Selectivity in Nickel-Catalyzed Rearrangements of Cyclopropylen-yne

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SUPPORTING INFORMATION

General information. All reactions were conducted under an atmosphere of N₂ using standard Schlenk techniques or in a N₂ filled glove-box unless otherwise noted. Toluene was dried over neutral alumina under N₂ using a Grubbs-type solvent purification system. THF was freshly distilled from Na/benzophenone. Ni(COD)₂ was purchased from Strem and used without further purification. *N*-Heterocyclic carbene ligands (e.g., ICy, IrBu, IAd, IMes, IPr) were prepared as previously reported.¹ The cyclopropylen-yne **1a**, **1b**, **1e**, **8** and **10** were synthesized using literature procedures or under slightly modified procedures.² All other materials were purchased from commercial sources and used without further purification.

¹H and ¹³C NMR spectra were recorded on a GE-300 or a GE-500 NMR and referenced to residual protiated solvent (resonances downfield to the standard are reported as positive). All ¹³C NMR spectra were proton decoupled. Low-resolution mass spectra were obtained on a Hewlett Packard 5890 series II gas chromatograph interfaced with a Hewlett Packard 5989A mass spectrometer. IR spectra were recorded on a Bruker Tensor 27 FT-IR spectrometer. High-resolution mass spectra (HR-MS) (EI) were provided by the University of Utah Mass Spectrometry Facility.

[(1E)-3-(4-methyl-2-pentynyloxy)-1-propenyl]-cyclopropane (**1c**).

To a stirred suspension of NaH (0.4 g, 10 mmol) and dry THF (50 mL) in a 100 mL round-bottom flask was added 3-cyclopropyl-2-propen-1-ol (0.98 g, 10 mmol) dropwise under N₂ counter-flow. The resulting solution was stirred at room temperature for 0.5 h after which time 1-bromo-4-methyl-2-pentyne (1.61 g, 10 mmol) was added. The reaction mixture was then stirred at room temperature overnight. The solution was quenched with 20 mL sat. NH₄Cl solution. The aqueous layer was extracted 3 × 10 mL Et₂O. The collected organics were washed with brine, dried with anhydrous MgSO₄, and concentrated *in vacuo*. The resulting crude yellow oil was purified by flash column chromatography (10% Et₂O/pentanes) to yield **1c** (1.32 g, 74%) as a colorless oil. ¹H NMR (300 MHz, CDCl₃, ppm): δ 5.62 (dt, 6.6 Hz, 15.0 Hz, 1H), 5.25 (dd, 9.0 Hz, 15.0 Hz, 1H), 4.09 (d, 2.1 Hz, 2H), 3.95 (d, 6.6 Hz, 2H), 2.58 (m, 1H), 1.38 (m, 1H), 1.16 (d, 6.6 Hz, 6H), 0.71 (m, 2H), 0.37 (m, 2H); ¹³C {¹H} NMR (75 MHz, CDCl₃, ppm): δ 139.2, 123.1, 92.1, 75.1, 70.0, 57.3, 22.9, 20.5, 13.4, 6.7; IR (neat): 2872, 2935, 2851, 2249, 1668, 1461, 1357, 1320, 1184, 1080, 965, 812; HR-MS calcd. for C₁₂H₁₉O (M+H): 179.1436, found 179.1428.

[(1E)-3-(4,4-dimethyl-2-pentynyloxy)-1-propenyl]-cyclopropane (**1d**).

Prepared analogously to **1c** with 0.40 g NaH (10 mmol), 0.98 g 3-cyclopropyl-2-propen-1-ol (10 mmol), and 1.75 g 1-bromo-4,4-dimethyl-2-pentyne³ (10 mmol). Purified by column chromatography eluting with 10% Et₂O/pentanes to yield **1d** (1.48 g, 77%) as a colorless oil. ¹H NMR (300 MHz, CDCl₃, ppm): δ 5.64 (dt, 6.6 Hz, 15.3 Hz, 1H), 5.25 (dd, 9.0 Hz, 15.3 Hz, 1H), 4.10 (s, 2H), 3.96 (d, 6.6 Hz, 2H), 1.40 (m, 1H), 1.22 (s, 9H), 0.71 (m, 2H), 0.39 (m, 2H); ¹³C {¹H} NMR (75 MHz, CDCl₃, ppm): δ 139.3, 123.2, 95.1, 74.4, 69.9, 57.3, 30.9, 27.4, 13.4, 6.7; IR (neat): 2970, 2855, 2243, 1652, 1456, 1361, 1265, 1087, 811; HR-MS calcd. for C₁₃H₂₁O (M+H): 193.1592, found 193.1578.

[(1E)-3-(4-methoxy-2-pentynyloxy)-1-propenyl]-cyclopropane (12).

To a solution of [3-(2-propynyloxy)-1-propenyl]-cyclopropane (1.36 g, 10 mmol) and dry THF (50 mL) in a 100 mL round-bottom flask was added BuLi (4 mL, 2.5 M in hexane, 10 mmol) dropwise at -78 °C. The resulting solution was stirred at -78 °C for 0.5 h after which time bromomethoxy-methane (1.37 g, 11 mmol) was added. The reaction mixture was then stirred at room temperature for 4 h. The solution was quenched with 20 mL sat. NH₄Cl solution. The aqueous layer was extracted 3 × 10 mL Et₂O. The collected organics were washed with brine, dried with anhydrous MgSO₄, and concentrated *in vacuo*. The resulting crude yellow oil was purified by flash column chromatography (10% Et₂O/pentanes) to yield **12**. (1.18 g, 66%). ¹H NMR (300 MHz, CDCl₃, ppm): δ 5.62 (dt, 6.6 Hz, 15.3 Hz, 1H), 5.26 (dd, 8.7 Hz, 15.3 Hz, 1H), 4.17 (t, 1.8 Hz, 2H), 4.14 (t, 1.8 Hz, 2H), 3.98 (d, 6.6 Hz, 2H), 3.38 (s, 3H), 1.41 (m, 1H), 0.72 (m, 2H), 0.39 (m, 2H); ¹³C {¹H} NMR (75 MHz, CDCl₃, ppm): δ 139.9, 123.0, 82.8, 82.1, 70.4, 60.1, 57.8, 57.1, 13.7, 7.0; IR (neat): 3003, 2934, 2849, 1666, 1449, 1351, 1188, 1098, 966, 906; HR-MS calcd. for C₁₁H₁₇O₂ (M+H): 181.1229, found 181.1224.

4-cyclopropyl-3-buten-2-ol (20).

To a solution of cyclopropylacetylene (3.3 g, 50 mmol) and dry THF (100 mL) in a 250 mL round-bottom flask was added BuLi (20 mL, 2.5 M in hexane, 50 mmol) dropwise at -78 °C. The resulting solution was stirred at -78 °C for 0.5 h after which time acetaldehyde (2.2 g, 50 mol) was added. The reaction mixture was stirred at room temperature for 3 h. The solvent was removed *in vacuo* and dry ether (50 mL) was added. A suspension of LiAlH₄ (3.8 g, 10 mmol) in dry ether (50 mL) was added carefully at 0 °C. The resulting solution was stirred at room temperature for 4 h then quenched with 100 mL saturated Rochelle's salt solution. The aqueous layer was extracted 3 × 10 mL Et₂O. The collected organics were washed with saturated Rochelle's salt solution and brine, dried with anhydrous MgSO₄, and concentrated *in vacuo*. The resulting crude yellow oil was then distilled to yield **20** (3.5 g, 63%). ¹H NMR (300 MHz, CDCl₃, ppm): δ 5.60 (dd, 6.3 Hz, 15.3 Hz, 1H), 5.15 (dd, 9.0 Hz, 15.3 Hz, 1H), 4.24 (m, 1H), 1.46 (br, 1H), 1.41 (m, 1H), 1.25 (d, 6.3 Hz, 3H), 0.70 (m, 2H), 0.37 (m, 2H); ¹³C {¹H} NMR (75 MHz, CDCl₃, ppm): δ 135.1, 131.9, 69.1, 23.6, 13.5, 6.9; IR (neat): 3387, 1598, 1067; HR-MS calcd. for C₇H₁₃O (M+H): 113.0966, found 113.0959.

[(1E)-3-(2-butynyloxy)-1-propenyl]-cyclopropane (14).

Prepared analogously to **1c** with 0.40 g NaH (10 mmol), 1.12 g **20** (10 mmol), and 1.33 g 1-bromo-2-butyne (10 mmol). Purified by column chromatography eluting with 10% Et₂O/pentanes to yield **14** (1.47 g, 89%). ¹H NMR (300 MHz, CDCl₃, ppm): δ 5.36 (dd, 8.1 Hz, 15.3 Hz, 1H), 5.16 (dd, 8.7 Hz, 15.3 Hz, 1H), 4.10 (dq, 2.4 Hz, 15.0 Hz, 1H), 3.93 (m, 2H), 1.84 (t, 2.4 Hz, 3H), 1.39 (m, 1H), 1.24 (d, 6.3 Hz, 3H), 0.71 (m, 2H), 0.38 (m, 2H); ¹³C {¹H} NMR (75 MHz, CDCl₃, ppm): δ 137.8, 128.7, 81.8, 75.8, 75.4, 55.6, 21.8, 13.5, 7.1, 7.0, 3.9; IR (neat): 2973, 2841, 1439, 1383, 1100, 1028, 998, 906, 858; HR-MS calcd. for C₁₁H₁₇O (M+H): 165.1279, found 165.1270.

8-cyclopropyl-7-octen-2-yn-4-ol (21).

To a solution of BuLi (4 mL, 2.5 M in hexane, 10 mmol) and dry THF (50 mL) in a 100 mL round-bottom flask was added propyne (approximate 0.4 g, 10 mmol) at -78°C. The resulting solution was stirred at -78°C for 0.5 h after which time 5-cyclopropyl-4-pentalenol (1.24 g, 10 mol) was added. The reaction mixture was then stirred at room temperature for 3 h. The solution was quenched with 20 mL sat. NH₄Cl solution. The aqueous layer was extracted 3 × 10 mL Et₂O. The collected organics were washed with brine, dried with anhydrous MgSO₄, and concentrated *in vacuo*. The resulting crude yellow oil was purified by flash column chromatography (20% Et₂O/pentanes) to yield **21** (1.22 g, 74%). ¹H NMR (300 MHz, CDCl₃, ppm): δ 5.31 (dt, 7.5 Hz, 10.5 Hz, 1H), 4.78 (t, 9.0 Hz, 10.5 Hz, 1H),

4.38 (m, 1H), 2.34 (m, 2H), 1.85 (d, 2.1 Hz, 3H), 1.76 (m, 3H), 1.60 (m, 1H), 0.74 (m, 2H), 0.33 (m, 2H); ^{13}C { ^1H } NMR (75 MHz, CDCl_3 , ppm): δ 135.2, 126.9, 81.3, 80.5, 62.5, 38.2, 23.6, 9.8, 7.1, 3.8; IR (neat): 3357, 3081, 3005, 2922, 2859, 1650, 1423, 1153, 1064, 1019, 937, 810; HR-MS calcd. for $\text{C}_{11}\text{H}_{17}\text{O}$ (M+H): 165.1279, found 165.1283.

1-Cyclopropyl-5-methoxy-1-octen-6-yne (**16**).

To a stirred suspension of NaH (0.2 g, 5 mmol) and dry THF (25 mL) in a 100 mL round-bottom flask was added **21** (0.82 g, 5 mmol) dropwise under N_2 counter-flow. The resulting solution was stirred at room temperature for 0.5 h after which time methyl iodide (0.71 g, 5 mol) was added. The reaction mixture was then stirred at room temperature for 3 h. The solution was quenched with 20 mL sat. NH_4Cl solution. The aqueous layer was extracted 3×10 mL Et_2O . The collected organics were washed with brine, dried with anhydrous MgSO_4 , and concentrated *in vacuo*. The resulting crude yellow oil was purified by flash column chromatography (10% Et_2O /pentanes) to yield **16** (0.72 g, 81%) as a colorless oil. ^1H NMR (300 MHz, CDCl_3 , ppm): δ 5.28 (dt, 7.5 Hz, 10.5 Hz, 1H), 4.76 (t, 10.5 Hz, 1H), 3.93 (m, 1H), 3.39 (s, 3H), 2.30 (m, 2H), 1.87 (d, 2.1 Hz, 3H), 1.75 (m, 2H), 1.59 (m, 1H), 0.73 (m, 2H), 0.31 (m, 2H); ^{13}C { ^1H } NMR (75 MHz, CDCl_3 , ppm): δ 135.0, 126.9, 82.0, 78.2, 71.0, 56.5, 36.1, 23.6, 9.8, 7.0, 3.8; IR (neat): 3005, 2925, 1446, 1338, 1102, 937; HR-MS calcd. for $\text{C}_{12}\text{H}_{19}\text{O}$ (M+H): 179.1436, found 179.1436.

[3-(3-cyclopropyl-2-pentenyl)-1-propynyl]-cyclopropane (**18**).

Prepared analogously to **1c** with 0.40 g NaH (10 mmol), 0.98 g 3-cyclopropyl-2-propen-1-ol (10 mmol), and 1.6 g 3-bromo-1-propynyl-cyclopropane (10 mmol). Purified by column chromatography eluting with 10% Et_2O /pentanes to yield **1d** (1.53 g, 87%) as a colorless oil. ^1H NMR (300 MHz, CDCl_3 , ppm): δ 5.62 (dt, 6.6 Hz, 15.3 Hz, 1H), 5.25 (dd, 8.7 Hz, 15.3 Hz, 1H), 4.07 (d, 1.8 Hz, 2H), 3.95 (d, 6.6 Hz, 2H), 1.41 (m, 1H), 1.25 (m, 1H), 0.75 (m, 6H), 0.39 (m, 2H); ^{13}C { ^1H } NMR (75 MHz, CDCl_3 , ppm): δ 139.5, 123.3, 90.1, 71.5, 70.2, 57.5, 13.7, 8.4, 7.0; IR (neat): 3085, 3008, 2850, 2247, 1357, 1074, 966, 878, 812; HR-MS calcd. for $\text{C}_{12}\text{H}_{17}\text{O}$ (M+H): 177.1279, found 177.1271.

General Procedure for Ni-catalyzed Rearrangement. In a glove box, the cyclopropyl-ene-yne was added to an oven dried scintillation vial equipped with a magnetic stir bar and dissolved in toluene. To the stirring solution, a solution of $\text{Ni}(\text{COD})_2$ and NHC ligand (previously equilibrated for at least 6 h)⁴ was added. The reaction mixture was stirred at room temperature for 2 h (or until complete consumption of starting material was observed as judged by GC or TLC). The mixture was concentrated *in vacuo* and purified by silica gel column chromatography.

Rearrangement of [(1E)-3-(2-butynyloxy)-1-propenyl]-cyclopropane (1a**) catalyzed by $\text{Ni}(\text{COD})_2/\text{SIPr}$.** The general procedure was used with [(1E)-3-(2-butynyloxy)-1-propenyl]-cyclopropane (**1a**, 100 mg, 0.67 mmol), $\text{Ni}(\text{COD})_2$ (10 mg, 0.036 mmol), SIPr (14 mg, 0.036 mmol), and 0.5 mL of toluene. The reaction mixture was purified by column chromatography on silica

gel (10% Et_2O /pentanes) to afford 3-(1,3-butadienyl)-4-ethylidenetetrahydrofuran (**2a**, 57 mg, 57%) as a colorless oil. ^1H NMR (300 MHz, CDCl_3 , ppm): δ 6.64 (dt, 10.5 Hz, 16.8 Hz, 1H), 6.17 (t, 10.5 Hz, 1H), 5.21 (m, 4H), 4.37 (m, 2H), 4.10 (t, 7.8 Hz, 1H), 3.74 (m, 1H), 3.41 (t, 9.0 Hz, 1H), 1.58 (m, 3H); ^{13}C { ^1H } NMR (75 MHz, CDCl_3 , ppm): δ 142.1, 132.2, 131.5, 130.4, 118.7, 115.9, 73.8, 69.6, 43.2, 14.9; IR (neat): 2851, 1668, 1363, 1070, 919; HR-MS calcd. for $\text{C}_{10}\text{H}_{15}\text{O}$ (M+H): 151.1123, found 151.1140.

COSY summary; the following cross peaks were observed: H(1) and H(2); H(1) and H(4); H(5) and H(6); H(7) and H(8); H(8) and H(9); H(9) and H(10).

HMBC summary; the following cross peaks were observed: H(1) and C(2), C(3); H(2) and C(4), C(6); H(4) and C(2), C(3), C(5), C(6); H(5) and C(3), C(4), C(6), C(7); H(6) and C(3); H(7) and C(3), C(5), C(6), C(8), C(9); H(8) and C(6), C(9), C(10); H(9) and C(7), C(8); H(10) and C(8).

Rearrangement of [(1E)-3-(2-pentynyloxy)-1-propenyl]-cyclopropane (1b**) catalyzed by $\text{Ni(COD)}_2/\text{SIPr}$.** The general procedure was used with [(1E)-3-(2-pentynyloxy)-1-propenyl]-cyclopropane^{2a} (**1b**, 100 mg, 0.6 mmol), Ni(COD)_2 (8 mg, 0.03 mmol), SIPr (12 mg, 0.03 mmol), and

0.5 mL of toluene. The reaction mixture was purified by column chromatography on silica gel (10% Et_2O /pentanes) to afford 3-(1,3-butadienyl)-tetrahydro-4-propylidenefuran (**2b**, 34 mg, 34%) and 8-ethyl-3,3a,6,7-tetrahydro-1H-cyclohepta[c]furan^{2a} (**3b**, 27 mg, 27%).

2b: ^1H NMR (300 MHz, CDCl_3 , ppm): δ 6.65 (dt, 10.8 Hz, 16.5 Hz, 1H), 6.17 (t, 10.8 Hz, 1H), 5.18 (m, 4H), 4.43 (d, 10.2 Hz, 1H), 4.32 (d, 10.2 Hz, 1H), 4.09 (t, 8.4 Hz, 1H), 3.75 (m, 1H), 3.40 (t, 8.4 Hz, 1H), 1.93 (m, 2H), 0.96 (t, 7.5 Hz, 3H); ^{13}C $\{^1\text{H}\}$ NMR (75 MHz, CDCl_3 , ppm): δ 140.6, 132.2, 131.5, 130.4, 123.4, 118.7, 73.6, 69.4, 43.1, 23.1, 14.1; IR (neat): 2965, 2845, 1642, 1454, 1357, 1075, 1000, 918; HR-MS calcd. for $\text{C}_{11}\text{H}_{17}\text{O}$ (M+H): 165.1279, found 165.1280.

HMBC summary; the following cross peaks were observed: H(1) and C(2), C(3); H(2) and C(1), C(3), C(4); H(3) and C(5), C(7); H(5) and C(3), C(4), C(6), C(7); H(6) and C(4), C(5), C(7), C(8); H(7) and C(4), C(6), C(8); H(8) and C(4), C(6), C(7), C(10); H(9) and C(7), C(10), C(11); H(10) and C(8), C(9); H(11) and C(9).

3b^{2a}: ^1H NMR (300 MHz, CDCl_3 , ppm): δ 5.70 (m, 1H), 5.38 (m, 1H), 4.41 (d, 12.9 Hz, 1H), 4.30 (d, 12.9 Hz, 1H), 4.21 (t, 8.4 Hz), 3.70 (m, 1H), 3.45 (t, 8.4 Hz, 1H), 2.52 (m, 1H), 2.32 (m, 1H), 2.17 (m, 1H), 2.07 (m, 1H), 2.01 (q, 7.5 Hz, 2H), 0.99 (t, 7.5 Hz, 3H); ^{13}C $\{^1\text{H}\}$ NMR (75 MHz, CDCl_3 , ppm): δ 135.9, 132.9, 131.0, 128.2, 75.3, 70.8, 41.4, 29.8, 28.7, 27.0, 12.4; IR (neat): 2958, 2840, 1455, 1064, 938, 678; HR-MS calcd. for $\text{C}_{11}\text{H}_{17}\text{O}$ (M+H): 165.1279, found 165.1285.

COSY summary; the following cross peaks were observed: H(1) and H(2); H(2) and H(5); H(6) and H(7); H(8) and H(9); H(9) and H(10); H(10) and H(11); H(11) and H(5).

HMBC summary; the following cross peaks were observed: H(1) and C(2), C(3); H(2) and C(1), C(2), C(4), C(11); H(5) and C(3), C(4), C(6), C(7); H(6) and C(4), C(5), C(7), C(8); H(8) and C(4), C(6), C(7), C(10); H(9) and C(7); H(10) and C(3), C(8), C(9), C(11); H(11) and C(2), C(3), C(4), C(9), C(10).

Rearrangement of [(1E)-3-(2-pentynyloxy)-1-propenyl]-cyclopropane (1b**) catalyzed by $\text{Ni(COD)}_2/\text{IrBu}$.** The general procedure was used with [(1E)-3-(2-pentynyloxy)-1-propenyl]-cyclopropane (**1b**, 100 mg, 0.6 mmol), Ni(COD)_2 (8 mg, 0.03 mmol), IrBu (5.4 mg, 0.03 mmol), and 0.5 mL of toluene. The reaction mixture was purified by column chromatography on

silica gel (10% Et_2O /pentanes) to afford 3-(1,3-butadienyl)-tetrahydro-4-propylidenefuran (**2b**, 52 mg, 52%) as a colorless oil. ^1H NMR (300 MHz, CDCl_3 , ppm): δ 6.65 (dt, 10.8 Hz, 16.5 Hz, 1H), 6.17 (t, 10.8 Hz, 1H), 5.18 (m, 4H), 4.43 (d, 10.2 Hz, 1H), 4.32 (d, 10.2 Hz, 1H), 4.09 (t, 8.4 Hz, 1H), 3.75 (m, 1H), 3.40 (t, 8.4 Hz, 1H), 1.93 (m, 2H), 0.96 (t, 7.5 Hz, 3H); ^{13}C $\{^1\text{H}\}$ NMR (75 MHz, CDCl_3 , ppm): δ 140.6, 132.2, 131.5, 130.4, 123.4, 118.7, 73.6, 69.4, 43.1, 23.1, 14.1; IR (neat): 2965, 2845, 1642, 1454, 1357, 1075, 1000, 918; HR-MS calcd. for $\text{C}_{11}\text{H}_{17}\text{O}$ (M+H): 165.1279, found 165.1280.

HMBC summary; the following cross peaks were observed: H(1) and C(2), C(3); H(2) and C(1), C(3), C(4); H(3) and C(5), C(7); H(5) and C(3), C(4), C(6), C(7); H(6) and C(4), C(5), C(7),

C(8); H(7) and C(4), C(6), C(8); H(8) and C(4), C(6), C(7), C(10); H(9) and C(7), C(10), C(11); H(10) and C(8), C(9); H(11) and C(9).

Rearrangement of [(1E)-3-(4-methyl-2-pentynyloxy)-1-propenyl]-cyclopropane (1c) catalyzed by Ni(COD)₂/SIPr. The general procedure was used with [(1E)-3-(4-methyl-2-pentynyloxy)-1-propenyl]-cyclopropane (**1c**, 100 mg, 0.56 mmol), Ni(COD)₂ (8 mg, 0.03 mmol),

SIPr (12 mg, 0.03 mmol), and 0.5 mL of toluene. The reaction mixture was purified by column chromatography on silica gel (10% Et₂O/pentanes) to afford 3-(1,3-butadienyl)-tetrahydro-4-(2-methylpropylidene)furan (**2c**, 28 mg, 28%) and 8-isopropyl-3,3a,6,7-tetrahydro-1H-cyclohepta[c]furan (**3c**, 38 mg, 38%).

2c: ¹H NMR (300 MHz, CDCl₃, ppm): δ 6.63 (dt, 10.5 Hz, 16.8 Hz, 1H), 6.16 (t, 10.5 Hz, 1H), 5.24 (m, 3H), 4.99 (m, 1H), 4.44 (d, 13.2 Hz, 1H), 4.32 (dt, 13.2 Hz, 2.1 Hz, 1H), 4.08 (t, 8.4 Hz, 1H), 3.74 (m, 1H), 3.38 (t, 8.4 Hz, 1H), 2.21 (m, 1H), 0.94 (d, 6.6 Hz, 6H); ¹³C {¹H} NMR (75 MHz, CDCl₃, ppm): δ 139.0, 132.3, 131.5, 130.5, 129.1, 118.6, 73.5, 69.2, 43.0, 29.6, 23.0; IR (neat): 2958, 2849, 1463, 1359, 1067, 919; HR-MS calcd. for C₁₂H₁₉O (M+H): 179.1436, found 179.1421.

COSY summary; the following cross peaks were observed: H(1) and H(2); H(2) and H(3); H(3) and H(5); H(6) and H(7); H(7) and H(8); H(8) and H(9); H(9) and H(10); H(10) and H(11).

HMBC summary; the following cross peaks were observed: H(1) and C(2), C(3); H(2) and C(1), C(3), C(4); H(3) and C(5), C(7); H(5) and C(3), C(4), C(6), C(7); H(6) and C(4), C(5), C(7), C(8); H(7) and C(3), C(4), C(6), C(8); H(8) and C(4), C(6), C(7), C(10); H(9) and C(7), C(10), C(11); H(10) and C(8), C(9); H(11) and C(9).

3c: ¹H NMR (300 MHz, CDCl₃, ppm): δ 5.64 (m, 1H), 5.33 (m, 1H), 4.43 (d, 12.6 Hz, 1H), 4.20 (t, 8.4 Hz), 3.70 (m, 1H), 3.44 (t, 8.4 Hz), 2.35 (m, 3H), 2.10 (m, 2H), 0.99 (d, 6.6 Hz, 3H), 0.94 (d, 6.6 Hz); ¹³C {¹H} NMR (75 MHz, CDCl₃, ppm): δ 136.6, 136.2, 130.9, 127.5, 75.4, 70.5, 41.6, 33.1, 28.0, 24.9, 20.5, 20.4; IR (neat): 2957, 2839, 1462, 1362, 1073, 937; HR-MS calcd. for C₁₂H₁₉O (M+H): 179.1436, found 179.1407.

COSY summary; the following cross peaks were observed: H(1) and H(2); H(6) and H(7); H(8) and H(9); H(9) and H(10); H(10) and H(11); H(11) and H(5).

HMBC summary; the following cross peaks were observed: H(1) and C(2), C(3); H(2) and C(1), C(2), C(4), C(11); H(5) and C(3), C(4), C(6), C(7); H(6) and C(4), C(5), C(7), C(8); H(8) and C(4), C(6), C(7), C(10); H(9) and C(7); H(10) and C(3), C(8), C(9), C(11); H(11) and C(2), C(3), C(4), C(9), C(10).

Rearrangement of [(1E)-3-(4-methyl-2-pentynyloxy)-1-propenyl]-cyclopropane (1c) catalyzed by Ni(COD)₂/IrBu.

The general procedure was used with [(1E)-3-(4-methyl-2-pentynyloxy)-1-propenyl]-cyclopropane (**1c**, 100 mg, 0.56 mmol), Ni(COD)₂ (8 mg, 0.03 mmol), IrBu (5.4 mg, 0.03 mmol), and 0.5 mL of toluene. The reaction mixture was purified by column chromatography on silica gel (10% Et₂O/pentanes) to afford 3-(1,3-butadienyl)-tetrahydro-4-(2-methylpropylidene)furan (**2c**, 65 mg, 65%) as a colorless oil. ¹H NMR (300 MHz, CDCl₃, ppm): δ 6.63 (dt, 10.5 Hz, 16.8 Hz, 1H), 6.16 (t, 10.5 Hz, 1H), 5.24 (m, 3H), 4.99 (m, 1H), 4.44 (d, 13.2 Hz, 1H), 4.32 (dt, 13.2 Hz, 2.1 Hz, 1H), 4.08 (t, 8.4 Hz, 1H), 3.74 (m, 1H), 3.38 (t, 8.4 Hz, 1H), 2.21 (m, 1H), 0.94 (d, 6.6 Hz, 6H); ¹³C {¹H} NMR (75 MHz, CDCl₃, ppm): δ 139.0, 132.3, 131.5, 130.5, 129.1, 118.6, 73.5, 69.2, 43.0, 29.6, 23.0; IR (neat): 2958, 2849, 1463, 1359, 1067, 919; HR-MS calcd. for C₁₂H₁₉O (M+H): 179.1436, found 179.1421.

COSY summary; the following cross peaks were observed: H(1) and H(2); H(2) and H(3); H(3) and H(5); H(6) and H(7); H(7) and H(8); H(8) and H(9); H(9) and H(10); H(10) and H(11).

HMBC summary; the following cross peaks were observed: H(1) and C(2), C(3); H(2) and C(1), C(3), C(4); H(3) and C(5), C(7); H(5) and C(3), C(4), C(6), C(7); H(6) and C(4), C(5), C(7), C(8); H(7) and C(3), C(4), C(6), C(8); H(8) and C(4), C(6), C(7), C(10); H(9) and C(7), C(10), C(11); H(10) and C(8), C(9); H(11) and C(9).

Rearrangement of [(1E)-3-(4,4-dimethyl-2-pentynyloxy)-1-propenyl]-cyclopropane (1d) catalyzed by Ni(COD)₂/SIPr.

The general procedure was used with [(1E)-3-(4,4-dimethyl-2-pentynyloxy)-1-propenyl]-cyclopropane (**1d**, 100 mg, 0.5 mmol), Ni(COD)₂ (8 mg, 0.03 mmol), SIPr (11 mg, 0.03 mmol), and 0.5 mL of toluene. The reaction mixture was purified by column chromatography on silica gel (10% Et₂O/pentanes) to afford 8-(1',1'-dimethyl-ethyl)-1,3,6,7-tetrahydro-2H-cyclohepta[c]furan (**4d**, 82 mg, 82%) as a colorless oil. ¹H NMR (300 MHz, CDCl₃, ppm): δ 5.82 (m, 1H), 5.69 (d, 11.4 Hz, 1H), 4.96 (m, 1H), 4.80 (m, 2H), 4.60 (m, 1H), 2.51 (m, 2H), 2.19 (m, 1H), 1.62 (m, 1H), 0.95 (s, 9H); ¹³C {¹H} NMR (75 MHz, CDCl₃, ppm): δ 138.6, 134.5, 130.2, 119.6, 81.9, 79.1, 47.4, 35.7, 30.0, 29.4, 27.6; IR (neat): 2964, 2852, 1645, 1432, 1048, 922; HR-MS calcd. for C₁₃H₂₁O (M+H): 193.1592, found 193.1596.

COSY summary; the following cross peaks were observed: H(3) and H(11); H(8) and H(9); H(9) and H(10); H(10) and H(11).

HMBC summary; the following cross peaks were observed: H(1) and C(2), C(3); H(3) and C(1), C(2), C(4), C(5), C(7), C(10); H(5) and C(4), C(6), C(7); H(6) and C(4), C(5), C(7); H(8) and C(4), C(6), C(10); H(9) and C(7), C(10); H(10) and C(3), C(8), C(9); H(11) and C(2), C(3), C(4), C(9), C(10).

Rearrangement of [(1E)-3-(4,4-dimethyl-2-pentynyloxy)-1-propenyl]-cyclopropane (1d) catalyzed by Ni(COD)₂/ItBu.

The general procedure was used with [(1E)-3-(4,4-dimethyl-2-pentynyloxy)-1-propenyl]-cyclopropane (**1d**, 100 mg, 0.5 mmol), Ni(COD)₂ (8 mg, 0.03 mmol), ItBu (5.4 mg, 0.03 mmol), and 0.5 mL of toluene. The reaction mixture was purified by column chromatography on silica gel (10% Et₂O/pentanes) to afford 3-(1,3-butadienyl)-tetrahydro-4-(2,2-dimethylpropylidene)furan (**2d**, 79 mg, 79%) as a colorless oil. ¹H NMR (300 MHz, CDCl₃, ppm): δ 6.63 (dt, 10.5 Hz, 16.8 Hz, 1H), 6.17 (t, 16.8 Hz, 1H), 5.18 (m, 4H), 4.57 (m, 1H), 4.42 (dt, 2.4 Hz, 13.2 Hz, 1H), 4.02 (t, 8.4 Hz, 1H), 3.73 (m, 1H), 3.30 (t, 8.4 Hz, 1H), 1.03 (s, 9H); ¹³C {¹H} NMR (75 MHz, CDCl₃, ppm): δ 138.0, 132.7, 132.2, 131.6, 130.9, 118.7, 72.4, 68.9, 44.6, 33.5, 30.4; IR (neat): 2960, 2858, 1596, 1360, 1078, 935; HR-MS calcd. for C₁₃H₂₁O (M+H): 193.1592, found 193.1580.

COSY summary; the following cross peaks were observed: H(6) and H(7); H(7) and H(8); H(8) and H(9); H(9) and H(10); H(10) and H(11).

HMBC summary; the following cross peaks were observed: H(1) and C(2), C(3); H(3) and C(5), C(7); H(5) and C(3), C(4), C(6); H(6) and C(4), C(5), C(7), C(8); H(7) and C(4), C(6), C(8); H(8) and C(4), C(6), C(10); H(9) and C(10), C(11); H(10) and C(8), C(9); H(11) and C(9).

Rearrangement of [3-[[[(2E)-3-cyclopropyl-2-propenyl]oxy]-1-propynyl]trimethylsilane (1e) catalyzed by Ni(COD)₂/SIPr.

The general procedure was used with [3-[[[(2E)-3-cyclopropyl-2-propenyl]oxy]-1-propynyl]trimethylsilane (**1e**, 100 mg, 0.5 mmol), Ni(COD)₂ (8 mg, 0.03 mmol), SIPr (11 mg, 0.03 mmol), and 0.5 mL of toluene. The reaction mixture was purified by column chromatography on silica gel (10% Et₂O/pentanes) to afford **4e** (88 mg, 88%) as a colorless oil. ¹H NMR (300 MHz, CDCl₃,

ppm): δ 5.74 (m, 1H), 5.68 (m, 1H), 4.70 (m, 3H), 4.55 (m, 1H), 2.47 (m, 2H), 2.04 (m, 1H), 1.89 (m, 2H), 0.04 (s, 9H); ^{13}C { ^1H } NMR (75 MHz, CDCl_3 , ppm): δ 138.3, 132.8, 126.0, 119.9, 80.5, 79.3, 31.4, 30.2, 26.1, -1.4; IR (neat): 3014, 2840, 1756, 1655, 1427, 1357, 1252, 1062, 930, 844; HR-MS calcd. for $\text{C}_{12}\text{H}_{21}\text{OSi}$ (M+H): 209.1362, found 209.1340.

COSY summary; the following cross peaks were observed: H(2) and H(10); H(7) and H(8); H(8) and H(9); H(9) and H(10).

HMBC summary; the following cross peaks were observed: H(2) and C(3), C(6), C(9), C(10); H(4) and C(3), C(5), C(6); H(5) and C(3), C(4), C(6), C(7); H(7) and C(3), C(5), C(9); H(8) and C(6), C(9), C(10); H(9) and C(7), C(8), C(10); H(10) and C(2), C(3), C(8), C(9).

Rearrangement of [(2E)-3-cyclopropyl-2-propenyl](2-butynyl)-malonic acid dimethyl ester (8) catalyzed by $\text{Ni}(\text{COD})_2/\text{SIPr}$. The general procedure was used with [(2E)-3-cyclopropyl-2-propenyl](2-butynyl)-malonic acid dimethyl ester (**8**, 100 mg, 0.38 mmol), $\text{Ni}(\text{COD})_2$ (5 mg, 0.018 mmol), SIPr (7 mg, 0.018 mmol), and 0.5 mL of toluene. The reaction mixture was purified by column chromatography on silica gel

(10% Et_2O /pentanes) to afford 3-(1,3-butadienyl)-4-ethylidene-1,1-cyclopentenedicarboxylic acid dimethyl ester (**9**, 82 mg, 82%) as a colorless oil. ^1H NMR (300 MHz, CDCl_3 , ppm): δ 6.59 (dt, 10.8 Hz, 16.8 Hz, 1H), 6.11 (t, 10.8 Hz, 1H), 5.18 (m, 4H), 3.76 (s, 3H), 3.74 (s, 3H), 3.59 (m, 1H), 3.04 (m, 1H), 2.86 (m, 1H), 2.56 (ddd, 1.2 Hz, 7.2 Hz, 12.3 Hz, 1H), 1.88 (t, 12.3 Hz, 1H), 1.59 (dd, 1.8 Hz, 6.6 Hz, 3H); ^{13}C { ^1H } NMR (75 MHz, CDCl_3 , ppm): δ 172.3, 172.2, 141.1, 133.3, 132.3, 130.5, 118.0, 58.6, 52.9, 52.8, 41.7, 40.9, 36.7, 14.6; IR (neat): 2955, 1738, 1438, 1264, 1170, 912; HR-MS calcd. for $\text{C}_{15}\text{H}_{21}\text{O}_4$ (M+H): 265.1440, found 265.1429.

COSY summary; the following cross peaks were observed: H(1) and H(2); H(1) and H(4); H(6) and H(7); H(7) and H(8); H(8) and H(9); H(9) and H(10); H(10) and H(11).

HMBC summary; the following cross peaks were observed: H(1) and C(2), C(3); H(2) and C(4); H(4) and C(2), C(3), C(5), C(6), C(12); H(6) and C(3), C(4), C(5), C(7), C(12); H(8) and C(3), C(6), C(10); H(9) and C(7), C(10), C(11); H(10) and C(8), C(9); H(11) and C(9); H(13) and C(5), C(12).

Rearrangement of N-2-butynyl-N-[(2E)-3-cyclopropyl-2-propenyl]-4-methyl-benzenesulfonamide (10) catalyzed by $\text{Ni}(\text{COD})_2/\text{SIPr}$. The general procedure was used with N-2-butynyl-N-[(2E)-3-cyclopropyl-2-propenyl]-4-methyl-benzenesulfonamide (**10**, 100 mg, 0.3 mmol), $\text{Ni}(\text{COD})_2$ (5 mg, 0.018 mmol), SIPr (7 mg, 0.018 mmol), and 0.5 mL of toluene. The reaction mixture was

purified by column chromatography on silica gel (25% Et_2O /pentanes) to afford **11** (88 mg, 88%) as a white crystalline solid. ^1H NMR (300 MHz, CDCl_3 , ppm): δ 7.72 (d, 8.1 Hz, 2H), 7.35 (d, 8.1 Hz, 2H), 6.50 (dt, 10.5 Hz, 16.5 Hz, 1H), 6.12 (t, 10.5 Hz, 1H), 5.26 (d, 16.5 Hz, 1H), 5.15 (m, 3H), 3.98 (m, 1H), 3.66 (m, 3H), 2.66 (m, 1H), 2.45 (s, 3H), 1.53 (m, 3H); ^{13}C { ^1H } NMR (75 MHz, CDCl_3 , ppm): δ 143.9, 138.2, 133.0, 132.2, 131.8, 129.9, 129.4, 128.1, 119.4, 118.6, 53.6, 49.7, 41.8, 21.8, 14.7; IR (neat): 2925, 2856, 1706, 1597, 1447, 1346, 1162, 1099, 1025, 911, 818, 623, 588, 548; HR-MS calcd. For $\text{C}_{17}\text{H}_{22}\text{NO}_2\text{S}$ (M+H): 304.1371, found 304.1367.

COSY summary; the following cross peaks were observed: H(1) and H(2); H(1) and H(4); H(2) and H(4); H(5) and H(6); H(6) and H(7); H(7) and H(8); H(8) and H(9); H(9) and H(10).

HMBC summary; the following cross peaks were observed: H(1) and C(2), C(3); H(4) and C(2), C(3); H(5) and C(3), C(6), C(7); H(7) and C(5), C(6), C(9); H(8) and C(6), C(9), C(10); H(9) and C(7), C(8); H(10) and C(8).

Rearrangement of [(1E)-3-(4-methoxy-2-pentynyloxy)-1-propenyl]-cyclopropane (12) catalyzed by

Ni(COD)₂/tBu. The general procedure was used with [(1E)-3-(4-methoxy-2-pentynyloxy)-1-propenyl]-cyclopropane (**12**, 100 mg, 0.56 mmol), Ni(COD)₂ (8 mg, 0.03 mmol), tBu (5.4 mg, 0.03 mmol), and 0.5 mL of toluene. The reaction mixture was purified by column chromatography on silica gel (10% Et₂O/pentanes) to afford 3-(1,3-butadienyl)-tetrahydro-4-(2-methoxypropylidene)furan (**13**, 66 mg, 66%) as a colorless oil. ¹H NMR (300 MHz, CDCl₃, ppm): δ 6.63 (dt, 10.8 Hz, 16.8 Hz, 1H), 6.20 (t, 10.8 Hz, 1H), 5.34 (m, 4H), 4.49 (d, 13.8 Hz, 1H), 4.37 (d, 13.2 Hz, 1H), 4.12 (t, 8.4 Hz, 1H), 3.85 (m, 3H), 3.41 (t, 8.4 Hz, 1H), 3.32 (s, 3H); ¹³C {¹H} NMR (75 MHz, CDCl₃, ppm): δ 145.7, 132.15, 132.06, 119.1, 117.9, 73.3, 70.2, 69.4, 58.4, 43.6; IR (neat): 2978, 2844, 1596, 1440, 1381, 1195, 1115, 1073, 999, 912; HR-MS calcd. for C₁₁H₁₇O₂ (M+H): 181.1229, found 181.1224.

COSY summary; the following cross peaks were observed: H(2) and H(3); H(6) and H(7); H(7) and H(8); H(8) and H(9); H(9) and H(10); H(10) and H(11).

HMBC summary; the following cross peaks were observed: H(1) and C(2); H(2) and C(1), C(3), C(4); H(5) and C(3), C(4), C(6); H(6) and C(4), C(5), C(8); H(8) and C(6); H(9) and C(7), C(10), C(11); H(10) and C(8), C(9).

Rearrangement of [(1E)-3-(2-butynyloxy)-1-propenyl]-cyclopropane (14**) catalyzed by Ni(COD)₂/tBu.** The general procedure was used with [(1E)-3-(2-butynyloxy)-1-propenyl]-cyclopropane (**14**, 100 mg, 0.61 mmol), Ni(COD)₂ (8 mg, 0.03 mmol), tBu (5.4 mg, 0.03 mmol), and 0.5 mL of toluene. The reaction mixture was purified by column chromatography on

silica gel (10% Et₂O/pentanes) to afford 3-(1,3-butadienyl)-tetrahydro-2-methyl-4-propylidene-furan (**15**, 64 mg, 64%) as a colorless oil. ¹H NMR (300 MHz, CDCl₃, ppm): δ 6.60 (dt, 10.5 Hz, 17.1 Hz, 1H), 6.23 (t, 10.5 Hz, 1H), 5.27 (d, 17.1 Hz, 1H), 5.15 (m, 3H), 4.52 (d, 13.2 Hz, 1H), 4.34 (dt, 13.2 Hz, 1.5 Hz, 1H), 3.56 (m, 1H), 3.22 (m, 1H), 1.56 (m, 3H), 1.27 (d, 6.0 Hz, 3H); ¹³C {¹H} NMR (75 MHz, CDCl₃, ppm): δ 143.0, 132.5, 132.4, 129.9, 118.7, 115.7, 81.0, 69.5, 50.7, 18.8, 14.5; IR (neat): 3082, 2977, 2925, 2857, 1664, 1443, 1369, 1136, 1070, 962, 861; HR-MS calcd. for C₁₁H₁₇O (M+H): 165.1279, found 165.1277.

COSY summary; the following cross peaks were observed: H(1) and H(2); H(5) and H(6); H(6) and H(7); H(7) and H(8); H(8) and H(9); H(9) and H(10); H(10) and H(11).

HMBC summary; the following cross peaks were observed: H(1) and C(2), C(3); H(4) and C(2), C(3), C(6); H(5) and C(6), C(7); H(6) and C(8); H(8) and C(6), C(7), C(10); H(9) and C(7), C(10), C(11); H(10) and C(8), C(9).

Rearrangement of 1-Cyclopropyl-5-methoxy-1-octen-6-yne (16**) catalyzed by Ni(COD)₂/tBu.** The general procedure was used with 1-Cyclopropyl-5-methoxy-1-octen-6-yne (**16**, 100 mg, 0.56 mmol), Ni(COD)₂ (8 mg, 0.03 mmol), SiPr (11 mg, 0.03 mmol), and 0.5 mL of toluene. The reaction mixture was purified by column chromatography on

silica gel (10% Et₂O/pentanes) to afford 1-(1,3-butadienyl)-3-methoxy-2-propylidene-cyclopentane (**17**, 58 mg, 58%) as a colorless oil. ¹H NMR (300 MHz, CDCl₃, ppm): δ 6.33 (dt, 10.5 Hz, 16.8 Hz, 1H), 6.04 (dd, 10.2 Hz, 15.3 Hz, 1H), 5.59 (dd, 9.0 Hz, 15.3 Hz, 1H), 5.50 (m, 1H), 5.11 (d, 16.8 Hz, 1H), 4.96 (d, 10.2 Hz, 1H), 4.25 (m, 1H), 3.31 (s, 3H), 2.93 (m, 1H), 1.99 (m, 1H), 1.86 (m, 1H), 1.74 (m, 3H), 1.56 (m, 2H); ¹³C {¹H} NMR (75 MHz, CDCl₃, ppm): δ 145.5, 138.8, 137.4, 130.9, 124.4, 115.2, 79.9, 56.2, 47.7, 31.2, 31.0, 15.4; IR (neat): 2960, 2927, 2815, 1647, 1446, 1195, 1091, 1003, 950, 897, 841; HR-MS calcd. for C₁₂H₁₉O (M+H): 179.1436, found 179.1432.

COSY summary; the following cross peaks were observed: H(1) and H(2); H(4) and H(5); H(5) and H(6); H(6) and H(7); H(7) and H(8); H(8) and H(9); H(9) and H(10); H(10) and H(11).

HMBC summary; the following cross peaks were observed: H(1) and C(2), C(3); H(2) and C(4), C(7); H(4) and C(5), C(7), C(MeO); H(5) and C(3), C(4), C(6), C(7); H(6) and C(3), C(4), C(5), C(7), C(8); H(8) and C(7), C(10); H(9) and C(7), C(10), C(11); H(10) and C(8), C(9); H(11) and C(9).

Rearrangement of [3-(3-cyclopropyl-2-pentenyl)-1-propynyl]-cyclopropane (**18**) catalyzed by $\text{Ni}(\text{COD})_2/\text{tBu}$.

The general procedure was used with [3-(3-cyclopropyl-2-pentenyl)-1-propynyl]-cyclopropane (**18**, 100 mg, 0.57 mmol), $\text{Ni}(\text{COD})_2$ (8 mg, 0.03 mmol), tBu (5.4 mg, 0.03 mmol), and 0.5 mL of toluene. The reaction mixture was purified by column chromatography on silica gel (10% Et_2O /pentanes) to afford 3-(1,3-butadienyl)-4-(2-cyclopropylpropylidene)-tetrahydrofuran (**19**, 65 mg, 65%) as a colorless oil. ^1H NMR (300 MHz, CDCl_3 , ppm): δ 6.62 (dt, 10.5 Hz, 16.8 Hz, 1H), 6.15 (t, 10.5 Hz, 1H), 5.22 (m, 3H), 4.58 (m, 2H), 4.46 (m, 1H), 4.10 (t, 8.4 Hz, 1H), 3.76 (m, 1H), 3.41 (t, 8.4 Hz, 1H), 1.17 (m, 1H), 0.73 (m, 4H), 0.34 (m, 2H); ^{13}C { ^1H } NMR (75 MHz, CDCl_3 , ppm): δ 139.8, 132.2, 131.5, 130.3, 125.3, 118.7, 73.7, 69.8, 43.3, 11.7, 6.99, 6.86; IR (neat): 3083, 3006, 2843, 1643, 1433, 1072, 1000, 961, 907, 807; HR-MS calcd. for $\text{C}_{12}\text{H}_{17}\text{O}$ ($\text{M}+\text{H}$): 177.1279, found 177.1279.

COSY summary; the following cross peaks were observed: H(1) and H(2); H(2) and H(3); H(6) and H(7); H(7) and H(8); H(8) and H(9); H(9) and H(10); H(10) and H(11).

HMBC summary; the following cross peaks were observed: H(1) and C(2), C(3); H(3) and C(4), C(5); H(5) and C(3), C(4), C(6), C(7); H(6) and C(4), C(5), C(7), C(8); H(7) and C(3), C(4), C(6), C(8), C(9); H(8) and C(6), C(7); H(9) and C(7), C(10), C(11); H(10) and C(8), C(9); H(11) and C(9).

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^{13}C NMR spectrum of **1c** in CDCl_3

^{13}C NMR spectrum of **1d** in CDCl_3

Pulse Sequence: relayh

Solvent: CDCl₃
 Ambient temperature
 File: Me-ether-pl-cosy
 INOVA-500 "i500b"

Relax. delay 1.000 sec

COSY 90-90

Acq. time 0.186 sec

Width 5499.8 Hz

2D Width 5499.8 Hz

4 repetitions

512 increments

OBSERVE H1, 500.0912275 MHz

Single precision data

DATA PROCESSING

Resol. enhancement 27.3 Hz

Gauss apodization 0.047 sec

F1 DATA PROCESSING

Resol. enhancement 54.7 Hz

Gauss apodization 0.023 sec

FT size 2048 x 2048

Total time 42 min, 28 sec

COSY spectrum of 2a in CDCl₃

Pulse Sequence: gHMQC

Solvent: CDCl₃

Ambient temperature

File: Me-ether-P-HMQC

INOVA-500 "1500P"

Relax. delay 1.000 sec

Acq. time 0.184 sec

Width 2778.5 Hz

2D Width 21356.1 Hz

16 repetitions

2 x 128 increments

OBSERVE H1, 499.6156701 MHz

DECOUPLE C13, 125.6378518 MHz

Power 47 dB

on during acquisition

off during delay

GARP-1 modulated

DATA PROCESSING

Gauss apodization 0.085 sec

F1 DATA PROCESSING

Gauss apodization 0.011 sec

FT size 1024 x 2048

Total time 1 hr, 25 min, 12 sec

HMQC spectrum of 2a in CDCl₃

Pulse Sequence: gHMBC

Solvent: CDCl₃

Ambient temperature

File: Me-ether-P-HMBC

INOVA-500 "i500b"

Relax. delay 1.000 sec

Acq. time 0.184 sec

Width 2778.5 Hz

2D Width 30154.5 Hz

32 repetitions

400 increments

OBSERVE H1, 499.6156701 MHz

DATA PROCESSING

Sine bell 0.092 sec

F1 DATA PROCESSING

Sine bell 0.007 sec

FT size 1024 x 2048

Total time 4 hr, 32 min, 10 sec

HMBC spectrum of 2a in CDCl₃

Pulse Sequence: gHMQC

Solvent: CDCl₃
 Ambient temperature
 User: 1-14-87
 File: Et-ether-P2-HMQC
 INOVA-500 "1500b"

Relax. delay 1.000 sec
 Acq. time 0.170 sec
 Width 3008.8 Hz
 2D Width 21356.1 Hz
 16 repetitions
 2 x 128 increments

OBSERVE H1, 499.6156701 MHz
 DECOUPLE C13, 125.6378518 MHz
 Power 47 dB

on during acquisition
 off during delay
 GARP-1 modulated

DATA PROCESSING

Gauss apodization 0.079 sec

F1 DATA PROCESSING

Gauss apodization 0.011 sec

FT size 1024 x 2048

Total time 1 hr, 24 min, 13 sec

HMQC NMR spectrum of 2b in CDCl₃

Pulse Sequence: gHMBC

Solvent: CDCl₃

Ambient temperature

File: Et-ether-P2-HMBC

INOVA-500 "i500b"

Relax. delay 1.000 sec

Acq. time 0.170 sec

Width 3008.8 Hz

2D Width 30154.5 Hz

64 repetitions

400 increments

OBSERVE H1, 499.6156701 MHz

DATA PROCESSING

Sine bell 0.085 sec

F1 DATA PROCESSING

Sine bell 0.007 sec

FT size 1024 x 2048

Total time 8 hr, 57 min, 38 sec

HMBC NMR spectrum of 2b in CDCl₃

Pulse Sequence: gCOSY
 Solvent: CDCl₃
 Ambient temperature
 File: Pr-ether-PI-COSY
 INOVA-500 "1500B"

 Relax. delay 1.000 sec
 Acq. time 0.170 sec
 Width 3018.1 Hz
 2D Width 3018.1 Hz
 16 repetitions
 128 increments
 OBSERVE H1, 499.6156701 MHz
 DATA PROCESSING
 Sq. sine bell 0.085 sec
 F1 DATA PROCESSING
 Sq. sine bell 0.042 sec
 FT size 1024 x 1024
 Total time 41 min, 24 sec

COSY spectrum of 2c in CDCl₃

Pulse Sequence: gHMQC

Solvent: CDCl₃
Ambient temperature
File: Pr-ether-PI-HMQC
INOVA-500 "1500E"

Relax. delay 1.000 sec
Acq. time 0.170 sec
Width 3018.1 Hz
2D Width 21356.1 Hz
16 repetitions
2 x 128 increments
OBSERVE H1, 499.6156701 MHz
DECOUPLE C13, 125.6378518 MHz
Power 47 dB
on during acquisition
off during delay
GARP-1 modulated
DATA PROCESSING
Gauss apodization 0.078 sec
F1 DATA PROCESSING
Gauss apodization 0.011 sec
FT size 1024 x 2048
Total time 1 hr, 24 min, 11 sec

HMQC spectrum of 2c in CDCl₃

Pulse Sequence: gHMBC

Solvent: CDCl₃

Ambient temperature

File: Pr-ether-PI-HMBC

INOVA-500 "1500P"

Relax. delay 1.000 sec

Acq. time 0.170 sec

Width 3018.1 Hz

2D Width 30154.5 Hz

64 repetitions

400 increments

OBSERVE H1, 499.6156701 MHz

DATA PROCESSING

Sine bell 0.085 sec

F1 DATA PROCESSING

Sine bell 0.007 sec

FT size 1024 x 2048

Total time 8 hr, 57 min, 25 sec

HMBC spectrum of 2c in CDCl₃

STANDARD PROTON PARAMETERS

Pulse Sequence: gCOSY
 Solvent: CDCl₃
 Temp. 26.0 C / 299.1 K
 File: Bu-ether-P1-COSY
 INOVA-500 "i500b"

Relax. delay 1.000 sec
 Acq. time 0.159 sec
 Width 3218.8 Hz
 2D Width 3218.8 Hz
 8 repetitions
 128 increments

OBSERVE H1, 499.6156701 MHz
 DATA PROCESSING
 Sg. sine bell 0.080 sec
 F1 DATA PROCESSING
 Sg. sine bell 0.040 sec
 FT size 1024 x 1024
 Total time 20 min, 39 sec

COSY spectrum of 2d in CDCl₃

STANDARD PROTON PARAMETERS

Pulse Sequence: gHMOC

Solvent: CDCl₃
 Temp. 26.0 C / 299.1 K
 File: Bu-ether-P1-HMOC
 INOVA-500 "i500b"

Relax. delay 1.000 sec
 Acq. time 0.159 sec
 Width 3218.8 Hz
 2D Width 21356.1 Hz
 8 repetitions
 2 x 128 increments
 OBSERVE H1, 499.6156701 MHz
 DECOUPLE C13, 125.6378518 MHz
 Power 47 dB
 on during acquisition
 off during delay
 GARP-1 modulated
 DATA PROCESSING
 Gauss apodization 0.073 sec
 F1 DATA PROCESSING
 Gauss apodization 0.011 sec
 FT size 1024 x 2048
 Total time 42 min, 3 sec

HMOC spectrum of 2d in CDCl₃

2d

STANDARD PROTON PARAMETERS

Pulse Sequence: gHMBC
 Solvent: CDCl₃
 Temp. 26.0 C / 299.1 K
 File: Bu-ether-P1-HMBC
 INOVA-500 "1500b"

Relax. delay 1.000 sec
 Acq. time 0.159 sec
 Width 3218.8 Hz
 2D Width 30154.5 Hz
 16 repetitions
 400 increments
 OBSERVE H1, 499.6156701 MHz
 DATA PROCESSING
 Sine bell 0.080 sec
 F1 DATA PROCESSING
 Sine bell 0.007 sec
 FT size 1024 x 2048
 Total time 2 hr, 13 min, 43 sec

HMBC spectrum of 2d in CDCl₃

File: PROTON

Pulse Sequence: gCOSY

Solvent: CDCl₃

Temp. 26.0 C / 299.1 K

INOVA-500 "1500B"

Relax. delay 1.000 sec

Acq. time 0.202 sec

Width 2537.4 Hz

2D Width 2537.4 Hz

8 repetitions

128 increments

OBSERVE H1, 499.6156679 MHz

DATA PROCESSING

Sq. sine bell 0.101 sec

F1 DATA PROCESSING

Sq. sine bell 0.050 sec

FT size 1024 x 1024

Total time 21 min, 28 sec

COSY spectrum of 3b in CDCl₃

File: PROTON

Pulse Sequence: gHMQC

Solvent: CDCl₃

Temp. 26.0 C / 299.1 K

INOVA-500 "1500b"

Relax. delay 1.000 sec

Acq. time 0.202 sec

Width 2537.4 Hz

2D Width 21356.1 Hz

8 repetitions

2 x 128 increments

OBSERVE H1, 499.6156679 MHz

DECOUPLE C13, 125.6378518 MHz

Power 47 dB

on during acquisition

off during delay

GARP-1 modulated

DATA PROCESSING

Gauss apodization 0.093 sec

F1 DATA PROCESSING

Gauss apodization 0.011 sec

FT size 1024 x 2048

Total time 43 min, 32 sec

HMQC spectrum of 3b in CDCl₃

File: PROTON

Pulse Sequence: gHMBC

Solvent: CDCl₃

Temp. 26.0 C / 299.1 K

User: 1-14-87

INOVA-500 "i500b"

Relax. delay 1.000 sec

Acq. time 0.202 sec

Width 2537.4 Hz

2D Width 30154.5 Hz

16 repetitions

400 increments

OBSERVE H1, 499.6156679 MHz

DATA PROCESSING

Sine bell 0.101 sec

F1 DATA PROCESSING

Sine bell 0.007 sec

FT size 1024 x 2048

Total time 2 hr, 18 min, 18 sec

HMBC spectrum of 3b in CDCl₃

Pulse Sequence: gCOSY

Solvent: CDCl₃

Ambient temperature

File: Pr-ether-P3-COSY

INOVA-500 "1500B"

Relax. delay 1.000 sec

Acq. time 0.208 sec

Width 2460.1 Hz

2D Width 2460.1 Hz

16 repetitions

128 increments

OBSERVE H1, 499.6156701 MHz

DATA PROCESSING

Sq. sine bell 0.104 sec

F1 DATA PROCESSING

Sq. sine bell 0.052 sec

FT size 1024 x 1024

Total time 42 min, 53 sec

COSY spectrum of 3c in CDCl₃

3c

Pulse Sequence: gHMQC

Solvent: CDCl₃

Ambient temperature

User: 1-14-87

File: Pr-ether-P3-HMQC

INOVA-500 "i500b"

Relax. delay 1.000 sec

Acq. time 0.208 sec

Width 2460.1 Hz

2D Width 21356.1 Hz

16 repetitions

2 x 128 increments

OBSERVE H1, 499.6156701 MHz

DECOUPLE C13, 125.6378518 MHz

Power 47 dB

on during acquisition

off during delay

GARP-1 modulated

DATA PROCESSING

Gauss apodization 0.096 sec

F1 DATA PROCESSING

Gauss apodization 0.011 sec

FT size 1024 x 2048

Total time 1 hr, 26 min, 50 sec

HMQC spectrum of 3c in CDCl₃

3c

Pulse Sequence: gHMBC

Solvent: CDCl₃

Ambient temperature

User: 1-14-87

File: Pr-ether-P3-HMBC

INOVA-500 "1500b"

Relax. delay 1.000 sec

Acq. time 0.208 sec

Width 2460.1 Hz

2D Width 30154.5 Hz

32 repetitions

400 increments

OBSERVE H1, 499.6156701 MHz

DATA PROCESSING

Sine bell 0.104 sec

F1 DATA PROCESSING

Sine bell 0.007 sec

FT size 1024 x 2048

Total time 4 hr, 37 min, 16 sec

HMBC spectrum of 3c in CDCl₃

Pulse Sequence: gCOSY

Solvent: CDCl₃

Ambient temperature

INOVA-500 "1500b"

Relax. delay 1.000 sec

Acq. time 0.128 sec

Width 7993.6 Hz

2D Width 7993.6 Hz

16 repetitions

128 increments

OBSERVE H1, 499.6156679 MHz

DATA PROCESSING

Sq. sine bell 0.064 sec

F1 DATA PROCESSING

Sq. sine bell 0.016 sec

FT size 2048 x 2048

Total time 39 min, 31 sec

COSY spectrum of **4d** in CDCl₃

Pulse Sequence: gHMQC

Solvent: CDCl₃
 Ambient temperature
 User: 1-14-87
 INOVA-500 "1500B"

Relax. delay 1.000 sec
 Acq. time 0.195 sec
 Width 2621.4 Hz
 2D Width 21356.1 Hz
 16 repetitions
 2 x 128 increments

OBSERVE H1, 499.6156701 MHz
 DECOUPLE C13, 125.6378518 MHz
 Power 47 dB

on during acquisition
 off during delay
 GARP-1 modulated

DATA PROCESSING

Gauss apodization 0.090 sec
 F1 DATA PROCESSING
 Gauss apodization 0.011 sec
 FT size 1024 x 2048
 Total time 1 hr, 25 min, 57 sec

HMQC spectrum of 4d in CDCl₃

Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "i500b"

```

Relax. delay 1.000 sec
Acq. time 0.195 sec
Width 2321.4 Hz
2D Width 30154.5 Hz
64 repetitions
400 increments
OBSERVE H1, 499.6156701 MHz
DATA PROCESSING
Sine bell 0.098 sec
F1 DATA PROCESSING
Sine bell 0.007 sec
FFT size 1024 x 2048
Total time 9 hr, 8 min, 23 sec

```

HMBC spectrum of **4d** in CDCl_3

1H LINESHAPE
CHCL3/ACETONE-D6

Pulse Sequence: gCOSY
Solvent: CDCl3
Temp. 26.0 C / 299.1 K
File: TMS-ether-P-cosy
INOVA-500 "i500b"

Relax. delay 1.000 sec
Acq. time 0.186 sec
Width 5499.8 Hz
2D Width 5499.8 Hz
4 repetitions
128 increments

OBSERVE H1, 499.6156718 MHz
Single precision data
DATA PROCESSING
Sq. sine bell 0.093 sec
F1 DATA PROCESSING
Sq. sine bell 0.023 sec
FT size 2048 x 2048
Total time 10 min, 39 sec

COSY spectrum of 4e in CDCl₃

Pulse Sequence: gHMQC

Solvent: CDCl₃

Ambient temperature

User: 1-14-87

File: TMS-ether-Fl-HMQC

INOVA-500 "i500b"

Relax. delay 1.000 sec

Acq. time 0.168 sec

Width 3052.2 Hz

2D Width 21356.1 Hz

16 repetitions

2 x 128 increments

OBSERVE H1, 499.6156679 MHz

DECOUPLE C13, 125.6378518 MHz

Power 47 dB

on during acquisition

off during delay

GARP-1 modulated

DATA PROCESSING

Gauss apodization 0.078 sec

F1 DATA PROCESSING

Gauss apodization 0.011 sec

FT size 1024 x 2048

Total time 1 hr, 24 min, 3 sec

HMQC spectrum of 4e in CDCl₃

Pulse Sequence: gHMBC

Solvent: CDCl₃

Ambient temperature

File: TMS-ether-Pl-HMBC

INOVA-500 "i500p"

Relax. delay 1.000 sec

Acq. time 0.168 sec

Width 3052.2 Hz

2D Width 30154.5 Hz

32 repetitions

400 increments

OBSERVE H1, 499.6156679 MHz

DATA PROCESSING

Sine bell 0.084 sec

F1 DATA PROCESSING

Sine bell 0.007 sec

FT size 1024 x 2048

Total time 4 hr, 28 min, 38 sec

HMBC spectrum of 4e in CDCl₃

1H LINESHAPE

CHCL3 / ACETONE -D6

Pulse Sequence: relayh

Solvent: CDCl₃

Ambient temperature

File: Me-ester-P1-Cosy

INOVA-500 "i500b"

Relax. delay 1.000 sec

COSY 90-90

Acq. time 0.186 sec

Width 5499.8 Hz

2D Width 5499.8 Hz

4 repetitions

512 increments

OBSERVE H1, 500.0912275 MHz

Single precision data

DATA PROCESSING

Resol. enhancement 27.3 Hz

Gauss apodization 0.047 sec

F1 DATA PROCESSING

Resol. enhancement 54.7 Hz

Gauss apodization 0.023 sec

FT size 2048 x 2048

Total time 42 min, 28 sec

COSY spectrum of **9** in CDCl_3

Pulse Sequence: gHMQC

Solvent: CDCl₃

Ambient temperature

User: 1-14-87

File: Me-ester-P2-HMQC

INOVA-500 "1500b"

Relax. delay 1.000 sec

Acq. time 0.182 sec

Width 2813.1 Hz

2D Width 21356.1 Hz

16 repetitions

2 x 128 increments

OBSERVE H1, 499.6156701 MHz

DECOUPLE C13, 125.6378518 MHz

Power 47 dB

on during acquisition

off during delay

GARP-1 modulated

DATA PROCESSING

Gauss apodization 0.084 sec

F1 DATA PROCESSING

Gauss apodization 0.011 sec

FT size 1024 x 2048

Total time 1 hr, 25 min, 2 sec

HMQC spectrum of 9 in CDCl₃

MeOOC COOMe

9

Pulse Sequence: gHMBC

Solvent: CDCl₃

Ambient temperature

File: Me-ester-P2-HMBC

INOVA-500 "1500E"

Relax. delay 1.000 sec

Acq. time 0.182 sec

Width 2813.1 Hz

2D Width 30154.5 Hz

32 repetitions

400 increments

OBSERVE H1, 499.6156701 MHz

DATA PROCESSING

Sine bell 0.091 sec

F1 DATA PROCESSING

Sine bell 0.007 sec

FT size 1024 x 2048

Total time 4 hr, 31 min, 41 sec

HMBC spectrum of **9** in CDCl₃

MeOOC COOMe

9

Pulse Sequence: gCOSY

Solvent: CDCl₃
Temp. 26.0 C / 299.1 K
INOVA-500 "1500B"

Relax. delay 1.000 sec
Acq. time 0.152 sec
Width 3376.0 Hz
2D Width 3376.0 Hz
8 repetitions

128 increments

OBSERVE H1, 499.6156721 MHz

DATA PROCESSING

Sq. sine bell 0.076 sec

F1 DATA PROCESSING

Sq. sine bell 0.038 sec

FT size 1024 x 1024

Total time 20 min, 30 sec

COSY spectrum of 11 in CDCl₃

Pulse Sequence: gHMOC

Solvent: CDCl₃
Temp. 26.0 C / 299.1 K
User: 1-14-87
INOVA-500 "i500b"

Relax. delay 1.000 sec
Acq. time 0.153 sec
Width 3347.8 Hz
2D Width 21356.1 Hz
16 repetitions
2 x 128 increments
OBSERVE H1, 499.6156679 MHz
DECOUPLE C13, 125.6378518 MHz
Power 47 dB
on during acquisition
off during delay
GARP-1 modulated
DATA PROCESSING
Gauss apodization 0.071 sec
F1 DATA PROCESSING
Gauss apodization 0.011 sec
FT size 1024 x 2048
Total time 1 hr, 23 min, 2 sec

HMOC spectrum of 11 in CDCl₃

Pulse Sequence: gHMBC

Solvent: CDCl₃
Temp. 26.0 C / 299.1 K
User: 1-14-87
INOVA-500 "i500b"

Relax. delay 1.000 sec
Acq. time 0.152 sec
Width 3372.3 Hz
2D Width 30154.5 Hz
32 repetitions
400 increments
OBSERVE H1, 499.6156679 MHz
DATA PROCESSING
Sine bell 0.076 sec
F1 DATA PROCESSING
Sine bell 0.007 sec
FT size 1024 x 2048
Total time 4 hr, 25 min, 14 sec

HMBC spectrum of 11 in CDCl₃

¹H NMR spectrum of 12 in CDCl₃

¹³C NMR spectrum of 13 in CDCl₃

Pulse Sequence: gCOSY

Solvent: CDCl₃

Temp. 26.0 C / 299.1 K

INOVA-500 "i500b"

Relax. delay 1.000 sec

Acq. time 0.137 sec

Width 1868.3 Hz

2D Width 1868.3 Hz

16 repetitions

128 increments

OBSERVE H1, 499.6156679 MHz

DATA PROCESSING

Sq. sine bell 0.069 sec

F1 DATA PROCESSING

Sq. sine bell 0.069 sec

FT size 1024 x 1024

Total time 40 min, 43 sec

COSY spectrum of 13 in CDCl₃

13

Pulse Sequence: gHMQC

Solvent: CDCl₃
Temp. 26.0 C / 299.1 K
User: 1-14-87
INOVA-500 "i500b"

Relax. delay 1.000 sec
Acq. time 0.137 sec
Width 1868.3 Hz
2D Width 21356.1 Hz
16 repetitions
2 x 128 increments
OBSERVE H1, 499.6156679 MHz
DECOUPLE C13, 125.6378518 MHz
Power 47 dB
on during acquisition
off during delay
GARP-1 modulated
DATA PROCESSING
Gauss apodization 0.063 sec
F1 DATA PROCESSING
Gauss apodization 0.011 sec
FT size 512 x 2048
Total time 1 hr, 21 min, 56 sec
HMQC spectrum of 13 in CDCl₃

Pulse Sequence: gHMBC

Solvent: CDCl₃
Temp. 26.0 C / 299.1 K
User: 1-14-87
INOVA-500 "i500b"

Relax. delay 1.000 sec
Acq. time 0.137 sec
Width 1868.3 Hz
2D Width 30154.5 Hz
16 repetitions
400 increments

OBSERVE H1, 499.6156679 MHz

DATA PROCESSING

Sine bell 0.069 sec

F1 DATA PROCESSING

Sine bell 0.007 sec

FT size 512 x 2048

Total time 2 hr, 11 min, 21 sec

HMBC spectrum of 13 in CDCl₃

13

Pulse Sequence: gCOSY
 Solvent: CDCl₃
 Temp. 26.0 C / 299.1 K
 INOVA-500 "1500E"

 Relax. delay 1.000 sec
 Acq. time 0.166 sec
 Width 3084.2 Hz
 2D Width 3084.2 Hz
 16 repetitions
 128 increments
 OBSERVE H1, 499.6156679 MHz
 DATA PROCESSING
 Sq. sine bell 0.083 sec
 F1 DATA PROCESSING
 Sq. sine bell 0.042 sec
 FT size 1024 x 1024
 Total time 41 min, 15 sec

COSY spectrum of 15 in CDCl₃

Pulse Sequence: gHMQC

Solvent: CDCl₃

Temp. 26.0 C / 299.1 K

User: 1-14-87

INOVA-500 "1500b"

Relax. delay 1.000 sec

Acq. time 0.166 sec

Width 3084.2 Hz

2D Width 21356.1 Hz

16 repetitions

2 x 128 increments

OBSERVE H1, 499.6156679 MHz

DECOUPLE C13, 125.6378518 MHz

Power 47 dB

on during acquisition

off during delay

GARP-1 modulated

DATA PROCESSING

Gauss apodization 0.077 sec

F1 DATA PROCESSING

Gauss apodization 0.011 sec

FT size 1024 x 2048

Total time 1 hr, 23 min, 56 sec

HMQC spectrum of 15 in CDCl₃

Pulse Sequence: gHMEC

Solvent: CDCl₃
Temp. 26.0 C / 299.1 K
User: 1-14-87
INOVA-500 "1500b"

Relax. delay 1.000 sec
Acq. time 0.166 sec
Width 3084.2 Hz
2D Width 30154.5 Hz
32 repetitions
400 increments
OBSERVE H1, 499.6156679 MHz
DATA PROCESSING
Sine bell 0.083 sec
F1 DATA PROCESSING
Sine bell 0.007 sec
FT size 1024 x 2048
Total time 4 hr, 28 min, 16 sec

HMBC spectrum of 15 in CDCl₃

Archive directory: /home/louie/zuogang/vnmrsys/data F2
 Sample directory:
 File: PROTON

Pulse Sequence: gCOSY
 Solvent: CDCl₃
 Temp. 26.0 C / 299.1 K
 INOVA-500 "1500b"

Relax. delay 1.000 sec
 Acq. time 0.166 sec
 Width 3085.5 Hz
 2D Width 3085.5 Hz
 8 repetitions
 128 increments
 OBSERVE HL, 499.6156679 MHz
 DATA PROCESSING
 Sg. sine bell 0.083 sec
 F1 DATA PROCESSING
 Sg. sine bell 0.041 sec
 FT size 1024 x 1024
 Total time 20 min, 47 sec

COZY spectrum of 17 in CDCl₃

Pulse Sequence: gHMQC

Solvent: CDCl₃
Temp. 26.0 C / 299.1 K
User: 1-14-87
INOVA-500 "1500b"

Relax. delay 1.000 sec
Acq. time 0.166 sec
Width 3085.5 Hz

2D Width 21356.1 Hz
16 repetitions

2 x 128 increments

OBSERVE H1, 499.6156679 MHz

DECOUPLE C13, 125.6378518 MHz

Power 47 dB

on during acquisition

off during delay

GARP-1 modulated

DATA PROCESSING

Gauss apodization 0.077 sec

F1 DATA PROCESSING

Gauss apodization 0.011 sec

FT size 1024 x 2048

Total time 1 hr, 23 min, 56 sec

HMQC spectrum of 17 in CDCl₃

Pulse Sequence: gHMBC

Solvent: CDCl₃

Temp. 26.0 C / 299.1 K

INOVA-500 "i500E"

Relax. delay 1.000 sec

Acq. time 0.166 sec

Width 3085.5 Hz

2D Width 30154.5 Hz

16 repetitions

400 increments

OBSERVE H1, 499.6156679 MHz

DATA PROCESSING

Sine bell 0.083 sec

F1 DATA PROCESSING

Sine bell 0.007 sec

FT size 1024 x 2048

Total time 2 hr, 14 min, 27 sec

HMBC spectrum of 17 in CDCl₃

¹³C NMR spectrum of **19** in CDCl₃

Pulse Sequence: gHMQC

Solvent: CDCl₃
Temp. 26.0 C / 299.1 K
User: 1-14-87
INOVA-500 "1500b"

Relax. delay 1.000 sec
Acq. time 0.154 sec
Width 3332.1 Hz
2D Width 21356.1 Hz
16 repetitions
2 x 128 increments
OBSERVE H1, 499.6156679 MHz
DECOUPLE C13, 125.6378518 MHz
Power 47 dB
on during acquisition
off during delay
GARP-1 modulated
DATA PROCESSING
Gauss apodization 0.071 sec
F1 DATA PROCESSING
Gauss apodization 0.011 sec
FT size 1024 x 2048
Total time 1 hr, 23 min, 5 sec

HMQC spectrum of 19 in CDCl₃

19

Pulse Sequence: gHMBC

Solvent: CDCl₃
Temp. 26.0 C / 299.1 K
User: 1-14-87
INOVA-500 "i500b"

Relax. delay 1.000 sec
Acq. time 0.154 sec
Width 3332.1 Hz
2D Width 30154.5 Hz
16 repetitions
400 increments
OBSERVE H1, 499.6156679 MHz
DATA PROCESSING
Sine bell 0.077 sec
F1 DATA PROCESSING
Sine bell 0.007 sec
FT size 1024 x 2048
Total time 2 hr, 13 min, 8 sec

HMBC spectrum of 19 in CDCl₃

¹³C NMR spectrum of **21** in CDCl₃

21