Supporting Information

Copper-Catalyzed Deacetonative Sonogashira Coupling

Yury N. Kotovshchikov,* Artem A. Binyakovsky, Gennadij V. Latyshev,
Nikolay V. Lukashev, and Irina P. Beletskaya

Chemistry Department, M. V. Lomonosov Moscow State University,
Leninskiye Gory 1/3, Moscow 119991, Russia

E-mail: kotovshchikov@org.chem.msu.ru

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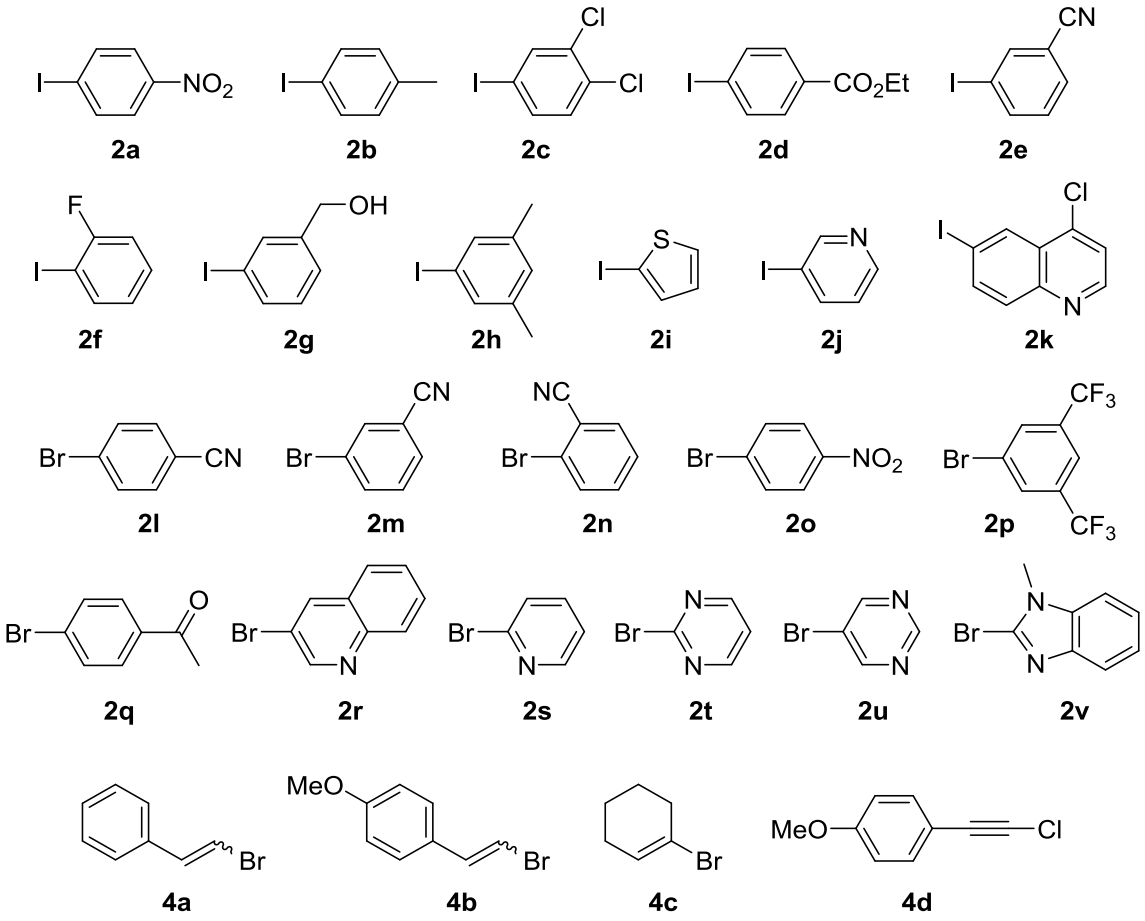
General information

NMR spectra were recorded with Bruker Avance 400, Agilent 400MR (^{1}H 400 MHz, ^{13}C 100 MHz), and Bruker Avance 600 (^{1}H 600 MHz, ^{13}C 151 MHz) spectrometers at ambient temperature. Chemical shifts are presented in ppm (δ scale) and referenced to tetramethylsilane (δ = 0 ppm) in the ^{1}H NMR spectra and to the solvent signal in the ^{13}C NMR spectra. MALDI-TOF spectra were recorded with a Bruker Daltonics UltraFlex instrument in a dithranol or a cinnamic acid matrix using PEG 300, PEG 400 or PEG 600 as the internal standard. ESI mass spectra were obtained from Thermo Scientific LTQ Orbitrap and Sciex TripleTOF 5600+ spectrometers. Column chromatography was carried out on Macherey–Nagel silica gel 60 (0.040–0.063 mm).

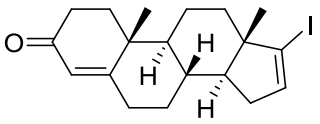
Experimental procedures and characterization data for compounds

Starting compounds

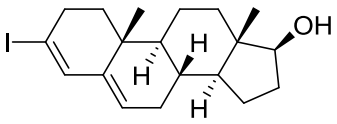
List of organic halides **2a-v** and **4a-d** used as coupling partners:



Vinyl iodides **4e** and **4f** were prepared according to reported procedures through iodination of hydrazones obtained from the corresponding ketosteroids.[1]

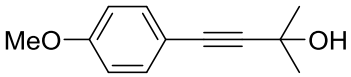


4e

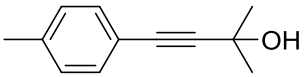


4f

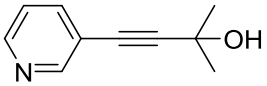
Propargyl alcohols **1a-d**,**f**,**g** were prepared from 2-methylbut-3-yn-2-ol and the corresponding (het)aryl iodides (for **1a-c**,**g**) or bromides (for **1d**,**f**) using standard protocol for Pd/Cu-cocatalyzed Sonogashira cross-coupling. Compound **1e** was obtained by oxidative alkynylation of ferrocene according to the literature.[2]



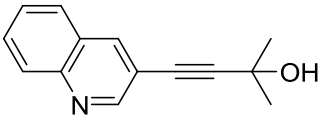
1a



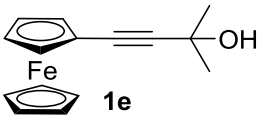
1b



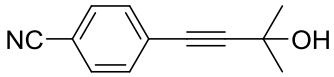
1c



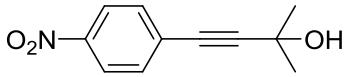
1d



1e

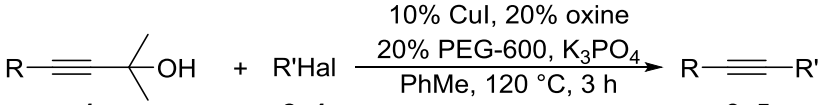


1f



1g

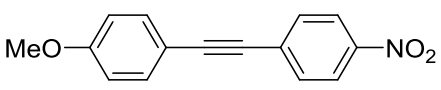
Cu-catalyzed deacetonative Sonogashira coupling



1 **2, 4** **3, 5**

General procedure. In a vial with a screw cap propargyl alcohol **1** (0.24 mmol, 1.2 eqiuv), organic halide **2** or **4** (0.20 mmol, 1.0 equiv), CuI (3.8 mg, 0.020 mmol, 10 mol%), oxine (5.8 mg, 0.040 mmol, 20 mol%), PEG-600 (21.6 μL, 0.040 mmol, 20 mol%), and K_3PO_4 (170 mg, 0.80 mmol, 4.0 equiv) were mixed under an Ar atmosphere in toluene (1 mL). The reaction mixture was stirred at 120 °C for 3 h, then diluted with CH_2Cl_2 (20 mL) and washed with water (20 mL). The organic layer was dried with anhydrous Na_2SO_4, and the solvents were evaporated *in vacuo*. The residue was purified by column chromatography.

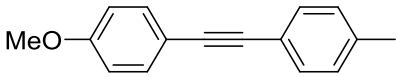
1-Methoxy-4-[(4-nitrophenyl)ethynyl]benzene (3a):



Prepared from 4-(4-methoxyphenyl)-2-methylbut-3-yn-2-ol **1a** (45.6 mg, 0.24 mmol) and 1-iodo-4-nitrobenzene **2a** (49.8 mg, 0.20 mmol) according to the general procedure; eluent: hexanes–PhH = 2:1. Yield 43 mg (85%). Prepared from 4-(4-methoxyphenyl)-2-methylbut-3-yn-2-ol **1a** (45.6 mg, 0.24 mmol)

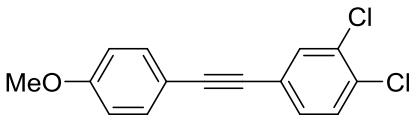
and 1-bromo-4-nitrobenzene **2o** (40.4 mg, 0.20 mmol) according to the general procedure; eluent: hexanes–PhH = 2:1. Yield 14.4 mg (28%). Yellow solid; mp 119–121 °C (lit.[3] 119.5–120.8 °C); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 (m, 2H), 7.62 (m, 2H), 7.49 (m, 2H), 6.90 (m, 2H), 3.84 (s, 3H).

1-Methoxy-4-[(4-methylphenyl)ethynyl]benzene (3b):



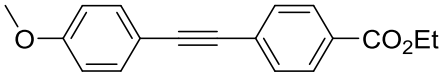
Prepared from 4-(4-methoxyphenyl)-2-methylbut-3-yn-2-ol **1a** (45.6 mg, 0.24 mmol) and 4-iodotoluene **2b** (43.6 mg, 0.20 mmol) according to the general procedure; eluent: hexanes–PhH = 3:1. Yield 39 mg (88%). White solid; mp 126–128 °C (lit.[4] 125.7–128.1 °C); ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.45 (m, 2H), 7.40 (m, 2H), 7.13 (m, 2H), 6.86 (m, 2H), 3.81 (s, 3H), 2.35 (s, 3H).

1,2-Dichloro-4-[(4-methoxyphenyl)ethynyl]benzene (3c):



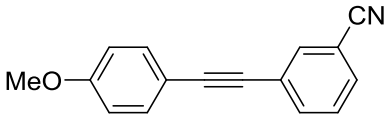
Prepared from 4-(4-methoxyphenyl)-2-methylbut-3-yn-2-ol **1a** (45.6 mg, 0.24 mmol) and 1,2-dichloro-4-iodobenzene **2c** (54.6 mg, 0.20 mmol) according to the general procedure; eluent: hexanes–CH_2Cl_2 = 4:1. Yield 31 mg (56%). White solid; mp 68–70 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (d, J = 1.8 Hz, 1H, 3-CH_{Ar}), 7.45 (m, 2H), 7.39 (d, J = 8.3 Hz, 1H, 6-CH_{Ar}), 7.31 (dd, J = 8.3, 1.8 Hz, 1H, 5-CH_{Ar}), 6.88 (m, 2H), 3.83 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.0 (C_{quat}), 133.1 (2C), 132.9, 132.4 (C_{quat}), 132.1 (C_{quat}), 130.5, 130.3, 123.6 (C_{quat}), 114.5 (C_{quat}), 114.1 (2C), 91.4 (C≡C), 85.8 (C≡C), 55.3 (CH_3); HRMS (ESI-TOF) calcd for $C_{15}H_{11}Cl_2O$ $[M+H]^+$ 277.0181; found 277.0180.

Ethyl 4-[(4-methoxyphenyl)ethynyl]benzoate (3d):



Prepared from 4-(4-methoxyphenyl)-2-methylbut-3-yn-2-ol **1a** (45.6 mg, 0.24 mmol) and ethyl 4-iodobenzoate **2d** (55.2 mg, 0.20 mmol) according to the general procedure; eluent: hexanes–PhH = 1:1. Yield 32.3 mg (58%). White solid; mp 86–87 °C (lit.[5] 90–92 °C); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (m, 2H), 7.56 (m, 2H), 7.48 (m, 2H), 6.89 (m, 2H), 4.38 (q, J = 7.1 Hz, 2H), 3.83 (s, 3H, MeO), 1.40 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.1 (C_{quat}), 159.9 (C_{quat}), 133.2 (2C), 131.2 (2C), 129.4 (2C+C_{quat}), 128.2 (C_{quat}), 114.7 (C_{quat}), 114.0 (2C), 92.4 (C≡C), 87.5 (C≡C), 61.1 (CH_2), 55.3 (CH_3O), 14.3 ($\underline{C}H_3CH_2$).

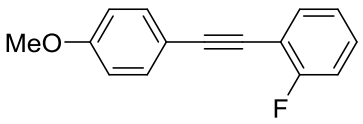
3-[(4-Methoxyphenyl)ethynyl]benzonitrile (3e):



Prepared from 4-(4-methoxyphenyl)-2-methylbut-3-yn-2-ol **1a** (45.6 mg, 0.24 mmol) and 3-iodobenzonitrile **2e** (45.8 mg, 0.20 mmol) according to the general procedure; eluent: hexanes–PhH = 1:2. Yield 33 mg (71%). Prepared from 4-(4-methoxyphenyl)-2-methylbut-3-yn-2-ol **1a** (45.6

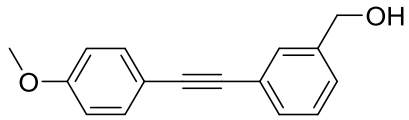
mg, 0.24 mmol) and 3-bromobenzonitrile **2m** (36.4 mg, 0.20 mmol) according to the general procedure; eluent: hexanes–PhH = 1:2. Yield 35.9 mg (77%). Beige solid; mp 66–67 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (m, 1H, 2-CH_{Ar}), 7.70 (m, 1H, 6-CH_{Ar}), 7.57 (m, 1H, 4-CH_{Ar}), 7.47 (m, 2H), 7.44 (m, 1H, 5-CH_{Ar}), 6.89 (m, 2H), 3.83 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.1 (C_{quat}), 135.4, 134.7, 133.2 (2C), 131.0, 129.2, 125.2 (C_{quat}), 118.1 (C_{quat}), 114.3 (C_{quat}), 114.1 (2C), 112.8 (C_{quat}), 91.9 (C≡C), 85.7 (C≡C), 55.3 (CH_3); HRMS (MALDI-TOF, cinnamic acid) calcd for $C_{16}H_{11}KNO$ $[M+K]^+$ 272.0472; found 272.0470.

1-Fluoro-2-[(4-methoxyphenyl)ethynyl]benzene (3f):



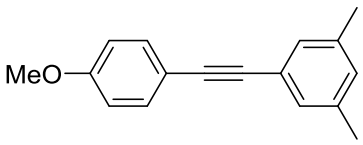
Prepared from 4-(4-methoxyphenyl)-2-methylbut-3-yn-2-ol **1a** (45.6 mg, 0.24 mmol) and 1-fluoro-2-iodobenzene **2f** (44.4 mg, 0.20 mmol) according to the general procedure; eluent: hexanes–PhH = 3:1. Yield 27.4 mg (61%). White solid; mp 57–58 °C (lit.[6] 56–57 °C); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53–7.47 (m, 3H), 7.28 (m, 1H), 7.14–7.06 (m, 2H), 6.88 (m, 2H), 3.83 (s, 3H).

{3-[(4-Methoxyphenyl)ethynyl]phenyl}methanol (3g):



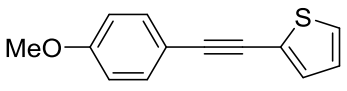
Prepared from 4-(4-methoxyphenyl)-2-methylbut-3-yn-2-ol **1a** (45.6 mg, 0.24 mmol) and (3-iodophenyl)methanol **2g** (46.8 mg, 0.20 mmol) according to the general procedure; eluent: PhH–MeCN = 40:1. Yield 45.9 mg (96%). Light-yellow solid; mp 59–62 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52–7.40 (m, 4H), 7.35–7.26 (m, 2H), 6.86 (m, 2H), 4.65 (s, 2H), 3.80 (s, 3H), 2.06 (br s, 1H); ^{13}C (APT, 100 MHz, $CDCl_3$) δ 159.6 (C_{quat}), 141.0 (C_{quat}), 133.0 (2C), 130.5, 129.8, 128.5, 126.4, 123.7 (C_{quat}), 115.2 (C_{quat}), 114.0 (2C), 89.5 (C_{quat}), 87.9 (C_{quat}), 64.8 (CH_2), 55.2 (CH_3). Spectral data are in accordance with the literature.[7]

1-[(4-Methoxyphenyl)ethynyl]-3,5-dimethylbenzene (3h):



Prepared from 4-(4-methoxyphenyl)-2-methylbut-3-yn-2-ol **1a** (45.6 mg, 0.24 mmol) and 1-iodo-3,5-dimethylbenzene **2h** (46.4 mg, 0.20 mmol) according to the general procedure; eluent: hexanes–PhH = 3:1. Yield 38 mg (81%). White solid; mp 45–46 °C (lit.[8] 41–42 °C); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (m, 2H), 7.14 (br s, 2H), 6.94 (br s, 1H), 6.86 (m, 2H), 3.80 (s, 3H), 2.30 (s, 6H).

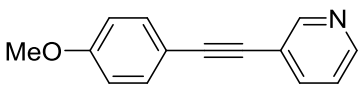
2-[(4-Methoxyphenyl)ethynyl]thiophene (3i):



Prepared from 4-(4-methoxyphenyl)-2-methylbut-3-yn-2-ol **1a** (45.6 mg, 0.24 mmol) and 2-iodothiophene **2i** (42.0 mg, 0.20 mmol) according to the general procedure; eluent: hexanes–CH_2Cl_2 = 1:1. Yield 31.2 mg (73%). Off-white solid; mp 55–57 °C (lit.[9] 53–54 °C); ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.44 (m, 2H), 7.25 (dd,

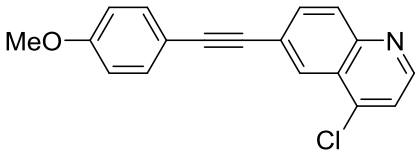
J = 5.2, 1.1 Hz, 1H), 7.24 (dd, *J* = 3.7, 1.1 Hz, 1H), 6.99 (dd, *J* = 5.2, 3.7 Hz, 1H), 6.87 (m, 2H), 3.82 (s, 3H).

3-[(4-Methoxyphenyl)ethynyl]pyridine (3j):



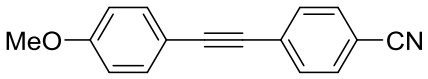
Prepared from 4-(4-methoxyphenyl)-2-methylbut-3-yn-2-ol **1a** (45.6 mg, 0.24 mmol) and 3-iodopyridine **2j** (41.0 mg, 0.20 mmol) according to the general procedure; eluent: hexanes–EtOAc = 1:1. Yield 36.9 mg (88%). Brownish solid; mp 53–55 °C (lit.[10] 51–52 °C); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.75 (br s, 1H, 2-CH_{Py}), 8.52 (d, *J* = 4.0 Hz, 1H, 6-CH_{Py}), 7.79 (m, 1H, 4-CH_{Py}), 7.49 (m, 2H), 7.26 (dd, *J* = 7.9, 5.6 Hz, 1H, 5-CH_{Py}), 6.90 (m, 2H), 3.84 (s, 3H).

4-Chloro-6-[(4-methoxyphenyl)ethynyl]quinoline (3k):



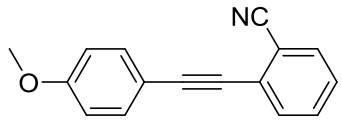
Prepared from 4-(4-methoxyphenyl)-2-methylbut-3-yn-2-ol **1a** (45.6 mg, 0.24 mmol) and 4-chloro-6-iodoquinoline **2k** (57.9 mg, 0.20 mmol) according to the general procedure; eluent: hexanes–EtOAc = 2:1. Yield 30 mg (51%). Light brown solid; mp 135–137 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.74 (d, *J* = 4.7 Hz, 1H, 2-CH_{Ar}), 8.36 (d, *J* = 1.7 Hz, 1H, 5-CH_{Ar}), 8.06 (d, *J* = 8.7 Hz, 1H, 8-CH_{Ar}), 7.83 (dd, *J* = 8.7, 1.7 Hz, 1H, 7-CH_{Ar}), 7.53 (m, 2H), 7.48 (d, *J* = 4.7 Hz, 1H, 3-CH_{Ar}), 6.91 (m, 2H), 3.84 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.9 (C_{quat}), 150.0, 148.2 (C_{quat}), 142.1 (C_{quat}), 133.2 (2C), 133.0, 129.8, 126.9, 126.4 (C_{quat}), 123.1 (C_{quat}), 121.8, 114.7 (C_{quat}), 114.1 (2C), 91.7 (C≡C), 87.6 (C≡C), 55.3 (CH_3); HRMS (MALDI-TOF, cinnamic acid) calcd for $C_{18}H_{13}ClNO$ $[M+H]^+$ 294.0680; found 294.0685.

4-[(4-Methoxyphenyl)ethynyl]benzonitrile (3l):



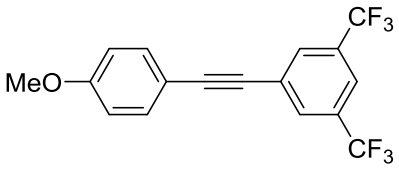
Prepared from 4-(4-methoxyphenyl)-2-methylbut-3-yn-2-ol **1a** (45.6 mg, 0.24 mmol) and 4-bromobenzonitrile **2l** (36.4 mg, 0.20 mmol) according to the general procedure; eluent: hexanes–PhH = 1:2. Yield 36.9 mg (79%). Off-white solid; mp 121–123 °C (lit.[11] 121.7–122.5 °C); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (m, 2H), 7.57 (m, 2H), 7.48 (m, 2H), 6.90 (m, 2H), 3.84 (s, 3H).

2-[(4-Methoxyphenyl)ethynyl]benzonitrile (3m):



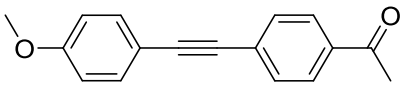
Prepared from 4-(4-methoxyphenyl)-2-methylbut-3-yn-2-ol **1a** (45.6 mg, 0.24 mmol) and 2-bromobenzonitrile **2n** (36.4 mg, 0.20 mmol) according to the general procedure; eluent: hexanes–PhH = 1:2. Yield 40.9 mg (88%). Light-yellow solid; mp 79–81 °C (lit.[12] 78–79 °C); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (ddd, *J* = 7.8, 1.3, 0.6 Hz, 1H), 7.61–7.51 (m, 4H), 7.37 (ddd, *J* = 7.8, 7.4, 1.5 Hz, 1H), 6.89 (m, 2H), 3.84 (s, 3H).

1-[(4-Methoxyphenyl)ethynyl]-3,5-bis(trifluoromethyl)benzene (3n):



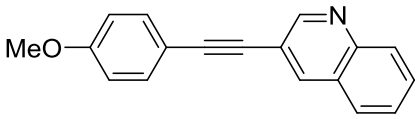
Prepared from 4-(4-methoxyphenyl)-2-methylbut-3-yn-2-ol **1a** (45.6 mg, 0.24 mmol) and 3,5-bis(trifluoromethyl)benzene **2p** (25.5 μL, 0.20 mmol) according to the general procedure; eluent: hexanes–PhH = 1:1. Yield 42 mg (61%). White solid; mp 68–69 °C (lit.[13] 67.9–68.3 °C); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (br s, 2H), 7.78 (br s, 1H), 7.49 (m, 2H), 6.91 (m, 2H), 3.84 (s, 3H).

1-{4-[(4-Methoxyphenyl)ethynyl]phenyl}ethanone (3o):



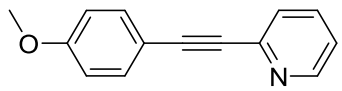
Prepared from 4-(4-methoxyphenyl)-2-methylbut-3-yn-2-ol **1a** (45.6 mg, 0.24 mmol) and 4-bromoacetophenone **2q** (39.8 mg, 0.20 mmol) according to the general procedure; eluent: PhH–MeCN = 40:1. Yield 47.2 mg (94%). Light-yellow solid; mp 127–129 °C (lit.[14] 125–128 °C); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (m, 2H), 7.58 (m, 2H), 7.49 (m, 2H), 6.91 (m, 2H), 3.84 (s, 3H, MeO), 2.61 (s, 3H, Ac).

3-[(4-Methoxyphenyl)ethynyl]quinoline (3p):



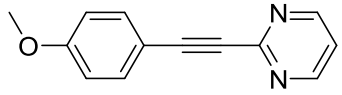
Prepared from 4-(4-methoxyphenyl)-2-methylbut-3-yn-2-ol **1a** (45.6 mg, 0.24 mmol) and 3-bromoquinoline **2r** (27.1 μL, 0.20 mmol) according to the general procedure; eluent: hexanes–EtOAc = 1:1. Yield 41.3 mg (80%). Off-white solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.99 (d, J = 2.0 Hz, 1H, 2-CH_{Ar}), 8.26 (d, J = 2.0 Hz, 1H, 4-CH_{Ar}), 8.09 (m, 1H), 7.78 (m, 1H), 7.70 (m, 1H), 7.55 (m, 1H), 7.53 (m, 2H), 6.91 (m, 2H), 3.83 (s, 3H). Spectral data are in accordance with the literature.[15]

2-[(4-Methoxyphenyl)ethynyl]pyridine (3q):



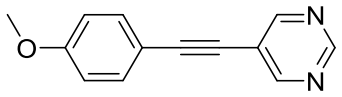
Prepared from 4-(4-methoxyphenyl)-2-methylbut-3-yn-2-ol **1a** (45.6 mg, 0.24 mmol) and 2-bromopyridine **2s** (31.6 mg, 0.20 mmol) according to the general procedure; eluent: hexanes–EtOAc = 3:1. Yield 25.3 mg (61%). Yellow liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.60 (ddd, J = 4.9, 1.8, 1.1 Hz, 1H), 7.66 (td, J = 7.7, 1.8 Hz, 1H), 7.54 (m, 2H), 7.49 (dt, J = 7.7, 1.1 Hz, 1H), 7.21 (ddd, J = 7.7, 4.9, 1.1 Hz, 1H), 6.89 (m, 2H), 3.83 (s, 3H). Spectral data are in accordance with the literature.[10]

2-[(4-Methoxyphenyl)ethynyl]pyrimidine (3r):



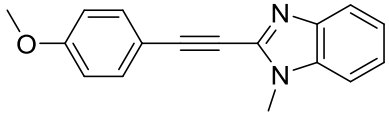
Prepared from 4-(4-methoxyphenyl)-2-methylbut-3-yn-2-ol **1a** (45.6 mg, 0.24 mmol) and 2-bromopyrimidine **2t** (31.8 mg, 0.20 mmol) according to the general procedure; eluent: hexanes–EtOAc = 2:1. Yield 6.8 mg (16%). White solid; mp 70–73 °C (lit.[10] 70–72 °C); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.74 (d, J = 4.9 Hz, 2H), 7.62 (m, 2H), 7.22 (t, J = 4.9 Hz, 1H), 6.91 (m, 2H), 3.84 (s, 3H).

5-[(4-Methoxyphenyl)ethynyl]pyrimidine (3s):



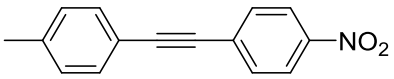
Prepared from 4-(4-methoxyphenyl)-2-methylbut-3-yn-2-ol **1a** (45.6 mg, 0.24 mmol) and 5-bromopyrimidine **2u** (31.8 mg, 0.20 mmol) according to the general procedure; eluent: hexanes–EtOAc = 3:1. Yield 30.2 mg (72%). Light-yellow solid; mp 115–117 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.12 (s, 1H), 8.83 (s, 2H), 7.50 (m, 2H), 6.91 (m, 2H), 3.84 (s, 3H); ^{13}C (100 MHz, $CDCl_3$) δ 160.3 (C_{quat}), 158.4 (2C), 156.3, 133.3 (2C), 120.2 (C_{quat}), 114.2 (2C), 113.7 (C_{quat}), 96.5 (C_{quat}), 81.4 (C_{quat}), 55.3. Spectral data are in accordance with the literature.[16]

2-[(4-Methoxyphenyl)ethynyl]-1-methyl-1*H*-benzimidazole (3t):



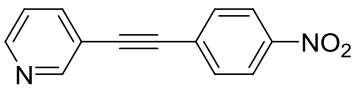
Prepared from 4-(4-methoxyphenyl)-2-methylbut-3-yn-2-ol **1a** (45.6 mg, 0.24 mmol) and 2-bromo-1-methyl-1*H*-benzimidazole **2v** (42.2 mg, 0.20 mmol) according to the general procedure; eluent: hexanes–EtOAc = 3:1. Yield 20.5 mg (39%). Light-yellow solid; mp 160–163 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (m, 1H), 7.57 (m, 2H), 7.33–7.27 (m, 3H), 6.91 (m, 2H), 3.90 (s, 3H), 3.84 (s, 3H). Spectral data are in accordance with the literature.[17]

1-Methyl-4-[(4-nitrophenyl)ethynyl]benzene (3u):



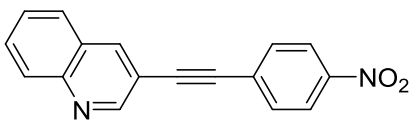
Prepared from 2-methyl-4-(4-methylphenyl)but-3-yn-2-ol **1b** (41.8 mg, 0.24 mmol) and 1-iodo-4-nitrobenzene **2a** (49.8 mg, 0.20 mmol) according to the general procedure; eluent: hexanes–CH_2Cl_2 = 3:1. Yield 30.8 mg (65%). Yellow solid; mp 153–155 °C (lit.[18] 154–156 °C); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.20 (m, 2H), 7.64 (m, 2H), 7.45 (m, 2H), 7.19 (m, 2H), 2.39 (s, 3H).

3-[(4-Nitrophenyl)ethynyl]pyridine (3v):



Prepared from 2-methyl-4-pyridine-3-ylbut-3-yn-2-ol **1c** (38.6 mg, 0.24 mmol) and 1-iodo-4-nitrobenzene **2a** (49.8 mg, 0.20 mmol) according to the general procedure. Yield 36.3 mg (81%). Light orange solid; mp 135–136 °C (lit.[18] 107–109 °C); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.87 (br s, 1H), 8.69 (br s, 1H), 8.26 (m, 2H), 7.86 (br d, J = 7.6 Hz, 1H), 7.70 (m, 2H), 7.37 (br s, 1H).

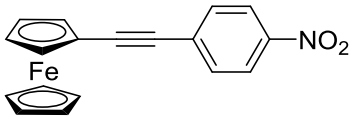
3-[(4-Nitrophenyl)ethynyl]quinoline (3w):



Prepared from 4-quinolin-3-yl-2-methylbut-3-yn-2-ol **1d** (50.6 mg, 0.24 mmol) and 1-iodo-4-nitrobenzene **2a** (49.8 mg, 0.20 mmol) according to the general procedure; eluent: CH_2Cl_2. Yield 45 mg (82%). Light brown solid; mp 192–194 °C; ^{1}H NMR (400 MHz, DMSO-d_6–CCl_4) δ 9.03 (d, J = 1.5 Hz, 1H, 2-CH_{Ar}), 8.69 (d, J = 1.5 Hz, 1H, 4-CH_{Ar}), 8.29 (m, 2H), 8.07–8.00 (m, 2H, 5,8-CH_{Ar}), 7.88 (m, 2H), 7.83 (m, 1H), 7.68 (m, 1H); ^{13}C NMR (100 MHz, DMSO-d_6–CCl_4)

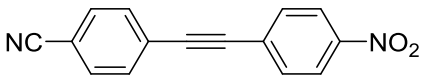
δ 151.4, 147.1 (C_{quat}), 146.6 (C_{quat}), 139.2, 132.7 (2C), 131.0, 128.9, 128.6 (C_{quat}), 128.2, 127.6, 126.7 (C_{quat}), 124.0 (2C), 115.5 (C_{quat}), 91.5 (C≡C), 90.5 (C≡C); HRMS (MALDI-TOF, cinnamic acid) calcd for $C_{17}H_{11}N_2O_2$ $[M+H]^+$ 275.0815; found 275.0818.

[(4-Nitrophenyl)ethynyl]ferrocene (3x):



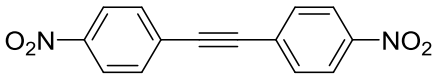
Prepared from 4-ferrocenyl-2-methylbut-3-yn-2-ol **1e** (64.4 mg, 0.24 mmol) and 1-iodo-4-nitrobenzene **2a** (49.8 mg, 0.20 mmol) according to the general procedure; eluent: hexanes–CH_2Cl_2 = 3:1. Yield 29.9 mg (45%). Dark red solid; mp 211–212 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 (m, 2H), 7.59 (m, 2H), 4.55 (m, 2H), 4.32 (m, 2H), 4.26 (s, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.4 (C_{quat}), 131.8 (2C), 131.0 (C_{quat}), 123.6 (2C), 95.2 (C≡C), 84.4 (C≡C), 71.8 (2C), 70.1 (5C), 69.5 (2C), 63.6 (C_{quat}); HRMS (MALDI-TOF, dithranol) calcd for $C_{18}H_{13}FeNO_2$ $[M]^+$ 331.0290; found 331.0282.

4-[(4-Nitrophenyl)ethynyl]benzonitrile (3y):



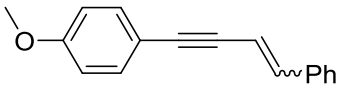
Prepared from 4-(3-hydroxy-3-methylbut-1-yn-1-yl)benzonitrile **1f** (44.5 mg, 0.24 mmol) and 1-iodo-4-nitrobenzene **2a** (49.8 mg, 0.20 mmol) according to the general procedure; eluent: hexanes–CH_2Cl_2 = 1:1. Yield 32.2 mg (65%). Beige solid; mp 211–213 °C (lit.[19] 212–214 °C); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.25 (m, 2H), 7.73–7.63 (m, 6H).

1,1'-Ethyne-1,2-diylbis(4-nitrobenzene) (3z):



Prepared from 2-methyl-4-(4-nitrophenyl)but-3-yn-2-ol **1g** (49.3 mg, 0.24 mmol) and 1-iodo-4-nitrobenzene **2a** (49.8 mg, 0.20 mmol) according to the general procedure; eluent: hexanes–CH_2Cl_2 = 1:1. Yield 33.5 mg (62%). Pale yellow solid; mp 213–214 °C (lit.[20] 214–215 °C); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.27 (m, 4H), 7.72 (m, 4H).

1-Methoxy-4-[4-phenylbut-3-en-1-yn-1-yl]benzene (*E*:*Z* = 10:1) (5a):

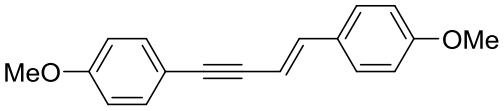


Prepared from 4-(4-methoxyphenyl)-2-methylbut-3-yn-2-ol **1a** (45.6 mg, 0.24 mmol) and β-bromostyrene **4a** (36.6 mg, 0.20 mmol, *E*:*Z* = 85:15) according to the general procedure; eluent: hexanes–PhH = 4:1. Yield 40.5 mg (87%). White solid (*E*:*Z* = 10:1).

1-Methoxy-4-[(3*E*)-4-phenylbut-3-en-1-yn-1-yl]benzene: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44–7.38 (m, 4H), 7.36–7.30 (m, 2H), 7.30–7.25 (m, 1H), 7.00 (d, *J* = 16.2 Hz, 1H), 6.86 (m, 2H), 6.38 (d, *J* = 16.2 Hz, 1H), 3.81 (s, 3H). Spectral data are in accordance with the literature.[20]

1-Methoxy-4-[(3*Z*)-4-phenylbut-3-en-1-yn-1-yl]benzene: partial ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (m, 2H), 6.88 (m, 2H), 6.66 (d, J = 11.9 Hz, 1H), 5.90 (d, J = 11.9 Hz, 1H), 3.82 (s, 3H). Spectral data are in accordance with the literature.[21]

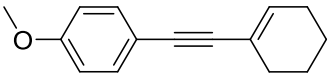
1,1'-(*1E*)-But-1-en-3-yne-1,4-diylbis(4-methoxybenzene) and 1,1'-(*1Z*)-but-1-en-3-yne-1,4-diylbis(4-methoxybenzene) (5b):



Prepared from 4-(4-methoxyphenyl)-2-methylbut-3-yn-2-ol **1a** (45.6 mg, 0.24 mmol) and (*E*)-1-(2-bromovinyl)-4-methoxybenzene **4b** (42.6 mg, 0.20 mmol) according to the general procedure; eluent: hexanes–PhH = 3:1. Yield 41.7 mg (79%). White plates; mp 151–153 °C (lit.[22] 153–155 °C); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (m, 2H), 7.37 (m, 2H), 6.97 (d, J = 16.2 Hz, 1H), 6.87 (m, 4H), 6.24 (d, J = 16.2 Hz, 1H), 3.829 (s, 3H), 3.826 (s, 3H).

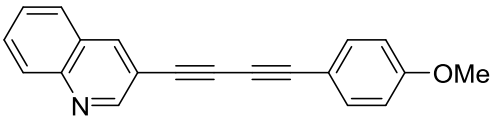
Mixture of *E*/*Z* isomers (2:5) was prepared by the same procedure from 1-(2-bromovinyl)-4-methoxybenzene (*E*/*Z* = 1:10, 42.6 mg, 0.20 mmol). Yield 13.4 mg (25%). (*Z*)-isomer: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (m, 2H), 7.43 (m, 2H), 6.90 (m, 4H), 6.60 (d, J = 11.9 Hz, 1H), 5.79 (d, J = 11.9 Hz, 1H), 3.84 (s, 3H), 3.83 (s, 3H). Spectral data are in accordance with the literature.[22]

1-(Cyclohex-1-en-1-ylethynyl)-4-methoxybenzene (5c):



Prepared from 4-(4-methoxyphenyl)-2-methylbut-3-yn-2-ol **1a** (45.6 mg, 0.24 mmol) and 1-bromocyclohexene **4c** (32.2 mg, 0.20 mmol) according to the general procedure; eluent: hexanes–PhH = 5:1. Yield 7.6 mg (18%). White glass; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 (m, 2H), 6.82 (m, 2H), 6.17 (m, 1H), 3.80 (s, 3H), 2.25–2.19 (m, 2H), 2.17–2.10 (m, 2H), 1.71–1.57 (m, 4H). Spectral data are in accordance with the literature.[23]

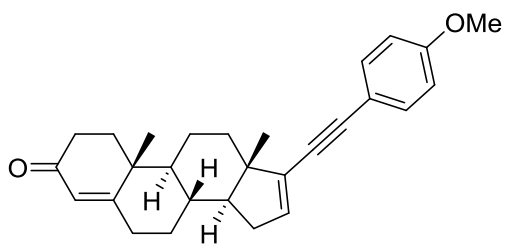
3-[4-(4-Methoxyphenyl)buta-1,3-diyn-1-yl]quinoline (5d):



Prepared from 4-quinolin-3-yl-2-methylbut-3-yn-2-ol **1d** (50.6 mg, 0.24 mmol) and 1-(chloroethynyl)-4-methoxybenzene **4d** (33.3 mg, 0.20 mmol) according to the general procedure; eluent: hexanes–EtOAc = 3:1. Yield 24.1 mg (43%). Light-yellow solid; mp 112–114 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.96 (d, J = 1.5 Hz, 1H, 2-CH_{Ar}), 8.29 (d, J = 1.5 Hz, 1H, 4-CH_{Ar}), 8.09 (m, 1H), 7.78 (m, 1H), 7.73 (m, 1H), 7.57 (m, 1H), 7.50 (m, 2H), 6.87 (m, 2H), 3.83 (s, 3H); ^{13}C (100 MHz, $CDCl_3$) δ 160.6 (C_{quat}), 152.2, 146.9 (C_{quat}), 139.5, 134.2 (2C), 130.5, 129.4, 127.6, 127.5, 127.0 (C_{quat}), 116.3 (C_{quat}), 114.2 (2C), 113.2 (C_{quat}), 83.2 (C_{quat}),

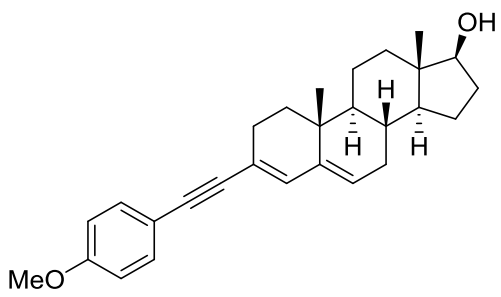
78.2 (C_{quat}), 77.4 (C_{quat}), 72.4 (C_{quat}), 55.3; HRMS (ESI-TOF) calcd for $C_{20}H_{14}NO$ $[M+H]^+$ 284.1070; found 284.1073.

17-[(4-Methoxyphenyl)ethynyl]androsta-4,16-dien-3-one (5e):



Prepared from 4-(4-methoxyphenyl)-2-methylbut-3-yn-2-ol **1a** (45.6 mg, 0.24 mmol) and 17-iodoandrosta-3,16-dien-3-one **4e** (79.2 mg, 0.20 mmol) according to the general procedure; eluent: hexanes–EtOAc = 4:1. Yield 67.0 mg (84%). White solid; mp 221–223 °C (lit.[24] 221–223 °C); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (m, 2H), 6.83 (m, 2H), 6.04 (dd, J = 3.3, 2.1 Hz, 1H, 16-CH), 5.74 (m, 1H, 4-CH), 3.80 (s, 3H, CH_3O), 2.49–2.21 (m, 5H), 2.11–1.84 (m, 4H), 1.81–1.64 (m, 3H), 1.60–1.37 (m, 3H), 1.22 (s, 3H, CH_3), 1.18–0.98 (m, 2H), 0.94 (s, 3H, CH_3).

3-[(4-Methoxyphenyl)ethynyl]androsta-3,5-dien-17β-ol (5f):

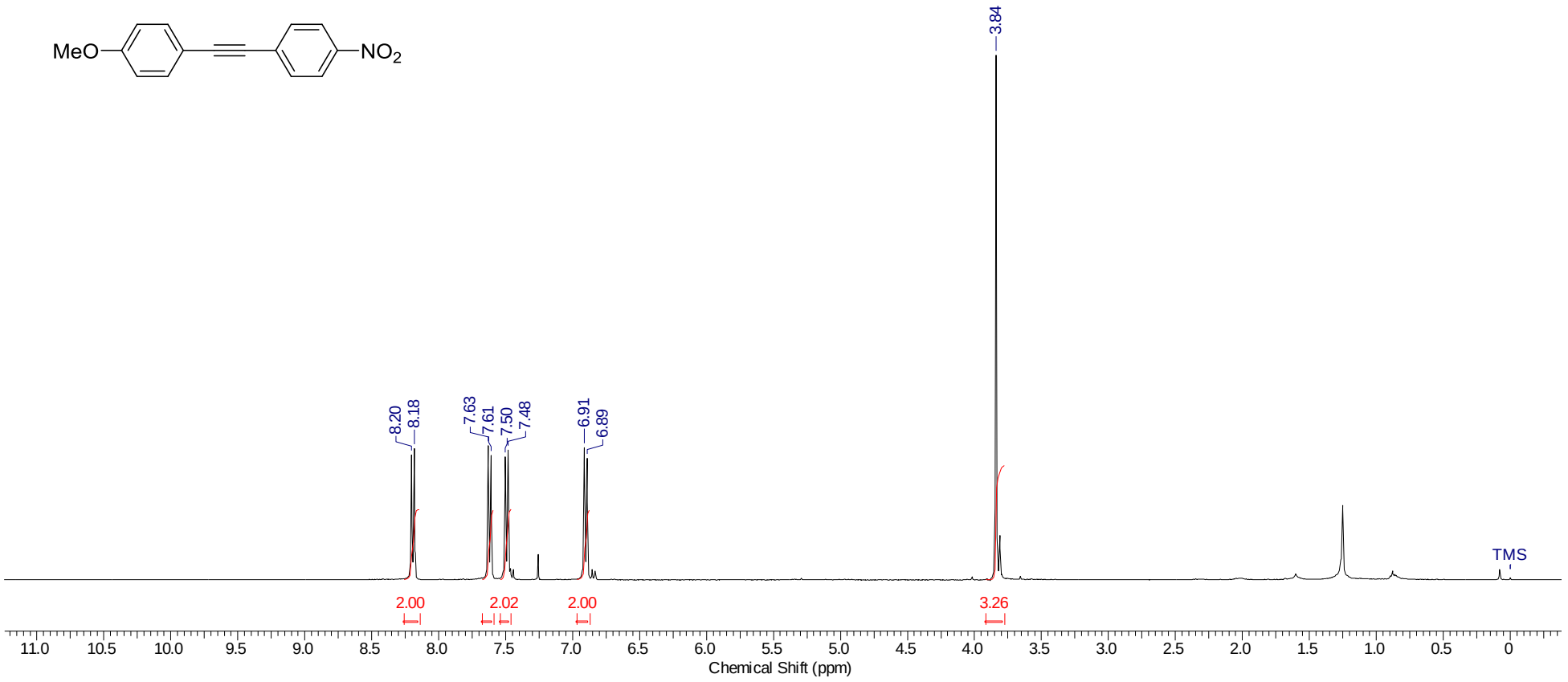


Prepared from 4-(4-methoxyphenyl)-2-methylbut-3-yn-2-ol **1a** (45.6 mg, 0.24 mmol) and 3-iodoandrosta-3,5-dien-17β-ol **4f** (79.7 mg, 0.20 mmol) according to the general procedure; eluent: hexanes–EtOAc = 3:1. Yield 67.3 mg (84%). White solid; mp 213–215 °C; ^{1}H NMR (400 MHz, $CDCl_3$–CD_3OD) δ 7.37 (m, 2H), 6.83 (m, 2H), 6.34 (d, J = 2.1 Hz, 1H, 4-CH), 5.51 (m, 1H, 6-CH), 3.81 (s, 3H, CH_3O), 3.64 (t, J = 8.6 Hz, 1H, 17-C*H*OH), 2.46–2.18 (m, 3H), 2.06 (m, 1H), 1.89–1.82 (m, 2H), 1.76–1.57 (m, 4H), 1.52–1.18 (m, 5H), 1.16–0.96 (m, 2H), 0.98 (s, 3H, CH_3), 0.78 (s, 3H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$–CD_3OD) δ 159.1 (C_{quat}), 141.4 (C_{quat}), 134.7, 132.7 (2C), 125.6, 117.3 (C_{quat}), 115.8 (C_{quat}), 113.8 (2C), 90.1 (C≡C), 89.1 (C≡C), 81.5 (17-CHOH), 55.2 (CH_3O), 51.4, 48.1, 42.7 (C_{quat}), 36.4, 34.5 (C_{quat}), 33.5, 31.7, 31.5, 30.1, 27.0, 23.2, 20.5, 19.0, 10.9; HRMS (MALDI-TOF, dithranol) calcd for $C_{28}H_{35}O_2$ $[M+H]^+$ 403.2632; found 403.2638.

Copies of NMR spectra

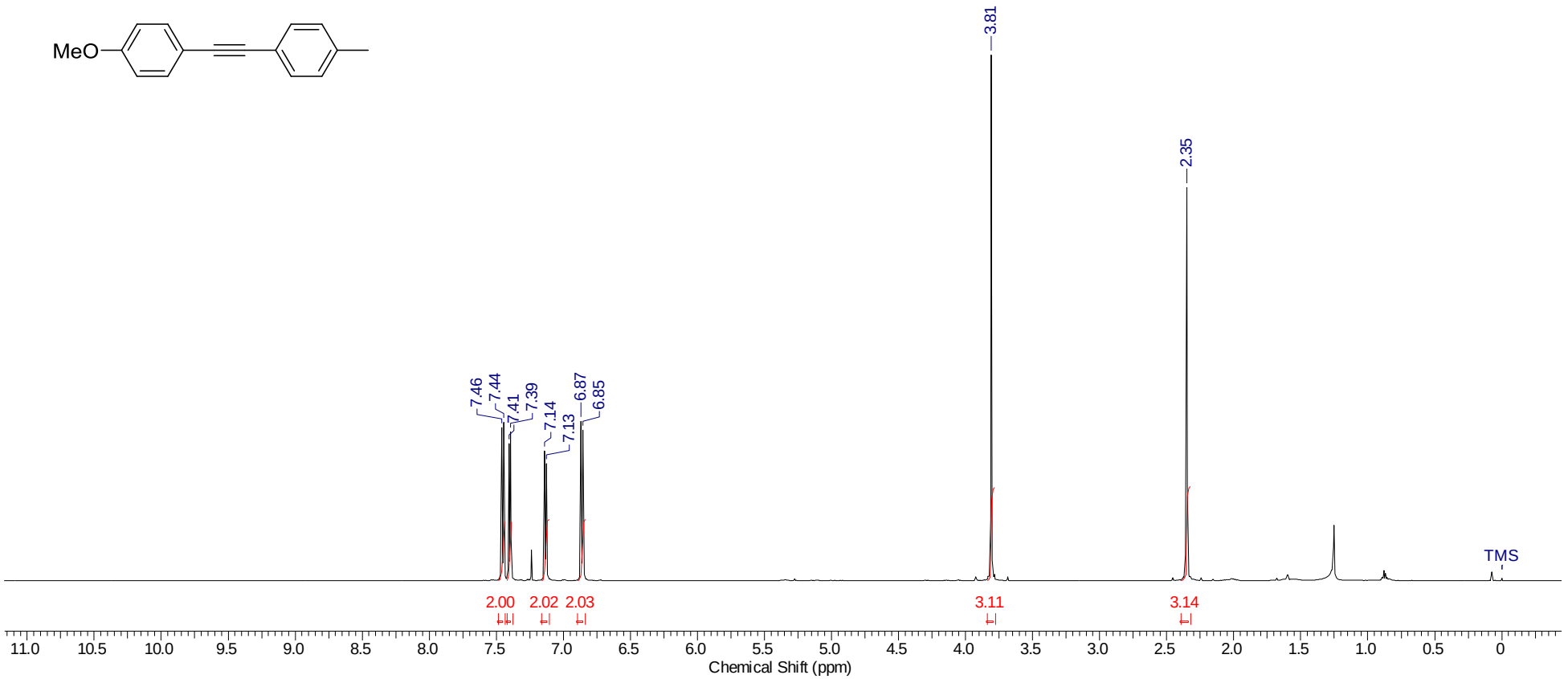
1-Methoxy-4-[(4-nitrophenyl)ethynyl]benzene (3a)

^{1}H NMR (400 MHz, $CDCl_3$)



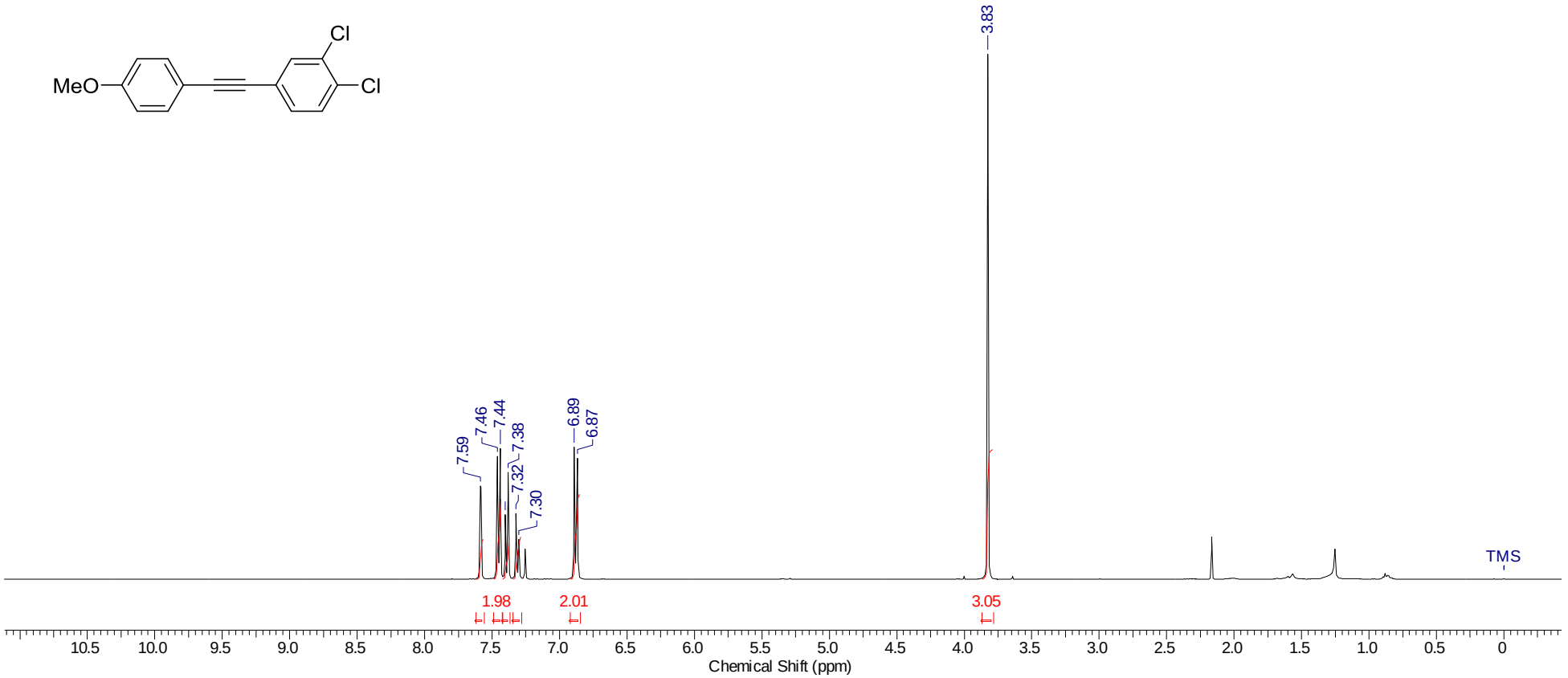
1-Methoxy-4-[(4-methylphenyl)ethynyl]benzene (3b)

^{1}H NMR (600 MHz, $CDCl_3$)



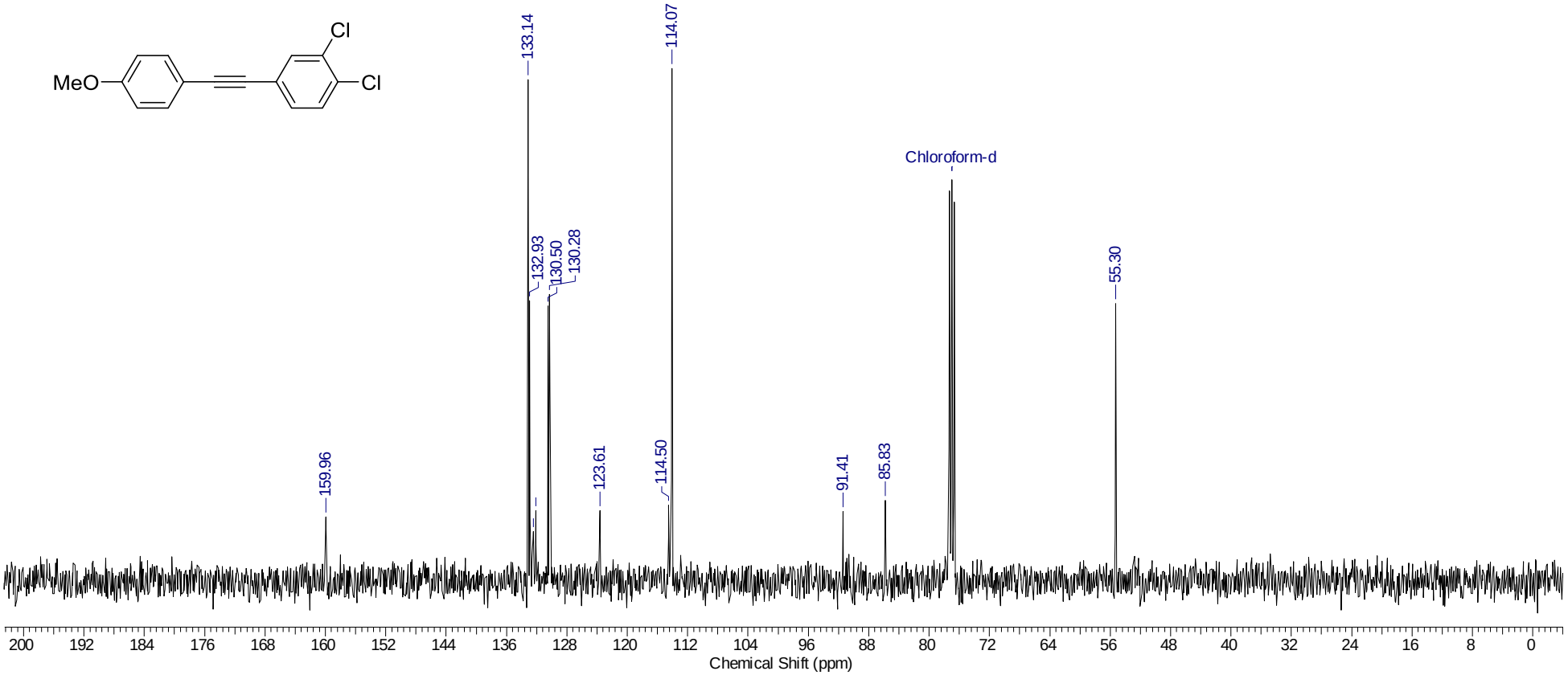
1,2-Dichloro-4-[(4-methoxyphenyl)ethynyl]benzene (3c)

^{1}H NMR (400 MHz, $CDCl_3$)



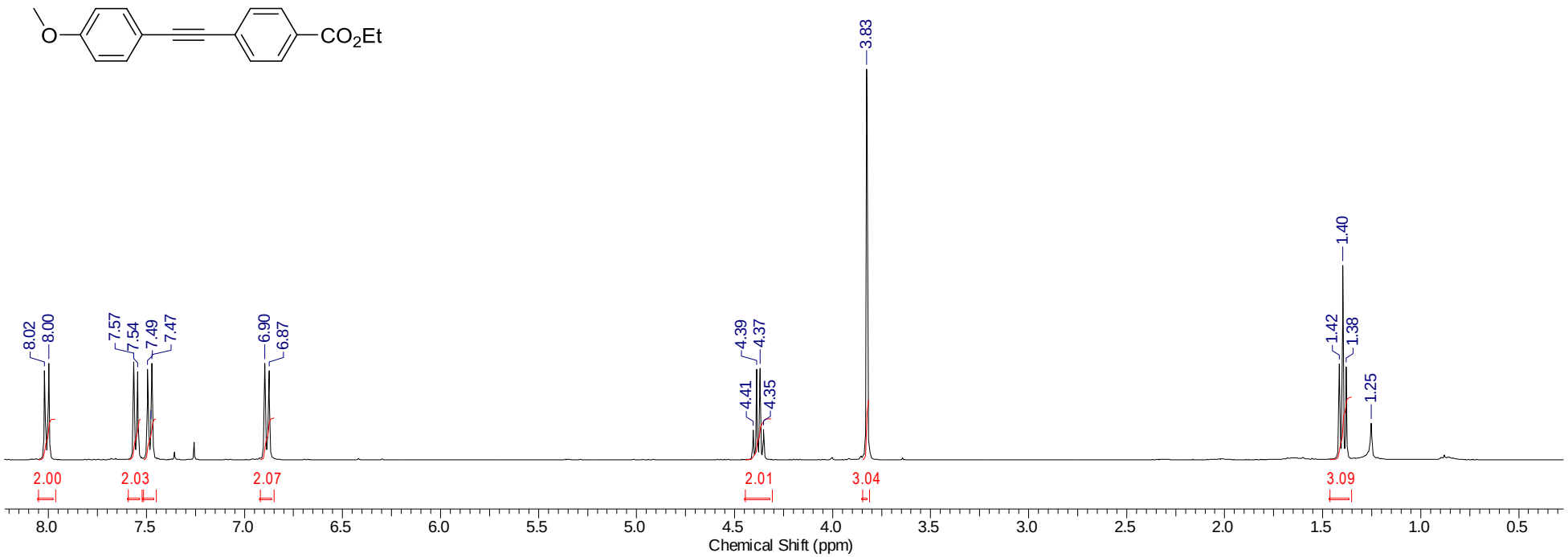
1,2-Dichloro-4-[(4-methoxyphenyl)ethynyl]benzene (3c)

^{13}C NMR (400 MHz, $CDCl_3$)



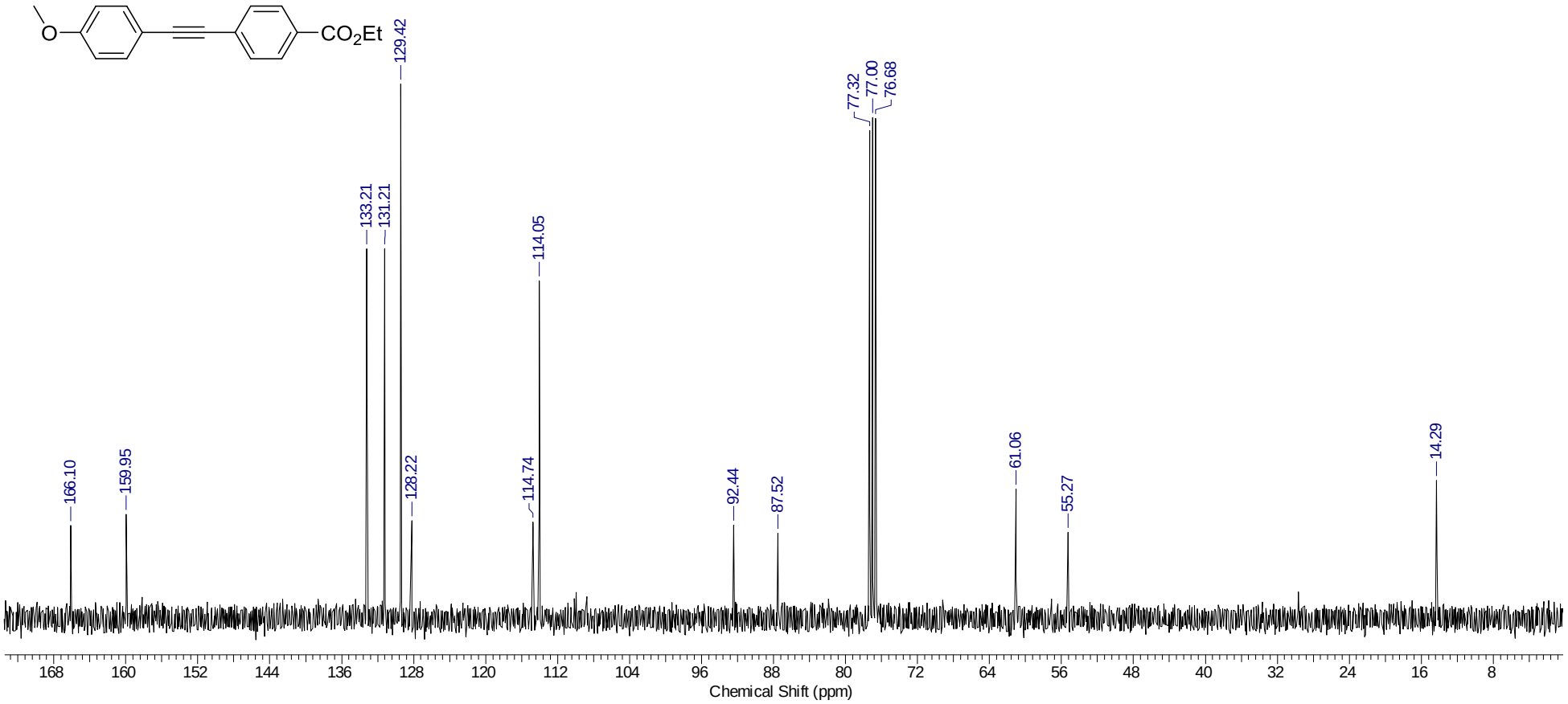
Ethyl 4-[(4-methoxyphenyl)ethynyl]benzoate (3d)

^{1}H NMR (400 MHz, $CDCl_3$)



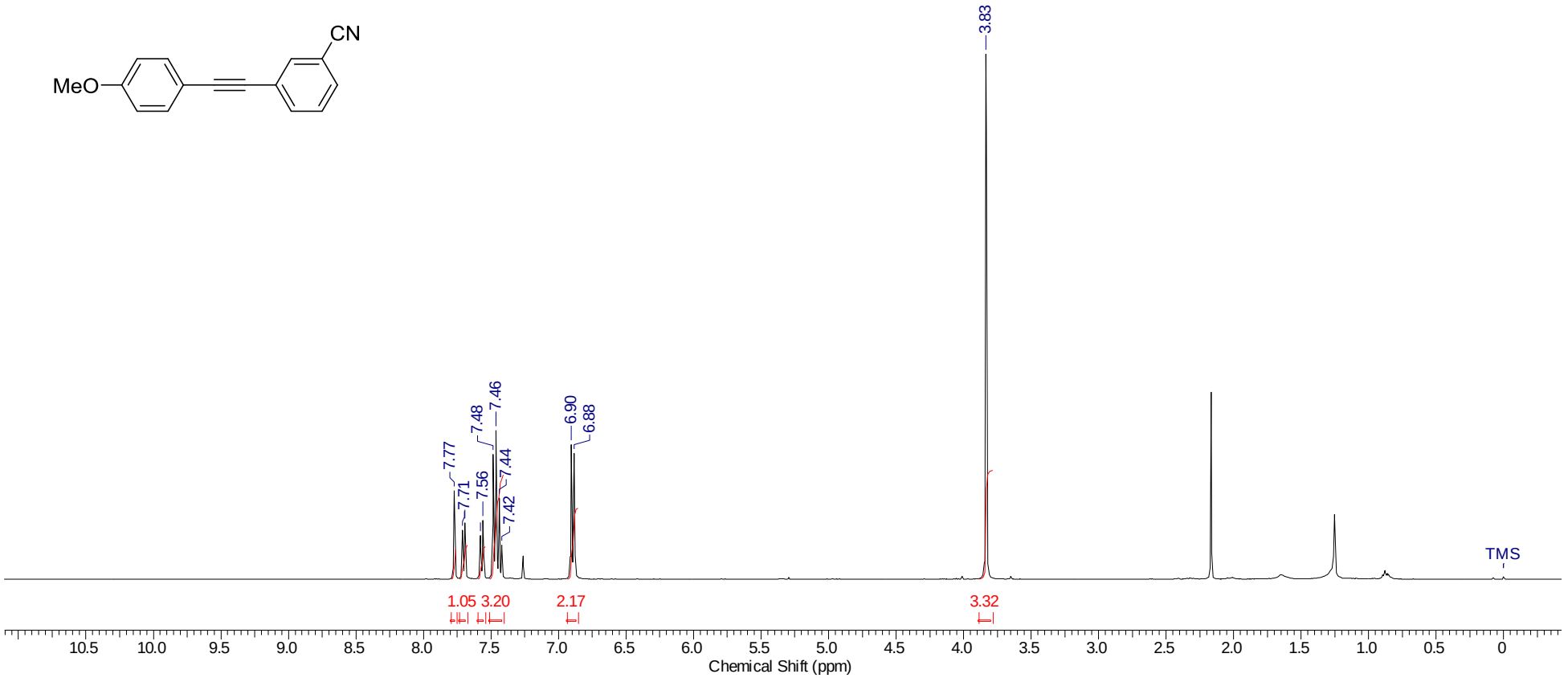
Ethyl 4-[(4-methoxyphenyl)ethynyl]benzoate (3d)

^{13}C NMR (100 MHz, $CDCl_3$)



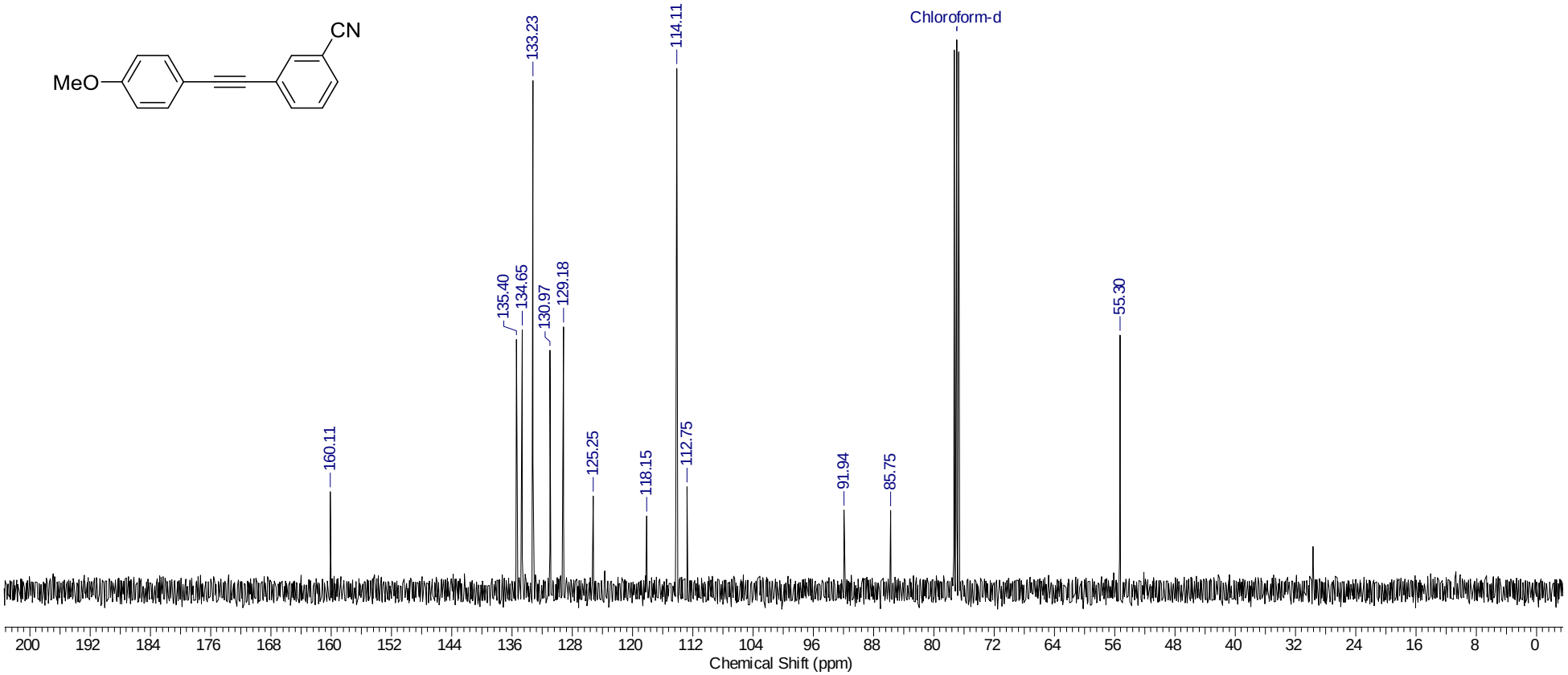
3-[(4-Methoxyphenyl)ethynyl]benzonitrile (3e)

^{1}H NMR (400 MHz, $CDCl_3$)



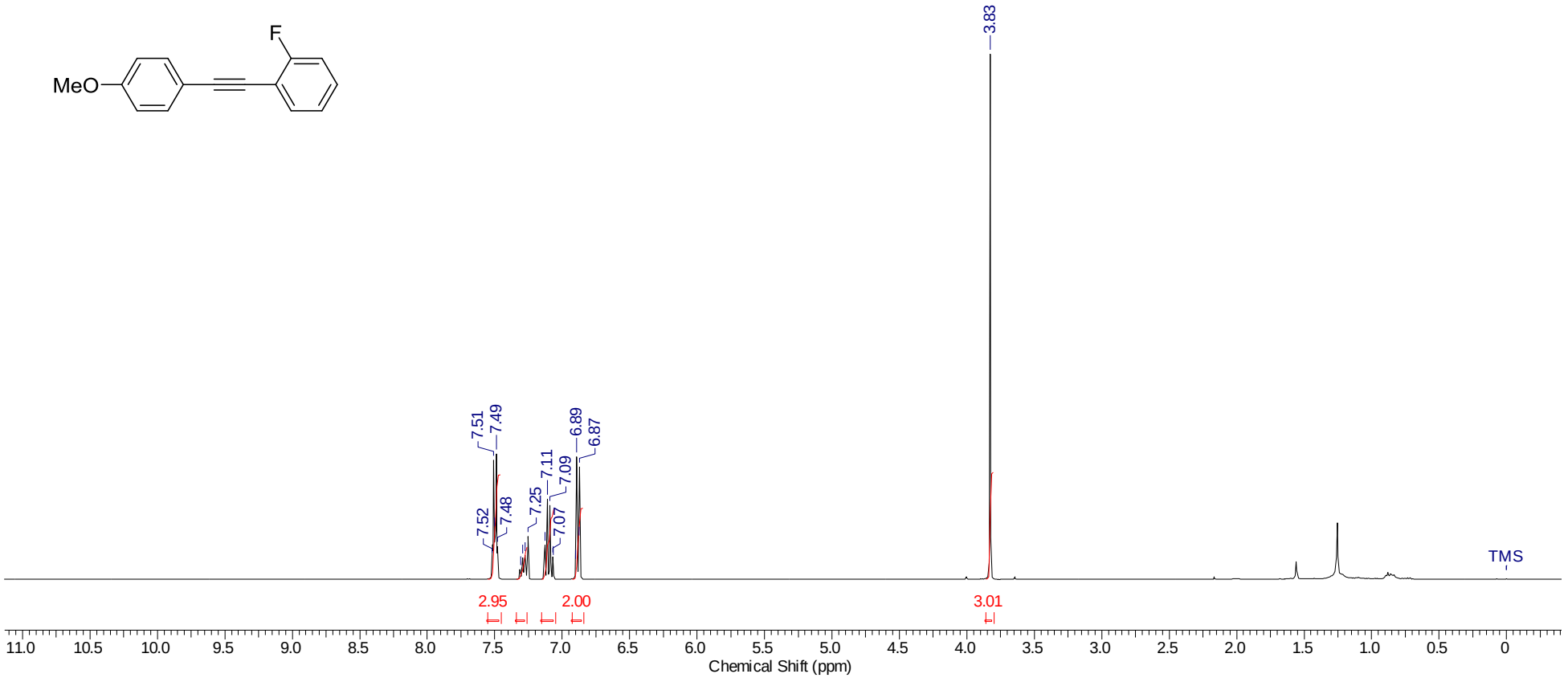
3-[(4-Methoxyphenyl)ethynyl]benzonitrile (3e)

^{13}C NMR (100 MHz, $CDCl_3$)



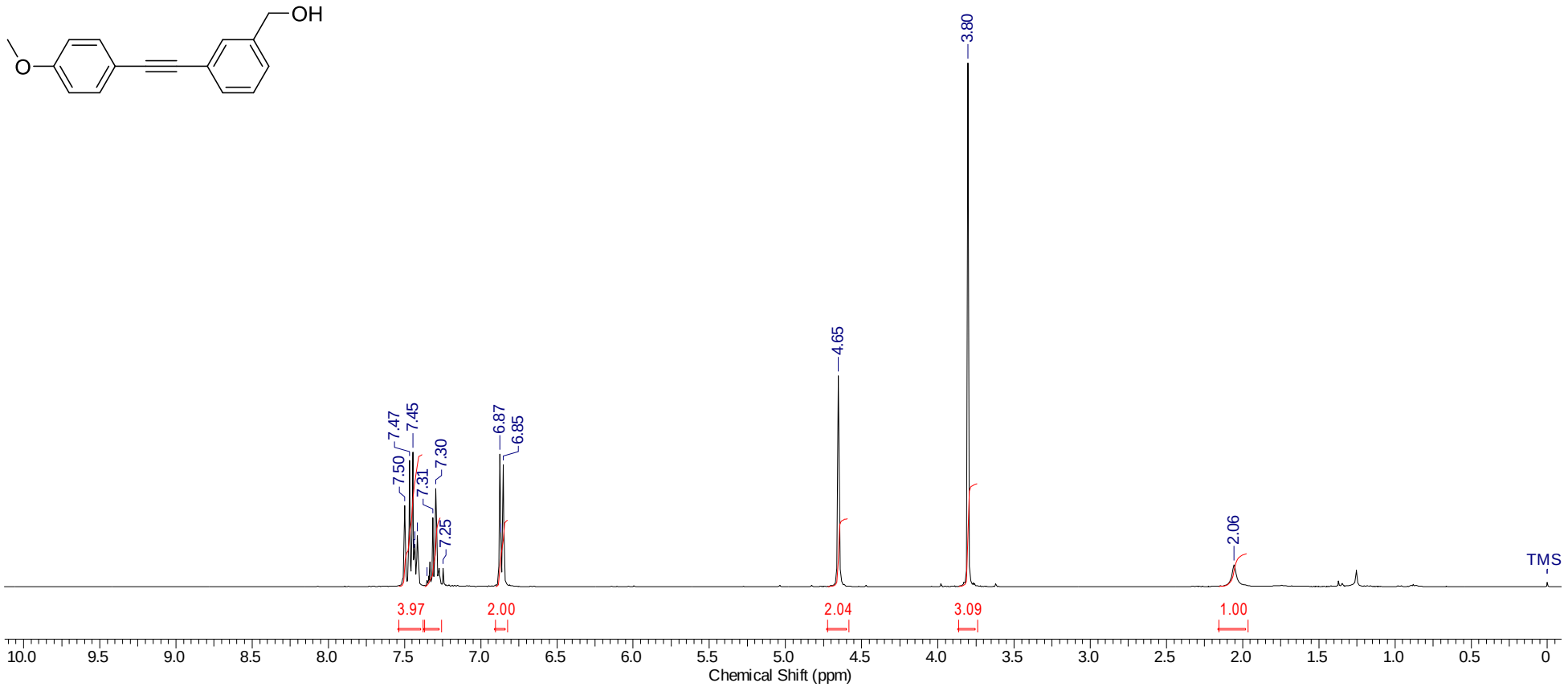
1-Fluoro-2-[(4-methoxyphenyl)ethynyl]benzene (3f)

^{1}H NMR (400 MHz, $CDCl_3$)



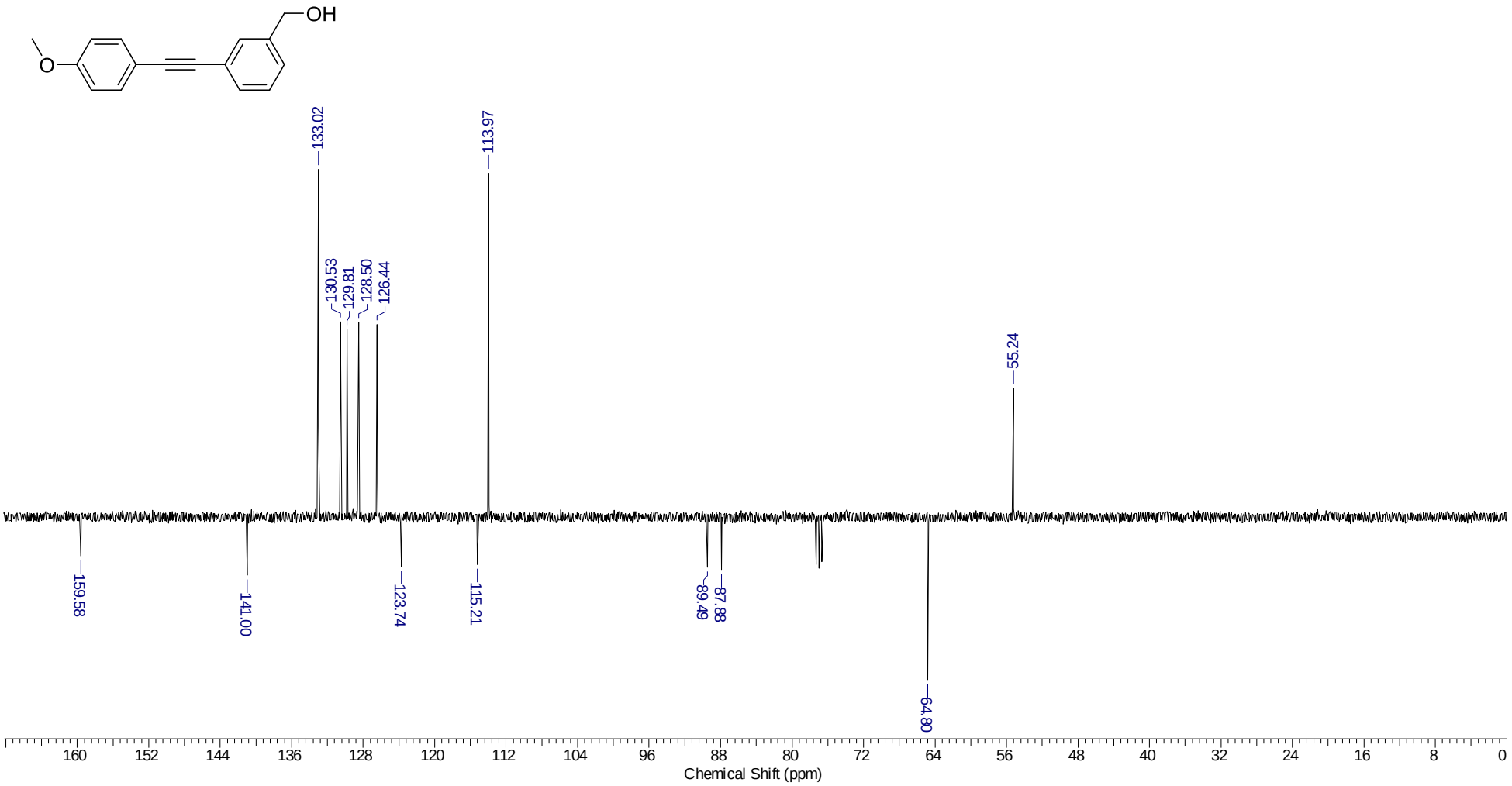
{3-[(4-Methoxyphenyl)ethynyl]phenyl}methanol (3g)

^{1}H NMR (400 MHz, $CDCl_3$)



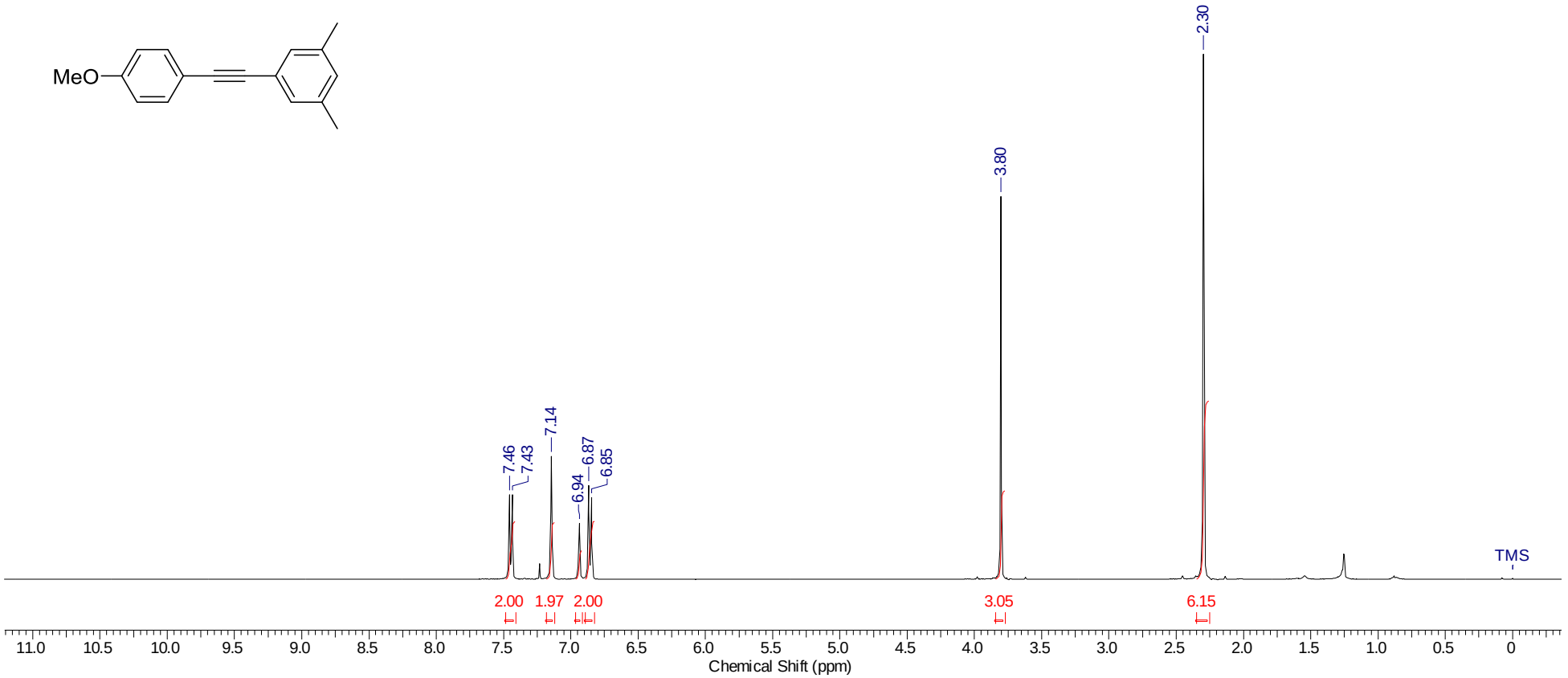
{3-[(4-Methoxyphenyl)ethynyl]phenyl}methanol (3g)

^{13}C NMR (APT, 100 MHz, $CDCl_3$)



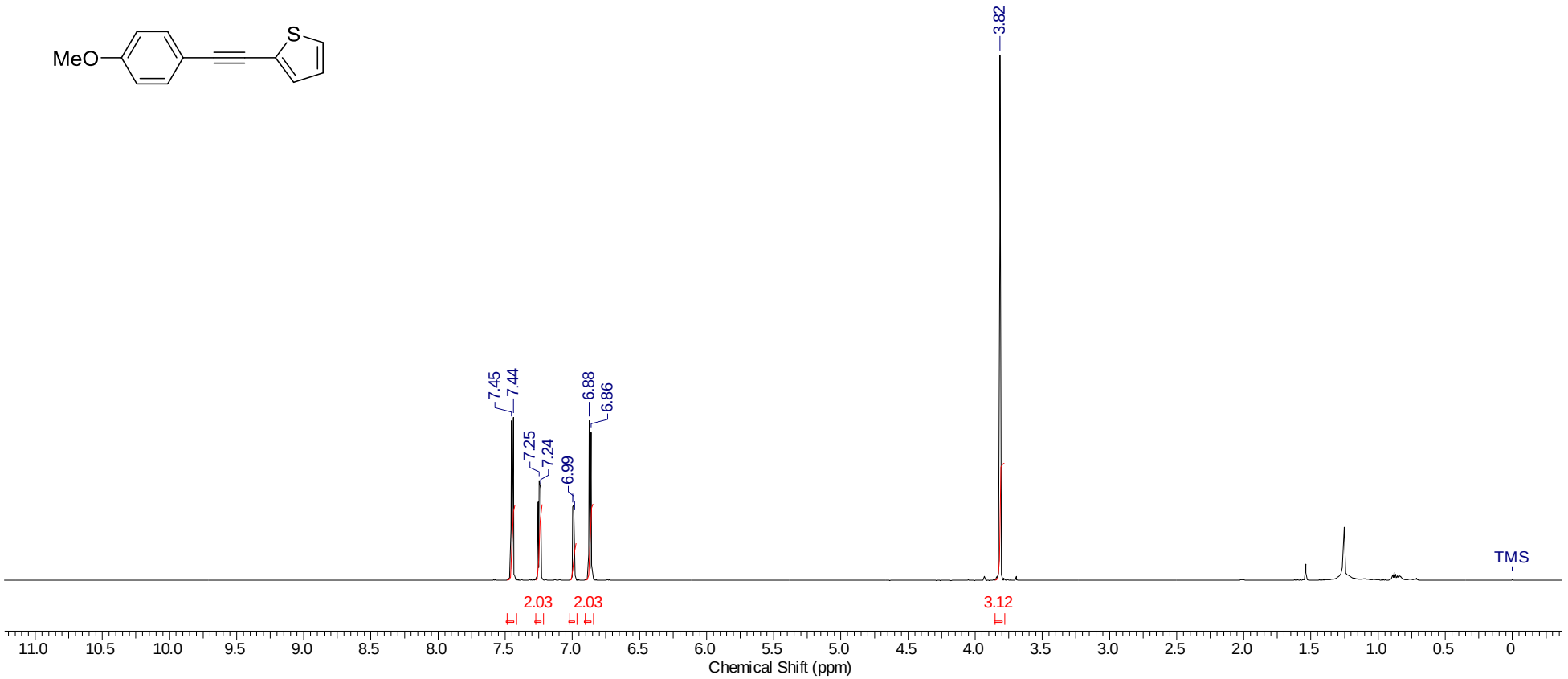
1-[(4-Methoxyphenyl)ethynyl]-3,5-dimethylbenzene (3h)

^{1}H NMR (400 MHz, $CDCl_3$)



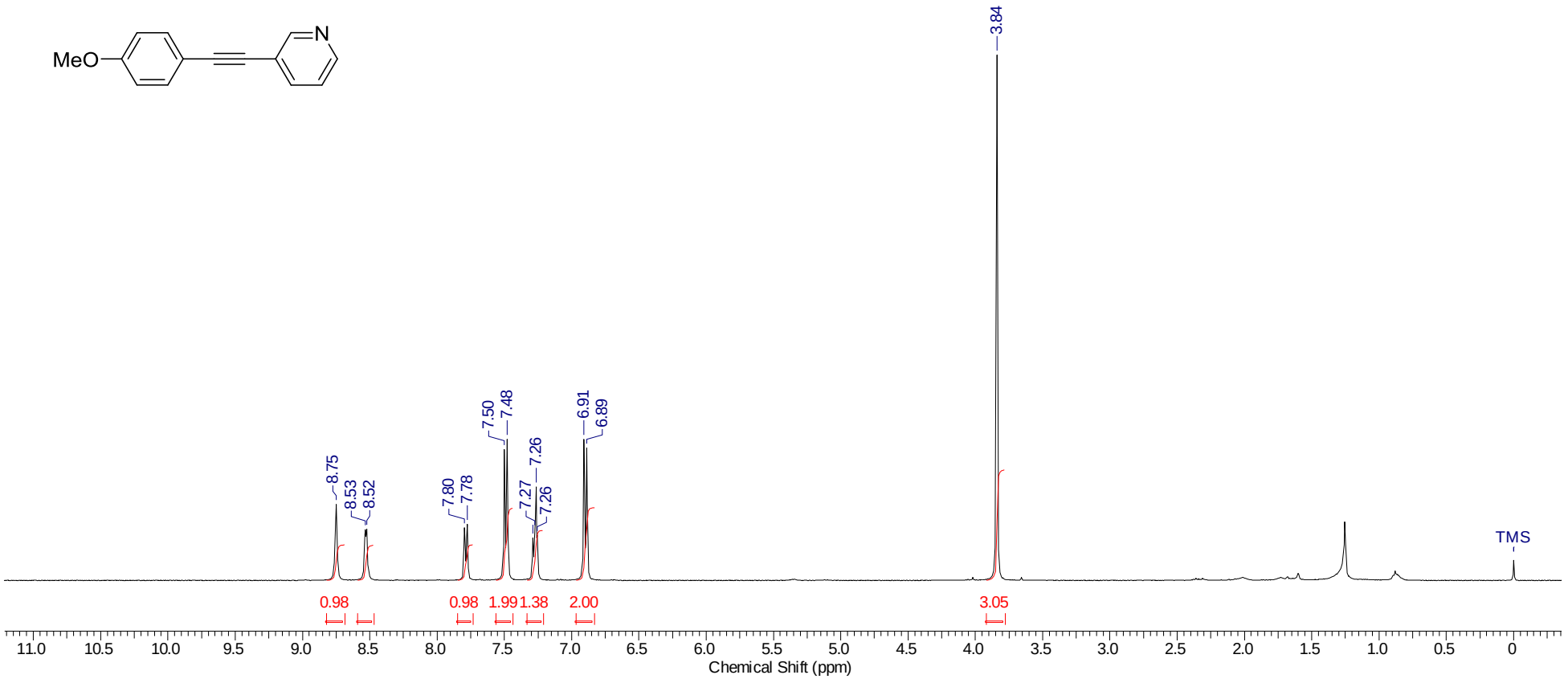
2-[(4-Methoxyphenyl)ethynyl]thiophene (3i)

^{1}H NMR (600 MHz, $CDCl_3$)



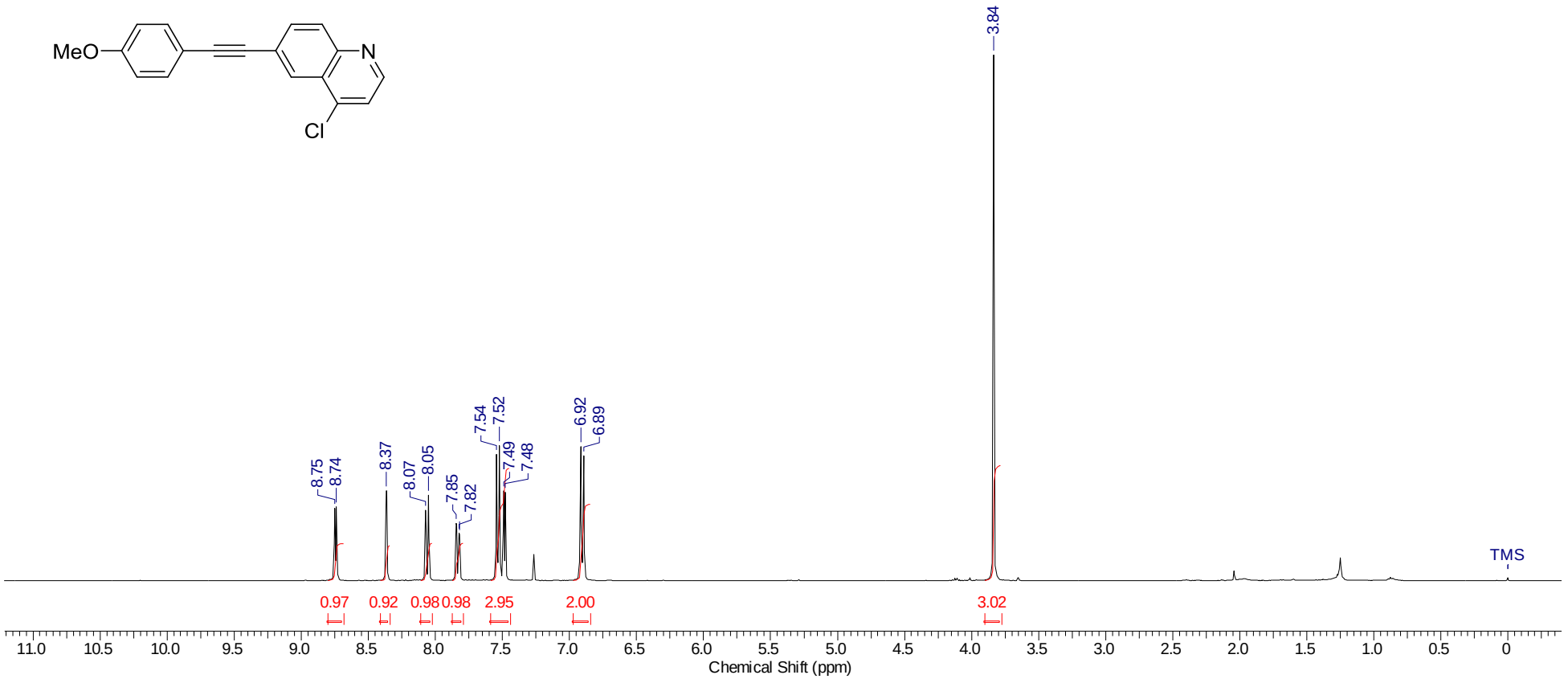
3-[(4-Methoxyphenyl)ethynyl]pyridine (3j)

^{1}H NMR (400 MHz, $CDCl_3$)



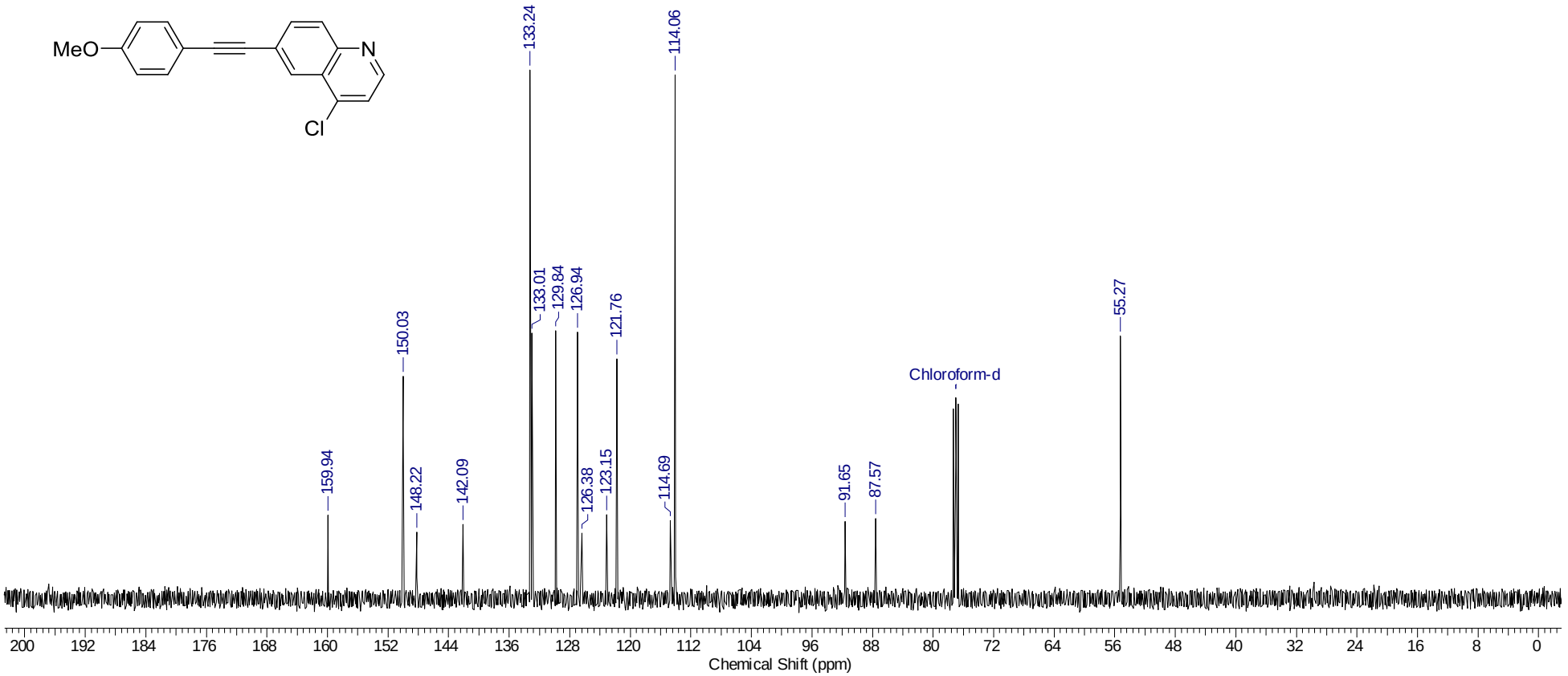
4-Chloro-6-[(4-methoxyphenyl)ethynyl]quinoline (3k)

^{1}H NMR (400 MHz, $CDCl_3$)



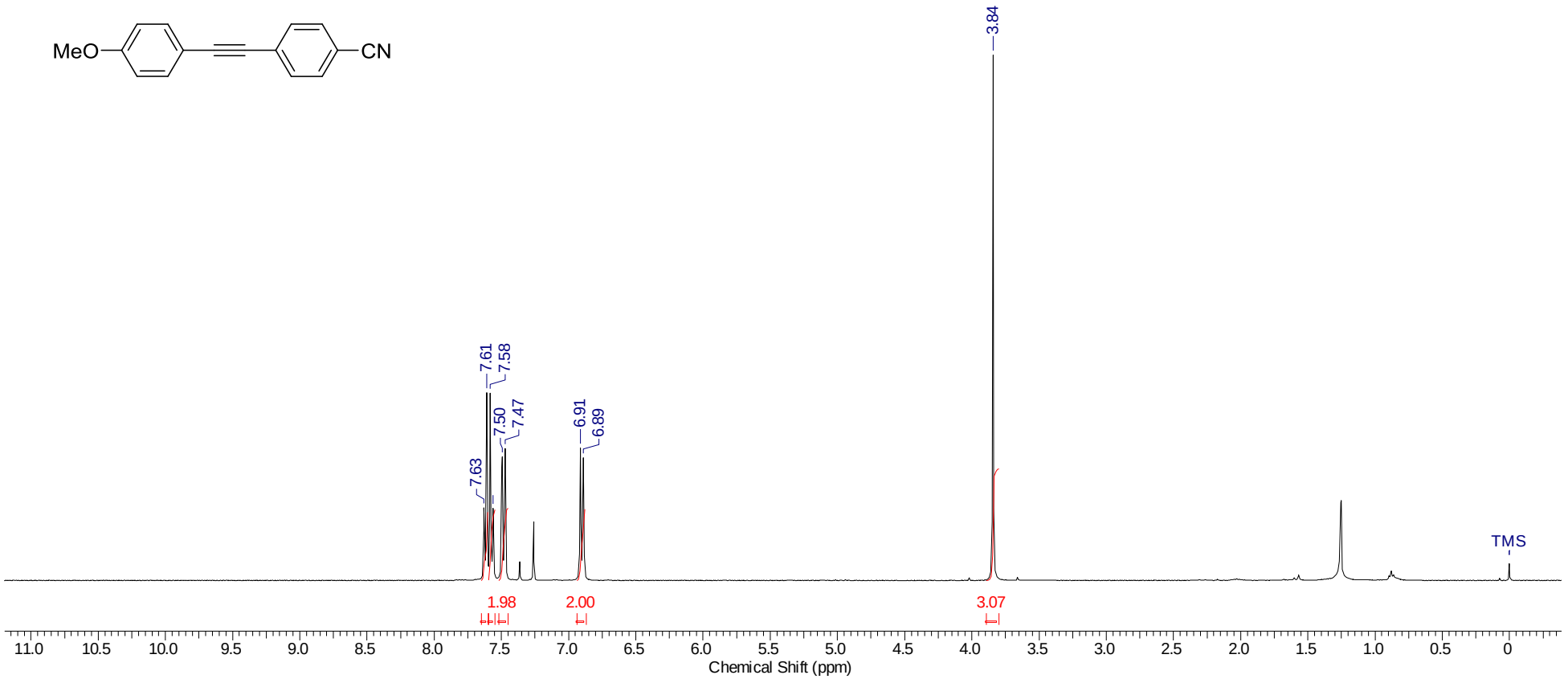
4-Chloro-6-[(4-methoxyphenyl)ethynyl]quinoline (3k)

^{13}C NMR (100 MHz, $CDCl_3$)



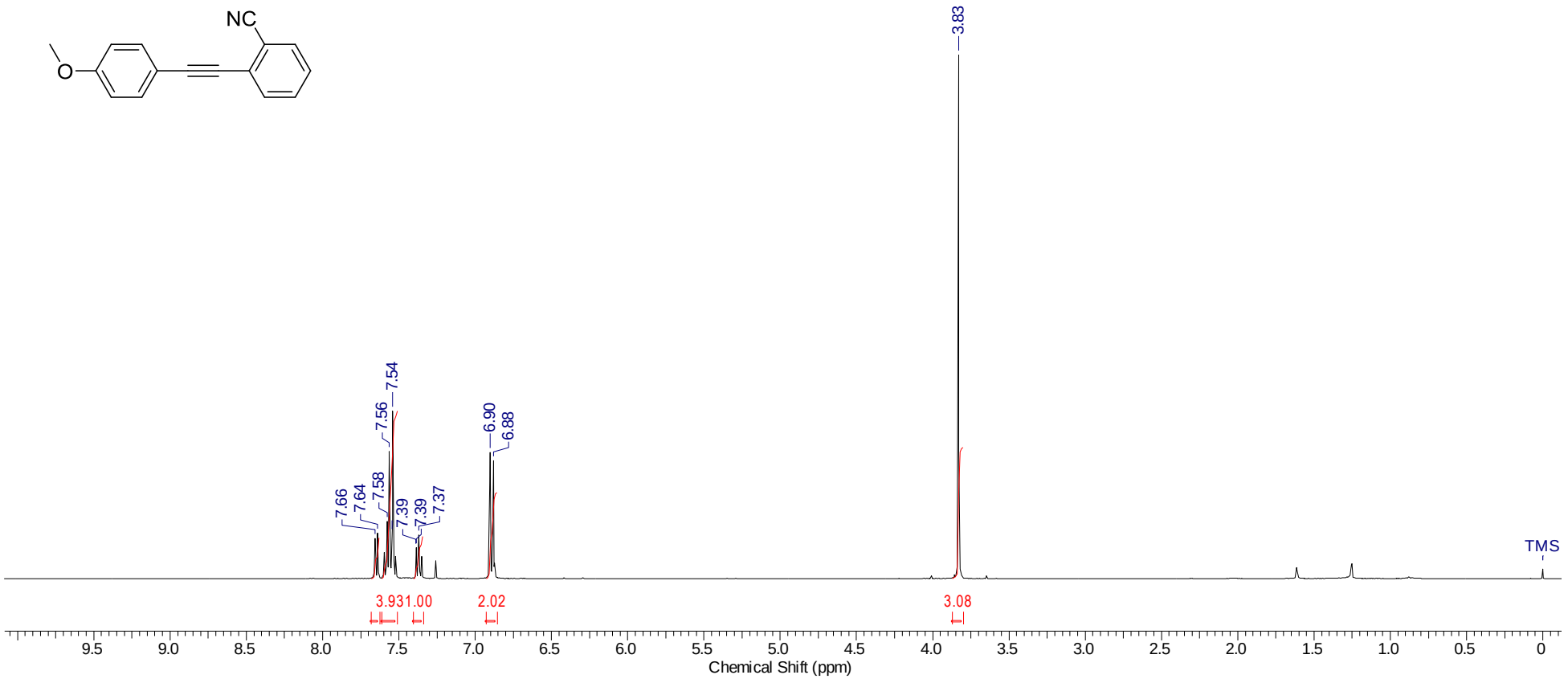
4-[(4-Methoxyphenyl)ethynyl]benzonitrile (3l)

^{1}H NMR (400 MHz, $CDCl_3$)



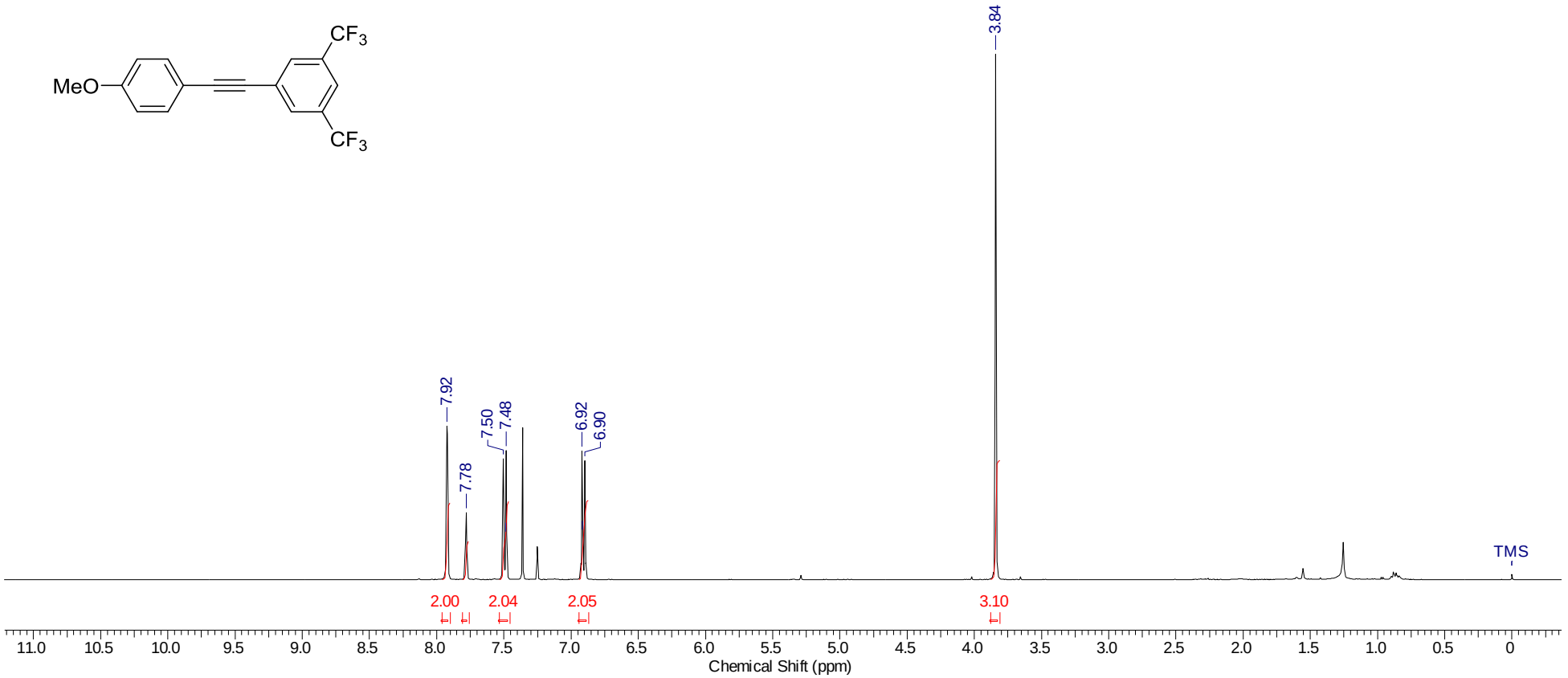
2-[(4-Methoxyphenyl)ethynyl]benzonitrile (3m)

^{1}H NMR (400 MHz, $CDCl_3$)



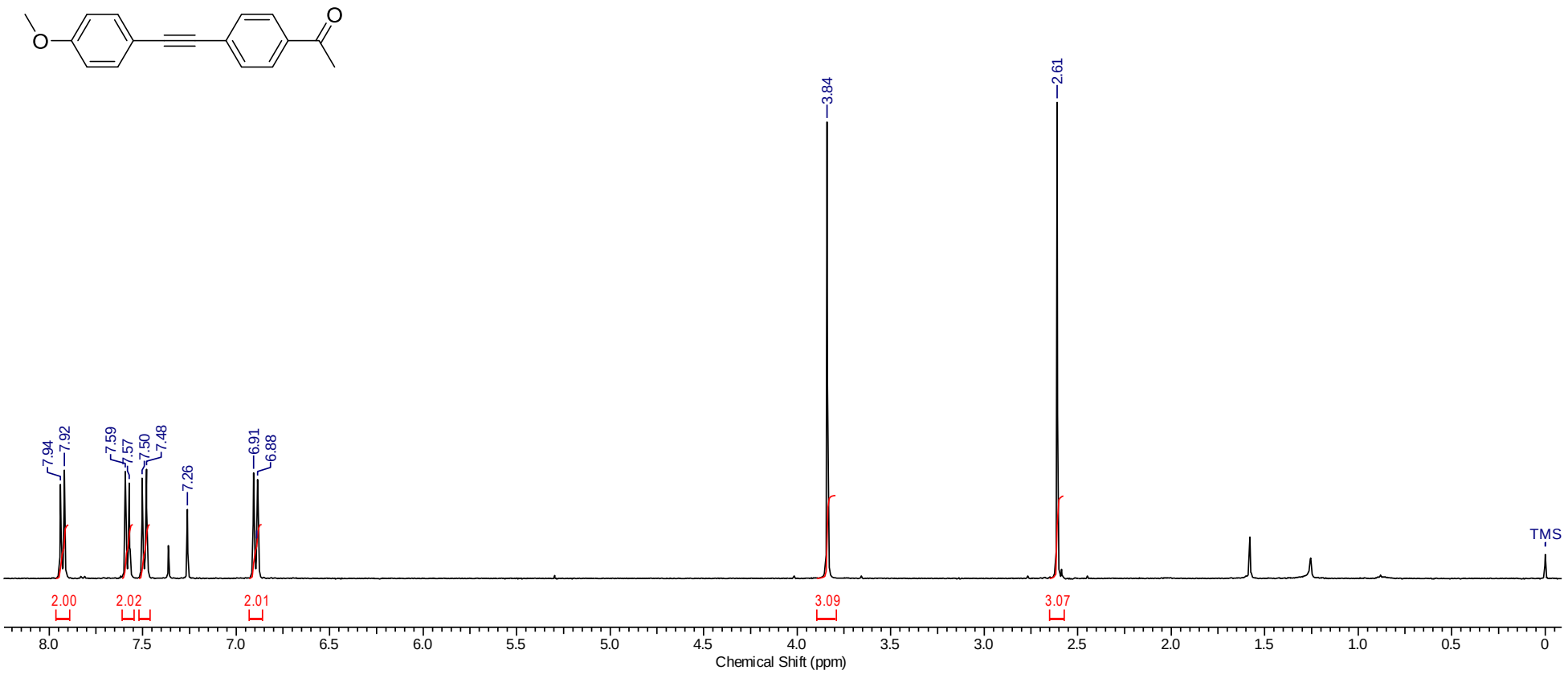
1-[(4-Methoxyphenyl)ethynyl]-3,5-bis(trifluoromethyl)benzene (3n)

^{1}H NMR (400 MHz, $CDCl_3$)



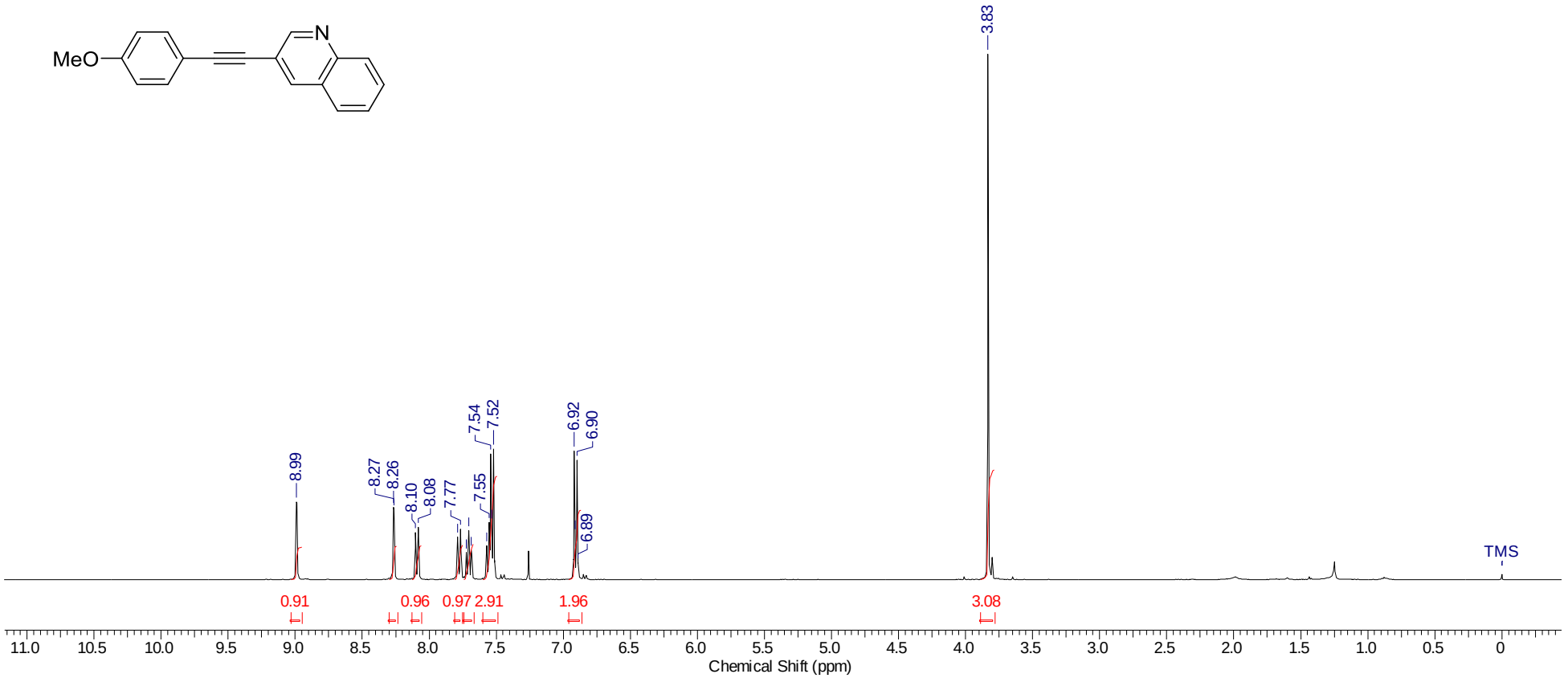
1-{4-[(4-Methoxyphenyl)ethynyl]phenyl}ethanone (3o)

^{1}H NMR (400 MHz, $CDCl_3$)



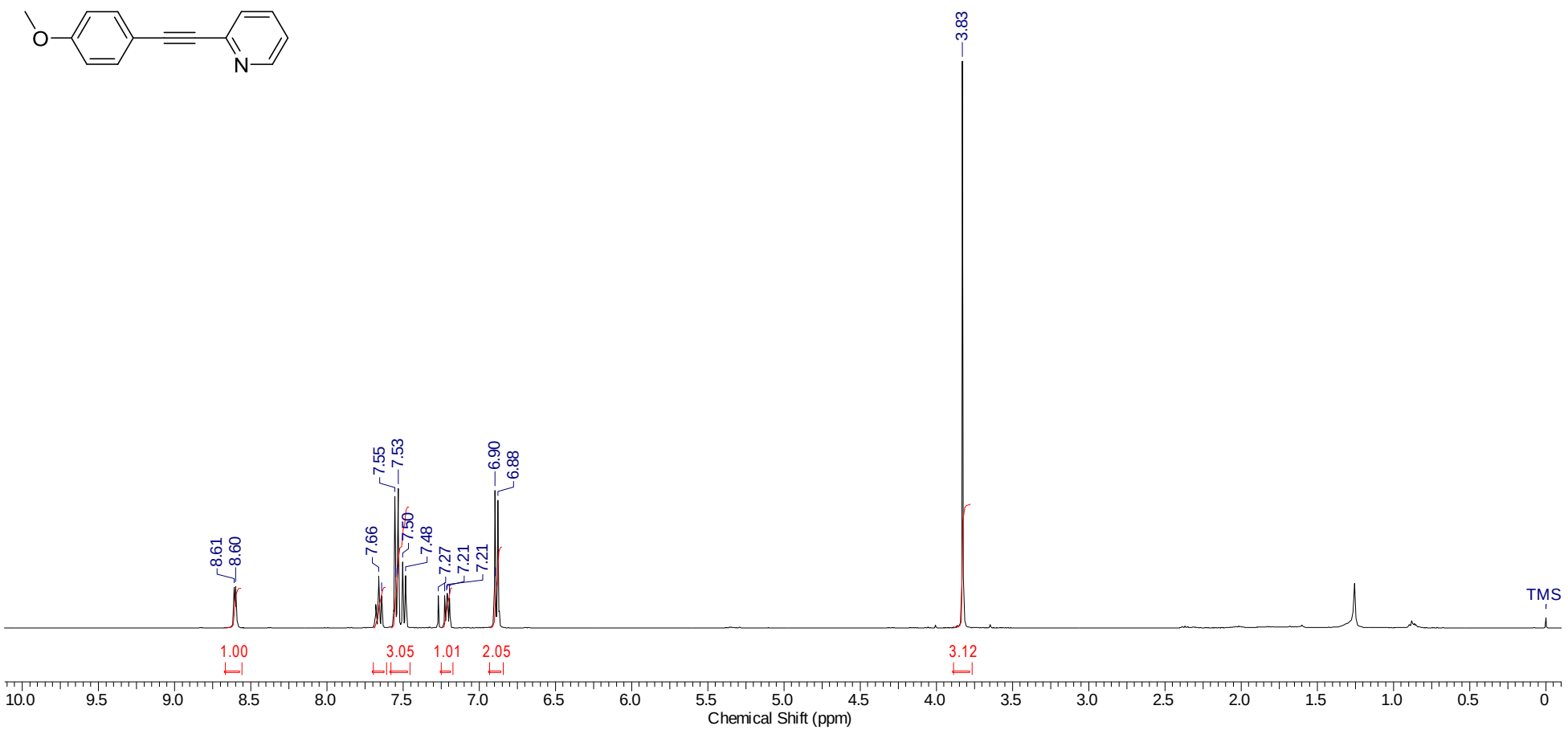
3-[(4-Methoxyphenyl)ethynyl]quinoline (3p)

^{1}H NMR (400 MHz, $CDCl_3$)



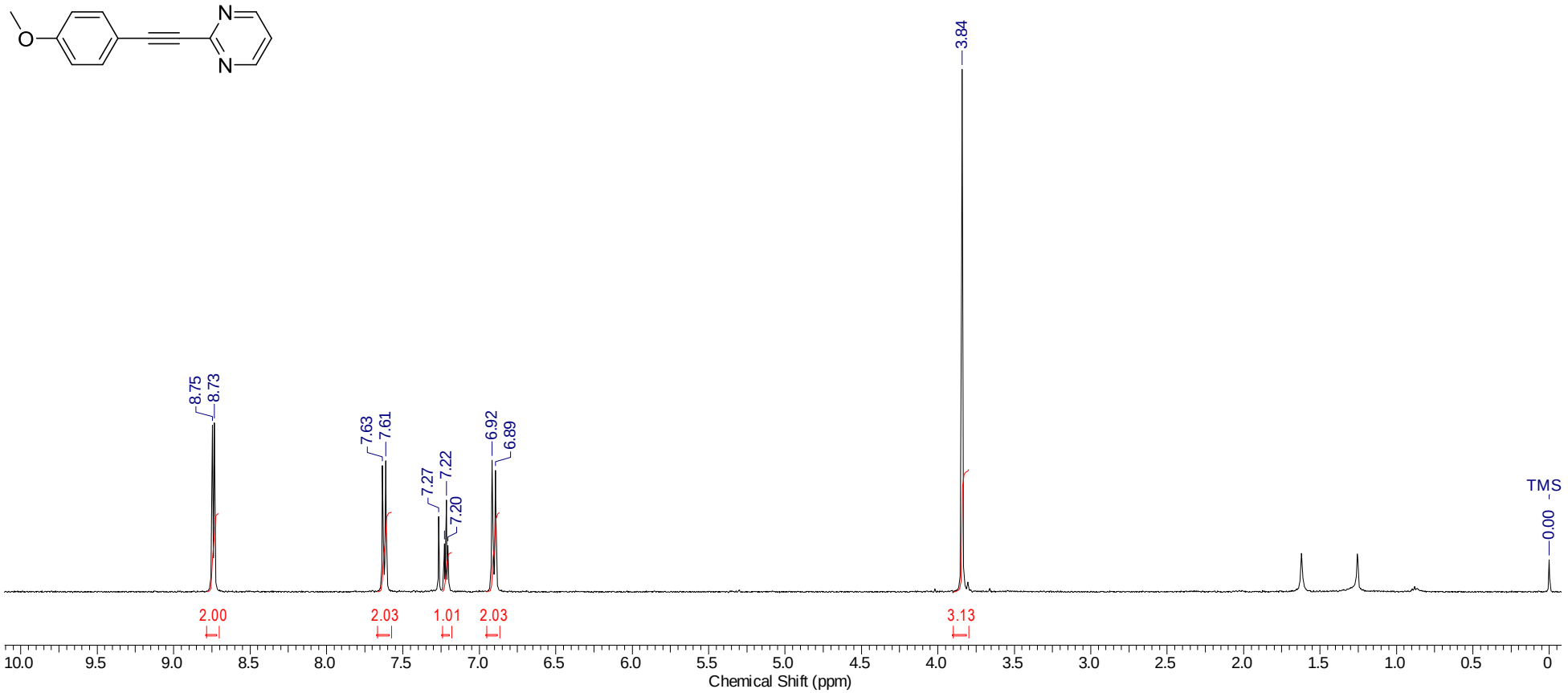
2-[(4-Methoxyphenyl)ethynyl]pyridine (3q)

^{1}H NMR (400 MHz, $CDCl_3$)



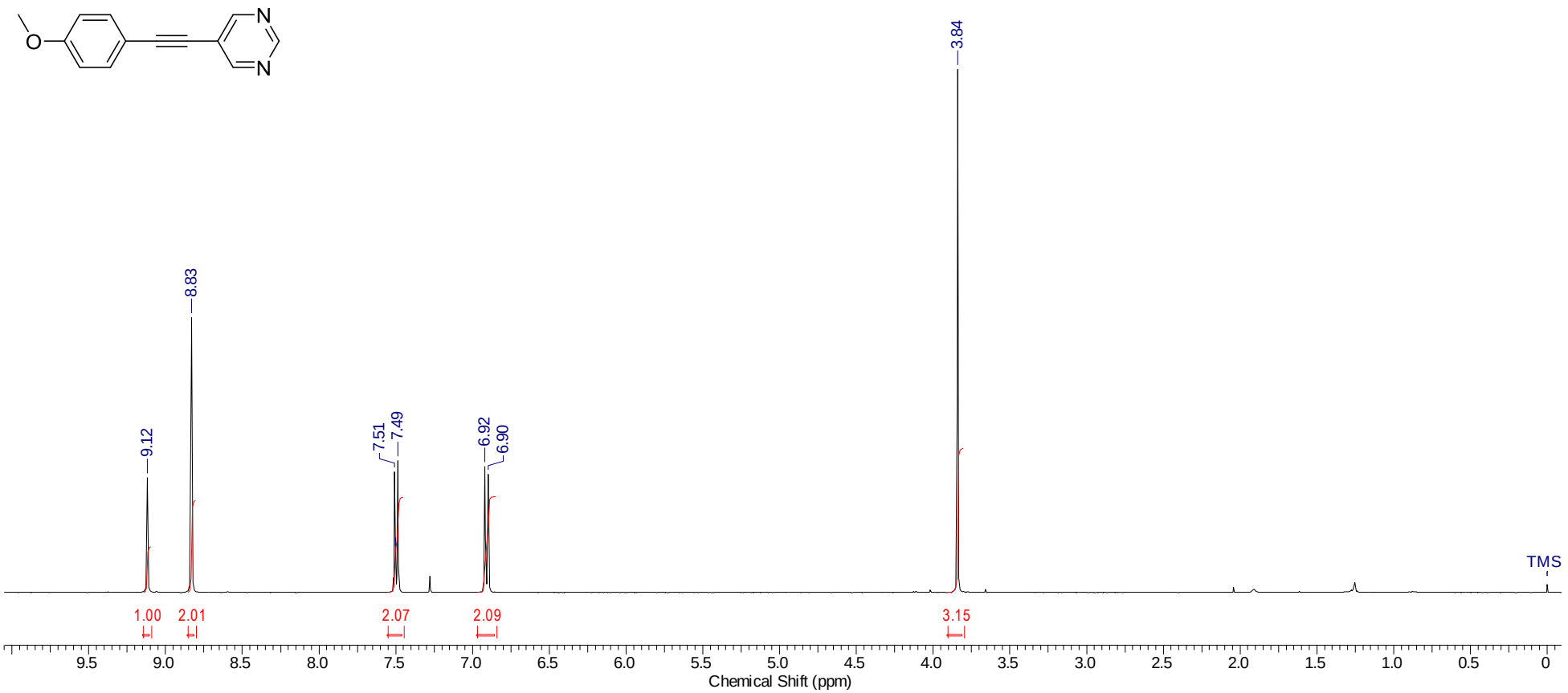
2-[(4-Methoxyphenyl)ethynyl]pyrimidine (3r)

^{1}H NMR (400 MHz, $CDCl_3$)



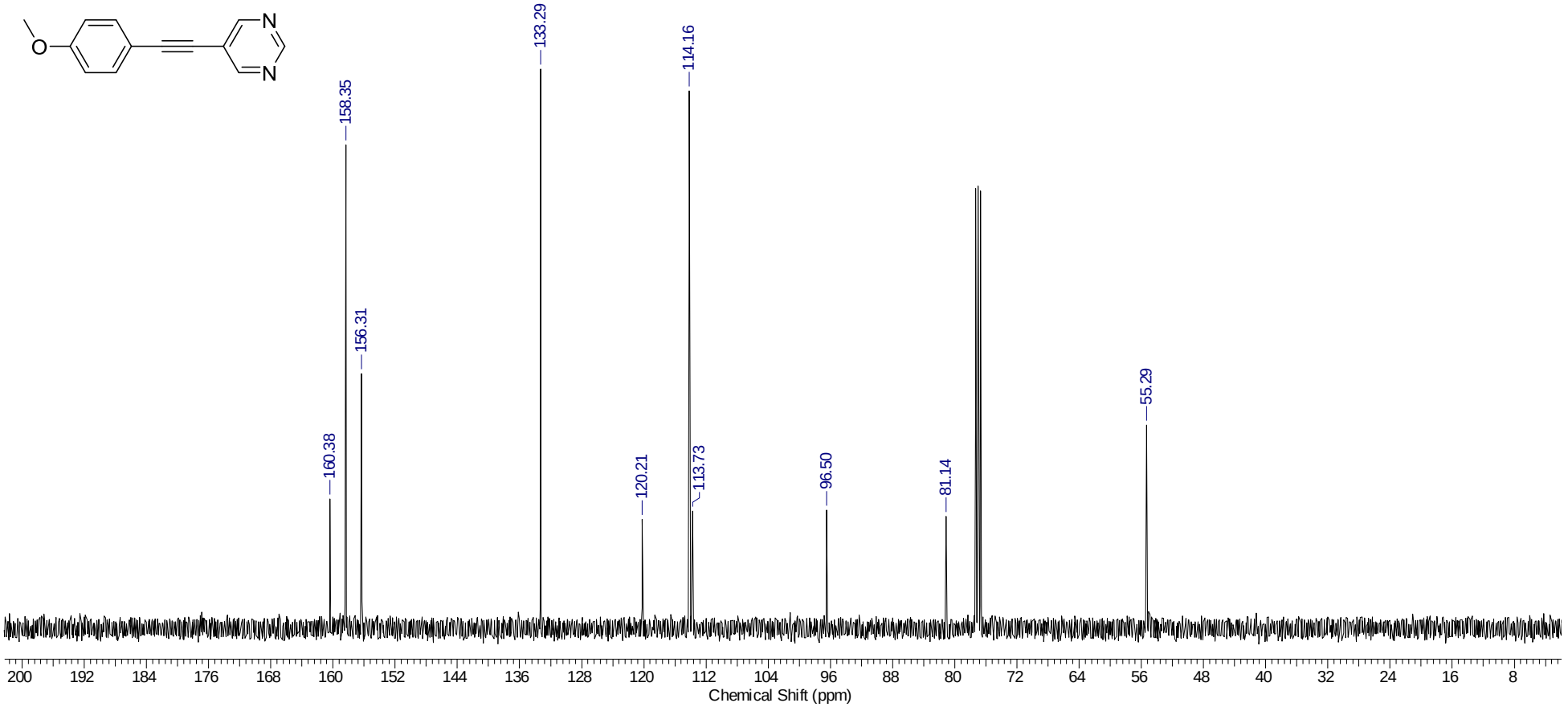
5-[(4-Methoxyphenyl)ethynyl]pyrimidine (3s)

^{1}H NMR (400 MHz, $CDCl_3$)



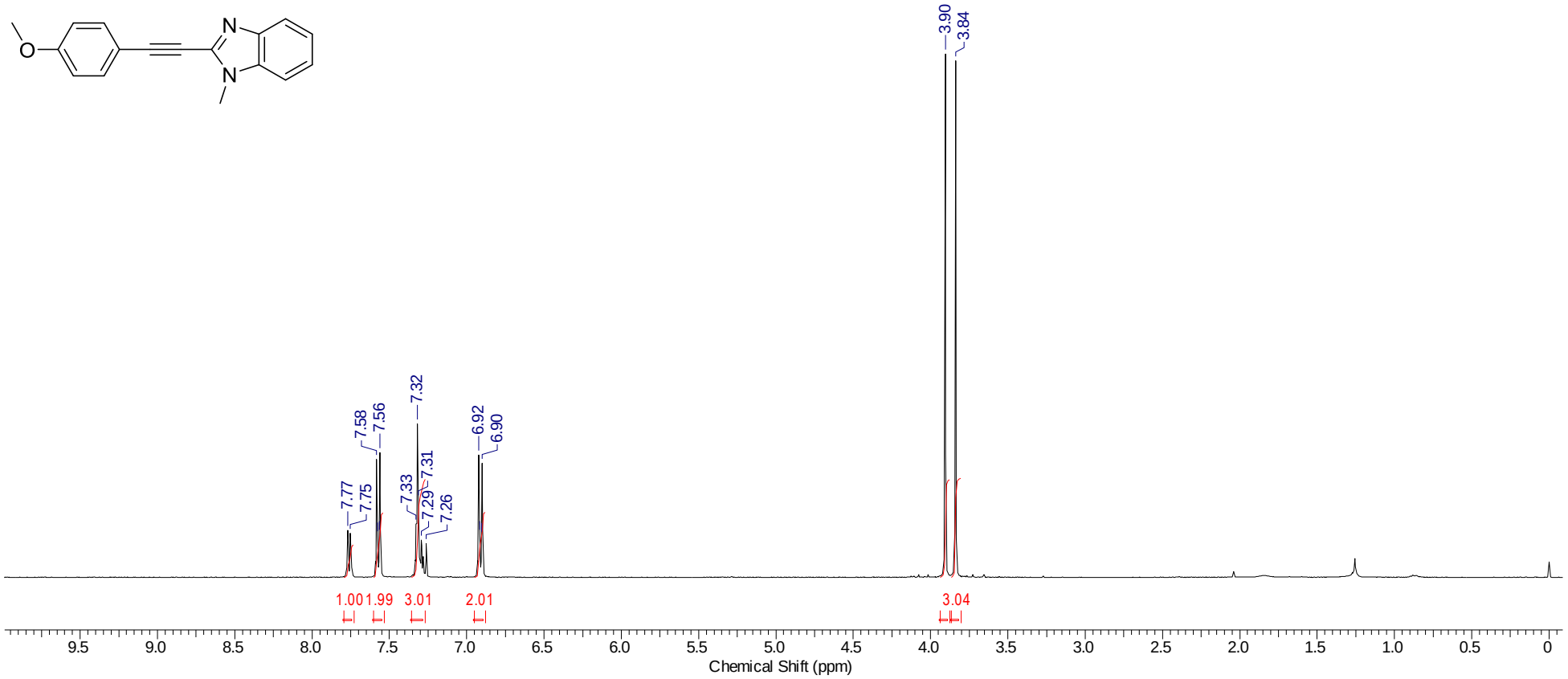
5-[(4-Methoxyphenyl)ethynyl]pyrimidine (3s)

^{13}C NMR (100 MHz, $CDCl_3$)



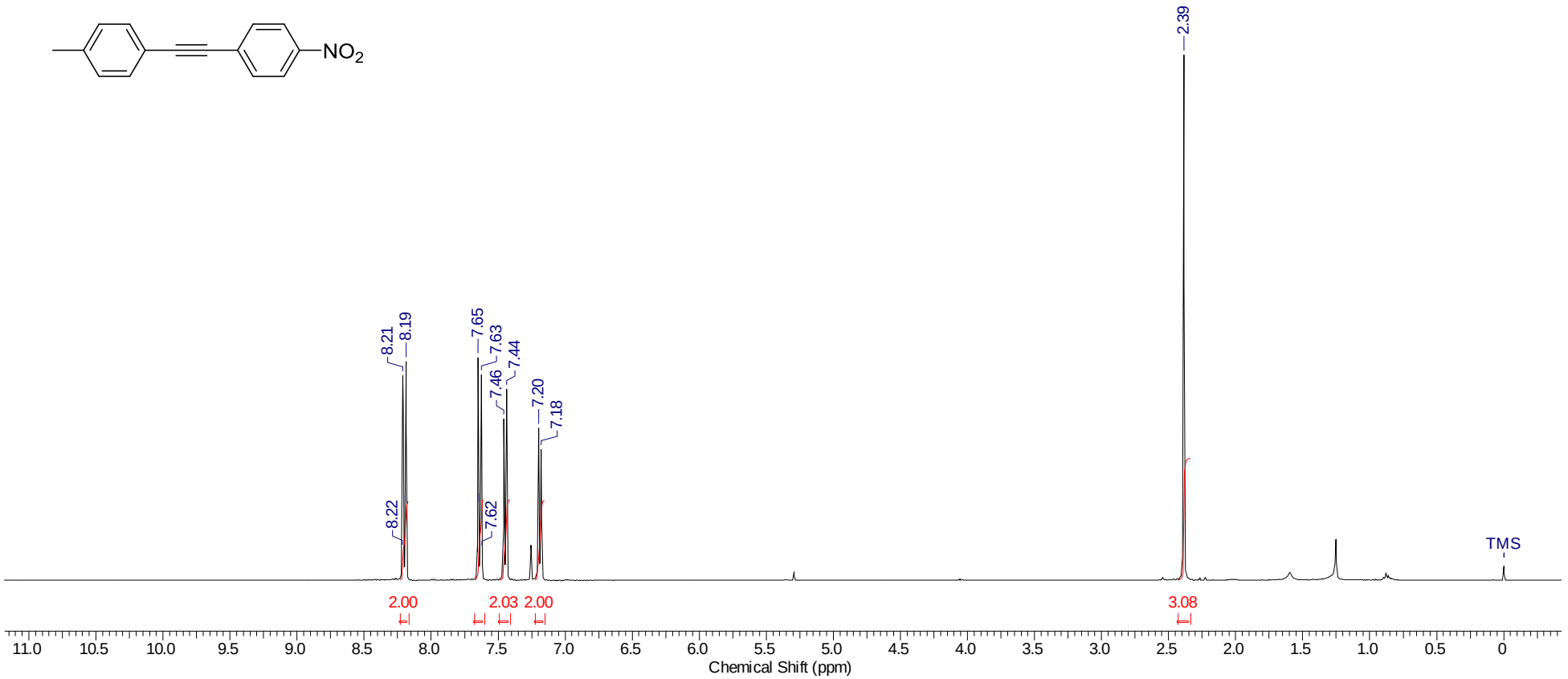
2-[(4-Methoxyphenyl)ethynyl]-1-methyl-1*H*-benzimidazole (3t)

^{1}H NMR (400 MHz, $CDCl_3$)



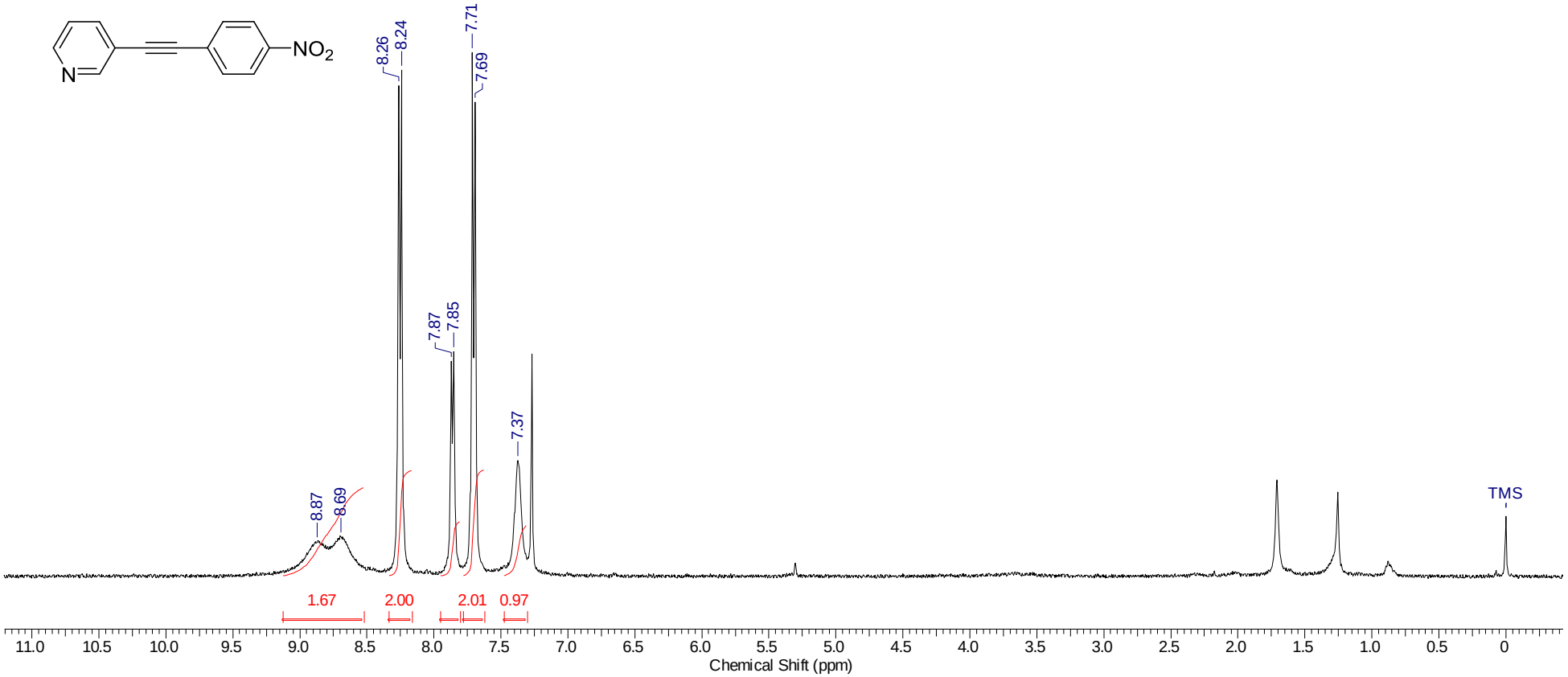
1-Methyl-4-[(4-nitrophenyl)ethynyl]benzene (3u)

^{1}H NMR (400 MHz, $CDCl_3$)



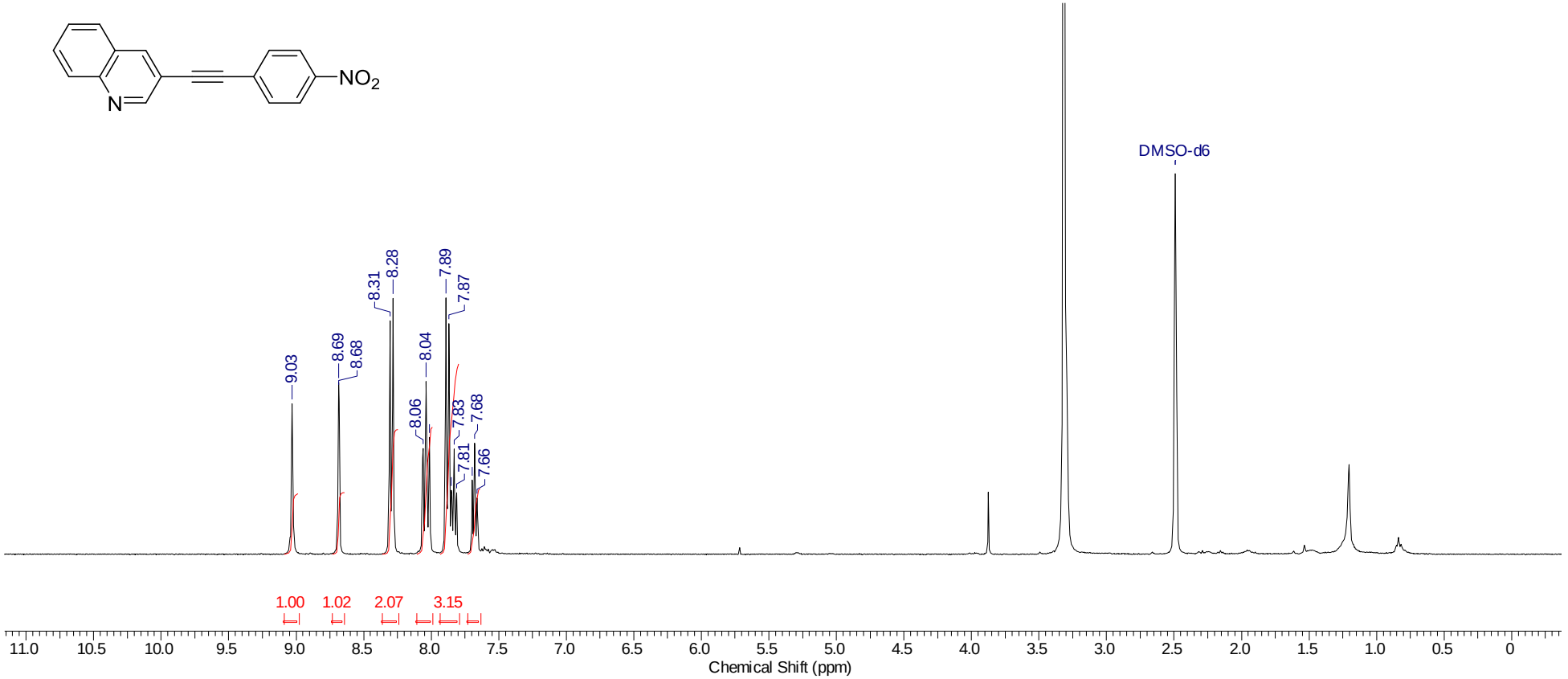
3-[(4-Nitrophenyl)ethynyl]pyridine (3v)

^{1}H NMR (400 MHz, $CDCl_3$)



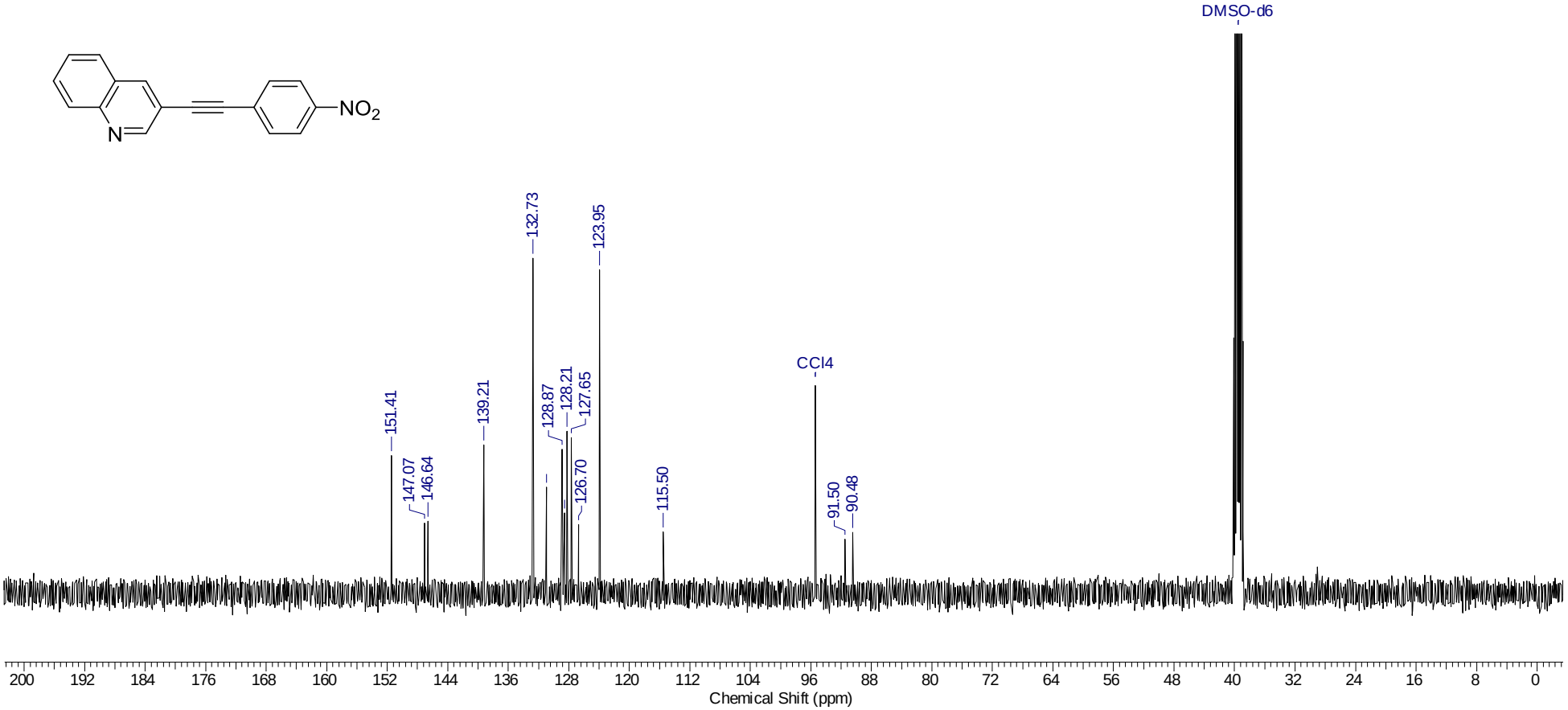
3-[(4-Nitrophenyl)ethynyl]quinoline (3w)

^{1}H NMR (400 MHz, DMSO-d_6–CCl_4)



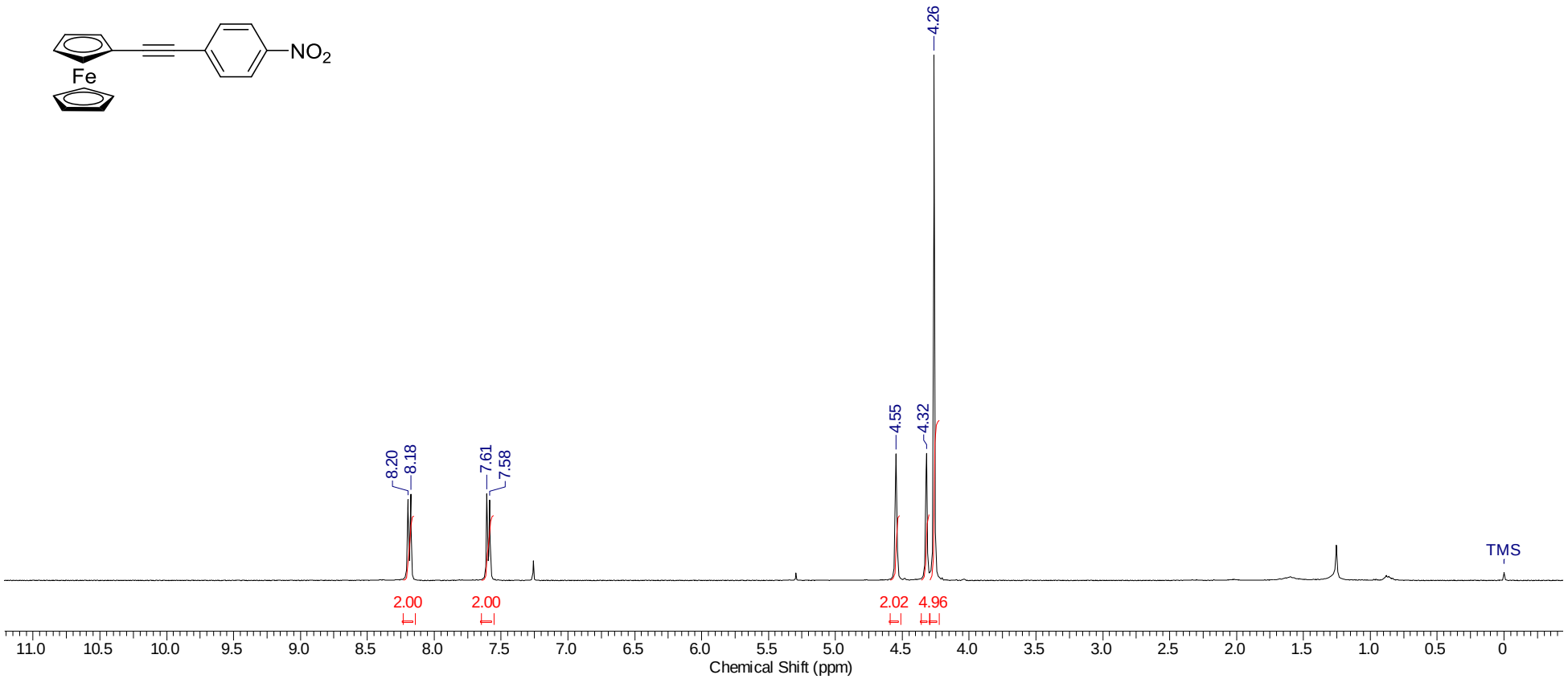
3-[(4-Nitrophenyl)ethynyl]quinoline (3w)

^{13}C NMR (100 MHz, DMSO-d_6–CCl_4)



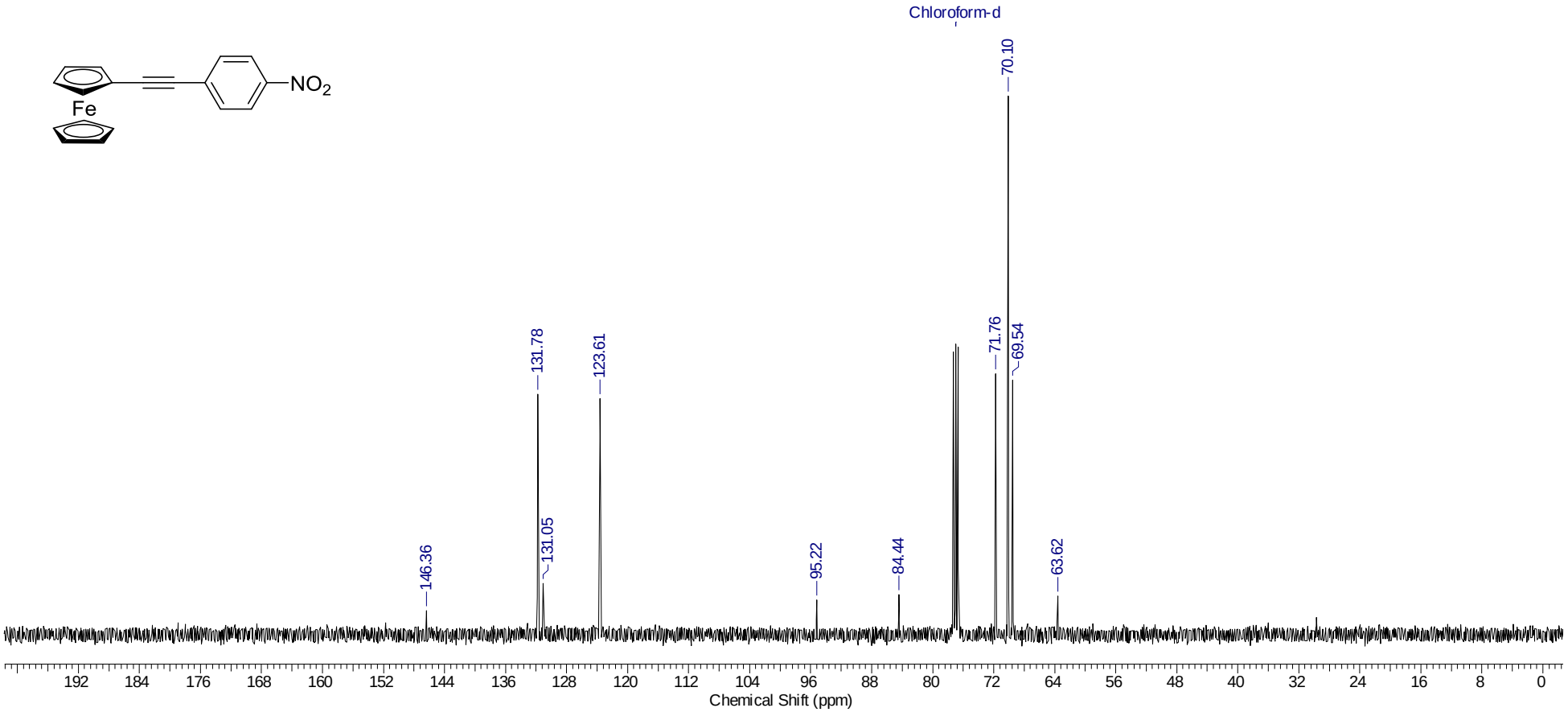
[(4-Nitrophenyl)ethynyl]ferrocene (3x)

^{1}H NMR (400 MHz, $CDCl_3$)



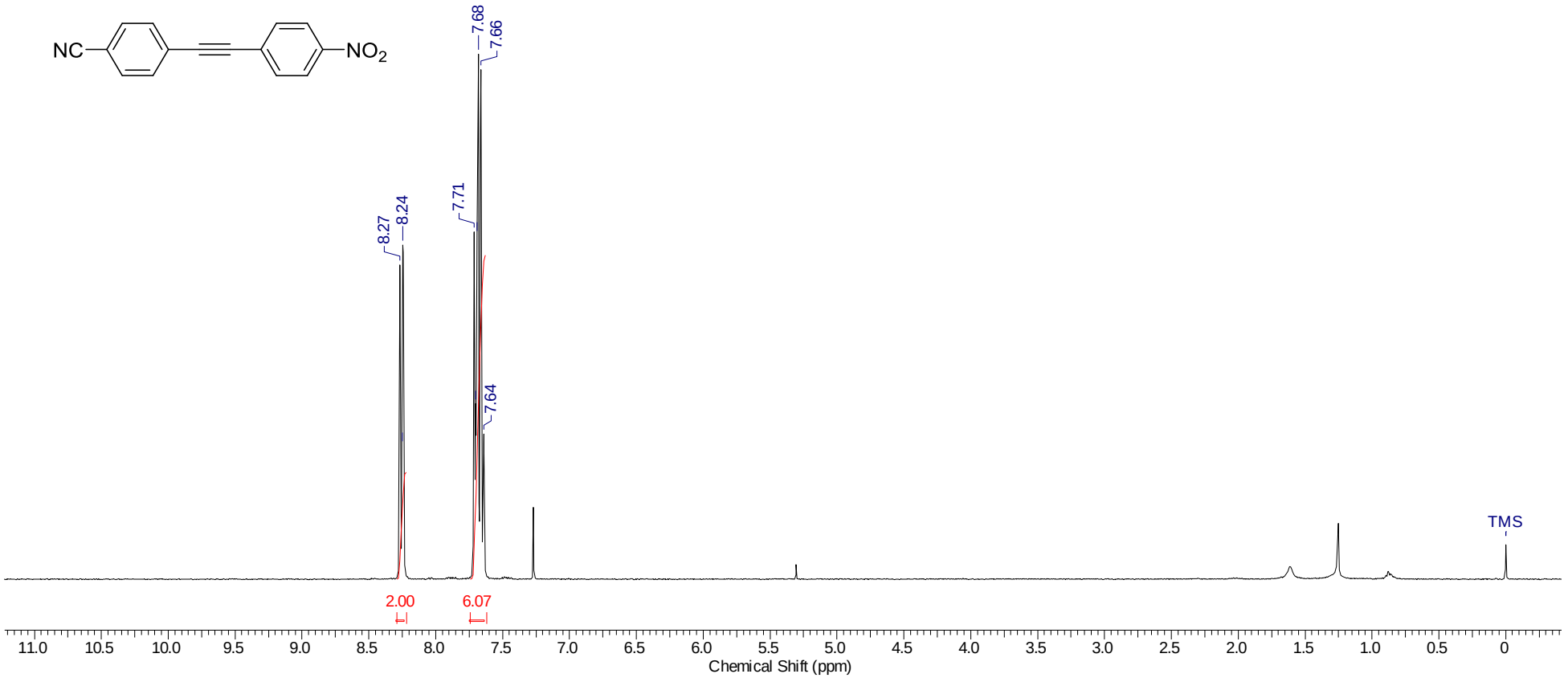
[(4-Nitrophenyl)ethynyl]ferrocene (3x)

^{13}C NMR (100 MHz, $CDCl_3$)



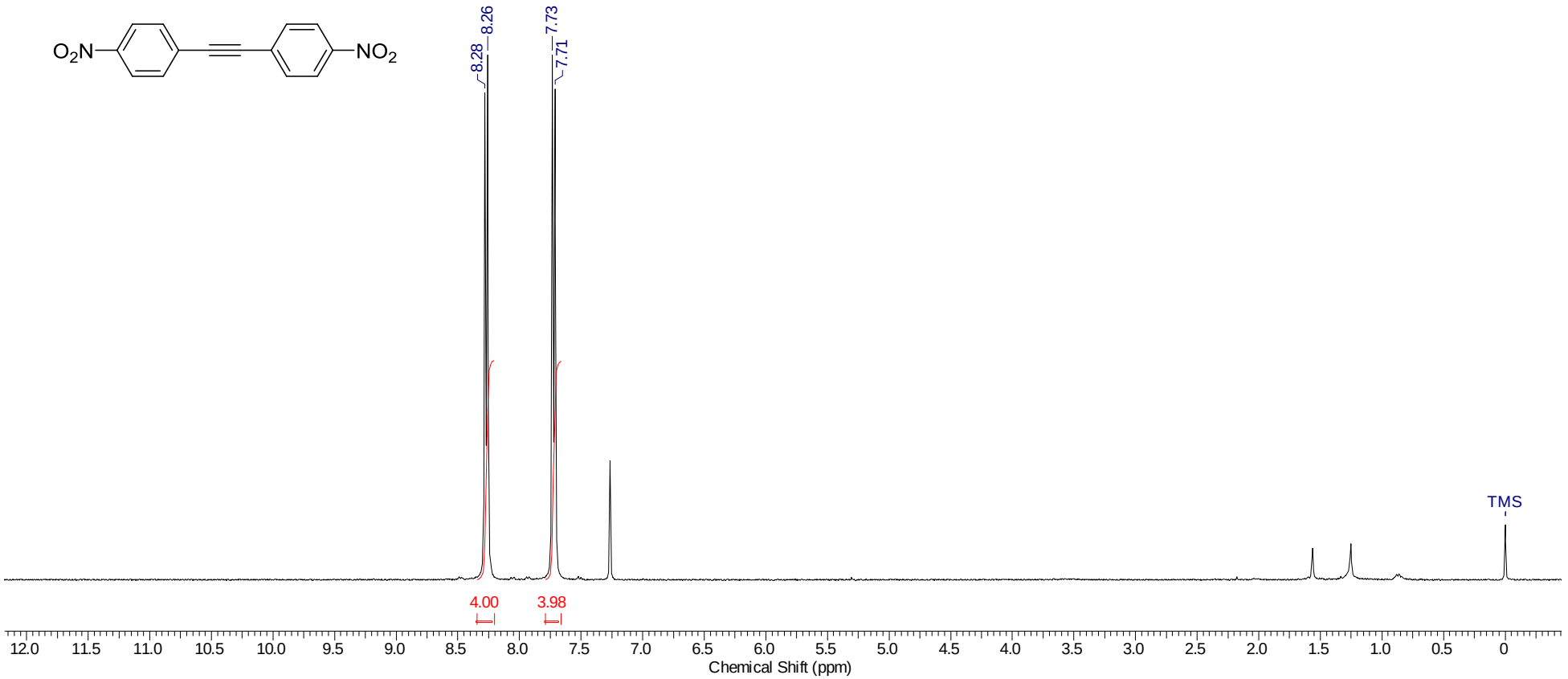
4-[(4-Nitrophenyl)ethynyl]benzonitrile (3y)

^{1}H NMR (400 MHz, $CDCl_3$)



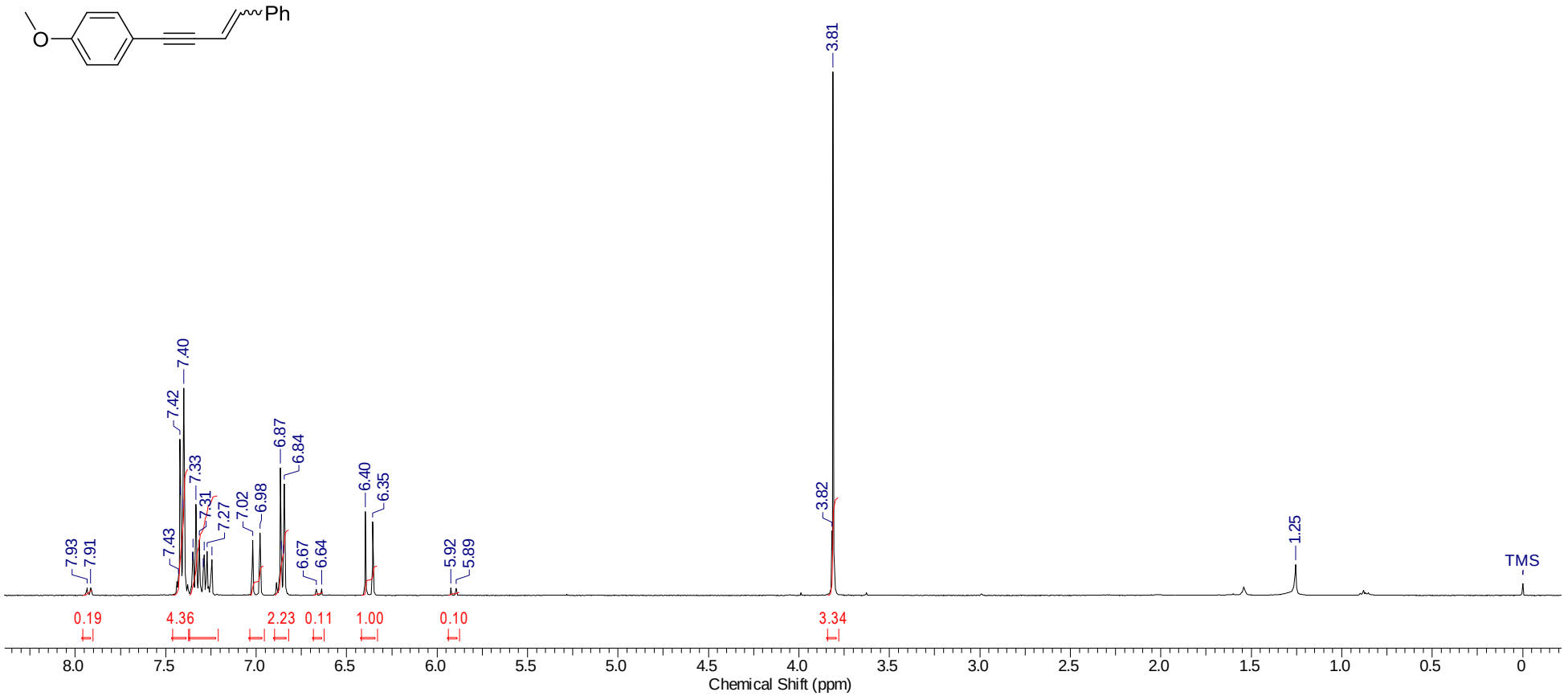
1,1'-Ethyne-1,2-diylbis(4-nitrobenzene) (3z)

^{1}H NMR (400 MHz, $CDCl_3$)



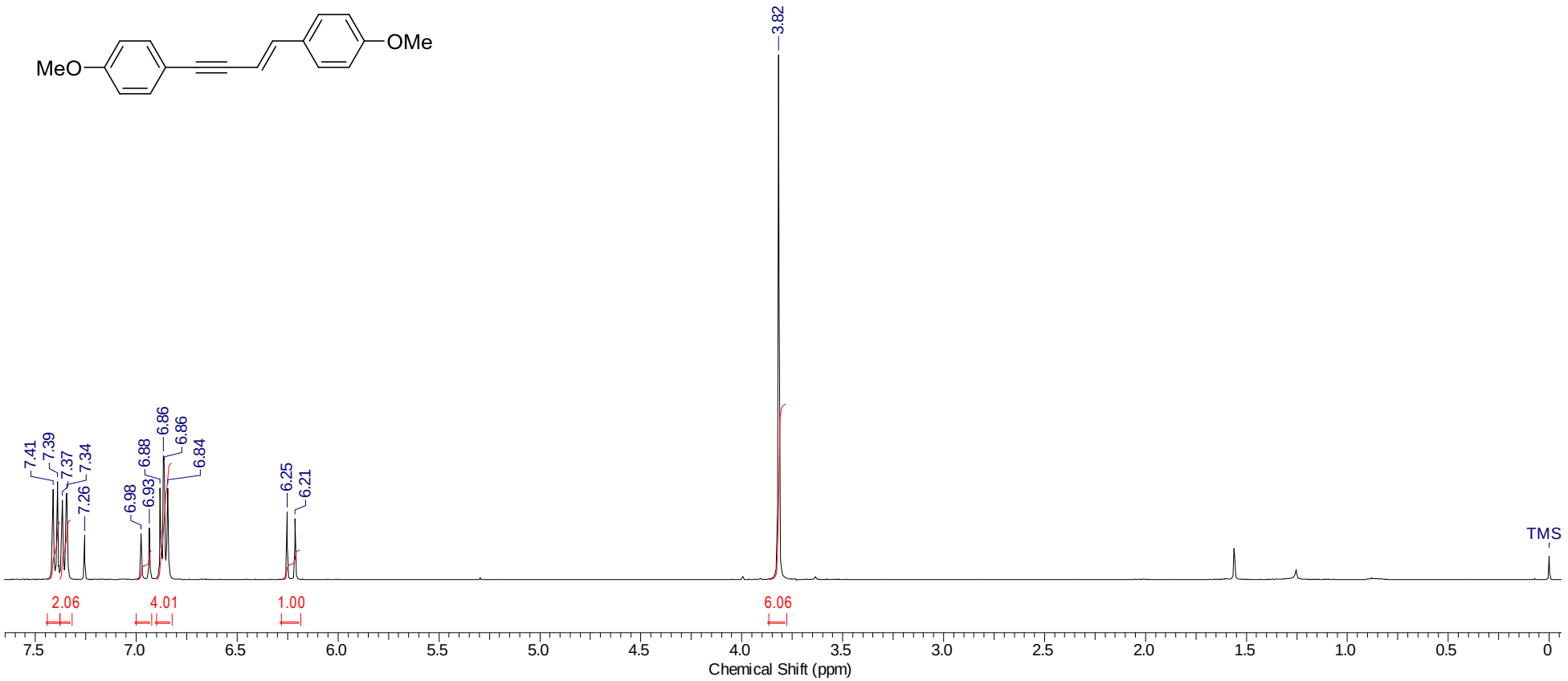
1-Methoxy-4-[4-phenylbut-3-en-1-yn-1-yl]benzene (*E*:*Z* = 10:1) (5a)

^{1}H NMR (400 MHz, $CDCl_3$)



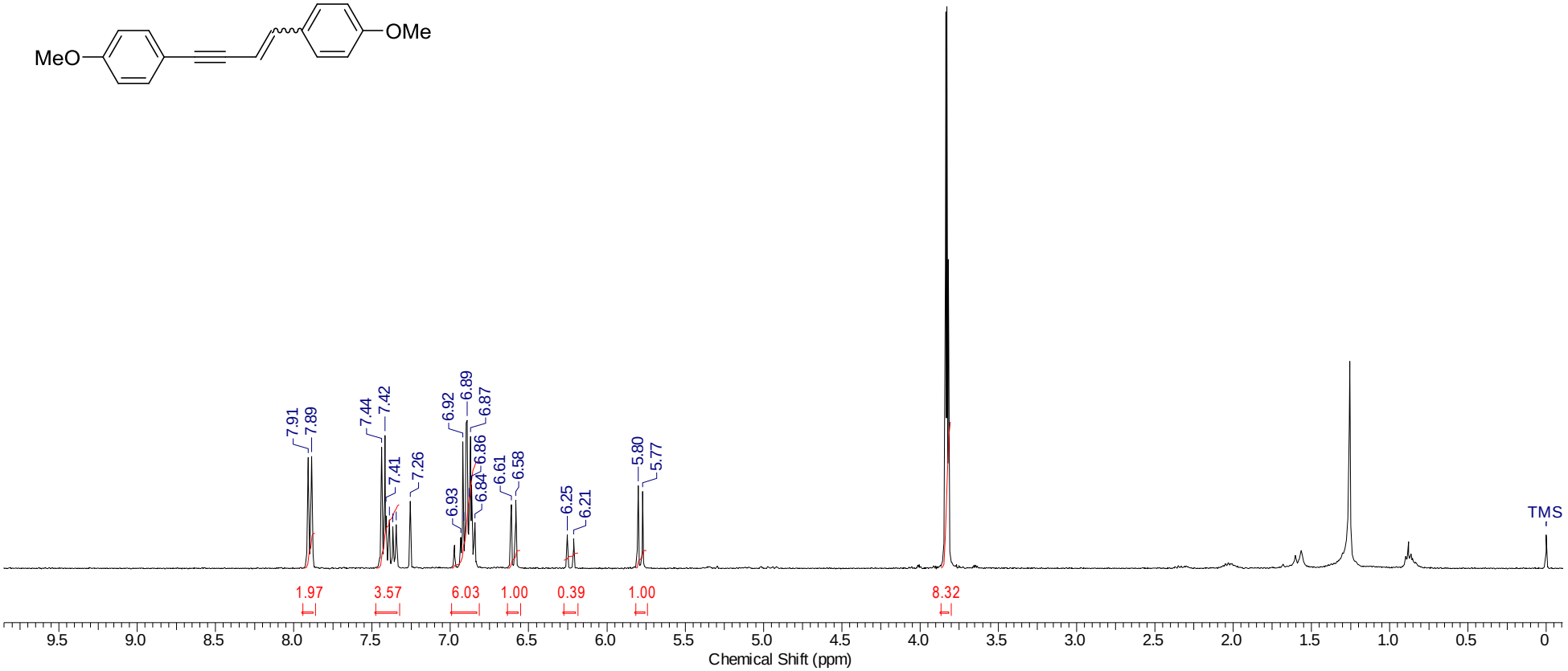
1,1'-(*1E*)-But-1-en-3-yne-1,4-diylbis(4-methoxybenzene) ((*E*)-5b)

^{1}H NMR (400 MHz, $CDCl_3$)



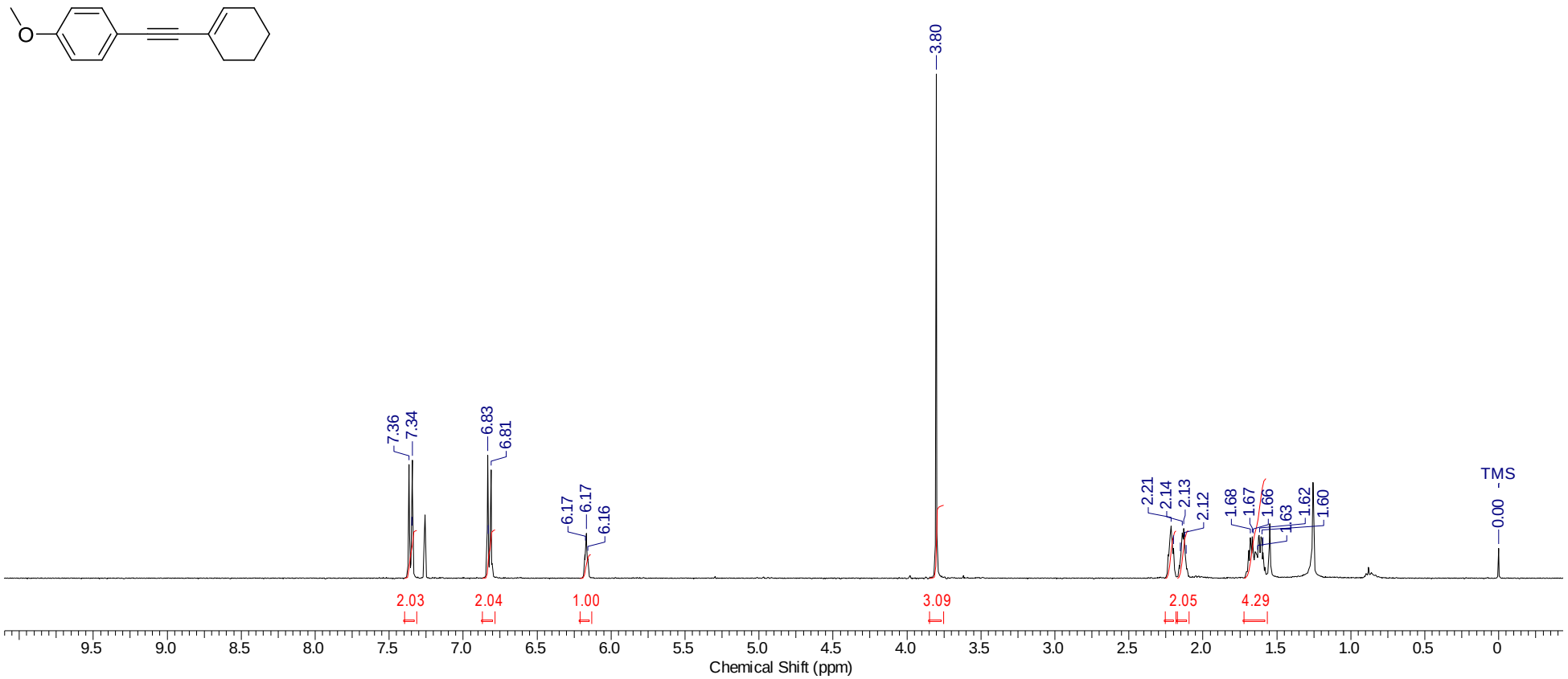
1,1'-But-1-en-3-yne-1,4-diylbis(4-methoxybenzene) *E*/*Z* = 2:5 (5b)

^{1}H NMR (400 MHz, $CDCl_3$)



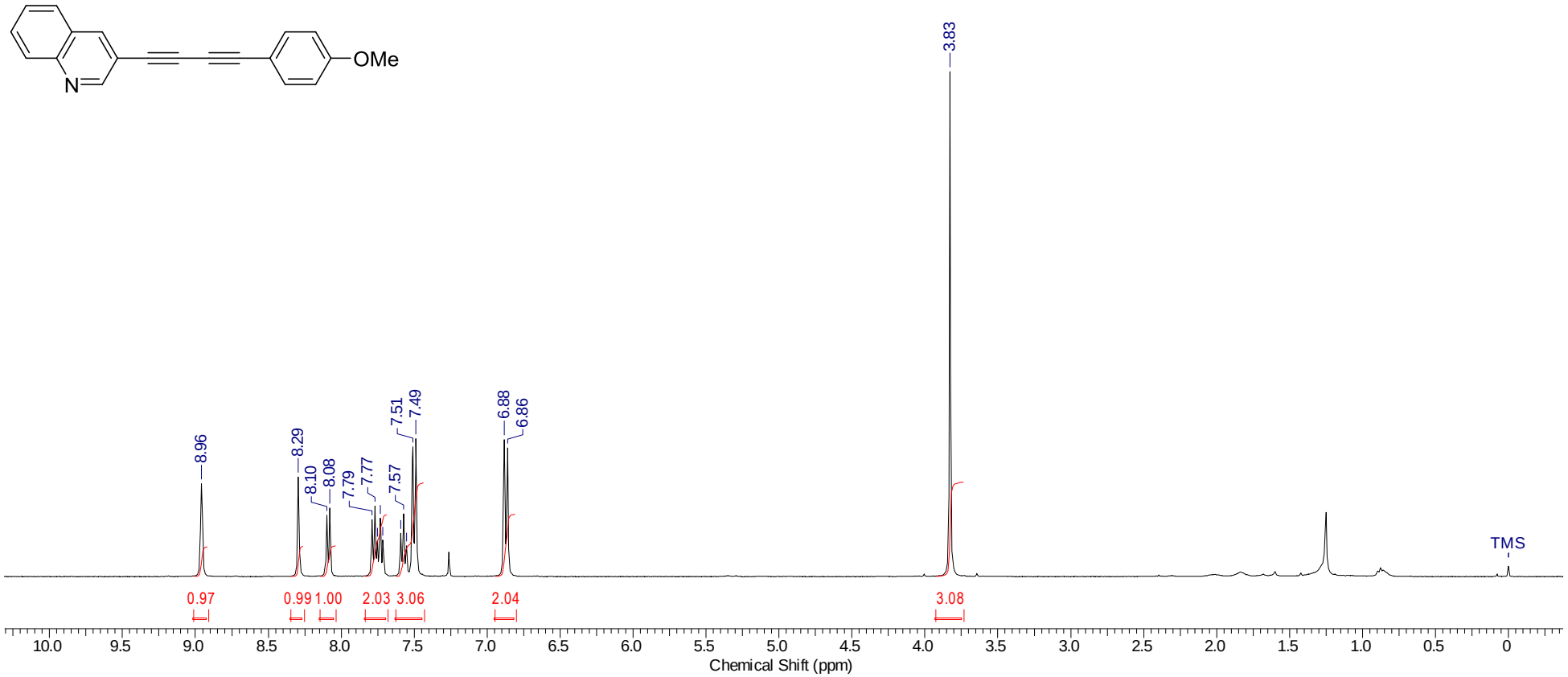
1-(Cyclohex-1-en-1-ylethynyl)-4-methoxybenzene (5c)

^{1}H NMR (400 MHz, $CDCl_3$)



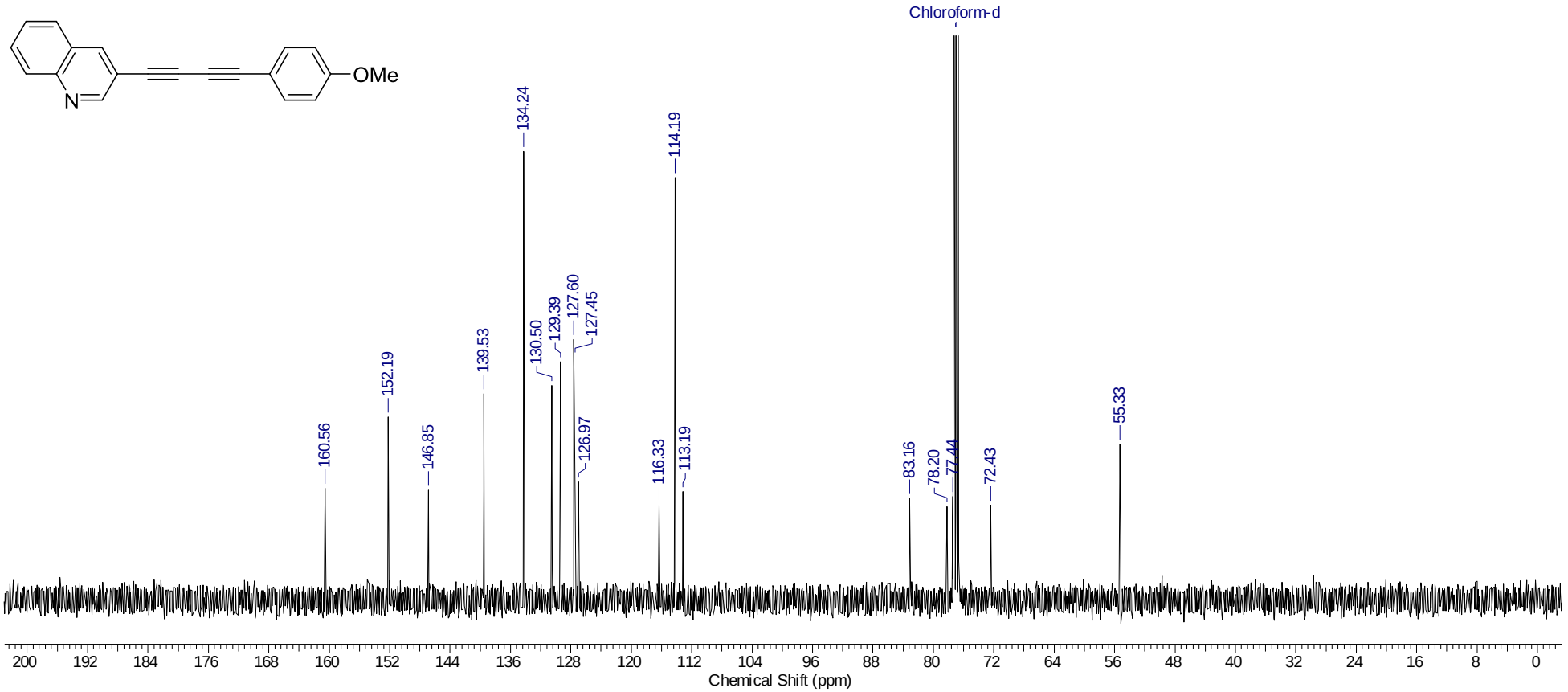
3-[4-(4-Methoxyphenyl)buta-1,3-diyn-1-yl]quinoline (5d)

^{1}H NMR (400 MHz, $CDCl_3$)



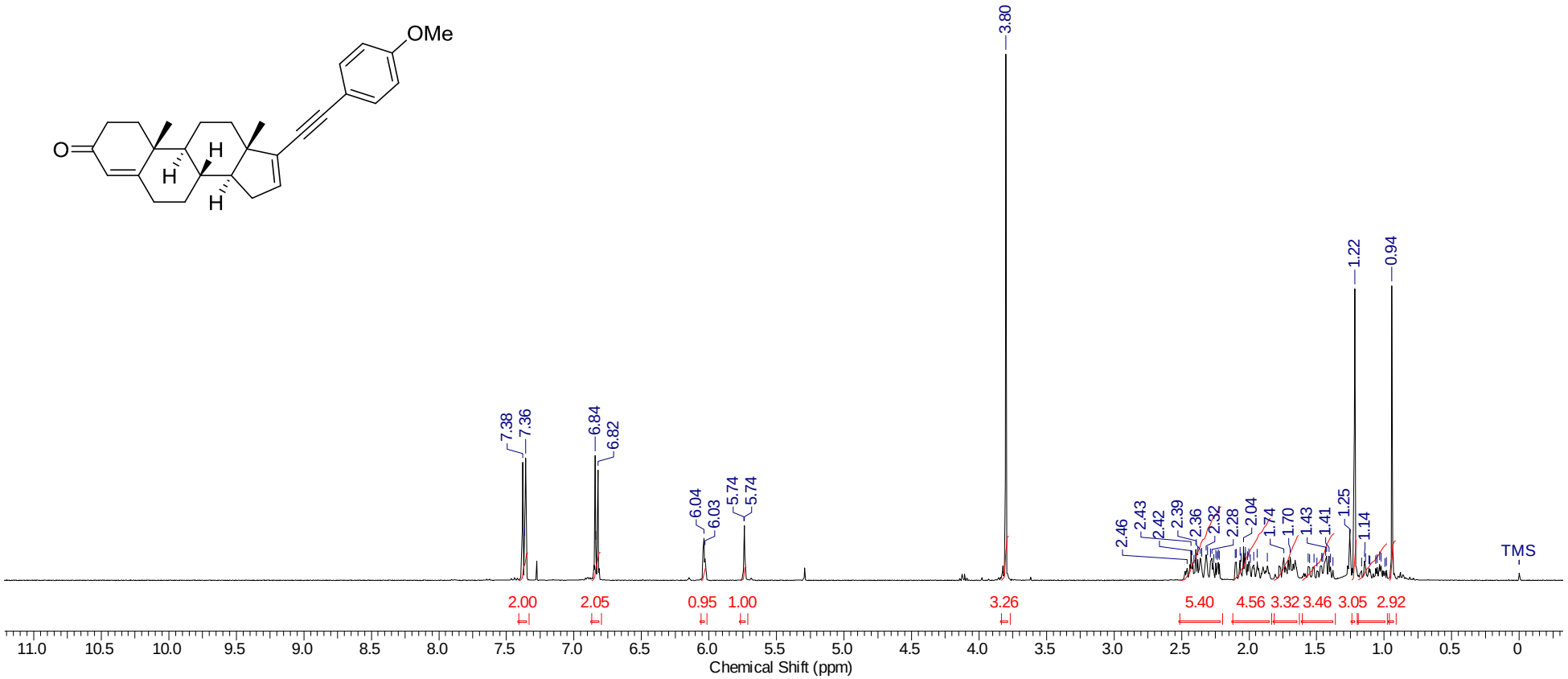
3-[4-(4-Methoxyphenyl)buta-1,3-diyn-1-yl]quinoline (5d)

^{13}C NMR (100 MHz, $CDCl_3$)



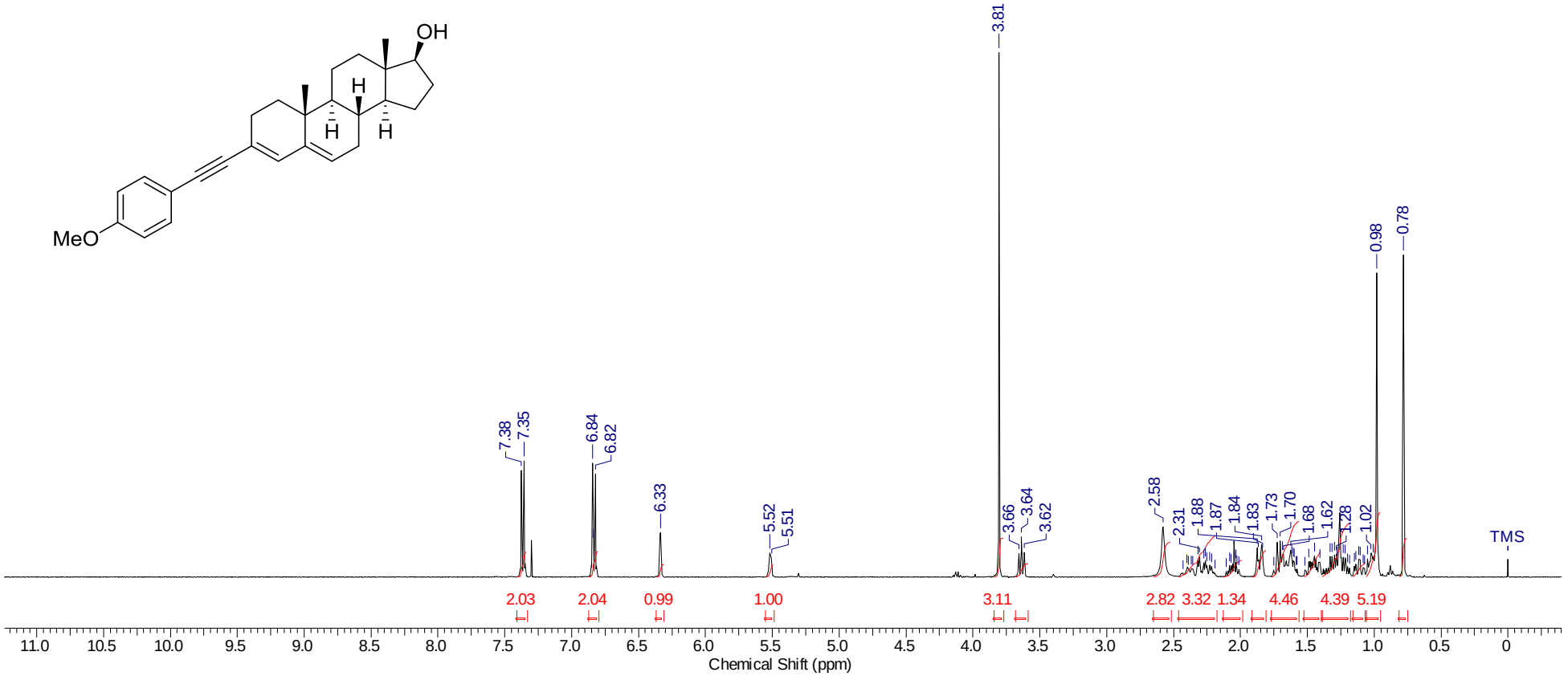
17-[(4-Methoxyphenyl)ethynyl]androsta-4,16-dien-3-one (5e)

^{1}H NMR (400 MHz, $CDCl_3$)



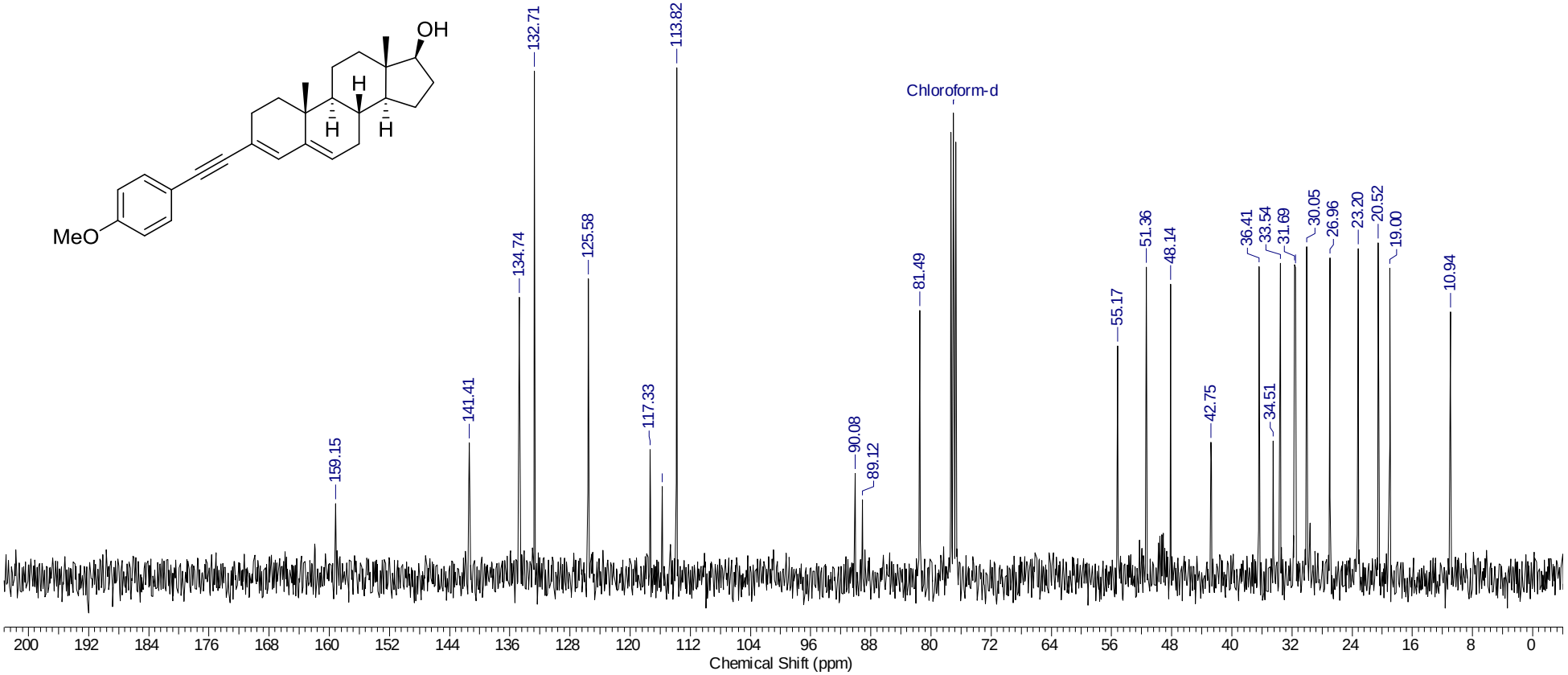
3-[(4-Methoxyphenyl)ethynyl]androsta-3,5-dien-17β-ol (5f)

^{1}H NMR (400 MHz, $CDCl_3$–CD_3OD)



3-[(4-Methoxyphenyl)ethynyl]androsta-3,5-dien-17β-ol (5f)

^{13}C NMR (100 MHz, $CDCl_3$–CD_3OD)



References

[1] Y. N. Kotovshchikov, G. V. Latyshev, N. V. Lukashev and I. P. Beletskaya, *Eur. J. Org. Chem.*, 2013, 7823–7832.

[2] V. P. Dyadchenko, M. A. Dyadchenko, V. N. Okulov and D. A. Lemenovskii, *J. Organomet. Chem.*, 2011, **696**, 468–472.

[3] S. Ruengsangtongkul, N. Chaisan, C. Thongsornkleeb, J. Tummatorn and S. Ruchirawat, *Org. Lett.*, 2019, **21**, 2514–2517.

[4] L. Liang, H.-Y. Niu, R.-L. Li, Y.-F. Wang, J.-K. Yan, C.-G. Li and H.-M. Guo, *Org. Lett.*, 2020, **22**, 6842–6846.

[5] Y.-H. Chen, C. P. Tüllmann, M. Ellwart and P. Knochel, *Angew. Chem. Int. Ed.*, 2017, **56**, 9236–9239.

[6] M. Csékei, Z. Novák and A. Kotschy, *Tetrahedron*, 2008, **64**, 975–982.

[7] D. Jung, S. Y. Park and S. Kim, *Bull. Korean Chem. Soc.*, 2022, **43**, 110–116.

[8] B.-B. Wang, Y.-M. Ye, J.-J. Chen, X.-X. Zhou, J.-M. Lu and L.-X. Shao, *Bull. Chem. Soc. Jpn.*, 2011, **84**, 526–530.

[9] Y. Xu, J. Zhao, X. Tang, W. Wu and H. Jiang, *Adv. Synth. Catal.*, 2014, **356**, 2029–2039.

[10] H. Peng, Y.-Q. Chen, S.-L. Mao, Y.-X. Pi, Y. Chen, Z.-Y. Lian, T. Meng, S.-H. Liu and G.-A. Yu, *Org. Biomol. Chem.*, 2014, **12**, 6944–6952.

[11] R. Bernini, S. Cacchi, G. Fabrizi, G. Forte, F. Petrucci, A. Prastaro, S. Niembro, A. Shafir and A. Vallribera, *Org. Biomol. Chem.*, 2009, **7**, 2270–2273.

[12] X.-T. Zhu, Q. Zhao, F. Liu, A.-F. Wang, P.-J. Cai, W.-J. Hao, S.-J. Tu and B. Jiang, *Chem. Commun.*, 2017, **53**, 6828–6831.

[13] Y. Takemoto, H. Yoshida and K. Takaki, *Chem. Eur. J.*, 2012, **18**, 14841–14844.

[14] H. Hu, F. Yang and Y. Wu, *J. Org. Chem.*, 2013, **78**, 10506–10511.

[15] A. Elangovan, S.-W. Yang, J.-H. Lin, K.-M. Kao and T.-I. Ho, *Org. Biomol. Chem.*, 2004, **2**, 1597–1602.

[16] S. Prateeptongkum, K. M. Driller, R. Jackstell, A. Spannenberg and M. Beller, *Chem. Eur. J.*, 2010, **16**, 9606–9615.

[17] F. Bellina, M. Biagetti, S. Guariento, M. Lessi, M. Fausti, P. Ronchi and E. Rosadoni, *RSC Adv.*, 2021, **11**, 25504–25509.

[18] A. Sapegin and M. Krasavin, *J. Org. Chem.*, 2019, **84**, 8788–8795.

[19] S. S. Bag, R. Kundu and M. Das, *J. Org. Chem.*, 2011, **76**, 2332–2337.

[20] S. Qiu, C. Zhang, R. Qiu, G. Yin and J. Huang, *Adv. Synth. Catal.*, 2018, **360**, 313–321.

[21] S. Ahammed, D. Kundu and B. C. Ranu, *J. Org. Chem.*, 2014, **79**, 7391–7398.

[22] S. M. Weber, J. Queder and G. Hilt, *Chem. Eur. J.*, 2020, **26**, 12129–12133.

[23] C. Savarin, J. Srogl and L. S. Liebeskind, *Org. Lett.*, 2001, **3**, 91–93.

[24] Y. N. Kotovshchikov, G. V. Latyshev, N. V. Lukashev and I. P. Beletskaya, *Org. Biomol. Chem.*, 2015, **13**, 5542–5555.