Design, Synthesis, and Biological Evaluation of Water-Soluble Amino Acid Prodrug Conjugates Derived from Combretastatin, Dihydronaphthalene, and Benzosuberene-Based Parent Vascular Disrupting Agents

Laxman Devkota[a,†], Chen-Ming Lin[a,†], Tracy E. Strecker[a], Yifan Wang[a], Justin K. Tidmore[a], Zhi Chen[a], Rajsekhar Guddneppanavar[a], Christopher J. Jelinek[a], Ramona Lopez[b], Li Liu[b], Ernest Hamel[c], Ralph P. Mason[b], David J. Chaplin[a,d], Mary Lynn Trawick[a,*], and Kevin G. Pinney[a,*]

[a] Department of Chemistry and Biochemistry, Baylor University, One Bear Place #97348, Waco, Texas 76798-7348, United States

[b] Department of Radiology, The University of Texas Southwestern Medical Center, 5323 Harry Hines Boulevard, Dallas, Texas 75390-9058, United States

[c] Screening Technologies Branch, Developmental Therapeutics Program, Division of Cancer Treatment and Diagnosis, National Cancer Institute, Frederick National Laboratory for Cancer Research, National Institutes of Health, Frederick, MD 21702, United States

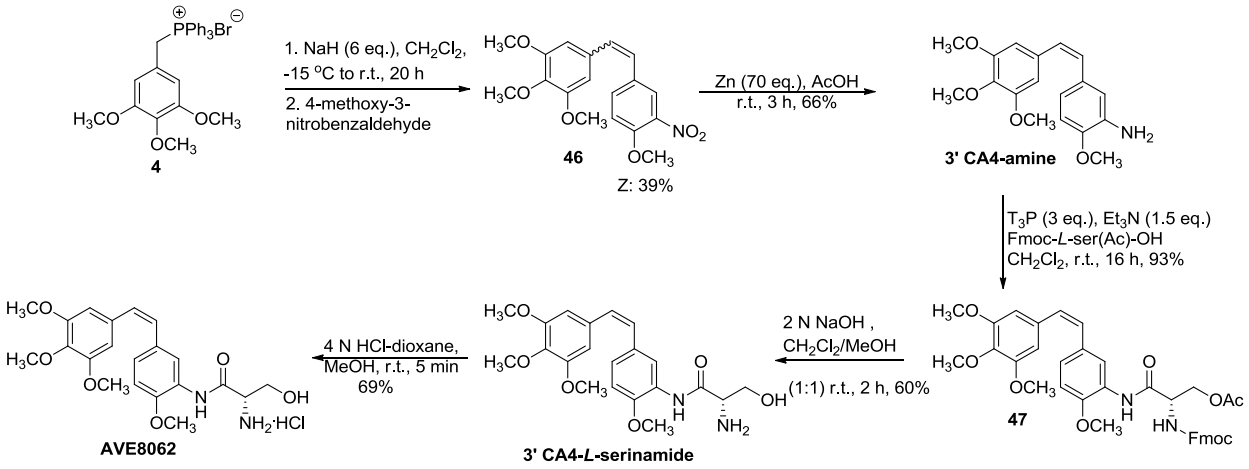
[d] OXiGENE Inc., 701 Gateway Boulevard, Suite 210, South San Francisco, California 94080, United States

*Co-corresponding authors. Tel.: +1 254 710 4117; Kevin_Pinney@baylor.edu and Tel.: +1 254 710 6857; Mery_Lynn_Trawick@baylor.edu

[†]Authors contributed equally

Table of Contents

Synthesis of AVE8062



Scheme. Synthesis of AVE8062

Experimental Section for the Synthesis of AVE8062

(*Z*)-1,2,3-trimethoxy-5-(4-methoxy-3-nitrostyryl)benzene (46)

NaH (1.54 g, 64.2 mmol) was added into an oven-dried round-bottom reaction flask. Anhydrous CH_2Cl_2 (100 mL) and 3,4,5-trimethoxybenzyltriphenylphosphonium bromide (6.94 g, 13.3 mmol) were added to the reaction flask, and the reaction mixture was stirred for 1 h. The reaction mixture was cooled to -15 ºC, and 4-methoxy-3-nitrobenzaldehyde (2.01 g 11.1 mmol) was added to the reaction flask. The reaction mixture was stirred for 20 h while warming to ambient temperature under N_2. Water (100 mL) was slowly added to the reaction, and the product was extracted with CH_2Cl_2 (3 × 100 mL). The organic phase was rinsed with brine, dried with Na_2SO_4, and concentrated under reduced pressure, and the residue was purified by flash column chromatography using a pre-packed 100 g silica column [solvent A: EtOAc; solvent B: hexanes; gradient: 10%A / 90%B (1 CV), 10%A / 90%B → 70%A / 30%B (10 CV), 70%A / 30%B (5 CV); flow rate: 100 mL/min; monitored at 254 and 280 nm] affording 3' CA4-nitro **46** (1.51 g,

4.34 mmol, 39% yield) as a yellow solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.80 (1H, d, *J*=2.0 Hz), 7.44 (1H, dd, *J*=8.7, 2.1 Hz), 6.95 (1H, d, *J*=8.7 Hz), 6.58 (1H, d, *J*=12.1 Hz), 6.47 (2H, s), 6.45 (1H, d, *J*=12.1 Hz), 3.94 (3H, s), 3.85 (3H, s), 3.72 (6H, s). ^{13}C NMR (150 MHz, $CDCl_3$) δ 153.50, 152.00, 139.67, 137.89, 134.99, 132.12, 131.59, 129.97, 127.15, 126.29, 113.36, 106.03, 61.29, 56.88, 56.31.

(*Z*)-2-methoxy-5-(3,4,5-trimethoxystyryl)aniline (3' CA4-amine)

To a well-stirred solution of 3' CA4-nitro **46** (0.560 g, 1.78 mmol) in AcOH (40 mL), Zn (8.20 g, 125 mmol) was added, and the reaction mixture was stirred for 3 h at room temperature. The reaction was filtered using Celite®, and the Celite® was washed with EtOAc. The filtrate was concentrated under reduced pressure, and the residue was purified by flash column chromatography using a pre-packed 25 g silica column [solvent A: EtOAc; solvent B: hexanes; gradient: 7%A / 93%B (1 CV), 7%A / 93%B → 60%A / 40%B (15 CV), 60%A / 40%B (2 CV); flow rate: 75 mL/min; monitored at 254 and 280 nm] affording 3' CA4-amine (0.370 g, 1.17 mmol, 66% yield) as a brown solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 6.71 – 6.65 (2H, m), 6.67 (1H, d, *J*=1.8 Hz), 6.55 (2H, s), 6.45 (1H, d, *J*=12.2 Hz), 6.37 (1H, d, *J*=12.2 Hz), 3.84 (3H, s), 3.82 (3H, s), 3.70 (6H, s). ^{13}C NMR (150 MHz, $CDCl_3$) δ 153.09, 146.91, 137.18, 136.02, 133.30, 130.32, 130.29, 128.64, 119.82, 115.53, 110.29, 106.20, 61.23, 56.20, 55.80. HRMS (ESI) calculated for $C_{18}H_{21}NO_4$ 315.1471, $(M+H)^+$ 316.1549, found 316.1543.

(*S*,*Z*)-2-((((9*H*-fluoren-9-yl)methoxy)carbonyl)amino)-3-((2-methoxy-5-(3,4,5-trimethoxystyryl)phenyl)amino)-3-oxopropyl acetate (47)

3' CA4-amine (0.11 g, 0.35 mmol) was dissolved in CH_2Cl_2 (10 mL), and then Fmoc-serine acetate (0.19 g, 0.52 mmol), T3P (0.62 mL, 1.0 mmol), and Et_3N (0.073 mL, 0.52 mmol) were added. After stirring for 16 h at room temperature, water (10 mL) was added, and the reaction

mixture was extracted with CH_2Cl_2 (3 × 10 mL). The combined organic phase was rinsed with brine, dried with Na_2SO_4, and concentrated under reduced pressure, and the residue was purified by flash column chromatography using a pre-packed 10 g silica column [solvent A: EtOAc; solvent B: hexanes; gradient: 12%A / 88%B (1 CV), 12%A / 88%B → 100%A / 0%B (10 CV), 100%A / 0%B (2 CV); flow rate: 36 mL/min; monitored at 254 and 280 nm] affording the desired Fmoc-*L*-serinamide acetate **47** (0.22 g, 0.32 mmol, 93%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.42 (1H, s), 8.31 (1H, d, *J*=1.8 Hz), 7.77 (2H, d, *J*=7.0 Hz), 7.60 (2H, d, *J*=6.7 Hz), 7.40 (2H, t, *J*=7.0 Hz), 7.31 (2H, d, *J*=5.6 Hz), 7.03 (1H, d, *J*=8.2 Hz), 6.70 (1H, d, *J*=8.5 Hz), 6.51 (2H, s), 6.50 (2H, d, *J*=12.9 Hz), 6.45 (1H, d, *J*=12.1 Hz), 5.79 (1H, d, *J*=7.0 Hz), 4.65 (1H, d, *J*=4.8 Hz), 4.53 – 4.48 (2H, m), 4.35 – 4.30 (1H, m), 4.24 (1H, t, *J*=7.0 Hz), 4.02 (1H, dd, *J*=9.4, 2.7 Hz), 3.84 (3H, s), 3.79 (3H, s), 3.68 (6H, s), 2.09 (3H, s). ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.99, 166.74, 153.17, 147.41, 143.93, 143.84, 141.59, 137.32, 133.05, 130.45, 129.75, 129.61, 128.13, 127.42, 125.35, 125.28, 121.14, 120.37, 120.35, 109.87, 106.19, 67.78, 64.23, 61.21, 56.19, 56.16, 55.01, 47.34, 21.05.

(*S,Z*)-2-amino-3-hydroxy-*N*-(2-methoxy-5-(3,4,5-trimethoxystyryl)phenyl)propanamide (3' CA4-*L*-serinamide)

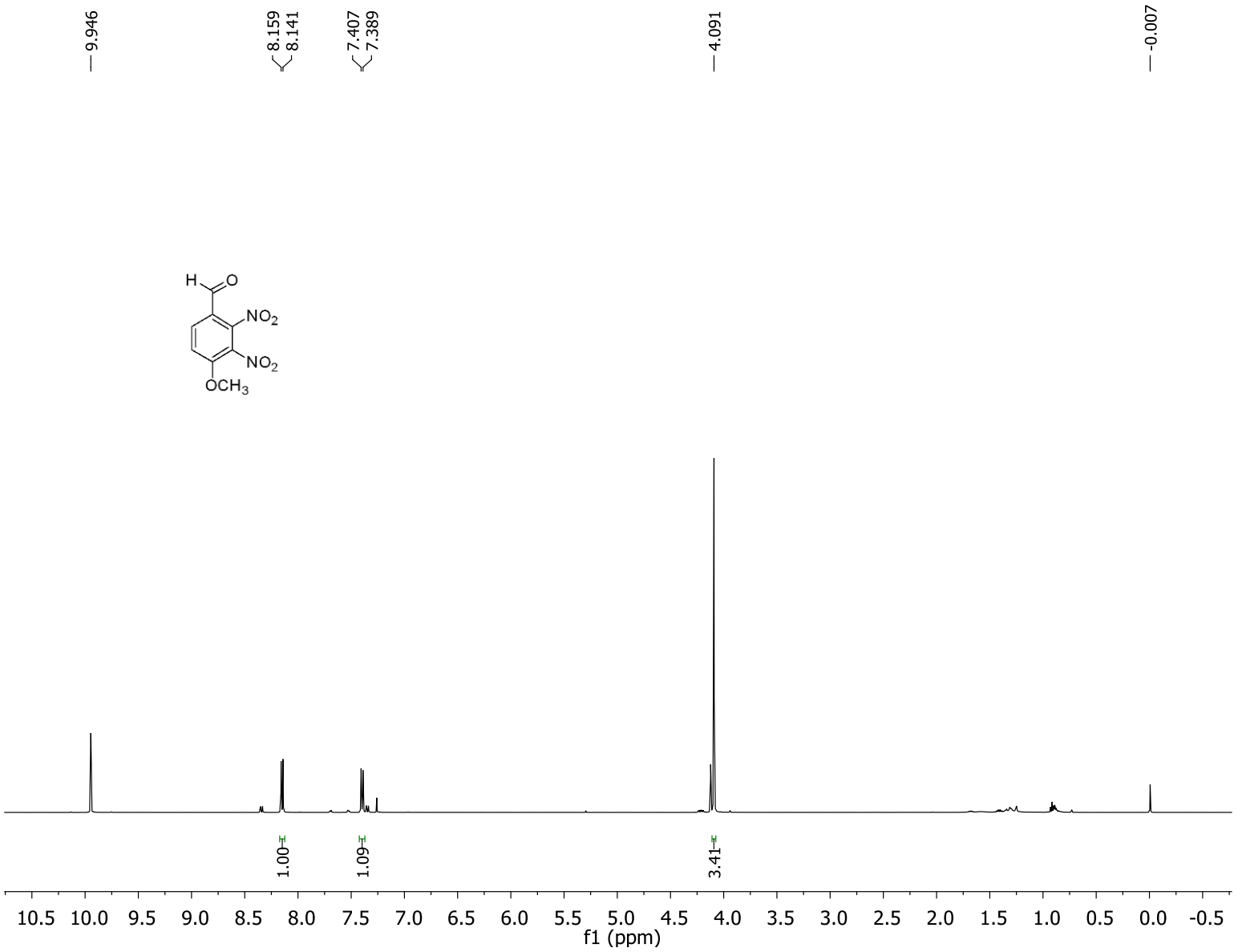
To a well-stirred solution of Fmoc-*L*-serinamide acetate **47** (0.22 g, 0.32 mmol) in CH_2Cl_2/MeOH mixture (4 mL, 1:1 ratio), 2 N NaOH (2.00 eq.) was added. The reaction mixture was stirred for 2 h at room temperature under N_2. The solvent was evaporated under reduced pressure, and sat. $NaHCO_3$ (10 mL) was added. The solution was extracted with CH_2Cl_2 (3 × 10 mL), and then the combined organic phase was rinsed with brine, dried with Na_2SO_4, and concentrated under reduced pressure. The residue was purified by preparative TLC [CH_2Cl_2/MeOH mixture (9:1 ratio)] to give the desired 3' CA4-*L*-serinamide prodrug (0.077 g,

0.19 mmol, 60%) as a yellow oil. ^{1}H NMR (600 MHz, CD_3OD) δ 8.21 (1H, d, *J*=1.9 Hz), 6.97 (1H, d, *J*=8.4 Hz), 6.88 (1H, d, *J*=8.3 Hz), 6.53 (2H, s), 6.49 (1H, d, *J*=12.1 Hz), 6.43 (1H, d, *J*=12.1 Hz), 3.84 (3H, s), 3.72 (2H, d, *J*=6 Hz), 3.71 (3H, s), 3.61 (6H, s), 3.49 (1H, t, *J*=5.3 Hz). ^{13}C NMR (CD_3OD, 150 MHz) δ 172.37, 152.72, 148.32, 136.81, 132.96, 129.88, 129.24, 128.95, 126.76, 124.82, 120.56, 109.89, 106.02, 63.84, 59.74, 57.03, 55.04, 54.93. HRMS (ESI) calculated for $C_{21}H_{26}N_2O_6$ 402.1791, $(M+H)^+$ 403.1864, found 403.1869.

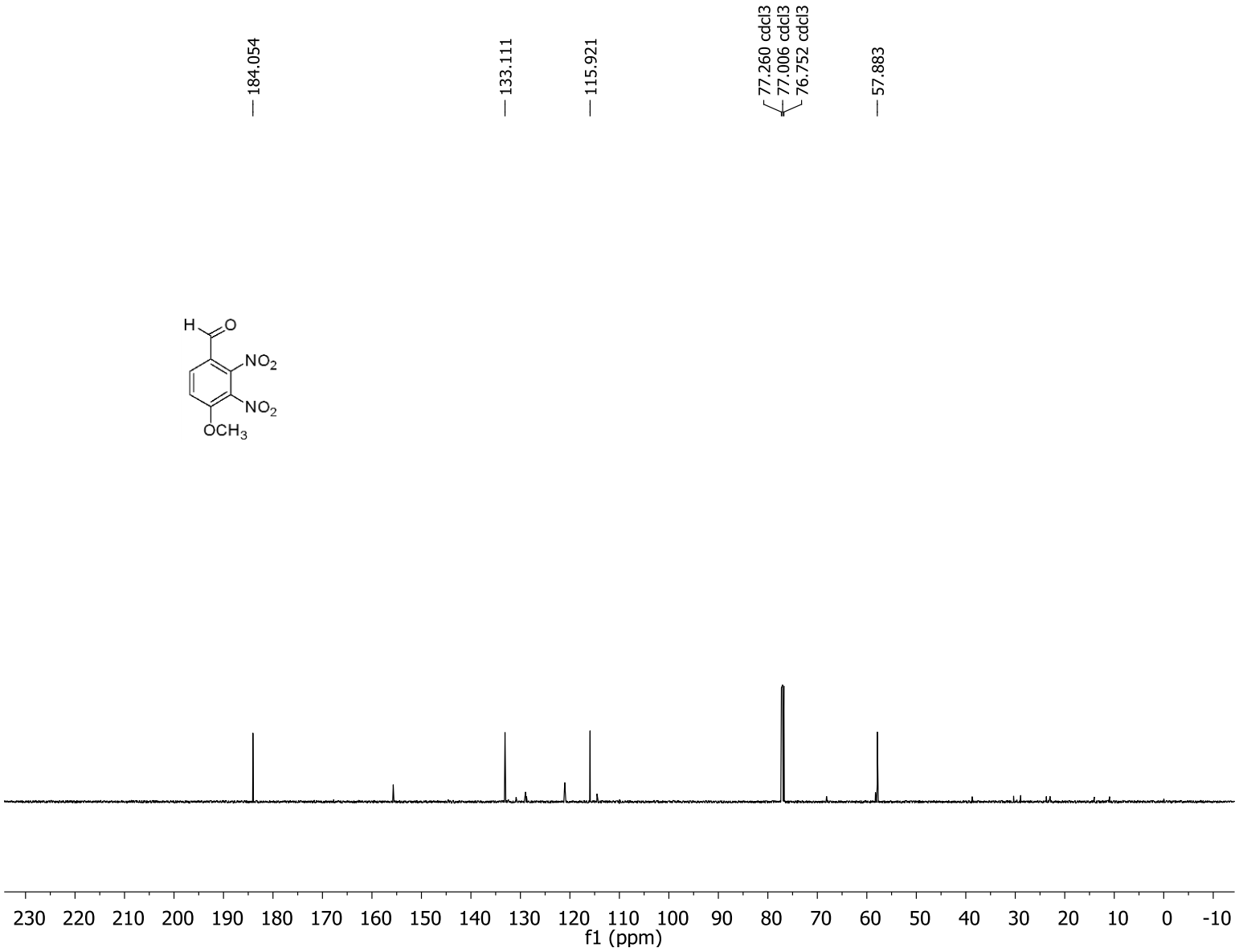
(*S,Z*)-2-amino-3-hydroxy-*N*-(2-methoxy-5-(3,4,5-trimethoxystyryl)phenyl)propanamide hydrochloride (AVE8062)

3' CA4-*L*-serinamide prodrug (0.027 g, 0.067 mmol) was dissolved in MeOH (0.50 mL), and 4 N HCl in dioxane (0.08 mL, 0.32 mmol) was added to the solution. After stirring for 5 min, the solvent was evaporated to dryness, and the residue was washed with diethyl ether (3 × 2 mL) to give the desired 3' CA4-*L*-serinamide prodrug salt **AVE8062** (0.020 g, 0.046 mmol, 69%) as a colorless solid. ^{1}H NMR (600 MHz, CD_3OD) δ 7.97 (1H, d, *J*=2.0 Hz), 7.04 (1H, dd, *J*=8.5, 2.0 Hz), 6.93 (1H, d, *J*=8.5 Hz), 6.52 (2H, s), 6.49 (1H, d, *J*=12.2 Hz), 6.45 (1H, d, *J*=12.2 Hz), 4.14 (1H, dd, *J*=6.3, 5.0 Hz), 3.94 (1H, dd, *J*=11.2, 4.7 Hz), 3.87 – 3.83 (4H, m), 3.71 (3H, s), 3.62 (6H, s). ^{13}C NMR (150 MHz, CD_3OD) δ 165.22, 152.77, 149.21, 136.74, 132.96, 129.80, 129.07, 128.93, 126.15, 125.87, 122.42, 110.28, 105.90, 60.38, 59.78, 55.04, 55.02, 54.93. HRMS (ESI) calculated for $C_{21}H_{27}ClN_2O_6$ 438.1558, $(M-Cl)^+$ 403.1864, found 403.1865.

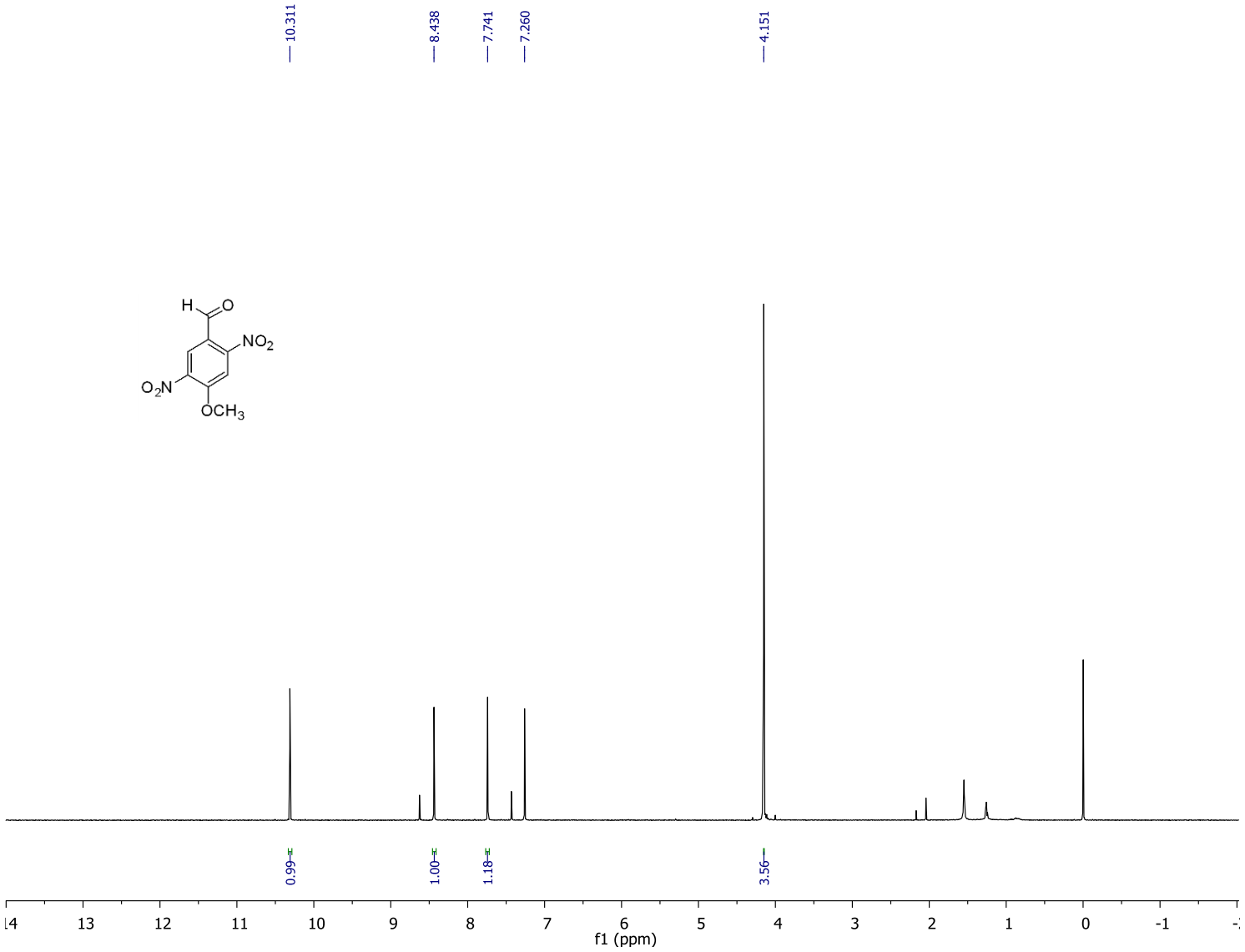
^{1}H NMR ($CDCl_3$, 500 MHz) Compound **1**



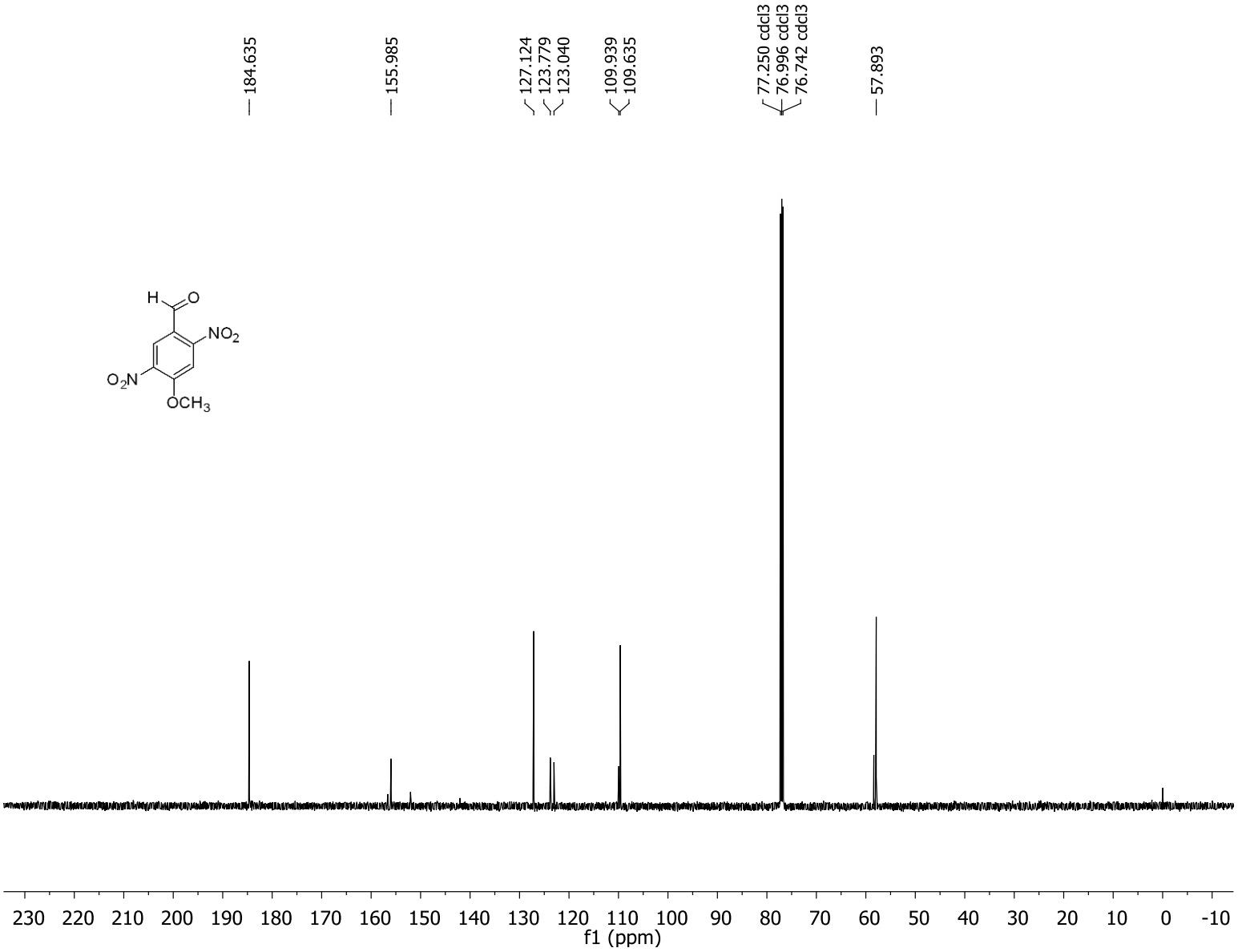
^{13}C NMR ($CDCl_3$, 125 MHz) Compound **1**



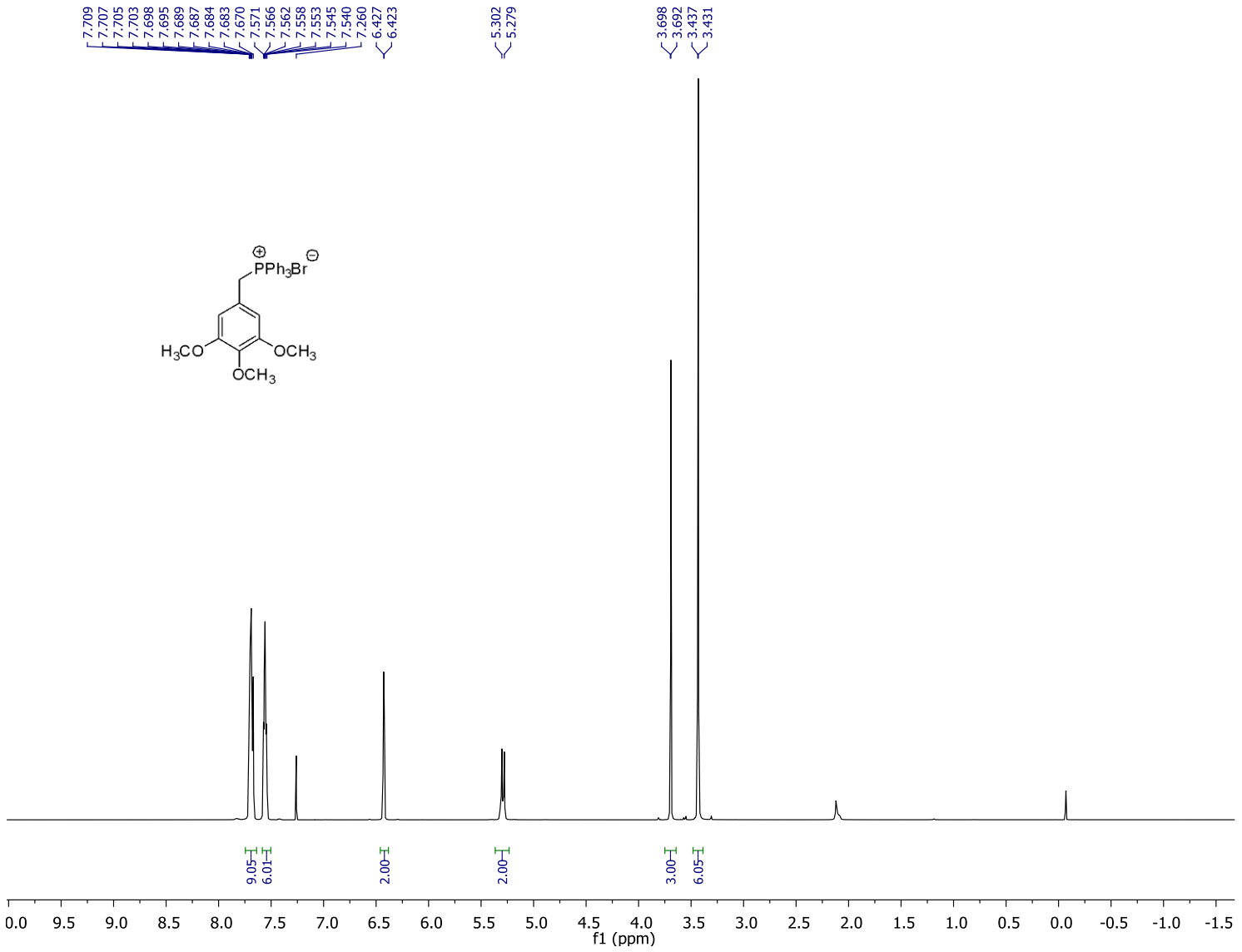
^{1}H NMR ($CDCl_3$, 500 MHz) Compound **2**



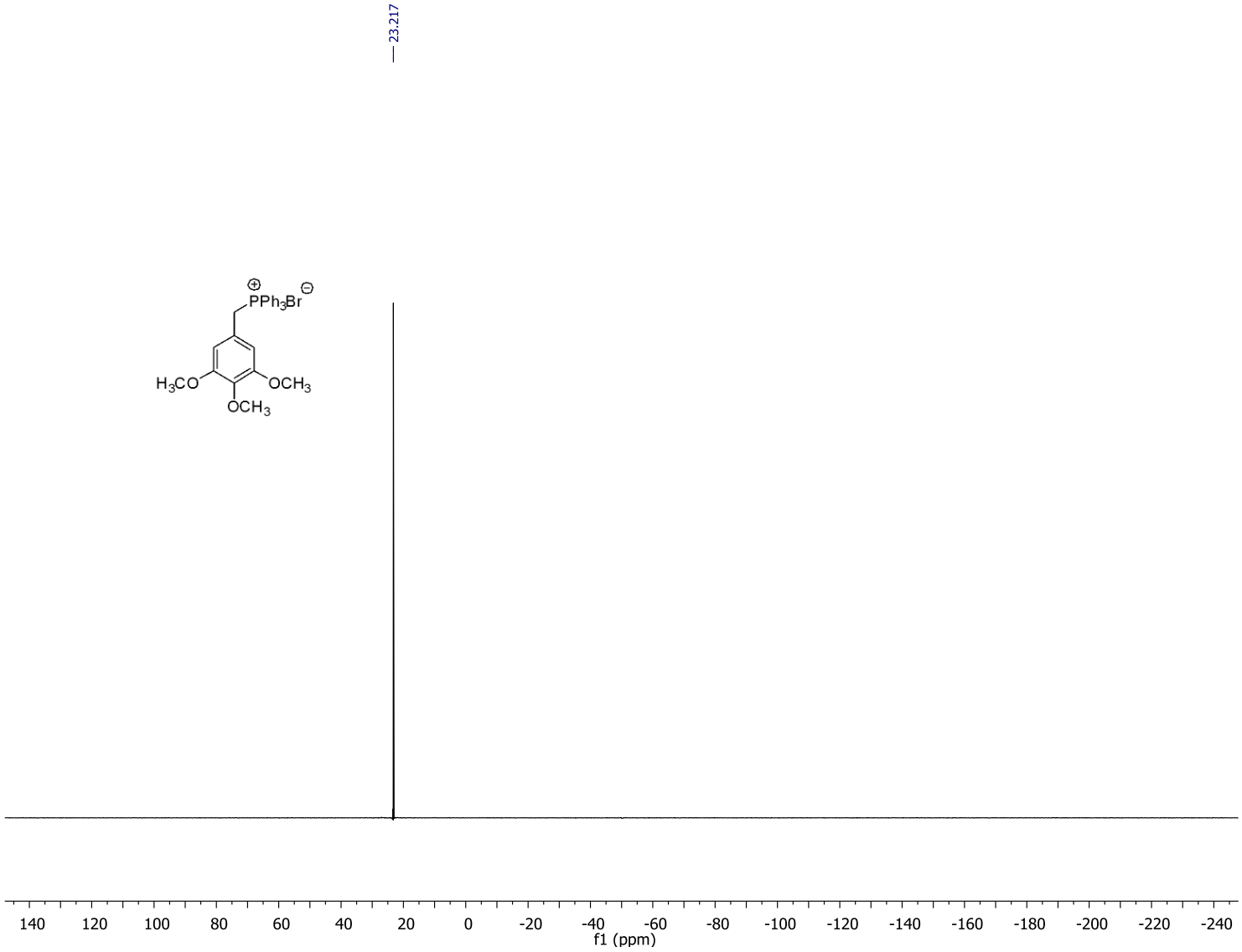
^{13}C NMR ($CDCl_3$, 125 MHz) Compound **2**



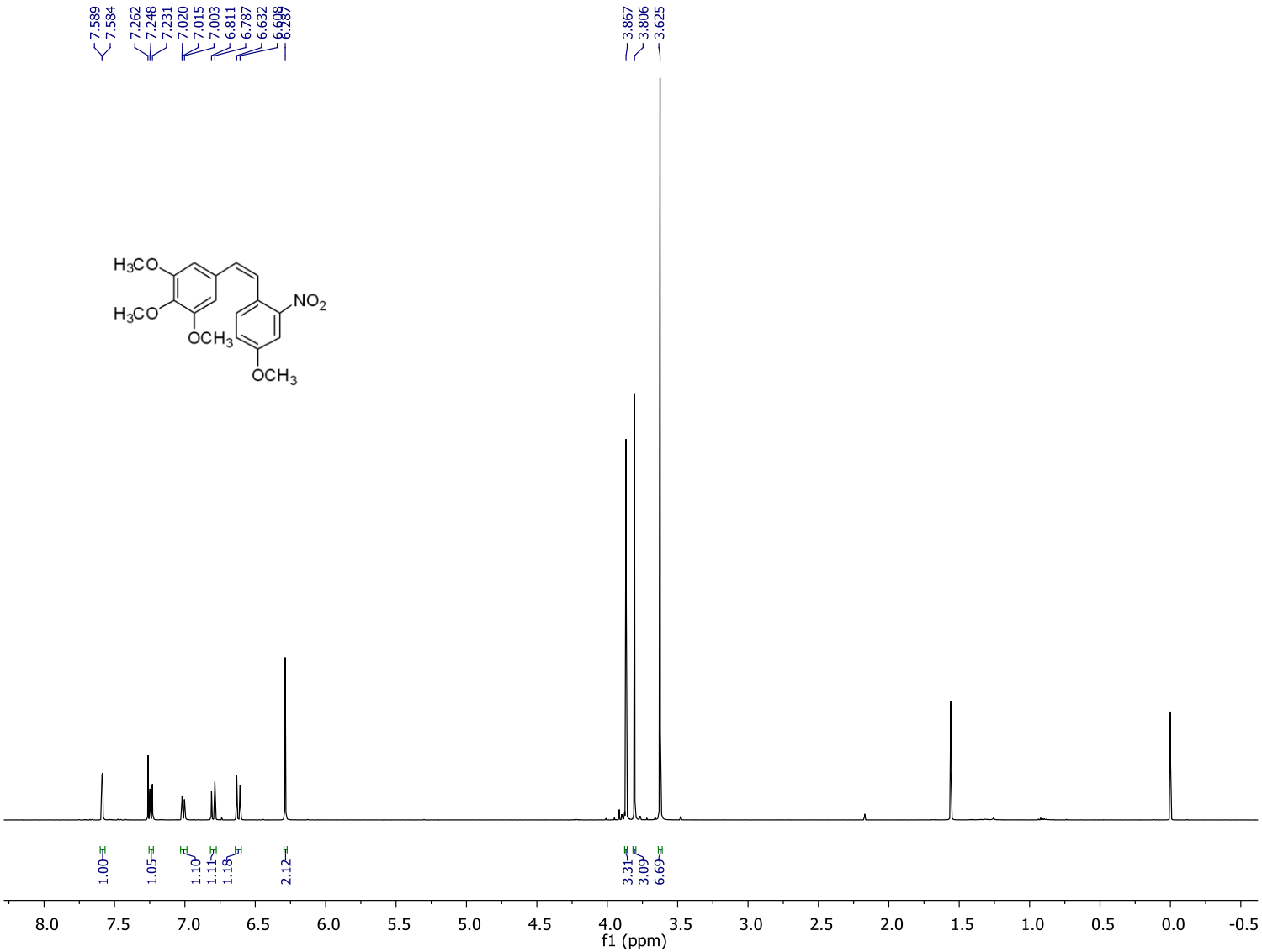
^{1}H NMR ($CDCl_3$, 600 MHz) Compound **4**



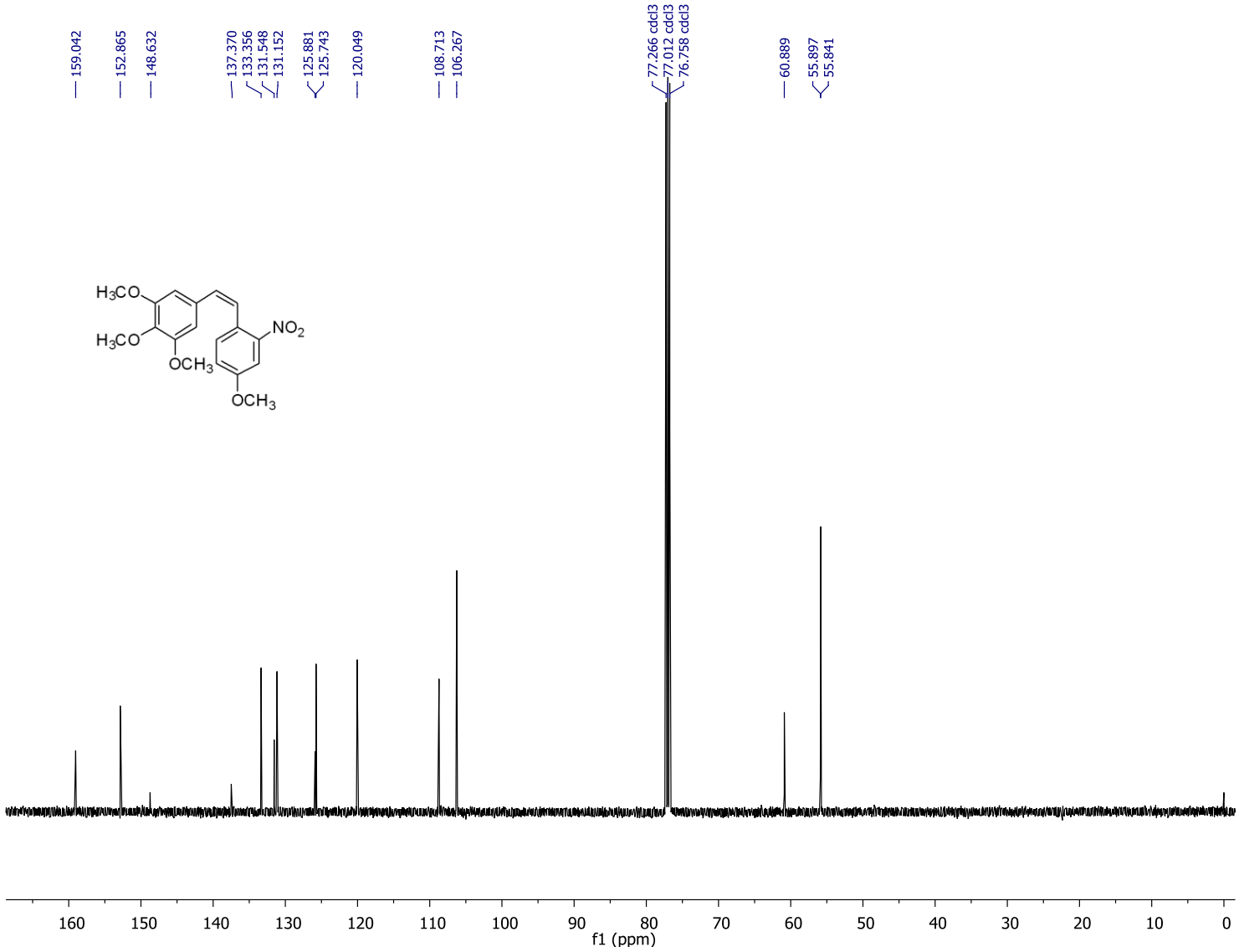
^{31}P NMR ($CDCl_3$, 240 MHz) Compound **4**



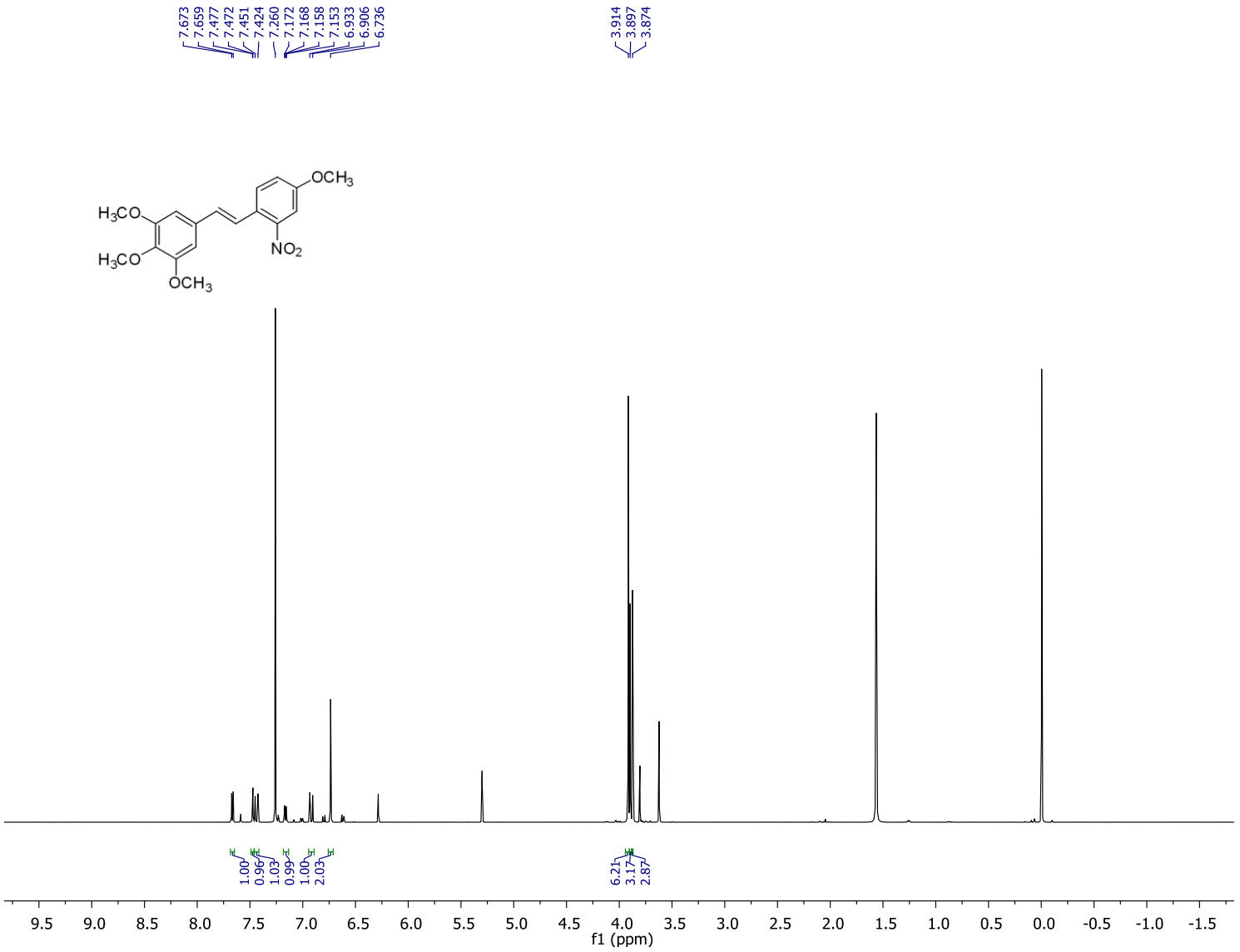
^{1}H NMR ($CDCl_3$, 500 MHz) Compound **5a**



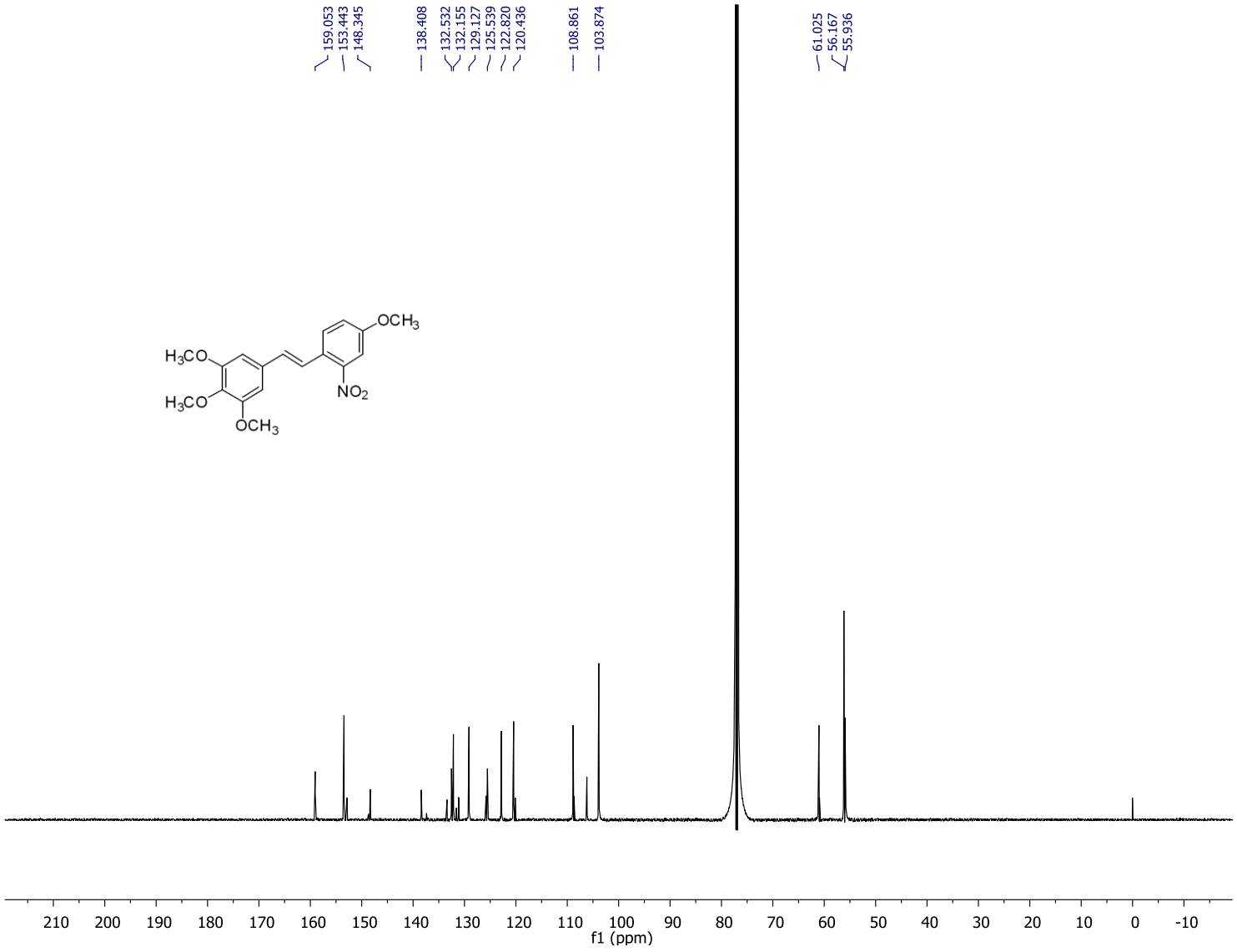
^{13}C NMR ($CDCl_3$, 125 MHz) Compound **5a**



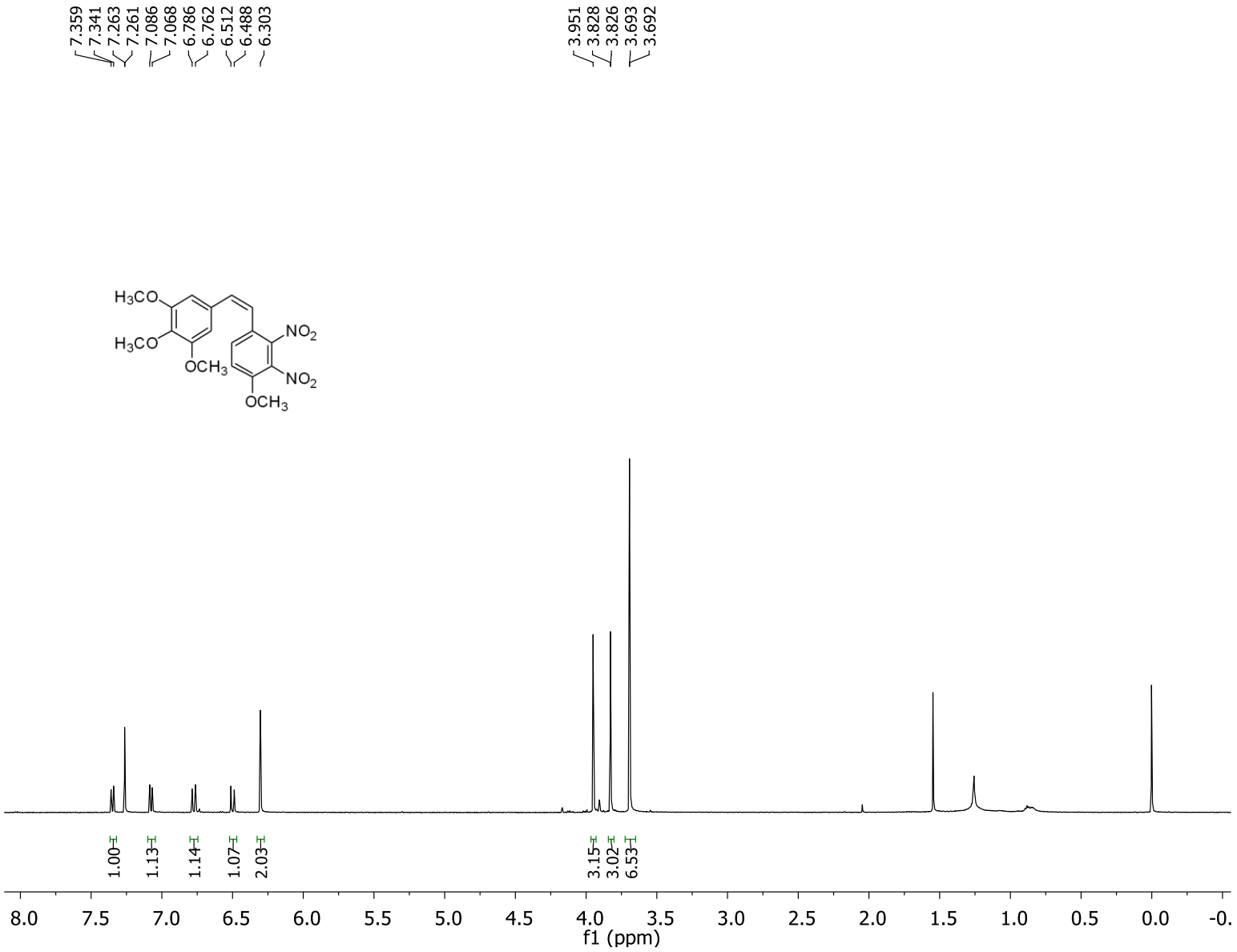
^{1}H NMR ($CDCl_3$, 600 MHz) Compound **5b**



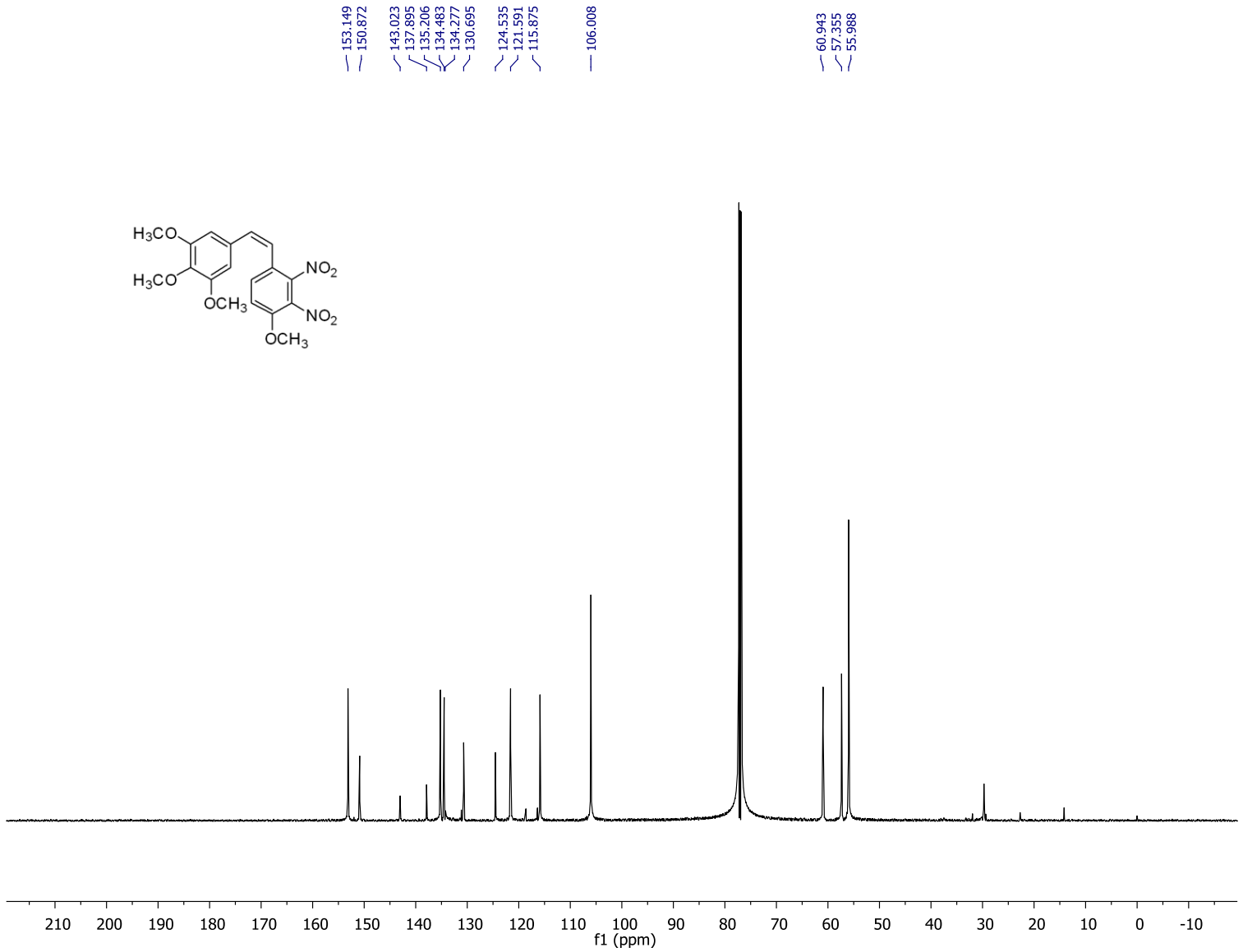
^{13}C NMR ($CDCl_3$, 150 MHz) Compound **5b**



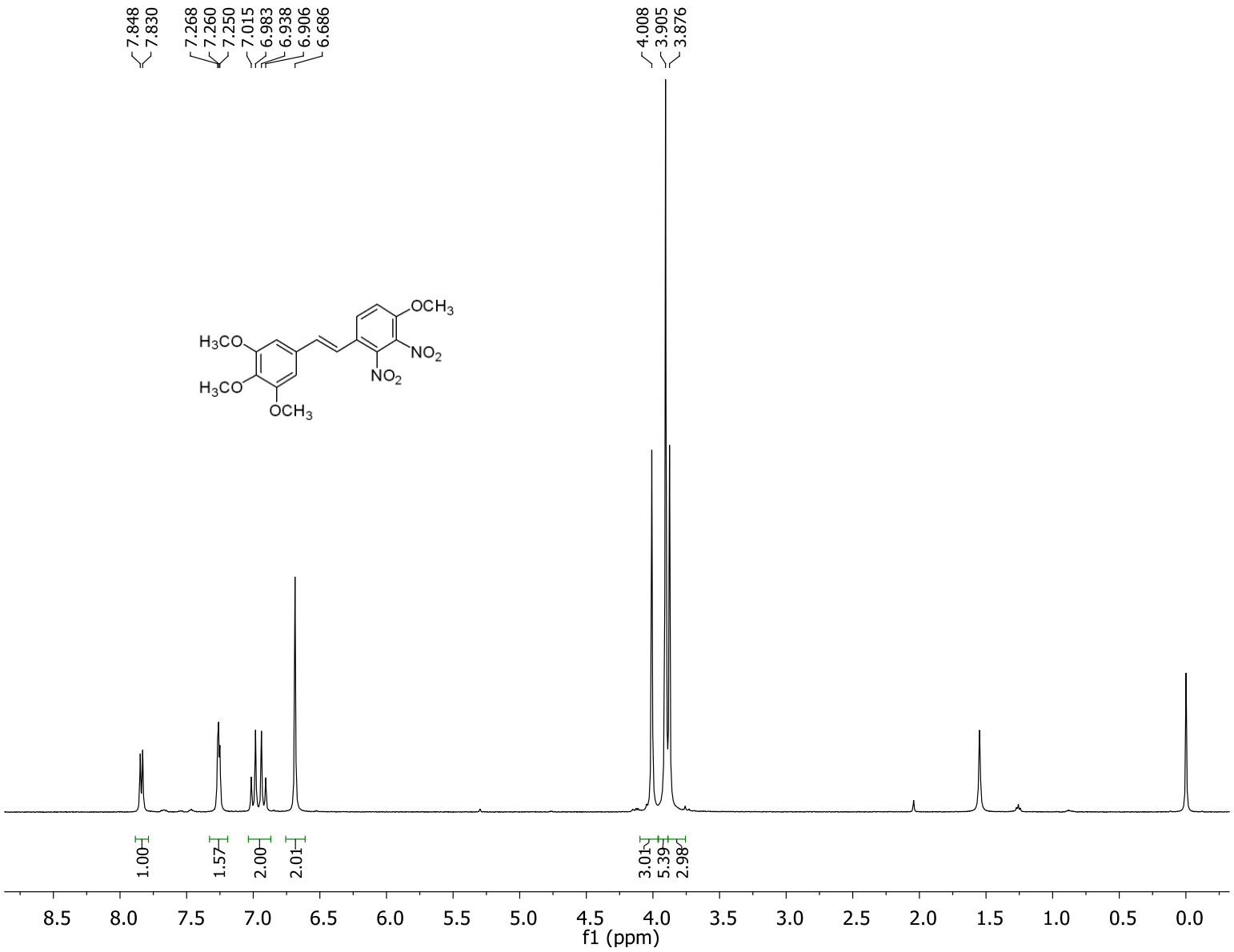
^{1}H NMR ($CDCl_3$, 500 MHz) Compound **6a**



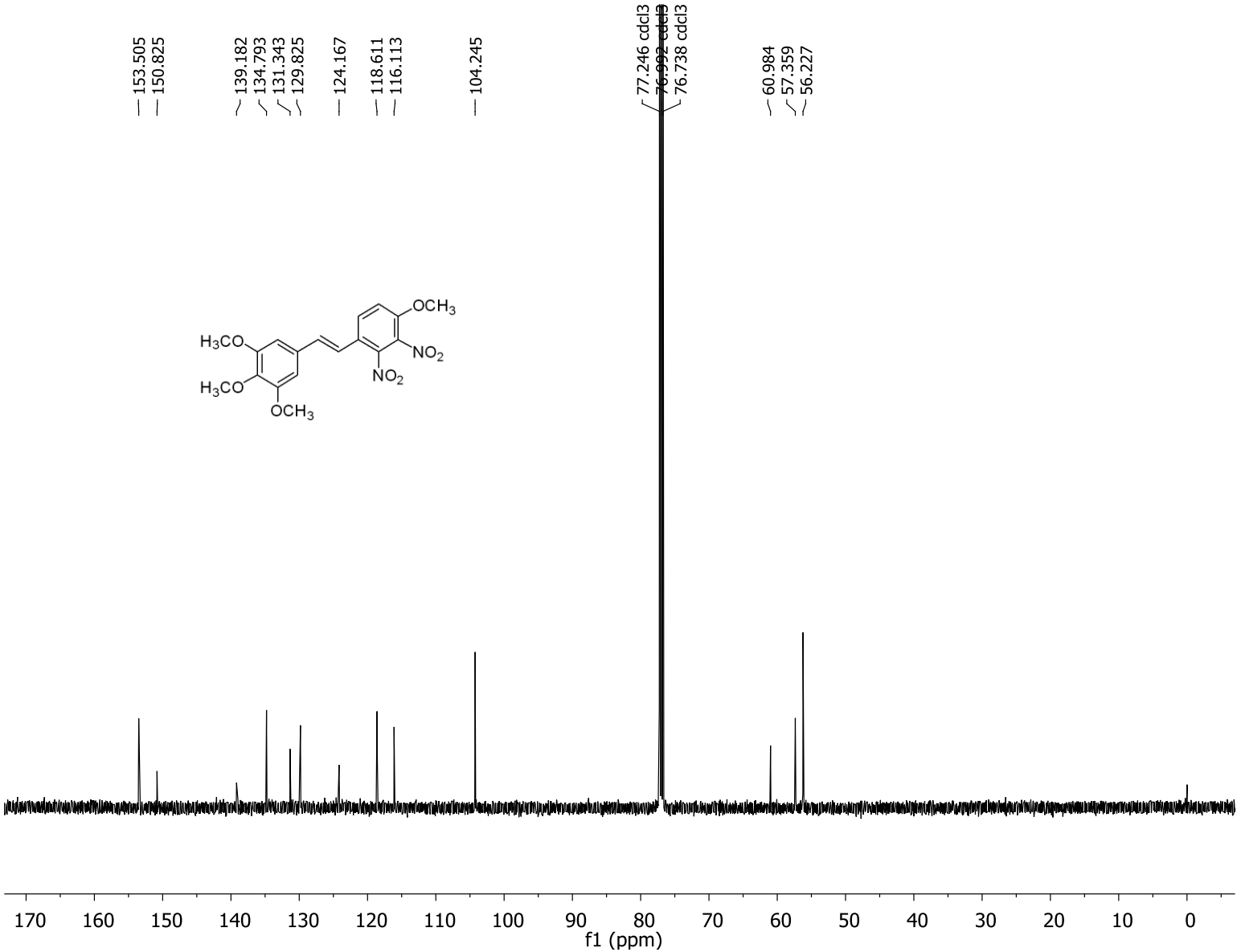
^{13}C NMR ($CDCl_3$, 150 MHz) Compound **6a**



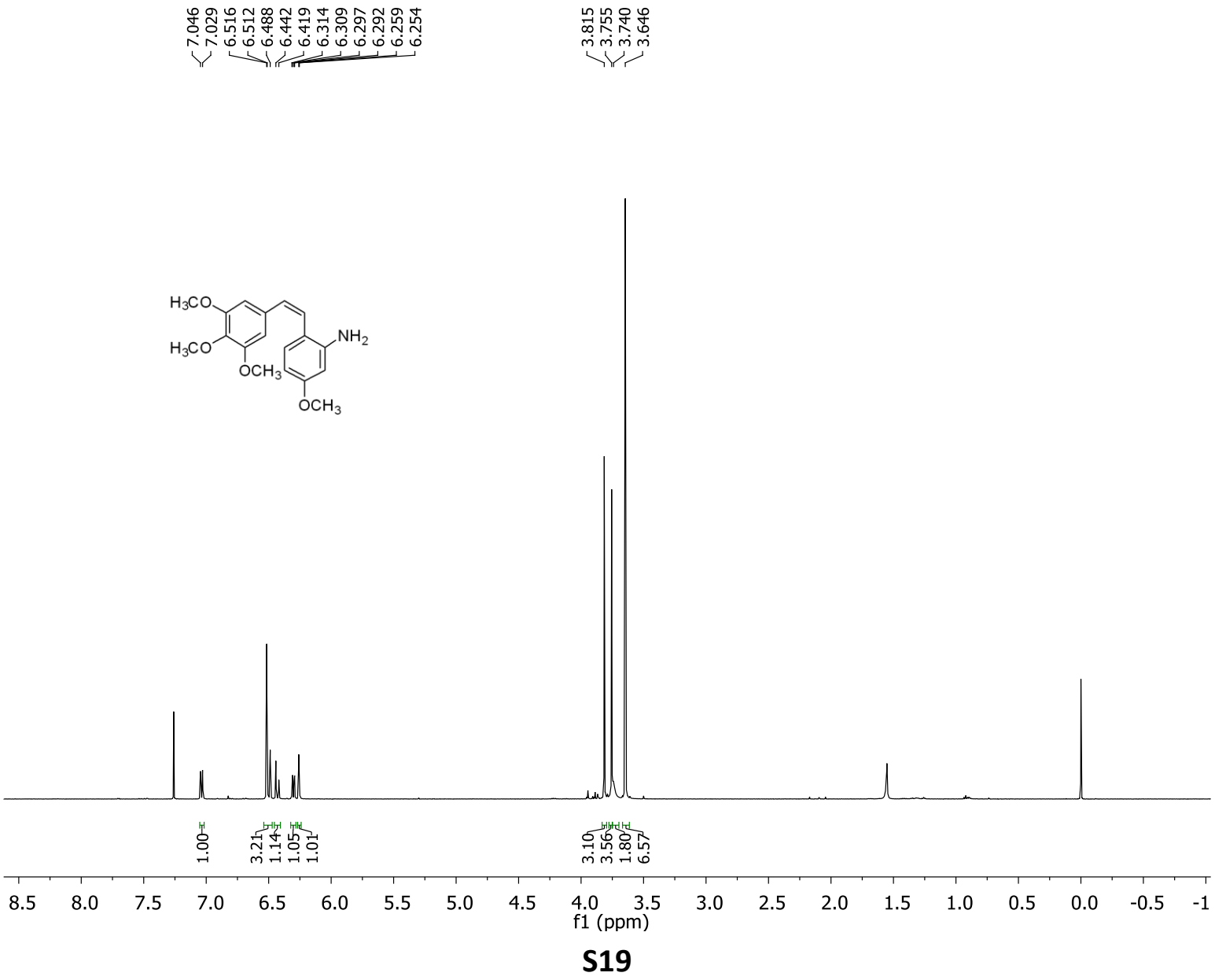
^{1}H NMR ($CDCl_3$, 500 MHz) Compound **6b**



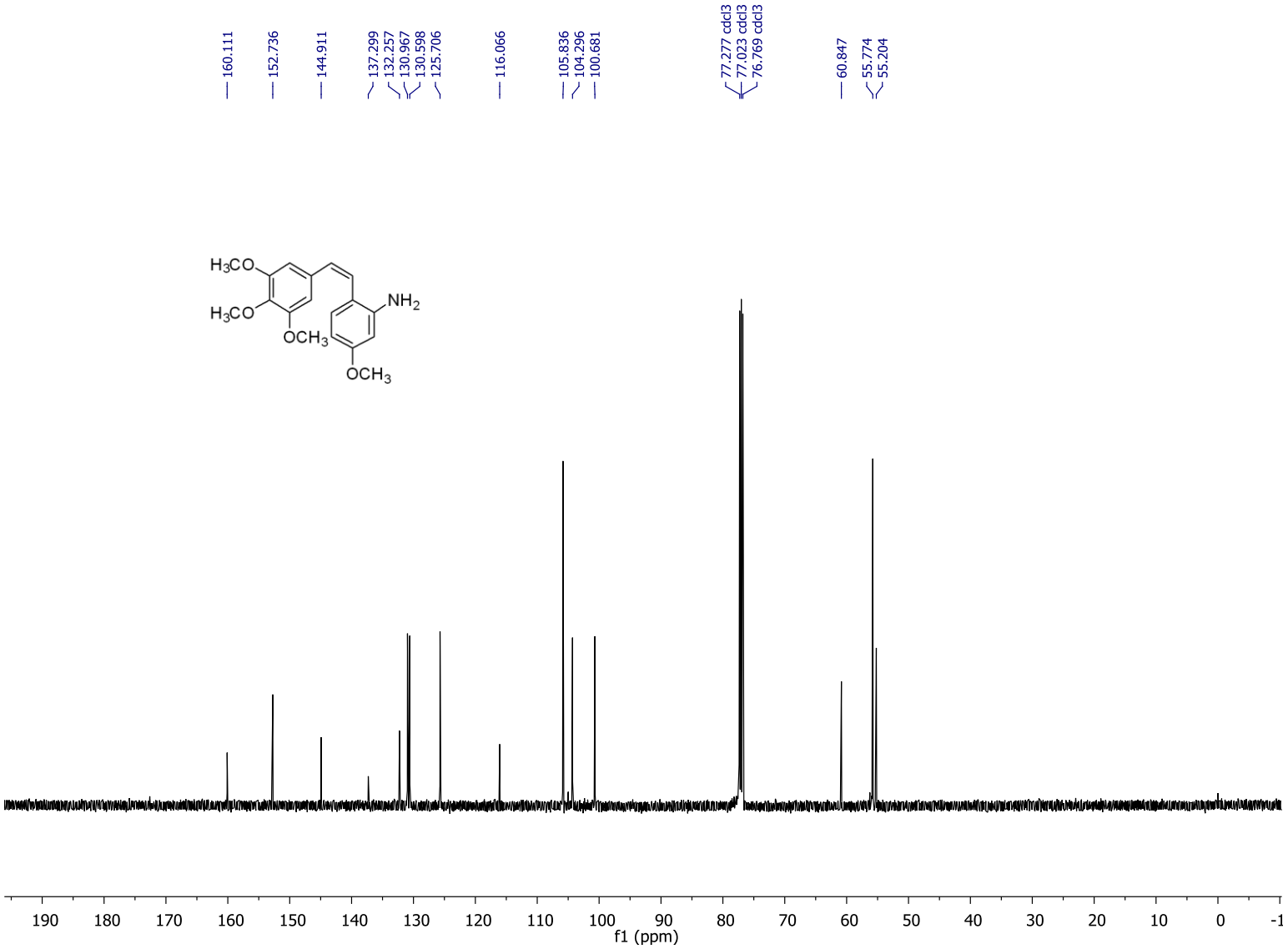
^{13}C NMR ($CDCl_3$, 125 MHz) Compound **6b**



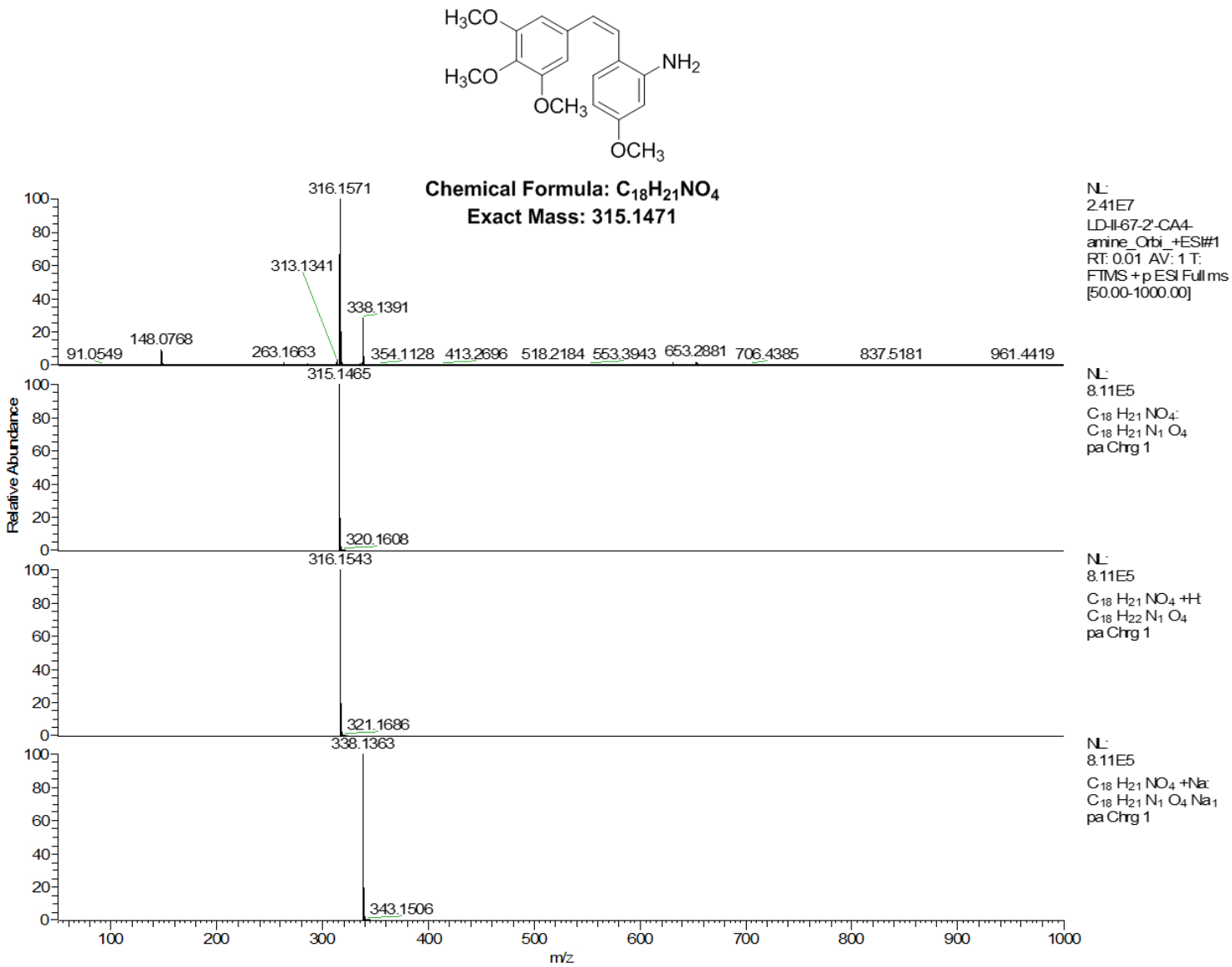
^{1}H NMR ($CDCl_3$, 500 MHz) Compound **7**



^{13}C NMR ($CDCl_3$, 125 MHz) Compound **7**



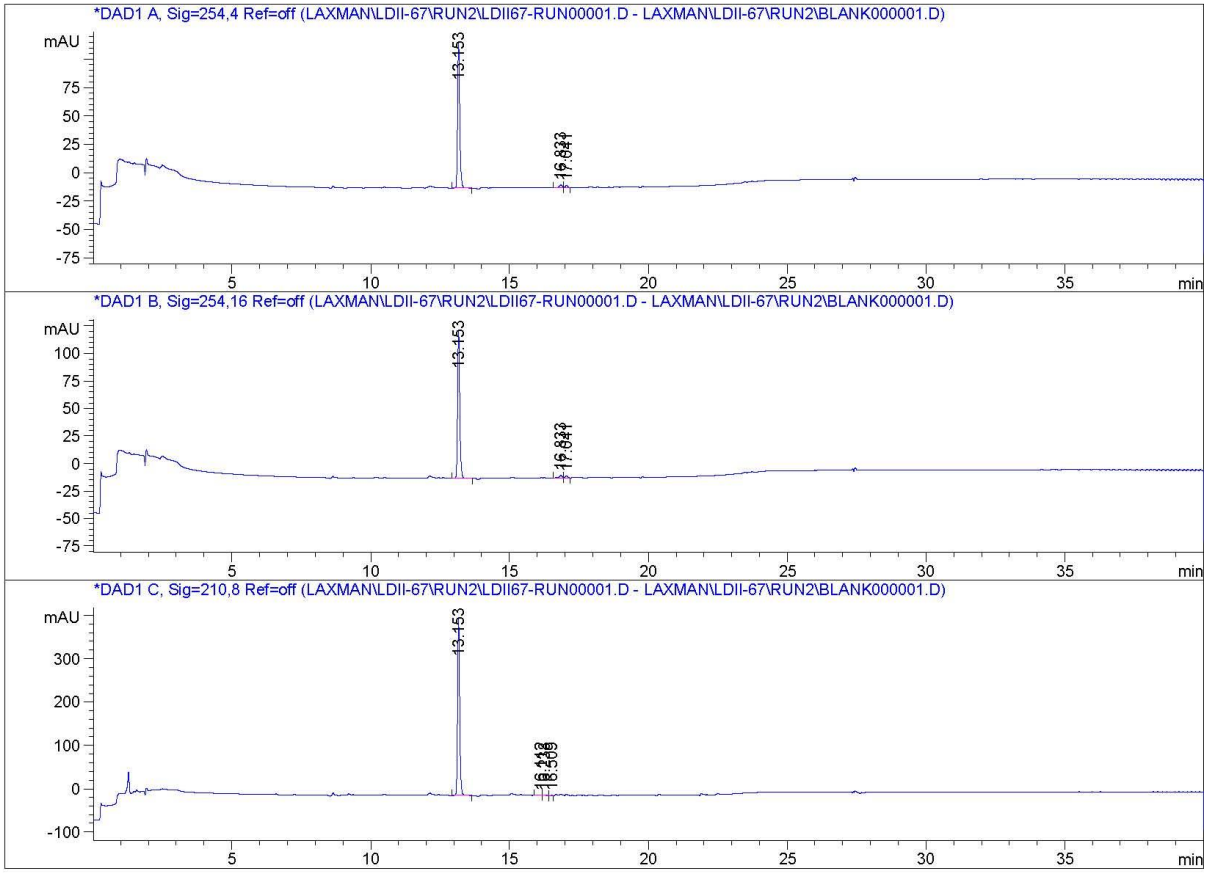
HRMS Compound **7**



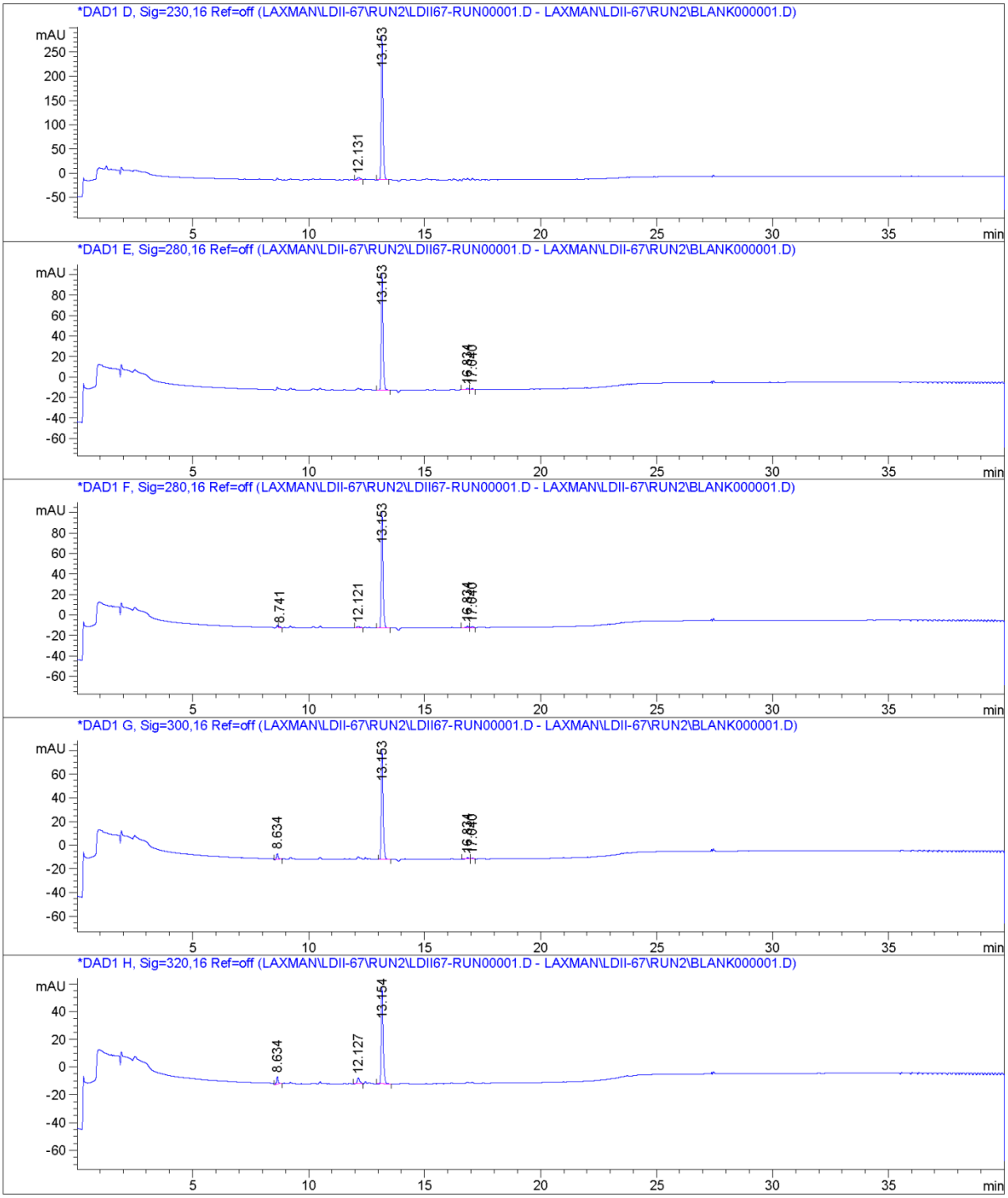
HPLC for Compound **7**

```
Data File C:\CHEM32\1\DATA\LAXMAN\LDII-67\RUN2\LDII67-RUN00001.D
Sample Name: LD-II-67-1-run2

    ======================================================================
    Acq. Operator   : Laxman
    Acq. Instrument : Instrument 1                    Location :   -
    Injection Date  : 7/3/2013 10:53:44 AM
    Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD.M
    Last changed    : 7/3/2013 10:38:50 AM by Laxman
    Analysis Method : C:\CHEM32\1\DATA\LAXMAN\LDII-67\RUN2\LDII67-RUN00001.D\DA.M (MASTERMETHOD.M)
    Last changed    : 7/3/2013 11:43:58 AM by Laxman
    Sample Info     :
                      mastermethod
```



Data File C:\CHEM32\1\DATA\LAXMAN\LDII-67\RUN2\LDII67-RUN00001.D
Sample Name: LD-II-67-1-run2



Data File C:\CHEM32\1\DATA\LAXMAN\LDII-67\RUN2\LDII67-RUN00001.D
Sample Name: LD-II-67-1-run2

==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.153	BB	0.0801	684.21429	128.44377	95.8285
2	16.833	BV	0.1042	18.81558	2.54608	2.6352
3	17.041	VB	0.0833	10.96856	2.02302	1.5362
Totals :				713.99843	133.01288	

Signal 2: DAD1 B, Sig=254,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.153	BB	0.0801	715.88000	134.46823	95.9599
2	16.833	BV	0.1076	19.28228	2.51098	2.5847
3	17.041	VB	0.0835	10.85781	1.99546	1.4554
Totals :				746.02010	138.97467	

Signal 3: DAD1 C, Sig=210,8 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.153	BB	0.0786	2124.96387	408.88397	98.1593
2	16.112	BV	0.1162	16.52923	1.96462	0.7635
3	16.238	VB	0.1149	15.90247	1.95409	0.7346
4	16.509	BB	0.0770	7.41531	1.63260	0.3425
Totals :				2164.81088	414.43528	

Data File C:\CHEM32\1\DATA\LAXMAN\LDII-67\RUN2\LDII67-RUN00001.D
Sample Name: LD-II-67-1-run2

Signal 4: DAD1 D, Sig=230,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.131	VV	0.1468	52.76670	4.93102	3.2789
2	13.153	BB	0.0790	1556.49365	297.68036	96.7211
Totals :				1609.26036	302.61138	

Signal 5: DAD1 E, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.153	BB	0.0796	603.33063	114.21896	97.2505
2	16.834	BV	0.0993	10.60114	1.56055	1.7088
3	17.040	VB	0.0835	6.45647	1.18643	1.0407
Totals :				620.38824	116.96593	

Signal 6: DAD1 F, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.741	VB	0.0797	5.59504	1.05788	0.8730
2	12.121	BB	0.1405	14.87934	1.48978	2.3218
3	13.153	BB	0.0796	603.33063	114.21896	94.1435
4	16.834	BV	0.0993	10.60114	1.56055	1.6542
5	17.040	VB	0.0835	6.45647	1.18643	1.0075
Totals :				640.86262	119.51359	

Signal 7: DAD1 G, Sig=300,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.634	BB	0.0769	27.28100	5.22612	4.8981
2	13.153	BB	0.0824	514.18933	93.18947	92.3180
3	16.834	BV	0.1026	9.83546	1.35601	1.7659

Data File C:\CHEM32\1\DATA\LAXMAN\LDII-67\RUN2\LDII67-RUN00001.D
Sample Name: LD-II-67-1-run2

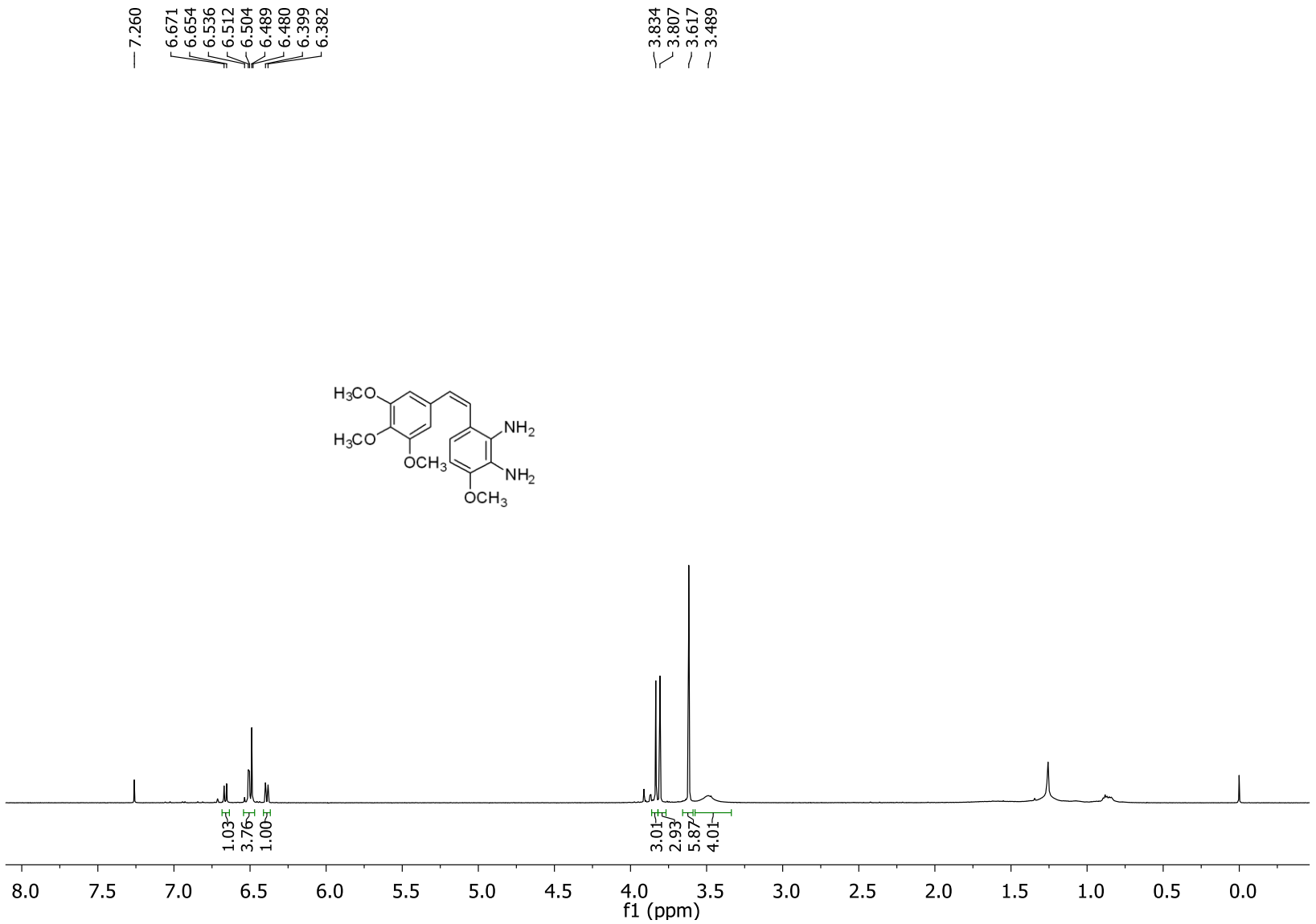
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
4	17.040	VB	0.0835	5.67047	1.04138	1.0181
Totals :				556.97626	100.81297	

Signal 8: DAD1 H, Sig=320,16 Ref=off
Signal has been modified after loading from rawdata file!

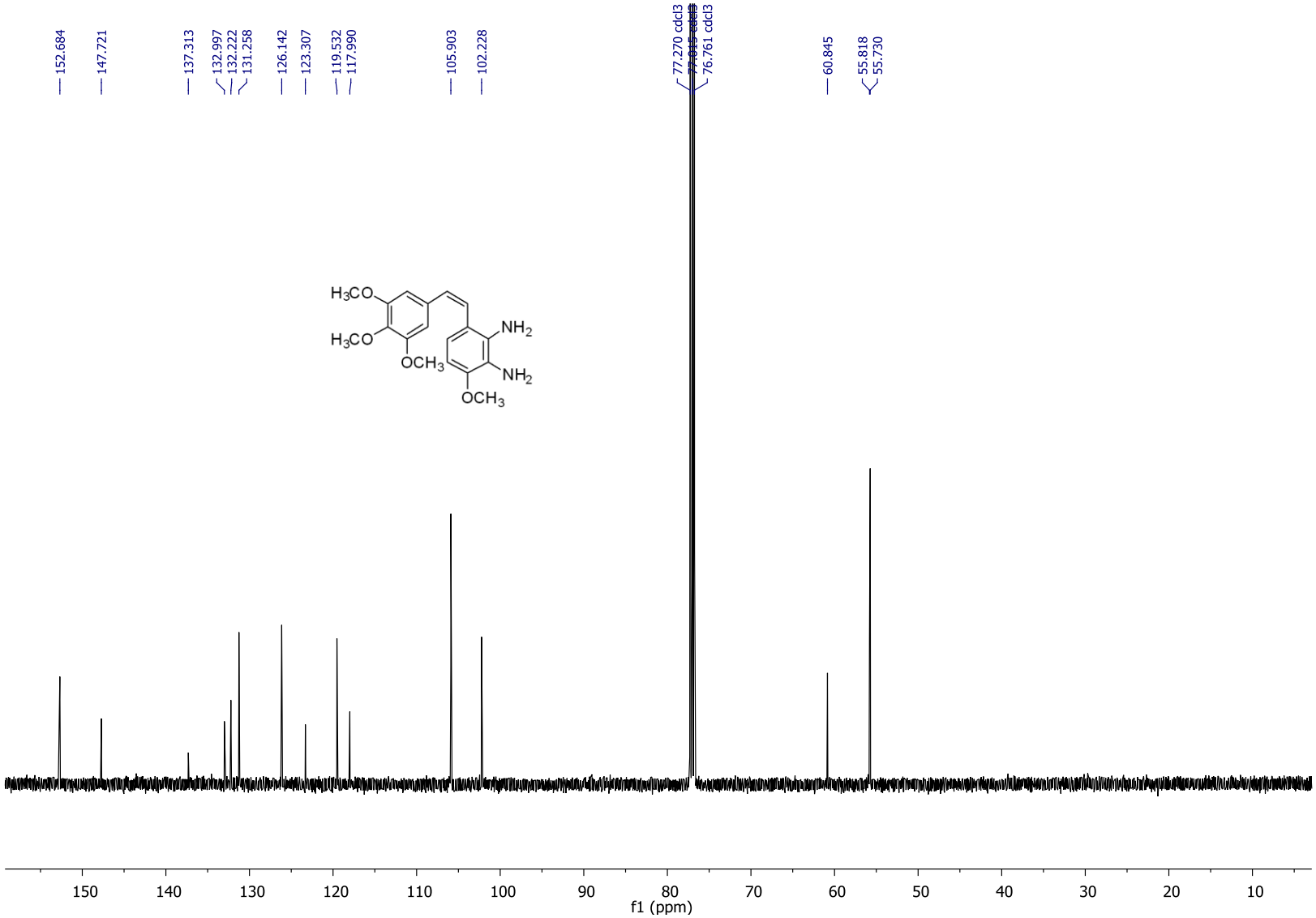
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.634	BB	0.0773	29.33656	5.58494	6.4560
2	12.127	BB	0.1151	33.01547	4.22876	7.2656
3	13.154	BB	0.0836	392.05457	69.73837	86.2784
Totals :				454.40660	79.55207	

*** End of Report ***

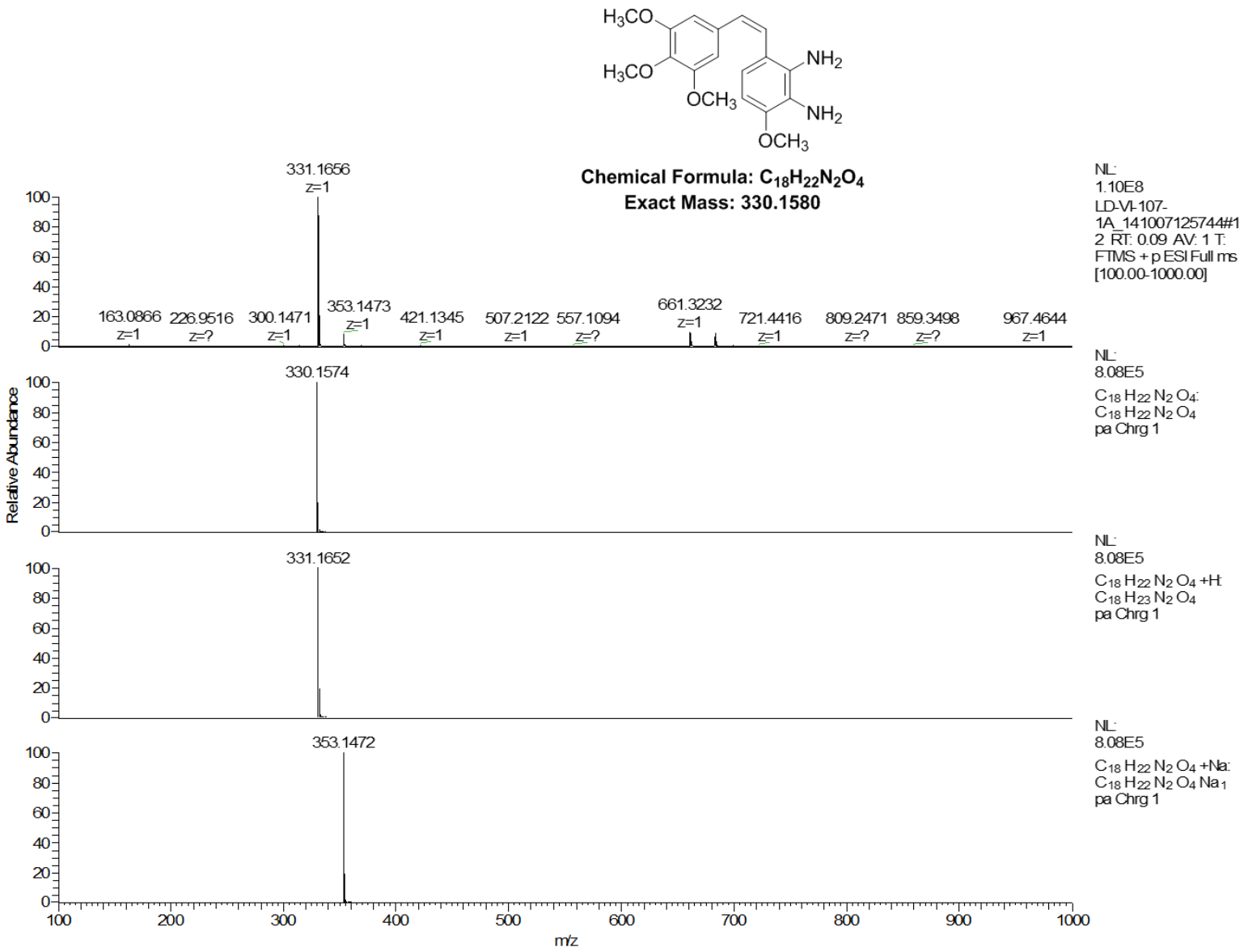
^{1}H NMR ($CDCl_3$, 500 MHz) Compound **8**



^{13}C NMR ($CDCl_3$, 125 MHz) Compound **8**



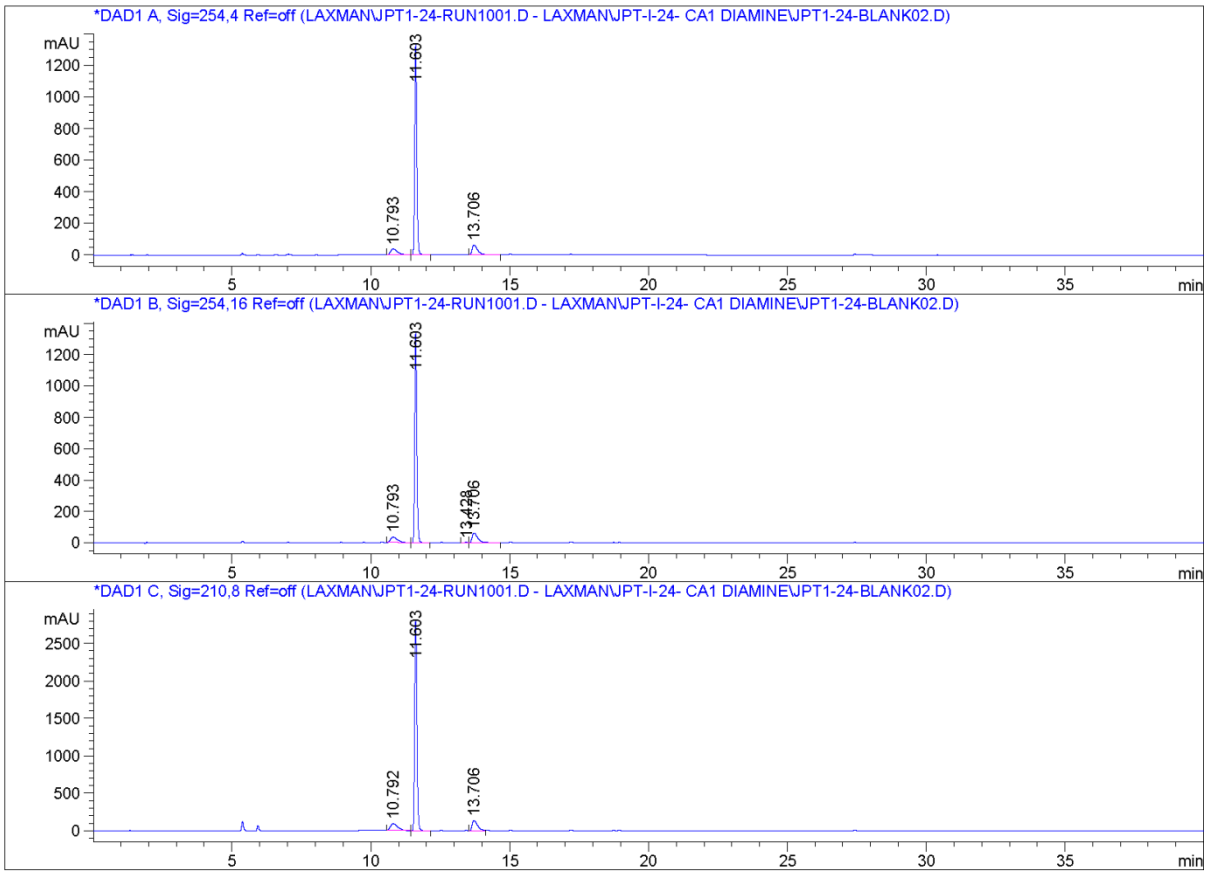
HRMS Compound **8**



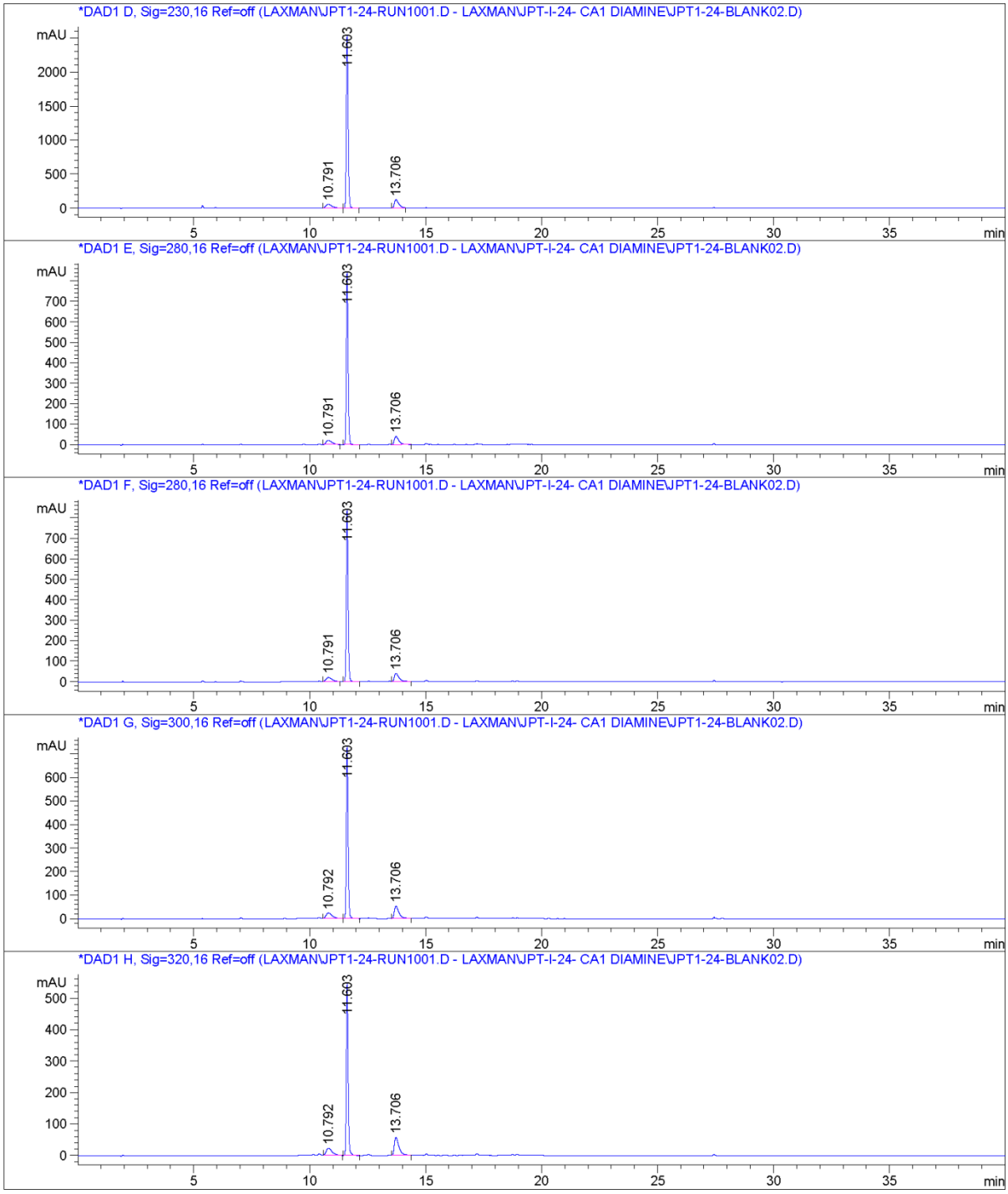
HPLC for Compound **8**

```
Data File C:\CHEM32\1\DATA\LAXMAN\JPT1-24-RUN1001.D
Sample Name: JPT-I-24-2- run1

    ======================================================================
    Acq. Operator   : Laxman
    Acq. Instrument : Instrument 1                    Location :   -
    Injection Date  : 3/8/2013 11:34:37 AM
    Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD.M
    Last changed    : 3/8/2013 10:58:56 AM by Laxman
    Analysis Method : C:\CHEM32\1\DATA\LAXMAN\JPT1-24-RUN1001.D\DA.M (MASTERMETHOD.M)
    Last changed    : 3/8/2013 12:22:23 PM by Laxman
                      (modified after loading)
    Sample Info     : CA1 Diamine
                      Run 1 - 10% ACN/Water
```



Data File C:\CHEM32\1\DATA\LAXMAN\JPT1-24-RUN1001.D
Sample Name: JPT-I-24-2- run1



Data File C:\CHEM32\1\DATA\LAXMAN\JPT1-24-RUN1001.D
Sample Name: JPT-I-24-2- run1

==========
Area Percent Report
==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.793	VB	0.2658	636.82605	36.46028	7.4422
2	11.603	BB	0.0813	7032.83594	1338.65552	82.1882
3	13.706	VB	0.2133	887.33014	61.89165	10.3697
Totals :				8556.99213	1437.00745	

Signal 2: DAD1 B, Sig=254,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.793	VB	0.2683	634.71381	36.23970	7.3312
2	11.603	BB	0.0813	7088.87402	1349.12854	81.8791
3	13.428	BV	0.1080	28.47644	3.95348	0.3289
4	13.706	VB	0.2133	905.67200	63.14117	10.4608
Totals :				8657.73626	1452.46289	

Signal 3: DAD1 C, Sig=210,8 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.792	BB	0.2721	1686.11475	93.64143	8.4590
2	11.603	BB	0.0918	1.63806e4	2819.08179	82.1797
3	13.706	VV	0.2051	1865.96326	136.85098	9.3613
Totals :				1.99327e4	3049.57420	

Data File C:\CHEM32\1\DATA\LAXMAN\JPT1-24-RUN1001.D
Sample Name: JPT-I-24-2- run1

Signal 4: DAD1 D, Sig=230,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.791	VB	0.2794	1063.52295	56.54724	6.3234
2	11.603	BB	0.0863	1.40428e4	2549.48462	83.4944
3	13.706	VV	0.2068	1712.51416	124.25601	10.1821
Totals :				1.68188e4	2730.28787	

Signal 5: DAD1 E, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.791	VB	0.2803	362.46127	19.91856	6.7384
2	11.603	BB	0.0812	4438.77637	845.72125	82.5199
3	13.706	VB	0.2105	577.80243	40.99035	10.7417
Totals :				5379.04007	906.63016	

Signal 6: DAD1 F, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.791	VB	0.2803	362.46127	19.91856	6.7384
2	11.603	BB	0.0812	4438.77637	845.72125	82.5199
3	13.706	VB	0.2105	577.80243	40.99035	10.7417
Totals :				5379.04007	906.63016	

Signal 7: DAD1 G, Sig=300,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.792	VB	0.2793	422.46881	23.11047	8.3678
2	11.603	BB	0.0815	3879.14600	736.06525	76.8335
3	13.706	VB	0.2095	747.15320	53.30036	14.7987

Data File C:\CHEM32\1\DATA\LAXMAN\JPT1-24-RUN1001.D
Sample Name: JPT-I-24-2- run1

Totals : 5048.76801 812.47607

Signal 8: DAD1 H, Sig=320,16 Ref=off
Signal has been modified after loading from rawdata file!

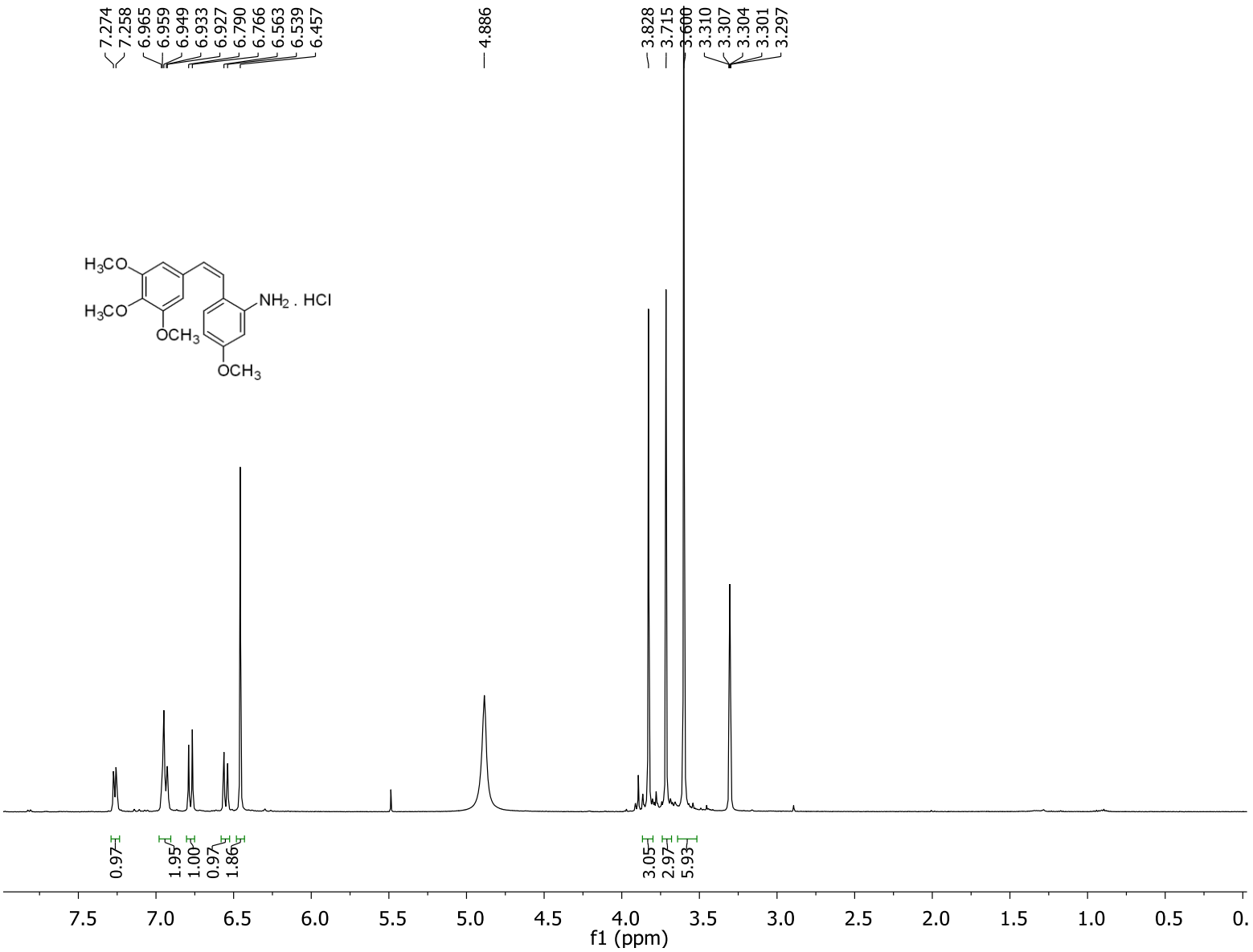
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.792	VB	0.2743	400.08136	22.62302	9.6661
2	11.603	BB	0.0822	2933.52075	549.82037	70.8749
3	13.706	VB	0.2086	805.40729	57.80400	19.4589

Totals : 4139.00940 630.24739

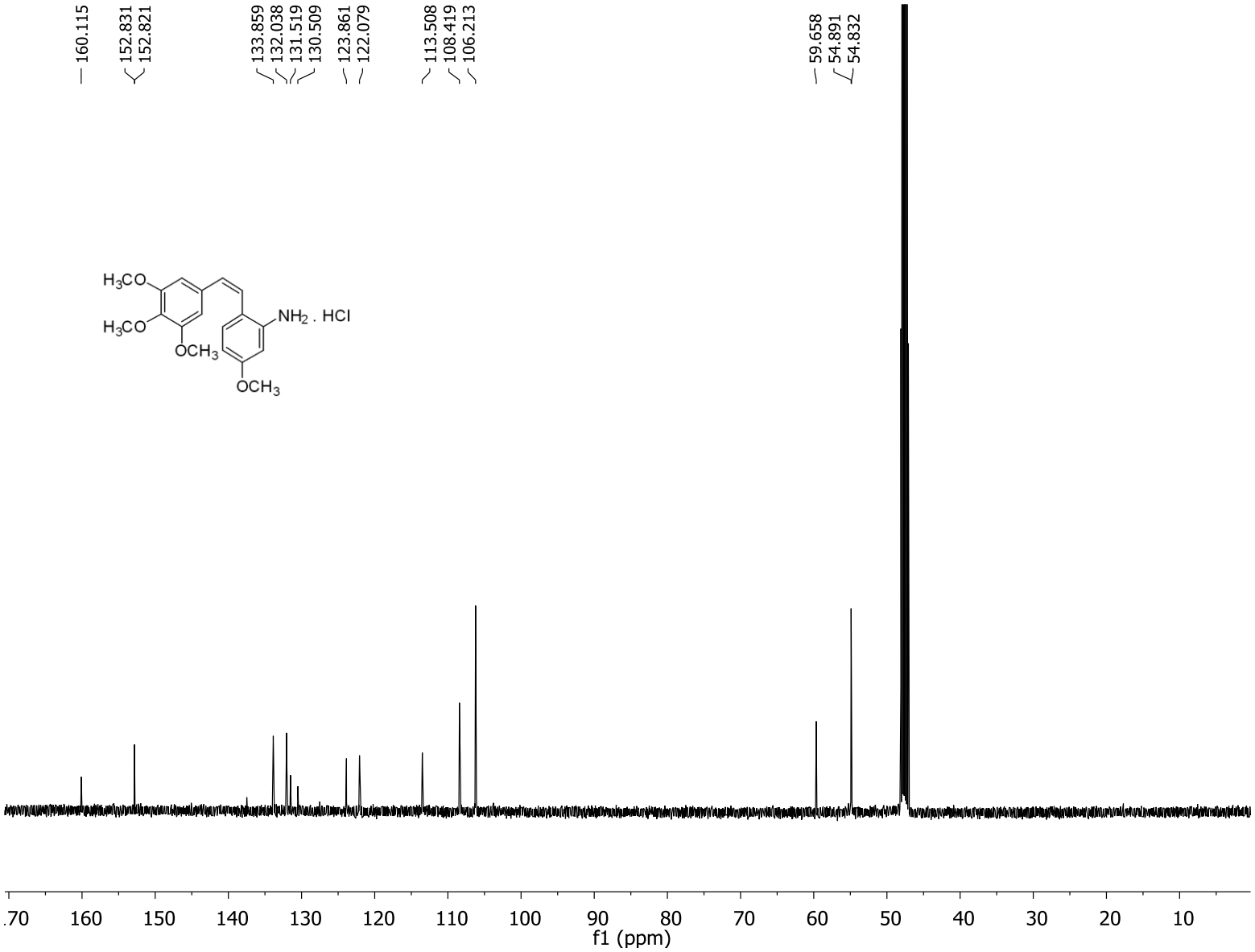
==========

*** End of Report ***

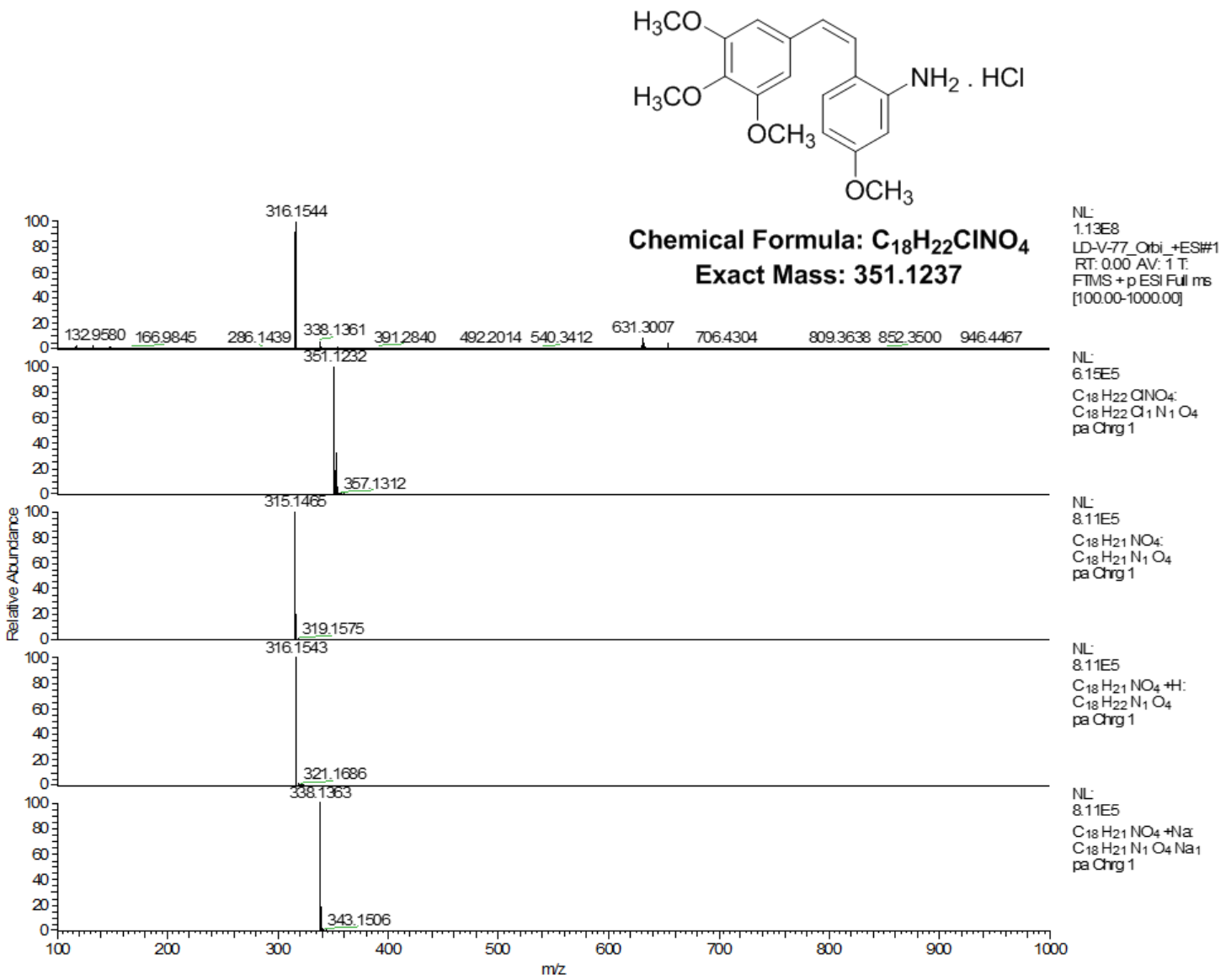
^{1}H NMR (CD_3OD, 500 MHz) Compound **9**



^{13}C NMR (CD_3OD, 125 MHz) Compound **9**



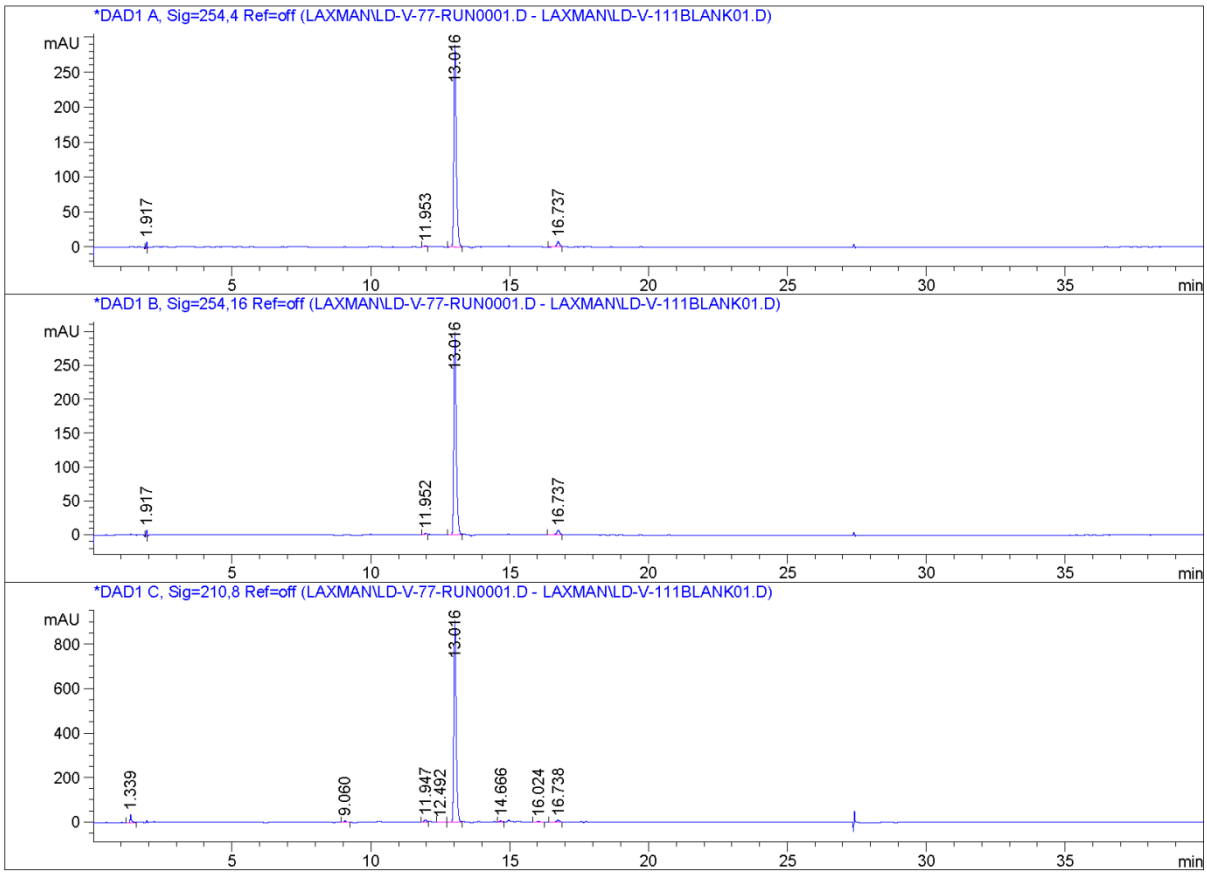
HRMS Compound **9**



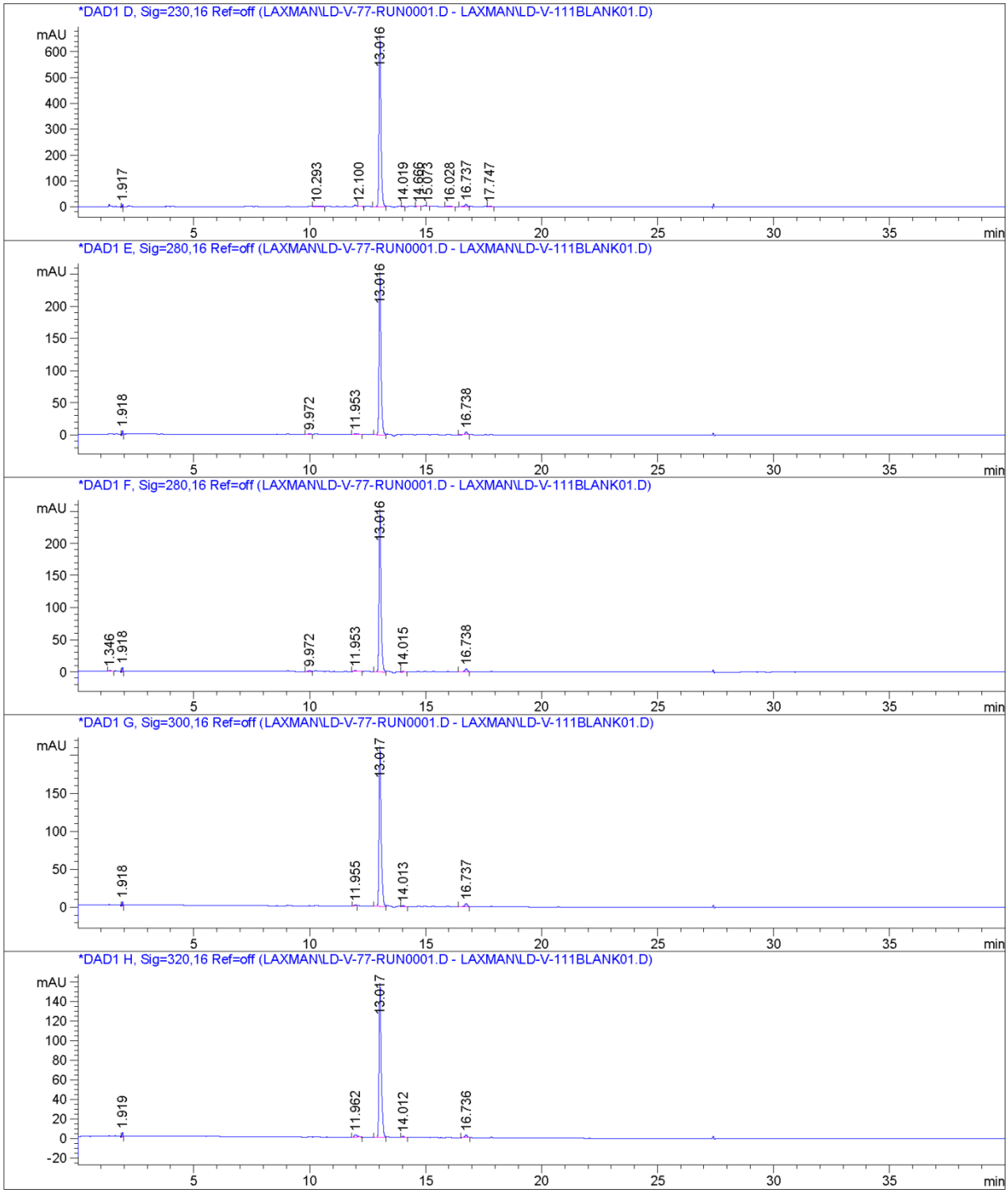
HPLC for Compound **9**

```
Data File C:\CHEM32\1\DATA\LAXMAN\LD-V-77-RUN0001.D
Sample Name: LD-V-77-1A-run1

    ======================================================================
    Acq. Operator   : Laxman
    Acq. Instrument : Instrument 1                    Location :   -
    Injection Date  : 7/23/2014 10:58:30 AM
    Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD.M
    Last changed    : 7/23/2014 10:57:14 AM by Laxman
    Analysis Method : C:\CHEM32\1\DATA\LAXMAN\LD-V-77-RUN0001.D\DA.M (MASTERMETHOD.M)
    Last changed    : 7/23/2014 11:48:44 AM by Laxman
    Sample Info     : Method- Mastermethod
```



Data File C:\CHEM32\1\DATA\LAXMAN\LD-V-77-RUN0001.D
Sample Name: LD-V-77-1A-run1



Data File C:\CHEM32\1\DATA\LAXMAN\LD-V-77-RUN0001.D
Sample Name: LD-V-77-1A-run1

==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.917	BB	0.0398	21.88001	9.19392	1.2022
2	11.953	BB	0.0959	9.57182	1.59724	0.5259
3	13.016	BV	0.0896	1734.00476	290.73328	95.2719
4	16.737	BV	0.1027	54.60278	7.70488	3.0001
Totals :				1820.05937	309.22931	

Signal 2: DAD1 B, Sig=254,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.917	BB	0.0398	21.54620	9.05059	1.1550
2	11.952	BB	0.0936	10.00687	1.67807	0.5364
3	13.016	BV	0.0894	1780.96240	299.31604	95.4728
4	16.737	BV	0.1033	52.89812	7.40820	2.8357
Totals :				1865.41360	317.45291	

Signal 3: DAD1 C, Sig=210,8 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.339	BV	0.0678	178.51530	37.33842	3.1609
2	9.060	VB	0.0861	49.31191	8.45259	0.8731
3	11.947	BV	0.1015	70.69033	10.36870	1.2517
4	12.492	VB	0.1447	22.89972	2.17788	0.4055
5	13.016	BV	0.0865	5184.50049	909.86755	91.8002
6	14.666	BB	0.0940	28.75089	4.79432	0.5091
7	16.024	VB	0.1287	33.65904	3.61362	0.5960

Data File C:\CHEM32\1\DATA\LAXMAN\LD-V-77-RUN0001.D
Sample Name: LD-V-77-1A-run1

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
8	16.738	BV	0.1192	79.26709	9.51872	1.4036
Totals :				5647.59478	986.13178	

Signal 4: DAD1 D, Sig=230,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.917	BV	0.0412	27.87641	11.13194	0.6840
2	10.293	VB	0.2294	37.74120	2.37437	0.9261
3	12.100	VB	0.0996	12.79910	1.92545	0.3141
4	13.016	BV	0.0878	3866.90088	665.83099	94.8834
5	14.019	VB	0.0972	7.92666	1.22969	0.1945
6	14.666	BB	0.0922	13.23263	2.19939	0.3247
7	15.073	VB	0.0824	7.13054	1.37812	0.1750
8	16.028	BB	0.1512	16.67805	1.46031	0.4092
9	16.737	BV	0.1155	71.10069	8.87124	1.7446
10	17.747	BB	0.1336	14.03684	1.44144	0.3444
Totals :				4075.42300	697.84294	

Signal 5: DAD1 E, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.918	BB	0.0398	16.67188	6.99573	1.0558
2	9.972	BV	0.1246	10.85515	1.31168	0.6874
3	11.953	BB	0.1431	16.43839	1.61048	1.0410
4	13.016	BV	0.0890	1503.59937	254.13380	95.2219
5	16.738	BB	0.1006	31.48283	4.67115	1.9938
Totals :				1579.04762	268.72284	

Signal 6: DAD1 F, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Data File C:\CHEM32\1\DATA\LAXMAN\LD-V-77-RUN0001.D
Sample Name: LD-V-77-1A-run1

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.346	BV	0.1634	13.47963	1.03886	0.8410
2	1.918	BB	0.0398	16.67188	6.99573	1.0401
3	9.972	BV	0.1246	10.85515	1.31168	0.6772
4	11.953	BB	0.1431	16.43839	1.61048	1.0256
5	13.016	BV	0.0890	1503.59937	254.13380	93.8061
6	14.015	VB	0.1280	10.35211	1.13919	0.6458
7	16.738	BB	0.1006	31.48283	4.67115	1.9641
Totals :				1602.87935	270.90089	

Signal 7: DAD1 G, Sig=300,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.918	BB	0.0399	13.10246	5.47127	0.9396
2	11.955	BB	0.0952	8.87040	1.49495	0.6361
3	13.017	BV	0.0933	1331.21448	211.96465	95.4686
4	14.013	VB	0.1117	12.31849	1.60284	0.8834
5	16.737	BB	0.1013	28.89387	4.14858	2.0721
Totals :				1394.39970	224.68228	

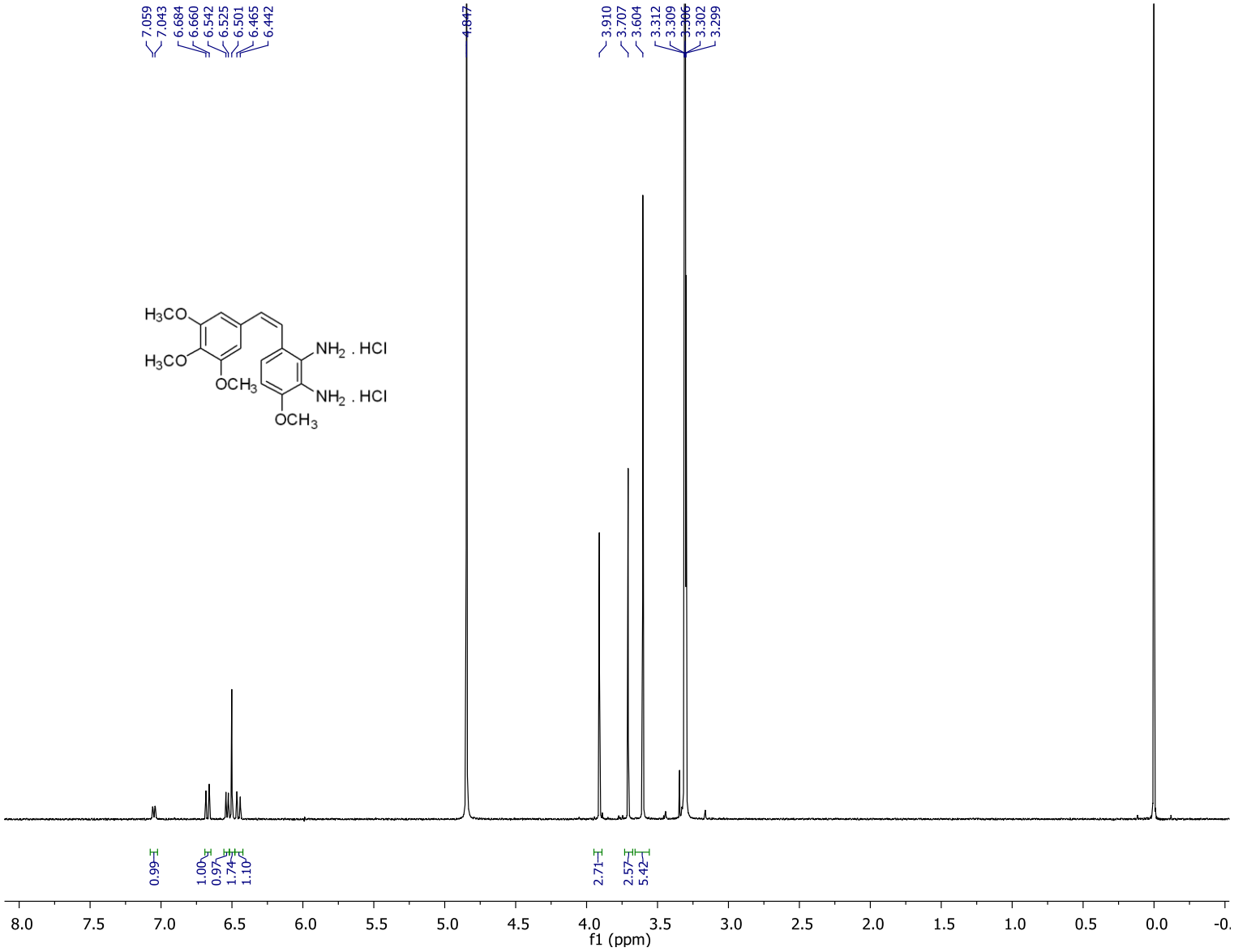
Signal 8: DAD1 H, Sig=320,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.919	BB	0.0399	11.75484	4.92294	1.0894
2	11.962	BB	0.1461	25.13461	2.52625	2.3294
3	13.017	BV	0.0946	1012.13507	158.33987	93.8010
4	14.012	BB	0.0878	7.24201	1.28391	0.6712
5	16.736	BB	0.1061	22.75686	3.08307	2.1090
Totals :				1079.02340	170.15604	

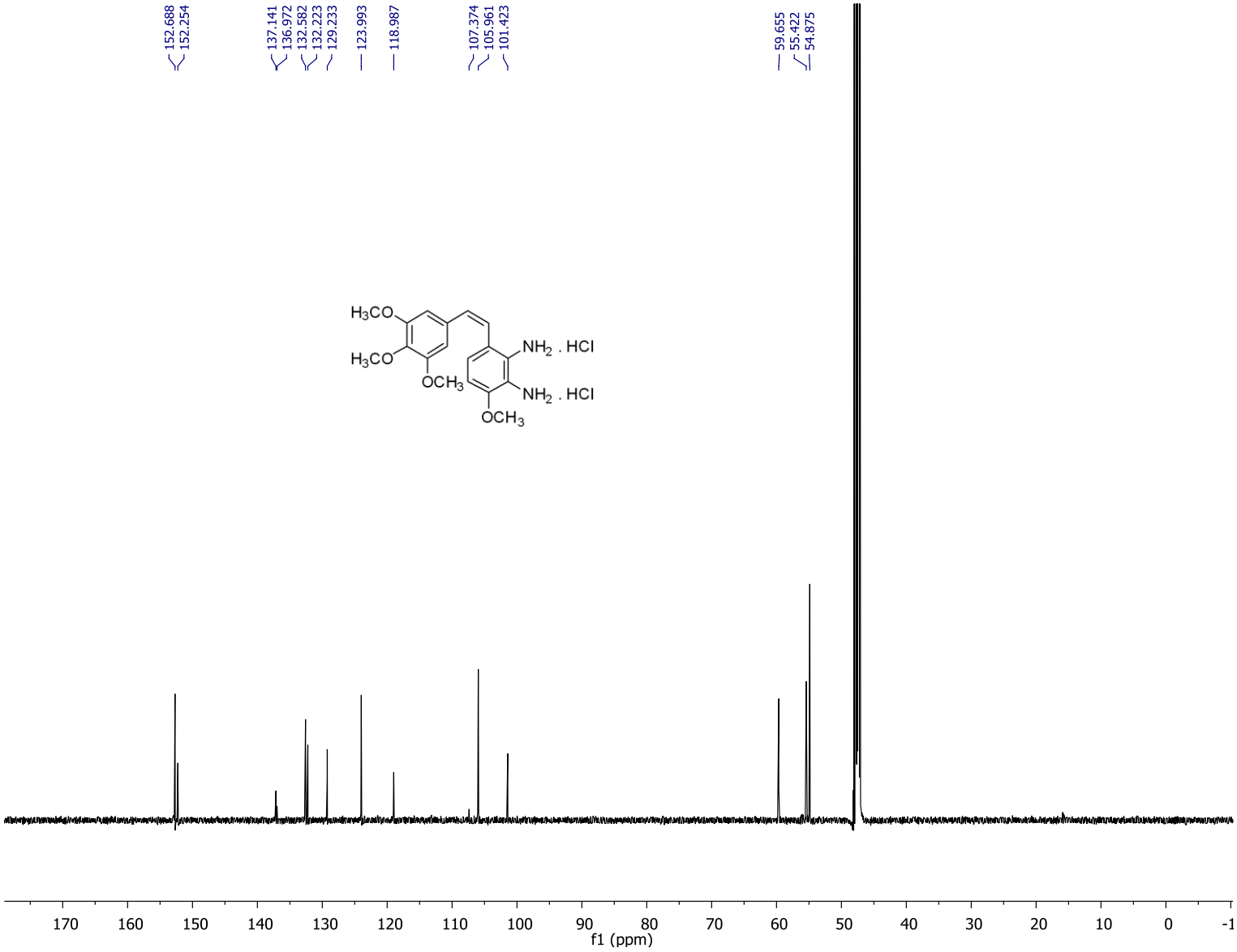
==========

*** End of Report ***

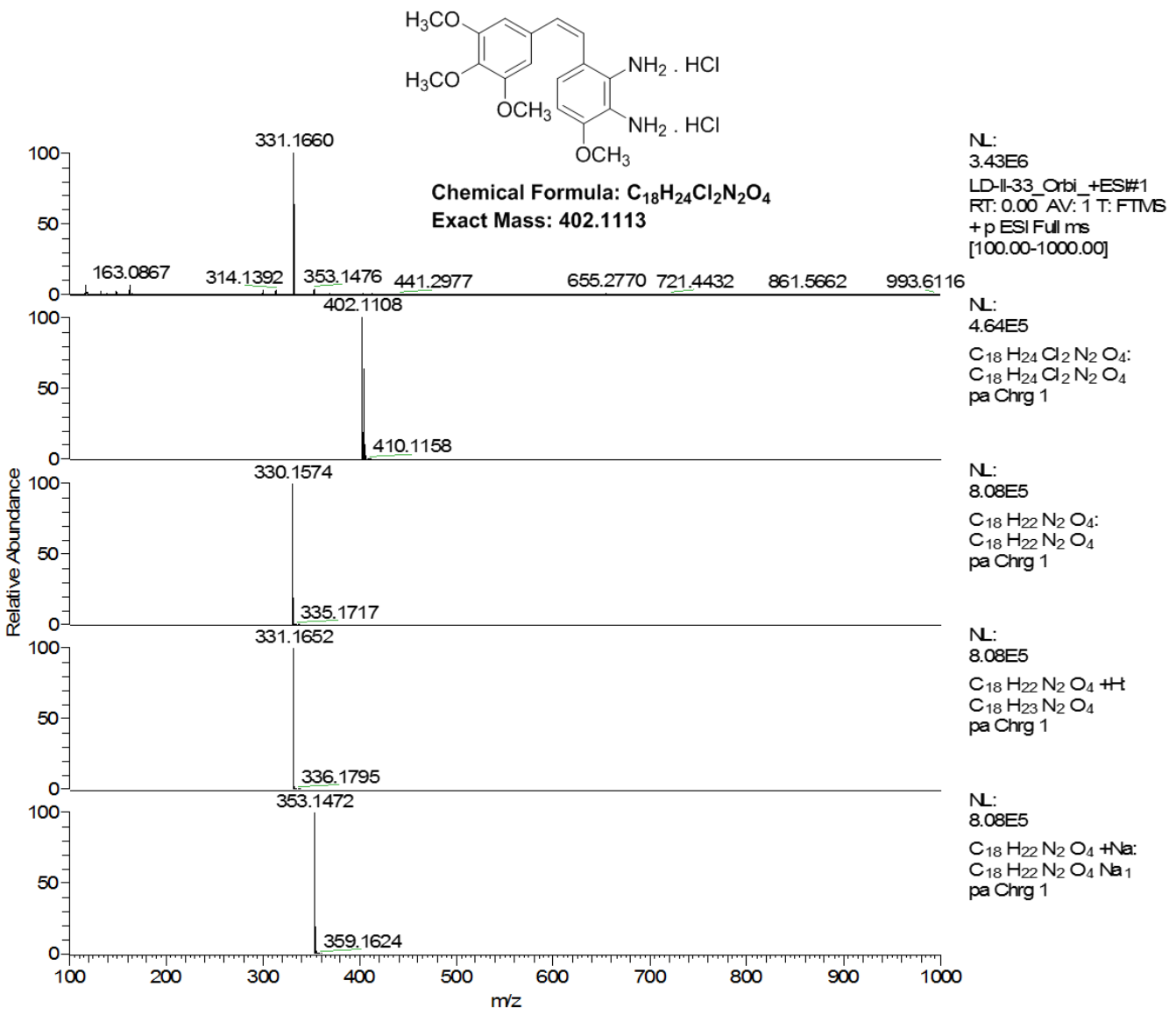
^{1}H NMR (CD_3OD, 600 MHz) Compound **10**



^{13}C NMR (CD_3OD, 150 MHz) Compound **10**



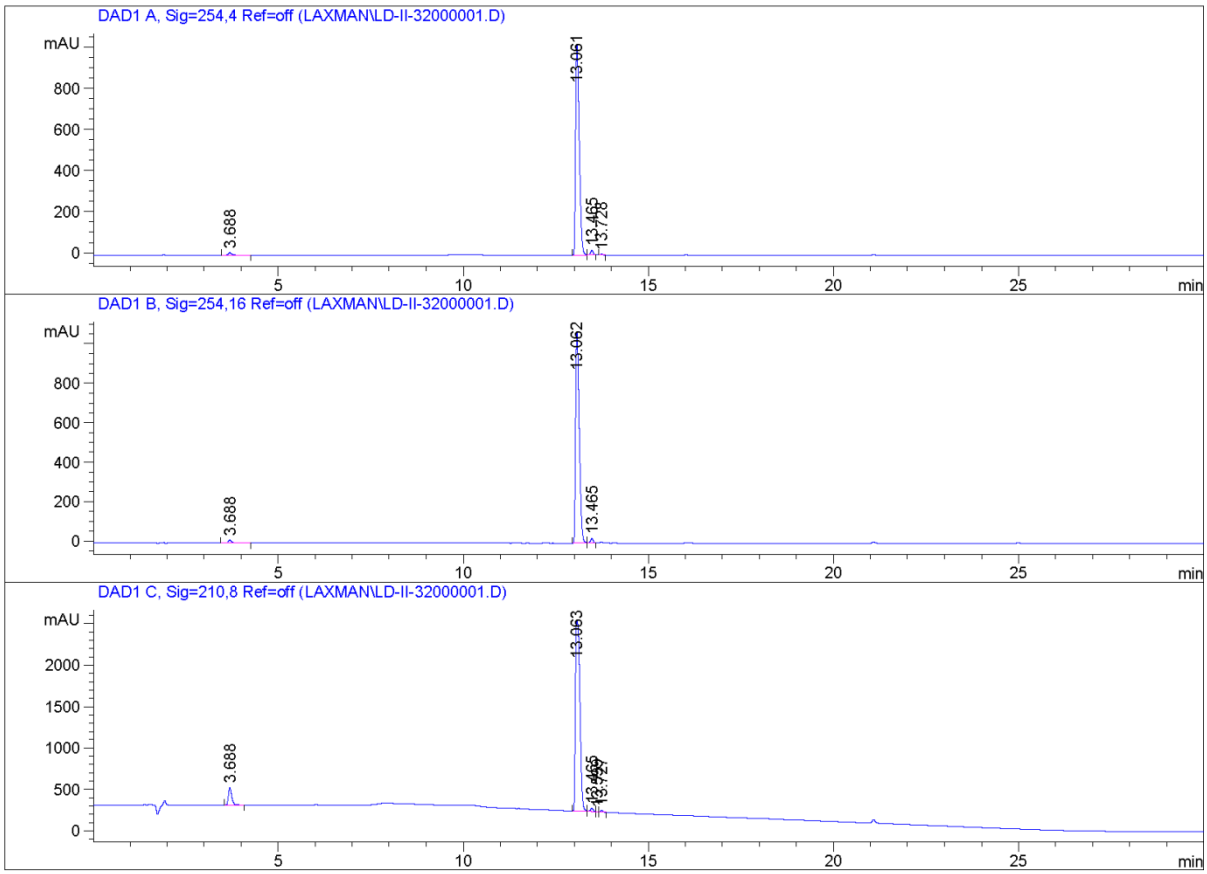
HRMS Compound **10**



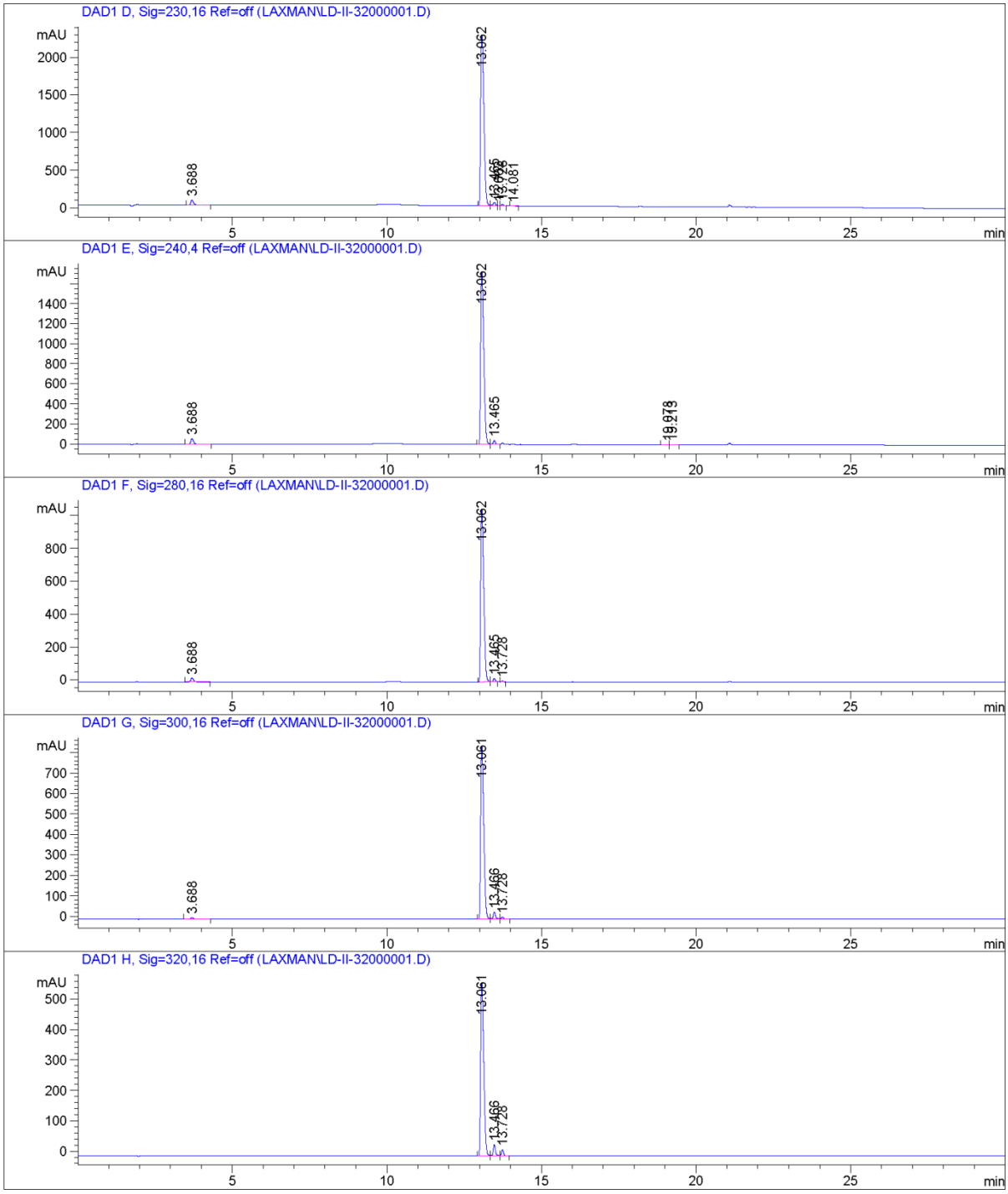
HPLC for Compound **10**

```
Data File C:\CHEM32\1\DATA\LAXMAN\LD-II-32000001.D
Sample Name: LD-II-32-run1

    ========================================================================
    Acq. Operator   : Laxman
    Acq. Instrument : Instrument 1                    Location :   -
    Injection Date  : 6/28/2015 4:33:05 PM
    Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD2.M
    Last changed    : 6/28/2015 4:06:36 PM by Eric Lin
    Analysis Method : C:\CHEM32\1\DATA\LAXMAN\LD-II-32000001.D\DA.M (MASTERMETHOD2.M)
    Last changed    : 6/28/2015 5:21:03 PM by Laxman
    Sample Info     : Method-Mastermethod2
```



Data File C:\CHEM32\1\DATA\LAXMAN\LD-II-32000001.D
Sample Name: LD-II-32-run1



Data File C:\CHEM32\1\DATA\LAXMAN\LD-II-32000001.D
Sample Name: LD-II-32-run1

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.688	BB	0.0941	70.04690	11.33046	0.9650
2	13.061	BV	0.1080	7049.17139	1027.60767	97.1129
3	13.465	VV	0.0763	120.15994	24.05628	1.6554
4	13.728	BB	0.0639	19.36081	4.71832	0.2667
Totals :				7258.73903	1067.71272	

Signal 2: DAD1 B, Sig=254,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.688	BB	0.0939	90.60506	14.70064	1.1994
2	13.062	BV	0.1080	7342.37061	1070.50842	97.1965
3	13.465	VV	0.0764	121.17745	24.21496	1.6041
Totals :				7554.15312	1109.42402	

Signal 3: DAD1 C, Sig=210,8 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.688	BB	0.0958	1287.63904	209.26445	6.1710
2	13.063	BV	0.1355	1.92646e4	2304.68799	92.3255
3	13.465	VV	0.0819	230.33527	42.03444	1.1039
4	13.599	VB	0.0544	11.39503	3.29091	0.0546
5	13.727	BB	0.0648	71.98167	17.20097	0.3450
Totals :				2.08659e4	2576.47876	

Data File C:\CHEM32\1\DATA\LAXMAN\LD-II-32000001.D
Sample Name: LD-II-32-run1

Signal 4: DAD1 D, Sig=230,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.688	BB	0.0936	411.70078	67.10372	2.3965
2	13.062	BV	0.1164	1.64493e4	2272.70361	95.7515
3	13.465	VV	0.0778	231.34738	45.13335	1.3467
4	13.602	VB	0.0543	8.60591	2.49010	0.0501
5	13.728	BB	0.0650	70.94254	16.90570	0.4130
6	14.081	BV	0.1019	7.25227	1.00877	0.0422
Totals :				1.71791e4	2405.34525	

Signal 5: DAD1 E, Sig=240,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.688	BB	0.0935	340.03543	55.47417	2.7247
2	13.062	BV	0.1086	1.18929e4	1721.22375	95.2988
3	13.465	VV	0.0842	224.57724	39.56317	1.7996
4	19.078	BV	0.1209	10.11429	1.24328	0.0810
5	19.213	VB	0.1184	11.95831	1.41727	0.0958
Totals :				1.24796e4	1818.92165	

Signal 6: DAD1 F, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.688	BB	0.0936	149.57484	24.38544	1.9959
2	13.062	BV	0.1079	7213.50439	1053.68127	96.2542
3	13.465	VV	0.0766	108.89276	21.67045	1.4530
4	13.728	BB	0.0643	22.24905	5.37009	0.2969
Totals :				7494.22105	1105.10725	

Signal 7: DAD1 G, Sig=300,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.688	BB	0.0965	37.61830	6.05143	0.6159
2	13.061	BV	0.1083	5836.52344	848.33795	95.5620

Data File C:\CHEM32\1\DATA\LAXMAN\LD-II-32000001.D
Sample Name: LD-II-32-run1

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
3	13.466	VV	0.0789	182.86238	35.00428	2.9940
4	13.728	VB	0.0714	50.57355	10.64560	0.8280
Totals :				6107.57767	900.03926	

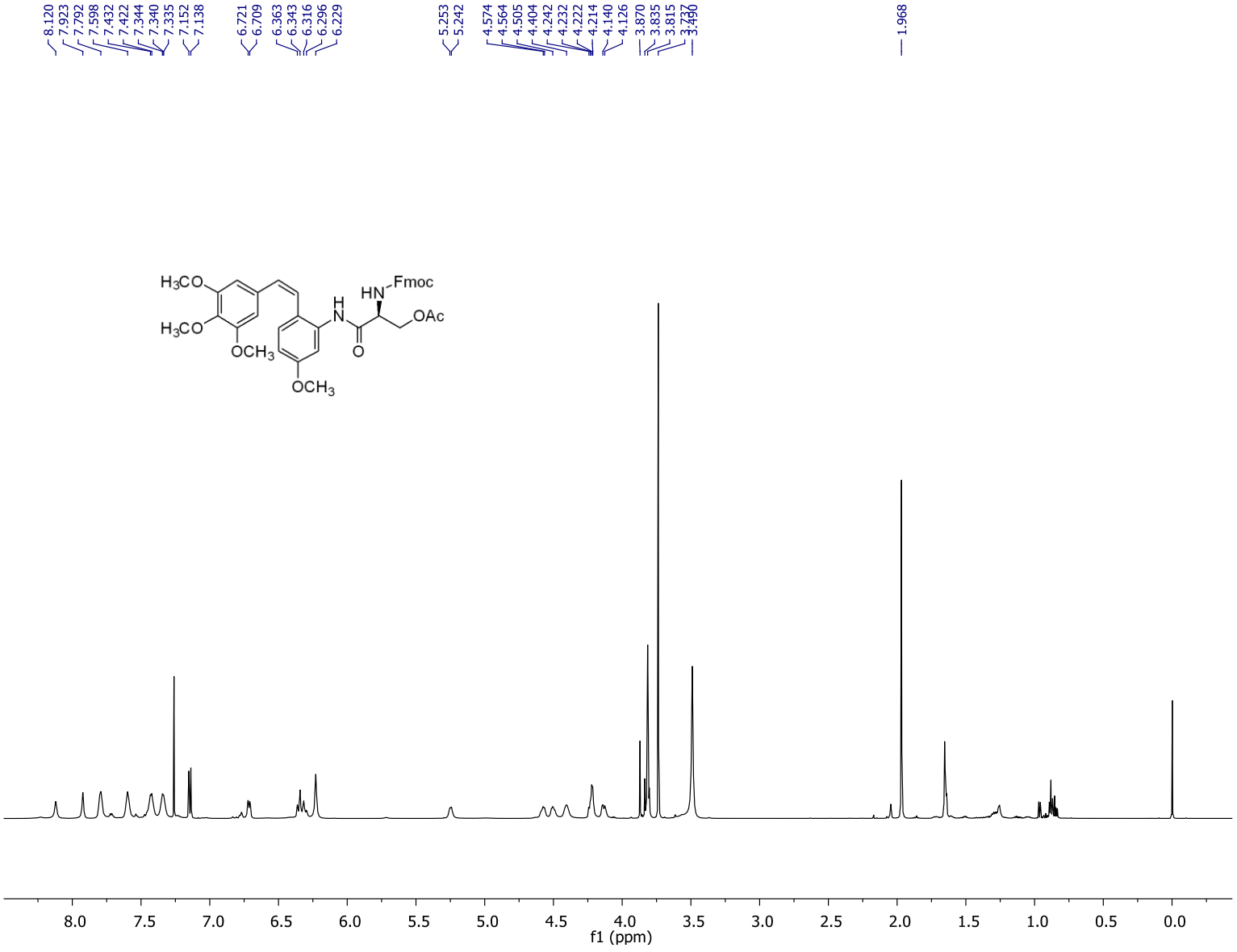
Signal 8: DAD1 H, Sig=320,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.061	BV	0.1086	3937.61230	569.77728	93.4290
2	13.466	VV	0.0762	185.44067	37.14345	4.4000
3	13.728	VB	0.0697	91.49677	19.89258	2.1710
Totals :				4214.54975	626.81331	

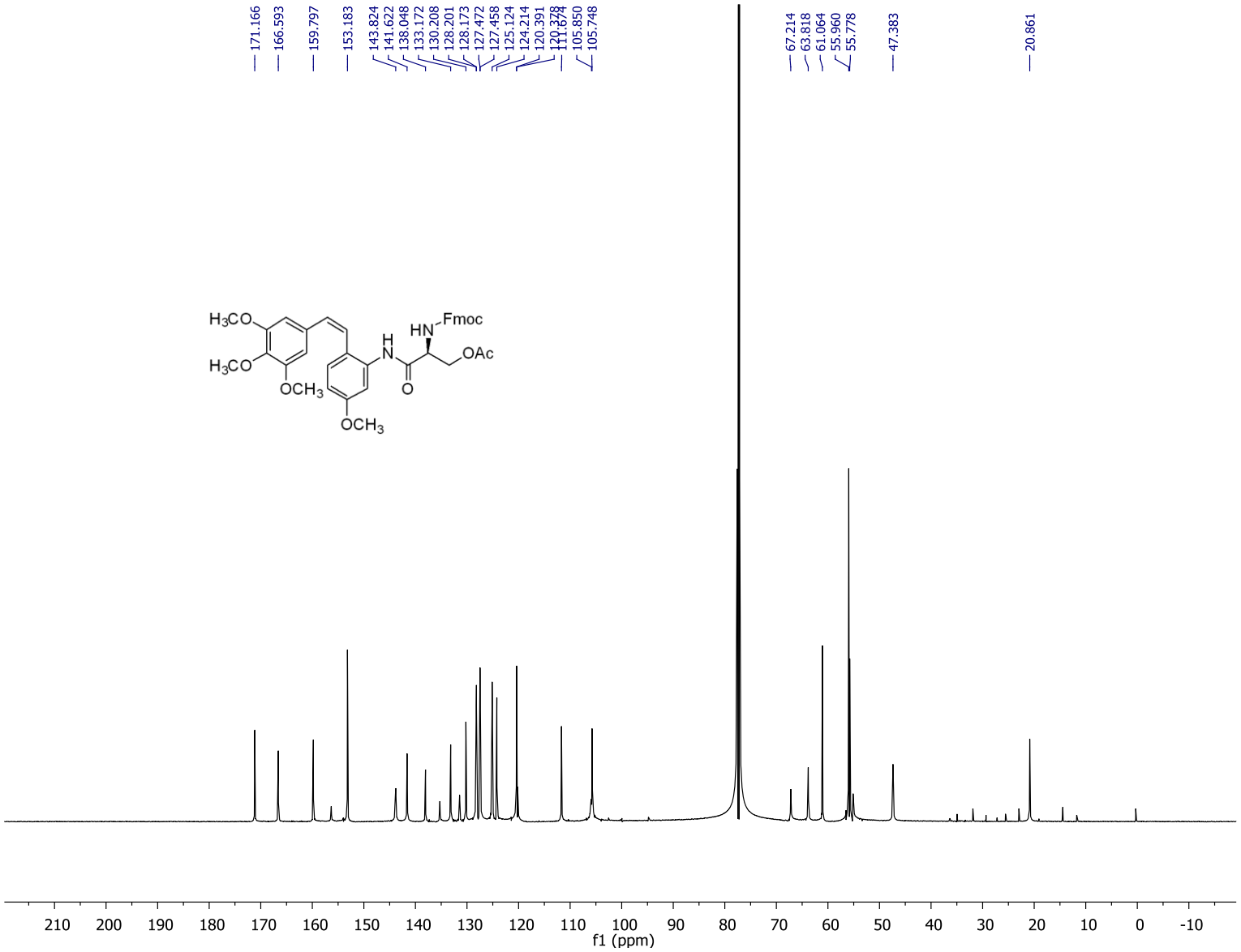
==========

*** End of Report ***

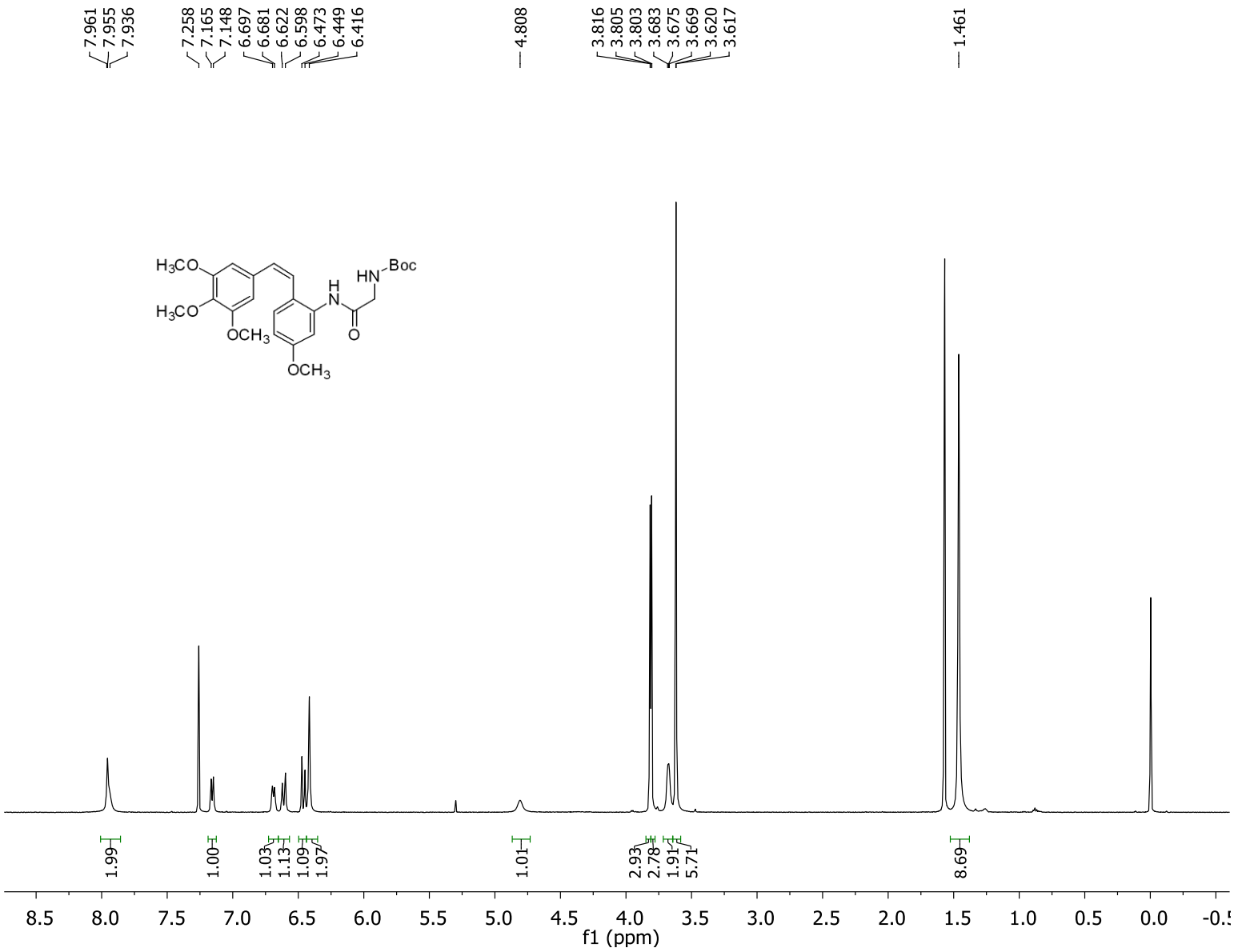
^{1}H NMR ($CDCl_3$, 500 MHz) Compound **11**



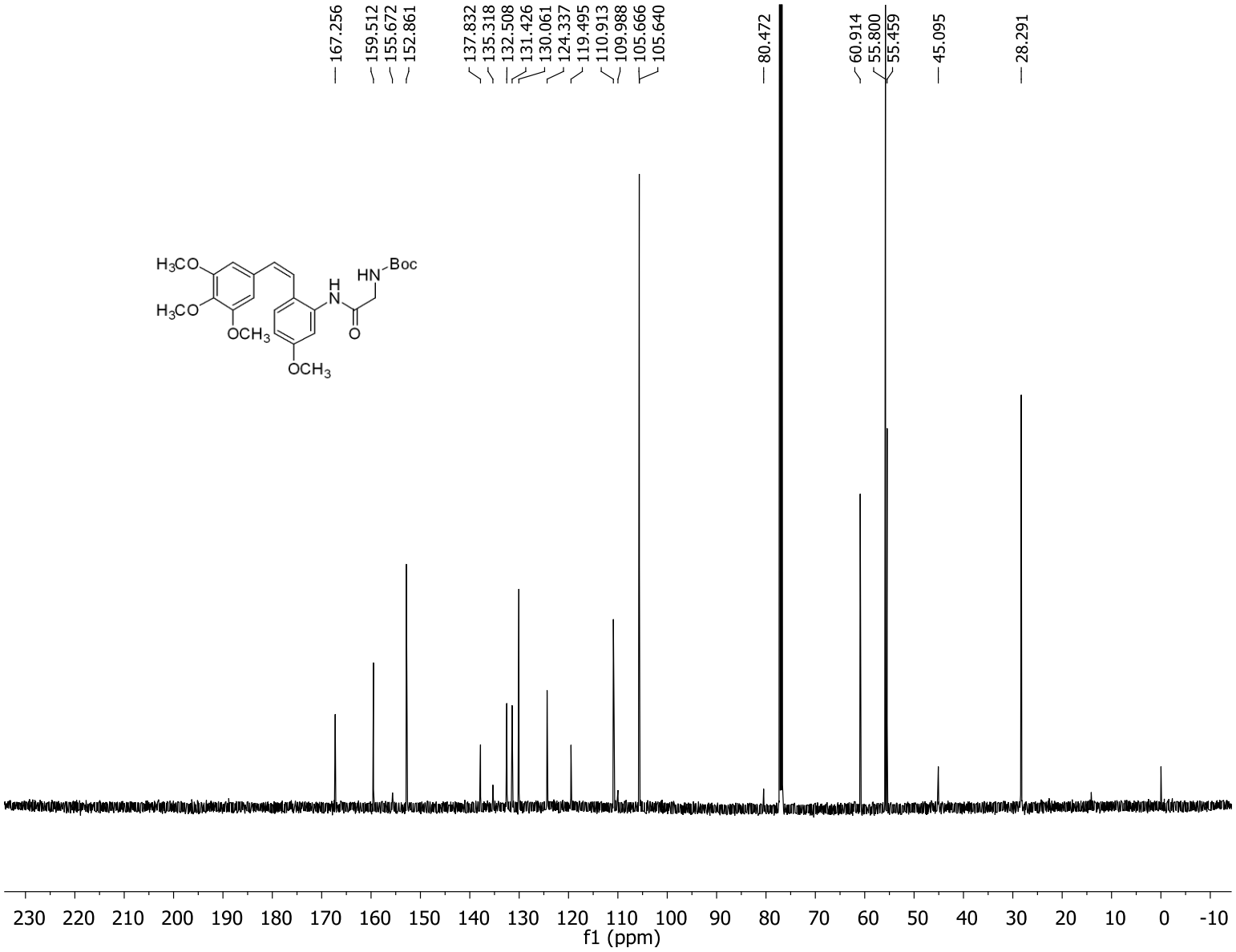
^{13}C NMR ($CDCl_3$, 150 MHz) Compound **11**



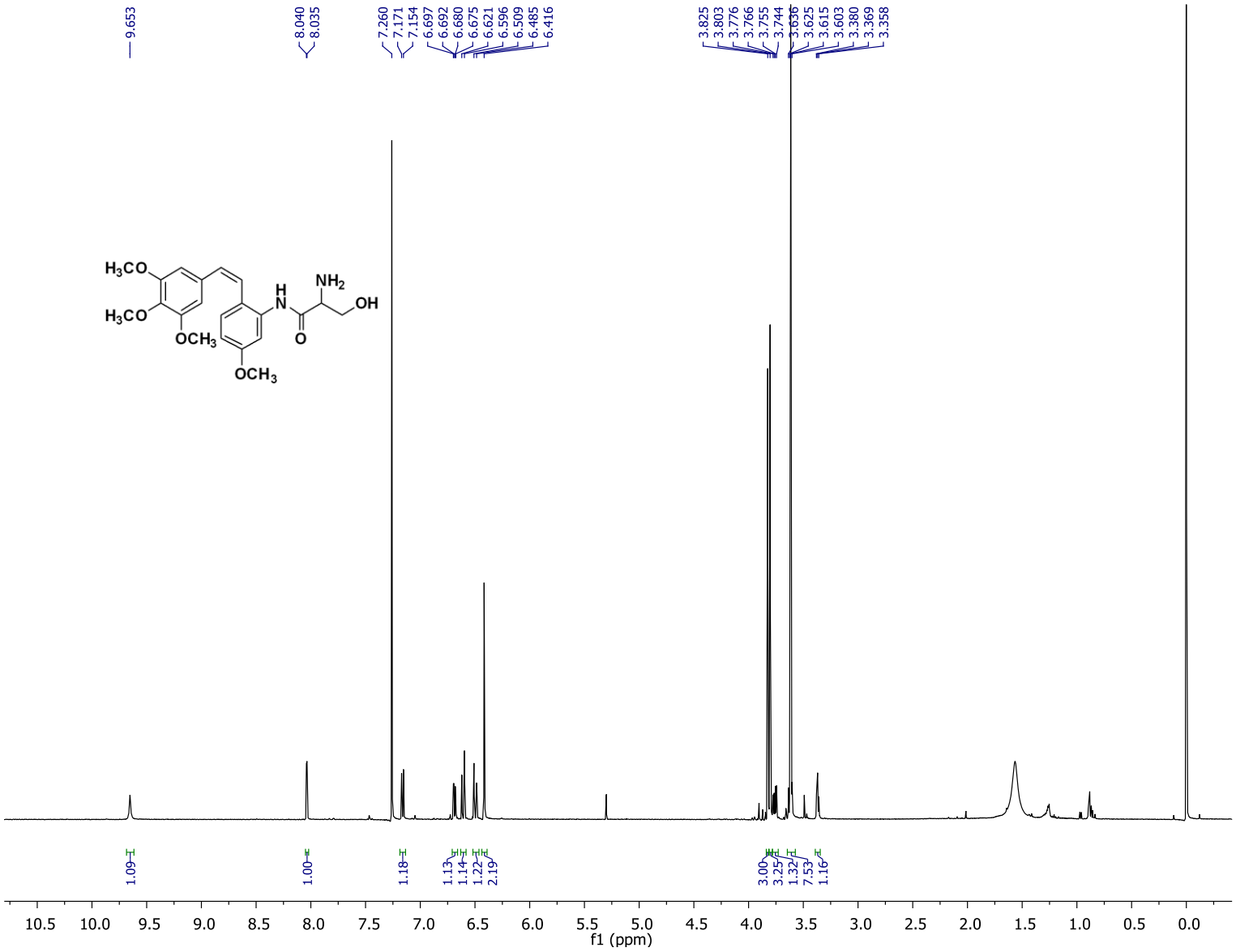
^{1}H NMR ($CDCl_3$, 500 MHz) Compound **12**



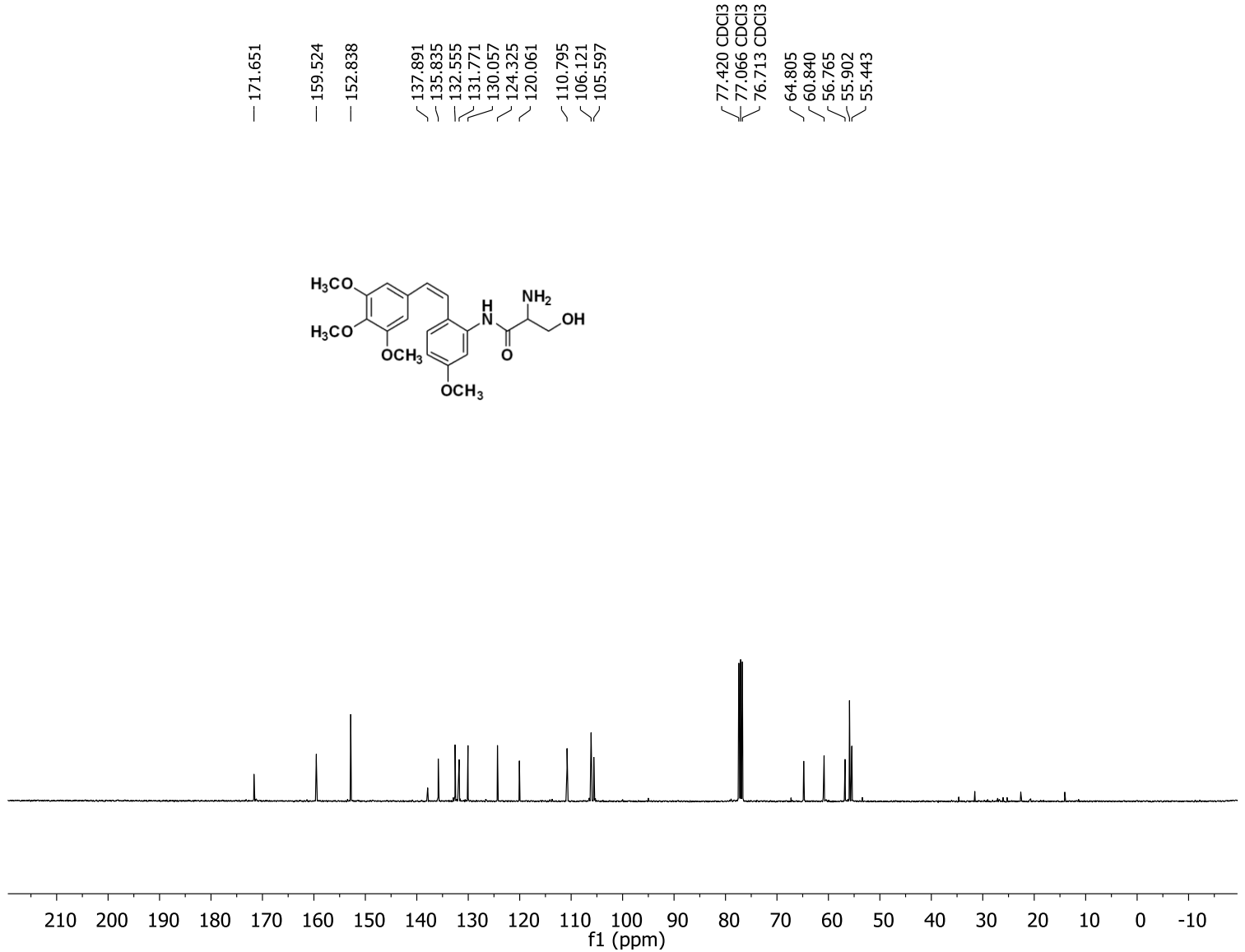
^{13}C NMR ($CDCl_3$, 125 MHz) Compound **12**



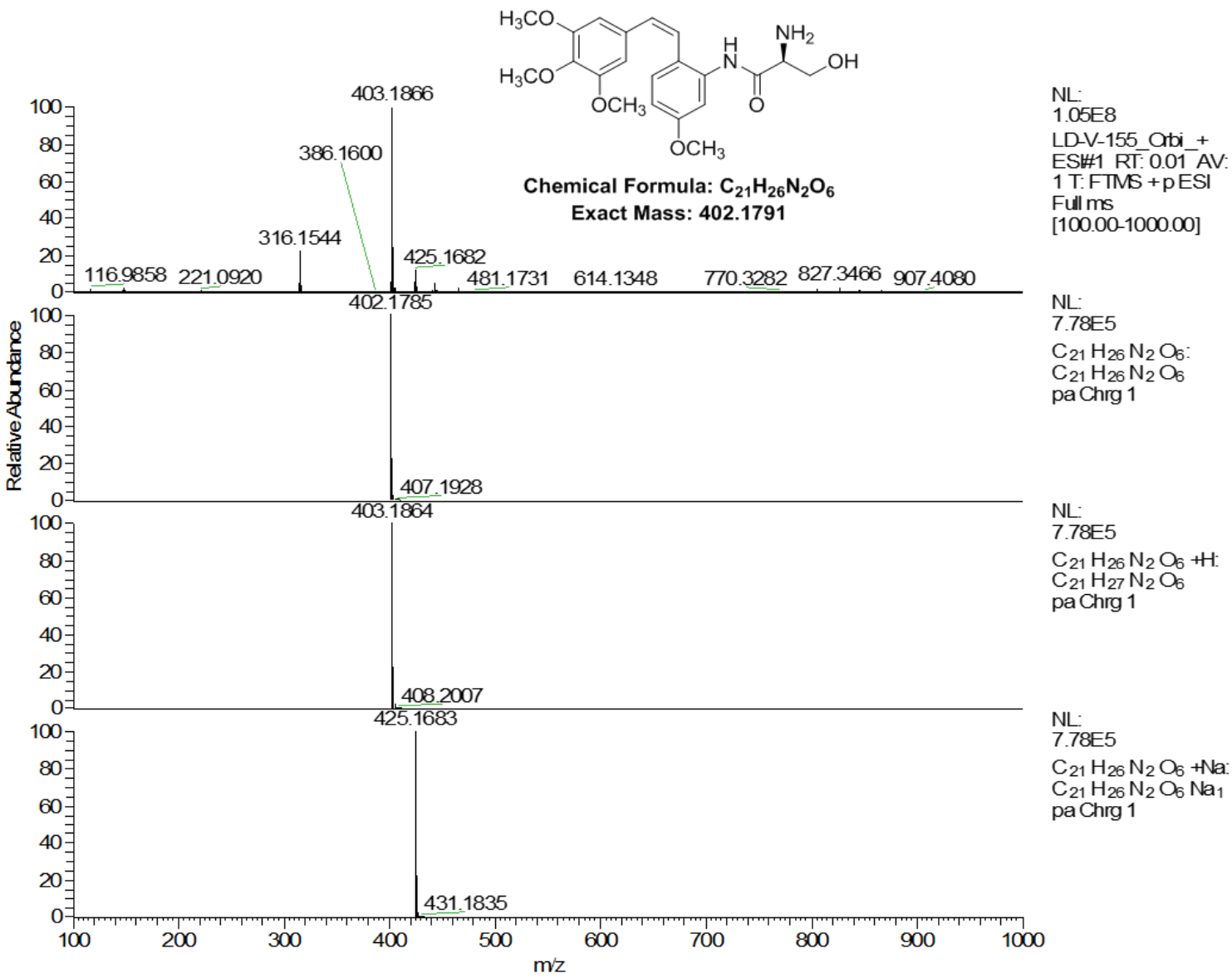
^{1}H NMR ($CDCl_3$, 500 MHz) Compound **13**



^{13}C NMR ($CDCl_3$, 90 MHz) Compound **13**



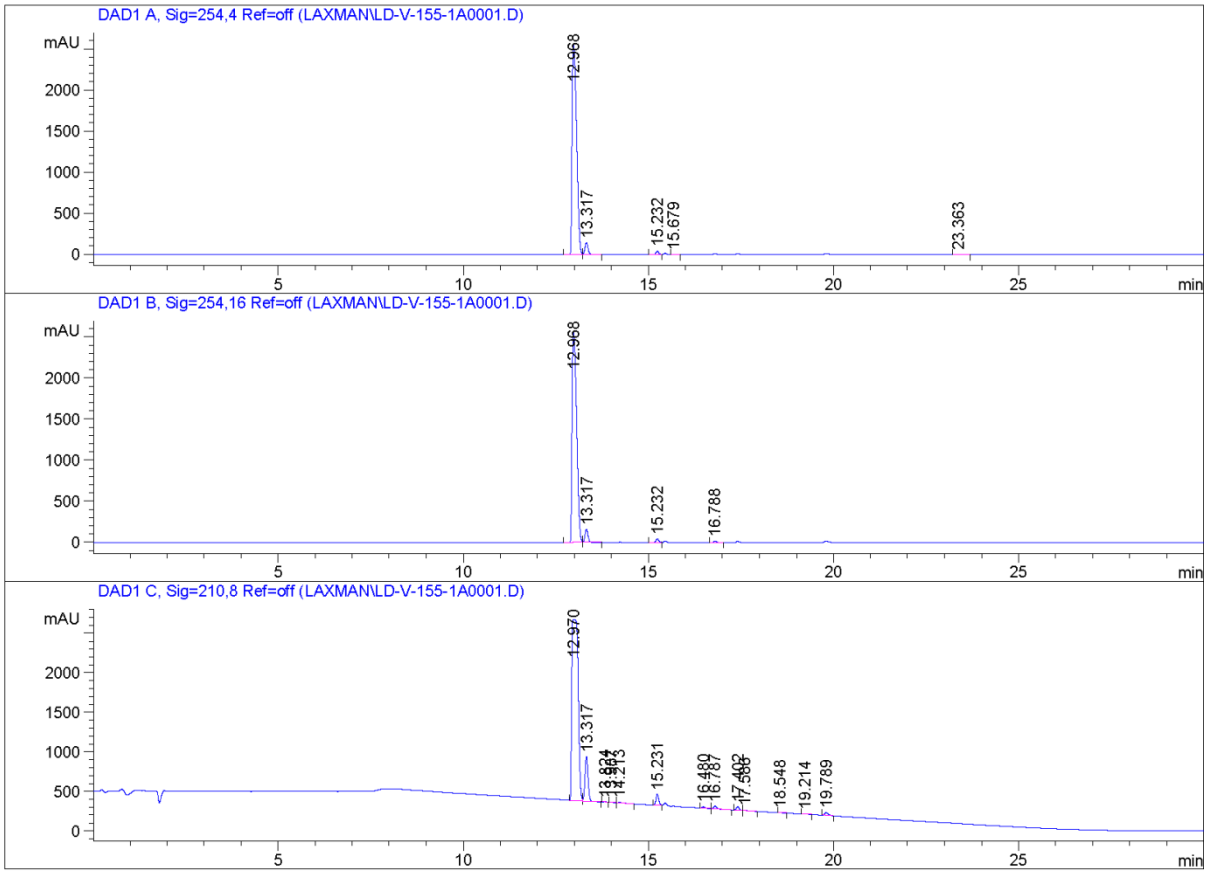
HRMS Compound **13**



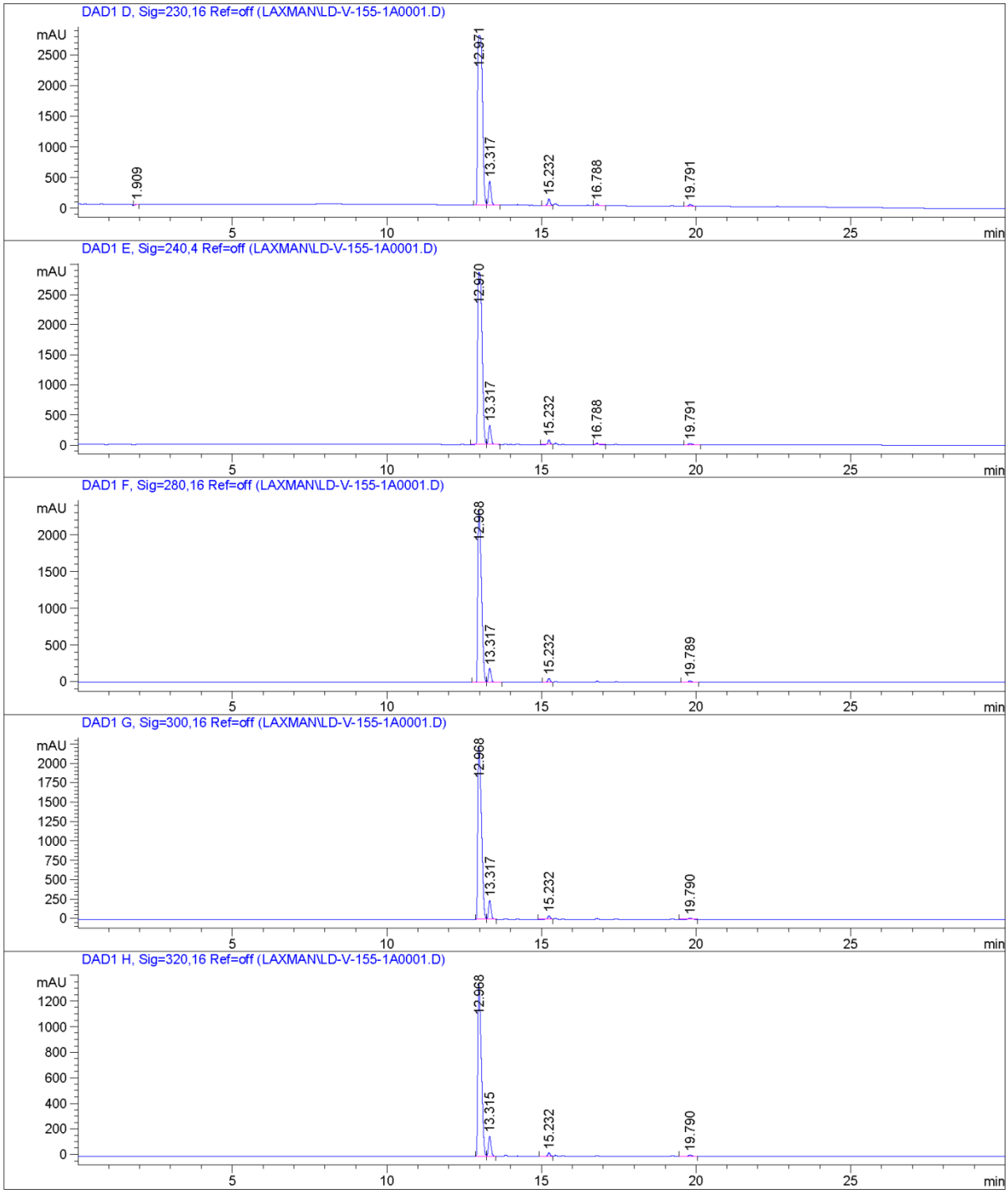
HPLC for Compound **13**

```
Data File C:\CHEM32\1\DATA\LAXMAN\LD-V-155-1A0001.D
Sample Name: LD-V-155-1A-run1

    ========================================================================
    Acq. Operator   : Laxman
    Acq. Instrument : Instrument 1                    Location :   -
    Injection Date  : 7/31/2014 1:28:27 PM
    Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD2.M
    Last changed    : 7/31/2014 1:12:57 PM by Laxman
    Analysis Method : C:\CHEM32\1\DATA\LAXMAN\LD-V-155-1A0001.D\DA.M (MASTERMETHOD2.M)
    Last changed    : 7/31/2014 2:17:20 PM by Laxman
    Sample Info     : Method- Mastermethod2
                      0-5 min 10:90 ACN: 0.1% TFA in Water
                      5-25 min 10:90 to 100:0 ACN: 0.1% TFA in Water
                      25-30 min 100:0 ACN: 0.1% TFA in Water
```



Data File C:\CHEM32\1\DATA\LAXMAN\LD-V-155-1A0001.D
Sample Name: LD-V-155-1A-run1



Data File C:\CHEM32\1\DATA\LAXMAN\LD-V-155-1A0001.D
Sample Name: LD-V-155-1A-run1

==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.968	BV	0.1362	2.16384e4	2571.50366	95.2518
2	13.317	VB	0.0888	820.29114	143.31441	3.6109
3	15.232	BV	0.0812	234.08575	43.19005	1.0304
4	15.679	VB	0.0841	9.86208	1.79634	0.0434
5	23.363	VB	0.1591	14.41530	1.20862	0.0635

Totals : 2.27171e4 2761.01308

Signal 2: DAD1 B, Sig=254,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.968	BV	0.1368	2.17823e4	2571.05396	94.5653
2	13.317	VB	0.0885	910.27527	159.73578	3.9519
3	15.232	BV	0.0812	252.29407	46.55761	1.0953
4	16.788	BB	0.0815	89.26048	16.92814	0.3875

Totals : 2.30341e4 2794.27549

Signal 3: DAD1 C, Sig=210,8 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.970	VV	0.1930	2.69550e4	2294.62939	84.3049
2	13.317	VB	0.0884	3234.33423	568.48431	10.1158
3	13.824	BB	0.0694	35.76576	7.82016	0.1119
4	13.987	BV	0.1578	19.65191	1.68774	0.0615
5	14.213	VB	0.1124	88.28443	10.91145	0.2761
6	15.231	VV	0.0804	750.52655	140.31758	2.3474
7	16.480	BV	0.0813	87.05302	16.56549	0.2723
8	16.787	VB	0.0843	200.55069	36.40348	0.6272
9	17.402	BV	0.0832	244.03952	45.06736	0.7633

Data File C:\CHEM32\1\DATA\LAXMAN\LD-V-155-1A0001.D
Sample Name: LD-V-155-1A-run1

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
10	17.586	VB	0.1047	37.94374	5.22775	0.1187
11	18.548	BB	0.0999	15.19789	2.27553	0.0475
12	19.214	BB	0.0957	43.13522	6.65227	0.1349
13	19.789	BB	0.1042	261.74756	36.26916	0.8186
Totals :				3.19732e4	3172.31167	

Signal 4: DAD1 D, Sig=230,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.909	BB	0.1103	64.22646	9.81579	0.1960
2	12.971	BV	0.1748	2.95450e4	2762.10352	90.1549
3	13.317	VB	0.0877	2179.68066	387.20401	6.6512
4	15.232	BV	0.0813	588.52106	108.53487	1.7958
5	16.788	VB	0.0830	164.31732	30.45298	0.5014
6	19.791	VV	0.1089	229.63806	30.12724	0.7007
Totals :				3.27713e4	3328.23841	

Signal 5: DAD1 E, Sig=240,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.970	BV	0.1595	2.78349e4	2861.06689	91.6270
2	13.317	VB	0.0877	1778.19116	315.66486	5.8535
3	15.232	BV	0.0815	446.13184	81.93338	1.4686
4	16.788	BV	0.0820	134.76234	25.35521	0.4436
5	19.791	VB	0.1138	184.50830	22.95249	0.6074
Totals :				3.03785e4	3306.97284	

Signal 6: DAD1 F, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.968	BV	0.1313	1.93204e4	2365.42163	92.6736
2	13.317	VB	0.0878	1085.78162	192.58340	5.2082
3	15.232	BV	0.0810	302.56723	56.02794	1.4513
4	19.789	BB	0.1053	139.03084	19.01400	0.6669

Data File C:\CHEM32\1\DATA\LAXMAN\LD-V-155-1A0001.D
Sample Name: LD-V-155-1A-run1

Totals : 2.08477e4 2633.04697

Signal 7: DAD1 G, Sig=300,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.968	BV	0.1321	1.83907e4	2233.27051	91.3035
2	13.317	VB	0.0875	1356.34009	241.75505	6.7338
3	15.232	BV	0.0823	263.98782	47.88708	1.3106
4	19.790	BB	0.1094	131.35910	17.14645	0.6522

Totals : 2.01423e4 2540.05910

Signal 8: DAD1 H, Sig=320,16 Ref=off

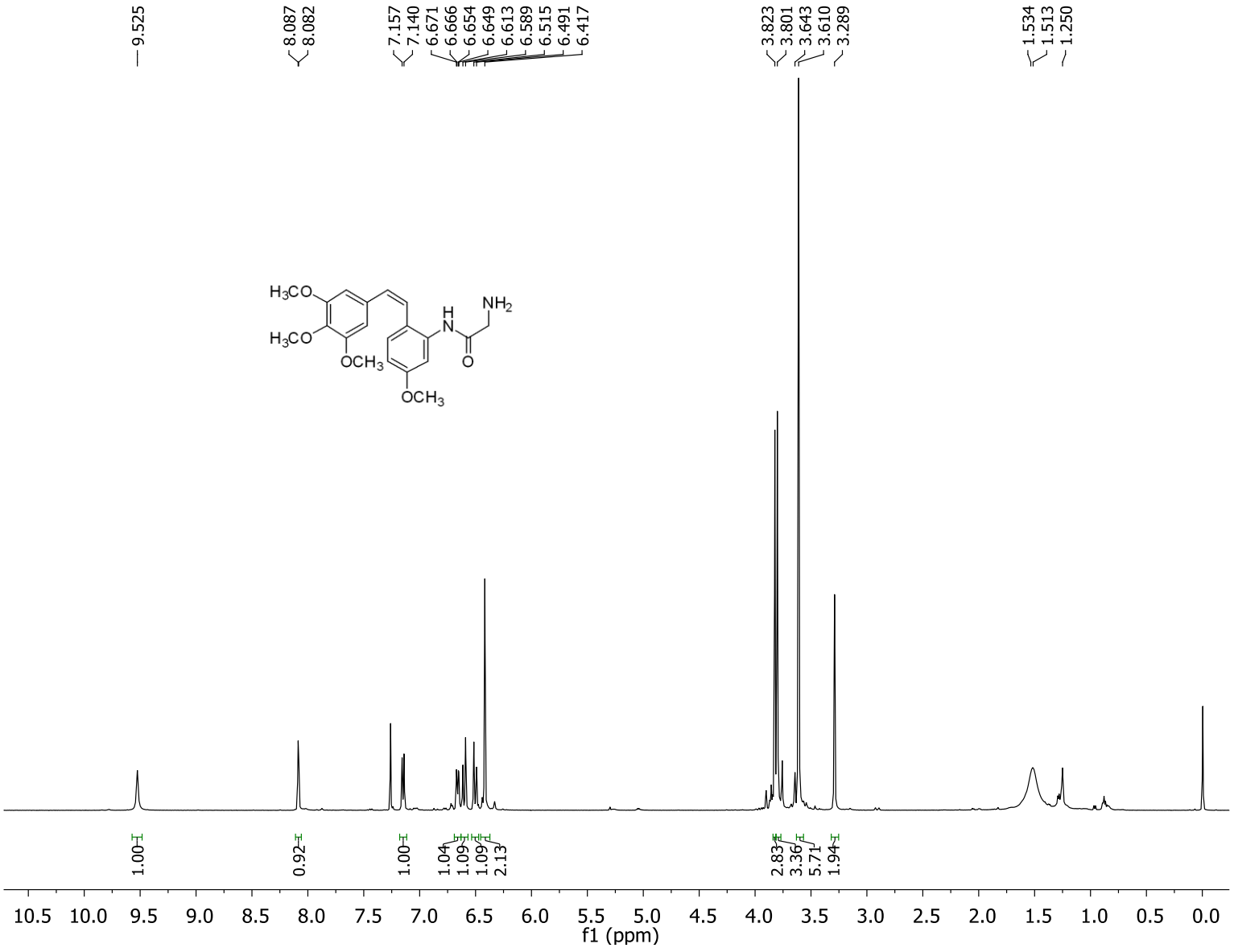
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.968	BV	0.1217	1.06545e4	1356.26221	90.4901
2	13.315	VB	0.0905	890.75409	156.25275	7.5653
3	15.232	BV	0.0826	148.63684	26.85264	1.2624
4	19.790	BB	0.1090	80.32684	10.53331	0.6822

Totals : 1.17742e4 1549.90090

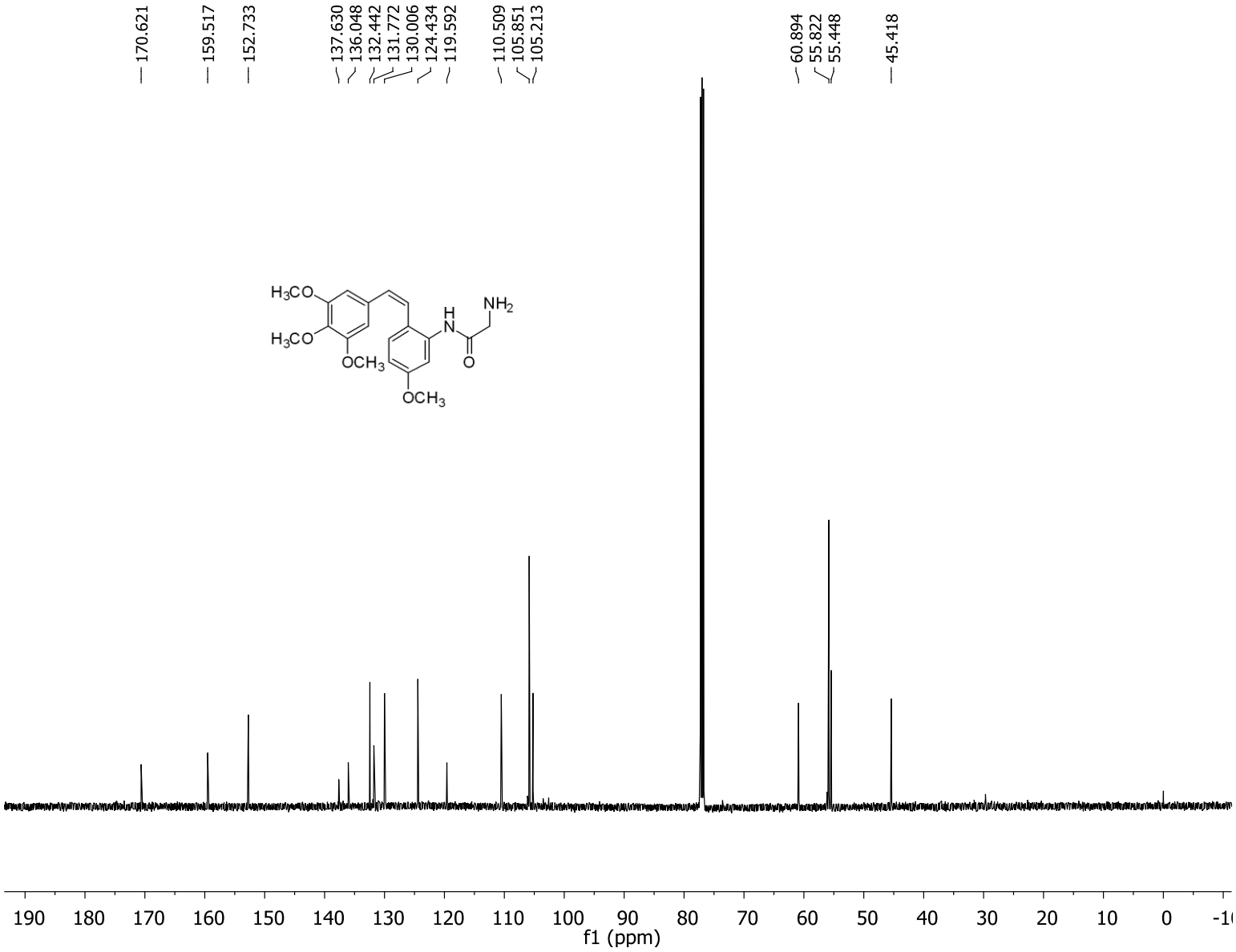
==========

*** End of Report ***

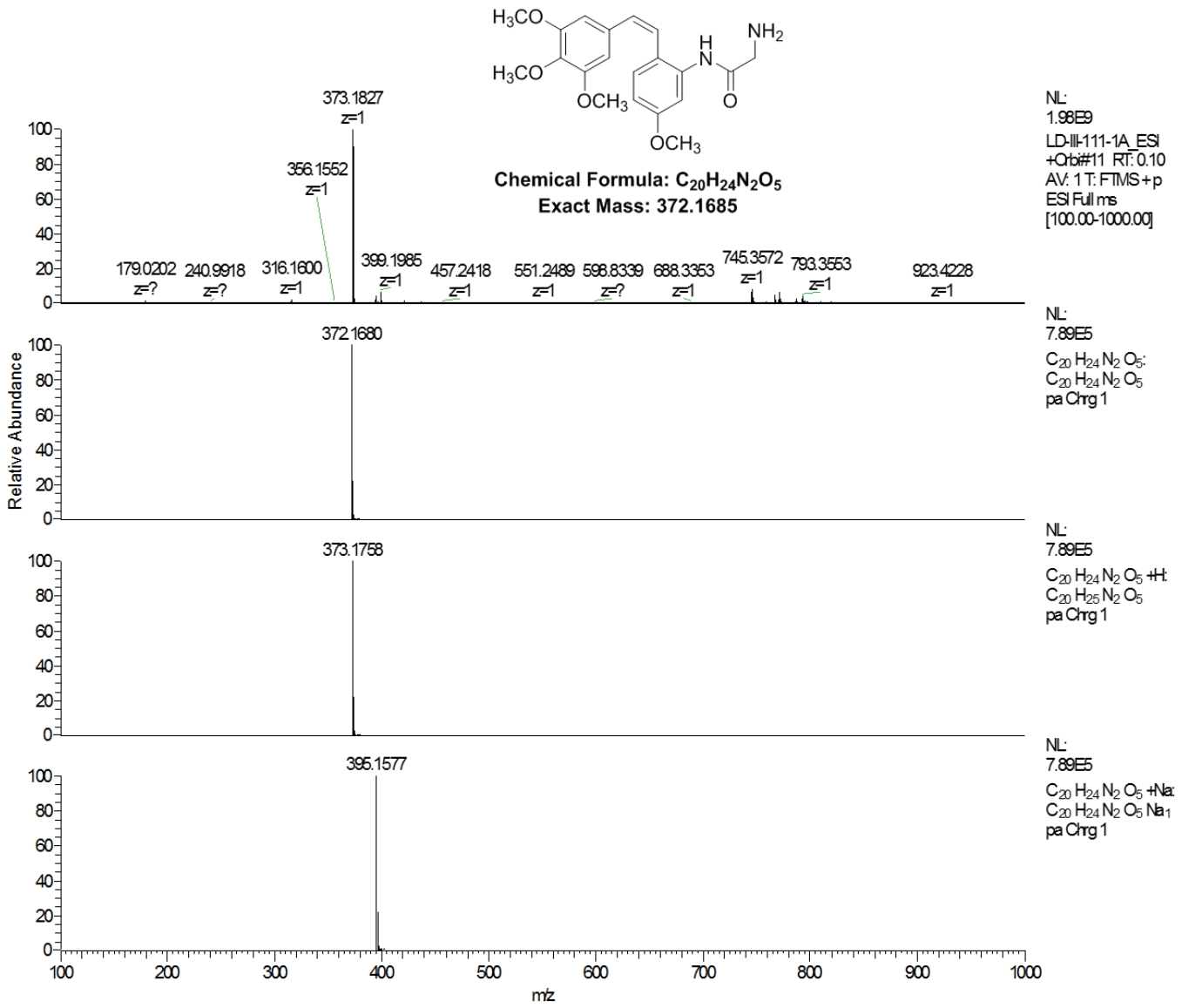
^{1}H NMR ($CDCl_3$, 500 MHz) Compound **14**



^{13}C NMR ($CDCl_3$, 125 MHz) Compound **14**



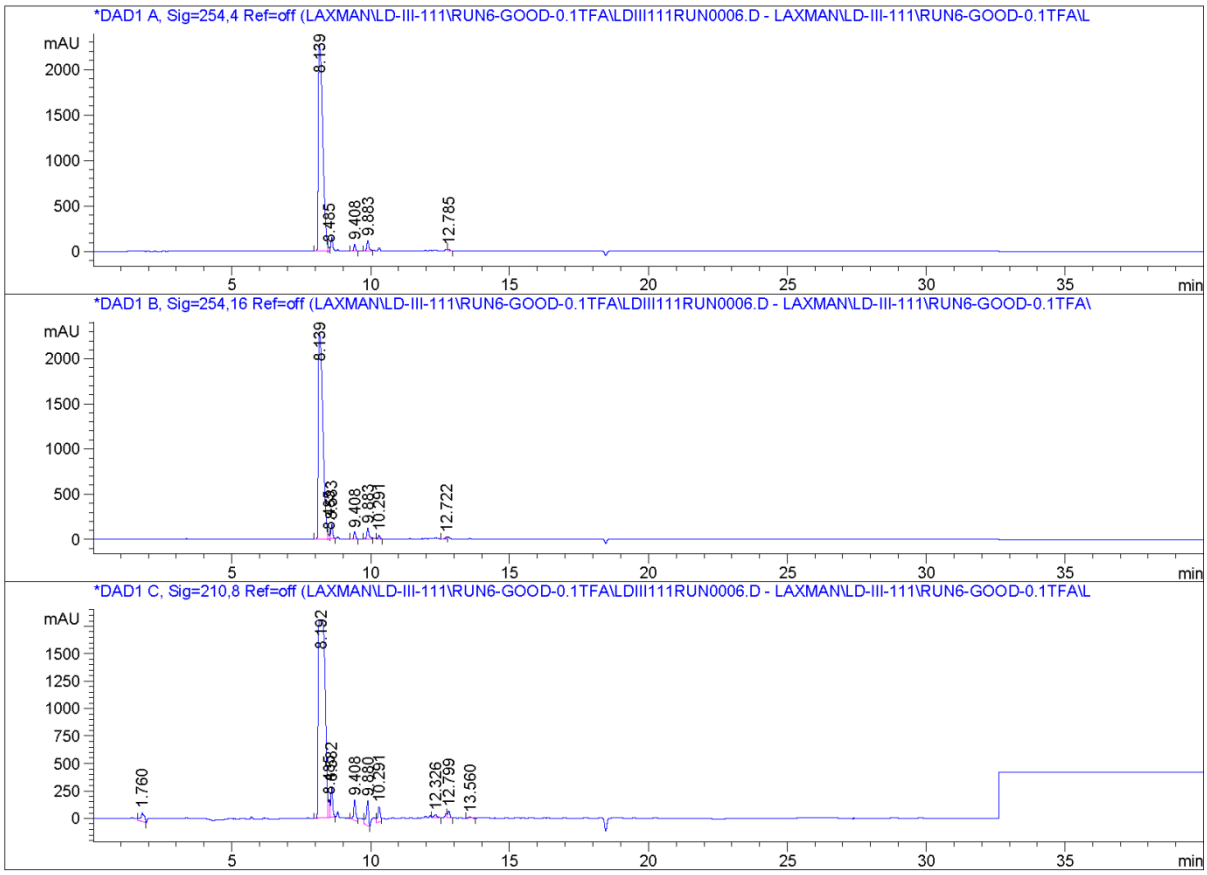
HRMS Compound **14**



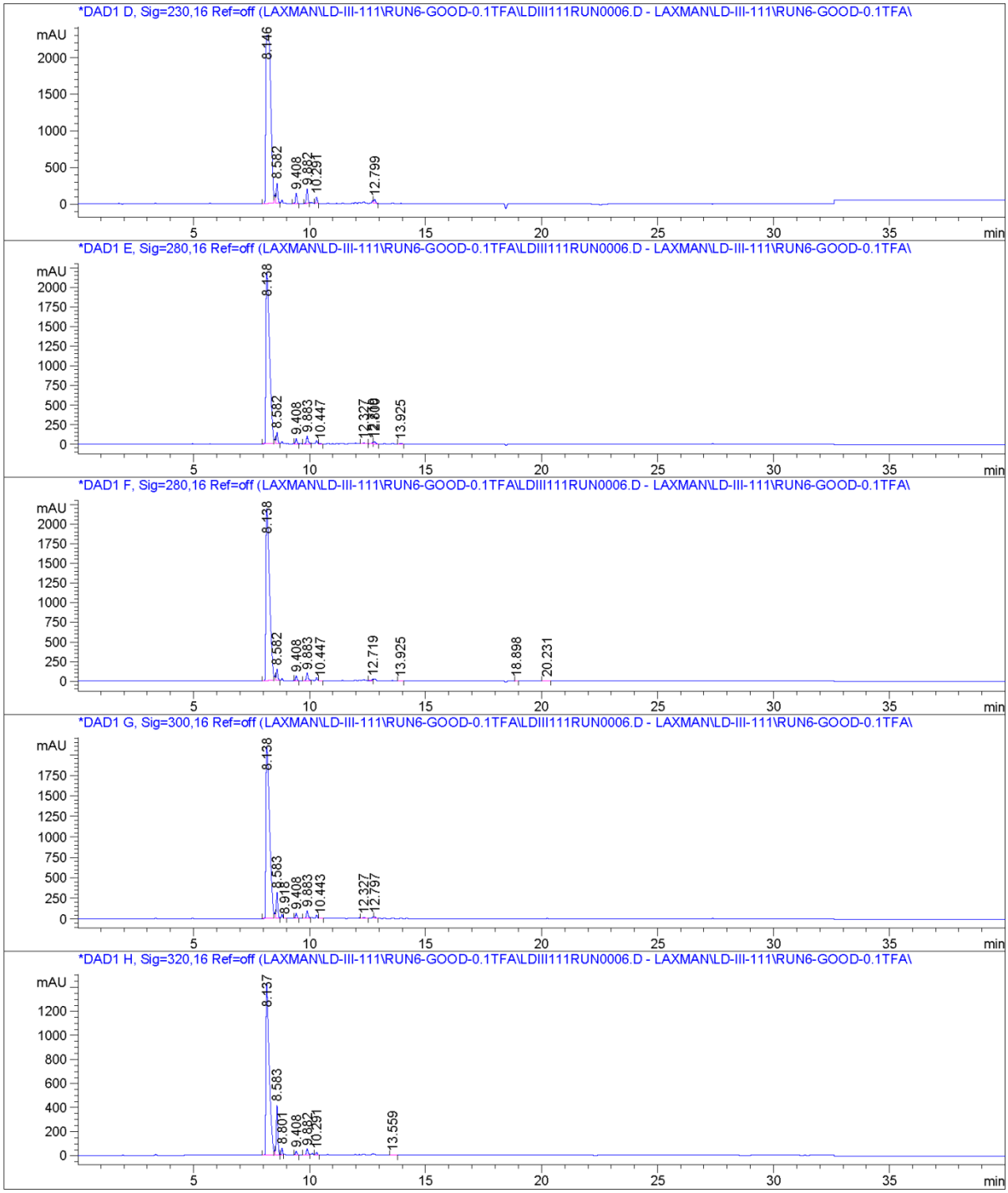
HPLC for Compound **14**

```
Data File C:\CHEM32\1\DATA\LAXMAN\LD-III-111\RUN6-GOOD-0.1TFA\LDIII111RUN0006.D
Sample Name: LD-III-111-run6

    ======================================================================
    Acq. Operator   : Laxman
    Acq. Instrument : Instrument 1                    Location :   -
    Injection Date  : 7/9/2013 9:00:02 PM
    Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD.M
    Last changed    : 7/9/2013 6:17:01 PM by Laxman
    Analysis Method : C:\CHEM32\1\DATA\LAXMAN\LD-III-111\RUN6-GOOD-0.1TFA\LDIII111RUN0006.D\DA.M (
                      MASTERMETHOD.M)
    Last changed    : 4/1/2014 10:55:34 AM by ZHE
    Sample Info     : Method- Mastermethod
                      0.1% TFA in Water
```



Data File C:\CHEM32\1\DATA\LAXMAN\LD-III-111\RUN6-GOOD-0.1TFA\LDIII111RUN0006.D
Sample Name: LD-III-111-run6



Data File C:\CHEM32\1\DATA\LAXMAN\LD-III-111\RUN6-GOOD-0.1TFA\LDIII111RUN0006.D
Sample Name: LD-III-111-run6

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.139	BV	0.1754	2.51894e4	2271.24341	95.4505
2	8.485	VV	0.0515	142.75304	42.16708	0.5409
3	9.408	BV	0.0674	326.38943	74.15012	1.2368
4	9.883	BV	0.0796	640.42310	117.46875	2.4268
5	12.785	VB	0.0730	91.04413	19.30842	0.3450
Totals :				2.63900e4	2524.33778	

Signal 2: DAD1 B, Sig=254,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.139	BV	0.1799	2.58701e4	2288.13721	92.2516
2	8.485	VV	0.0526	167.77173	48.26380	0.5983
3	8.583	VB	0.0693	721.26178	158.01703	2.5720
4	9.408	BV	0.0674	337.57236	76.65775	1.2038
5	9.883	BV	0.0794	656.34247	120.76386	2.3405
6	10.291	VB	0.0718	171.31871	37.16681	0.6109
7	12.722	BV	0.0809	118.60592	21.33296	0.4229
Totals :				2.80430e4	2750.33941	

Signal 3: DAD1 C, Sig=210,8 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.760	VB	0.1147	713.67432	81.08369	1.9280
2	8.192	BV	0.2641	3.05965e4	1801.82019	82.6552
3	8.485	VV	0.0556	607.23651	162.55066	1.6404

Data File C:\CHEM32\1\DATA\LAXMAN\LD-III-111\RUN6-GOOD-0.1TFA\LDIII111RUN0006.D
Sample Name: LD-III-111-run6

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
4	8.582	VB	0.0698	1298.44763	281.44962	3.5077
5	9.408	BV	0.0794	991.03918	182.45256	2.6772
6	9.880	VB	0.0838	1236.96521	226.01939	3.3416
7	10.291	VV	0.0889	863.00427	141.99573	2.3314
8	12.326	VB	0.1201	283.79422	32.43545	0.7667
9	12.799	VV	0.0854	353.40680	63.03465	0.9547
10	13.560	BV	0.0934	72.98273	11.92350	0.1972
Totals :				3.70171e4	2984.76544	

Signal 4: DAD1 D, Sig=230,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.146	BV	0.2404	3.43759e4	2300.19604	90.7117
2	8.582	VB	0.0695	1254.74963	273.39069	3.3111
3	9.408	BV	0.0685	651.16071	144.70052	1.7183
4	9.882	BV	0.0693	926.08765	202.89774	2.4438
5	10.291	BV	0.0704	385.79929	85.91025	1.0181
6	12.799	VB	0.0832	302.09579	54.06322	0.7972
Totals :				3.78958e4	3061.15845	

Signal 5: DAD1 E, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.138	BV	0.1715	2.42665e4	2186.85913	93.0409
2	8.582	VB	0.0692	656.98676	143.97168	2.5190
3	9.408	VB	0.0680	282.83658	63.43553	1.0844
4	9.883	BV	0.0788	524.90851	97.49480	2.0126
5	10.447	VB	0.0706	8.61436	1.84189	0.0330
6	12.327	VB	0.1122	90.34814	11.42952	0.3464
7	12.719	BV	0.0755	94.46789	19.16719	0.3622
8	12.800	VB	0.0819	142.46277	26.00443	0.5462
9	13.925	BB	0.1102	14.42253	2.09712	0.0553
Totals :				2.60816e4	2552.30129	

Data File C:\CHEM32\1\DATA\LAXMAN\LD-III-111\RUN6-GOOD-0.1TFA\LDIII111RUN0006.D
Sample Name: LD-III-111-run6

Signal 6: DAD1 F, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.138	BV	0.1715	2.42665e4	2186.85913	93.7362
2	8.582	VB	0.0692	656.98676	143.97168	2.5378
3	9.408	VB	0.0680	282.83658	63.43553	1.0925
4	9.883	BV	0.0788	524.90851	97.49480	2.0276
5	10.447	VB	0.0706	8.61436	1.84189	0.0333
6	12.719	BV	0.0755	94.46789	19.16719	0.3649
7	13.925	BB	0.1102	14.42253	2.09712	0.0557
8	18.898	VB	0.1124	30.22587	3.81837	0.1168
9	20.231	BB	0.0976	9.12180	1.40804	0.0352
Totals :				2.58881e4	2520.09375	

Signal 7: DAD1 G, Sig=300,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.138	BV	0.1723	2.34731e4	2101.46777	90.7065
2	8.583	VB	0.0679	1408.04065	316.57404	5.4410
3	8.918	VB	0.0565	13.70495	3.76272	0.0530
4	9.408	VB	0.0681	275.41730	61.68370	1.0643
5	9.883	BV	0.0795	513.82751	94.44556	1.9856
6	10.443	VB	0.0686	8.27514	1.83509	0.0320
7	12.327	VB	0.1099	91.84422	11.91732	0.3549
8	12.797	VB	0.0747	93.86009	18.64861	0.3627
Totals :				2.58781e4	2610.33480	

Signal 8: DAD1 H, Sig=320,16 Ref=off
Signal has been modified after loading from rawdata file!

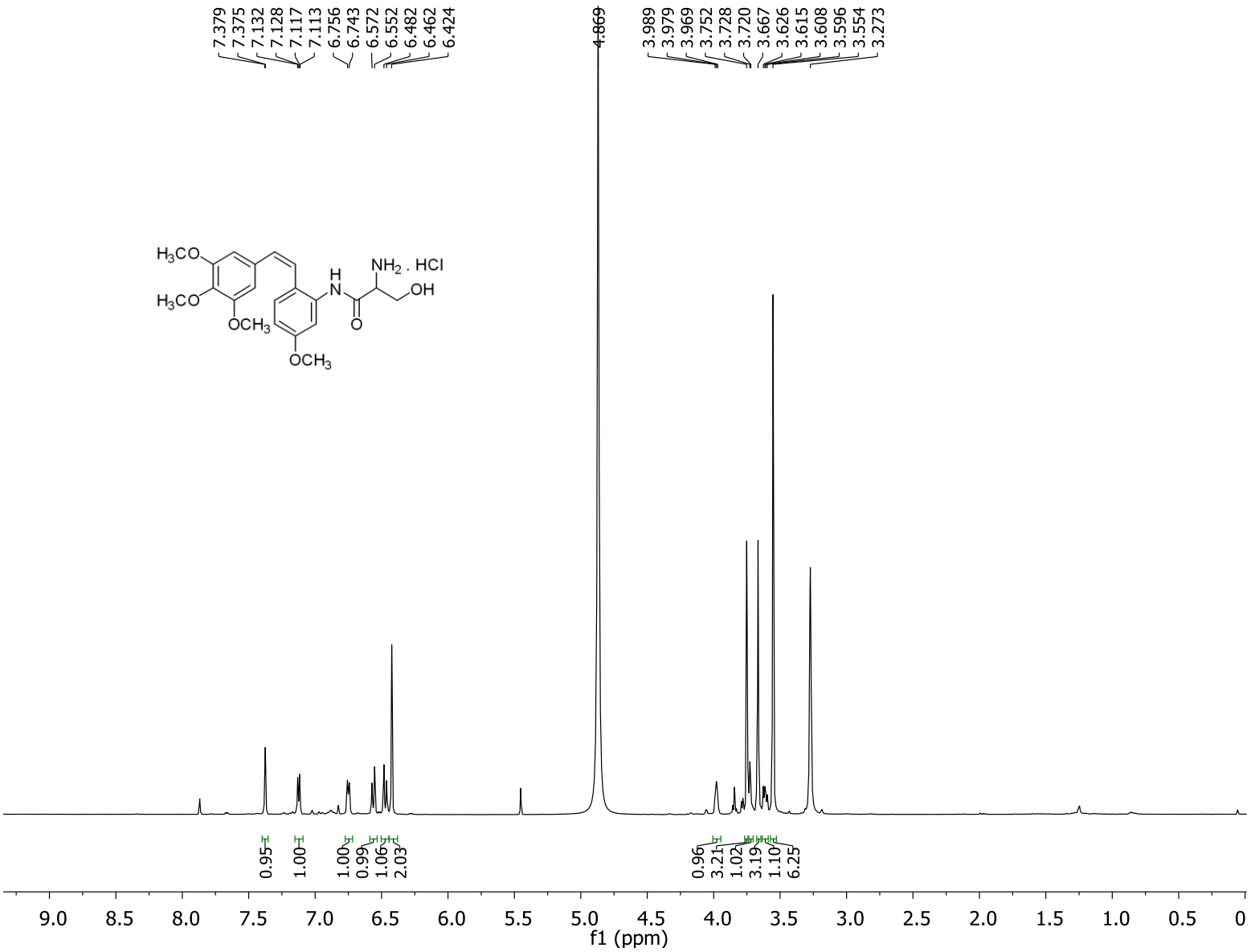
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.137	BV	0.1477	1.41791e4	1430.12402	84.7866
2	8.583	VB	0.0668	1786.22986	410.09262	10.6811
3	8.801	BV	0.0635	214.57774	52.71780	1.2831
4	9.408	VB	0.0681	151.69165	33.99868	0.9071
5	9.882	BV	0.0780	268.97385	50.61902	1.6084
6	10.291	VV	0.0737	110.94556	23.25360	0.6634
7	13.559	BB	0.0920	11.75747	1.90529	0.0703

Data File C:\CHEM32\1\DATA\LAXMAN\LD-III-111\RUN6-GOOD-0.1TFA\LDIII111RUN0006.D
Sample Name: LD-III-111-run6

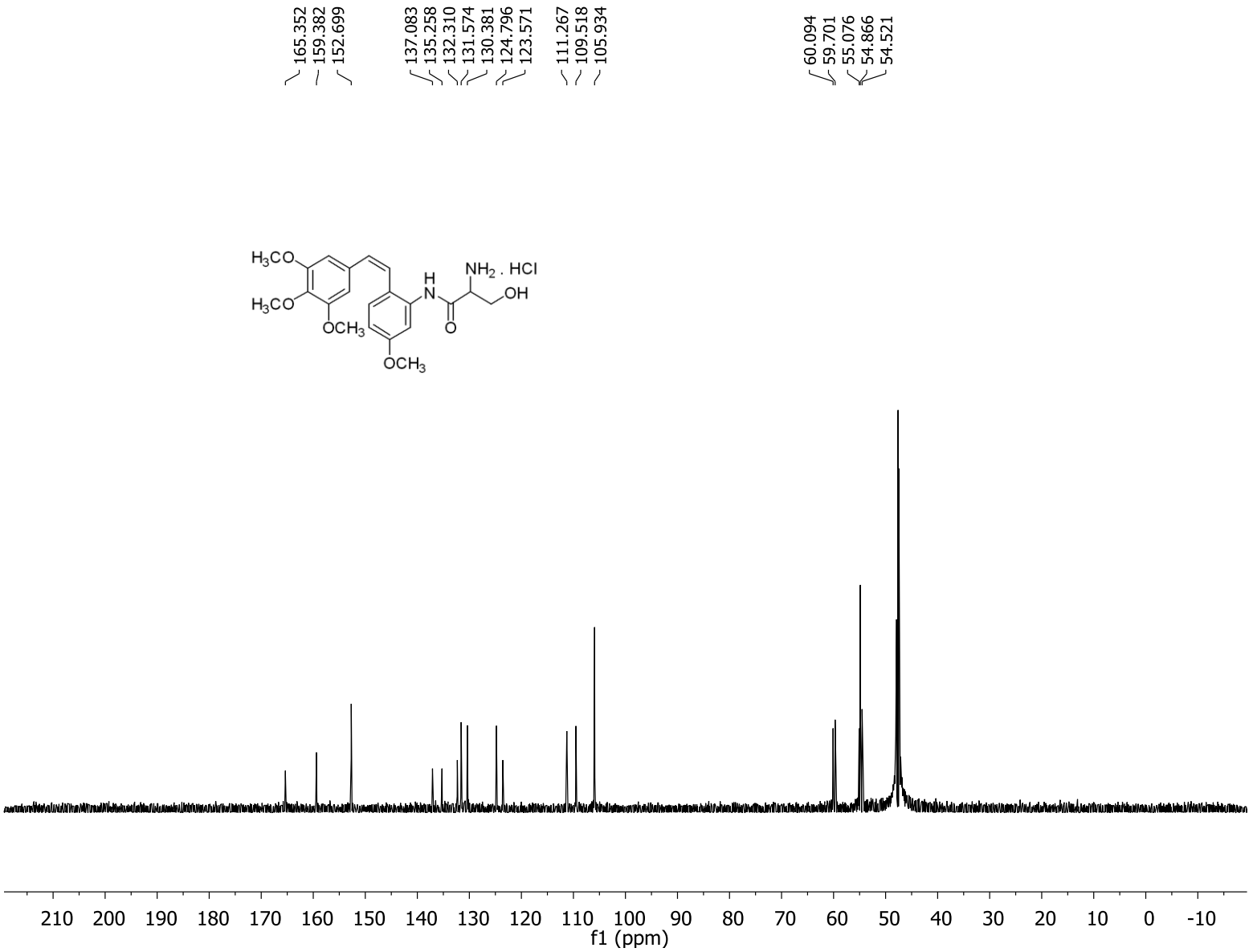
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
Totals :				1.67233e4	2002.71104	

*** End of Report ***

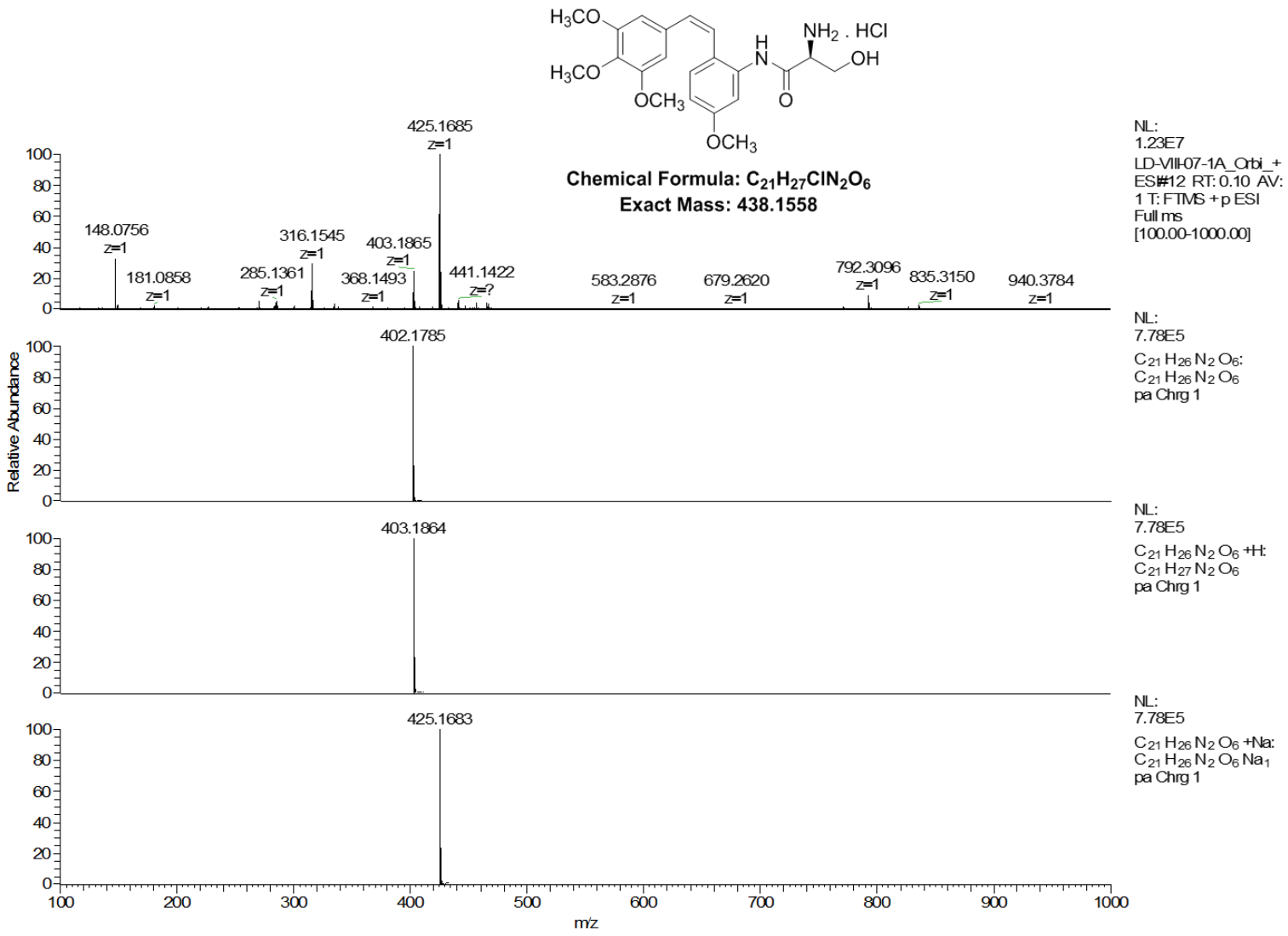
^{1}H NMR (CD_3OD, 600 MHz) Compound **15**



^{13}C NMR (CD_3OD, 150 MHz) Compound **15**



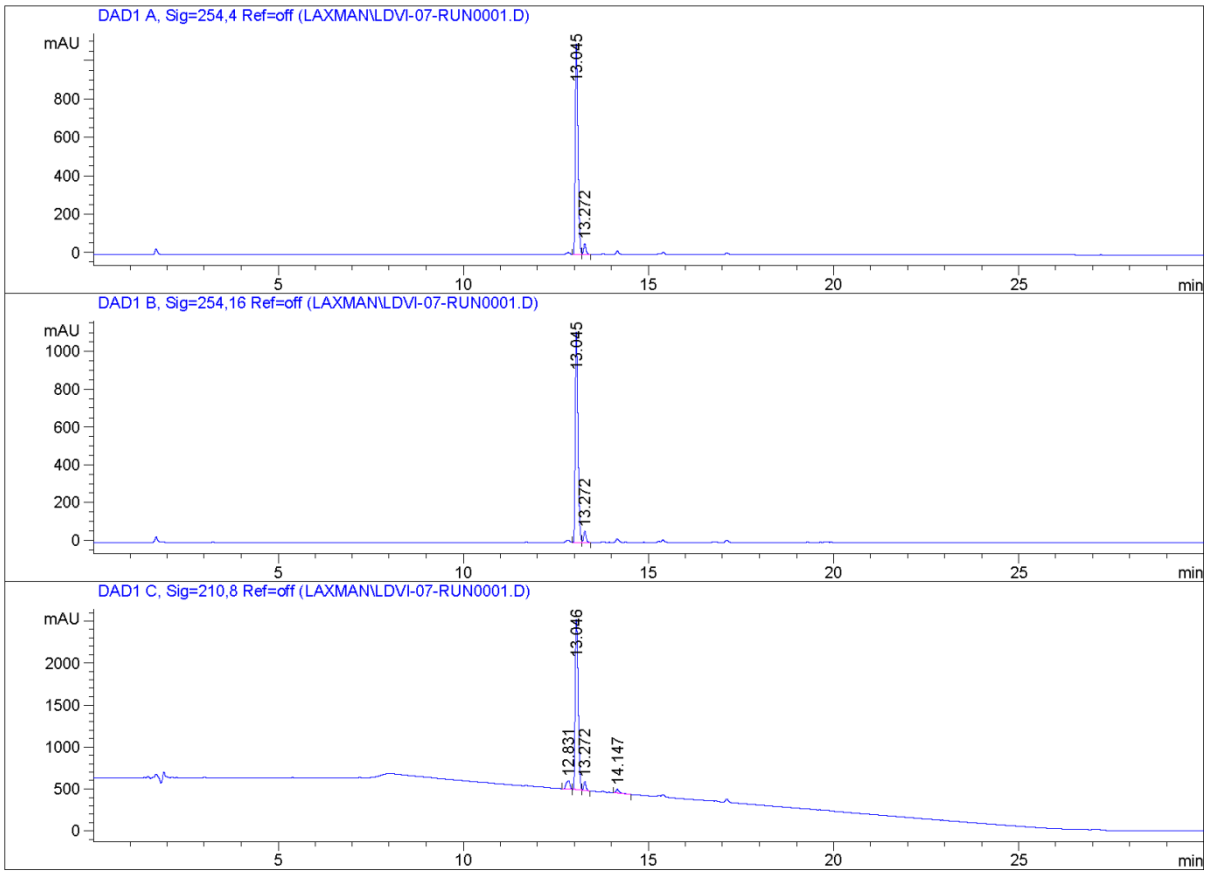
HRMS Compound **15**



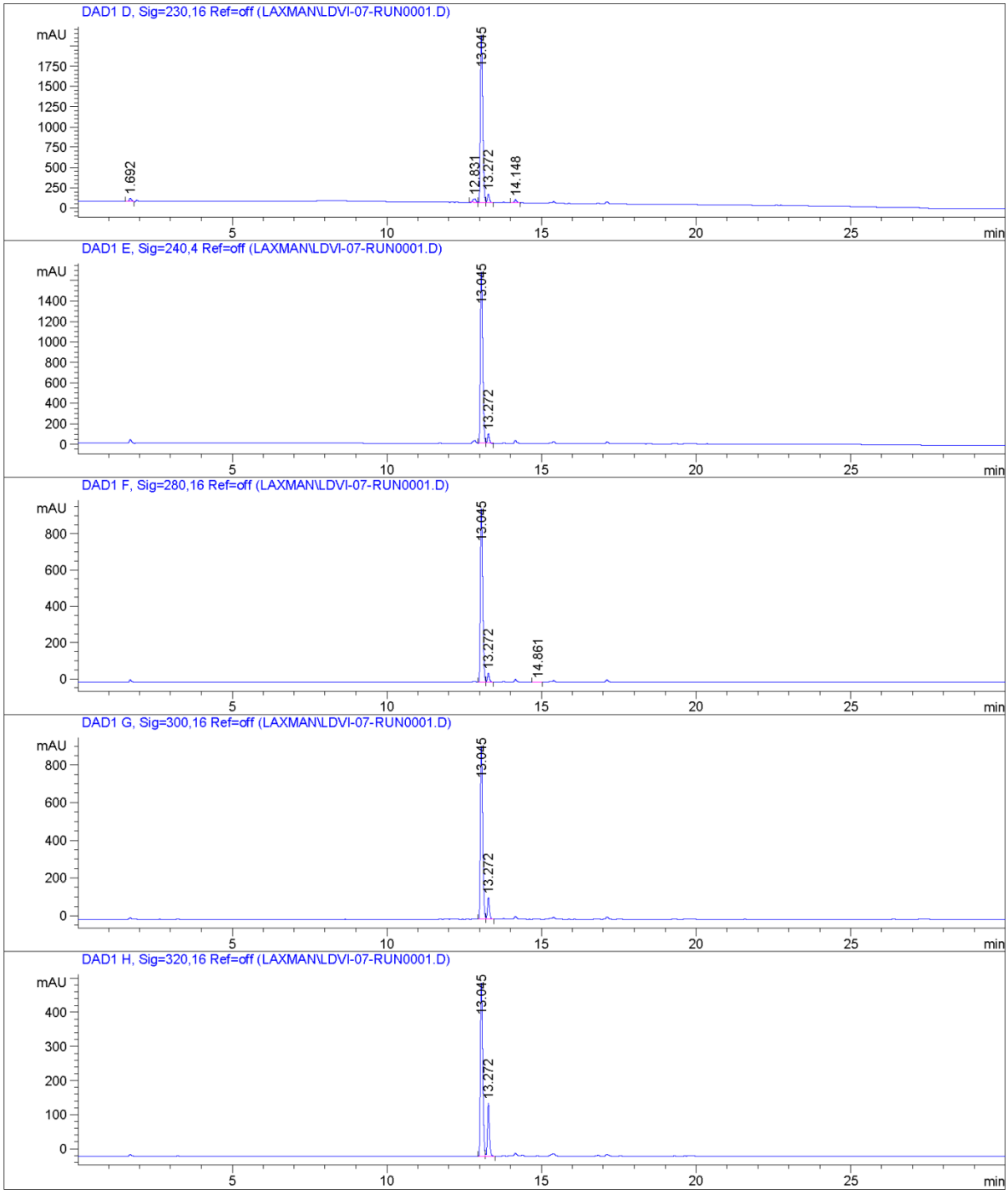
HPLC for Compound **15**

Data File C:\CHEM32\1\DATA\LAXMAN\LDVI-07-RUN0001.D
Sample Name: LD-VII-07-1A-run1

```
======================================================================
Acq. Operator   : Laxman
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 1/30/2015 11:04:00 AM
Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD2.M
Last changed    : 1/30/2015 11:00:11 AM by Laxman
Analysis Method : C:\CHEM32\1\DATA\LAXMAN\LDVI-07-RUN0001.D\DA.M (MASTERMETHOD2.M)
Last changed    : 1/30/2015 11:44:15 AM by Laxman
Sample Info     : Method-Mastermethod2.M
                  0.1% TFA/H2O
```



Data File C:\CHEM32\1\DATA\LAXMAN\LDVI-07-RUN0001.D
Sample Name: LD-VII-07-1A-run1



Data File C:\CHEM32\1\DATA\LAXMAN\LDVI-07-RUN0001.D
Sample Name: LD-VII-07-1A-run1

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.045	VV	0.0798	5641.81982	1100.64673	95.6171
2	13.272	VV	0.0679	258.60953	58.10617	4.3829
Totals :				5900.42935	1158.75290	

Signal 2: DAD1 B, Sig=254,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.045	VV	0.0798	5725.09717	1116.79260	95.6048
2	13.272	VV	0.0679	263.19711	59.14703	4.3952
Totals :				5988.29428	1175.93963	

Signal 3: DAD1 C, Sig=210,8 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.831	BB	0.1014	707.34418	101.35224	5.4677
2	13.046	BV	0.0917	1.14252e4	2030.26953	88.3164
3	13.272	VB	0.0674	483.14075	109.64965	3.7347
4	14.147	VB	0.0927	320.98795	48.82853	2.4812
Totals :				1.29367e4	2290.09996	

Data File C:\CHEM32\1\DATA\LAXMAN\LDVI-07-RUN0001.D
Sample Name: LD-VII-07-1A-run1

Signal 4: DAD1 D, Sig=230,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.692	VB	0.0868	273.36813	45.01778	2.2595
2	12.831	VV	0.1009	308.57629	43.41265	2.5505
3	13.045	VV	0.0831	1.08210e4	2067.49194	89.4404
4	13.272	VB	0.0677	469.66138	105.94901	3.8819
5	14.148	VV	0.0830	225.95723	39.32711	1.8676
Totals :				1.20986e4	2301.19848	

Signal 5: DAD1 E, Sig=240,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.045	VV	0.0800	8622.23145	1675.22180	95.2748
2	13.272	VV	0.0678	427.61954	96.27460	4.7252
Totals :				9049.85098	1771.49640	

Signal 6: DAD1 F, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.045	BV	0.0797	4888.63330	955.13538	95.3672
2	13.272	VB	0.0677	227.55379	51.38308	4.4391
3	14.861	BB	0.0931	9.93156	1.54229	0.1937
Totals :				5126.11865	1008.06074	

Signal 7: DAD1 G, Sig=300,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.045	BV	0.0799	4717.08740	919.34308	90.2095
2	13.272	VB	0.0672	511.94711	116.60598	9.7905
Totals :				5229.03452	1035.94906	

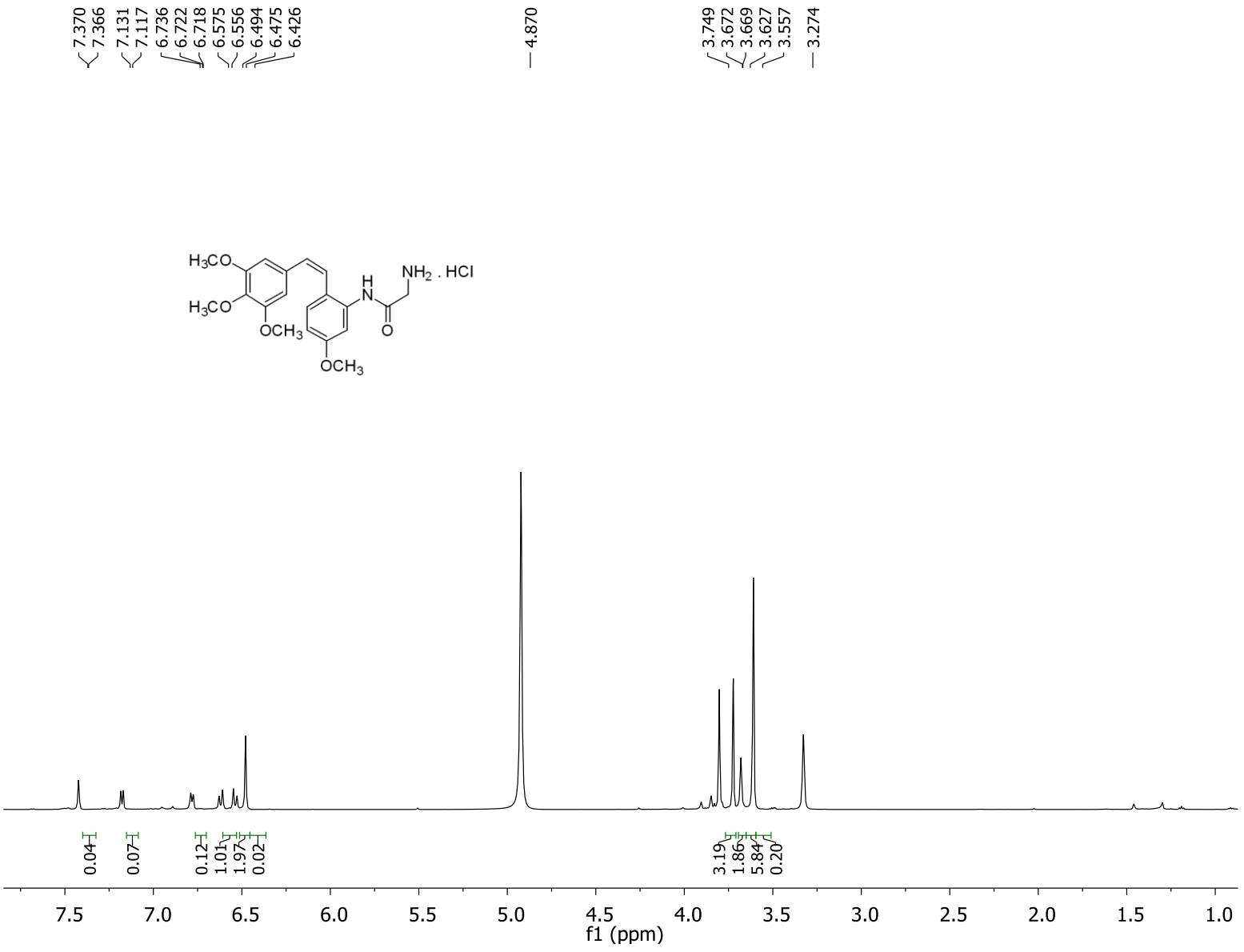
Data File C:\CHEM32\1\DATA\LAXMAN\LDVI-07-RUN0001.D
Sample Name: LD-VII-07-1A-run1

Signal 8: DAD1 H, Sig=320,16 Ref=off

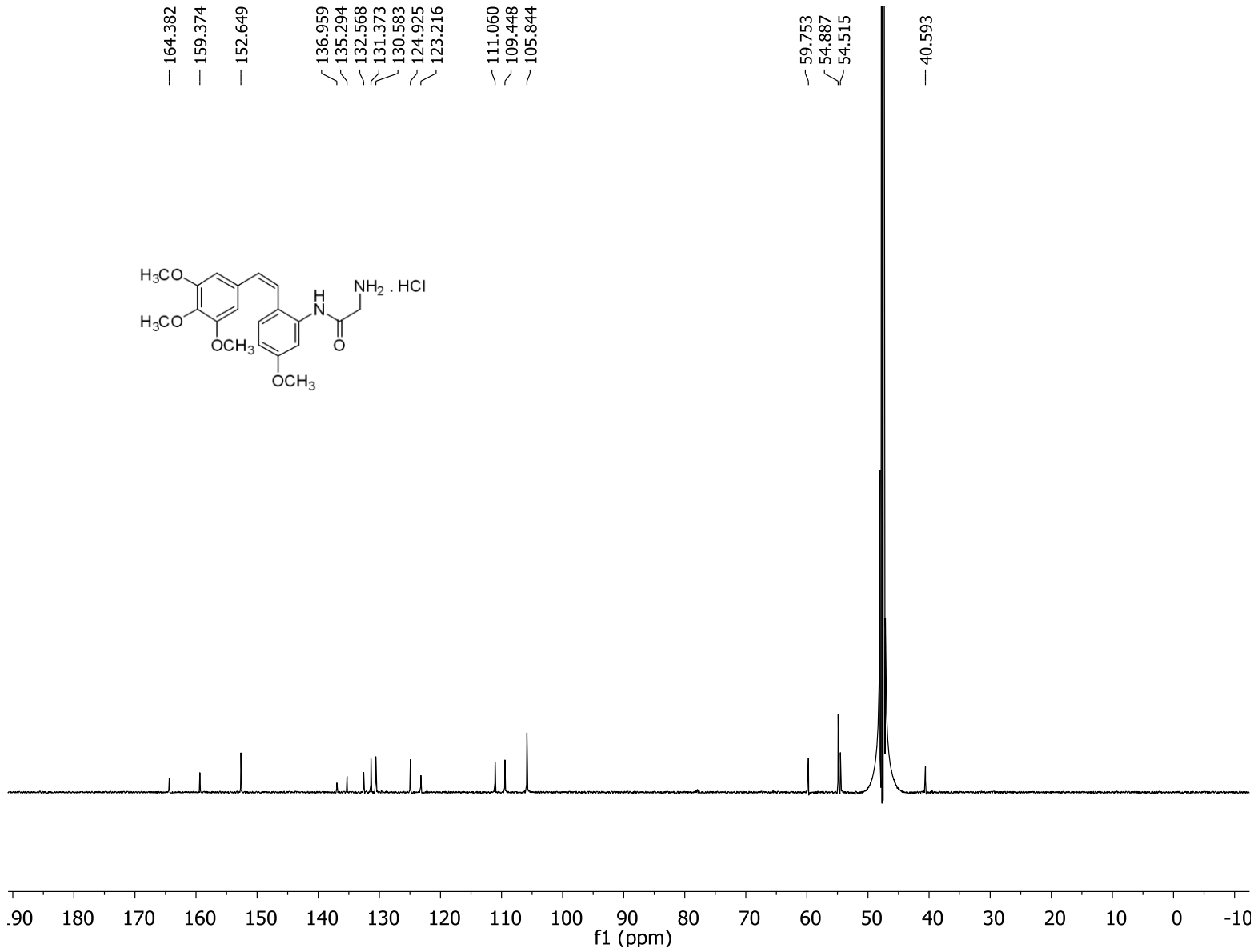
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.045	BV	0.0801	2620.03906	508.46661	79.2579
2	13.272	VB	0.0670	685.67603	156.98068	20.7421
Totals :				3305.71509	665.44730	

*** End of Report ***

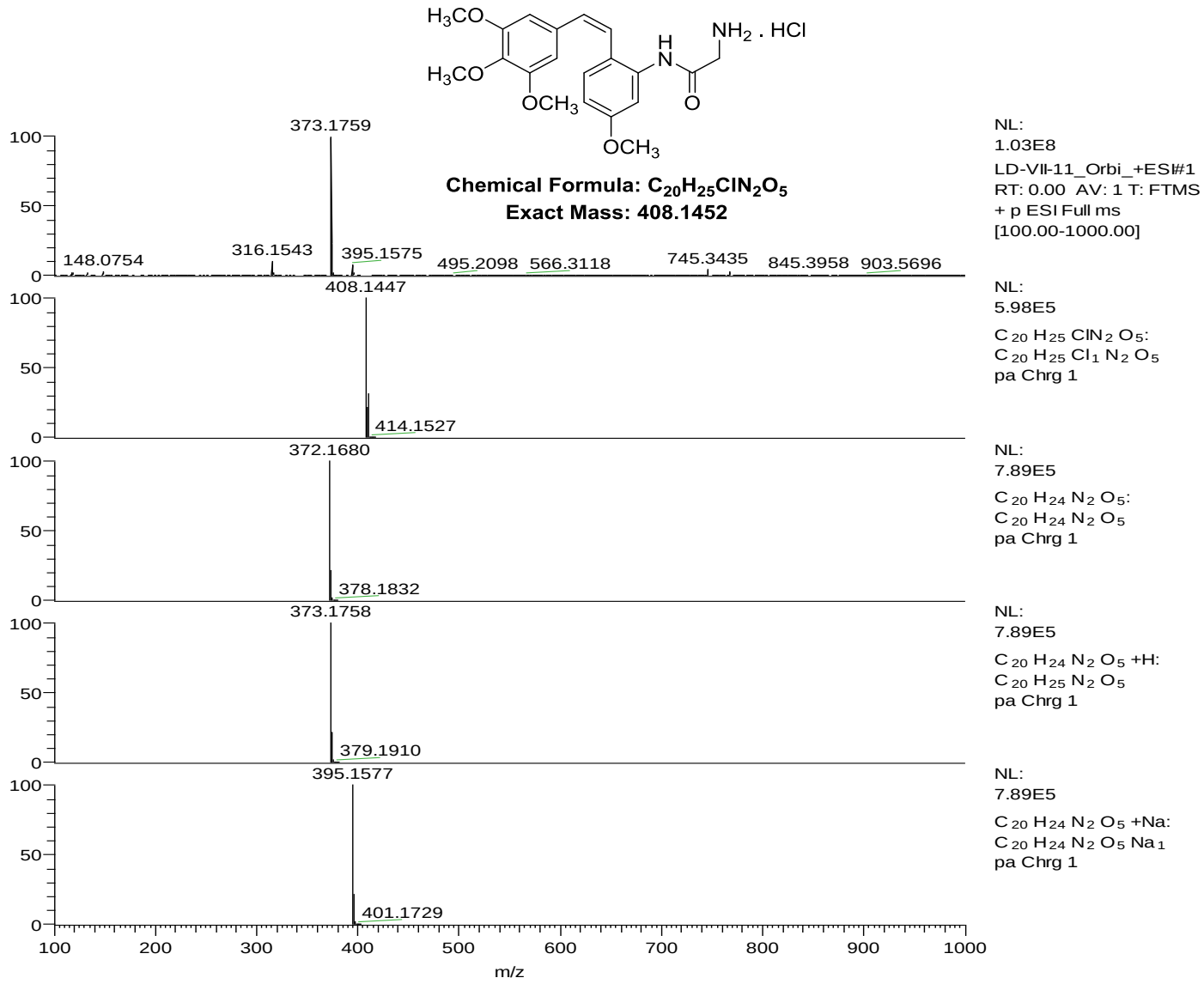
^{1}H NMR (CD_3OD, 600 MHz) Compound **16**



^{13}C NMR (CD_3OD, 150 MHz) Compound **16**



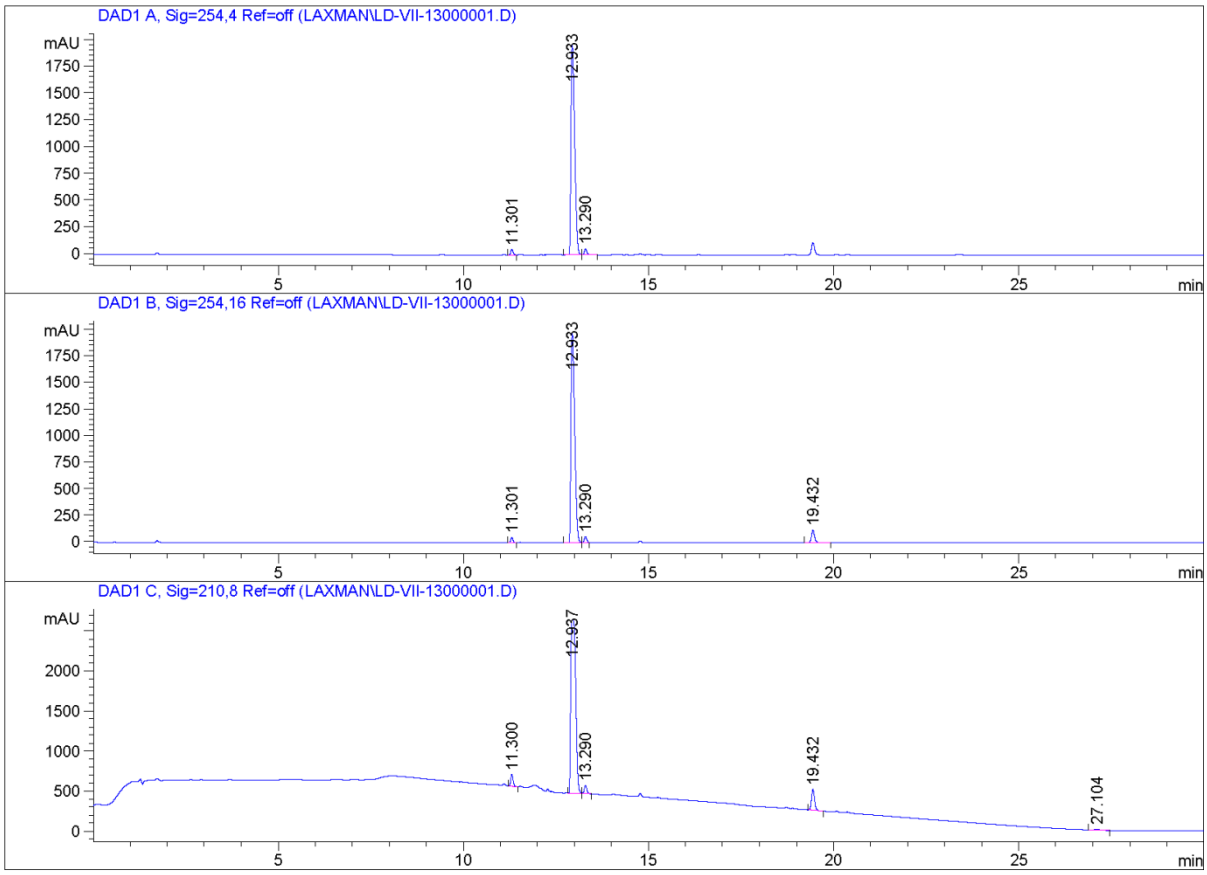
HRMS Compound **16**



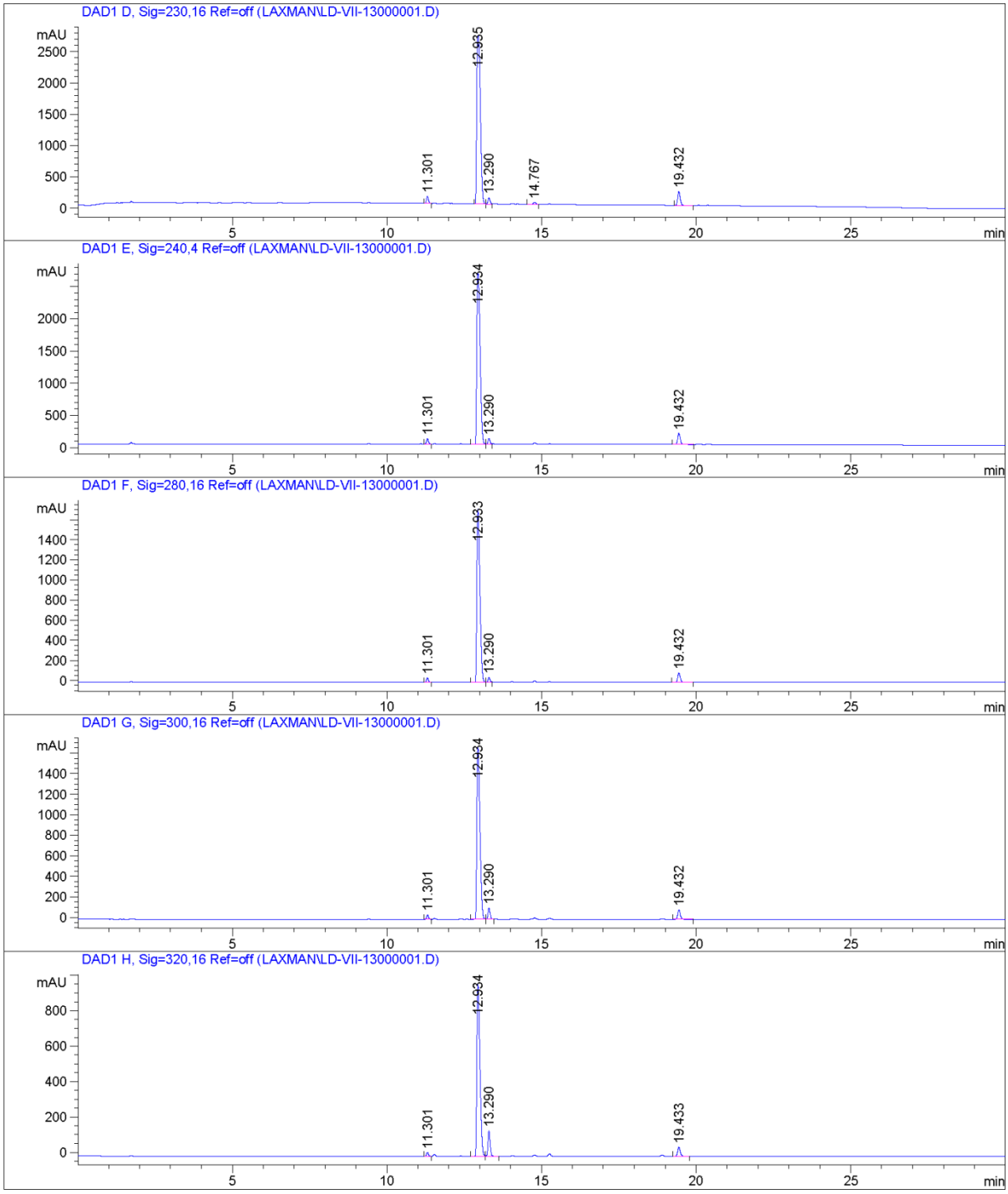
HPLC for Compound **16**

Data File C:\CHEM32\1\DATA\LAXMAN\LD-VII-13000001.D
Sample Name: LD-VII-13-1A-run1

```
======================================================================
Acq. Operator   : Laxman
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 1/30/2015 2:24:28 PM
Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD2.M
Last changed    : 1/30/2015 2:22:10 PM by Laxman
Analysis Method : C:\CHEM32\1\DATA\LAXMAN\LD-VII-13000001.D\DA.M (MASTERMETHOD2.M)
Last changed    : 2/3/2015 10:01:31 AM by Laxman
Sample Info     : Method-Mastermethod2.M
```



Data File C:\CHEM32\1\DATA\LAXMAN\LD-VII-13000001.D
Sample Name: LD-VII-13-1A-run1



Data File C:\CHEM32\1\DATA\LAXMAN\LD-VII-13000001.D
Sample Name: LD-VII-13-1A-run1

==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.301	VV	0.0688	216.40208	49.69061	1.5422
2	12.933	BV	0.1082	1.35325e4	1968.75415	96.4400
3	13.290	VB	0.0767	283.14041	56.29315	2.0178
Totals :				1.40320e4	2074.73792	

Signal 2: DAD1 B, Sig=254,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.301	VV	0.0688	220.70474	50.70074	1.4896
2	12.933	BB	0.1083	1.36721e4	1986.53516	92.2783
3	13.290	BV	0.0707	249.83946	55.35923	1.6863
4	19.432	BB	0.0870	673.50708	117.34465	4.5458
Totals :				1.48161e4	2209.93979	

Signal 3: DAD1 C, Sig=210,8 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.300	BV	0.0709	674.63080	148.83742	2.9450
2	12.937	VV	0.1521	2.01127e4	2169.80444	87.7994
3	13.290	VB	0.0728	479.12888	102.04993	2.0916
4	19.432	BB	0.0865	1503.00769	263.54004	6.5612
5	27.104	BBA	0.2629	138.09706	8.43884	0.6028
Totals :				2.29076e4	2692.67067	

Data File C:\CHEM32\1\DATA\LAXMAN\LD-VII-13000001.D
Sample Name: LD-VII-13-1A-run1

Signal 4: DAD1 D, Sig=230,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.301	VB	0.0686	494.89111	114.18317	2.0390
2	12.935	VV	0.1305	2.18511e4	2696.80225	90.0305
3	13.290	VV	0.0733	470.43481	99.17249	1.9383
4	14.767	BB	0.0844	173.55266	31.42022	0.7151
5	19.432	BB	0.0869	1280.80042	223.38777	5.2771
Totals :				2.42708e4	3164.96590	

Signal 5: DAD1 E, Sig=240,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.301	VV	0.0687	374.85321	86.20531	1.7492
2	12.934	BB	0.1178	1.96632e4	2673.26465	91.7548
3	13.290	BV	0.0707	396.78284	87.91900	1.8515
4	19.432	BB	0.0869	995.31000	173.60373	4.6444
Totals :				2.14301e4	3020.99268	

Signal 6: DAD1 F, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.301	VV	0.0689	198.91370	45.63385	1.5660
2	12.933	BB	0.1075	1.17454e4	1722.68103	92.4673
3	13.290	BV	0.0707	217.02390	48.02873	1.7085
4	19.432	BB	0.0868	540.88361	94.40138	4.2582
Totals :				1.27023e4	1910.74500	

Signal 7: DAD1 G, Sig=300,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.301	VV	0.0693	190.55530	43.33076	1.4858
2	12.934	BV	0.1082	1.15952e4	1687.50012	90.4100
3	13.290	VV	0.0725	517.15173	110.68778	4.0323
4	19.432	BB	0.0868	522.22614	91.22451	4.0719

Data File C:\CHEM32\1\DATA\LAXMAN\LD-VII-13000001.D
Sample Name: LD-VII-13-1A-run1

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
Totals :				1.28252e4	1932.74317	

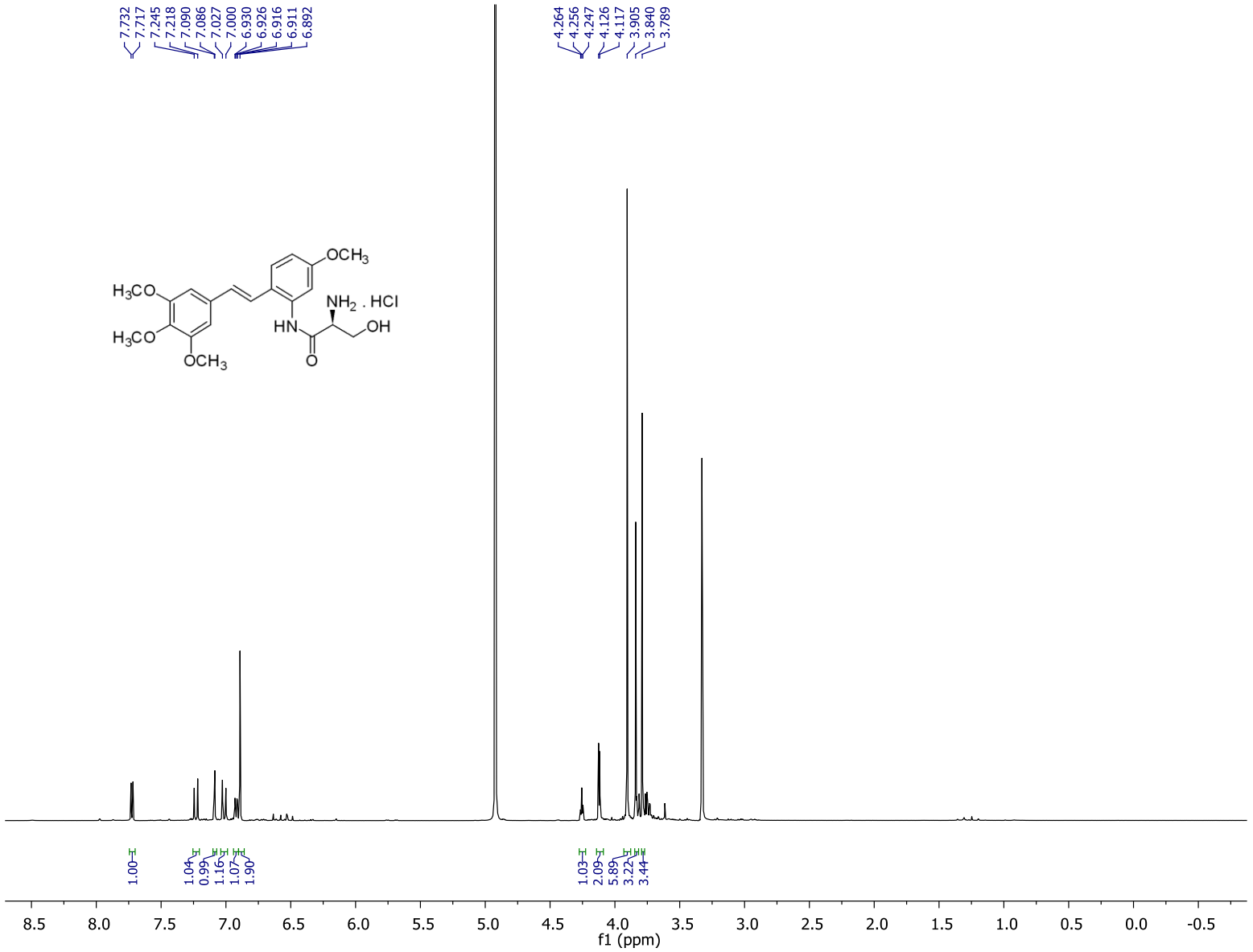
Signal 8: DAD1 H, Sig=320,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.301	BV	0.0690	96.04256	21.98625	1.2361
2	12.934	BV	0.1078	6687.89258	978.16895	86.0774
3	13.290	VB	0.0727	684.77283	146.05211	8.8135
4	19.433	BB	0.0867	300.91791	52.62127	3.8730
Totals :				7769.62588	1198.82858	

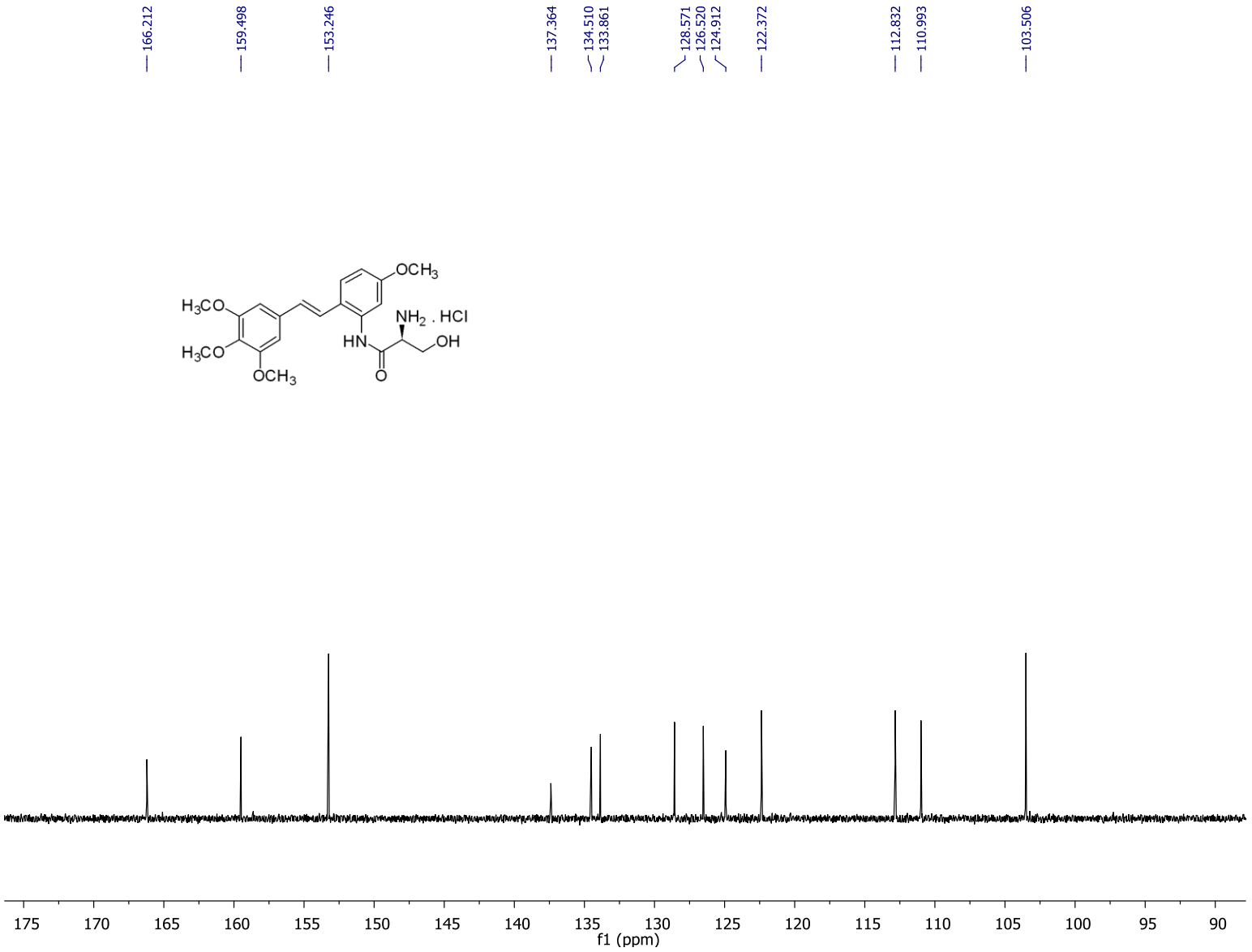
==========

*** End of Report ***

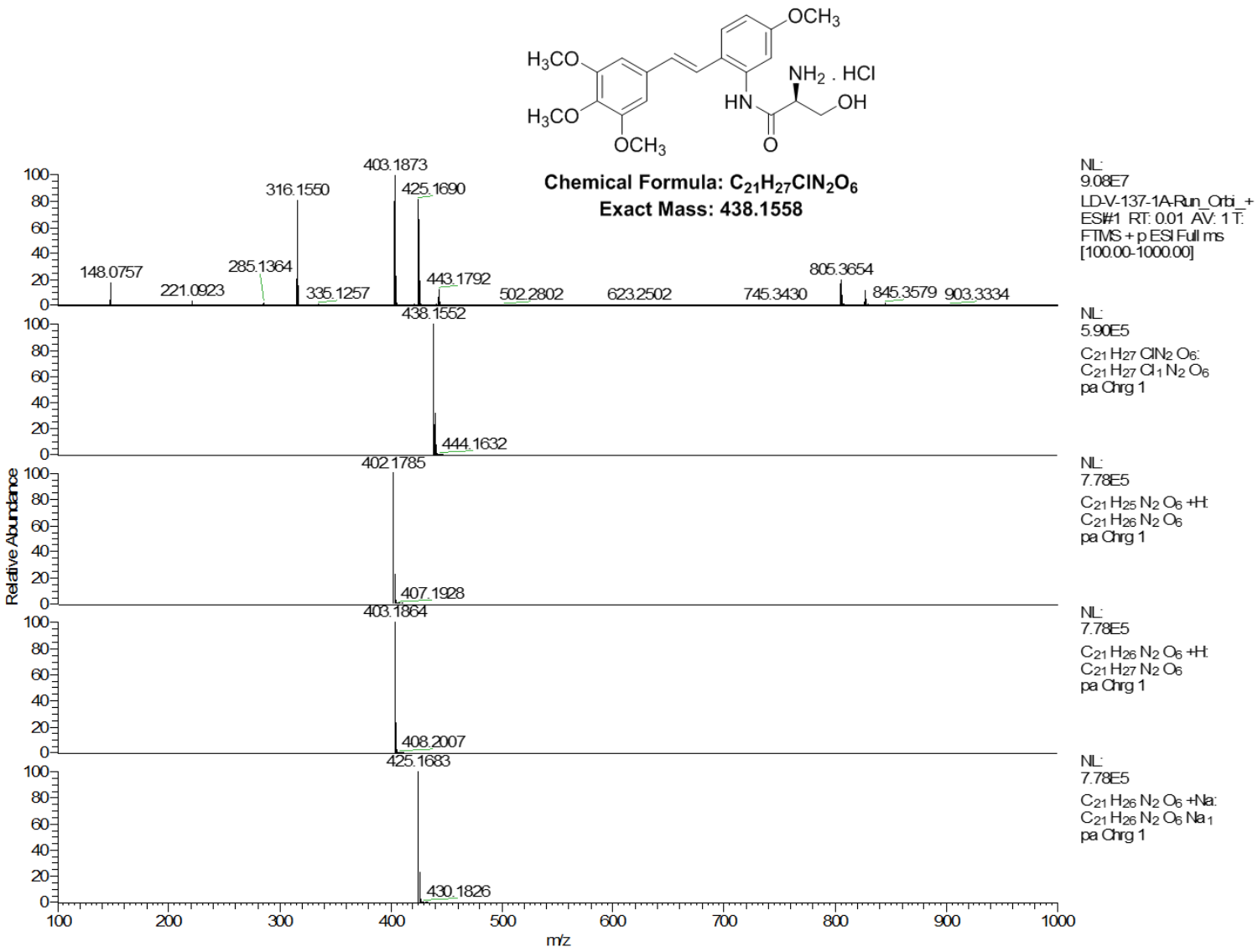
^{1}H NMR (CD_3OD, 600 MHz) Compound **17**



^{13}C NMR (CD_3OD, 150 MHz) Compound **17**



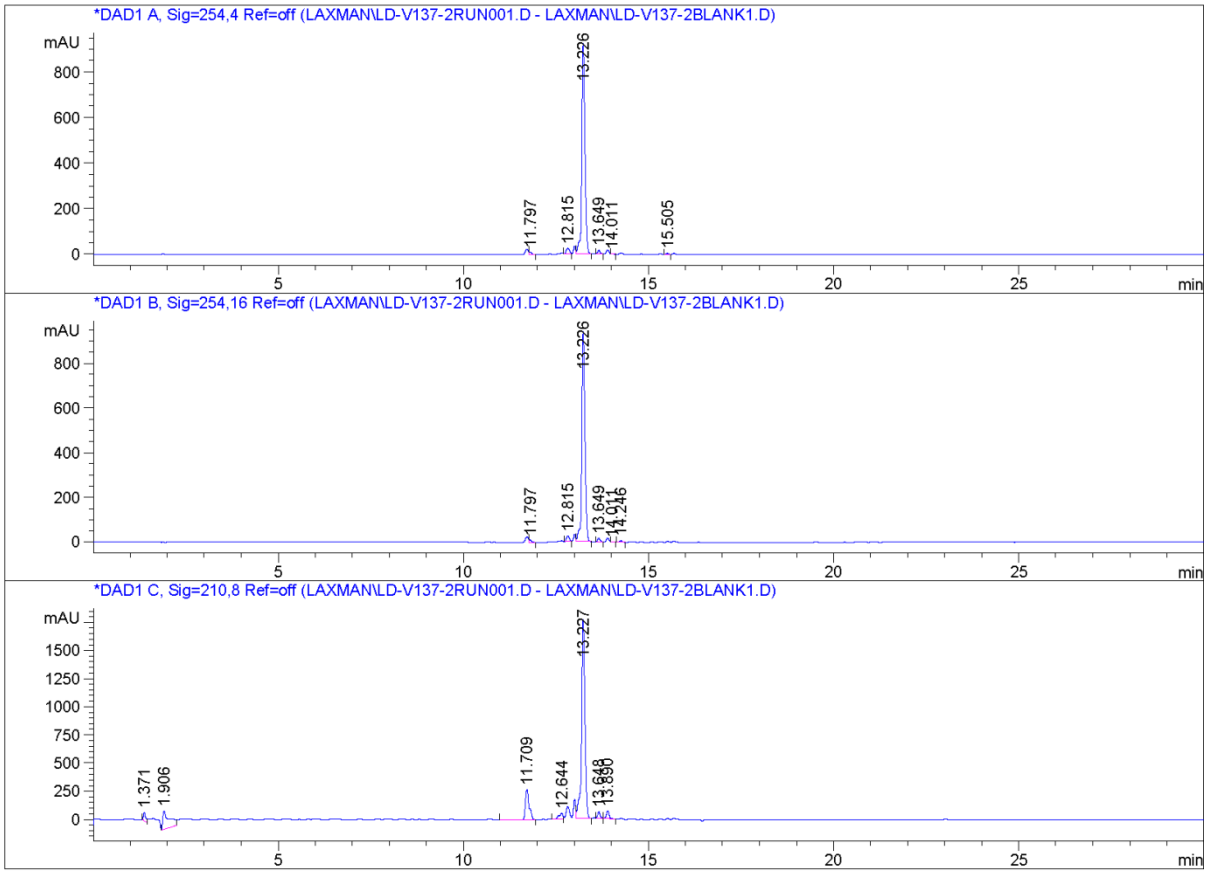
HRMS Compound **17**



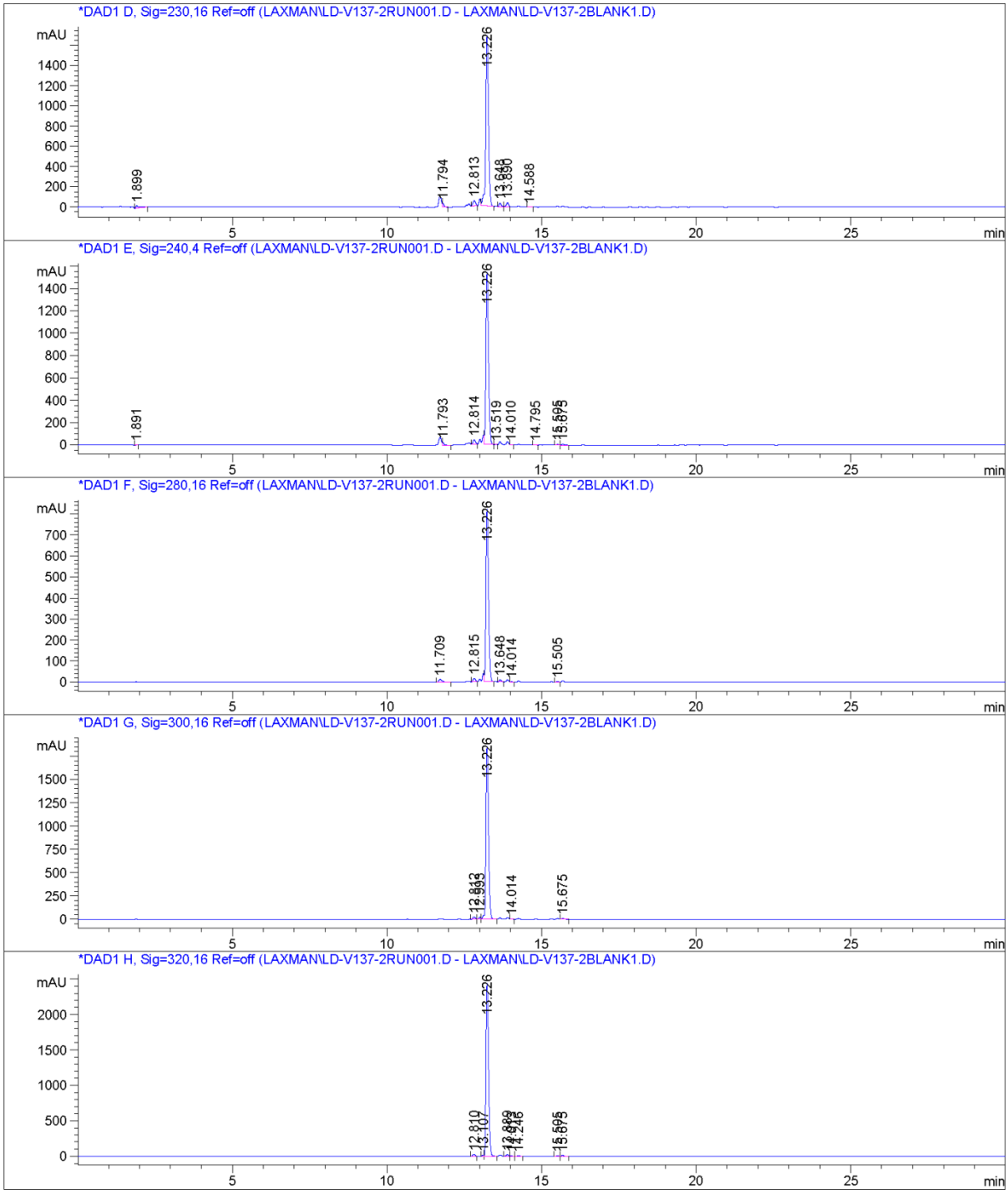
HPLC for Compound **17**

Data File C:\CHEM32\1\DATA\LAXMAN\LD-V137-2RUN001.D
Sample Name: LD-V-137-2-run1

```
======================================================================
Acq. Operator   : Laxman
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 7/14/2014 10:03:32 PM
Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD2.M
Last changed    : 7/14/2014 9:54:41 PM by Laxman
Analysis Method : C:\CHEM32\1\DATA\LAXMAN\LD-V137-2RUN001.D\DA.M (MASTERMETHOD2.M)
Last changed    : 7/14/2014 10:43:46 PM by Laxman
Sample Info     : Mastermethod2
                  0-5 min 10:90  ACN: 0.1% TFA in Water
                  5-25 min 10:90 to 100:0 ACN: 0.1% TFA in Water
                  25-30 min 100:0 ACN: 0.1% TFA in Water
```



Data File C:\CHEM32\1\DATA\LAXMAN\LD-V137-2RUN001.D
Sample Name: LD-V-137-2-run1



Data File C:\CHEM32\1\DATA\LAXMAN\LD-V137-2RUN001.D
Sample Name: LD-V-137-2-run1

==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.797	VB	0.0571	37.39809	10.12354	0.6524
2	12.815	BB	0.0943	141.96701	24.23329	2.4765
3	13.226	VB	0.0908	5449.38037	924.33972	95.0589
4	13.649	BB	0.0717	75.91022	16.47810	1.3242
5	14.011	VB	0.0582	5.78434	1.52517	0.1009
6	15.505	BV	0.0677	22.19742	5.01273	0.3872
Totals :				5732.63745	981.71255	

Signal 2: DAD1 B, Sig=254,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.797	VB	0.0543	37.51292	10.34681	0.6424
2	12.815	BB	0.0946	141.00084	23.99567	2.4146
3	13.226	VB	0.0907	5548.84961	942.08569	95.0223
4	13.649	BB	0.0717	74.49856	16.18236	1.2758
5	14.011	VB	0.0583	5.93264	1.56384	0.1016
6	14.246	BB	0.0727	31.73079	6.76175	0.5434
Totals :				5839.52536	1000.93613	

Signal 3: DAD1 C, Sig=210,8 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.371	BV	0.0676	340.71149	74.14693	2.0426
2	1.906	BV	0.1686	2097.38916	164.71938	12.5738
3	11.709	BB	0.1048	1859.09814	268.37698	11.1453

Data File C:\CHEM32\1\DATA\LAXMAN\LD-V137-2RUN001.D
Sample Name: LD-V-137-2-run1

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
4	12.644	BV	0.1103	423.80072	53.58047	2.5407
5	13.227	VB	0.0960	1.12451e4	1772.58142	67.4141
6	13.648	VV	0.0770	315.91071	62.49385	1.8939
7	13.890	VB	0.0834	398.60931	73.31020	2.3897
Totals :				1.66806e4	2469.20923	

Signal 4: DAD1 D, Sig=230,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.899	BB	0.1205	206.66499	23.52119	1.8437
2	11.794	VB	0.0507	132.52130	39.94218	1.1822
3	12.813	BB	0.0956	323.16006	54.13917	2.8830
4	13.226	VB	0.0918	1.01466e4	1696.98718	90.5198
5	13.648	VV	0.0736	178.25475	37.39213	1.5902
6	13.890	VV	0.0767	213.33083	42.40094	1.9032
7	14.588	VV	0.1051	8.73081	1.28800	0.0779
Totals :				1.12093e4	1895.67081	

Signal 5: DAD1 E, Sig=240,4 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.891	BB	0.0504	19.58302	6.28240	0.2135
2	11.793	VB	0.0534	76.00732	23.73110	0.8287
3	12.814	BB	0.0957	236.46078	39.57375	2.5782
4	13.226	VB	0.0880	8730.77148	1542.62524	95.1954
5	13.519	BV	0.0590	6.40798	1.82516	0.0699
6	14.010	VB	0.0571	10.10926	2.73744	0.1102
7	14.795	VB	0.0677	9.97771	2.34226	0.1088
8	15.505	VV	0.0689	35.71164	7.87916	0.3894
9	15.675	VB	0.0800	46.39381	8.72317	0.5059
Totals :				9171.42301	1635.71969	

Data File C:\CHEM32\1\DATA\LAXMAN\LD-V137-2RUN001.D
Sample Name: LD-V-137-2-run1

Signal 6: DAD1 F, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.709	BB	0.1042	97.78605	14.22911	2.0067
2	12.815	BB	0.0938	97.29163	16.73797	1.9965
3	13.226	VB	0.0873	4604.29199	823.04236	94.4843
4	13.648	BB	0.0710	46.71126	10.28558	0.9586
5	14.014	VB	0.0629	6.04078	1.50296	0.1240
6	15.505	VV	0.0699	20.95428	4.53886	0.4300
Totals :				4873.07599	870.33683	

Signal 7: DAD1 G, Sig=300,16 Ref=off
Signal has been modified after loading from rawdata file!

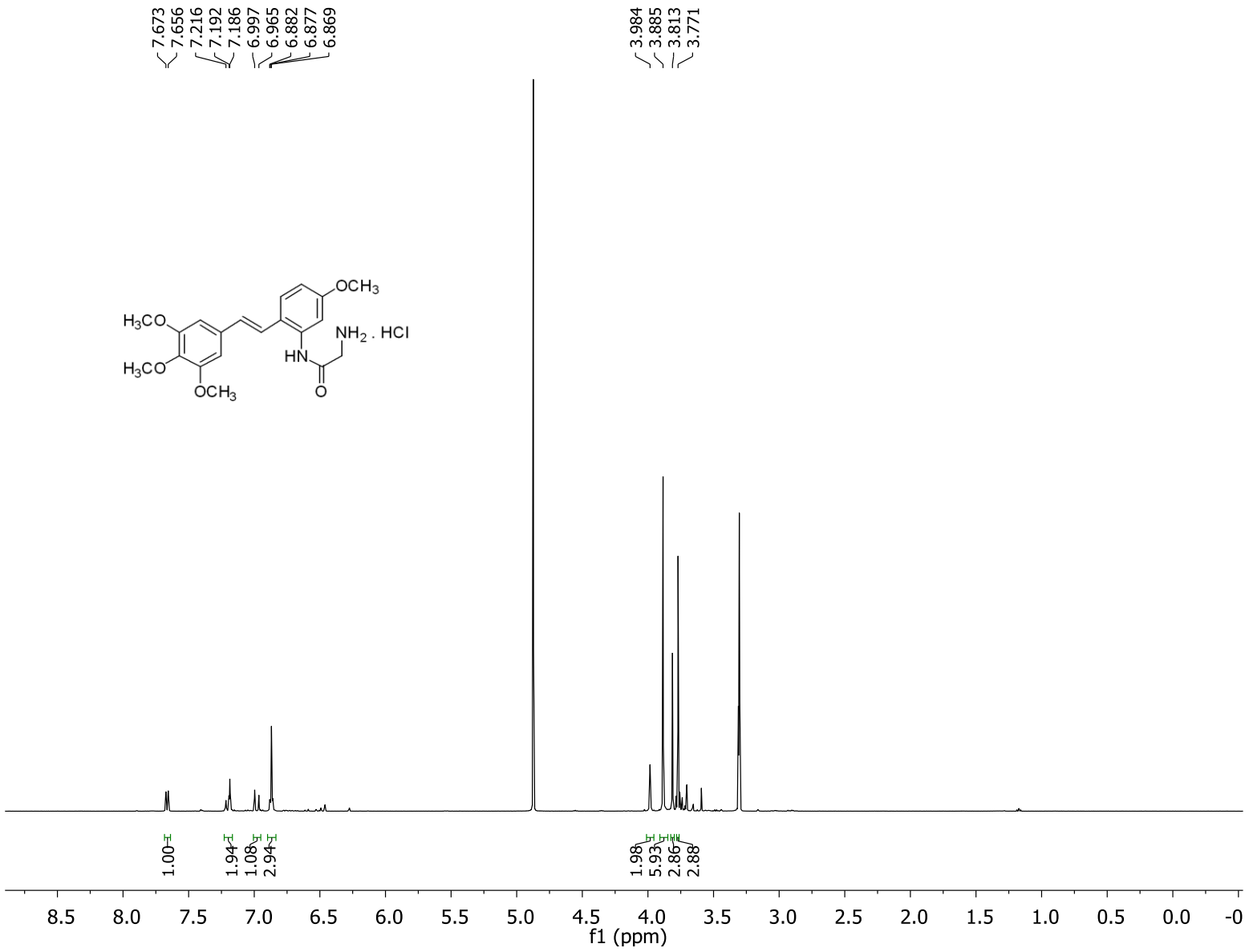
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.812	BB	0.0888	138.41168	25.71216	1.2652
2	12.993	BV	0.0620	88.38454	22.39664	0.8079
3	13.226	VB	0.0887	1.06324e4	1859.13770	97.1902
4	14.014	VB	0.0646	15.00118	3.60392	0.1371
5	15.675	VB	0.0780	65.59080	12.75470	0.5996
Totals :				1.09398e4	1923.60512	

Signal 8: DAD1 H, Sig=320,16 Ref=off
Signal has been modified after loading from rawdata file!

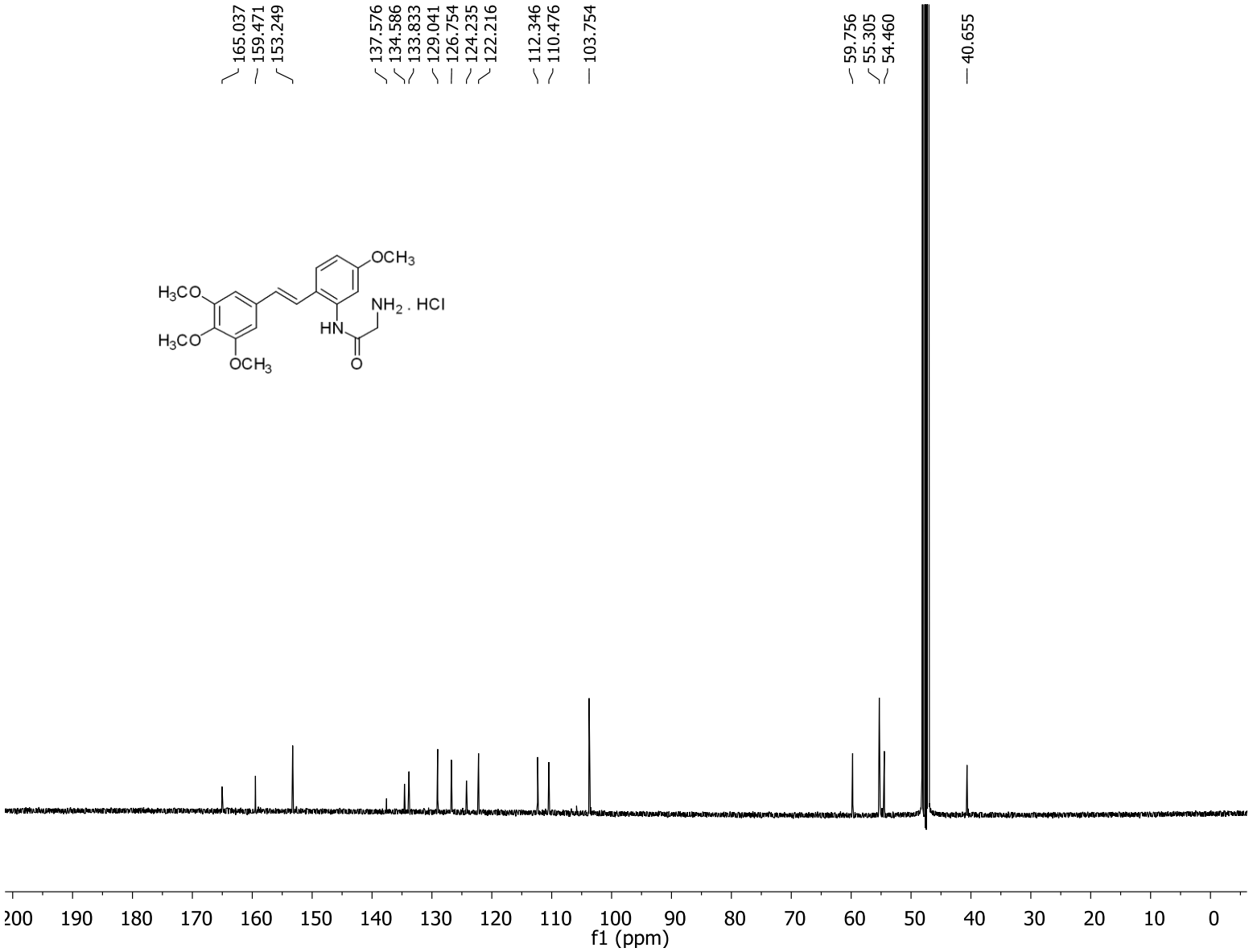
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.810	BB	0.0877	154.40775	29.18997	1.0733
2	13.107	BV	0.0546	66.88392	19.22609	0.4649
3	13.226	VB	0.0901	1.38478e4	2443.69946	96.2531
4	13.889	BV	0.0742	109.93297	22.84040	0.7641
5	14.013	VB	0.0647	19.88695	4.76055	0.1382
6	14.246	BB	0.0723	54.22525	11.65682	0.3769
7	15.505	VV	0.0698	57.71371	12.52080	0.4012
8	15.675	VB	0.0779	76.01604	14.81508	0.5284
Totals :				1.43869e4	2558.70918	

==========

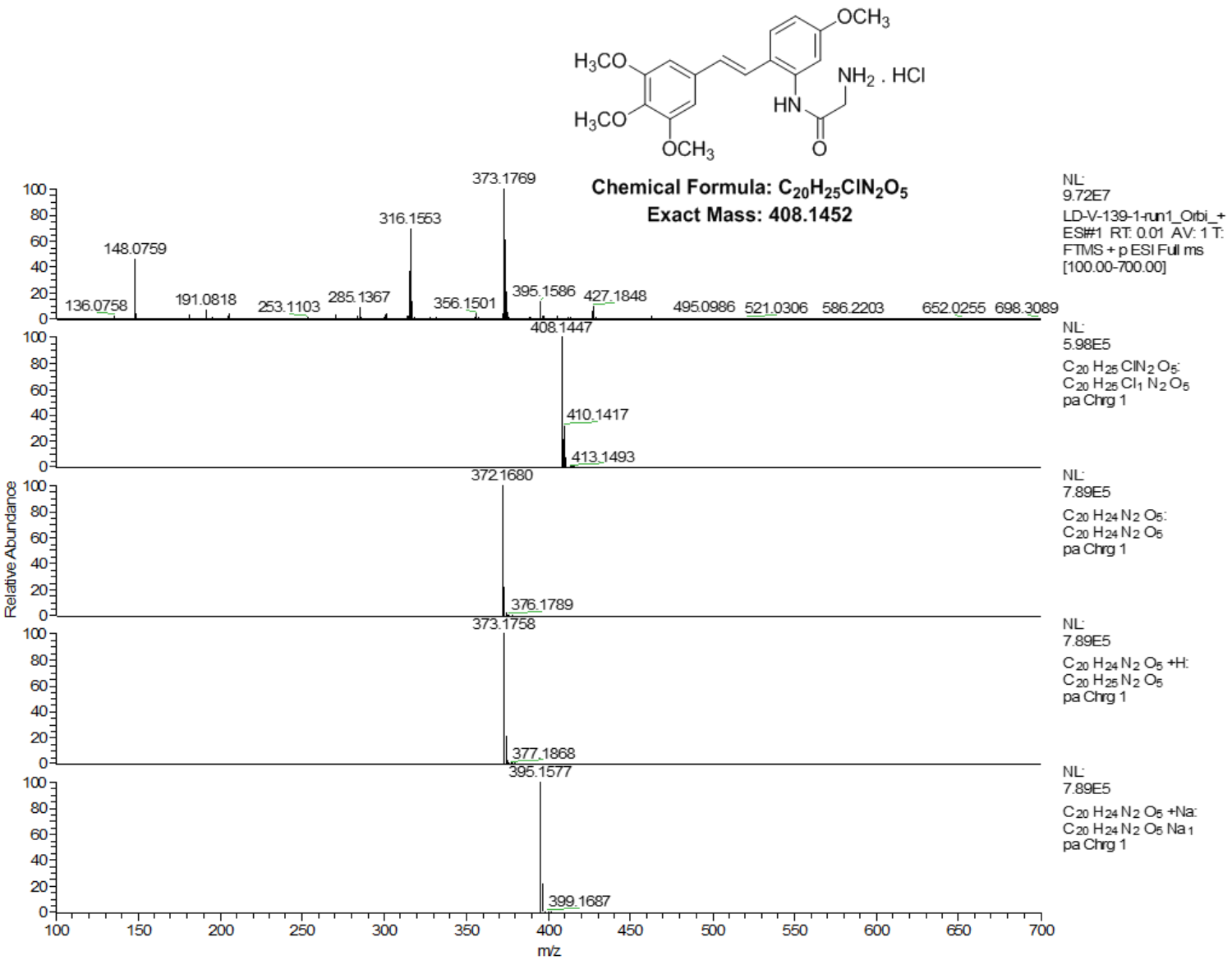
^{1}H NMR (CD_3OD, 500 MHz) Compound **18**



^{13}C NMR (CD_3OD, 125 MHz) Compound **18**



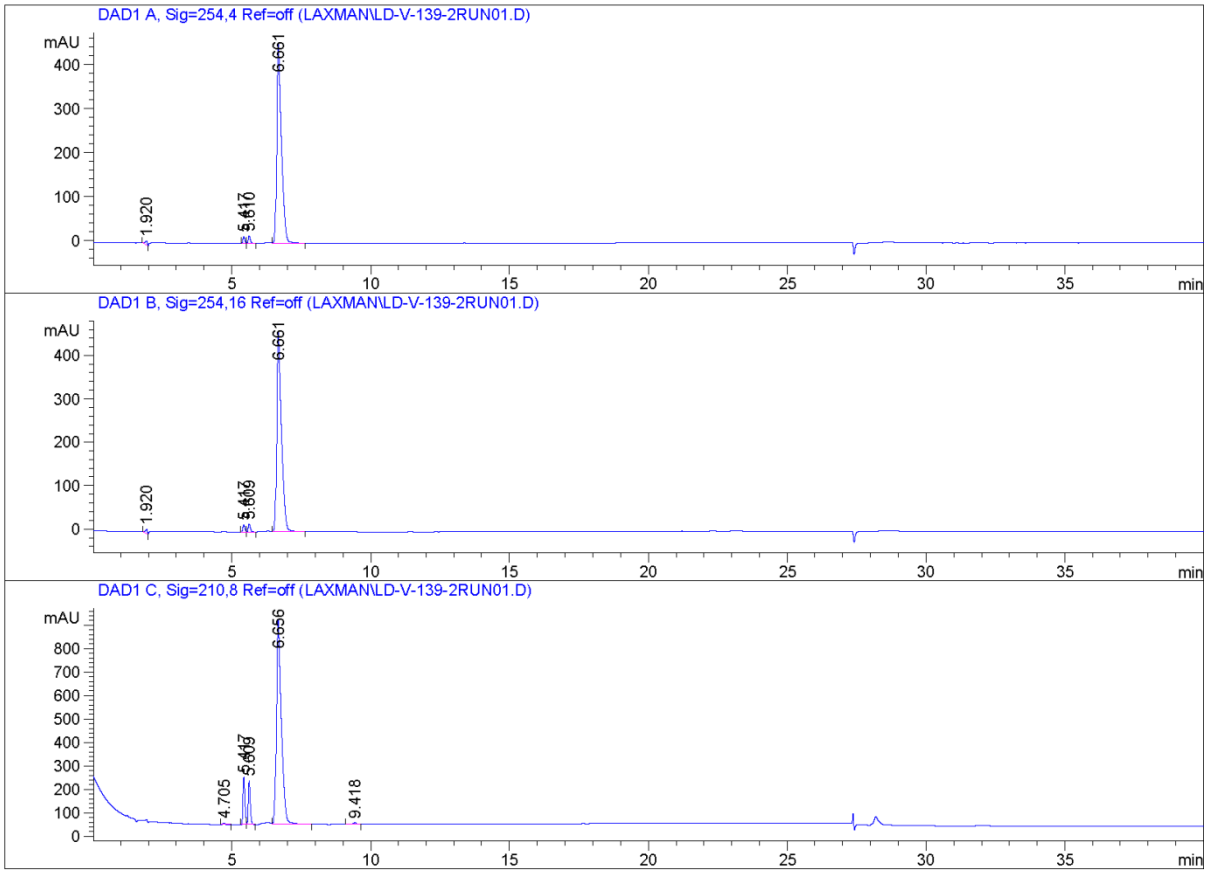
HRMS Compound **18**



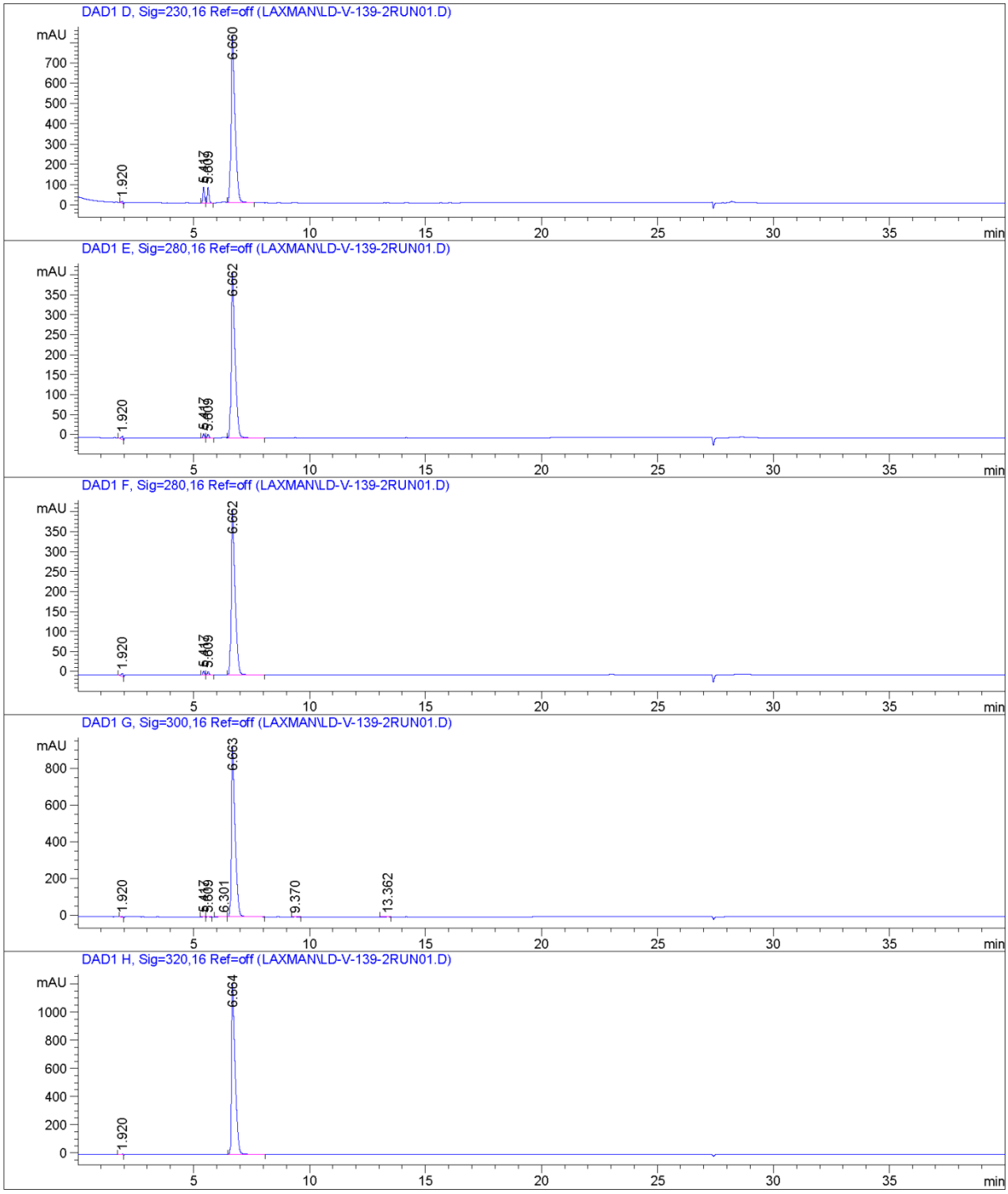
HPLC for Compound **18**

Data File C:\CHEM32\1\DATA\LAXMAN\LD-V-139-2RUN01.D
Sample Name: LD-V-139-2run1

```
======================================================================
Acq. Operator   : Laxman
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 7/13/2014 12:11:09 PM
Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed    : 7/13/2014 12:04:39 PM by Laxman
Analysis Method : C:\CHEM32\1\DATA\LAXMAN\LD-V-139-2RUN01.D\DA.M (MASTERMETHOD.M)
Last changed    : 7/13/2014 1:05:46 PM by Laxman
Sample Info     : Method
                  Mastermethod.M
```



Data File C:\CHEM32\1\DATA\LAXMAN\LD-V-139-2RUN01.D
Sample Name: LD-V-139-2run1



Data File C:\CHEM32\1\DATA\LAXMAN\LD-V-139-2RUN01.D
Sample Name: LD-V-139-2run1

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.920	BB	0.0775	46.97380	9.52554	0.8652
2	5.417	BV	0.0785	73.83537	14.72783	1.3600
3	5.610	VB	0.0783	87.99896	17.59993	1.6209
4	6.661	VB	0.1616	5220.14502	455.76682	96.1538
Totals :				5428.95316	497.62012	

Signal 2: DAD1 B, Sig=254,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.920	BB	0.0764	44.75594	9.26610	0.8119
2	5.417	BV	0.0785	80.30719	16.00931	1.4569
3	5.609	VB	0.0784	90.60959	18.10985	1.6438
4	6.661	VB	0.1615	5296.55029	462.70700	96.0874
Totals :				5512.22301	506.09225	

Signal 3: DAD1 C, Sig=210,8 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.705	BB	0.0736	31.72051	6.42426	0.2502
2	5.417	BV	0.0786	1011.34918	201.44394	7.9766
3	5.609	VB	0.0784	932.56183	186.28250	7.3552
4	6.656	VB	0.1680	1.06542e4	875.27972	84.0301
5	9.418	BB	0.1088	49.18766	6.46056	0.3879
Totals :				1.26790e4	1275.89098	

Data File C:\CHEM32\1\DATA\LAXMAN\LD-V-139-2RUN01.D
Sample Name: LD-V-139-2run1

Signal 4: DAD1 D, Sig=230,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.920	BB	0.0702	44.30437	10.30551	0.4233
2	5.417	BV	0.0786	388.58575	77.37257	3.7130
3	5.609	VB	0.0784	381.26849	76.18825	3.6430
4	6.660	VB	0.1644	9651.52832	825.70642	92.2207
Totals :				1.04657e4	989.57275	

Signal 5: DAD1 E, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.920	BB	0.0868	42.78221	7.47017	0.8821
2	5.417	BV	0.0786	51.21740	10.19095	1.0560
3	5.609	VB	0.0786	44.75695	8.91765	0.9228
4	6.662	VB	0.1583	4711.51318	415.42648	97.1392
Totals :				4850.26974	442.00525	

Signal 6: DAD1 F, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.920	BB	0.0868	42.78221	7.47017	0.8821
2	5.417	BV	0.0786	51.21740	10.19095	1.0560
3	5.609	VB	0.0786	44.75695	8.91765	0.9228
4	6.662	VB	0.1583	4711.51318	415.42648	97.1392
Totals :				4850.26974	442.00525	

Signal 7: DAD1 G, Sig=300,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.920	BB	0.0774	26.29604	5.34806	0.2568
2	5.417	BV	0.0789	11.63756	2.30688	0.1137
3	5.609	VB	0.0787	10.18231	2.02301	0.0995
4	6.301	BV	0.2271	25.72116	1.55393	0.2512
5	6.663	VB	0.1552	1.01509e4	930.72180	99.1451

Data File C:\CHEM32\1\DATA\LAXMAN\LD-V-139-2RUN01.D
Sample Name: LD-V-139-2run1

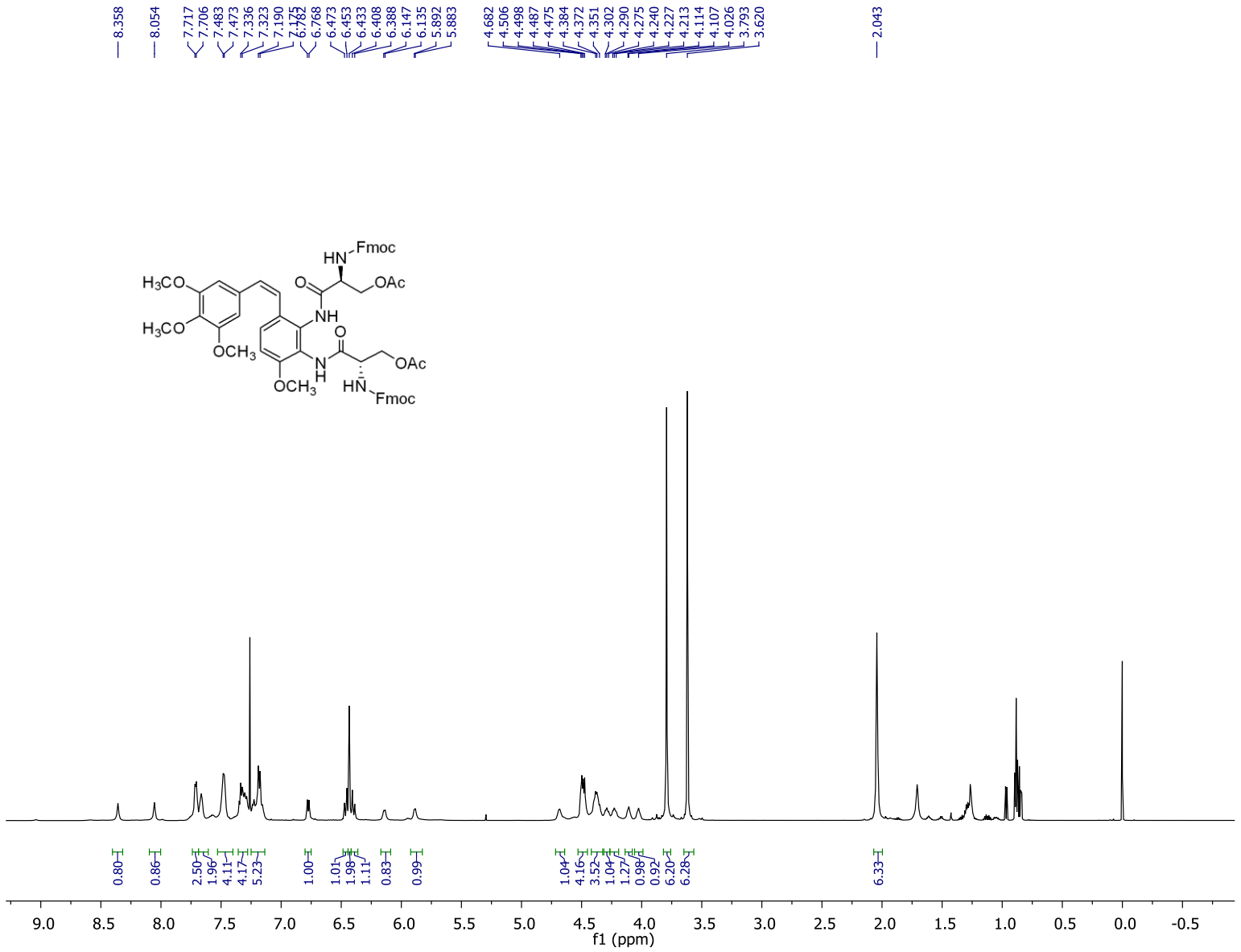
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
6	9.370	BB	0.0844	5.83878	1.05772	0.0570
7	13.362	BB	0.1049	7.84864	1.07818	0.0767
Totals :				1.02384e4	944.08958	

Signal 8: DAD1 H, Sig=320,16 Ref=off

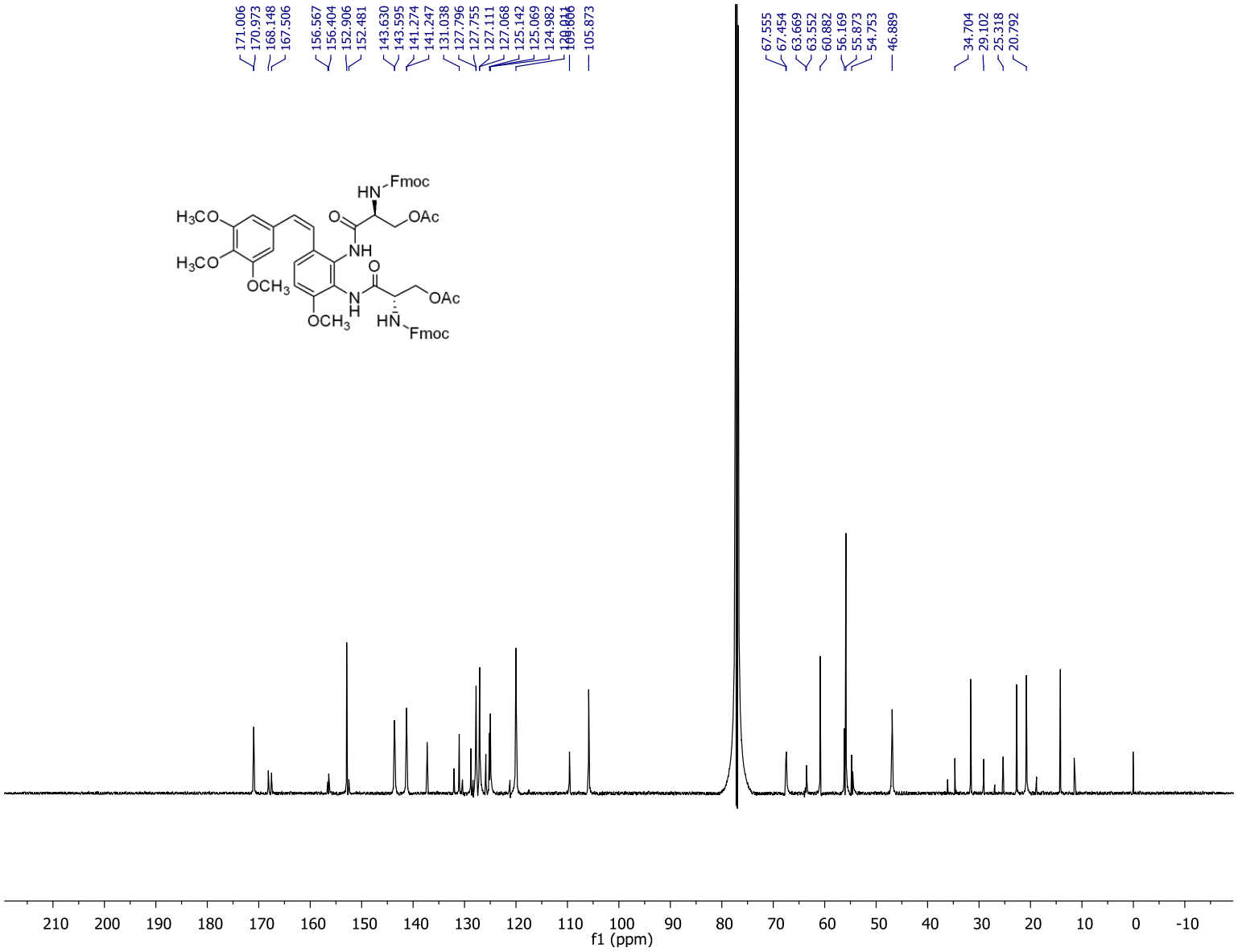
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.920	BB	0.0894	29.69990	4.99136	0.2281
2	6.664	VB	0.1527	1.29935e4	1215.47620	99.7719
Totals :				1.30232e4	1220.46756	

*** End of Report ***

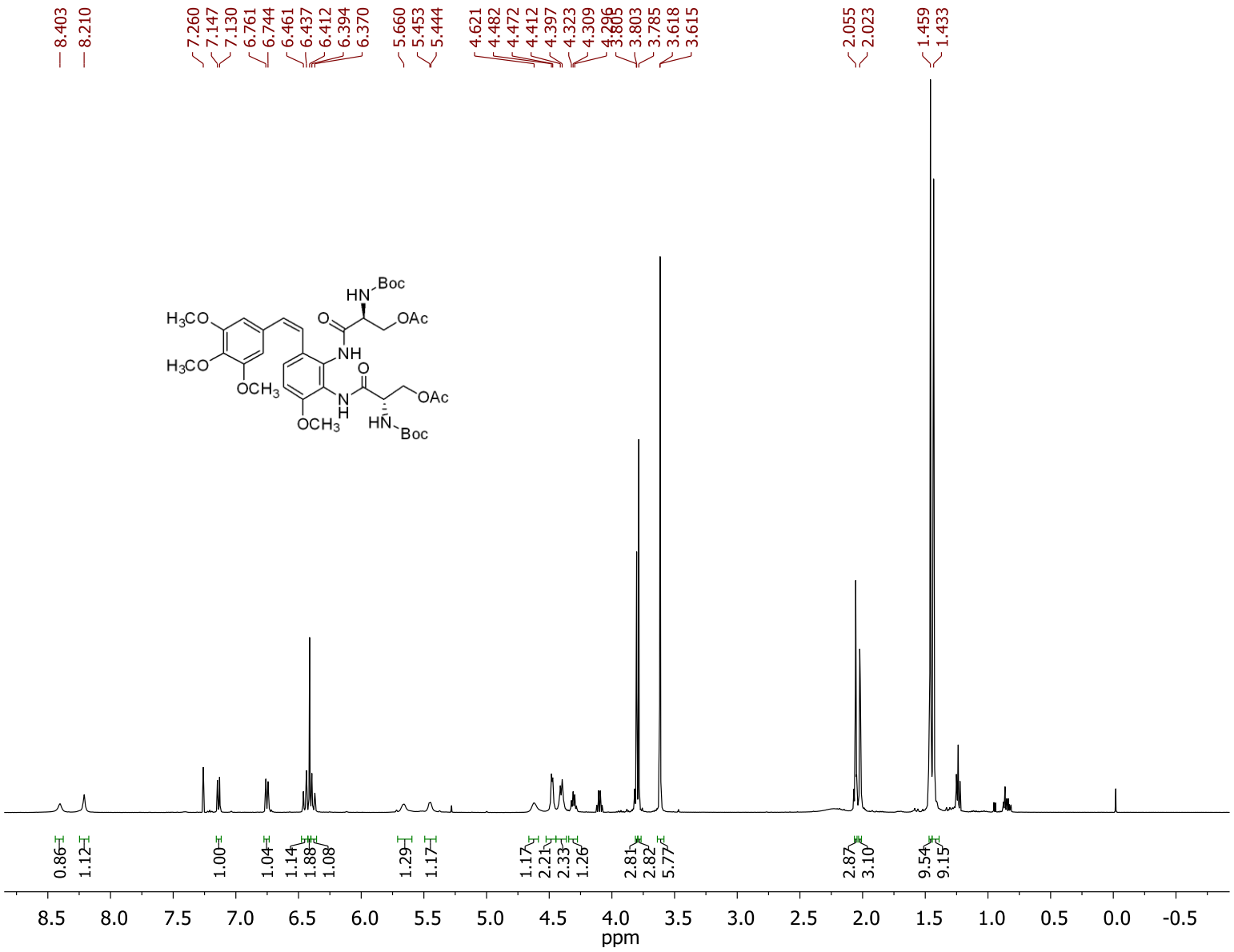
^{1}H NMR ($CDCl_3$, 500 MHz) Compound **19**



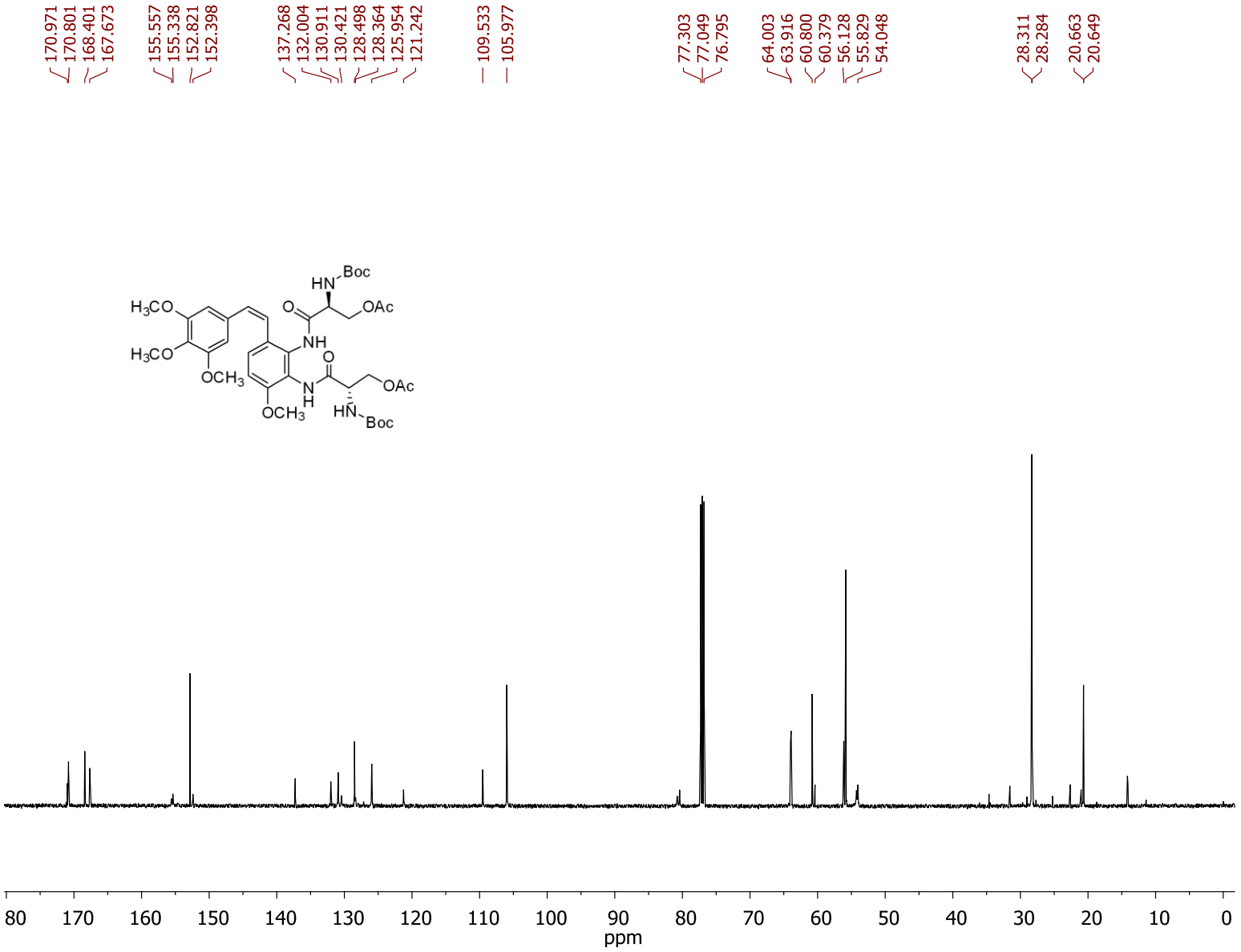
^{13}C NMR ($CDCl_3$, 150 MHz) Compound **19**



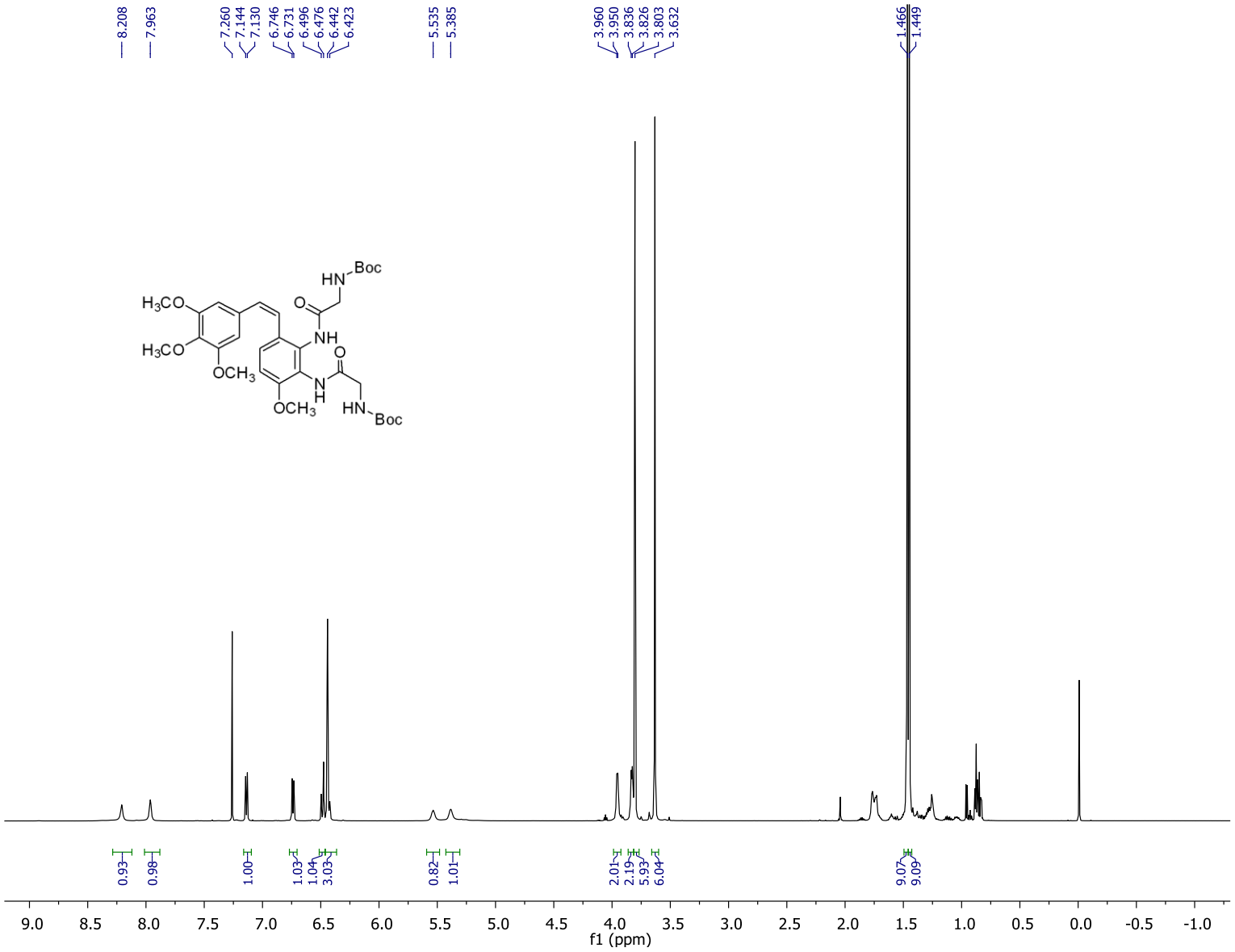
^{1}H NMR ($CDCl_3$, 500 MHz) Compound **20**



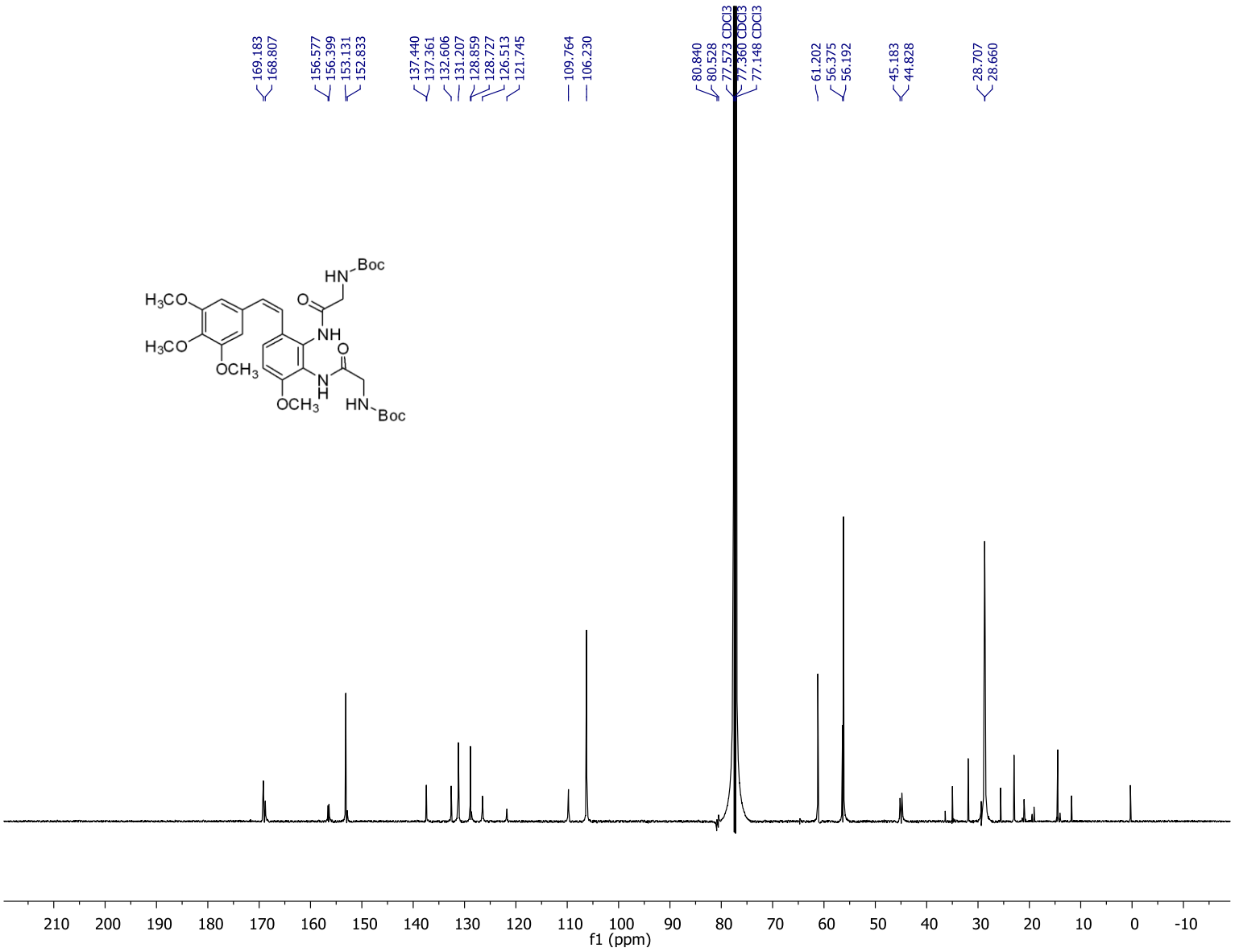
^{13}C NMR ($CDCl_3$, 125 MHz) Compound **20**



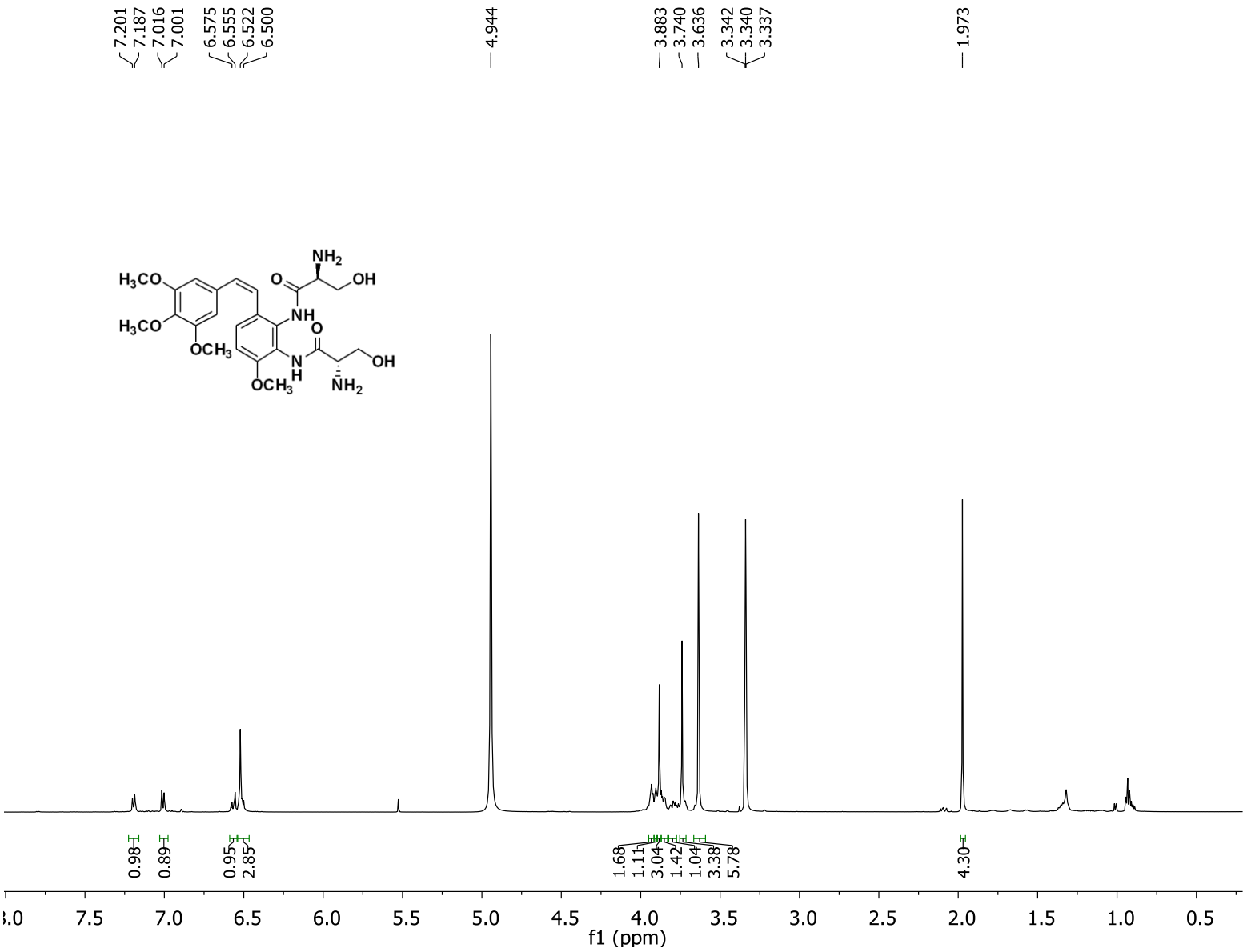
^{1}H NMR ($CDCl_3$, 600 MHz) Compound **21**



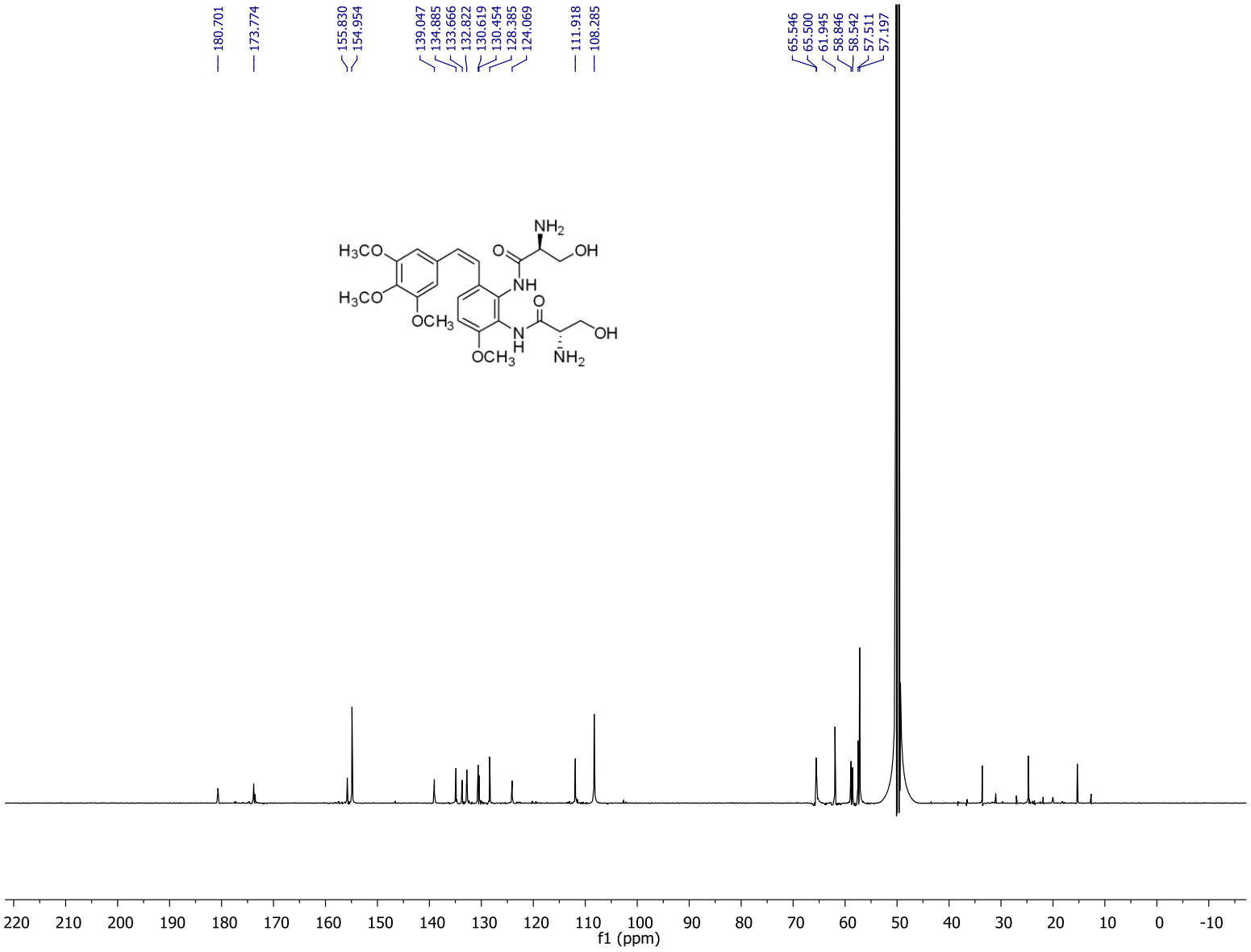
^{13}C NMR ($CDCl_3$, 150 MHz) Compound **21**



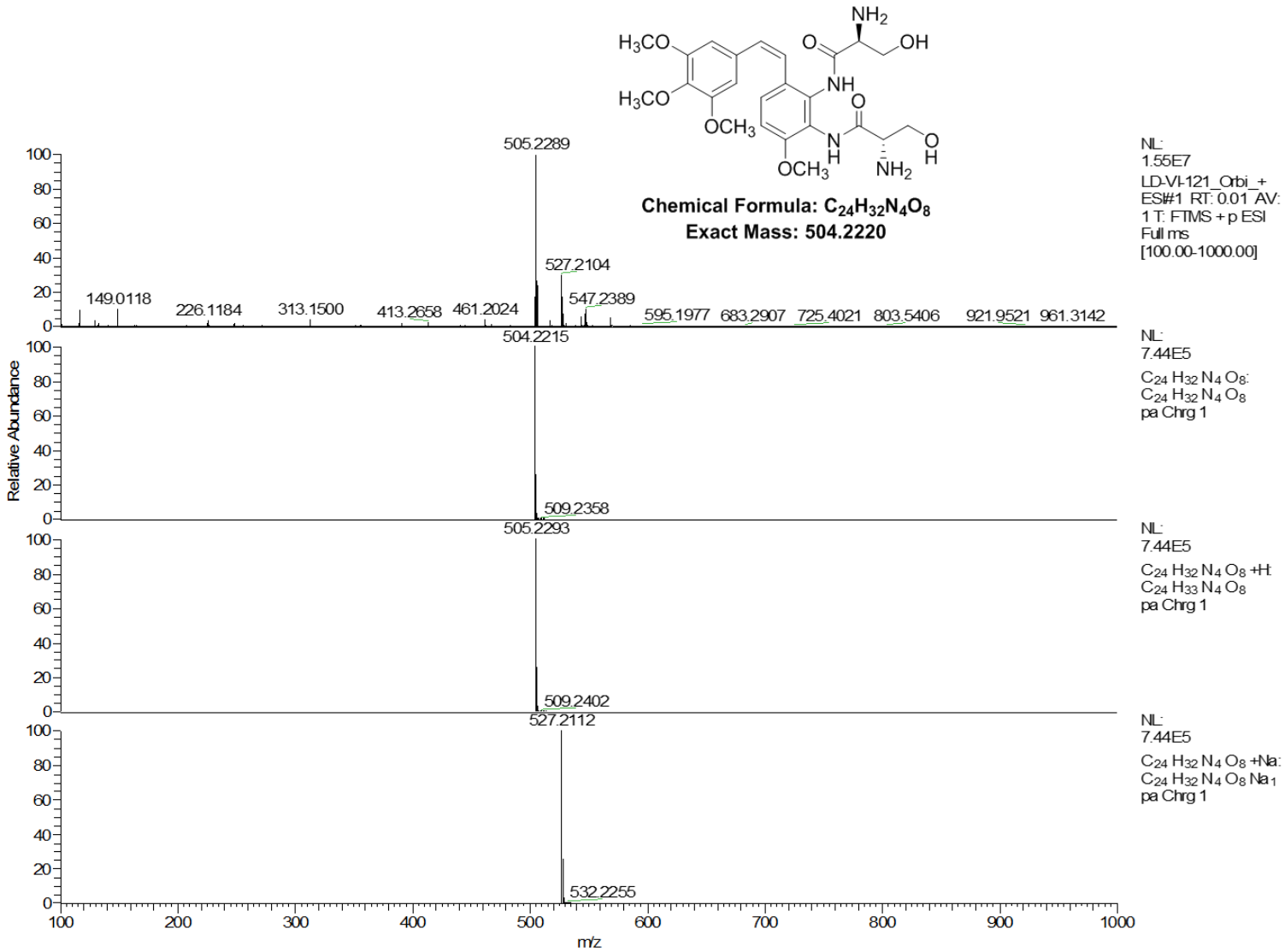
^{1}H NMR (CD_3OD, 600 MHz) Compound **22**



^{13}C NMR (CD_3OD, 150 MHz) Compound **22**



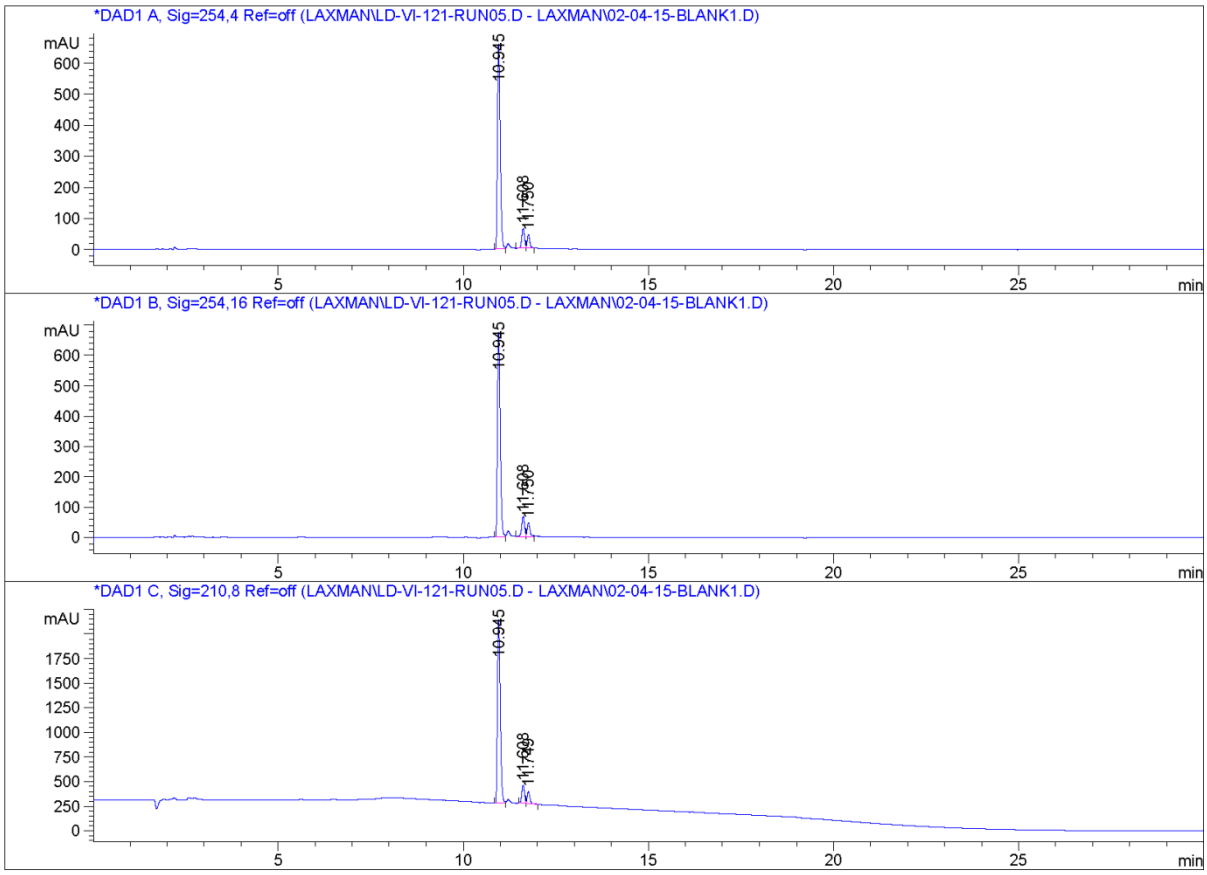
HRMS Compound **22**



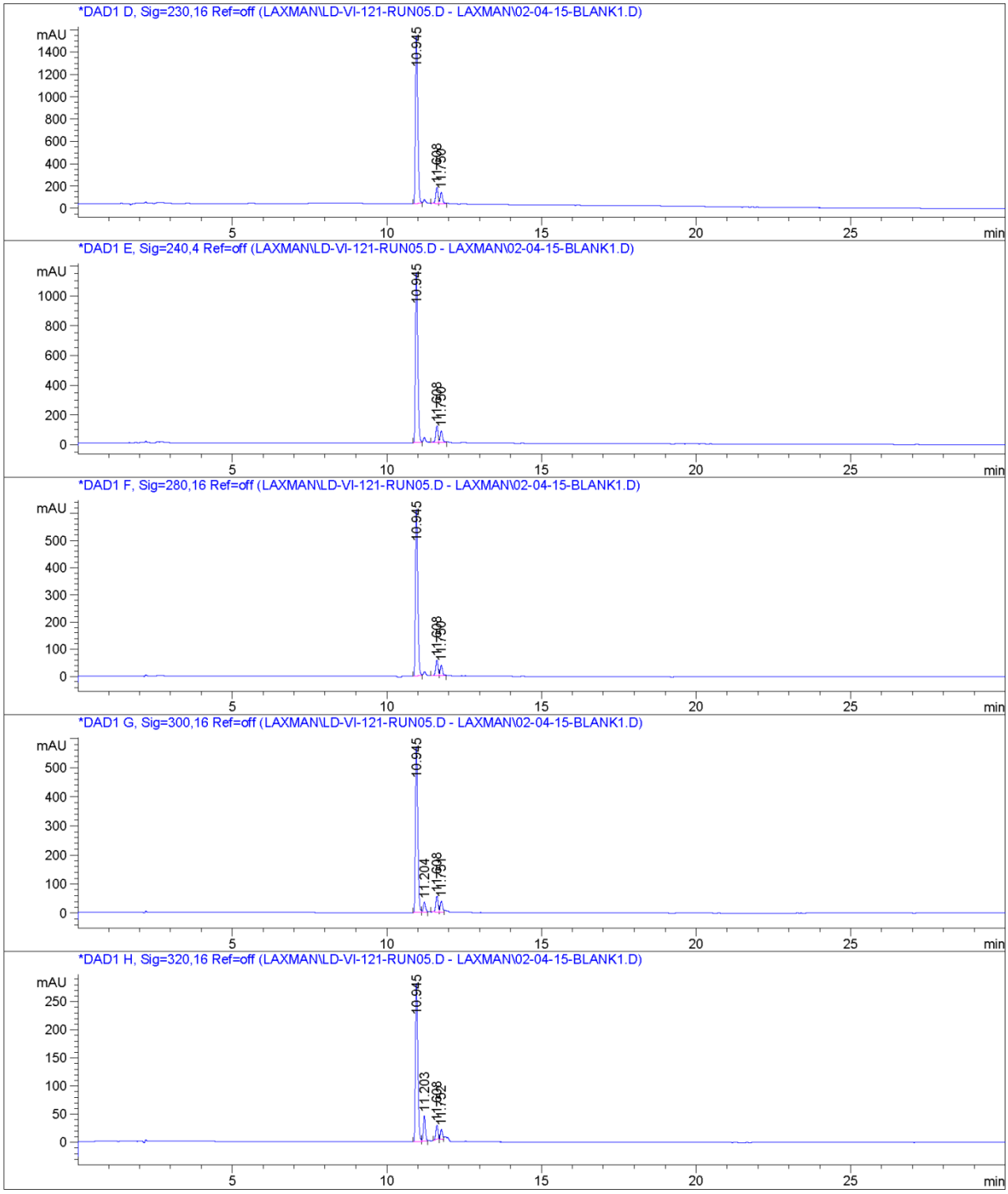
HPLC for Compound **22**

Data File C:\CHEM32\1\DATA\LAXMAN\LD-VI-121-RUN05.D
Sample Name: LD-VI-121-run5

```
======================================================================
Acq. Operator   : Laxman
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 2/4/2015 9:18:24 AM
Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD2.M
Last changed    : 2/4/2015 9:15:35 AM by Laxman
Analysis Method : C:\CHEM32\1\DATA\LAXMAN\LD-VI-121-RUN05.D\DA.M (MASTERMETHOD2.M)
Last changed    : 2/4/2015 10:03:37 AM by Laxman
Sample Info     : Method-Mastermethod2
                  0.05% TFA/H2O
```



Data File C:\CHEM32\1\DATA\LAXMAN\LD-VI-121-RUN05.D
Sample Name: LD-VI-121-run5



Data File C:\CHEM32\1\DATA\LAXMAN\LD-VI-121-RUN05.D
Sample Name: LD-VI-121-run5

==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.945	BV	0.0806	3429.38184	660.40332	85.7238
2	11.608	BV	0.0818	342.65540	64.67617	8.5653
3	11.750	VV	0.0771	228.46587	45.06903	5.7109
Totals :				4000.50310	770.14853	

Signal 2: DAD1 B, Sig=254,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.945	BV	0.0806	3503.13452	674.69476	85.7581
2	11.608	BV	0.0818	348.86224	65.84724	8.5403
3	11.750	VV	0.0771	232.90524	45.94286	5.7016
Totals :				4084.90201	786.48487	

Signal 3: DAD1 C, Sig=210,8 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.945	BV	0.0842	9926.38086	1861.91199	86.6126
2	11.608	BV	0.0789	927.72119	183.65503	8.0948
3	11.749	VB	0.0748	606.56696	124.58265	5.2926
Totals :				1.14607e4	2170.14967	

Data File C:\CHEM32\1\DATA\LAXMAN\LD-VI-121-RUN05.D
Sample Name: LD-VI-121-run5

Signal 4: DAD1 D, Sig=230,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.945	BV	0.0808	7864.29395	1509.11328	85.7517
2	11.608	BV	0.0822	780.64148	146.49777	8.5121
3	11.750	VV	0.0783	526.07617	101.73031	5.7363
Totals :				9171.01160	1757.34136	

Signal 5: DAD1 E, Sig=240,4 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.945	BV	0.0806	5953.35156	1145.60352	85.4289
2	11.608	BV	0.0823	600.10669	112.39833	8.6114
3	11.750	VV	0.0793	415.31732	79.05017	5.9597
Totals :				6968.77557	1337.05202	

Signal 6: DAD1 F, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.945	BV	0.0805	3185.41992	613.96997	86.4240
2	11.608	BV	0.0817	305.75058	57.77947	8.2954
3	11.750	VV	0.0770	194.63496	38.46372	5.2807
Totals :				3685.80547	710.21316	

Signal 7: DAD1 G, Sig=300,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.945	BV	0.0809	2988.00757	572.34558	81.8322
2	11.204	VV	0.0725	169.99767	35.08196	4.6557
3	11.608	BV	0.0823	296.85989	55.58070	8.1301
4	11.751	VV	0.0785	196.51675	37.88121	5.3820

Data File C:\CHEM32\1\DATA\LAXMAN\LD-VI-121-RUN05.D
Sample Name: LD-VI-121-run5

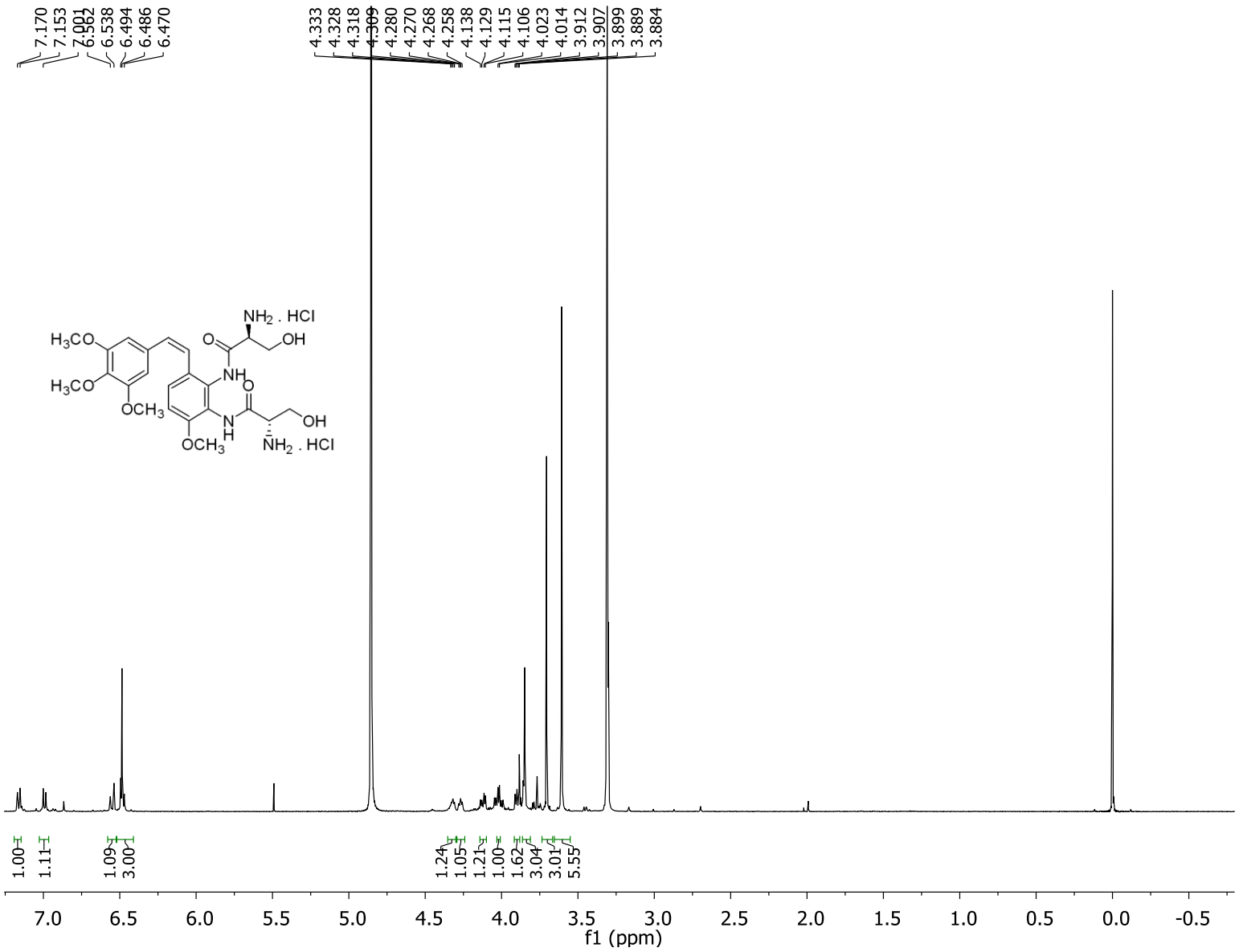
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
Totals :				3651.38188	700.88945	

Signal 8: DAD1 H, Sig=320,16 Ref=off
Signal has been modified after loading from rawdata file!

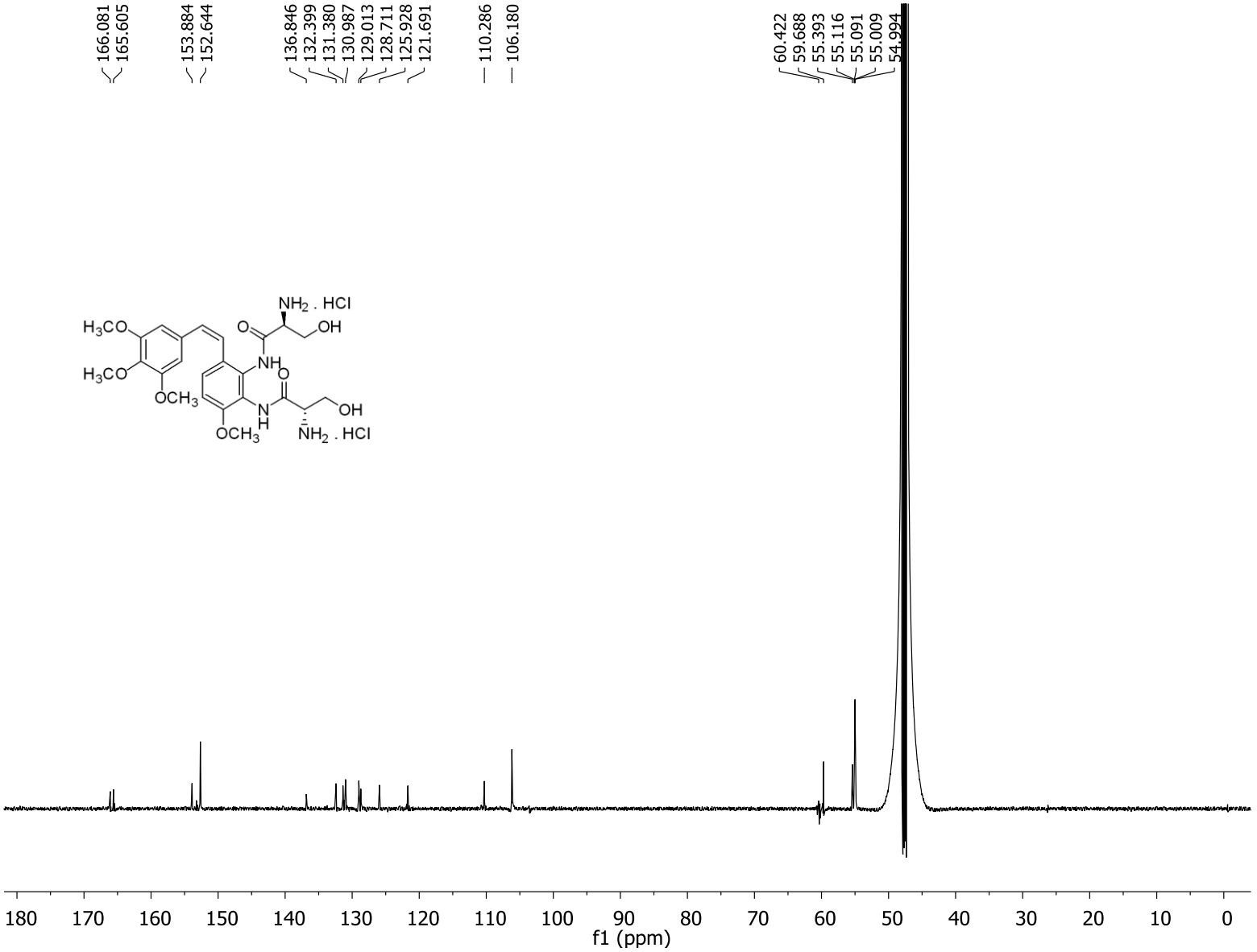
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.945	BV	0.0814	1481.12476	281.65887	78.3325
2	11.203	VV	0.0685	204.32208	45.42173	10.8060
3	11.608	BB	0.0749	121.84870	25.89132	6.4442
4	11.752	BV	0.0733	83.52104	17.62517	4.4172
Totals :				1890.81658	370.59708	

*** End of Report ***

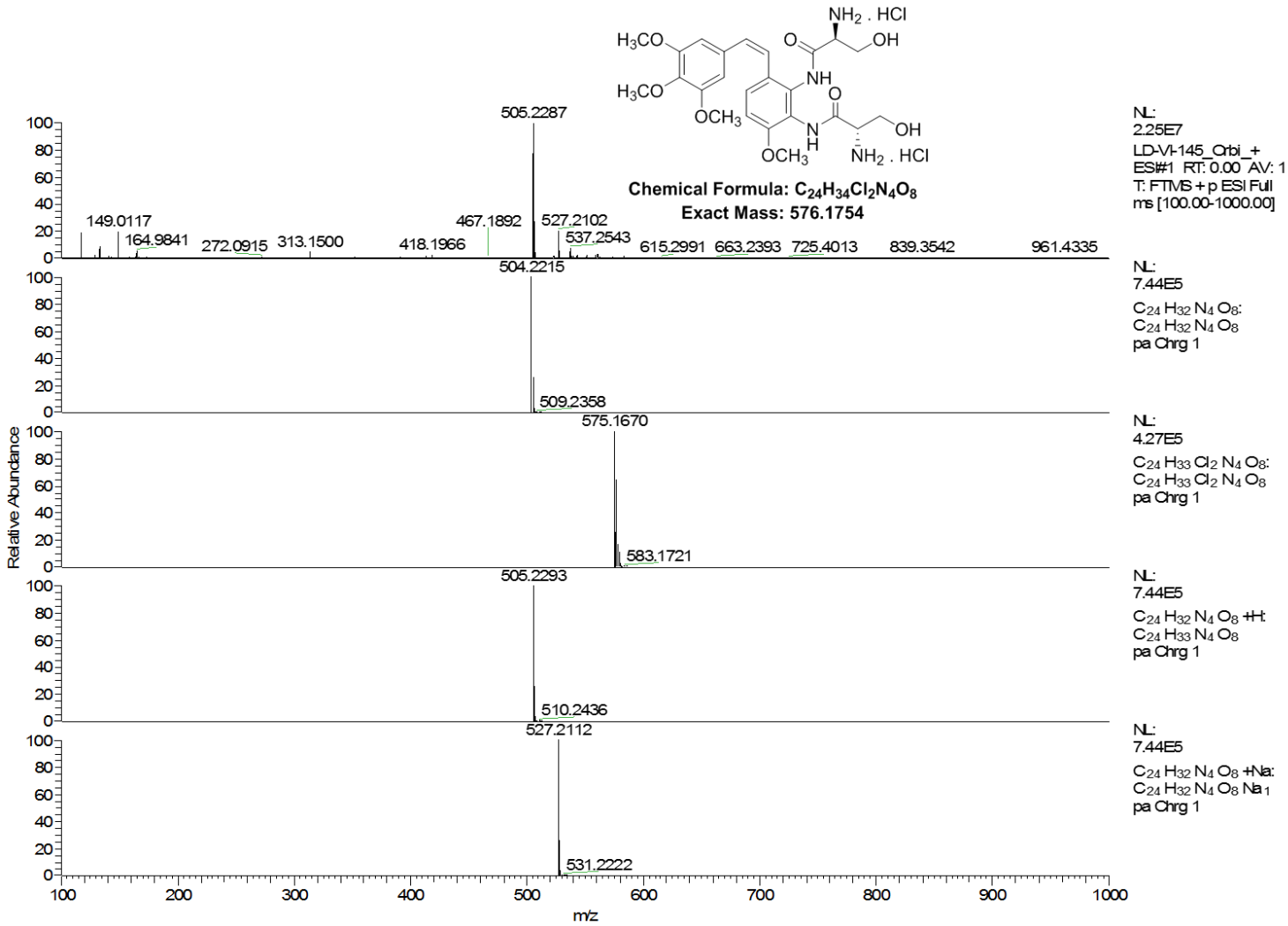
^{1}H NMR (CD_3OD, 500 MHz) Compound **23**



^{13}C NMR (CD_3OD, 150 MHz) Compound **23**



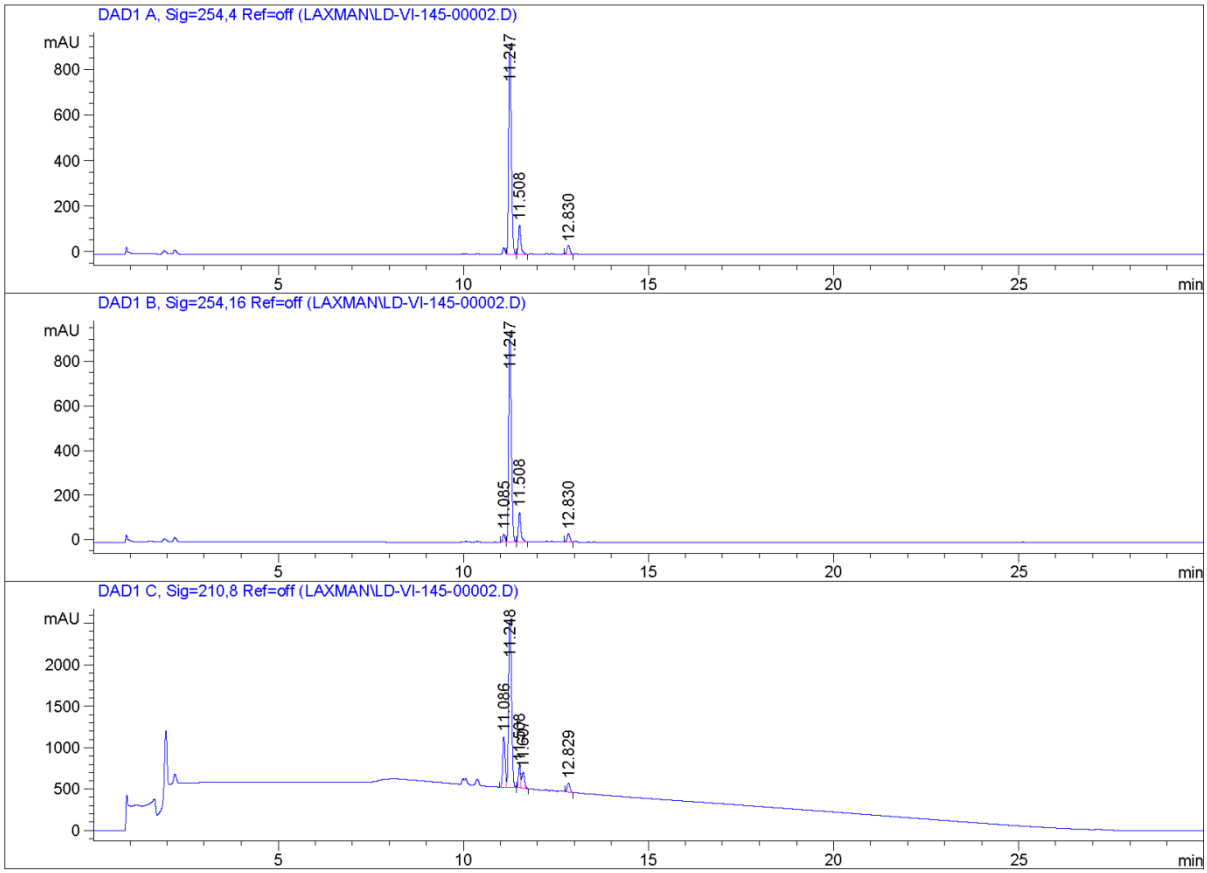
HRMS Compound **23**



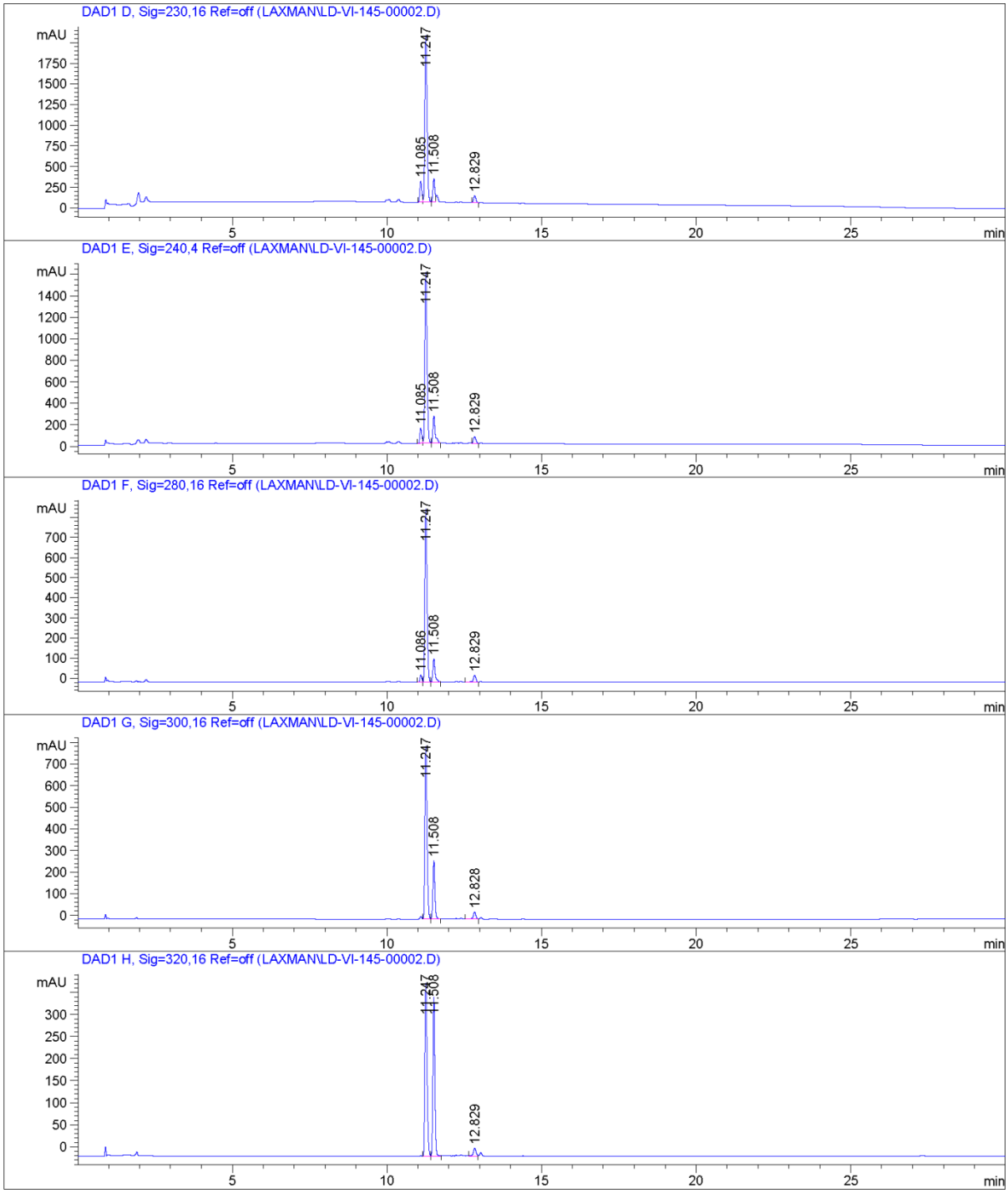
HPLC for Compound **23**

Data File C:\CHEM32\1\DATA\LAXMAN\LD-VI-145-00002.D
Sample Name: LD-VI-145-run2

```
======================================================================
Acq. Operator   : Laxman
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 2/3/2015 10:54:00 AM
Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD2.M
Last changed    : 2/3/2015 10:52:45 AM by Laxman
Analysis Method : C:\CHEM32\1\DATA\LAXMAN\LD-VI-145-00002.D\DA.M (MASTERMETHOD2.M)
Last changed    : 2/4/2015 9:20:40 AM by Laxman
Sample Info     : Method-Mastermethod2
```



Data File C:\CHEM32\1\DATA\LAXMAN\LD-VI-145-00002.D
Sample Name: LD-VI-145-run2



Data File C:\CHEM32\1\DATA\LAXMAN\LD-VI-145-00002.D
Sample Name: LD-VI-145-run2

==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.247	VV	0.0750	4529.85498	927.61292	84.7722
2	11.508	VB	0.0703	604.70068	129.86781	11.3164
3	12.830	VV	0.0862	209.00600	38.00933	3.9114
Totals :				5343.56166	1095.49006	

Signal 2: DAD1 B, Sig=254,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.085	BV	0.0666	158.56816	36.54854	2.8155
2	11.247	VV	0.0750	4626.72656	947.20282	82.1503
3	11.508	VB	0.0711	634.57526	134.26546	11.2673
4	12.830	VV	0.0861	212.15869	38.62234	3.7670
Totals :				5632.02867	1156.63916	

Signal 3: DAD1 C, Sig=210,8 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.086	BV	0.0678	2736.85107	615.96875	16.5802
2	11.248	VB	0.0883	1.11715e4	2027.12781	67.6783
3	11.508	BV	0.0638	1073.51233	262.24918	6.5035
4	11.607	VB	0.0726	937.48230	193.35208	5.6794
5	12.829	VB	0.0846	587.43060	109.54142	3.5587
Totals :				1.65068e4	3208.23923	

Data File C:\CHEM32\1\DATA\LAXMAN\LD-VI-145-00002.D
Sample Name: LD-VI-145-run2

Signal 4: DAD1 D, Sig=230,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.085	BV	0.0671	1128.58777	257.43103	8.7839
2	11.247	VB	0.0801	1.00606e4	2019.74780	78.3026
3	11.508	BV	0.0658	1199.73914	281.16333	9.3377
4	12.829	VB	0.0847	459.43491	85.48837	3.5758
Totals :				1.28483e4	2643.83054	

Signal 5: DAD1 E, Sig=240,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.085	BV	0.0672	634.01740	144.39835	6.2688
2	11.247	VV	0.0754	7837.16797	1593.89563	77.4891
3	11.508	VB	0.0754	1302.56641	255.79105	12.8790
4	12.829	VB	0.0847	340.14511	63.32522	3.3631
Totals :				1.01139e4	2057.41024	

Signal 6: DAD1 F, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.086	BV	0.0671	158.43652	36.17655	3.0899
2	11.247	VV	0.0748	4190.94482	860.92053	81.7330
3	11.508	VV	0.0736	576.60266	116.70569	11.2451
4	12.829	BV	0.0917	201.62273	33.76098	3.9321
Totals :				5127.60674	1047.56375	

Signal 7: DAD1 G, Sig=300,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.247	VV	0.0750	3908.86646	800.75116	74.1305
2	11.508	VB	0.0668	1175.77905	270.14182	22.2983
3	12.828	BV	0.0913	188.30544	31.69815	3.5712
Totals :				5272.95094	1102.59113	

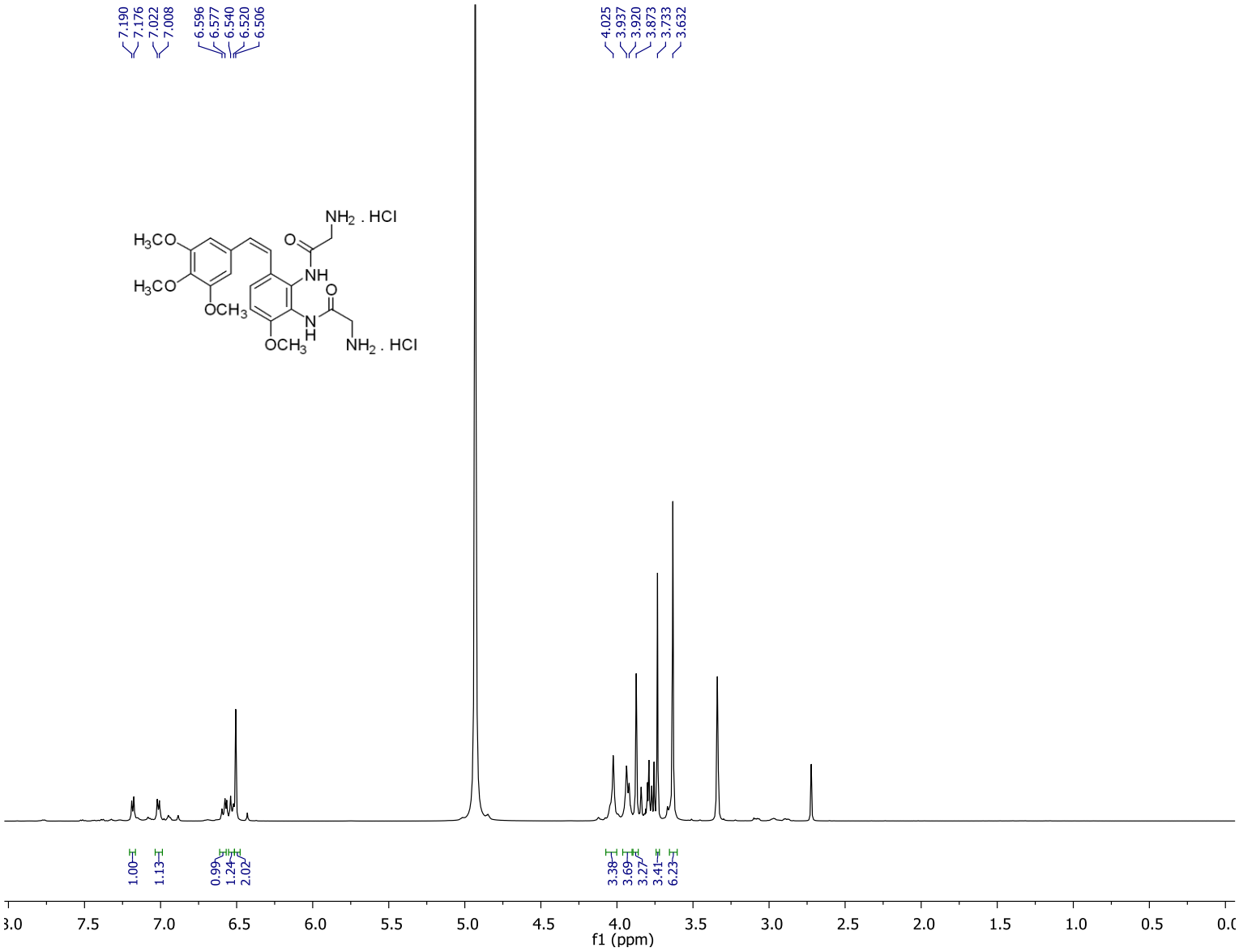
Data File C:\CHEM32\1\DATA\LAXMAN\LD-VI-145-00002.D
Sample Name: LD-VI-145-run2

Signal 8: DAD1 H, Sig=320,16 Ref=off

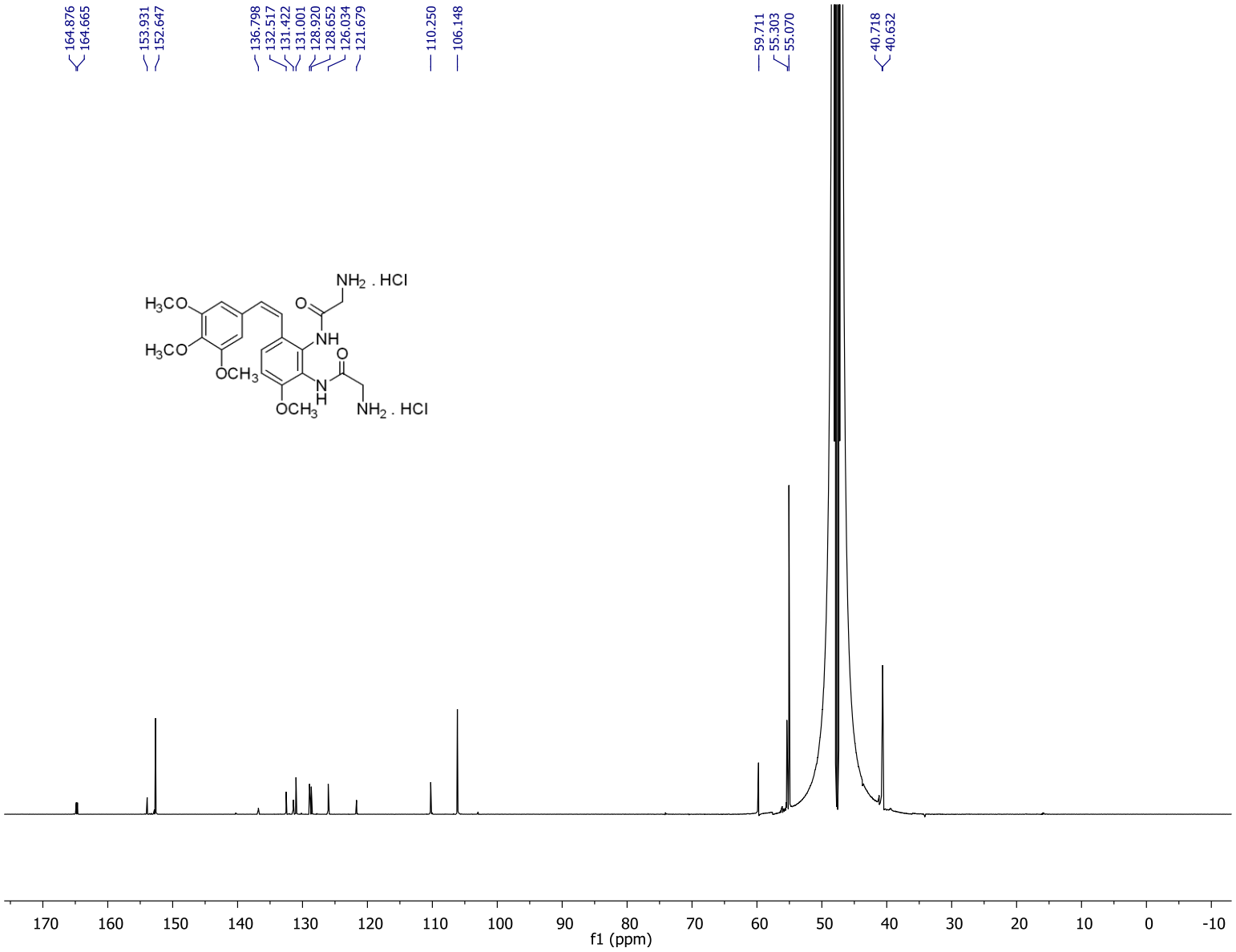
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.247	VV	0.0753	1927.66699	392.75601	53.8382
2	11.508	VV	0.0661	1547.21765	360.35504	43.2126
3	12.829	VV	0.0940	105.59404	17.09910	2.9492
Totals :				3580.47868	770.21016	

*** End of Report ***

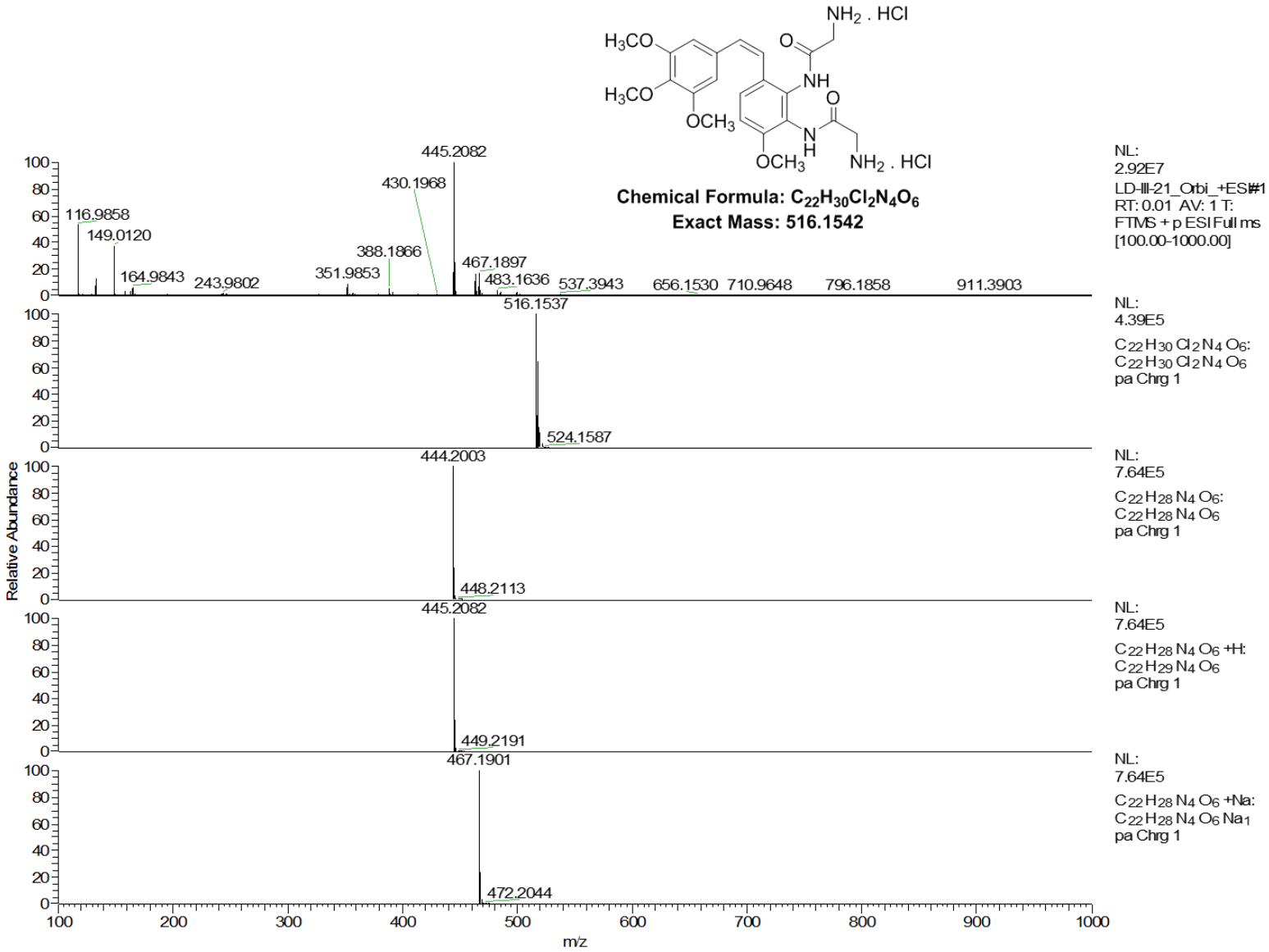
^{1}H NMR (CD_3OD, 600 MHz) Compound **24**



^{13}C NMR (CD_3OD, 150 MHz) Compound **24**



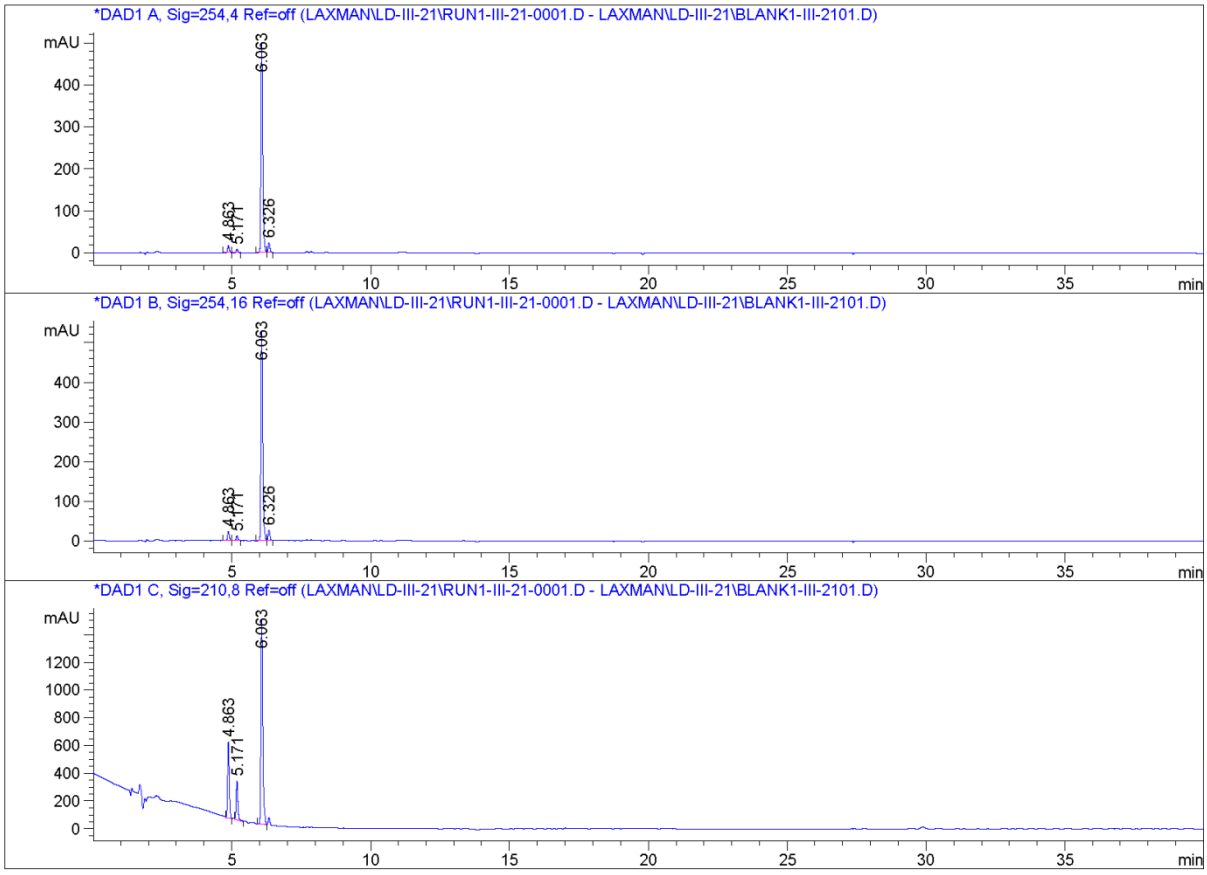
HRMS Compound **24**



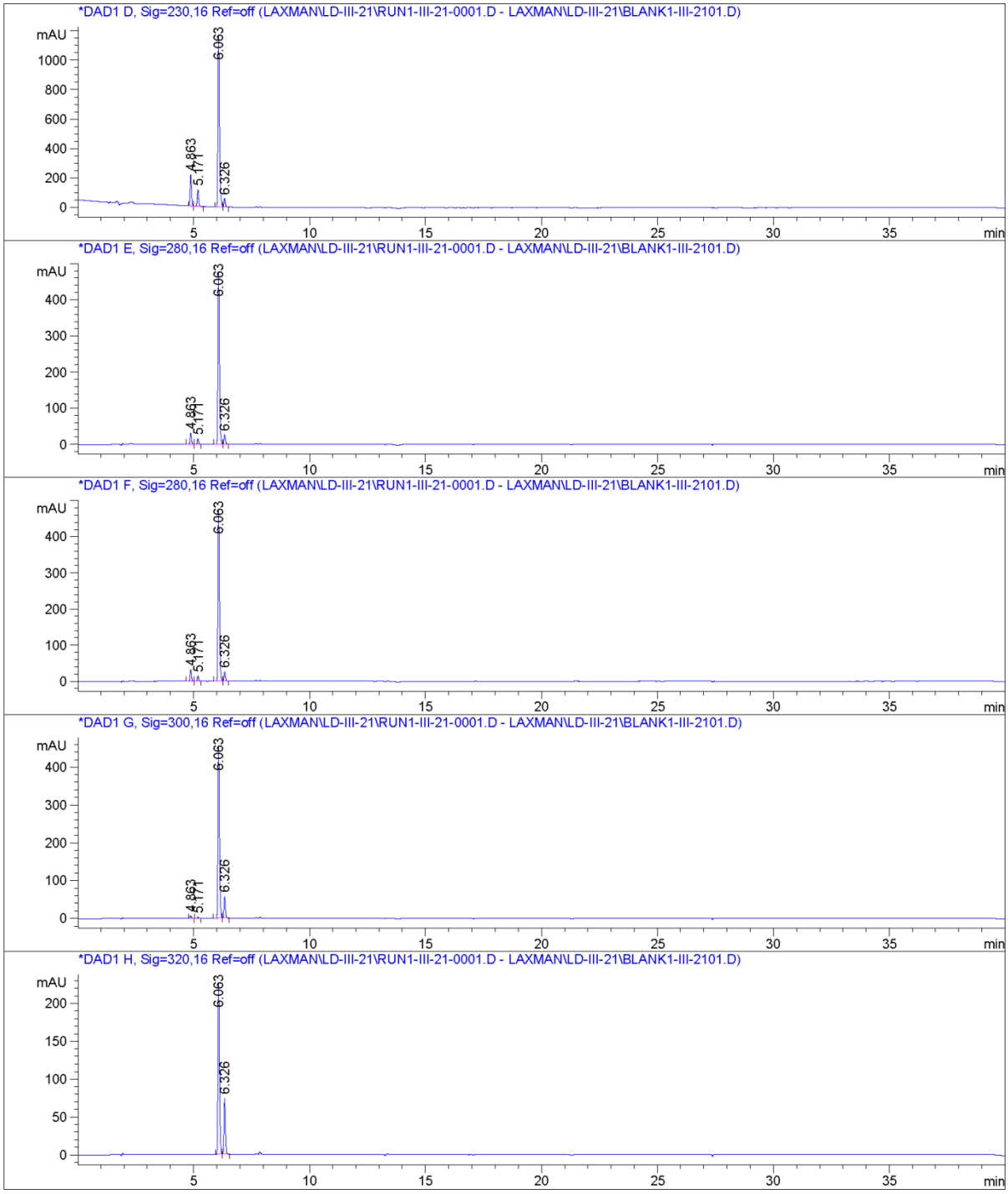
HPLC for Compound **24**

Data File C:\CHEM32\1\DATA\LAXMAN\LD-III-21\RUN1-III-21-0001.D
Sample Name: run1-LD-III-21-1A

```
======================================================================
Acq. Operator   : Laxman
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 12/21/2012 11:37:50 AM
Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed    : 12/21/2012 11:34:21 AM by Laxman
Analysis Method : C:\CHEM32\1\DATA\LAXMAN\LD-III-21\RUN1-III-21-0001.D\DA.M (MASTERMETHOD.M)
Last changed    : 1/18/2013 10:56:37 AM by song
                  (modified after loading)
Sample Info     : run1
                  10%ACN in 0.1%TFA water solution
```



Data File C:\CHEM32\1\DATA\LAXMAN\LD-III-21\RUN1-III-21-0001.D
Sample Name: run1-LD-III-21-1A



Data File C:\CHEM32\1\DATA\LAXMAN\LD-III-21\RUN1-III-21-0001.D
Sample Name: run1-LD-III-21-1A

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.863	BB	0.0643	71.16022	17.17359	2.8439
2	5.171	BB	0.0645	37.81452	9.08890	1.5113
3	6.063	BV	0.0693	2286.42432	500.08197	91.3767
4	6.326	VB	0.0649	106.79793	24.49240	4.2682
Totals :				2502.19698	550.83686	

Signal 2: DAD1 B, Sig=254,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.863	BB	0.0642	98.68317	23.90006	3.6702
2	5.171	BB	0.0639	50.66319	12.33097	1.8843
3	6.063	BV	0.0694	2422.45068	529.47382	90.0960
4	6.326	VB	0.0648	116.94734	26.84951	4.3495
Totals :				2688.74438	592.55435	

Signal 3: DAD1 C, Sig=210,8 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.863	VB	0.0650	2312.86865	551.00055	21.8635
2	5.171	VV	0.0671	1246.27808	284.80121	11.7810
3	6.063	BV	0.0732	7019.53174	1483.43799	66.3555
Totals :				1.05787e4	2319.23975	

Data File C:\CHEM32\1\DATA\LAXMAN\LD-III-21\RUN1-III-21-0001.D
Sample Name: run1-LD-III-21-1A

Signal 4: DAD1 D, Sig=230,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.863	VV	0.0648	884.47058	211.53062	12.5062
2	5.171	VV	0.0674	505.99442	114.94447	7.1546
3	6.063	BV	0.0721	5426.37646	1169.56604	76.7275
4	6.326	VB	0.0645	255.42552	59.02868	3.6116
Totals :				7072.26698	1555.06981	

Signal 5: DAD1 E, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.863	BB	0.0643	129.42253	31.24761	5.2490
2	5.171	BB	0.0635	60.69393	14.92229	2.4616
3	6.063	BV	0.0688	2160.37939	477.03757	87.6182
4	6.326	VB	0.0654	115.17860	26.14397	4.6713
Totals :				2465.67446	549.35143	

Signal 6: DAD1 F, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.863	BB	0.0643	129.42253	31.24761	5.2490
2	5.171	BB	0.0635	60.69393	14.92229	2.4616
3	6.063	BV	0.0688	2160.37939	477.03757	87.6182
4	6.326	VB	0.0654	115.17860	26.14397	4.6713
Totals :				2465.67446	549.35143	

Signal 7: DAD1 G, Sig=300,16 Ref=off
Signal has been modified after loading from rawdata file!

Data File C:\CHEM32\1\DATA\LAXMAN\LD-III-21\RUN1-III-21-0001.D
Sample Name: run1-LD-III-21-1A

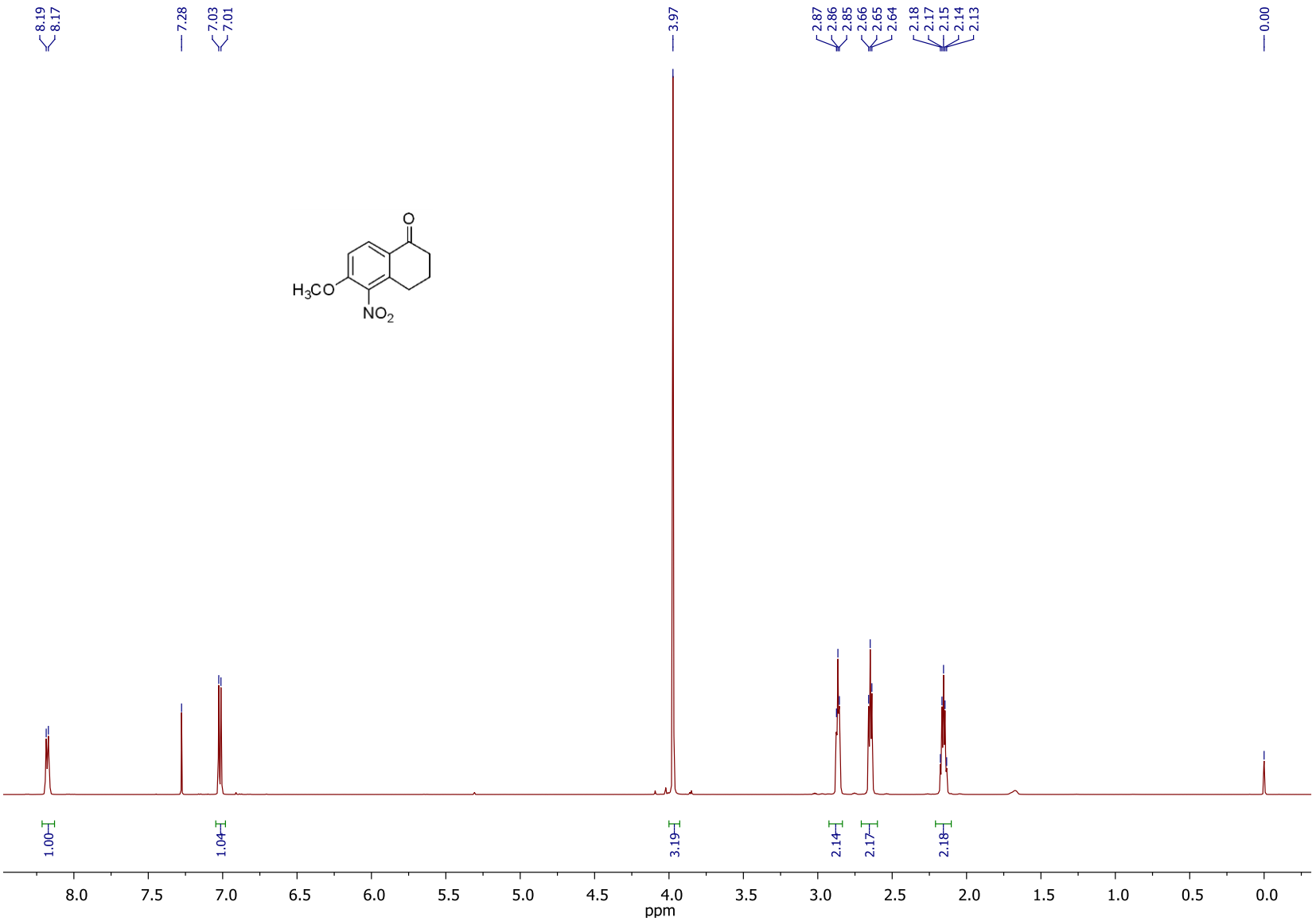
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.863	BB	0.0639	28.77723	7.00664	1.2205
2	5.171	BB	0.0640	11.33881	2.75776	0.4809
3	6.063	BV	0.0709	2067.91895	455.82397	87.7011
4	6.326	VB	0.0640	249.88266	58.33168	10.5976
Totals :				2357.91764	523.92005	

Signal 8: DAD1 H, Sig=320,16 Ref=off
Signal has been modified after loading from rawdata file!

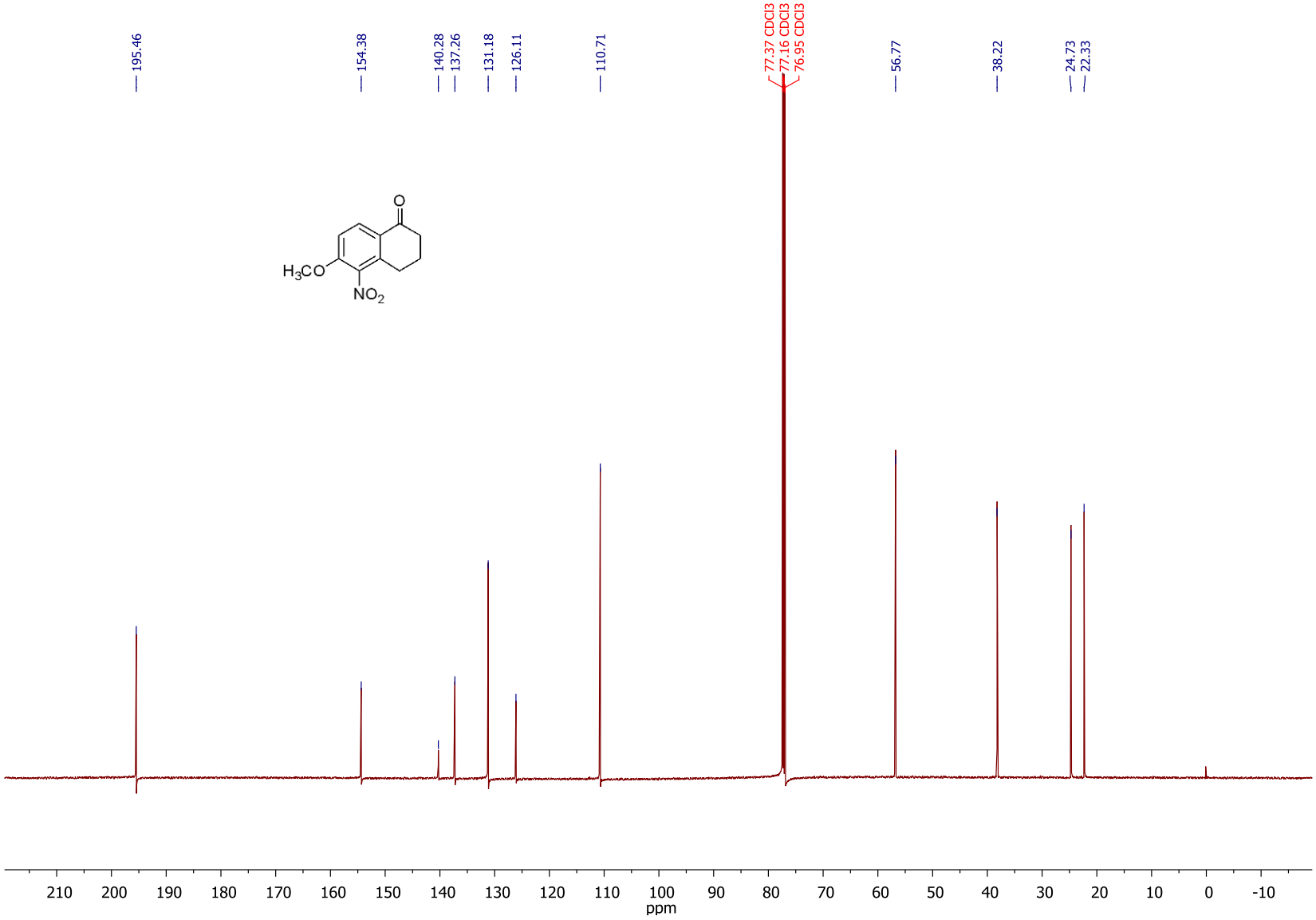
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.063	BV	0.0706	1020.73212	226.40169	76.5450
2	6.326	VB	0.0635	312.77420	73.71690	23.4550
Totals :				1333.50632	300.11858	

*** End of Report ***

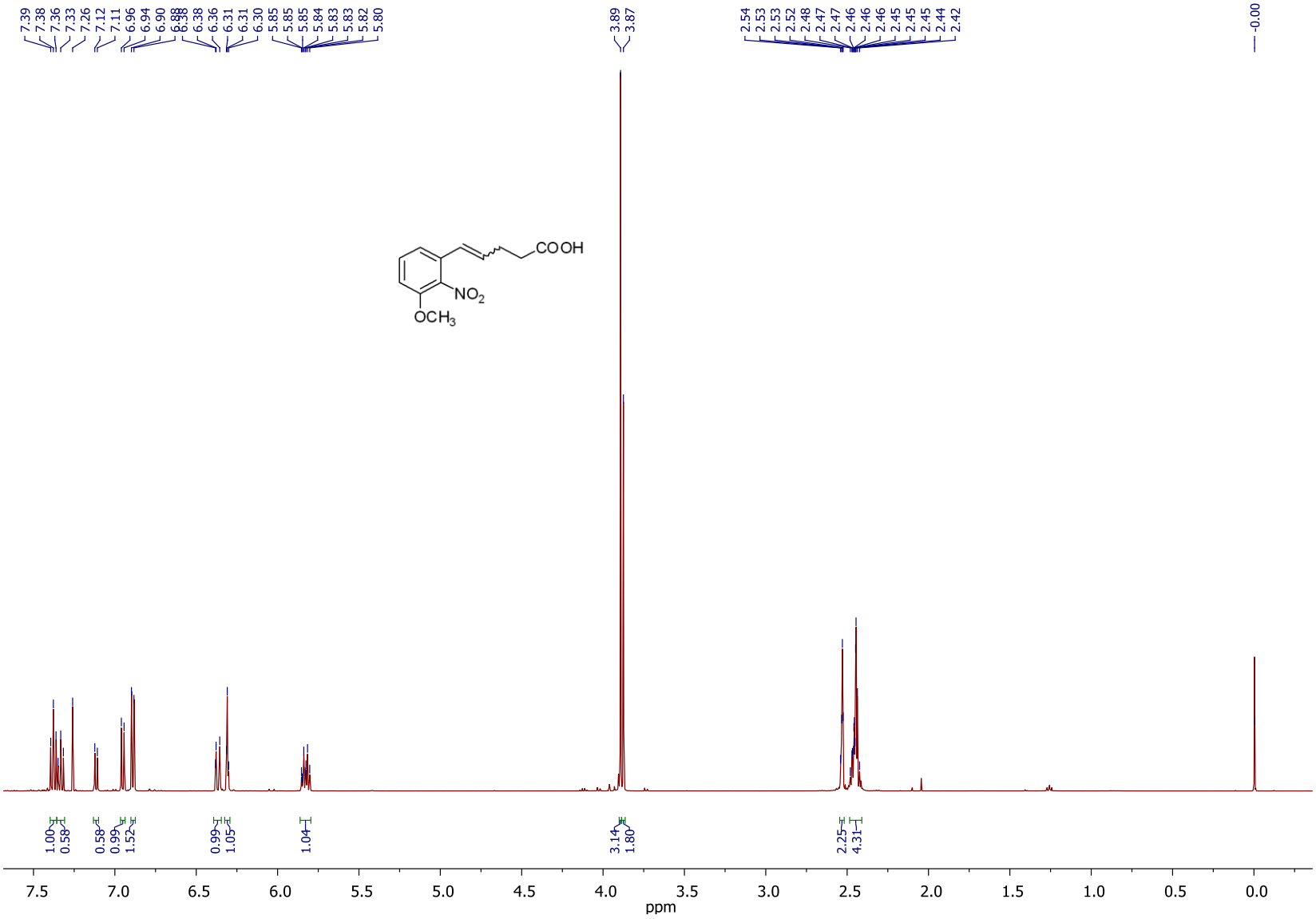
^{1}H NMR ($CDCl_3$, 600 MHz) Compound **25**



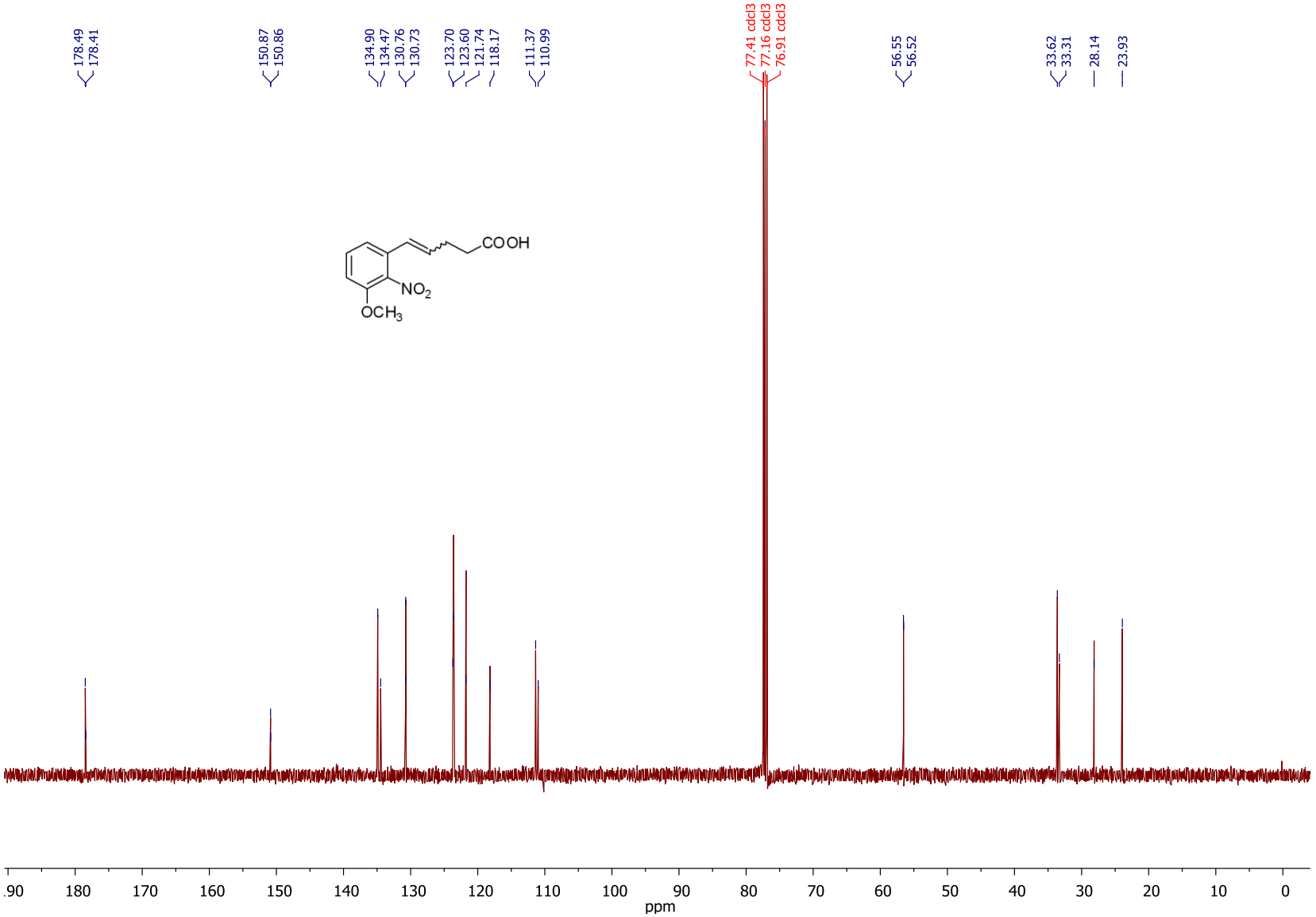
^{13}C NMR ($CDCl_3$, 150 MHz) Compound **25**



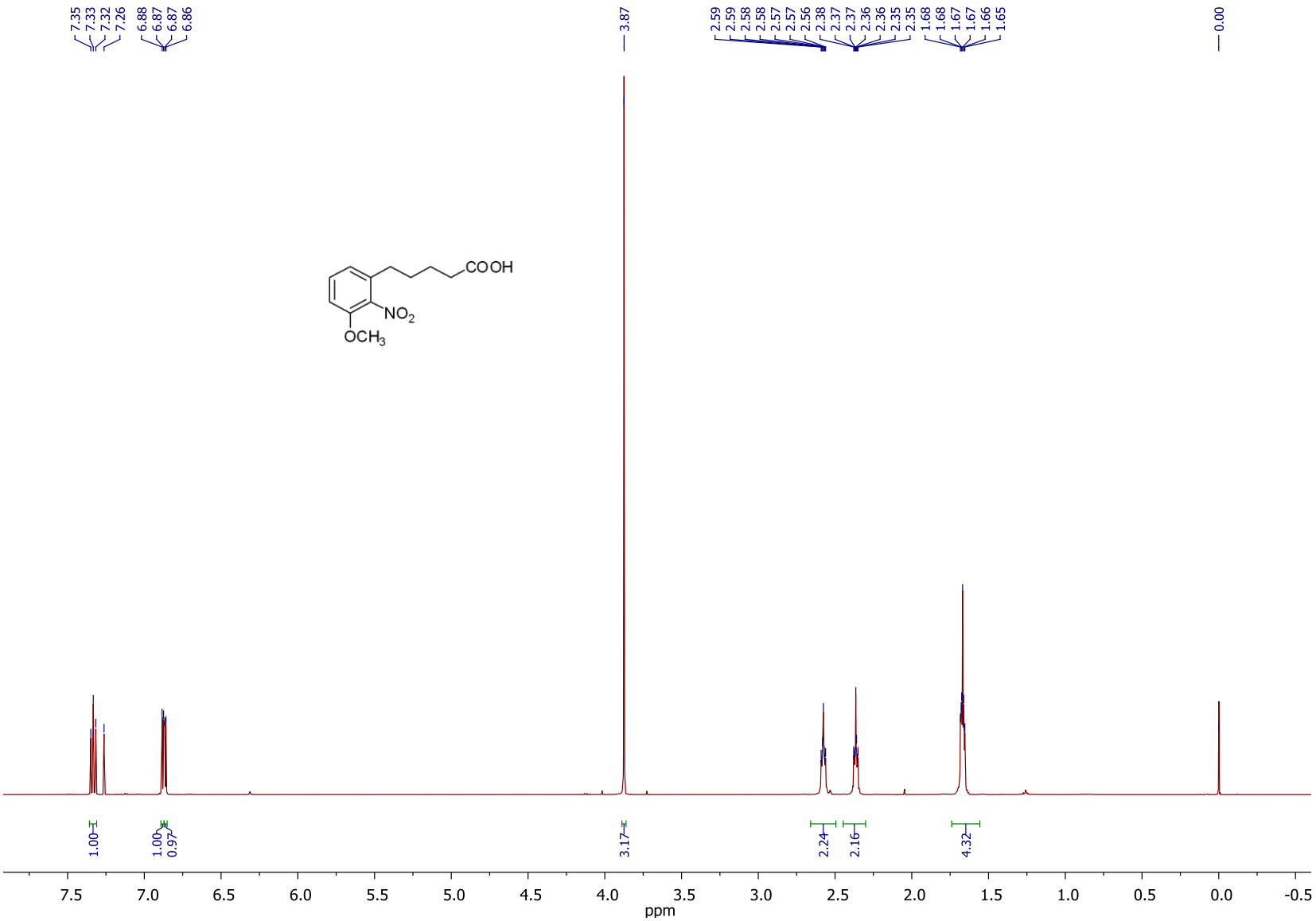
^{1}H NMR ($CDCl_3$, 500 MHz) Compound **27**



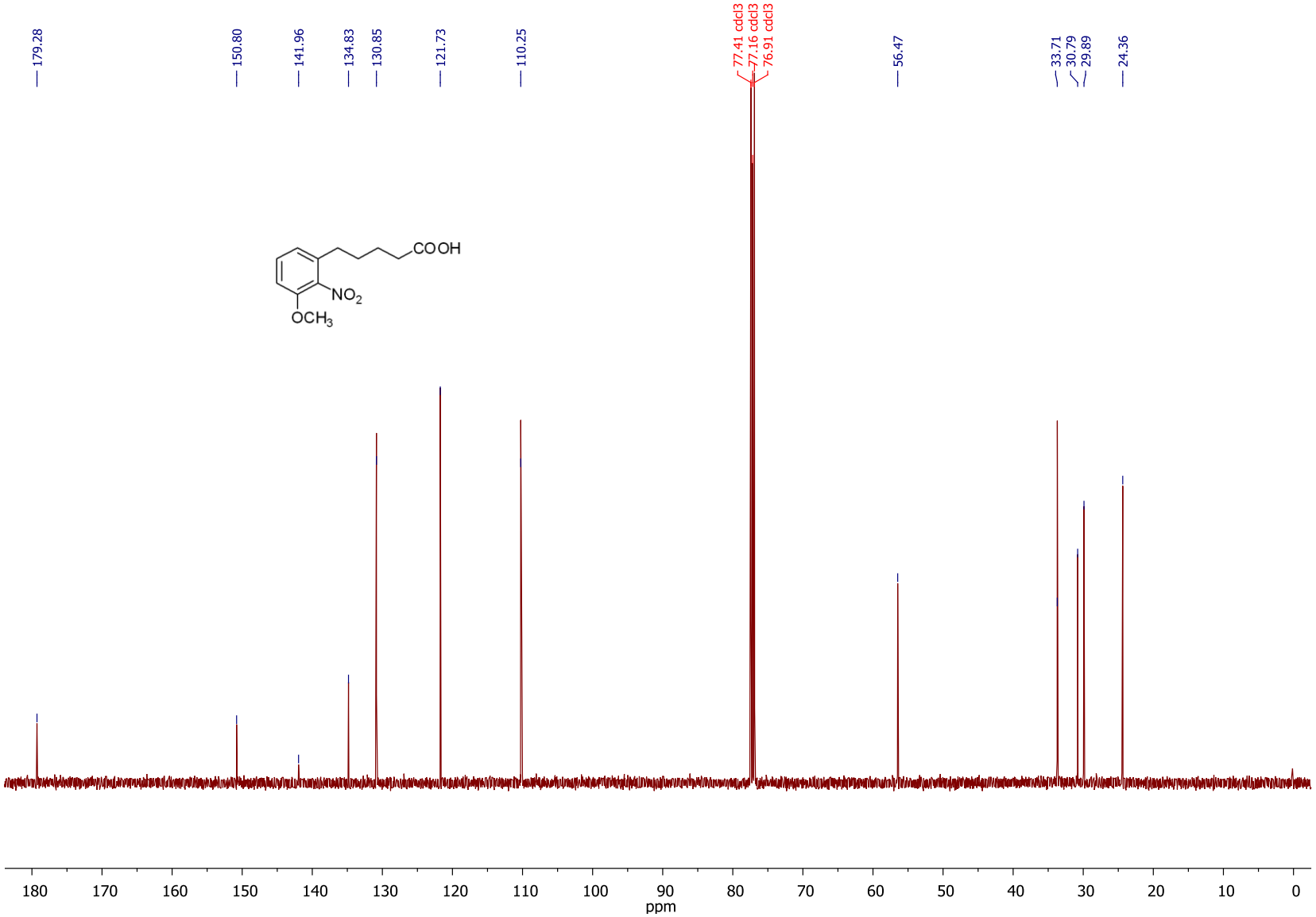
^{13}C NMR ($CDCl_3$, 125 MHz) Compound **27**



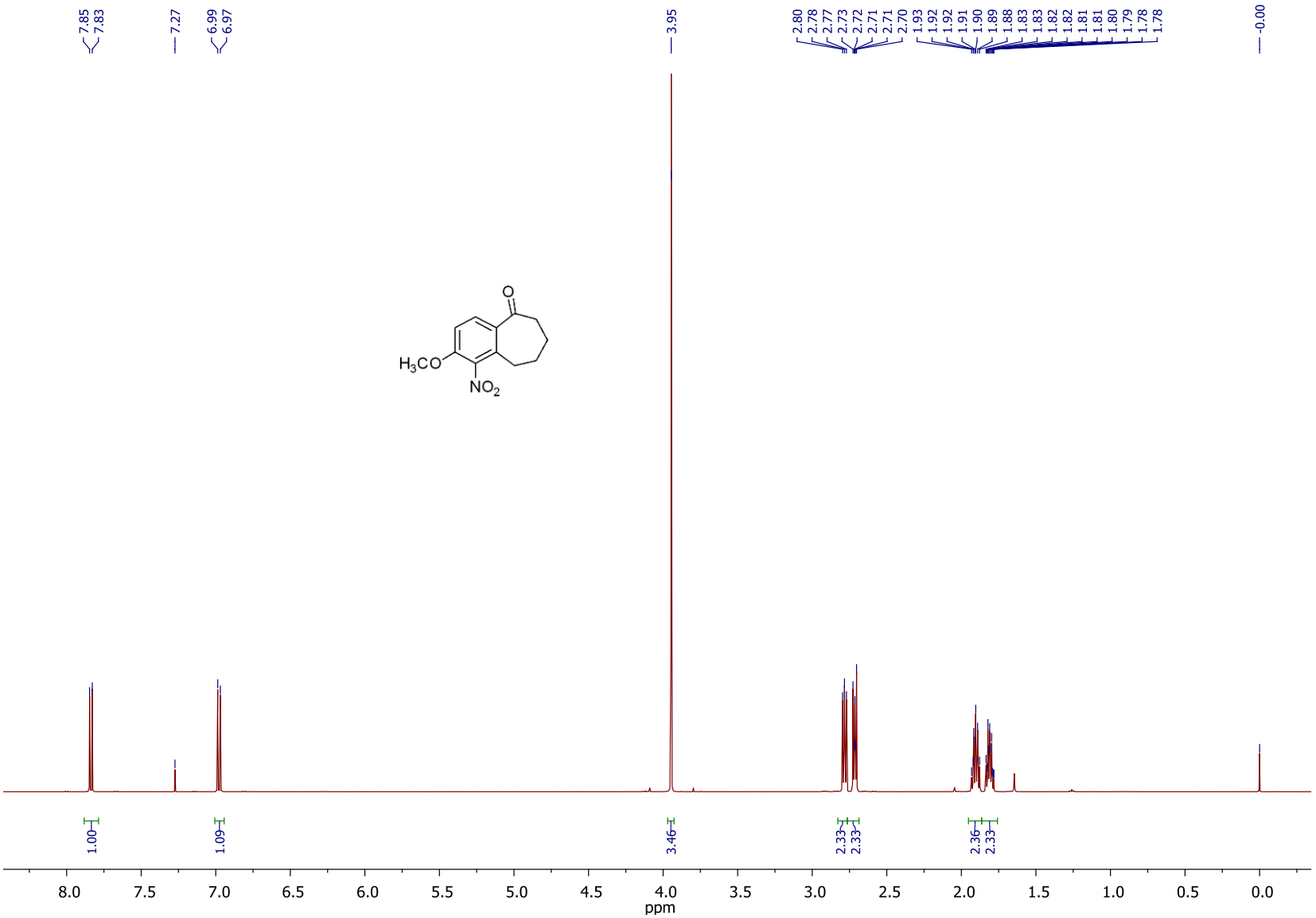
^{1}H NMR ($CDCl_3$, 500 MHz) Compound **28**



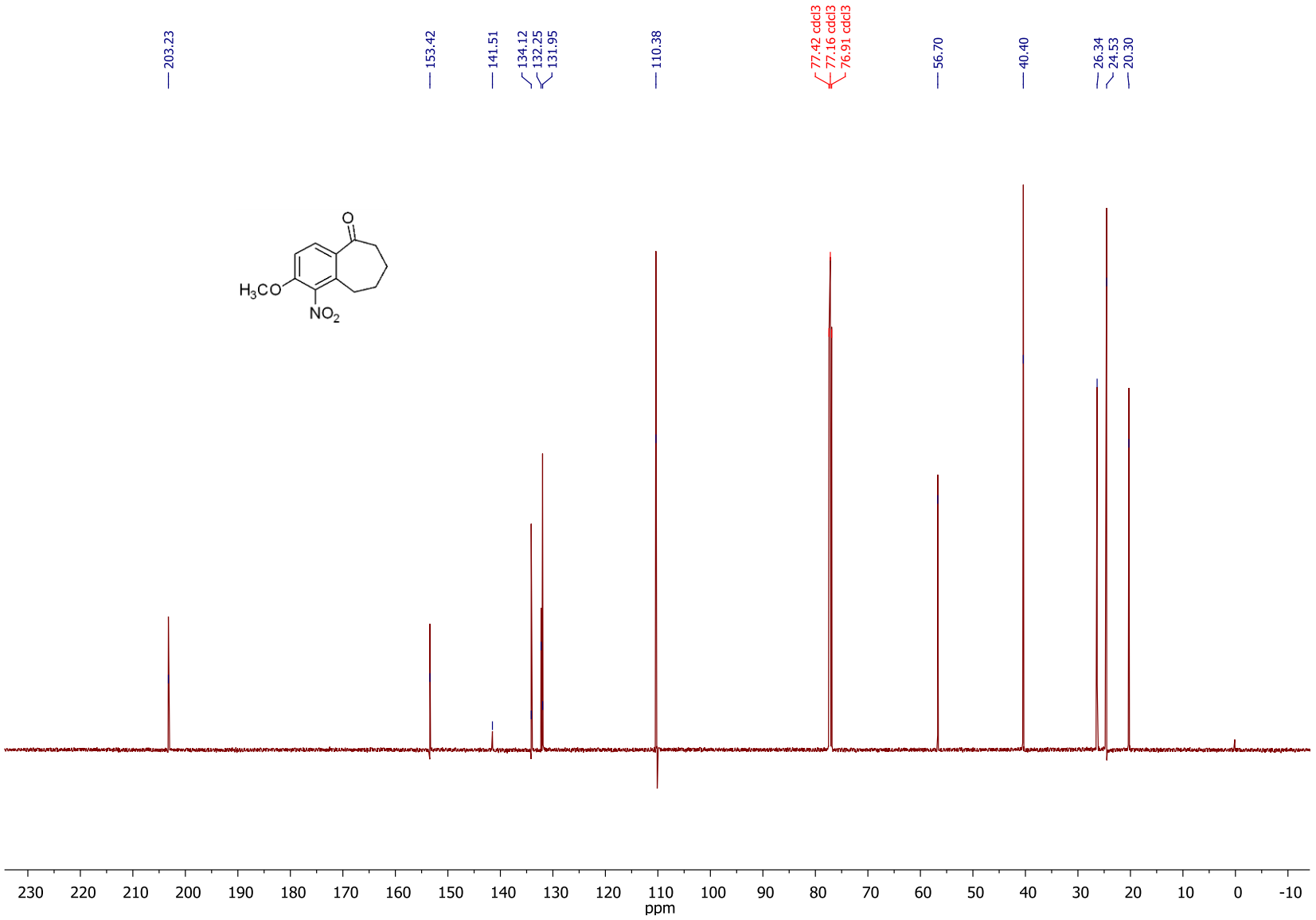
^{13}C NMR ($CDCl_3$, 125 MHz) Compound **28**



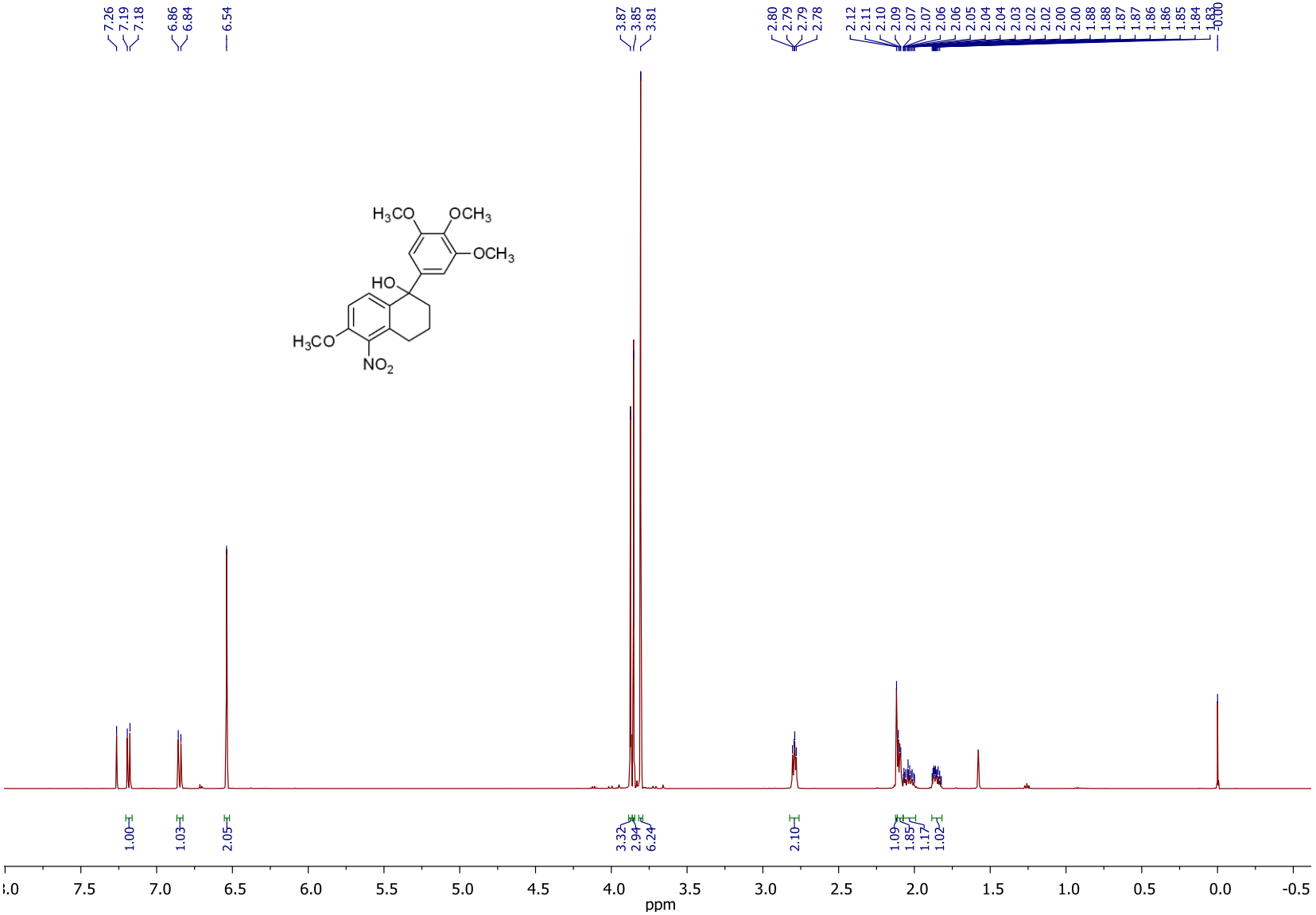
^{1}H NMR ($CDCl_3$, 500 MHz) Compound **29**



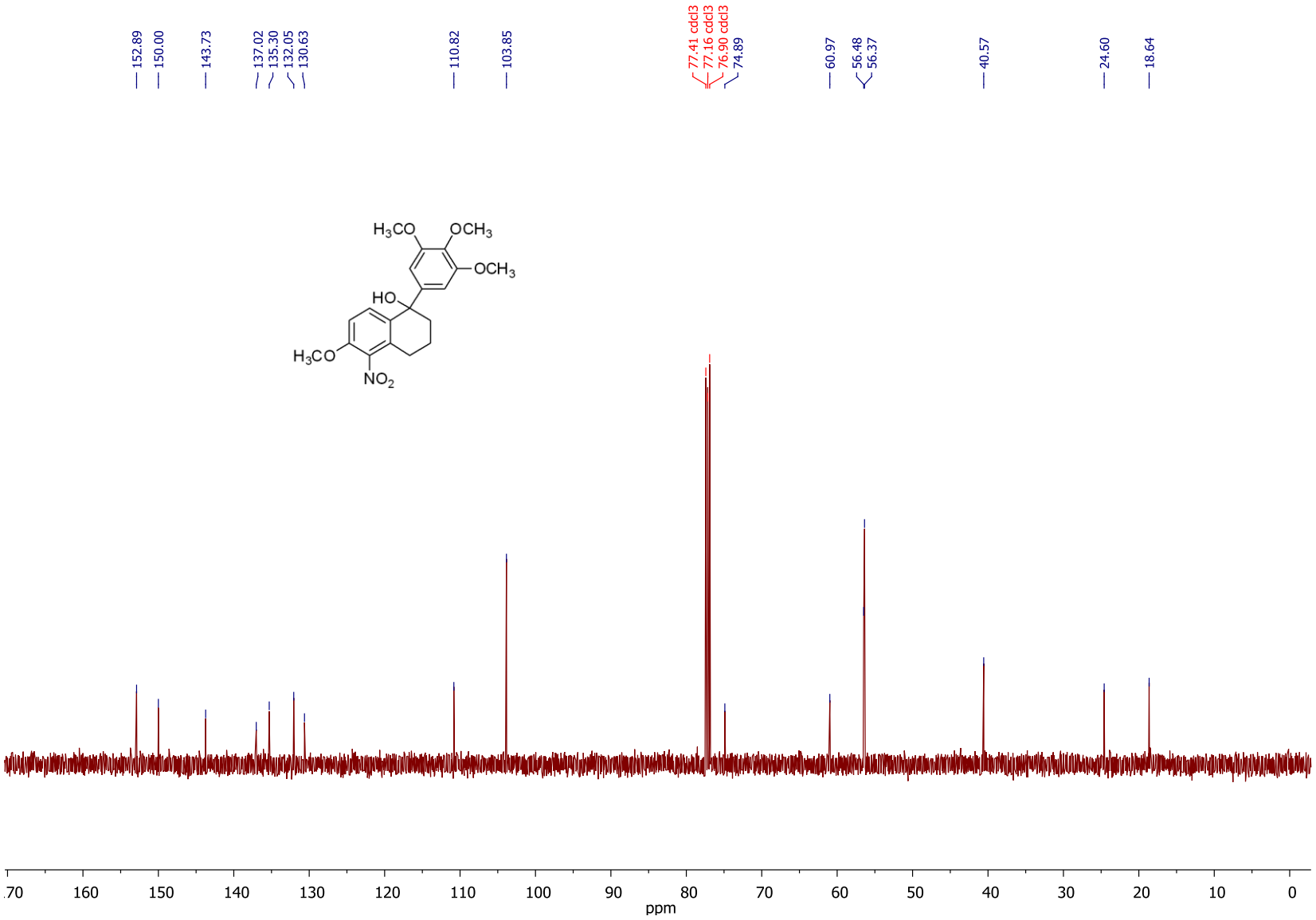
^{13}C NMR ($CDCl_3$, 125 MHz) Compound **29**



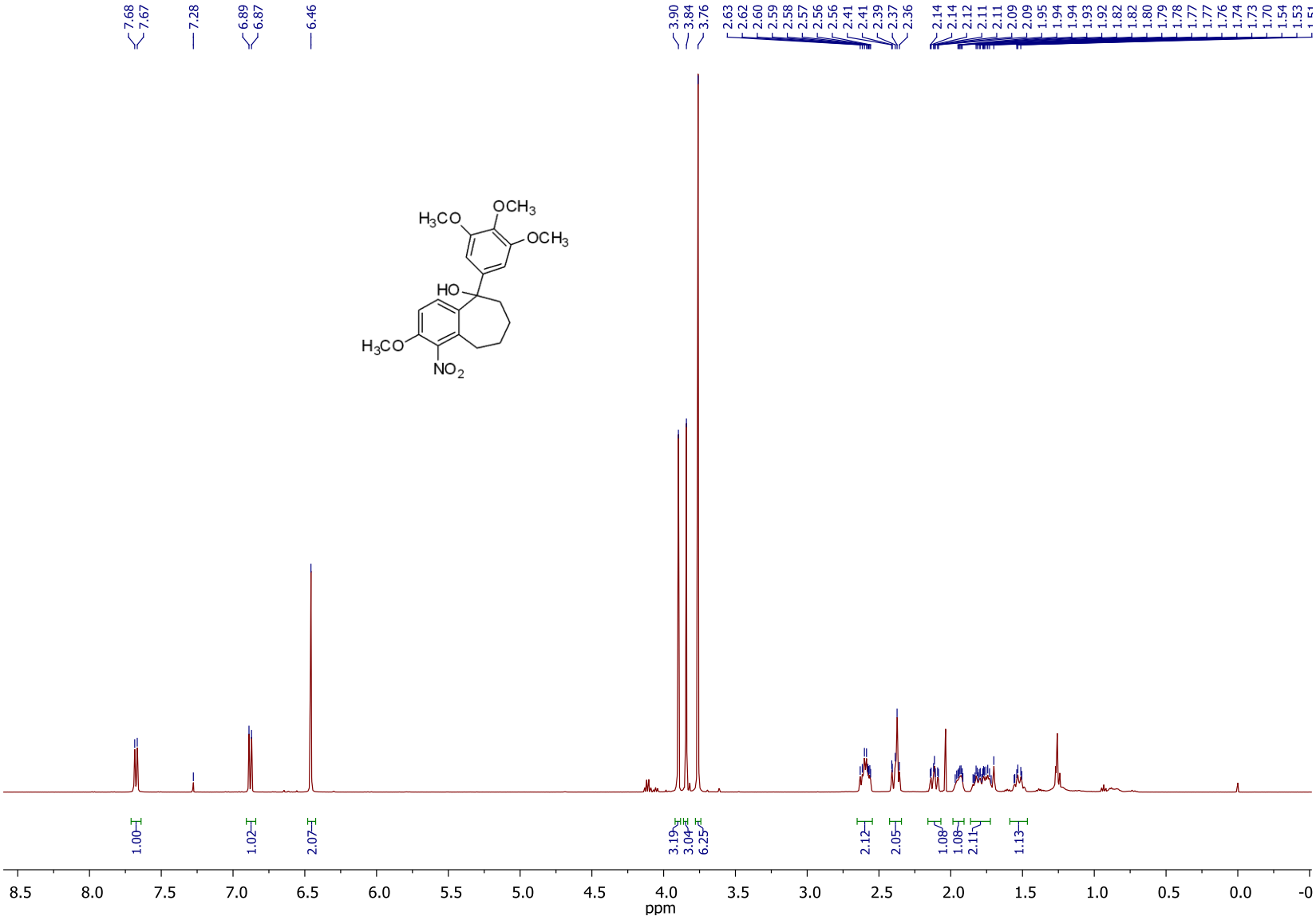
^{1}H NMR ($CDCl_3$, 500 MHz) Compound **30**



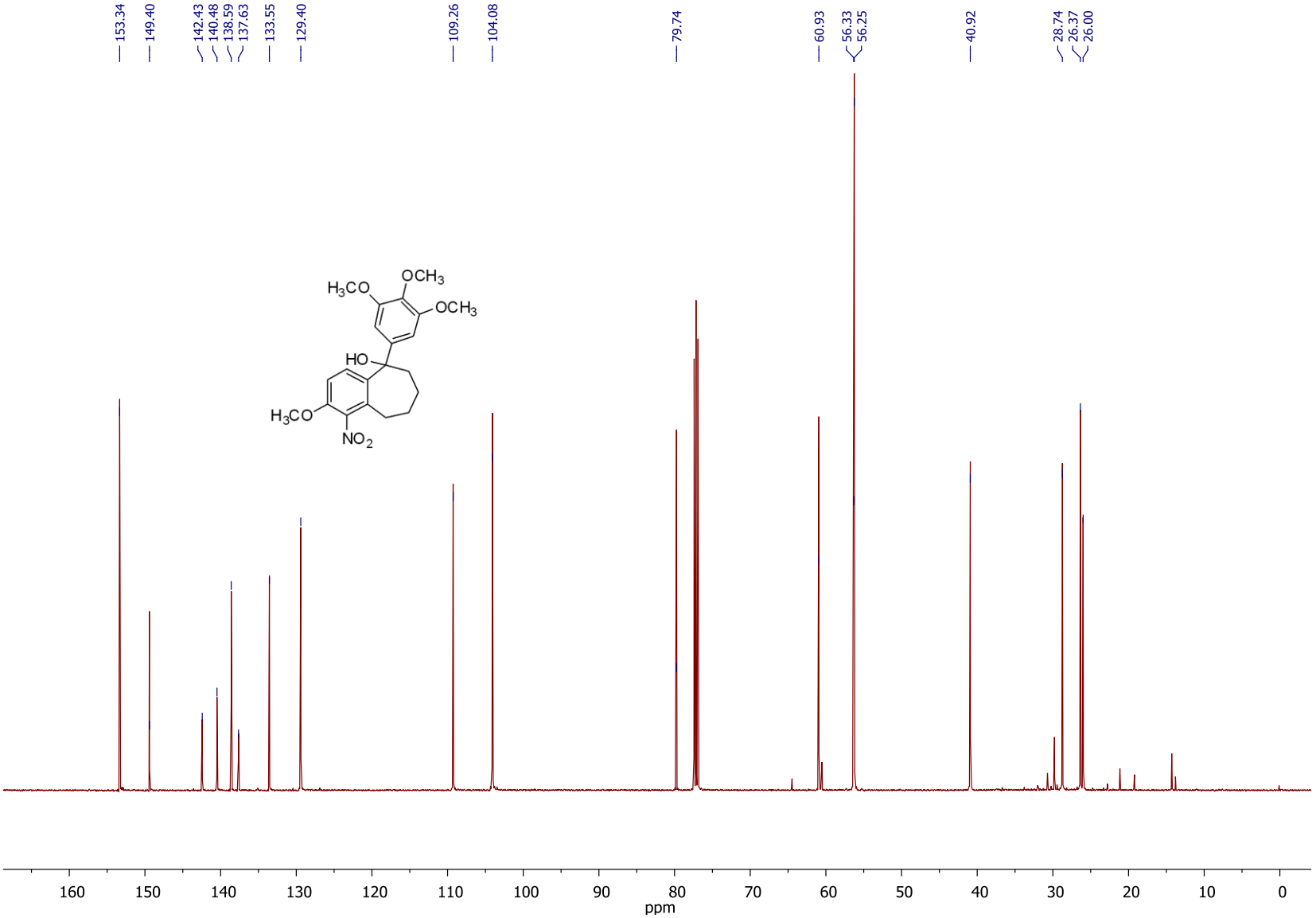
^{13}C NMR ($CDCl_3$, 125 MHz) Compound **30**



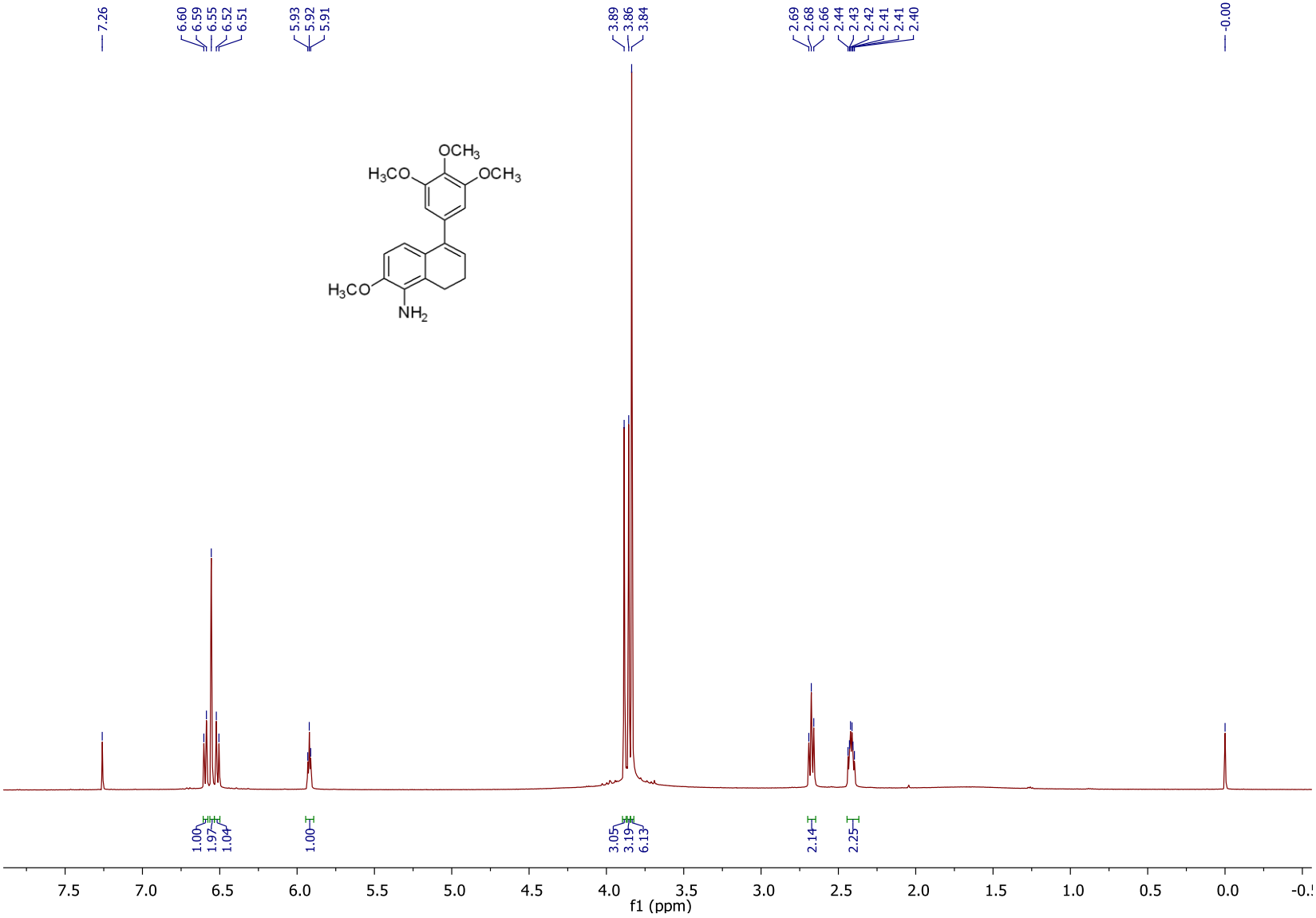
^{1}H NMR ($CDCl_3$, 500 MHz) Compound **31**



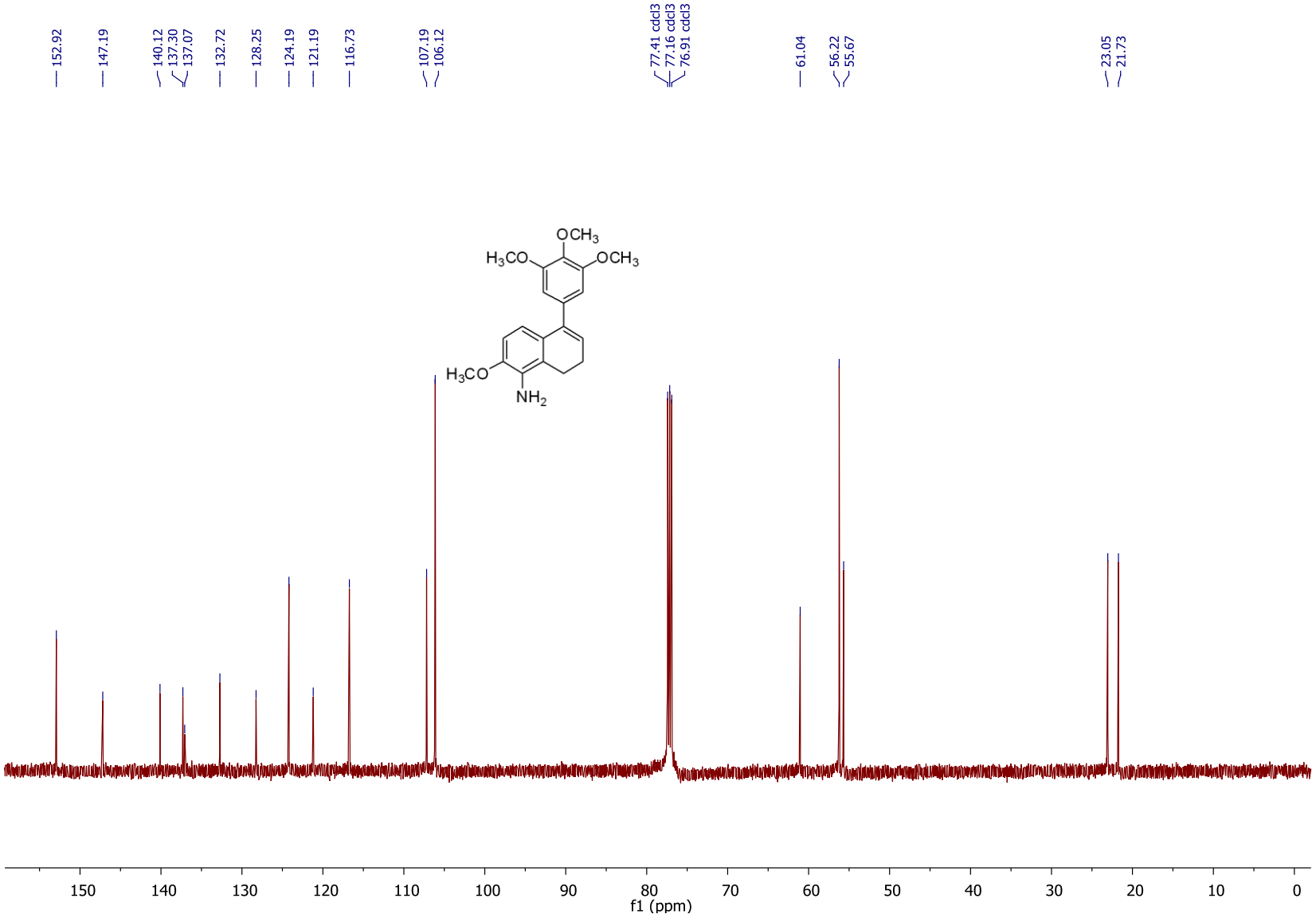
^{13}C NMR ($CDCl_3$, 125 MHz) Compound **31**



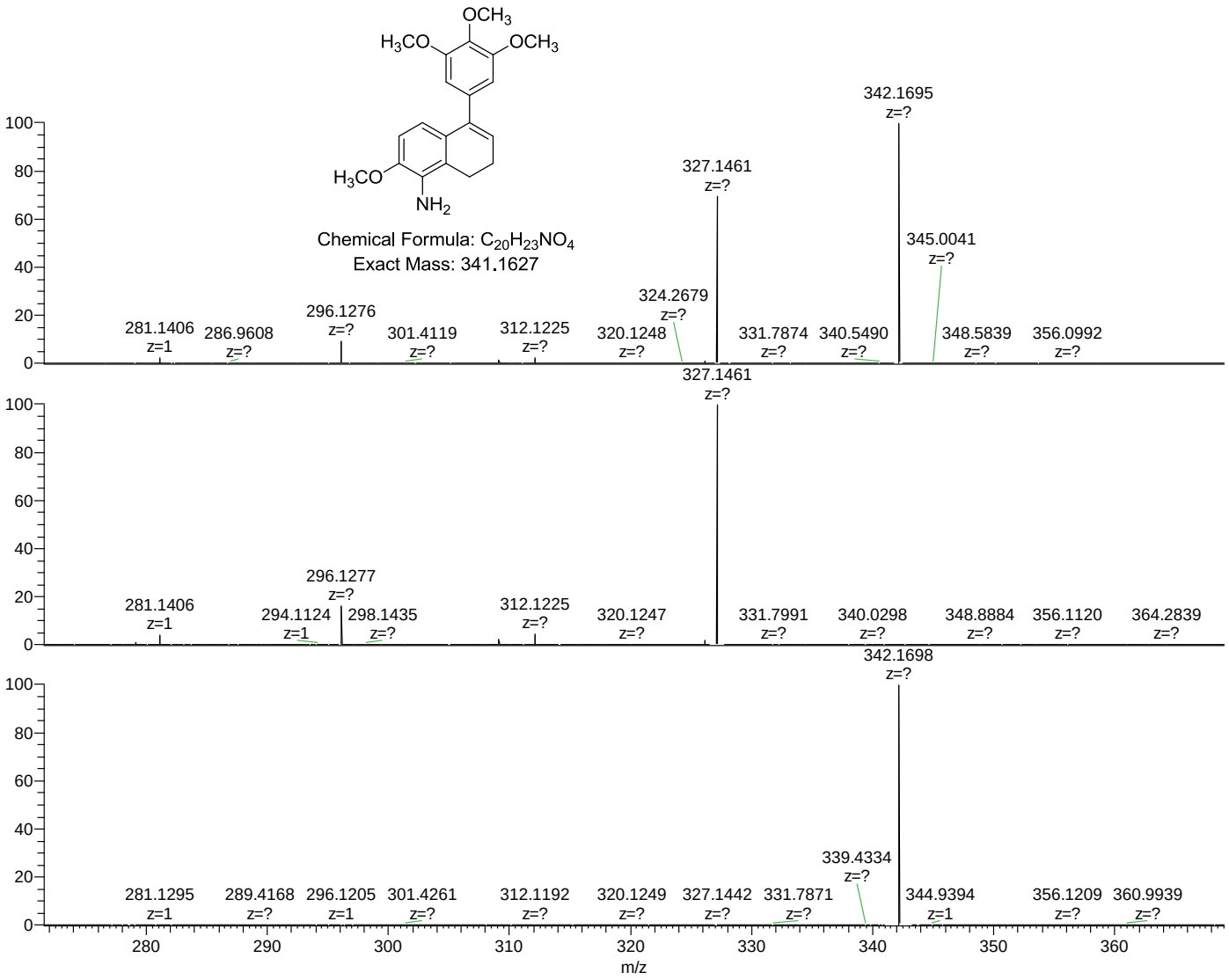
^{1}H NMR ($CDCl_3$, 500 MHz) Compound **32**



^{13}C NMR (DMSO-d6, 125 MHz) Compound **32**



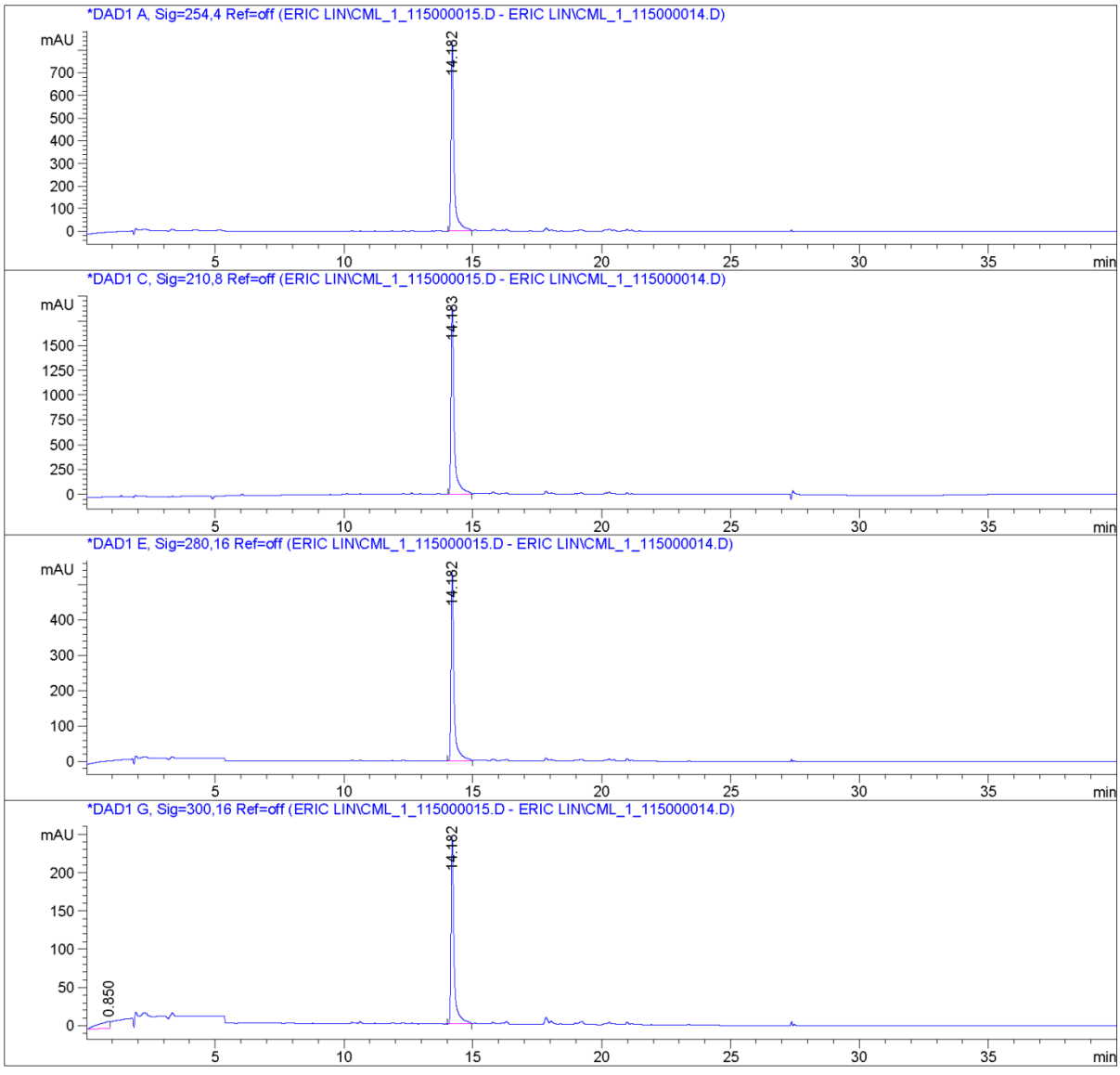
HRMS Compound **32**



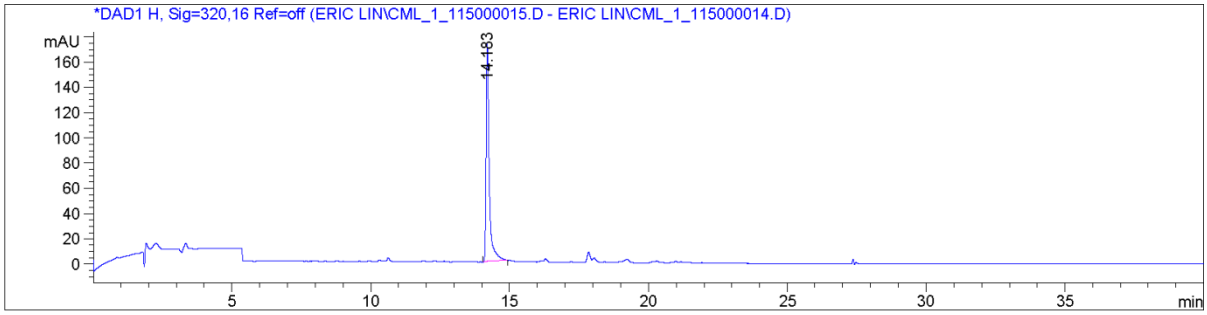
HPLC for Compound **32**

Data File C:\CHEM32\1\DATA\ERIC LIN\CML_1_115000015.D
Sample Name: CML_1_115_run1

```
==========================================================================
Acq. Operator   : ERIC LIN
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 7/24/2012 12:57:54 PM
Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed    : 7/24/2012 12:53:18 PM by ERIC LIN
Analysis Method : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed    : 8/7/2012 10:31:04 PM by ERIC LIN
                  (modified after loading)
```



Data File C:\CHEM32\1\DATA\ERIC LIN\CML_1_115000015.D
Sample Name: CML_1_115_run1




```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=off
 Signal has been modified after loading from rawdata file!

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  14.182 BB    0.1152 6719.51270  841.00653 100.0000

Totals :                  6719.51270  841.00653


Signal 2: DAD1 C, Sig=210,8 Ref=off
 Signal has been modified after loading from rawdata file!

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  14.183 VB    0.1246 1.60874e4  1902.56287 100.0000

Totals :                  1.60874e4  1902.56287


Signal 3: DAD1 E, Sig=280,16 Ref=off
 Signal has been modified after loading from rawdata file!
```

Data File C:\CHEM32\1\DATA\ERIC LIN\CML_1_115000015.D
Sample Name: CML_1_115_run1

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.182	BB	0.1150	4291.95557	538.29181	100.0000
Totals :				4291.95557	538.29181	

Signal 4: DAD1 G, Sig=300,16 Ref=off
Signal has been modified after loading from rawdata file!

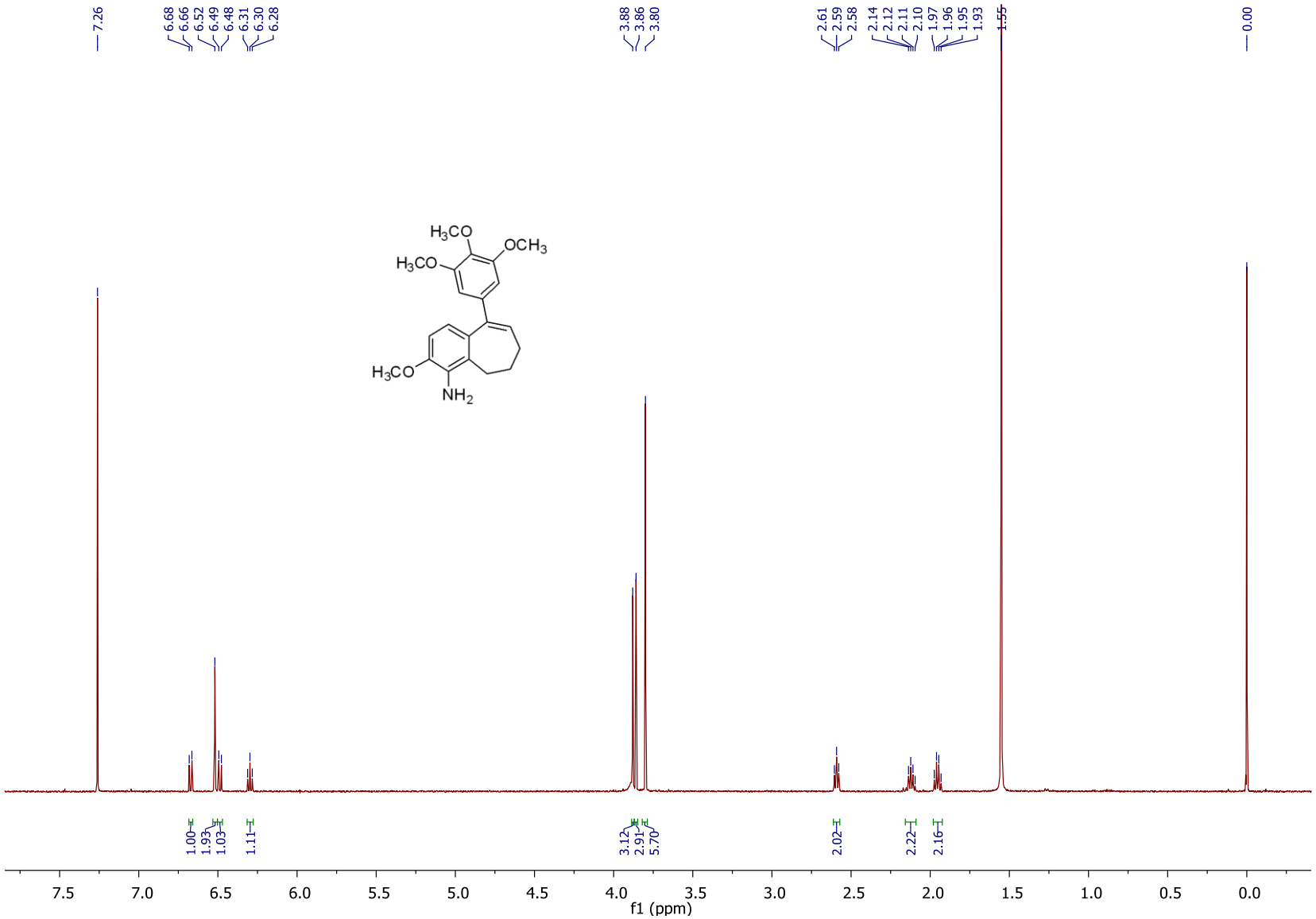
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	0.850	BV	0.4104	317.42105	9.63334	13.9350
2	14.182	BB	0.1150	1960.44995	245.89526	86.0650
Totals :				2277.87100	255.52861	

Signal 5: DAD1 H, Sig=320,16 Ref=off
Signal has been modified after loading from rawdata file!

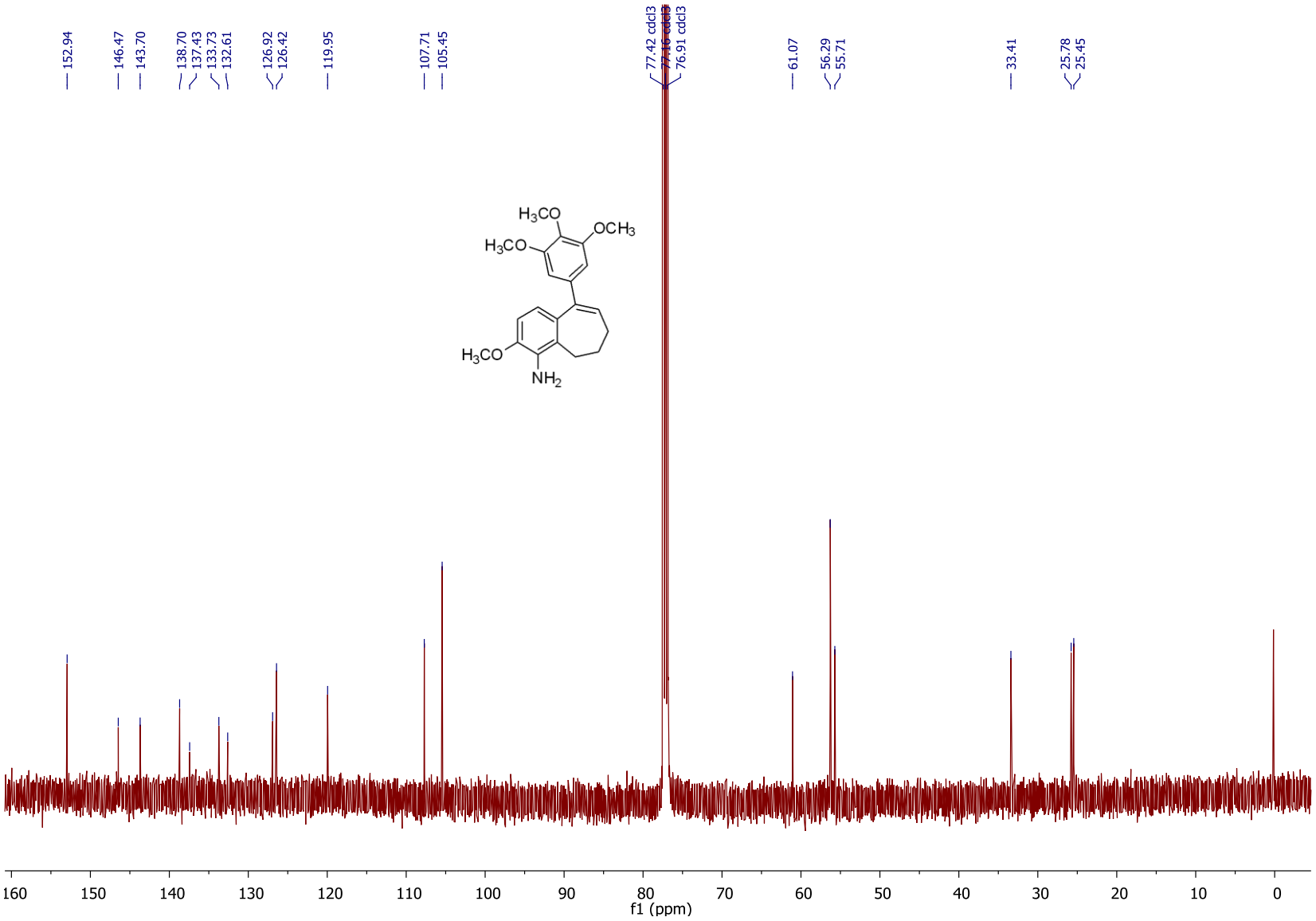
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.183	BB	0.1132	1353.12427	173.10461	100.0000
Totals :				1353.12427	173.10461	

*** End of Report ***

^{1}H NMR ($CDCl_3$, 500 MHz) Compound **33**



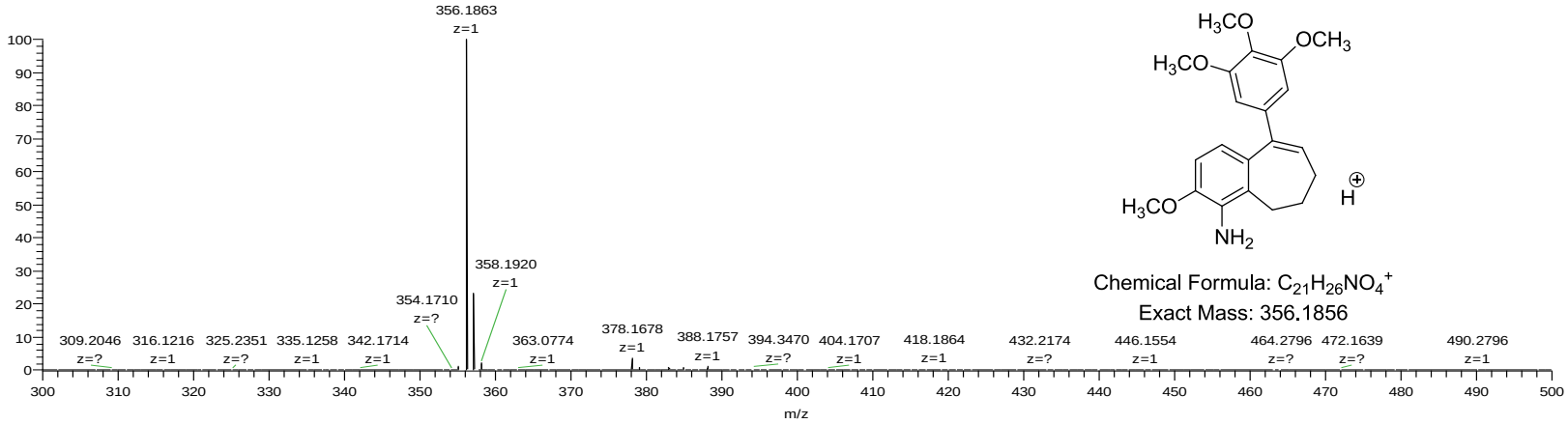
^{13}C NMR ($CDCl_3$, 125 MHz) Compound **33**



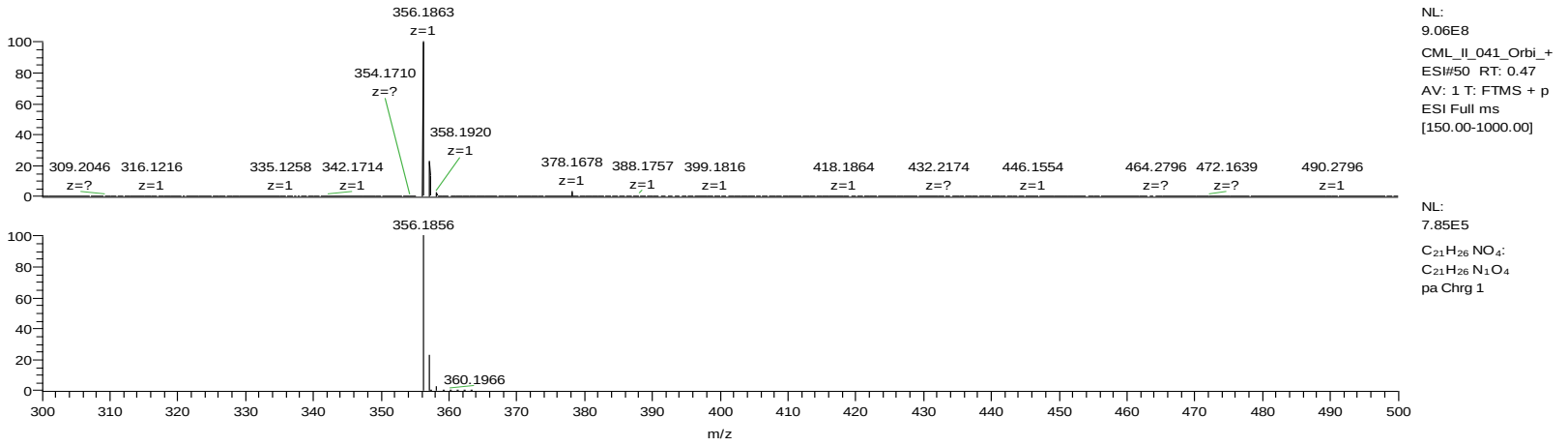
HRMS Compound **33**

C:\Xcalibur\...\CML_II_041_Orbi_+ESI 8/21/2013 4:30:17 PM CML_II_041

CML_II_041_Orbi_+ESI #50 RT: 0.47 AV: 1 NL:
T: FTMS + p ESI Full ms [150.00-1000.00]



Chemical Formula: $C_{21}H_{26}NO_4^+$
Exact Mass: 356.1856



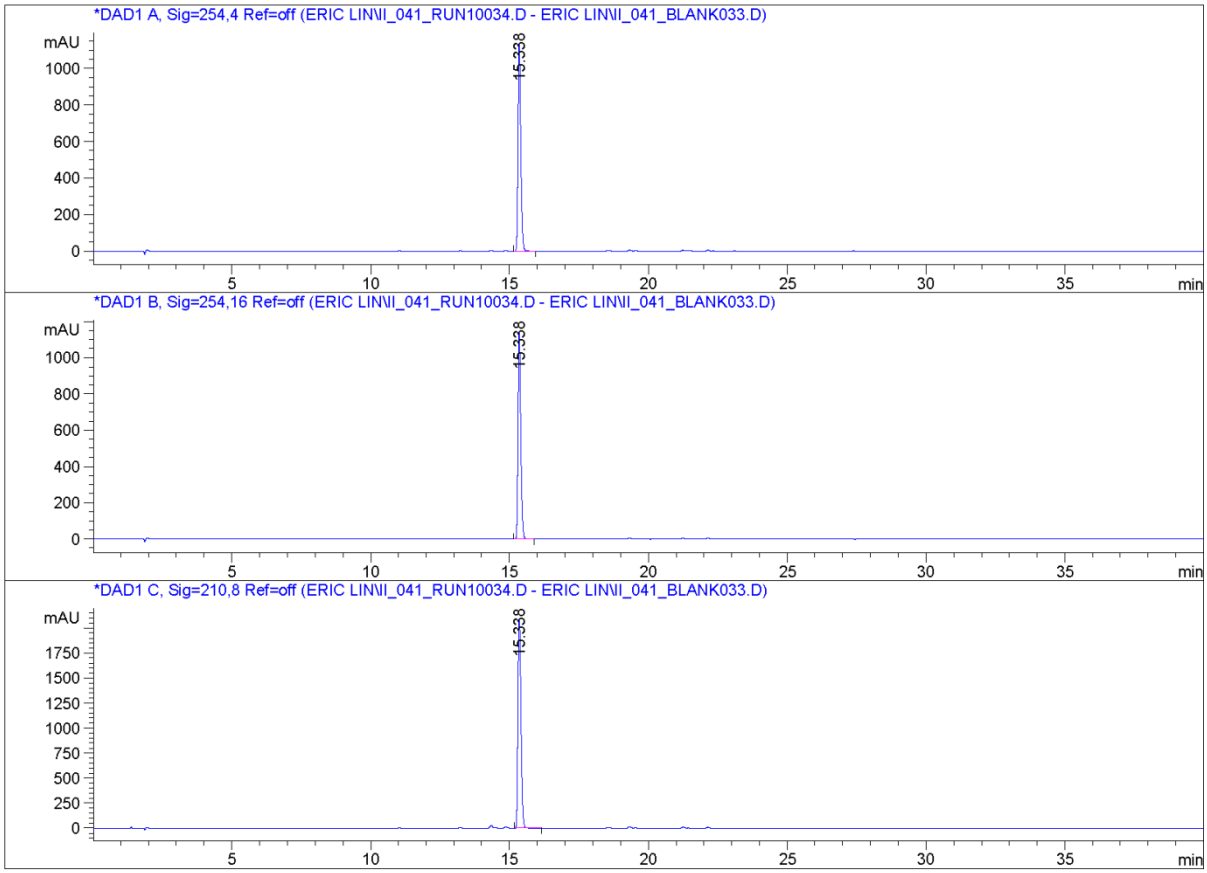
NL:
9.06E8
CML_II_041_Orbi_+
ESI#50 RT: 0.47
AV: 1 T: FTMS + p
ESI Full ms
[150.00-1000.00]

NL:
7.85E5
$C_{21}H_{26}NO_4$:
$C_{21}H_{26}N_1O_4$
pa Chrg 1

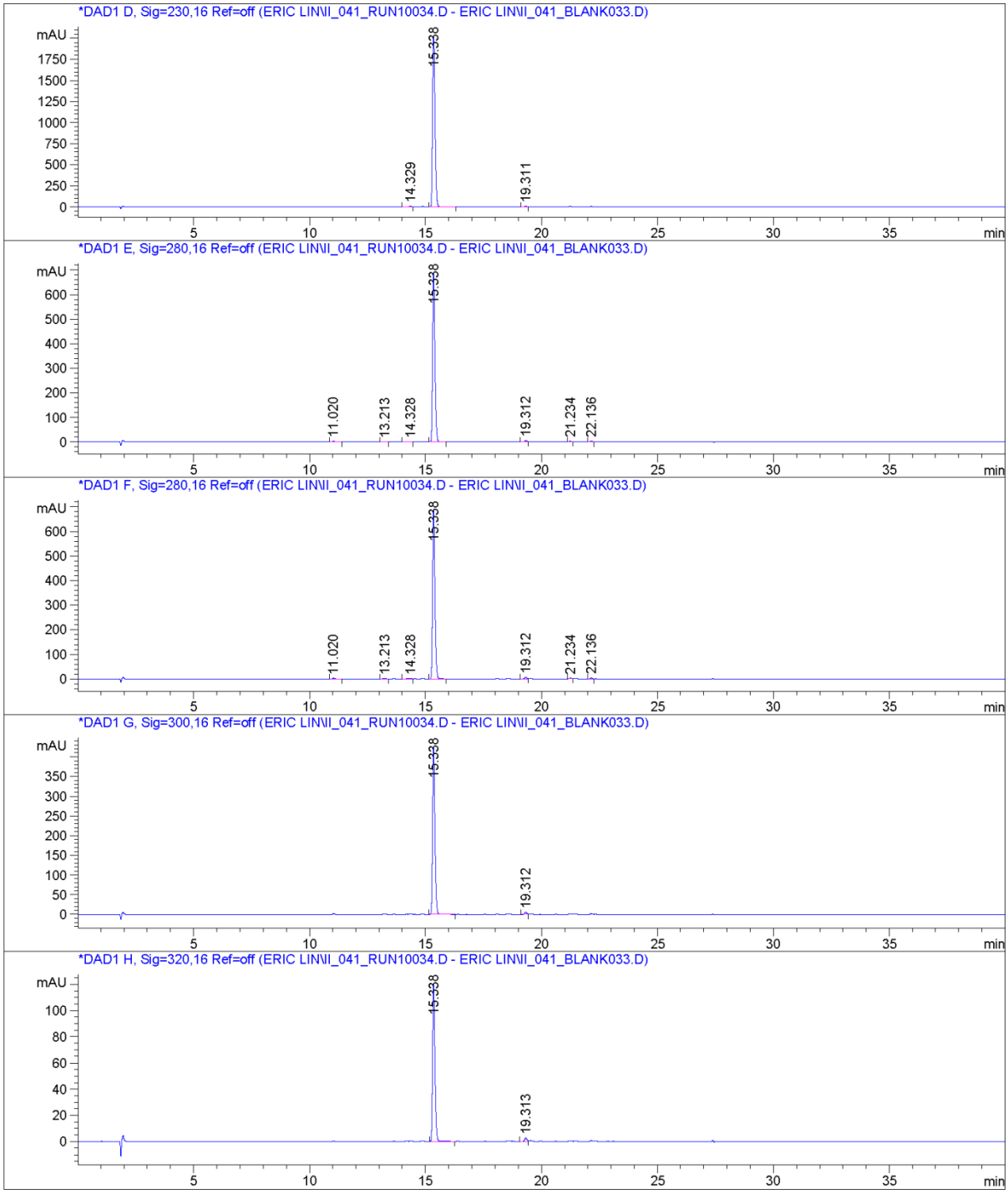
HPLC for Compound **33**

Data File C:\CHEM32\1\DATA\ERIC LIN\II_041_RUN10034.D
Sample Name: CML_II_041_run1

```
==========================================================================
Acq. Operator   : ERIC LIN
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 10/3/2012 10:17:19 PM
Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed    : 10/3/2012 10:08:22 PM by ERIC LIN
                  (modified after loading)
Analysis Method : C:\CHEM32\1\DATA\ERIC LIN\II_041_RUN10034.D\DA.M (MASTERMETHOD.M)
Last changed    : 10/3/2012 11:09:57 PM by Matt Mac
Sample Info     :
```



Data File C:\CHEM32\1\DATA\ERIC LIN\II_041_RUN10034.D
Sample Name: CML_II_041_run1



Data File C:\CHEM32\1\DATA\ERIC LIN\II_041_RUN10034.D
Sample Name: CML_II_041_run1

==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.338	BB	0.1028	7705.96338	1140.77222	100.0000
Totals :				7705.96338	1140.77222	

Signal 2: DAD1 B, Sig=254,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.338	BB	0.1028	7757.90479	1148.84033	100.0000
Totals :				7757.90479	1148.84033	

Signal 3: DAD1 C, Sig=210,8 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.338	BB	0.1211	1.59885e4	2092.96558	100.0000
Totals :				1.59885e4	2092.96558	

Signal 4: DAD1 D, Sig=230,16 Ref=off
Signal has been modified after loading from rawdata file!

Data File C:\CHEM32\1\DATA\ERIC LIN\II_041_RUN10034.D
Sample Name: CML_II_041_run1

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.329	BV	0.1151	81.64388	10.45497	0.5524
2	15.338	BB	0.1118	1.46307e4	2036.75854	98.9872
3	19.311	BV	0.1073	68.05374	9.76025	0.4604
Totals :				1.47804e4	2056.97377	

Signal 5: DAD1 E, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.020	BB	0.0916	21.07620	3.53085	0.4414
2	13.213	BB	0.1162	14.41269	1.78559	0.3019
3	14.328	BB	0.1234	15.15916	1.77981	0.3175
4	15.338	BB	0.1024	4643.60498	691.30487	97.2565
5	19.312	BV	0.1072	40.08195	5.75978	0.8395
6	21.234	BB	0.0913	17.97745	3.02522	0.3765
7	22.136	BV	0.0880	22.28565	3.93942	0.4668
Totals :				4774.59807	711.12554	

Signal 6: DAD1 F, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.020	BB	0.0916	21.07620	3.53085	0.4414
2	13.213	BB	0.1162	14.41269	1.78559	0.3019
3	14.328	BB	0.1234	15.15916	1.77981	0.3175
4	15.338	BB	0.1024	4643.60498	691.30487	97.2565
5	19.312	BV	0.1072	40.08195	5.75978	0.8395
6	21.234	BB	0.0913	17.97745	3.02522	0.3765
7	22.136	BV	0.0880	22.28565	3.93942	0.4668
Totals :				4774.59807	711.12554	

Signal 7: DAD1 G, Sig=300,16 Ref=off
Signal has been modified after loading from rawdata file!

Data File C:\CHEM32\1\DATA\ERIC LIN\II_041_RUN10034.D
Sample Name: CML_II_041_run1

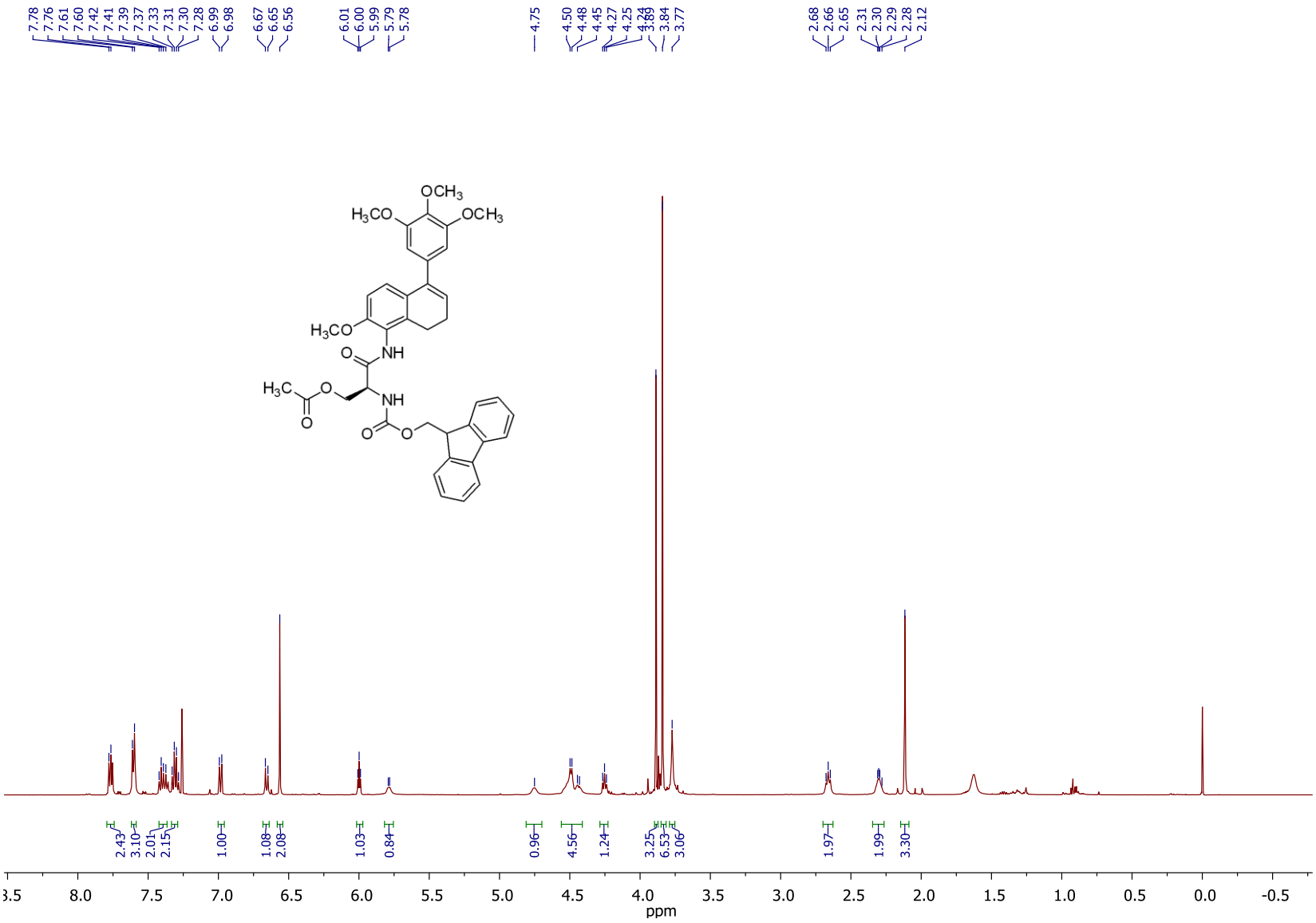
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.338	BB	0.1025	2873.33496	427.19150	98.6222
2	19.312	BV	0.1067	40.14083	5.80112	1.3778
Totals :				2913.47579	432.99261	

Signal 8: DAD1 H, Sig=320,16 Ref=off
Signal has been modified after loading from rawdata file!

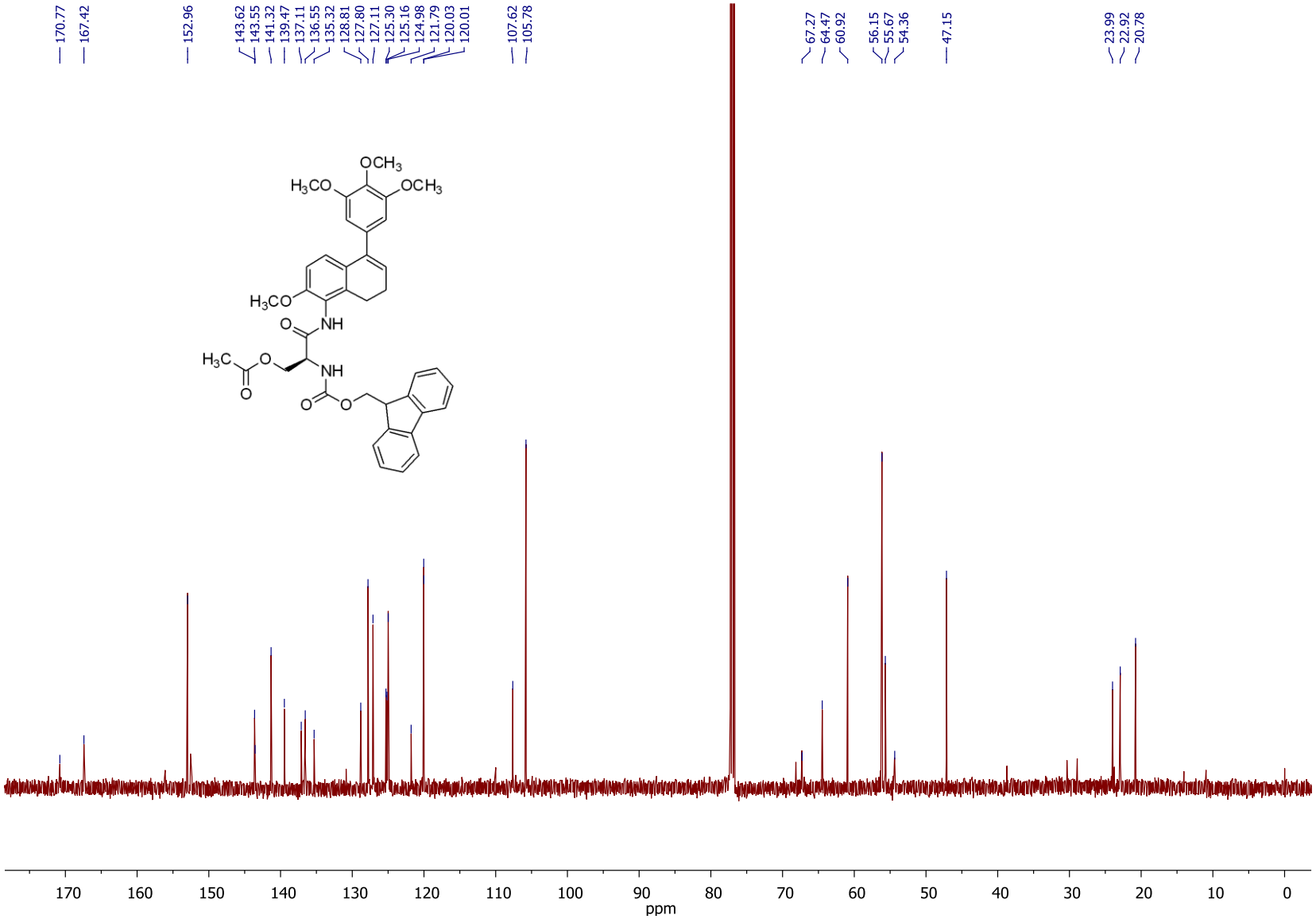
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.338	BB	0.1027	814.71570	120.79123	97.7850
2	19.313	BV	0.1074	18.45501	2.64384	2.2150
Totals :				833.17071	123.43507	

*** End of Report ***

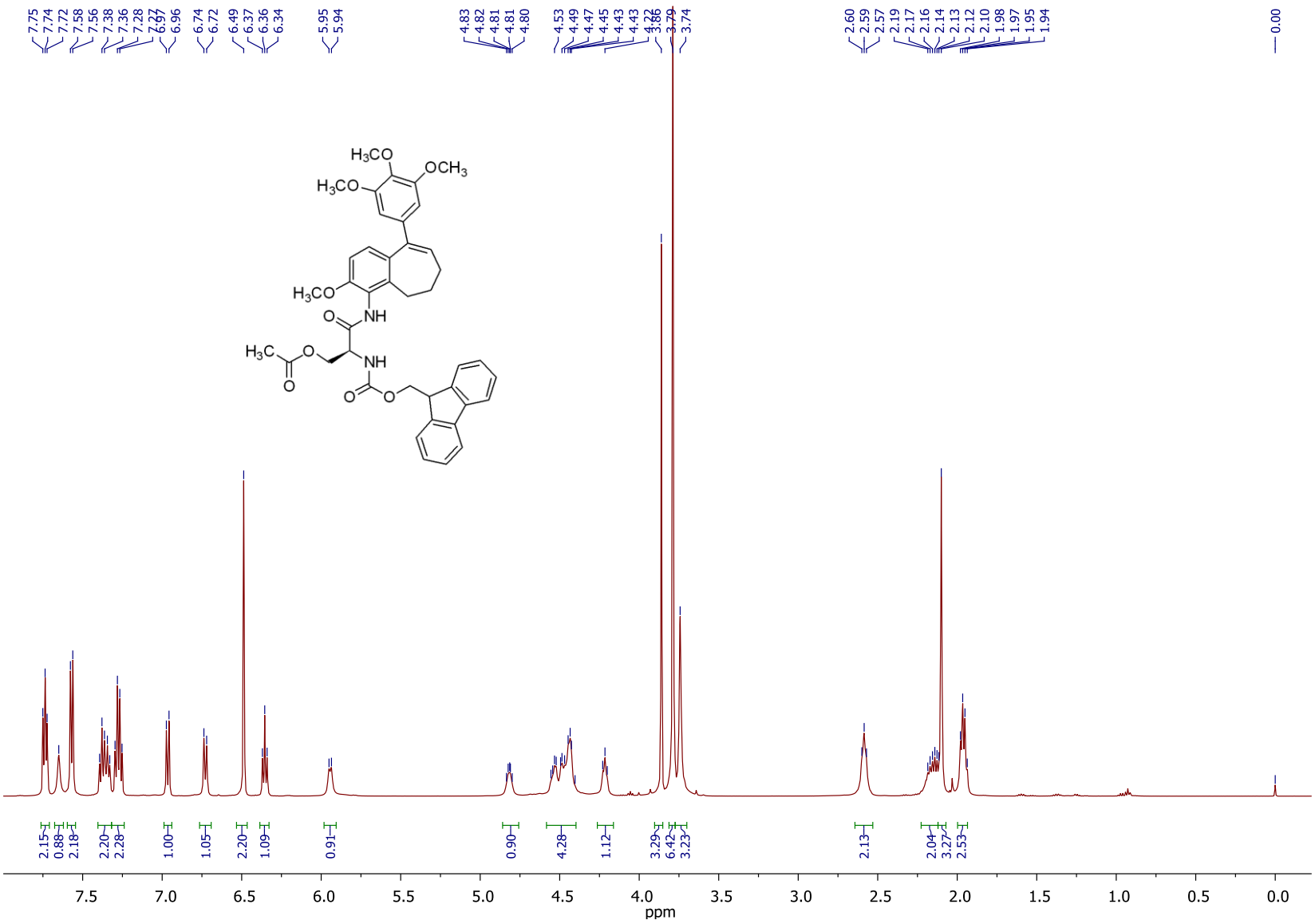
^{1}H NMR ($CDCl_3$, 500 MHz) Compound **34**



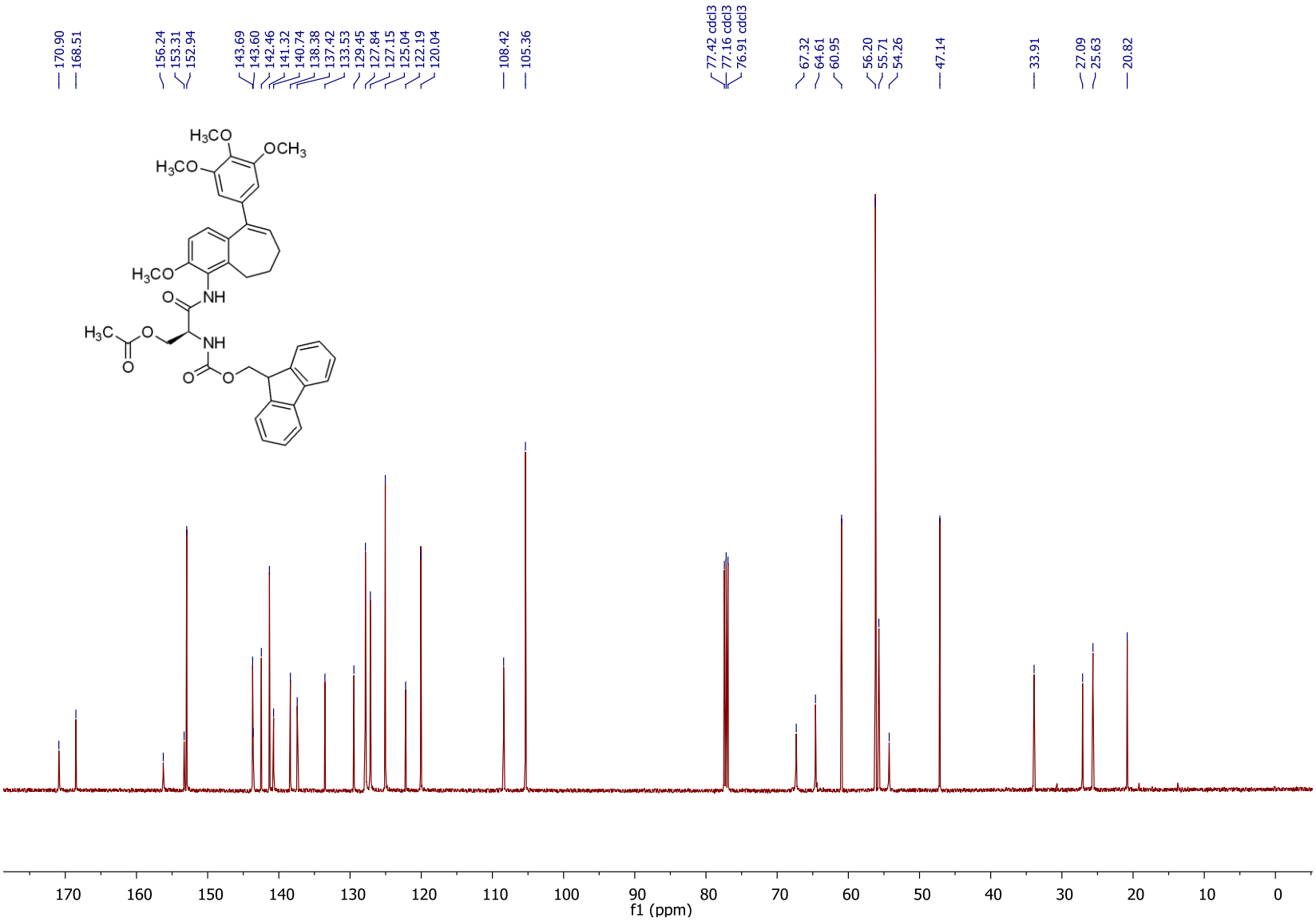
^{13}C NMR ($CDCl_3$, 125 MHz) Compound **34**



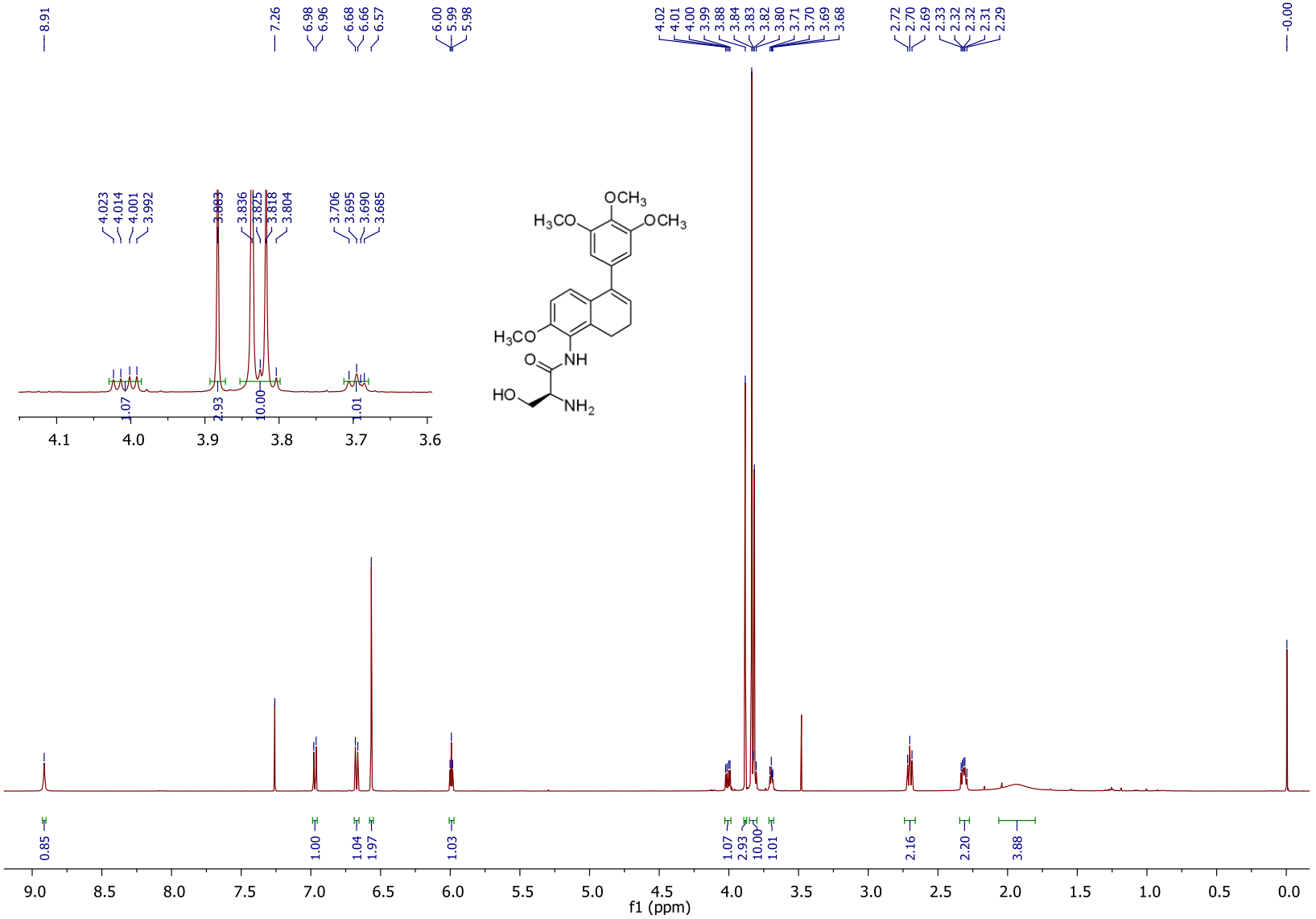
^{1}H NMR ($CDCl_3$, 500 MHz) Compound **35**



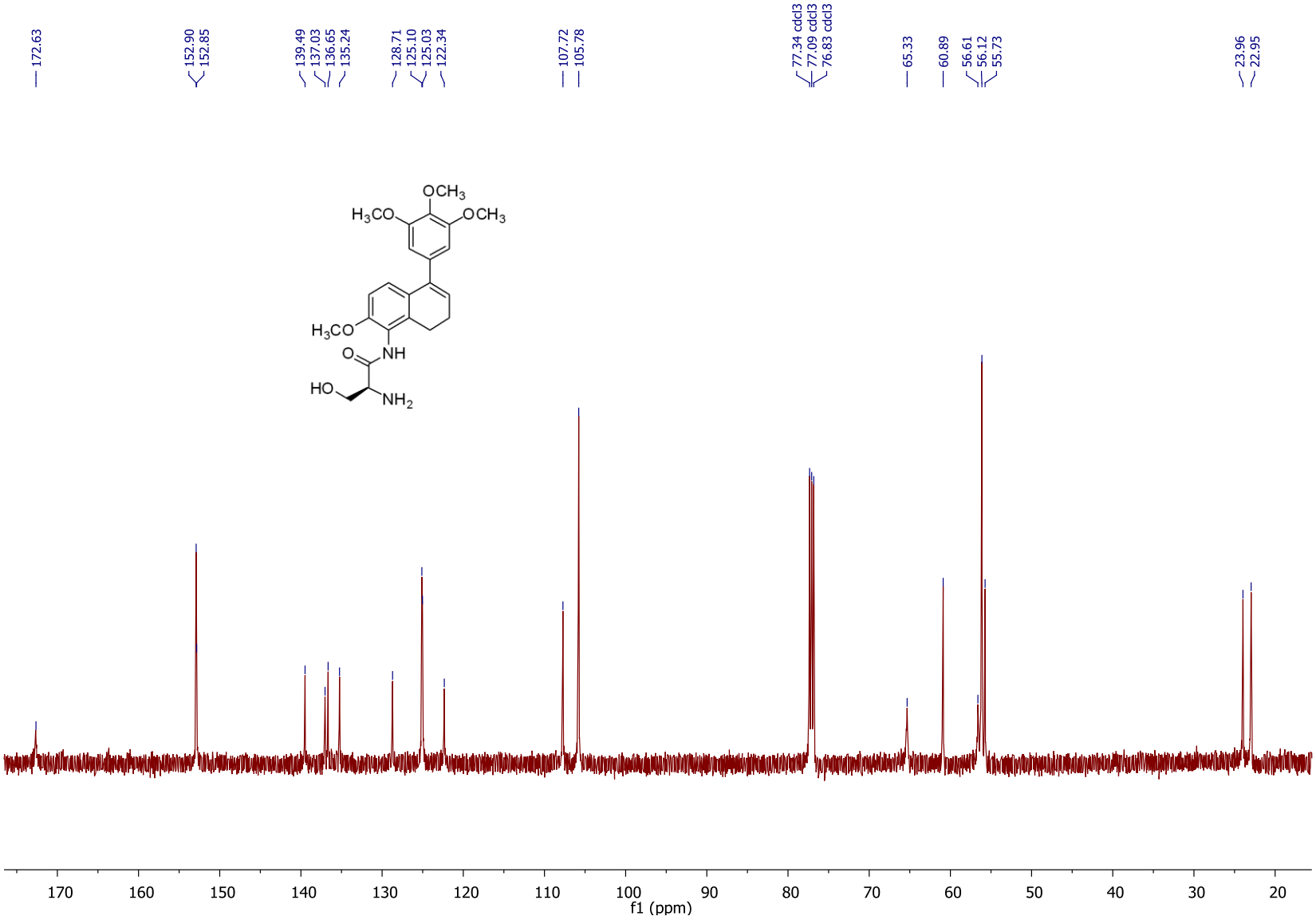
^{13}C NMR ($CDCl_3$, 125 MHz) Compound **35**



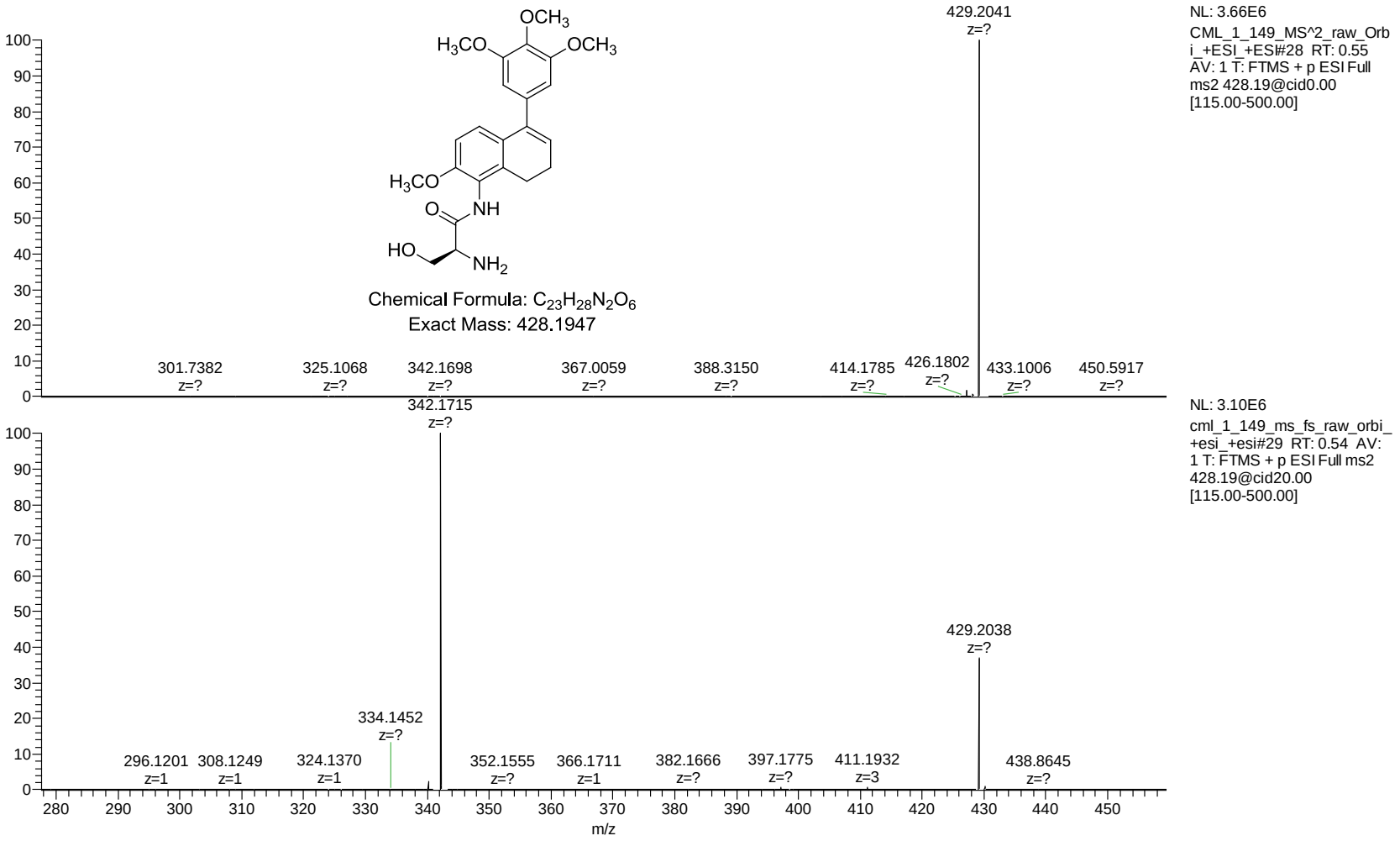
^{1}H NMR ($CDCl_3$, 500 MHz) Compound **36**



^{13}C NMR ($CDCl_3$, 125 MHz) Compound **36**



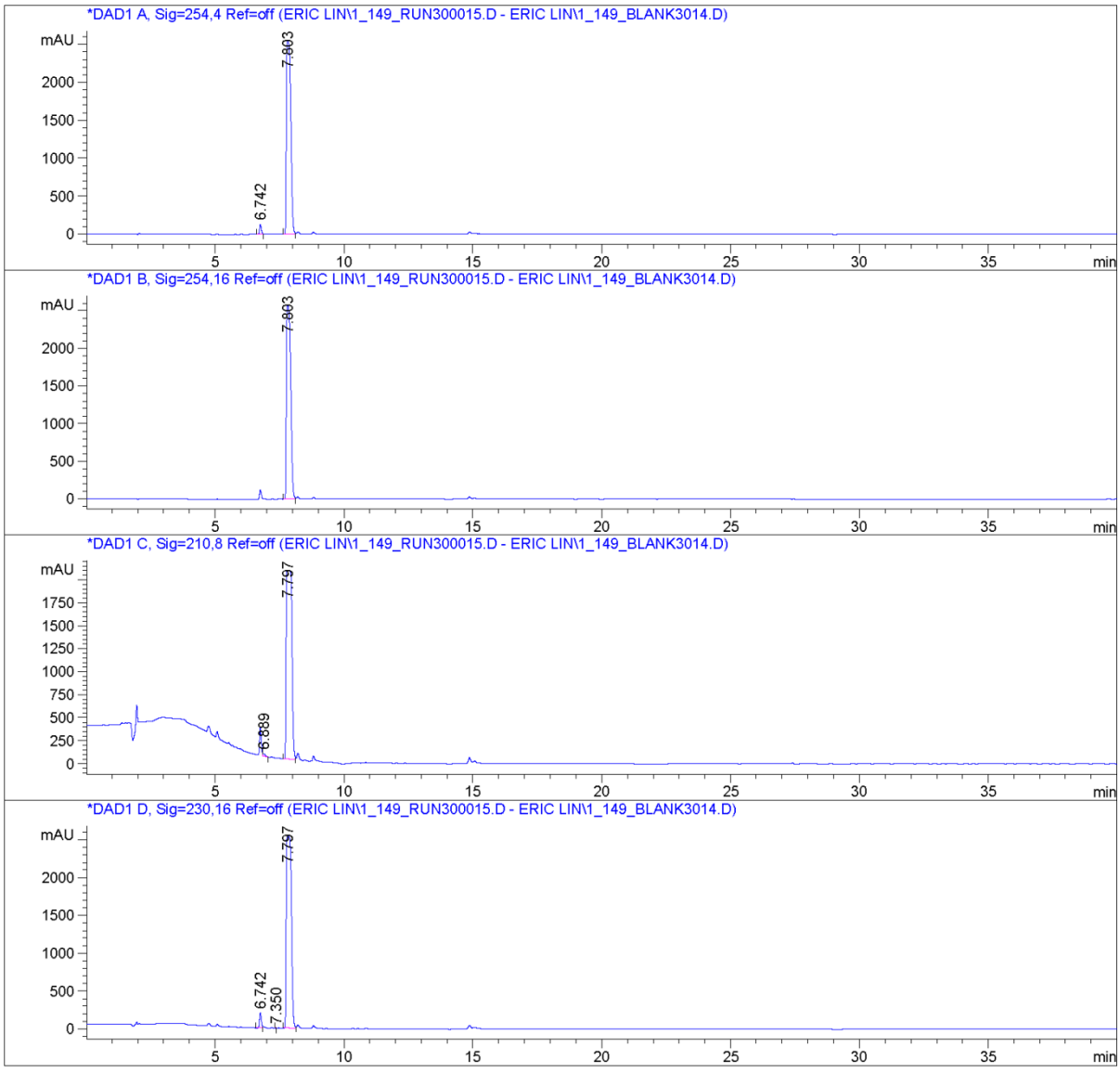
HRMS Compound **36**



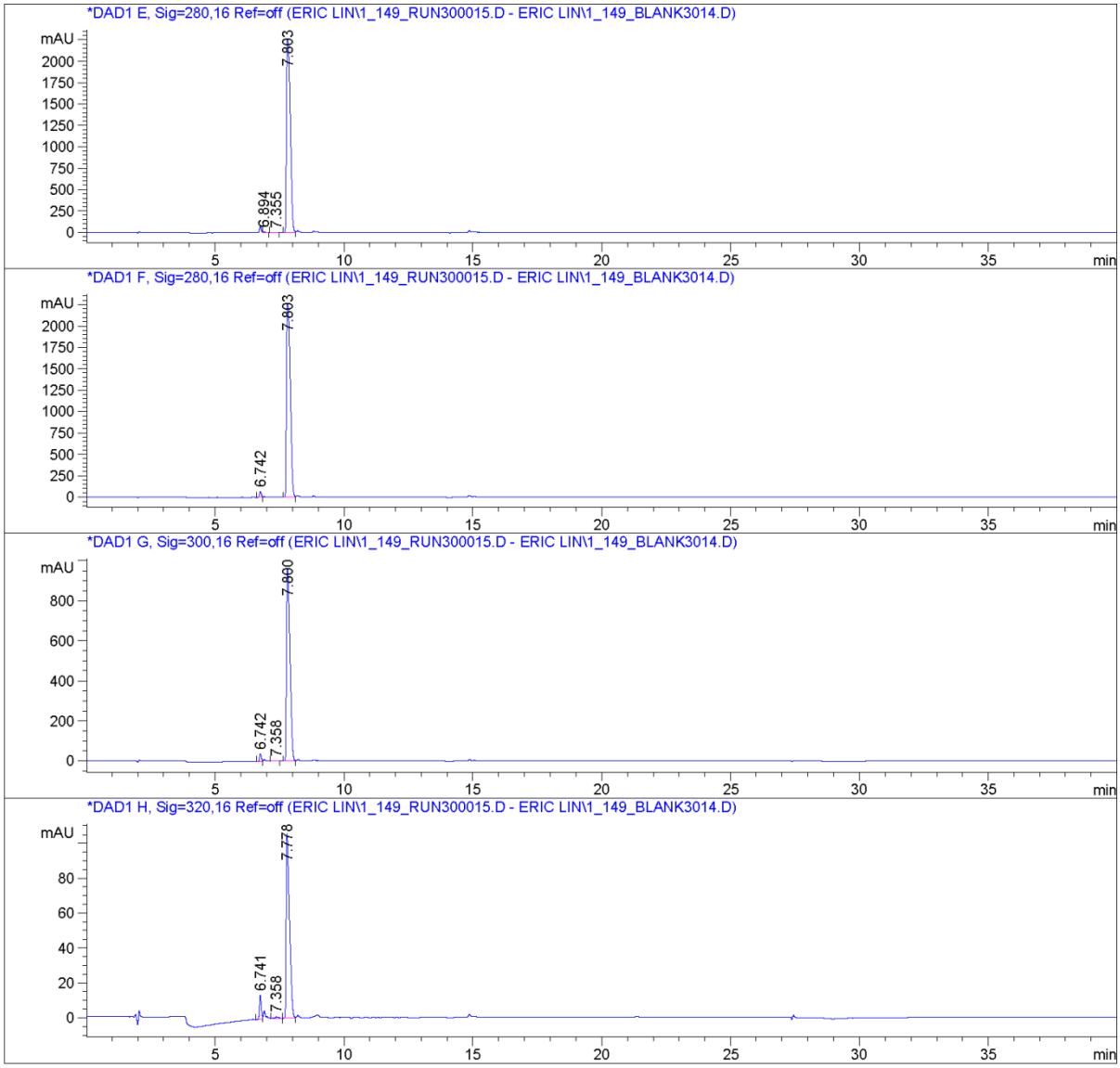
HPLC for Compound **36**

Data File C:\CHEM32\1\DATA\ERIC LIN\1_149_RUN300015.D
Sample Name: CML_1_149_run3

```
==========================================================================
Acq. Operator   : ERIC LIN
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 8/6/2012 11:41:44 AM
Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed    : 8/6/2012 11:35:23 AM by ERIC LIN
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed    : 3/28/2012 11:50:33 AM by Erica P
```



Data File C:\CHEM32\1\DATA\ERIC LIN\1_149_RUN300015.D
Sample Name: CML_1_149_run3



==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Data File C:\CHEM32\1\DATA\ERIC LIN\1_149_RUN300015.D
Sample Name: CML_1_149_run3

Signal 1: DAD1 A, Sig=254,4 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.742	VV	0.0717	625.44885	131.10748	2.0644
2	7.803	BV	0.1916	2.96707e4	2551.87720	97.9356
Totals :				3.02962e4	2682.98468	

Signal 2: DAD1 B, Sig=254,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.803	BV	0.1880	2.95208e4	2569.66016	100.0000
Totals :				2.95208e4	2569.66016	

Signal 3: DAD1 C, Sig=210,8 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.889	VB	0.0780	144.33923	27.16252	0.4637
2	7.797	BV	0.2001	3.09806e4	2052.53906	99.5363
Totals :				3.11250e4	2079.70159	

Signal 4: DAD1 D, Sig=230,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.742	BV	0.0721	968.08734	201.15717	2.7443
2	7.350	VV	0.0622	9.36047	2.17735	0.0265
3	7.797	BV	0.2211	3.42987e4	2547.35425	97.2292
Totals :				3.52762e4	2750.68876	

Data File C:\CHEM32\1\DATA\ERIC LIN\1_149_RUN300015.D
Sample Name: CML_1_149_run3

Signal 5: DAD1 E, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.894	VB	0.0813	45.02870	8.04697	0.1943
2	7.355	BV	0.1814	15.41385	1.07444	0.0665
3	7.803	BV	0.1693	2.31112e4	2259.27368	99.7392
Totals :				2.31717e4	2268.39509	

Signal 6: DAD1 F, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.742	BV	0.0718	337.84677	70.68241	1.4408
2	7.803	BV	0.1693	2.31112e4	2259.27368	98.5592
Totals :				2.34491e4	2329.95609	

Signal 7: DAD1 G, Sig=300,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.742	BV	0.0715	176.22855	37.06408	1.9206
2	7.358	VV	0.1462	16.24239	1.45576	0.1770
3	7.800	BV	0.1471	8983.08105	960.55737	97.9024
Totals :				9175.55199	999.07721	

Signal 8: DAD1 H, Sig=320,16 Ref=off
Signal has been modified after loading from rawdata file!

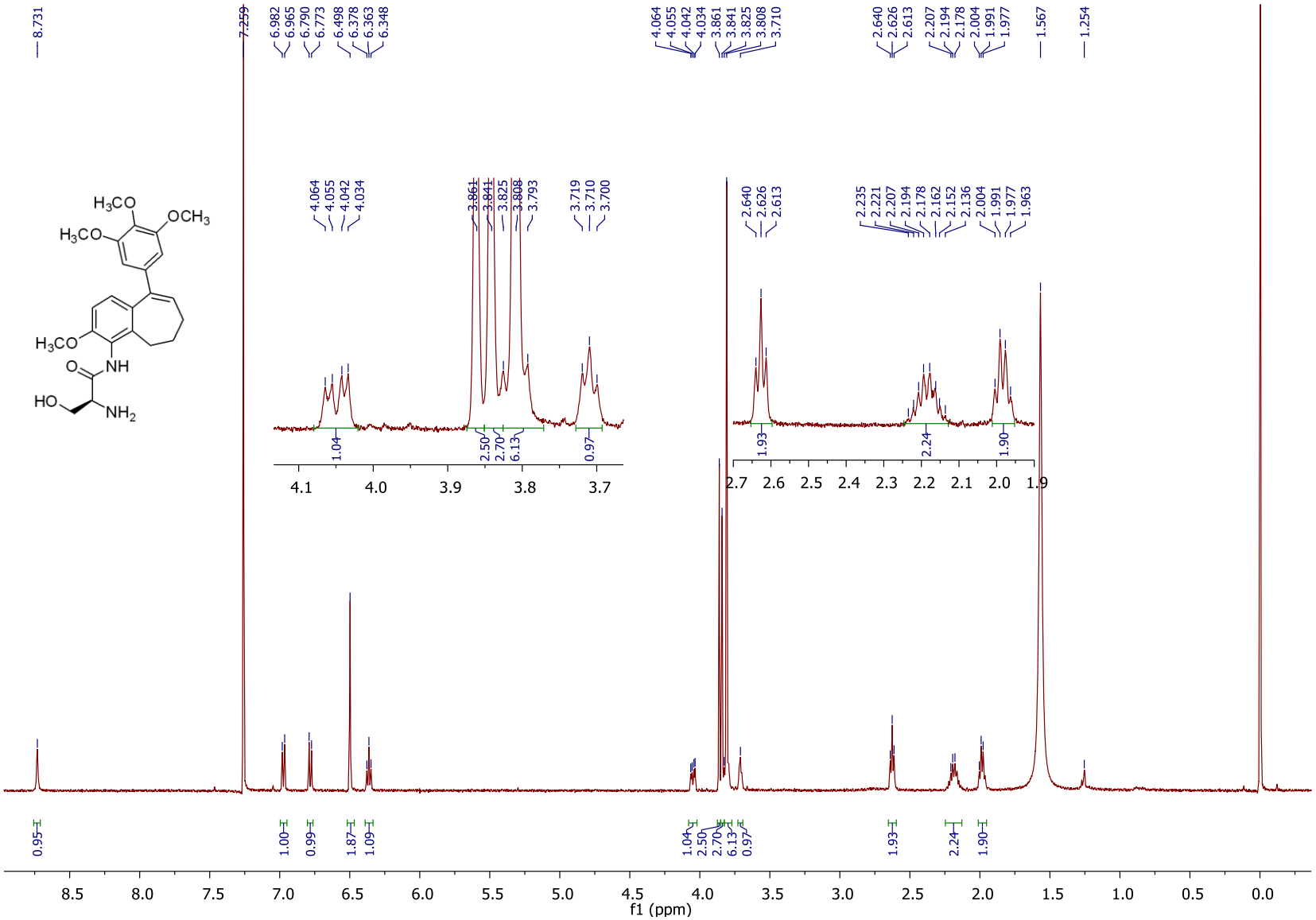
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.741	BV	0.0729	65.90073	14.00128	6.4953
2	7.358	VB	0.1497	12.81287	1.11823	1.2629
3	7.778	BV	0.1271	935.87689	105.88159	92.2418
Totals :				1014.59050	121.00111	

Data File C:\CHEM32\1\DATA\ERIC LIN\1_149_RUN300015.D
Sample Name: CML_1_149_run3

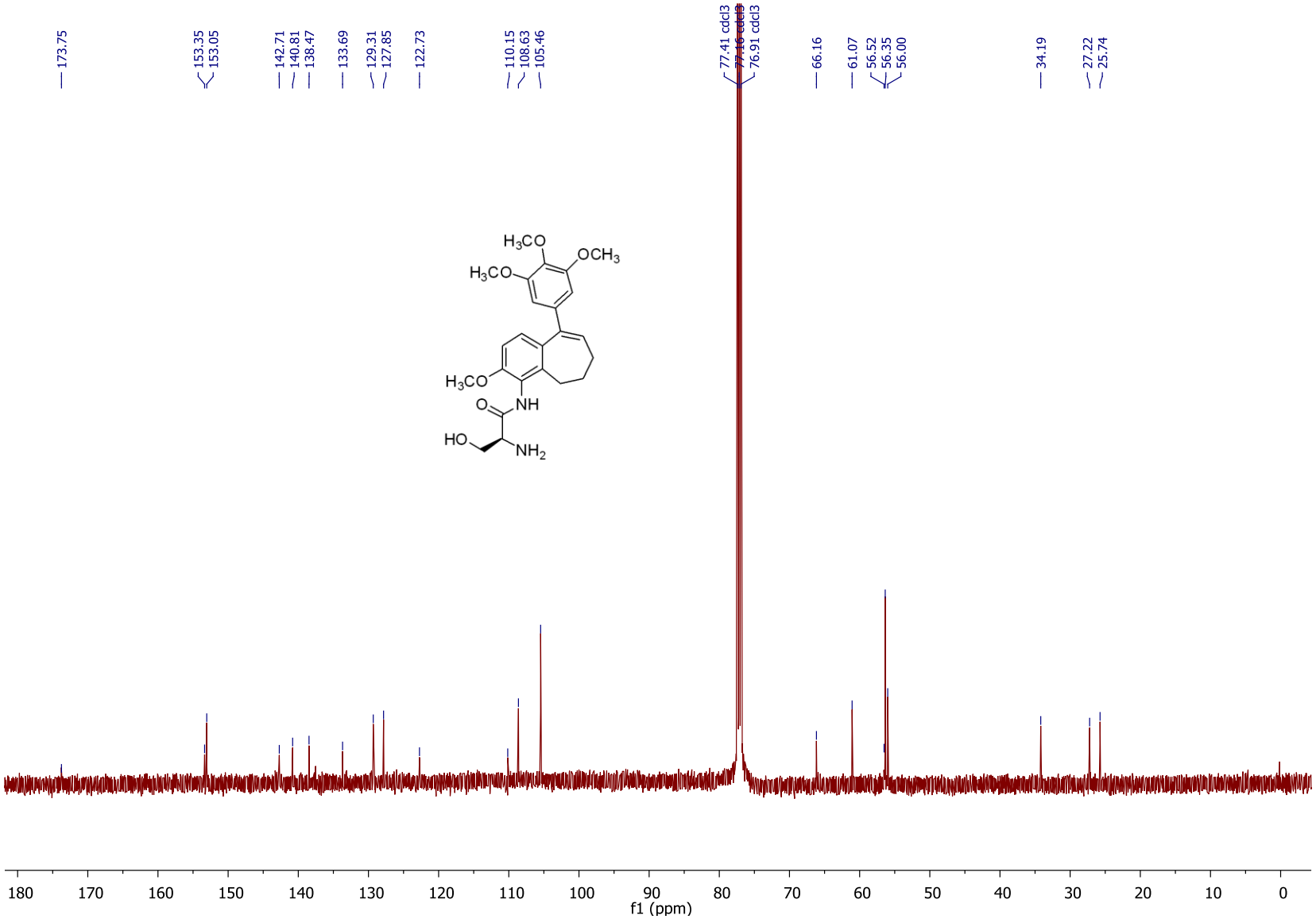
==========

*** End of Report ***

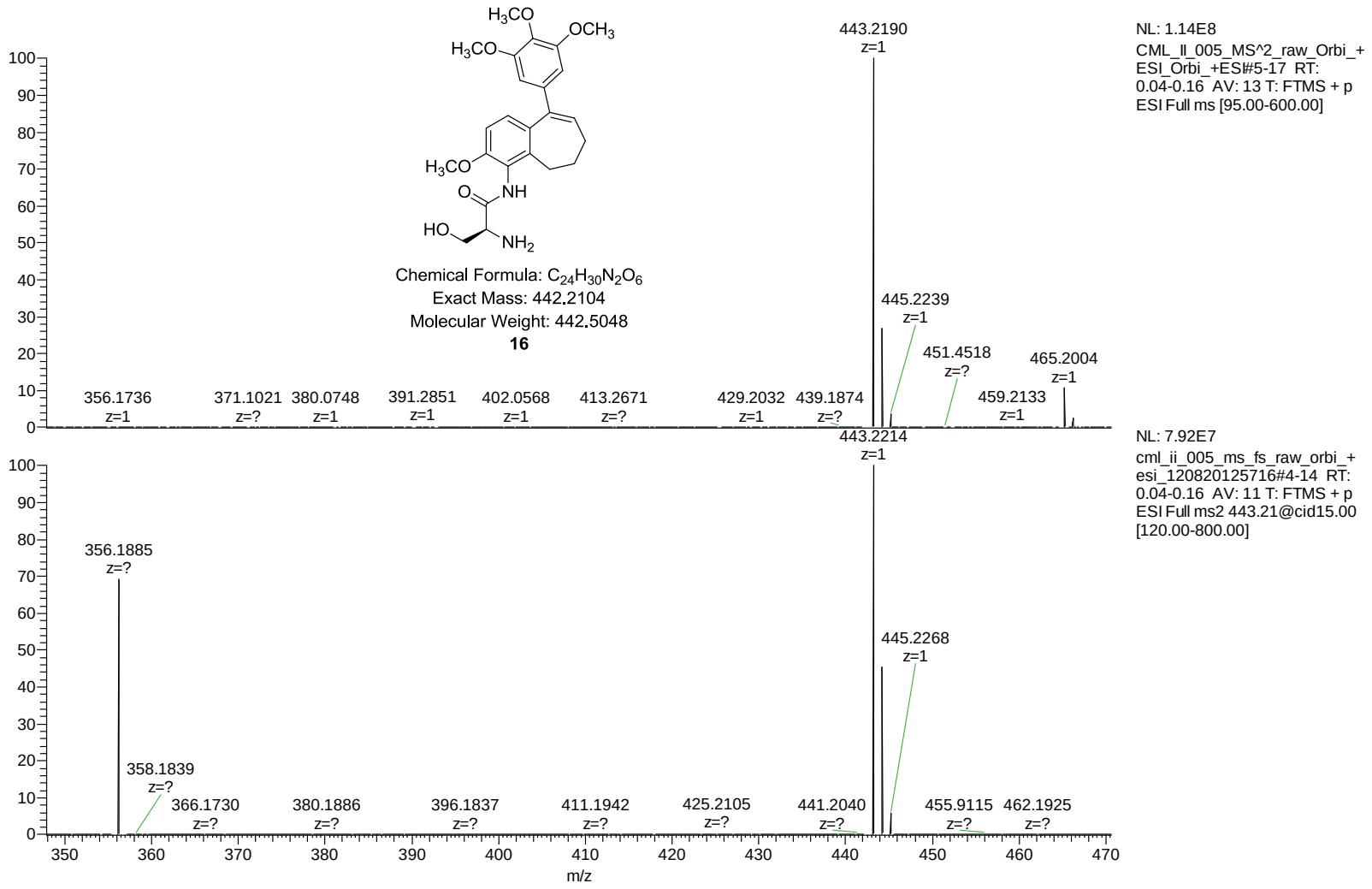
^{1}H NMR ($CDCl_3$, 500 MHz) Compound **37**



^{13}C NMR ($CDCl_3$, 125 MHz) Compound **37**



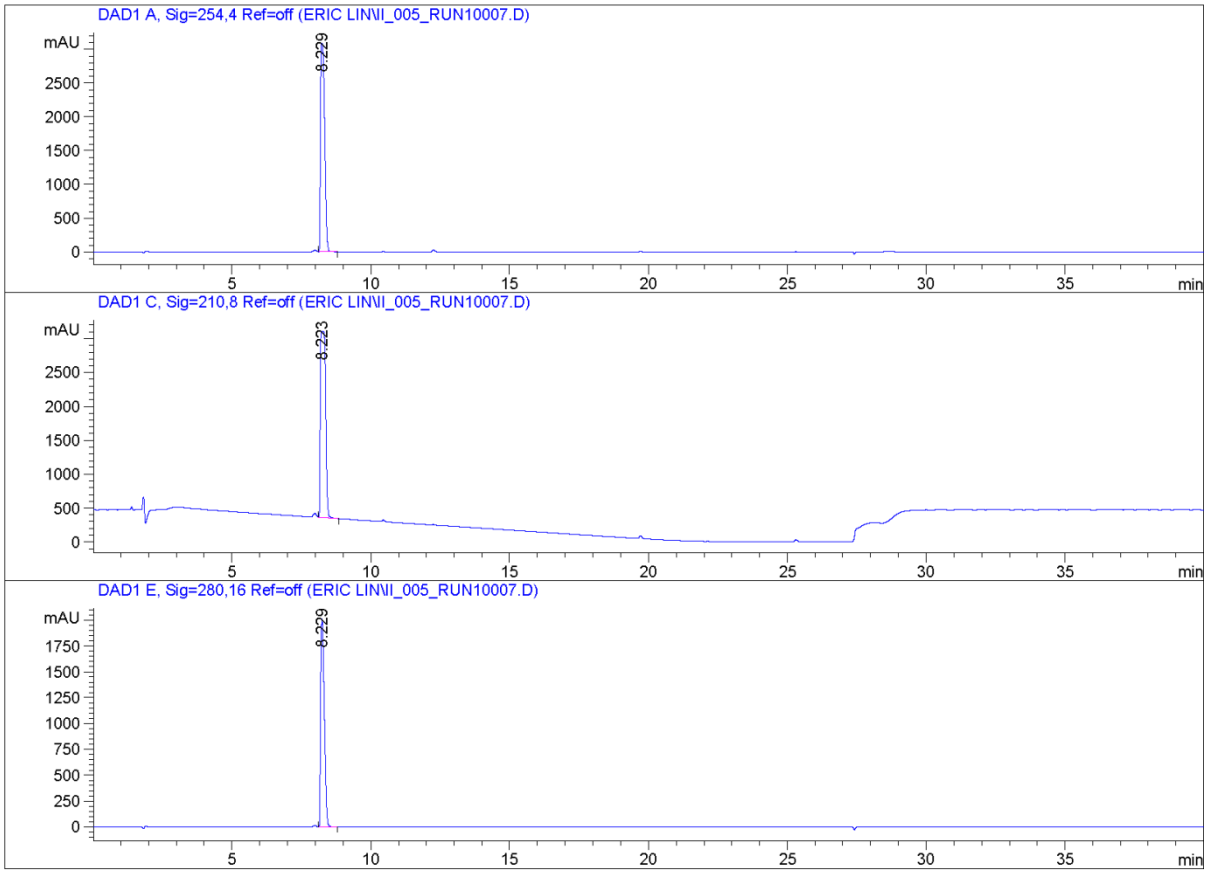
HRMS Compound **37**



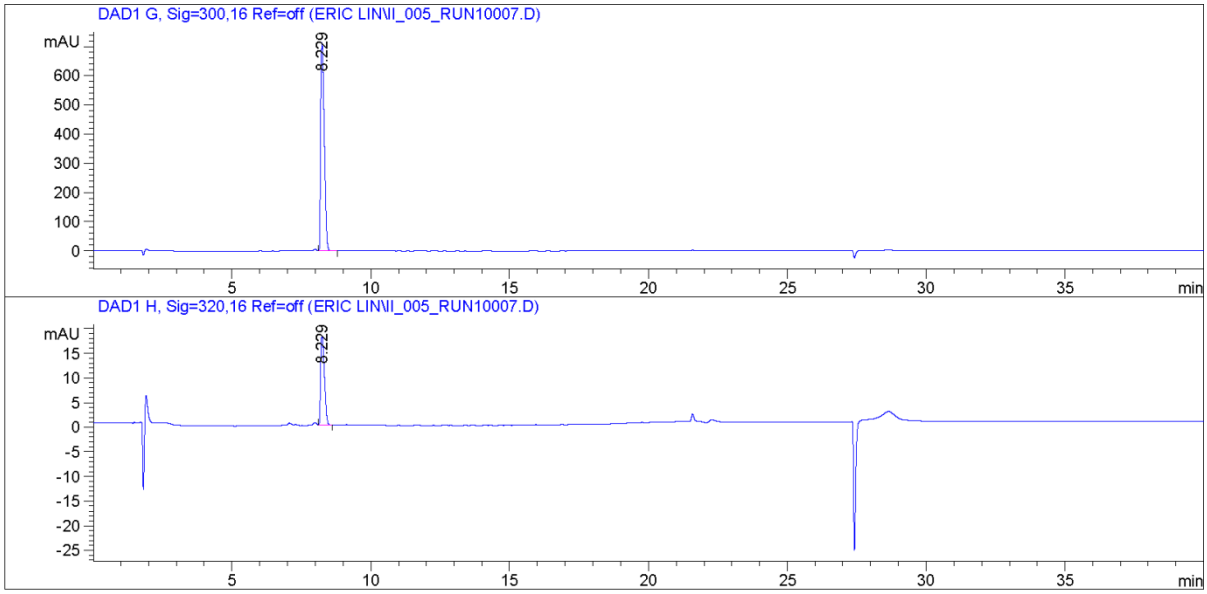
HPLC for Compound **37**

Data File C:\CHEM32\1\DATA\ERIC LIN\II_005_RUN10007.D
Sample Name: CML_II_005_run1

```
==========================================================================
Acq. Operator   : ERIC LIN
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 8/26/2012 6:28:39 PM
Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed    : 8/26/2012 6:29:45 PM by ERIC LIN
                  (modified after loading)
Analysis Method : C:\CHEM32\1\DATA\ERIC LIN\II_005_RUN10007.D\DA.M (MASTERMETHOD.M)
Last changed    : 8/26/2012 8:08:08 PM by ERIC LIN
Sample Info     :
```



Data File C:\CHEM32\1\DATA\ERIC LIN\II_005_RUN10007.D
Sample Name: CML_II_005_run1



==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.229	VV	0.1674	3.16497e4	3092.45068	100.0000
Totals :				3.16497e4	3092.45068	

Signal 2: DAD1 C, Sig=210,8 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.223	VV	0.1718	3.58401e4	2754.71655	100.0000
Totals :				3.58401e4	2754.71655	

Data File C:\CHEM32\1\DATA\ERIC LIN\II_005_RUN10007.D
Sample Name: CML_II_005_run1

Signal 3: DAD1 E, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.229	VV	0.1462	1.83095e4	2011.87817	100.0000
Totals :				1.83095e4	2011.87817	

Signal 4: DAD1 G, Sig=300,16 Ref=off

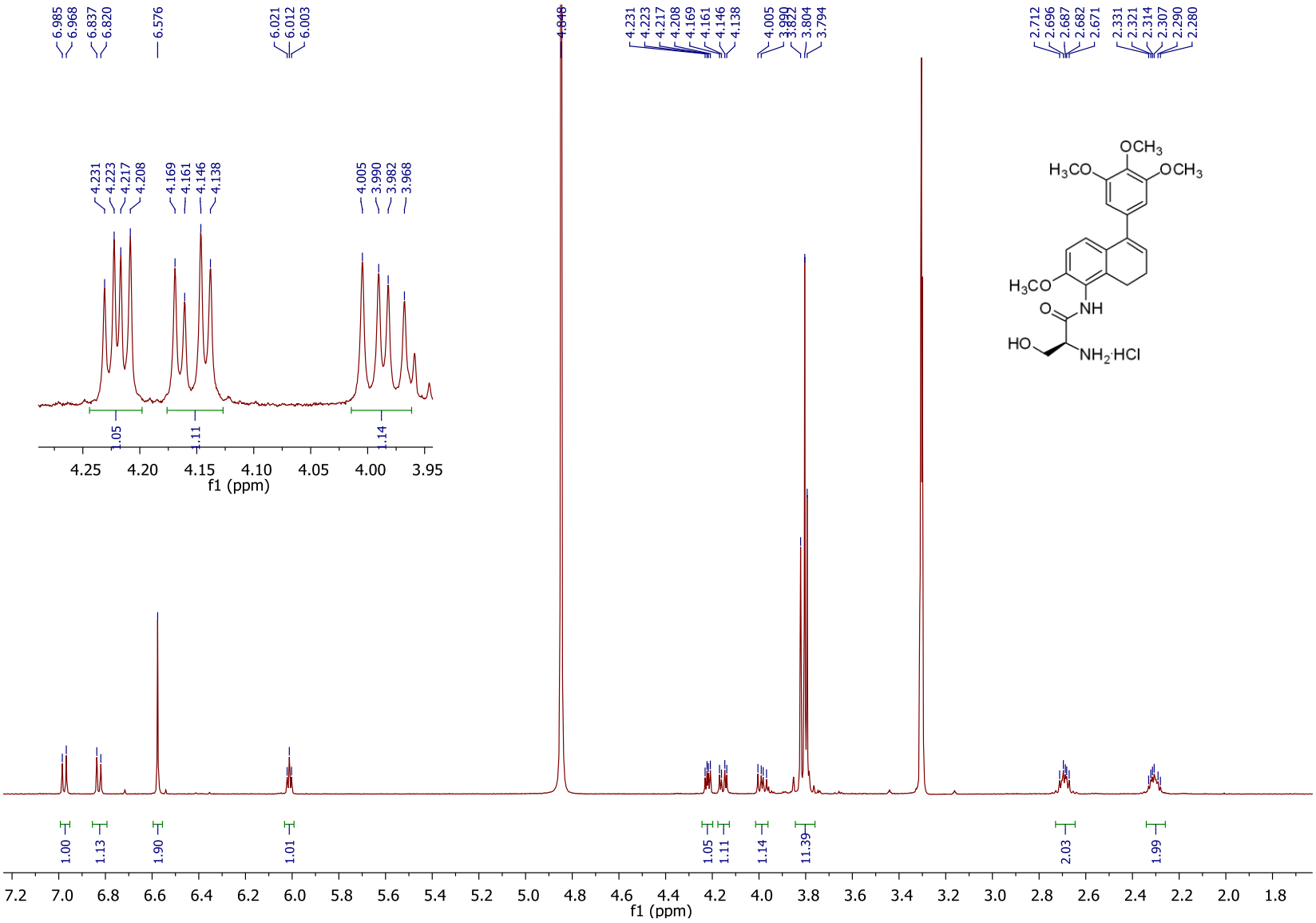
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.229	VB	0.1406	6393.18701	712.86285	100.0000
Totals :				6393.18701	712.86285	

Signal 5: DAD1 H, Sig=320,16 Ref=off

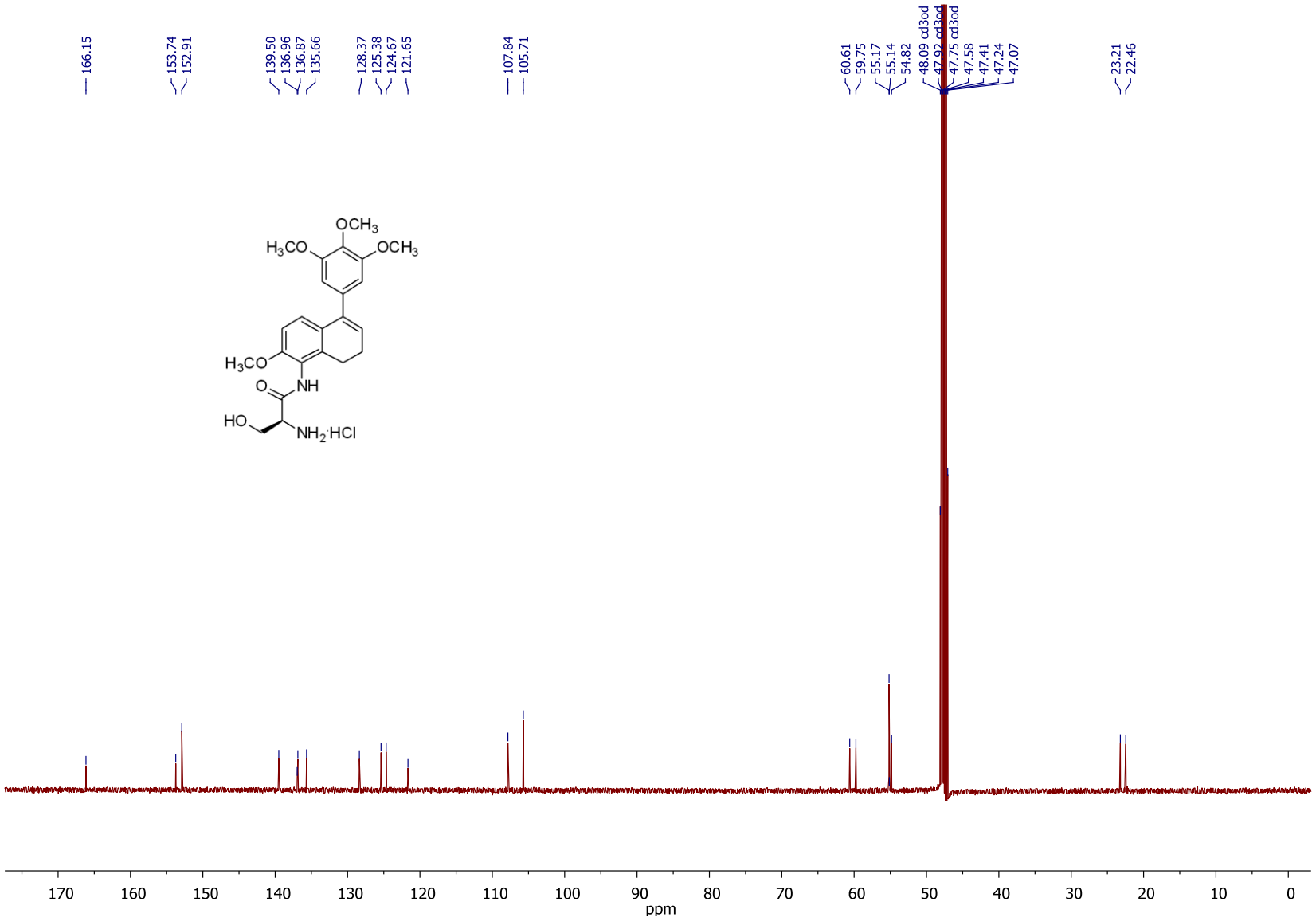
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.229	VB	0.1486	169.87639	18.24767	100.0000
Totals :				169.87639	18.24767	

*** End of Report ***

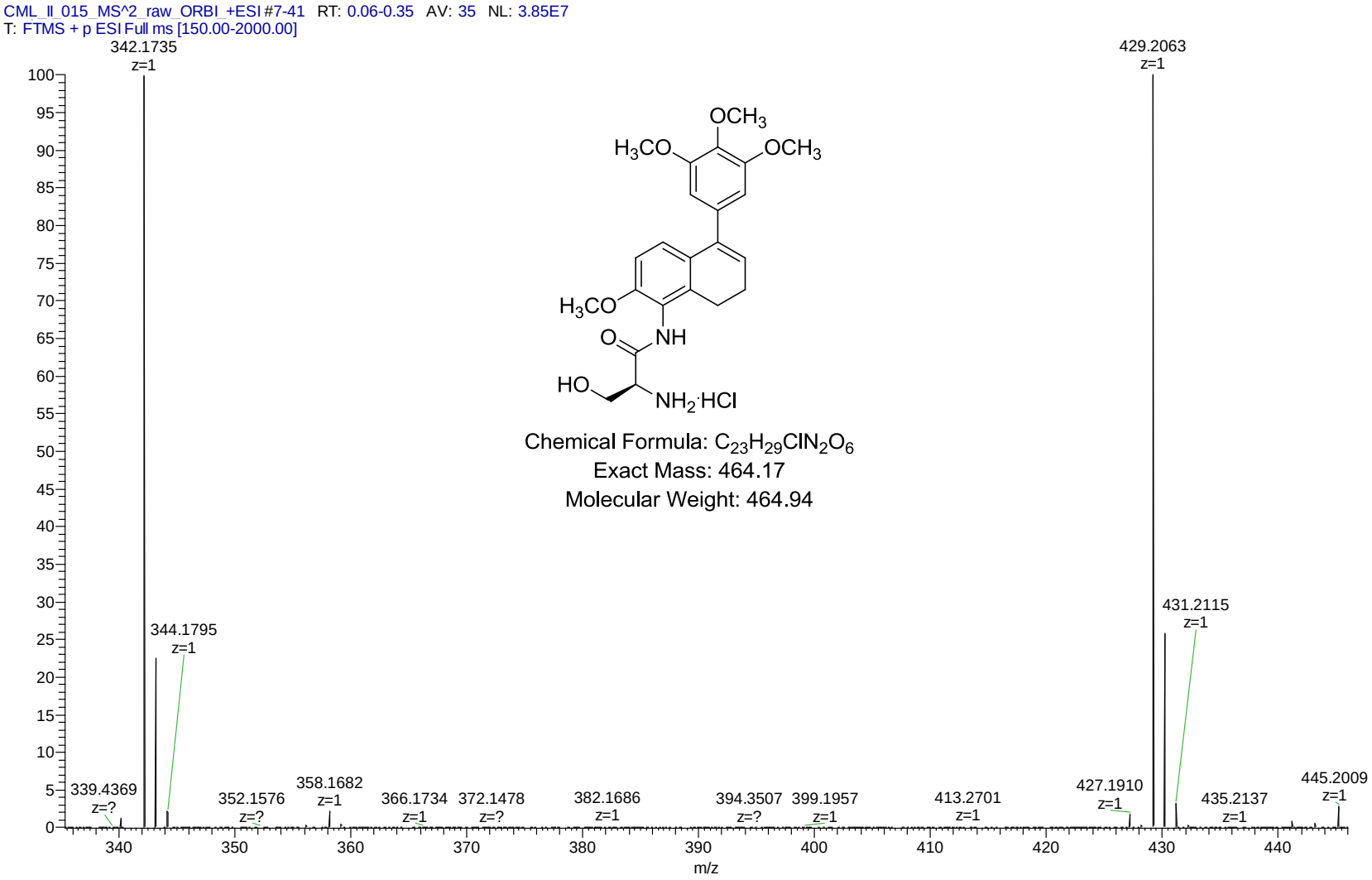
^{1}H NMR (CD_3OD, 500 MHz) Compound **38**



^{13}C NMR (CD_3OD, 125 MHz) Compound **38**



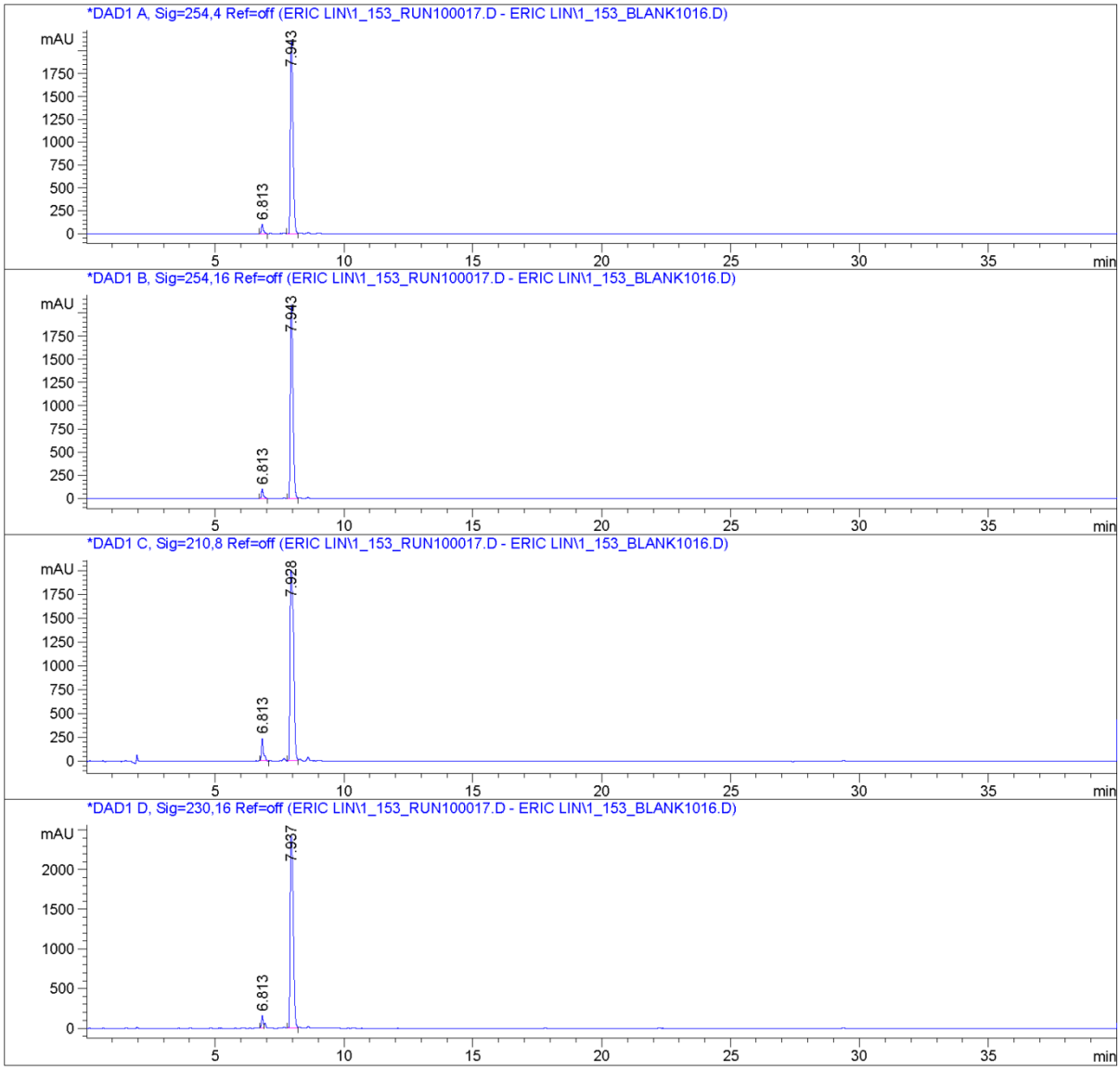
HRMS Compound **38**



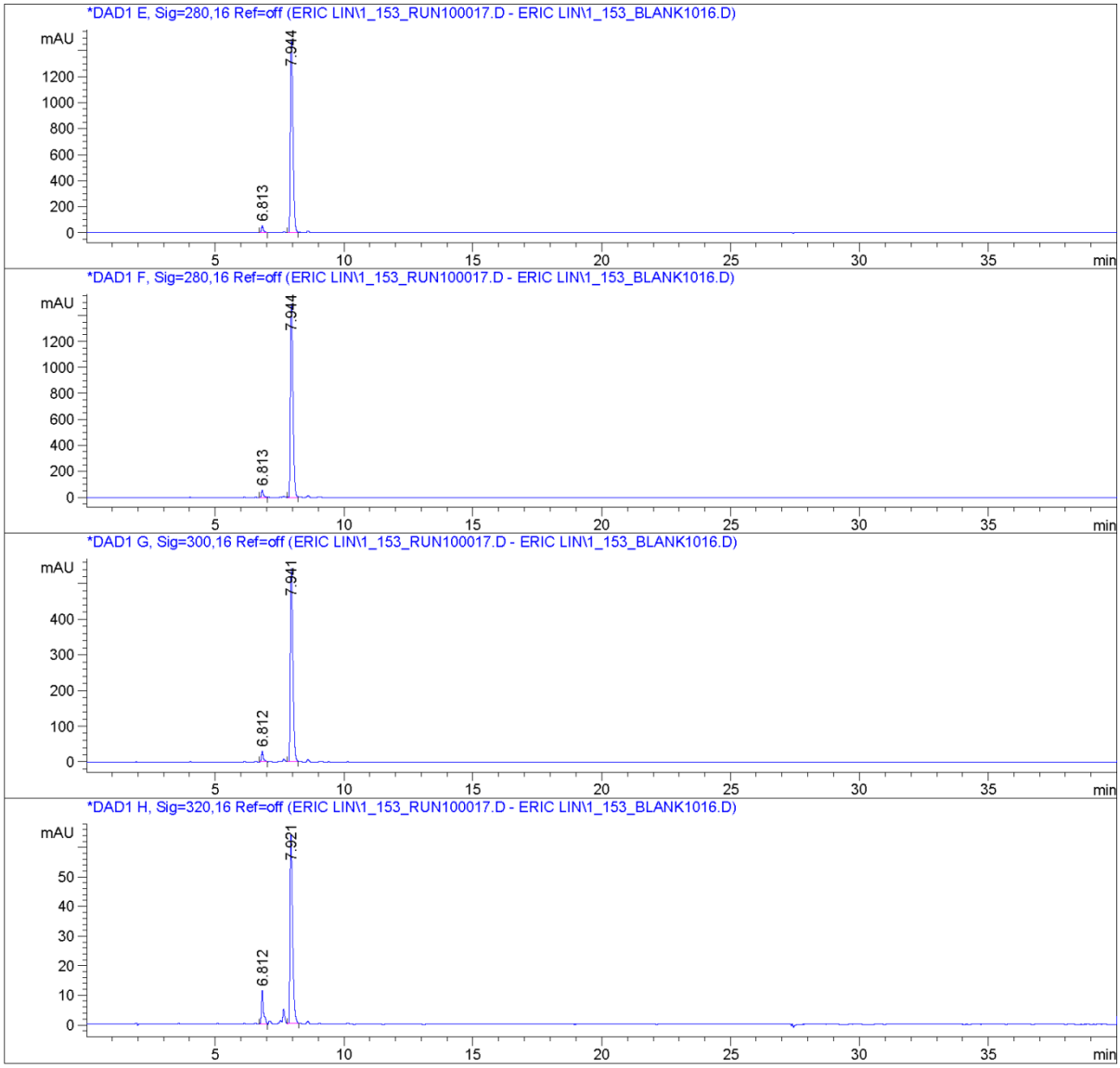
HPLC for Compound **38**

Data File C:\CHEM32\1\DATA\ERIC LIN\1_153_RUN100017.D
Sample Name: CML_1_153_run1

```
==========================================================================
Acq. Operator   : ERIC LIN
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 8/6/2012 1:58:05 PM
Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed    : 8/6/2012 1:47:21 PM by ERIC LIN
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed    : 8/6/2012 1:00:08 PM by ERIC LIN
                  (modified after loading)
```



Data File C:\CHEM32\1\DATA\ERIC LIN\1_153_RUN100017.D
Sample Name: CML_1_153_run1



==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Data File C:\CHEM32\1\DATA\ERIC LIN\1_153_RUN100017.D
Sample Name: CML_1_153_run1

Signal 1: DAD1 A, Sig=254,4 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.813	BV	0.0760	537.58142	104.52646	3.3615
2	7.943	BV	0.1131	1.54548e4	2117.94653	96.6385
Totals :				1.59924e4	2222.47299	

Signal 2: DAD1 B, Sig=254,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.813	BV	0.0777	540.95929	102.36874	3.4391
2	7.943	BV	0.1128	1.51888e4	2089.05884	96.5609
Totals :				1.57297e4	2191.42757	

Signal 3: DAD1 C, Sig=210,8 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.813	VV	0.0816	1336.63696	237.73367	6.3785
2	7.928	BV	0.1420	1.96189e4	2007.60486	93.6215
Totals :				2.09555e4	2245.33853	

Signal 4: DAD1 D, Sig=230,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.813	VV	0.0693	728.46539	159.36909	3.3904
2	7.937	BV	0.1392	2.07575e4	2440.93188	96.6096
Totals :				2.14860e4	2600.30098	

Data File C:\CHEM32\1\DATA\ERIC LIN\1_153_RUN100017.D
Sample Name: CML_1_153_run1

Signal 5: DAD1 E, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.813	BV	0.0770	292.07108	55.82075	2.6804
2	7.944	VV	0.1111	1.06043e4	1488.94983	97.3196
Totals :				1.08964e4	1544.77058	

Signal 6: DAD1 F, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.813	BV	0.0770	292.07108	55.82075	2.6804
2	7.944	VV	0.1111	1.06043e4	1488.94983	97.3196
Totals :				1.08964e4	1544.77058	

Signal 7: DAD1 G, Sig=300,16 Ref=off
Signal has been modified after loading from rawdata file!

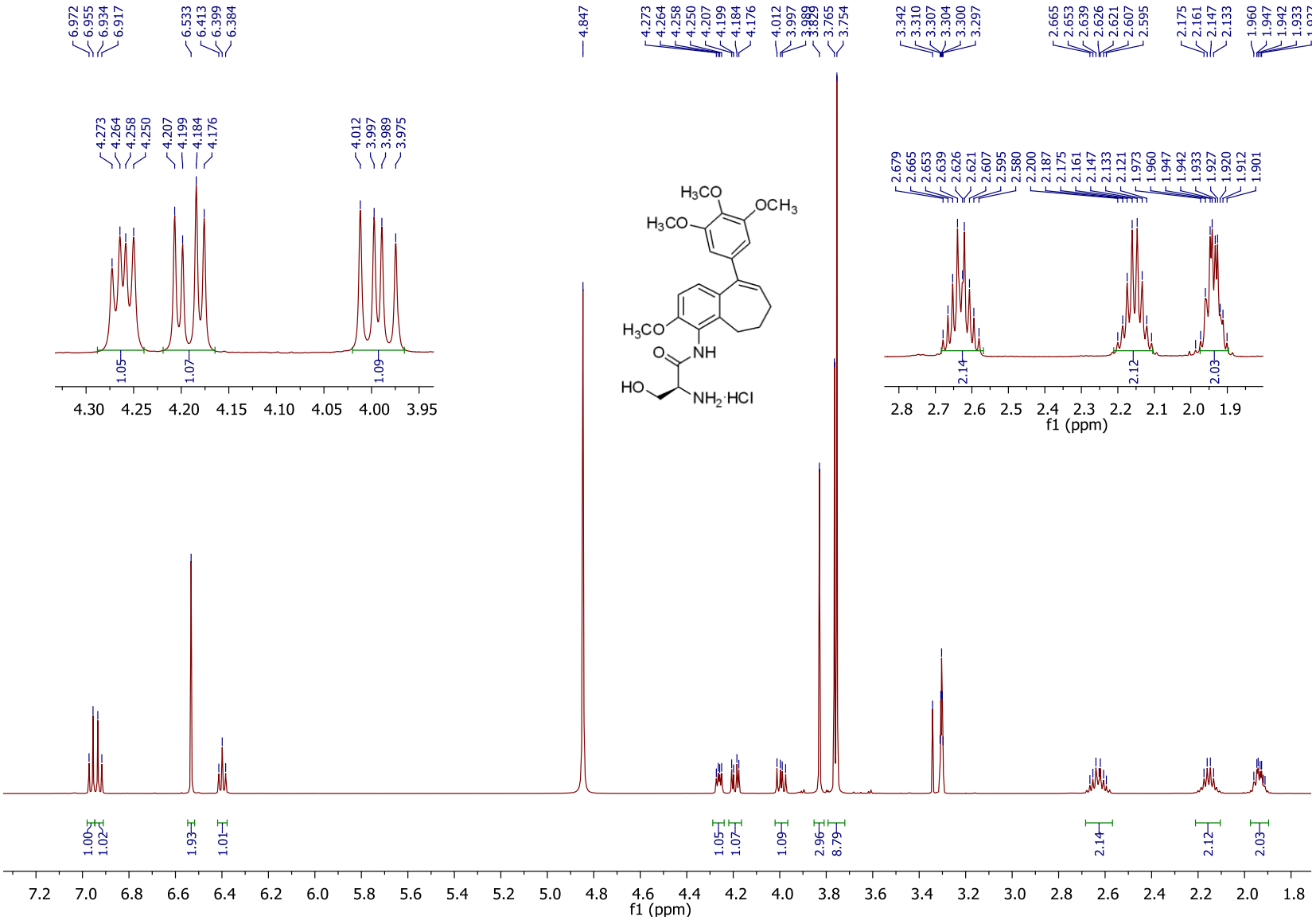
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.812	VV	0.0791	160.51169	29.68318	3.9846
2	7.941	BV	0.1093	3867.79565	542.01520	96.0154
Totals :				4028.30734	571.69837	

Signal 8: DAD1 H, Sig=320,16 Ref=off
Signal has been modified after loading from rawdata file!

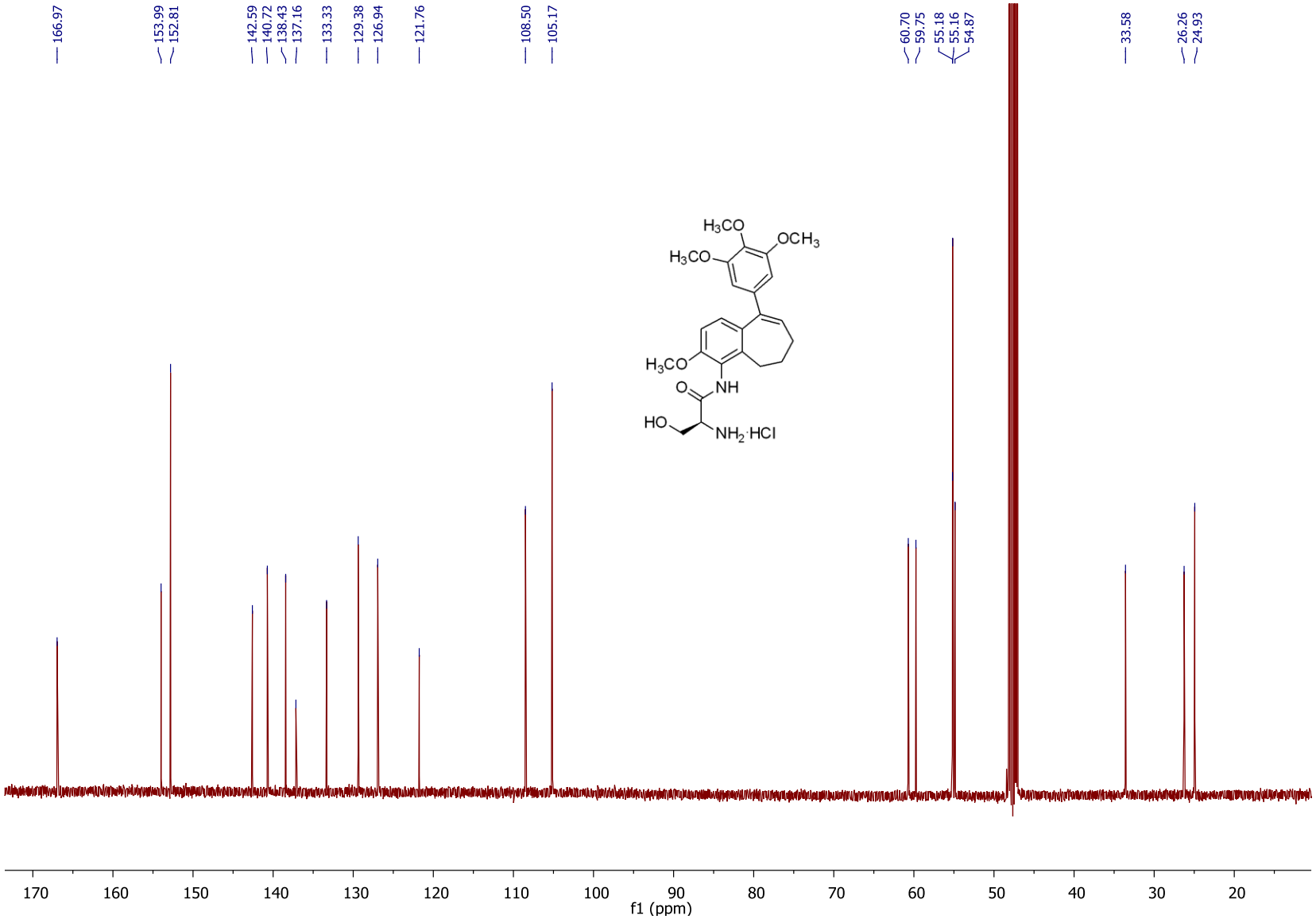
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.812	BB	0.0828	65.37932	11.41679	12.7347
2	7.921	VB	0.1073	448.01462	64.29078	87.2653
Totals :				513.39394	75.70757	

==========

^{1}H NMR (CD_3OD, 500 MHz) Compound **39**

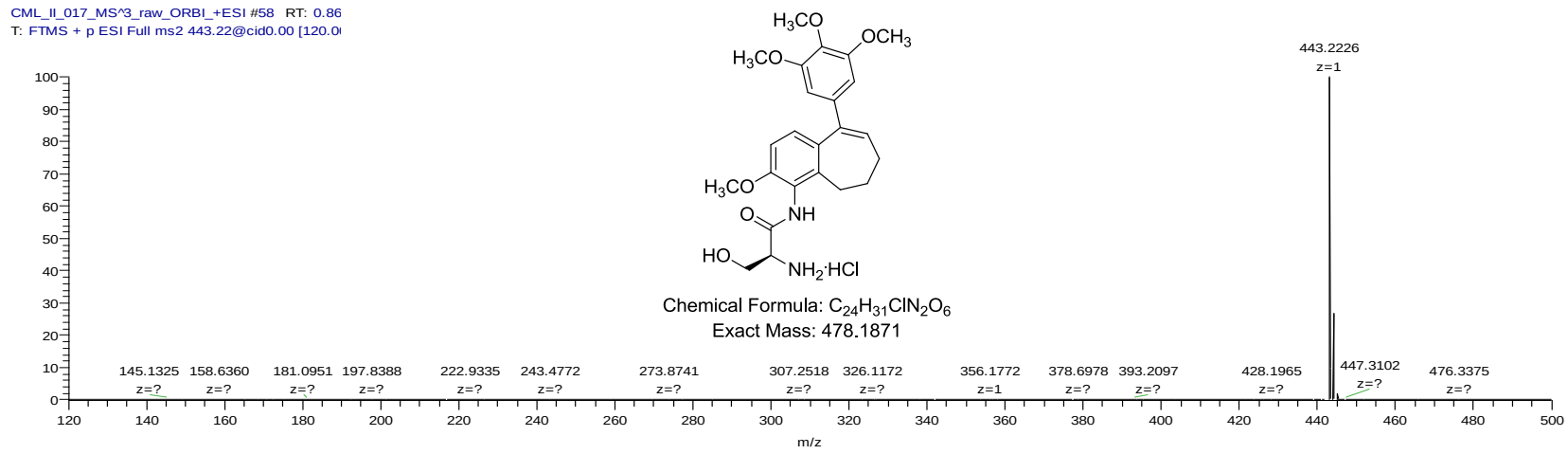


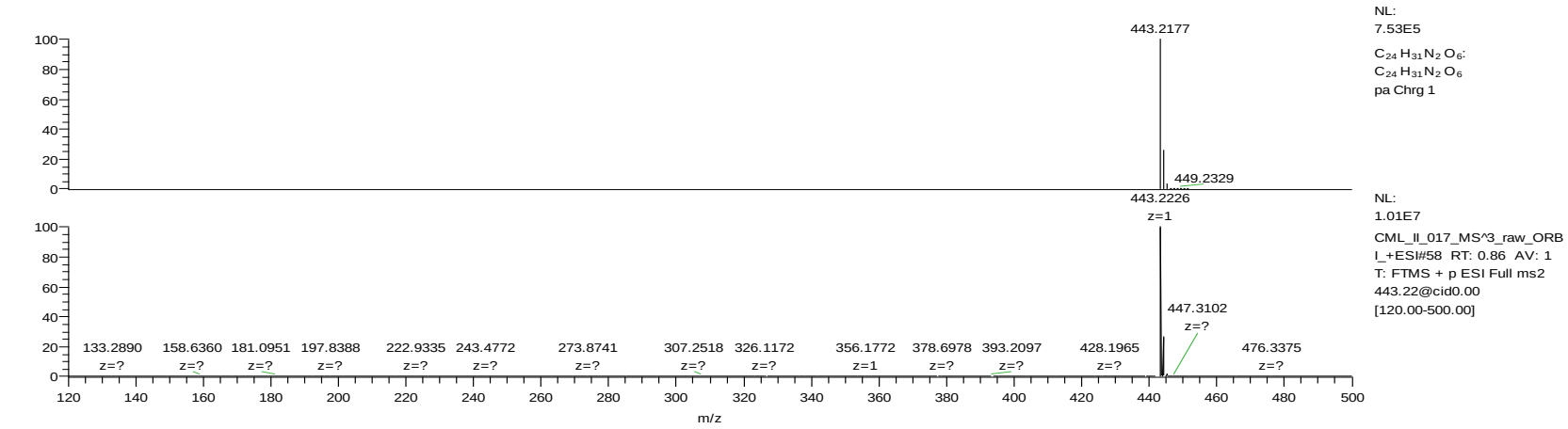
^{13}C NMR (CD_3OD, 125 MHz) Compound **39**



HRMS Compound **39**

CML_II_017_MS^3_raw_ORBI_+ESI 8/26/2012 11:33:23 PM

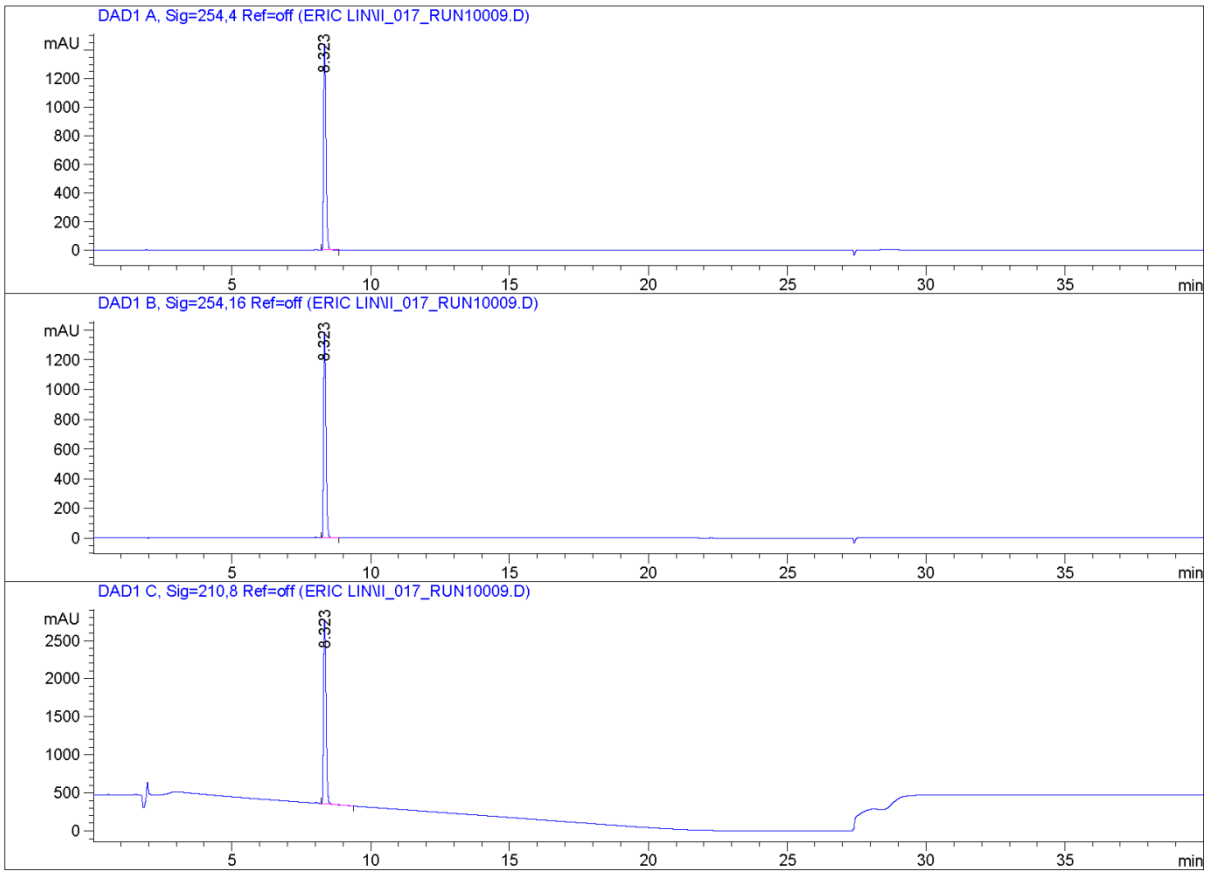




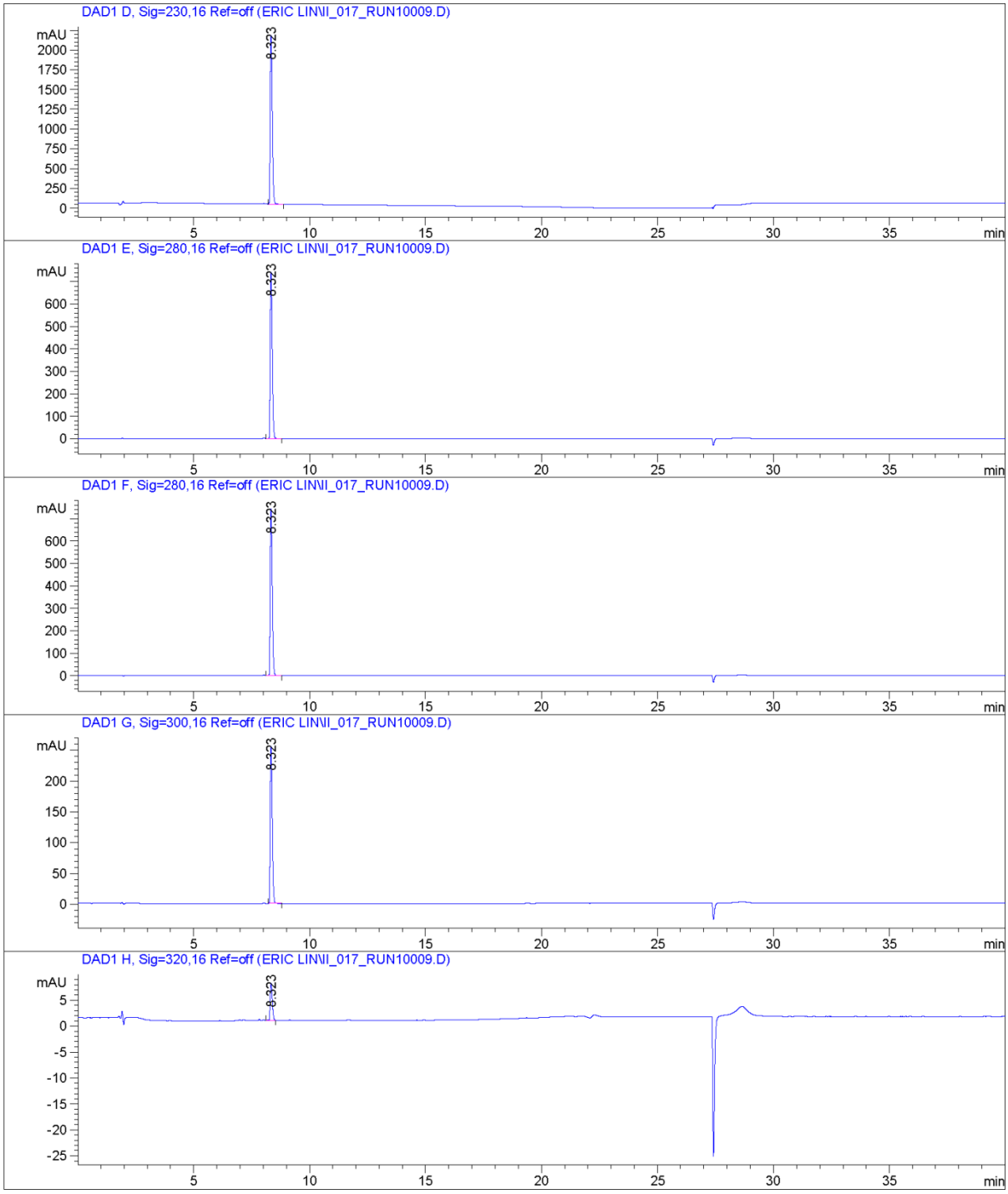
HPLC for Compound **39**

Data File C:\CHEM32\1\DATA\ERIC LIN\II_017_RUN10009.D
Sample Name: CML_II_017_run1

```
    ======================================================================
    Acq. Operator   : ERIC LIN
    Acq. Instrument : Instrument 1                    Location :   -
    Injection Date  : 8/26/2012 8:14:29 PM
    Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD.M
    Last changed    : 8/26/2012 8:11:49 PM by ERIC LIN
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\DATA\ERIC LIN\II_017_RUN10009.D\DA.M (MASTERMETHOD.M)
    Last changed    : 8/26/2012 9:04:41 PM by ERIC LIN
    Sample Info     :
```



Data File C:\CHEM32\1\DATA\ERIC LIN\II_017_RUN10009.D
Sample Name: CML_II_017_run1



Data File C:\CHEM32\1\DATA\ERIC LIN\II_017_RUN10009.D
Sample Name: CML_II_017_run1

==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.323	VV	0.0949	8745.86328	1439.63110	100.0000
Totals :				8745.86328	1439.63110	

Signal 2: DAD1 B, Sig=254,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.323	VB	0.0948	8403.83496	1384.48010	100.0000
Totals :				8403.83496	1384.48010	

Signal 3: DAD1 C, Sig=210,8 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.323	VB	0.1035	1.56445e4	2416.47046	100.0000
Totals :				1.56445e4	2416.47046	

Signal 4: DAD1 D, Sig=230,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.323	VV	0.0977	1.31259e4	2134.97778	100.0000
Totals :				1.31259e4	2134.97778	

Data File C:\CHEM32\1\DATA\ERIC LIN\II_017_RUN10009.D
Sample Name: CML_II_017_run1

Signal 5: DAD1 E, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.323	VB	0.0948	4502.56006	741.66730	100.0000
Totals :				4502.56006	741.66730	

Signal 6: DAD1 F, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.323	VB	0.0948	4502.56006	741.66730	100.0000
Totals :				4502.56006	741.66730	

Signal 7: DAD1 G, Sig=300,16 Ref=off

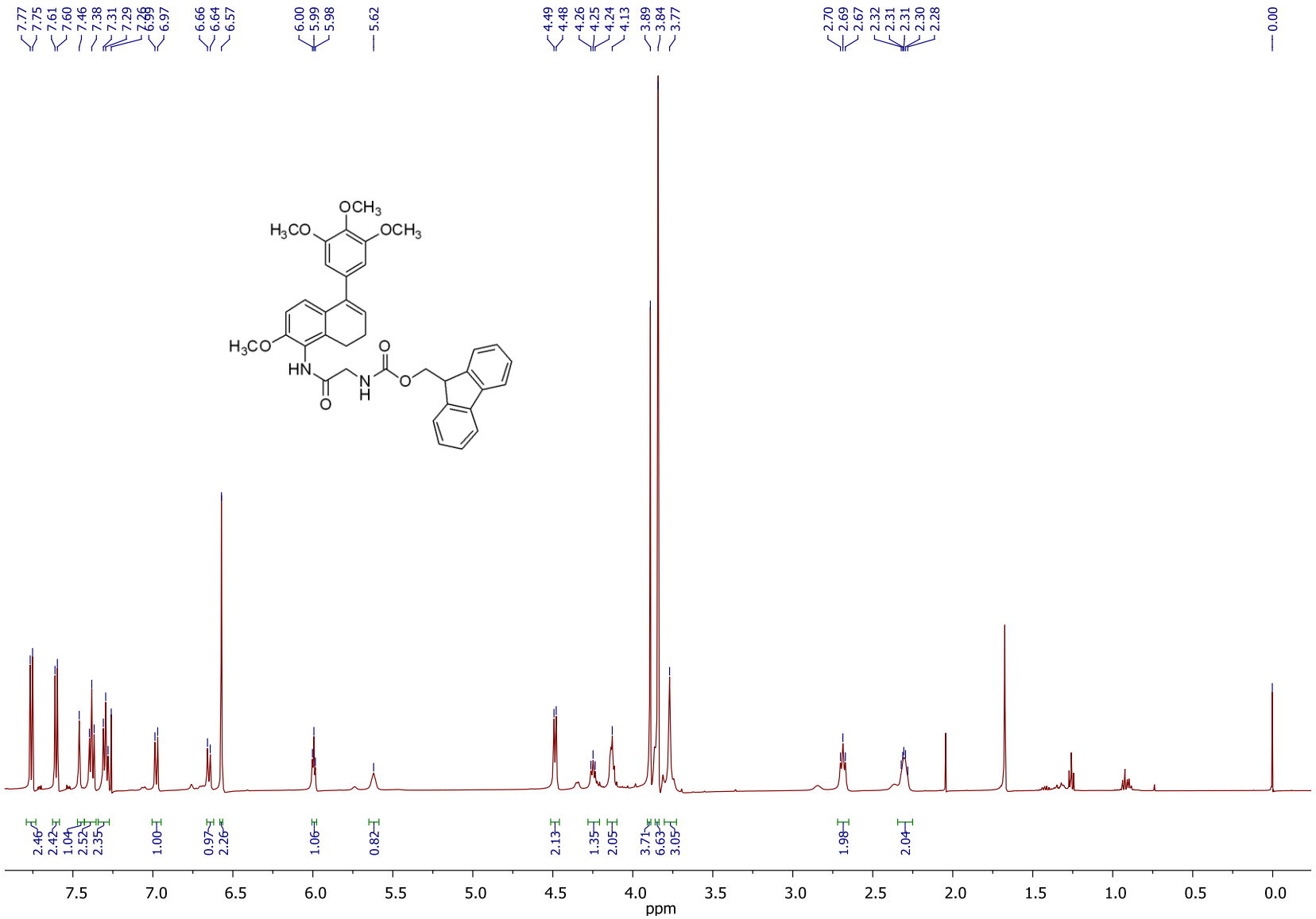
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.323	BB	0.0949	1553.26367	255.71465	100.0000
Totals :				1553.26367	255.71465	

Signal 8: DAD1 H, Sig=320,16 Ref=off

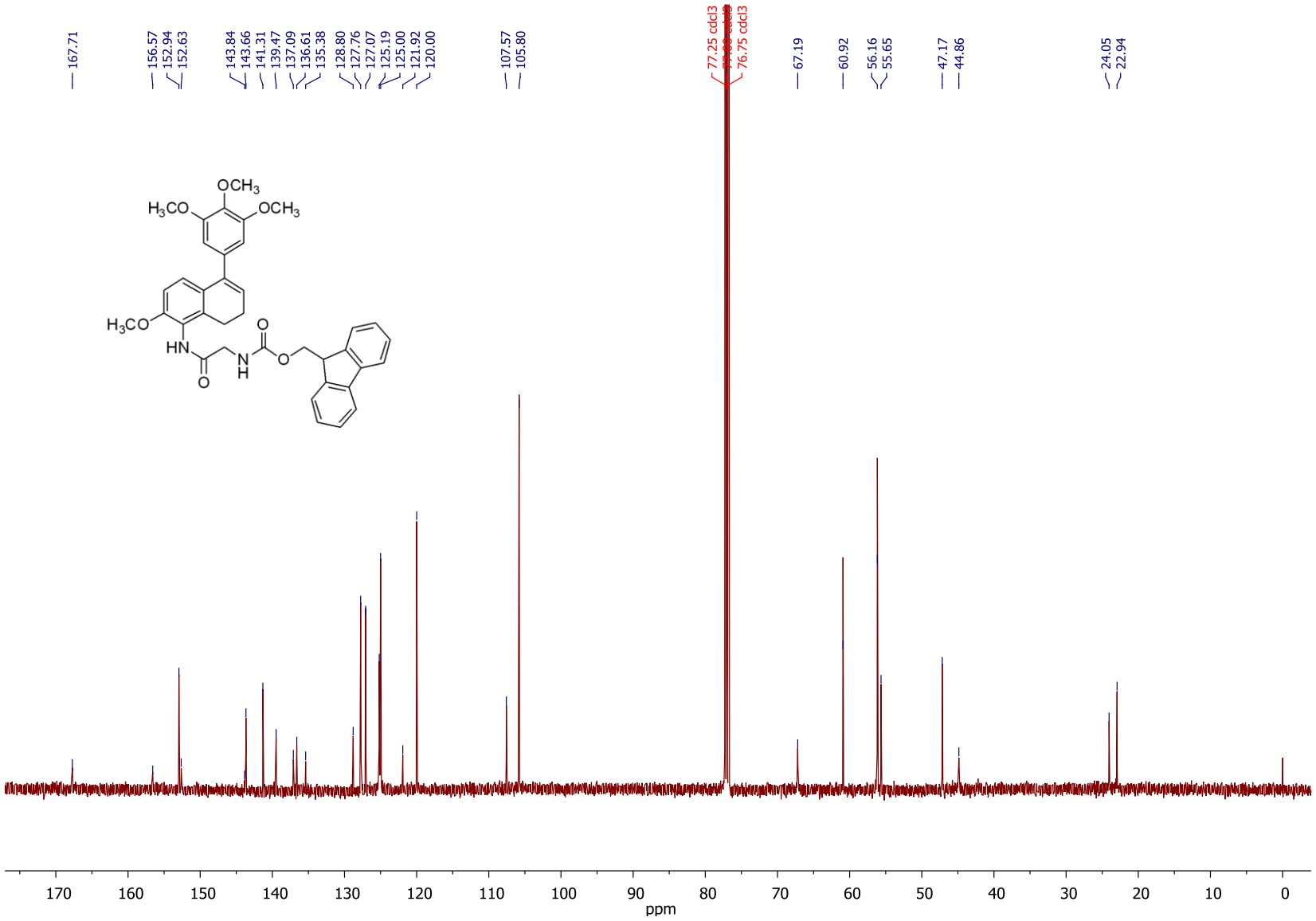
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.323	BB	0.0998	45.32738	7.16882	100.0000
Totals :				45.32738	7.16882	

*** End of Report ***

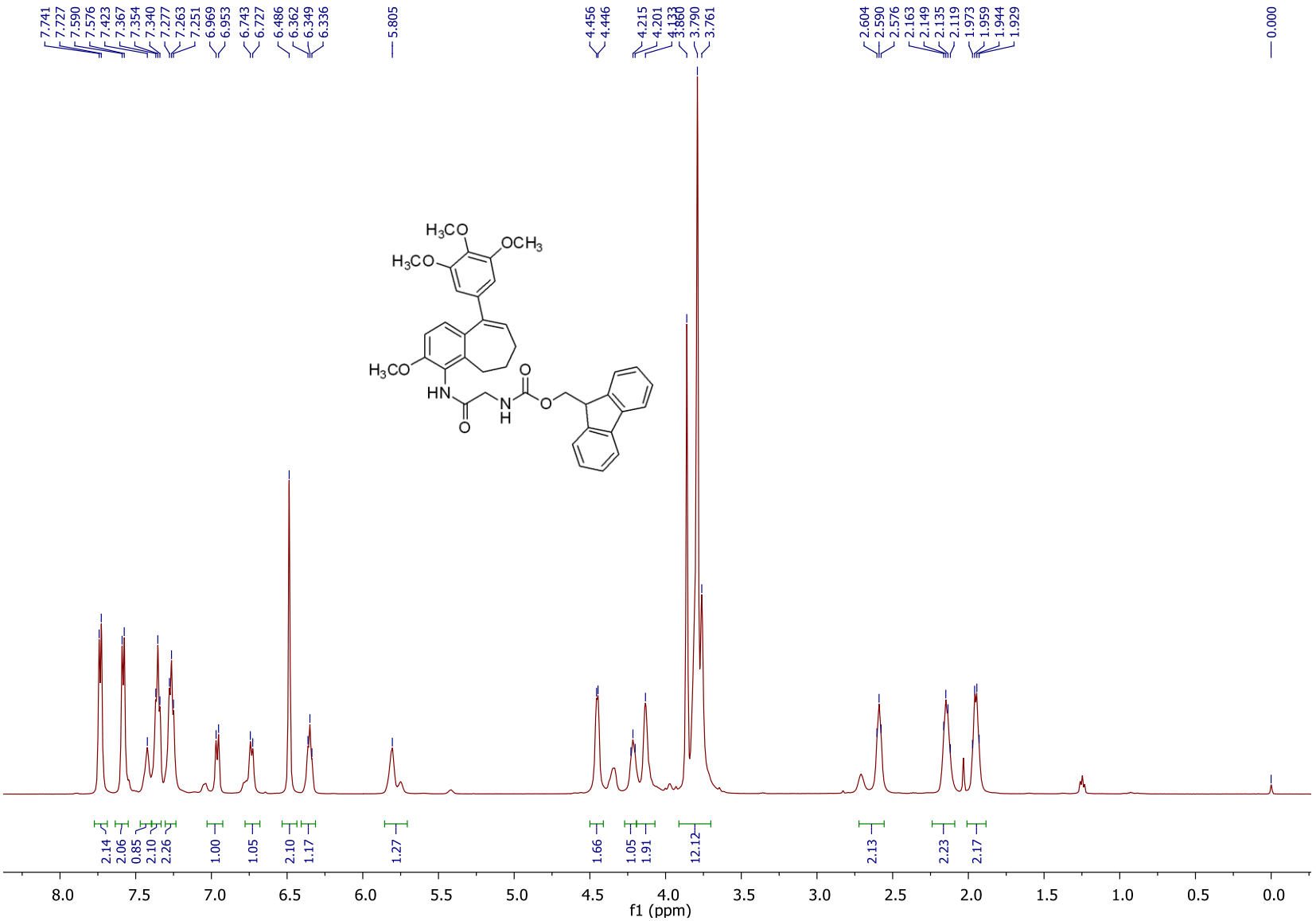
^{1}H NMR ($CDCl_3$, 500 MHz) Compound **40**



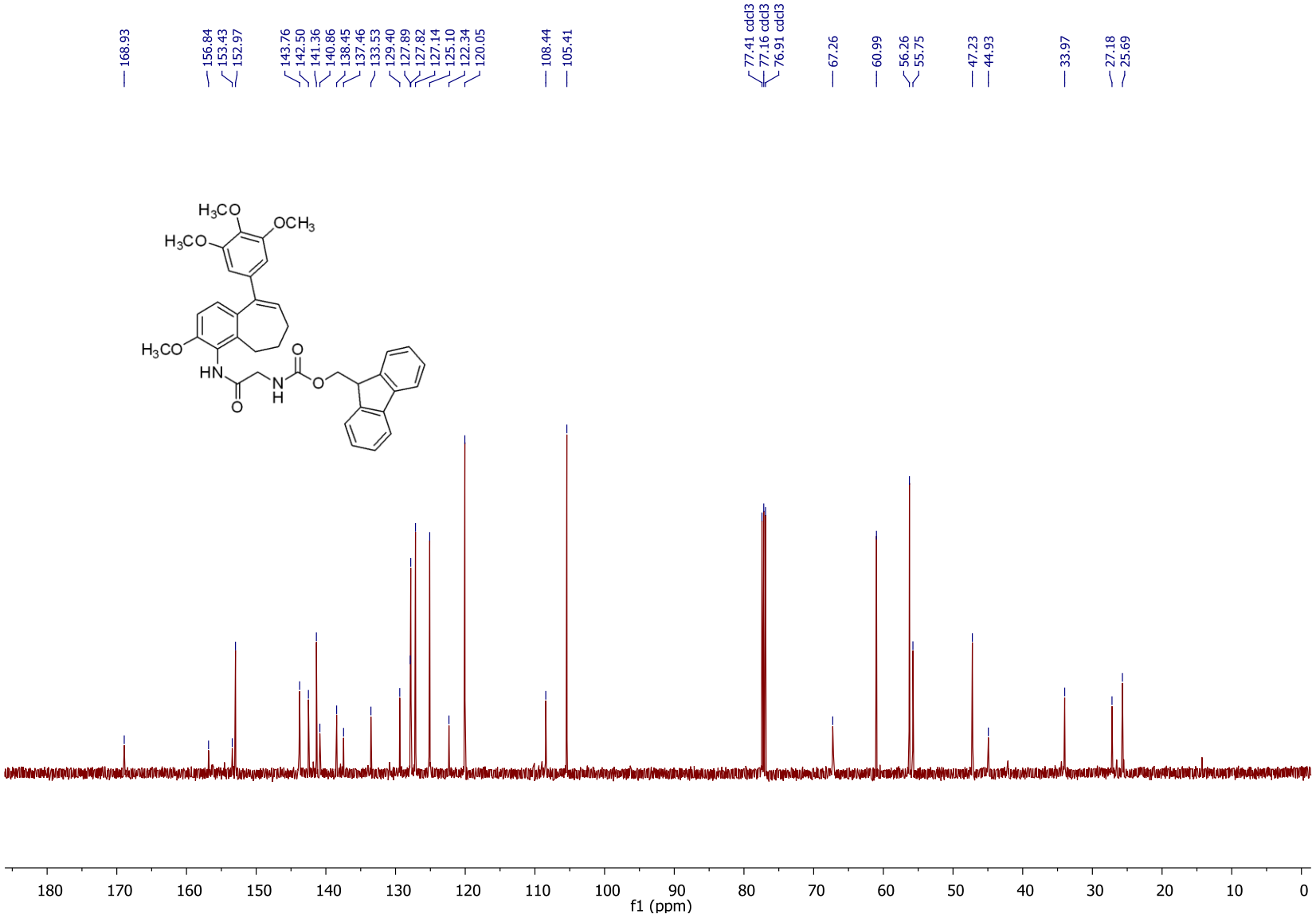
^{13}C NMR ($CDCl_3$, 125 MHz) Compound **40**



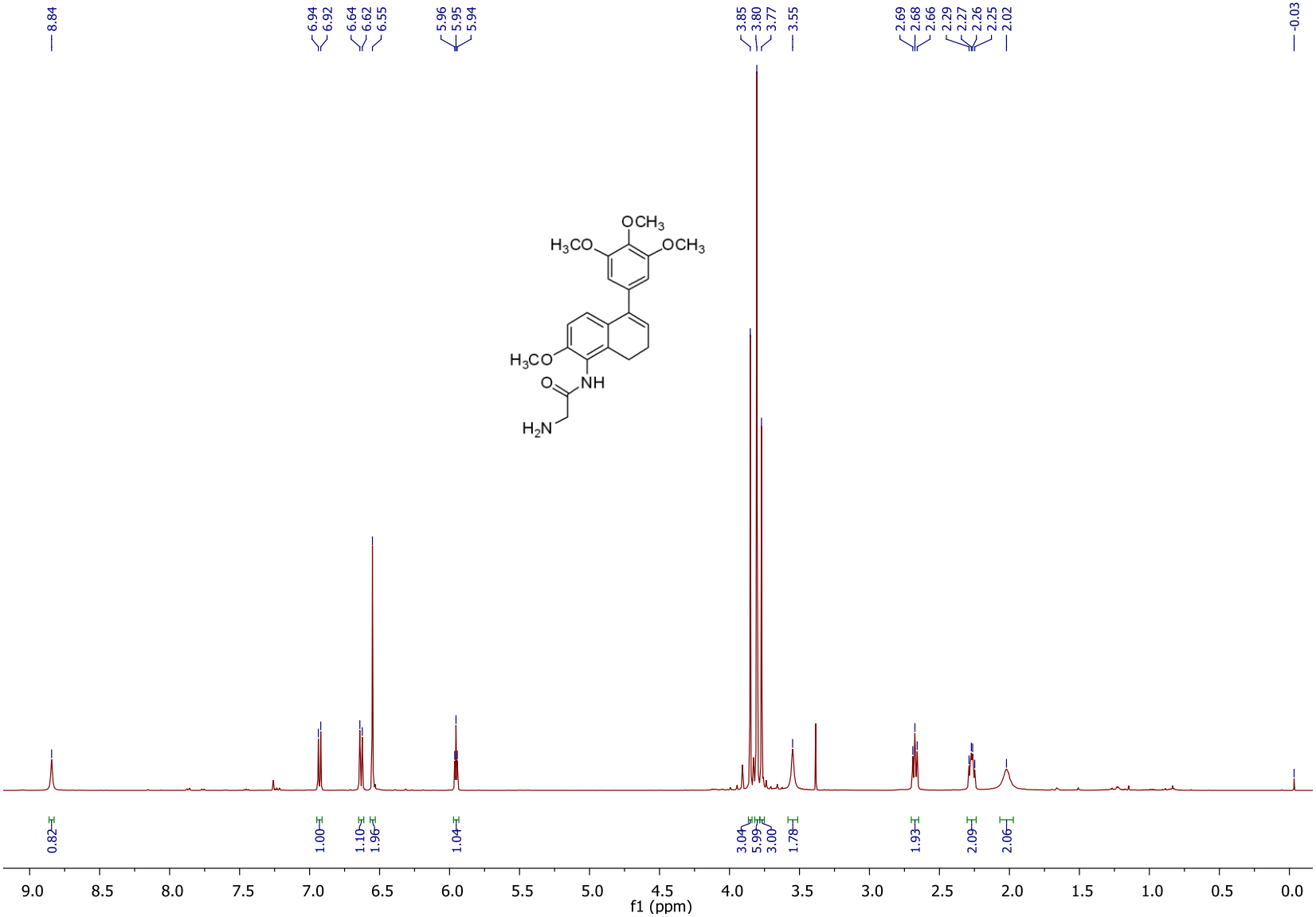
^{1}H NMR ($CDCl_3$, 500 MHz) Compound **41**



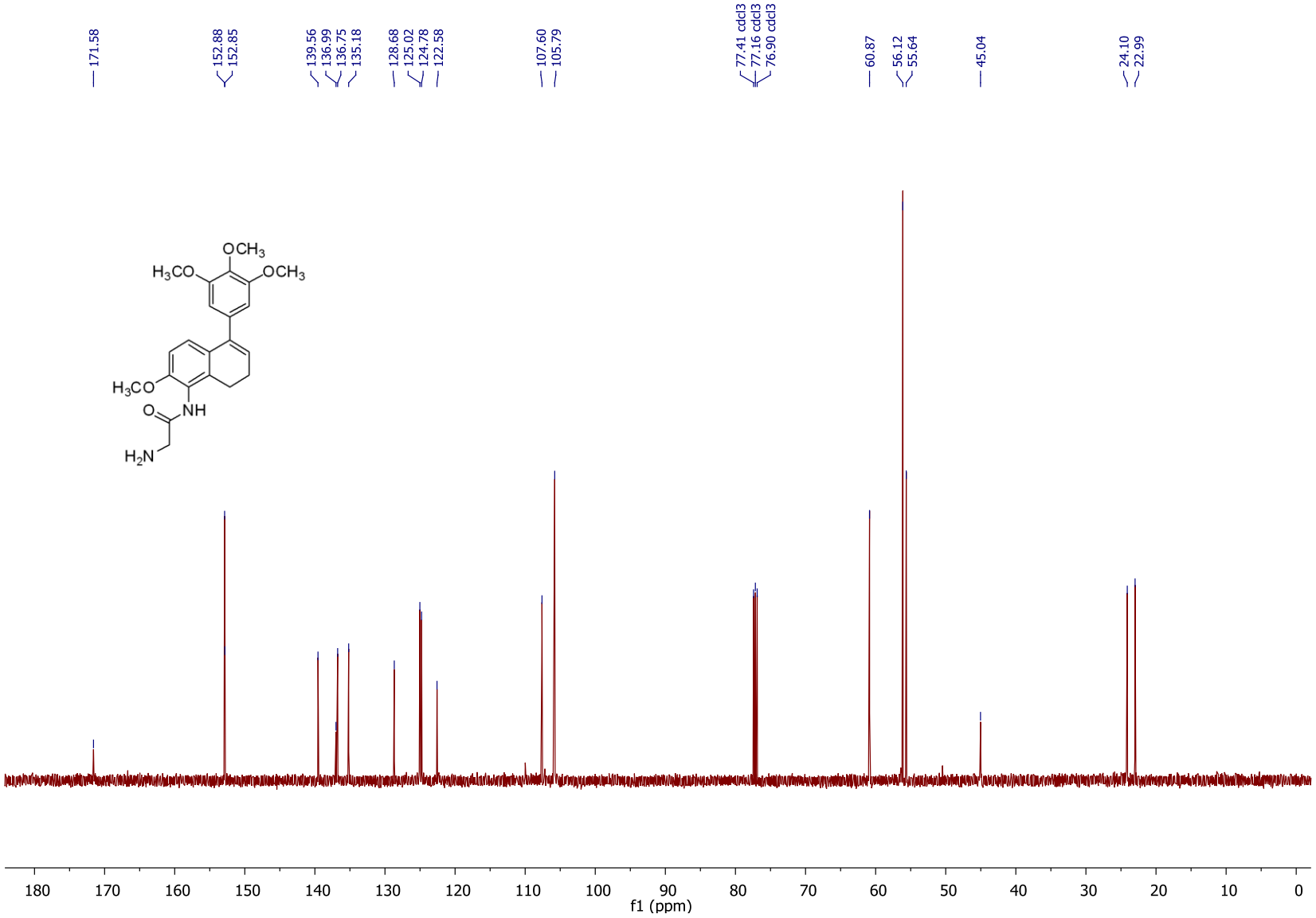
^{13}C NMR ($CDCl_3$, 125 MHz) Compound **41**



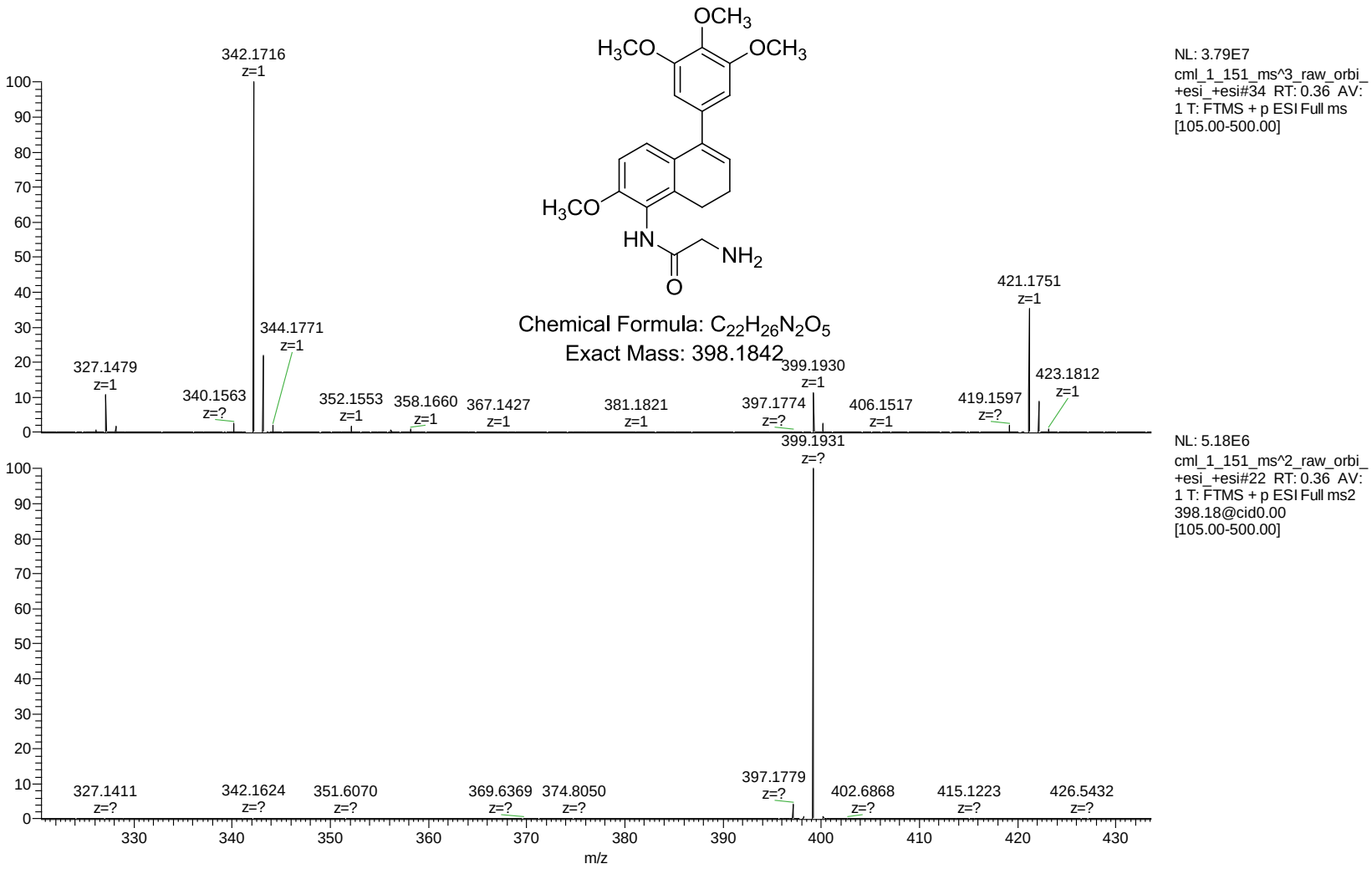
^{1}H NMR ($CDCl_3$, 500 MHz) Compound **42**



^{13}C NMR ($CDCl_3$, 125 MHz) Compound **42**



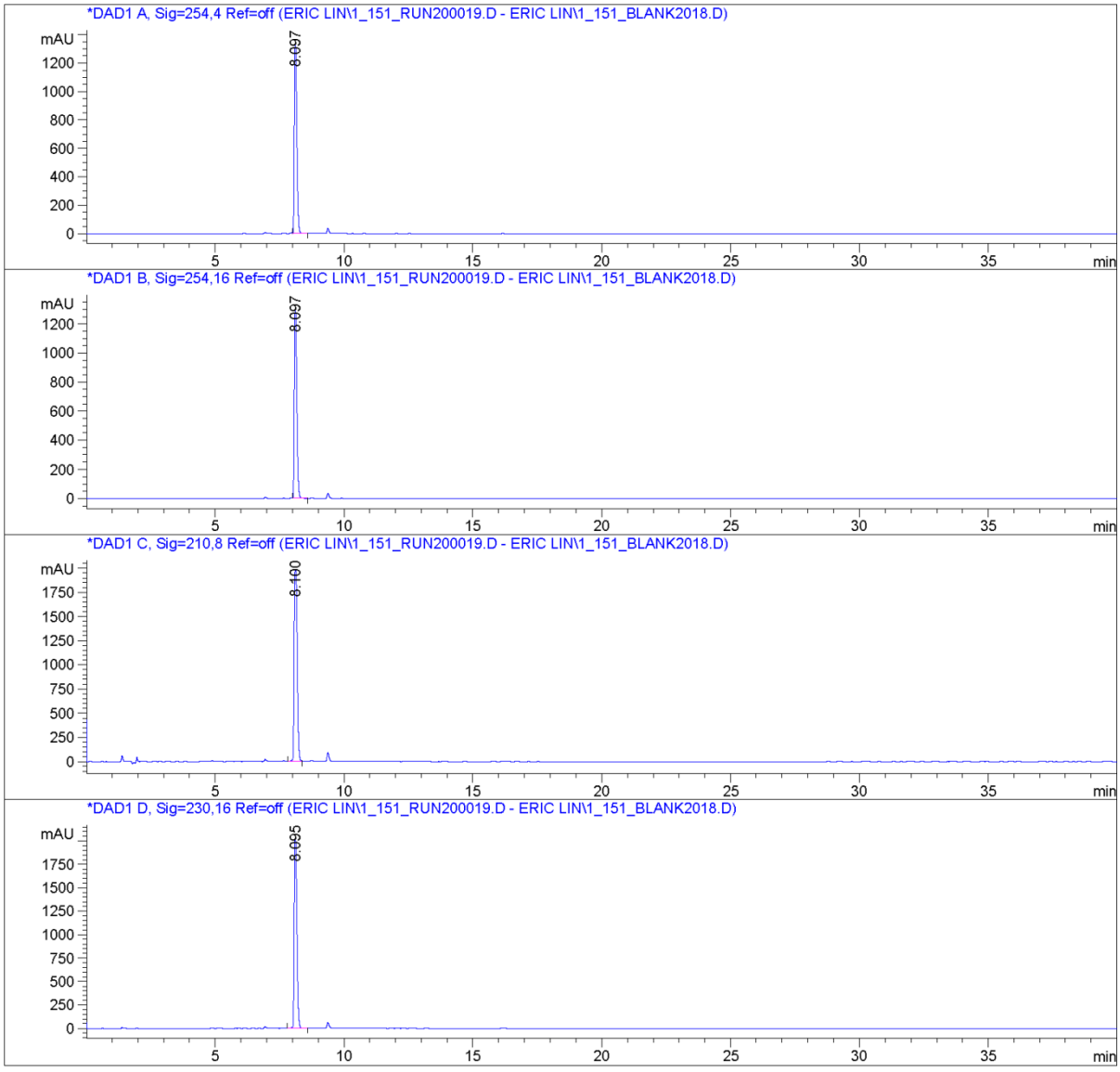
HRMS Compound **42**



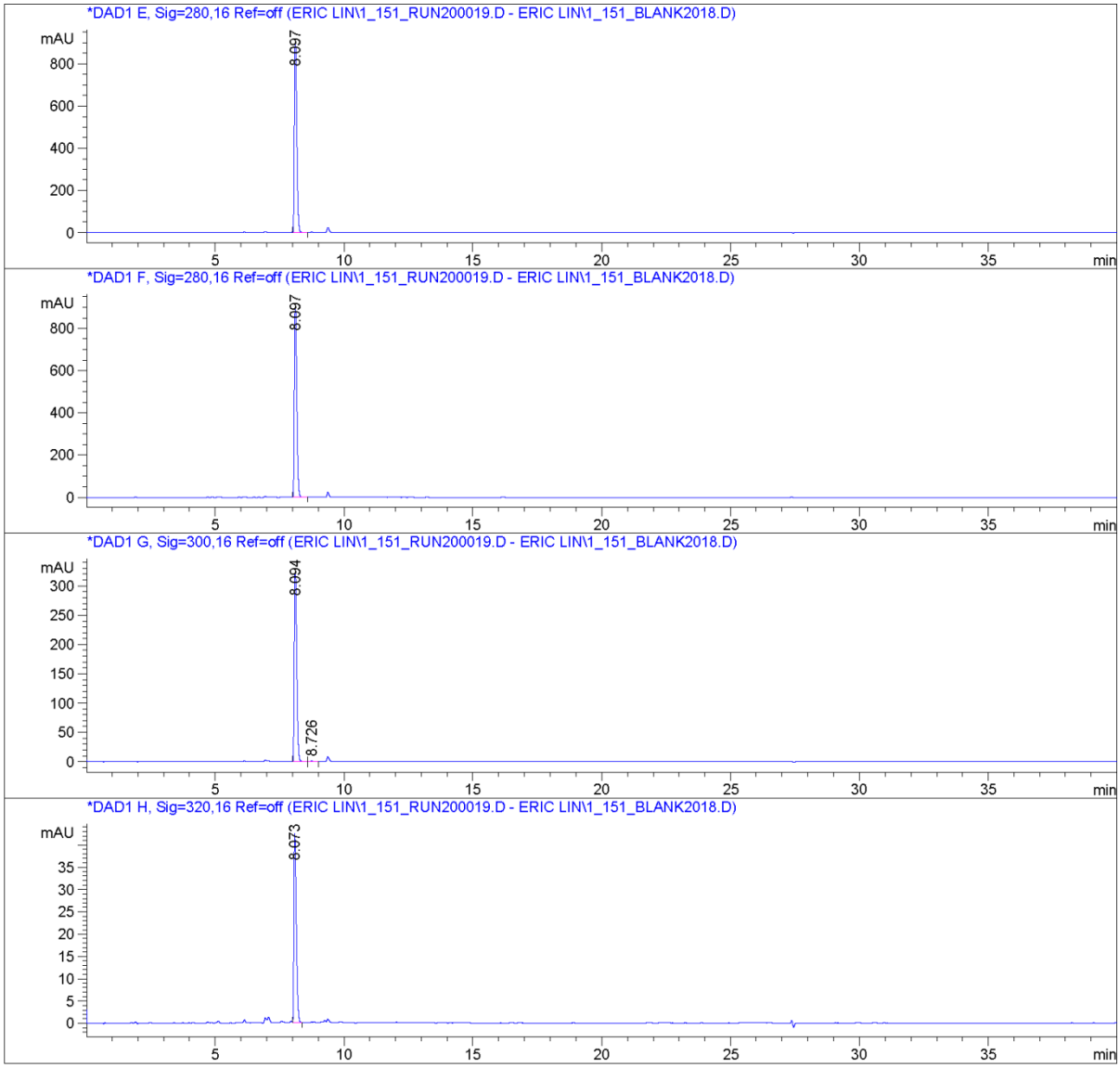
HPLC for Compound **42**

Data File C:\CHEM32\1\DATA\ERIC LIN\1_151_RUN200019.D
Sample Name: CML_1_151_run2

```
==========================================================================
Acq. Operator   : ERIC LIN
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 8/6/2012 3:44:48 PM
Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed    : 8/6/2012 3:41:45 PM by ERIC LIN
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed    : 8/6/2012 1:00:08 PM by ERIC LIN
                  (modified after loading)
```



Data File C:\CHEM32\1\DATA\ERIC LIN\1_151_RUN200019.D
Sample Name: CML_1_151_run2



==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Data File C:\CHEM32\1\DATA\ERIC LIN\1_151_RUN200019.D
Sample Name: CML_1_151_run2

Signal 1: DAD1 A, Sig=254,4 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.097	VB	0.1019	8863.30371	1363.22180	100.0000
Totals :				8863.30371	1363.22180	

Signal 2: DAD1 B, Sig=254,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.097	VB	0.1039	8660.84570	1330.41003	100.0000
Totals :				8660.84570	1330.41003	

Signal 3: DAD1 C, Sig=210,8 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.100	BV	0.1328	1.61150e4	1982.65149	100.0000
Totals :				1.61150e4	1982.65149	

Signal 4: DAD1 D, Sig=230,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.095	BV	0.1118	1.44809e4	2065.76636	100.0000
Totals :				1.44809e4	2065.76636	

Data File C:\CHEM32\1\DATA\ERIC LIN\1_151_RUN200019.D
Sample Name: CML_1_151_run2

Signal 5: DAD1 E, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.097	VB	0.1017	5946.59521	916.38574	100.0000
Totals :				5946.59521	916.38574	

Signal 6: DAD1 F, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.097	VB	0.1017	5946.59521	916.38574	100.0000
Totals :				5946.59521	916.38574	

Signal 7: DAD1 G, Sig=300,16 Ref=off
Signal has been modified after loading from rawdata file!

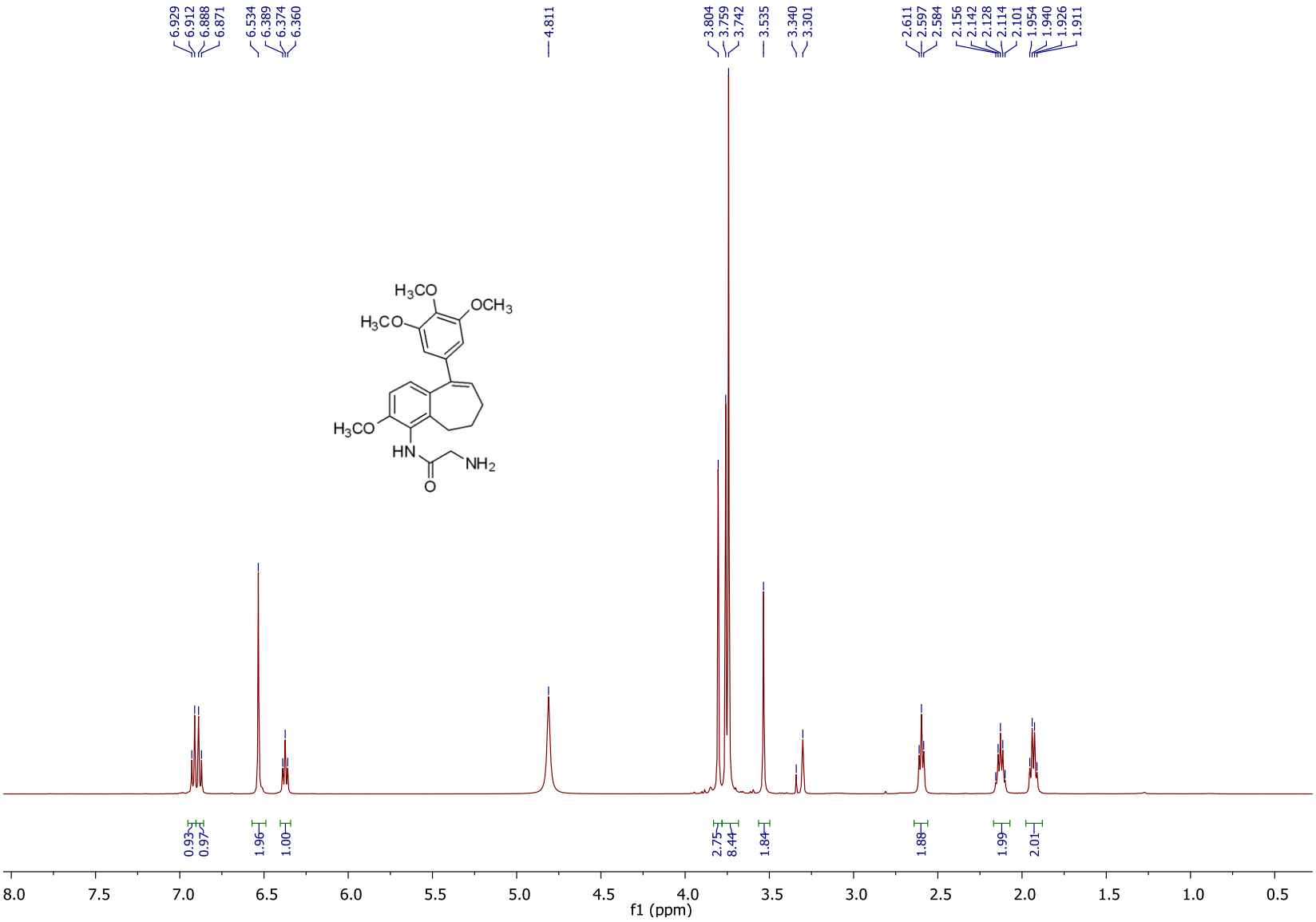
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.094	VB	0.1049	2171.17407	329.57730	99.5934
2	8.726	BB	0.1166	8.86310	1.02736	0.4066
Totals :				2180.03717	330.60466	

Signal 8: DAD1 H, Sig=320,16 Ref=off
Signal has been modified after loading from rawdata file!

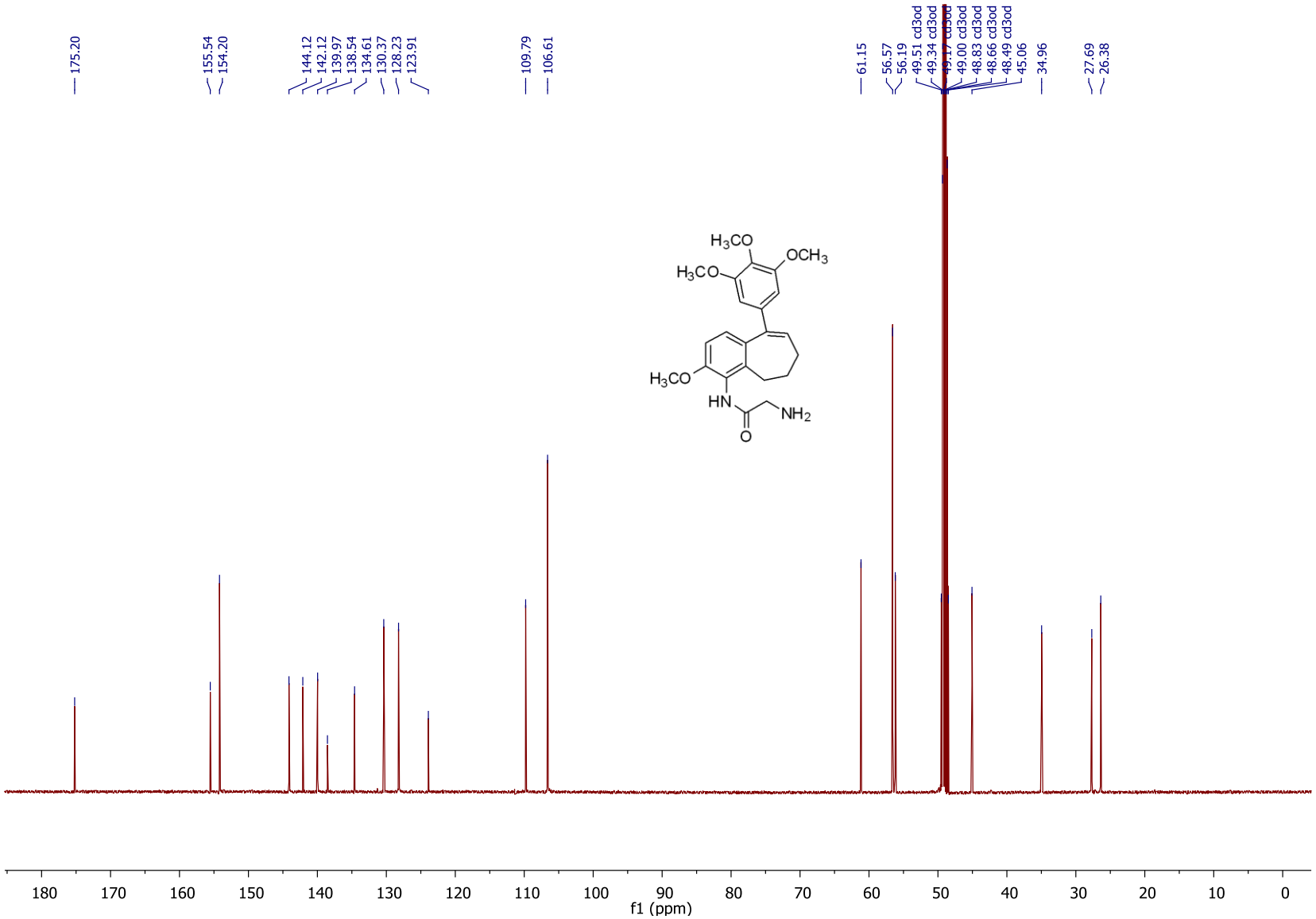
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.073	VB	0.1002	270.49811	42.54499	100.0000
Totals :				270.49811	42.54499	

*** End of Report ***

^{1}H NMR (CD_3OD, 500 MHz) Compound **43**



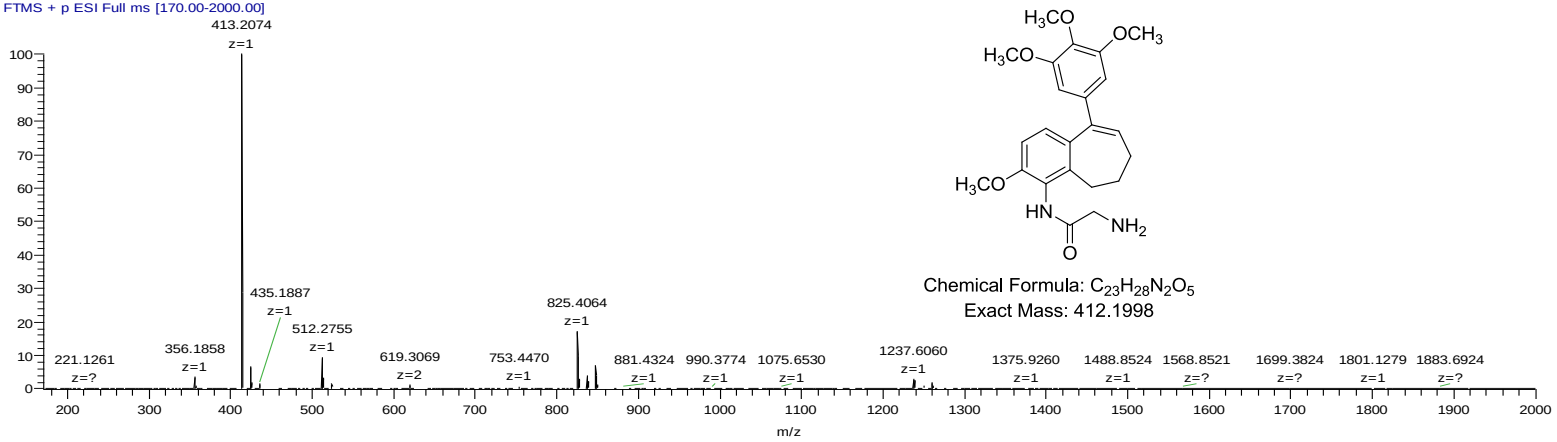
^{13}C NMR (CD_3OD, 125 MHz) Compound **43**



HRMS Compound **43**

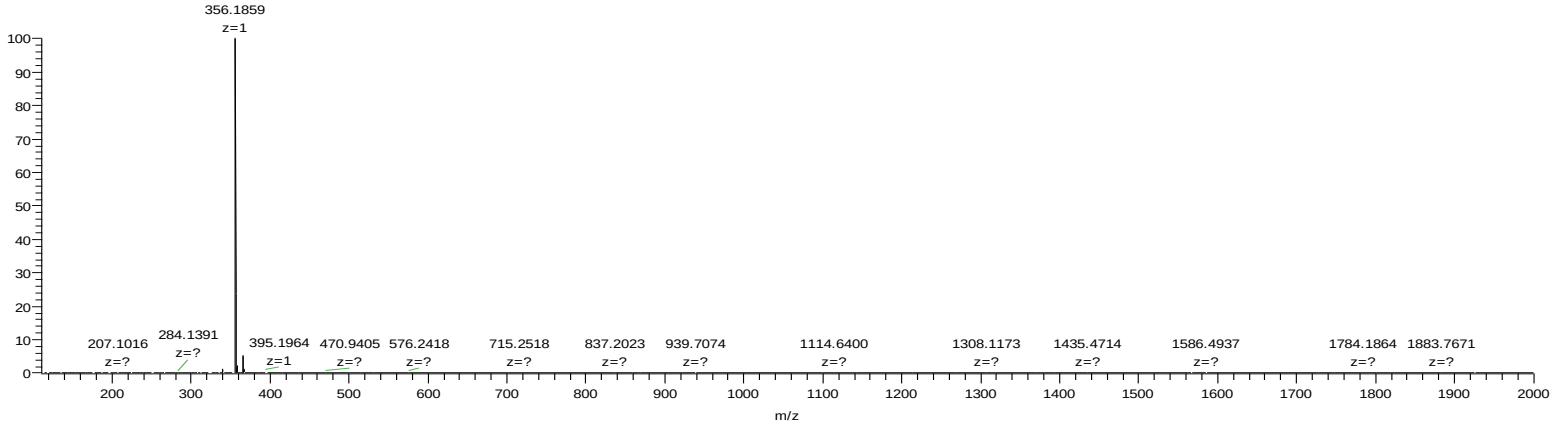
C:\Xcalibur\...\CML_II_091_ORBI_+ESI 2/13/2013 5:26:54 PM CML_II_091

CML_II_091_ORBI_+ESI #20 RT: 0.18 AV: 1 NL
T: FTMS + p ESI Full ms [170.00-2000.00]



Chemical Formula: $C_{23}H_{28}N_2O_5$
Exact Mass: 412.1998

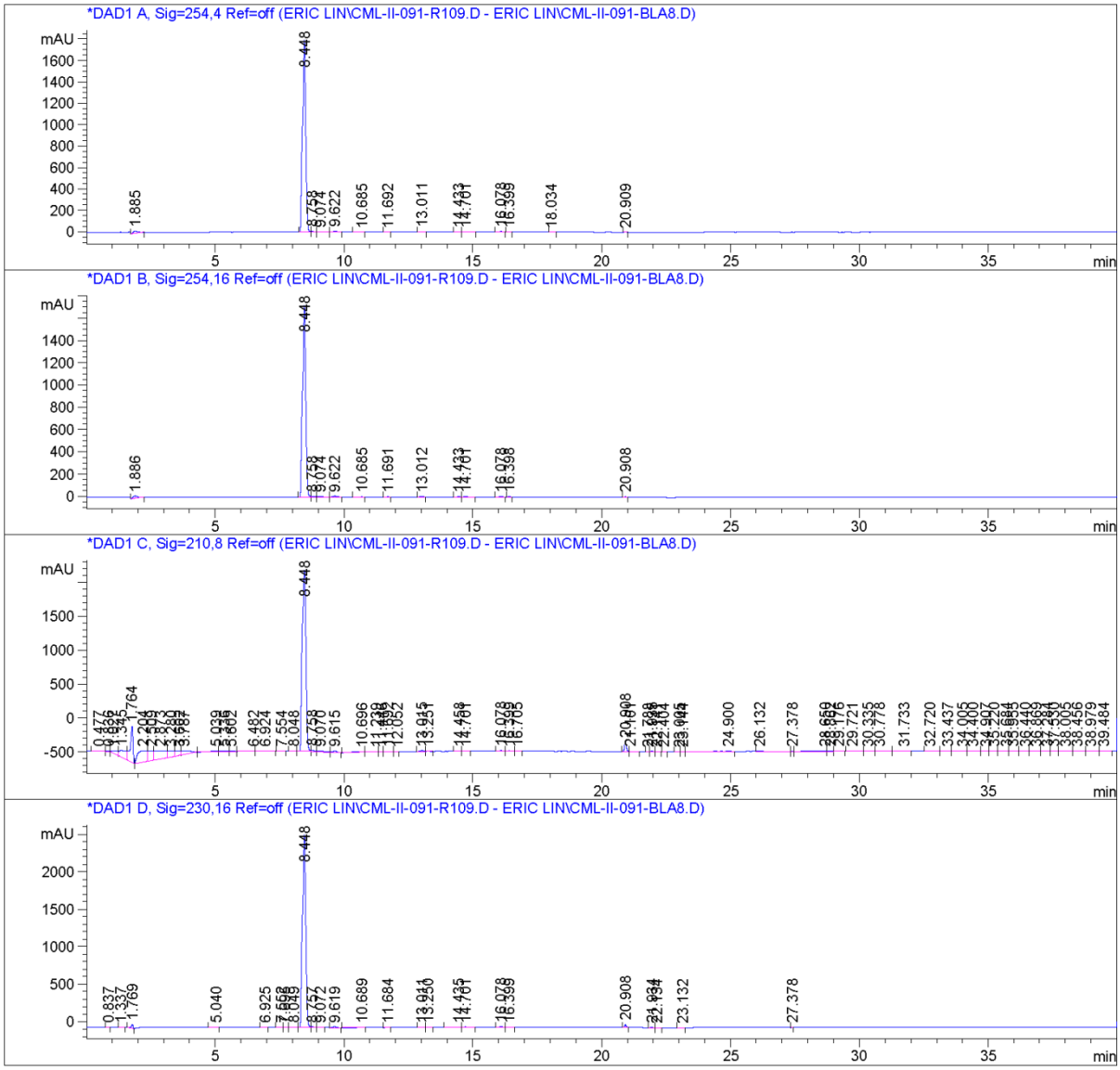
cml_ii_091_fs_orbi_+esi #16 RT: 0.18 AV: 1 NL: 2.41E9
T: FTMS + p ESI Full ms2 413.21@cid35.00 [110.00-2000.00]



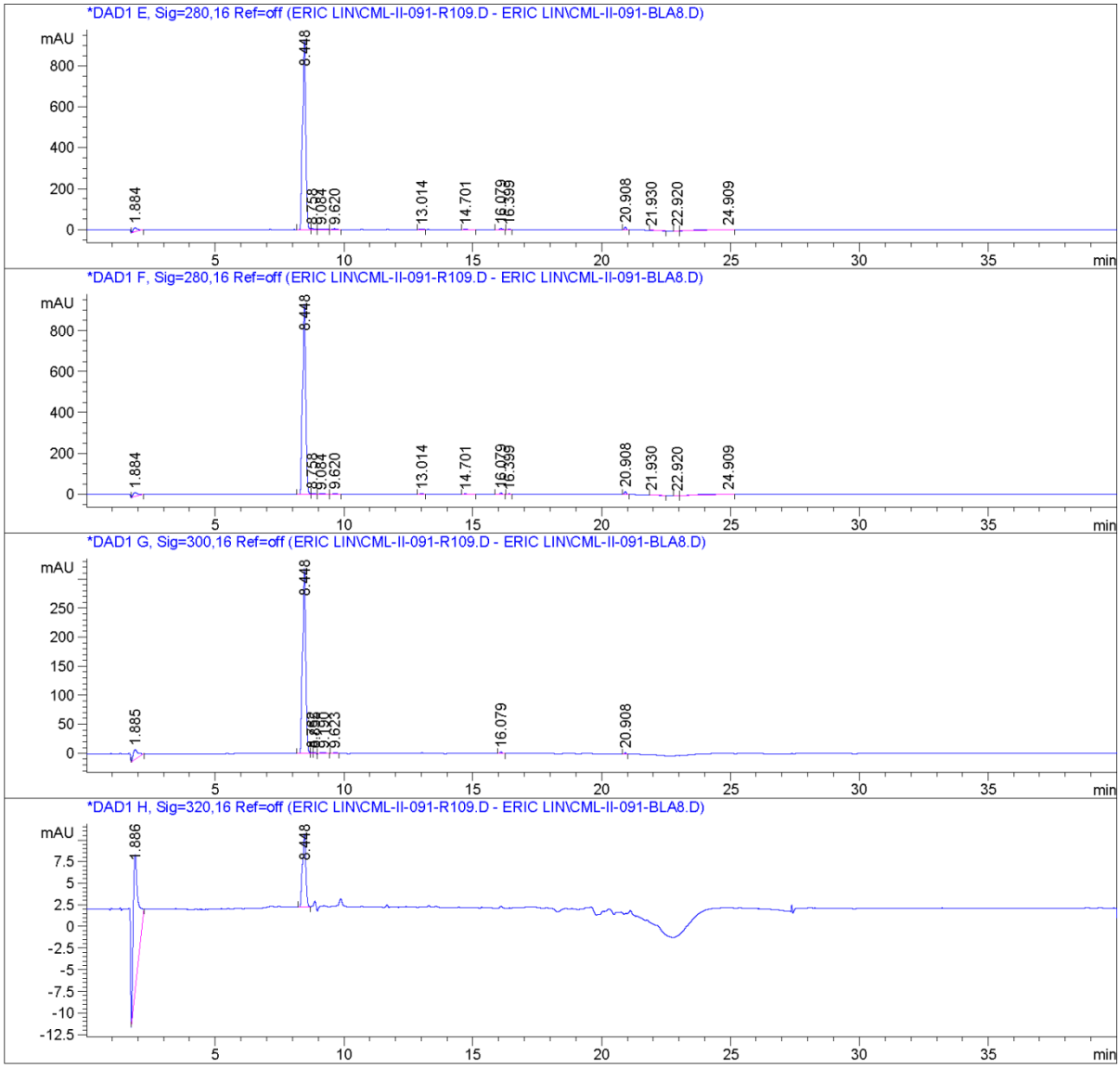
HPLC for Compound **43**

Data File C:\CHEM32\1\DATA\ERIC LIN\CML-II-091-R109.D
Sample Name: CML-II-091-run1

```
======================================================================
Acq. Operator   : Eric Lin
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 2/13/2013 3:57:47 PM
Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed    : 2/13/2013 3:54:29 PM by Eric Lin
Analysis Method : C:\CHEM32\1\DATA\ERIC LIN\CML-II-091-R109.D\DA.M (MASTERMETHOD.M)
Last changed    : 2/13/2013 4:48:00 PM by Eric Lin
```



Data File C:\CHEM32\1\DATA\ERIC LIN\CML-II-091-R109.D
Sample Name: CML-II-091-run1



==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Data File C:\CHEM32\1\DATA\ERIC LIN\CML-II-091-R109.D
Sample Name: CML-II-091-run1

Signal 1: DAD1 A, Sig=254,4 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.885	BB	0.2087	303.67535	22.32442	1.8885
2	8.448	BV	0.1222	1.54340e4	1796.28943	95.9830
3	8.758	VV	0.1012	43.91451	5.87406	0.2731
4	9.074	VB	0.1413	30.87476	2.92129	0.1920
5	9.622	BB	0.1462	81.67364	7.67224	0.5079
6	10.685	BB	0.1368	10.20960	1.03738	0.0635
7	11.692	BB	0.1086	8.05322	1.19470	0.0501
8	13.011	BV	0.1354	46.89869	5.62008	0.2917
9	14.433	BV	0.0991	17.61300	2.66349	0.1095
10	14.701	VB	0.0991	31.21455	4.85003	0.1941
11	16.078	BB	0.0893	42.34123	7.33771	0.2633
12	16.399	BB	0.0791	11.89078	2.34849	0.0739
13	18.034	BB	0.0979	7.92484	1.18623	0.0493
14	20.909	BB	0.0737	9.65385	2.09784	0.0600
Totals :				1.60800e4	1863.41738	

Signal 2: DAD1 B, Sig=254,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.886	BB	0.2082	292.19580	21.55061	1.8910
2	8.448	BV	0.1222	1.48160e4	1725.56189	95.8863
3	8.758	VV	0.1040	46.97472	6.09177	0.3040
4	9.074	VV	0.1676	40.01356	3.12126	0.2590
5	9.622	VB	0.1509	84.10874	7.61149	0.5443
6	10.685	BB	0.1363	9.81310	1.00172	0.0635
7	11.691	BB	0.1095	8.22453	1.17736	0.0532
8	13.012	BV	0.1353	44.79118	5.37278	0.2899
9	14.433	BV	0.1025	14.41688	2.14135	0.0933
10	14.701	VB	0.0986	30.12296	4.71390	0.1950
11	16.078	BB	0.0892	41.94569	7.27688	0.2715
12	16.398	BB	0.0787	11.48548	2.28234	0.0743
13	20.908	BB	0.0722	11.53259	2.48224	0.0746
Totals :				1.54516e4	1790.38559	

Data File C:\CHEM32\1\DATA\ERIC LIN\CML-II-091-R109.D
Sample Name: CML-II-091-run1

Signal 3: DAD1 C, Sig=210,8 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	0.477	BV	0.2906	92.21676	4.09649	0.1728
2	0.836	VB	0.0805	53.20325	9.33237	0.0997
3	1.027	BV	0.3305	670.57526	27.30559	1.2565
4	1.345	VV	0.2675	2115.05591	99.71152	3.9631
5	1.764	VB	0.1102	3927.80103	531.70923	7.3597
6	2.204	BV	0.4097	4284.61621	164.61928	8.0282
7	2.509	VV	0.2153	2180.02832	143.45102	4.0848
8	2.873	VV	0.3862	3652.52368	119.61574	6.8439
9	3.280	VV	0.2287	1475.25244	88.38440	2.7642
10	3.602	VV	0.2097	989.25763	65.68133	1.8536
11	3.787	VB	0.3572	1286.01184	52.61129	2.4096
12	5.039	BV	0.3271	351.56982	13.18396	0.6587
13	5.336	VV	0.2700	179.99951	8.26615	0.3373
14	5.602	VB	0.1725	54.66105	4.54023	0.1024
15	6.482	BV	0.3940	113.11806	3.49148	0.2120
16	6.924	VB	0.2796	163.18980	7.16936	0.3058
17	7.554	BB	0.1327	20.66869	2.30251	0.0387
18	8.048	BB	0.1226	46.82283	5.12485	0.0877
19	8.448	BV	0.1479	2.70409e4	2675.88184	50.6674
20	8.758	VV	0.1307	207.94093	20.79608	0.3896
21	9.070	VV	0.2763	388.74979	17.56201	0.7284
22	9.615	VB	0.1742	307.48911	23.89076	0.5762
23	10.696	BV	0.4404	335.67877	9.31619	0.6290
24	11.239	VV	0.4456	157.50700	4.35884	0.2951
25	11.446	VB	0.1477	21.31094	1.97792	0.0399
26	11.692	BV	0.1490	33.04355	3.24023	0.0619
27	12.052	VB	0.0919	7.33788	1.26097	0.0137
28	13.015	BV	0.1273	93.54889	11.94884	0.1753
29	13.251	VB	0.1058	10.98901	1.53050	0.0206
30	14.458	BV	0.1543	10.71459	1.09359	0.0201
31	14.701	VB	0.0971	60.09192	9.59263	0.1126
32	16.078	BV	0.0922	86.43350	14.35692	0.1620
33	16.399	VV	0.0996	68.47499	10.03284	0.1283
34	16.705	VB	0.1007	15.76245	2.27747	0.0295
35	20.908	BV	0.0773	689.77765	140.49957	1.2925
36	21.101	VB	0.1272	18.98328	2.06597	0.0356
37	21.788	BV	0.0849	7.57358	1.36091	0.0142
38	21.935	VV	0.0838	61.65607	11.26755	0.1155
39	22.141	VB	0.1169	15.22546	1.87094	0.0285
40	22.404	BB	0.0748	12.27476	2.61129	0.0230
41	23.005	BV	0.0896	12.24500	1.99634	0.0229
42	23.144	VV	0.1077	20.31472	2.70290	0.0381
43	24.900	VB	1.7347	340.25699	2.32597	0.6376
44	26.132	VB	0.9003	208.87184	2.81955	0.3914
45	27.378	BV	0.0418	19.50561	6.35763	0.0365
46	28.650	VV	1.0018	297.79904	3.55320	0.5580

Data File C:\CHEM32\1\DATA\ERIC LIN\CML-II-091-R109.D
Sample Name: CML-II-091-run1

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
47	28.862	VV	0.1958	47.13171	3.62181	0.0883
48	29.176	VB	0.2655	66.63641	3.78308	0.1249
49	29.721	BV	0.2670	154.19702	8.28990	0.2889
50	30.335	VV	0.2156	48.99574	3.14722	0.0918
51	30.778	VB	0.2976	74.64709	3.35520	0.1399
52	31.733	BB	0.3347	92.02336	3.45542	0.1724
53	32.720	BB	0.2971	61.00483	2.92722	0.1143
54	33.437	BV	0.2381	46.26827	2.61943	0.0867
55	34.005	VB	0.3248	74.92151	3.09050	0.1404
56	34.400	BB	0.2370	51.44723	3.50920	0.0964
57	34.907	BV	0.1647	32.06065	3.15101	0.0601
58	35.220	VB	0.2098	23.26075	1.56117	0.0436
59	35.684	BV	0.1994	24.30363	1.71397	0.0455
60	35.955	VB	0.1758	27.64740	2.24256	0.0518
61	36.440	BV	0.1933	25.35880	1.90497	0.0475
62	36.869	VB	0.1773	39.94121	3.39431	0.0748
63	37.284	BV	0.2028	38.22689	2.84392	0.0716
64	37.530	VV	0.1852	34.93135	2.76789	0.0655
65	38.005	VV	0.2773	62.55053	2.95101	0.1172
66	38.456	VB	0.2762	47.11165	2.49561	0.0883
67	38.979	BV	0.2542	45.89429	2.49911	0.0860
68	39.484	VB	0.2220	43.82164	3.15564	0.0821
Totals :				5.33694e4	4407.62637	

Signal 4: DAD1 D, Sig=230,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	0.837	BB	0.0781	5.56431	1.20046	0.0231
2	1.337	BB	0.1077	13.39890	1.86771	0.0556
3	1.769	BB	0.0773	272.98090	53.73421	1.1318
4	5.040	BB	0.0987	10.24736	1.44641	0.0425
5	6.925	VB	0.1197	13.83992	1.52722	0.0574
6	7.552	BV	0.1321	16.18779	1.77921	0.0671
7	7.695	VV	0.0873	7.71480	1.26159	0.0320
8	8.049	VB	0.1263	19.13415	2.02378	0.0793
9	8.448	BV	0.1282	2.29623e4	2571.97168	95.2053
10	8.757	VV	0.1048	72.72637	9.34707	0.3015
11	9.072	VB	0.1160	37.48595	4.46290	0.1554
12	9.619	BB	0.1460	136.21893	12.81127	0.5648
13	10.689	BB	0.3011	48.31770	2.01960	0.2003
14	11.684	BB	0.1430	24.21141	2.59154	0.1004
15	13.011	BV	0.1363	53.04623	6.29419	0.2199
16	13.250	VB	0.0996	7.95180	1.19580	0.0330
17	14.435	BV	0.3300	32.17361	1.30322	0.1334

Data File C:\CHEM32\1\DATA\ERIC LIN\CML-II-091-R109.D
Sample Name: CML-II-091-run1

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
18	14.701	VB	0.1034	54.05129	7.93924	0.2241
19	16.078	BB	0.0889	73.22536	12.76157	0.3036
20	16.399	BV	0.1012	36.36917	5.22610	0.1508
21	20.908	BB	0.0765	171.37622	35.40434	0.7106
22	21.934	BV	0.0841	17.05586	3.10618	0.0707
23	22.134	VV	0.1344	11.14204	1.17699	0.0462
24	23.132	BB	0.1046	11.67745	1.64870	0.0484
25	27.378	BB	0.0323	10.31213	4.56581	0.0428
Totals :				2.41187e4	2748.66680	

Signal 5: DAD1 E, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.884	BB	0.1919	257.12570	19.75065	2.9011
2	8.448	BV	0.1221	7991.19873	931.65533	90.1645
3	8.758	VV	0.1162	37.21556	4.24881	0.4199
4	9.084	VV	0.2509	55.30942	2.74731	0.6241
5	9.620	VV	0.1660	57.15676	4.62927	0.6449
6	13.014	BV	0.1304	25.37619	3.13567	0.2863
7	14.701	BB	0.0984	14.28490	2.24054	0.1612
8	16.079	BB	0.0889	33.30441	5.80521	0.3758
9	16.399	BB	0.0788	7.59325	1.50537	0.0857
10	20.908	BB	0.0766	66.62645	13.74501	0.7517
11	21.930	BB	0.2128	18.59188	1.12523	0.2098
12	22.920	BV	0.1313	11.14157	1.33532	0.1257
13	24.909	VB	2.4737	287.98969	1.36046	3.2494
Totals :				8862.91449	993.28419	

Signal 6: DAD1 F, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.884	BB	0.1919	257.12570	19.75065	2.9011
2	8.448	BV	0.1221	7991.19873	931.65533	90.1645
3	8.758	VV	0.1162	37.21556	4.24881	0.4199
4	9.084	VV	0.2509	55.30942	2.74731	0.6241
5	9.620	VV	0.1660	57.15676	4.62927	0.6449
6	13.014	BV	0.1304	25.37619	3.13567	0.2863
7	14.701	BB	0.0984	14.28490	2.24054	0.1612

Data File C:\CHEM32\1\DATA\ERIC LIN\CML-II-091-R109.D
Sample Name: CML-II-091-run1

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
8	16.079	BB	0.0889	33.30441	5.80521	0.3758
9	16.399	BB	0.0788	7.59325	1.50537	0.0857
10	20.908	BB	0.0766	66.62645	13.74501	0.7517
11	21.930	BB	0.2128	18.59188	1.12523	0.2098
12	22.920	BV	0.1313	11.14157	1.33532	0.1257
13	24.909	VB	2.4737	287.98969	1.36046	3.2494
Totals :				8862.91449	993.28419	

Signal 7: DAD1 G, Sig=300,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.885	BB	0.1997	241.76862	17.66125	7.8587
2	8.448	BV	0.1224	2746.77637	319.03760	89.2840
3	8.762	VV	0.0701	9.90111	1.98929	0.3218
4	8.855	VB	0.0902	14.08478	2.41078	0.4578
5	9.190	BB	0.2484	28.60969	1.50082	0.9300
6	9.623	BB	0.1341	11.87181	1.23573	0.3859
7	16.079	BB	0.0887	15.36226	2.68666	0.4994
8	20.908	BB	0.0719	8.07447	1.74746	0.2625
Totals :				3076.44910	348.26959	

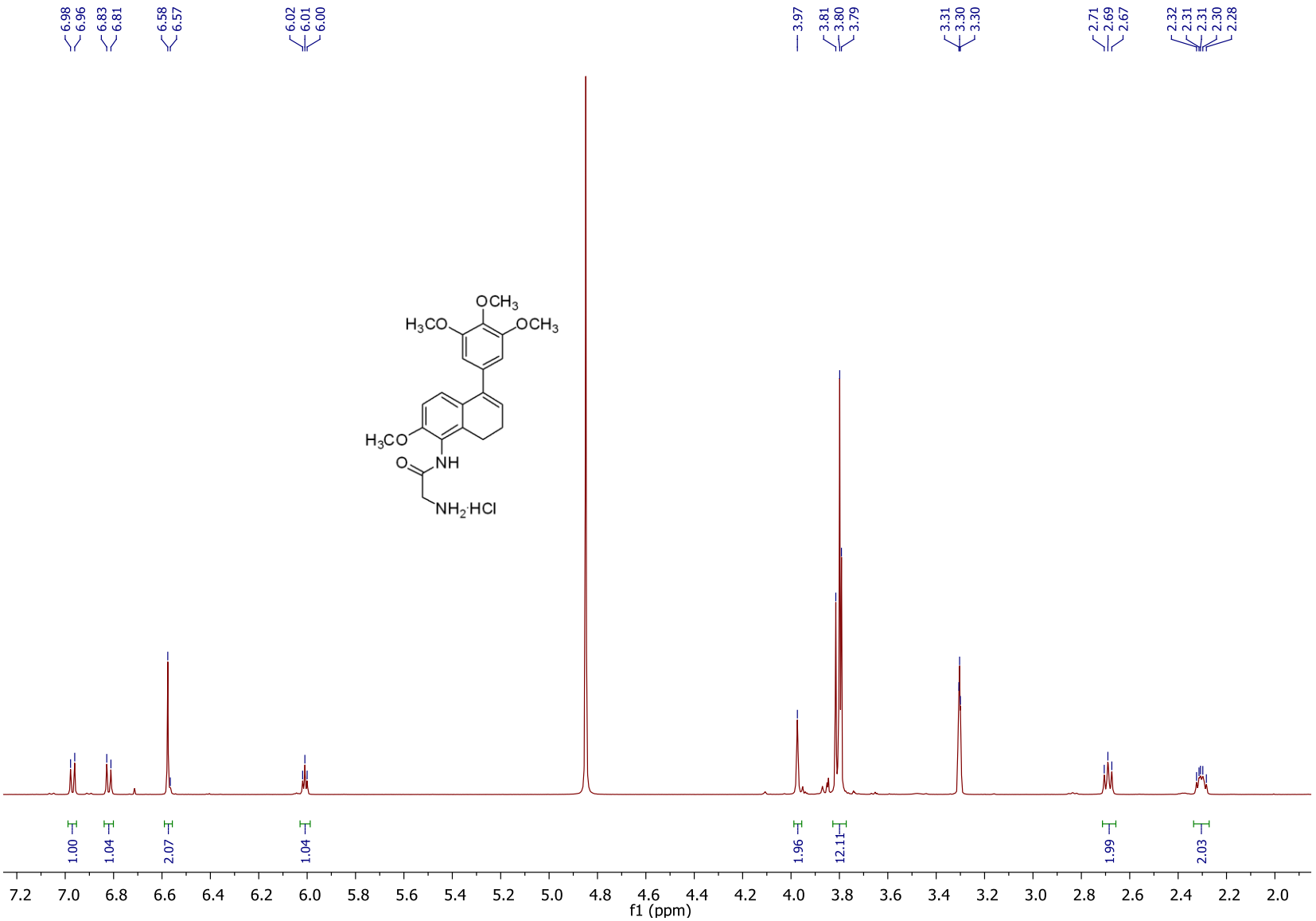
Signal 8: DAD1 H, Sig=320,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.886	BB	0.1964	216.25700	15.92820	73.9221
2	8.448	BB	0.1286	76.29005	8.50886	26.0779
Totals :				292.54706	24.43706	

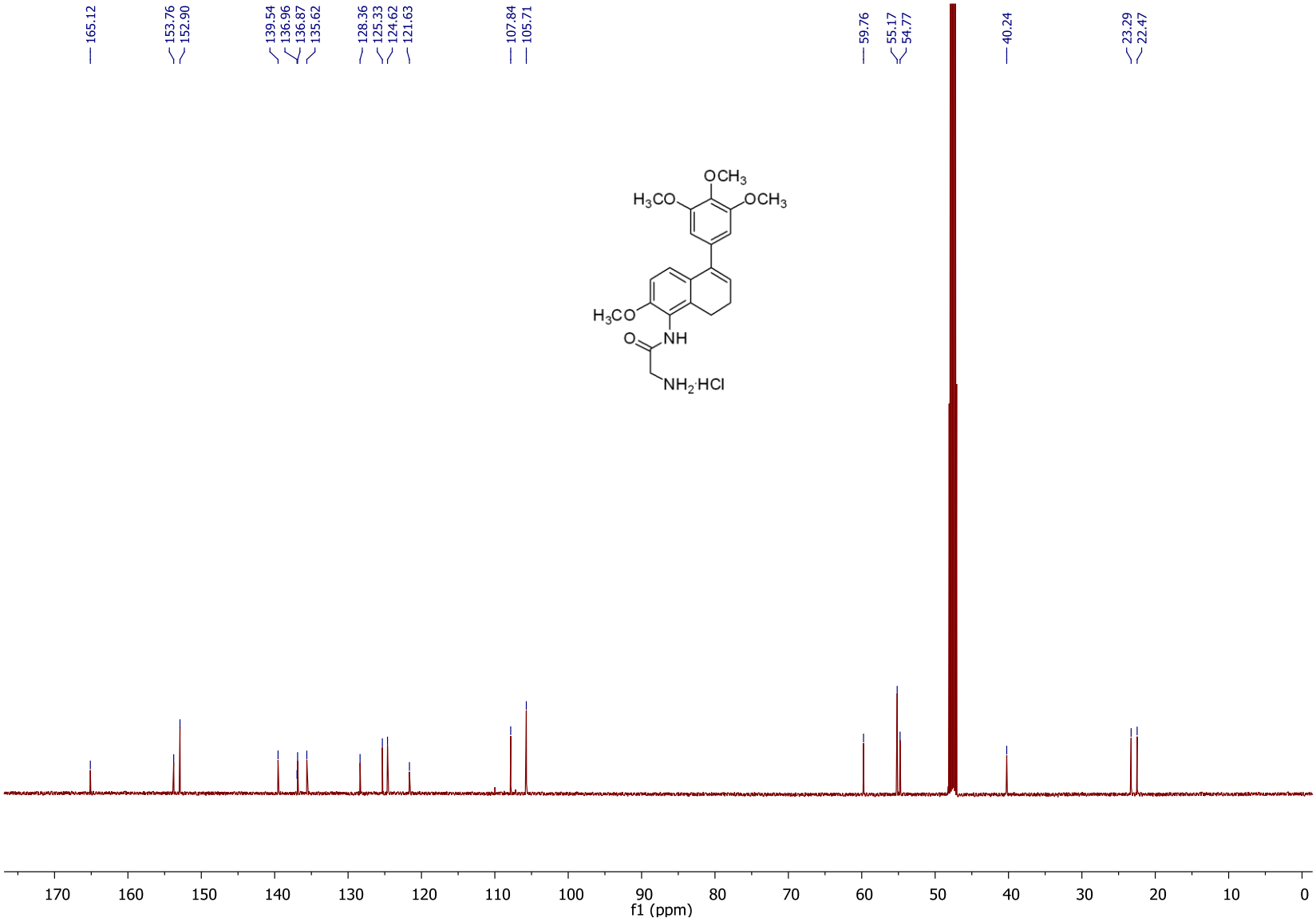
==========

*** End of Report ***

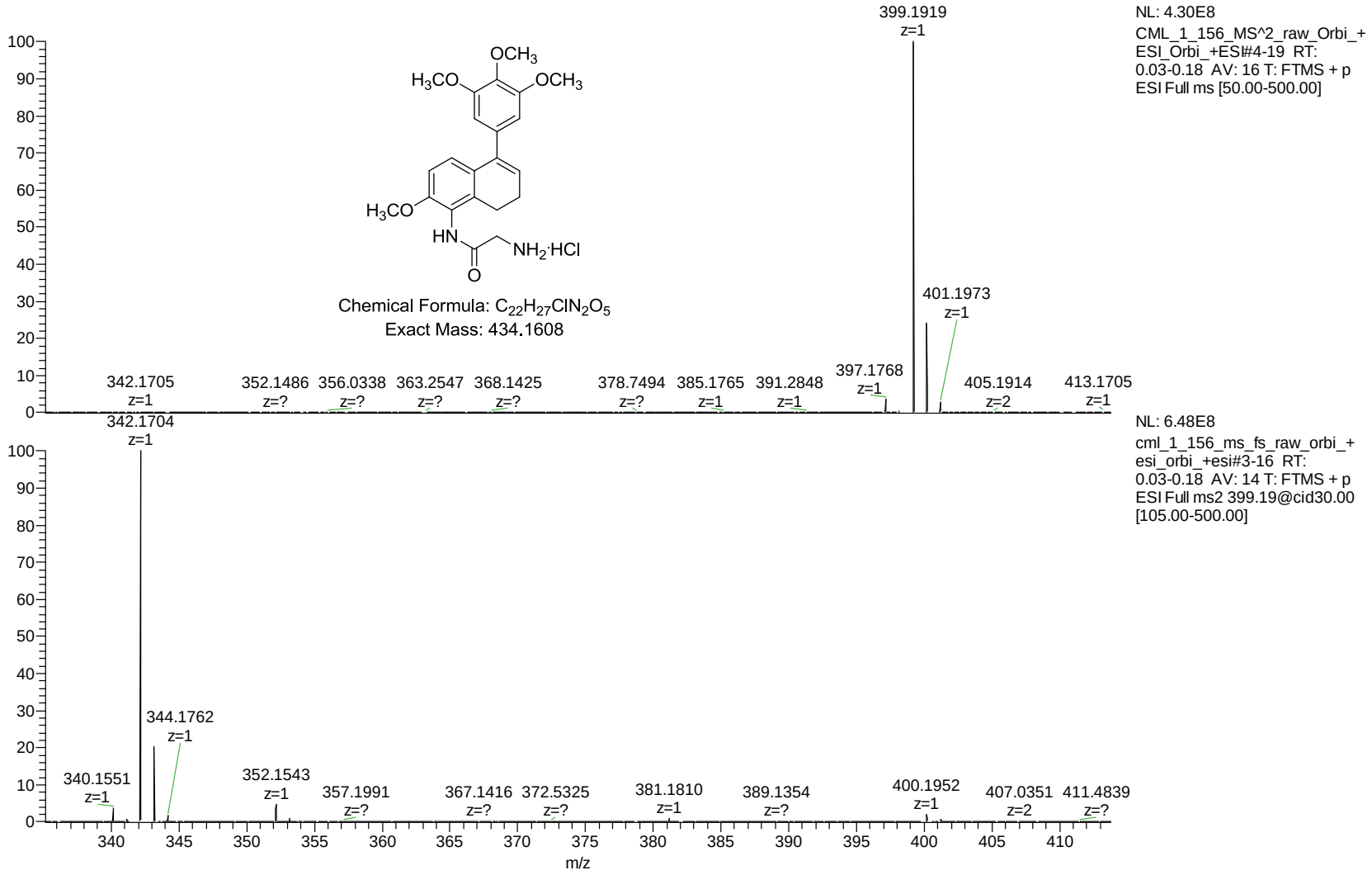
^{1}H NMR (CD_3OD, 500 MHz) Compound **44**



^{13}C NMR (CD_3OD, 125 MHz) Compound **44**



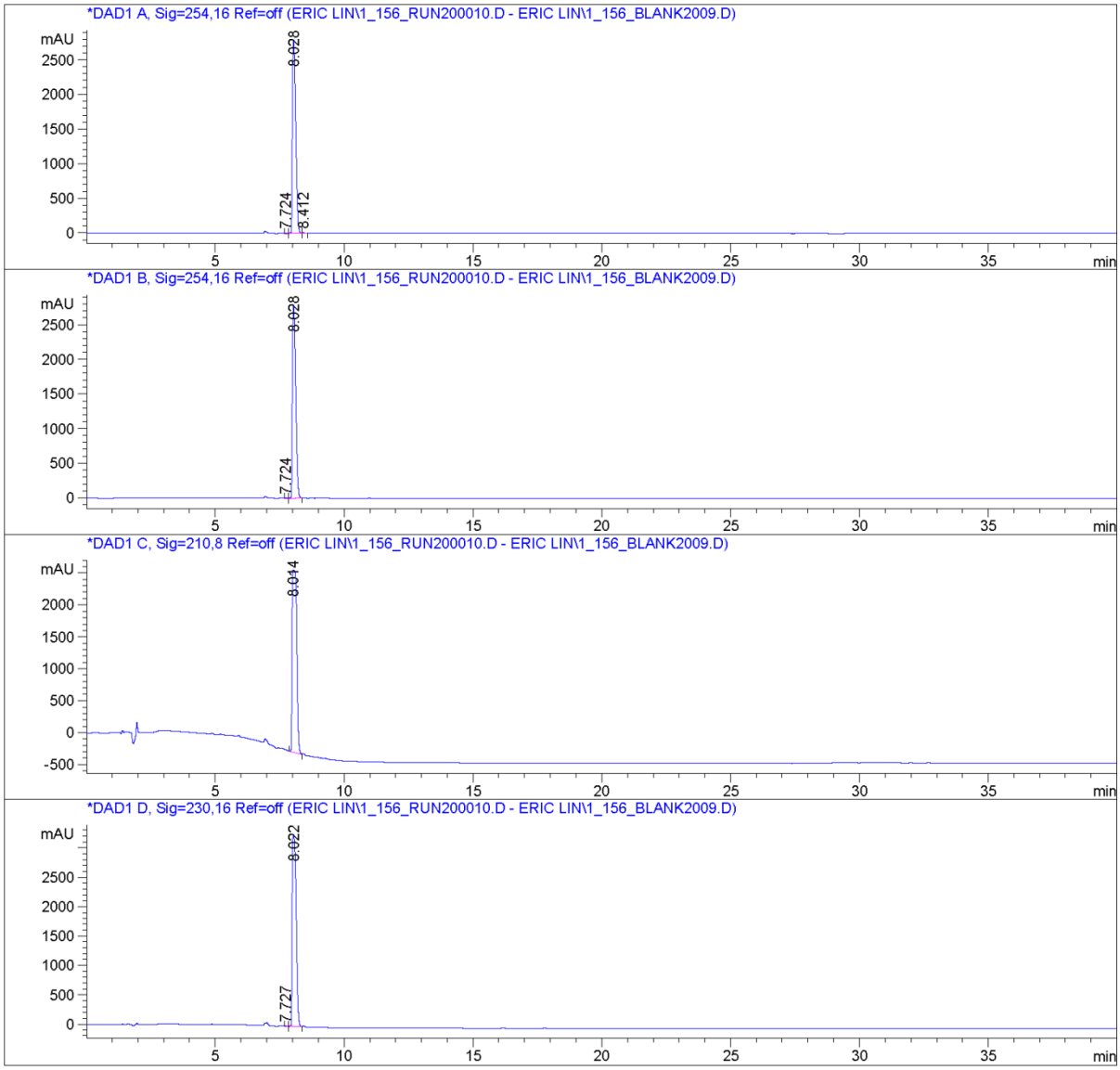
HRMS Compound **44**



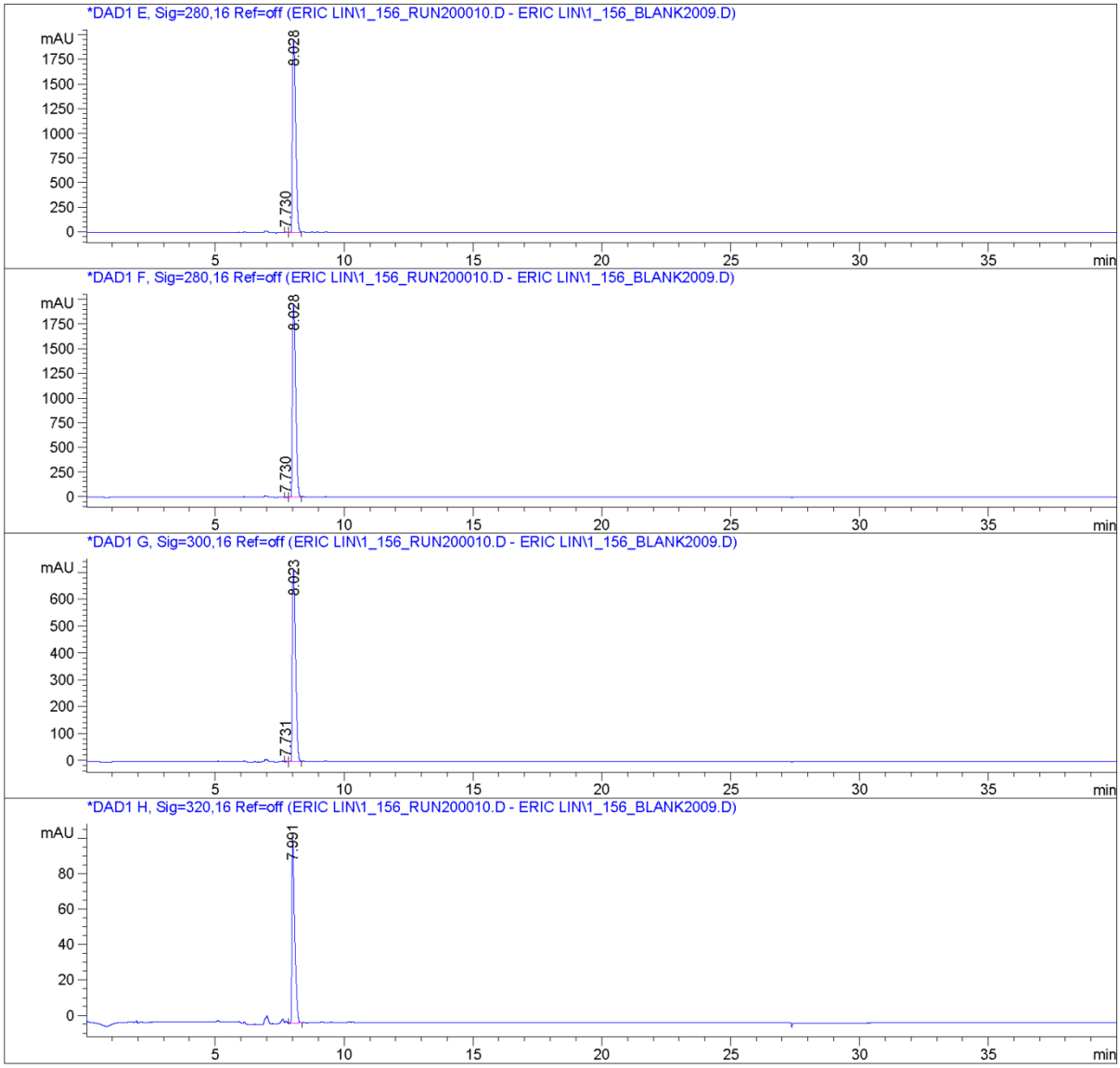
HPLC for Compound **44**

Data File C:\CHEM32\1\DATA\ERIC LIN\1_156_RUN200010.D
Sample Name: CML_1_156_run2

```
==========================================================================
Acq. Operator   : ERIC LIN
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 8/7/2012 9:41:12 PM
Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed    : 8/7/2012 9:37:06 PM by ERIC LIN
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed    : 8/7/2012 10:31:04 PM by ERIC LIN
                  (modified after loading)
```



Data File C:\CHEM32\1\DATA\ERIC LIN\1_156_RUN200010.D
Sample Name: CML_1_156_run2



==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Data File C:\CHEM32\1\DATA\ERIC LIN\1_156_RUN200010.D
Sample Name: CML_1_156_run2

Signal 1: DAD1 A, Sig=254,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.724	VV	0.1122	58.09821	6.90150	0.2336
2	8.028	VV	0.1413	2.47516e4	2794.96948	99.5184
3	8.412	VB	0.0864	61.67973	10.51692	0.2480
Totals :				2.48714e4	2812.38790	

Signal 2: DAD1 B, Sig=254,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.724	VV	0.1122	58.09821	6.90150	0.2342
2	8.028	VV	0.1413	2.47516e4	2794.96948	99.7658
Totals :				2.48097e4	2801.87099	

Signal 3: DAD1 C, Sig=210,8 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.014	VV	0.1645	3.54314e4	2861.09155	100.0000
Totals :				3.54314e4	2861.09155	

Signal 4: DAD1 D, Sig=230,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.727	VB	0.0929	50.91706	7.52660	0.1468
2	8.022	BV	0.1757	3.46356e4	3266.68286	99.8532
Totals :				3.46865e4	3274.20946	

Data File C:\CHEM32\1\DATA\ERIC LIN\1_156_RUN200010.D
Sample Name: CML_1_156_run2

Signal 5: DAD1 E, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.730	VV	0.1146	51.66423	6.11322	0.3020
2	8.028	VV	0.1375	1.70565e4	1960.77515	99.6980
Totals :				1.71082e4	1966.88837	

Signal 6: DAD1 F, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.730	VV	0.1146	51.66423	6.11322	0.3020
2	8.028	VV	0.1375	1.70565e4	1960.77515	99.6980
Totals :				1.71082e4	1966.88837	

Signal 7: DAD1 G, Sig=300,16 Ref=off
Signal has been modified after loading from rawdata file!

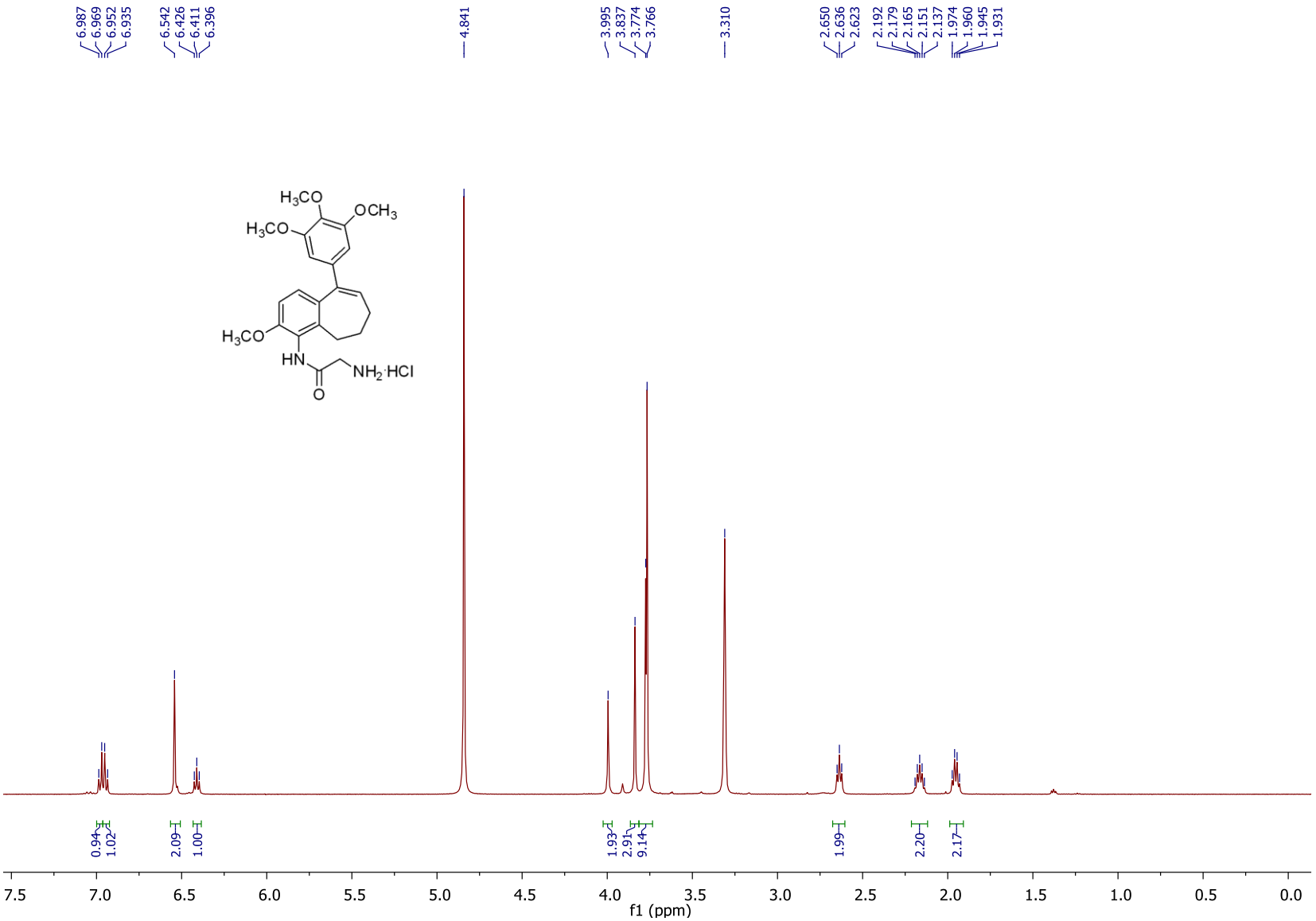
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.731	VV	0.0987	26.19738	3.69741	0.4170
2	8.023	VV	0.1374	6256.75488	719.75366	99.5830
Totals :				6282.95226	723.45107	

Signal 8: DAD1 H, Sig=320,16 Ref=off
Signal has been modified after loading from rawdata file!

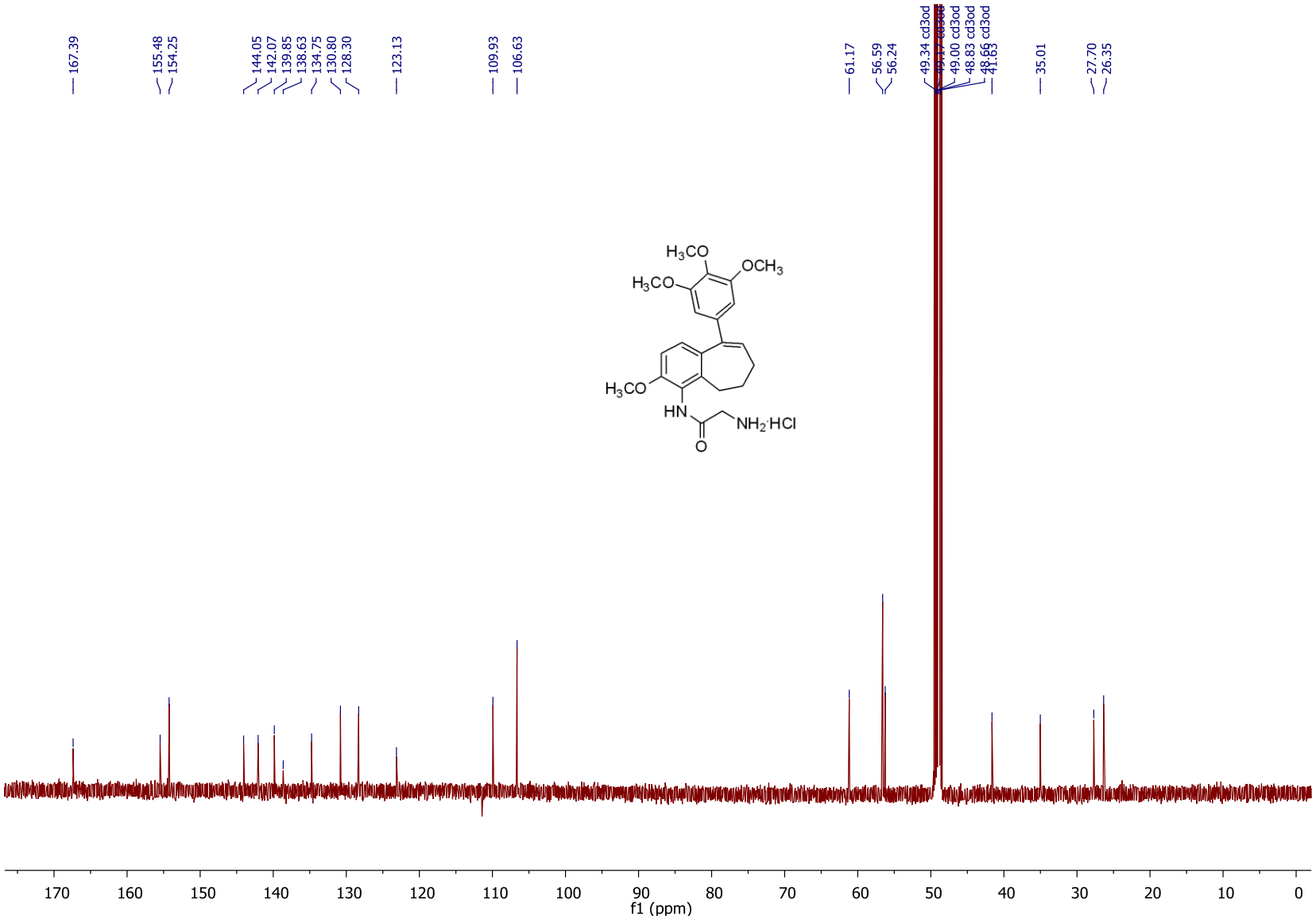
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.991	VV	0.1072	785.54877	107.56760	100.0000
Totals :				785.54877	107.56760	

*** End of Report ***

^{1}H NMR (CD_3OD, 500 MHz) Compound **45**



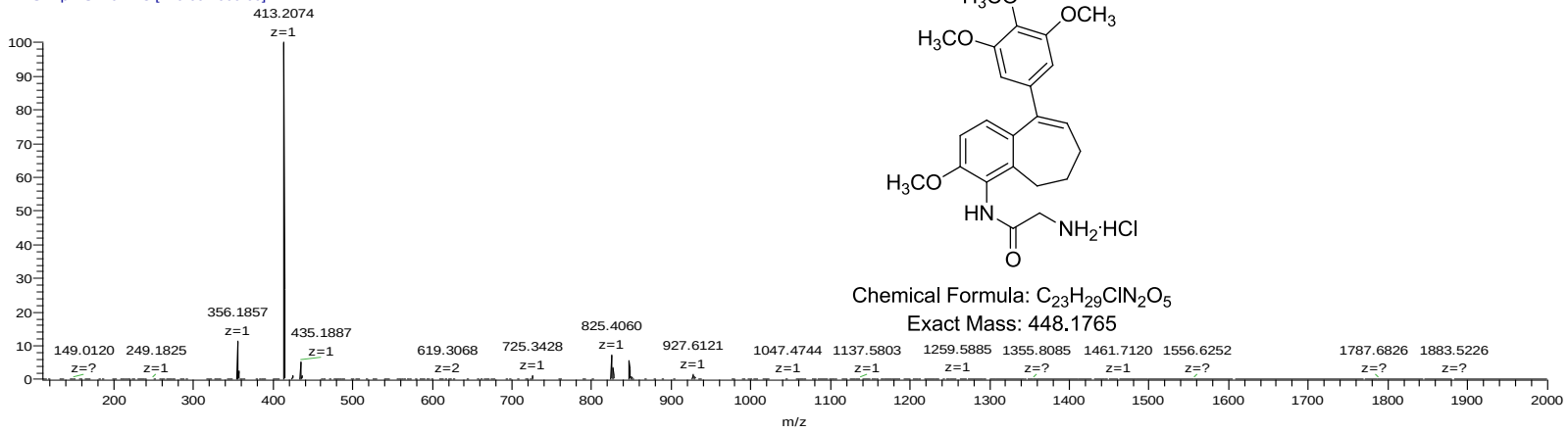
^{13}C NMR (CD_3OD, 125 MHz) Compound **45**



HRMS Compound **45**

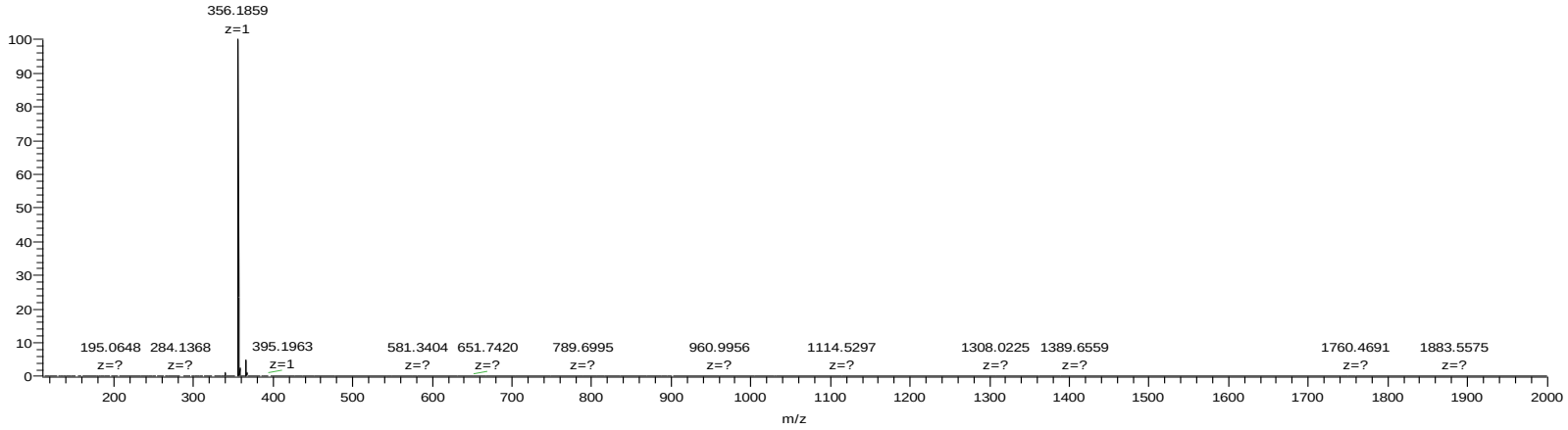
C:\Xcalibur\...\CML_II_097_ORBI_+ESI 2/13/2013 5:40:17 PM CML_II_097

CML_II_097_ORBI_+ESI #20 RT: 0.16 AV: 1 NL
T: FTMS + p ESI Full ms [110.00-2000.00]



Chemical Formula: $C_{23}H_{29}ClN_2O_5$
Exact Mass: 448.1765

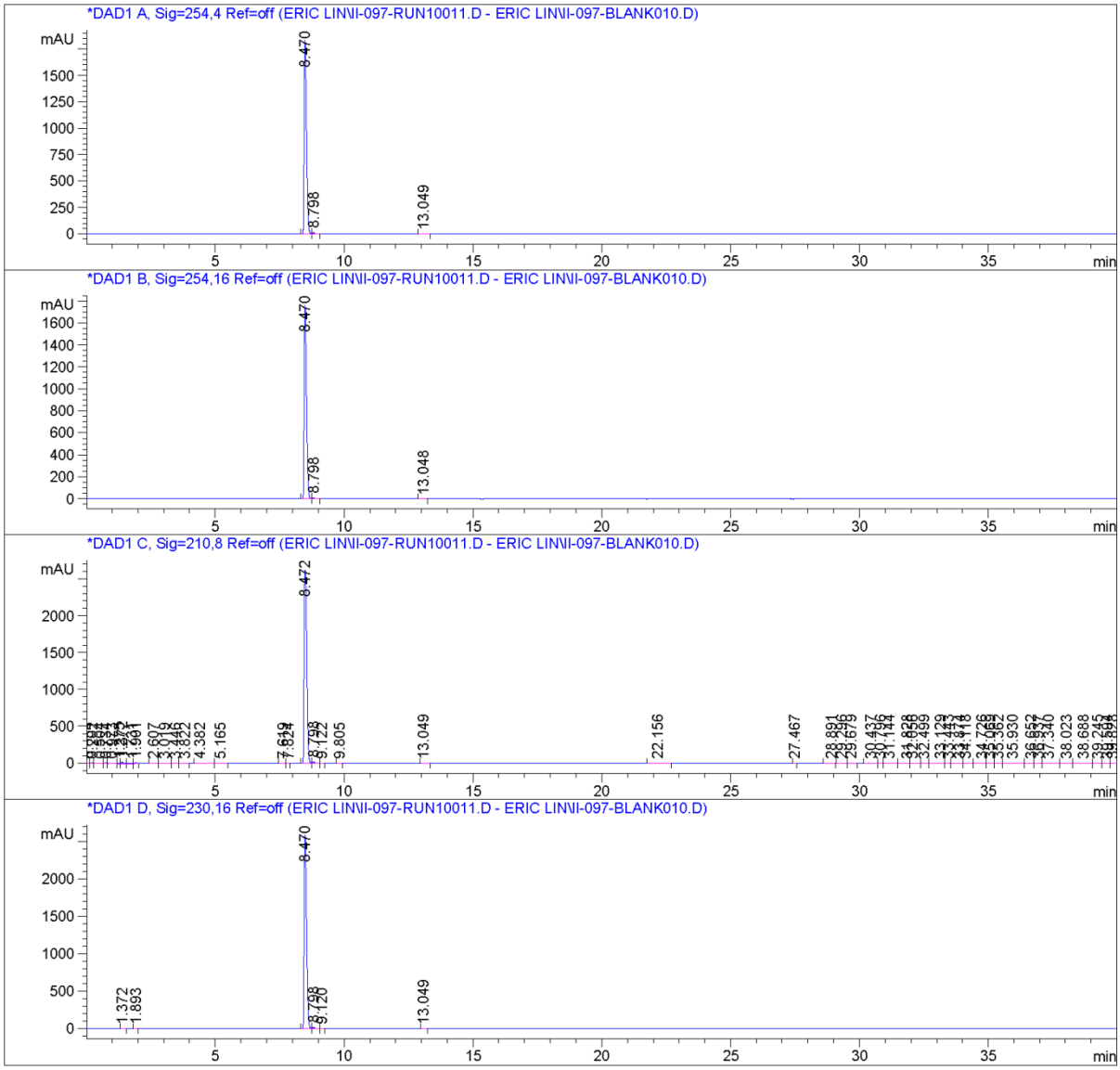
cml_ii_097_fs_orbi_+esi #14 RT: 0.16 AV: 1 NL: 3.56E8
T: FTMS + p ESI Full ms2 413.20@cid35.00 [110.00-2000.00]



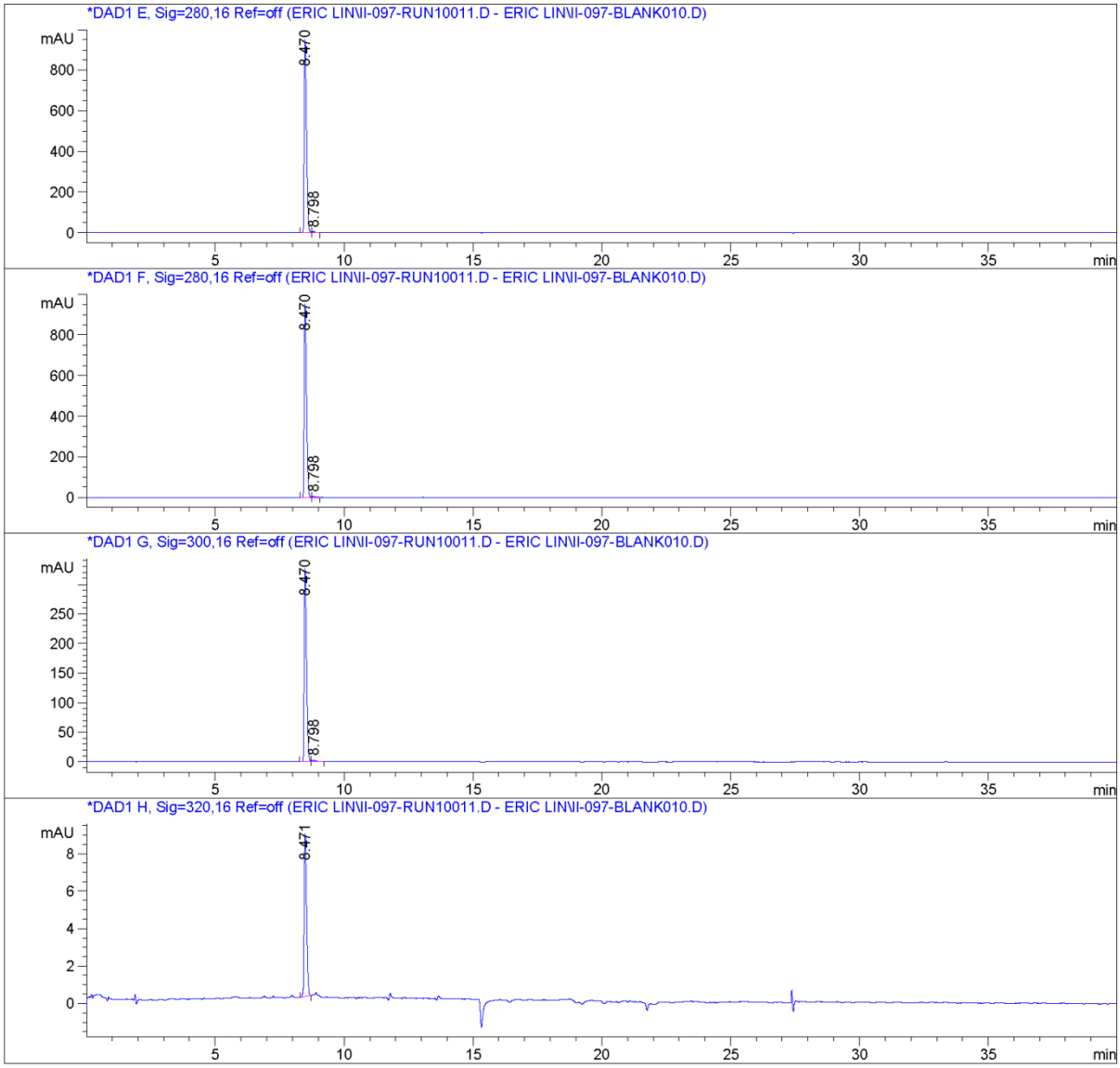
HPLC for Compound **45**

Data File C:\CHEM32\1\DATA\ERIC LIN\II-097-RUN10011.D
Sample Name: II-097-run1

```
======================================================================
Acq. Operator   : Eric Lin
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 2/13/2013 6:00:26 PM
Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed    : 2/13/2013 5:46:30 PM by Eric Lin
Analysis Method : C:\CHEM32\1\DATA\ERIC LIN\II-097-RUN10011.D\DA.M (MASTERMETHOD.M)
Last changed    : 2/13/2013 6:53:22 PM by Eric Lin
```



Data File C:\CHEM32\1\DATA\ERIC LIN\II-097-RUN10011.D
Sample Name: II-097-run1



==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Data File C:\CHEM32\1\DATA\ERIC LIN\II-097-RUN10011.D
Sample Name: II-097-run1

Signal 1: DAD1 A, Sig=254,4 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.470	BV	0.1008	1.17827e4	1837.96814	99.4176
2	8.798	VV	0.0826	62.69828	10.97642	0.5290
3	13.049	BB	0.0837	6.32477	1.12330	0.0534
Totals :				1.18517e4	1850.06786	

Signal 2: DAD1 B, Sig=254,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.470	BV	0.1007	1.13146e4	1766.50464	99.4144
2	8.798	VV	0.0832	60.84603	10.56088	0.5346
3	13.048	BB	0.0822	5.79796	1.05424	0.0509
Totals :				1.13813e4	1778.11976	

Signal 3: DAD1 C, Sig=210,8 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	0.054	BB	0.0750	11.16905	2.55559	0.0520
2	0.202	BV	0.0817	55.52967	9.57559	0.2587
3	0.504	VV	0.2634	105.27843	5.21736	0.4904
4	0.734	VB	0.0770	51.02904	9.44696	0.2377
5	0.973	BV	0.2570	99.27760	5.93896	0.4625
6	1.275	VV	0.1060	41.00343	5.56546	0.1910
7	1.372	VV	0.0851	147.69992	23.58000	0.6880
8	1.731	VB	0.1449	112.58514	9.88511	0.5245
9	1.901	BB	0.0702	78.50723	16.88350	0.3657
10	2.607	BB	0.1757	41.11032	3.81595	0.1915
11	3.019	BB	0.1998	64.92876	4.98890	0.3025
12	3.446	BV	0.1491	23.71148	2.63236	0.1105
13	3.822	VB	0.1872	15.85303	1.19137	0.0738
14	4.382	BB	0.3332	51.56498	2.16998	0.2402
15	5.165	BB	0.2393	32.31220	2.03490	0.1505
16	7.619	BV	0.1153	11.13670	1.30909	0.0519
17	7.824	VB	0.0996	6.59859	1.04646	0.0307
18	8.472	BV	0.1199	1.92972e4	2621.11572	89.8915

Data File C:\CHEM32\1\DATA\ERIC LIN\II-097-RUN10011.D
Sample Name: II-097-run1

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
19	8.798	VV	0.0861	131.16293	21.82805	0.6110
20	9.122	VB	0.0850	15.38597	2.60056	0.0717
21	9.805	BV	0.1435	9.15189	1.01217	0.0426
22	13.049	BB	0.0798	12.42094	2.34633	0.0579
23	22.156	BB	0.3403	45.85422	1.72430	0.2136
24	27.467	BB	0.0955	14.02170	2.35320	0.0653
25	28.891	BB	0.2091	30.57117	2.32981	0.1424
26	29.296	BV	0.2434	50.76952	3.16065	0.2365
27	29.679	VB	0.1904	36.37481	2.71358	0.1694
28	30.437	BV	0.3088	49.40651	2.06713	0.2301
29	30.796	VB	0.1172	7.50973	1.00419	0.0350
30	31.144	BB	0.2749	30.46164	1.78690	0.1419
31	31.828	BV	0.2434	49.71067	2.88011	0.2316
32	32.056	VV	0.2347	56.01570	3.28860	0.2609
33	32.499	VV	0.1936	24.38632	1.85169	0.1136
34	33.129	VB	0.2906	45.53355	2.05459	0.2121
35	33.443	BV	0.1474	19.68425	2.09988	0.0917
36	33.874	VV	0.2797	65.24351	3.12388	0.3039
37	34.118	VB	0.1626	20.04897	1.76423	0.0934
38	34.726	BV	0.2280	25.88588	1.64124	0.1206
39	35.069	VV	0.1740	25.54245	2.29200	0.1190
40	35.362	VB	0.1951	16.67540	1.17863	0.0777
41	35.930	BB	0.3034	46.04016	1.97945	0.2145
42	36.652	BV	0.2354	27.88365	1.92074	0.1299
43	36.937	VB	0.1720	32.49429	2.82890	0.1514
44	37.340	BB	0.3804	86.93623	3.22579	0.4050
45	38.023	BV	0.2396	65.75677	4.04595	0.3063
46	38.688	VB	0.3647	75.06680	2.63271	0.3497
47	39.245	BV	0.2034	36.32860	2.83767	0.1692
48	39.594	VV	0.2061	39.79980	2.86473	0.1854
49	39.828	VBA	0.1623	28.59319	2.77197	0.1332
Totals :				2.14672e4	2821.16288	

Signal 4: DAD1 D, Sig=230,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.372	BB	0.0580	10.34708	2.62183	0.0596
2	1.893	BB	0.0638	10.50224	2.56492	0.0605
3	8.470	BV	0.1075	1.72373e4	2593.79321	99.2714
4	8.798	VV	0.0849	92.27396	15.63470	0.5314
5	9.120	VB	0.0763	7.66288	1.48331	0.0441
6	13.049	BB	0.0763	5.72853	1.14636	0.0330
Totals :				1.73638e4	2617.24433	

Data File C:\CHEM32\1\DATA\ERIC LIN\II-097-RUN10011.D
Sample Name: II-097-run1

Signal 5: DAD1 E, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.470	BV	0.1006	6110.43115	955.03564	99.2556
2	8.798	VV	0.0974	45.82551	6.57189	0.7444
Totals :				6156.25666	961.60754	

Signal 6: DAD1 F, Sig=280,16 Ref=off
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.470	BV	0.1006	6110.43115	955.03564	99.2556
2	8.798	VV	0.0974	45.82551	6.57189	0.7444
Totals :				6156.25666	961.60754	

Signal 7: DAD1 G, Sig=300,16 Ref=off
Signal has been modified after loading from rawdata file!

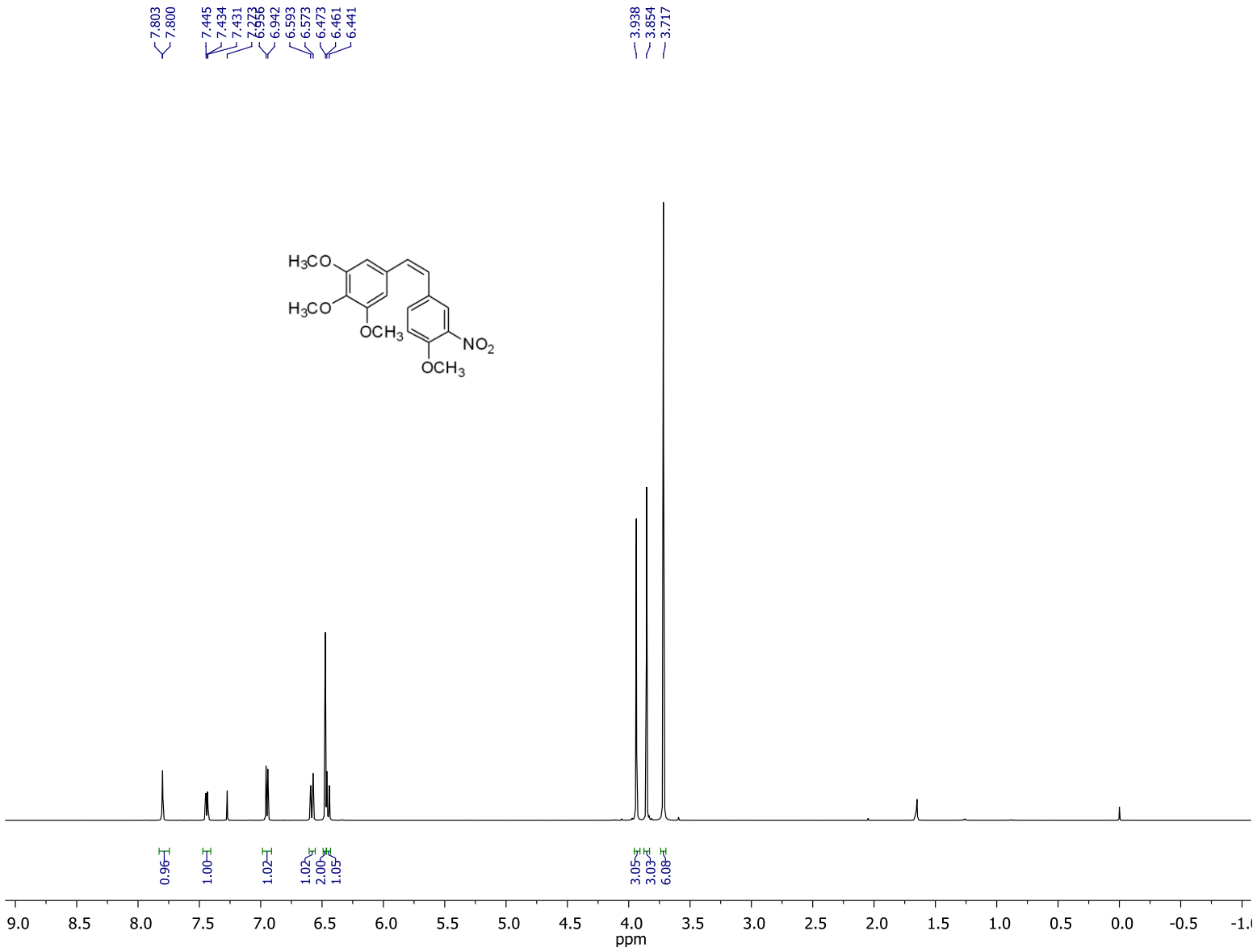
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.470	BV	0.1009	2099.83813	326.97824	98.7479
2	8.798	VB	0.1206	26.62493	2.97049	1.2521
Totals :				2126.46306	329.94873	

Signal 8: DAD1 H, Sig=320,16 Ref=off
Signal has been modified after loading from rawdata file!

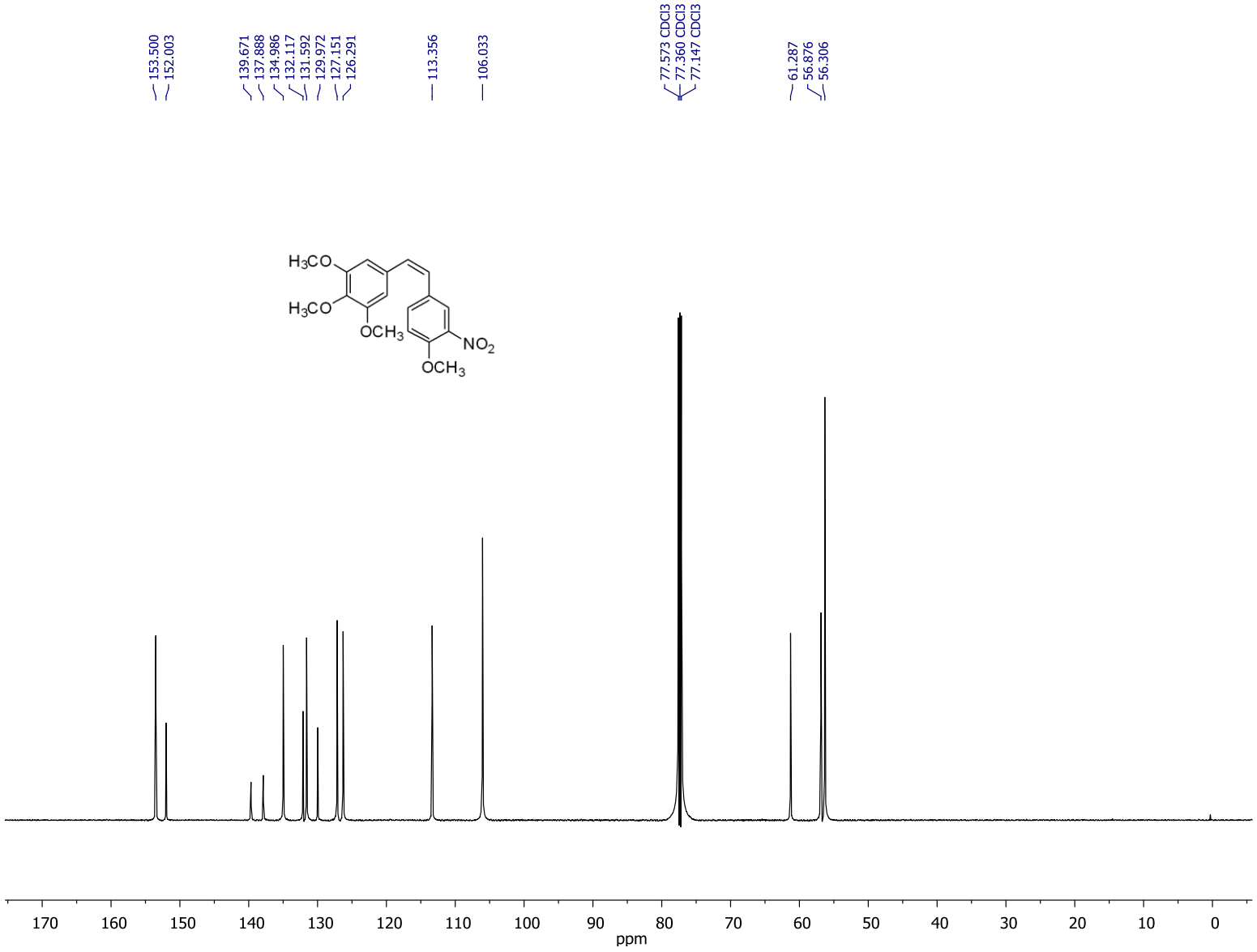
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.471	BB	0.1060	58.33273	8.73021	100.0000
Totals :				58.33273	8.73021	

==========

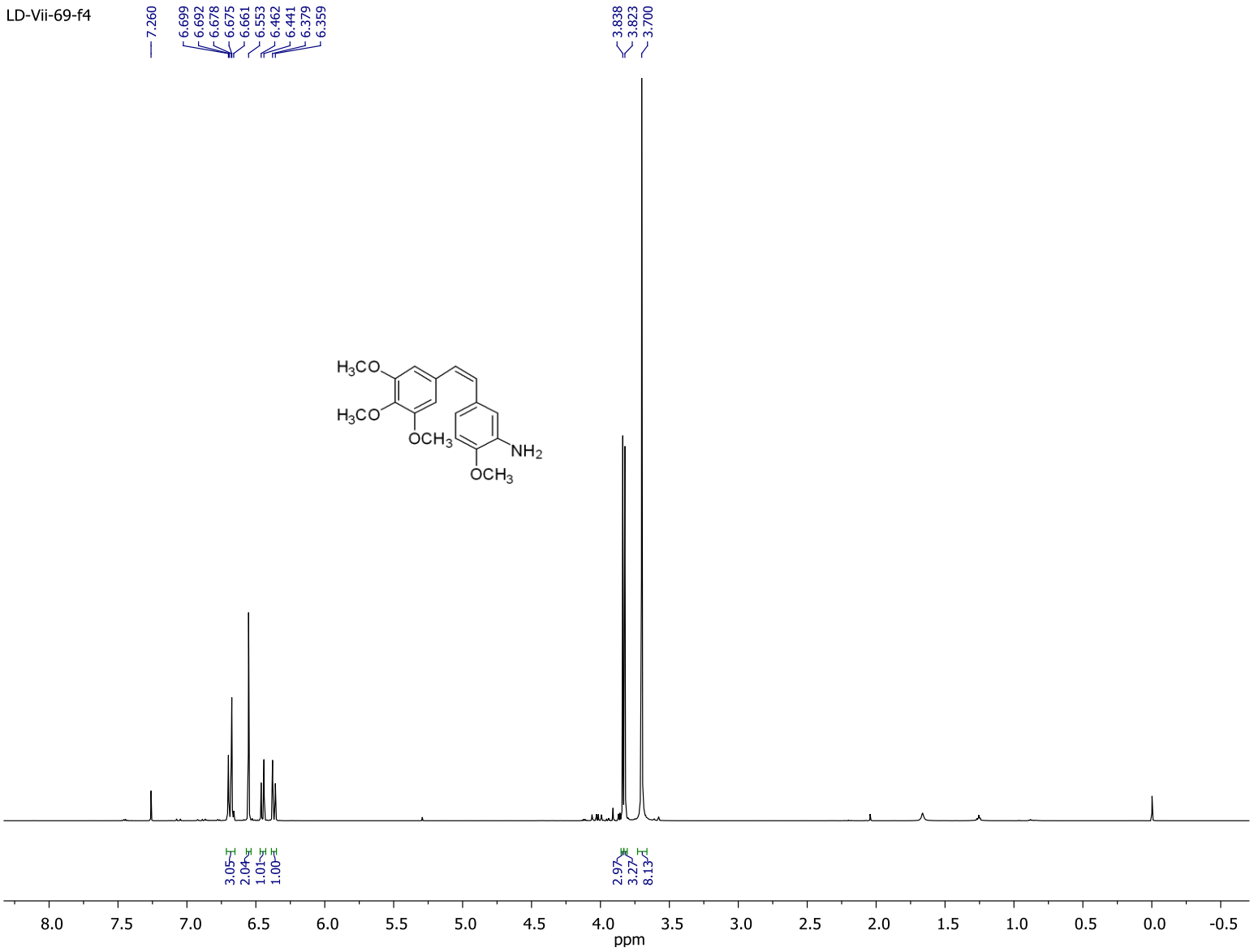
^{1}H NMR ($CDCl_3$, 600 MHz) Compound **46**



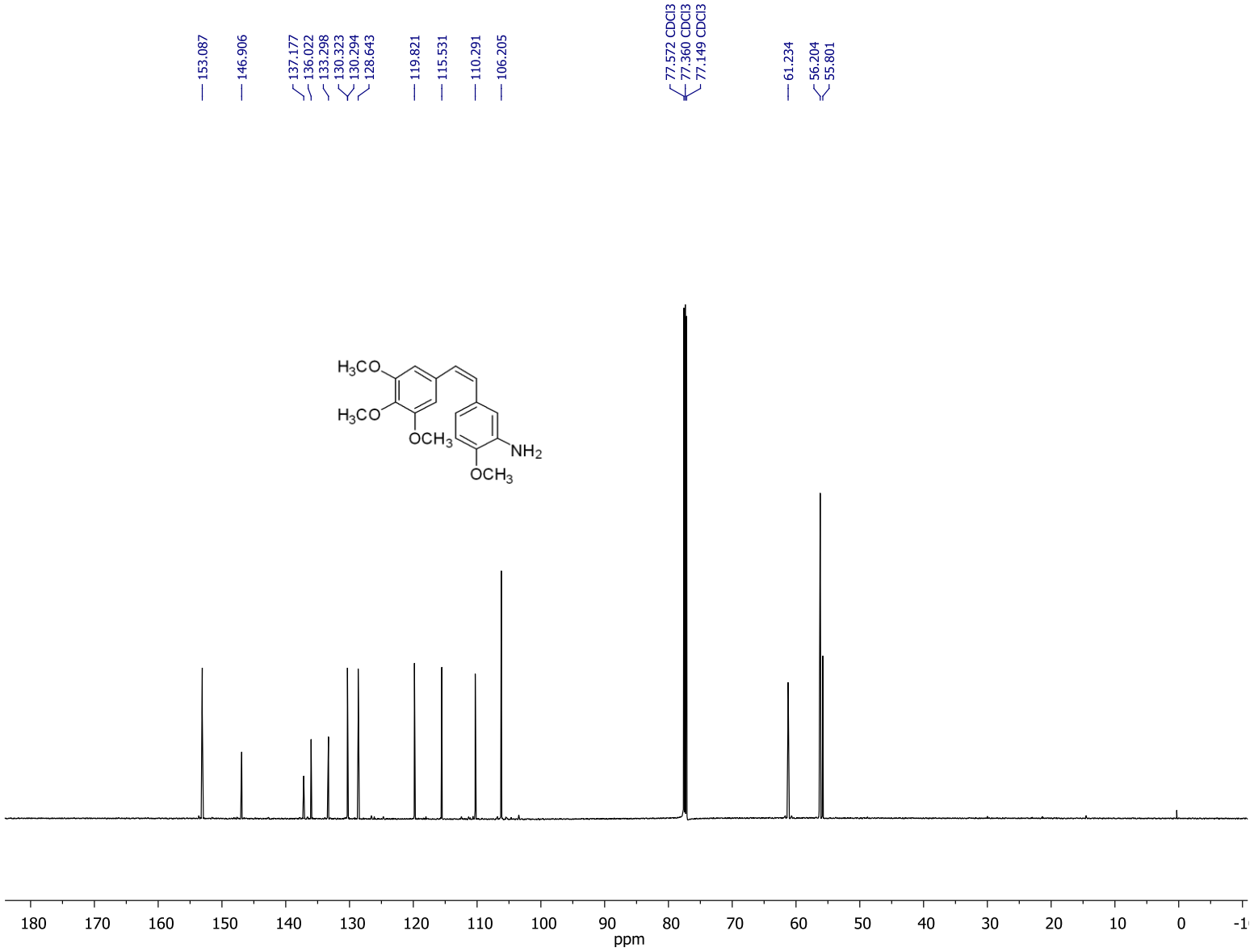
^{13}C NMR ($CDCl_3$, 150 MHz) Compound **46**



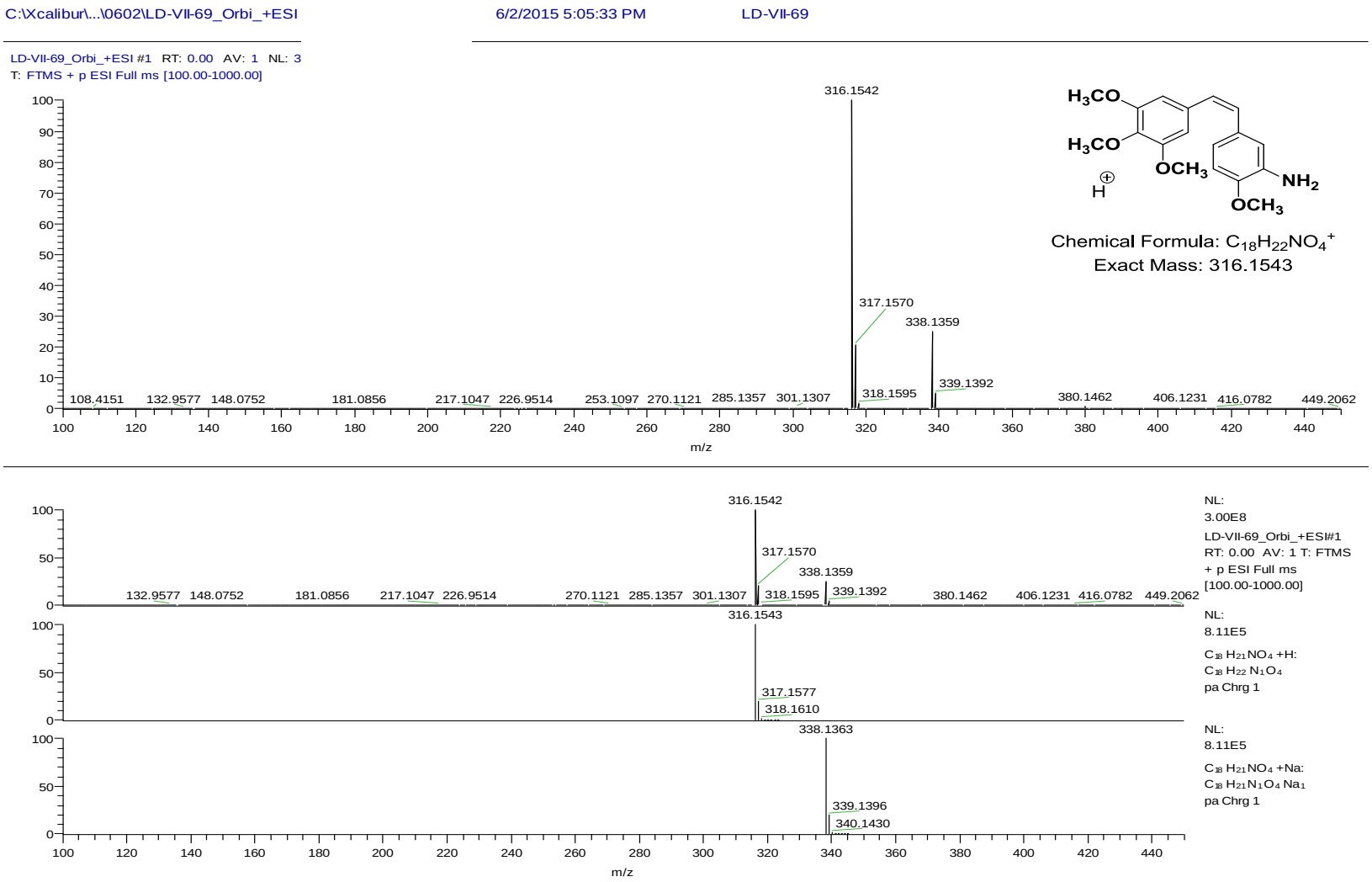
^{1}H NMR ($CDCl_3$, 600 MHz) for 3' CA4-amine



^{13}C NMR ($CDCl_3$, 150 MHz) for 3' CA4-amine



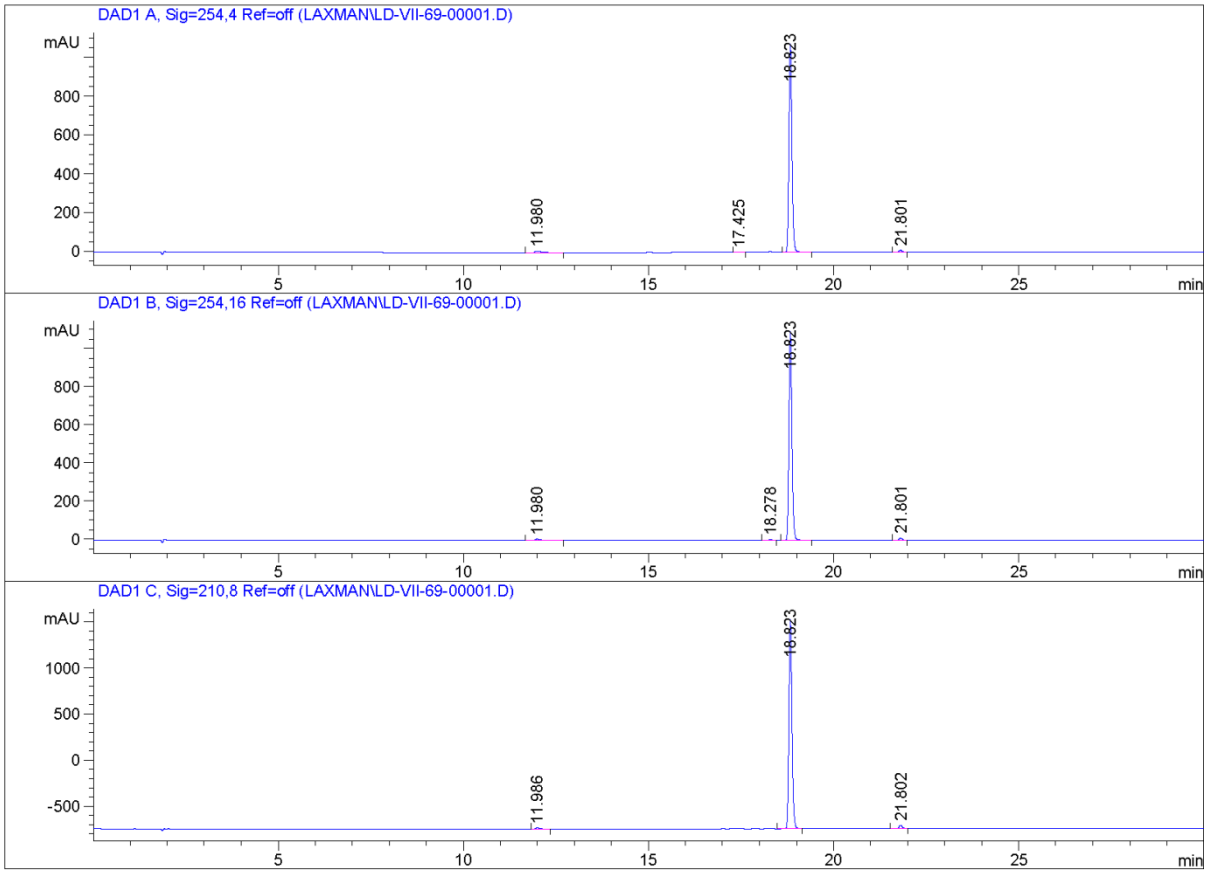
HRMS for 3' CA4-amine



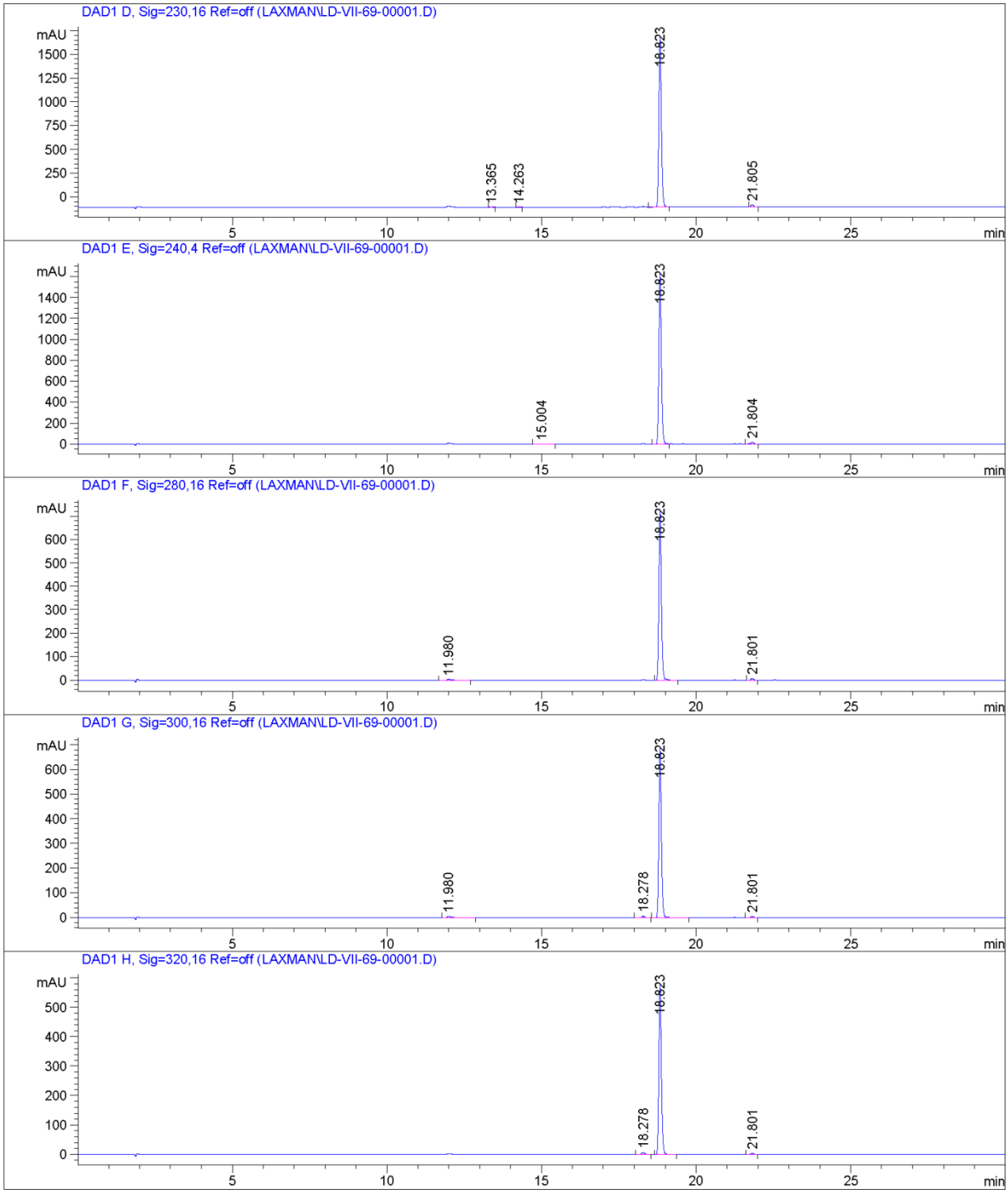
HPLC for 3' CA4-amine

Data File C:\CHEM32\1\DATA\LAXMAN\LD-VII-69-00001.D
Sample Name: LD-VII-69-1 3'CA4-Amine

```
==========================================================================
Acq. Operator   : Laxman
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 6/1/2015 2:33:28 PM
Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD2.M
Last changed    : 6/1/2015 2:13:25 PM by Eric Lin
Analysis Method : C:\CHEM32\1\DATA\LAXMAN\LD-VII-69-00001.D\DA.M (MASTERMETHOD2.M)
Last changed    : 6/1/2015 3:22:30 PM by Eric Lin
Sample Info     : Method-Mastermethod2
```



Data File C:\CHEM32\1\DATA\LAXMAN\LD-VII-69-00001.D
Sample Name: LD-VII-69-1 3'CA4-Amine



Data File C:\CHEM32\1\DATA\LAXMAN\LD-VII-69-00001.D
Sample Name: LD-VII-69-1 3'CA4-Amine

==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.980	BB	0.1751	89.59236	7.62506	1.4503
2	17.425	BB	0.0961	10.77919	1.65243	0.1745
3	18.823	BB	0.0851	6008.21289	1077.08765	97.2588
4	21.801	BB	0.0943	68.96559	11.13570	1.1164
Totals :				6177.55003	1097.50084	

Signal 2: DAD1 B, Sig=254,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.980	BB	0.1756	89.20059	7.56309	1.4138
2	18.278	BB	0.0868	26.60785	4.64551	0.4217
3	18.823	BB	0.0851	6123.01221	1097.30920	97.0493
4	21.801	BB	0.0947	70.35337	11.29626	1.1151
Totals :				6309.17402	1120.81407	

Signal 3: DAD1 C, Sig=210,8 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.986	BB	0.1510	134.73405	13.20541	1.0140
2	18.823	BV	0.0883	1.29139e4	2273.72705	97.1937
3	21.802	VB	0.0974	238.12749	36.85693	1.7922
Totals :				1.32868e4	2323.78940	

Data File C:\CHEM32\1\DATA\LAXMAN\LD-VII-69-00001.D
Sample Name: LD-VII-69-1 3'CA4-Amine

Signal 4: DAD1 D, Sig=230,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.365	BB	0.0656	9.52854	2.24285	0.0928
2	14.263	BB	0.0820	5.22144	1.01602	0.0508
3	18.823	BV	0.0872	1.01168e4	1810.49146	98.5021
4	21.805	VB	0.0987	139.09056	21.72524	1.3543
Totals :				1.02707e4	1835.47557	

Signal 5: DAD1 E, Sig=240,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.004	BB	0.1330	14.18810	1.51891	0.1524
2	18.823	BV	0.0870	9179.72461	1646.90674	98.5840
3	21.804	VB	0.1042	117.66814	17.11329	1.2637
Totals :				9311.58084	1665.53893	

Signal 6: DAD1 F, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.980	BB	0.1822	64.04824	4.97383	1.5266
2	18.823	BB	0.0852	4088.56470	731.74805	97.4517
3	21.801	BB	0.0907	42.86423	7.27667	1.0217
Totals :				4195.47717	743.99854	

Signal 7: DAD1 G, Sig=300,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.980	BB	0.1797	62.46052	5.14319	1.5541
2	18.278	BB	0.0856	34.96275	6.22234	0.8699
3	18.823	BB	0.0852	3891.12744	695.78998	96.8142
4	21.801	BB	0.0907	30.62026	5.19646	0.7619
Totals :				4019.17097	712.35197	

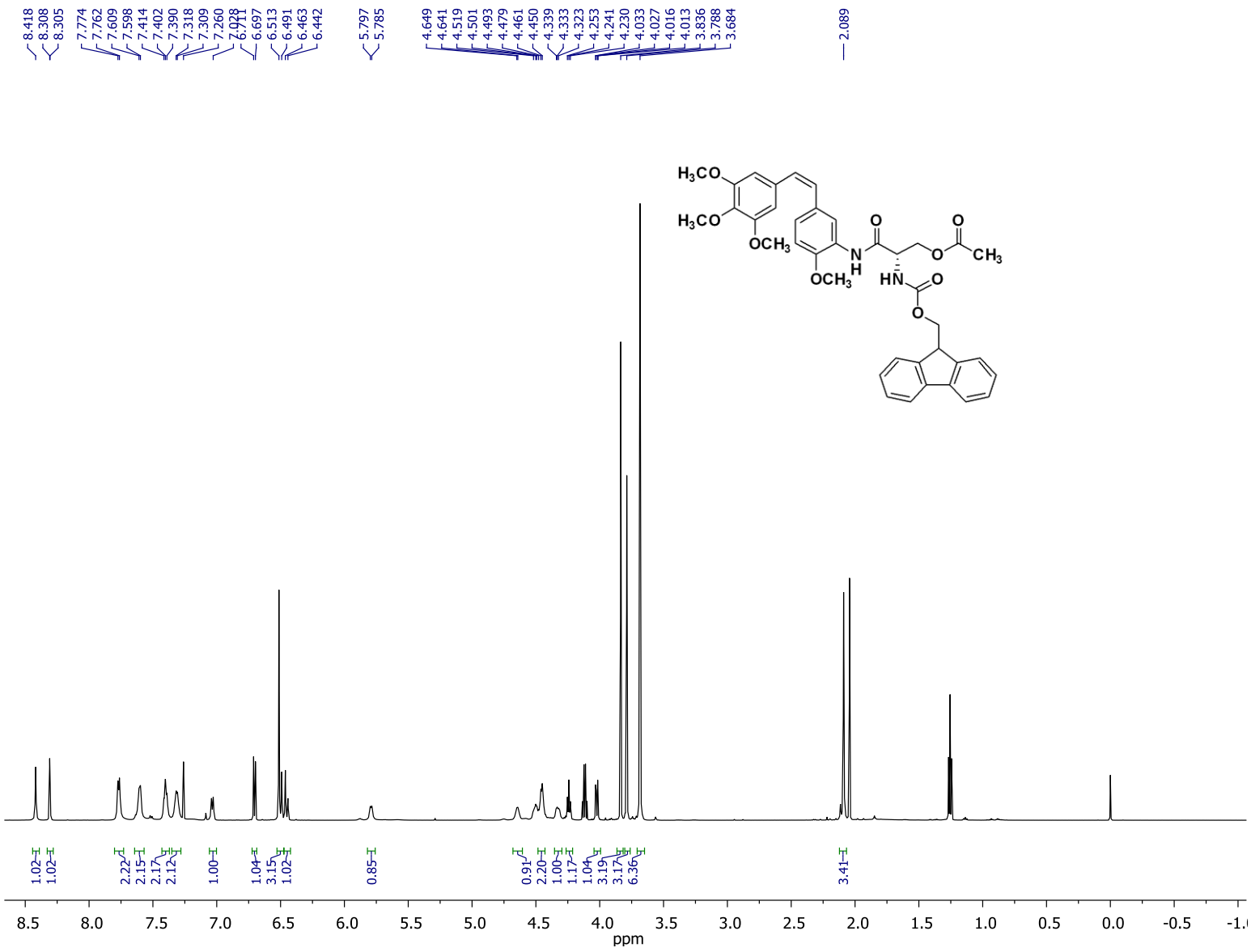
Data File C:\CHEM32\1\DATA\LAXMAN\LD-VII-69-00001.D
Sample Name: LD-VII-69-1 3'CA4-Amine

Signal 8: DAD1 H, Sig=320,16 Ref=off

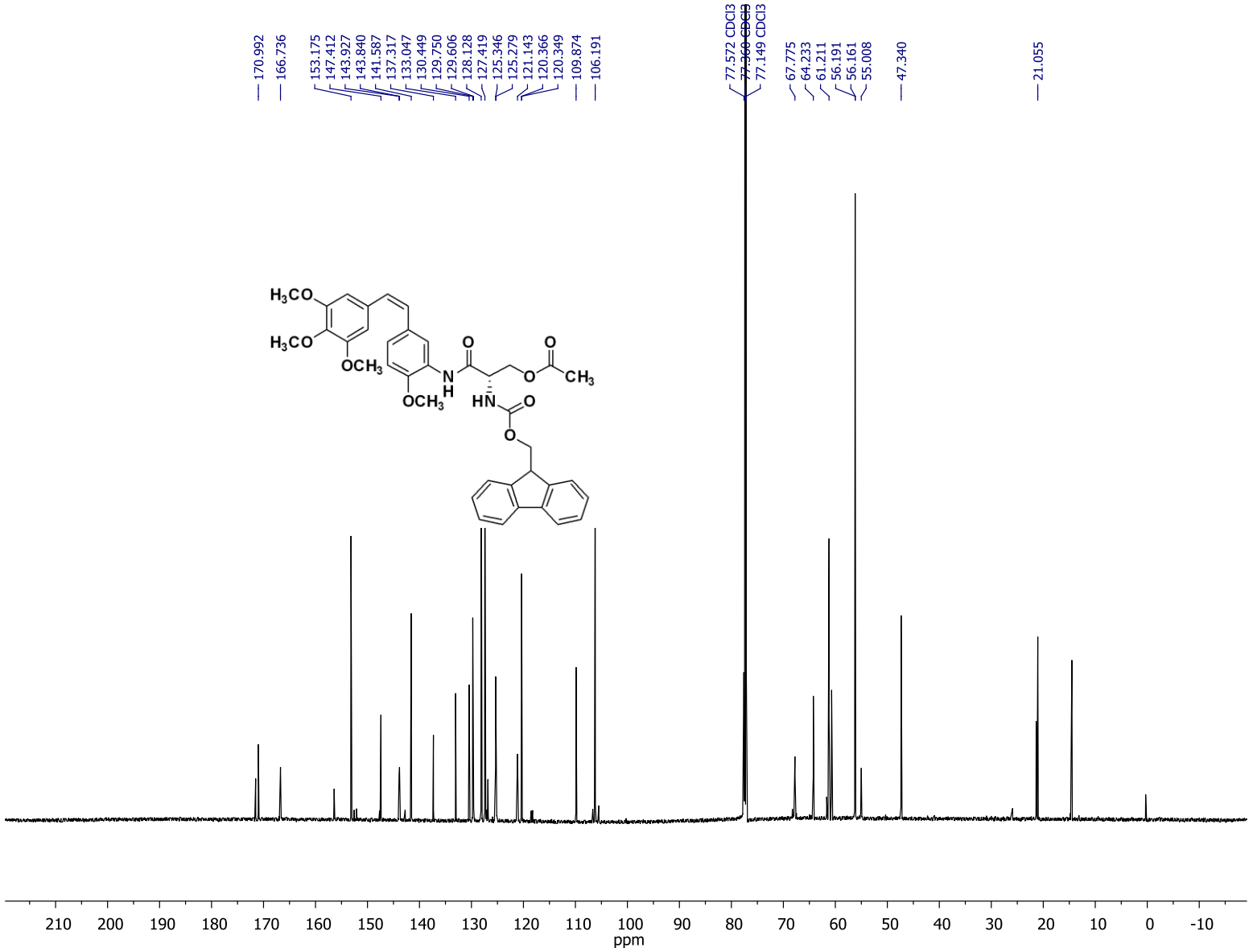
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.278	BB	0.0849	42.04350	7.55673	1.2647
2	18.823	BB	0.0851	3256.91406	583.29913	97.9669
3	21.801	BB	0.0909	25.54603	4.32569	0.7684
Totals :				3324.50358	595.18155	

*** End of Report ***

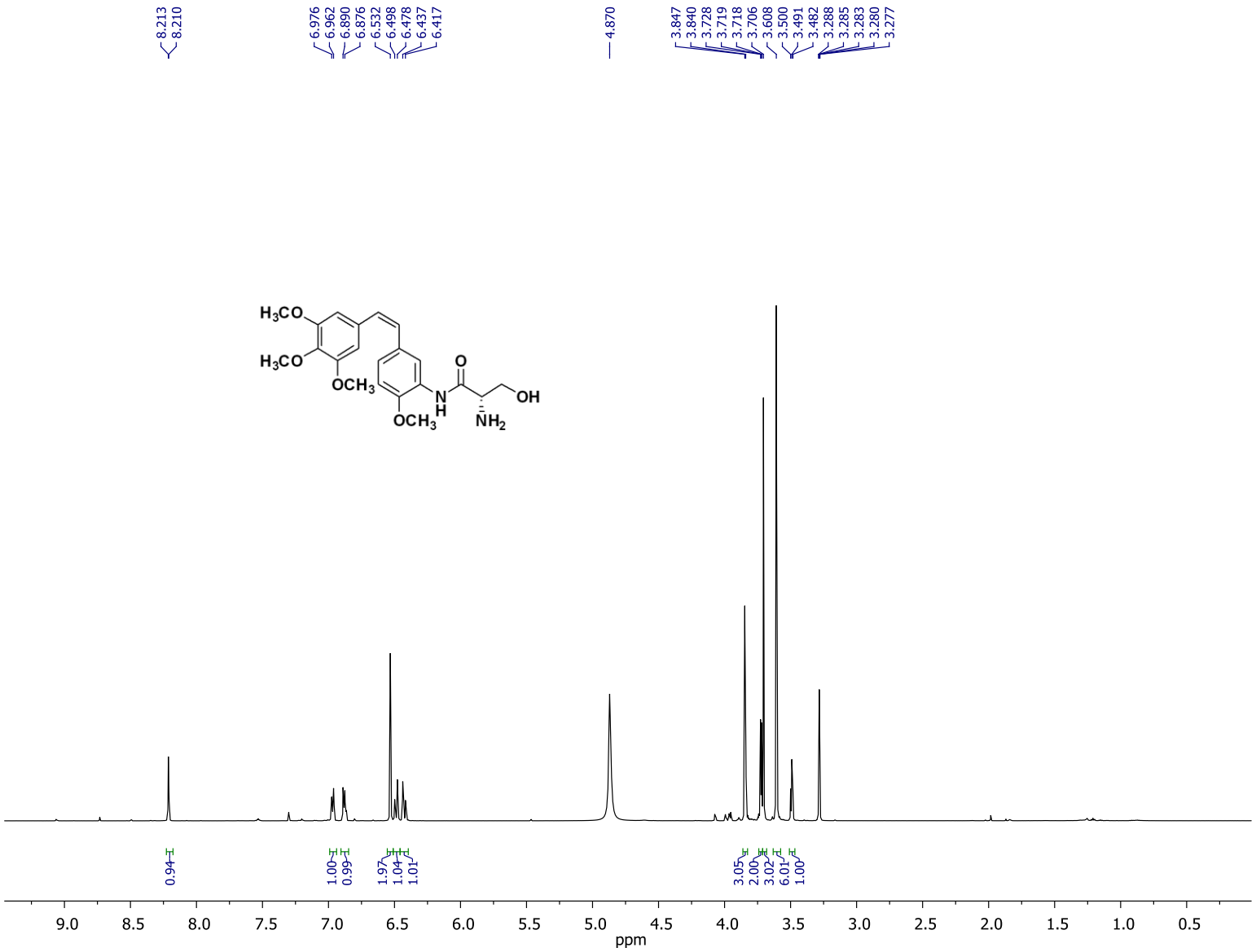
^{1}H NMR ($CDCl_3$, 600 MHz) Compound **47**



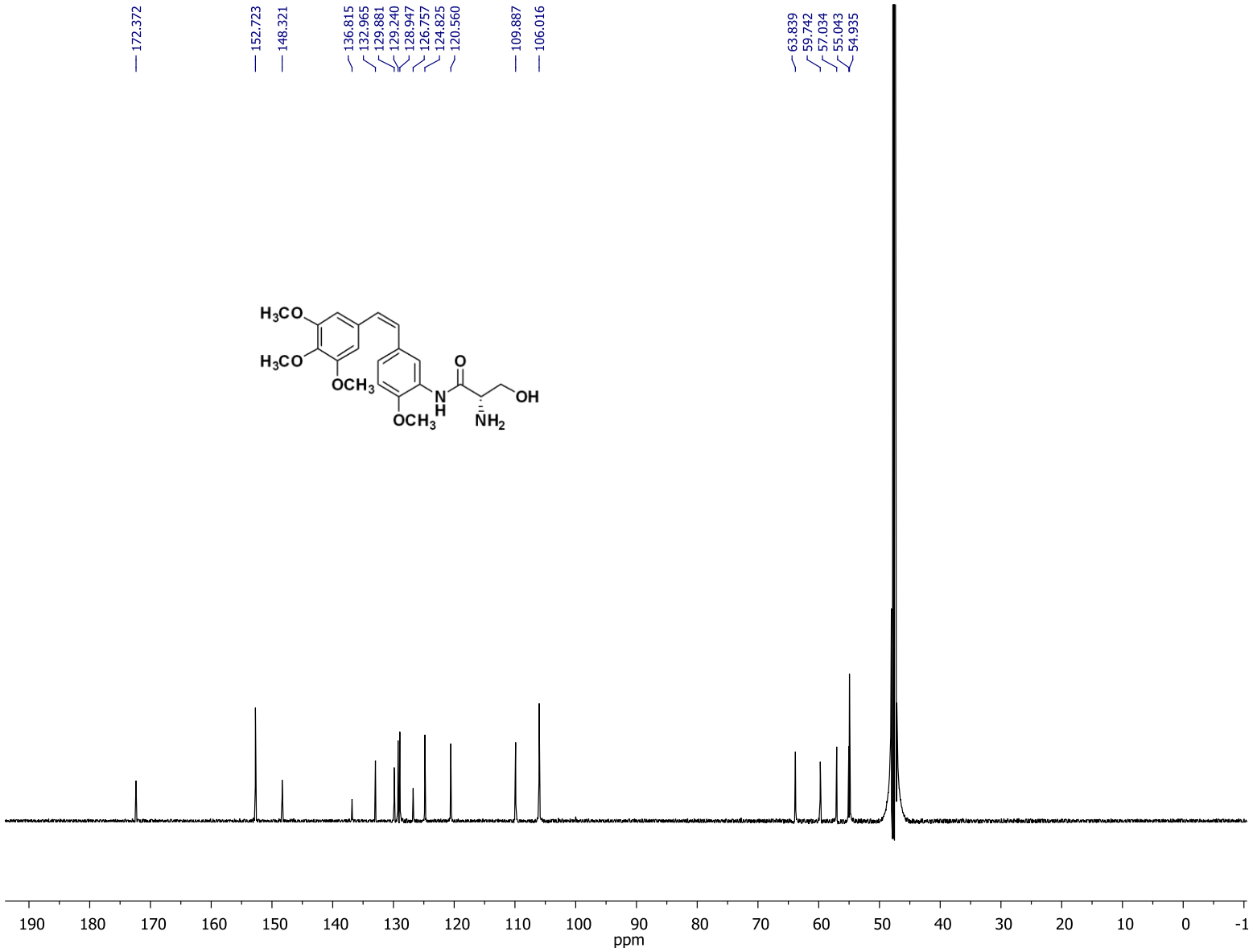
^{13}C NMR ($CDCl_3$, 150 MHz) Compound **47**



^{1}H NMR (CD_3OD, 600 MHz) for 3' CA4-*L*-serinamide



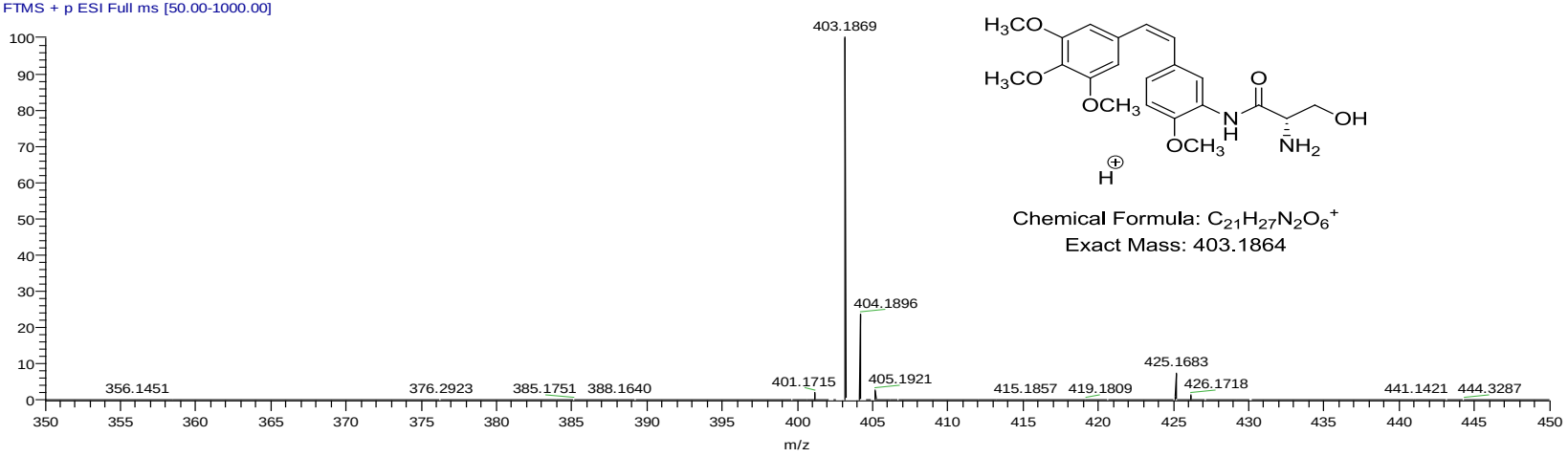
^{13}C NMR (CD_3OD, 150 MHz) for 3' CA4-*L*-serinamide



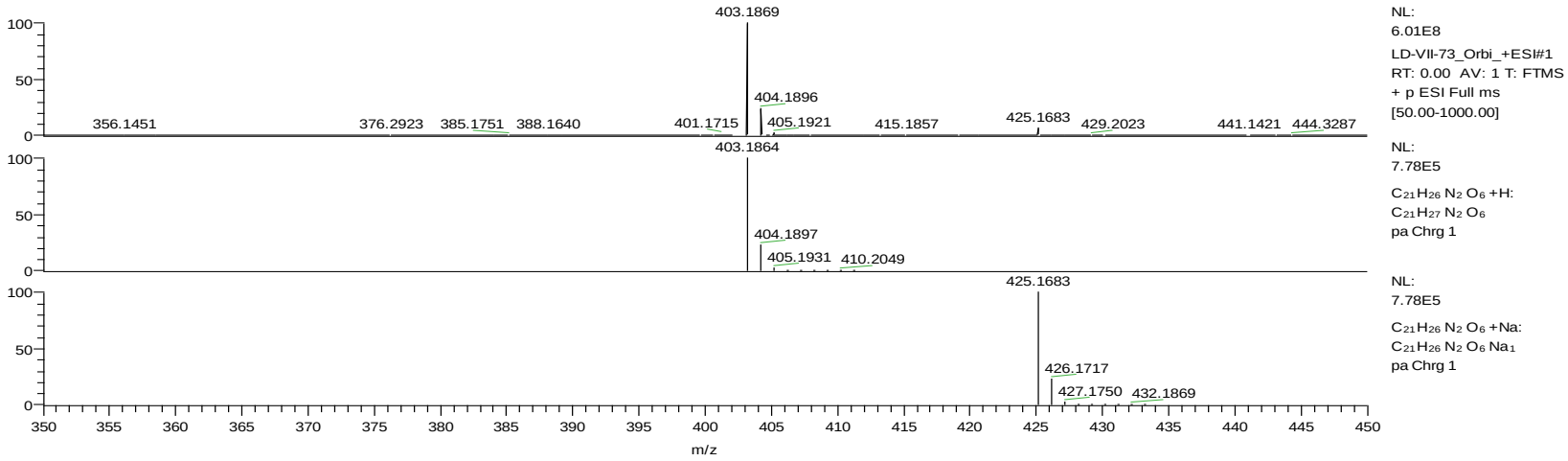
HRMS for 3' CA4-*L*-serinamide

C:\Xcalibur\...\0602\LD-VII-73_Orbi_+ESI 6/2/2015 5:10:31 PM LD-VII-73

LD-VII-73_Orbi_+ESI #1 RT: 0.00 AV: 1 NL: 6
T: FTMS + p ESI Full ms [50.00-1000.00]



Chemical Formula: $C_{21}H_{27}N_2O_6^+$
Exact Mass: 403.1864



NL:
6.01E8
LD-VII-73_Orbi_+ESI#1
RT: 0.00 AV: 1 T: FTMS
+ p ESI Full ms
[50.00-1000.00]

NL:
7.78E5
$C_{21}H_{26}N_2O_6$ +H:
$C_{21}H_{27}N_2O_6$
pa Chrg 1

NL:
7.78E5
$C_{21}H_{26}N_2O_6$ +Na:
$C_{21}H_{26}N_2O_6Na_1$
pa Chrg 1

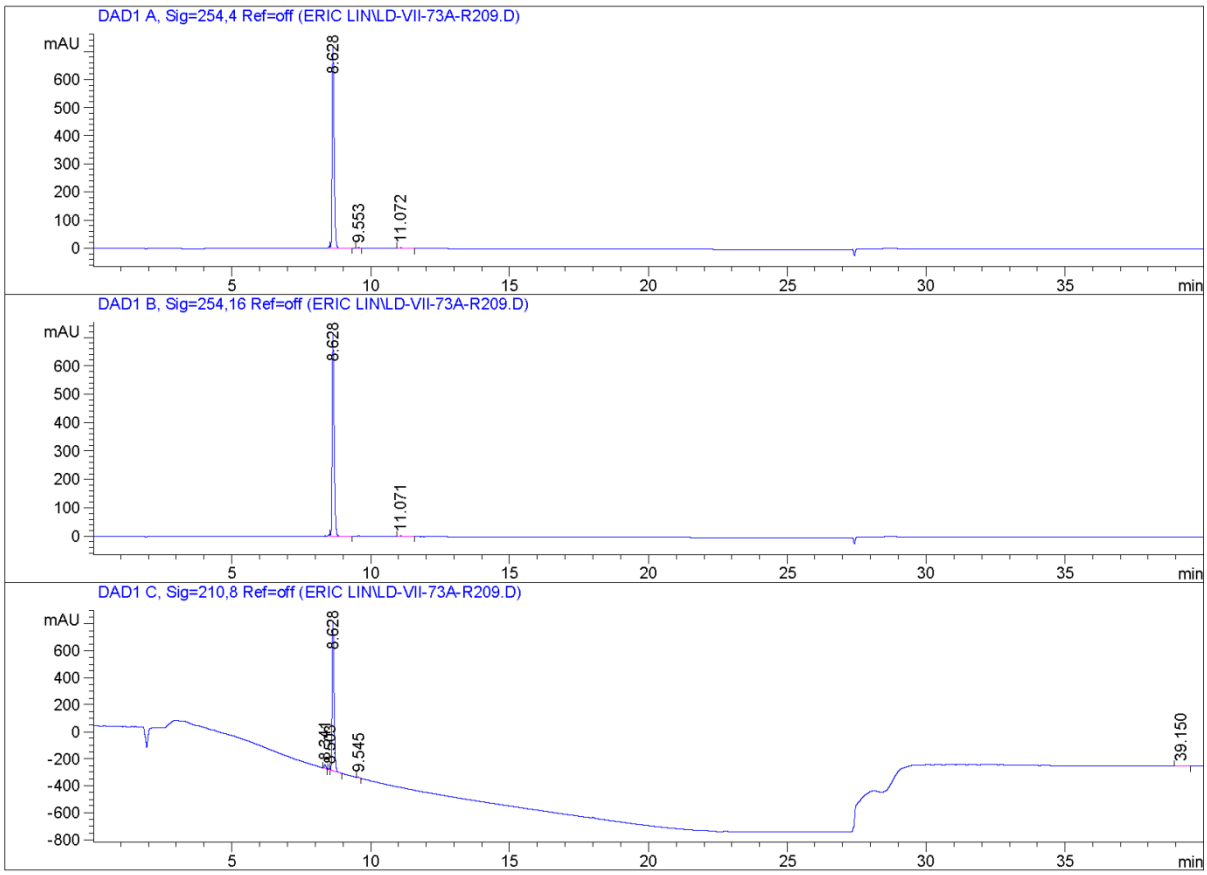
HPLC for 3' CA4-*L*-serinamide

Data File C:\CHEM32\1\DATA\ERIC LIN\LD-VII-73A-R209.D
Sample Name: LD-VII-73A-R2

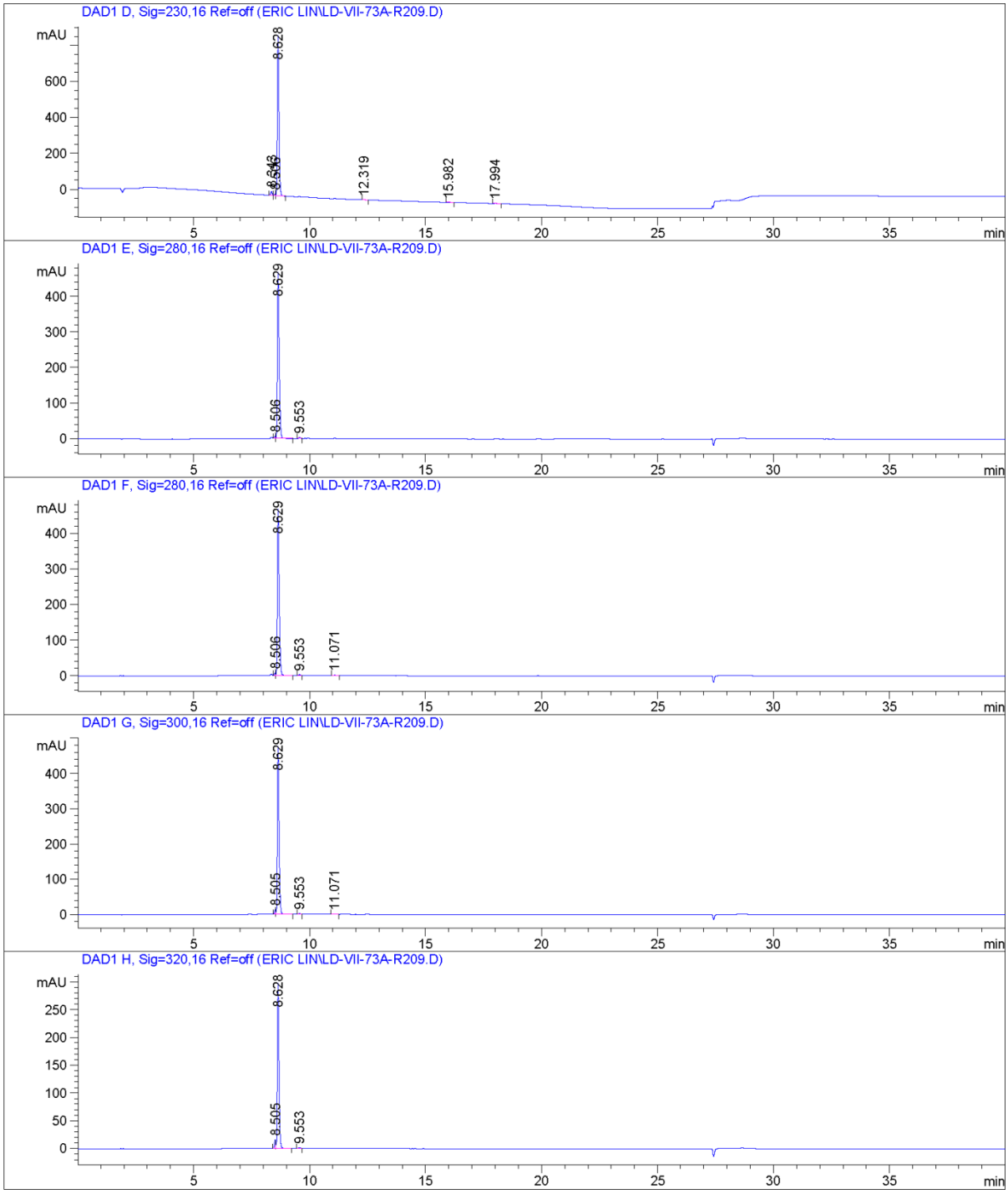
```
==========================================================================
Acq. Operator   : Eric Lin
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 6/1/2015 12:29:03 PM
Acq. Method     : C:\CHEM32\1\METHODS\MASTERMETHOD.M
Last changed    : 6/1/2015 11:52:37 AM by Eric Lin
Analysis Method : C:\CHEM32\1\DATA\ERIC LIN\LD-VII-73A-R209.D\DA.M (MASTERMETHOD.M)
Last changed    : 6/1/2015 1:19:18 PM by Eric Lin
Sample Info     : wash

                  Method:

                  0-5 min, 10:90 ACN/Water
                  5-25 min, gradient, 10:90 to 100:00 ACN/Water
                  25-30 min, 100:00 ACN/Water
```



Data File C:\CHEM32\1\DATA\ERIC LIN\LD-VII-73A-R209.D
Sample Name: LD-VII-73A-R2



Data File C:\CHEM32\1\DATA\ERIC LIN\LD-VII-73A-R209.D
Sample Name: LD-VII-73A-R2

==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.628	VB	0.0761	3615.06738	725.35986	99.2925
2	9.553	BB	0.0725	8.57860	1.83688	0.2356
3	11.072	BB	0.1303	17.18025	1.78545	0.4719
Totals :				3640.82624	728.98219	

Signal 2: DAD1 B, Sig=254,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.628	VB	0.0764	3597.92407	718.34595	99.5364
2	11.071	BB	0.1264	16.75720	1.77018	0.4636
Totals :				3614.68127	720.11613	

Signal 3: DAD1 C, Sig=210,8 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.341	BB	0.0702	125.12968	27.94563	2.2352
2	8.503	BV	0.0582	32.47391	8.98720	0.5801
3	8.628	VB	0.0744	5386.43848	1113.73535	96.2202
4	9.545	BB	0.0701	25.45053	5.69919	0.4546
5	39.150	BB	0.2871	28.53901	1.39256	0.5098
Totals :				5598.03160	1157.75993	

Data File C:\CHEM32\1\DATA\ERIC LIN\LD-VII-73A-R209.D
Sample Name: LD-VII-73A-R2

Signal 4: DAD1 D, Sig=230,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.343	BB	0.0704	97.43347	21.70703	2.1572
2	8.506	BV	0.0582	33.81914	9.34663	0.7487
3	8.628	VB	0.0748	4350.40234	893.63580	96.3168
4	12.319	VB	0.1081	10.71834	1.41883	0.2373
5	15.982	BB	0.1050	6.85785	1.01244	0.1518
6	17.994	VB	0.0977	17.53352	2.63260	0.3882
Totals :				4516.76467	929.75333	

Signal 5: DAD1 E, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.506	VV	0.0609	26.17398	6.80126	1.0126
2	8.629	VB	0.0813	2547.08740	469.12259	98.5353
3	9.553	BB	0.0732	11.68830	2.47169	0.4522
Totals :				2584.94968	478.39554	

Signal 6: DAD1 F, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.506	VV	0.0609	26.17398	6.80126	1.0099
2	8.629	VB	0.0813	2547.08740	469.12259	98.2801
3	9.553	BB	0.0732	11.68830	2.47169	0.4510
4	11.071	BB	0.0906	6.71148	1.07787	0.2590
Totals :				2591.66116	479.47341	

Signal 7: DAD1 G, Sig=300,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.505	BV	0.0613	50.99851	13.13386	2.0432
2	8.629	VB	0.0773	2427.22803	477.66873	97.2444
3	9.553	BB	0.0727	11.26238	2.40055	0.4512
4	11.071	BB	0.0867	6.51913	1.10683	0.2612

Data File C:\CHEM32\1\DATA\ERIC LIN\LD-VII-73A-R209.D
Sample Name: LD-VII-73A-R2

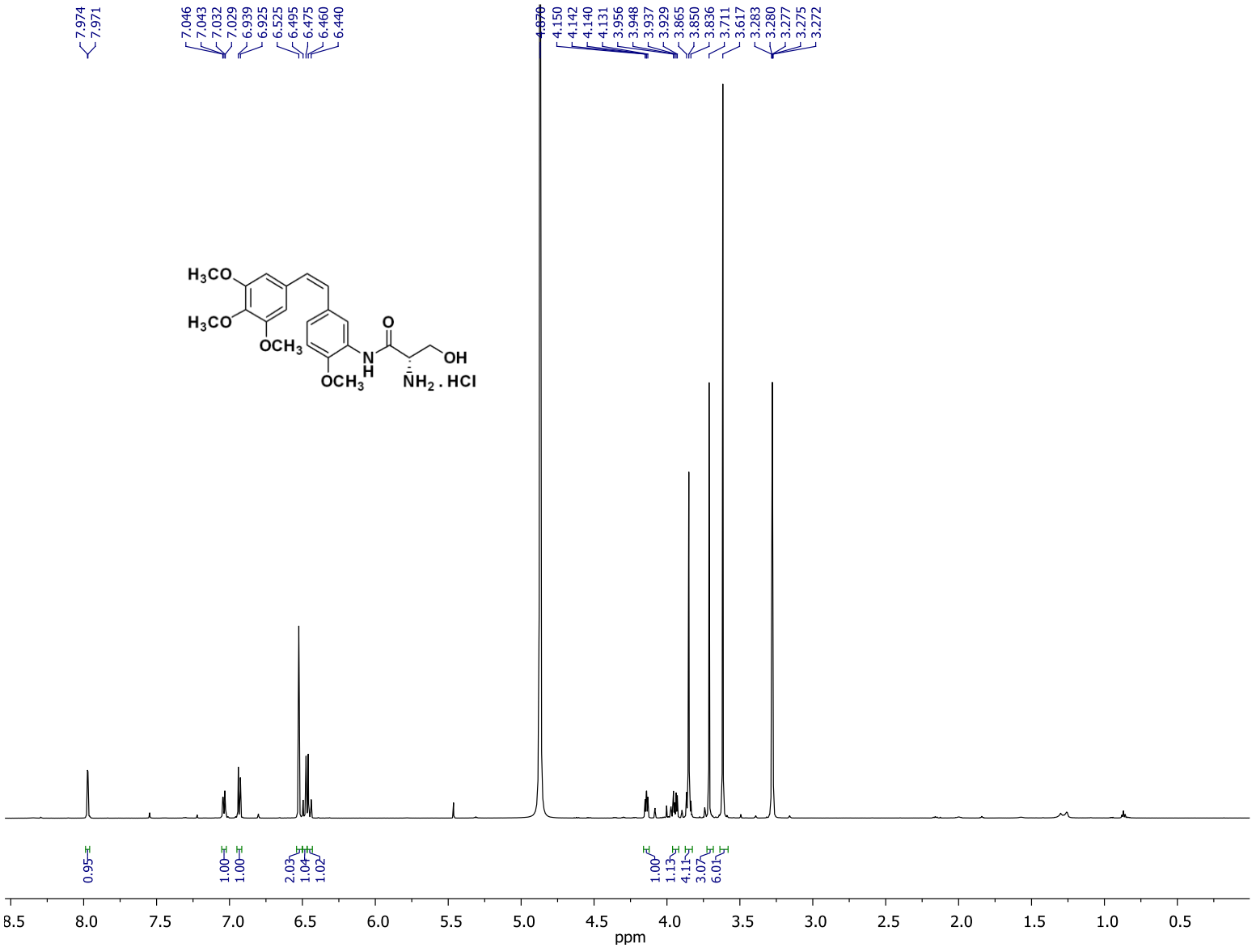
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
Totals :				2496.00805	494.30997	

Signal 8: DAD1 H, Sig=320,16 Ref=off

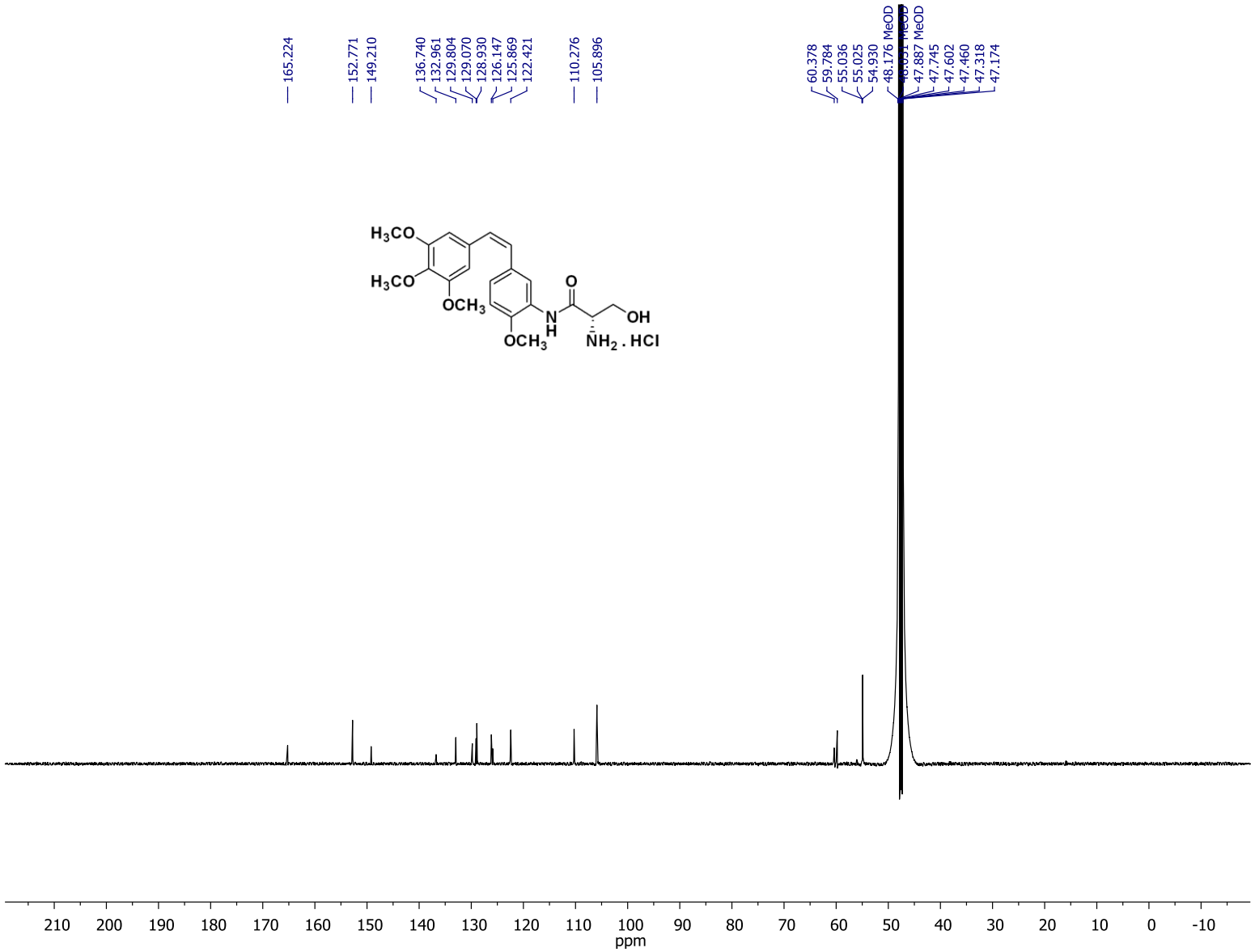
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.505	BV	0.0623	62.80982	15.83393	4.0939
2	8.628	VB	0.0754	1464.79163	297.60944	95.4747
3	9.553	BB	0.0730	6.61850	1.40323	0.4314
Totals :				1534.21994	314.84660	

*** End of Report ***

^{1}H NMR (CD_3OD, 600 MHz) for **AVE8062**



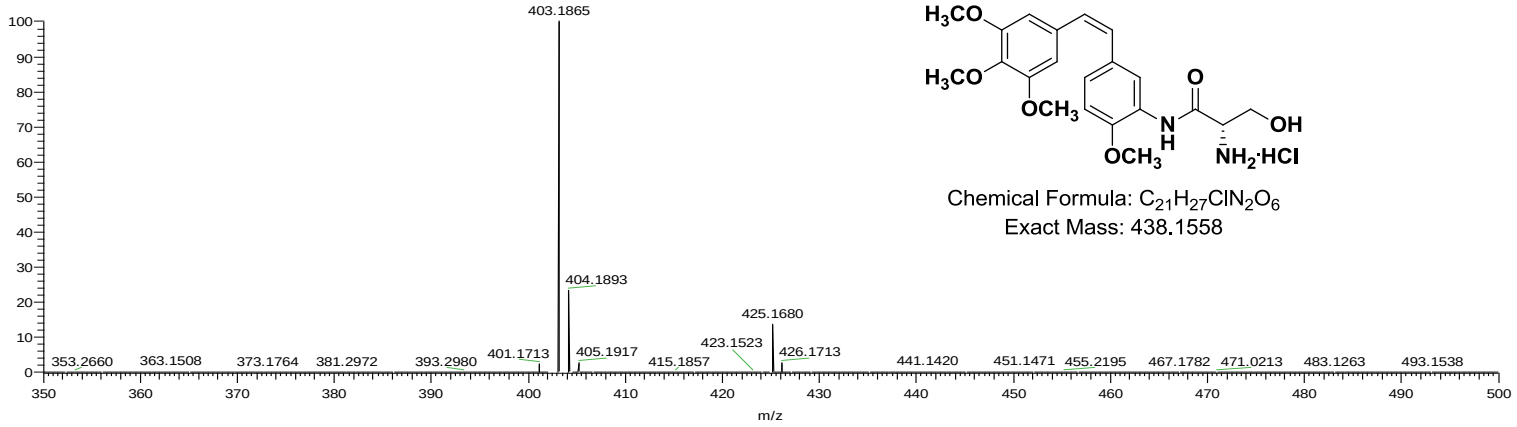
^{13}C NMR (CD_3OD, 150 MHz) for **AVE8062**



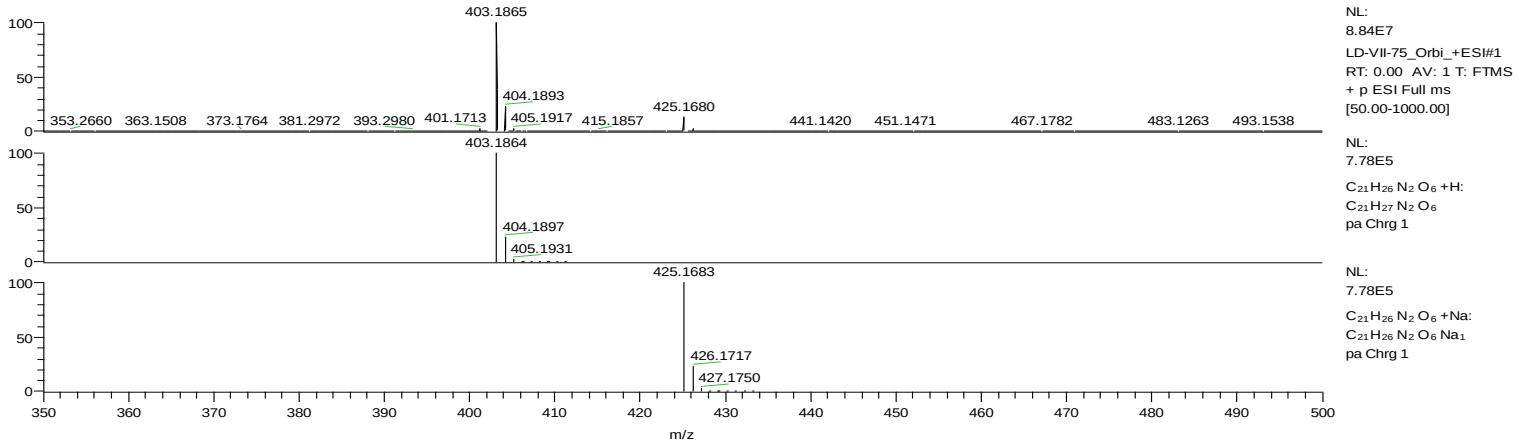
HRMS for **AVE8062**

C:\Xcalibur\...\0602\LD-VII-75_Orbi_+ESI 6/2/2015 5:17:27 PM LD-VII-75

LD-VII-75_Orbi_+ESI #1 RT: 0.00 AV: 1 NL: 8
T: FTMS + p ESI Full ms [50.00-1000.00]



Chemical Formula: $C_{21}H_{27}ClN_2O_6$
Exact Mass: 438.1558



NL:
8.84E7
LD-VII-75_Orbi_+ESI#1
RT: 0.00 AV: 1 T: FTMS
+ p ESI Full ms
[50.00-1000.00]

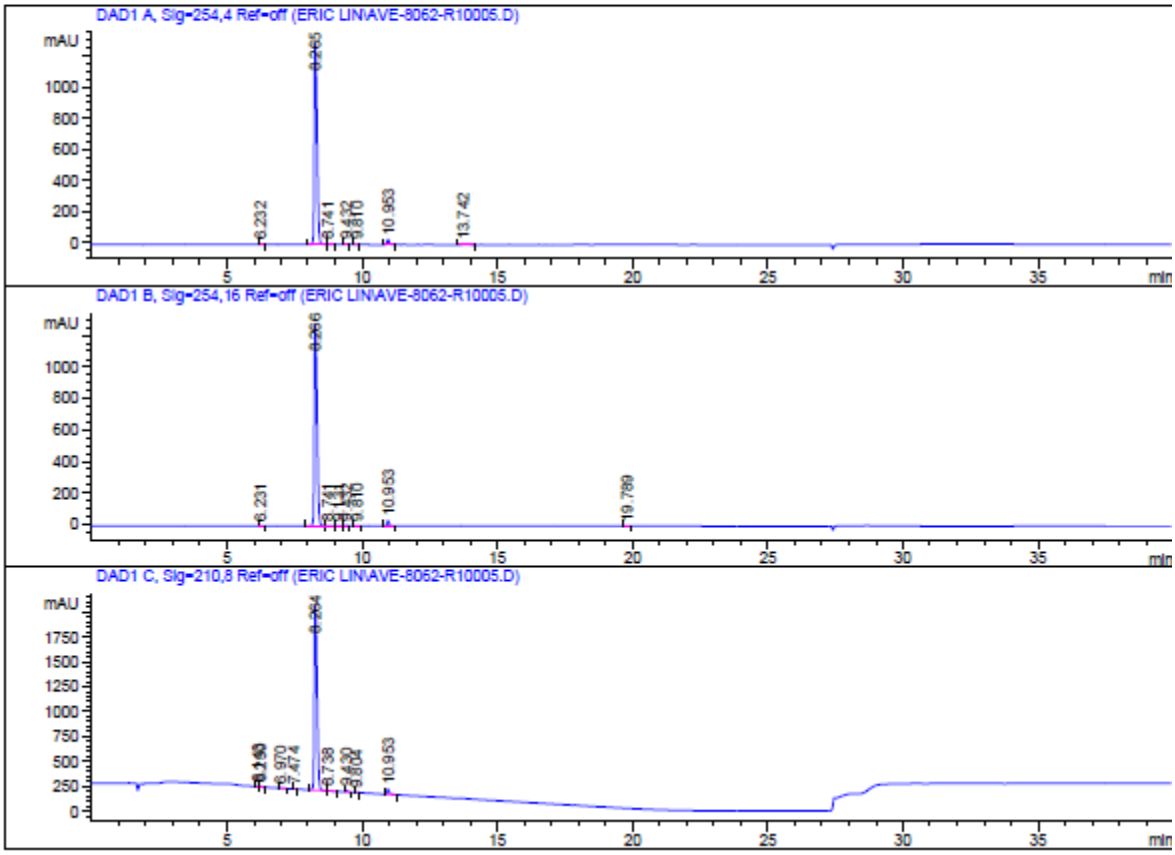
NL:
7.78E5
$C_{21}H_{26}N_2O_6$ +H:
$C_{21}H_{27}N_2O_6$
pa Chrg 1

NL:
7.78E5
$C_{21}H_{26}N_2O_6$ +Na:
$C_{21}H_{26}N_2O_6Na_1$
pa Chrg 1

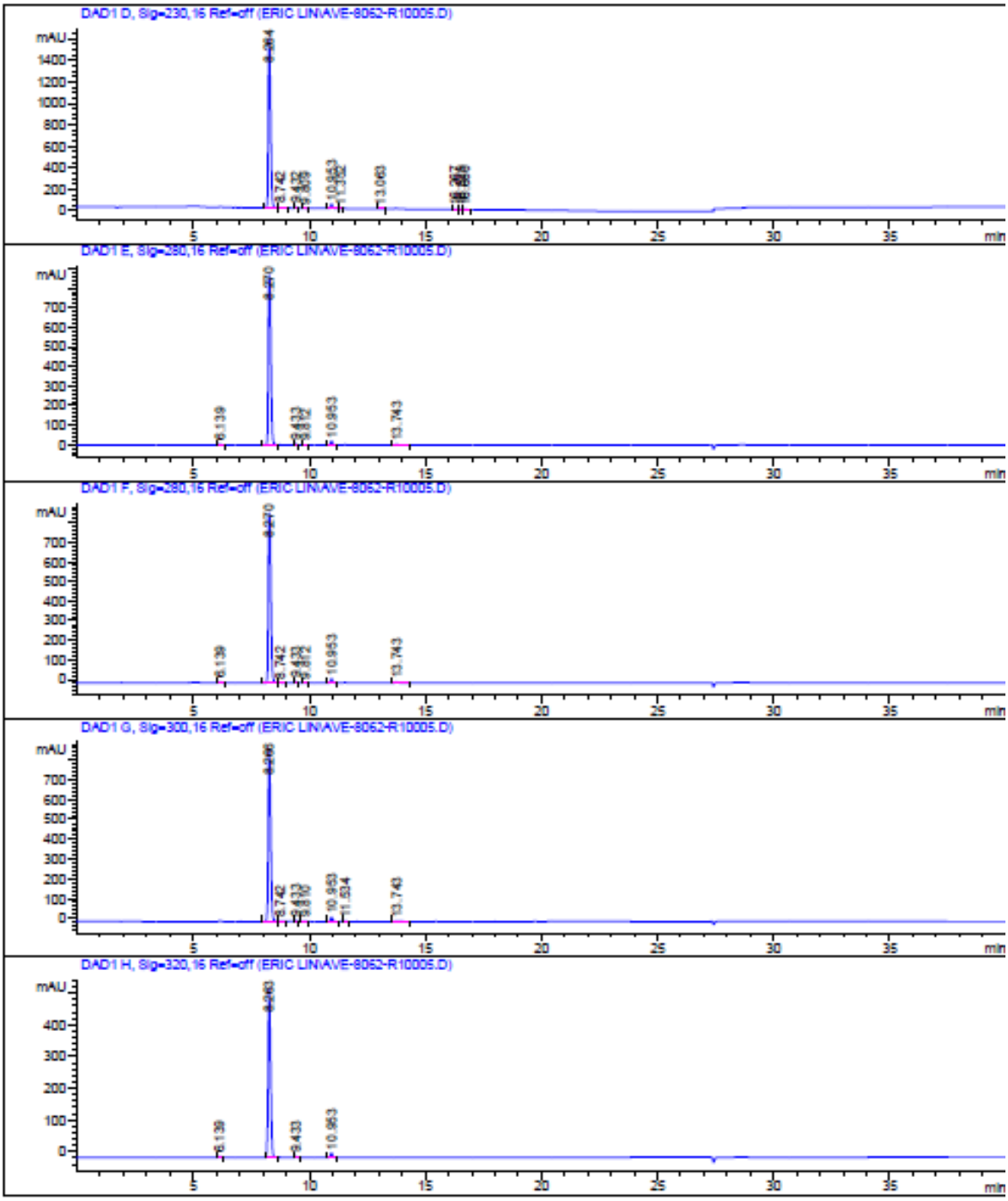
HPLC for **AVE8062**

```
Data File C:\CHEM32\1\DATA\ERIC LIN\AVE-8062-R10005.D
Sample Name: AVE-8062-R1

    ========================================================================
    Acq. Operator    : Eric Lin
    Acq. Instrument : Instrument 1                    Location :    -
    Injection Date   : 6/2/2015 12:52:41 PM
    Acq. Method      : C:\CHEM32\1\METHODS\MASTERMETHOD.M
    Last changed     : 6/2/2015 12:44:32 PM by Eric Lin
                       (modified after loading)
    Analysis Method : C:\CHEM32\1\DATA\ERIC LIN\AVE-8062-R10005.D\DA.M (MASTERMETHOD.M)
    Last changed     : 6/2/2015 2:28:03 PM by Eric Lin
    Sample Info      : WASH
```



Data File C:\CHEM32\1\DATA\ERIC LIN\AVE-8062-R10005.D
Sample Name: AVE-8062-R1



Data File C:\CHEM32\1\DATA\ERIC LIN\AVE-8062-R10005.D
Sample Name: AVE-8062-R1

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.232	VB	0.0727	7.52764	1.54801	0.0804
2	8.265	BV	0.1118	9095.58008	1296.55225	97.1534
3	8.741	VV	0.1199	15.38805	1.72812	0.1644
4	9.432	VB	0.0763	19.83443	3.97215	0.2119
5	9.810	BB	0.0796	11.97414	2.26929	0.1279
6	10.953	BB	0.0813	187.22200	34.52772	1.9998
7	13.742	BB	0.1673	24.55193	2.11709	0.2622
Totals :				9362.07827	1342.71463	

Signal 2: DAD1 B, Sig=254,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.231	VB	0.0696	7.30522	1.53343	0.0782
2	8.266	BV	0.1122	9068.53711	1286.86707	97.0999
3	8.741	VV	0.1311	19.50147	1.97604	0.2088
4	9.131	VV	0.1193	8.92628	1.00854	0.0956
5	9.432	VV	0.0819	25.36259	4.63422	0.2716
6	9.810	VB	0.0887	14.70501	2.42779	0.1575
7	10.953	BB	0.0812	185.16338	34.15549	1.9826
8	19.789	BB	0.0890	9.88684	1.72187	0.1059
Totals :				9339.38790	1334.32444	

Signal 3: DAD1 C, Sig=210,8 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.143	BV	0.0688	29.88397	6.60556	0.2195
2	6.230	VB	0.0716	43.34299	9.09865	0.3183
3	6.970	VB	0.1280	22.19154	2.31232	0.1630

Data File C:\CHEM32\1\DATA\ERIC LIN\AVE-8062-R10005.D
Sample Name: AVE-8062-R1

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
4	7.474	BB	0.0867	7.42790	1.19249	0.0546
5	8.264	BV	0.1121	1.31104e4	1863.22046	96.2920
6	8.738	VB	0.1087	29.73862	3.74297	0.2184
7	9.430	BB	0.0793	74.17680	14.59327	0.5448
8	9.804	BB	0.0708	12.46281	2.75495	0.0915
9	10.953	BV	0.0808	285.62799	53.03270	2.0979
Totals :				1.36152e4	1956.55338	

Signal 4: DAD1 D, Sig=230,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.264	BV	0.1086	1.09526e4	1585.03564	96.7945
2	8.742	VV	0.1154	24.17192	2.83805	0.2136
3	9.432	VB	0.0771	47.90776	9.44978	0.4234
4	9.809	BB	0.0822	14.80064	2.69172	0.1308
5	10.953	BV	0.0824	231.90778	42.03893	2.0495
6	11.352	VV	0.0903	10.09067	1.72249	0.0892
7	13.063	BB	0.1017	8.28207	1.24403	0.0732
8	16.267	BB	0.0863	7.61213	1.38036	0.0673
9	16.529	BV	0.0940	7.70709	1.28438	0.0681
10	16.680	VV	0.1121	10.23456	1.29656	0.0904
Totals :				1.13153e4	1648.98195	

Signal 5: DAD1 E, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.139	BB	0.0786	27.36969	5.27338	0.4053
2	8.270	BV	0.1215	6523.29443	869.58826	96.6053
3	9.433	BB	0.0751	25.17395	5.13926	0.3728
4	9.812	BB	0.0839	7.86839	1.39310	0.1165
5	10.953	BB	0.0813	117.99647	21.75365	1.7474
6	13.743	BB	0.1650	50.81811	4.39317	0.7526
Totals :				6752.52103	907.54081	

Data File C:\CHEM32\1\DATA\ERIC LIN\AVE-8062-R10005.D
Sample Name: AVE-8062-R1

Signal 6: DAD1 F, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.139	BB	0.0786	27.37036	5.27350	0.4045
2	8.270	BV	0.1215	6523.28369	869.58813	96.4126
3	8.742	VB	0.1035	13.48111	1.84035	0.1992
4	9.433	BB	0.0751	25.17586	5.13947	0.3721
5	9.812	BB	0.0839	7.87010	1.39290	0.1163
6	10.953	BB	0.0813	118.00455	21.75415	1.7441
7	13.743	BB	0.1650	50.82064	4.39342	0.7511
Totals :				6766.00630	909.38193	

Signal 7: DAD1 G, Sig=300,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.266	BV	0.1147	6275.62891	864.02368	96.8008
2	8.742	VB	0.1013	11.85244	1.66092	0.1828
3	9.433	BB	0.0753	24.58060	5.00164	0.3792
4	9.810	BB	0.0814	7.66697	1.41149	0.1183
5	10.953	BB	0.0824	125.00182	22.64232	1.9281
6	11.534	VB	0.0826	7.59221	1.37027	0.1171
7	13.743	BB	0.1655	30.70807	2.64326	0.4737
Totals :				6483.03103	898.75359	

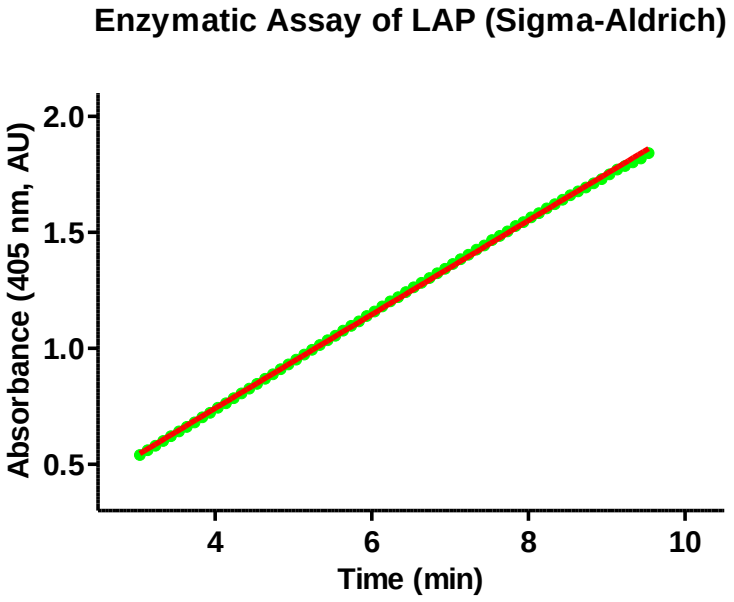
Signal 8: DAD1 H, Sig=320,16 Ref=off

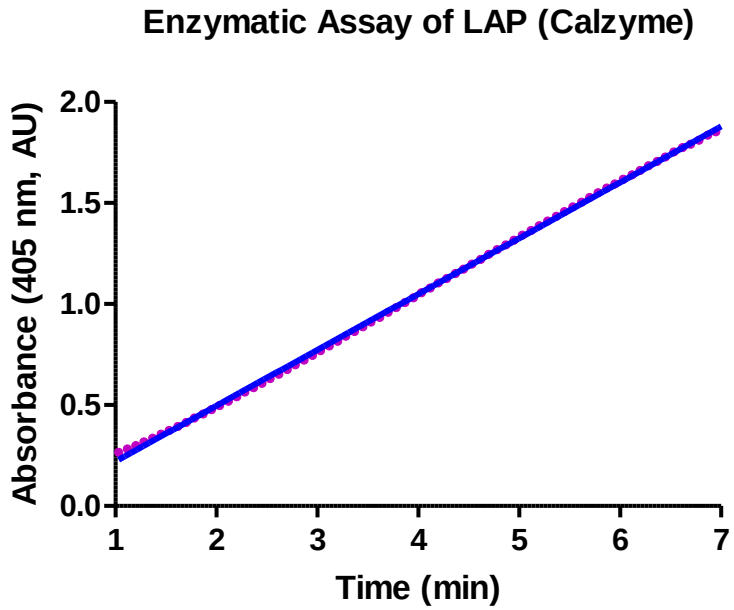
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.139	BB	0.0714	17.48327	3.81790	0.4340
2	8.263	BV	0.1134	3913.47534	534.81958	97.1548
3	9.433	BB	0.0760	14.63547	2.94218	0.3633
4	10.953	BB	0.0840	82.49001	14.57937	2.0479
Totals :				4028.08409	556.15903	

*** End of Report ***

Enzymatic Assays of Leucine Aminopeptidase

(Determination of Enzyme Units; Substrate: *L*-leucine *p*-nitroanilide)





HPLC Conditions

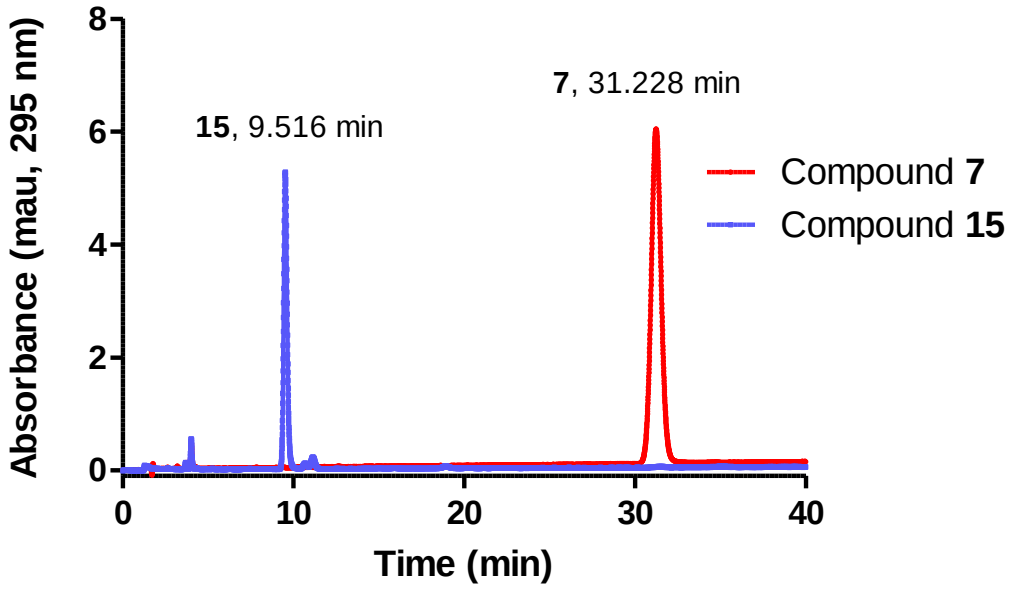
Method 1: solvent A acetonitrile, solvent B 0.05% TFA in H_2O; isocratic, 30%A/70%B.

Method 2: solvent A acetonitrile, solvent B 0.05% TFA in H_2O; isocratic, 28%A/72%B.

Method 3: solvent A acetonitrile, solvent B 0.05% TFA in H_2O; isocratic, 26%A/74%B.

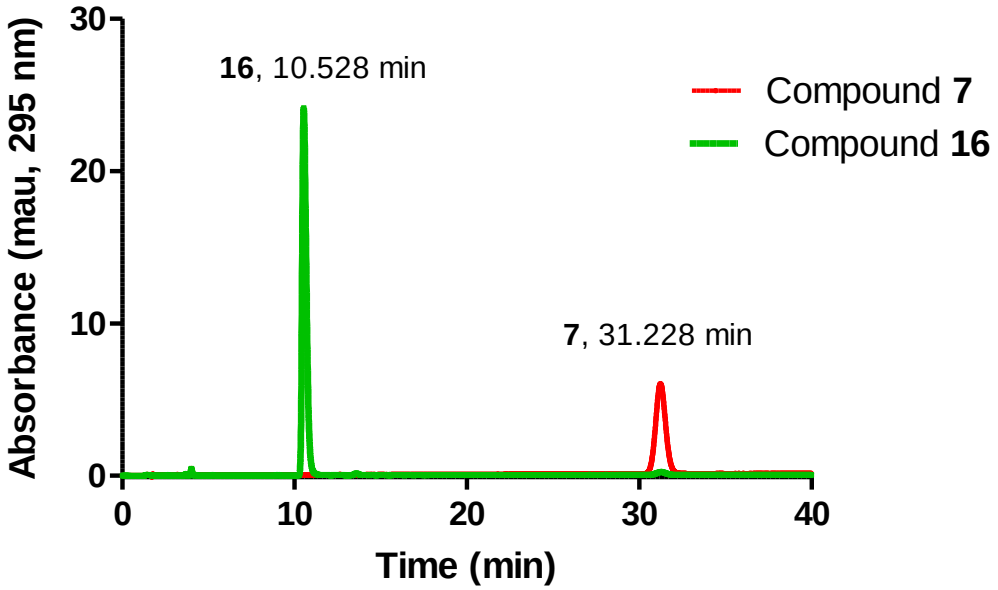
HPLC Chromatograms of Compounds 7, 15 and 16

Chromatogram of 40 µM Each of Compounds 7 and 15 (Method 3)



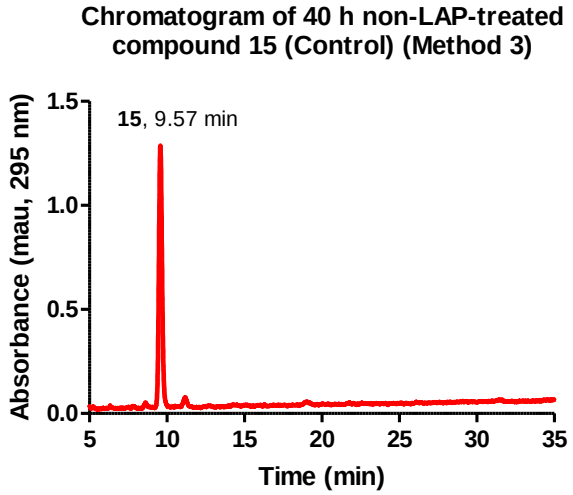
[Note: Retention times of compounds **7** and **15** were determined separately as standards in HPLC with the same mobile phase. The chromatograms were combined for comparison purposes.]

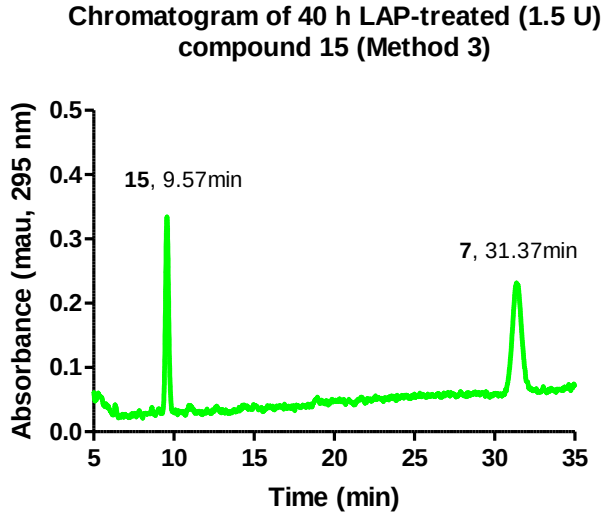
Chromatogram of 40 µM Each of Compounds 7 and 16 (Method 3)

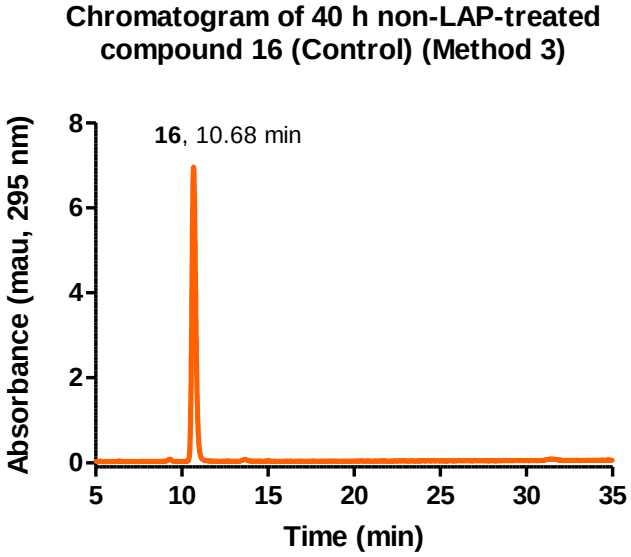


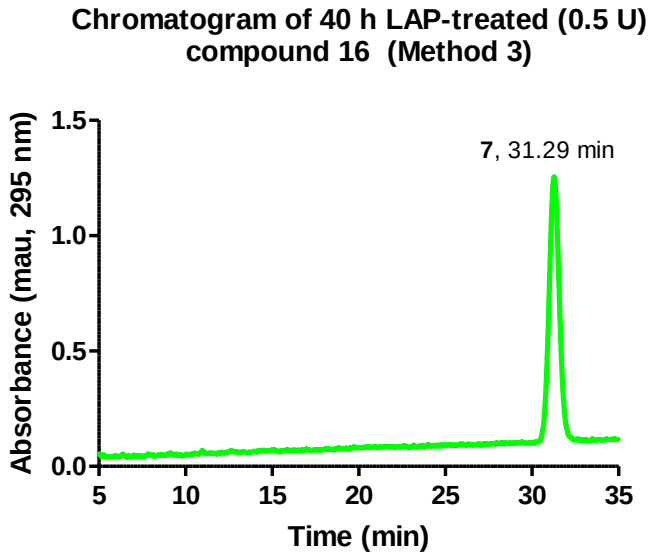
[Note: Retention times of compounds **7** and **16** were determined separately as standards in HPLC with the same mobile phase. The chromatograms were combined for comparison purposes.]

HPLC Chromatograms of Compounds 15 and 16 Before and After Leucine Aminopeptidase Digestion



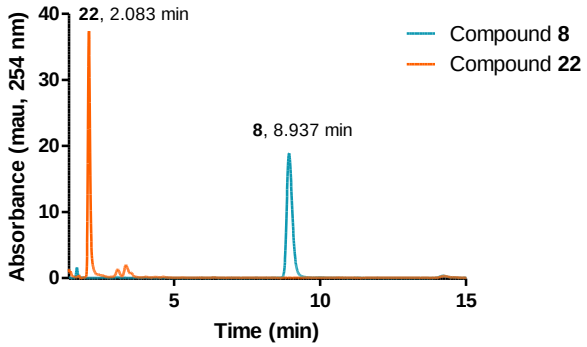






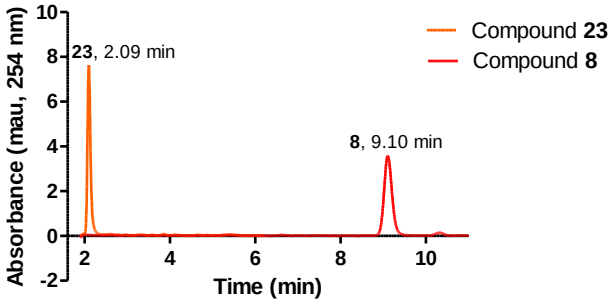
HPLC Chromatograms of Compounds 8, 22, 23 and 24

Chromatogram of 40 μM Each of Compounds 8 and 22 (Method 3)



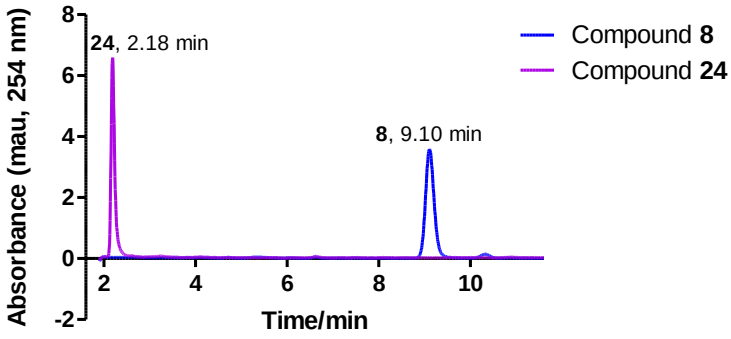
[Note: Retention times of compounds **8** and **22** were determined separately as standards in HPLC with the same mobile phase. The chromatograms were combined for comparison purposes.]

Chromatogram of 20 μM Each of Compounds 8 and 23 (Method 3)



[Note: Retention times of compounds **8** and **23** were determined separately as standards in HPLC with the same mobile phase. The chromatograms were combined for comparison purposes.]

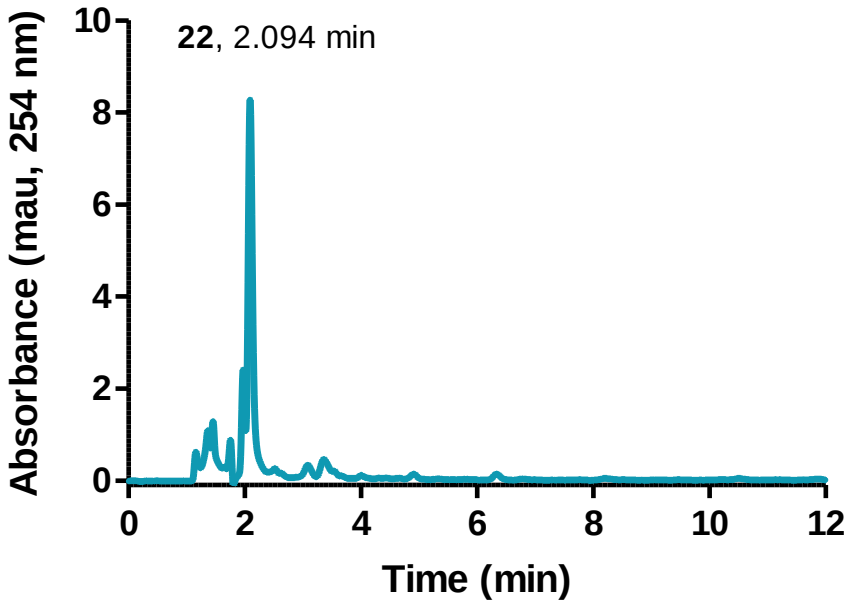
Chromatogram of 20 μM Each of Compounds 8 and 24 (Method 3)



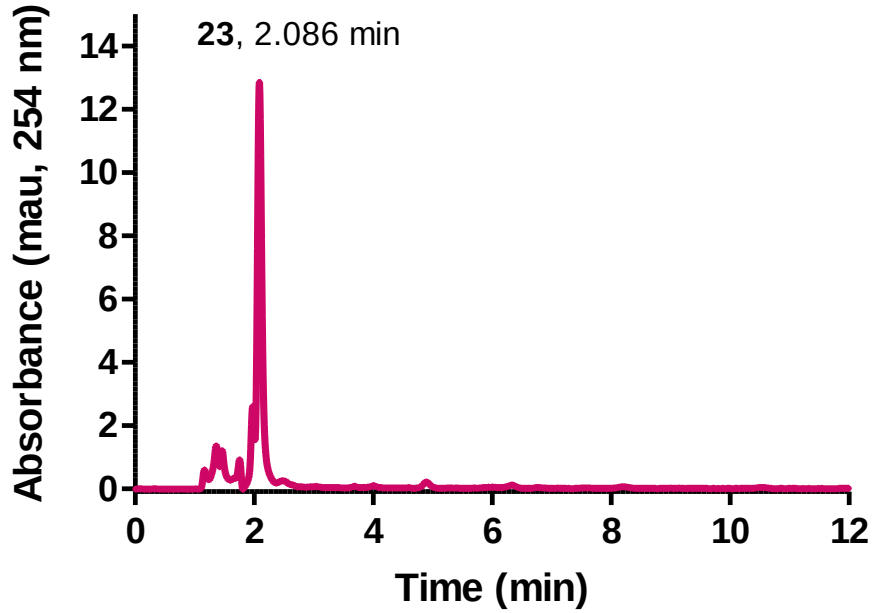
[Note: Retention times of compounds **8** and **24** were determined separately as standards in HPLC with the same mobile phase. The chromatograms were combined for comparison purposes.]

HPLC Chromatograms of Compounds 22 and 23 After Leucine Aminopeptidase Digestion

Chromatogram of 40 h LAP-treated (1.5 U) compound 22: No cleavage observed (Method 3)

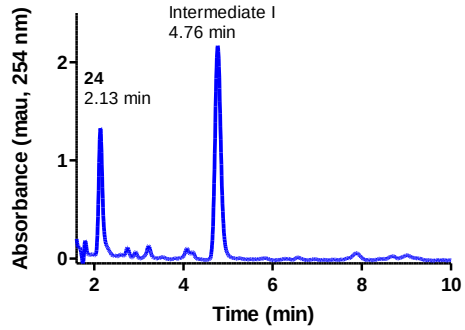


Chromatogram of 40 h LAP-treated (1.5 U) compound 23 : No cleavage observed (Method 3)

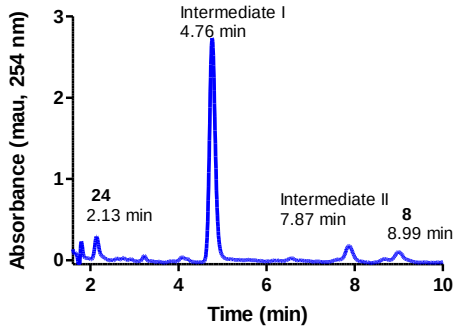


HPLC Chromatograms of Compound 24 After Leucine Aminopeptidase Digestion

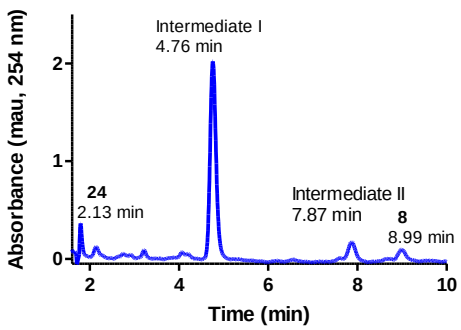
Chromatogram of LAP-treated (0.12 U) compound 24 incubated for 3 h (Method 3)



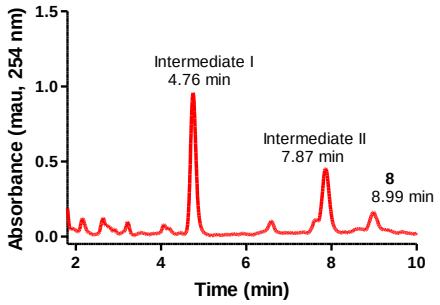
Chromatogram of LAP-treated (0.12 U) compound 24 incubated for 7 h (Method 3)



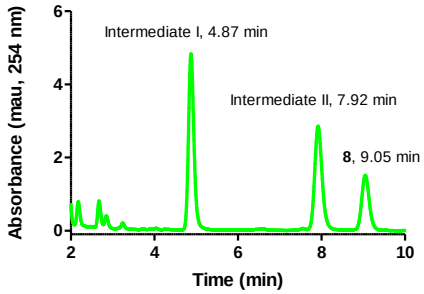
Chromatogram of LAP-treated (0.12 U) compound 24 incubated for 10 h (Method 3)



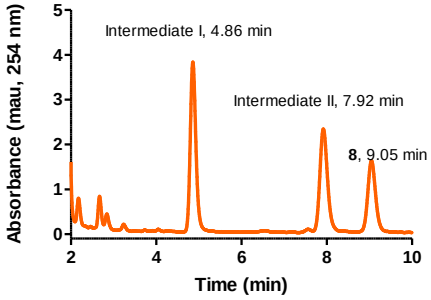
Chromatogram of LAP-treated (0.12 U) compound 24 incubated for 25 h (Method 3)



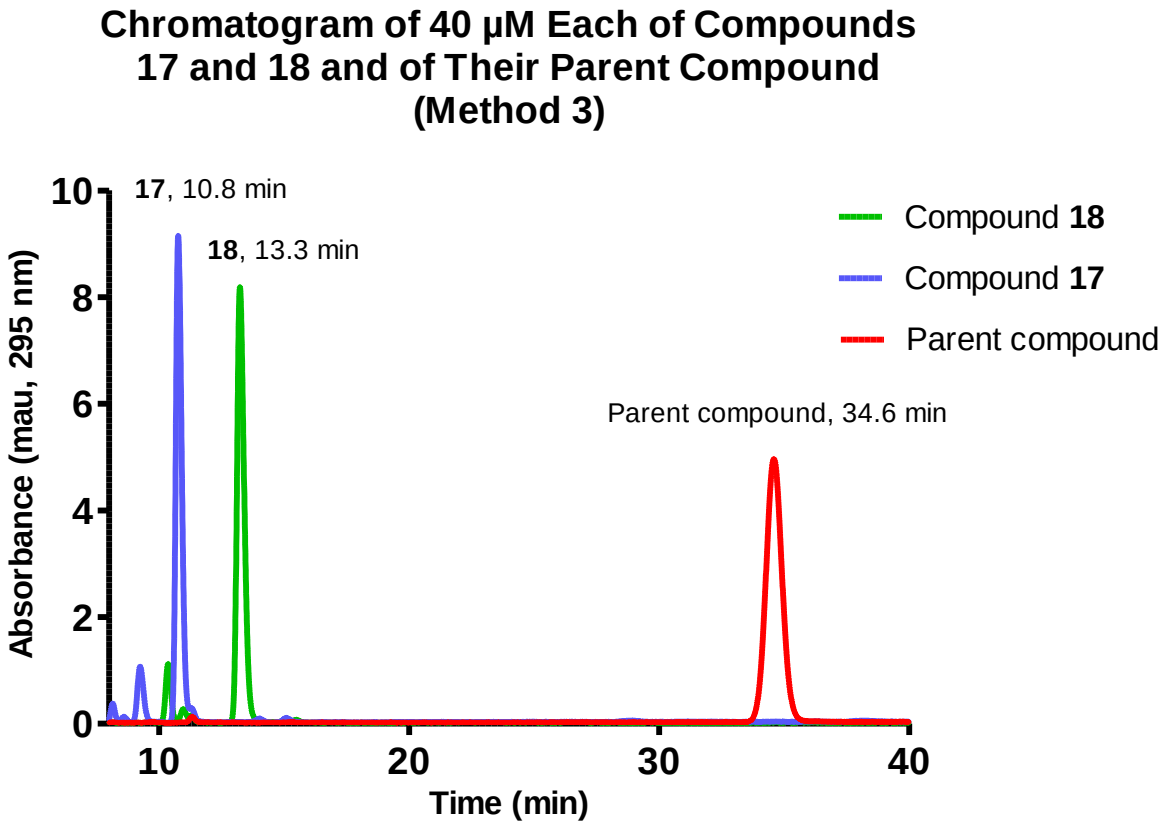
Chromatogram of LAP-treated (1.5 U) compound 24 incubated for 20 h (Method 3)



Chromatogram of LAP-treated (3.7 U) compound 24 incubated for 20 h (Method 3)

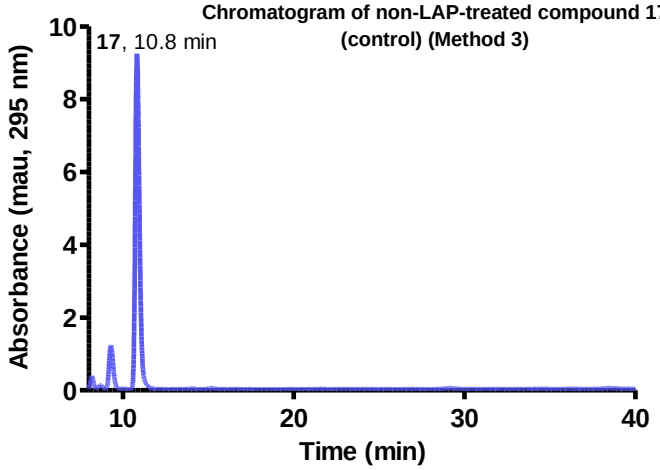


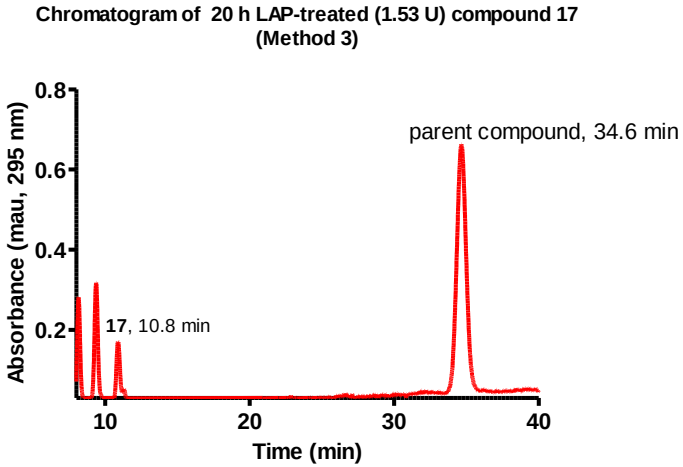
HPLC Chromatograms of Compounds 17, 18 and of Their Parent Compound

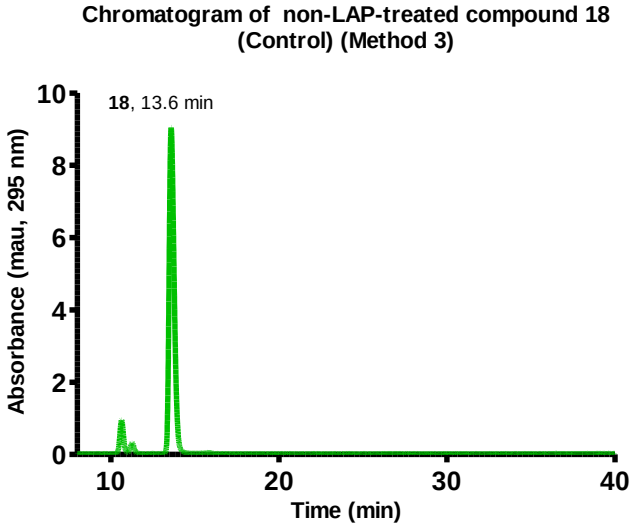


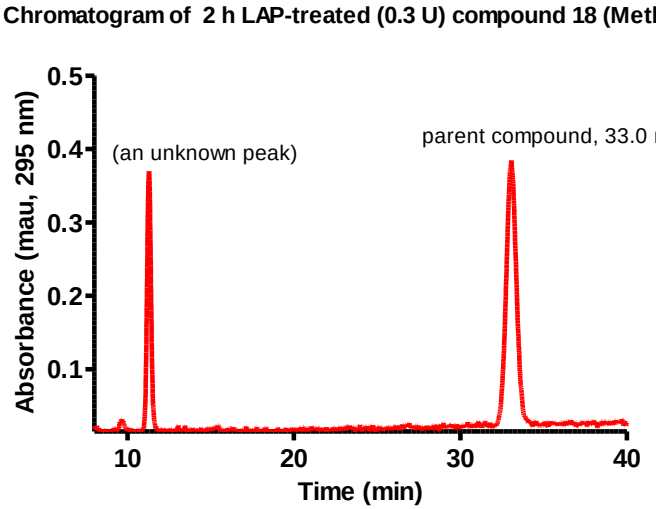
[Note: Retention times of compounds **17** and **18** and of their parent compound were determined separately as standards in HPLC with the same mobile phase. The chromatograms were combined for comparison purposes.]

HPLC Chromatograms of Compounds 17 and 18 Before and After Leucine Aminopeptidase Digestion



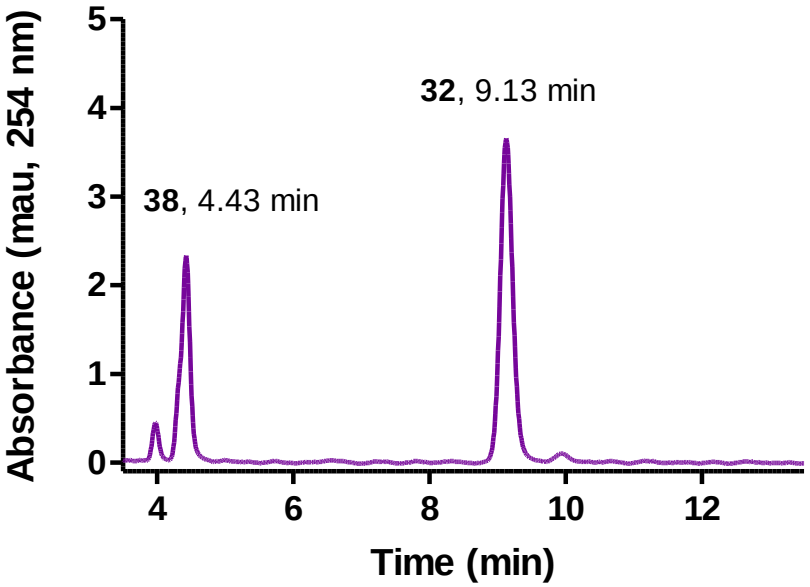




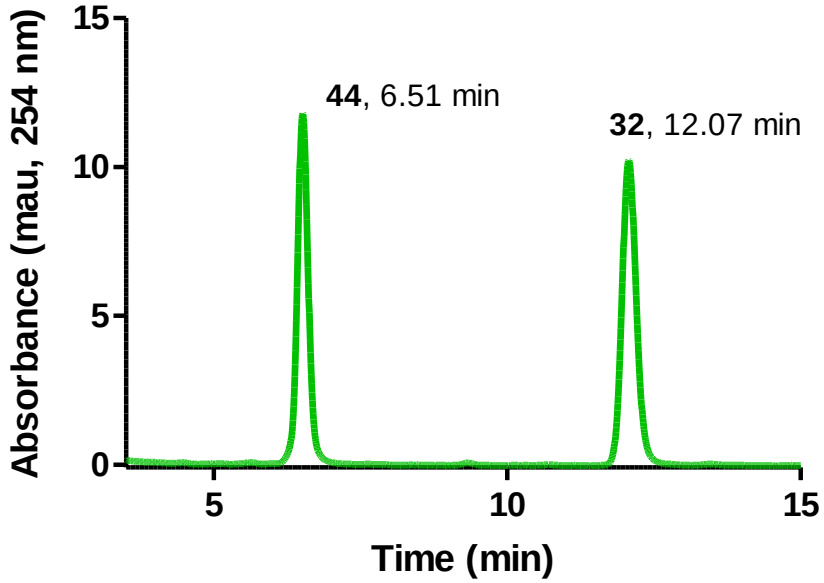


HPLC Chromatograms of Compounds 32, 38 and 44

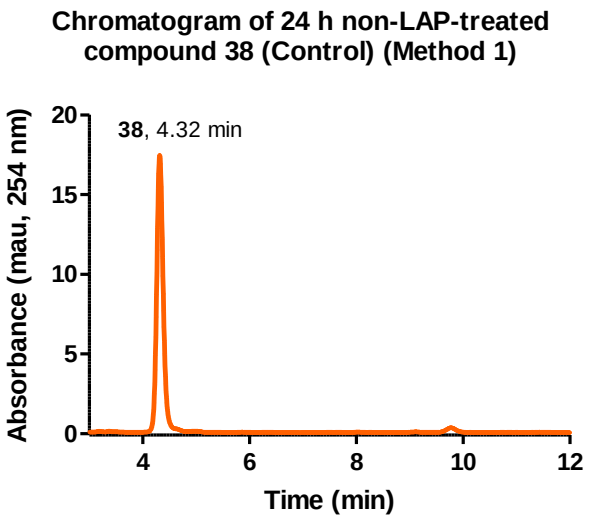
Chromatogram of Compounds 32 and 38 (Method 1)

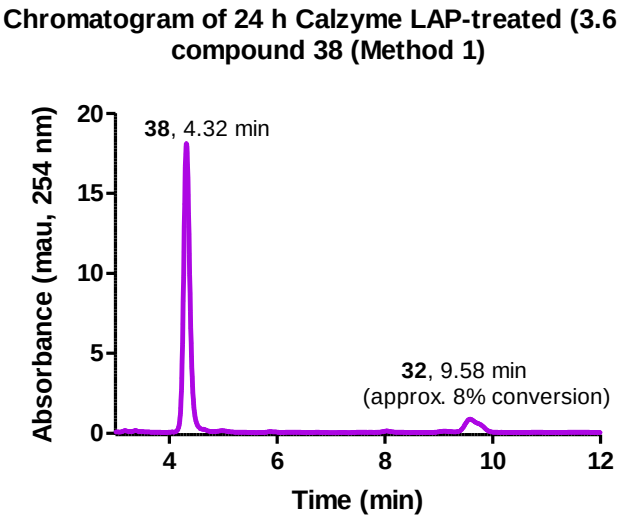


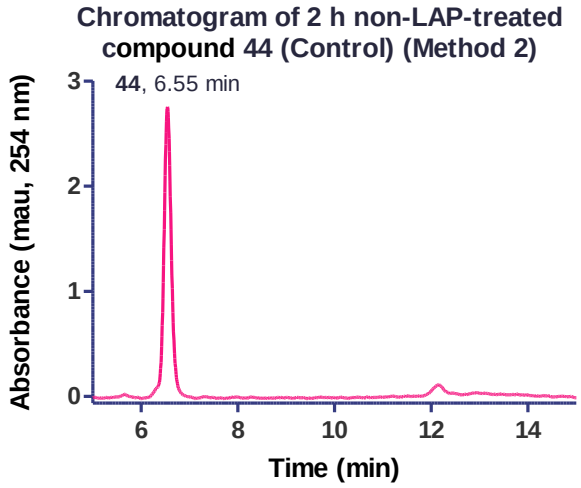
Chromatogram of Compounds 32 and 44 (Method 2)

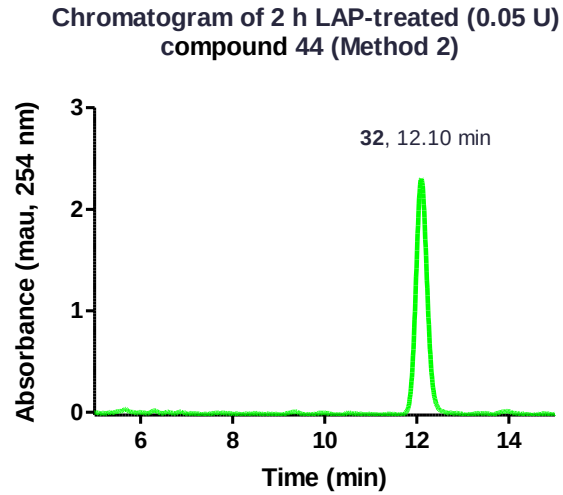


HPLC Chromatograms of Compounds 38 and 44 Before and After Leucine Aminopeptidase Digestion



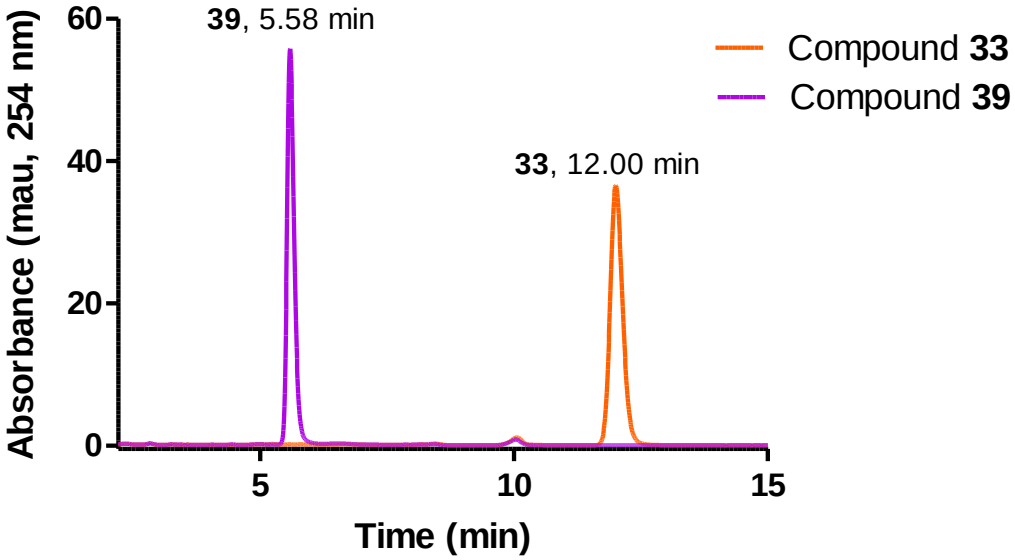






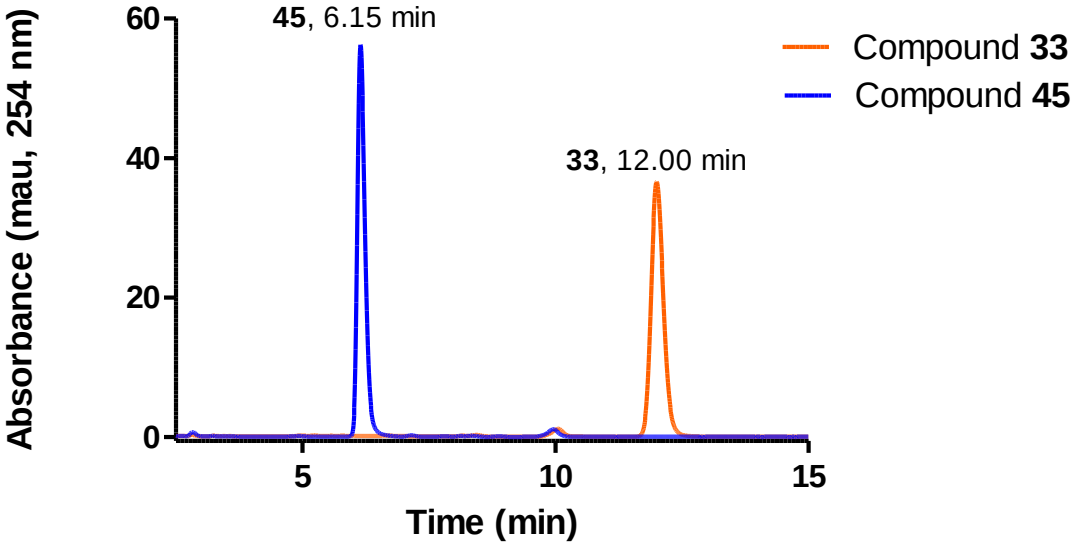
HPLC Chromatograms of Compounds 33, 39 and 45

Chromatogram of 20 μM Each of Compounds 33 and 39 (Method 1)



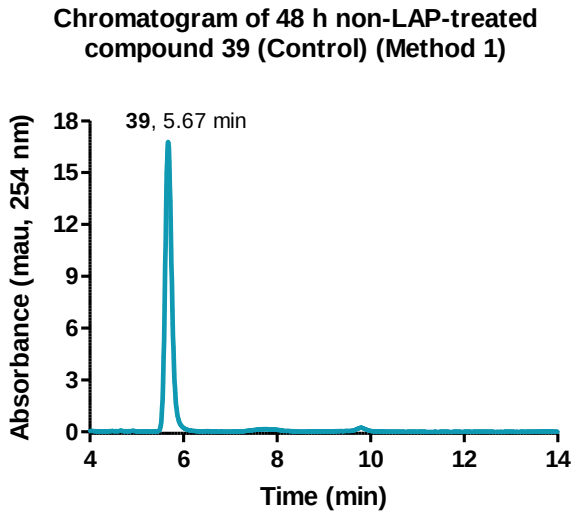
[Note: Retention times of compounds **33** and **39** were determined separately as standards in HPLC with the same mobile phase. The chromatograms were combined for comparison purposes.]

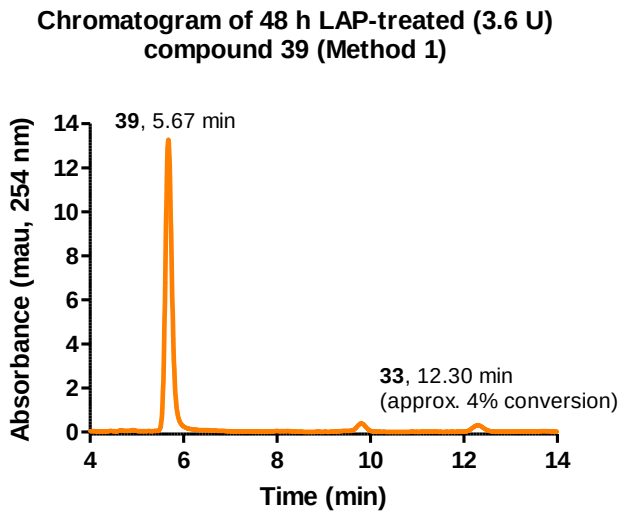
Chromatogram of 20 μM Each of Compounds 33 and 45 (Method 1)

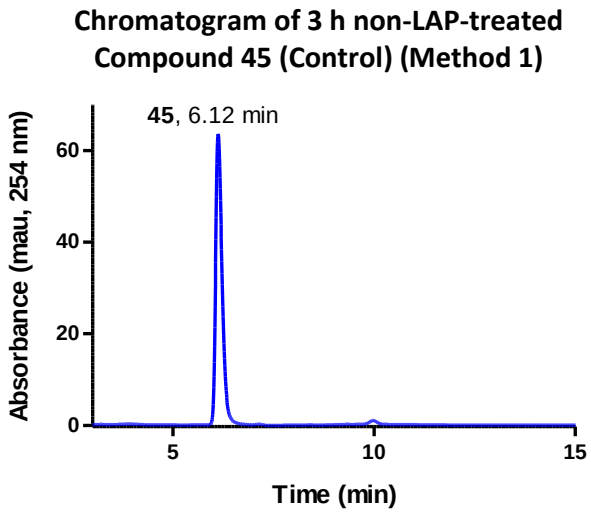


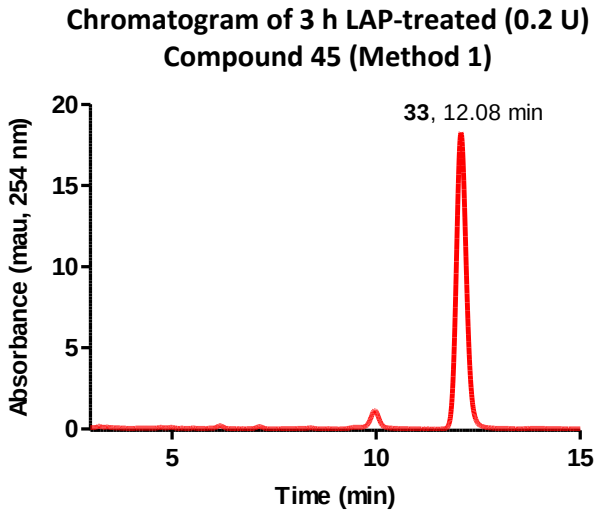
[Note: Retention times of compounds **33** and **45** were determined separately as standards in HPLC with the same mobile phase. The chromatograms were combined for comparison purposes.]

HPLC Chromatograms of Compounds 39 and 45 Before and After Leucine Aminopeptidase Digestion



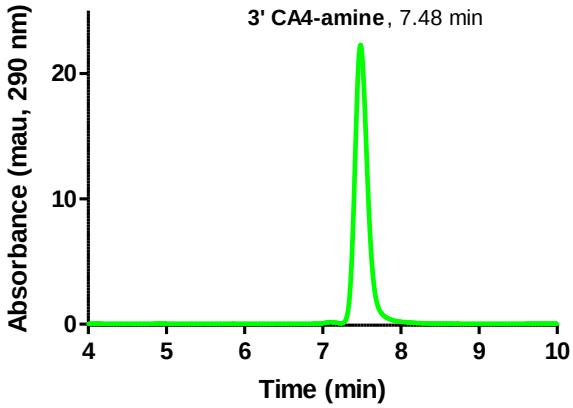




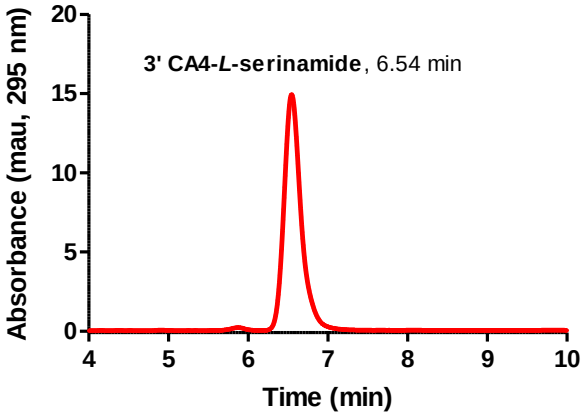


HPLC Chromatograms of 3' CA4-amine, 3' CA4-*L*-serinamide and AVE8062

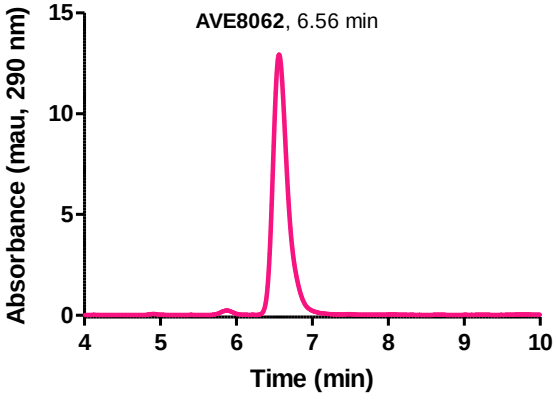
Chromatogram of 40 μM Standard 3' CA4-amine (Method 1)



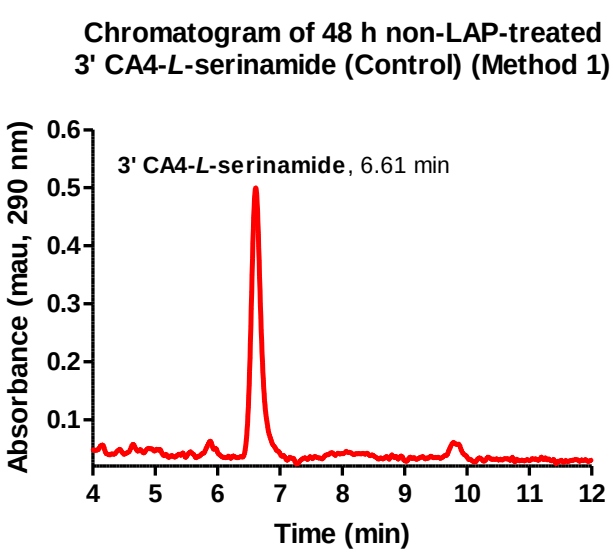
Chromatogram of 40 μM Standard 3' CA4-*L*-serinamide (Method 1)

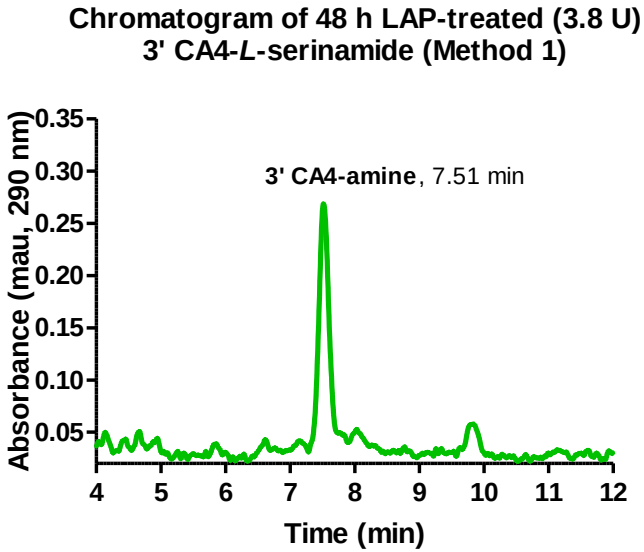


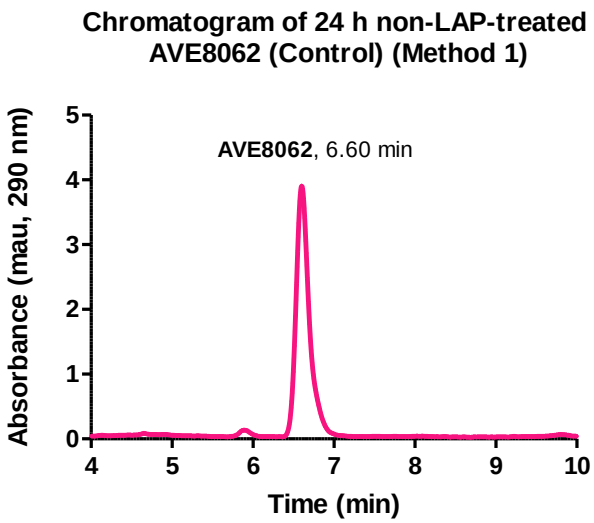
Chromatogram of 40 μM Standard AVE8062 (Method 1)

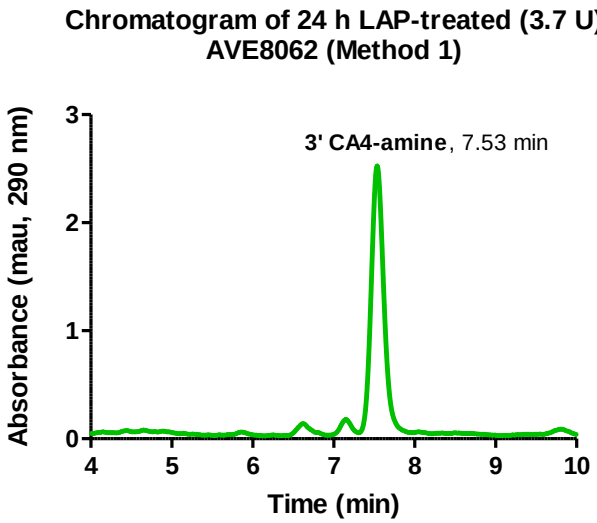


HPLC Chromatograms of 3' CA4-*L*-serinamide and AVE8062 Before and After Leucine Aminopeptidase Digestion

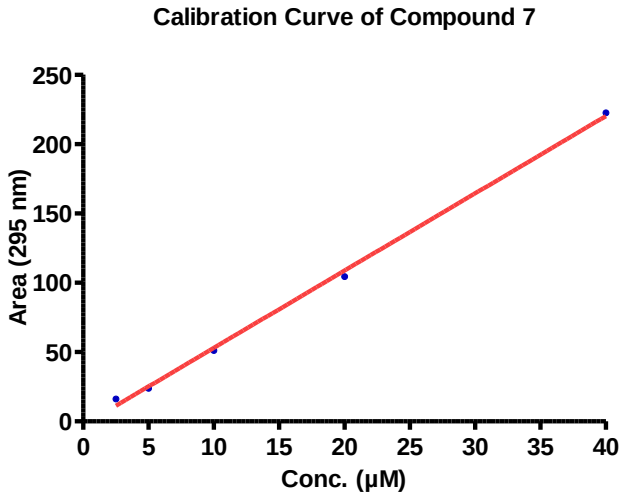


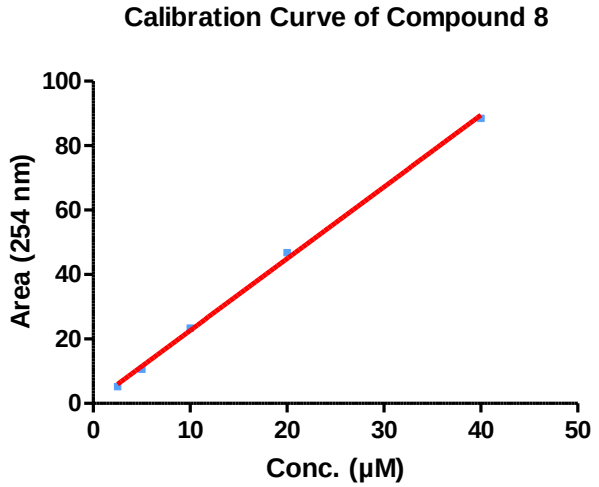


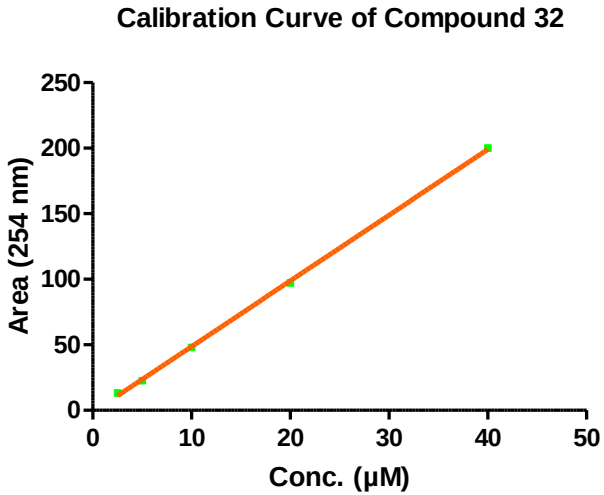


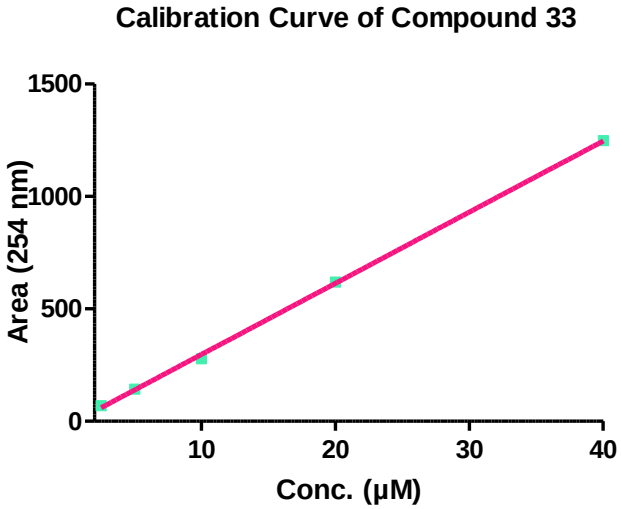


Calibration Curves of Parent Compounds 7, 8, 32 and 33

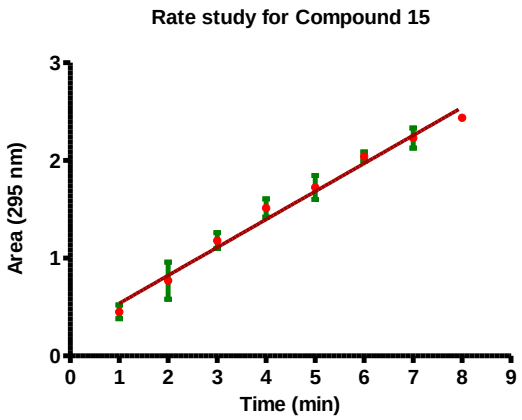


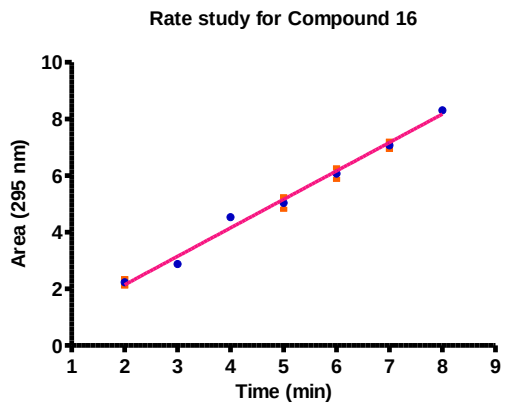


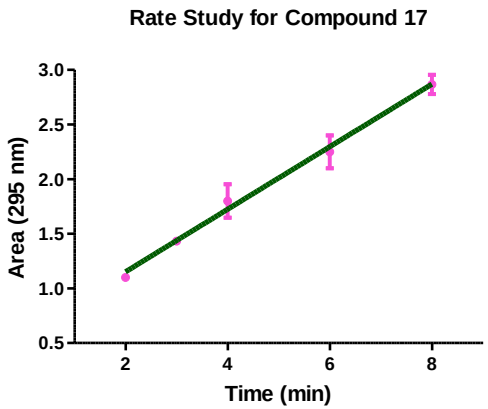


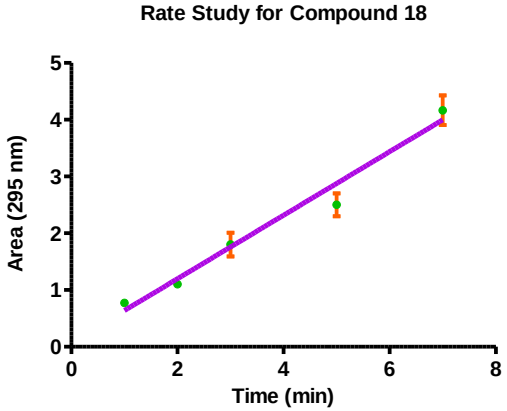


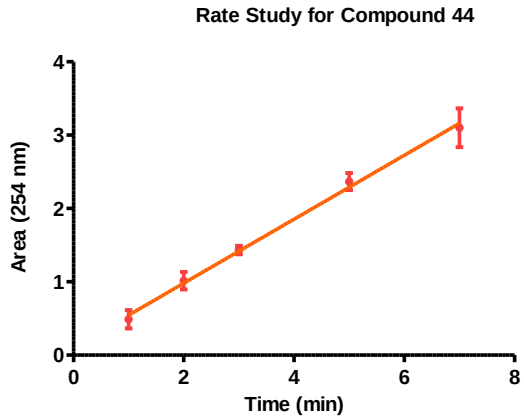
Rate Studies for Cleavable Compounds 15, 16, 17, 18, 44 and 45

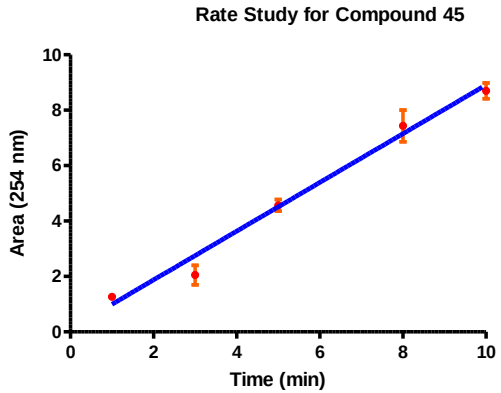












Evaluation of Aqueous Solubility

A preliminary evaluation of the water solubility of two sets of the most promising parent drugs and their respective prodrug conjugates (as representative examples) was carried out, which utilized solution cloudiness (as evidenced by visual examination with the naked eye) as the transition between soluble and non-soluble.

Compound **7** (parent drug): 0.29 mg of compound **7** was weighed out in a vial and 580 µL of DI water was added in several sequential aliquots. Only partial solubility of compound **7** in water was observed even at the lowest concentration, thus aqueous solubility was < 0.5 g/L. Compound **16** (prodrug of compound **7**): 0.57 mg of prodrug **16** was weighed out in a vial, and upon the addition of a total of 90 µL of DI water (added in several aliquots), the solution remained cloudy. Upon the addition of a total of 190 µL of DI water, complete solubility of prodrug **16** was achieved. Thus, the calculated approximate solubility of prodrug **16** in water was 3 g/L.

Compound **32** (parent drug): 0.24 mg of compound **32** was weighed out in a vial and 480 µL of DI water was added (in several aliquots). Only a partial solubility of compound **32** in water was observed even at the lowest concentration, thus aqueous solubility was < 0.5 g/L. Compound **44** (prodrug of compound **32**): 0.54 mg of prodrug **44** was weighed out in a vial. Upon the addition of a total of 120 µL of DI water, complete solubility of prodrug **44** was observed. Thus, the calculated approximate solubility of prodrug **44** in water was 4.5 g/L.