CO₂ Incorporation Reaction using Arynes: Straightforward Access to Benzoxazinone

Hiroto Yoshida,* Hiroyuki Fukushima, Joji Ohshita, and Atsutaka Kunai*

*Department of Applied Chemistry, Graduate School of Engineering, Hiroshima University,
Higashi-Hiroshima 739-8527, Japan.*

Supporting Information

General Remarks. All manipulations of oxygen- and moisture-sensitive materials were conducted with a standard Schlenk technique under a purified argon atmosphere. Nuclear magnetic resonance spectra were taken on a JEOL EX-270 (¹H, 270 MHz; ¹³C, 67.8 MHz) spectrometer or a JEOL Lambda-400 (¹H, 400 MHz; ¹³C, 99.5 MHz) spectrometer using residual benzene (¹H) or C₆D₆ (¹³C) as an internal standard. High-resolution mass spectra were obtained with a JEOL JMS-SX102A spectrometer. Melting points were measured with Yanaco Micro Melting Point apparatus and uncorrected. The preparative recycling gel permeation chromatography was performed with GL Science PU 614 equipped with Shodex GPC H-2001L and -2002L columns (benzene as an eluent). Unless otherwise noted, commercially available reagents were used without purification. 18-Crown-6 was recrystallized from distilled MeCN. KF (spray-dried) was vacuum dried at 100 °C for 12 h. THF was distilled from sodium/benzophenone ketyl. MeCN was distilled from phosphorus pentoxide.

Aryne Precursors. 2-(Trimethylsilyl)phenyl triflate (**1a**),¹ 4-methyl-2-(trimethylsilyl)phenyl triflate (**1b**),² 4-fluoro-2-(trimethylsilyl)phenyl triflate (**1c**),³ 6-methyl-2-(trimethylsilyl)phenyl triflate (**1d**),⁴ 3-methoxy-2-(trimethylsilyl)phenyl triflate (**1e**),⁵ 4,5-dimethyl-2-(trimethylsilyl)phenyl triflate (**1f**),⁶ 6-(trimethylsilyl)-5-indanyl triflate (**1g**),⁶ 3-(trimethylsilyl)-5,6,7,8-tetrahydro-2-naphthyl triflate (**1h**),³ 3-(trimethylsilyl)-2-naphthyl triflate (**1i**)³ and 3,6-dimethyl-2-(trimethylsilyl)phenyl triflate (**1j**)³ were prepared according to literature procedures.

Imines. Imines were synthesized by condensation of corresponding aldehydes and amines.

Three-Component Coupling of Arynes, Imines and CO₂. A General Procedure. A THF solution (1 mL) of an imine (0.15 mmol), 18-crown-6 (0.158 g, 0.60 mmol) and KF (0.035 g, 0.60 mmol) was degassed through two freeze-thaw cycles, and the reaction flask was filled with CO₂ by connecting to a balloon (1 L). To this solution was added an aryne precursor (0.30 mmol), and the resulting mixture was stirred at 0 °C for the period as specified in Table 1 or Scheme 1. The mixture was diluted with ethyl acetate, filtered through a Celite

plug, washed three times with brine and dried over MgSO_4 . Evaporation of the solvent followed by gel permeation chromatography (benzene as an eluent) gave the corresponding product.

1-Methyl-2-(2,4,6-trimethylphenyl)-1,2-dihydro-(4*H*)-benzo[*d*]-3,1-oxazin-4-one (3aa).

Isolated in 82% yield as a pale green solid: m.p. 108–110 °C; ^1H NMR (C_6D_6) δ 2.07 (s, 3 H), 2.13 (brs, 9 H), 6.18 (s, 1 H), 6.35 (d, $J = 8.4$ Hz, 1 H), 6.65 (s, 2 H), 6.70 (d, $J = 7.3$ Hz, 1 H), 7.09–7.12 (m, 1 H), 8.33 (dd, $J = 8.1, 1.6$ Hz, 1 H); ^{13}C NMR (C_6D_6) δ 20.5, 20.9, 31.3, 90.0, 113.0, 114.4, 119.6, 128.5, 130.5, 131.6, 135.0, 138.3, 139.3, 151.0, 163.9; HRMS Calcd for $\text{C}_{18}\text{H}_{19}\text{NO}_2$: M^+ , 281.1416. Found: m/z 281.1416.

1-Methyl-2-(2,4-dimethoxyphenyl)-1,2-dihydro-(4*H*)-benzo[*d*]-3,1-oxazin-4-one (3ab).

Isolated in 80% yield as a pale green solid: m.p. 134–137 °C; ^1H NMR (C_6D_6) δ 2.31 (s, 3 H), 3.15 (s, 3 H), 3.23 (s, 3 H), 6.27 (dd, $J = 8.6, 2.3$ Hz, 1 H), 6.32–6.38 (m, 3 H), 6.64 (t, $J = 7.5$ Hz, 1 H), 7.09 (td, $J = 7.7, 1.7$ Hz, 1 H), 7.48 (d, $J = 8.4$ Hz, 1 H), 8.26 (dd, $J = 7.8, 1.4$ Hz, 1 H); ^{13}C NMR (C_6D_6) δ 32.7, 54.94, 57.96, 86.2, 98.9, 104.9, 113.3, 114.9, 116.6, 119.4, 128.5, 129.6, 131.4, 135.0, 150.6, 159.0, 162.3, 163.9; HRMS Calcd for $\text{C}_{17}\text{H}_{17}\text{NO}_4$: M^+ , 299.1158. Found: m/z 299.1167.

1-Methyl-2-(2,4-dimethylphenyl)-1,2-dihydro-(4*H*)-benzo[*d*]-3,1-oxazin-4-one (3ac).

Isolated in 76% yield as a pale green solid: m.p. 115–117 °C; ^1H NMR (C_6D_6) δ 2.05 (s, 3 H), 2.10 (s, 3 H), 2.12 (s, 3 H), 5.77 (s, 1 H), 6.35 (d, $J = 8.4$ Hz, 1 H), 6.66 (t, $J = 7.3$ Hz, 1 H), 6.75–6.79 (m, 2 H), 7.08 (td, $J = 8.5, 1.6$ Hz, 1 H), 7.18 (s, 1 H), 8.27 (dd, $J = 8.1, 1.6$ Hz, 1 H); ^{13}C NMR (C_6D_6) δ 19.2, 21.0, 33.1, 90.3, 114.1, 120.0, 126.9, 128.5, 128.5, 130.7, 131.4, 132.2, 135.0, 137.3, 139.3, 150.5, 163.5; HRMS Calcd for $\text{C}_{17}\text{H}_{17}\text{NO}_2$: M^+ , 267.1259. Found: m/z 267.1253.

1-Methyl-2-(4-methoxyphenyl)-1,2-dihydro-(4H)-benzo[d]-3,1-oxazin-4-one (3ad).

Isolated in 73% yield as a pale yellow solid: m.p. 99–102 °C; ^1H NMR (C_6D_6) δ 2.13 (s, 3 H), 3.20 (s, 3 H), 5.46 (s, 1 H), 6.32 (d, $J = 8.4$ Hz, 1 H), 6.58–6.65 (m, 3 H), 7.05 (td, $J = 7.8, 1.6$ Hz, 1 H), 7.11–7.16 (m, 2 H), 8.21 (dd, $J = 7.8, 1.6$ Hz, 1 H); ^{13}C NMR (C_6D_6) δ 34.2, 54.8, 91.5, 114.1, 114.2, 115.4, 119.9, 128.6, 129.2, 131.2, 135.0, 150.0, 160.8, 163.2; HRMS Calcd for $\text{C}_{16}\text{H}_{15}\text{NO}_3$: M^+ , 269.1052. Found: m/z 269.1042.

1-Methyl-2-phenyl-1,2-dihydro-(4H)-benzo[d]-3,1-oxazin-4-one (3ae).

Isolated in 72% yield as a pale yellow solid: m.p. 101–103 °C; ^1H NMR (C_6D_6) δ 2.12 (s, 3 H), 5.49 (s, 1 H), 6.32 (d, $J = 8.2$ Hz, 1 H), 6.60 (t, $J = 7.5$ Hz, 1 H), 7.01–7.07 (m, 4 H), 7.21–7.23 (m, 2 H), 8.18 (dd, $J = 7.9, 1.5$ Hz, 1 H); ^{13}C NMR (C_6D_6) δ 34.5, 91.5, 114.3, 115.5, 120.1, 127.8, 128.8, 129.5, 131.2, 135.0, 136.7, 149.8, 162.9; HRMS Calcd for $\text{C}_{15}\text{H}_{13}\text{NO}_2$: M^+ , 239.0946. Found: m/z 239.0960.

1-Methyl-2-(1-naphthyl)-1,2-dihydro-(4H)-benzo[d]-3,1-oxazin-4-one (3af).

Isolated in 70% yield as a pale yellow solid: m.p. 109–111 °C; ^1H NMR (C_6D_6) δ 2.04 (s, 3 H), 6.10 (s, 1 H), 6.36 (d, $J = 8.2$ Hz, 1 H), 6.69 (td, $J = 7.5, 1.0$ Hz, 1 H), 7.08–7.12 (m, 2 H), 7.21–7.24 (m, 2 H), 7.33 (d, $J = 7.0$ Hz, 1 H), 7.55–7.61 (m, 2 H), 8.21 (t, $J = 4.9$ Hz, 1 H), 8.29 (dd, $J = 8.0, 1.2$ Hz, 1 H); ^{13}C NMR (C_6D_6) δ 33.5, 91.0, 114.5, 120.3, 124.8, 125.1, 126.3, 127.0, 127.3, 128.5, 129.0, 130.68, 130.74, 131.3, 131.4, 134.4, 135.0, 150.6, 163.4; HRMS Calcd for $\text{C}_{19}\text{H}_{15}\text{NO}_2$: M^+ , 289.1103. Found: m/z 289.1100.

1-Methyl-2-(2-thienyl)-1,2-dihydro-(4H)-benzo[d]-3,1-oxazin-4-one (3ag).

Isolated in 49% yield as a yellow solid: m.p. 95–98 °C; ^1H NMR (C_6D_6) δ 2.21–2.22 (m, 3 H), 5.71 (s, 1 H), 6.26 (d, $J = 8.5$ Hz, 1 H), 6.47–6.50 (m, 1 H), 6.56 (td, $J = 7.5, 1.0$ Hz, 1 H), 6.67 (dt, $J = 5.1, 1.2$ Hz, 1 H), 6.76 (dt, $J = 3.6, 0.9$ Hz, 1 H), 6.99–7.04 (m, 1 H), 8.12 (dd, $J = 7.7, 1.7$ Hz, 1 H); ^{13}C NMR (C_6D_6) δ 35.1, 88.0, 114.4, 115.4, 120.2, 126.6, 126.9, 128.5, 131.0, 135.2, 140.2, 148.5, 162.2; HRMS Calcd for $\text{C}_{13}\text{H}_{11}\text{NO}_2\text{S}$: M^+ , 245.0511. Found: m/z 245.0503.

1-Methyl-2-[4-(trifluoromethyl)phenyl]-1,2-dihydro-(4H)-benzo[d]-3,1-oxazin-4-one (3ah).

Isolated in 31% yield as a pale yellow solid: m.p. 94–96 °C; ^1H NMR ($\text{THF}-d_8$) δ 2.96 (s, 3 H), 6.32 (s, 1 H), 6.92 (t, $J = 7.6$ Hz, 1 H), 7.03 (d, $J = 8.1$ Hz, 1 H), 7.53 (td, $J = 7.8, 1.6$ Hz, 1 H), 7.66 (d, $J = 8.6$ Hz, 2 H), 7.72 (d, $J = 8.6$ Hz, 2 H), 7.85 (dd, $J = 7.8, 1.6$ Hz, 1 H); ^{13}C NMR ($\text{THF}-d_8$) δ 35.9, 91.2, 115.5, 115.7, 120.6, 125.0 ($J_{\text{C-F}} = 272.2$ Hz), 126.3 ($J_{\text{C-F}} = 3.8$ Hz), 128.9, 131.1, 131.7 ($J_{\text{C-F}} = 32.2$ Hz), 136.0, 142.4 ($J_{\text{C-F}} = 1.6$ Hz), 150.0, 162.5; Anal. Calcd for $\text{C}_{16}\text{H}_{12}\text{F}_3\text{NO}_2$: C, 62.54; H, 3.94; N, 4.56. Found: C, 62.44; H, 4.10; N, 4.39.

1-Benzyl-2-(4-methoxyphenyl)-1,2-dihydro-(4H)-benzo[d]-3,1-oxazin-4-one (3ai).

3ai

Isolated in 72% yield as a pale yellow viscous oil: ^1H NMR (THF- d_8) δ 3.73 (s, 3 H), 4.44 (d, $J = 16.3$ Hz, 1 H), 4.66 (d, $J = 16.3$ Hz, 1 H), 6.36 (s, 1 H), 6.83-6.91 (m, 4 H), 7.19-7.39 (m, 8 H), 7.86 (dd, $J = 7.6, 1.7$ Hz, 1 H); ^{13}C NMR (THF- d_8) δ 52.9, 55.3, 91.0, 114.5, 116.4, 116.6, 120.4, 127.8, 127.9, 128.9, 129.2, 129.5, 131.1, 135.4, 138.7, 149.2, 161.4, 163.2; HRMS Calcd for $\text{C}_{22}\text{H}_{19}\text{NO}_3$: M^+ , 345.1365. Found: m/z 345.1360.

1-*n*-Butyl-2-(4-methoxyphenyl)-1,2-dihydro-(4*H*)-benzo[*d*]-3,1-oxazin-4-one (3aj).

3aj

Isolated in 62% yield as a pale yellow solid: m.p. 74–75 °C; ^1H NMR (C_6D_6) δ 0.65 (t, $J = 7.2$ Hz, 3 H), 0.88-1.01 (m, 2 H), 1.17-1.28 (m, 2 H), 2.65-2.77 (m, 1 H), 2.96-3.07 (m, 1 H), 3.18 (s, 3 H), 5.81 (s, 1 H), 6.54-6.65 (m, 4 H), 7.05 (td, $J = 7.4, 1.6$ Hz, 1 H), 7.28 (d, $J = 8.4$ Hz, 2 H), 8.21 (dd, $J = 7.8, 1.5$ Hz, 1 H); ^{13}C NMR (C_6D_6) δ 13.7, 20.2, 30.3, 49.5, 54.7, 90.2, 114.2, 115.7, 117.1, 120.1, 129.0, 129.4, 131.2, 134.8, 148.4, 160.6, 163.3; HRMS Calcd for $\text{C}_{19}\text{H}_{21}\text{NO}_3$: M^+ , 311.1521. Found: m/z 311.1523.

1-*i*-Propyl-2-(4-methoxyphenyl)-1,2-dihydro-(4*H*)-benzo[*d*]-3,1-oxazin-4-one (3ak).

3ak

Isolated in 36% yield as a pale yellow viscous oil: ^1H NMR (C_6D_6) δ 0.91 (d, $J = 6.7$ Hz, 6 H), 3.14 (s, 3 H), 3.52 (quintet, $J = 6.7$ Hz, 1 H), 6.19 (s, 1 H), 6.53 (t, $J = 7.2$ Hz, 1 H), 6.57 (d, $J = 8.6$ Hz, 2 H), 6.65 (d, $J = 8.2$ Hz, 1 H), 7.00 (t, $J = 8.2$ Hz, 1 H), 7.58 (d, $J = 8.6$ Hz, 2 H), 8.08 (d, $J = 7.7$ Hz, 1 H); ^{13}C NMR (C_6D_6) δ 20.9, 21.4, 54.5, 54.6, 85.2, 114.1, 120.4, 120.7, 121.9, 128.5, 130.3, 131.5, 134.4, 147.4, 160.1, 163.3; HRMS Calcd for $\text{C}_{18}\text{H}_{19}\text{NO}_3$: M^+ , 297.1365. Found: m/z 297.1366.

A mixture of 1,6-dimethyl-2-(2,4,6-trimethylphenyl)-1,2-dihydro-(4*H*)-benzo[*d*]-3,1-oxazin-4-one (3ba) and 1,7-dimethyl-2-(2,4,6-trimethylphenyl)-1,2-dihydro-(4*H*)-benzo[*d*]-3,1-oxazin-4-one (3'ba).

Isolated in 74% yield as a pale yellow solid: m.p. 121–123 °C; ^1H NMR (C_6D_6) δ 2.02 (s, 6 H), 2.06 (s, 6 H), 2.14–2.16 (m, 18 H), 6.19 (s, 1 H), 6.21 (s, 1 H), 6.23 (s, 1 H), 6.31 (d, $J = 8.3$ Hz, 1 H), 6.51 (d, $J = 8.0$ Hz, 1 H), 6.66 (s, 4 H), 6.97 (dd, $J = 8.3, 1.7$ Hz, 1 H), 8.20 (s, 1 H), 8.29 (d, $J = 8.0$ Hz, 1 H); ^{13}C NMR (C_6D_6) δ 20.1, 20.5, 20.6, 20.9, 22.1, 31.3, 31.5, 88.9, 89.2, 112.1, 113.3, 114.4, 121.0, 121.1, 128.2, 128.5, 129.0, 130.5, 130.7, 131.6, 131.7, 136.0, 138.4, 139.2, 139.2145.9, 149.1, 151.0, 163.97, 164.04; HRMS Calcd for $\text{C}_{19}\text{H}_{21}\text{NO}_2$: M^+ , 295.1572. Found: m/z 295.1587.

A mixture of 1-methyl-6-fluoro-2-(2,4,6-trimethylphenyl)-1,2-dihydro-(4*H*)-benzo[*d*]-3,1-oxazin-4-one (3ca) and 1-methyl-7-fluoro-2-(2,4,6-trimethylphenyl)-1,2-dihydro-(4*H*)-benzo[*d*]-3,1-oxazin-4-one (3'ca).

Isolated in 63% yield as a yellow solid: m.p. 111–115 °C; ^1H NMR (C_6D_6) δ 1.94 (s, 3 H, *minor*), 2.04–2.13 (m, 21 H), 6.03–6.08 (m, 2 H), 6.09 (s, 1 H, *major*), 6.12 (s, 1 H, *minor*), 6.31 (td, $J = 8.5, 2.2$ Hz, 1 H, *minor*), 6.64 (s, 2 H, *minor*), 6.65 (s, 2 H, *major*), 6.78–6.83 (m, 1 H, *major*), 8.00 (dd, $J = 8.2, 3.1$ Hz, 1 H, *major*), 8.14 (dd, $J = 8.7, 6.5$ Hz, 1 H, *minor*); ^{13}C NMR (C_6D_6) δ 20.4, 20.5, 20.9, 31.3, 31.6, 88.7, 89.3, 99.7, 100.1, 107.0, 107.3, 114.5, 114.6, 115.1, 115.2, 116.7, 117.1, 122.2, 122.6, 128.5, 130.5, 134.4, 134.5, 138.3, 139.4, 147.5, 152.6, 152.8, 154.9, 158.5, 162.9, 165.8, 169.5; HRMS Calcd for $\text{C}_{18}\text{H}_{18}\text{FNO}_2$: M^+ , 299.1322. Found: m/z 299.1317.

A mixture of 1,5-dimethyl-2-(2,4,6-trimethylphenyl)-1,2-dihydro-(4*H*)-benzo[*d*]-3,1-oxazin-4-one (3da) and 1,8-dimethyl-2-(2,4,6-trimethylphenyl)-1,2-dihydro-(4*H*)-benzo[*d*]-3,1-oxazin-4-one (3'da).

Isolated in 71% yield as a pale yellow solid: m.p. 138–141 °C; ^1H NMR (C_6D_6) δ 1.92 (s, 3 H, *minor*), 2.06 (s, 3 H, *major*), 2.09 (s, 3 H, *minor*), 2.17 (s, 12 H), 2.31 (s, 6 H, *minor*), 2.89 (s, 3 H, *major*), 6.14 (s, 1 H, *major*), 6.16 (s, 1 H, *minor*), 6.29 (d, $J = 8.5$ Hz, 1 H, *major*), 6.60 (d, $J = 7.5$ Hz, 1 H, *major*), 6.66 (s, 2 H, *major*), 6.71 (s, 2 H, *minor*), 6.83 (t, $J = 7.6$ Hz, 1 H, *minor*), 6.95 (d, $J = 6.7$ Hz, 1 H, *minor*), 7.03 (t, $J = 7.9$ Hz, 1 H, *major*), 8.25 (d, $J = 7.8$ Hz, 1 H, *minor*); ^{13}C NMR (C_6D_6) δ 16.8, 20.6, 20.8, 20.9, 21.3, 22.8, 31.8, 33.5, 88.3, 91.6, 110.9, 113.5, 122.1, 123.4, 124.9, 128.1, 128.5, 128.6, 130.5, 130.6, 131.6, 133.8, 136.1, 137.6, 138.3, 138.4, 139.2, 144.7, 150.4, 152.4, 163.3, 164.4; Anal. Calcd for $\text{C}_{19}\text{H}_{21}\text{NO}_2$: C, 77.26; H, 7.17; N, 4.74. Found: C, 77.16; H, 7.15; N, 4.84.

5-Methoxy-1-methyl-2-(2,4,6-trimethylphenyl)-1,2-dihydro-(4*H*)-benzo[*d*]-3,1-oxazin-4-one (3ea).

Isolated in 73% yield as a white solid: m.p. 196–198 °C; ^1H NMR (C_6D_6) δ 2.06 (s, 3 H), 2.15 (s, 6 H), 2.20 (s, 3 H), 3.47 (s, 3 H), 6.09 (d, $J = 8.2$ Hz, 1 H), 6.12 (s, 2 H), 6.15 (d, $J = 8.2$ Hz, 1 H), 7.08 (t, $J = 8.2$ Hz, 1 H); ^{13}C NMR (C_6D_6) δ 20.6, 20.9, 31.9, 55.7, 87.9, 103.2, 105.1, 128.1, 128.5, 130.4, 135.0, 138.4, 139.1, 153.4, 160.4, 163.4; HRMS Calcd for $\text{C}_{19}\text{H}_{21}\text{NO}_3$: M^+ , 311.1521. Found: m/z 311.1546.

1,6,7-Trimethyl-2-(2,4,6-trimethylphenyl)-1,2-dihydro-(4*H*)-benzo[*d*]-3,1-oxazin-4-one (3fa).

Isolated in 76% yield as a white solid: m.p. 147–150 °C; ^1H NMR (C_6D_6) δ 1.92 (s, 3 H), 1.96 (s, 3 H), 2.08 (s, 3 H), 2.20 (s, 9 H), 6.24 (s, 1 H), 6.30 (s, 3 H), 6.67 (s, 2 H), 8.14 (s, 1 H); ^{13}C NMR (C_6D_6) δ 18.5, 20.5, 20.6, 20.9, 31.5, 89.2, 112.4, 114.1, 128.1, 128.4, 130.5, 132.1, 138.4, 139.1, 144.7, 149.5, 164.1; Anal. Calcd for $\text{C}_{20}\text{H}_{23}\text{NO}_2$: C, 77.64; H, 7.49; N, 4.53. Found: C, 77.46; H, 7.57; N, 4.63.

1-Methyl-6,7-trimethylene-2-(2,4,6-trimethylphenyl)-1,2-dihydro-(4H)-benzo[d]-3,1-oxazin-4-one (3ga).

Isolated in 64% yield as a white solid: m.p. 131–133 °C; ^1H NMR (C_6D_6) δ 1.76 (quintet, J = 7.3 Hz, 2 H), 2.09 (s, 3 H), 2.206 (s, 6 H), 2.214 (s, 3 H), 2.56–2.63 (m, 4 H), 6.24 (s, 1 H), 6.36 (s, 1 H), 6.68 (s, 2 H), 8.22 (s, 1 H); ^{13}C NMR (C_6D_6) δ 20.6, 20.9, 25.8, 31.69, 31.73, 33.8, 89.1, 108.9, 127.0, 128.4, 128.5, 130.5, 135.6, 138.4, 139.1, 150.4, 152.6, 164.4; HRMS Calcd for $\text{C}_{21}\text{H}_{23}\text{NO}_2$: M^+ , 321.1729. Found: m/z 321.1723.

1-Methyl-6,7-tetramethylene-2-(2,4,6-trimethylphenyl)-1,2-dihydro-(4H)-benzo[d]-3,1-oxazin-4-one (3ha).

Isolated in 47% yield as a white solid: m.p. 197–200 °C; ^1H NMR (C_6D_6) δ 1.47–1.56 (m, 4 H), 2.08 (s, 3 H), 2.20 (s, 3 H), 2.22 (s, 6 H), 2.47–2.53 (m, 4 H), 6.22 (s, 1 H), 6.26 (s, 1 H), 6.68 (s, 2 H), 8.11 (s, 1 H); ^{13}C NMR (C_6D_6) δ 20.6, 20.9, 23.2, 23.4, 28.4, 30.5, 31.5, 89.3, 112.7, 113.0, 128.4, 128.9, 130.5, 132.0, 138.4, 139.1, 145.1, 148.9, 164.1; Anal. Calcd for $\text{C}_{22}\text{H}_{25}\text{NO}_2$: C, 78.77; H, 7.51; N, 4.18. Found: C, 78.71; H, 7.58; N, 4.09.

1-Methyl-2-(2,4,6-trimethylphenyl)-1,2-dihydro-(4H)-naphtho[2,3-d]-3,1-oxazin-4-one (3ia).

Isolated in 82% yield as a yellow solid: m.p. 165–168 °C; ^1H NMR (C_6D_6) δ 2.09 (s, 3 H), 2.21 (s, 6 H), 2.25 (s, 3 H), 6.29 (s, 1 H), 6.68 (s, 2 H), 6.73 (s, 1 H), 7.03–7.07 (m, 1 H), 7.25–7.28 (m, 1 H), 7.51 (t, J = 8.4 Hz, 2 H), 8.89 (s, 1 H); ^{13}C NMR (C_6D_6) δ 20.5, 20.9, 31.8, 89.4, 108.2, 116.4, 124.2, 126.6, 128.0, 128.5, 129.3, 129.9, 130.6, 133.7, 137.9, 138.3, 139.3, 146.8, 163.9; HRMS Calcd for $\text{C}_{22}\text{H}_{21}\text{NO}_2$: M^+ , 331.1572. Found: m/z 331.1574.

1,5,8-Trimethyl-2-(2,4,6-trimethylphenyl)-1,2-dihydro-(4H)-benzo[*d*]-3,1-oxazin-4-one (3ja).

Isolated in 57% yield as a pale yellow solid: m.p. 115–118 °C; ^1H NMR (C_6D_6) δ 1.95 (s, 3 H), 2.08 (s, 3 H), 2.21 (s, 3 H), 2.37 (s, 6 H), 2.85 (s, 3 H), 6.13 (s, 1 H), 6.71 (s, 2 H), 6.74 (d, J = 7.7 Hz, 1 H), 6.88 (d, J = 7.7 Hz, 1 H); ^{13}C NMR ($\text{THF}-d_8$) δ 17.0, 20.8, 21.3, 22.1, 30.6, 33.8, 91.3, 121.2, 125.8, 128.6, 130.1, 130.9, 135.6, 138.2, 138.7, 141.1, 151.8, 163.8; HRMS Calcd for $\text{C}_{20}\text{H}_{23}\text{NO}_2$: M^+ , 309.1729. Found: m/z 309.1722.

References

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040807-9-322-GPC1

File Name
Comment
Slice History
EXMODE

POINT
SAMPD
FREQU
FILTR
DELAY
DEADT
INTVL
TIMES
DUMMY
PD
ACQTM
PREDL
INTWT
RESOL
PW1
OBNUC
OBFRQ
QBSET
RGAIN
SCANS
SLVNT
SPINNING
TEMP

32768 points
32768 points
7917.7 Hz
3950 Hz
50.6 usec
73.2 usec
126.3 usec
16 times
4 times
2.8614 sec
4138.5986 msec
0.01000 msec
1000.0000 msec
0.24 Hz
5.50 usec
1H
395.75 MHz
134498.00 Hz
17
16 times
C606
17 Hz
25.6 C

163.907
150.976
139.290
138.334
135.054
131.610
129.481
128.536
128.247
128.000
127.761
119.602
114.435
112.985
88.953
31.348
20.881
20.510

FileName
Comment
SliceHistory
EXMODE

LoadingFID.rmd
040807-9-332-GPC1
bcm

POINT 32768 points
SAMP 32768 points
FREQ 26881.7 Hz
FILT 13450 Hz
DELT 14.9 usec
DEAD 19.7 usec
TNTL 37.2 usec
TIMES 20000 times
DUMMY 4 times
PD 1.7810 sec
ACQ 1218.9696 msec
PRED 0.01000 msec
ININ 1000.0000 msec
RESOL 0.82 Hz
PW1 5.25 usec
13C 99.45 MHz
104750.00 Hz
1H 26
395.75 MHz
134498.00 Hz
75.0 usec
0

SCANS 790 times
SLVNT C6D6
SPIN 17 Hz
TEMP 26.0 C

200 150 100 50 0 ppm

040731-9-325-GPC1

Loading: ID: nmdata
 040731-9-325-GPC1
 non
 POINT 32768 points
 SAMPD 32768 points
 FREQU 7917.7 Hz
 DELAY 3950 Hz
 DEADT 50.6 usec
 INTVL 73.2 usec
 TIMES 126.3 usec
 DUMMY 16 times
 PD 1 times
 ACQTM 2.8614 sec
 PREOL 4138.5986 msec
 INTWT 0.01000 msec
 RESOL 1000.0000 msec
 PW1 0.24 Hz
 OBNUC 5.50 usec
 OBFREQ 395.75 MHz
 OBFSET 134498.00 Hz
 RGAIN 17
 SCANS 16 times
 SLVNT C6D6
 SPINNING 15 Hz
 TEMP 25.9 C

File Name LoadingFID.nddata
Comment 040802-9-326-GPC1
SliceHistory
EXMODE bcm

POINT 32768 points
SAMPD 32768 points
FREQ 26881.7 Hz
FILTR 13450 Hz
DELAY 14.9 usec
DEADT 19.7 usec
INVT 37.2 usec
TIMES 20000 times
DUMMY 4 times
PD 3.5620 sec
ACQTM 1218.9696 msec
PREDL 0.01000 msec
TNMT 1000.0000 msec
RESOL 0.82 Hz
PW1 5.25 usec

13C 99.45 MHz
104750.00 Hz
25
1H 395.75 MHz
134498.00 Hz
75.0 usec
0

SCANS 621 times
SLVNT C6D6
SPINNING 14 Hz
TEMP 26.3 C

ppm

0

50

100

150

200

File Name: LoadingFID.nmdata
Comment: 040803-9-328-GPC2
Slice History: non
EXMODE: non

POINT 32768 points
SAMPD 32768 points
FREQ 7917.7 Hz
FILT 3950 Hz
DELTA 50.6 usec
DEAD 73.2 usec
INTVL 126.3 usec
TIMES 16 times
DUMMY 4 times
PD 2.8614 sec
ACQTH 4138.5986 msec
PREDL 0.01000 msec
ININT 1000.0000 msec
RESOL 0.24 Hz
PM1 5.50 usec
OBNUC 1H
OBFRQ 395.75 MHz
OBSET 134496.00 Hz
RGAIN 17
SCANS 16 times
SLVNT C506
SPINNT 14 Hz
TEMP 25.8 C

8.2792
8.2749
8.2590
8.2553
7.1756
7.1561
7.1524
7.1500
7.1152
7.1109
7.1091
7.1048
7.0926
7.0920
7.0841
7.0735
7.0719
7.0576
7.0552
6.7887
6.7685
6.7502
6.6812
6.6623
6.6434
6.3700
6.3645
6.3492
6.3431
5.7724

2.1408
2.1250
2.1189
2.1030
2.0542

File Name : LoadingID.mdata
Comment : 040803-9-328-GPC2
Slice History :
EXMODE : bcm

POINT : 32768 points
SAMPD : 32768 points
FREQU : 26881.7 Hz
FILT : 13450 Hz
DELTA : 14.9 usec
DEADT : 19.7 usec
INVT : 37.2 usec
TIMES : 20000 times
DUMMY : 4 times
PD : 1.7810 sec
ACQTM : 1218.9696 msec
PREDL : 0.01000 msec
ININT : 1000.0000 msec
RESOL : 0.82 Hz
PW1 : 5.25 usec

13C : 99.45 MHz
OBFRQ : 104750.00 Hz
OBSET : 26
RGAIN : 1H
IRFRO : 395.75 MHz
IRFSE : 134498.00 Hz
IRPPM : 75.0 usec
IRPNS : 0

SCANS : 827 times

SLVNT : C6D6
SPINNING : 15 Hz
TEMP : 26.1 C

040707-8-307-GPC1

OFILF JJJJ
 QBNUC 1H
 EXMOD NON
 QFR 270.05 MHZ
 QBSET 112.00 KHZ
 QBFIN 5800.0 HZ
 POINT 32768
 FREQU 5405.4 HZ
 SCANS 16
 ACQTM 3.031 sec
 PD 3.969 sec
 PW1 5.2 us
 IRNUC 1H
 CTEMP 20.4 C
 SLVNT C6D6
 EXREF 7.15 ppm
 BF 0.16 HZ
 RGAIN 17
 OPERATOR :

File Name LoadingFID.rmdata
Comment 040708-8-307-GPC1
Spectral History
EXMODE bcm

POINT 32768 points
SAMPD 32768 points
FREQ 26881.7 Hz
FILTR 13450 Hz
DELAY 14.9 usec
DEADT 19.7 usec
INTVL 37.2 usec
TIMES 20000 times
DUMMY 4 times
PD 1.7810 sec
ACQTM 1218.9696 msec
PREOL 0.01000 msec
ININT 1000.0000 msec
RESOL 0.82 Hz
PM1 5.25 usec

13C 99.45 MHz
104750.00 Hz
26
1H 395.75 MHz
134498.00 Hz
75.0 usec
0

SCANS 569 times
SLVNT C6D6
SPINNING 15 Hz
TEMP 23.6 C

34.224

54.769

91.467

114.130

114.237

115.440

119.932

127.753

128.000

128.239

128.560

129.236

131.173

135.030

149.996

160.849

163.206

ppm

0

50

100

150

200

040718-9-318-GPC1

3.2547

2.1244

File Name
Comment
Slice History
EXMODE

.LoadingFID.nmdata
040718-9-318-GPC1
non

POINT 32768 points
SAMPD 32768 points
FREQ 7917.7 Hz
FILT 3950 Hz
DEL 50.6 usec
DEAD 73.2 usec
INTVL 126.3 usec
TIMES 15 times
DUMMY 4 times
PD 2.8614 sec
ACQTM 4138.5986 msec
PREOL 0.01000 msec
INW 1000.0000 msec
RESOL 0.24 Hz
PW1 5.50 usec
OBNUC 1H
OBPRQ 395.75 MHz
OBSET 134498.00 Hz
RGAIN 18
SCANS 16 times
SLVNT CDCl3
SPINNT 13 Hz
TEMP 26.4 C

ppm

0

2

4

6

8

File Name
Comment
Slice History
EXMODE

POINT
SAMPL
FREQ
FILT
DELAY
DEAD
INVT
TIMES
DUMM
PD
ACQ
PRED
INVT
RESOL
PW1
OBNUC
OBFRQ
OBSET
RGAIN
IRNUC
IRFRQ
IRSET
IRPRW
IRRS

32768 points
32768 points
26881.7 Hz
13450 Hz
14.9 usec
19.7 usec
37.2 usec
20000 times
4 times
1.7810 sec
1218.9596 msec
0.01000 msec
1000.0000 msec
0.82 Hz
5.25 usec
13C
99.45 MHz
104750.00 Hz
26
1H
395.75 MHz
134498.00 Hz
75.0 usec
0

bcm

SCANS
SLVNT
SPINNING
TEMP

725 lines
C6D6
14 Hz
26.5 C

34.512

91.483

114.344

115.548

120.097

127.753

127.810

128.000

128.239

128.775

129.508

131.156

135.038

136.686

149.814

162.943

ppm

0

50

100

150

200

040831-9-341-GPC1

```

DFILE JUL
QBNUC 1H
EXMOD NON
QFR
QBSCT
QBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC 1H
CTEMP
SLVNT C6D6
EXREF
BF
RGAIN
OPERATOR :

```

File Name : .LoadingFID.nmdata
Comment : 040901-9-341-GPC1
Slice History :
EXMODE : hcm

POINT : 32768 points
SAMPD : 32768 points
FREQ : 26881.7 Hz
FILT : 13450 Hz
DELAY : 14.9 usec
DEAD : 19.7 usec
INTVL : 37.2 usec
TIMES : 20000 times
DUMMY : 4 times
P0 : 1.7810 sec
ACQTM : 1218.9696 msec
PREDL : 0.01000 msec
IN1WT : 1000.0000 msec
RESOL : 0.83 Hz
PWI : 5.25 usec

13C : 99.45 MHz
104750.00 Hz
25

1H : 395.75 MHz
134498.00 Hz
75.0 usec
0

SCANS : 11786 times

SLVNT : C6D6
SPINNING : 13 Hz
TEMP : 26.3 C

8.1369
8.1327
8.1174
8.11317.1531
7.1500
7.0383
7.0353
7.0310
7.0169
7.0139
7.0102
6.9986
6.9956
6.9919
6.7679
6.7655
6.7630
6.7588
6.7563
6.7539
6.6788
6.6758
6.6727
6.6660
6.6629
6.6599
6.6575
6.5799
6.5775
6.5592
6.5421
6.5396
6.4957
6.4933
6.4865
6.4835
6.4804
6.4743
6.4713
6.2705
6.2491
5.7102
5.7077 2.2153
2.2116

3.00

1.00

1.03

0.95

1.04

1.04

0.98

1.01

1.08

File Name : LoadingID.nmdata
Comment : 040904-10-343-GPC1
Slice history : non
EXMODE :
POINT : 32768 points
SAMPD : 32768 points
FREQ : 7917.7 Hz
FILT : 3950 Hz
DELTA : 50.6 usec
DEAD : 73.2 usec
INTVL : 126.3 usec
TIMES : 16 times
DUMMY : 4 times
PD : 2.8614 sec
ACQTM : 4138.5986 msec
PREDL : 0.01000 msec
ININT : 1000.0000 msec
RESOL : 0.24 Hz
PM1 : 5.50 usec
CBNUC : 1H
CBFRQ : 395.75 MHz
OBSET : 134498.00 Hz
RGAIN : 19
SCANS : 16 times
SLVNT : C6D6
SPINNT : 15 Hz
SPINNG : 24.9 C
TEMP :

File Name
Comment
SliceHistory
EXMODE

LoadingID: mdata
040904-10-343-GPC1
bcm

POINT 32768 points
SAMPD 32768 points
FREQ 26881.7 Hz
FILT 13450 Hz
DELAY 14.9 usec
DEADT 19.7 usec
INVT 37.2 usec
TIMES 20000 times
DUMY 4 times
PD 1.7810 sec
ACQTM 1218.9696 msec
PREDL 0.01000 msec
INVT 1000.0000 msec
RESOL 0.82 Hz
PM1 5.25 usec

13C 99.45 MHz
104750.00 Hz
26
1H 395.75 MHz
134498.00 Hz
75.0 usec
0

SCANS 1411 times

SLVNT C6D6
SPINNING 14 Hz
TEMP 25.8 C

162.481
150.013
142.439
136.019
132.220
131.898
131.577
131.247
131.107
128.948
126.385
126.352
126.311
126.270
120.592
115.729
115.474

91.154

67.733
67.510
67.288
67.065
66.843

35.938
25.604
25.406
25.200
24.796

File Name
Comment
Slice History
EXMODE

Loading FID.nmdata
040827-9-336-GPC1
bcm

POINT 32768 points
SAMPL 32768 points
FREQ 26681.7 Hz
FILT 13450 Hz
DEL 14.9 usec
DELT 19.7 usec
INVT 37.2 usec
TIMES 20000 times
DUMMY 4 times
PD 1.7810 sec
ACQTM 1218.9695 msec
PREDL 0.01000 msec
ININT 1000.0000 msec
RESOL 0.82 Hz
PW1 5.25 usec
13C 13C 99.45 MHz
104750.00 Hz
28
1H 395.75 MHz
134498.00 Hz
75.0 usec
0
SCANS 12954 times
SLVNT DMSO
SPINNING 3 Hz
TEMP 26.2 C

ppm

040704-8-304-GPC1

DFILE JJJJ
 DENDUC 1H
 EXMOD HMN
 OFR 270.05 MHZ
 OBSET 112.00 KHZ
 DBFIN 5800.0 HZ
 POINT 32768
 FREQU 5405.4 HZ
 SCANS 16
 ACQTM 3.031 sec
 PD 3.969 sec
 PW1 5.2 us
 INNUC 1H
 CTEMP 21.0 C
 SLVIT C6D6
 EXREF 7.15 ppm
 BF 0.16 HZ
 RGAIN 16
 OPERATOR :

040521-8-276-GPC2

DFILE JJJ
 QBNUC 13C
 EXMOD BCM
 QFR 67.80 MHz
 QBCET 135.00 kHz
 QBFIN 5200.0 Hz
 POINT 32768
 FREQU 20000.0 Hz
 SCANS 12361
 ACQTM 0.819 sec
 PD 2.181 sec
 PW1 3.8 us
 IRNUC 1H
 CTEMP 28.6 C
 SLVNT CD300
 EXREF 25.20 ppm
 BF 1.22 Hz
 RGAIN 24
 OPERATOR :

3aj

040702-8-301-GPC1

OFILE .J.J.J
 QBNUC 1H
 EXMOD NON
 QFR 270.05 MHz
 QBSSET 112.00 KHz
 QBFIN 5800.0 Hz
 POINT 32768
 FREQU 5405.4 Hz
 SCANS 16
 ACQTM 3.031 sec
 PD 3.959 sec
 PW1 5.2 us
 IRNUC 1H
 CTEMP 21.7 C
 SLVNT CDCl₃
 EXREF 7.15 ppm
 BF 0.16 Hz
 RGAIN 18
 OPERATOR :

OFILF	JJL				
OBNUC	L3C				
EXMOD	BCM				
QFR		67.80	MHz		
OBSCT		135.00	KHz		
OBFIN		5200.0	HZ		
PONT		32768			
FREQU		20000.0	HZ		
SCANS		13773			
ACQTU		0.819	sec		
PD		2.181	sec		
PW1		3.8	us		
IRNUC	1H				
CTEMP		28.6	C		
SLVNT	C6D6				
EXREF		128.00	ppm		
Bf		1.22	HZ		
RGAIn	24				
OPERATOR :

LoadingFID.ndata
040715-9-314-GPC1

non

File Name
Comment
Slice History
EXMODEPOINT 32768 points
SAMPD 32768 points
FREQU 7917.7 Hz
FILTR 3950 Hz
DELAY 50.6 usec
DEAD 73.2 usec
INVT 126.3 usec
TIMES 16 times
DUMMY 4 timesPD 2.8614 sec
ACQTM 4138.5986 msec
FREDL 0.01000 msec
ININT 1000.0000 msec
RESOL 0.24 Hz
PW1 5.50 usec1H 395.75 MHz
134498.00 Hz
18

SCANS 16 times

C6D6

SPINNING 0 Hz
TEMP 25.8 C0.9146
0.89763.5660
3.5495
3.5324
3.5154
3.4983
3.4812
3.4647
3.10697.1500
7.0163
6.9956
6.9773
6.6629
6.6422
6.5848
6.5628
6.5512
6.5311
6.5128
6.18697.430
7.42168.0893
8.0698

6.15

3.11

1.06

1.20

2.03

1.00

3.02

1.01

ppm

0

2

4

6

8

File Name: .LoadingID.mdata
Comment: 040715-9-314-GPC1
Slice History: bcm
EXMODE: POINT 32768 points
SAMPLING: 32768 points
FREQ: 26881.7 Hz
FILT: 13450 Hz
DELTA: 14.9 usec
DEAD: 19.7 usec
INTVL: 37.2 usec
TIMES: 20000 times
DUMMY: 4 times
PD: 1.7810 sec
ACQTH: 1218.9696 msec
PREDL: 0.01000 msec
ININT: 1000.0000 msec
RESOL: 0.82 Hz
PWL: 5.25 usec
13C: 99.45 MHz
OBFRQ: 104750.00 Hz
RGAIN: 26
1H: 395.75 MHz
134498.00 Hz
IRPWL: 75.0 usec
IRPWS: 0
SCANS: 1746 times
SLVNT: CDCl3
SPINNING: 0 Hz
TEMP: 26.2 C

ppm

DFILE JJJ

OBNUC 1H

EXMOD NON

270.05 MHz OFR

OBSET 112.00 KHZ

5800.0 Hz
OBFIN

POINT 32768

FREQU 5405.4 HZ

SCANS
16

ACQTM 3.031 sec

3.969 sec

PW1 5.2 us

IRNUC 1H

25.7 C CTMP

SI VNT C6D6

FREE

0.16 Hz

RGATN 16

OPERATOR :

FileName
Comment
SliceHistory
EXMODE

.LoadingFID.nmdata
041008-10-373-GPC1
bcm

POINT 32768 points
SAMP0 32768 points
FREQ0 26881.7 Hz
FILTR 13450 Hz
DELAY 14.9 usec
DEADT 19.7 usec
INTVL 37.2 usec
TIMES 20000 times
DUMMY 4 times
PO 1.7810 sec
ACQTM 1218.9696 msec
PREDL 0.01000 msec
ININT 1000.0000 msec
RESOL 0.82 Hz
PW1 5.25 usec
13C 99.45 MHz
OBFRQ 104750.00 Hz
OBSET 26
1H 395.75 MHz
IRFRQ 134498.00 Hz
IRBPW 75.0 usec
IRPNS 0
SCANS 1401 times
SLWNT 17 Hz
SPINNING 23.8 C
TEMP

20.123
20.527
20.552
20.889
20.896
22.076
31.331
31.488

164.047
163.973
151.018
149.130
145.941
139.216
139.200
138.351
136.010
131.692
131.601
130.711
130.481
128.964
128.536
128.247
128.165
128.000
127.761
121.069
120.954
114.435
113.298
113.067
112.078
89.209
88.912

ppm

0

50

100

150

200

2.1311
2.1018
2.0700
2.0615
2.0511
1.9443

8.1552
8.1382
8.1333
8.1168
8.0210
8.0143
8.0063
8.0002
7.9935
7.9856

7.1537
7.1500
6.8326
6.8253
6.8100
6.8052
6.8021
6.7899
6.7820
6.6550
6.6385
6.6340
6.6301
6.6187
6.6126
6.6067
6.5912
6.5858
6.0953
6.0892
6.0782
6.0660
6.0556
6.0410
6.0355

62.51

2.96

1.00
4.11

4.50
10.76

1.12
1.47

9.63

File Name
Comment
Slice History
EXMODE

Loading F10.rmd data
041021-11-389-GPC1
non

POINT
SAMPD
FREQ
FILTR
DELT
INTVL
TIMES
DUMMY

32768 points
32768 points
7917.7 Hz
3950 Hz
50.6 usec
73.2 usec
126.3 usec
16 times
4 times

PO
ACQTM
PREDL
INWIT
RESOL
PW1

2.8614 sec
4138.5986 msec
0.01000 msec
1000.0000 msec
0.24 Hz
5.50 usec

OBNUC
OBFRQ
OBSET
RGAIN

1H
395.75 MHz
134498.00 Hz
16

SCANS

16 times

SLVNT
SPINNT
TEMP

C506
17 Hz
25.1 C

ppm

041022-11-389-GPC1

DFILE	JJJ
OBNUC	13C
EXMOD	BCM
QFR	67.80 MHz
OBSET	135.00 kHz
OBFIN	5200.0 Hz
POINT	32768
FREQ	20000.0 Hz
SCANS	12410
ACQTM	0.819 sec
PD	2.181 sec
PW1	3.8 us
IRNUC	1H
CTEMP	26.0 c
SLVNT	C6D6
EXREF	128.00 ppm
BF	1.22 Hz
RGAIN	26
OPERATOR	:

LoadingID: mdata
041109-11-403-GPC1File Name
Comment
Slice History
EXMODE

non
POINT 32768 points
SAMPD 32768 points
FREQ 7917.7 Hz
FILTR 3950 Hz
DELAY 50.6 usec
DEADT 73.2 usec
TNTVL 126.3 usec
TIMES 16 times
DUMMY 1 times
PD 2.8614 sec
ACQTM 4138.5986 msec
PREDL 0.01000 msec
ININT 1000.0000 msec
RESOL 0.24 Hz
PM1 5.50 usec
1H 395.75 MHz
134498.00 Hz
16
SCANS 16 times
SLVNT C6D6
SPINNING 14 Hz
TEMP 25.9 C

0.5339

2.3178
2.1683
2.0896
2.0633
1.9150

2.8934

7.1500
7.0499
7.0310
7.0102
6.9541
6.9370
6.9358
6.8515
6.8326
6.8131
6.7105
6.6550
6.6141
6.5952
6.2986
6.2772
6.1618
6.13808.2376
8.2572 5.53
5.20
1.21
2.14
5.46
5.24
5.389

1.00

ppm

File Name: 041110-11-403-GPC1
Comment: bcm
Slice History: EXMODE
POINT: 32768 points
SAMPLING: 32768 points
FREQ: 26881.7 Hz
FILT: 13450 Hz
DELTA: 14.9 usec
DEAD: 19.7 usec
INTVL: 37.2 usec
TIMES: 20000 times
DUMY: 4 times
PD: 1.7810 sec
ACQTM: 1218.9696 msec
PREDL: 0.01000 msec
INWIT: 1000.0000 msec
RESOL: 0.62 Hz
PM1: 5.25 usec
OBNUC: 13C
OBFRQ: 99.45 MHz
OBSET: 104750.00 Hz
RGAIN: 26
IRNUC: 1H
IRFRQ: 305.75 MHz
IRSET: 134498.00 Hz
IRPW: 75.0 usec
IRNS: 0
SCANS: 8928 times
SLVNT: C5D6
SPINNT: 15 Hz
TEMP: 25.0 C

041006-10-371-GPC1

1.000
2.105
3.236

3.113

1.000
2.105
3.236

DFILE JJJ
OBNUC 1H
EXMOD NON
OFR 270.05 MHz
OBSET 112.00 kHz
OBFIN 5800.0 Hz
POINT 32768
FREQU 5405.4 Hz
SCANS 16
ACQTM 3.031 sec
PD 3.969 sec
PW1 5.2 us
IRNUC 1H
CTEMP 26.0 c
SLVNT C6D6
EXREF 7.15 ppm
B1 0.16 Hz
RGAIN 16
OPERATOR :

PPM
8
7
6
5
4
3
2
1
0

7.150
7.115
7.080
7.045
6.980
6.945
6.910
6.875
6.840
6.805
6.770
6.735
6.700
6.665
6.630
6.595
6.560
6.525
6.490
6.455
6.420
6.385
6.350
6.315
6.280
6.245
6.210
6.175
6.140
6.105
6.070
6.035
6.000
5.965
5.930
5.895
5.860
5.825
5.790
5.755
5.720
5.685
5.650
5.615
5.580
5.545
5.510
5.475
5.440
5.405
5.370
5.335
5.300
5.265
5.230
5.195
5.160
5.125
5.090
5.055
5.020
4.985
4.950
4.915
4.880
4.845
4.810
4.775
4.740
4.705
4.670
4.635
4.600
4.565
4.530
4.495
4.460
4.425
4.390
4.355
4.320
4.285
4.250
4.215
4.180
4.145
4.110
4.075
4.040
4.005
3.970
3.935
3.900
3.865
3.830
3.795
3.760
3.725
3.690
3.655
3.620
3.585
3.550
3.515
3.480
3.445
3.410
3.375
3.340
3.305
3.270
3.235
3.200
3.165
3.130
3.095
3.060
3.025
2.990
2.955
2.920
2.885
2.850
2.815
2.780
2.745
2.710
2.675
2.640
2.605
2.570
2.535
2.500
2.465
2.430
2.395
2.360
2.325
2.290
2.255
2.220
2.185
2.150
2.115
2.080
2.045
2.010
1.975
1.940
1.905
1.870
1.835
1.800
1.765
1.730
1.695
1.660
1.625
1.590
1.555
1.520
1.485
1.450
1.415
1.380
1.345
1.310
1.275
1.240
1.205
1.170
1.135
1.100
1.065
1.030
1.000
0.965
0.930
0.895
0.860
0.825
0.790
0.755
0.720
0.685
0.650
0.615
0.580
0.545
0.510
0.475
0.440
0.405
0.370
0.335
0.300
0.265
0.230
0.195
0.160
0.125
0.090
0.055
0.020

3.469

041006-10-371-GPC1

DFILE JJJ
 OBNUC 13C
 EXMOD BCM
 OFR 67.80 MHz
 OBSET 135.00 kHz
 OBFIN 5200.0 Hz
 POINT 32768
 FREQU 20000.0 Hz
 SCANS 11487
 ACQTM 0.819 sec
 PD 2.181 sec
 PW1 3.8 us
 IRNUC 1H
 CTEMP 26.0 c
 SLVNT C6D6
 EXREF 128.00 ppm
 BF 1.22 Hz
 RGAIN 27
 OPERATOR :

File Name: LoadingF10.nmdata
Comment: 040917-10-355-GPC1
Slice History: non
EXMODE: non

POINT: 32768 points
SAMP0: 32768 points
FREQ0: 7917.7 Hz
FILT0: 3950 Hz
DELT0: 50.6 usec
DEAD0: 73.2 usec
INTVL: 126.3 usec
TIMES: 16 times
DUMY: 4 times
PD: 2.8614 sec
ACQTM: 4138.5986 msec
PREDL: 0.01000 msec
TINT: 1000.0000 msec
RESOL: 0.24 Hz
PM1: 5.50 usec
OBNUC: ¹H
QBFRG: 395.75 MHz
QBSET: 134498.00 Hz
RGAIN: 15
SCANS: 16 times

SLVNT: C6D6
SPINNT: 15 Hz
TEMP: 24.7 C

Filename LoadingFID.nmdata
Comment 040917-10-355-GPC1
SliceHistory
EXMODE bcm

POINT 32768 points
SAMPD 32768 points
FREQD 26881.7 Hz
FILTR 13450 Hz
DELAY 14.9 usec
DEADT 19.7 usec
INTVL 37.2 usec
TIMES 20000 times
DUMMY 4 times
PD 1.7810 sec
ACQTM 1218.9696 msec
PREDL 0.01000 msec
INVT 1000.0000 msec
RESOL 0.82 Hz
PW1 5.25 usec
13C 99.45 MHz
OBFRQ 104750.00 Hz
RGAIN 26
1H 395.75 MHz
IRFRQ 134498.00 Hz
IRSET 75.0 usec
IRPW 0
IRRS 0
SCANS 928 times
SLVNT C6D6
SPINNING 14 Hz
TEMP 24.3 C

ppm

0

50

100

150

200

040910-10-351-GPC1

LoadingFID.nmdata
 Comment 040910-10-351-GPC1
 SliceHistory non
 EXMODE
 POINT 32768 points
 SAMPD 32768 points
 FREQU 7917.7 Hz
 FILTR 3950 Hz
 DELAY 50.5 usec
 DEAT 73.2 usec
 INTVL 126.3 usec
 TIMES 16 times
 DUMMY 4 times
 PD 2.8614 sec
 ACQTM 4138.5986 msec
 PREDL 0.01000 msec
 INIWT 1000.0000 msec
 RESOL 0.24 Hz
 PW1 5.50 usec
 1H 395.75 MHz
 13C 134498.00 Hz
 15
 SCANS 16 times
 SLVNT C6D6
 SPINNT 17 Hz
 TEMP 241.7 C

2.6303
 2.6108
 2.5943
 2.5815
 2.5620
 2.5400
 2.5223
 2.2141
 2.2061
 2.0853
 1.8015
 1.7832
 1.7643
 1.7459
 1.7264

8.2157
 7.1500
 7.1469
 6.6764
 6.3596
 6.3492
 6.2351

1.00
 2.43
 1.02
 1.00

4.36
 9.15
 3.25
 2.08

ppm

File Name
Comment
Slice History
EXMODE

Loading ID: nmdata
040910-10-351-GPC1
bcm

POINT 32768 points
SAMPD 32768 points
FREQ 26881.7 Hz
FILTR 13450 Hz
DEADT 14.9 usec
INVL 19.7 usec
TIMES 37.2 usec
20000 times
DUMMY 4 times
PD 1.7810 sec
ACQTM 1218.5656 msec
PREDL 0.01000 msec
ININT 1000.0000 msec
RESOL 0.82 Hz
PH1 5.25 usec
13C 99.45 MHz
104750.00 Hz
26
1H 395.75 MHz
134498.00 Hz
75.0 usec
0
SCANS 1154 times
SLVNT
SPINNING
TEMP 16 Hz
25.3 C

20.593
20.898
25.793
31.694
31.735
33.795

89.110

108.913

127.011
127.753
128.000
128.239
128.404
128.536
130.472
135.607
138.359
139.126
150.350
152.633

164.377

ppm

1.4713
1.4872
1.5018
1.5013
1.5158
1.5293
1.5470
1.5610
1.5781
2.0841
2.1976
2.2202
2.4698
2.4845
2.5113
2.5266

8.1101
7.1543
7.1500
5.6776
6.2571
6.2235

4.19
3.01
4.31

1.00
2.03
2.09

Filename : LoadingCID.nmdata
Comment : 040914-10-353-GPC1
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPD : 32768 points
FREQU : 7917.7 Hz
FILTR : 3950 Hz
DELAY : 50.6 usec
DEADT : 73.2 usec
INVL : 126.3 usec
TIMES : 16 times
DUMMY : 4 times
PD : 2.8614 sec
ACQTM : 4138.5986 msec
PREDL : 0.01000 msec
TNINT : 1000.0000 msec
RESOL : 0.24 Hz
PWI : 5.50 usec
DBNUC : 395.75 MHz
DBFRO : 134488.00 Hz
ORSET : 17
RGAIN :
SCANS : 16 times
SLVMT : C6D6
SPINNT : 15 Hz
TEMP : 23.9 C

File Name : LoadingFTD.mdata
Comment : 040914-10-353-GPC1
Slice history : bcm
EXMODE :
POINT : 32768 points
SAMPO : 32768 points
FREQ : 26881.7 Hz
FILT : 13450 Hz
DELAY : 14.9 usec
DEADT : 19.7 usec
INTVL : 37.2 usec
TIMES : 20000 times
DUMMY : 4 times
PD : 1.7810 sec
ACQTM : 1218.9696 msec
PREDL : 0.01000 msec
ININT : 1000.0000 msec
RESOL : 0.82 Hz
PWI : 5.25 usec
13C : 99.45 MHz
104750.00 Hz
1H : 395.75 MHz
134498.00 Hz
75.0 usec
0
SCANS : 760 times
SLVNT : C6D6
SPINNING : 14 Hz
TEMP : 24.2 C

File Name LoadingF10.nmdata
Comment 040925-10-363-GPC1
Slice History non
EXMODE

POINT 32768 points
SAMPD 32768 points
FREQ 7917.7 Hz
FILTR 3950 Hz
DELAY 50.6 usec
DEADT 73.2 usec
INTVL 126.3 usec
TIMES 16 times
DUMMY 4 times
PO 2.0614 sec
ACQTM 4138.5986 msec
PREDL 0.01000 msec
ININT 1000.0000 msec
RESOL 0.24 Hz
PWI 5.50 usec
1H 395.75 MHz
134498.00 Hz
17

SCANS 16 times

SLVNT C6D6
SPINNT 16 Hz
TEMP 24.0 C

7.5309
7.5107
7.4867
7.2843
7.2666
7.2635
7.2458
7.1500
7.0688
7.0517
7.0487
7.0316
6.7282
6.6825
6.2918

2.2458
2.2110
2.0877

1.00

2.02

1.09

1.10

3.00

0.97

9.01

2.96

ppm

FileName : LoadingID_nodata
Comment : 040925-10-363-GPC1
SliceHistory :
EXMODE : bcm

POINT : 32768 points
SAMPD : 32768 points
FREQ : 26881.7 Hz
FILT : 13450 Hz
DELT : 14.9 usec
DEAD : 19.7 usec
INVL : 37.2 usec
TIMES : 20000 times
DUMY : 4 times
PO : 1.7810 sec
ACQTM : 1218.9596 msec
PREDL : 0.01000 msec
ININT : 1000.0000 msec
RESOL : 0.82 Hz
PW1 : 5.25 usec
13C : 99.45 MHz
OBFRQ : 104750.00 Hz
OBSET : 26
RGAIN : 395.75 MHz
IRNUC : 134498.00 Hz
IRFRO : 75.0 usec
IRRPW : 0
IRHNS : 0

SCANS : 1849 times
SLWNT : 6606
SPINNT : 16 Hz
TEMP : 24.4 C

31.801
20.906
20.510

146.848
139.307
138.326
137.865
133.719
130.555
129.895
129.327
128.527
128.239
128.049
128.000
127.753
126.624
124.193
116.380
108.155
89.407

163.874

200 150 100 50 0 ppm


```

DFILE JJJ
OBNUC 1H
EXMOD NON
OFR
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC 1H
CTEMP
SLVNT C6D6
EXREF
BF
RGAIN 1
OPERATOR :

```

0.973
1.000
2.972

→
→
→

Aja

041019-11-387-GPC2

3ja

DFILE JJJ
 OBNUC 13C
 EXMOD BCM
 OFR 67.80 MHz
 OBSET 135.00 kHz
 OBFIN 5200.0 Hz
 POINT 32768
 FREQU 20000.0 Hz
 SCANS 13157
 ACQTM 0.819 sec
 PD 2.181 sec
 PW1 3.8 us
 IRNUC 1H
 CTEMP 26.2 c
 SLVNT C6D6
 EXREF 128.00 ppm
 BF 1.22 Hz
 RGAIN 26
 OPERATOR :