Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

A benzo[*c*]carbazolyl-based phosphine ligand for Pd-catalyzed tetra-*ortho*-substituted biaryl syntheses

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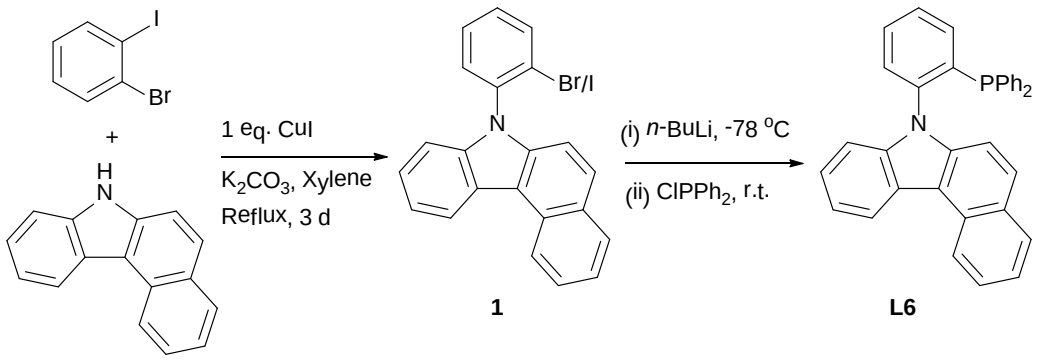
1. General considerations

Unless otherwise noted, all reagents were purchased from commercial suppliers and used without purification. All catalytic reactions were performed in re-sealable screw-capped Schlenk tube (approx. 20 mL volume) in the presence of Teflon-coated magnetic stirrer bar (4 mm × 10 mm). Dioxane and THF were freshly distilled from sodium benzophenone ketyl under nitrogen before use. Toluene was freshly distilled from sodium under nitrogen before use. *t*-BuOH and hexane were freshly distilled from calcium hydride under nitrogen before use. Xylene was distilled from sodium under nitrogen and stored under nitrogen. NEt_3, Cs_2CO_3, K_2CO_3, CsF, $K_3PO_4 \bullet H_2O$, KOH, Na_3PO_4 and K_3PO_4 were purchased from Aldrich. DABCO was purchased from Acros. $Pd(OAc)_2$, $Pd(dba)_2$, $Pd_2(dba)_3$ was purchased from Strem Chemical. $PdCl_2(ACN)_2$ and $[PdCl(cinnamyl)]_2$ were purchased from Aldrich. Ligands **L1**-**L5** were prepared according to reported literature procedures.[1] Commercially available aryl halides and aryl boronic acids were used as received. Thin layer chromatography was performed on Merck precoated silica gel 60 F_{254} plates. Silica gel (Merck, 70-230 and 230-400 mesh) was used for column chromatography. Melting points were recorded on an uncorrected Büchi Melting Point B-545 instrument. ^{1}H NMR spectra were recorded on a Bruker (400 MHz) spectrometer. Spectra were referenced internally to the residual proton resonance in $CDCl_3$ (δ 7.26 ppm), or with tetramethylsilane (TMS, δ 0.00 ppm) as the internal standard. Chemical shifts (δ) were reported as part per million (ppm) in δ scale downfield from TMS. ^{13}C NMR spectra were referenced to $CDCl_3$ (δ 77.00 ppm, the middle peak). ^{31}P NMR spectra were referenced to 85% H_3PO_4 externally. Coupling constants (*J*) were reported in Hertz (Hz). Mass spectra (EI-MS and ES-MS) were recorded on a HP 5989B Mass Spectrometer. High-resolution mass spectra (HRMS) were obtained on a Brüker APEX 47e FTICR mass spectrometer (ESIMS). GC-MS analysis was conducted on a HP 5973 GCD system using a HP5MS column (30 m × 0.25 mm). The products described in GC yield were accorded to the authentic samples/dodecane calibration standard from HP 6890 GC-FID system. All yields reported refer to isolated yield of compounds estimated to be greater than 95% purity as determined by capillary gas chromatography (GC) or ^{1}H NMR. Compounds described in the literature were characterized by comparison of their ^{1}H, and or ^{13}C NMR spectra to the previously reported data. The procedures in this section are representative, and thus the yields may differ from those reported in tables.

2. Synthesis of benzo[*c*]carbazolyl-based phosphine ligand



*General procedure for synthesis of 7-(2-bromophenyl)-7H-benzo[c]carbazole/7-(2-bromophenyl)-7H-benzo[c]carbazole (**1**):* 7*H*-benzo[c]carbazole (6.52 g, 30 mmol), CuI (5.72 g, 30 mmol), K_2CO_3 (8.28 g, 60 mmol) and Teflon-coated magnetic stir bar were charged to a two-necked round bottom flask (250 mL) equipped with a condenser and fitted with a septum. The system was carefully evacuated and backfilled with nitrogen (3 cycles). 2-Bromoiodobenzene (7.7 mL, 60 mmol) and xylene (100 mL) were added by syringe via septum. The septum was switched with a stopper and the reaction mixture was allowed to reflux in a preheated oil bath (185 ^{o}C) for 3 days. After completion of reaction, the copper powder was filtered by Celite® and the xylene was removed by distillation under high vacuum. The crude products were purified by flash column chromatography on silica gel (230-400 mesh) to afford the product as a white solid (5.85 g, 50%). The product was afforded as a bromo-/iodo- mixture. As determined by NMR spectroscopy, the bromo-/iodo- ratio was found to be 0.559:0.441. R_f = 0.63 (Hexane:EA = 9:1); Melting point 69.0 – 72.3 °C; HRMS: calcd. for $C_{22}H_{14}NBrH^+$: 372.0382, found 372.0385, calcd. for $C_{22}H_{14}NIH^+$: 420.0244, found 420.0248.

General procedure for synthesis of 7-(2-(diphenylphosphino)phenyl)-7*H*-benzo[c]carbazole (**L6**): The ligand precursors (**1**) obtained from the previous step (2.95 mmol) was dissolved in freshly distilled THF (15 mL) at room temperature under nitrogen atmosphere. The solution was cooled to -78 °C in a dry ice/acetone bath. Titrated *n*-BuLi (3.3 mmol) was added dropwise with a syringe and the reaction mixture was allowed to stir for 30 min at -78 °C. Chlorodiphenylphosphine (654 μL, 3.54 mmol) was then added dropwise to the reaction mixture with a syringe. The reaction was allowed to warm to room temperature and stirred overnight. The solvent was then removed under reduced pressure. Methanol (10 mL) was added to the

residue and the mixture was stirred at 1250 rpm for 10 min. The white solid product was filtered and washed with cold methanol successively. The white solid was collected and dried over vacuum to afford 7-(2-(diphenylphosphino)phenyl)-7*H*-benzo[c]carbazole (1.29 g, 92%). R_f = 0.66 (Hexane:EA = 2:1); Melting point 147.9 – 149.8 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.99 (d, *J* = 8.1 Hz, 1H), 7.07 – 7.14 (m, 5H), 7.17 – 7.27 (m, 7H), 7.37 (t, *J* = 7.5 Hz, 1H), 7.39 – 7.43 (m, 2H), 7.47 – 7.62 (m, 3H), 7.66 (d, *J* = 8.8 Hz, 1H), 7.74 (t, *J* = 7.6 Hz, 1H), 7.98 (d, *J* = 8.0 Hz, 1H), 8.59 (d, *J* = 7.9 Hz, 1H), 8.85 (d, *J* = 8.3 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 110.8 , 112.1 , 115.2 , 120.2 , 121.7 , 122.8 , 123.4 , 123.6 , 124.0 , 126.7 (d, *J* = 19.3 Hz), 128.2 (d, *J* = 2.2 Hz), 128.2 (d, *J* = 11.7 Hz), 128.6 , 129.1 (d, *J* = 9.6 Hz), 129.3 , 129.8 , 130.2 (d, *J* = 2.8 Hz), 130.4 , 133.6 (d, *J* = 5.4 Hz), 133.9 (d, *J* = 5.4 Hz), 134.9 , 136.0 (d, *J* = 12.0 Hz), 139.3 , 140.8, 140.8 (d, *J* = 55.9 Hz), 140.9 (d, *J* = 99.2 Hz); ^{31}P NMR (162 MHz, C_6D_6) δ -16.6; HRMS: calcd. for $C_{34}H_{24}NPH^+$: 478.1719, found 478.1719.

3. General procedures for reaction condition screenings

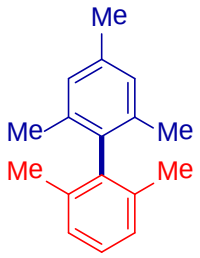
General procedure for screening: An array of Schlenk tubes were equipped with a Teflon-coated magnetic stir bar and fitted with a screw cap. Pd source (0.018 mmol) in 6.0 mL DCM (1.0 mol% Pd per 1.0 mL stock solution) was prepared with stirring until all of the solids were dissolved. The corresponding volume of palladium stock solution was then immediately added to the Schlenk tubes by syringe (In case of 0.5 mol% Pd, 0.5 mL of stock solution was transferred). The tubes were then carefully evacuated. After that, 2,6-dimethylphenyl boronic acid (0.45 mmol), ligand and base (0.9 mmol) were loaded to the Schlenk tubes. The tubes were carefully evacuated and backfilled with nitrogen for three cycles. 2-Bromomesitylene (46 μL, 0.3 mmol) followed by solvent (0.3 M, 1 mL) was added by syringe and the tubes were placed into a preheated oil bath (110 °C) and stirred for 18 h. After completion of reaction, the reaction tubes were allowed to reach room temperature. Ethyl acetate (~3 mL), dodecane (114 μL, internal standard), water (~2 mL) were added. The organic layer was subjected to GC analysis. The GC yield was previously calibrated by authentic sample/dodecane calibration curve.

4. General procedures for sterically hindered Suzuki–Miyaura cross-coupling

General procedure for sterically hindered Suzuki–Miyaura cross-coupling: An array of Schlenk tubes were equipped with a Teflon-coated magnetic stir bar and fitted with a screw cap. Aryl halide (if solid, 0.3 mmol), arylboronic acid (0.45 mmol), and K_3PO_4 (192 mg, 0.9 mmol) were loaded to the Schlenk tubes. The tubes were carefully evacuated and backfilled with nitrogen for three cycles. $Pd(OAc)_2$ (5.39 mg, 0.024 mmol) and ligand **L6** (45.8 mg, 0.096 mmol) in 8.0 mL Dioxane (1.0 mol% Pd per 1.0 mL stock solution) was prepared under nitrogen with stirring until all of the solids were dissolved (usually within 3 min). The corresponding volume of stock solution was then immediately added to the Schlenk tubes by syringe. Aryl bromide (if liquid, 0.3 mmol) was added by syringe and the tubes were placed into a preheated oil bath (110 °C) and stirred for 18 h. After completion of reaction, the reaction tube was allowed to reach room temperature. Ethyl acetate (~3 mL), water (~2 mL) were added. The organic layer was separated and the aqueous layer was washed with ethyl acetate. The filtrate was concentrated under reduced pressure. The crude products were purified by flash column chromatography on silica gel (230-400 mesh) to afford the desired product.

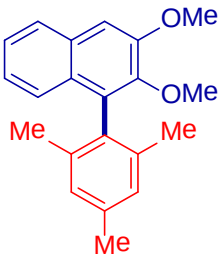
5. Characterization data of coupling products

2,2',4,6,6'-Pentamethylbiphenyl (Scheme 3, entry 3a)[2]



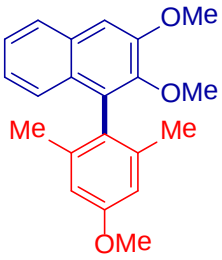
R_f = 0.70 (Hexane); ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.88 (s, 6H), 1.91 (s, 6H), 2.35 (s, 3H), 6.96 (s, 2H), 7.12 – 7.19 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.7, 19.8, 19.9, 21.1, 126.7, 127.3, 128.2, 128.2, 135.2, 135.5, 135.7, 136.1, 136.9, 140.0

1-Mesityl-2,3-dimethoxynaphthalene (Scheme 3, entry 3b)



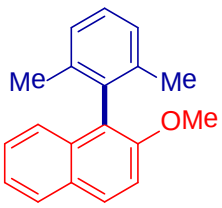
R_f = 0.59 (Hexane:EA = 9:1); Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.92 (s, 6H), 2.39 (s, 3H), 3.63 (s, 3H), 4.03 (s, 3H), 7.01 (s, 2H), 7.12 – 7.19 (m, 2H), 7.26 (s, 1H), 7.35 – 7.39 (m, 1H), 7.75 (d, *J* = 8.1 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.2, 21.2, 55.6, 60.0, 106.4, 124.0, 125.0, 125.2, 126.0, 126.6, 126.7, 128.1, 130.2, 131.4, 132.2, 136.8, 136.9, 152.3; HRMS: calcd. for $C_{21}H_{22}O_2Na$: 329.1512, found 329.1524.

2,3-Dimethoxy-1-(4-methoxy-2,6-dimethylphenyl)naphthalene (Scheme 3, entry 3c)



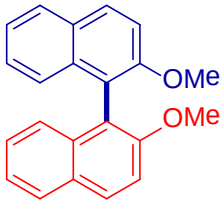
R_f = 0.33 (Hexane:EA = 9:1); White solid; Melting point 148.2 – 149.8 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.94 (s, 6H), 3.64 (s, 3H), 3.88 (s, 3H), 4.04 (s, 3H), 6.77 (s, 2H), 7.18 – 7.22 (m, 3H), 7.38 (s, 1H), 7.76 (d, *J* = 7.5 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.5, 55.0, 55.5, 56.0, 106.4, 112.6, 124.0, 124.9, 125.2, 126.6, 127.5, 128.2, 129.9, 131.3, 138.5, 146.2, 152.2, 158.7; HRMS: calcd. for $C_{21}H_{22}O_3Na$: 345.1461, found 341.1472.

1-(2,6-Dimethylphenyl)-2-methoxynaphthalene (Scheme 3, entry 3d)[2]



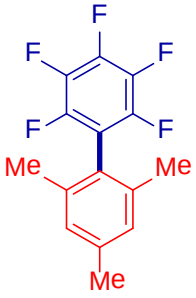
R_f = 0.66 (Hexane:EA = 9:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.95 (s, 6H), 3.89 (s, 3H), 7.21 – 7.26 (m, 3H), 7.28 – 7.41 (m, 3H), 7.44 (d, *J* = 9.1 Hz, 1H), 7.89 (d, *J* = 8.7 Hz, 1H), 7.95 (d, *J* = 9.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.1, 56.4, 113.5, 123.5, 124.4, 126.5, 127.2, 127.3, 128.0, 128.9, 129.1, 132.8, 135.7, 137.4, 137.5, 153.4

2,2'-Dimethoxy-1,1'-binaphthyl (Scheme 3, entry 3e)[3]



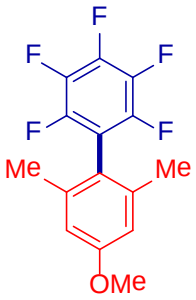
R_f = 0.34 (Hexane:EA = 9:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.77 (s, 6H), 7.12 (d, *J* = 8.4 Hz, 2H), 7.22 (t, *J* = 7.2 Hz, 2H), 7.33 (t, *J* = 7.8 Hz, 2H), 7.47 (d, *J* = 9.0 Hz, 2H), 7.88 (d, *J* = 8.2 Hz, 2H), 7.99 (d, *J* = 9.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 56.9, 114.3, 119.6, 123.5, 125.2, 126.3, 127.9, 129.2, 129.4, 134.0, 155.0

2,3,4,5,6-Pentafluoro-2',4',6'-trimethylbiphenyl (Scheme 3, entry 3f)[4]



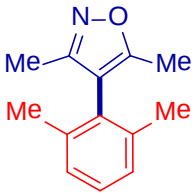
R_f = 0.69 (Hexane:EA = 9:1); Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.08 (s, 6H), 2.37 (s, 3H), 7.02 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.0, 21.1, 114.4, 122.6, 128.6, 137.2, 139.3, 141.8, 142.6, 145.1

2,3,4,5,6-Pentafluoro-4'-methoxy-2',6'-dimethylbiphenyl (Scheme 3, entry 3g)



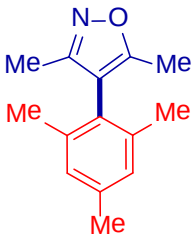
R_f = 0.75 (Hexane:EA = 9:1); Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.08 (s, 6H), 3.84 (s, 3H), 6.74 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.0, 21.1, 114.4, 122.6, 128.6, 137.2, 139.3, 141.8, 142.6, 145.1; HRMS: calcd. for $C_{15}H_{10}F_5OH^+$: 302.0725, found 302.0745.

4-(2,6-Dimethylphenyl)-3,5-dimethylisoxazole (Scheme 3, entry 3h)[2]



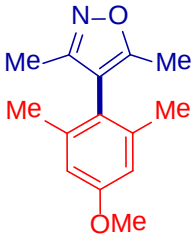
R_f = 0.30 (Hexane:EA = 2:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.02 (s, 3H), 2.04 (s, 6H), 2.17 (s, 3H), 7.11 (d, *J* = 7.5 Hz, 2H), 7.18 – 7.22 (m, 1H), 7.66 – 7.71 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 10.3, 11.1, 20.2, 114.6, 127.4, 128.2, 128.5, 138.1, 159.4, 165.0

4-Mesityl-3,5-dimethylisoxazole (Scheme 3, entry 3i)[5]



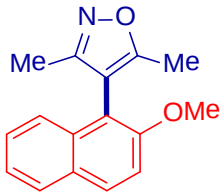
R_f = 0.30 (Hexane:EA = 2:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.00 (s, 6H), 2.02 (s, 3H), 2.17 (s, 3H), 2.32 (s, 3H), 6.95 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 10.3, 11.1, 20.1, 21.0, 114.5, 125.5, 128.2, 137.78, 137.85, 159.6, 165.0

4-(4-Methoxy-2,6-dimethylphenyl)-3,5-dimethylisoxazole (Scheme 3, entry 3j)



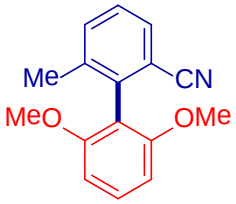
R_f = 0.47 (Hexane:EA = 9:1); White solid; Melting point 112.2 – 113.3 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.00 (s, 9H), 2.16 (s, 3H), 3.80 (s, 3H), 6.67 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 10.3, 11.1, 20.2, 20.5, 55.0, 112.8, 114.3, 120.7, 139.5, 159.2, 159.8, 165.2; HRMS: calcd. for $C_{14}H_{17}O_2NH^+$: 232.1332, found 232.1339.

4-(2-Methoxynaphthalen-1-yl)-3,5-dimethylisoxazole (Scheme 3, entry 3k)



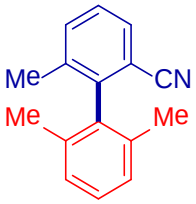
R_f = 0.30 (Hexane:EA = 2:1); Light pink solid; Melting point 141.3 – 143.4 ^oC; ^1H NMR (400 MHz, $CDCl_3$) δ 2.07 (s, 3H), 2.20 (s, 3H), 3.89 (s, 3H), 7.33 – 7.47 (m, 4H), 7.85 (d, *J* = 7.9 Hz, 1H), 7.93 (d, *J* = 9.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 10.5, 11.6, 56.1, 110.4, 112.0, 113.0, 123.6, 124.1, 126.9, 128.2, 128.9, 130.2, 133.4, 155.2, 160.7, 166.6; HRMS: calcd. for $C_{16}H_{15}NO_2Na$: 276.0995, found 276.1003.

2',6'-Dimethoxy-6-methylbiphenyl-2-carbonitrile (Scheme 3, entry 3l)



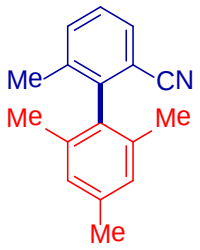
R_f = 0.31 (Hexane:EA = 9:1); White solid; Melting point 137.2 – 139.0 ^oC; ^1H NMR (400 MHz, $CDCl_3$) δ 2.12 (s, 3H), 3.75 (s, 6H), 6.67 (s, 1H), 6.70 (s, 1H), 7.32 (t, *J* = 7.7 Hz, 1H), 7.38 (t, *J* = 8.4 Hz, 1H), 7.48 (d, *J* = 7.6 Hz, 1H), 7.56 (d, *J* = 7.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.7, 55.8, 104.1, 114.5, 114.8, 118.8, 127.3, 129.9, 130.3, 133.7, 138.6, 139.2, 157.4; HRMS: calcd. for $C_{16}H_{15}NO_2Na$: 276.0995, found 276.1003.

2',6,6'-Trimethylbiphenyl-2-carbonitrile (Scheme 3, entry 3m)



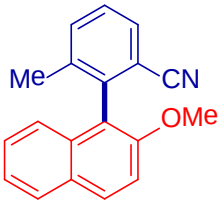
R_f = 0.62 (Hexane:EA = 9:1); Colorless oil; ^1H NMR (400 MHz, $CDCl_3$) δ 1.97 (s, 6H), 2.02 (s, 3H), 7.16 (s, 1H), 7.17 (s, 1H), 7.22 – 7.26 (m, 1H), 7.37 (t, *J* = 7.6 Hz, 1H), 7.54 (d, *J* = 7.6 Hz, 1H), 7.62 (d, *J* = 7.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.4, 19.8, 113.0, 117.8, 127.6, 127.7, 128.3, 130.6, 134.4, 135.3, 136.7, 137.6, 144.4; HRMS: calcd. for $C_{16}H_{15}NNa$: 244.1097, found 244.1104.

2',4',6,6'-Tetramethylbiphenyl-2-carbonitrile (Scheme 3, entry 3n)



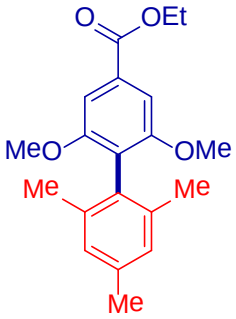
R_f = 0.63 (Hexane:EA = 9:1); White solid; Melting point 58.3 – 63.5 ^oC; ^1H NMR (400 MHz, $CDCl_3$) δ 1.91 (s, 6H), 2.02 (s, 3H), 2.33 (s, 3H), 6.97 (s, 2H), 7.34 (t, *J* = 7.7 Hz, 1H), 7.51 (d, *J* = 7.6 Hz, 1H), 7.59 (d, *J* = 7.7 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.5, 19.8, 21.2, 113.3, 118.0, 127.5, 128.6, 130.6, 133.9, 134.3, 135.1, 137.8, 137.9, 144.7; HRMS: calcd. for $C_{17}H_{17}NNa$: 258.1253, found 258.1260.

2-(2-Methoxynaphthalen-1-yl)-3-methylbenzonitrile (Scheme 3, entry 3o)



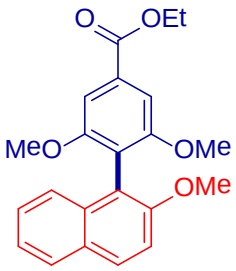
R_f = 0.32 (Hexane:EA = 9:1); Light yellow solid; Melting point 147.0 – 150.8 ^oC; ^1H NMR (400 MHz, $CDCl_3$) δ 2.02 (s, 3H), 3.91 (s, 3H), 7.09 – 7.11 (m, 1H), 7.35 – 7.40 (m, 2H), 7.41 – 7.45 (m, 2H), 7.59 (d, *J* = 7.7 Hz, 1H), 7.68 (d, *J* = 7.7 Hz, 1H), 7.87 – 7.89 (m, 1H), 7.99 (d, *J* = 9.1 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.8, 56.4, 113.3, 114.7, 118.4, 119.9, 123.6, 123.7, 127.1, 127.8, 128.3, 128.9, 130.4, 130.6, 132.5, 134.1, 139.4, 140.3, 153.9; HRMS: calcd. for $C_{19}H_{15}NONa$: 296.1046, found 296.1041.

Ethyl 2,6-dimethoxy-2',4',6'-trimethylbiphenyl-4-carboxylate (Scheme 3, entry 3p)



R_f = 0.44 (Hexane:EA = 9:1); White solid; Melting point 114.3 – 119.6 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.49 (t, *J* = 7.0 Hz, 3H), 2.00 (s, 6H), 2.38 (s, 3H), 3.82 (s, 6H), 4.50 (q, *J* = 7.1 Hz, 2H), 6.99 (s, 2H), 7.42 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.3, 19.7, 21.1, 55.8, 61.0, 105.1, 122.6, 127.8, 130.1, 130.8, 136.4, 136.7, 157.3, 166.5; HRMS: calcd. for $C_{20}H_{24}O_4Na$: 351.1567, found 351.1579.

Ethyl 3,5-dimethoxy-4-(2-methoxynaphthalen-1-yl)benzoate (Scheme 3, entry 3q)



R_f = 0.19 (Hexane:EA = 9:1); White solid; Melting point 181.8 – 183.4 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.48 (t, *J* = 7.1 Hz, 3H), 3.74 (s, 6H), 3.86 (s, 3H), 4.50 (q, *J* = 7.1 Hz, 2H), 7.28 (s, 1H), 7.32 – 7.35 (m, 2H), 7.41 (d, *J* = 9.0 Hz, 1H) 7.46 (s, 2H), 7.83 – 7.85 (m, 1H), 7.92 (d, *J* = 8.9 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 14.3, 56.0, 56.8, 61.0, 105.4, 114.1, 117.4, 118.8, 123.3, 124.6, 126.0, 127.9, 129.0, 129.3, 131.2, 133.0, 154.4, 158.4, 166.5; HRMS: calcd. for $C_{22}H_{22}O_5Na$: 389.1359, found 389.1365.

6. X-ray crystallographic data of ligand L6

Table S1. Crystal data and structure refinement for **L6**.

Empirical formula	C_{34} H_{24} N P	
Formula weight	477.51	
Temperature	298(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 9.9764(5) Å	α= 115.716(2)°.
	b = 11.8382(6) Å	β= 100.125(2)°.
	c = 12.5698(6) Å	γ = 93.876(2)°.
Volume	1299.22(11) $Å^3$	
Z	2	
Density (calculated)	1.221 Mg/m^3	
Absorption coefficient	0.129 mm^{-1}	
F(000)	500	
Crystal size	0.32 x 0.26 x 0.24 mm^3	
Theta range for data collection	2.10 to 30.60°.	
Index ranges	-14<=h<=14, -16<=k<=16, -17<=l<=17	
Reflections collected	115406	
Independent reflections	7967 [R(int) = 0.0308]	
Completeness to theta = 30.60°	99.8 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7461 and 0.7174	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	7967 / 12 / 326	
Goodness-of-fit on F^2	1.002	
Final R indices [I>2sigma(I)]	R1 = 0.0595, wR2 = 0.1549	
R indices (all data)	R1 = 0.0766, wR2 = 0.1680	
Extinction coefficient	0.032(3)	
Largest diff. peak and hole	0.423 and -0.328 e.$Å^{-3}$	

Table S2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **L6**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
P(1)	2821(1)	7025(1)	4449(1)	57(1)
N(1)	2000(1)	6258(1)	1757(1)	48(1)
C(1)	2537(1)	5397(1)	3206(1)	45(1)
C(2)	2142(1)	5201(1)	2008(1)	45(1)
C(3)	1897(2)	3990(1)	1044(1)	56(1)
C(4)	2048(2)	2954(1)	1258(1)	58(1)
C(5)	2427(1)	3122(1)	2430(1)	52(1)
C(6)	2668(1)	4327(1)	3388(1)	51(1)
C(7)	2950(1)	6808(1)	1371(1)	47(1)
C(8)	4204(1)	6447(2)	1115(1)	60(1)
C(9)	4987(2)	7173(2)	773(2)	71(1)
C(10)	4554(2)	8217(2)	684(2)	71(1)
C(11)	3311(1)	8577(1)	926(1)	58(1)
C(12)	2475(1)	7860(1)	1276(1)	44(1)
C(13)	1167(1)	7942(1)	1623(1)	41(1)
C(14)	192(1)	8790(1)	1737(1)	43(1)
C(15)	319(2)	9817(1)	1449(1)	56(1)
C(16)	-662(2)	10590(1)	1576(2)	70(1)
C(17)	-1808(2)	10393(2)	1995(2)	74(1)
C(18)	-1963(2)	9416(2)	2288(1)	65(1)
C(19)	-985(1)	8582(1)	2158(1)	49(1)
C(20)	-1180(1)	7539(1)	2420(1)	56(1)
C(21)	-260(1)	6724(1)	2301(1)	53(1)
C(22)	921(1)	6945(1)	1910(1)	43(1)
C(23)	4683(2)	7495(1)	4718(1)	56(1)
C(24)	5601(2)	6686(1)	4268(1)	60(1)
C(25)	7001(2)	7121(2)	4546(2)	74(1)
C(26)	7508(2)	8384(2)	5280(2)	93(1)

C(27)	6625(3)	9199(2)	5731(2)	102(1)
C(28)	5223(2)	8777(2)	5461(2)	83(1)
C(29)	2704(1)	6683(1)	5725(1)	53(1)
C(30)	3835(2)	6732(1)	6562(1)	59(1)
C(31)	3681(2)	6516(2)	7536(1)	71(1)
C(32)	2400(2)	6239(2)	7691(2)	78(1)
C(33)	1261(2)	6174(2)	6868(2)	78(1)
C(34)	1402(2)	6400(2)	5896(2)	70(1)

Table S3. Bond lengths [Å] and angles [°] for **L6**.

P(1)-C(23)	1.8306(16)
P(1)-C(29)	1.8364(16)
P(1)-C(1)	1.8423(11)
N(1)-C(7)	1.3826(17)
N(1)-C(22)	1.3833(16)
N(1)-C(2)	1.4264(16)
C(1)-C(6)	1.3920(18)
C(1)-C(2)	1.3978(17)
C(2)-C(3)	1.3872(16)
C(3)-C(4)	1.377(2)
C(3)-H(3A)	0.9300
C(4)-C(5)	1.376(2)
C(4)-H(4A)	0.9300
C(5)-C(6)	1.3802(16)
C(5)-H(5A)	0.9300
C(6)-H(6A)	0.9300
C(7)-C(8)	1.3941(19)
C(7)-C(12)	1.4064(18)
C(8)-C(9)	1.371(2)
C(8)-H(8A)	0.9300
C(9)-C(10)	1.380(3)
C(9)-H(9A)	0.9300
C(10)-C(11)	1.379(2)

C(10)-H(10A)	0.9300
C(11)-C(12)	1.4030(19)
C(11)-H(11A)	0.9300
C(12)-C(13)	1.4431(16)
C(13)-C(22)	1.3944(17)
C(13)-C(14)	1.4247(17)
C(14)-C(15)	1.4145(19)
C(14)-C(19)	1.4182(18)
C(15)-C(16)	1.368(2)
C(15)-H(15A)	0.9300
C(16)-C(17)	1.386(3)
C(16)-H(16A)	0.9300
C(17)-C(18)	1.364(3)
C(17)-H(17A)	0.9300
C(18)-C(19)	1.414(2)
C(18)-H(18A)	0.9300
C(19)-C(20)	1.419(2)
C(20)-C(21)	1.356(2)
C(20)-H(20A)	0.9300
C(21)-C(22)	1.4044(18)
C(21)-H(21A)	0.9300
C(23)-C(24)	1.381(2)
C(23)-C(28)	1.3958(19)
C(24)-C(25)	1.383(2)
C(24)-H(24A)	0.9300
C(25)-C(26)	1.371(3)
C(25)-H(25A)	0.9300
C(26)-C(27)	1.358(3)
C(26)-H(26A)	0.9300
C(27)-C(28)	1.383(3)
C(27)-H(27A)	0.9300
C(28)-H(28A)	0.9300
C(29)-C(30)	1.3798(19)
C(29)-C(34)	1.395(2)
C(30)-C(31)	1.386(2)

C(30)-H(30A)	0.9300
C(31)-C(32)	1.365(3)
C(31)-H(31A)	0.9300
C(32)-C(33)	1.369(3)
C(32)-H(32A)	0.9300
C(33)-C(34)	1.386(3)
C(33)-H(33A)	0.9300
C(34)-H(34A)	0.9300
C(23)-P(1)-C(29)	101.83(6)
C(23)-P(1)-C(1)	101.76(6)
C(29)-P(1)-C(1)	100.22(6)
C(7)-N(1)-C(22)	108.14(10)
C(7)-N(1)-C(2)	125.79(11)
C(22)-N(1)-C(2)	126.01(11)
C(6)-C(1)-C(2)	117.18(10)
C(6)-C(1)-P(1)	123.61(9)
C(2)-C(1)-P(1)	119.19(9)
C(3)-C(2)-C(1)	121.16(12)
C(3)-C(2)-N(1)	118.77(11)
C(1)-C(2)-N(1)	120.06(10)
C(4)-C(3)-C(2)	120.06(12)
C(4)-C(3)-H(3A)	120.0
C(2)-C(3)-H(3A)	120.0
C(5)-C(4)-C(3)	119.86(11)
C(5)-C(4)-H(4A)	120.1
C(3)-C(4)-H(4A)	120.1
C(4)-C(5)-C(6)	120.04(13)
C(4)-C(5)-H(5A)	120.0
C(6)-C(5)-H(5A)	120.0
C(5)-C(6)-C(1)	121.69(12)
C(5)-C(6)-H(6A)	119.2
C(1)-C(6)-H(6A)	119.2
N(1)-C(7)-C(8)	128.38(13)
N(1)-C(7)-C(12)	109.07(11)
C(8)-C(7)-C(12)	122.55(13)

C(9)-C(8)-C(7)	117.33(15)
C(9)-C(8)-H(8A)	121.3
C(7)-C(8)-H(8A)	121.3
C(8)-C(9)-C(10)	121.42(14)
C(8)-C(9)-H(9A)	119.3
C(10)-C(9)-H(9A)	119.3
C(11)-C(10)-C(9)	121.75(15)
C(11)-C(10)-H(10A)	119.1
C(9)-C(10)-H(10A)	119.1
C(10)-C(11)-C(12)	118.74(15)
C(10)-C(11)-H(11A)	120.6
C(12)-C(11)-H(11A)	120.6
C(11)-C(12)-C(7)	118.21(12)
C(11)-C(12)-C(13)	135.16(12)
C(7)-C(12)-C(13)	106.61(11)
C(22)-C(13)-C(14)	119.28(11)
C(22)-C(13)-C(12)	106.43(10)
C(14)-C(13)-C(12)	134.28(11)
C(15)-C(14)-C(19)	118.11(12)
C(15)-C(14)-C(13)	124.23(12)
C(19)-C(14)-C(13)	117.66(11)
C(16)-C(15)-C(14)	121.04(15)
C(16)-C(15)-H(15A)	119.5
C(14)-C(15)-H(15A)	119.5
C(15)-C(16)-C(17)	120.88(16)
C(15)-C(16)-H(16A)	119.6
C(17)-C(16)-H(16A)	119.6
C(18)-C(17)-C(16)	119.76(15)
C(18)-C(17)-H(17A)	120.1
C(16)-C(17)-H(17A)	120.1
C(17)-C(18)-C(19)	121.41(15)
C(17)-C(18)-H(18A)	119.3
C(19)-C(18)-H(18A)	119.3
C(18)-C(19)-C(14)	118.78(13)
C(18)-C(19)-C(20)	121.04(13)

C(14)-C(19)-C(20)	120.17(12)
C(21)-C(20)-C(19)	122.23(12)
C(21)-C(20)-H(20A)	118.9
C(19)-C(20)-H(20A)	118.9
C(20)-C(21)-C(22)	117.66(12)
C(20)-C(21)-H(21A)	121.2
C(22)-C(21)-H(21A)	121.2
N(1)-C(22)-C(13)	109.76(11)
N(1)-C(22)-C(21)	127.27(12)
C(13)-C(22)-C(21)	122.98(11)
C(24)-C(23)-C(28)	117.31(16)
C(24)-C(23)-P(1)	125.55(10)
C(28)-C(23)-P(1)	117.12(14)
C(23)-C(24)-C(25)	121.57(14)
C(23)-C(24)-H(24A)	119.2
C(25)-C(24)-H(24A)	119.2
C(26)-C(25)-C(24)	120.10(19)
C(26)-C(25)-H(25A)	120.0
C(24)-C(25)-H(25A)	120.0
C(27)-C(26)-C(25)	119.39(19)
C(27)-C(26)-H(26A)	120.3
C(25)-C(26)-H(26A)	120.3
C(26)-C(27)-C(28)	121.16(17)
C(26)-C(27)-H(27A)	119.4
C(28)-C(27)-H(27A)	119.4
C(27)-C(28)-C(23)	120.47(19)
C(27)-C(28)-H(28A)	119.8
C(23)-C(28)-H(28A)	119.8
C(30)-C(29)-C(34)	117.31(15)
C(30)-C(29)-P(1)	123.84(12)
C(34)-C(29)-P(1)	118.81(12)
C(29)-C(30)-C(31)	121.23(15)
C(29)-C(30)-H(30A)	119.4
C(31)-C(30)-H(30A)	119.4
C(32)-C(31)-C(30)	120.78(16)

C(32)-C(31)-H(31A)	119.6
C(30)-C(31)-H(31A)	119.6
C(31)-C(32)-C(33)	119.15(18)
C(31)-C(32)-H(32A)	120.4
C(33)-C(32)-H(32A)	120.4
C(32)-C(33)-C(34)	120.58(16)
C(32)-C(33)-H(33A)	119.7
C(34)-C(33)-H(33A)	119.7
C(33)-C(34)-C(29)	120.93(15)
C(33)-C(34)-H(34A)	119.5
C(29)-C(34)-H(34A)	119.5

Symmetry transformations used to generate equivalent atoms:

Table S4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for fwc3. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
P(1)	68(1)	42(1)	45(1)	12(1)	-6(1)	17(1)
N(1)	45(1)	54(1)	51(1)	29(1)	10(1)	11(1)
C(1)	44(1)	41(1)	40(1)	14(1)	-3(1)	8(1)
C(2)	41(1)	46(1)	43(1)	20(1)	2(1)	7(1)
C(3)	61(1)	55(1)	38(1)	15(1)	1(1)	5(1)
C(4)	63(1)	42(1)	49(1)	8(1)	0(1)	1(1)
C(5)	53(1)	41(1)	56(1)	19(1)	1(1)	5(1)
C(6)	57(1)	47(1)	43(1)	19(1)	-2(1)	10(1)
C(7)	42(1)	51(1)	39(1)	16(1)	6(1)	4(1)
C(8)	48(1)	67(1)	58(1)	21(1)	14(1)	15(1)
C(9)	47(1)	89(1)	70(1)	25(1)	22(1)	9(1)
C(10)	55(1)	83(1)	75(1)	33(1)	25(1)	-4(1)
C(11)	54(1)	59(1)	60(1)	26(1)	15(1)	-2(1)
C(12)	41(1)	46(1)	37(1)	14(1)	6(1)	0(1)
C(13)	40(1)	42(1)	34(1)	15(1)	4(1)	1(1)
C(14)	44(1)	41(1)	36(1)	12(1)	3(1)	2(1)

C(15)	59(1)	43(1)	58(1)	19(1)	8(1)	3(1)
C(16)	75(1)	45(1)	81(1)	24(1)	9(1)	11(1)
C(17)	71(1)	50(1)	84(1)	16(1)	12(1)	20(1)
C(18)	56(1)	63(1)	67(1)	19(1)	18(1)	16(1)
C(19)	46(1)	53(1)	43(1)	16(1)	9(1)	9(1)
C(20)	45(1)	76(1)	56(1)	36(1)	17(1)	10(1)
C(21)	49(1)	66(1)	57(1)	40(1)	12(1)	7(1)
C(22)	41(1)	50(1)	40(1)	23(1)	5(1)	6(1)
C(23)	78(1)	42(1)	38(1)	17(1)	-1(1)	-2(1)
C(24)	70(1)	53(1)	55(1)	27(1)	7(1)	-1(1)
C(25)	74(1)	87(1)	66(1)	41(1)	14(1)	-5(1)
C(26)	91(1)	106(1)	62(1)	34(1)	3(1)	-35(1)
C(27)	124(2)	70(1)	69(1)	9(1)	2(1)	-41(1)
C(28)	112(1)	48(1)	63(1)	8(1)	10(1)	-9(1)
C(29)	53(1)	46(1)	44(1)	7(1)	7(1)	13(1)
C(30)	52(1)	73(1)	47(1)	23(1)	12(1)	14(1)
C(31)	75(1)	86(1)	50(1)	29(1)	13(1)	10(1)
C(32)	92(1)	71(1)	62(1)	19(1)	29(1)	-1(1)
C(33)	68(1)	63(1)	86(1)	14(1)	31(1)	1(1)
C(34)	52(1)	60(1)	72(1)	11(1)	5(1)	4(1)

Table S5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for **L6**.

	x	y	z	U(eq)
H(3A)	1631	3878	253	67
H(4A)	1894	2143	611	70
H(5A)	2520	2422	2576	63
H(6A)	2925	4426	4175	61
H(8A)	4496	5743	1173	72
H(9A)	5829	6956	598	85
H(10A)	5114	8691	455	86

H(11A)	3033	9282	858	70
H(15A)	1082	9969	1170	67
H(16A)	-559	11258	1378	83
H(17A)	-2469	10924	2077	89
H(18A)	-2727	9296	2579	78
H(20A)	-1966	7410	2682	67
H(21A)	-405	6043	2473	63
H(24A)	5270	5827	3765	72
H(25A)	7599	6557	4234	89
H(26A)	8449	8680	5468	111
H(27A)	6969	10056	6231	122
H(28A)	4635	9352	5775	100
H(30A)	4717	6912	6470	70
H(31A)	4459	6562	8092	86
H(32A)	2303	6096	8346	94
H(33A)	385	5977	6961	94
H(34A)	618	6362	5350	84

Table S6. Torsion angles [°] for **L6**.

C(23)-P(1)-C(1)-C(6)	87.11(12)
C(29)-P(1)-C(1)-C(6)	-17.39(13)
C(23)-P(1)-C(1)-C(2)	-94.20(11)
C(29)-P(1)-C(1)-C(2)	161.31(10)
C(6)-C(1)-C(2)-C(3)	-0.27(19)
P(1)-C(1)-C(2)-C(3)	-179.05(10)
C(6)-C(1)-C(2)-N(1)	-179.45(11)
P(1)-C(1)-C(2)-N(1)	1.77(16)
C(7)-N(1)-C(2)-C(3)	-77.85(15)
C(22)-N(1)-C(2)-C(3)	105.29(14)
C(7)-N(1)-C(2)-C(1)	101.34(14)
C(22)-N(1)-C(2)-C(1)	-75.52(15)
C(1)-C(2)-C(3)-C(4)	-0.2(2)
N(1)-C(2)-C(3)-C(4)	178.94(12)

C(2)-C(3)-C(4)-C(5)	0.7(2)
C(3)-C(4)-C(5)-C(6)	-0.6(2)
C(4)-C(5)-C(6)-C(1)	0.1(2)
C(2)-C(1)-C(6)-C(5)	0.4(2)
P(1)-C(1)-C(6)-C(5)	179.08(11)
C(22)-N(1)-C(7)-C(8)	179.21(12)
C(2)-N(1)-C(7)-C(8)	1.88(19)
C(22)-N(1)-C(7)-C(12)	0.04(12)
C(2)-N(1)-C(7)-C(12)	-177.29(10)
N(1)-C(7)-C(8)-C(9)	-178.30(12)
C(12)-C(7)-C(8)-C(9)	0.77(19)
C(7)-C(8)-C(9)-C(10)	-0.1(2)
C(8)-C(9)-C(10)-C(11)	-0.4(2)
C(9)-C(10)-C(11)-C(12)	0.3(2)
C(10)-C(11)-C(12)-C(7)	0.36(18)
C(10)-C(11)-C(12)-C(13)	178.19(13)
N(1)-C(7)-C(12)-C(11)	178.33(10)
C(8)-C(7)-C(12)-C(11)	-0.90(17)
N(1)-C(7)-C(12)-C(13)	-0.08(12)
C(8)-C(7)-C(12)-C(13)	-179.30(11)
C(11)-C(12)-C(13)-C(22)	-177.92(13)
C(7)-C(12)-C(13)-C(22)	0.09(11)
C(11)-C(12)-C(13)-C(14)	0.7(2)
C(7)-C(12)-C(13)-C(14)	178.72(11)
C(22)-C(13)-C(14)-C(15)	-178.66(10)
C(12)-C(13)-C(14)-C(15)	2.85(19)
C(22)-C(13)-C(14)-C(19)	0.83(15)
C(12)-C(13)-C(14)-C(19)	-177.66(11)
C(19)-C(14)-C(15)-C(16)	0.18(18)
C(13)-C(14)-C(15)-C(16)	179.67(12)
C(14)-C(15)-C(16)-C(17)	0.3(2)
C(15)-C(16)-C(17)-C(18)	0.1(2)
C(16)-C(17)-C(18)-C(19)	-0.9(2)
C(17)-C(18)-C(19)-C(14)	1.3(2)
C(17)-C(18)-C(19)-C(20)	-177.55(14)

C(15)-C(14)-C(19)-C(18)	-0.92(16)
C(13)-C(14)-C(19)-C(18)	179.56(11)
C(15)-C(14)-C(19)-C(20)	177.92(11)
C(13)-C(14)-C(19)-C(20)	-1.60(16)
C(18)-C(19)-C(20)-C(21)	179.87(12)
C(14)-C(19)-C(20)-C(21)	1.05(18)
C(19)-C(20)-C(21)-C(22)	0.31(19)
C(7)-N(1)-C(22)-C(13)	0.02(12)
C(2)-N(1)-C(22)-C(13)	177.34(10)
C(7)-N(1)-C(22)-C(21)	-179.42(11)
C(2)-N(1)-C(22)-C(21)	-2.10(18)
C(14)-C(13)-C(22)-N(1)	-178.94(9)
C(12)-C(13)-C(22)-N(1)	-0.06(12)
C(14)-C(13)-C(22)-C(21)	0.53(16)
C(12)-C(13)-C(22)-C(21)	179.41(10)
C(20)-C(21)-C(22)-N(1)	178.26(11)
C(20)-C(21)-C(22)-C(13)	-1.11(17)
C(29)-P(1)-C(23)-C(24)	90.27(13)
C(1)-P(1)-C(23)-C(24)	-12.95(14)
C(29)-P(1)-C(23)-C(28)	-88.48(13)
C(1)-P(1)-C(23)-C(28)	168.29(13)
C(28)-C(23)-C(24)-C(25)	0.5(2)
P(1)-C(23)-C(24)-C(25)	-178.25(12)
C(23)-C(24)-C(25)-C(26)	-0.3(3)
C(24)-C(25)-C(26)-C(27)	0.1(3)
C(25)-C(26)-C(27)-C(28)	-0.1(3)
C(26)-C(27)-C(28)-C(23)	0.4(3)
C(24)-C(23)-C(28)-C(27)	-0.6(3)
P(1)-C(23)-C(28)-C(27)	178.30(16)
C(23)-P(1)-C(29)-C(30)	-3.49(13)
C(1)-P(1)-C(29)-C(30)	100.95(12)
C(23)-P(1)-C(29)-C(34)	174.07(11)
C(1)-P(1)-C(29)-C(34)	-81.49(11)
C(34)-C(29)-C(30)-C(31)	-0.4(2)
P(1)-C(29)-C(30)-C(31)	177.19(12)

C(29)-C(30)-C(31)-C(32)	0.5(2)
C(30)-C(31)-C(32)-C(33)	0.0(3)
C(31)-C(32)-C(33)-C(34)	-0.6(3)
C(32)-C(33)-C(34)-C(29)	0.8(2)
C(30)-C(29)-C(34)-C(33)	-0.2(2)
P(1)-C(29)-C(34)-C(33)	-177.94(12)

Symmetry transformations used to generate equivalent atoms:

7. References

1 S. C. To and F. Y. Kwong, *Chem. Commun.*, 2011, **47**, 5079-5081.

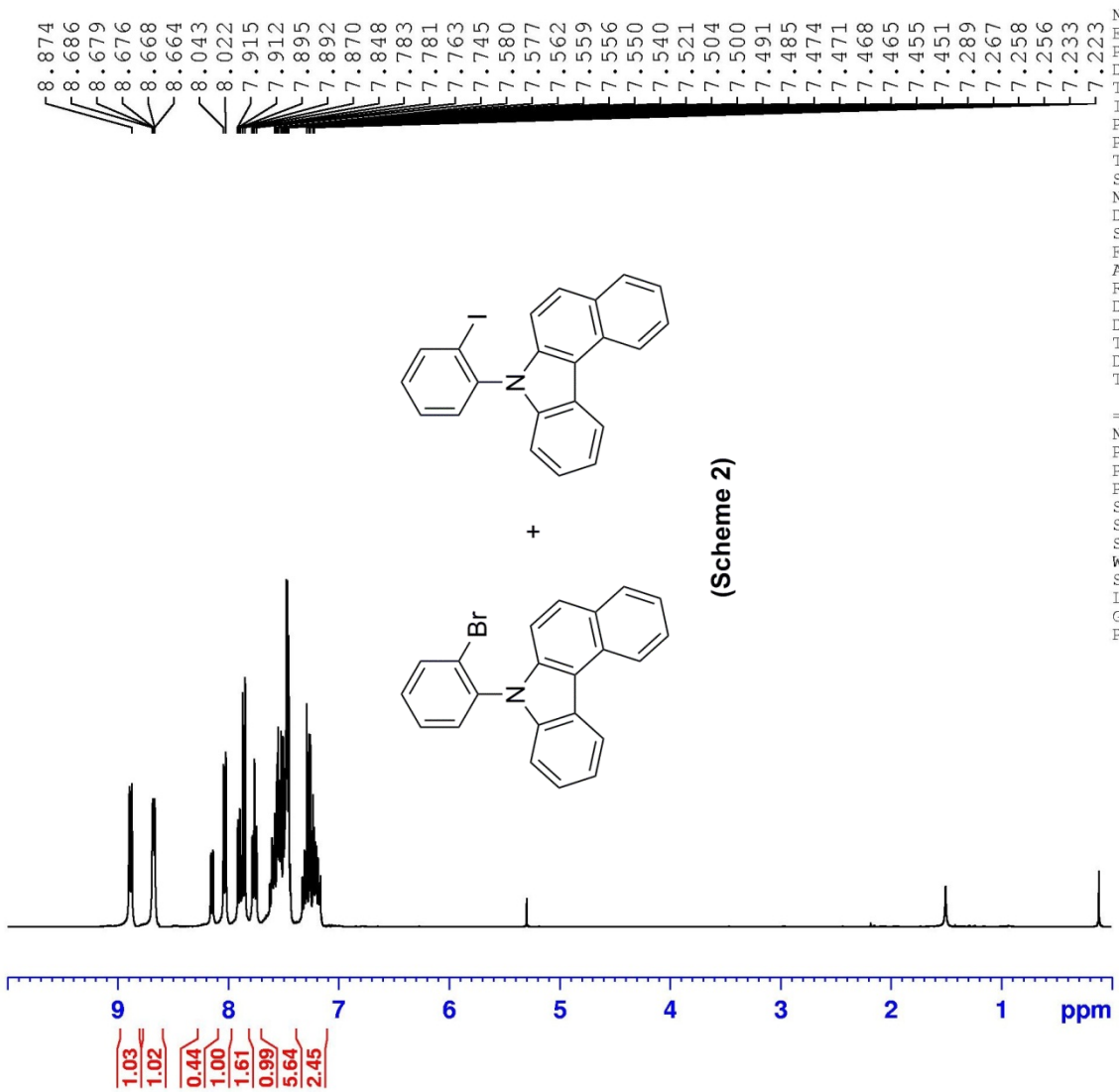
2 M. G. Organ, S. Calimsiz, M. Sayah, K. H. Hoi and A. J. Lough, *Angew. Chem. Int. Ed*., 2009, **121**, 2419-2423.

3 G. Gottarelli and G. P. Spada, *J. Org. Chem*. 1991, **56**, 2096-2098.

4 V. N. Kovtonyuk, L. S. Kobrina and G. Haufe, *Russ. Chem. Bull.* **57**, 1686-1688

5 S. Calimsiz, M. Sayah, D. Mallik and M. G. Organ, *Angew. Chem. Int. Ed*., 2010, **49**, 2014-2017.

8. ^{1}H, ^{13}C, ^{31}P NMR, HRMS spectra



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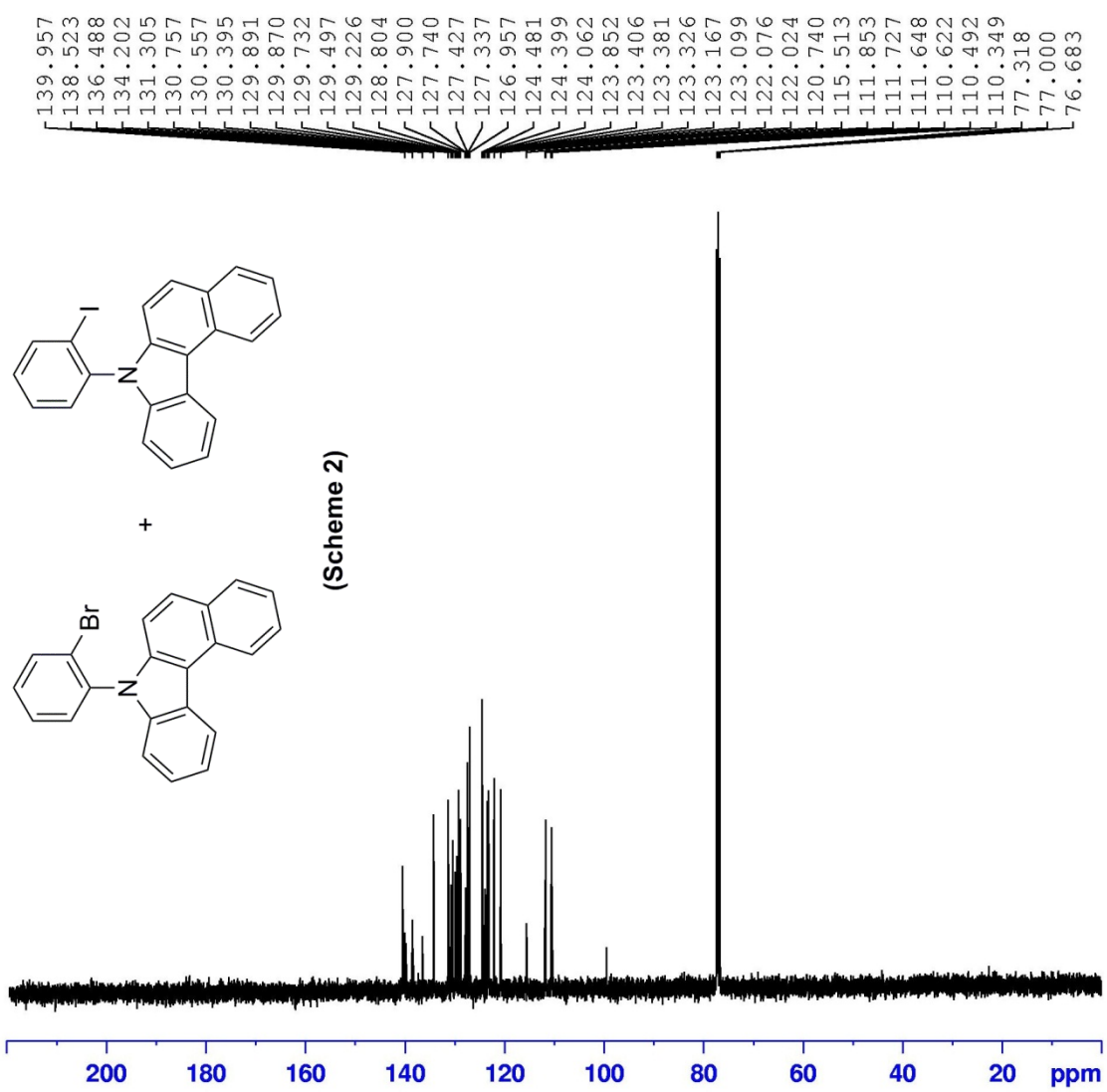
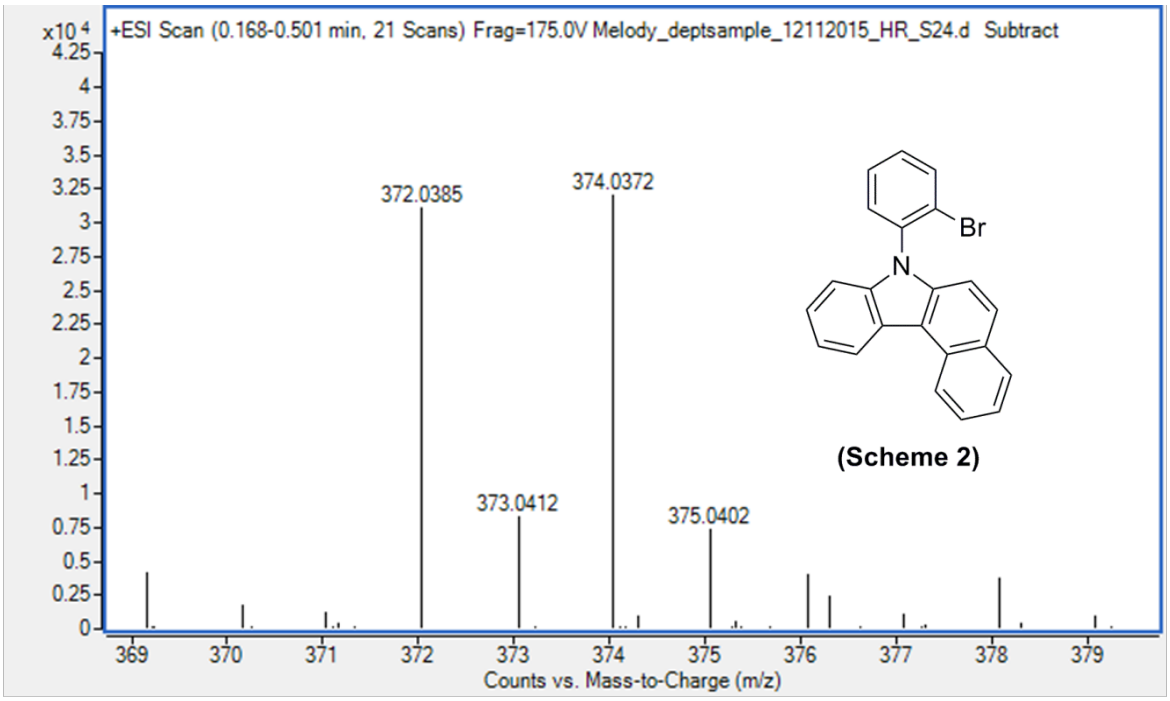
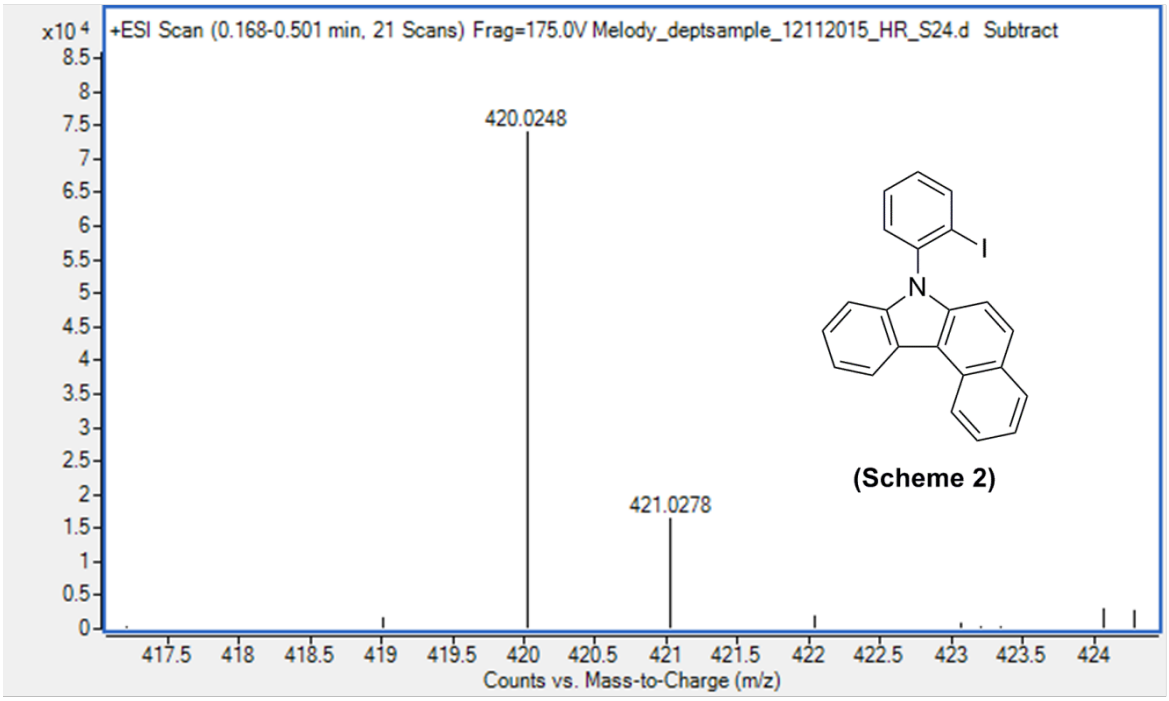
EXPNO	1
PROCNO	1
Date_	20140120
Time	20.17
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	16384
SOLVENT	CDC13
NS	16
DS	2
SWH	8012.820 Hz
FIDRES	0.489064 Hz
AQ	1.0224116 sec
RG	128
DW	62.400 usec
DE	6.50 usec
TE	297.8 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.70 usec
PL1	0.00 dB
PL1W	11.88122272 W
SFO1	400.1336012 MHz
SI	32768
SF	400.1300102 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

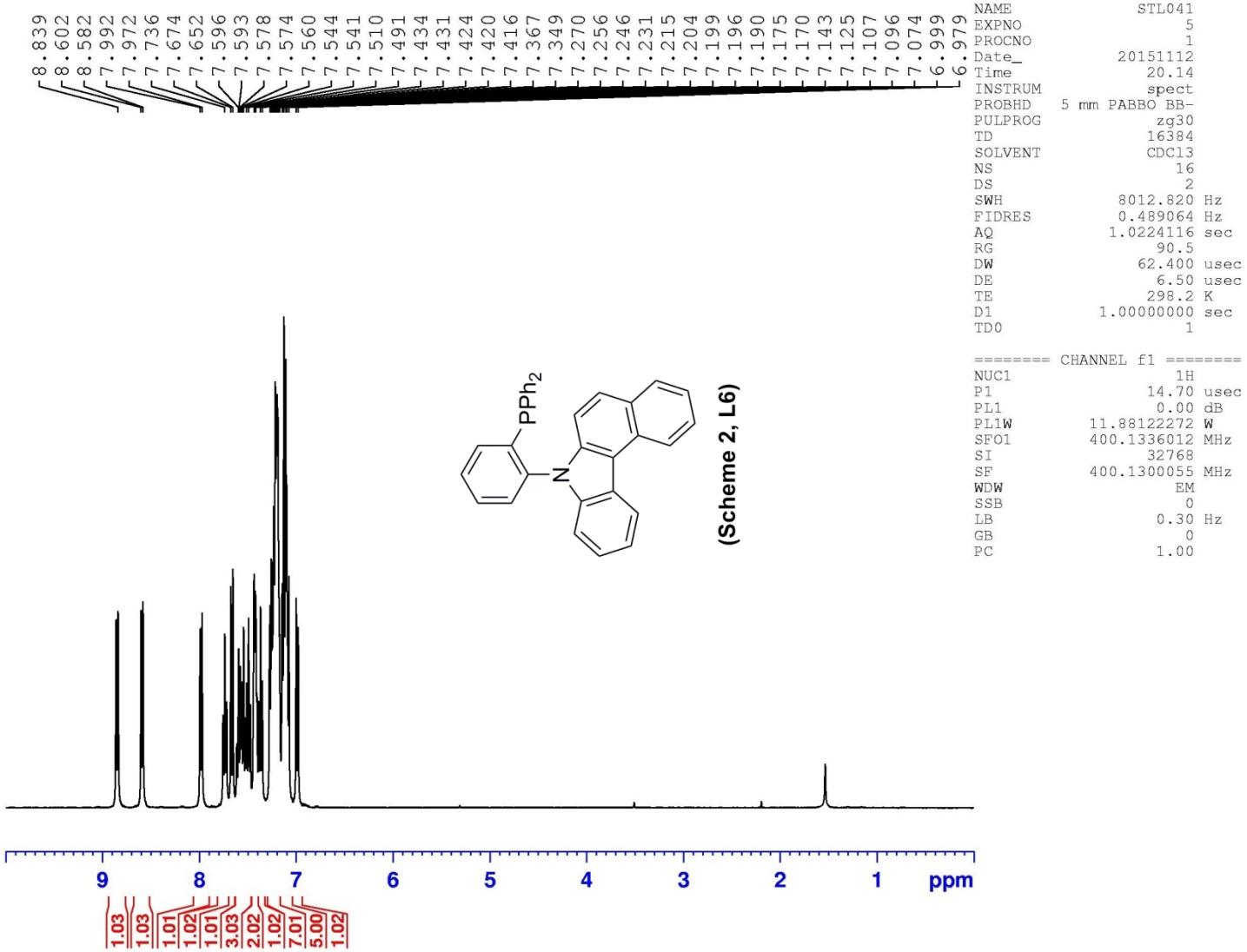


NAME	STL041pre
EXPNO	2
PROCNO	1
Date_	20151112
Time	20.07
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	82
DS	2
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	228
DW	20.800 usec
DE	6.50 usec
TE	298.6 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.50 usec
PL1	-2.00 dB
PL1W	58.52175522 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	15.00 dB
PL13	15.00 dB
PL2W	11.88122272 W
PL12W	0.37571725 W
PL13W	0.37571725 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127766 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

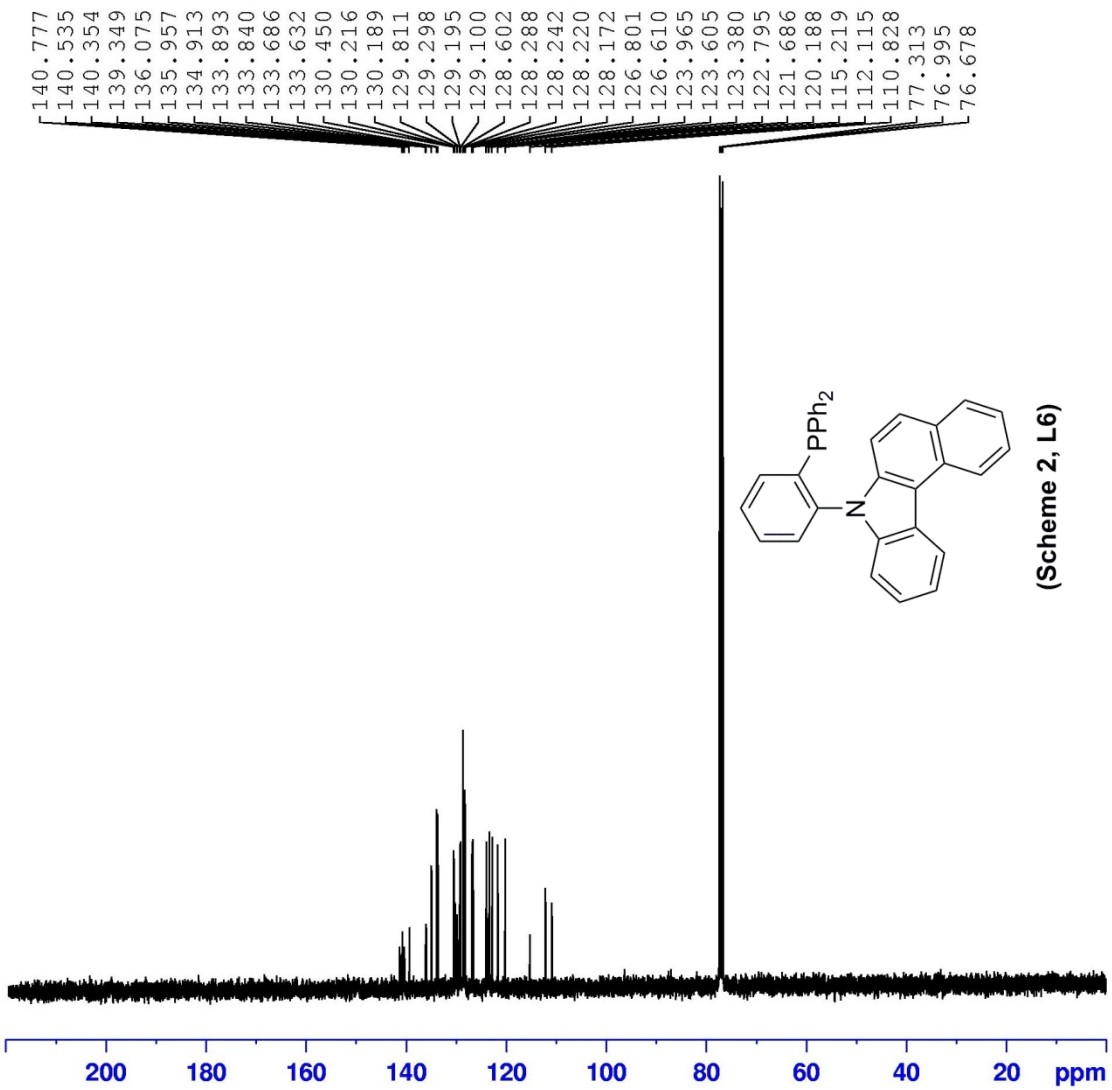




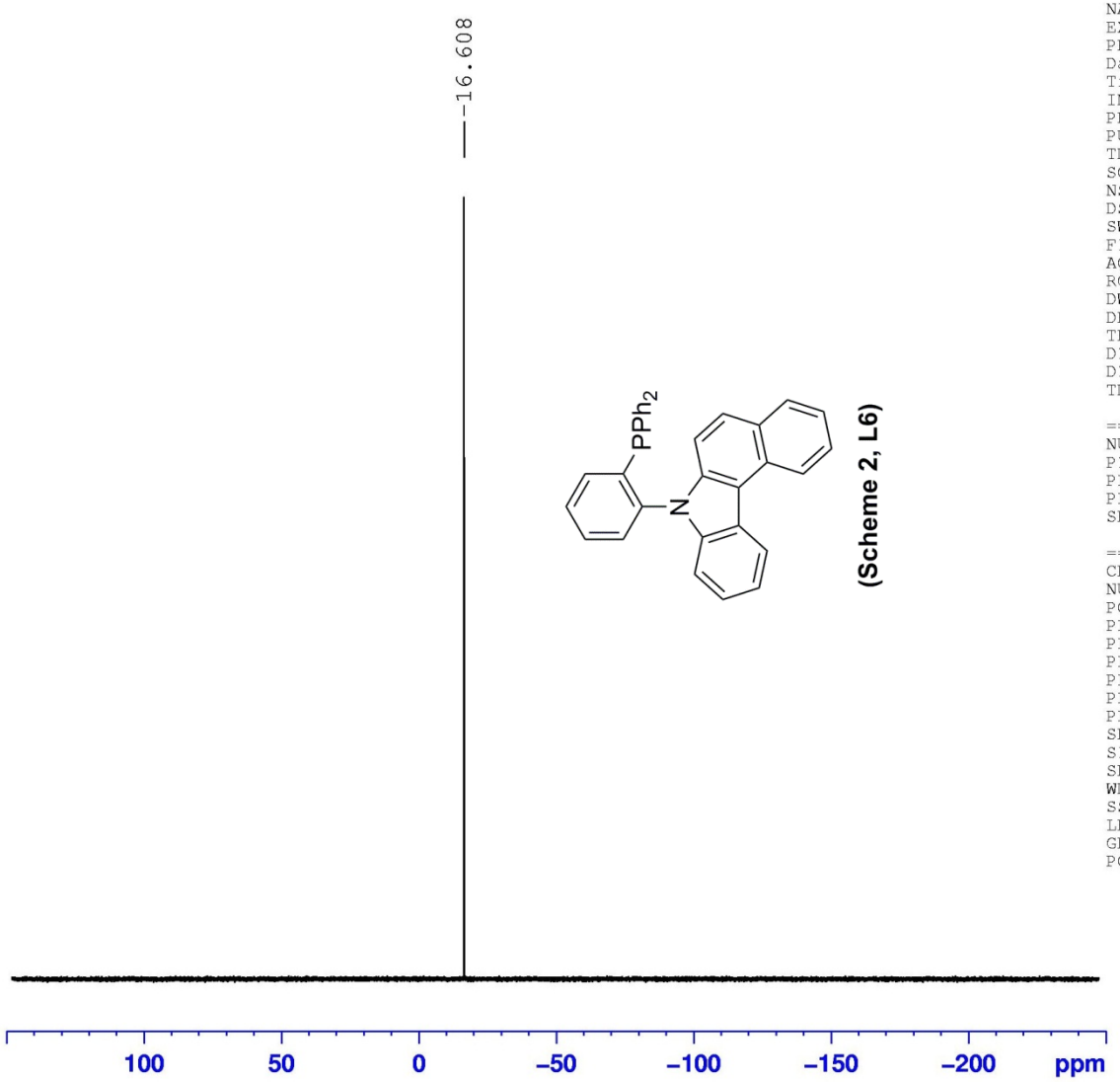
NO.	DATE	FORMULA	THEO	MEASURED	DIFF (mDa)	PPM	OTHER
S24	12112015	C22 H15 I N	420.0244	420.0248	0.4	1.0	
S24	12112015	C22 H15 Br N	372.0382	372.0385	0.3	0.8	



NAME	STL041
EXPNO	5
PROCNO	1
Date_	20151112
Time	20.14
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	16384
SOLVENT	CDC13
NS	16
DS	2
SWH	8012.820 Hz
FIDRES	0.489064 Hz
AQ	1.0224116 sec
RG	90.5
DW	62.400 usec
DE	6.50 usec
TE	298.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.70 usec
PL1	0.00 dB
PL1W	11.88122272 W
SFO1	400.1336012 MHz
SI	32768
SF	400.1300055 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

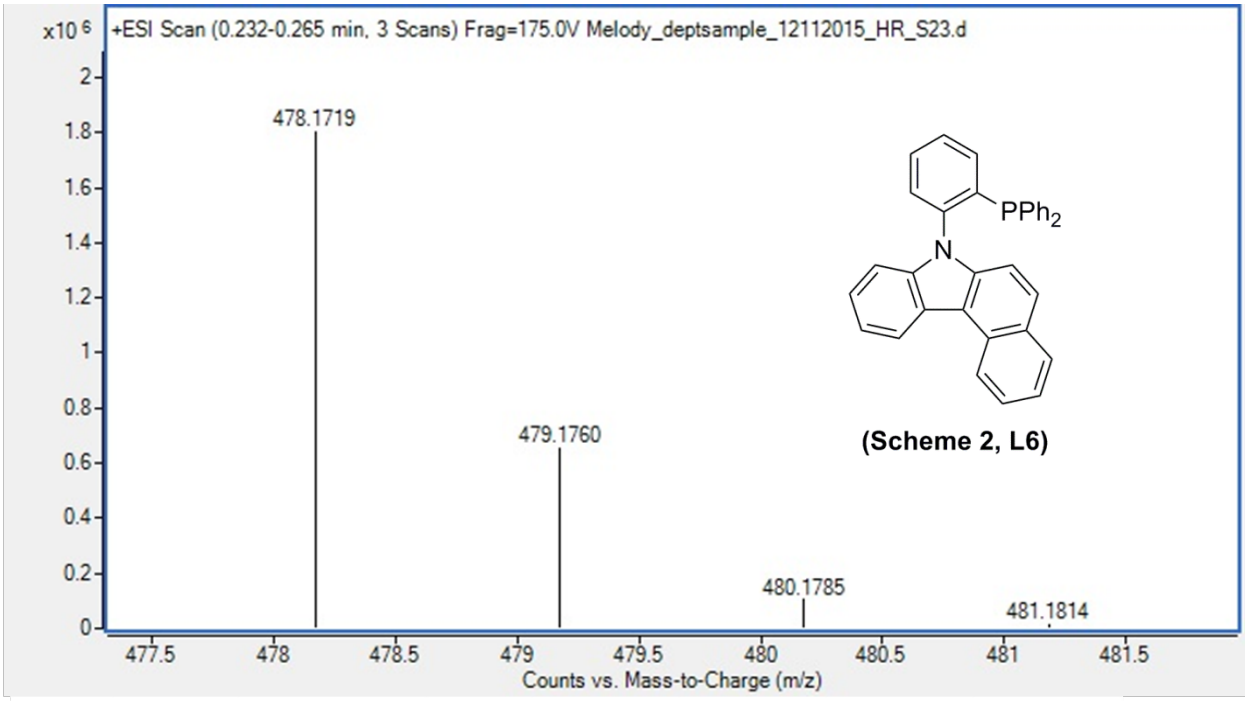


NAME	STL041
EXPNO	6
PROCNO	1
Date_	20151112
Time	20.18
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	129
DS	2
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	228
DW	20.800 usec
DE	6.50 usec
TE	299.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.50 usec
PL1	-2.00 dB
PL1W	58.52175522 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	15.00 dB
PL13	15.00 dB
PL2W	11.88122272 W
PL12W	0.37571725 W
PL13W	0.37571725 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127737 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

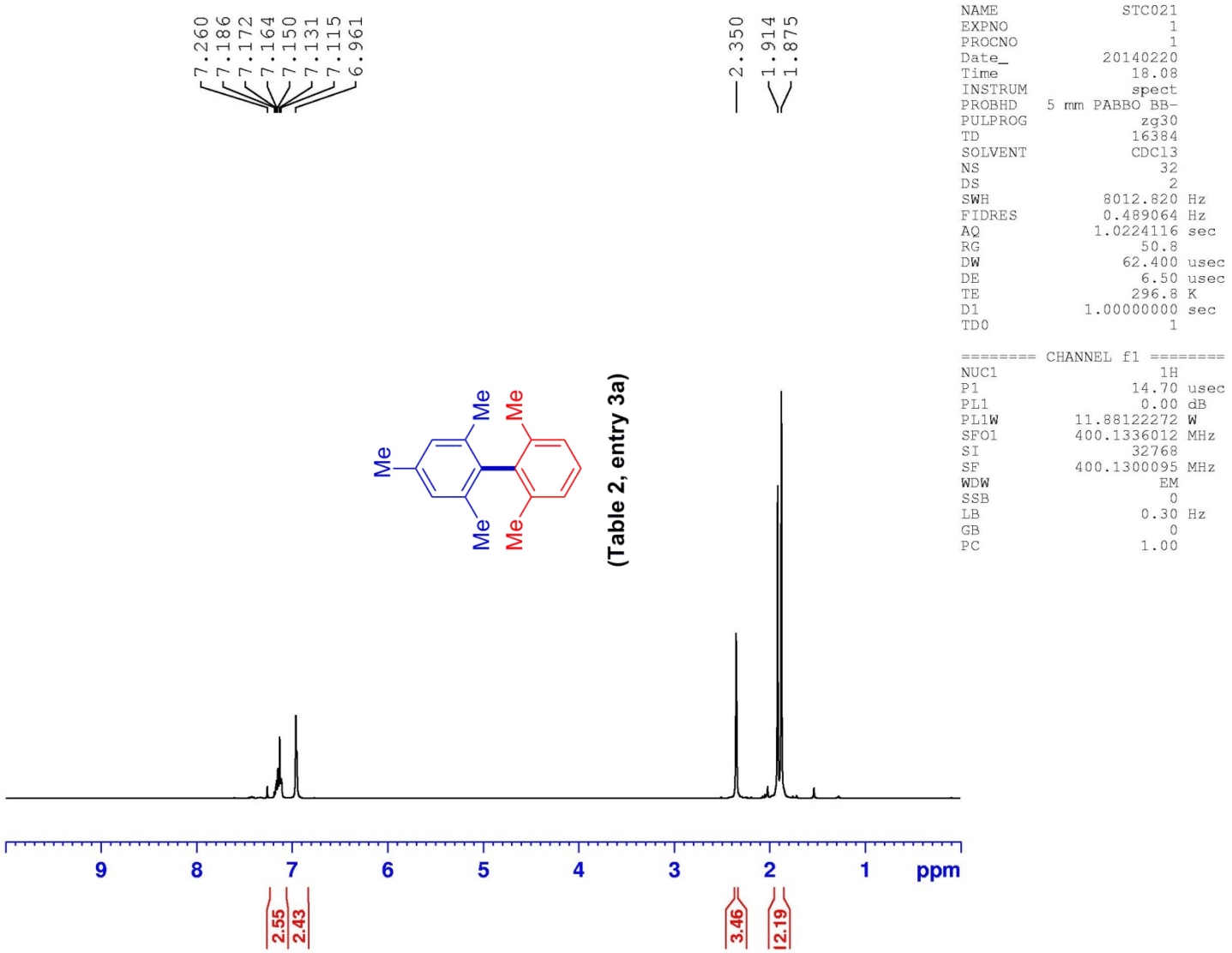


(Scheme 2, L6)

NAME	STL041
EXPNO	7
PROCNO	1
Date_	20151112
Time	20.24
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDC13
NS	32
DS	4
SWH	64102.563 Hz
FIDRES	0.978127 Hz
AQ	0.5112308 sec
RG	2050
DW	7.800 usec
DE	6.50 usec
TE	298.8 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	31P
P1	14.70 usec
PL1	3.00 dB
PL1W	12.96693134 W
SFO1	161.9674942 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	15.00 dB
PL13	15.00 dB
PL2W	11.88122272 W
PL12W	0.37571725 W
PL13W	0.37571725 W
SFO2	400.1316005 MHz
SI	32768
SF	161.9755930 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



NO.	DATE	FORMULA	THEO	MEASURED	DIFF (mDa)	PPM	OTHER
S23	12112015	C34 H25 N P	478.1719	478.1719	0	0.0	

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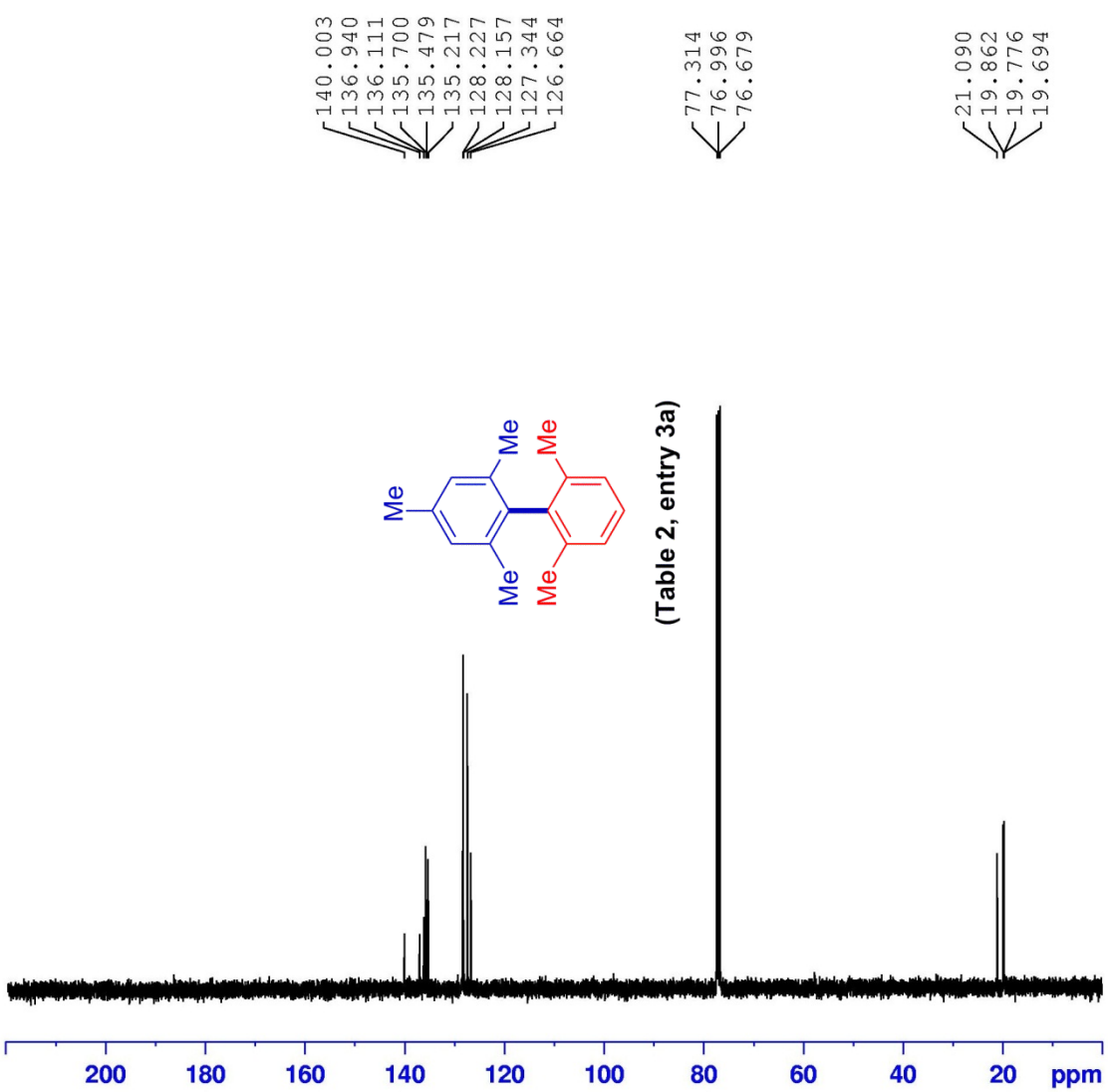
NAME                STC021
EXPNO                    1
PROCNO                   1
Date_             20140220
Time                 18.08
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG               zg30
TD                   16384
SOLVENT              CDC13
NS                      32
DS                       2
SWH               8012.820 Hz
FIDRES               0.489064 Hz
AQ                1.0224116 sec
RG                      50.8
DW                   62.400 usec
DE                    6.50 usec
TE                   296.8 K
D1                1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   14.70 usec
PL1                   0.00 dB
PL1W             11.88122272 W
SFO1            400.1336012 MHz
SI                   32768
SF               400.1300095 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```




```
NAME                STC021
EXPNO                    2
PROCNO                   1
Date_             20140220
Time                 18.13
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT                CDCl3
NS                      64
DS                       2
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631988 sec
RG                     203
DW                  20.800 usec
DE                   6.50 usec
TE                  298.0 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    13C
P1                    9.50 usec
PL1                  -2.00 dB
PL1W           58.52175522 W
SFO1           100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                   0.00 dB
PL12                 15.00 dB
PL13                 15.00 dB
PL2W           11.88122272 W
PL12W           0.37571725 W
PL13W           0.37571725 W
SFO2           400.1316005 MHz
SI                   32768
SF             100.6127722 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
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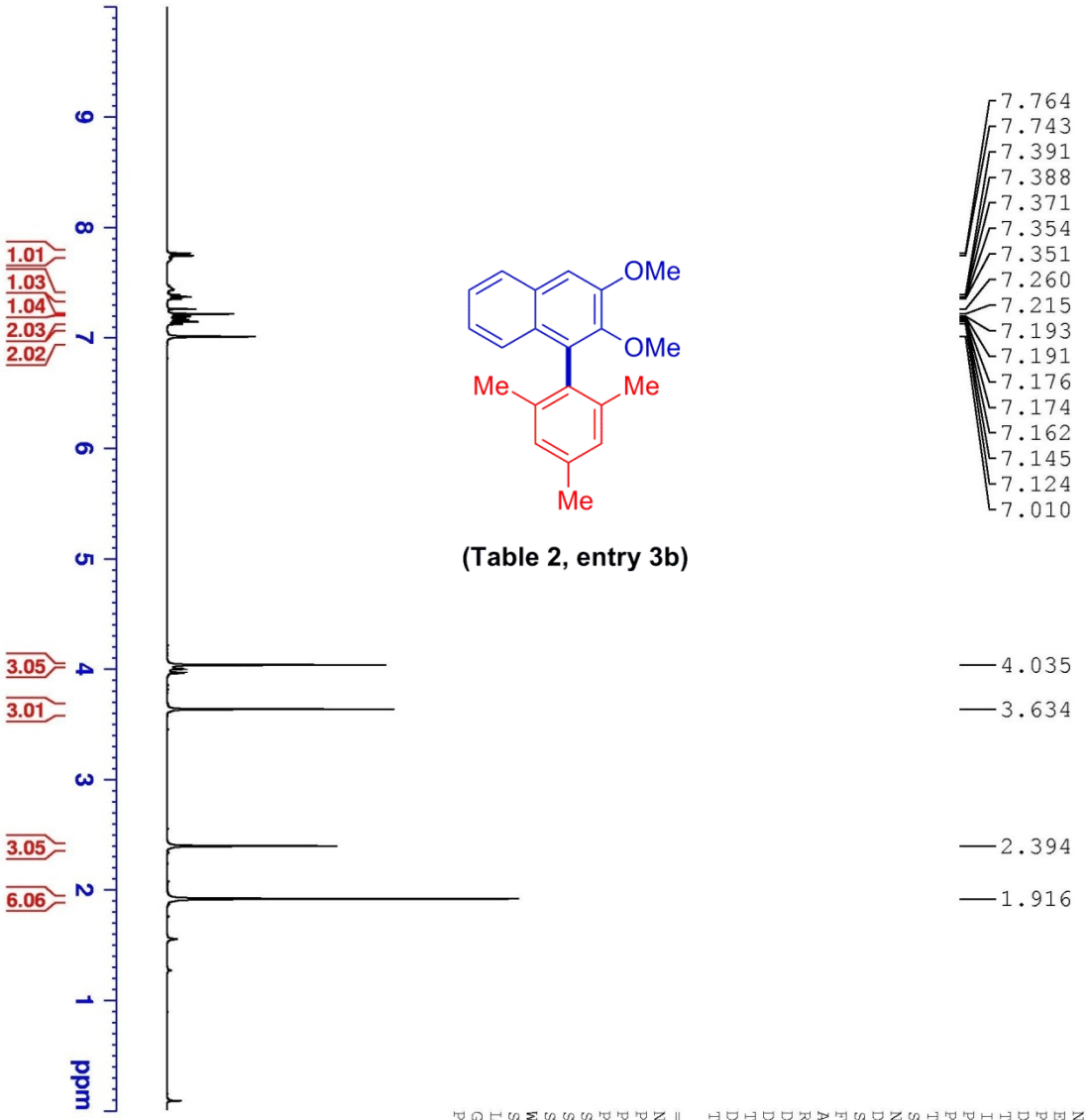
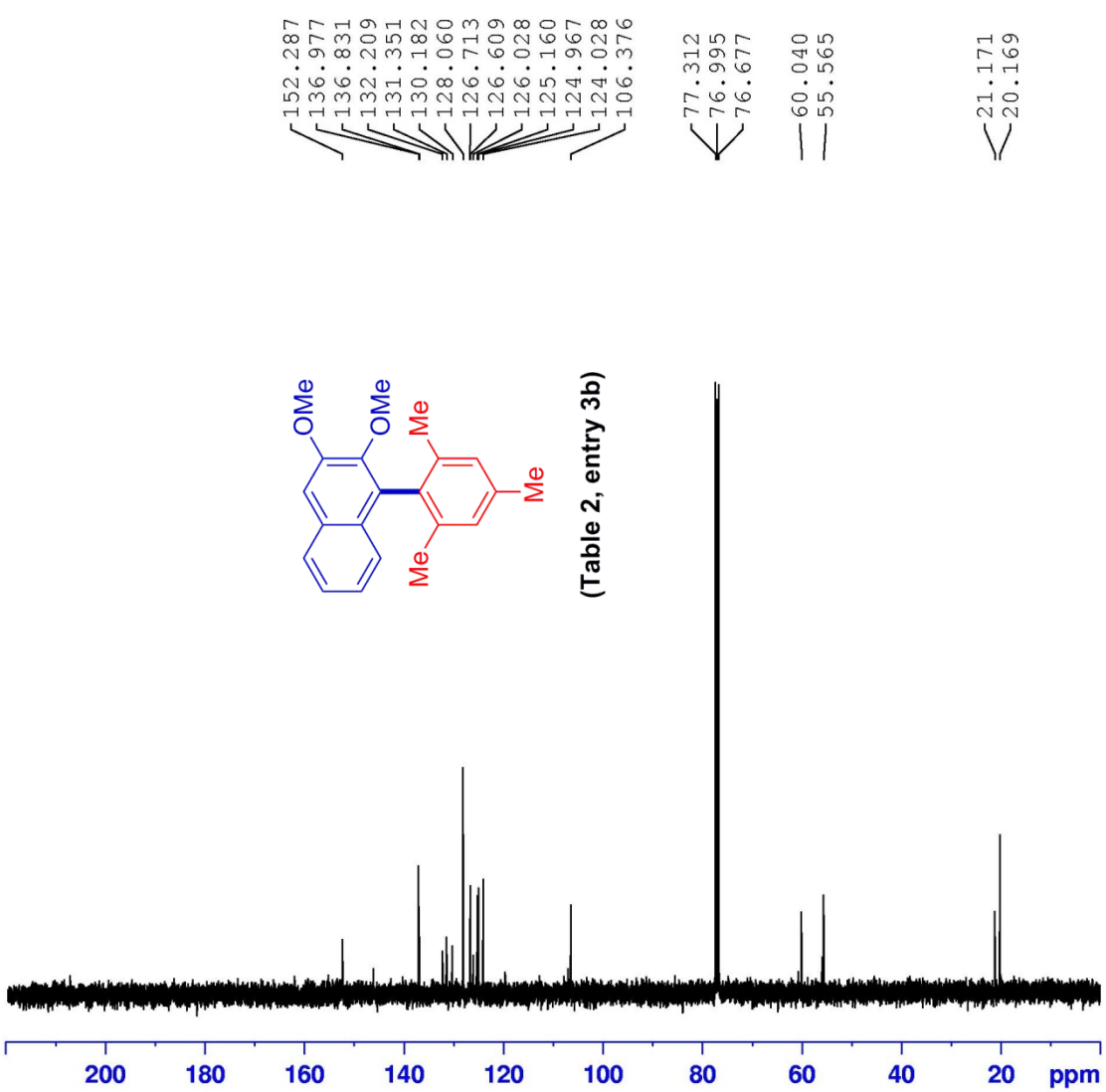
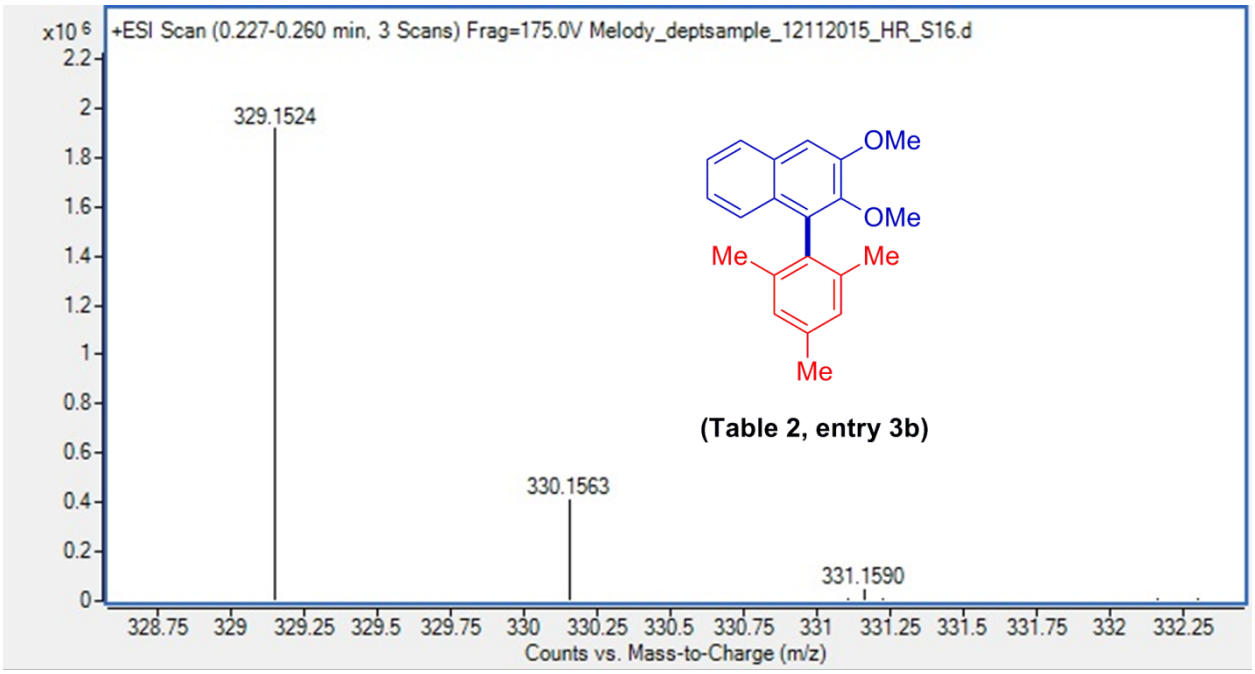


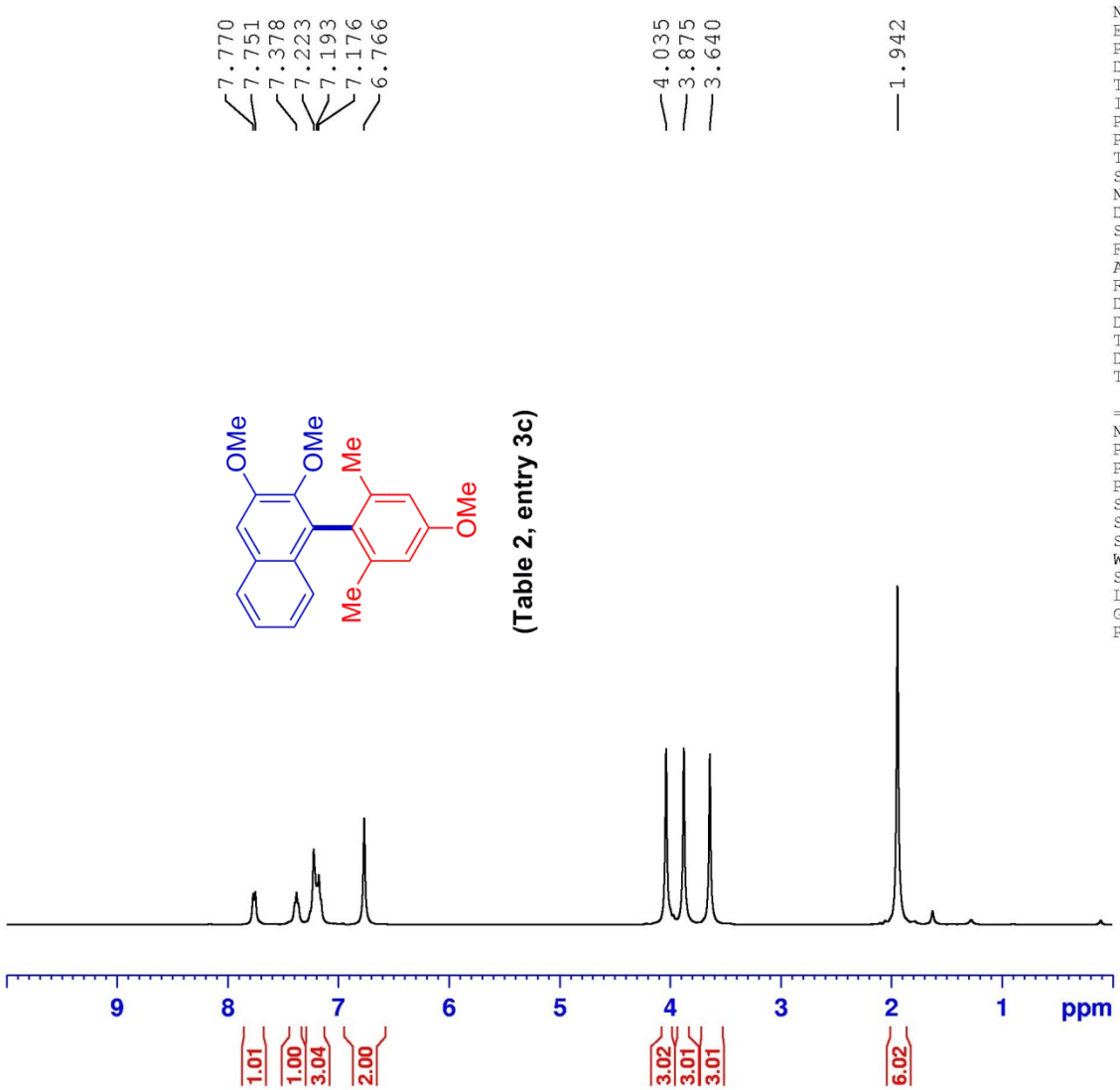
(Table 2, entry 3b)



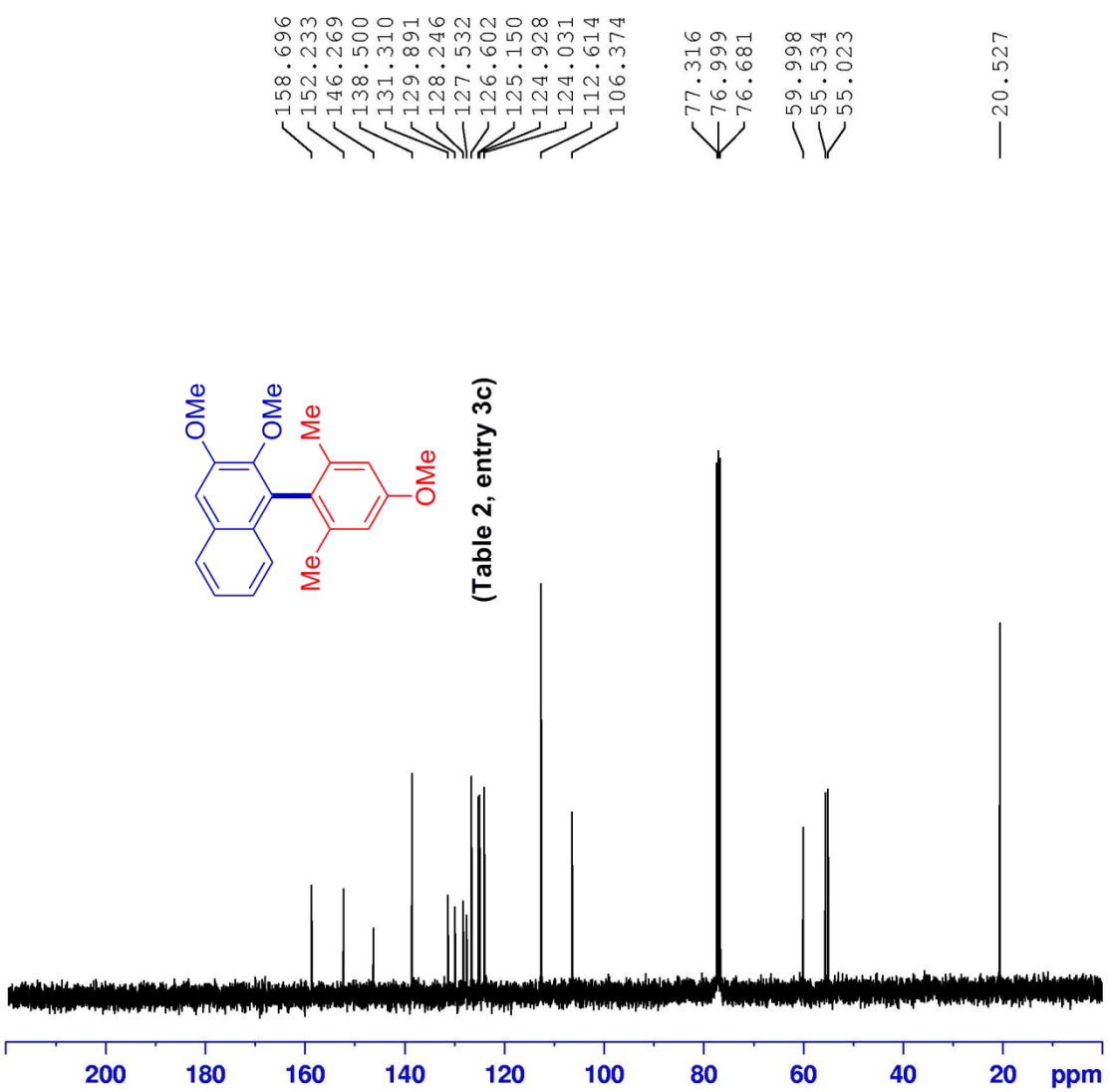
NAME	STC514C
EXPNO	2
PROCNO	1
Date_	20151110
Time	17.38
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	64
DS	2
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	181
DW	20.800 usec
DE	6.50 usec
TE	299.6 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.50 usec
PL1	-2.00 dB
PL1W	58.52175522 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	15.00 dB
PL13	15.00 dB
PL2W	11.88122272 W
PL12W	0.37571725 W
PL13W	0.37571725 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127715 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



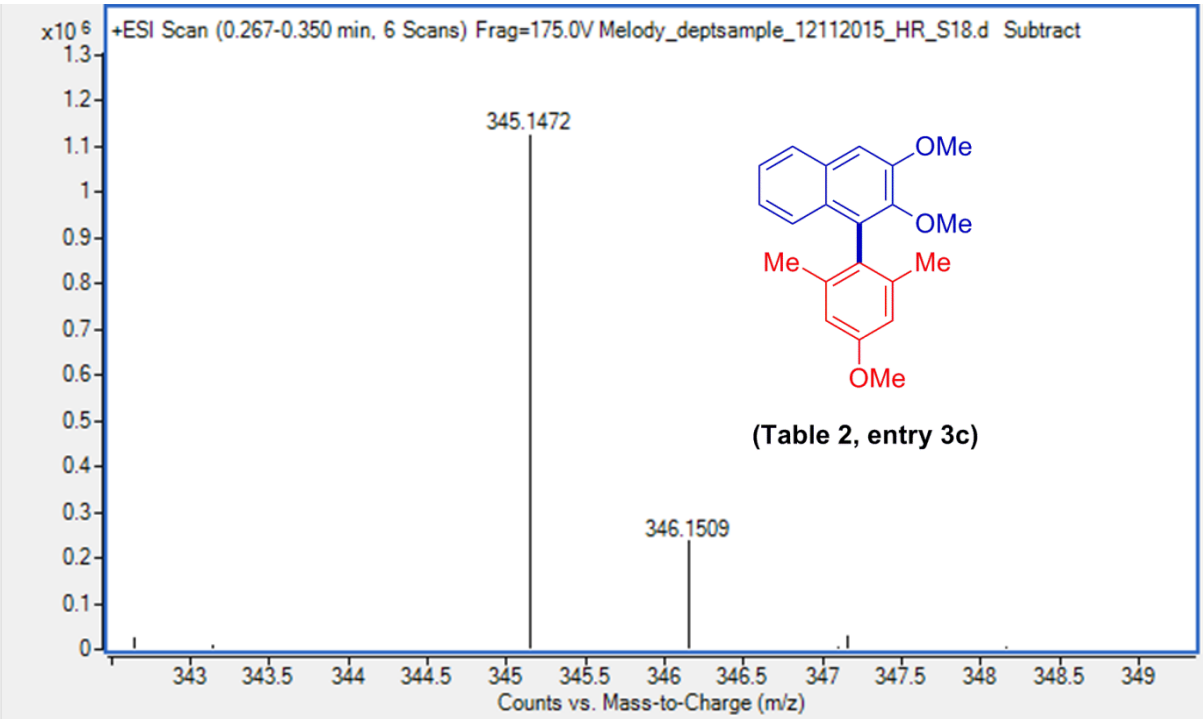
NO.	DATE	FORMULA	THEO	MEASURED	DIFF (mDa)	PPM	OTHER
S16	12112015	C21H22O2Na	329.1512	329.1524	1.2	3.6	



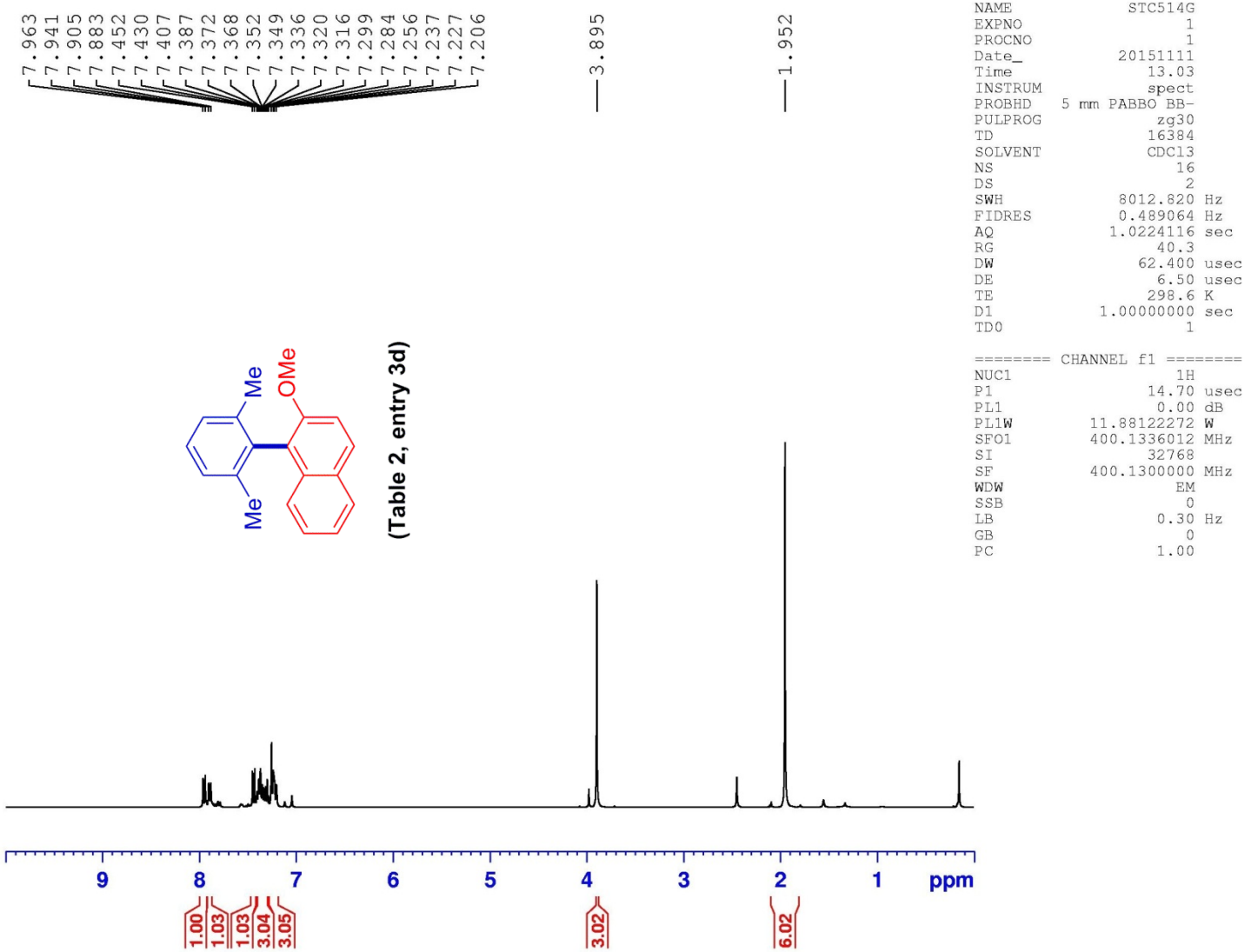
NAME	STC527E
EXPNO	1
PROCNO	1
Date_	20151109
Time	12.04
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	16384
SOLVENT	CDC13
NS	16
DS	2
SWH	8012.820 Hz
FIDRES	0.489064 Hz
AQ	1.0224116 sec
RG	32
DW	62.400 usec
DE	6.50 usec
TE	297.1 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.70 usec
PL1	0.00 dB
PL1W	11.88122272 W
SFO1	400.1336012 MHz
SI	32768
SF	400.1300097 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



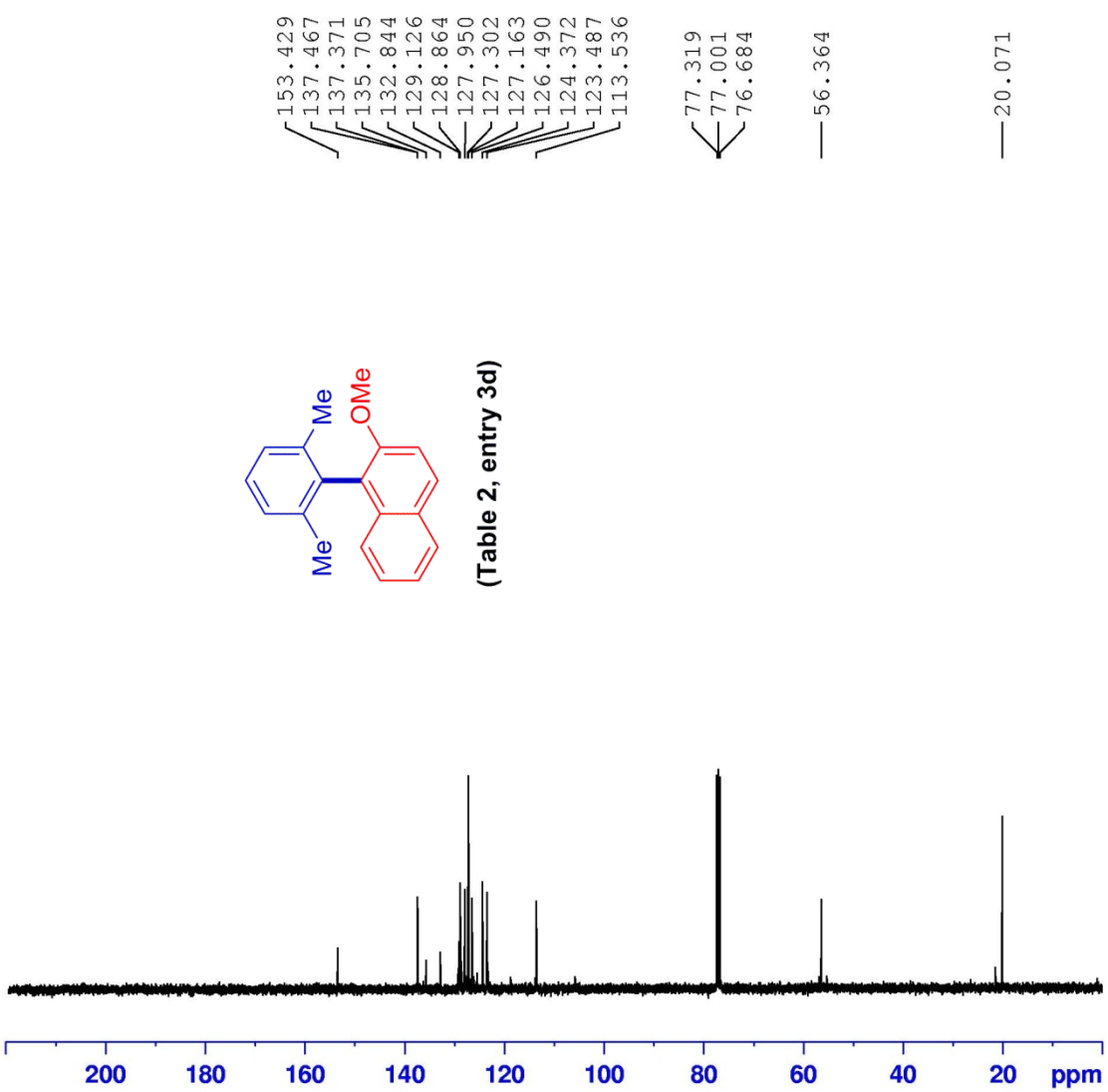

```
NAME              STC527E
EXPNO                   2
PROCNO                  1
Date_            20151109
Time                12.08
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     2
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                   181
DW                20.800 usec
DE                 6.50 usec
TE                297.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.50 usec
PL1                -2.00 dB
PL1W        58.52175522 W
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 0.00 dB
PL12               15.00 dB
PL13               15.00 dB
PL2W        11.88122272 W
PL12W        0.37571725 W
PL13W        0.37571725 W
SFO2       400.1316005 MHz
SI                 32768
SF         100.6127765 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



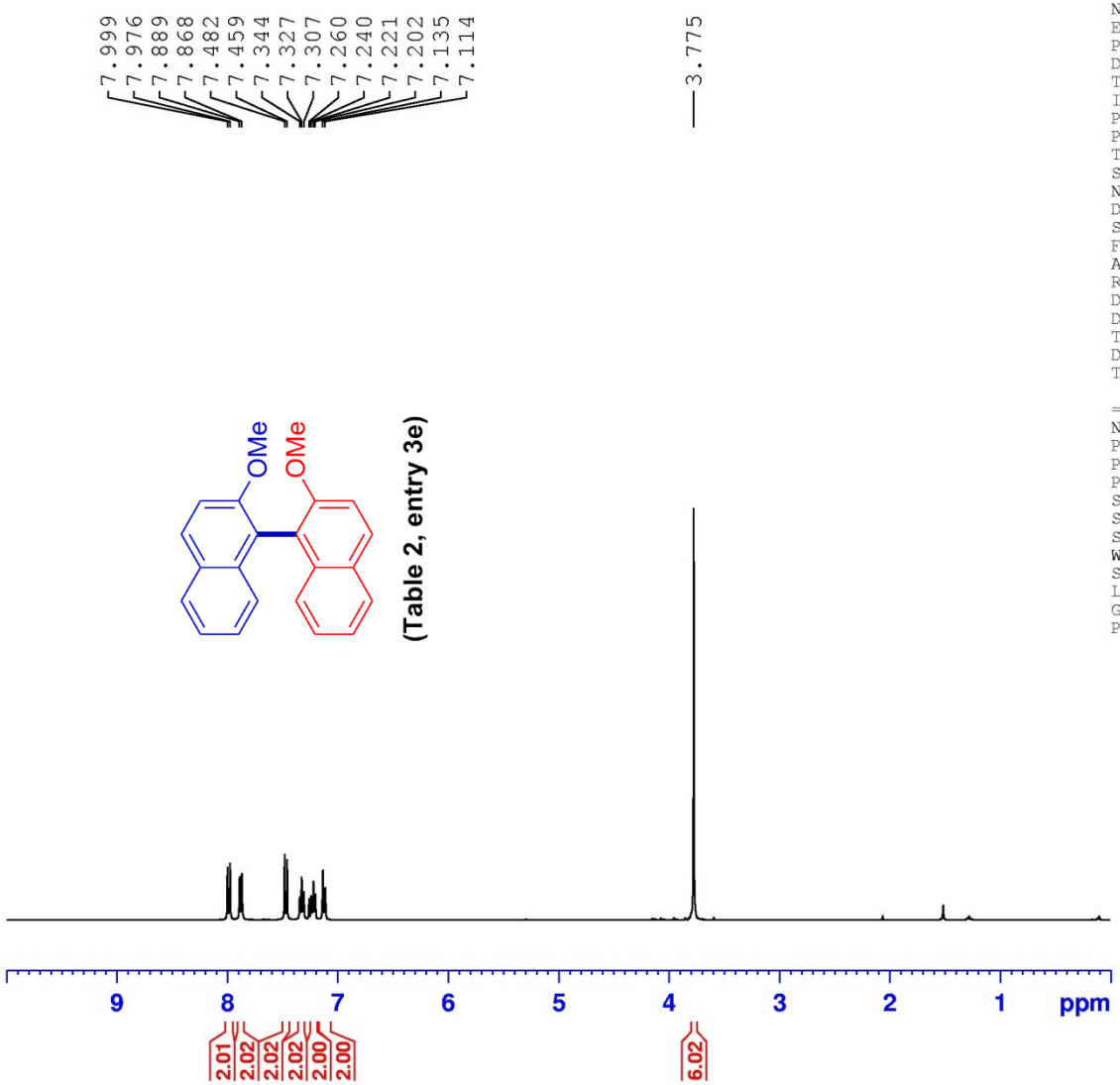
NO.	DATE	FORMULA	THEO	MEASURED	DIFF (mDa)	PPM	OTHER
S18	12112015	C21H22O3Na	345.1461	345.1472	1.1	3.2	



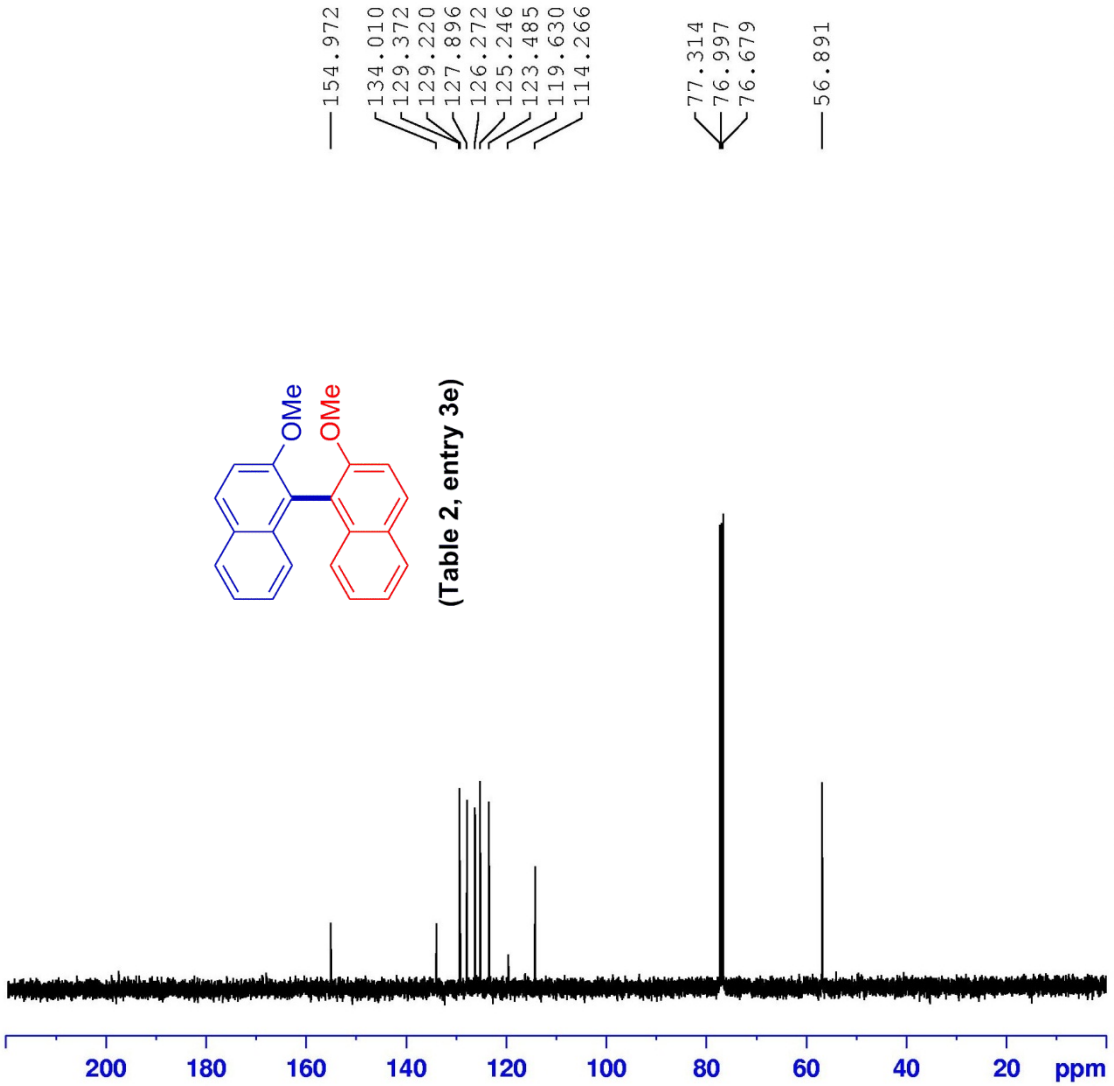
NAME	STC514G
EXPNO	1
PROCNO	1
Date_	20151111
Time	13.03
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	16384
SOLVENT	CDCl3
NS	16
DS	2
SWH	8012.820 Hz
FIDRES	0.489064 Hz
AQ	1.0224116 sec
RG	40.3
DW	62.400 usec
DE	6.50 usec
TE	298.6 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.70 usec
PL1	0.00 dB
PL1W	11.88122272 W
SFO1	400.1336012 MHz
SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



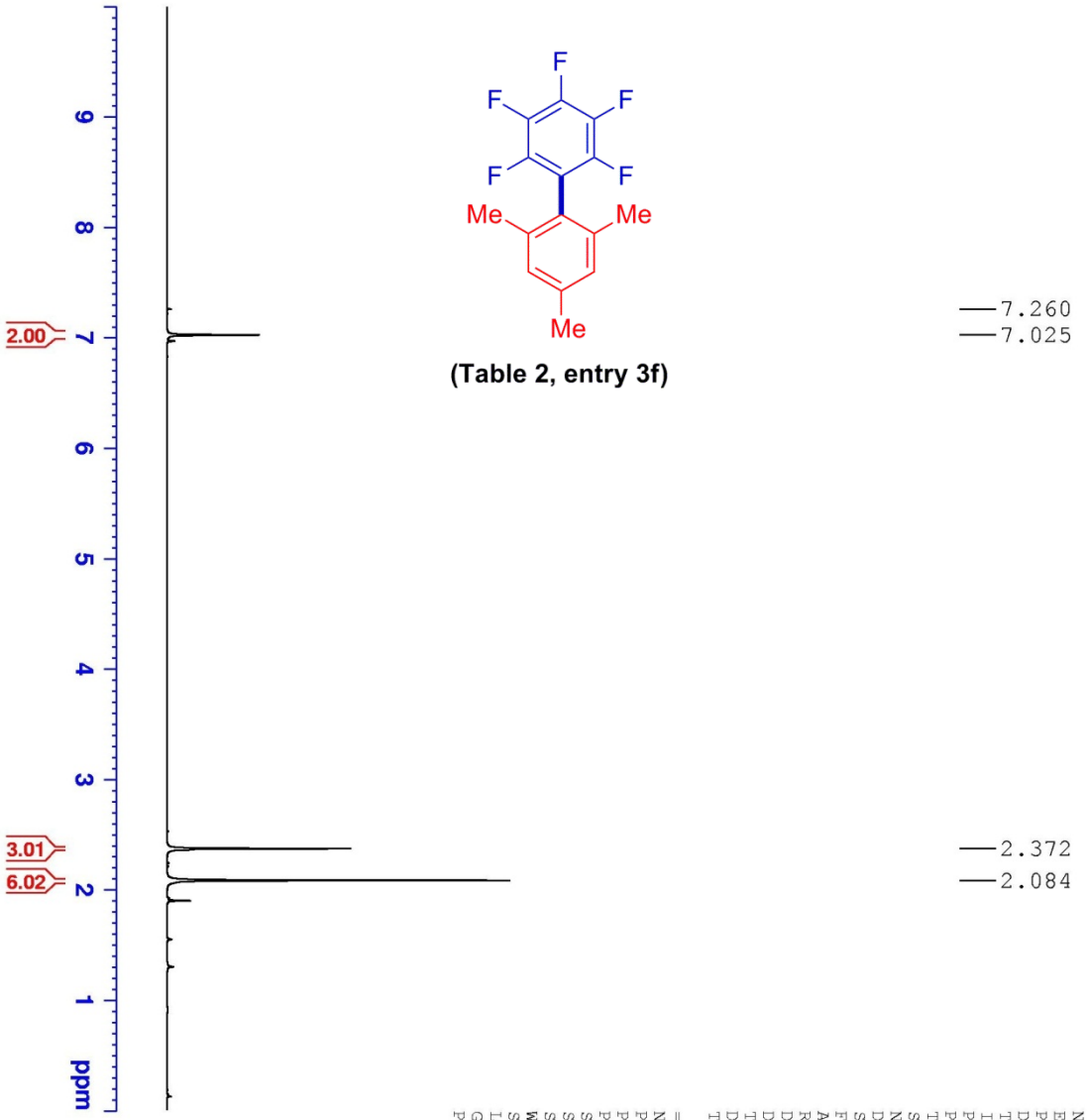

```
NAME              STC514G
EXPNO                   3
PROCNO                  1
Date_            20151111
Time                13.10
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT             CDCl3
NS                     34
DS                      2
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                    128
DW                 20.800 usec
DE                  6.50 usec
TE                 299.5 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.50 usec
PL1                 -2.00 dB
PL1W         58.52175522 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 0.00 dB
PL12               15.00 dB
PL13               15.00 dB
PL2W         11.88122272 W
PL12W         0.37571725 W
PL13W         0.37571725 W
SFO2        400.1316005 MHz
SI                 32768
SF          100.6127751 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



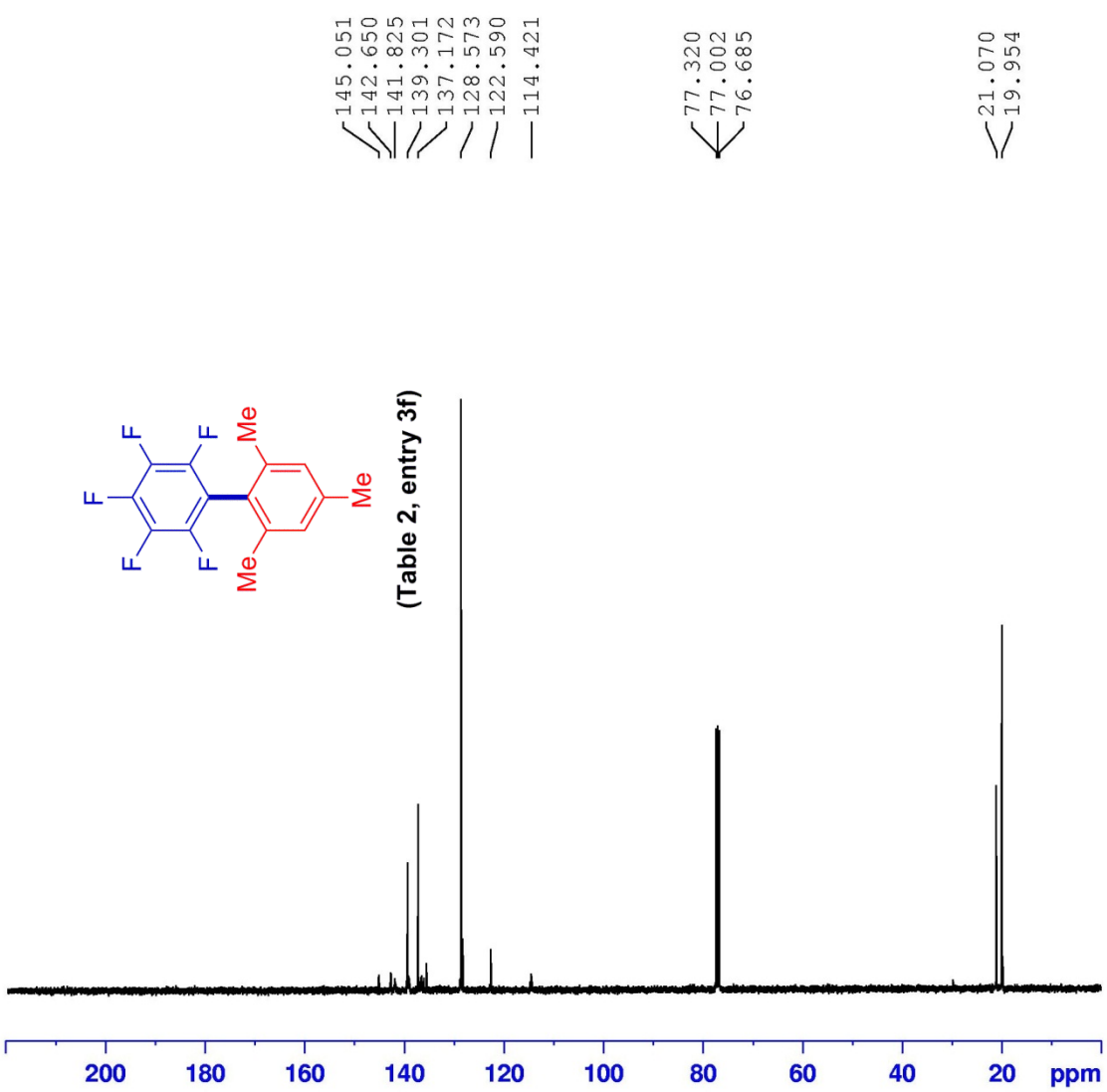

```
NAME              STC532F
EXPNO                   1
PROCNO                  1
Date_            20151110
Time                17.30
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG               zg30
TD                   16384
SOLVENT              CDC13
NS                      16
DS                       2
SWH               8012.820 Hz
FIDRES               0.489064 Hz
AQ                1.0224116 sec
RG                      128
DW                   62.400 usec
DE                    6.50 usec
TE                   299.0 K
D1                1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                    1H
P1                   14.70 usec
PL1                   0.00 dB
PL1W             11.88122272 W
SFO1             400.1336012 MHz
SI                   32768
SF               400.1300097 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



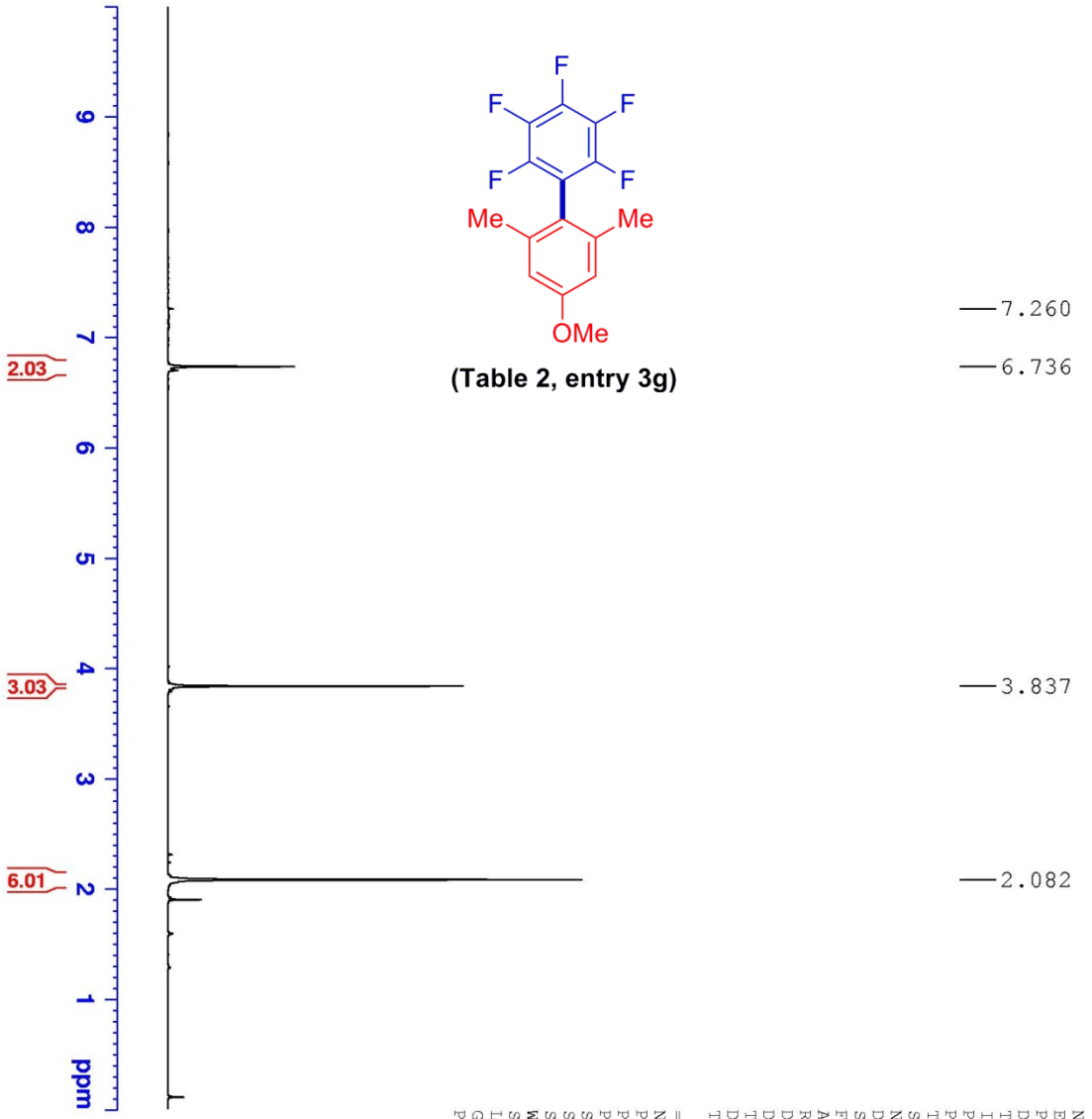

```
NAME              STC532F
EXPNO                   2
PROCNO                  1
Date_            20151110
Time                17.33
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT             CDCl3
NS                     50
DS                      2
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                    203
DW                 20.800 usec
DE                  6.50 usec
TE                 300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  9.50 usec
PL1                -2.00 dB
PL1W        58.52175522 W
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 0.00 dB
PL12               15.00 dB
PL13               15.00 dB
PL2W        11.88122272 W
PL12W        0.37571725 W
PL13W        0.37571725 W
SFO2       400.1316005 MHz
SI                 32768
SF         100.6127729 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



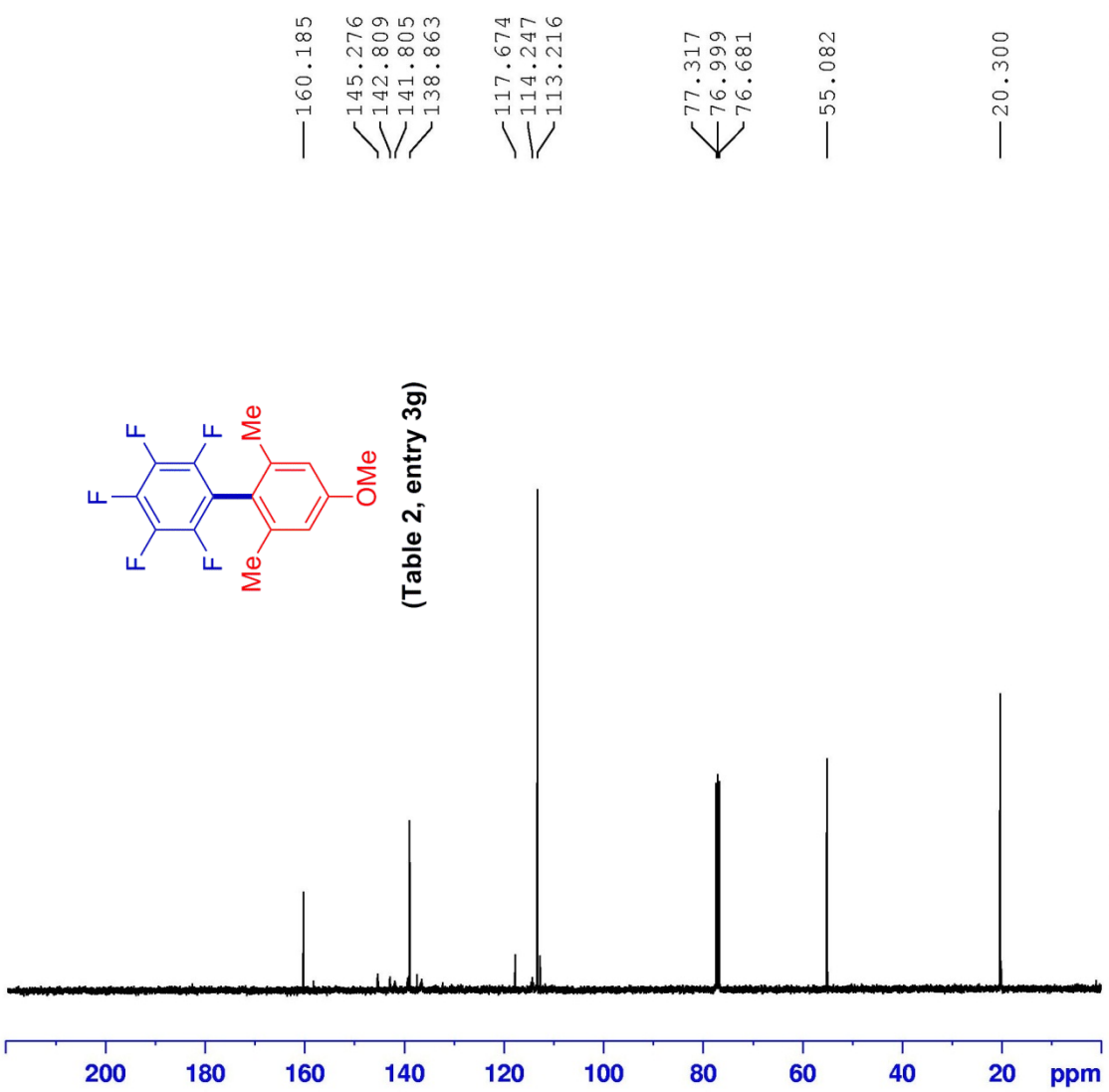
(Table 2, entry 3f)



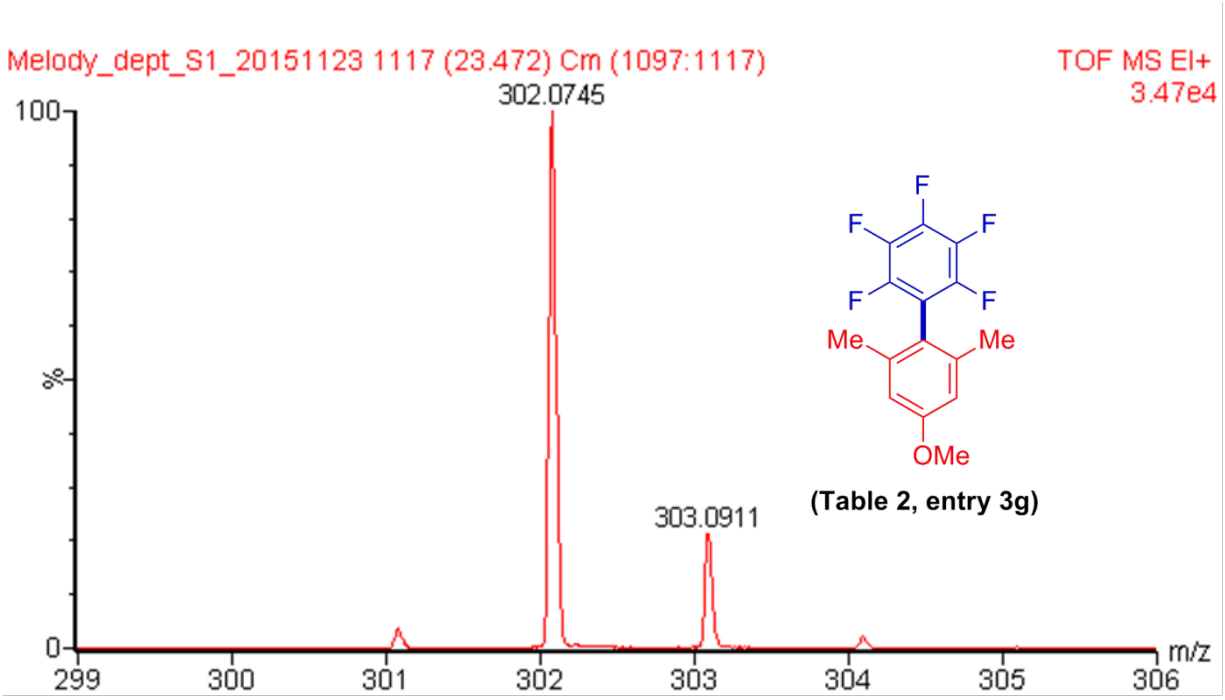

```
NAME              STC527A
EXPNO                   4
PROCNO                  1
Date_            20151105
Time                22.57
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT             CDC13
NS                    128
DS                      2
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                    203
DW               20.800 usec
DE                6.50 usec
TE                299.5 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                 9.50 usec
PL1                -2.00 dB
PL1W        58.52175522 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2             80.00 usec
PL2                0.00 dB
PL12              15.00 dB
PL13              15.00 dB
PL2W        11.88122272 W
PL12W        0.37571725 W
PL13W        0.37571725 W
SFO2        400.1316005 MHz
SI                 32768
SF          100.6127700 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                 1.40
```




```
NAME                                    STC530A
EXPNO                                         1
PROCNO                                        1
Date_                                  20151111
Time                                      13.30
INSTRUM                                   spect
PROBHD                 5 mm PABBO BB-
PULPROG                                    zg30
TD                                        16384
SOLVENT                                   CDCl3
NS                                           16
DS                                            2
SWH                                    8012.820 Hz
FIDRES                                 0.489064 Hz
AQ                                     1.0224116 sec
RG                                           40.3
DW                                     62.400 usec
DE                                      6.50 usec
TE                                     298.9 K
D1                                     1.00000000 sec
TD0                                           1

======== CHANNEL f1 ========
NUC1                                         1H
P1                                     14.70 usec
PL1                                     0.00 dB
PL1W                                11.88122272 W
SFO1                                400.1336012 MHz
SI                                        32768
SF                                  400.1300097 MHz
WDW                                          EM
SSB                                           0
LB                                     0.30 Hz
GB                                            0
PC                                         1.00
```

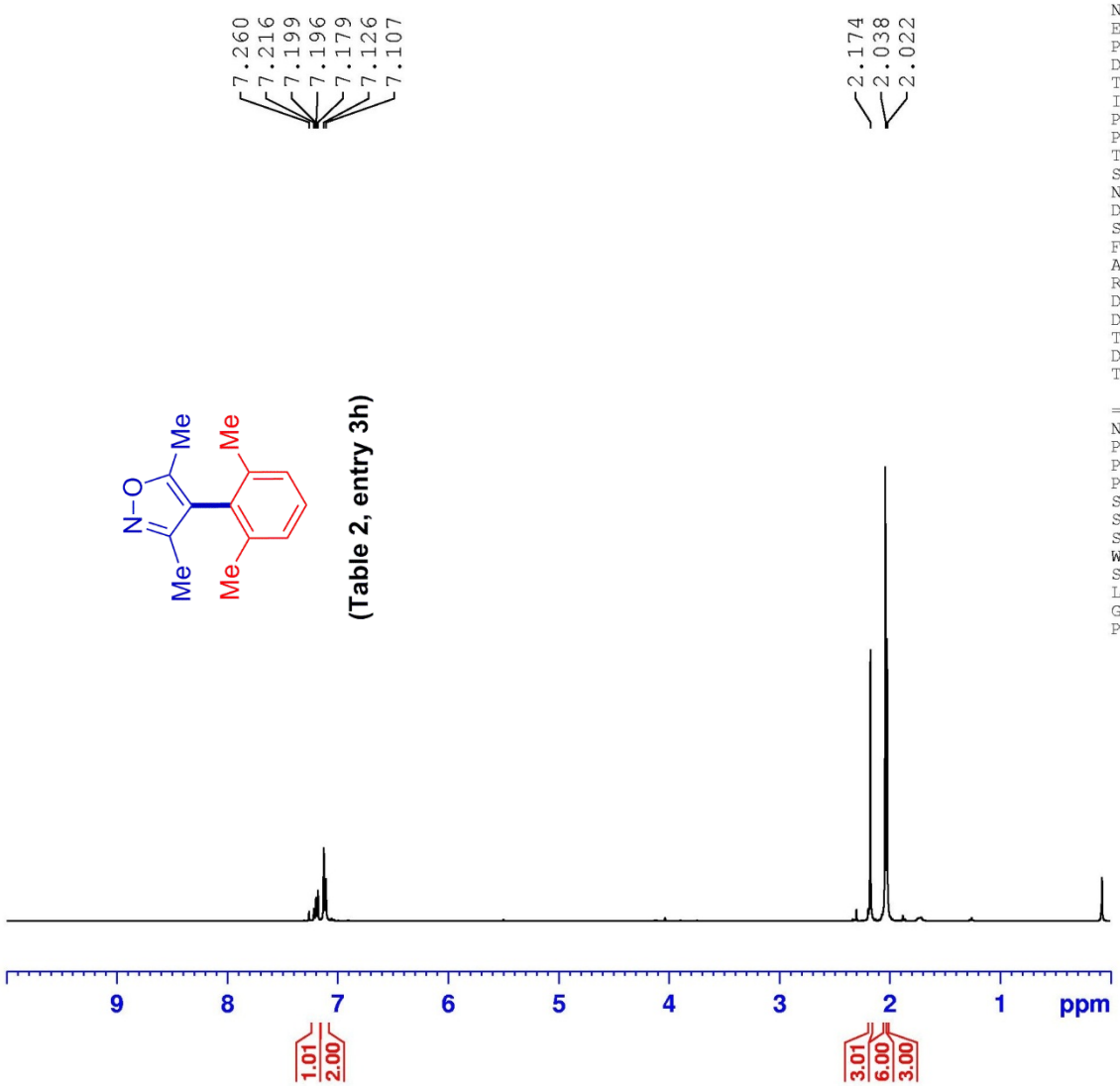


NAME	STC530A
EXPNO	2
PROCNO	1
Date_	20151111
Time	13.33
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	94
DS	2
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	299.8 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.50 usec
PL1	-2.00 dB
PL1W	58.52175522 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	15.00 dB
PL13	15.00 dB
PL2W	11.88122272 W
PL12W	0.37571725 W
PL13W	0.37571725 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127706 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

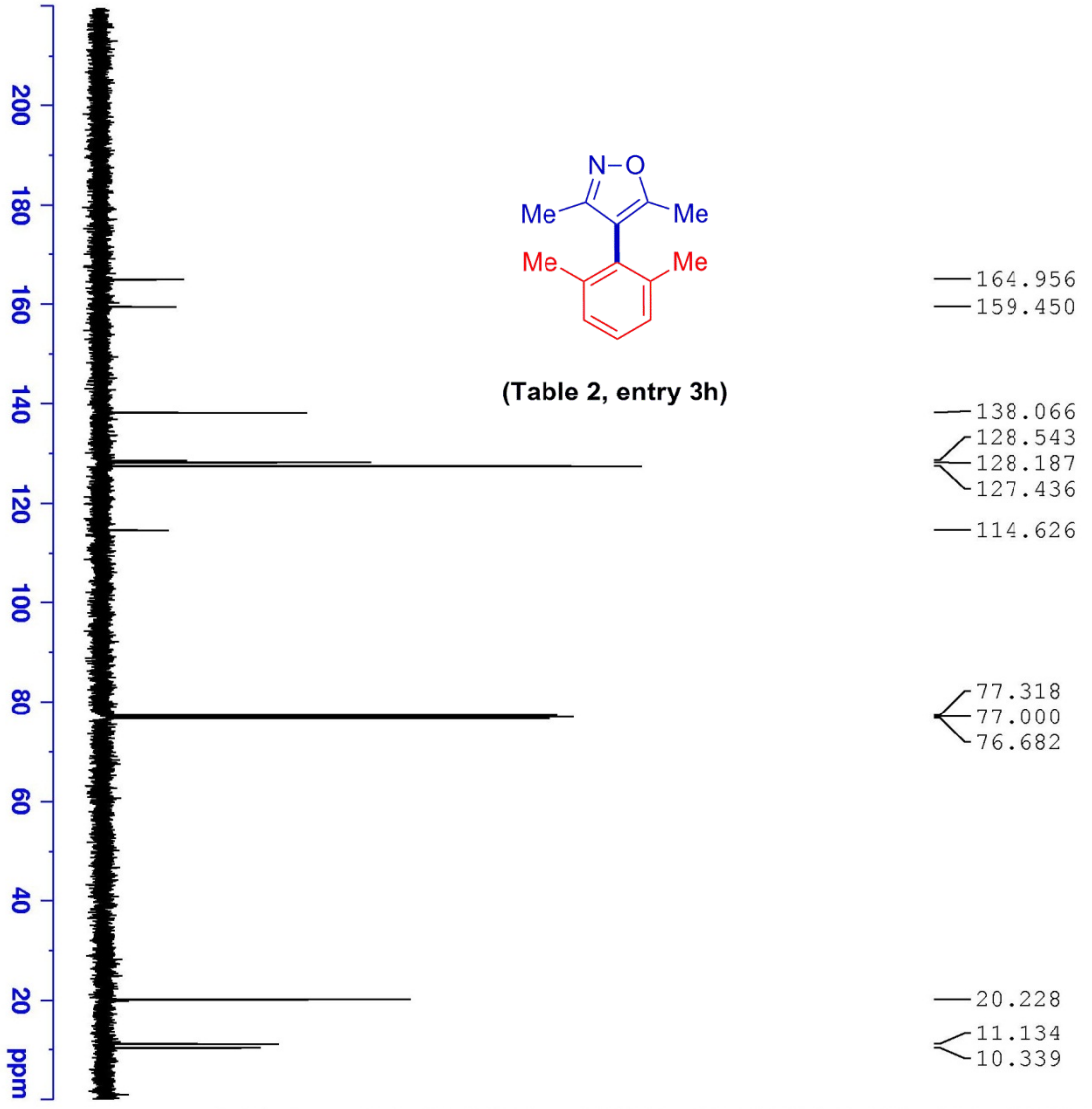


(Table 2, entry 3g)

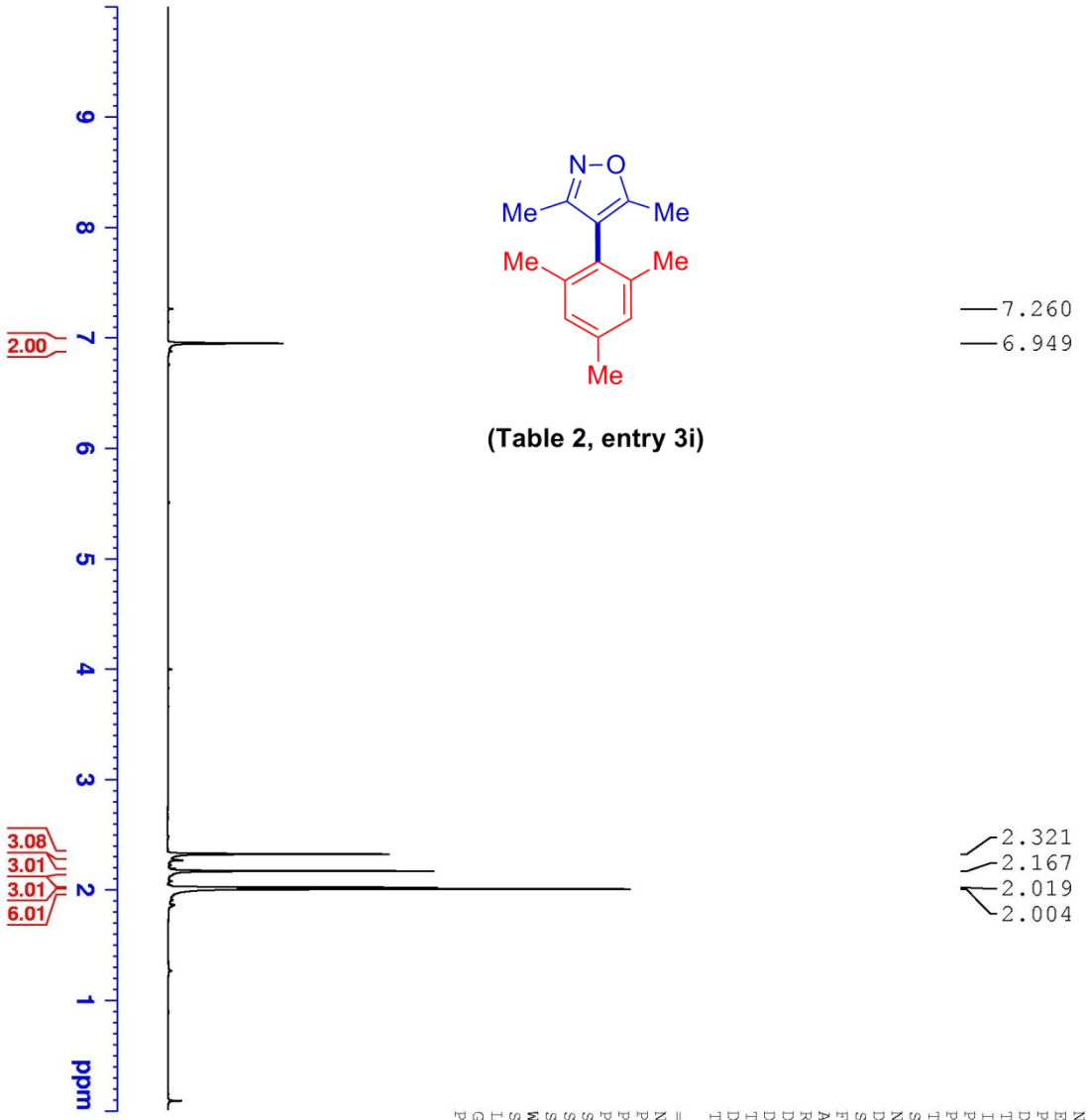
Mass	Calc. Mass	mDa	PPM	Formula
302.0745	302.0725	2.0	6.6	C15H11F5O

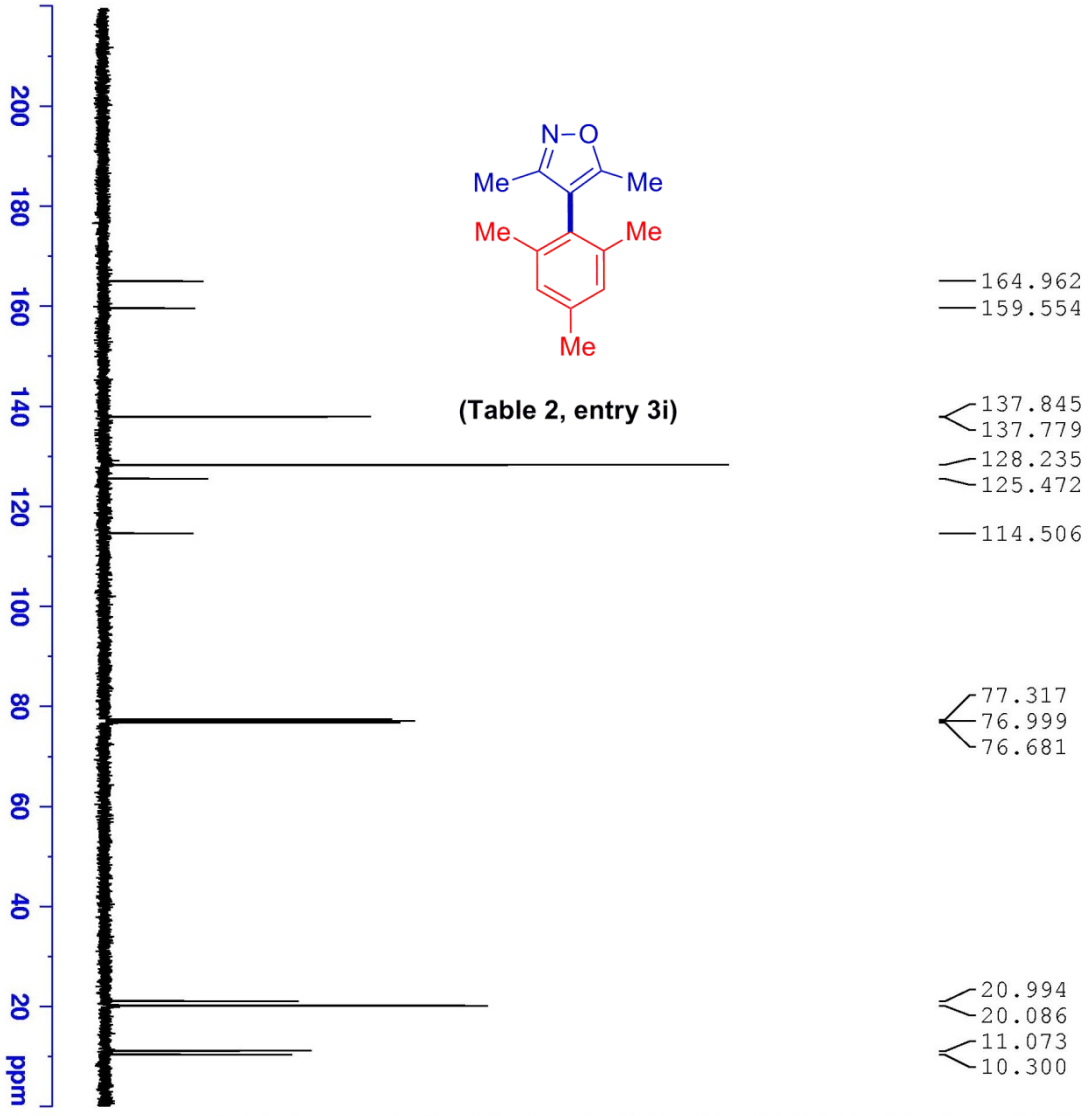


NAME	STC529A
EXPNO	1
PROCNO	1
Date_	20151110
Time	17.23
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	16384
SOLVENT	CDCl3
NS	16
DS	2
SWH	8012.820 Hz
FIDRES	0.489064 Hz
AQ	1.0224116 sec
RG	64
DW	62.400 usec
DE	6.50 usec
TE	298.8 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.70 usec
PL1	0.00 dB
PL1W	11.88122272 W
SFO1	400.1336012 MHz
SI	32768
SF	400.1300097 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

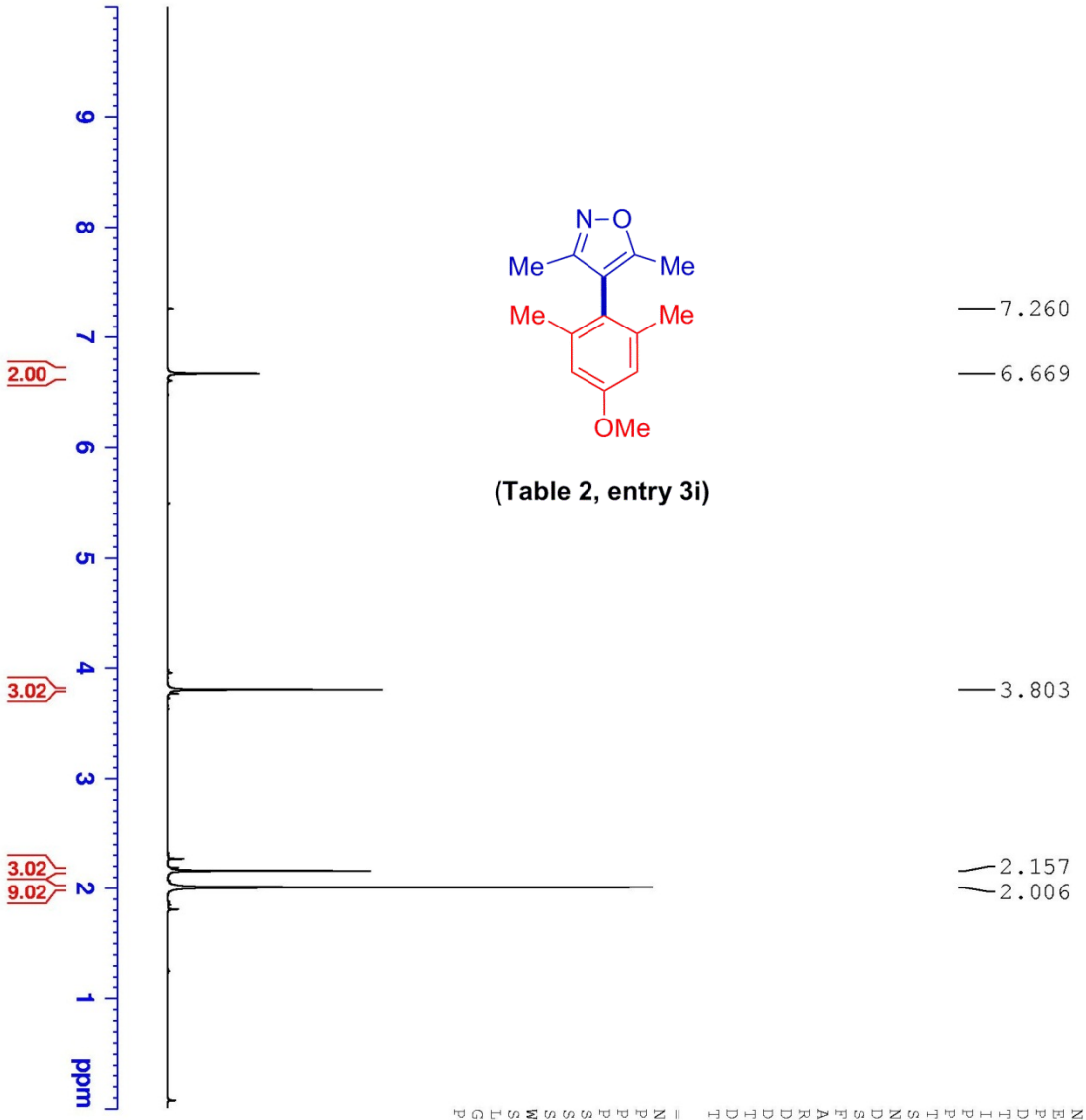



```
NAME                                    STC529A
EXPNO                                         2
PROCNO                                        1
Date_                               20151110
Time                                    17.26
INSTRUM                                  spect
PROBHD                  5 mm PABBO BB-
PULPROG                                zgpg30
TD                                      65536
SOLVENT                                 CDCl3
NS                                         33
DS                                          2
SWH                                 24038.461 Hz
FIDRES                                0.366798 Hz
AQ                                    1.3631988 sec
RG                                        203
DW                                     20.800 usec
DE                                      6.50 usec
TE                                     299.7 K
D1                                    2.00000000 sec
D11                                   0.03000000 sec
TD0                                        1

======== CHANNEL f1 ========
NUC1                                      13C
P1                                       9.50 usec
PL1                                     -2.00 dB
PL1W                                 58.52175522 W
SFO1                                100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                                waltz16
NUC2                                       1H
PCPD2                                  80.00 usec
PL2                                     0.00 dB
PL12                                   15.00 dB
PL13                                   15.00 dB
PL2W                                11.88122272 W
PL12W                                0.37571725 W
PL13W                                0.37571725 W
SFO2                                400.1316005 MHz
SI                                         32768
SF                                  100.6127736 MHz
WDW                                        EM
SSB                                         0
LB                                       1.00 Hz
GB                                          0
PC                                       1.40
```

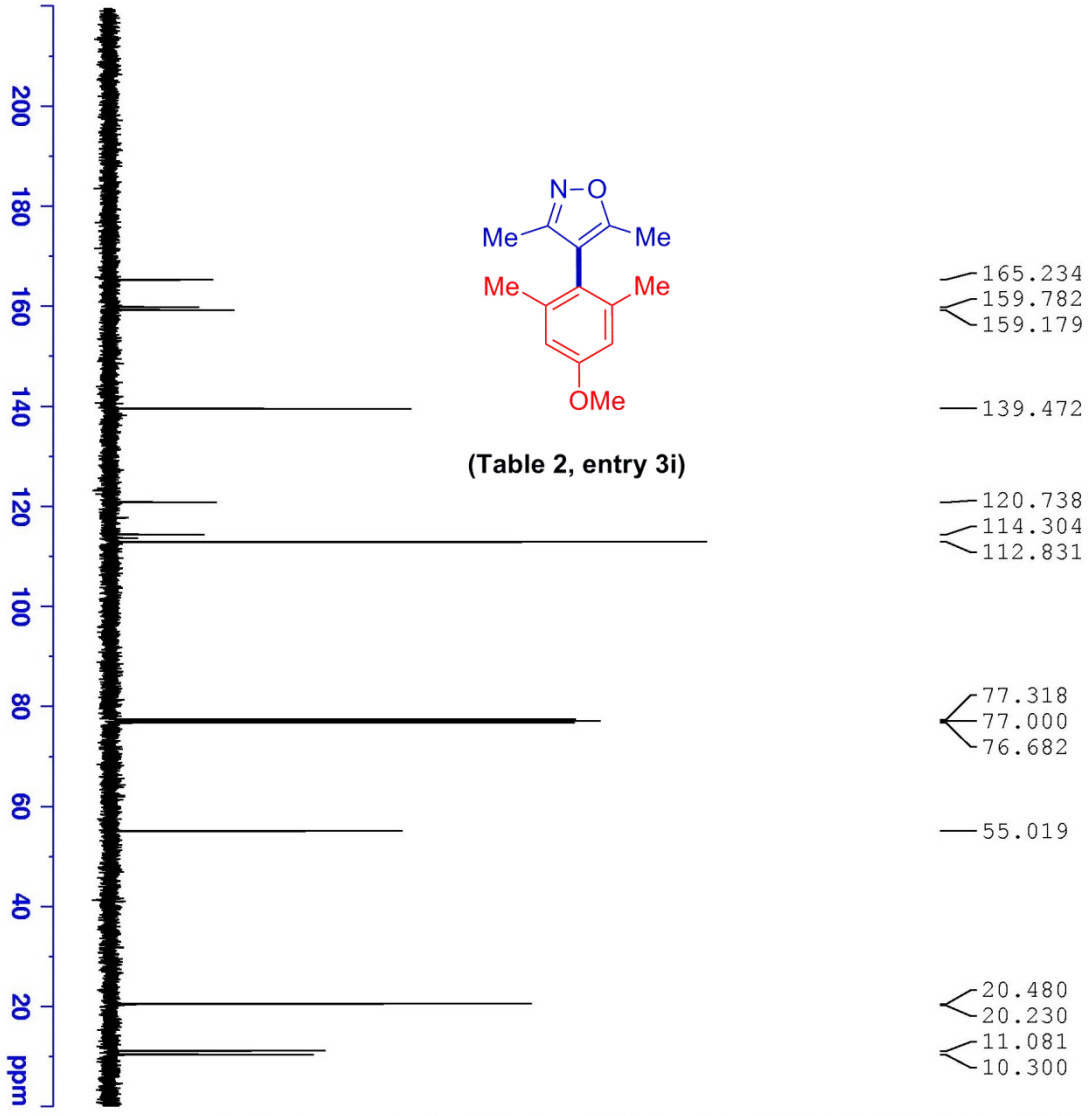

N–O
Me
Me
Me
Me
Me
(Table 2, entry 3i)
7.260
6.949
2.321
2.167
2.019
2.004
2.00
3.08
3.01
3.01
6.01
9
8
7
6
5
4
3
2
1
ppm
NAME STC526A
EXPNO 1
PROCNO 1
Date_ 20151111
Time_ 13.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDC13
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 1.0224116 sec
RG 40.3
DW 62.400 usec
DE 6.50 usec
TE 299.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.70 usec
PL1 0.00 dB
PL1W 11.88122272 W
SFO1 400.1336012 MHz
SI 32768
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



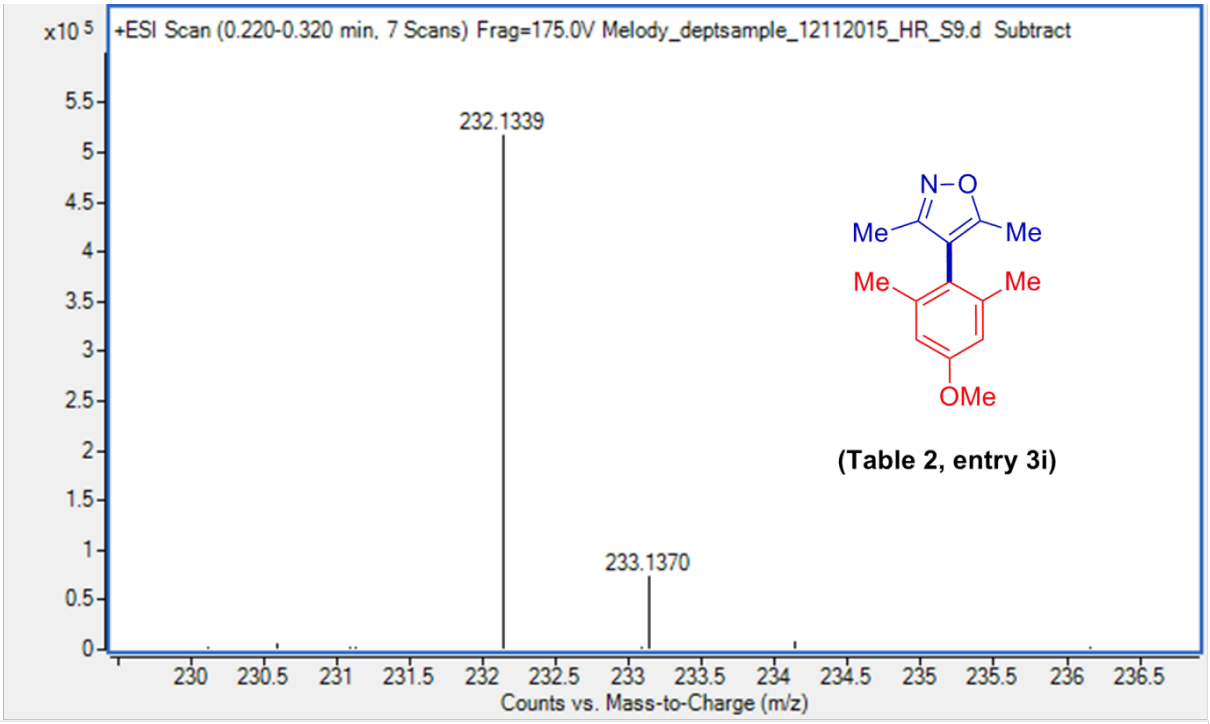

```
NAME                                    STC526A
EXPNO                                         2
PROCNO                                        1
Date_                               20151111
Time                                    13.22
INSTRUM                                  spect
PROBHD                  5 mm PABBO BB-
PULPROG                              zgpg30
TD                                    65536
SOLVENT                              CDCl3
NS                                       35
DS                                        2
SWH                                 24038.461 Hz
FIDRES                                0.366798 Hz
AQ                                    1.3631988 sec
RG                                      228
DW                                   20.800 usec
DE                                    6.50 usec
TE                                   299.6 K
D1                                    2.00000000 sec
D11                                   0.03000000 sec
TD0                                   1

======== CHANNEL f1 ========
NUC1                                    13C
P1                                    9.50 usec
PL1                                  -2.00 dB
PL1W                                58.52175522 W
SFO1                               100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                             waltz16
NUC2                                    1H
PCPD2                                80.00 usec
PL2                                   0.00 dB
PL12                                 15.00 dB
PL13                                 15.00 dB
PL2W                                11.88122272 W
PL12W                                0.37571725 W
PL13W                                0.37571725 W
SFO2                               400.1316005 MHz
SI                                       32768
SF                                 100.6127773 MHz
WDW                                     EM
SSB                                      0
LB                                    1.00 Hz
GB                                       0
PC                                    1.40
```



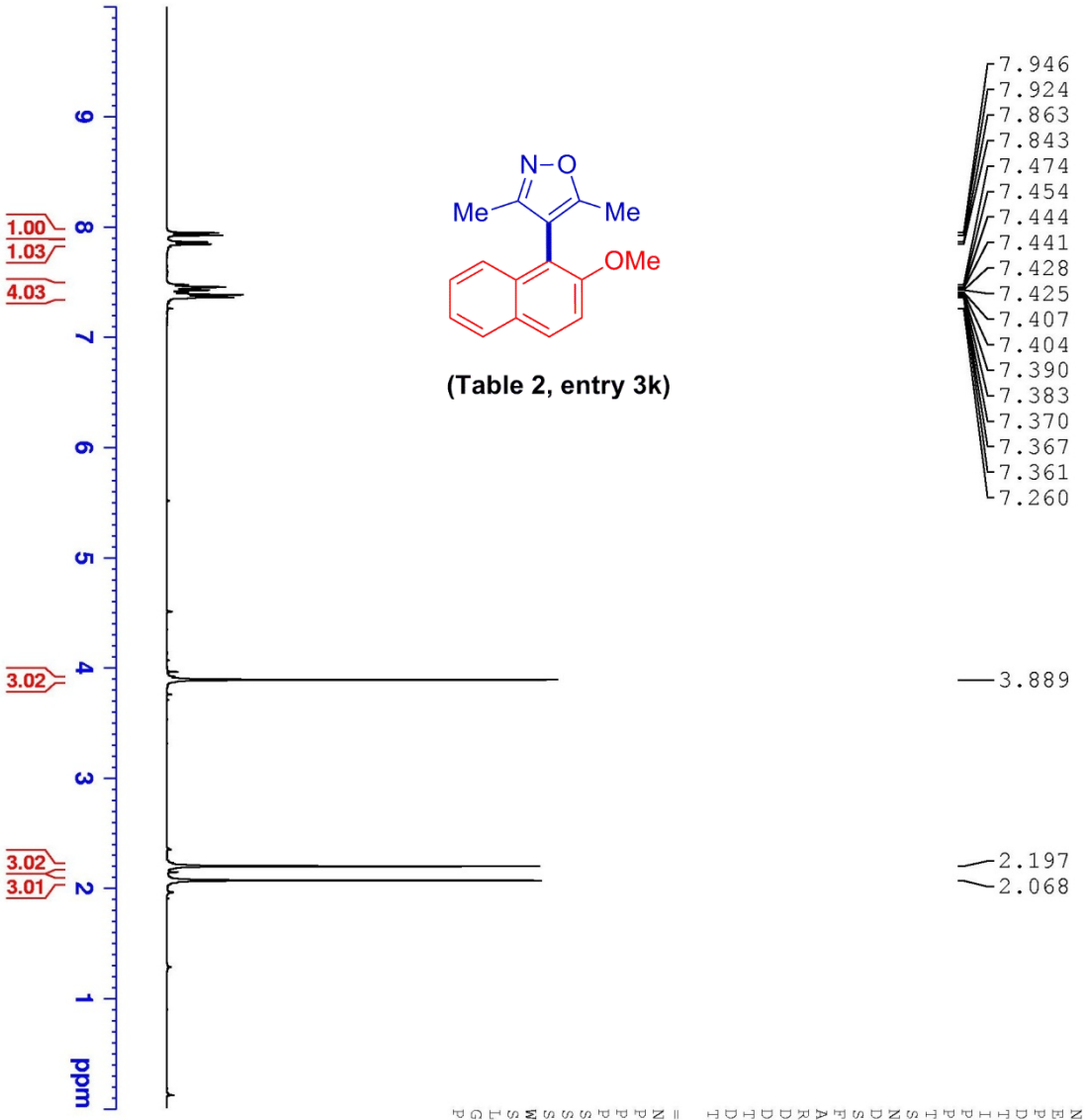

```
NAME                                  STC530B
EXPNO                                       1
PROCNO                                      1
Date_                              20151105
Time_                                  22.14
INSTRUM                                 spect
PROBHD                  5 mm PABBO BB-
PULPROG                                 zg30
TD                                     16384
SOLVENT                                CDC13
NS                                         16
DS                                          2
SWH                                8012.820 Hz
FIDRES                             0.489064 Hz
AQ                                 1.0224116 sec
RG                                         32
DW                                 62.400 usec
DE                                  6.50 usec
TE                                  298.8 K
D1                                 1.00000000 sec
TD0                                         1

======== CHANNEL f1 ========
NUC1                                      1H
P1                                 14.70 usec
PL1                                 0.00 dB
PL1W                           11.88122272 W
SFO1                           400.1336012 MHz
SI                                     32768
SF                             400.1300097 MHz
WDW                                       EM
SSB                                        0
LB                                  0.30 Hz
GB                                         0
PC                                      1.00
```

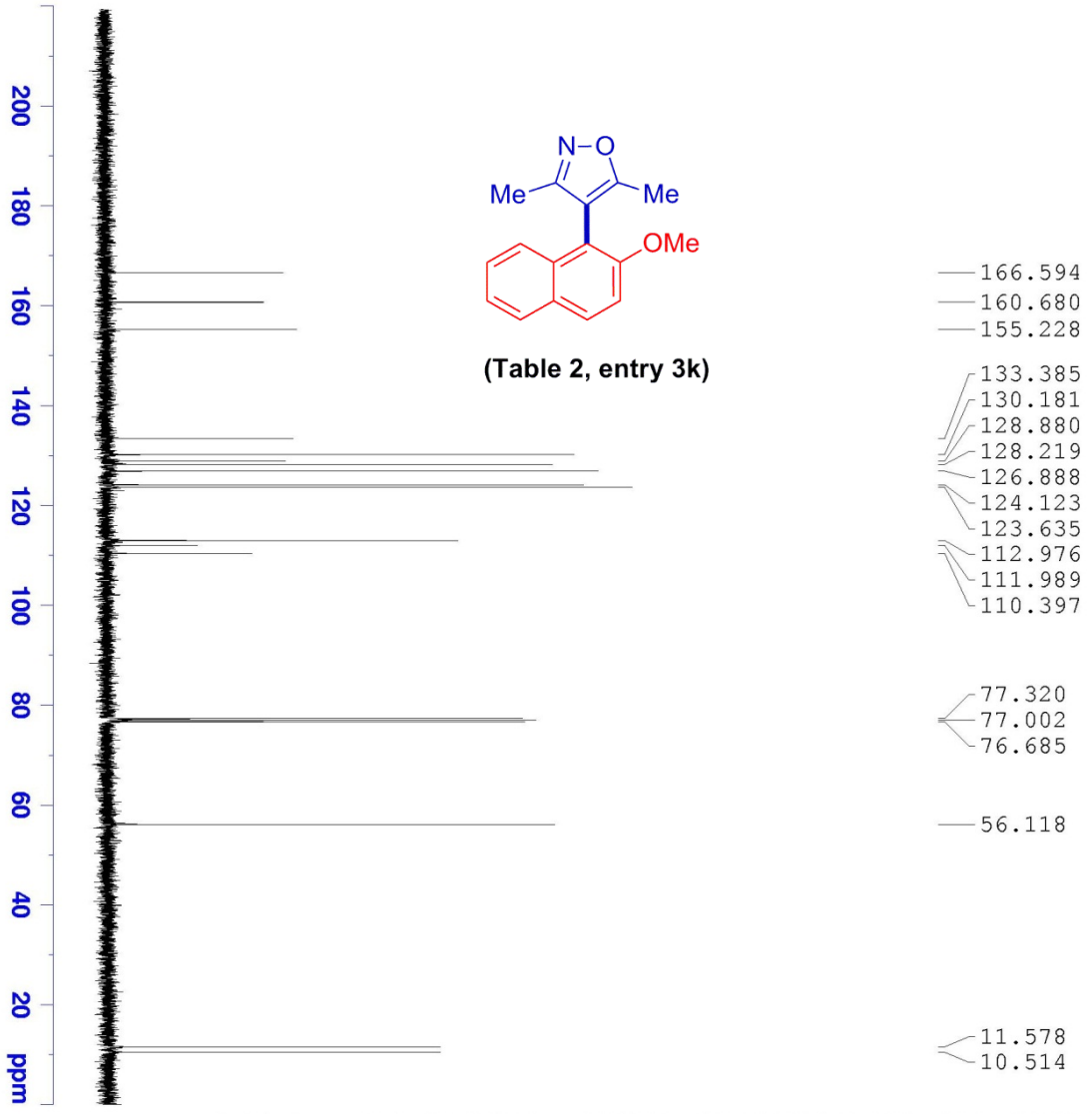



```
NAME                                    STC530B
EXPNO                                         2
PROCNO                                        1
Date_                               20151105
Time                                    22.16
INSTRUM                                  spect
PROBHD                 5 mm PABBO BB-
PULPROG                              zgpg30
TD                                    65536
SOLVENT                              CDCl3
NS                                       32
DS                                        2
SWH                              24038.461 Hz
FIDRES                               0.366798 Hz
AQ                               1.3631988 sec
RG                                      203
DW                                   20.800 usec
DE                                    6.50 usec
TE                                   299.6 K
D1                               2.00000000 sec
D11                              0.03000000 sec
TD0                                       1

======== CHANNEL f1 ========
NUC1                                    13C
P1                                    9.50 usec
PL1                                  -2.00 dB
PL1W                            58.52175522 W
SFO1                           100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                             waltz16
NUC2                                    1H
PCPD2                                80.00 usec
PL2                                   0.00 dB
PL12                                 15.00 dB
PL13                                 15.00 dB
PL2W                            11.88122272 W
PL12W                            0.37571725 W
PL13W                            0.37571725 W
SFO2                           400.1316005 MHz
SI                                    32768
SF                             100.6127751 MHz
WDW                                      EM
SSB                                       0
LB                                     1.00 Hz
GB                                        0
PC                                     1.40
```

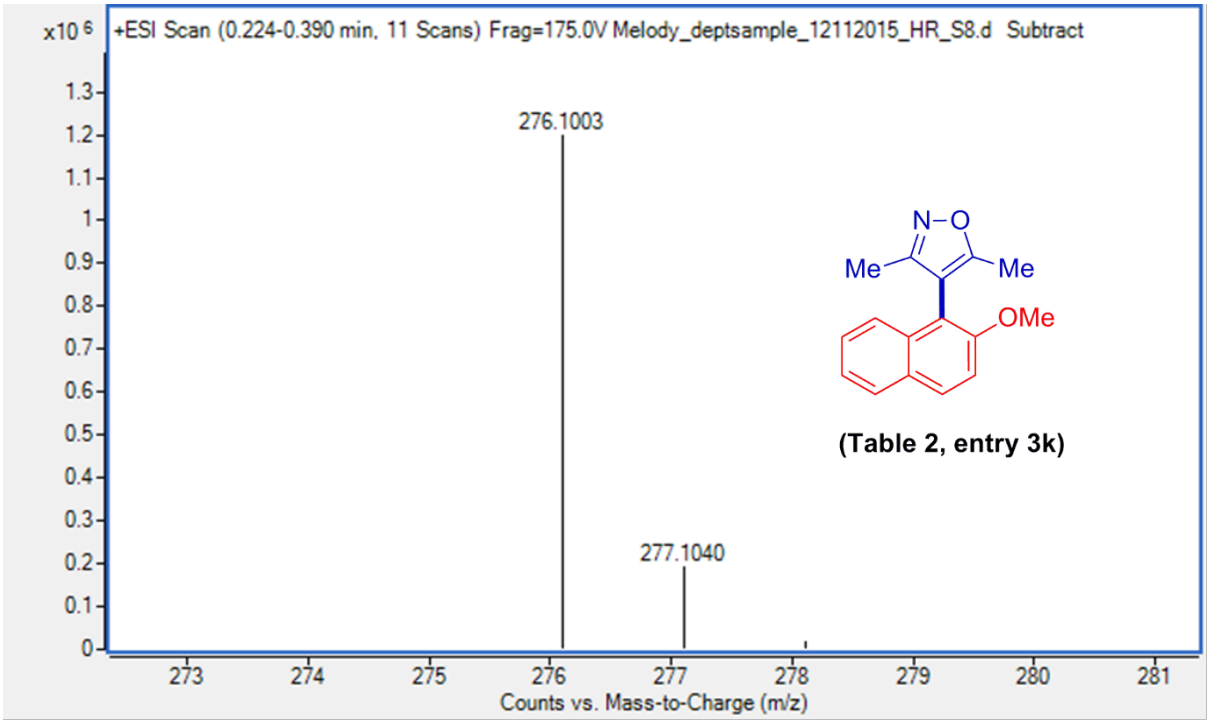


NO.	DATE	FORMULA	THEO	MEASURED	DIFF (mDa)	PPM	OTHER
S9	12112015	C14 H18 N O2	232.1332	232.1339	0.7	3.0	


7.946
7.924
7.863
7.843
7.474
7.454
7.444
7.441
7.428
7.425
7.407
7.404
7.390
7.383
7.370
7.367
7.361
7.260
3.889
2.197
2.068
1.00
1.03
4.03
3.02
3.02
3.01
9
8
7
6
5
4
3
2
1
ppm
N–O
Me
Me
OMe
(Table 2, entry 3k)
NAME STC526B
EXPNO 1
PROCNO 1
Date_ 20151105
Time_ 22.30
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDC13
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 1.0224116 sec
RG 36
DW 62.400 usec
DE 6.50 usec
TE 298.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.70 usec
PL1 0.00 dB
PL1W 11.88122272 W
SFO1 400.1336012 MHz
SI 32768
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

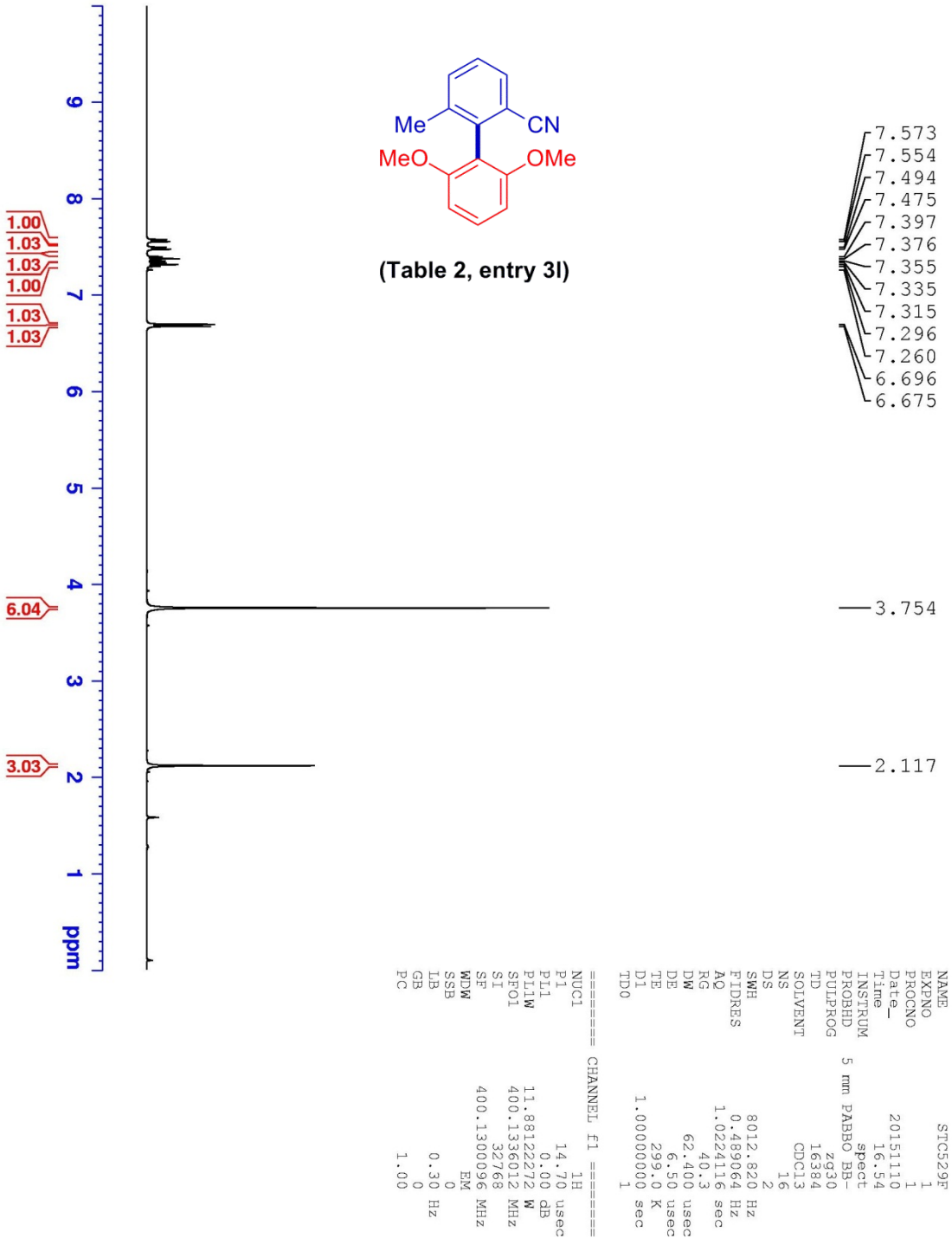


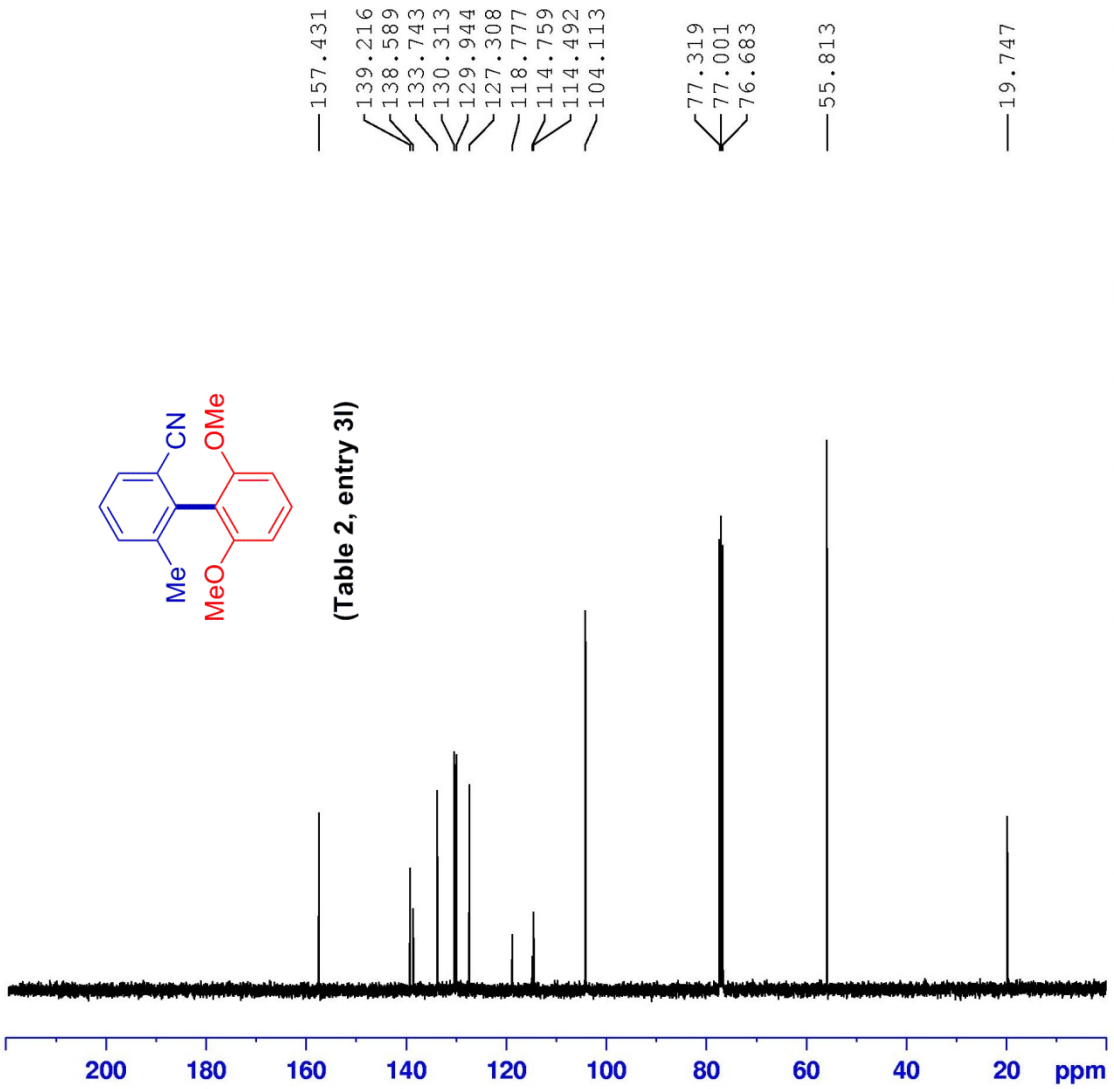

```
NAME                                                    STC526B
EXPNO                                                         2
PROCNO                                                        1
Date_                                              20151105
Time                                                  22.32
INSTRUM                                                 spect
PROBHD                                 5 mm PABBO BB-
PULPROG                                             zgpg30
TD                                                   65536
SOLVENT                                             CDCl3
NS                                                      32
DS                                                       2
SWH                                              24038.461 Hz
FIDRES                                               0.366798 Hz
AQ                                               1.3631988 sec
RG                                                     203
DW                                                  20.800 usec
DE                                                   6.50 usec
TE                                                 299.6 K
D1                                               2.00000000 sec
D11                                              0.03000000 sec
TD0                                                      1

========== CHANNEL f1 ==========
NUC1                                                   13C
P1                                                   9.50 usec
PL1                                                 -2.00 dB
PL1W                                             58.52175522 W
SFO1                                            100.6228298 MHz

========== CHANNEL f2 ==========
CPDPRG2                                             waltz16
NUC2                                                   1H
PCPD2                                               80.00 usec
PL2                                                  0.00 dB
PL12                                                15.00 dB
PL13                                                15.00 dB
PL2W                                             11.88122272 W
PL12W                                             0.37571725 W
PL13W                                             0.37571725 W
SFO2                                            400.1316005 MHz
SI                                                   32768
SF                                              100.6127831 MHz
WDW                                                    EM
SSB                                                     0
LB                                                   1.00 Hz
GB                                                      0
PC                                                   1.40
```



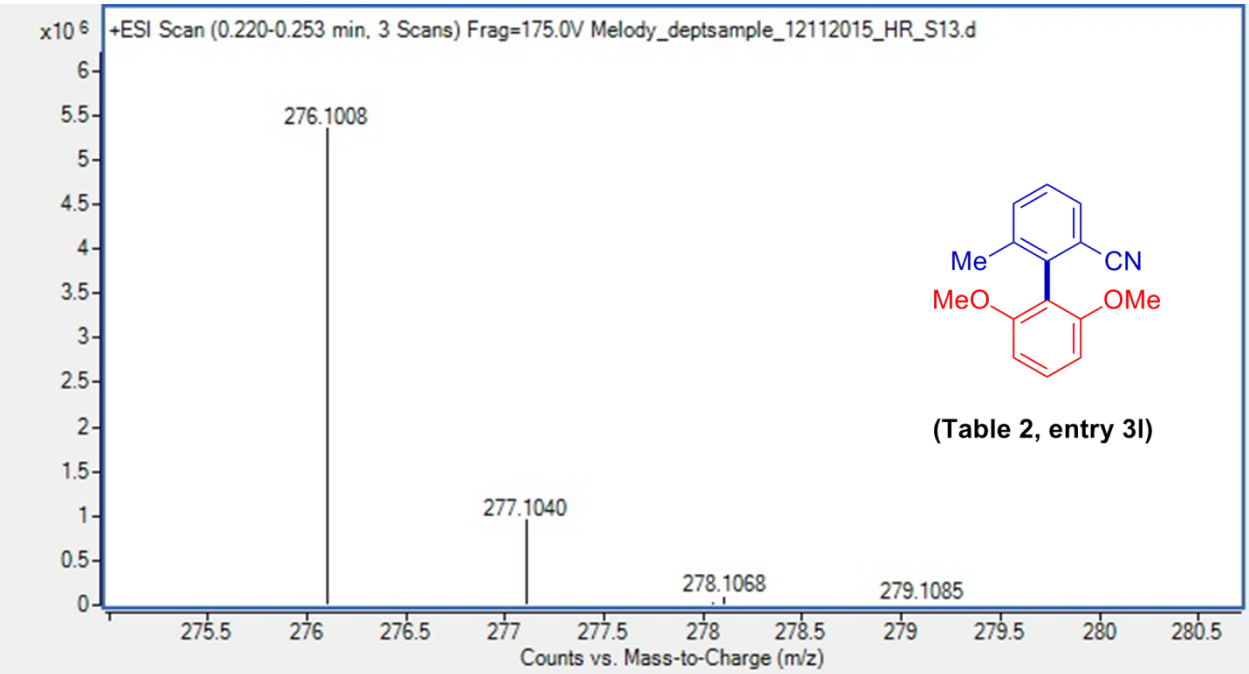
(Table 2, entry 3k)

NO.	DATE	FORMULA	THEO	MEASURED	DIFF (mDa)	PPM	OTHER
S8	12112015	C16H15NO2Na	276.0995	276.1003	0.8	2.9	

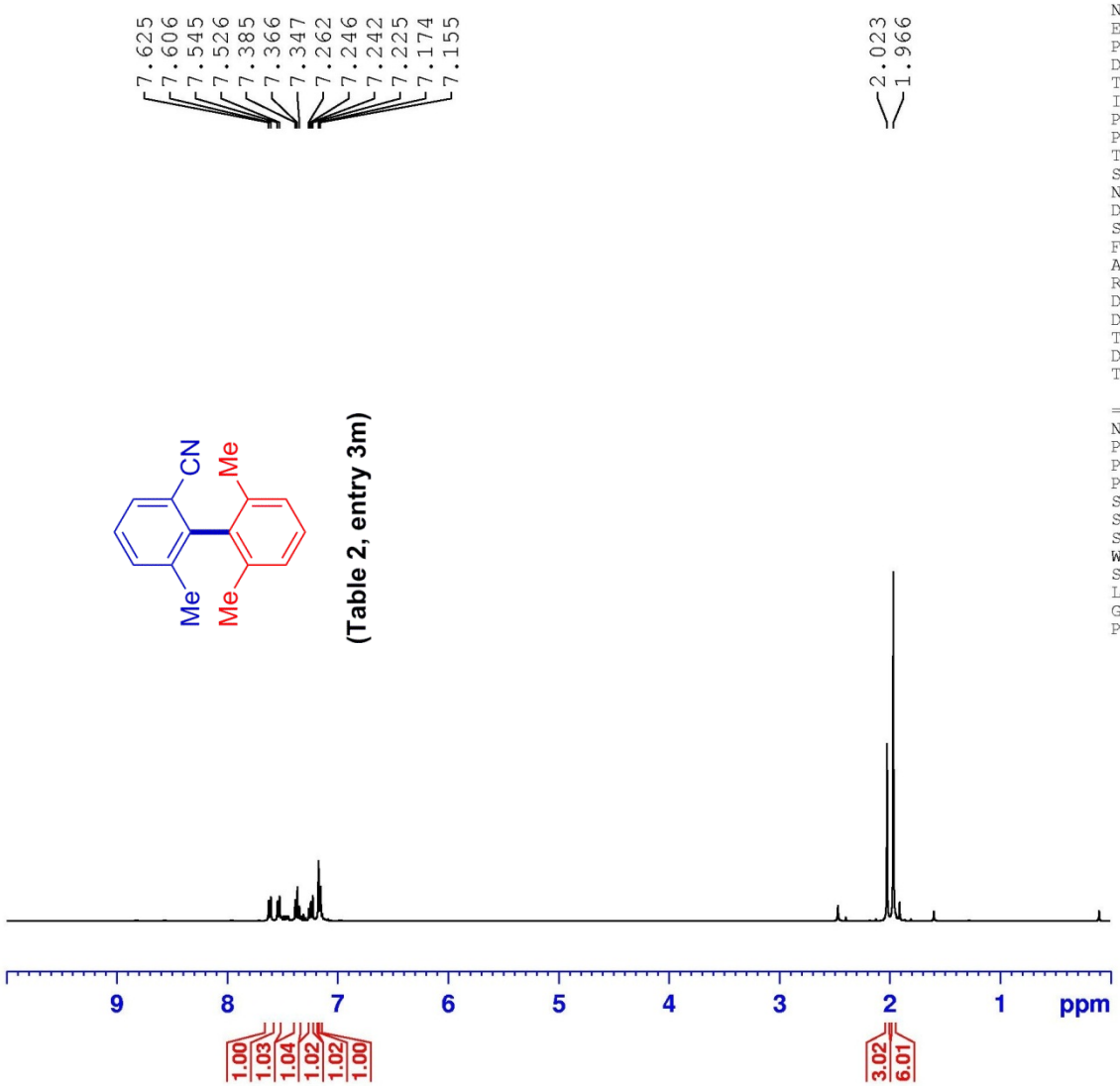

Me
CN
MeO
OMe
(Table 2, entry 3l)
7.573
7.554
7.494
7.475
7.397
7.376
7.355
7.335
7.315
7.296
7.260
6.696
6.675
3.754
2.117
1.00
1.03
1.03
1.00
1.03
1.03
6.04
3.03
9
8
7
6
5
4
3
2
1
ppm
NAME STC529F
EXPNO 1
PROCNO 1
Date_ 20151110
Time 16.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 1.0224116 sec
RG 40.3
DW 62.400 usec
DE 6.50 usec
TE 299.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.70 usec
PL1 0.00 dB
PL1W 11.88122272 W
SFO1 400.1336012 MHz
SI 32768
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



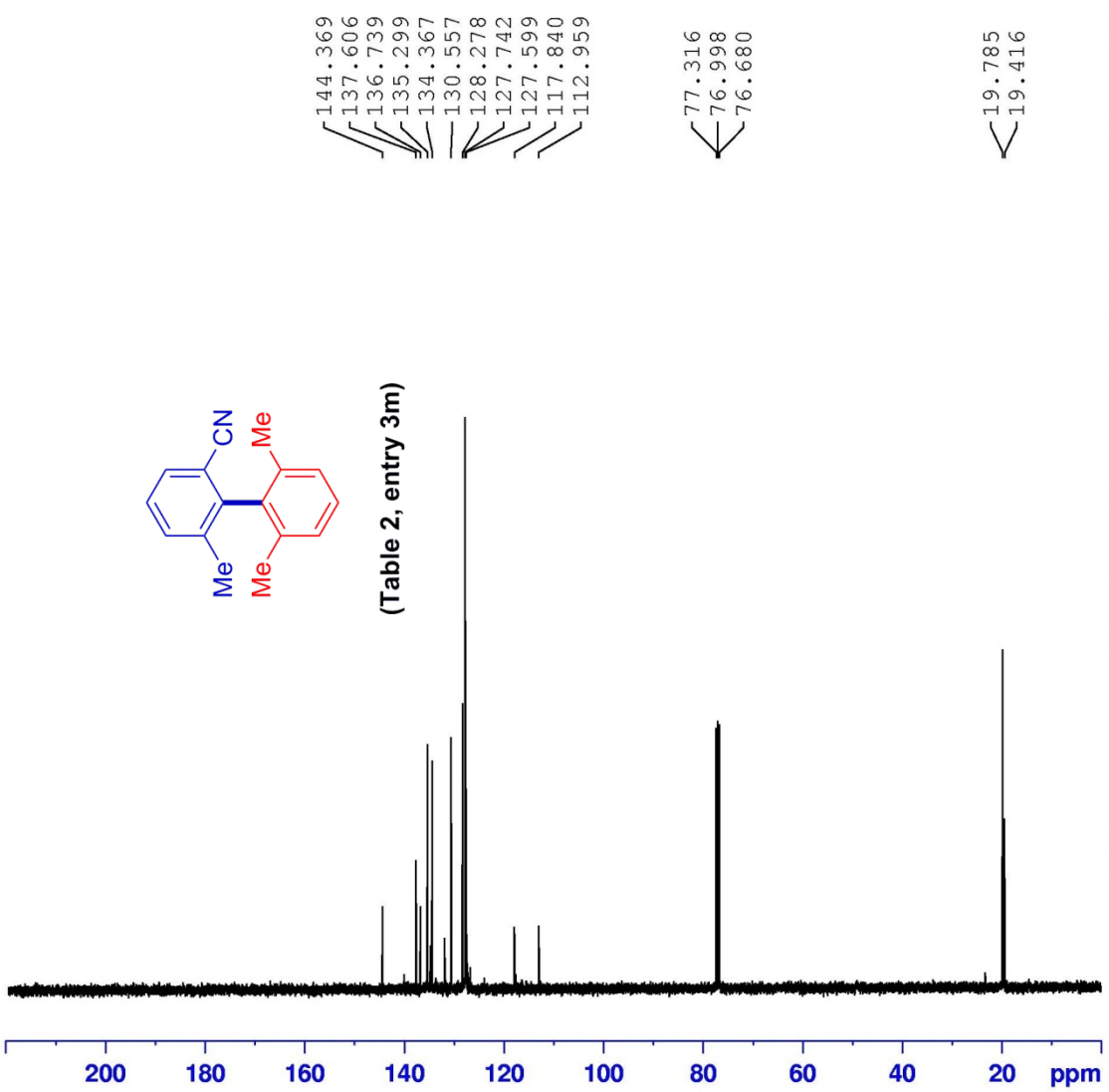

```
NAME              STC529F
EXPNO                   2
PROCNO                  1
Date_            20151110
Time                16.58
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT             CDCl3
NS                     64
DS                      2
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                    181
DW                 20.800 usec
DE                  6.50 usec
TE                 300.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.50 usec
PL1                 -2.00 dB
PL1W         58.52175522 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                  0.00 dB
PL12                15.00 dB
PL13                15.00 dB
PL2W         11.88122272 W
PL12W         0.37571725 W
PL13W         0.37571725 W
SFO2        400.1316005 MHz
SI                 32768
SF          100.6127765 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



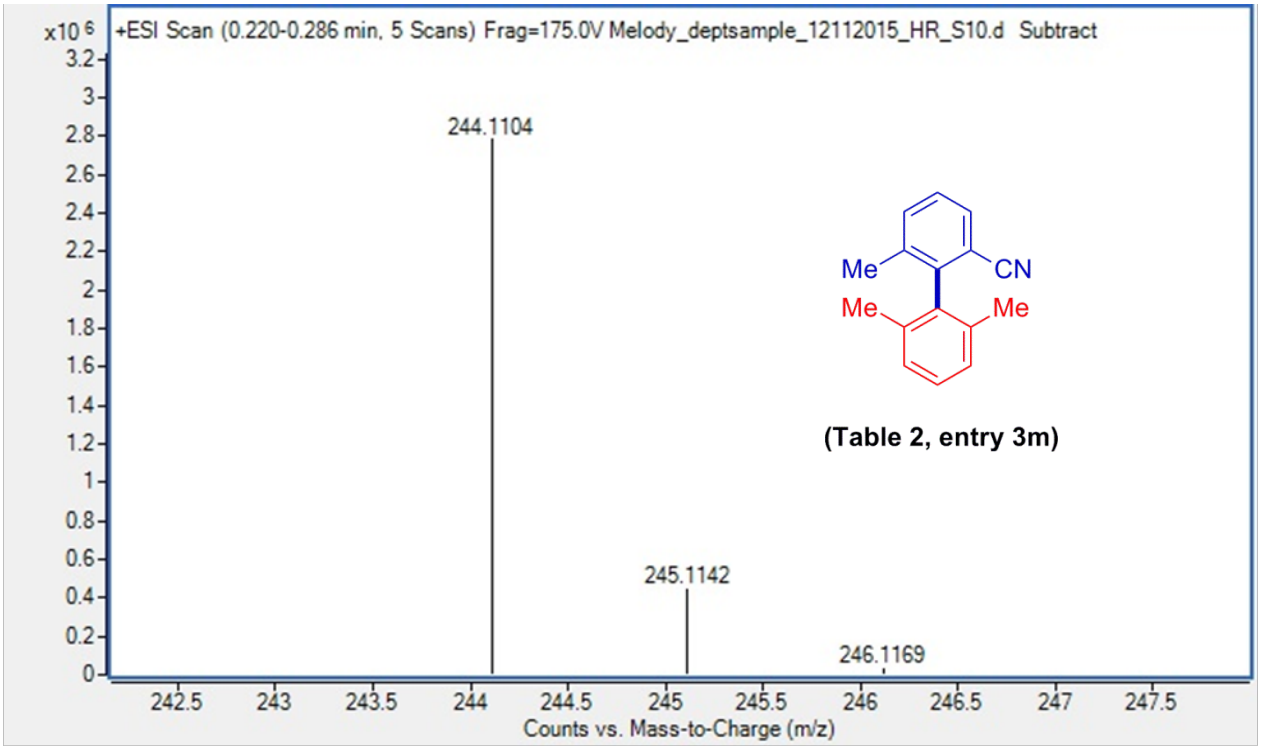
NO.	DATE	FORMULA	THEO	MEASURED	DIFF (mDa)	PPM	OTHER
S13	12112015	C16H15NO2Na	276.0995	276.1008	1.3	4.7	




```
NAME              STC530C
EXPNO                   1
PROCNO                  1
Date_            20151105
Time                22.05
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  16384
SOLVENT             CDC13
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.489064 Hz
AQ               1.0224116 sec
RG                     32
DW                 62.400 usec
DE                  6.50 usec
TE                 298.6 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.70 usec
PL1                 0.00 dB
PL1W          11.88122272 W
SFO1         400.1336012 MHz
SI                  32768
SF           400.1300094 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                  1.00
```

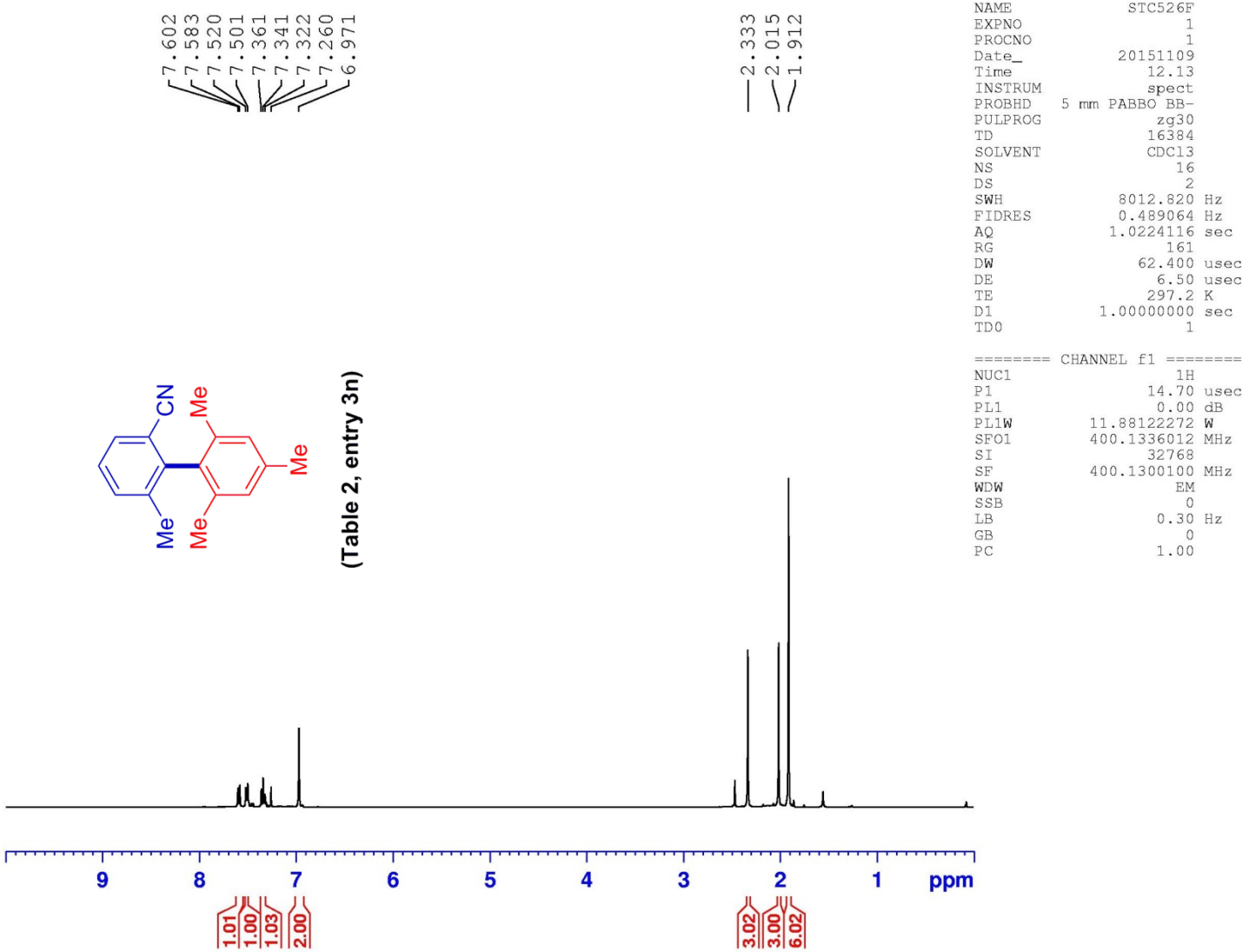


NAME	STC530C
EXPNO	2
PROCNO	1
Date_	20151105
Time	22.08
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	64
DS	2
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	299.4 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.50 usec
PL1	-2.00 dB
PL1W	58.52175522 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	15.00 dB
PL13	15.00 dB
PL2W	11.88122272 W
PL12W	0.37571725 W
PL13W	0.37571725 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127795 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



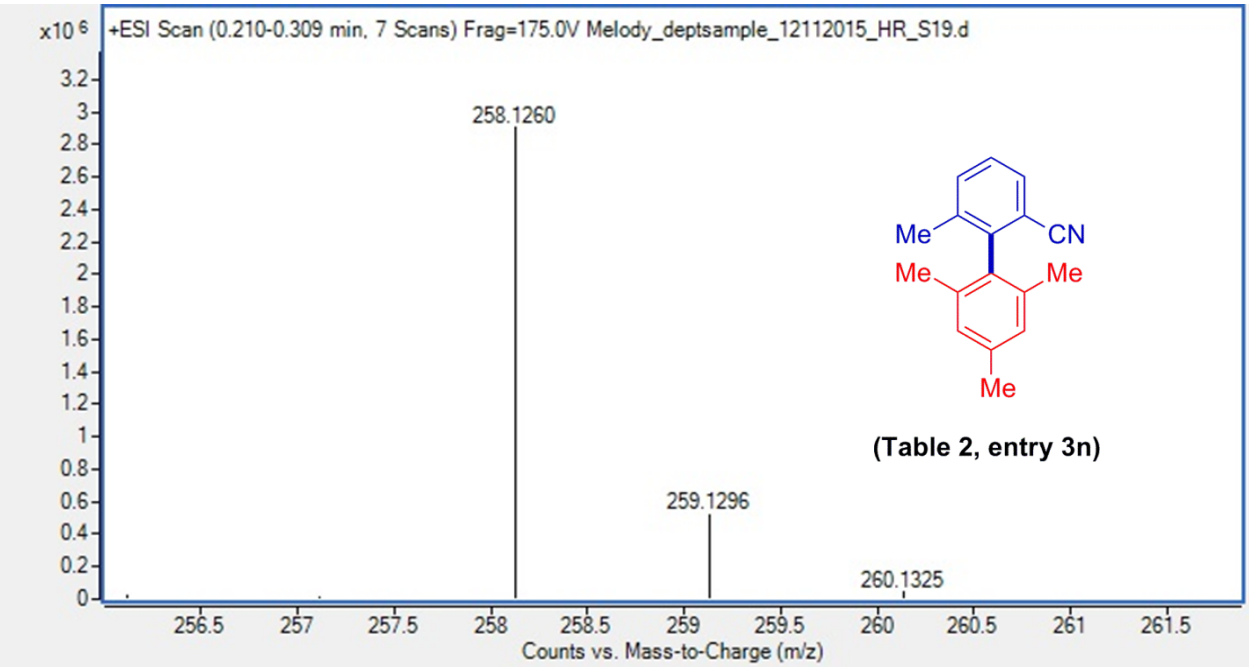
(Table 2, entry 3m)

NO.	DATE	FORMULA	THEO	MEASURED	DIFF (mDa)	PPM	OTHER
S10	12112015	C16H15NNa	244.1097	244.1104	0.7	2.9	

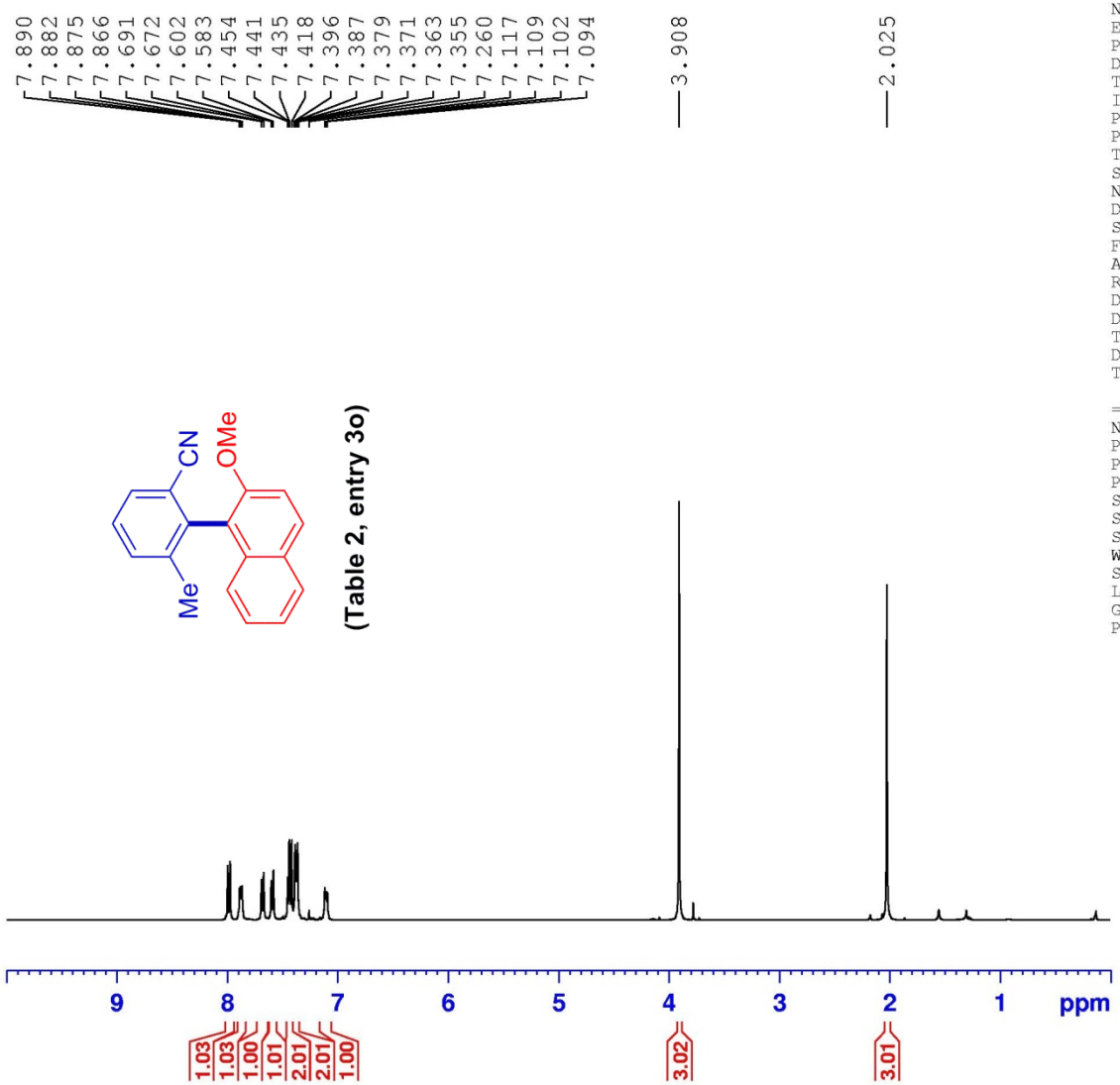



```
NAME              STC526F
EXPNO                   1
PROCNO                  1
Date_            20151109
Time                12.13
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG               zg30
TD                   16384
SOLVENT              CDC13
NS                      16
DS                       2
SWH               8012.820 Hz
FIDRES               0.489064 Hz
AQ                1.0224116 sec
RG                     161
DW                  62.400 usec
DE                   6.50 usec
TE                  297.2 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  14.70 usec
PL1                  0.00 dB
PL1W           11.88122272 W
SFO1          400.1336012 MHz
SI                   32768
SF            400.1300100 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

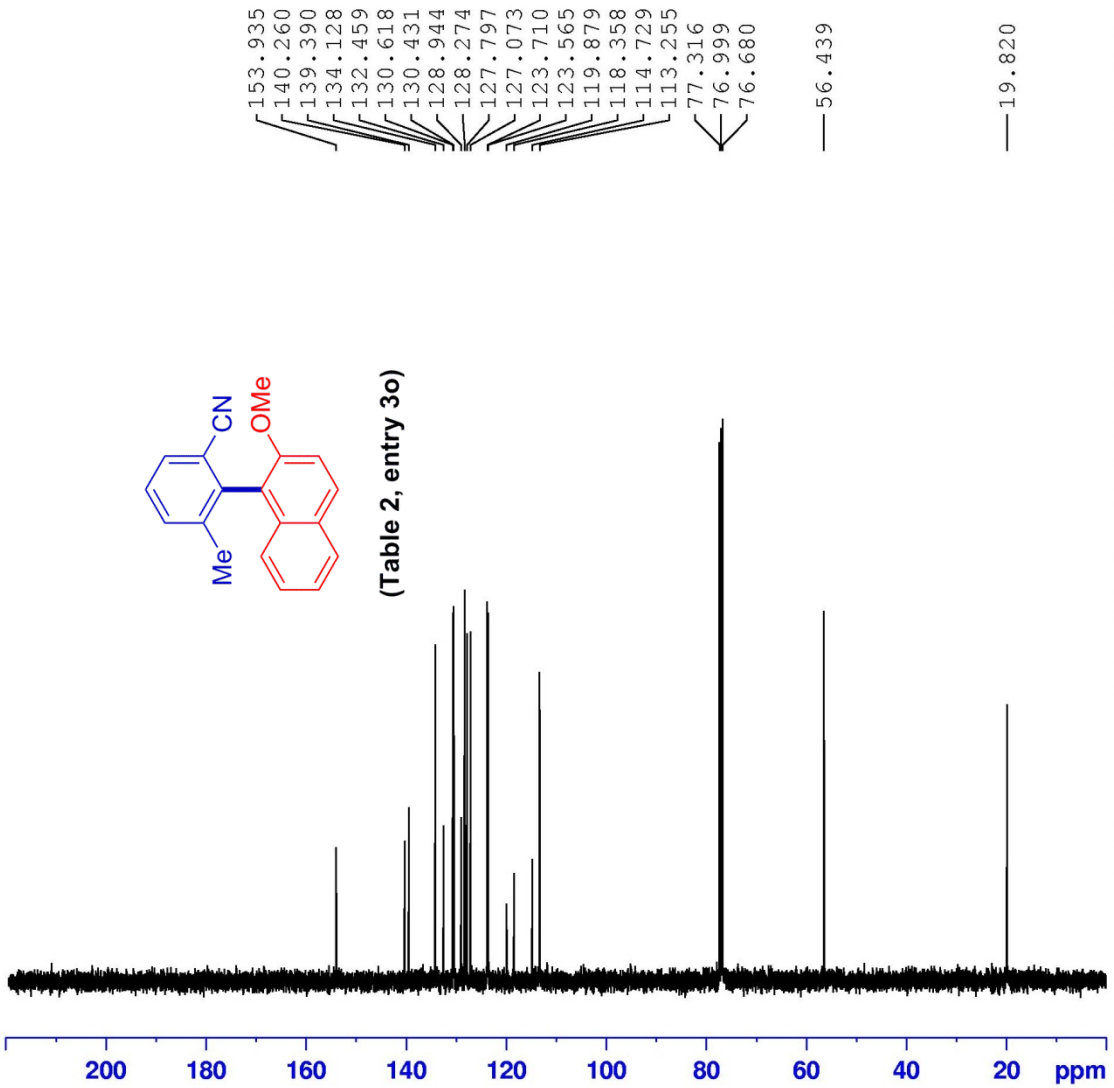
(Table 2, entry 3n)



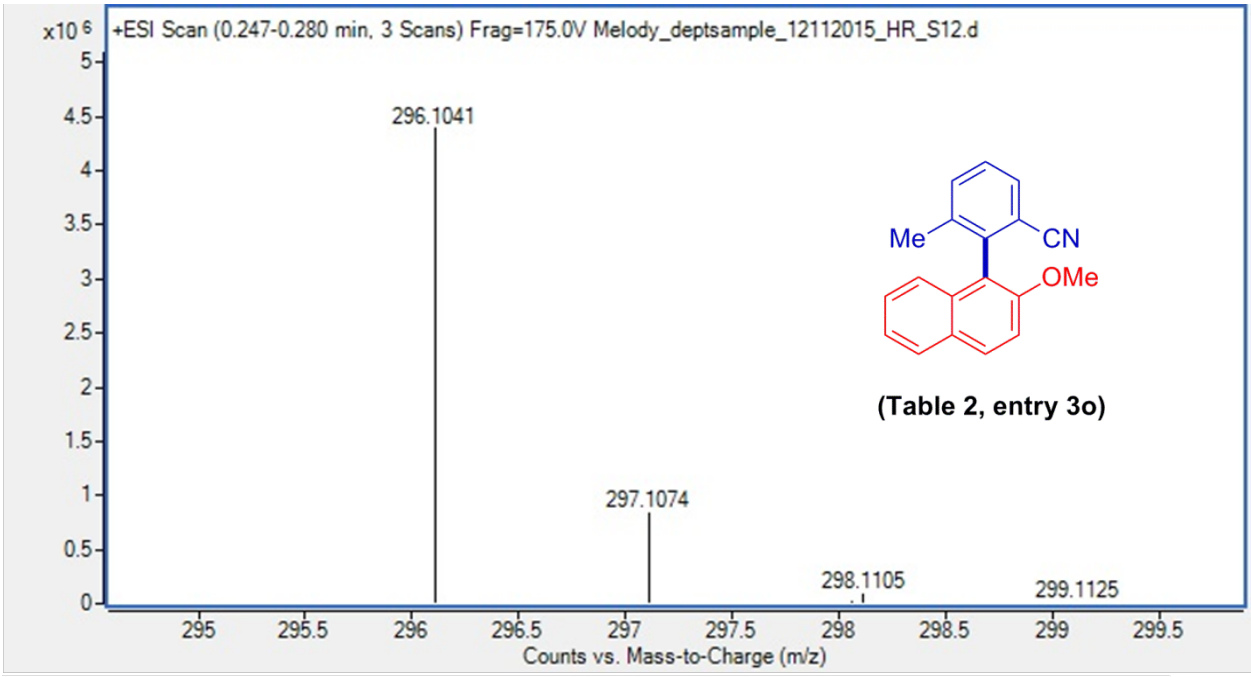
NO.	DATE	FORMULA	THEO	MEASURED	DIFF (mDa)	PPM	OTHER
S19	12112015	C17H17NNa	258.1253	258.126	0.7	2.7	



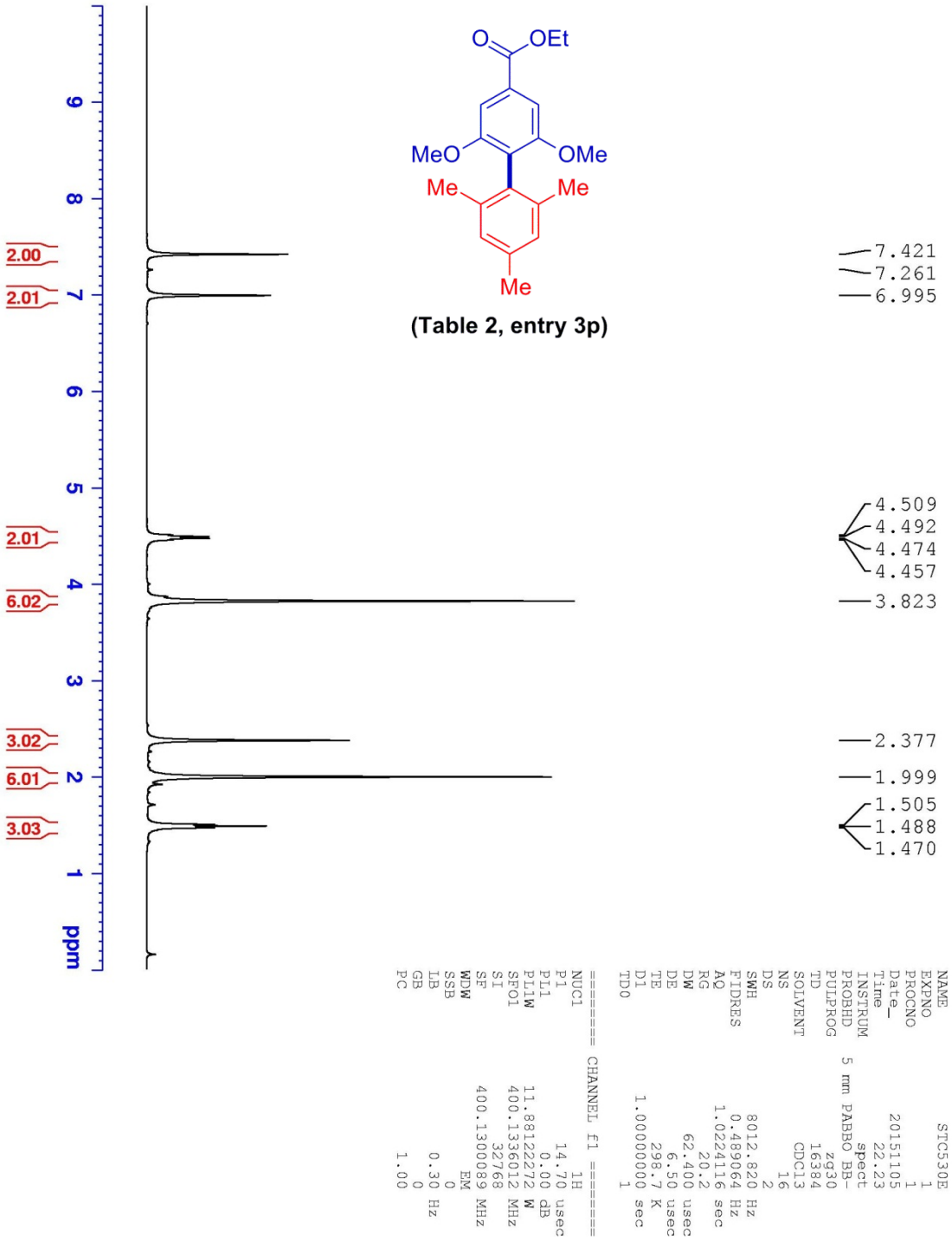
NAME	STC522C
EXPNO	1
PROCNO	1
Date_	20151105
Time	22.42
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	16384
SOLVENT	CDCl3
NS	16
DS	2
SWH	8012.820 Hz
FIDRES	0.489064 Hz
AQ	1.0224116 sec
RG	36
DW	62.400 usec
DE	6.50 usec
TE	298.8 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.70 usec
PL1	0.00 dB
PL1W	11.88122272 W
SFO1	400.1336012 MHz
SI	32768
SF	400.1300098 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

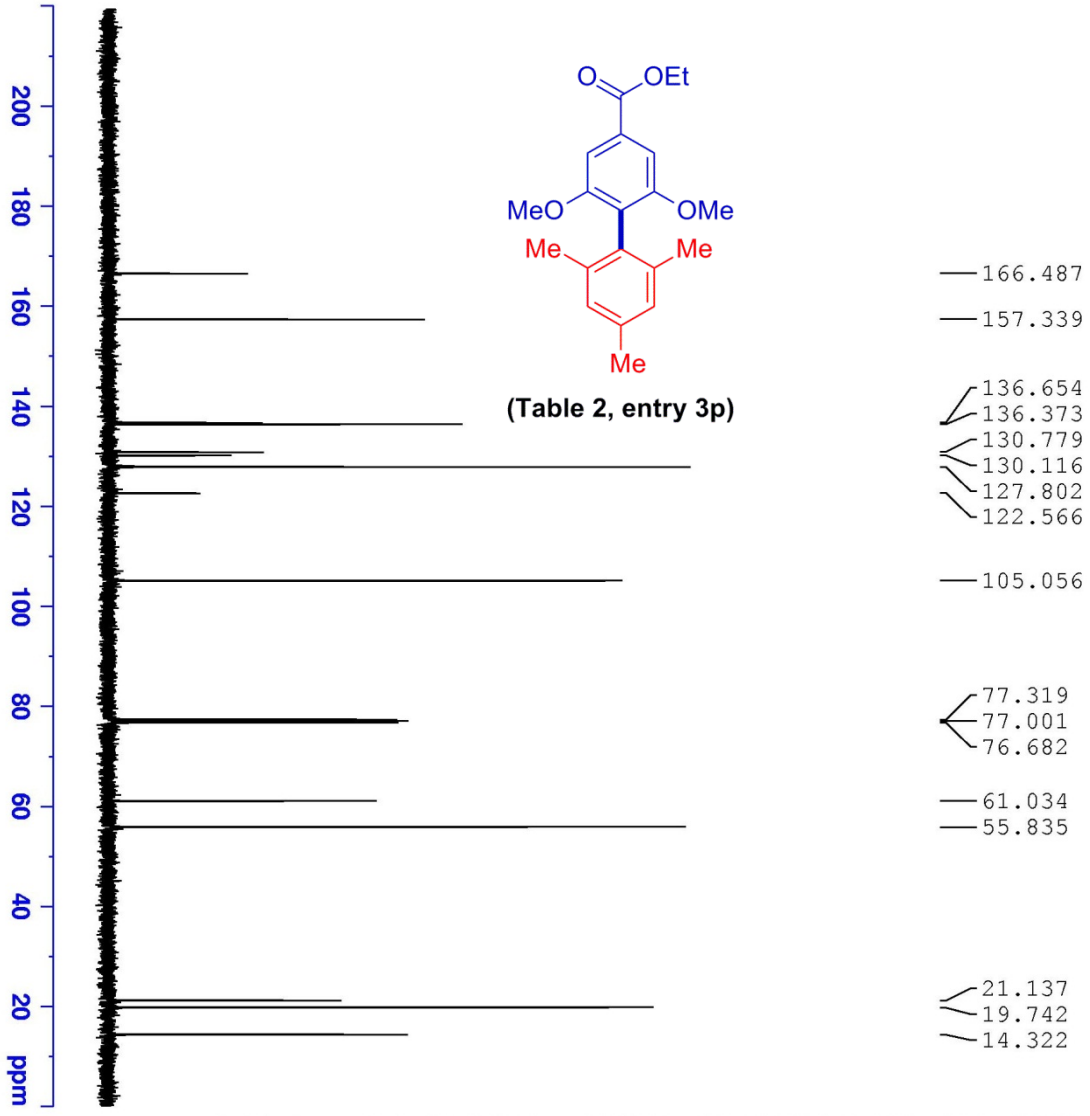


NAME	STC522C
EXPNO	2
PROCNO	1
Date_	20151105
Time	22.44
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	32
DS	2
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	299.6 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.50 usec
PL1	-2.00 dB
PL1W	58.52175522 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	15.00 dB
PL13	15.00 dB
PL2W	11.88122272 W
PL12W	0.37571725 W
PL13W	0.37571725 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127809 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

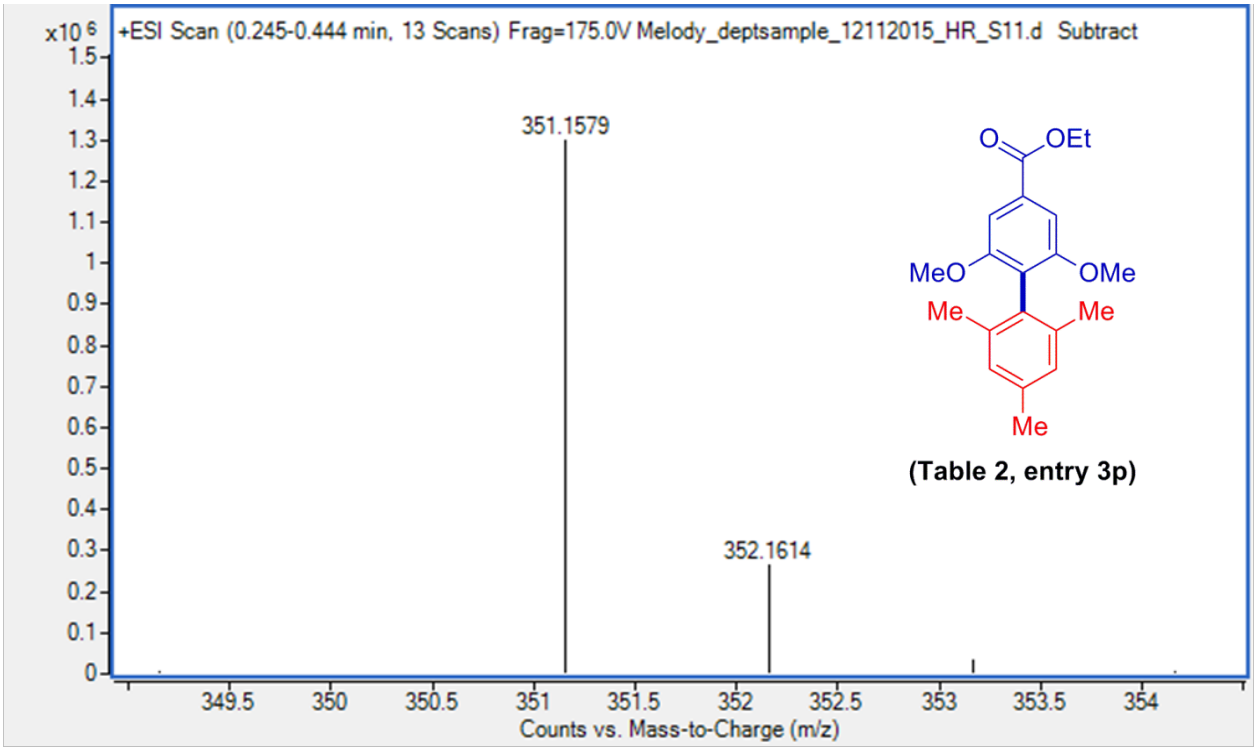


NO.	DATE	FORMULA	THEO	MEASURED	DIFF (mDa)	PPM	OTHER
S12	12112015	C19H15NONa	296.1046	296.1041	-0.5	-1.7	

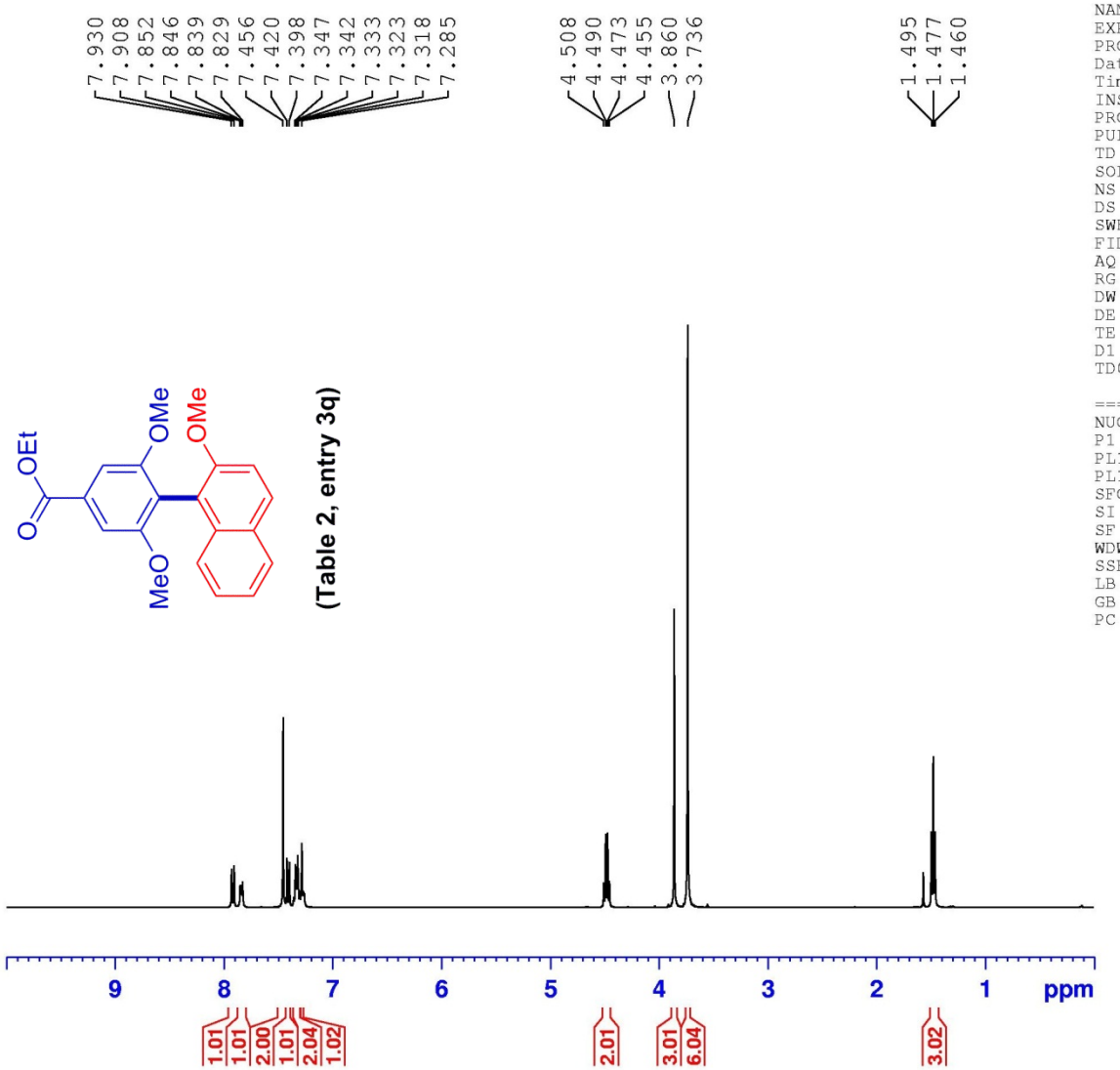

O
OEt
MeO
OMe
Me
Me
Me
(Table 2, entry 3p)
7.421
7.261
6.995
4.509
4.492
4.474
4.457
3.823
2.377
1.999
1.505
1.488
1.470
2.00
2.01
2.01
6.02
3.02
6.01
3.03
9
8
7
6
5
4
3
2
1
ppm
NAME STC530E
EXPNO 1
PROCNO 1
Date_ 20151105
Time_ 22.23
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDC13
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 1.0224116 sec
RG 20.2
DW 62.400 usec
DE 6.50 usec
TE 298.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.70 usec
PL1 0.00 dB
PL1W 11.88122272 W
SFO1 400.1336012 MHz
SI 32768
SF 400.1300089 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00




```
NAME                                    STC530E
EXPNO                                         2
PROCNO                                        1
Date_                               20151105
Time                                    22.26
INSTRUM                                  spect
PROBHD                 5 mm PABBO BB-
PULPROG                              zgpg30
TD                                    65536
SOLVENT                               CDCl3
NS                                       32
DS                                        2
SWH                                24038.461 Hz
FIDRES                                0.366798 Hz
AQ                                    1.3631988 sec
RG                                      203
DW                                   20.800 usec
DE                                    6.50 usec
TE                                   299.6 K
D1                                    2.00000000 sec
D11                                   0.03000000 sec
TD0                                   1

======== CHANNEL f1 ========
NUC1                                    13C
P1                                    9.50 usec
PL1                                  -2.00 dB
PL1W                                 58.52175522 W
SFO1                                100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                             waltz16
NUC2                                    1H
PCPD2                                80.00 usec
PL2                                   0.00 dB
PL12                                 15.00 dB
PL13                                 15.00 dB
PL2W                                 11.88122272 W
PL12W                                 0.37571725 W
PL13W                                 0.37571725 W
SFO2                                400.1316005 MHz
SI                                       32768
SF                                  100.6127861 MHz
WDW                                      EM
SSB                                       0
LB                                    1.00 Hz
GB                                        0
PC                                    1.40
```



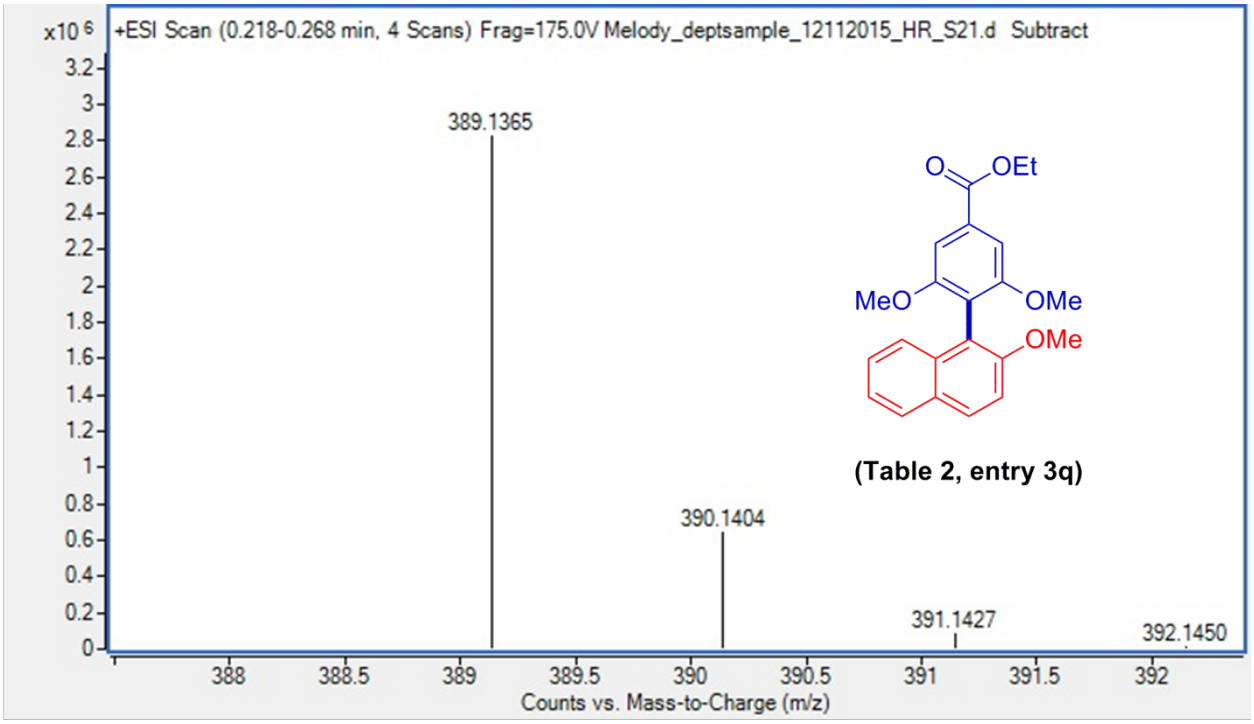
NO.	DATE	FORMULA	THEO	MEASURED	DIFF (mDa)	PPM	OTHER
S11	12112015	C20H24O4Na	351.1567	351.1579	1.2	3.4	



NAME	STC532A
EXPNO	3
PROCNO	1
Date_	20151110
Time	16.51
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	16384
SOLVENT	CDCl3
NS	16
DS	2
SWH	8012.820 Hz
FIDRES	0.489064 Hz
AQ	1.0224116 sec
RG	114
DW	62.400 usec
DE	6.50 usec
TE	299.1 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.70 usec
PL1	0.00 dB
PL1W	11.88122272 W
SFO1	400.1336012 MHz
SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

(Table 2, entry 3q)


```
NAME                                                STC532A
EXPNO                                                     2
PROCNO                                                    1
Date_                                              20151109
Time                                                  12.26
INSTRUM                                               spect
PROBHD                                 5 mm PABBO BB-
PULPROG                                            zgpg30
TD                                                  65536
SOLVENT                                             CDCl3
NS                                                     32
DS                                                      2
SWH                                            24038.461 Hz
FIDRES                                          0.366798 Hz
AQ                                              1.3631988 sec
RG                                                    181
DW                                             20.800 usec
DE                                              6.50 usec
TE                                              297.8 K
D1                                              2.00000000 sec
D11                                             0.03000000 sec
TD0                                                     1

======== CHANNEL f1 ========
NUC1                                                  13C
P1                                               9.50 usec
PL1                                             -2.00 dB
PL1W                                        58.52175522 W
SFO1                                       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                                           waltz16
NUC2                                                  1H
PCPD2                                          80.00 usec
PL2                                             0.00 dB
PL12                                           15.00 dB
PL13                                           15.00 dB
PL2W                                        11.88122272 W
PL12W                                        0.37571725 W
PL13W                                        0.37571725 W
SFO2                                       400.1316005 MHz
SI                                                 32768
SF                                         100.6127868 MHz
WDW                                                   EM
SSB                                                    0
LB                                              1.00 Hz
GB                                                     0
PC                                              1.40
```



NO.	DATE	FORMULA	THEO	MEASURED	DIFF (mDa)	PPM	OTHER
S21	12112015	C22H22O5Na	389.1359	389.1365	0.6	1.5	