0840 sec
 PD 1.0000 sec
 PW1 3.17 usec
 IRNUC 1H
 CTEMP 26.3 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 29

3b-H

DFILE C:\Documents and
 COMNT 3b-H
 DATIM 17-JAN-2004 07:32
 OBNUC 1H
 EXMOD single_pulse.exp
 OBFRQ 500.00 MHz
 OBSET 162.00 KHz
 OBFIN 416.02 Hz
 POINT 16384
 FREQU 7507.51 Hz
 SCANS 16
 ACQTM 2.1823 sec
 PD 2.0000 sec
 PW1 6.25 usec
 IRNUC 1H
 CTEMP 24.2 C
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 11

3c-C

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

C:\Documents and
3c-C
4-DEC-2003 08:55
13C
single_pulse_dec
125.00 MHz
777.00 KHz
875.47 Hz
65536
31446.54 Hz
256
2.0840 sec
1.0000 sec
3.07 usec
1H
24.2 c
CDCL3
77.00 ppm
0.12 Hz
28

3c-H

DFILE C:\Documents and
 COMNT 3c-H
 DATIM 4-DEC-2003 08:41
 OBNUC 1H
 EXMOD single_pulse.exp
 OBFRQ 500.00 MHz
 OBSET 162.00 KHz
 OBFIN 416.02 Hz
 POINT 16384
 FREQU 7507.51 Hz
 SCANS 16
 ACQTM 2.1823 sec
 PD 2.0000 sec
 PW1 5.60 usec
 IRNUC 1H
 CTEMP 23.6 C
 SLVNT CDCl3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 13

3e-C

DFILE C:\Documents and
 COMNT 3e-C
 DATIM 29-JAN-2004 19:53
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFRO 125.00 MHz
 OBSET 777.00 KHz
 OBFIN 875.47 Hz
 POINT 65536
 FREQU 31446.54 Hz
 SCANS 256
 ACQTM 2.0840 sec
 PD 1.0000 sec
 PW1 3.17 usec
 IRNUC 1H
 CTEMP 25.2 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 29

3e-H

DFILE C:\Documents and
COMNT 3e-H
DATIM 29-JAN-2004 19:39
OBNUC 1H
EXMOD single_pulse.exp
OBFRO 500.00 MHz
OBSET 162.00 KHz
OBFIN 416.02 Hz
POINT 16384
FREQU 7507.51 Hz
SCANS 16
ACQTM 2.1823 sec
PD 2.0000 sec
PW1 6.25 usec
IRNUC 1H
CTEMP 23.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 13

DFILE C:\Documents and
 COMNT 3f-C
 DATIM 15-JAN-2004 12:15
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFRQ 125.00 MHz
 OBSET 777.00 KHz
 OBFIN 875.47 Hz
 POINT 65536
 FREQU 31446.54 Hz
 SCANS 256
 ACQTM 2.0840 sec
 PD 1.0000 sec
 PW1 3.17 usec
 IRNUC 1H
 CTEMP 26.2 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 30

DFILE C:\Documents and
COMNT 3f-H
DATIM 15-JAN-2004 12:00
OBNUC 1H
EXMOD single_pulse.exp
OBFRO 500.00 MHz
OBSET 162.00 KHz
OBFIN 416.02 Hz
POINT 16384
FREQU 7507.51 Hz
SCANS 16
ACQTM 2.1823 sec
PD 2.0000 sec
PW1 6.25 usec
IRNUC 1H
CTEMP 23.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 13

diastereo-Ph-noesy

DFILE C:\Documents and Se
 COMNT diastereo-Ph-noesy
 DATIM 20-OCT-2004 03:24:4
 EXMOD noesy_phase_sens
 OBNUC 1H
 OBFRQ 500.00 MHz
 OBSET 162.00 KHz
 OBFIN 416.02 Hz
 POINT 512
 FREQU 7507.51 Hz
 CLPNT 512
 TODAT 256
 CLFRQ 7507.51 Hz
 SCANS 16
 ACQTM 0.0682 sec
 PD 1.5000 sec
 PW1 12.50 usec
 PW2 12.50 usec
 PW3 12.50 usec
 PI1 1.0000 msec
 PI2 1.0000 msec
 PI3 1.0000 msec
 IRNUC 1H
 CTEMP 25.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 CLEXR 0.00
 RGAIN 15

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

C:\Documents and
diastereo-Ph-C
11-NOV-2004 08:07
13C
single_pulse_dec
125.00 MHz
777.00 KHz
875.47 Hz
65536
31446.54 Hz
12000
2.0840 sec
1.0000 sec
3.17 usec
1H
28.8 c
CDCL3
77.00 ppm
0.12 Hz
29

DFILE C:\Documents and
COMNT diastereo-Ph-H
DATIM 10-NOV-2004 21:49
OBNUC 1H
EXMOD single_pulse.exp
OBFRQ 500.00 MHz
OBSET 162.00 KHz
OBFIN 416.02 Hz
POINT 16384
FREQU 7507.51 Hz
SCANS 16
ACQTM 2.1823 sec
PD 2.0000 sec
PW1 6.25 usec
IRNUC 1H
CTEMP 24.5 c
SIVNT CDCL3
EXREF 7.24 ppm
BF 0.12 Hz
RGAIN 13