Supporting Information

Palladium-catalyzed cross-coupling reaction of triorganoindium reagents with propargylic esters

Ricardo Riveiros, David Rodríguez, José Pérez Sestelo,* and Luis A. Sarandeses*

Departamento de Química Fundamental, Universidade da Coruña, E-15071 A Coruña, Spain

Contents

General experimental procedures	S1–S3
Data for compounds prepared	S3–S8
Copies of NMR spectra	S9–S52

General Considerations.¹ NMR spectra were recorded in CDCl₃ at 200 MHz or 300 MHz for ¹H and at 50 MHz or 75 MHz for ¹³C. DEPT was used to assign carbon multiplicity. The electron-impact mass spectra were measured at 70 eV.

Propargylic acetates and benzoates (**1a–b**, **3b**, **15a–b**, and **17**) were prepared from the corresponding alcohols by treatment with acetyl or benzoyl chloride in pyridine, in the presence of a catalytic amount of DMAP.² Carbonate **1c** was prepared by the reaction of the corresponding lithium alkoxide with methyl chloroformate.³

-
- (1) For General Experimental Methods, see: Rodríguez, D.; Pérez Sestelo, J.; Sarandeses, L. A. *J. Org. Chem.* **2003**, *68*, 2518–2520.
 - (2) Höffe, G.; Steglich, W.; Vorbrüggen, H. *Angew. Chem. Int. Ed.* **1978**, *17*, 569–583.
 - (3) Matsuda, I.; Komori, K. -I.; Itoh, K. *J. Am. Chem. Soc.* **2002**, *124*, 9072–9073.

Triorganoindium Reagents. Triorganoindium compounds were prepared according to previously published methods⁴ by treatment of the corresponding organolithium or Grignard reagents (3 equiv) with InCl₃ (1.1 equiv) in dry THF at –78 °C and warming to room temperature. In this procedure triphenyl-, trimethyl-, tributyl-, tris[(trimethylsilyl)ethynyl]- and tri(phenylethynyl)indium were prepared from the corresponding organolithium reagents and trivinylindium and tri(2-methoxyphenyl)indium were prepared from vinylmagnesium bromide and 2-methoxyphenylmagnesium bromide, respectively. All organolithium and Grignard solutions were purchased from Aldrich and used as received, except (trimethylsilyl)ethynyl- and (phenylethynyl)lithium which were prepared, prior to use, by metallation of (trimethylsilyl)acetylene and phenylacetylene respectively with *n*-BuLi in dry THF at –78 °C, and warming to room temperature.

General Procedure for the Palladium-Catalyzed Cross-Coupling Reaction of Propargylic Esters with Indium(III) Organometallics. To a suspension of Pd(DPEphos)Cl₂ (14.3 mg, 0.02 mmol) and the propargylic ester (1 mmol) in dry THF (7 mL) was slowly added a solution of R₃In (1.2 mmol, approx 0.24 M in dry THF) and the resulting solution was stirred at room temperature for 8–10 h. The reaction was quenched by addition of a few drops of MeOH and the mixture was concentrated. The residue was purified by flash chromatography (hexanes) affording, after concentration and high-vacuum drying, the corresponding allenes.

(4) (a) Pérez, I.; Pérez Sestelo, J.; Sarandeses, L. A. *Org. Lett.* **1999**, *1*, 1267–1269. (b) Pérez, I.; Pérez Sestelo, J.; Sarandeses, L. A. *J. Am. Chem. Soc.* **2001**, *123*, 4155–4160.

¹H NMR determination of the enantiomeric excess when chiral, non racemic allenes are prepared. A solution of the allene (**16** or **18**, ~0.03 mmol) in CDCl₃ (1 mL) with (+)-Yb(hfbc)₃ (1 equiv), and Ag(fod) (1 equiv) was prepared at room temperature. Under these conditions, the ¹H NMR was recorded showing that the signals at 6.58 and 6.09 ppm (allenic protons for **16** and **18**, respectively) appear splitted as two broad singlets. The enantiomeric excess was measured by integration. See copy of ¹H NMR attached.

¹ H NMR (200 MHz) δ		
	(<i>R</i>)	(<i>S</i>)
16	6.92 ppm	6.84 ppm
18	6.76 ppm	6.57 ppm

2-Phenyl-3-butyn-2-yl benzoate (1a).⁵ ¹H NMR (CDCl₃, 300 MHz) δ 2.07 (s, 3 H), 2.88 (s, 1 H), 7.27–7.69 (m, 8 H), 8.07 (m, 2 H); ¹³C NMR (CDCl₃, 75 MHz) δ 32.2 (CH₃), 75.8 (C), 75.9 (CH), 82.9 (C), 124.8 (2 $\times$ CH), 128.0 (CH), 128.4 (2 $\times$ CH), 128.3 (2 $\times$ CH), 129.7 (2 $\times$ CH), 130.4 (C), 133.1 (CH), 142.1 (C), 164.2 (C); MS (EI, 70 eV) *m/z* 250 (M⁺, 2), 128 (64), 105 (C₇H₅O⁺, 100), 77 (C₆H₅⁺, 39).

2-Phenyl-3-butyn-2-yl acetate (1b).⁶ ¹H NMR (CDCl₃, 300 MHz) δ 1.91 (s, 3 H), 2.09 (s, 3 H), 2.82 (s, 1 H), 7.27–7.40 (m, 3 H), 7.60 (d, *J* = 6.9 Hz, 2 H); ¹³C NMR (CDCl₃, 75 MHz) δ 21.7 (CH₃), 32.0 (CH₃), 75.3 (C), 75.5 (C), 82.9 (CH), 124.7 (2 $\times$ CH), 127.9 (CH), 128.4 (2 $\times$ CH), 142.1 (C), 168.6 (C); MS (EI, 70 eV) *m/z* 188 (M⁺, 3), 173 (M⁺ – CH₃, 2), 145 (M⁺ – C₂H₃O, 100).

(5) Olivero, S.; Duñach, E. *Tetrahedron Lett.* **1997**, 38, 6193–6196.

(6) Henke, E.; Bornscheuer, U. T.; Schmid, R. D.; Pleiss, J. *ChemBioChem* **2003**, 4, 485–493.

Methyl 2-phenyl-3-butyn-2-yl carbonate (1c). ¹H NMR (CDCl₃, 300 MHz) δ 1.94 (s, 3 H), 2.87 (s, 1 H), 3.73 (s, 3 H), 7.42–7.30 (m, 3 H), 7.65–7.61 (m, 2 H); ¹³C NMR (CDCl₃, 75 MHz) δ 32.0 (CH₃), 54.5 (CH₃), 76.1 (CH), 77.6 (C), 82.4 (C), 124.9 (2 $\times$ CH), 128.3 (CH), 128.4 (2 $\times$ CH), 141.4 (C), 153.2 (C); MS (EI, 70 eV) m/z 204 (M⁺, 2), 159 (M⁺ – CH₃, 1), 145 (47), 128 (100).

1,1'-(1-Methyl-1,2-propadiene-1,3-diyl)bisbenzene (2).⁷ ¹H NMR (CDCl₃, 300 MHz) δ 2.26 (d, J = 2.8 Hz, 3 H), 6.51 (d, J = 2.8 Hz, 1 H), 7.22–7.45 (m, 8 H), 7.49 (d, 2 H, J = 8.0 Hz); ¹³C NMR (CDCl₃, 75 MHz) δ 16.7 (CH₃), 96.6 (CH), 104.5 (C), 125.8 (2 $\times$ CH), 126.9 (2 $\times$ CH), 127.00 (CH), 127.02 (CH), 128.4 (2 $\times$ CH), 128.7 (2 $\times$ CH), 134.5 (C), 136.4 (C), 206.8 (C); MS (EI, 70 eV) m/z 206 (M⁺, 78), 191 (M⁺ – CH₃, 100), 77 (C₆H₅⁺, 30).

1-Ethynylcyclohexyl acetate (3b).⁸ ¹H NMR (CDCl₃, 300 MHz) δ 1.34 (m, 1 H), 1.52 (m, 1 H), 1.63 (m, 4 H), 1.85 (m, 2 H), 2.05 (s, 3 H), 2.13 (m, 2 H), 2.60 (s, 1H); ¹³C NMR (CDCl₃, 75 MHz) δ 21.9 (CH₃), 22.4 (2 $\times$ CH₂), 25.1 (CH₂), 36.9 (2 $\times$ CH₂), 74.2 (C), 75.1 (C), 83.7 (CH), 169.3 (C); MS (EI, 70 eV) m/z 166 (M⁺, 2), 159 (M⁺ – C₂H₂O, 63), 91 (100).

1-Methoxy-2-(3-phenyl-1,2-butadienyl)benzene (4).⁹ ¹H NMR (CDCl₃, 300 MHz) δ 2.23 (d, J = 2.7 Hz, 3 H), 3.88 (s, 3 H), 6.87–6.92 (m, 3 H), 7.17–7.49 (m, 7 H); ¹³C NMR (CDCl₃, 75 MHz) δ 16.8 (CH₃), 55.6 (CH₃), 90.5 (CH), 103.7 (C), 111.1 (CH), 120.8 (CH), 122.9 (C), 125.8 (2 $\times$ CH), 126.8 (CH), 127.9 (CH), 128.1 (CH), 128.4 (2 $\times$ CH), 136.7 (C), 156.2 (C), 207.3 (C); MS (EI, 70 eV) m/z 236 (M⁺, 48), 221 (M⁺ – CH₃, 74), 84 (100).

(7) Keinan, E.; Bosch, E. *J. Org. Chem.* **1986**, *51*, 4006–4016.

(8) Ishihara, K.; Kuboto, M.; Kurihara, H.; Yamamoto, H. *J. Am. Chem. Soc.* **1995**, *117*, 4413–4414.

(9) Yoshida, M.; Gotou, T.; Ihara, M. *Tetrahedron Lett.* **2004**, *45*, 5573–5575.

(1-Methyl-1,2,4-pentatrienyl)benzene (5).¹⁰ ¹H NMR (CDCl₃, 300 MHz) δ 2.1 (d, J = 3.0 Hz, 3 H), 5.05–5.09 (dd, J = 10.2, 1.7 Hz, 1 H), 5.26–5.32 (dd, J = 16.6, 1.7 Hz, 1 H), 6.13–6.38 (m, 2 H), 7.43–7.20 (m, 5 H); ¹³C NMR (CDCl₃, 75 MHz) δ 16.9 (CH₃), 96.5 (CH), 101.9 (C), 116.1 (CH₂), 125.9 (2 $\times$ CH), 126.9 (CH), 128.4 (2 $\times$ CH), 132.9 (CH), 136.6 (C), 208.4 (C); MS (EI, 70 eV) m/z 156 (M⁺, 7), 129 (M⁺ – C₂H₃, 32), 115 (100).

1,1'-(3,4-Hexadien-1-yn-1,5-diyl)bisbenzene (6). ¹H NMR (CDCl₃, 300 MHz) δ 2.20 Hz (d, J = 2.4 Hz, 3 H), 5.94 (q, J = 2.4 Hz, 1 H), 7.27–7.48 (m, 10 H); ¹³C NMR (CDCl₃, 75 MHz) δ 16.7 (CH₃), 77.9 (CH), 82.2 (C), 89.9 (C), 103.1 (C), 123.4 (C), 126.2 (2 $\times$ CH), 127.4 (CH), 128.1 (CH), 128.3 (2 $\times$ CH), 128.5 (2 $\times$ CH), 131.5 (2 $\times$ CH), 135.4 (C), 213.9 (C); MS (EI, 70 eV) m/z 230 (M⁺, 10), 191 (M⁺ – CH₃, 39), 83 (100).

(5-Phenyl-3,4-hexadien-1-ynyl)trimethylsilane (7). ¹H NMR (CDCl₃, 300 MHz) δ 0.21 (s, 9 H), 2.16 (d, J = 2.7 Hz, 3 H), 5.75 (q, J = 2.7 Hz, 1 H), 7.43 (m, 5 H); ¹³C NMR (CDCl₃, 75 MHz) δ 0.0 (3 $\times$ CH₃), 16.5 (CH₃), 77.8 (CH), 95.2 (C), 97.5 (C), 103.0 (C), 126.2 (2 $\times$ CH), 127.4 (CH), 128.4 (2 $\times$ CH), 135.3 (C), 214.2 (C); MS (EI, 70 eV) m/z 226 (M⁺, 6), 211 (M⁺ – CH₃, 31), 73 (100).

(1-Methyl-1,2-propadienyl)benzene (8).¹¹ ¹H NMR (CDCl₃, 300 MHz) δ 2.11 (t, J = 3.0 Hz, 3 H), 5.04 (q, J = 3.0 Hz, 2 H), 7.17–7.44 (m, 5 H); ¹³C NMR (CDCl₃, 75 MHz) δ 16.6 (CH₃), 76.9 (CH₂), 99.8 (C), 125.6 (2 $\times$ CH), 126.5 (CH), 128.3 (2 $\times$ CH), 136.7 (C), 204.1 (C); MS (EI, 70 eV) m/z 130 (M⁺, 57), 12 (M⁺ – H, 100), 115 (M⁺ – CH₃, 71).

(10) Tanigawa, Y.; Murahashi, S. *J. Org. Chem.* **1980**, *45*, 4536–4538.

(11) Petasis, N. A.; Hu, Y.-H. *J. Org. Chem.* **1997**, *62*, 782–783.

(1-Methyl-1,2-butadienyl)benzene (9).¹¹ ¹H NMR (CDCl₃, 300 MHz) δ 1.79 (d, J = 6.86 Hz, 3 H), 2.12 (d, J = 3.02 Hz, 3 H), 5.49–5.41 (m, 1 H), 7.45–7.19 (m, 5 H); ¹³C NMR (CDCl₃, 75 MHz) δ 14.3 (CH₃), 17.1 (CH₃), 87.5 (CH), 99.7 (C), 125.7 (2 $\times$ CH), 126.3 (CH), 128.2 (2 $\times$ CH), 137.8 (C), 205.0 (C); MS (EI, 70 eV) m/z 144 (M⁺, 38), 128 (M⁺ – CH₃, 100), 115 (M⁺ – C₂H₅, 100), 77 (C₆H₅⁺, 14).

(Cyclohexylideneethenyl)benzene (10).¹² ¹H NMR (CDCl₃, 300 MHz) δ 1.51–1.78 (m, 6 H), 2.15–2.33 (m, 4 H), 6.00 (t, J = 1.9 Hz, 1 H), 7.14–7.62 (m, 5 H); ¹³C NMR (CDCl₃, 75 MHz) δ 26.1 (CH₂), 27.7 (2 $\times$ CH₂), 31.3 (2 $\times$ CH₂), 92.3 (CH), 106.5 (C), 126.3 (CH), 126.5 (2 $\times$ CH), 128.5 (2 $\times$ CH), 136.2 (C), 199.7 (C); MS (EI, 70 eV) m/z 184 (M⁺, 8), 141 (M⁺ – C₃H₇, 32), 105 (100).

1-Methoxy-2-(2-cyclohexylideneethenyl)benzene (11). ¹H NMR (CDCl₃, 300 MHz) δ 1.55–1.69 (m, 6 H), 2.18–2.34 (m, 4 H), 3.86 (s, 3 H), 6.41 (t, J = 2.0 Hz, 1 H), 6.85–6.96 (m, 2 H), 7.12–7.21 (m, 1 H), 7.39 (dd, J = 7.8, 1.5 Hz, 1 H); ¹³C NMR (CDCl₃, 75 MHz) δ 26.2 (CH₂), 27.6 (2 $\times$ CH₂), 31.3 (2 $\times$ CH₂), 55.5 (CH₃), 86.2 (CH), 105.5 (C), 110.9 (CH), 120.7 (CH), 124.6 (C), 127.3 (CH), 127.5 (CH), 156.0 (C), 200.1 (C); MS (EI, 70 eV) m/z 214 (M⁺, 23), 199 (M⁺ – CH₃, 70), 131 (100).

(1,3-Butadienylidene)cyclohexane (12).¹³ ¹H NMR (CDCl₃, 300 MHz) δ 1.57–1.72 (m, 6 H), 2.05–2.33 (m, 4 H), 4.92 (d, J = 10.3 Hz, 1 H), 5.17 (dd, J = 15.6, 1.5 Hz, 1 H), 5.69 (d, J = 10.3 Hz, 1 H), 6.08–6.27 (m, 1 H); ¹³C NMR (CDCl₃, 75 MHz) δ 26.0 (CH₂), 27.3 (2 $\times$ CH₂), 31.3 (2 $\times$ CH₂), 92.5 (CH₂), 103.6 (C), 113.8 (CH), 134.8 (CH), 202.0 (C); MS (EI, 70 eV) m/z 134 (M⁺, 18), 105 (M⁺ – C₂H₅, 26), 91 (100).

(12) Minami, T.; Shikita, S.; So, S.; Nakayama, M.; Yamamoto, I. *J. Org. Chem.* **1988**, 53, 2937–2942.

(13) Pashayan, A. A.; Voskanyan, M. G.; Badanyan, S. O. *Arm. Khim. Zh.* **1977**, 30, 992–999.

(4-Cyclohexylidene-3-buten-1-ynyl)benzene (13).¹⁴ ¹H NMR (CDCl₃, 300 MHz) δ 1.55–1.76 (m, 6 H), 2.17–2.21 (m, 4 H), 5.46 (t, J = 2.4 Hz, 1 H), 7.27–7.31 (m, 3 H), 7.42–7.47 (m, 2 H); ¹³C NMR (CDCl₃, 75 MHz) δ 25.9 (CH₂), 26.9 (2 $\times$ CH₂), 30.8 (2 $\times$ CH₂), 73.3 (CH), 84.1 (C), 88.4 (C), 104.4 (C), 123.8 (C), 127.8 (CH), 128.2 (2 $\times$ CH), 131.4 (2 $\times$ CH), 207.1 (C); MS (EI, 70 eV) m/z 208 (M⁺, 96), 202 (38), 180 (M⁺ – C₅H₁₀, 84), 165 (100).

(4-Cyclohexylidene-3-buten-1-ynyl)trimethylsilane (14).¹⁵ ¹H NMR (CDCl₃, 300 MHz) δ 0.19 (s, 9 H), 1.46–1.70 (m, 6 H), 2.14–2.18 (m, 4 H), 5.27 (t, J = 2.2 Hz, 1 H); ¹³C NMR (CDCl₃, 75 MHz) δ 0.0 (3 $\times$ CH₃), 25.8 (CH₂), 26.8 (2 $\times$ CH₂), 30.6 (2 $\times$ CH₂), 73.2 (CH), 93.2 (C), 99.6 (C), 104.3 (C), 207.4 (C); MS (EI, 70 eV) m/z 204 (M⁺, 21), 189 (M⁺ – CH₃, 100).

(R)-1-Phenyl-2-propynyl benzoate (15a). ¹H NMR (CDCl₃, 300 MHz) δ 2.71 (d, J = 2.2 Hz, 1 H), 6.73 (d, J = 2.2 Hz, 1 H), 7.39–7.66 (m, 8 H), 8.09 (dd, J = 5.3, 1.6 Hz, 2 H); ¹³C NMR (CDCl₃, 75 MHz) δ 65.8 (CH), 75.6 (C), 80.3 (CH), 127.7 (2 $\times$ CH), 128.4 (2 $\times$ CH), 128.7 (2 $\times$ CH), 129.1 (CH), 129.6 (C), 129.9 (2 $\times$ CH), 133.3 (CH), 136.5 (C), 165.3 (C); MS (EI, 70 eV) m/z 236 (M⁺, 18), 208 (11), 115 (100), 77 (C₆H₅⁺, 49); $[\alpha]_D^{20}$ = –21.0 (c = 4.41, CHCl₃).

(R)-1-Phenyl-2-propynyl acetate (15b).¹⁶ ¹H NMR (CDCl₃, 300 MHz) δ 2.12 (s, 3 H), 2.66 (d, J = 2.2 Hz, 1 H), 6.46 (d, J = 2.2 Hz, 1 H), 7.37–7.44 (m, 3 H), 7.54 (m, 2 H); ¹³C NMR (CDCl₃, 75 MHz) δ 21.0 (CH₃), 65.3 (CH), 75.3 (CH), 80.2 (C), 127.7 (2 $\times$ CH), 128.7 (2 $\times$ CH), 129.1 (CH), 136.5 (C), 169.7 (C); MS (EI, 70 eV) m/z 174 (M⁺, 7), 159 (M⁺ – CH₃, 2),

(14) Satoh, T.; Hanaki, N.; Kuramochi, Y.; Inoue, Y.; Hosoya, K.; Sakai, K. *Tetrahedron* **2002**, *58*, 2533–2549.

(15) Wang, K. K.; Zhang, Q.; Liao, J. *Tetrahedron Lett.* **1996**, *37*, 4087–4090.

(16) Smulik, J. A.; Diver, S. T. *J. Org. Chem.* **2000**, *65*, 1788–1792.

132 (38), 114 ($M^+ - C_2H_4O_2$, 100); $[\alpha]^{21}_D = +4.8$ ($c = 1.61$, $CHCl_3$); Lit.¹⁷ $[\alpha]^{25}_D = +4.4$ ($c = 4.33$, $CHCl_3$).

(R)-1,1'-(1,2-Propadiene-1,3-diyl)bisbenzene (16).¹⁸ 1H NMR ($CDCl_3$, 300 MHz) δ 6.58 (s, 2 H), 7.21–7.36 (m, 10 H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 98.4 ($2 \times CH$), 127.0 ($4 \times CH$), 127.3 ($2 \times CH$), 128.7 ($4 \times CH$), 133.6 ($2 \times C$), 207.8 (C); MS (EI, 70 eV) m/z 192 (M^+ , 59), 191 ($M^+ - H$, 67), 105 (100); MS (EI, 70 eV) m/z 192 (M^+ , 59), 191 ($M^+ - H$, 67), 105 (100).

(S)-3-Butyn-2-yl benzoate (17). 1H NMR ($CDCl_3$, 300 MHz) δ 1.65 (d, $J = 6.9$ Hz, 3 H), 2.51 (d, $J = 2.2$ Hz, 1 H), 5.69 (m, 1 H), 7.44 (m, 2 H), 7.57 (m, 1 H), 8.07 (m, 2 H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 21.2 (CH_3), 60.5 (CH), 73.0 (CH), 82.1 (C), 128.3 ($2 \times CH$), 129.68 ($2 \times CH$), 129.74 (C), 133.1 (CH), 165.3 (C); MS (EI, 70 eV) m/z 174 (M^+ , 6), 105 (100), 77 ($C_6H_5^+$, 57); $[\alpha]^{21}_D = -31.1$ ($c = 3.92$, $CHCl_3$).

(R)-4-Phenyl-2,3-butadiene (18).¹⁸ 1H NMR ($CDCl_3$, 300 MHz) δ 1.78 (dd, $J = 6.9$, 3.0 Hz, 3 H), 5.53 (qd, $J = 6.9$, 6.6 Hz, 1 H), 6.09 (dq, $J = 6.6$, 3.0 Hz, 1 H), 7.14–7.32 (m, 5 H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 14.1 (CH_3), 89.6 (CH), 93.9 (CH), 126.6 ($3 \times CH$), 128.5 ($2 \times CH$), 135.1 (C), 206.0 (C); MS (EI, 70 eV) m/z 130 (M^+ , 71), 115 ($M^+ - CH_3$, 86), 84 (100).

(17) Raminelli, C.; Comasseto, J. V.; Andrade, L. H.; Porto, A. L. M. *Tetrahedron: Asymmetry* **2004**, *15*, 3117–3122.

(18) Ohno, H.; Miyamura, K.; Tanaka, T. *J. Org. Chem.* **2002**, *67*, 1359–1367.

RR131C1-1H

7.5507
7.4803
7.4757
7.4584
7.4547
7.4355
7.4300
7.4154
7.4108
7.4044
7.3870
7.3825
7.3669
7.3624
7.3541
7.3459
7.3413
7.3367
7.3267
7.3185
7.3075
7.2983
7.2938
7.2691

RR139C1-1H

7.5800
7.5681
7.4017
7.3971
7.3907
7.3742
7.3678
7.3486
7.3303
7.3258
7.3212
7.3102
7.3020
7.2773
7.2691

RR139C1-13C

RR158C1-1H

7.6065
7.4190
7.4136
7.4062
7.3898
7.3852
7.3706
7.3660
7.3578
7.3523
7.3477
7.3386
7.3303
7.3057
7.2691

RR158C1-13C

RR060C1-1H

7.4419
7.3934
7.3770
7.3523
7.3285
7.3020
7.2691
7.2581
7.2508
7.2298
7.2151
7.2087
7.2014
6.5156
6.5065
6.4973
6.4882

2.2609
2.2509

RR060C1-13C

RR1978-1H

RR1978

RR075C1-13C

207.2896

156.1761

136.6967
128.3753
128.0626
127.8807
126.7605
125.7786
122.8763
120.7814

111.0999

103.6951

90.4930

77.4219
77.0000
76.5781

55.6366

16.7651

RR80C1-1H

7.3633
7.3386
7.3130
7.2691
7.2508
7.2270
7.2032

6.3291
6.2952
6.2733
6.2623
6.2395
6.2075
6.1892
6.1800
6.1709
6.1627
6.1453
6.1361
6.1279

5.3205
5.3150
5.2647
5.2601
5.0892
5.0837
5.0526

RR080C1-13C

RR096C1-1H

7.3733
7.3678
7.3477
7.3313
7.3212
7.3139
7.3075
7.2993
7.2919
7.2874
7.2691
7.2435

5.9642
5.9542
5.9450
5.9350

RR096C1-13C

RR115C1-1H

7.379
7.3614
7.3550
7.3404
7.3349
7.2883
7.2837
7.2782
7.2691
7.2599
7.2517
7.2417
7.2371

5.7695
5.7594
5.7503
5.7402

RR115C1-13C

RR162C1-1H

7.4044
7.3870
7.3605
7.3496
7.2846
7.2673
7.2609
7.2417

5.1020
5.0919
5.0818
5.0709

2.1777
2.1677
2.1622
2.1567

0.0 0.5 1.0 1.5 2.0 2.5 3.0 3.5 4.0 4.5 5.0 5.5 6.0 6.5 7.0 7.5 (ppm)

RR078C3-13C

RR161C-13C

RR120C1-1H

7.3090
7.2871
7.2724
7.2285
7.2090
7.1871
7.1651
7.1431
7.1236

6.0184
6.0062
5.9964

2.3319
2.2904
2.2660
2.2318
2.1635

1.6951
1.6756
1.6438
1.6146
1.5609
1.5365

RR054C1

RR076C1-1H

RR076C1-13C

RR103Cl-13C

RR102C 1-H

7.3194
7.3148
7.3066
7.2993
7.2892
7.2828
7.2691
7.2554

5.4814
5.4741
5.4668
5.4604
5.4531

2.2362
2.2298
2.2161
2.2106
2.1969
2.1896
1.7388
1.7251
1.7178
1.7004
1.6830
1.6638
1.6400
1.6254
1.6199
1.6071
1.5952
1.5824
1.5687
1.5559
1.5367
1.5221
1.5139

RR102C1-13C

RR116C1-1H

RR116C1-13C

RR083C 1-1H

7.6220
7.6111
7.6065
7.6019
7.5891
7.5818
7.5754
7.5617
7.5571
7.5525
7.4757
7.4712
7.4666
7.4538
7.4501
7.4364
7.4309
7.4254
7.4200
7.4081
7.3953
7.3907
7.2691
6.7305
6.7223

2.7145
2.7071

(ppm)

RR083Cl-13C

15a

RR113C1-1H

7.5306
7.4254
7.4209
7.4090
7.4035
7.3998
7.3907
7.3852
7.3770
7.3697
7.2682

6.4699
6.4626

2.6669
2.6596

2.1238

(ppm)

RR051C2-1H

7.3596
7.3532
7.3459
7.3294
7.3239
7.3093
7.3038
7.2691
7.2618
7.2554
7.2471
7.2389
7.2298
7.2206
7.2160
6.6135

16

RR051C2-13C

RR144C1-1H

7.5919
7.5873
7.5736
7.5672
7.5599
7.5471
7.5425
7.5379
7.4675
7.4629
7.4419
7.4227
7.4172
7.2682

5.7320
5.7247
5.7091
5.7027
5.6872
5.6799
5.6643
5.6579

17

2.5124
2.5051

1.6346
1.6374

(ppm)

RR144C1-13C

RR155C1-1H

7.2801
7.2691
7.2316
7.2170
7.2032
7.1886
7.1786
7.1731
7.1594

6.1307
6.1206
6.1096
6.0987
6.0886
6.0776

5.5930
5.5701
5.5473
5.5244
5.5006

1.8165
1.8055
1.7927
1.7818

RR149C1

RR153C1

RR155C3

RR156C2