SUPPORTING INFORMATION

Organocatalytic Synthesis of Alkynes

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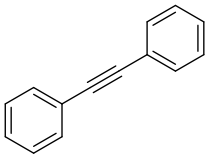
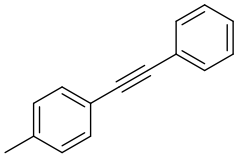
General Methods: All reactions were carried out under dry nitrogen. Anhydrous tetrahydrofuran **(**THF) was purchased from Sigma-Aldrich and directly used without further purification. Unless otherwise stated, reagents were commercially available and used as purchased without further purification. Chemicals were purchased from Sigma-Aldrich, Acros, Alfa Aesar or Matrix Scientific. Aldehydes were newly purchased or distilled under nitrogen atmosphere. The progress of the reactions was monitored by thin-layer chromatography using Whatman Partisil K6F 250 μm precoated 60 Å silica gel plates and visualized by short-wave ultraviolet light as well as by treatment with iodine. Flash chromatography was performed with silica gel (230–400 mesh, Silicycle). The NMR spectra were obtained using a Brüker 500 MHz Fourier-transform NMR spectrometer. Chemical shifts are reported in units of parts per million (ppm) downfield from tetramethylsilane (TMS), and all coupling constants are reported in hertz. Reactions were conducted in 2–5 mL microwave vials that were purchased from VWR International.

Preparation of sulfoxides: Sulfoxides were prepared according to the literature procedures.[1]

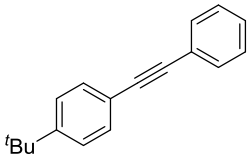
General Procedure for catalysis: To an oven-dried microwave vial equipped with a stir bar was added 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (180 mg, 1.60 mmol, 3.2 equiv), under a nitrogen atmosphere followed by 1.0 mL dry THF. The microwave vial was sealed with a rubber septum and an aluminum cap. The vial was then heated at 80 ^{o}C for 10 minutes for catalyst activation. The benzyl chloride (58 μL, 0.50 mmol) and the benzaldehyde (105 μL, 1.0 mmol) were combined with 1.0 mL dry THF and loaded into a 1.0 mL syringe. This stock solution was added to the reaction vial in a portionwise fashion (0.1 mL every 6 minutes). The reaction mixture was therefore heated for 1 h in total, cooled to room temperature, and opened to air. The reaction mixture was vacuumed filtered through a plug of Celite packed in a 15 mL Buchner funnel into a 100 mL round bottom flask. The pad was then rinsed with 20 mL ethyl acetate and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (64.2 mg, 72% yield) as a white solid. The spectroscopic data match the previously reported data.[2]



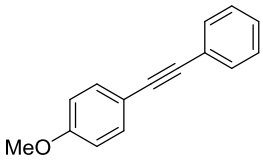
Diphenyl acetylene (3a): The reaction was performed following the General Procedure with 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (180 mg, 1.60 mmol). The stock solution was made with benzyl chloride (**1a**) (58 μL, 0.50 mmol) and benzaldehyde (**2a**) (105 μL, 1.0 mmol). The crude product was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (64.2 mg, 72% yield) as a white solid. The spectroscopic data match the previously reported data.[2]



1-Methyl-4-(phenylethynyl)benzene (3b): The reaction was performed following the General Procedure with 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (180 mg, 1.60 mmol). The stock solution was made with 4-methyl benzyl chloride (**1b**) (63 μL, 0.50 mmol) and benzaldehyde (**2a**) (105 μL, 1.0 mmol). The crude product was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (58.6 mg, 61% yield) as a white solid. The spectroscopic data match the previously reported data.[3]

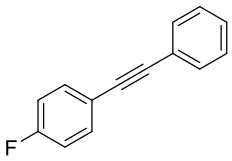


1-(1,1-Dimethylethyl)-4-(2-phenylethynyl)benzene (3c): The reaction was performed following the General Procedure with 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (180 mg, 1.60 mmol). The stock solution was made with 4-*tert*-butyl benzyl chloride (**1c**) (97 μL, 0.50 mmol) and benzaldehyde (**2a**) (105 μL, 1.0 mmol). The crude product was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (80.8 mg, 69% yield) as a white solid. The spectroscopic data match the previously reported data.[4]

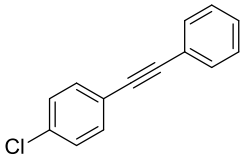


4-Methoxy-1-(phenylethynyl)benzene (3d): The reaction was performed following the General Procedure with 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (180 mg, 1.60 mmol). The stock solution was made with 4-methoxy benzyl chloride (**1d**) (68 μL, 0.50 mmol) and benzaldehyde (**2a**) (105 μL, 1.0 mmol). The crude

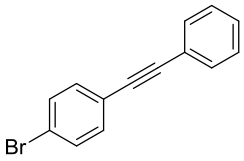
product was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (80.2 mg, 77% yield) as a white solid. The spectroscopic data match the previously reported data.[2]



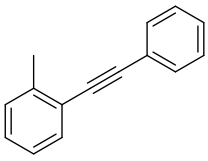
1-Fluoro-4-(phenylethynyl)benzene (3e): The reaction was performed following the General Procedure with 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (180 mg, 1.60 mmol). The stock solution was made with 4-fluoro benzyl chloride (**1e**) (60 μL, 0.50 mmol) and benzaldehyde (**2a**) (105 μL, 1.0 mmol). The crude product was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (68.7 mg, 70% yield) as a white solid. The spectroscopic data match the previously reported data.[2]



1-Chloro-4-(phenylethynyl)benzene (3f): The reaction was performed following the General Procedure with 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (180 mg, 1.60 mmol). The stock solution was made with 4-chloro benzyl chloride (**1f**) (80.5 mg, 0.50 mmol) and benzaldehyde (**2a**) (105 μL, 1.0 mmol). The crude product was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (73.4 mg, 69% yield) as a white solid. The spectroscopic data match the previously reported data.[4]

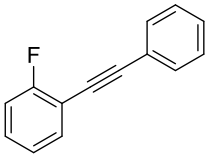


1-Bromo-4-(phenylethynyl)benzene (3g): The reaction was performed following the General Procedure with 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (180 mg, 1.60 mmol). The stock solution was made with 4-bromo benzyl chloride (**1g**) (102.7 mg, 0.50 mmol) and benzaldehyde (**2a**) (105 μL, 1.0 mmol). The crude product was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (93.9 mg, 73% yield) as a white solid. The spectroscopic data match the previously reported data.[4]

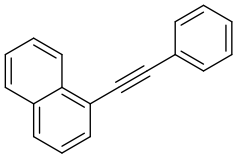


1-Methyl-2-(phenylethynyl)benzene (3h): The reaction was performed following the General Procedure with 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (180 mg, 1.60 mmol). The stock solution was made with 2-methyl benzyl chloride (**1h**) (67 μL, 0.50 mmol) and benzaldehyde (**2a**) (105 μL, 1.0 mmol). The crude product was purified by flash

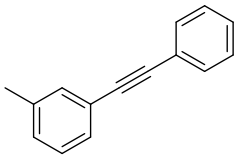
chromatography on silica gel (eluted with hexanes) to give the product (22.1 mg, 23% yield) as a white solid. The spectroscopic data match the previously reported data.[3]



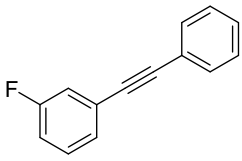
1-Fluoro-2-(phenylethynyl)benzene (3i): The reaction was performed following the General Procedure with 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (180 mg, 1.60 mmol). The stock solution was made with 2-fluoro benzyl chloride (**1i**) (60 μL, 0.50 mmol) and benzaldehyde (**2a**) (105 μL, 1.0 mmol). The crude product was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (61.8 mg, 63% yield) as a white solid. The spectroscopic data match the previously reported data. [5]



1-(Phenylethynyl)naphthalene (3j): The reaction was performed following the General Procedure with 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (180 mg, 1.60 mmol). The stock solution was made with 1-(chloromethyl) naphthalene (**1j**) (88 mg, 0.50 mmol) and benzaldehyde (**2a**) (105 μL, 1.0 mmol). The crude product was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (77.6 mg, 68% yield) as a white solid. The spectroscopic data match the previously reported data.[3]

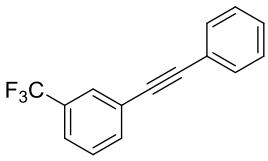


1-Methyl-3-(phenylethynyl)benzene (3k): The reaction was performed following the General Procedure with 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (180 mg, 1.60 mmol). The stock solution was made with 3-methyl benzyl chloride (**1k**) (67 μL, 0.50 mmol) and benzaldehyde (**2a**) (105 μL, 1.0 mmol). The crude product was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (62.5 mg, 65% yield) as a white oil. The spectroscopic data match the previously reported data.[3]

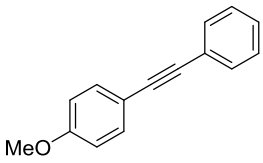


1-Fluoro-3-(phenylethynyl)benzene (3l): The reaction was performed following the General Procedure with 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (180 mg, 1.60 mmol). The stock solution was made with 3-fluoro benzyl chloride (**1l)** (61 μL, 0.50 mmol) and benzaldehyde (**2a**) (105 μL, 1.0 mmol). The crude product was

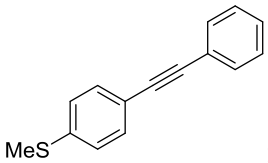
purified by flash chromatography on silica gel (eluted with hexanes) to give the product (60.1 mg, 62% yield) as a white oil. The spectroscopic data match the previously reported data.[6]



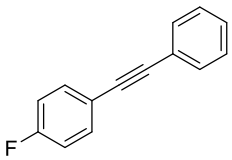
1-(2-Phenylethynyl)-3-(trifluoromethyl)benzene (3m): The reaction was performed following the General Procedure with 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (180 mg, 1.60 mmol). The stock solution was made with 3-(trifluoromethyl) benzyl chloride (**1m**) (78 μL, 0.50 mmol) and benzaldehyde (**2a**) (105 μL, 1.0 mmol). The crude product was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (65.3 mg, 53% yield) as a while oil. The spectroscopic data match the previously reported data.[6]



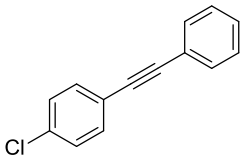
4-Methoxy-1-(phenylethynyl)benzene (3d) (from 4-methoxybenzaldehyde): The reaction was performed following the General Procedure with 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (180 mg, 1.60 mmol). The stock solution was made with benzyl chloride (**1a**) (58 μL, 0.50 mmol) and 4-methoxybenzaldehyde (**2d**) (121 μL, 1.0 mmol). The crude product was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (63.5 mg, 61% yield) as a white solid. The spectroscopic data match the previously reported data.[2]



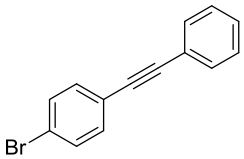
4-Methylthio-1-(phenylethynyl)benzene (3n): The reaction was performed following the General Procedure with 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (180 mg, 1.60 mmol). The stock solution was made with benzyl chloride (**1a**) (58 μL, 0.50 mmol) and 4-methylthiobenzaldehyde (**2n**) (133 μL, 1.0 mmol). The crude product was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (78.5 mg, 70% yield) as a white solid. The spectroscopic data match the previously reported data.[8]



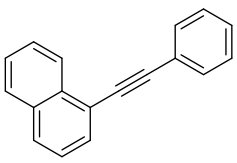
1-Fluoro-4-(phenylethynyl)benzene (3e) (from 4-flurorbenzaldehyde): The reaction was performed following the General Procedure with 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (180 mg, 1.60 mmol). The stock solution was made with benzyl chloride (**1a**) (58 μL, 0.50 mmol) and 4-fluoro benzaldehyde (**2e**) (108 μL, 1.0 mmol). The crude product was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (65.7 mg, 67% yield) as a white solid. The spectroscopic data match the previously reported data.[2]



1-Chloro-4-(phenylethynyl)benzene (3f) (from 4-chlorobenzaldehyde): The reaction was performed following the General Procedure with 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (180 mg, 1.60 mmol). The stock solution was made with benzyl chloride (**1a**) (58 μL, 0.50 mmol) and 4-chlorobenzaldehyde (**2f**) (140.6 mg, 1.0 mmol). The crude product was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (78.7 mg, 74% yield) as a white solid. The spectroscopic data match the previously reported data.[4]

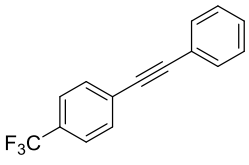


1-Bromo-4-(phenylethynyl)benzene (3g) (from 4-bromobenzaldehyde): The reaction was performed following the General Procedure with 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (180 mg, 1.60 mmol). The stock solution was made with benzyl chloride (**1a**) (58 μL, 0.50 mmol) and 4-bromobenzaldehyde (**2g**) (185 mg, 1.0 mmol). The crude product was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (97.7 mg, 76% yield) as a white solid. The spectroscopic data match the previously reported data.[4]

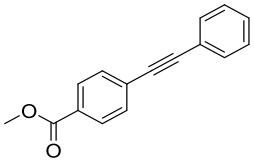


1-(Phenylethynyl)naphthalene (3j) (from 1-naphthaldehyde): The reaction was performed following the General Procedure with 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (180 mg, 1.60 mmol). The stock solution was made with

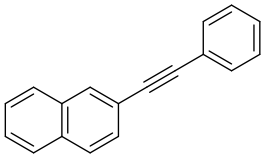
benzyl chloride (**1a**) (58 μL, 0.50 mmol) and 1-naphthaldehyde (**2j**) (136 μL, 1.0 mmol). The crude product was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (92.5 mg, 81% yield) as a white solid. The spectroscopic data match the previously reported data.[3]



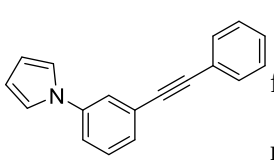
1-(2-Phenylethynyl)-4-(trifluoromethyl)benzene (3o): The reaction was performed following the General Procedure with 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (180 mg, 1.60 mmol). The stock solution was made with benzyl chloride (**1a**) (58 μL, 0.50 mmol) and 4-(trifluoromethyl)benzaldehyde (**2o**) (137 μL, 1.0 mmol). The crude product was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (88.6 mg, 72% yield) as a white solid. The spectroscopic data match the previously reported data.[4]



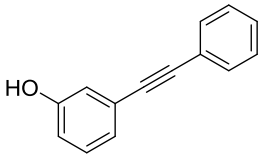
1-(4-Methoxycarbonylphenyl)-2-phenylacetylene (3p): The reaction was performed following the General Procedure with 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (180 mg, 1.60 mmol). The stock solution was made with benzyl chloride (**1a**) (58 μL, 0.50 mmol) and methyl 4-formylbenzoate (**2p**) (164 mg, 1.0 mmol). The crude product was purified by flash chromatography on silica gel (eluted with hexanes:EtOAc = 100:1) to give the product (36.6 mg, 31% yield) as a white solid. The spectroscopic data match the previously reported data.[3]



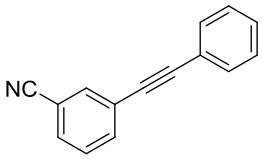
2-(2-Phenylethynyl)naphthalene (3q): The reaction was performed following the General Procedure with 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (180 mg, 1.60 mmol). The stock solution was made with benzyl chloride (**1a**) (58 μL, 0.50 mmol) and 2-naphthaldehyde (**2q**) (156 mg, 1.0 mmol). The crude product was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (85.6 mg, 75% yield) as a white solid. The spectroscopic data match the previously reported data.[6]



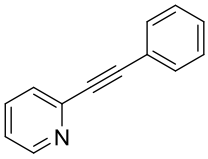
1-[3-(2-Phenylethynyl)phenyl]pyrrole (3r): The reaction was performed following the General Procedure with 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (180 mg, 1.60 mmol). The stock solution was made with benzyl chloride (**1a**) (58 μL, 0.50 mmol) and 3-(1*H*-pyrrol-1-yl)benzaldehyde (**2r**) (172 mg, 1.0 mmol). The crude product was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (92.5 mg, 76% yield) as a white solid; m.p. = 69 –71 ^{o}C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.55 – 7.53 (m, 3H), 7.38 – 7.30 (m, 6H), 7.07 (t, *J* = 2.5 Hz, 2H), 6.34 (t, *J* = 2.5 Hz, 2H) ppm; ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): 140.7, 131.6, 129.5, 128.6, 128.5, 128.4, 124.6, 123.2, 122.9, 120.2, 119.1, 110.7, 90.2, 88.6 ppm; IR (thin film): 3150, 3130, 3100, 3070, 1940, 1603, 1582, 1498, 1488, 1441, 1345, 1257, 1085, 1074, 1028, 887, 788, 757, 727, 691, 684 cm^{-1}; HRMS calculated for $C_{18}H_{14}N$ 244.1126, found 240.1125 $[M+H]^+$.



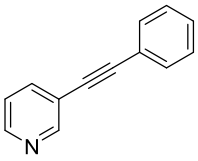
1-Phenyl-2-(m-hydroxyphenyl)acetylene (3s): The reaction was performed following the General Procedure with 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (292 mg, 2.60 mmol). Note that an extra equivalent of base was used to deprotonate the phenolic hydroxyl group. The stock solution was made with benzyl chloride (**1a**) (58 μL, 0.50 mmol) and 3-hydroxybenzaldehyde (**2s**) (122 mg, 1.0 mmol). The crude product was purified by flash chromatography on silica gel (eluted with hexanes:EtOAc = 10:1) to give the product (64.1 mg, 66% yield) as a white solid. The spectroscopic data match the previously reported data.[7]



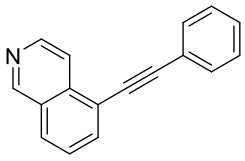
(3-Cyanophenyl)phenylacetylene (3t): The reaction was performed following the General Procedure with 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (180 mg, 1.60 mmol). The stock solution was made with benzyl chloride (**1a**) (58 μL, 0.50 mmol) and 3-cyanobenzaldehyde (**2t**) (131 mg, 1.0 mmol). The crude product was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (69.1 mg, 68% yield) as a white solid. The spectroscopic data match the previously reported data.[8]



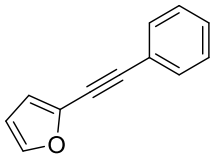
2-(2-phenylethynyl)pyridine (3u): The reaction was performed following the General Procedure with 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (180 mg, 1.60 mmol). The stock solution was made with benzyl chloride (**1a**) (58 μL, 0.50 mmol) and 2-pyridinecarboxaldehyde (**2u**) (96 μL, 1.0 mmol). The crude product was purified by flash chromatography on silica gel (eluted with hexanes:EtOAc = 10:1) to give the product (48.4 mg, 54% yield) as a white solid. The spectroscopic data match the previously reported data.[9]



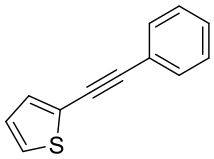
3-(2-Phenylethynyl)pyridine (3v): The reaction was performed following the General Procedure with 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (180 mg, 1.60 mmol). The stock solution was made with benzyl chloride (**1a**) (58 μL, 0.50 mmol) and 3-pyridinecarboxaldehyde (**2v**) (94 μL, 1.0 mmol). The crude product was purified by flash chromatography on silica gel (eluted with hexanes:EtOAc = 10:1) to give the product (62.7 mg, 70% yield) as a white solid. The spectroscopic data match the previously reported data.[3]



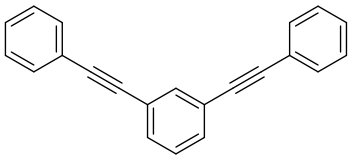
(5-Isoquinolinyl)phenylacetylene (3w): The reaction was performed following the General Procedure with 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (180 mg, 1.60 mmol). The stock solution was made with benzyl chloride (**1a**) (58 μL, 0.50 mmol) and isoquinoline-5-carboxaldehyde (**2w**) (157 mg, 1.0 mmol). The crude product was purified by flash chromatography on silica gel (eluted with hexanes:EtOAc = 10:1) to give the product (83.7 mg, 73% yield) as a white oil. ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.23 (s, 1H), 8.62 (d, J = 6 Hz, 1H), 8.14 (d, J = 6 Hz, 1H), 7.87 (d, J = 7.5 Hz, 2H), 7.62 (dd, J = 7.5, 2.5 Hz, 2H), 7.51 (t, J = 8.5 Hz, 1H), 7.38 – 7.36 (m, 3H) ppm; ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): 152.6, 143.8, 135.8, 133.8, 131.5, 128.6, 128.3, 128.2, 127.8, 126.5, 122.6, 120.1, 118.7, 95.3, 85.8 ppm; IR (thin film): 3062, 2215, 1612, 1598, 1579, 1571, 1492, 1442, 1381, 1368, 1024, 829, 755, 691 cm^{-1}; HRMS calculated for $C_{17}H_{12}N$ 230.0969, found 230.0975 $[M+H]^+$.



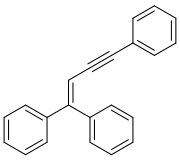
2-(2-Phenylethynyl)furan (3x): The reaction was performed following the General Procedure with 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (180 mg, 1.60 mmol). The stock solution was made with benzyl chloride (**1a**) (58 μL, 0.50 mmol) and 2-furaldehyde (**2x**) (83 μL, 1.0 mmol). The crude product was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (67.3 mg, 80% yield) as a pale yellow oil. The spectroscopic data match the previously reported data.[10]



2-(2-Phenylethynyl)thiophene (3y): The reaction was performed following the General Procedure with 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (180 mg, 1.60 mmol). The stock solution was made with benzyl chloride (**1a**) (58 μL, 0.50 mmol) and 2-thiophenecarboxaldehyde (**2y**) (94 μL, 1.0 mmol). The crude product was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (70.9 mg, 77% yield) as a white solid. The spectroscopic data match the previously reported data.[3]

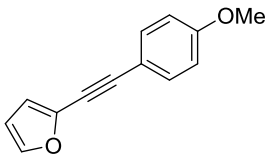


1,3-Bis(2-phenylethynyl)benzene (5a): The reaction was performed following the General Procedure with 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (360 mg, 3.20 mmol). The stock solution was made with 1,3-bis(chloromethyl)benzene (**4a**) (88 mg, 0.50 mmol) and benzaldehyde (**2a**) (210 μL, 2.0 mmol). The crude product was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (58.5 mg, 42% yield) as a white solid. The spectroscopic data match the previously reported data.[13]

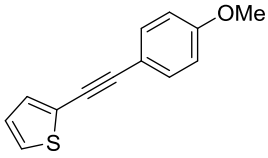


1,1',4-Triphenylbut-3-en-1-yne (5b): The reaction was performed following the General Procedure with 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (180 mg, 1.60 mmol). The stock solution was made with 1,1-diphenyl-3-chloro-1-propene (**4b**) (115

mg, 0.50 mmol) and benzaldehyde (**2a**) (105 μL, 1.0 mmol). The crude product was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (64.5 mg, 46% yield) as a white solid. The spectroscopic data match the previously reported data.[14]

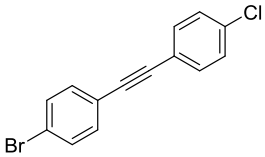


2-(2-(4-Methoxyphenyl)ethynyl)furan (3dx) (gram scale reaction): To an oven-dried Schlenk flask equipped with a stir bar was added 2-phenylethyl phenyl sulfoxide (184 mg, 0.80 mmol) and KO^{t}Bu (2.87 g, 25.6 mmol, 3.2 equiv) under nitrogen atmosphere, followed by 17.0 mL dry THF via syringe. The Schlenk flask neck was equipped with a condenser, whose top was connected to a nitrogen line, with side arm closed. And the flask was then heated at 80 ^{o}C for 10 minutes for catalyst activation. 4-Methoxy benzyl chloride **1d** (1.08 mL, 8.0 mmol) and 2-furaldehyde **2x** (1.33 mL, 16.0 mmol) were diluted to 15.0 mL with dry THF and loaded into a syringe. This stock solution was added to the vial every 6 minutes through the sidearm. The reaction was heated for 1 h and then cooled to room temperature, opened to air. The reaction mixture was vacuumed filtered through a plug of Celite packed in a 15 mL Buchner funnel into a 100 mL round bottom flask. The pad of Celite was then rinsed with 50 mL ethyl acetate, the filtrates combined, and the volatile materials removed under reduced pressure. The crude product was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (1.23 g, 78% yield) as a pale yellow oil. The spectroscopic data match the previously reported data.[11]

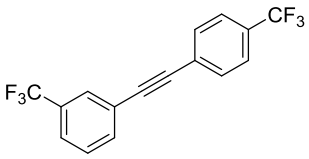


2-(2-(4-Methoxyphenyl)ethynyl)thiophene (3dy) (gram scale sythesis): To an oven-dried Schlenk flask equipped with a stir bar was added 2-phenylethyl phenyl sulfoxide (184 mg, 0.80 mmol) and KO^{t}Bu (2.87 g, 25.6 mmol, 3.2 equiv), under nitrogen atmosphere followed by 17.0 mL dry THF via syringe. The Schlenk flask neck was equipped with a condenser, whose top was connected to a nitrogen line, with side arm closed. And the flask was then heated at 80 ^{o}C for 10 minutes for catalyst activation. 4-Methoxy benzyl chloride **1d** (1.08 mL, 8.0

mmol) and 2-thiophenecarboxaldehyde **2y** (1.50 mL, 16.0 mmol) was diluted to 15.0 mL with dry THF and loaded to a syringe as the stock solution. 1.5 mL stock solution was added to the vial every 6 minutes through the sidearm. The reaction was heated for 1 h and then cooled to room temperature, opened to air. The reaction mixture was vacuumed filtered through a plug of Celite packed in a 15 mL Buchner funnel into a 100 mL round bottom flask. The pad was then rinsed with 50 mL ethyl acetate and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (1.28 g, 75% yield) as a white solid. The spectroscopic data match the previously reported data.[11]

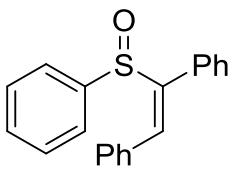


1-(4-Bromophenyl)-2-(4-chlorophenyl)ethyne (3gf) (gram scale synthesis): To an oven-dried 100 Schlenk flask equipped with a stir bar was added 2-phenylethyl phenyl sulfoxide (184 mg, 0.80 mmol) and KO^{t}Bu (2.87 g, 25.6 mmol, 3.2 equiv) under nitrogen atmosphere, followed by 17.0 mL dry THF via syringe. The Schlenk flask neck was equipped with a condenser, whose top was connected to a nitrogen line, with side arm closed. And the flask was then heated at 80 ^{o}C for 10 minutes for catalyst activation. 4-Bromo benzyl chloride **1g** (1.64 g, 8.0 mmol) and **2f** 4-chlorobenzaldehyde (2.25 g, 16.0 mmol) was diluted to 15.0 mL with dry THF and loaded to a syringe as the stock solution. 1.5 mL stock solution was added to the vial every 6 minutes through the sidearm. The reaction was heated for 1 h and then cooled to room temperature, opened to air. The reaction mixture was vacuumed filtered through a plug of Celite packed in a 15 mL Buchner funnel into a 100 mL round bottom flask. The pad was then rinsed with 50 mL ethyl acetate and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (2.06 g, 71% yield) as a white solid. The spectroscopic data match the previously reported data.[12]



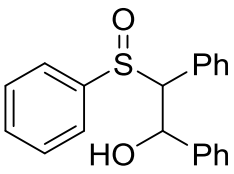
1-(3-Trifluoromethyl)-2-(4-trifluorophenyl)ethyne (3mo) (gram scale synthesis): To an oven-dried Schlenk flask equipped with a stir bar was added 2-phenylethyl phenyl sulfoxide (184 mg, 0.80 mmol) and KO^{t}Bu (2.87 g, 25.6 mmol, 3.2 equiv), under nitrogen atmosphere followed by 17.0 mL dry THF via syringe. The Schlenk flask neck was equipped with a condenser, whose top was connected to a nitrogen line, with side arm closed. And the flask was then heated at 80 ^{o}C for 10 minutes for catalyst activation. 3-Trifluoromethyl benzyl chloride **1m** (1.24 mL, 8.0 mmol) and 4-(trifluoromethy)lbenzaldehyde **2o** (2.19 mL, 16.0 mmol) was diluted to 15.0 mL with dry THF and loaded to a syringe as the stock solution. 1.5 mL stock solution was added to the vial every 6 minutes through the sidearm. The reaction was heated for 1 h and then cooled to room temperature, opened to air. The reaction mixture was vacuumed filtered through a plug of Celite packed in a 15 mL Buchner funnel into a 100 mL round bottom flask. The pad was then rinsed with 50 mL ethyl acetate and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (1.41 g, 56% yield) as a white oil. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.80 (s, 1H), 7.68 (d, J = 7.5 Hz, 1H), 7.63 – 7.56 (m, 5H), 7.45 (t, J = 7.5 Hz, 1H), 7.51 (t, J = 4 Hz, 1H) ppm; ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): 134.8, 131.9, 131.1 (q, $J_{C\text{-}F}$ = 31.2 Hz), 130.4 (q, $J_{C\text{-}F}$ = 32.5 Hz), 129.0, 128.5 (q, $J_{C\text{-}F}$ = 3.7 Hz), 126.5, 125.4 (q, $J_{C\text{-}F}$ = 3.7 Hz), 125.2 (q, $J_{C\text{-}F}$ = 3.7 Hz), 123.9 (q, $J_{C\text{-}F}$ = 270 Hz), 123.7 (q, $J_{C\text{-}F}$ = 270 Hz), 123.6 ppm; IR (thin film): 3076, 1616, 1434, 1343, 1324, 1129, 1169, 1067, 842, 902, 707, 695 cm^{-1}; HRMS calculated for $C_{16}H_8F_6$ 314.0530, found 314.0535 [M]$^+$.

Mechanism Study



(*Z*)-1,2-Diphenyl-1-(phenylsulphinyl)ethene (I) : (*Z*)-1,2-diphenyl-1-(phenylthio)ethene was synthesized according to literature[15] with diphenyl acetylene (3.56 g, 20 mmol) and thiophenol (2.04 mL, 20 mmol). The resulting sulfide was

oxidized with sodium periodate (4.28 g, 20 mmol) in 70 mL MeOH:H_2O = 19:1 for 12 h, giving (*Z*)-1,2-diphenyl-1-(phenylsulphinyl)ethene (3.16 g, 52%); m.p. = 125 – 127 ^oC. ^1H NMR (500 MHz, $CDCl_3$): δ 7.42 (s, 1H), 7.38 – 7.29 (m, 6H), 7.25 (t, *J* = 7.5 Hz, 2H), 7.20 – 7.15 (m, 5H), 6.88 (d, *J* = 7.5 Hz, 2H) ppm; $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$): 145.5, 142.5, 133.8, 131.8, 131.0, 129.7, 129.6, 129.5, 128.75, 128.72, 128.68, 128.64, 128.3, 125.3 ppm; IR (thin film): 3056, 1443, 1082, 1053, 750, 692 cm^{-1}; HRMS calculated for $C_{20}H_{17}OS$ 305.1000, found 314.0997 $[M+H]^+$. The vinyl sulfoxide isolated from reaction was demonstrated as the same compound with (*Z*)-1,2-diphenyl-1-(phenylsulphinyl)ethene by comparing ^1H and $^{13}C\{^1H\}$ NMR spectra.



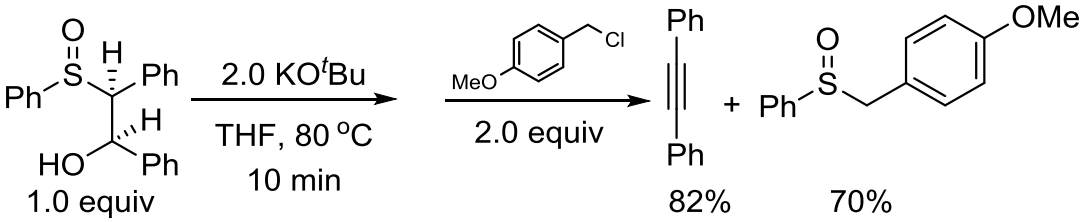
2-Phenylsulfinyl-1,2-diphenyl-1-ethanol (H) : Both threo- and erythro-2-phenylsulfinyl-1,2-diphenyl-1-ethanol were synthesized according to literature.[16]

Resting state study:

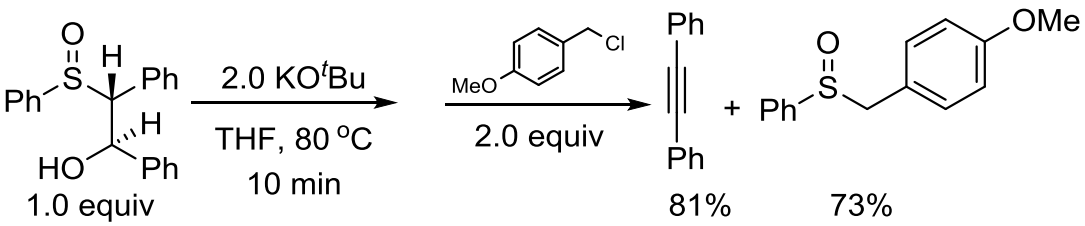
To an oven-dried microwave vial equipped with a stir bar was added 2-phenylethyl phenyl sulfoxide (23 mg, 0.10 mmol) and KO^{t}Bu (180 mg, 1.60 mmol, 3.2 equiv), under a nitrogen atmosphere followed by 1.0 mL dry THF. The microwave vial was sealed with a rubber septum and an aluminum cap. The vial was then heated at 80 ^oC for 10 minutes for catalyst activation. The benzyl chloride (58 μL, 0.50 mmol) and the benzaldehyde (105 μL, 1.0 mmol) were combined with 1.0 mL dry THF and loaded into a 1.0 mL syringe. This stock solution was added to the reaction vial in a portionwise fashion (0.1 mL every 6 minutes). At the 12th minute and the first 3 doses of stock solution, the rest stock solution was injected into reaction mixture at once and the reaction was quenched 3 min later. The reaction mixture was filtered through Celite The reaction mixture was vacuumed filtered through a plug of Celite packed in a 15 mL Buchner funnel into a 100 mL round bottom flask. The pad was then rinsed with 20 mL ethyl acetate and the solvent was removed under reduced pressure. The crude product was purified by flash

chromatography on silica gel (eluted with hexanes:EtOAc = 4:1) to give (*Z*)-1,2-diphenyl-1-(phenylsulphinyl)ethene (18.8 mg, 62% yield) as a white solid and benzyl phenyl sulfoxide (7.2 mg, 33% yield) as a white solid.

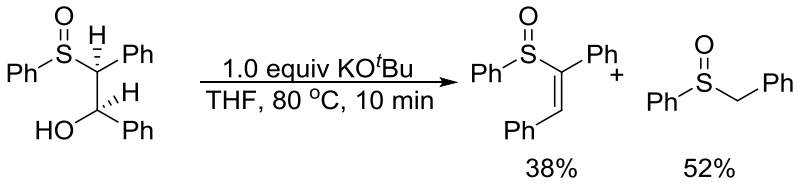
Studies of Reaction Intermediates.



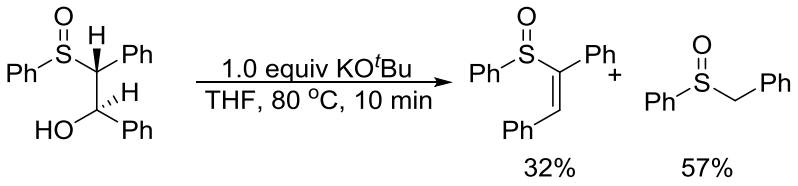
To an oven-dried microwave vial equipped with a stir bar was added *erythro*-2-phenylsulfinyl-1,2-diphenyl-1-ethanol (33.2 mg, 0.1 mmol) and KO^{t}Bu (22.4 mg, 0.20 mmol, 2.0 equiv) under nitrogen atmosphere, followed by 2.0 mL dry THF via syringe. The microwave vial was sealed with rubber septum and aluminum cap, and heated to 80 °C for 10 min. The sealed vial was cooled to room temperature, quenched with 4-methoxy benzyl chloride (28 μL, 0.20 mmol, 2.0 equiv), and opened to air. The reaction mixture was passed through a short pad of silica gel, the pad was rinsed with 10 mL ethyl acetate, and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography on silica gel (eluted with hexanes) to give diphenyl acetylene (14.6 mg, 82% yield) as a white solid. The column was then eluted with hexanes:EtOAc = 2:1 to give 4-methoxy-benzyl phenyl sulfoxide (17.2 mg, 70% yield) as a white solid.



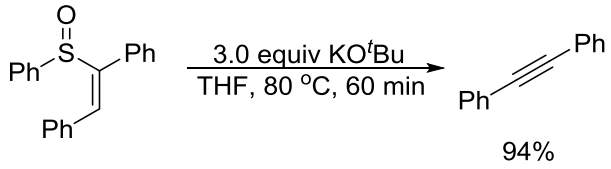
To an oven-dried microwave vial equipped with a stir bar was added *erythro*-2-phenylsulfinyl-1,2-diphenyl-1-ethanol (33.2 mg, 0.1 mmol) and KO^{t}Bu (22.4 mg, 0.20 mmol, 2.0 equiv) under nitrogen atmosphere, followed by 2.0 mL dry THF via syringe. The microwave vial was sealed with rubber septum and aluminum cap, and heated to 80 ^{o}C for 10 min. The sealed vial was cooled to room temperature, quenched with 4-methoxy benzyl chloride (28 μL, 0.20 mmol, 2.0 equiv), and opened to air. The reaction mixture was passed through a short pad of silica gel, the pad was rinsed with 10 mL ethyl acetate, and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography on silica gel (eluted with hexanes) to give diphenyl acetylene (14.4 mg, 81% yield) as a white solid. The column was then eluted with hexanes:EtOAc = 2:1 to give 4-methoxy-benzyl phenyl sulfoxide (18.0 mg, 73% yield) as a white solid.



To an oven-dried microwave vial equipped with a stir bar was added erythro-2-phenylsulfinyl-1,2-diphenyl-1-ethanol (33.2 mg, 0.1 mmol) and 1 equiv. KO^{t}Bu (11.2 mg, 0.10 mmol, 1.0 equiv) under a nitrogen atmosphere, followed by 2.0 mL dry THF via syringe. The microwave vial was sealed with rubber septum and aluminum cap, and heated to 80 ^{o}C for 10 min. The sealed vial was cooled to room temperature and opened to air. The reaction mixture was passed through a short pad of silica gel. The pad was then rinsed with 10 mL ethyl acetate and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography on silica gel (eluted with hexanes:EtOAc = 4:1) to give the (Z)-1,2-diphenyl-1-(phenylsulphinyl)ethene (11.6 mg, 38% yield) as a white solid. The column was then eluted with hexanes:EtOAc = 2:1 to give benzyl phenyl sulfoxide (11.2 mg, 52 %) as a white solid.



To an oven-dried microwave vial equipped with a stir bar was added threo-2-phenylsulfinyl-1,2-diphenyl-1-ethanol (33.2 mg, 0.1 mmol) and KO^{t}Bu (11.2 mg, 0.10 mmol, 1.0 equiv), under nitrogen atmosphere followed by 2.0 mL dry THF. The microwave vial was sealed with rubber septum and aluminum cap, and heated to 80 °C for 10 min. The sealed vial was cooled to room temperature and opened to air. The reaction mixture was passed through a short pad of silica gel. The pad was then rinsed with 10 mL ethyl acetate and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography on silica gel (eluted with hexanes:EtOAc = 4:1) to give the (*Z*)-1,2-diphenyl-1-(phenylsulphinyl)ethene (9.7 mg, 32% yield) as a white solid. The column was then eluted with hexanes:EtOAc = 2:1 to give benzyl phenyl sulfoxide (12.3 mg, 57 %) as a white solid.



To an oven-dried microwave vial equipped with a stir bar was added (*Z*)-1,2-diphenyl-1-(phenylsulphinyl)ethene (30.4 mg, 0.1 mmol) and KO^{t}Bu (33.6 mg, 0.30 mmol, 3.0 equiv) under a nitrogen atmosphere, followed by 2.0 mL dry THF via syringe. The microwave vial was sealed with rubber septum and aluminum cap, and heated to 80 °C for 60 min. The sealed vial was cooled to room temperature and opened to air. The reaction mixture was passed through a short pad of silica gel. The pad was then rinsed with 10 mL ethyl acetate and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography on silica gel (eluted with hexanes) to give diphenyl acetylene (16.7 mg, 94% yield) as a white solid.

High-throughput Experimentation Screenings

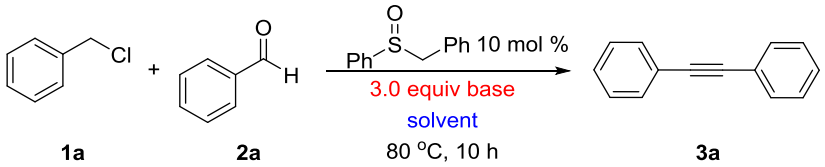
Set up:

Experiments were set up in a glove box under a nitrogen atmosphere. A 24-well aluminum block containing 1 mL glass vials was dosed with 1 μmol benzyl phenyl sulfoxide in THF. The solvent was removed to dryness using a GeneVac. Next, base (30 μmol) in THF was added to the vials. The solvent was again removed to dryness using a GeneVac and a parylene stir bar was then added to each reaction vial. Benzyl chloride (10 μmol/reaction), benzaldehyde (20 μmol/reaction) and biphenyl (1 μmol/reaction, used as an internal standard to measure HPLC yields) were then dosed together into each reaction vial as a solution in each solvent (100 μL, 0.1 M). The 24-well plate was then sealed and stirred for 10 h at 80 ^{o}C.

Work up:

The plate was cooled to room temperature. Upon opening the plate to air, 500 μL of acetonitrile was added into each vial. The plate was covered again and the vials stirred for 10 min to ensure good homogenization. Into a separate 24-well LC block was added 700 μL of acetonitrile, followed by 40 μL of the diluted reaction mixtures. The LC block was then sealed with a silicon-rubber storage mat and mounted on an automated HPLC instrument for analysis.

Base and solvent screening



Base: LiO^{t}Bu, NaO^{t}Bu, KO^{t}Bu, LiN(SiMe$_3$)$_2$, NaN(SiMe$_3$)$_2$, KN(SiMe$_3$)$_2$.

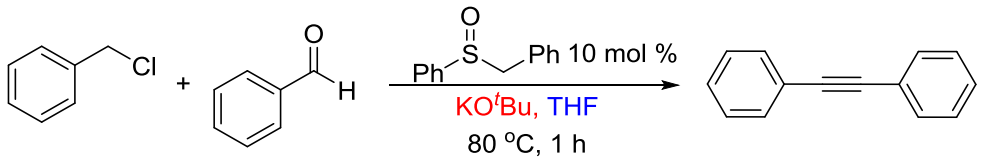
Solvent: THF, DME, dioxane, CPME.

Well	Base	Solvent	Prod/IS	AY 3a (%)
A01	LiO^tBu	THF	0	--
B01		DME	0	--
C01		Dioxane	0	--
D01		CPME	0	--
A02	NaO^tBu	THF	0	--
B02		DME	0	--
C02		Dioxane	0	--
D02		CPME	0	--
A03	KO^tBu	THF	1.26	20
B03		DME	0.96	15
C03		Dioxane	1.15	18
D03		CPME	0	--
A04	$LiN(SiMe_3)_2$	THF	0	--
B04		DME	0	--
C04		Dioxane	0	--
D04		CPME	0	--

A05	$NaN(SiMe_3)_2$	THF	0	--
B05		DME	0	--
C05		Dioxane	0	--
D05		CPME	0	--
A06	$KN(SiMe_3)_2$	THF	0	--
B06		DME	0	--
C06		Dioxane	0	--
D06		CPME	0	--

Ratio screening:

Reactions were set up on lab scale following the General Procedure. The stock solutions of benzyl chloride and benzaldehyde were prepared in different ratios as listed below.



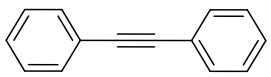
1a **2a** **3a**

Entry	**1a : 2a : base**	**AY 3a (%)**
1	1:2:3	67
2	2:1:3	37

3	1:2:4	58
4	1:3:3	65

NMR

Diphenyl acetylene (3a)



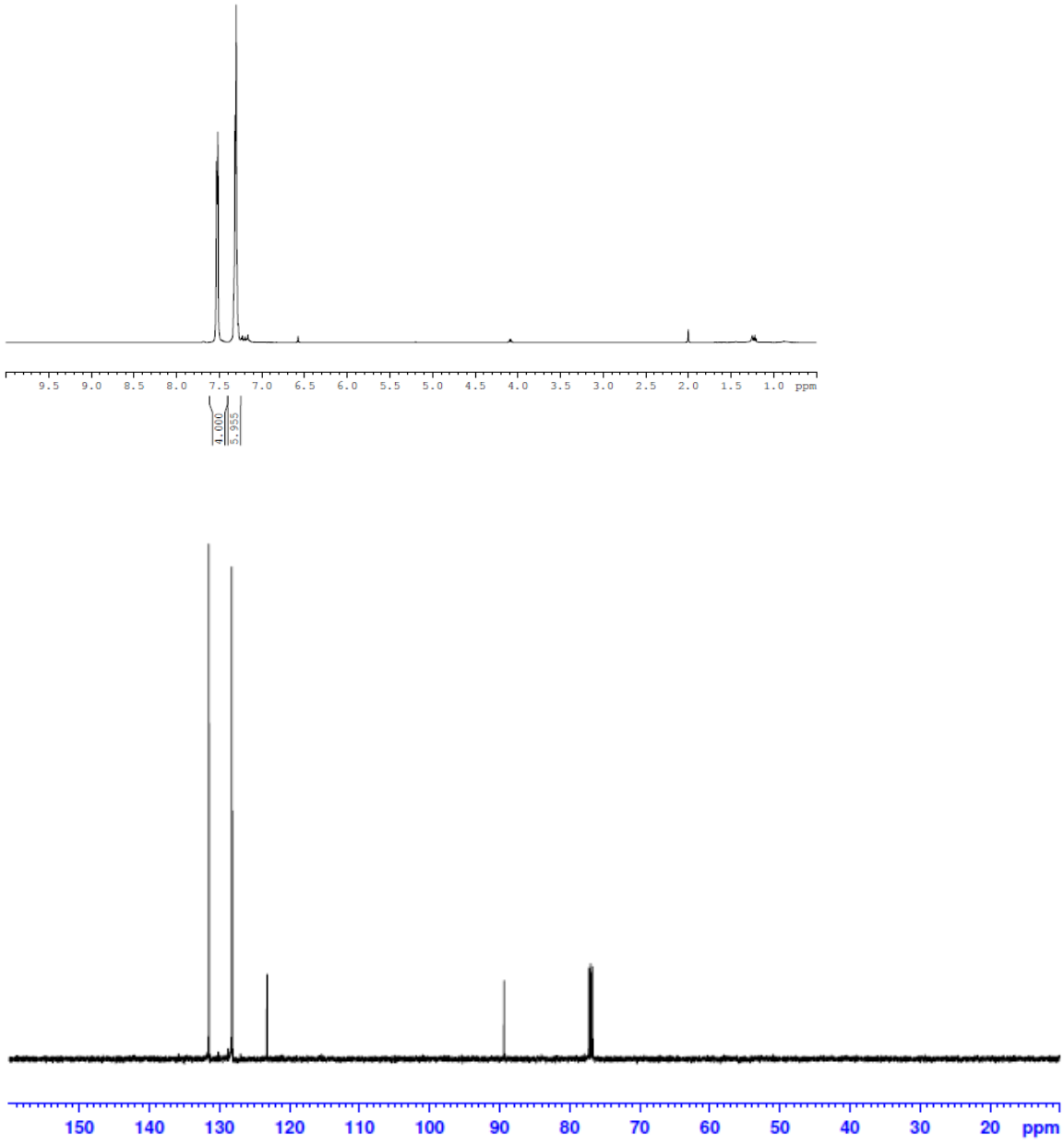
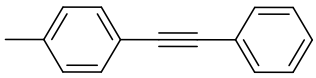


Figure S1. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of 3a in $CDCl_3$.

1-Methyl-4-(phenylethynyl)benzene (3b)



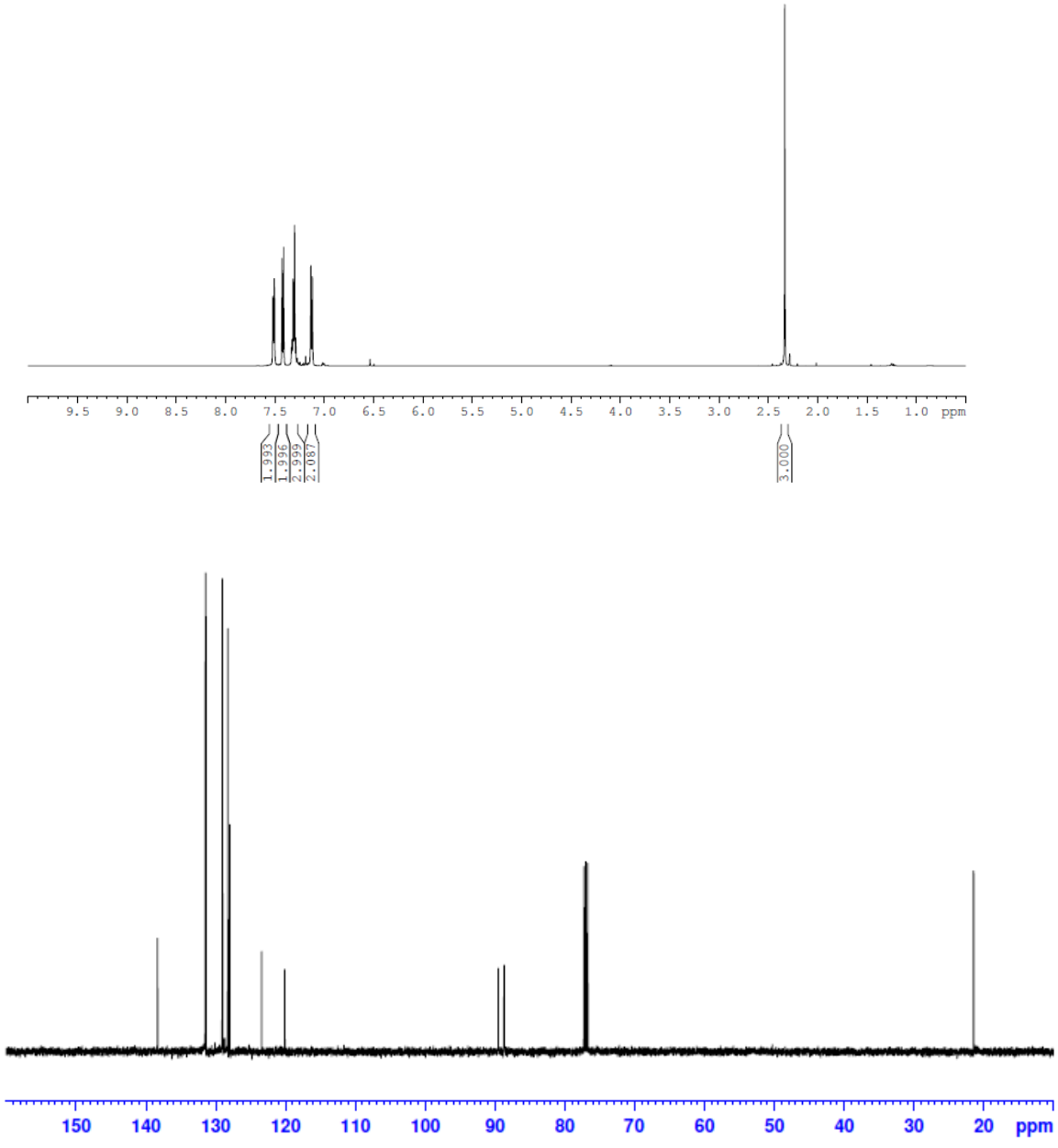
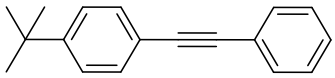


Figure S2. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of 3b in $CDCl_3$.

1-(1,1-Dimethylethyl)-4-(2-phenylethynyl)benzene (3c)



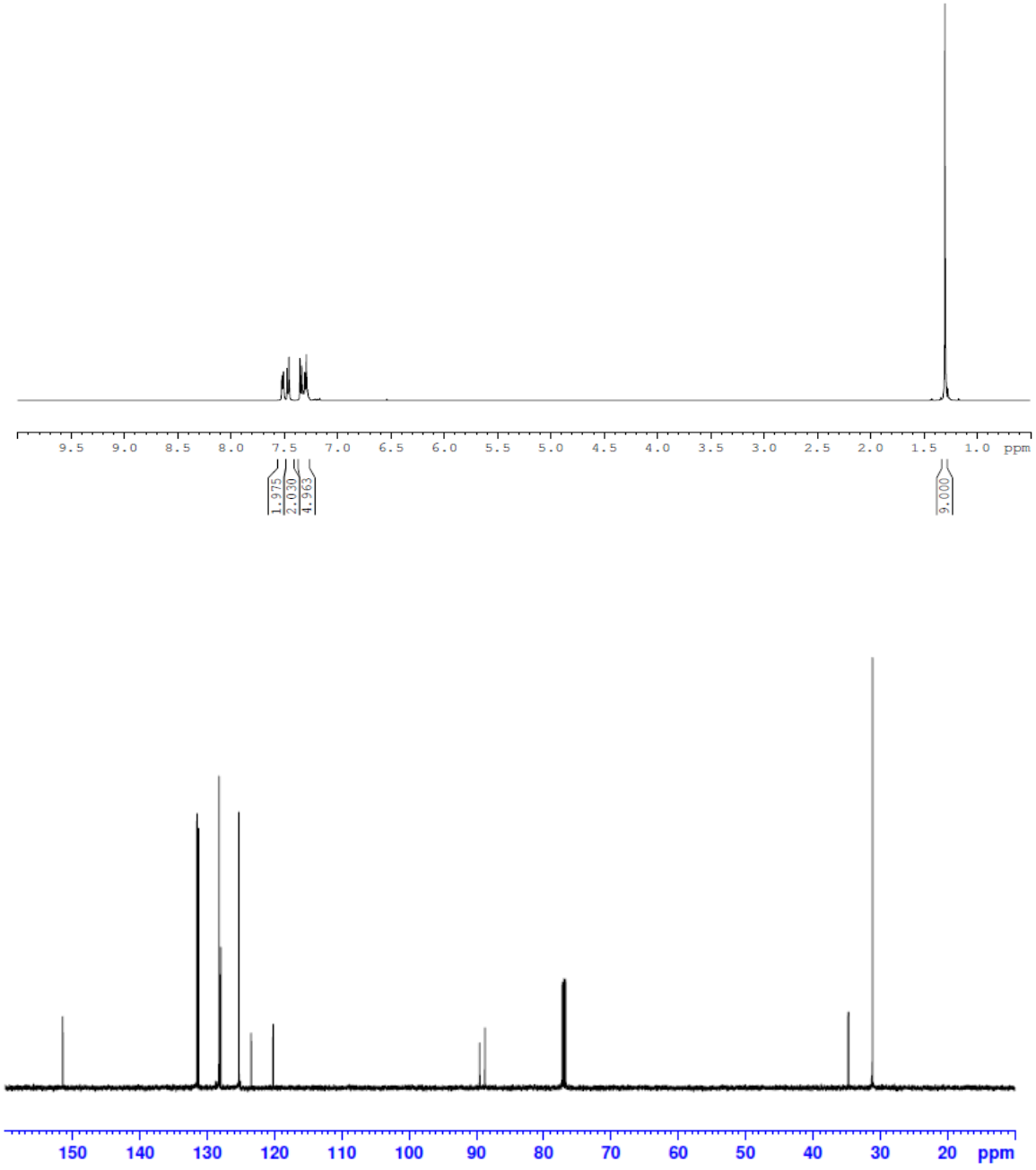
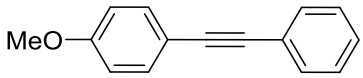


Figure S3. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of 3c in $CDCl_3$.

4-Methoxy-1-(phenylethynyl)benzene (3d)



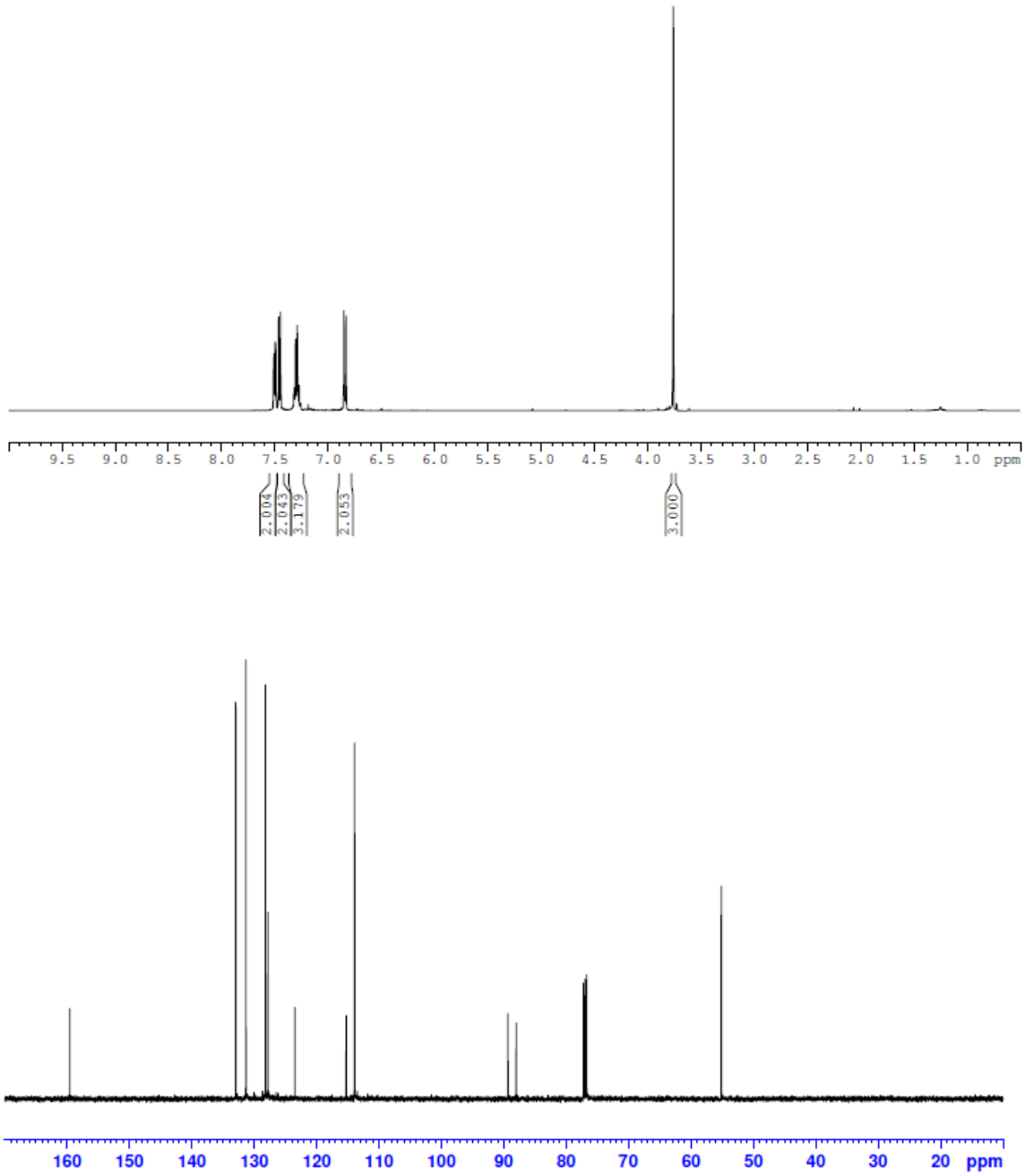
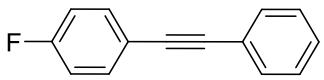


Figure S4. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of 3d in $CDCl_3$.

1-Fluoro-4-(phenylethynyl)benzene (3e)



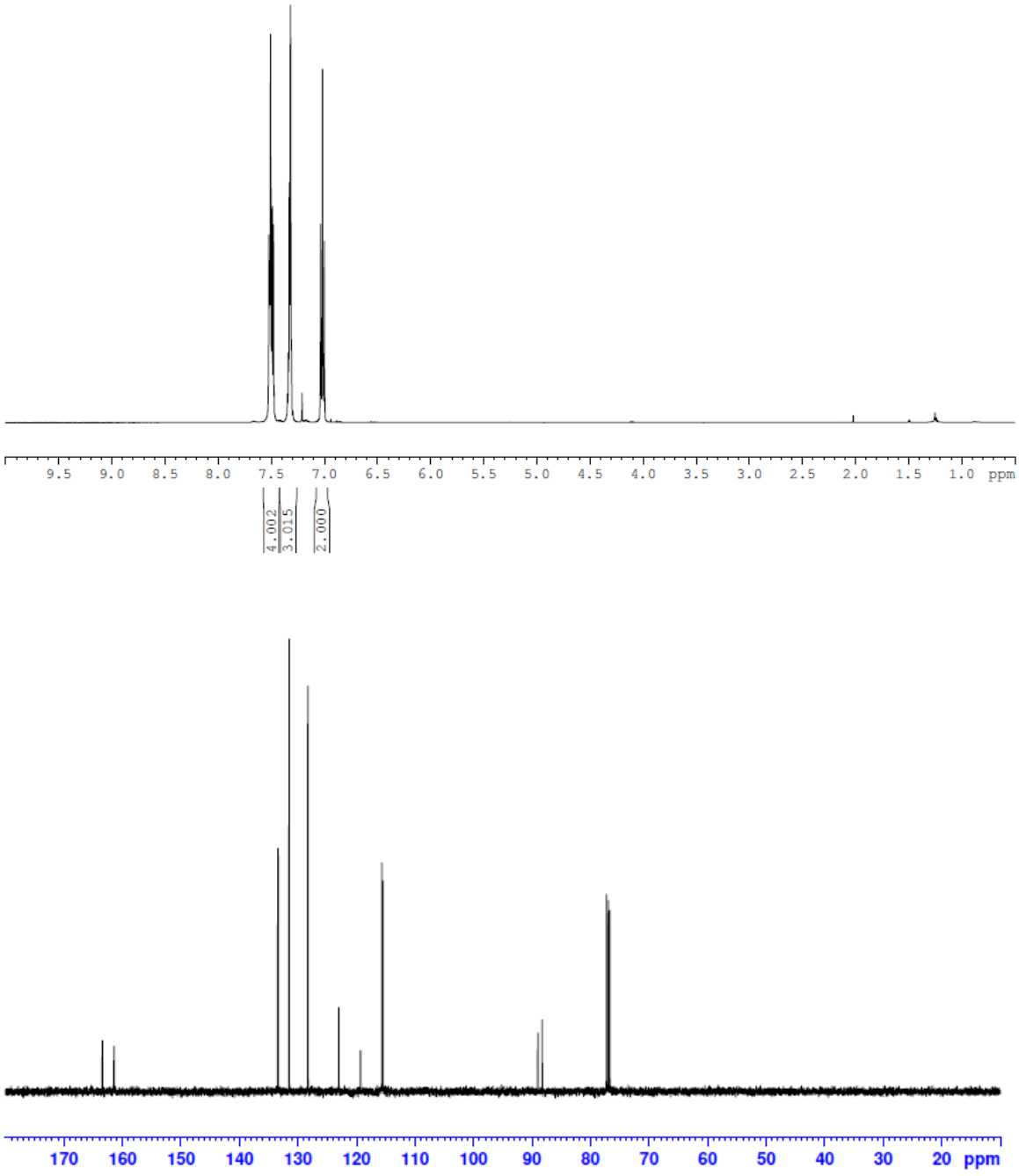


Figure S5. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of 3e in $CDCl_3$.

1-Chloro-4-(phenylethynyl)benzene (3f)

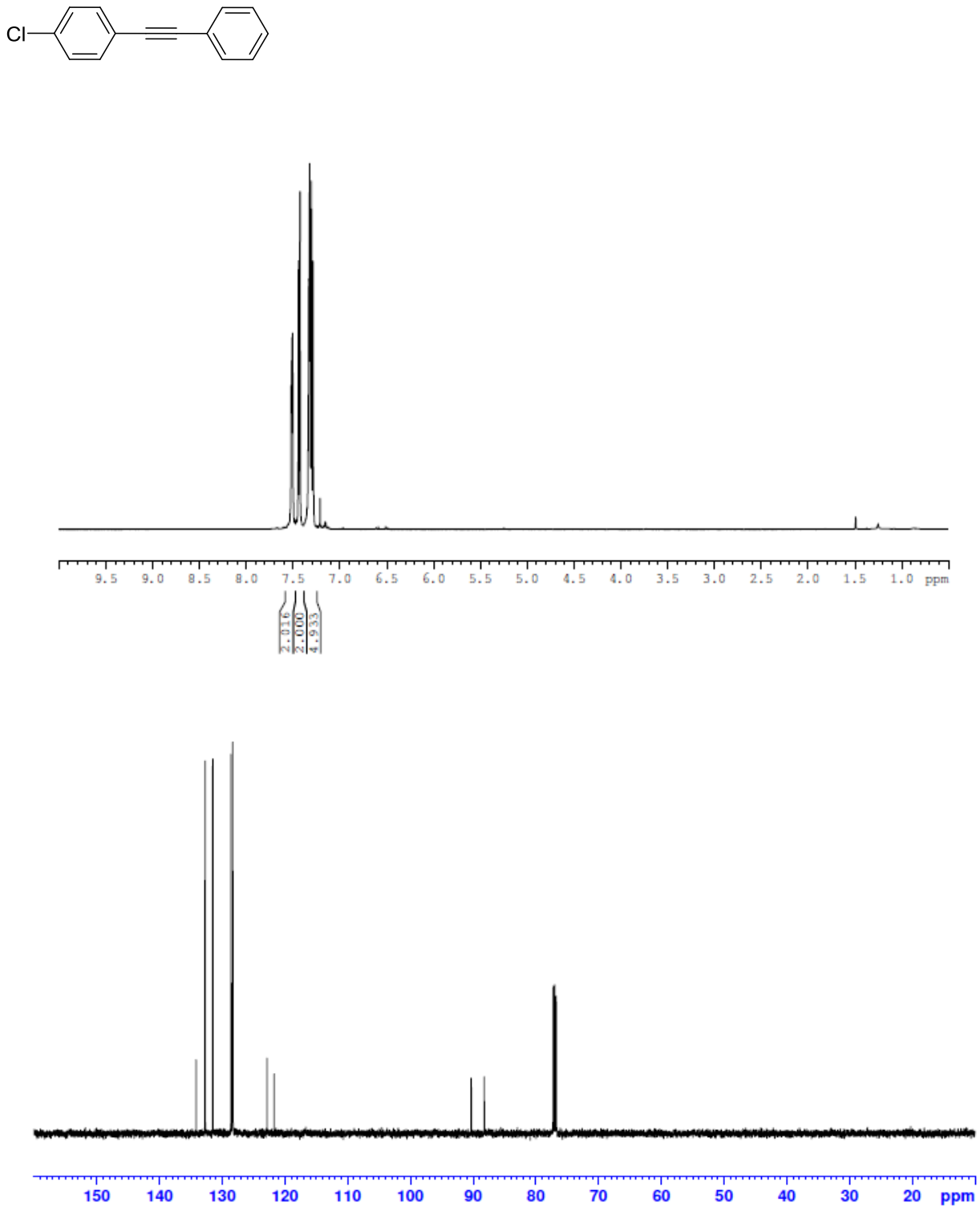


Figure S6. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of 3f in $CDCl_3$.

1-Bromo-4-(phenylethynyl)benzene (3g)

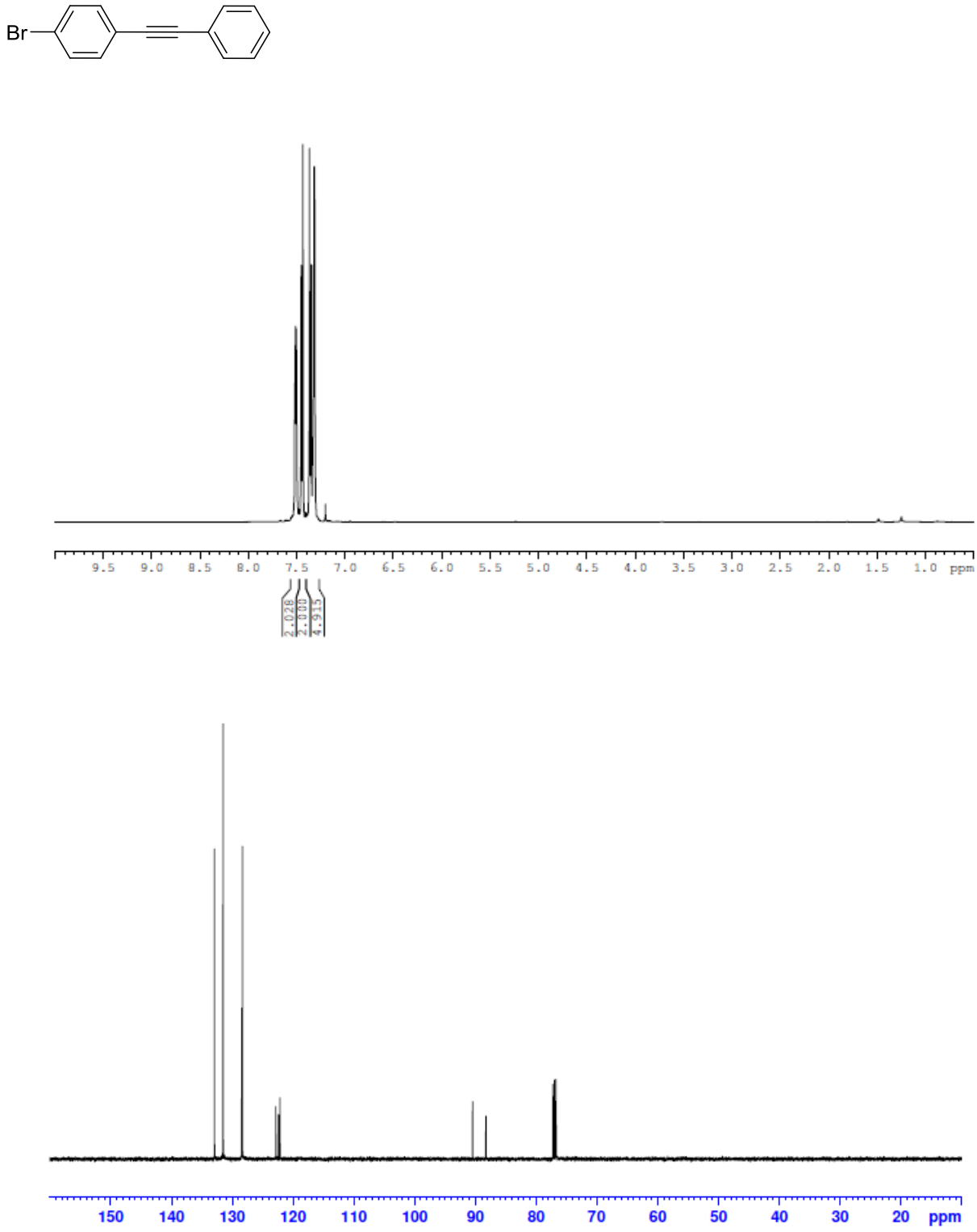
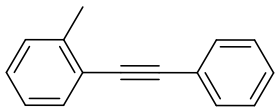


Figure S7. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of 3g in $CDCl_3$.

1-Methyl-2-(phenylethynyl)benzene (3h)



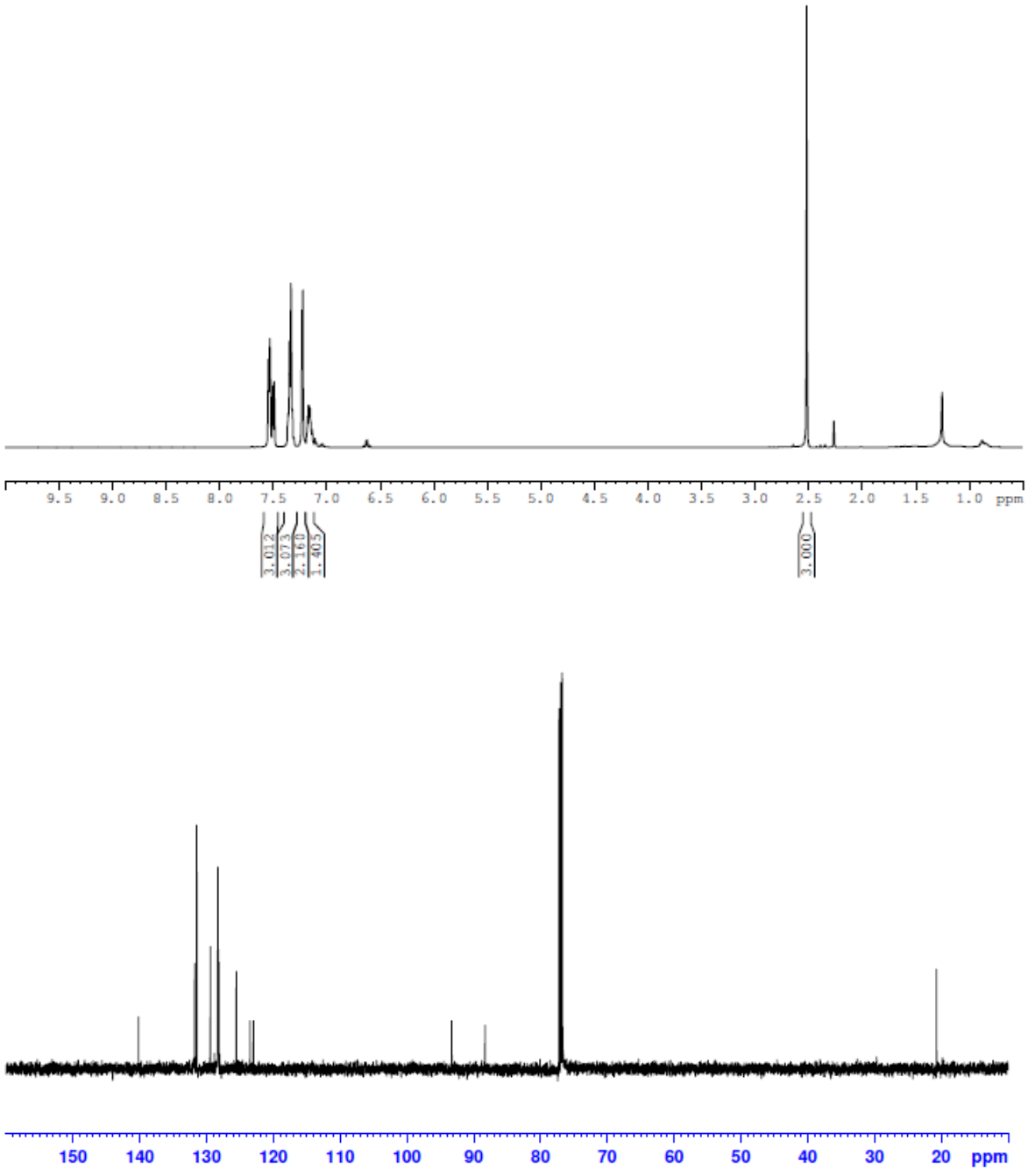


Figure S8. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of 3h in $CDCl_3$.

1-Fluoro-2-(phenylethynyl)benzene (3i)

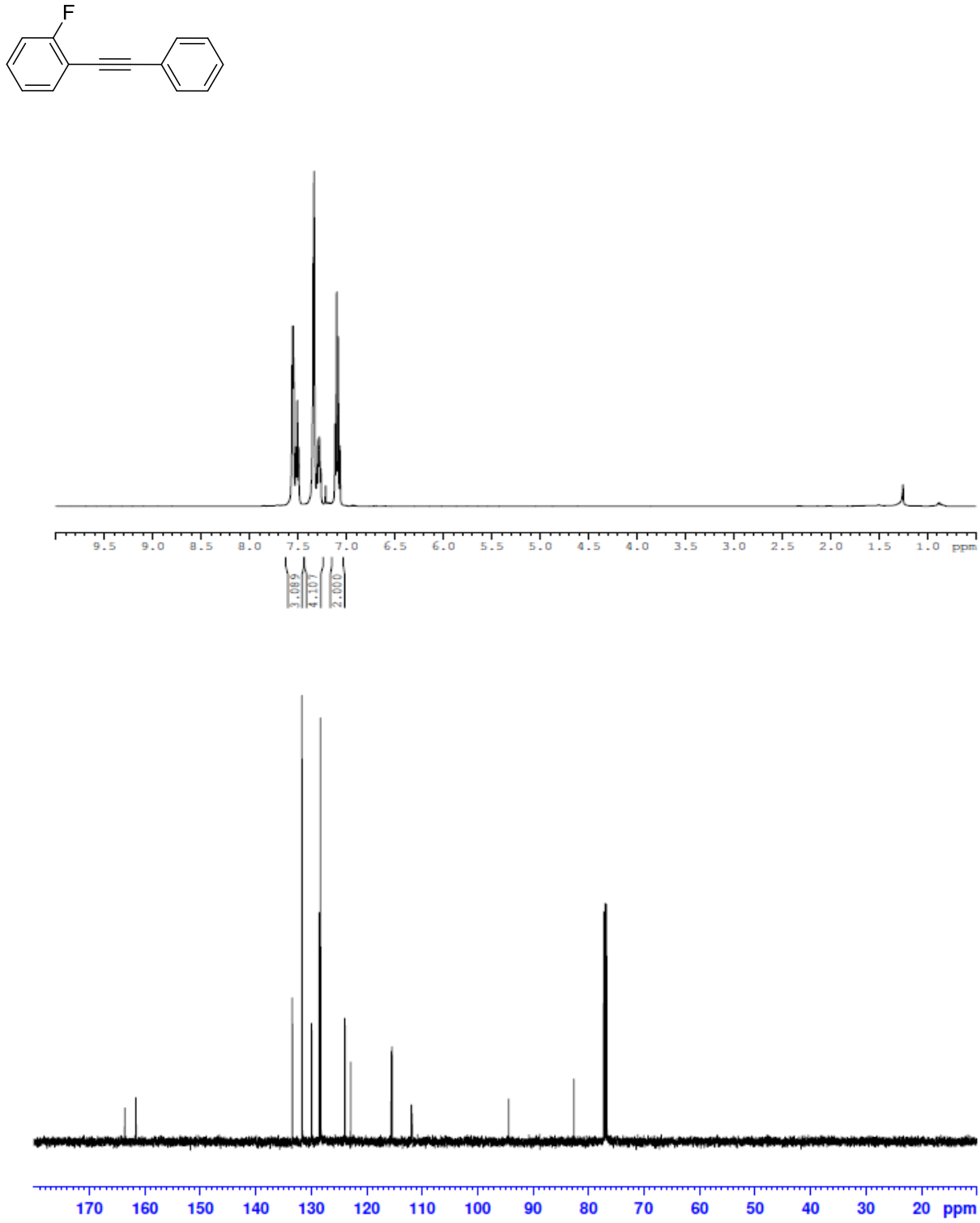


Figure S9. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of 3i in $CDCl_3$.

1-(Phenylethynyl)naphthalene (3j)

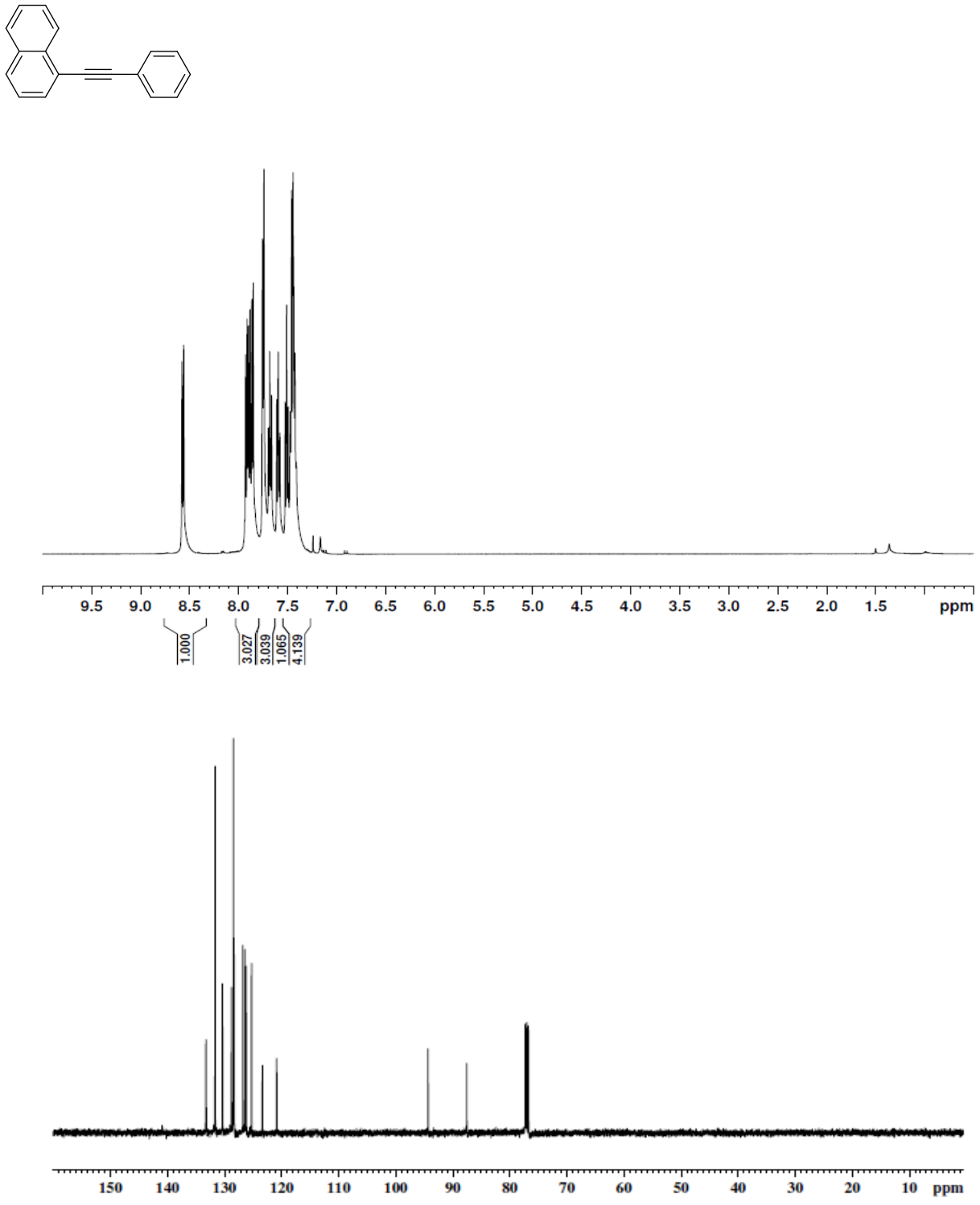


Figure S10. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of 3j in $CDCl_3$.

1-Methyl-3-(phenylethynyl)benzene (3k)

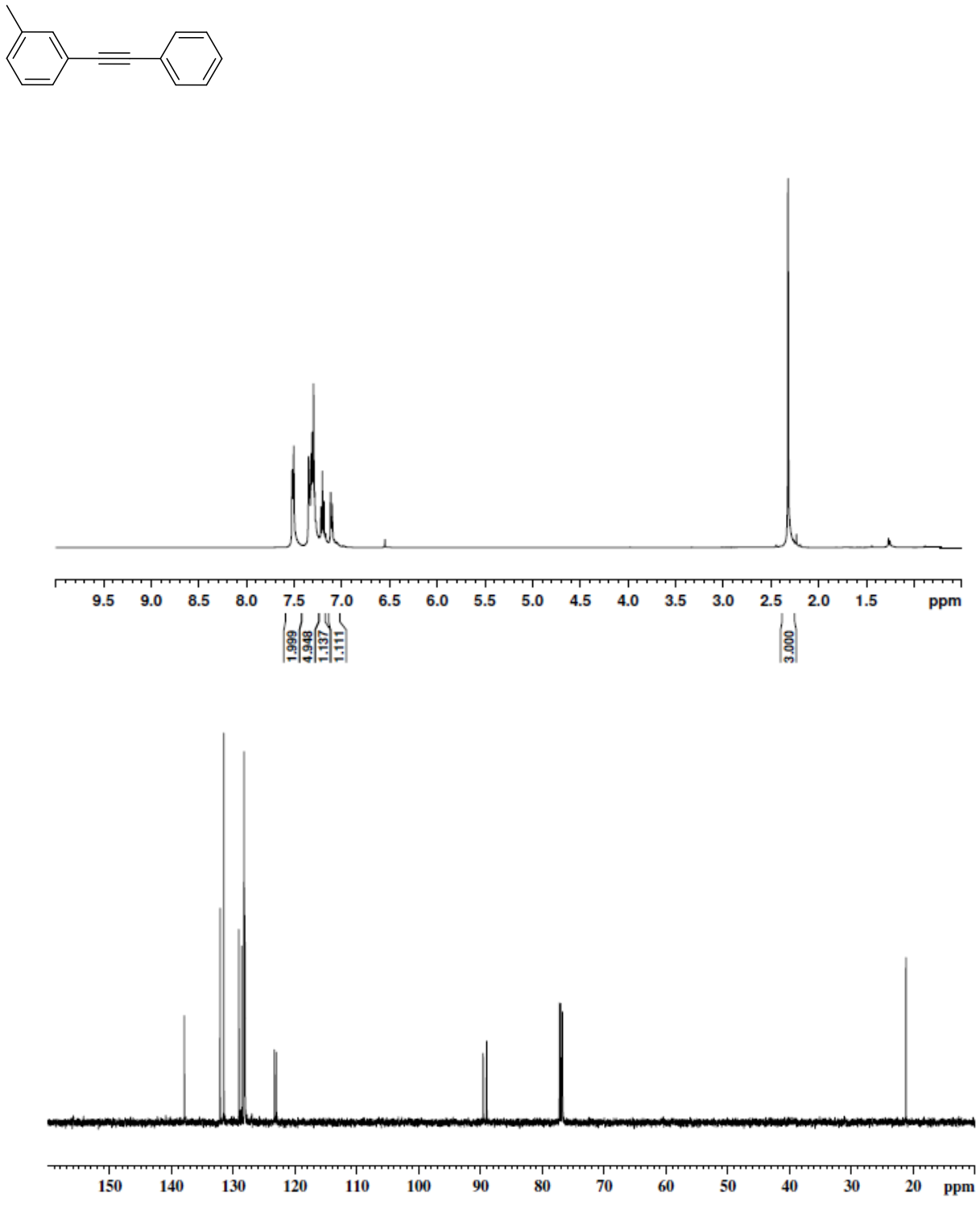


Figure S11. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of 3k in $CDCl_3$.

1-Fluoro-3-(phenylethynyl)benzene (3l)

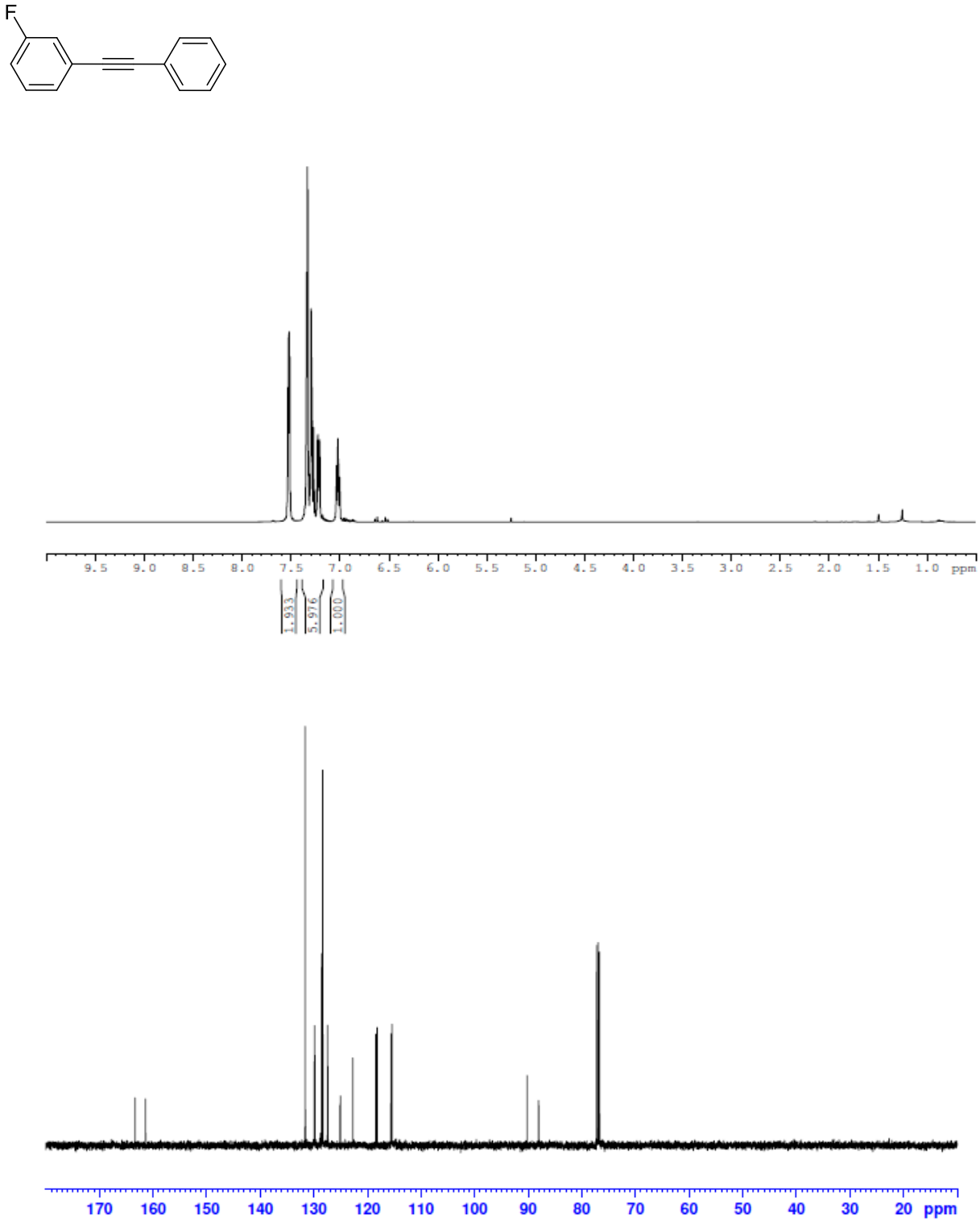


Figure S12. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of 3l in $CDCl_3$.

1-(2-Phenylethynyl)-3-(trifluoromethyl)benzene (3m)

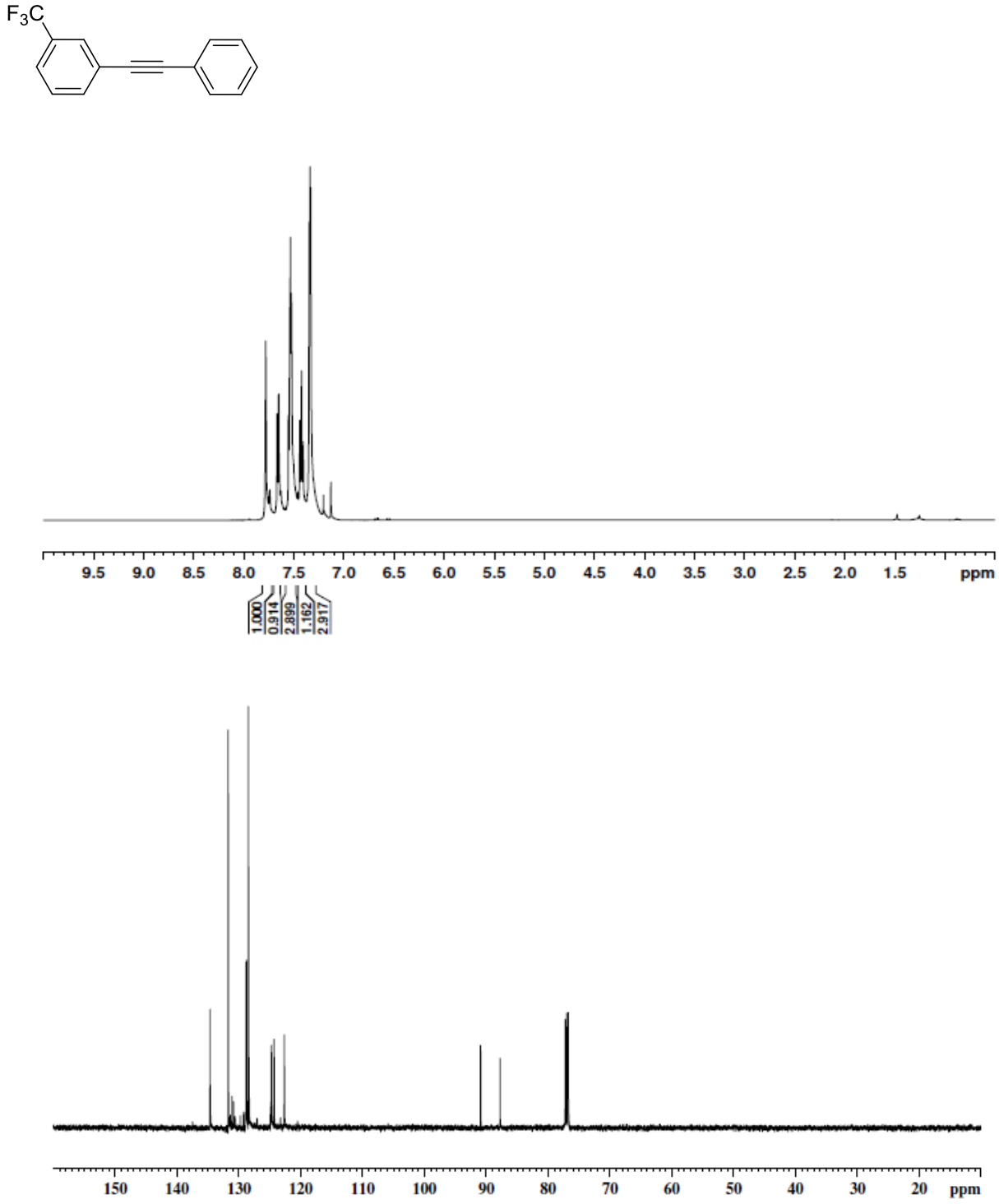
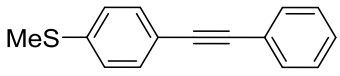


Figure S13. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of 3m in $CDCl_3$.

4-Methylthio-1-(phenylethynyl)benzene (3n)



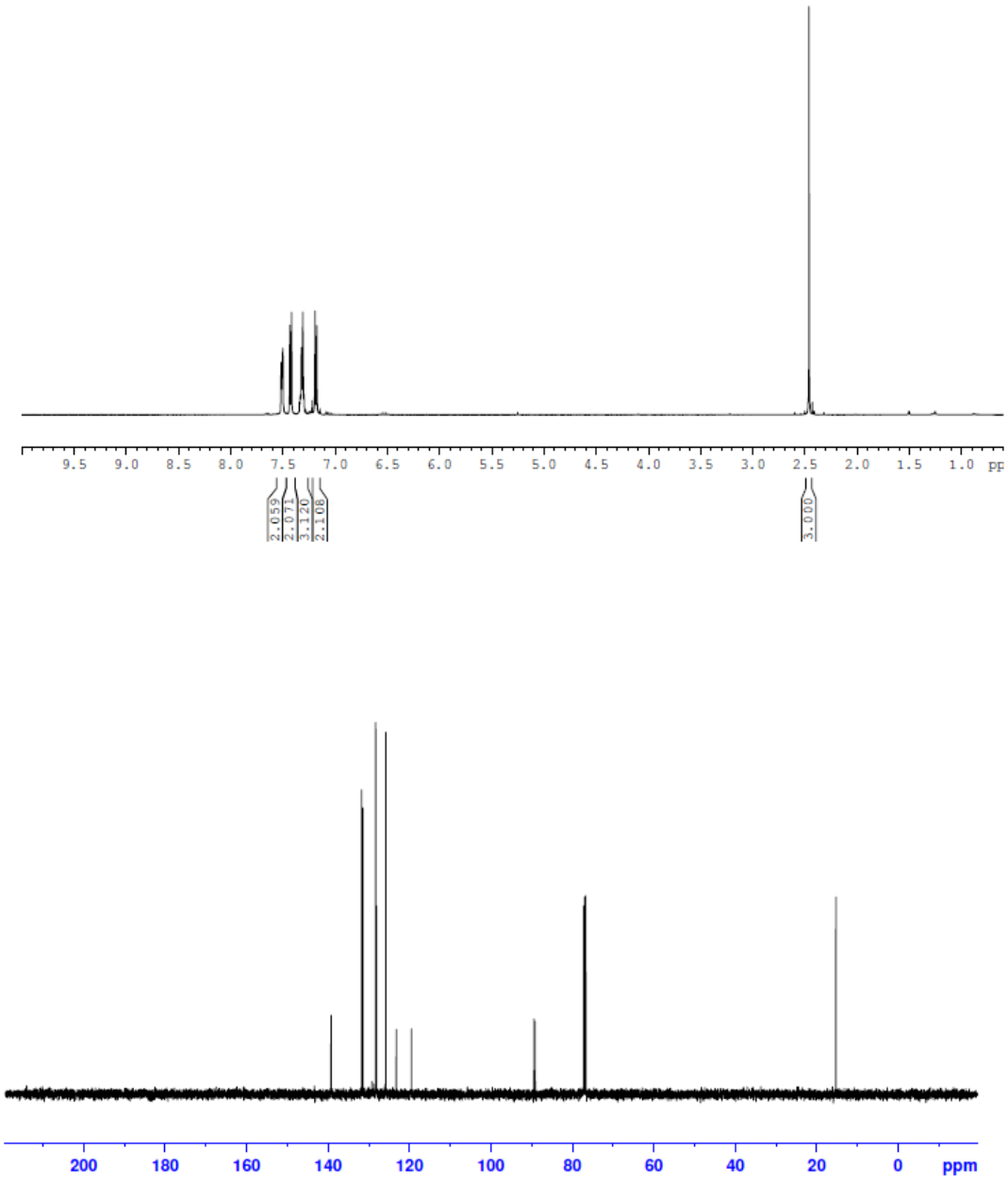


Figure S14. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of 3n in $CDCl_3$.

1-(2-Phenylethynyl)-4-(trifluoromethyl)benzene (3o)

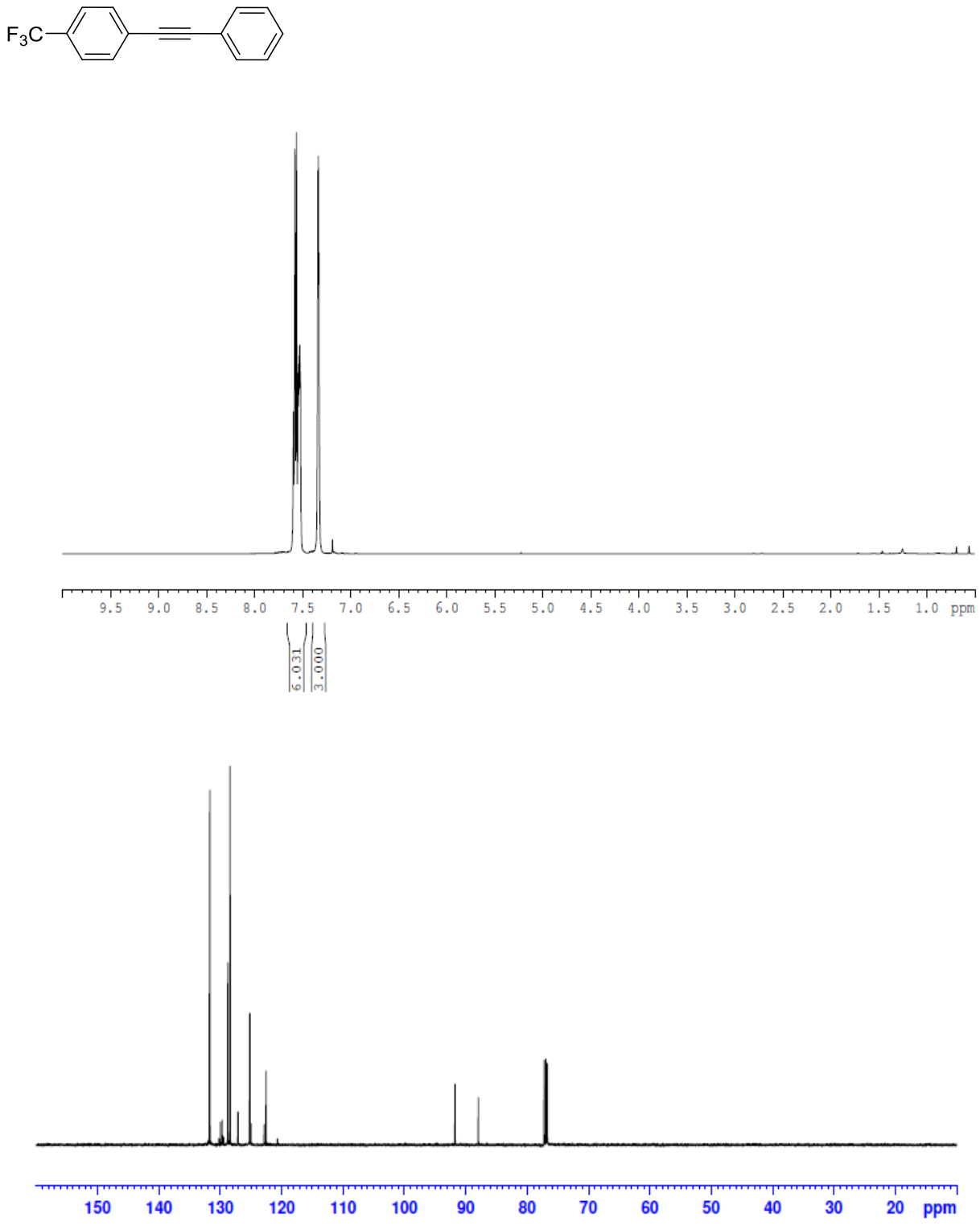
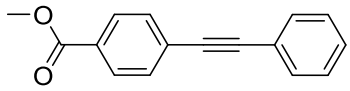


Figure S15. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of 3o in $CDCl_3$.

1-(4-Methoxycarbonylphenyl)-2-phenylacetylene (3p)



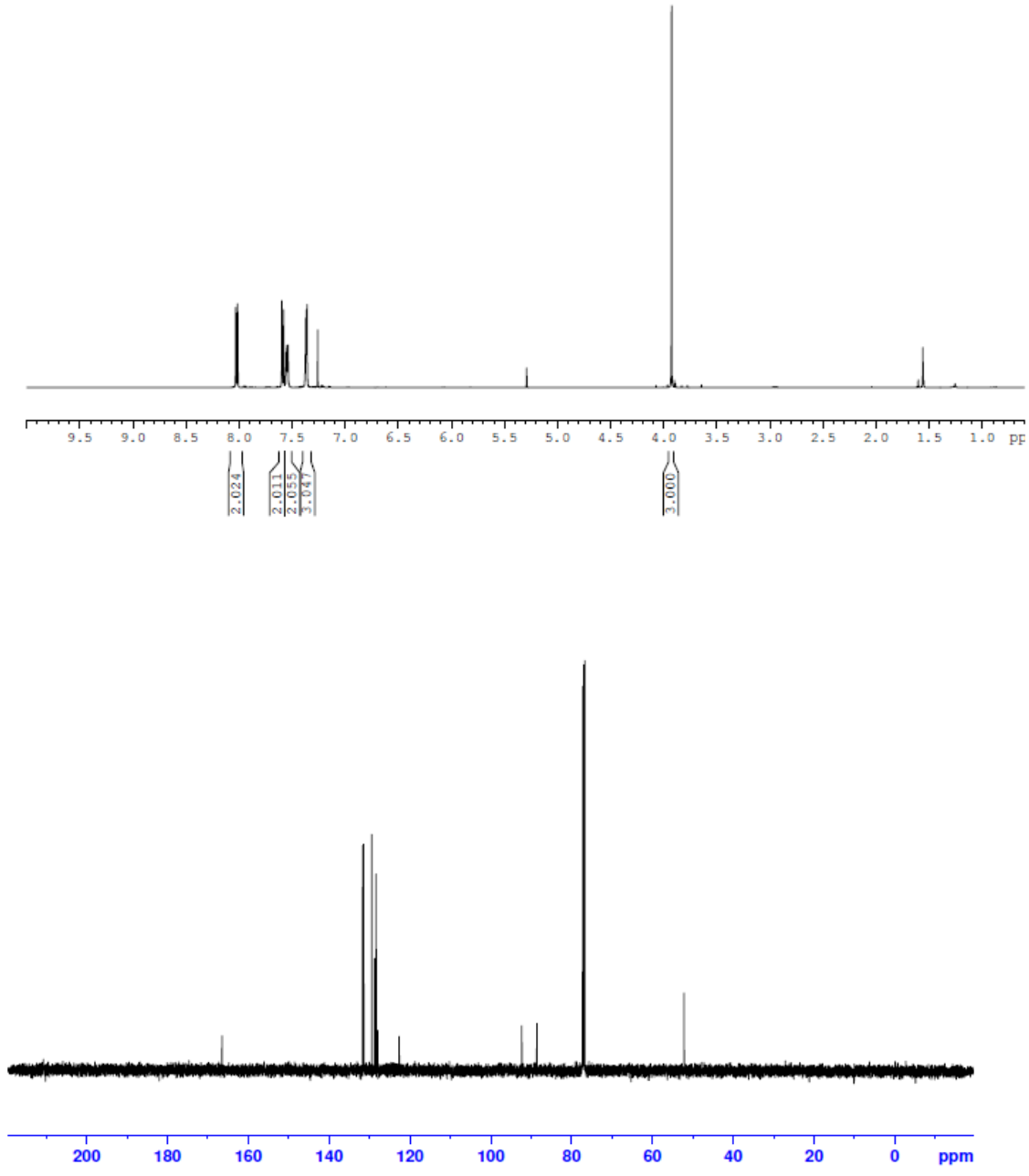


Figure S16. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of 3p in $CDCl_3$.

2-(2-Phenylethynyl)naphthalene (3q)

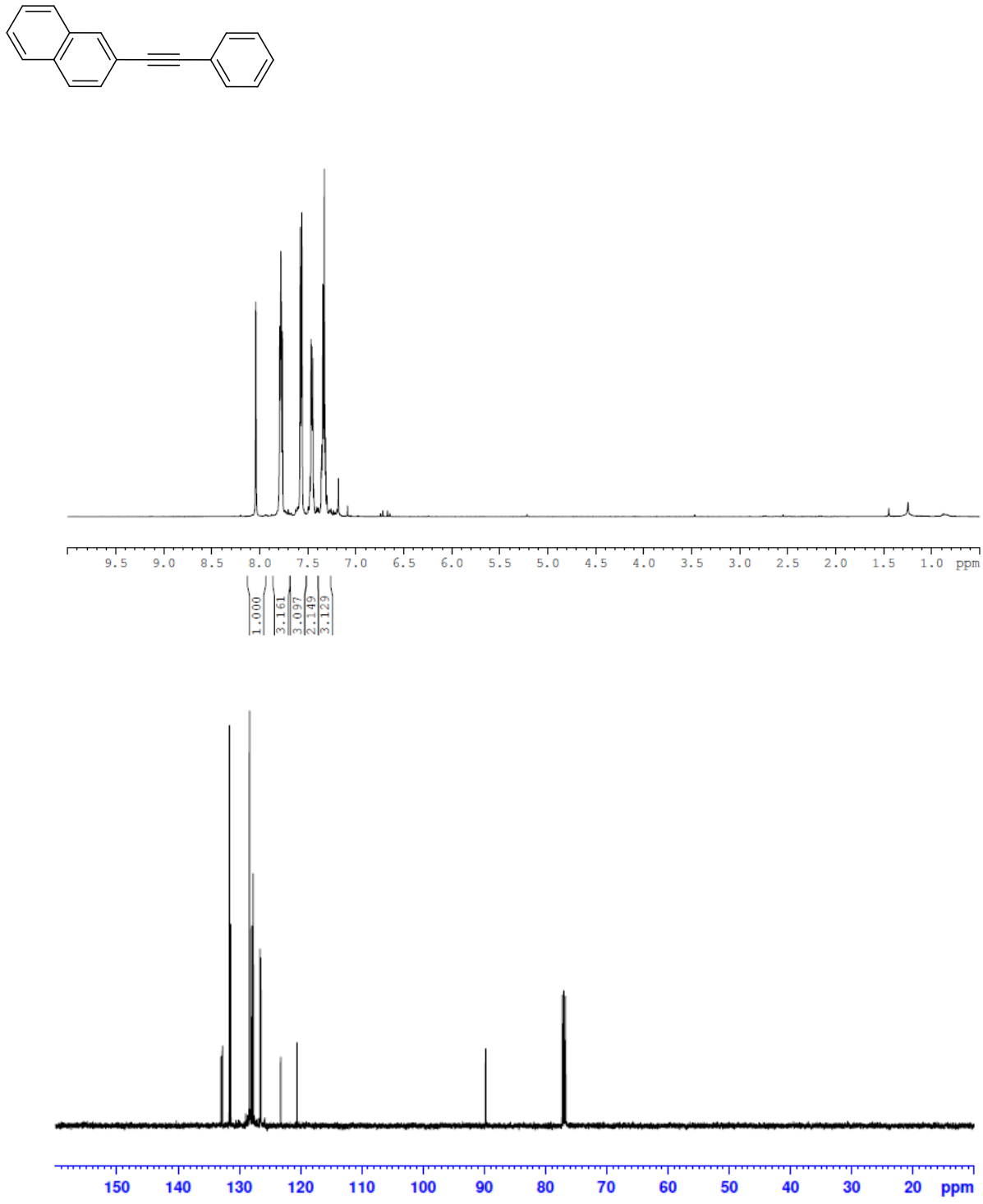


Figure S17. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of 3q in $CDCl_3$.

1-[3-(2-Phenylethynyl)phenyl]pyrrole (3r)

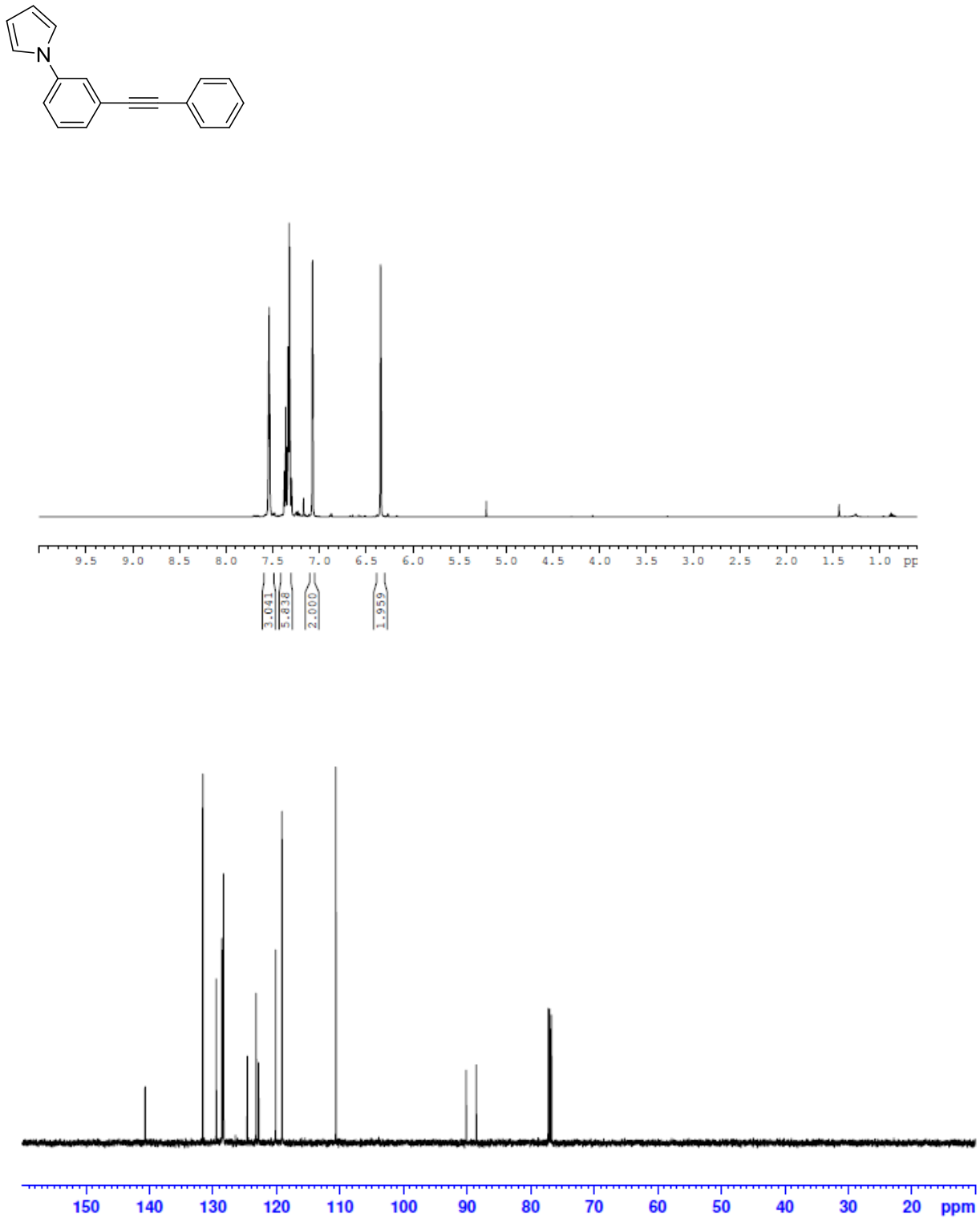


Figure S18. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of 3r in $CDCl_3$.

1-phenyl-2-(m-hydroxyphenyl)acetylene (3s)

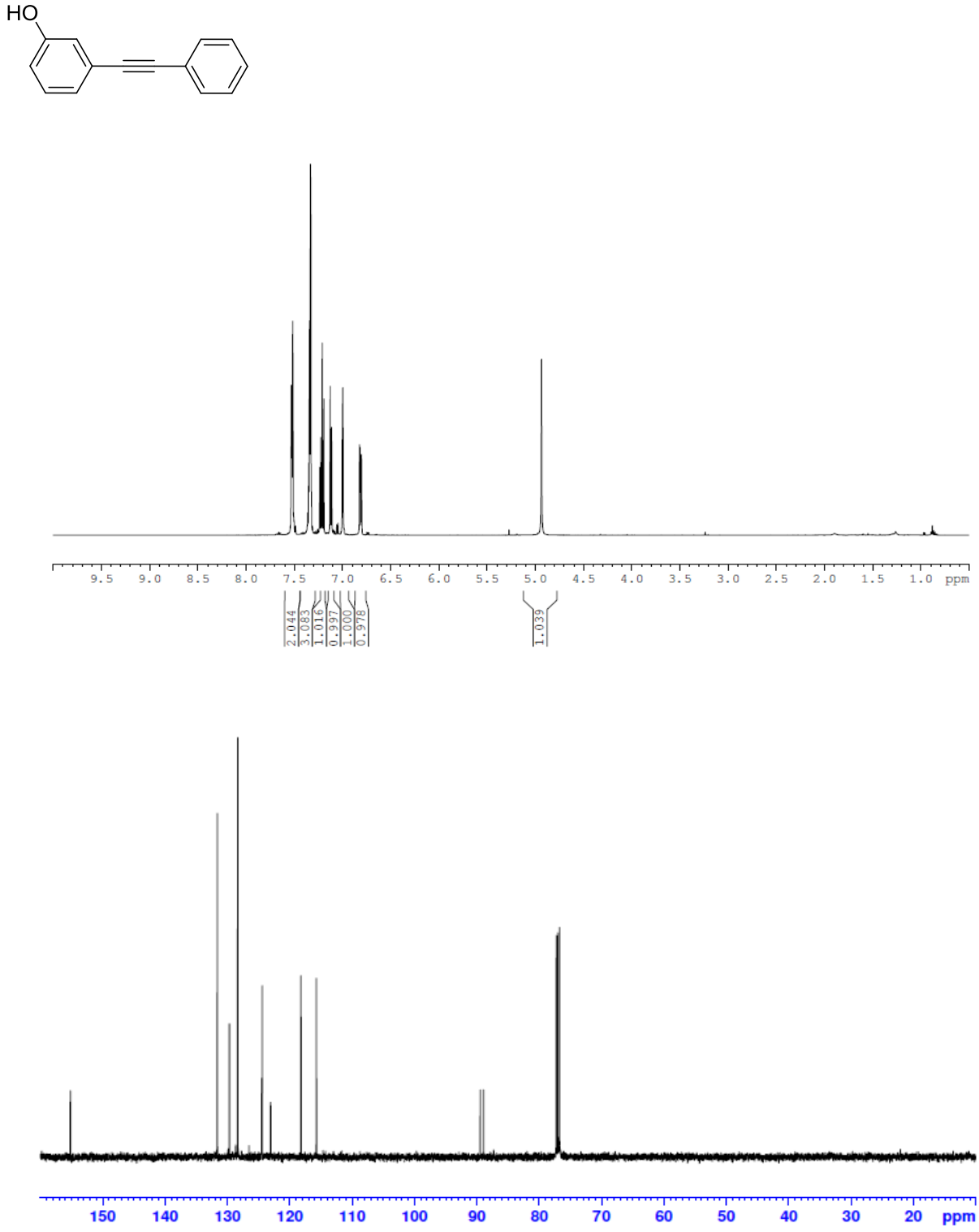


Figure S19. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of 3p in $CDCl_3$.

(3-cyanophenyl)phenylacetylene (3t)

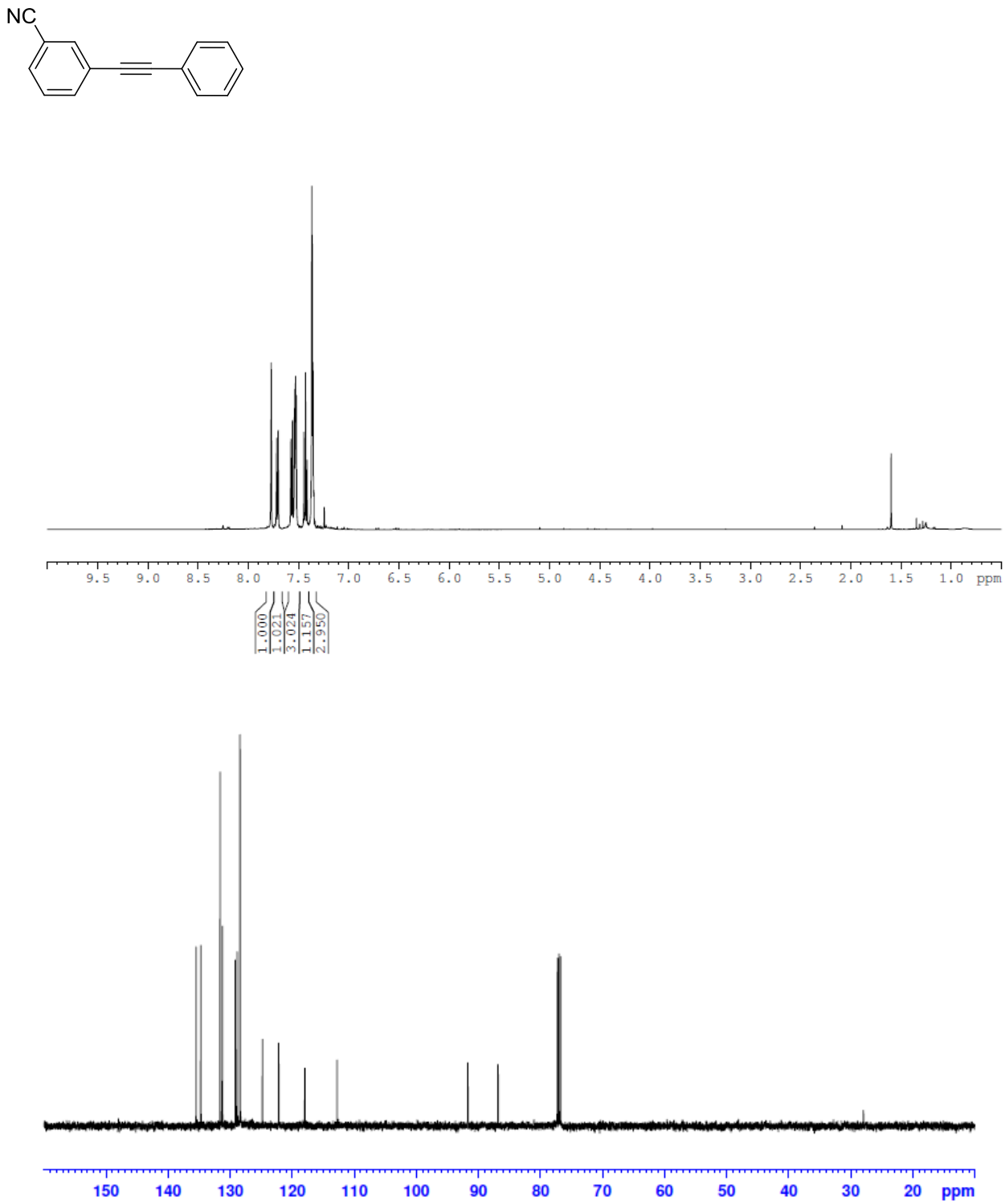


Figure S20. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of 3t in $CDCl_3$.

2-(2-phenylethynyl)pyridine (3u)

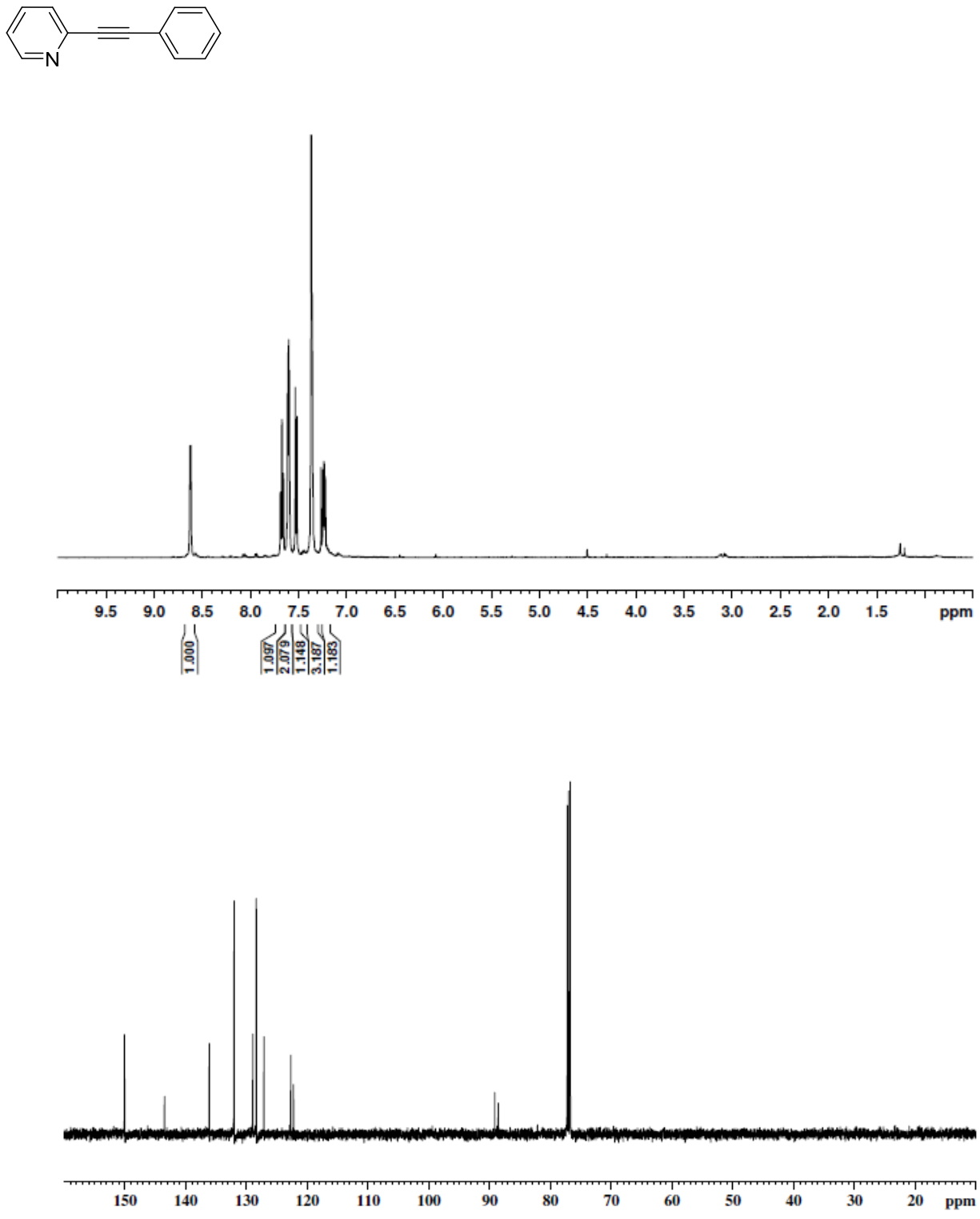


Figure S21. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of 3u in $CDCl_3$.

3-(2-phenylethynyl)pyridine (3v)

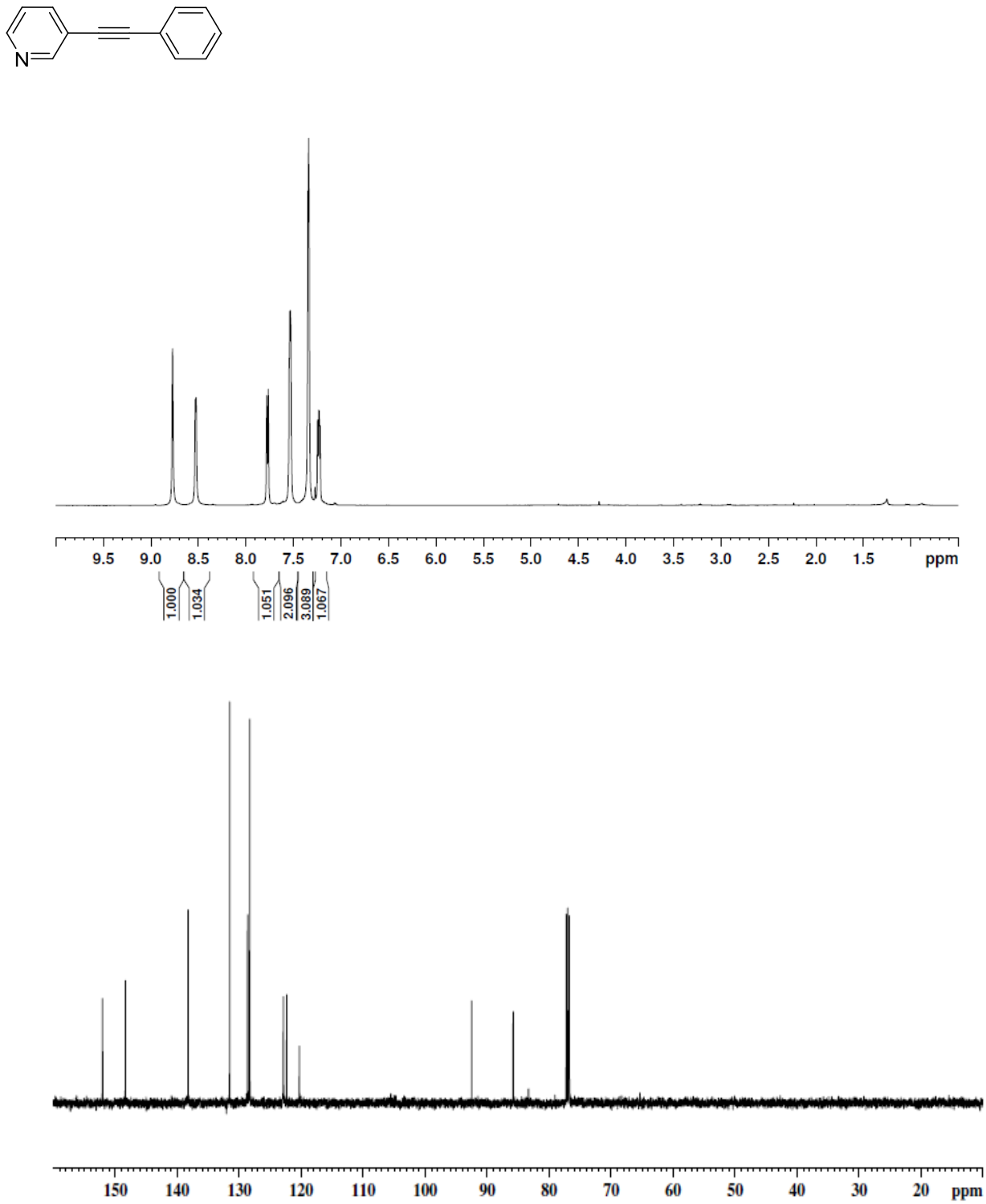


Figure S22. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of 3v in $CDCl_3$.

(5-isoquinolinyl)phenylacetylene (3w)

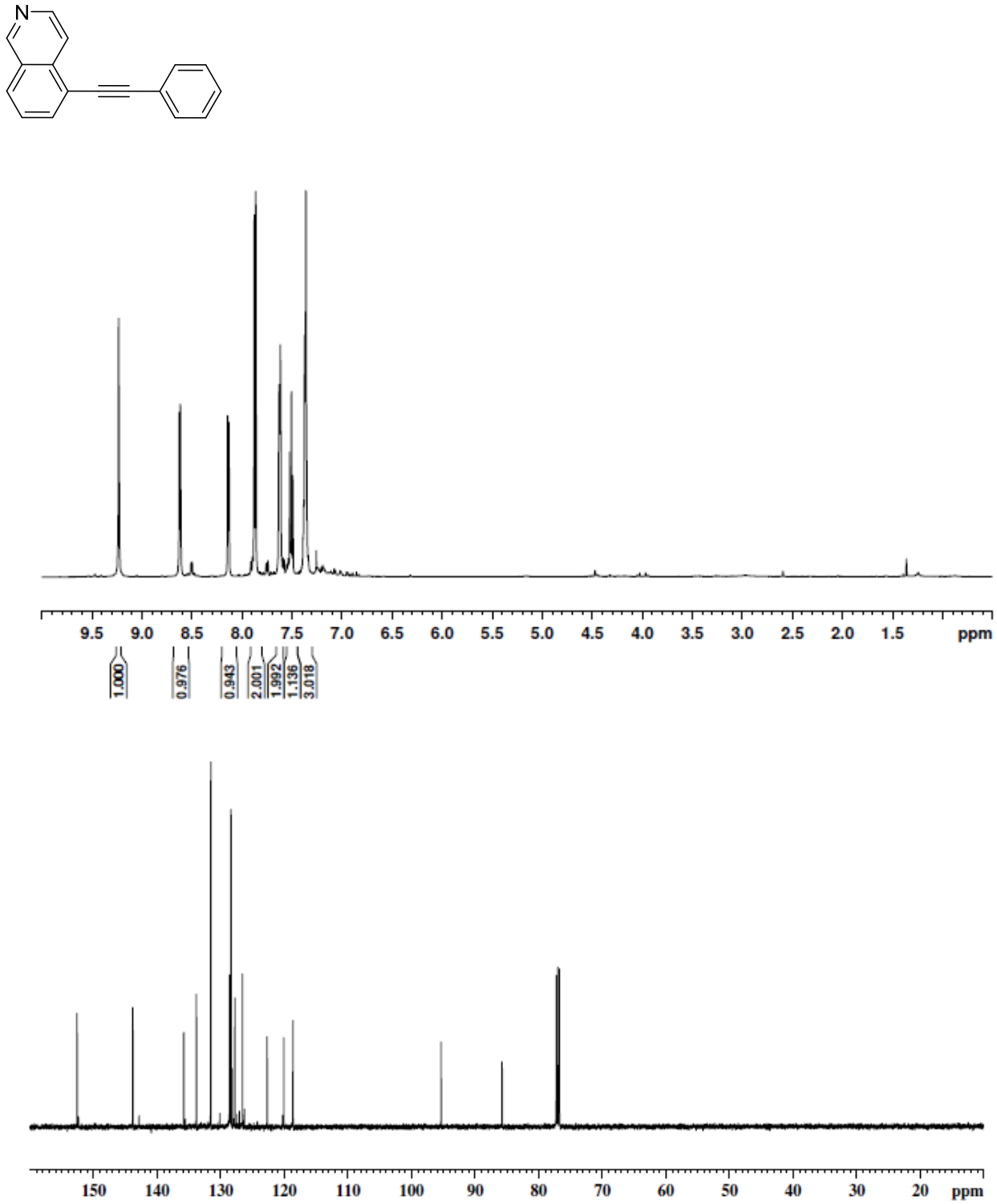


Figure S23. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of 3w in $CDCl_3$.

2-(2-phenylethynyl)furan (3x)

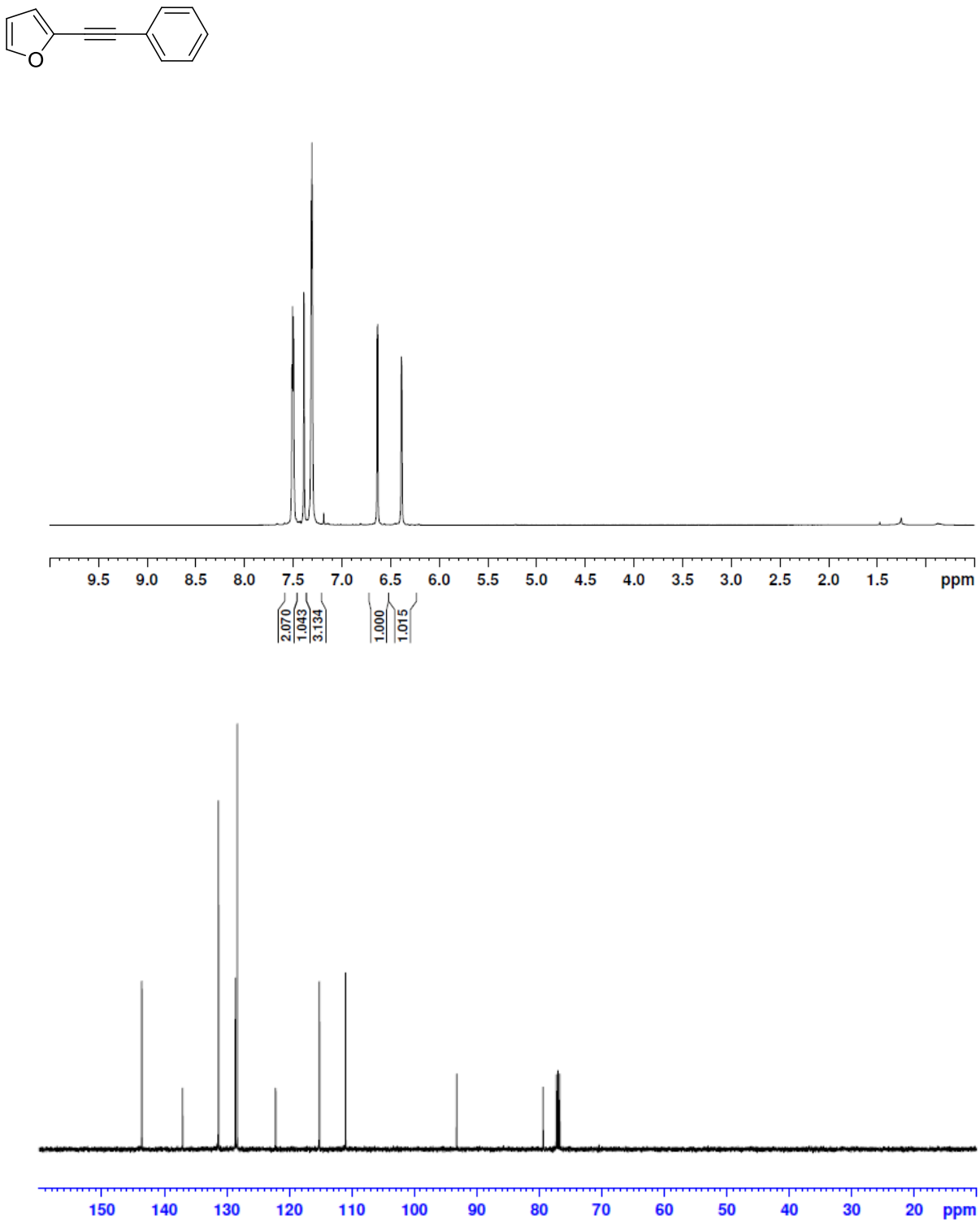
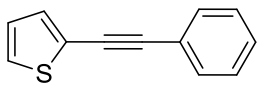


Figure S24. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of 3x in $CDCl_3$.

2-(2-phenylethynyl)thiophene (3y)



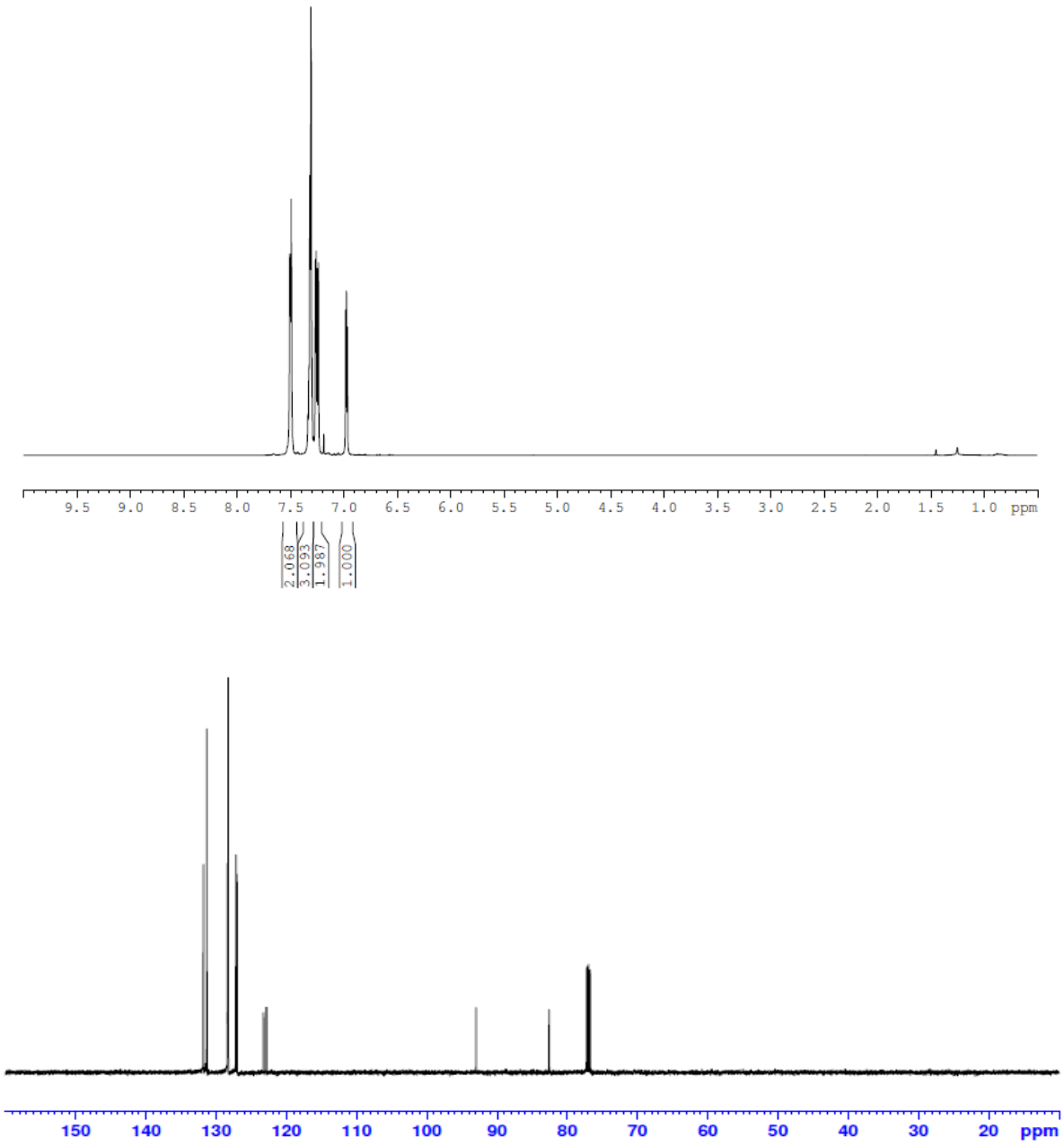


Figure S25. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of 3y in $CDCl_3$.

1,3-bis(2-phenylethynyl)benzene (5a)

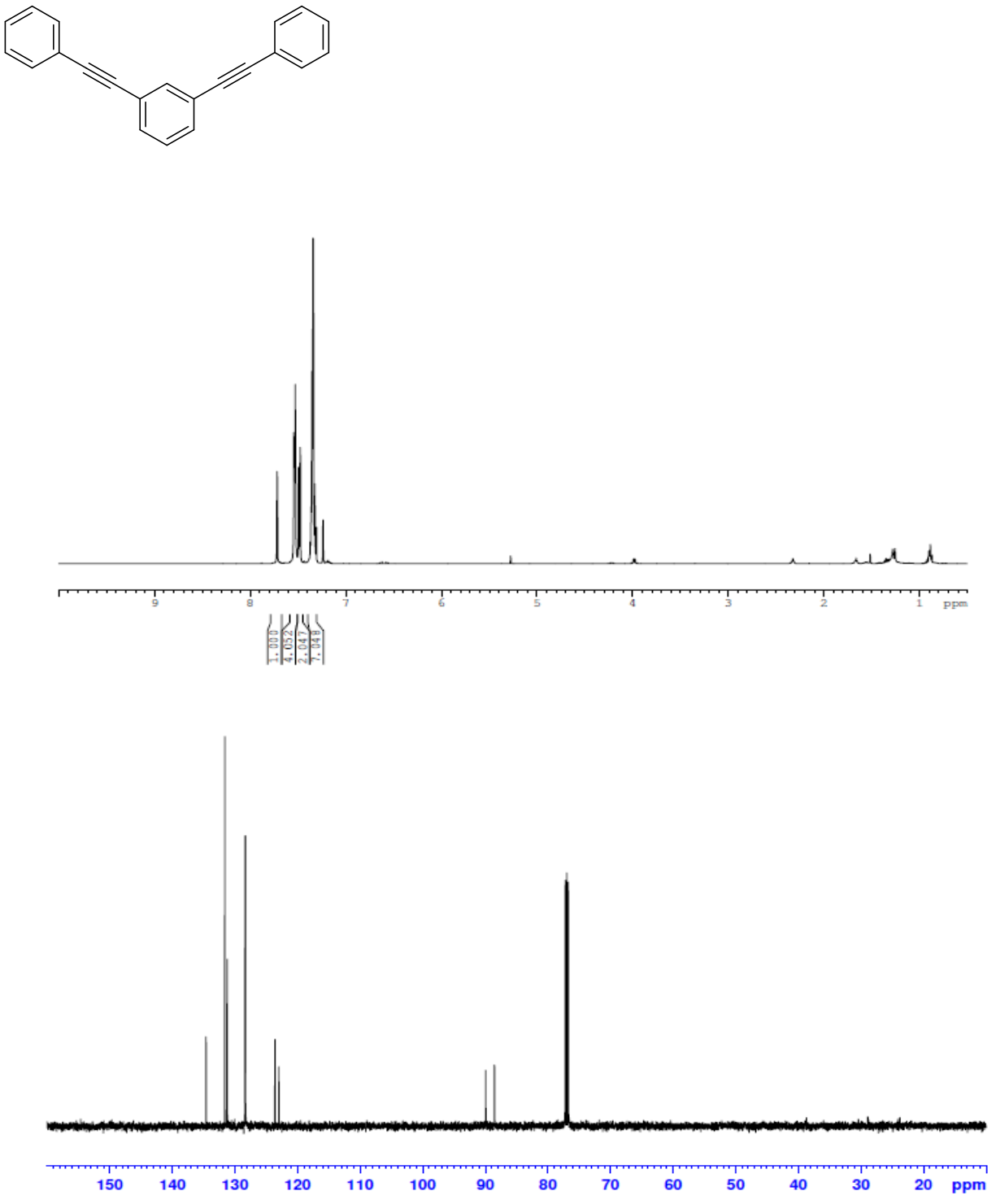


Figure S26. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of 5a in $CDCl_3$.

1,1',4-triphenylbut-3-en-1-yne (5b)

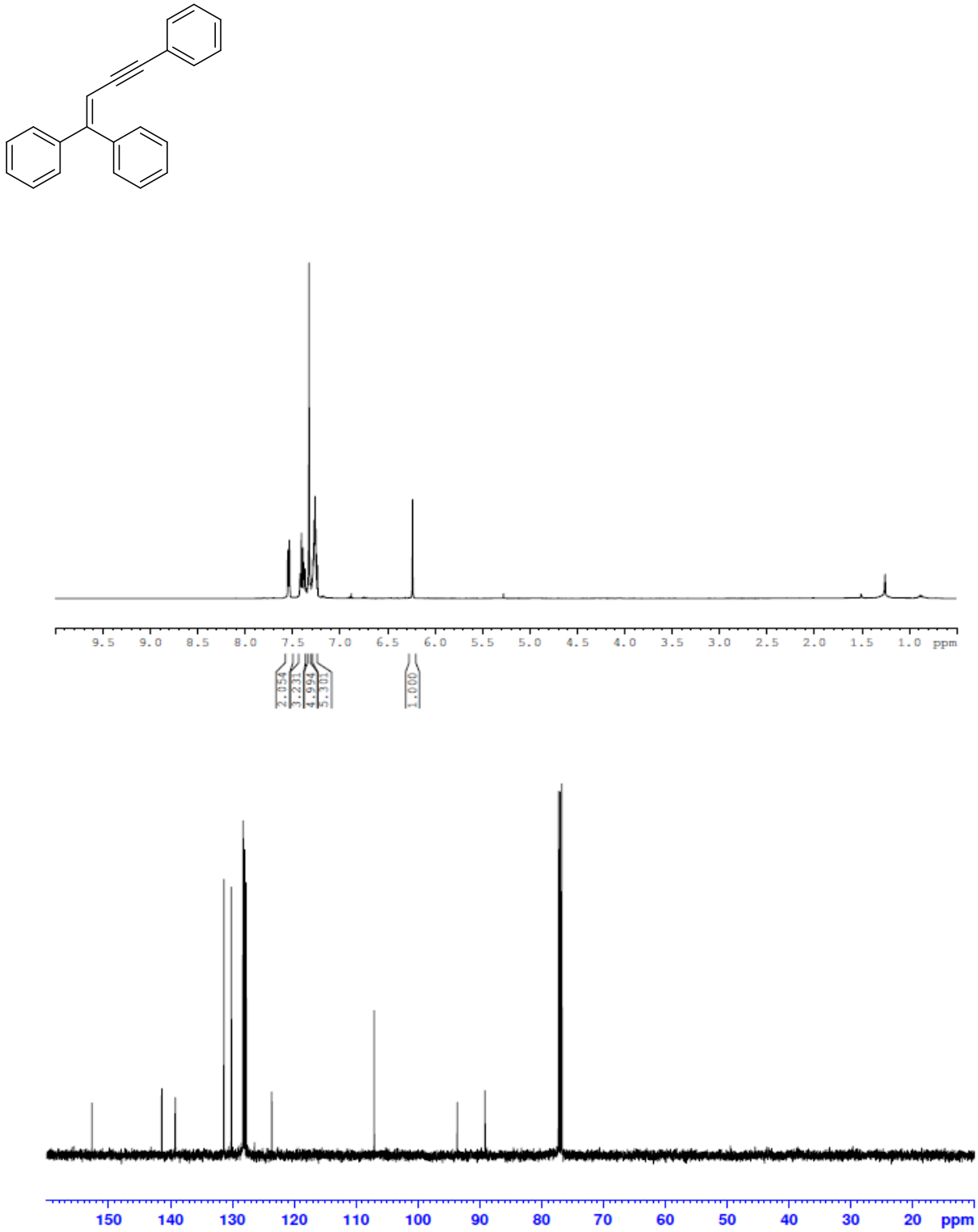
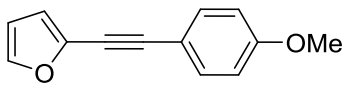


Figure S27. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of 5b in $CDCl_3$.

2-(2-(4-methoxyphenyl)ethynyl)furan (3dx)



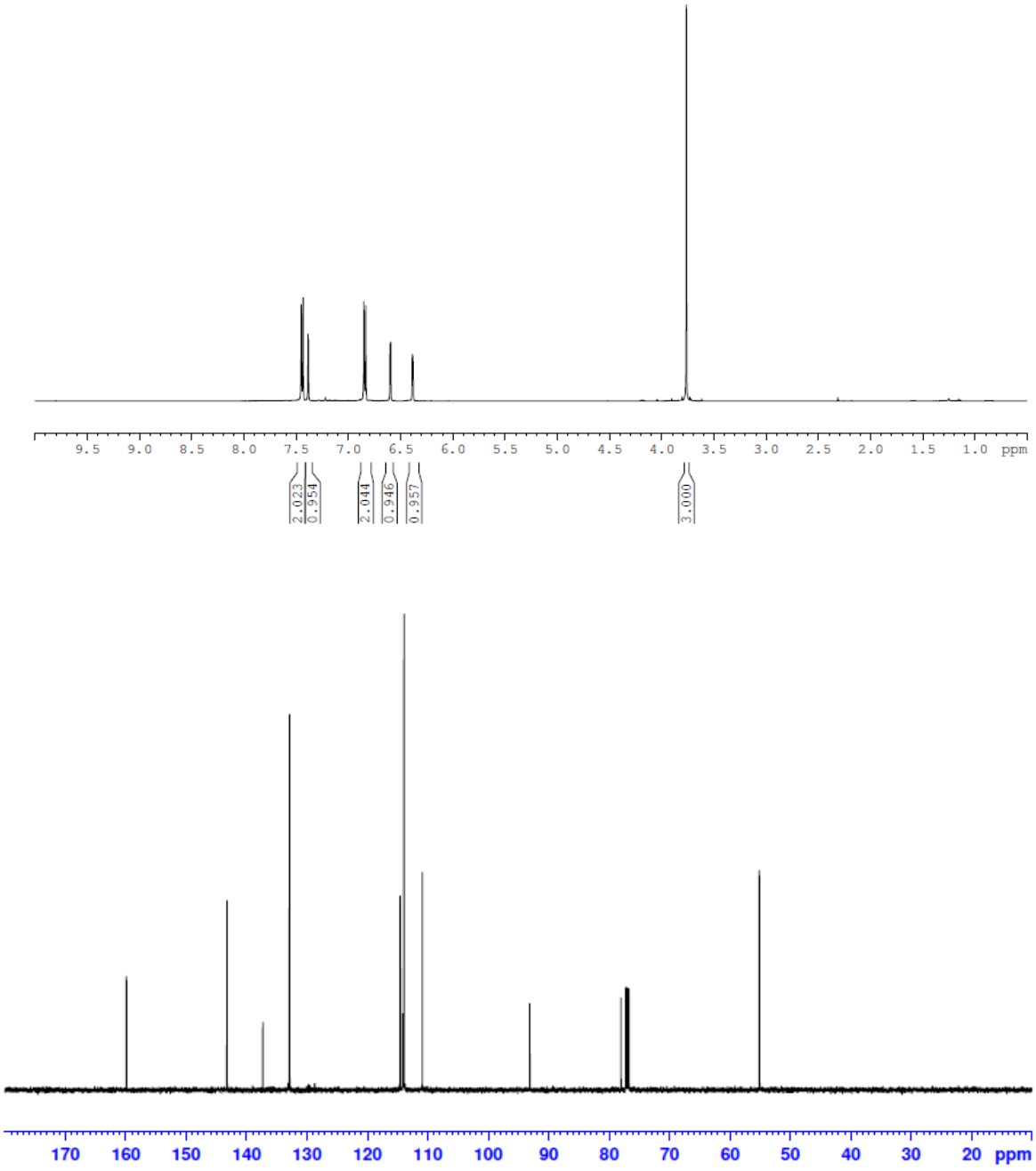
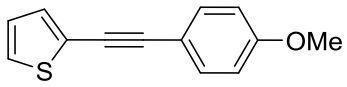


Figure S28. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of 3dx in $CDCl_3$.

2-(2-(4-methoxyphenyl)ethynyl)thiophene (3dy)



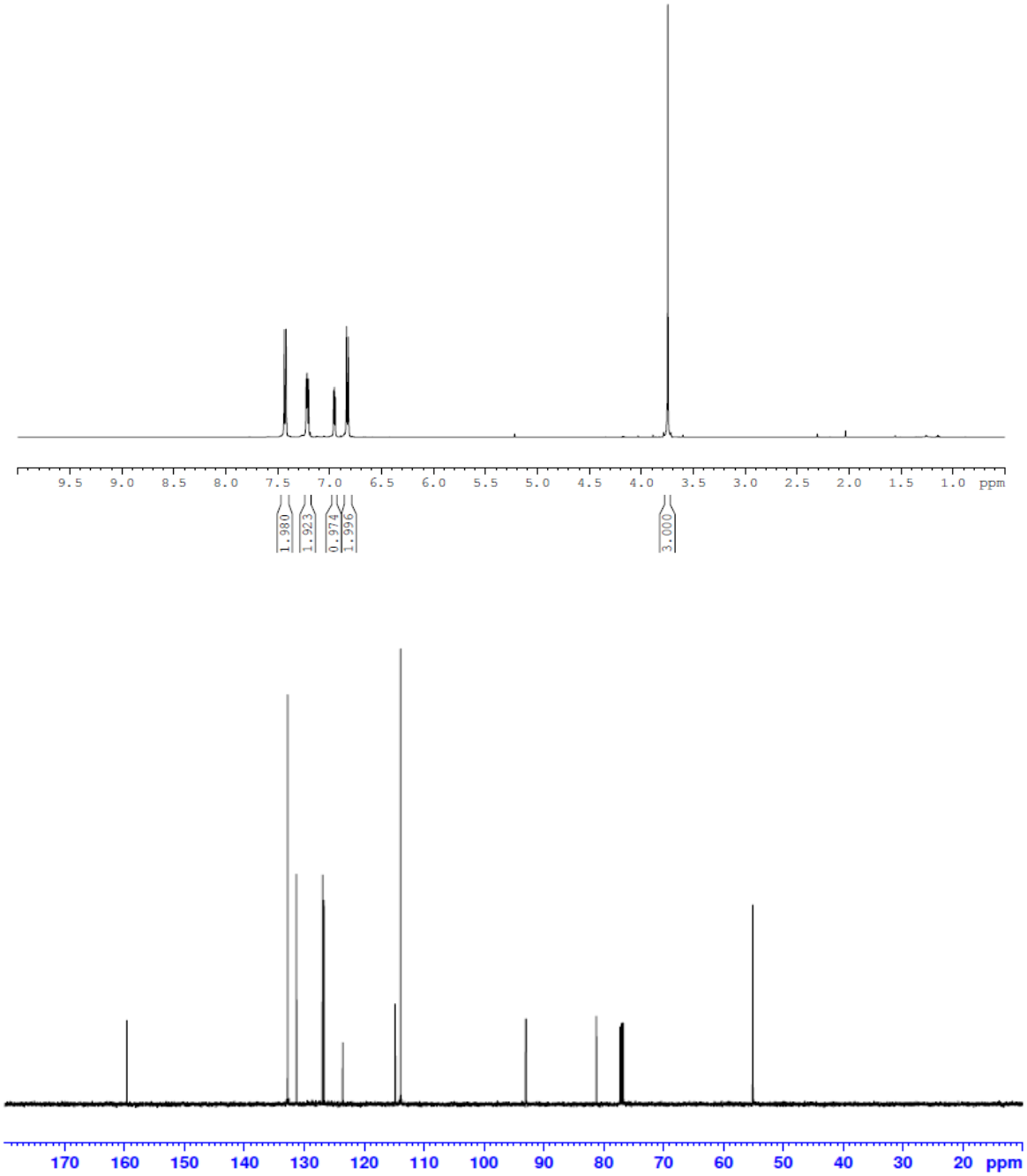


Figure S29. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of 3dy in $CDCl_3$.

1-(4-bromophenyl)-2-(4-chlorophenyl)ethyne (3gf)

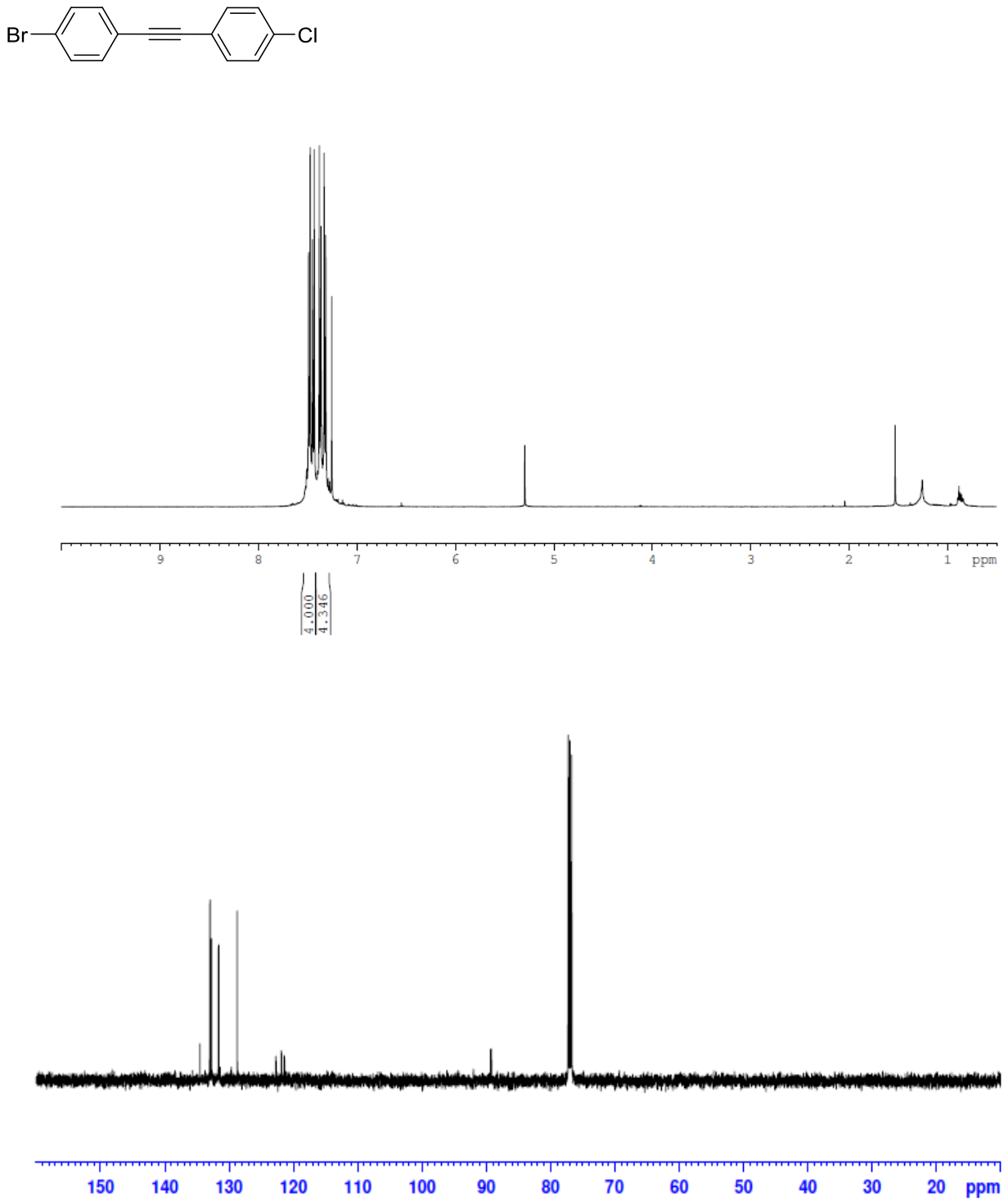


Figure S30. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of 3gf in $CDCl_3$.

1-(3-trifluoromethyl)-2-(4-trifluorophenyl)ethyne (3mo)

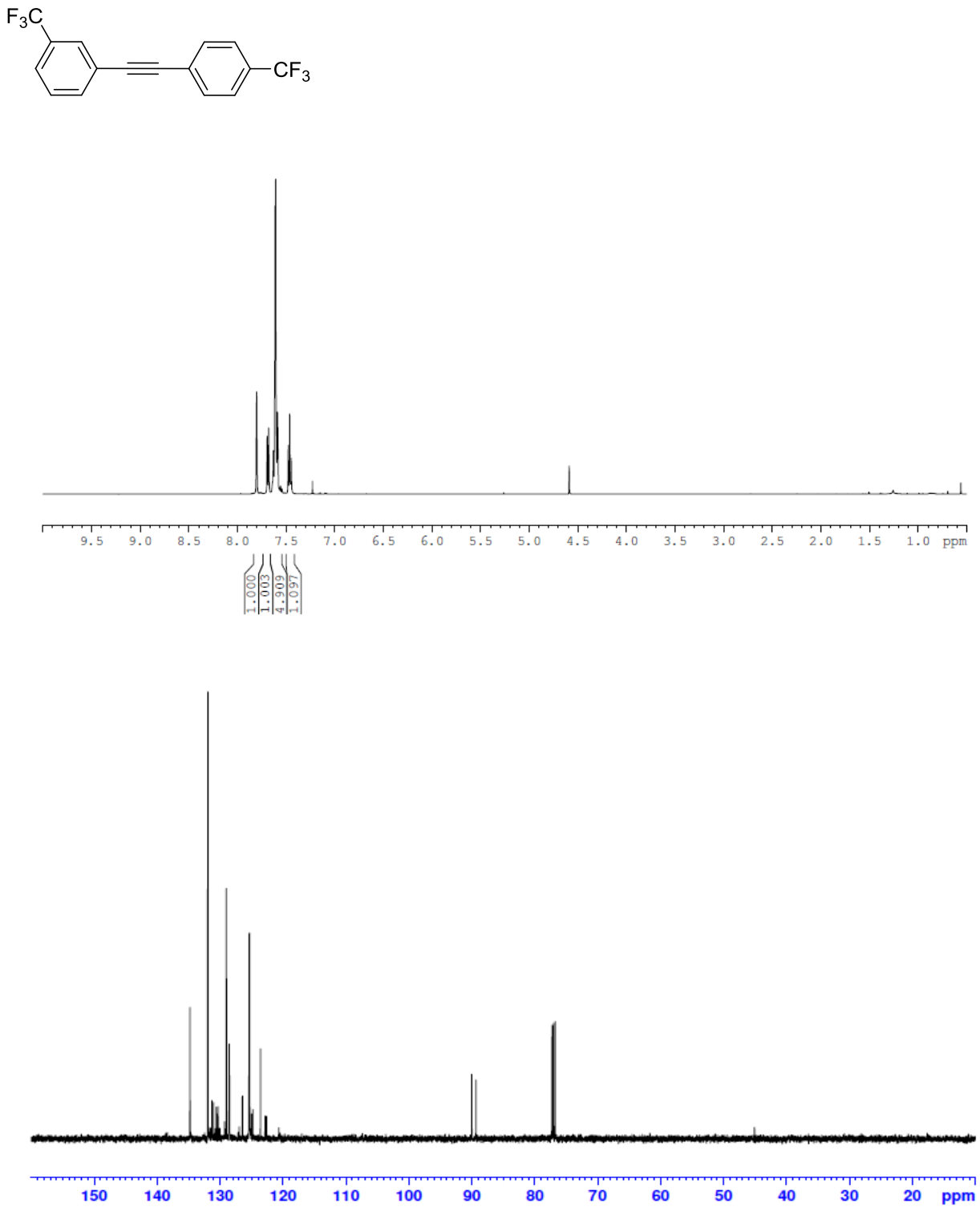
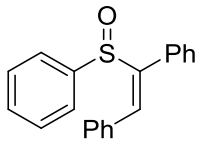


Figure S31. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of 3mo in $CDCl_3$.

(Z)-1,2-diphenyl-1-(phenylsulphinyl)ethene (I)



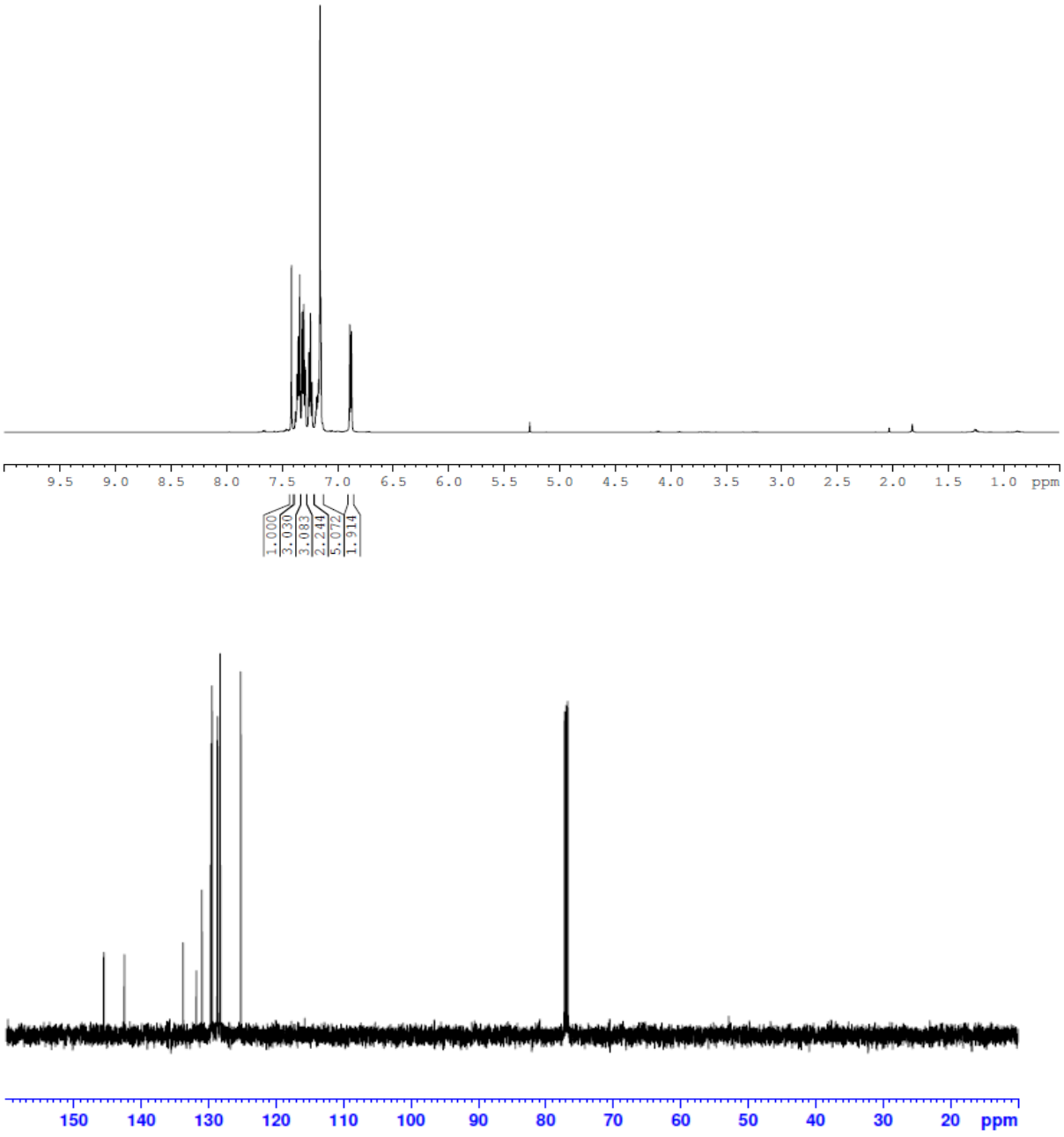


Figure S32. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of I in $CDCl_3$.

Erythro-2-phenylsulfinyl-1,2-diphenyl-1-ethanol (H1)

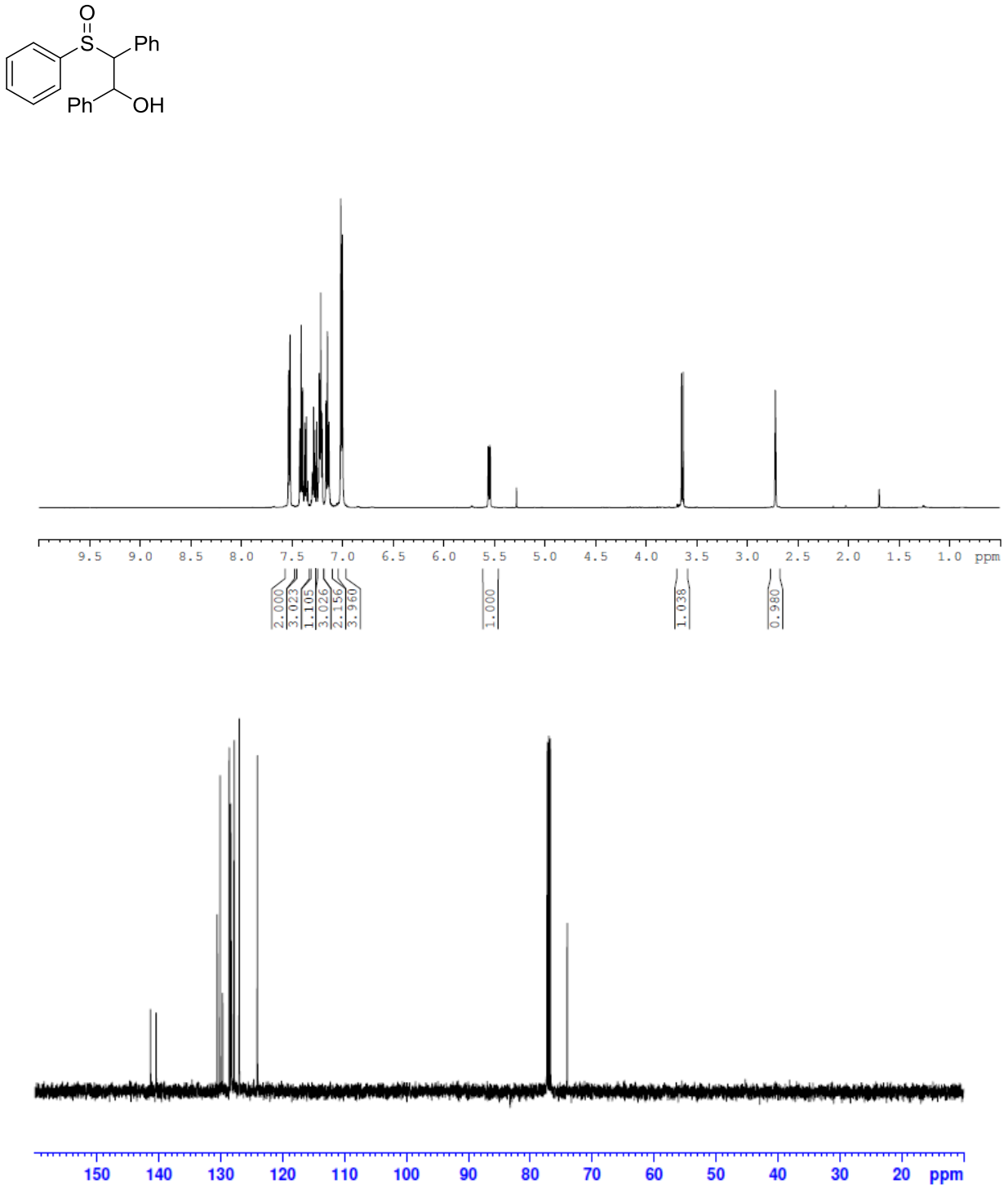
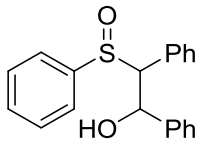


Figure S33. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of H1 in $CDCl_3$.

Threo-2-phenylsulfinyl-1,2-diphenyl-1-ethanol (H2)



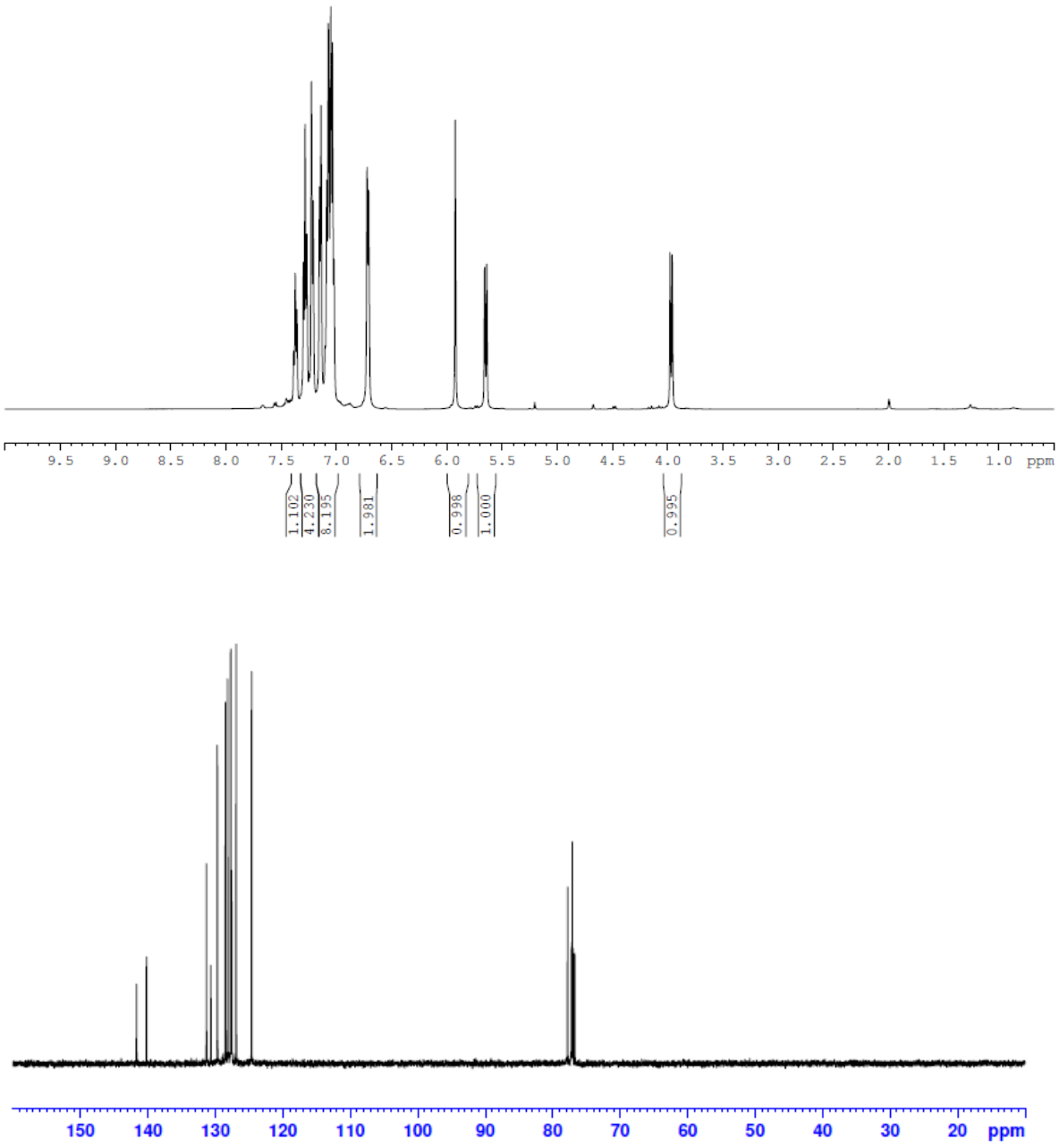


Figure S34. ^{1}H (500 MHz) and ^{13}C {1H} (125 MHz) NMR spectra of H2 in $CDCl_3$.

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