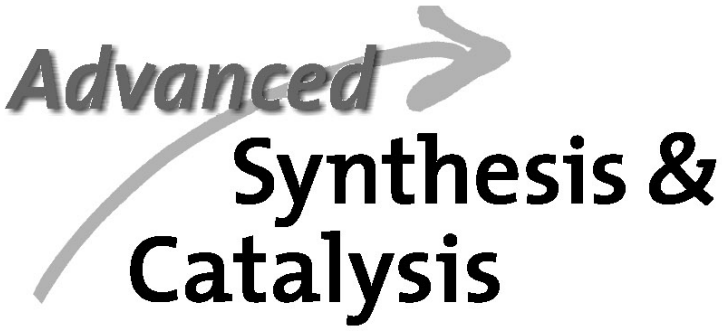

Supporting Information

Supporting Information for:

Combining Transition Metal Catalysis with Radical Chemistry: Dramatic Acceleration of Palladium-Catalyzed C–H Arylation with Diaryliodonium Salts

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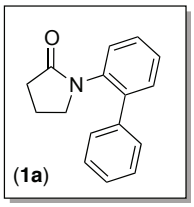
I. General Procedures

NMR spectra were obtained on a Varian vnmrs 700 (699.76 MHz for ^{1}H; 175.95 MHz for ^{13}C; 658.43 for ^{19}F), Varian vnmrs 500 (500.10 MHz for ^{1}H; 125.75 MHz for ^{13}C, 470.56 MHz for ^{19}F), Varian Inova 500 (499.90 MHz for ^{1}H; 125.70 MHz for ^{13}C), or a Varian MR400 (400.52 MHz for ^{1}H; 100.71 for ^{13}C, 376.87 MHz for ^{19}F) spectrometer. ^{1}H NMR chemical shifts are reported in parts per million (ppm) relative to TMS, with the residual solvent peak used as an internal reference. Multiplicities are reported as follows: singlet (s), doublet (d), doublet of doublets (dd), doublet of doublets of doublets (ddd), doublet of triplets (dt), triplet (t), triplet of doublets (td), triplet of triplets (tt), quartet (q), quintet (quin), multiplet (m), and broad resonance (br). IR spectra were obtained on a Perkin-Elmer Spectrum BX FT-IR spectrometer. Melting points were determined with a Mel-Temp 3.0, a Laboratory Devices Inc, USA instrument, and are uncorrected. HRMS data were obtained on a Micromass AutoSpec Ultima Magnetic Sector mass spectrometer. Gas chromatography was carried out on a Shimadzu 17A using a Restek Rtx®-5 (Crossbond 5% diphenyl – 95% dimethyl polysiloxane; 15 m, 0.25 mm ID, 0.25 μm df) column. GC calibrated yields are reported relative to hexadecane as an internal standard.

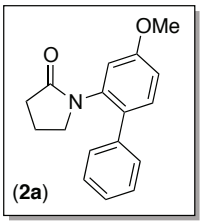
Materials and Methods: Substrates **2**[1] and **8**[2] were prepared according to literature procedures. Substrate **9** was prepared by a palladium-catalyzed Suzuki coupling between 2-methoxyboronic acid and 2-bromopyridine. Oxime ethers **10** and **11** were prepared by the reaction of the corresponding ketones with $MeONH_2$•HCl in pyridine.[3] The remaining substrates were obtained from Aldrich (**1**, **5**, and **7**), Alfa Aesar (**3** and **4**), or Acros (**6**) and were used as received. $[Ph_2I]BF_4$ and [Mes–I–Ph]BF_4 were prepared by the reaction of $PhI(OAc)_2$ or $MesI(OAc)_2$ with $PhB(OH)_2$ in the presence of BF_3•Et_2O.[4] $[Ph_2I]OTf$ and $[Mes_2I]OTf$ were prepared by the reaction of iodobenzene or iodomesitylene with *m*CPBA and benzene or mesitylene in the presence of TfOH.[5] Unsymmetrical [Ar–I–Ph]BF_4 salts were prepared by the reaction of an aryl iodide with *m*-CPBA and $PhB(OH)_2$ in the presence of BF_3•Et_2O.[6] Symmetrical $[Ar_2I]BF_4$ salts were prepared by the reaction of an aryl iodide with *m*-CPBA and the corresponding arylboronic acid in the presence of BF_3•Et_2O.[6] $Pd(OAc)_2$, obtained from Pressure Chemical, and $Pd(NO_3)_2$ and $Ru(bpy)_3Cl_2$•$6H_2O$, obtained from Strem, were used as received. $Ir(ppy)_3$[7] and $Ir(ppy)_2(dtbbpy)PF_6$[8] were prepared according to literature procedures. Solvents were obtained from Fisher Chemical and used without further purification. Flash chromatography was performed on EM Science silica gel 60 (0.040–0.063 mm particle size, 230–400 mesh) and thin layer chromatography was performed on Merck TLC plates pre-coated with silica gel 60 F_{254}.

II. Synthesis and Characterization of Products in Table 2

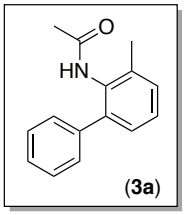
General Procedure: Substrate (1 equiv), $[Ph_2I]BF_4$ or $[Ph_2I]OTf$ (2 equiv), $Ir(ppy)_2(dtbbpy)PF_6$ (0.05 equiv), and $Pd(NO_3)_2•2H_2O$ (0.10 equiv) were combined in MeOH in a 4 mL scintillation vial. For substrates containing *N*-acetyl moieties (noted below), MgO (1 equiv) was also included and appeared to help prevent substrate and/or product degradation. The reaction mixture was cooled in an ice bath (to prevent evaporation) and sparged with N_2 using a submerged needle for 10 min, and the vial was then immediately sealed with a Teflon-lined cap. The vial was placed on a stir plate with two 26 W compact fluorescent light bulbs (one on either side of the vial about 5–8 cm away), and the reaction mixture was allowed to stir at room temperature for 15 h. The reaction mixture was diluted with EtOAc (50 mL) and washed with 10% aqueous Na_2SO_3 (2 x 25 mL) and brine (1 x 25 mL). The combined aqueous layers were extracted with EtOAc (3 x 10 mL), and the organic layers were then combined, dried over $MgSO_4$, filtered, concentrated, and purified by column chromatography on silica gel.



Pyrrolidinone 1a. The general procedure was followed utilizing substrate **1** (80.6 mg, 0.50 mmol, 1.0 equiv), $[Ph_2I]OTf$ (430 mg, 1.00 mmol, 2 equiv), $Ir(ppy)_2(dtbbpy)PF_6$ (22.8 mg, 0.025 mmol, 0.05 equiv), $Pd(NO_3)_2•2H_2O$ (13.3 mg, 0.05 mmol, 0.10 equiv), and MeOH (2.5 mL). Product **1a** was obtained as a pale yellow oil (96.3 mg, 81% yield, R_f = 0.17 in 20% hexanes/80% Et_2O). ^1H and ^{13}C NMR data matched those reported in the literature.[9]

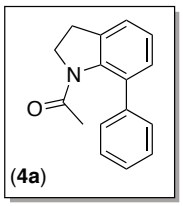


Pyrrolidinone 2a. The general procedure was followed utilizing substrate **2** (47.8 mg, 0.25 mmol, 1.0 equiv), $[Ph_2I]OTf$ (215 mg, 0.50 mmol, 2 equiv), $Ir(ppy)_2(dtbbpy)PF_6$ (11.4 mg, 0.0125 mmol, 0.05 equiv), $Pd(NO_3)_2•2H_2O$ (6.7 mg, 0.025 mmol, 0.10 equiv), and MeOH (1.8 mL). Product **2a** was obtained as a pale yellow solid [62.5 mg, 94% yield, R_f = 0.10 in 20% hexanes/80% Et_2O, mp = 72.9-74.7 ºC (lit.[11] 61–64 ºC)]. ^1H and ^{13}C NMR data matched those reported in the literature.[9]

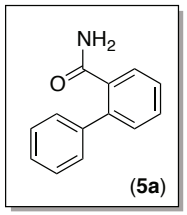


Acetanilide 3a. The general procedure was followed utilizing substrate **3** (37.3 mg, 0.25 mmol, 1.0 equiv), $[Ph_2I]BF_4$ (184 mg, 0.50 mmol, 2 equiv), $Ir(ppy)_2(dtbbpy)PF_6$ (11.4 mg, 0.0125 mmol, 0.05 equiv), $Pd(NO_3)_2•2H_2O$ (6.7 mg, 0.025 mmol, 0.10 equiv), and MeOH (1.25 mL), with the addition of MgO (10.1 mg, 0.25 mmol, 1.0 equiv). Product **3a** was obtained as a pale yellow solid [40.6 mg, 72% yield, R_f = 0.17 in 30% hexanes/70% Et_2O, mp = 134.5-136.0 ºC (lit. 139-140 ºC)].[10] ^1H NMR (700 MHz, CD_3CN): δ

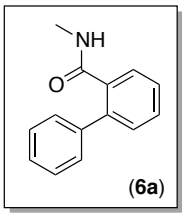
7.64 (br s, 1H); 7.42–7.39 (multiple peaks, 2H); 7.35 (t, J = 7.4 Hz, 1H); 7.32–7.31 (multiple peaks, 2H); 7.27 (d, J = 4.9 Hz, 2H); 7.17 (t, J = 4.9 Hz, 1H); 2.23 (s, 3H); 1.85 (s, 3H). $^{13}C\{^1H\}$ NMR (176 MHz, CD_3CN): δ 170.04; 141.30; 140.97; 138.16; 134.58; 130.52; 129.67; 129.03; 128.72; 128.14; 128.05; 22.76; 18.54. IR (thin film, CH_2Cl_2) 3246, 3026, 2922, 1652, 1522 cm^{-1}. HRMS $[M+H]^+$ Calcd for $C_{15}H_{16}NO$: 226.1226; Found: 226.1234.



Acetylindoline 4a. The general procedure was followed utilizing substrate **4** (80.5 mg, 0.50 mmol, 1.0 equiv), $[Ph_2I]BF_4$ (368 mg, 1.00 mmol, 2 equiv), $Ir(ppy)_2(dtbbpy)PF_6$ (22.8 mg, 0.025 mmol, 0.05 equiv), $Pd(NO_3)_2•2H_2O$ (26.6 mg, 0.100 mmol, 0.20 equiv), and MeOH (2.5 mL), with the addition of MgO (20.2 mg, 0.50 mmol, 1.0 equiv). Product **4a** was obtained as a pale yellow solid [51.7 mg, 44% yield, R_f = 0.30 in 20% hexanes/80% Et_2O, mp = 116.3-117.8 ºC (lit. 117-119 ºC)].[11] ^1H and ^{13}C NMR data matched those reported in the literature.[11]

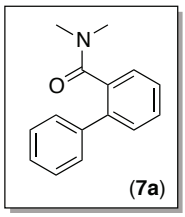


Benzamide 5a. The general procedure was followed utilizing substrate **5** (33.8 mg, 0.50 mmol, 1.0 equiv), $[Ph_2I]BF_4$ (368 mg, 1.00 mmol, 2 equiv), $Ir(ppy)_2(dtbbpy)PF_6$ (22.8 mg, 0.025 mmol, 0.05 equiv), $Pd(NO_3)_2•2H_2O$ (13.3 mg, 0.050 mmol, 0.10 equiv), and MeOH (2.5 mL). Product **5a** was obtained as a white solid (39 mg, 40% yield, R_f = 0.26 in 1:1:1 benzene:CH_2Cl_2:Et_2O, mp = 169.0-173.0 ºC). ^1H NMR (700 MHz, $CDCl_3$): δ 7.79 (d, J = 7.7 Hz, 1H), 7.50 (td, J = 7.7, 0.7 Hz, 1H), 7.46-7.42 (multiple peaks, 5H), 7.39 (m, 1H), 7.37 (dd, J = 7.7, 0.7 Hz, 1H), 5.62 (br s, 1H), 5.25 (br s, 1H). $^{13}C\{^1H\}$ NMR (176 MHz, $CDCl_3$): δ 171.21, 140.15, 139.80, 134.30, 130.54, 130.38, 129.08, 128.77, 128.69, 127.93, 127.62. IR (thin film, $CDCl_3$) 3383, 3178, 1653, 1643 cm^{-1}. HRMS $[M+H]^+$ Calcd for $C_{13}H_{12}NO$: 198.0913; Found: 198.0920.

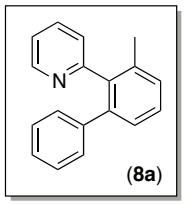


Benzamide 6a. The general procedure was followed utilizing substrate **6** (67.6 mg, 0.50 mmol, 1.0 equiv), $[Ph_2I]OTf$ (430 mg, 1.00 mmol, 2 equiv), $Ir(ppy)_2(dtbbpy)PF_6$ (22.8 mg, 0.025 mmol, 0.05 equiv), $Pd(NO_3)_2•2H_2O$ (13.3 mg, 0.05 mmol, 0.10 equiv), and MeOH (2.5 mL). Product **6a** was obtained as a pale yellow solid (56.7 mg, 54% yield, R_f = 0.27 in 20% hexanes/80% Et_2O, mp = 164.5-166.8 ºC). ^1H NMR (400 MHz, $CDCl_3$): δ 7.69 (dd, J = 7.6, 1.6 Hz, 1H), 7.47 (td, J = 7.6, 1.2 Hz, 1H), 7.42-7.35 (multiple peaks, 7H), 5.19 (br s, 1H), 2.67 (d, J = 4.8 Hz, 3H). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 170.24, 140.12, 139.29, 135.68, 130.11, 130.10, 128.82, 128.60, 128.58, 127.75, 127.59, 26.64. IR (thin film, $CDCl_3$)

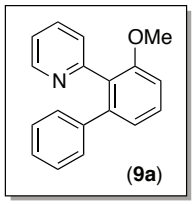
3286, 3060, 2936, 1636, 1540, 1313 cm^{-1}. HRMS $[M+H]^+$ Calcd for $C_{14}H_{14}NO$: 212.1070; Found: 212.1074.



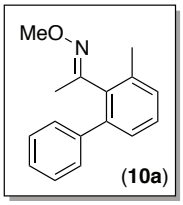
Benzamide 7a. The general procedure was followed utilizing substrate **7** (74.6 mg, 0.50 mmol, 1.0 equiv), $[Ph_2I]OTf$ (430 mg, 1.00 mmol, 2 equiv), $Ir(ppy)_2(dtbbpy)PF_6$ (22.8 mg, 0.025 mmol, 0.05 equiv), $Pd(NO_3)_2•2H_2O$ (13.3 mg, 0.05 mmol, 0.10 equiv), and MeOH (2.5 mL). Product **7a** was obtained as a yellow oil (9.8 mg, 9% yield, R_f = 0.27 in 20% hexanes/80% Et_2O). ^1H NMR (400 MHz, $CDCl_3$): δ 7.48-7.44 (multiple peaks, 3H), 7.42-7.32 (multiple peaks, 6H), 2.85 (s, 3H), 2.39 (s, 3H). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 171.33, 139.93, 138.67, 135.74, 129.30, 128.47, 128.36, 127.70, 127.58, 127.41, 37.94, 24.53. Two aromatic ^{13}C resonances are coincidentally overlapping. IR (thin film, $CDCl_3$) 3057, 2924, 1624, 1394 cm^{-1}. HRMS $[M+H]^+$ Calcd for $C_{15}H_{16}NO$: 226.1226; Found: 226.1232.



Pyridine 8a. The general procedure was followed utilizing substrate **8** (84.6 mg, 0.50 mmol, 1.0 equiv), $[Ph_2I]OTf$ (430 mg, 1.00 mmol, 2 equiv), $Ir(ppy)_2(dtbbpy)PF_6$ (22.8 mg, 0.025 mmol, 0.05 equiv), $Pd(NO_3)_2•2H_2O$ (13.3 mg, 0.050 mmol, 0.10 equiv), and MeOH (2.5 mL). Product **8a** was obtained as a clear viscous oil (76.0 mg, 62% yield, R_f = 0.09 in 90% hexanes/10% Et_2O). ^1H and ^{13}C NMR data matched those reported in the literature.[9]

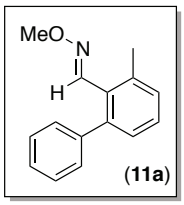


Pyridine 9a. The general procedure was followed utilizing substrate **9** (92.6 mg, 0.50 mmol, 1.0 equiv), $[Ph_2I]OTf$ (430 mg, 1.00 mmol, 2 equiv), $Ir(ppy)_2(dtbbpy)PF_6$ (22.8 mg, 0.025 mmol, 0.05 equiv), $Pd(NO_3)_2•2H_2O$ (13.3 mg, 0.050 mmol, 0.10 equiv), and MeOH (2.5 mL). Product **9a** was obtained as a pale yellow solid [88.0 mg, 67% yield, R_f = 0.11 in 60% hexanes/40% Et_2O, mp = 83.5-86.4 ºC (lit. 77.7-85.4 ºC)].[9] ^1H and ^{13}C NMR data matched those reported in the literature.[9]



Oxime ether 10a. The general procedure was followed utilizing substrate **10** (81.6 mg, 0.50 mmol, 1.0 equiv), $[Ph_2I]OTf$ (430 mg, 1.00 mmol, 2 equiv), $Ir(ppy)_2(dtbbpy)PF_6$ (22.8 mg, 0.025 mmol, 0.05 equiv), $Pd(NO_3)_2•2H_2O$ (13.3 mg, 0.050 mmol, 0.10 equiv), and MeOH (2.5 mL). Product **10a** was obtained as a colorless oil (71.4 mg, 60% yield, R_f = 0.14 in 98% hexanes/2% Et_2O). ^1H NMR (700 MHz, $CDCl_3$): δ 7.39–7.36 (multiple peaks, 4H), 7.32 (m, 1H), 7.29 (d, J = 7.7 Hz, 1H), 7.23 (dd, J = 7.0, 0.7 Hz, 1H), 7.20 (dd, J = 7.7, 0.7 Hz, 1H), 3.92 (s, 3H), 2.37 (s, 3H), 1.69 (s, 3H). $^{13}C\{^1H\}$ NMR (176 MHz, $CDCl_3$): δ 156.52,

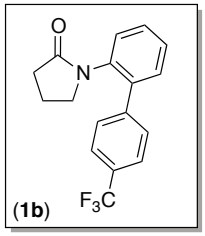
141.21, 140.97, 136.19, 136.07, 129.38, 129.34, 128.13, 127.94, 127.63, 126.92, 61.62, 20.08, 16.56. IR (thin film, neat) 3060, 2936, 1459, 1041 cm^{-1}. HRMS $[M+H]^+$ Calcd for $C_{16}H_{18}NO$: 240.1383; Found: 240.1387.



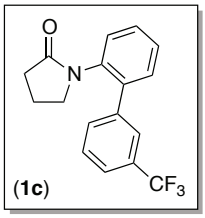
Oxime ether 11a. The general procedure was followed utilizing substrate **11** (74.6 mg, 0.50 mmol, 1.0 equiv), $[Ph_2I]OTf$ (430 mg, 1.00 mmol, 2 equiv), $Ir(ppy)_2(dtbbpy)PF_6$ (22.8 mg, 0.025 mmol, 0.05 equiv), $Pd(NO_3)_2•2H_2O$ (13.3 mg, 0.050 mmol, 0.10 equiv), and MeOH (2.5 mL). Product **11a** was obtained as a colorless oil consisting of a ~1.5:1 mixture of oxime stereoisomers (64.7 mg, 57% yield, R_f = 0.28 (major) and 0.14 (minor) in 6:1:0.2 hexanes/benzene/methylene chloride). Major Isomer: ^1H NMR (700 MHz, C_6D_6): δ 8.26 (s, 1H); 7.22 (m, 2H), 7.09 (tt, *J* = 7.4, 1.4 Hz, 2H); 7.06-7.05 (multiple peaks, 2H); 7.04-7.02 (multiple peaks, 2H); 3.76 (s, 3H), 2.62 (s, 3H). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 149.09, 143.18, 140.66, 137.83, 130.33, 129.83, 128.48, 128.12, 127.84, 127.21, 61.85, 22.46. Two aromatic ^{13}C resonances are coincidentally overlapping. IR (thin film, neat) 3059, 2935, 1460, 1048 cm^{-1}. HRMS $[M+H]^+$ Calcd for $C_{15}H_{16}NO$: 226.1226; Found: 226.1227. Minor Isomer: ^1H NMR (700 MHz, C_6D_6): δ 7.40 (d, *J* = 7.7 Hz, 2H); 7.24 (s, 1H); 7.19 (t, *J* = 7.7 Hz, 2H); 7.13-7.10 (multiple peaks, 2H); 7.08 (t, *J* = 7.7 Hz, 1H); 6.98 (d, *J* = 7.7 Hz, 1H); 3.66 (s, 3H); 2.22 (s, 3H). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 147.58, 140.70, 140.66, 136.43, 130.19, 128.87, 128.84, 128.74, 128.05, 127.36, 126.99, 61.79, 20.13. IR (thin film, $CDCl_3$) 3059, 2935, 1460, 1057 cm^{-1}. HRMS $[M+H]^+$ Calcd for $C_{15}H_{16}NO$: 226.1226; Found: 226.1228.

III. Synthesis and Characterization of Products in Table 3

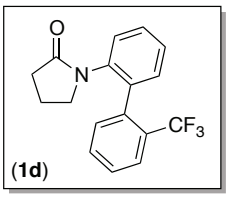
General Procedure: Substrate (1 equiv), $[Ar_2I]BF_4$ (2 equiv), $Ir(ppy)_2(dtbbpy)PF_6$ (0.05 equiv), and $Pd(NO_3)_2•2H_2O$ (0.10 equiv) were combined in MeOH in a 4 mL scintillation vial. The reaction mixture was cooled in an ice bath (to prevent evaporation) and sparged with N_2 using a submerged needle for 10 min, and the vial was then immediately sealed with a Teflon-lined cap. The vial was placed on a stir plate with two 26 W compact fluorescent light bulbs (one on either side of the vial about 5–8 cm away), and the reaction mixture was allowed to stir at room temperature for 15 h. The reaction mixture was diluted with EtOAc (50 mL) and washed with 10% aqueous Na_2SO_3 (2 x 25 mL) and brine (1 x 25 mL). The combined aqueous layers were extracted with EtOAc (3 x 10 mL), and the organic layers were then combined, dried over $MgSO_4$, filtered, concentrated, and purified by column chromatography on silica gel.



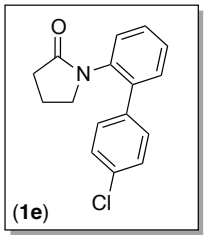
Pyrrolidinone 1b. The general procedure was followed utilizing substrate **1** (80.6 mg, 0.50 mmol, 1.0 equiv), $[(p\text{-}CF_3C_6H_4)_2I]BF_4$ (504 mg, 1.00 mmol, 2 equiv), $Ir(ppy)_2(dtbbpy)PF_6$ (22.8 mg, 0.025 mmol, 0.05 equiv), $Pd(NO_3)_2•2H_2O$ (13.3 mg, 0.050 mmol, 0.10 equiv), and MeOH (2.5 mL). Product **1b** was obtained as a tan solid [106 mg, 69% yield, R_f = 0.17 in 20% hexanes/80% Et_2O, mp = 87.6-89.2 ºC (lit. 86.1–88.0 ºC)].[9] ^1H and ^{13}C NMR data matched those reported in the literature.[9]



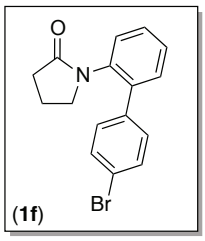
Pyrrolidinone 1c. The general procedure was followed utilizing substrate **1** (80.6 mg, 0.50 mmol, 1.0 equiv), $[(m\text{-}CF_3C_6H_4)_2I]BF_4$ (504 mg, 1.00 mmol, 2 equiv), $Ir(ppy)_2(dtbbpy)PF_6$ (22.8 mg, 0.025 mmol, 0.05 equiv), $Pd(NO_3)_2•2H_2O$ (13.3 mg, 0.050 mmol, 0.10 equiv), and MeOH (2.5 mL). Product **1c** was obtained as a tan solid [86.1 mg, 56% yield, R_f = 0.23 in 20% hexanes/80% Et_2O, mp = 79.2-83.5 ºC]. ^1H NMR (700 MHz, $CDCl_3$): δ 7.64 (br s, 1H), 7.62 (d, J = 8.4 Hz, 1H), 7.60 (d, J = 7.7 Hz, 1H), 7.53 (t, J = 7.7 Hz, 1H), 7.45 (m, 1H), 7.41–7.40 (multiple peaks, 2H), 7.33 (d, J = 7.7 Hz, 1H), 3.28 (t, J = 7.0 Hz, 2H), 2.40 (t, J = 8.1 Hz, 2H), 1.91 (tt, J = 8.1, 7.0 Hz, 2H). $^{13}C\{^1H\}$ NMR (176 MHz, $CDCl_3$): δ 175.42, 139.84, 138.11, 136.29, 131.80, 130.71 (q, $J_{C–F}$ = 32 Hz), 130.60, 129.25, 128.96, 128.30, 128.25, 124.99 (q, $J_{C–F}$ = 3.6 Hz), 124.21 (q, $J_{C–F}$ = 3.8 Hz), 123.97 (q, $J_{C–F}$ = 272 Hz), 50.30, 30.93, 18.83. ^{19}F NMR (376 MHz, $CDCl_3$): δ –62.65 (s). IR (thin film, $CDCl_3$) 2918, 1692, 1333, 1117 cm^{-1}. HRMS $[M+H]^+$ Calcd for $C_{17}H_{15}F_3NO$: 306.1100; Found: 306.1110.



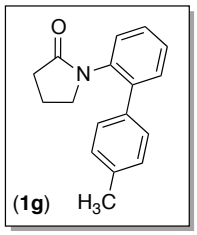
Pyrrolidinone 1d. The general procedure was followed utilizing substrate **1** (40.3 mg, 0.25 mmol, 1.0 equiv), $[(o\text{-}CF_3C_6H_4)_2I]BF_4$ (252 mg, 0.50 mmol, 2 equiv), $Ir(ppy)_2(dtbbpy)PF_6$ (11.4 mg, 0.0125 mmol, 0.05 equiv), $Pd(NO_3)_2•2H_2O$ (6.7 mg, 0.025 mmol, 0.10 equiv), and MeOH (1.25 mL). Product **1d** was obtained as a white solid (35.0 mg, 46% yield, R_f = 0.13 in 20% hexanes/80% Et_2O, mp = 61.8-63.9 ºC). ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.76 (d, *J* = 7.7 Hz, 1H), 7.54 (t, *J* = 7.7 Hz, 1H), 7.49 (t, *J* = 7.7 Hz, 1H), 7.46 (td, *J* = 7.7, 1.4 Hz, 1H), 7.41 (d, *J* = 7.7 Hz, 1H), 7.36 (td, *J* = 7.4, 1.4 Hz, 1H), 7.33–7.32 (multiple peaks, 2H), 3.36 (ddd, *J* = 14.0, 7.7, 5.6 Hz, 1H), 3.03 (ddd, *J* = 14.0, 8.4, 5.6 Hz, 1H), 2.40 (ddd, *J* = 16.4, 9.1, 6.3 Hz, 1H), 2.22 (ddd, *J* = 16.4, 9.1, 6.3 Hz, 1H), 1.94 (m, 1H), 1.67 (m, 1H). $^{13}C\{^1H\}$ NMR (176 MHz, $CDCl_3$): δ 175.57, 137.43, 136.99, 136.76, 132.08, 131.25, 131.05 (q, $J_{C–F}$ = 2.1 Hz), 129.25, 128.28 (q, $J_{C–F}$ = 30 Hz), 128.08, 127.96, 127.17, 126.21 (q, $J_{C–F}$ = 5.3 Hz), 124.06 (q, $J_{C–F}$ = 274 Hz), 49.90, 30.98, 19.05. ^{19}F NMR (376 MHz, $CDCl_3$): δ –57.09 (s). IR (thin film, $CDCl_3$) 2920, 1697, 1313, 1111 cm^{-1}. HRMS $[M+H]^+$ Calcd for $C_{17}H_{15}F_3NO$: 306.1100; Found: 306.1112.



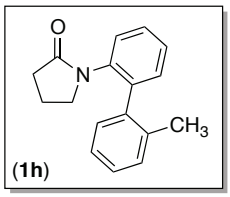
Pyrrolidinone 1e. The general procedure was followed utilizing substrate **1** (80.6 mg, 0.50 mmol, 1.0 equiv), $[(p\text{-}ClC_6H_4)_2I]BF_4$ (437 mg, 1.00 mmol, 2 equiv), $Ir(ppy)_2(dtbbpy)PF_6$ (22.8 mg, 0.025 mmol, 0.05 equiv), $Pd(NO_3)_2•2H_2O$ (13.3 mg, 0.050 mmol, 0.10 equiv), and MeOH (2.5 mL). Product **1e** was obtained as a tan solid [104 mg, 77% yield, R_f = 0.13 in 20% hexanes/80% Et_2O, mp = 95.6-97.4 ºC (lit. 93.9-96.0 ºC)].[9] ^{1}H and ^{13}C NMR data matched those reported in the literature.[9]



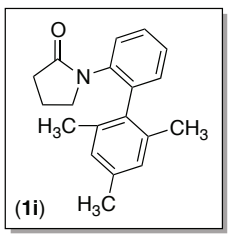
Pyrrolidinone 1f. The general procedure was followed utilizing substrate **1** (80.6 mg, 0.50 mmol, 1.0 equiv), $[(p\text{-}BrC_6H_4)_2I]BF_4$ (526 mg, 1.00 mmol, 2 equiv), $Ir(ppy)_2(dtbbpy)PF_6$ (22.8 mg, 0.025 mmol, 0.05 equiv), $Pd(NO_3)_2•2H_2O$ (13.3 mg, 0.050 mmol, 0.10 equiv), and MeOH (2.5 mL). Product **1f** was obtained as a pale yellow oil (125 mg, 79% yield, R_f = 0.13 in 20% hexanes/80% Et_2O). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.54 (d, *J* = 8.5 Hz, 2H), 7.44–7.35 (multiple peaks, 3H) 7.33 (d, *J* = 7.5 Hz, 1H), 7.27 (d, *J* = 8.5 Hz, 2H), 3.27 (t, *J* = 7.0 Hz, 2H), 2.44 (t, *J* = 8.0 Hz, 2H), 1.93 (tt, *J* = 8.0, 7.0 Hz, 2H). $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$): δ 175.61, 138.48, 138.01, 136.18, 131.56, 130.61, 130.00, 128.95, 128.40, 128.18, 121.87, 50.26, 31.09, 18.94. IR (thin film, neat) 2879, 1680, 1402 cm^{-1}. HRMS $[M+H]^+$ Calcd for $C_{16}H_{15}BrNO$: 316.0332; Found: 316.0340.



Pyrrolidinone 1g. The general procedure was followed utilizing substrate **1** (80.6 mg, 0.50 mmol, 1.0 equiv), [(*p*-$MeC_6H_4)_2I]BF_4$ (396 mg, 1.00 mmol, 2 equiv), $Ir(ppy)_2(dtbbpy)PF_6$ (22.8 mg, 0.025 mmol, 0.05 equiv), $Pd(NO_3)_2$•$2H_2O$ (13.3 mg, 0.050 mmol, 0.10 equiv), and MeOH (2.5 mL). Product **1g** was obtained as a tan solid (109.8 mg, 87% yield, R_f = 0.17 in 20% hexanes/80% Et_2O, mp = 78.6-80.4 ºC). ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.40-7.35 (multiple peaks, 3H), 7.31 (d, *J* = 7.3 Hz, 1H), 7.27 (d, *J* = 8.0 Hz, 2H), 7.20 (d, *J* = 8.0 Hz, 2H), 3.22 (t, *J* = 6.9 Hz, 2H), 2.43 (t, *J* = 8.0 Hz, 2H), 2.39 (s, 3H), 1.88 (tt, *J* = 8.0, 6.9 Hz, 2H). $^{13}C\{^1H\}$ NMR (176 MHz, $CDCl_3$): δ 175.61, 139.52, 137.28, 136.27, 136.16, 130.86, 129.12, 128.34, 128.30, 128.18, 127.98, 50.06, 31.20, 21.18, 18.97. IR (thin film, $CDCl_3$) 3026, 2920, 1694, 1487, 1407, 1301 cm^{-1}. HRMS $[M+H]^+$ Calcd for $C_{17}H_{18}NO$: 252.1383; Found: 252.1391.

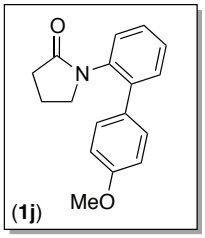


Pyrrolidinone 1h. The general procedure was followed utilizing substrate **1** (80.6 mg, 0.50 mmol, 1.0 equiv), [(*o*-$MeC_6H_4)_2I]BF_4$ (396 mg, 1.00 mmol, 2 equiv), $Ir(ppy)_2(dtbbpy)PF_6$ (22.8 mg, 0.025 mmol, 0.05 equiv), $Pd(NO_3)_2$•$2H_2O$ (13.3 mg, 0.050 mmol, 0.10 equiv), and MeOH (2.5 mL). Product **1h** was obtained as a pale yellow oil (107 mg, 85% yield, R_f = 0.20 in 20% hexanes/80% Et_2O). NMR (500 MHz, $CDCl_3$): δ 7.40 (ddd, *J* = 7.7, 7.0, 1.4 Hz, 1H), 7.36 (dd, *J* = 8.4, 1.4 Hz, 1H), 7.34 (td, *J* = 7.7, 1.4 Hz, 1H), 7.27–7.24 (multiple peaks, 3H), 7.19 (m, 1H), 7.16 (d, 7.0 Hz, 1H), 3.23 (ddd, *J* = 9.1, 8.4, 5.6 Hz, 1H), 3.09 (ddd, *J* = 9.1, 7.7, 5.6 Hz, 1H) 2.32 (m, 2H), 2.15 (s, 3H), 1.82 (m, 1H), 1.75 (m, 1H). $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$): δ 175.10, 138.89, 138.60, 136.96, 135.88, 131.11, 130.13, 129.39, 128.3, 128.1, 127.72, 127.29, 125.47, 49.94, 31.15, 19.93, 19.02. IR (thin film, neat) 2952, 1696, 1398 cm^{-1}. HRMS $[M+H]^+$ Calcd for $C_{17}H_{18}NO$: 252.1383; Found: 252.1392.



Pyrrolidinone 1i. The general procedure was followed utilizing substrate **1** (40.3 mg, 0.25 mmol, 1.0 equiv), $[Mes_2I]OTf$ (257 mg, 0.50 mmol, 2 equiv), $Ir(ppy)_2(dtbbpy)PF_6$ (11.3 mg, 0.0125 mmol, 0.05 equiv), $Pd(NO_3)_2$•$2H_2O$ (6.7 mg, 0.025 mmol, 0.10 equiv), and MeOH (1.25 mL). Product **1i** was obtained as a white solid (8 mg, 11% yield, R_f = 0.17 in 96% CH_2Cl_2/4% Et_2O, mp = 121.2-123.8 ºC). ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.44–7.39 (multiple peaks, 2H), 7.34 (td, *J* = 7.5, 1.5 Hz, 1H), 7.13 (dd, *J* = 7.5, 1.2 Hz, 1H), 6.92 (s, 2H), 3.12 (t, *J* = 6.9 Hz, 2H), 2.36 (t, *J* = 8.0 Hz, 2H), 2.34 (s, 3H), 1.98 (s, 6H), 1.80 (tt, *J* = 8.0, 6.9 Hz, 2H). $^{13}C\{^1H\}$ NMR (176 MHz, $CDCl_3$): δ 174.82, 137.32, 137.16, 137.01, 136.23, 135.62, 131.27, 128.25, 128.06, 127.97, 127.33, 49.16, 31.35,

21.06, 20.41, 19.07. IR (thin film, CH_2Cl_2) 2918, 1699, 1398, 1301 cm^{-1}. HRMS $[M+H]^+$ Calcd for $C_{19}H_{22}NO$: 280.1699; Found: 280.1705.



Pyrrolidinone 1j. The general procedure was followed utilizing substrate **1** (80.6 mg, 0.50 mmol, 1.0 equiv), $[(p\text{-}OMeC_6H_4)_2I]BF_4$ (428 mg, 1.00 mmol, 2 equiv), $Ir(ppy)_2(dtbbpy)PF_6$ (22.8 mg, 0.025 mmol, 0.05 equiv), $Pd(NO_3)_2 \bullet 2H_2O$ (13.3 mg, 0.050 mmol, 0.10 equiv), and MeOH (2.5 mL). Product **1j** was obtained as a tan viscous oil (54.3 mg, 41% yield, R_f = 0.07 in 20% hexanes/80% Et_2O). ^1H and ^{13}C NMR data matched those reported in the literature.[9]

IV. Experimental Details for Table 4

Radical/Photocatalytic Procedure for Reactions in Table 4 (entries 1–5): Substrate **8** (8.5 mg, 0.050 mmol, 1 equiv), $[Ph_2I]BF_4$ (36.8 mg, 0.100 mmol, 2 equiv), $Ir(ppy)_2(dtbbpy)PF_6$ (2.3 mg, 0.0025 mmol, 0.05 equiv), $Pd(NO_3)_2•2H_2O$ (1.3 mg, 0.005 mmol, 0.10 equiv), and galvinoxyl (0, 2.1, or 5.3 mg; 0, 0.005, or 0.0125 mmol; 0, 0.10, or 0.25 equiv) or TEMPO (0, 3.9, or 7.8 mg; 0, 0.025, or 0.050 mmol; 0, 0.50, or 1.0 equiv) were combined in MeOH (0.25 mL) in a 4 mL scintillation vial. The reaction mixture was cooled in an ice bath (to prevent evaporation) and sparged with N_2 using a submerged needle for 1 min, and the vial was then immediately sealed with a Teflon-lined cap. The vial was placed on a stir plate with two 26 W compact fluorescent light bulbs (one on either side of the vial about 5–8 cm away), and the reaction mixture was allowed to stir at room temperature for 15 h. Reactions were then quenched with 10% aqueous Na_2SO_3 (0.25 mL), diluted with EtOAc (3.5 mL), and analyzed by GC-FID. GC calibrated yields are reported relative to hexadecane as an internal standard. The yields reported in Table 4 are the averages of three separate trials.

Ionic/Thermal Procedure for Reactions in Table 4 (entries 6–9). Substrate **8** (8.5 mg, 0.050 mmol, 1 equiv), $[Ph_2I]BF_4$ (20.2 mg, 0.055 mmol, 1.1 equiv), $Pd(OAc)_2$ (1.1 mg, 0.005 mmol, 0.10 equiv), and galvinoxyl (0 or 5.3 mg; 0 or 0.0125 mmol; 0 or 0.25 equiv) or TEMPO (0 or 7.8 mg; 0 or 0.050 mmol; 0 or 1.0 equiv) were combined in AcOH (0.42 mL) in a 4 mL scintillation vial. The reaction was heated to 100 ºC for 15 h, then quenched with 10% aqueous Na_2SO_3 (0.25 mL), diluted with EtOAc (3.5 mL), and analyzed by GC-FID. GC calibrated yields are reported relative to hexadecane as an internal standard. The yields reported in Table 4 are the averages of three separate trials. These conditions are similar to those reported previously for 2-arylpyridine substrates;[11] however, the catalyst loading was increased to 10% (instead of 5%) to more closely resemble the conditions of the photocatalytic/radical trials.

V. Experimental Details for Equation 1

Radical/Photocatalytic Procedure for Reaction in Equation 1. Substrate **1** (8.1 mg, 0.050 mmol, 1 equiv), [(*o*-$CF_3C_6H_4$)–I–Ph]BF_4 (43.6 mg, 0.100 mmol, 2 equiv), $Ir(ppy)_2(dtbbpy)PF_6$ (2.3 mg, 0.0025 mmol, 0.05 equiv), and $Pd(NO_3)_2$•$2H_2O$ (1.3 mg, 0.005 mmol, 0.10 equiv) were combined in MeOH (0.25 mL) in a 4 mL scintillation vial. The reaction mixture was cooled in an ice bath (to prevent evaporation) and sparged with N_2 using a submerged needle for 1 min, and the vial was then immediately sealed with a Teflon-lined cap. The vial was placed on a stir plate with two 26 W compact fluorescent light bulbs (one on either side of the vial about 5–8 cm away), and the reaction mixture was allowed to stir at room temperature for 15 h. Reactions were then quenched with 10% aq. Na_2SO_3 (0.25 mL), diluted with EtOAc (3.5 mL), and analyzed by GC-FID. GC calibrated yields are reported relative to hexadecane as an internal standard.

Ionic/Thermal Procedure for Reaction in Equation 1. Substrate **1** (8.1 mg, 0.050 mmol, 1 equiv), [(*o*-$CF_3C_6H_4$)–I–Ph]BF_4 (43.6 mg, 0.100 mmol, 2 equiv), $Pd(OAc)_2$ (1.1 mg, 0.005 mmol, 0.10 equiv), and $NaHCO_3$ (6.3 mg, 0.075 mmol, 1.5 equiv) were combined in toluene (0.42 mL). The reaction was heated to 100 ºC for 15 h, then quenched with 10% aq. Na_2SO_3 (0.25 mL), diluted with EtOAc (3.5 mL), and analyzed by GC-FID. GC calibrated yields are reported relative to hexadecane as an internal standard. These conditions are similar to the conditions reported previously for substrate **1**;[11] however, the equivalents of oxidant were increased to 2 (instead of 1.5) and the catalyst loading was increased to 10% (instead of 5%) to more closely resemble the conditions of the photocatalytic/radical trials.

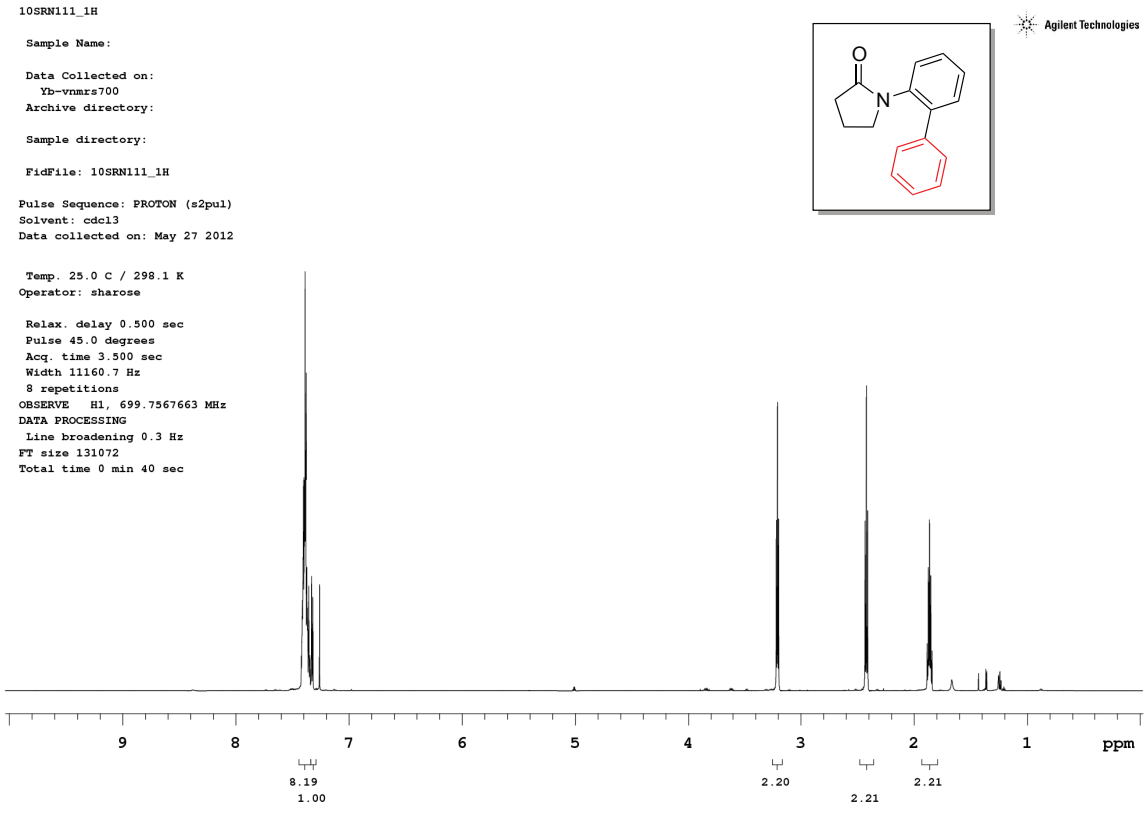
VI. Experimental Details for Table 5

PhN_2^+ procedure.[9] Substrate (0.050 mmol, 1 equiv), $Pd(OAc)_2$ (1.1 mg, 0.005 mmol, 0.10 equiv), $Ru(bpy)_3Cl_2 \cdot 6H_2O$ (0.94 mg, 0.00125 mmol, 0.025 equiv), and $[PhN_2]BF_4$ (38.4 mg, 0.200 mmol, 4 equiv) were combined in MeOH (500 μL) in a 4 mL scintillation vial. The reaction mixture was cooled in an ice bath (to prevent evaporation) and sparged with N_2 using a submerged needle for 1 min, and the vial was then immediately sealed with a Teflon-lined cap. The vial was placed on a stir plate with two 26 W compact fluorescent light bulbs (one on either side of the vial about 5–8 cm away), and the reaction mixture was allowed to stir at room temperature for 15 h. Reactions were then quenched with 10% aq. Na_2SO_3 (0.25 mL), diluted with EtOAc (3.5 mL), and analyzed by GC-FID. GC calibrated yields are reported relative to hexadecane as an internal standard.

Ph_2I^+ procedure. GC calibrated yields were obtained from the reactions described in Section II and are reported relative to hexadecane as an internal standard.

VII. References

1. Shakespeare, W. C. *Tetrahedron Lett.* **1999**, *40*, 2035.
2. Stowers K. J.; Sanford, M. S. *Org. Lett.* **2009**, *11*, 4584.
3. Neufeldt, S. R.; Sanford, M. S. *Org. Lett.* **2010**, *12*, 532.
4. Chen, D. W.; Ochiai, M. J. Org. Chem. **1999**, 64, 6804.
5. Bielawski, M.; Olofsson, B. *Chem. Commun.* **2007**, 2521.
6. Bielawski, M.; Aili, D.; Olofsson, B. *J. Org. Chem.* **2008**, *73*, 4602.
7. Stoessel, P.; Fortte, R.; Parham, A.; Breuning, E.; Heil, H.; Vestweber, H. U.S. Patent 20080312396A1, December 18, 2008.
8. Slinker, J. D.; Gorodetsky, A. A.; Lowry, M. S.; Wang, J.; Parker, S.; Rohl, R.; Bernhard, S.; Malliaras, G. G. *J. Am. Chem. Soc.* **2004**, *126*, 2763.
9. Kalyani, D.; McMurtrey, K. B.; Neufeldt, S. R.; Sanford, M. S. *J. Am. Chem. Soc.* **2011**, *133*, 18566.
10. Daugulis, O.; Zaitsev, V. *Angew. Chem. Int. Ed.* **2005**, *44*, 4046.
11. Kalyani, D.; Deprez, N. R.; Desai, L. V.; Sanford, M. S. *J. Am. Chem. Soc.* **2005**, *127*, 7330.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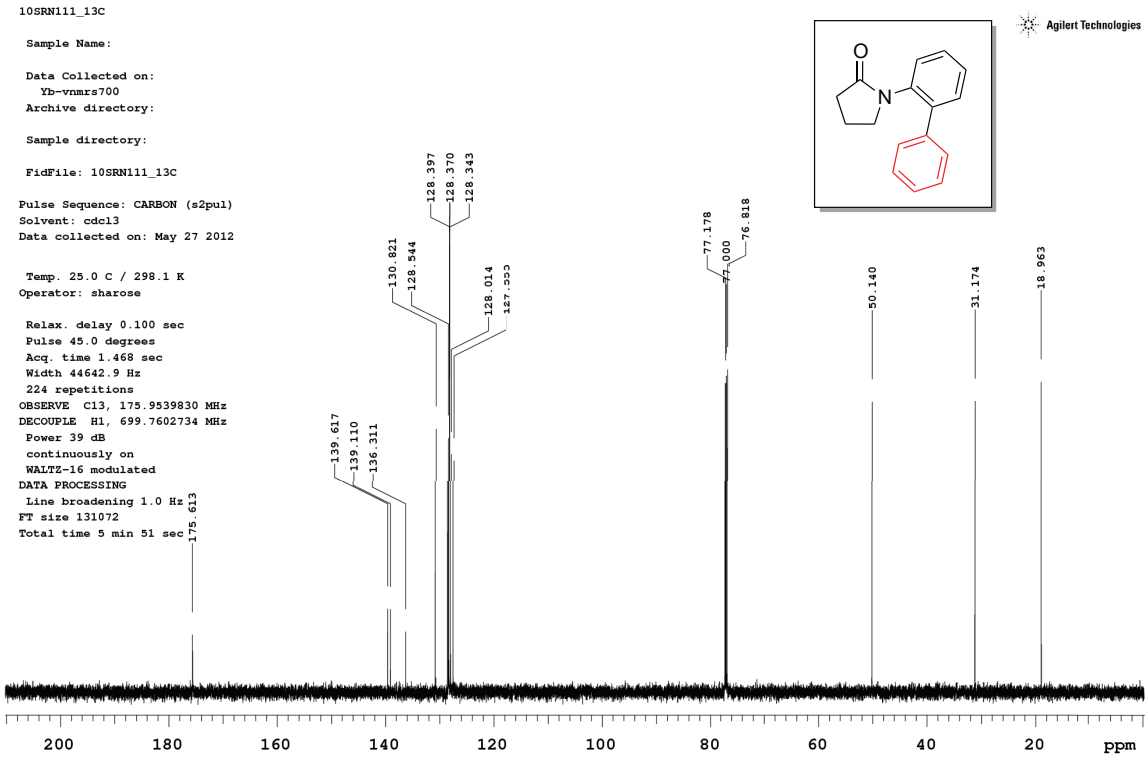
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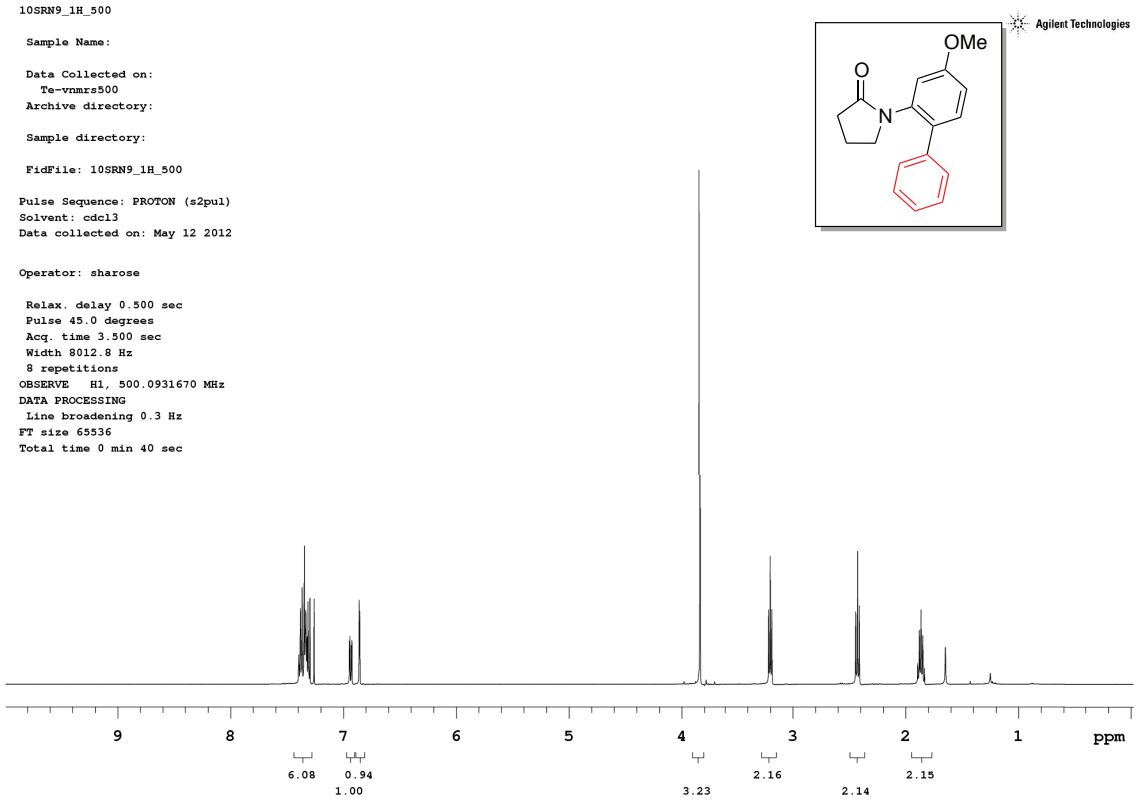
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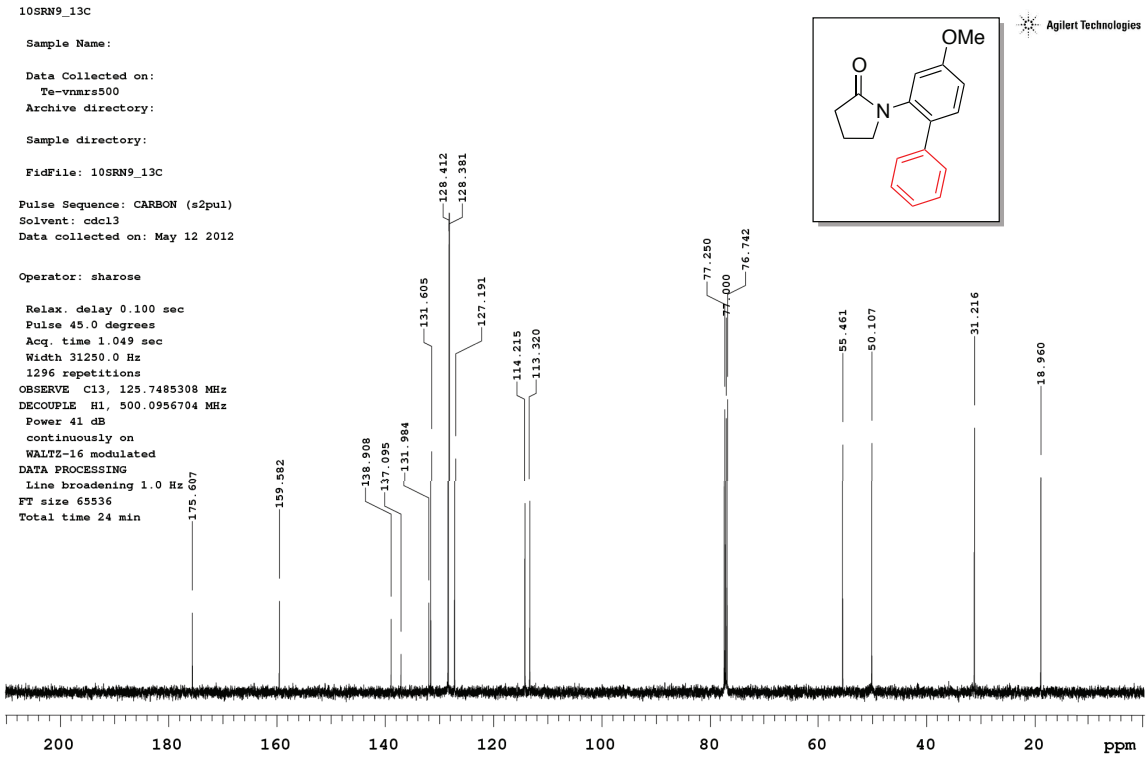
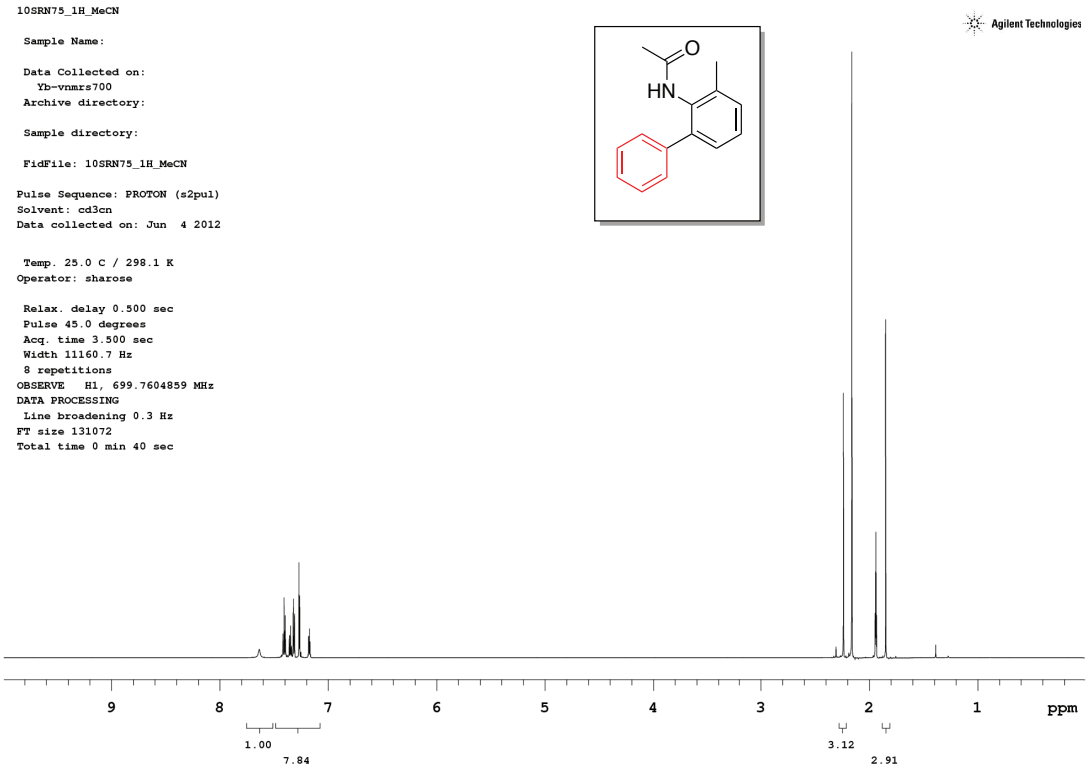
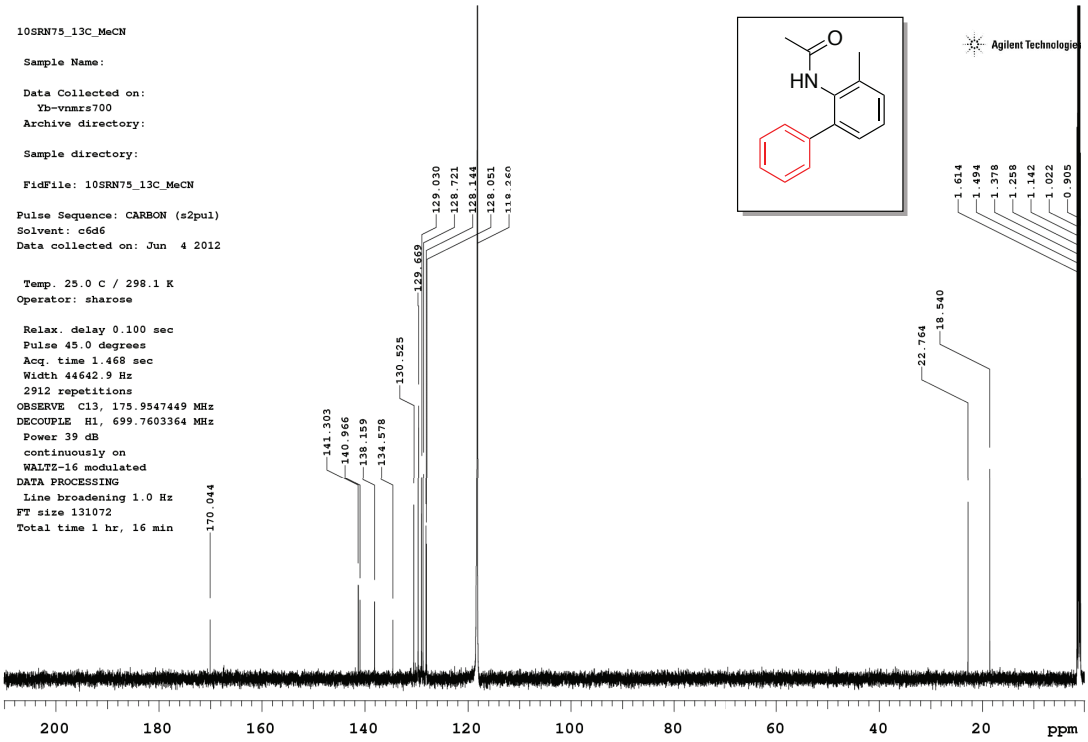
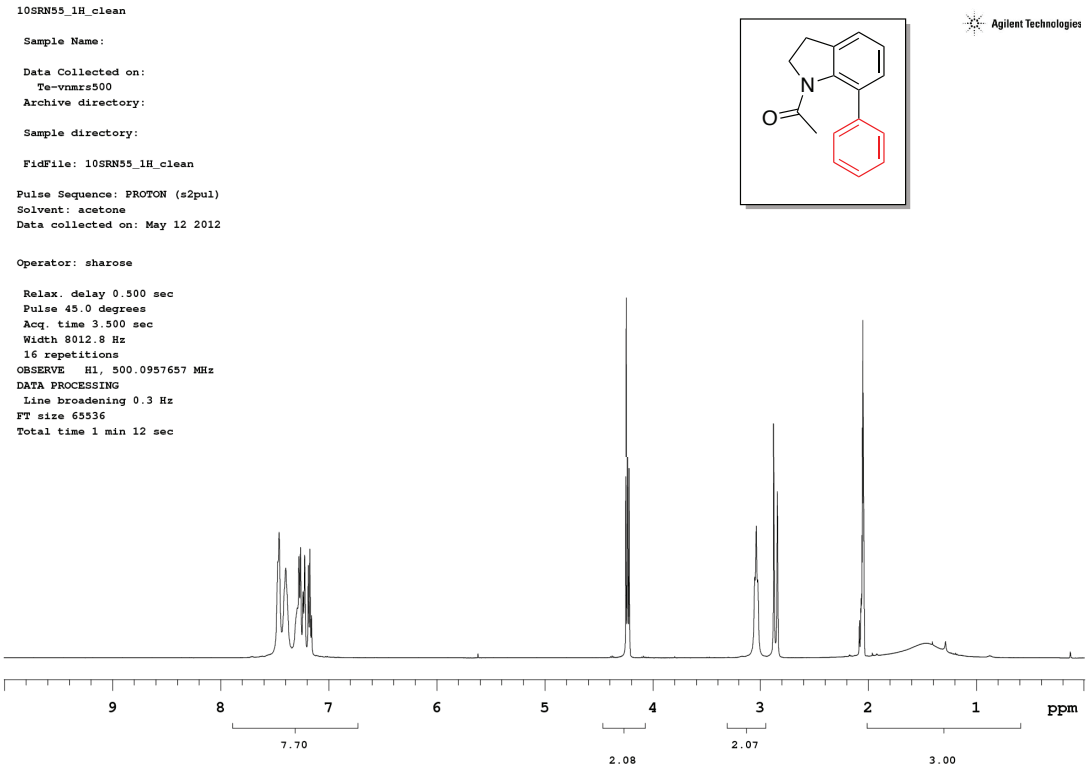
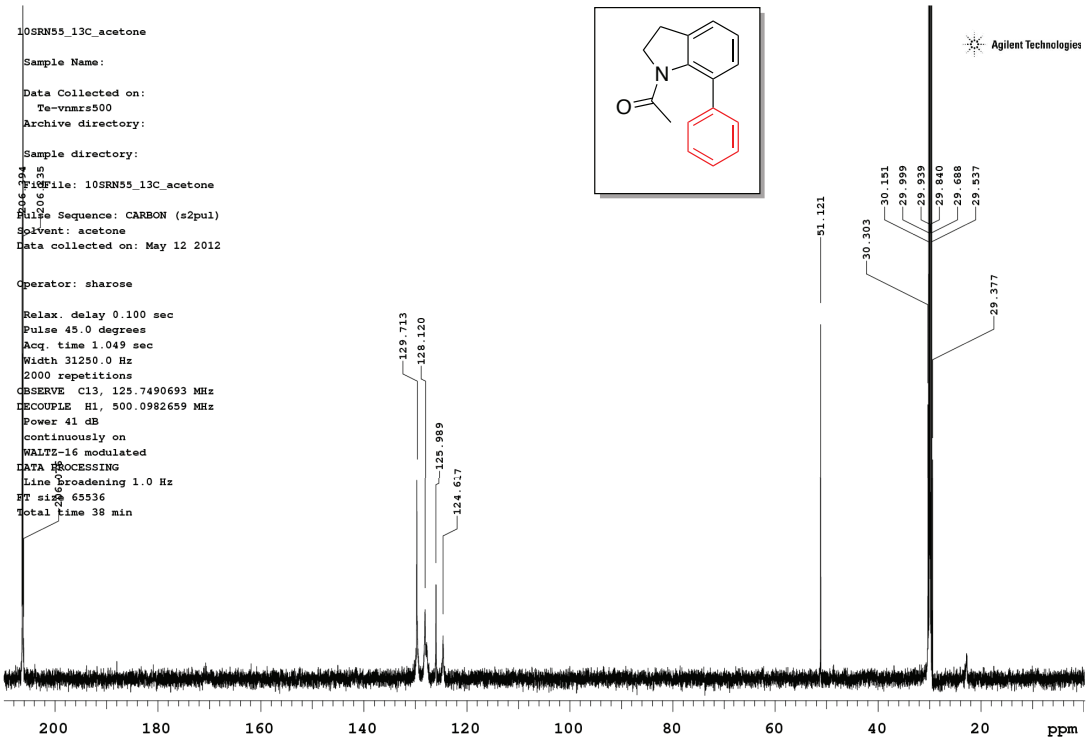
2.14
2.15

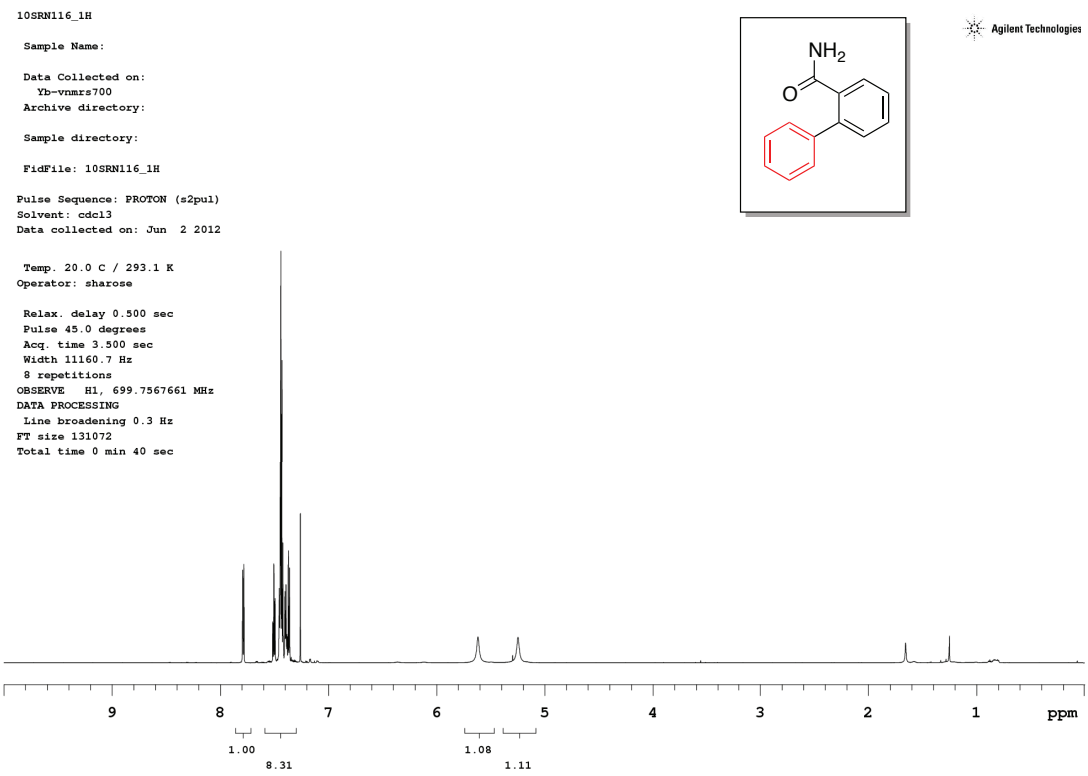

10SRN9_13C
Sample Name:
Data Collected on:
Te-vnmrs500
Archive directory:
Sample directory:
FidFile: 10SRN9_13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 12 2012
Operator: sharose
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.049 sec
Width 31250.0 Hz
1296 repetitions
OBSERVE C13, 125.7485308 MHz
DECOUPLE H1, 500.0956704 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 24 min
Agilent Technologies
OMe
O
N
175.607
159.582
138.908
137.095
131.984
131.605
128.412
128.381
127.191
114.215
113.320
77.250
77.000
76.742
55.461
50.107
31.216
18.960
200
180
160
140
120
100
80
60
40
20
ppm

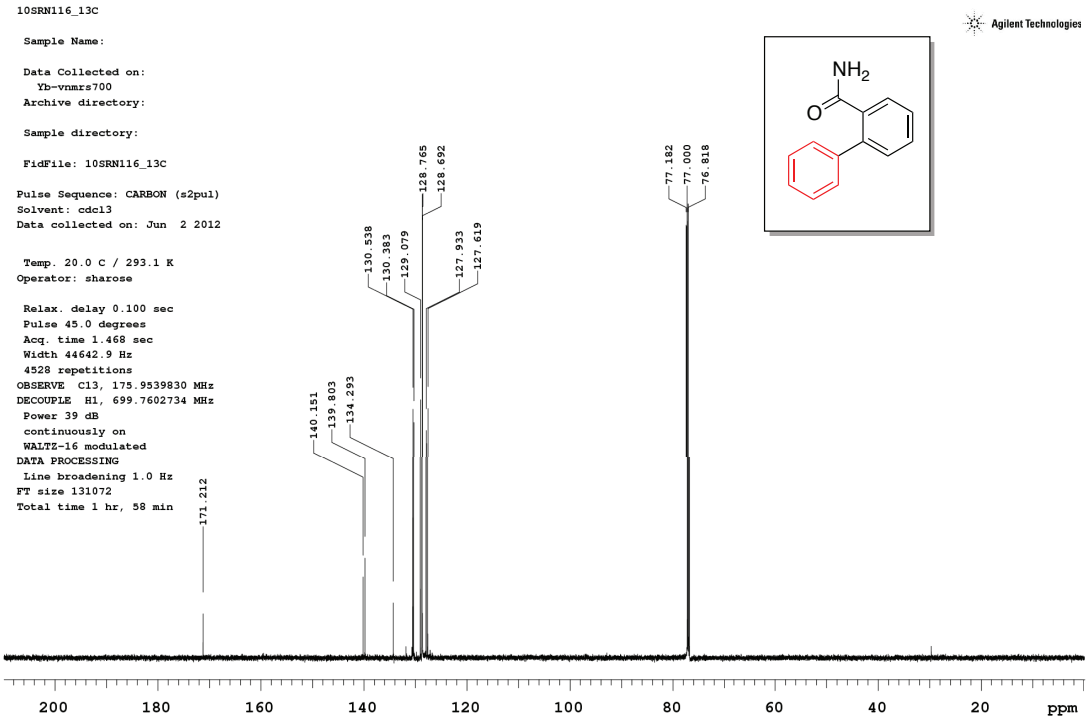

10SRN75_1H_MeCN
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: 10SRN75_1H_MeCN
Pulse Sequence: PROTON (s2pul)
Solvent: cd3cn
Data collected on: Jun 4 2012
Temp. 25.0 C / 298.1 K
Operator: sharose
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 11160.7 Hz
8 repetitions
OBSERVE H1, 699.7604859 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 0 min 40 sec
Agilent Technologies
O
HN
9
8
7
6
5
4
3
2
1
ppm
1.00
7.84
3.12
2.91

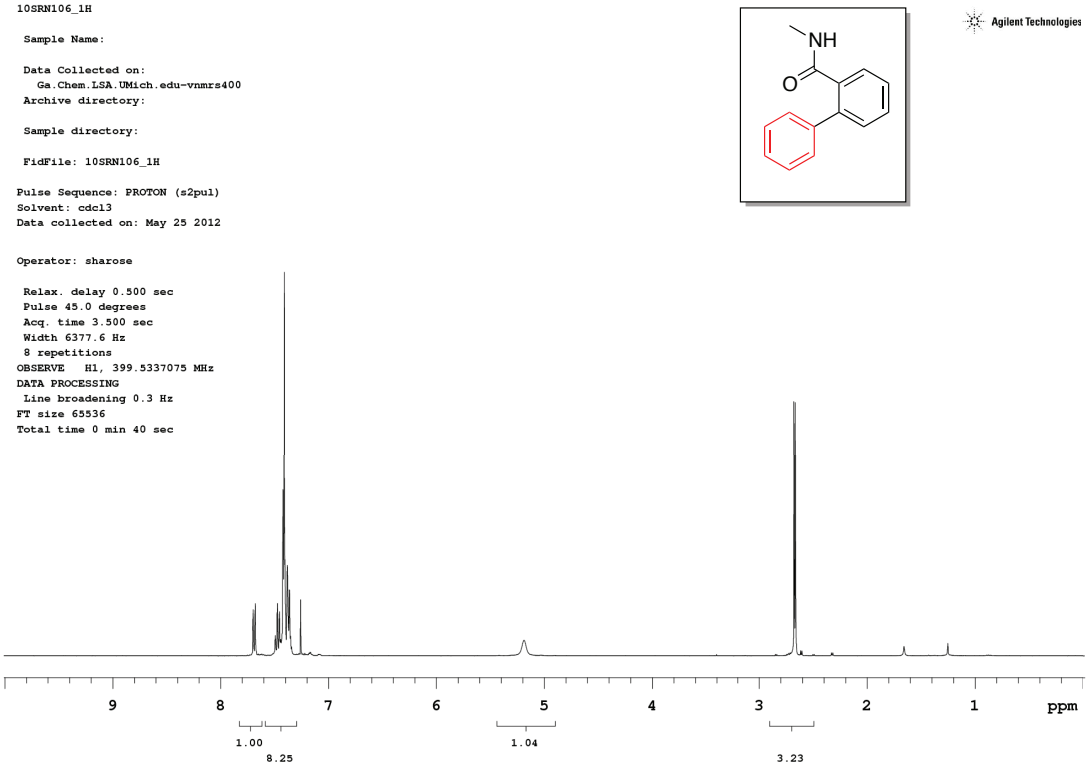

10SRN75_13C_MeCN
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: 10SRN75_13C_MeCN
Pulse Sequence: CARBON (s2pul)
Solvent: c6d6
Data collected on: Jun 4 2012
Temp. 25.0 C / 298.1 K
Operator: sharose
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.468 sec
Width 44642.9 Hz
2912 repetitions
OBSERVE C13, 175.9547449 MHz
DECOUPLE H1, 699.7603364 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 16 min
O
HN
Agilent Technologies
170.044
141.303
140.966
138.159
134.578
130.525
129.669
129.030
128.721
128.144
128.051
22.764
18.540
1.614
1.494
1.378
1.258
1.142
1.022
0.905
200
180
160
140
120
100
80
60
40
20
ppm

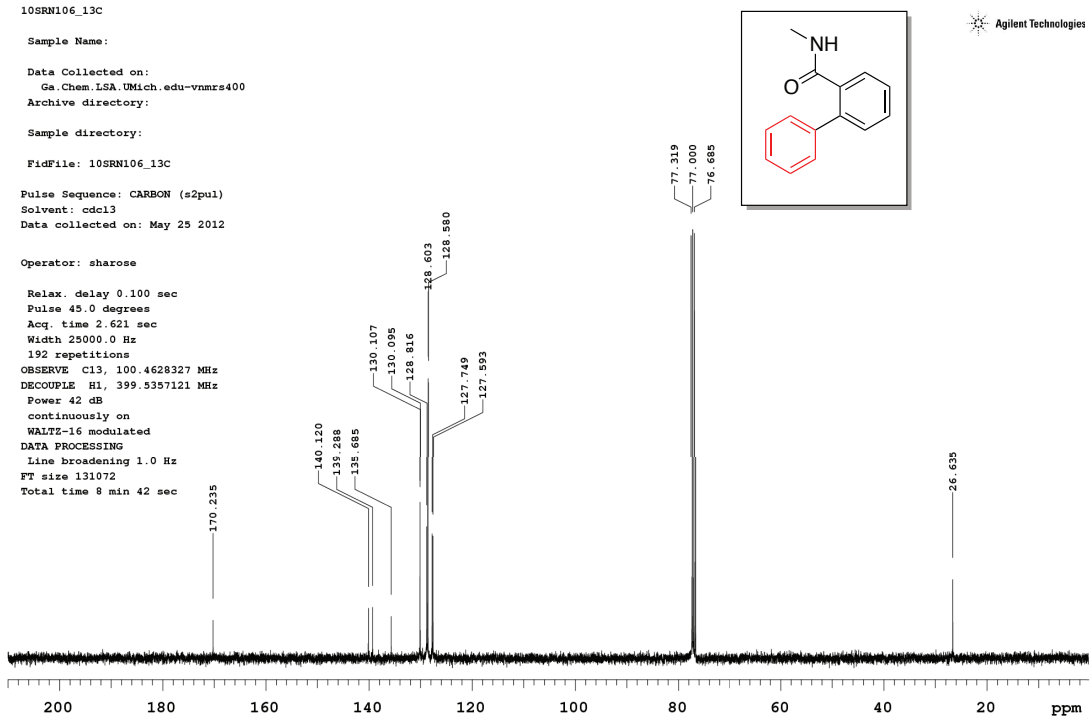

10SRN55_1H_clean
Sample Name:
Data Collected on:
Te-vnmrs500
Archive directory:
Sample directory:
FidFile: 10SRN55_1H_clean
Pulse Sequence: PROTON (s2pul)
Solvent: acetone
Data collected on: May 12 2012
Operator: sharose
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 8012.8 Hz
16 repetitions
OBSERVE H1, 500.0957657 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min 12 sec
Agilent Technologies
N
O
9
8
7
6
5
4
3
2
1
ppm
7.70
2.08
2.07
3.00

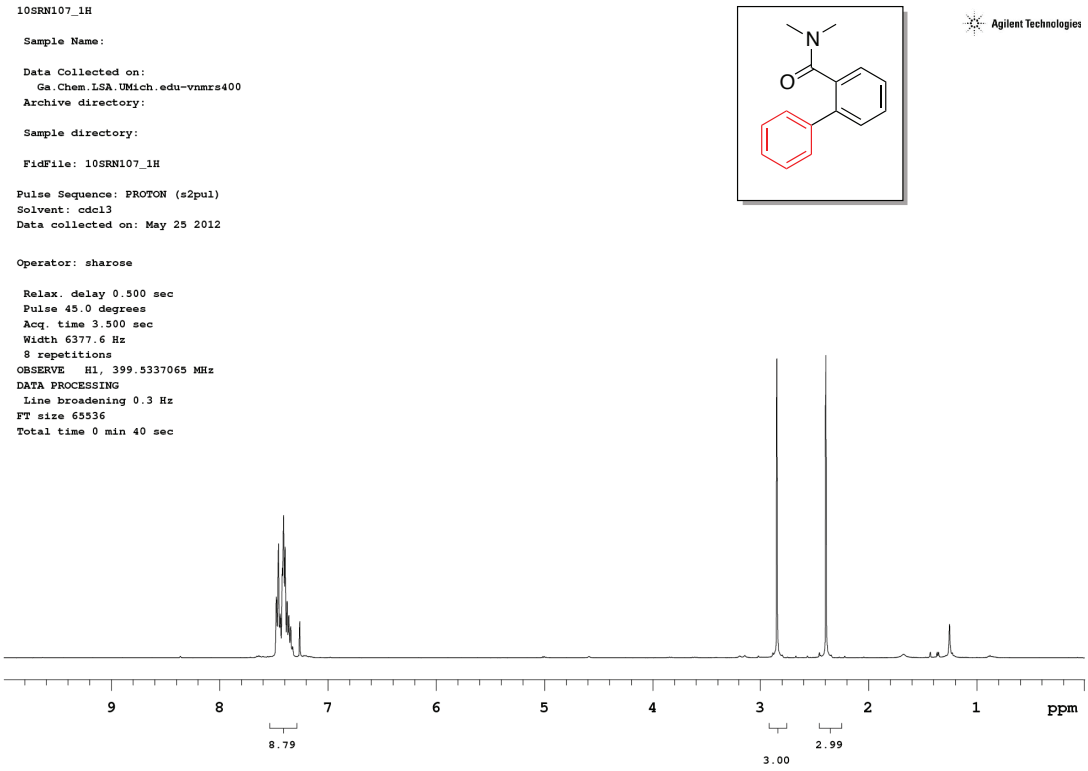

10SRN55_13C_acetone
Sample Name:
Data Collected on:
Te-vnmrs500
Archive directory:
Sample directory:
FidFile: 10SRN55_13C_acetone
Pulse Sequence: CARBON (s2pul)
Solvent: acetone
Data collected on: May 12 2012
Operator: sharose
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.049 sec
Width 31250.0 Hz
2000 repetitions
OBSERVE C13, 125.7490693 MHz
DECOUPLE H1, 500.0982659 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 38 min
206.394
206.035
205.676
129.713
128.120
125.989
124.617
51.121
30.303
30.151
29.999
29.939
29.840
29.688
29.537
29.377
Agilent Technologies
200
180
160
140
120
100
80
60
40
20
ppm

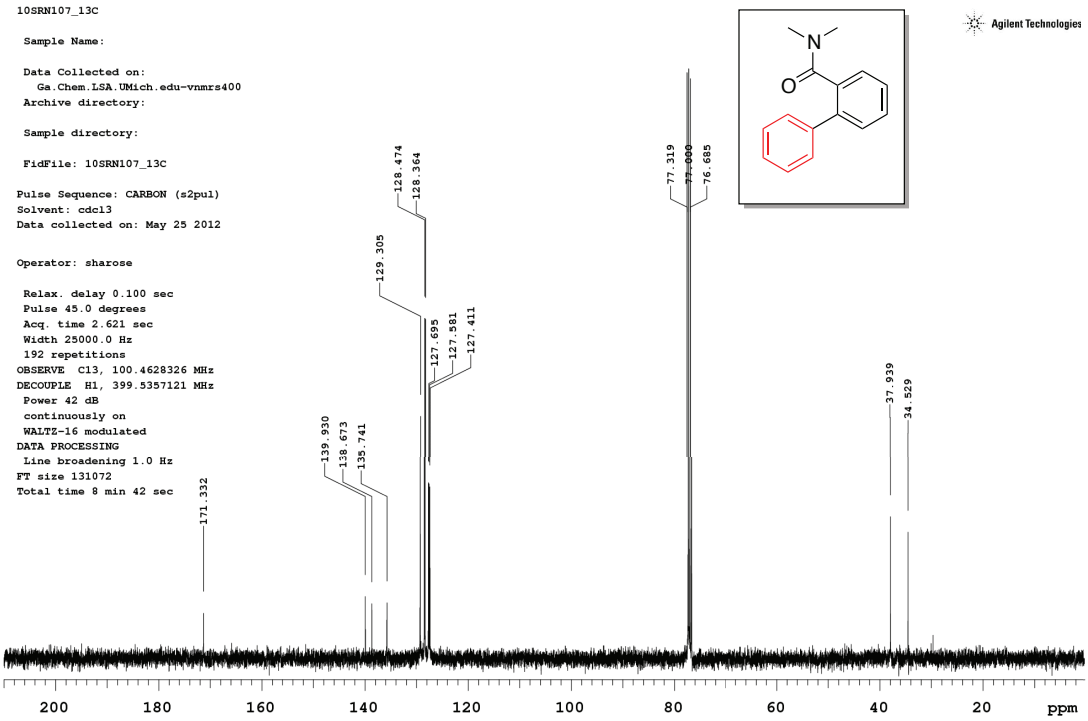

10SRN116_1H
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: 10SRN116_1H
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jun 2 2012
Temp. 20.0 C / 293.1 K
Operator: sharose
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 11160.7 Hz
8 repetitions
OBSERVE H1, 699.7567661 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 0 min 40 sec
NH2
O
Agilent Technologies
9
8
7
6
5
4
3
2
1
ppm
1.00
8.31
1.08
1.11

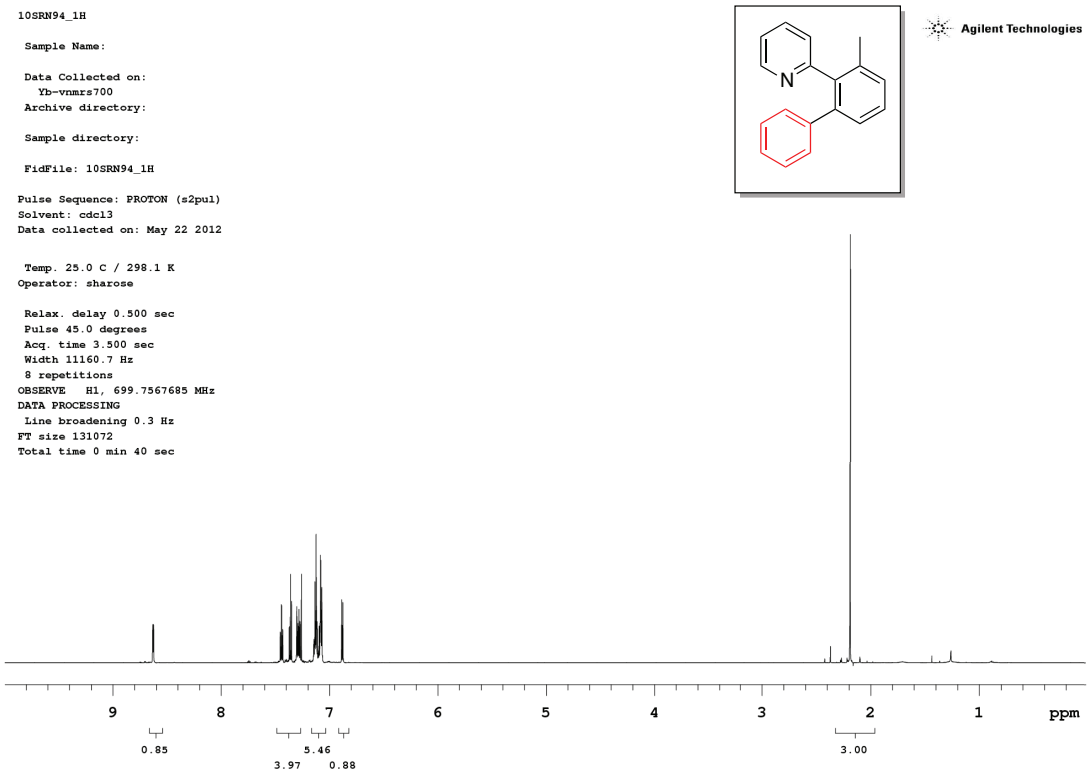

10SRN116_13C
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: 10SRN116_13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jun 2 2012
Temp. 20.0 C / 293.1 K
Operator: sharose
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.468 sec
Width 44642.9 Hz
4528 repetitions
OBSERVE C13, 175.9539830 MHz
DECOUPLE H1, 699.7602734 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 58 min
Agilent Technologies
NH2
O
171.212
140.151
139.803
134.293
130.538
130.383
129.079
128.765
128.692
127.933
127.619
77.182
77.000
76.818
200
180
160
140
120
100
80
60
40
20
ppm

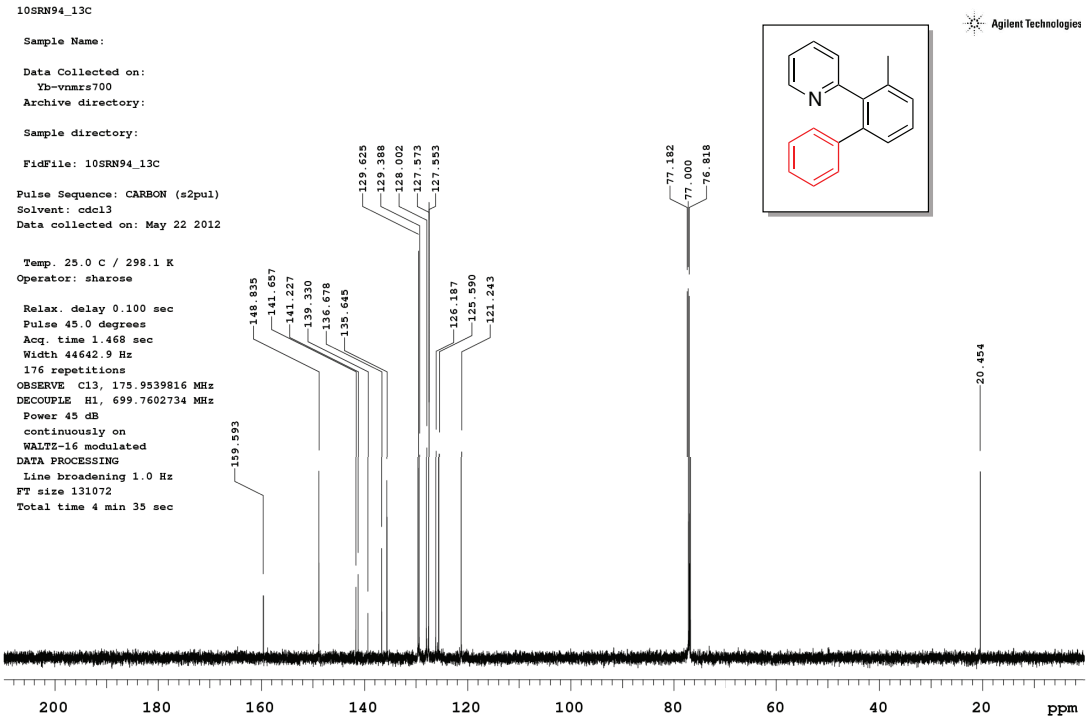

10SRN106_1H
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: 10SRN106_1H
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 25 2012
Operator: sharose
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6377.6 Hz
8 repetitions
OBSERVE H1, 399.5337075 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 0 min 40 sec
NH
O
Agilent Technologies
9
8
7
6
5
4
3
2
1
ppm
1.00
8.25
1.04
3.23

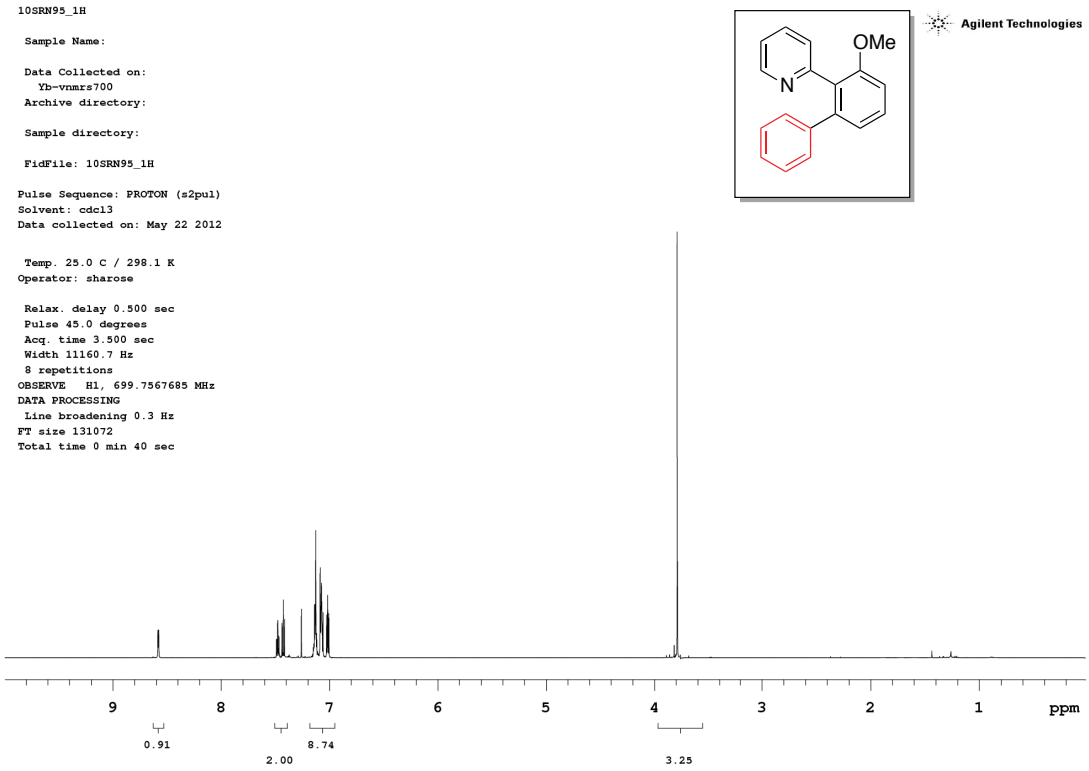

10SRN106_13C
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: 10SRN106_13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 25 2012
Operator: sharose
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.621 sec
Width 25000.0 Hz
192 repetitions
OBSERVE C13, 100.4628327 MHz
DECOUPLE H1, 399.5357121 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 8 min 42 sec
Agilent Technologies
NH
O
170.235
140.120
135.288
135.685
130.107
130.095
128.816
128.603
128.580
127.749
127.593
77.319
77.000
76.685
26.635
200
180
160
140
120
100
80
60
40
20
ppm

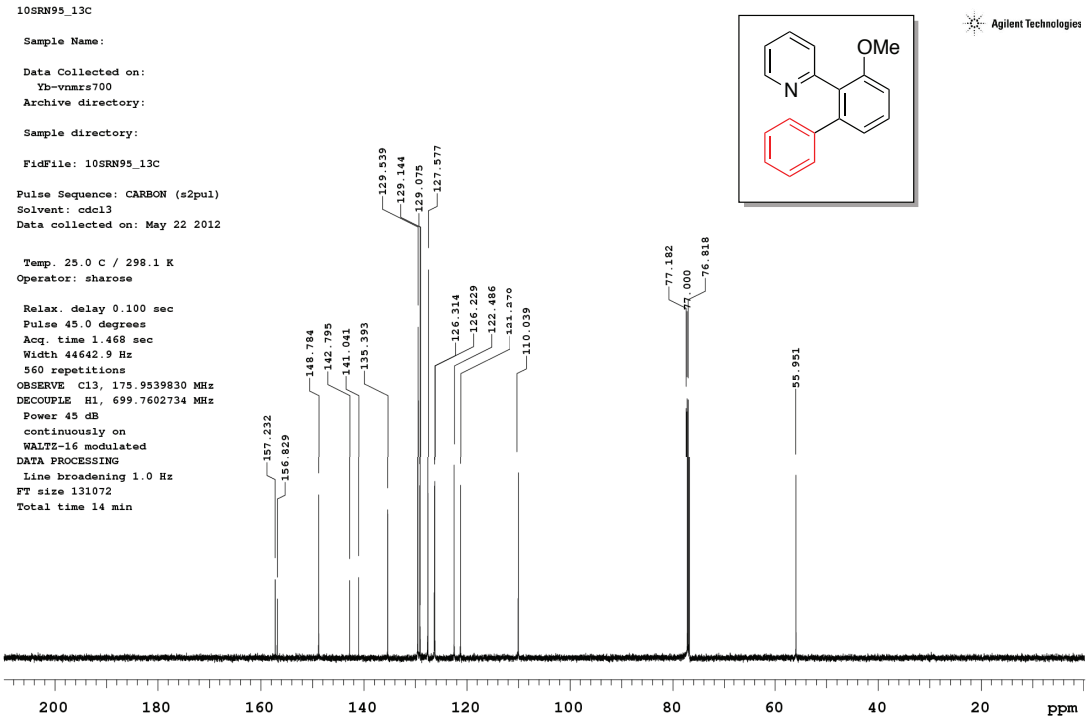

10SRN107_1H
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: 10SRN107_1H
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 25 2012
Operator: sharose
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6377.6 Hz
8 repetitions
OBSERVE H1, 399.5337065 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 0 min 40 sec
N
O
Agilent Technologies
9
8
7
6
5
4
3
2
1
ppm
8.79
3.00
2.99

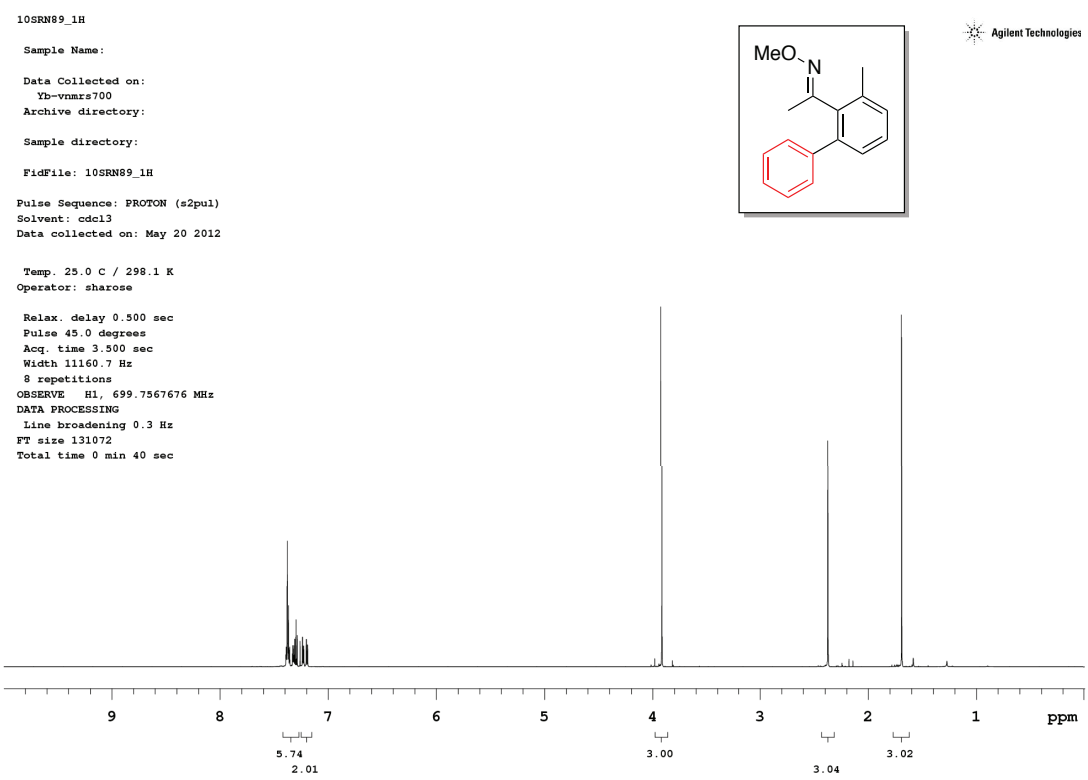

10SRN107_13C
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: 10SRN107_13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 25 2012
Operator: sharose
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.621 sec
Width 25000.0 Hz
192 repetitions
OBSERVE C13, 100.4628326 MHz
DECOUPLE H1, 399.5357121 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 8 min 42 sec
Agilent Technologies
N
O
171.332
139.930
138.673
135.741
129.305
128.474
128.364
127.695
127.581
127.411
77.319
77.000
76.685
37.939
34.529
200
180
160
140
120
100
80
60
40
20
ppm

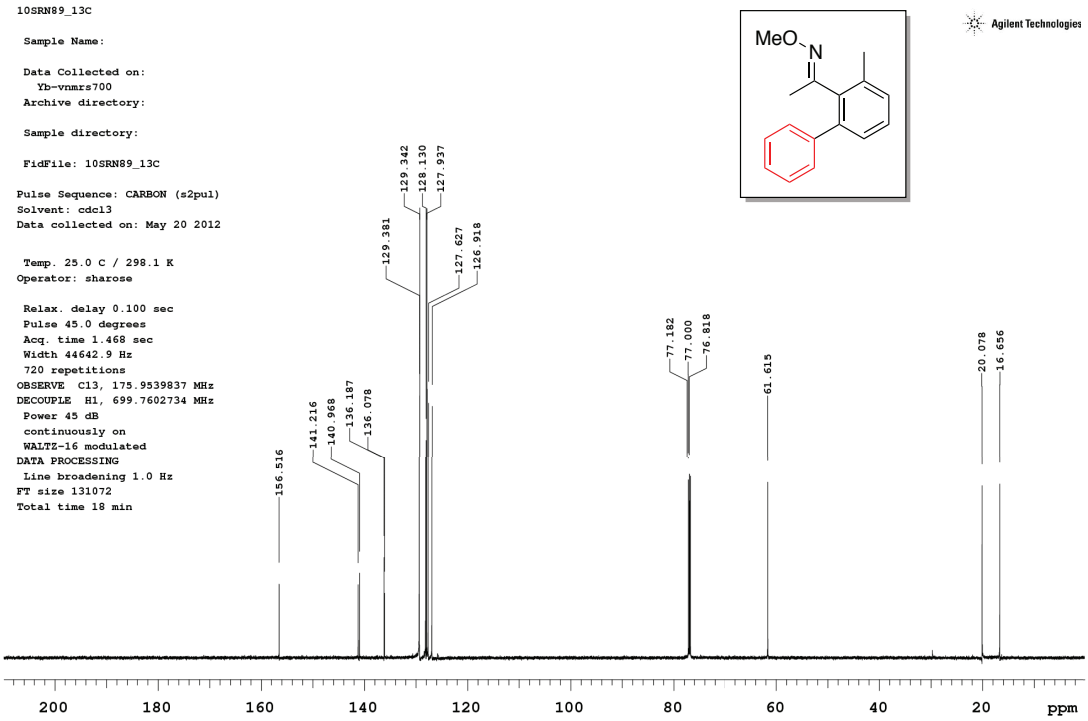

10SRN94_1H
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: 10SRN94_1H
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 22 2012
Temp. 25.0 C / 298.1 K
Operator: sharose
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 11160.7 Hz
8 repetitions
OBSERVE H1, 699.7567685 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 0 min 40 sec
N
Agilent Technologies
9
8
7
6
5
4
3
2
1
ppm
0.85
3.97
5.46
0.88
3.00

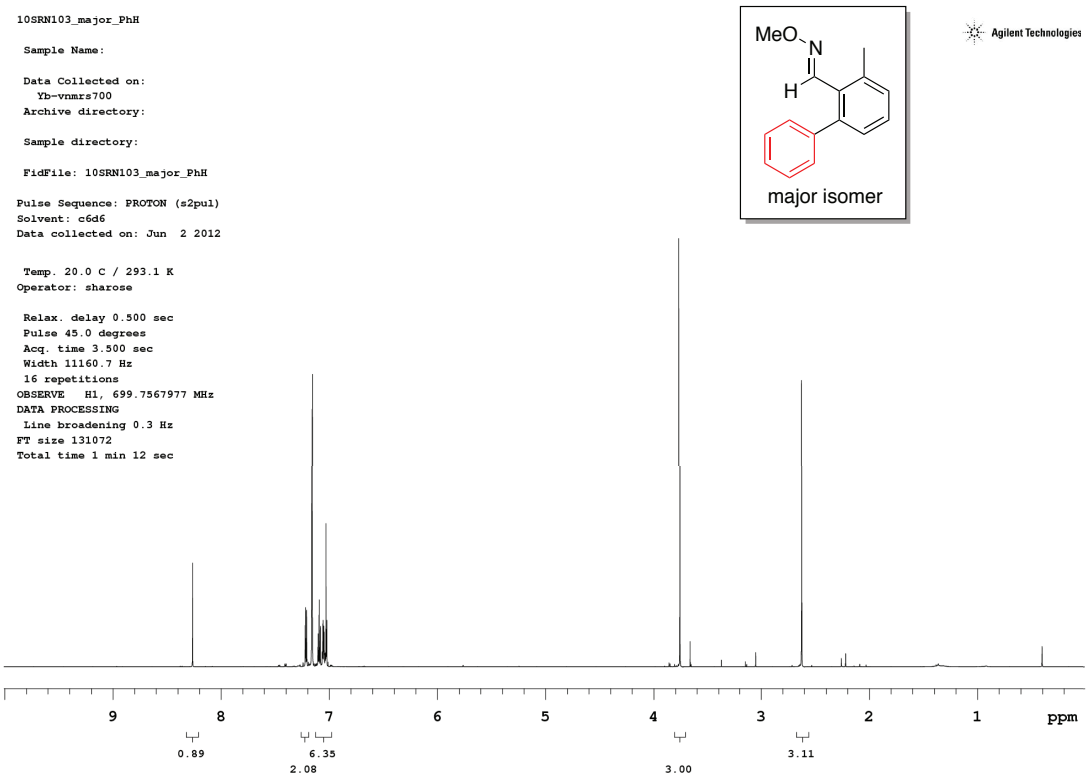

10SRN94_13C
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: 10SRN94_13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 22 2012
Temp. 25.0 C / 298.1 K
Operator: sharose
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.468 sec
Width 44642.9 Hz
176 repetitions
OBSERVE C13, 175.9539816 MHz
DECOUPLE H1, 699.7602734 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 4 min 35 sec
Agilent Technologies
N
159.593
148.835
141.657
141.227
139.330
136.678
135.645
129.625
129.388
128.002
127.573
127.553
126.187
125.590
121.243
77.182
77.000
76.818
20.454
200
180
160
140
120
100
80
60
40
20
ppm

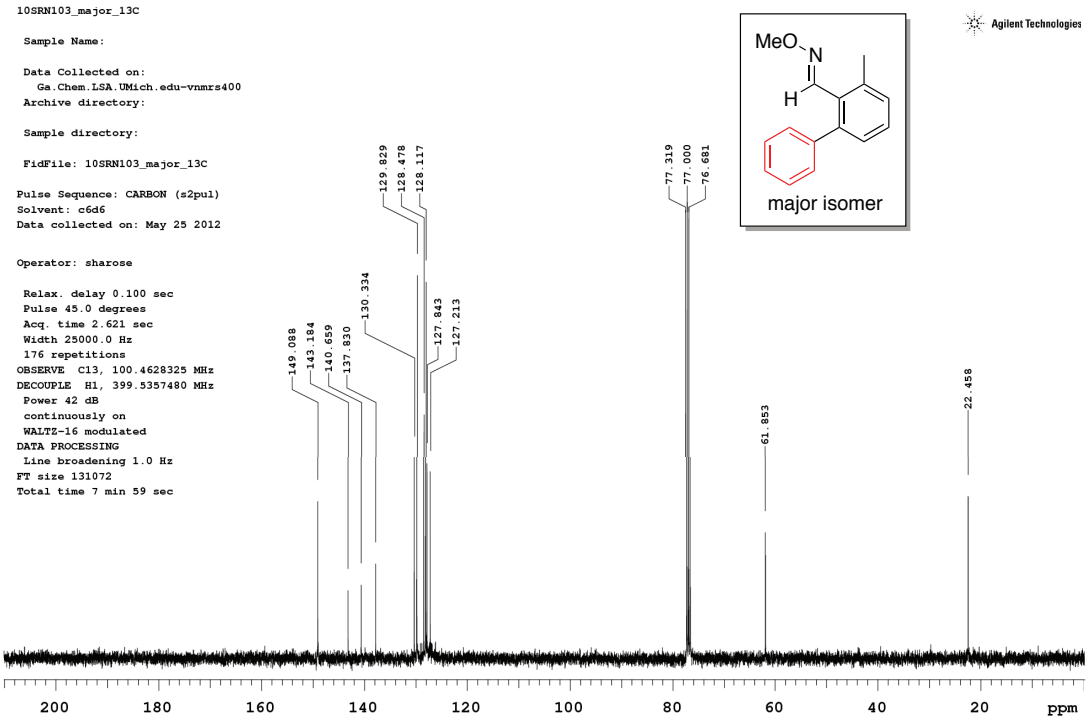

10SRN95_1H
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: 10SRN95_1H
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 22 2012
Temp. 25.0 C / 298.1 K
Operator: sharose
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 11160.7 Hz
8 repetitions
OBSERVE H1, 699.7567685 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 0 min 40 sec
OMe
N
Agilent Technologies
9
8
7
6
5
4
3
2
1
ppm
0.91
2.00
8.74
3.25

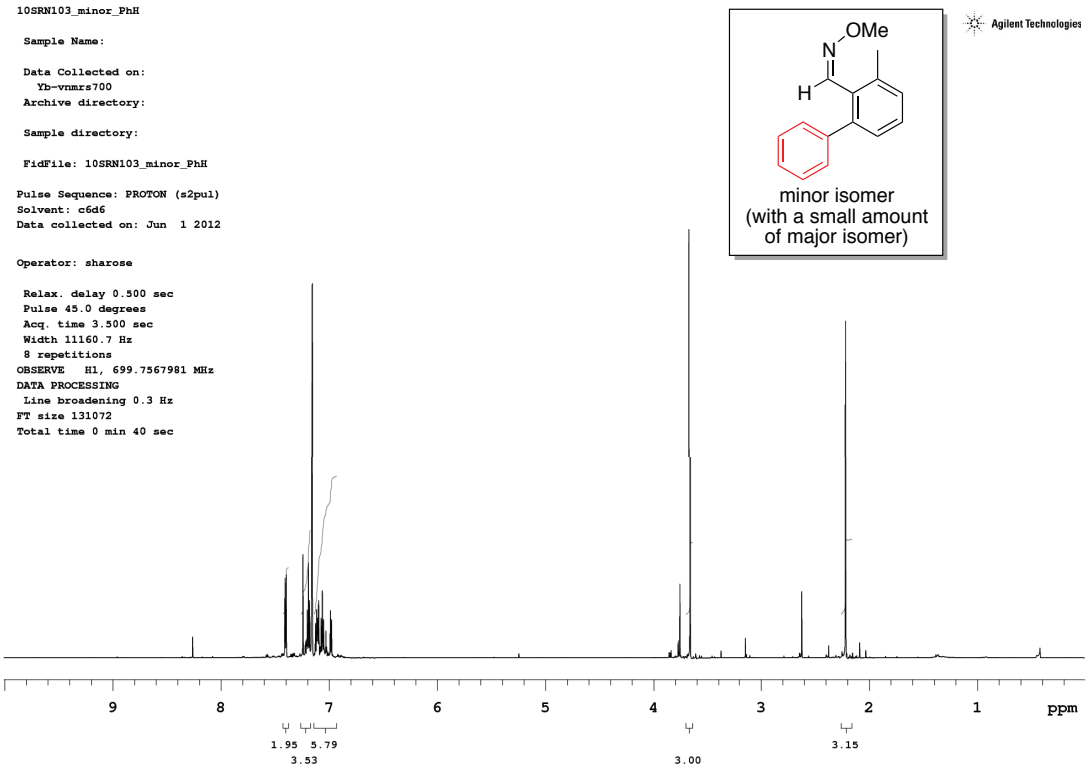

10SRN95_13C
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: 10SRN95_13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 22 2012
Temp. 25.0 C / 298.1 K
Operator: sharose
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.468 sec
Width 44642.9 Hz
560 repetitions
OBSERVE C13, 175.9539830 MHz
DECOUPLE H1, 699.7602734 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 14 min
OMe
N
Agilent Technologies
157.232
156.829
148.784
142.795
141.041
135.393
129.539
129.144
129.075
127.577
126.314
126.229
122.486
121.270
110.039
77.182
77.000
76.818
55.951
200
180
160
140
120
100
80
60
40
20
ppm

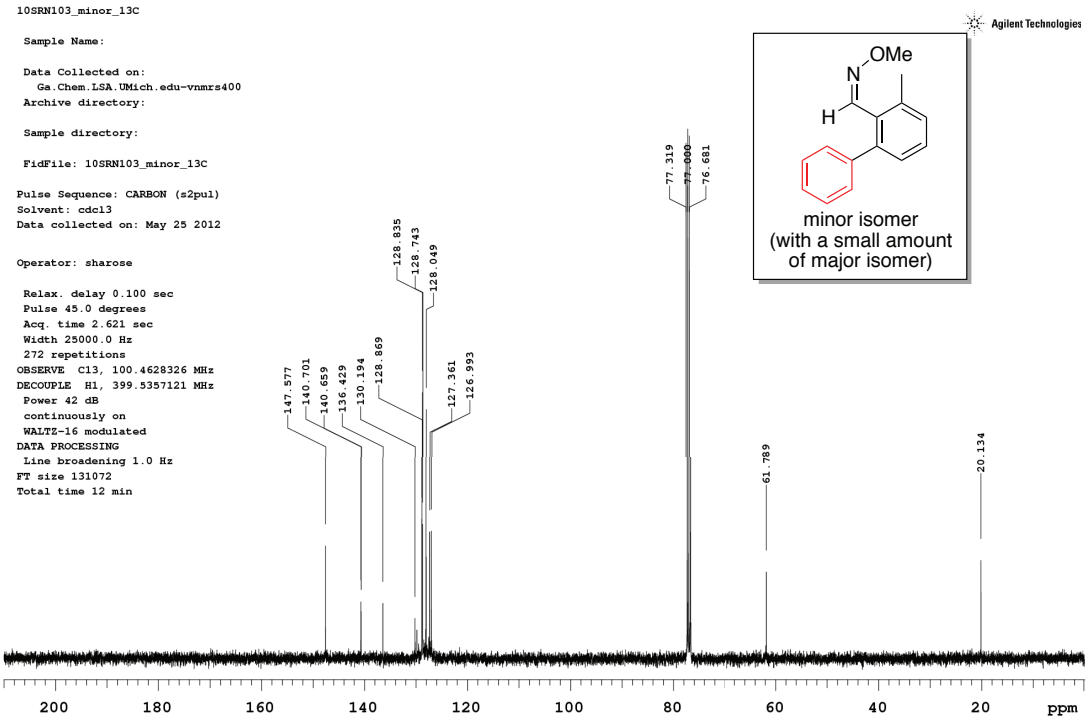

10SRN89_1H
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: 10SRN89_1H
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 20 2012
Temp. 25.0 C / 298.1 K
Operator: sharose
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 11160.7 Hz
8 repetitions
OBSERVE H1, 699.7567676 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 0 min 40 sec
MeO
N
Agilent Technologies
9
8
7
6
5
4
3
2
1
ppm
5.74
2.01
3.00
3.04
3.02

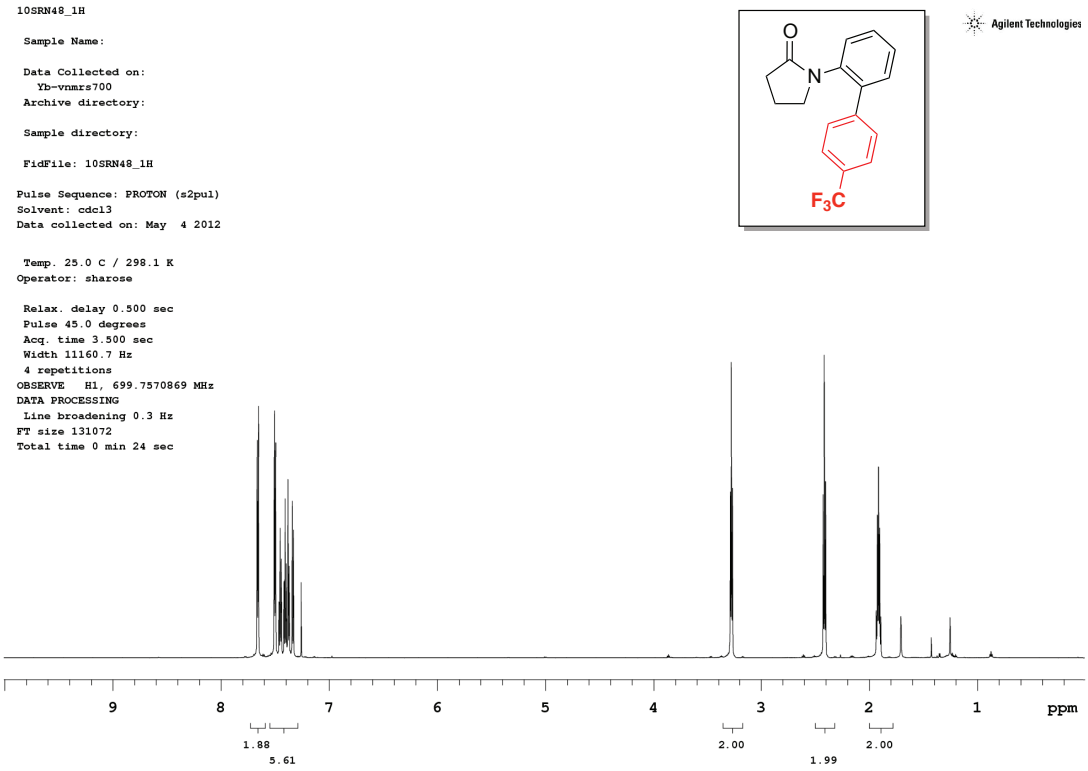

10SRN89_13C
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: 10SRN89_13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 20 2012
Temp. 25.0 C / 298.1 K
Operator: sharose
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.468 sec
Width 44642.9 Hz
720 repetitions
OBSERVE C13, 175.9539837 MHz
DECOUPLE H1, 699.7602734 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 18 min
MeO
N
Agilent Technologies
156.516
141.216
140.968
136.187
136.078
129.342
129.381
128.130
127.937
127.627
126.918
77.182
77.000
76.818
61.615
20.078
16.656
200
180
160
140
120
100
80
60
40
20
ppm

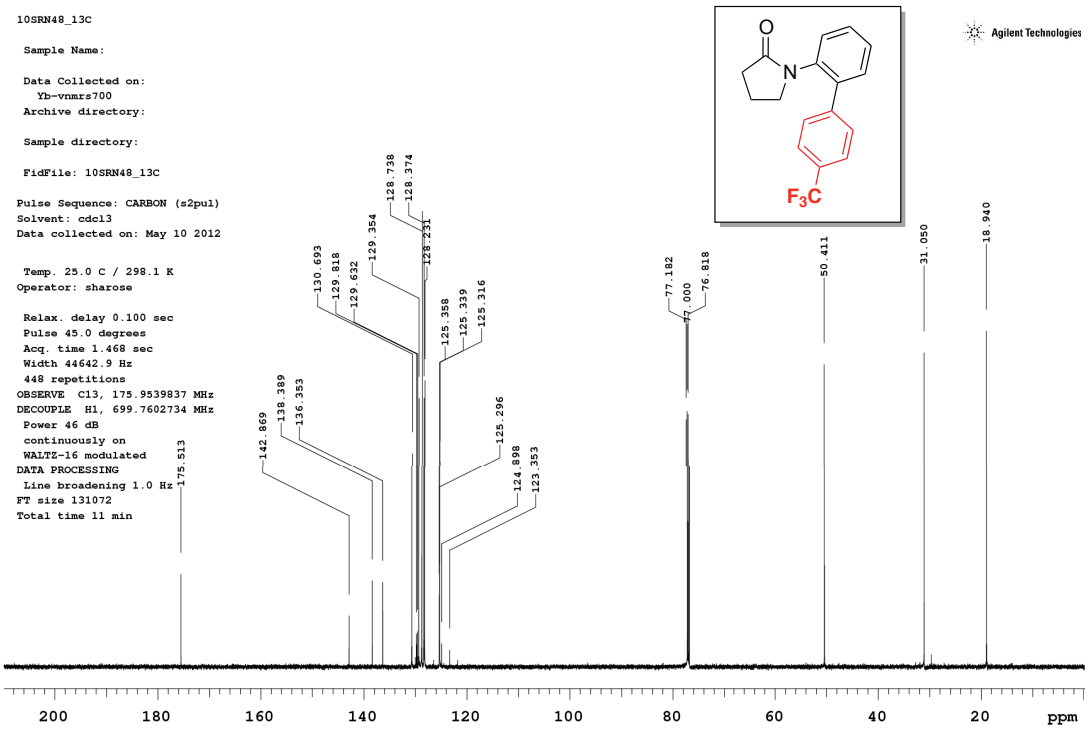

10SRN103_major_PhH
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: 10SRN103_major_PhH
Pulse Sequence: PROTON (s2pul)
Solvent: c6d6
Data collected on: Jun 2 2012
Temp. 20.0 C / 293.1 K
Operator: sharose
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 11160.7 Hz
16 repetitions
OBSERVE H1, 699.7567977 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 1 min 12 sec
MeO
N
H
major isomer
Agilent Technologies
9
8
7
6
5
4
3
2
1
ppm
0.89
6.35
2.08
3.00
3.11

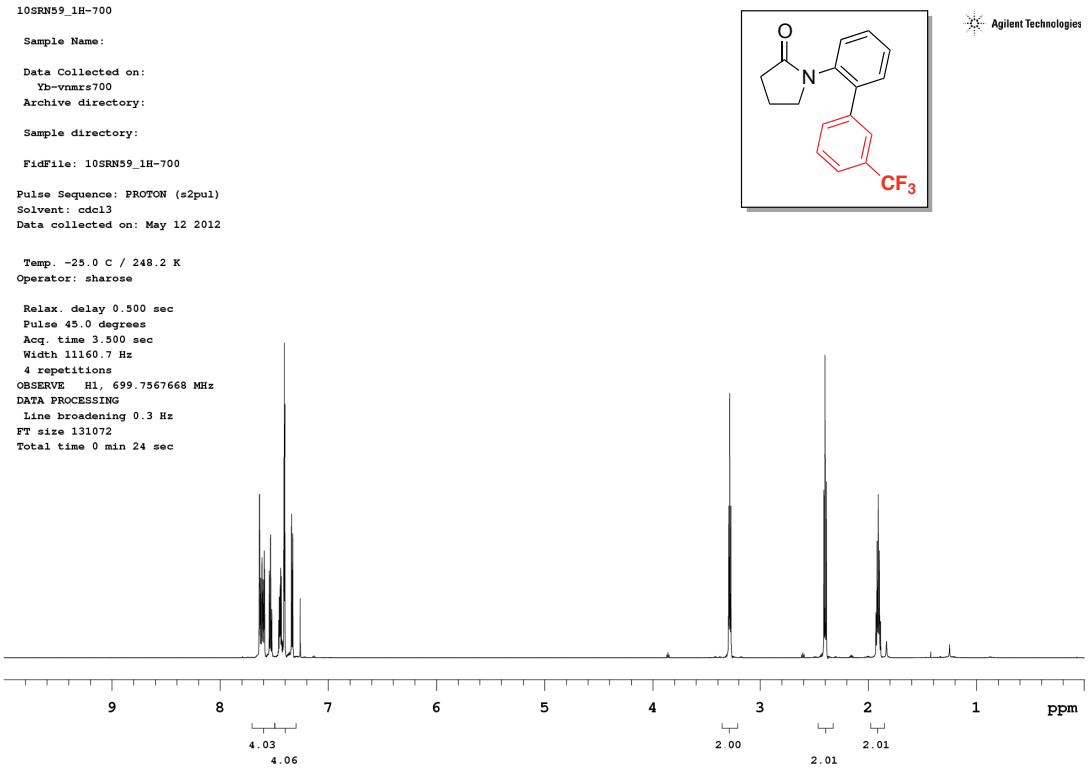

10SRN103_major_13C
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: 10SRN103_major_13C
Pulse Sequence: CARBON (s2pul)
Solvent: c6d6
Data collected on: May 25 2012
Operator: sharose
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.621 sec
Width 25000.0 Hz
176 repetitions
OBSERVE C13, 100.4628325 MHz
DECOUPLE H1, 399.5357480 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 7 min 59 sec
MeO
N
H
major isomer
Agilent Technologies
149.088
143.184
140.659
137.830
130.334
129.829
128.478
128.117
127.843
127.213
77.319
77.000
76.681
61.853
22.458
200
180
160
140
120
100
80
60
40
20
ppm

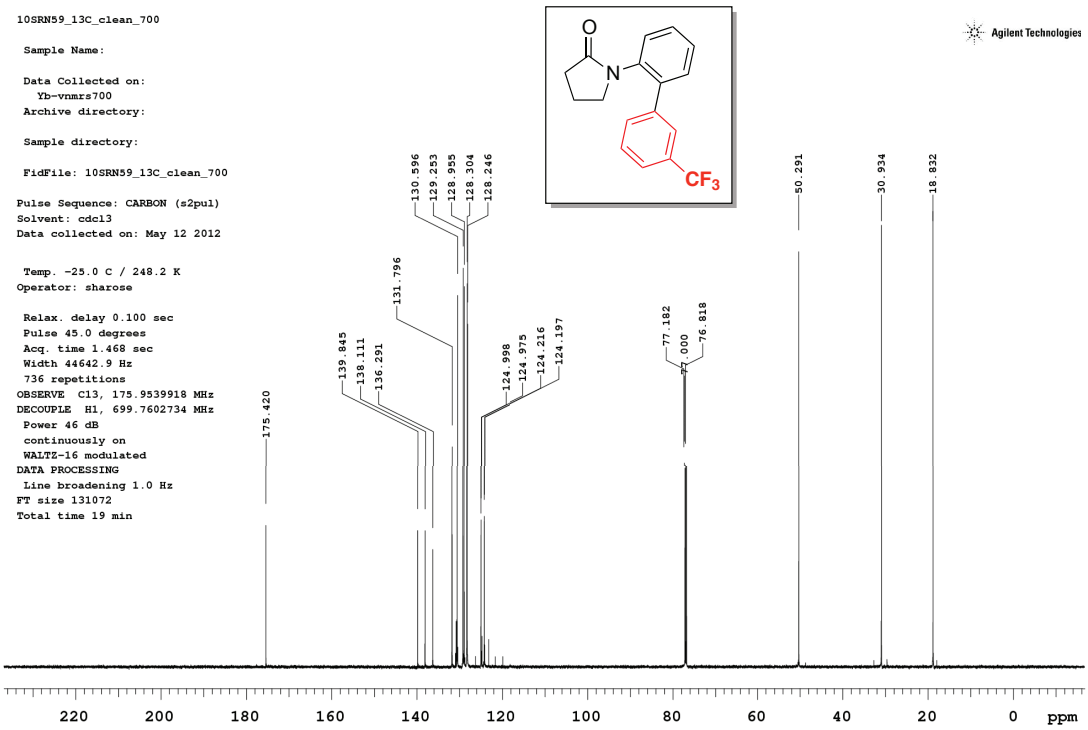

10SRN103_minor_PhH
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: 10SRN103_minor_PhH
Pulse Sequence: PROTON (s2pul)
Solvent: c6d6
Data collected on: Jun 1 2012
Operator: sharose
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 11160.7 Hz
8 repetitions
OBSERVE H1, 699.7567981 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 0 min 40 sec
OMe
N
H
minor isomer
(with a small amount
of major isomer)
Agilent Technologies
9
8
7
6
5
4
3
2
1
ppm
1.95
5.79
3.53
3.00
3.15

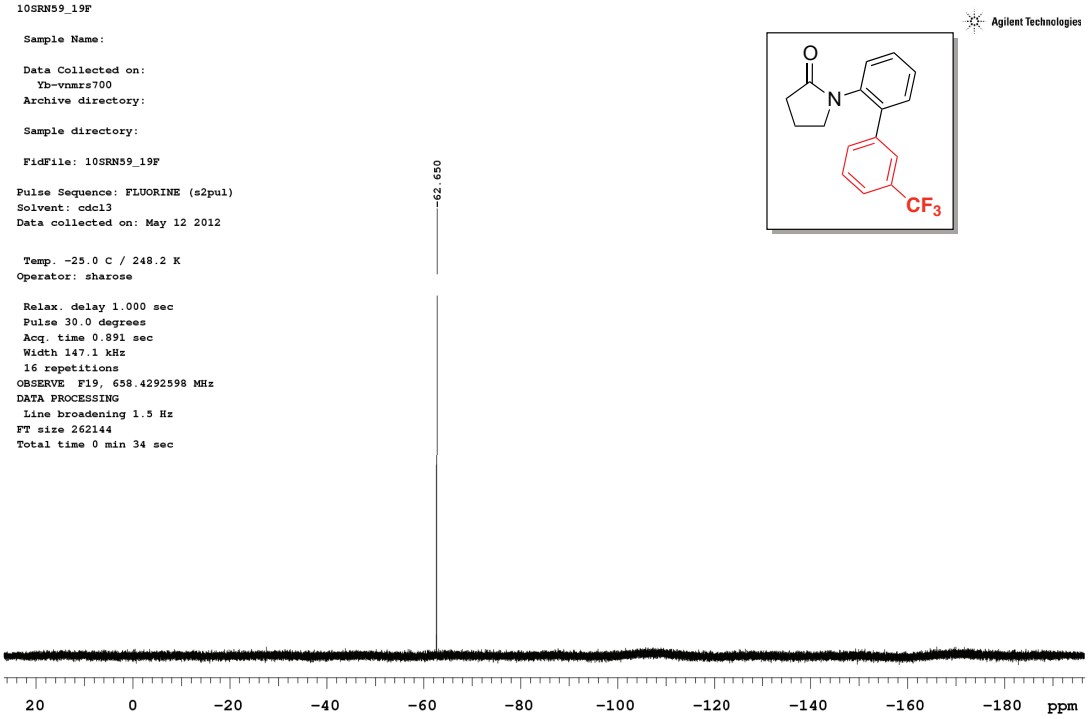

10SRN103_minor_13C
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: 10SRN103_minor_13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 25 2012
Operator: sharose
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.621 sec
Width 25000.0 Hz
272 repetitions
OBSERVE C13, 100.4628326 MHz
DECOUPLE H1, 399.5357121 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 12 min
Agilent Technologies
OMe
N
H
minor isomer
(with a small amount
of major isomer)
147.577
140.701
140.659
136.429
130.194
128.869
128.835
128.743
128.049
127.361
126.993
77.319
77.000
76.681
61.789
20.134
200
180
160
140
120
100
80
60
40
20
ppm

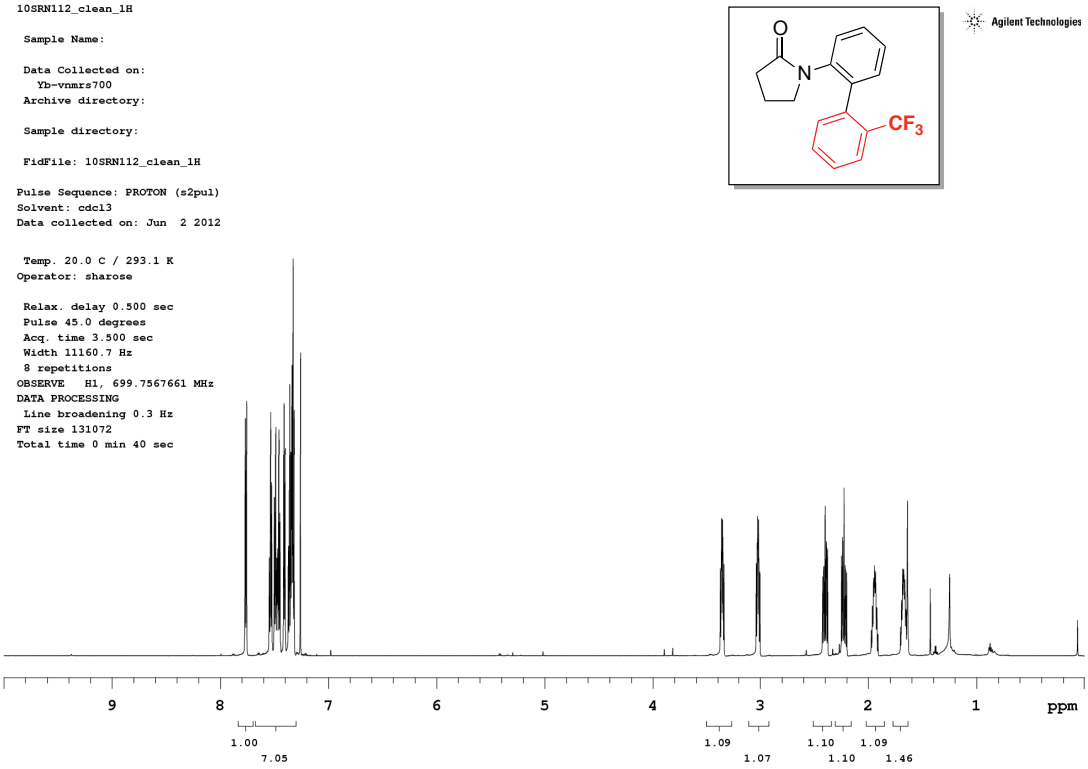

10SRN48_1H
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: 10SRN48_1H
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 4 2012
Temp. 25.0 C / 298.1 K
Operator: sharose
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 11160.7 Hz
4 repetitions
OBSERVE H1, 699.7570869 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 0 min 24 sec
O
N
F3C
Agilent Technologies
9
8
7
6
5
4
3
2
1
ppm
1.88
5.61
2.00
1.99
2.00

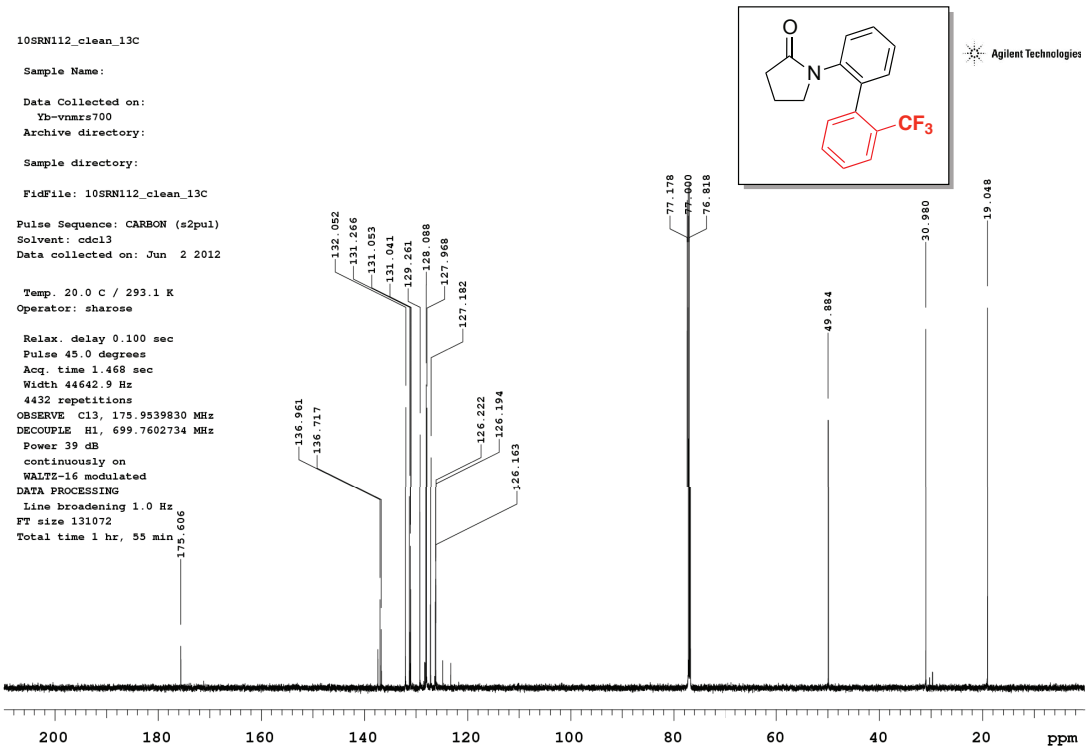

10SRN48_13C
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: 10SRN48_13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 10 2012
Temp. 25.0 C / 298.1 K
Operator: sharose
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.468 sec
Width 44642.9 Hz
448 repetitions
OBSERVE C13, 175.9539837 MHz
DECOUPLE H1, 699.7602734 MHz
Power 46 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 11 min
O
N
F3C
Agilent Technologies
175.513
142.869
138.389
136.353
130.693
129.818
129.632
129.354
128.738
128.374
128.231
125.358
125.339
125.316
125.296
124.898
123.353
77.182
77.000
76.818
50.411
31.050
18.940
200
180
160
140
120
100
80
60
40
20
ppm

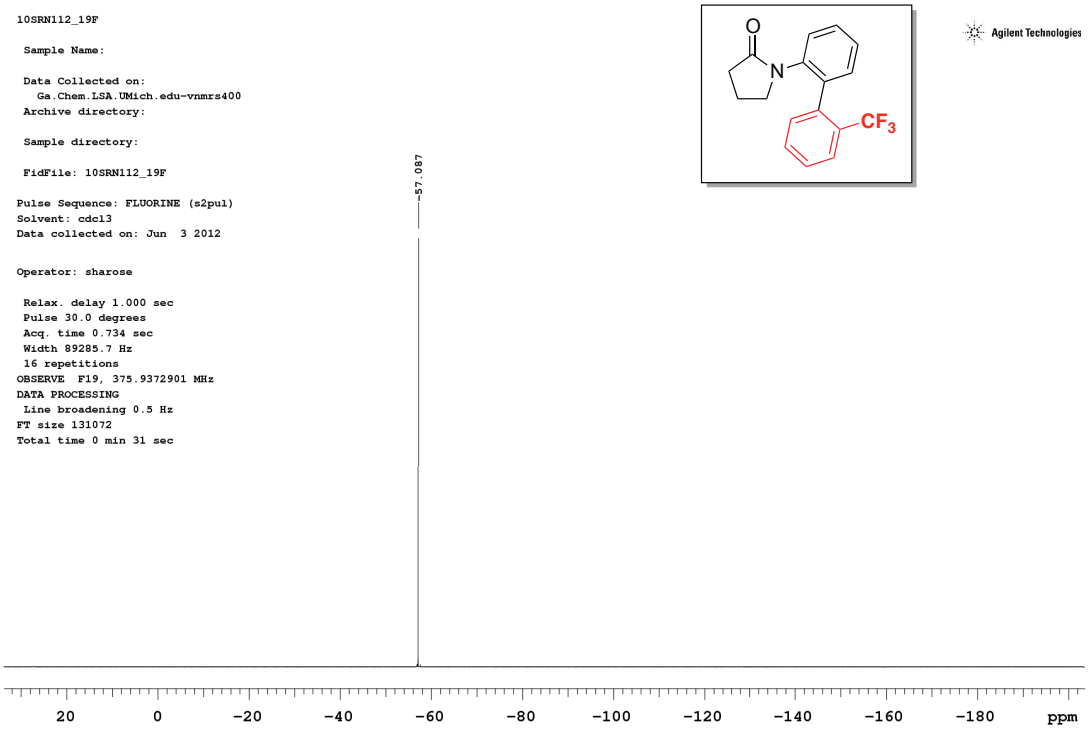

10SRN59_1H-700
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: 10SRN59_1H-700
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 12 2012
Temp. -25.0 C / 248.2 K
Operator: sharose
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 11160.7 Hz
4 repetitions
OBSERVE H1, 699.7567668 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 0 min 24 sec
O
N
CF3
Agilent Technologies
9
8
7
6
5
4
3
2
1
ppm
4.03
4.06
2.00
2.01
2.01

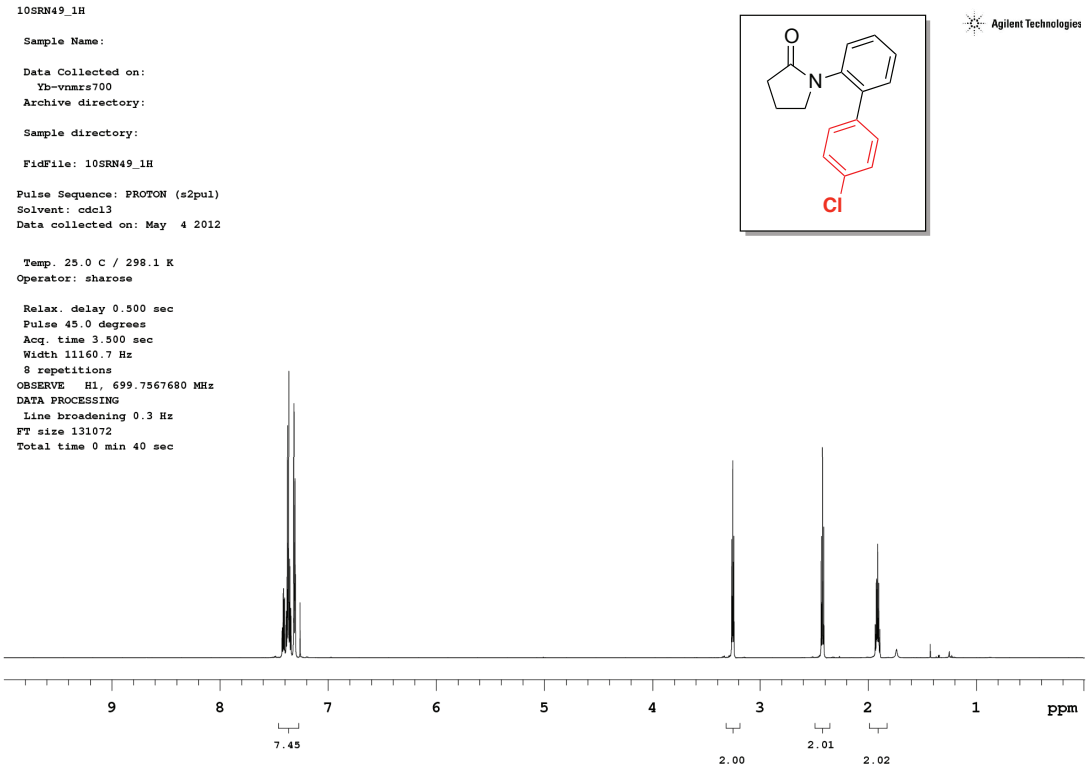

10SRN59_13C_clean_700
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: 10SRN59_13C_clean_700
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 12 2012
Temp. -25.0 C / 248.2 K
Operator: sharose
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.468 sec
Width 44642.9 Hz
736 repetitions
OBSERVE C13, 175.9539918 MHz
DECOUPLE H1, 699.7602734 MHz
Power 46 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 19 min
Agilent Technologies
O
N
CF3
175.420
139.845
138.111
136.291
131.796
130.596
129.253
128.955
128.304
128.246
124.998
124.975
124.216
124.197
77.182
77.000
76.818
50.291
30.934
18.832
220
200
180
160
140
120
100
80
60
40
20
0
ppm

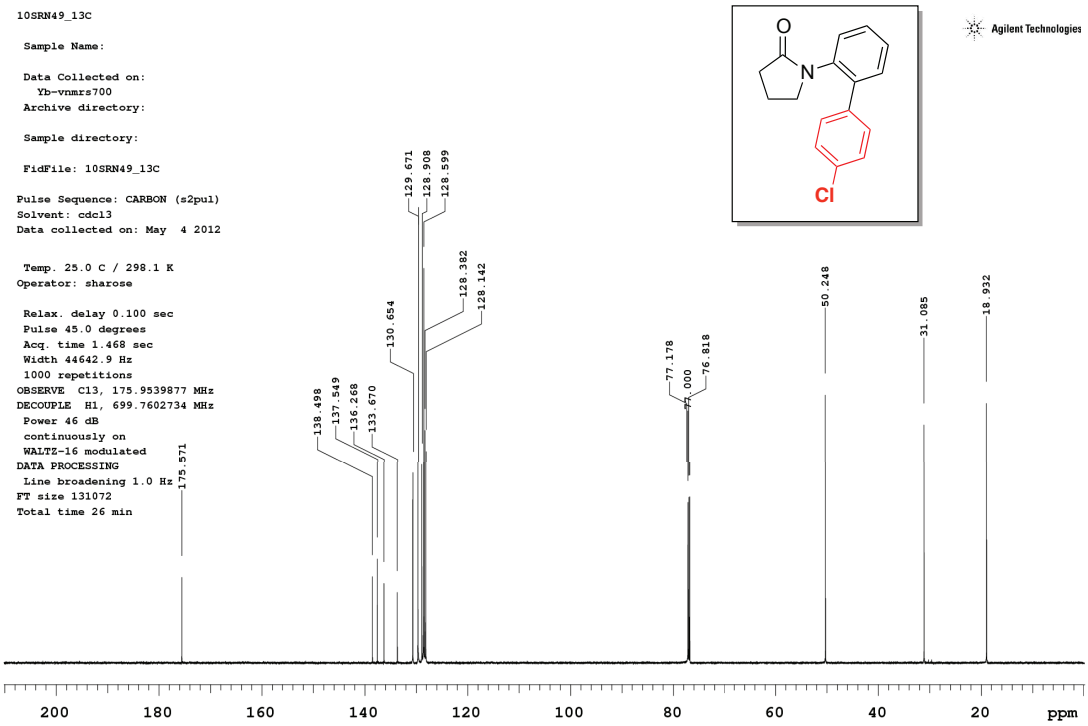

10SRN59_19F
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: 10SRN59_19F
Pulse Sequence: FLUORINE (s2pul)
Solvent: cdcl3
Data collected on: May 12 2012
Temp. -25.0 C / 248.2 K
Operator: sharose
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 0.891 sec
Width 147.1 kHz
16 repetitions
OBSERVE F19, 658.4292598 MHz
DATA PROCESSING
Line broadening 1.5 Hz
FT size 262144
Total time 0 min 34 sec
-62.650
Agilent Technologies
O
N
CF3
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
ppm

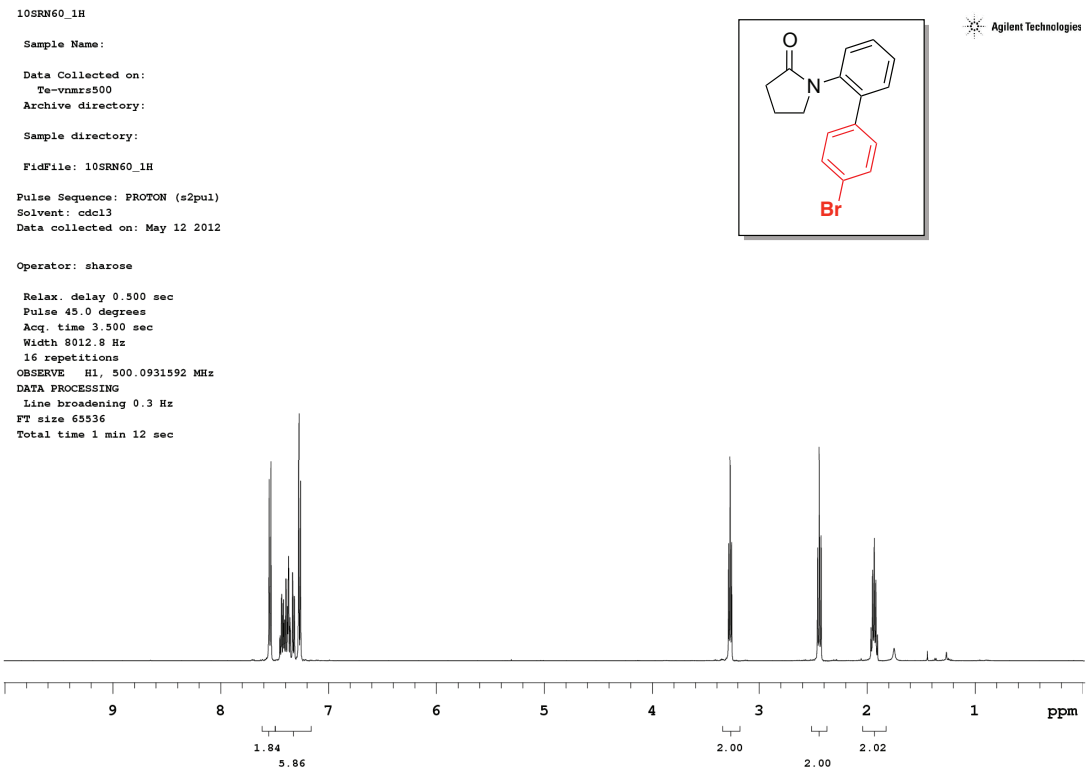

10SRN112_clean_1H
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: 10SRN112_clean_1H
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jun 2 2012
Temp. 20.0 C / 293.1 K
Operator: sharose
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 11160.7 Hz
8 repetitions
OBSERVE H1, 699.7567661 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 0 min 40 sec
O
N
CF3
Agilent Technologies
9
8
7
6
5
4
3
2
1
ppm
1.00
7.05
1.09
1.07
1.10
1.10
1.09
1.46

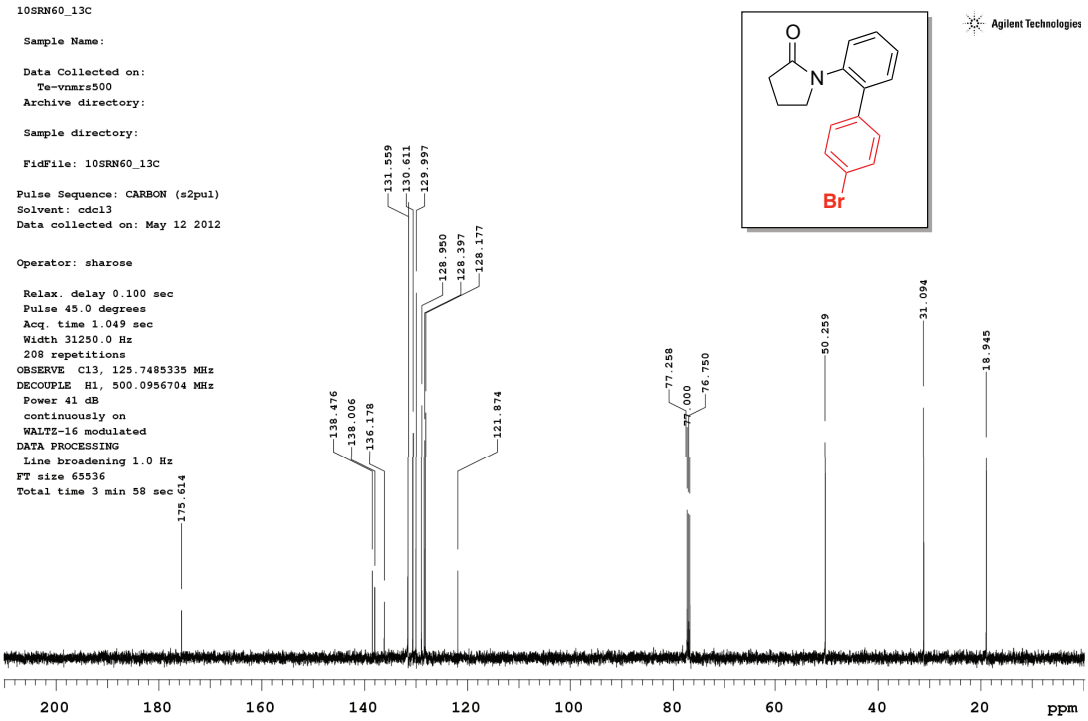

10SRN112_clean_13C
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: 10SRN112_clean_13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jun 2 2012
Temp. 20.0 C / 293.1 K
Operator: sharose
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.468 sec
Width 44642.9 Hz
4432 repetitions
OBSERVE C13, 175.9539830 MHz
DECOUPLE H1, 699.7602734 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 55 min
O
N
CF3
Agilent Technologies
175.606
136.961
136.717
132.052
131.266
131.053
131.041
129.261
128.088
127.968
127.182
126.222
126.194
126.163
77.178
77.000
76.818
49.884
30.980
19.048
200
180
160
140
120
100
80
60
40
20
ppm

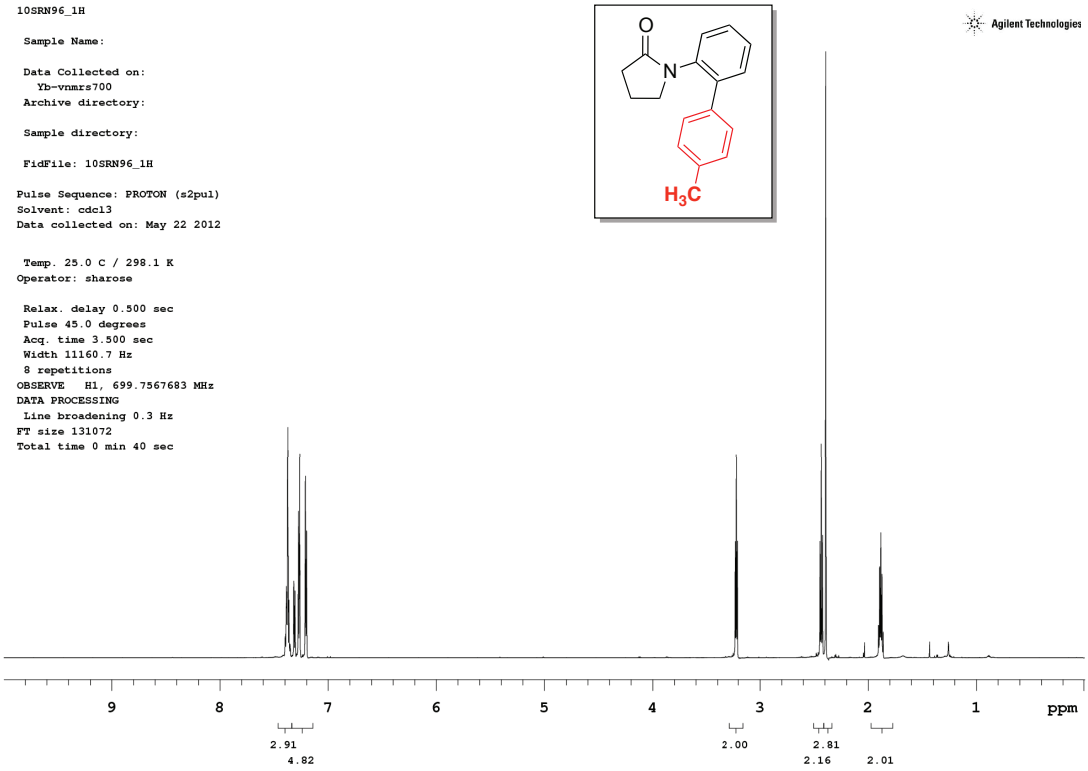

10SRN112_19F
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: 10SRN112_19F
Pulse Sequence: FLUORINE (s2pul)
Solvent: cdcl3
Data collected on: Jun 3 2012
Operator: sharose
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 0.734 sec
Width 89285.7 Hz
16 repetitions
OBSERVE F19, 375.9372901 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 0 min 31 sec
-57.087
O
N
CF3
Agilent Technologies
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
ppm

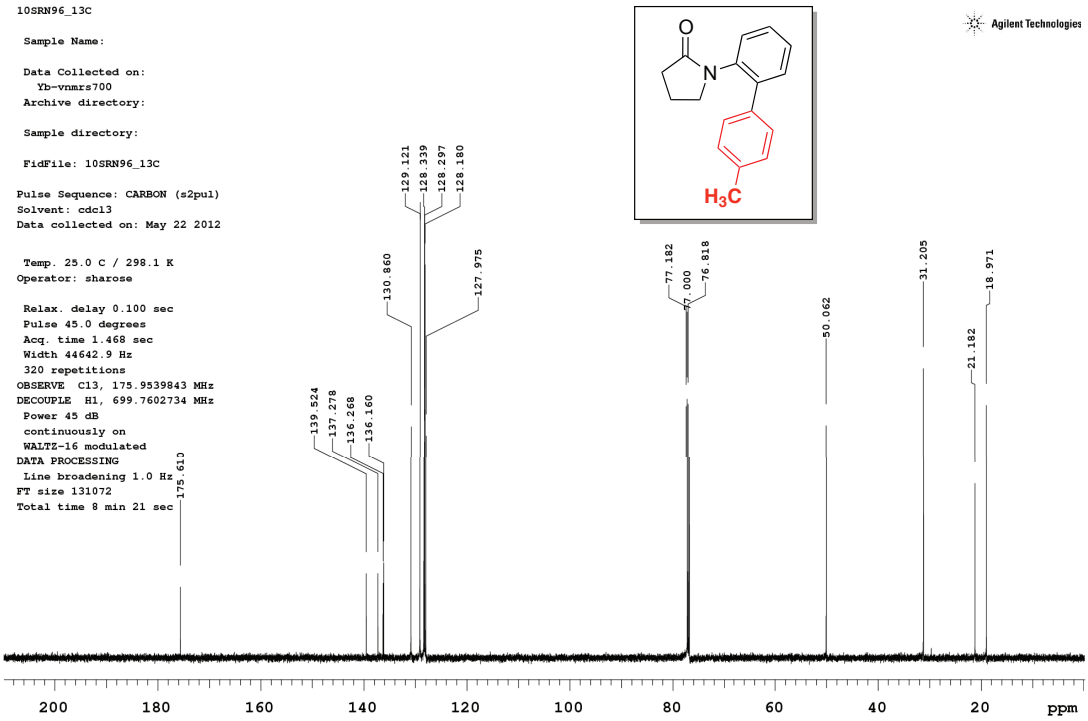

10SRN49_1H
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: 10SRN49_1H
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 4 2012
Temp. 25.0 C / 298.1 K
Operator: sharose
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 11160.7 Hz
8 repetitions
OBSERVE H1, 699.7567680 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 0 min 40 sec
O
N
Cl
Agilent Technologies
9
8
7
6
5
4
3
2
1
ppm
7.45
2.00
2.01
2.02

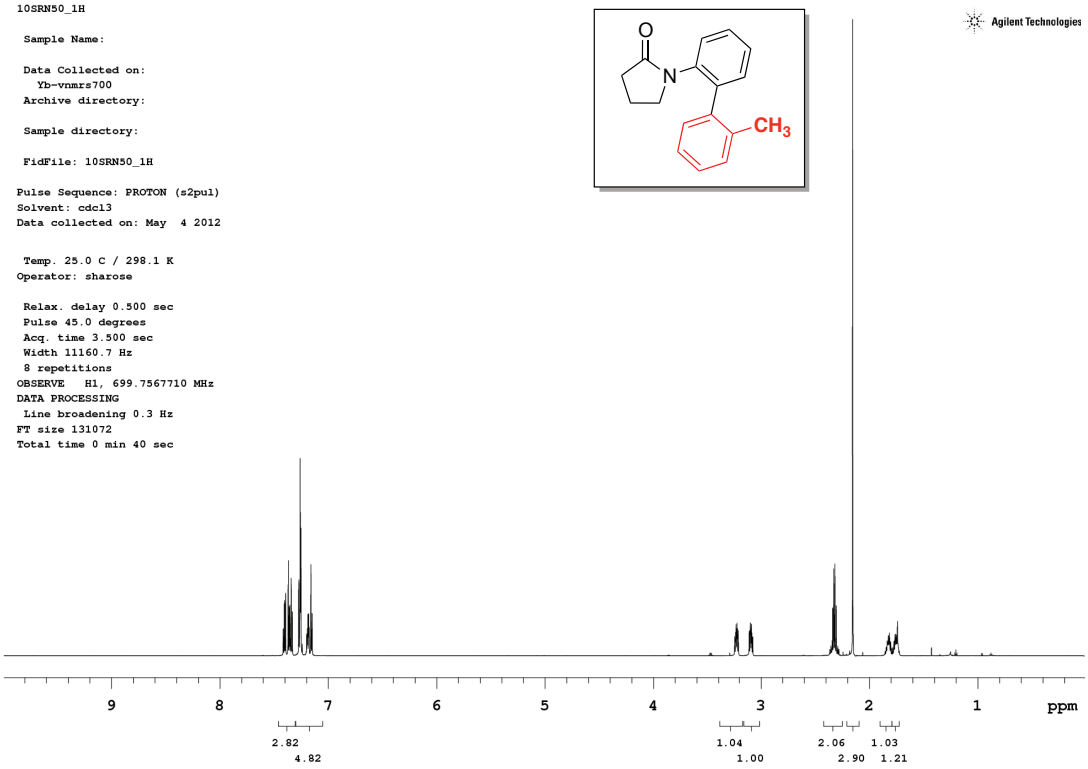

10SRN49_13C
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: 10SRN49_13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 4 2012
Temp. 25.0 C / 298.1 K
Operator: sharose
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.468 sec
Width 44642.9 Hz
1000 repetitions
OBSERVE C13, 175.9539877 MHz
DECOUPLE H1, 699.7602734 MHz
Power 46 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 26 min
O
N
Cl
Agilent Technologies
175.571
138.498
137.549
136.268
133.670
130.654
129.671
128.908
128.599
128.382
128.142
77.178
77.000
76.818
50.248
31.085
18.932
200
180
160
140
120
100
80
60
40
20
ppm

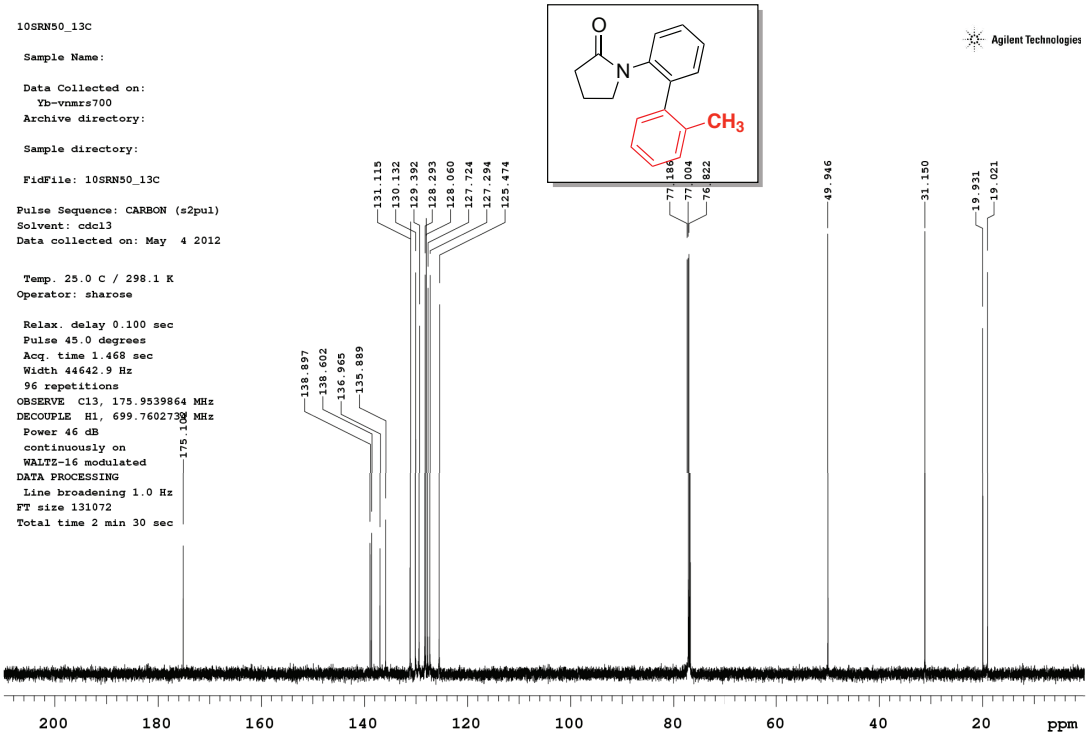

10SRN60_1H
Sample Name:
Data Collected on:
Te-vnmrs500
Archive directory:
Sample directory:
FidFile: 10SRN60_1H
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 12 2012
Operator: sharose
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 8012.8 Hz
16 repetitions
OBSERVE H1, 500.0931592 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min 12 sec
O
N
Br
Agilent Technologies
9
8
7
6
5
4
3
2
1
ppm
1.84
5.86
2.00
2.00
2.02

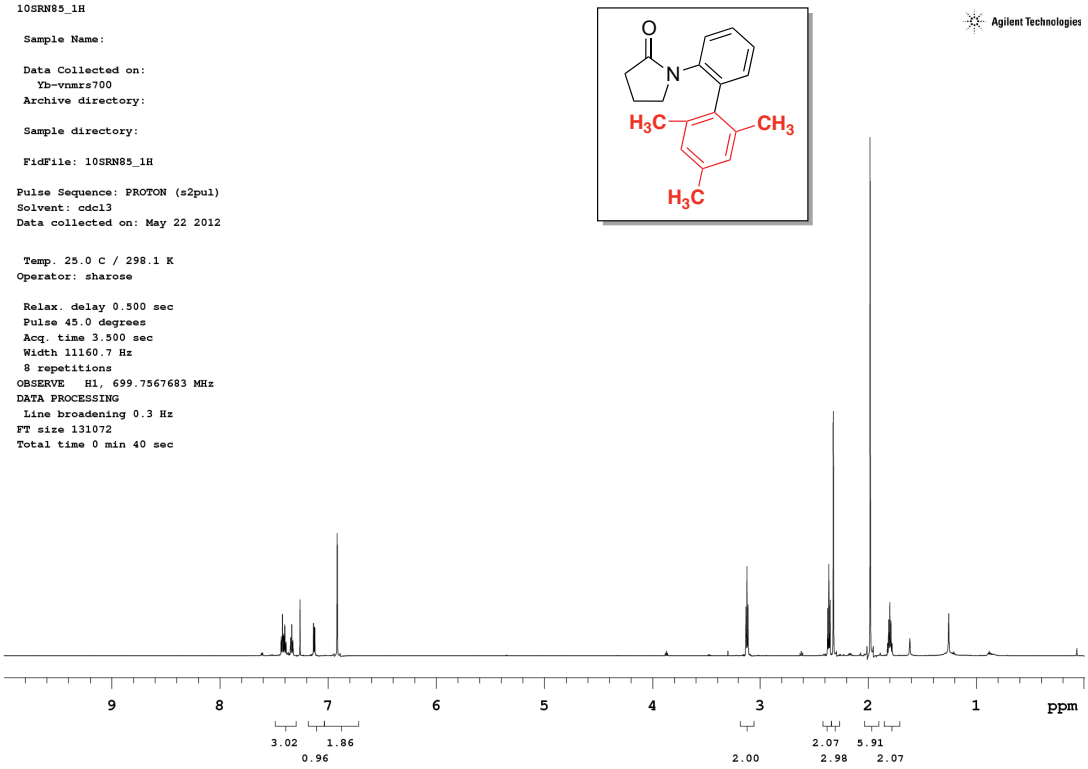

10SRN60_13C
Sample Name:
Data Collected on:
Te-vnmrs500
Archive directory:
Sample directory:
FidFile: 10SRN60_13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 12 2012
Operator: sharose
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.049 sec
Width 31250.0 Hz
208 repetitions
OBSERVE C13, 125.7485335 MHz
DECOUPLE H1, 500.0956704 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 3 min 58 sec
Agilent Technologies
O
N
Br
175.614
138.476
138.006
136.178
131.559
130.611
129.997
128.950
128.397
128.177
121.874
77.258
77.000
76.750
50.259
31.094
18.945
200
180
160
140
120
100
80
60
40
20
ppm

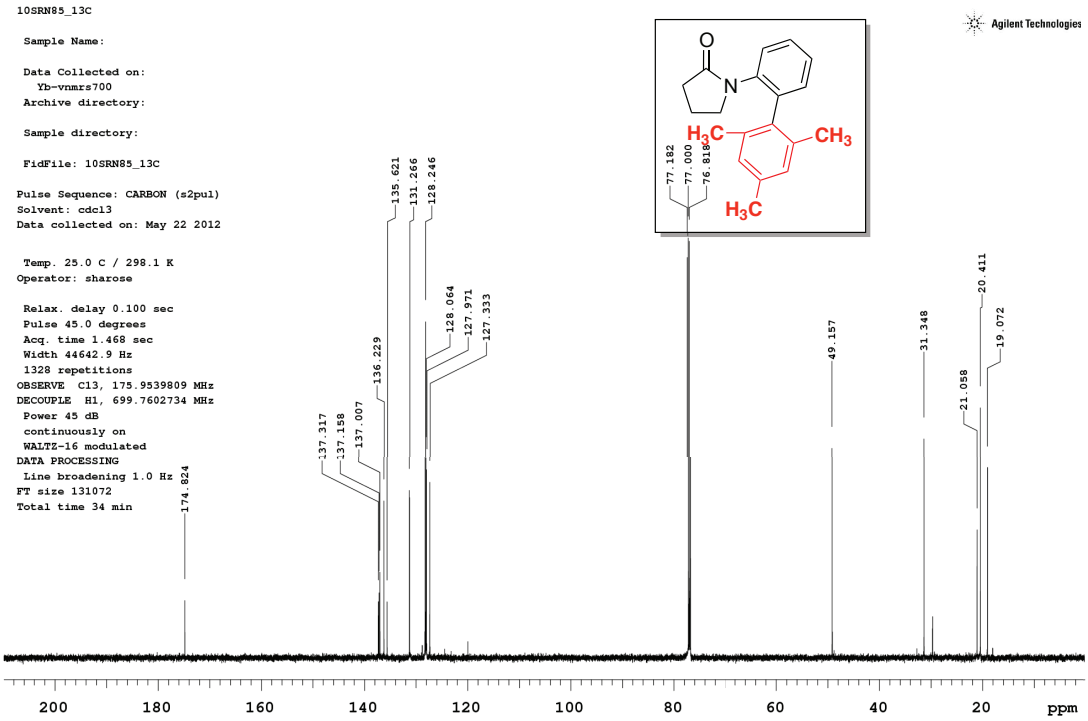

10SRN96_1H
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: 10SRN96_1H
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 22 2012
Temp. 25.0 C / 298.1 K
Operator: sharose
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 11160.7 Hz
8 repetitions
OBSERVE H1, 699.7567683 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 0 min 40 sec
O
N
H3C
Agilent Technologies
9
8
7
6
5
4
3
2
1
ppm
2.91
4.82
2.00
2.81
2.16
2.01

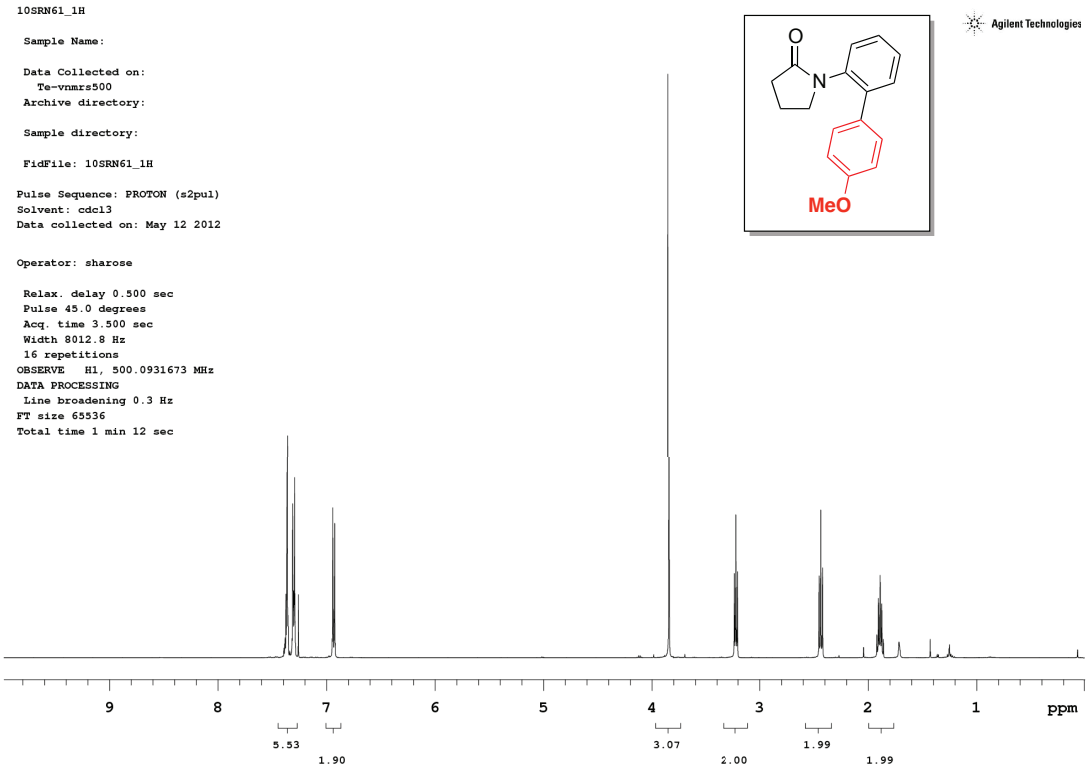

10SRN96_13C
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: 10SRN96_13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 22 2012
Temp. 25.0 C / 298.1 K
Operator: sharose
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.468 sec
Width 44642.9 Hz
320 repetitions
OBSERVE C13, 175.9539843 MHz
DECOUPLE H1, 699.7602734 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 8 min 21 sec
O
N
H3C
Agilent Technologies
175.610
139.524
137.278
136.268
136.160
130.860
129.121
128.339
128.297
128.180
127.975
77.182
77.000
76.818
50.062
31.205
21.182
18.971
200
180
160
140
120
100
80
60
40
20
ppm

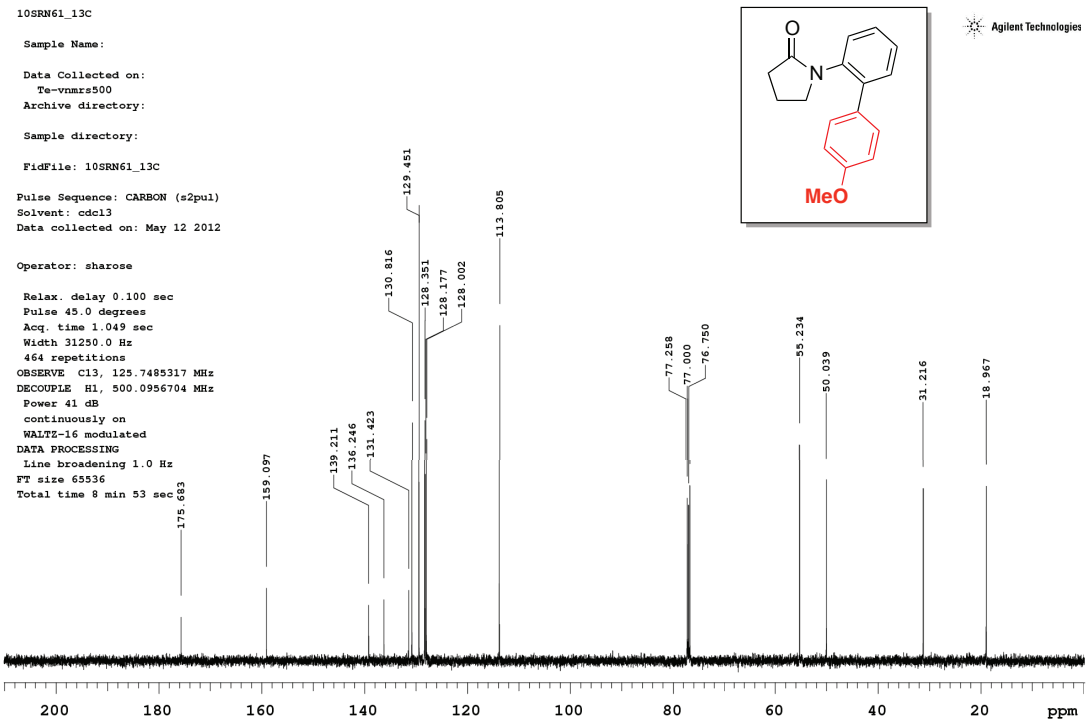

10SRN50_1H
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: 10SRN50_1H
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 4 2012
Temp. 25.0 C / 298.1 K
Operator: sharose
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 11160.7 Hz
8 repetitions
OBSERVE H1, 699.7567710 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 0 min 40 sec
O
N
CH3
Agilent Technologies
9
8
7
6
5
4
3
2
1
ppm
2.82
4.82
1.04
1.00
2.06
2.90
1.03
1.21


10SRN50_13C
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: 10SRN50_13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 4 2012
Temp. 25.0 C / 298.1 K
Operator: sharose
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.468 sec
Width 44642.9 Hz
96 repetitions
OBSERVE C13, 175.9539864 MHz
DECOUPLE H1, 699.7602738 MHz
Power 46 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 2 min 30 sec
Agilent Technologies
O
N
CH3
175.104
138.897
138.602
136.965
135.889
131.115
130.132
129.392
128.293
128.060
127.724
127.294
125.474
77.186
77.004
76.822
49.946
31.150
19.931
19.021
200
180
160
140
120
100
80
60
40
20
ppm


10SRN85_1H
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: 10SRN85_1H
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 22 2012
Temp. 25.0 C / 298.1 K
Operator: sharose
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 11160.7 Hz
8 repetitions
OBSERVE H1, 699.7567683 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 0 min 40 sec
Agilent Technologies
O
N
H3C
CH3
H3C
9
8
7
6
5
4
3
2
1
ppm
3.02
1.86
0.96
2.00
2.07
2.98
5.91
2.07


10SRN85_13C
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: 10SRN85_13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 22 2012
Temp. 25.0 C / 298.1 K
Operator: sharose
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.468 sec
Width 44642.9 Hz
1328 repetitions
OBSERVE C13, 175.9539809 MHz
DECOUPLE H1, 699.7602734 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 34 min
Agilent Technologies
O
N
H3C
CH3
H3C
77.182
77.000
76.818
174.824
137.317
137.158
137.007
136.229
135.621
131.266
128.246
128.064
127.971
127.333
49.157
31.348
21.058
20.411
19.072
200
180
160
140
120
100
80
60
40
20
ppm


10SRN61_1H
Sample Name:
Data Collected on:
Te-vnmrs500
Archive directory:
Sample directory:
FidFile: 10SRN61_1H
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 12 2012
Operator: sharose
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 8012.8 Hz
16 repetitions
OBSERVE H1, 500.0931673 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min 12 sec
O
N
MeO
Agilent Technologies
9
8
7
6
5
4
3
2
1
ppm
5.53
1.90
3.07
2.00
1.99
1.99


10SRN61_13C
Sample Name:
Data Collected on:
Te-vnmrs500
Archive directory:
Sample directory:
FidFile: 10SRN61_13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 12 2012
Operator: sharose
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.049 sec
Width 31250.0 Hz
464 repetitions
OBSERVE C13, 125.7485317 MHz
DECOUPLE H1, 500.0956704 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 8 min 53 sec
175.683
159.097
139.211
136.246
131.423
130.816
129.451
128.351
128.177
128.002
113.805
77.258
77.000
76.750
55.234
50.039
31.216
18.967
MeO
Agilent Technologies
200
180
160
140
120
100
80
60
40
20
ppm