Supporting Information

Synthesis of Tetrasubstituted Furans through One-pot Formal [3+2] Cycloaddition Utilizing [1,2]-Phospha-Brook Rearrangement

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General Information

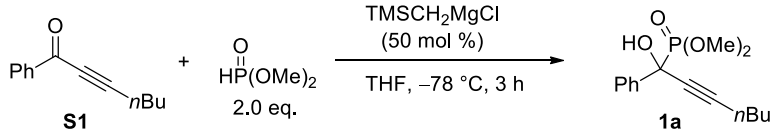
Unless otherwise noted, the reactions were carried out with dried glassware under argon atmosphere. ^{1}H NMR spectra were recorded on a JEOL JNM-ECA600 (600 MHz) spectrometer. Chemical shifts are reported in ppm from the solvent resonance or tetramethylsilane (TMS) as the internal standard ($CDCl_3$: 7.26 ppm, TMS: 0.00 ppm). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, sep = septet, m = multiplet), coupling constants (Hz) and integration. ^{13}C NMR spectra were recorded on a JEOL JNM-ECA600 (150 MHz) spectrometer with complete proton decoupling. Chemical shifts are reported in ppm from the solvent resonance as the internal standard ($CDCl_3$: 77.0 ppm; CD_3OD: 49.0 ppm). ^{31}P NMR spectra were recorded on a JEOL JNM-ECA600 (243 MHz) spectrometer with complete proton decoupling. Chemical shifts are reported in ppm with 85% H_3PO_4 solution as an external standard (0.0 ppm in $CDCl_3$). Analytical thin layer chromatography (TLC) was performed on Merck precoated TLC plates (silica gel 60 GF_{254}, 0.25 mm). Flash column chromatography was performed on silica gel 60N (spherical, neutral, 40-50 μm; Kanto Chemical Co., Inc.). High resolution mass spectra analysis was performed on a Bruker Daltonics solariX 9.4T FT-ICR-MS spectrometer at the Research and Analytical Center for Giant Molecules, Graduate School of Science, Tohoku University.

Materials: Unless otherwise noted, materials were purchased from Wako Pure Chemical Industries, Ltd., Tokyo Chemical Industry Co., LTD., Aldrich Inc., and other commercial suppliers and were used without purification. Dichloromethane, tetrahydrofuran and toluene were supplied from Kanto Chemical Co., Inc. as “Dehydrated solvent system”. Other solvents were purchased from commercial suppliers as dehydrated solvents, and used under argon atmosphere.

Experimental Procedure

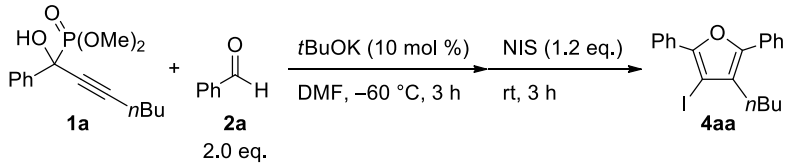
Procedure for Preparation of Propargyl Alcohols 1.

Propargyl alcohols **1** were prepared by the 1,2-addition of a dimethyl phosphite to alkynyl ketones by using a substoichiometric amount of (trimethylsilyl)methylmagnesium chloride as a Brønsted base.[S1] Synthesis of **1a** is representative.



A mixture of **S1** (1.9 g, 10 mmol) and dimethyl phosphite (1.8 mL, 20 mmol) in THF (20 mL) was stirred at –78 °C for 20 min. Then a solution of (trimethylsilyl)methylmagnesium chloride (1.0 M in Et_2O, 5.0 mL, 5.0 mmol) was added dropwise to the mixture, and the resulting mixture was stirred at that temperature for 3 h. The reaction was quenched with sat. aq. NH_4Cl, and the product was extracted with AcOEt. The combined organic layer was washed with brine, dried over Na_2SO_4 and evaporated. The crude solid was purified by reprecipitation from methanol and hexane to afford **1a** (2.4 g, 8.1 mmol, 81%) as a white solid.

General Procedure for One-pot Formal [3+2] Cycloaddition of Propargyl Alcohol 1 with Aldehyde 2.



The reaction of **1a** with **2a** is representative.

A solution of **1a** (74 mg, 0.25 mmol) and benzaldehyde (**2a**) (51 μL, 0.50 mmol) in DMF (1.0 mL) was stirred at –60 °C for 20 min. Then, a solution of *t*BuOK in THF (1.0 M, 25 μL, 0.025 mmol) was added to the solution at –60 °C, and the resulting mixture was stirred at that temperature for 3 h. NIS (68 mg, 0.30 mmol) was added, and the mixture was allowed to warm to room temperature. After stirred for 3 h, the reaction was quenched with sat. aq. $Na_2S_2O_3$, and the product was extracted with AcOEt. The combined organic layer was washed with brine, dried over Na_2SO_4 and evaporated. The crude mixture was purified by silica-gel column chromatography (hexane/AcOEt = 30:1) to provide **4aa** (68 mg, 0.17 mmol, 67%) as a pale yellow solid.

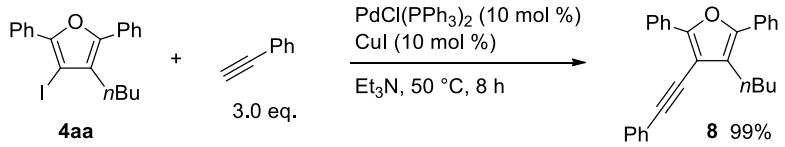
Gram-Scale Synthesis.

A solution of **1a** (1.2 g, 4.0 mmol) and benzaldehyde (**2a**) (0.82 mL, 8.0 mmol) in DMF (16 mL) was stirred at –60 °C for 20 min. Then, a solution of *t*BuOK in THF (1.0 M, 0.40 mL, 0.40 mmol) was added dropwise to the solution at –60 °C, and the resulting mixture was stirred at that temperature for 3 h. NIS (1.1 g, 4.8 mmol) was added, and the mixture was allowed to warm to room temperature. After stirred for 3 h, the reaction was quenched with sat. aq. $Na_2S_2O_3$, and the product was extracted with AcOEt. The combined organic layer was washed with brine, dried over Na_2SO_4 and evaporated. The crude mixture was purified by silica-gel column chromatography

(hexane/AcOEt = 30:1) to provide **4aa** (1.1 g, 2.6 mmol, 66%) as a pale yellow solid.

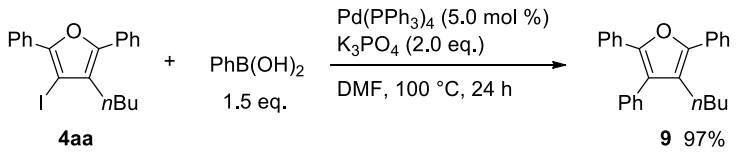
Procedure for Pd-Catalyzed Reactions (Scheme 5a).

Sonogashira Coupling Reaction (Condition A)



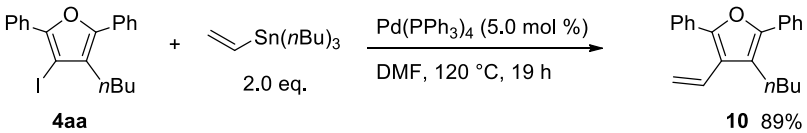
To a solution of **4aa** (40 mg, 0.10 mmol) in trimethylamine (1.0 mL) were sequentially added $PdCl_2(PPh_3)_2$ (7.0 mg, 10 μmol), CuI (1.9 mg, 1 μmol) and phenylacetylene (33 μL, 0.30 mmol). The resulting mixture then was stirred at 50 °C with oil bath for 8 h. The reaction was quenched with sat. aq. NH_4Cl, and the product was extracted with AcOEt. The combined organic layer was washed with brine, dried over Na_2SO_4 and evaporated. The crude mixture was purified by silica-gel column chromatography (hexane/AcOEt = 50:1) to furnish **8** (37 mg, 0.099 mmol, 99%) as an orange solid.

Suzuki-Miyaura Coupling Reaction (Condition B)



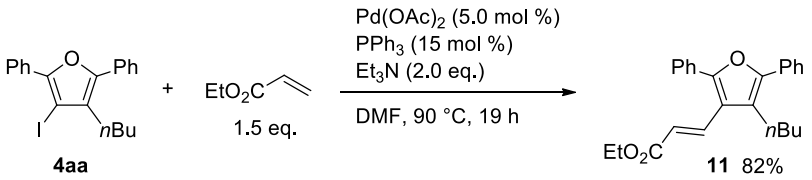
$Pd(PPh_3)_4$ (5.8 mg, 5.0 μmol), K_3PO_4 (43 mg, 0.20 mmol) and $PhB(OH)_2$ (18 mg, 0.15 mmol) were added to a solution of **4aa** (40 mg, 0.10 mmol) in DMF (1.0 mL), and the resulting mixture was heated at 100 °C with oil bath for 24 h. After cooled to room temperature, the reaction was quenched with sat. aq. NH_4Cl, and the product was extracted with AcOEt. The combined organic layer was washed with brine, dried over Na_2SO_4 and evaporated. The residue was purified by silica-gel column chromatography (hexane/AcOEt = 50:1) to provide **9** (34 mg, 0.097 mmol, 97%) as a pale yellow solid.

Migita-Kosugi-Stille Coupling Reaction (Condition C)



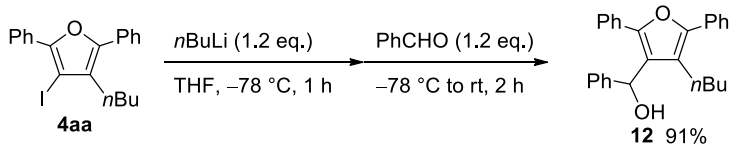
To a mixture of **4aa** (40 mg, 0.10 mmol) and $Pd(PPh_3)_4$ (5.8 mg, 5.0 μmol) in DMF (1.0 mL) was added tributyl(vinyl)tin (58 μL, 0.20 mmol). The resulting mixture was then heated at 120 °C with oil bath for 19 h. After cooled to room temperature, the reaction was quenched with H_2O, and the product was extracted with AcOEt. The combined organic layer was washed with brine, dried over Na_2SO_4 and evaporated. The residue was purified by silica-gel column chromatography (hexane/AcOEt = 50:1) to provide **10** (27 mg, 0.089 mmol, 89%) as a pale yellow oil.

Mizoroki-Heck Reaction (Condition D)



To a solution of **4aa** (40 mg, 0.10 mmol), $Pd(OAc)_2$ (1.1 mg, 5.0 μmol), and triphenylphosphine (3.9 mg, 0.015 mmol) in DMF (2.0 mL) was added triethylamine (28 μL, 0.20 mmol) and ethyl acrylate (16 μL, 0.15 mmol). The resulting mixture was then heated at 90 °C with oil bath for 19 h. After cooled to room temperature, the reaction was quenched with sat. aq. NH_4Cl, and the product was extracted with AcOEt. The combined organic layer was washed with brine, dried over Na_2SO_4 and evaporated. The residue was purified by silica-gel column chromatography (hexane/AcOEt = 30:1) to provide **11** (31 mg, 0.082 mmol, 82%) as a yellow oil.

Procedure for Lithiation of 4aa and Trapping with Electrophiles (Scheme 5b).

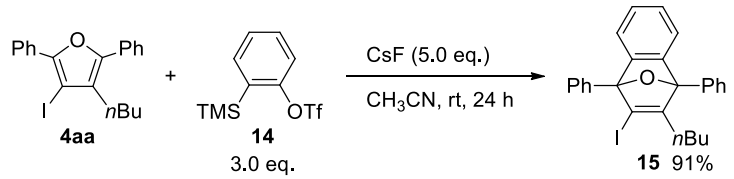


The reaction of **4aa** with benzaldehyde is representative.

To a solution of **4aa** (40 mg, 0.10 mmol) in THF (1.0 mL) was added a solution of *n*BuLi in hexane (1.6 M, 80 μL, 0.12 mmol) at −78 °C. After stirring at −78 °C for 1 h, benzaldehyde (15 μL, 0.15 mmol) was added. The resulting mixture was allowed to warm to room temperature, and stirred for 2 h. The reaction was quenched with sat. aq. NH_4Cl, and the product was extracted with AcOEt. The combined organic layer was washed with brine, dried over Na_2SO_4 and evaporated. The crude mixture was purified by silica-gel column chromatography (hexane/AcOEt = 10:1) to provide **12** (35 mg, 0.091 mmol, 91%) as a colorless oil.

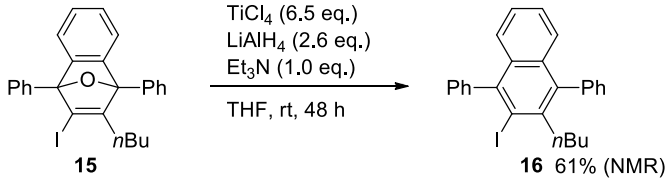
Procedure for Diels-Alder Reaction and Deoxygenative Aromatization (Scheme 5c).

Diels-Alder Reaction



To a solution of **4aa** (0.20 g, 0.50 mmol) in acetonitrile (10 mL) were sequentially added **14** (0.36 mL, 1.5 mmol), and cesium fluoride (0.38 g, 2.5 mmol). The resulting mixture then was stirred at room temperature for 24 h. The reaction was quenched with H_2O, and the product was extracted with AcOEt. The combined organic layer was washed with brine, dried over Na_2SO_4 and evaporated. The crude mixture was purified by silica-gel column chromatography (hexane/AcOEt = 30:1) followed by gel permeation chromatography to proviede **15** (0.22 g, 0.46 mmol, 91%) as a yellow sticky oil.

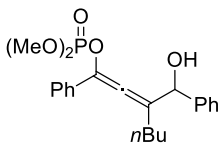
Deoxygenative Aromatization[S2]



To a reaction flask containing THF (0.50 mL) were added titanium tetrachloride (71 μL, 0.65 mmol), a solution of lithium aluminum hydride (9.9 mg, 0.26 mmol) in THF (1.0 mL) and triethylamine (14 μL, 0.10 mmol), and the mixture was stirred at room temperature for 10 min, and then at reflux for 30 min. After cooled to room temperature, a solution of **15** (40 mg, 0.10 mmol) in THF (0.50 mL) was added, and the resulting mixture was stirred at room temperature for 48 h. The reaction was then quenched with sat. aq. K_2CO_3 at 0 °C, and the product was extracted with CH_2Cl_2. The combined organic layer was washed with brine, dried over Na_2SO_4 and evaporated. The residue was purified by silica-gel column chromatography (hexane/AcOEt = 30:1) to provide **16** (61% NMR yield) along with a small amount of impurities (<5%). Further purification by recrystallization from hexane provided analytically pure **16** as a white solid.

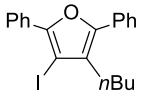
Analytical Data

2-Butyl-4-dimethoxyphosphoryloxy-1,4-diphenylbuta-2,3-dien-1-ol (3aa):



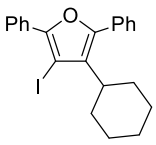
31 mg, 31% yield, Eluent, hexane/AcOEt = 1:1; Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) major diastereomer δ 0.79 (t, J = 7.2 Hz, 3H), 1.28 (qt, J = 7.2, 7.2 Hz, 2H), 1.36-1.45 (m, 2H), 1.87-1.94 (m, 1H), 1.98-2.05 (m, 1H), 3.85 (d, J = 11.4 Hz, 6H), 4.75 (brs, 1H), 5.33 (s, 1H), 7.26-7.31 (m, 2H), 7.32-7.40 (m, 4H), 7.49-7.52 (m, 4H); minor diastereomer δ 0.81 (t, J = 7.2 Hz, 3H), 1.20-1.30 (m, 2H), 1.36-1.51 (m, 2H), 1.98-2.05 (m, 1H), 2.11-2.18 (m, 1H), 3.85 (d, J = 11.4 Hz, 3H), 3.86 (d, J = 11.4 Hz, 3H), 4.75 (brs, 1H), 5.22 (s, 1H), 7.26-7.31 (m, 2H), 7.32-7.40 (m, 4H), 7.42-7.46 (m, 4H); ^{13}C NMR (150 MHz, $CDCl_3$) identifiable peaks for major diastereomer δ 13.8, 22.33, 29.6 (2C), 54.7 (d, J = 7.2 Hz), 54.8 (d, J = 5.7 Hz), 76.0, 189.9 (d, J = 2.9 Hz); identifiable peaks for minor diastereomer δ 13.8, 22.29, 29.4, 31.0, 54.8 (d, J = 7.2 Hz), 54.9 (d, J = 5.7 Hz), 77.2, 188.4 (d, J = 2.9 Hz); other peaks δ 124.5, 124.6, 126.9, 127.3, 127.4, 127.5, 127.6 (d, J = 7.2 Hz), 127.9, 128.1, 128.31, 128.33, 128.37, 128.42, 129.0 (d, J = 7.2 Hz), 129.2, 132.5 (d, J = 8.7 Hz), 141.3, 141.6; ^{31}P NMR (243 MHz, $CDCl_3$) δ -0.9 (major), -0.6 (minor); IR (ATR): 3370, 2954, 2930, 2865, 1952, 1497, 1451, 1268, 1191, 1038, 1009, 894, 853, 795, 763, 730, 698 cm^{-1}; HRMS (ESI) *m/z*: $[M+Na]^+$ Calcd for $C_{22}H_{27}O_5P$ 425.1488, Found 425.1488.

4-Butyl-3-iodo-2,5-diphenylfuran (4aa):



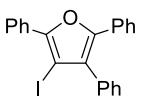
68 mg, 67% yield, Eluent, hexane/AcOEt = 50:1; Pale yellow solid; ^{1}H NMR (600 MHz, $CDCl_3$) δ 1.00 (t, J = 7.2 Hz, 3H), 1.50 (qt, J = 7.2, 7.2 Hz, 2H), 1.61-1.67 (m, 2H), 2.65-2.69 (m, 2H), 7.32 (tt, J = 7.2, 1.2 Hz, 1H), 7.35 (tt, J = 7.8, 1.2 Hz, 1H), 7.45 (dd, J = 7.8, 7.8 Hz, 4H), 7.68 (dd, J = 7.8, 1.2 Hz, 2H), 8.07 (dd, J = 7.8, 1.2 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 13.9, 22.8, 26.9, 31.7, 71.5, 125.6, 126.0, 126.4, 127.5, 128.1, 128.3, 128.7, 130.4, 130.9, 148.0, 150.1; IR (ATR): 3056, 2955, 2928, 2859, 1669, 1604, 1492, 1481, 1445, 1265, 1038, 946, 795, 764, 690 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{20}H_{19}IO$ 402.0481, Found 402.0480; mp. 52.0-55.0 °C.

4-Cyclohexyl-3-iodo-2,5-diphenylfuran (4ba):



65 mg, 61% yield, Eluent, hexane/AcOEt = 50:1; Pale yellow solid; ^{1}H NMR (600 MHz, $CDCl_3$) δ 1.23-1.31 (m, 1H), 1.32-1.39 (m, 2H), 1.69-1.76 (m, 3H), 1.81-1.87 (m, 2H), 2.00-2.08 (m, 2H), 2.86 (tt, J = 12.0, 3.6 Hz, 1H), 7.33 (tt, J = 7.2, 1.2 Hz, 1H), 7.38 (tt, J = 7.2, 1.2 Hz, 1H), 7.42 (tt, J = 7.2, 7.2 Hz, 2H), 7.44 (dd, J = 7.2, 7.2 Hz, 2H), 7.54 (dd, J = 7.2, 1.2 Hz, 2H), 7.99 (dd, J = 7.2, 1.2 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 25.9, 26.9, 31.1, 37.2, 67.0, 127.0, 128.08, 128.12, 128.2, 128.32, 128.35 (2C), 130.6, 131.3, 149.2, 151.1; IR (ATR): 3049, 2927, 2847, 1604, 1478, 1444, 1111, 1040, 936, 908, 761, 696, 666 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{22}H_{21}IO$ 428.0637, Found 428.0638; mp. 123.0-126.0 °C.

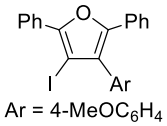
3-Iodo-2,4,5-triphenylfuran (4ca):



56 mg, 53% yield, Eluent, hexane/AcOEt = 30:1; Pale yellow solid; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.21 (tt, J = 7.2, 1.2 Hz, 1H), 7.25 (dd, J = 7.2, 7.2 Hz, 2H), 7.37 (dd, J = 7.2, 1.2 Hz, 2H), 7.39 (tt, J = 7.2, 1.2 Hz, 1H), 7.43-7.50 (m, 7H), 8.14 (dd, J = 7.2, 1.2 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 71.2, 125.5, 126.5, 127.7, 127.9, 128.1, 128.3, 128.38, 128.41, 128.7, 130.0, 130.2, 130.5, 133.9, 148.4, 150.5; IR

(ATR): 3064, 3025, 2939, 2852, 1600, 1486, 1442, 1176, 1063, 1025, 940, 761, 688, 660 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{22}H_{15}IO$ 422.0168, Found 422.0167; mp. 157.0-160.0 °C.

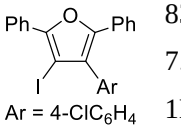
3-Iodo-4-(4-methoxyphenyl)-2,5-diphenylfuran (4da):



Ar = 4-$MeOC_6H_4$

43% NMR yield, 49 mg, abt. 95% purity, Eluent, hexane/AcOEt = 30:1 (analytically pure product was isolated after purification by recrystallization from AcOEt and hexane, 22 mg, 19%); Yellow solid; ^{1}H NMR (600 MHz, $CDCl_3$) δ 3.88 (s, 3H), 7.00 (d, *J* = 9.0 Hz, 2H), 7.20 (tt, *J* = 7.2, 1.2 Hz, 1H), 7.25 (dd, *J* = 7.2, 7.2 Hz, 2H), 7.26 (d, *J* = 9.0 Hz, 2H), 7.37 (t, *J* = 7.2 Hz, 1H), 7.44-7.49 (m, 4H), 8.13 (d, *J* = 7.2 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 56.2, 72.0, 114.2, 125.4, 126.0, 126.5, 127.6, 128.3, 128.38, 128.40 (2C), 130.2, 130.3, 131.7, 148.4, 150.3, 159.4; IR (ATR): 3077, 2954, 2836, 1604, 1510, 1489, 1940, 1290, 1247, 1175, 1061, 1027,939, 832, 761, 689 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{23}H_{17}IO_2$ 452.0273, Found 452.0273; mp. 139.0-142.0 °C.

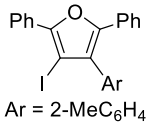
4-(4-Chlorophenyl)-3-iodo-2,5-diphenylfuran (4ea):



Ar = 4-ClC_6H_4

83 mg, 73% yield, Eluent, hexane/AcOEt = 30:1; Pale yellow solid; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.24 (t, *J* = 7.2 Hz, 1H), 7.28 (dd, *J* = 7.2, 7.2 Hz, 2H), 7.31 (d, *J* = 8.4 Hz, 2H), 7.39 (t, *J* = 7.2 Hz, 1H), 7.43 (d, *J* = 7.2 Hz, 2H), 7.45 (d, *J* = 8.4 Hz, 2H), 7.48 (dd, *J* = 7.2, 7.2 Hz, 2H), 8.11 (d, *J* = 7.2 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 70.7, 125.6, 126.5, 126.6, 127.9, 128.4 (2C), 128.5, 129.0, 129.7, 130.1, 131.9, 132.3, 134.2, 148.7, 150.8; IR (ATR): 3082, 2972, 2902, 1599, 1484, 1442, 1398, 1088, 1059, 1014, 941, 839, 766, 688 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{22}H_{14}ClIO$ 455.9778, Found 455.9778; mp. 138.0-142.0 °C.

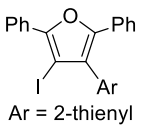
3-Iodo-2,5-diphenyl-4-(2-tolyl)furan (4fa):



Ar = 2-MeC_6H_4

34 mg, 32% yield, Eluent, hexane/AcOEt = 30:1; Yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 2.15 (s, 3H), 7.19 (tt, *J* = 7.2, 1.2 Hz, 1H), 7.22 (dd, *J* = 7.2, 1.2 Hz, 1H), 7.24 (dd, *J* = 7.2, 7.2 Hz, 2H), 7.32 (ddd, *J* = 7.2, 7.2, 1.2 Hz, 1H), 7.35-7.41 (m, 5H), 7.49 (dd, *J* = 7.8, 7.2 Hz, 2H), 8.18 (dd, *J* = 7.2, 1.2 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 20.0, 71.6, 124.6, 126.28, 126.32, 127.5, 127.6, 128.3, 128.4, 128.5, 128.6, 130.2, 130.3, 130.4, 130.8, 133.7, 137.7, 148.0, 150.3; IR (ATR): 3057, 3020, 2921, 2849, 1670, 1599, 1483, 1444, 1245, 1179, 1062, 1029, 939, 910, 761, 688 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{23}H_{17}IO$ 436.0324, Found 436.0324.

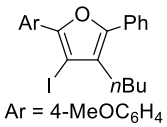
3-Iodo-2,5-diphenyl-4-(2-thienyl)furan (4ga):



Ar = 2-thienyl

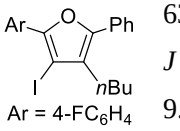
86 mg, 80% yield, Eluent, hexane/AcOEt = 30:1; Brown solid; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.10 (dd, *J* = 3.6, 0.6 Hz, 1H), 7.18 (dd, *J* = 5.4, 3.6 Hz, 1H), 7.23-7.25 (m, 1H), 7.30 (dd, *J* = 7.2, 7.2 Hz, 2H), 7.39 (t, *J* = 7.2 Hz, 1H), 7.48 (dd, *J* = 7.8, 7.2 Hz, 2H), 7.49 (dd, *J* = 5.4, 0.6 Hz, 1H), 7.53 (d, *J* = 7.8 Hz, 2H), 8.11 (dd, *J* = 8.4, 1.2 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 72.5, 121.0, 125.6, 126.5, 127.2, 127.5, 128.1, 128.4 (3C), 129.0, 129.6, 130.0, 134.2, 150.0, 150.6; IR (ATR): 3074, 3026, 2927, 2849, 1599, 1478, 1442, 1218, 1156, 1054, 943, 917, 849, 761, 687 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{20}H_{13}IOS$ 427.9732, Found 427.9731; mp. 123.0-125.0 °C.

4-Butyl-3-iodo-2-(4-methoxyphenyl)-5-phenylfuran (4ha):



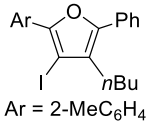
76 mg, 70% yield, Eluent, hexane/AcOEt = 30:1; Yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 0.99 (t, J = 7.2 Hz, 3H), 1.49 (qt, J = 7.2, 7.2 Hz, 2H), 1.61-1.66 (m, 2H), 2.64-2.68 (m, 2H), 3.86 (s, 3H), 6.97 (d, J = 9.0 Hz, 2H), 7.30 (t, J = 7.8 Hz, 1H), 7.43 (dd, J = 7.8, 7.8 Hz, 2H), 7.66 (d, J = 7.8 Hz, 2H), 7.99 (d, J = 9.0 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 13.9, 22.8, 26.9, 31.7, 55.3, 70.0, 113.7, 123.2, 125.4, 125.8, 127.3, 127.9, 128.6, 131.0, 147.5, 150.3, 159.4; IR (ATR): 3033, 2952, 2856, 1604, 1549, 1493, 1459, 1303, 1240, 1176, 1100, 1034, 944, 818, 764, 689 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{21}H_{21}IO_2$ 432.0586, Found 432.0587.

4-Butyl-2-(4-fluorophenyl)-3-iodo-5-phenylfuran (4ia):



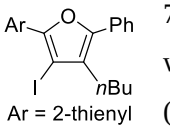
63 mg, 60% yield, Eluent, hexane/AcOEt = 50:1; Yellow solid; ^{1}H NMR (600 MHz, $CDCl_3$) δ 0.99 (t, J = 7.2 Hz, 3H), 1.48 (qt, J = 7.2, 7.2 Hz, 2H), 1.60-1.66 (m, 2H), 2.64-2.67 (m, 2H), 7.13 (dd, J = 9.0, 9.0 Hz, 2H), 7.32 (tt, J = 7.8, 1.2 Hz, 1H), 7.44 (dd, J = 7.8, 7.2 Hz, 2H), 7.66 (dd, J = 7.2, 1.2 Hz, 2H), 8.04 (dd, J = 9.0, 5.4 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 13.9, 22.8, 26.9, 31.7, 71.2, 115.4 (d, J = 21.5 Hz), 125.6, 126.0, 126.7 (d, J = 2.9 Hz), 127.6, 128.3 (d, J = 8.6 Hz), 128.7, 130.8, 148.1, 149.4, 162.4 (d, J = 247.1 Hz); IR (ATR): 3072, 2949, 2858, 1591, 1545, 1486, 1445, 1234, 1159, 1093, 1038, 942, 834, 764, 691 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{20}H_{18}FIO$ 420.0386, Found 420.0385; mp. 51.0-53.0 °C.

4-Butyl-3-iodo-5-phenyl-2-(2-tolyl)furan (4ja):



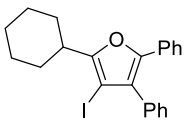
79 mg, 76% yield, Eluent, hexane/AcOEt = 40:1; Yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 1.01 (t, J = 7.2 Hz, 3H), 1.50 (qt, J = 7.2, 7.2 Hz, 2H), 1.64-1.69 (m, 2H), 2.40 (s, 3H), 2.66-2.70 (m, 2H), 7.27-7.34 (m, 4H), 7.42 (dd, J = 7.8, 7.8 Hz, 2H), 7.55 (d, J = 7.2 Hz, 1H), 7.63 (d, J = 7.8 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 14.0, 20.7, 22.8, 27.0, 31.8, 74.3, 124.7, 125.4 (2C), 127.4, 128.6, 129.2, 130.0, 130.6, 130.8, 131.1, 137.9, 148.3, 152.7; IR (ATR): 3060, 2957, 2929, 2870, 1492, 1454, 1086, 948, 908, 761, 723, 693 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{21}H_{21}IO$ 416.0637, Found 416.0638.

4-Butyl-3-iodo-5-phenyl-2-(2-thienyl)furan (4ka):



70% NMR yield, 71 mg, abt. 95% purity, Eluent, hexane/AcOEt = 40:1 (analytically pure product was isolated after purification by recrystallization from hexane, 17 mg, 17%); Brown solid; ^{1}H NMR (600 MHz, $CDCl_3$) δ 0.99 (t, J = 7.2 Hz, 3H), 1.48 (qt, J = 7.2, 7.2 Hz, 2H), 1.60-1.65 (m, 2H), 2.63-2.67 (m, 2H), 7.12 (dd, J = 4.8, 3.6 Hz, 1H), 7.31 (t, J = 7.2 Hz, 1H), 7.34 (dd, J = 4.8, 0.6 Hz, 1H), 7.44 (dd, J = 7.2, 7.2 Hz, 2H), 7.66 (d, J = 7.2 Hz, 2H), 7.76 (dd, J = 3.6, 0.6 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 13.9, 22.7, 26.7, 31.6, 71.4, 124.8, 125.1, 125.6, 126.0, 127.3, 127.5, 128.7, 130.6, 132.5, 147.4, 147.5; IR (ATR): 3073, 2944, 2857, 1589, 1485, 1199, 1074, 1038, 930, 896, 767, 688 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{18}H_{17}IOS$ 408.0045, Found 408.0046; mp. 61.0-64.0 °C.

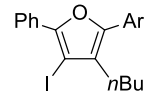
2-Cyclohexyl-3-iodo-4,5-diphenylfuran (4ma):



74 mg, 69% yield, Eluent, hexane/AcOEt = 30:1; Pale yellow solid; ^{1}H NMR (600 MHz, $CDCl_3$) δ 1.29-1.37 (m, 1H), 1.38-1.46 (m, 2H), 1.67-1.73 (m, 2H), 1.74-1.77 (m, 1H), 1.86-1.90 (m, 2H), 1.93-1.96 (m, 2H), 2.88 (tt, J = 12.0, 3.6 Hz, 1H), 7.16 (tt, J = 7.2, 1.2 Hz, 1H), 7.21

(dd, *J* = 7.2, 7.2 Hz, 2H), 7.33 (dd, *J* = 7.2, 1.2 Hz, 2H), 7.35 (dd, *J* = 7.2, 1.2 Hz, 2H), 7.39 (tt, *J* = 7.2, 1.2 Hz, 1H), 7.42 (dd, *J* = 7.2, 7.2 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 25.9, 26.2, 31.0, 37.8, 69.9, 125.1, 125.2, 127.1, 127.8, 128.3, 128.5, 130.3, 130.6, 134.0, 147.3, 158.8; IR (ATR): 3054, 2925, 2854, 1599, 1557, 1479, 1442, 1249, 1170, 1068, 1011, 985, 951, 766, 689 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{22}H_{21}IO$ 428.0637, Found 428.0637; mp. 90.0-93.0 °C.

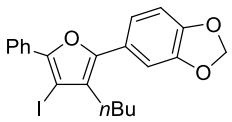
4-Butyl-3-iodo-5-(4-methoxyphenyl)-2-phenylfuran (4ab):



Ar = 4-MeOC$_6$H$_4$

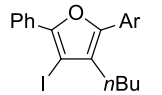
77 mg, 72% yield, Eluent, hexane/AcOEt = 40:1; Yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 0.99 (t, *J* = 7.2 Hz, 3H), 1.48 (qt, *J* = 7.2, 7.2 Hz, 2H), 1.59-1.66 (m, 2H), 2.61-2.65 (m, 2H), 3.86 (s, 3H), 6.98 (d, *J* = 9.0 Hz, 2H), 7.33 (tt, *J* = 7.8, 1.2 Hz, 1H), 7.43 (dd, *J* = 7.8, 7.2 Hz, 2H), 7.60 (d, *J* = 9.0 Hz, 2H), 8.05 (dd, *J* = 7.2, 1.2 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 13.9, 22.8, 26.8, 31.8, 55.3, 71.3, 114.1, 123.8, 124.6, 126.3, 127.1, 127.9, 128.3, 130.6, 148.2, 149.5, 159.1; IR (ATR): 3062, 2955, 2929, 2858, 2834, 1609, 1504, 1462, 1293, 1251, 1177, 1090, 1039, 890, 763, 690 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{21}H_{21}IO_2$ 432.0586, Found 432.0586.

4-Butyl-3-iodo-5-(3,4-methylenedioxyphenyl)-2-phenylfuran (4ac):



74 mg, 66% yield, Eluent, hexane/AcOEt = 30:1; Pale yellow solid; ^{1}H NMR (600 MHz, $CDCl_3$) δ 0.99 (t, *J* = 7.2 Hz, 3H), 1.48 (qt, *J* = 7.2, 7.2 Hz, 2H), 1.58-1.64 (m, 2H), 2.59-2.64 (m, 2H), 6.00 (s, 2H), 6.88 (d, *J* = 9.0 Hz, 2H), 7.14 (d, *J* = 9.0 Hz, 1H), 7.15 (s, 1H), 7.33 (t, *J* = 7.2 Hz, 1H), 7.43 (dd, *J* = 7.8, 7.2 Hz, 2H), 8.05 (d, *J* = 7.8 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 13.9, 22.8, 26.8, 31.7, 71.3, 101.2, 106.4, 108.6, 119.7, 124.9, 125.1, 126.3, 128.0, 128.3, 130.4, 147.1, 147.9 (2C), 149.6; IR (ATR): 2956, 2925, 2858, 1599, 1478, 1387, 1222, 1037, 947, 869, 818, 762, 687 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{21}H_{19}IO_3$ 446.0379, Found 446.0379; mp. 91.0-93.0 °C.

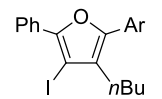
4-Butyl-5-(4-cyanophenyl)-3-iodo-2-phenylfuran (4ad):



Ar = 4-CN-C$_6$H$_4$

55 mg, 51% yield, Eluent, hexane/AcOEt = 30:1; Yellow solid; ^{1}H NMR (600 MHz, $CDCl_3$) δ 1.01 (t, *J* = 7.2 Hz, 3H), 1.51 (qt, *J* = 7.2, 7.2 Hz, 2H), 1.60-1.65 (m, 2H), 2.69-2.73 (m, 2H), 7.39 (tt, *J* = 7.2, 1.2 Hz, 1H), 7.47 (dd, *J* = 7.8, 7.2 Hz, 2H), 7.71 (d, *J* = 9.0 Hz, 2H), 7.77 (d, *J* = 9.0 Hz, 2H), 8.06 (dd, *J* = 7.8, 1.2 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 13.9, 22.8, 27.2, 31.4, 72.1, 110.3, 118.8, 125.3, 126.6, 128.5, 128.7, 129.4, 129.8, 132.5, 134.8, 145.9, 151.6; IR (ATR): 2965, 2928, 2857, 2224, 1606, 1477, 1262, 1179, 1075, 943, 838, 767, 688 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{21}H_{18}INO$ 427.0433, Found 427.0433; mp. 95.0-98.0 °C.

4-Butyl-3-iodo-5-(4-nitrophenyl)-2-phenylfuran (4ae):

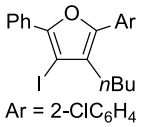


Ar = 4-NO$_2$-C$_6$H$_4$

34 mg, 31% yield, Eluent, hexane/AcOEt = 30:1; Yellow solid; ^{1}H NMR (600 MHz, $CDCl_3$) δ 1.02 (t, *J* = 7.2 Hz, 3H), 1.53 (qt, *J* = 7.2, 7.2 Hz, 2H), 1.61-1.67 (m, 2H), 2.72-2.76 (m, 2H), 7.41 (t, *J* = 7.8 Hz, 1H), 7.48 (dd, *J* = 7.8, 7.8 Hz, 2H), 7.83 (d, *J* = 9.0 Hz, 2H), 8.08 (d, *J* = 7.8 Hz, 2H), 8.31 (d, *J* = 9.0 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 13.9, 22.8, 27.4, 31.3, 72.4, 124.3, 125.2, 126.7, 128.5, 128.9, 129.8, 130.2, 136.6, 145.7, 146.1, 152.1; IR (ATR): 3099, 3050, 1962, 2925, 2859, 1672, 1596, 1509, 1448, 1335, 1259, 1105, 943, 850, 766, 690 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{20}H_{18}INO_3$ 447.0331, Found

447.0331; mp. 103.0-106.0 °C.

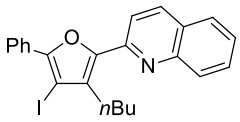
4-Butyl-5-(2-chlorophenyl)-3-iodo-2-phenylfuran (4af):



Ar = 2-ClC$_6$H$_4$

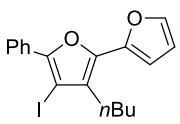
75 mg, 69% yield, Eluent, hexane/AcOEt = 50:1; Yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 0.85 (t, *J* = 7.2 Hz, 3H), 1.29 (qt, *J* = 7.2, 7.2 Hz, 2H), 1.49-1.54 (m, 2H), 2.43-2.47 (m, 2H), 7.32-7.37 (m, 3H), 7.41-7.44 (m, 3H), 7.50 (dd, *J* = 7.2, 1.8 Hz, 1H), 8.04 (dd, *J* = 7.2, 1.2 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 13.7, 22.3, 26.1, 31.5, 69.0, 126.4, 126.5, 127.6, 128.1, 128.3, 130.0, 130.1, 130.2, 130.4, 131.9, 134.4, 146.5, 151.1; IR (ATR): 3059, 2955, 2926, 2858, 1483, 1438, 1236, 1091, 907, 759, 731, 690 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{20}H_{18}ClIO$ 436.0091, Found 436.0091.

4-Butyl-3-iodo-2-phenyl-5-(quinolin-2-yl)furan (4ag):



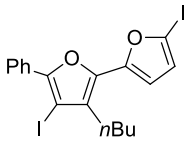
77 mg, 68% yield, Eluent, hexane/AcOEt = 50:1; Brown solid; ^{1}H NMR (600 MHz, $CDCl_3$) δ 1.04 (t, *J* = 7.2 Hz, 3H), 1.57 (qt, *J* = 7.2, 7.2 Hz, 2H), 1.69-1.75 (m, 2H), 3.17-3.21 (m, 2H), 7.39 (tt, *J* = 7.8, 1.2 Hz, 2H), 7.48 (dd, *J* = 7.8, 7.8 Hz, 2H), 7.49 (ddd, *J* = 7.8, 7.2, 1.2 Hz, 1H), 7.70 (ddd, *J* = 8.4, 7.8, 1.2 Hz, 1H), 7.79 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.96 (d, *J* = 8.4 Hz, 1H), 8.04 (d, *J* = 8.4 Hz, 1H), 8.14 (dd, *J* = 7.2, 1.2 Hz, 2H), 8.17 (d, *J* = 8.4 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 14.1, 22.9, 27.1, 31.4, 72.9, 117.8, 126.1, 126.6, 126.7, 127.5, 128.4, 128.5, 129.4, 129.6, 130.3, 132.1, 136.1, 147.3, 148.1, 149.9, 150.9; IR (ATR): 3052, 2952, 2914, 2849, 1596, 1551, 1528, 1494, 1420, 1312, 1086, 954, 928, 825, 750, 681 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{23}H_{20}INO$ 453.0590, Found 453.0589; mp. 87.0-90.0 °C.

4-Butyl-5-(2-furyl)-3-iodo-2-phenylfuran (4ah):



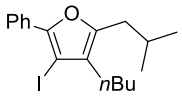
50 mg, 50% yield, Eluent, hexane; Brown oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 0.98 (t, *J* = 7.2 Hz, 3H), 1.45 (qt, *J* = 7.2, 7.2 Hz, 2H), 1.56-1.61 (m, 2H), 2.67-2.71 (m, 2H), 6.51 (dd, *J* = 3.6, 2.4 Hz, 1H), 6.60 (dd, *J* = 3.6, 0.6 Hz, 1H), 7.34 (tt, *J* = 7.8, 1.2 Hz, 1H), 7.44 (dd, *J* = 7.8, 7.8 Hz, 2H), 7.48 (dd, *J* = 2.4, 0.6 Hz, 1H), 8.04 (dd, *J* = 7.8, 1.2 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 14.0, 22.6, 26.1, 31.8, 70.7, 106.2, 111.3, 126.1, 126.4, 128.1, 128.3, 130.2, 141.3, 142.0, 146.2, 150.0; IR (ATR): 2956, 2926, 2863, 1668, 1598, 1528, 1462, 1376, 1168, 1009, 952, 729, 689 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{18}H_{17}IO_2$ 392.0273, Found 392.0273.

4-Butyl-3-iodo-5-(2-iodofur-5-yl)-2-phenylfuran (4ah'):



77 mg, 60% yield, Eluent, hexane; Ocher solid; ^{1}H NMR (600 MHz, $CDCl_3$) δ 1.00 (t, *J* = 7.2 Hz, 3H), 1.46 (qt, *J* = 7.2, 7.2 Hz, 2H), 1.54-1.61 (m, 2H), 2.65-2.69 (m, 2H), 6.49 (d, *J* = 3.6 Hz, 1H), 6.64 (d, *J* = 3.6 Hz, 1H), 7.35 (tt, *J* = 7.8, 1.2 Hz, 1H), 7.44 (dd, *J* = 7.8, 7.2 Hz, 2H), 8.03 (dd, *J* = 7.8, 1.2 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 14.0, 22.5, 26.1, 31.7, 70.7, 87.1, 108.8, 121.9, 126.4, 127.1, 128.30, 128.34, 130.0, 140.2, 150.3, 151.3; IR (ATR): 2955, 2928, 2859, 1672, 1600, 1522, 1462, 1195, 1088, 1009, 956, 913, 762, 687 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{18}H_{16}I_2O_2$ 517.9240, Found 517.9239; mp. 49.0-52.0 °C.

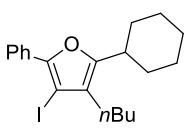
4-Butyl-3-iodo-5-isobutyl-2-phenylfuran (4ai):



29 mg, 30% yield, Eluent, hexane; Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 0.96 (t, *J* = 7.2 Hz, 3H), 0.96 (d, *J* = 7.2 Hz, 6H), 1.39 (qt, *J* = 7.2, 7.2 Hz, 2H), 1.47-1.52 (m, 2H), 2.04 (dsept,

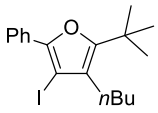
J = 7.2, 7.2 Hz, 1H), 2.33-2.36 (m, 2H), 2.52 (d, *J* = 7.2 Hz, 2H), 7.29 (tt, *J* = 7.8, 1.2 Hz, 1H), 7.40 (dd, *J* = 7.8, 7.8 Hz, 2H), 7.96 (dd, *J* = 7.8, 1.2 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 14.0, 22.5, 22.6, 25.7, 28.2, 32.3, 35.6, 68.6, 124.5, 126.0, 127.5, 128.2, 130.9, 149.1, 150.6; IR (ATR): 2955, 2928, 2871, 1603, 1465, 1384, 1235, 1089, 890, 761, 689 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{18}H_{23}IO$ 382.0794, Found 382.0794.

4-Butyl-5-cyclohexyl-3-iodo-2-phenylfuran (4aj):



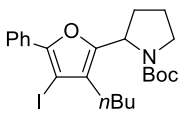
68 mg, 66% yield, Eluent, hexane/AcOEt = 30:1; Yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 0.96 (t, *J* = 7.2 Hz, 3H), 1.25-1.43 (m, 5H), 1.46-1.51 (m, 2H), 1.61-1.68 (m, 2H), 1.70-1.76 (m, 1H), 1.77-1.88 (m, 4H), 2.35-2.38 (m, 2H), 2.67 (tt, *J* = 6.0, 3.6 Hz, 1H), 7.28 (t, *J* = 7.8 Hz, 1H), 7.39 (dd, *J* = 7.8, 7.8 Hz, 2H), 7.96 (d, *J* = 7.8 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 14.0, 22.5, 25.6, 25.9, 26.4, 31.8, 32.6, 36.6, 68.9, 122.3, 125.9, 127.4, 128.2, 130.9, 148.6, 155.1; IR (ATR): 2927, 2853, 1601, 1447, 1378, 1256, 1093, 1069, 954, 760, 689 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{20}H_{25}IO$ 408.0950, Found 408.0951.

4-Butyl-5-(*tert*-butyl)-3-iodo-2-phenylfuran (4ak):



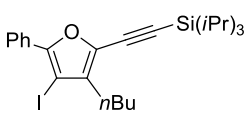
23 mg, 24% yield, Eluent, hexane/AcOEt = 30:1; Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 0.98 (t, *J* = 7.2 Hz, 3H), 1.39 (s, 9H), 1.42-1.52 (m, 4H), 2.46-2.50 (m, 2H), 7.29 (tt, *J* = 7.8, 1.2 Hz, 1H), 7.40 (dd, *J* = 7.8, 7.8 Hz, 2H), 7.96 (dd, *J* = 7.8, 1.2 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 13.9, 23.0, 26.7, 29.7, 33.2, 34.3, 71.0, 122.2, 126.0, 127.5, 128.2, 130.9, 148.0, 156.3; IR (ATR): 2958, 2925, 2868, 1484, 1363, 1236, 1089, 895, 762, 691 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{18}H_{23}IO$ 382.0794, Found 382.0793.

5-(1-*tert*-Butoxycarbonylpyrrolidin-2-yl)-4-butyl-3-iodo-2-phenylfuran (4al):



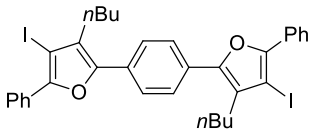
63 mg, 51% yield, Eluent, hexane/AcOEt = 20:1; Colorless oil; Ratio of rotamers = 70:30; ^{1}H NMR (600 MHz, $CDCl_3$) δ 0.96 (t, *J* = 7.2 Hz, 3H), 1.20-1.60 (m, 5H), 1.28 (s, 6.3H), 1.44 (s, 2.7H), 1.88-2.05 (m, 2H), 2.10-2.30 (m, 2H), 2.32-2.63 (m, 2H), 3.43-3.69 (m, 2H), 4.84 (m, 0.70 H), 5.03 (m, 0.30 H), 7.30 (t, *J* = 7.2 Hz, 1H), 7.40 (dd, *J* = 7.8, 7.2 Hz, 2H), 7.94 (d, *J* = 7.8 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 13.9, 22.5, 23.7, 24.5, 25.5, 25.7, 28.3, 28.5, 32.0, 32.4, 33.3, 46.6, 46.7, 52.9, 53.2, 68.5, 69.5, 79.3, 79.6, 124.0, 124.7, 126.0, 127.7, 127.8, 128.3, 130.4, 130.7, 149.1, 150.5, 150.8, 154.2; IR (ATR): 2957, 2925, 2869, 1692, 1477, 1442, 1392, 1251, 1161, 1108, 1072, 950, 689 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{23}H_{30}INO_3$ $[M]^+$ 495.1270, Found 495.1271.

4-Butyl-3-iodo-2-phenyl-5-(triisopropylsilylethynyl)furan (4am):



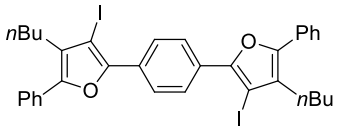
89 mg, 70% yield, Eluent, hexane/AcOEt = 50:1; Brown oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 0.95 (t, *J* = 7.2 Hz, 3H), 1.11-1.19 (m, 21H), 1.41 (qt, *J* = 7.2, 7.2 Hz, 2H), 1.60 (tt, *J* = 7.2, 7.2 Hz, 2H), 2.51 (t, *J* = 7.2 Hz, 2H), 7.33 (tt, *J* = 7.2, 1.2 Hz, 1H), 7.42 (dd, *J* = 7.8, 7.2 Hz, 2H), 8.00 (dd, *J* = 7.8, 1.2 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 11.2, 13.9, 18.6, 22.5, 26.8, 31.3, 67.9, 95.3, 100.2, 126.7, 128.3, 128.5, 130.0, 134.0, 136.0, 151.5; IR (ATR): 2943, 2863, 2145, 1459, 1136, 1066, 957, 882, 763, 731, 688 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{25}H_{35}IOSi$ 506.1502, Found 506.1502.

1,4-Bis(4-butyl-3-iodo-2-phenylfur-5-yl)benzene (4an):



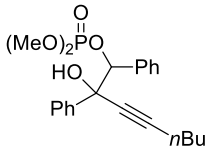
22 mg, 30% yield, Eluent, hexane/AcOEt = 60:1; Yellow solid; ^{1}H NMR (600 MHz, $CDCl_3$) δ 1.02 (t, J = 7.2 Hz, 6H), 1.52 (qt, J = 7.2, 7.2 Hz, 4H), 1.64-1.70 (m, 4H), 2.69-2.74 (m, 4H), 7.36 (tt, J = 7.2, 1.2 Hz, 2H), 7.46 (dd, J = 7.8, 7.2 Hz, 4H), 7.76 (s, 4H), 8.09 (dd, J = 7.8, 1.2 Hz, 4H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 13.9, 22.8, 27.1, 31.7, 71.8, 125.6, 126.4, 126.7, 128.2, 128.4, 129.7, 130.4, 147.6, 150.3; IR (ATR): 2955, 2925, 2858, 1671, 1591, 1492, 1259, 1105, 1034, 945, 833, 796, 692 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{34}H_{32}I_2O_2$ 726.0492, Found 726.0491; mp. 150.0-153.0 °C.

1,4-Bis(4-butyl-3-iodo-5-phenylfur-2-yl)benzene (4na):



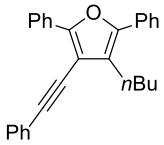
26 mg, 36% yield, Eluent, hexane/AcOEt = 60:1; Yellow solid; ^{1}H NMR (600 MHz, $CDCl_3$) δ 1.00 (t, J = 7.2 Hz, 6H), 1.50 (qt, J = 7.2, 7.2 Hz, 4H), 1.62-1.68 (m, 4H), 2.67-2.70 (m, 4H), 7.33 (tt, J = 7.2, 1.2 Hz, 2H), 7.46 (dd, J = 7.8, 7.2 Hz, 4H), 7.70 (dd, J = 7.8, 1.2 Hz, 4H), 8.19 (s, 4H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 13.9, 22.8, 26.9, 31.7, 72.0, 125.6, 126.0, 126.4, 127.6, 128.7, 129.7, 130.8, 148.2, 149.6; IR (ATR): 3056, 2952, 2930, 2858, 1662, 1591, 1493, 1443, 1262, 1104, 1038, 942, 834, 799, 694 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{34}H_{32}I_2O_2$ 726.0492, Found 726.0490; mp. 150.0-153.0 °C.

1-Dimethoxyphosphoryloxy-1,2-diphenyl-3-octyn-2-ol (5aa):



The isolated amount is not available for this byproduct. This compound was finally isolated from the combined crude mixtures which were obtained during the optimized reaction conditions. Eluent, hexane/AcOEt = 1:1; Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) major diastereomer δ 0.92 (t, J = 7.2 Hz, 3H), 1.38-1.45 (m, 2H), 1.51-1.56 (m, 2H), 2.29 (t, J = 7.2 Hz, 2H), 2.80 (brs, 1H), 3.37 (d, J = 10.8 Hz, 3H), 3.46 (d, J = 10.8 Hz, 3H), 5.40 (d, J = 8.4 Hz, 1H), 7.23-7.31 (m, 8H), 7.54 (dd, J = 7.2, 1.8 Hz, 2H); minor diastereomer δ 0.93 (t, J = 7.2 Hz, 3H), 1.38-1.45 (m, 2H), 1.51-1.56 (m, 2H), 2.29 (t, J = 7.2 Hz, 2H), 3.54 (d, J = 11.4 Hz, 3H), 3.60 (d, J = 11.4 Hz, 3H), 5.36 (d, J = 8.4 Hz, 1H), 7.11 (d, J = 7.2 Hz, 2H), 7.18 (dd, J = 7.2, 7.2 Hz, 2H), 7.22-7.32 (m, 3H), 7.41-7.43 (m, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) major diastereomer δ 13.5, 18.5, 22.0, 30.37, 54.0 (d, J = 5.7 Hz), 54.1 (d, J = 5.7 Hz), 75.3 (d, J = 7.1 Hz), 79.8, 85.2 (d, J = 5.7 Hz), 89.5, 127.16, 127.3, 127.6, 128.0, 128.55, 128.57, 135.6, 140.1; minor diastereomer δ 13.5, 18.5, 21.9, 30.40, 54.25 (d, J = 5.9 Hz), 54.31 (d, J = 5.7 Hz), 76.3 (d, J = 7.2 Hz), 78.8, 86.5 (d, J = 5.7 Hz), 89.5, 126.9, 127.19, 127.7, 128.0, 128.1, 128.4, 135.4 (d, J = 2.9 Hz), 140.2; ^{31}P NMR (243 MHz, $CDCl_3$) δ 1.3 (major), 1.8 (minor); IR (ATR): 3384, 2958, 2929, 2866, 1496, 1449, 1258, 1181, 1040, 1014, 962, 882, 846, 696 cm^{-1}; HRMS (ESI) *m/z*: $[M+Na]^+$ Calcd for $C_{22}H_{27}O_5P$425.1488, Found 425.1488.

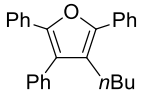
3-Butyl-2,5-diphenyl-4-(phenylethynyl)furan (8):



37 mg, 99% yield, Eluent, hexane; Orange solid; ^{1}H NMR (600 MHz, $CDCl_3$) δ 0.99 (t, J = 7.2 Hz, 3H), 1.51 (qt, J = 7.2, 7.2 Hz, 2H), 1.74-1.80 (m, 2H), 2.81-2.85 (m, 2H), 7.31 (tt, J = 7.2, 1.2 Hz, 2H), 7.36-7.42 (m, 3H), 7.43-7.48 (m, 4H), 7.57 (dd, J = 7.2, 1.2 Hz, 2H), 7.73 (dd, J = 7.2, 1.2 Hz, 2H), 8.20 (dd, J = 7.2, 1.2 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 14.0, 22.8, 24.6, 31.8, 82.3, 96.1, 106.9, 123.6, 124.8, 125.3, 125.6, 127.3, 127.9, 128.2, 128.5, 128.6, 128.7, 130.5, 131.1, 131.3, 147.4, 152.7;

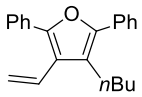
IR (ATR): 3060, 2956, 2925, 2854, 2208, 1596, 1483, 1442, 1259, 1027, 911, 752, 686 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{28}H_{24}O$ 376.1827, Found 376.1827; mp. 55.0-58.0 °C.

3-Butyl-2,4,5-triphenylfuran (9):



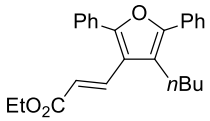
34 mg, 97% yield, Eluent, hexane; Pale yellow solid; ^{1}H NMR (600 MHz, $CDCl_3$) δ 0.77 (t, *J* = 7.2 Hz, 3H), 1.23 (qt, *J* = 7.2, 7.2 Hz, 2H), 1.39-1.44 (m, 2H), 2.53-2.56 (m, 2H), 7.16 (tt, *J* = 7.2, 1.2 Hz, 1H), 7.22 (dd, *J* = 7.2, 7.2 Hz, 2H), 7.30 (tt, *J* = 7.2, 1.2 Hz, 1H), 7.35 (dd, *J* = 7.2, 1.2 Hz, 2H), 7.40 (tt, *J* = 7.2, 1.2 Hz, 1H), 7.43-7.46 (m, 6H), 7.76 (dd, *J* = 7.8, 1.2 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 13.7, 22.6, 23.8, 32.0, 124.3, 125.3, 125.5, 125.8, 126.87, 126.91, 127.4, 128.2, 128.6, 128.8, 130.2, 131.0, 131.7, 134.1, 147.1, 147.4; IR (ATR): 3049, 2948, 2860, 1600, 1491, 1444, 1257, 1073, 1024, 952, 911, 764, 677 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{26}H_{24}O$ 352.1827, Found 352.1828; mp. 89.0-92.0 °C.

3-Butyl-2,5-diphenyl-4-vinylfuran (10):



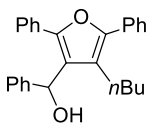
27 mg, 89% yield, Eluent, hexane/AcOEt = 50:1; Pale yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 0.97 (t, *J* = 7.2 Hz, 3H), 1.45 (qt, *J* = 7.2, 7.2 Hz, 2H), 1.63-1.68 (m, 2H), 2.72-2.76 (m, 2H), 5.41 (dd, *J* = 12.0, 1.2 Hz, 1H), 5.55 (dd, *J* = 18.0, 1.2 Hz, 1H), 6.75 (dd, *J* = 18.0, 12.0 Hz, 1H), 7.28-7.32 (m, 2H), 7.41 (dd, *J* = 7.8, 7.2 Hz, 2H), 7.44 (dd, *J* = 7.8, 7.2 Hz, 2H), 7.69 (dd, *J* = 7.8, 1.2 Hz, 2H), 7.74 (dd, *J* = 7.8, 1.2 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 13.9, 22.9, 24.1, 32.1, 117.4, 121.7, 122.8, 125.8, 126.5, 127.0, 127.4, 128.4, 128.6 (2C), 131.3, 131.6, 148.1, 148.9; IR (ATR): 3056, 2952, 2954, 2871, 1672, 1599, 1493, 1446, 1382, 1239, 1094, 890, 764, 696 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{22}H_{22}O$ 302.1671, Found 302.1671.

(*E*)-3-Butyl-4-(1-ethoxycarbonylethen-2-yl)-2,5-diphenylfuran (11):



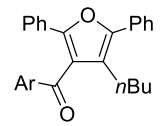
31 mg, 82% yield, Eluent, hexane/AcOEt = 30:1; Yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 0.98 (t, *J* = 7.2 Hz, 3H), 1.34 (t, *J* = 7.2 Hz, 3H), 1.48 (qt, *J* = 7.2, 7.2 Hz, 2H), 1.65-1.71 (m, 2H), 2.77-2.81 (m, 2H), 4.27 (q, *J* = 7.2 Hz, 2H), 6.29 (d, *J* = 16.2 Hz, 1H), 7.34 (tt, *J* = 7.2, 1.2 Hz, 1H), 7.39 (tt, *J* = 7.2, 1.2 Hz, 1H), 7.45 (dd, *J* = 7.8, 7.2 Hz, 2H), 7.47 (dd, *J* = 7.8, 7.2 Hz, 2H), 7.67 (dd, *J* = 7.8, 1.2 Hz, 4H), 7.84 (d, *J* = 16.2 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 13.8, 14.4, 22.8, 24.3, 32.0, 60.4, 118.7, 118.8, 122.1, 126.2, 127.6 (2C), 128.57, 128.64, 128.8, 130.3, 130.9, 136.5, 149.3, 153.6, 167.4; IR (ATR): 3053, 2956, 2932, 2867, 1710, 1632, 1490, 1444, 1276, 1178, 1032, 912, 763, 692 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{25}H_{26}O_3$ [M] 374.1882, Found 374.1882.

(4-Butyl-2,5-diphenylfur-3-yl)(phenyl)methanol (12):



35 mg, 91% yield, Eluent, hexane/AcOEt = 10:1; Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 0.77 (t, *J* = 7.2 Hz, 3H), 1.01-1.09 (m, 1H), 1.12-1.26 (m, 2H), 1.40-1.49 (m, 1H), 2.20 (s, 1H), 2.33-2.40 (m, 1H), 2.51-2.57 (m, 1H), 6.24 (s, 1H), 7.25-7.29 (m, 2H), 7.32 (tt, *J* = 7.2, 1.2 Hz, 1H), 7.35 (dd, *J* = 7.8, 7.2 Hz, 2H), 7.40 (dd, *J* =7.8, 7.2 Hz, 2H), 7.41 (dd, *J* = 7.8, 7.2 Hz, 2H), 7.49 (d, *J* = 7.8 Hz, 2H), 7.67 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.68 (dd, *J* = 7.8, 1.2 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 13.7, 23.0, 24.3, 31.8, 67.8, 123.3, 124.5, 125.5, 125.9, 127.0, 127.2, 128.0, 128.2, 128.56, 128.58 (2C), 130.7, 131.5, 142.4, 148.6, 150.0; IR (ATR): 3446, 3061, 2953, 2926, 2872, 1600, 1491, 1448, 1069, 1018, 910, 765, 733, 692 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{27}H_{26}O_2$ [M] 382.1933, Found 382.1933.

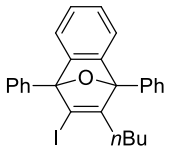
4-Butyl-3-((4-methoxyphenyl)carbonyl)-2,5-diphenylfuran (13):



Ar = 4-MeOC$_6$H$_4$

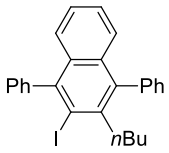
34 mg, 83% yield, Eluent, hexane/AcOEt = 20:1; Pale yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 0.80 (t, J = 7.2 Hz, 3H), 1.28 (qt, J = 7.2, 7.2 Hz, 2H), 1.45-1.51 (m, 2H), 2.62-2.66 (m, 2H), 3.83 (s, 3H), 6.85 (d, J = 9.0 Hz, 2H), 7.19 (tt, J = 7.2, 1.2 Hz, 1H), 7.24 (dd, J = 7.8, 7.2 Hz, 2H), 7.34 (tt, J = 7.2, 1.2 Hz, 1H), 7.47 (dd, J = 7.2, 7.2 Hz, 2H), 7.51 (dd, J = 7.8, 1.2 Hz, 2H), 7.73 (dd, J = 7.8, 1.2 Hz, 2H), 7.91 (d, J = 9.0 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 13.7, 22.6, 24.0, 32.3, 55.4, 113.8, 123.5, 123.7, 126.0, 126.3, 127.5, 128.0, 128.4, 128.7, 129.8, 130.7, 131.0, 132.2, 148.5, 150.4, 163.9, 192.7; IR (ATR): 3060, 2958, 2931, 2869, 1653, 1598, 1488, 1314, 1254, 1171, 1028, 904, 794, 692 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{28}H_{26}O_3$ 410.1882, Found 410.1882.

3-Butyl-2-iodo-1,4-diphenyl-1,4-dihydro-1,4-epoxynaphthalene (15):



22 mg, 91% yield, Eluent, hexane/AcOEt = 30:1; Yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 0.74 (t, J = 7.2 Hz, 3H), 0.94-1.07 (m, 2H), 1.09-1.18 (m, 2H), 2.24-2.37 (m, 2H), 7.04 (dd, J = 4.2, 4.2 Hz, 1H), 7.06 (dd, J = 4.2, 4.2 Hz, 1H), 7.41-7.47 (m, 4H), 7.51-7.55 (m, 4H), 7.82 (d, J = 7.2 Hz, 2H), 7.91 (d, J = 7.8 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 13.7, 22.2, 28.5, 30.0, 92.9, 94.1, 111.3, 120.0, 120.5, 125.0, 125.2, 126.5, 127.9, 128.0, 128.2, 128.4, 128.5, 134.5, 134.9, 149.8, 150.0, 161.8; IR (ATR): 3065, 2955, 2928, 2864, 1605, 1452, 1304, 1002, 907, 742, 701 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{26}H_{23}IO$ 478.0794, Found 478.0794.

3-Butyl-2-iodo-1,4-diphenylnaphthalene (16):

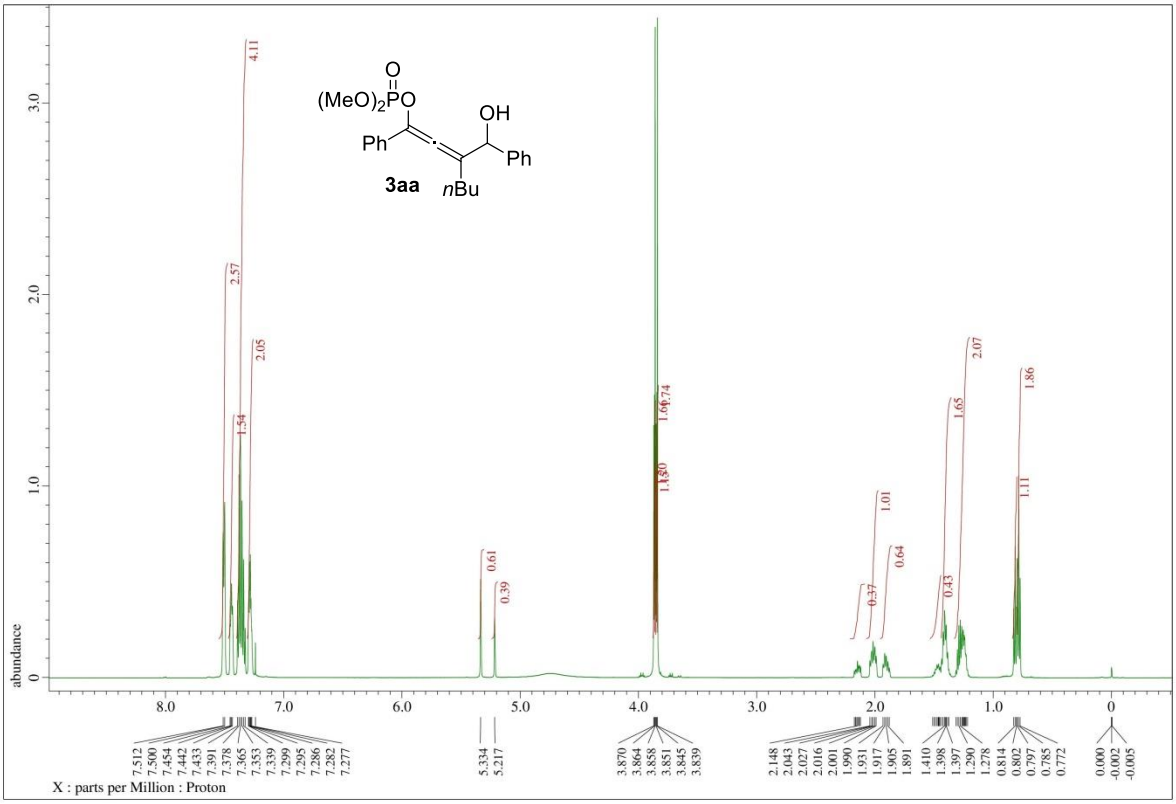


61% NMR yield, 27 mg, abt. 95% purity, Eluent, hexane (analytically pure product was isolated after purification by recrystallization from hexane, 1.6 mg, 3%); White solid; ^{1}H NMR (600 MHz, $CDCl_3$) δ 0.76 (t, J = 7.2 Hz, 3H), 1.22 (qt, J = 7.2, 7.2 Hz, 2H), 1.47-1.54 (m, 2H), 2.75-2.79 (m, 2H), 7.22 (ddd, J = 8.4, 8.4, 1.8 Hz, 1H), 7.26-7.34 (m, 7H), 7.46 (tt, J = 7.2, 1.2 Hz, 1H), 7.49-7.53 (m, 3H), 7.55 (dd, J = 7.2, 7.2 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 13.6, 22.8, 32.4, 39.7, 107.5, 125.6, 126.0, 126.6, 127.3, 127.5, 127.6, 128.3, 128.4, 130.0, 130.1, 131.6, 132.8, 138.4, 139.7, 140.0, 145.4, 145.5; IR (ATR): 3058, 2957, 2923, 2853, 1600, 1544, 1493, 1441, 1372, 1262, 1069, 1029, 909, 801, 752, 699 cm^{-1}; HRMS (FD+) *m/z*: [M] Calcd for $C_{26}H_{23}I$ 462.0844, Found 462.0844; mp. 161.0-164.0 °C.

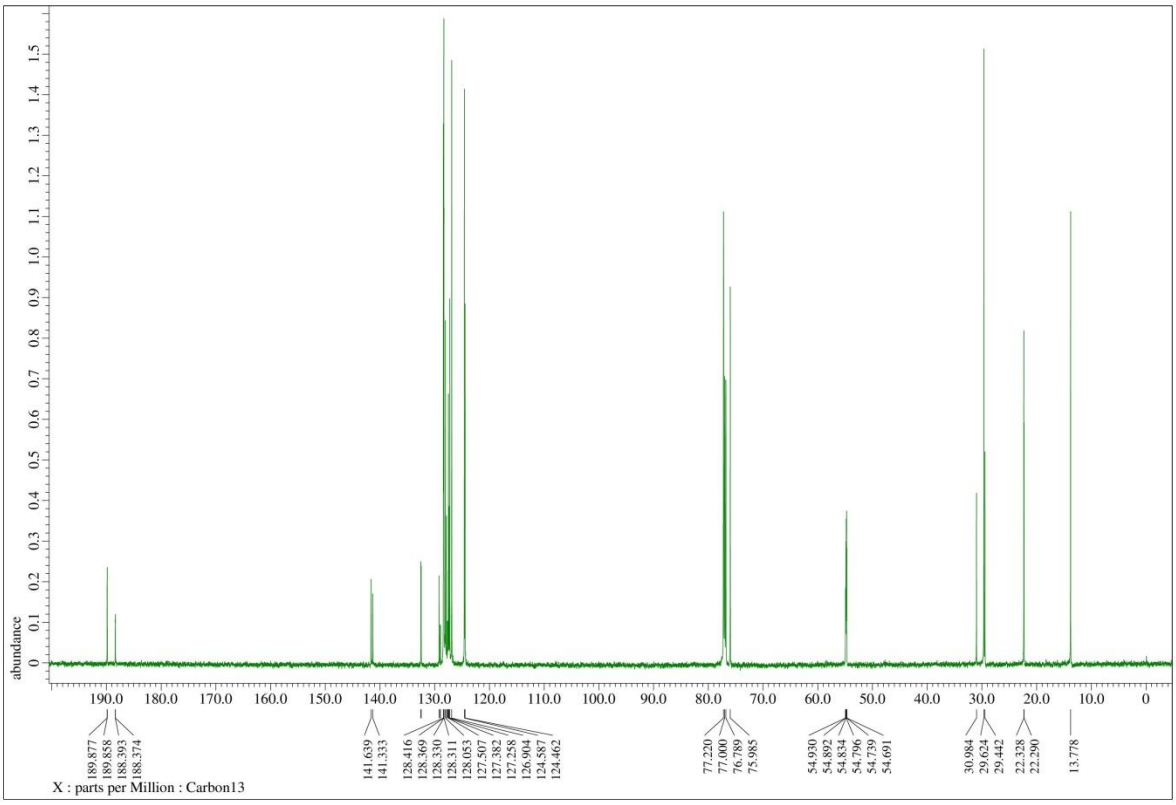
References

S1. Kondoh, A.; Iino, A.; Ishikawa, S.; Aoki, T.; Terada, M. *Chem. Eur. J.* **2018**, *24*, 15246-15253.

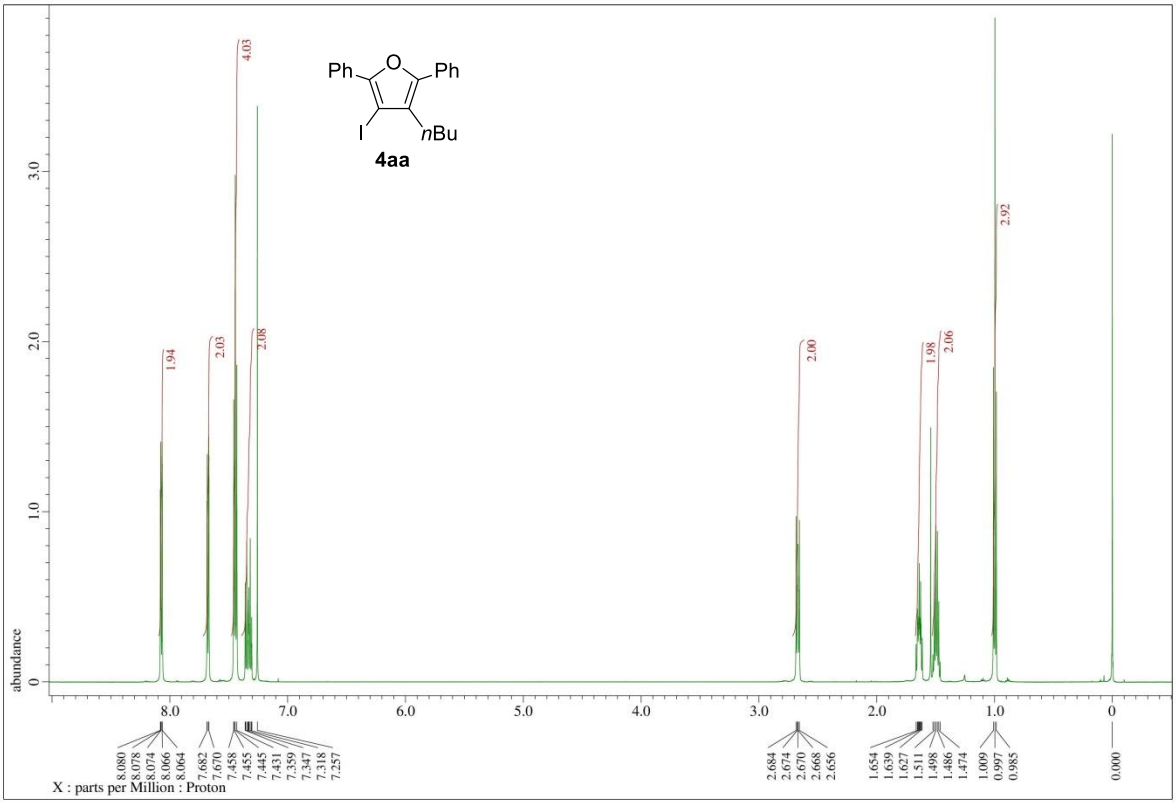
S2. Xing, Y. D.; Huang, N. Z. *J. Org. Chem.* **1982**, *47*, 140-142.



600 MHz, $CDCl_3$



150 MHz, $CDCl_3$



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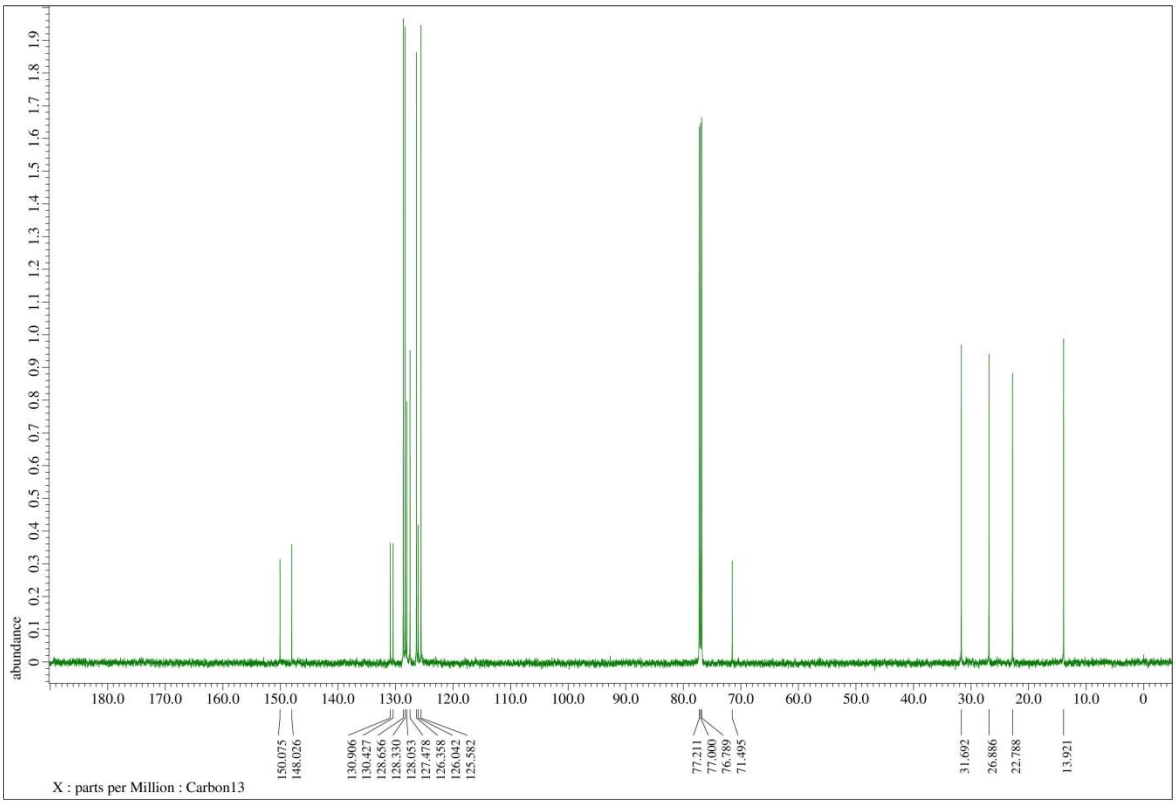

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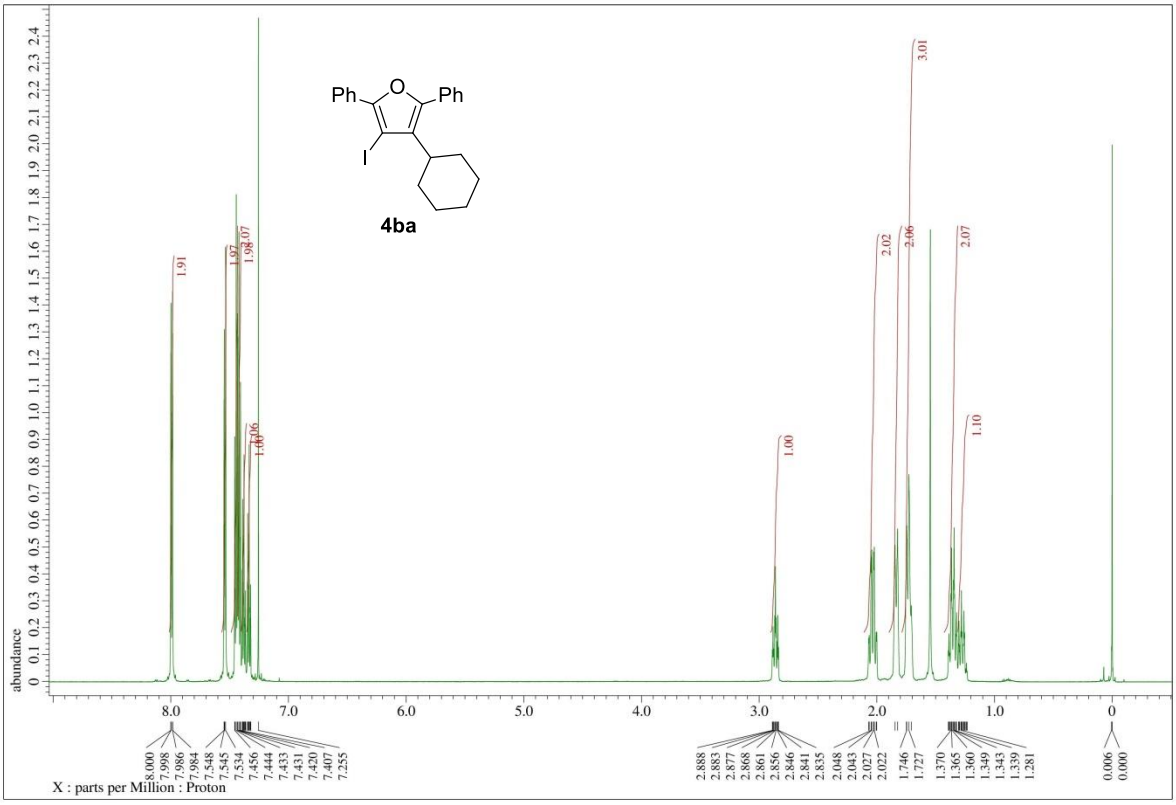

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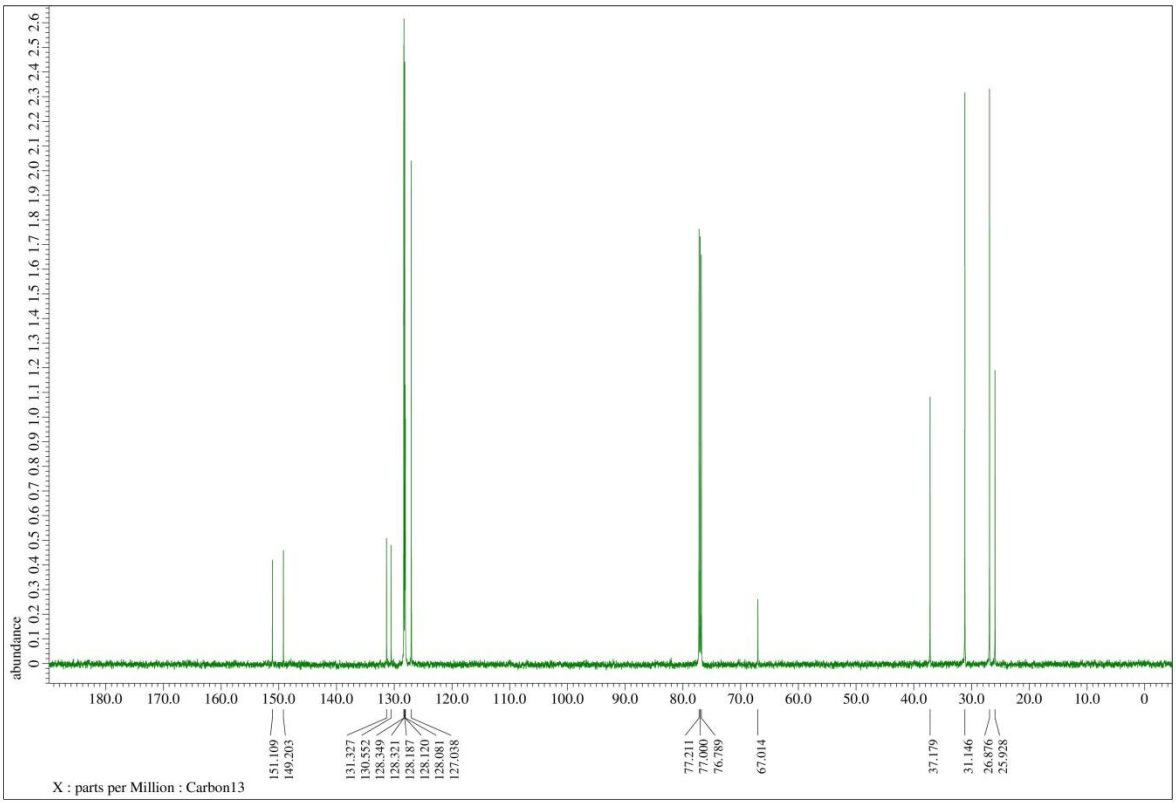

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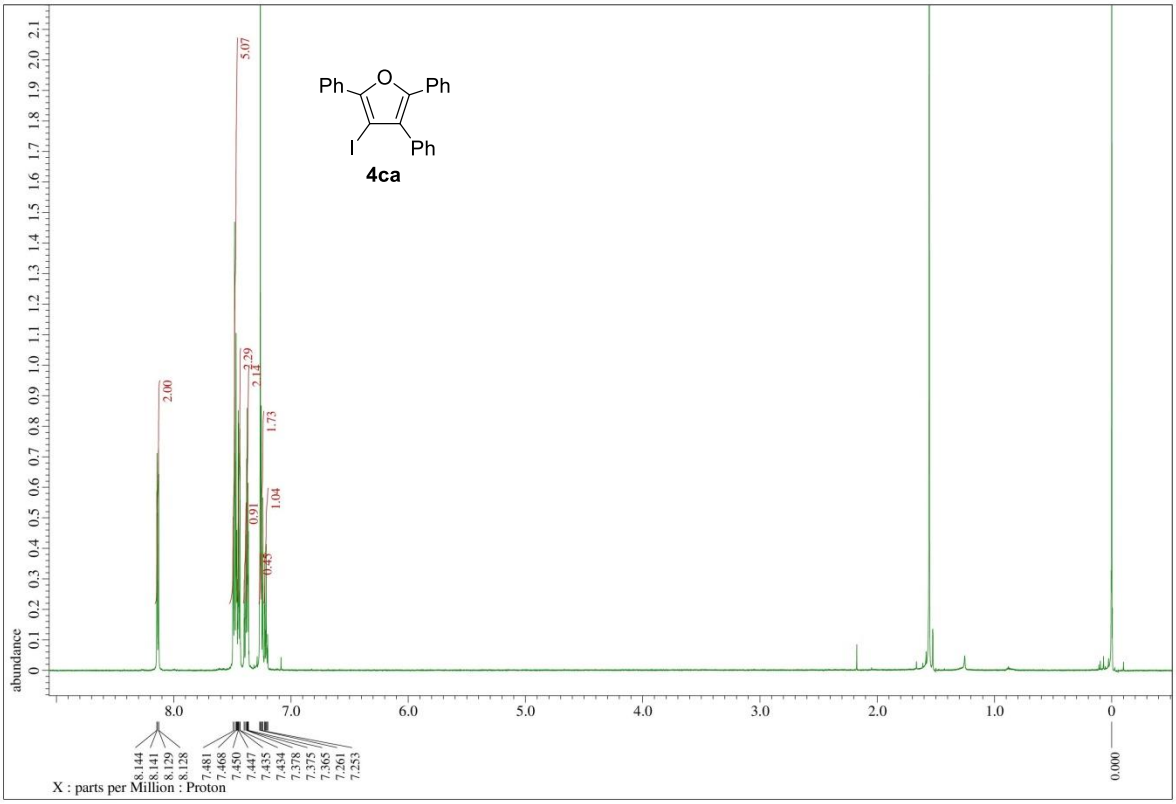

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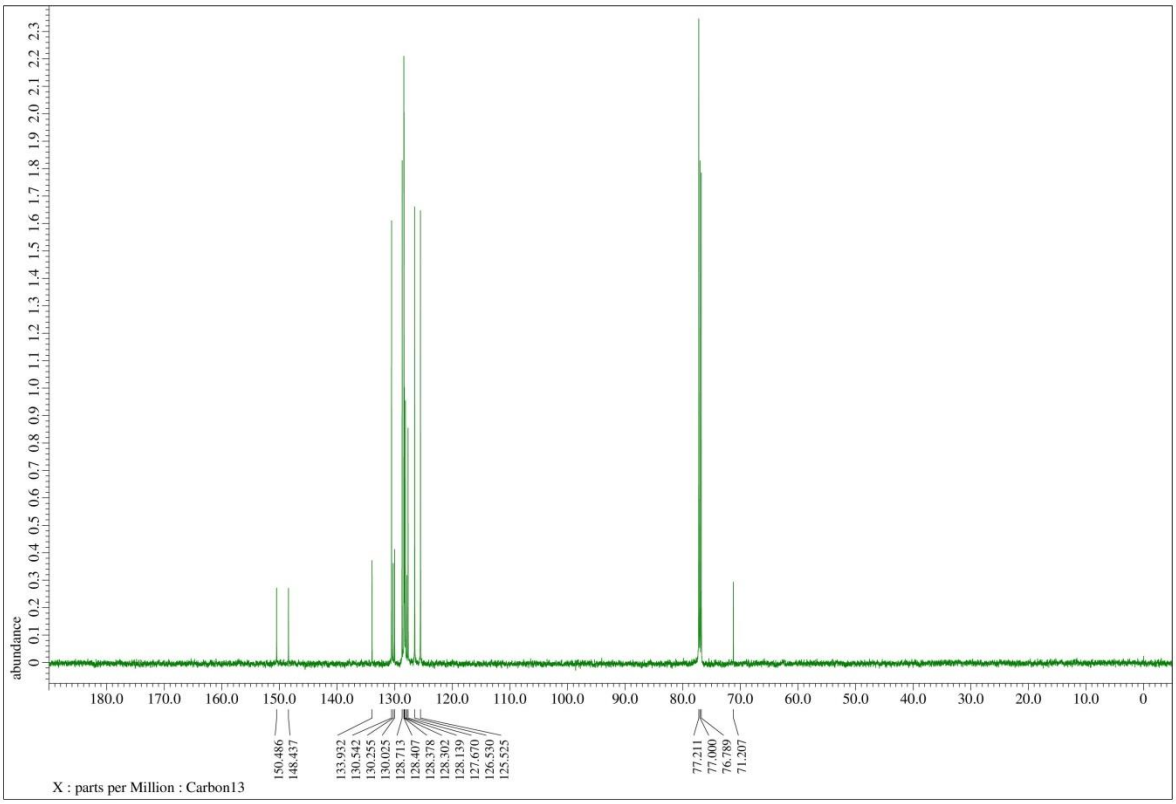

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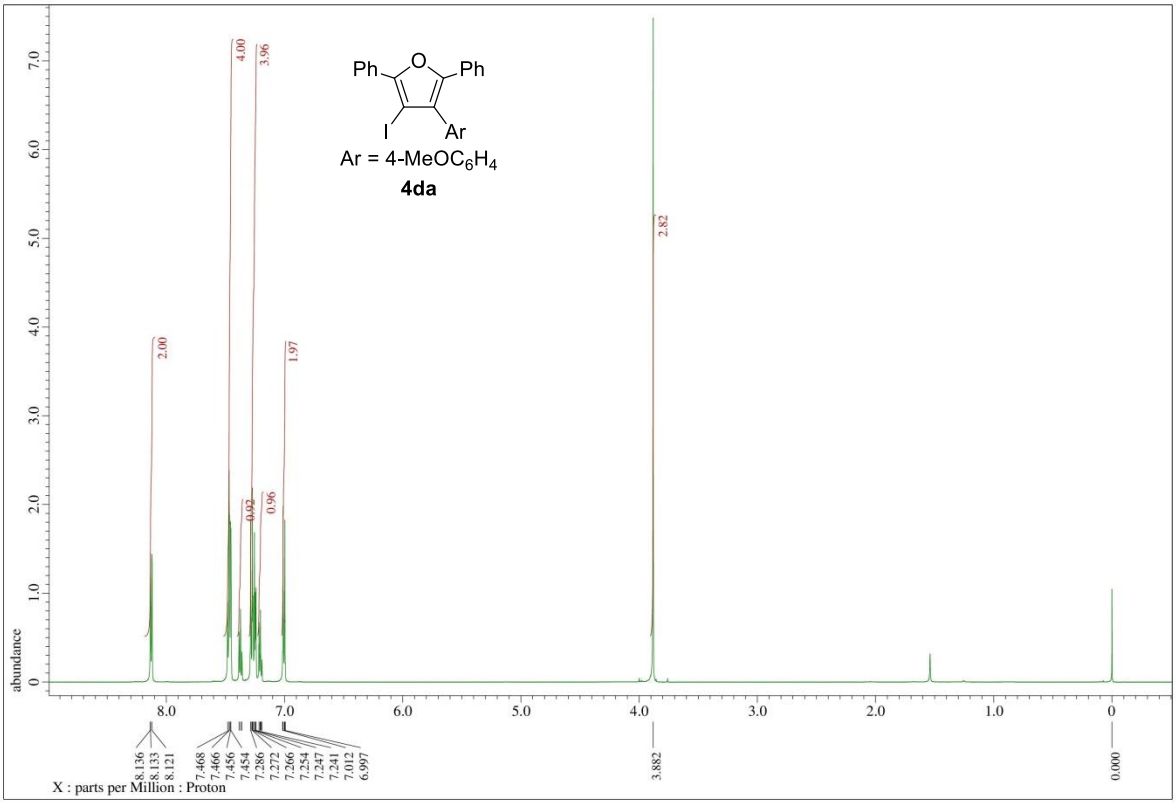

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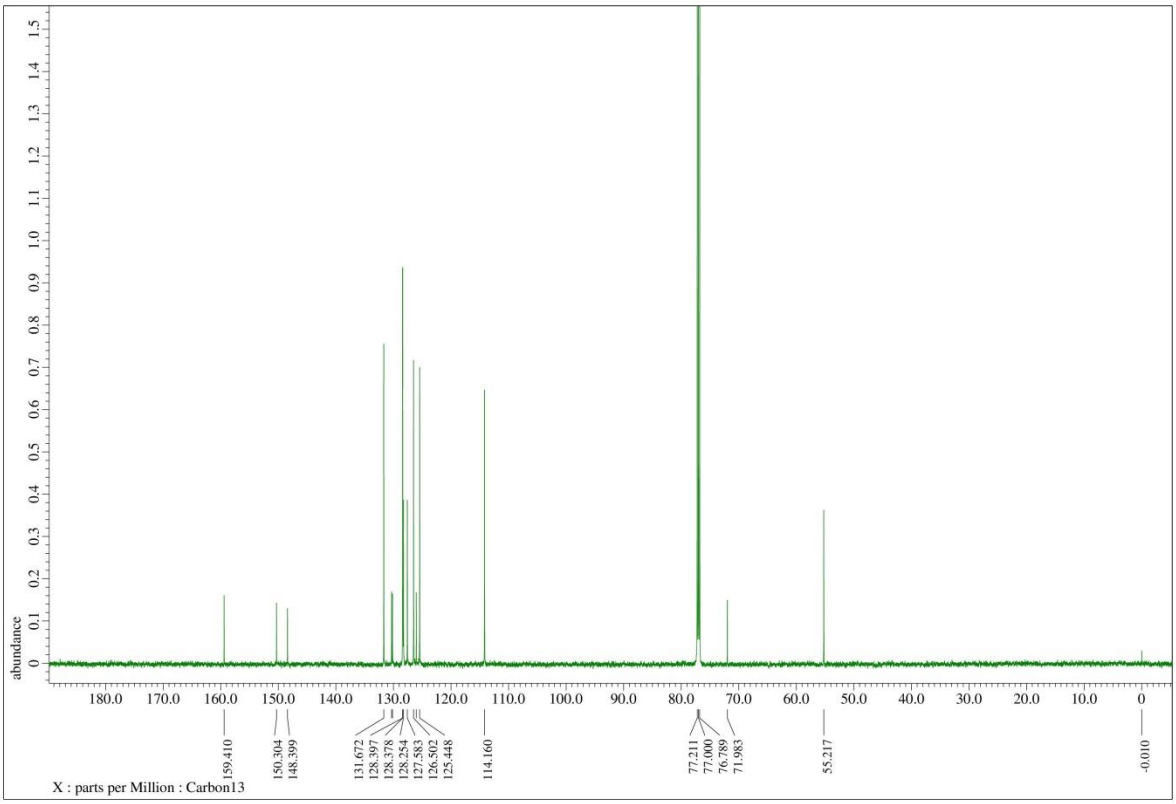

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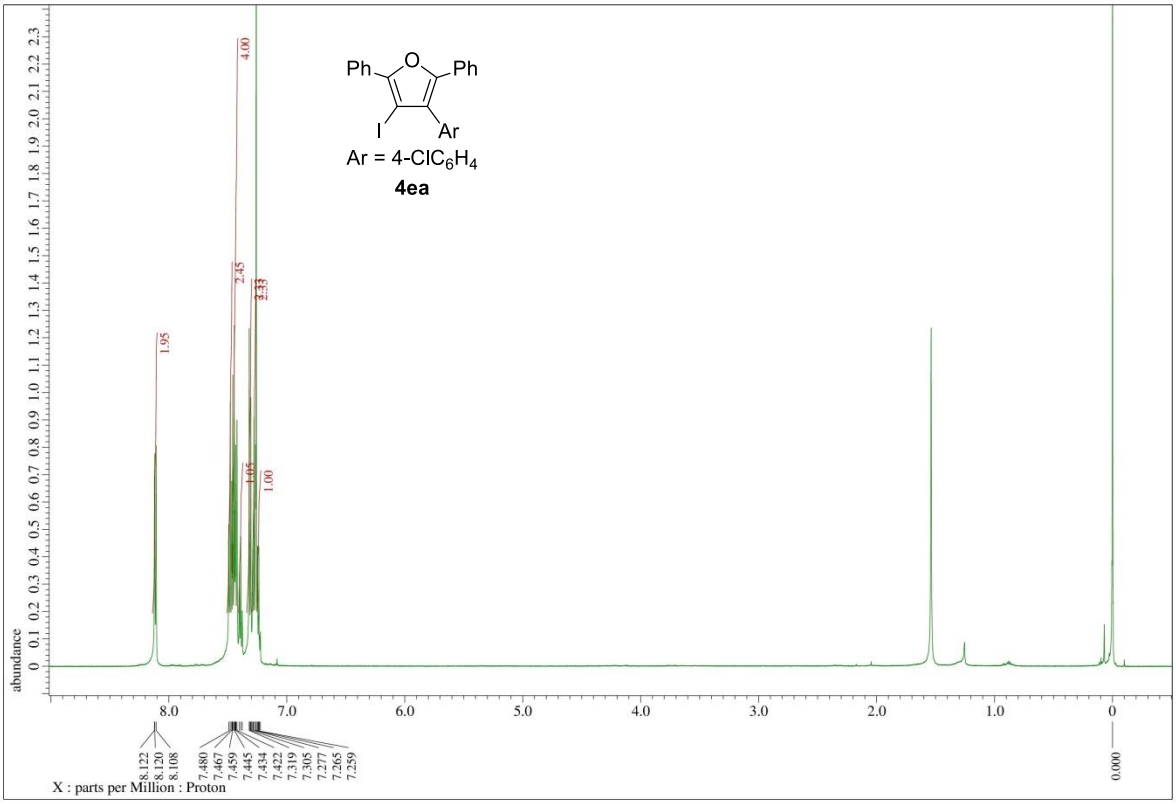

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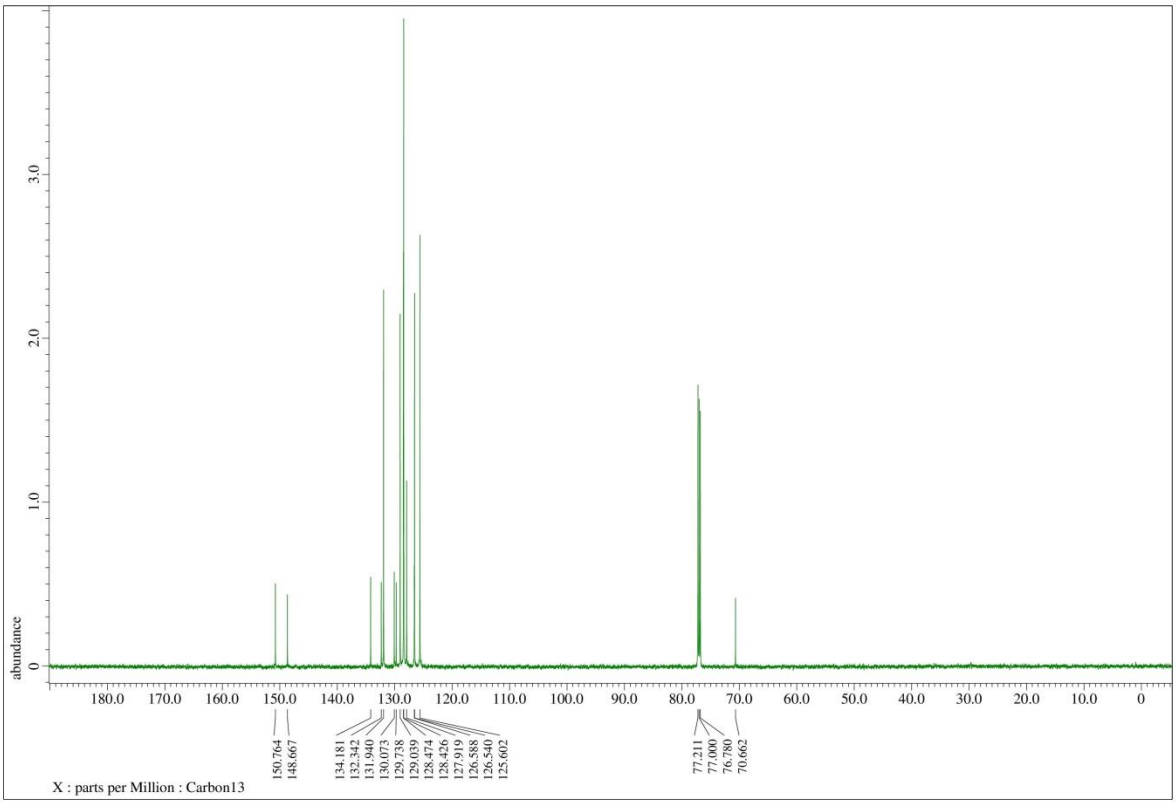

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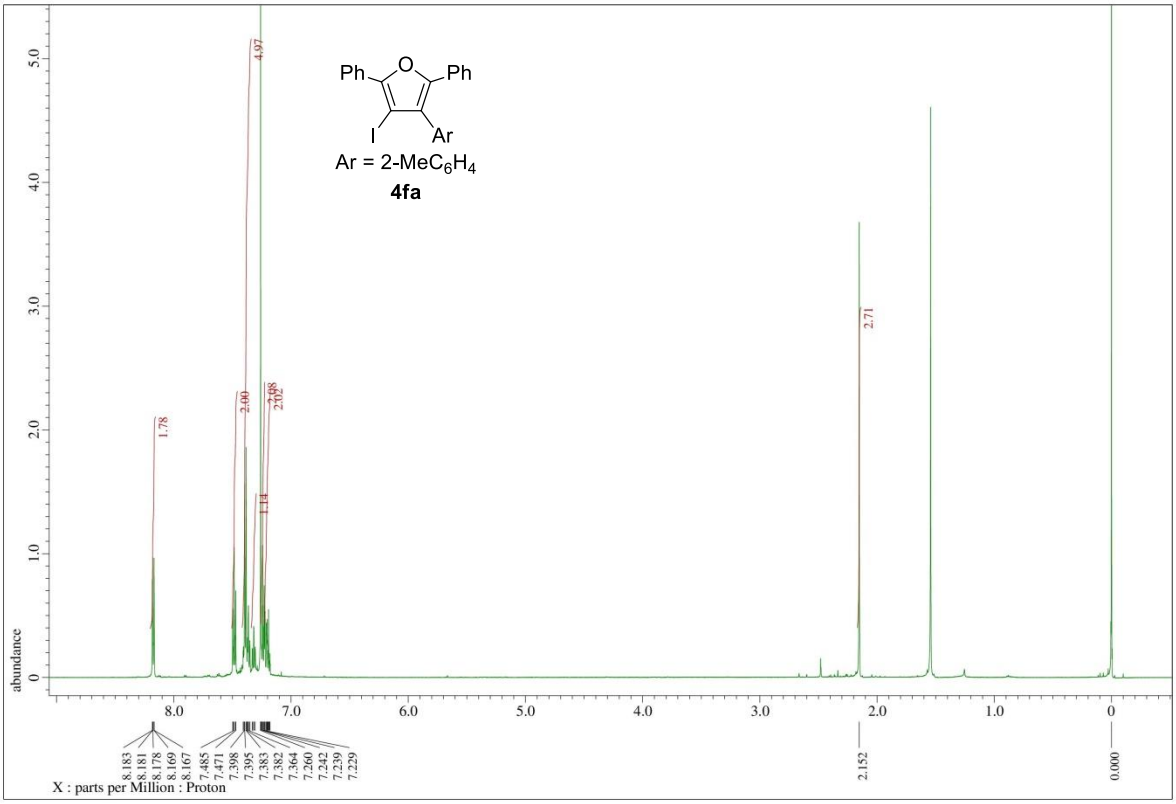

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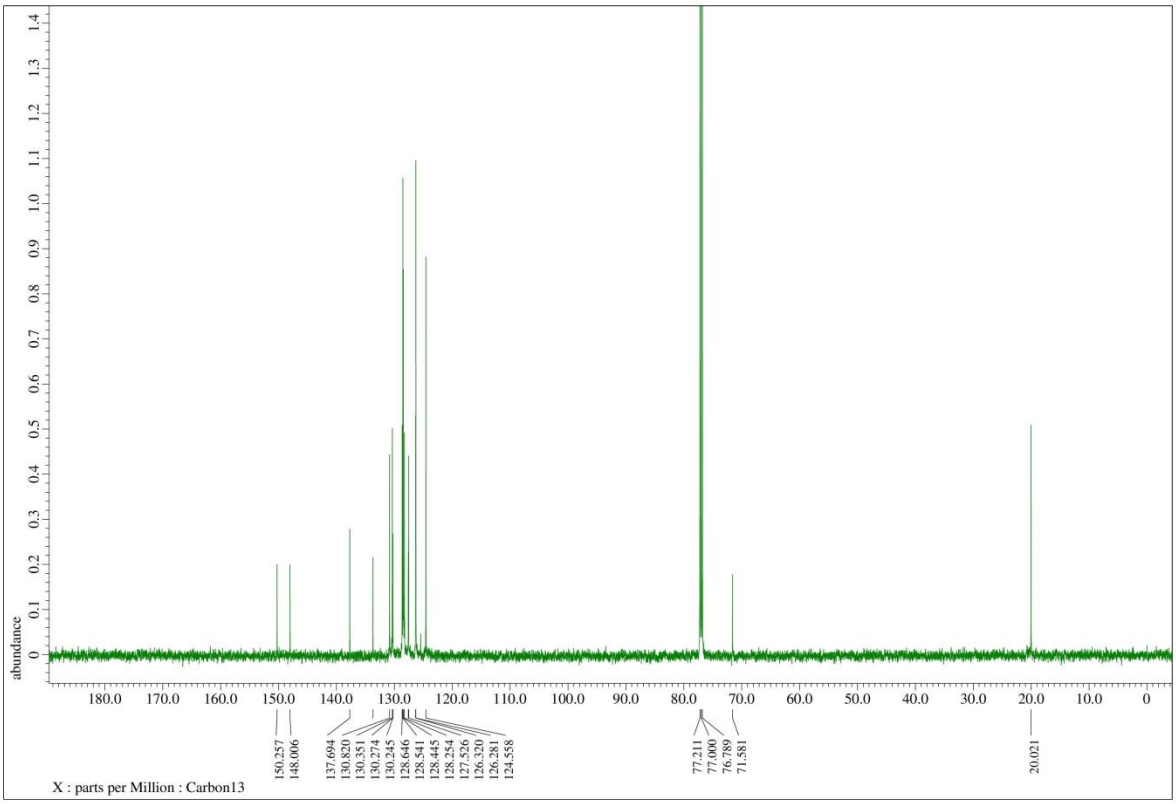

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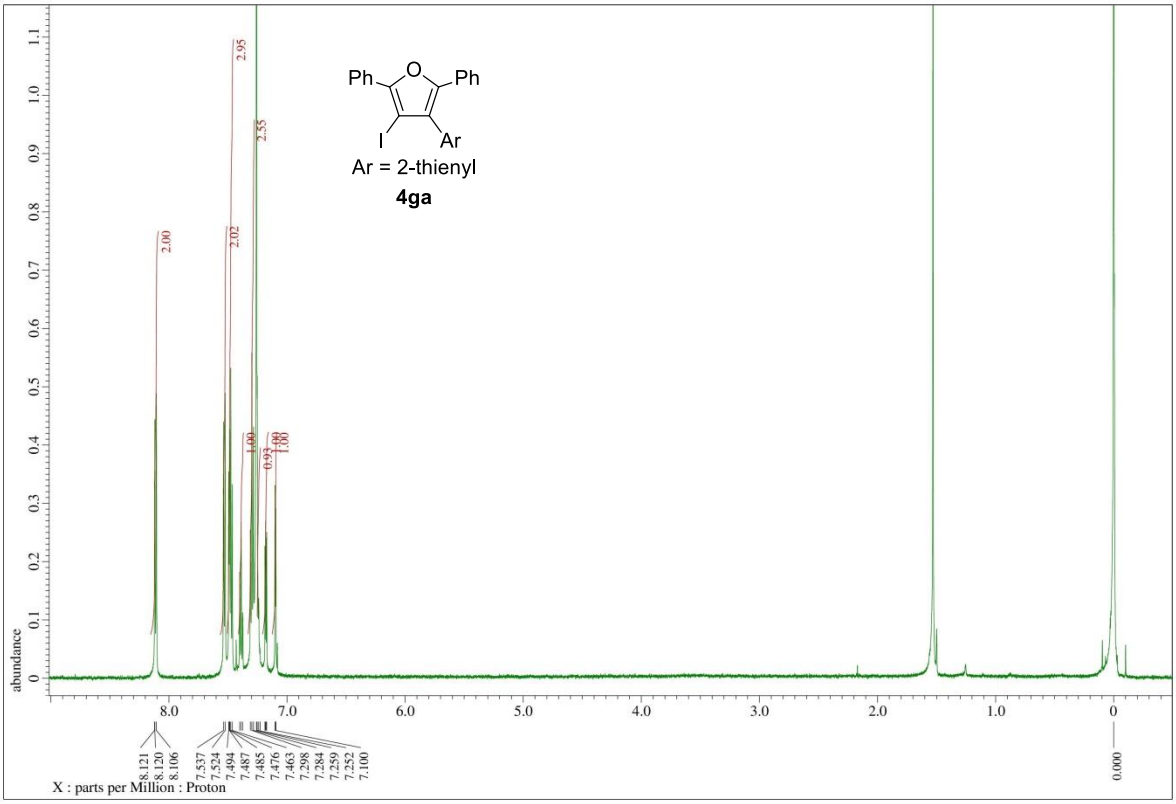

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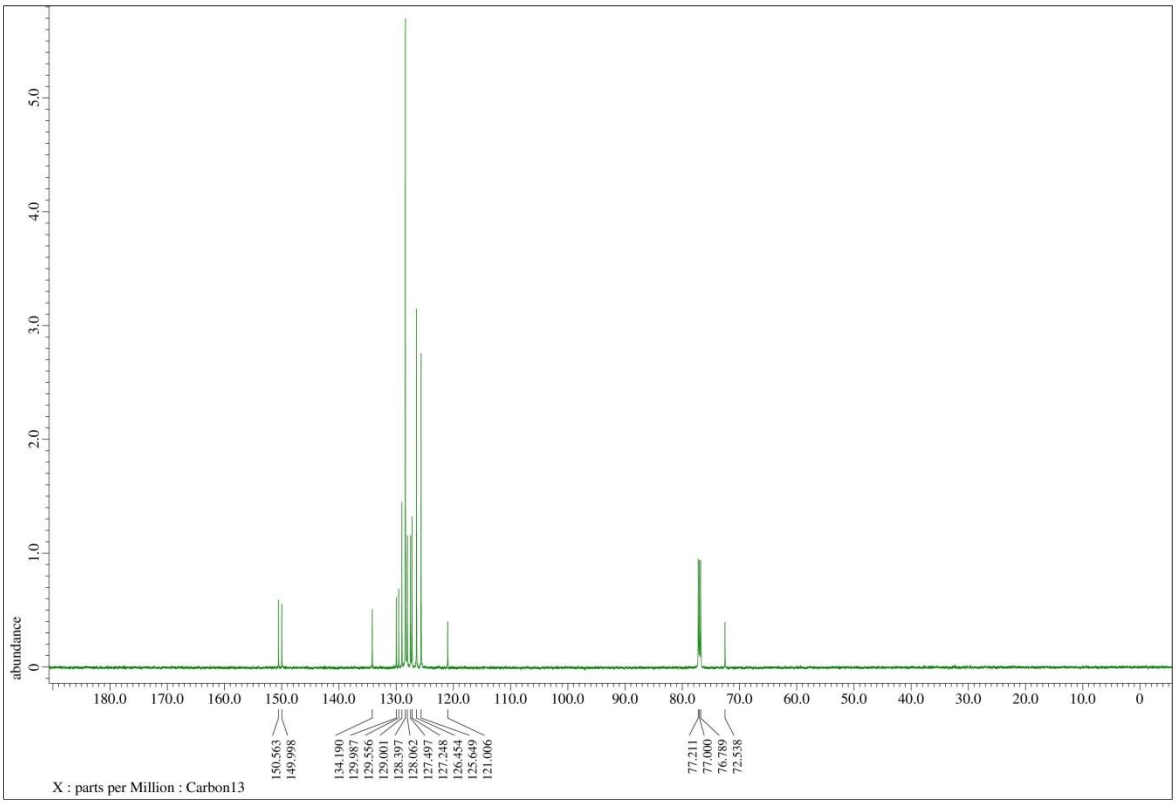

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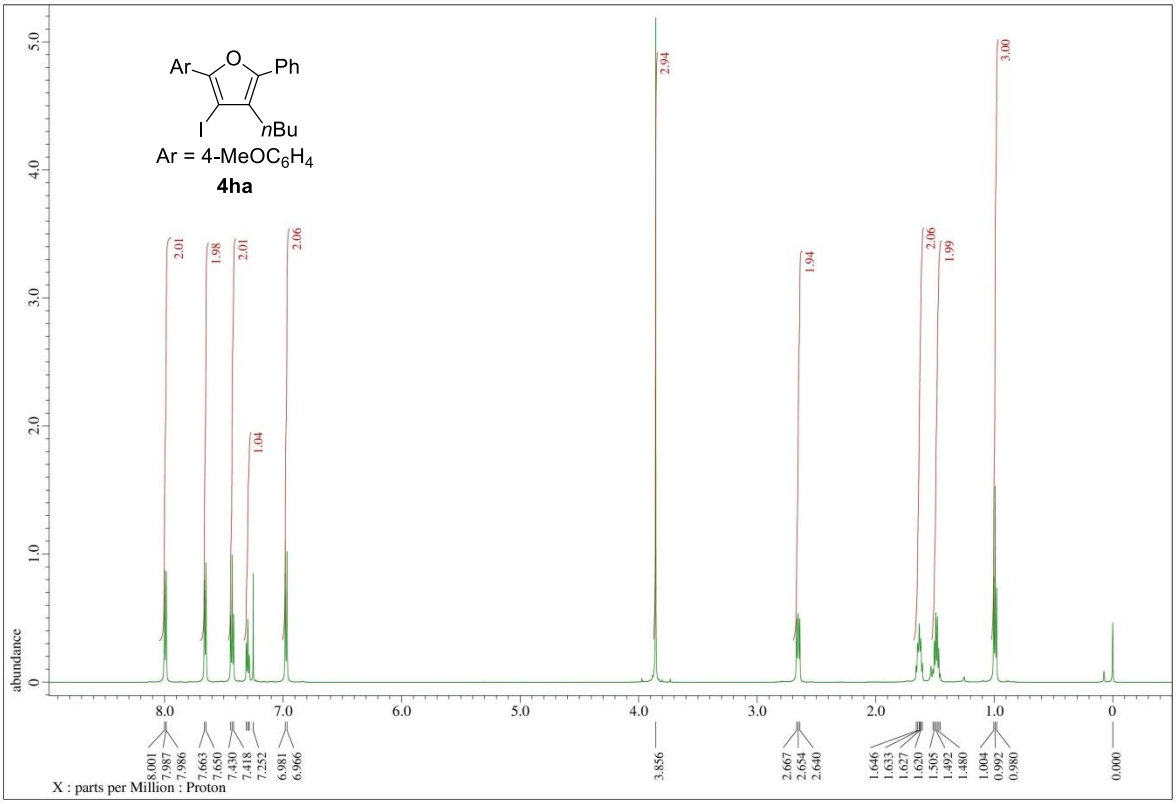

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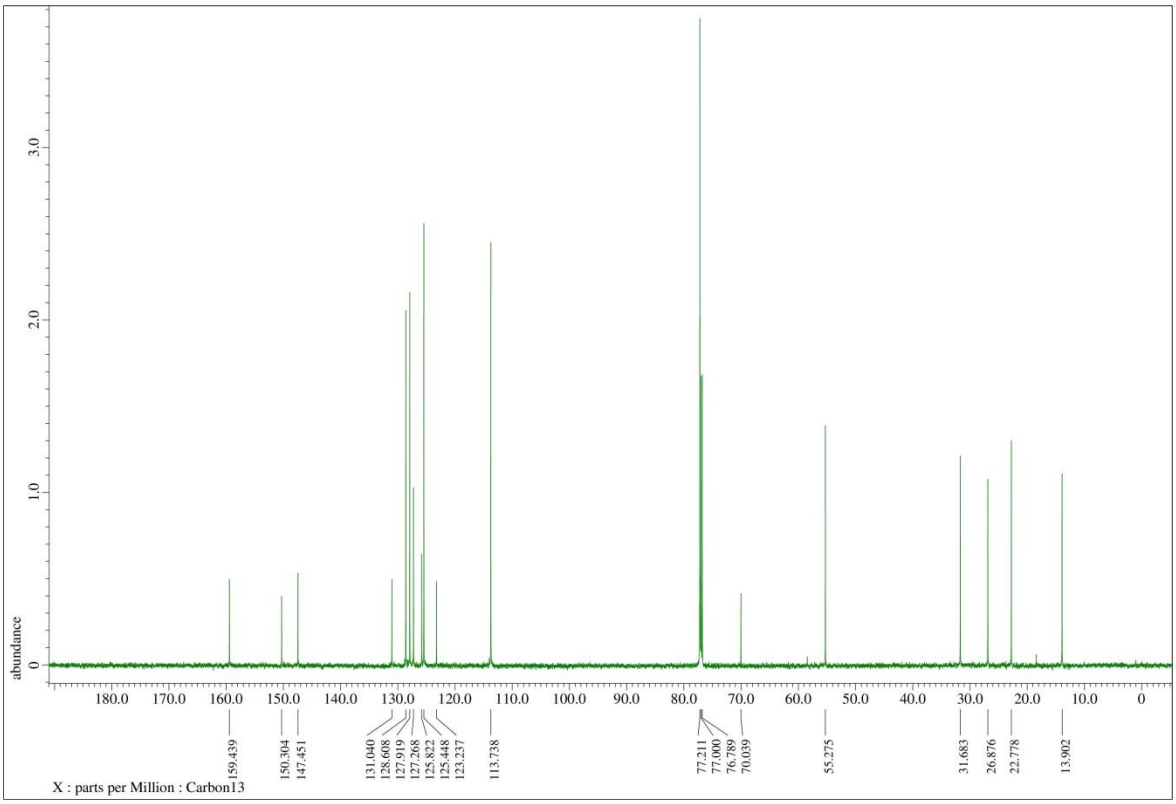

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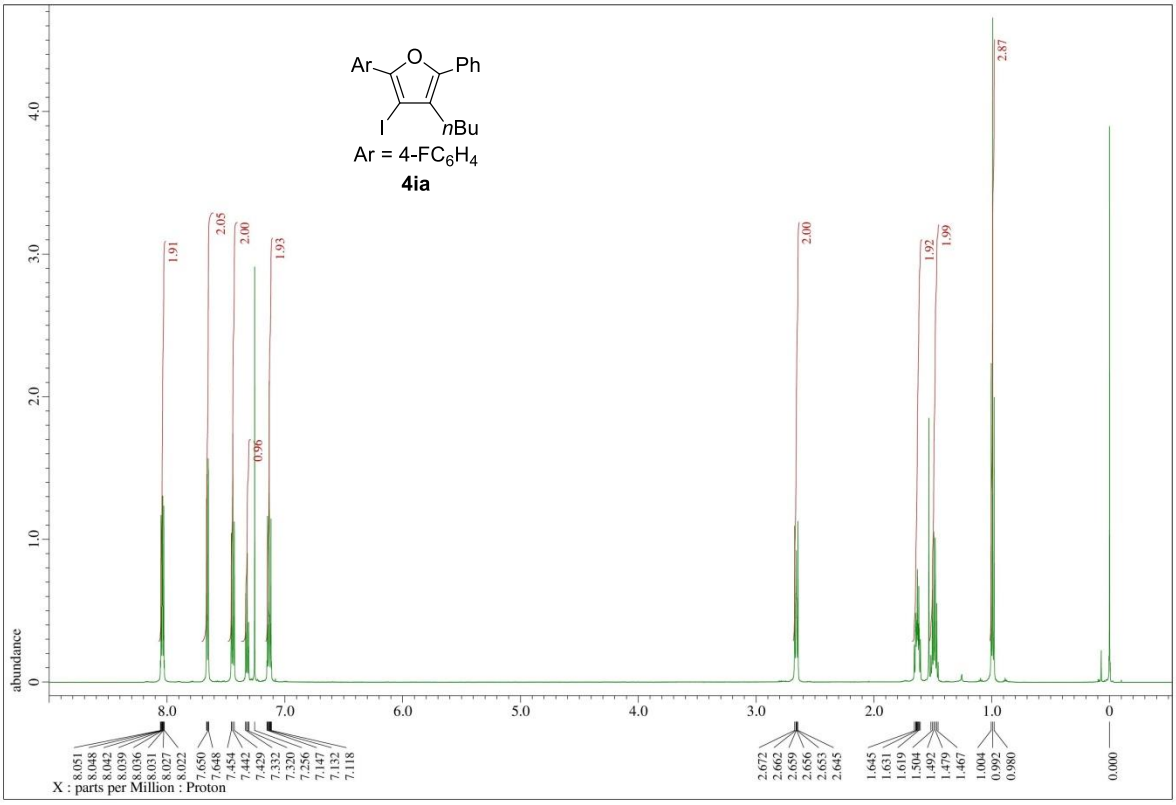

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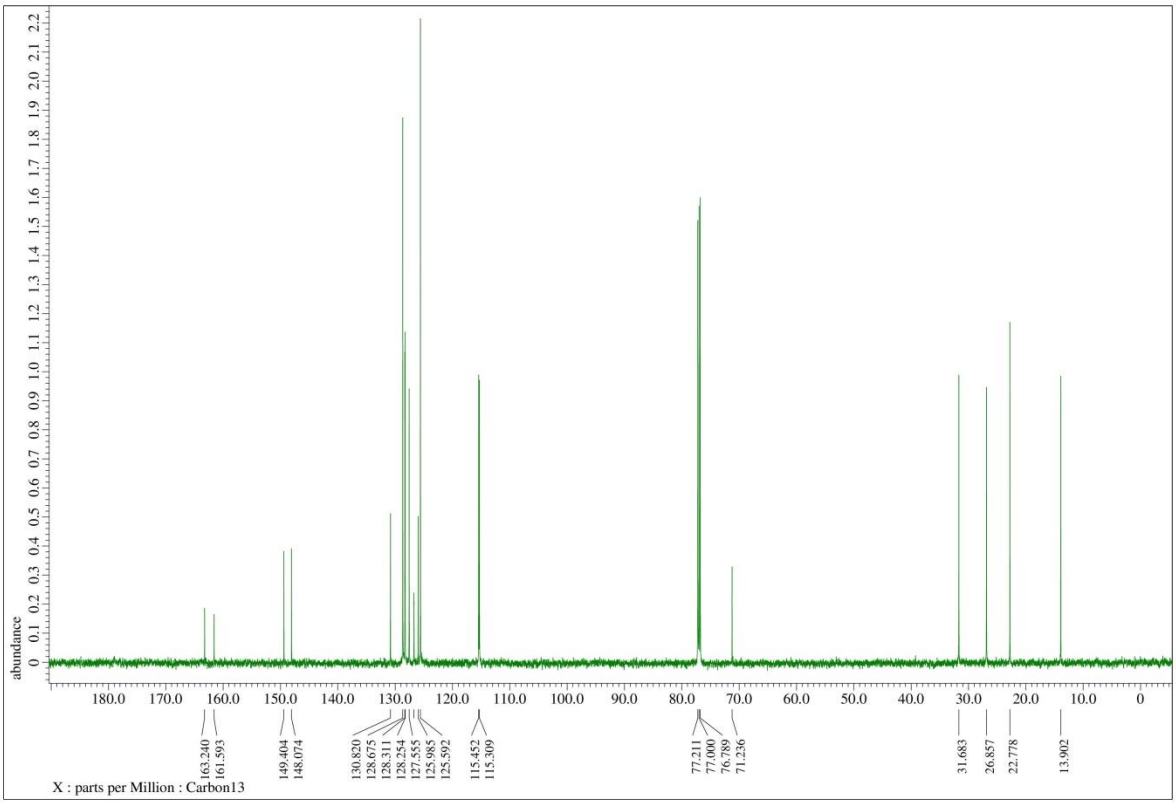

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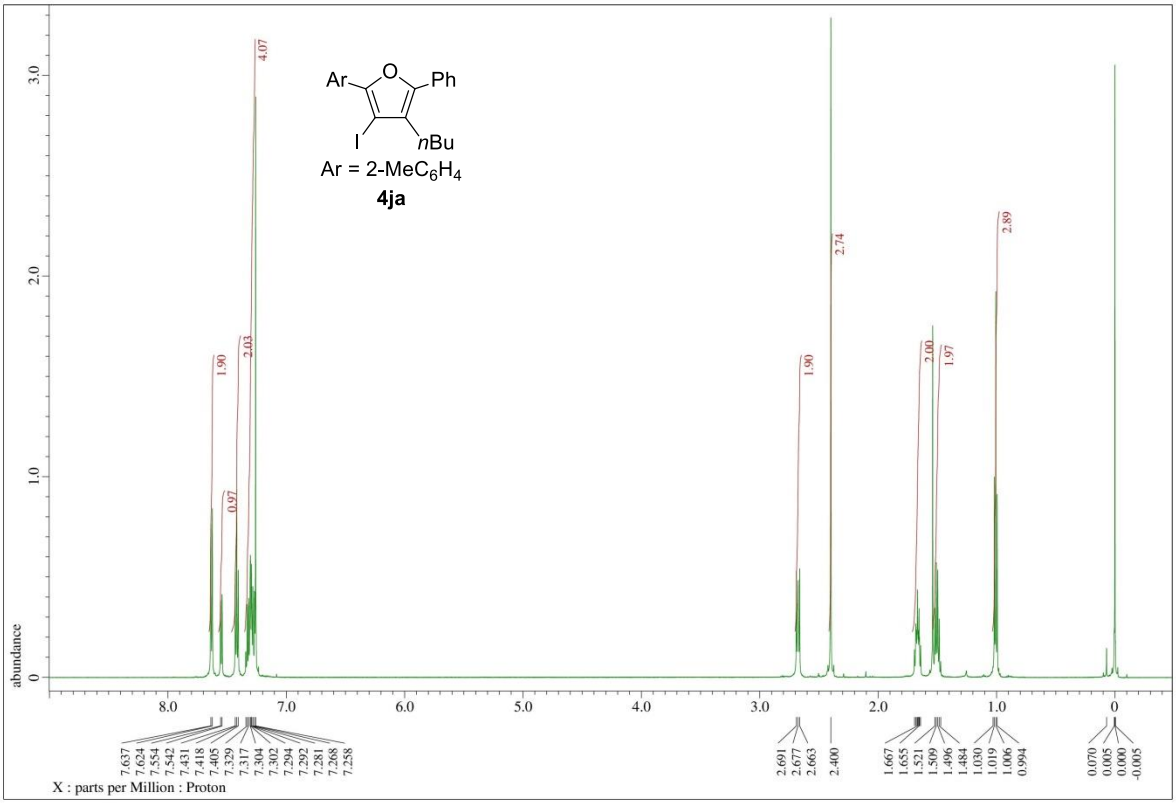

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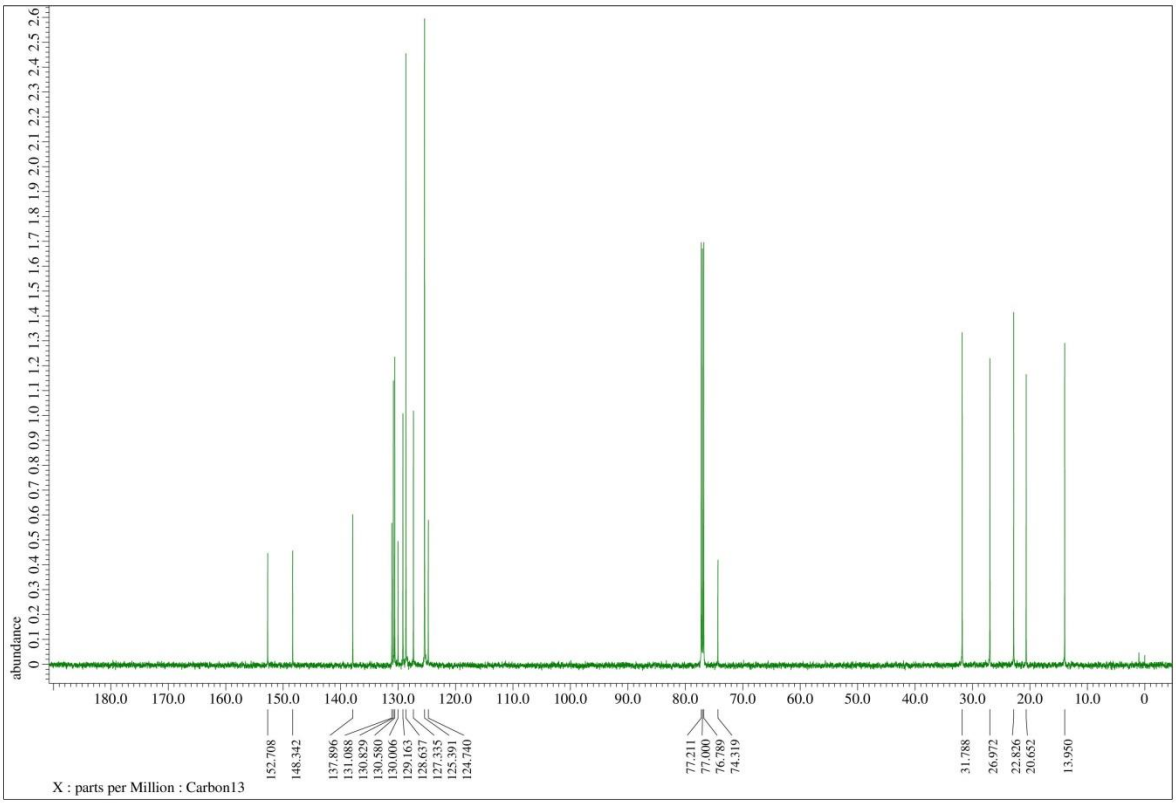

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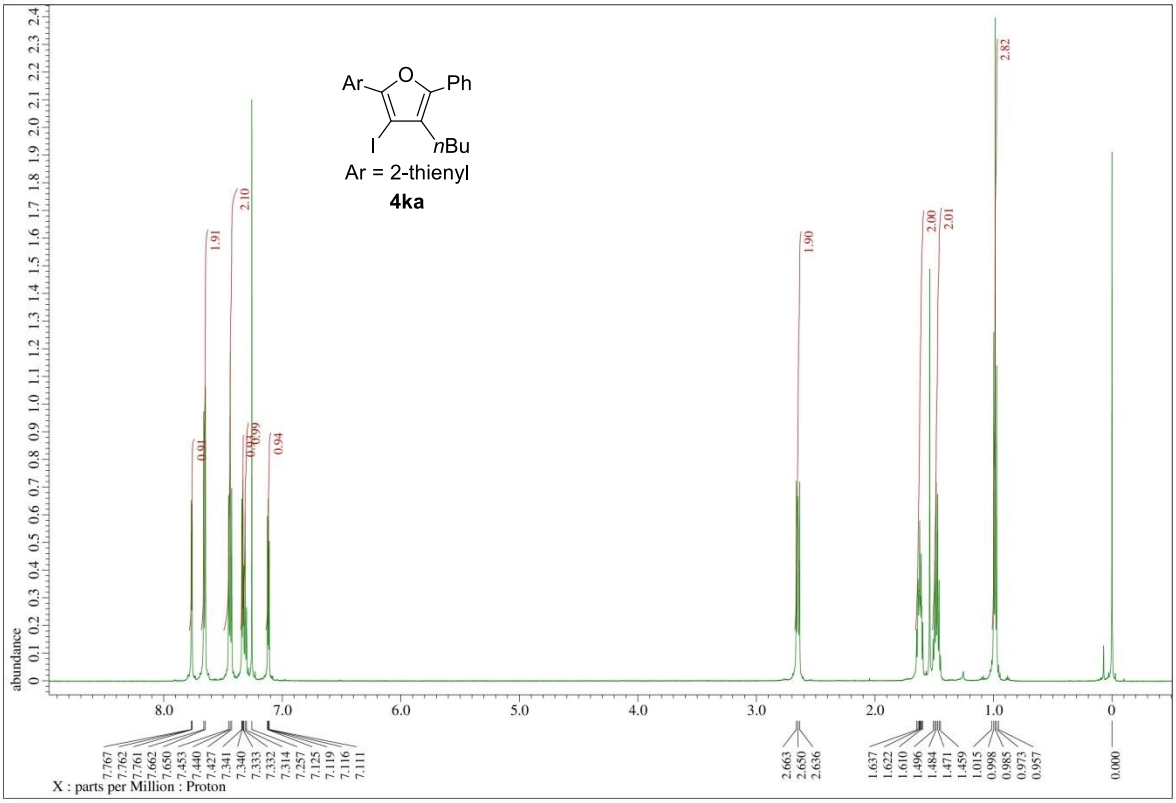

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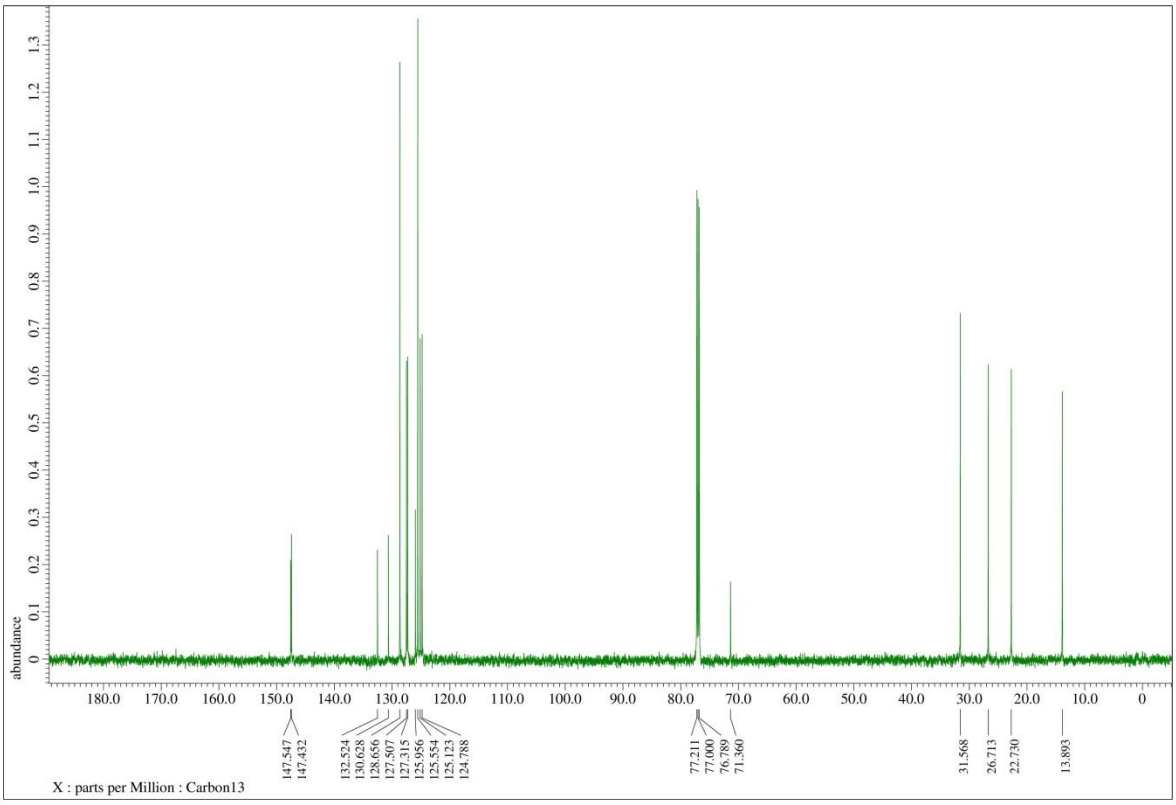

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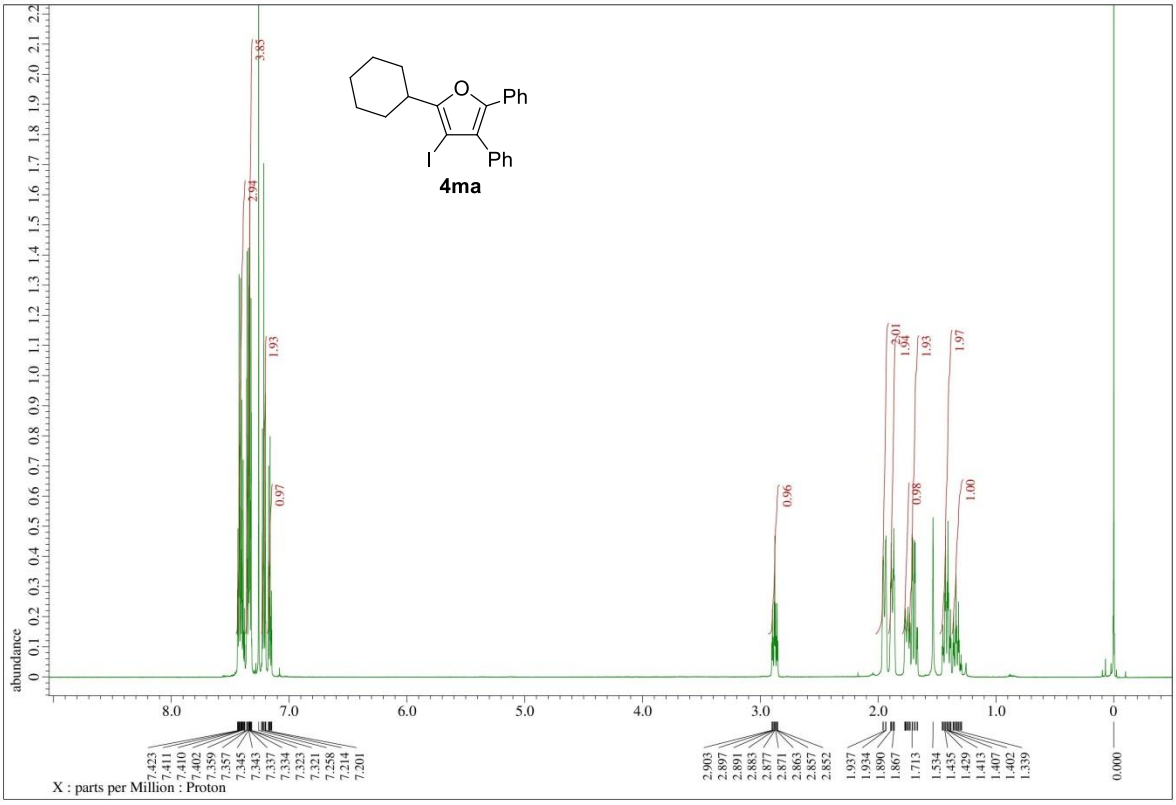

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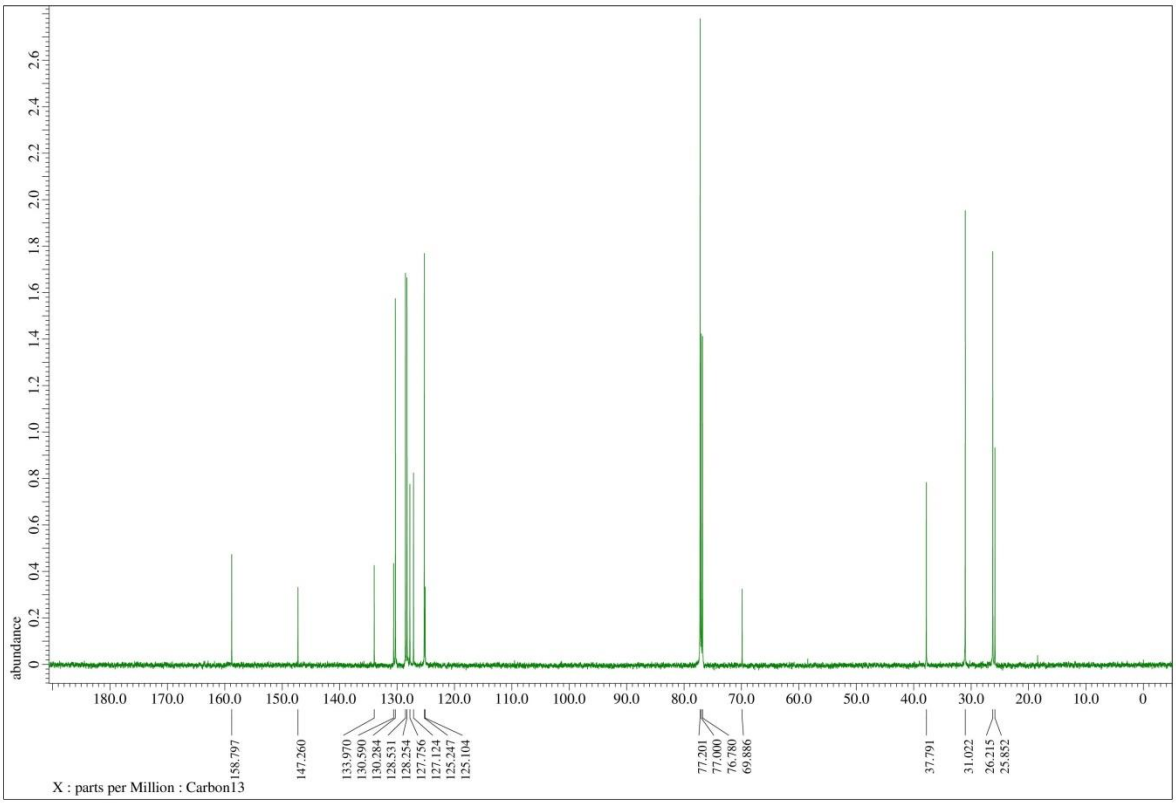

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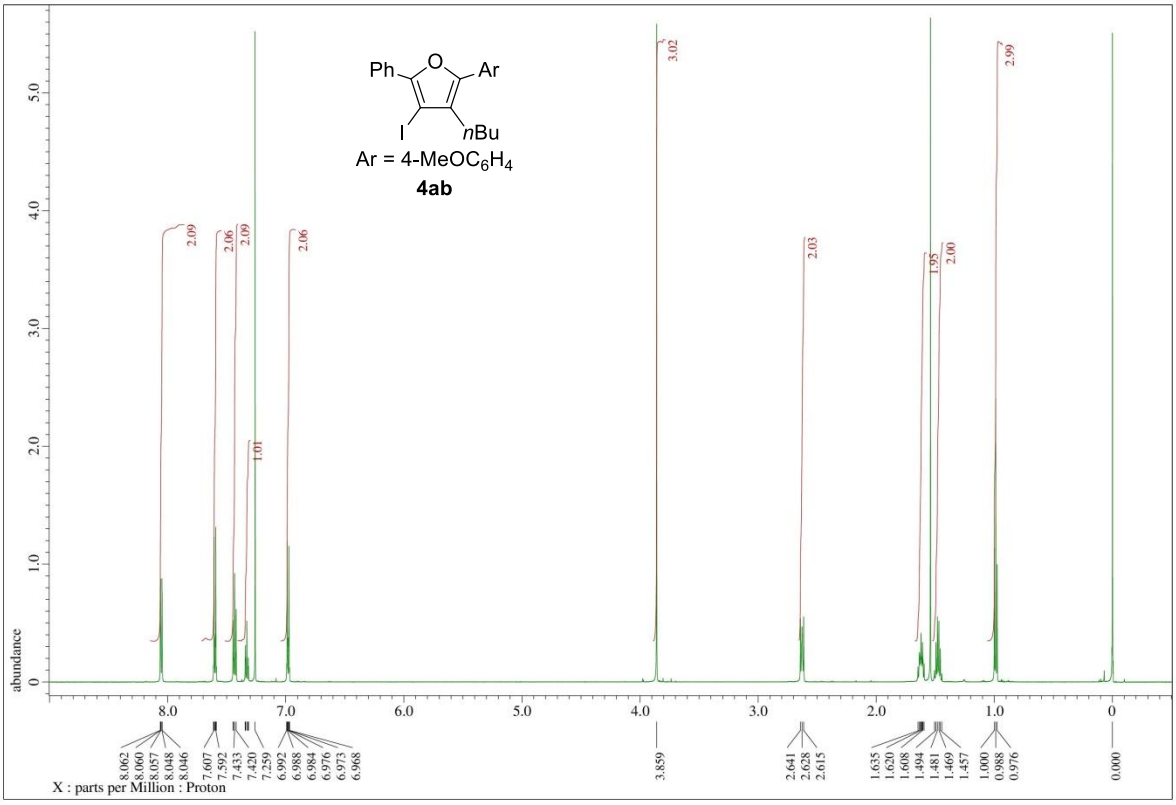

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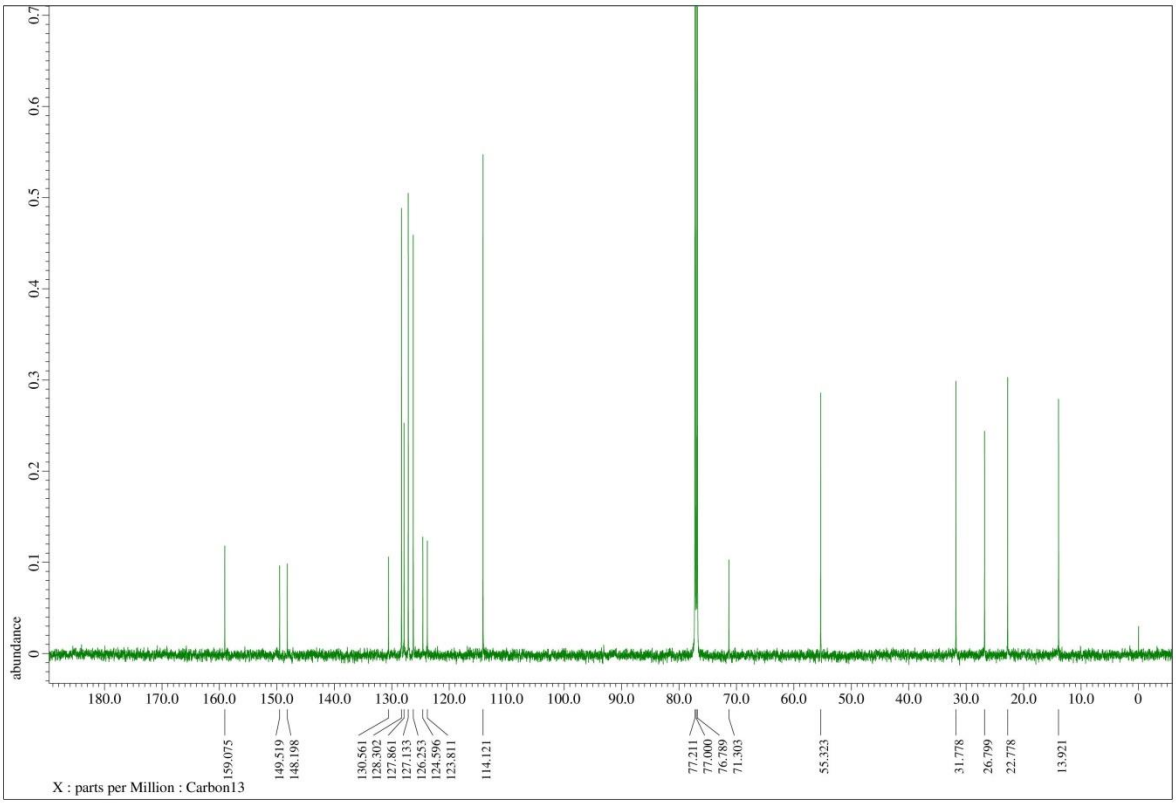

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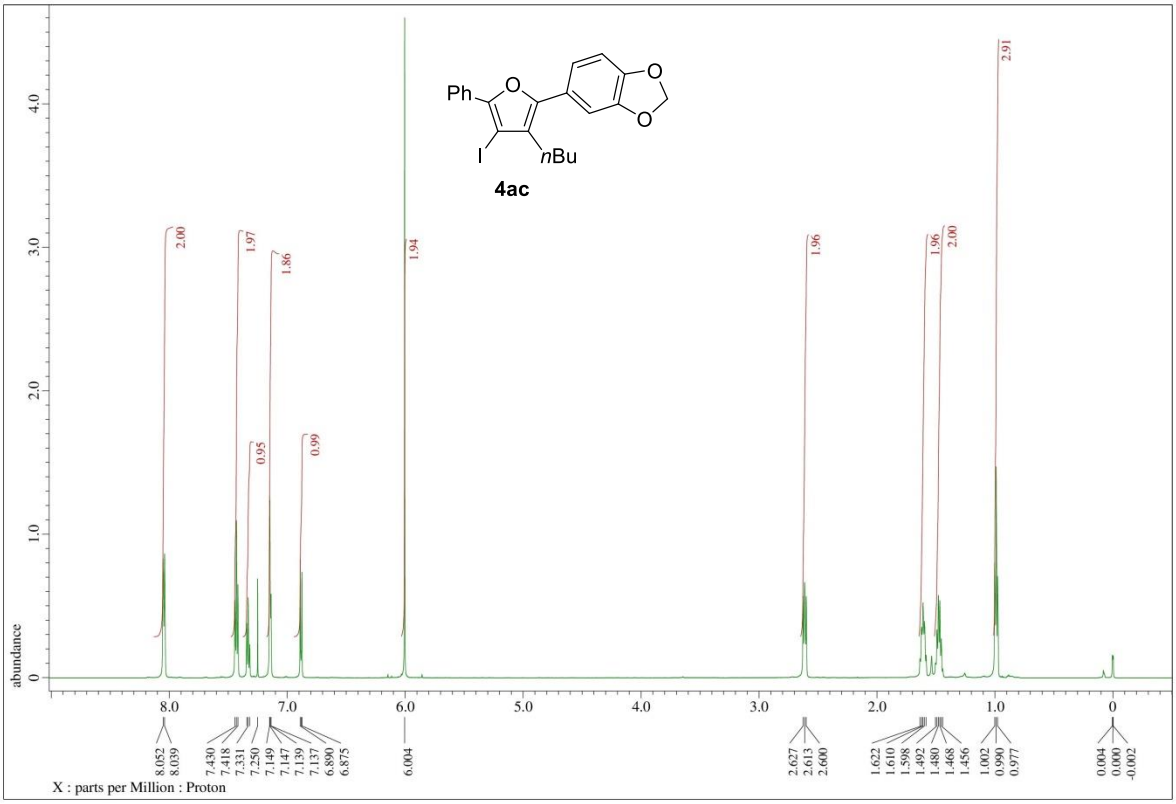

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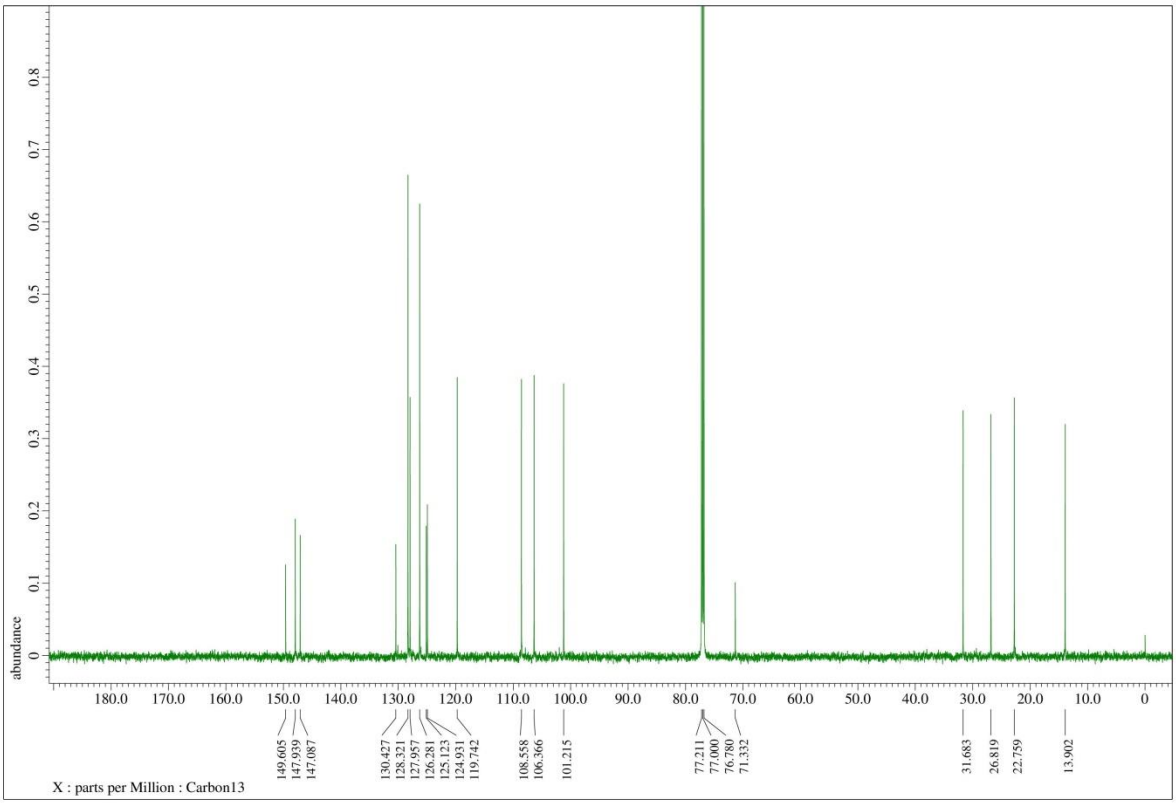

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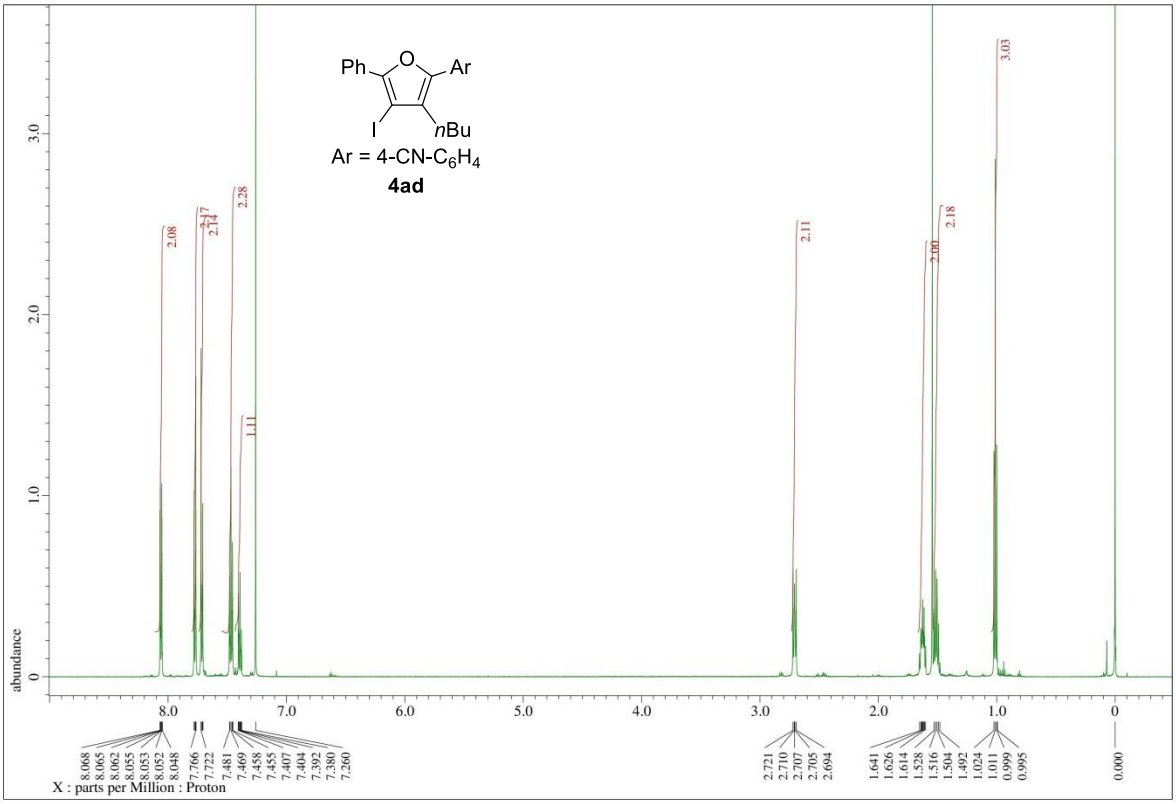

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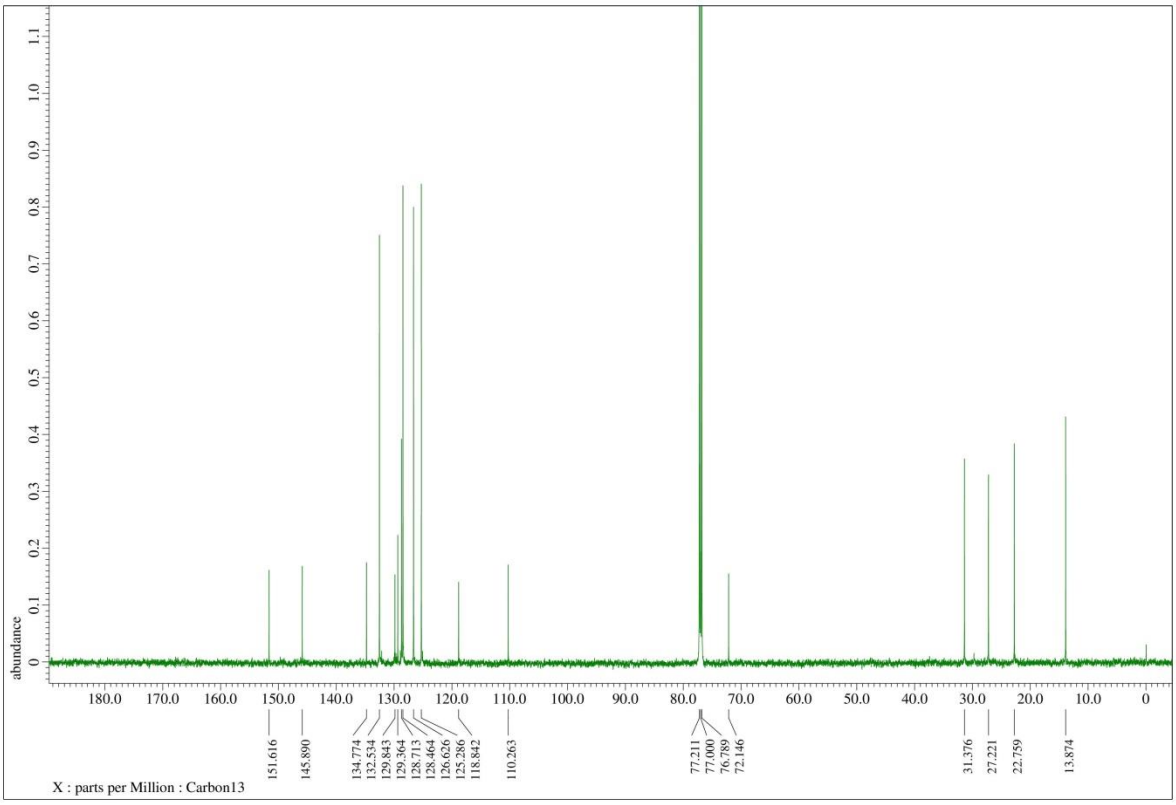

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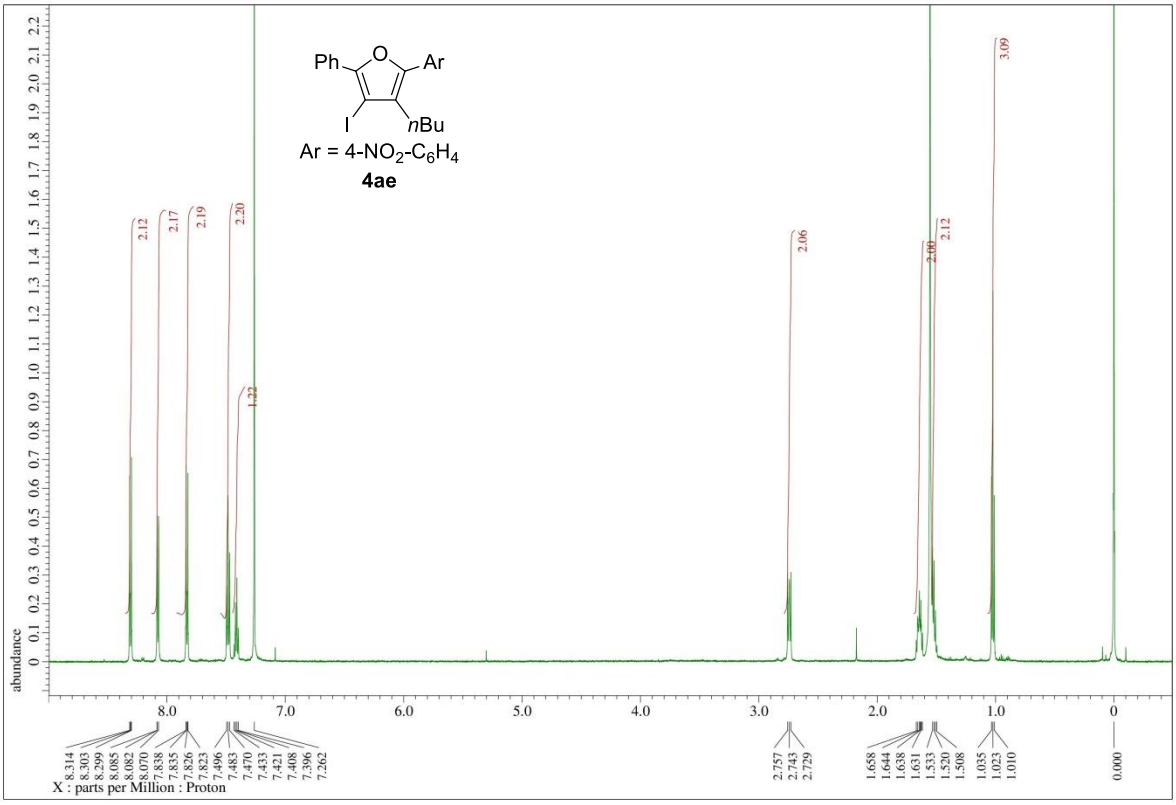

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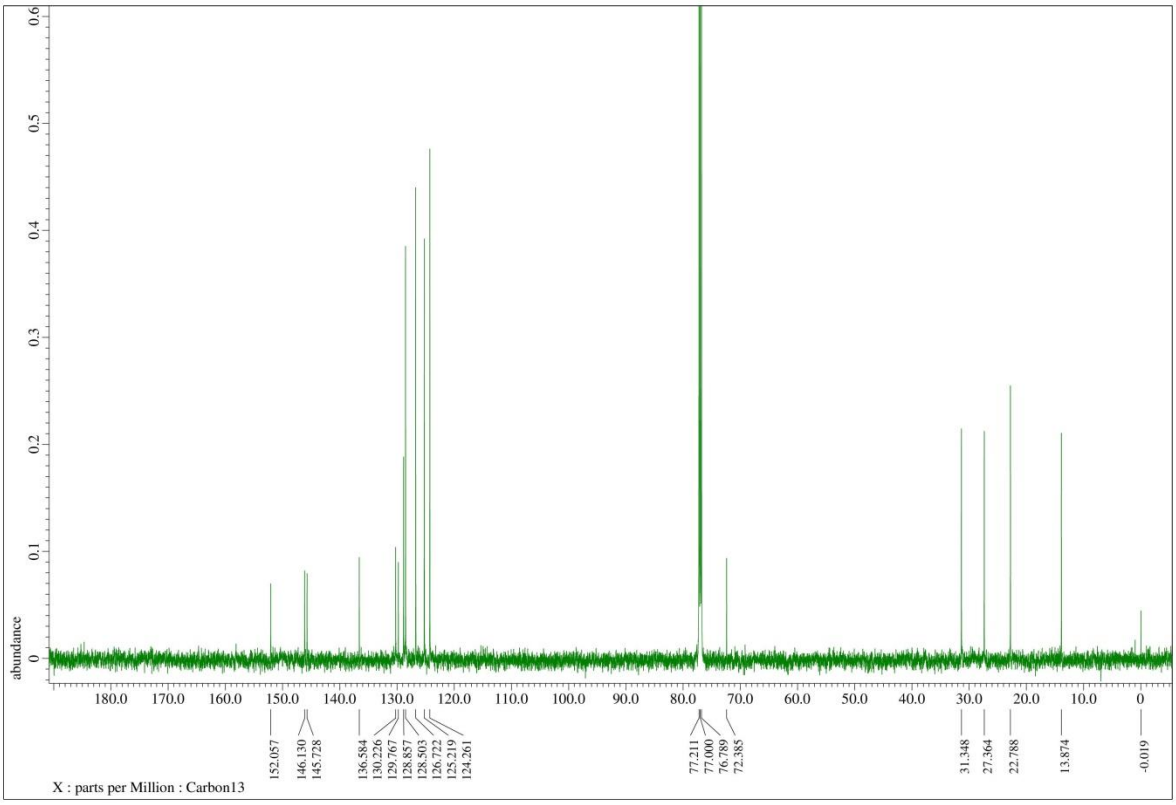

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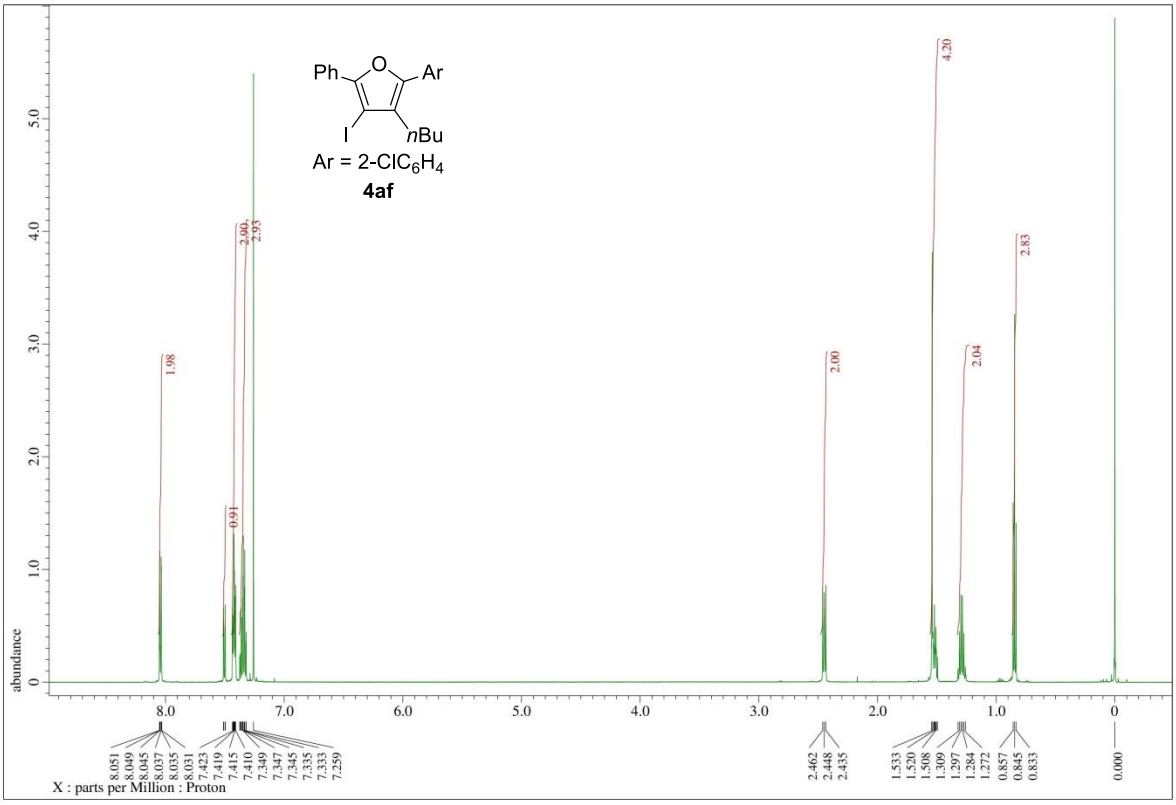

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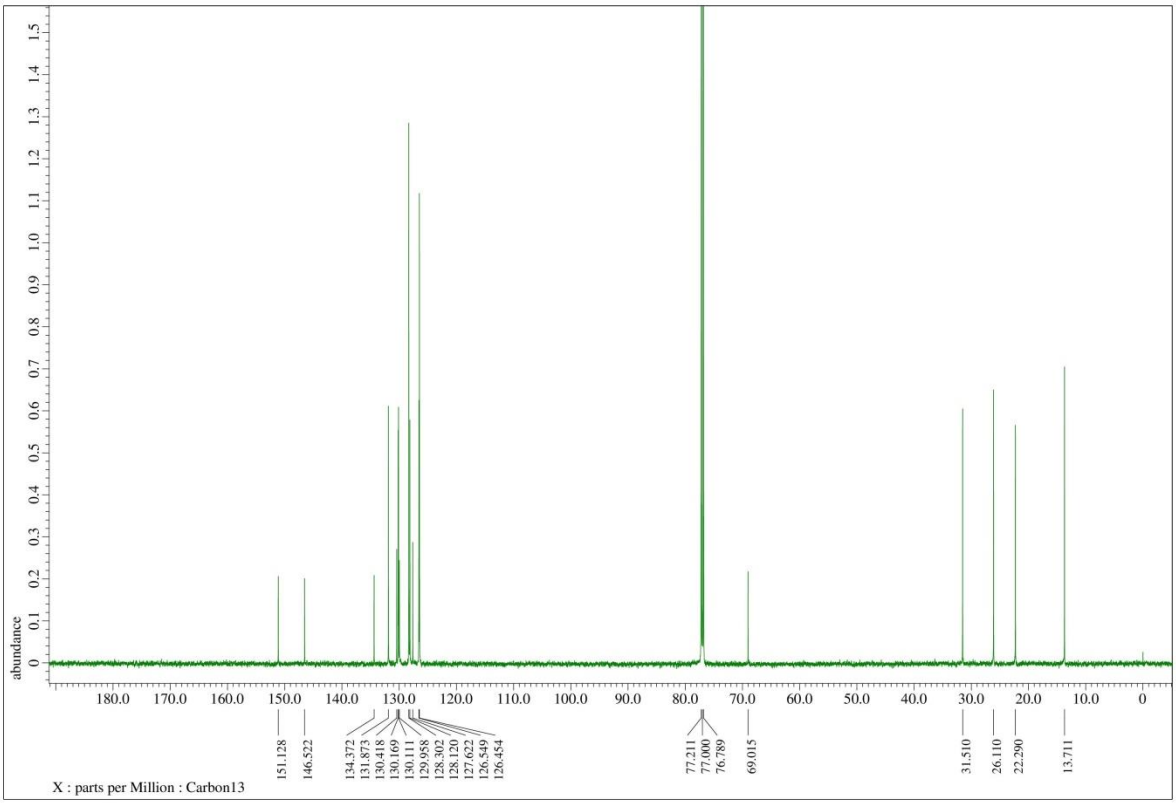

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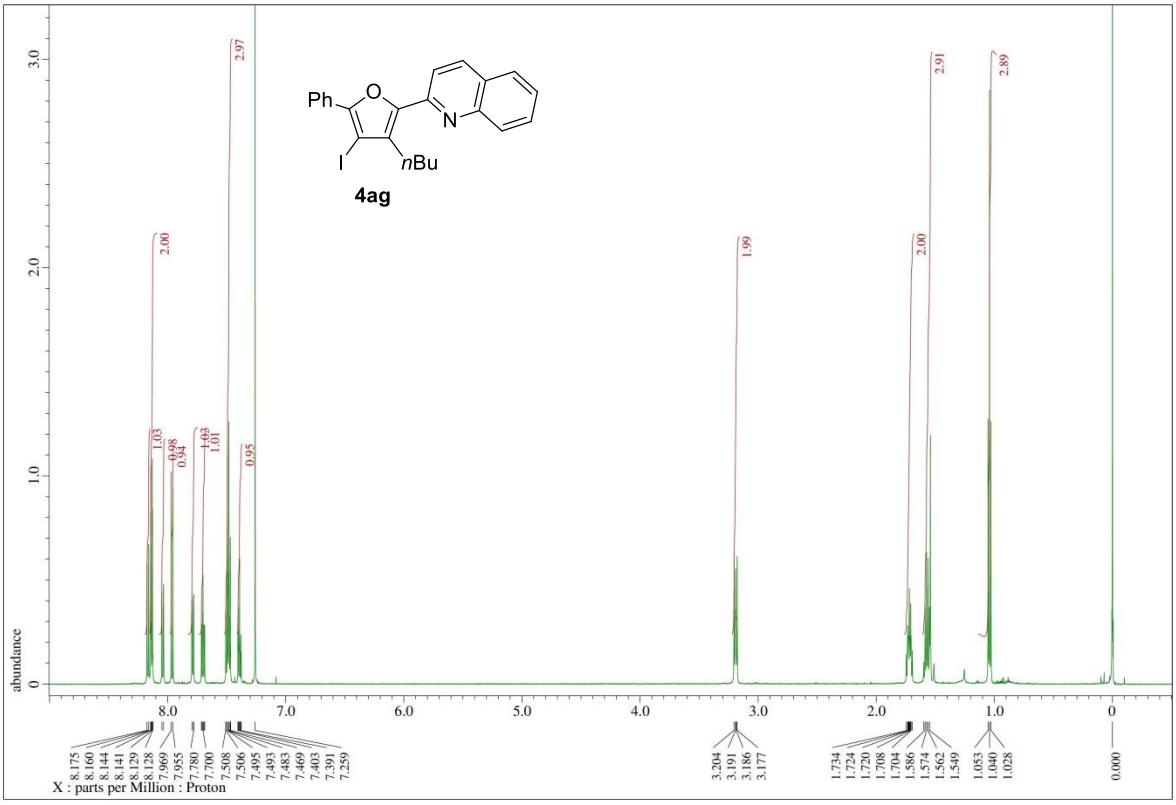

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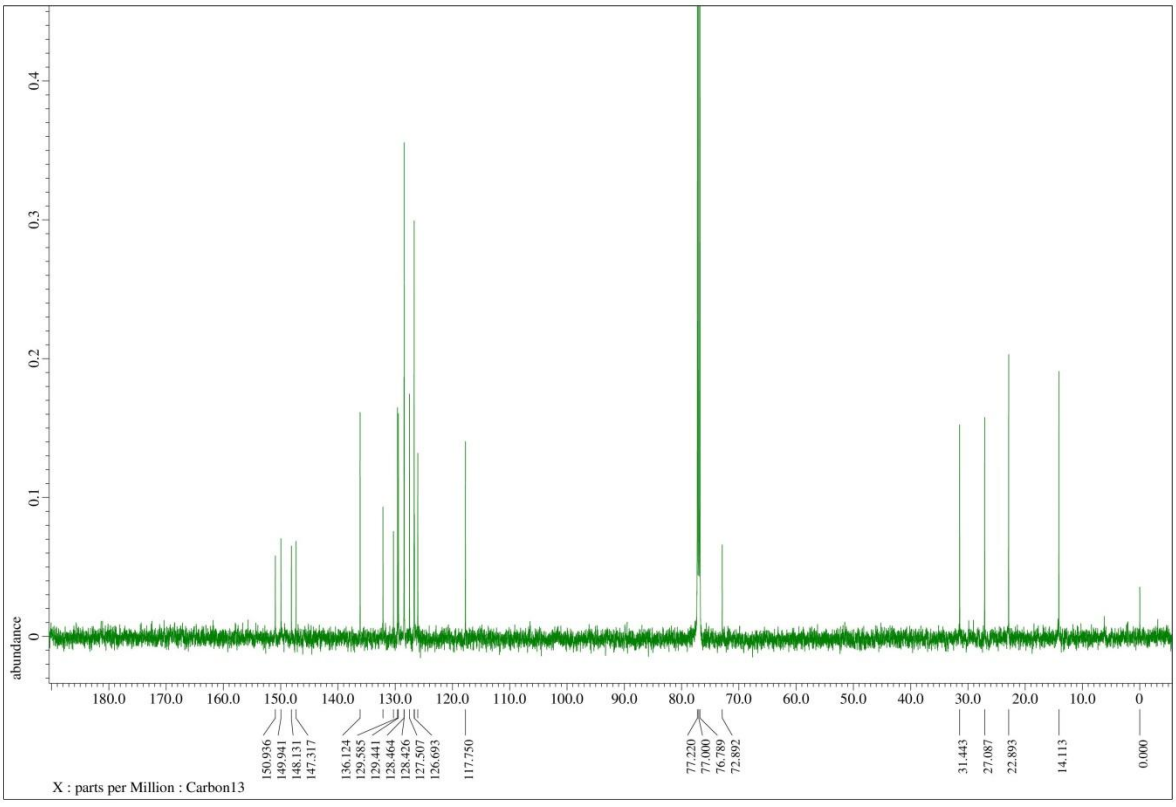

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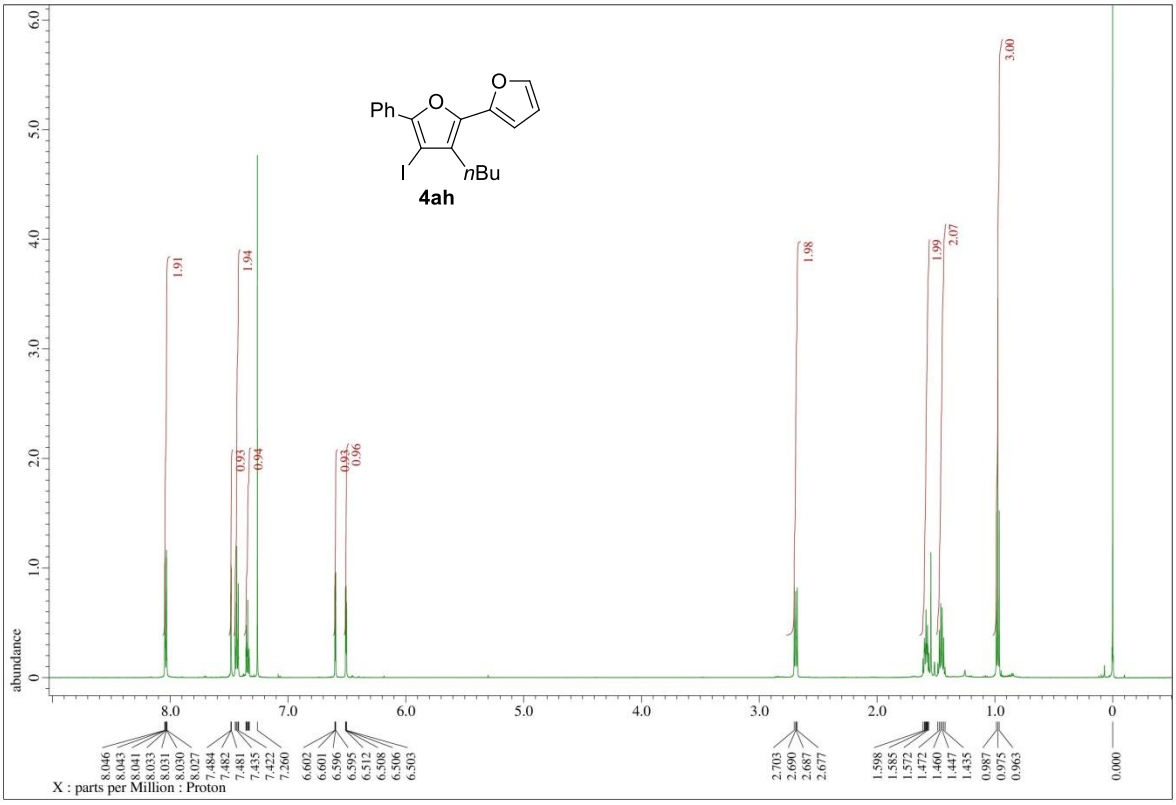
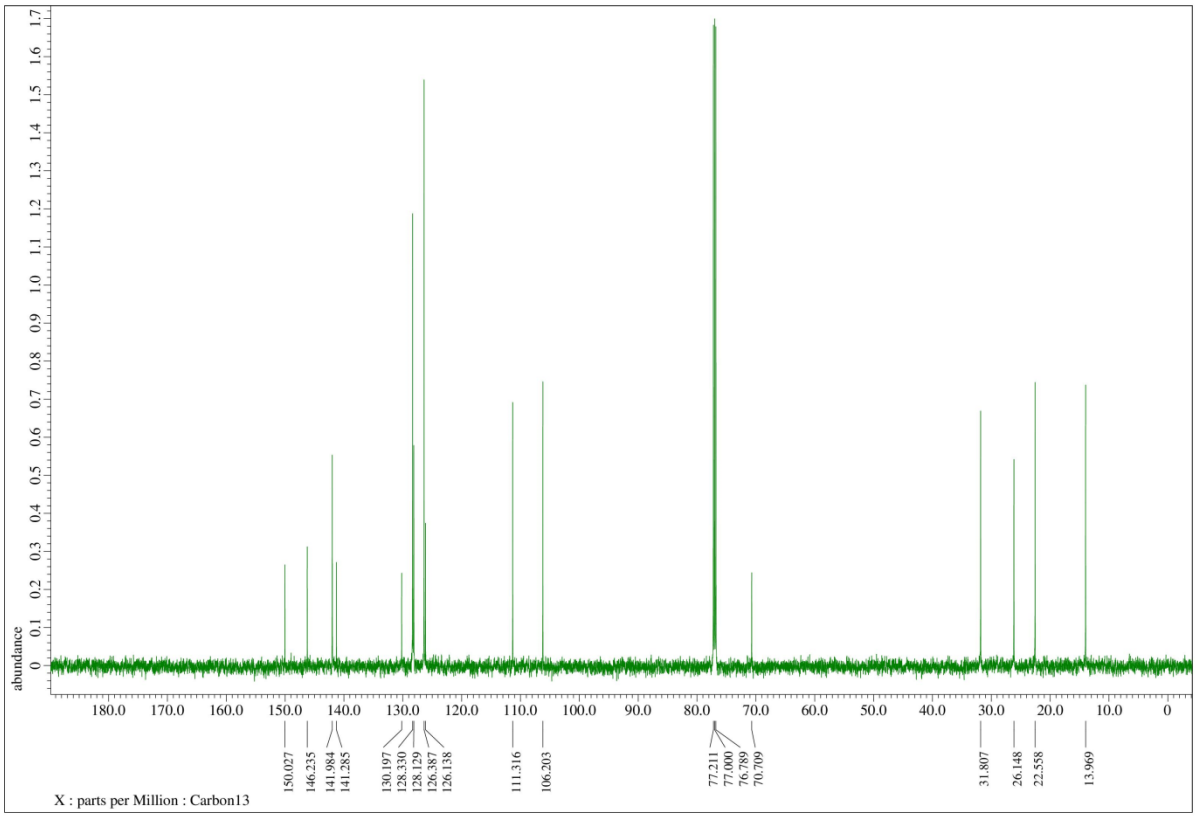
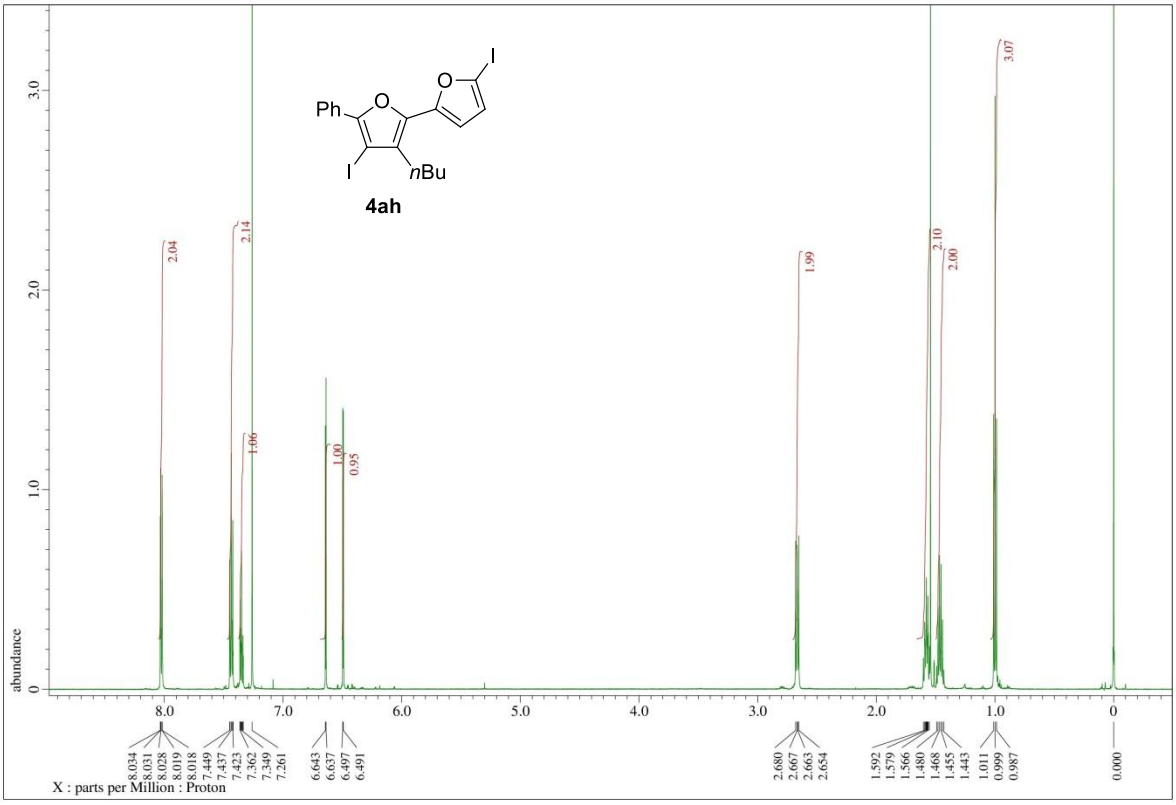


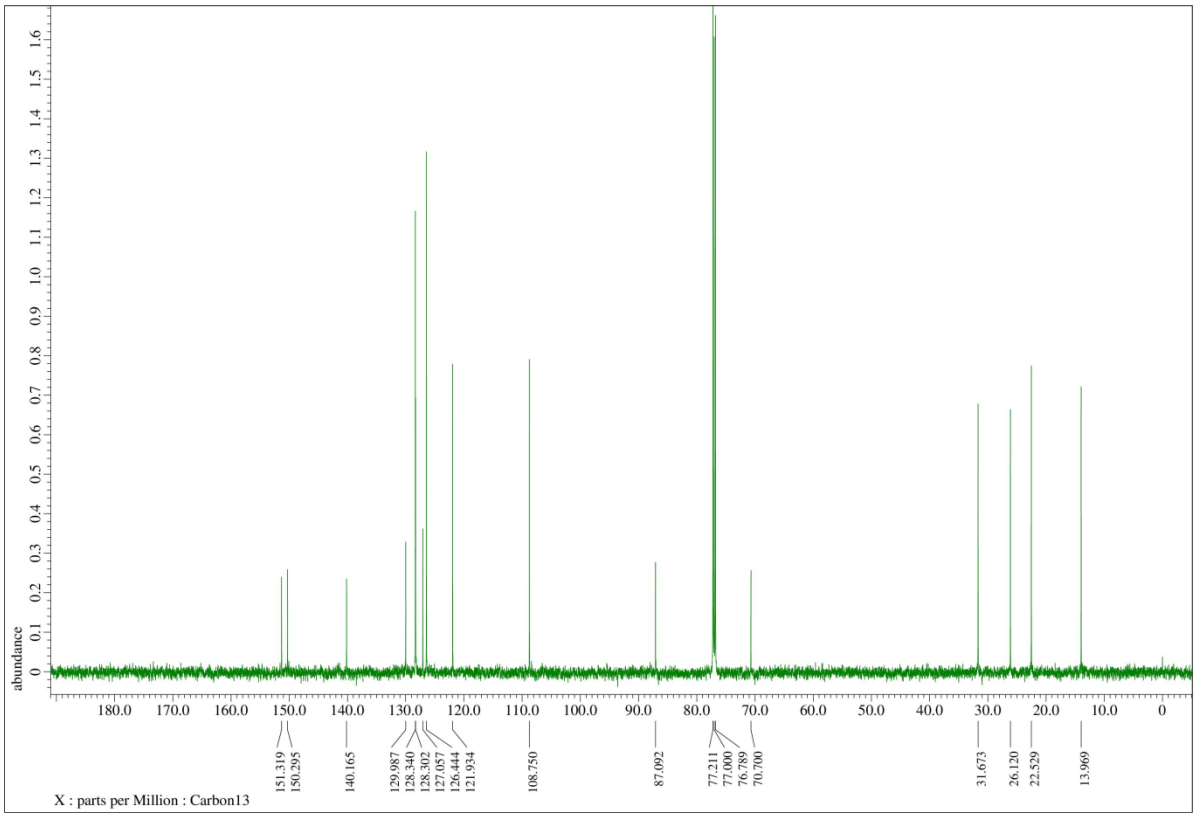
600 MHz, $CDCl_3$



150 MHz, $CDCl_3$



600 MHz, $CDCl_3$



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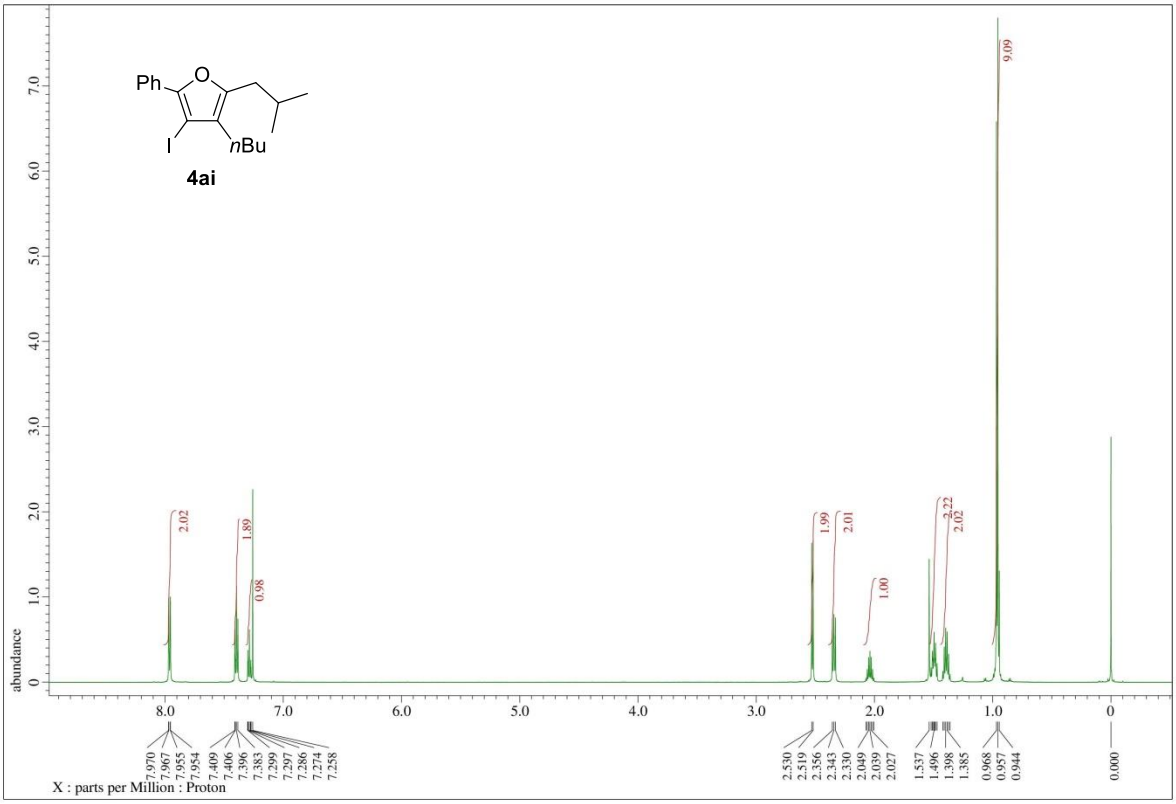
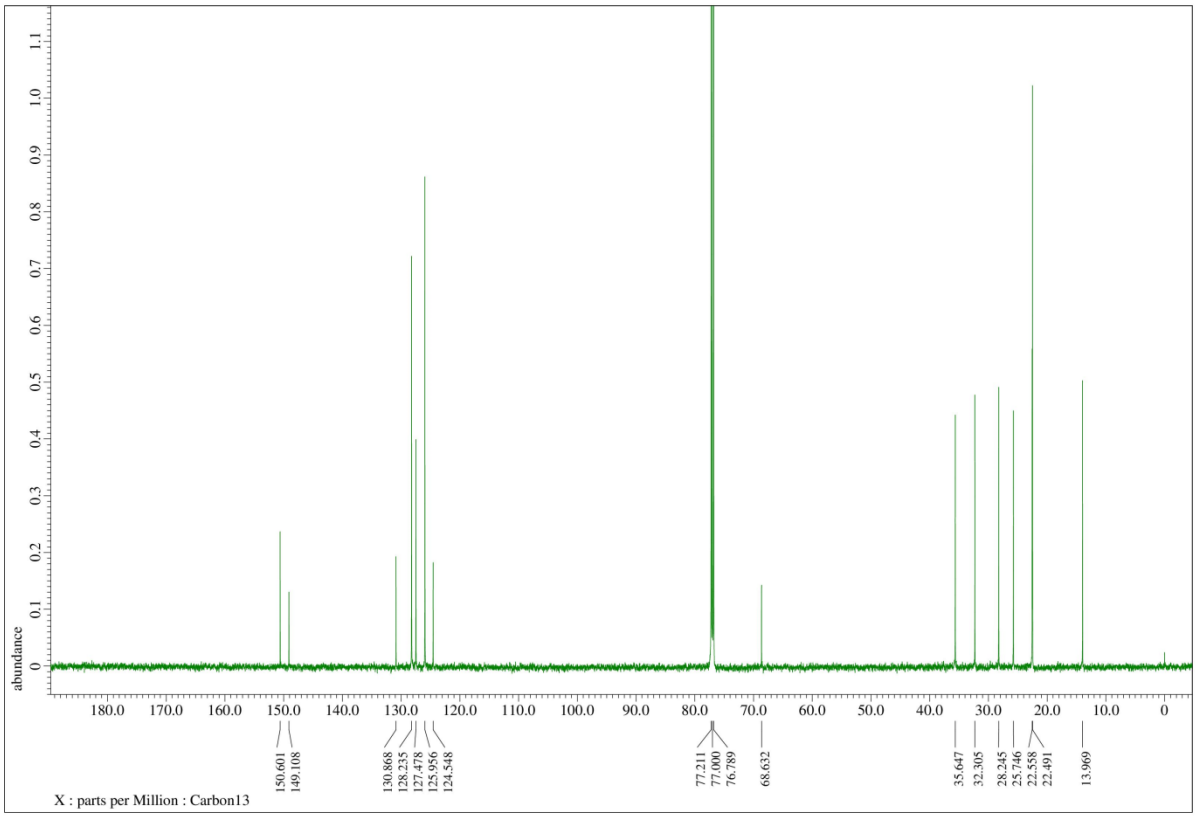


600 MHz, $CDCl_3$



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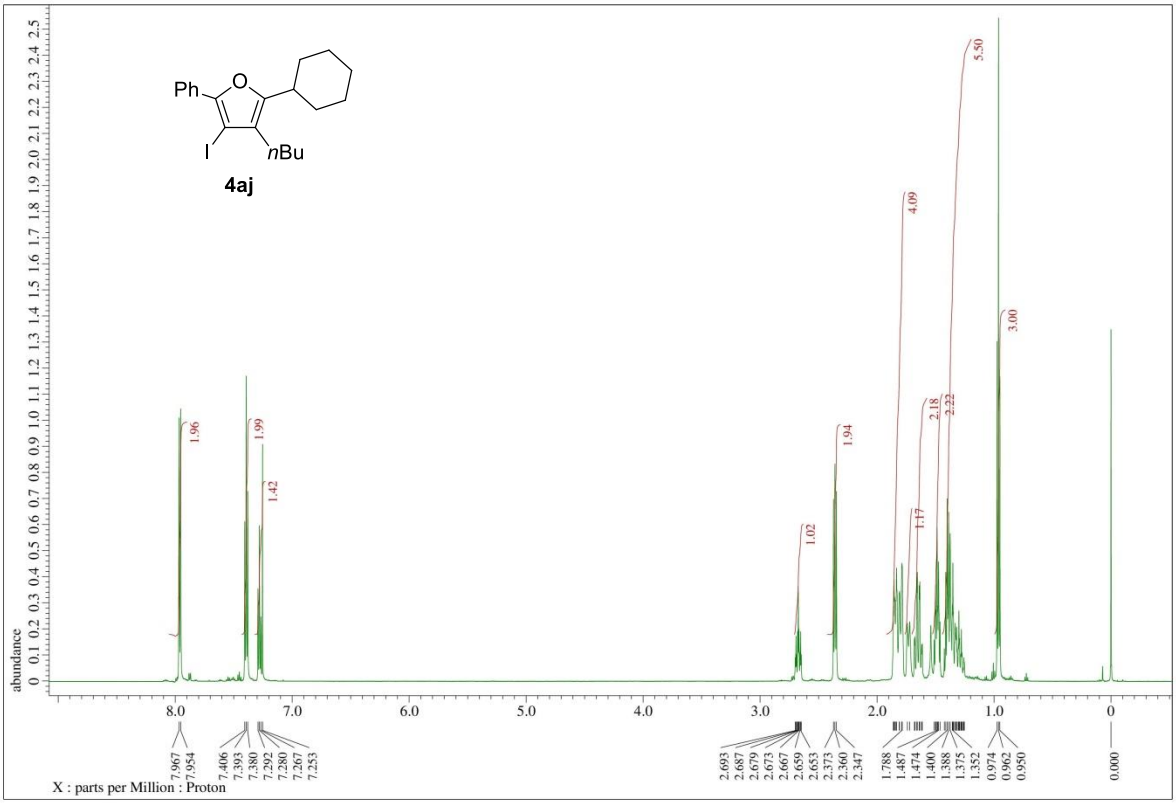

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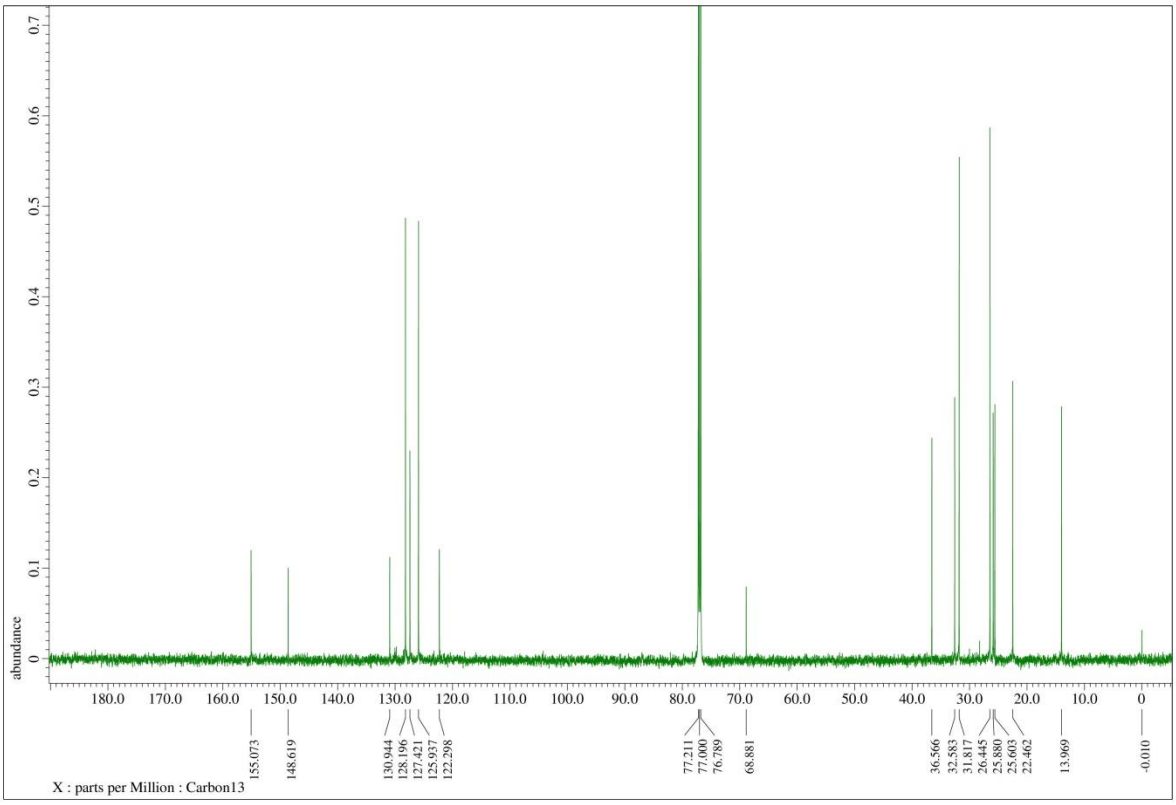

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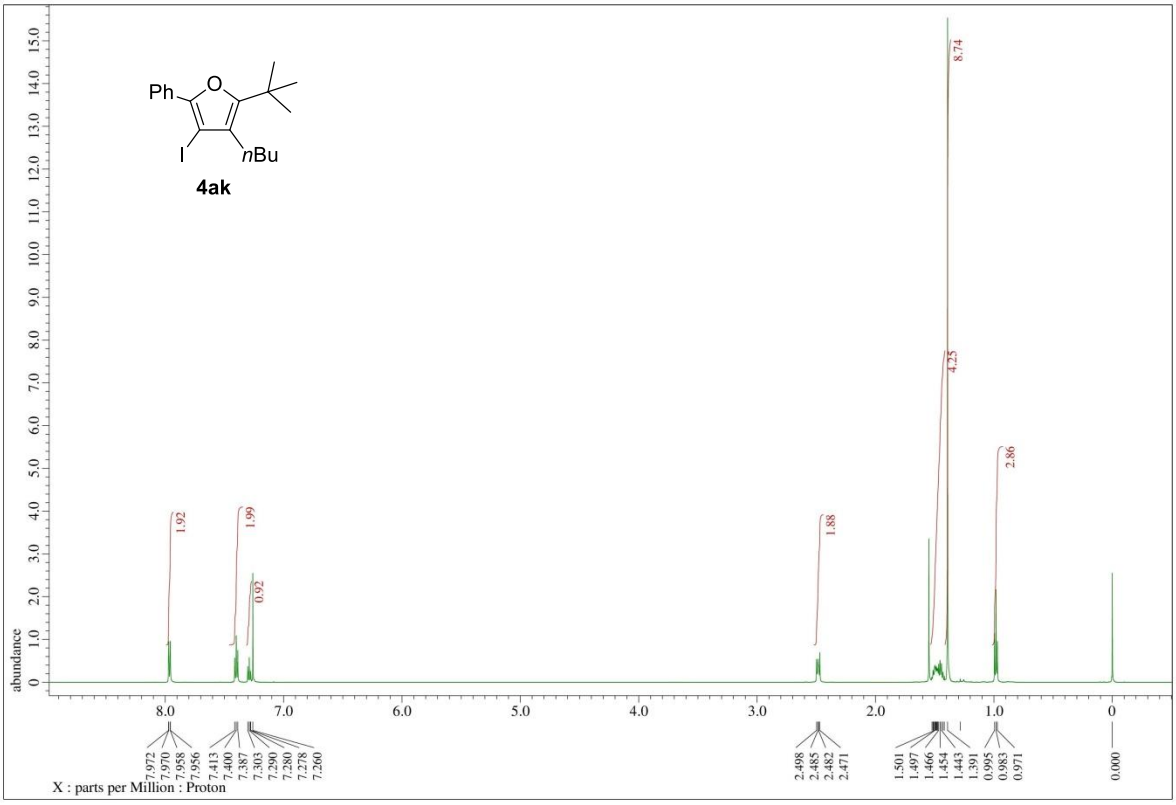

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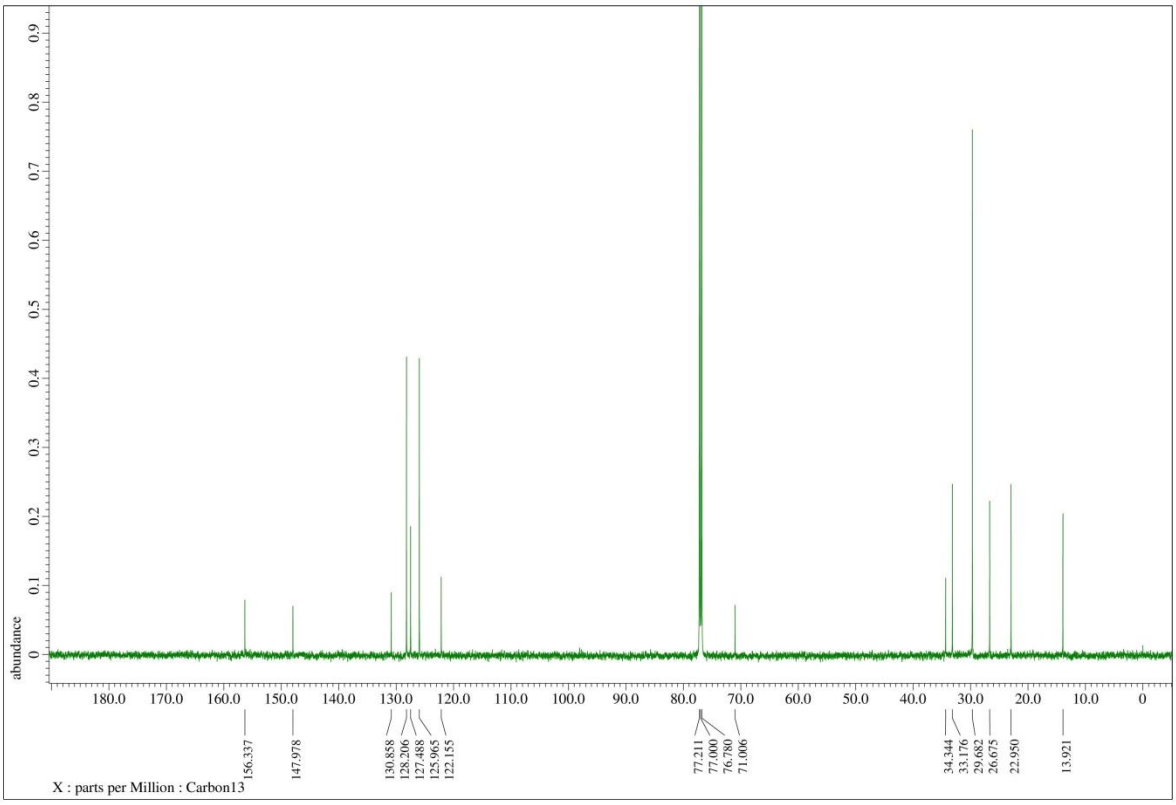

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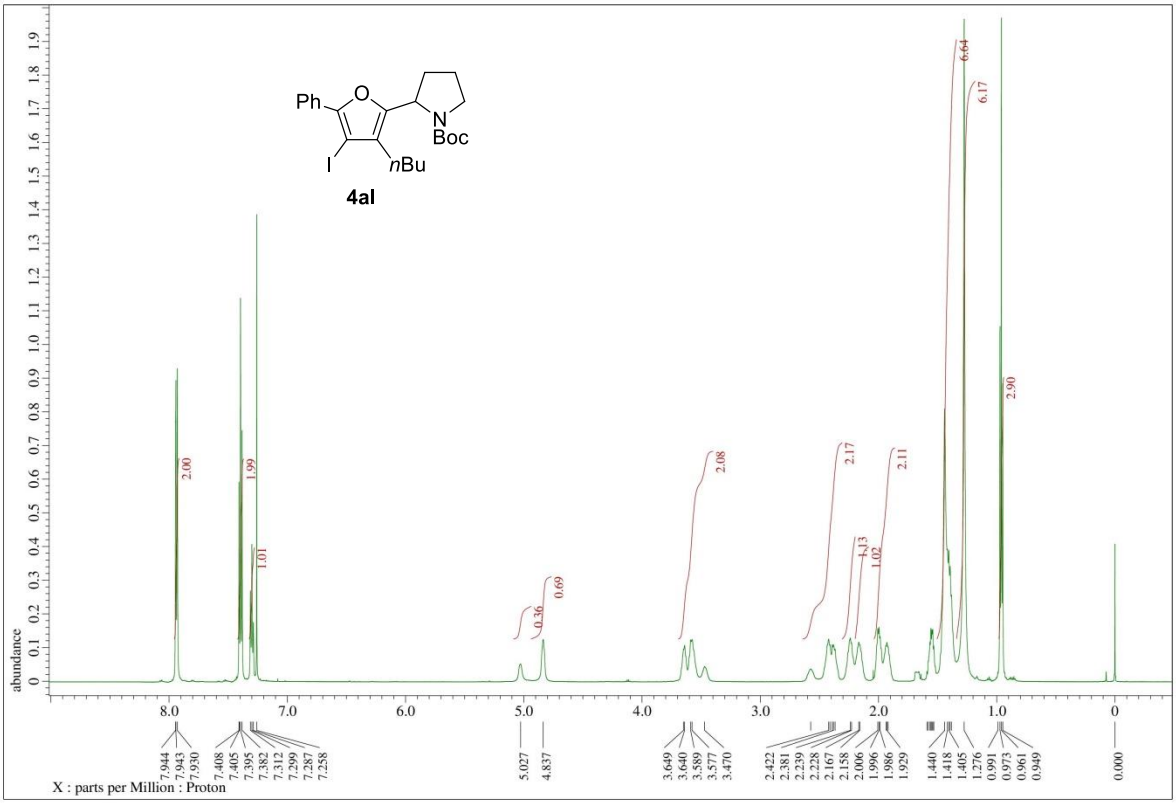
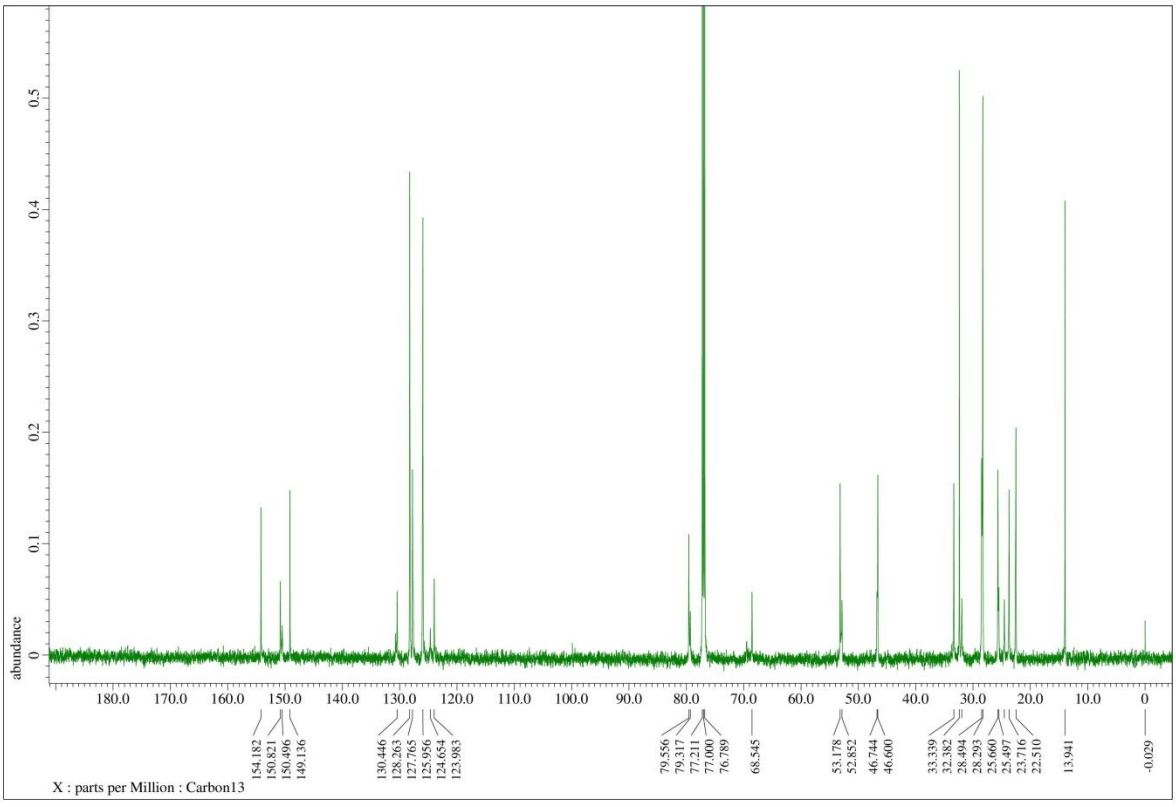


600 MHz, $CDCl_3$



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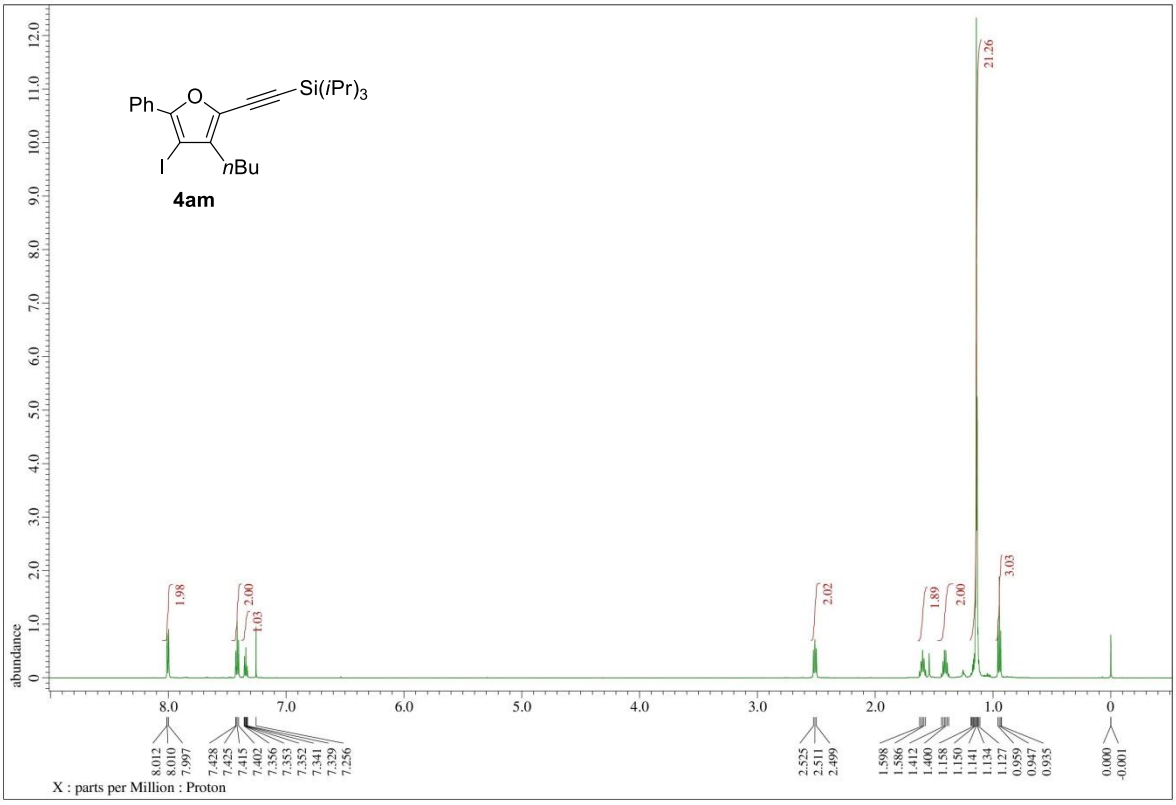
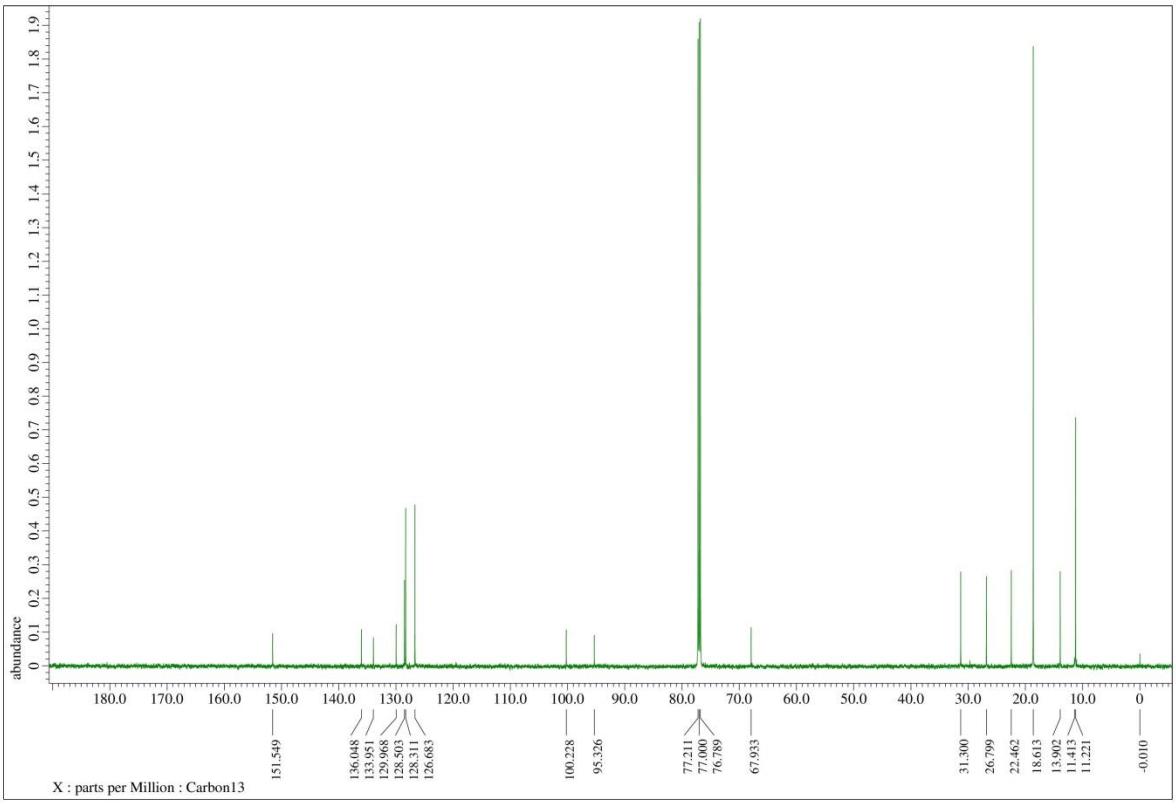


600 MHz, $CDCl_3$



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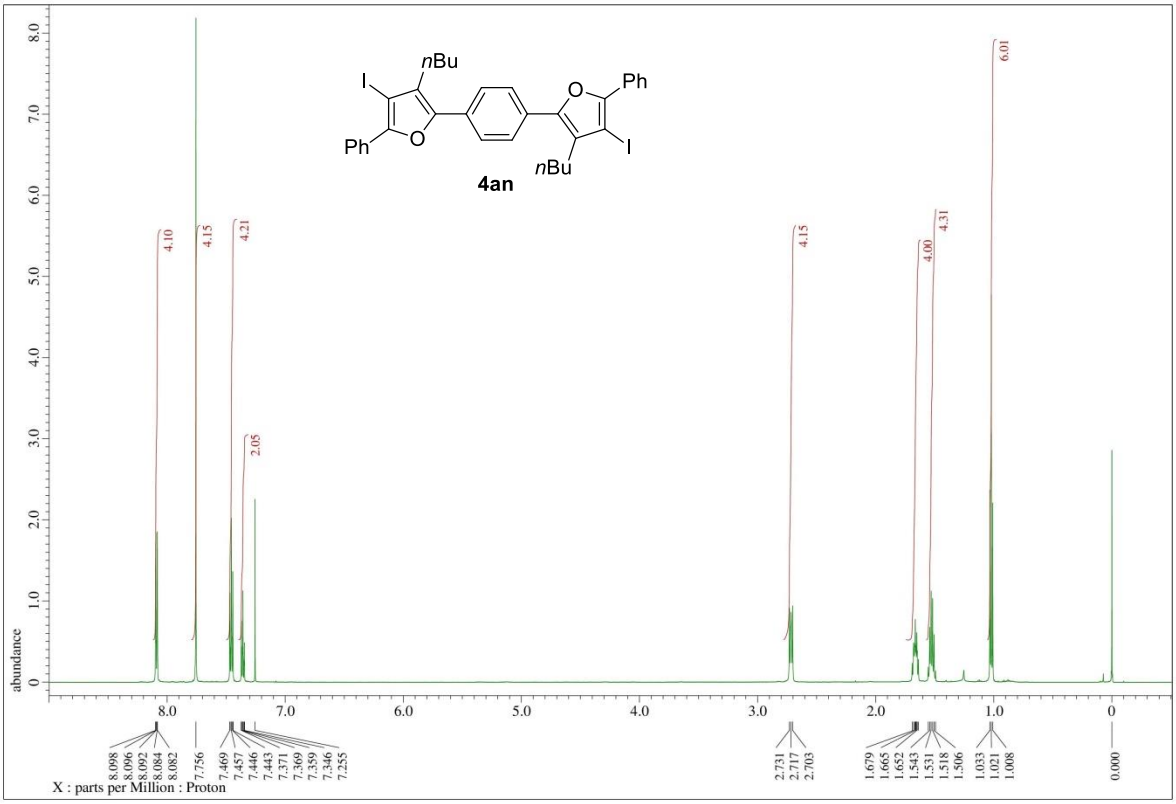
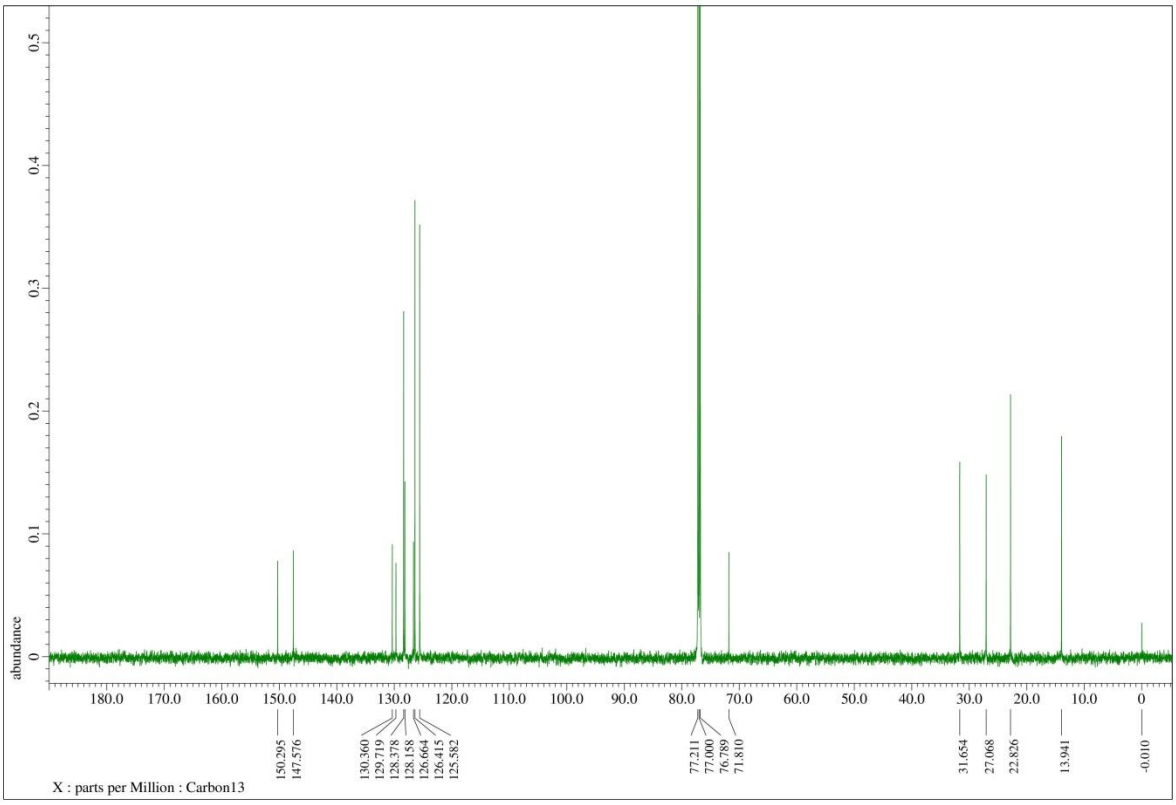
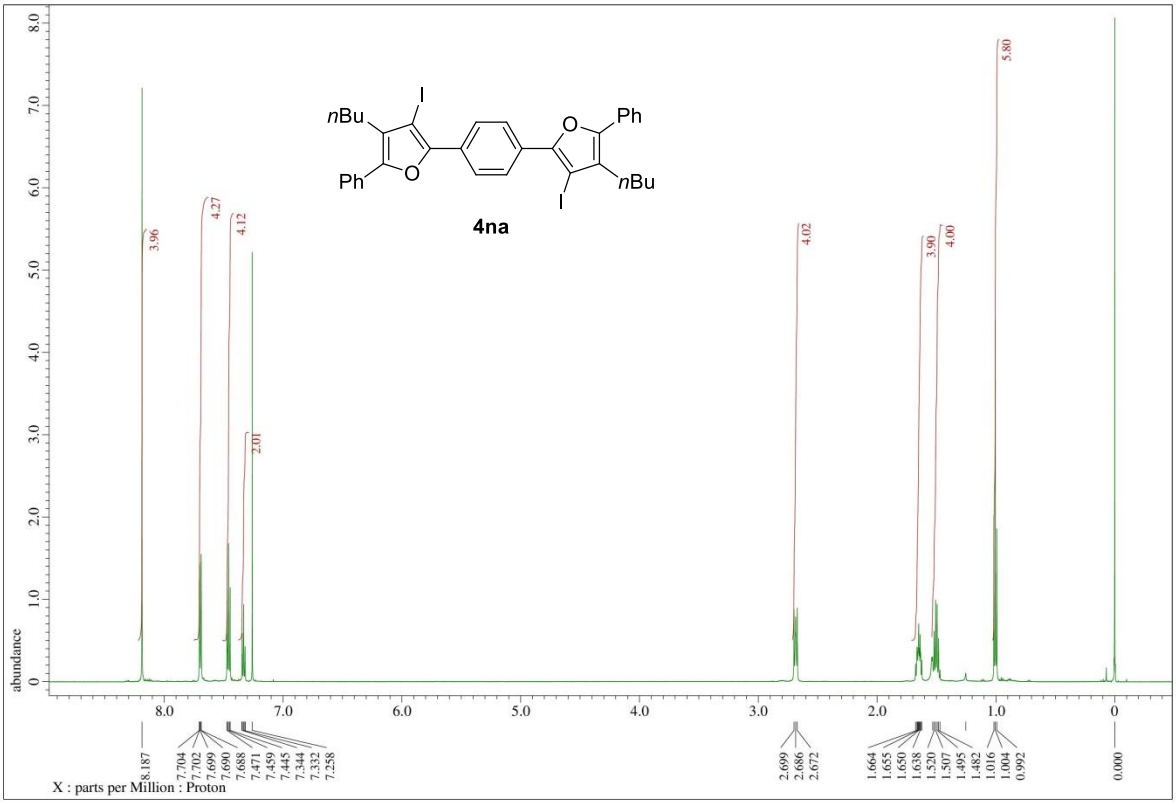


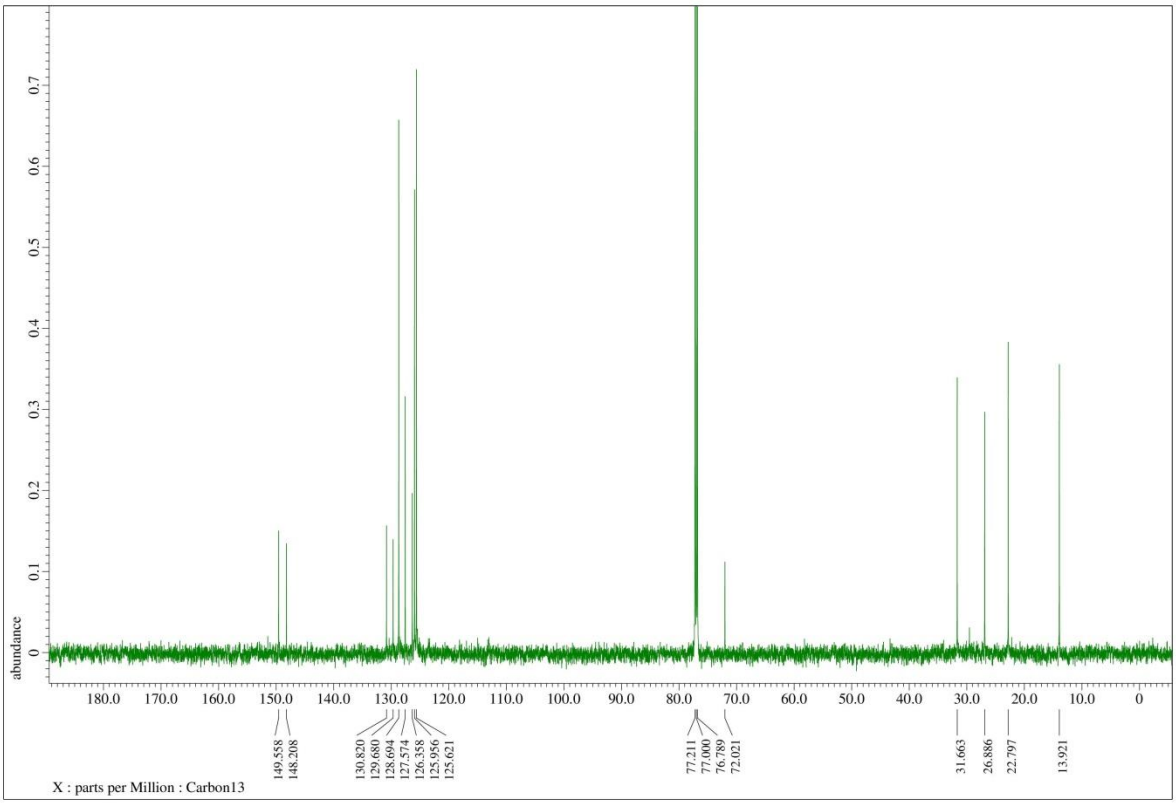
600 MHz, $CDCl_3$



150 MHz, $CDCl_3$



600 MHz, $CDCl_3$



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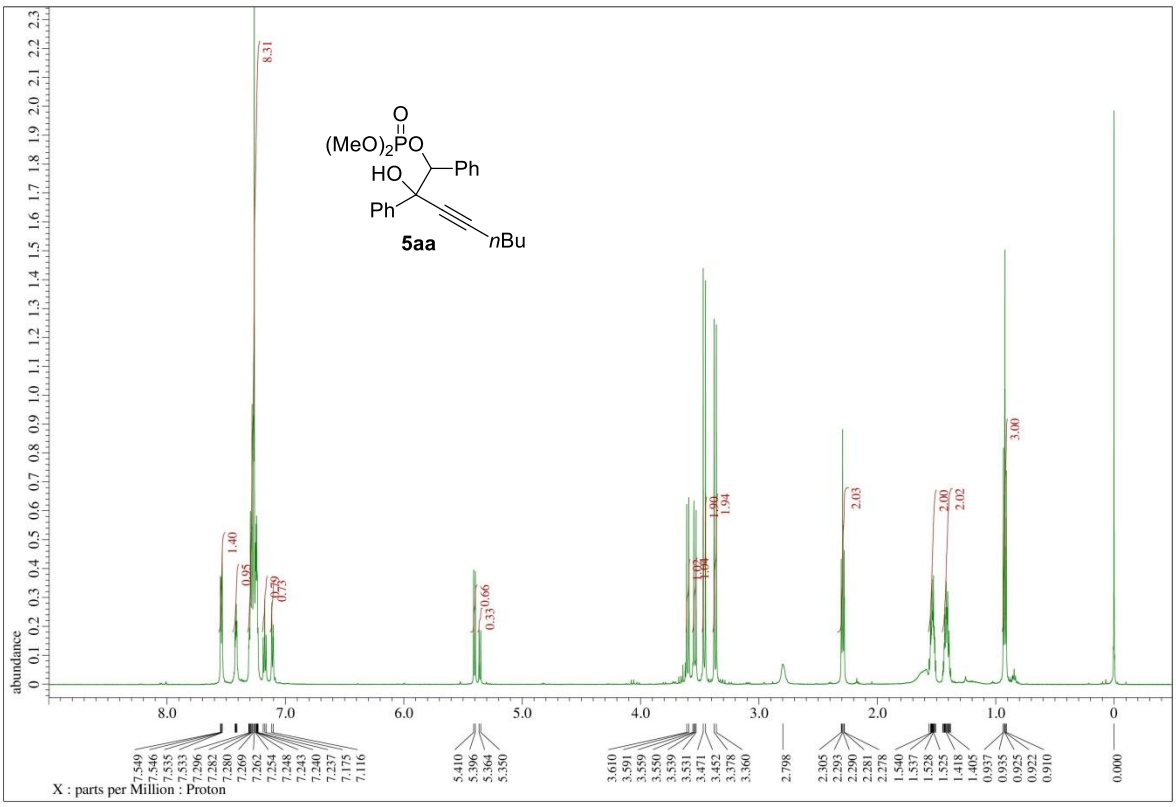
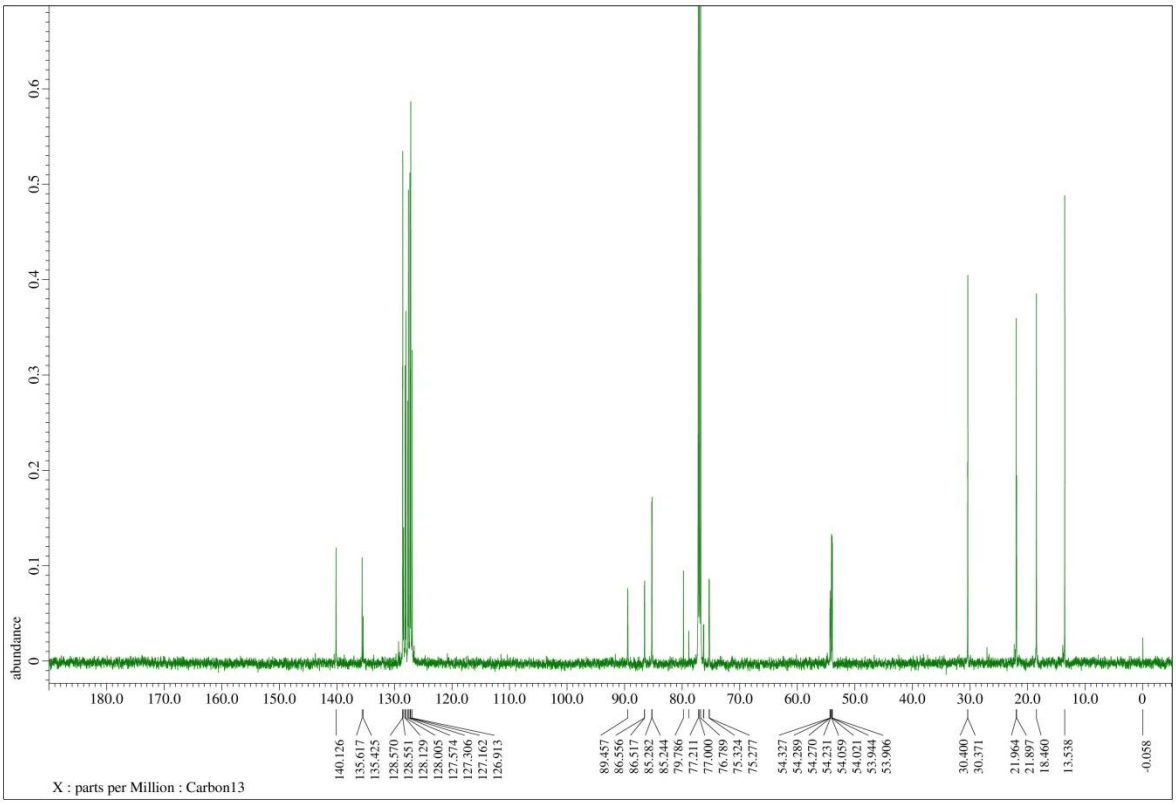
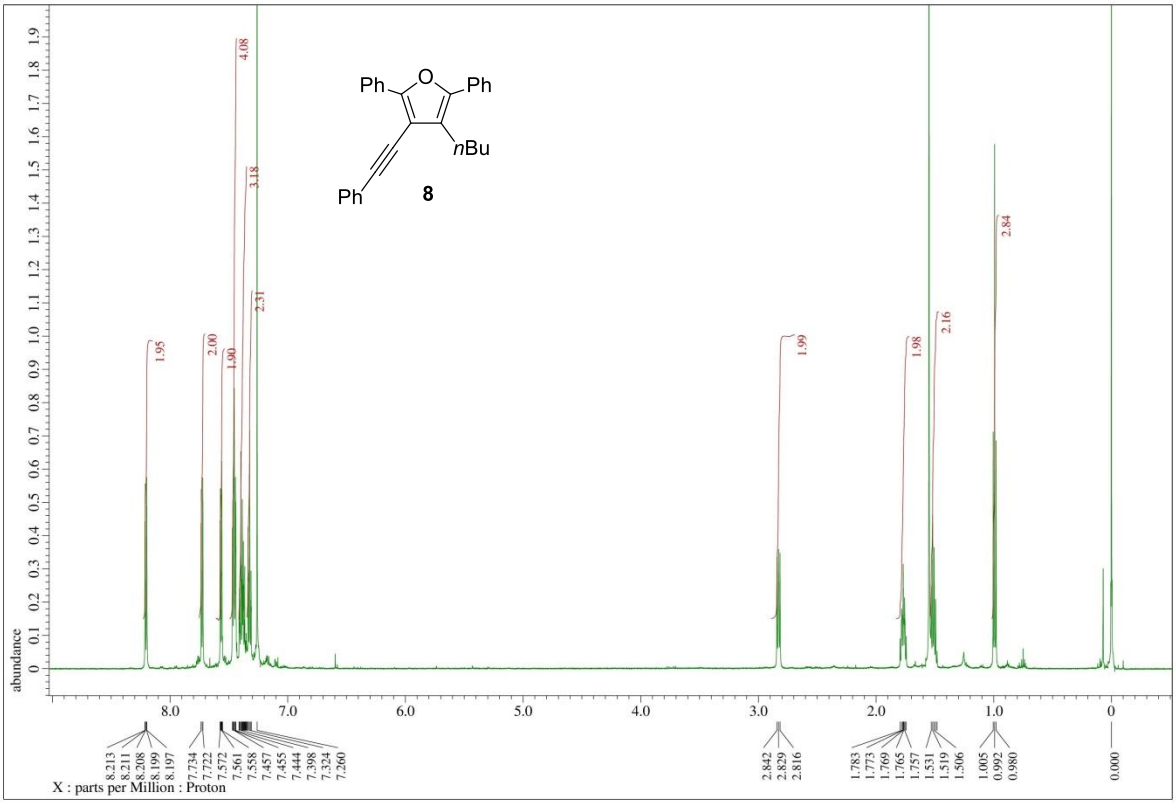


600 MHz, $CDCl_3$



150 MHz, $CDCl_3$



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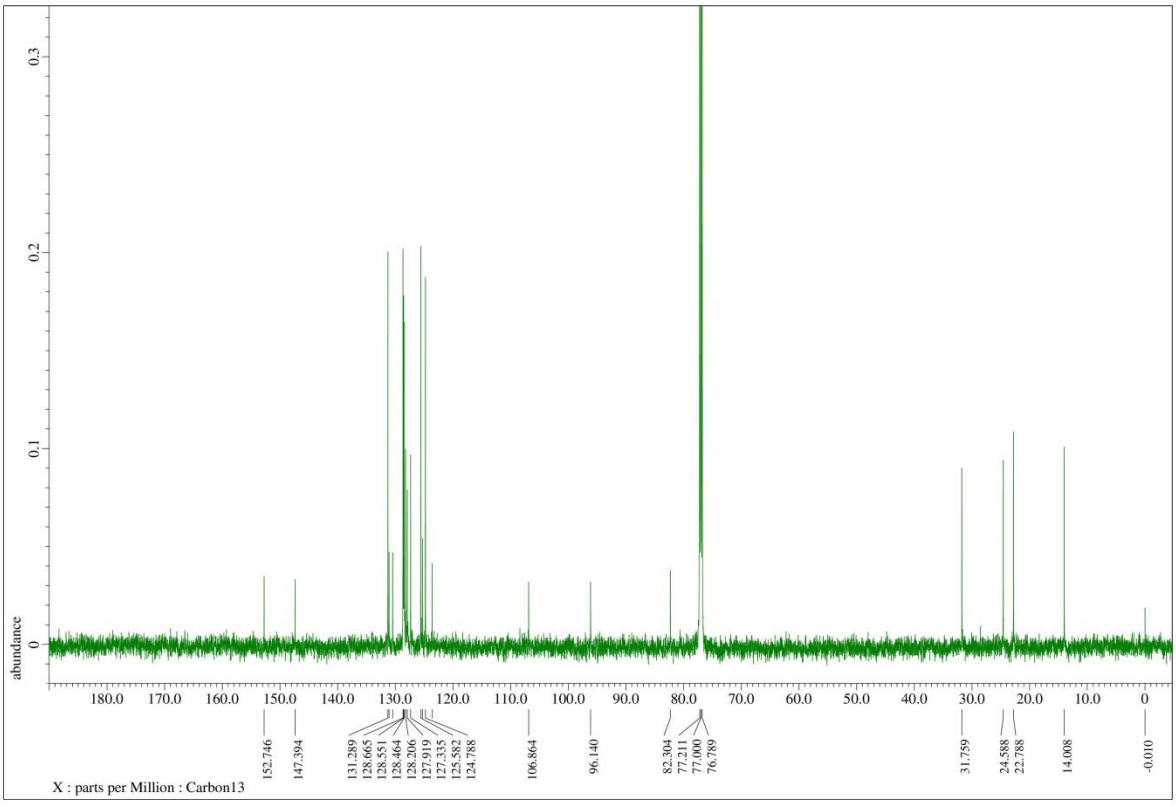

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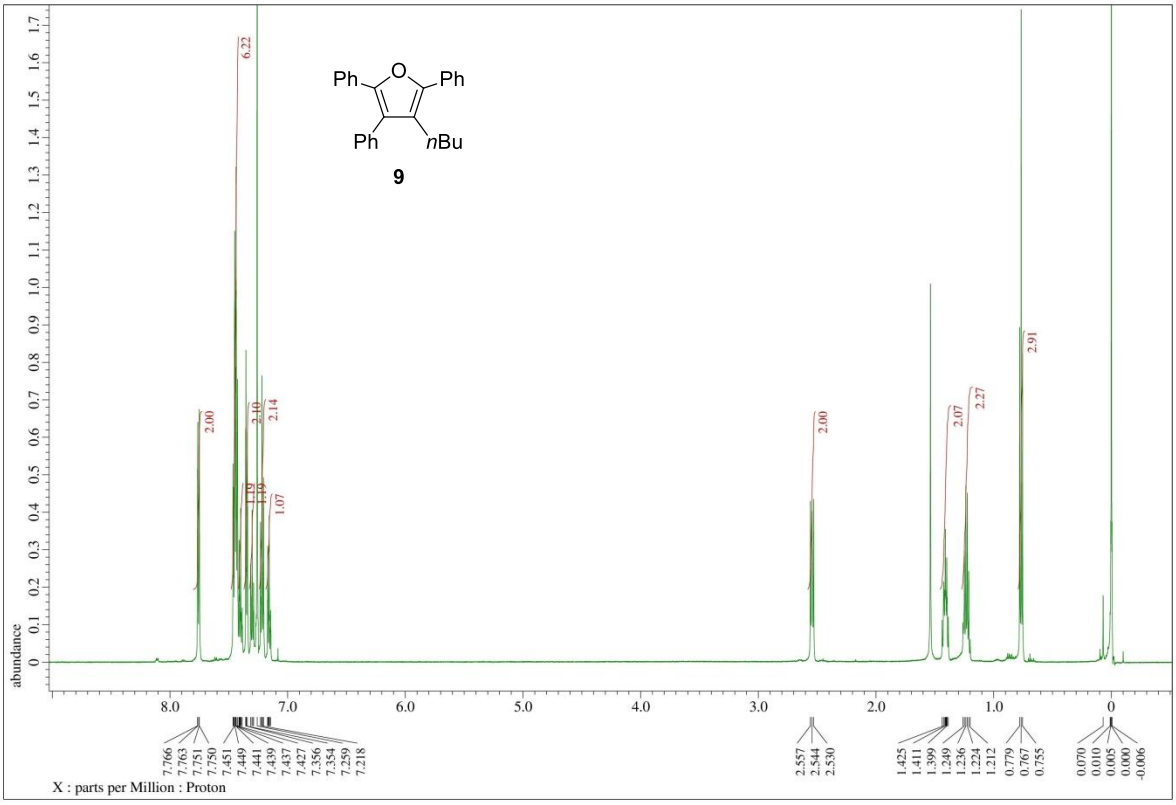

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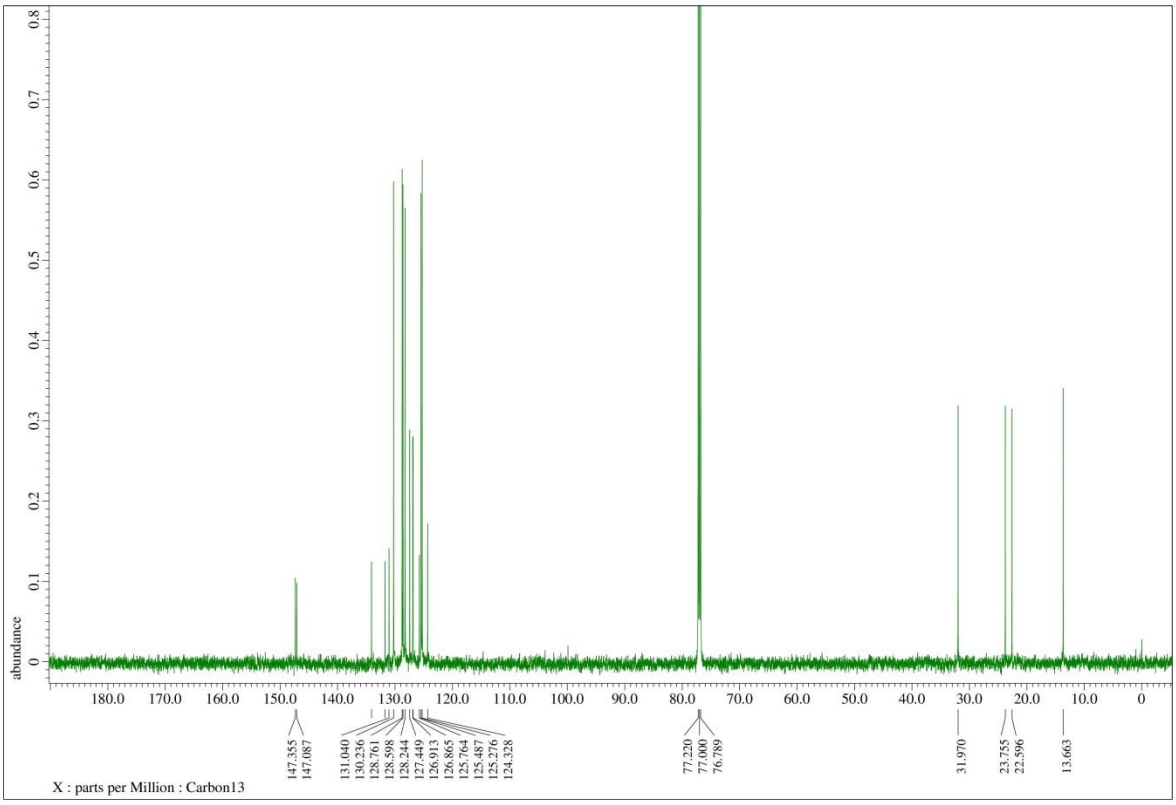
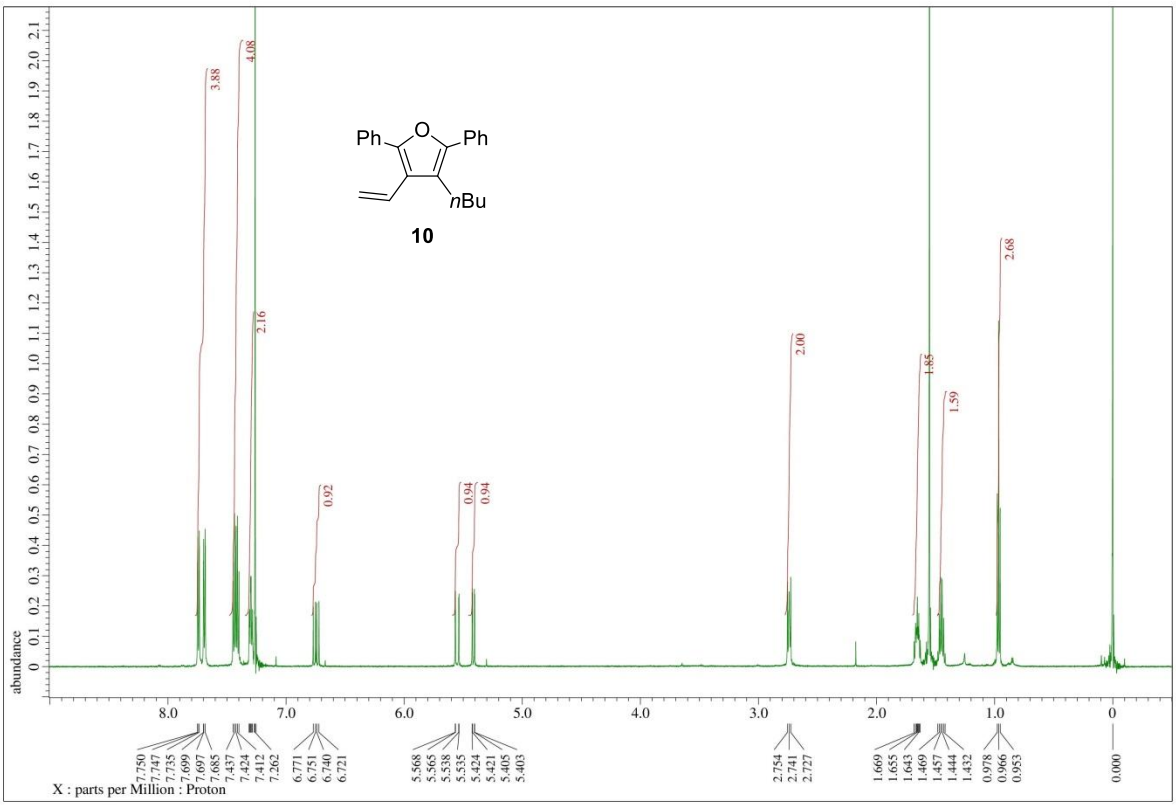


150 MHz, $CDCl_3$



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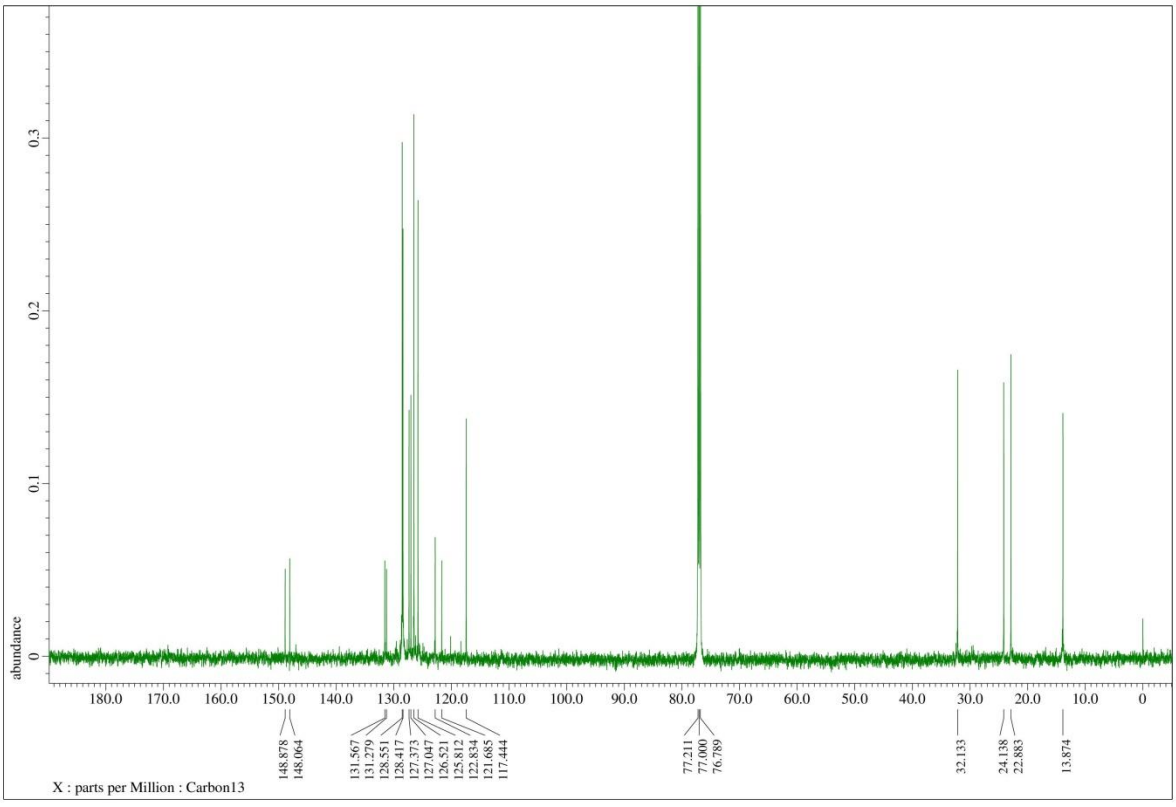

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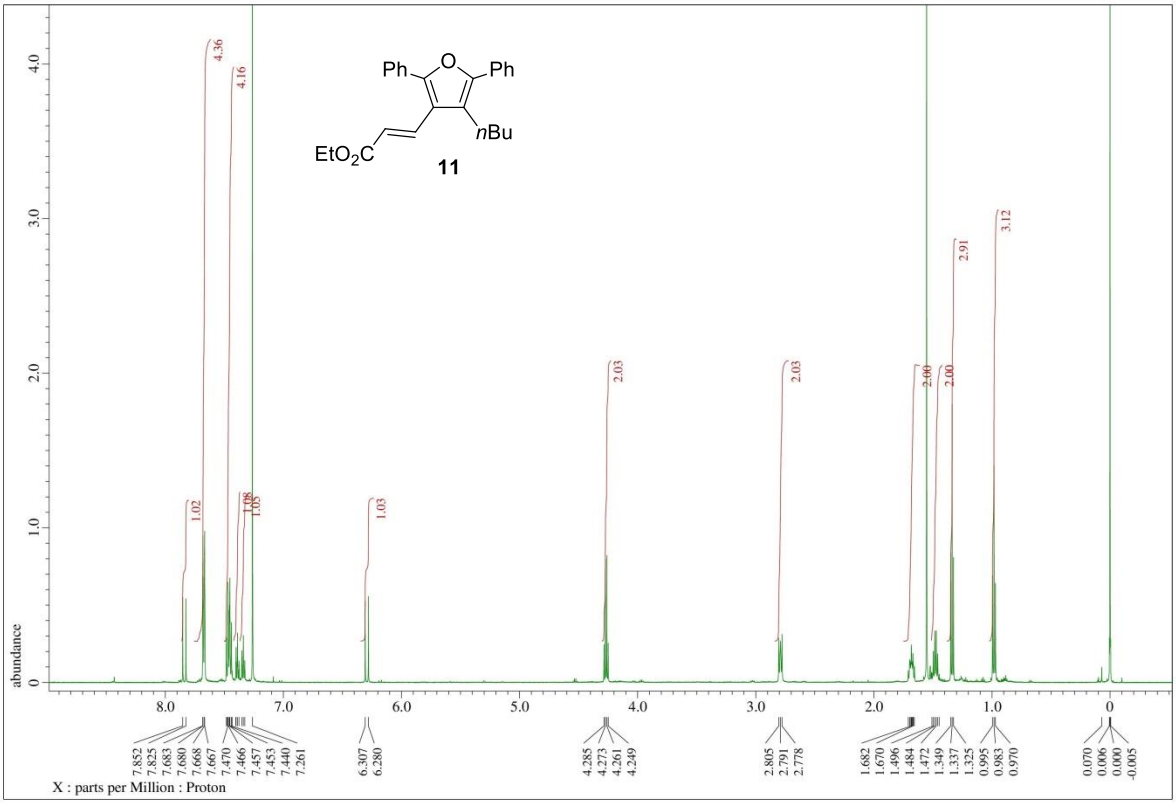

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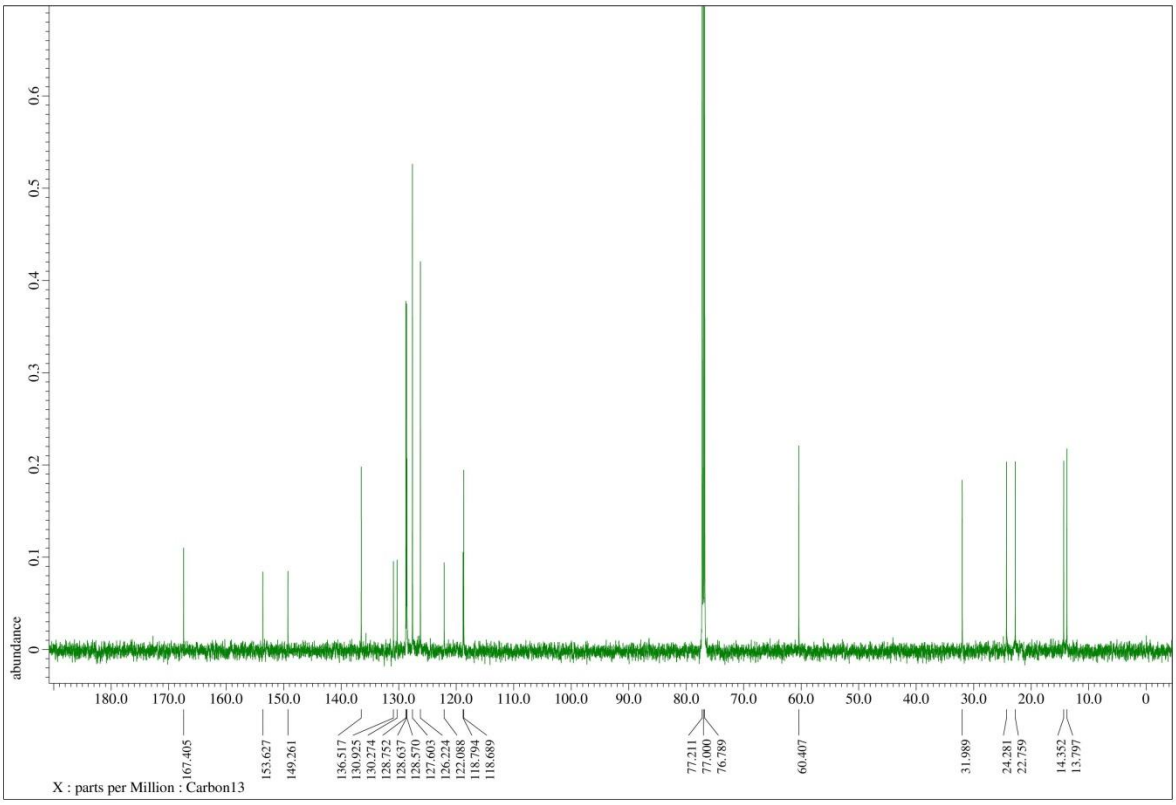

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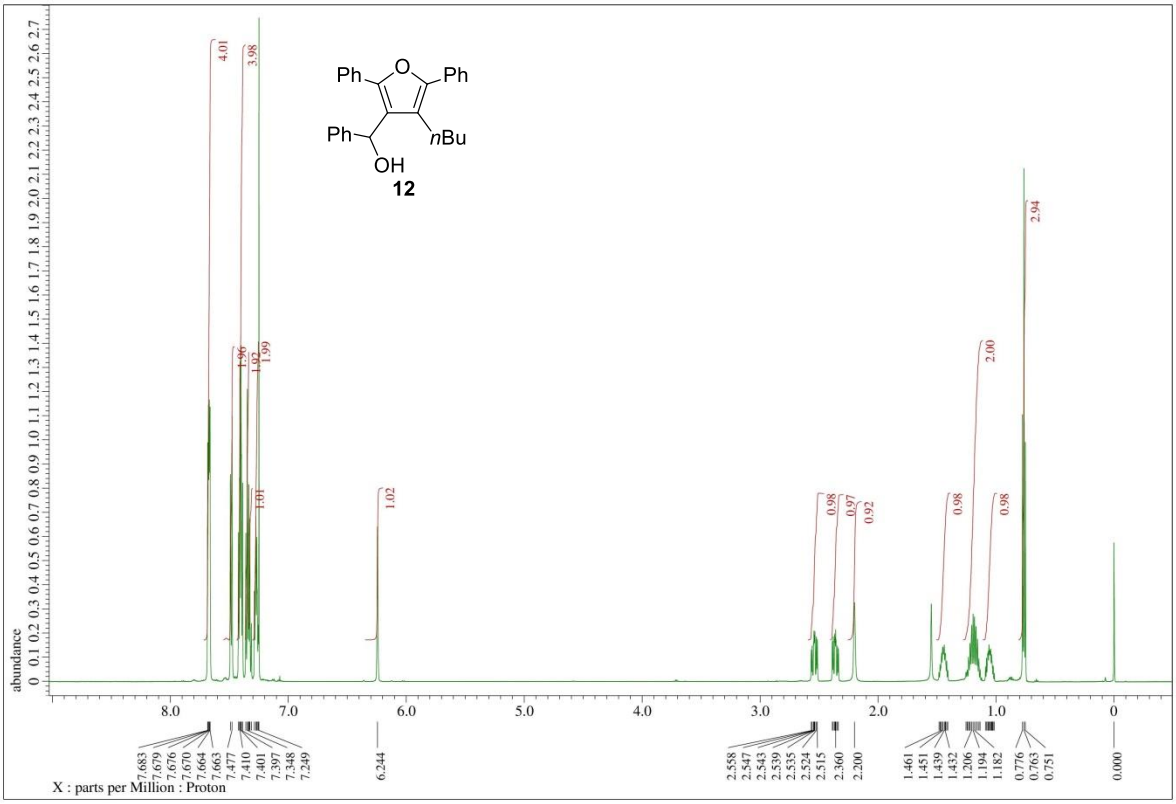

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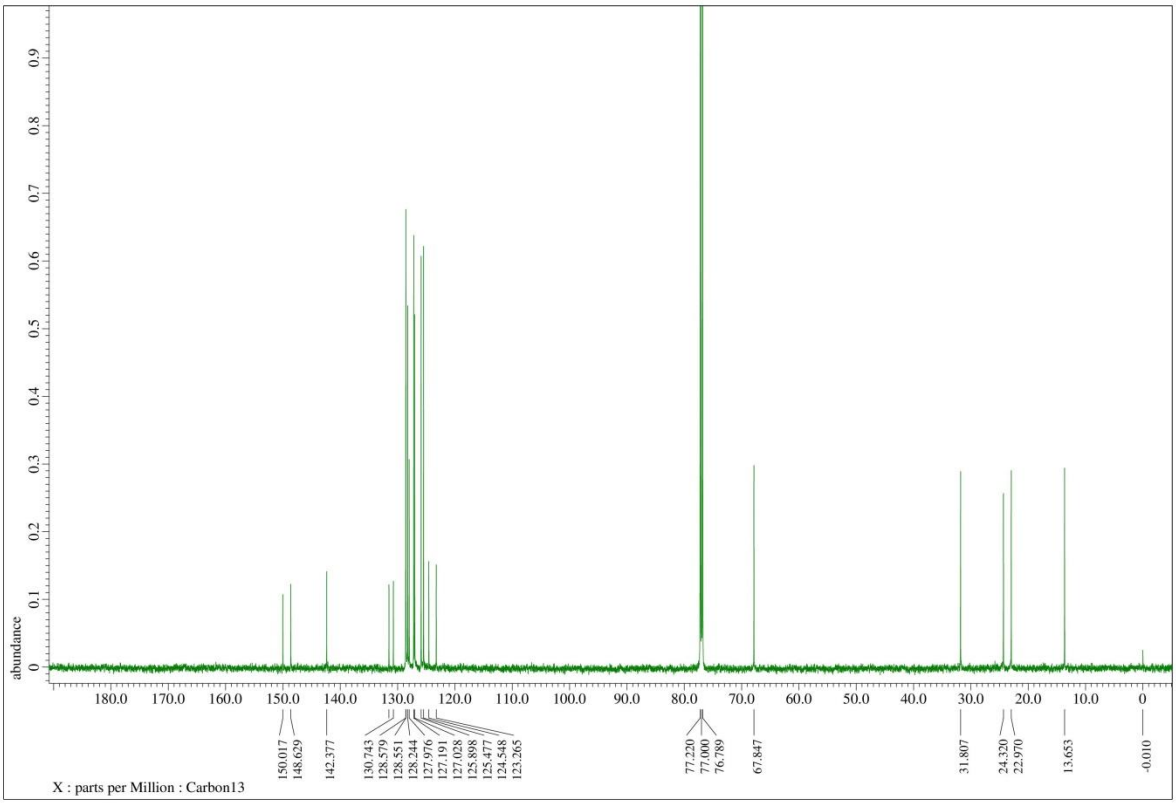

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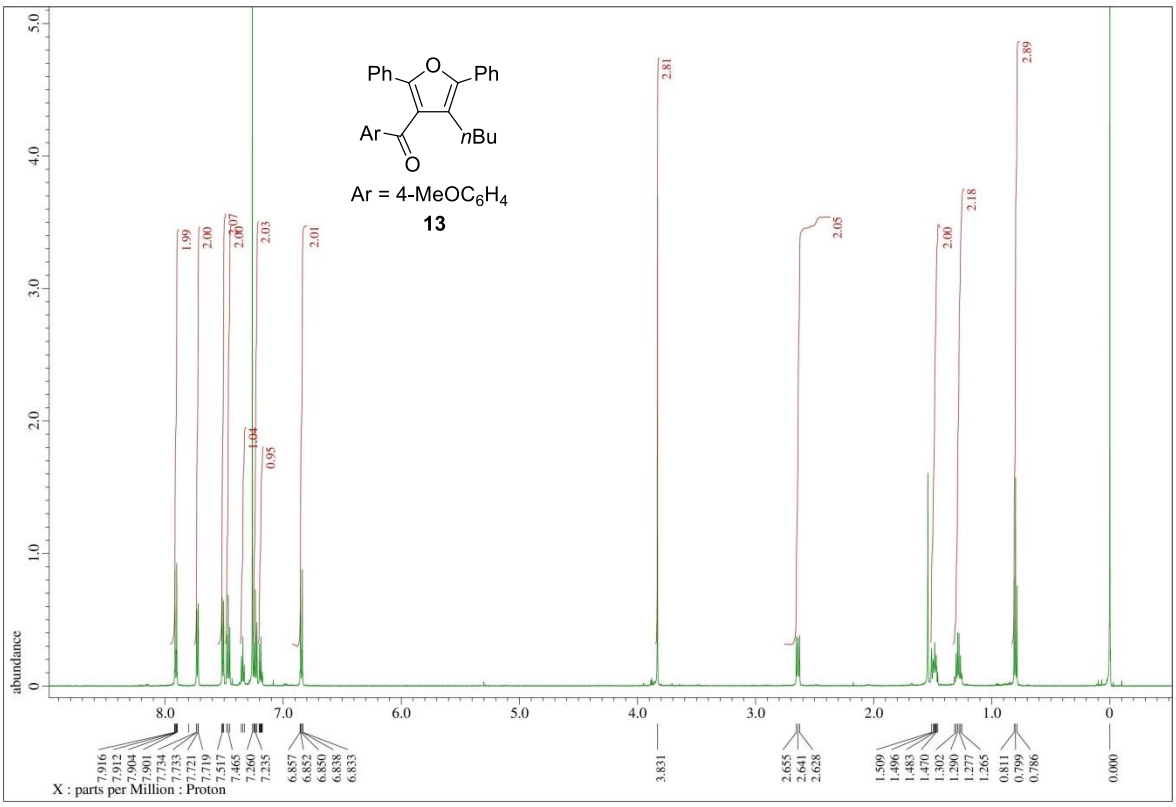

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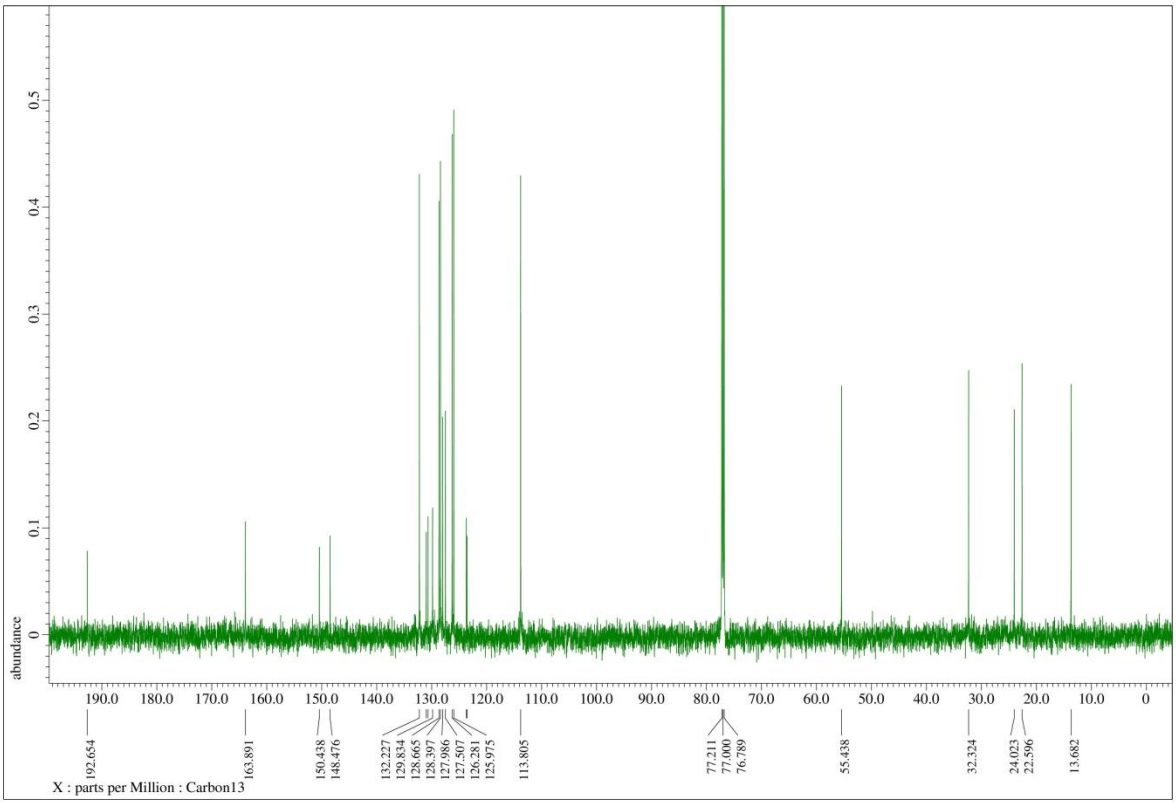

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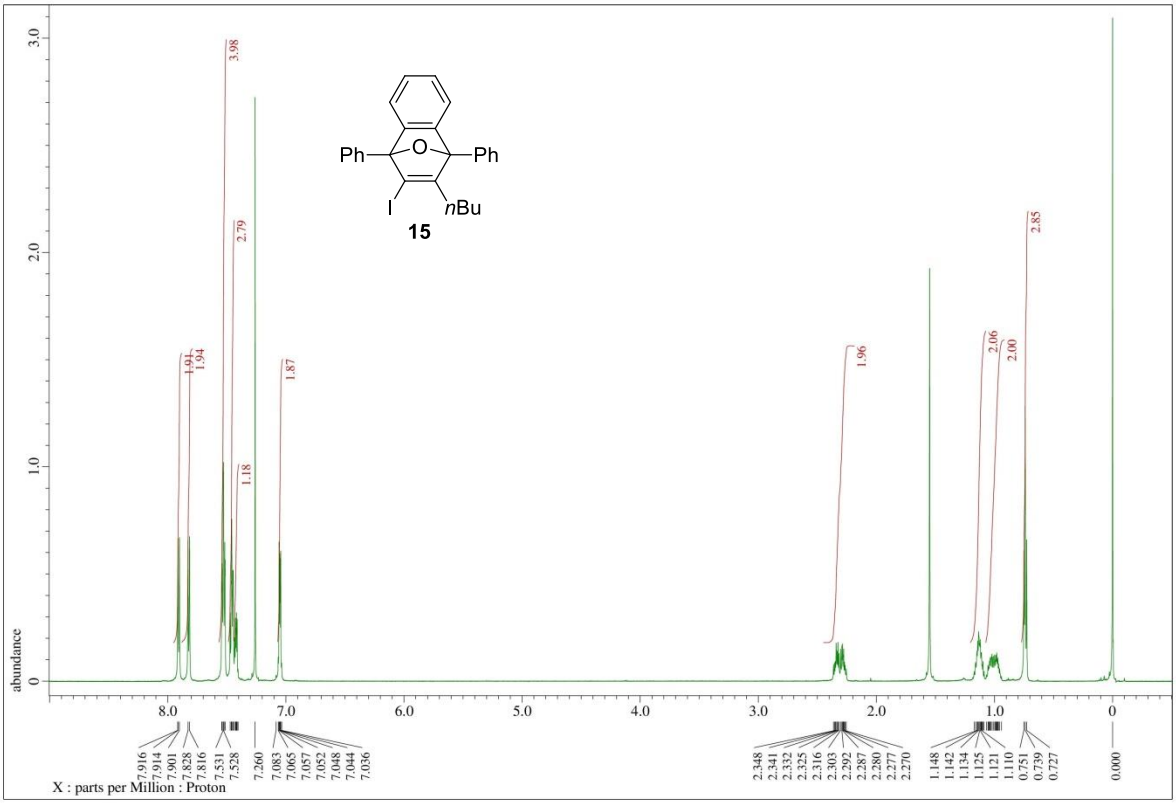

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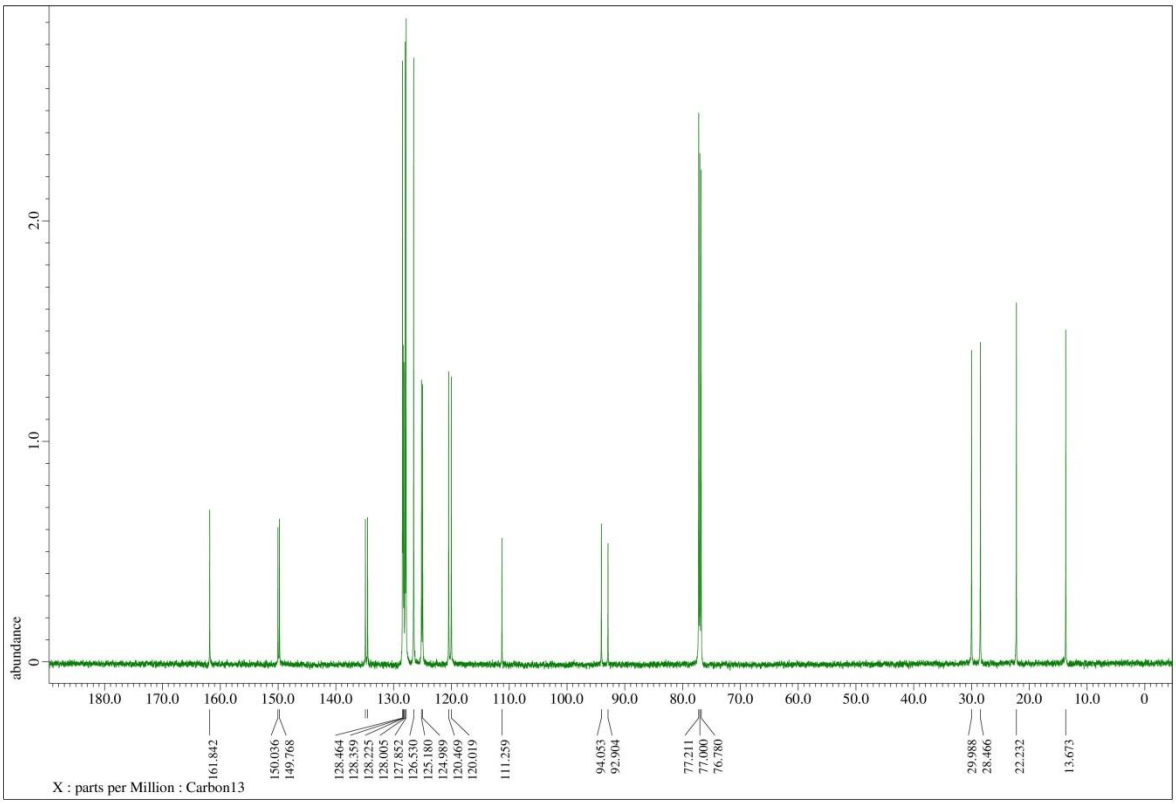

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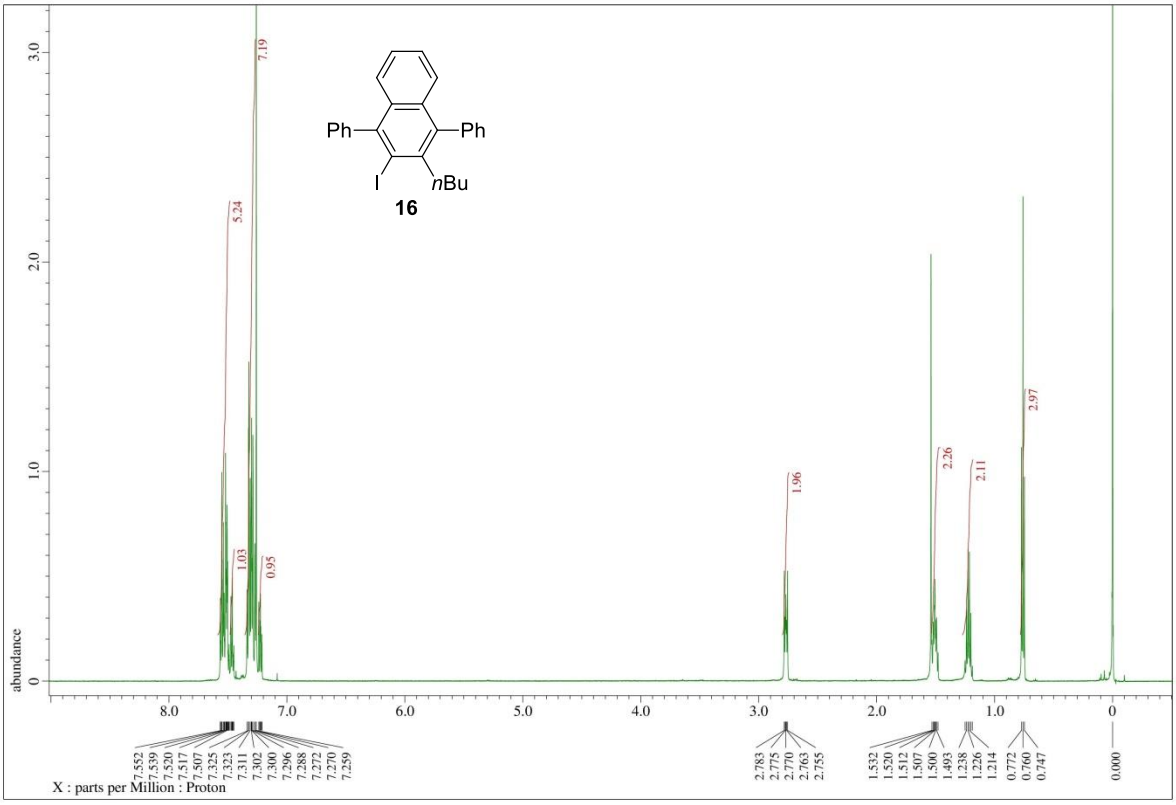
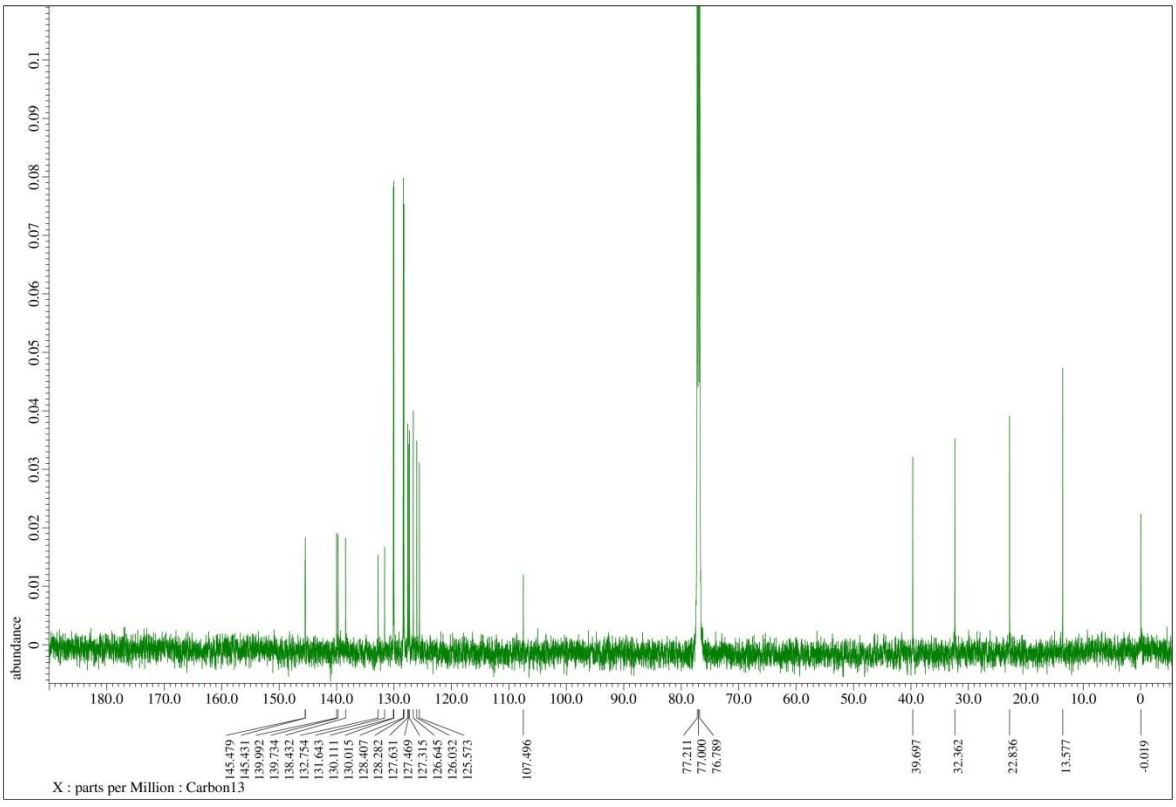


600 MHz, $CDCl_3$



150 MHz, $CDCl_3$