An Efficient Synthesis of 4-Halo-5-hydroxyfuran-2(5H)-ones via the Sequential Halolactonization and γ-Hydroxylation of 4-Aryl-2,3-alkadienoic Acids

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Supporting Information

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Experimental Section

Starting Materials. 2,3-Allenoic acids were prepared according to the known method by treatment of the acid chlorides with ethyl 2-(triphenylphoranylidene)propionte and subsequent hydrolysis of the 2,3-allenoic esters with 1.5 equiv of NaOH.\(^1\)

General procedure for 3-methyl-4-iodo-5-phenyl-5-hydroxyl-2(5\(\text{H}\))-furanone 2a from the optically active salt of 2-methyl-4-phenylbuta-2,3-dienoic acid and (L)-(\(-\))-cinchonidine (+)-1aa: A mixture of 2-methyl-4-phenyl-2,3-butadienoic acid 1a (5.22g, 30 mmol) and (L)-(\(-\))-cinchonidine (4.416g, 15 mmol) in 25 ml ethyl acetate was stirred at room temperature overnight to afford a salt, which could be readily recrystallized in ethyl acetate / methanol (15:1) for 4 times to afford 2.50 g (35.5\%) of the optically active (+)-1aa: \([\alpha]_D^{20} = 94.6^\circ (c = 1, \text{EtOH}).\)

A mixture of the optically active salt (+)-1aa (468 mg, 1.0 mmol) and I\(_2\) (280 mg, 1.1 mmol) in 15 ml THF and 5 ml DMF was stirred at room temperature overnight. Then the mixture was diluted with diethyl ether, washed subsequently with 5\% Na\(_2\)S\(_2\)O\(_3\), brine, and dried over Na\(_2\)SO\(_4\). After evaporation, the residue was purified via flash chromatography on silica gel with petroleum ether / diethyl ether (3.5:1) as the eluent to afford 265 mg (84\%) of 2a. The structure of 2a was unambiguously determined by the X-ray single crystal diffraction study.

The following compounds were prepared according to Procedure A.

Procedure A.

(1) Synthesis of 3-methyl-4-iodo-5-phenyl-5-hydroxyl-2(5\(\text{H}\))-furanone (2a). A
mixture of 2-methyl-4-phenyl-2,3-butadienoic acid 1a (86 mg, 0.494 mmol), LiOAc·2H₂O (63 mg, 0.62 mmol), and I₂ (250 mg, 0.98 mmol) in THF (2 mL) was stirred at room temperature for 3 h. Then DMF (1 mL) was added, and the mixture was stirred at 40°C for 36 h with O₂ (1 atm). Then the mixture was diluted with diethyl ether, washed subsequently with 5% Na₂S₂O₃, brine, and dried over Na₂SO₄. After evaporation, the residue was purified via flash chromatography on silica gel with petroleum ether / diethyl ether (3.5:1) as the eluent to afford 136 mg (87%) of 2a: solid, mp 150-152 °C (acetone/n-hexane); ¹H NMR (300 MHz, CD₃COCD₃) δ 7.60-7.38 (m, 5 H), 7.25-7.15 (bs, 1 H), 1.94 (s, 3 H); ¹³C NMR (75.4 MHz, CD₃COCD₃) δ 169.9, 137.9, 137.1, 130.2, 129.3, 127.3, 127.2, 107.2, 13.2; MS (m/z) 316 (M⁺, 4.65), 67 (100); IR (KBr) 3282, 1742, 1732, 1643 cm⁻¹; Anal. Calcd for C₁₁H₉IO₃: C, 41.80; H, 2.87. Found: C, 41.83; H, 2.62.

(2) 3-(n-Propyl)-4-ido-5-phenyl-5-hydroxyl-2(5H)-furanone (2b): The reaction of 1b (101 mg, 0.50 mmol), I₂ (257 mg, 1.01 mmol), and LiOAc-2H₂O (65 mg, 0.64 mmol) afforded 143 mg (83%) of 2b: liquid; ¹H NMR (300 MHz, CD₃COCD₃) δ 7.62-7.35 (m, 5 H), 7.22 (s, 1 H), 2.34 (t, J = 7.7 Hz, 2 H), 2.34 (t, J = 7.7 Hz, 2 H), 1.73-1.52 (m, 2 H), 0.96 (t, J = 7.4 Hz, 3 H); ¹³C NMR (75.4 MHz, CD₃COCD₃) δ 169.0, 139.6, 137.3, 129.5, 128.6, 127.3, 126.5, 106.4, 29.3, 20.7, 13.5; MS (m/z) 344 (M⁺, 4.14), 95 (100); IR (neat) 3364, 2961, 1744, 1637, 955, 867 cm⁻¹; HRMS calcd for C₁₃H₁₃IO₃ 343.9909.
(3) 3-Benzyl-4-iodo-5-phenyl-5-hydroxyl-2(5H)-furanone (2c): The reaction of 1c (100 mg, 0.40 mmol), I₂ (124 mg, 0.49 mmol), and LiOAc·2H₂O (55 mg, 0.54 mmol) afforded 113 mg (72%) of 2c: solid, mp 151-155 °C (acetone/n-hexane); ¹H NMR (300 MHz, CD₃COCD₃) δ 7.60-7.18 (m, 10 H), 3.72 (s, 2 H), 2.86 (s, 1 H); ¹³C NMR (75.4 MHz, CD₃COCD₃) δ 170.0, 139.9, 138.4, 138.1, 130.7, 130.1, 129.7, 129.4, 128.2, 127.6, 107.8, 34.3; MS (m/z) 392 (M⁺, 1.16), 105 (100); IR (KBr) 3234, 1724, 1642 cm⁻¹; Anal. Calcd for C₁₇H₁₃IO₃: C, 52.06; H, 3.34. Found: C, 52.39; H, 3.48.

(4) 3-(n-Butyl)-4-iodo-5-(4'-methoxylphenyl)-5-hydroxyl-2(5H)-furanone (2d): The reaction of 1d (187 mg, 0.76 mmol), I₂ (388 mg, 1.53 mmol), and LiOAc·2H₂O (93 mg, 0.91 mmol) afforded 160 mg (54%) of 2d: liquid; ¹H NMR (300 MHz, CD₃COCD₃) δ 7.39 (d, J = 6.9 Hz, 2 H), 6.87 (d, J = 6.9 Hz, 2 H), 5.20-4.30 (bs, 1 H), 3.79 (s, 3 H), 2.31 (t, J = 7.7 Hz, 2 H), 1.60-1.21 (m, 4 H), 0.91 (t, J = 7.4 Hz, 3 H); ¹³C NMR (75.4 MHz, CD₃COCD₃) δ 169.8, 161.8, 140.5, 130.0, 129.0, 128.1, 115.2, 107.7, 56.0, 30.4, 28.3, 23.4, 14.5; MS (m/z) 388 (M⁺, 17.57), 135 (100); IR
(5) **3-(n-Butyl)-4-iodo-5-(4′-fluorophenyl)-5-hydroxyl-2(5H)-furanone (2e):** The reaction of 1e (117 mg, 0.50 mmol), I₂ (159 mg, 0.63 mmol), and LiOAc·2H₂O (64 mg, 0.63 mmol) afforded 138 mg (73%) of 2e: liquid; ¹H NMR (300 MHz, CD₃COCD₃) δ 7.40 (dd, J = 5.0 and 8.9 Hz, 2 H), 7.00 (t, J = 8.6 Hz, 2 H), 5.70-4.50 (bs, 1 H), 2.26 (t, J = 7.7 Hz, 2 H), 1.55-1.39 (m, 2 H), 1.39-1.18 (m, 2 H), 0.86 (t, J = 7.4 Hz, 3 H); ¹³C NMR (75.4 MHz, CD₃COCD₃) δ 169.7, 164.4 (d, J = 246.3 Hz), 140.9, 134.5 (d, J = 3.4 Hz), 129.8 (d, J = 8.7 Hz), 127.6, 116.3 (d, J = 21.9 Hz), 107.0, 30.3, 28.1, 23.3, 14.5; ¹⁹F NMR (282 MHz, CD₃COCD₃) δ -114.36; MS (m/z) 376 (M⁺, 2.25), 123 (100); IR (neat) 3389, 1732, 1624, 1605, 1235 cm⁻¹; HRMS calcd for C₁₄H₁₄FIO₃ 375.9972. Found 376.0008.

(6) **3-(n-Butyl)-4-iodo-5-(4′-i-propylphenyl)-5-hydroxyl-2(5H)-furanone (2f):** The reaction of 1f (129 mg, 0.50 mmol), I₂ (155 mg, 0.61 mmol), and LiOAc·2H₂O (64 mg, 0.63 mmol) afforded 139 mg (70%) of 2f: solid, mp 113-114 °C (acetone/n-hexane); ¹H NMR (300 MHz, CD₃COCD₃) δ 7.44 (d, J = 8.7 Hz, 2 H), 7.31 (d, J =
9 Hz, 2 H), 7.25-7.10 (bs, 1 H), 3.05-2.85 (m, 1 H), 2.37 (t, J = 7.4 Hz, 2 H), 1.68-1.50 (m, 2 H), 1.50-1.33 (m, 2 H), 1.24 (d, J = 6.9 Hz, 6 H), 0.94 (t, J = 7.2 Hz, 3 H); ¹³C NMR (75.4 MHz, CD₃COCD₃) δ 170.0, 151.4, 140.8, 135.8, 128.0, 127.8, 127.7, 107.6, 35.0, 28.3, 24.7, 23.5, 14.6; MS (m/z) 400 (M⁺, 24.14), 147 (100); IR (KBr) 3380, 1733, 1632, 1220, cm⁻¹; Anal. Calcd for C₁₇H₂₁IO₃: C, 51.01; H, 5.29. Found: C, 50.89; H, 5.28.

![Image](https://example.com/image1.png)

(7) 3-(n-Butyl)-4-iodo-5-(4′-methylphenyl)-5-hydroxyl-2(5H)-furanone (2g): The reaction of 1g (115 mg, 0.50 mmol), I₂ (156 mg, 0.61 mmol), and LiOAc·2H₂O (61 mg, 0.60 mmol) afforded 135 mg (73%) of 2g: solid, mp 111-112 °C (acetone/n-hexane); ¹H NMR (300 MHz, CD₃COCD₃) δ 7.39 (d, J = 8.4 Hz, 2 H), 7.24 (d, J =7.8 Hz, 2 H), 7.20-7.10 (bs, 1 H), 2.45-2.25 (m, 5 H), 1.62-1.48 (m, 2 H), 1.48-1.26 (m, 2 H), 0.94 (t, J = 7.4 Hz, 3 H); ¹³C NMR (75.4 MHz, CD₃COCD₃) δ 169.9, 140.6, 140.3, 135.3, 130.1, 128.1, 127.4, 107.4, 30.3, 28.0, 23.3, 21.6, 14.5; MS (m/z) 372 (M⁺, 5.89), 119 (100); IR (KBr) 3374, 1720, 1627, 1209 cm⁻¹; Anal. Calcd for C₁₅H₁₇IO₃: C, 48.41; H, 4.60. Found: C, 48.35; H, 4.53.

![Image](https://example.com/image2.png)

(8) 3-Methyl-4-iodo-5-(1′-naphthyl)-5-hydroxyl-2(5H)-furanone (2h): The reaction
of 1h (88 mg, 0.39 mmol), I₂ (177 mg, 0.70 mmol), and LiOAc·2H₂O (70 mg, 0.69 mmol) afforded 112 mg (78%) of 2h: solid, mp 158-160 °C (acetone/n-hexane); \(^1\)H NMR (300 MHz, CD₃COCD₃) δ 8.60-8.30 (bs, 1 H), 8.10-7.85 (m, 3 H), 7.70-7.50 (m, 3 H), 745 (bs, 1 H), 2.09 (s, 3 H); \(^{13}\)C NMR (75.4 MHz, CD₃COCD₃) δ 169.7, 138.9, 138.2, 135.7, 133.2, 132.2, 131.9, 130.0, 127.6, 127.0, 126.9, 125.7, 108.7, 13.8; MS (m/z) 366 (M⁺, 31.19), 67 (100); IR (KBr) 3289, 1747, 1732, 1643, 1287 cm⁻¹; Anal. Calcd for C₁₅H₁₁IO₃: C, 49.20; H, 3.03. Found: C, 49.22; H, 2.72.

(9) 3-(n-Propyl)-4-iodo-5-(1′-naphthyl)-5-hydroxyl-2(5H)-furanone (2i): The reaction of 1i (100 mg, 0.397 mmol), I₂ (177 mg, 0.70 mmol), and LiOAc·2H₂O (75 mg, 0.74 mmol) afforded 111 mg (71%) of 2i: solid, mp 162-164 °C (acetone/n-hexane); \(^1\)H NMR (300 MHz, CD₃COCD₃) δ 8.60-8.40 (bs, 1 H), 8.10-7.90 (m, 3 H), 7.67-7.10 (m, 4 H), 2.49 (t, \(J = 7.4\) Hz, 2 H), 1.80-1.60 (m, 2 H), 1.03 (t, \(J = 7.2\) Hz, 3 H); \(^{13}\)C NMR (75.4 MHz, CD₃COCD₃) δ 168.5, 141.3, 137.3, 134.8, 132.2, 131.4, 130.9, 129.2, 127.3, 126.6, 126.1, 125.9, 124.8, 20.7, 13.7; MS (m/z) 394 (M⁺, 36.78), 155 (100); IR (KBr) 3345, 1701, 1634, 1236 cm⁻¹; Anal. Calcd for C₁₇H₁₅IO₃: C, 51.80; H, 3.84. Found: C, 51.86; H, 3.66.

The following compounds were prepared according to Procedure B.

Procedure B.
(1) Synthesis of 3-methyl-4-chloro-5-phenyl-5-hydroxyl-2(5H)-furanone (4a). A solution of 1a (139 mg, 0.8 mmol) and CuCl₂ (432 mg, 3.2 mmol) in acetone / H₂O (2:1, 4.5 mL) was stirred at 65°C for 4 h (in the case of CuCl₂) or 2 h (in the case of CuBr₂). The mixture was then diluted with diethyl ether, washed subsequently with diluted HCl, saturated NaHCO₃ and brine, and dried over Na₂SO₄. After evaporation, the residue was treated with LiOAc·2H₂O (44 mg, 0.43 mmol) in THF / DMF (2:1, 3 mL) at 40°C with O₂ (1 atm) for 8.5 h. Then the mixture was diluted with diethyl ether, washed with brine, and dried over Na₂SO₄. After filtration and evaporation, the residue was purified via flash chromatography on silica gel with hexane / ethyl acetate (10:1) as the eluent to afford 138 mg (77%) of 4a: solid, mp 104-105 °C (diethyl ether/n-hexane); ¹H NMR (300 MHz, CD₃COCD₃) δ 7.62-7.51 (m, 2 H), 7.51-7.38 (m, 3 H), 7.32 (s, 1 H), 1.91 (s, 3 H); ¹³C NMR (75.4 MHz, CD₃COCD₃) δ 169.9, 154.0, 137.3, 130.7, 129.6, 127.2, 126.9, 105.6, 9.2; MS (m/z) 226 (M⁺(³⁷Cl), 6.41), 224 (M⁺(³⁵Cl), 19.32), 105 (100); IR (KBr) 3283, 1741, 1722, 1666 cm⁻¹; Anal. Calcd for C₁₁H₉ClO₃ C, 58.81; H, 4.04. Found C, 59.07; H, 4.08.

(2) 3-Methyl-4-bromo-5-phenyl-5-hydroxyl-2(5H)-furanone (4b): The reaction of 1a (87 mg, 0.50 mmol), CuBr₂ (448 mg, 2.0 mmol), and LiOAc·2H₂O (53 mg, 0.52 mmol) afforded 73 mg (54%) of 4b: solid, mp 116-117 °C (acetone/n-hexane); ¹H NMR (300 MHz, CD₃COCD₃) δ 7.60-7.50 (m, 2 H), 7.50-7.40 (m, 3 H), 7.40-7.28
(bs, 1 H), 1.92 (s, 3 H); $^{13}$C NMR (75.4 MHz, CD$_3$COCD$_3$) δ 170.0, 146.6, 137.6, 130.8, 130.7, 129.6, 127.4, 106.4, 10.8; MS (m/z) 270 (M$^+$(81Br), 11.95), 268 (M$^+$(79Br), 12.39), 105 (100); IR (KBr) 3284, 1747, 1735, 1660, cm$^{-1}$; Anal. Calcd for C$_{11}$H$_9$BrO$_3$ C, 49.10; H, 3.37. Found C, 49.20; H, 3.27.

(3) 3-(n-Propyl)-4-chloro-5-phenyl-5-hydroxyl-2(5H)-furanone (4c): The reaction of 1b (162 mg, 0.80 mmol), CuCl$_2$ (432 mg, 3.2 mmol), and LiOAc·2H$_2$O (43 mg, 0.42 mmol) afforded 95 mg (47%) of 4c: liquid; $^1$H NMR (300 MHz, CD$_3$COCD$_3$) δ 7.60-7.52 (m, 2 H), 7.50-7.42 (m, 3 H), 3.50-3.00 (bs, 1 H), 2.35 (t, $J$ = 7.4 Hz, 2 H), 1.70-1.54 (m, 2 H), 0.94 (t, $J$ = 7.4 Hz, 3 H); $^{13}$C NMR (75.4 MHz, CD$_3$COCD$_3$) δ 169.7, 154.6, 137.4, 130.7, 130.2, 129.7, 127.2, 105.5, 26.4, 21.3, 14.3; MS (m/z) 254 (M$^+$(37Cl), 4.81), 252 (M$^+$(35Cl), 14.53), 105 (100); IR (neat) 3353, 1748, 1661, 1222 cm$^{-1}$; HRMS calcd for C$_{13}$H$_{13}$ClO$_3$ 252.0553. Found 252.0576.

(4) 3-(n-Propyl)-4-bromo-5-phenyl-5-hydroxyl-2(5H)-furanone (4d): The reaction of 1b (122 mg, 0.60 mmol), CuBr$_2$ (532 mg, 2.38 mmol), and LiOAc·2H$_2$O (32 mg, 0.31 mmol) afforded 89 mg (50%) of 4d: liquid; $^1$H NMR (300 MHz, CD$_3$COCD$_3$) δ
7.58-7.50 (m, 2 H), 7.50-7.42 (m, 3 H), 7.38-7.34 (bs, 1 H), 2.34 (t, \(J = 7.5\) Hz, 2 H), 1.70-1.54 (m, 2 H), 0.95 (t, \(J = 7.7\) Hz, 3 H); \(^{13}\)C NMR (75.4 MHz, CD\(_3\)COCD\(_3\)) \(\delta\)

169.8, 147.2, 137.7, 134.1, 130.6, 129.6, 127.3, 106.5, 27.8, 21.4, 14.3; MS (m/z) 298 (M\(^+\)(\(^{81}\)Br), 7.24), 296 (M\(^+\)(\(^{79}\)Br), 7.51), 95 (100); IR (neat) 3358, 1747, 1654, 1222 cm\(^{-1}\); HRMS calcd for C\(_{13}\)H\(_{13}\)\(^{79}\)BrO\(_3\) 296.0048. Found 296.0077.

\[\text{(5) 3-Benzyl-4-chloro-5-phenyl-5-hydroxyl-2(5H)-furanone (4e):} \]

The reaction of 1c (151 mg, 0.60 mmol), CuCl\(_2\) (327 mg, 2.42 mmol), and LiOAc·2H\(_2\)O (60 mg, 0.59 mmol) afforded 143 mg (79\%) of 4e: solid, mp 136-138 °C (acetone/n-hexane); \(^1\)H NMR (300 MHz, CD\(_3\)COCD\(_3\)) \(\delta\)

7.58-7.51 (m, 3 H), 7.48-7.42 (m, 3 H), 7.36-7.30 (m, 4 H), 7.30-7.22 (m, 1 H), 3.72 (s, 2 H); \(^{13}\)C NMR (75.4 MHz, CD\(_3\)COCD\(_3\)) \(\delta\)

169.5, 155.1, 137.8, 137.3, 130.8, 129.9, 129.7, 129.5, 128.0, 127.2, 105.7; MS (m/z) 302 (M\(^+\)(\(^{37}\)Cl), 1.38), 300 (M\(^+\)(\(^{35}\)Cl), 4.01), 105 (100); IR (KBr) 3375, 1752, 1741, 1665, 1153 cm\(^{-1}\); Anal. Calcd for C\(_{17}\)H\(_{13}\)ClO\(_3\) C, 67.89; H, 4.36. Found C, 67.82; H, 4.19.

\[\text{(6) 3-Benzyl-4-bromo-5-phenyl-5-hydroxyl-2(5H)-furanone (4f):} \]

The reaction of 1c (150 mg, 0.60 mmol), CuBr\(_2\) (538 mg, 2.41 mmol), and LiOAc·2H\(_2\)O (61 mg, 0.60
mmol) afforded 101 mg (49%) of 4f: solid, mp 138-140 °C (acetone/n-hexane); $^1$H NMR (300 MHz, CD$_3$COCD$_3$) $\delta$ 7.58-7.48 (m, 3 H), 7.48-7.40 (m, 3 H), 7.37-7.30 (m, 4 H), 7.30-7.21 (m, 1 H), 3.72 (s, 2 H); $^{13}$C NMR (75.4 MHz, CD$_3$COCD$_3$) $\delta$ 169.8, 148.1, 137.8, 137.7, 133.5, 130.8, 130.0, 129.9, 129.8, 128.1, 127.4, 106.6, 31.9; MS ($m/z$) 346 (M$^+$($^{81}$Br), 0.51), 344 (M$^+$($^{79}$Br), 0.53), 105 (100); IR (KBr) 3382, 1745, 1735, 1657 cm$^{-1}$; Anal. Calcd for C$_{17}$H$_{13}$BrO$_3$ C, 59.15; H, 3.80. Found C, 59.36; H, 3.77.

(7) 3-(n-Propyl)-4-chloro-5-(1′-naphthyl)-5-hydroxy-2(5H)-furanone (4g): The reaction of 1i (126 mg, 0.50 mmol), CuCl$_2$ (270 mg, 2.0 mmol), and LiOAc·2H$_2$O (51 mg, 0.50 mmol) afforded 123 mg (81%) of 4g: solid, mp 120-122 °C (acetone/n-hexane); $^1$H NMR (300 MHz, CD$_3$COCD$_3$) $\delta$ 8.61 (bs, 1 H), 8.10-7.45 (m, 7 H), 2.44 (t, $J = 7.2$ Hz, 2 H), 1.78-1.60 (m, 2 H), 0.99 (t, $J = 7.4$ Hz, 3 H); $^{13}$C NMR (75.4 MHz, CD$_3$COCD$_3$) $\delta$ 169.3, 154.3, 153.8, 132.9, 132.3, 131.9, 130.0, 127.5, 127.4, 127.1, 125.7, 107.3, 26.9, 21.4, 14.4; MS ($m/z$) 304 (M$^+$($^{37}$Cl), 14.52), 302 (M$^+$($^{35}$Cl), 43.05), 155 (100); IR (KBr) 3329, 1719, 1654 cm$^{-1}$; Anal. Calcd for C$_{17}$H$_{15}$ClO$_3$ C, 67.44; H, 4.99. Found C, 67.21; H, 4.98.
(8) 3-(n-Propyl)-4-bromo-5-(1′-naphthyl)-5-hydroxyl-2(5H)-furanone (4h): The reaction of 1i (126 mg, 0.50 mmol), CuBr₂ (450 mg, 2.0 mmol), and LiOAc·2H₂O (51 mg, 0.50 mmol) afforded 133 mg (77%) of 4h: solid, mp 148-150 °C (acetone/n-hexane); ¹H NMR (300 MHz, CD₃COCD₃) δ 8.55 (bs, 1 H), 8.10-7.40 (m, 7 H), 2.45 (bs, 2 H), 1.80-1.60 (m, 2 H), 1.01 (t, J = 7.5 Hz, 3 H); ¹³C NMR (75.4 MHz, CD₃COCD₃) δ 169.6, 147.2, 135.8, 133.0, 132.4, 132.0, 130.1, 127.6, 127.23, 127.16, 125.8, 107.7, 28.3, 21.5, 14.6; MS (m/z) 348 (M⁺(⁸¹Br), 20.13), 346 (M⁺(⁷⁹Br), 20.65), 155 (100); IR (KBr) 3313, 1711, 1652, cm⁻¹; Anal. Calcd for C₁₇H₁₅BrO₃ C, 58.81; H, 4.35. Found C, 58.91; H, 4.24.

References:


-d-acetone-
wb-3-56-2-3-c

Pulse Sequence: 1H-3L

Solvent: Acetone-

Ambient temperature

File: wb-3-56-2-3-c

Relax. delay 1.000 sec

Pulse 90, 2.0 degrees

Add. time 1.000 sec

VTEX 18.677 Hertz

208 repetitions

BASELINE 15, 15, 444480 Hz

DECoupling 1H, 300.001070 Hz

Power 40 dB

continuously on

WALTZ-16 Amplitude

DATA PROCESSING

Line broadening 1.0 Hz

Run time 10 min

Total time 0 min, 0 sec
Pulse Sequence: 62u1
Solvent: Acetone
Ambient Temperature
Date: 7/21/94
Mercury-300B 7.0030S

Relax. delay 1.000 sec
Pulse 90.0 degrees
Acq. time 1.665 sec
Width 10000.0 Hz
10240 repetitions
OBSERVE 13C, 70.4429870 MHz
DECOUPLE 31P, 399.0318270 MHz
Power 34 dB
T1 pulse on
WBL2-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FI size 85538
Total time 47 hr, 32 min, 47 sec
Pulse Sequence: 2pul
Solvent: Acetone
Ambient Temperature
Field Strength 400 MHz
Mercury-400B, "DNC300"

Relax, delay 1.000 sec
Pulse of 3 degrees
Acq. time 1.180 sec
Width 2697.8 Hz
11676 repetitions

RESERVE CIF, 75.4423844 MHz
DECUPLE H1, 300.0301870 MHz
Power 36 dB
Continuously on

Line broadening 1.0 Hz
F3 size 1024
Total time 47 hr, 12 min, 47 sec

2f

38 37 36 35 34 33 32 31 30 29 28 ppm
Pulse Sequence: zpul
Solvent: Acetone
Ambient temperature
File: wb-3-5-c
Mercury-3000 "QNC300"

Relax. delay: 30 sec
Pulse 45.8 degrees
Acq. lines: 3,650
Run time: 39.7 hours
1168 repetitions

SOME 0.1, 75.5534011 MHz
DECOUPLING: X1, 396.0310278 MHz
Power 26 dB continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 131072
Total time 107 hr, 53 min, 19 sec
Pulse Sequence: 90, 180
Solvent: CDCl3
Temperature: 27 °C
Magnetic Field: 600 MHz
Sample: 2

1H NMR (600 MHz, CDCl3) δ 7.34 (d, J = 8.0 Hz, 1H), 7.10 (d, J = 8.0 Hz, 1H), 7.03 (s, 1H), 6.94 (d, J = 7.0 Hz, 1H), 6.86 (d, J = 7.0 Hz, 1H), 6.27 (s, 1H), 3.86 (s, 3H), 3.48 (s, 3H), 3.18 (s, 3H), 2.47 (s, 3H), 2.40 (s, 1H), 2.03 (s, 3H), 1.90 (s, 3H), 1.72 (s, 3H), 1.48 (s, 9H), 1.44 (s, 9H), 1.25 (s, 9H), 1.20 (s, 3H), 1.15 (s, 3H), 1.08 (s, 3H), 1.03 (s, 3H), 0.98 (s, 3H), 0.95 (s, 3H), 0.90 (s, 3H), 0.86 (s, 3H), 0.80 (s, 3H), 0.70 (s, 3H), 0.60 (s, 3H), 0.50 (s, 3H), 0.40 (s, 3H), 0.30 (s, 3H), 0.20 (s, 3H), 0.10 (s, 3H), 0.00 (s, 3H).
S35
Pulse Sequence: s2pol
Solvent: Acetone
Ambient temperature
File: wb-5-51-c
Mercury-300B "CH2Cl2"

Relax. delay 2.00 sec
Pulse 45.0 degrees
Acq. time 2.00 sec
Width 1800/3 Hz
Sweep width 10000 Hz

Observation 10.0 Hz
DQcouple 4.93 Hz
Power 63 dB
Continuously on
Wait 16 modulation
DATA PROCESSING
Line Broadening 1.0 Hz
Files *.cbf
Total time 14 hr, 26 min, 48 sec

4c

Cl
Ph
HO
HO

220 200 180 160 140 120 100 80 60 40 20 0 ppm
S41