Supporting Information

**Efficient Synthesis of Cyclopentenones from Enynyl Acetates via
Tandem Au(I)-Catalyzed 3,3-Rearrangement and the Nazarov Reaction**

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General. Ethyl acetate (ACS grade), hexanes (ACS grade) and diethyl ether (ACS grade) were purchased from Fisher Scientific and used without further purification. Anhydrous dichloromethane (HPLC grade) was purified by distillation over calcium hydride. Anhydrous tetrahydrofuran in Pure-Pac™ from Aldrich was used directly without further purification. Commercially available reagents were used without further purification. Reactions were monitored by thin layer chromatography(TLC) using whatman precoated silica gel plates. Flash column chromatography was performed over silacyle silica gel (230-400 mesh). ¹H NMR and ¹³C NMR spectra were recorded on a Varian 500 MHz Unity plus spectrometer using residue solvent peaks as internal standards. Infrared spectra were recorded with a Perkin Elmer FT-IR spectrum 2000 spectrometer and are reported in reciprocal centimeter (cm⁻¹). Mass spectra were recorded with Waters micromass ZQ detector using electron spray method.

General Procedure A: Preparation of terminally 2-propenyl-substituted propargyl acetates

To a solution of 2-methyl-1-buten-3-yne (0.24 mL, 2.5 mmol) in THF (5.0 mL) was added *n*-BuLi (1.60 M solution in hexanes, 1.56 mL, 2.5 mmol) at -78°C and the resulting solution was stirred at the same temperature for 15 min. To this solution was added the aldehyde (2.5 mmol) neat via syringe and the reaction was stirred for another 15 min. Aqueous NH₄Cl solution was added to quench the reaction. The resulting mixture was extracted with diethyl ether (3 x 20 mL). The combined organic layers were washed with water (40 mL) and brine (40 mL) successively, dried (anhydrous MgSO₄) and concentrated. The residue was purified through silica gel flash column chromatography (hexanes/ethyl acetate = 4/1) to yield the corresponding enynyl alcohol.

To a solution of the above enynyl alcohol (2.0 mmol), pyridine (1.65 mL, 20.0 mmol) and catalytic amount of DMAP in anhydrous CH₂Cl₂ (6.0 mL) at 0°C was slowly added acetyl chloride (0.29 mL, 4.0 mmol). The reaction was stirred at the same temperature for 30 min before being diluted with hexanes (30 mL). The solid precipitates were filtered off and the filtrate obtained was concentrated. The residue was purified through silica gel flash column chromatography (hexanes/ethyl acetate = 20/1) to yield the desired acetate.

Compound **1** was prepared in 91% yield according to the general procedure A. ^1H NMR (500MHz, CDCl_3) δ 5.48 (t, 1H, $J = 7.0$ Hz), 5.31 (s, 1H), 5.24 (s, 1H), 2.08 (s, 3H), 1.88 (s, 3H), 1.79 - 1.74 (m, 2H), 1.43 (p, 2H, $J = 7.0$ Hz), 1.34 - 1.30 (m, 4H), 0.90 (t, 3H, $J = 7.0$ Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 170.0, 126.1, 122.6, 86.4, 85.6, 64.5, 34.8, 31.3, 24.7, 23.3, 22.5, 21.1, 13.9; IR (neat): 3099, 2956, 2932, 2863, 2128, 1744, 1615, 1457, 1372; MS (ES^+) Calculated for $[\text{C}_{13}\text{H}_{20}\text{NaO}_2]^+$: 231.1; Found: 231.0.

Compound **4** was prepared in 95% yield according to the general procedure A. ^1H NMR (500MHz, CDCl_3) δ 5.34 (d, 1H, $J = 6.5$ Hz), 5.31 (s, 1H), 5.24 (s, 1H), 2.09 (s, 3H), 1.88 (s, 3H), 1.87 - 1.83 (m, 1H), 1.80 - 1.73 (m, 3H), 1.71 - 1.64 (m, 2H), 1.30 - 1.07 (m, 5H); ^{13}C NMR (125 MHz, CDCl_3) δ 170.1, 126.2, 122.5, 87.0, 84.6, 68.6, 42.0, 28.6, 28.1, 26.2, 25.8, 25.7, 23.3, 21.0; IR (neat): 3098, 2930, 2855, 2228, 1741, 1615, 1452, 1372; MS (ES^+) Calculated for $[\text{C}_{14}\text{H}_{20}\text{NaO}_2]^+$: 243.1; Found: 243.0.

Compound **6** was prepared in 52% yield according to the general procedure A. ^1H NMR (500MHz, CDCl_3) δ 5.31 (s, 1H), 5.24 (s, 1H), 5.23 (s, 1H), 2.11 (s, 3H), 1.88 (s, 3H), 1.02 (s, 9H); ^{13}C NMR (125 MHz, CDCl_3) δ 170.2, 126.2, 122.4, 86.8, 84.5, 72.1, 35.3, 25.6, 23.3, 20.9; IR (neat): 3099, 2972, 2935, 2872, 2231, 1743, 1615, 1480, 1464, 1370; MS (ES^+) Calculated for $[\text{C}_{12}\text{H}_{18}\text{NaO}_2]^+$: 217.1; Found: 217.0.

General Procedure B: Preparation of enynyl acetates with substituent other than methyl

To a solution of alkenyl bromide or triflate (0.6 mmol) in THF (3 mL) was added diisopropylamine (1 mL), 1-octyn-3-ol (0.5 mmol), $\text{PdCl}_2(\text{PPh}_3)_2$ (7 mg, 0.01 mmol) and CuI (3.8 mg, 0.02 mmol). The resulting mixture was stirred at ambient temperature for 2 h before being diluted with Et_2O (20 mL) and treated with H_2O (20 mL). The ethereal layer was separated and the aqueous layer was extracted with Et_2O (3 x 10 mL). The combined organic layers was washed with H_2O (20 mL), brine (20 mL), dried (MgSO_4) and concentrated. The residue was purified through silica gel flash column chromatography (EtOAc /hexanes = 1/3) to yield the corresponding enynyl alcohol as a clear liquid.

To a solution of the above enynyl alcohol, pyridine (0.41 mL, 5.0 mmol) and catalytic amount of DMAP in anhydrous CH_2Cl_2 (5.0 mL) at 0°C was slowly added acetyl chloride (0.07 mL, 1 mmol). The solution was stirred at the same temperature for 30 min and diluted with hexanes (30 mL). The solid precipitates were filtered off and the filtrate obtained was concentrated. The residue was purified through silica gel flash column chromatography (hexanes/ethyl acetate = 20/1) to yield the desired acetate.

Compound **8** was prepared in 72% yield from vinyl bromide according to the general procedure B. ^1H NMR (500 MHz, CDCl_3) 5.80 (dd, 1H, J = 11.0, 20.0 Hz), 5.65 (d, 1H, J = 16.8 Hz), 5.52 - 5.46 (m, 2H), 2.08 (s, 3H), 1.80 - 1.74 (m, 2H), 1.43 (p, 2H, J = 7.0 Hz), 1.32 - 1.31 (m, 4H), 0.89 (t, 3H, J = 6.5 Hz); ^{13}C NMR (125 MHz, CDCl_3) 170.0, 127.8, 116.4, 87.2, 83.8, 64.5, 34.7, 31.2, 24.7, 22.4, 21.0, 13.9; IR (neat) 2957, 2933, 2863, 1746, 1600, 1371; MS (ES^+) Calculated for $[\text{C}_{12}\text{H}_{18}\text{NaO}_2]^+$: 217.1; Found: 217.1.

10

Compound **10** was prepared in 79% yield from cyclopenten-1-yl triflate¹ according to the general procedure B. ¹H NMR (500MHz, CDCl₃) δ 6.07 (bs, 1H), 5.51 (t, 1H, *J* = 7.0 Hz), 2.48 - 2.39 (m, 4H), 2.08 (s, 3H), 1.89 (p, 2H, *J* = 7.5 Hz), 1.82 - 1.71 (m, 2H), 1.44 (p, 2H, *J* = 7.5 Hz), 1.36 - 1.27 (m, 4H), 0.89 (t, 3H, *J* = 7.0 Hz); ¹³C NMR (125 MHz, CDCl₃) δ 170.1, 138.9, 123.7, 87.7, 82.6, 64.7, 36.3, 34.9, 33.2, 31.3, 24.7, 23.2, 22.5, 21.1, 13.9; IR (neat): 2956, 2932, 2860, 2225, 1746, 1370; MS (ES⁺) Calculated for [C₁₅H₂₂NaO₂]⁺: 257.1; Found: 257.0.

12

Compound **12** was prepared in 30% yield according to the general procedure A where 1-ethynyl-1-cyclohexene was used instead of 2-methyl-1-buten-3-yne. ¹H NMR (500MHz, CDCl₃) δ 6.12 (bs, 1H), 5.48 (t, 1H, *J* = 6.5 Hz), 2.12 - 2.05 (m, 6H), 1.80 - 1.69 (m, 2H), 1.64 - 1.54 (m, 4H), 1.43 (p, 2H, *J* = 7.0 Hz), 1.36 - 1.24 (m, 5H), 0.89 (t, 3H, *J* = 7.0 Hz); ¹³C NMR (125 MHz, CDCl₃) δ 170.1, 135.8, 119.9, 87.0, 83.9, 64.7, 35.0, 31.3, 29.0, 25.6, 24.7, 22.5, 22.2, 21.4, 21.1, 13.9; IR (neat): 3028, 2932, 2861, 2223, 1743, 1370; MS (ES⁺) Calculated for [C₁₆H₂₄NaO₂]⁺: 271.1 Found: 270.9.

14

Compound **14** was prepared in 85% yield from hepten-1-yl triflate¹ according to the general procedure B. ¹H NMR (500MHz, CDCl₃) δ 6.30 (t, 1H, *J* = 6.5 Hz), 5.49 (t, 1H, *J* = 7.0 Hz), 2.32 - 2.29 (m, 2H), 2.17 (q, 2H, *J* = 6.5 Hz), 2.07 (s, 3H), 1.78 - 1.70 (m, 4H), 1.57 - 1.47 (m, 4H), 1.46 - 1.39 (p, 2H, *J* = 7.0 Hz), 1.34 - 1.28 (m, 4H), 0.89 (t, 3H, *J* = 7.0 Hz); ¹³C NMR (125 MHz, CDCl₃) δ 170.1, 140.9, 126.0, 88.7, 83.7, 64.8, 35.0, 34.1, 32.0, 31.3, 29.1, 26.5, 26.4, 24.7, 22.5, 21.2, 13.9; IR (neat): 3027, 2925, 2854, 2219, 1743, 1447, 1370; MS (ES⁺) Calculated for [C₁₇H₂₆NaO₂]⁺: 285.2 Found: 285.1.

16

Compound **16** was prepared in 95% yield from α -bromostyrene according to the general procedure B. ^1H NMR (500MHz, CDCl_3) δ 7.63 - 7.61 (m, 2H), 7.37 - 7.34 (m, 2H), 7.32 - 7.29 (m, 1H), 5.94 (s, 1H), 5.69 (s, 1H), 5.59 (t, 1H, J = 6.0 Hz), 2.11 (s, 3H), 1.88 - 1.83 (m, 2H), 1.53 - 1.48 (m, 2H), 1.36 - 1.31 (m, 4H), 0.91 (t, 3H, J = 7.0 Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 170.0, 136.9, 129.9, 128.37, 128.35, 126.0, 121.5, 88.2, 84.3, 64.5, 34.8, 31.3, 24.8, 22.5, 21.1, 13.9; IR (neat): 3062, 2956, 2932, 2862, 1743, 1598, 1495, 1371; MS (ES^+) Calculated for $[\text{C}_{18}\text{H}_{22}\text{NaO}_2]^+$: 293.1 Found: 293.1.

18

Compound **18** was prepared in 66% yield from TIPS-protected 3-bromobut-3-en-1-ol according to the general procedure B. ^1H NMR (500MHz, CDCl_3) δ 5.48 (t, 1H, J = 7.0 Hz), 5.39 (s, 1H), 5.32 (s, 1H), 3.82 (t, 2H, J = 7.0 Hz), 2.38 (t, 2H, J = 7.0 Hz), 2.07 (s, 3H), 1.78 - 1.72 (m, 2H), 1.43 (p, 2H, J = 7.0 Hz), 1.33 - 1.28 (m, 4H), 1.12 - 1.00 (m, 21H), 0.90 (t, 3H, J = 7.0 Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 170.0, 127.5, 123.8, 86.4, 85.3, 64.4, 61.7, 40.7, 34.8, 31.3, 24.7, 22.5, 21.1, 18.0, 14.0, 12.0; IR (neat): 3097, 2944, 2867, 1745, 1612, 1464, 1370; MS (ES^+) Calculated for $[\text{C}_{23}\text{H}_{42}\text{NaO}_3\text{Si}]^+$: 417.2 Found: 417.1.

20

Compound **20** was prepared in 86% yield according to the general procedure A. ^1H NMR (500MHz, CDCl_3) δ 7.53 (d, 2H, J = 7.0 Hz), 7.41 - 7.36 (m, 3H), 6.59 (s, 1H), 5.37 (s, 1H), 5.29 (s, 1H), 2.11 (s, 3H), 1.91 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 169.8, 137.2, 128.9, 128.6, 127.8, 125.9, 123.2, 88.2, 84.5, 66.0, 23.2, 21.1; IR (neat): 3093, 3067, 3036, 2976, 2955, 2923, 2231, 1742, 1614, 1495, 1456, 1370; MS (ES^+) Calculated for $[\text{C}_{14}\text{H}_{14}\text{NaO}_2]^+$: 237.1 Found: 237.0.

23

Compound **23** was prepared in 82% yield according to the general procedure A. ^1H NMR (500MHz, CDCl_3) δ 7.65 (s, 4H), 6.62 (s, 1H), 5.38 (s, 1H), 5.31 (s, 1H), 2.13 (s, 3H), 1.91 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 169.6, 141.1, 131.1 (q, $J_{\text{C-F}} = 32.6$ Hz), 128.0, 125.7 (q, $J_{\text{C-F}} = 4.0$ Hz), 123.9 (q, $J_{\text{C-F}} = 272.3$ Hz), 123.7, 88.8, 83.6, 65.2, 23.1, 21.0; IR (neat): 3100, 2958, 2924, 2850, 2232, 1745, 1622, 1372; MS (ES^+) Calculated for $[\text{C}_{15}\text{H}_{13}\text{F}_3\text{NaO}_2]^+$: 305.1 Found: 305.0.

General Procedure C: $\text{AuCl}(\text{PPh}_3)/\text{AgSbF}_6$ -catalyzed formation of cyclopentenones from enynyl acetates

To a solution of the enynyl acetate (0.2 mmol) in wet CH_2Cl_2 (4.0 mL, 0.05 M) was added $\text{AuCl}(\text{PPh}_3)/\text{AgSbF}_6$ [pregenerated as 0.01 M solution in CH_2Cl_2 by mixing $\text{AuCl}(\text{PPh}_3)$ with AgSbF_6 , 0.2 mL] at room temperature and the reaction mixture was stirred for 30 min. The mixture was treated with one drop of Et_3N and concentrated. The residue was purified by silica gel flash column chromatography (ethyl acetate/hexanes = 10/1) to give the corresponding cyclopentenone.

3

Compound **3** was prepared in 92% yield according to the general procedure C. ^1H NMR (500MHz, CDCl_3) δ 5.89 (s, 1H), 2.81 (dd, 1H, $J = 6.5, 18.0$ Hz), 2.43 - 2.36 (m, 3H), 2.17 (d, 1H, $J = 18.0$ Hz), 1.60 - 1.54 (m, 2H), 1.36 - 1.30 (m, 4H), 1.17 (d, 3H, $J = 7.5$ Hz), 0.91 (t, 3H, $J = 7.0$ Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 212.7, 181.3, 128.1, 40.7, 40.4, 33.4, 31.6, 26.7, 22.4, 16.5, 13.9; IR (neat) 2959, 2930, 2873, 1705, 1616, 1459, 1432; MS (ES^+) Calculated for $[\text{C}_{11}\text{H}_{18}\text{NaO}]^+$: 189.1; Found: 189.0.

5

Compound **5** was prepared in 95% yield according to the general procedure C. ^1H NMR (500MHz, CDCl_3) δ 5.88 (s, 1H), 2.84 (ddd, 1H, J = 1.8, 6.5, 18.0 Hz), 2.38 (dp, 1H, J = 1.8, 7.3 Hz), 2.26 (t, 1H, J = 11.0 Hz), 2.17 (d, 1H, J = 18.0 Hz), 1.93 – 1.87 (m, 2H), 1.85 – 1.79 (m, 2H), 1.77 – 1.70 (m, 1H), 1.41 - 1.20 (m, 5H), 1.17 (d, 3H, J = 7.3 Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 212.8, 185.6, 126.5, 41.8, 40.4, 38.4, 31.21, 31.18, 26.0, 25.9, 16.5; IR (neat): 2928, 2853, 1705, 1608, 1450; MS (ES^+) Calculated for $[\text{C}_{12}\text{H}_{18}\text{NaO}]^+$: 201.1; Found: 201.0.

7

Compound **7** was prepared in 88% yield according to the general procedure C. ^1H NMR (500MHz, CDCl_3) δ 5.90 (s, 1H), 2.89 (ddd, 1H, J = 1.4, 7.0, 18.1 Hz), 2.42 (dp, 1H, J = 1.4, 7.0 Hz), 2.20 (dd, 1H, J = 1.4, 18.0 Hz), 1.18 (s, 9H), 1.17 (d, 3H, J = 8.5 Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 213.1, 189.2, 125.8, 40.8, 36.5, 35.0, 28.7, 16.5; IR (neat): 3080, 2967, 2933, 2909, 2871, 1705, 1603, 1479, 1459, 1367; MS (ES^+) Calculated for $[\text{C}_{10}\text{H}_{16}\text{NaO}]^+$: 175.1; Found: 174.9.

9

Compound **9** was prepared in 79% yield according to the general procedure C. ^1H NMR (500 MHz, CDCl_3) δ 5.93 (s, 1H), 2.57 - 2.55 (m, 2H), 2.40 - 2.37 (m, 4H), 1.60 - 1.54 (m, 2H), 1.35 - 1.28 (m, 4H), 0.89 (t, 3H, J = 6.5 Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 210.1, 183.2, 129.4, 35.2, 33.4, 31.44, 31.43, 26.7, 22.3, 13.9; IR (neat) 2957, 2929, 2861, 1708, 1675, 1616, 1467, 1438; MS (ES^+) Calculated for $[\text{C}_{10}\text{H}_{16}\text{NaO}]^+$: 175.1; Found: 175.1.

Compound **11** was prepared in 57% yield according to the general procedure C. ^1H NMR (500MHz, CDCl_3) δ 5.89 (s, 1H), 3.19 (q, 1H, J = 5.5 Hz), 2.77- 2.74 (m, 1H), 2.47 - 2.40 (m, 1H), 2.32 - 2.26 (m, 1H), 1.90 - 1.86 (m, 1H), 1.72 - 1.51 (m, 6H), 1.38 - 1.22 (m, 5H), 0.90 (t, 3H, J = 7.0 Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 212.8, 185.0, 129.6, 51.1, 48.8, 31.7, 31.5, 29.3, 28.7, 26.7, 24.0, 22.4, 14.0; IR (neat): 2956, 2934, 2869, 1699, 1613, 1467, 1450; MS (ES^+) Calculated for $[\text{C}_{13}\text{H}_{20}\text{NaO}]^+$: 215.1 Found: 214.9.

Compound **13** was prepared in 84% yield according to the general procedure C. ^1H NMR (500MHz, CDCl_3) δ 5.88 (s, 1H), 2.81 (q, 1H, J = 6.0 Hz), 2.46 - 2.32 (m, 2H), 2.31 - 2.25 (m, 1H), 2.02 - 1.91 (m, 2H), 1.68 - 1.48 (m, 5H), 1.38 - 1.29 (m, 5H), 1.28 - 1.19 (m, 1H), 1.14 - 1.06 (m, 1H), 0.90 (t, 3H, J = 7.0 Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 211.1, 185.6, 126.9, 46.6, 43.0, 31.6, 31.3, 28.0, 26.4, 22.5, 22.4, 21.7, 21.4, 13.9; IR (neat): 2928, 2856, 1709, 1611, 1460, 1449; MS (ES^+) Calculated for $[\text{C}_{14}\text{H}_{22}\text{NaO}]^+$: 229.2 Found: 229.0.

Compound **15** in combination of inseparable *trans*-fused isomer (6%) and double bond isomer (see below, 6%) was prepared in 90% yield according to the general procedure C. Spectra data of **13**: ^1H NMR (500MHz, CDCl_3) δ 5.95 (s, 1H), 2.97 (bs, 1H), 2.56 - 2.51 (m, 1H), 2.45 - 2.38 (m, 1H), 2.29 - 2.21 (m, 1H), 1.98 - 1.90 (m, 2H), 1.72 - 1.24 (m, 14H), 0.90 (t, 3H, J = 6.8 Hz) ; ^{13}C NMR (125 MHz, CDCl_3) δ 211.7, 185.3, 128.8, 51.6, 49.2, 31.6, 31.4, 31.15, 31.14, 29.0, 28.2, 27.8, 26.7, 22.4, 13.9; IR (neat) 2926, 2853, 1703, 1699, 1615, 1456; MS (ES^+) Calculated for $[\text{C}_{15}\text{H}_{24}\text{NaO}]^+$: 243.2 Found: 243.1.

Treatment of enone **15** with base with the intention to epimerize the fused ring carbon center resulted to the conversion of the mixture obtained in the Au(I)-catalyzed reaction completely to the enone where the double bond is at the ring juncture. This is presumably due to the stability of the product and 1,5-hydride shift could account for its formation. The following is the experimental procedure and characterization of the enone: a solution of enone **15** (11 mg, 0.05 mmol) in ^tBuOH (1 mL) was treated with KO^tBu (5 mg) for 4 h at ambient temperature. A yellow color developed. The reaction was quenched with aqueous NH₄Cl. The resulting mixture was extracted with Et₂O (3 x 10 mL). The combined organic layers were washed with H₂O (10 mL), brine (10 mL), dried (MgSO₄) and concentrated. The residue was purified through silica gel flash column chromatography (EtOAc/hexanes = 1/8) to give the 5,7-fused double bond isomer of compound **15** (10 mg, 90%). ¹H NMR (500MHz, CDCl₃) δ 2.65 - 2.60 (m, 1H), 2.50 (dd, 1H, *J* = 6.4, 18.6 Hz), 2.50 - 2.37 (m, 2H), 2.34 - 2.24 (m, 2H), 2.08 (dd, 1H, *J* = 1.5, 18.6 Hz), 1.84 - 1.71 (m, 3H), 1.66 - 1.60 (m, 2H), 1.55 - 1.48 (m, 2H), 1.31 - 1.22 (m, 6H), 1.18 - 1.10 (m, 1H), 0.89 (t, 3H, *J* = 6.7 Hz); ¹³C NMR (125 MHz, CDCl₃) δ 208.2, 180.0, 142.3, 42.6, 40.8, 32.9, 31.9, 31.5, 31.4, 27.0, 26.6, 26.4, 23.2, 22.5, 14.0; IR (neat) 2955, 2925, 2854, 1700, 1645, 1447, 1386; MS (EI) Calculated for [C₁₅H₂₄O]⁺: 220.2 Found: 220.

Compound **17** was prepared in 74% yield according to the general procedure C. However, 5 mol% of AuCl(PPh₃)/AgSbF₆ were used to drive this reaction to completion. ¹H NMR (500MHz, CDCl₃) δ 7.32 (t, 2H, *J* = 7.0 Hz), 7.24 (t, 1H, *J* = 7.0 Hz), 7.14 (d, 2H, *J* = 7.0 Hz), 6.03 (s, 1H), 3.61 (dd, 1H, *J* = 3.0, 7.2 Hz), 3.12 (dd, 1H, *J* = 7.2, 18.5 Hz), 2.69 (d, 1H, *J* = 18.5 Hz), 2.48 (t, 2H, *J* = 7.5 Hz), 1.68 - 1.61 (m, 2H), 1.40 - 1.32 (m, 4H), 0.93 (t, 3H, *J* = 7.0 Hz); ¹³C NMR (125 MHz, CDCl₃) δ 209.1, 182.5, 139.8, 128.8, 128.4, 127.5, 126.8, 52.2, 41.3, 33.5, 31.5, 26.7, 22.4, 14.0; IR (neat): 3064, 3029, 2956, 2929, 2859, 1703, 1699, 1616, 1496, 1454, 1171, 1075, 866; MS (ES⁺) Calculated for [C₁₆H₂₀NaO]⁺: 251.1 Found: 251.0.

To a solution of propargylic acetate **18** (0.09 mmol) in wet CH_2Cl_2 (1.8 mL, 0.05 M) was added $\text{AuCl}(\text{PPh}_3)/\text{AgSbF}_6$ [pregenerated as 0.01 M solution in CH_2Cl_2 by mixing $\text{AuCl}(\text{PPh}_3)$ with AgSbF_6 , 0.45 mL] at room temperature and the reaction mixture was stirred for 30 min. The mixture was concentrated. The residue was dissolved in $\text{THF}/\text{H}_2\text{O}/\text{TFA}$ (3/3/1, 2 mL) and the solution was stirred for 1 h. The reaction mixture was diluted with EtOAc (10 mL) and neutralized with saturated NaHCO_3 (10 mL). Separation of the two layers and the aqueous layer was extracted with EtOAc (3 x 10 mL). The combined organic layers were washed with brine (10 mL), dried (MgSO_4) and concentrated. The residue was purified by silica gel flash column chromatography (hexanes/ethyl acetate = 1/2) to give hydroxyl cyclopentenone **19** (14.3 mg, 81% yield). ^1H NMR (500MHz, CDCl_3) δ 5.93 (s, 1H), 3.84 - 3.72 (m, 2H), 2.89 - 2.82 (m, 2H), 2.56 (ddd, 1H, J = 3.0, 6.7, 14.3 Hz), 2.39 (t, 2H, J = 7.5 Hz), 2.31 (d, 1H, J = 18.5 Hz), 1.93 - 1.86 (m, 1H), 1.74 - 1.68 (m, 1H), 1.58 (p, 2H, J = 7.5 Hz), 1.36 - 1.29 (m, 4H), 0.90 (t, 3H, J = 7.0 Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 212.8, 182.5, 128.4, 61.8, 44.9, 39.1, 34.3, 33.5, 31.5, 26.7, 22.4, 13.9; IR (neat): 3407, 2956, 2930, 2861, 1699, 1613, 1467, 1428; MS (ES^+) Calculated for $[\text{C}_{12}\text{H}_{20}\text{NaO}_2]^+$: 219.1 Found: 218.9.

To a solution of acetate (**20**, 43 mg, 0.2 mmol) in wet CH_2Cl_2 (20.0 mL) was added $\text{AuCl}(\text{PPh}_3)/\text{AgSbF}_6$ [pregenerated as 0.01M solution in CH_2Cl_2 by mixing $\text{AuCl}(\text{PPh}_3)$ with AgSbF_6 , 1.0 mL] at room temperature and the reaction was stirred overnight. The reaction mixture was then concentrated and the residue was purified by silica gel flash column chromatography (hexane/ethyl acetate = 20/1) to give cyclopentadienylic acetate **21** (39 mg, 91% yield). ^1H NMR (500MHz, CD_2Cl_2) δ 7.46 (d, 2H, J = 7.5 Hz), 7.32 (t, 2H, J = 7.5 Hz), 7.20 (t, 1H, J = 7.5 Hz), 6.70 (s, 1H), 3.32 (s, 2H), 2.24 (s, 3H), 1.93 (s, 3H); ^{13}C NMR (125 MHz, CD_2Cl_2) δ 169.2, 147.2, 142.8, 136.2, 129.1, 127.4, 125.0, 125.0, 124.9, 41.9, 21.1, 11.8; IR (neat) 3039, 3013, 2914, 2857, 1753, 1654, 1647, 1445, 1370; MS (ES^+) Calculated for $[\text{C}_{14}\text{H}_{14}\text{NaO}_2]^+$: 237.1 Found: 237.0.

To a solution of compound **21** (21.4 mg, 0.1 mmol) in CH_2Cl_2 was added 0.10 mL solution of Tf_2NH (pregenerated as 0.01 M in CH_2Cl_2) at room temperature and the solution was stirred for 4.0 h. The reaction mixture was concentrated and the residue was purified by silica gel flash column chromatography (hexanes/ethyl acetate = 10/1) to give cyclopentenone **22** (15.5 mg, 90% yield). ^1H NMR (500MHz, CDCl_3) δ 7.66 -7.64 (m, 2H), 7.46 - 7.42 (m, 3H), 6.54 (s, 1H), 3.30 (dd, 1H, J = 7.5, 18.0 Hz), 2.66 - 2.58 (m, 2H), 1.28 (d, 3H, J = 7.0 Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 211.9, 172.0, 134.0, 131.1, 128.9, 126.8, 126.2, 40.7, 37.5, 16.7; IR (neat) 3073, 3030, 2972, 2917, 2871, 1684, 1594, 1571, 1448, 1372; MS (ES^+) Calculated for $[\text{C}_{12}\text{H}_{12}\text{NaO}]^+$: 195.1 Found: 194.9.

To a solution of enynyl acetate **23** (57 mg, 0.2 mmol) in wet CH_2Cl_2 (20.0 mL) was added $\text{AuCl(PPh}_3\text{)/AgSbF}_6$ [pregenerated as 0.01M solution in CH_2Cl_2 by mixing $\text{AuCl(PPh}_3\text{)}$ with AgSbF_6 , 1.0 mL] at room temperature and the reaction was stirred overnight. To the reaction mixture was added 0.20 mL solution of Tf_2NH (pregenerated as 0.01 M in CH_2Cl_2) and the solution was stirred for another 4.0 h. The resulting solution was concentrated and the residue was purified by silica gel flash column chromatography (hexanes/ethyl acetate = 10/1) to give cyclopentenone **24** (46 mg, 96% yield). ^1H NMR (500MHz, CDCl_3) δ 7.75 (d, 2H, J = 8.4 Hz), 7.70 (d, 2H, J = 8.4 Hz), 6.60 (s, 1H), 3.31 (ddd, 1H, J = 2.0, 7.3, 18.4 Hz), 2.67 - 2.62 (m, 2H), 1.29 (d, 3H, J = 7.5 Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 211.4, 169.9, 137.4, 132.6 (q, $J_{\text{C-F}}$ = 32.6 Hz), 128.2, 127.0, 125.9 (q, $J_{\text{C-F}}$ = 4.0 Hz), 123.7 (q, $J_{\text{C-F}}$ = 272.4 Hz), 40.9, 37.5, 16.5; IR (neat) 3077, 2969, 2938, 2875, 1709, 1602, 1573, 1327; MS (ES^+) Calculated for $[\text{C}_{13}\text{H}_{11}\text{F}_3\text{NaO}]^+$: 263.1 Found: 263.1.

To a solution of enynyl acetate **1** (0.332 g, 1.6 mmol) in anhydrous CH_2Cl_2 (32.0 mL, 0.05 M) was added $\text{AuCl(PPh}_3\text{)/AgSbF}_6$ [pregenerated as 0.01 M solution in dry CH_2Cl_2 by mixing $\text{AuCl(PPh}_3\text{)}$ with AgSbF_6 , 1.6 mL] at room temperature and the reaction mixture was stirred for 30 min. The mixture was treated with Et_3N (0.05 mL) and concentrated. The residue was passed quickly through a silica gel column (CH_2Cl_2 /pentane = 2/1) to give cyclopentadienylic acetate **2** (0.215 g,

65% yield). ^1H NMR (500MHz, CD_2Cl_2) δ 5.92 (s, 1H), 2.80 (s, 2H), 2.30 (t, 2H, $J = 7.0$ Hz), 2.17 (s, 3H), 1.80 (s, 3H), 1.52 - 1.46 (m, 2H), 1.34 - 1.26 (m, 4H), 0.89 (t, 3H, $J = 7.0$ Hz); ^{13}C NMR (125 MHz, CD_2Cl_2) δ 169.3, 146.9, 146.2, 123.9, 122.0, 44.0, 32.2, 31.4, 29.5, 23.1, 21.1, 14.4, 11.5; IR (neat) 2957, 2930, 2873, 2860, 1758, 1654, 1369; MS (ES^+) Calculated for $[\text{C}_{13}\text{H}_{20}\text{NaO}_2]^+$: 231.1; Found: 230.9.

References:

- (1) Stang, P. J.; Treptow, W. *Synthesis* 283-284.
- (2) Generated by shaking distilled CH_2Cl_2 with deionized water in a separatory funnel.

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Temp. 25.0 C / 298.1 K

UNITYplus-500 "ernst"

PULSE SEQUENCE

Pulse 70.7 degrees

Acq. time 1.999 sec

Width 7476.6 Hz

Single scan

OBSERVE H1, 499.8006811 MHz

DATA PROCESSING

Line broadening 1.0 Hz

FT size 32768

Total time 0 min, 2 sec

¹³C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
UNITYplus-500 "ernst"

PULSE SEQUENCE

Relax. delay 0.100 sec
Pulse 41.5 degrees
Acq. time 2.187 sec
Width 29261.2 Hz
672 repetitions

OBSERVE C13, 125.6749801 MHz

DECOUPLE H1, 499.8037245 MHz

Power 36 dB

on during acquisition

off during delay

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 31 hr, 55 min, 2 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pu1

Solvent: CDCl3

Temp. 25.0 C / 298.1 K

UNITYplus-500 "ernst"

PULSE SEQUENCE

Relax. delay 3.000 sec

Pulse 70.7 degrees

Acq. time 1.999 sec

Width 7476.6 Hz

32 repetitions

OBSERVE H1, 499.8016480 MHz

DATA PROCESSING

Line broadening 1.0 Hz

FT size 32768

Total time 4 min, 16 sec

13C OBSERVE

Pulse Sequence: s2pu1

Solvent: CDC13

Temp. 25.0 C / 298.1 K

UNITYplus-500 "ernst"

PULSE SEQUENCE

Relax. delay 0.100 sec

Pulse 41.5 degrees

Acq. time 2.187 sec

Width 29261.2 Hz

992 repetitions

OBSERVE C13, 125.6751452 MHz

DECOUPLE H1, 499.8037245 MHz

Power 36 dB

Power 30 dB
on during acquisition

off during delay

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 31 hr, 55 min, 2 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pu1

Solvent: CDC13

Temp. 25.0 C / 298.1 K

UNITYplus-500 "ernst"

PULSE SEQUENCE

Pulse 70.7 degrees

Acq. time 1.999 sec

Width 7476.6 Hz

32 repetitions

OBSERVE H1, 499.8006811 MHz

DATA PROCESSING

Line broadening 1.0 Hz

FT size 32768

Total time 1 min, 4 sec

¹³C OBSERVE

Pulse Sequence: s2pu1

Solvent: CDC13

Temp. 25.0 C / 298.1 K

UNITYplus-500 "ernst"

PULSE SEQUENCE

Relax. delay 0.100 sec

Pulse 41.5 degrees

Acq. time 2.187 sec

Width 29261.2 Hz

1776 repetitions

OBSERVE C13, 125.6749815 MHz

DECOUPLE H1, 499.8037245 MHz

Power 36 dB

on during acquisition

off during delay

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 31 hr, 55 min, 2 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pu1

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
UNITYplus-500 "ernst"

PULSE SEQUENCE

Pulse 70.7 degrees
Acq. time 1.999 sec
Width 7476.6 Hz

Single scan
OBSERVE H1, 499.8006816 MHz
DATA PROCESSING

Line broadening 1.0 Hz
FT size 32768

Total time 0 min, 2 sec

¹³C OBSERVE

Pulse Sequence: s2pt1
Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
File: 1
UNITYplus-500 "ernst"
PULSE SEQUENCE
Relax. delay 0.100 sec
Pulse 41.5 degrees
Acq. time 2.187 sec
Width 29261.2 Hz
1648 repetitions
OBSERVE C13, 125.6749806 MHz
DECOUPLE H1, 499.8037245 MHz
Power 36 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 31 hr, 55 min, 2 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pu1

Solvent: CDCl3

Temp. 25.0 C / 298.1 K

UNITYplus-500 "ernst"

PULSE SEQUENCE

Pulse 70.7 degrees

Acq. time 1.999 sec

Width 7476.6 Hz

32 repetitions

OBSERVE H1, 499.8006811 MHz

DATA PROCESSING

Line broadening 1.0 Hz

FT size 32768

Total time 1 min, 4 sec

¹³C OBSERVE

Pulse Sequence: s2pu1

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

UNITYplus-500 "ernst"

PULSE SEQUENCE

Relax. delay 0.100 sec

Pulse 41.5 degrees

Acq. time 2.187 sec

Width 29261.2 Hz

192 repetitions

OBSERVE C13, 125.6749833 MHz

DECOUPLE H1, 499.8037245 MHz

Power 36 dB

on during acquisition

off during delay

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 31 hr, 55 min, 2 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Temp. 25.0 C / 298.1 K

UNITYplus-500 "ernst"

PULSE SEQUENCE

Pulse 70.7 degrees

Acq. time 1.999 sec

Width 7476.6 Hz

32 repetitions

OBSERVE H1, 499.8006811 MHz

DATA PROCESSING

Line broadening 1.0 Hz

FT size 32768

Total time 1 min, 4 sec

13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
UNITYplus-500 "ernst"

PULSE SEQUENCE

Relax. delay 0.100 sec

Pulse 41.5 degrees

Acq. time 2.187 sec

Width 29261.2 Hz

352 repetitions

OBSERVE C13, 125.6749801 MHz

DECOUPLE H1, 499.8037245 MHz

Power 36 dB

on during acquisition

off during delay

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 31 hr, 55 min, 2 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pu1

Solvent: CDCl3

Temp. 25.0 C / 298.1 K

UNITYplus-500 "ernst"

PULSE SEQUENCE

Pulse 70.7 degrees

Acq. time 1.999 sec

Width 7476.6 Hz

64 repetitions

OBSERVE H1, 499.8006811 MHz

DATA PROCESSING

Line broadening 1.0 Hz

FT size 32768

Total time 2 min, 8 sec

¹³C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
UNITYplus-500 "ernst"

PULSE SEQUENCE

Relax. delay 0.100 sec

Pulse 41.5 degrees

Acq. time 2.187 sec

Width 29261.2 Hz

1824 repetitions

OBSERVE C13, 125.6749842 MHz

DECOUPLE H1, 499.8037245 MHz

Power 36 dB

on during acquisition

off during delay

WALTZ-16 modulated

DATA PROCESSING

Line broadening 2.0 Hz

FT size 131072

Total time 31 hr, 55 min, 2 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDC13

Temp. 25.0 C / 298.1 K

UNITYplus-500 "ernst"

PULSE SEQUENCE

Pulse 67.5 degrees

Acq. time 1.999 sec

Width 7476.6 Hz

32 repetitions

OBSERVE H1, 499.8006802 MHz

DATA PROCESSING

Line broadening 1.0 Hz

FT size 32768

Total time 1 min, 4 sec

¹³C OBSERVE

Pulse Sequence: s2pu1

Solvent: CDC13
Temp. 25.0 C / 298.1 K
UNITYplus-500 "ernst"

PULSE SEQUENCE

Relax. delay 0.100 sec
Pulse 41.5 degrees
Acq. time 1.600 sec
Width 40000.0 Hz
6480 repetitions

OBSERVE C13, 125.6749827 MHz

DECOUPLE H1, 499.8037245 MHz

Power 36 dB

on during acquisition

off during delay

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 23 hr, 45 min, 42 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pu1

Solvent: CDC13

Temp. 25.0 C / 298.1 K

UNITYplus-500 "ernst"

PULSE SEQUENCE

Relax. delay 5.000 sec

Pulse 67.5 degrees

Acq. time 1.999 sec

Width 7476.6 Hz

16 repetitions

OBSERVE H1, 499.8006807 MHz

DATA PROCESSING

Line broadening 1.0 Hz

FT size 32768

Total time 3 min, 44 sec

¹³C OBSERVE

Pulse Sequence: s2pu1

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

UNITYplus-500 "ernst"

PULSE SEQUENCE

Relax. delay 0.100 sec

Pulse 41.5 degrees

Acq. time 2.187 sec

Width 29261.2 Hz

320 repetitions

OBSERVE C13, 125.6749868 MHz

DECOUPLE H1, 499.8037245 MHz

Power 36 dB

on during acquisition

off during delay

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 31 hr, 55 min, 2 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDC13

Temp. 25.0 C / 298.1 K

UNITYplus-500 "ernst"

PULSE SEQUENCE

Pulse 70.7 degrees

Acq. time 1.999 sec

Width 7476.6 Hz

32 repetitions

OBSERVE H1, 499.8006811 MHz

DATA PROCESSING

Line broadening 1.0 Hz

FT size 32768

Total time 1 min, 4 sec

13C OBSERVE

Pulse Sequence: s2pu1

Solvent: CDC13

Temp. 25.0 C / 298.1 K

UNITYplus-500 "ernst"

PULSE SEQUENCE

Relax. delay 0.100 sec

Pulse 41.5 degrees

Acq. time 2.187 sec

Width 29261.2 Hz

320 repetitions

OBSERVE C13, 125.6749801 MHz

DECOUPLE H1, 499.8037245 MHz

Power 36 dB

on during acquisition

off during delay

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 31 hr, 55 min, 2 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pu1

Solvent: CDC13

Temp. 25.0 C / 298.1 K

UNITYplus-500 "ernst"

PULSE SEQUENCE

Pulse 70.7 degrees

Acq. time 1.999 sec

Width 7476.6 Hz

32 repetitions

OBSERVE H1, 499.6006811 MHz

DATA PROCESSING

Line broadening 1.0 Hz

FT size 32768

Total time 1 min, 4 sec

13C OBSERVE

Pulse Sequence: s2pu1

Solvent: CDC13

Temp. 25.0 C / 298.1 K

UNITYplus-500 "ernst"

PULSE SEQUENCE

Relax. delay 0.100 sec

Pulse 41.5 degrees

Acq. time 2.187 sec

Width 29261.2 Hz

6944 repetitions

OBSERVE C13, 125.6749833 MHz

DECOUPLE H1, 499.8037245 MHz

Power 36 dB

on during acquisition

off during delay

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 76 hr, 36 min, 5 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pu1

Solvent: CDC13

Temp. 25.0 C / 298.1 K

UNITYplus-500 "ernst"

PULSE SEQUENCE

Pulse 70.7 degrees

Acq. time 1.999 sec

Width 7476.6 Hz

32 repetitions

OBSERVE H1, 499.8006811 MHz

DATA PROCESSING

Line broadening 1.0 Hz

FT size 32768

Total time 1 min, 4 sec

¹³C OBSERVE

Pulse Sequence: s2pu1

Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
UNITYplus-500 "ernst"

PULSE SEQUENCE

Relax. delay 0.100 sec
Pulse 41.5 degrees
Acq. time 2.187 sec
Width 29261.2 Hz
176 repetitions

OBSERVE C13, 125.6749801 MHz

DECOUPLE H1, 499.8037245 MHz

Power 36 dB

on during acquisition

off during delay

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 31 hr, 55 min, 2 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Temp. 25.0 C / 298.1 K

UNITYplus-500 "ernst"

PULSE SEQUENCE

Pulse 70.7 degrees

Acq. time 1.999 sec

Width 7476.6 Hz

32 repetitions

OBSERVE H1, 499.8006816 MHz

DATA PROCESSING

Line broadening 1.0 Hz

FT size 32768

Total time 1 min, 4 sec

¹³C OBSERVE

Pulse Sequence: s2pu1

Solvent: CDC13

Temp. 25.0 C / 298.1 K

UNITYplus-500 "ernst"

PULSE SEQUENCE

Relax. delay 0.100 sec

Pulse 41.5 degrees

Acq. time 2.187 sec

Width 29261.2 Hz

112 repetitions

OBSERVE C13, 125.6749855 MHz

DECOUPLE H1, 499.8037245 MHz

Power 36 dB

on during acquisition

off during delay

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 31 hr, 55 min, 2 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pu1

Solvent: CDC13

Temp. 25.0 C / 298.1 K

UNITYplus-500 "ernst"

PULSE SEQUENCE

Pulse 70.7 degrees

Acq. time 1.999 sec

Width 7476.6 Hz

32 repetitions

OBSERVE H1, 499.8006811 MHz

DATA PROCESSING

Line broadening 1.0 Hz

FT size 32768

Total time 1 min, 4 sec

13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
UNITYplus-500 "ernst"

PULSE SEQUENCE

Relax. delay 0.100 sec

Pulse 41.5 degrees

Acq. time 2.187 sec

Width 29261.2 Hz

256 repetitions

OBSERVE C13, 125.6749801 MHz

DECOUPLE H1, 499.8037245 MHz

Power 36 dB

on during acquisition

off during delay

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 31 hr, 55 min, 2 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pu1

Solvent: CDC13
Temp. 25.0 C / 298.1 K
UNITYplus-500 "ernst"

PULSE SEQUENCE

Pulse 70.7 degrees

Acq. time 1.999 sec

Width 7476.6 Hz

32 repetitions

OBSERVE H1, 499.8006816 MHz

DATA PROCESSING

Line broadening 1.0 Hz

FT size 32768

Total time 1 min, 4 sec

¹³C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

UNITYplus-500 "ernst"

PULSE SEQUENCE

Relax. delay 0.100 sec

Pulse 41.5 degrees

Acq. time 2.187 sec

Width 29261.2 Hz

2288 repetitions

OBSERVE C13, 125.6749810 MHz

DECOUPLE H1, 499.8037245 MHz

Power 36 dB

on during acquisition

off during delay

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 31 hr, 55 min, 2 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Temp. 25.0 C / 298.1 K

File: PB-cyclopentenone-hexanal-heptane-H1

UNITYplus-500 "ernst"

PULSE SEQUENCE

Pulse 22.5 degrees

Acq. time 2.000 sec

Width 10000.0 Hz

32 repetitions

OBSERVE H1, 499.8006812 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 1 min, 4 sec

13C OBSERVE

Pulse Sequence: s2pu1

Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
UNITYplus-500 "ernst"

PULSE SEQUENCE

Relax. delay 0.100 sec
Pulse 41.5 degrees
Acq. time 2.187 sec
Width 29261.2 Hz
1520 repetitions

OBSERVE C13, 125.6749801 MHz
DECOUPLE H1, 499.8037245 MHz
Power 36 dB

on during acquisition
off during delay
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz
FT size 131072
Total time 31 hr, 55 min, 2 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pu1

Solvent: CDCl3

Temp. 25.0 C / 298.1 K

File: PB-hexanal-phenylalkynol-acetate

UNITYplus-500 "ernst"

PULSE SEQUENCE

Pulse 22.5 degrees

Acq. time 1.999 sec

Width 7476.6 Hz

32 repetitions

OBSERVE H1, 499.8006807 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 32768

Total time 1 min, 4 sec

¹³C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

File: PB-hexanal-phenylalkynol-acetate-C13
UNITYplus-500 "ernst"

PULSE SEQUENCE

Relax. delay 0.100 sec

Pulse 41.5 degrees

Acq. time 2.187 sec

Width 29261.2 Hz

50000 repetitions

OBSERVE C13, 125.6749824 MHz

DECOUPLE H1, 499.8037245 MHz

Power 36 dB

on during acquisition

off during delay

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 31 hr, 55 min, 2 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pu1

Solvent: CDCl3

Temp. 25.0 C / 298.1 K

UNITYplus-500 "ernst"

PULSE SEQUENCE

Pulse 70.7 degrees

Acq. time 1.999 sec

Width 7476.6 Hz

32 repetitions

OBSERVE H1, 499.8006811 MHz

DATA PROCESSING

Line broadening 1.0 Hz

FT size 32768

Total time 1 min, 4 sec

¹³C OBSERVE

Pulse Sequence: s2pu1

Solvent: CDC13
Temp. 25.0 C / 298.1 K
UNITYplus-500 "ernst"

PULSE SEQUENCE
Relax. delay 0.100 sec
Pulse 41.5 degrees
Acq. time 2.187 sec
Width 29261.2 Hz
128 repetitions

OBSERVE C13, 125.6749873 MHz
DECOUPLE H1, 499.8037245 MHz

Power 36 dB
on during acquisition
off during delay
WALTZ-16 modulated

DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072

Total time 31 hr, 55 min, 2 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pu1

Solvent: CDC13

Temp. 25.0 C / 298.1 K

UNITYplus-500 "ernst"

PULSE SEQUENCE

Pulse 70.7 degrees

Acq. time 1.999 sec

Width 7476.6 Hz

32 repetitions

OBSERVE H1, 499.8006816 MHz

DATA PROCESSING

Line broadening 1.0 Hz

FT size 32768

Total time 1 min, 4 sec

13C OBSERVE

Pulse Sequence: s2pu1

Solvent: CDC13

Temp. 25.0 C / 298.1 K

UNITYplus-500 "ernst"

PULSE SEQUENCE

Relax. delay 0.100 sec

Pulse 41.5 degrees

Acq. time 2.187 sec

Width 29261.2 Hz

336 repetitions

OBSERVE C13, 125.6749828 MHz

DECOUPLE H1, 499.8037245 MHz

Power 36 dB

on during acquisition

off during delay

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 31 hr, 55 min, 2 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pu1

Solvent: CDC13
Temp. 25.0 C / 298.1 K
UNITYplus-500 "ernst"

PULSE SEQUENCE

Pulse 22.5 degrees
Acq. time 1.999 sec
Width 7476.6 Hz
32 repetitions
OBSERVE H1, 499.8006816 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 32768
Total time 1 min, 4 sec

13C OBSERVE

Pulse Sequence: s2pu1

Solvent: CDC13
Temp. 25.0 C / 298.1 K
UNITYplus-500 "ernst"

PULSE SEQUENCE

Relax. delay 0.100 sec

Pulse 41.5 degrees

Acq. time 2.187 sec

Width 29261.2 Hz

2064 repetitions

OBSERVE C13, 125.6749810 MHz

DECOUPLE H1, 499.8037245 MHz

Power 36 dB

on during acquisition

off during delay

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 31 hr, 55 min, 2 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pu1

Solvent: CDC13

Temp. 25.0 C / 298.1 K

UNITYplus-500 "ernst"

PULSE SEQUENCE

Pulse 70.7 degrees

Acq. time 1.999 sec

Width 7476.6 Hz

32 repetitions

OBSERVE H1, 499.8006811 MHz

DATA PROCESSING

Line broadening 1.0 Hz

FT size 32768

Total time 1 min, 4 sec

13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDC13

Temp. 25.0 C / 298.1 K

UNITYplus-500 "ernst"

PULSE SEQUENCE

Relax. delay 0.100 sec

Pulse 41.5 degrees

Acq. time 2.187 sec

Width 29261.2 Hz

528 repetitions

OBSERVE C13, 125.6749806 MHz

DECOUPLE H1, 499.8037245 MHz

Power 36 dB

on during acquisition

off during delay

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 31 hr, 55 min, 2 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDC13
Temp. 25.0 C / 298.1 K
UNITYplus-500 "ernst"

PULSE SEQUENCE

Pulse 70.7 degrees
Acq. time 1.999 sec
Width 7476.6 Hz
Single scan

OBSERVE H1, 499.8016480 MHz

DATA PROCESSING

Line broadening 1.0 Hz
FT size 32768
Total time 0 min, 2 sec

¹³C OBSERVE

Pulse Sequence: s2pu1

Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
UNITYplus-500 "ernst"

PULSE SEQUENCE

Relax. delay 0.100 sec
Pulse 41.5 degrees
Acq. time 2.187 sec
Width 29261.2 Hz
400 repetitions

OBSERVE C13, 125.6751479 MHz

DECOUPLE H1, 499.8037245 MHz

Power 36 dB

on during acquisition

off during delay

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 31 hr, 55 min, 2 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pu1

Solvent: CDC13
Temp. 25.0 C / 298.1 K
UNITYplus-500 "ernst"

PULSE SEQUENCE

Pulse 70.7 degrees
Acq. time 1.999 sec
Width 7476.6 Hz
Single scan

OBSERVE H1, 499.8006802 MHz

DATA PROCESSING

Line broadening 1.0 Hz
FT size 32768

Total time 0 min, 2 sec

¹³C OBSERVE

Pulse Sequence: s2pul

Solvent: CDC13

Temp. 25.0 C / 298.1 K

UNITYplus-500 "ernst"

PULSE SEQUENCE

Relax. delay 0.100 sec

Pulse 41.5 degrees

Acq. time 2.187 sec

Width 29261.2 Hz

240 repetitions

OBSERVE C13, 125.6749824 MHz

DECOUPLE H1, 499.8037245 MHz

Power 36 dB

on during acquisition

off during delay

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 31 hr, 55 min, 2 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pu1

Solvent: CDCl3

Temp. 25.0 C / 298.1 K

UNITYplus-500 -"ernst"

PULSE SEQUENCE

Pulse 70.7 degrees

Acq. time 1.999 sec

Width 7476.6 Hz

Single scan

OBSERVE H1, 499.8006807 MHz

DATA PROCESSING

Line broadening 1.0 Hz

FT size 32768

Total time 0 min, 2 sec

¹³C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

File: CF3benzaldehyde-enynol-acetate-C13

UNITYplus-500 "ernst"

PULSE SEQUENCE

Relax. delay 0.100 sec

Pulse 41.5 degrees

Acq. time 2.187 sec

Width 29261.2 Hz

14624 repetitions

OBSERVE C13, 125.6749797 MHz

DECOUPLE H1, 499.8037245 MHz

Power 36 dB

on during acquisition

off during delay

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 31 hr, 55 min, 2 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pu1

Solvent: CDCl3

Temp. 25.0 C / 298.1 K

UNITYplus-500 "ernst"

PULSE SEQUENCE

Pulse 70.7 degrees

Acq. time 1.999 sec

Width 7476.6 Hz

Single scan

OBSERVE H1, 499.8006807 MHz

DATA PROCESSING

Line broadening 1.0 Hz

FT size 32768

Total time 0 min, 2 sec

13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDC13

Temp. 25.0 C / 298.1 K

UNITYplus-500 "ernst"

PULSE SEQUENCE

Relax. delay 0.100 sec

Pulse 41.5 degrees

Acq. time 2.187 sec

Width 29261.2 Hz

14816 repetitions

OBSERVE C13, 125.6749815 MHz

DECOUPLE H1, 499.8037245 MHz

Power 36 dB

on during acquisition

off during delay

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 31 hr, 55 min, 2 sec