Supporting Information
for DOI: 10.1055/s-0039-1690236
© 2019. Thieme. All rights reserved.
Georg Thieme Verlag KG, Rüdigerstraße 14, 70469 Stuttgart, Germany
Supporting Information

An Efficient Deprotection of 2,6-Bis(trifluoromethyl)phenylboronic Esters via Catalytic Protodeboronation using Tetrabutylammonium Fluoride

Sari Urata,† Shinya Nojima,† Kazuishi Makino† and Naoyuki Shimada *†

†Laboratory of Organic Chemistry for Drug Development and Medical Research Laboratories, Department of Pharmaceutical Sciences, Kitasato University, Tokyo 108-8641, Japan

Table of Contents

1. General information S2
2. Deprotection of o-FFylboronic ester using stoichiometric amount of TBAF S2
3. Catalytic deprotection of o-FFylboronic esters and characterization data for compounds 2a-m S3
4. 1H-NMR studies for the catalytic deprotection of 1a using TBAF (Figure 1) S10
5. Deprotection of o-FFylboronic ester 1a in the presence of D2O (Scheme 4) S11
6. Deprotection of phenylboronic ester using TBAF S12
7. 1H and 13C NMR spectra S13
8. Reference S39
1. General information

NMR spectra were recorded on Agilent Technologies 400-MR DD2 (400 MHz for $^1$H, 100 MHz for $^{13}$C), 400-MR (400 MHz for $^1$H, 100 MHz for $^{13}$C) spectrometers. $^1$H-NMR data are reported as follows; chemical shift in parts per million (ppm) downfield or upfield from CDCl$_3$ ($\delta$ 7.26), CD$_3$OD ($\delta$ 3.31) integration, multiplicity (br = broad, s = singlet, d = doublet, t = triplet, dd = double doublet, ddd = double double doublet, ddt = double double triplet, dq = double quintet, and m = multiplet), and coupling constants (Hz). $^{13}$C-NMR chemical shifts are reported in ppm downfield or upfield from CDCl$_3$ ($\delta$ 77.0) or CD$_3$OD ($\delta$ 49.0). Mass spectra were measured with JEOL JMS-AX505HA, JMS-700 MStation, and JEOL JMS-T100LP spectrometers. Thin-layer chromatography (TLC) was carried out on Merck 60F-254 precoated silica gel plates and were visualized by fluorescence quenching under UV light. TBAF ($ca$. 1 mol/L in tetrahydrofuran including maximum 10% of water) was purchased from TOKYO CHEMICAL INDUSTRY CO., LTD. 0.20 M solution of TBAF in THF was prepared by diluting 1.0 M solution of TBAF with THF (1.0 mL) in THF (4.0 mL). Amino silica gel (NH, DM1020, HU50100) was purchased from Fuji Silysia Chemical LTD. Column chromatography was performed using Silica Gel 60N (spherical, neutral, 63-210 $\mu$m) (Kanto Chemical Co., Inc.). $\omega$-FXylboronic esters 1a-m were synthesized according to the literature$^1$.

2. Deprotection of $\omega$-FXylboronic ester using stoichiometric amount of TBAF

6-(Benzylxy)hexane-1,3-diol (2a)

\[
\begin{align*}
\text{TBAF (120 mo\%)} & \quad \text{THF (0.1 M)} \\
& \quad \text{rt, 10 min}
\end{align*}
\]

TBAF (1.0 M in THF, 0.24 mL, 0.240 mmol, 120 mol%) was added to a solution of 1a (89.2 mg, 0.200 mmol, 1.0 equiv) in dry THF (1.8 mL, total 0.10 M) at room temperature. After stirring for 2 h under reflux, cooling to room temperature, the reaction mixture was filtered through a short pad of amino silica gel (800 mg) eluting with EtOAc (20 mL), and the filtrate was concentrated under reduced pressure to give 2a (44.8 mg, 0.200 mmol, >99% yield) as a colorless oil.
3. Catalytic deprotection of \( \alpha \)-FXylboronic esters using TBAF

6-(Benzyloxy)hexane-1,3-diol (2a)

![Diagram of the reaction](image)

TBAF (0.20 M in THF, 100 \( \mu \)L, 0.0200 mmol, 10 mol\%) and \( \text{H}_2\text{O} \) (6.0 M in THF, 100 \( \mu \)L, 0.600 mmol, 3.0 equiv) were added to a solution of \( 1\text{a} \) (89.2 mg, 0.200 mmol, 1.0 equiv) in dry THF (1.8 mL, total 0.10 M) at room temperature. After stirring for 2 h under reflux, cooling to room temperature, the reaction mixture was filtered through a short pad of amino silica gel (800 mg) eluting with EtOAc (20 mL), and the filtrate was concentrated under reduced pressure to give \( 2\text{a} \) (46.8 mg, 0.200 mmol, >99% yield) as a colorless oil.

Data for \( 2\text{a} \): \( R_f 0.13 \) (4/1 n-hexane/EtOAc); \(^1\)H-NMR (400 MHz, CDCl\(_3\)) \( \delta 7.38-7.27 \) (m, 5H), 4.53 (s, 2H), 3.90-3.79 (m, 3H), 3.57-3.49 (m, 2H), 2.34 (brs, 2H), 1.79-1.52 (m, 6H); \(^1\)C NMR (100 MHz, CDCl\(_3\)) \( \delta 137.9, 128.4, 127.8, 127.7, 73.1, 71.9, 70.5, 61.7, 38.3, 35.2, 26.2 \); IR (neat) \( \nu = 3372, 2942, 2865, 1278, 1099 \) cm\(^{-1}\); HRMS (ESI) m/z Calcd for \( \text{C}_{13}\text{H}_{20}\text{O}_3\text{Na} \) [M+Na]\(^+\) 247.1310, found 247.1311.

6-(benzyloxy)hexane-1,3-diol (2b)

![Diagram of the reaction](image)

TBAF (0.20 M in THF, 100 \( \mu \)L, 0.0200 mmol, 10 mol\%) and \( \text{H}_2\text{O} \) (6.0 M in THF, 100 \( \mu \)L, 0.600 mmol, 3.0 equiv) were added to a solution of \( 1\text{b} \) (79.6 mg, 0.200 mmol, 1.0 equiv) in dry THF (1.8 mL, total 0.10 M) at room temperature. After stirring for 1.5 h under reflux, cooling to room temperature, the reaction mixture was filtered through a short pad of amino silica gel (800 mg) eluting with EtOAc (20 mL), and the filtrate was concentrated under reduced pressure to give \( 2\text{a} \) (33.2 mg, 0.188 mmol, 94% yield) as a colorless oil.

Data for \( 2\text{b} \): \( R_f 0.13 \) (4/1 n-hexane/EtOAc); \(^1\)H-NMR (400 MHz, CDCl\(_3\)) \( \delta 4.10 \) (t, \( J = 6.8 \) Hz, 2H), 3.93-3.80 (m, 3H), 2.26 (s, 2H), 2.05 (s, 3H), 1.83-1.65 (m, 4H), 1.57-1.52 (m, 2H); \(^1\)C NMR (100 MHz, CDCl\(_3\)) \( \delta 137.9, 128.4, 127.8, 127.7, 73.1, 71.9, 70.5, 61.7, 38.3, 35.2, 26.2 \); IR (neat) \( \nu = 3372, 2942, 2865, 1278, 1099 \) cm\(^{-1}\); HRMS (ESI) m/z Calcd for \( \text{C}_{13}\text{H}_{20}\text{O}_3\text{Na} \) [M+Na]\(^+\) 247.1310, found 247.1311.
MHz, CDCl$_3$) δ 171.3, 71.6, 64.4, 61.8, 38.3, 34.0, 24.8, 21.0; IR (neat) ν = 3384, 2940, 1732, 1254, 1050 cm$^{-1}$; HRMS (EI) m/z Calcd for C$_8$H$_{17}$O$_4$ [M+H]$^+$ 177.1127, found 177.1122.

**N-benzyl-4,6-dihydroxyhexanamide (2c)**

![Chemical structure of 2c]

TBAF (0.20 M in THF, 100 µL, 0.0200 mmol, 10 mol%) and H$_2$O (6.0 M in THF, 100 µL, 0.600 mmol, 3.0 equiv) were added to a solution of 1c (91.8 mg, 0.200 mmol, 1.0 equiv) in dry THF (1.8 mL, total 0.10 M) at room temperature. After stirring for 1.5 h under reflux, cooling to room temperature, the reaction mixture was filtered through a short pad of amino silica gel (800 mg) eluting with EtOAc (20 mL), and the filtrate was concentrated under reduced pressure to give 2c (44.6 mg, 0.188 mmol, 94% yield) as a colorless oil.

Data for 2c: R$_f$ 0.43 (10/1 CH$_2$Cl$_2$/MeOH); $^1$H-NMR (400 MHz, CD$_3$OD) δ 7.33-7.21 (m, 5H), 4.36 (s, 2H), 3.75-3.67 (m, 3H), 2.44-2.29 (m, 2H), 1.87-1.78 (m, 1H), 1.74-1.59 (m, 3H); $^{13}$C NMR (100 MHz, CD$_3$OD) δ 176.0, 140.1, 129.5, 128.6, 128.2, 69.4, 60.2, 44.1, 40.7, 34.6, 33.4; IR (neat) ν = 3308, 2927, 1645, 1553, 1454, 1359, 1261, 1058 cm$^{-1}$; HRMS (EI) m/z Calcd for C$_{13}$H$_{19}$NO$_3$Na [M+Na]$^+$ 260.1263, found 260.1262.

**6-(benzylamino)hexane-1,3-diol (2d)**

![Chemical structure of 2d]

TBAF (0.20 M in THF, 100 µL, 0.0200 mmol, 10 mol%) and H$_2$O (6.0 M in THF, 100 µL, 0.600 mmol, 3.0 equiv) were added to a solution of 1d (89.0 mg, 0.200 mmol, 1.0 equiv) in dry THF (1.8 mL, total 0.10 M) at room temperature. After stirring for 2 h under reflux, cooling to room temperature, the reaction mixture was filtered through a short pad of amino silica gel (800 mg) eluting with EtOAc (20 mL), and the filtrate was concentrated under reduced pressure to give 2d (40.7 mg, 0.182 mmol, 91% yield) as a colorless oil.

Data for 2d: R$_f$ 0.24 (5/1 CH$_2$Cl$_2$/MeOH); $^1$H-NMR (400 MHz, CDCl$_3$) δ 7.36-7.23 (m, 5H), 3.86-3.80 (m, 2H), 3.79 (s, 2H), 3.23 (brs, 2H), 2.85 (ddd, J = 12.0, 6.0, 3.2 Hz, 1H), 2.60 (ddd, J = 12.0, 6.0, 3.2 Hz, 1H).
12.0, 9.2, 2.8 Hz, 1H), 1.87-1.48 (m, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 138.4, 128.6, 128.4, 127.5, 72.4, 62.2, 53.6, 49.2, 38.4, 37.7, 27.3; IR (neat) $\nu$ = 3343, 2931, 1496, 1454, 1058, 744 cm$^{-1}$; HRMS (ESI) m/z Calcd for C$_{13}$H$_{22}$NO$_2$ [M+H]$^+$ 224.1651, found 224.1641.

**Hept-6-ene-1,3-diol (2e)**

TBAF (0.20 M in THF, 100 µL, 0.0200 mmol, 10 mol%) and H$_2$O (6.0 M in THF, 100 µL, 0.600 mmol, 3.0 equiv) were added to a solution of 1e (70.4 mg, 0.200 mmol, 1.0 equiv) in dry THF (1.8 mL, total 0.10 M) at room temperature. After stirring for 2 h under reflux, cooling to room temperature, the reaction mixture was filtered through a short pad of amino silica (800 mg) eluting with EtOAc (20 mL), and the filtrate was concentrated under reduced pressure to give 2e (27.4 mg, 0.200 mmol, 99% yield) as a colorless oil.

**Hexane-1,3,6-triol (2f)**

TBAF (0.20 M in THF, 100 µL, 0.0200 mmol, 10 mol%) and H$_2$O (6.0 M in THF, 100 µL, 0.600 mmol, 3.0 equiv) were added to a solution of 1f (71.2 mg, 0.200 mmol, 1.0 equiv) in dry THF (1.8 mL, total 0.10 M) at room temperature. After stirring for 6 h under reflux, cooling to room temperature, the reaction mixture was filtered through a short pad of amino silica gel (800 mg) eluting with MeOH (20 mL). Concentration under reduced pressure furnished the crude product, which was purified by silica gel column chromatography (CH$_2$Cl$_2$/MeOH = 9:1) to give 2f (21.4 mg, 0.159 mmol, 80% yield) as a colorless oil.
Data for 2f: Rf 0.24 (10/1 CH₂Cl₂/Methanol); ^1^H-NMR (400 MHz, CD₃OD) δ 3.76-3.68 (m, 3H), 3.57 (t, J = 6.4 Hz, 2H), 1.75-1.42 (m, 6H); ^1^C NMR (100 MHz, CD₃OD) δ 69.8, 63.0, 60.3, 40.8, 35.1, 29.8; IR (neat) ν = 3343, 2942, 1052 cm⁻¹; HRMS (ESI) m/z Calcd for C₆H₁₄O₃Na [M+Na]^+ 157.0841, found 157.0843.

6-((tert-butyldimethylsilyl)oxy)hexane-1,3-diol (2g)

TBAF (0.20 M in THF, 100 µL, 0.0200 mmol, 10 mol%) and H₂O (6.0 M in THF, 100 µL, 0.600 mmol, 3.0 equiv) were added to a solution of 1g (94.1 mg, 0.200 mmol, 1.0 equiv) in dry THF (1.8 mL, total 0.10 M) at room temperature. After stirring for 2 h under reflux, cooling to room temperature, the reaction mixture was filtered through a short pad of amino silica gel (800 mg) eluting with EtOAc (20 mL), and the filtrate was concentrated under reduced pressure to give 2g (43.6 mg, 0.175 mmol, 88% yield) as a colorless oil.

Data for 2g: Rf 0.24 (2/1 n-hexane/EtOAc); ^1^H-NMR (400 MHz, CDCl₃) δ 3.90-3.79 (m, 3H), 3.73-3.62 (m, 2H), 2.74 (brs, 2H), 1.74-1.54 (m, 6H), 0.90 (s, 9H), 0.07 (s, 6H); ^1^C NMR (100 MHz, CDCl₃) δ 72.3, 63.6, 62.0, 38.4, 35.6, 29.2, 25.9, 18.3, -5.44, -5.46; IR (neat) ν = 3359, 2930, 2858, 1255, 1096, 1006, 836 cm⁻¹; HRMS (ESI) m/z Calcd for C₁₂H₂₅O₃SiNa [M+Na]^+ 271.1702, found 271.1705.

2,2-Dimethylpropane-1,3-diol (2h)

TBAF (0.20 M in THF, 100 µL, 0.0200 mmol, 10 mol%) and H₂O (6.0 M in THF, 100 µL, 0.600 mmol, 3.0 equiv) were added to a solution of 1h (65.2 mg, 0.200 mmol, 1.0 equiv) in dry THF (1.8 mL, total 0.10 M) at room temperature. After stirring for 0.5 h under reflux, cooling to room temperature, the reaction mixture was filtered through a short pad of amino silica gel (800 mg) eluting with EtOAc (20 mL), and the filtrate was concentrated under reduced pressure to give 2h (24.5 mg, 0.200 mmol, 99% yield) as a white solid.
Data for 2h: Rf 0.37 (1/2 n-hexane/EtOAc); 1H-NMR (400 MHz, CDCl3) δ 3.49 (s, 4H), 2.53 (s, 2H), 0.90 (s, 6H); 13C NMR (100 MHz, CDCl3) δ 71.6, 36.4, 21.3; IR (neat) ν = 3371, 2959, 2926, 2873, 1652, 1471, 1261 cm⁻¹; HRMS (EI) m/z Calcd for C₅H₁₂O₂ [M]⁺ 104.0837, found 104.0835.

1-Phenylethane-1,2-diol (2i)

TBAF (0.20 M in THF, 100 µL, 0.0200 mmol, 10 mol%) and H₂O (6.0 M in THF, 100 µL, 0.600 mmol, 3.0 equiv) were added to a solution of 1i (72.0 mg, 0.200 mmol, 1.0 equiv) in dry THF (1.8 mL, total 0.10 M) at room temperature. After stirring for 2 h under reflux, cooling to room temperature, the reaction mixture was filtered through a short pad of amino silica gel (800 mg) eluting with EtOAc (20 mL), and the filtrate was concentrated under reduced pressure to give 2i (24.2 mg, 0.175 mmol, 88% yield) as a white solid.

Data for 2i: Rf 0.47 (2/3 n-hexane/EtOAc); 1H-NMR (400 MHz, CDCl₃) δ 7.39-7.29 (m, 5H), 4.83 (dd, J = 8.4, 3.6 Hz, 1H), 3.78 (dd, J = 11.2, 3.6 Hz, 1H), 3.67 (dd, J = 11.2, 8.4 Hz, 1H), 2.03 (brs, 2H); 13C NMR (100 MHz, CDCl₃) δ 140.4, 128.5, 128.0, 126.0, 74.7, 68.0; IR (KBr) ν = 3204, 2963, 2931, 1448, 1262, 1101, 1054 cm⁻¹; HRMS (EI) m/z Calcd for C₈H₁₀O₂ [M]⁺ 138.0681, found 138.0681.

(1R,2S)-1,2-Diphenylethane-1,2-diol (2j)

TBAF (0.20 M in THF, 100 µL, 0.0200 mmol, 10 mol%) and H₂O (6.0 M in THF, 100 µL, 0.600 mmol, 3.0 equiv) were added to a solution of 1j (88.2 mg, 0.200 mmol, 1.0 equiv) in dry THF (1.8 mL, total 0.10 M) at room temperature. After stirring for 5 h under reflux, cooling to room temperature, the reaction mixture was filtered through a short pad of amino silica gel (800 mg) eluting with EtOAc (20 mL), and the filtrate was concentrated under reduced pressure to give 2j (43.9 mg, 0.200 mmol, 99% yield) as a white solid.

Data for 2j: Rf 0.27 (3/1 n-hexane/EtOAc); 1H-NMR (400 MHz, CDCl₃) δ 7.33-7.24 (m, 10H), 4.84
(s, 2H), 2.18 (s, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 139.7, 128.2, 128.1, 127.1, 78.0; IR (KBr) $\nu$ = 3375, 3312, 2899, 1451, 1279, 1034 cm$^{-1}$; HRMS (EI) m/z Calcd for C$_{14}$H$_{14}$O$_2$ [M]$^+$ 214.0994, found 214.0999.

**1R,2S-Cyclopentane-1,2-diol (2k)**

![Chemical Structure](attachment:1k.png)

TBAF (0.20 M in THF, 100 µL, 0.0200 mmol, 10 mol%) and H$_2$O (6.0 M in THF, 100 µL, 0.600 mmol, 3.0 equiv) were added to a solution of 1k (64.8 mg, 0.200 mmol, 1.0 equiv) in dry THF (1.8 mL, total 0.10 M) at room temperature. After stirring for 0.5 h under reflux, cooling to room temperature, the reaction mixture was filtered through a short pad of amino silica gel (800 mg) eluting with EtOAc (20 mL), and the filtrate was concentrated under reduced pressure to give 2k (18.3 mg, 0.179 mmol, 90% yield) as a colorless oil.

Data for 2k: R$_f$ 0.38 (1/3 n-hexane/EtOAc); $^1$H-NMR (400 MHz, CDCl$_3$) $\delta$ 4.06-4.02 (m, 2H), 2.27 (s, 2H), 1.91-1.79 (m, 3H), 1.71-1.62 (m, 2H), 1.54-1.44 (m, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 73.1, 30.1, 26.2, 23.7; IR (neat) $\nu$ = 3390, 2962, 2925, 1344, 1298, 1261, 1200, 1092 cm$^{-1}$; HRMS (EI) m/z Calcd for C$_5$H$_{10}$O$_2$ [M]$^+$ 102.0681, found 102.0672.

**1R,2S-Cyclohexane-1,2-diol (2l)**

![Chemical Structure](attachment:1l.png)

TBAF (0.20 M in THF, 100 µL, 0.0200 mmol, 10 mol%) and H$_2$O (6.0 M in THF, 100 µL, 0.600 mmol, 3.0 equiv) were added to a solution of 1l (67.6 mg, 0.200 mmol, 1.0 equiv) in dry THF (1.8 mL, total 0.10 M) at room temperature. After stirring for 20 h under reflux, cooling to room temperature, the reaction mixture was filtered through a short pad of amino silica gel (800 mg) eluting with EtOAc (20 mL), and the filtrate was concentrated under reduced pressure to give 2l (24.3 mg, 0.200 mmol, 99% yield) as a white solid.

Data for 2l: R$_f$ 0.46 (1/3 n-hexane/EtOAc); $^1$H-NMR (400 MHz, CDCl$_3$) $\delta$ 3.78-3.76 (m, 2H), 1.93 (s, 2H), 1.79-1.72 (m, 2H), 1.66-1.52 (m, 4H), 1.35-1.25 (m, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 70.6,
29.9, 21.4; IR (KBr) ν = 3399, 3267, 2931, 2865, 1350, 1260, 1116, 1076 cm⁻¹; HRMS (EI) m/z Calcd for C₆H₁₂O₂ [M]+ 116.1587, found 116.0832.

**(1R,2S)-Cyclooctane-1,2-diol (2m)**

TBAF (0.20 M in THF, 100 µL, 0.0200 mmol, 10 mol%) and H₂O (6.0 M in THF, 100 µL, 0.600 mmol, 3.0 equiv) were added to a solution of 1m (73.2 mg, 0.200 mmol, 1.0 equiv) in dry THF (1.8 mL, total 0.10 M) at room temperature. After stirring for 1 h under reflux, cooling to room temperature, the reaction mixture was filtered through a short pad of amino silica gel (800 mg) eluting with EtOAc (20 mL), and the filtrate was concentrated under reduced pressure to give 2m (28.9 mg, 0.200 mmol, 99% yield) as a colorless solid.

Data for 2m: Rₐ 0.49 (1/2 n-hexane/EtOAc); ¹H-NMR (400 MHz, CDCl₃) δ 3.90 (brd, J = 10.0 Hz, 2H), 1.97-1.86 (m, 4H), 1.69-1.64 (m, 4H), 1.56-1.48 (m, 6H); ¹³C NMR (100 MHz, CDCl₃) δ 73.1, 30.1, 26.2, 23.7; IR (KBr) ν = 3388, 3277, 2925, 2855, 1459, 1260, 1099 cm⁻¹; HRMS (EI) m/z Calcd for C₆H₁₀O₂ [M]+ 144.1150, found 144.1148.
4. $^1$H-NMR studies for the catalytic deprotection of 1a using TBAF (Figure 1)

- **1a**
  - TBAF (10 mol%)
  - H$_2$O (3.0 equiv)
  - THF-d$_8$ (0.1 M)
  - reflux, 2 h

filtration through a pad of silica gel (amino silica gel)

then evaporation

$^1$H-NMR of reaction mixture in THF-d$_8$

$^1$H-NMR of crude mixture in THF-d$_8$
4. Deprotection of \( \sigma \)-FXylboronic ester 1a in the presence of D\(_2\)O (Scheme 4)

\[
\begin{align*}
\text{TBAF (10 mol\%)} & \quad \text{D}_2\text{O (3.0 equiv)} \\
\text{THF-d}_8 (0.1 M) & \quad \text{reflux, 2 h}
\end{align*}
\]

H\(_c\) : H\(_b\) = 1:2
H\(_a\) = 1H
H\(_a\) + 2H\(_b\) = 2.22H
Therefore
H\(_a\) = 0.22H
D\(_a\) = 0.78D
5. Deprotection of phenylboronic ester using TBAF

\[
\begin{align*}
\text{HO} & \quad \text{OH} \quad \text{OBn} \\
\text{HO} & \quad \text{OH} \quad \text{OBn} \\
\end{align*}
\]

TBAF (10 mol%) 
H\textsubscript{2}O (3.0 equiv) 
THF-\textsubscript{d}_8 (0.1 M) 
rt, 24 h

\[
\begin{align*}
\text{HO} & \quad \text{OH} \\
\text{HO} & \quad \text{OH} \\
\text{HO} & \quad \text{OH} \\
\end{align*}
\]

reaction mixture

\[
\begin{align*}
\text{HO} & \quad \text{OH} \\
\text{HO} & \quad \text{OH} \\
\end{align*}
\]

SM: 2a = 84:16
6. $^1$H and $^{13}$C NMR spectra
Comment  su55021pure13C_201009.8
Date  2010/Sep/18
ObsNuc  13C
ExMode  CAMEBON.001
ObsFreq  100.46 MHz
Scan  612
AcqLine  1.3081 s
Acc. Interval  3.3081 s
Spinning  20.0 Hz
Temperature  25.0 °C
Solvent  dcl3
Comment: au-trial_20180730_01
Date: 2018/Jul/30
ObsNuc: 13C
Exp.Mode: CARBON
ObsFreq: 100.66 MHz
Scan: 410
Acq.Time: 1.3331 s
Acc. Interval: 2.3331 s
Spinning: 20.0 Hz
Temperature: 40.0 °C
Solvent: chde
Comment: au26000_10100008_02
Date: 2010/Dec/30
Hardware: NMR
Ex-Mode: PROTON
ObaFreq: 400.27 MHz
Scan: 8
AcqTime: 2.5555 µs
Acc. Interval: 2.5555 µs
Spinning: 16.0 Hz
Temperature: 40.0 °C
Solvent: cdc6
Comment: au26010pere150_36100017_01
Date: 2019/Sept/17
ObaNucl: 140C
Experiments: CARBON_13C
ObaFreq: 100.45 MHz
Scan: 612
AcqTime: 1.3631 s
Acc. Interval: 3.3651 s
Spinning: 20.0 Hz
Temperature: 25.0 °C
Solvent: cdcl3
7. Reference