A New Complex of Palladium-Thiourea and Carbon Tetrabromide Catalyzed Carbonylative Annulation of α-Hydroxyl-Arylacetylenes

Efficient New Synthetic Technology for the Synthesis of 2,3-Disubstituted Benzo[*b*]furans

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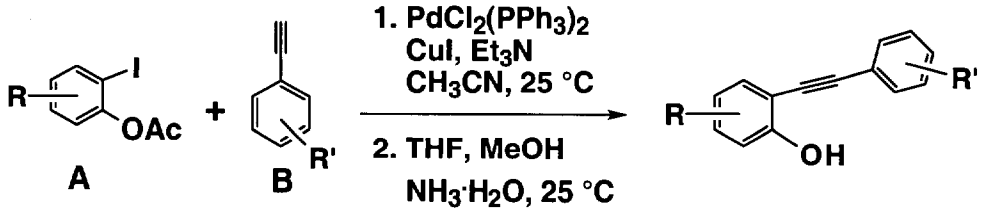
Supporting Information

General Methods

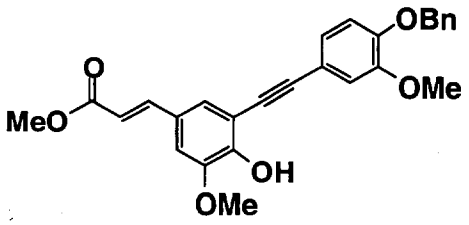
Unless stated otherwise, reactions were performed in flame-dried glassware under nitrogen or an argon atmosphere. Reaction solvents were commercially purchased from *Aldrich* without further purification and reagents were used as received. Reaction were monitored by thin-layer chromatography (TLC) on 0.25 mm precoated Merck Silica Gel 60 F_{254}, visualizing with ultraviolet light, *p*-anisaldehyde stain, or phosphomolybdic acid stain. Flash column chromatography was performed on Merck Silica Gel 60 (230-400mesh) using reagent grade hexanes, dichloromethane, and ACS grade ethyl acetate, methanol and diethyl ether. High-resolution mass spectra were performed at Harvard University Mass Spectrometry. ^{1}H and ^{13}C NMR spectra were recorded on a Varian Unity INOVA 500MHz spectrometer and are referenced to residual solvent peaks ($CDCl_3$: 1H: δ 7.24, ^{13}C: δ 77.0) or to an internal reference

of tetramethylsilan in $CDCl_3$ (1H: δ 0.00). ^1H-^1H couplings are assumed to be first order, and peak multiplicity is reported as s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), or b (broad).

General Procedure for Synthesis of α-Hydroxyl-Arylacetylenes 1a to 8a

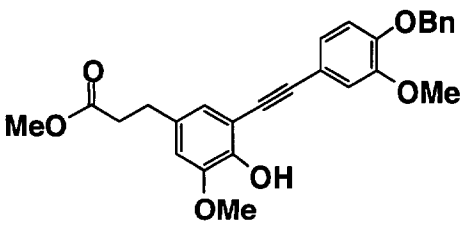


Compounds 1a-8a are synthesized followed a general procedure as illustrated below.



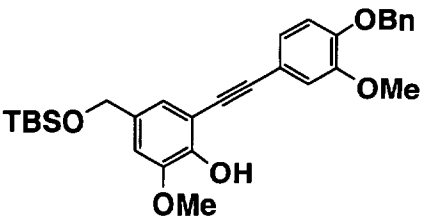
compound 1a

Compound 1a. Purification by flash chromatography (EtOAc/hexanes= 1/2) gave **1a** in 89% yield as a white solid; R_f = 0.4 (EtOAc/hexanes = 1/2); ^1H NMR (500 MHz, $CDCl_3$) δ 7.58 (d, *J* = 16.0 Hz, 1H), 7.42-7.44 (m, 2H), 7.35-7.38 (m, 2H), 7.29-7.32 (m, 1H), 7.24 (d, *J* =1.0 Hz, 1H), 7.08-7.11 (m, 2H), 6.99 (d, *J* = 1.0 Hz, 1H), 6.85 (d, *J* = 8.5 Hz, 1H), 6.32 (d, *J* = 16.0 Hz, 1H), 6.23 (s, 1H), 5.17 (s, 2H), 3.93 (s, 3H), 3.90 (s, 3H), 3.80 (s, 3H); ^{13}C NMR (125.7 MHz) δ 167.4, 149.2, 148.8, 148.4, 146.8, 144.1, 136.5, 128.5, 127.8, 127.1, 126.5, 125.5, 124.9, 116.0, 115.3, 114.7, 113.5, 110.3, 109.5, 95.0, 82.0, 70.8, 56.1, 55.9, 51.5; HRMS (FAB) for $[C_{27}H_{24}O_6 + Na]^+$, *m/z* calcd 467.1471, found: 467.1462.



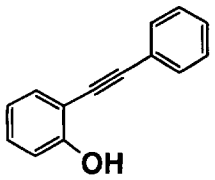
compound 2a

Compound 2a. Purification by flash chromatography (EtOAc/hexanes = 1/2) gave **2a** in 90% as a white solid; R_f = 0.3 (EtOAc/hexanes= 1/2); ^{1}HNMR (500 MHz, $CDCl_3$) δ 7.42-7.44 (m, 2H), 7.35-7.38 (m, 2H), 7.29-7.32 (m, 1H), 7.06-7.09 (m, 2H), 6.88 (d, *J* = 2.0 Hz, 1H), 6.84 (d, *J* = 9.0 Hz, 1H), 6.70 (d, *J* = 2.0 Hz, 1H); 5.84 (s, 1H), 5.17 (s, 2H), 3.90 (s, 3 H), 3.88 (s, 3H), 3.67 (s, 3H), 2.87 (t, *J* = 8.0 Hz, 2H), 2.61 (t, *J* = 8.0 Hz, 2H); ^{13}C NMR (125.7 MHz, $CDCl_3$) δ 173.1, 149.1, 148.6, 146.4, 144.9, 136.6, 132.0, 128.5, 127.8, 127.1, 124.8, 123.6, 115.6, 114.7, 113.5, 111.6, 109.6, 94.3, 82.8, 70.8, 56.0, 51.5, 35.7, 30.4; HRMS (ES) for $[C_{27}H_{27}O_6 + H]^+$, *m/z* calcd 447.1807, found: 447.1788.



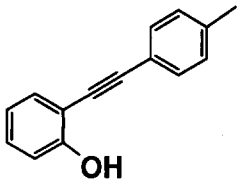
compound 3a

Compound 3a, Purification by flash chromatography (EtOAc/hexanes = 4/6) gave **3a** in 90% yield as a white solid; R_f = 0.7 (EtOAc/hexanes= 4/6); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43-7.44 (m, 2H), 7.35-7.38 (m, 2H), 7.30 (m, 1H), 7.08-7.10 (m, 2H), 6.96 (s, 1H), 6.89 (s, 1H), 6.85 (d, *J* = 9.0 Hz, 1H), 5.17 (s, 2H), 4.65 (s, 2H), 3.90 (s, 3H), 0.95 (s, 9H), 0.11 (s, 6H); ^{13}C NMR (125.7 MHz, $CDCl_3$) δ 149.1, 148.5, 146.5, 145.4, 136.6, 133.0, 128.4, 127.8, 127.1, 124.8, 121.7, 115.7, 114.7, 113.5, 109.5, 109.2, 94.1, 82.9, 70.8, 64.5, 55.9, 25.8, 18.3, -5.3; HRMS submitted.



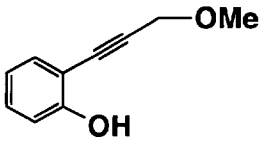
compound 4a

Known compound. See reference5c.



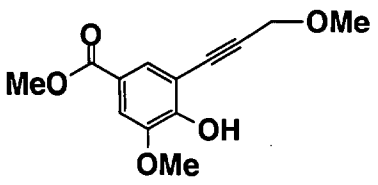
compound 5a

Compound **5**, Purification by flash chromatography (EtOAc/hexanes=1/4) gave **5a** in 87% yield as a oil; R_f = 0.4 (EtOAc/hexanes= 1/4); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 (d, J = 8.0 Hz, 2H), 7.41 (d, J = 8.0 Hz, 1H), 7.26 (m, 1H), 7.18 (d, J = 8.0 Hz, 2H), 6.98 (d, J = 8.0 Hz, 1H), 6.91 (t, J = 7.5 Hz, 1H), 5.84 (s, 1H), 2.39 (s, 3H); ^{13}C NMR (125.7 MHz, $CDCl_3$) δ 156.3, 138.9, 131.5, 131.4, 130.2, 129.2, 120.3, 119.2, 114.5, 109.7, 96.5, 82.2, 21.4; HRMS (EI) for $[C_{15}H_{12}O]^+$, *m/z* calcd 208.0888, found: 208.0881.



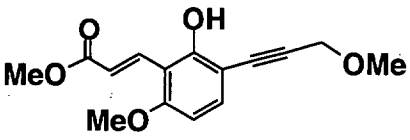
compound 6a

Compound **6a**, Purification by flash chromatography (EtOAc/hexanes=1/10) gave **6a** in 91% as a oil ; R_f = 0.5 (EtOAc/hexanes= 1/10); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.35 (dd, J_1 = 7.5 Hz, J_2 = 1.0 Hz, 1H), 7.26 (t, J = 7.5 Hz, 1H), 6.95 (d, J = 7.5 Hz, 1H), 6.87 (t, J = 7.5 Hz, 1H), 5.77 (s, 1H), 4.39 (s, 2H), 3.46(s, 3H); ^{13}C NMR (125.7 MHz, $CDCl_3$) δ 156.8, 131.9, 130.6, 120.2, 114.7, 108.7, 92.0, 80.5, 60.3, 57.7; HRMS (EI) for $[C_{10}H_{10}O_2]^+$, *m/z* calcd 162.0681, found: 162.0678.



compound 7a

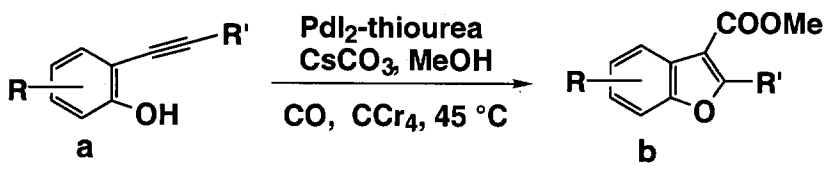
Compound **7a**, Purification by flash chromatography (EtOAc/hexanes) gave **7a** in 93% yield as a white solid; R_f = 0.3 (EtOAc/hexanes= 3/7); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.78 (d, *J* = 2.0 Hz, 1H), 7.53 (d, *J* = 2.0 Hz, 1H), 6.38 (s, 1H), 4.40 (s, 2H), 3.97 (s, 3H), 3.91 (s, 3H), 3.49 (s, 3H); ^{13}C NMR (125.7 MHz, $CDCl_3$) δ 166.4, 151.1, 146.5, 127.6, 122.1, 112.2, 109.2, 90.6, 80.8, 60.6, 57.8, 56.5, 52.3; HRMS (EI) for $[C_{13}H_{14}O_5]^+$, *m/z* calcd 250.0841, found 250.0845.



Compound 8a

Compound **8a**, Purification by flash chromatography (EtOAc/hexanes=3/7) gave **8a** in 92% yield as a white solid; R_f = 0.4 (EtOAc/hexanes = 4/6); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.08 (d, *J* = 16.5 Hz, 1H), 7.33 (d, *J* = 8.5 Hz, 1H), 6.94 (d, *J* = 16.5 Hz, 1H), 6.47 (d, *J* = 8.5 Hz, 1 H), 6.46 (s, 1H), 4.39 (s, 2H), 3.89 (s, 3H), 3.81 (s, 3H), 3.46 (s, 3H); ^{13}C NMR (125.7 MHz, $CDCl_3$) δ 168.9, 160.7, 157.6, 135.0, 133.8, 121.4, 110.7, 103.4, 102.5, 92.3, 80.2, 60.6, 58.0, 56.1, 51.7; HRMS submitted.

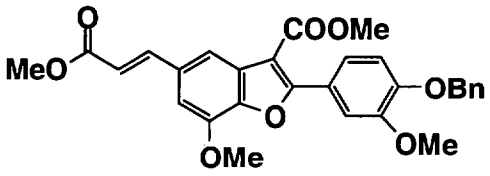
General procedure for the synthesis of compounds 1b, 2b, 3b, 4b, 5b, 6b, 7b and 8b.



a **b**

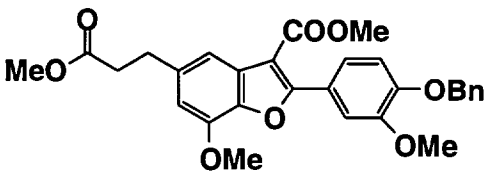
A round bottom flask (25 ml) was flame dried under high vacuum. Upon cooling, α-hydroxyl-phenylacetylen **a** (1 mmol), PdI_2 (18mg, 0.05 mmol), thiourea (3.8 mg, 0.05 mmol), CBr_4

(1.66g, 5 mmol) and methanol (8 mL) were added to the flask, and the mixture was degassed under vacuum with CO for 4 times. The reaction mixture was stirred at 45 °C for 25 min. Following completion of the reaction as monitored by TLC, the reaction mixture was cooled, dilute with Et_2O (30 mL), and filtrated through a short silica gel bed. The filtrate was concentrated to a residue that was purified as stated below.



Compound 1b

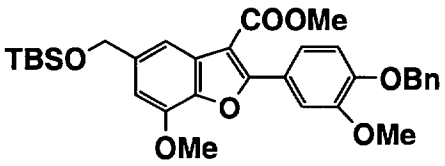
Compound 1b, Purification by flash chromatography (EtOAc/hexanes=1/2) gave **1b** in 84% yield as a white solid; R_f = 0.7 (EtOAc/hexanes= 1/2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.81 (d, J = 16 Hz, 1H), 7.78 (d, J = 1 Hz, 1H), 7.76 (d, J = 2 Hz, 1H), 7.64 (dd, J_1 = 9, 2 Hz, J_2 = 2.0 Hz, 1H), 7.45-7.7.46 (m, 2H), 7.37-7.40 (m, 2 H), 7.30-7.33 (m, 1H), 7.00 (m, 1H), 6.97 (d, J = 9 Hz, 1H), 6.46 (d, J = 16 Hz, 1H), 5.24 (s, 2H), 4.04 (s, 3H), 3.98 (s, 3H), 3.96 (s, 3H), 3.83 (s, 3H); ^{13}C NMR (125.7 MHz, $CDCl_3$) δ 167.3, 164.1, 161.5, 150.1, 148.8, 145.0, 143.9, 136.5, 131.2, 129.1, 128.5, 127.8, 127.1, 122.8, 121.8, 116.9, 116.0, 112.9, 112.7, 107.9, 105.7, 70.7, 56.0, 55.9, 51.6, 51.5; HRMS (ES) for $[C_{29}H_{26}O_8+Na]^+$, *m/z* calcd 502.1628, found: 502.1619.



Compound 2b

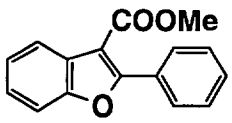
Compound 2b, Purification by flash chromatography (EtOAc/hexanes=3/7) gave **2b** in 81% yield as a white solid; R_f = 0.6 (EtOAc/hexanes= 3/7); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.70 (d, J = 2 Hz, 1H), 7.60 (dd, J_1 = 9.0 Hz J_2 = 2.0 Hz, 1H), 7.43-7.46 (m, 3H), 7.36-7.39 (m, 2H), 7.31

(m, 1H), 6.96 (d, J = 9.0 Hz, 1H), 6.70 (d, J = 1.5 Hz, 1H), 5.24 (s, 3H), 4.01(s, 3H), 3.98 (s, 3H), 3.93 (s, 3H), 3.70 (s, 3H), 3.06 (t, J = 8.0 Hz, 2H), 2.71 (t, J = 8.0 Hz, 2H); ^{13}C NMR (125.7 MHz, $CDCl_3$) δ 173.2, 164.5, 161.0, 149.9, 148.8, 144.6, 141.6, 137.3, 136.6, 128.9, 128.5, 127.8, 127.1, 122.8, 122.3, 113.6, 112.9, 112.8, 107.9, 107.7, 70.7, 56.1, 56.0, 51.5, 36.3, 31.5; HRMS (ES) for $[C_{29}H_{29}O_8 + H]^+$, m/z calcd 505.1862, found: 505.1884.



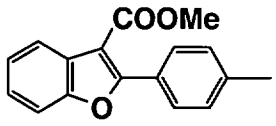
Compound 3b

Compound 3b, Purification by flash chromatography (EtOAc/hexanes =1/4) gave **3b** 85% yield as a soft solid; R_f = 0.7 (EtOAc/hexanes= 1/4); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.74 (d, J = 2.0 Hz, 1H), 7.63 (dd, J_1 = 9.0 Hz, J_2 = 2.0 Hz, 1H), 7.55 (s, 1H), 7.45-7.46 (m, 2H), 7.36-7.39 (m, 3H), 7.31 (s, 1H), 6.96 (d, J = 9.0 Hz, 1H), 6.88 (s, 1H), 5.23 (s, 1H), 4.85 (s, 2H), 4.01 (s, 3H), 3.98 (s, 3H), 3.93 (s, 3H), 0.98 (s, 9H), 0.14 (s, 6H); ^{13}C NMR (125.7 MHz, $CDCl_3$) δ 164.6, 161.0, 149.8, 148.8, 144.7, 141.9, 138.3, 136.6, 128.5, 127.8, 127.1, 122.8, 122.3, 112.9, 112.8, 111.6, 108.0, 105.4, 70.7, 65.1., 56.1, 55.9, 51.4, 25.8, 18.3, -5.26; HRMS (FAB) for $[C_{32}H_{38}O_7Si + Na]^+$, m/z calcd 585.2285, found: 585.2285.



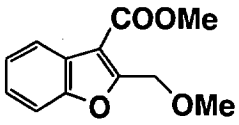
Compound 4b

Known compound, see reference 5c.



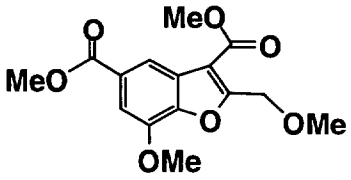
Compound 5b

Compound 5b, Purification by flash chromatography (EtOAc/hexanes=1:19) gave **5b** in 78% yield as a white solid; R_f = 0.8 (EtOAc/hexanes = 1/19); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.05 (m, 1H), 7.93 (d, *J* = 8.5 Hz, 2H), 7.53 (m, 1H), 7.35 (m, 2H), 7.30 (d, *J* = 8.5 Hz, 1H), 3.94 (s, 3H), 2.45 (s, 3H); ^{13}C NMR (125.7 MHz, $CDCl_3$) δ 164.5, 161.1, 153.5, 140.5, 129.3, 128.7, 127.0, 126.6, 124.9, 123.8, 122.5, 111.0, 108.1, 51.4, 21.5; HRMS (EI) for $[C_{17}H_{14}O_3]^+$, *m/z* calcd 226.0943, found: 226.0931.



Compound 6b

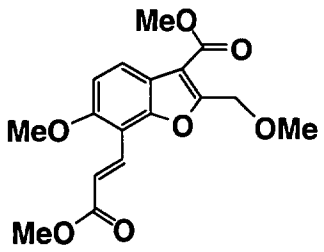
Compound 6b, Purification by flash chromatography (EtOAc/hexanes=1/4) gave **6b** in 84% yield) as a oil; R_f = 0.75 (EtOAc/hexanes= 1/4); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.01 (m, 1H), 7.52 (m, 1H), 7.31-7.36 (m, 2H), 4.97 (s, 2H), 3.97 (s, 3H), 3.49 (s, 3H); ^{13}C NMR (125.7 MHz, $CDCl_3$) δ 164.1, 160.6, 154.2, 125.3, 125.2, 124.0, 122.3, 111.4, 111.1 65.5, 58.8, 51.6; HRMS (EI) for $[C_{12}H_{12}O_4]^+$, *m/z* calcd 220.0736, found: 220.0730.



Compound 7b

Compound 7b, Purification by flash chromatography (EtOAc/hexanes=3/7) gave **7b** in 80% yield as a white solid; R_f = 0.65 (EtOAc/hexanes= 3/7); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31 (d, *J* = 1.5 Hz, 1H), 7.56 (d, *J* = 1.5 Hz, 1H), 4.95 (s, 2 H), 4.05 (s, 3H), 4.0 (s, 3H), 3.96 (s, 3H),

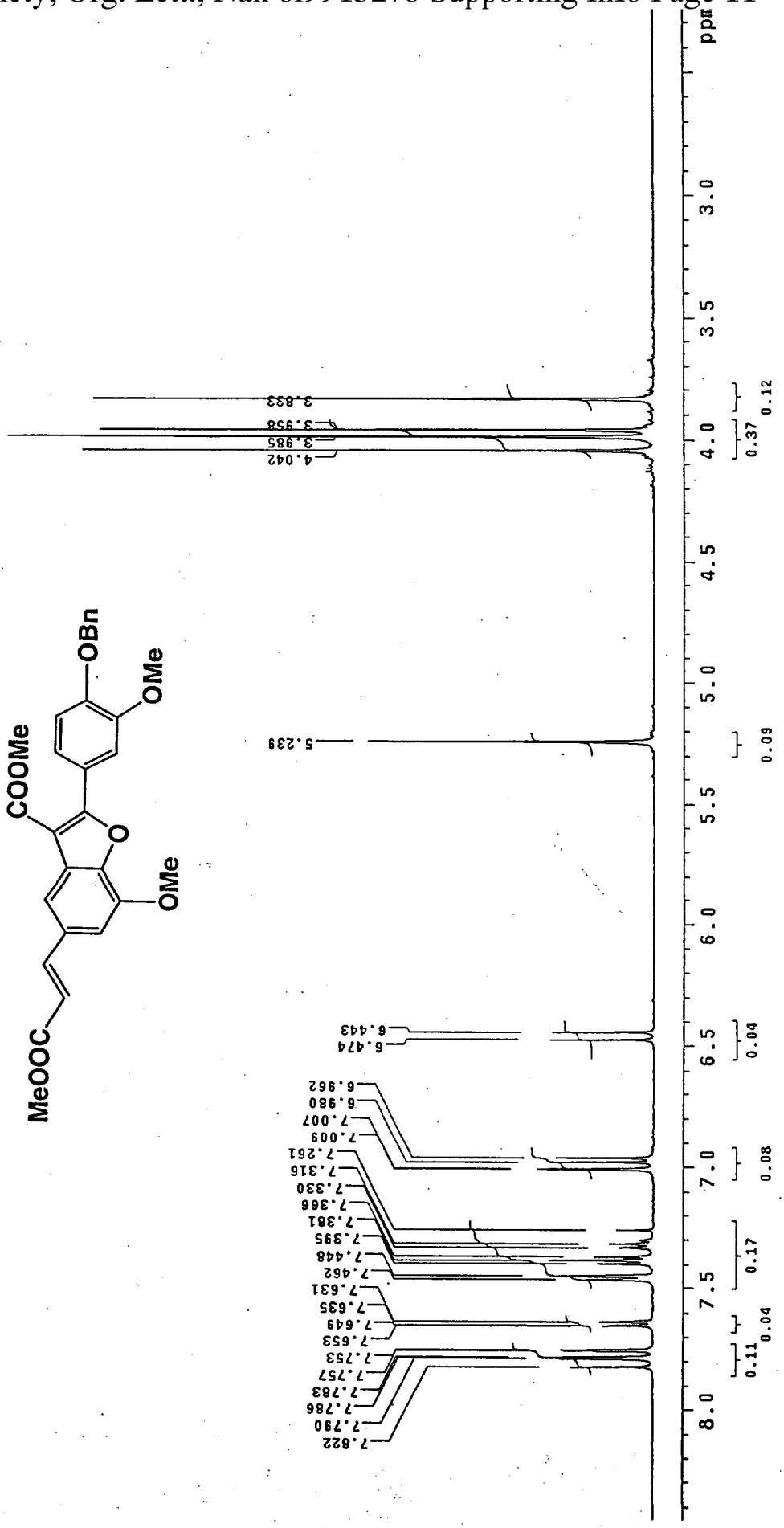
3.48 (s, 3); ^{13}C NMR (125.7 MHz, $CDCl_3$) δ 166.9, 163.5, 161.8, 146.1, 144.9, 127.2, 126.6, 117.0, 112.1, 108.2, 65.1, 58.9, 56.1, 52.2, 51.9; HRMS (EI) for $[C_{15}H_{16}O_7]^+$, *m/z* calcd 308.0896 found 308.0891.



Compound 8b

Compound 8b, Purification by flash chromatography (EtOAc/hexanes=3/7) gave **8b** in 79% yield as a white solid solid; R_f = 0.45 (EtOAc/hexanes = 4/6); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.17 (d, *J* = 16.5 Hz, 1H), 7.94 (d, *J* = 8.5 Hz, 1H), 7.06 (d, *J* = 16.5 Hz, 1H), 7.06 (d, *J* = 16.5 Hz, 1H), 6.97 (d, *J* = 8.5 Hz, 1H), 4.92 (s, 2H), 3.96 (s, 3H), 3.95 (s, 3H), 3.83 (s, 3H), 3.48 (s, 3H); ^{13}C NMR (125.7 MHz, $CDCl_3$) δ 168.3, 164.0, 160.2, 157.5, 153.6, 134.0, 124.5, 121.5, 119.6, 111.6, 108.9, 108.8, 65.4, 59.0, 56.7, 51.9, 51.8; HRMS submitted.

Table of Spectra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OMe
COOMe
O
OMe
MeOOC
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5.239
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6.980
7.007
7.009
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7.330
7.366
7.381
7.395
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ppm
3.0
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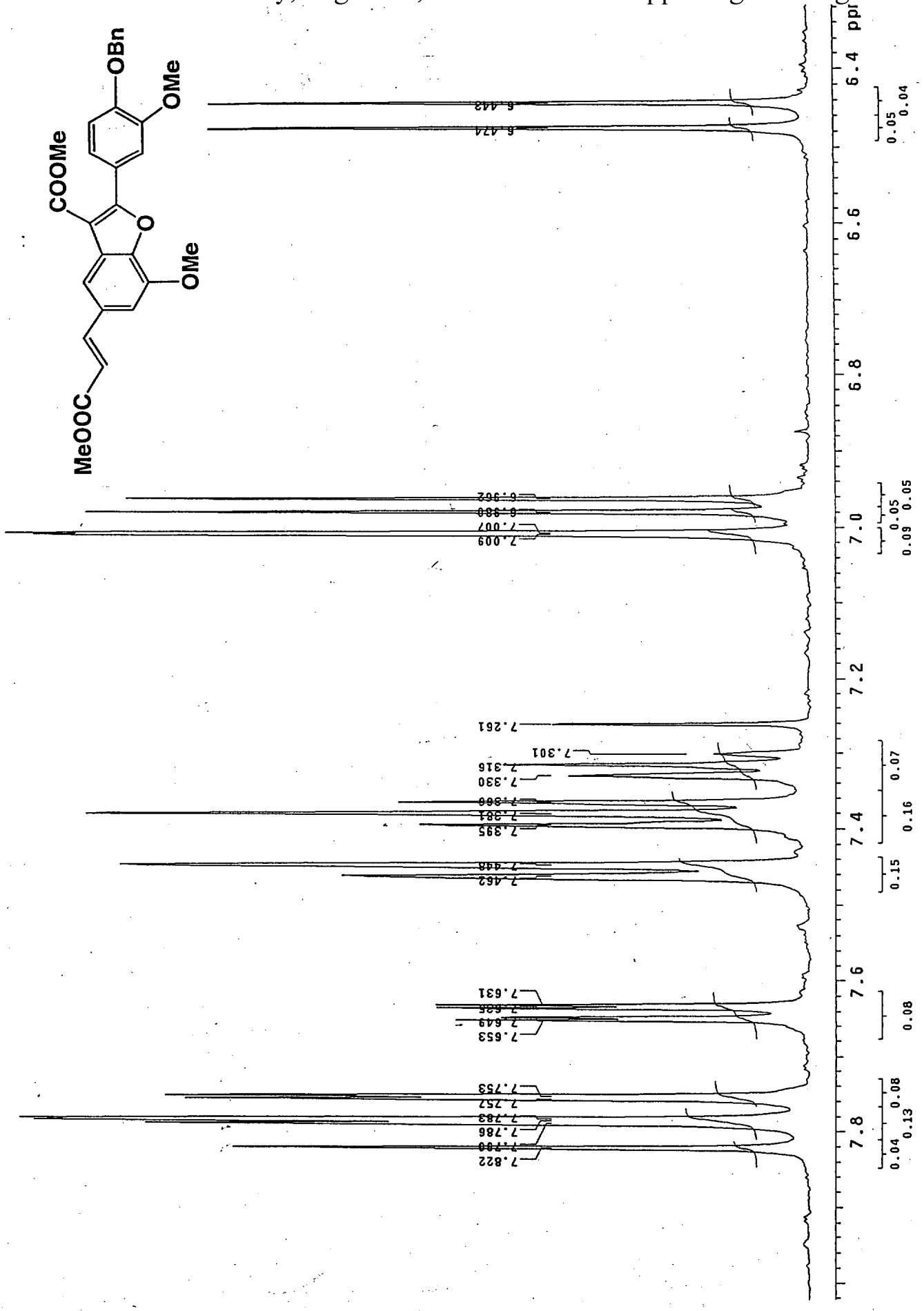
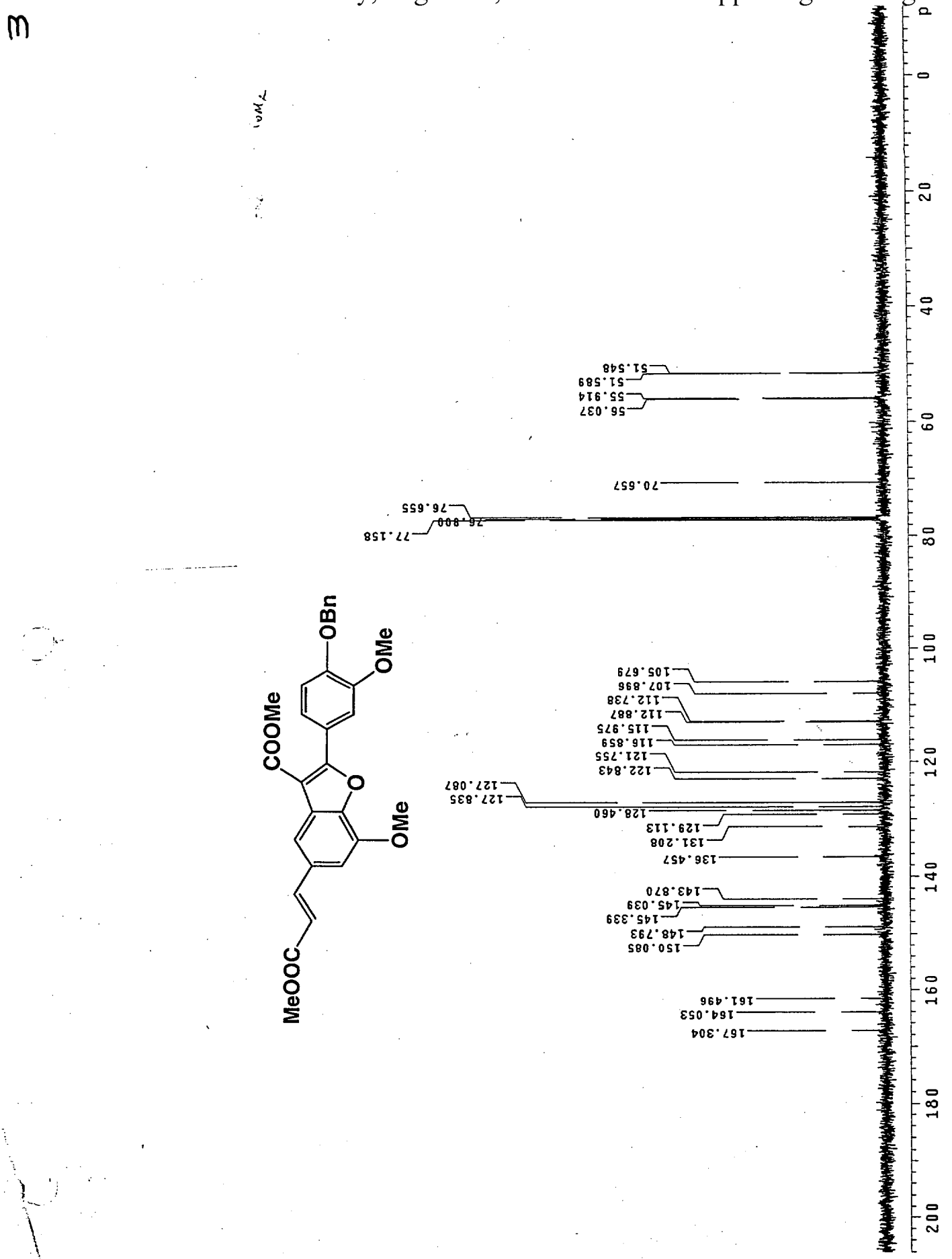
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
0.37
0.12
0.09
0.04
0.08
0.17
0.11
0.04





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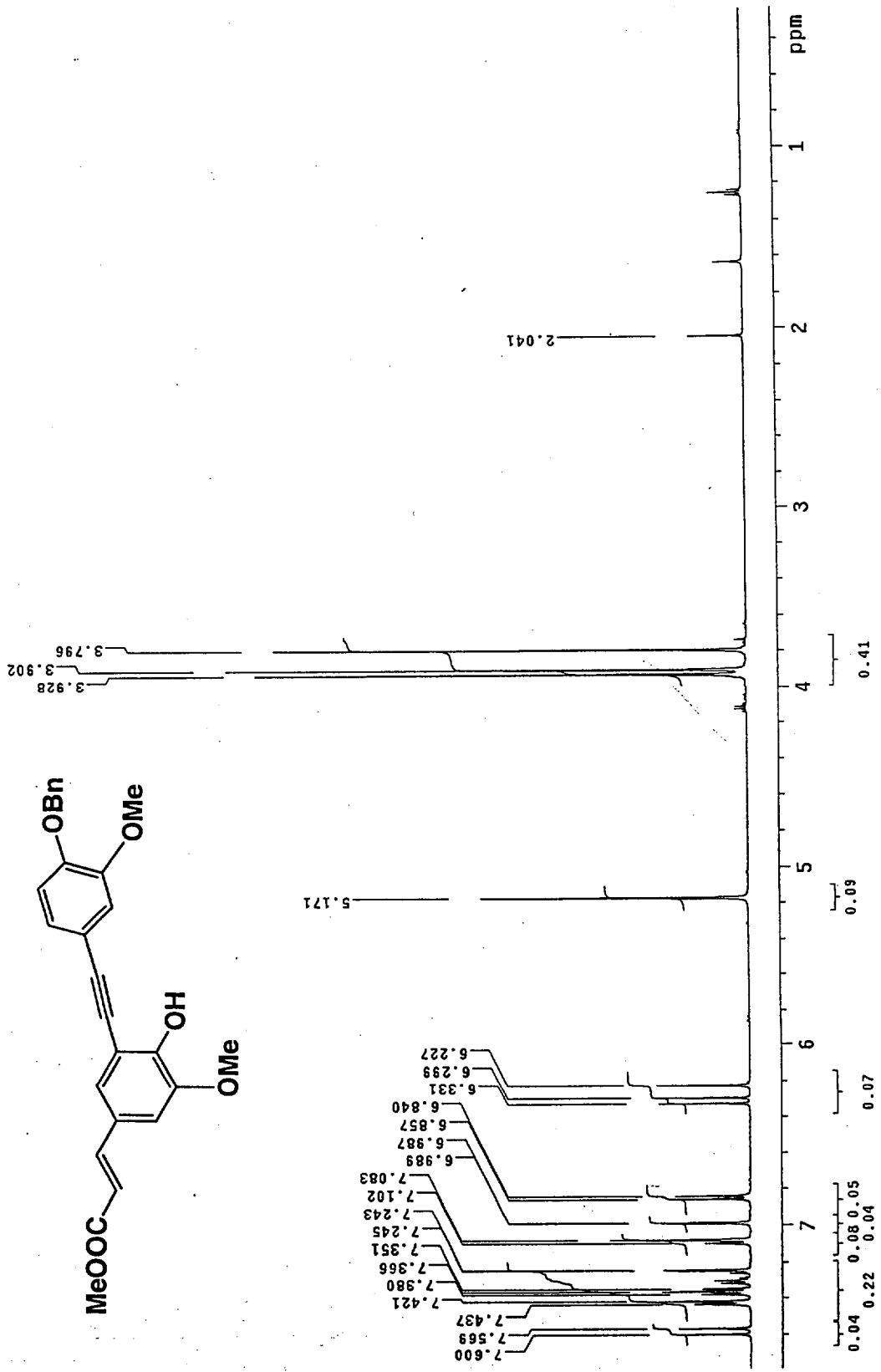

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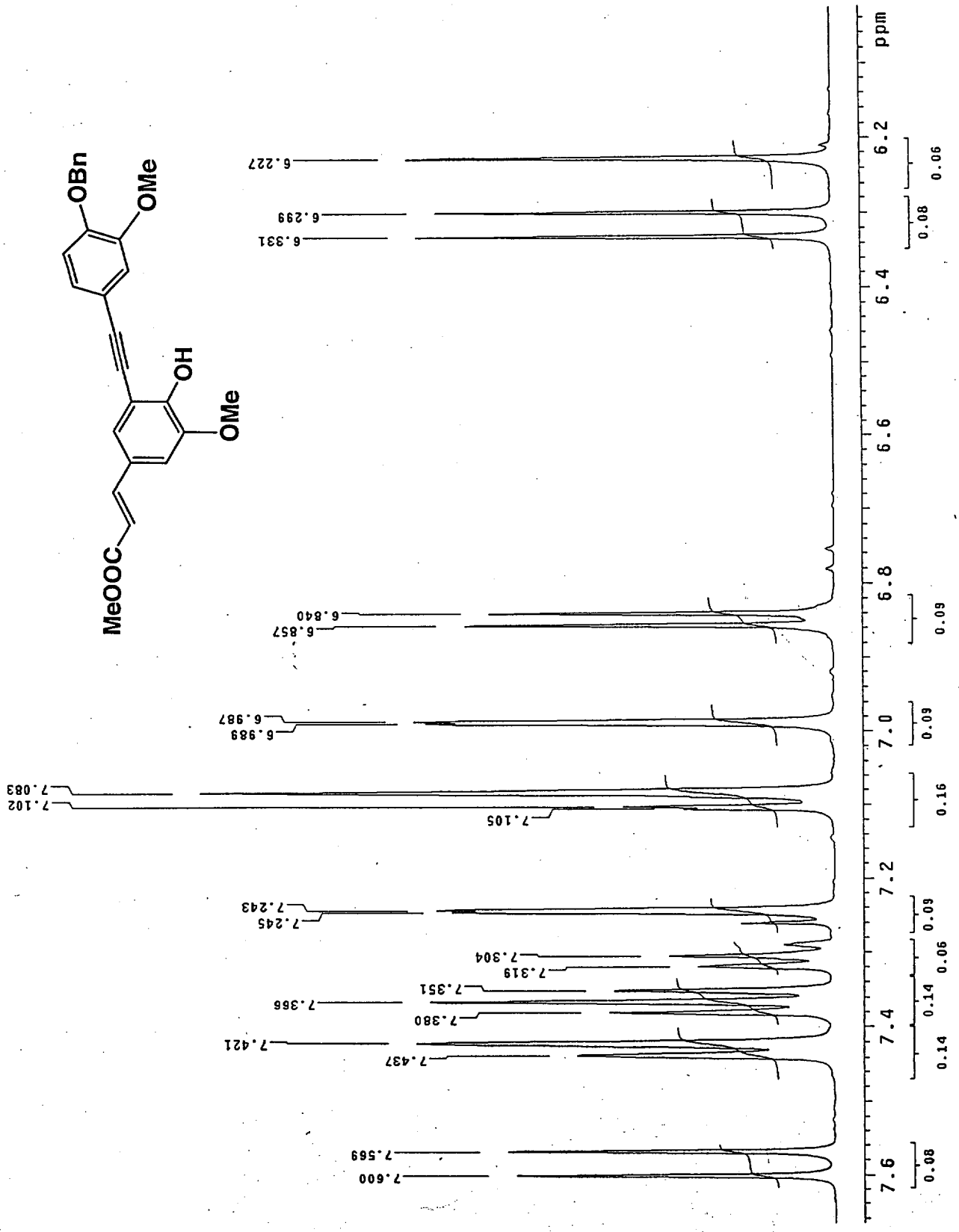
OBn
OMe
OH
OMe
MeOOC
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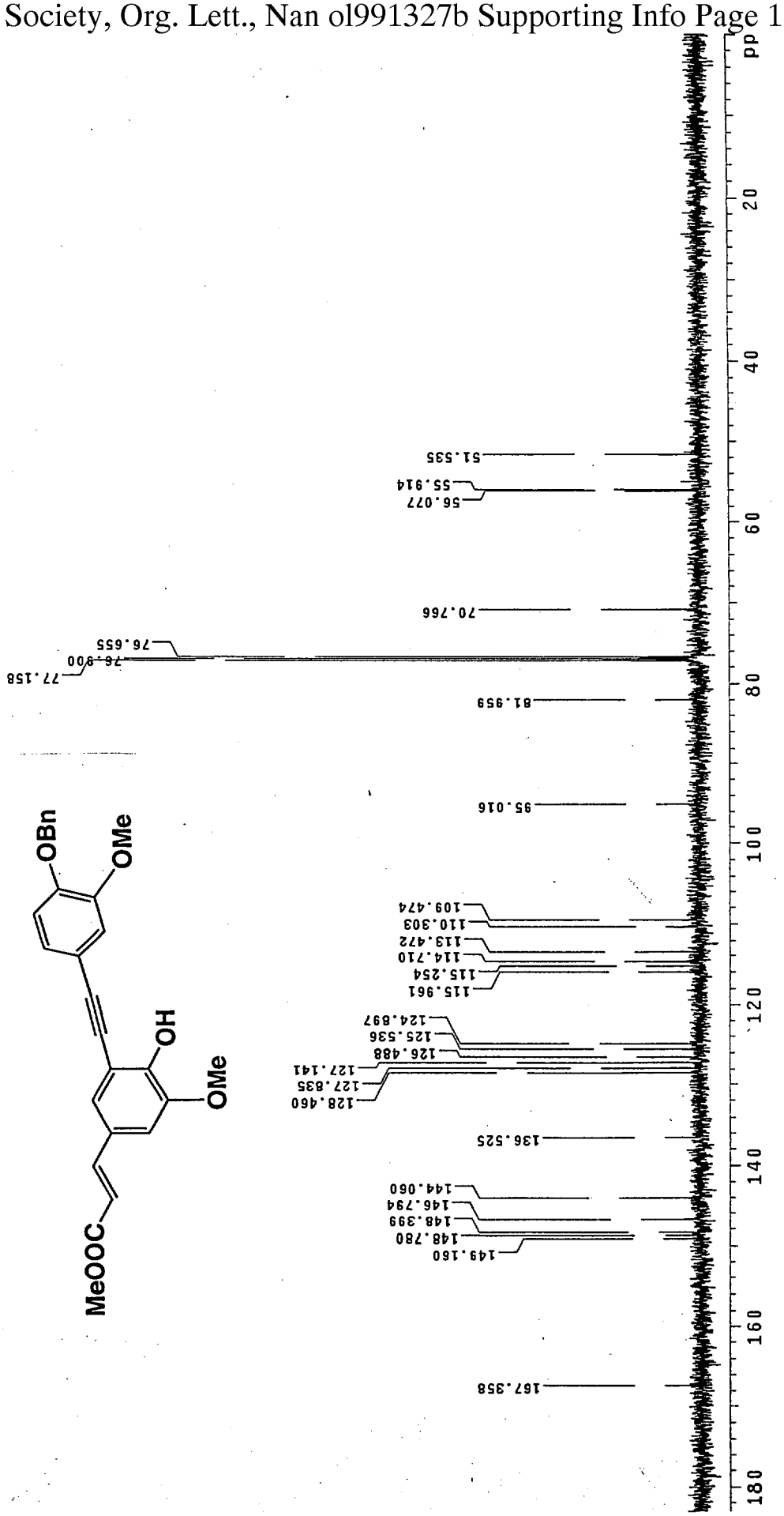
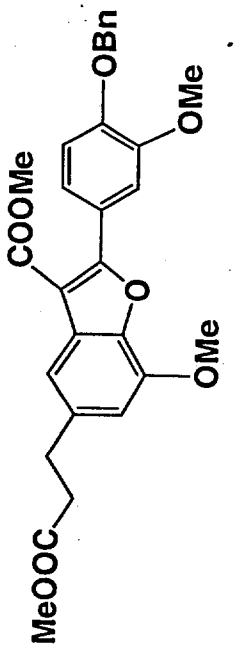
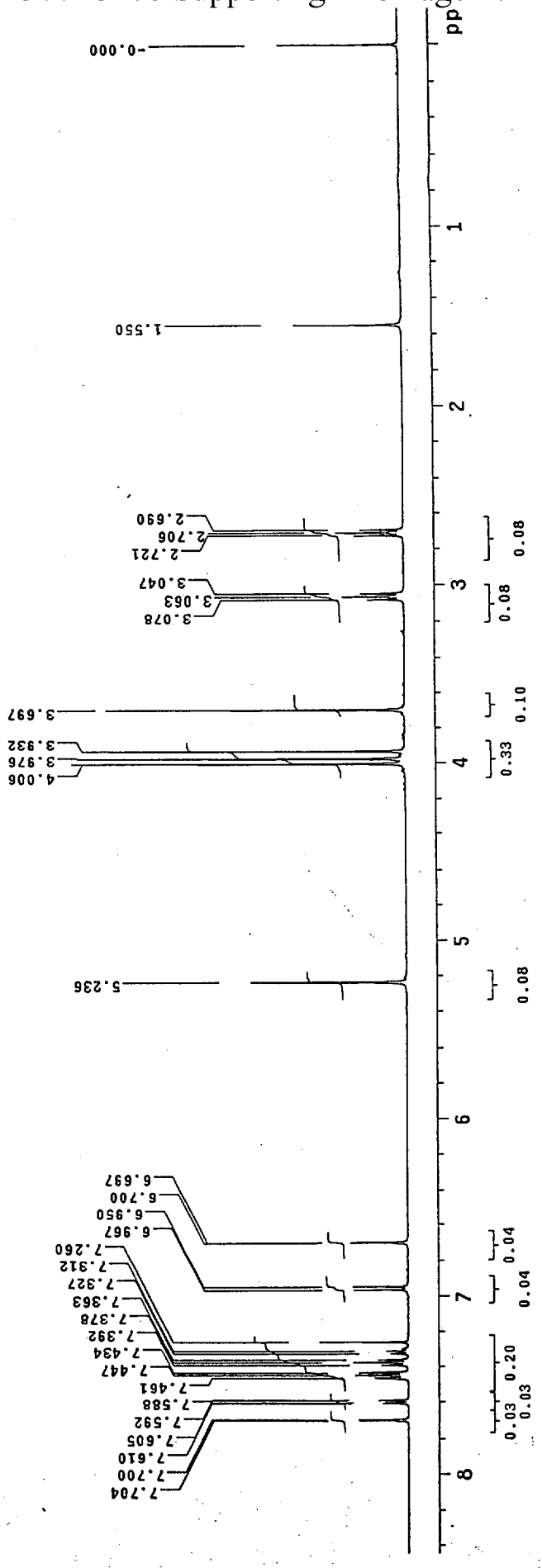
6.299
6.331
6.840
6.857
6.983
6.987
7.083
7.102
7.105
7.243
7.245
7.304
7.319
7.351
7.356
7.380
7.421
7.437
7.569
7.600
ppm
6.2
6.4
6.6
6.8
7.0
7.2
7.4
7.6
0.08
0.06
0.08
0.09
0.09
0.16
0.09
0.06
0.14
0.14
0.08




OBn
OMe
COOMe
O
OMe
MeOOC


ppm
0.000
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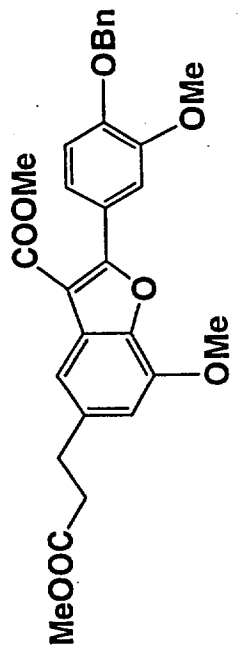
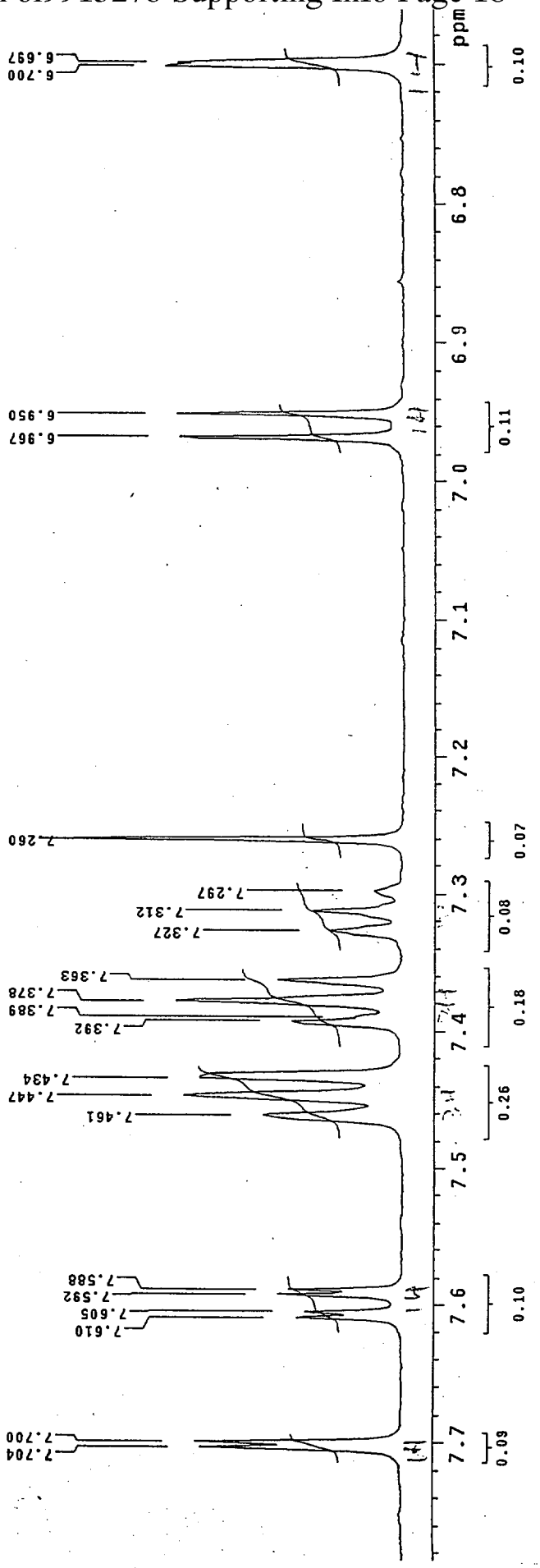
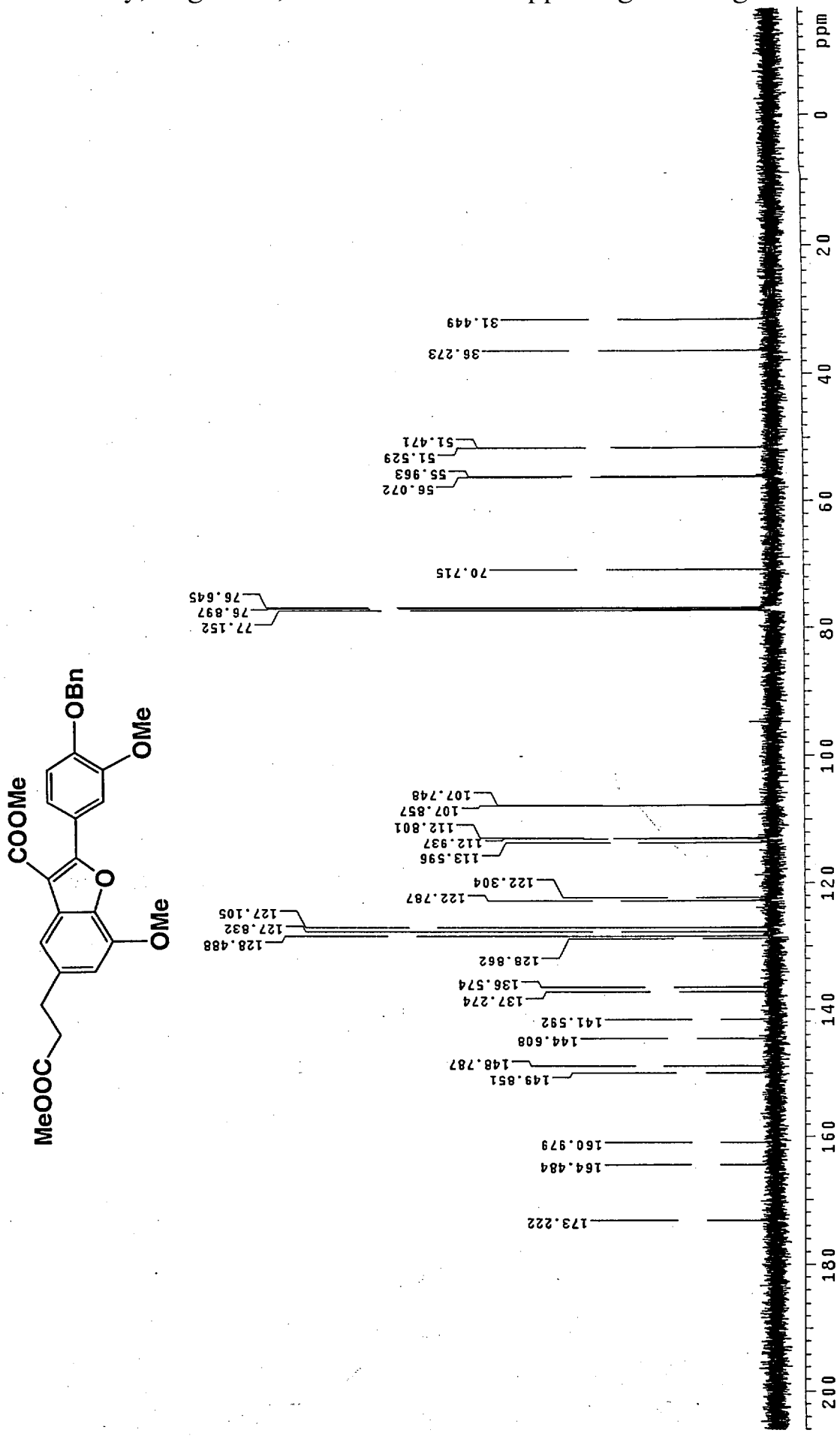
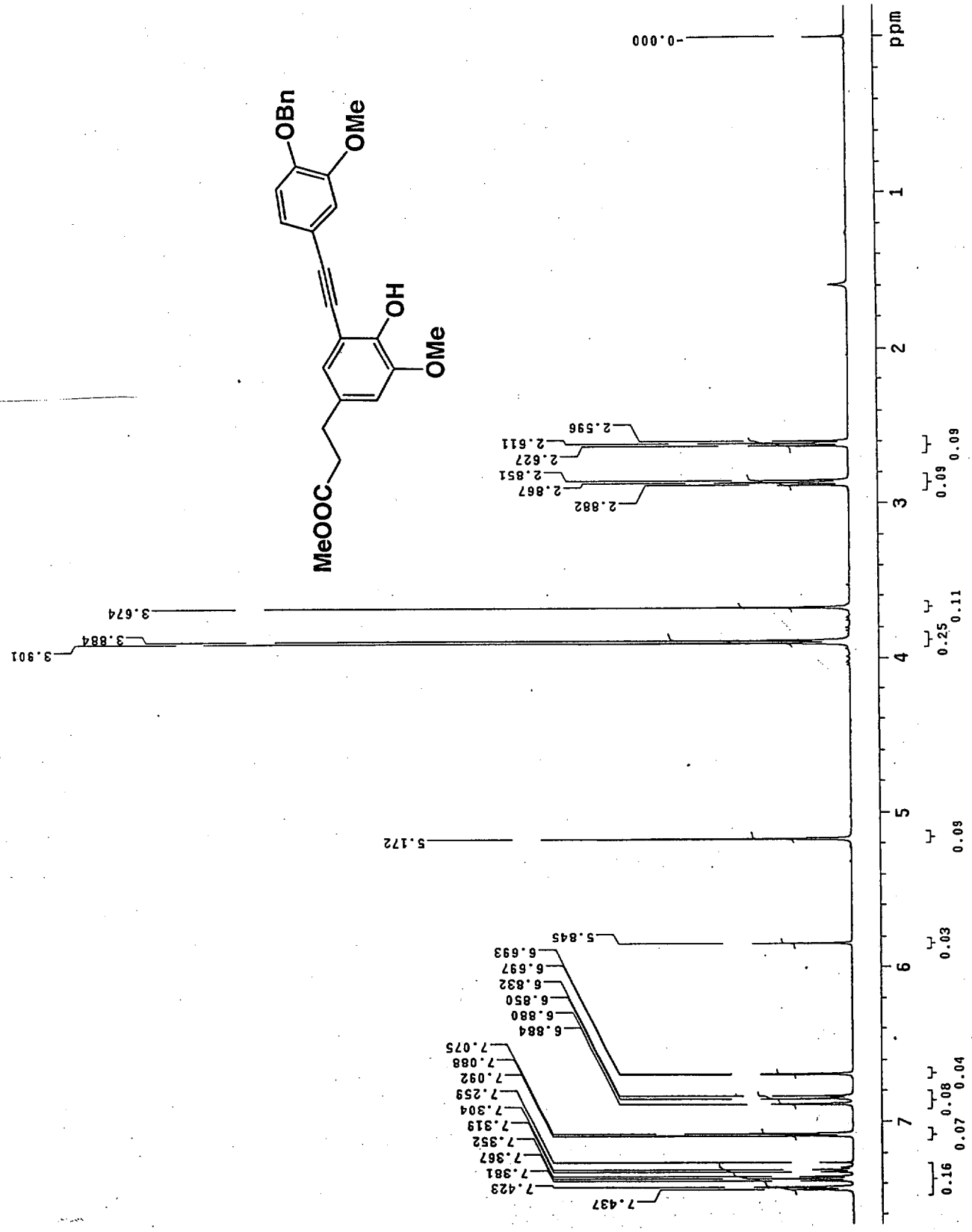
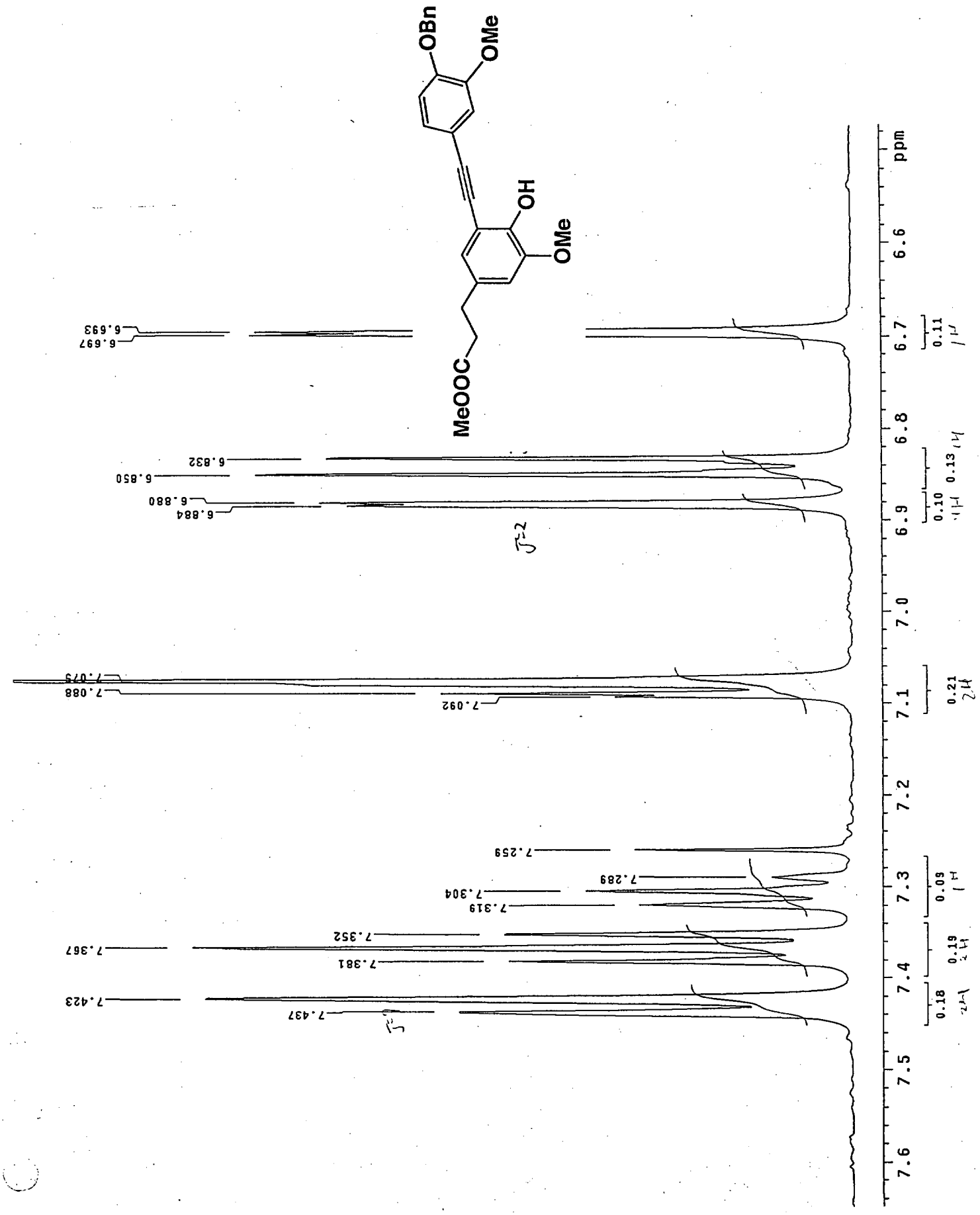
2.690
2.706
2.721
3.047
3.063
3.078
3.697
3.932
3.976
4.006
5.236
6.697
6.700
6.950
6.967
7.260
7.312
7.327
7.363
7.378
7.392
7.434
7.447
7.461
7.588
7.592
7.605
7.610
7.700
7.704
0.08
0.08
0.10
0.33
0.08
0.04
0.04
0.20
0.03
0.03






OBn
OMe
COOMe
O
OMe
MeOOC
31.449
36.273
51.471
51.529
55.963
56.072
70.715
76.645
76.897
77.152
107.748
107.857
112.801
112.937
113.596
122.304
122.787
127.105
127.832
128.488
128.862
136.574
137.274
141.592
144.608
148.787
149.851
160.979
164.484
173.222
ppm
0
20
40
60
80
100
120
140
160
180
200


OBn
OMe
OH
OMe
MeOOC
ppm
0
1
2
3
4
5
6
7
0.000
2.596
2.611
2.627
2.851
2.867
2.882
3.674
3.884
3.901
5.172
5.845
6.693
6.697
6.832
6.850
6.880
6.884
7.075
7.088
7.092
7.259
7.304
7.319
7.352
7.367
7.381
7.423
7.437
0.09
0.09
0.11
0.25
0.09
0.03
0.04
0.08
0.07
0.16


OBn
OMe
OH
OMe
MeOOC
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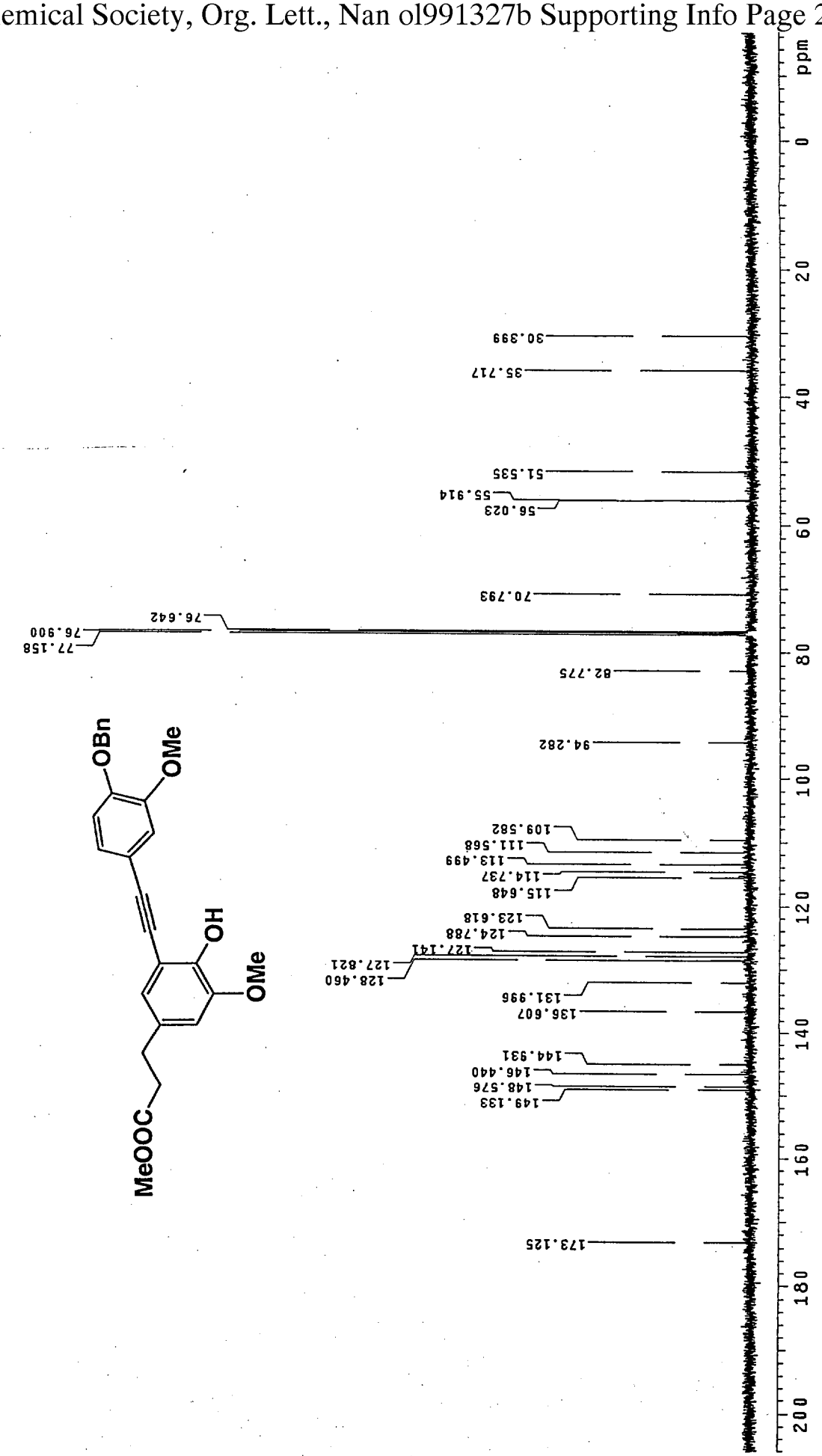
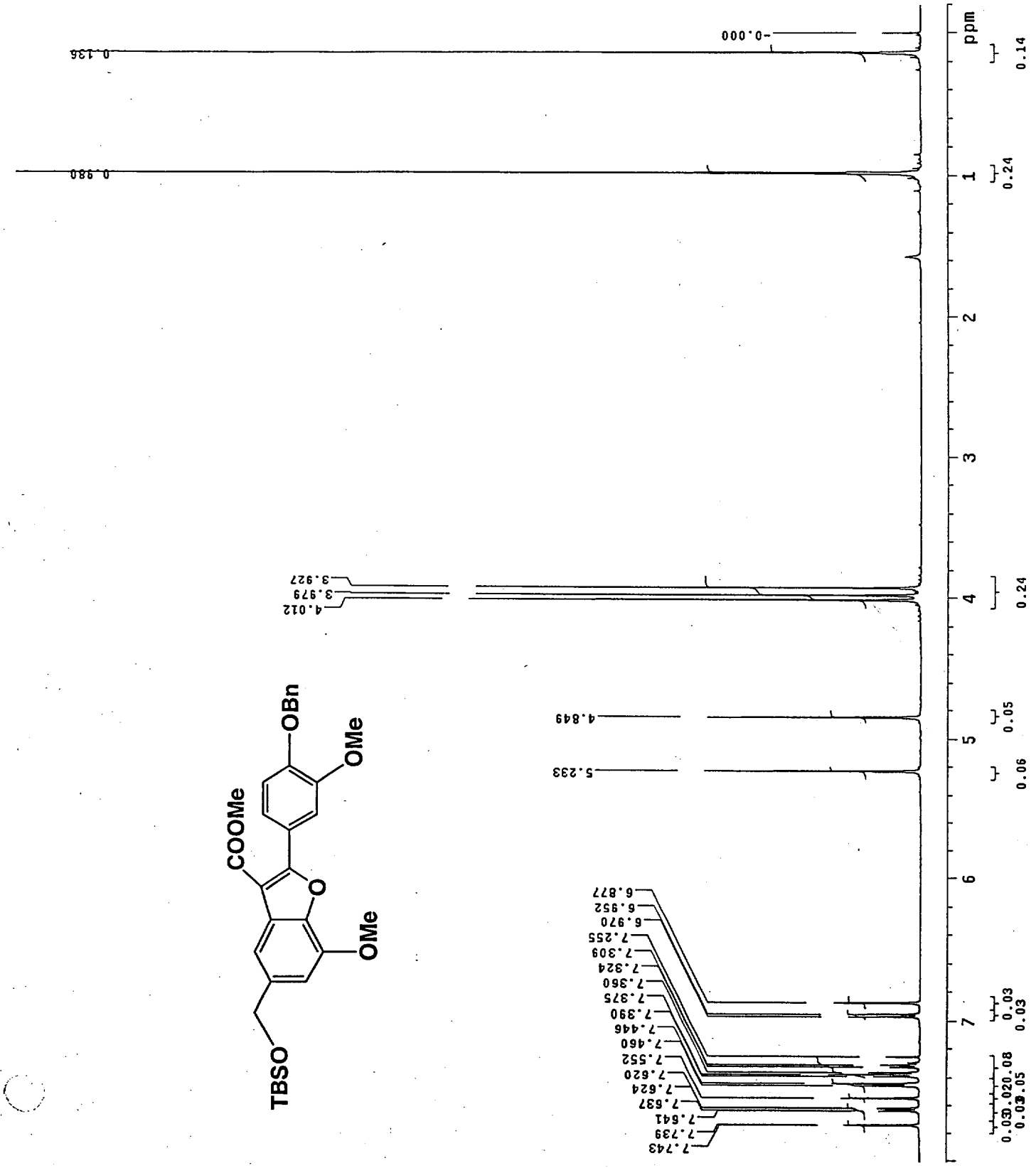
6.697
6.832
6.850
6.880
6.884
7.075
7.088
7.092
7.259
7.289
7.304
7.319
7.352
7.367
7.381
7.423
7.437
ppm
6.6
6.7
6.8
6.9
7.0
7.1
7.2
7.3
7.4
7.5
7.6
0.11
0.13
0.10
0.21
0.09
0.19
0.18


OBn
OMe
OH
OMe
MeOOC
ppm
0
20
40
60
80
100
120
140
160
180
200
30.399
35.717
51.535
55.914
56.023
70.793
76.642
76.900
77.158
82.775
94.282
109.582
111.568
113.499
114.737
115.648
123.618
124.788
127.141
127.821
128.460
131.995
136.607
144.931
146.440
148.576
149.133
173.125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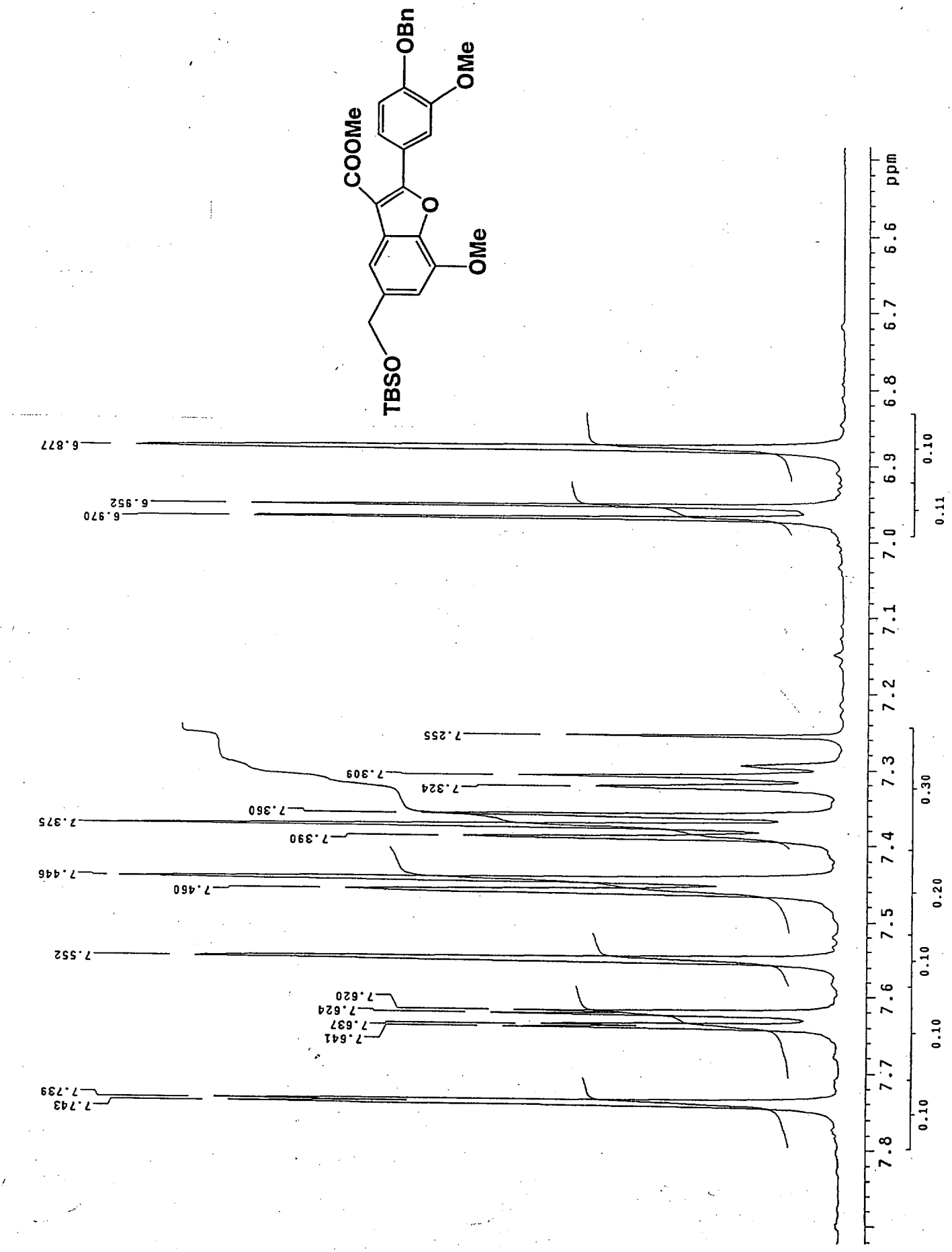
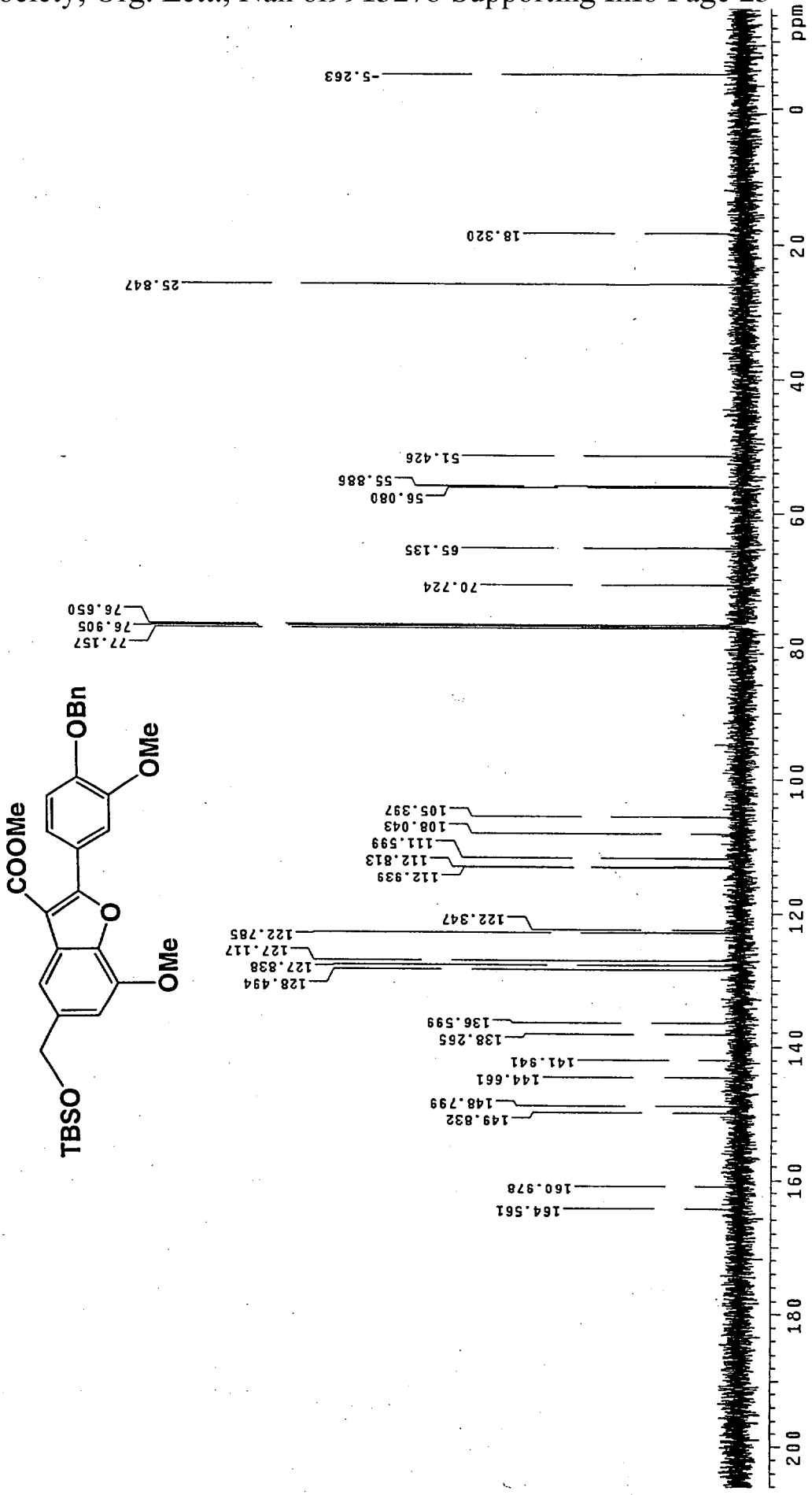
0.136
0.980
3.927
3.979
4.012
4.849
5.233
6.877
6.952
6.970
7.255
7.309
7.324
7.360
7.375
7.390
7.446
7.460
7.552
7.620
7.624
7.637
7.641
7.739
7.743
ppm
0.14
0.24
0.24
0.05
0.06
0.03
0.03
0.03
0.02
0.08
0.05
0.03
OBn
OMe
COOMe
OMe
TBSO

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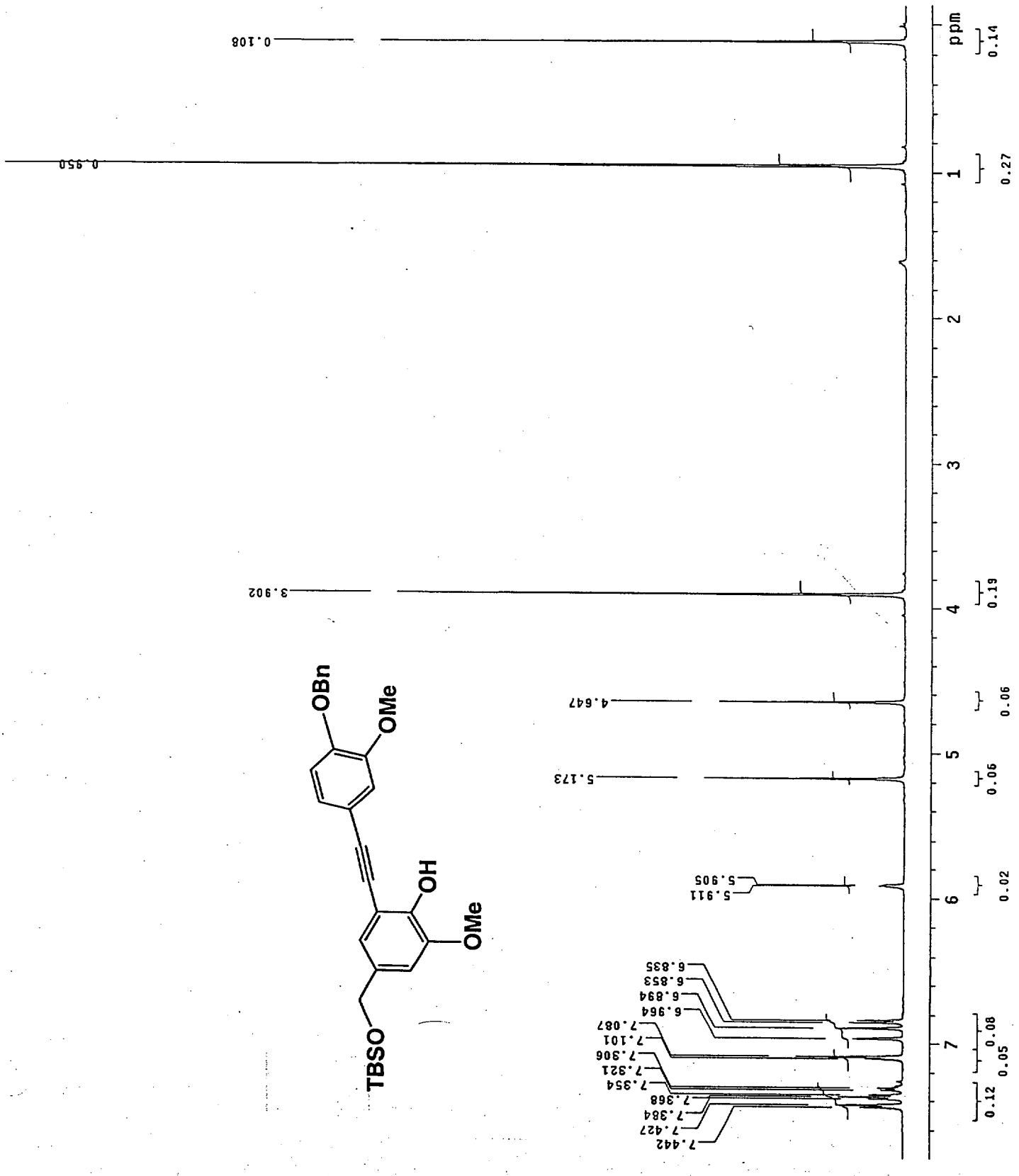
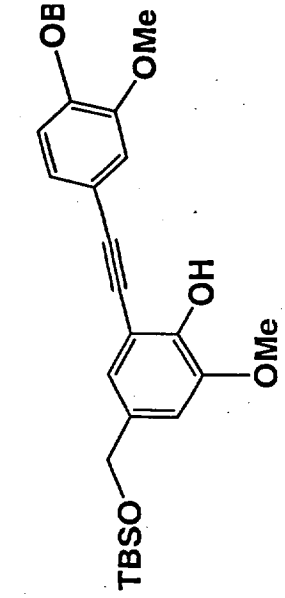
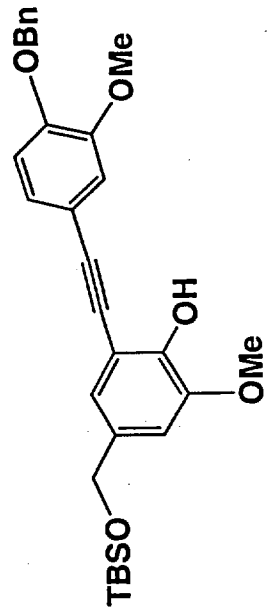
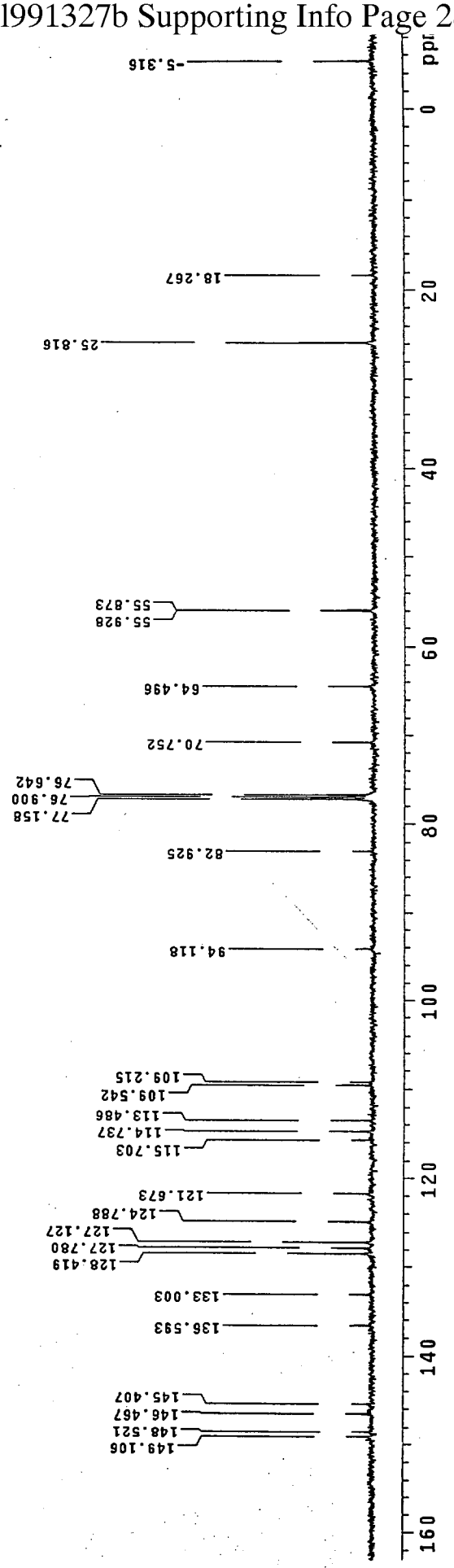
25.847
51.426
55.886
56.080
65.135
70.724
76.650
76.905
77.157
105.397
108.043
111.599
112.813
112.939
122.347
122.785
127.117
127.838
128.494
136.599
138.265
141.941
144.661
148.799
149.832
160.978
164.561
ppm
0
20
40
60
80
100
120
140
160
180
200
OBn
OMe
COOMe
O
OMe
TBSO

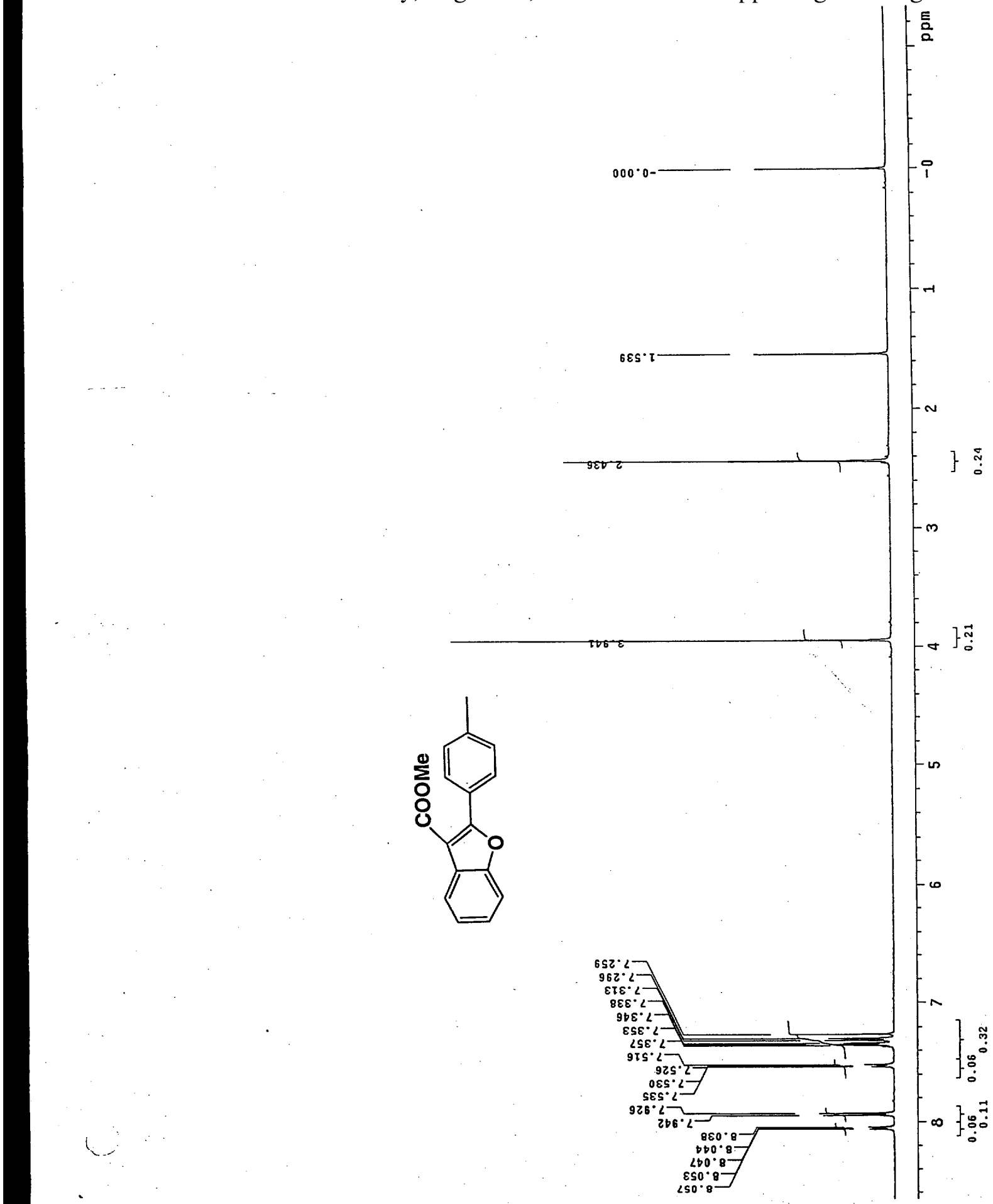



OB
OMe
OH
OMe
TBSO

6.835
6.853
6.894
6.964
7.082
7.101
7.306
7.321
7.354
7.368
7.384
7.427
7.442
ppm
6.8
6.9
7.0
7.1
7.2
7.3
7.4
7.5
7.6
0.13
0.11
0.09
0.21
0.09
0.19
0.18


OBn
OMe
OH
OMe
TBSO


-5.316
18.267
25.816
55.873
55.928
64.496
70.752
76.642
76.900
77.158
82.925
94.118
109.215
109.542
113.486
114.737
115.703
121.673
124.788
127.127
127.780
128.419
133.003
136.593
145.407
146.467
148.521
149.106
ppm
0
20
40
60
80
100
120
140
160


ppm
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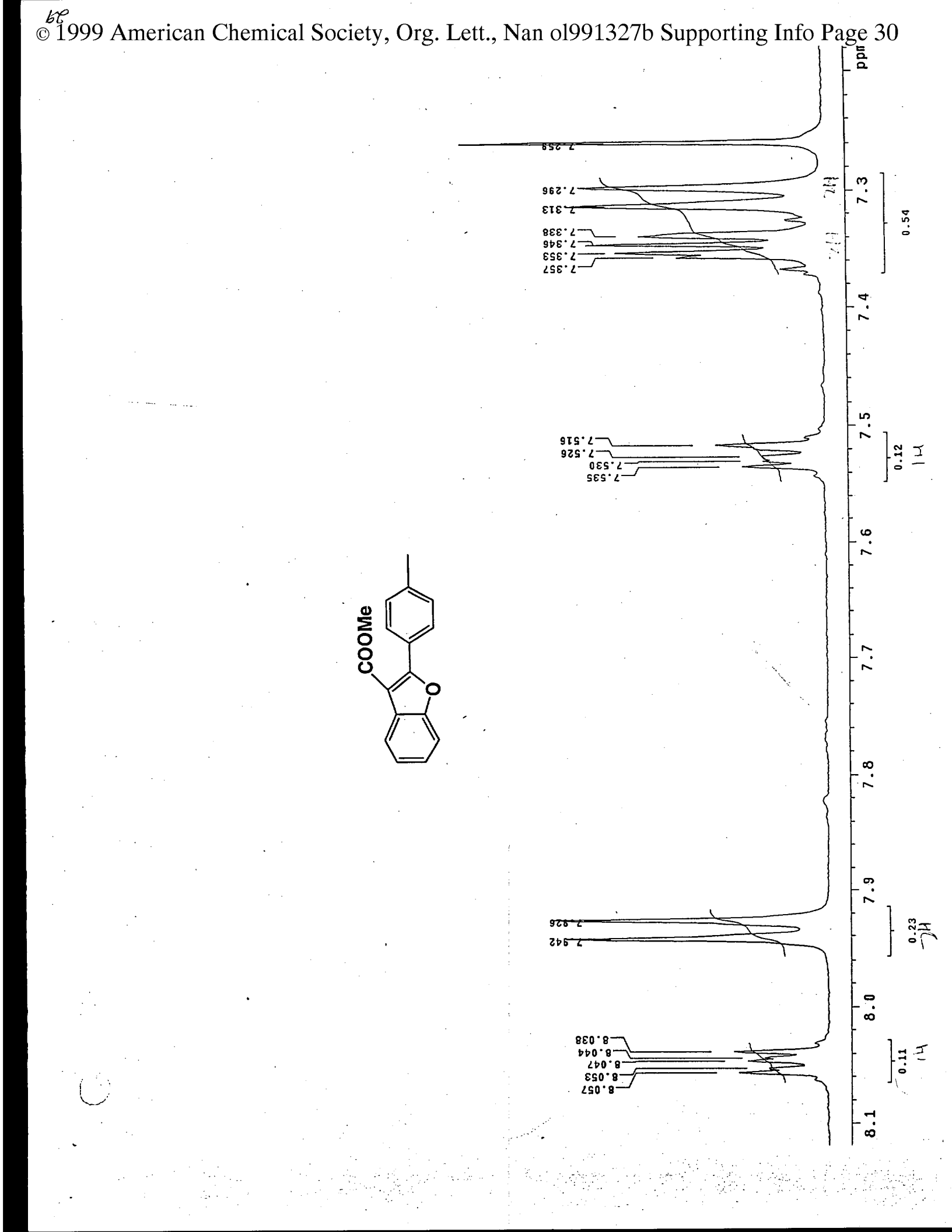
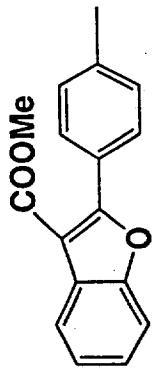
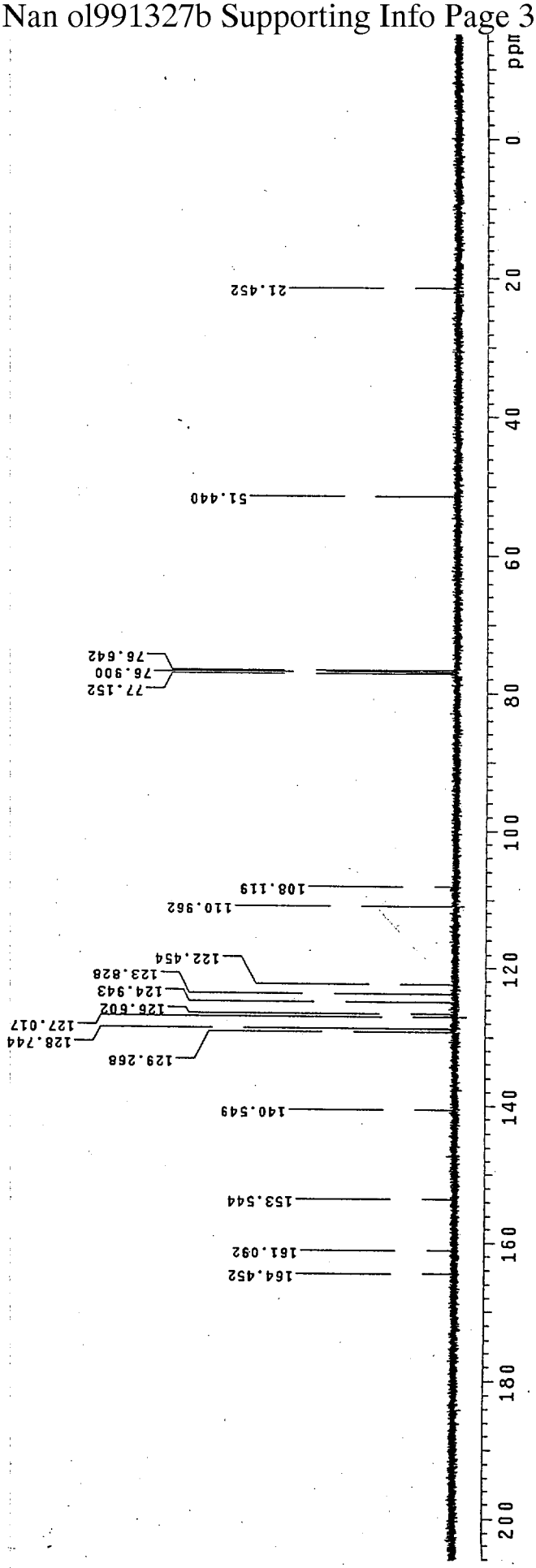
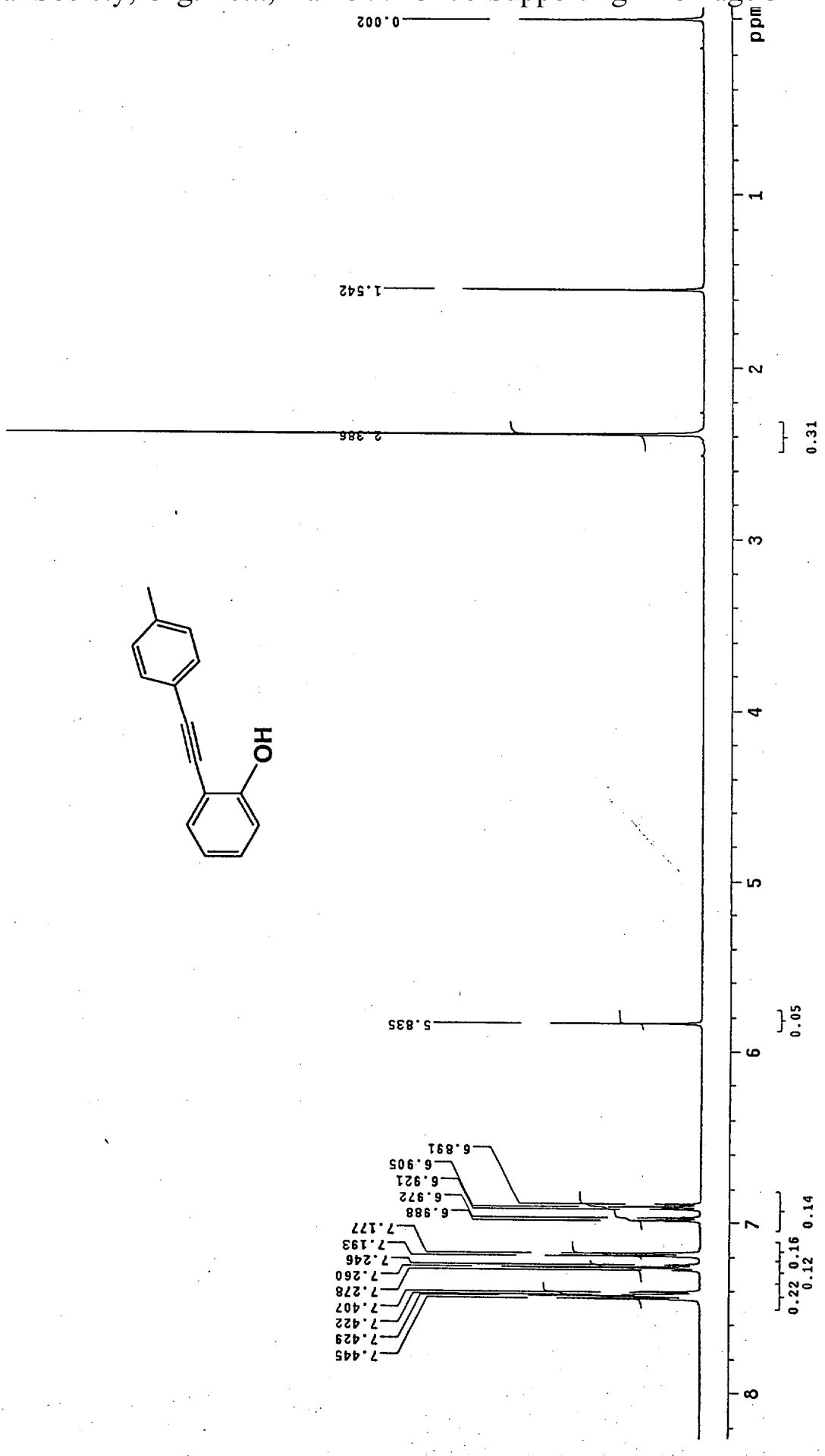
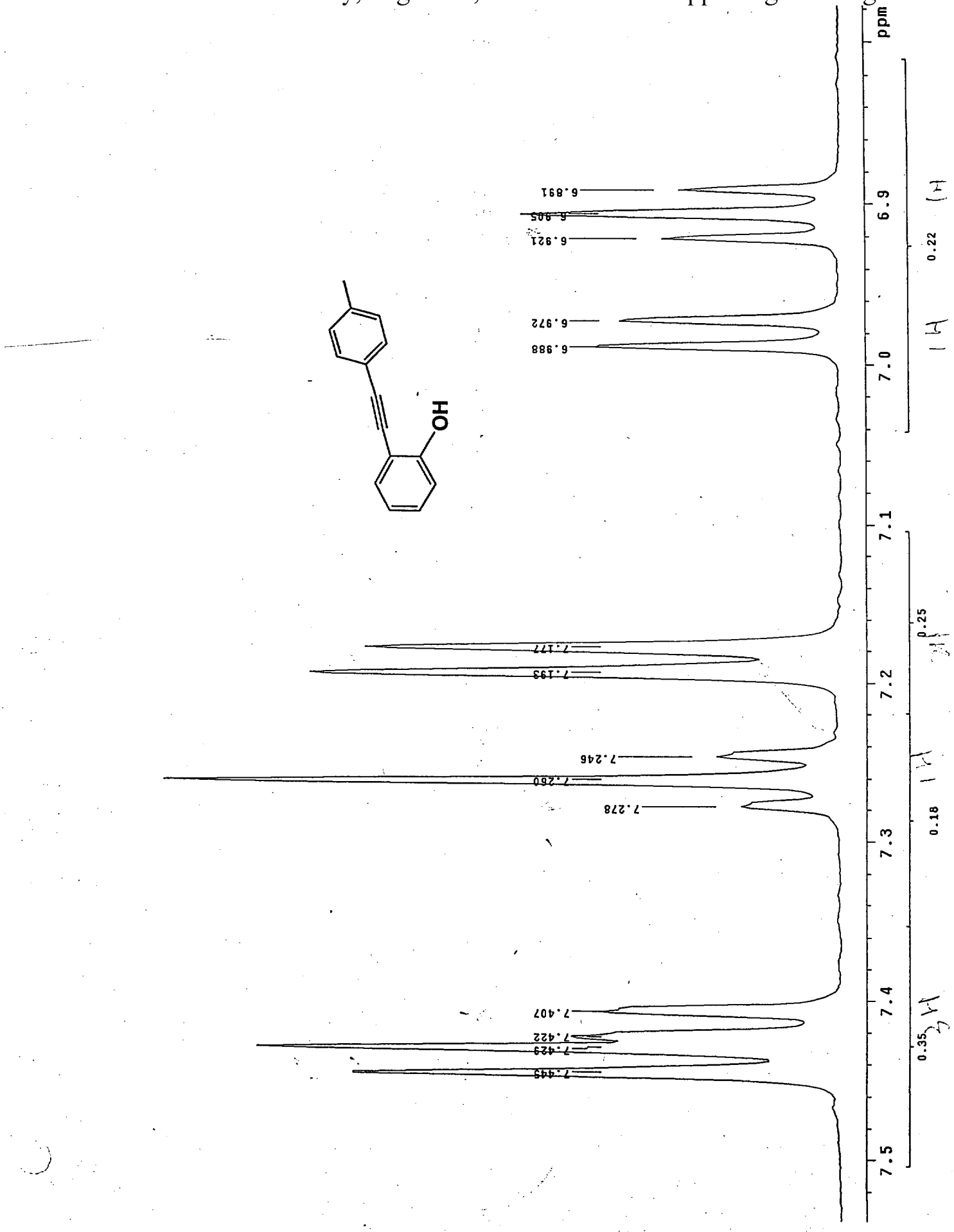
2.436
3.941
7.259
7.296
7.313
7.338
7.346
7.353
7.357
7.516
7.526
7.530
7.535
7.926
7.942
8.038
8.044
8.047
8.053
8.057
0.24
0.21
0.32
0.06
0.11
0.06
COOMe
O

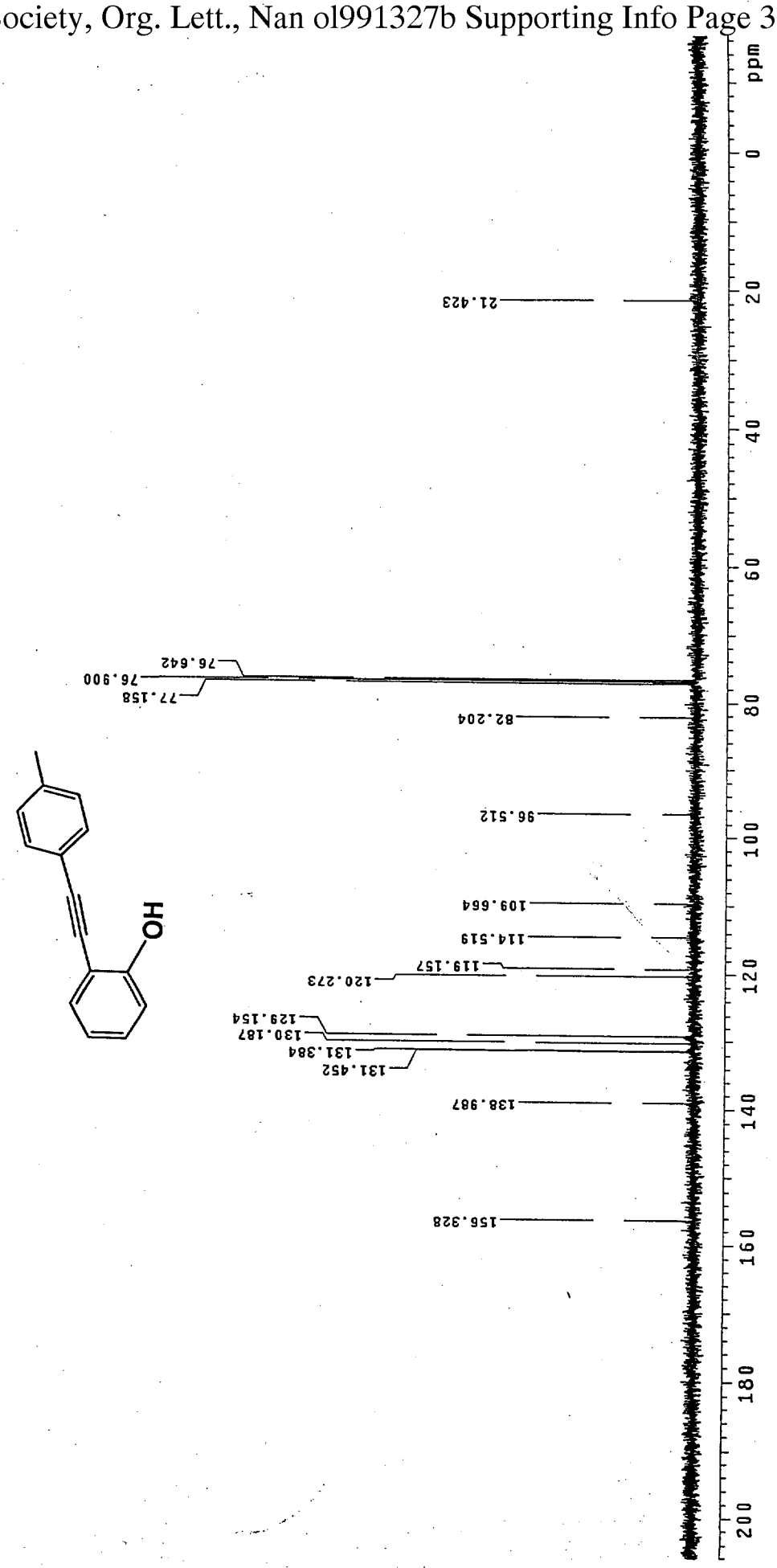

ppm
7.259
7.296
7.313
7.338
7.346
7.353
7.357
7.516
7.526
7.530
7.535
7.926
7.942
8.038
8.044
8.047
8.053
8.057
0.54
0.12
0.23
0.11
COOMe
O

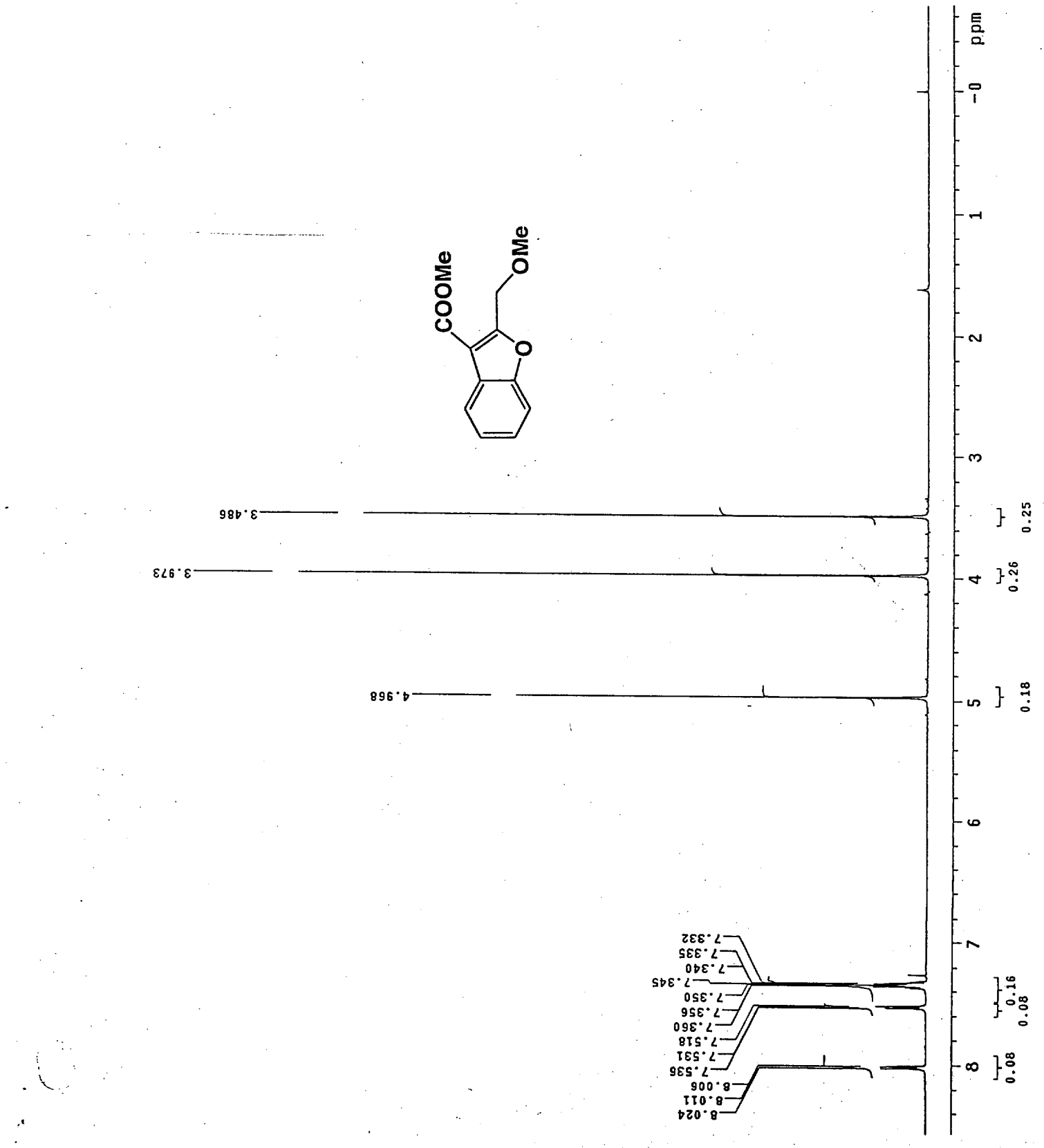


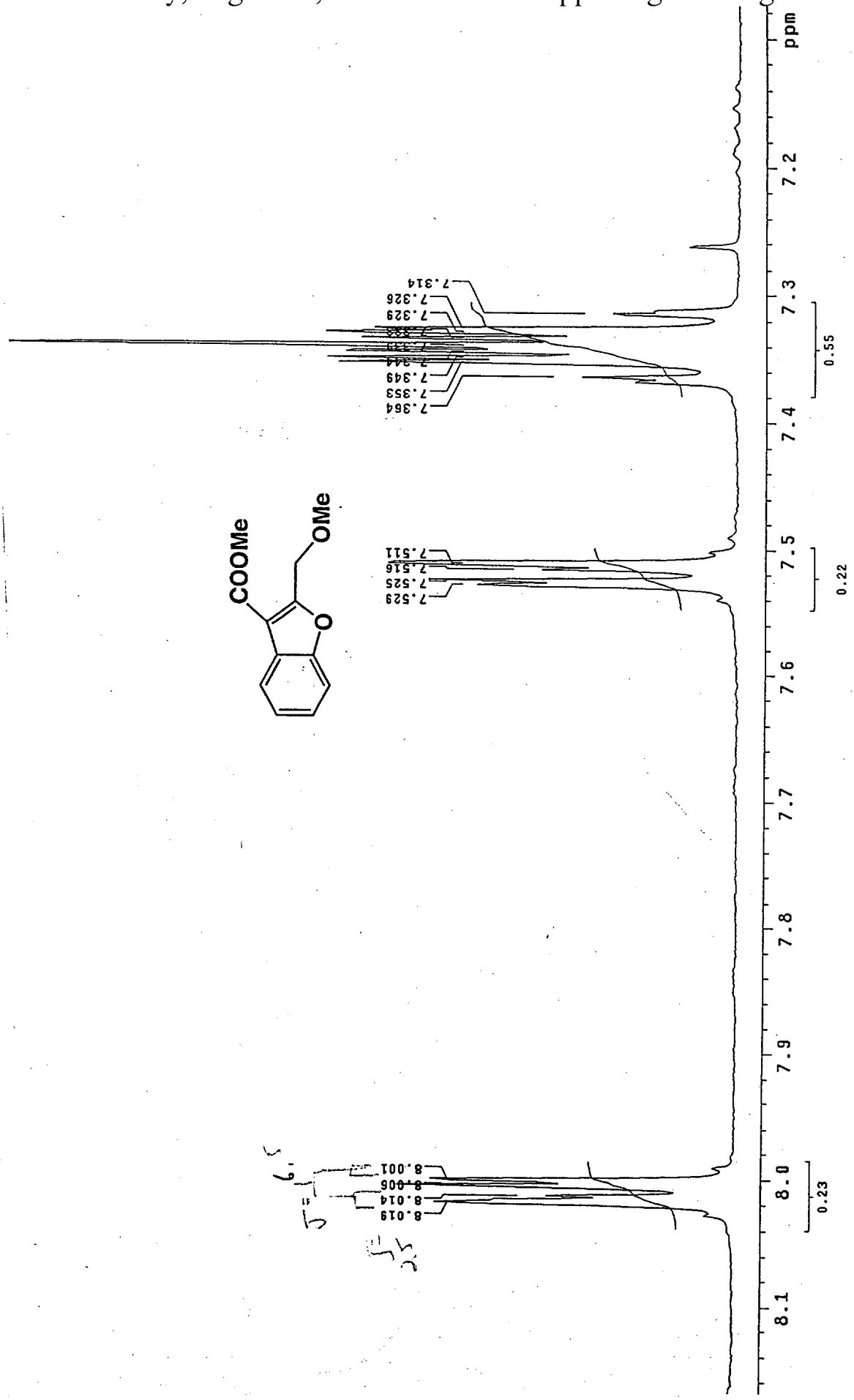


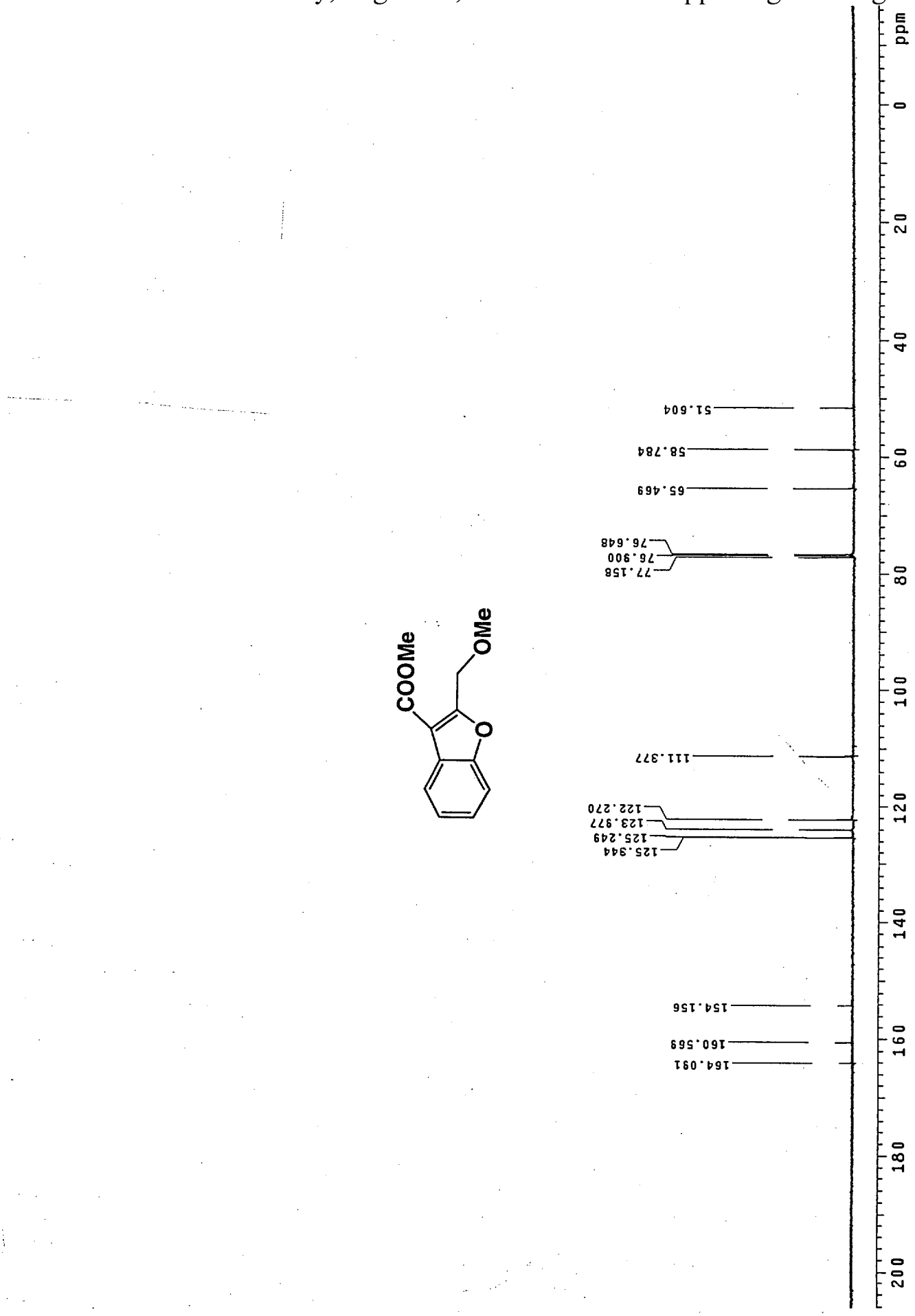


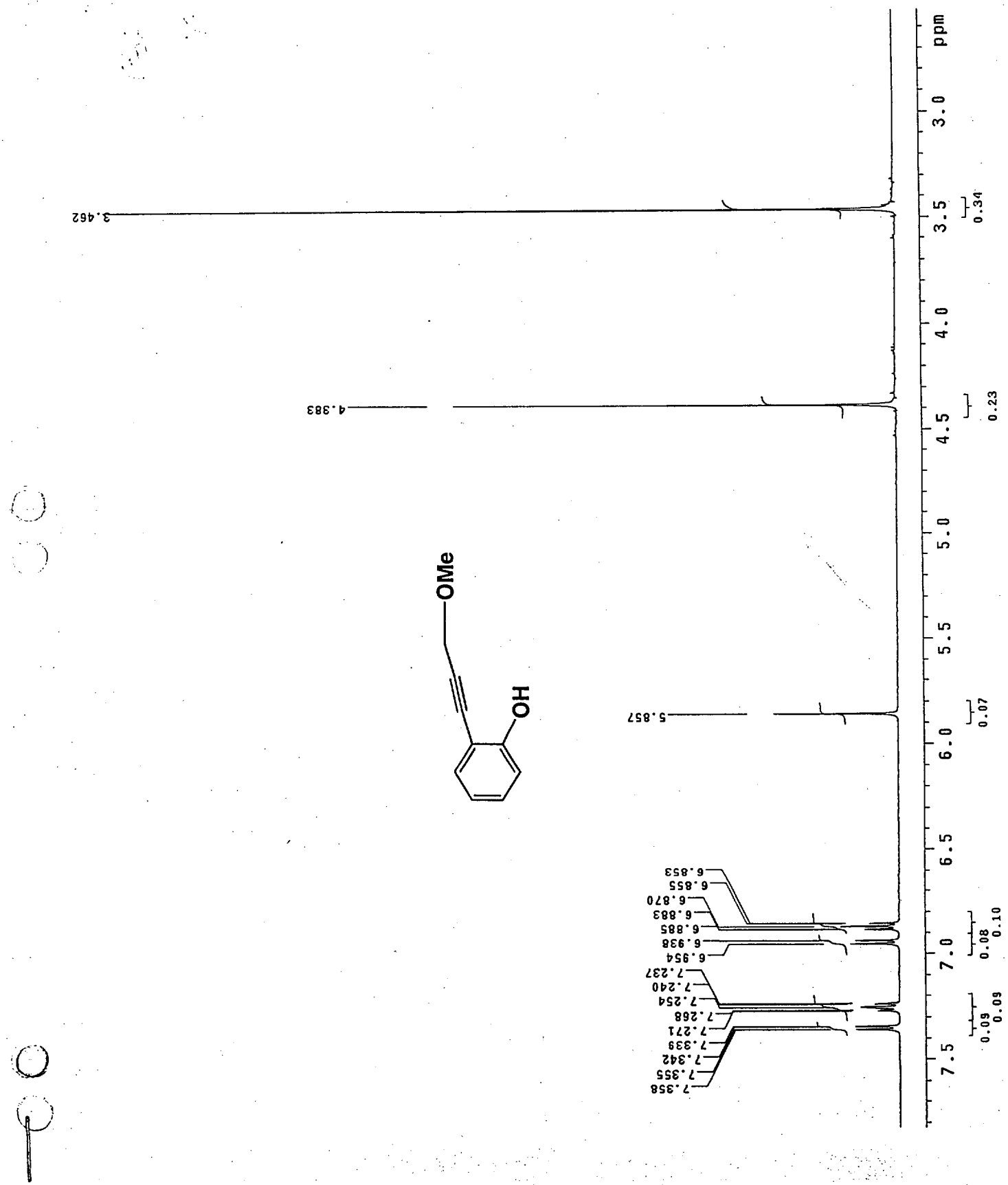


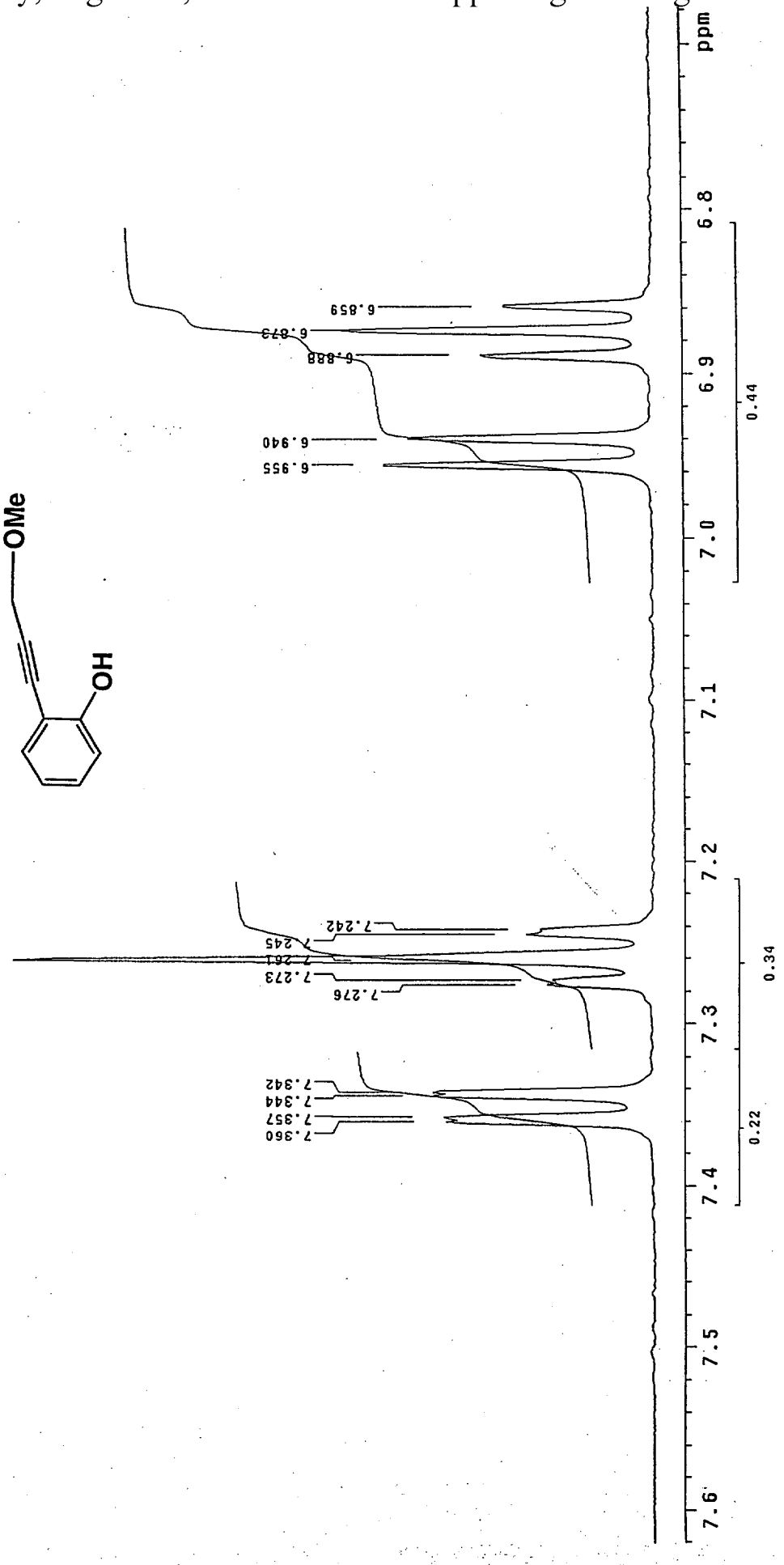



COOMe
OMe
O
3.486
3.973
4.968
8.024
8.011
8.006
7.536
7.531
7.518
7.360
7.356
7.350
7.345
7.340
7.335
7.332
ppm
0
1
2
3
4
5
6
7
8
0.25
0.26
0.18
0.16
0.08
0.08


7.314
7.326
7.329
7.349
7.353
7.364
7.511
7.516
7.525
7.529
8.001
8.006
8.014
8.019
COOMe
OMe
O
ppm
7.2
7.3
7.4
7.5
7.6
7.7
7.8
7.9
8.0
8.1
0.55
0.22
0.23




3.462
4.383
5.857
6.853
6.855
6.870
6.883
6.885
6.938
6.954
7.237
7.240
7.254
7.268
7.271
7.339
7.342
7.355
7.358
OMe
OH
ppm
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
0.34
0.23
0.07
0.08
0.10
0.09
0.09


OMe
OH
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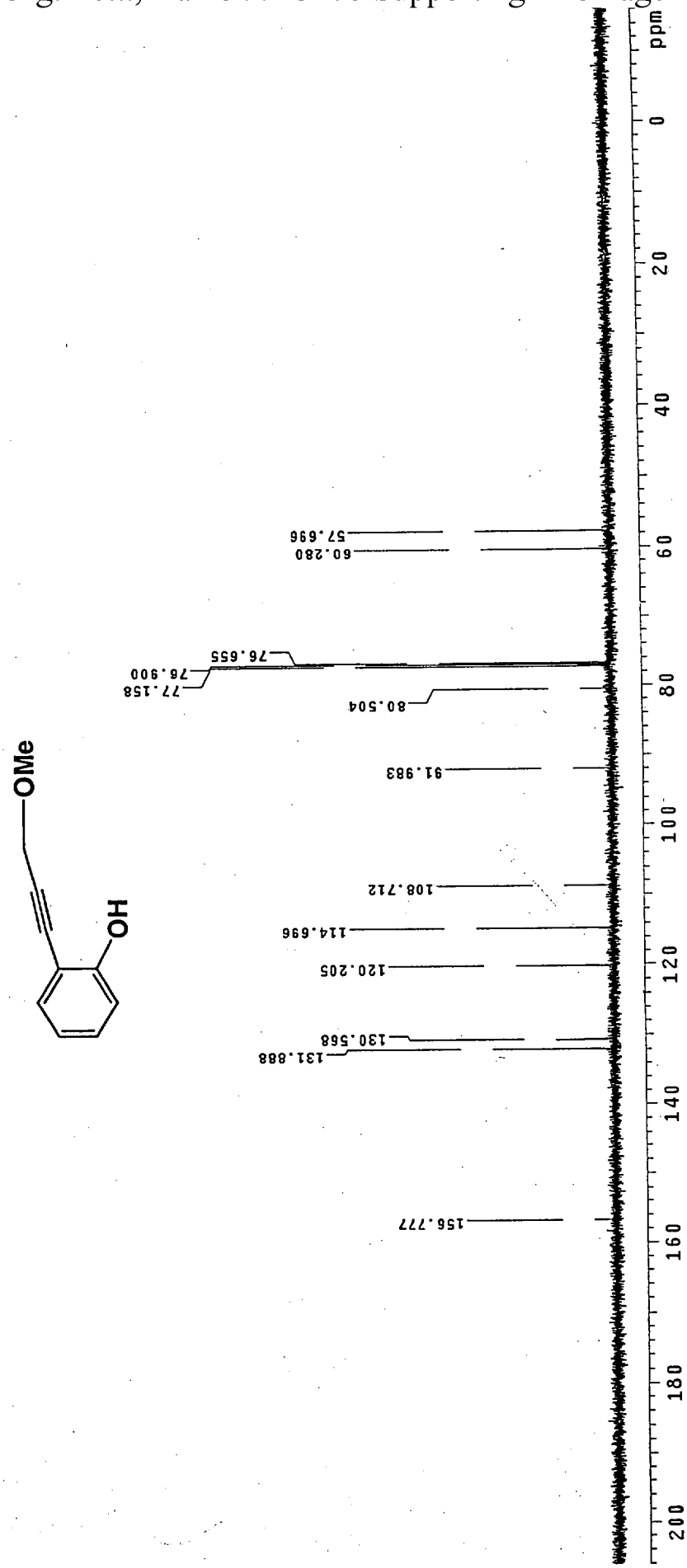
6.873
6.888
6.940
6.955
7.242
7.245
7.261
7.273
7.276
7.342
7.344
7.357
7.360
ppm
6.8
6.9
7.0
7.1
7.2
7.3
7.4
7.5
7.6
0.44
0.34
0.22


OMe
OH
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60.280
76.655
76.900
77.158
80.504
91.983
108.712
114.696
120.205
130.568
131.888
156.777
ppm
0
20
40
60
80
100
120
140
160
180
200