From Alkenes to Isoxazolines via Copper-Mediated Alkene Cleavage and Dipolar Cycloaddition

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Supporting Information

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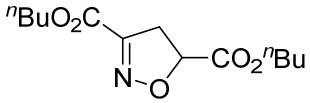
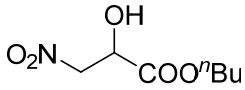
1. General Information

All reagents and metal salts were obtained from commercial sources without further purification, and commercially available solvents were purified before use. All new compounds were fully characterized. All melting points were taken on a SGW_R X-4A Digital Melting Point Apparatus without correction. Infrared spectra were obtained using a Nicolet AVATAR 370 FT-IR spectrometer. ^1H, ^{13}C, and ^{19}F NMR spectra were recorded with a Bruker AV-500 spectrometer operating at 500 MHz, 125 MHz and 470 MHz, respectively, with chemical shift values being reported in ppm relative to chloroform (δ = 7.26 ppm), dimethyl sulfoxide (δ = 2.50 ppm) or TMS (δ = 0.00 ppm) for ^1H NMR; chloroform (δ = 77.16 ppm) or dimethyl sulfoxide (δ = 39.52 ppm) for ^{13}C NMR; and C_6F_6 (δ = -164.9 ppm) for ^{19}F NMR. Mass spectra and high resolution mass spectra (HRMS) were recorded with an Agilent 5975N using an Electron impact (EI) or Electrospray ionization (ESI) techniques. Silica gel plate GF254 were used for thin layer chromatography (TLC) and silica gel H or 300-400 mesh were used for flash column chromatography. Yields refer to chromatographically and spectroscopically pure compounds, unless otherwise indicated. Unless commercially available starting materials, 1-phenylethyl acrylate,[1] mesityl acrylate,[2] 1-(4-chlorophenyl)prop-2-en-1-one,[3] 1-(*p*-tolyl)prop-2-en-1-one,[3] 1-(furan-2-yl)prop-2-en-1-one,[4] 3-vinylbenzonitrile[5] and 1-cyclopropyl-1*H*-pyrrole-2,5-dione[6] were all prepared according to the literature reported procedures.

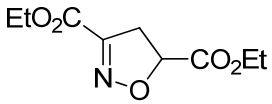
2. Synthesis and Characterization of Substrates and Products



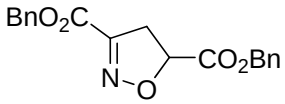
Dibutyl 4,5-dihydroisoxazole-3,5-dicarboxylate (2a) (General Procedure): To a test tube were added **1a** (58 μL, 0.4 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (96.6 mg, 0.4 mmol), KI (66.4 mg, 0.4 mmol), $B(OH)_3$ (24.7 mg, 0.4 mmol) and CH_3CN/PhCN (2:1, v/v, 2.0 mL). The reaction was stirred at 80 ^oC under air as monitored by TLC. Upon completion, the reaction mixture was cooled down to room temperature, quenched with ammonium hydroxide and extracted with ethyl acetate (3×10 mL). The combined organic phase was washed with water and brine, and dried over anhydrous Na_2SO_4. After filtration through a thin pad of celite, the filtrate was evaporated under reduced pressure to give the crude product, which was further purified by flash column chromatography on silica gel (PE/EtOAc = 10:1, v/v) to give **2a** as a pale yellow oil (47.4 mg, 87%). IR (KBr, cm^{-1}): 2962, 1732, 1597, 1254, 1206, 916, 746; ^1H NMR ($CDCl_3$, 500 MHz): δ 5.21-5.11 (m, 1H), 4.26 (t, J = 6.5 Hz, 2H), 4.17 (t, J = 6.5 Hz, 2H), 3.53-3.40 (m, 2H), 1.72-1.59 (m, 4H), 1.44-1.31 (m, 4H), 0.96-0.87 (m, 6H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 169.1, 160.1, 151.1, 80.0, 66.3, 66.2, 37.7, 30.5, 19.1, 19.0, 13.7; LC-MS (ESI) *m/z* 272 $[M+H]^+$; HRMS (ESI) *m/z* calcd for $C_{13}H_{22}NO_5$ $[M+H]^+$ 272.1492, found 272.1493.



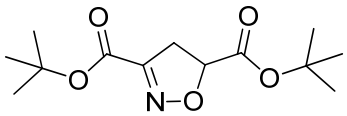
Butyl 2-hydroxy-3-nitropropanoate (2a'):[7] Following the general procedure, the reaction of **1a** (58 μL, 0.4 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (96.6 mg, 0.4 mmol) in CH_3CN (2.0 mL) afforded the desired product **2a** (17.8 mg, 33%) and the by-product **2a'** by flash column chromatography (PE/EtOAc = 5:1, v/v) as a pale yellow oil (20.4 mg, 27%). IR (KBr, cm^{-1}): 3479, 2964, 2875, 1743, 1561, 1464, 1422, 1380, 1215, 1124, 1061, 943, 845, 679, 540; ^1H NMR ($CDCl_3$, 500 MHz): δ 4.80-4.71 (m, 2H), 4.65-4.61 (m, 1H), 4.33-4.23 (m, 2H), 3.38 (d, J = 5.0 Hz, 1H), 1.71-1.64 (m, 2H), 1.42-1.33 (m, 2H), 0.94 (t, J = 7.5 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.9, 76.9, 67.7, 67.1, 30.5, 19.1, 13.7; LC-MS (ESI) *m/z* 214 $[M+Na]^+$.



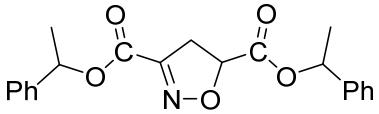
Diethyl 4,5-dihydroisoxazole-3,5-dicarboxylate (2b):[8] Following the general procedure, the reaction of **1b** (43 μL, 0.4 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (96.6 mg, 0.4 mmol), KI (66.4 mg, 0.4 mmol), $B(OH)_3$ (24.7 mg, 0.4 mmol) in CH_3CN/PhCN (2:1, v/v, 2.0 mL) afforded the desired product **2b** by flash column chromatography on silica gel (PE/EtOAc = 10:1, v/v) as a pale yellow oil (34.4 mg, 80%). IR (KBr, cm^{-1}): 1726, 1593, 1255, 1018, 802; ^1H NMR ($CDCl_3$, 500 MHz): δ 5.18 (dd, J = 11.5, 8.0 Hz, 1H), 4.35 (q, J = 7.0 Hz, 2H), 4.26 (q, J = 7.0 Hz, 2H), 3.54-3.44 (m, 2H), 1.36 (t, J = 7.0 Hz, 3H), 1.31 (t, J = 7.0 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 169.0, 160.0, 151.2, 80.0, 62.5, 62.4, 37.7, 14.2, 14.1; LC-MS (ESI) *m/z* 216 $[M+H]^+$.



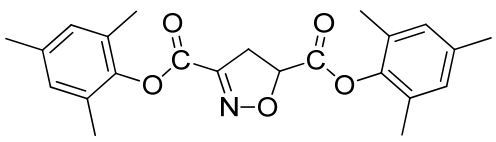
Dibenzyl 4,5-dihydroisoxazole-3,5-dicarboxylate (2c): Following the general procedure, the reaction of **1c** (64.9 mg, 0.4 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (96.6 mg, 0.4 mmol), KI (66.4 mg, 0.4 mmol), $B(OH)_3$ (24.7 mg, 0.4 mmol) in CH_3CN/PhCN (2:1, v/v, 2.0 mL) afforded the desired product **2c** by flash column chromatography on silica gel (PE/EtOAc = 10:1, v/v) as a pale yellow oil (52.9 mg, 78%). IR (KBr, cm^{-1}): 1734, 1597, 1257, 1124, 916, 745; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.43-7.33 (m, 10H), 5.32 (s, 2H), 5.26-5.19 (m, 3H), 3.55-3.43 (m, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 168.7, 159.8, 151.0, 134.8, 134.7, 128.9, 128.8, 128.7, 128.6, 80.0, 68.0, 67.9, 37.7; LC-MS (ESI) *m/z* 340 $[M+H]^+$; HRMS (ESI) *m/z* calcd for $C_{19}H_{18}NO_5$ $[M+H]^+$ 340.1179, found 340.1176.



Di-*tert*-butyl 4,5-dihydroisoxazole-3,5-dicarboxylate (2d): Following the general procedure, the reaction of **1d** (58 μL, 0.4 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (96.6 mg, 0.4 mmol), KI (66.4 mg, 0.4 mmol), $B(OH)_3$ (24.7 mg, 0.4 mmol) in CH_3CN/PhCN (2:1, v/v, 2.0 mL) afforded the desired product **2d** by flash column chromatography on silica gel (PE/EtOAc = 10:1, v/v) as a pale yellow oil (32.5 mg, 60%). IR (KBr, cm^{-1}): 2981, 2934, 1724, 1596, 1467, 1379, 1261, 1136, 920, 835; ^1H NMR ($CDCl_3$, 500 MHz): δ 5.03 (t, J = 10.0 Hz, 1H), 3.39 (d, J = 10.0 Hz, 2H), 1.54 (s, 9H), 1.48 (s, 9H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 168.2, 159.2, 152.1, 83.9, 83.3, 80.5, 37.8, 28.1, 28.0; LC-MS (ESI) *m/z* 272 $[M+H]^+$; HRMS (ESI) *m/z* calcd for $C_{13}H_{22}NO_5$ $[M+H]^+$ 272.1492, found 272.1492.

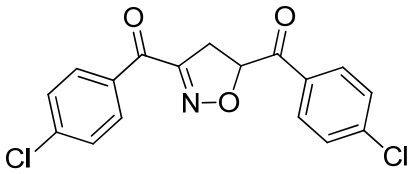


bis(1-Phenylethyl) 4,5-dihydroisoxazole-3,5-dicarboxylate (2e): Following the general procedure, the reaction of **1e** (70.4 mg, 0.4 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (96.6 mg, 0.4 mmol), KI (66.4 mg, 0.4 mmol), $B(OH)_3$ (24.7 mg, 0.4 mmol) in CH_3CN/PhCN (2:1, v/v, 2.0 mL) afforded the desired product **2e** by flash column chromatography on silica gel (PE/EtOAc = 10:1, v/v) as a pale yellow oil (48.5 mg, 66%). IR (KBr, cm^{-1}): 3032, 2982, 2931, 1728, 1596, 1254, 1061, 920, 756; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.45-7.27 (m, 10H), 6.08-5.99 (m, 1H), 5.99-5.91 (m, 1H), 5.24-5.12 (m, 1H), 3.52-3.37 (m, 2H), 1.65 (d, J = 6.5 Hz, 3H), 1.62-1.57 (m, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 168.2, 159.3, 151.2, 140.6, 128.8, 128.5, 128.4, 126.3, 126.2, 126.1, 80.1, 80.0, 74.9, 74.7, 74.6, 37.7, 37.6, 37.5, 22.3, 22.2, 22.1; LC-MS (ESI) *m/z* 368 $[M+H]^+$; HRMS (ESI) *m/z* calcd for $C_{21}H_{22}NO_5$ $[M+H]^+$ 368.1492, found 368.1490.

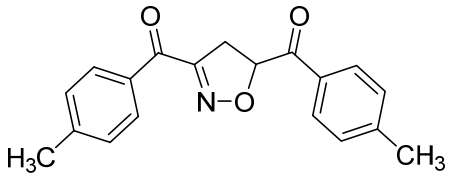


Dimesityl 4,5-dihydroisoxazole-3,5-dicarboxylate (2f): Following the general procedure, the reaction of **1f** (76.0 mg, 0.4 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (96.6 mg, 0.4 mmol), KI (66.4 mg, 0.4

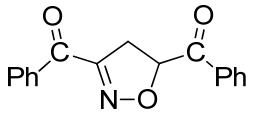
mmol), $B(OH)_3$ (24.7 mg, 0.4 mmol) in CH_3CN/PhCN (2:1, v/v, 2.0 mL) afforded the desired product **2f** by flash column chromatography on silica gel (PE/EtOAc = 10:1, v/v) as a pale yellow solid (37.9 mg, 48%). m.p.: 123-124 ^oC; IR (KBr, cm^{-1}): 1735, 1481, 1192, 1138, 913; ^1H NMR ($CDCl_3$, 500 MHz): δ 6.91 (s, 2H), 6.90 (s, 2H), 5.56 (dd, J = 12.0, 7.5 Hz, 1H), 3.86-3.70 (m, 2H), 2.29 (s, 3H), 2.28 (s, 3H), 2.15 (s, 6H), 2.14 (s, 6H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 166.9, 158.0, 150.7, 145.4, 145.3, 136.3, 136.2, 129.6, 126.5, 129.4, 80.4, 37.9, 20.9, 16.4 16.3; LC-MS (ESI) *m/z* 396 $[M+H]^+$; HRMS (ESI) *m/z* calcd for $C_{23}H_{26}NO_5$ $[M+H]^+$ 396.1805, found 396.1800.



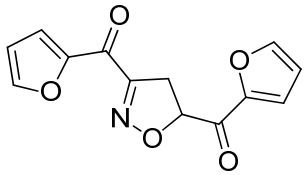
(**4,5-Dihydroisoxazole-3,5-diyl)bis((4-chlorophenyl)methanone) (2g):** Following the general procedure, the reaction of **1g** (66.6 mg, 0.4 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (96.6 mg, 0.4 mmol), KI (66.4 mg, 0.4 mmol), $B(OH)_3$ (24.7 mg, 0.4 mmol) in CH_3CN/PhCN (2:1, v/v, 2.0 mL) afforded the desired product **2g** by flash column chromatography on silica gel (PE/EtOAc = 10:1, v/v) as a pale yellow solid (41.3 mg, 60%). m.p.: 129-130 ^oC; IR (KBr, cm^{-1}): 3866, 3740, 3682, 2935, 1688, 1652, 1583, 1400, 1260, 1226, 1090, 920, 849, 736, 670, 583, 482; ^1H NMR ($CDCl_3$, 500 MHz): δ 8.16 (d, J = 8.5 Hz, 2H), 8.03 (d, J = 9.0 Hz, 2H), 7.52 (d, J = 8.5 Hz, 2H), 7.44 (d, J = 8.5 Hz, 2H), 5.91 (dd, J = 12.5, 7.5 Hz, 1H), 4.02 (dd, J = 18.0, 7.0 Hz, 1H), 3.60 (dd, J = 18.0, 12.0 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 191.2, 184.3, 157.7, 141.1, 140.7, 133.8, 132.6, 131.9, 131.1, 129.4, 129.0, 82.3, 35.7; LC-MS (ESI) *m/z* 365 (100) $[M+NH_4$ (^{35}Cl, ^{35}Cl)$]^+$, 367 (68) $[M+NH_4$ (^{35}Cl, ^{37}Cl)$]^+$, 369 (9) $[M+NH_4$ (^{37}Cl, ^{37}Cl)$]^+$; HRMS (ESI) *m/z* calcd for $C_{17}H_{15}Cl_2N_2O_3$ $[M+NH_4]^+$ 365.0454, found 365.0455.



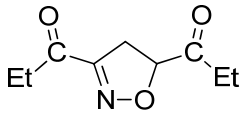
(4,5-Dihydroisoxazole-3,5-diyl)bis(*p*-tolylmethanone) (2h): Following the general procedure, the reaction of **1h** (58.4 mg, 0.4 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (96.6 mg, 0.4 mmol), KI (66.4 mg, 0.4 mmol), $B(OH)_3$ (24.7 mg, 0.4 mmol) in CH_3CN/PhCN (2:1, v/v, 2.0 mL) afforded the desired product **2h** by flash column chromatography on silica gel (PE/EtOAc = 10:1, v/v) as a pale yellow solid (40.9 mg, 67%). m.p.: 108-110 ^oC; IR (KBr, cm^{-1}): 3741, 3435, 3038, 2963, 1688, 1644, 1600, 1419, 1362, 1264, 1226, 1151, 1033, 977, 921, 877, 831, 739, 673, 590, 473; ^1H NMR ($CDCl_3$, 500 MHz): δ 8.11 (d, J = 8.0 Hz, 2H), 7.97 (d, J = 8.0 Hz, 2H), 7.32 (d, J = 8.0 Hz, 2H), 7.25 (d, J = 8.5 Hz, 2H), 5.92 (dd, J = 12.0, 7.5 Hz, 1H), 3.95 (dd, J = 18.0, 7.5 Hz, 1H), 3.61 (dd, J = 18.0, 12.0 Hz, 1H), 2.44 (s, 3H), 2.41 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 192.1, 185.3, 157.6, 145.4, 145.0, 133.1, 131.8, 130.6, 129.7, 129.6, 129.3, 82.1, 36.3, 21.9, 21.8; EI-MS *m/z* (%): 307 (1) $[M^+]$, 219 (12), 119 (100), 91 (95); HRMS (EI) *m/z* calcd for $C_{19}H_{17}NO_3$ M^+ 307.1208, found 307.1212.



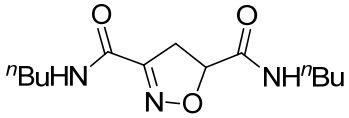
(4,5-Dihydroisoxazole-3,5-diyl)bis(phenylmethanone) (2i):[9] Following the general procedure, the reaction of **1i** (52.8 mg, 0.4 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (96.6 mg, 0.4 mmol), KI (66.4 mg, 0.4 mmol), $B(OH)_3$ (24.7 mg, 0.4 mmol) in CH_3CN/PhCN (2:1, v/v, 2.0 mL) afforded the desired product **2i** by flash column chromatography on silica gel (PE/EtOAc = 10:1, v/v) as a pale yellow solid (34.0 mg, 61%). m.p.: 78-80 ^oC; IR (KBr, cm^{-1}): 1693, 1655, 1587, 1362, 1258, 907, 696; ^1H NMR ($CDCl_3$, 500 MHz): δ 8.26-8.15 (m, 2H), 8.13-8.03 (m, 2H), 7.69-7.63 (m, 1H), 7.63-7.57 (m, 1H), 7.57-7.52 (m, 2H), 7.50-7.42 (m, 2H), 5.97 (dd, J = 12.0, 7.5 Hz, 1H), 4.00 (dd, J = 17.5, 7.5 Hz, 1H), 3.64 (dd, J = 18.0, 12.0 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 192.5, 185.8, 157.6, 135.7, 134.4, 134.3, 134.0, 130.5, 129.6, 129.0, 128.6, 82.3, 36.1; LC-MS (ESI) *m/z* 280 $[M+H]^+$.



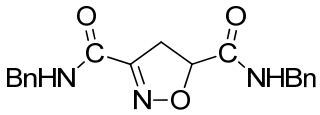
(4,5-Dihydroisoxazole-3,5-diyl)bis(furan-2-ylmethanone) (2j): Following the general procedure, the reaction of **1j** (48.8 mg, 0.4 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (96.6 mg, 0.4 mmol), KI (66.4 mg, 0.4 mmol), $B(OH)_3$ (24.7 mg, 0.4 mmol) in CH_3CN/PhCN (2:1, v/v, 2.0 mL) afforded the desired product **2j** by flash column chromatography on silica gel (PE/EtOAc = 10:1, v/v) as a pale yellow solid (37.6 mg, 73%). m.p.: 131-133 ^oC; IR (KBr, cm^{-1}): 3853, 3741, 3435, 3038, 2963, 1688,1644, 1600, 1419, 1362, 1264, 1226, 1151, 1033, 921, 877, 739, 673, 473 ; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.80-7.66 (m, 3H), 7.48 (d, J = 4.0 Hz, 1H), 6.63 (dd, J = 4.0, 2.0 Hz, 1H), 6.58 (dd, J = 3.5, 2.0 Hz, 1H), 5.74 (dd, J = 12.0, 7.5 Hz, 1H), 3.84 (dd, J = 18.0, 7.5 Hz, 1H), 3.62 (dd, J = 18.0, 7.0 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 181.9, 172.0, 156.7, 150.5, 150.2, 148.7, 148.2, 123.4, 121.0, 113.0, 112.8, 82.3, 36.1; EI-MS m/z (%): 259 (1) $[M^+]$, 121 (14), 95 (100); HRMS (EI) m/z calcd for $C_{13}H_9NO_5$ M^+ 259.0481, found 259.0484.



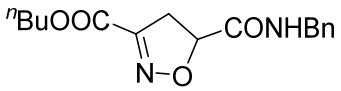
1,1'-(4,5-Dihydroisoxazole-3,5-diyl)bis(propan-1-one) (2k): Following the general procedure, the reaction of **1k** (41 μL, 0.4 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (96.6 mg, 0.4 mmol), KI (66.4 mg, 0.4 mmol), $B(OH)_3$ (24.7 mg, 0.4 mmol) in CH_3CN/PhCN (2:1, v/v, 2.0 mL) afforded the desired product **2k** by flash column chromatography on silica gel (PE/EtOAc = 10:1, v/v) as a pale yellow solid (17.2 mg, 47%). m.p.: 22-24 ^oC; IR (KBr, cm^{-1}): 1725, 1694, 1584, 1222, 912, 867; ^1H NMR ($CDCl_3$, 500 MHz): δ 5.08 (dd, J = 12.5, 7.5 Hz, 1H), 3.42 (dd, J = 18.0, 7.5 Hz, 1H), 3.28 (dd, J = 18.0, 12.5 Hz, 1H), 2.96-2.86 (m, 2H), 2.73-2.63 (m, 2H), 1.14 (t, J = 7.5 Hz, 3H), 1.09 (t, J = 7.5 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 207.5, 195.4, 157.4, 85.9, 35.0, 32.9, 32.5, 7.8, 7.2; LC-MS (ESI) *m/z* 184 $[M+H]^+$; HRMS (ESI) *m/z* calcd for $C_9H_{14}NO_3$ $[M+H]^+$ 184.0968, found 184.0969.



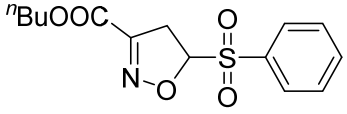
***N*^{3},*N*^{5}-Dibutyl-4,5-dihydroisoxazole-3,5-dicarboxamide (2l):** Following the general procedure, the reaction of **1l** (50.8 mg, 0.4 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (96.6 mg, 0.4 mmol), KI (66.4 mg, 0.4 mmol), $B(OH)_3$ (24.7 mg, 0.4 mmol) in CH_3CN (2.0 mL) afforded the desired product **2l** by flash column chromatography on silica gel (PE/EtOAc = 2:1, v/v) as a pale yellow solid (20.2 mg, 38%). m.p.: 168-170 ^oC; IR (KBr, cm^{-1}): 3739, 3262, 2958, 2867, 1652, 1542, 1431, 1368, 1146, 883, 730; ^1H NMR ($CDCl_3$, 500 MHz): δ 6.54 (s, 2H), 5.10 (dd, J = 10.5, 8.0 Hz, 1H), 3.61-3.49 (m, 2H), 3.37-3.18 (m, 4H), 1.58-1.45 (m, 4H), 1.42-1.29 (m, 4H), 0.98-0.86 (m, 6H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 169.6, 158.6.1, 154.9, 80.6, 39.4, 39.2, 38.7, 31.6, 31.5, 20.1, 13.8 ; LC-MS (ESI) *m/z* 287 $[M+NH_4]^+$; HRMS (ESI) *m/z* calcd for $C_{13}H_{27}N_4O_3$ $[M+NH_4]^+$ 287.2078, found 287.2077.



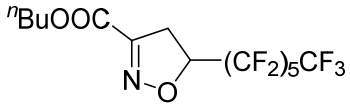
***N*^{3},*N*^{5}-Dibenzyl-4,5-dihydroisoxazole-3,5-dicarboxamide (2m):**[10] Following the general procedure, the reaction of **1m** (64.4 mg, 0.4 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (96.6 mg, 0.4 mmol), KI (66.4 mg, 0.4 mmol), $B(OH)_3$ (24.7 mg, 0.4 mmol) in CH_3CN/PhCN (2:1, v/v, 2.0 mL) afforded the desired product **2m** by flash column chromatography on silica gel (PE/EtOAc = 2:1, v/v) as a pale yellow solid (22.9 mg, 34%). m.p.: 152-154 ^oC; IR (KBr, cm^{-1}): 3329, 3257, 1653, 1535, 1428, 134, 698; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.53-7.16 (m, 10 H), 6.87 (br, 2H), 5.16 (dd, J = 11.5, 7.0 Hz, 1H), 4.59-4.47 (m, 3H), 4.44-4.34 (m, 1H), 3.69-3.54 (m, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 169.5, 158.6, 154.7, 137.4, 137.3, 129.0, 128.0 (2C) 127.9 (2C), 80.7, 43.7, 43.5, 38.6; LC-MS (ESI) *m/z* 338 $[M+H]^+$.



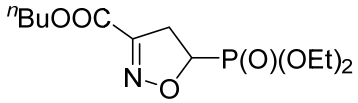
Butyl 5-(benzylcarbamoyl)-4,5-dihydroisoxazole-3-carboxylate (2o): Following the general procedure, the reaction of butyl acrylate **1a** (43 μL, 0.3 mmol), *N*-benzylacrylamide **1o** (193.4 mg, 1.2 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (290.0 mg, 1.2 mmol), KI (49.8 mg, 0.3 mmol), $B(OH)_3$ (18.5 mg, 0.3 mmol) in CH_3CN/PhCN (2:1, v/v, 3.0 mL) afforded the desired product **2o** by flash column chromatography on silica gel (PE/EtOAc = 4:1, v/v) as a pale yellow oil (44.0 mg, 48%). IR (KBr, cm^{-1}): 3740, 3319, 2958, 1743, 1671, 1532, 1204, 909, 739, 699; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.38-7.27 (m, 5H), 6.96 (s, 1H), 5.15 (dd, J = 11.0, 8.5 Hz, 1H), 4.52 (d, J = 6.0 Hz, 2H), 4.20 (t, J = 6.5 Hz, 2H), 3.60-3.48 (m, 2H), 1.69-1.61 (m, 2H), 1.43-1.33 (m, 2H), 0.93 (t, J = 7.5 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 169.2, 158.6, 153.3, 137.2, 128.8, 127.9, 127.8, 79.6, 66.0, 43.6, 37.8, 30.4, 19.0, 13.7; LC-MS (ESI) *m/z* 322 $[M+NH_4]^+$; HRMS (ESI) *m/z* calcd for $C_{16}H_{21}N_2O_4$ $[M+H]^+$ 305.1496, found 305.1496.



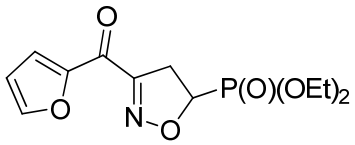
Butyl 5-(phenylsulfonyl)-4,5-dihydroisoxazole-3-carboxylate (2p): Following the general procedure, the reaction of butyl acrylate **1a** (43 μL, 0.3 mmol), (vinylsulfonyl)benzene **1p** (201.6 mg, 1.2 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (290.0 mg, 1.2 mmol), KI (49.8 mg, 0.3 mmol), $B(OH)_3$ (18.5 mg, 0.3 mmol) in CH_3CN/PhCN (2:1, v/v, 3.0 mL) afforded the desired product **2p** by flash column chromatography on silica gel (PE/EtOAc = 10:1, v/v) as a pale yellow solid (46.2 mg, 50%). m.p.: 85-87 ^oC; IR (KBr, cm^{-1}): 3852, 3740, 3682, 2965, 2871, 1734, 1608, 1461, 1353, 1317, 1279, 1156, 1080, 953, 870, 773, 734, 686, 568; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.98 (d, J = 7.5 Hz, 2H), 7.73 (t, J = 7.5 Hz, 1H), 7.61 (t, J = 7.5 Hz, 2H), 5.55 (dd, J = 11.5, 5.0 Hz, 1H), 4.28 (t, J = 7.0 Hz, 2H), 3.94 (dd, J = 19.5, 5.5 Hz, 1H), 3.65 (dd, J = 19.5, 11.5 Hz, 1H), 1.76-1.63 (m, 2H), 1.47-1.34 (m, 2H), 0.95 (t, J = 7.5 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 158.9, 152.0, 135.0, 134.8, 129.9, 129.5, 94.1, 66.6, 35.8, 30.4, 19.0, 13.7; LC-MS (ESI) *m/z* 329 $[M+NH_4]^+$; HRMS (ESI) *m/z* calcd for $C_{14}H_{21}SN_2O_5$ $[M+NH_4]^+$ 329.1166, found 329.1165.



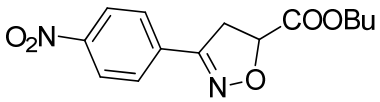
Butyl 5-(perfluorohexyl)-4,5-dihydroisoxazole-3-carboxylate (2q): Following the general procedure, the reaction of butyl acrylate **1a** (43 μL, 0.3 mmol), 3,3,4,4,5,5,6,6,7,7,8,8,8-tridecafluorooct-1-ene **1q** (415.3 mg, 1.2 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (290.0 mg, 1.2 mmol), KI (49.8 mg, 0.3 mmol), $B(OH)_3$ (18.5 mg, 0.3 mmol) in CH_3CN/PhCN (2:1, v/v, 3.0 mL) afforded the desired product **2q** by flash column chromatography on silica gel (PE/EtOAc = 10:1, v/v) as a white solid (58.0 mg, 40%). m.p.: 32-34 ^oC; IR (KBr, cm^{-1}): 3740, 2966, 2878, 1733, 1462, 1354, 1242, 1202, 1142, 912, 740, 703, 650; ^1H NMR ($CDCl_3$, 500 MHz): δ 5.32-5.16 (m, 1H), 4.29 (t, J = 7.0 Hz, 2H), 3.57-3.47 (m, 2H), 1.77-1.64 (m, 2H), 1.46-1.36 (m, 2H), 0.93 (t, J = 7.5 Hz, 3H); ^{19}F NMR ($CDCl_3$, 470 MHz): δ -80.9 (t, $J_{F\text{-}F}$ = 9.9 Hz, 2F), -121.9 (m, 4F), -122.3 (m, 2F), -122.9 (m, 2F), -126.3 (m, 2F); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 159.6, 151.5, 120.7-108.4 (m, $(CF_2)_6F$), 78.5 (dd, $J_{C\text{-}F}$ = 28.7 Hz, $J_{C\text{-}F}$ = 22.5 Hz), 66.7, 35.0, 30.5, 19.2, 13.7; LC-MS (ESI) *m/z* 507 $[M+NH_4]^+$; HRMS (ESI) *m/z* calcd for $C_{14}H_{16}F_{13}N_2O_3$ $[M+NH_4]^+$ 507.0948, found 507.0939.



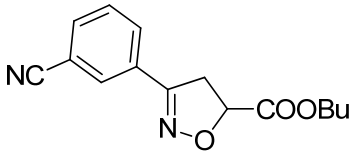
Butyl 5-(diethoxyphosphoryl)-4,5-dihydroisoxazole-3-carboxylate (2r): Following the general procedure, the reaction of butyl acrylate **1a** (43 μL, 0.3 mmol), diethyl vinylphosphonate **1r** (196.9 mg, 1.2 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (290.0 mg, 1.2 mmol), KI (49.8 mg, 0.3 mmol), $B(OH)_3$ (18.5 mg, 0.3 mmol) in CH_3CN (3.0 mL) afforded the desired product **2r** by flash column chromatography on silica gel (PE/EtOAc = 5:1, v/v) as a pale yellow oil (45.7 mg, 50%). IR (KBr, cm^{-1}): 3465, 2966, 2874, 1723, 1599, 1451, 1399, 1256, 1127, 1022, 971, 912, 786, 746, 546; ^1H NMR ($CDCl_3$, 500 MHz): δ 4.90 (t, J = 11.5 Hz, 1H), 4.29 (t, J = 6.5 Hz, 2H), 4.27-4.16 (m, 4H), 3.59-3.42 (m, 2H), 1.76-1.66 (m, 2H), 1.46-1.38 (m, 2H), 1.35 (td, J = 7.0, 2.5 Hz, 6H), 0.94 (t, J = 7.5 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 159.9, 151.4 (d, $^3J_{P\text{-}C}$ = 6.3 Hz), 77.3 (d, $^1J_{P\text{-}C}$ = 168.6 Hz), 66.2, 63.7 (d, $^2J_{P\text{-}C}$ = 7.4 Hz), 63.4 (d, $^2J_{P\text{-}C}$ = 6.4 Hz), 36.4, 30.4, 19.0, 16.5 (d, $^3J_{P\text{-}C}$ = 5.9 Hz), 13.6; LC-MS (ESI) *m/z* 325 $[M+NH_4]^+$; HRMS (ESI) *m/z* calcd for $C_{12}H_{23}PNO_6$ $[M+H]^+$ 308.1258, found 308.1256.



Diethyl (3-(furan-2-carbonyl)-4,5-dihydroisoxazol-5-yl)phosphonate (2s): Following the general procedure, the reaction of 1-(furan-2-yl)prop-2-en-1-one **1j** (36.6 mg, 0.3 mmol), diethyl vinylphosphonate **1r** (196.9 mg, 1.2 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (290.0 mg, 1.2 mmol), KI (49.8 mg, 0.3 mmol), $B(OH)_3$ (18.5 mg, 0.3 mmol) in CH_3CN/PhCN (2:1, v/v, 3.0 mL) afforded the desired product **2s** by flash column chromatography on silica gel (PE/EtOAc = 5:1, v/v) as a pale yellow oil (54.6 mg, 60%). IR (KBr, cm^{-1}): 3141, 2986, 2934, 1740, 1646, 1557, 1462, 1398, 1250, 1158, 1022, 972, 915, 835, 781, 588, 544; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.80-7.67 (m, 2H), 6.58 (dd, J = 4.0, 2.0 Hz, 1H), 4.89 (t, J = 11.5 Hz, 1H), 4.29-4.18 (m, 4H), 3.68-3.58 (m, 2H), 1.39-1.33 (m, 6H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 171.8, 156.5 (d, $^3J_{P\text{-}C}$ = 6.4 Hz), 150.0, 148.6, 123.3, 112.7, 76.5 (d, $^1J_{P\text{-}C}$ = 169.5 Hz), 63.6 (d, $^2J_{P\text{-}C}$ = 7.1 Hz), 63.4 (d, $^2J_{P\text{-}C}$ = 6.5 Hz), 36.4, 16.5 (d, $^3J_{P\text{-}C}$ = 5.5 Hz); EI-MS *m/z* (%): 301 (1) [M^+], 221 (18), 112 (66), 95 (100); HRMS (EI) *m/z* calcd for $C_{12}H_{16}PNO_6$ [M^+] 301.0715, found 301.0714.

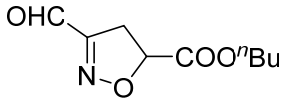


Butyl 3-(4-nitrophenyl)-4,5-dihydroisoxazole-5-carboxylate (2t): Following the general procedure, the reaction of butyl acrylate **1a** (43 μL, 0.3 mmol), 1-nitro-4-vinylbenzene **1t** (178.9 mg, 1.2 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (290.0 mg, 1.2 mmol), KI (49.8 mg, 0.3 mmol), $B(OH)_3$ (37.1 mg, 0.6 mmol) in CH_3CN/PhCN (2:1, v/v, 3.0 mL) afforded the desired product **2t** by flash column chromatography on silica gel (PE/EtOAc = 5:1, v/v) as a pale yellow solid (53.1 mg, 61%). m.p.: 120-122 ^{o}C; IR (KBr, cm^{-1}): 2958, 2927, 2861, 1743, 1602, 1575, 1520, 1342, 1207, 1166, 1109, 1017, 971, 898,848, 749, 691, 570, 534, 496, 430; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.26 (d, J = 9.0 Hz, 2H), 7.85 (d, J = 8.5 Hz, 2H), 5.25 (dd, J = 11.0, 7.5 Hz, 1H), 4.22 (t, J = 7.0 Hz, 2H), 3.75-3.57 (m, 2H), 1.71-1.64 (m, 2H), 1.44-1.34 (m, 2H), 0.93 (t, J = 7.5 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 169.6, 154.6, 148.7, 134.6, 127.7, 124.1, 79.0, 66.1, 38.2, 30.5, 19.0, 13.7; LC-MS (ESI) *m/z* 310 [$M+NH_4$]$^+$; HRMS (ESI) *m/z* calcd for $C_{14}H_{17}N_2O_5$ [$M+H$]$^+$ 293.1132, found 293.1130.

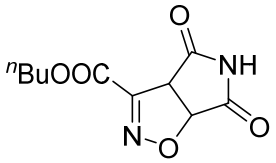


Butyl 3-(3-cyanophenyl)-4,5-dihydroisoxazole-5-carboxylate (2u): Following the general procedure, the reaction of butyl acrylate **1a** (43 μL, 0.3 mmol), 4-vinylbenzonitrile **1u** (154.9 mg, 1.2 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (290.0 mg, 1.2 mmol), KI (49.8 mg, 0.3 mmol), $B(OH)_3$ (37.1 mg, 0.6 mmol) in CH_3CN/PhCN (2:1, v/v, 3.0 mL) afforded the desired product **2u** by flash column chromatography on silica gel (PE/EtOAc = 5:1, v/v) as a pale yellow oil (39.6 mg, 49%). IR (KBr, cm^{-1}): 3489, 3073, 2961, 2872, 2232, 1742, 1601, 1437, 1352, 1206, 1064, 1017, 909, 804, 686, 503; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.94 (d, J = 8.0 Hz, 1H), 7.92 (s, 1H), 7.71 (d, J = 7.5 Hz, 1H), 7.54 (t, J = 8.0 Hz, 1H), 5.22 (dd, J = 11.5, 7.5 Hz, 1H), 4.21 (t, J = 7.0 Hz, 2H), 3.68-3.57 (m, 2H), 1.71-1.63 (m, 2H), 1.47-1.33 (m, 2H), 0.93 (t, J = 7.5 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125

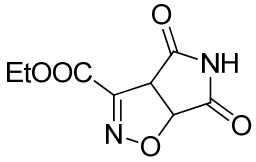
MHz): δ 169.7, 154.4, 133.6, 130.8, 130.3, 130.1, 129.8, 118.0, 113.3, 78.7, 66.1, 38.2, 30.5, 19.0, 13.7; LC-MS (ESI) *m/z* 290 $[M+NH_4]^+$; HRMS (ESI) *m/z* calcd for $C_{15}H_{17}N_2O_3$ $[M+H]^+$ 273.1234, found 273.1232.



Butyl 3-formyl-4,5-dihydroisoxazole-5-carboxylate (2v): Following the general procedure, the reaction of butyl acrylate **1a** (43 μL, 0.3 mmol), vinyl acetate **1v** (111 μL, 1.2 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (290.0 mg, 1.2 mmol), KI (49.8 mg, 0.3 mmol), $B(OH)_3$ (37.1 mg, 0.6 mmol) in CH_3CN/PhCN (2:1, v/v, 3.0 mL) at 70 ^oC afforded the desired product **2v** by flash column chromatography on silica gel (PE/EtOAc = 5:1, v/v) as a pale yellow oil (28.2 mg, 47%). IR (KBr, cm^{-1}): 2924, 2857, 2363, 1744, 1697, 1577,1461, 1361, 1211, 1021, 923, 802, 714; ^1H NMR ($CDCl_3$, 500 MHz): δ 9.93 (s, 1H), 5.22 (dd, *J* = 11.0, 8.5 Hz, 1H), 4.21 (t, *J* = 6.5 Hz, 2H), 3.44-3.33 (m, 2H), 1.70-1.62 (m, 2H), 1.43-1.34 (m, 2H), 0.94 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 184.7, 168.6, 158.3, 80.5, 66.2, 34.6, 30.4, 19.0, 13.6; LC-MS (ESI) *m/z* 217 $[M+NH_4]^+$; HRMS (ESI) *m/z* calcd for $C_9H_{14}NO_4$ $[M+H]^+$ 200.0917, found 200.0918.

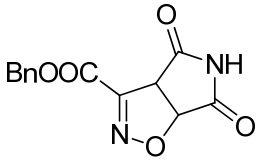


Butyl 4,6-dioxo-4,5,6,6a-tetrahydro-3a*H*-pyrrolo[3,4-*d*]isoxazole-3-carboxylate (4a): Following the general procedure, the reaction of butyl acrylate **1a** (43 μL, 0.3 mmol), 1*H*-pyrrole-2,5-dione **3a** (116.5 mg, 1.2 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (290.0 mg, 1.2 mmol), KI (49.8 mg, 0.3 mmol), $B(OH)_3$ (37.1 mg, 0.6 mmol) in CH_3CN/PhCN (2:1, v/v, 3.0 mL) afforded the desired product **4a** by flash column chromatography on silica gel (PE/EtOAc = 1:1, v/v) as a pale yellow oil (40.1 mg, 56%). IR (KBr, cm^{-1}): 3740, 3260, 2964, 1733, 1339, 1227, 1126, 930, 760, 614; ^1H NMR ($CDCl_3$, 500 MHz): δ 9.46 (s, 1H), 5.63 (d, *J* = 9.5 Hz, 1H), 4.79 (d, *J* = 9.5 Hz, 1H), 4.34-4.21 (m, 2H), 1.73-1.63 (m, 2H), 1.45-1.33 (m, 2H), 0.91 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 172.1, 170.6, 158.8, 147.8, 83.6, 67.0, 55.0, 30.3, 18.9, 13.6; LC-MS (ESI) *m/z* 258 $[M+NH_4]^+$; HRMS (ESI) *m/z* calcd for $C_{10}H_{13}N_2O_5$ $[M+H]^+$ 241.0819, found 241.0812.

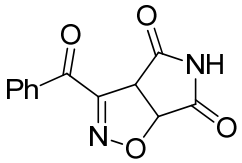


Ethyl 4,6-dioxo-4,5,6,6a-tetrahydro-3a*H*-pyrrolo[3,4-*d*]isoxazole-3-carboxylate (4b): Following the general procedure, the reaction of ethyl acrylate **1b** (32 μL, 0.3 mmol), 1*H*-pyrrole-2,5-dione **3a** (116.5 mg, 1.2 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (290.0 mg, 1.2 mmol), KI (49.8 mg, 0.3 mmol), $B(OH)_3$ (37.1 mg, 0.6 mmol) in CH_3CN/PhCN (2:1, v/v, 3.0 mL) afforded the desired product **4b** by flash column chromatography on silica gel (PE/EtOAc = 1:1, v/v) as a pale yellow oil (28.3 mg, 44%). IR (KBr, cm^{-1}): 3743, 3268, 3101, 2987, 2753, 1739, 1584, 1465, 1376, 1337, 1227, 1185, 1124, 1010, 930, 763, 607; ^1H NMR ($CDCl_3$, 500 MHz): δ 5.62 (d, *J* = 9.5 Hz, 1H), 4.79 (d, *J* = 10.0 Hz, 1H), 4.43-4.32 (m, 2H), 1.36 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 171.6, 170.2, 158.6, 147.7, 83.4, 63.3, 55.0, 14.0; LC-MS (ESI) *m/z* 230 $[M+NH_4]^+$;

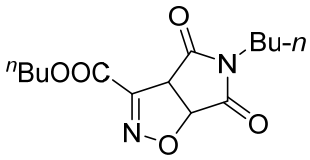
HRMS (ESI) *m/z* calcd for $C_8H_9N_2O_5$ $[M+H]^+$ 213.0506, found 213.0505.



Benzyl 4,6-dioxo-4,5,6,6a-tetrahydro-3a*H*-pyrrolo[3,4-*d*]isoxazole-3-carboxylate (4c): Following the general procedure, the reaction of benzyl acrylate **1c** (45 μL, 0.3 mmol), 1*H*-pyrrole-2,5-dione **3a** (116.5 mg, 1.2 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (290.0 mg, 1.2 mmol), KI (49.8 mg, 0.3 mmol), $B(OH)_3$ (37.1 mg, 0.6 mmol) in CH_3CN/PhCN (2:1, v/v, 3.0 mL) afforded the desired product **4c** by flash column chromatography on silica gel (PE/EtOAc = 1:1, v/v) as a pale yellow solid (41.4 mg, 50%). m.p.: 148-150 ^oC; IR (KBr, cm^{-1}): 3740, 3249, 3098, 2979, 1733, 1338, 1221, 1182, 1122, 930, 748, 702; ^1H NMR (d_6-DMSO, 500 MHz): δ 11.9 (s, 1H), 7.49-7.32 (m, 5H), 5.57 (d, J = 9.5 Hz, 1H), 5.39-5.25 (m, 2H), 4.74 (d, J = 9.5 Hz, 1H); ^{13}C NMR (d_6-DMSO, 125 MHz): δ 173.8, 172.2, 158.6, 149.0, 135.6, 128.9, 128.8, 128.6, 84.9, 67.7, 55.8; LC-MS (ESI) *m/z* 292 $[M+NH_4]^+$; HRMS (ESI) *m/z* calcd for $C_{13}H_{14}N_3O_5$ $[M+NH_4]^+$ 292.0928, found 292.0926.

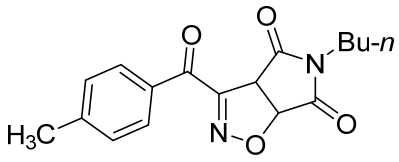


3-Benzoyl-3a*H*-pyrrolo[3,4-*d*]isoxazole-4,6(5*H*,6a*H*)-dione (4d):[9] Following the general procedure, the reaction of 1-phenylprop-2-en-1-one **1i** (39.6 mg, 0.3 mmol), 1*H*-pyrrole-2,5-dione **3a** (116.5 mg, 1.2 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (290.0 mg, 1.2 mmol), KI (49.8 mg, 0.3 mmol), $B(OH)_3$ (37.1 mg, 0.6 mmol) in CH_3CN/PhCN (2:1, v/v, 3.0 mL) afforded the desired product **4d** by flash column chromatography on silica gel (PE/EtOAc = 1:1, v/v) as a pale yellow solid (32.8 mg, 45%). m.p.: 163-165 ^oC; IR (KBr, cm^{-1}): 3518, 3283, 2969, 2923, 1815, 1732, 1645, 1592, 1558, 1449, 1337, 1184, 926, 879, 837, 704, 597; ^1H NMR (d_6-DMSO, 500 MHz): δ 11.92 (s, 1H), 8.04 (d, J = 7.5 Hz, 2H), 7.72 (t, J = 7.5 Hz, 1H), 7.56 (t, J = 8.0 Hz, 2H), 5.59 (d, J = 9.5 Hz, 1H), 4.97 (d, J = 9.5 Hz, 1H); ^{13}C NMR (d_6-DMSO, 125 MHz): δ 184.6, 173.9, 172.5, 153.9, 135.6, 134.8, 130.4, 129.1, 83.9, 56.7; LC-MS (ESI) *m/z* 262 $[M+NH_4]^+$.

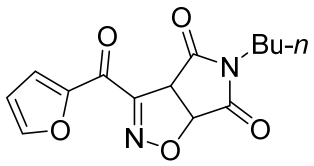


Butyl 5-butyl-4,6-dioxo-4,5,6,6a-tetrahydro-3a*H*-pyrrolo[3,4-*d*]isoxazole-3-carboxylate (4e): Following the general procedure, the reaction of butyl acrylate **1a** (43 μL, 0.3 mmol), 1-butyl-1*H*-pyrrole-2,5-dione **3e** (183.7 mg, 1.2 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (290.0 mg, 1.2 mmol), KI (49.8 mg, 0.3 mmol), $B(OH)_3$ (37.1 mg, 0.6 mmol) in CH_3CN/PhCN (2:1, v/v, 3.0 mL) afforded the desired product **4e** by flash column chromatography on silica gel (PE/EtOAc = 3:1, v/v) as a pale yellow solid (57.0 mg, 64%). m.p.: 67-70 ^oC; IR (KBr, cm^{-1}): 3744, 34823, 2962, 2871, 1714, 1580, 1460, 1406, 1349, 1230, 1196, 1147, 1018, 920, 842, 737, 635, 607, 409; ^1H NMR ($CDCl_3$, 500 MHz): δ 5.56 (d, J = 9.5 Hz, 1H), 4.71 (d, J = 9.5 Hz, 1H), 4.38-4.23 (m, 2H), 3.55-3.42 (m, 2H), 1.75-1.64 (m, 2H), 1.57-1.45 (m, 2H), 1.45-1.33 (m, 2H), 1.30-1.18 (m, 2H),

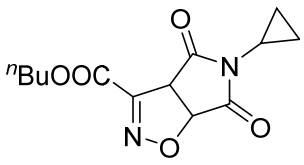
0.92 (t, J = 7.5 Hz, 3H), 0.87 (t, J = 7.5 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.8, 169.5, 158.5, 147.7, 82.2, 66.8, 53.8, 39.6, 30.3, 29.3, 19.9, 18.9, 13.6, 13.5; EI-MS *m/z* (%): 296 (1) [M^+], 185 (29), 135 (100), 124 (85), 56 (80); HRMS (EI) *m/z* calcd for $C_{14}H_{20}N_2O_5$ [M^+] 296.1372, found 296.1366.



5-Butyl-3-(4-methylbenzoyl)-3a*H*-pyrrolo[3,4-*d*]isoxazole-4,6(5*H*,6a*H*)-dione (4f): Following the general procedure, the reaction of 1-(*p*-tolyl)prop-2-en-1-one **1h** (43.8 mg, 0.3 mmol), 1-butyl-1*H*-pyrrole-2,5-dione **3e** (183.7 mg, 1.2 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (290.0 mg, 1.2 mmol), KI (49.8 mg, 0.3 mmol), $B(OH)_3$ (37.1 mg, 0.6 mmol) in CH_3CN/PhCN (2:1, v/v, 3.0 mL) afforded the desired product **4f** by flash column chromatography on silica gel (PE/EtOAc = 3:1, v/v) as a pale yellow solid (58.5 mg, 63%). m.p.: 88-90 ^{o}C; IR (KBr, cm^{-1}): 3739, 3295, 2971, 1731, 1643, 1558, 1346, 1185, 924, 882, 833, 726, 596; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.06 (d, J = 8.5 Hz, 2H), 7.27 (d, J = 8.5 Hz, 2H), 5.54 (d, J = 9.5 Hz, 1H), 5.06 (d, J = 10.0 Hz, 1H), 3.58-3.48 (m, 2H), 2.42 (s, 3H), 1.59-1.47 (m, 2H), 1.31-1.21 (m, 2H), 0.88 (t, J = 7.5 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 182.9, 171.0, 169.8, 153.0, 145.8, 132.3, 130.5, 129.4, 81.0, 54.9, 39.5, 29.4, 21.8, 19.9, 13.5; LC-MS (ESI) *m/z* 315 [M+H]$^+$; HRMS (ESI) *m/z* calcd for $C_{17}H_{19}N_2O_4$ [M+H]$^+$ 315.1339, found 315.1336.

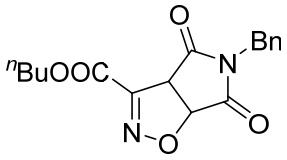


5-Butyl-3-(furan-2-carbonyl)-3a*H*-pyrrolo[3,4-*d*]isoxazole-4,6(5*H*,6a*H*)-dione (4g): Following the general procedure, the reaction of 1-(furan-2-yl)prop-2-en-1-one **1j** (36.6 mg, 0.3 mmol), 1-butyl-1*H*-pyrrole-2,5-dione **3e** (183.7 mg, 1.2 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (290.0 mg, 1.2 mmol), KI (49.8 mg, 0.3 mmol), $B(OH)_3$ (37.1 mg, 0.6 mmol) in CH_3CN/PhCN (2:1, v/v, 3.0 mL) afforded the desired product **4g** by flash column chromatography on silica gel (PE/EtOAc = 1:1, v/v) as a pale yellow solid (50.1 mg, 58%). m.p.: 108-109 ^{o}C; IR (KBr, cm^{-1}): 3860, 3744, 3145, 2960, 2871, 2357, 1792, 1716, 1648, 1461, 1396, 1348, 1254, 1187, 1137, 1080, 1026, 925, 834, 771, 632, 590; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.76 (d, J = 1.5 Hz, 1H), 7.67 (d, J = 3.5 Hz, 1H), 6.60 (dd, J = 3.5, 2.0 Hz, 1H), 5.53 (d, J = 9.5 Hz, 1H), 4.99 (d, J = 9.5 Hz, 1H), 3.60-3.47 (m, 2H), 1.58-1.51 (m, 2H), 1.30-1.23 (m, 2H), 0.89 (t, J = 7.5 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.6, 169.7, 169.3, 152.3, 149.9, 149.1, 123.6, 112.9, 81.3, 54.0, 39.6, 29.3, 19.9, 13.5; EI-MS *m/z* (%): 290 (1) [M^+], 178 (18), 136 (93), 95 (100); HRMS (EI) *m/z* calcd for $C_{14}H_{14}N_2O_5$ [M^+] 290.0903, found 290.0904.

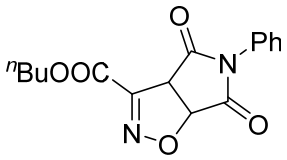


Butyl 5-cyclopropyl-4,6-dioxo-4,5,6,6a-tetrahydro-3a*H*-pyrrolo[3,4-*d*]isoxazole-3-carboxylate

(4h): Following the general procedure, the reaction of butyl acrylate **1a** (43 μL, 0.3 mmol), 1-cyclopropyl-1*H*-pyrrole-2,5-dione **3h** (164.4 mg, 1.2 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (290.0 mg, 1.2 mmol), KI (49.8 mg, 0.3 mmol), $B(OH)_3$ (37.1 mg, 0.6 mmol) in CH_3CN/PhCN (2:1, v/v, 3.0 mL) afforded the desired product **4h** by flash column chromatography on silica gel (PE/EtOAc = 3:1, v/v) as a pale yellow solid (47.0 mg, 56%). m.p.: 76-78 ^oC; IR (KBr, cm^{-1}): 3845, 3741, 3679, 3619, 3499, 3415, 2965, 2874, 2615, 1797, 1724, 1576, 1462, 1403, 1351, 1224, 1125, 1030, 983, 928, 831, 798, 744, 666, 632; ^1H NMR ($CDCl_3$, 500 MHz): δ 5.50 (d, J = 9.5 Hz, 1H), 4.66 (d, J = 10.0 Hz, 1H), 4.38-4.26 (m, 2H), 2.69-2.57 (m, 1H), 1.75-1.67 (m, 2H), 1.46-1.37 (m, 2H), 1.00-0.85 (m, 7H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 171.0, 169.8, 158.4, 147.8, 81.8, 66.8, 53.4, 30.3, 23.1, 19.0, 13.6, 4.9, 4.8; EI-MS *m/z* (%): 280 (8) [M^+], 136 (52), 56 (100); HRMS (EI) *m/z* calcd for $C_{13}H_{16}N_2O_5$ [M^+] 280.1059, found 280.1057.



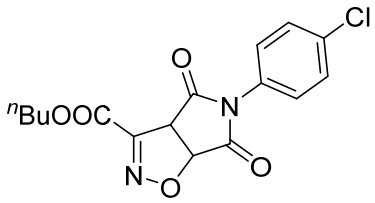
Butyl 5-benzyl-4,6-dioxo-4,5,6,6a-tetrahydro-3a*H*-pyrrolo[3,4-*d*]isoxazole-3-carboxylate (4i): Following the general procedure, the reaction of butyl acrylate **1a** (43 μL, 0.3 mmol), 1-benzyl-1*H*-pyrrole-2,5-dione **3i** (224.5 mg, 1.2 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (290.0 mg, 1.2 mmol), KI (49.8 mg, 0.3 mmol), $B(OH)_3$ (37.1 mg, 0.6 mmol) in CH_3CN/PhCN (2:1, v/v, 3.0 mL) afforded the desired product **4i** by flash column chromatography on silica gel (PE/EtOAc = 3:1, v/v) as a pale yellow solid (57.3 mg, 58%). m.p.: 110-112 ^oC; IR (KBr, cm^{-1}): 2963, 2876, 1717, 1581, 1495, 1461, 1398, 1349, 1313, 1221, 1171, 1124, 919, 825, 745, 701, 665, 626, 582, 525, 459; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.34-7.26 (m, 5H), 5.51 (d, J = 10.0 Hz, 1H), 4.66 (d, J = 9.5 Hz, 1H), 4.62 (s, 2H), 4.37-4.25 (m, 2H), 1.76-1.66 (m, 2H), 1.47-1.35 (m, 2H), 0.95 (t, J = 7.5 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.4, 169.2, 158.4, 147.6, 134.4, 129.0, 128.9, 128.5, 82.2, 66.8, 53.9, 43.3, 30.3, 19.0, 13.6; EI-MS *m/z* (%): 330 (1) [M^+], 212 (97), 91 (100); HRMS (EI) *m/z* calcd for $C_{17}H_{18}N_2O_5$ [M^+] 330.1216, found 330.1221.



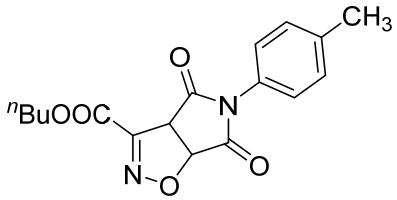
Butyl 4,6-dioxo-5-phenyl-4,5,6,6a-tetrahydro-3a*H*-pyrrolo[3,4-*d*]isoxazole-3-carboxylate (4j): Following the general procedure, the reaction of butyl acrylate **1a** (43 μL, 0.3 mmol), 1-phenyl-1*H*-pyrrole-2,5-dione **3j** (207.6 mg, 1.2 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (290.0 mg, 1.2 mmol), KI (49.8 mg, 0.3 mmol), $B(OH)_3$ (37.1 mg, 0.6 mmol) in CH_3CN/PhCN (2:1, v/v, 3.0 mL) afforded the desired product **4j** by flash column chromatography on silica gel (PE/EtOAc = 3:1, v/v) as a pale yellow solid (55.4 mg, 58%). m.p.: 96-97 ^oC; IR (KBr, cm^{-1}): 3740, 2965, 1731, 1501, 1382, 1212, 1120, 926, 735, 685; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.49-7.34 (m, 3H), 7.20 (d, J = 7.0 Hz, 2H), 5.68 (d, J = 10.0 Hz, 1H), 4.87 (d, J = 10.0 Hz, 1H), 4.39-4.21 (m, 2H), 1.78-1.64 (m, 2H), 1.48-1.33 (m, 2H), 0.93 (t, J = 7.5 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.0, 168.7, 158.6, 147.8, 130.7, 129.4, 129.3, 126.3, 82.4, 66.9, 53.9, 30.3, 19.0, 13.6; EI-MS *m/z* (%): 316 (6) [M^+], 198 (38), 119 (100); HRMS (EI) *m/z* calcd for $C_{16}H_{16}N_2O_5$ [M^+] 316.1059, found 316.1065.

Gram-scale reaction

To a 100-mL round bottom flask were added butyl acrylate **1a** (1.0 g, 7.8 mmol), 1-phenyl-1*H*-pyrrole-2,5-dione **3j** (5.4 g, 31.2 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (7.54 g, 31.2 mmol), KI (1.29 g, 7.8 mmol), $B(OH)_3$ (0.96 g, 15.6 mmol) and CH_3CN/PhCN (2:1, v/v, 78 mL). The reaction was stirred at 80 ^oC under air as monitored by TLC. Upon completion, the reaction mixture was cooled down to room temperature and the solvent was removed under reduced pressure. After filtration to remove insoluble solid, the mixture was washed with ammonium hydroxide and extracted with ethyl acetate (3×10 mL). The combined organic phase was washed with water and brine, and dried over anhydrous Na_2SO_4. After filtration through a thin pad of celite, the filtrate was evaporated under reduced pressure to give the crude product, which was further purified by flash column chromatography on silica gel (PE/EtOAc = 3:1, v/v) to give **4j** as a pale yellow solid (1.03 g, 42%).

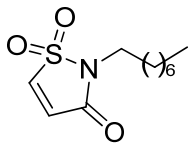


Butyl-5-(4-chlorophenyl)-4,6-dioxo-4,5,6,6a-tetrahydro-3a*H*-pyrrolo[3,4-*d*]isoxazole-3-carbo xylate (4k): Following the general procedure, the reaction of butyl acrylate **1a** (43 μL, 0.3 mmol), 1-(4-chlorophenyl)-1*H*-pyrrole-2,5-dione **3k** (248.4 mg, 1.2 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (290.0 mg, 1.2 mmol), KI (49.8 mg, 0.3 mmol), $B(OH)_3$ (37.1 mg, 0.6 mmol) in CH_3CN/PhCN (2:1, v/v, 3.0 mL) afforded the desired product **4k** by flash column chromatography on silica gel (PE/EtOAc = 3:1, v/v) as a pale yellow solid (45.3 mg, 43%). m.p.: 147-149 ^oC; IR (KBr, cm^{-1}): 3506, 2964, 2873, 1731, 1585, 1495, 1461, 1383, 1225, 1191, 1122, 1092, 1013, 924, 828, 791, 737, 705, 639, 506; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.44 (d, J = 9.0 Hz, 2H), 7.23 (d, J = 9.0 Hz, 2H), 5.71 (d, J = 10.0 Hz, 1H), 4.89 (d, J = 10.0 Hz, 1H), 4.42-4.27 (m, 2H), 1.78-1.68 (m, 2H), 1.49-1.38 (m, 2H), 0.95 (t, J = 7.5 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 169.4, 168.1, 158.4, 147.7, 135.3, 129.6, 129.0, 127.4, 82.1, 67.0, 53.8, 30.3, 19.0, 13.6; EI-MS *m/z* (%): 352 (1) [M^+ (^{37}Cl)], 350 (5) [M^+ (^{35}Cl)], 234 (16), 232 (66), 155 (10), 153 (100); HRMS (EI) *m/z* calcd for $C_{16}H_{15}ClN_2O_5$ [M^+] 350.0669, found 350.0678.

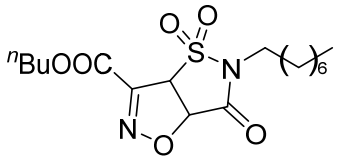


Butyl 4,6-dioxo-5-(*p*-tolyl)-4,5,6,6a-tetrahydro-3a*H*-pyrrolo[3,4-*d*]isoxazole-3-carboxylate (4l): Following the general procedure, the reaction of butyl acrylate **1a** (43 μL, 0.3 mmol), 1-(*p*-tolyl)-1*H*-pyrrole-2,5-dione **3l** (224.5 mg, 1.2 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (290.0 mg, 1.2 mmol), KI (49.8 mg, 0.3 mmol), $B(OH)_3$ (37.1 mg, 0.6 mmol) in CH_3CN/PhCN (2:1, v/v, 3.0 mL) afforded the desired product **4l** by flash column chromatography on silica gel (PE/EtOAc = 3:1, v/v) as a pale yellow solid (49.6 mg, 50%). m.p.: 115-118 ^oC; IR (KBr, cm^{-1}): 3495, 3413, 3004, 2965, 2869, 2360, 1791, 1724, 1580, 1515, 1459, 1390, 1226, 1197, 1121, 1057, 935, 816, 780, 741, 630, 499, 430; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.25 (d, J = 8.5 Hz, 2H), 7.10 (d, J = 8.5 Hz,

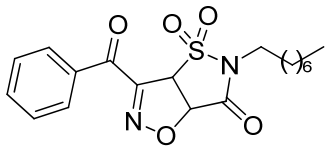
2H), 5.67 (d, J = 10.0 Hz, 1H), 4.85 (d, J = 10.0 Hz, 1H), 4.39-4.26 (m, 2H), 2.37 (s, 3H), 1.77-1.67 (m, 2H), 1.48-1.37 (m, 2H), 0.94 (t, J = 7.5 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.0, 168.7, 158.5, 147.9, 139.6, 130.0, 128.0, 126.0, 82.3, 66.9, 53.8, 30.3, 21.2, 19.0, 13.6; EI-MS *m/z* (%): 330 (7) [M^+], 212 (100), 133 (76); HRMS (EI) *m/z* calcd for $C_{17}H_{18}N_2O_5$ [M^+] 330.1216, found 330.1212.



Preparation of 2-octylisothiazol-3(2*H*)-one 1,1-dioxide (3m):[11] To a flask were added 2-octyl-4-isothiazolin-3-one (640.0 mg, 3.0 mmol) and DCM (30.0 mL), then *m*-chloroperoxybenzoic acid (1.55 g, 7.6 mmol) was added slowly at room temperature. After add up, the reaction was stirred for another 18 h. Upon completion, the reaction mixture was quenched with saturated $Na_2S_2O_3$ aqueous solution and extracted with DCM (3 × 10 mL). The combined organic phase was washed with saturated $NaHCO_3$ aqueous solution and brine, and dried over Na_2SO_4. After filtration through a thin pad of celite, the filtrate was evaporated under reduced pressure to give the crude product which was purified by column chromatography on silica gel (PE/EtOAc = 5:1, v/v) to give **3m** as a white solid (675.2 mg, 92%). m.p.: 22-24 ^{o}C; IR (KBr, cm^{-1}): 3448, 3170, 3091, 2927, 2857, 1738, 1588, 1461, 1340, 1165, 1116, 1029, 979, 931, 852, 793, 724, 672, 615, 571, 537, 404; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.39 (d, J = 7.0 Hz, 1H), 6.77 (d, J = 7.5 Hz, 1H), 3.63 (t, J = 7.5 Hz, 2H), 1.81-1.66 (m, 2H), 1.41-1.15 (m, 10H), 0.86 (t, J = 7.0 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 159.2, 138.4, 129.3, 39.8, 31.7, 29.1, 28.9, 28.1, 26.7, 22.6, 14.1; LC-MS (ESI) *m/z* 246 [M+H]$^+$.

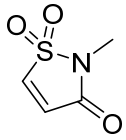


Butyl 5-octyl-6-oxo-3a,5,6,6a-tetrahydroisothiazolo[5,4-*d*]isoxazole-3-carboxylate 4,4-dioxide (4m): Following the general procedure, the reaction of butyl acrylate **1a** (43 μL, 0.3 mmol), **3m** (294.1 mg, 1.2 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (290.0 mg, 1.2 mmol), KI (49.8 mg, 0.3 mmol), $B(OH)_3$ (37.1 mg, 0.6 mmol) in CH_3CN/PhCN (2:1, v/v, 3.0 mL) afforded the desired product **4m** by flash column chromatography on silica gel (PE/EtOAc = 3:1, v/v) as a pale yellow oil (48.1 mg, 41%). IR (KBr, cm^{-1}): 2928, 2862, 1739, 1589, 1461, 1406, 1353, 1221, 1160, 1123, 1070, 983, 930, 553; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 5.81 (d, J = 11.0 Hz, 1H), 5.47 (d, J = 10.5 Hz, 1H), 4.49-4.27 (m, 2H), 3.71-3.54 (m, 2H), 1.80-1.65 (m, 4H), 1.48-1.38 (m, 2H), 1.36-1.17 (m, 10H), 0.95 (t, J = 7.5 Hz, 3H), 0.86 (t, J = 7.0 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 159.2, 157.9, 146.1, 84.1, 68.1, 67.4, 41.2, 31.7, 30.3, 29.0, 28.9, 27.9, 26.6, 22.6, 18.9, 14.1, 13.6; LC-MS (ESI) *m/z* 406 [M^+NH_4]; HRMS (ESI) *m/z* calcd for $C_{17}H_{32}SN_3O_6$ [$M+NH_4$]$^+$ 406.2006, found 406.2002.

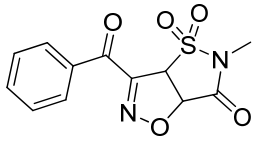


3-Benzoyl-5-octyl-5,6a-dihydroisothiazolo[5,4-*d*]isoxazol-6(3a*H*)-one 4,4-dioxide (4n):

Following the general procedure, the reaction of 1-phenylprop-2-en-1-one **1i** (39.6 mg, 0.3 mmol), **3m** (294.1 mg, 1.2 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (290.0 mg, 1.2 mmol), KI (49.8 mg, 0.3 mmol), $B(OH)_3$ (37.1 mg, 0.6 mmol) in CH_3CN/PhCN (2:1, v/v, 3.0 mL) afforded the desired product **4n** by flash column chromatography on silica gel (PE/EtOAc = 3:1, v/v) as a pale yellow solid (37.9 mg, 32%). m.p.: 80-81^oC; IR (KBr, cm^{-1}): 2927, 2859, 1742, 1715, 1658, 1574, 1452, 1354, 1307, 1228, 1156, 1079, 979, 934, 862, 712, 686, 664; ^1H NMR ($CDCl_3$, 500 MHz): δ 8.26 (dd, J = 8.0, 1.3 Hz, 2H), 7.70-7.65 (m, 1H), 7.52 (t, J = 8.0 Hz, 2H), 5.75 (dd, J = 11.0, 10.5 Hz, 2H), 3.70-3.56 (m, 2H), 1.79-1.69 (m, 2H), 1.36-1.24 (m, 10H), 0.87 (t, J = 7.0 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 183.1, 159.5, 152.0, 134.9, 134.1, 130.6, 128.8, 82.9, 69.0, 41.2, 31.6, 29.0, 28.9, 27.9, 26.6, 22.6, 14.1; LC-MS (ESI) *m/z* 415 $[M+Na]^+$; HRMS (ESI) *m/z* calcd for $C_{19}H_{24}N_2SNaO_5$ $[M+Na]^+$ 415.1298, found 415.1302.

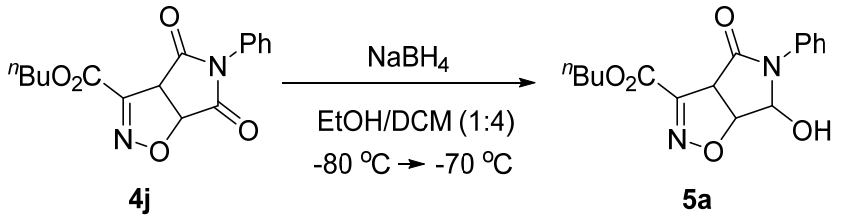


Preparation of 2-methylisothiazol-3(2*H*)-one 1,1-dioxide (3o): To a flask were added 2-methyl-4-isothiazolin-3-one (345.5 mg, 3.0 mmol) and DCM (30.0 mL), then *m*-chloroperoxy-benzoic acid (1.52 g, 7.5 mmol) was added slowly at 30 ^oC. After add up, the reaction was stirred for another 17.5 h. Upon completion, the reaction mixture was quenched with saturated $Na_2S_2O_3$ aqueous solution and extracted with DCM (3 × 10 mL). The combined organic phase was washed with saturated $NaHCO_3$ aqueous solution and brine, and dried over Na_2SO_4. After filtration through a thin pad of celite, the filtrate was evaporated under reduced pressure to give the crude product, which was further purified by column chromatography on silica gel (PE/EtOAc = 3:1, v/v) to give **3o** as a white solid (375.6 mg, 85%). m.p.: 111-113 ^oC; IR (KBr, cm^{-1}): 3450, 3173, 3095, 2925, 2856, 1737, 1584, 1435, 1331, 1298, 1213, 1161, 1033, 915, 855, 785, 668, 614, 565, 512, 483, 407; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.42 (d, J = 7.0 Hz, 1H), 6.81 (d, J = 7.5 Hz, 1H), 3.15 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 158.9, 138.4, 129.4, 23.5; LC-MS (ESI) *m/z* 148 $[M+H]^+$; HRMS (ESI) *m/z* calcd for $C_4H_6NO_3S$ $[M+H]^+$ 148.0063, found 148.0062.



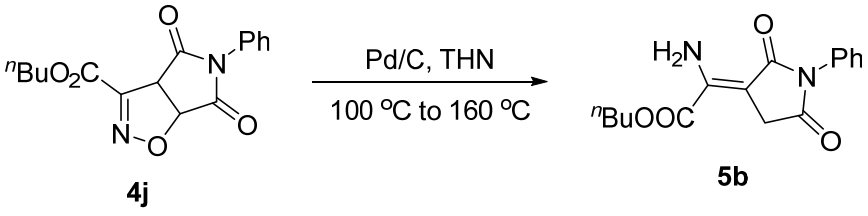
3-Benzoyl-5-methyl-5,6a-dihydroisothiazolo[5,4-*d*]isoxazol-6(3a*H*)-one 4,4-dioxide (4o): Following the general procedure, the reaction of 1-phenylprop-2-en-1-one **1i** (39.6 mg, 0.3 mmol), **3o** (176.4 mg, 1.2 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (290.0 mg, 1.2 mmol), KI (49.8 mg, 0.3 mmol), $B(OH)_3$ (37.1 mg, 0.6 mmol) in CH_3CN/PhCN (2:1, v/v, 3.0 mL) afforded the desired product **4o** by flash column chromatography on silica gel (PE/EtOAc = 2:1, v/v) as a pale yellow solid (20.5 mg, 23%). m.p.: 201-203 ^oC; IR (KBr, cm^{-1}): 3438, 2965, 1730, 1661, 1585, 1344, 1236, 1155, 1076, 922, 847, 716, 682, 636, 551; ^1H NMR (d_6-DMSO, 500 MHz): δ 8.13 (d, J = 7.0 Hz, 2H), 7.77 (t, J = 7.5 Hz, 1H), 7.61 (t, J = 7.5 Hz, 2H), 6.04 (dd, J = 11.5, 11.0 Hz, 2H), 3.04 (s, 3H); ^{13}C NMR (d_6-DMSO, 125 MHz): δ 183.9, 160.5, 153.4, 135.2, 134.7, 130.4, 129.4, 85.2, 70.0, 24.9; LC-MS (ESI) *m/z* 312 $[M+NH_4]^+$; HRMS (ESI) *m/z* calcd for $C_{12}H_{11}N_2O_5S$ $[M+H]^+$ 295.0383, found 295.0380.

3. Further Transformation of Products



4j **5a**

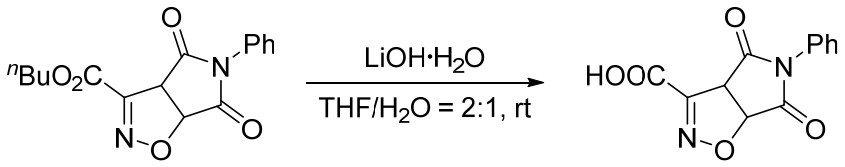
Butyl-6-hydroxy-4-oxo-5-phenyl-4,5,6,6a-tetrahydro-3a*H*-pyrrolo[3,4-*d*]isoxazole-3-carboxylate (5a): To a flask was added **4j** (63.2 mg, 0.2 mmol) and DCM/EtOH (4:1, v/v, 5.0 mL). Then to the mixture was added sodium borohydride (9.1 mg, 0.24 mmol) dissolved in EtOH (1 mL) dropwise at -80 °C during 0.5 h. The mixture was strictly controlled between -80 °C and -70 °C for 1.5 h. Upon completion, the reaction mixture was warmed to room temperature, quenched with saturated NH_4Cl aqueous solution (5 mL) and extracted with DCM (3 × 10 mL). The combined organic phase was washed with brine and dried over Na_2SO_4. After filtration through a thin pad of celite, the filtrate was evaporated under reduced pressure to give the crude product, which was further purified by column chromatography on silica gel (PE/EtOAc = 3:1, v/v) to give **5a** as a pale yellow solid (61.2 mg, 96%). m.p.: 73-75 °C; IR (KBr, cm^{-1}): 3548, 3432, 2965, 2872, 1724, 1691, 1590, 1499, 1463, 1410, 1356, 1303, 1223, 1119, 997, 925, 849, 791, 760, 692, 643, 562; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.44-7.22 (m, 5H), 5.59-5.44 (m, 1H), 5.19 (d, J = 9.0 Hz, 1H), 5.06 (d, J = 9.5 Hz, 1H), 4.74 (d, J = 9.0 Hz, 1H), 4.30 (t, J = 7.0 Hz, 2H), 1.78-1.63 (m, 2H), 1.49-1.35 (m, 2H), 0.94 (t, J = 7.5 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 167.6, 159.3, 149.2, 135.6, 129.3, 127.6, 124.8, 88.7, 88.6, 66.6, 54.3, 30.4, 19.0, 13.7; LC-MS (ESI) *m/z* 319 $[M+H]^+$; HRMS (ESI) *m/z* calcd for $C_{16}H_{19}N_2O_5$ $[M+H]^+$ 319.1288, found 319.1287.



4j **5b**

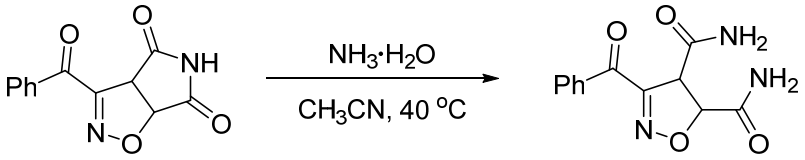
(*Z*)-Butyl 2-amino-2-(2,5-dioxo-1-phenylpyrrolidin-3-ylidene)acetate (5b): To a test tube were added **4j** (63.2 mg, 0.2 mmol), Pd-C catalyst (Pd: 10%, 42.4 mg, 0.04 mmol) and tetrahydronaphthalene (THN, 2 mL). The mixture was stirred at 100 °C for 2 h. Then the reaction temperature was raised to 160 °C for 3 h. Upon completion, the reaction mixture was cooled down to room temperature, extracted with EA (3 × 10 mL). The combined organic phase was washed with brine and dried over Na_2SO_4. After filtration through a thin pad of celite, the filtrate was evaporated under reduced pressure to give the crude product, which was further purified by column chromatography on silica gel (PE/EtOAc = 5:1, v/v) to give **5b** as a white solid (45.8 mg, 76%). m.p.: 96-97 °C; IR (KBr, cm^{-1}): 3425, 3310, 3062, 2958, 2864, 1729, 1685, 1628, 1555, 1500, 1457, 1399, 1279, 1187, 1113, 1063, 912, 761, 699, 669, 619, 504; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.91 (brs, 1H), 7.52-7.44 (m, 2H), 7.42-7.30 (m, 3H), 5.67 (brs, 1H), 4.34 (t, J = 7.0 Hz, 2H), 3.71 (s, 2H), 1.81-1.68 (m, 2H), 1.52-1.37 (m, 2H), 0.98 (t, J = 7.5 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 174.2, 172.3, 163.2, 140.5, 132.0, 129.1, 128.3, 126.5, 95.2, 66.9, 35.0, 30.5, 19.2, 13.7; LC-MS (ESI) *m/z* 303 $[M+H]^+$; HRMS (ESI) *m/z* calcd for $C_{16}H_{19}N_2O_4$ $[M+H]^+$

303.1339, found 303.1336.



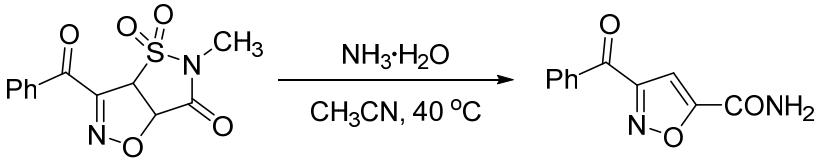
4j **5c**

4,6-Dioxo-5-phenyl-4,5,6,6a-tetrahydro-3a*H*-pyrrolo[3,4-*d*]isoxazole-3-carboxylic acid (5c):[12] To a test tube were added **4j** (63.2 mg, 0.2 mmol), lithium hydroxide monohydrate (25.2 mg, 0.6 mmol) and THF/H_2O (2:1, v/v, 2 mL). The mixture was stirred at room temperature for 12.5 h. Upon completion, the reaction mixture was acidified with dilute HCl. After removal of THF, the resulting residue was extracted with EA and washed with brine. The organic phase was dried over Na_2SO_4. After filtration through a thin pad of celite, the filtrate was evaporated under reduced pressure to give the crude product, which was further purified by column chromatography on silica gel (DCM/MeOH = 20:1, v/v) to give **5c** as a yellow solid (51.6 mg, 99%). m.p.: 115-117 ^oC; IR (KBr, cm^{-1}): 3332, 3064, 2926, 1718, 1600, 1544, 1495, 1444, 1387, 1317, 1195, 752, 691; ^1H NMR (d_6-DMSO, 500 MHz): δ 10.54 (s, 1H), 7.60 (d, J = 8.0 Hz, 2H), 7.33 (t, J = 8.0Hz, 2H), 7.10 (t, J = 7.5 Hz, 1H), 5.33 (d, J = 7.0 Hz, 1H), 4.66 (d, J = 7.0 Hz, 1H); ^{13}C NMR (d_6-DMSO, 125 MHz): δ 169.8, 166.3, 160.8, 151.2, 138.8, 129.4, 124.6, 119.9, 84.7, 57.9; LC-MS (ESI) *m/z* 279 $[M+H+H_2O]^+$.



4d **5d**

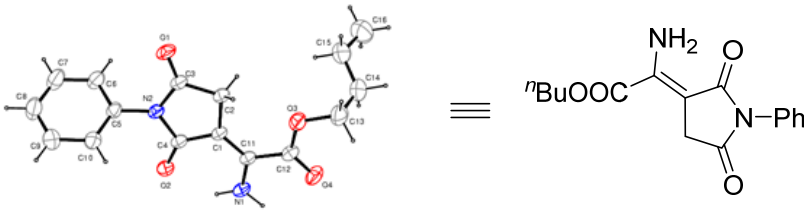
3-Benzoyl-4,5-dihydroisoxazole-4,5-dicarboxamide (5d) (31.2 mg, 60%): To a test tube were added **4d** (48.8 mg, 0.2 mmol), ammonium hydroxide (25-28%, 0.4 mL) and CH_3CN (2 mL). The reaction was stirred at 40 ^oC as monitored by TLC. Upon completion, the solvent was removed under reduced pressure, and the residue was washed with CH_3OH and EA to give **5d** as a white solid (31.2 mg, 60%). m.p.: 213-215 ^oC; IR (KBr, cm^{-1}): 3423, 3187, 1681, 1612, 1443, 1400, 1349, 1310, 1220, 1140, 1099, 942, 907, 873, 793, 689, 633, 493; ^1H NMR (d_6-DMSO, 500 MHz): δ 8.04 (d, J = 7.5 Hz, 2H), 7.95 (s, 1H), 7.91(s, 1H), 7.72 (t, J = 7.5 Hz, 1H), 7.64 (s, 1H), 7.57 (t, J = 7.5 Hz, 2H), 7.41 (s, 1H), 5.13 (d, J = 7.0 Hz, 1H), 4.62 (d, J = 7.0 Hz, 1H); ^{13}C NMR (d_6-DMSO, 125 MHz): δ 185.8, 169.8, 169.7, 156.4, 135.9, 134.5, 130.3, 129.1, 85.2, 57.5; LC-MS (ESI) *m/z* 260 $[M-H]^-$; HRMS (ESI) *m/z* calcd for $C_{12}H_{10}N_3O_4$ $[M-H]^-$ 260.0677, found 260.0685.



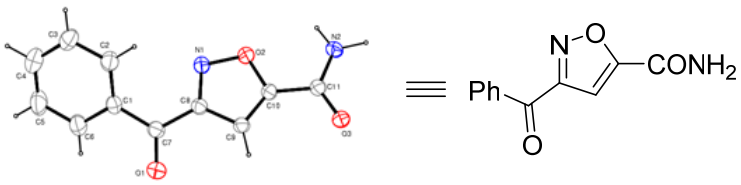
4o **5e**

3-Benzoylisoxazole-5-carboxamide (5e): To a test tube were added **4o** (58.8 mg, 0.2 mmol), ammonium hydroxide (25%-28%, 0.4 mL) and CH_3CN (2.0 mL). The reaction was stirred at 40 ^oC as monitored by TLC. Upon completion, the reaction mixture was cooled down to room temperature, diluted with EA and washed with brine. The aqueous phase was then extracted with EA (3×10 mL). The combined organic phase was dried over Na_2SO_4. After filtration through a thin pad of celite, the filtrate was evaporated under reduced pressure to give the crude product, which was purified by column chromatography on silica gel (PE/EtOAc = 1:1, v/v) to give **5e** as a white solid (31.4 mg, 73%). m.p.: 191-193 ^oC; IR (KBr, cm^{-1}): 3742, 3439, 3177, 3099, 2923, 1719, 1679, 1365, 1266, 1224, 1187, 942, 891, 775, 699, 545; ^1H NMR (d_6-DMSO, 500 MHz): δ 8.49 (s, 1H), 8.15 (d, J = 7.0 Hz, 2H), 8.12 (s, 1H), 7.77 (t, J = 7.5 Hz, 1H), 7.62 (t, J = 8.0 Hz, 2H), 7.51 (s, 1H); ^{13}C NMR (d_6-DMSO, 125 MHz): δ 185.5, 164.9, 162.3, 157.0, 135.6, 135.0, 130.7, 129.3, 107.1; LC-MS (ESI) m/z 234 $[M+NH_4]^+$; HRMS (ESI) m/z calcd for $C_{11}H_9N_2O_3$ $[M+H]^+$ 217.0608, found 217.0606.

4. X-Ray Crystallographic Analysis for Compounds 5b and 5e

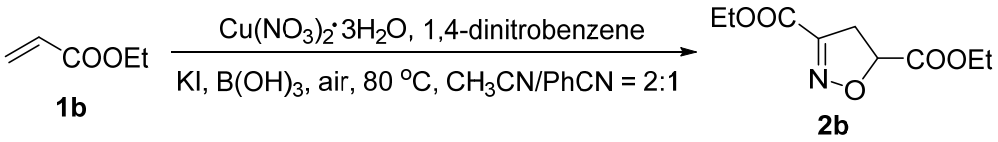


Crystallographic data for **5b**: $C_{16}H_{18}N_2O_4$, M = 302.32, triclinic, P-1 (No. 2), a = 7.033 (16) Å, b = 8.85 (4) Å, c = 13.91 (5) Å, α = 74.89 (2)^o, β = 80.27 (2)^o, γ = 73.83 (2)^o ,V = 799 (5) $Å^3$, Z = 2, Crystal size: 0.25 0.21 0.15 mm, T = 295 K, ρ_{calcd} = 1.257 g·cm^{-3}, R_1 = 0.0573 (I>4σ(I)), wR_2 = 0.1716 (all data), GOF = 1.031, reflections collected/unique: 4014 /2743 (Rint = 0.0225), Data: 1878, restraints: 0, parameters: 208. CCDC 1895015 contains the supplementary crystallographic data for this paper. The data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

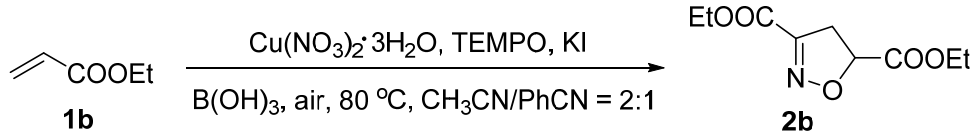


Crystallographic data for **5e**: $C_{11}H_8N_2O_3$, M = 216.19, Orthorhombic, Pbca (No. 61), a = 7.851 (13) Å, b = 7.073 (12) Å, c = 37.45 (6) Å, V = 2079 (6) $Å^3$, Z = 8, Crystal size: 0.27 0.15 0.13 mm, T = 295 K, ρ_{calcd} = 1.381 g·cm^{-3}, R_1 = 0.0741 (I>4σ(I)), wR_2 = 0.1992 (all data), GOF = 1.015, reflections collected/unique: 11624 / 2415 (Rint = 0.0872), Data: 1199, restraints: 0, parameters: 154. CCDC 1895016 contains the supplementary crystallographic data for this paper. The data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

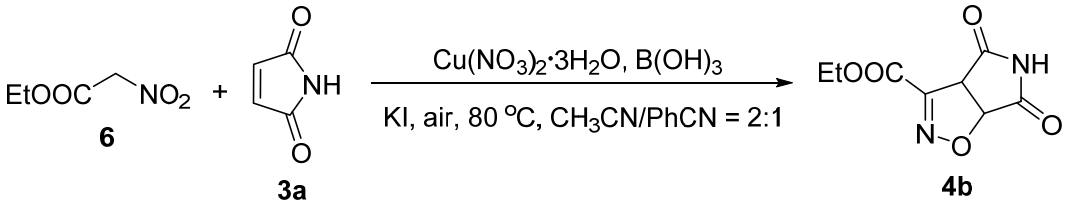
5. Mechanistic Studies



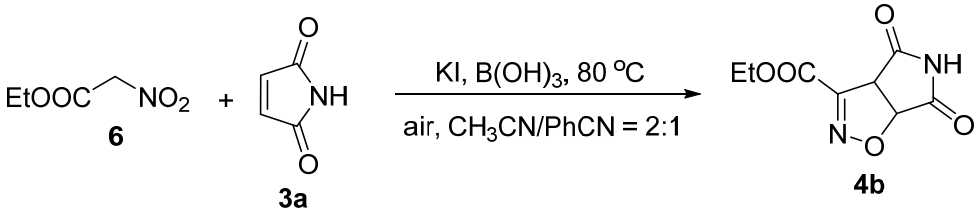
Following the general procedure, the reaction of **1b** (43 μL, 0.4 mmol), $Cu(NO_3)_2·3H_2O$ (96.6 mg, 0.4 mmol), KI (66.4 mg, 0.4 mmol), $B(OH)_3$ (24.7 mg, 0.4 mmol), 1,4-dinitrobenzene (134.5 mg, 0.8 mmol) in CH_3CN/PhCN (2:1, v/v, 2.0 mL) afforded the desired product **2b** by flash column chromatography on silica gel (PE/EtOAc = 10:1, v/v) as a pale yellow oil (36.5 mg, 85%).



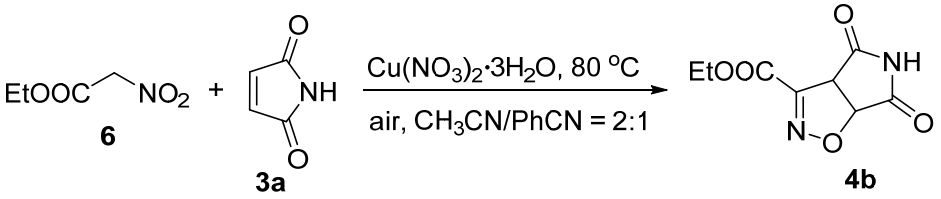
Following the general procedure, the reaction of **1b** (43 μL, 0.4 mmol), $Cu(NO_3)_2·3H_2O$ (96.6 mg, 0.4 mmol), KI (66.4 mg, 0.4 mmol), $B(OH)_3$ (24.7 mg, 0.4 mmol), TEMPO (125.0 mg, 0.8 mmol) in CH_3CN/PhCN (2:1, v/v, 2.0 mL) afforded the desired product **2b** by flash column chromatography on silica gel (PE/EtOAc = 10:1, v/v) as a pale yellow oil (24.6 mg, 57%).



Following the general procedure, the reaction of ethyl nitroacetate **6** (34.5 mg, 0.26 mmol), **3a** (77.7 mg, 0.8 mmol), $Cu(NO_3)_2·3H_2O$ (193.3 mg, 0.8 mmol), KI (33.2 mg, 0.2 mmol), $B(OH)_3$ (24.7 mg, 0.4 mmol) in CH_3CN/PhCN (2:1, v/v, 2.0 mL) afforded the desired product **4b** (52.4 mg, 95%) by flash column chromatography on silica gel (PE/EtOAc = 1:1, v/v).



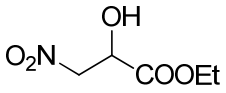
Following the general procedure, the reaction of ethyl nitroacetate **6** (26.2 mg, 0.2 mmol), **3a** (77.7 mg, 0.8 mmol), KI (33.2 mg, 0.2 mmol), $B(OH)_3$ (24.7 mg, 0.4 mmol) in CH_3CN/PhCN (2:1, v/v, 2.0 mL) afforded no product.



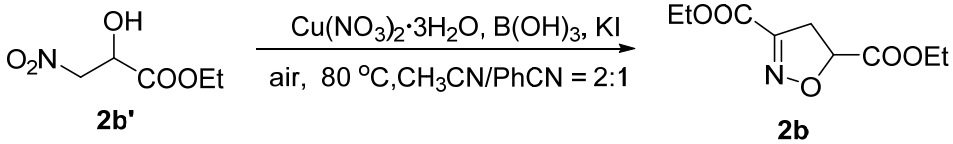
Following the general procedure, the reaction of ethyl nitroacetate **6** (34.6 mg, 0.26 mmol), **3a**

(77.7 mg, 0.8 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (14.5 mg, 0.06 mmol) in CH_3CN/PhCN (2:1, v/v, 2.0 mL) afforded the desired product **4b** (38.0 mg, 90%) by flash column chromatography on silica gel (PE/EtOAc = 1:1, v/v).

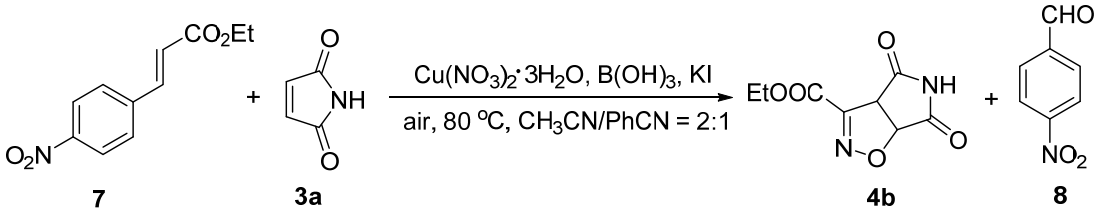
Following the general procedure, the reaction of **1b** (43 μL, 0.4 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (96.6 mg, 0.4 mmol), $B(OH)_3$ (24.7 mg, 0.4 mmol) in CH_3CN/PhCN (2:1, v/v, 2.0 mL) afforded the desired product **2b** as a pale yellow oil (15.2 mg, 35%) and the by-product **2b'** as a pale yellow oil (20.4 mg, 31%) by flash column chromatography on silica gel (PE/EtOAc = 10:1 to 5:1, v/v).



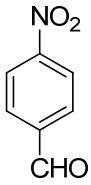
Ethyl 2-hydroxy-3-nitropropanoate (2b'):[13] IR (KBr, cm^{-1}): 3493, 2982, 2926, 1743, 1560, 1378, 1221, 1123, 1018, 908, 859, 683; ^1H NMR ($CDCl_3$, 500 MHz): δ 4.82-4.72 (m, 2H), 4.65-4.61 (m, 1H), 4.41-4.30 (m, 2H), 3.35 (d, J = 5.0 Hz, 1H), 1.33 (t, J = 7.5 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.7, 76.7, 67.5, 63.1, 14.0; LC-MS (ESI) *m/z* 186 $[M+Na]^+$.



Following the general procedure, the reaction of **2b'** (16.3 mg, 0.1 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (24.2 mg, 0.1 mmol), KI (16.6 mg, 0.1 mmol), $B(OH)_3$ (6.2 mg, 0.1 mmol in CH_3CN/PhCN (2:1, v/v, 1.0 mL) afforded no product.



To a test tube were added (*E*)-3-(4-nitrophenyl) acrylate (44.2 mg, 0.2 mmol), maleimide (77.7 mg, 0.8 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (193.3 mg, 0.8 mmol), KI (33.2 mg, 0.2 mmol), $B(OH)_3$ (24.7 mg, 0.4 mmol) and CH_3CN/PhCN (2:1, v/v, 2.0 mL). The mixture was stirred at 80 °C under air as monitored by TLC. Upon completion, the reaction mixture was cooled down to room temperature, quenched with ammonium hydroxide, and extracted with EA (3×10 mL). The combined organic phase was washed with water and brine, and dried over Na_2SO_4. After filtration through a thin pad of celite, the filtrate was evaporated under reduced pressure to give the crude product, which was purified by column chromatography on silica gel to give **4b** (16.6 mg, 39%) and *p*-nitrobenzaldehyde **8** (PE/EtOAc = 10:1, v/v) as a white solid (23.8 mg, 78%).



4-Nitrobenzaldehyde (8):[14] m.p.: 100-102 ^{o}C; IR (KBr, cm^{-1}): 3396, 3105, 2923, 2848, 1708, 1604, 1534, 1415, 1350, 1292, 1103, 1007, 851, 816, 736, 679, 512; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 10.15 (s, 1H), 8.38 (d, J = 9.0 Hz, 2H), 8.07 (d, J = 8.5 Hz, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 190.4, 151.1, 140.1, 130.5, 124.3. The obtained data were according with the literature reported results.

6. Reference

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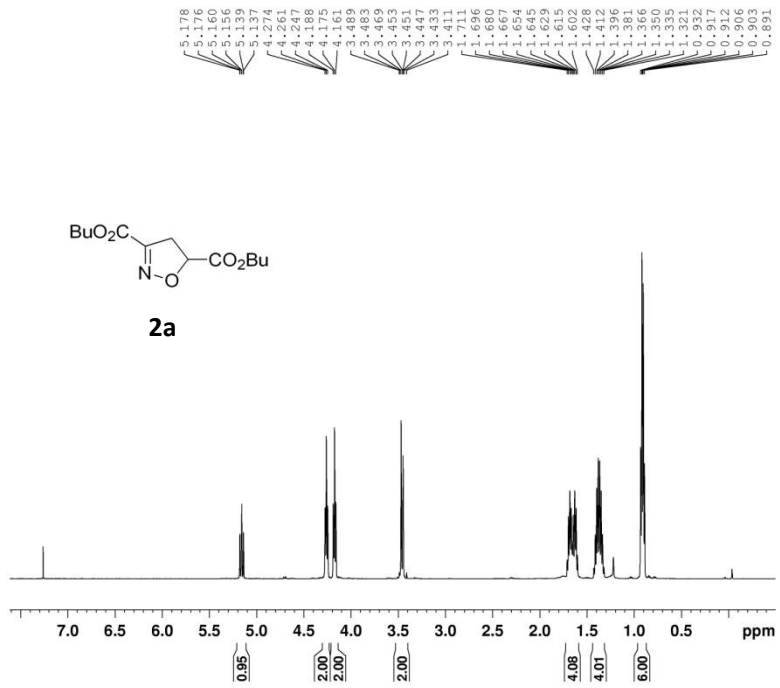

GYS-1-99
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TD0	1
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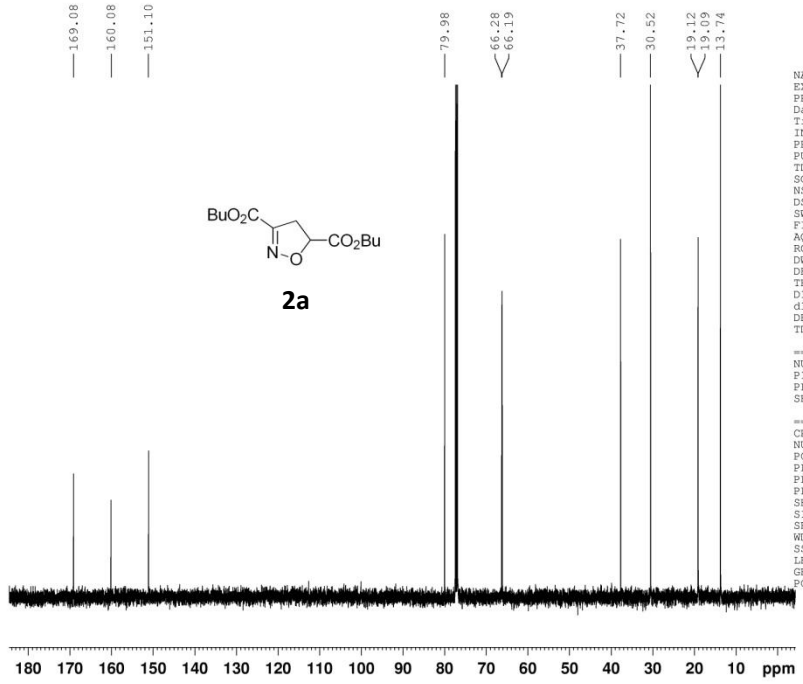

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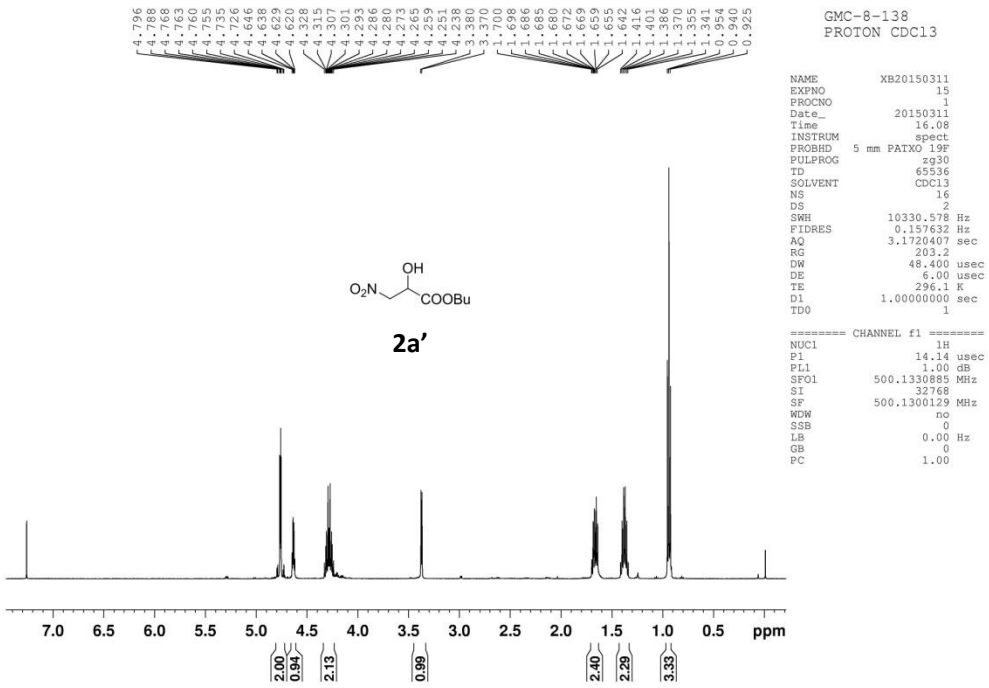

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AQ	3.1720407 sec
RG	203.2
DW	48.400 usec
DE	6.00 usec
TE	296.1 K
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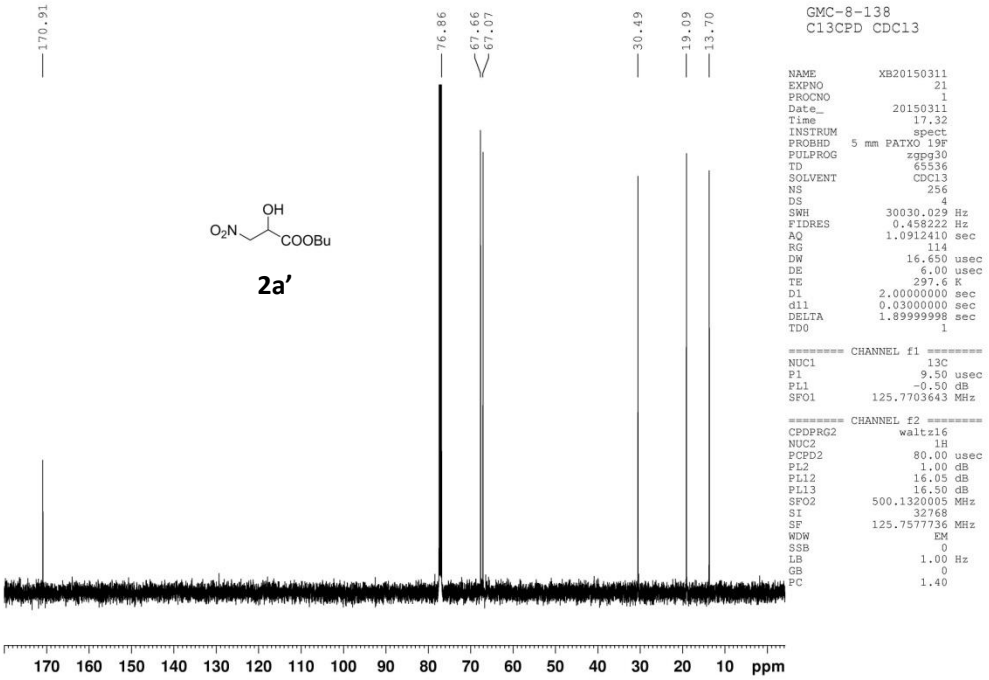

GMC-8-138
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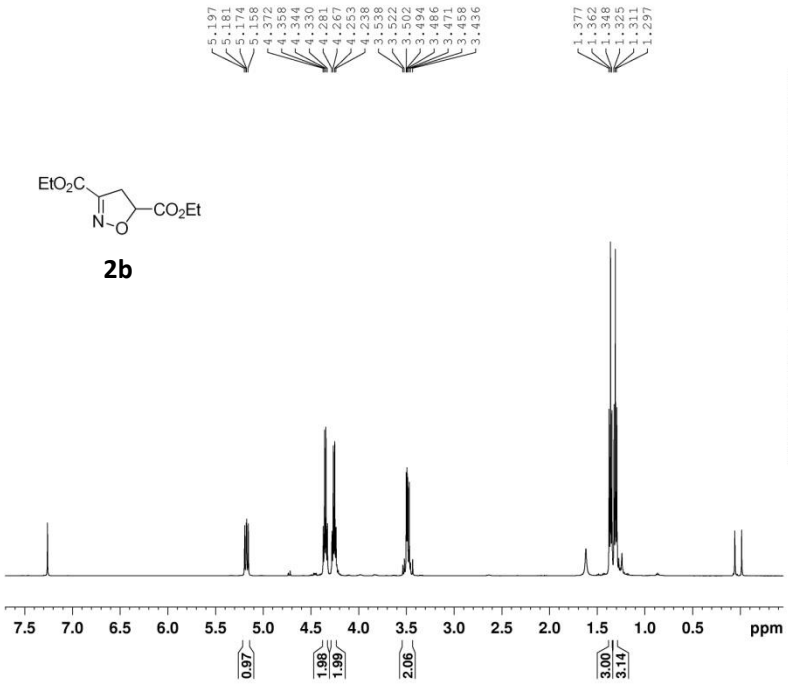

GMC-10-30-1
PROTON CDC13

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AQ	3.1720407 sec
RG	203.2
DW	48.400 usec
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TE	295.9 K
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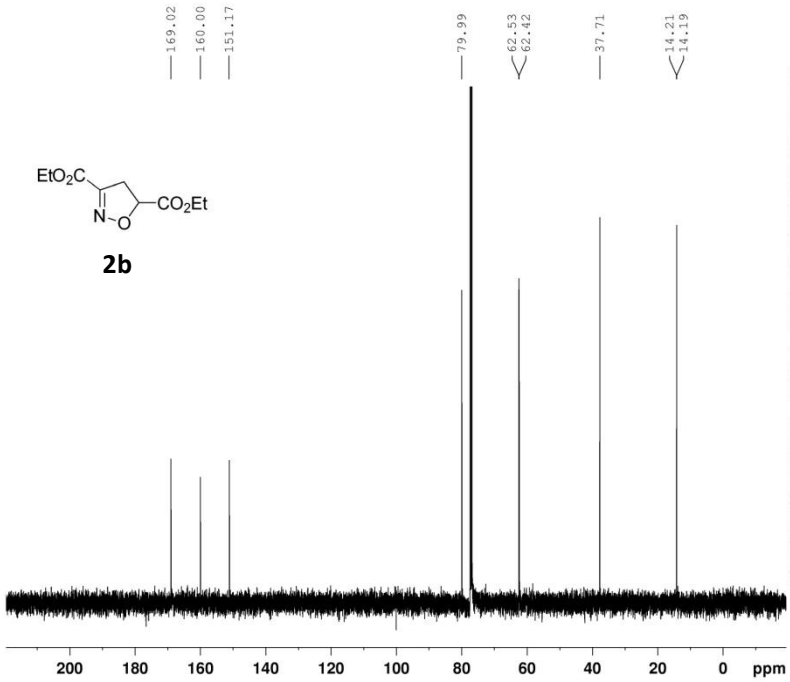

GMC-10-30-1
C13CPD CDC13

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TE	297.2 K
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SSB	0
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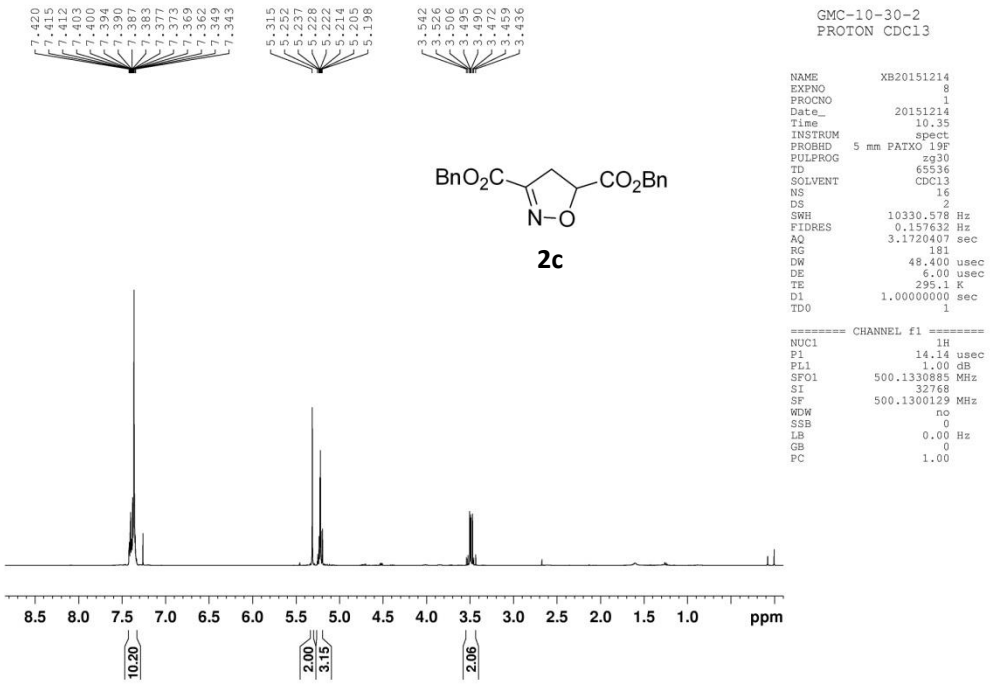

GMC-10-30-2
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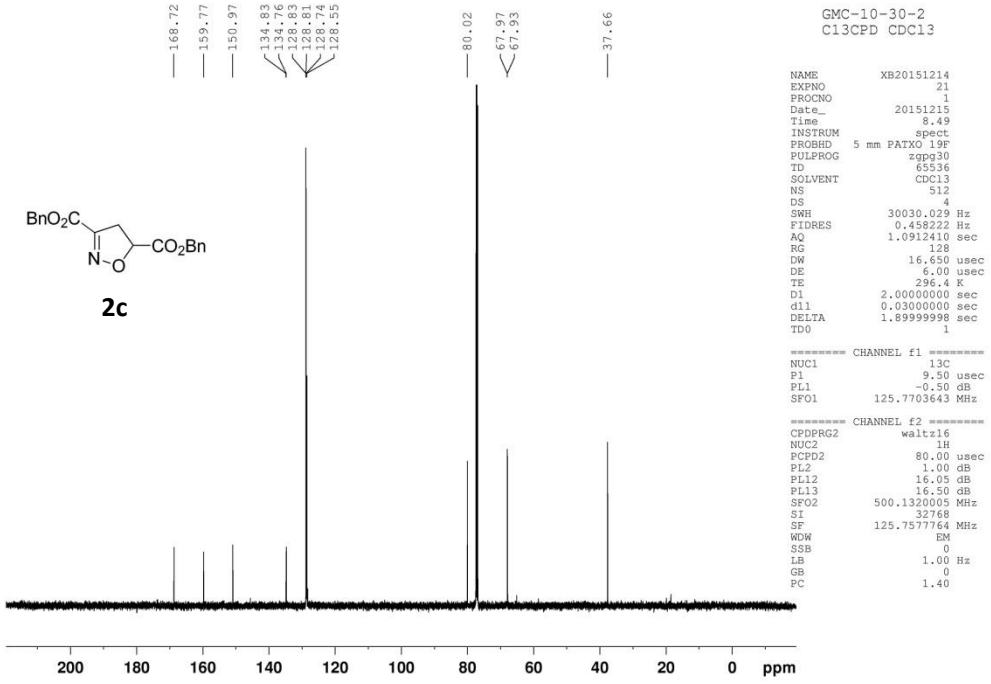

GMC-10-30-2
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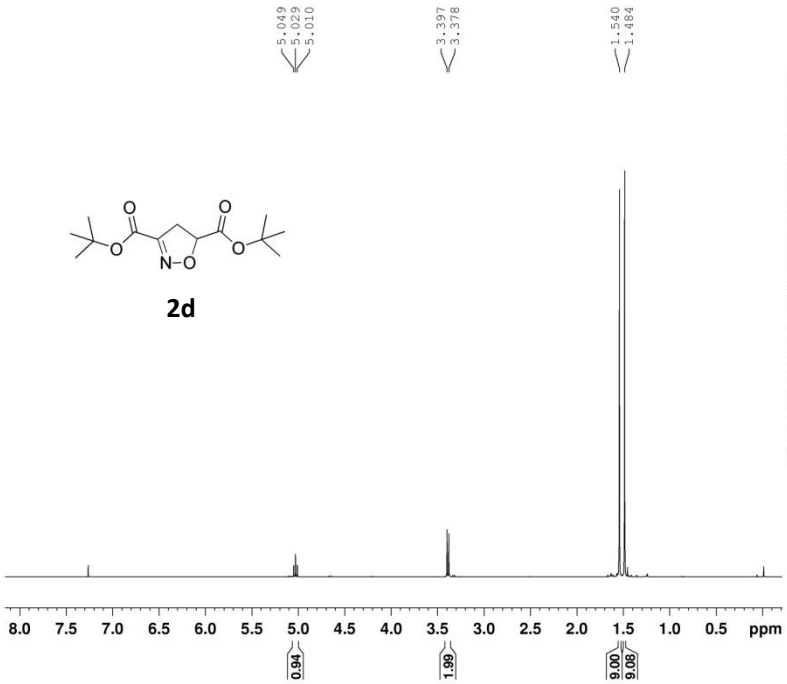
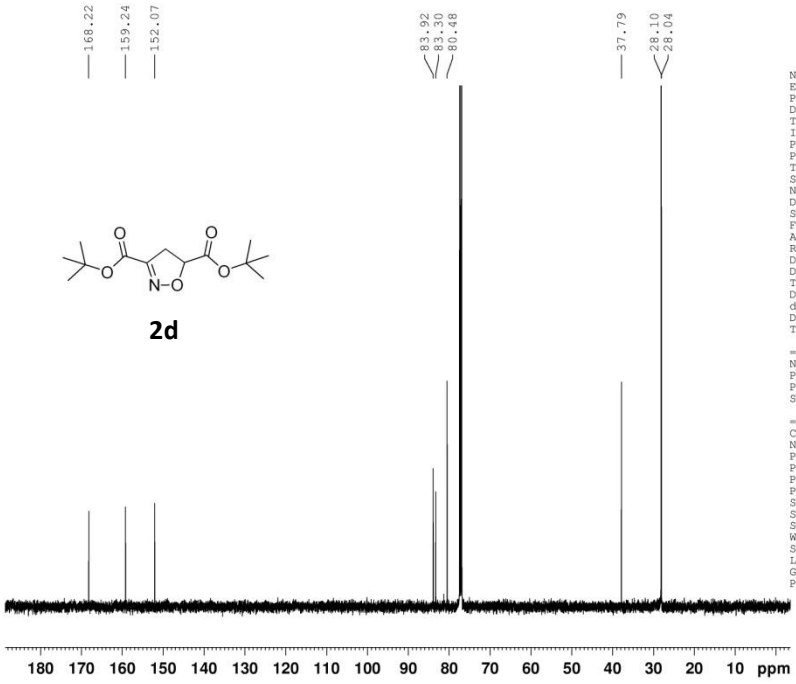
Date_	20151215
Time	8.49
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	512
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	128
DW	16.650 usec
DE	6.00 usec
TE	296.4 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.50 usec
PL1	-0.50 dB
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577764 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



2d

GMC-11-9
PROTON CDCl3

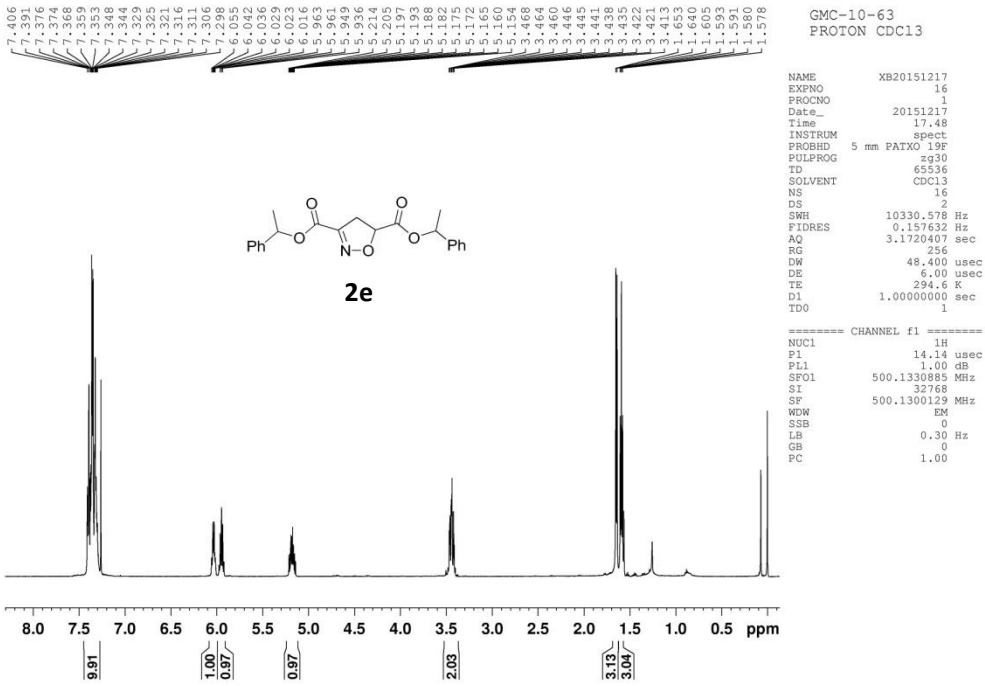
NAME	XB20151230
EXPNO	7
PROCNO	1
Date_	20151230
Time	10.00
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	128
DW	48.400 usec
DE	6.00 usec
TE	295.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300129 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

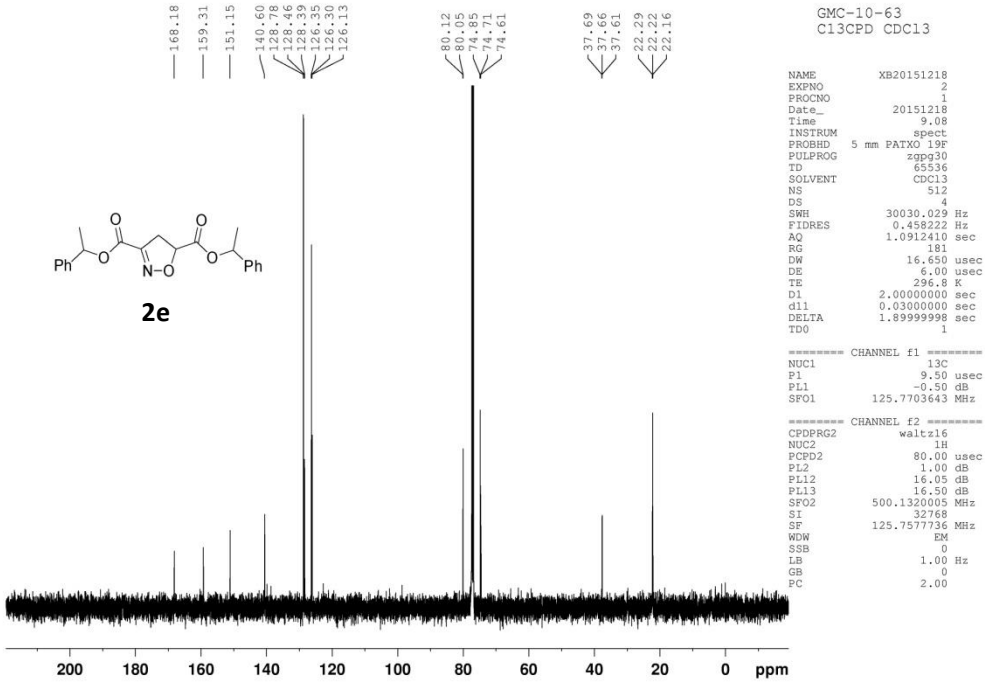


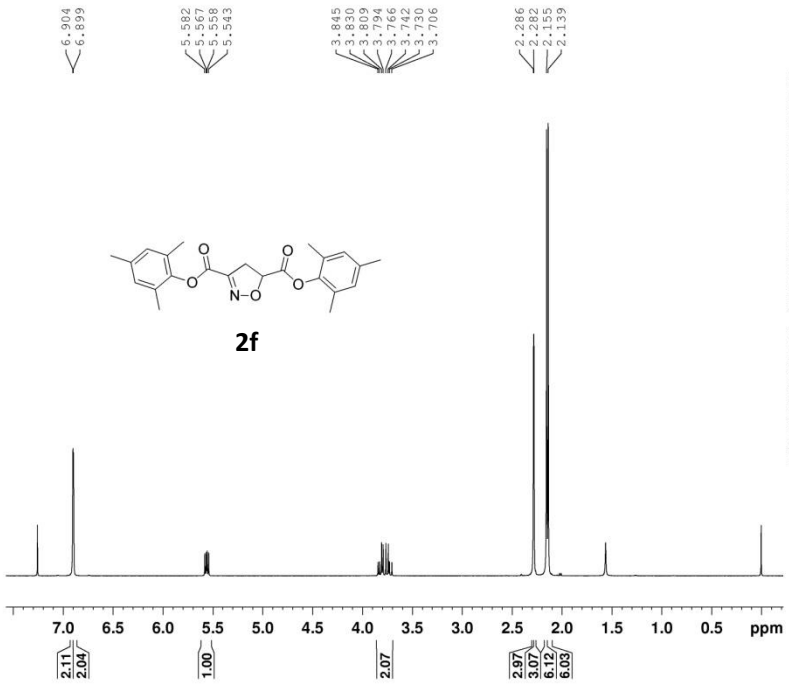
2d

GMC-11-9
C13CPD CDCl3

NAME	XB20151230
EXPNO	14
PROCNO	1
Date_	20151230
Time	11.54
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	259
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	128
DW	16.650 usec
DE	6.00 usec
TE	296.4 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.50 usec
PL1	-0.50 dB
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577736 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40


7.406
7.391
7.376
7.374
7.368
7.359
7.353
7.348
7.344
7.329
7.325
7.321
7.316
7.311
7.306
7.298
6.055
6.042
6.036
6.029
6.023
6.016
5.963
5.961
5.949
5.936
5.214
5.205
5.197
5.193
5.188
5.182
5.175
5.172
5.165
5.160
5.154
3.468
3.464
3.460
3.446
3.445
3.441
3.438
3.435
3.422
3.421
3.413
1.653
1.640
1.605
1.593
1.591
1.580
1.578
GMC-10-63
PROTON CDCl3
NAME XB20151217
EXPNO 16
PROCNO 1
Date_ 20151217
Time 17.48
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 256
DW 48.400 usec
DE 6.00 usec
TE 294.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.14 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300129 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Ph
O
O
N–O
O
O
Ph
2e
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
9.91
1.00
0.97
0.97
2.03
3.13
3.04

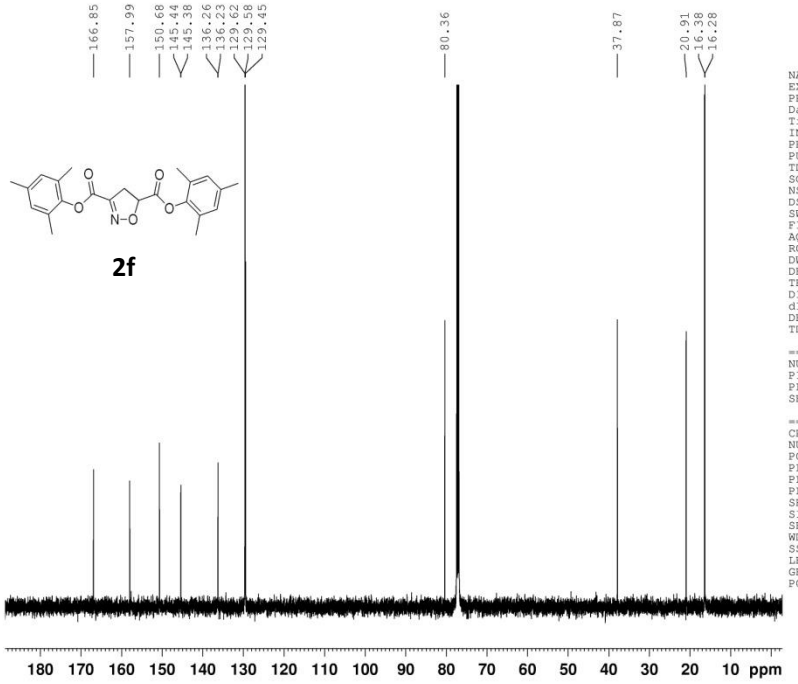

168.18
159.31
151.15
140.60
128.78
128.46
128.39
126.35
126.30
126.13
80.12
80.05
74.85
74.71
74.61
37.69
37.66
37.61
22.29
22.22
22.16
GMC-10-63
C13CPD CDC13
NAME XB20151218
EXPNO 2
PROCNO 1
Date_ 20151218
Time 9.08
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 181
DW 16.650 usec
DE 6.00 usec
TE 296.8 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.50 usec
PL1 -0.50 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 16.05 dB
PL13 16.50 dB
SFO2 500.1320005 MHz
SI 32768
SF 125.7577736 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
Ph
O
O
N–O
O
O
Ph
2e
200
180
160
140
120
100
80
60
40
20
0
ppm



2f

GMC-10-101
PROTON CDC13

NAME	XB20151110
EXPNO	2
PROCNO	1
Date_	20151110
Time	10.40
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	CDC13
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	203.2
DW	48.400 usec
DE	6.00 usec
TE	294.9 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300129 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

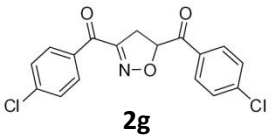


2f

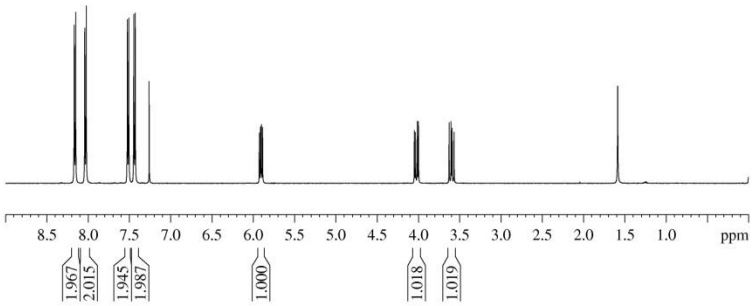
GMC-10-101
C13CPD CDC13

NAME	XB20151110
EXPNO	9
PROCNO	1
Date_	20151111
Time	7.17
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	CDC13
NS	1024
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	228.1
DW	16.650 usec
DE	6.00 usec
TE	296.2 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.50 usec
PL1	-0.50 dB
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577736 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	2.00

GYS−3−68
PROTON CDCl3



2g

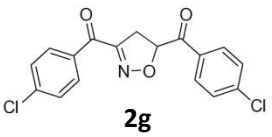


NAME	XB20160428
EXPNO	2
PROCNO	1
Date_	20160428
Time	10.45
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	256
DW	48.400 usec
DE	6.00 usec
TE	295.0 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========

NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300129 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

GYS−3−68
C13CPD CDCl3



2g

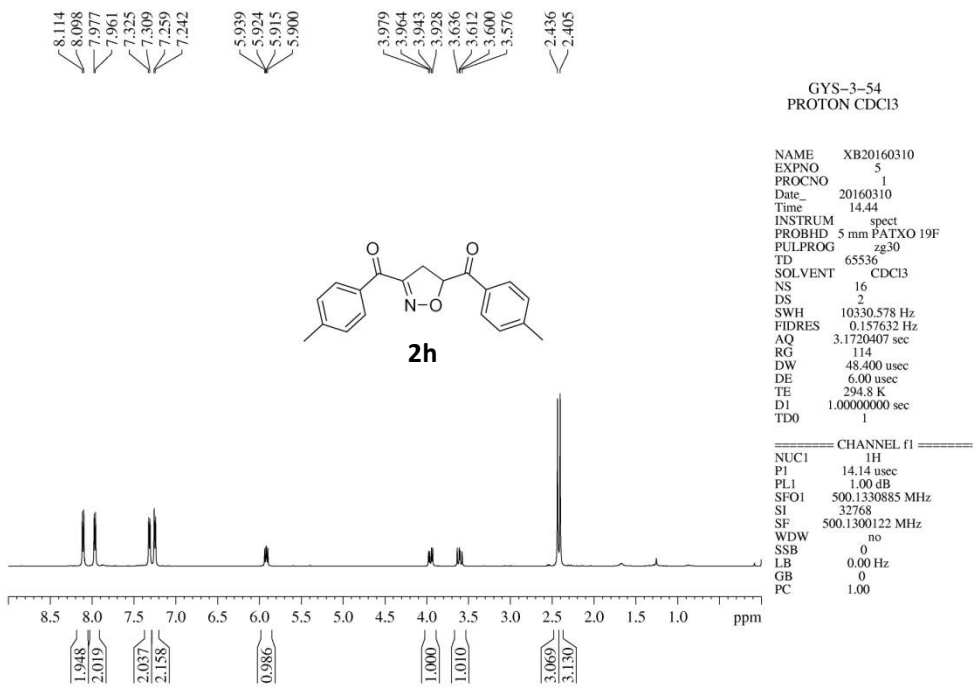
NAME	XB20160428
EXPNO	10
PROCNO	1
Date_	20160429
Time	6.35
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	512
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	101.6
DW	16.650 usec
DE	6.00 usec
TE	297.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

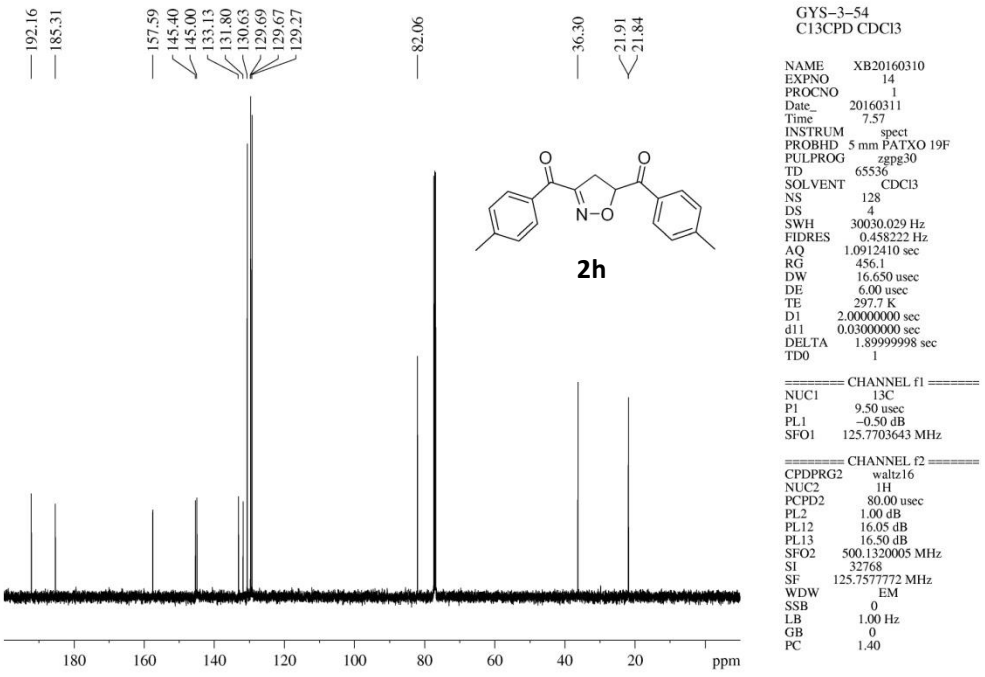
======== CHANNEL f1 ========

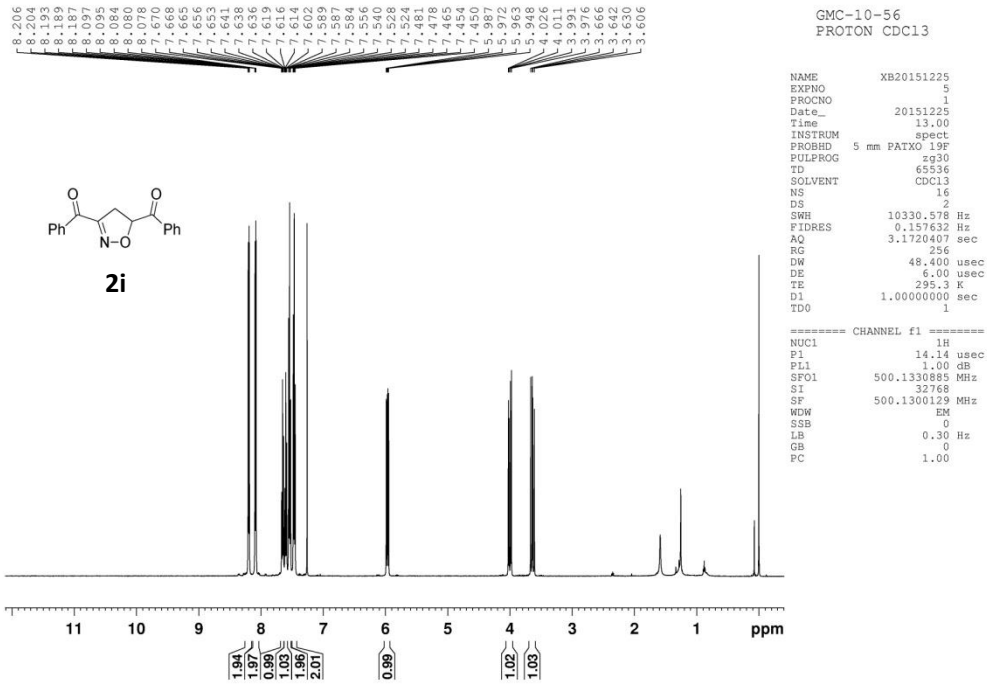
NUC1	13C
P1	9.50 usec
PL1	−0.50 dB
SFO1	125.7703643 MHz

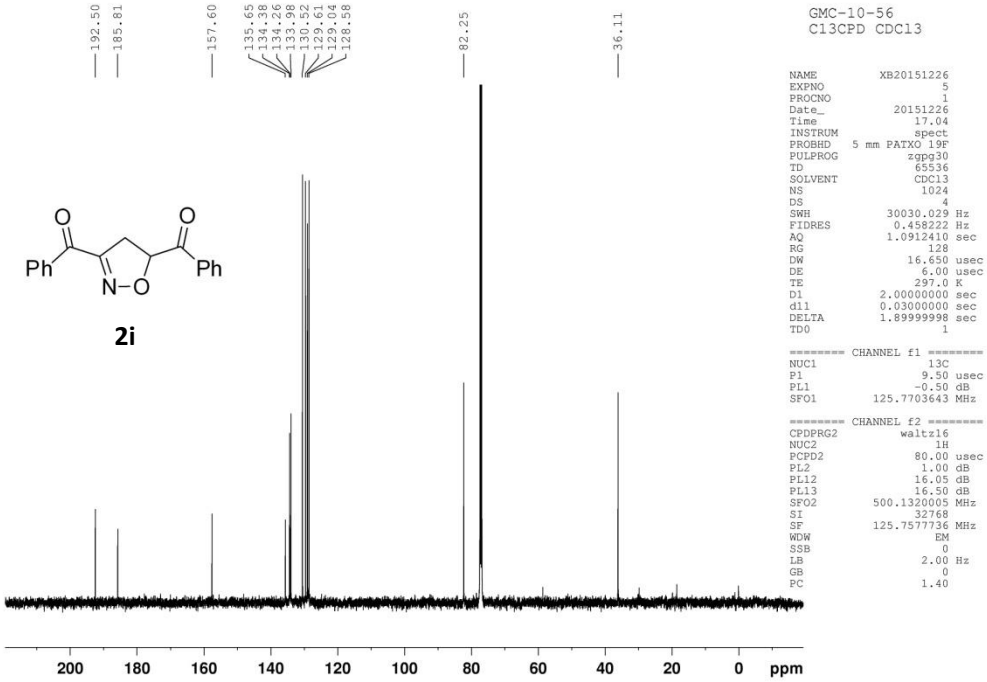
======== CHANNEL f2 ========

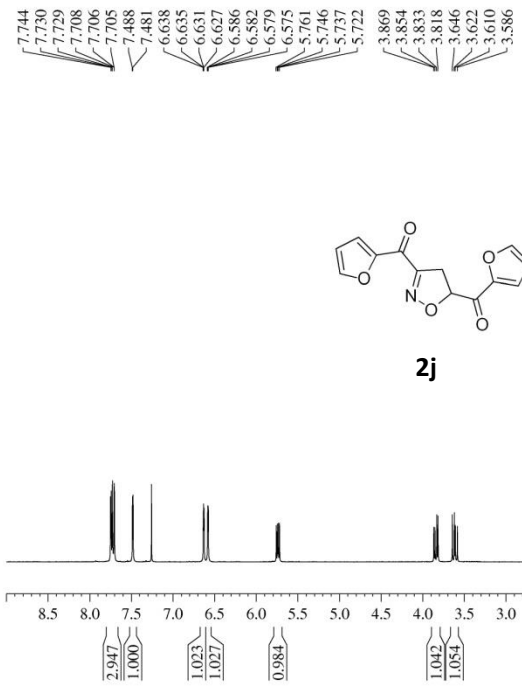
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577727 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

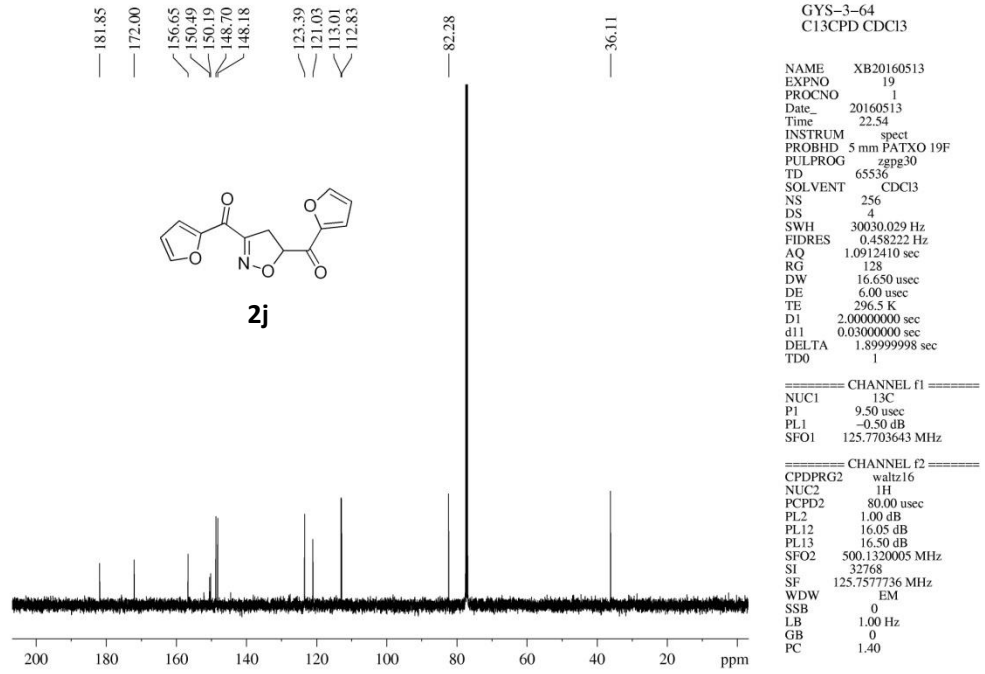

8.114
8.098
7.977
7.961
7.325
7.309
7.259
7.242
5.939
5.924
5.915
5.900
3.979
3.964
3.943
3.928
3.636
3.612
3.600
3.576
2.436
2.405
GYS-3-54
PROTON CDCl3
NAME XB20160310
EXPNO 5
PROCNO 1
Date_ 20160310
Time 14.44
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 114
DW 48.400 usec
DE 6.00 usec
TE 294.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.14 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300122 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
2h
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.948
2.019
2.037
2.158
0.986
1.000
1.010
3.069
3.130

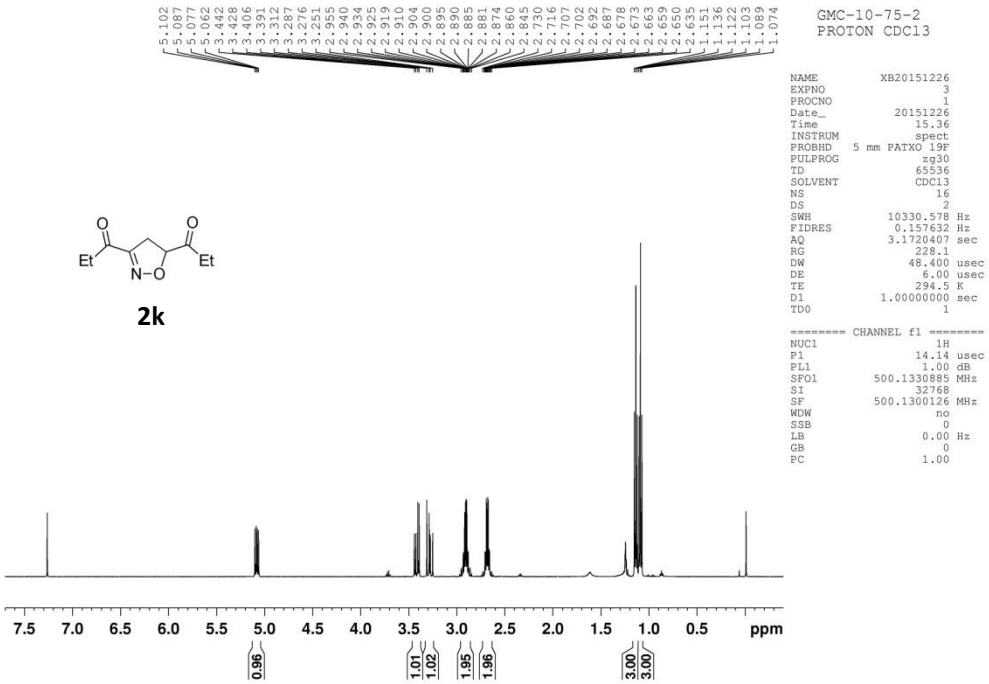

192.16
185.31
157.59
145.40
145.00
133.13
131.80
130.63
129.69
129.67
129.27
82.06
36.30
21.91
21.84
GYS-3-54
C13CPD CDCl3
NAME XB20160310
EXPNO 14
PROCNO 1
Date_ 20160311
Time 7.57
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 456.1
DW 16.650 usec
DE 6.00 usec
TE 297.7 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.50 usec
PL1 -0.50 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 16.05 dB
PL13 16.50 dB
SFO2 500.1320005 MHz
SI 32768
SF 125.7577772 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
2h
180 160 140 120 100 80 60 40 20 ppm

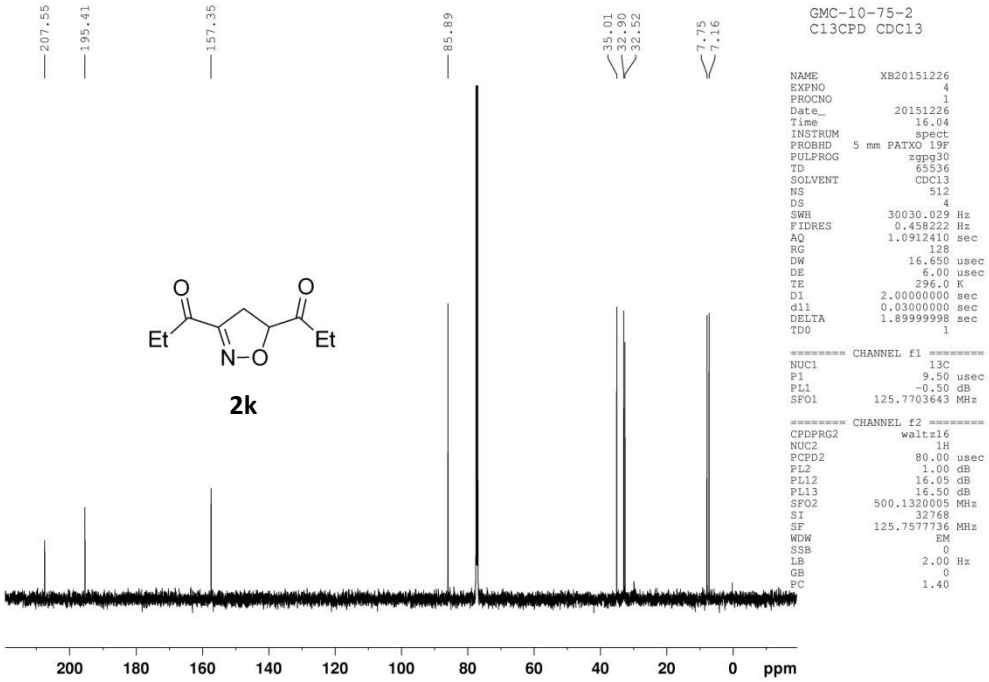

8.206
8.204
8.193
8.189
8.187
8.097
8.095
8.084
8.080
8.078
7.670
7.668
7.665
7.656
7.653
7.641
7.638
7.636
7.619
7.616
7.614
7.602
7.589
7.587
7.584
7.556
7.540
7.528
7.524
7.481
7.478
7.465
7.454
7.450
5.987
5.972
5.963
5.948
4.026
4.011
3.991
3.976
3.666
3.642
3.630
3.606
GMC-10-56
PROTON CDC13
NAME XB20151225
EXPNO 5
PROCNO 1
Date_ 20151225
Time 13.00
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 256
DW 48.400 usec
DE 6.00 usec
TE 295.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.14 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300129 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Ph
O
N–O
2i
11
10
9
8
7
6
5
4
3
2
1
ppm
1.94
1.97
0.98
1.03
1.96
2.01
0.99
1.02
1.03


192.50
185.81
157.60
135.65
134.38
134.26
133.98
130.52
129.61
129.04
128.58
82.25
36.11
GMC-10-56
C13CPD CDC13
NAME XB20151226
EXPNO 5
PROCNO 1
Date_ 20151226
Time 17.04
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 128
DW 16.650 usec
DE 6.00 usec
TE 297.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.50 usec
PL1 -0.50 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 16.05 dB
PL13 16.50 dB
SFO2 500.1320005 MHz
SI 32768
SF 125.7577736 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40
Ph
O
N–O
2i
200
180
160
140
120
100
80
60
40
20
0
ppm


7.744
7.730
7.729
7.708
7.706
7.705
7.488
7.481
6.638
6.635
6.631
6.627
6.586
6.582
6.579
6.575
5.761
5.746
5.737
5.722
3.869
3.854
3.833
3.818
3.646
3.622
3.610
3.586
GYS−3−64
PROTON CDCl3
NAME XB20160513
EXPNO 12
PROCNO 1
Date_ 20160513
Time 17.21
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 256
DW 48.400 usec
DE 6.00 usec
TE 295.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.14 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300129 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
2j
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.947
1.000
1.023
1.027
0.984
1.042
1.054


181.85
172.00
156.65
150.49
150.19
148.70
148.18
123.39
121.03
113.01
112.83
82.28
36.11
GYS−3−64
C13CPD CDCl3
NAME XB20160513
EXPNO 19
PROCNO 1
Date_ 20160513
Time 22.54
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 128
DW 16.650 usec
DE 6.00 usec
TE 296.5 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.50 usec
PL1 −0.50 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 16.05 dB
PL13 16.50 dB
SFO2 500.1320005 MHz
SI 32768
SF 125.7577736 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
2j
200 180 160 140 120 100 80 60 40 20 ppm

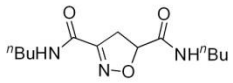

5.102
5.087
5.077
5.062
3.442
3.428
3.406
3.391
3.312
3.287
3.276
3.251
2.955
2.940
2.934
2.925
2.919
2.910
2.904
2.900
2.895
2.890
2.885
2.881
2.874
2.860
2.845
2.730
2.716
2.707
2.702
2.692
2.687
2.678
2.673
2.663
2.659
2.650
2.635
2.151
2.136
2.122
2.103
2.089
1.074
GMC-10-75-2
PROTON CDC13
NAME XB20151226
EXPNO 3
PROCNO 1
Date_ 20151226
Time 15.36
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 228.1
DW 48.400 usec
DE 6.00 usec
TE 294.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.14 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300126 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
O
O
Et
Et
N–O
2k
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.96
1.01
1.02
1.95
1.96
3.00
3.00


207.55
195.41
157.35
85.89
35.01
32.90
32.52
7.75
7.16
GMC-10-75-2
C13CPD CDC13
NAME XB20151226
EXPNO 4
PROCNO 1
Date_ 20151226
Time 16.04
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 512
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 128
DW 16.650 usec
DE 6.00 usec
TE 296.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.50 usec
PL1 -0.50 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 16.05 dB
PL13 16.50 dB
SFO2 500.1320005 MHz
SI 32768
SF 125.7577736 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40
O
O
Et
Et
N–O
2k
200 180 160 140 120 100 80 60 40 20 0 ppm

GYS−3−24
PROTON CDCl3

NAME XB20160509
EXPNO 10
PROCNO 1
Date_ 20160509
Time 10.43
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 322.5
DW 48.400 usec
DE 6.00 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 =======
NUC1 1H
P1 14.14 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300129 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



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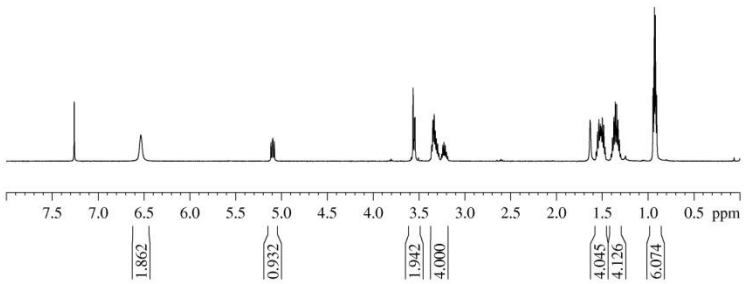
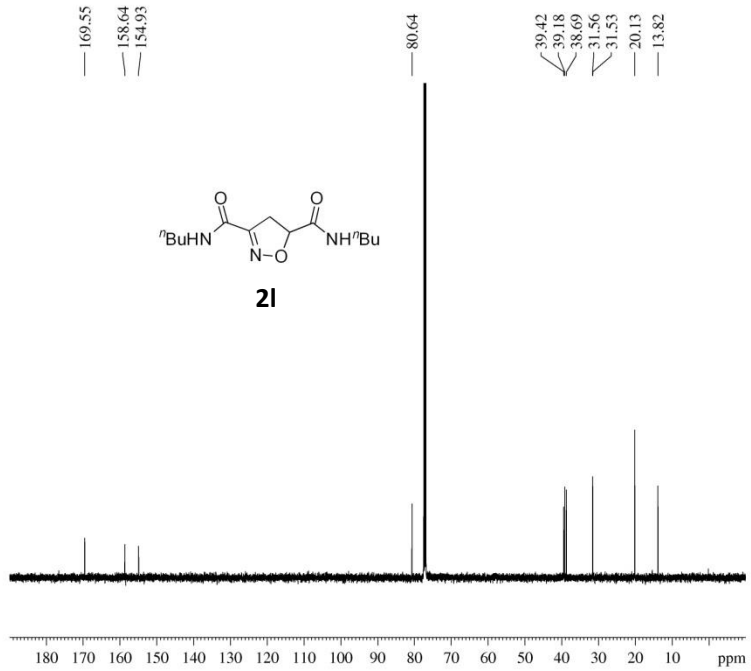




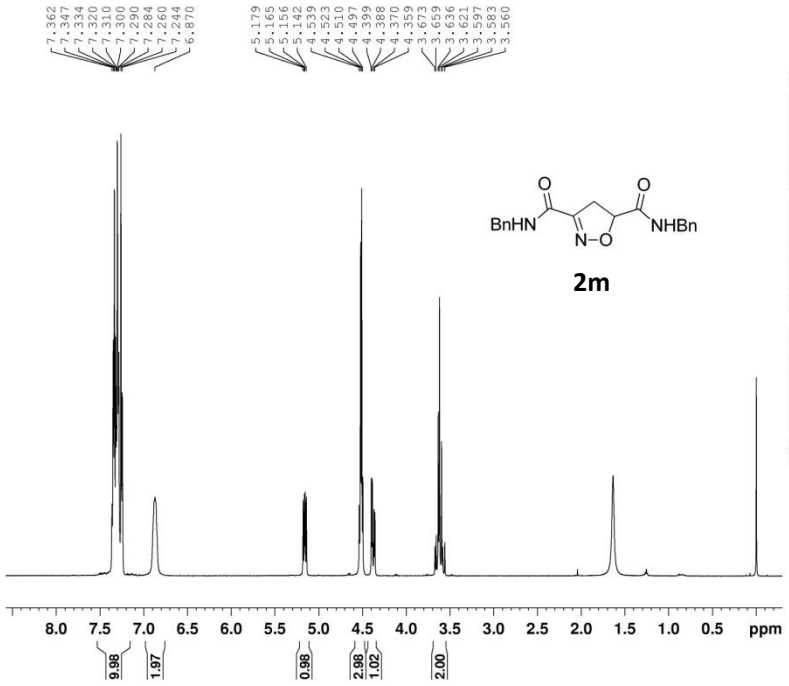
GYS−3−24
C13CPD CDCl3

NAME XB20160509
EXPNO 41
PROCNO 1
Date_ 20160510
Time 5.25
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 143.7
DW 16.650 usec
DE 6.00 usec
TE 296.5 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 =======
NUC1 13C
P1 9.50 usec
PL1 −0.50 dB
SFO1 125.7703643 MHz

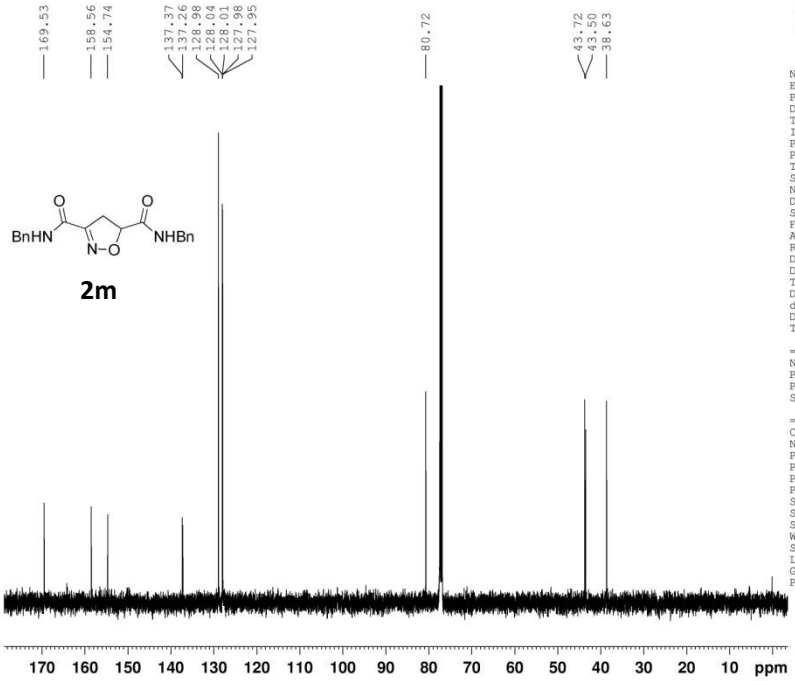
======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 16.05 dB
PL13 16.50 dB
SFO2 500.1320005 MHz
SI 32768
SF 125.7577727 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

2l



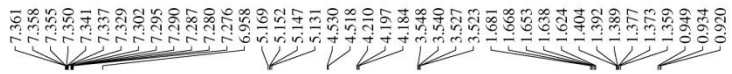
GMC-10-48-2-2
PROTON CDCl3

NAME	XB20150930
EXPNO	7
PROCNO	1
Date_	20150930
Time	16.22
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	256
DW	48.400 usec
DE	6.00 usec
TE	295.9 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300129 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



GMC-10-48-2-2
C13CPD CDCl3

NAME	XB20150930
EXPNO	15
PROCNO	1
Date_	20150930
Time	19.43
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	512
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	143.7
DW	16.650 usec
DE	6.00 usec
TE	297.6 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.50 usec
PL1	-0.50 dB
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577736 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

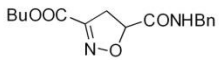


GYS–2–52
PROTON CDCl3

NAME	XB20160127
EXPNO	3
PROCNO	1
Date_	20160127
Time	14.17
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	161.3
DW	48.400 usec
DE	6.00 usec
TE	294.8 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ======

NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300126 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



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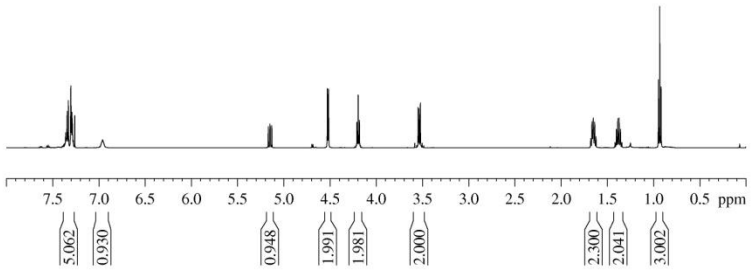


GYS–2–52
C13CPD CDCl3

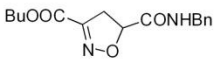
NAME	XB20160127
EXPNO	22
PROCNO	1
Date_	20160127
Time	18.49
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	512
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	128
DW	16.650 usec
DE	6.00 usec
TE	296.9 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

======== CHANNEL f1 ========

NUC1	13C
P1	9.50 usec
PL1	−0.50 dB
SFO1	125.7703643 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



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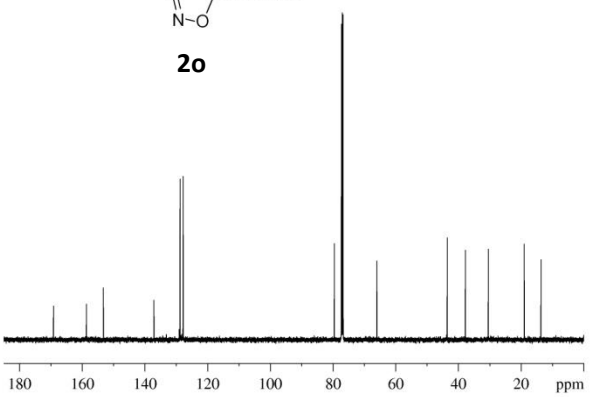
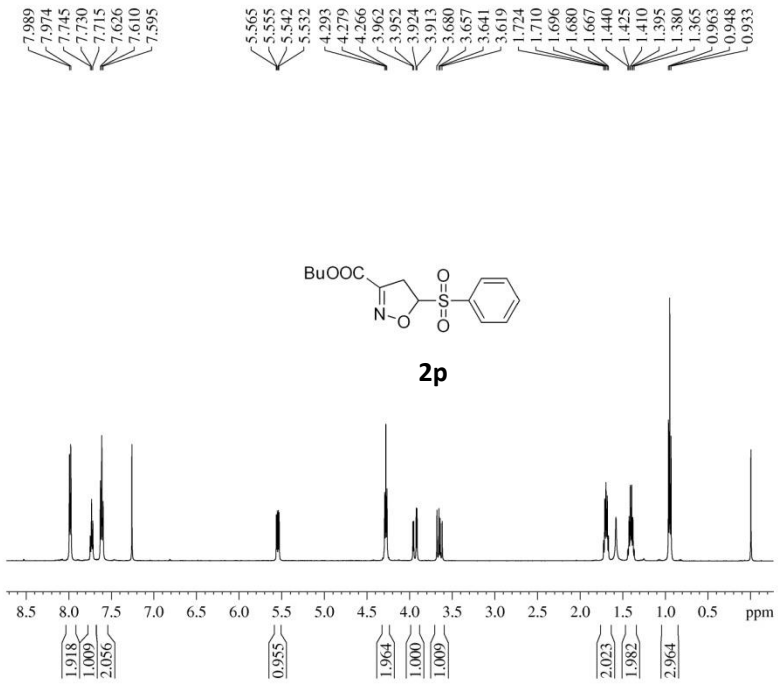


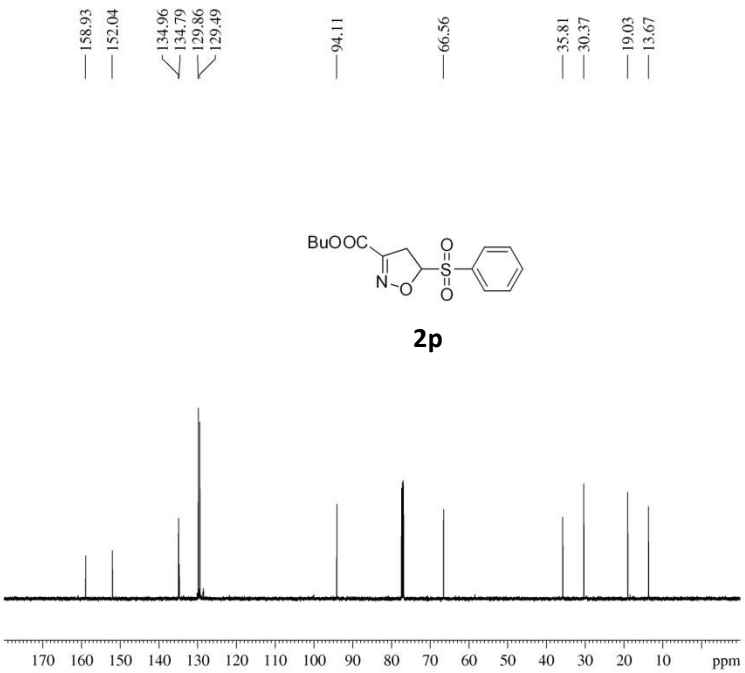


GYS-3-74
PROTON CDCl3

NAME	XB20160602
EXPNO	4
PROCNO	1
Date_	20160602
Time	10.48
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	322.5
DW	48.400 usec
DE	6.00 usec
TE	295.5 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 =======

NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300129 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



GYS-3-74
C13CPD CDCl3

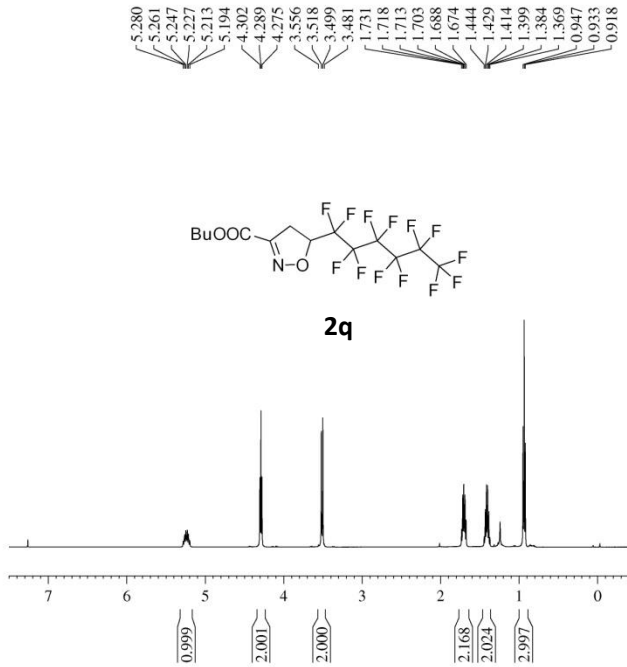
NAME	XB20160324
EXPNO	1
PROCNO	1
Date_	20160324
Time	9.08
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	128
DS	4
SWH	30030.029 H
FIDRES	0.458222 H
AQ	1.0912410 s
RG	128
DW	16.650 u
DE	6.00 u
TE	295.6 K
D1	2.00000000 s
d11	0.03000000 s
DELTA	1.89999998 s
TD0	1

======== CHANNEL f1 =====

NUC1	13C
P1	9.50 u
PL1	-0.50 d
SFO1	125.7703643 M

======== CHANNEL f2 =====

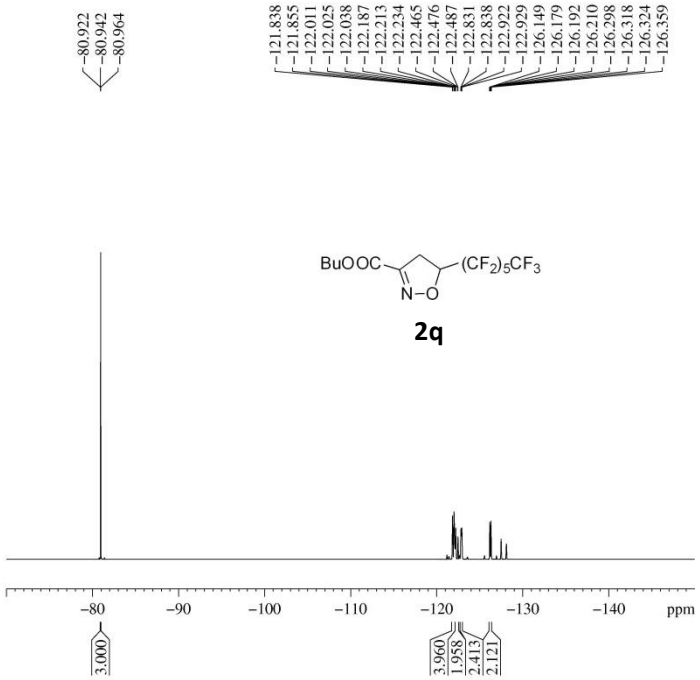
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 u
PL2	1.00 d
PL12	16.05 d
PL13	16.50 d
SFO2	500.1320005 M
SI	32768
SF	125.7577828 M
WDW	EM
SSB	0
LB	1.00 H
GB	0
PC	1.40



GYS–3–85
PROTON CDCl3

NAME XB20160801
EXPNO 5
PROCNO 1
Date_ 20160801
Time 14.52
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 64
DW 48.400 usec
DE 6.00 usec
TE 295.8 K
D1 1.00000000 sec
TD0 1

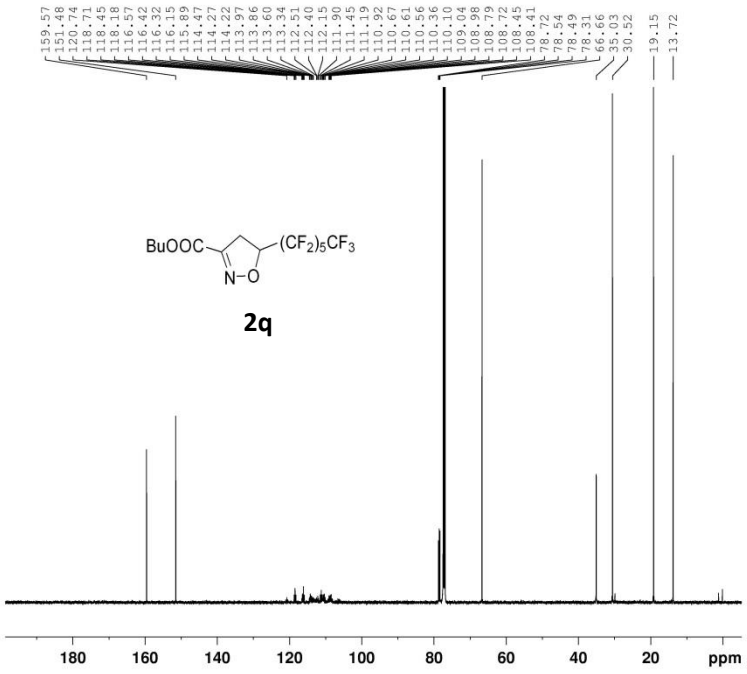
======== CHANNEL f1 ========
NUC1 1H
P1 14.24 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300126 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



GYS–3–85
19Fdeft CDCl3 D

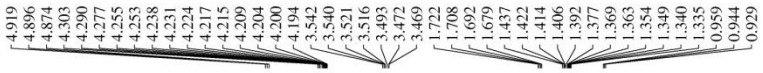
NAME XB20161104
EXPNO 5
PROCNO 1
Date_ 20161104
Time 15.09
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg
TD 131072
SOLVENT CDCl3
NS 8
DS 4
SWH 100000.000 Hz
FIDRES 0.762939 Hz
AQ 0.6554150 sec
RG 90.5
DW 5.000 usec
DE 6.00 usec
TE 296.4 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 19F
P1 19.30 usec
PL1 4.00 dB
SFO1 470.5453180 MHz
SI 65536
SF 470.5923770 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



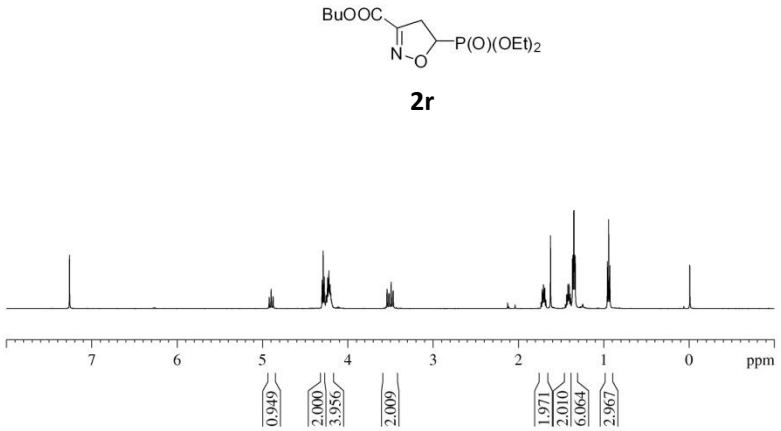
GYS-3-85
C13CPD CDCl3

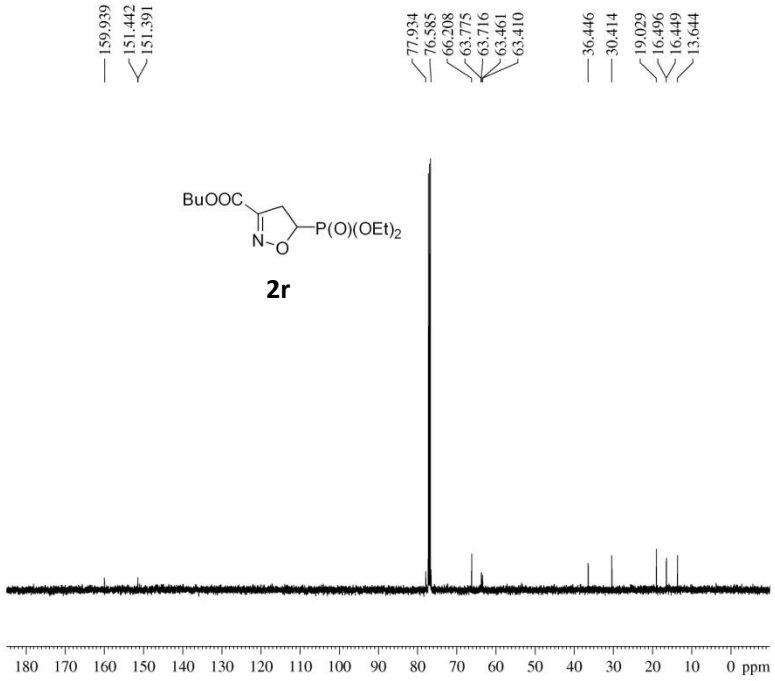
NAME	XB20190429
EXPNO	30
PROCNO	1
Date_	20190429
Time	18.28
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	8800
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	20642.5
DW	16.650 usec
DE	6.50 usec
TE	298.5 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.50 usec
PL1	-0.50 dB
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	15.76 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	0.70



GYS-3-98
PROTON CDCl3

NAME	XB20160513
EXPNO	11
PROCNO	1
Date_	20160513
Time	17.15
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	256
DW	48.400 usec
DE	6.00 usec
TE	295.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300129 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

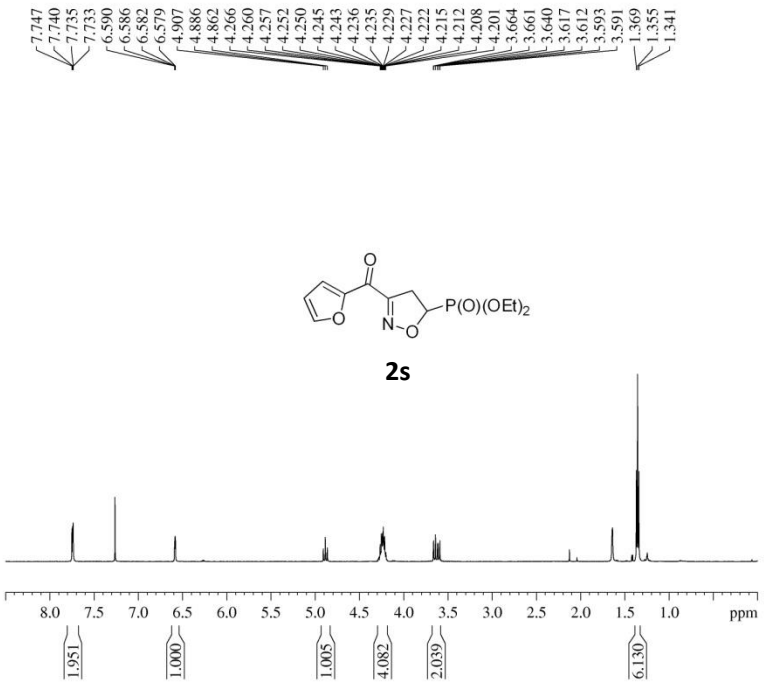




2r

GYS−3−98
C13CPD CDCl3

NAME	XB20161201
EXPNO	20
PROCNO	1
Date_	20160513
Time	23.11
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	256
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	128
DW	16.650 usec
DE	6.00 usec
TE	296.6 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 =======	
NUC1	13C
P1	9.50 usec
PL1	−0.50 dB
SFO1	125.7703643 MHz
======== CHANNEL f2 =======	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	0.10



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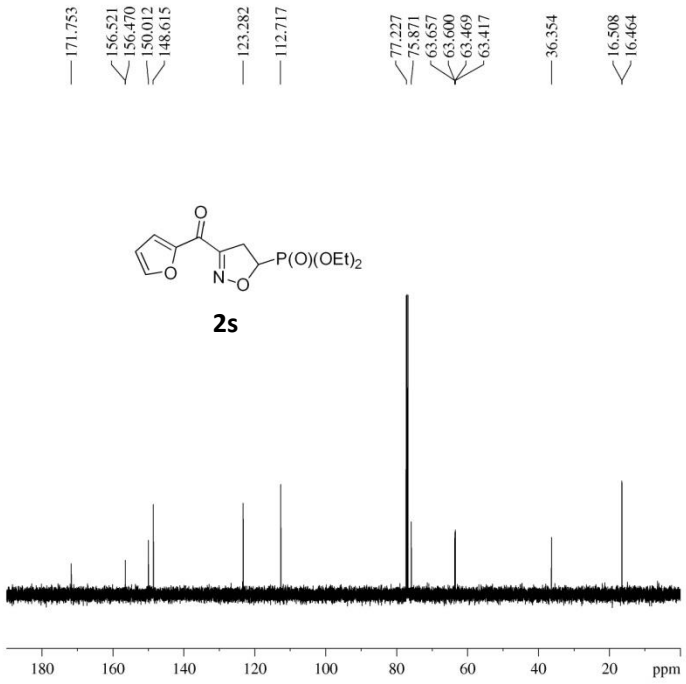
GYS−3−116
PROTON CDCl3

NAME	XB20160520
EXPNO	16
PROCNO	1
Date_	20160520
Time	17.15
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	256
DW	48.400 usec
DE	6.00 usec
TE	294.8 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 =======	
NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300129 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00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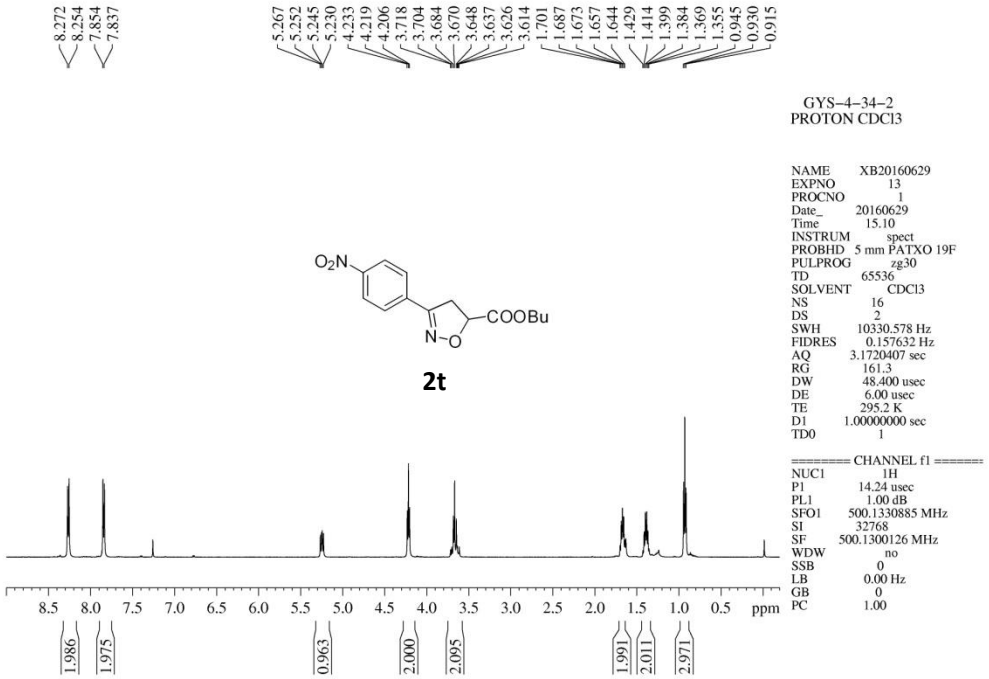
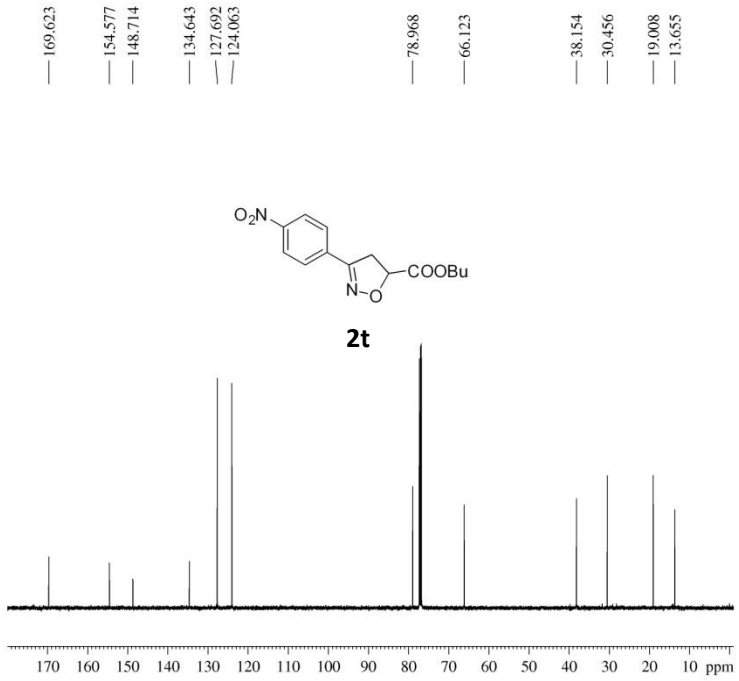
156.521
156.470
150.012
148.615
123.282
112.717
77.227
75.871
63.657
63.600
63.469
63.417
36.354
16.508
16.464
GYS-3-116
C13CPD CDCl3
NAME XB20160511
EXPNO 20
PROCNO 1
Date_ 20160511
Time 23.02
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 143.7
DW 16.650 usec
DE 6.00 usec
TE 296.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.50 usec
PL1 -0.50 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 16.05 dB
PL13 16.50 dB
SFO2 500.1320005 MHz
SI 32768
SF 125.7577890 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
O
O
N
O
P(O)(OEt)2
2s
180
160
140
120
100
80
60
40
20
ppm


8.272
8.254
7.854
7.837
5.267
5.252
5.245
5.230
4.233
4.219
4.206
3.718
3.704
3.684
3.670
3.648
3.637
3.626
3.614
1.701
1.687
1.673
1.657
1.644
1.429
1.414
1.399
1.384
1.369
1.355
0.945
0.930
0.915
GYS-4-34-2
PROTON CDCl3
NAME XB20160629
EXPNO 13
PROCNO 1
Date_ 20160629
Time 15.10
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 161.3
DW 48.400 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.24 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300126 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
O2N
COOBu
N
O
2t
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.986
1.975
0.963
2.000
2.095
1.991
2.011
2.971



GYS−4−34−2
C13CPD CDCl3

NAME	XB20160702
EXPNO	2
PROCNO	1
Date_	20160703
Time	10.27
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	512
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	203.2
DW	16.650 usec
DE	6.00 usec
TE	296.7 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

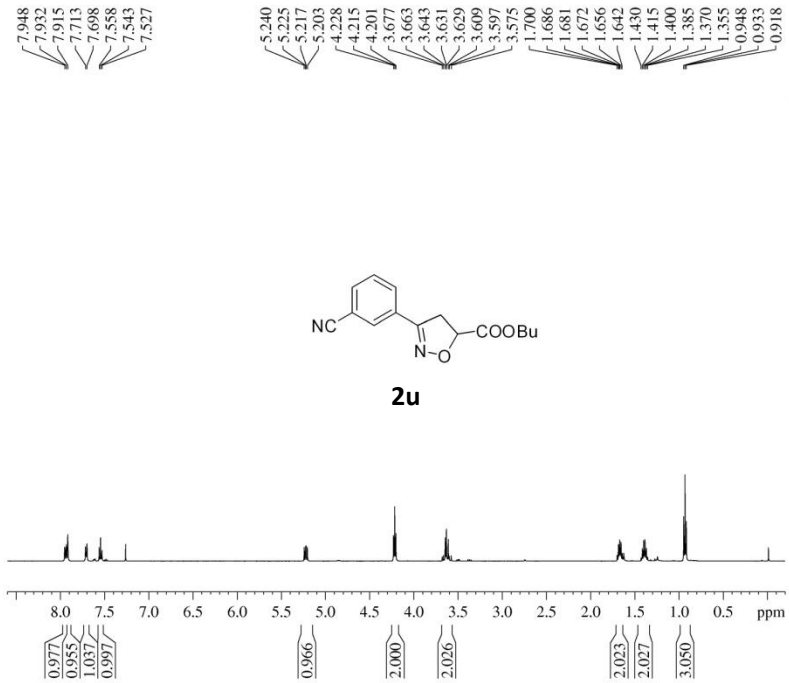
======== CHANNEL f1 ========

NUC1	13C
P1	9.50 usec
PL1	−0.50 dB
SFO1	125.7703643 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	15.99 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

2t



GYS−3−140−3
PROTON CDCl3

NAME	XB20160519
EXPNO	9
PROCNO	1
Date_	20160519
Time	14.18
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	181
DW	48.400 usec
DE	6.00 usec
TE	295.8 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========

NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300129 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

2u

GYS-3-140-3
C13CPD CDCl3

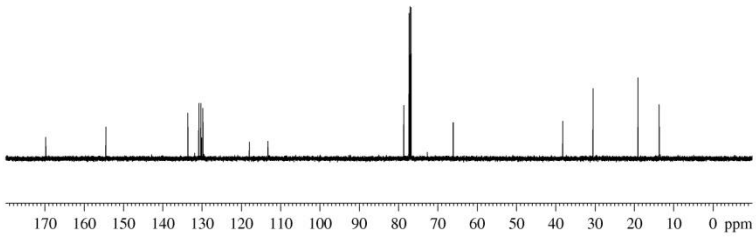
NAME	XB20160520
EXPNO	23
PROCNO	1
Date_	20160521
Time	8.34
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	256
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	287.4
DW	16.650 usec
DE	6.00 usec
TE	297.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

======== CHANNEL f1 ========

NUC1	13C
P1	9.50 usec
PL1	−0.50 dB
SFO1	125.7703643 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.40



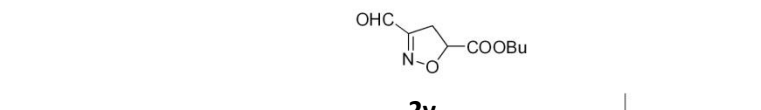
2u

GYS-2-91
PROTON CDCl3

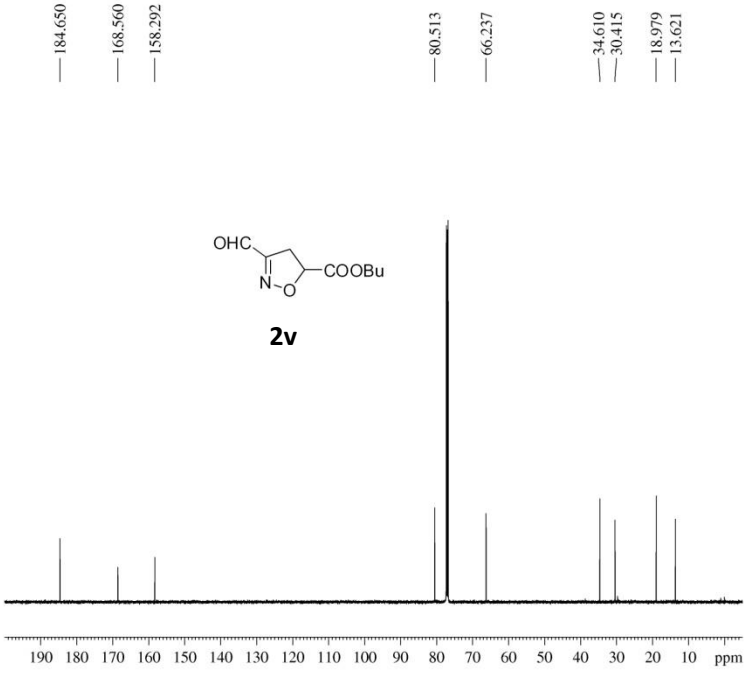
NAME	XB20161104
EXPNO	4
PROCNO	1
Date_	20161104
Time	15.04
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	203.2
DW	48.400 usec
DE	6.00 usec
TE	295.9 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ======

NUC1	1H
P1	14.24 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300129 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



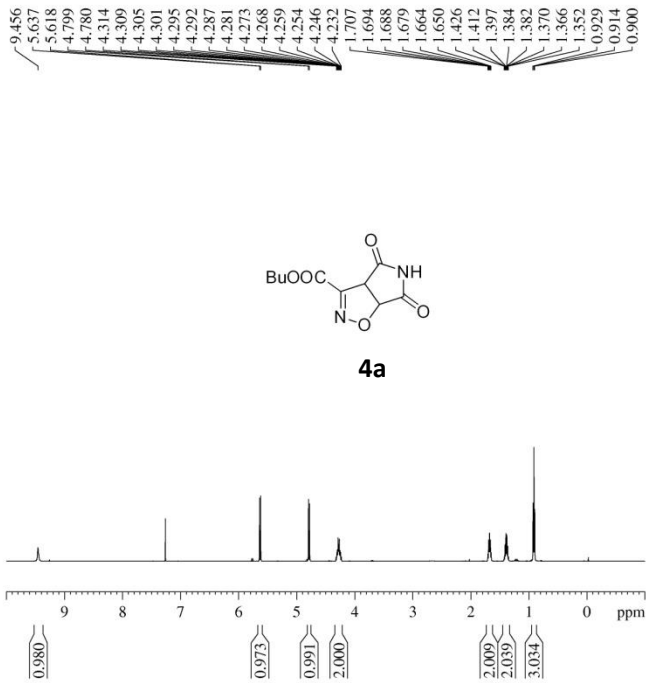
2v



2v

GYS-2-91
C13CPD CDCl3

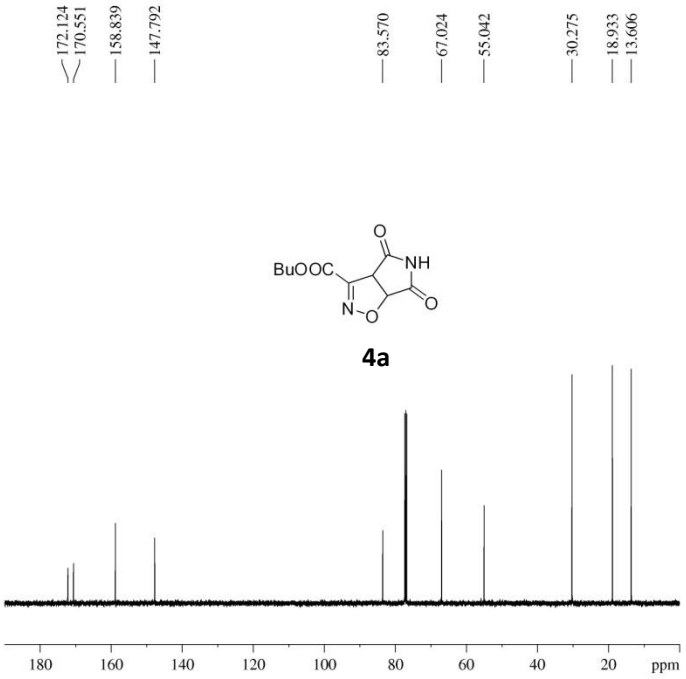
NAME	XB20161105
EXPNO	1
PROCNO	1
Date_	20161105
Time	10.20
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	322.5
DW	16.650 usec
DE	6.00 usec
TE	298.4 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.50 usec
PL1	-0.50 dB
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	15.99 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



4a

GYS-2-97
PROTON CDCl3

NAME	XB20160127
EXPNO	4
PROCNO	1
Date_	20160127
Time	14.23
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	64
DW	48.400 usec
DE	6.00 usec
TE	295.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ======	
NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300126 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



4a

GYS-2-97
C13CPD CDCl3

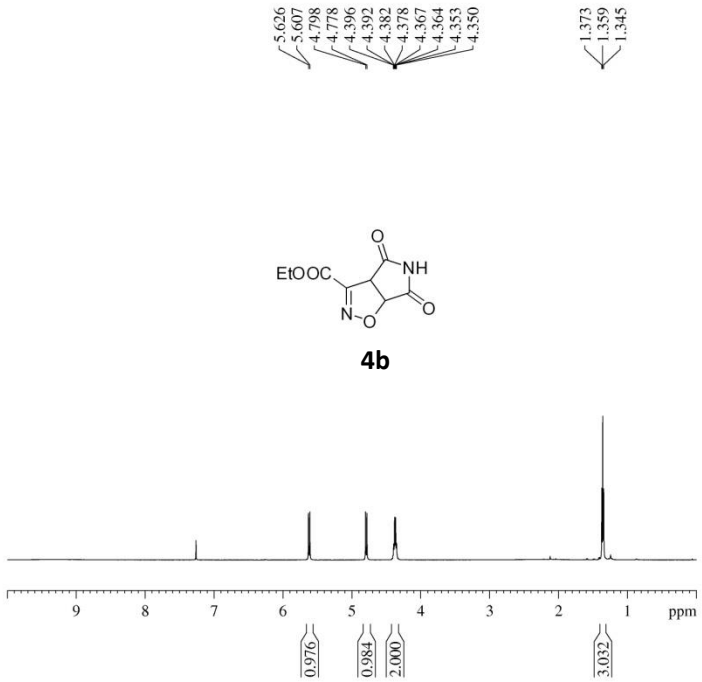
NAME	XB20160127
EXPNO	28
PROCNO	1
Date_	20160128
Time	1.42
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	128
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	143.7
DW	16.650 usec
DE	6.00 usec
TE	296.8 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

======== CHANNEL f1 ========

NUC1	13C
P1	9.50 usec
PL1	-0.50 dB
SFO1	125.7703643 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



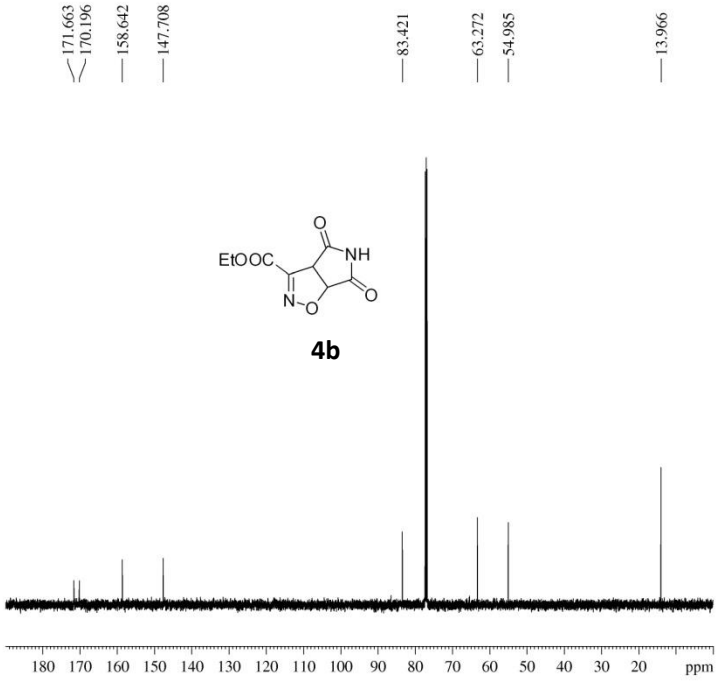
4b

GYS-2-160-1
PROTON CDCl3

NAME	XB20160510
EXPNO	17
PROCNO	1
Date_	20160510
Time	15.07
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	256
DW	48.400 usec
DE	6.00 usec
TE	295.1 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========

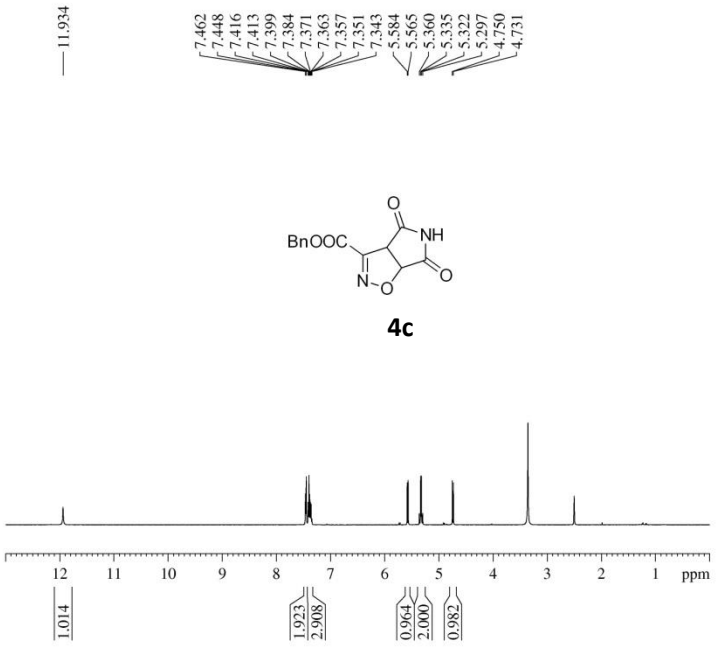
NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300129 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



4b

GYS-2-160-1
C13CPD CDCl3

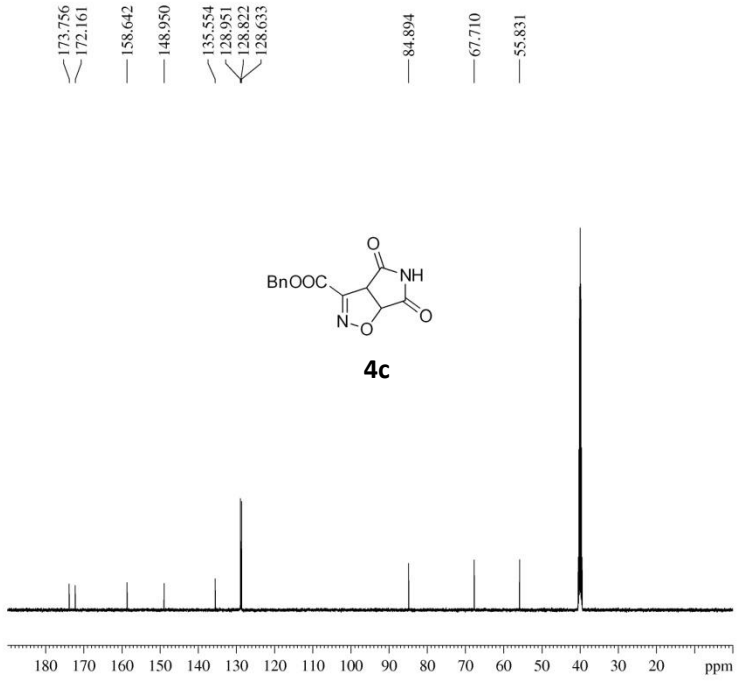
NAME	XB20160511
EXPNO	1
PROCNO	1
Date_	20160511
Time	8.23
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	128
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	322.5
DW	16.650 usec
DE	6.00 usec
TE	296.9 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.50 usec
PL1	-0.50 dB
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



4c

GYS-3-157
PROTON DMSO

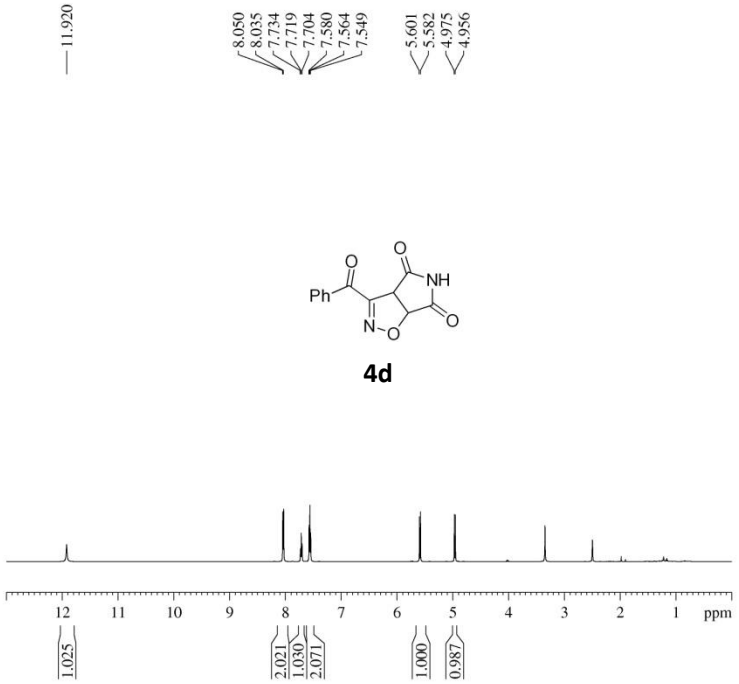
NAME	XB20160527
EXPNO	23
PROCNO	1
Date_	20160527
Time	14.42
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	DMSO
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	228.1
DW	48.400 usec
DE	6.00 usec
TE	295.7 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300044 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



4c

GYS-3-157
C13CPD DMSO

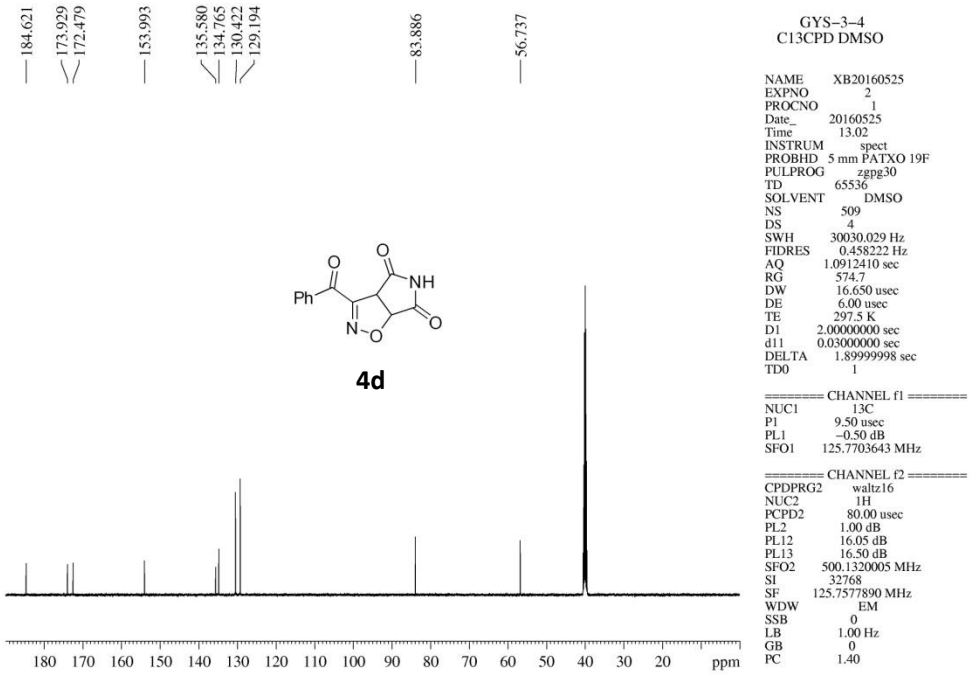
NAME	XB20160527
EXPNO	32
PROCNO	1
Date_	20160527
Time	21.59
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	DMSO
NS	512
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	322.5
DW	16.650 usec
DE	6.00 usec
TE	297.2 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.50 usec
PL1	-0.50 dB
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



4d

GYS-3-4
PROTON DMSO

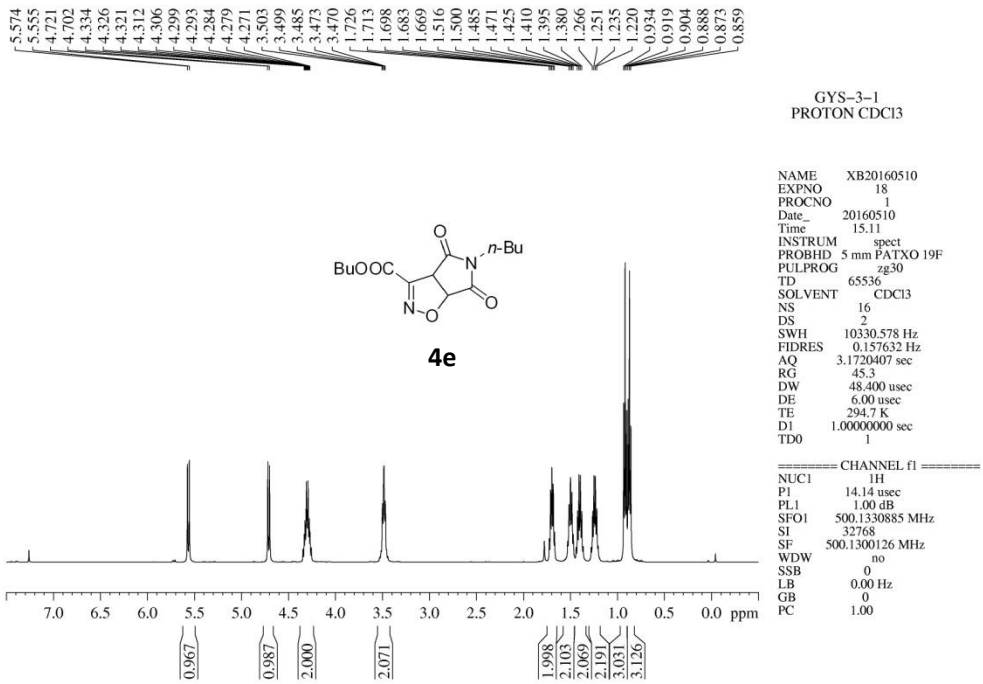
NAME	XB20160523
EXPNO	17
PROCNO	1
Date_	20160523
Time	11.16
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	DMSO
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	228.1
DW	48.400 usec
DE	6.00 usec
TE	295.5 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ======	
NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300041 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



4d

GYS−3−4
C13CPD DMSO

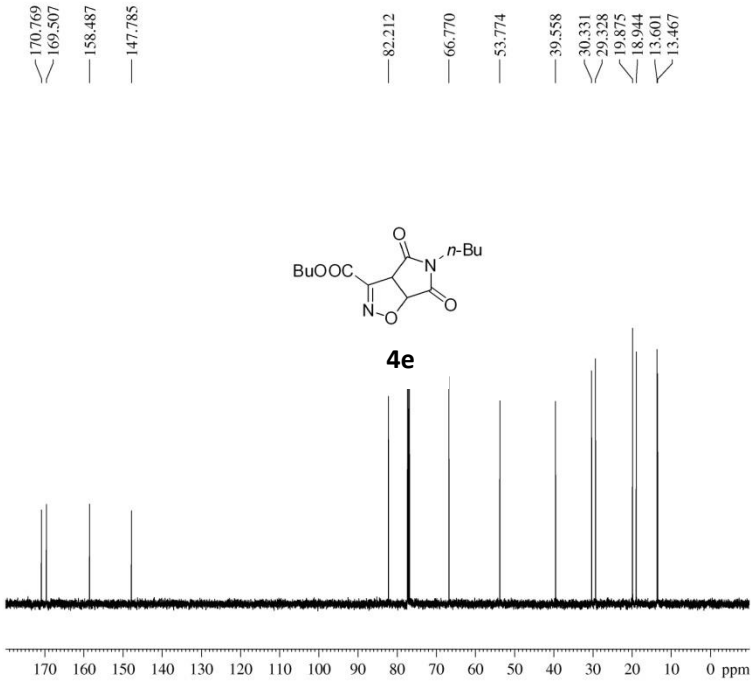
NAME	XB20160525
EXPNO	2
PROCNO	1
Date_	20160525
Time	13.02
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	DMSO
NS	509
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	574.7
DW	16.650 usec
DE	6.00 usec
TE	297.5 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.50 usec
PL1	−0.50 dB
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



4e

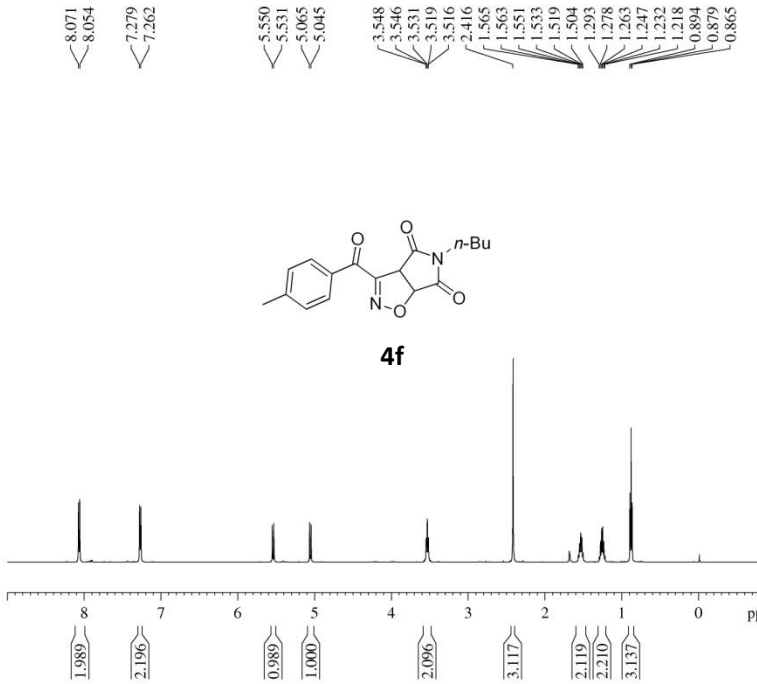
GYS−3−1
PROTON CDCl3

NAME	XB20160510
EXPNO	18
PROCNO	1
Date_	20160510
Time	15.11
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	45.3
DW	48.400 usec
DE	6.00 usec
TE	294.7 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300126 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



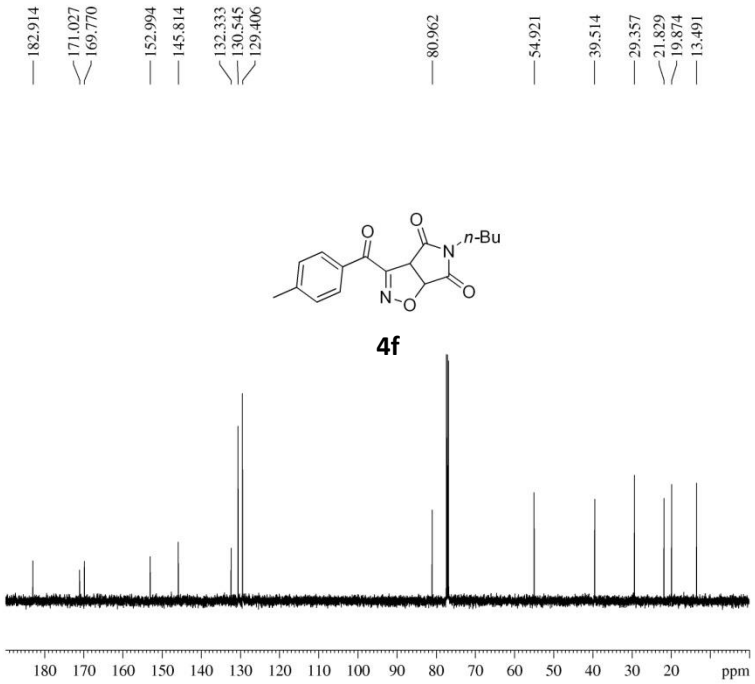
GYS−3−1
C13CPD CDCl3

NAME	XB20160511
EXPNO	17
PROCNO	1
Date_	20160511
Time	22.24
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	64
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	161.3
DW	16.650 usec
DE	6.00 usec
TE	296.3 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.50 usec
PL1	−0.50 dB
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



GYS−3−94
PROTON CDCl3

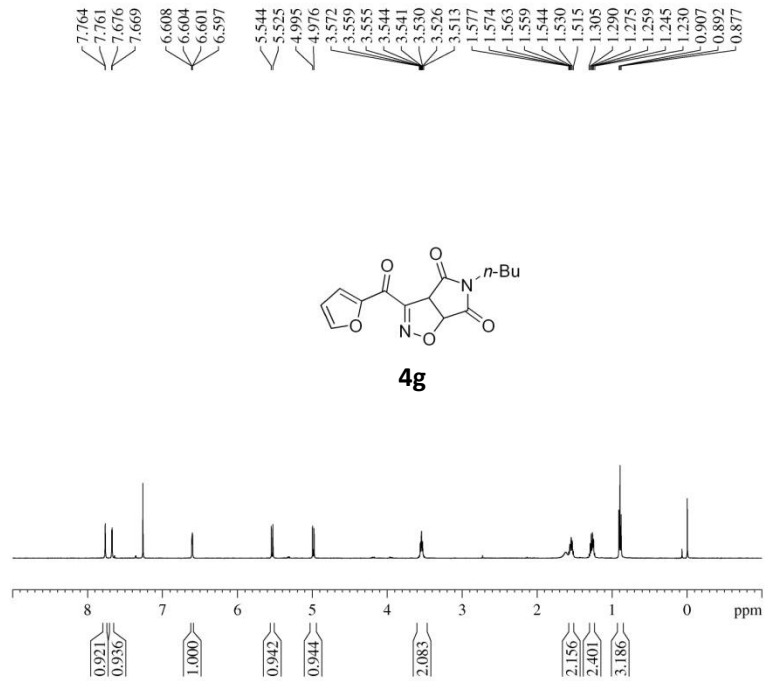
NAME	XB20160512
EXPNO	6
PROCNO	1
Date_	20160512
Time	8.58
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	143.7
DW	48.400 usec
DE	6.00 usec
TE	295.5 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300129 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



4f

GYS−3−94
C13CPD CDCl3

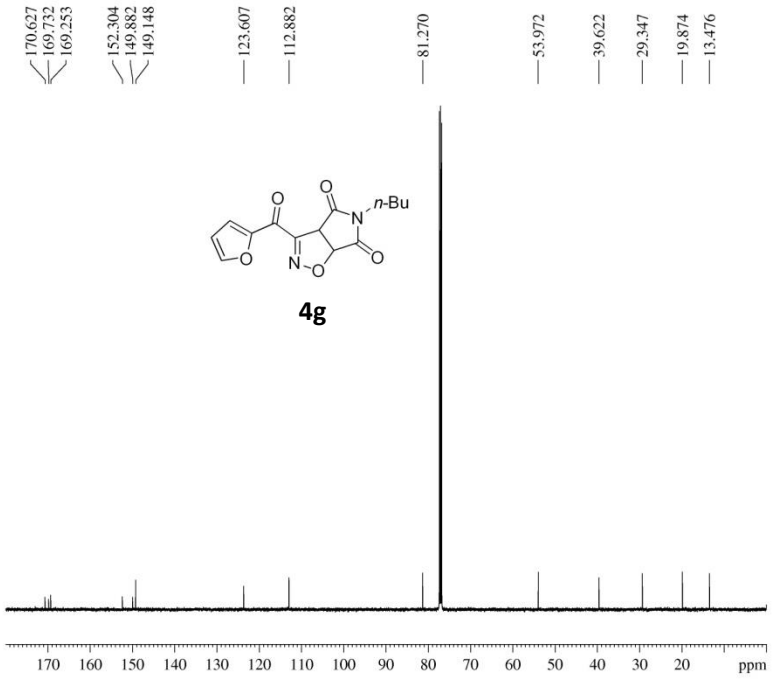
NAME	XB20160512
EXPNO	11
PROCNO	1
Date_	20160513
Time	3.43
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	64
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	101.6
DW	16.650 usec
DE	6.00 usec
TE	296.1 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.50 usec
PL1	−0.50 dB
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



4g

GYS−3−111
PROTON CDCl3

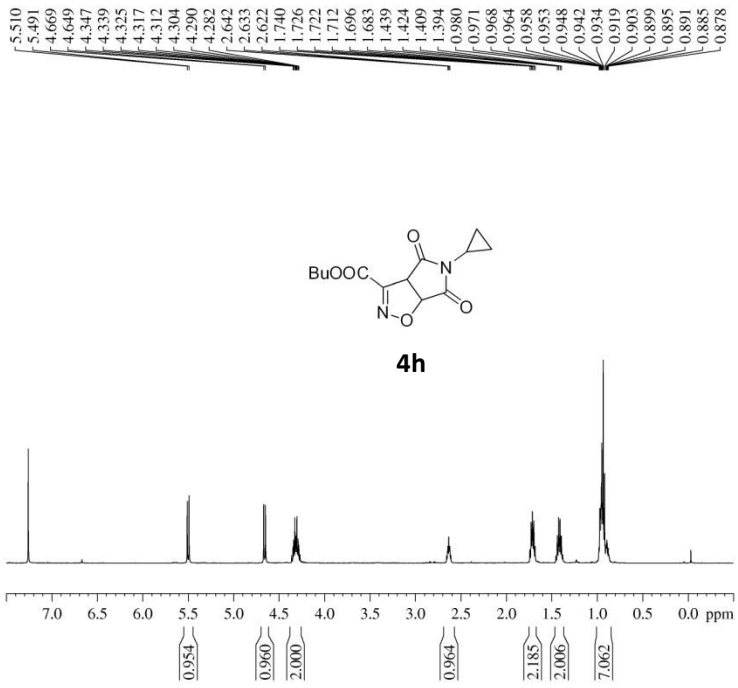
NAME	XB20160523
EXPNO	13
PROCNO	1
Date_	20160523
Time	10.53
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	362
DW	48.400 usec
DE	6.00 usec
TE	295.5 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ======	
NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300129 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



4g

GYS-3-111
C13CPD CDCl3

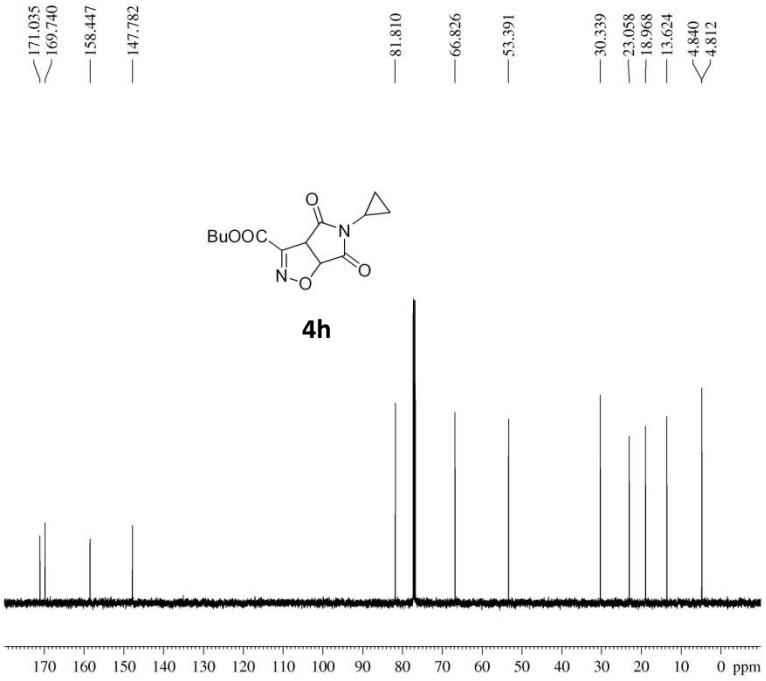
NAME	XB20160525
EXPNO	13
PROCNO	1
Date_	20160526
Time	6.10
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	2087
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	114
DW	16.650 usec
DE	6.00 usec
TE	297.9 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.50 usec
PL1	-0.50 dB
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	0.20



4h

GYS-3-115
PROTON CDCl3

NAME	XB20160427
EXPNO	5
PROCNO	1
Date_	20160427
Time	10.02
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	101.6
DW	48.400 usec
DE	6.00 usec
TE	294.8 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300129 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



4h

GYS−3−115
C13CPD CDCl3

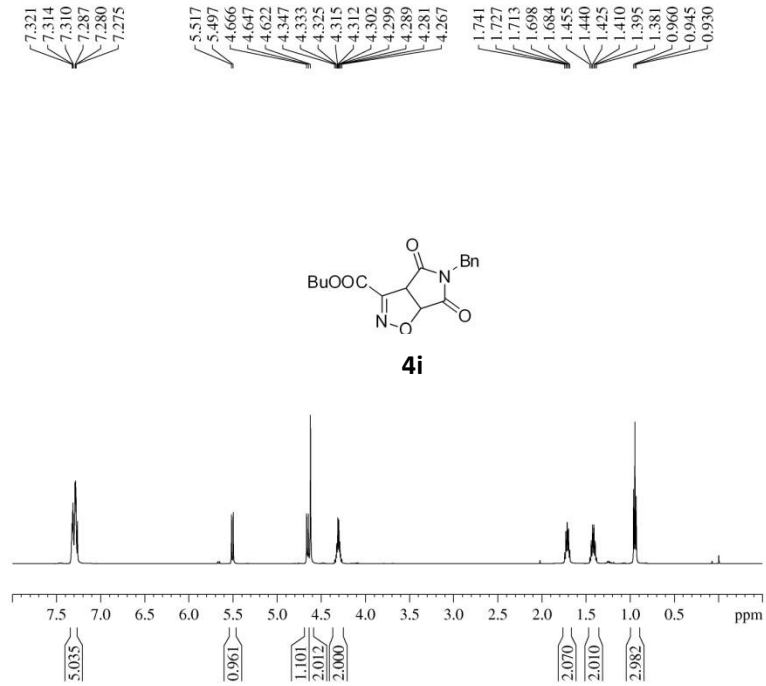
NAME	XB20160427
EXPNO	10
PROCNO	1
Date_	20160428
Time	5.25
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	128
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	101.6
DW	16.650 usec
DE	6.00 usec
TE	296.2 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

======== CHANNEL f1 ========

NUC1	13C
P1	9.50 usec
PL1	−0.50 dB
SFO1	125.7703643 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



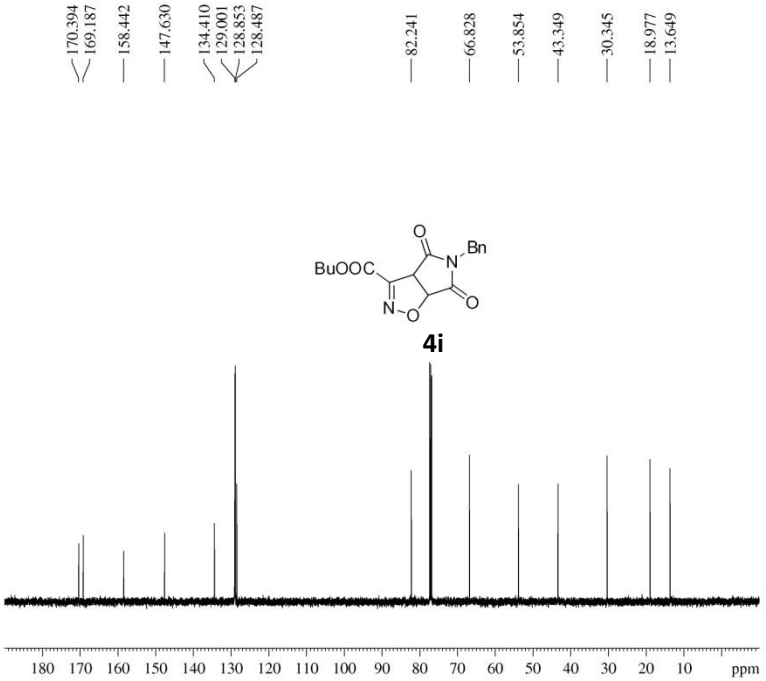
4i

GYS−3−113
PROTON CDCl3

NAME	XB20160427
EXPNO	3
PROCNO	1
Date_	20160427
Time	9.50
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	90.5
DW	48.400 usec
DE	6.00 usec
TE	294.9 K
D1	1.00000000 sec
TD0	1

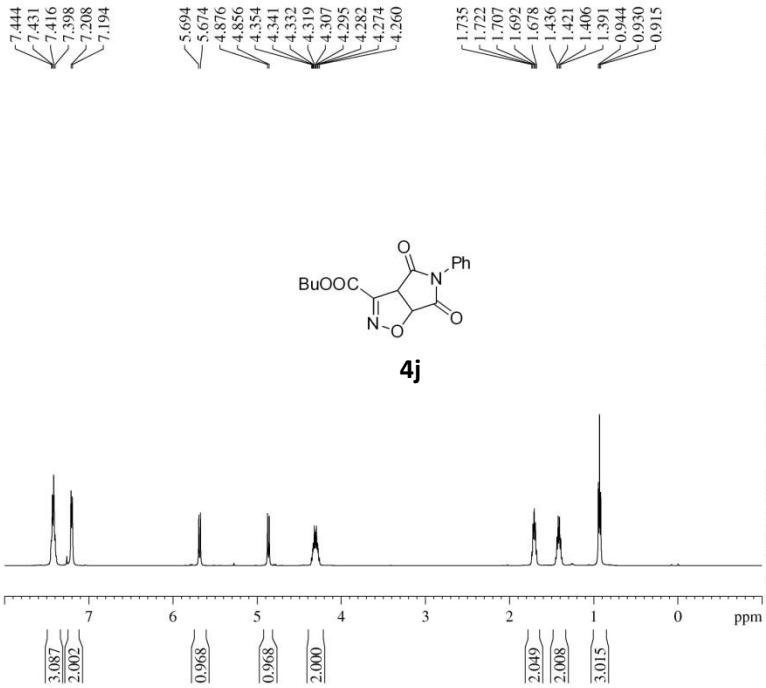
======== CHANNEL f1 ========

NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300126 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



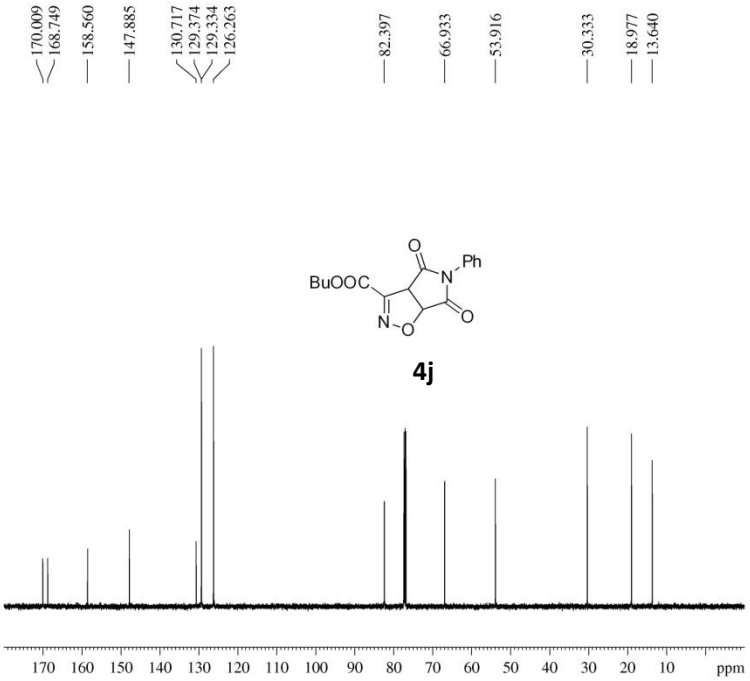
GYS−3−113
C13CPD CDCl3

NAME	XB20160427
EXPNO	8
PROCNO	1
Date_	20160428
Time	5.01
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	64
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	101.6
DW	16.650 usec
DE	6.00 usec
TE	296.2 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.50 usec
PL1	−0.50 dB
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



GYS−2−159
PROTON CDCl3

NAME	XB20160122
EXPNO	20
PROCNO	1
Date_	20160122
Time	14.52
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	64
DW	48.400 usec
DE	6.00 usec
TE	295.6 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300120 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



GYS−2−159
C13CPD CDCl3

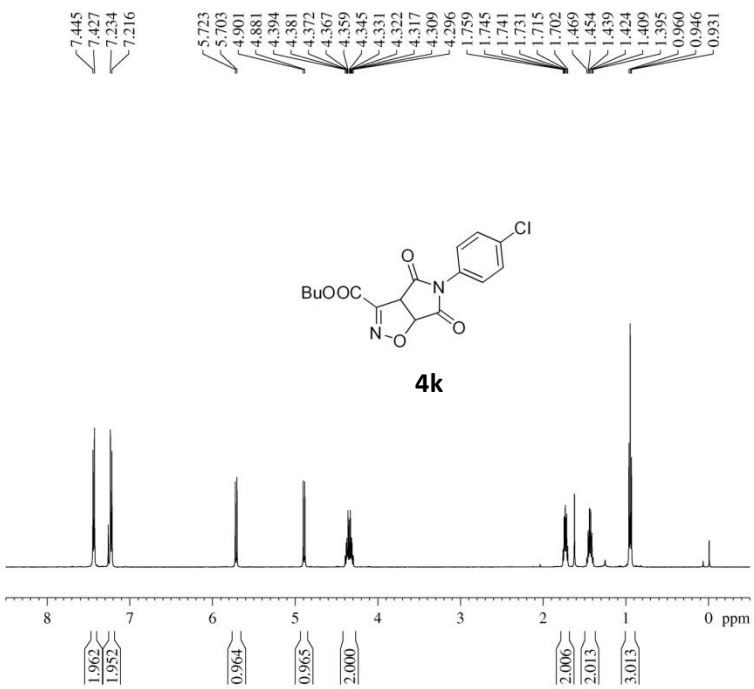
NAME	XB20160122
EXPNO	30
PROCNO	1
Date_	20160122
Time	15.57
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	128
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	181
DW	16.650 usec
DE	6.00 usec
TE	296.4 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

======== CHANNEL f1 ========

NUC1	13C
P1	9.50 usec
PL1	−0.50 dB
SFO1	125.7703643 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

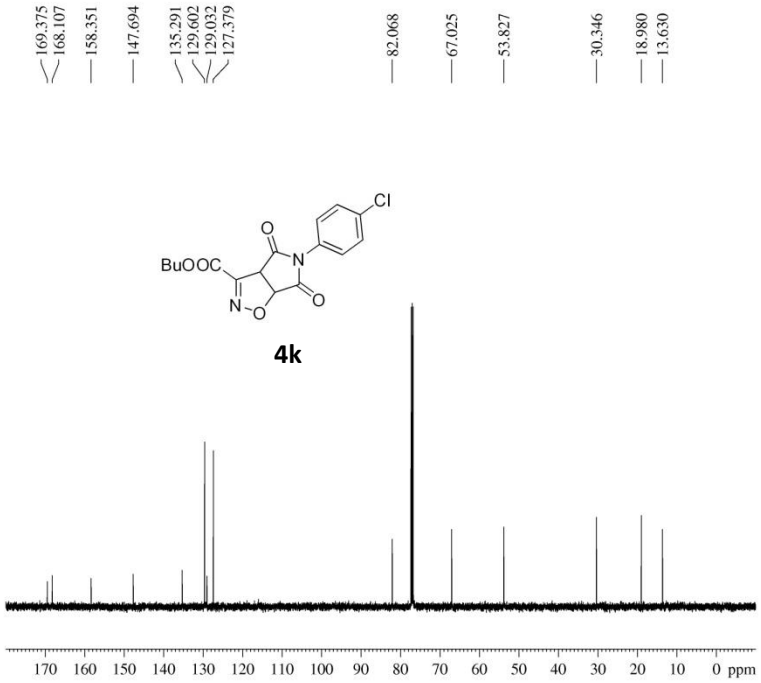


GYS−3−107
PROTON CDCl3

NAME	XB20160510
EXPNO	21
PROCNO	1
Date_	20160510
Time	15.29
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	203.2
DW	48.400 usec
DE	6.00 usec
TE	294.9 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========

NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300129 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



GYS−3−107
C13CPD CDCl3

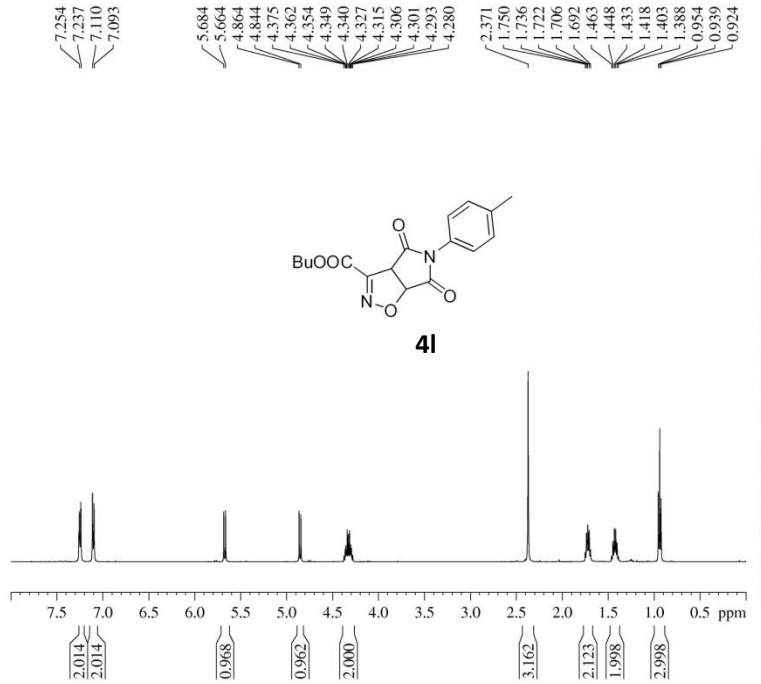
NAME	XB20160511
EXPNO	19
PROCNO	1
Date_	20160511
Time	22.50
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	128
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	143.7
DW	16.650 usec
DE	6.00 usec
TE	296.8 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

======== CHANNEL f1 ========

NUC1	13C
P1	9.50 usec
PL1	−0.50 dB
SFO1	125.7703643 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

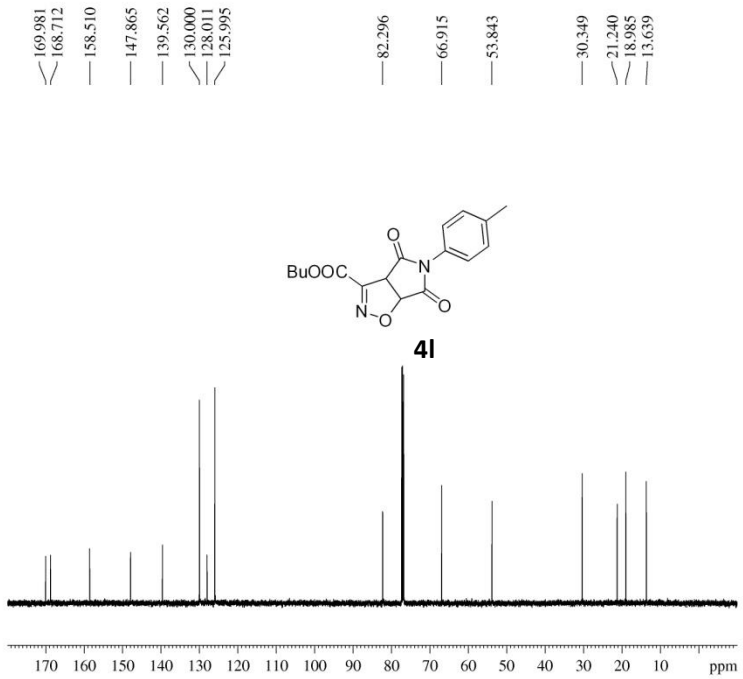


GYS−3−114
PROTON CDCl3

NAME	XB20160427
EXPNO	4
PROCNO	1
Date_	20160427
Time	9.56
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	101.6
DW	48.400 usec
DE	6.00 usec
TE	295.2 K
D1	1.00000000 sec
TD0	1

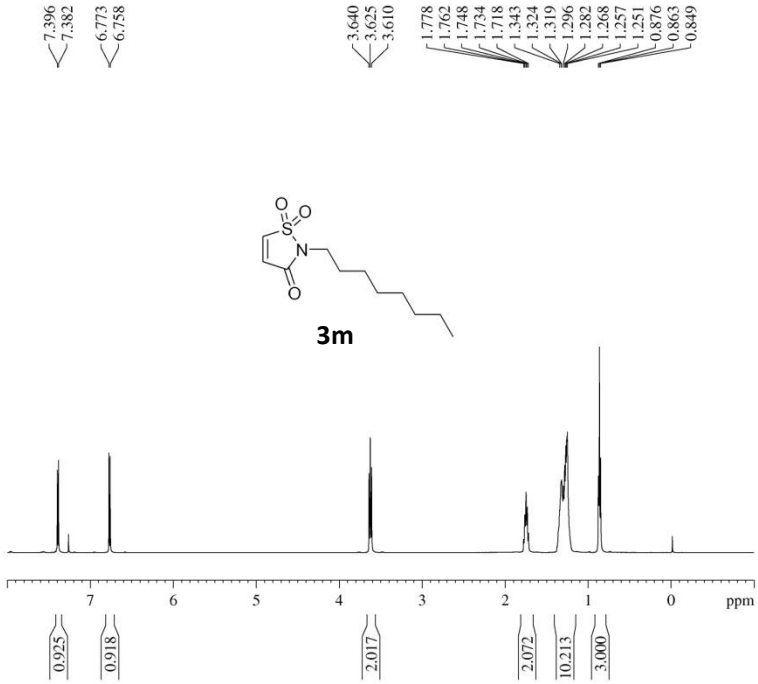
======== CHANNEL f1 ========

NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300129 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



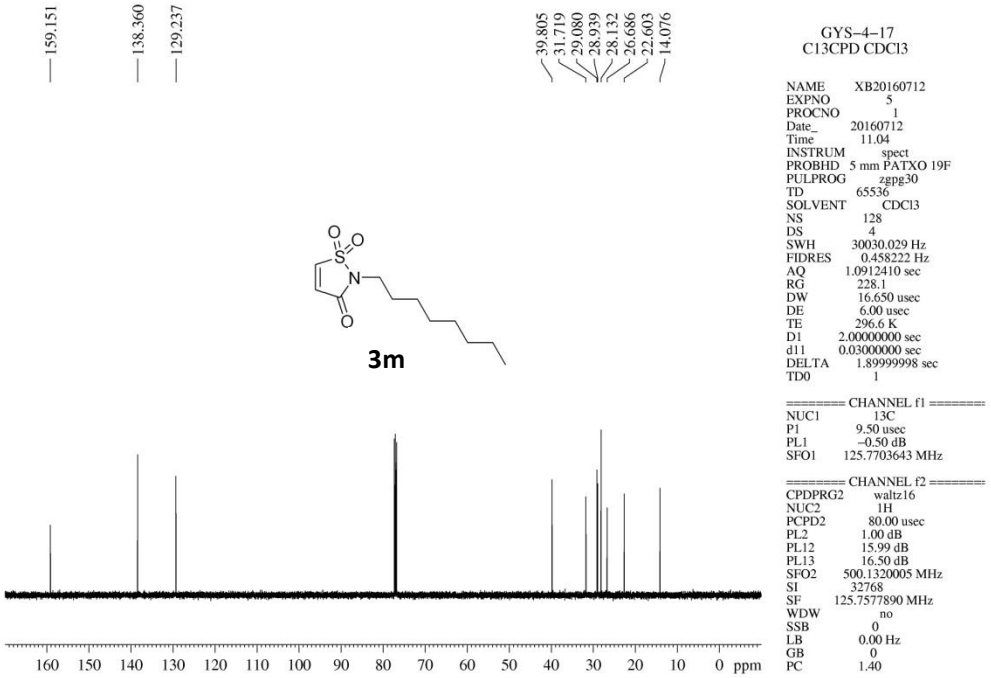
GYS-3-114
C13CPD CDCl3

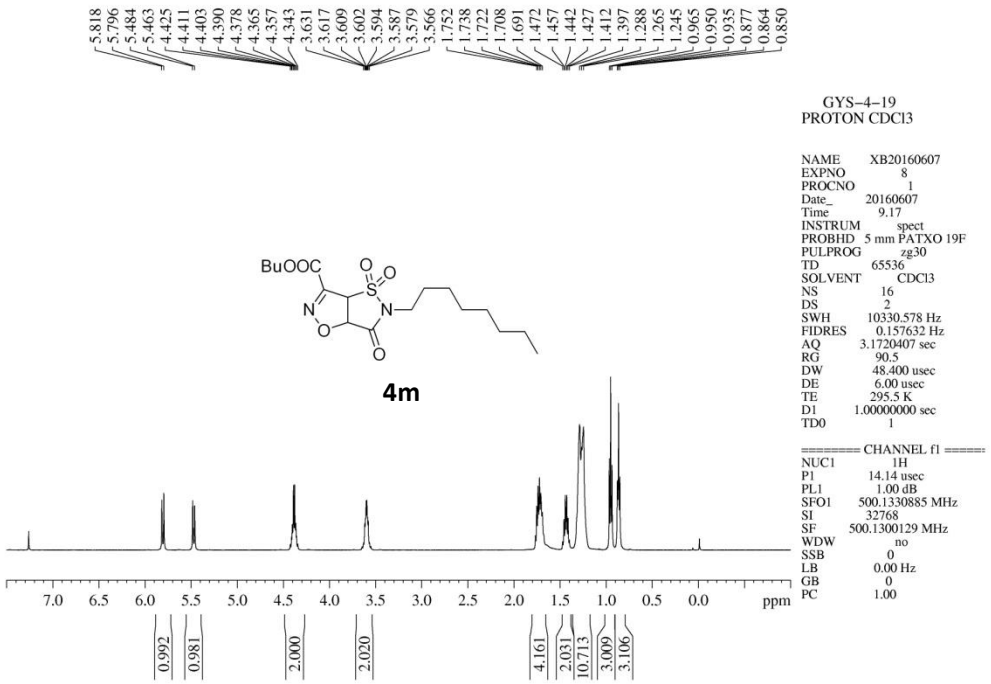
NAME	XB20160427
EXPNO	9
PROCNO	1
Date_	20160428
Time	5.13
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	128
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	101.6
DW	16.650 usec
DE	6.00 usec
TE	296.8 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.50 usec
PL1	−0.50 dB
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

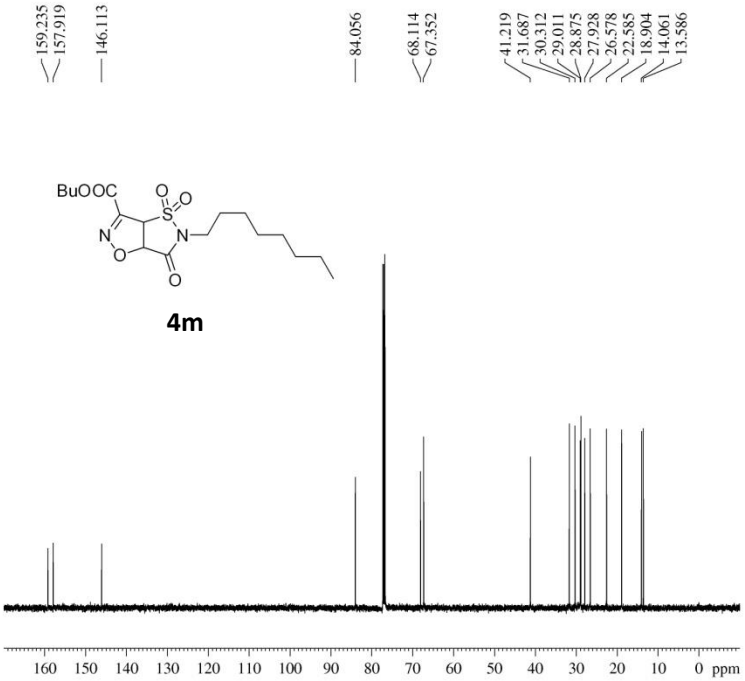


GYS-4-17
PROTON CDCl3

NAME	XB20160712
EXPNO	2
PROCNO	1
Date_	20160712
Time	10.41
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	71.8
DW	48.400 usec
DE	6.00 usec
TE	295.4 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.24 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300129 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

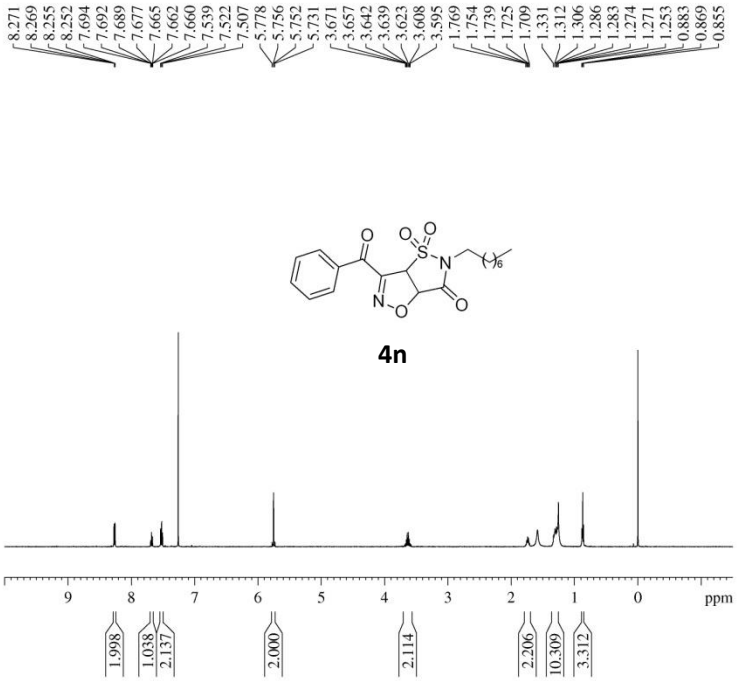

159.151
138.360
129.237
39.805
31.719
29.080
28.939
28.132
26.686
22.603
14.076
3m
GYS-4-17
C13CPD CDCl3
NAME XB20160712
EXPNO 5
PROCNO 1
Date_ 20160712
Time 11.04
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 228.1
DW 16.650 usec
DE 6.00 usec
TE 296.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 9.50 usec
PL1 -0.50 dB
SFO1 125.7703643 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 15.99 dB
PL13 16.50 dB
SFO2 500.1320005 MHz
SI 32768
SF 125.7577890 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


5.818
5.796
5.484
5.463
4.425
4.411
4.403
4.390
4.378
4.365
4.357
4.343
3.631
3.617
3.609
3.602
3.594
3.587
3.579
3.566
1.752
1.738
1.722
1.708
1.691
1.472
1.457
1.442
1.427
1.412
1.397
1.288
1.265
1.245
0.965
0.950
0.935
0.877
0.864
0.850
BuOOC
4m
GYS-4-19
PROTON CDCl3
NAME XB20160607
EXPNO 8
PROCNO 1
Date_ 20160607
Time 9.17
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 90.5
DW 48.400 usec
DE 6.00 usec
TE 295.5 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 14.14 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300129 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.992
0.981
2.000
2.020
4.161
2.031
10.713
3.009
3.106



GYS-4-19
C13CPD CDCl3

NAME	XB20160608
EXPNO	4
PROCNO	1
Date_	20160608
Time	12.36
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	420
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	114
DW	16.650 usec
DE	6.00 usec
TE	296.5 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.50 usec
PL1	-0.50 dB
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



GYS-4-135
PROTON CDCl3

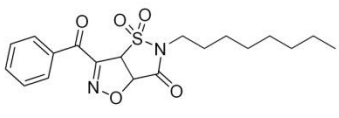
NAME	XB20161223
EXPNO	1
PROCNO	1
Date_	20161223
Time	10.32
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	456.1
DW	48.400 usec
DE	6.00 usec
TE	296.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ======	
NUC1	1H
P1	14.24 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300129 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

GYS-4-135
C13CPD CDCl3

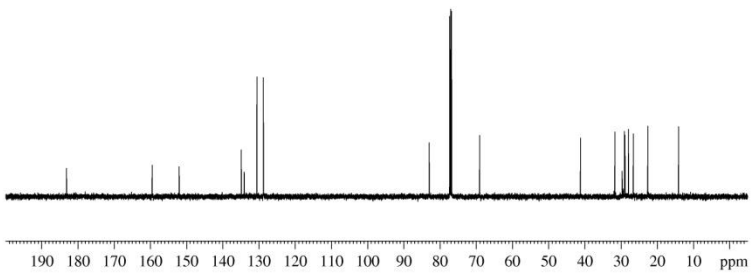
NAME XB20161130
EXPNO 20
PROCNO 1
Date_ 20161130
Time 15.54
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 456.1
DW 16.650 usec
DE 6.00 usec
TE 295.7 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.50 usec
PL1 -0.50 dB
SFO1 125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 15.99 dB
PL13 16.50 dB
SFO2 500.1320005 MHz
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



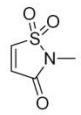
4n



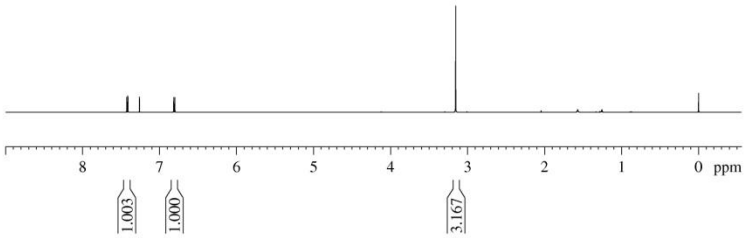
GYS-4-144
PROTON CDCl3

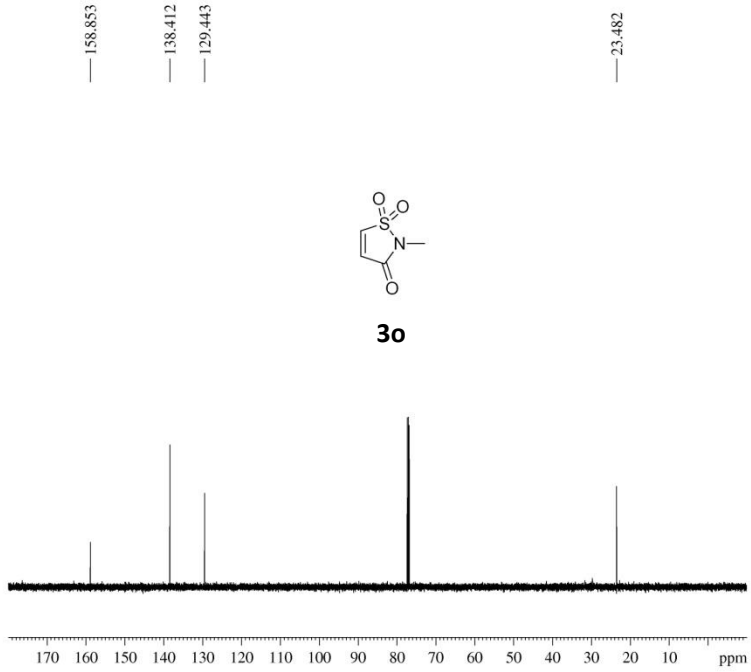
NAME XB20161213
EXPNO 1
PROCNO 1
Date_ 20161213
Time 9.34
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 456.1
DW 48.400 usec
DE 6.00 usec
TE 296.3 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.24 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300129 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



3o





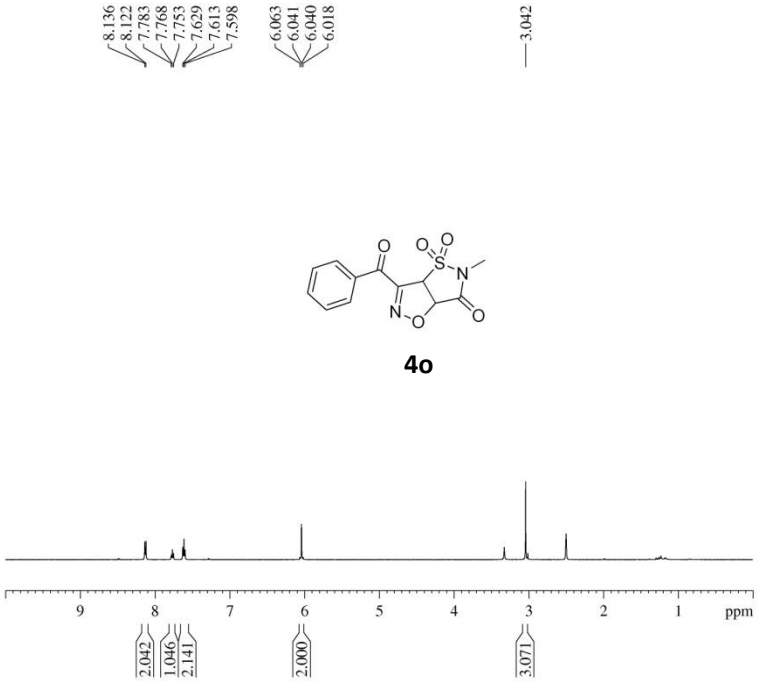
GYS-4-144
C13CPD CDCl3

NAME XB20161215
EXPNO 20
PROCNO 1
Date_ 20161215
Time 17.00
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 512
DW 16.650 usec
DE 6.00 usec
TE 297.1 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.50 usec
PL1 -0.50 dB
SFO1 125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 15.99 dB
PL13 16.50 dB
SFO2 500.1320005 MHz
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 0.50

3o

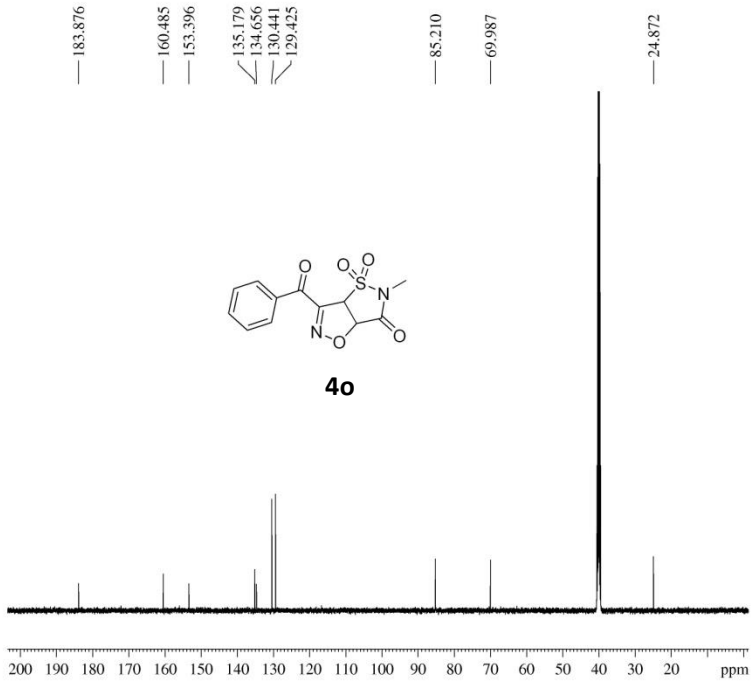


GYS-4-147
PROTON DMSO

NAME XB20161215
EXPNO 14
PROCNO 1
Date_ 20161215
Time 13.44
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 362
DW 48.400 usec
DE 6.00 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.24 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300039 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

4o



GYS-4-147
C13CPD DMSO

NAME	XB20161215
EXPNO	22
PROCNO	1
Date_	20161215
Time	22.05
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	DMSO
NS	1024
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	724.1
DW	16.650 usec
DE	6.00 usec
TE	297.6 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

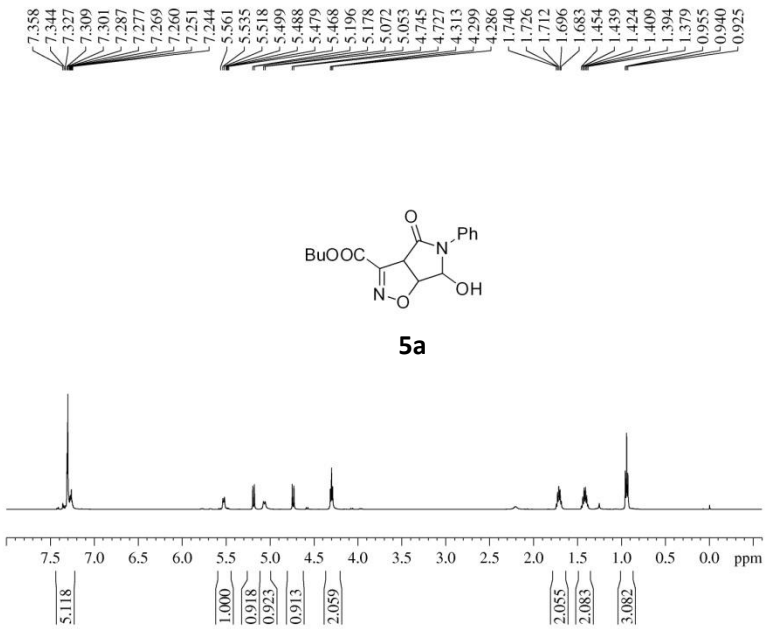
======== CHANNEL f1 ========

NUC1	13C
P1	9.50 usec
PL1	-0.50 dB
SFO1	125.7703643 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	15.99 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

4o



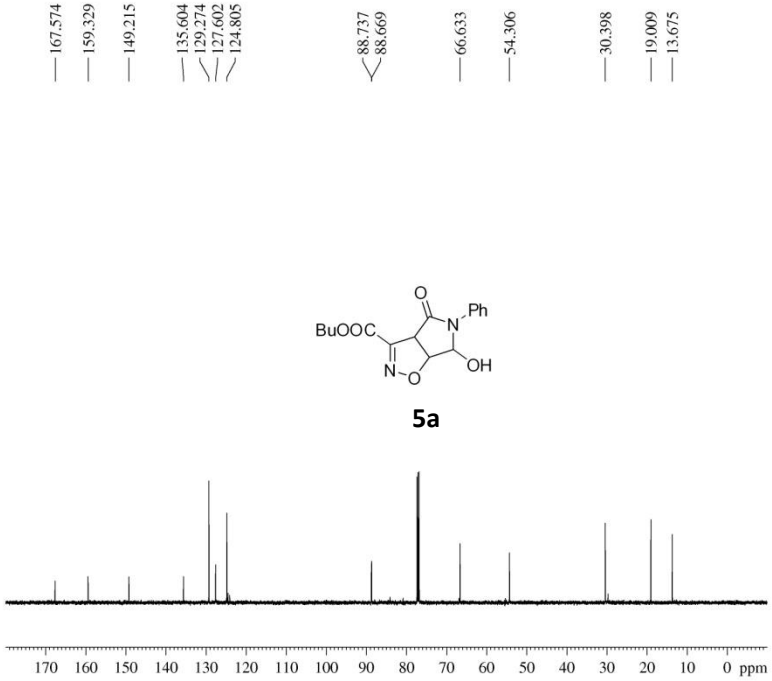
GYS-4-58
PROTON CDCl3

NAME	XB20160712
EXPNO	4
PROCNO	1
Date_	20160712
Time	10.52
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	80.6
DW	48.400 usec
DE	6.00 usec
TE	295.1 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========

NUC1	1H
P1	14.24 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300126 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

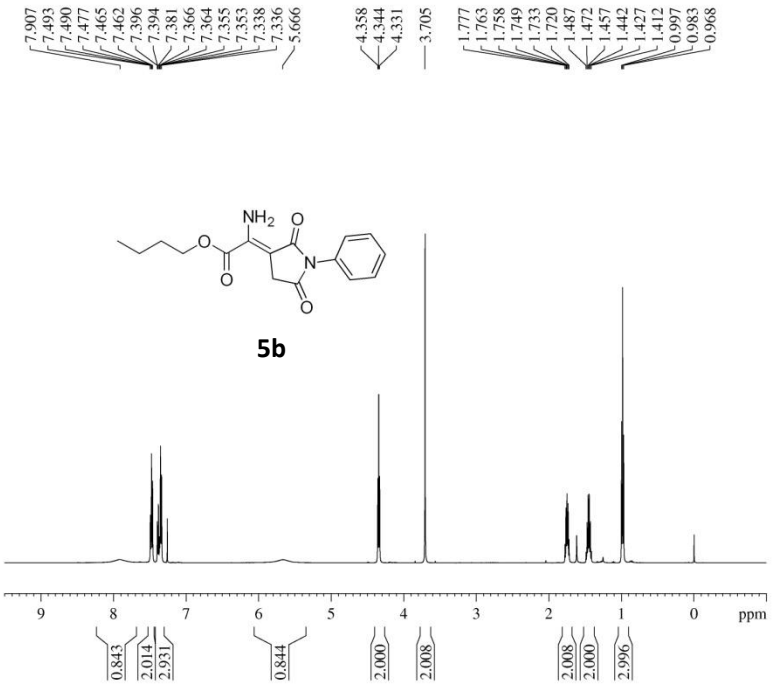
5a



5a

GYS-4-58
C13CPD CDCl3

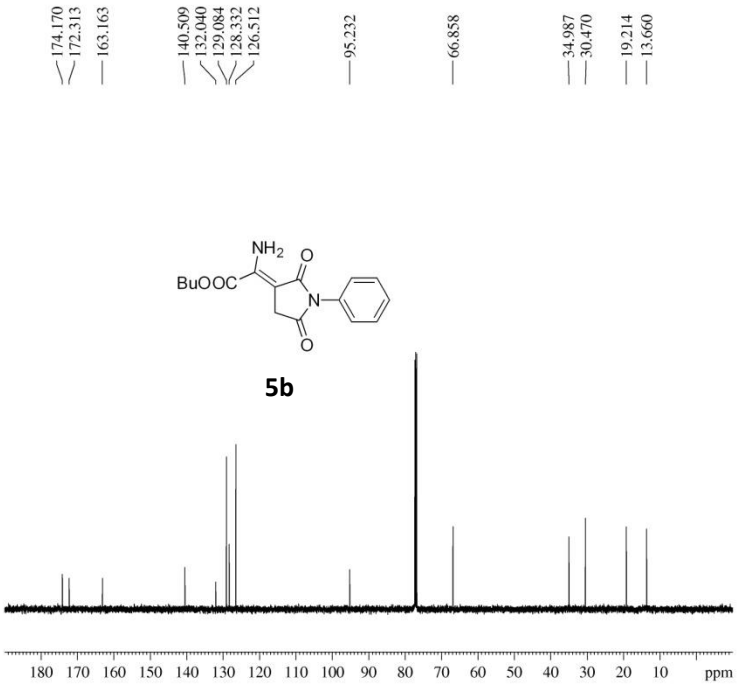
NAME	XB20160630
EXPNO	3
PROCNO	1
Date_	20160630
Time	10.26
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	128
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	101.6
DW	16.650 usec
DE	6.00 usec
TE	296.7 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.50 usec
PL1	-0.50 dB
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	15.99 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



5b

GYS-3-152
PROTON CDCl3

NAME	XB20160523
EXPNO	15
PROCNO	1
Date_	20160523
Time	11.04
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	181
DW	48.400 usec
DE	6.00 usec
TE	295.1 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300129 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



GYS−3−152
C13CPD CDCl3

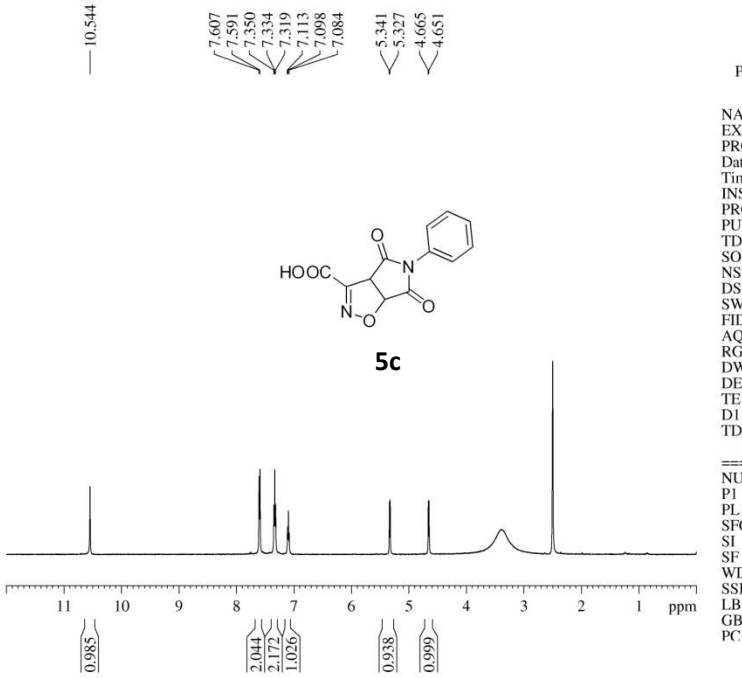
NAME	XB20160524
EXPNO	10
PROCNO	1
Date_	20160524
Time	10.55
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	128
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	181
DW	16.650 usec
DE	6.00 usec
TE	296.6 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

======== CHANNEL f1 ========

NUC1	13C
P1	9.50 usec
PL1	−0.50 dB
SFO1	125.7703643 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

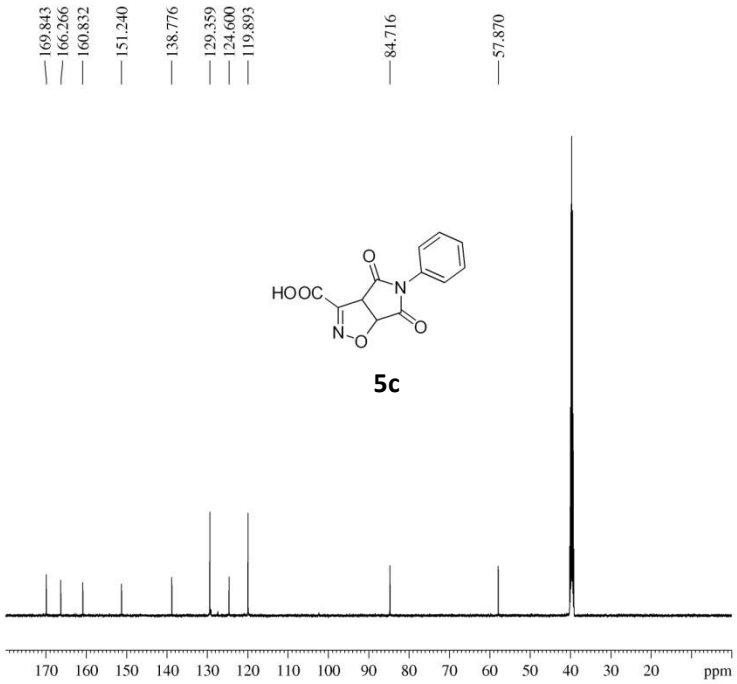


GYS−3−144
PROTON DMSO

NAME	XB20160523
EXPNO	18
PROCNO	1
Date_	20160523
Time	11.22
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	DMSO
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	287.4
DW	48.400 usec
DE	6.00 usec
TE	295.5 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========

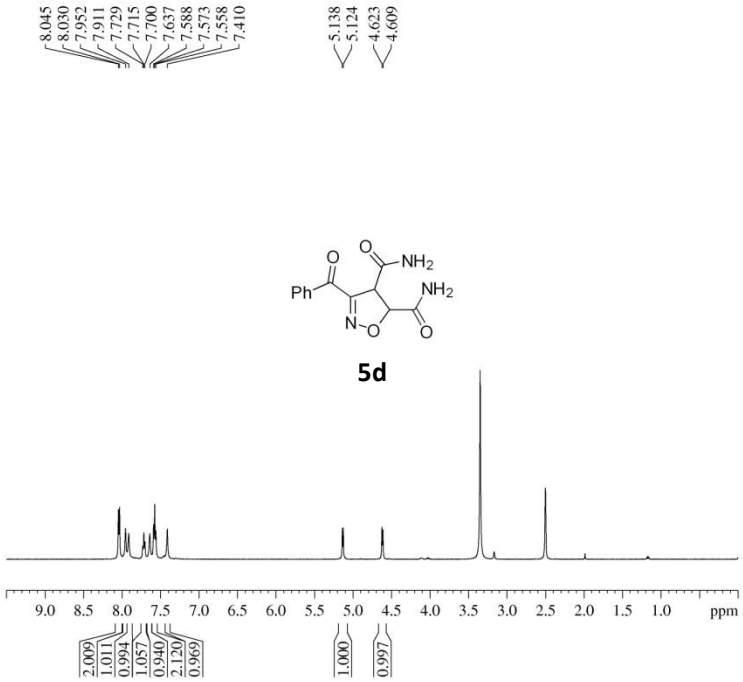
NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300038 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



5c

GYS–3–144
C13CPD DMSO

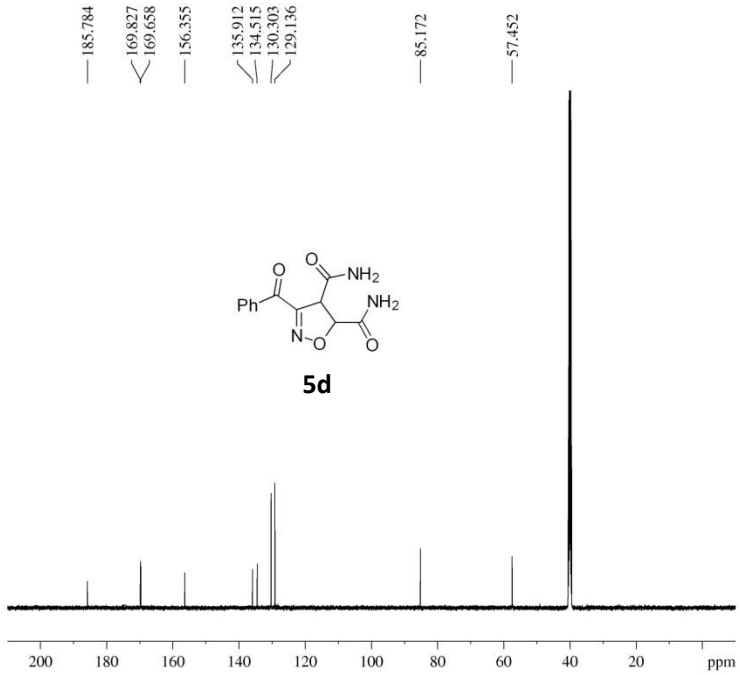
NAME	XB20160524
EXPNO	8
PROCNO	1
Date_	20160524
Time	10.07
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	DMSO
NS	512
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	322.5
DW	16.650 usec
DE	6.00 usec
TE	296.2 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.50 usec
PL1	−0.50 dB
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



5d

GYS–4–114
PROTON DMSO

NAME	XB20161027
EXPNO	14
PROCNO	1
Date_	20161027
Time	14.21
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	DMSO
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	256
DW	48.400 usec
DE	6.00 usec
TE	295.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.24 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300034 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



5d

GYS-4-114
C13CPD DMSO

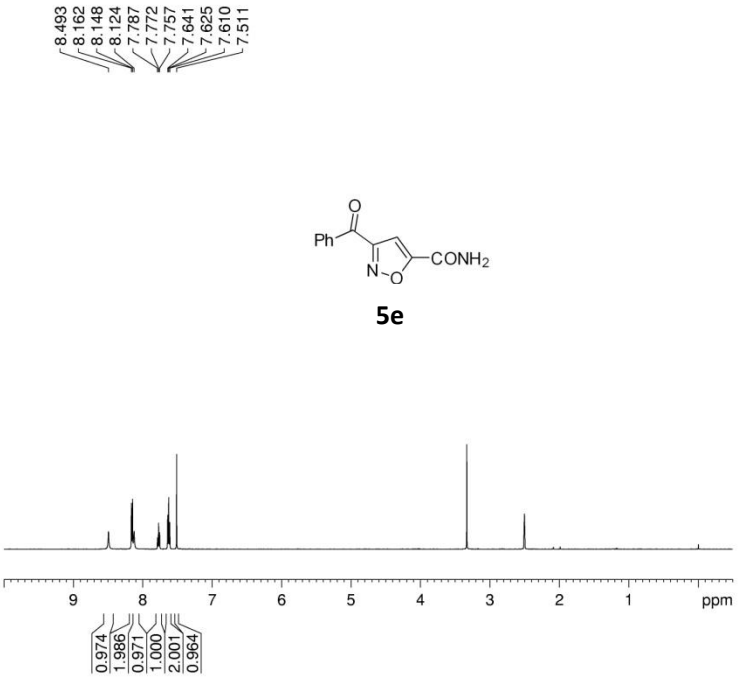
NAME	XB20161027
EXPNO	23
PROCNO	1
Date_	20161027
Time	22.36
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	DMSO
NS	2100
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	406.4
DW	16.650 usec
DE	6.00 usec
TE	297.5 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

======== CHANNEL f1 ========

NUC1	13C
P1	9.50 usec
PL1	-0.50 dB
SFO1	125.7703643 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	15.99 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



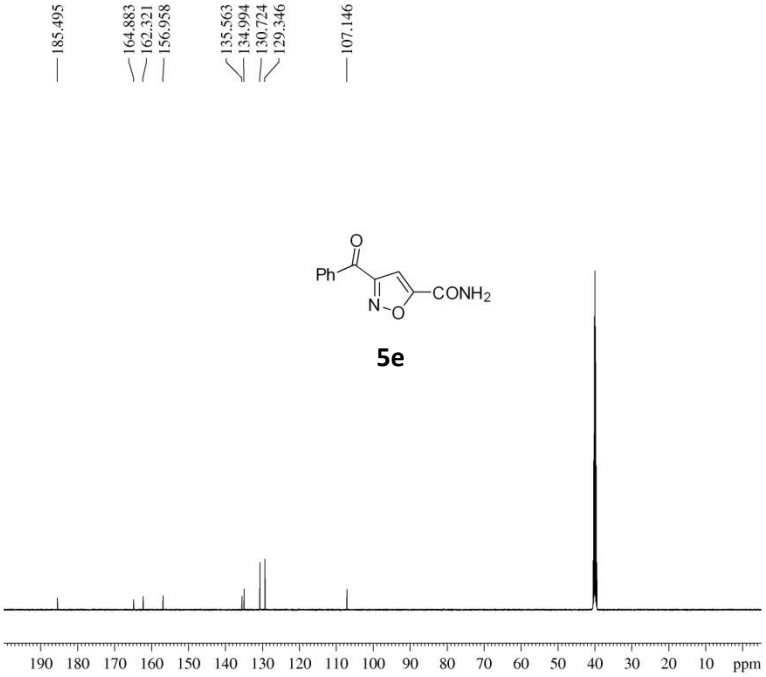
5e

GYS-5-22
PROTON DMSO

NAME	XB20170315
EXPNO	15
PROCNO	1
Date_	20170315
Time	15.23
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	DMSO
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	256
DW	48.400 usec
DE	6.00 usec
TE	296.5 K
D1	1.00000000 sec
TD0	1

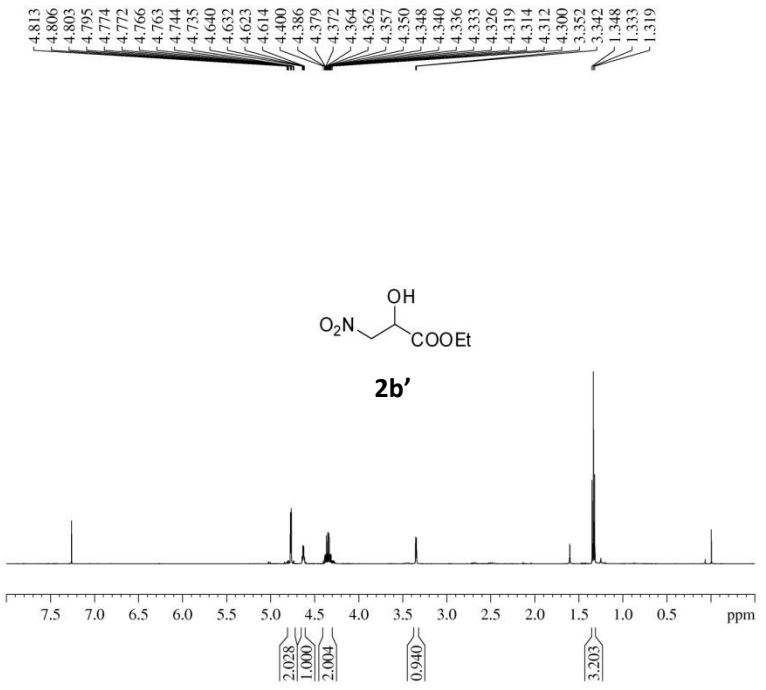
======== CHANNEL f1 ========

NUC1	1H
P1	14.24 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300039 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



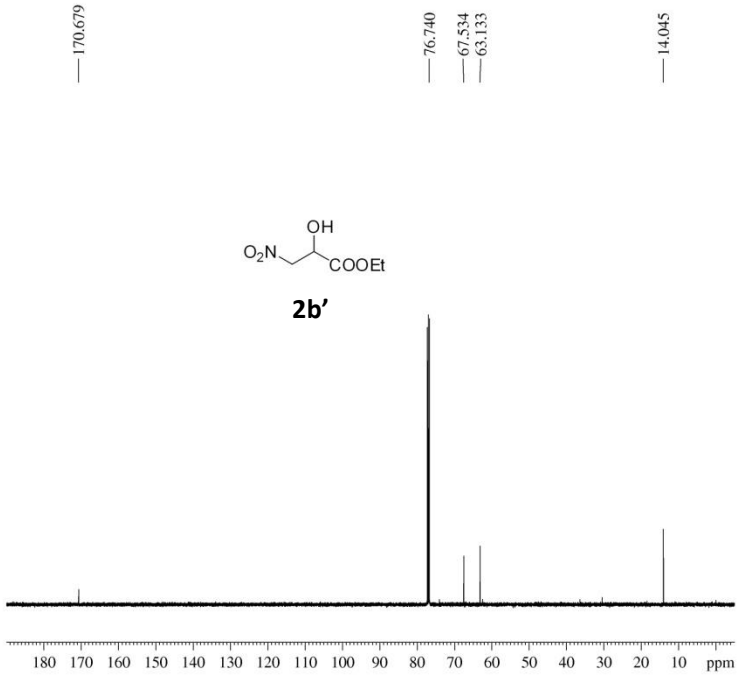
GYS−5−22
C13CPD DMSO

NAME	XB20170315
EXPNO	18
PROCNO	1
Date_	20170315
Time	21.09
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	DMSO
NS	2048
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	1824.6
DW	16.650 usec
DE	6.00 usec
TE	298.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.50 usec
PL1	−0.50 dB
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	15.99 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



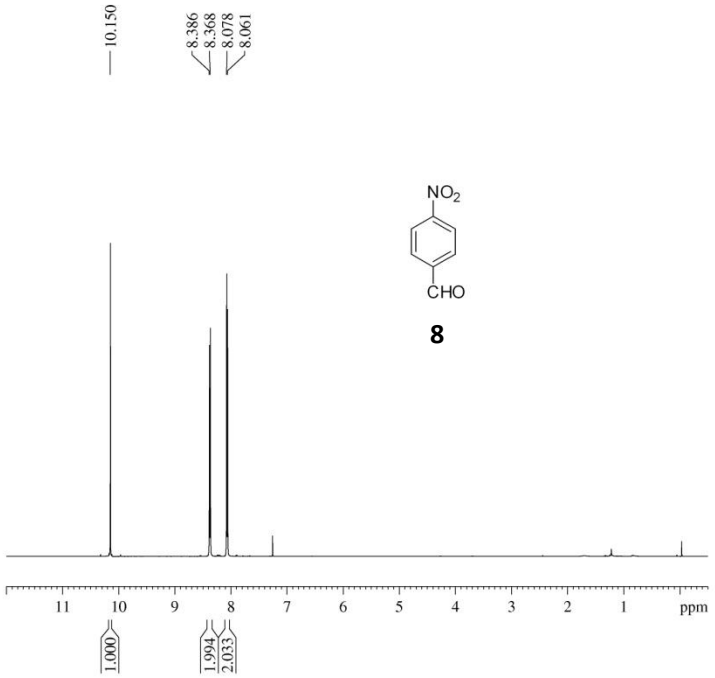
GYS−4−112−2
PROTON CDCl3

NAME	XB20161103
EXPNO	10
PROCNO	1
Date_	20161103
Time	15.15
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	287.4
DW	48.400 usec
DE	6.00 usec
TE	296.4 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.24 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300129 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



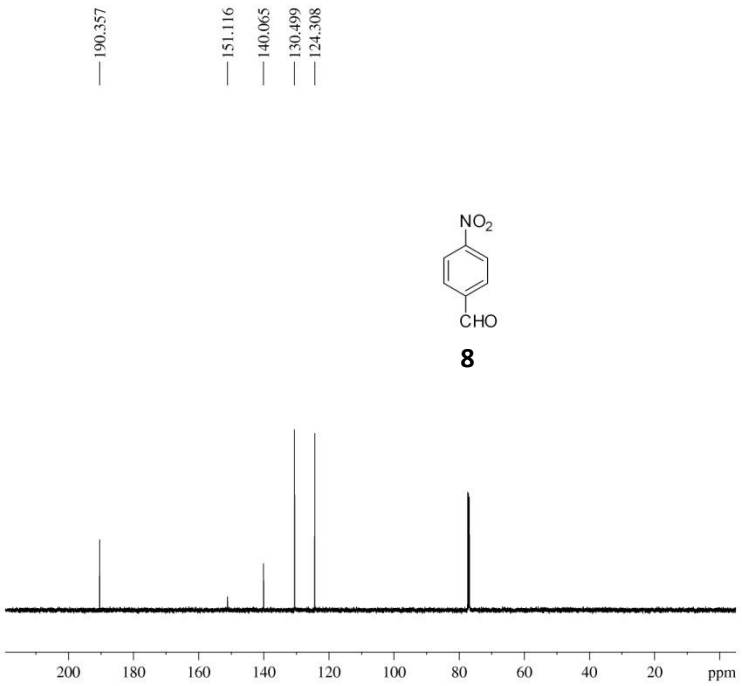
GYS-4-112-2
C13CPD CDCl3

NAME	XB20161103
EXPNO	18
PROCNO	1
Date_	20161103
Time	23.02
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	406.4
DW	16.650 usec
DE	6.00 usec
TE	298.7 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.50 usec
PL1	-0.50 dB
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	15.99 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.40



GYS-4-142-2
PROTON CDCl3

NAME	XB20161230
EXPNO	13
PROCNO	1
Date_	20161230
Time	10.45
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	161.3
DW	48.400 usec
DE	6.00 usec
TE	296.1 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 =======	
NUC1	1H
P1	14.24 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300126 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



GYS-4-142-2
C13CPD CDCl3

NAME	XB20161230
EXPNO	36
PROCNO	1
Date_	20161231
Time	10.16
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	32
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	724.1
DW	16.650 usec
DE	6.00 usec
TE	297.8 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ======	
NUC1	13C
P1	9.50 usec
PL1	-0.50 dB
SFO1	125.7703643 MHz
======== CHANNEL f2 ======	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	15.99 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40