

ELECTRONIC SUPPORTING INFORMATION

γ -Valerolactone as an alternative biomass-derived medium for the Sonogashira reaction

Giacomo Strappaveccia,^[a] Lorenzo Luciani,^[a] Elena Bartollini,^[a] Assunta Marrocchi,^[a] Ferdinando Pizzo,^[a] Luigi Vaccaro*^[a]

^[a] Laboratory of Green Synthetic Organic Chemistry, CEMIN – Dipartimento di Chimica, Università di Perugia Via Elce di Sotto 8, 06123 Perugia, Italia, E-mail: luigi.vaccaro@unipg.it

CONTENTS

page ESI 1 Experimental Section

page ESI 2-ESI 21 Full characterization data (¹H NMR, ¹³C NMR, ¹⁹F NMR, EA, GC-EIMS) for Compounds **3 a-q**

page ESI 23-ESI 53 Copies of the ¹H, ¹³C NMR and ¹⁹F NMR for compounds **3 a-q**

Experimental Section

Unless otherwise stated, all solvents and reagents were used as obtained from Sigma-Aldrich Co. without further purification. Commercial Pd/C (10% wt) was not activated prior to use, and the value of palladium loading was taken from its certificate of analysis available at <http://www.sigmaaldrich.com>. GLC analyses were performed by using Hewlett-Packard HP 5890A equipped with a capillary column DB-35MS (30 m, 0.53 mm), a FID detector and hydrogen as gas carrier. GC-EIMS analyses were carried out by using a Hewlett-Packard HP 6890N Network GC system/5975 Mass Selective Detector equipped with an electron impact ionizer at 70 eV. NMR spectra were recorded on a Bruker DRX-ADVANCE 400 MHz (^1H at 400 MHz, ^{13}C at 100.6 MHz and ^{19}F at 376.4 MHz) in CDCl_3 using TMS as the internal standard. Elemental Analyses were conducted on a Fisons EA1108CHN. Melting points are not corrected and they were measured on a Büchi 510. An Inductive Coupled Plasma-Optical Emission Spectrometer (ICP-OES 710 Agilent Technology) was used to determine the amount of leached palladium into the reaction products.

Compounds **3a**,¹ **3b**,¹ **3c**,¹ **3d**,² **3e**,¹ **3f**,¹ **3g**,³ **3h**,⁴ **3i**,⁴ **3j**,⁴ **3k**,⁵ **3l**,⁴ **3m**,⁵ **3n**,⁶ **3o**,⁶ **3p**,⁶ **3q**⁷ are known compounds.

Characterization data and copies of the ^1H and ^{13}C NMR are reported below.

Reference:

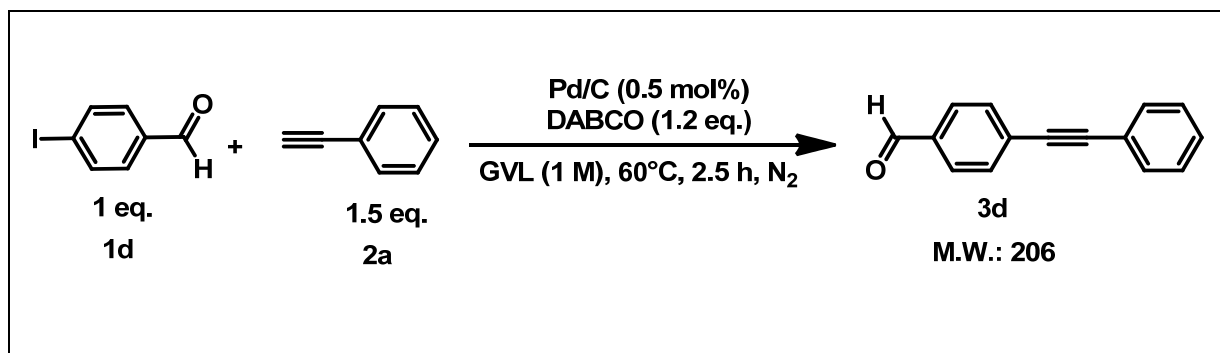
- (1) A. Modak, J. Mondal, A. Bhaumik, *Green Chem.* **2012**, *14*, 2840–2855
- (2) K. Worm-Leonhard, M. Meldal, *Eur. J. Org. Chem.*, **2008**, *31*, 5244-5253
- (3) R. Thorwirth, A. Stolle, B. Ondruschka, *Green Chem.*, **2010**, *12*, 985-991
- (4) S. Mori, T. Yanase, S. Aoyagi, Y. Monguchi, T. Maegawa, H. Sajiki, *Chem. Eur. J.*, **2008**, *14*, 6994-6999
- (5) Y. Shi, X. Li, J. Liu, W. Jiang, L. Sun, *Appl. Organometal. Chem.*, **2011**, *25*, 514-520
- (6) S. Urgaonkar, J. G. Verkade, *J. Org. Chem.*, **2004**, *69*, 5752-5755
- (7) L. Yang, Y. Li, Q. Chen, Y. Du, C. Cao, Y. Shi, G. Pang, *Tetrahedron*, **2013**, *69*, 5178-5184
- (8) K. Takahashi, Y. Kuroyama, K. Sonogashira, N. Hagihara, *Synthesis*, **1980**, 627-630

Chem. Name	1,2-diphenylethyne (3a)			
Lit. Ref.	<i>Green Chem.</i> , 2012 , <i>14</i> , 2840–2855			
<p style="text-align: center;"> $\text{I-C}_6\text{H}_5$ (1 eq. 1a) + $\text{C}_6\text{H}_5\text{-C}\equiv\text{C-H}$ (1.5 eq. 2a) $\xrightarrow[\text{GVL (1 M), 60 }^\circ\text{C, 4 h, N}_2]{\text{Pd/C (0.5 mol\%), DABCO (1.2 eq.)}}$ $\text{C}_6\text{H}_5\text{-C}\equiv\text{C-C}_6\text{H}_5$ (3a) M.W.: 178 </p>				
METHOD:				
<p>In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.005 mmol), GVL (1 mL, 98% purity), DABCO (139 mg, 1.2 mmol, 99% purity), iodobenzene (1a) (139 mg, 0.114 mL, 1 mmol, 98% purity), and ethynylbenzene (2a) (156 mg, 0.168 mL, 1.5 mmol, 98% purity) were consecutively added, the resulting mixture was purged with nitrogen and left under stirring at 60 °C. After 4 hours, petroleum ether was added and the reaction mixture was filtered over a small pad of celite, washed with neutral water and, subsequently, with acidic water (1 M HCl). The organic layer was dried over sodium sulphate and the solvent was removed under vacuum. The crude oil was purified by column chromatography on silica gel (petroleum ether) to afford 3a as a white solid (124 mg, 70% yield).</p>				
Mol Formula	C ₁₄ H ₁₀	m.p.	61-63 °C	
Elemental Analysis: Calc.: C: 94.34; H: 5.66; found C: 94.24; H: 5.60				
¹H NMR 400 MHz CDCl₃	δ value	No. H	Mult.	j value/Hz
	7.34-7.36	6	<i>m</i>	
	7.53-7.55	4	<i>m</i>	
¹³C NMR (100.6 MHz, CDCl₃) δ : 89.37; 123.28; 128.25; 128.34; 131.61				
GC-EIMS (m/z, %): 178 (M ⁺ , 100), 177 (34), 176 (50), 152 (29), 151 (21), 150 (15), 126 (12), 89 (15)				

Chem. Name	1-nitro-4-(phenylethynyl)benzene (3b)			
Lit. Ref.	<i>Green Chem.</i> , 2012 , <i>14</i> , 2840–2855			
<p style="text-align: center;"> $\text{I-C}_6\text{H}_4\text{-NO}_2$ (1 eq. 1b) + $\text{C}_6\text{H}_5\text{-C}\equiv\text{C-H}$ (1.5 eq. 2a) $\xrightarrow[\text{GVL (1 M), 60 }^\circ\text{C, 2 h, N}_2]{\text{Pd/C (0.5 mol\%), DABCO (1.2 eq.)}}$ $\text{O}_2\text{N-C}_6\text{H}_4\text{-C}\equiv\text{C-C}_6\text{H}_5$ (3b) M.W.: 223 </p>				
METHOD:				
<p>In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.005 mmol), GVL (1 mL, 98% purity), DABCO (139 mg, 1.2 mmol, 99% purity), 1-iodo-4-nitrobenzene (1b) (254 mg, 1 mmol, 98% purity), and ethynylbenzene (2a) (156 mg, 0.168 mL, 1.5 mmol, 98% purity) were consecutively added, the resulting mixture was purged with nitrogen and left under stirring at 60 °C. After 2 hours, petroleum ether was added and the reaction mixture was filtered over a small pad of celite, washed with neutral water and, subsequently, with acidic water (1 M HCl). The organic layer was dried over sodium sulphate and the solvent was removed under vacuum. The crude oil was purified by column chromatography on silica gel (1:1 / toluene / petroleum ether) to afford 3b as a white solid (210 mg, 94% yield).</p>				
Mol Formula	$\text{C}_{14}\text{H}_9\text{NO}_2$	m.p.	116-118 °C	
Elemental Analysis: Calc.: C: 75.33; H: 4.06; N:6.27; found C: 75.10; H: 4.99; N: 6.20				
¹H NMR 400 MHz CDCl₃	δ value	No. H	Mult.	j value/Hz
	7.39-7.41	3	<i>m</i>	
	7.55-7.58	2	<i>m</i>	
	7.67	2	<i>d</i>	8.7
	8.23	2	<i>d</i>	8.7
¹³C NMR (100.6 MHz, CDCl₃) δ : 87.6, 94.7, 122.1, 123.6, 128.6, 129.3, 130.3, 131.9, 132.3, 147.0				
GC-EIMS (m/z, %): 223 (M ⁺ , 100), 193 (29), 176 (60), 165 (27), 177 (19), 151 (20), 150 (16)				

Chem. Name	1-(4-(phenylethynyl)phenyl)ethanone (3c)			
Lit. Ref.	<i>Green Chem.</i> , 2012 , 14, 2840–2855			
<p>1 eq. 1c + 1.5 eq. 2a $\xrightarrow[\text{GVL (1 M), 60^\circ\text{C}, 2.5 h, N_2}]{\text{Pd/C (0.5 mol\%), DABCO (1.2 eq.)}}$ 3c M.W.: 220</p>				
METHOD:				
<p>In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.005 mmol), GVL (1 mL, 98% purity), DABCO (139 mg, 1.2 mmol, 99% purity), 1-(4-iodophenyl)ethanone (1c) (251 mg, 1 mmol, 98% purity), and ethynylbenzene (2a) (156 mg, 0.168 mL, 1.5 mmol, 98% purity) were consecutively added, the resulting mixture was purged with nitrogen and left under stirring at 60 °C. After 2.5 hours, petroleum ether was added and the reaction mixture was filtered over a small pad of celite, washed with neutral water and, subsequently, with acidic water (1 M HCl). The organic layer was dried over sodium sulphate and the solvent was removed under vacuum. The crude oil was purified by column chromatography on silica gel (1:1 / toluene : petroleum ether) to afford 3c as a white solid (209 mg, 95% yield).</p>				
Mol Formula	C ₁₆ H ₁₂ O	m.p.	98-101 °C	
Elemental Analysis: Calc.: C, 87.25; H, 5.49; found C: 87.00; H: 5.40				
¹H NMR 400 MHz CDCl ₃	δ value	No. H	Mult.	j value/Hz
	2.63	3	<i>s</i>	
	7.37-7.38	3	<i>m</i>	
	7.55-7.57	2	<i>m</i>	
	7.61	2	<i>d</i>	8.3
	7.94	2	<i>d</i>	8.3
¹³C NMR (100.6 MHz, CDCl₃) δ : 26.6, 88.6, 92.7, 122.6, 128.2, 128.3, 128.4, 128.8, 131.69, 131.73, 136.2, 197.3				
GC-EIMS (m/z, %): 220 (M ⁺ , 61), 205 (100), 177 (24), 176 (55), 151 (21), 150 (17), 88 (10), 43 (10)				

Chem. Name	4-(phenylethynyl)benzaldehyde (3d)
Lit. Ref.	<i>Eur. J. Org. Chem.</i> , 2008 , 31, 5244-5253



METHOD:

In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.005 mmol), GVL (1 mL, 98% purity), DABCO (139 mg, 1.2 mmol, 99% purity), 4-iodobenzaldehyde (**1d**) (234 mg, 1 mmol, 96% purity), and ethynylbenzene (**2a**) (156 mg, 0.168 mL, 1.5 mmol, 98% purity) were consecutively added, the resulting mixture was purged with nitrogen and left under stirring at 60 °C. After 2.5 hours, petroleum ether was added and the reaction mixture was filtered over a small pad of celite, washed with neutral water and, subsequently, with acidic water (1 M HCl). The organic layer was dried over sodium sulphate and the solvent was removed under vacuum. The crude oil was purified by column chromatography on silica gel (1:1 / toluene : petroleum ether) to afford **3d** as a white solid (186 mg, 90% yield).

Mol Formula	C ₁₅ H ₁₀ O	m.p.	95-97 °C
--------------------	-----------------------------------	-------------	----------

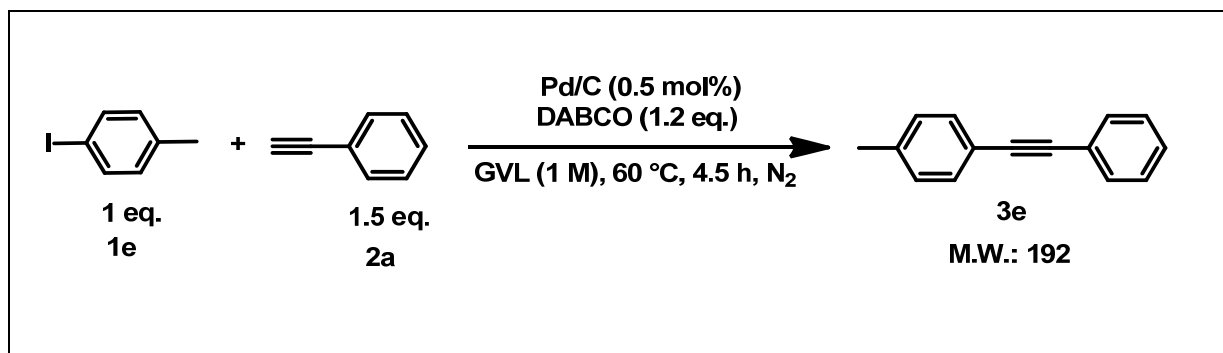
Elemental Analysis: Calc.: C, 87.36; H, 4.89; found C: 87.10; H:4.80

	δ value	No. H	Mult.	j value/Hz
¹H NMR 400 MHz CDCl ₃	7.38	3	<i>br</i>	
	7.56	2	<i>br</i>	
	7.68	2	<i>d</i>	7.8
	7.87	2	<i>d</i>	7.8
	10.02	1	<i>s</i>	

¹³C NMR (100.6 MHz, CDCl₃) δ : 88.5, 93.5, 122.5, 128.5, 129.0, 129.6, 131.8, 132.1, 135.4, 191.5

GC-EIMS (m/z, %): 206 (M⁺, 100), 205 (71), 178 (14), 177 (20), 176 (51), 151 (20), 150 (17)

Chem. Name	1-methyl-4-(phenylethynyl)benzene (3e)
Lit. Ref.	<i>Green Chem.</i> , 2012 , <i>14</i> , 2840–2855



METHOD:

In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.005 mmol), GVL (1 mL, 98% purity), DABCO (139 mg, 1.2 mmol, 99% purity), 1-iodo-4-methylbenzene (**1e**) (220 mg, 1 mmol, 99% purity), and ethynylbenzene (**2a**) (156 mg, 0.168 mL, 1.5 mmol, 98% purity) were consecutively added, the resulting mixture was purged with nitrogen and left under stirring at 60 °C. After 4.5 hours, petroleum ether was added and the reaction mixture was filtered over a small pad of celite, washed with neutral water and, subsequently, with acidic water (1 M HCl). The organic layer was dried over sodium sulphate and the solvent was removed under vacuum. The crude oil was purified by column chromatography on silica gel (petroleum ether) to afford **3e** as a white solid (154 mg, 80% yield).

Mol Formula	C ₁₅ H ₁₂	m.p.	67-70 °C
--------------------	---------------------------------	-------------	----------

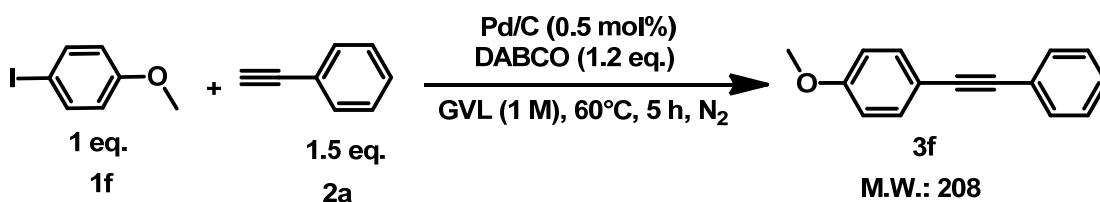
Elemental Analysis: Calc.: C: 93.71; H: 6.29; found C: 93.63; H: 6.19

	δ value	No. H	Mult.	j value/Hz
¹H NMR 400 MHz CDCl₃	2.37	3	s	
	7.16	2	d	7.8
	7.33-7.36	3	m	
	7.43	2	d	7.9
	7.52-7.54	2	m	

¹³C NMR (100.6 MHz, CDCl₃) δ : 21.5, 88.7, 89.6, 120.2, 123.5, 128.1, 128.3, 129.1, 131.5, 131.6, 138.4

GC-EIMS (m/z, %): 192 (M⁺, 100), 191 (100), 190 (32), 189 (47), 165 (27), 115 (10), 94 (11), 82 (10)

Chem. Name	1-methoxy-4-(phenylethynyl)benzene (3f)
Lit. Ref.	<i>Green Chem.</i> , 2012 , <i>14</i> , 2840–2855



METHOD: In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.005 mmol), GVL (1 mL, 98% purity), DABCO (139 mg, 1.2 mmol, 99% purity), 1-iodo-4-methoxybenzene (**1f**) (238 mg, 1 mmol, 98% purity), and ethynylbenzene (**2a**) (156 mg, 0.168 mL, 1.5 mmol, 98% purity) were consecutively added, the resulting mixture was purged with nitrogen and left under stirring at 60 °C. After 5 hours, petroleum ether was added and the reaction mixture was filtered over a small pad of celite, washed with neutral water and, subsequently, with acidic water (1 M HCl). The organic layer was dried over sodium sulphate and the solvent was removed under vacuum. The crude oil was purified by column chromatography on silica gel (1:1 / toluene : petroleum ether) to afford **3f** as a white solid (156 mg, 75% yield).

Mol Formula	C ₁₅ H ₁₂ O	m.p.	58-61 °C
--------------------	-----------------------------------	-------------	----------

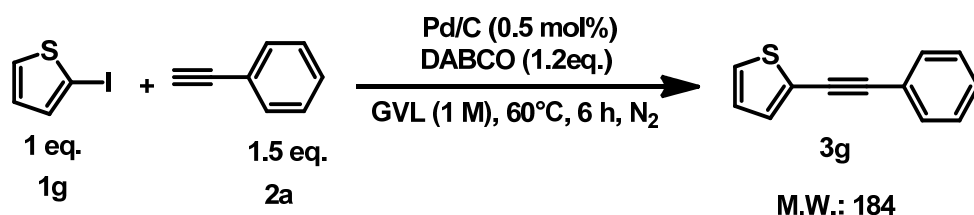
Elemental Analysis: Calc.: C: 86.51; H: 5.81; found C: 86.40; H: 5.75

	δ value	No. H	Mult.	j value/Hz
¹H NMR 400 MHz	3.83	3	s	
	6.88	2	d	8.6
CDCl₃	7.26-7.35	3	m	
	7.46-7.52	4	m	

¹³C NMR (100.6 MHz, CDCl₃) δ : 55.3, 88.0, 89.3, 114.0, 115.4, 123.6, 127.9, 128.3, 131.4, 133.0, 159.6

GC-EIMS (m/z, %): 208 (M⁺, 100), 193 (10), 165 (48), 164 (17), 163 (13), 139 (13)

Chem. Name	2-(phenylethynyl)thiophene (3g)
Lit. Ref.	<i>Green Chem.</i> , 2010 , <i>12</i> , 985-991



METHOD:

In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.005 mmol), GVL (1 mL, 98% purity), DABCO (139 mg, 1.2 mmol, 99% purity), 2-iodothiophene (**1g**) (215 mg, 0.113 mL, 1 mmol, 98% purity), and ethynylbenzene (**2a**) (156 mg, 0.168 mL, 1.5 mmol, 98% purity) were consecutively added, the resulting mixture was purged with nitrogen and left under stirring at 60 °C. After 6 hours, petroleum ether was added and the reaction mixture was filtered over a small pad of celite, washed with neutral water and, subsequently, with acidic water (1 M HCl). The organic layer was dried over sodium sulphate and the solvent was removed under vacuum. The crude oil was purified by column chromatography on silica gel (petroleum ether) to afford **3g** as a white solid (120 mg, 65% yield).

Mol Formula	C ₁₂ H ₈ S	m.p.	50-52 °C
--------------------	----------------------------------	-------------	----------

Elemental Analysis: Calc.: C: 78.22; H: 4.38; S: 17.40 found C: 78.19; H: 4.30, S:17.38

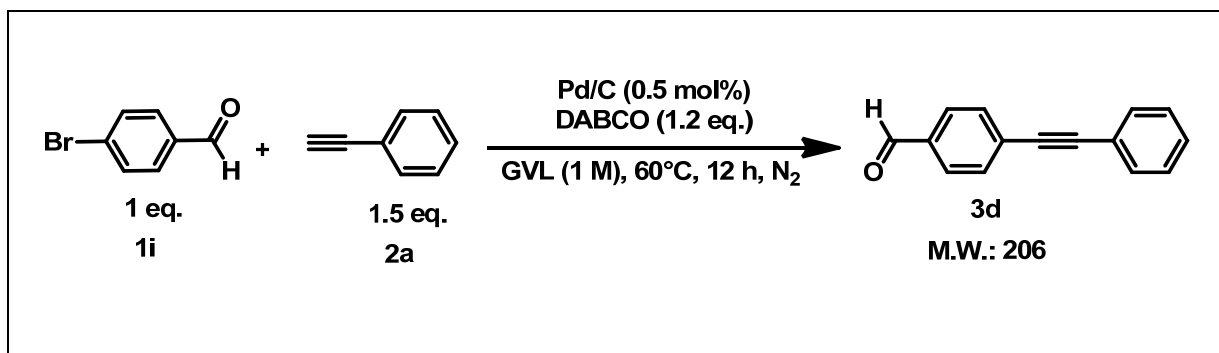
	δ value	No. H	Mult.	j value/Hz
¹H NMR 400 MHz CDCl ₃	7.00-7.02	1	<i>m</i>	
	7.29-7.30	2	<i>m</i>	
	7.34-7.35	3	<i>m</i>	
	7.51-7.53	2	<i>m</i>	

¹³C NMR (100.6 MHz, CDCl₃) δ : 82.6, 93.0, 122.9, 123.3, 127.1, 127.2, 128.4, 128.4, 129.2, 131.4, 131.9, 132.5

GC-EIMS (m/z, %): 184 (M⁺, 100), 183 (8), 152 (16), 139 (25), 126 (10), 113 (5), 98 (5)

Chem. Name	1-nitro-4-(phenylethynyl)benzene (3b)			
Lit. Ref.	<i>Green Chem.</i> , 2012 , <i>14</i> , 2840–2855			
<p style="text-align: center;"> $\text{Br}-\text{C}_6\text{H}_4-\text{NO}_2$ (1h) + $\text{C}_6\text{H}_5\text{C}\equiv\text{CH}$ (2a) $\xrightarrow[\text{GVL (1 M), 60 }^\circ\text{C, 12 h, N}_2]{\text{Pd/C (0.5 mol\%), DABCO (1.2 eq.)}}$ $\text{O}_2\text{N}-\text{C}_6\text{H}_4-\text{C}\equiv\text{C}-\text{C}_6\text{H}_5$ (3b) </p> <p style="text-align: center;">M.W.: 223</p>				
METHOD:				
<p>In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.005 mmol), GVL (1 mL, 98% purity), DABCO (139 mg, 1.2 mmol, 99% purity), 1-Bromo-4-nitrobenzene (1h) (204 mg, 1 mmol, 99% purity), and ethynylbenzene (2a) (156 mg, 0.168 mL, 1.5 mmol, 98% purity) were consecutively added, the resulting mixture was purged with nitrogen and left under stirring at 60 °C. After 12 hours, petroleum ether was added and the reaction mixture was filtered over a small pad of celite, washed with neutral water and, subsequently, with acidic water (1 M HCl). The organic layer was dried over sodium sulphate and the solvent was removed under vacuum. The crude oil was purified by column chromatography on silica gel (1:1 / toluene / petroleum ether) to afford 3b as a white solid (167 mg, 75% yield).</p>				
Mol Formula	$\text{C}_{14}\text{H}_9\text{NO}_2$	m.p.	116-118 °C	
Elemental Analysis: Calc.: C: 75.33; H: 4.06; N:6.27; found C: 75.10; H: 4.99; N: 6.20				
¹H NMR 400 MHz CDCl₃	δ value	No. H	Mult.	j value/Hz
	7.39-7.41	3	<i>m</i>	
	7.55-7.58	2	<i>m</i>	
	7.67	2	<i>d</i>	8.7
	8.23	2	<i>d</i>	8.7
¹³C NMR (100.6 MHz, CDCl₃) δ : 87.6, 94.7, 122.1, 123.6, 128.6, 129.3, 130.24, 131.9, 132.3, 147.0				
GC-EIMS (m/z, %): 223 (M ⁺ , 100), 193 (29), 176 (60), 165 (27), 177 (19), 151 (20), 150 (16)				

Chem. Name	4-(phenylethynyl)benzaldehyde (3d)
Lit. Ref.	<i>Eur. J. Org. Chem.</i> , 2008 , <i>31</i> , 5244-5253



METHOD:

In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.005 mmol), GVL (1 mL, 98% purity), DABCO (139 mg, 1.2 mmol 4-Bromobenzaldehyde (**1i**) (187 mg, 1 mmol, 99% purity), and ethynylbenzene (**2a**) (156 mg, 0.168 mL, 1.5 mmol, 98% purity) were consecutively added, the resulting mixture was purged with nitrogen and left under stirring at 60 °C. After 12 hours, petroleum ether was added and the reaction mixture was filtered over a small pad of celite, washed with neutral water and, subsequently, with acidic water (1 M HCl). The organic layer was dried over sodium sulphate and the solvent was removed under vacuum. The crude oil was purified by column chromatography on silica gel (1:1 / toluene : petroleum ether) to afford **3d** as a white solid (145 mg, 70% yield).

Mol Formula	C ₁₅ H ₁₀ O	m.p.	95-97 °C
--------------------	-----------------------------------	-------------	----------

Elemental Analysis: Calc.: C, 87.36; H, 4.89; found C: 87.10; H:4.80

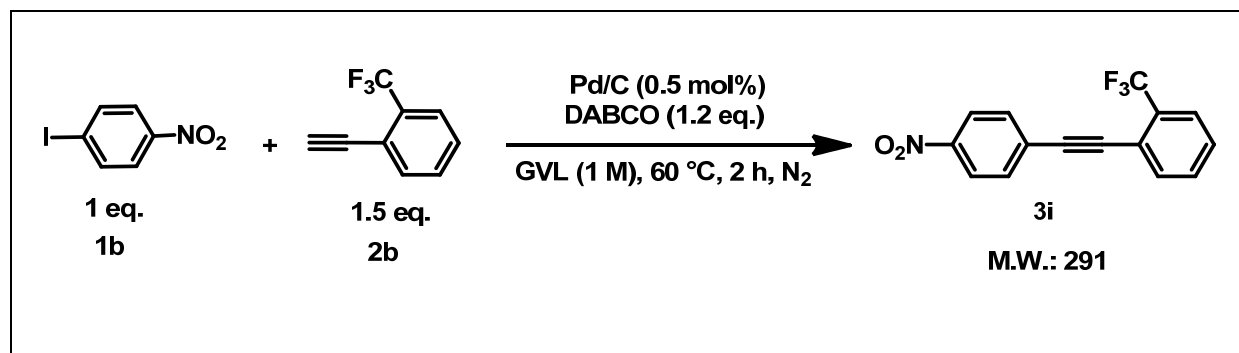
	δ value	No. H	Mult.	j value/Hz	
¹H NMR 400 MHz CDCl₃	7.38	3	<i>br</i>		
	7.55	2	<i>br</i>		
	7.68	2	<i>d</i>	7.8	
	7.87	2	<i>d</i>	7.8	
	10.02	1	<i>s</i>		

¹³C NMR (100.6 MHz, CDCl₃) δ : 88.5, 93.5, 122.5, 128.5, 129.0, 129.6, 131.8, 132.1, 135.4, 191.5

GC-EIMS (m/z, %): 206 (M⁺, 100), 205 (71), 178 (14), 177 (20), 176 (51), 151 (20), 150 (17)

Chem. Name	1-(phenylethynyl)-2-(trifluoromethyl)benzene (3h)			
Lit. Ref.	<i>Chem. Eur. J.</i> , 2008 , <i>14</i> , 6994-6999			
<p style="text-align: center;"> Pd/C (0.5 mol\%) DABCO (1.2 eq.) $\text{GVL (1 M), 60 }^\circ\text{C, 4 h, N}_2$ </p> <p style="text-align: center;"> 1 eq. 1a 1.5 eq. 2b 3h M.W.: 246 </p>				
METHOD:				
<p>In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.005 mmol), GVL (1 mL, 98% purity), DABCO (139 mg, 1.2 mmol, 99% purity), iodobenzene (1a) (139 mg, 0.114 mL, 1 mmol, 98% purity), and 1-ethynyl-2-(trifluoromethyl)benzene (2b) (263 mg, 0.215 mL, 1.5 mmol, 97% purity) were consecutively added, the resulting mixture was purged with nitrogen and left under stirring at 60 °C. After 4 hours, petroleum ether was added and the reaction mixture was filtered over a small pad of celite, washed with neutral water and, subsequently, with acidic water (1 M HCl). The organic layer was dried over sodium sulphate and the solvent was removed under vacuum. The crude oil was purified by column chromatography on silica gel (1:1 / petroleum ether : toluene) to obtain 3h as a white solid (182 mg, 74% yield).</p>				
Mol Formula	C ₁₅ H ₉ F ₃	m.p.	70-73 °C	
Elemental Analysis: Calc.: C: 73.17; H: 3.68; found C: 73.00; H: 3.60				
¹H NMR 400 MHz CDCl₃	δ value	No. H	Mult.	j value/Hz
	7.36-7.43	4	<i>m</i>	
	7.50-7.56	3	<i>m</i>	
	7.66-7.69	2	<i>m</i>	
¹³C NMR (100.6 MHz, CDCl₃) δ: 85.3; 94.9; 121.6, 122.2, 122.7; 125.0, 125.9 (q, <i>j</i> _{F-C} = 5.0 Hz), 127.9, 128.4, 128.8, 131.4, 131.7, 133.7				
¹⁹F NMR(376.4, CDCl₃) δ : -62.7				
GC-EIMS (m/z, %): 246 (M ⁺ , 100), 245 (51), 227 (18), 225 (23), 207 (6), 196 (10)				

Chem. Name	1-((4-nitrophenyl)ethynyl)-2-(trifluoromethyl)benzene (3i)
Lit. Ref.	<i>Chem. Eur. J.</i> , 2008 , <i>14</i> , 6994-6999



METHOD:

In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.005 mmol), GVL (1 mL, 98% purity), DABCO (139 mg, 1.2 mmol, 99% purity), 1-iodo-4-nitrobenzene (**1b**) (254 mg, 1 mmol, 98% purity), and 1-ethynyl-2-(trifluoromethyl)benzene (**2b**) (263 mg, 0.215 mL, 1.5 mmol, 97% purity) were consecutively added, the resulting mixture was purged with nitrogen and left under stirring at 60 °C. After 2 hours, petroleum ether was added and the reaction mixture was filtered over a small pad of celite, washed with neutral water and, subsequently, with acidic water (1 M HCl). The organic layer was dried over sodium sulphate and the solvent was removed under vacuum. The crude oil was purified by column chromatography on silica gel (1:1 / petroleum ether: toluene) to afford **3i** as a pale yellow solid (265 mg, 91% yield).

Mol Formula	C ₁₅ H ₈ F ₃ NO ₂	m.p.	95-98 °C
--------------------	---	-------------	----------

Elemental Analysis: Calc.: C: 61.86; H: 2.77; N: 4.81; found C: 61.76; H: 2.70; N: 4.79

	δ value	No. H	Mult.	j value/Hz
¹H NMR 400 MHz CDCl ₃	7.47-7.51	1	<i>m</i>	
	7.54-7.58	1	<i>m</i>	
	7.66-7.73	4	<i>m</i>	
	8.23	2	<i>d</i>	8.5

¹³C NMR (100.6 MHz, CDCl₃) δ : 90.6, 93.0, 120.7, 122.5, 124.1, 125.2, 126.6 (q, *j*_{F-C} = 4.9 Hz), 129.4, 129.9, 132.0, 132.4, 132.8, 134.4, 147.7

¹⁹F NMR(376.4, CDCl₃) δ : -62.6

GC-EIMS (m/z, %): 291 (M⁺, 100), 261 (19), 245 (24), 243 (10), 233 (24), 232 (24), 225 (34), 214 (12)

Chem. Name	1-(4-((2-(trifluoromethyl)phenyl)ethynyl)phenyl)ethanone (3j)			
Lit. Ref.	<i>Chem. Eur. J.</i> , 2008 , 14, 6994-6999			
<p>1 eq. 1c + 1.5 eq. 2b $\xrightarrow[\text{GVL (1 M), 60}^\circ\text{C, 2.5 h, N}_2]{\text{Pd/C (0.5 mol\%), DABCO (1.2 eq.)}}$ 3j M.W.: 288</p>				
METHOD:				
<p>In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.005 mmol), GVL (1 mL, 98% purity), DABCO (139 mg, 1.2 mmol, 99% purity), 1-(4-iodophenyl)ethanone (1c) (251 mg, 1 mmol, 98% purity), and 1-ethynyl-2-(trifluoromethyl)benzene (2b) (263 mg, 0.215 mL, 1.5 mmol, 97% purity) were consecutively added, the resulting mixture was purged with nitrogen and left under stirring at 60 °C. After 2.5 hours, petroleum ether was added and the reaction mixture was filtered over a small pad of celite, washed with neutral water and, subsequently, with acidic water (1 M HCl). The organic layer was dried over sodium sulphate and the solvent was removed under vacuum. The crude oil was purified by column chromatography on silica gel (1:1 / petroleum ether : toluene) to obtain 3j as a white solid (277 mg, 96% yield).</p>				
Mol Formula	C ₁₇ H ₁₁ F ₃ O	m.p.	80-83 °C	
Elemental Analysis: Calc.: C: 70.83; H: 3.85; found C:70.79; H: 3.80				
¹H NMR 400 MHz CDCl ₃	δ value	No. H	Mult.	j value/Hz
	2.62	3	s	
	7.46-7.48	1	m	
	7.53-7.57	1	m	
	7.63	2	d	8.1
	7.68-7.72	2	m	
	7.96	2	d	8.1
¹³C NMR (100.6 MHz, CDCl₃) δ : 26.7, 88.4, 93.8, 120.9, 122.2, 124.9, 126.0 (q, <i>j</i> _C = 4.9 Hz), 127.5, 128.3, 128.5, 131.5, 131.8, 133.8, 136.6, 197.3				
¹⁹F NMR(376.4, CDCl₃) δ : -62.7				
GC-EIMS (m/z, %): 288 (M ⁺ , 49), 274 (17), 273 (100), 245 (29), 243 (8), 225 (29), 219 (6), 214 (6)				

Chem. Name	1-(p-tolyethynyl)-2-(trifluoromethyl)benzene (3k)			
Lit. Ref.	<i>Appl. Organometal. Chem.</i> , 2011 , <i>25</i> , 514-520			
<p style="text-align: center;"> $\text{1 eq. 1e} + \text{1.5 eq. 2b} \xrightarrow[\text{GVL (1 M), 60^\circ\text{C, 4.5 h, N}_2]{\text{Pd/C (0.5 mol\%), DABCO (1.2 eq.)}} \text{3k}$ M.W.: 260 </p>				
METHOD:				
<p>In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.005 mmol), GVL (1 mL, 98% purity), DABCO (139 mg, 1.2 mmol, 99% purity), 1-iodo-4-methylbenzene (1e) (220 mg, 1 mmol, 99% purity), and 1-ethynyl-2-(trifluoromethyl)benzene (2b) (263 mg, 0.215 mL, 1.5 mmol, 97% purity) were consecutively added, the resulting mixture was purged with nitrogen and left under stirring at 60 °C. After 4.5 hours, petroleum ether was added and the reaction mixture was filtered over a small pad of celite, washed with neutral water and, subsequently, with acidic water (1 M HCl). The organic layer was dried over sodium sulphate and the solvent was removed under vacuum. The crude oil was purified by column chromatography on silica gel (petroleum ether) to afford 3k as a white solid (182 mg, 70% yield).</p>				
Mol Formula	C ₁₆ H ₁₁ F ₃	m.p.	73-75 °C	
Elemental Analysis: Calc.: C: 73.84; H: 4.26; found C: 73.79; H: 4.25				
¹H NMR 400 MHz CDCl ₃	δ value	No. H	Mult.	j value/Hz
	2.38	3	s	
	7.18	2	d	7.6
	7.38-7.53	4	m	
	7.65-7.69	2	m	
¹³C NMR (100.6 MHz, CDCl₃) δ : 21.5, 84.8, 95.2, 119.7, 121.8, 122.3, 125.0, 125.9 (q, <i>j</i> _{F-C} = 4.8 Hz), 127.7, 129.2, 131.4, 131.6, 133.6, 139.1				
¹⁹F NMR(376.4, CDCl₃) δ : -62.6				
GC-EIMS (m/z, %): 260 (M ⁺ , 100), 259 (26), 239 (21), 238 (25), 221 (9), 220 (20), 189 (11)				

Chem. Name	1-((4-methoxyphenyl)ethynyl)-2-(trifluoromethyl)benzene (3I)
Lit. Ref.	<i>Chem. Eur. J.</i> , 2008 , <i>14</i> , 6994-6999

METHOD:

In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.005 mmol), GVL (1 mL, 98% purity), DABCO (139 mg, 1.2 mmol, 99% purity), 1-iodo-4-methoxybenzene (**1f**) (238 mg, 1 mmol, 98% purity), and 1-ethynyl-2-(trifluoromethyl)benzene (**2b**) (263 mg, 0.215 mL, 1.5 mmol, 97% purity) were consecutively added, the resulting mixture was purged with nitrogen and left under stirring at 60 °C. After 5 hours, petroleum ether was added and the reaction mixture was filtered over a small pad of celite, washed with neutral water and, subsequently, with acidic water (1 M HCl). The organic layer was dried over sodium sulphate and the solvent was removed under vacuum. The crude oil was purified by column chromatography on silica gel (1:1 / petroleum ether : toluene) to afford **3I** as a white solid (180 mg, 65% yield).

Mol Formula	C ₁₆ H ₁₁ F ₃ O	m.p.	64-67 °C
--------------------	--	-------------	----------

Elemental Analysis: Calc.: C: 69.56; H: 4.01; found C: 69.49; H: 3.99

	δ value	No. H	Mult.	j value/Hz
¹H NMR 400 MHz CDCl ₃	3.84	3	<i>s</i>	
	6.89	2	<i>d</i>	8
	7.36-7.40	1	<i>m</i>	
	7.49	3	<i>d</i>	8
	7.63-7.68	2	<i>m</i>	

¹³C NMR (100.6 MHz, CDCl₃) δ : 55.3, 84.2, 95.1, 114.0, 114.8, 121.9, 122.3, 125.0, 125.9 (q, j_{F-C} = 4.8 Hz), 127.5, 131.3, 133.2, 133.4, 160.1

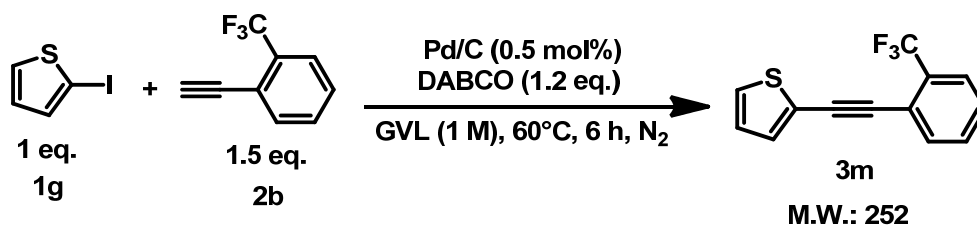
¹⁹F NMR(376.4,CDCl₃) δ : -62.9

GC-EIMS (m/z, %): 276 (M⁺, 100), 261 (19), 233 (27), 232 (43), 214 (9), 183 (14)

Chem. Name	2-((2-(trifluoromethyl)phenyl)ethynyl)thiophene (3m)
-------------------	---

Lit. Ref.

Appl. Organometal. Chem., 2011, 25, 514-520



METHOD:

In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.005 mmol), GVL (1 mL, 98% purity), DABCO (139 mg, 1.2 mmol, 99% purity), 2-iodothiophene (**1g**) (215 mg, 0.113 mL, 1 mmol, 98% purity), and 1-ethynyl-2-(trifluoromethyl)benzene (**2b**) (263 mg, 0.215 mL, 1.5 mmol, 97% purity) were consecutively added, the resulting mixture was purged with nitrogen and left under stirring at 60 °C. After 6 hours, petroleum ether was added and the reaction mixture was filtered over a small pad of celite, washed with neutral water and, subsequently, with acidic water (1 M HCl). The organic layer was dried over sodium sulphate and the solvent was removed under vacuum. The crude oil was purified by column chromatography on silica gel (petroleum ether) to obtain **3m** as a white solid (151 mg, 60% yield).

Mol Formula	C ₁₃ H ₇ F ₃ S	m.p.	55-56 °C
--------------------	---	-------------	----------

Elemental Analysis: Calc.: C: 61.90; H: 2.80; S: 12.71; found C: 61.88, H: 2.78, S: 12.67

	δ value	No. H	Mult.	j value/Hz
¹H NMR 400 MHz CDCl ₃	7.04-7.06	1	<i>m</i>	
	7.35-7.36	2	<i>m</i>	
	7.42-7.43	1	<i>m</i>	
	7.50-7.54	1	<i>m</i>	
	7.65-7.70	2	<i>m</i>	

¹³C NMR (100.6 MHz, CDCl₃) δ : 88.3, 89.1, 121.2, 122.2, 122.6, 124.9, 125.9 (q, $j_{\text{C-F}} = 4.9$ Hz), 127.2, 128.0, 128.2, 131.4, 132.7, 133.4

¹⁹F NMR (376.4, CDCl₃) δ : -62.7

GC-EIMS (m/z, %): 252 (M⁺, 100), 233 (32), 219 (11), 207 (9), 206 (11), 202 (10), 188 (10)

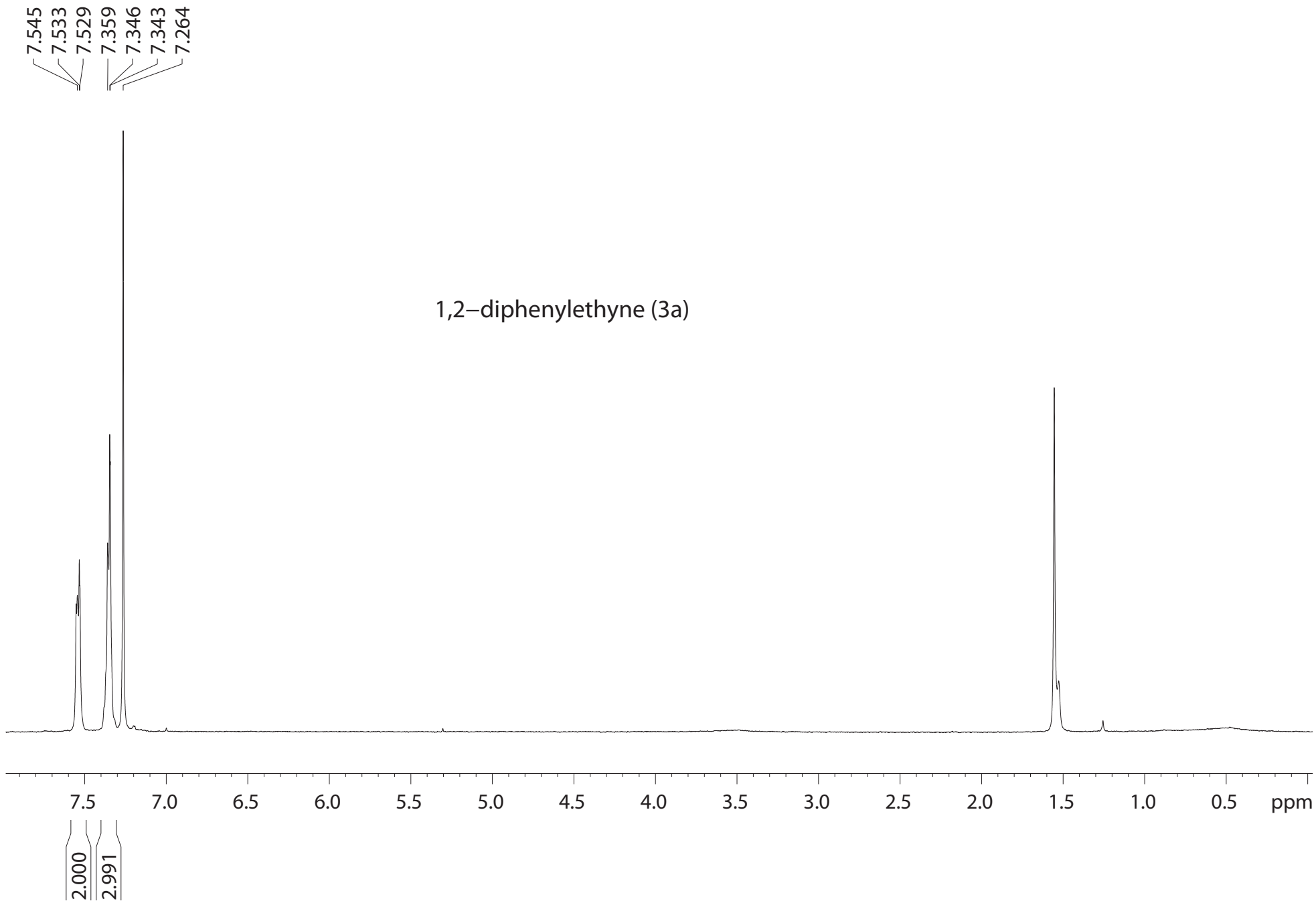
Chem. Name	1-((4-nitrophenyl)ethynyl)-2-(trifluoromethyl)benzene (3i)			
Lit. Ref.	<i>Chem. Eur. J.</i> , 2008 , <i>14</i> , 6994-6999			
<p style="text-align: center;"> $\text{Br}-\text{C}_6\text{H}_4-\text{NO}_2$ (1 eq. 1h) + $\text{C}_6\text{H}_4(\text{F}_3\text{C})-\text{C}\equiv\text{CH}$ (1.5 eq. 2b) $\xrightarrow[\text{GVL (1 M), 60 }^\circ\text{C, 12 h, N}_2]{\text{Pd/C (0.5 mol\%), DABCO (1.2 eq.)}}$ $\text{O}_2\text{N}-\text{C}_6\text{H}_4-\text{C}\equiv\text{C}-\text{C}_6\text{H}_4(\text{F}_3\text{C})$ (3i) M.W.: 291 </p>				
METHOD:				
<p>In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.005 mmol), GVL (1 mL, 98% purity), DABCO (139 mg, 1.2 mmol, 99% purity), 1-bromo-4-nitrobenzene (1h) (204 mg, 1 mmol, 99% purity), and 1-ethynyl-2-(trifluoromethyl)benzene (2b) (263 mg, 0.215 mL, 1.5 mmol, 97% purity) were consecutively added, the resulting mixture was purged with nitrogen and left under stirring at 60 °C. After 12 hours, petroleum ether was added and the reaction mixture was filtered over a small pad of celite, washed with neutral water and, subsequently, with acidic water (1 M HCl). The organic layer was dried over sodium sulphate and the solvent was removed under vacuum. The crude oil was purified by column chromatography on silica gel (1:1 / petroleum ether : toluene) to afford 3i as a white solid (224 mg, 77% yield).</p>				
Mol Formula	C ₁₅ H ₈ F ₃ NO ₂	m.p.	95-98 °C	
Elemental Analysis: Calc.: C: 61.86; H: 2.77; N: 4.81; found C: 61.76; H: 2.70; N: 4.79				
¹H NMR 400 MHz CDCl ₃	δ value	No. H	Mult.	j value/Hz
	7.47-7.51	1	<i>m</i>	
	7.54-7.58	1	<i>m</i>	
	7.66-7.73	4	<i>m</i>	
	8.23	2	<i>d</i>	8.5
¹³C NMR (100.6 MHz, CDCl₃) δ : 90.6, 93.0, 120.7, 122.5, 124.1, 125.2, 126.6 (q, <i>j</i> _{F-C} = 4.9 Hz), 129.4, 129.9, 132.0, 132.4, 132.8, 134.4, 147.7				
¹⁹F NMR(376.4, CDCl₃) δ : 62.6				
GC-EIMS (m/z, %): 291 (M ⁺ , 100), 261 (19), 245 (24), 243 (10), 233 (24), 232 (24), 225 (34), 214 (12)				

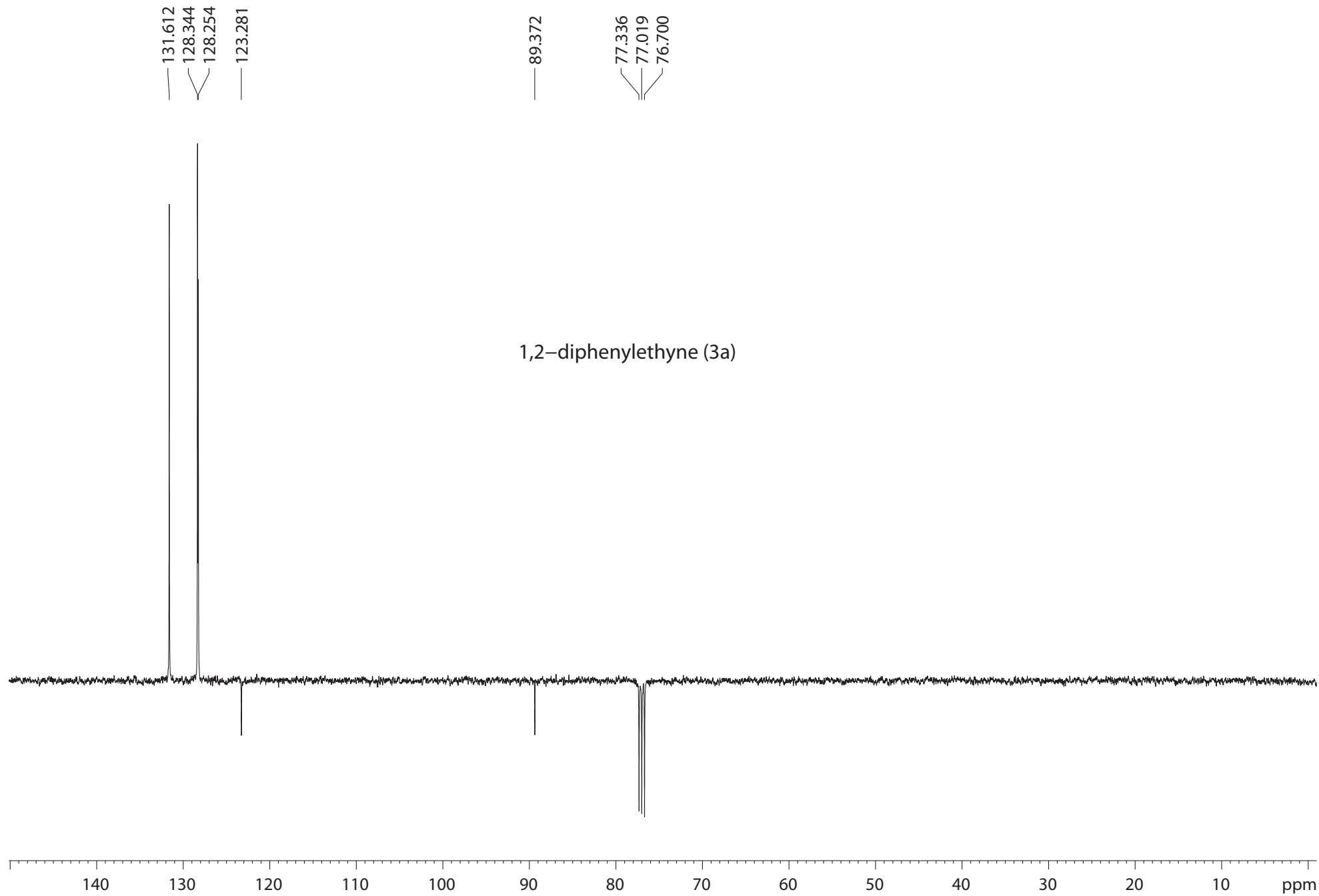
Chem. Name	1-nitro-4-(oct-1-ynyl)benzene (3n)			
Lit. Ref.	<i>J. Org. Chem.</i> , 2004 , 69, 5752-5755			
<p style="text-align: center;"> Pd/C (0.5 mol\%) DABCO (2 eq.) $\text{GVL (1 M), 60 }^\circ\text{C, 4.5 h, N}_2$ </p> <p style="text-align: center;"> 1b 2c 3n M.W.: 231 </p>				
METHOD:				
<p>In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.005 mmol), GVL (1 mL, 98% purity), DABCO (231 mg, 2 mmol, 99% purity), 1-iodo-4-nitrobenzene (1b) (254 mg, 1 mmol, 98% purity), and oct-1-yne (2c) (172 mg, 0.230 mL, 1.5 mmol, 97% purity) were consecutively added, the resulting mixture was purged with nitrogen and left under stirring at 60 °C. After 4.5 hours, petroleum ether was added and the reaction mixture was filtered over a small pad of celite, washed with neutral water and, subsequently, with acidic water (1 M HCl). The organic layer was dried over sodium sulphate and the solvent was removed under vacuum. The crude oil was purified by column chromatography on silica gel (1:1 / petroleum ether : toluene) to afford 3n as an oil (218 mg, 94% yield).</p>				
Mol Formula	C ₁₄ H ₁₇ NO ₂	m.p.	oil	
Elemental Analysis: Calc.: C: 71.87; H: 6.96; N: 6.06; found C: 71.80; H: 6.90; N: 6.00				
¹H NMR 400 MHz CDCl₃	δ value	No. H	Mult.	j value/Hz
	0.91	3	<i>t</i>	6.6
	1.32-1.34	4	<i>m</i>	
	1.42-1.49	2	<i>m</i>	
	1.58 -1.66	2	<i>m</i>	
	2.44	2	<i>t</i>	7.1
	7.51	2	<i>d</i>	8.7
8.15	2	<i>d</i>	8.7	
¹³C NMR (100.6 MHz, CDCl₃) δ : 14.0; 19.6; 22.5; 28.3; 28.6; 31.3; 79.3; 96.2; 123.5; 131.2; 132.2; 146.6				
GC-EIMS (m/z, %): 231 (M ⁺ , 21), 202 (60), 188 (51), 156 (45), 142 (71), 141 (53), 128 (100), 129 (54), 114 (45), 115 (75), 116 (46)				

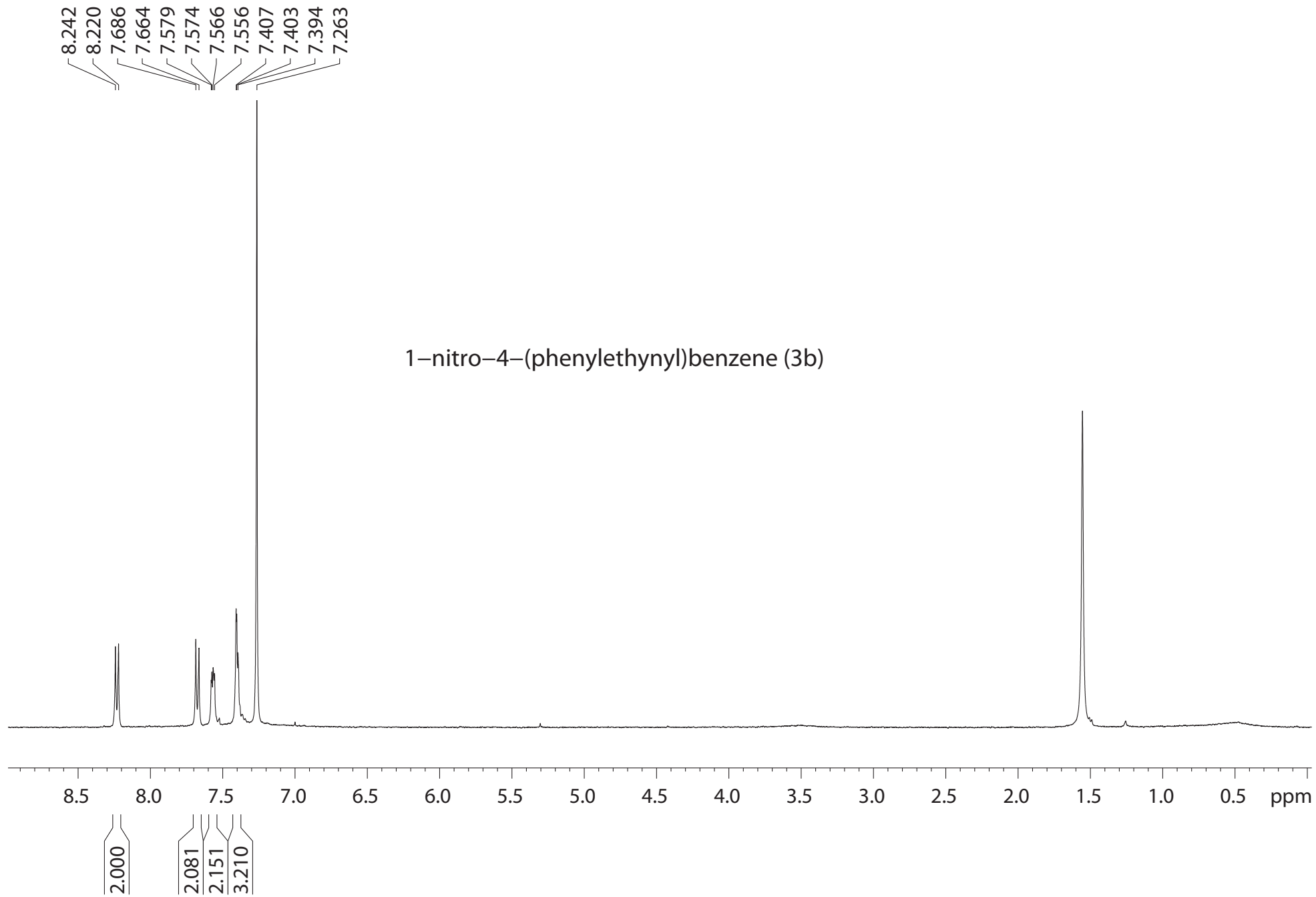
Chem. Name	1-(4-(oct-1-ynyl)phenyl)ethanone (3o)			
Lit. Ref.	<i>J. Org. Chem.</i> , 2004 , 69, 5752-5755			
<p>1 eq. 1c + 1.5 eq. 2c $\xrightarrow[\text{GVL (1 M), 60 }^\circ\text{C, 4.5 h, N}_2]{\text{Pd/C (0.5 mol\%), DABCO (2 eq.)}}$ 3o M.W.: 228</p>				
METHOD:				
<p>In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.005 mmol), GVL (1 mL, 98% purity), DABCO (231 mg, 2 mmol, 99% purity), 1-(4-iodophenyl)ethanone (1c) (251 mg, 1 mmol, 98% purity), and oct-1-yne (2c) (172 mg, 0.230 mL, 1.5 mmol, 97% purity) were consecutively added, the resulting mixture was purged with nitrogen and left under stirring at 60 °C. After 4.5 hours, petroleum ether was added and the reaction mixture was filtered over a small pad of celite, washed with neutral water and, subsequently, with acidic water (1 M HCl). The organic layer was dried over sodium sulphate and the solvent was removed under vacuum. The crude oil was purified by column chromatography on silica gel (1:1 / petroleum ether : toluene) to afford 3o as an oil (220 mg, 96% yield).</p>				
Mol Formula	C ₁₆ H ₂₀ O	m.p.	oil	
Elemental Analysis: Calc.: C: 84.16; H: 8.83; found C: 84.10; H: 8.79				
¹H NMR 400 MHz CDCl₃	δ value	No. H	Mult.	j value/Hz
	0.91	3	<i>t</i>	6.4
	1.24-1.33	4	<i>m</i>	
	1.42-1.49	2	<i>m</i>	
	1.59-1.65	2	<i>m</i>	
	2.43	2	<i>t</i>	7.1
	2.61	3	<i>s</i>	
	7.46	2	<i>d</i>	8.2
7.87	2	<i>d</i>	8.2	
¹³C NMR (100.6 MHz, CDCl₃) δ : 14.0; 19.5; 22.5; 26.6; 28.5; 28.6; 31.3; 80.1; 94.4; 128.1; 129.2; 131.6; 135.6; 197.4				
GC-EIMS (m/z, %): 228 (M ⁺ , 26), 213 (48), 157 (25), 143 (25), 129 (51), 128 (28), 115 (28), 114 (33)				

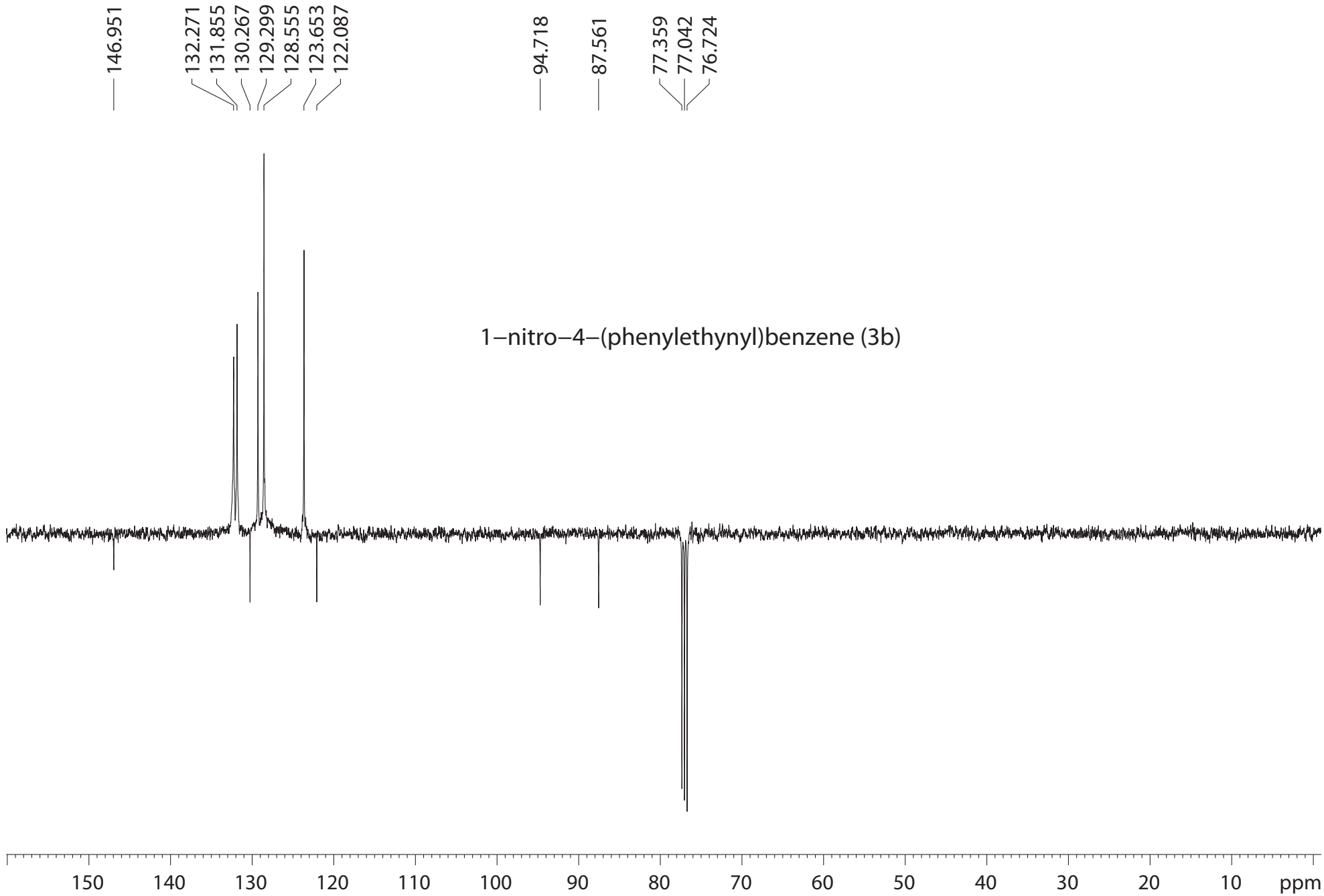
Chem. Name	1-(hex-1-ynyl)-4-nitrobenzene (3p)			
Lit. Ref.	<i>Tetrahedron</i> , 2013 , 69, 5178-5184			
<p>1 eq. 1b + 1.5 eq. 2d $\xrightarrow[\text{GVL (1 M), 60 }^\circ\text{C, 4.5 h, N}_2]{\text{Pd/C (0.5 mol\%), DABCO (2 eq.)}}$ 3p M.W.: 203</p>				
METHOD:				
<p>In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.005 mmol), GVL (1 mL, 98% purity), DABCO (231 mg, 2 mmol, 99% purity), 1-iodo-4-nitrobenzene (1b) (254 mg, 1 mmol, 98% purity), and hex-1-yne (2d) (127 mg, 0.178 mL, 1.5 mmol, 97% purity) were consecutively added, the resulting mixture was purged with nitrogen and left under stirring at 60 °C. After 4.5 hours, petroleum ether was added and the reaction mixture was filtered over a small pad of celite, washed with neutral water and, subsequently, with acidic water (1 M HCl). The organic layer was dried over sodium sulphate and the solvent was removed under vacuum. The crude oil was purified by column chromatography on silica gel (1:1 / petroleum ether : toluene) to afford 3p as an oil (177 mg, 87% yield).</p>				
Mol Formula	C ₁₂ H ₁₃ NO ₂	m.p.	oil	
Elemental Analysis: Calc.: C: 70.92; H: 6.45; N: 6.89; found C: 70.89; H: 6.40; N: 6.80				
¹ H NMR 400 MHz CDCl ₃	δ value	No. H	Mult.	j value/Hz
	0.96	3	<i>t</i>	7.2
	1.45-1.53	2	<i>m</i>	
	1.57-1.65	2	<i>m</i>	
	2.45	2	<i>t</i>	7.1
	7.51	2	<i>d</i>	8.7
	8.14	2	<i>d</i>	8.7
¹³ C NMR (100.6 MHz, CDCl ₃) δ :13.6; 19.2; 22.0; 30.4; 79.3; 96.8; 123.5; 131.2; 132.2; 146.5				
GC-EIMS (m/z, %): 203 (M ⁺ , 66), 188 (100), 156 (33), 142 (94), 128 (93), 114 (46), 115 (71), 102 (39), 63 (29)				

Chem. Name	trimethyl((4-nitrophenyl)ethynyl)silane (3q)			
Lit. Ref.	<i>Synthesis</i> , 1980, 627-630			
<p style="text-align: center;"> $\text{I-C}_6\text{H}_4\text{-NO}_2$ (1 eq. 1b) + $\text{C}\equiv\text{C-Si(CH}_3\text{)}_3$ (1.5 eq. 2e) $\xrightarrow[\text{GVL (1 M), 60 }^\circ\text{C, 6 h, N}_2]{\text{Pd/C (0.5 mol\%), DABCO (2 eq.)}}$ $\text{O}_2\text{N-C}_6\text{H}_4\text{-C}\equiv\text{C-Si(CH}_3\text{)}_3$ (3q) M.W.: 219 </p>				
METHOD:				
<p>In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.005 mmol), GVL (1 mL, 98% purity), DABCO (231 mg, 2 mmol, 99% purity), 1-iodo-4-nitrobenzene (1b) (254 mg, 1 mmol, 98% purity), and ethynyltrimethylsilane (2e) (150 mg, 0.216 mL, 1.5 mmol, 98% purity) were consecutively added, the resulting mixture was purged with nitrogen and left under stirring at 60 °C. After 6 hours, petroleum ether was added and the reaction mixture was filtered over a small pad of celite, washed with neutral water and, subsequently, with acidic water (1 M HCl). The organic layer was dried over sodium sulphate and the solvent was removed under vacuum. The crude oil was purified by column chromatography on silica gel (1:1 / petroleum ether : toluene) to afford 3q as a white solid (134 mg, 61% yield).</p>				
Mol Formula	$\text{C}_{11}\text{H}_{13}\text{NO}_2\text{Si}$	m.p.	98-100 °C	
Elemental Analysis: Calc.: C: 60.24; H: 5.97; N: 6.39; found C: 60.00; H: 5.90; N: 6.00				
¹H NMR 400 MHz CDCl_3 CDCl_3	δ value	No. H	Mult.	j value/Hz
	0.27	9	s	
	7.59	2	d	8.6
	8.17	2	d	8.6
¹³C NMR (100.6 MHz, CDCl_3) δ : -0.3, 100.6, 102.7, 123.5, 130.0, 132.7, 147.1				
GC-EIMS (m/z, %): 219 (M^+ , 16), 205 (22), 204 (100), 158 (35), 145 (9), 143 (17), 117 (7)				





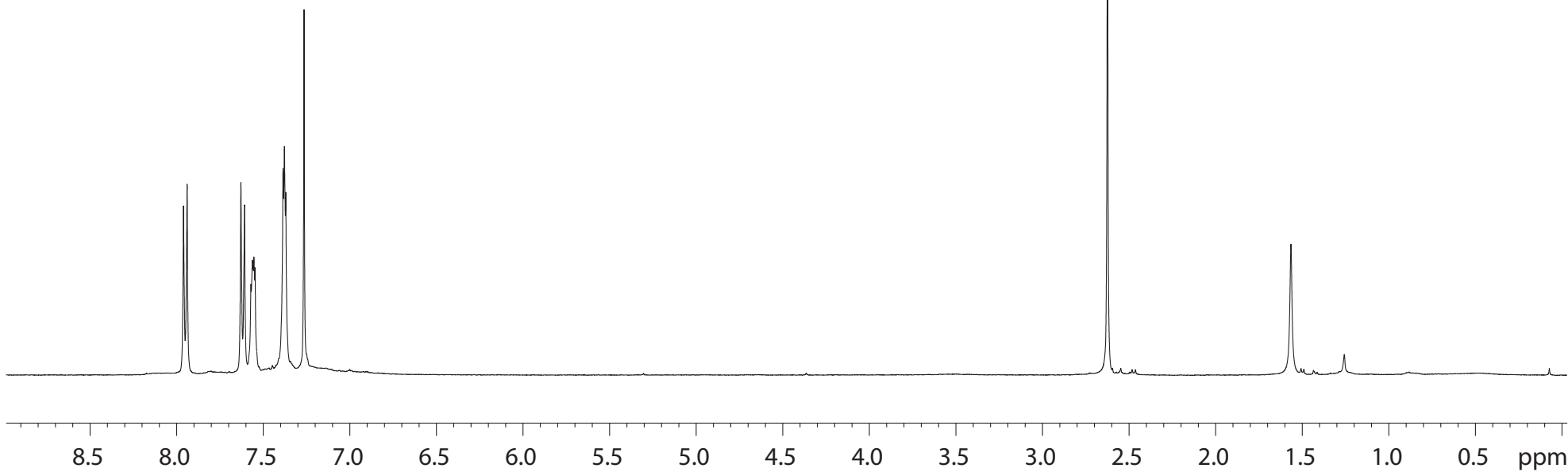




7.960
7.939
7.628
7.607
7.570
7.562
7.557
7.553
7.547
7.523
7.385
7.377
7.369
7.264

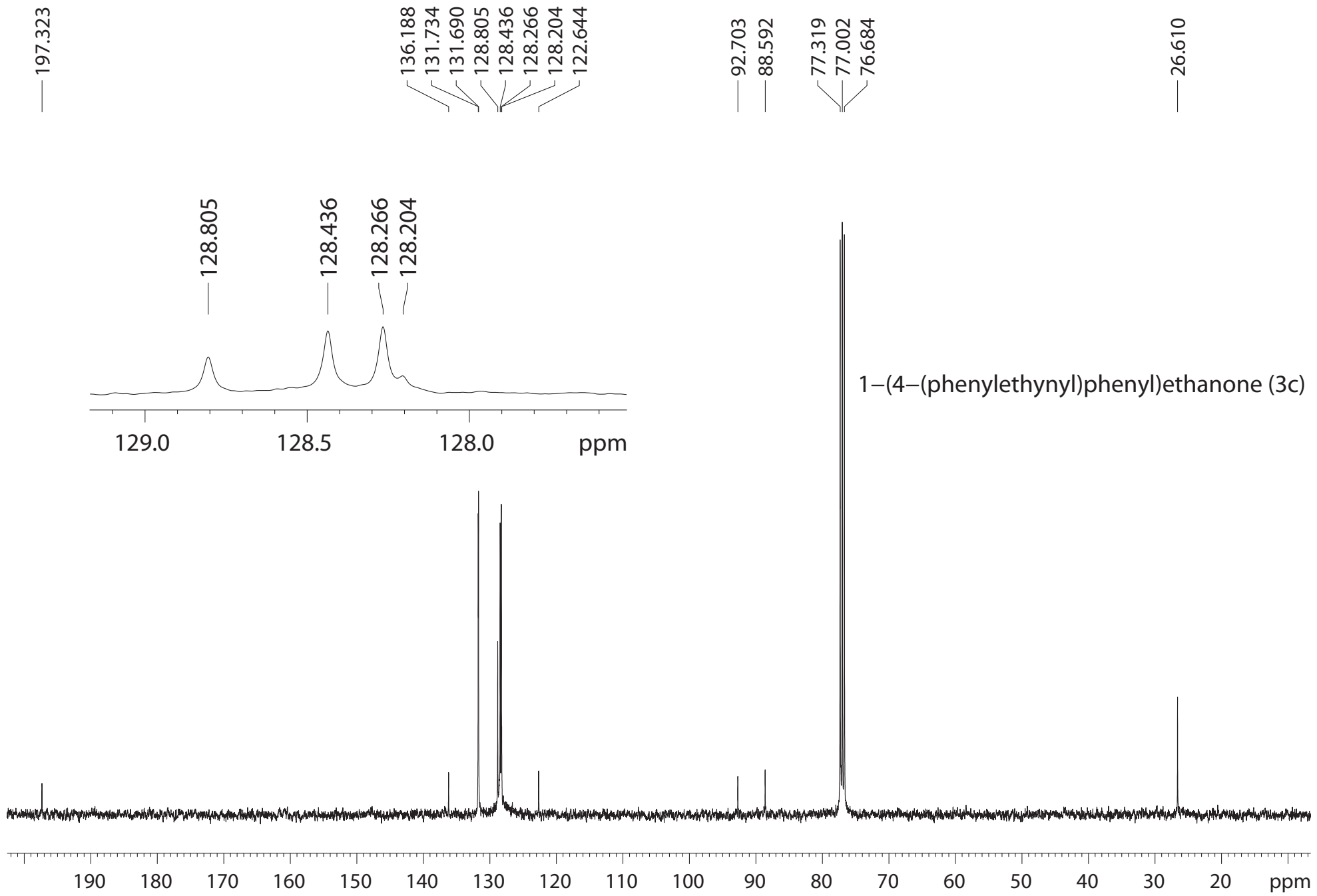
1-(4-(phenylethynyl)phenyl)ethanone (3c)

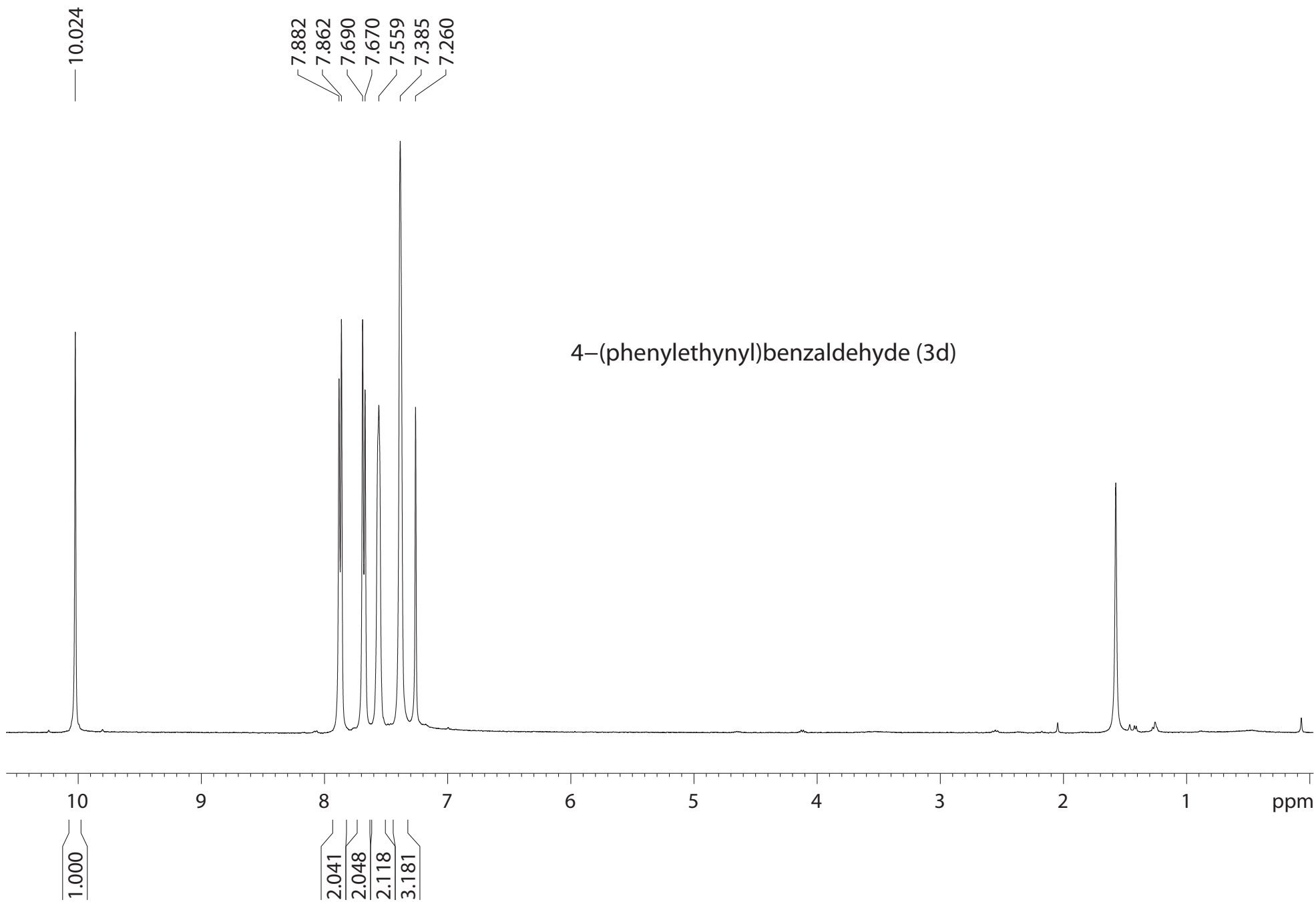
2.624

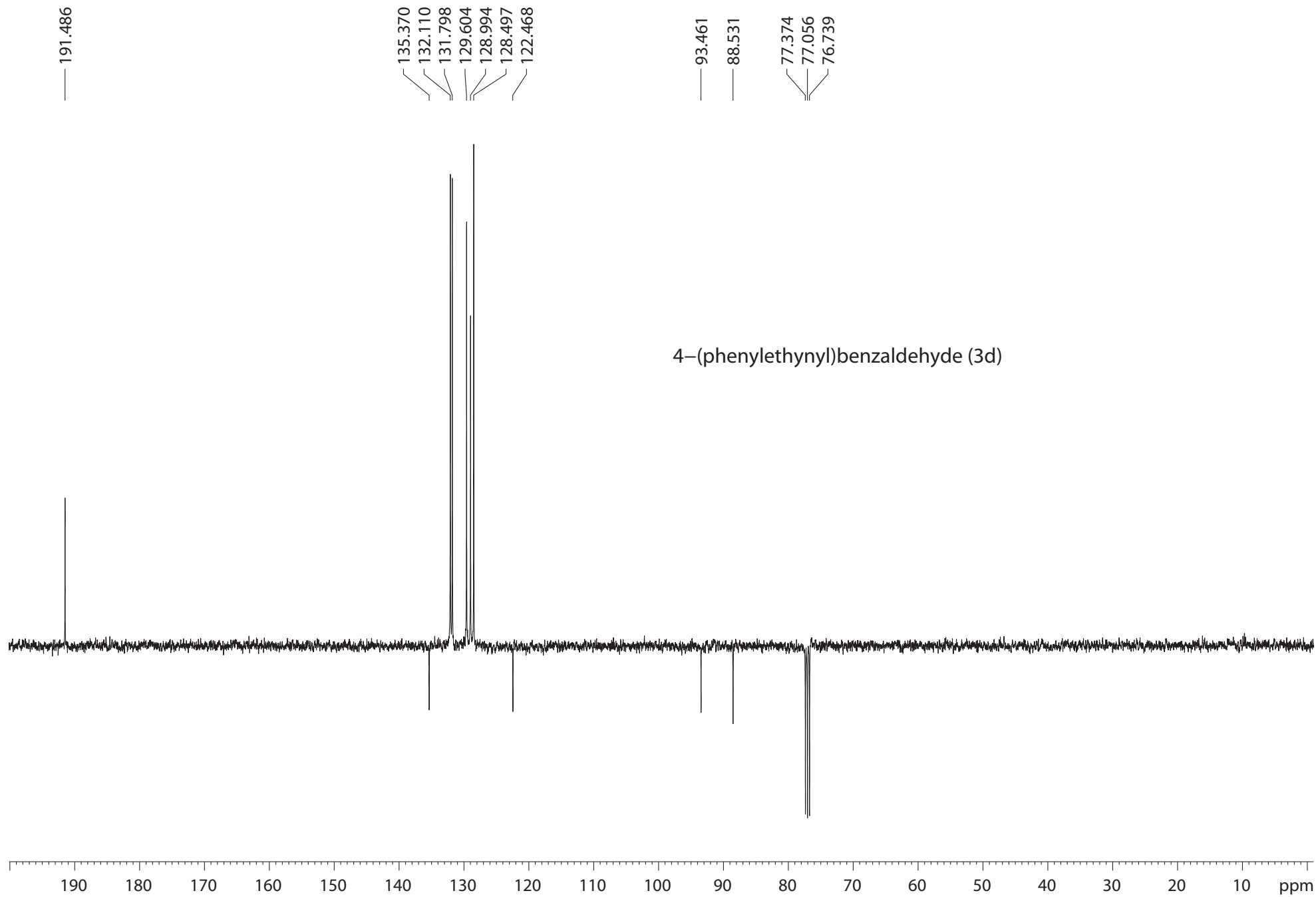


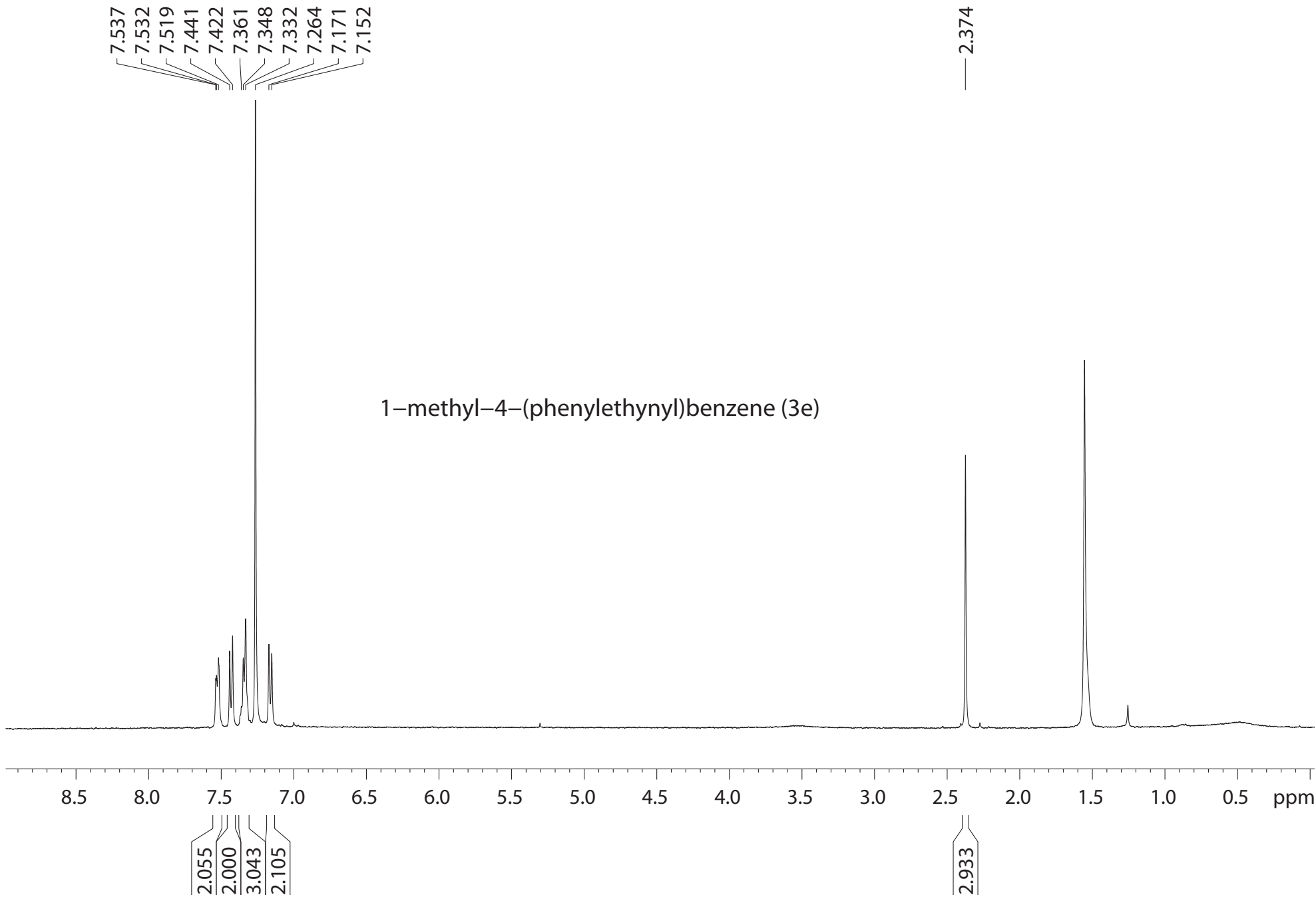
2.000
2.040
2.144
3.306

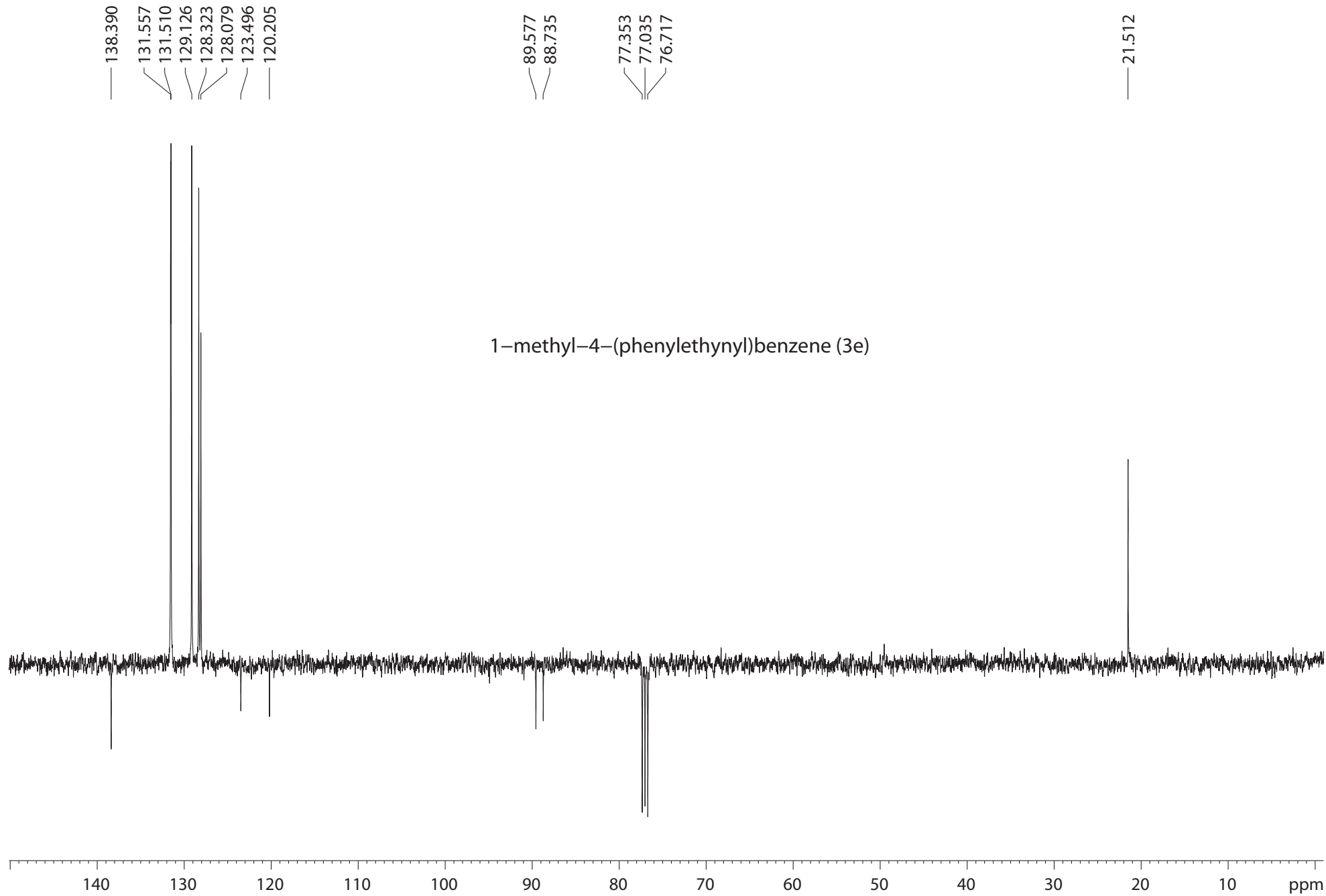
2.879

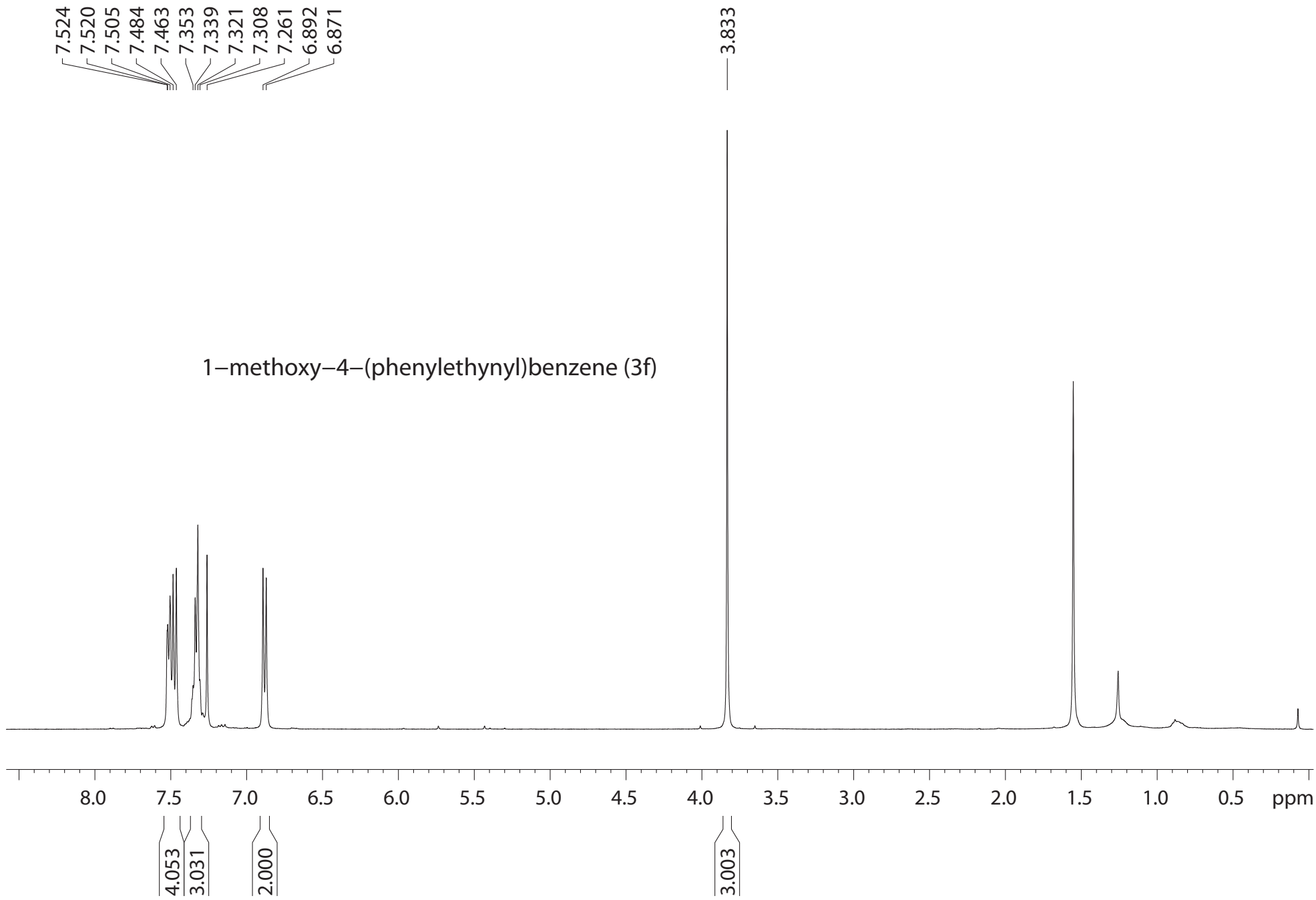


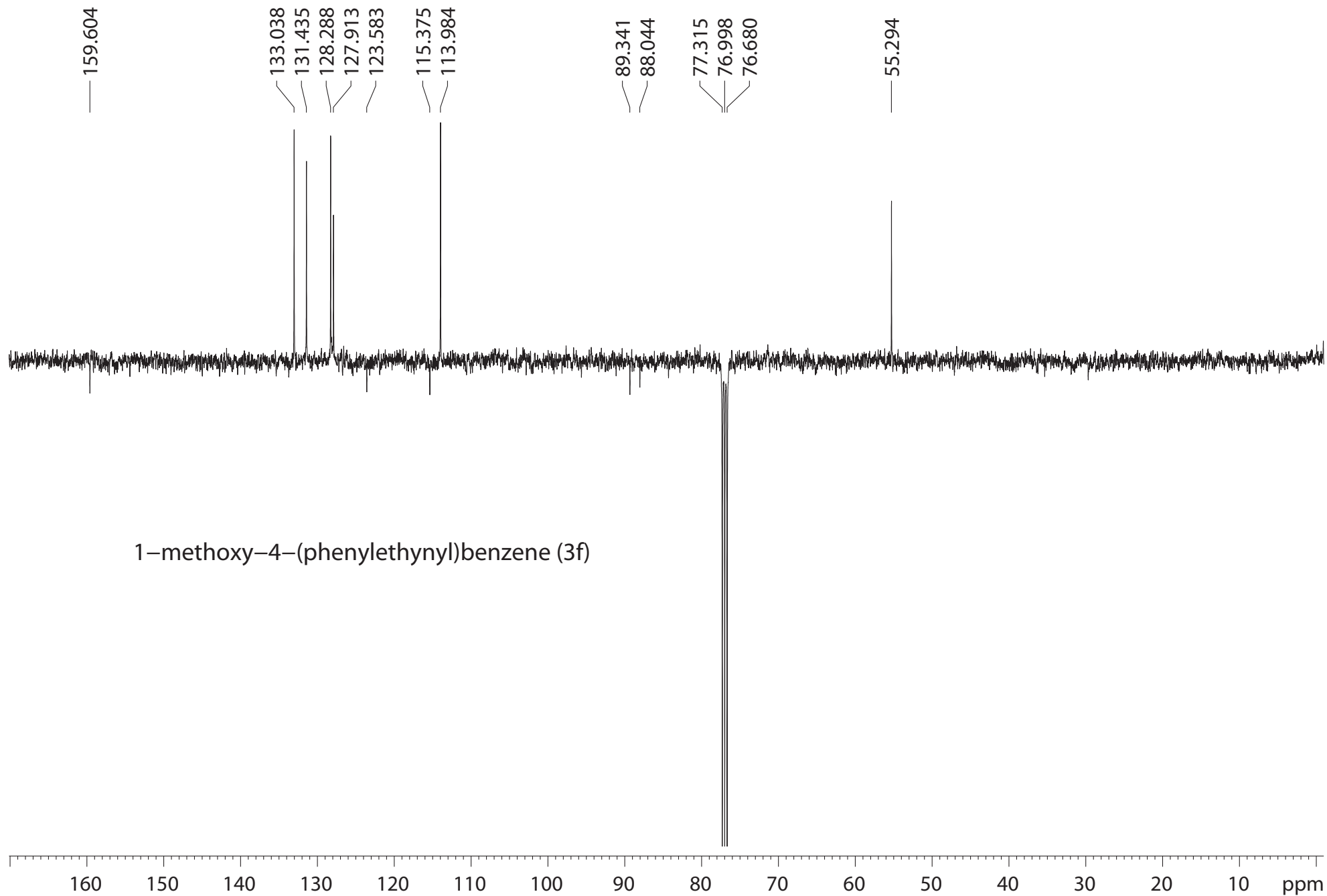


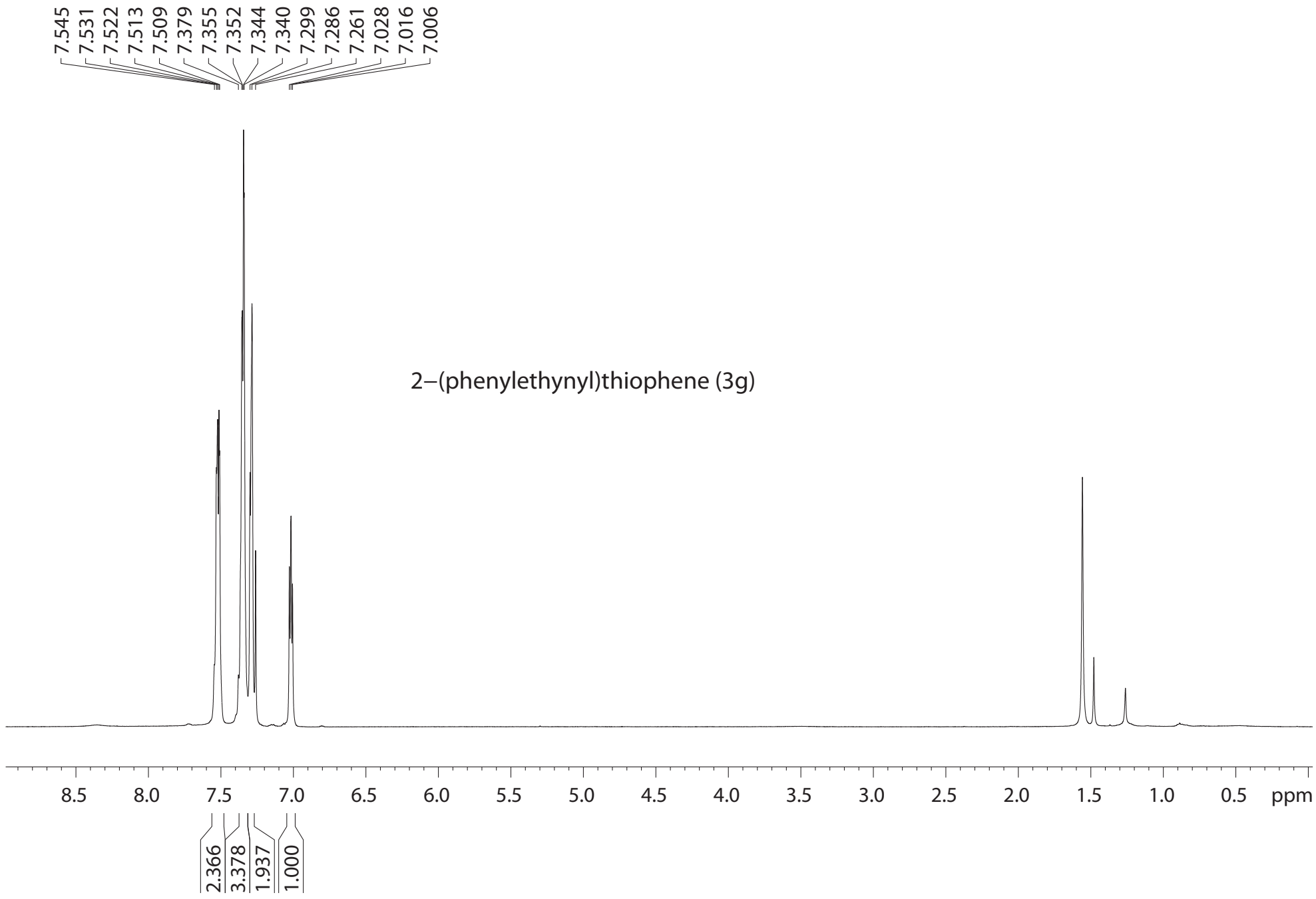


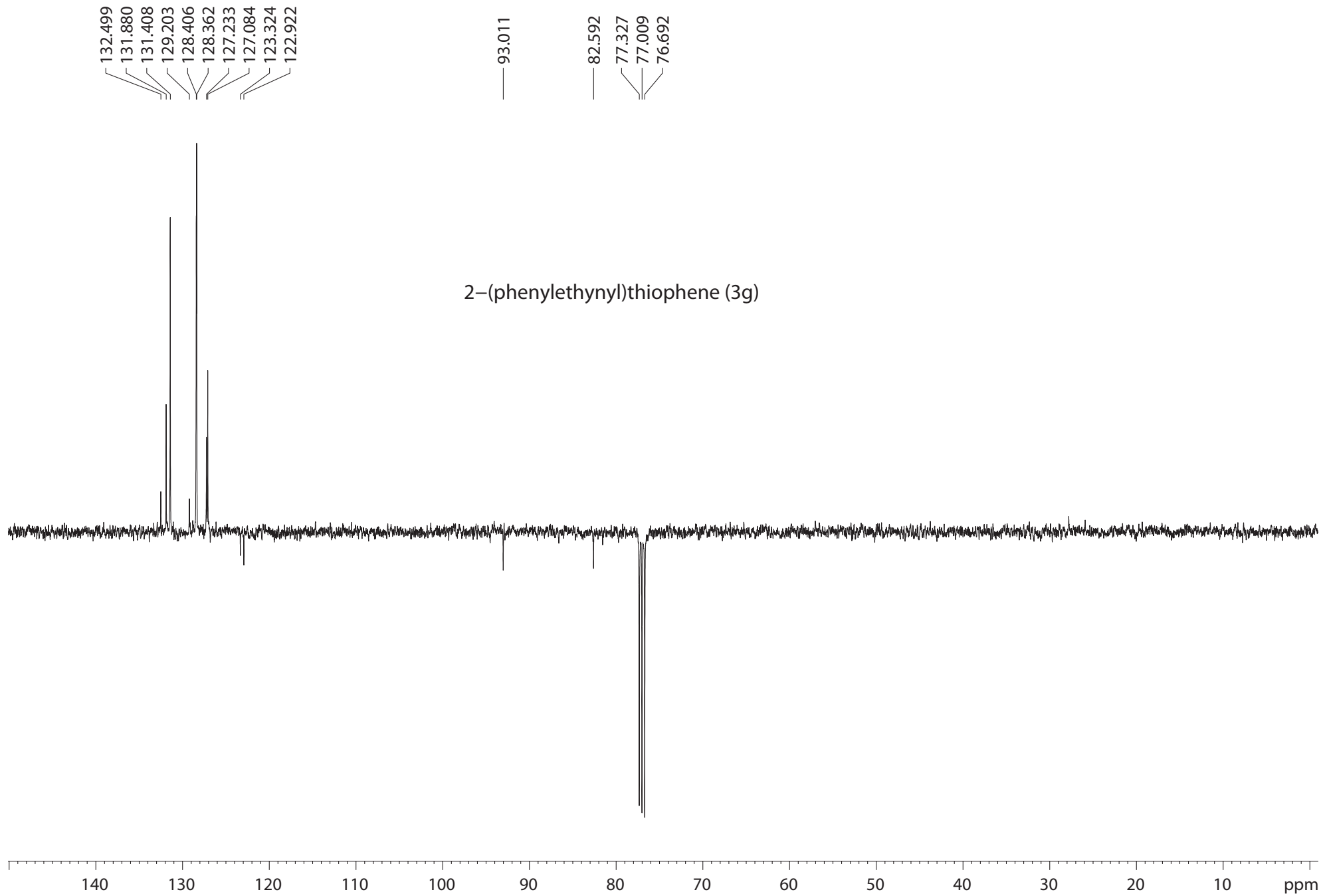


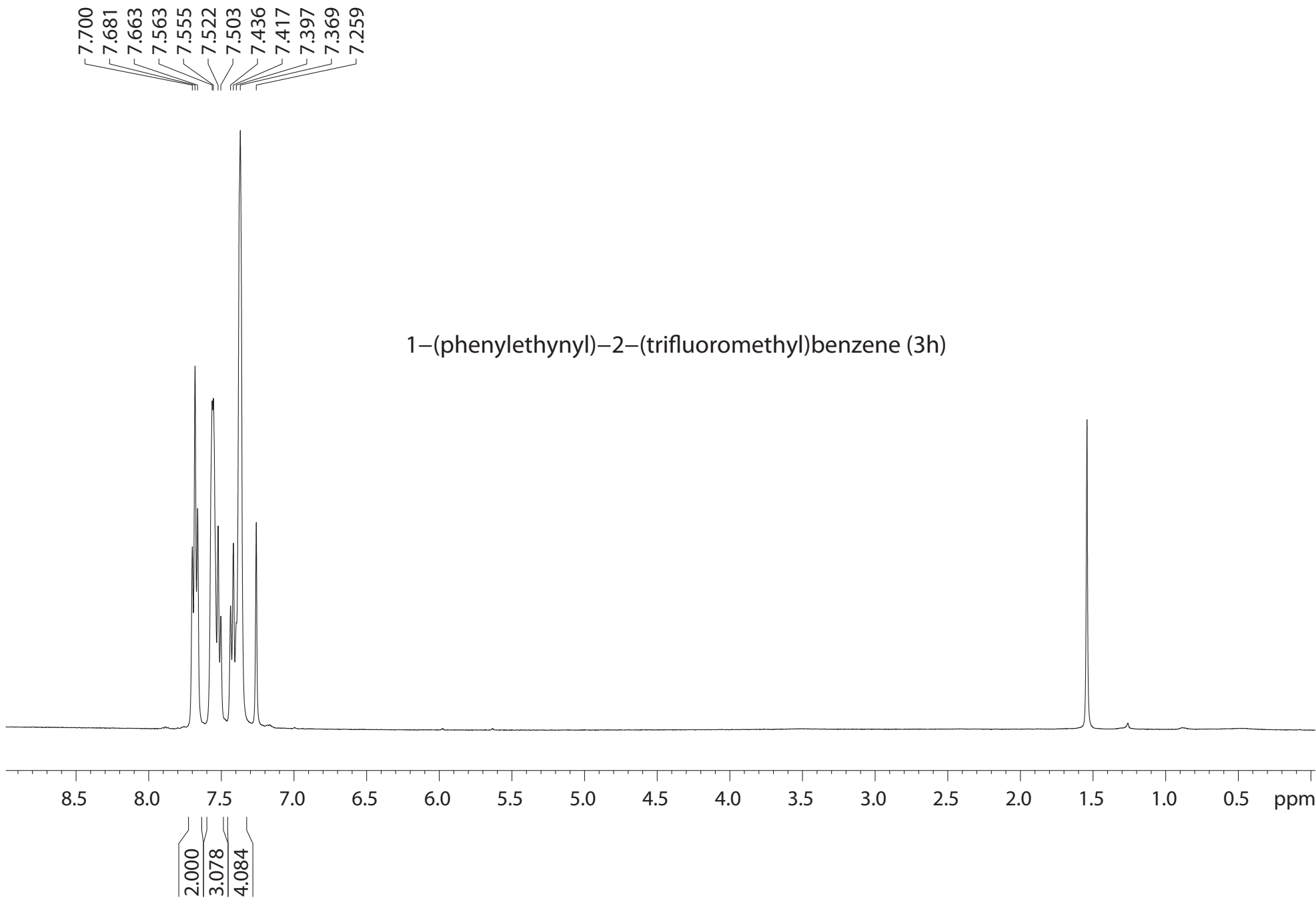






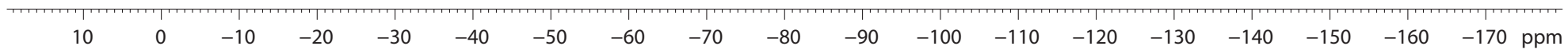


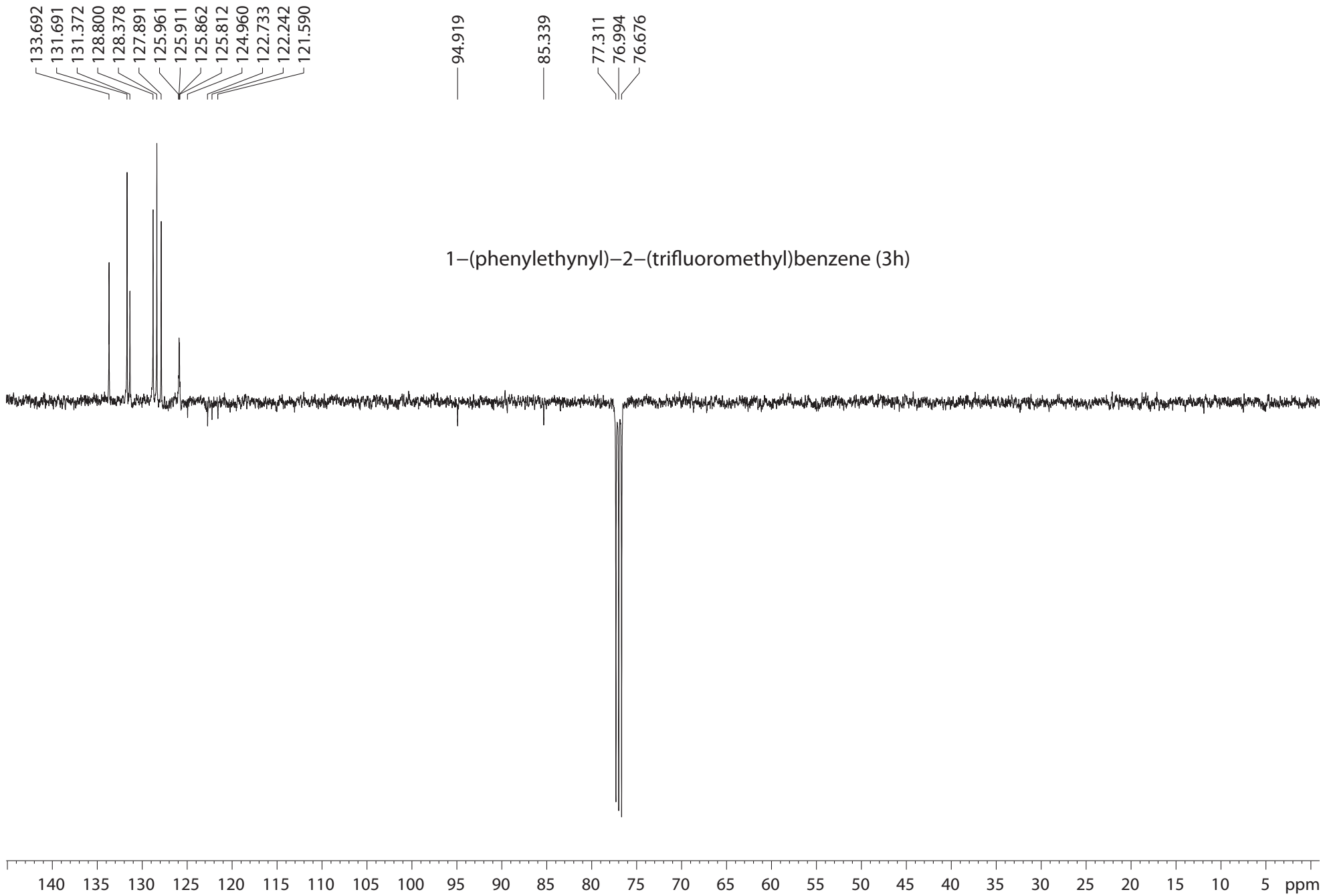




-62.704

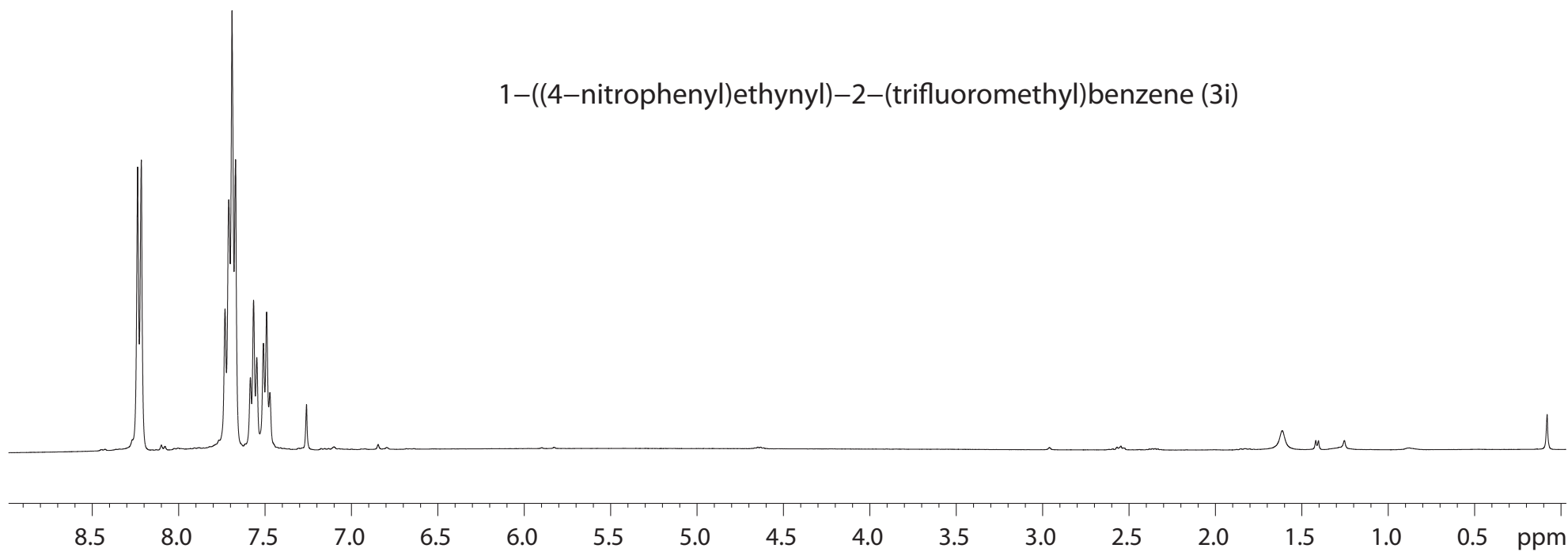
1-(phenylethynyl)-2-(trifluoromethyl)benzene (3h)





8.237
8.215
7.731
7.709
7.690
7.670
7.584
7.566
7.547
7.509
7.490
7.471
7.259

1-((4-nitrophenyl)ethynyl)-2-(trifluoromethyl)benzene (3i)

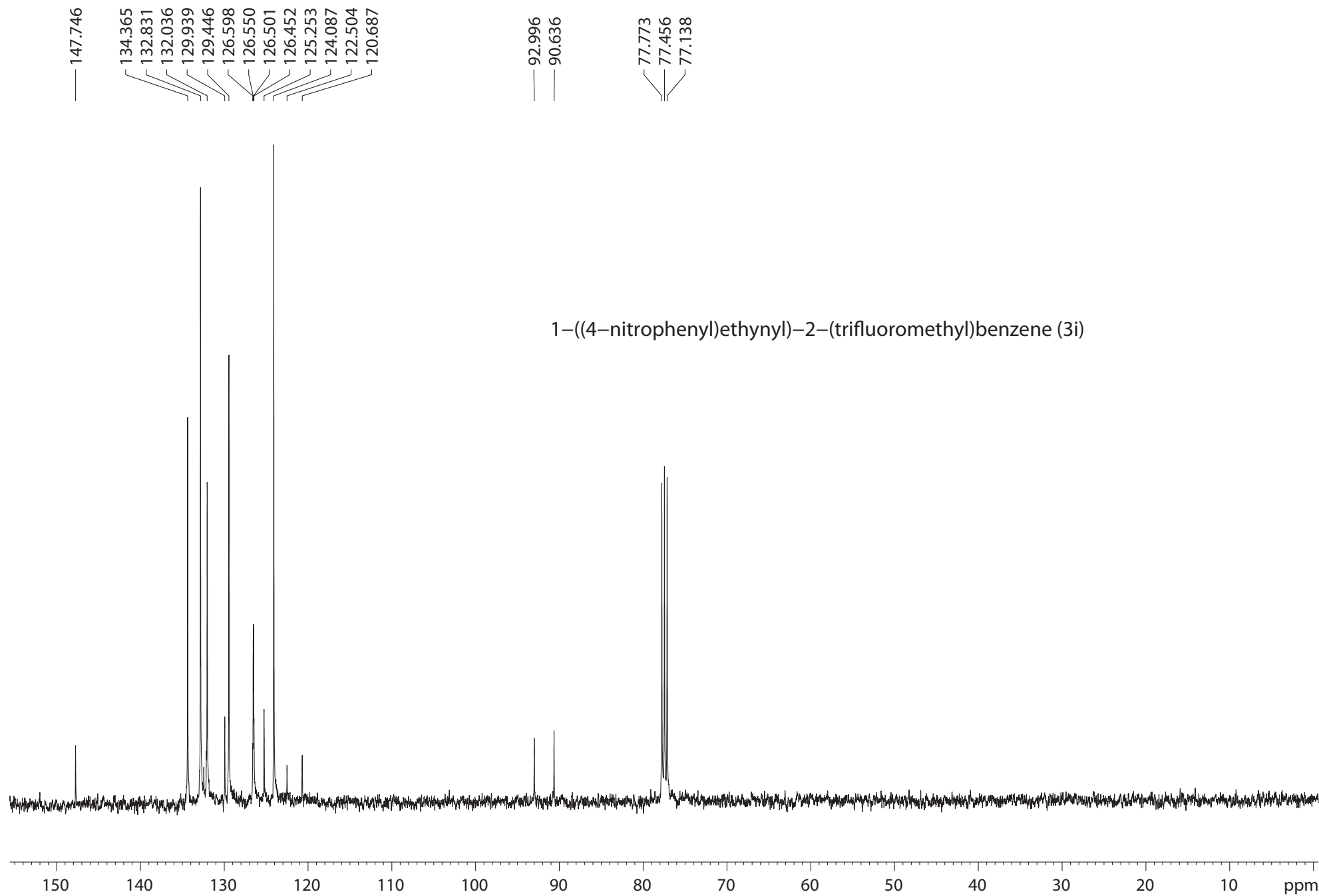


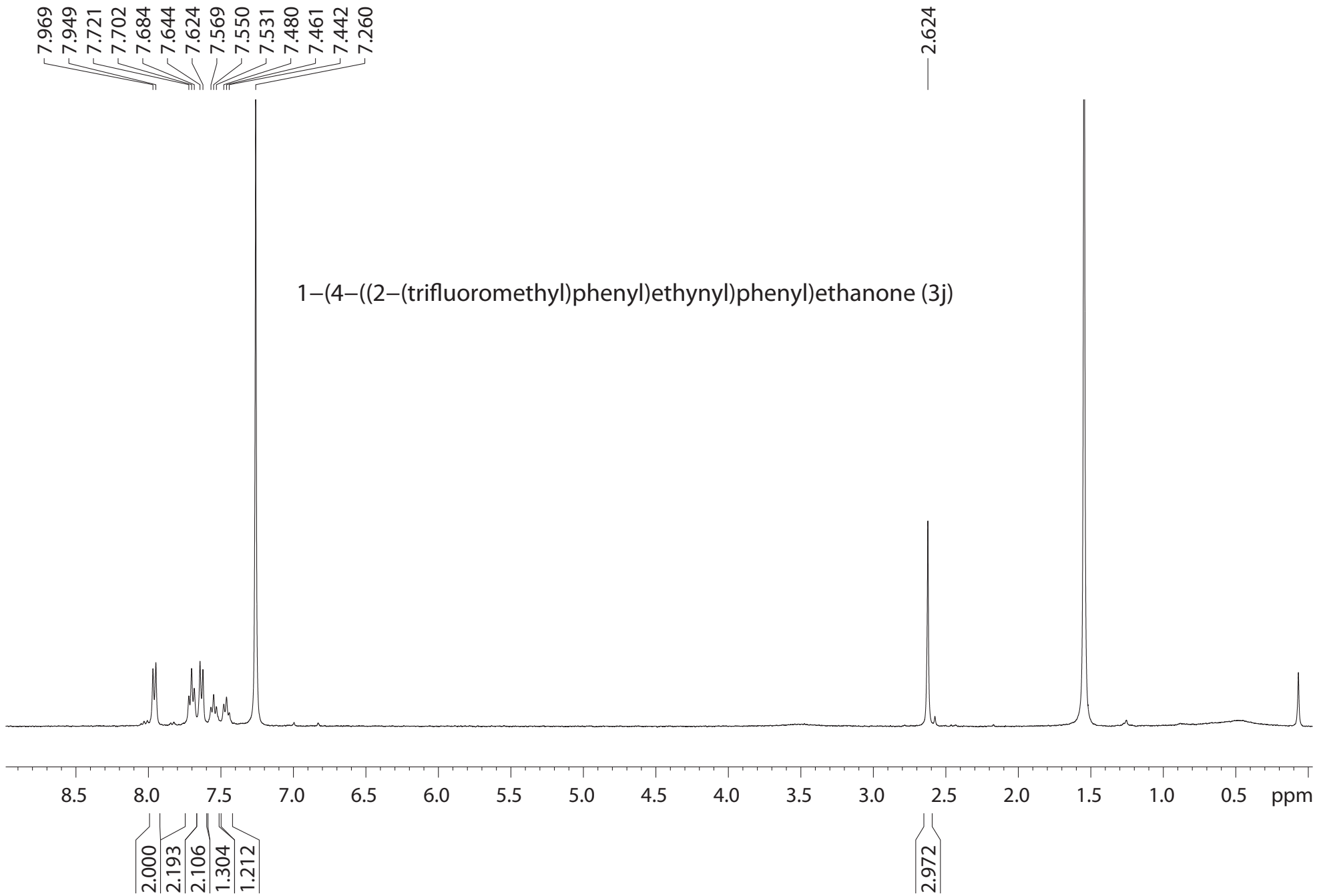
2.000
4.142
1.093
1.118

-62.626

1-((4-nitrophenyl)ethynyl)-2-(trifluoromethyl)benzene (3i)

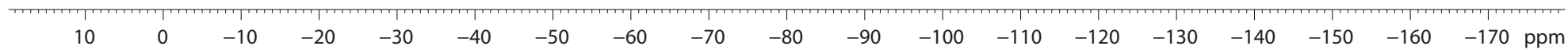


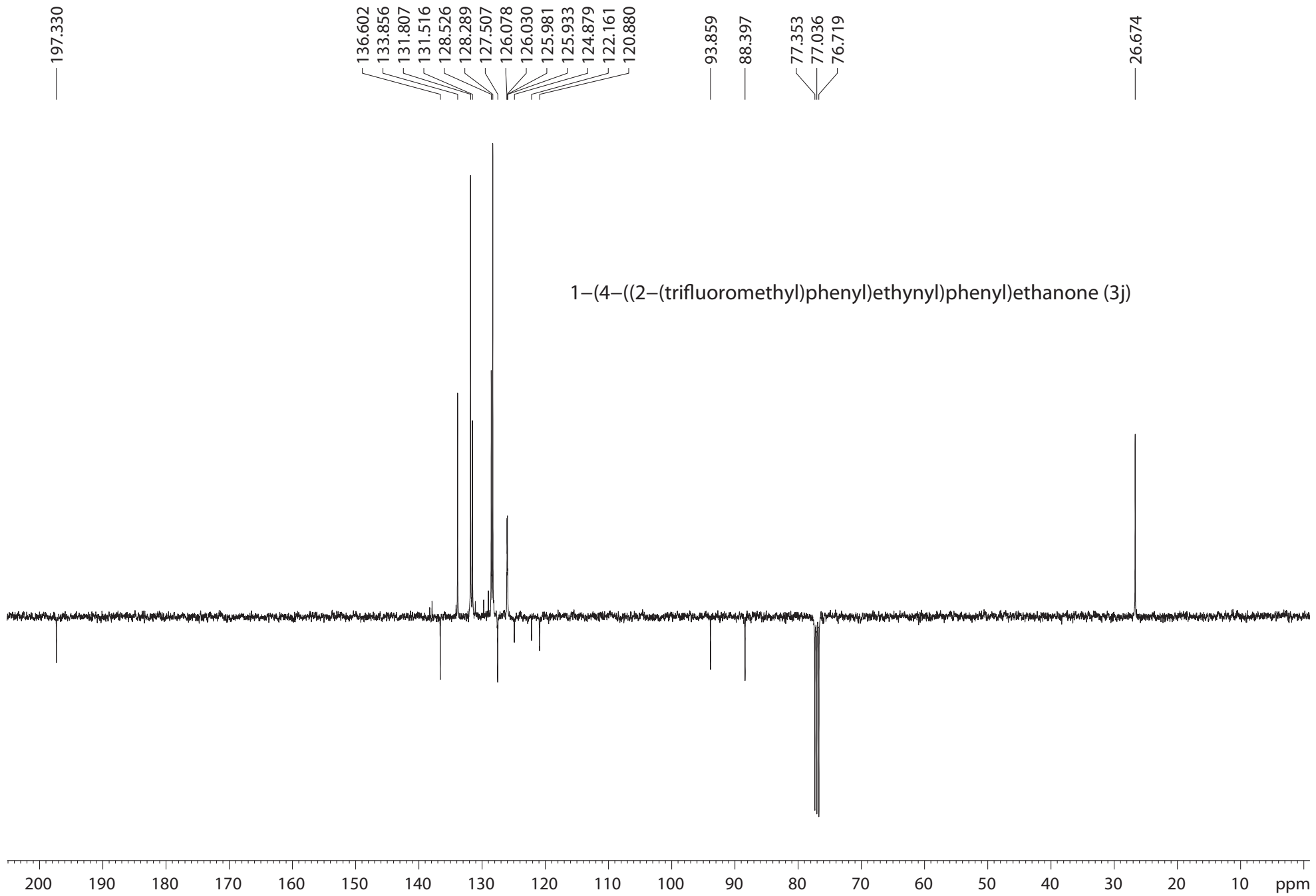




-62.699

1-(4-((2-(trifluoromethyl)phenyl)ethynyl)phenyl)ethanone (3j)

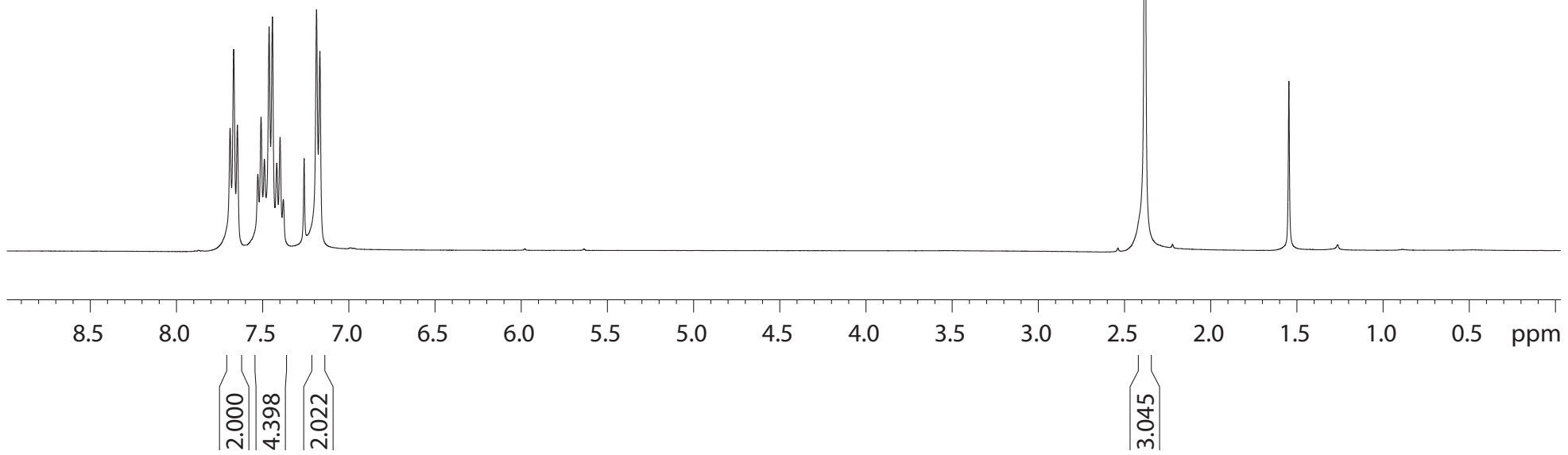




7.689
7.668
7.647
7.528
7.509
7.490
7.463
7.443
7.418
7.399
7.380
7.259
7.187
7.168

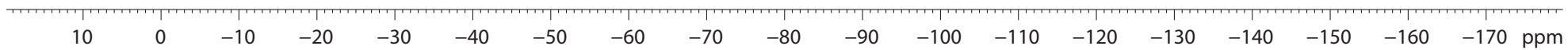
2.382

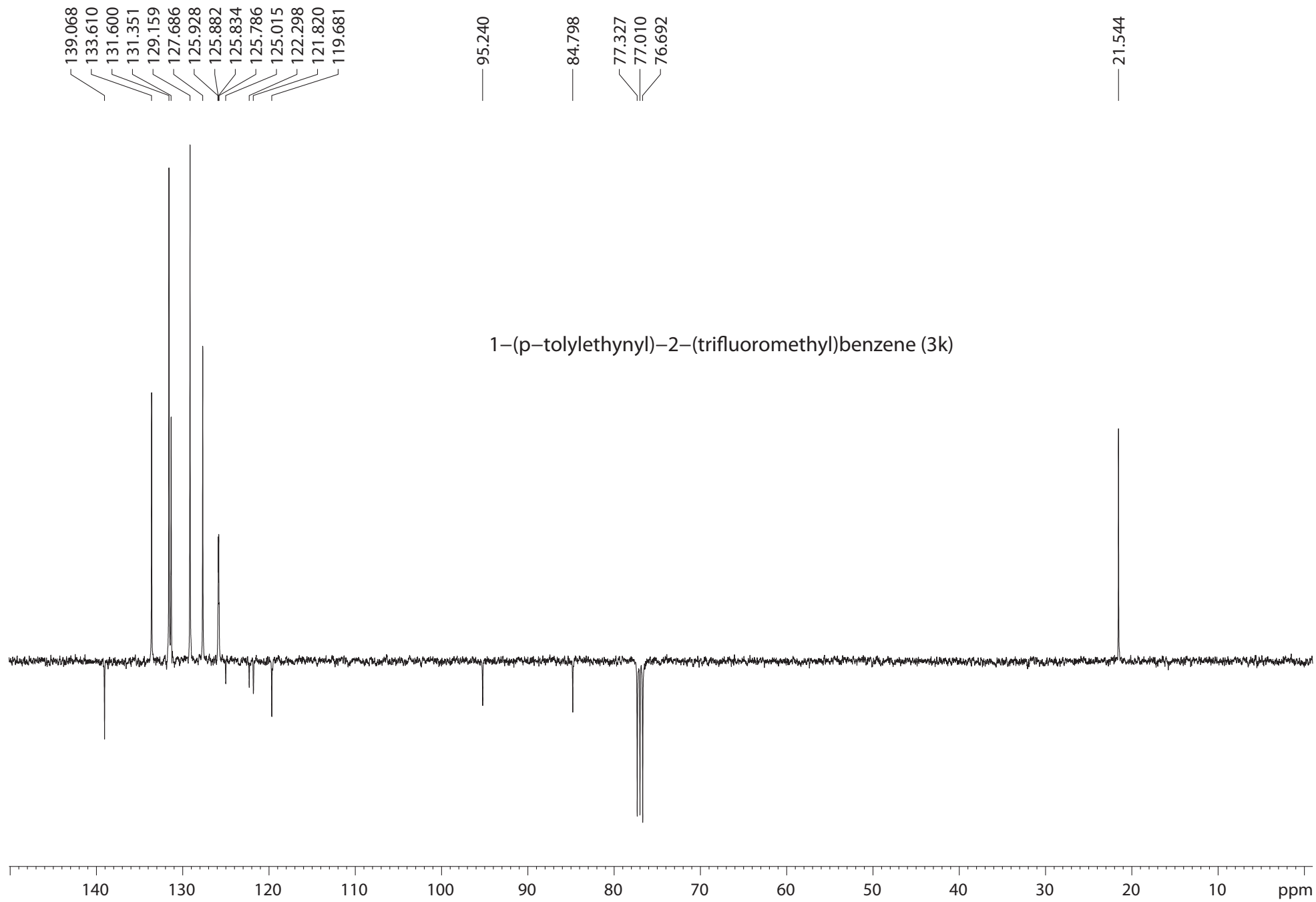
1-(p-tolylethynyl)-2-(trifluoromethyl)benzene (3k)

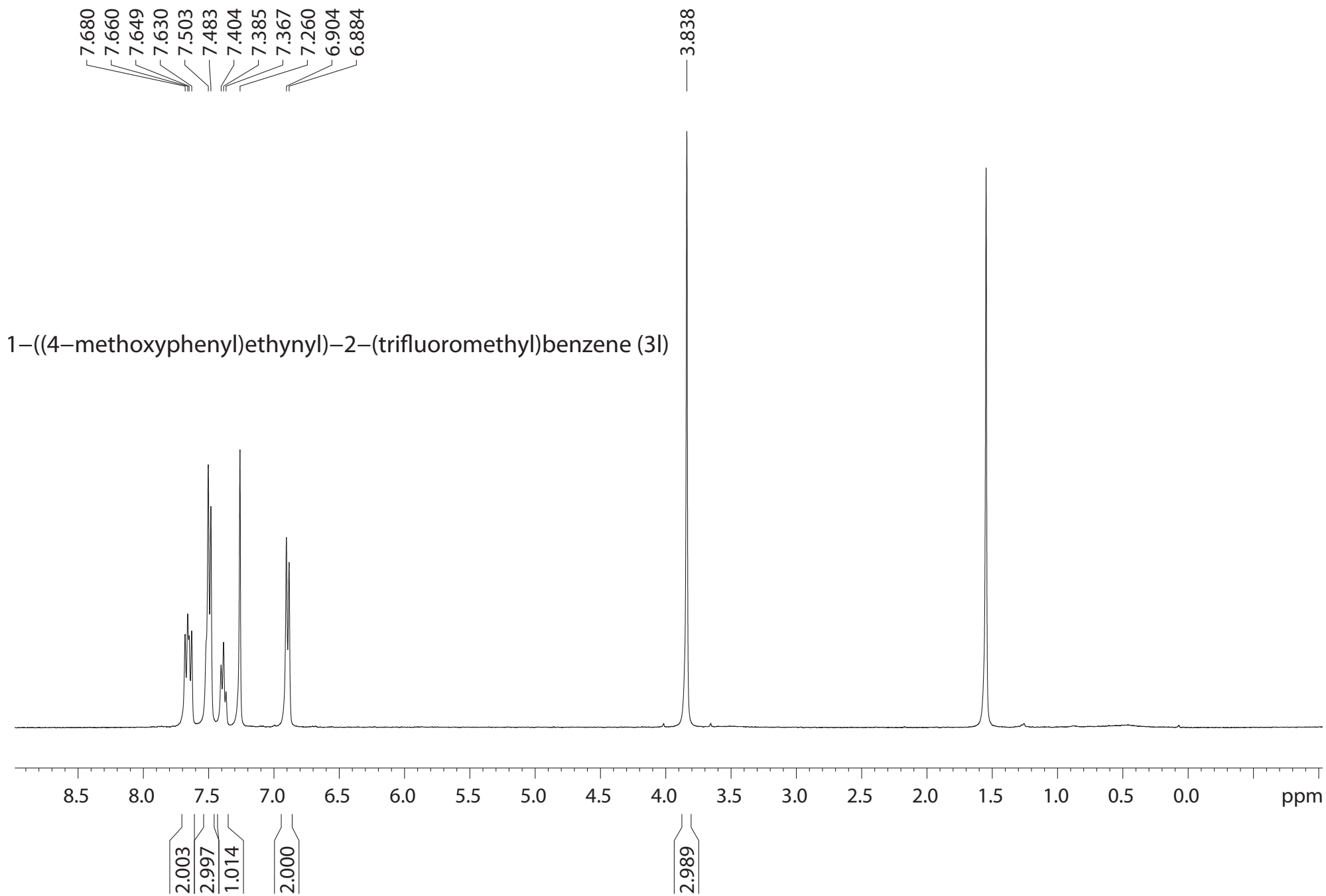


1-(p-tolylolethynyl)-2-(trifluoromethyl)benzene (3k)

-62.654

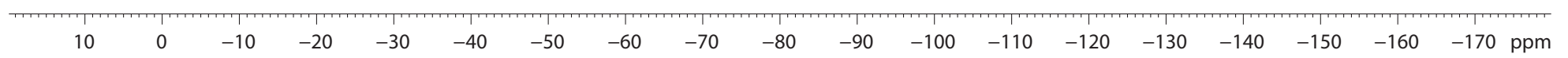


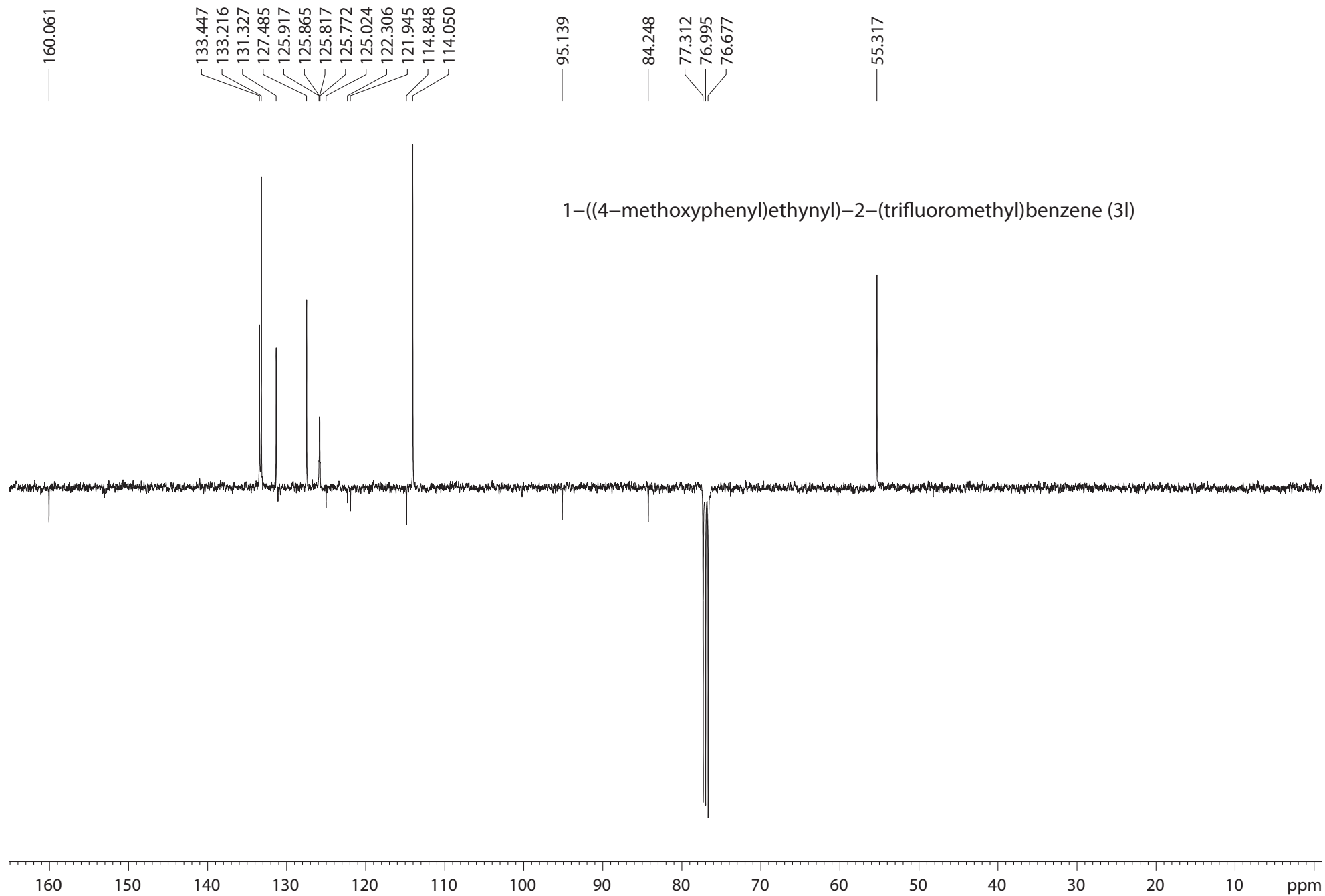


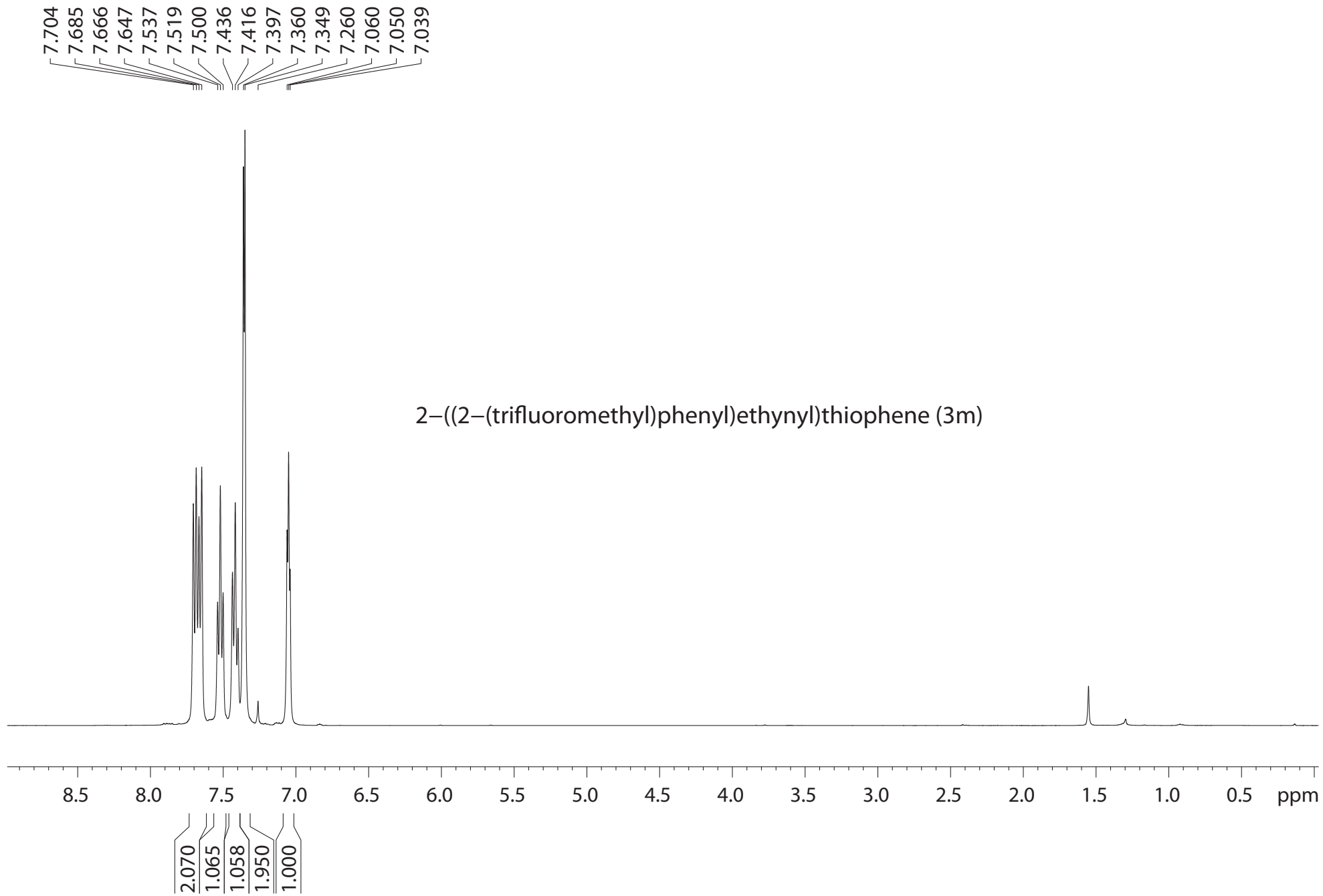


-62.867

1-((4-methoxyphenyl)ethynyl)-2-(trifluoromethyl)benzene (3l)







-62.721

2-((2-(trifluoromethyl)phenyl)ethynyl)thiophene (3m)

