An Efficient One-pot Synthesis of Unsymmetrical Ethers: A Directly Reductive Deoxygenation of Esters Using an InBr₃/Et₃SiH Catalytic System

Supporting Information

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Experimental Section

General Methods: Column chromatography was performed using Silica gel 60. Chloroform and THF were distilled from P₂O₅ and sodium-benzophenone, respectively. Other organic solvents were dried and distilled prior to use. InBr₃, InCl₃, and In(OTf)₃ were commercial available, and used without a further purification. All reactions were carried out under a nitrogen atmosphere, unless otherwise noted. ¹H-NMR spectra were measured at 500 MHz using tetramethylsilane as an internal standard. ¹³C-NMR spectra were measured at 125 MHz using the center peak of chloroform (77.0 ppm) as an internal standard. High-resolution mass was measured using NBA (3-nitrobenzylalcohol) as a matrix. The ester compounds **1** and **3a-c**, and **5a,b**, which were prepared from the corresponding alcohol/thiol and acetyl chloride, are known compounds, and were identified by ¹H NMR and ¹³C NMR by comparison to literature data. 2-Phenylethyl acetate (**1a**), benzyl acetate (**1b**), ethyl benzoate (**1i**), 3-isochromanone (**1o**), ethyl thiophenyl-2-acetate (**1m**), 3-phenylpropanoic acid (**5c**), and 4-phenyl-2-butanone (**5d**) were commercial available and used without a further purification. Phenethyl butyrate (**1e**), phenethyl isobutyrate (**1f**), and γ -lactone (**1n**) were also purchased.

General Procedure for the preparation of esters: An alcohol (10 mmol), acetyl chloride (20 mmol), and Et₃N (20 mmol) were successively added via a syringe to anhydrous Et₂O (20 mL) at room temperature under a N₂ atmosphere. The reaction mixture was stirred for 2 h. The resulting precipitation was filtered, and the filtrate was washed with H₂O. The organic layer was dried over anhydrous Na₂SO₄, and evaporated under reduced pressure. The crude product was purified by a silica gel chromatography (hexane : AcOEt = 8 : 2) to give the corresponding ester as shown in Table 2, Scheme 1, and Scheme 2.

4-Nitrophenethyl acetate¹⁾ (**1c**): 89% yield; colorless needles; ¹H NMR (500 MHz, CDCl₃) δ 2.01 (s, 3H), 3.03 (t, 2H, J = 6.5 Hz), 4.30 (t, 2H, J = 6.5 Hz), 7.36 (d, 2H, J = 8.5 Hz), 8.14 (d, 2H, J = 8.5 Hz); ¹³C NMR (125 MHz, CDCl₃) δ 20.8, 34.9, 63.8, 123.7, 129.7, 145.7, 146.9, 170.8; MS (EI) m/z 209 (M⁺), 151 (100%).

3-Phenylpropyl acetate²⁾ (**1d**): 99% yield; yellow oil; ¹H NMR (500 MHz, CDCl₃) δ 1.97 (quint, 2H, J = 7.0 Hz), 2.05 (s, 3H), 2.69 (t, 2H, J = 7.0 Hz), 4.09 (t, 2H, J = 7.0), 7.18-7.30 (m, 5H); ¹³C NMR (125 MHz, CDCl₃) δ 20.9, 30.2, 32.2, 63.8, 126.0, 128.3, 128.4, 141.2, 171.1; MS (EI): m/z 178 (M⁺), 91 (100%).

Methyl (4-bromophenyl)acetate³⁾ (**1h**): 89% yield; colorless oil; ¹H NMR (500 MHz, CDCl₃) δ 3.58 (s, 2H), 3.69 (s, 3H), 7.15 (d, 2H, J = 8.5 Hz), 7.44 (d, 2H, J = 8.5 Hz); ¹³C NMR (125 MHz, CDCl₃) δ 40.5, 52.1, 121.2, 130.9, 131.4, 132.9, 171.4; MS (EI) m/z 229, 231 (M⁺, M⁺+2).

n-Decyl acetate⁴⁾ (**1j**): 99% yield; yellow oil; ¹H NMR (500 MHz, CDCl₃) δ 0.85 (t, 3H, J = 7.0 Hz),

1.23-1.40 (m, 14H), 1.58 (quint, 2H, $J = 7.0$ Hz), 2.01 (s, 3H), 4.02 (t, 2H, $J = 7.0$ Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 14.0, 20.9, 22.6, 25.9, 28.6, 29.2, 29.3, 29.5 (d), 31.8, 64.6, 171.2; MS (FAB): m/z 201 ($\text{M}+\text{H}$, 100%).

1-Acetoxy-10-bromo decane⁵⁾ (**1k**): 99% yield; colorless oil; ^1H NMR (500 MHz, CDCl_3) δ 1.20-1.30 (m, 10H), 1.35 (m, 2H), 1.55 (t, 2H, $J = 7.0$ Hz), 1.78 (quint, 2H, $J = 7.0$ Hz), 1.98 (s, 3H), 3.33 (t, 2H, $J = 7.0$ Hz), 3.98 (t, 2H, $J = 7.0$ Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 21.0, 25.8, 28.1, 28.5, 28.7, 29.1, 29.2, 29.3, 32.8, 33.9, 64.5, 171.1; MS (EI) m/z 278 (M^+), 280 (M^++2), 107 (100%).

2-Decyl acetate⁵⁾ (**1l**): 99% yield; colorless oil; ^1H NMR (500 MHz, CDCl_3) δ 0.88 (t, 3H, $J = 7.0$ Hz), 1.21 (t, 3H, $J = 6.5$ Hz), 1.26-1.30 (m, 12H), 1.46 (m, 1H), 1.58 (m, 1H), 2.03 (s, 3H), 4.89 (sex, 1H, $J = 6.5$ Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 14.1, 19.9, 21.4, 22.6, 25.4, 29.2, 29.4, 29.5, 31.8, 35.9, 71.1, 170.8; MS (EI): m/z 200 (M^+), 103 (100%).

Diphenylmethyl acetate⁶⁾ (**3a**): 88% yield; yellow oil; ^1H NMR (500 MHz, CDCl_3) δ 2.16 (s, 3H), 6.88 (s, 1H), 7.28 (m, 2H), 7.33-7.35 (m, 8H); ^{13}C NMR (125 MHz, CDCl_3) δ 21.3, 76.7, 127.1, 127.9, 128.5, 140.2, 170.0; MS (EI): m/z 226 (M^+), 166 (100%).

α -Methylbenzyl acetate⁶⁾ (**3b**): 92% yield; yellow oil; ^1H NMR (500 MHz, CDCl_3) δ 1.53 (d, 3H, $J = 6.5$ Hz), 2.07 (s, 3H), 5.89 (q, 1H, $J = 6.5$ Hz), 7.29 (m, 1H), 7.33 (m, 4H); ^{13}C NMR (125 MHz, CDCl_3) δ 21.3, 22.2, 72.3, 126.1, 127.8, 128.5, 141.7, 170.3; MS (EI): m/z 164 (M^+), 104 (100%).

4-Phenyl-3-butene-2-ol acetate⁷⁾ (**3c**): 99% yield; colorless oil; ^1H NMR (500 MHz, CDCl_3) δ 1.33 (d, 3H, $J = 6.5$ Hz), 2.00 (s, 3H), 5.45 (quint, 1H, $J = 6.5$ Hz), 6.12 (dd, 1H, $J = 16.0, 7.5$ Hz), 6.53 (d, 1H, $J = 16.0$ Hz), 7.17 (m, 1H), 7.25 (m, 2H), 7.30 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 20.4, 21.4, 71.0, 126.5, 127.9, 128.5, 128.8, 131.5, 136.3, 170.3; MS (EI) m/z 190 (M^+), 129 (100%).

Phenethyl thioacetate⁸⁾ (**5a**): 86% yield; colorless oil; ^1H NMR (500 MHz, CDCl_3) δ 2.33 (s, 3H), 2.87 (t, 2H, $J = 7.5$ Hz), 3.12 (t, 2H, $J = 7.5$ Hz), 7.23 (m, 3H), 7.30 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 30.5, 30.7, 35.8, 126.5, 128.5, 128.6, 140.0, 195.7; MS (EI): m/z 180 (M^+), 75 (100%).

***N*-Phenethylacetamide**⁹⁾ (**5b**): 91% yield; colorless oil; ^1H NMR (500 MHz, CDCl_3) δ 1.90 (s, 3H), 2.80 (t, 2H, $J = 7.0$ Hz), 3.48 (d, 2H, $J = 7.0$ Hz), 7.17-7.23 (m, 3H), 7.27-7.31 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 23.1, 35.5, 40.6, 126.3, 128.5, 128.6, 138.8, 170.1; MS (EI): m/z 163 (M^+).

General procedure for the reduction of esters using the $\text{InBr}_3\text{-Et}_3\text{SiH}$ system: To a freshly distilled CHCl_3 solution (0.6 mL) in a screw-capped vial under N_2 atmosphere, ester **1** (0.6 mmol), InBr_3 (10.6 mg, 0.0300 mmol) and Et_3SiH (380 μL , 2.40 mmol) was successively added, and the vial was sealed with a cap containing a PTFE septum. During the stirring of the reaction mixture at 60 $^\circ\text{C}$ (bath temperature), the solution turned from colorless to yellow, then to orange. The reaction was monitored

by GC analysis until consumption of the starting ester. After the reaction, H₂O (3 mL) was added, and the resulting orange suspension was stirred continuously until the disappearance of the color. The aqueous layer was extracted with CH₂Cl₂ (15 mL), the combined organic phase was dried over anhydrous Na₂SO₄ filtered, and then evaporated under reduced pressure. The crude product was purified by flash column chromatography (SiO₂ / hexane : AcOEt = 99 : 1) to give the corresponding ether **2** as shown in Table 2, and in Scheme 2.

Ethyl phenethyl ether¹⁰ (**2a**): 86% yield; colorless oil; ¹H NMR (500 MHz, CDCl₃) δ 1.20 (t, 3H, *J* = 7.5 Hz), 2.90 (t, 2H, *J* = 7.5 Hz), 3.50 (q, 2H, *J* = 7.5 Hz), 3.63 (t, 2H, *J* = 7.5 Hz), 7.22 (m, 3H), 7.28 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 15.2, 36.4, 66.2, 71.6, 126.1, 128.3, 128.9, 139.0; MS (EI): *m/z* 150 (M⁺), 105 (100%).

Ethyl benzyl ether¹¹ (**2b**): 69% yield; colorless oil; ¹H NMR (500 MHz, CDCl₃) δ 1.25 (t, 3H, *J* = 7.0 Hz), 3.54 (q, 2H, *J* = 7.0 Hz), 4.51 (s, 2H), 7.26 (m, 1H), 7.33 (m, 4H); ¹³C NMR (125 MHz, CDCl₃) δ 15.2, 65.7, 72.7, 127.5, 127.7, 128.3, 138.6; MS (EI): *m/z* 136 (M⁺), 91 (100%).

Ethyl 4-nitrophenethyl ether (**2c**): 61% yield; brown oil; ¹H NMR (500 MHz, CDCl₃) δ 1.19 (t, 3H, *J* = 7.0 Hz), 2.98 (t, 2H, *J* = 7.0 Hz), 3.49 (t, 2H, *J* = 7.0 Hz), 3.67 (t, 2H, *J* = 7.0 Hz), 7.39 (d, 2H, *J* = 8.5 Hz), 8.15 (d, 2H, *J* = 8.5 Hz); ¹³C NMR (125 MHz, CDCl₃) δ 15.1, 36.2, 66.4, 70.3, 123.5, 129.7, 146.6, 147.3; MS (FAB): *m/z* 196 (M⁺+1, 100%), HRMS (FAB): Calcd for C₁₀H₁₄NO₃: 196.0974, Found: 196.0978.

Ethyl 3-phenylpropyl ether¹² (**2d**): 89% yield; colorless oil; ¹H NMR (500 MHz, CDCl₃) δ 1.21 (t, 3H, *J* = 7.0 Hz), 1.90 (m, 2H), 2.69 (t, 2H, *J* = 7.0 Hz), 3.42 (t, 2H, *J* = 7.0 Hz), 3.46 (q, 2H, *J* = 7.0 Hz), 7.17 (m, 3H), 7.27 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 15.2, 31.3, 32.4, 66.1, 69.7, 125.7, 128.3, 128.4, 142.0; MS (EI): *m/z* 164 (M⁺), 118 (100%).

***n*-Butyl phenethyl ether**¹³ (**2e**): 71% yield; colorless oil; ¹H NMR (500 MHz, CDCl₃) δ 0.91 (t, 3H, *J* = 7.0 Hz), 1.35 (sex, 2H, *J* = 7.0 Hz), 1.56 (quint, 2H, *J* = 7.0 Hz), 2.89 (t, 2H, *J* = 7.0 Hz), 3.43 (t, 2H, *J* = 7.0 Hz), 3.62 (t, 2H, *J* = 7.0 Hz), 7.20 (m, 3H), 7.28 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 13.9, 19.3, 31.8, 36.4, 70.8, 71.8, 126.1, 128.3, 128.9, 139.1; MS (EI): *m/z* 178 (M⁺), 57 (100%).

Phenethyl isobutyl ether (2f): 71% yield; colorless oil; ^1H NMR (500 MHz, CDCl_3) δ 0.89 (d, 6H, $J = 7.0$ Hz), 1.84 (m, 1H), 2.89 (t, 2H, $J = 7.0$ Hz), 3.20 (d, 2H, $J = 6.5$ Hz), 3.62 (t, 2H, $J = 7.0$ Hz), 7.20 (m, 3H), 7.28 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 19.4, 28.4, 36.4, 71.9, 76.7, 126.1, 128.3, 128.9, 139.2; MS (FAB): m/z 178 (M^+), 105 (100%); HRMS (FAB) Calcd for $\text{C}_{12}\text{H}_{18}\text{O}$ 178.1358, Found 178.1337.

Methyl 4-bromophenethyl ether¹⁴ (2h): 62% yield; colorless oil; ^1H NMR (500 MHz, CDCl_3) δ 2.83 (t, 2H, $J = 7.0$ Hz), 3.34 (s, 3H), 3.57 (t, 2H, $J = 7.0$ Hz), 7.09 (d, 2H, $J = 8.0$ Hz), 7.40 (d, 2H, $J = 8.0$ Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 35.6, 58.7, 73.1, 120.0, 130.9, 131.4, 138.0; MS (EI) m/z 215, 217 (M^+ , $\text{M}^+ + 2$).

1-Ethoxydecane¹⁰ (2j): 77% yield; colorless oil; ^1H NMR (500 MHz, CDCl_3) δ 0.88 (t, 3H, $J = 7.0$ Hz), 1.20 (t, 3H, $J = 7.0$ Hz), 1.25-1.40 (m, 14H), 1.57 (quint, 2H, $J = 7.0$ Hz), 3.40 (t, 2H, $J = 7.0$ Hz), 3.46 (q, 2H, $J = 7.0$ Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 14.1, 15.2, 22.7, 26.2, 29.3, 29.5, 29.7, 29.6, 29.8, 31.9, 66.0, 70.8; MS (EI) m/z 186 (M^+ , 100%).

1-Bromo-10-ethoxydecane¹⁵ (2k): 73% yield; colorless oil; ^1H NMR (500 MHz, CDCl_3) δ 1.20 (t, 3H, $J = 7.5$ Hz), 1.25-1.30 (m, 10H), 1.42 (m, 2H), 1.56 (t, 2H, $J = 7.5$ Hz), 1.85 (quint, 2H, $J = 7.5$ Hz), 3.40 (t, 4H, $J = 7.5$ Hz), 3.46 (q, 2H, $J = 7.5$ Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 15.1, 26.1, 28.0, 28.6, 29.2, 29.3, 29.4, 29.7, 32.7, 33.7, 65.9, 70.6; MS (EI): m/z 264 (M^+), 266 ($\text{M}^+ + 2$), 71 (100%).

2-Ethoxydecane⁵ (2l): 69% yield; colorless oil; ^1H NMR (500 MHz, CDCl_3) δ 0.81 (t, 3H, $J = 7.0$ Hz), 1.05 (d, 3H, $J = 6.0$ Hz), 1.12 (t, 3H, $J = 7.0$ Hz), 1.20-1.25 (m, 11H), 1.28 (brs, 2H), 1.45 (brs, 1H), 3.29 (sex, 1H, $J = 6.0$ Hz), 3.34 (quint, 1H, $J = 7.0$ Hz), 3.46 (quint, 1H, $J = 7.0$ Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 14.1, 15.6, 19.8, 22.7, 25.6, 29.3, 29.6, 29.8, 31.9, 36.7, 63.5, 75.2; MS (EI) m/z 186 (M^+), 73 (100%).

1-Ethoxy-2-(2-thiophenyl)ethane (2m): 55% yield; colorless oil; ^1H NMR (500 MHz, CDCl_3) δ 1.22 (t, 3H, $J = 7.0$ Hz), 3.10 (t, 2H, $J = 7.0$ Hz), 3.53 (dd, 2H, $J = 7.0, 7.0$ Hz), 3.66 (t, 2H, $J = 7.0$ Hz), 6.86 (d, 1H, $J = 3.5$ Hz), 6.93 (dd, 1H, $J = 5.5, 3.5$ Hz), 7.14 (d, 1H, $J = 5.5$ Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 15.2, 30.5, 66.3, 71.2, 123.5, 125.0, 126.6, 141.3; MS (EI) m/z 156 (M^+). HRMS (FAB):

Calcd for C₈H₁₃OS: 157.0687, Found: 157.0690.

Isochroman¹⁶⁾ (**2o**): 84% yield; colorless oil; ¹H NMR (500 MHz, CDCl₃) δ 2.86 (t, 2H, *J* = 5.5 Hz), 3.97 (t, 2H, *J* = 5.5 Hz), 4.77 (s, 2H), 6.97 (m, 1H), 7.11 (m, 1H), 7.16 (m, 2H); ¹³C NMR (CDCl₃) δ 28.3, 65.4, 67.9, 124.4, 125.9, 126.3, 128.9, 133.2, 134.9; MS (EI) *m/z* 134 (M⁺), 104 (100%).

Diphenylmethane¹⁷⁾ (**4a**): 98% yield; colorless oil; ¹H NMR (500 MHz, CDCl₃) δ 3.98 (s, 2H), 7.20 (m, 6H), 7.27 (m, 4H); ¹³C NMR (125 MHz, CDCl₃) δ 41.9, 126.0, 128.4, 128.9, 141.1; MS (EI) *m/z* 168 (M⁺, 100%).

Ethylbenzene¹⁷⁾ (**4b**): 76% yield; colorless oil; ¹H NMR (500 MHz, CDCl₃) δ 1.24 (t, 3H, *J* = 7.5 Hz), 2.65 (q, 2H, *J* = 7.5 Hz), 7.18 (m, 3H), 7.28 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 15.6, 28.9, 125.6, 127.8, 128.3, 131.8, 144.2; MS (EI) *m/z* 106 (M⁺), 91 (100%).

β-Ethyl styrene¹⁸⁾ (**4c**): 41% yield; colorless oil; ¹H NMR (500 MHz, CDCl₃) δ 1.09 (t, 3H, *J* = 7.5 Hz), 2.23 (quint, 2H, *J* = 7.5 Hz), 6.27 (dt, 1H, *J* = 6.5 Hz, 16 Hz), 6.37 (d, 1H, *J* = 16 Hz), 7.18 (m, 2H), 7.27 (m, 2H), 7.34 (m, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 13.6, 26.1, 125.9, 126.7, 128.5, 128.8, 132.6, 137.9; MS (EI): *m/z* 132 (M⁺).

1-Phenyl-2-butene¹⁹⁾ (**4c'**): 18% yield; colorless oil; ¹H NMR (500 MHz, CDCl₃) δ 1.68 (d, 3H, *J* = 6.0 Hz), 3.31 (d, 2H, *J* = 6.5 Hz), 5.48-5.54 (m, 1H), 5.56-5.62 (m, 1H), 7.18 (m, 2H), 7.27 (m, 2H), 7.34 (m, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 17.9, 39.0, 125.9, 126.3, 128.3, 128.4, 130.0, 141.1; MS (EI): *m/z* 132 (M⁺).

3-Phenyl-1-propanol²⁰⁾ (**6c**): 32% yield; colorless oil; ¹H NMR (500 MHz, CDCl₃) δ 1.45 (brs, 1H), 1.90 (quint, 2H, *J* = 7.5 Hz), 2.71 (t, 2H, *J* = 7.5 Hz), 3.67 (t, 2H, *J* = 7.5 Hz), 7.20 (m, 3H), 7.28 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 32.1, 34.2, 62.2, 125.8, 128.4, 141.8; MS (EI): *m/z* 136 (M⁺).

Bis(4-phenyl-2-butyl)ether²¹⁾ (**6d**): 88% yield; colorless oil; ¹H NMR (500 MHz, CDCl₃) δ 1.15 (dd, 3H, *J* = 6, 16 Hz), 1.72 (m, 1H), 1.82 (m, 1H), 2.63 (m, 1H), 2.73 (m, 1H), 3.48 (m, 1H), 7.17 (m, 3H), 7.27 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 20.7 (d), 32.0 (d), 38.9 (d), 72.5 (d), 125.6 (d), 128.3 (d), 128.4 (d), 142.4 (d); MS (FAB): *m/z* 283 (M⁺+H), 133 (100%); HRMS (FAB): Calcd for C₂₀H₂₇O: 283.2061, Found: 283.2062.

References

- 1) Kawada, A.; Takeda, S.; Yamashita, K.; Abe, H.; Harayama, T. *Chem. Pharm. Bull.* **2002**, *50*, 1060.
- 2) Davies, H. M. L.; Beckwith, R. E. J.; Antoulinakis, E. G.; Jin, Q. *J. Org. Chem.* **2003**, *68*, 6126.
- 3) Tamura, Y.; Yakura, T.; Shirouchi, Y.; Haruta, J.; *Chem. Pharm. Bull.* **1985**, *33*, 1097.
- 4) Nikolaides, N.; Ganem, B. *J. Org. Chem.* **1989**, *54*, 5996.
- 5) Gasparrini, F.; Caglioti, L.; Misiti, D.; Palmieri, G.; Ballini, R. *Tetrahedron* **1982**, *38*, 3609.
- 6) Karimi, B.; Maleki, J. *J. Org. Chem.* **2003**, *68*, 4591.
- 7) Onaran, M.; Seto, C. T. *J. Org. Chem.* **2003**, *68*, 8136.
- 8) Holmes, B. T.; Snow, A. W. *Tetrahedron* **2005**, *61*, 12339.
- 9) Kita, Y.; Akai, S.; Ajimura, N.; Yoshigi, M.; Tsugoshi, T.; Yasuda, H.; Tamura, Y. *J. Org. Chem.* **1986**, *51*, 4150.
- 10) Oka, K.; Nakao, R. *J. Organomet. Chem.* **1989**, *362*, 31.
- 11) Ochiai, M.; Ito, T.; Takahashi, H.; Nakanishi, A.; Toyonari, M.; Sueda, T.; Goto, S.; Shiro, M. *J. Am. Chem. Soc.* **1996**, *118*, 7716.
- 12) (a) Ishikawa, H.; Mukaiyama, T. *Bull. Chem. Soc. Jpn.* **1978**, *51*, 2059. (b) Matsubara, K.; Iura, T.; Maki, T.; Nagashima, H. *J. Org. Chem.* **2002**, *67*, 4985.
- 13) Inoue, K.; Sawada, A.; Shibata, I.; Baba, A. *J. Am. Chem. Soc.* **2002**, *124*, 906.
- 14) Yato, M.; Homma, K.; Ishida, A. *Tetrahedron* **2001**, *57*, 5353.
- 15) Murayama, T.; Nagai, M.; Fujiyama, T.; Sakai, Y. *JP Patent*, 2000, 16969 (*Chem. Abstr.* 2000, 132, 100535).
- 16) Adcock, W.; Gupta, B. D.; Kitching, W. *J. Org. Chem.* **1976**, *41*, 1498.
- 17) Shapiro, M. J. *J. Org. Chem.* **1976**, *41*, 3197.
- 18) J. M. Concellón, ; H. Rodríguez-Solla; C. Simal; H. Huerta, H. *Org. Lett.* **2005**, *7*, 5833.
- 19) McEnroe, F. J.; Sha, C.-K.; Hall, S. S. *J. Org. Chem.* **1976**, *41*, 3465.
- 20) Chen, G.; Fu, C.; Ma, S. *Tetrahedron* **2006**, *62*, 4444.
- 21) Wada, M.; Nagayama, S.; Mizutani, K.; Hiroi, R.; Miyoshi, N. *Chem. Lett.* **2002**, *31*, 248.

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 PD 4.000 sec
 FW1 7.5 us
 IRNUC 1H
 CTEMP 23.6 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 RF 0.23 Hz
 RGAIN 11

1k

Single Pulse Experiment

DFILE E:\CX\J\157-1H-2.als
 COMNT Single Pulse Experiment
 DATIM 6-NOV-2006 17:17:52
 OBNUC 1H
 EXMOD single pulse.exp
 OBFRQ 500.00 MHz
 OBSET 162.00 KHz
 OBFIN 416.0 Hz
 POINT 16384
 FREQU 7507.5 Hz
 SCANS 8
 ACQTM 2.182 sec
 PD 4.000 sec
 FW1 7.5 us
 IRNUC 1H
 CTEMP 24.4 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 RF 0.23 Hz
 RGAIN 14

1l

Single Pulse Experiment

DFILE E:\tm-185-1H.als
 COMNT Single Pulse Experiment
 DATIM 24-NOV-2006 15:55:20
 OBNUC 1H
 EXMOD single_pulse.exp
 OBFRQ 500.00 MHz
 OBSET 162.00 KHz
 OBFIN 416.0 Hz
 POINT 16384
 FREQU 7507.5 Hz
 SCANS 8
 ACQTM 2.182 sec
 PD 4.000 sec
 PW1 7.5 us
 IRNUC 1H
 CTEMP 24.2 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.23 Hz
 RGAIN 15

2a

Single Pulse with Broadband Decoupling

DFILE E:\tm-185-13C.als
 COMNT Single Pulse with Broadband
 DATIM 24-NOV-2006 16:04:32
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFRQ 125.00 MHz
 OBSET 777.00 KHz
 OBFIN 875.5 Hz
 POINT 32768
 FREQU 31446.5 Hz
 SCANS 237
 ACQTM 1.042 sec
 PD 1.000 sec
 PW1 4.2 us
 IRNUC 1H
 CTEMP 26.9 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.50 Hz
 RGAIN 30

2a

Single Pulse Experiment

DFILE E:\tm-276-3-1H.als
 COMNT Single Pulse Experiment
 DATIM 30-JAN-2007 19:52:57
 OBNUC 1H
 EXMOD single pulse.exp
 OBFRQ 500.00 MHz
 OBSET 162.00 KHz
 OBFIN 416.0 Hz
 POINT 16384
 FREQU 7507.5 Hz
 SCANS 8
 ACQTM 2.182 sec
 PD 4.000 sec
 PW1 7.5 us
 IRNUC 1H
 CTEMP 26.2 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.23 Hz
 RGAIN 17

2b

Single Pulse with Broadband Decoupling

DFILE E:\tm-276-3-13C.als
 COMNT Single Pulse with Broadb
 DATIM 30-JAN-2007 20:07:37
 OBNUC 13C
 EXMOD single pulse dec
 OBFRQ 125.00 MHz
 OBSET 777.00 KHz
 OBFIN 875.5 Hz
 POINT 32768
 FREQU 31446.5 Hz
 SCANS 351
 ACQTM 1.042 sec
 PD 1.000 sec
 PW1 4.2 us
 IRNUC 1H
 CTEMP 28.1 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.50 Hz
 RGAIN 30

2b

Single Pulse Experiment

DFILE E:\tm-301-r2-1H.als
 COMNT Single Pulse Experiment
 DATIM 1-MAR-2007 16:27:04
 OBNUC 1H
 EXMOD single pulse.exp
 OBFRQ 500.00 MHz
 OBSET 162.00 KHz
 OBFIN 416.0 Hz
 POINT 16384
 FREQU 7507.5 Hz
 SCANS 8
 ACQTM 2.182 sec
 PD 4.000 sec
 PW1 7.5 us
 IRNUC 1H
 CTEMP 24.1 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 RF 0.00 Hz
 RGAIN 15

2c

Single Pulse with Broadband Decoupling

DFILE C:\Windows\Adm\A\18\tm-
 COMNT Single Pulse with Broad
 DATIM 1-MAR-2007 16:45:16
 OBNUC 13C
 EXMOD single pulse dec
 OBFRQ 125.00 MHz
 OBSET 777.00 KHz
 OBFIN 875.5 Hz
 POINT 32768
 FREQU 31446.5 Hz
 SCANS 509
 ACQTM 1.042 sec
 PD 1.000 sec
 PW1 4.2 us
 IRNUC 1H
 CTEMP 25.2 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 RF 0.50 Hz
 RGAIN 30

2c

Single Pulse Experiment

DFILE E:\tm-225-1H.als
 COMNT Single Pulse Experiment
 DATIM 26-DEC-2006 18:46:10
 OBNUC 1H
 EXMOD single_pulse.exp
 OBFRQ 500.00 MHz
 OBSET 162.00 KHz
 OBFIN 416.0 Hz
 POINT 16384
 FREQU 7507.5 Hz
 SCANS 8
 ACQTM 2.182 sec
 PD 4.000 sec
 PW1 7.5 us
 IRNUC 1H
 CTEMP 25.4 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 12

2d

Single Pulse with Broadband Decoupling

DFILE E:\tm-225-13C.als
 COMNT Single Pulse with Br
 DATIM 26-DEC-2006 18:56:07
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFRQ 125.00 MHz
 OBSET 777.00 KHz
 OBFIN 875.5 Hz
 POINT 32768
 FREQU 31446.5 Hz
 SCANS 267
 ACQTM 1.042 sec
 PD 1.000 sec
 PW1 4.2 us
 IRNUC 1H
 CTEMP 27.5 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.50 Hz
 RGAIN 30

2d

Single Pulse Experiment

DFILE E:\tm-070129-1H.als
 COMNT Single Pulse Experime
 DATIM 29-JAN-2007 15:20:03
 OBNUC 1H
 EXMOD single_pulse.exp
 OBFRQ 500.00 MHz
 OBSET 162.00 KHz
 OBFIN 416.0 Hz
 POINT 16384
 FREQU 7507.5 Hz
 SCANS 8
 ACQTM 2.182 sec
 PD 4.000 sec
 FW1 7.5 us
 IRNUC 1H
 CTEMP 24.3 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.23 Hz
 RGAIN 17

2e

Single Pulse with Broadband Decoupling

DFILE E:\tm-070129-13C.als
 COMNT Single Pulse with Broa
 DATIM 29-JAN-2007 15:30:18
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFRQ 125.00 MHz
 OBSET 777.00 KHz
 OBFIN 875.5 Hz
 POINT 32768
 FREQU 31446.5 Hz
 SCANS 275
 ACQTM 1.042 sec
 PD 1.000 sec
 FW1 4.2 us
 IRNUC 1H
 CTEMP 27.1 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.50 Hz
 RGAIN 30

2e

Single Pulse Experiment

DFILE E:\tm-280-1H.als
 COMNT Single Pulse Experim
 DATIM 9-FEB-2007 15:50:58
 OBNUC 1H
 EXMOD single_pulse.exp
 OBFRQ 500.00 MHz
 OBSET 162.00 KHz
 OBFIN 416.0 Hz
 POINT 16384
 FREQU 7507.5 Hz
 SCANS 8
 ACQTM 2.182 sec
 PD 4.000 sec
 FWL 7.5 us
 IRNUC 1H
 CTEMP 23.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.23 Hz
 RGAIN 16

2f

Single Pulse with Broadband Decoupling

DFILE E:\tm-280-13C.als
 COMNT Single Pulse with Br
 DATIM 9-FEB-2007 16:02:21
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFRQ 125.00 MHz
 OBSET 777.00 KHz
 OBFIN 875.5 Hz
 POINT 32768
 FREQU 31446.5 Hz
 SCANS 309
 ACQTM 1.042 sec
 PD 1.000 sec
 FWL 4.2 us
 IRNUC 1H
 CTEMP 24.8 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.50 Hz
 RGAIN 30

2f

Single Pulse Experiment

DFILE E:\tm-277-1H.als
 COMNT Single Pulse Experime
 DATIM 1-FEB-2007 16:09:14
 OBNUC 1H
 EXMOD single_pulse.exp
 OBFRQ 500.00 MHz
 OBSET 162.00 KHz
 OBFIN 416.0 Hz
 POINT 16384
 FREQU 7507.5 Hz
 SCANS 8
 ACQTM 2.182 sec
 PD 4.000 sec
 PW1 7.5 us
 IRNUC 1H
 CTEMP 24.7 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.23 Hz
 RGAIN 13

2j

Single Pulse with Broadband Decoupling

DFILE E:\tm-277-13C.als
 COMNT Single Pulse with Brc
 DATIM 1-FEB-2007 16:17:12
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFRQ 125.00 MHz
 OBSET 777.00 KHz
 OBFIN 875.5 Hz
 POINT 32768
 FREQU 31446.5 Hz
 SCANS 209
 ACQTM 1.042 sec
 PD 1.000 sec
 PW1 4.2 us
 IRNUC 1H
 CTEMP 26.3 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.50 Hz
 RGAIN 30

2k

Single Pulse Experiment

DFILE E:\CX\J\tm-070
 COMNT Single Pulse E
 DATIM 5-MAR-2007 18
 OBNUC 1H
 EXMOD single pulse.e
 OBFRQ 500.00 MHz
 OBSET 162.00 KHz
 OBFIN 416.0 Hz
 POINT 16384
 FREQU 7507.5 Hz
 SCANS 8
 ACQTM 2.182 sec
 PD 4.000 sec
 PW1 7.5 us
 IRNUC 1H
 CTEMP 24.3 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.23 Hz
 RGAIN 21

21

Single Pulse with Broadband Decoupling

DFILE C:\Windows\Apw\A-1
 COMNT Single Pulse with
 DATIM 2-MAR-2007 20:33:
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFRQ 125.00 MHz
 OBSET 777.00 KHz
 OBFIN 875.5 Hz
 POINT 32768
 FREQU 31446.5 Hz
 SCANS 103
 ACQTM 1.042 sec
 PD 1.000 sec
 PW1 4.2 us
 IRNUC 1H
 CTEMP 24.5 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.05 Hz
 RGAIN 30

DFILE Ty-010-2-1H-1
 COMNT Single Pulse Expe
 DATIM 22-05-2007 13:22:
 1H
 EXMOD single_pulse.exp
 OBFRQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 16364
 FREQU 7507.51 Hz
 SCANS 8
 ACQTM 2.1823 sec
 PD 4.0000 sec
 PW1 7.50 usec
 IRNUC
 CTEMP 22.3 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.23 Hz
 RGAIN 18

2m

DFILE Ty-010-2-13C-1
 COMNT Single Pulse with Br
 DATIM 22-05-2007 13:35:4
 13C
 EXMOD single_pulse_dec
 OBFRQ 125.77 MHz
 OBSET 7.87 KHz
 OBFIN 4.21 Hz
 POINT 32768
 FREQU 31446.54 Hz
 SCANS 354
 ACQTM 1.0420 sec
 PD 1.0000 sec
 PW1 4.17 usec
 IRNUC 1H
 CTEMP 25.5 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.93 Hz
 RGAIN 30

Single Pulse Experiment

DFILE C:\Windows\AD4_A\18\
 COMNT Single Pulse Experiment
 DATIM 16-MAR-2007 11:20:22
 OBNUC 1H
 EXMOD single_pulse.exp
 OBFRQ 500.00 MHz
 OBSET 162.00 KHz
 OBFIN 416.0 Hz
 POINT 16384
 FREQU 7507.5 Hz
 SCANS 8
 ACQTM 2.182 sec
 PD 4.000 sec
 PW1 7.5 us
 IRNUC 1H
 CTEMP 23.5 c
 SLVNT CDCL3
 EXREF 7.24 ppm
 BF 1.00 Hz
 RGAIN 13

2n

Single Pulse with Broadband Decoupling

DFILE C:\Windows\AD4_A\18\
 COMNT Single Pulse with Br
 DATIM 16-MAR-2007 11:26:16
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFRQ 125.00 MHz
 OBSET 777.00 KHz
 OBFIN 875.5 Hz
 POINT 32768
 FREQU 31446.5 Hz
 SCANS 147
 ACQTM 1.042 sec
 PD 1.000 sec
 PW1 4.2 us
 IRNUC 1H
 CTEMP 24.9 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.00 Hz
 RGAIN 30

20

Single Pulse Experiment

DFILE E:\tm-101-1h.als
COMNT Single Pulse Experim
DATIM 11-JAN-2007 16:24:48
OBNUC 1H
EXMOD single_pulse.exp
OBFRQ 500.00 MHz
OBSET 162.00 KHz
OBFIN 416.0 Hz
POINT 16384
FREQU 7507.5 Hz
SCANS 8
ACQTM 2.182 sec
PD 4.000 sec
FWL 7.5 us
IRNUC 1H
CTEMP 24.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 17

Single Pulse Experiment

DFILE E:\tm-149-1h.als
COMNT Single Pulse Experim
DATIM 12-JAN-2007 16:20:10
OBNUC 1H
EXMOD single_pulse.exp
OBFRQ 500.00 MHz
OBSET 162.00 KHz
OBFIN 416.0 Hz
POINT 16384
FREQU 7507.5 Hz
SCANS 8
ACQTM 2.182 sec
PD 4.000 sec
FWL 7.5 us
IRNUC 1H
CTEMP 24.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.23 Hz
RGAIN 14

Single Pulse Experiment

DFILE E:\tm-242-1H.als
 COMNT Single Pulse Experiment
 DATIM 16-JAN-2007 18:21:47
 OBNUC 1H
 EXMOD single pulse.exp
 OBFRQ 500.00 MHz
 OBSET 162.00 KHz
 OBFIN 416.0 Hz
 POINT 16384
 FREQU 7507.5 Hz
 SCANS 8
 ACQTM 2.182 sec
 PD 4.000 sec
 FW1 7.5 us
 IRNUC 1H
 CTEMP 24.6 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.23 Hz
 RGAIN 17

3c

Single Pulse Experiment

DFILE E:\tm-070111-1H.als
 COMNT Single Pulse Experiment
 DATIM 11-JAN-2007 16:38:51
 OBNUC 1H
 EXMOD single pulse.exp
 OBFRQ 500.00 MHz
 OBSET 162.00 KHz
 OBFIN 416.0 Hz
 POINT 16384
 FREQU 7507.5 Hz
 SCANS 8
 ACQTM 2.182 sec
 PD 4.000 sec
 FW1 7.5 us
 IRNUC 1H
 CTEMP 25.2 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.23 Hz
 RGAIN 14

4a

DFILE	E:\tm-070118-1H.als
COMNT	Single Pulse Experiment
DATEI	18-JAN-2007 14:48:25
OBSCN	1H
EXPNO	single pulse.exp
OBFRQ	500.00 MHz
OBSET	162.00 KHz
OBFIN	416.0 Kz
POINT	16384
FREQU	7507.5 Hz
SCANS	8
ACQTM	2.182 sec
PD	4.000 sec
FW1	7.5 us
IRNUC	1H
CTEMP	25.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.23 Hz
RGAIN	21

```

PFILE C:\Windows\AP\A\18\atm-246
CNAME Single Pulse Experiment
DATE_ 10-MAR-2007 16:16:05
ORIGIN 1H
EXMOD single_pulse.exp
OBFREQ 500.00 MHz
OBSET 162.00 KHz
OBFIN 416.0 Hz
POINT 16384
FREQU 7507.5 Hz
SCANS 8
ACQTM 2.182 sec
PD 4.000 sec
PW1 7.5 us
IRNUC 1H
CTEMP 22.9 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.23 Hz
RGAIN 15

```

Single Pulse Experiment

DFILE E:\tm-290-2-1H.al
COMNT Single Pulse Expe
DATIM 24-FEB-2007 17:41
OBNOC 1H
EXMOD single_pulse.exp
OBFRQ 500.00 MHz
OBSET 162.00 KHz
OBFIN 416.0 Hz
POINT 16384
FREQU 7507.5 Hz
SCANS 8
ACQTM 2.182 sec
PD 4.000 sec
PW1 7.5 us
IRNUC 1H
CTEMP 23.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.23 Hz
RGAIN 17

5a

DFILE KP-7-1H.1
COMNT Single Pulse Experi
DATIM 02-06-2007 18:45:31
OBNOC 1H
EXMOD single_pulse.exp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 7507.61 Hz
SCANS 8
ACQTM 2.1823 sec
PD 4.0000 sec
PW1 7.50 usec
IRNUC 1H
CTEMP 23.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.93 Hz
RGAIN 12

5b

Single Pulse Experiment

DFILE C:\Windows\AD4_A\18\tr
 COMNT Single Pulse Experiment
 DATIM 8-MAR-2007 18:55:52
 OBNUC 1H
 EXMOD single_pulse.exp
 OBFRQ 500.00 MHz
 OBSET 162.00 KHz
 OBFIN 416.0 Hz
 POINT 16384
 FREQU 7507.5 Hz
 SCANS 8
 ACQTM 2.182 sec
 PD 4.000 sec
 PW1 7.5 us
 IRNUC 1H
 CTEMP 23.4 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.23 Hz
 RGAIN 18

6c

Single Pulse with Broadband Decoupling

DFILE C:\Windows\AD4_A\18\tr
 COMNT Single Pulse with B
 DATIM 8-MAR-2007 19:00:4
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFRQ 125.00 MHz
 OBSET 777.00 KHz
 OBFIN 875.5 Hz
 POINT 32768
 FREQU 31446.5 Hz
 SCANS 111
 ACQTM 1.042 sec
 PD 1.000 sec
 PW1 4.2 us
 IRNUC 1H
 CTEMP 25.5 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.00 Hz
 RGAIN 30

6c

Single Pulse Experiment

DFILE E:\CX'J\m-273-4-1H.a
 COMNT Single Pulse Experime
 DATIM 2-FEB-2007 15:46:37
 OBNUC 1H
 EXMOD single_pulse.exp
 OBFRQ 500.00 MHz
 OBSET 162.00 KHz
 OBFIN 416.0 Hz
 POINT 16384
 FREQU 7507.5 Hz
 SCANS 8
 ACQTM 2.182 sec
 PD 4.000 sec
 PW1 7.5 us
 IRNUC 1H
 CTEMP 25.9 c
 SLVNT CDCL3
 EXREF 7.24 ppm
 BF 0.23 Hz
 RGAIN 18

Single Pulse with Broadband Decoupling

DFILE E:\tm-273-5-13C.a
 COMNT Single Pulse with
 DATIM 3-FEB-2007 09:58
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFRQ 125.00 MHz
 OBSET 777.00 KHz
 OBFIN 875.5 Hz
 POINT 32768
 FREQU 31446.5 Hz
 SCANS 202
 ACQTM 1.042 sec
 PD 1.000 sec
 PW1 4.2 us
 IRNUC 1H
 CTEMP 26.9 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.50 Hz
 RGAIN 30