**Highly Regio- and Chemoselective Palladium-Catalyzed
Propargylallylation of Activated Olefins: A Novel Route to 1,7-Enyne
Derivatives**

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Supporting Information

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General Procedure for the Propargylallylation of Arylethylidene Malononitriles, Allylic Chlorides and Allenylstannanes.

A 25-mL round-bottom flask containing Pd(PPh₃)₄ (0.050 mmol) and arylethylidene malononitrile (1.00 mmol) was dissolved in 3 ml toluene and purged with nitrogen gas several times. To the flask were then added allyl chloride (1.20 mmol) and allenylstannane (1.20 mmol) via syringe. The reaction mixture was stirred at ambient temperature for 8 h. The reaction mixture was then treated with 10 mL of saturated KF solution and stirred for 30 min at ambient temperature. The solution was filtered through a Celite and silica gel pad and washed with 50 mL of ether. The filtrate was washed with water, dried (MgSO₄) and concentrated in vacuo. The residue was purified on a silica gel column (hexane/EtOAc = 9/1) to give the desired product. Products **6**, **8** and **10** were synthesized according to similar procedures by using substituted 2-(phenylmethylene)-1,3-indanedione (**5**) substituted 2-2-dimethyl-5-(phenylmethylene)-1,3-dioxane-4,6-dione (**7**) and conjugated activated olefins (**9**) as the amphiphilic reagents, respectively.

Products **4a-m**, **6a-m**, **8a-d**, **10a-h** and **11** were obtained according to this procedure. Product yields of these reactions are listed in Tables **2**, **3**, **4**, and **5**, while spectral data of these 1,7-enyne derivatives are shown below or listed in supporting information.

¹H NMR spectra of compounds **4a-d**, **4 f-i**, **4k**, **4m**, **10a-c** and **11** are available in the supporting information of *Org. Lett.* **2003**, 5, 881-884

2-(1-Phenyl-3-butylnyl)-2-((E)-3-phenyl-2-propenyl)propanedinitrile (4d): pale yellow oil; ¹H NMR (500 MHz, CDCl₃): δ 7.45 - 7.28 (m, 10H), 6.57 (d, *J* = 16.0 Hz, 1H),

6.17 (m, 1H), 3.31 (dd, $J = 10.5, 4.5$ Hz, 1H), 3.11 - 2.99 (m, 2H), 2.69 (dd, $J = 14.0, 7.5$ Hz, 1H), 2.60 (dd, $J = 14.0, 7.5$ Hz, 1H), 1.92 (t, $J = 3.0$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3): δ 138.0, 135.7, 134.6, 129.3, 129.2, 128.8, 128.7, 128.5, 126.7, 118.9, 114.9, 113.9, 79.3, 71.7, 50.0, 42.9, 40.1, 22.5; HRMS calcd for $\text{C}_{22}\text{H}_{18}\text{N}_2$ 310.1470, found 310.1460.

2-(2-Cyclohexenylmethyl)-2-(1-phenyl-3-butynyl)propanedinitrile (4e): pale yellow oil; ^1H NMR (500 MHz, CDCl_3): δ 7.46 - 7.38 (m, 5H), 6.08 - 6.06 (d, $J = 5.5$ Hz, 1H), 5.73 (d, $J = 11$ Hz, 1H), 3.50 - 3.37 (m, 1H), 3.04 - 2.98 (m, 2H), 2.45 - 2.43 (m, 2H), 2.09 - 2.02 (m, 4H), 1.91 - 1.87 (m, 2H), 1.76 - 1.42 (m, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 134.8, 134.7, 134.2, 129.1, 129.0, 128.8, 128.6, 123.1, 121.5, 114.5, 114.3, 114.2, 113.6, 79.5, 71.4, 48.1, 47.9, 47.2, 47.2, 40.2, 39.8, 26.3, 24.8, 24.7, 24.5, 23.2, 22.4, 20.9; HRMS calcd for $\text{C}_{19}\text{H}_{18}\text{N}_2$ 274.1470, found 274.1476. (1:1 diastereomers).

2-(1-(4-Chlorophenyl)-3-butynyl)-2-(2-methyl-2-propenyl)propanedinitrile (4f): pale yellow oil; ^1H NMR (500 MHz, CDCl_3): δ 7.40 (d, $J = 7.5$ Hz, 2H), 7.32 (d, $J = 7.5$ Hz, 2H), 5.13 (s, 1H), 5.02 (s, 1H), 3.27 (dd, $J = 11.0, 4.5$ Hz, 1H), 3.09 - 2.93 (m, 2H), 2.50 (d, $J = 14.5$ Hz, 1H), 2.31 (d, $J = 14.5$ Hz, 1H), 1.92 (t, $J = 2.5$ Hz, 1H), 1.89 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 136.9, 135.4, 133.1, 130.2, 129.3, 118.9, 114.8, 114.1, 78.9, 71.9, 50.7, 44.1, 41.7, 23.0, 22.1; HRMS calcd for $\text{C}_{17}\text{H}_{15}\text{ClN}_2$ 282.0924, found 282.0917.

2-(1-(4-(Methoxy)phenyl)-3-butynyl)-2-(2-methyl-2-propenyl)propanedinitrile (4g): pale yellow oil; ^1H NMR (500 MHz, CDCl_3): δ 7.30 (d, $J = 7.5$ Hz, 2H), 6.92 (d, $J = 7.5$ Hz, 2H), 5.11 (s, 1H), 5.01 (s, 1H), 3.81 (s, 3H), 3.24 (dd, $J = 11.5, 5.0$ Hz, 1H), 3.09 - 3.00 (m, 2H), 2.50 (d, $J = 14.5$ Hz, 1H), 2.31 (d, $J = 14.5$ Hz, 1H), 1.86 (t, $J = 3.0$

Hz, 1H), 1.85 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 160.1, 137.2, 129.9, 126.4, 118.6, 115.2, 114.4, 114.3, 79.5, 71.4, 55.3, 50.9, 44.2, 42.1, 23.1, 22.4; HRMS calcd for $\text{C}_{18}\text{H}_{18}\text{N}_2\text{O}$ 278.1419, found 278.1420.

2-(2-Methyl-2-propenyl)-2-(1-(4-nitrophenyl)-3-butynyl)propanedinitrile (4h):

pale yellow oil; ^1H NMR (500 MHz, CDCl_3): δ 8.29 (d, $J = 7.5$ Hz, 2H), 7.60 (d, $J = 7.5$ Hz, 2H), 5.16 (s, 1H), 5.05 (s, 1H), 3.42 (dd, $J = 11.5, 4.5$ Hz, H), 3.13 - 2.98 (m, 2H), 2.59 (d, $J = 14.0$ Hz, 1H), 2.35 (d, $J = 14.5$ Hz, 1H), 1.93 (t, $J = 3.0$ Hz, 1H), 1.91 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 148.5, 141.9, 136.5, 130.0, 124.2, 119.3, 114.3, 113.8, 78.3, 72.6, 50.8, 44.1, 41.5, 23.0, 21.9; HRMS calcd for $\text{C}_{17}\text{H}_{15}\text{N}_3\text{O}_2$ 293.1164, found 293.1153.

2-(2-Methyl-2-propenyl)-2-(1-(1-naphthyl)-3-butynyl)propanedinitrile (4i):

pale yellow oil; ^1H NMR (500 MHz, CDCl_3): δ 7.91 - 7.84 (m, 4H), 7.54 - 7.50 (m, 3H), 5.12 (s, 1H), 5.01 (s, 1H), 3.47 (dd, $J = 10.0, 5.0$ Hz, 1H), 3.18 - 3.14 (m, 2H), 2.54 (d, $J = 14.5$ Hz, 1H), 2.31 (d, $J = 14.5$ Hz, 1H), 1.87 (s, 3H), 1.86 (t, $J = 2.5$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3): δ 137.1, 133.5, 133.1, 131.9, 129.1, 128.9, 128.2, 127.8, 126.9, 126.8, 125.5, 118.7, 115.2, 114.3, 79.3, 71.7, 51.7, 44.3, 41.8, 23.1, 22.5; HRMS calcd for $\text{C}_{21}\text{H}_{18}\text{N}_2$ 298.1470, found 298.1473.

2-Allyl-2-[1-(2-furyl)-3-butynyl]malononitrile (4j):

pale yellow oil; ^1H NMR (500 MHz, CDCl_3): δ 7.45 (d, $J = 2.0$ Hz, 1H), 6.41 (d, $J = 3.5$ Hz, 1H), 6.38 (d, $J = 3.0$ Hz, 1H), 5.89 - 5.82 (m, 1H), 5.42 - 5.35 (m, 2H), 3.50 (dd, $J = 10.5, 4.5$ Hz, 1H), 3.89 - 2.79 (m, 2H), 2.58 - 2.47 (m, 2H), 1.99 (t, $J = 3.0$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3): δ 147.8, 143.5, 128.2, 123.7, 114.3, 113.4, 110.9, 110.7, 78.4, 71.7, 45.3, 43.3, 41.8, 40.1, 19.7; HRMS calcd for $\text{C}_{14}\text{H}_{12}\text{N}_2\text{O}$ 224.0950, found 224.0947.

2-(1-(2-Furanyl)-3-butynyl)-2-(2-methyl-2-propenyl)propanedinitrile (4k): pale yellow oil; ^1H NMR (500MHz, CDCl_3): δ 7.45 (d, $J = 2.0$ Hz, 1H), 6.50 (dd, $J = 5.5$, 3.5 Hz, 1H), 6.41 (d, $J = 3.0$ Hz, 1H), 5.13 (s, 1H), 5.04 (s, 1H), 3.50 (t, $J = 4.5$ Hz, 1H), 3.00 - 2.98 (m, 2H), 2.50 (d, $J = 14.5$ Hz, 1H), 2.36 (d, $J = 14.5$ Hz, 1H), 1.99 (t, $J = 3.0$ Hz, 1H), 1.90 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 147.9, 143.4, 136.9, 118.7, 114.6, 113.8, 110.8, 110.6, 78.7, 71.6, 45.3, 43.4, 41.1, 22.9, 21.0; HRMS calcd for $\text{C}_{15}\text{H}_{14}\text{N}_2\text{O}$ 238.1106, found 238.1107.

2-Allyl-2-[1-(2-thienyl)-3-butynyl]malononitrile (4l): pale yellow oil; ^1H NMR (500 MHz, CDCl_3): δ 7.36 (d, $J = 4.5$ Hz, 1H), 7.19 (d, $J = 3.5$ Hz, 1H), 7.06 (dd, $J = 5.0$, 3.5 Hz, 1H), 5.90 – 5.83 (m, 1H), 5.43 – 5.34 (m, 2H), 3.59 (dd, $J = 11.0$, 4.0 Hz, 1H), 3.10 - 2.92 (m, 2H), 2.60 – 2.54 (m, 2H), 1.99 (t, $J = 3.0$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3): δ 136.0, 127.9, 127.7, 127.1, 126.1, 123.5, 114.2, 113.4, 78.5, 71.8, 45.4, 42.7, 40.1, 24.0; HRMS calcd for $\text{C}_{14}\text{H}_{12}\text{N}_2\text{S}$ 240.0721, found 240.0727.

2-(2-Methyl-2-propenyl)-2-(1-(2-thiophenyl)-3-butynyl)propanedinitrile (4m): pale yellow oil; ^1H NMR (500 MHz, CDCl_3): δ 7.35 (d, $J = 4.5$ Hz, 1H), 7.19 (d, $J = 3.5$ Hz, 1H), 7.06 (dd, $J = 5.0$, 3.5 Hz, 1H), 5.14 (s, 1H), 5.05 (s, 1H), 3.60 (dd, $J = 11.0$, 4.0 Hz, 1H), 3.14 - 2.94 (m, 2H), 2.52 (d, $J = 14.0$ Hz, 1H), 2.42 (d, $J = 14.0$ Hz, 1H), 1.97 (t, $J = 3.0$ Hz, 1H), 1.90 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 137.0, 136.5, 128.1, 127.4, 126.4, 118.8, 114.9, 114.1, 78.8, 71.9, 47.3, 43.9, 42.2, 24.2, 23.1; HRMS calcd for $\text{C}_{15}\text{H}_{14}\text{N}_2\text{S}$ 254.0878, found 254.0886

2-(2-Cyclohexenyl)-2-(1-phenyl-3-butynyl)-1,3-indanedione (6d): pale yellow oil; ^1H NMR (600 MHz, CDCl_3): δ 7.90 – 7.57 (m, 4H), 7.03 – 6.95 (m, 4H), 6.09 – 5.89 (m,

1H), 5.55 – 4.97 (m, 1H), 3.77 (dd, $J = 12, 4.2$ Hz, 1H), 3.53 (dd, $J = 9, 6.6$ Hz, 1H), 3.19 – 2.87 (m, 2H), 2.01 – 1.71 (m, 9H); ^{13}C NMR (125 MHz, CDCl_3): δ 204.9, 204.5, 202.6, 202.3, 142.6, 142.5, 137.8, 135.3, 135.1, 130.9, 130.3, 129.3, 129.3, 127.9, 127.9, 127.1, 127.0, 125.5, 124.8, 122.5, 122.3, 122.1, 122.0, 82.3, 69.9, 63.2, 62.4, 46.5, 46.1, 39.5, 38.2, 26.3, 24.9, 23.3, 22.2, 19.2, 18.6, 14.1; HRMS calcd for $\text{C}_{25}\text{H}_{22}\text{O}_2$ 354.1620, found 354.1616. (1:1 diastereomers).

2-[1-(4-Chlorophenyl)-3-butynyl]-2-(2-methylallyl)-1,3-indanedione (6e):

viscous oil; ^1H NMR (500 MHz, CDCl_3): δ 7.80 (d, $J = 7.2$, 1H), 7.69 – 7.61 (m, 3H), 7.05 – 7.01 (m, 5H), 4.53 (s, 2H), 3.42 (dd, $J = 9.0, 7.5$ Hz, 1H), 2.83 – 2.81 (m, 2H), 2.75 (d, $J = 13.5$ Hz, 1H), 2.49 (d, $J = 13.5$ Hz, 1H), 1.72 (t, $J = 2.5$ Hz, 1H), 1.31 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 203.5, 202.5, 142.8, 142.5, 139.4, 136.7, 135.6, 135.6, 133.0, 130.6, 128.1, 122.7, 122.5, 116.9, 81.7, 70.5, 60.9, 49.8, 42.1, 23.9, 20.1; HRMS calcd for $\text{C}_{23}\text{H}_{19}\text{ClO}_2$ 362.1074, found 362.1070.

2-[1-(4-Bromophenyl)-3-butynyl]-2-(2-methylallyl)-1,3-indanedione (6f):

viscous oil; ^1H NMR (600 MHz, CDCl_3): δ 7.81 – 7.66 (m, 4H), 7.19 (d, $J = 8.4$ Hz, 2H), 6.97 (d, $J = 8.4$ Hz, 2H), 4.53 (s, 2H), 3.40 (dd, $J = 9.0, 6.6$ Hz, 1H), 2.82 – 2.80 (m, 2H), 2.74 (d, $J = 13.5$ Hz, 1H), 2.48 (d, $J = 13.0$ Hz, 1H), 1.71 (t, $J = 2.4$ Hz, 1H), 1.30 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 203.5, 202.4, 142.8, 142.5, 139.4, 137.2, 135.6, 135.6, 131.1, 127.8, 122.7, 121.3, 116.9, 81.7, 70.5, 60.8, 49.8, 42.1, 23.9, 20.2; HRMS calcd for $\text{C}_{23}\text{H}_{19}\text{BrO}_2$ 406.0568, found 406.0570.

2-[1-(4-Methoxyphenyl)-3-butynyl]-2-(2-methylallyl)-1,3-indanedione (6g):

viscous oil; ^1H NMR (500 MHz, CDCl_3): δ 7.79 (d, $J = 7.2$ Hz, 1H), 7.70 – 7.62 (m, 3H),

6.98 (d, $J = 8.4$ Hz, 2H), 6.59 (d, $J = 8.4$ Hz, 2H), 4.53 (s, 2H), 3.63 (s, 3H), 3.39 (dd, $J = 9.5, 7.0$ Hz, 1H), 2.85 – 2.82 (m, 2H), 2.79 (d, $J = 13.0$ Hz, 1H), 2.52 (d, $J = 13.0$ Hz, 1H), 1.72 (t, $J = 2.5$ Hz, 1H), 1.33 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 203.9, 202.8, 158.5, 142.9, 142.7, 139.8, 135.4, 135.3, 130.2, 122.6, 122.5, 116.7, 113.3, 82.3, 70.1, 61.3, 54.9, 49.8, 41.9, 24.0, 20.2; HRMS calcd for $\text{C}_{24}\text{H}_{22}\text{O}_3$ 358.1569, found 358.1571.

2-(2-Methylallyl)-2-[1-(4-nitrophenyl)-3-butynyl]-1,3-indanedione (6h): viscous oil; ^1H NMR (500 MHz, CDCl_3): δ 7.95 (d, $J = 8.5$ Hz, 2H), 7.82 (d, $J = 7.5$ Hz, 1H), 7.73 – 7.67 (m, 3H), 7.30 (d, $J = 9.0$ Hz, 2H), 4.54 (s, 2H), 3.57 (dd, $J = 9.5, 7.5$ Hz, 1H), 2.89 – 2.80 (m, 2H), 2.75 (d, $J = 13.0$ Hz, 1H), 2.47 (d, $J = 13.0$ Hz, 1H), 1.70 (t, $J = 2.5$ Hz, 1H), 1.33 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 203.0, 202.1, 147.0, 145.8, 142.6, 142.4, 139.0, 135.9, 135.9, 130.3, 123.1, 122.9, 122.7, 117.4, 81.0, 70.9, 60.6, 49.9, 42.2, 23.9, 19.9; HRMS calcd for $\text{C}_{23}\text{H}_{19}\text{NO}_4$ 373.1311, found 373.1311.

4-1-[2-(2-Methylallyl)-1,3-dioxo-2,3-dihydro-1H-2-indenyl]-3-butynylbenzonitrile (6i): viscous oil; ^1H NMR (600 MHz, CDCl_3): δ 7.81 – 7.67 (m, 4H), 7.36 (d, $J = 8.4$ Hz, 2H), 7.21 (d, $J = 8.4$ Hz, 2H), 4.53 – 4.52 (m, 2H), 3.49 (dd, $J = 10.6, 5.4$ Hz, 1H), 2.86 – 2.81 (m, 2H), 2.73 (d, $J = 12.6$ Hz, 1H), 2.45 (d, $J = 12.6$ Hz, 1H), 1.70 (t, $J = 2.4$ Hz, 1H), 1.29 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 202.9, 202.9, 143.7, 142.6, 142.3, 139.1, 135.9, 135.8, 131.7, 130.1, 122.8, 122.6, 118.4, 117.2, 111.1, 81.1, 70.8, 60.6, 50.2, 42.0, 23.9, 19.7; HRMS calcd for $\text{C}_{24}\text{H}_{19}\text{NO}_2$ 353.1416, found 353.1415.

2-(2-Methylallyl)-2-[1-(4-biphenyl)-3-butynyl]-1,3-indanedione (6j): viscous oil; ^1H NMR (500 MHz, CDCl_3): δ 7.82 (d, $J = 8.0$ Hz, 1H), 7.65 – 7.60 (m, 3H), 7.43 (d, $J =$

8.5 Hz, 2H), 7.36 – 7.25 (m, 5H), 7.15 (d, J = 8.5, 2H), 4.56 (d, J = 8.0 Hz, 2H), 3.50 (t, J = 8.0 Hz, 1H), 2.92 – 2.90 (dd, J = 7.5, 2.5 Hz 2H), 2.84 (d, J = 13.5 Hz, 1H), 2.57 (d, J = 13.0 Hz, 1H), 1.76 (t, J = 2.5 Hz, 1H), 1.36 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 203.7, 202.7, 142.9, 142.7, 140.4, 139.8, 139.7, 137.3, 135.4, 135.4, 129.6, 128.6, 127.2, 126.8, 126.5, 122.7, 122.4, 116.8, 82.2, 70.3, 61.2, 50.3, 41.9, 24.0, 20.1; HRMS calcd for $\text{C}_{29}\text{H}_{24}\text{O}_2$ 404.1776, found 404.1778.

2-(2-Methylallyl)-2-[1-(1-naphthyl)-3-butynyl]-1,3-indanedione (6k): viscous oil; ^1H NMR (500 MHz, CDCl_3): δ 8.33 (d, J = 9.0 Hz, 1H), 7.87 (d, J = 8.0 Hz, 1H), 7.73 – 7.45 (m, 8H), 7.21 (t, J = 6 Hz, 1H), 4.60 (dd, J = 9.5, 6.0 Hz, 1H), 4.50 (d, J = 7.5, Hz, 2H), 2.99 – 2.83 (m, 2H), 2.81 (d, J = 13.0 Hz, 1H), 2.53 (d, J = 13.5 Hz, 1H), 1.54 (t, J = 2.5 Hz, 1H), 1.30 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 204.2, 202.2, 143.2, 142.7, 139.6, 135.3, 135.3, 133.6, 132.7, 128.6, 127.8, 126.1, 125.9, 125.4, 124.9, 124.7, 123.8, 122.7, 122.4, 116.8, 82.0, 70.2, 61.2, 42.4, 34.0, 22.6, 14.2; HRMS calcd for $\text{C}_{27}\text{H}_{22}\text{O}_2$ 378.1620, found 378.1622.

2-(2-Methylallyl)-2-[1-(2-thienyl)-3-butynyl]-1,3-indanedione (6l): viscous oil; ^1H NMR (600 MHz, CDCl_3): δ 7.83 – 7.67 (m, 4H), 6.94 (dd, J = 4.2, 3.6 Hz, 1H), 6.78 – 6.77 (dd, J = 3.6, 1.2 Hz, 1H), 6.69 (dd, J = 5.4, 3.6 Hz, 1H), 4.54 – 4.52 (m, 1H), 3.75 (dd, J = 10.2, 5.4 Hz, 1H), 2.85 – 2.77 (m, 3H), 2.56 (d, J = 13.0 Hz, 1H), 1.78 (t, J = 2.4 Hz, 1H), 1.32 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 203.3, 202.4, 143.0, 142.6, 140.9, 139.5, 135.4, 127.7, 126.1, 124.5, 122.7, 122.5, 116.9, 81.6, 70.4, 61.1, 45.8, 41.9, 23.9, 21.9; HRMS calcd for $\text{C}_{21}\text{H}_{18}\text{O}_2\text{S}$ 334.1027, found 334.1029.

2-(1-Benzo[*b*]furan-2-yl-3-butynyl)-2-(2-methylallyl)-1,3-indanedione (6m):

viscous oil; ^1H NMR (500 MHz, CDCl_3): δ 7.85 (d, $J = 8.0$ Hz, 1H), 7.68 (d, $J = 7.5$ Hz, 1H), 7.62 – 7.52 (m, 2H), 7.33 – 7.31 (m, 1H), 7.11 – 7.01 (m, 3H), 6.47 (s, 1H), 4.58 (s, 2H), 3.66 – 3.63. (dd, $J = 11.0, 5.0$ Hz, 1H), 3.00 – 2.85 (m, 3H), 2.62 (d, $J = 13.0$ Hz, 1H), 1.81 (t, $J = 2.5$ Hz, 1H), 1.38 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 201.9, 201.7, 154.4, 154.2, 142.5, 141.9, 139.4, 135.3, 135.2, 127.6, 123.7, 122.6, 122.5, 120.7, 116.8, 110.6, 106.4, 81.2, 70.5, 59.9, 44.9, 40.9, 24.0, 18.5; HRMS calcd for $\text{C}_{25}\text{H}_{20}\text{O}_3$ 368.1412, found 368.1413.

2,2-Dimethyl-5-[1-(4-nitrophenyl)-3-butynyl]-5-[(E)-3-phenyl-2-propenyl]-1,3-dioxane-4,6-dione (8c): pale yellow oil; ^1H NMR (600 MHz, CDCl_3): δ 8.13 (d, $J = 8.4$ Hz, 2H), 7.34 (d, $J = 8.4$ Hz, 2H), 7.23 – 7.17 (m, 5H), 6.48 (d, $J = 15.6$ Hz, 1H), 5.95 – 5.92 (m, 1H), 3.78 – 3.76 (dd, $J = 9.4, 6.6$ Hz, 1H), 3.13 – 3.05 (m, 3H), 2.91 – 2.88 (m, 1H), 1.76 (t, $J = 2.4$ Hz, 1H), 1.47 (s, 3H), 0.78 (s, 3H); ^{13}C NMR (150 MHz, CDCl_3): δ 167.5, 166.9, 147.8, 144.9, 136.9, 135.8, 130.5, 128.7, 128.6, 126.3, 123.8, 120.9, 106.5, 80.6, 71.2, 59.4, 51.7, 39.9, 30.2, 28.4, 19.5; HRMS calcd for $\text{C}_{25}\text{H}_{23}\text{NO}_6$ 433.1525, found 233.1530.

5-[1-(4-Methoxyphenyl)-3-butynyl]-2,2-dimethyl-5-(2-methylallyl)-1,3-dioxane-4,6-dione (8d): pale yellow oil; ^1H NMR (600 MHz, CDCl_3): δ 7.04 (d, $J = 8.4$ Hz, 2H), 6.77 (d, $J = 8.4$ Hz, 2H), 4.89 (s, 1H), 4.82 (s, 1H), 3.70 (s, 3H), 3.57 (dd, $J = 9.6, 6.6$ Hz, 1H), 3.01 – 2.98 (m, 3H), 2.70 (d, $J = 12.6$, 1H), 1.74 (t, $J = 2.4$ Hz, 1H), 1.66 (s, 3H), 1.43 (s, 3H), 0.67 (s, 3H); ^{13}C NMR (150 MHz, CDCl_3): δ 167.9, 167.3, 159.3, 139.3, 130.3, 129.8, 118.3, 114.1, 106.1, 81.9, 69.9, 59.5, 55.2, 51.4, 44.2, 29.9, 28.3, 19.6; HRMS calcd for $\text{C}_{21}\text{H}_{24}\text{O}_5$ 356.1624, found 356.1626.

2-(2-Methyl-2-propenyl)-2-(1-((*E*)-2-phenyl-1-ethenyl)-3-butynyl)propanedinitrile (10b): pale yellow oil; ^1H NMR (500 MHz, CDCl_3): δ 7.42 (d, $J = 7.5$ Hz, 2H), 7.36 – 7.30 (m, 3H), 6.69 (d, $J = 15.5$ Hz, 1H), 6.04 (dd, $J = 15.5, 9.5$ Hz, 1H), 5.15 (s, 1H), 5.08 (s, 1H), 2.93 – 2.84 (m, 2H), 2.73 – 2.65 (m, 2H), 2.55 (d, $J = 14.5$ Hz, 1H), 2.07 (t, $J = 3.0$ Hz, 1H), 1.94 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 138.0, 137.1, 135.4, 128.8, 128.7, 126.8, 122.1, 118.8, 114.7, 114.1, 79.0, 72.2, 49.5, 43.9, 40.8, 23.1, 22.2; HRMS calcd for $\text{C}_{19}\text{H}_{18}\text{N}_2$ 274.1470, found 274.1477.

2-(2-Methyl-2-propenyl)-2-(1-((*E*)-2-phenyl-1-ethenyl)-3-butynyl)propanedinitrile (10c): pale yellow oil; ^1H NMR (500 MHz, CDCl_3): δ 7.38 – 7.26 (m, 10H), 6.68 (t, $J = 15.5$ Hz, 2H), 6.20 (m, 1H), 6.07 (dd, $J = 16.0, 9.5$ Hz, 1H), 2.97 – 2.70 (m, 5H), 2.07 (t, $J = 3.0$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3): δ 138.1, 138.0, 135.6, 135.4, 128.8, 128.7, 128.6, 127.4, 126.8, 126.7, 121.9, 118.7, 114.4, 113.8, 78.9, 72.3, 48.1, 41.7, 39.9, 22.3; HRMS calcd for $\text{C}_{24}\text{H}_{20}\text{N}_2$ 336.1626, found 336.1626

2-(2-Methylallyl)-2-1-[(*E*)-2-phenyl-1-ethenyl]-3-butynyl-1,3-indanedione (10e): pale yellow oil; ^1H NMR (600 MHz, CDCl_3): δ 7.89 – 7.84 (m, 2H), 7.74 – 7.72 (m, 2H), 7.25 – 7.12 (m, 5H), 6.42 (d, $J = 15.6$ Hz, 1H), 6.13 (dd, $J = 10.2, 5.4$ Hz, 1H), 4.49 (s, 2H), 2.91 – 2.87 (m, 2H), 2.63 (d, $J = 13.2$ Hz, 1H), 2.54 (d, $J = 13.2$ Hz, 1H), 2.28 – 2.16 (m, 2H), 1.76 (t, $J = 2.4$ Hz, 1H), 1.29 (s, 3H); ^{13}C NMR (150 MHz, CDCl_3): δ 203.6, 202.8, 143.1, 142.9, 139.8, 136.7, 135.8, 135.6, 134.3, 128.4, 127.5, 126.8, 126.5, 122.6, 122.7, 116.6, 81.3, 71.0, 60.5, 48.6, 42.6, 24.0, 20.9; HRMS calcd for $\text{C}_{24}\text{H}_{20}\text{O}_2$ 340.1463, found 340.1460.

2,2-Dimethyl-5-(2-methylallyl)-5-1-[(*E*)-2-phenyl-1-ethenyl]-3-butynyl-1,3-dioxane

ne-4,6-dione (10g): pale yellow oil; ^1H NMR (600 MHz, CDCl_3): δ 7.33 (d, $J = 7.8$ Hz, 2H), 7.25 (t, $J = 7.2$ Hz 1H), 7.18 (t, $J = 7.2$ Hz 1H), 6.47 (d, $J = 15.6$ Hz, 1H), 6.03 (dd, $J = 15.6, 9.6$ Hz, 2H), 5.63 – 5.58 (m, 1H), 5.18 – 5.11 (m, 2H), 3.08 – 3.06 (m, 1H), 2.71 (s, 2H), 2.60 – 2.56 (m, 1H), 2.45 – 2.42 (m, 1H), 1.91 (t, $J = 2.4$ Hz, 1H), 1.65 (s, 3H), 1.58 (s, 3H), 1.54 (s, 3H); ^{13}C NMR (150 MHz, CDCl_3): δ 168.1, 167.4, 139.6, 136.1, 135.5, 128.6, 127.9, 126.6, 125.6, 118.5, 106.3, 80.7, 71.2, 57.8, 51.5, 43.8, 30.4, 29.3, 23.9, 20.8; HRMS calcd for $\text{C}_{22}\text{H}_{24}\text{O}_4$ 352.1675, found 352.1670.

2,2-Dimethyl-5-1-[(E)-2-phenyl-1-ethenyl]-3-butynyl-5-[(E)-3-phenyl-2-propenyl]-1,3-dioxane-4,6-dione (10h): pale yellow oil; ^1H NMR (600 MHz, CDCl_3): δ 7.35 – 7.18 (m, 10H), 6.51 (d, $J = 15.6$ Hz, 1H), 6.44 (dd, $J = 15.6, 9.6$ Hz, 2H), 6.06 – 5.98 (m, 2H), 3.13 – 3.12 (m, 1H), 2.88 (d, $J = 7.8$ Hz, 2H), 2.64 – 2.60 (m, 1H), 2.47 – 2.44 (m, 1H), 1.93 (t, $J = 2.4$ Hz, 1H), 1.46 (s, 3H), 1.43 (s, 3H); ^{13}C NMR (150 MHz, CDCl_3): δ 168.6, 167.5, 136.1, 136.0, 135.5, 128.6, 127.3, 126.7, 126.3, 124.3, 121.3, 108.7, 80.1, 71.3, 56.9, 51.4, 41.8, 31.7, 29.8, 21.2; HRMS calcd for $\text{C}_{27}\text{H}_{26}\text{O}_4$ 414.1831, found 414.1836.

The regiochemistry of **4a,b** was established by ^1H - ^1H decoupling experiments. For example, on irradiating the H_a proton at δ 3.26 in **4a**, the multiplet at δ 3.03 (H_b and H_c protons) collapses partially. Irradiation of the two protons (H_b and H_c) multiplet at δ 3.03 causes the signal at δ 3.26 (doublet of a doublet) to a sharp singlet and also converts the triplet at δ 1.90 (H_d proton) to a sharp singlet. Irradiation of the H_d proton at δ 1.90 leads to partial collapse of the multiplet at δ 3.03 (H_b and H_c protons).

Similarly, irradiation of the H_a proton at δ 3.28 in **4b** led to partial collapse of the

multiplet at δ 3.05 (H_b and H_c protons). Irradiation of the two protons (H_b and H_c) multiplet at δ 3.05 converts the doublet of a doublet at δ 3.28 to a sharp singlet and also the triplet at δ 1.90 (H_d proton) to a sharp singlet. Irradiation of the H_d proton at δ 1.90 results in partial collapse of the multiplet at δ 3.05 (H_b and H_c protons). The above data strongly confirm the regiochemistry of **4a,b**.

Similarly, irradiation of the H_a proton at δ 3.43 in **6b** led to partial collapse of the multiplet at δ 2.89 (H_b and H_c protons). Irradiation of the two protons (H_b and H_c) multiplet at δ 2.89 converts the doublet of a doublet at δ 3.43 to a sharp singlet and also the triplet at δ 1.71 (H_d proton) to a sharp singlet. Irradiation of the H_d proton at δ 1.71 results in partial collapse of the multiplet at δ 3.43 (H_b and H_c protons).

Similarly, irradiating the H_a proton at δ 3.73 in **8a**, the multiplet at δ 3.05 (H_b and H_c protons) collapses partially. Irradiation of the two protons (H_b and H_c) multiplet at δ 3.05 causes the signal at δ 3.73 (doublet of a doublet) to a sharp singlet and also converts the triplet at δ 1.74 (H_d proton) to a sharp singlet. Irradiation of the H_d proton at δ 1.74 leads to partial collapse of the multiplet at δ 3.05 (H_b and H_c protons). The above data strongly confirm the regiochemistry of **6b** and **8a**.

6b

8a

The structures of 1,2-addition products **10a**, **b**, **e**, **g** were established by ^1H NMR NOE experiments. For example, selective irradiation of the H_a proton at δ 6.67 in **10a** caused 2.32% enhancement of the ortho protons of the phenyl ring at δ 7.30. Selective irradiation of the H_b proton at δ 6.00 caused 3.30% enhancement of the ortho protons at δ 7.30. Similar results were obtained for **10b**, **e**, **g** and were shown below. These NOE results strongly confirm the structures of **10a**, **b**, **e**, **g**.

10a

10b

10e

10g

Current Data Parameters
NAME CRJ-102
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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Time 15.35
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PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8389.262 Hz
FIDRES 0.256020 Hz
AQ 1.9530228 sec
RG 256
RW 59.600 usec
DE 5.50 usec
TE 300.1 K
D1 1.50000000 sec
WDECT 0.00000000 sec
MCPRK 0.01500000 sec

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PL1 -2.00 dB
SFO1 599.8023990 MHz

F2 - Processing parameters
SI 32768
SF 599.8000232 MHz
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LB 0.00 Hz
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PC 0.10

10 NMR plot parameters
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CY 9.00 cm
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F2P -0.500 ppm
F2 -299.90 Hz
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HZCH 299.90002 Hz/cm

Integration values:
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7.027
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7.012
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6.989
6.984
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IN              n
dp             y
vp             y
ns             nm
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VS             11.7
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SC             0
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pH

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Current Data Parameters
NAME CRJ-11
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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PULPROG zg
TD 32768
SOLVENT CDCl3
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FIDRES 0.256020 Hz
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CY 8.00 cm
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F1 5698.10 Hz
F2P -0.500 ppm
F2 -299.90 Hz
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HZCH 299.90002 Hz/cm

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Current Data Parameters
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EXPNO 1
PROCNO 1

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PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
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SWH 8389.262 Hz
FIDRES 0.256020 Hz
AQ 1.9530228 sec
RG 64
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DE 5.50 usec
TE 299.3 K
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F2 - Processing parameters
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LB 0.00 Hz
GB 0
PC 0.10

1D NMR plot parameters
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F1 5698.10 Hz
F2P -0.500 ppm
F2 -299.90 Hz
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HZCH 299.90002 Hz/cm

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Current Data Parameters
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EXPNO 1
PROCNO 1

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SOLVENT CDCl3
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FIDRES 0.236020 Hz
AQ 1.5530228 sec
RG 32
DQ 59.600 usec
DE 6.50 usec
TE 0.0 K
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MCREST 0.00000000 sec
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1.701
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Current Data Parameters
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EXPNO 1
PROCNO 1

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2.52

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Current Data Parameters
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PROCNO 1

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PULPROG zg
TD 32768
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SOLVENT CDCl3
NS 16
DS 0
SS 8399.262 Hz
SH1 0.256020 Hz
FIDRES 1.953028 sec
AQ 64
RG 59.600 usec
DE 5.50 usec
TE 300.0 K
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PL1 -2.00 dB
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F2 - Processing parameters
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SF 599.8000287 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 0.10

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
FIP 9.500 ppm
F1 5698.10 Hz
F2 -0.500 ppm
F2 -289.50 Hz
PPMCH 0.50000 ppm/cm
HZCH 299.50002 Hz/cm

Current Data Parameters
NAME CRJ-19
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030830
Time 9.55
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 9615.305 Hz
FIDRES 0.283439 Hz
AQ 1.703960 sec
RG 64
DM 52.000 usec
DE 6.50 usec
TE 299.1 K
D1 2.0000000 sec
d11 0.0000000 sec
MCREST 0.0150000 sec
MCIRK

***** CHANNEL f1 *****
NUC1 1H
P1 13.00 usec
PL1 -1.00 dB
SFO1 599.8029990 MHz

F2 - Processing parameters
SI 32768
SF 599.8000426 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D acq plot parameters
CX 60.00 ca
CY 10.00 ca
F1 9.500 MHz
F2 562.910 Hz
F3 -259.90 Hz
PRGCM 0.50000 pu/cu
KCM 299.80002 Hz/ca

Current Data Parameters
NAME CRJ-18
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030829
Time 18.50
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 9615.375 Hz
FIDRES 0.293438 Hz
AQ 1.7038077 sec
RG 64
DM 92.000 usec
DE 6.50 usec
TE 300.8 K
D1 2.00000000 sec
WDECT 0.00000000 sec
WDECK 0.01500000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 13.00 usec
PL1 -1.00 dB
SFO1 599.8024010 MHz

F2 - Processing parameters
SI 32768
SF 599.800544 MHz
WDW 70
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

10 MHz plot parameters
CX 20.00 cm
CY 10.00 cm
FIP 9.500 ppm
F1 5698.10 Hz
F2 -4.500 ppm
F2P -293.80 Hz
PPMCH 0.50000 ppm/cm
HZCM 299.90002 Hz/cm

Current Data Parameters
 Name: CHJ-201
 ExpNO: 1
 PROCNO: 1

F2 - Acquisition Parameters
 Date_: 20030910
 Time: 13.35
 INSTRUM: spect
 PULPROG: zgpg30
 SOLVENT: CDCl3
 NS: 16
 DS: 0
 SH: 0
 FIDRES: 0.256020 Hz
 AQ: 1.5030728 sec
 RG: 64
 DE: 59.500 usec
 TE: 300.2 K
 D1: 1.0000000 sec
 d11: 0.0300000 sec
 MCOREST: 0.0000000 sec
 MCORR: 0.0150000 sec

***** CHANNEL f1 *****
 NUC1: 1H
 P1: 6.00 usec
 PL1: -3.00 dB
 SFO1: 500.1360990 MHz

***** CHANNEL f2 *****
 CDPORG2: gird
 NUC2: 13C
 P2: 92.00 usec
 PL2: 120.00 dB
 PL12: 8.80 dB
 PL13: 10.00 dB
 SFO2: 500.1360990 MHz

F2 - Processing parameters
 SI: 32768
 SF: 500.1360990 MHz
 WDW: no
 SSB: 0
 LB: 0.00 Hz
 GB: 0
 PC: 0.50

1D NMR Plot parameters
 CX: 20.00 cm
 CY: 10.00 cm
 FIP: 9.300 ppm
 F1: 5659.10 Hz
 F2: -299.30 Hz
 PPMCH: 0.50000 ppm/cm
 HZCM: 299.50002 Hz/cm

Current Data Parameters
 NAME CRU-15
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030829
 Time 18.07
 INSTRUM spect
 PROBR40 5 mm QNP 1H/1
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SH 9615.375 Hz
 FIDRES 0.233438 Hz
 AQ 1.7038077 sec
 RG 64
 DW 52.000 usec
 DE 6.50 usec
 TE 300.7 K
 O1 2.00000000 sec
 MCOREST 0.00000000 sec
 MCORR 0.01500000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 13.00 usec
 PL1 -1.00 dB
 SF01 599.8024010 MHz

F2 - Processing parameters
 SI 32768
 SF 599.8000426 MHz
 KW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 10.00 cm
 F1P 9.500 ppm
 F1 5698.10 Hz
 F2P -0.500 ppm
 F2 -299.90 Hz
 PPKCH 0.50000 ppm/cm
 HZCH 299.90002 Hz/cm

Current Data Parameters
NAME CRJ-21
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030910
Time 15.04
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TO 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8389.262 Hz
FIDRES 0.256020 Hz
AQ 1.9330228 sec
RG 8
DW 59.600 usec
DE 6.50 usec
TE 300.7 K
D1 1.0000000 sec
d11 0.0300000 sec
HCES1 0.0000000 sec
HCES2 0.0150000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 6.00 usec
PL1 -3.00 dB
SFO1 500.13629950 MHz

***** CHANNEL f2 *****
CPOPRG2 gpgd
NUC2 13C
P2 92.00 usec
PL2 120.00 dB
PL12 8.80 dB
PL13 10.00 dB
SFO2 500.13629950 MHz

F2 - Processing parameters
SI 32768
SF 500.13629950 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 0.50

10 MHz plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 9.500 ppm
F1 5658.10 Hz
F2P -259.50 Hz
F2 -259.50 Hz
PUNCH 0.50000 ppm/cm
H2O 259.50002 Hz/cm

Current Data Parameters
NAME CRJ-15
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030829
Time 17.08
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 9615.375 Hz
FIDRES 0.293438 Hz
AQ 1.7039877 sec
RG 64
RW 52.000 usec
DE 6.50 usec
TE 300.5 K
O1 2.00000000 sec
MCREST 0.00000000 sec
MCNKK 0.01500000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 13.00 usec
PL1 -1.00 dB
SFO1 599.8024010 MHz

F2 - Processing parameters
SI 32768
SF 599.8000982 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
FIP 9.500 ppm
F1 5698.10 Hz
F2P -0.500 ppm
F2 -298.90 Hz
PPMCH 0.50000 ppm/cm
HZCH 299.90002 Hz/cm

Current Data Parameters
NAME CRJ-14
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030829
Time 16.43

INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg

TD 32768
SOLVENT CDCl3

NS 16
DS 0

SWH 9615.375 Hz
FIDRES 0.293438 Hz

AQ 1.7039877 sec
RG 64

OW 52.000 usec
TE 300.5 K

D1 2.00000000 sec
H1PROB 0.00000000 sec

MCPRK 0.01500000 sec
***** CHANNEL f1 *****

MFC 1H
P1 13.00 usec
PL1 -1.00 dB

ST01 599.8024010 MHz
F2 - Processing parameters

SI 32768
SF 599.8000596 MHz

WDW no
SSB 0

LB 0.00 Hz
GB 0

PC 1.00
10 NMR plot parameters

CX 20.00 cm
CY 10.00 cm

F1P 9.500 ppm
F1 5698.10 Hz

F2P -0.500 ppm
F2 -299.90 Hz

PPMCM 0.50000 ppm/cm
HZCM 299.90002 Hz/cm

ppm

109

Integral

Current Data Parameters
NAME CHU-17
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030831
Time 18.05
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 9615.375 Hz
FIDRES 0.293498 Hz
AQ 1.7039077 sec
RG 64
DW 52.000 usec
DE 6.50 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.00000000 sec
d12 0.01500000 sec
d13 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 13.00 usec
PL1 -1.00 dB
SF01 599.8024010 MHz

F2 - Processing parameters
SI 32768
SF 599.800623 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 9.500 ppm
F1 5698.10 Hz
F2P -0.500 ppm
F2 -299.90 Hz
PPMCH 0.50000 ppm/cm
HZCH 299.90002 Hz/cm