Supporting Information

An Efficient, Regio- and Stereoselective Palladium Catalyzed Route to Tetrasubstituted Olefins

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General

^{1}H and ^{13}C NMR spectra were recorded at 300 and 75 MHz, respectively. COESY and NOESY NMR spectra were recorded at 400 MHz. Thin layer chromatography was performed using commercially prepared 60-mesh silica gel plates (Whatman K6F), and visualization was effected with short wavelength UV light (254 nm). Low resolution mass spectra were recorded on a Finnigan TSQ700 triple quadruple mass spectrometer (Finnigan MAT, San Jose, CA). High resolution mass spectra were recorded on a Kratos MS50TC double focusing magnetic sector mass spectrometer using EI at 70 eV. All reagents were used directly as obtained commercially. Anhydrous forms of K_2CO_3, $KHCO_3$, DMF, ethyl ether, hexanes, and ethyl acetate were purchased from Fisher Scientific Co.. All palladium salts were donated by Johnson Matthey Inc. and Kawaken Fine Chemicals Co., Ltd.. All boronic acids were donated by Frontier Scientific Co., Ltd..

General procedure for the palladium-catalyzed formation of tetrasubstituted olefins (Table 2)

DMF (8 mL), H_2O (2 mL), $KHCO_3$ (0.75 mmol), the ArI (0.50 mmol), the boronic acid (0.75 mmol), and the internal alkyne (0.25 mmol) were placed in a 6 dram vial. The contents were stirred and heated in a 100 ˚C oil bath for 10 min. The $PdCl_2(PhCN)_2$ catalyst (0.0025 mmol, in 0.1 mL of DMF) was injected. The vial was then heated in an oil bath at 100 ˚C until palladium black appeared (usually in 3-24 h). The reaction mixture was cooled, quenched with 30 mL of satd NaCl solution, and the aqueous layer was extracted with ethyl ether three times. The combined organic layer was dried over

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anhydrous $MgSO_4$ and the solvent was evaporated under reduced pressure. The product was isolated by chromatography on a silica gel column.

(*Z*)-2-(4-Methylphenyl)-1,1-diphenyl-1-propene (1a). White solid after recrystallization from hexane: mp 83-85 ºC (lit.[1] mp 84-85 ºC); ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.13 (s, 3H), 2.28 (s, 3H), 6.90-7.06 (m, 9H), 7.24-7.28 (m, 3H), 7.33-7.38 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 21.36, 23.60, 125.93, 126.72, 127.65, 128.33, 128.81, 129.40, 130.25, 131.09, 135.78, 135.99, 139.17, 141.19, 143.52, 143.98; IR ($CDCl_3$, cm^{-1}) 3023, 2922, 2856, 1509, and 1490; MS (EI) m/z (rel intensity) 284 (M^+, 100), 269 (38); HRMS (EI) calcd for $C_{22}H_{20}$ 284.1565, found: 284.1570.

(*Z*)-1-(4-Methylphenyl)-1,2-diphenyl-1-propene (1b). White solid after recrystallization from hexane: mp 93-95 ºC; ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.15 (s, 3H), 2.22 (s, 3H), 6.79-6.87 (m, 4H), 7.18-7.37 (m, 10H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 21.33, 23.65, 126.34, 126.74, 128.12, 128.34, 128.40, 129.53, 130.24, 130.97, 135.38, 135.58, 139.43, 140.33, 144.04, 144.50; IR ($CDCl_3$, cm^{-1}) 3023, 2922, 1509, and 1490; MS (EI) m/z (rel intensity) 284 (M^+, 100), 269 (46), 254 (17); HRMS (EI) calcd for $C_{22}H_{20}$ 284.1565, found: 284.1573.

1-Methyl-4-(triphenylethenyl)-benzene (3). White solid: mp 146-148 ºC (lit.[2] mp 146-148 ºC); ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.29 (s, 3H), 6.95-7.16 (m, 19H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 21.47, 126.55, 126.60, 127.87, 127.93, 128.65, 131.49, 131.58, 131.62, 136.31, 140.73, 141.01, 141.17, 144.18, 144.21, 144.22 (3 C are missing due to overlap); IR ($CDCl_3$, cm^{-1}) 3023, 2920, 1508, and 1492; MS (EI) m/z (rel intensity) 346 (M^+, 100); HRMS (EI) calcd for $C_{27}H_{22}$ 346.1722, found: 346.1728.

1-Methoxy-4-(triphenylethenyl)benzene (4). Light yellow solid: mp 132-134 ºC (lit. [3] mp 132-134 ºC); ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.75 (s, 3H), 6.66 (d, J = 8.7 Hz, 2H), 6.96 (d, J = 8.7 Hz, 2H), 7.04-7.15 (m, 15H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 55.32, 113.28, 126.47, 126.48, 126.59, 127.84, 127.96, 129.29, 131.58, 131.60, 131.63, 132.77, 136.35, 140.33, 140.76, 144.22, 144.23, 144.28, 158.32; IR ($CDCl_3$, cm^{-1}) 3055, 3019, 2954, 2836, 1508, 1491; MS (EI) m/z (rel intensity) 362 (M^+, 100); HRMS (EI) calcd for $C_{27}H_{22}O$ 362.1671, found: 362.1677.

1-Chloro-4-(triphenylethenyl)benzene (5). White solid: mp 165-167 ºC; ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.95-7.15 (m, 19H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 126.82, 126.88, 126.92, 127.93, 128.02, 128.11, 128.15, 131.48, 131.50, 131.53, 132.42, 132.88, 139.88, 141.82, 142.46, 143.53, 143.60, 143.66; IR ($CDCl_3$, cm^{-1}) 3055, 3025 and 1491; MS (EI) m/z (rel intensity) 366 (M^+, 100); HRMS (EI) calcd for $C_{26}H_{19}Cl$ 366.1175, found: 366.1181.

(*Z*)-1-(3,5-Difluorophenyl)-2-(4-methoxyphenyl)-1,2-diphenylethylene (6). White solid: mp 131-133 ºC; ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.78 (s, 3H), 6.57-6.60 (m, 3H), 6.71 (d, *J* = 8.7 Hz, 2H), 6.96 (d, *J* = 8.7 Hz, 2H), 7.00-7.13 (m, 10H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 55.37, 101.77, 102.11, 102.45, 113.61, 114.15, 114.25, 114.38, 114.48, 126.96, 127.04, 127.93, 128.12, 128.17, 128.24, 131.41, 131.48, 132.52, 132.70, 135.38, 138.07, 142.59, 142.99, 143.44, 147.73, 158.86, 161.06, 161.24, 164.35, 164.52 (the more ^{13}C peaks are due to ^{19}F splitting); IR ($CDCl_3$, cm^{-1}) 3021, 2930, 2837, 1618, 1592, and 1508; MS (EI) m/z (rel intensity) 398 (M^+, 100); HRMS (EI) calcd for $C_{27}H_{20}F_2O$ 398.1482, found: 398.1490.

(*Z*)-2-(4-Methylphenyl)-1,1-diphenyl-1-butene (7a).[4] White solid after recrystallization from hexane: mp 114-116 ºC; ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.94 (t, *J* = 7.5 Hz, 3H), 2.28 (s, 3H), 2.48 (q, *J* = 7.5 Hz, 2H), 6.88-7.03 (m, 9H), 7.23-7.37 (m, 5H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 13.88, 21.39, 29.20, 125.82, 126.71, 127.56, 128.33, 128.75, 129.70, 129.76, 130.98, 135.86, 138.66, 139.24, 142.31, 143.43, 143.94; IR ($CDCl_3$, cm^{-1}) 3033, 2977, 1490, and 1442; MS (EI) m/z (rel intensity) 298 (M^+, 100), 283 (23); HRMS (EI) calcd for $C_{23}H_{22}$ 298.1722, found: 298.1726.

3-(4-Methylphenyl)-4,4-diphenyl-3-buten-2-one (8a) and (*E*)-4-(4-methylphenyl)-3,4-diphenyl-3-buten-2-one (8b). Yellow solid (mixture); ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.09 (2 sets of singlets, 3H), 2.28 (2 sets of singlets, 3H), 6.88-7.35 (m, 14H); IR ($CDCl_3$, cm^{-1}) 3023, 2922, 1683, 1509, 1490, and 1443; MS (EI) m/z (rel intensity) 312 (100), 311(88), 297 (45) and 269 (65); HRMS (EI) calcd for $C_{23}H_{20}O$ 312.1514, found: 312.1522.

Ethyl 2-(4-methylphenyl)-3,3-diphenylpropenoate (9a) and ethyl (*E*)-3-(4-methylphenyl)-2,3-diphenylpropenoate (9b). Yellow solid (mixture); ^{1}H NMR (300

MHz, $CDCl_3$) δ 0.97 (2 sets of triplets, *J* = 7.2 Hz, 3H), 2.28 (2 sets of singlets, 3H), 4.02 (2 sets of quartets, *J* = 7.2 Hz, 2H), 6.89-7.32 (m, 14H); IR ($CDCl_3$, cm^{-1}) 3023, 2985, 1717, 1508, 1490, and 1443; MS (EI) m/z (rel intensity) 342 (M^+, 14), 252 (100), 207 (27), 179 (45), 178 (53); HRMS (EI) calcd for $C_{24}H_{22}O_2$ 342.1620, found: 342.1629.

(*E*)-1-(4-Trifluoromethylphenyl)-1-(4-methylphenyl)-2-phenylpropene (10a). White solid after recrystallization from ethanol: mp 105-107 ºC; ^1H NMR (400 MHz, $CDCl_3$) δ 2.14 (s, 3H), 2.23 (s, 3H), 6.77 (d, *J* = 8.4 Hz, 2H), 6.87 (d, *J* = 8.4 Hz, 2H), 7.16-7.22 (m, 5H), 7.38 (d, *J* = 8.0 Hz, 2H), 7.62 (d, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.34, 23.58, 125.30, 125.34, 125.37, 125.41, 125.91, 126.67, 128.21, 128.60, 128.68, 129.40, 130.56, 130.90, 136.04, 136.66, 138.20, 139.55, 143.86, 147.71, 147.73 (the extra peaks are due to ^{19}F splitting); IR ($CDCl_3$, cm^{-1}) 3023, 2920, 1615, 1509, 1490, 1441, and 1325; MS (EI) m/z (rel intensity) 352 (M^+, 100), 351 (62), 337 (31), 336 (31), 322 (18); HRMS (EI) calcd for $C_{23}H_{19}F_3$ 352.1439, found: 352.1444.

(*E*)-1-(4-Methylphenyl)-2-phenyl-1-(5-pyrimidyl)propene (11a). White solid, recrystallized from 40:1 hexane/ethyl acetate: mp 109-111 ºC; ^1H NMR (400 MHz, $CDCl_3$) δ 2.18 (s, 3H), 2.22 (s, 3H), 6.73 (d, *J* = 8.0 Hz, 2H), 6.87 (d, *J* = 8.0 Hz, 2H), 7.14-7.20 (m, 5H), 8.64 (s, 2H), 9.13 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.33, 23.57, 127.11, 128.33, 128.93, 129.20, 130.86, 132.39, 136.66, 137.55, 138.41, 139.46, 143.00, 156.59, 157.84; IR ($CDCl_3$, cm^{-1}) 3022, 2920, 1547, 1510, 1442, and 1409; MS (EI) m/z (rel intensity) 286 (M^+, 100), 271 (32), 215 (59), 213 (31); HRMS (EI) calcd for $C_{20}H_{18}N_2$ 286.1470, found: 286.1476.

(*E*)-1-(4-Methylphenyl)-1-(4-nitrophenyl)-2-phenylpropene (12a). Yellow solid, recrystallized from 40:1 hexane/ethyl acetate: mp 136-138 ºC; ^1H NMR (400 MHz, $CDCl_3$) δ 2.15 (s, 3H), 2.22 (s, 3H), 6.73 (d, *J* = 8.0 Hz, 2H), 6.86 (d, *J* = 8.0 Hz, 2H), 7.15-7.20 (m, 5H), 7.42 (d, *J* = 8.8 Hz, 2H), 8.21 (d, *J* = 8.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.32, 23.59, 123.75, 126.92, 128.27, 128.76, 129.33, 130.91, 131.18, 136.40, 137.63, 137.71, 139.10, 143.46, 146.66, 151.03; IR ($CDCl_3$, cm^{-1}) 3022, 2920, 1592, 1515, and 1491; MS (EI) m/z (rel intensity) 329 (M^+, 100), 314 (17); HRMS (EI) calcd for $C_{22}H_{19}NO_2$ 329.1416, found: 329.1423.

(*E*)-2-(4-Methylphenyl)-1-(4-nitrophenyl)-1-phenylpropene (12b). Yellow solid, recrystallized from 10:1 hexane/acetone: mp 141-143 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.15 (s, 3H), 2.29 (s, 3H), 6.86-6.89 (m, 2H), 7.00-7.09 (m, 7H), 7.41 (d, *J* = 8.8 Hz, 2H), 8.22 (d, *J* = 8.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.37, 23.56, 123.75, 126.62, 128.04, 128.96, 129.24, 131.05, 131.20, 136.72, 137.35, 138.18, 140.19, 142.30, 146.63, 150.97; IR ($CDCl_3$, cm^{-1}) 3023, 2921, 1595, 1515, and 1493; MS (EI) m/z (rel intensity) 329 (M^+, 100), 314 (15); HRMS (EI) calcd for $C_{22}H_{19}NO_2$ 329.1416, found: 329.1423.

(*Z*)-1-(4-Methoxyphenyl)-1,2-diphenyl-1-butene (13a).[5] White solid, recrystallized from hexane: mp 116-118 ºC (lit. mp 116-119 ºC); ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.96 (t, *J* = 7.5 Hz, 3H), 2.48 (q, *J* = 7.5 Hz, 2H), 3.69 (s, 3H), 6.56 (d, *J* = 8.7 Hz, 2H), 6.80 (d, *J* = 8.7 Hz, 2H), 7.14-7.20 (m, 10H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 13.87, 29.30, 55.24, 113.01, 126.28, 126.78, 128.14, 128.36, 129.72, 129.96, 132.14, 135.72, 138.50, 141.56, 142.69, 144.10, 157.74; IR ($CDCl_3$, cm^{-1}) 3019, 2969, 2932, 2871, 2834, 1606, 1508, 1492, and 1441; MS (EI) m/z (rel intensity) 314 (M^+, 100), 299 (35), 284 (19); HRMS (EI) calcd for $C_{23}H_{22}O$ 314.1671, found: 314.1680.

References

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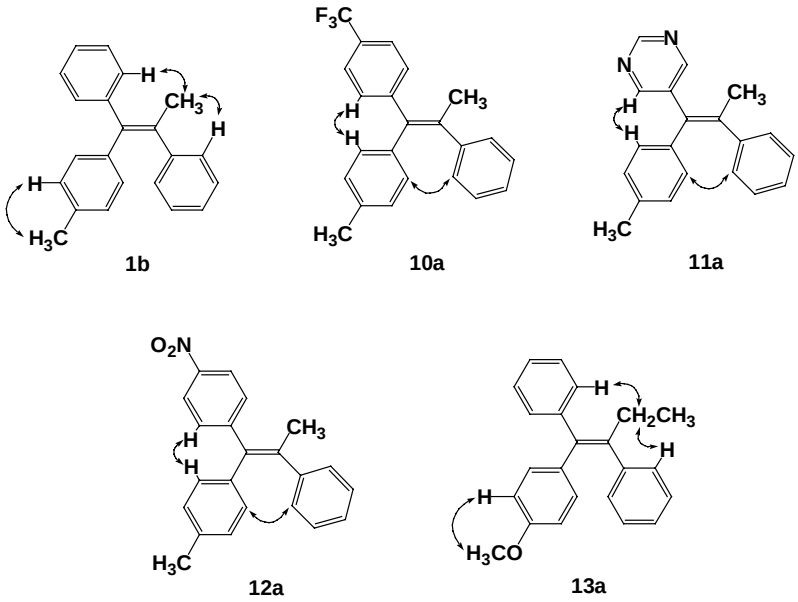

Figure 1. NOESY H-H interaction

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7.063
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7.050
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7.035
7.031
7.026
7.016
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6.933
6.923
6.917
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6.901
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H3C
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3
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6
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8
9
10
11
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2.972
3.000

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60
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20
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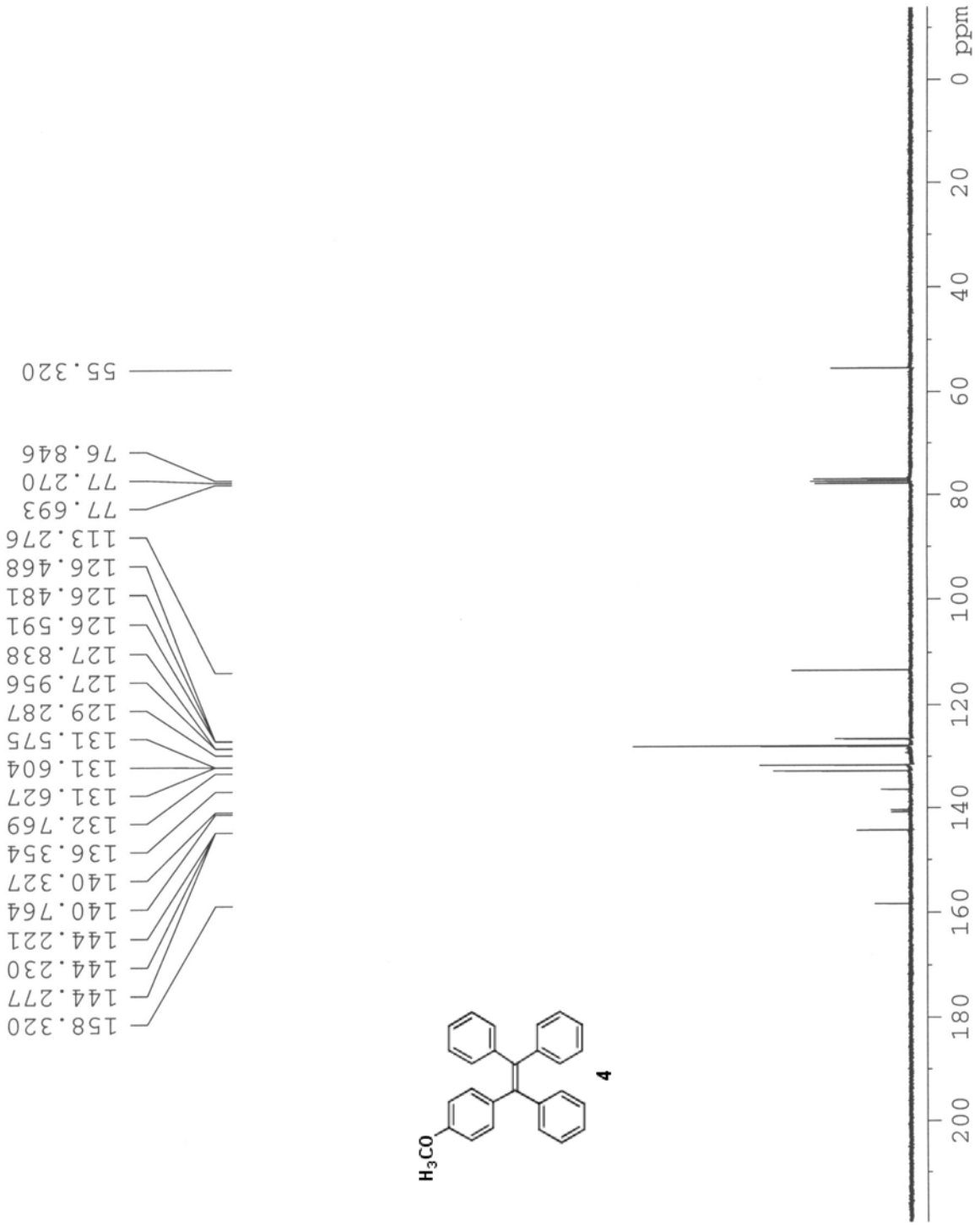
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40
60
80
100
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140
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180
200

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OCH3
F
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60
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100
120
140
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200
H3C
H3CH2C
7a

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7.199
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7.150
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7.025
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6.999
6.993
6.954
6.947
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6.883
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2.075
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COCH3
8b
+
H3C
H3COC
8a
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ppm
2.4
2.2
ppm

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7.186
7.180
7.170
7.159
7.154
7.142
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7.124
7.117
7.035
7.030
7.020
7.017
7.010
7.003
6.991
6.919
6.891
4.057
4.033
4.022
4.009
3.999
3.985
2.280
2.264
0.989
0.975
0.965
0.952
0.941
0.928
4.1
ppm
H3C
C2H5O2C
9a
+
H3C
CO2C2H5
9b
ppm
11
10
9
8
7
6
5
4
3
2
1
0
4.096
9.835
2.108
3.000
3.046

7.638
7.618
7.404
7.385
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7.197
7.191
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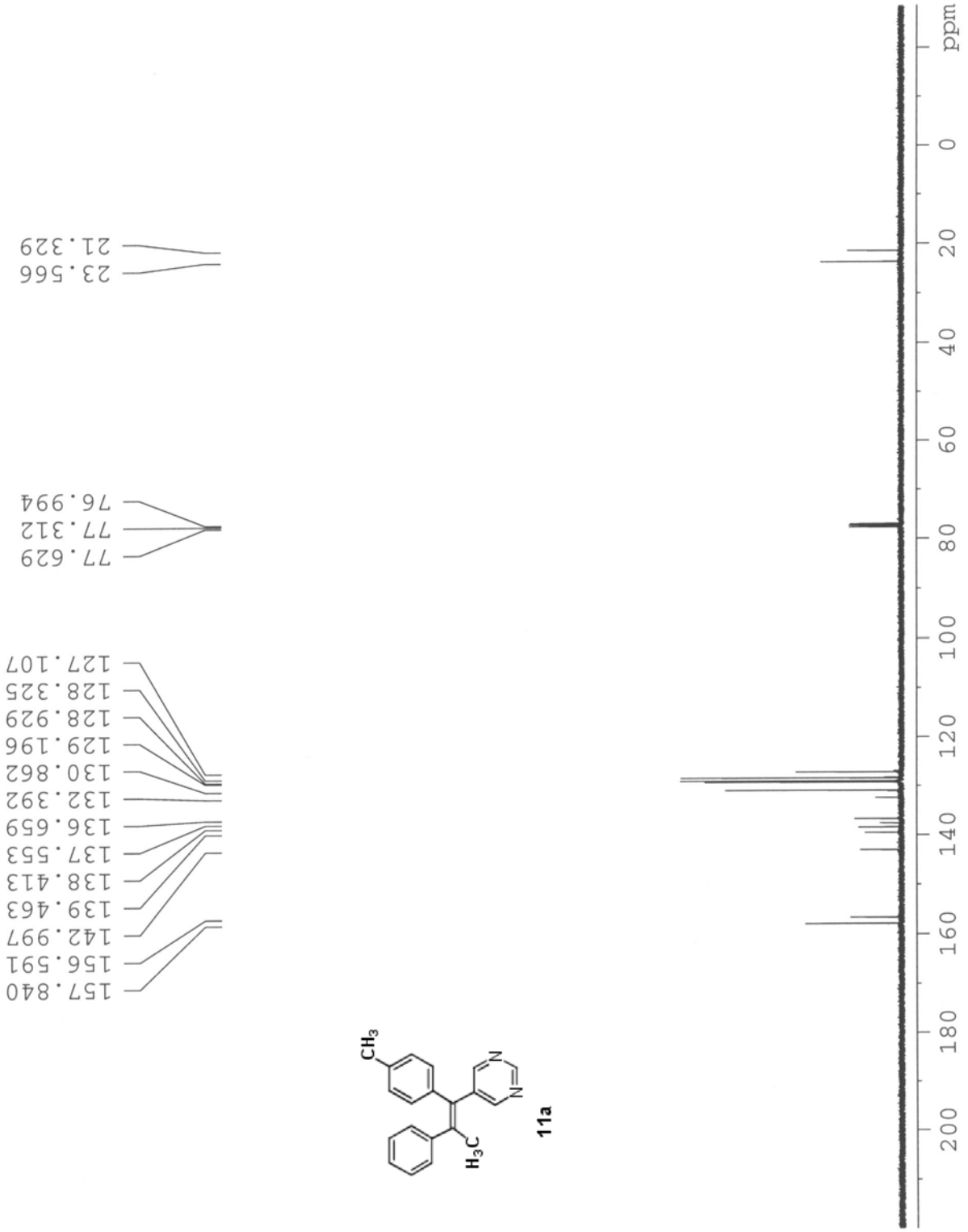
7.175
7.169
7.159
6.888
6.868
6.787
6.766
2.232
2.144
1.980
2.024
4.981
1.992
1.973
3.000
2.901
ppm
0
1
2
3
4
5
6
7
8
9
10
11
12
13
CH3
CF3
H3C
10a

147.728
147.714
143.854
139.545
138.196
136.659
136.043
130.897
130.559
129.399
128.679
128.595
128.210
126.669
125.412
125.374
125.337
125.299
77.596
77.279
76.961
23.586
21.345
CH3
CF3
H3C
10a
ppm
200
180
160
140
120
100
80
60
40
20
0

9.131
8.639
7.265
7.201
7.196
7.185
7.174
7.170
7.159
7.154
7.143
7.139
6.891
6.872
6.747
6.727
2.221
2.179
CH3
H3C
N
N
11a
0.934
1.867
5.065
1.947
1.911
2.922
3.014
ppm
0
1
2
3
4
5
6
7
8
9
10
11
12
13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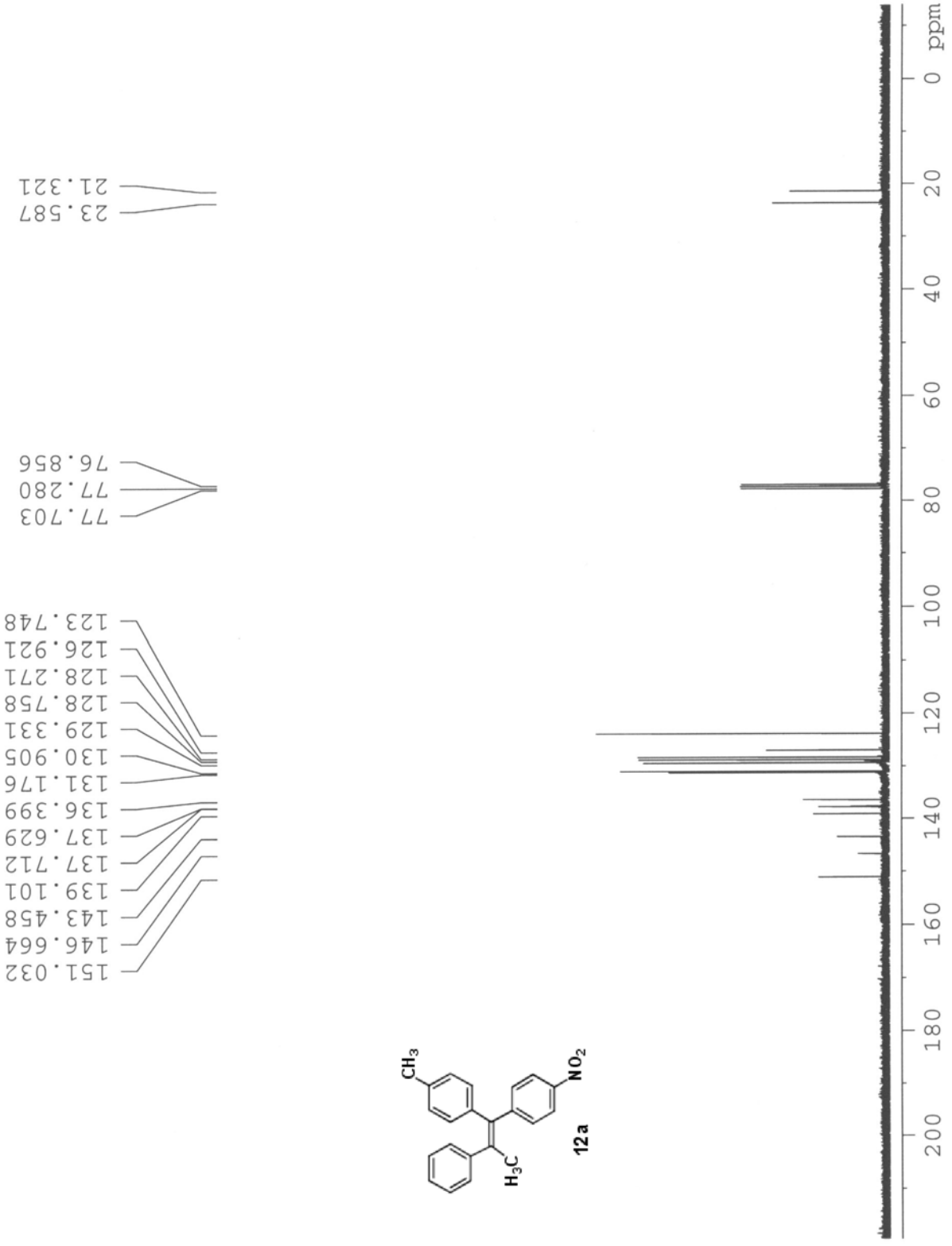
156.591
142.997
139.463
138.413
137.553
136.659
132.392
130.862
129.196
128.929
128.325
127.107
77.629
77.312
76.994
23.566
21.329
ppm
0
20
40
60
80
100
120
140
160
180
200
CH3
H3C
N
N
11a

2.148
2.223
6.729
6.749
6.862
6.882
7.150
7.155
7.157
7.162
7.168
7.171
7.187
7.200
7.202
7.265
7.415
7.420
7.437
8.209
8.213
8.231
CH3
NO2
H3C
12a
ppm
0
1
2
3
4
5
6
7
8
9
10
11
12
13
3.000
3.073
2.002
2.025
5.093
2.059
1.997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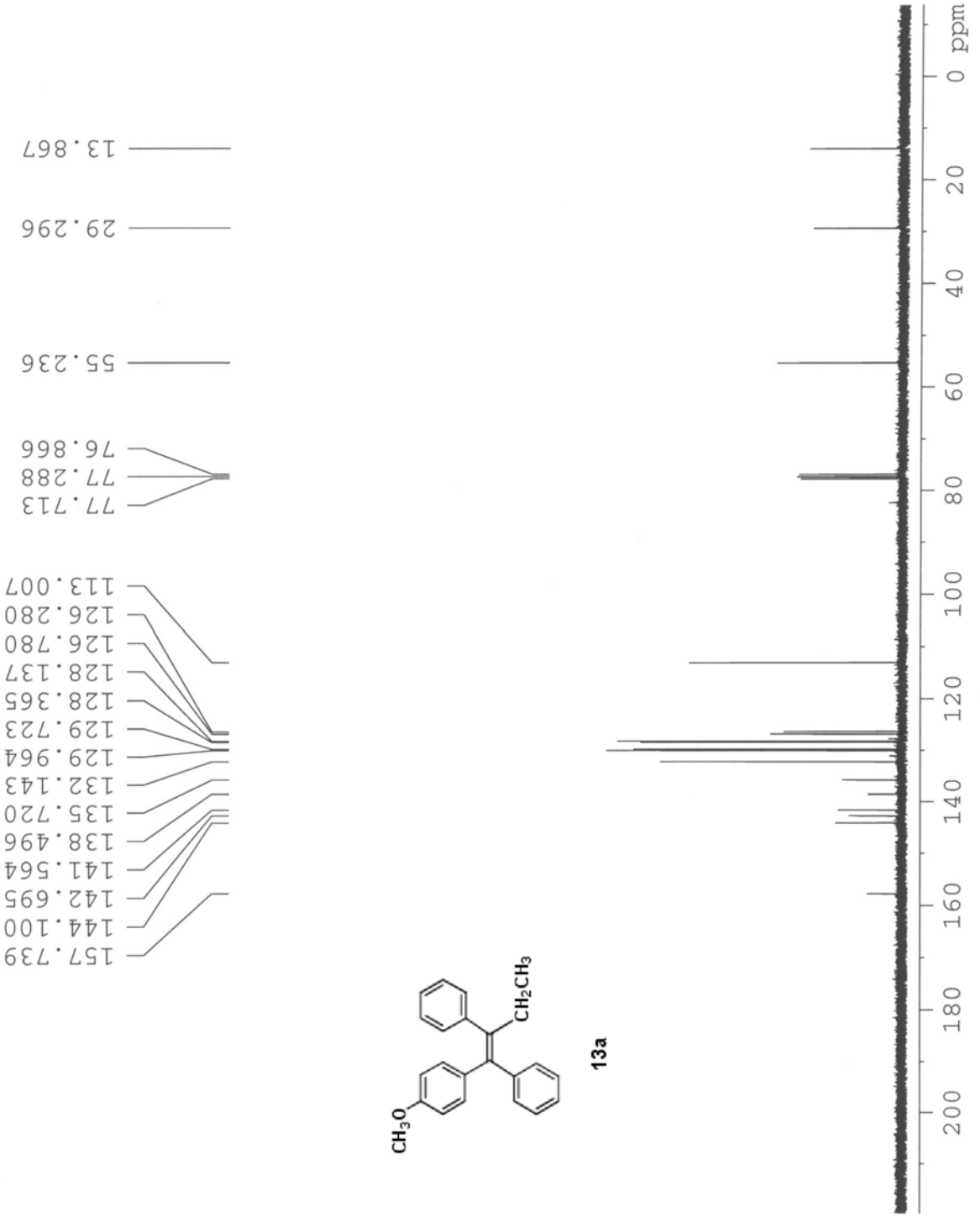
146.664
143.458
139.101
137.712
137.629
136.399
131.176
130.905
129.331
128.758
128.271
126.921
123.748
77.703
77.280
76.856
23.587
21.321
CH3
NO2
H3C
12a
0
20
40
60
80
100
120
140
160
180
200
ppm

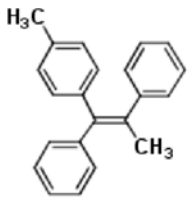
2.148
2.286
8.233
8.227
8.211
8.204
7.439
7.432
7.416
7.409
7.265
7.087
7.085
7.080
7.074
7.065
7.058
7.053
7.037
7.030
7.006
6.979
6.892
6.889
6.884
6.880
6.876
6.868
6.860
H3C
H3C
NO2
12b
ppm
11
10
9
8
7
6
5
4
3
2
1
0
1.951
2.059
7.082
1.923
3.000
3.035

150.965
146.633
142.302
140.188
138.179
137.347
136.721
131.197
131.048
129.235
128.955
128.038
126.621
123.752
77.689
77.265
76.841
23.555
21.372
0 ppm
20
40
60
80
100
120
140
160
180
200
NO_2
H_3C
H_3C
12b

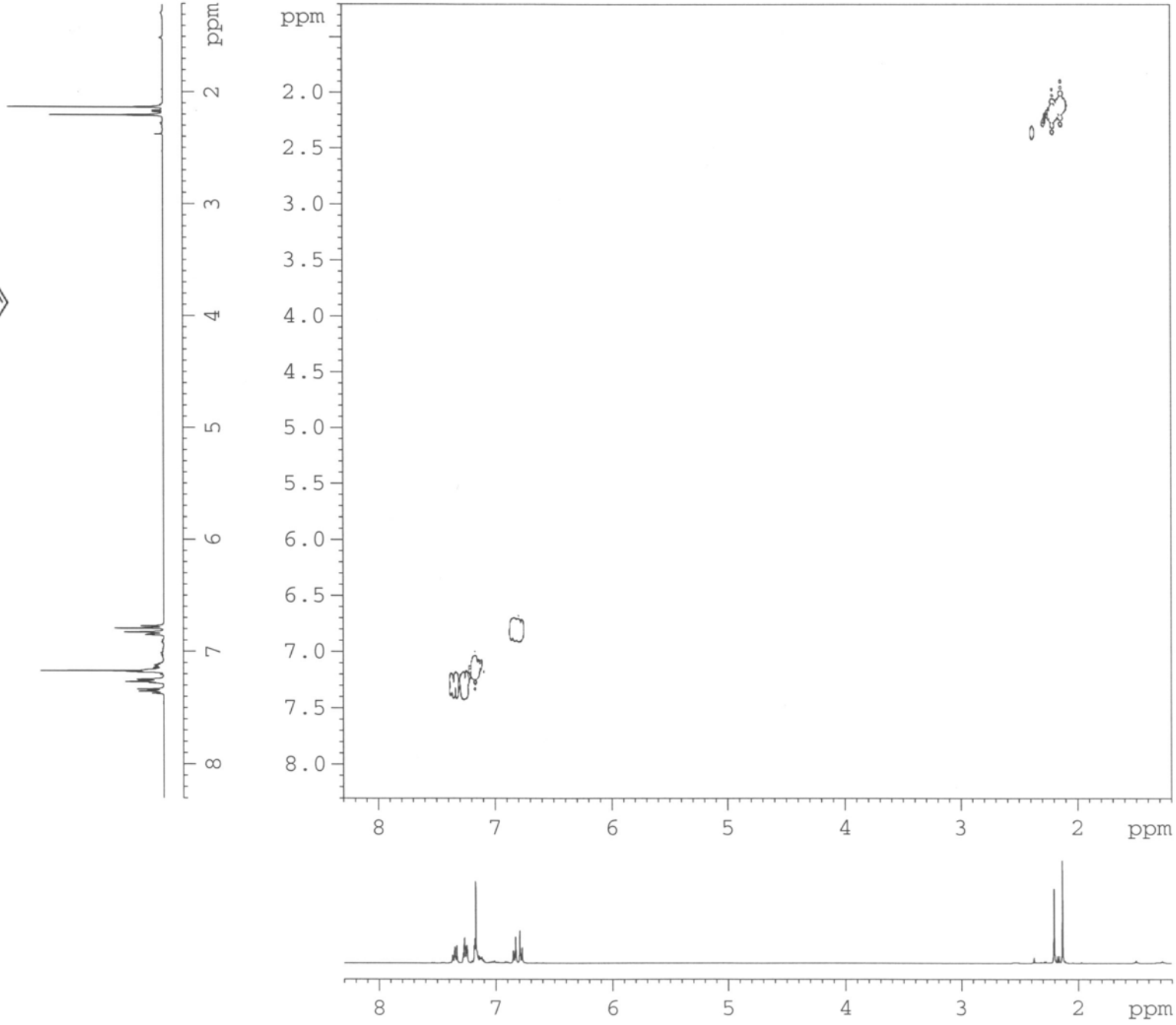
7.374
7.369
7.358
7.352
7.349
7.313
7.309
7.291
7.285
7.270
7.265
7.260
7.210
7.204
7.197
7.187
7.170
7.162
7.153
7.147
7.141
7.136
6.833
6.826
6.810
6.803
6.591
6.584
6.569
6.562
3.695
2.531
2.506
2.481
2.456
0.984
0.960
0.935
CH3O
CH2CH3
13a
ppm
0
1
2
3
4
5
6
7
8
9
10
11
9.953
1.916
1.927
3.000
2.046
3.069


13.867
29.296
55.236
76.866
77.288
77.713
113.007
126.280
126.780
128.137
128.365
129.723
129.964
132.143
135.720
138.496
141.564
142.695
144.100
157.739
CH2CH3
CH3O
13a
0 ppm
20
40
60
80
100
120
140
160
180
200

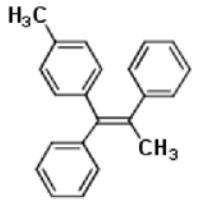
COESY


H3C
CH3

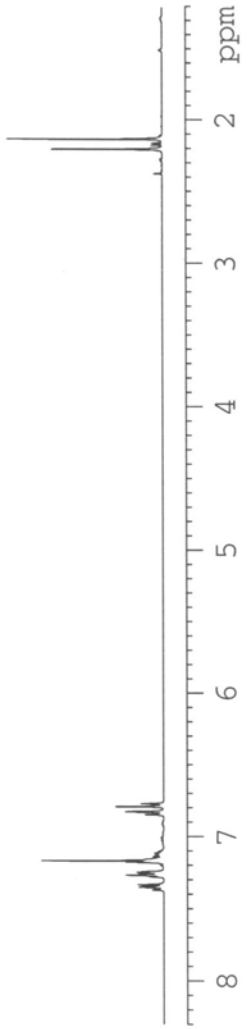
1b

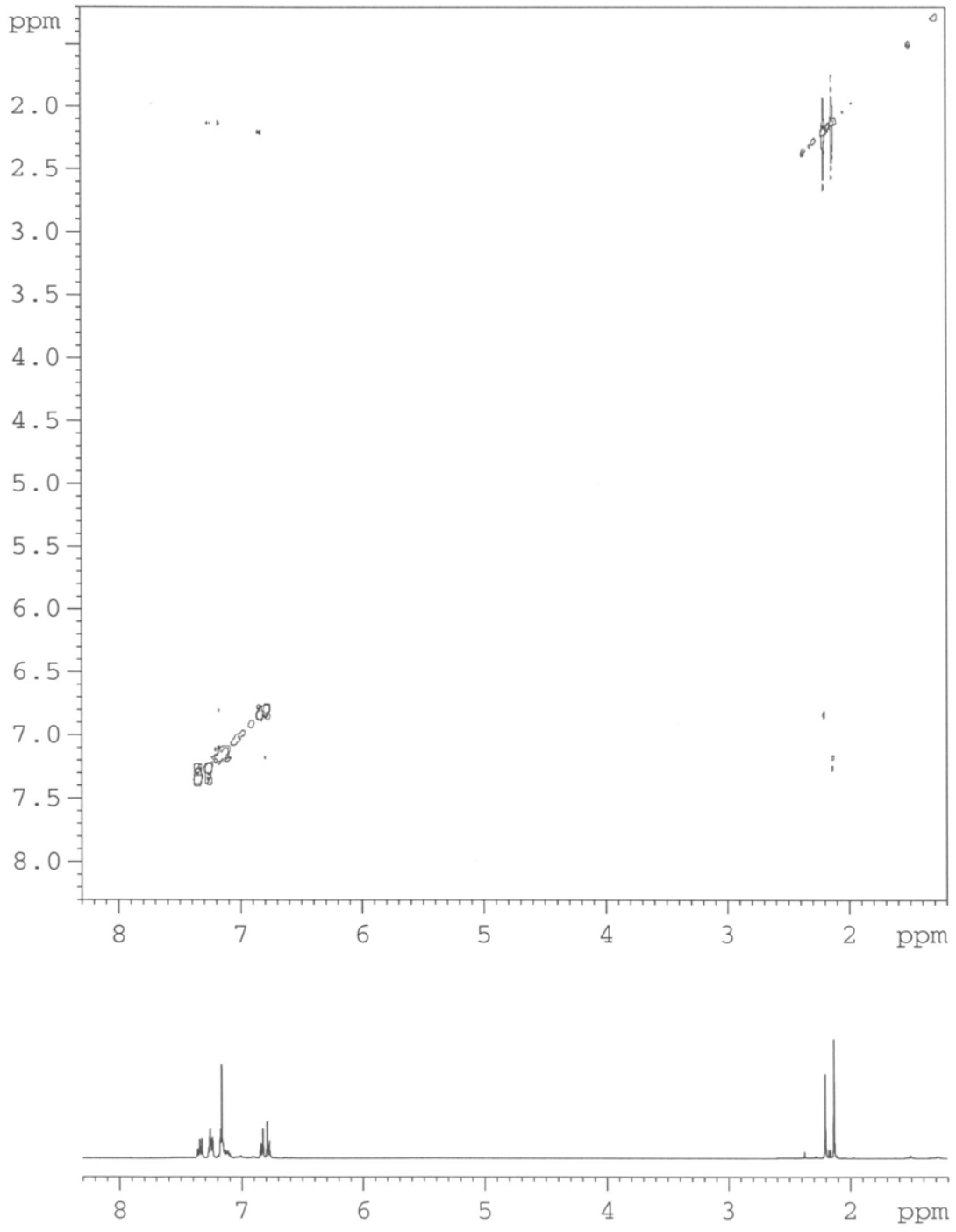

ppm
2
3
4
5
6
7
8
ppm
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8
7
6
5
4
3
2
ppm
8
7
6
5
4
3
2
ppm

NOESY


H3C
CH3

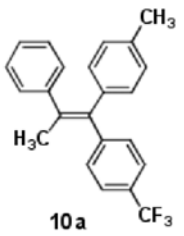
1b


ppm
2
3
4
5
6
7
8

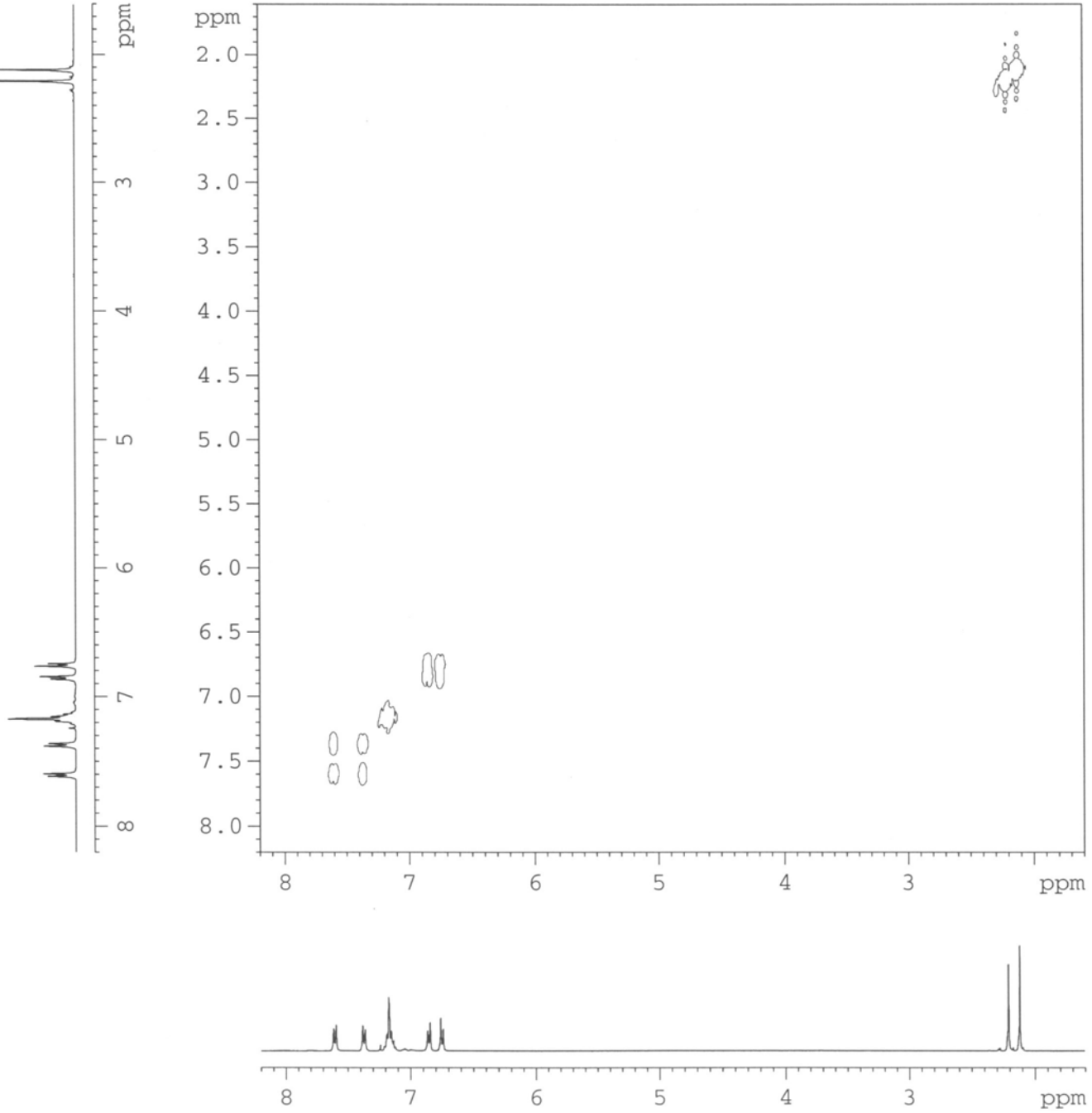

ppm
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8
7
6
5
4
3
2
ppm

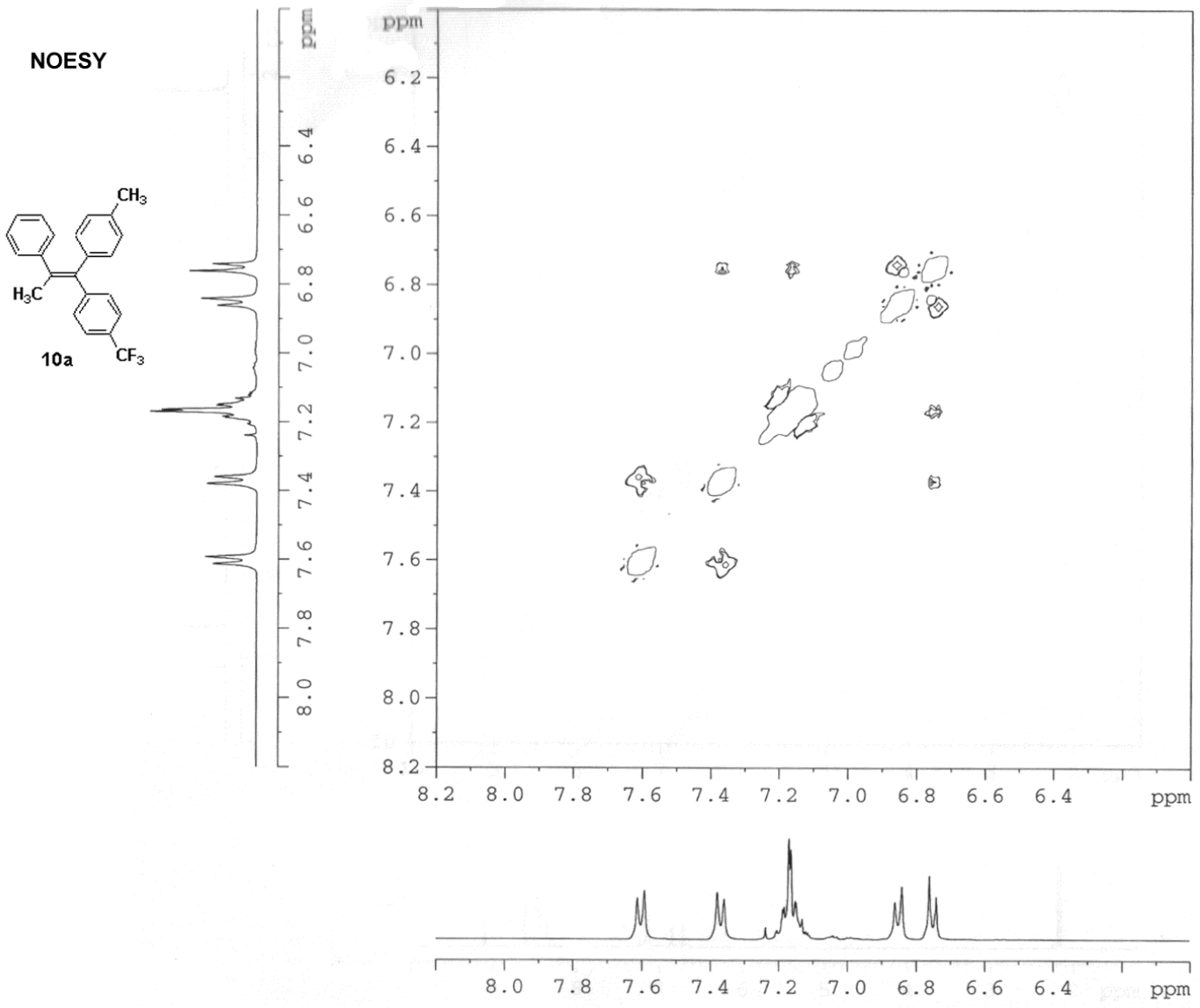
8
7
6
5
4
3
2
ppm

COESY

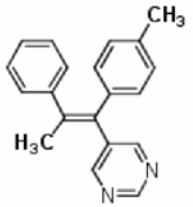


10a

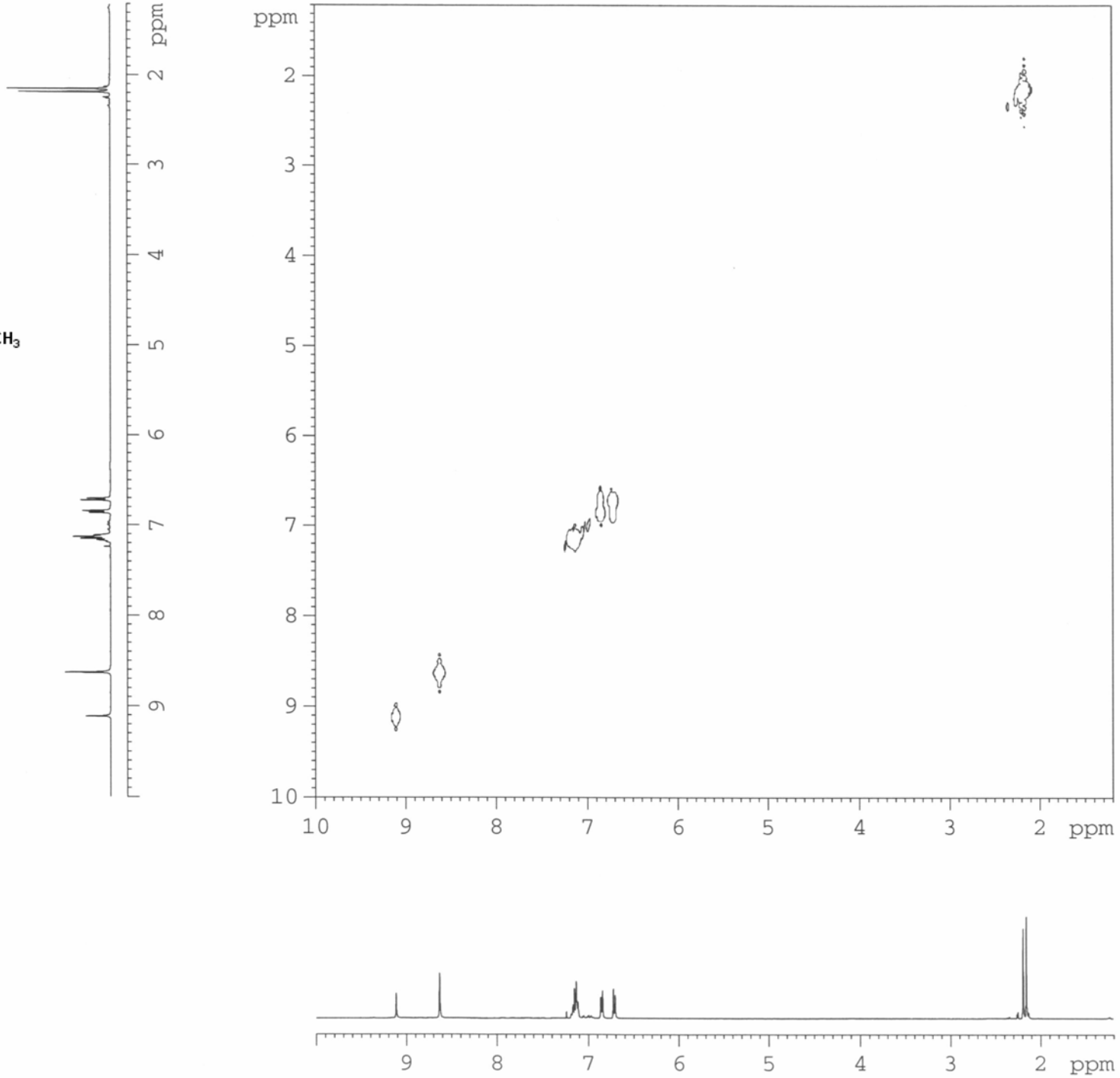



NOESY
CH3
H3C
CF3
10a
ppm
6.2
6.4
6.6
6.8
7.0
7.2
7.4
7.6
7.8
8.0
8.2
8.2 8.0 7.8 7.6 7.4 7.2 7.0 6.8 6.6 6.4 ppm
8.0 7.8 7.6 7.4 7.2 7.0 6.8 6.6 6.4 ppm

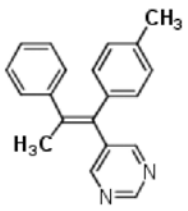
COESY



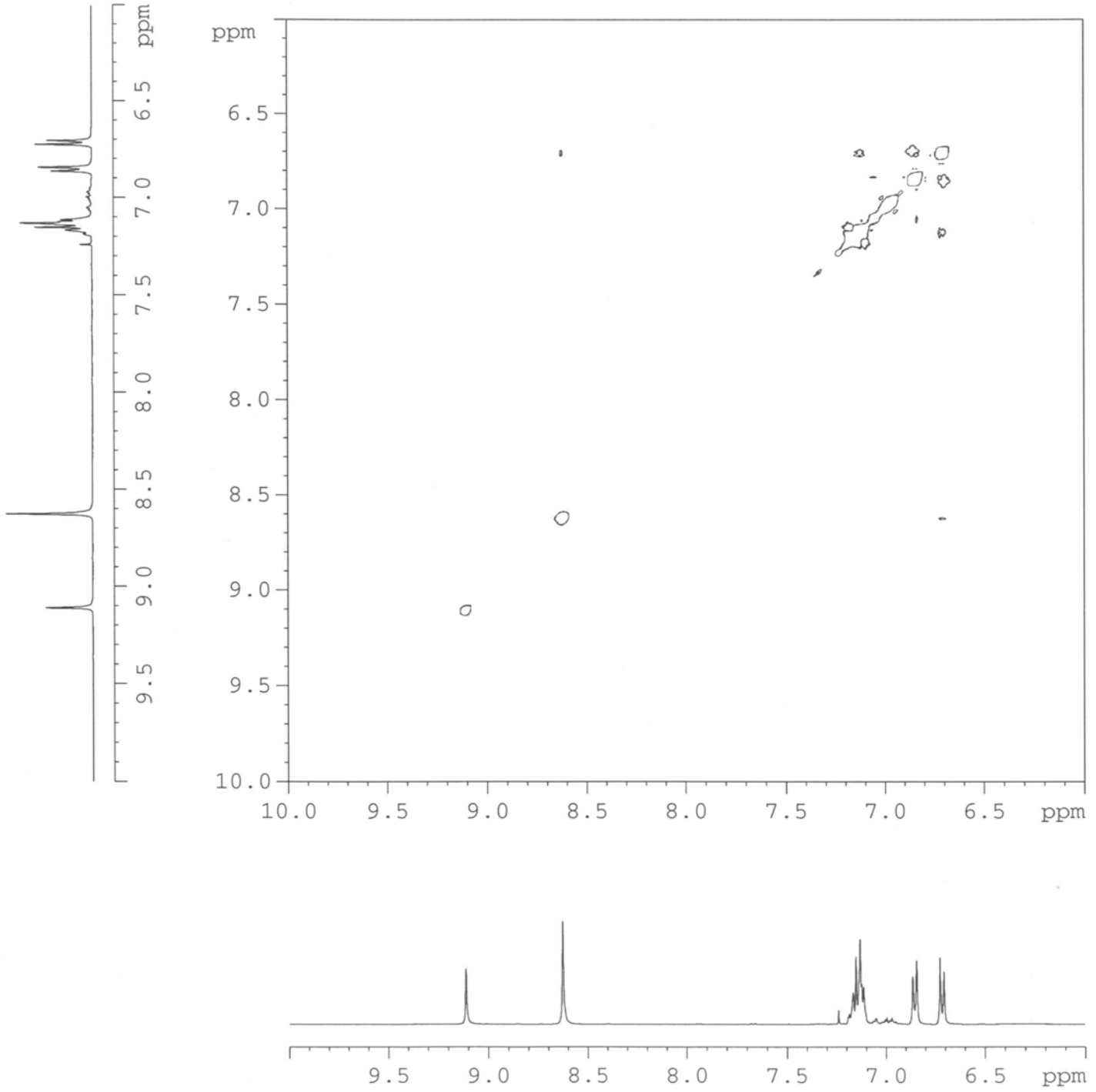
11a



NOESY


CH3
H3C
N
N

11a


ppm
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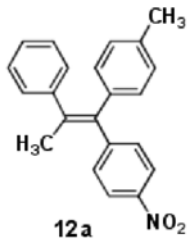
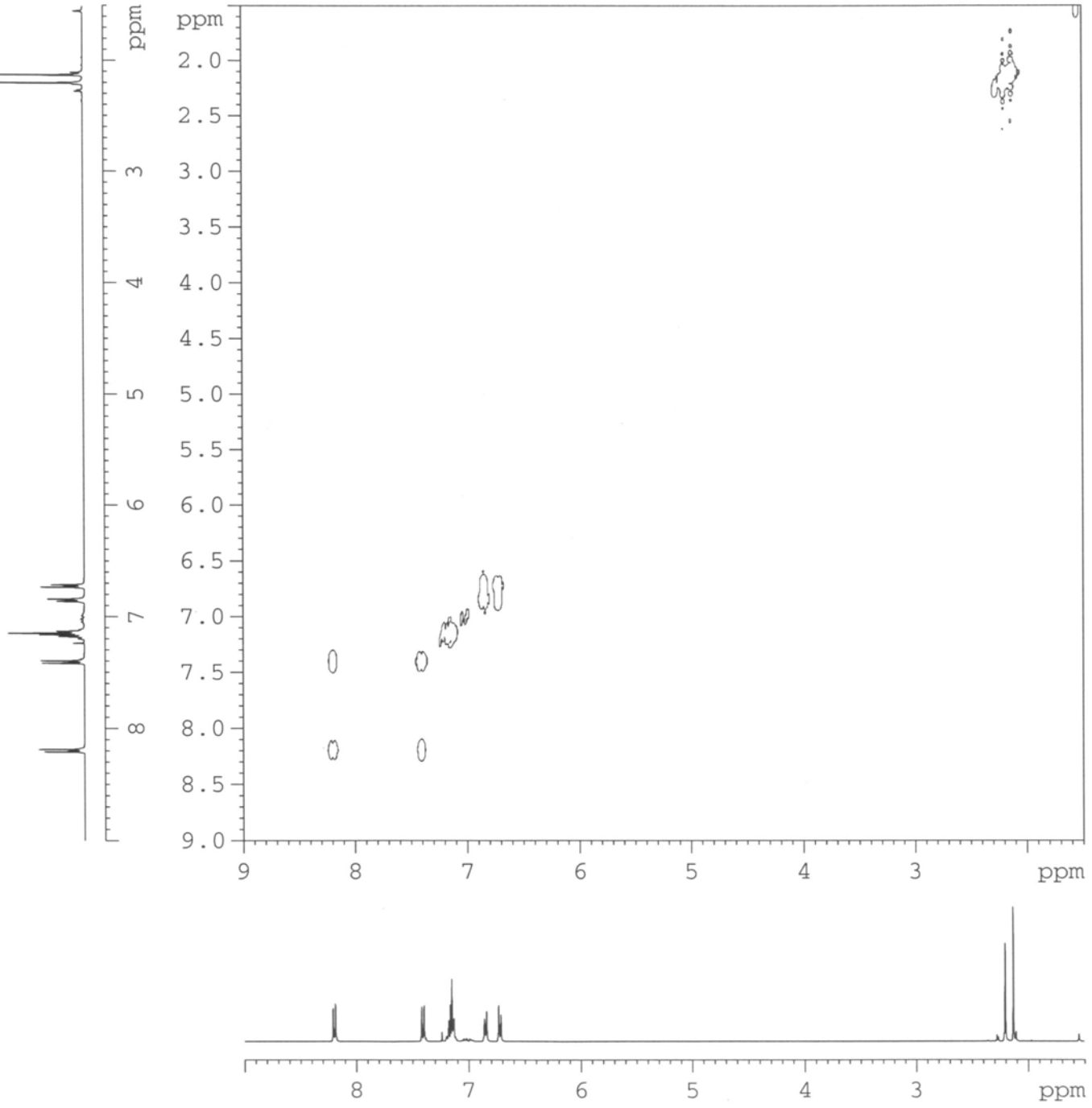
7.0
7.5
8.0
8.5
9.0
9.5
10.0
ppm
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
ppm

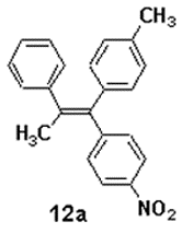
COESY


CH3
H3C
NO2

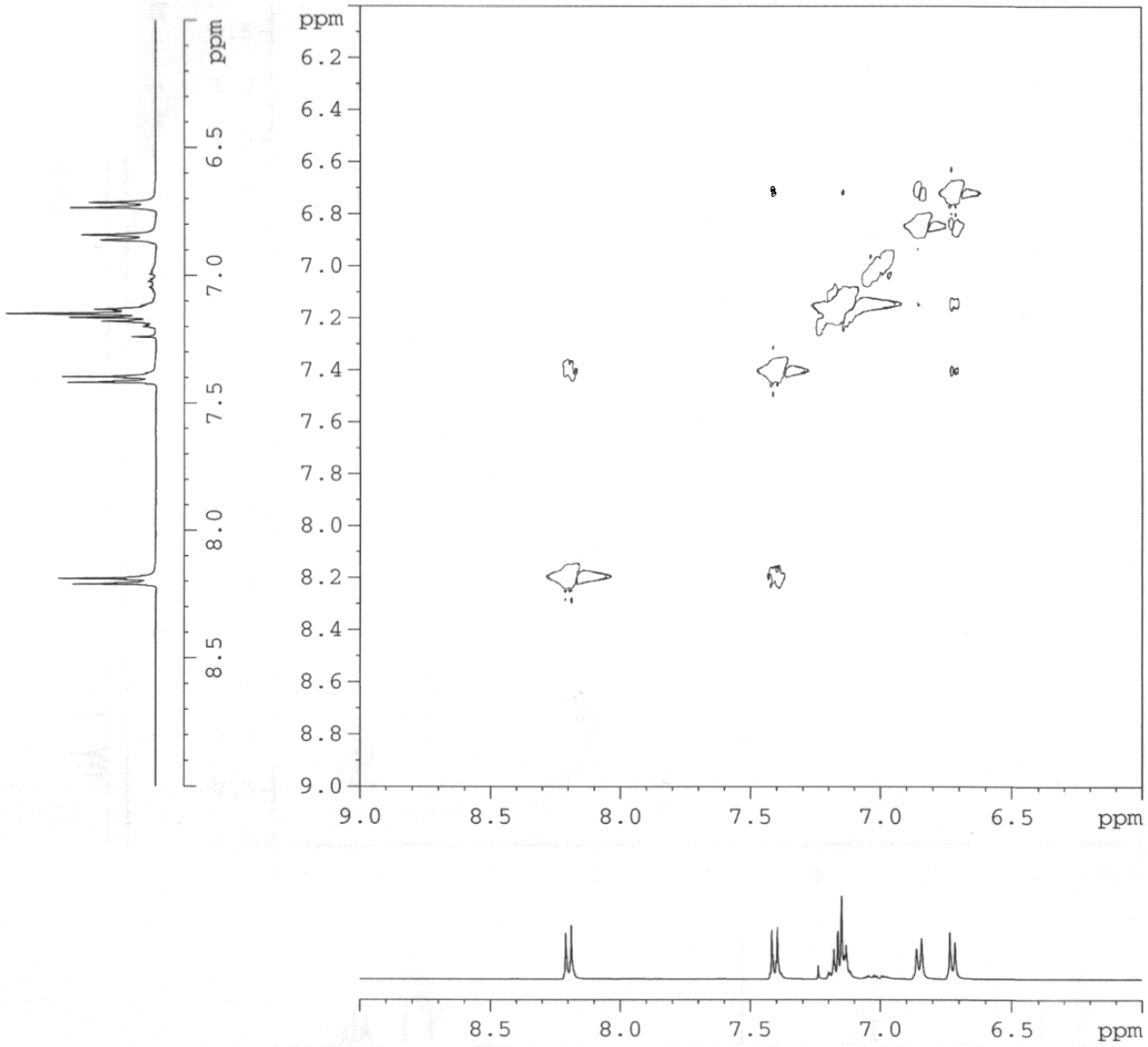
12a


ppm
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
9.0
9
8
7
6
5
4
3
ppm

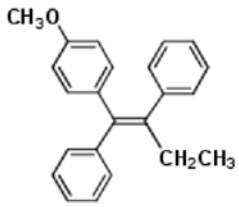
NOESY


CH3
H3C
NO2

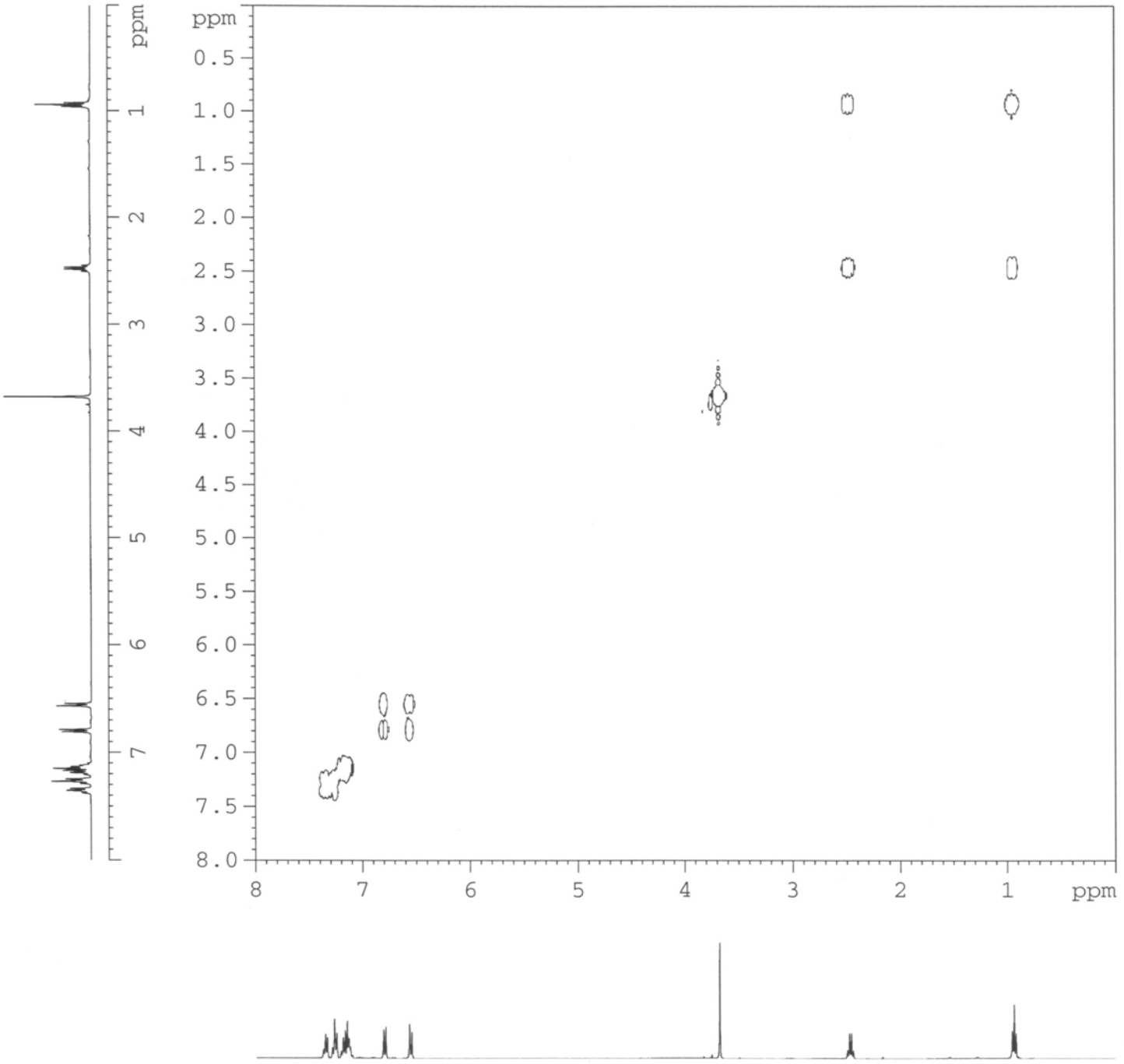
12a


ppm
6.5
7.0
7.5
8.0
8.5
ppm
6.2
6.4
6.6
6.8
7.0
7.2
7.4
7.6
7.8
8.0
8.2
8.4
8.6
8.8
9.0
9.0
8.5
8.0
7.5
7.0
6.5
ppm
8.5
8.0
7.5
7.0
6.5
ppm

COESY


CH3O
CH2CH3

13a


ppm
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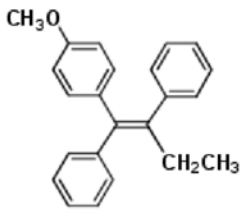
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8
7
6
5
4
3
2
1
ppm

NOESY



13a