Room-Temperature Stille Cross-Couplings of Alkenyltin Reagents and Functionalized Alkyl Bromides that Possess β Hydrogens

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Supporting Information

I. General

$[(\pi\text{-allyl})\text{PdCl}]_2$ (Strem, Johnson Matthey), $\text{N}(i\text{-Pr})_2\text{Et}$ (Aldrich), $\text{CsOH}\cdot\text{H}_2\text{O}$ (Aldrich), NaOMe (Aldrich), KF (Aldrich), CsF (Aldrich), $\text{Bu}_4\text{NF}\cdot 3\text{H}_2\text{O}$ (Aldrich), anhydrous Me_4NF (Aldrich), $\text{Me}_4\text{NF}\cdot 4\text{H}_2\text{O}$ (Aldrich, Alfa Aesar; can be employed if dried under vacuum at 120 °C for 2 days), anhydrous 3 Å molecular sieves (Aldrich), PCy_3 (Strem), $\text{P}(t\text{-Bu})_3$ (Strem), $\text{P}(t\text{-Bu})_2\text{Me}$ (Strem), $\text{P}(\text{OPh})_3$ (Aldrich), PPh_3 (Aldrich), $\text{P}(o\text{-tol})_3$ (Strem), $\text{P}(2\text{-furyl})_3$ (Strem), AsPh_3 (Strem), IMesHCl (Strem), $[\text{HP}(t\text{-Bu})_2\text{Me}]\text{BF}_4$ (Strem), and tributyl(vinyl)tin (Aldrich, Alfa Aesar) were used as received. 1-Bromodecane (Avocado), benzyloxy-4-bromobutane (Aldrich), 5-bromo-1-pentene (Avocado), 6-bromohexanonitrile (Avocado), 2-(2-bromoethyl)-1,3-dioxolane (Aldrich), ethyl 6-bromohexanoate (Aldrich), 3-phenylpropyl bromide (Aldrich) were sparged with argon before use (if a liquid). All other compounds were prepared according to literature procedures. THF was purified by passing it through a neutral alumina column under argon, and then it was subjected to several freeze-pump-thaw cycles.

II. Preparation of Substrates

Synthesis of (E) organotin reagents. Sample procedure. A solution of LiEt_3BH (1.0 M in THF; 12 mL, 12 mmol) was added dropwise to a solution of Cp_2ZrCl_2 (3.5 g, 12 mmol) in THF (50 mL). The reaction flask was covered with aluminum foil, and the mixture was stirred for 1 h at room temperature. To the resulting white suspension was added dropwise 2-(pent-4-ynyloxy)tetrahydropyran (1.05 g, 6.03 mmol). After stirring this reaction mixture for 1 h at room temperature, *N*-iodosuccinimide (3.0 g, 13.3 mmol) was added. After stirring for 5 min, the reaction mixture was poured into 20 mL of saturated aq NaHCO_3 . The phases were separated, and the aqueous layer was extracted three times with Et_2O (100 mL). The combined organic layers were dried, filtered, and concentrated. Flash chromatography (pentane/ Et_2O 5: 1, R_f = 0.60) furnished 1.78 g (5.80 mmol, 95%) of (*E*)-2-(5-iodopent-4-enyloxy)tetrahydropyran as a colorless liquid. The product was stored in a refrigerator.

Chromatography: Pentane/ Et_2O 3:1, colorless liquid, R_f = 0.60.

^1H NMR (CDCl_3 , 400 MHz): 6.52 (td, J = 7.2, 14.3 Hz, 1H), 6.01 (td, J = 1.4, 14.3 Hz, 1H), 4.56-4.54 (m, 1H), 3.97-3.81 (m, 1H), 3.74 (td, J = 6.4, 9.7 Hz, 1H), 3.52-3.47 (m, 1H), 3.37 (td, J = 6.3, 9.7 Hz, 1H), 2.18-2.12 (m, 2H), 1.84-1.79 (m, 1H), 1.72-1.65 (m, 3H), 1.59-1.49 (m, 4H).

^{13}C NMR (CDCl_3 , 100 MHz): 145.9, 98.8, 74.9, 66.4, 62.3, 32.8, 30.6, 28.4, 25.4, 19.6.

HRMS calc. for $\text{C}_{10}\text{H}_{17}\text{IO}_2\text{Na}$ [$\text{M}+\text{Na}$]: 319.0165, found: 319.0165.

IR: 2940, 2868, 1440, 1353, 1201, 1136, 1076, 1034.

A solution of *t*-BuLi (1.50 M in pentane; 27.2 mL, 40.7 mmol) was added over 1.5 h to a $-78\text{ }^{\circ}\text{C}$ solution of (*E*)-2-(5-iodopent-4-enyloxy)tetrahydropyran (5.80 g, 19.4 mmol) in Et₂O (60 mL) and pentane (150 mL). After 5 min, ClSnBu₃ (6.60 mL, 24.0 mmol) was added slowly at $-78\text{ }^{\circ}\text{C}$. The reaction mixture was stirred for 6 h at $-78\text{ }^{\circ}\text{C}$, and then it was allowed to gradually warm to room temperature overnight. A saturated aq NH₄Cl solution (30 mL) was then added, and the resulting phases were separated. The aqueous layer was extracted three times with Et₂O (60 mL), and the combined organic layers were dried, filtered, and concentrated. Flash chromatography (pentane/Et₂O 10:1, *R_f* = 0.50) furnished 5.78 g (12.6 mmol, 65%) of (*E*)-tributyl[5-(tetrahydropyran-2-yloxy)pent-1-enyl]stannane as a colorless liquid.

Chromatography: Pentane/Et₂O: 10:1, colorless liquid, *R_f* = 0.50.

¹H NMR (CDCl₃, 400 MHz): 5.99-5.87 (m, 2H), 4.58-4.57 (m, 1H), 3.89-3.84 (m, 1H), 3.74 (td, *J* = 6.6, 9.6 Hz, 1H), 3.52-3.46 (m, 1H), 3.38 (td, *J* = 6.7, 9.6 Hz, 1H), 2.24-2.19 (m, 2H), 1.88-1.78 (m, 1H), 1.73-1.69 (m, 3H), 1.59-1.44 (m, 10H), 1.29 (sext, *J* = 7.6 Hz, 6H), 0.90-0.83 (m, 15H).

¹³C NMR (CDCl₃, 100 MHz): 148.7, 127.7, 98.8, 67.0, 62.4, 34.4, 30.7, 29.1 (3x, multiplet, due to coupling to tin), 28.9, 27.4 (3x), 25.5, 19.6, 13.7 (3x), 9.3 (3x).

HRMS calc. for C₂₂H₄₄O₂SnNa [*M*+Na]: 483.2251, found: 483.2255.

IR: 2955, 2925, 2871, 1599, 1464, 1137, 1077, 1035.

Synthesis of (Z) organotin reagent. A solution of NaHMDS (1.0 M in THF; 29.9 mL, 29.9 mmol) was added to a suspension of iodomethylphosphonium iodide (15.8 g, 29.9 mmol) in THF (100 mL) at room temperature. The reaction mixture was cooled to -60°C , and then HMPA (420 μL , 2.40 mmol) was added. The reaction mixture was cooled to -78°C , and then hydrocinnamaldehyde (3.50 mL, 23.9 mmol) was added. After stirring at -78°C for 30 min, the reaction mixture was allowed to warm to r.t. over one hour. The suspension was then poured into brine (50 mL), and the phases were separated. The aqueous layer was extracted three times with pentane (60 mL), and the combined organic layers were dried, filtered, and concentrated. Flash chromatography (2% Et₂O in pentane, $R_f = 0.90$) furnished 5.05 g (19.6 mmol, 82%) of (Z)-(4-iodobut-3-enyl)benzene as a colorless liquid. The corresponding organotin reagent was then prepared, as described in the previous procedure.

Chromatography: Pentane, colorless liquid, $R_f = 0.88$.

¹H NMR (CDCl₃, 400 MHz): 7.32-7.28 (m, 2H), 7.22-7.19 (m, 3H), 6.57 (td, $J = 7.0, 12.4$ Hz, 1H), 5.85 (br d, $J = 12.4$ Hz, 1H), 2.70 (dd, $J = 7.5, 9.8$ Hz, 2H), 2.38-2.32 (m, 2H), 1.51-1.46 (m, 6H), 1.31 (sext, $J = 7.3$ Hz, 6H), 0.92-0.85 (m, 15H).

¹³C NMR (CDCl₃, 100 MHz): 148.0, 141.9, 128.8, 128.4 (2x), 128.3 (2x), 125.8, 39.0, 36.3, 29.2 (3x, multiplet, due to coupling to tin), 27.3 (3x), 13.7 (3x), 10.2 (3x).

HRMS calc. for C₂₂H₃₈SnNa [M+Na]: 445.1888, found: 445.1867.

IR: 2957, 2925, 2871, 2853, 1598, 1496, 1376, 1074, 697.

Chromatography: Pentane/Et₂O 10:1, colorless liquid, R_f = 0.36.

¹H NMR (CDCl₃, 400 MHz): 5.99-5.88 (m, 2H), 2.15-2.10 (m, 2H), 1.53-1.45 (m, 6H), 1.35-1.26 (m, 16H), 0.92-0.85 (m, 18H).

¹³C NMR (CDCl₃, 100 MHz): 149.9, 126.9, 37.9, 31.9, 29.2, 29.1 (3x), 29.0, 28.9, 27.3 (3x), 22.7, 14.1, 13.7 (3x), 9.4 (3x).

HRMS calc. for C₂₁H₄₄SnNa [M+Na]: 439.2357, found: 439.2360.

IR: 2957, 2926, 2855, 2872.

Chromatography: Pentane/Et₂O 3:1, colorless liquid, R_f = 0.60.

¹H NMR (CDCl₃, 400 MHz): 5.90-5.89 (m, 1H), 4.55-4.53 (m, 1H), 3.86-3.81 (m, 1H), 3.70 (td, J = 6.5, 9.7 Hz, 1H), 3.51-3.50 (m, 1H), 3.34 (td, J = 6.4, 9.7 Hz, 1H), 2.29 (ddt, J = 0.9, 4.0, 7.8 Hz, 2H), 1.83 (s, 3H), 1.72-1.68 (m, 4H), 1.58-1.49 (m, 4H).

¹³C NMR (CDCl₃, 100 MHz): 147.5, 98.9, 74.8, 66.6, 62.3, 36.2, 30.7, 27.7, 25.4, 23.8, 19.6.

HRMS calc. for C₁₁H₁₉IO₂Na [M+Na]: 333.0322, found: 333.0314.

IR: 2941, 2870, 1441, 1353, 1269, 1200, 1137, 1121, 1076, 1035.

Chromatography: Pentane/Et₂O: 10:1, colorless liquid, R_f = 0.73.

^1H NMR (CDCl_3 , 400 MHz): 5.46 (s, 1H), 4.57-4.56 (m, 1H), 3.89-3.84 (m, 1H), 3.76-3.70 (m, 1H), 3.51-3.46 (m, 1H), 3.41-3.35 (m, 1H), 2.22-2.17 (m, 2H), 1.79-1.70 (m, 7H), 1.59-1.45 (m, 10H), 1.30 (sext, $J = 7.3$ Hz, 6H), 0.90-0.85 (m, 15H).

^{13}C NMR (CDCl_3 , 100 MHz): 154.3, 121.7, 98.9, 67.2, 62.3, 38.4, 30.7, 29.2 (3x), 28.2, 27.3 (3x, multiplet, due to coupling to tin), 25.5, 24.5, 19.7, 13.7 (3x), 10.0 (3x).

HRMS calc. for $\text{C}_{23}\text{H}_{46}\text{O}_2\text{SnNa}$ [$\text{M}+\text{Na}$]: 497.2412, found: 497.2413.

IR: 2955, 2923, 2871, 2853, 1607, 1455, 1442, 1376, 1353, 1138, 1121, 1076, 1035, 1023.

III. Stille Reactions

General procedure. In a glove box, either $P(t\text{-Bu})_2\text{Me}$ (0.15 equiv) or $[\text{HP}(t\text{-Bu})_2\text{Me}]\text{BF}_4$ (0.15 equiv), and then anhydrous Me_4NF (1.9 equiv) were added in turn to a vial that contained a yellow solution of $[(\pi\text{-allyl})\text{PdCl}]_2$ (0.025 equiv) in THF (0.10 M). The resulting heterogeneous reaction mixture was stirred for 5 minutes, and then the alkenyltin reagent (1.1 equiv), activated powdered 3Å molecular sieves (500 mg/mmol), and the alkyl bromide (1.0 equiv) were added. The vial was closed with a Teflon-lined cap, removed from the glove box, and the reaction mixture was stirred vigorously for 24 hours at room temperature. Then, aq 10% KF (same volume as the volume of THF) was added. After 30 minutes of stirring, the phases were separated, and the aqueous phase was extracted three times with Et_2O . The combined organic phases were dried and filtered. The solvent was then evaporated, and the residue was purified by flash chromatography.

Table 3, entry 1. Chromatography: Pentane, colorless liquid, $R_f = 0.89$.

^1H NMR (CDCl_3 , 400 MHz): 5.81 (tdd, $J = 6.7, 10.2, 16.9$ Hz, 1H), 5.45-5.34 (m, 2H), 5.00 (qd, $J = 1.8, 17.1$ Hz, 1H), 4.96-4.93 (m, 2H), 2.08-1.95 (m, 5H), 1.45 (d, $J = 7.4$ Hz, 2H), 1.43-1.27 (m, 10H), 0.89 (t, $J = 6.7$ Hz, 3H).

^{13}C NMR (CDCl_3 , 100 MHz): 138.9, 130.8, 129.8, 114.3, 33.2, 32.6, 32.0, 31.9, 29.6, 29.2, 29.1, 28.8, 22.7, 14.1.

HRMS calc. for $\text{C}_{14}\text{H}_{26}$ [M]: 194.2029, found: 194.2031.

IR: 2957, 2926, 2855.

Table 3, entry 2. Chromatography: Pentane/Et₂O 10:1, colorless liquid, R_f = 0.13.

¹H NMR (CDCl₃, 400 MHz): 5.45-5.42 (m, 2H), 4.84 (t, J = 4.8 Hz, 1H), 4.56-4.54 (m, 1H), 3.96-3.79 (m, 5H), 3.71 (td, J = 6.8, 9.6 Hz, 1H), 3.50-3.45 (m, 1H), 3.36 (td, J = 6.6, 9.6 Hz, 1H), 2.13-2.03 (m, 4H), 1.84-1.77 (m, 1H), 1.71-1.62 (m, 5H), 1.57-1.48 (m, 4H).

¹³C NMR (CDCl₃, 100 MHz): 130.1, 129.5, 104.1, 98.8, 66.9, 64.8 (2x), 62.2, 33.7, 30.7, 29.5, 29.1, 27.1, 25.4, 19.6.

HRMS calc. for C₁₅H₂₆O₄Na [M+Na]: 293.1723, found: 293.1727.

IR: 3295, 2940, 1436, 1350, 1199, 1164, 1075, 1033.

Table 3, entry 3. Chromatography: Pentane/Et₂O 10:1, colorless liquid, R_f = 0.32.

¹H NMR (CDCl₃, 400 MHz): 7.30-7.26 (m, 2H), 7.19-7.17 (m, 3H), 5.48-5.39 (m, 2H), 4.59 (dd, J = 2.8, 4.2 Hz, 1H), 3.90-3.84 (m, 1H), 3.75 (td, J = 6.8, 9.6 Hz, 1H), 3.53-3.47 (m, 1H), 3.39 (t, J = 6.6, 9.6 Hz, 1H), 2.61 (dd, J = 7.6, 7.9 Hz, 2H), 2.13-2.01 (m, 4H), 1.88-1.79 (m, 1H), 1.72-1.64 (m, 5H), 1.61-1.50 (m, 4H).

¹³C NMR (CDCl₃, 100 MHz): 142.5, 130.9, 130.1, 128.4 (2x), 128.2 (2x), 125.5, 98.8, 66.9, 62.2, 35.8, 32.0, 31.2, 30.7, 29.6, 29.2, 25.4, 19.6.

HRMS calc. for C₁₉H₂₈O₂Na [M+Na]: 311.1982, found: 311.1982.

IR: 3026, 2938, 2855, 1604, 1453, 1441, 1353, 1201, 1137, 1121, 1077, 1034, 969, 699.

Table 3, entry 4. Chromatography: Pentane/Et₂O 10:1, colorless liquid, R_f = 0.22.

¹H NMR (CDCl₃, 400 MHz): 7.37-7.36 (m, 4H), 7.32-7.27 (m, 1H), 5.49-5.37 (m, 2H), 4.61-4.59 (m, 1H), 4.52 (s, 2H), 3.92-3.86 (m, 1H), 3.76 (td, J = 6.8, 9.6 Hz, 1H), 3.55-3.50 (m, 1H), 3.49 (t, J = 6.5 Hz, 2H), 3.41 (td, J = 6.6, 9.6 Hz, 1H), 2.21-2.07 (m, 2H), 2.05-2.00 (m, 2H), 1.87-1.84 (m, 1H), 1.72-1.44 (m, 11H).

¹³C NMR (CDCl₃, 100 MHz): 138.6, 130.4, 129.8, 128.2 (2x), 127.5 (2x), 127.4, 98.7, 72.8, 70.2, 66.9, 62.1, 32.3, 30.7, 29.5, 29.1 (2x), 26.0, 25.4, 19.5.

HRMS calc. for C₂₁H₃₂O₃Na [M+Na]: 355.2244, found: 355.2240.

IR: 2939, 2856, 1721, 1354, 1201, 1119, 1077, 1034, 698.

Table 3, entry 5. Chromatography: Pentane/Et₂O 10:1, colorless liquid, R_f = 0.36.

¹H NMR (CDCl₃, 400 MHz): 5.42-5.30 (m, 2H), 2.31 (t, J = 7.1 Hz, 2H), 1.99-1.93 (m, 4H), 1.67-1.60 (m, 2H), 1.47-1.40 (m, 2H), 1.33-1.25 (m, 14H), 0.86 (t, J = 6.9 Hz, 3H).

¹³C NMR (CDCl₃, 100 MHz): 130.8, 129.8, 119.8, 32.5, 32.3, 31.8, 29.6, 29.2 (2x), 29.1, 28.5, 28.1, 25.3, 22.6, 17.1, 14.1.

HRMS calc. for C₁₆H₂₉NNa [M+Na]: 258.2192, found: 258.2189.

IR: 2925, 2855, 2247, 1465.

Table 3, entry 6. Chromatography: Pentane/Et₂O 10:1, colorless liquid, R_f = 0.34.

¹H NMR (CDCl₃, 400 MHz): 7.33-7.28 (m, 2H), 7.23-7.19 (m, 3H), 5.47-5.37 (m, 2H), 4.16 (q, J = 7.1 Hz, 2H), 2.69 (t, J = 7.3 Hz, 2H), 2.41-2.36 (m, 2H), 2.31 (t, J = 7.6 Hz, 2H), 2.04-1.99 (m, 2H), 1.68-1.60 (m, 2H), 1.34-1.30 (m, 4H), 1.28 (t, J = 7.1 Hz, 3H).

¹³C NMR (CDCl₃, 100 MHz): 173.8, 142.0, 130.3, 128.8, 128.4 (2x), 128.2 (2x), 125.7, 60.1, 35.9, 34.3, 29.2, 29.1, 28.7, 27.0, 24.8, 14.2.

HRMS calc. for C₁₈H₂₆O₂Na [M+Na]: 297.1845, found: 297.1843.

IR: 2932, 2857, 1736, 1454, 1373, 1179, 699.

Table 3, entry 7. Chromatography: Pentane/Et₂O 10:1, colorless liquid, R_f = 0.20.

¹H NMR (CDCl₃, 400 MHz): 7.32-7.28 (m, 2H), 7.22-7.18 (m, 3H), 5.49-5.39 (m, 2H), 4.84 (t, J = 4.8 Hz, 1H), 4.01-3.81 (m, 4H), 2.69 (t, J = 7.7 Hz, 2H), 2.40 (dt, J = 6.7, 7.8 Hz, 2H), 2.16 (dt, J = 6.7, 8.4 Hz, 2H), 1.67 (ddd, J = 5.0, 8.0, 8.1 Hz, 2H).

¹³C NMR (CDCl₃, 100 MHz): 142.0, 129.4, 129.3, 128.4 (2x), 128.2 (2x), 125.7, 104.0, 64.8 (2x), 35.8, 33.6, 29.0, 21.9.

HRMS calc. for C₁₅H₂₀O₂ [M⁺]: calculated: 232.1458, found: 232.1458.

IR: 2926, 2886, 1810, 1776, 1735, 1603, 1496, 1454, 1141, 1075, 1032, 700.

Table 3, entry 8. Chromatography: Et₂O/pentane 9:1, colorless liquid, R_f = 0.13.

¹H NMR (CDCl₃, 400 MHz): 7.31-7.28 (m, 2H), 7.22-7.18 (m, 3H), 5.46-5.35 (m, 2H), 3.68-3.63 (m, 6H), 3.47-3.45 (m, 2H), 2.67 (t, J = 7.8 Hz, 2H), 2.39-2.34 (m, 2H), 2.31 (dd, J = 7.5, 7.9 Hz, 2H), 2.01-2.00 (m, 2H), 1.65-1.59 (m, 2H), 1.34-1.31 (m, 4H).

¹³C NMR (CDCl₃, 100 MHz): 171.7, 142.0, 130.3, 128.8, 128.4 (2x), 128.2 (2x), 125.7, 69.9, 66.6, 45.9, 41.8, 35.9, 33.0, 29.3, 29.1, 29.0, 27.0, 25.1.

HRMS calc. for C₂₀H₂₉NO₂Na [M+Na]: 338.2091, found: 338.2091.

IR: 3003, 2923, 2854, 1659, 1495, 1430, 1272, 1230, 1116, 1032, 700.

Table 3, entry 9. Chromatography: Pentane/Et₂O 10:1, colorless liquid, R_f = 0.13.

¹H NMR (CDCl₃, 400 MHz): 5.14 (dt, J = 1.0, 7.1 Hz, 1H), 4.83 (t, J = 4.8 Hz, 1H), 4.56-4.54 (m, 1H), 3.99-3.80 (m, 5H), 3.69 (td, J = 6.8, 9.6 Hz, 1H), 3.51-3.45 (m, 1H), 3.34 (td, J = 6.8, 9.6 Hz, 1H), 2.14-2.08 (m, 2H), 2.04-2.01 (m, 2H), 1.85-1.79 (m, 1H), 1.73-1.64 (m, 5H), 1.60 (s, 3H), 1.57-1.49 (m, 4H).

¹³C NMR (CDCl₃, 100 MHz): 135.3, 123.5, 104.2, 98.8, 67.2, 64.8 (2x), 62.3, 36.0, 33.9, 30.7, 27.9, 25.4, 22.6, 19.6, 15.8.

HRMS calc. for C₁₆H₂₈O₄Na [M+Na]: 307.1880, found: 307.1885.

IR: 2941, 1811, 1776, 1733, 1442, 1385, 1354, 1138, 1034.

ppm

ppm

Integral

Current Data Parameters

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PROCNO 1

F2 - Acquisition Parameters

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F2 - Processing parameters

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1D NMR plot parameters

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Current Data Parameters
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$C_{21}H_{32}O_3$

Current Data Parameters
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 SF01 400.1324710 MHz
 F2 - Processing parameters
 SI 32768
 SF 400.130000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00
 1D NMR plot parameters
 CX 20.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 0.50000 ppm/cm
 HZCM 200.06500 Hz/cm

C₁₆H₂₅N

Current Data Parameters

NAME km331

EXPNO 3

PROCNO 1

F2 - Acquisition Parameters

Date_ 20021109

Time 9.17

INSTRUM spect

PROBHD 5mm BBO BB-1

PULPROG zg30

TD 65536

SOLVENT CDCl3

NS 16

DS 2

SWH 8278.146 Hz

FIDRES 0.126314 Hz

AQ 3.9584243 sec

RG 4

DM 60.400 usec

DE 6.00 usec

TE 300.0 K

D1 1.00000000 sec

===== CHANNEL f1 =====

NUC1 1H

P1 7.90 usec

PL1 0.00 dB

SFO1 400.1324710 MHz

F2 - Processing parameters

SI 32768

SF 400.1300119 MHz

WDW EM

SSB 0

LB 0.30 Hz

GB 0

PC 1.00

1D NMR plot parameters

CX 20.00 cm

F1P 10.000 ppm

F1 4001.30 Hz

F2P 0.000 ppm

F2 0.00 Hz

PPMCM 0.50000 ppm/cm

HZCM 200.06500 Hz/cm

C₁₈H₂₆O₂

Current Data Parameters
 NAME km275
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020923
 Time 19.06
 INSTRUM spect
 PROBHD 5mm BBO BB-1
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SMH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.958423 sec
 RG 40.3
 DM 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.90 usec
 PL1 0.00 dB
 SF01 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300000 MHz
 MDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 0.50000 ppm/cm
 HZCM 200.06500 Hz/cm

C₁₅H₁₂O₂

Current Data Parameters

NAME	km326
EXPNO	5
PROCNO	1

F2 - Acquisition Parameters

Date_	20021109
Time	8.42
INSTRUM	spect
PROBHD	5mm BB0 BB-1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SMH	8278.146 Hz
FIDRES	0.126314 Hz
AG	3.9584243 sec
RG	4
DW	60.400 usec
DE	5.00 usec
TE	300.0 K
D1	1.00000000 sec

===== CHANNEL f1 =====

NUC1 1H

P1 7.90 usec

PL1 0.00 dB

SFO1 400.1324710 MHz

F2 - Processing parameters

SI	32768
SF	400.1300114 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	10.000 ppm
F1	4001.30 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	0.50000 ppm/cm
HZCM	200.06500 Hz/cm

C₁₀H₁₂O₂N

Current Data Parameters

NAME km340

EXPNO 3

PROCNO 1

F2 - Acquisition Parameters

Date_ 20021111

Time 18.10

INSTRUM spect

PROBHD 5mm BBO BB-1

PULPROG zg30

TD 65536

SOLVENT CDCl3

NS 16

DS 2

SWH 8278.146 Hz

FIDRES 0.126314 Hz

AQ 3.9584243 sec

RG 57

DW 60.400 usec

DE 6.00 usec

TE 300.0 K

1.00000000 sec

===== CHANNEL f1 =====

NUC1 1H

P1 7.90 usec

PL1 0.00 dB

SFO1 400.1324710 MHz

F2 - Processing parameters

SI 32768

SF 400.130000 MHz

WDW EM

SSB 0

LB 0.30 Hz

GB 0

PC 1.00

1D NMR plot parameters

CX 20.00 cm

F1P 10.000 ppm

F1 4001.30 Hz

F2P 0.000 ppm

F2 0.00 Hz

PPMCM 0.50000 ppm/cm

HZCM 200.06500 Hz/cm

C₁₆H₂₈O₂

Current Data Parameters
 NAME Km341
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20021111
 Time 18.26
 INSTRUM spect
 PROBHD 5mm BBO BB-1
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SMH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 4
 DM 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 MUC1 1H
 P1 7.90 usec
 PL1 0.00 dB
 SF01 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300099 MHz
 MDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 0.50000 ppm/cm
 HZCM 200.06500 Hz/cm