

Supporting Information

Copper immobilized on nano-silica triazine dendrimer (Cu(II)-TD@nSiO₂) catalyzed synthesis of symmetrical and unsymmetrical 1,3-diyne under aerobic conditions and ambient temperature

Mahboobeh Nasr-Esfahani,^a Iraj Mohammadpoor-Baltork,^{*a} Ahmad Reza Khosropour,^{*a} Majid Moghadam,^a Valliollah Mirkhani,^a Shahram Tangestaninejad,^a Vladislav Agabekov,^b and Hadi Amiri Rudbari^{a1}

^a Department of Chemistry, Catalysis Division, University of Isfahan, Isfahan 81746-73441, Iran

^b Institut für Organische Chemie, Albert-Ludwigs-Universität Freiburg, Albertstrasse 21, 79104, Freiburg, Germany

E-mail addresses: imbaltork@sci.ui.ac.ir; arkhosropour@sci.ui.ac.ir

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1. Experimental Section

Melting points were determined with a Stuart Scientific SMP2 apparatus. FT-IR spectra were recorded on a Nicolet-Impact 400D instrument in the range of 400-4000 cm^{-1} . ^1H and ^{13}C NMR (400 and 100 MHz) spectra were recorded on a Bruker Avance 400 spectrometer using CDCl_3 as solvent. Mass spectra were recorded on a Platform II spectrometer from Micromass; EI mode at 70 eV. Elemental analysis was done on a LECO, CHNS-932 analyzer. The Cu content of the catalyst was measured by an inductively coupled plasma optical emission spectrometry (ICP-OES), using a Jarrell-Ash 1100 ICP analyzer. The Cu(II)-TD@nSiO₂ catalyst was prepared according to the reported procedure.¹

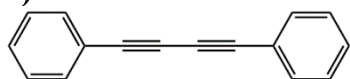
Typical procedure for synthesis of symmetrical 1,3-diyne 2a

A mixture of phenylacetylene **1a** (1 mmol), Cu(II)-TD@nSiO₂ (0.6 mol%), DBU (20 mol%) in acetonitrile (2 mL) was stirred under aerobic conditions at room temperature for 1.5 h. After completion of the reaction, as indicated by TLC (eluent: petroleum ether/ethyl acetate, 20:1), the catalyst was separated by centrifugation and washed with acetonitrile (5 mL). The solvent was evaporated and the residue was purified on a small bed of silica gel using petroleum ether/ethyl acetate (20:1) as eluent to afford the corresponding 1,3-diyne **2a** in 99% yield.

Typical procedure for synthesis of unsymmetrical 1,3-diyne 3an

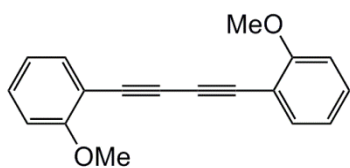
A mixture of phenylacetylene **1a** (1 mmol) and ethyl propiolate **1n** (0.5 mmol), Cu(II)-TD@nSiO₂ (0.6 mol%) and DBU (20 mol%) in acetonitrile (2 mL) was stirred under aerobic conditions at room temperature for 2 h. The progress of the reaction was monitored by TLC (eluent: petroleum ether/ethyl acetate, 20:1). After completion of the reaction, the catalyst was separated by centrifugation and washed with acetonitrile (5 mL). Evaporation of the solvent followed by purification of the crude product by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate, 20:1) afforded the desired unsymmetrical 1,3-diyne **3an** in 89% yield.

2. Spectroscopic data of the products 2a-2n (Table 2) and 3an-3dk (Table 4):



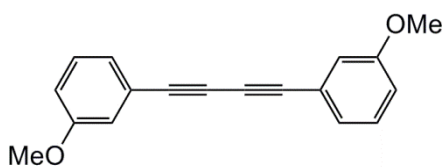
1,4-Diphenylbuta-1,3-diyne (Table 2, 2a):² Yield 99%. Mp 86-

87 °C. IR (KBr): ν_{\max} = 3077, 2924, 2146, 1590, 1482, 914, 754, 684, 524 cm^{-1} . ¹H NMR (400 MHz, CDCl_3): δ = 7.53 (dd, ¹*J* = 8.0, ²*J* = 1.6 Hz, 4H), 7.37-7.30 (m, 6H). MS: *m/z* (%): 204.13 ($[\text{M}+2]^+$, 2.40), 202.11 ($[\text{M}^+]$, 90.77), 200.11 (34.87), 174.09 (10.90), 150.11 (43.08), 122.07 (30.00), 110.06 (36.92), 98.05 (63.59), 74.04 (73.33), 63.09 (85.64), 50.10 (100.00).



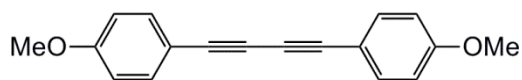
1,4-Bis(2-methoxyphenyl)buta-1,3-diyne (Table 2, 2b):³ Yield

97%. Mp 136-138 °C. IR (KBr): ν_{\max} = 3064, 3001, 2137, 1590, 1485, 1248, 1019, 753 cm^{-1} . ¹H NMR (400 MHz, CDCl_3): δ = 7.42 (dd, ¹*J* = 7.6, ²*J* = 1.6 Hz, 2H), 7.27 (td, ¹*J* = 7.6, ²*J* = 1.6 Hz, 2H), 6.86 (dd, ¹*J* = 7.6, ²*J* = 7.2 Hz, 4H), 3.82 (s, 6H). MS: *m/z* (%): 262.04 ($[\text{M}^+]$, 71.03), 247.11 (15.10), 235.69 (31.12), 172.04 (30.11), 108.05 (47.88), 69.13 (27.10), 57.15 (85.14), 41.15 (100.00).



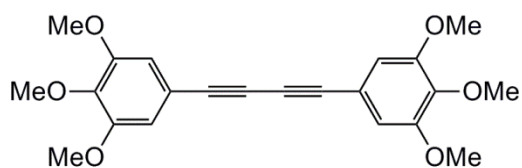
1,4-Bis(3-methoxyphenyl)buta-1,3-diyne (Table 2, 2c):⁴

Yield 98%. Mp 92-93 °C. IR (KBr): ν_{\max} = 3071, 2926, 2218, 1594, 1462, 1261, 1048, 782, 684 cm^{-1} . ¹H NMR (400 MHz, CDCl_3): δ = 7.25 (t, *J* = 8.0 Hz, 2H), 7.13 (dt, ¹*J* = 8.0, ²*J* = 1.2 Hz, 2H), 7.04 (dd, ¹*J* = 4.0, ²*J* = 1.6 Hz, 2H), 6.94 (ddd, ¹*J* = 8.0, ²*J* = 3.6, ³*J* = 1.2 Hz, 2H), 3.80 (s, 6H). MS: *m/z* (%): 263.13 ($[\text{M}+1]^+$, 5.86), 262.04 ($[\text{M}^+]$, 37.11), 218.98 (14.18), 189.09 (15.21), 176.00 (21.39), 150.05 (21.39), 111.03 (32.73), 85.08 (34.28), 71.07 (54.38), 57.07 (100.00), 43.12 (81.44).



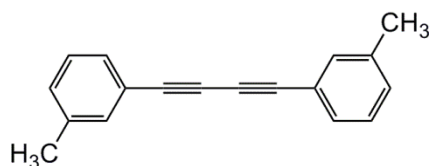
1,4-Bis(4-methoxyphenyl)buta-1,3-diyne (Table 2,

2d):² Yield 99%. Mp 139-141 °C. IR (KBr): ν_{\max} = 2999, 2929, 2134, 1597, 1500, 1250, 1023, 835, 534 cm^{-1} . ¹H NMR (400 MHz, CDCl₃): δ = 7.47 (dt, ¹*J* = 8.4, ²*J* = 2.4 Hz, 4H), 6.87 (dt, ¹*J* = 7.2, ²*J* = 2.0 Hz, 4H), 3.82 (s, 6H). MS: *m/z* (%): 263.03 ([M+1]⁺, 18.07), 262.04 ([M⁺], 89.11), 246.99 (53.47), 219.03 (18.32), 204.03 (14.60), 176.04 (33.17), 149.01 (42.08), 111.05 (21.78), 85.12 (19.31), 63.08 (32.18), 57.13 (73.21), 43.16 (100.00).



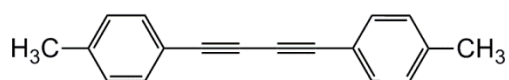
1,4-Bis(3,4,5-trimethoxyphenyl)buta-1,3-diyne

(Table 2, 2e):⁵ Yield 94%. Mp 200-202 °C. IR (KBr): ν_{\max} = 3092, 2959, 2928, 2141, 1573, 1462, 1272, 1127, 992, 819, 712 cm^{-1} . ¹H NMR (400 MHz, CDCl₃): δ = 7.71 (dd, ¹*J* = 8.4, ²*J* = 3.2 Hz, 2H), 7.54 (dd, ¹*J* = 8.4, ²*J* = 3.2 Hz, 2H), 3.87 (s, 6H), 3.86 (s, 12H). MS: *m/z* (%): 384.03 ([M+2]⁺, 4.49), 383.04 ([M+1]⁺, 22.18), 382.01 ([M⁺], 100.00), 367.01 (38.03), 308.98 (6.56), 221.06 (3.57), 191.10 (12.15), 139.11 (13.73), 111.06 (29.23), 69.04 (48.59), 53.06 (73.94), 41.12 (92.75).



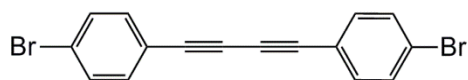
1,4-Di-*m*-tolylbuta-1,3-diyne (Table 2, 2f):² Yield 98%.

Mp 73-74 °C. IR (KBr): ν_{\max} = 3033, 2917, 2138, 1645, 1591, 1478, 1038, 903, 787, 684 cm^{-1} . ¹H NMR (400 MHz, CDCl₃): δ = 7.26 (s, 2H), 7.24 (s, 2H), 7.16 (d, *J* = 7.2 Hz, 2H), 7.11 (d, *J* = 8.0 Hz, 2H), 2.26 (s, 6H). MS: *m/z* (%): 231.08 ([M+1]⁺, 19.72), 230.67 ([M⁺], 100.00), 215.05 (11.00), 202.05 (7.79), 163.05 (10.12), 115.06 (20.42), 77.10 (18.66), 63.09 (50.70), 51.11 (63.38), 43.18 (67.61).



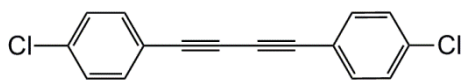
1,4-Di-*p*-tolylbuta-1,3-diyne (Table 2, 2g):² Yield

98%. Mp 183-184 °C. IR (KBr): ν_{\max} = 3077, 2959, 2925, 2133, 1500, 1273, 1120, 809, 521 cm^{-1} . ¹H NMR (400 MHz, CDCl₃): δ = 7.42 (d, J = 8.0 Hz, 4H), 7.14 (d, J = 8.0 Hz, 4H), 2.36 (s, 6H). MS: m/z (%): 232.17 ([M+2]⁺, 1.23), 230.67 ([M⁺], 63.68), 215.15 (12.15), 202.13 (8.14), 189.13 (6.57), 167.07 (65.09), 149.02 (97.17), 113.17 (37.26), 104.07 (50.94), 83.11 (41.51), 70.11 (84.43), 57.10 (100.00), 41.13 (96.23).



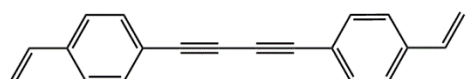
1,4-Bis(4-bromophenyl)buta-1,3-diyne (Table 2, 2h):⁶

Yield 92%. Mp 260-261 °C. IR (KBr): ν_{\max} = 3090, 2924, 1624, 1119, 824 cm^{-1} . ¹H NMR (400 MHz, CDCl₃): δ = 7.50 (dt, 1J = 8.4, 2J = 2.0 Hz, 4H), 7.39 (dt, 1J = 7.2, 2J = 2.4 Hz, 4H). MS: m/z (%): 361.76 ([M+4]⁺, 0.31), 359.75 ([M+2]⁺, 0.54), 357.81 ([M⁺], 0.41), 335.72 (0.55), 279.12 (1.43), 167.05 (6.27), 149.03 (21.74), 111.11 (6.52), 83.12 (17.32), 69.13(27.45), 57.15 (80.43), 43.20 (100.00).



1,4-Bis(4-chlorophenyl)buta-1,3-diyne (Table 2, 2i):⁵

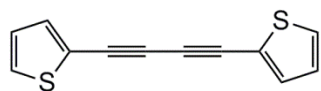
Yield 94%. Mp 250-251 °C. IR (KBr): ν_{\max} = 3084, 2929, 2141, 1590, 1110, 846, 530 cm^{-1} . ¹H NMR (400 MHz, CDCl₃): δ = 7.46 (d, J = 7.6 Hz, 4H), 7.33 (d, J = 8.4 Hz, 4H). MS: m/z (%): 273.03 ([M+2]⁺, 0.73), 271.03 ([M⁺], 0.41), 235.72 (20.18), 201.04 (43.12), 171.05 (18.24), 140.03 (21.11), 111.02 (33.52), 83.12 (17.32), 71.12 (34.59), 57.05 (68.54), 43.11 (100.00).



1,4-Bis(4-vinylphenyl)buta-1,3-diyne (Table 2, 2j):

Yield 90%. Mp 113-115 °C. IR (KBr): ν_{\max} = 3097, 3054, 2939, 2124, 1643, 1594, 1020, 909, 540 cm^{-1} . ¹H NMR (400 MHz, CDCl₃): δ = 7.42 (dd, 1J = 8.4, 2J = 2.0 Hz, 4H), 7.31 (d, J = 8.4 Hz, 4H), 6.66 (dd, 1J = 10.8, 2J = 6.8 Hz, 2H), 5.7 (dd, 1J = 18.0, 2J = 0.4 Hz, 2H), 5.26 (dd, 1J = 10.8, 2J = 0.4 Hz, 2H). ¹³C NMR (100 MHz, CDCl₃): δ = 138.38, 136.07, 132.71,

126.24, 120.98, 115.47, 82.01, 74.63. MS: m/z (%): 255.08 ($[M+1]^+$, 11.40), 254.06 ($[M]^+$, 79.08), 228.11 (10.11), 200.08 (23.35), 154.10 (21.44), 126.07 (44.68), 102.09 (50.94), 72.24 (82.41), 57.07 (100.00), 41.10 (76.23). Anal. Calcd for $C_{20}H_{14}$: C, 94.45; H, 5.55. Found: C, 94.21 H, 5.58.

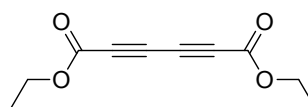


1,4-Di(thiophene-2-yl)buta-1,3-diyne (Table 2, 2k):² Yield 97%.

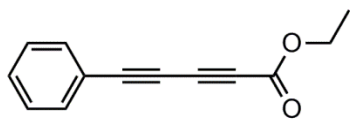
Mp 89-90 °C. IR (KBr): ν_{\max} = 3100, 2920, 2198, 2136, 1617, 1220, 836, 710 cm^{-1} . 1H NMR (400 MHz, $CDCl_3$): δ = 7.18-7.09 (m, 4H), 6.85 (t, J = 4.8 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 134.42, 128.93, 127.23, 121.93, 77.76, 76.64.

$n-C_5H_{11}-\equiv\equiv-C_5H_{11}-n$ **Tetradeca-6,8-diyne (Table 2, 2l):**⁷ Yield 86%. Oil. IR (KBr): ν_{\max} = 3050, 2964, 2936, 2219, 1473, 1362, 1150 cm^{-1} . 1H NMR (400 MHz, $CDCl_3$): δ = 2.19 (t, J = 7.2 Hz, 4H), 1.48 (quin, J = 7.2 Hz, 4H), 1.32-1.26 (m, 8H), 0.84 (t, J = 7.2 Hz, 6H). MS: m/z (%): 190.14 ($[M]^+$, 0.96), 181.04 (1.72), 119.06 (28.08), 105.04 (55.07), 91.01 (100.00), 79.08 (50.00), 55.08 (42.93), 41.12 (92.75).

$n-C_4H_9-\equiv\equiv-C_4H_9-n$ **Dodeca-5,7-diyne (Table 2, 2m):**⁸ Yield 87%. Oil. IR (KBr): ν_{\max} = 2960, 2932, 2232, 1462, 1255, 1168 cm^{-1} . 1H NMR (400 MHz, $CDCl_3$): δ = 2.20 (t, J = 7.6 Hz, 4H), 1.45-1.38 (m, 4H), 1.35-1.29 (m, 4H), 0.85 (t, J = 7.2 Hz, 6H).

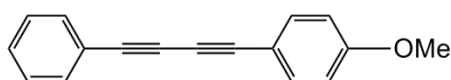


Diethyl hexa-2,4-diyne-1,6-dioate (Table 2, 2n):⁹ Yield 86%. Oil. IR (KBr): ν_{\max} = 2957, 2926, 2147, 1725, 1462, 1243, 1120, 739 cm^{-1} . 1H NMR (400 MHz, $CDCl_3$): δ = 4.28 (q, J = 7.2 Hz, 4H), 1.34 (t, J = 7.2 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 156.24, 71.60, 67.09, 61.67, 13.54.



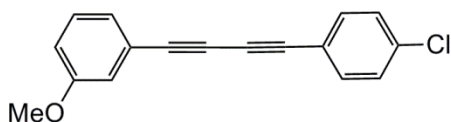
Ethyl 5-phenylpenta-2,4-dienoate (Table 4, 3an):¹⁰ Yield 89%.

Oil. IR (KBr): ν_{\max} = 3066, 2926, 2226, 2149, 1737, 1602, 1214, 1071, 756, 690 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): δ = 7.48 (dd, 1J = 8.0, 2J = 1.2 Hz, 2H), 7.31 (td, 1J = 7.6, 2J = 1.6 Hz, 3H), 4.23 (q, J = 7.2 Hz, 2H), 1.28 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ = 180.87, 133.09, 130.45, 128.61, 117.14, 78.64, 76.69, 62.47, 28.93, 14.00.



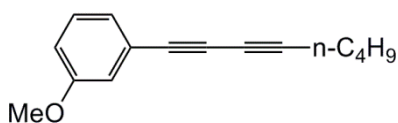
1-Methoxy-4-(phenylbuta-1,3-diyne-1-yl)benzene

(Table 4, 3ad):¹¹ Yield 95%. Mp 88-90 °C. IR (KBr): ν_{\max} = 3050, 2986, 2211, 2138, 1595, 1486, 1247, 1026, 826, 754 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): δ = 7.38 (dd, 1J = 8.0, 2J = 1.6 Hz, 2H), 7.34 (dt, 1J = 8.4, 2J = 2.0 Hz, 2H), 7.20 (m, 2H), 7.10 (s, 1H), 6.72 (dt, 1J = 7.2, 2J = 1.6 Hz, 2H), 3.67 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ = 160.38, 134.13, 132.44, 129.03, 128.42, 122.03, 114.17, 81.82, 81.03, 74.17, 72.74, 55.35.



1-((4-Chlorophenyl)buta-1,3-diyne-1-yl)-3-

methoxybenzene (Table 4, 3ci): Yield 93%. Mp 102-103 °C. IR (KBr): ν_{\max} = 3064, 2955, 2211, 2151, 1591, 1248, 1036, 822, 777 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): δ = 7.48 (dt, 1J = 8.4, 2J = 2.0 Hz, 2H), 7.35 (dd, 1J = 8.4, 2J = 2.0 Hz, 2H), 7.28 (d, J = 4.0 Hz, 1H), 7.16 (dt, 1J = 7.6, 2J = 1.2 Hz, 1H), 7.07 (dd, 1J = 4.0, 2J = 1.6 Hz, 1H), 6.97 (ddd, 1J = 8.0, 2J = 3.6, 3J = 0.8 Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3): δ = 159.31, 135.41, 133.69, 129.59, 128.88, 125.10, 122.53, 120.27, 117.10, 116.16, 82.03, 80.33, 74.84, 74.84, 73.45, 55.33. Anal. Calcd for $\text{C}_{17}\text{H}_{11}\text{ClO}$: C, 76.55; H, 4.16. Found: C, 76.39; H, 4.13.



1-Methoxy-3-(octa-1,3-diyne-1-yl)benzene (Table 4, 3cm):¹²

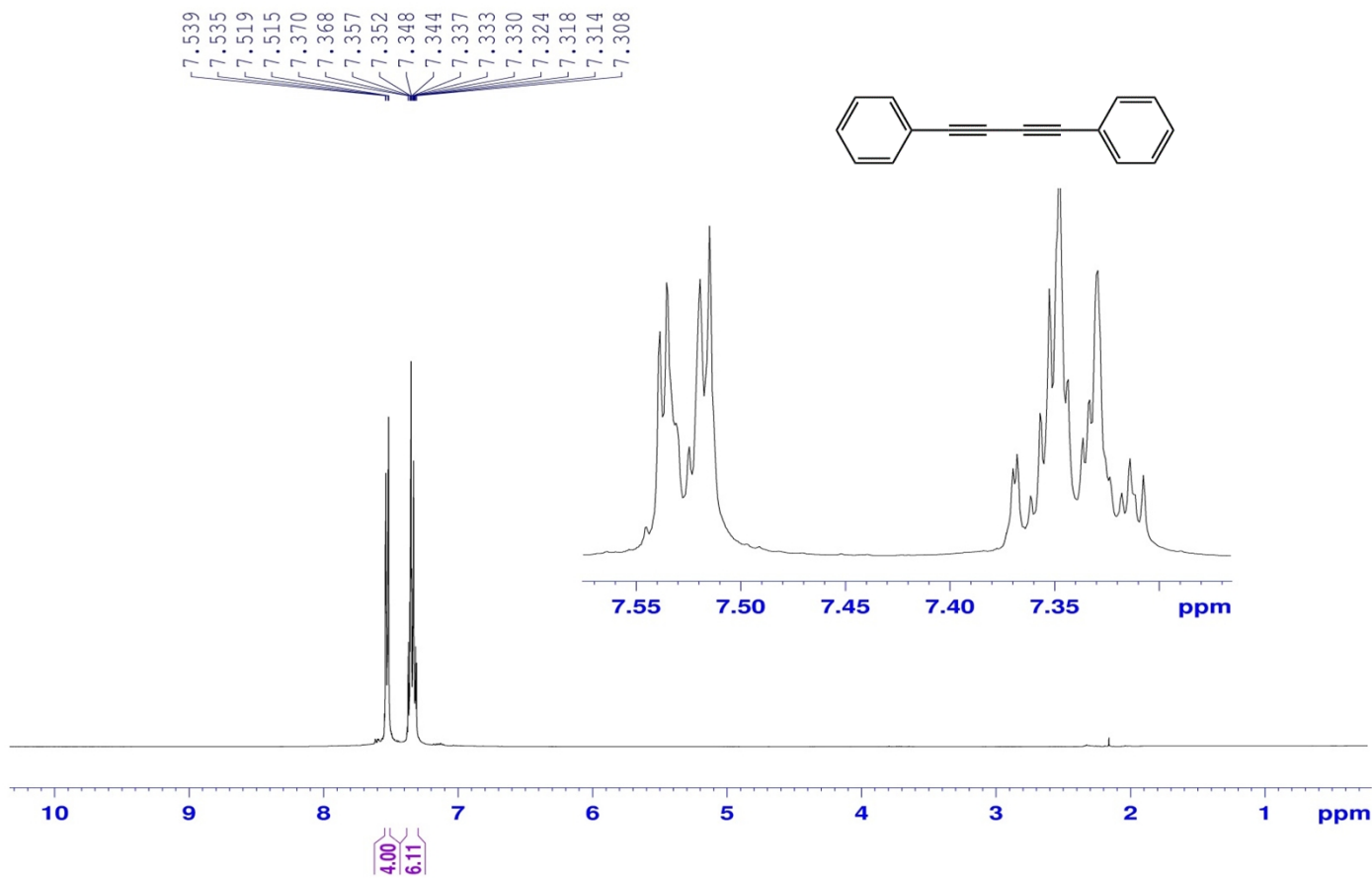
Yield 78%. Oil. IR (KBr): ν_{\max} = 3071, 2938, 2868, 2240, 2152, 1597, 1425, 1224, 1043, 781,

683 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): $\delta = 7.26$ (t, $J = 8.0$ Hz, 1H), 7.10 (dt, $^1J = 7.6$, $^2J = 1.2$ Hz, 1H), 7.02 (dd, $^1J = 4.0$, $^2J = 1.6$ Hz, 1H), 6.93 (ddd, $^1J = 8.4$, $^2J = 2.8$, $^3J = 0.8$ Hz, 1H), 3.81 (s, 3H), 2.40 (t, $J = 7.6$ Hz, 2H), 1.62-1.55(m, 2H), 1.53 (sex, $J = 7.2$, 2H), 0.97 (t, $J = 7.2$, 3H). ^{13}C NMR (100 MHz, CDCl_3): $\delta = 159.25$, 129.42, 125.06, 123.09, 117.11, 115.67, 84.93, 74.60, 74.20, 55.27, 30.27, 21.96, 19.28, 13.54.



(Table 4, 3dk): Yield 88%. Mp 74-75 $^{\circ}\text{C}$. IR (KBr): $\nu_{\text{max}} = 3092$, 2926, 2199, 2137, 1597, 1504, 1290, 1250, 832, 710, 466 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): $\delta = 7.32$ (dt, $^1J = 8.4$, $^2J = 2.8$ Hz, 2H), 7.17 (ddd, $^1J = 9.6$, $^2J = 4.8$, $^3J = 1.2$ Hz, 1H), 7.10 (s, 1H), 6.84 (dd, $^1J = 5.2$, $^2J = 3.6$ Hz, 1H), 6.71(dt, $^1J = 6.8$, $^2J = 2.4$ Hz, 2H), 3.66 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3): $\delta = 160.61$, 134.13, 134.01, 130.88, 128.81, 128.43, 127.13, 114.19, 83.89, 78.21, 74.00, 68.16, 55.36. Anal. Calcd for $\text{C}_{15}\text{H}_{10}\text{SO}$: C, 75.60; H, 4.23; S, 13.46. Found: C, 75.47; H, 4.25; S, 13.22.

2. ¹H and ¹³C NMR spectra of the products:
1,4-Diphenylbuta-1,3-diyne (Table 2, 2a): ¹H NMR (400 MHz, CDCl₃)



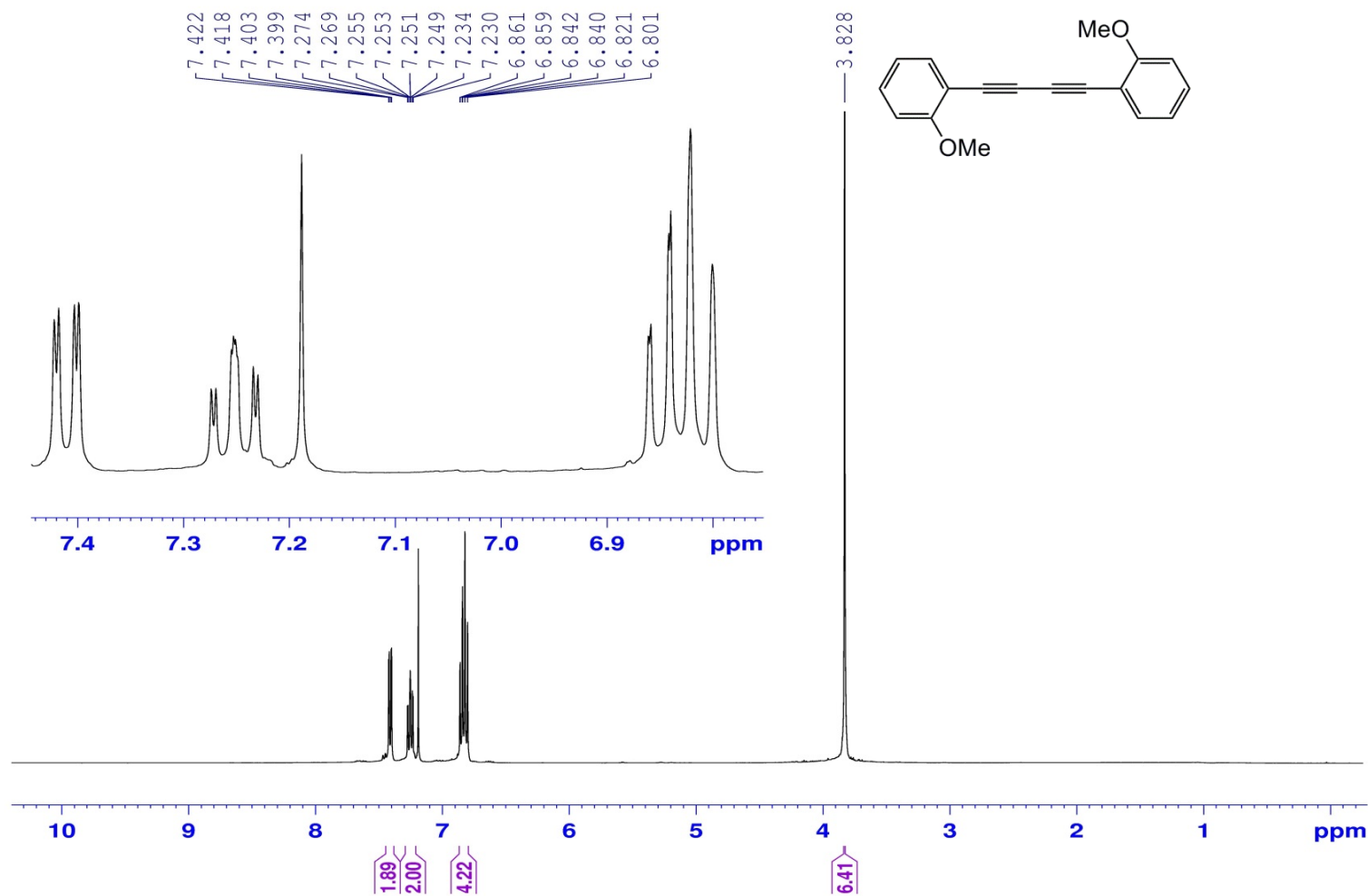
Instrument Specifications:
 NMR Spectrometer 500 MHz, Avance III 400
 Bruker, Germany

Type of experiment:
 1H Spectrum

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TD        65536
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1,4-Bis(2-methoxyphenyl)buta-1,3-diyne (Table 2 , 2b): ¹H NMR (400 MHz, CDCl₃)



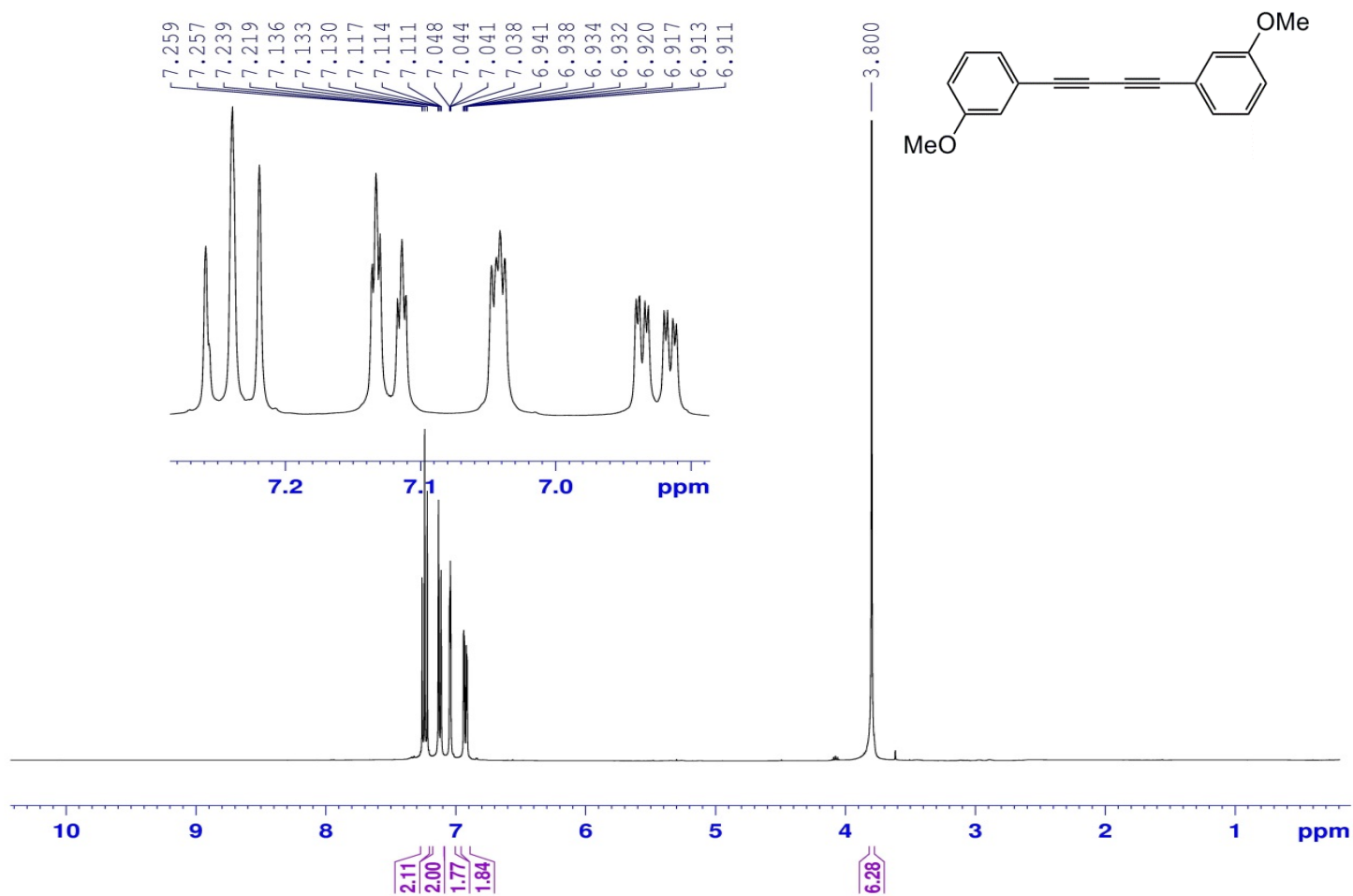
Instrument Specifications:
NMR Spectrometer 400 MHz, Avance III 400
Bruker, Germany

Type of experiment:
1H Spectrum

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NAME          Desktop
EXPNO         10
PROCNO        1
Date_         20130727
Time_         13.58
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg
TD            32768
SOLVENT       CDCl3
NS            32
DS            2
SWH           8417.509 Hz
FIDRES        0.256882 Hz
AQ            1.9464692 sec
RG            101
DW            59.400 usec
DE            6.50 usec
TE            295.4 K
D1            1.0000000 sec
TDO           1
----- CHANNEL f1 -----
NUC1          1H
P1            11.00 usec
PL1           -2.00 dB
PL1W          17.51671600 W
SFO1          400.1326008 MHz
S1            32768
SF            400.1300385 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

1,4-Bis(3-methoxyphenyl)buta-1,3-diyne (Table 2, 2c): ¹H NMR (400 MHz, CDCl₃)



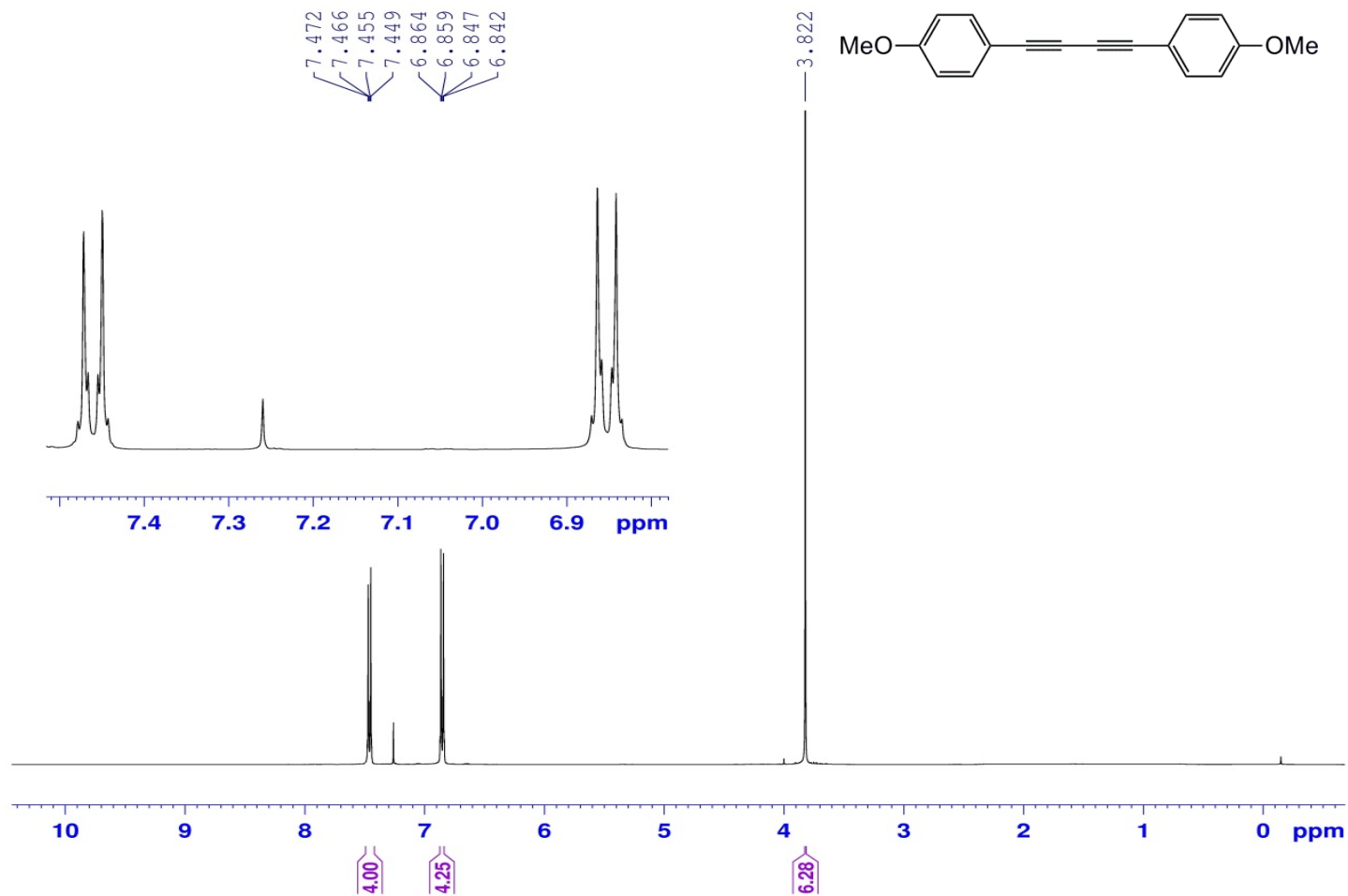
Instrument Specifications:
NMR Spectrometer 400 MHz, Avance III 400
Bruker, Germany

Type of experiment:
1H Spectrum

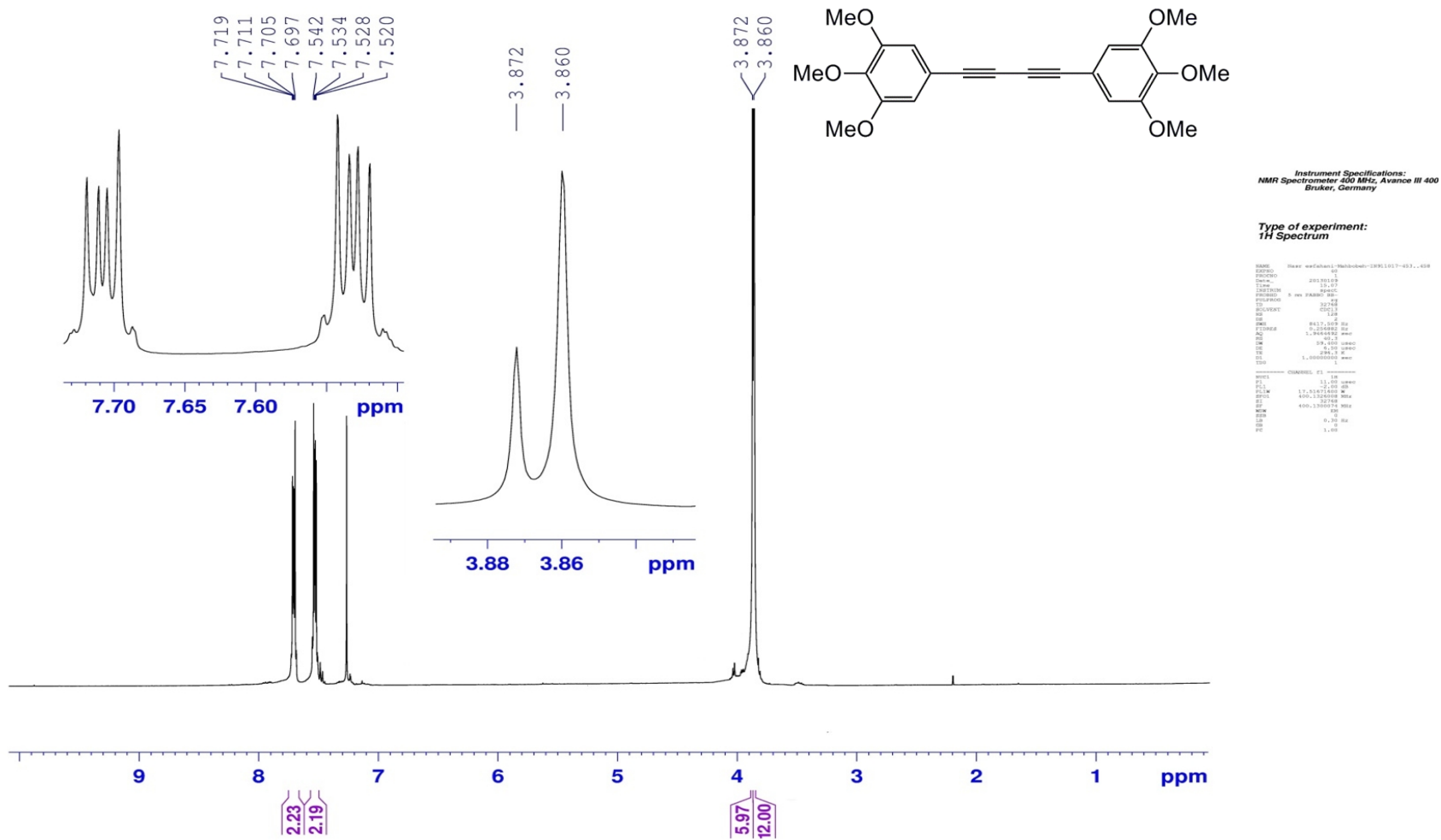
```

NAME      User: mstahani-Methoxybut-1,3-diyne-4,4'-diyl
EXPNO    1
PROCNO   1
F2      201.9143
TIME     14.78
PROBHD   5 mm BBO-400
PULPROG  zgpg30
PC       120.0
SOLVENT  CDCl3
NS       128
DS       4
SWH      813.1400 MHz
FIDRES   0.210000 Hz
AQ       1.190000 sec
RG       256
DM       59.400 ucm
DE       20.0
TE       300.2 K
DQ       1.0000000 sec
TD       1
----- CHANNEL f1 -----
NUC1      13
P1        12.00 usec
PL1       0.00 dB
PC1       17.000000 MHz
NUC2      1H
P2        6.00 usec
PL2       19.00 dB
PC2       400.142000 MHz
SFO       400.142000 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0.00 Hz
PC        1.00
    
```

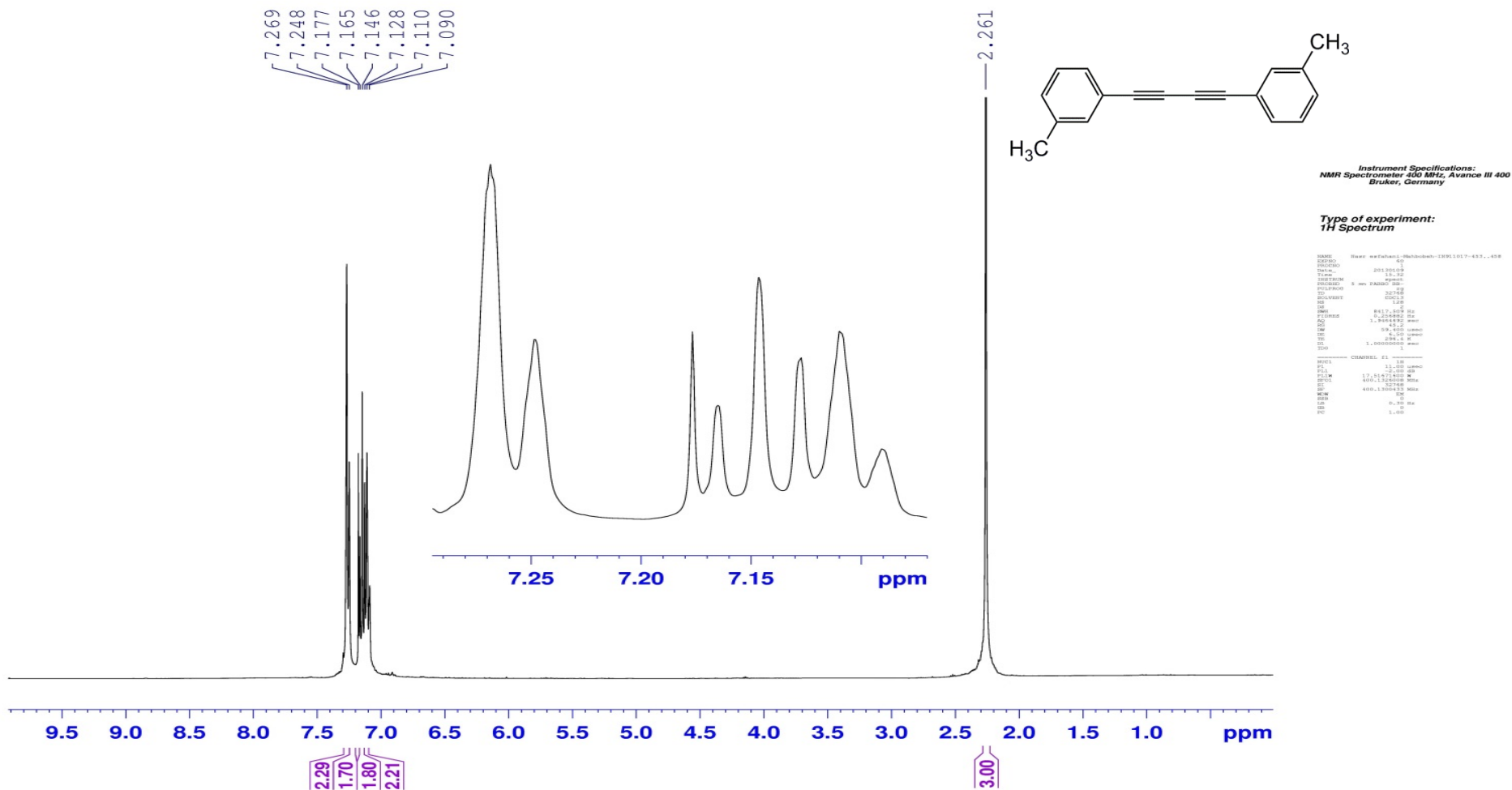
1,4-Bis(4-methoxyphenyl)buta-1,3-diyne (Table 2, 2d): ¹H NMR (400 MHz, CDCl₃)



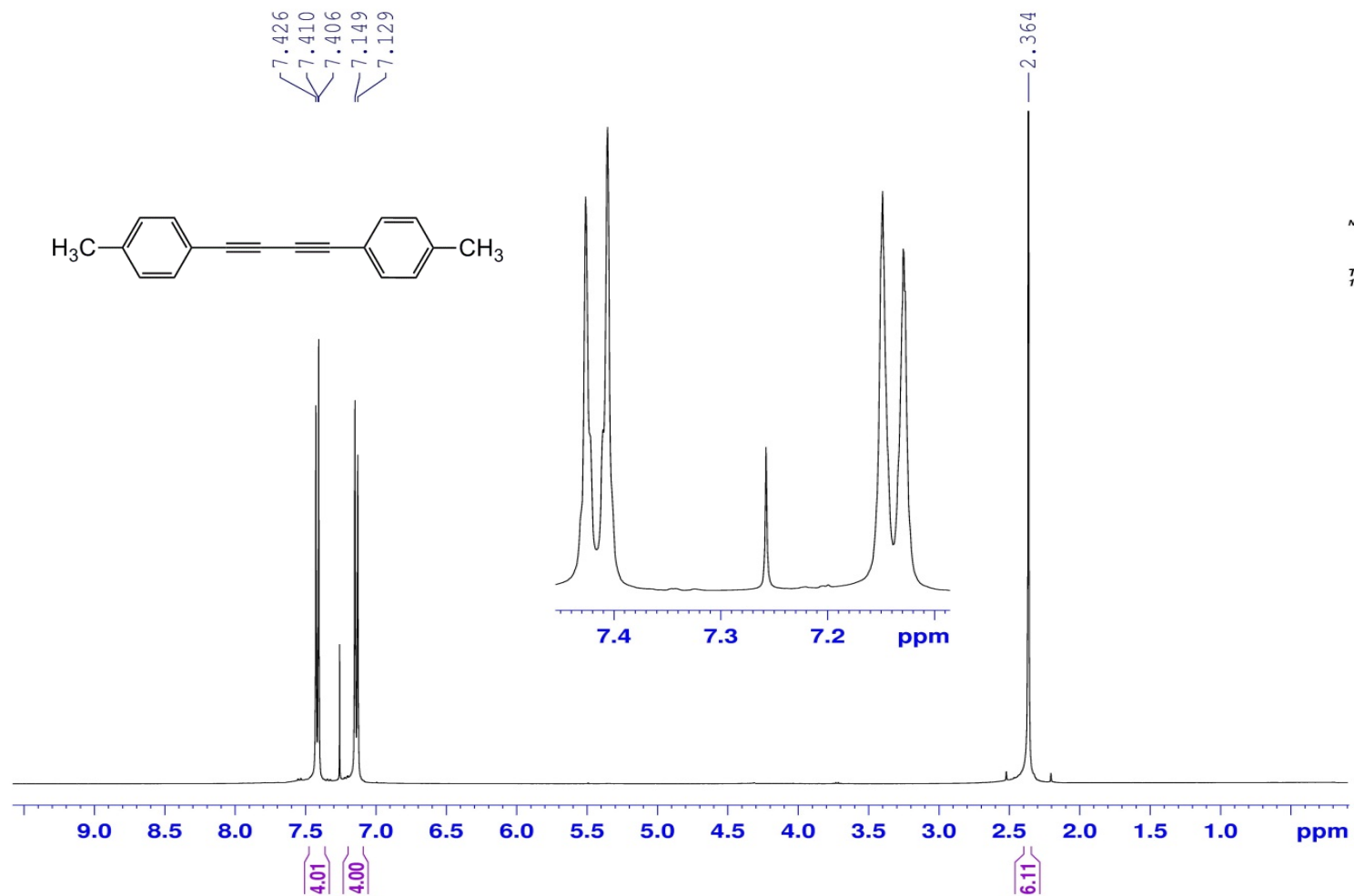
1,4-Bis(3,4,5-trimethoxyphenyl)buta-1,3-diyne (Table 2, 2e): ¹H NMR (400 MHz, CDCl₃)



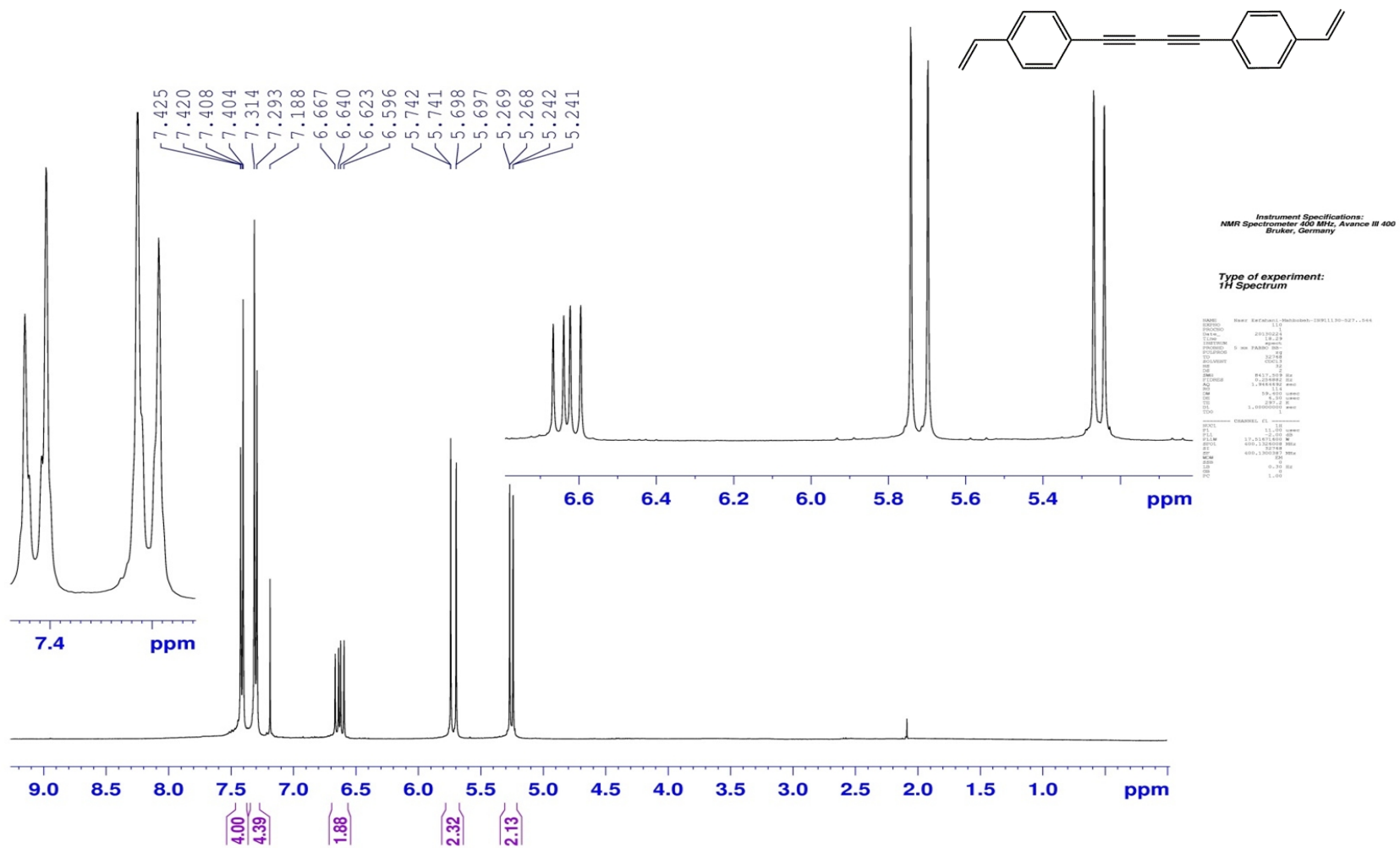
1,4-Di-*m*-tolylbuta-1,3-diyne (Table 2, 2f): ¹H NMR (400 MHz, CDCl₃)



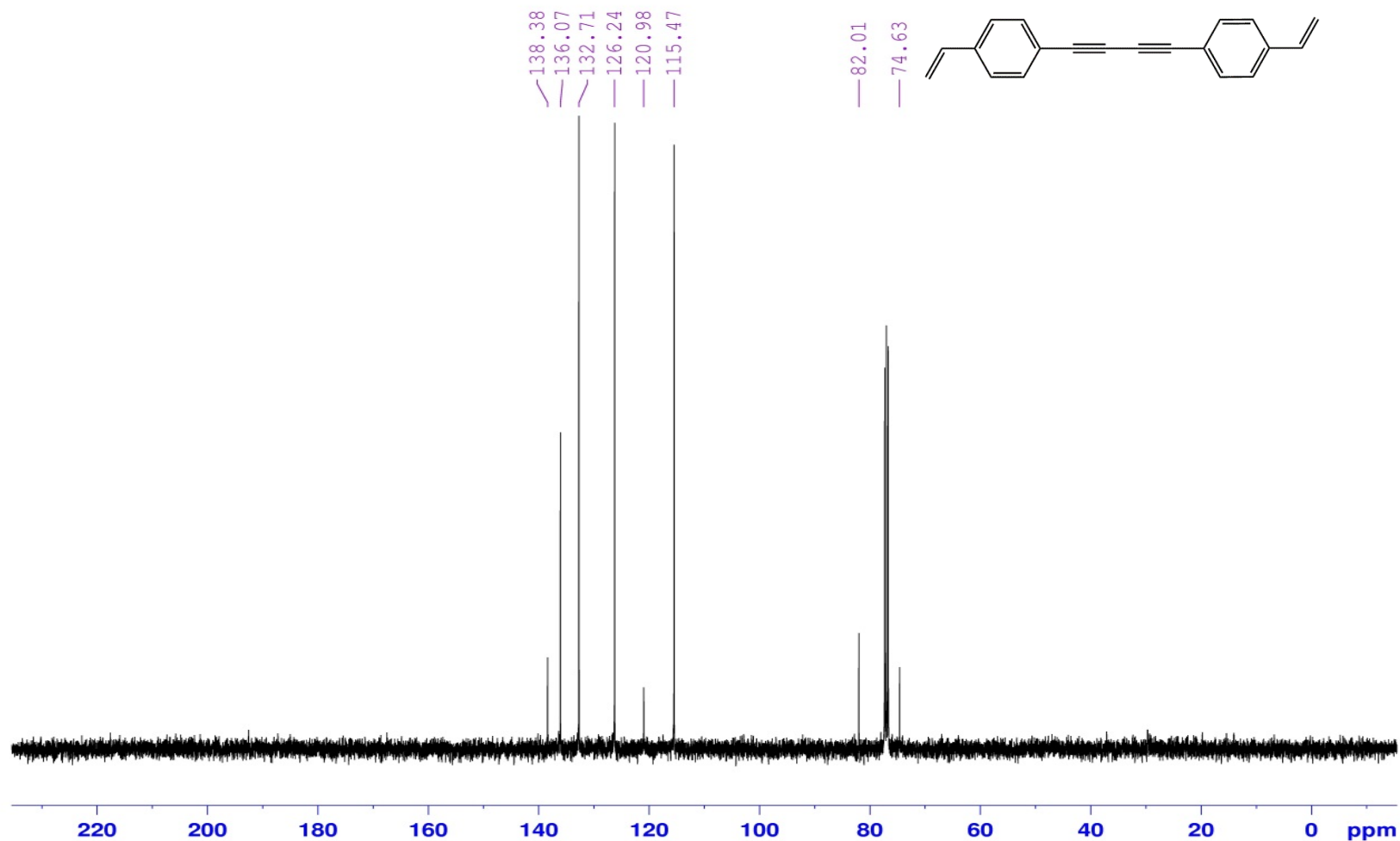
1,4-Di-*p*-tolylbuta-1,3-diyne (Table 2, 2g): ^1H NMR (400 MHz, CDCl_3)



1,4-Bis(4-vinylphenyl)buta-1,3-diyne (Table 2, 2j): ¹H NMR (400 MHz, CDCl₃)



1,4-Bis(4-vinylphenyl)buta-1,3-diyne (Table 2, 2j): ^{13}C NMR (100 MHz, CDCl_3)



Instrument Specific
MNR Spectrometer 400 MHz
Bruker, German

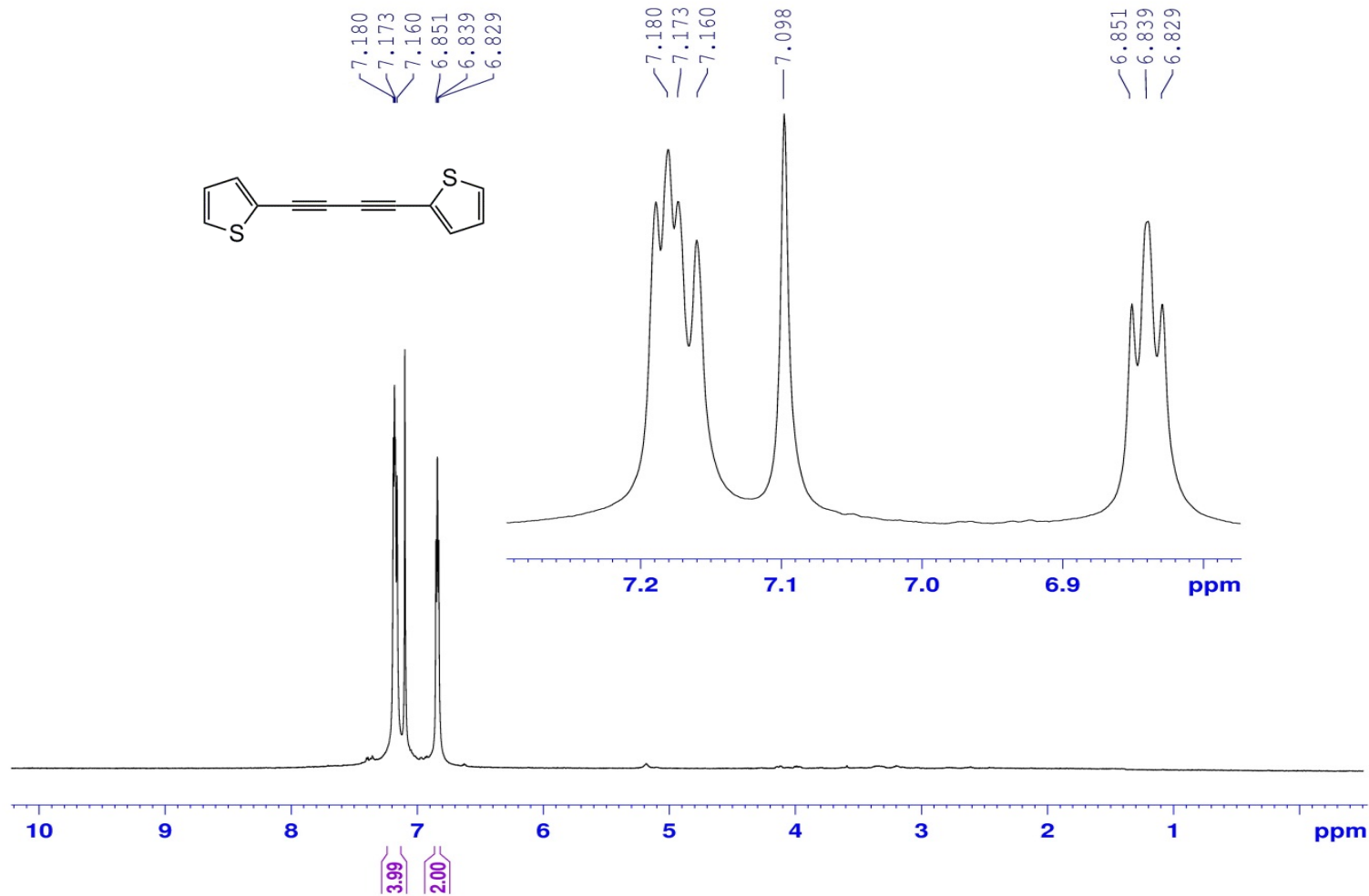
Type of experiment:
1H Decoupled 13C S

```

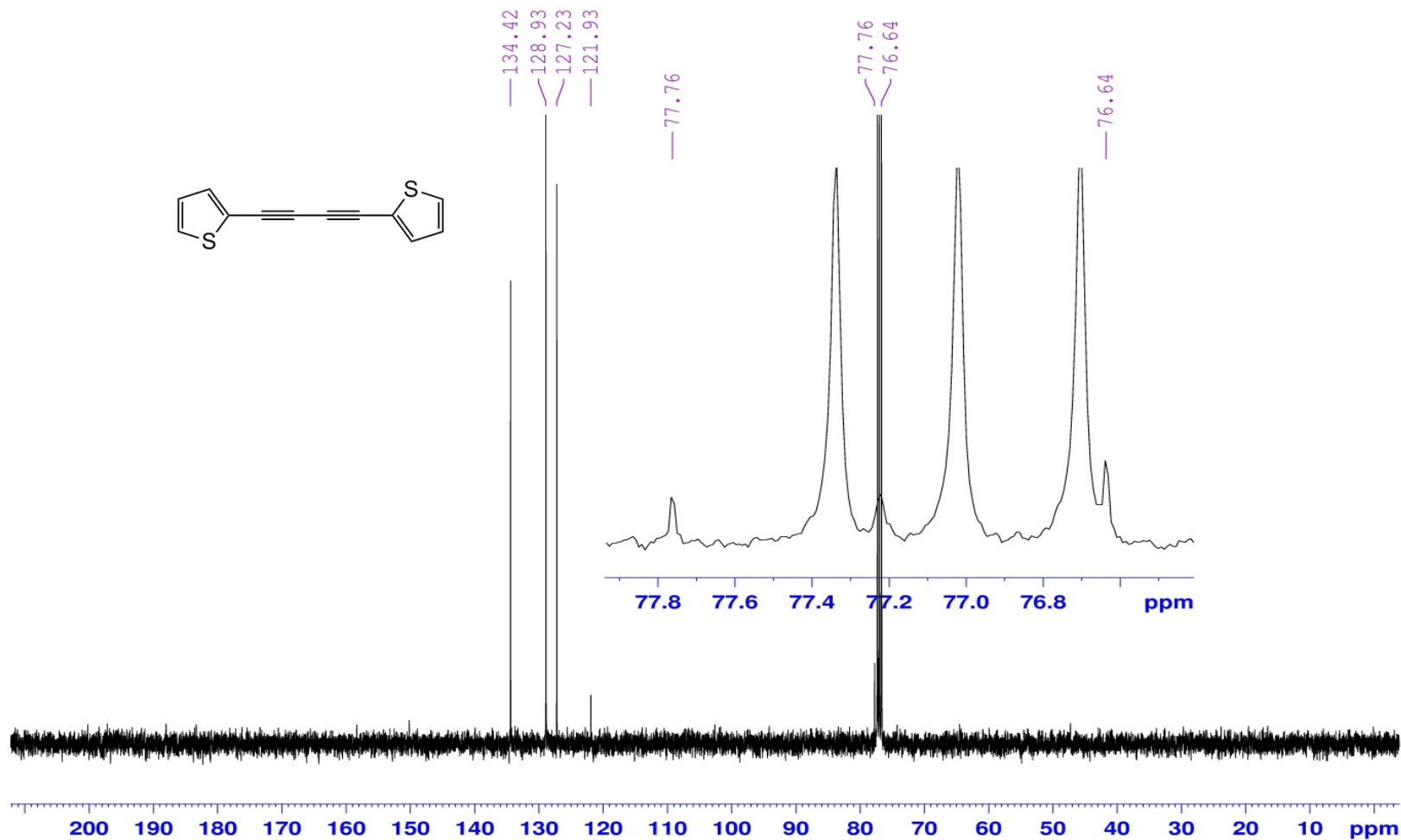
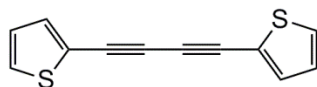
NAME      Name: 2aFahung-Halbbol
PROCNO    111
F2ACQD    20110921
TIME      19:15
INSTRUM   spect
PROBHD    5 mm QNP1HPC
PULPROG   zgpg30
PC        40000
AQ        0.0015
RG        327.5
SF        400.146
FIDRES    0.000188
AQRES     0.000188
SFORES    0.000188
NUC1       13
NUC2       13
NUC3       13
NUC4       13
NUC5       13
NUC6       13
NUC7       13
NUC8       13
NUC9       13
NUC10      13
NUC11      13
NUC12      13
NUC13      13
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NUC93      13
NUC94      13
NUC95      13
NUC96      13
NUC97      13
NUC98      13
NUC99      13
NUC100     13

```

1,4-Di(thiophene-2-yl)buta-1,3-diyne (Table 2, 2k): ^1H NMR (400 MHz, CDCl_3)



1,4-Di(thiophene-2-yl)buta-1,3-diyne (Table 2, 2k): ¹³C NMR (100 MHz, CDCl₃)



Instrument SpectPi
NMR Spectrometer 400 MHz
Bruker, German

Type of experiment:
1H Decoupled 13C S

```

NAME      Maxx Kafahani-06
EXPNO     20
PROCNO    1
F2 -      201809 1
TIME      0.21
INSTRUM   spect
PROBHD    5 mm PABBO HB-
PULPROG   zgpg30
ID         25788
SOLVENT   CDCl3
NS         3800
DS         3800
SFO       20250.1305 H
FIDRES    0.270644 H
AQ         0.4488664 s
RG         3800
SM         19.8500 G
DE         6.30 G
TE         297.0 K
SI         1.00000000 s
D1         0.03000000 s
TD         1
  
```

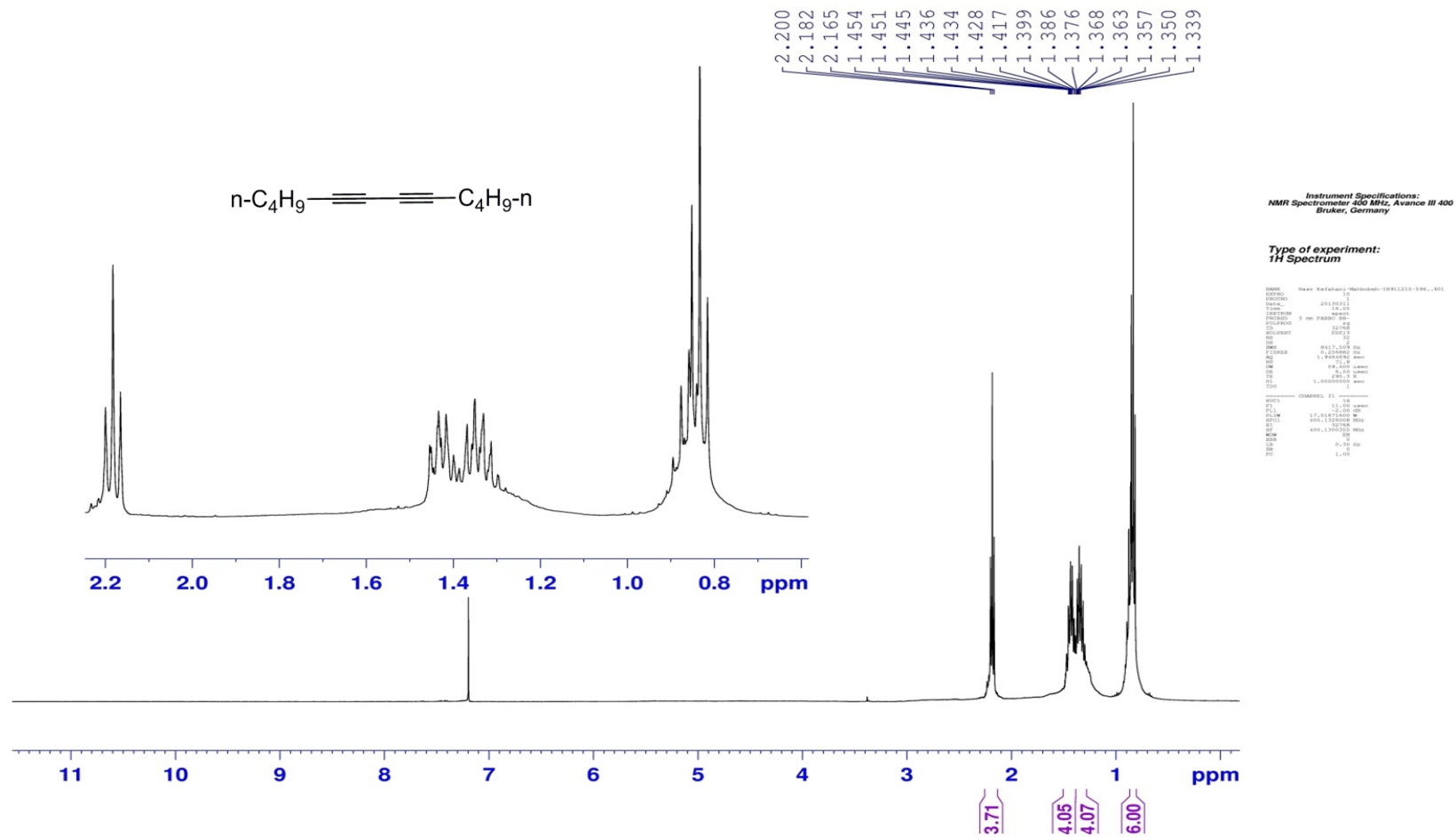
```

----- CHANNEL f1 -----
NUC1      13C
P1         8.70 G
P2         1.00 G
P1PW      42.69075012 W
SFO1      100.6283664 H
  
```

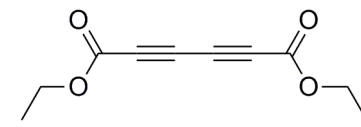
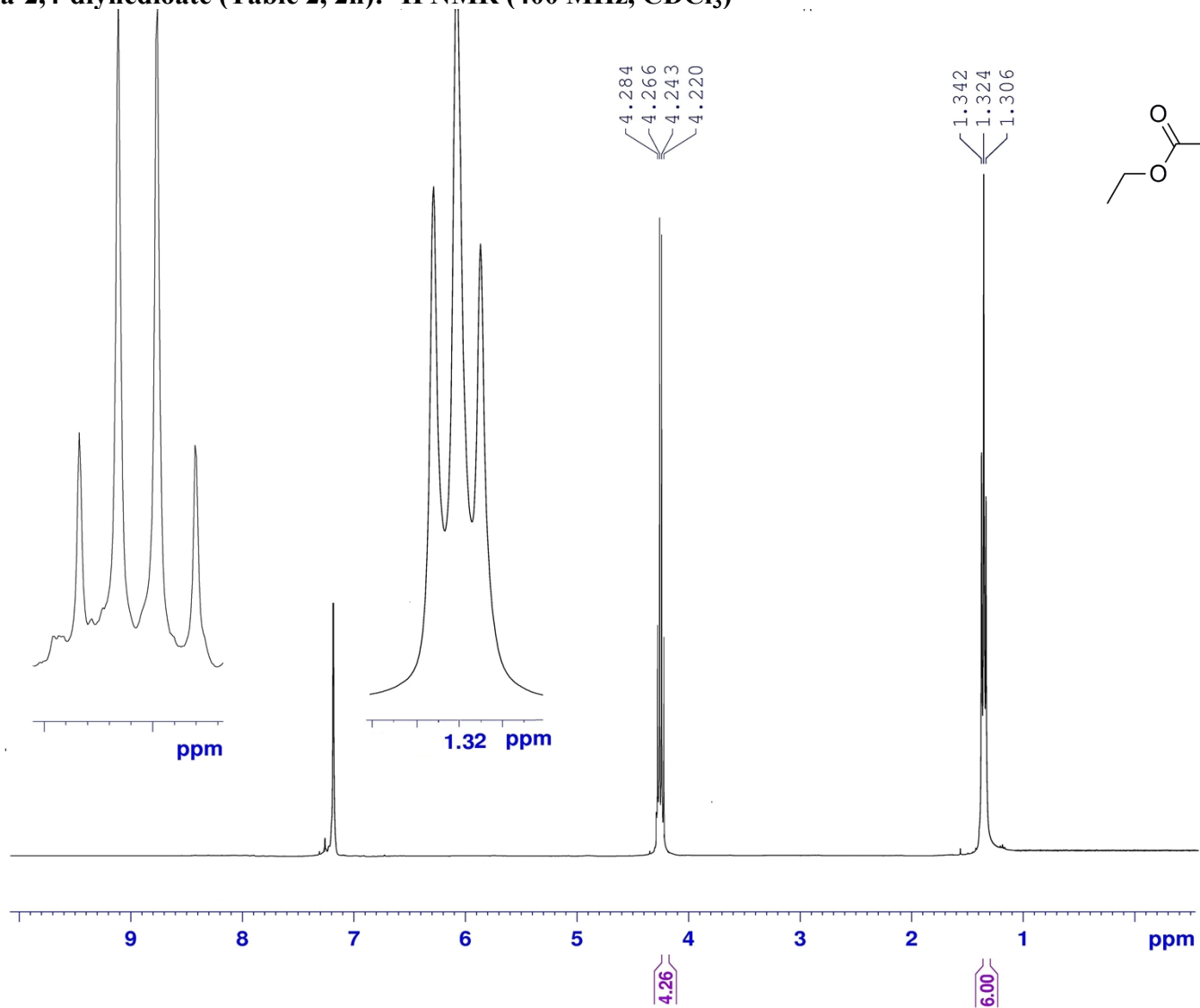
```

----- CHANNEL f2 -----
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 G
P12        0.00 G
P212       19.20 G
P1212     11.02330405 W
P1212M    0.12983629 W
P1212M    0.14698812 W
SFO2      400.1310000 H
SI         0.25788
TE         297.0 K
DE         6.30 G
SFO       100.6127600 H
NS         3800
DS         3800
SFO       1.40 G
PC         1.40
  
```


Dodeca-5,7-diyne (Table 2, 2m): ^1H NMR (400 MHz, CDCl_3)



Diethyl hexa-2,4-diynedioate (Table 2, 2n): ^1H NMR (400 MHz, CDCl_3)



Instrument Specifications:
NMR Spectrometer 400 MHz, Avance III 400
Bruker, Germany

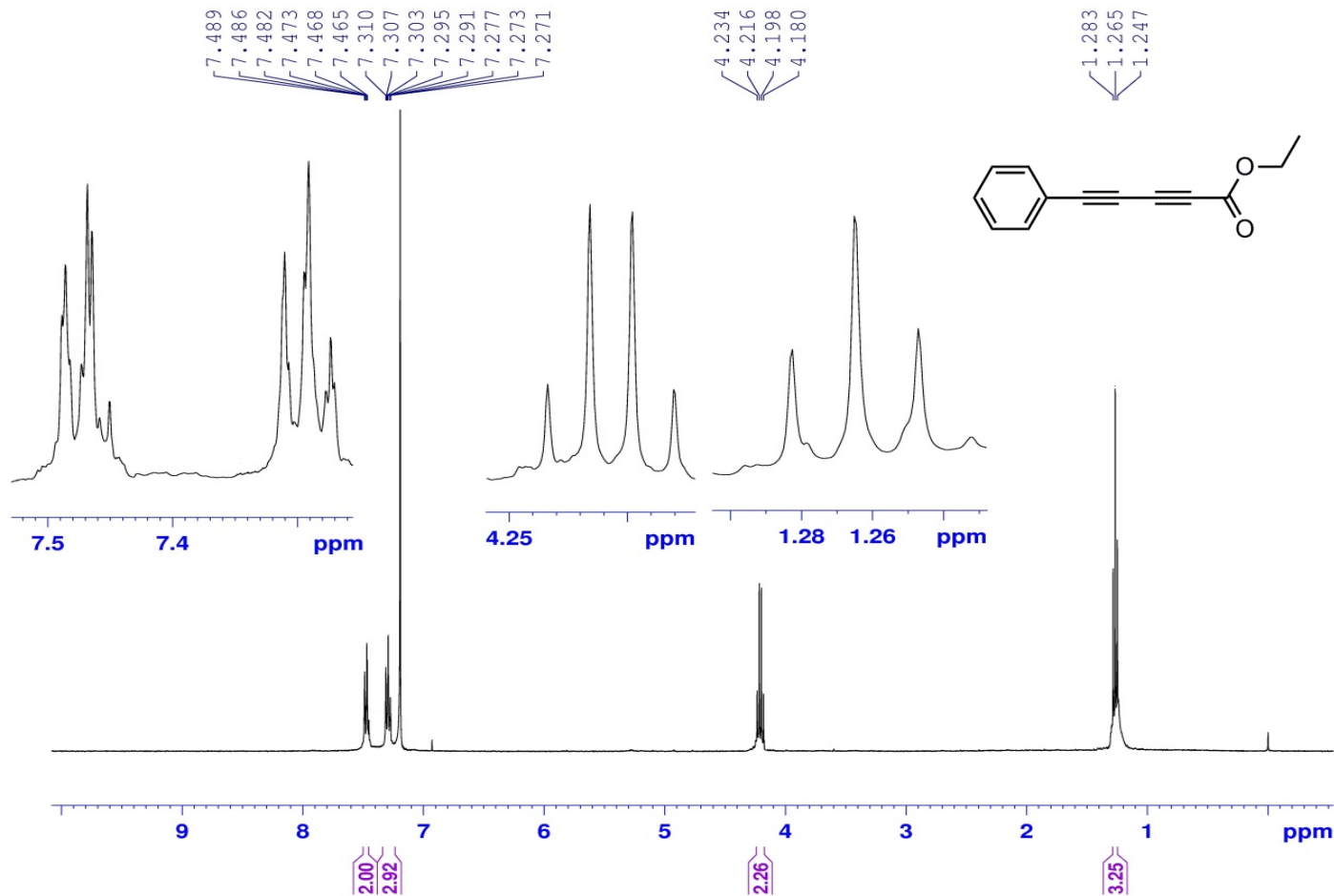
Type of experiment:
1H Spectrum

```

NAME      Hest Eafahael
EXPNO     1
PROCNO    1
DATE_     20130917
Time      12.00
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         32
DS         4
SHE        841.000 Hz
FIDRES    0.224892 Hz
AQ         1.9464652 sec
RG         114
DM         09.400 usec
DE         6.00 usec
TE         296.2 K
DQ         1.00000000 sec
DD         1
----- CHANNEL f1 -----
NUC1       1H
P1         11.00 usec
PL1        0
PL12       2.00 usec
PL122      0
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PL848      0
PL849      0
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PL875      0
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PL989      0
PL990      0
PL991      0
PL992      0
PL993      0
PL994      0
PL995      0
PL996      0
PL997      0
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PL999      0

```


Ethyl 5-phenylpenta-2,4-dienoate (Table 4, 3an): ¹H NMR (400 MHz, CDCl₃)



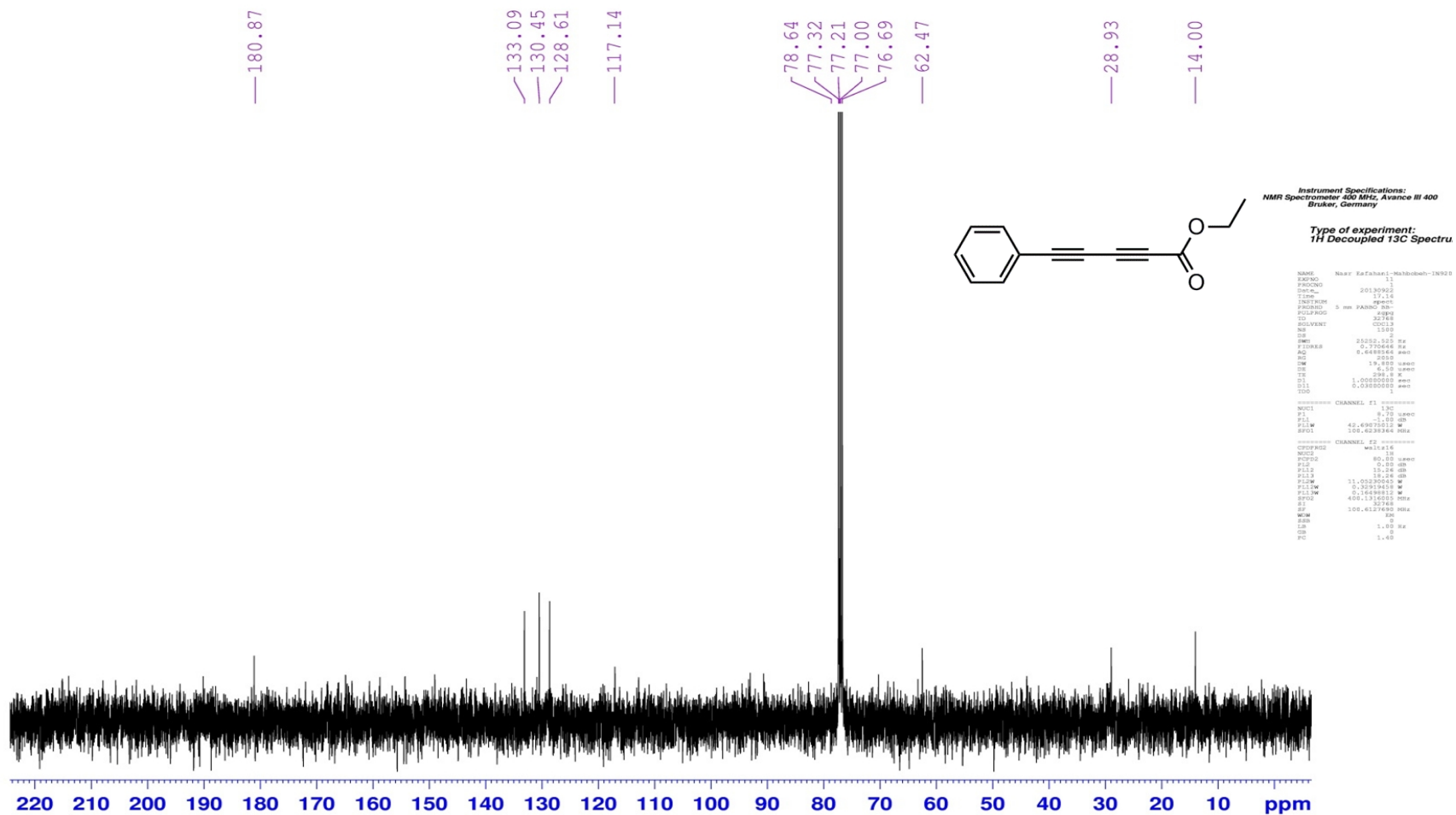
Instrument Specifications:
NMR Spectrometer 400 MHz, Avance III 400
Bruker, Germany

Type of experiment:
1H Spectrum

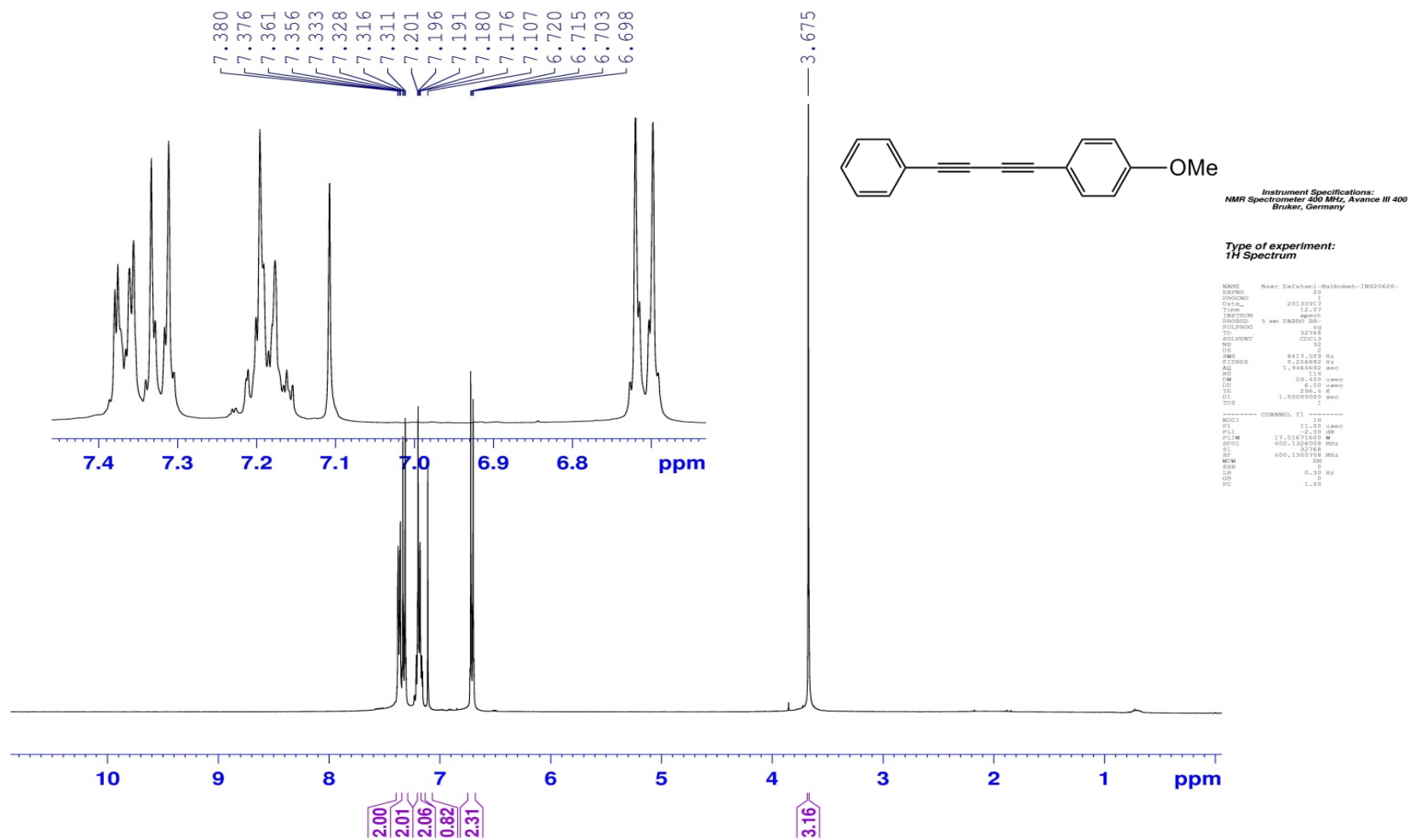
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NAME      Name: Kefaber-1 (Mahnobeh)-10020626-
EXPNO     1
PROCNO    1
Date_     20130913
TIME      12.10
INSTRUM   spect
PROBHD    5 mm PABBO AB-
PULPROG   zgpg30
TD        32768
SOLVENT   CDCl3
DS        2
SWH       8417.000 Hz
FIDRES    0.256892 Hz
AQ        1.244452 sec
RG         114
DM        59.400 umm
DE        6.00 umm
TE        298.15 K
D1        1.30000000 sec
TD0
----- CHANNEL f1 -----
NUC1      1H
P1        13.00 umm
PC1M      11.9167500 MHz
PC1S      400.1326000 MHz
SF        400.1326000 MHz
WDW       EM
SSB       0
MBM       0
SFO       0
GB        0.00 Hz
OB        0
PC        1.00
    
```

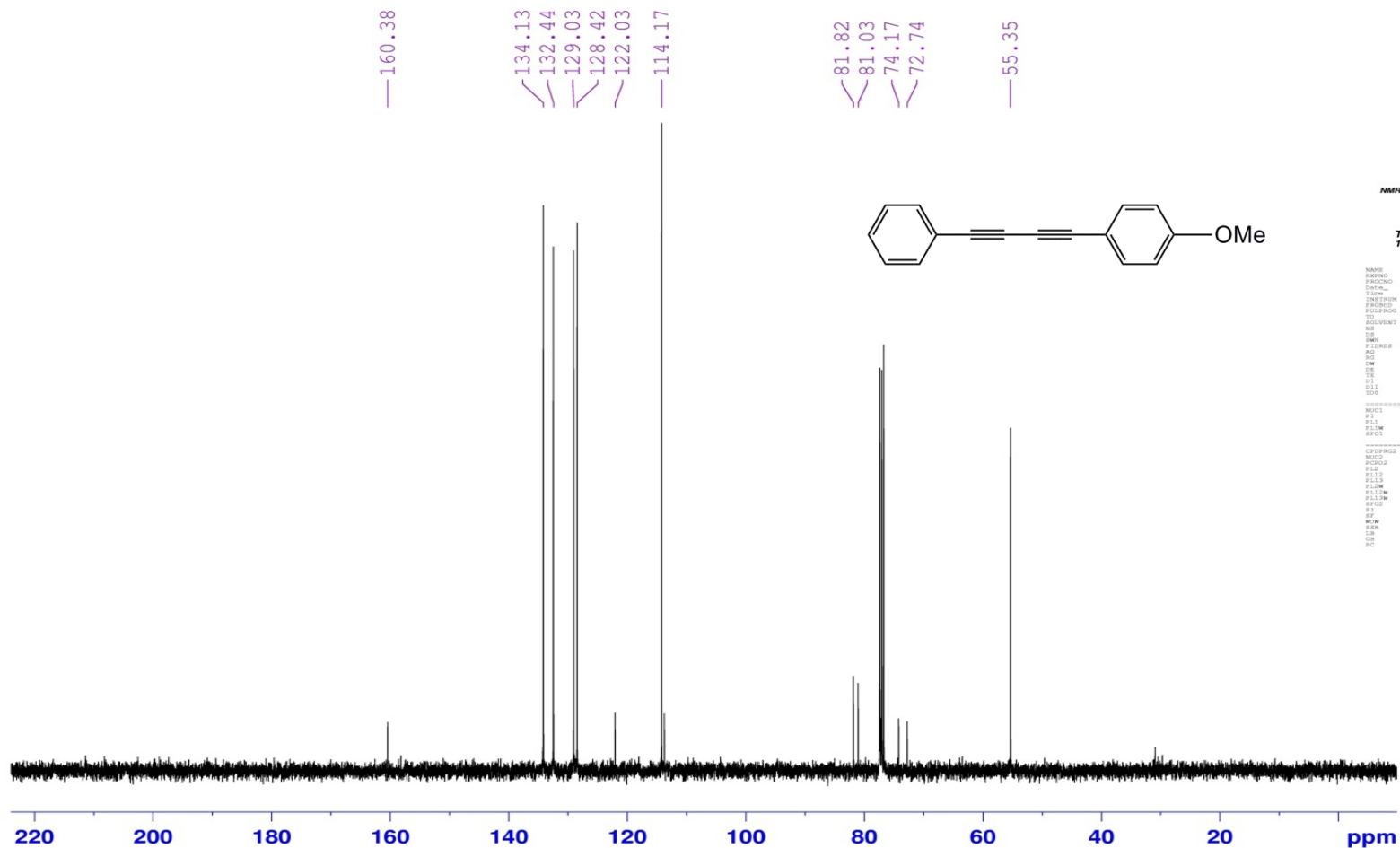
Ethyl 5-phenylpenta-2,4-dienoate (Table 4, 3an): ^{13}C NMR (100 MHz, CDCl_3)



1-Methoxy-4-(phenylbuta-1,3-diyne-1-yl)benzene (Table 4, 3ad): ¹H NMR (400 MHz, CDCl₃)



1-Methoxy-4-(phenylbuta-1,3-diy-1-yl)benzene (Table 4, 3ad): ^{13}C NMR (100 MHz, CDCl_3)



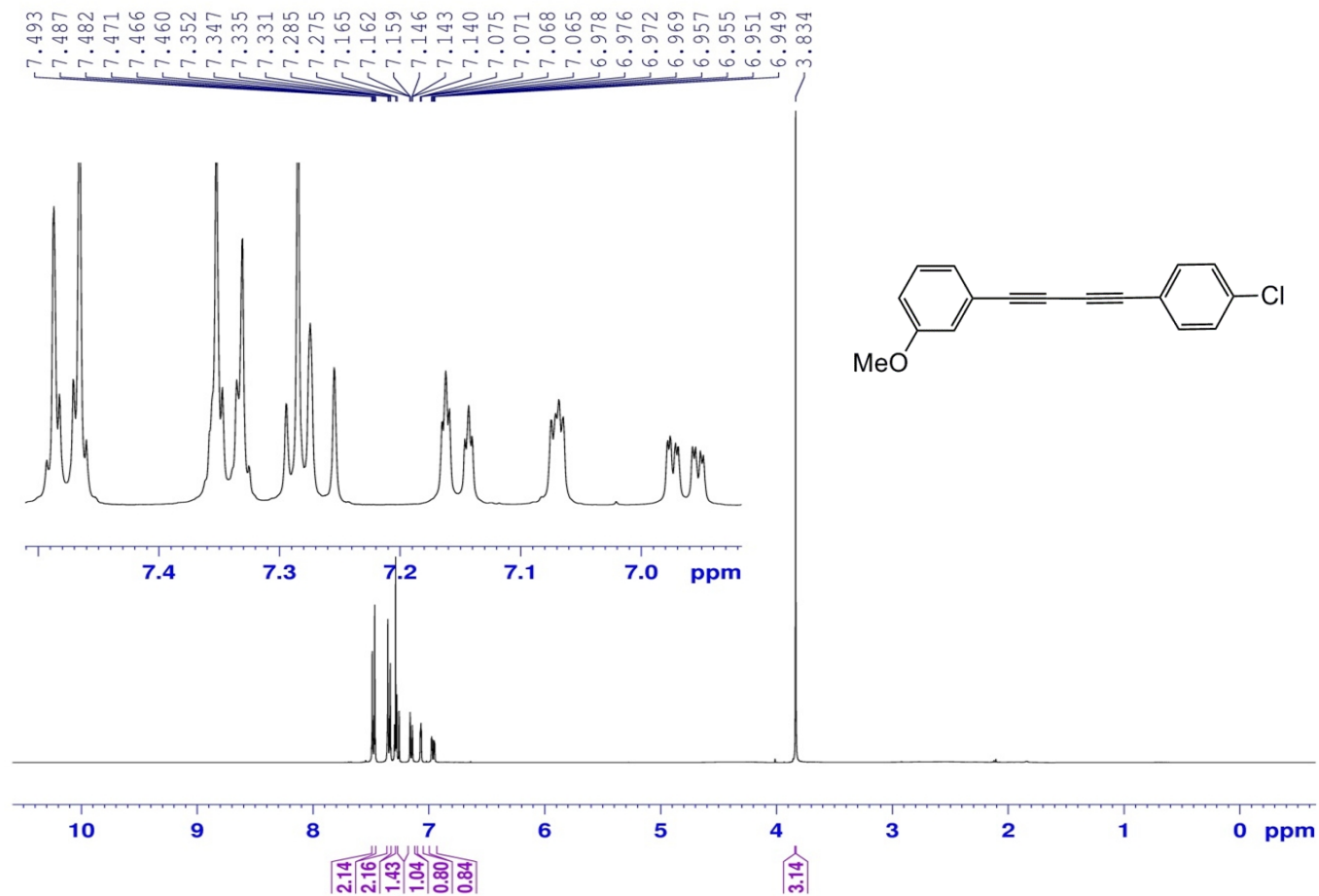
Instrument Specifications:
NMR Spectrometer 400 MHz, Avance II
Bruker, Germany

Type of experiment:
1H Decoupled 13C Spectru.

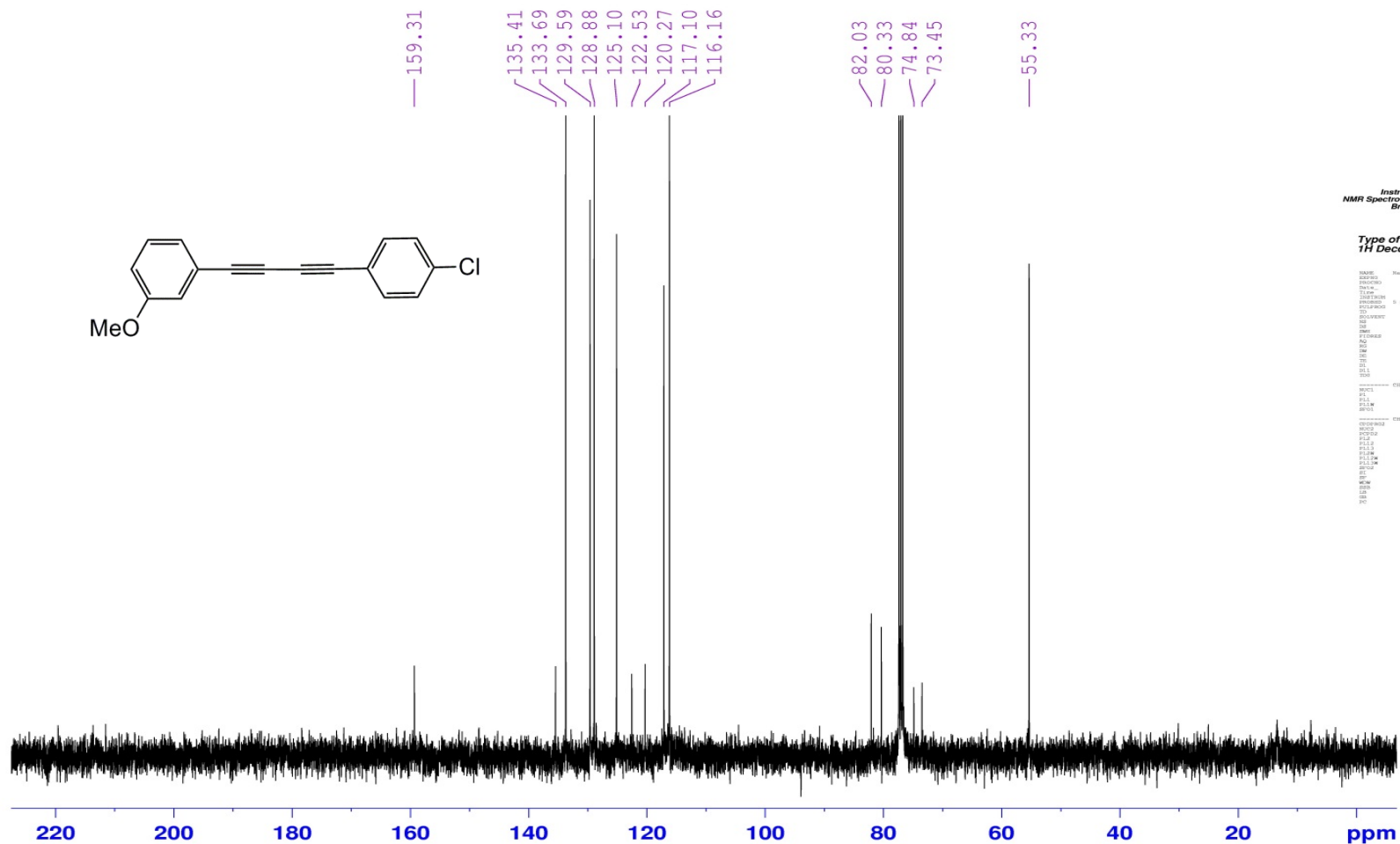
```

NAME      Name: ExFaharj_Mohibobeh-190701-
EXPNO     21
PROCNO    20199021
INSTRUM   spect
PROBHD    5 mm PABBO DQ-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         1360
DS         20202.025 Hz
FIDRES    0.77064 Hz
AQ         0.468864 sec
RG         600
DM         19.800 usec
DE         0.00 usec
TE         300.2 K
D1         1.00000000 sec
d11        0.00000000 sec
DDB
===== CHANNEL f1 =====
NUC1       13C
P1         1.00 usec
PL1        0.00 dB
PL12       0.00000000 W
SFO1       100.628364 MHz
===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2     80.00 usec
P2         10.00 dB
PL2        0.00 dB
PL12       0.00000000 W
PL13       11.00000000 W
PL14       0.00000000 W
PL15       0.00000000 W
PL16       0.00000000 W
SFO2       400.1314000 MHz
SS         0.00000000 sec
MW         100.4127800 MHz
SFO        0.00000000 MHz
DS         1.00 Hz
DB         1.40
PC         1.40
    
```

1-((4-Chlorophenyl)buta-1,3-diyn-1-yl)-3-methoxybenzene (Table 4, 3ci): ¹H NMR (400 MHz, CDCl₃)



1-((4-Chlorophenyl)buta-1,3-diyn-1-yl)-3-methoxybenzene (Table4, 3ci): ^{13}C NMR (100 MHz, CDCl_3)



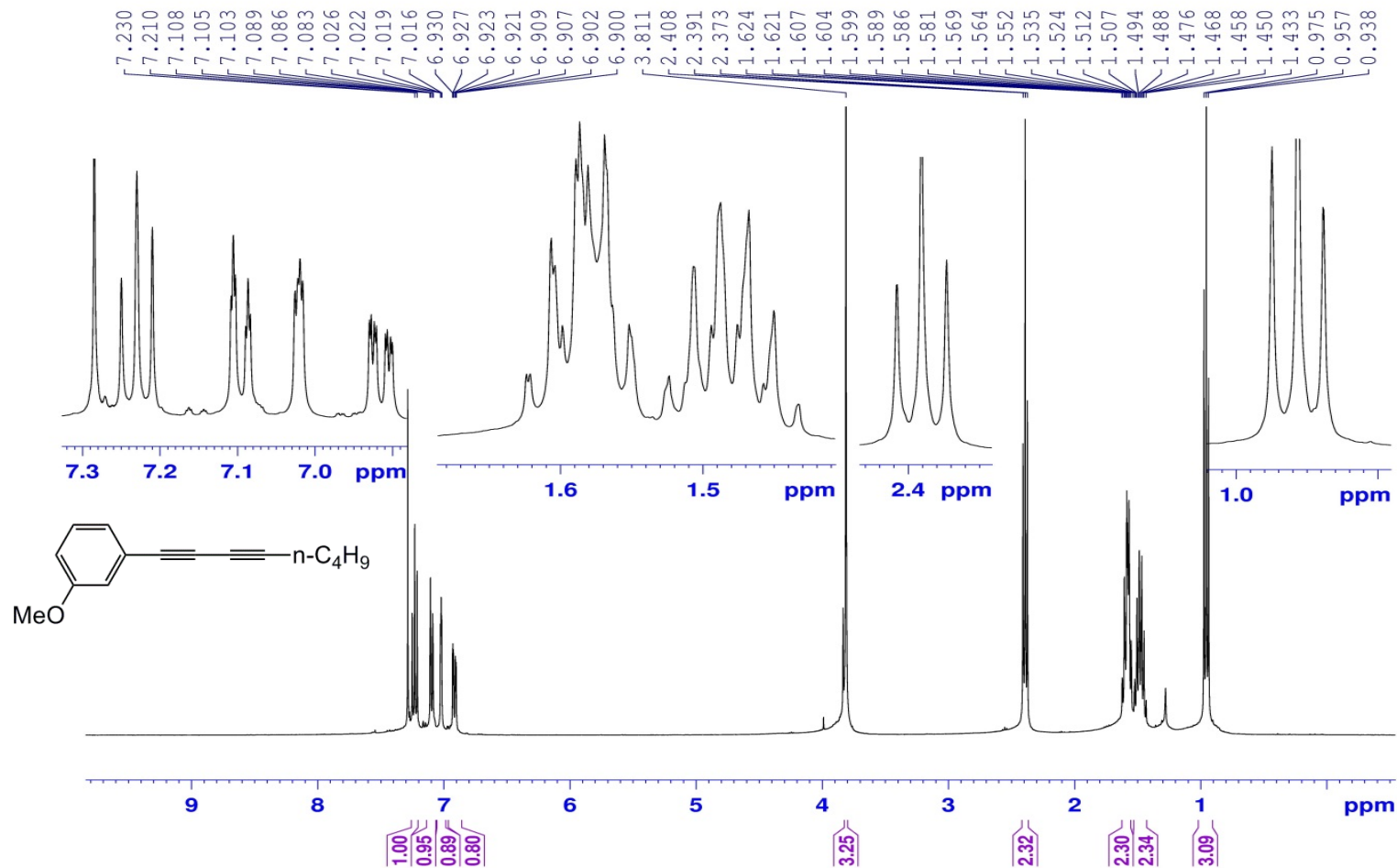
Instrument Specifications:
 NMR Spectrometer 400 MHz, Avance III 400
 Bruker, Germany

Type of experiment:
 1H Decoupled ^{13}C Spectrum

```

NAME      Name: Kefahari-Mabobeh-18920425-512_133
EXPNO    1
PROCNO   20130813
TIME     0.24
INSTRUM  spect
PROBHD   5 mm VAMBO
PULPROG  zgpg30
SOLVENT  CDCl3
AQ       15.00
RG       320
DS        4
SWH      20242.520 Hz
F2       0.717644 Hz
RG2      0.4480000 sec
AQ2      4.000
RG2      1.4000000 sec
DE       1.00
TE       300.2
D1       1.00000000 sec
D11      0.03000000 sec
TD       1
----- CHANNEL f1 -----
NUC1      13C
P1        8.00 sec
PL1W     40.0000000 dB
PL1      100.4236344 MHz
----- CHANNEL f2 -----
CPDPRG2  waltz16
NUC2      1H
PCPD2    80.00 sec
PL2      0.00 dB
PL2W     0.0000000 dB
PL2      100.6261260 MHz
PL12     11.05210463 Hz
PL12W    0.0000000 Hz
PL12     400.1418000 MHz
PCPD2    400.1418000 MHz
NUC3      13C
PCPD3    100.6174000 MHz
PL3      0.00 dB
PL3W     0.0000000 Hz
PL3      100.6174000 MHz
    
```

1-Methoxy-3-(octa-1,3-diyn-1-yl)benzene (Table 4, 3cm): ¹H NMR (400 MHz, CDCl₃)



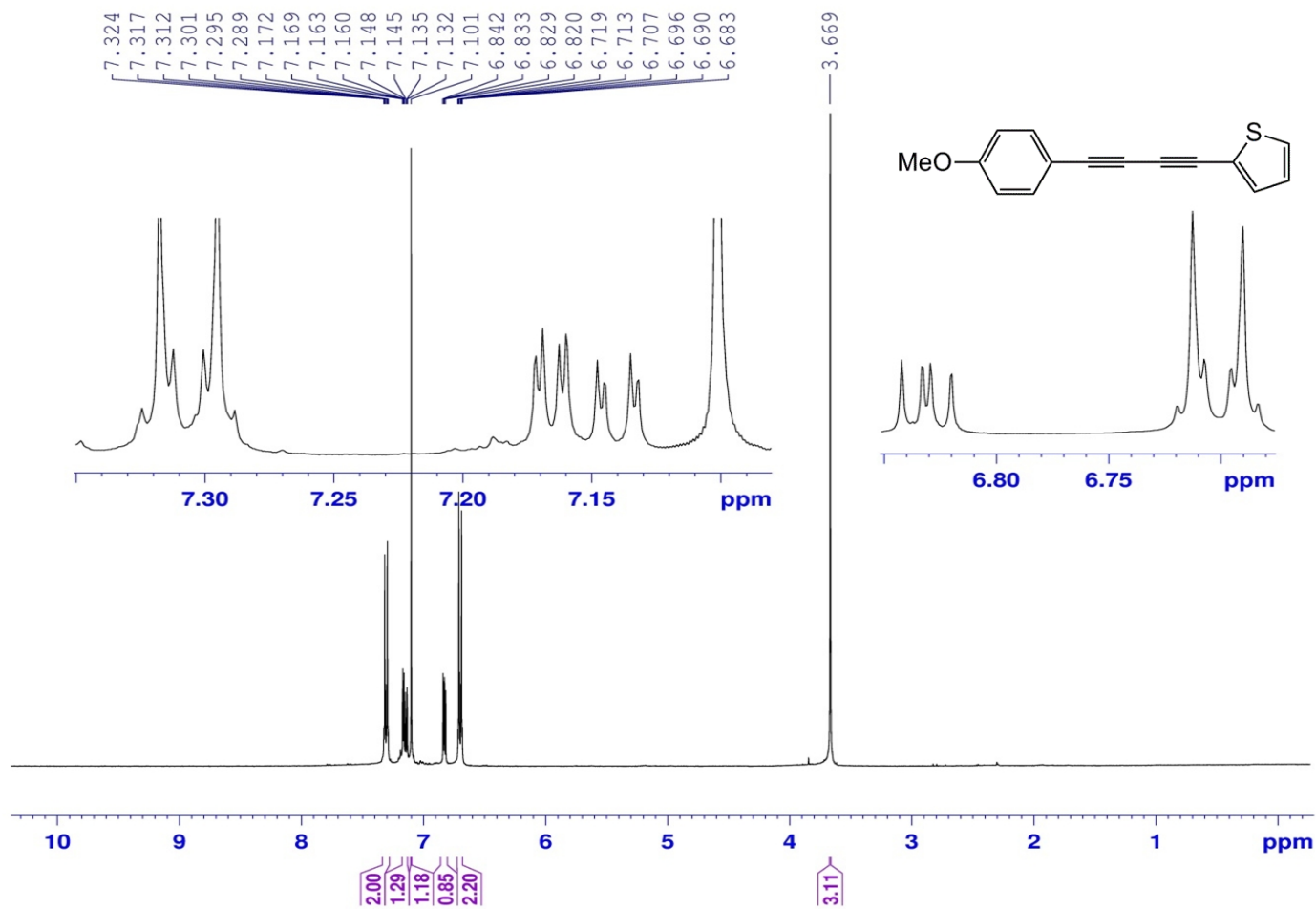
Instrument Specifications:
NMR Spectrometer 400 MHz, Avance III 400
Bruker, Germany

Type of experiment:
1H Spectrum

NAME	Desktop
EXPNO	170
PROCNO	1
Date_	20130804
Time	13:36
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	32
DS	2
SWH	8417.509 Hz
FIDRES	0.256882 Hz
AQ	1.946492 sec
RG	101
DW	59.400 usec
DE	6.50 usec
TE	297.5 K
D1	1.0000000 sec
TD0	1

-----	CHANNEL f1	-----
NUC1	1H	
P1	11.00 usec	
PL1	-2.00 dB	
PL1W	17.51671400 W	
SFO1	400.1324008 MHz	
SI	32768	
SF	400.1300000 MHz	
WDW	EM	
SSB	0	
LB	0.30 Hz	
GB	0	
PC	1.00	

2-((4-Methoxyphenyl)buta-1,3-diyn-1-yl)thiophene (Table 4, 3dk): ¹H NMR (400 MHz, CDCl₃)



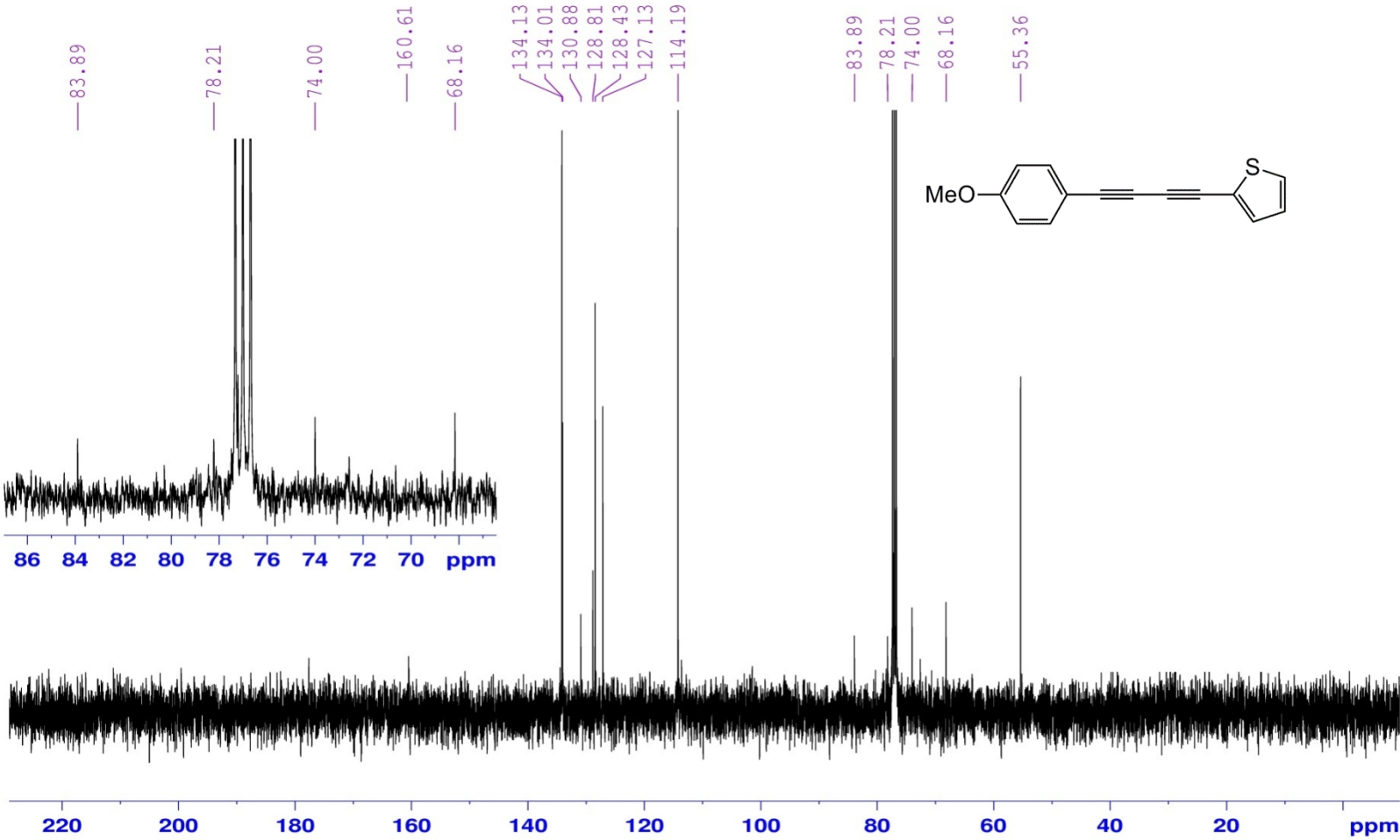
Instrument Specifications:
NMR Spectrometer 400 MHz, Avance III 400
Bruker, Germany

Type of experiment:
1H Spectrum

```

NAME      Meer-eufonia1-Mahabeh-190701-01_10
EXPNO    2
PROCNO   2
DATE_    20130703
TIME     22:44
PROBHD   5 mm PABBO 50
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        2048
DS        4
SWH       8171.000 Hz
F2       101.625000 MHz
AQ        0.199999999
RG         655.312
WDW       EM
SS        0.000000000
LB        0.300000000
GB        0
PC        1.000000000
===== CHANNEL f1 =====
NUC1      13
P1         12.00
NUC2      1H
P2         12.000000000 MHz
=====
  
```

2-((4-Methoxyphenyl)buta-1,3-diyn-1-yl)thiophene (Table 4, 3dk): ¹³C NMR (100 MHz, CDCl₃)



Instrument Specific
NMR Spectrometer 400 MHz
Bruker, German

Type of experiment:
1H Decoupled 13C S

```

NAME      Hase-wefahaj-13dkb06
EXPNO    241
PROCNO   20134924
TIME     24.58
INSTRUM  spect
PROBHD   5 mm BBOBO-1H
PULPROG  zgpg30
TD        32768
SOLVENT  CDCl3
NS       1800
DS       4
SWH      25297.325 Hz
FIDRES   0.773444 Hz
AQ        0.488558 sec
RG        400
DM        19.800 uspec
DC        25.90 uspec
DE        248.3 Hz
DIL       1.00000000 sec
SOL1     0.00000000 sec
TSD      1
----- CHANNEL f1 -----
NUC1      13C
P1        12.00 uspec
PL1       -1.00 dB
SFO1      100.6281624 MHz
----- CHANNEL f2 -----
NUC2      1H
P2        80.00 uspec
PL2       0.00 dB
PL12      19.24 dB
SFO2      400.1464018 MHz
PULPROG  zgpg30
PC        0.22914688 sec
PCPRG2   0.11193918 sec
SFO3      100.6187500 MHz
WDW       EM
SSB       0
MVM       0
MDE      1.00 uspec
WDW       EM
SSB       0
MVM       0

```

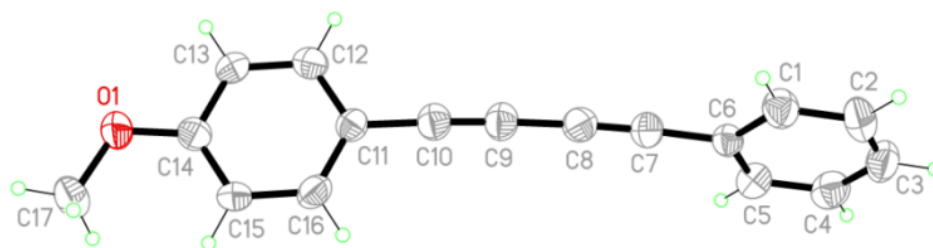


Figure 1 X-ray crystal structure of compound **3ad**. Thermal ellipsoids are drawn at the 30% probability level, while the hydrogen size is arbitrary.

Table 1 Crystal data and structure refinement for Compound **3ad**.

Empirical formula	C ₁₇ H ₁₂ O	
Temperature (K)	293(2)	
Formula weight	232.27	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 13.846(2) Å	α = 90°
	b = 9.3560(19) Å	β = 101.896(11)°
	c = 10.2300(15) Å	γ = 90°
Volume	1296.8(4) Å ³	
Z	4	
Density (calculated)	1.190 Mg/m ³	
Absorption coefficient	0.073 mm ⁻¹	
F(000)	488	
Theta range for data collection	2.65 to 25.00°	
Index ranges	-16 ≤ h ≤ 16, -11 ≤ k ≤ 11, -12 ≤ l ≤ 11	
Reflections collected	14290	
Independent reflections	2284 [R(int) = 0.1138]	
Completeness to theta = 25.00	100.0%	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	2284 / 0 / 165	
Goodness-of-fit on F ²	0.924	
Final R indices [I > 2σ(I)]	R1 = 0.0520, wR2 = 0.0806	
R indices (all data)	R1 = 0.1991, wR2 = 0.1092	
Largest diff. peak and hole	0.128 and -0.201 e.Å ³	

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