Electronic Supplementary Material (ESI) for MedChemComm.

Supplemental Data File

Synthesis and biological evaluation of structurally diverse conformationally restricted chalcones and related analogues.

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4. Experimental section

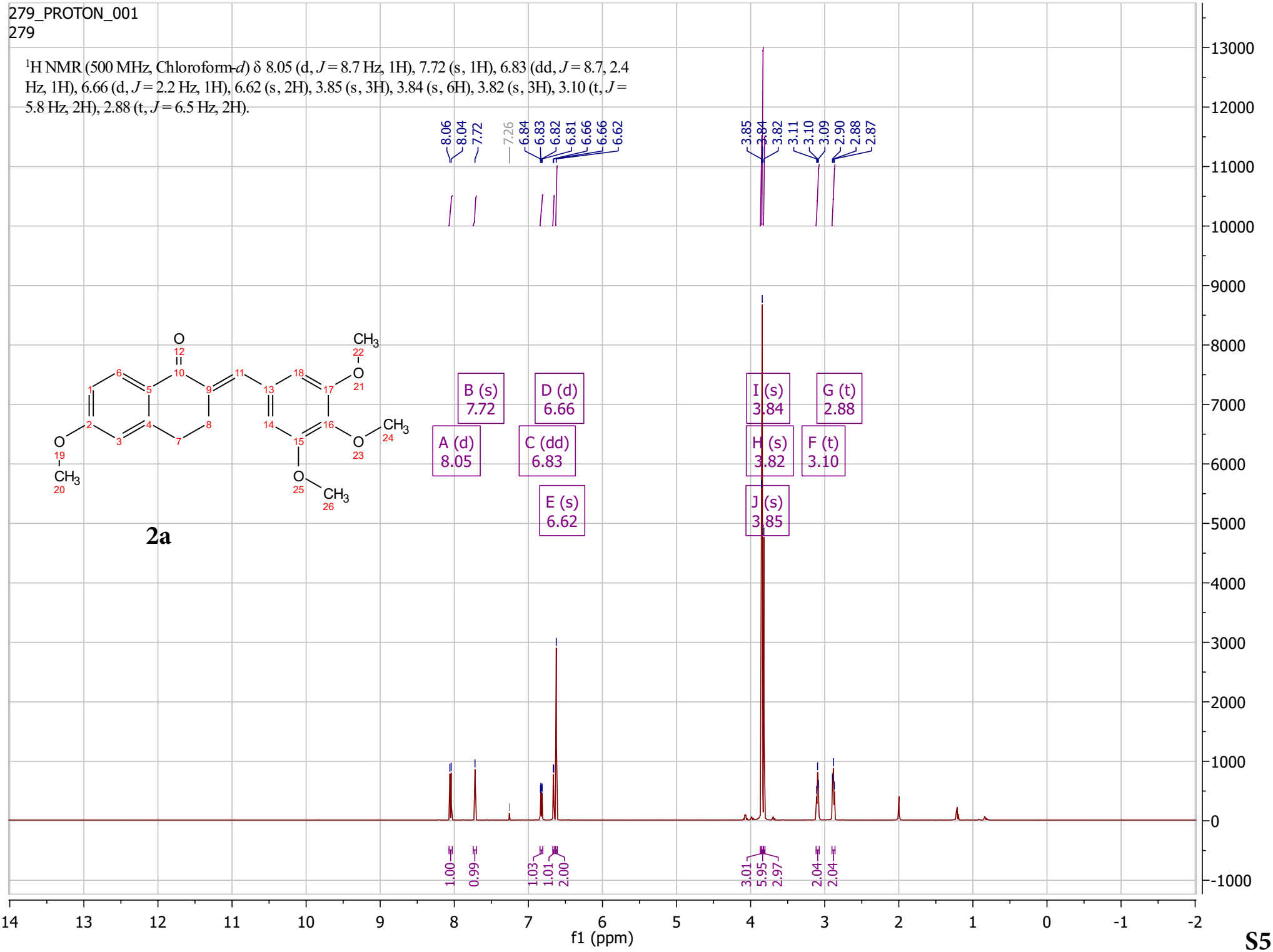
4.1. Chemistry

4.1.1. General materials and methods. Dichloromethane, methanol, dimethylformamide, and tetrahydrofuran (THF) were used in their anhydrous forms, as obtained from the chemical suppliers. Reactions were performed under an inert nitrogen atmosphere. Reactions carried out under microwave irradiation were performed with a Biotage Initiator Microwave Synthesizer. Thin-layer chromatography (TLC) plates (precoated glass plates with silica gel 60 F254, 0.25 mm thickness) were used to monitor reactions. Purification of intermediates and products was carried out with a Biotage Isolera flash purification system using silica gel (200-400 mesh, 60 Å) or manually in glass columns. Intermediates and products synthesized were characterized on the basis of their ^{1}H NMR (600, 500 MHz), ^{13}C NMR (151 or 126 MHz) spectroscopic data using a Bruker Avance III 600 MHz or a Varian VNMRs 500 MHz instrument. Spectra were recorded in $CDCl_3$ or $(CD_3)_2SO$. All chemical shifts are expressed in ppm (δ), coupling constants (*J*) are presented in Hz, and peak patterns are reported as singlet (s), doublet (d), triplet (t), quartet (q), septet (sept), double doublet (dd), and multiplet (m). Purity of the final compounds was further analyzed at 25 ºC using an Agilent 1200 HPLC system with a diode-array detector (λ = 190-400 nm), a Zorbax XDB-C18 HPLC column (4.6 mm x 150 mm, 5 µm), and a Zorbax reliance cartridge guard-column; method A: solvent A, acetonitrile, solvent B, H_2O; gradient, 50% A/50% B to 90% A/10% B over 0 to 30 min; post-time 10 min; flow rate 1.0 mL/min; injection volume 20 µL; monitored at wavelengths of 254, 210, 280, 300, and 320 nm. Mass spectrometry was carried out under positive ESI (electrospray ionization) using a Thermo Scientific LTQ Orbitrap Discovery instrument.

279_PROTON_001
279

^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.05 (d, *J* = 8.7 Hz, 1H), 7.72 (s, 1H), 6.83 (dd, *J* = 8.7, 2.4 Hz, 1H), 6.66 (d, *J* = 2.2 Hz, 1H), 6.62 (s, 2H), 3.85 (s, 3H), 3.84 (s, 6H), 3.82 (s, 3H), 3.10 (t, *J* = 5.8 Hz, 2H), 2.88 (t, *J* = 6.5 Hz, 2H).

2a



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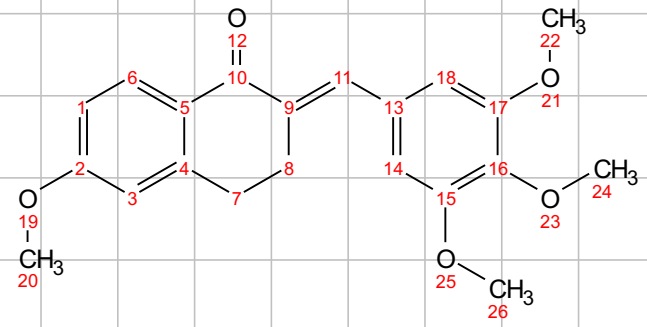
^{13}C NMR (126 MHz, cdcl$_3$) δ 186.46, 163.52, 153.00, 145.56, 138.38, 136.08, 135.02, 131.46, 130.63, 126.94, 113.28, 112.22, 107.14, 60.90, 56.15, 55.41, 29.19, 27.32.



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CJM-IV-279

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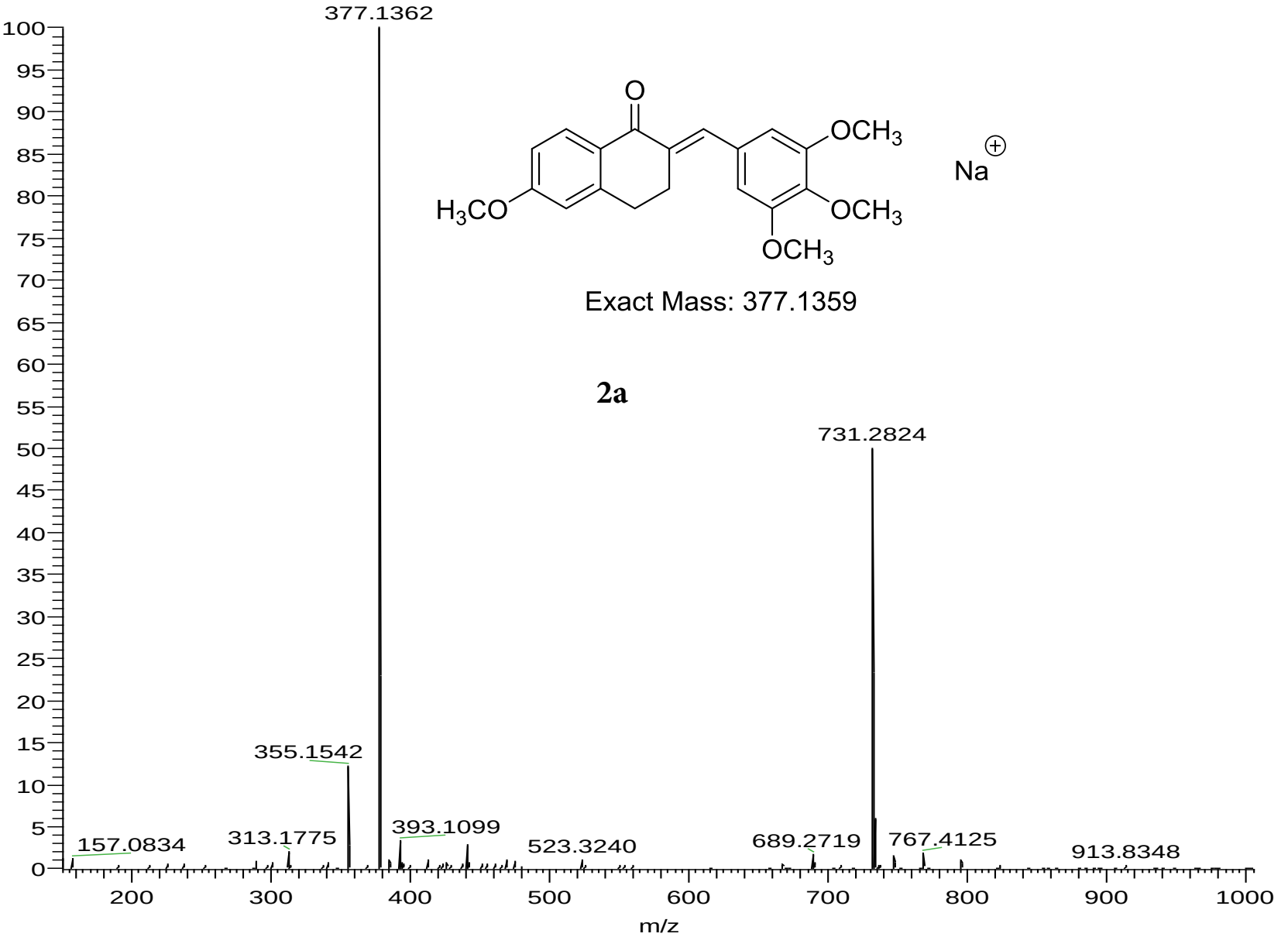

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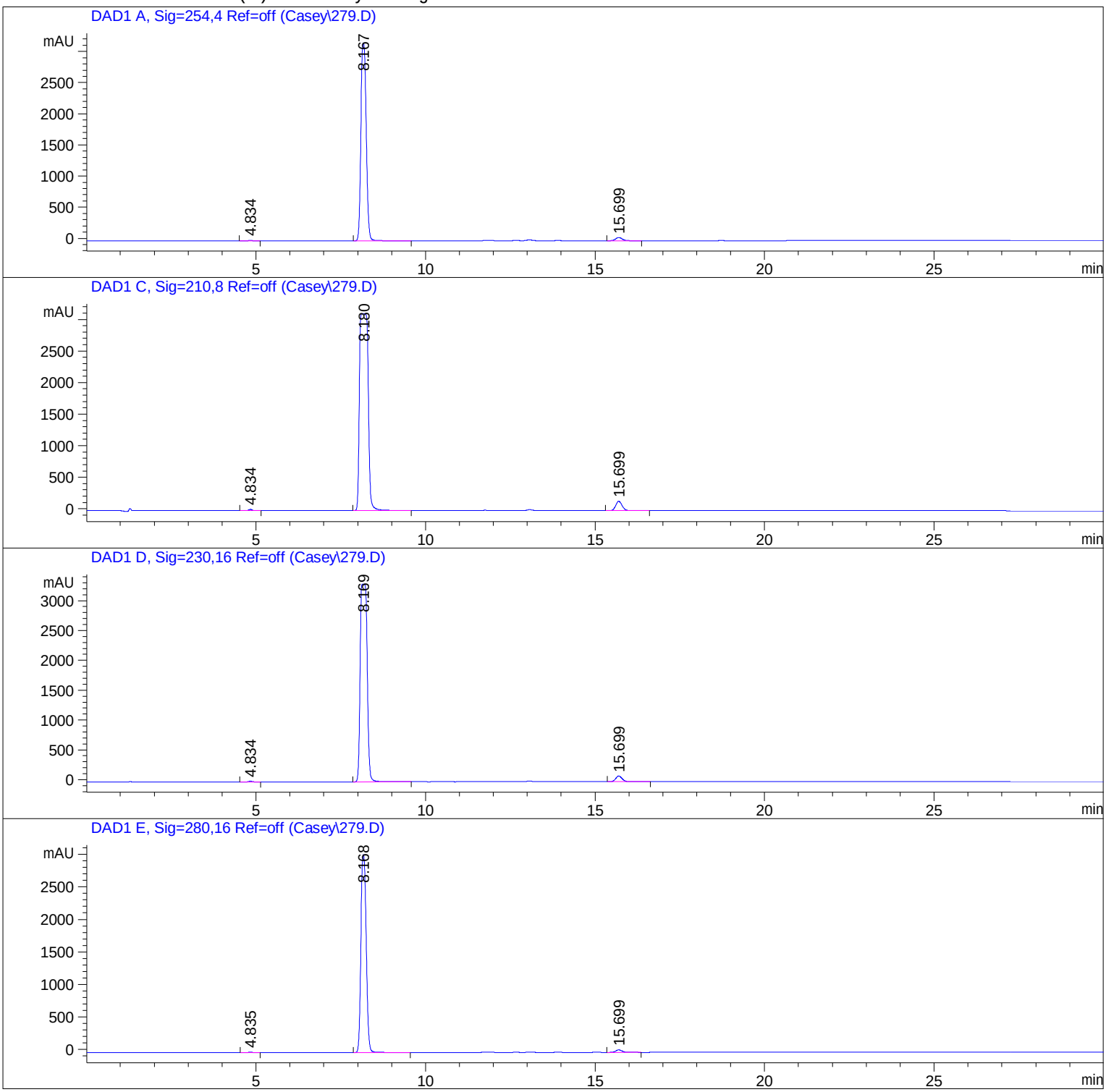

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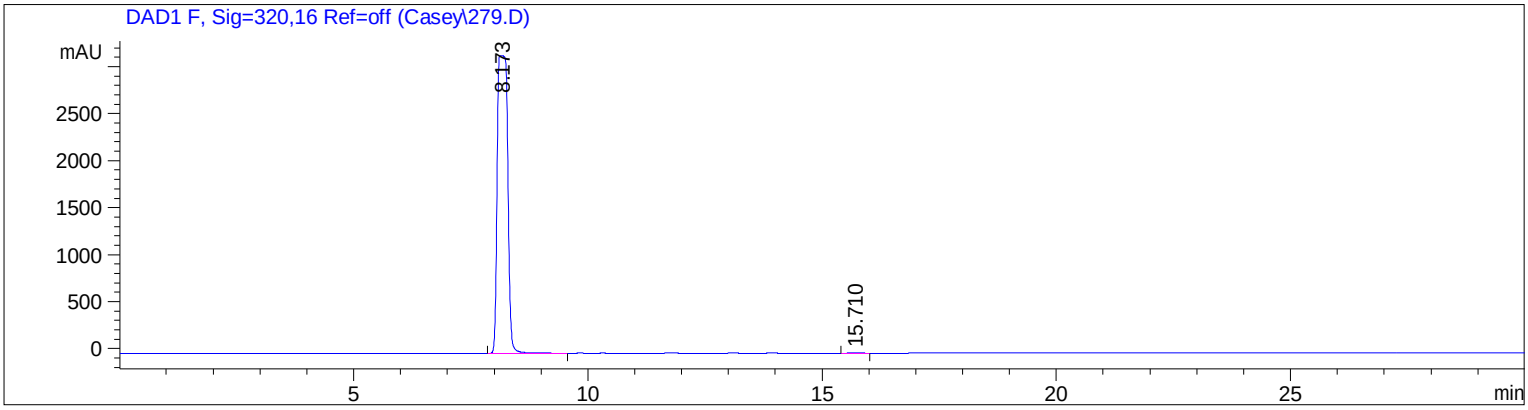

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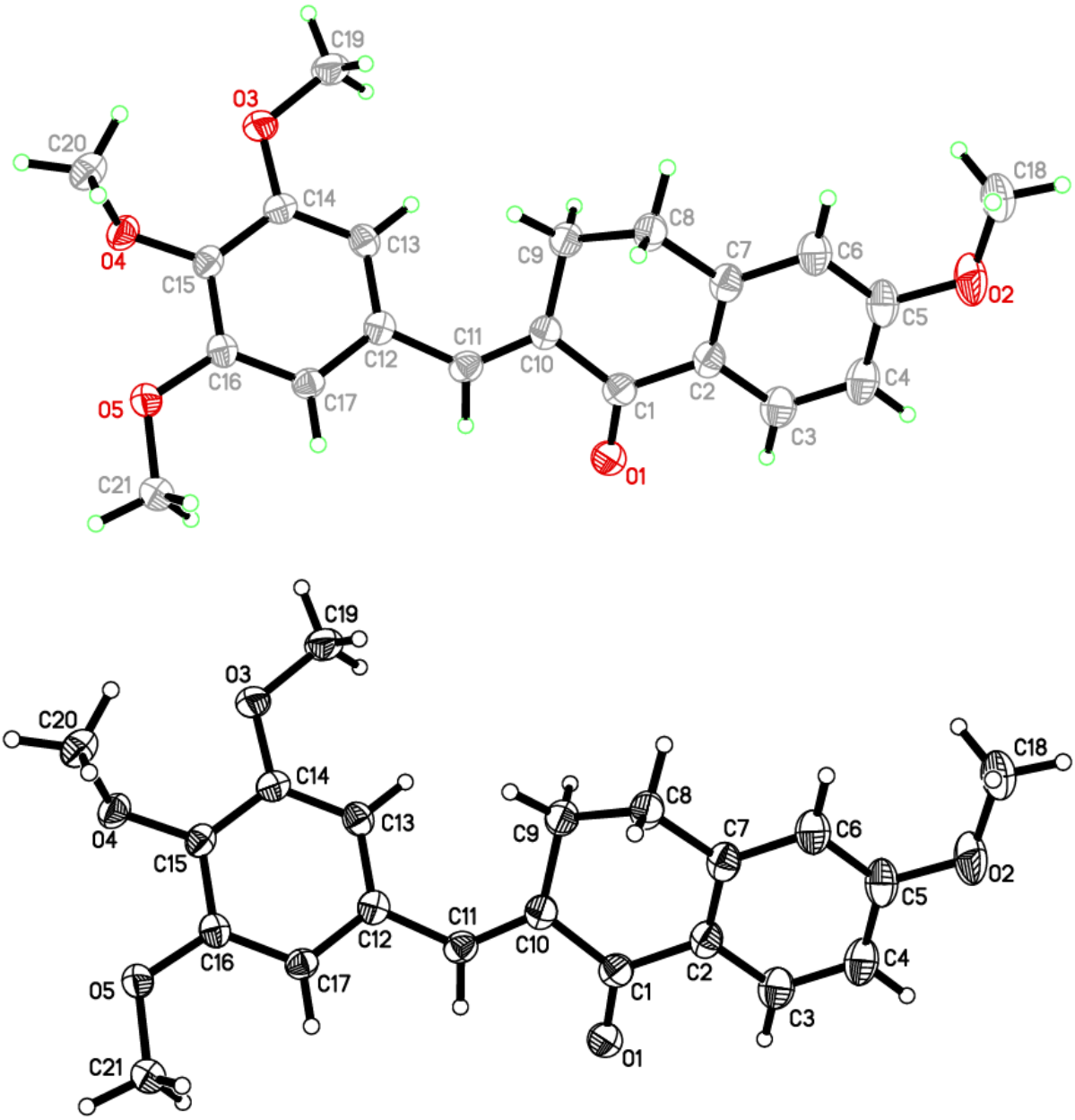

Compound 2a

X-ray crystal structure of **KGP146 (Compound 2a)**

X-ray Crystallographic Analysis:
X-ray crystallographic analysis of compound KGP146. Crystallographic data were collected on a crystal of KGP146 with dimensions 0.336 x 0.142 x 0.108 mm^3. Data were collected at 150(2) K on a Bruker X8 Apex using Mo KR radiation (λ = 0.71073 Å). The structure was solved by direct methods after correction of the data using SADABS. Crystallographic data and definement details for the complex mentioned herein is found in the Supporting

Information (Table S1-S5). All data were processed using the Bruker AXS SHELXTL software, version 6.10.

Table 1. Crystal data and structure refinement for KGP146.

Identification code	KGP146	
Empirical formula	C21 H22 O5	
Formula weight	354.38	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	Pbca	
Unit cell dimensions	a = 13.8321(7) Å	α= 90°.
	b = 8.4887(3) Å	β= 90°.
	c = 30.2097(13) Å	γ = 90°.
Volume	3547.1(3) $Å^3$	
Z	8	
Density (calculated)	1.327 Mg/m^3	
Absorption coefficient	0.094 mm^{-1}	
F(000)	1504	
Crystal size	0.336 x 0.142 x 0.108 mm^3	
Theta range for data collection	2.697 to 26.375°.	
Index ranges	-17<=h<=16, -10<=k<=10, -36<=l<=37	
Reflections collected	27657	
Independent reflections	3628 [R(int) = 0.0440]	
Completeness to theta = 25.242°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.959 and 0.938	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3628 / 0 / 239	
Goodness-of-fit on F^2	1.031	
Final R indices [I>2sigma(I)]	R1 = 0.0400, wR2 = 0.0906	
R indices (all data)	R1 = 0.0572, wR2 = 0.0990	

Extinction coefficient	n/a
Largest diff. peak and hole	0.223 and -0.160 e.Å^{-3}

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for KGP146. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	3898(1)	2374(1)	757(1)	37(1)
O(2)	2171(1)	5440(2)	-972(1)	45(1)
O(3)	-226(1)	-1688(1)	1941(1)	31(1)
O(4)	473(1)	-1332(1)	2774(1)	30(1)
O(5)	2006(1)	433(1)	2937(1)	28(1)
C(1)	3042(1)	2556(2)	659(1)	28(1)
C(2)	2774(1)	3345(2)	239(1)	28(1)
C(3)	3505(1)	3750(2)	-64(1)	36(1)
C(4)	3284(1)	4470(2)	-457(1)	39(1)
C(5)	2323(1)	4786(2)	-565(1)	36(1)
C(6)	1593(1)	4424(2)	-266(1)	34(1)
C(7)	1818(1)	3717(2)	139(1)	29(1)
C(8)	1039(1)	3434(2)	477(1)	32(1)
C(9)	1229(1)	1991(2)	765(1)	30(1)
C(10)	2246(1)	1970(2)	951(1)	26(1)
C(11)	2499(1)	1481(2)	1359(1)	26(1)
C(12)	1913(1)	798(2)	1715(1)	24(1)
C(13)	1080(1)	-93(2)	1637(1)	25(1)
C(14)	583(1)	-771(2)	1989(1)	24(1)
C(15)	908(1)	-573(2)	2423(1)	23(1)
C(16)	1739(1)	324(2)	2503(1)	23(1)
C(17)	2241(1)	998(2)	2151(1)	23(1)
C(18)	1204(1)	5825(2)	-1095(1)	40(1)
C(19)	-638(1)	-1774(2)	1508(1)	38(1)
C(20)	-456(1)	-720(2)	2898(1)	34(1)
C(21)	2832(1)	1379(2)	3036(1)	30(1)

Table 3. Bond lengths [Å] and angles [°] for KGP146.

O(1)-C(1)	1.2296(18)
O(2)-C(5)	1.3658(18)
O(2)-C(18)	1.427(2)
O(3)-C(14)	1.3704(17)
O(3)-C(19)	1.4279(18)
O(4)-C(15)	1.3783(16)
O(4)-C(20)	1.4349(18)
O(5)-C(16)	1.3647(17)
O(5)-C(21)	1.4291(17)
C(1)-C(2)	1.482(2)
C(1)-C(10)	1.497(2)
C(2)-C(7)	1.392(2)
C(2)-C(3)	1.407(2)
C(3)-C(4)	1.369(2)
C(4)-C(5)	1.396(2)
C(5)-C(6)	1.388(2)
C(6)-C(7)	1.397(2)
C(7)-C(8)	1.505(2)
C(8)-C(9)	1.526(2)
C(9)-C(10)	1.515(2)
C(10)-C(11)	1.346(2)
C(11)-C(12)	1.466(2)
C(12)-C(13)	1.3979(19)
C(12)-C(17)	1.403(2)
C(13)-C(14)	1.391(2)
C(14)-C(15)	1.397(2)
C(15)-C(16)	1.399(2)
C(16)-C(17)	1.392(2)
C(5)-O(2)-C(18)	118.08(12)
C(14)-O(3)-C(19)	116.81(11)
C(15)-O(4)-C(20)	114.97(11)
C(16)-O(5)-C(21)	117.15(11)
O(1)-C(1)-C(2)	120.22(13)

O(1)-C(1)-C(10)	121.66(13)
C(2)-C(1)-C(10)	118.11(13)
C(7)-C(2)-C(3)	119.04(14)
C(7)-C(2)-C(1)	121.77(13)
C(3)-C(2)-C(1)	119.19(14)
C(4)-C(3)-C(2)	120.87(16)
C(3)-C(4)-C(5)	120.03(15)
O(2)-C(5)-C(6)	124.36(15)
O(2)-C(5)-C(4)	115.75(14)
C(6)-C(5)-C(4)	119.89(15)
C(5)-C(6)-C(7)	120.16(15)
C(2)-C(7)-C(6)	119.95(14)
C(2)-C(7)-C(8)	119.75(13)
C(6)-C(7)-C(8)	120.23(14)
C(7)-C(8)-C(9)	113.07(13)
C(10)-C(9)-C(8)	112.44(12)
C(11)-C(10)-C(1)	116.77(13)
C(11)-C(10)-C(9)	125.86(13)
C(1)-C(10)-C(9)	117.37(12)
C(10)-C(11)-C(12)	130.44(14)
C(13)-C(12)-C(17)	119.29(13)
C(13)-C(12)-C(11)	123.13(13)
C(17)-C(12)-C(11)	117.51(13)
C(14)-C(13)-C(12)	120.21(13)
O(3)-C(14)-C(13)	123.94(13)
O(3)-C(14)-C(15)	115.48(12)
C(13)-C(14)-C(15)	120.57(13)
O(4)-C(15)-C(14)	121.65(12)
O(4)-C(15)-C(16)	118.78(12)
C(14)-C(15)-C(16)	119.40(12)
O(5)-C(16)-C(17)	124.77(13)
O(5)-C(16)-C(15)	115.06(12)
C(17)-C(16)-C(15)	120.16(13)
C(16)-C(17)-C(12)	120.37(13)

Table 4. Anisotropic displacement parameters ($Å^2 \times 10^3$) for KGP146. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	29(1)	54(1)	29(1)	2(1)	1(1)	-5(1)
O(2)	56(1)	56(1)	25(1)	10(1)	11(1)	9(1)
O(3)	28(1)	32(1)	32(1)	4(1)	-4(1)	-8(1)
O(4)	24(1)	33(1)	32(1)	11(1)	2(1)	1(1)
O(5)	30(1)	30(1)	23(1)	3(1)	-2(1)	-5(1)
C(1)	31(1)	28(1)	25(1)	-6(1)	2(1)	-3(1)
C(2)	36(1)	26(1)	22(1)	-5(1)	5(1)	-3(1)
C(3)	37(1)	41(1)	29(1)	-2(1)	9(1)	2(1)
C(4)	45(1)	46(1)	27(1)	2(1)	15(1)	3(1)
C(5)	51(1)	34(1)	23(1)	1(1)	9(1)	6(1)
C(6)	41(1)	34(1)	27(1)	0(1)	5(1)	4(1)
C(7)	36(1)	26(1)	24(1)	-3(1)	5(1)	-1(1)
C(8)	31(1)	34(1)	30(1)	5(1)	2(1)	0(1)
C(9)	31(1)	30(1)	28(1)	2(1)	2(1)	-2(1)
C(10)	30(1)	23(1)	25(1)	-2(1)	2(1)	-2(1)
C(11)	26(1)	25(1)	28(1)	-3(1)	2(1)	-2(1)
C(12)	26(1)	20(1)	26(1)	1(1)	2(1)	3(1)
C(13)	29(1)	22(1)	24(1)	1(1)	-2(1)	2(1)
C(14)	22(1)	20(1)	31(1)	2(1)	-1(1)	1(1)
C(15)	22(1)	21(1)	26(1)	5(1)	3(1)	3(1)
C(16)	25(1)	21(1)	23(1)	2(1)	0(1)	5(1)
C(17)	22(1)	21(1)	27(1)	1(1)	0(1)	0(1)
C(18)	53(1)	42(1)	25(1)	4(1)	2(1)	3(1)
C(19)	30(1)	51(1)	32(1)	-8(1)	1(1)	-12(1)
C(20)	28(1)	38(1)	36(1)	2(1)	7(1)	-2(1)
C(21)	31(1)	33(1)	26(1)	-1(1)	-2(1)	-5(1)

284_PROTON_001
284

^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.16 (d, *J* = 8.7 Hz, 1H), 7.75 (s, 1H), 6.93 (d, *J* = 8.7 Hz, 1H), 6.66 (s, 2H), 3.99 (s, 3H), 3.89 (d, *J* = 5.0 Hz, 9H), 3.18 – 3.14 (m, 2H), 3.09 (t, *J* = 6.2 Hz, 2H).

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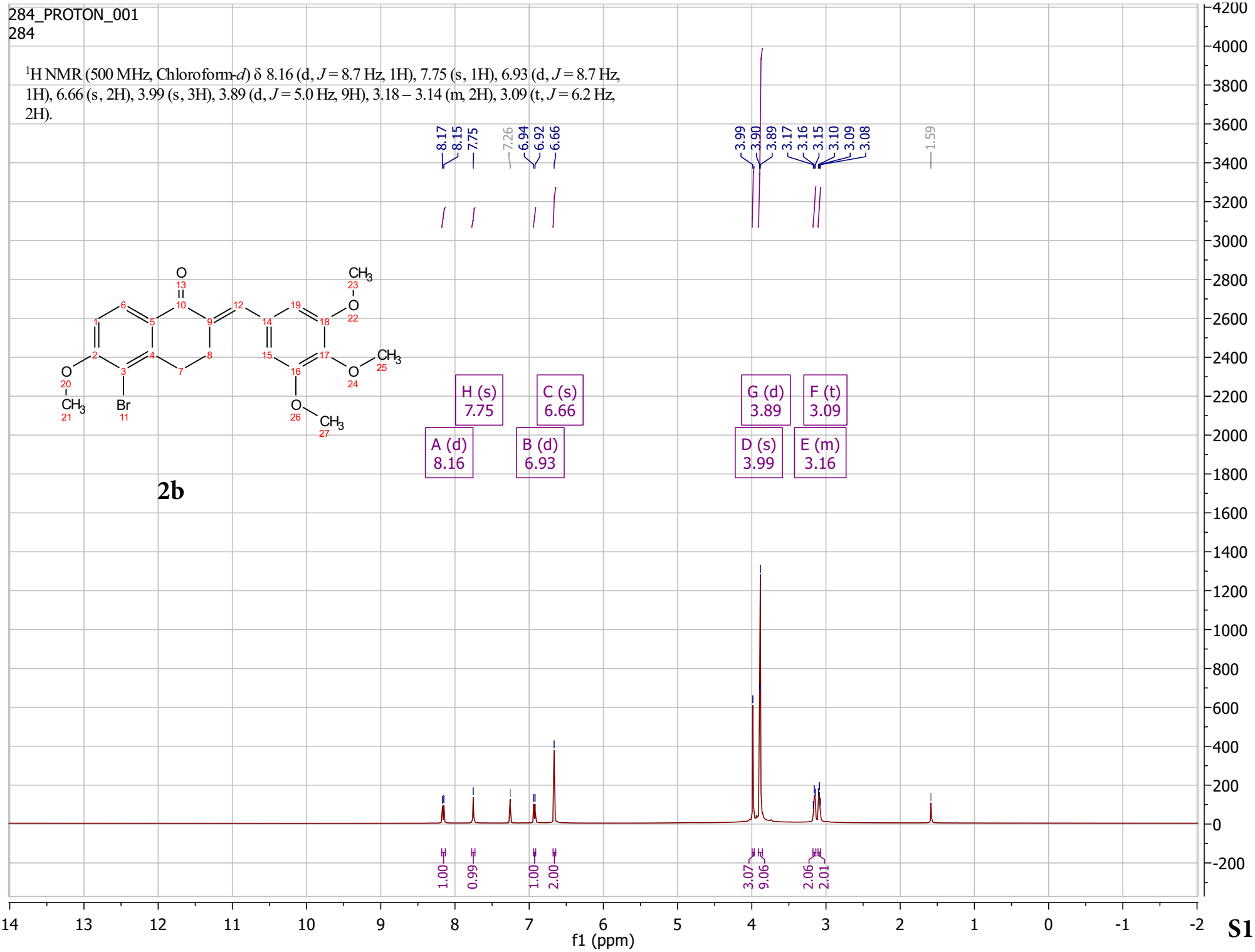

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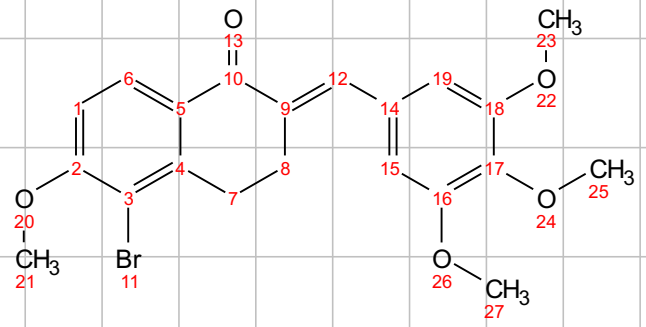
^{13}C NMR (126 MHz, cdcl$_3$) δ 186.06, 159.51, 152.88, 144.02, 136.50, 133.68, 131.00, 129.30, 129.27, 128.07, 109.80, 107.00, 77.08, 76.82, 76.57, 60.77, 56.33, 55.99, 28.83, 26.48.



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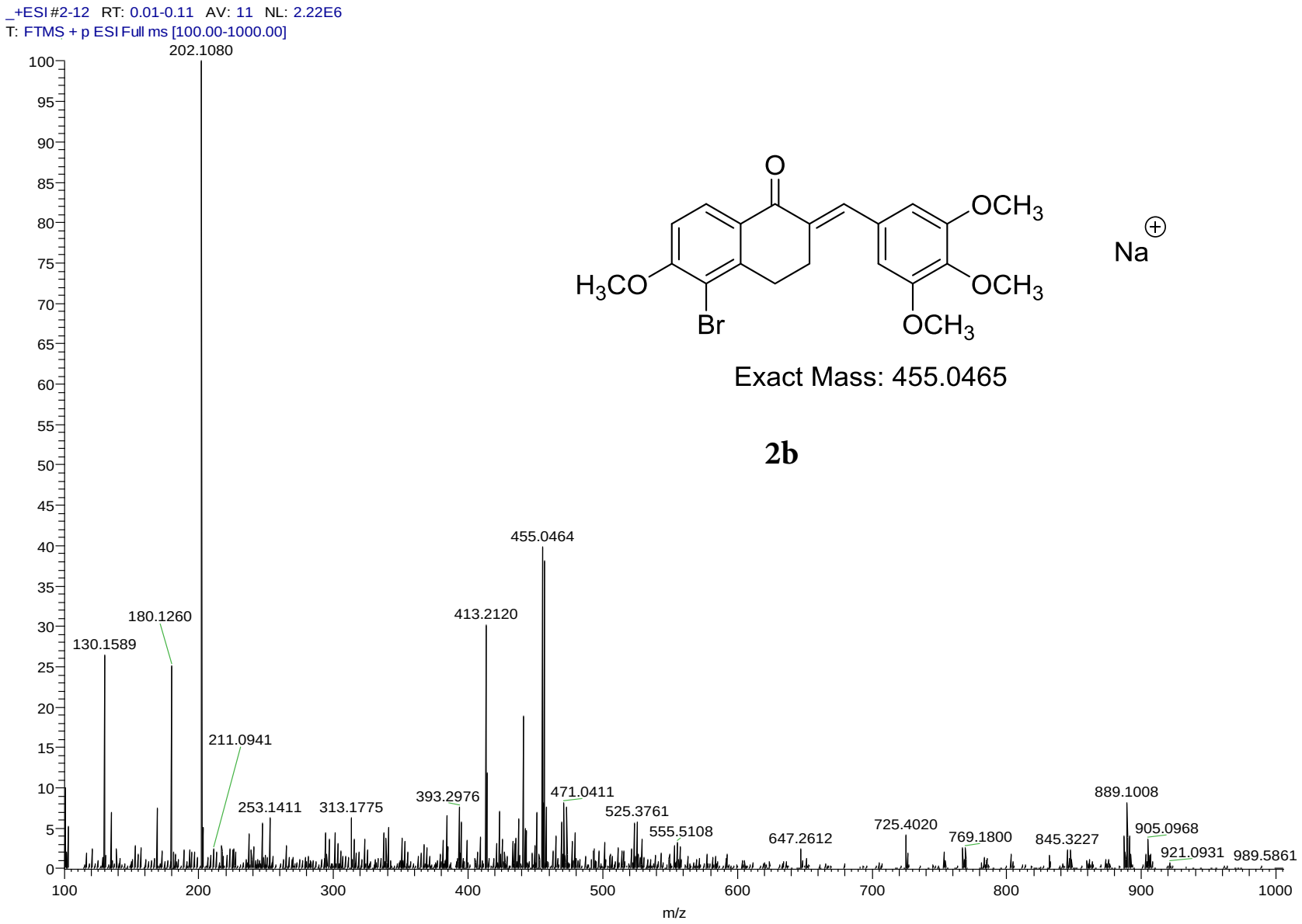

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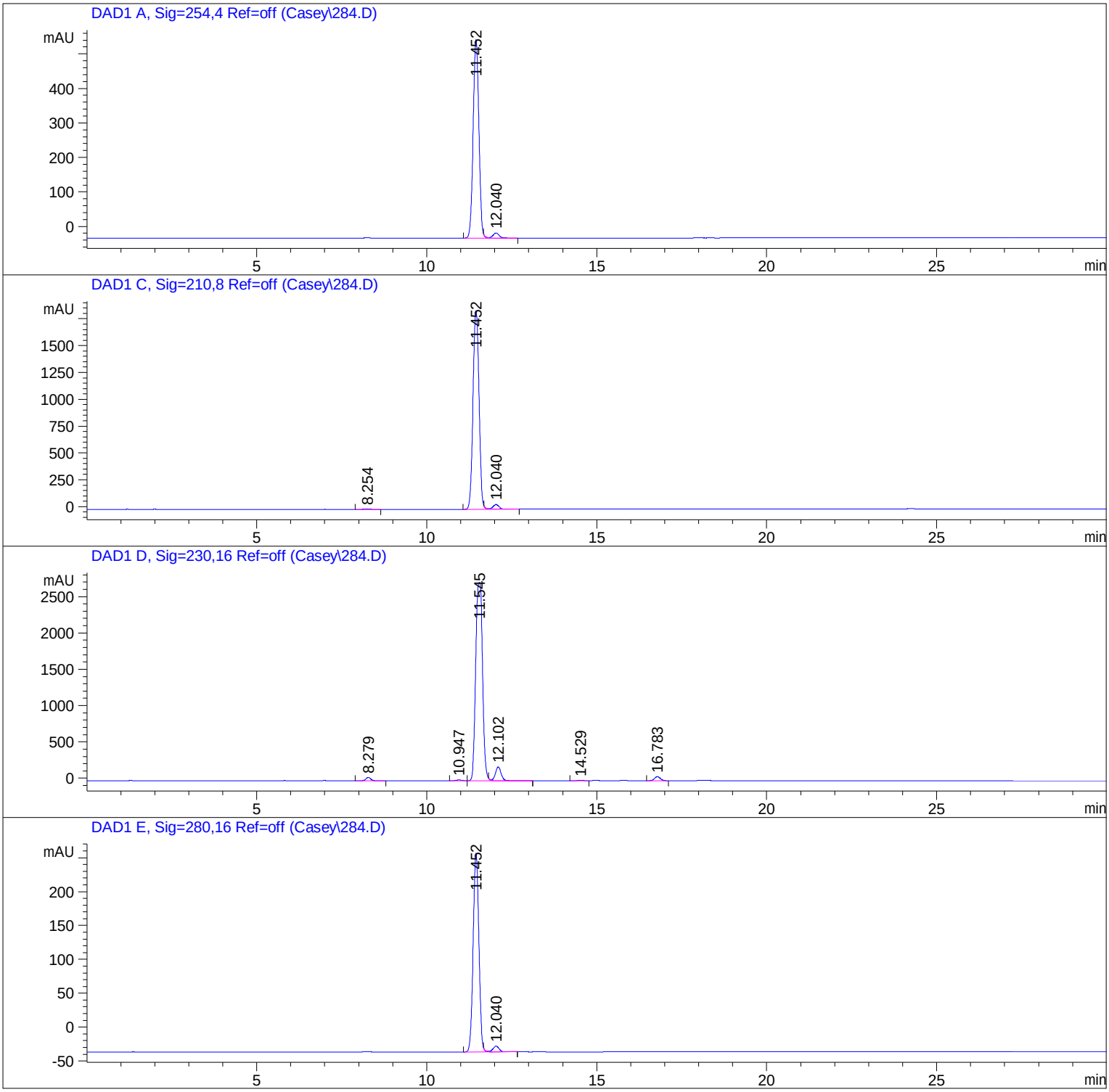

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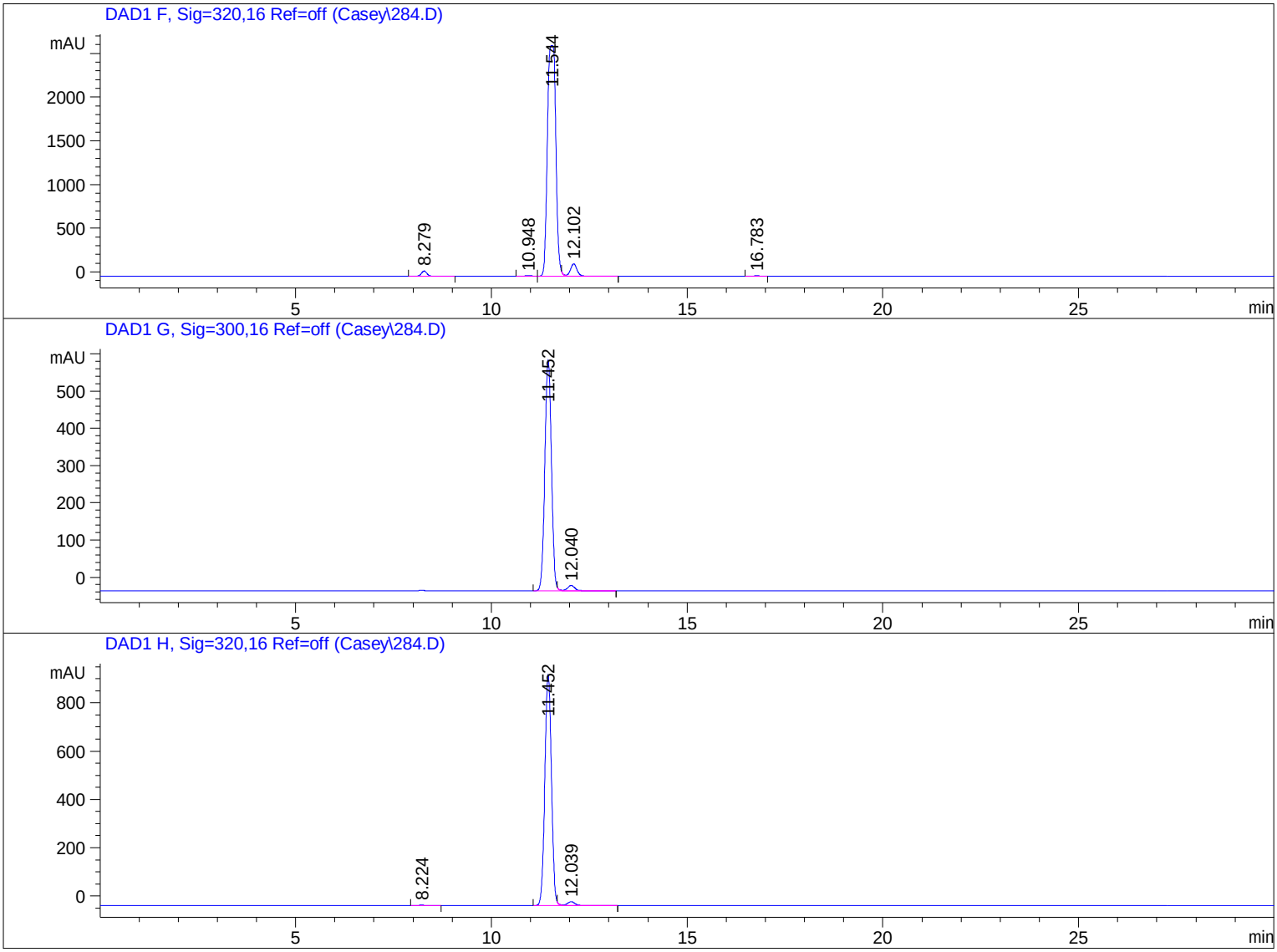

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Data File C:\Chem32\1\Data\Casey\284.D
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Signal 2: DAD1 C, Sig=210,8 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.254	BB	0.1906	56.62222	4.63146	0.2443
2	11.452	BV R	0.1944	2.25533e4	1847.64990	97.3096
3	12.040	VB E	0.1971	566.93726	43.76442	2.4461
Totals :				2.31768e4	1896.04578	

Signal 3: DAD1 D, Sig=230,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.279	BB	0.1518	436.85202	44.79024	1.0541
2	10.947	BV	0.1671	104.87866	9.78327	0.2531
3	11.545	VV R	0.2245	3.80441e4	2731.06079	91.7993
4	12.102	VB E	0.1782	2174.25171	188.96416	5.2464
5	14.529	BV	0.2110	34.07227	2.37991	0.0822
6	16.783	BB	0.1819	648.55060	55.69127	1.5649
Totals :				4.14427e4	3032.66964	

Signal 4: DAD1 E, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.452	BV R	0.1797	3404.77197	292.80515	96.9347
2	12.040	VB E	0.1938	107.66509	8.50091	3.0653
Totals :				3512.43707	301.30606	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.279	BB	0.1504	563.29327	58.44865	1.3541
2	10.948	BV	0.1689	68.31553	6.28307	0.1642
3	11.544	VV R	0.2398	3.92923e4	2637.03027	94.4575
4	12.102	VB E	0.1806	1636.07031	139.75050	3.9331
5	16.783	BB	0.1817	37.88853	3.25761	0.0911
Totals :				4.15979e4	2844.77010	

Data File C:\Chem32\1\Data\Casey\284.D
Sample Name: 284

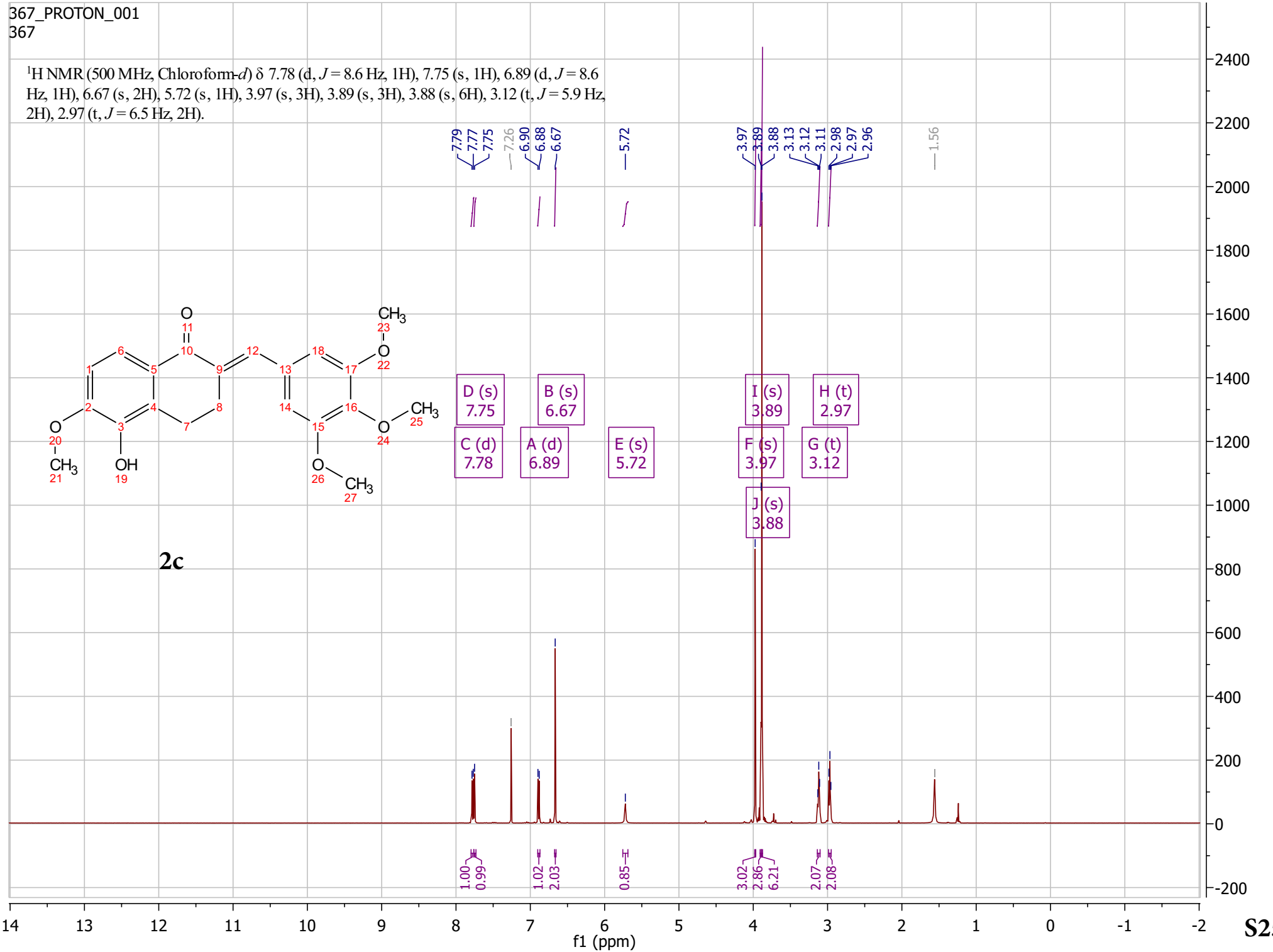
Signal 6: DAD1 G, Sig=300,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.452	BV R	0.1798	7214.64307	619.85413	97.4662
2	12.040	VB E	0.2012	187.55479	14.10318	2.5338
Totals :				7402.19786	633.95730	

Signal 7: DAD1 H, Sig=320,16 Ref=off

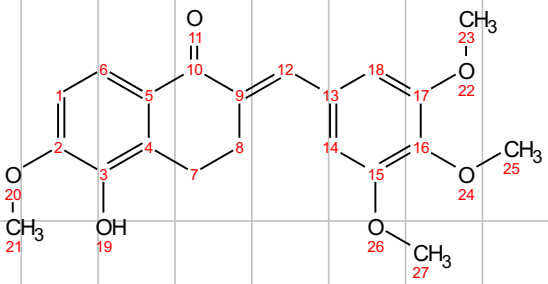
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.224	BB	0.1749	14.28738	1.27359	0.1256
2	11.452	BV R	0.1840	1.11393e4	955.79541	97.9331
3	12.039	VB E	0.2092	220.81247	15.78399	1.9413
Totals :				1.13744e4	972.85299	

*** End of Report ***


367_PROTON_001
367
[1]H NMR (500 MHz, Chloroform-d) δ 7.78 (d, J = 8.6 Hz, 1H), 7.75 (s, 1H), 6.89 (d, J = 8.6 Hz, 1H), 6.67 (s, 2H), 5.72 (s, 1H), 3.97 (s, 3H), 3.89 (s, 3H), 3.88 (s, 6H), 3.12 (t, J = 5.9 Hz, 2H), 2.97 (t, J = 6.5 Hz, 2H).
7.79
7.77
7.75
7.26
6.90
6.88
6.67
5.72
3.97
3.89
3.88
3.13
3.12
3.11
2.98
2.97
2.96
1.56
D (s) 7.75
B (s) 6.67
I (s) 3.89
H (t) 2.97
C (d) 7.78
A (d) 6.89
E (s) 5.72
F (s) 3.97
G (t) 3.12
J (s) 3.88
2c
1.00
0.99
1.02
2.03
0.85
3.02
2.86
6.21
2.07
2.08
f1 (ppm)

367_CARBON_001
367

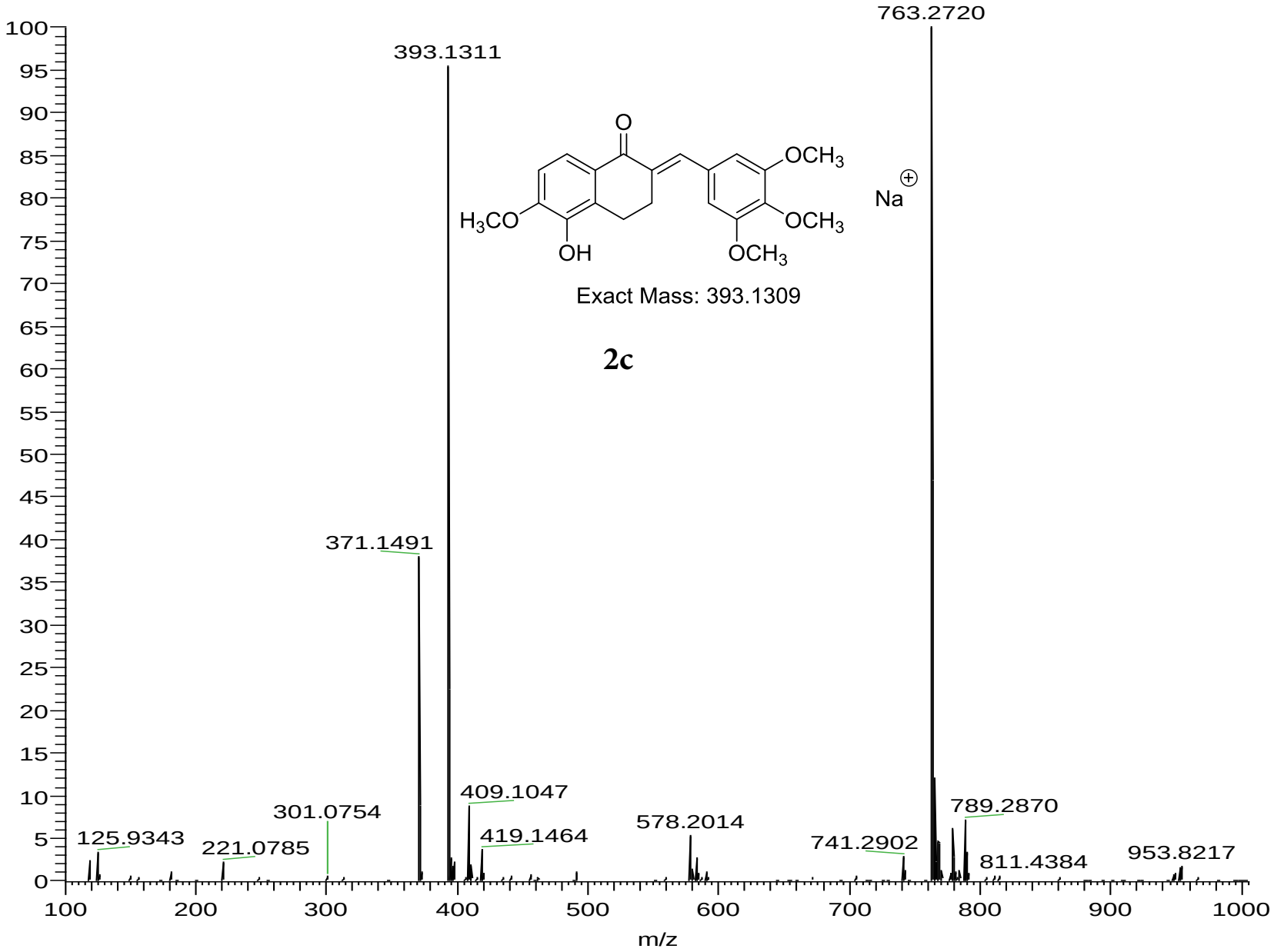
^{13}C NMR (126 MHz, cdcl$_3$) δ 187.20, 153.04, 141.48, 136.18, 135.19, 135.15, 131.51, 129.07, 121.19, 116.27, 115.26, 109.99, 108.72, 107.16, 60.97, 56.18, 56.09, 26.74, 21.62.



2c

CJM-VI-367

365_180723122610 #2-14 RT: 0.01-0.11 AV: 13 NL: 3.69E7
T: FTMS + p ESI Full ms [100.00-1000.00]



Data File C:\Chem32\1\Data\Casey\CJM-VI-367.D
Sample Name: CJM-VI-367

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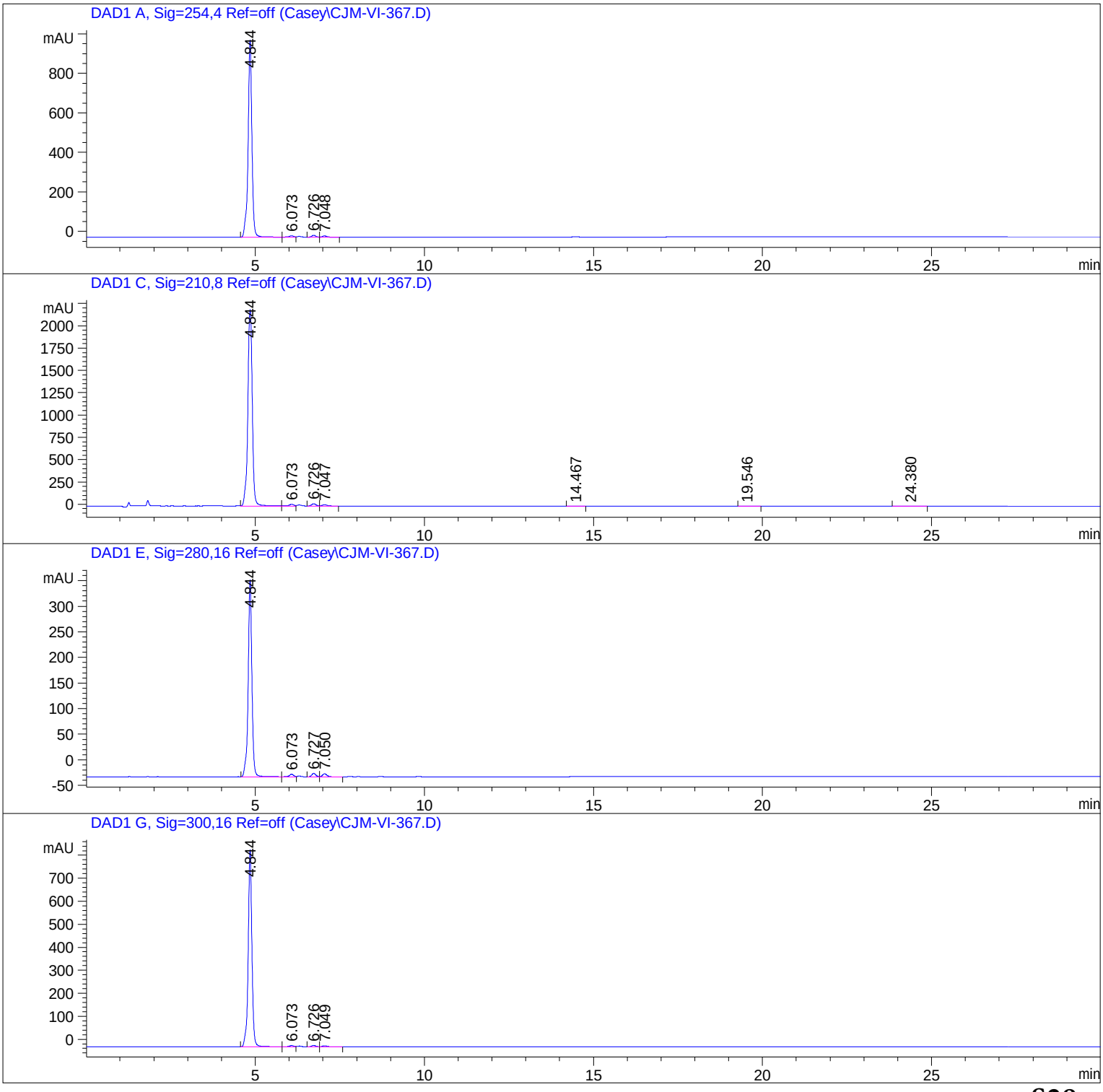
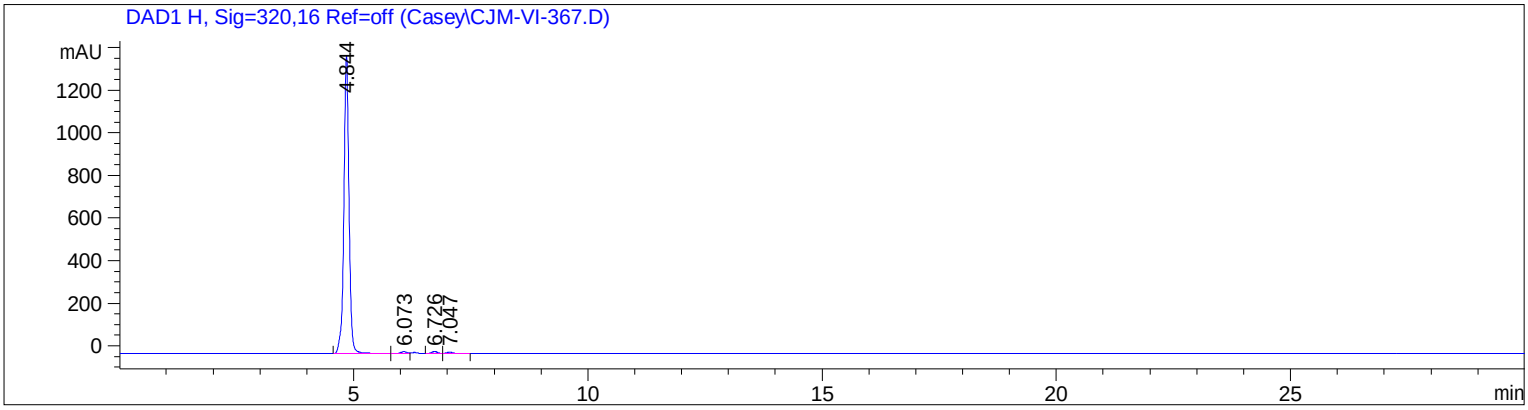
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : 1200 HPLC Location : 1
Injection Date : 7/25/2018 11:12:57 AM
Inj Volume : No inj
Acq. Method : C:\CHEM32\1\METHODS\GRAD 2 50-90 ACN.M
Last changed : 4/30/2014 1:53:57 AM by ERICAP
Analysis Method : C:\CHEM32\1\METHODS\RT-ACNWASH 2.M
Last changed : 7/9/2015 2:27:22 PM by Blake
Method Info : General Column Wash Method

Sample Info : GRAD 2 50-90

Additional Info : Peak(s) manually integrated



Data File C:\Chem32\1\Data\Casey\CJM-VI-367.D
Sample Name: CJM-VI-367



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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	4.844	BB	0.1214	8003.51855	1000.01160	97.4634
2	6.073	BV	0.1425	68.04012	7.05827	0.8286
3	6.726	BV	0.1325	78.62694	9.12359	0.9575
4	7.048	VB	0.1480	61.63052	6.42145	0.7505
Totals :				8211.81613	1022.61490	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.844	VV	0.1446	2.04755e4	2200.29761	96.9145
2	6.073	VV	0.1484	194.10883	19.46047	0.9188
3	6.726	BV	0.1327	226.82710	26.29191	1.0736
4	7.047	VB	0.1500	147.20825	15.06602	0.6968
5	14.467	BB	0.1887	23.60516	1.93033	0.1117
6	19.546	BB	0.2128	19.53611	1.45355	0.0925
7	24.380	BB	0.2642	40.60459	2.31945	0.1922
Totals :				2.11274e4	2266.81934	

Data File C:\Chem32\1\Data\Casey\CJM-VI-367.D
Sample Name: CJM-VI-367

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.844	BB	0.1197	3031.34229	385.61246	94.9974
2	6.073	BV	0.1359	44.51842	4.99980	1.3951
3	6.727	BV	0.1348	57.93388	6.70839	1.8156
4	7.050	VB	0.1458	57.17899	5.96761	1.7919
Totals :				3190.97358	403.28825	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.844	BB	0.1190	6701.68164	858.98492	97.7904
2	6.073	BV	0.1361	47.96641	5.37614	0.6999
3	6.726	BV	0.1346	55.47385	6.43208	0.8095
4	7.049	VB	0.1476	47.98608	4.92705	0.7002
Totals :				6853.10798	875.72019	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.844	BB	0.1188	1.08993e4	1399.92578	98.0340
2	6.073	BV	0.1334	73.64749	8.47660	0.6624
3	6.726	BV	0.1326	83.72591	9.71457	0.7531
4	7.047	VB	0.1480	61.20546	6.37443	0.5505
Totals :				1.11179e4	1424.49139	

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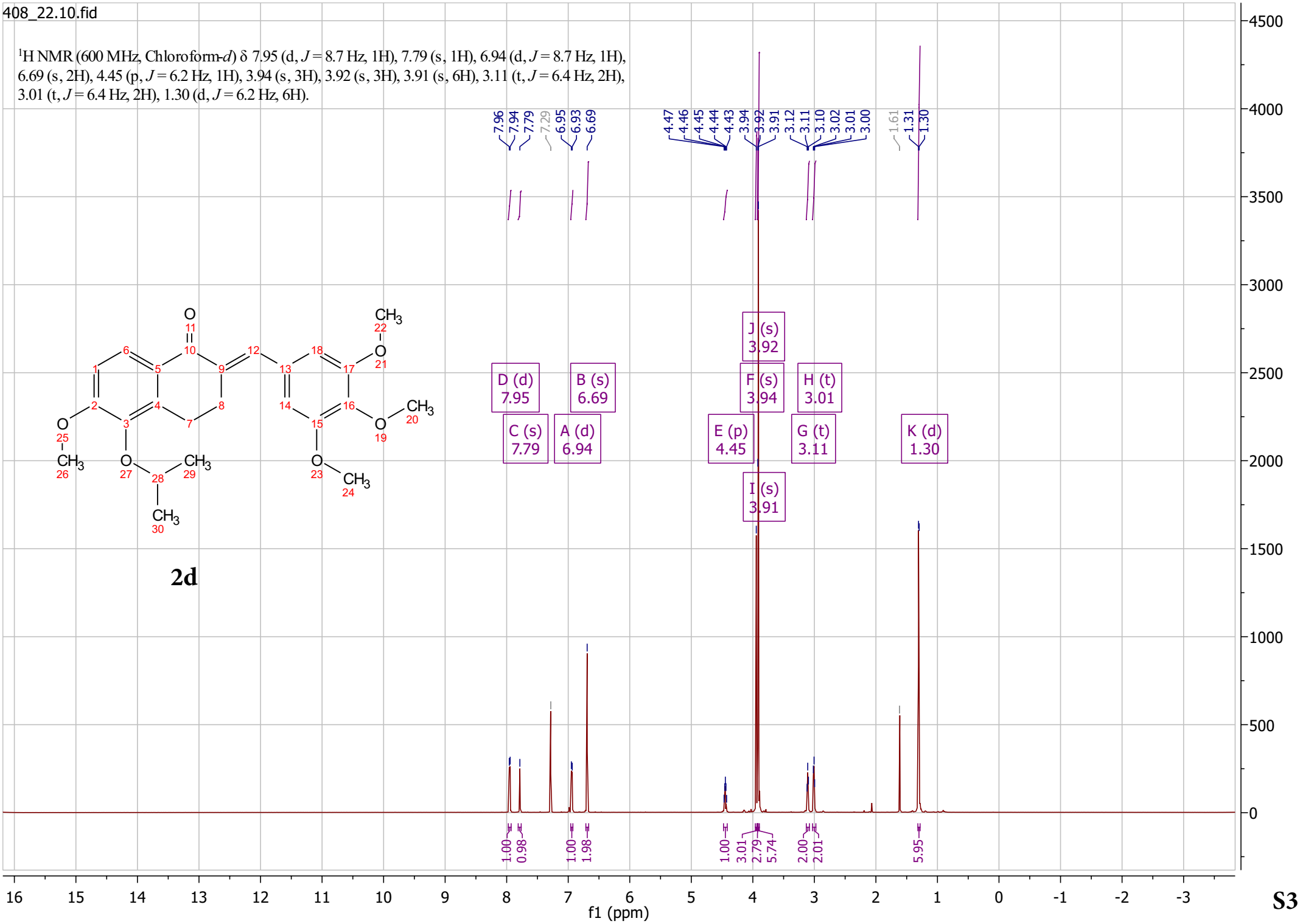
*** End of Report ***

408_22.10.fid

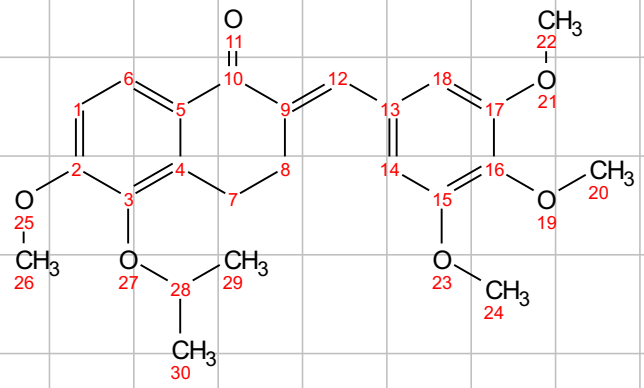
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.95 (d, *J* = 8.7 Hz, 1H), 7.79 (s, 1H), 6.94 (d, *J* = 8.7 Hz, 1H), 6.69 (s, 2H), 4.45 (p, *J* = 6.2 Hz, 1H), 3.94 (s, 3H), 3.92 (s, 3H), 3.91 (s, 6H), 3.11 (t, *J* = 6.4 Hz, 2H), 3.01 (t, *J* = 6.4 Hz, 2H), 1.30 (d, *J* = 6.2 Hz, 6H).

2d



408.10.fid

^{13}C NMR (151 MHz, $CDCl_3$) δ 187.09, 157.01, 153.06, 142.78, 138.48, 137.91, 136.09, 135.09, 131.52, 127.44, 125.15, 110.29, 107.24, 74.87, 60.96, 56.21, 55.75, 27.07, 22.96, 22.55.

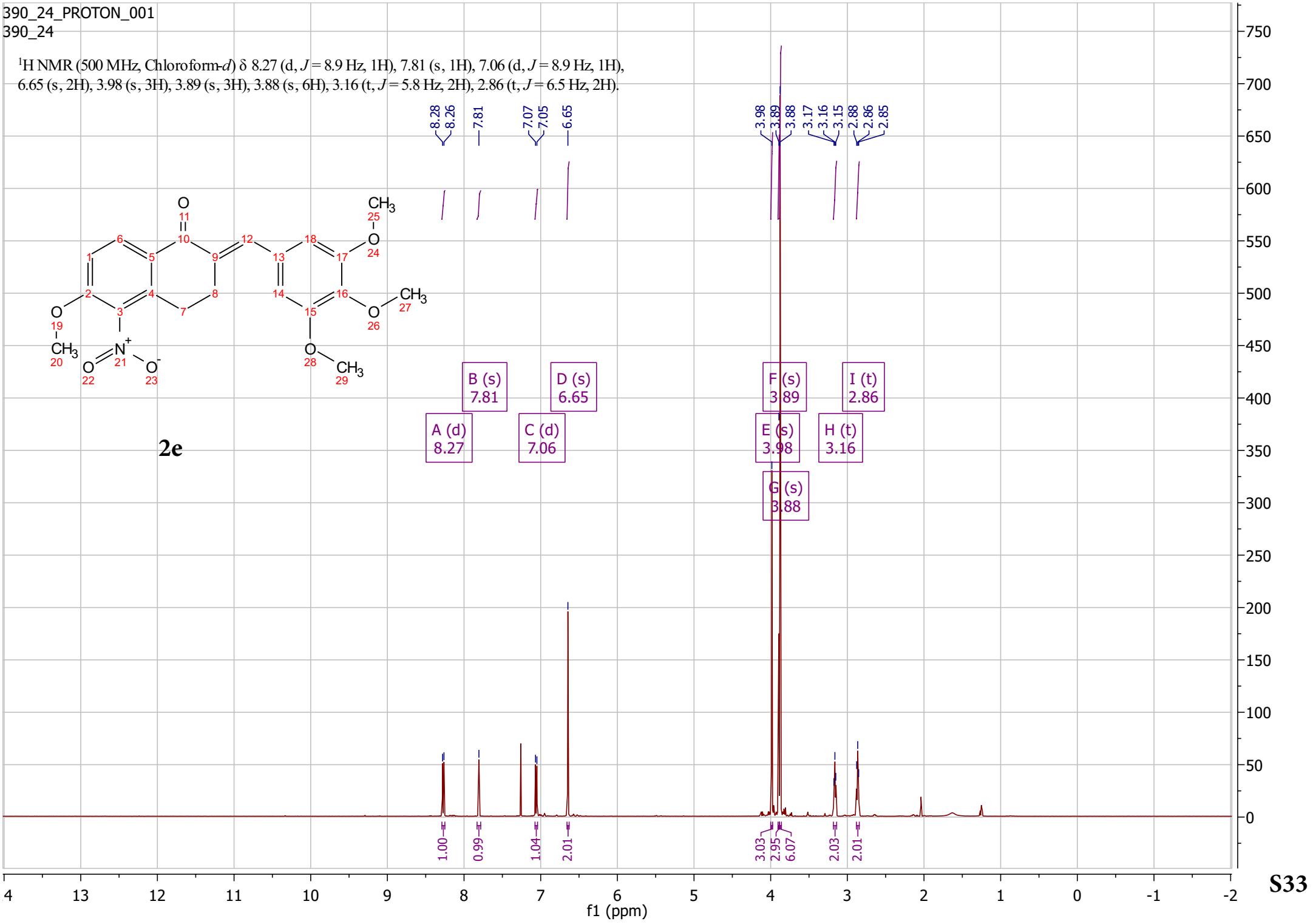


2d

390_24_PROTON_001
390_24

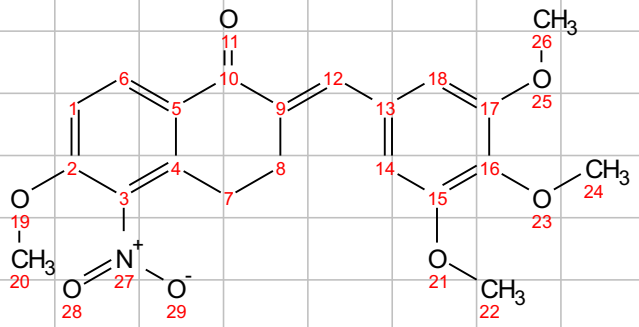
^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.27 (d, *J* = 8.9 Hz, 1H), 7.81 (s, 1H), 7.06 (d, *J* = 8.9 Hz, 1H), 6.65 (s, 2H), 3.98 (s, 3H), 3.89 (s, 3H), 3.88 (s, 6H), 3.16 (t, *J* = 5.8 Hz, 2H), 2.86 (t, *J* = 6.5 Hz, 2H).

2e

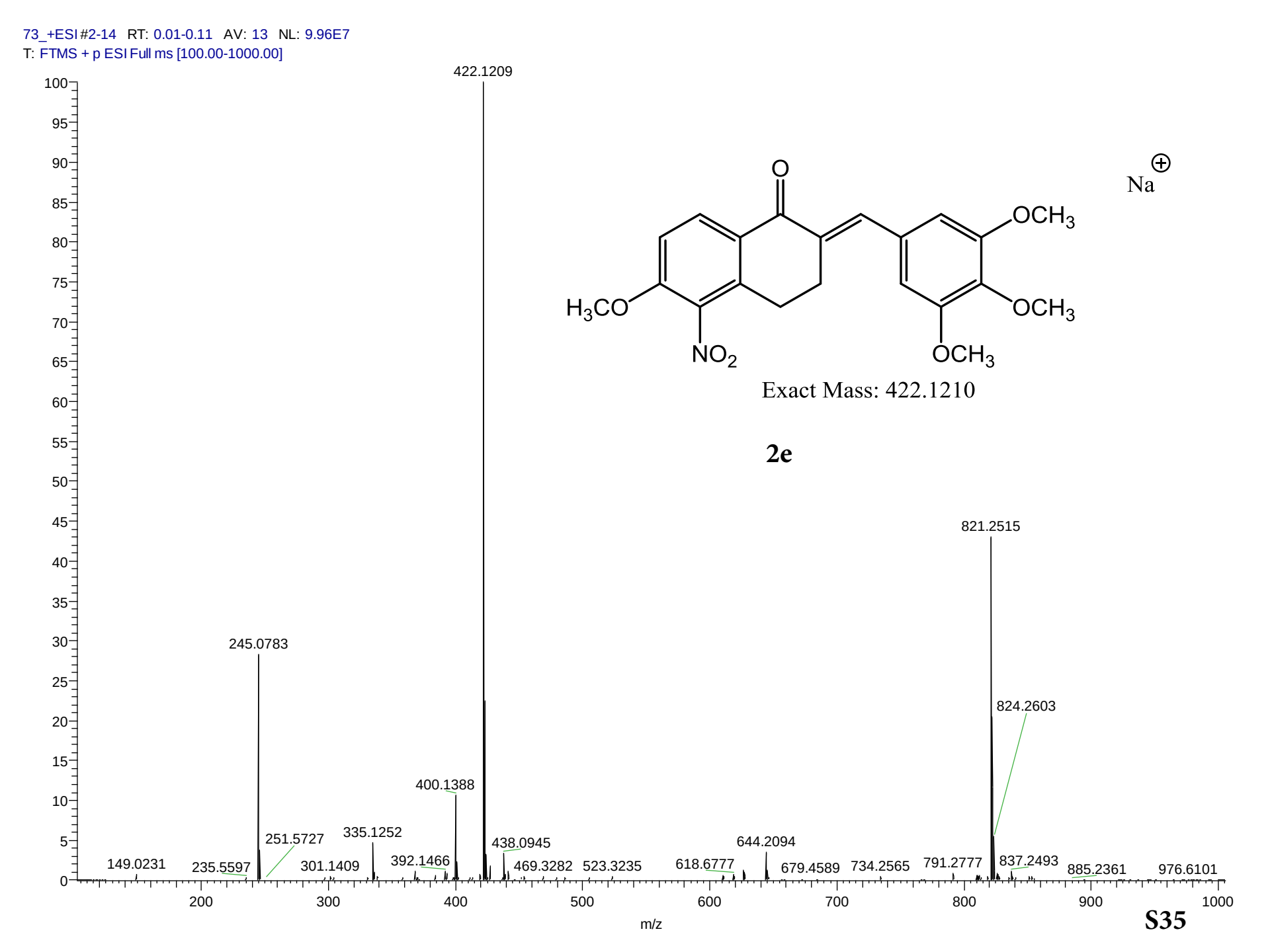


JWF-V-73.10.fid

^{13}C NMR (151 MHz, $CDCl_3$) δ 185.10, 154.28, 153.18, 139.81, 138.98, 137.99, 136.04, 132.99, 132.12, 130.79, 126.89, 110.94, 107.33, 61.00, 56.70, 56.24, 26.29, 23.76.



2e


73_+ESI #2-14 RT: 0.01-0.11 AV: 13 NL: 9.96E7
T: FTMS + p ESI Full ms [100.00-1000.00]
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821.2515
245.0783
824.2603
400.1388
251.5727
335.1252
438.0945
644.2094
149.0231
235.5597
301.1409
392.1466
469.3282
523.3235
618.6777
679.4589
734.2565
791.2777
837.2493
885.2361
976.6101
m/z
O
OCH3
H3CO
OCH3
NO2
OCH3
Na
Exact Mass: 422.1210
2e

Data File C:\Chem32\1\Data\Jake\JWF-V-73.D
Sample Name: JWF-V-73

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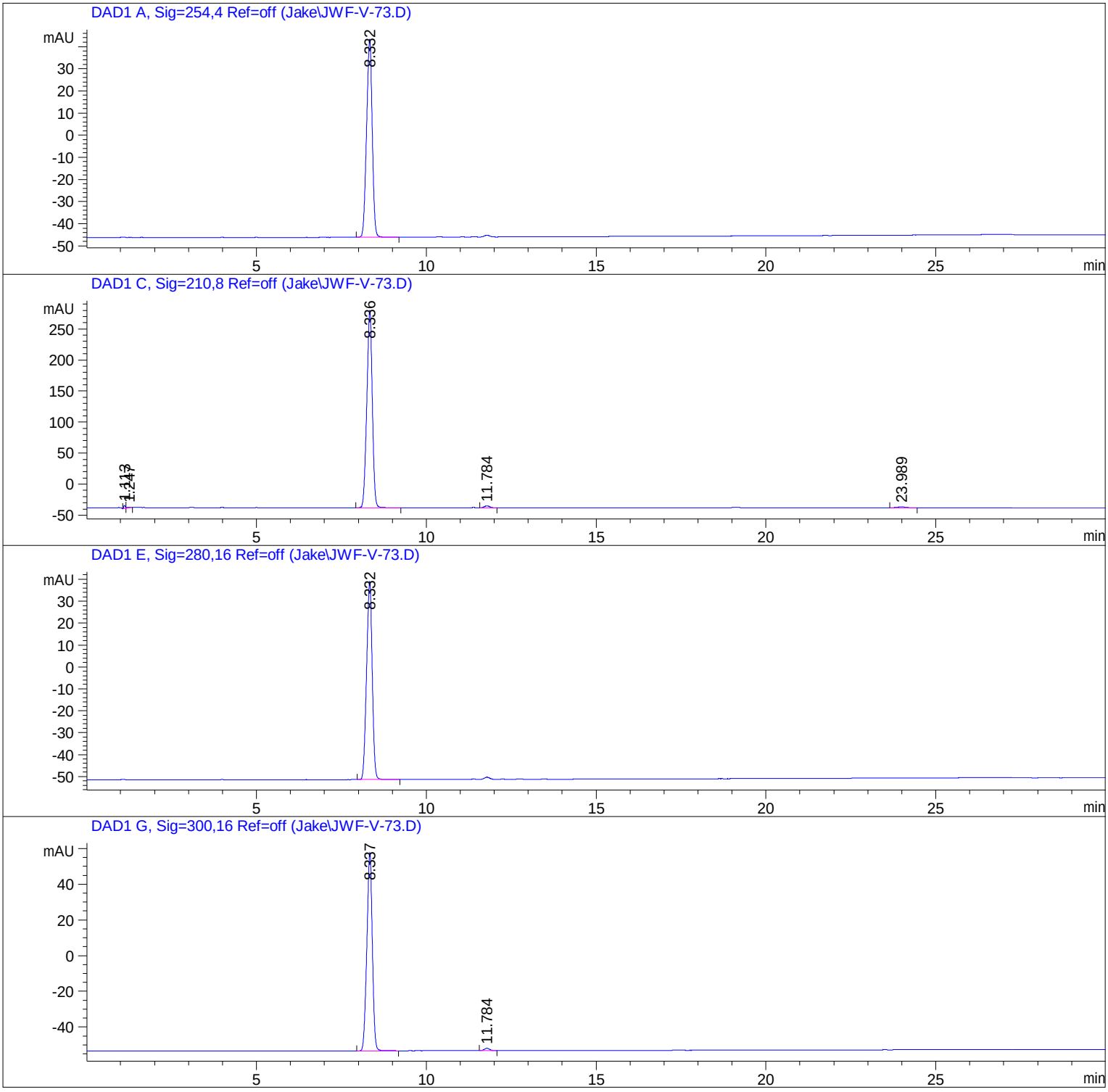
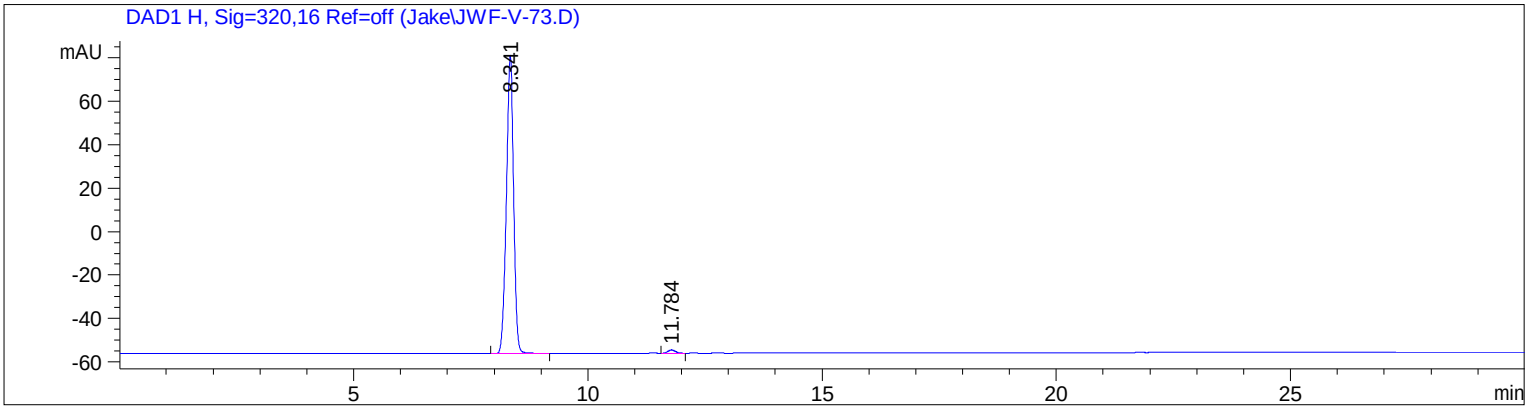
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : 1200 HPLC Location : 1
Injection Date : 9/14/2018 12:01:27 PM
Inj Volume : No inj
Method : C:\CHEM32\1\METHODS\GRAD 2 50-90 ACN.M
Last changed : 4/30/2014 1:53:57 AM by ERICAP



Data File C:\Chem32\1\Data\Jake\JWF-V-73.D
Sample Name: JWF-V-73



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Area Percent Report

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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	8.332	BB	0.1812	1037.73804	89.51611	100.0000
Totals :				1037.73804	89.51611	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.113	BV	0.0491	16.74559	5.25913	0.4564
2	1.247	VB	0.1094	11.86115	1.42085	0.3233
3	8.336	BB	0.1716	3584.98145	317.74329	97.7068
4	11.784	BB	0.1704	33.99076	3.13785	0.9264
5	23.989	BB	0.2392	21.54188	1.35722	0.5871
Totals :				3669.12083	328.91834	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.332	BB	0.1810	1046.46753	90.45219	100.0000
Totals :				1046.46753	90.45219	

Data File C:\Chem32\1\Data\Jake\JWF-V-73.D
Sample Name: JWF-V-73

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.337	BB	0.1708	1244.67322	111.00800	98.8331
2	11.784	BB	0.1674	14.69588	1.34616	1.1669
Totals :				1259.36910	112.35415	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.341	BB	0.1636	1476.06409	137.14726	98.8889
2	11.784	BB	0.1693	16.58478	1.52027	1.1111
Totals :				1492.64886	138.66754	

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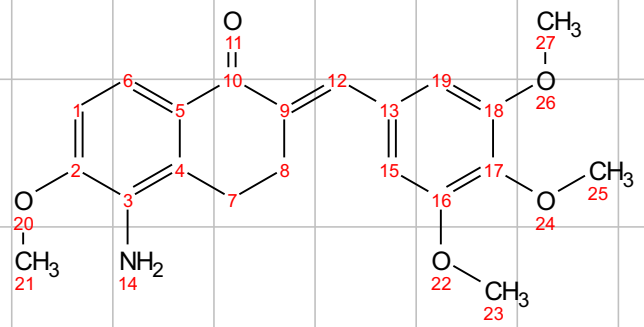
*** End of Report ***

JWF-81.11.fid

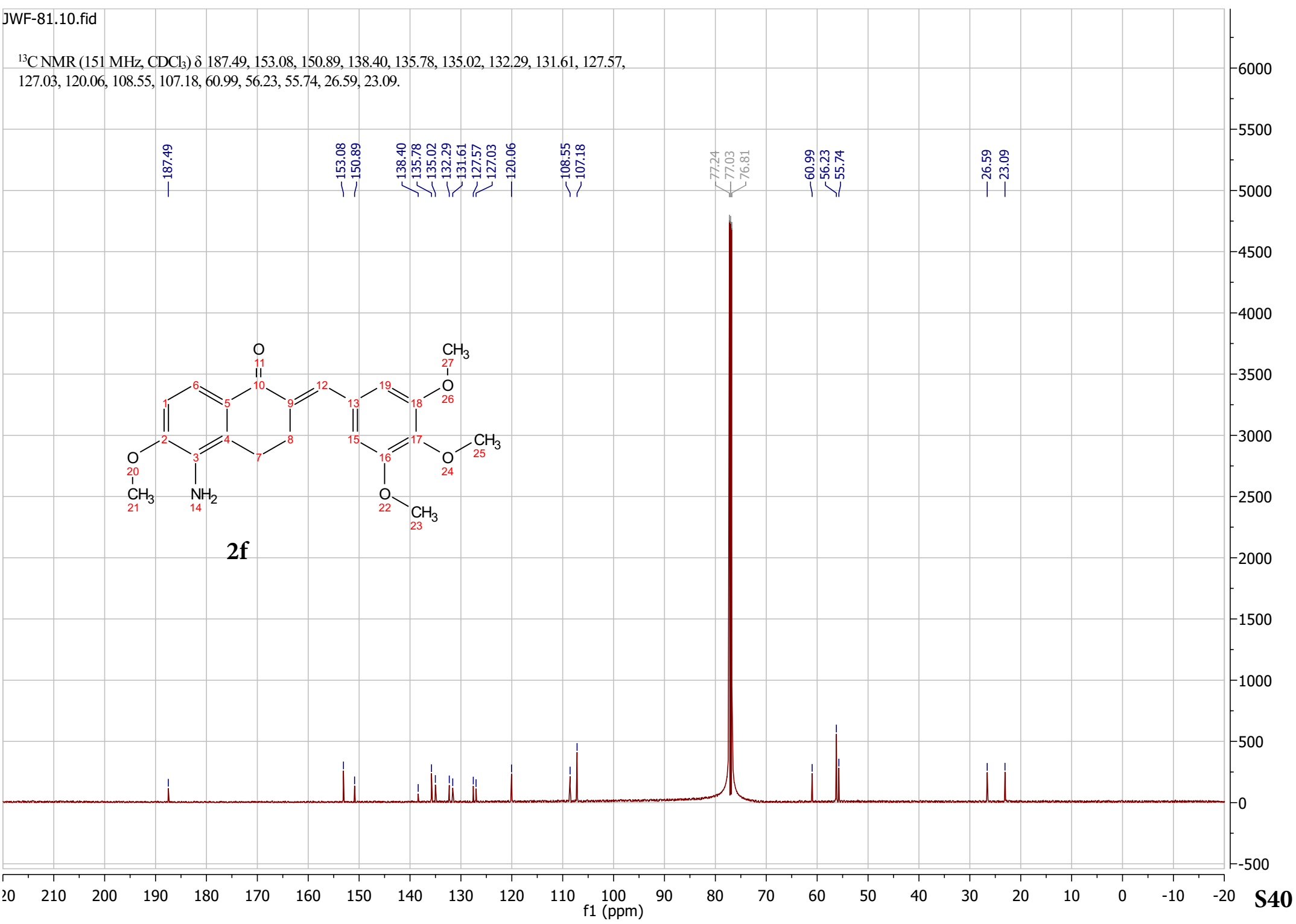
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.75 (d, *J* = 8.5 Hz, 2H), 6.88 (d, *J* = 8.6 Hz, 1H), 6.68 (s, 2H), 3.97 (s, 3H), 3.92 (s, 3H), 3.91 (s, 6H), 3.84 (s, 2H), 3.18 (t, *J* = 6.5 Hz, 2H), 2.78 (t, *J* = 6.5 Hz, 2H).



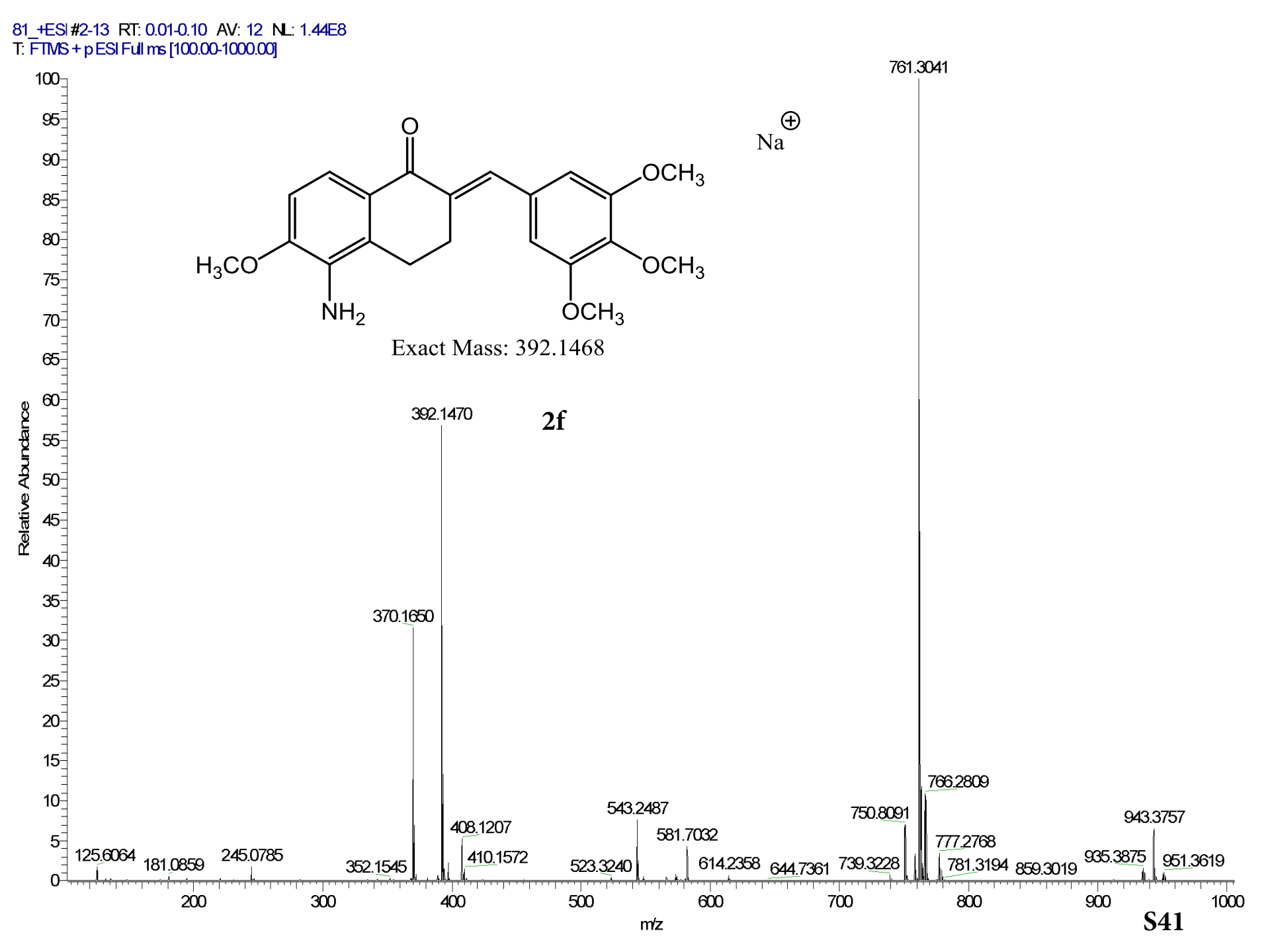
2f

JWF-81.10.fid

^{13}C NMR (151 MHz, $CDCl_3$) δ 187.49, 153.08, 150.89, 138.40, 135.78, 135.02, 132.29, 131.61, 127.57, 127.03, 120.06, 108.55, 107.18, 60.99, 56.23, 55.74, 26.59, 23.09.



2f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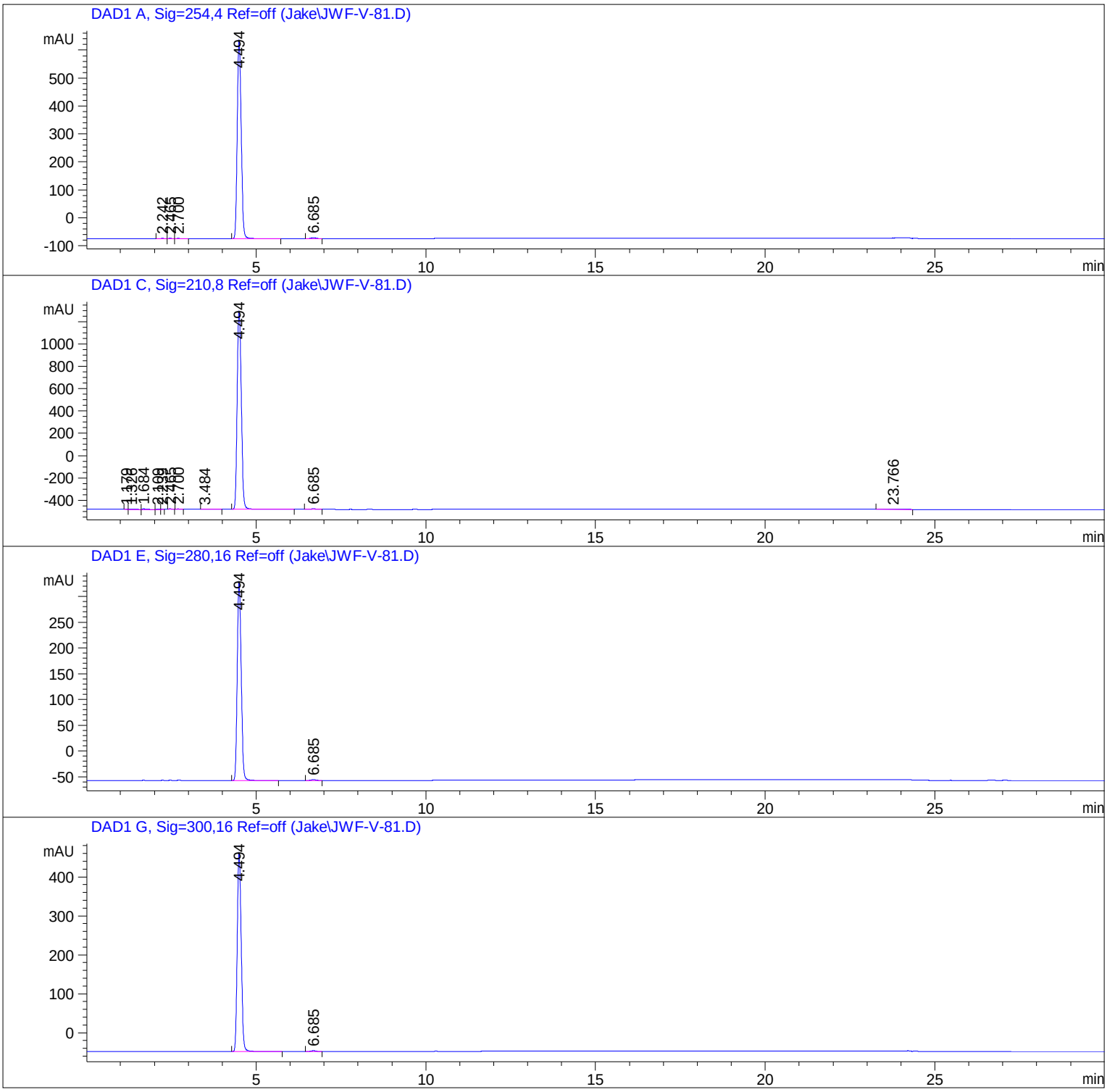
H3CO
OCH3
NH2
OCH3
Exact Mass: 392.1468
2f
Relative Abundance
m/z
761.3041
392.1470
370.1650
543.2487
766.2809
750.8091
943.3757
408.1207
581.7032
777.2768
410.1572
125.6064
181.0859
245.0785
352.1545
523.3240
614.2358
644.7361
739.3228
781.3194
859.3019
935.3875
951.3619

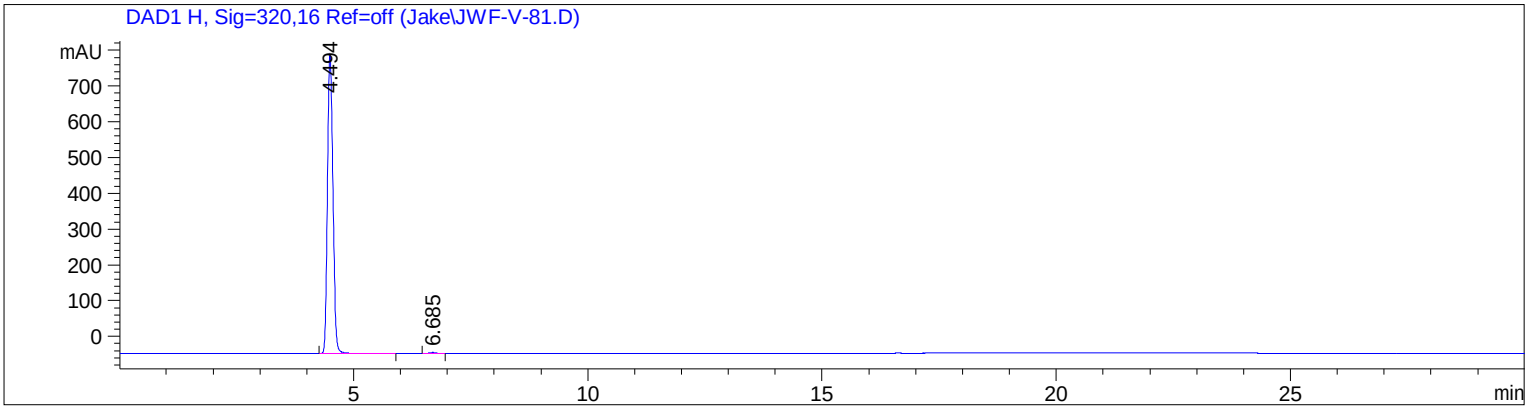
Data File C:\Chem32\1\Data\Jake\JWF-V-81.D
Sample Name: JWF-V-81

==========

Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : 1200 HPLC Location : 1
Injection Date : 10/24/2018 1:12:57 PM
Inj Volume : No inj
Method : C:\CHEM32\1\METHODS\GRAD 2 50-90 ACN.M
Last changed : 4/30/2014 1:53:57 AM by ERICAP
Sample Info : .0003g of sample used



Data File C:\Chem32\1\Data\Jake\JWF-V-81.D
Sample Name: JWF-V-81



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Area Percent Report

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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount: : 1.00000 [ng/ul] (not used in calc.)
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	2.242	BV	0.0814	7.54824	1.43405	0.1312
2	2.465	VV	0.0872	10.21161	1.82761	0.1774
3	2.700	VB	0.0959	9.12633	1.43989	0.1586
4	4.494	BB	0.1255	5692.61035	710.18085	98.9163
5	6.685	BB	0.1587	35.47947	3.48665	0.6165
Totals :				5754.97600	718.36904	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.179	BV	0.0608	11.14494	2.78117	0.0728
2	1.326	VB	0.2335	76.05427	4.63251	0.4967
3	1.684	BB	0.1057	64.83326	8.43432	0.4234
4	2.109	BV	0.0802	21.30974	3.99650	0.1392
5	2.239	VV	0.0812	10.45423	1.87118	0.0683
6	2.465	BB	0.0817	23.74887	4.64487	0.1551
7	2.700	BB	0.0921	21.28649	3.54392	0.1390
8	3.484	BB	0.1860	14.06727	1.02560	0.0919
9	4.494	VB	0.1321	1.49238e4	1775.04883	97.4650
10	6.685	BB	0.1586	51.79534	5.09382	0.3383
11	23.766	BB	0.5427	93.46161	2.25899	0.6104
Totals :				1.53120e4	1813.33170	

Data File C:\Chem32\1\Data\Jake\JWF-V-81.D
Sample Name: JWF-V-81

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.494	BB	0.1255	3086.66309	385.12259	99.4401
2	6.685	BB	0.1565	17.38092	1.71167	0.5599
Totals :				3104.04401	386.83426	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.494	BB	0.1256	4083.54492	509.14136	99.5169
2	6.685	BB	0.1565	19.82307	1.95135	0.4831
Totals :				4103.36800	511.09271	

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

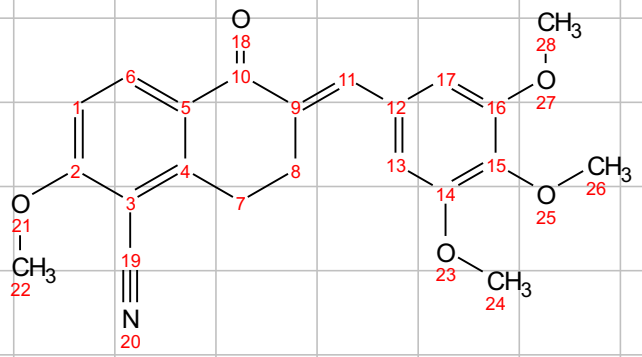
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.494	BB	0.1258	6706.89014	834.46460	99.5614
2	6.685	BB	0.1567	29.54779	2.90490	0.4386
Totals :				6736.43792	837.36950	

==========

*** End of Report ***

421.10.fid

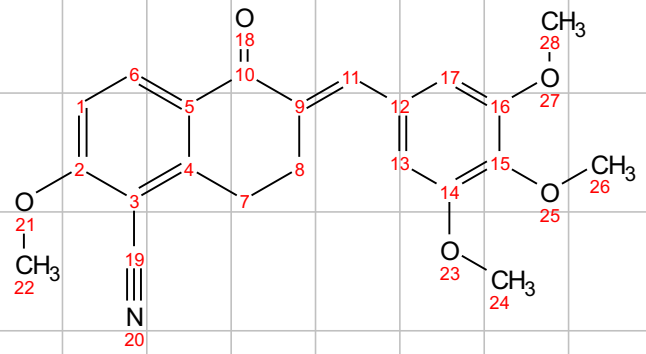
^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.34 (d, *J* = 8.9 Hz, 1H), 7.81 (s, 1H), 7.00 (d, *J* = 8.9 Hz, 1H), 6.68 (s, 2H), 4.04 (s, 3H), 3.91 (s, 3H), 3.90 (s, 6H), 3.21 (t, *J* = 6.5 Hz, 2H), 3.15 (t, *J* = 6.3 Hz, 2H).



2g

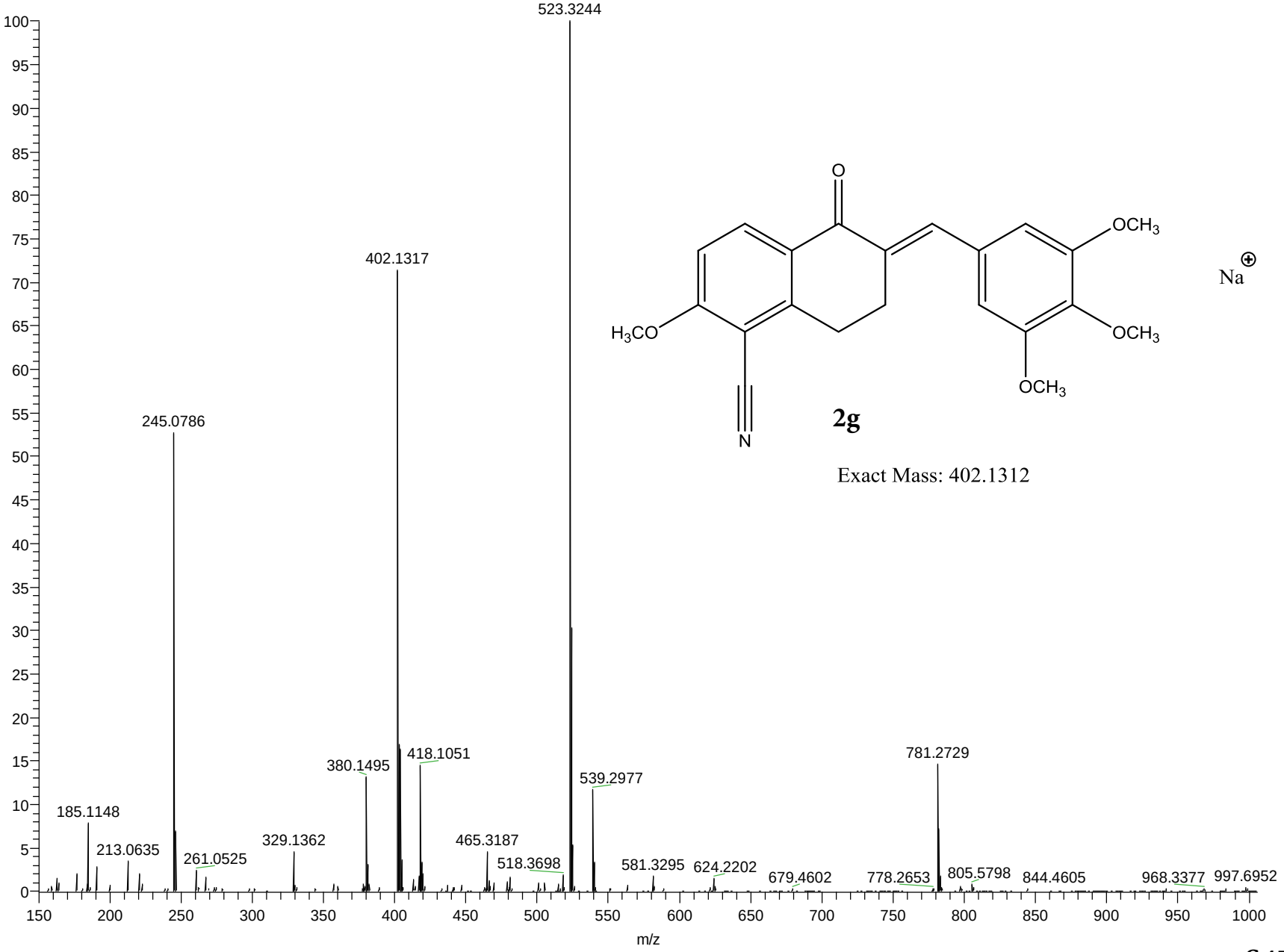
421.20.fid

^{13}C NMR (151 MHz, $CDCl_3$) δ 185.11, 164.94, 153.16, 148.66, 138.94, 137.79, 134.89, 133.21, 130.83, 127.25, 114.45, 109.88, 107.35, 100.92, 60.99, 56.56, 56.24, 27.20, 26.45.



2g

421_190109110802 #2-12 RT: 0.01-0.11 AV: 11 NL: 1.57E7
T: FTMS + p ESI Full ms [150.00-1000.00]



2g

Exact Mass: 402.1312

Data File C:\Chem32\1\Data\Casey\421.D
Sample Name: 421

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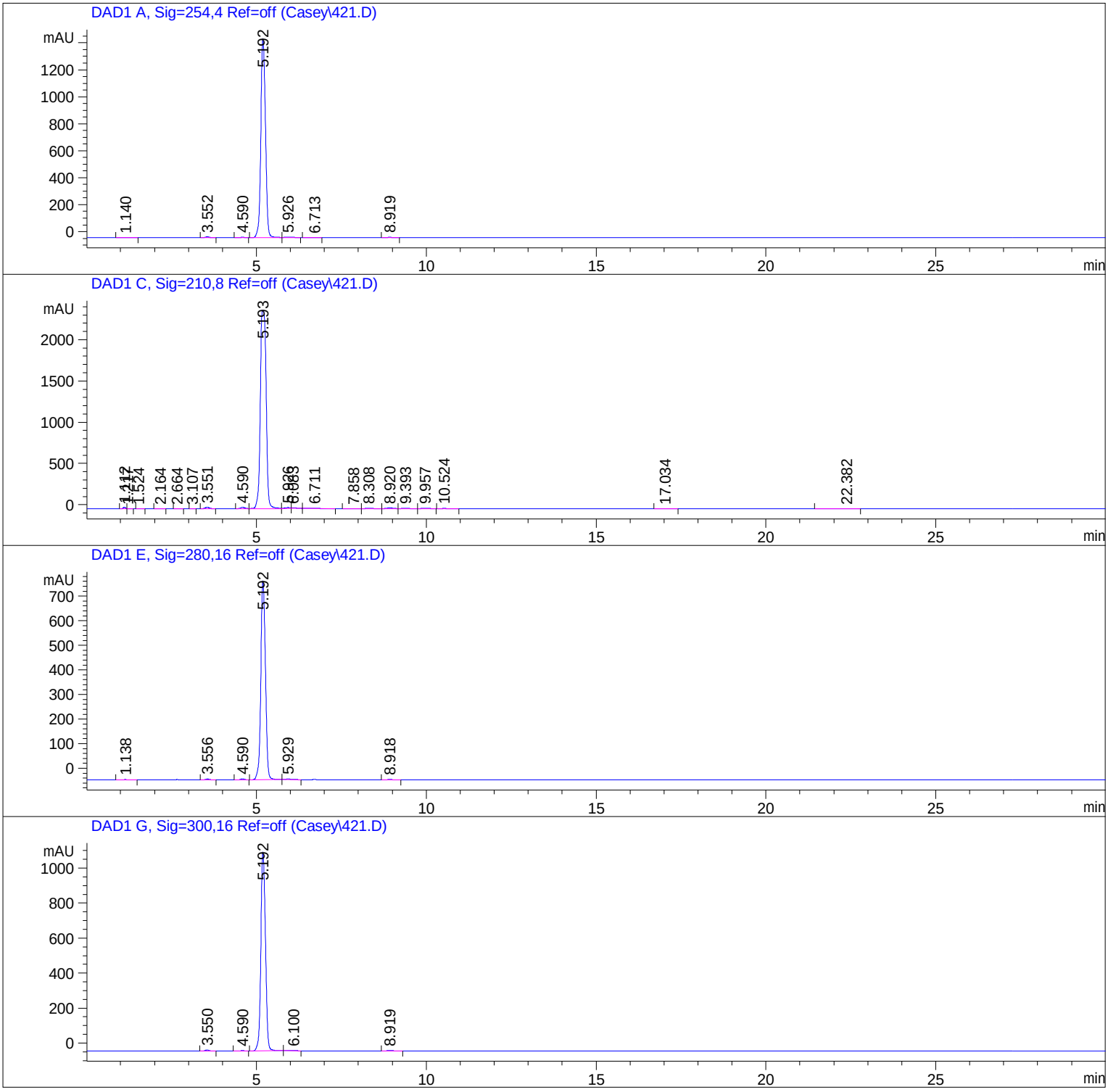
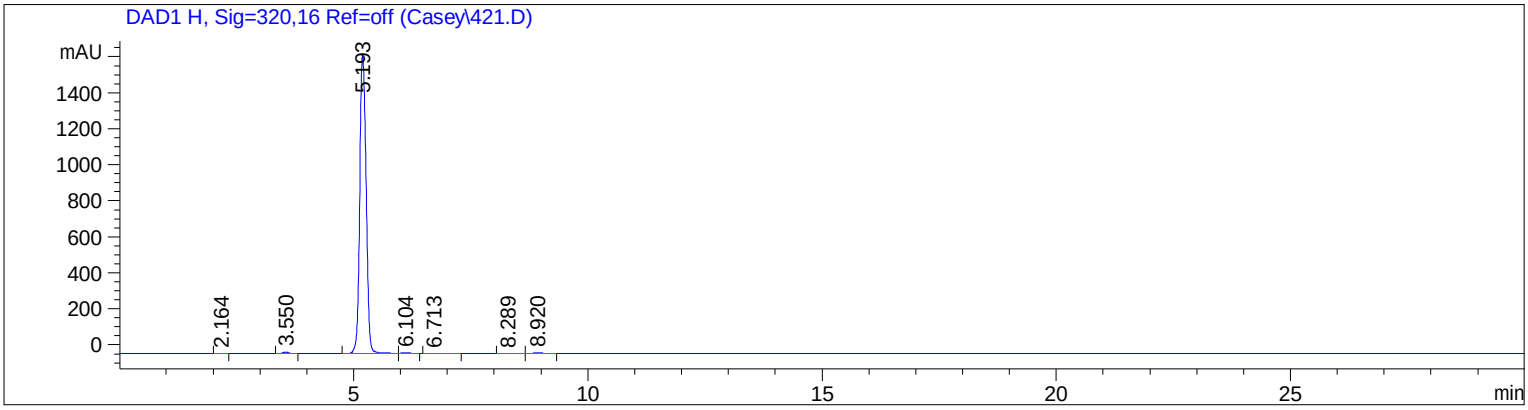
Sample Operator : SYSTEM
Acq. Instrument : 1200 HPLC Location : 1
Injection Date : 1/22/2019 12:38:47 PM
Inj Volume : No inj
Method : C:\Chem32\1\Methods\GRAD 2 50-90 ACN.M
Last changed : 4/30/2014 1:53:57 AM by ERICAP



Data File C:\Chem32\1\Data\Casey\421.D
Sample Name: 421



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Area Percent Report

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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount: : 1.00000 [ng/ul] (not used in calc.)
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	1.140	BB	0.0923	12.00705	1.74377	0.0848
2	3.552	BB	0.1289	50.23670	6.05119	0.3546
3	4.590	BB	0.1317	23.82469	2.90576	0.1682
4	5.192	BB	0.1529	1.39842e4	1471.24951	98.7210
5	5.926	BB	0.1894	45.53900	3.46372	0.3215
6	6.713	BB	0.1844	18.27536	1.56341	0.1290
7	8.919	BB	0.2101	31.28910	2.43230	0.2209
Totals :				1.41653e4	1489.40966	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.112	BV	0.0767	96.87898	19.26521	0.3267
2	1.217	VB	0.0717	15.89412	3.10466	0.0536
3	1.524	BB	0.0678	9.46560	2.05278	0.0319
4	2.164	BB	0.0906	13.62223	2.31717	0.0459
5	2.664	BB	0.0989	6.87014	1.07047	0.0232
6	3.107	VB	0.1019	7.20039	1.10654	0.0243
7	3.551	BB	0.1303	148.49734	17.98485	0.5008
8	4.590	BB	0.1332	124.15651	15.21809	0.4187
9	5.193	BV	0.1928	2.85762e4	2401.48853	96.3641
10	5.926	VV	0.1744	140.96933	11.55250	0.4754
11	6.083	VB	0.1599	87.80029	8.83651	0.2961

Data File C:\Chem32\1\Data\Casey\421.D
Sample Name: 421

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
12	6.711	BB	0.2199	56.52789	3.79321	0.1906
13	7.858	BB	0.2012	13.72396	1.08773	0.0463
14	8.308	BV	0.2354	47.79951	3.14350	0.1612
15	8.920	VV	0.2151	117.29104	8.82388	0.3955
16	9.393	VV	0.2780	43.13024	2.44305	0.1454
17	9.957	VB	0.2347	56.07365	3.92468	0.1891
18	10.524	BB	0.2423	24.64514	1.68787	0.0831
19	17.034	BB	0.2756	23.53830	1.40380	0.0794
20	22.382	BB	0.3168	44.12827	2.26960	0.1488
Totals :				2.96544e4	2512.57462	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.138	BB	0.0884	7.38130	1.12787	0.0952
2	3.556	BB	0.1284	24.02840	2.90925	0.3098
3	4.590	BB	0.1334	29.55334	3.61260	0.3810
4	5.192	BB	0.1526	7639.67920	806.12268	98.5010
5	5.929	BB	0.2044	39.02044	2.67170	0.5031
6	8.918	BB	0.2078	16.27808	1.25130	0.2099
Totals :				7755.94075	817.69540	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.550	BB	0.1285	39.52570	4.78016	0.3663
2	4.590	BB	0.1330	15.13307	1.82008	0.1402
3	5.192	BB	0.1518	1.06716e4	1133.85950	98.8967
4	6.100	BB	0.1937	29.36630	2.32025	0.2721
5	8.919	BB	0.2155	35.02502	2.66263	0.3246
Totals :				1.07906e4	1145.44262	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.164	BB	0.0821	5.65133	1.06130	0.0354
2	3.550	BB	0.1302	64.41965	7.81288	0.4040
3	5.193	BV	0.1529	1.57434e4	1655.88269	98.7230
4	6.104	VB	0.1805	50.00229	4.40176	0.3136

Data File C:\Chem32\1\Data\Casey\421.D
Sample Name: 421

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
5	6.713	BB	0.1831	14.19239	1.22515	0.0890
6	8.289	BB	0.2138	14.68582	1.09946	0.0921
7	8.920	BB	0.2143	54.68512	4.13423	0.3429
Totals :				1.59471e4	1675.61747	

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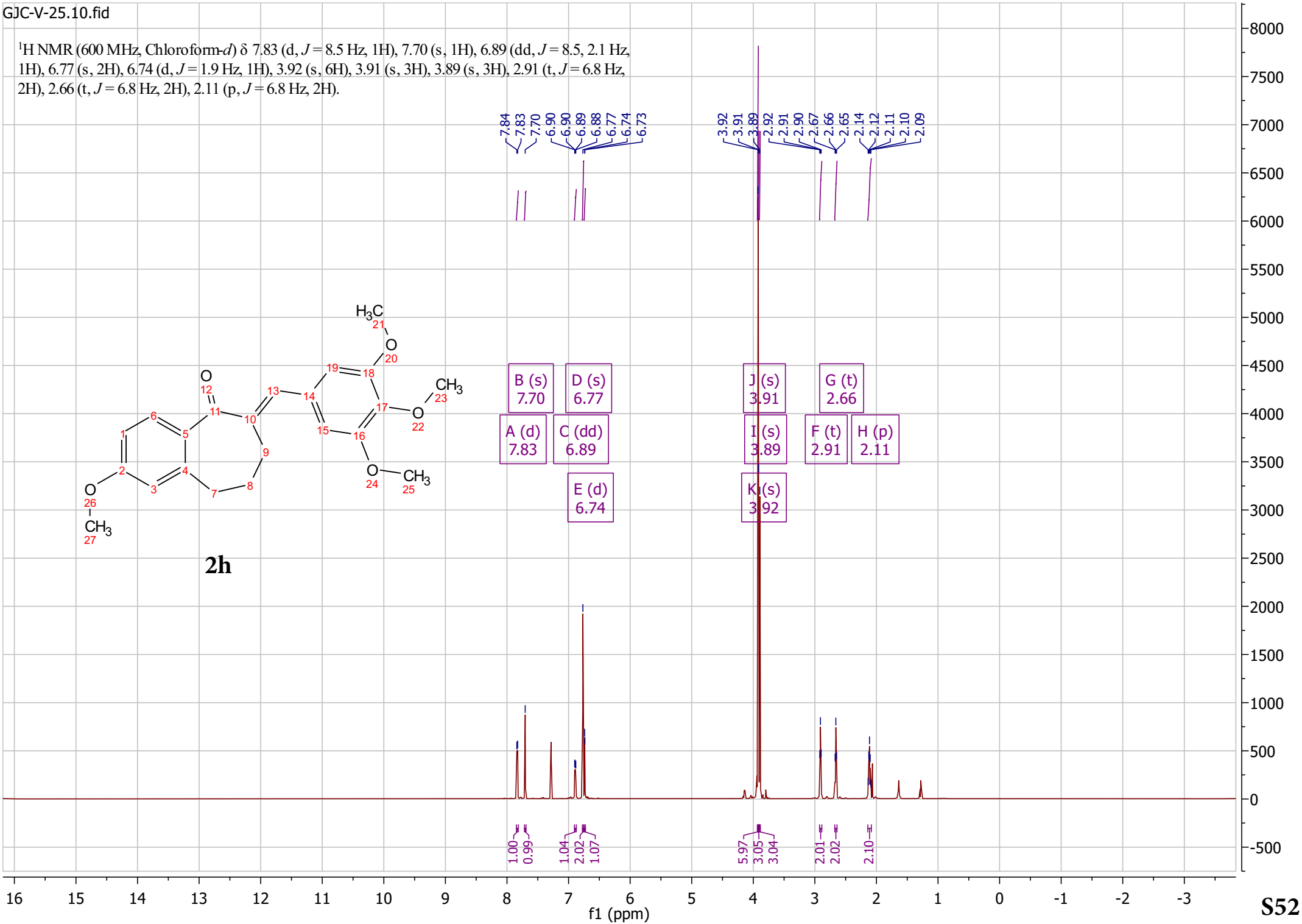
*** End of Report ***

GJC-V-25.10.fid

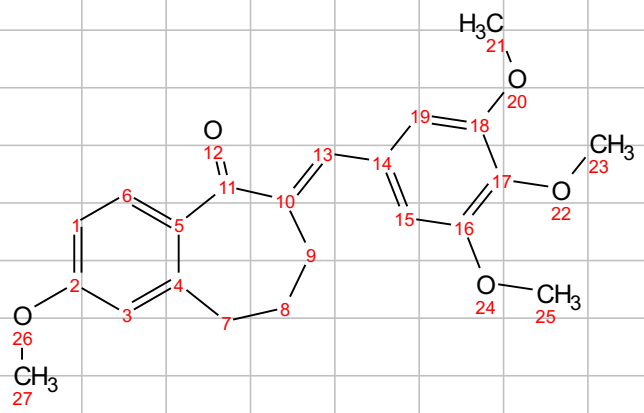
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.83 (d, *J* = 8.5 Hz, 1H), 7.70 (s, 1H), 6.89 (dd, *J* = 8.5, 2.1 Hz, 1H), 6.77 (s, 2H), 6.74 (d, *J* = 1.9 Hz, 1H), 3.92 (s, 6H), 3.91 (s, 3H), 3.89 (s, 3H), 2.91 (t, *J* = 6.8 Hz, 2H), 2.66 (t, *J* = 6.8 Hz, 2H), 2.11 (p, *J* = 6.8 Hz, 2H).

2h

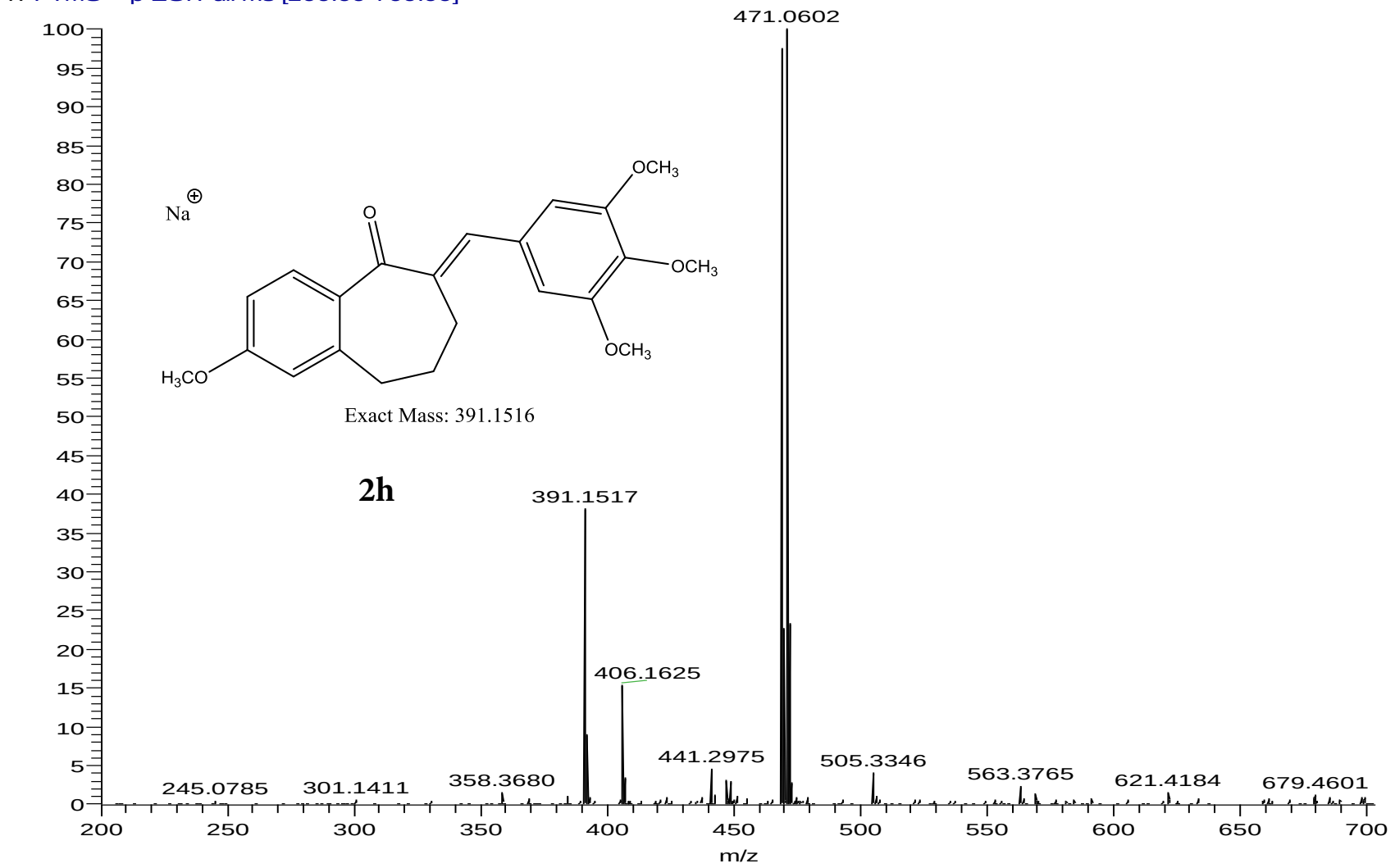


GJC-V-25.21.fid

^{13}C NMR (151 MHz, $CDCl_3$) δ 196.92, 163.02, 153.17, 142.21, 138.55, 137.92, 137.61, 131.75, 131.51, 131.35, 114.54, 111.99, 106.75, 60.98, 56.18, 55.40, 32.54, 26.49, 25.29.



2h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OCH3
OCH3
OCH3
H3CO
Exact Mass: 391.1516
2h
391.1517
406.1625
441.2975
505.3346
563.3765
621.4184
679.4601
245.0785
301.1411
358.3680
m/z

Data File C:\Chem32\1\Data\Graham\GJC-V-25.D
Sample Name: GJC-V-25

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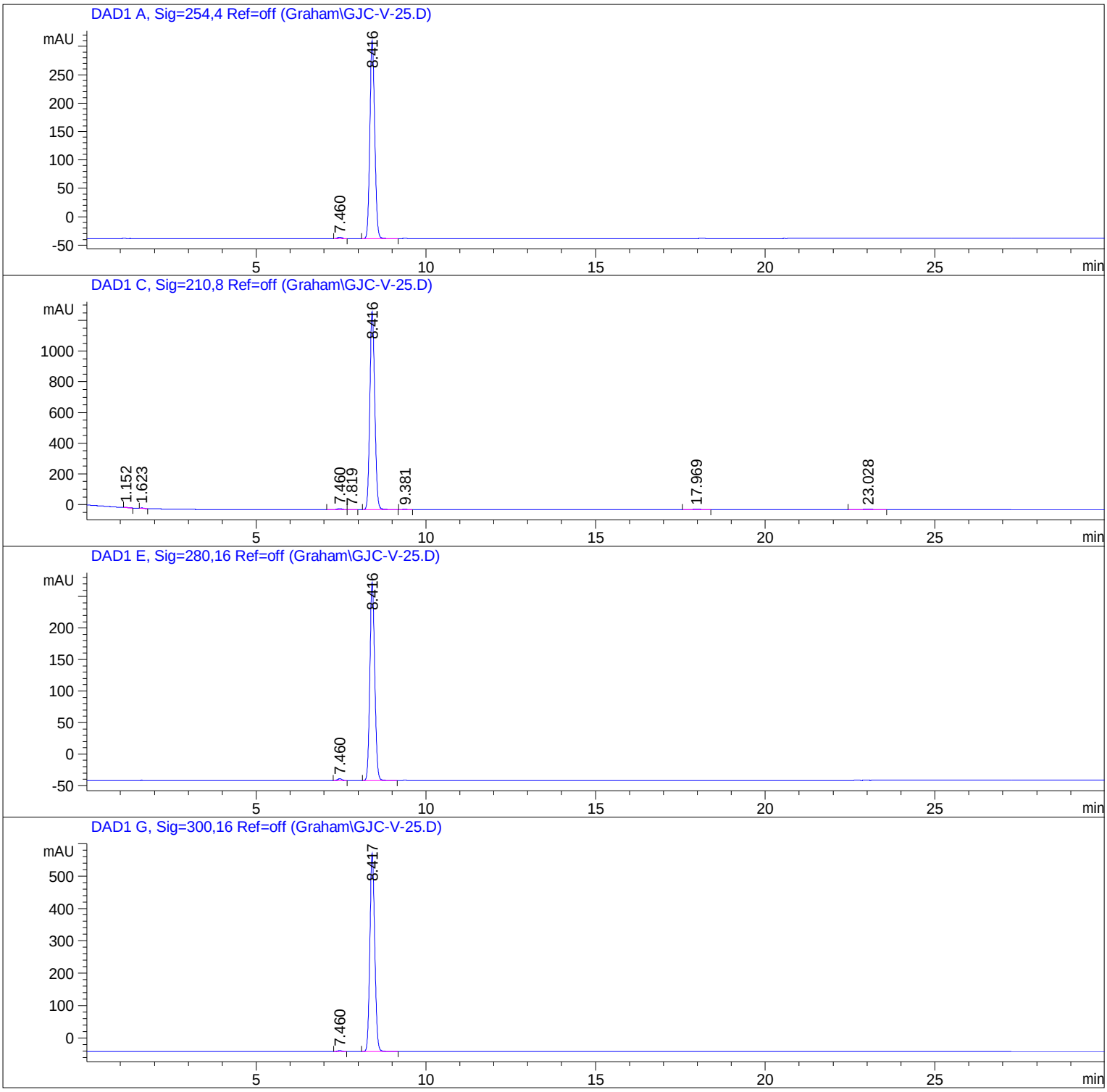
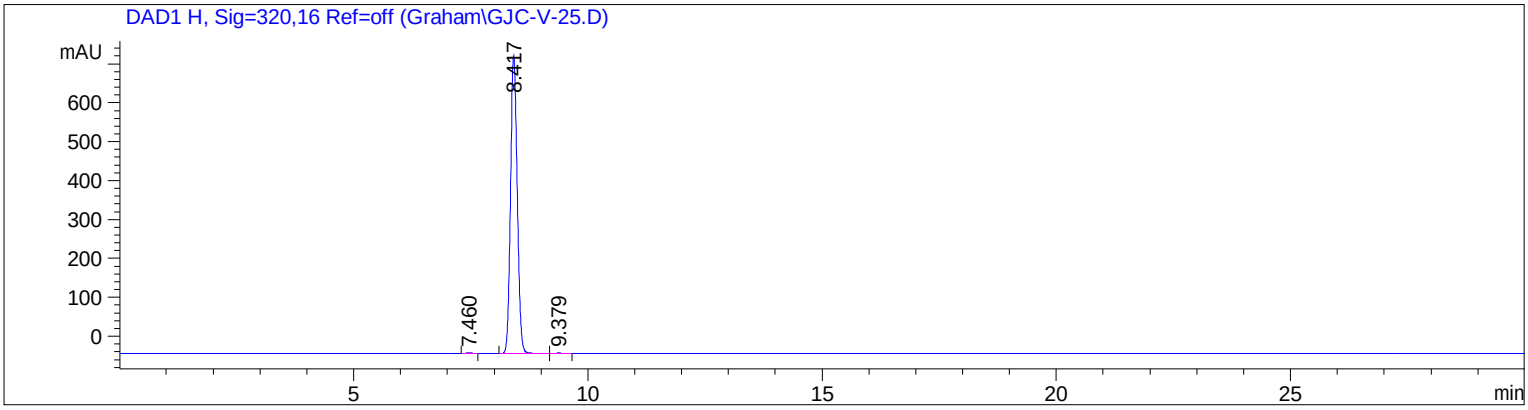
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : 1200 HPLC Location : 1
Injection Date : 9/18/2018 3:57:04 PM
Inj Volume : No inj
Method : C:\CHEM32\1\METHODS\GRAD 2 50-90 ACN.M
Last changed : 4/30/2014 1:53:57 AM by ERICAP
Sample Info : GJC-V-25



Data File C:\Chem32\1\Data\Graham\GJC-V-25.D
Sample Name: GJC-V-25



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Area Percent Report

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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount: : 1.00000 [ng/ul] (not used in calc.)
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	7.460	BB	0.1437	23.59247	2.60374	0.6684
2	8.416	BB	0.1573	3506.33521	348.86710	99.3316
Totals :				3529.92768	351.47084	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.152	BB	0.0872	18.39275	3.19479	0.1372
2	1.623	VB	0.0708	18.14385	3.86345	0.1354
3	7.460	BV	0.1529	65.57661	6.66080	0.4892
4	7.819	VB	0.1391	10.02179	1.15592	0.0748
5	8.416	BB	0.1608	1.31457e4	1291.40552	98.0654
6	9.381	BB	0.1614	19.73553	1.92772	0.1472
7	17.969	BB	0.2654	51.32008	2.91530	0.3828
8	23.028	BB	0.2487	76.14536	4.70704	0.5680
Totals :				1.34051e4	1315.83054	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.460	BB	0.1446	26.37920	2.88611	0.8276
2	8.416	BB	0.1573	3160.97168	314.45236	99.1724
Totals :				3187.35088	317.33847	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.460	BB	0.1432	26.59798	2.94901	0.4313
2	8.417	BB	0.1573	6140.50586	610.67761	99.5687
Totals :				6167.10384	613.62662	

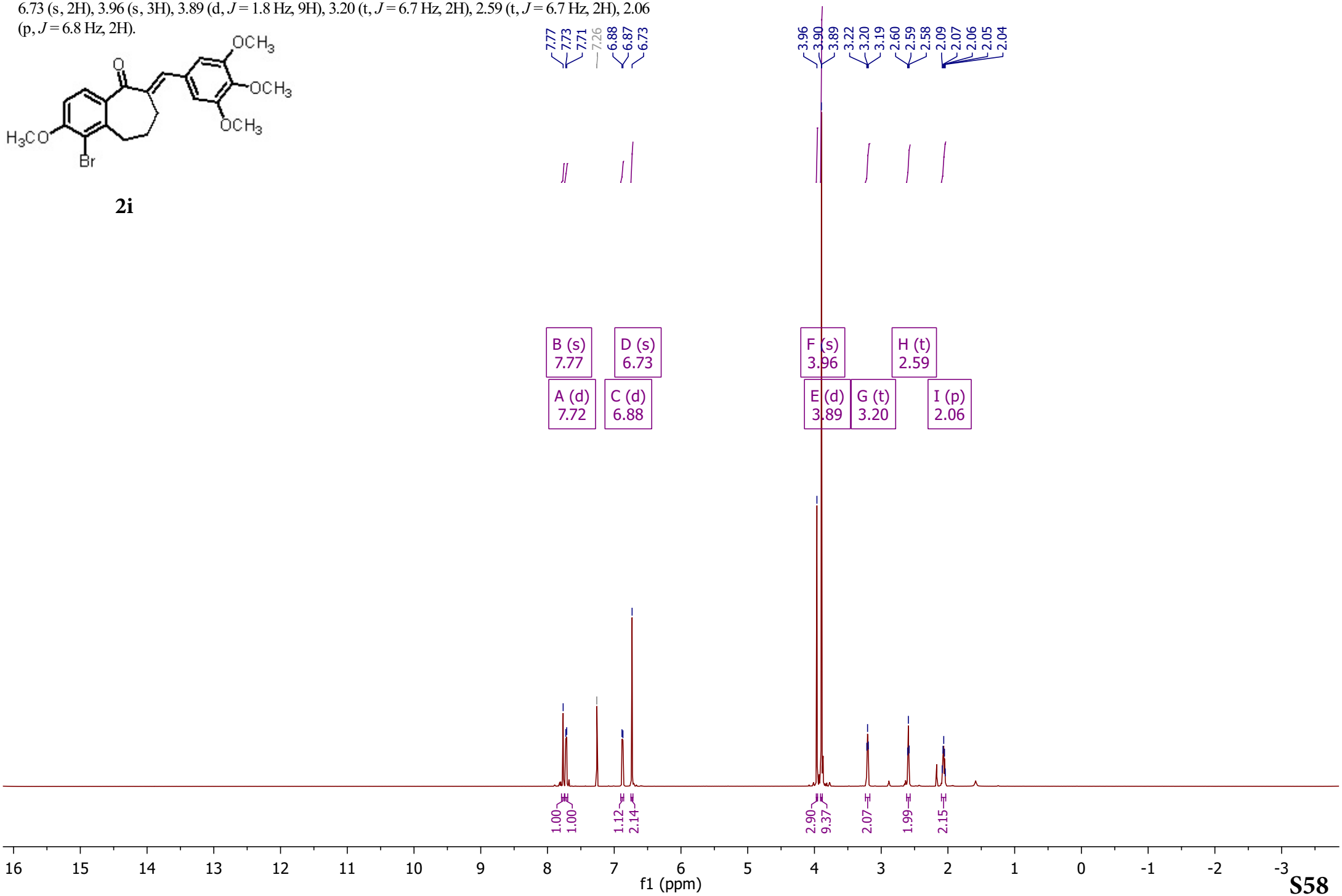
Signal 5: DAD1 H, Sig=320,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.460	BB	0.1385	16.15887	1.84009	0.2092
2	8.417	BB	0.1573	7696.69678	765.74493	99.6364
3	9.379	BB	0.1631	11.92932	1.14928	0.1544
Totals :				7724.78497	768.73431	

*** End of Report ***

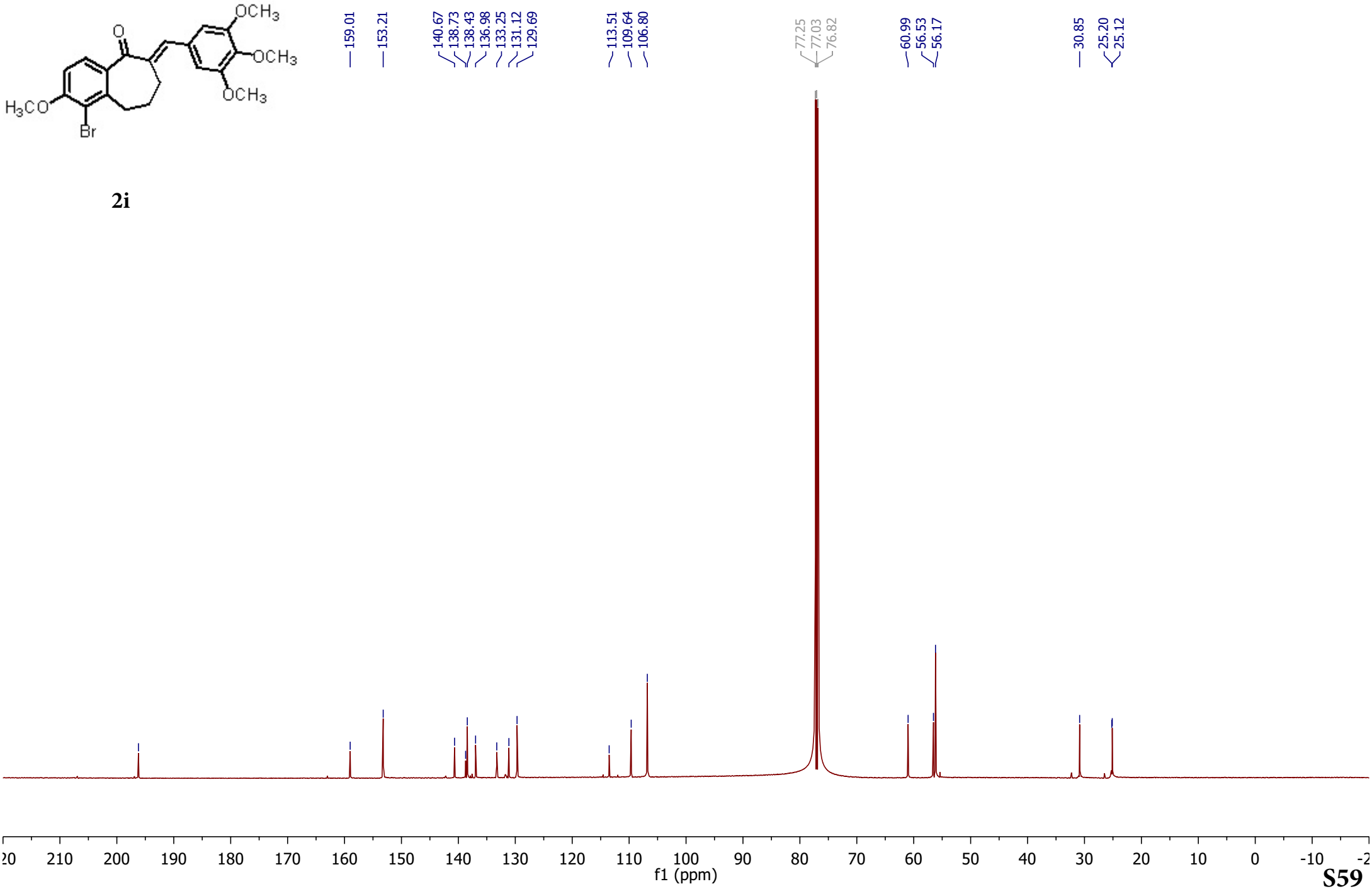
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.77 (s, 1H), 7.72 (d, *J* = 8.5 Hz, 1H), 6.88 (d, *J* = 8.6 Hz, 1H), 6.73 (s, 2H), 3.96 (s, 3H), 3.89 (d, *J* = 1.8 Hz, 9H), 3.20 (t, *J* = 6.7 Hz, 2H), 2.59 (t, *J* = 6.7 Hz, 2H), 2.06 (p, *J* = 6.8 Hz, 2H).

2i

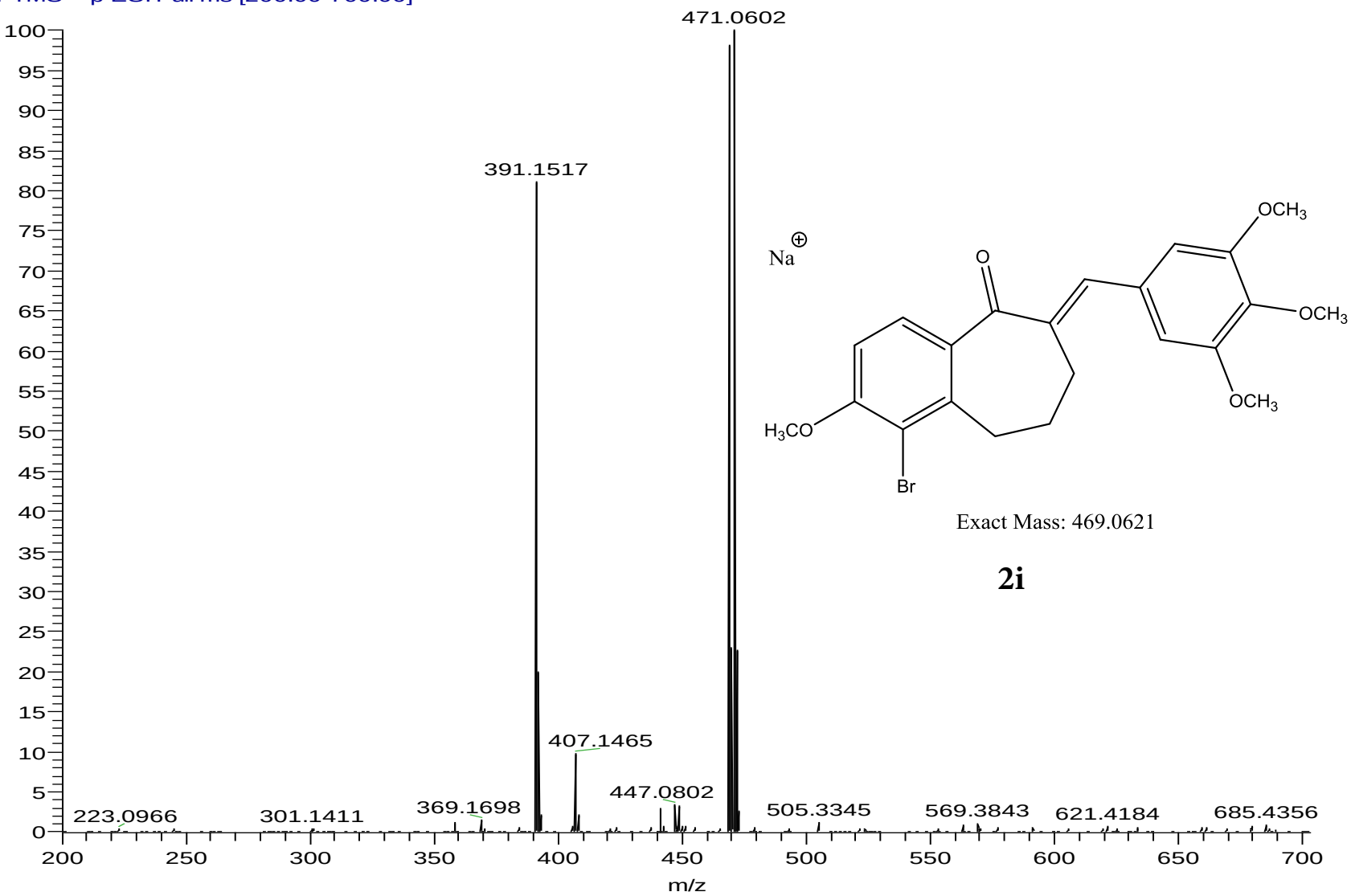


^{13}C NMR (151 MHz, $CDCl_3$) δ 196.20, 159.01, 153.21, 140.67, 138.73, 138.43, 136.98, 133.25, 131.12, 129.69, 113.51, 109.64, 106.80, 60.99, 56.53, 56.17, 30.85, 25.20, 25.12.

2i



GJC-V-18pure_+ESI #2-12 RT: 0.01-0.10 AV: 11 NL: 3.99E7
T: FTMS + p ESI Full ms [200.00-700.00]



Exact Mass: 469.0621

2i

Data File C:\Chem32\1\Data\Graham\GJC-V-18.2.D
Sample Name: GJC-V-18.2

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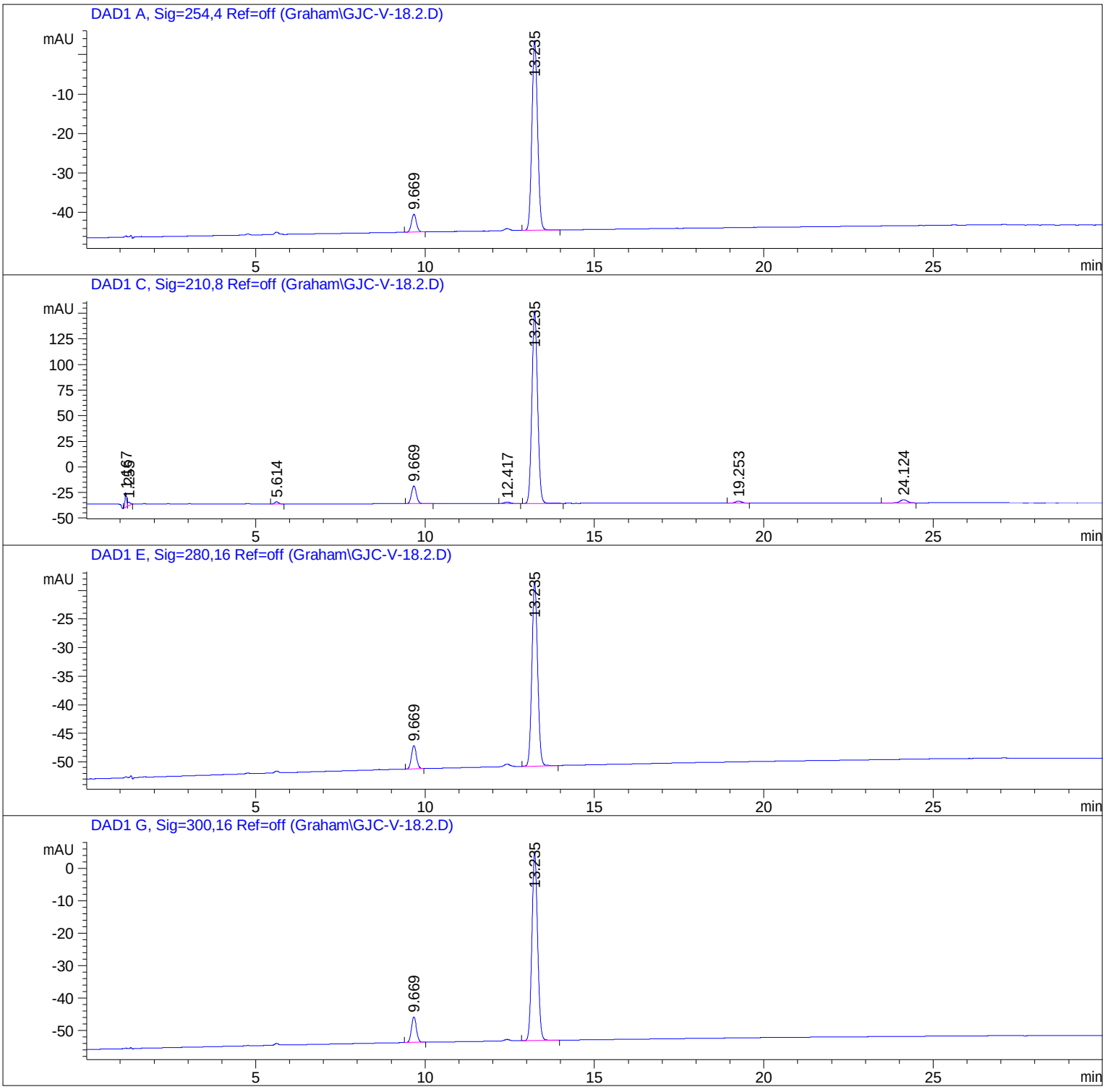
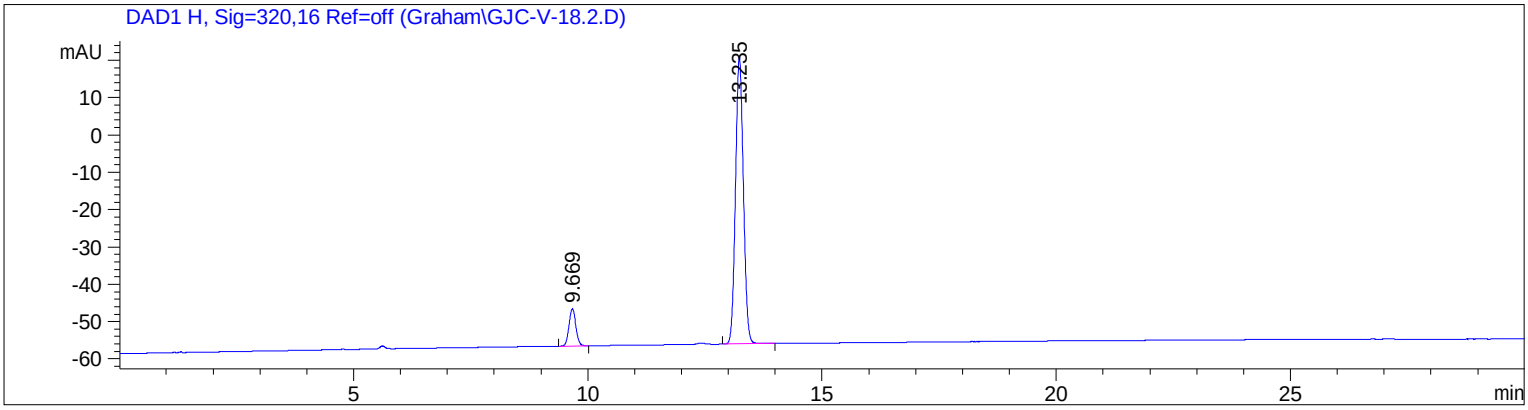
Sample Operator : SYSTEM
Acq. Instrument : 1200 HPLC Location : 1
Injection Date : 9/13/2018 2:42:32 PM
Inj Volume : No inj
Method : C:\CHEM32\1\METHODS\GRAD 2 50-90 ACN.M
Last changed : 4/30/2014 1:53:57 AM by ERICAP
Sample Info : GJC-V-18.2



Data File C:\Chem32\1\Data\Graham\GJC-V-18.2.D
Sample Name: GJC-V-18.2



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Area Percent Report

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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	9.669	BB	0.1591	46.19751	4.52631	7.5361
2	13.235	BB	0.1834	566.81787	48.11269	92.4639
Totals :				613.01538	52.63901	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.167	BV	0.0501	45.73252	14.00312	1.7817
2	1.255	VB	0.0774	20.64750	3.79848	0.8044
3	5.614	BB	0.1205	16.18366	2.08610	0.6305
4	9.669	BB	0.1594	176.81618	17.27099	6.8886
5	12.417	BB	0.1820	17.40522	1.47152	0.6781
6	13.235	BB	0.1834	2216.09253	188.16910	86.3369
7	19.253	BB	0.2119	27.45221	2.05482	1.0695
8	24.124	BB	0.2325	46.46711	3.10674	1.8103
Totals :				2566.79695	231.96088	

Data File C:\Chem32\1\Data\Graham\GJC-V-18.2.D
Sample Name: GJC-V-18.2

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.669	BB	0.1609	41.62261	4.08576	9.8550
2	13.235	BB	0.1834	380.72592	32.31737	90.1450
Totals :				422.34853	36.40313	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.669	BB	0.1592	80.96008	7.92405	10.5484
2	13.235	BB	0.1834	686.54950	58.31433	89.4516
Totals :				767.50958	66.23838	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.669	BB	0.1592	101.32680	9.91957	10.0259
2	13.235	BB	0.1833	909.32581	77.25915	89.9741
Totals :				1010.65260	87.17872	

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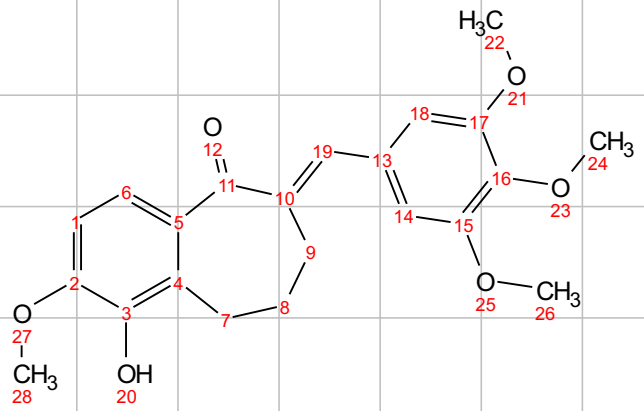
*** End of Report ***

GJC-V-13.20.fid

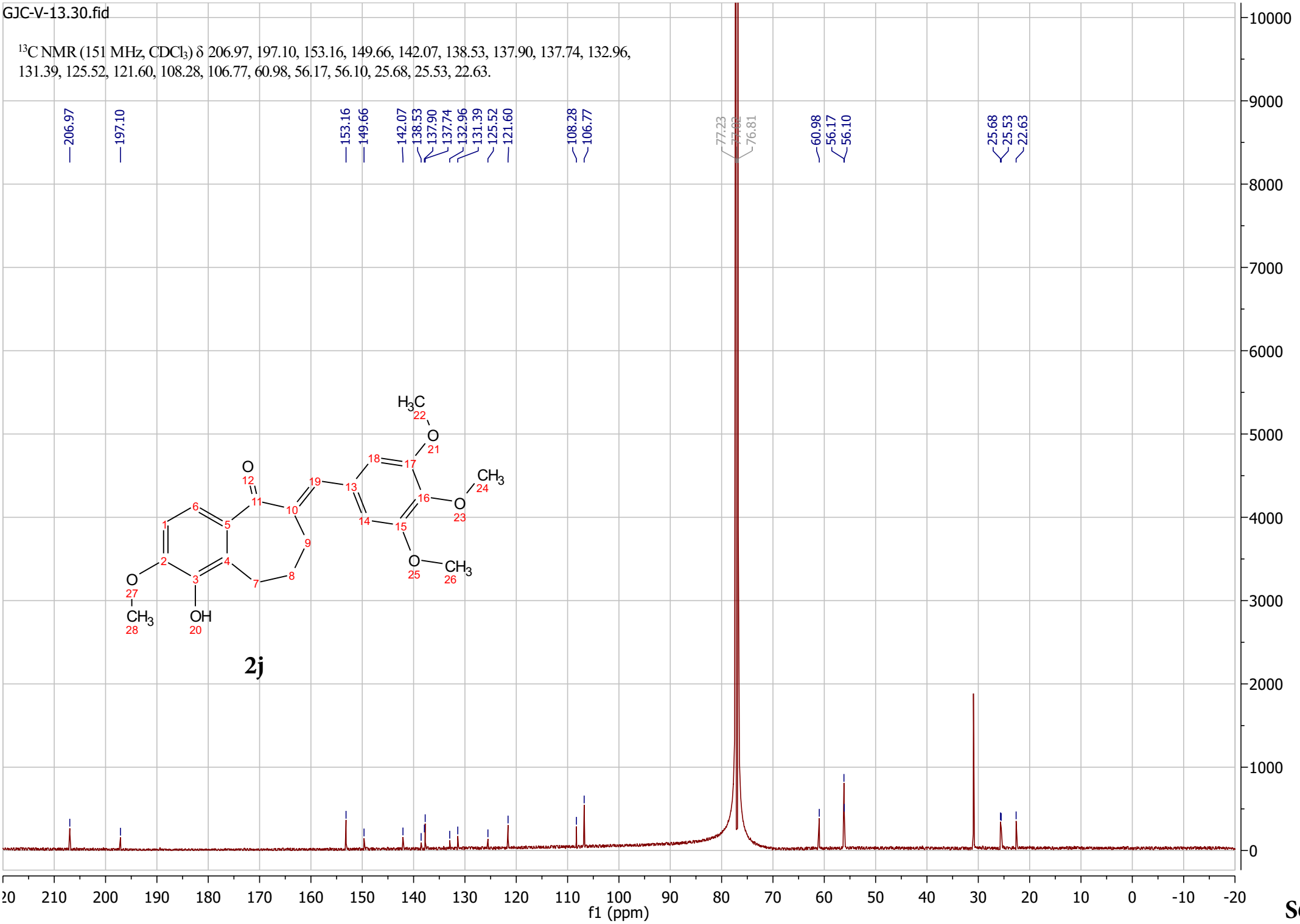
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.74 (s, 1H), 7.42 (d, *J* = 8.4 Hz, 1H), 6.87 (d, *J* = 8.4 Hz, 1H), 6.78 (s, 2H), 5.77 (s, 1H), 3.99 (s, 3H), 3.92 (s, 6H), 3.92 (s, 3H), 3.04 (t, *J* = 6.7 Hz, 2H), 2.65 (t, *J* = 6.7 Hz, 2H), 2.09 (p, *J* = 6.7 Hz, 2H).



2j

GJC-V-13.30.fid

^{13}C NMR (151 MHz, $CDCl_3$) δ 206.97, 197.10, 153.16, 149.66, 142.07, 138.53, 137.90, 137.74, 132.96, 131.39, 125.52, 121.60, 108.28, 106.77, 60.98, 56.17, 56.10, 25.68, 25.53, 22.63.



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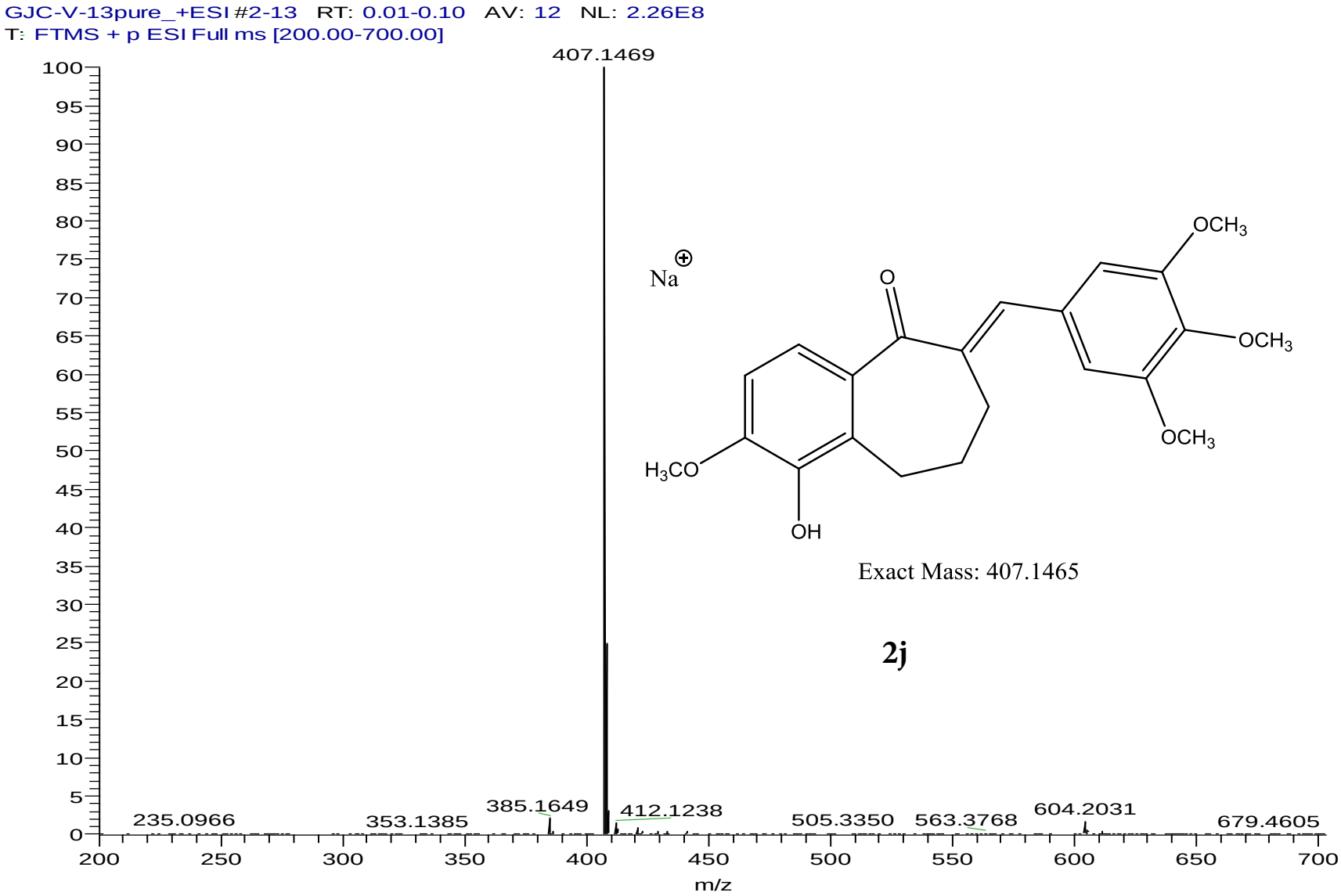

Exact Mass: 407.1465

2j

Data File C:\Chem32\1\Data\Graham\GJC-V-13.D
Sample Name: GJC-V-13

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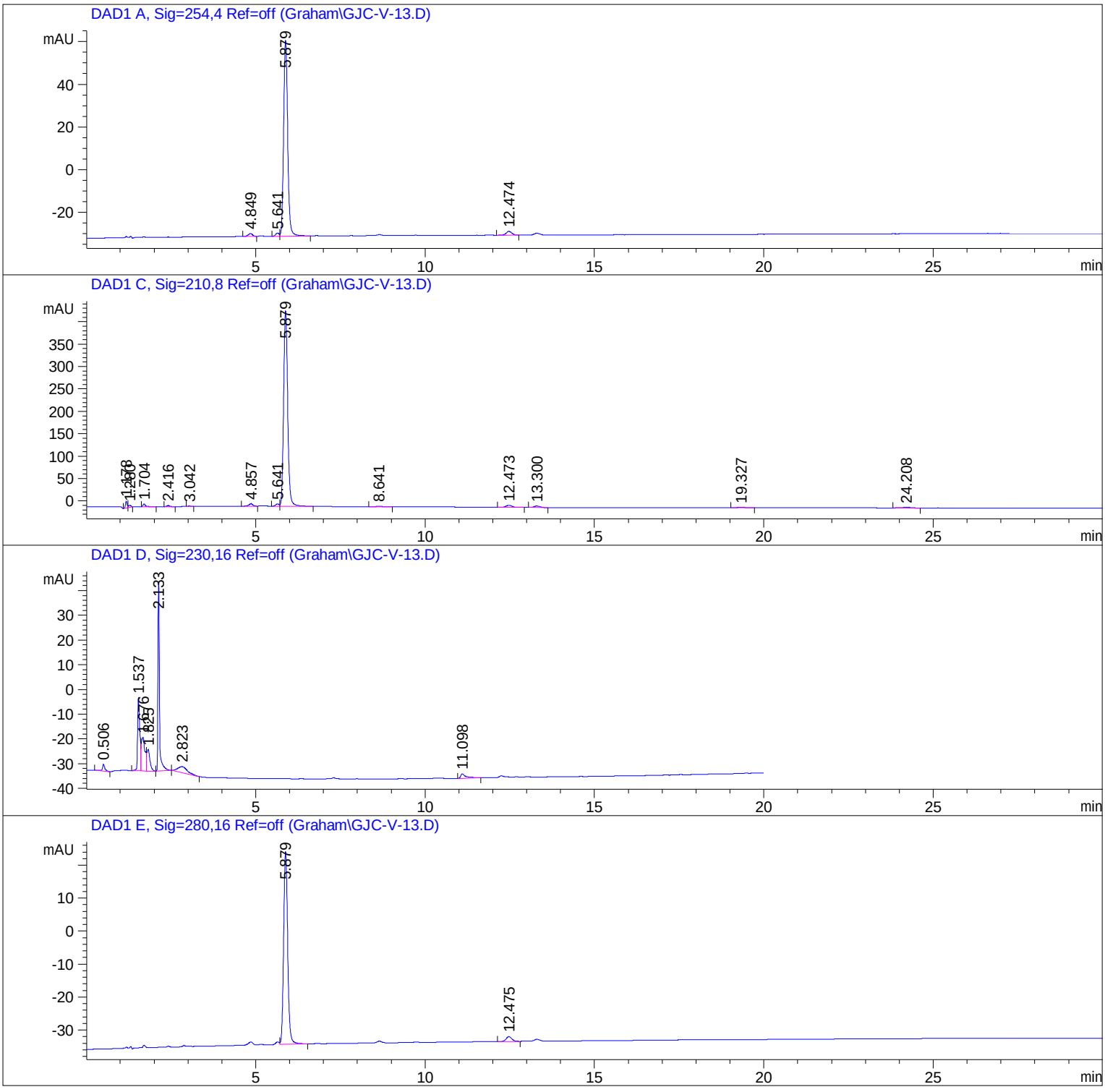
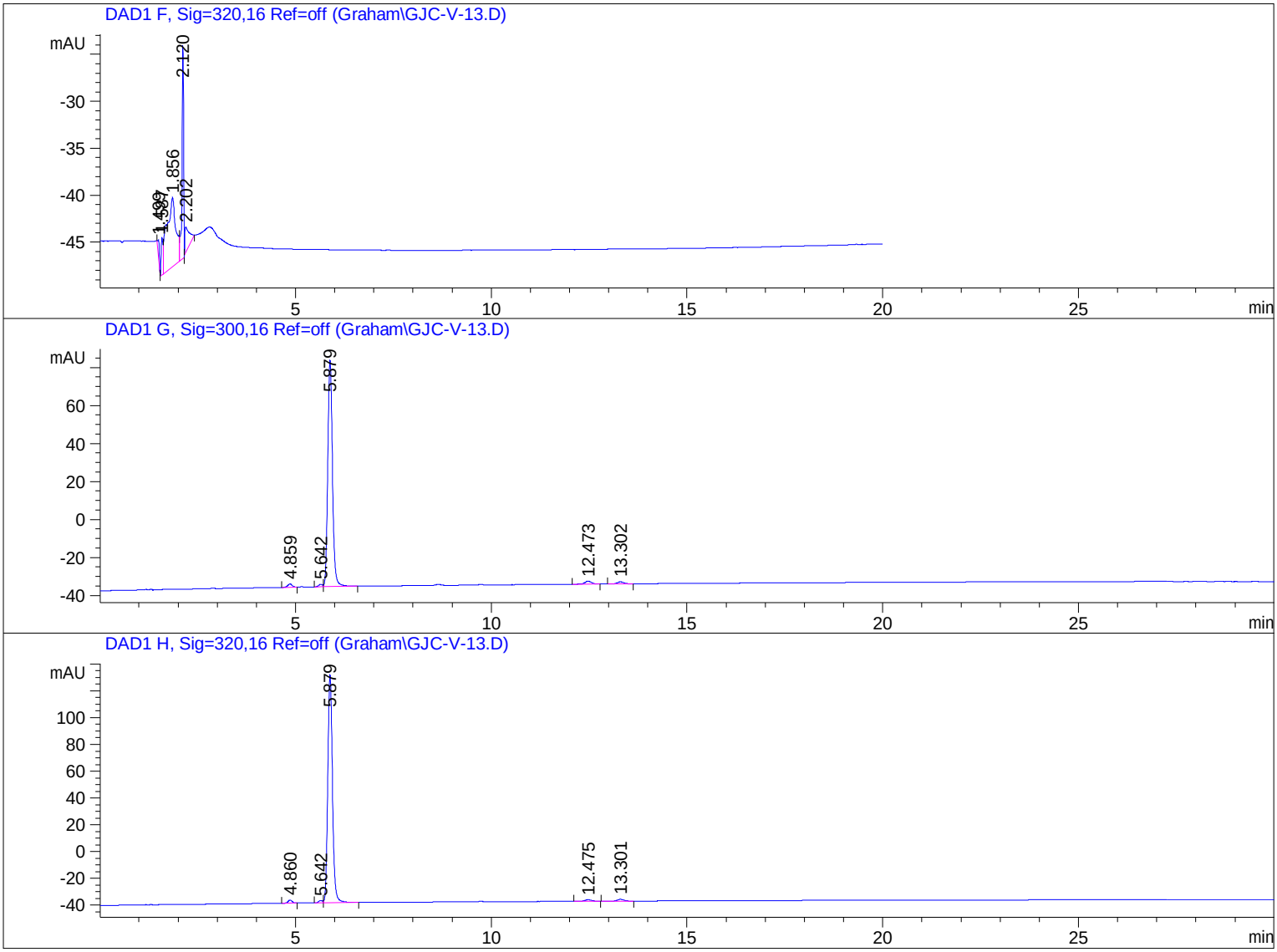
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : 1200 HPLC Location : 1
Injection Date : 9/14/2018 7:57:38 AM
Inj Volume : No inj
Method : C:\CHEM32\1\METHODS\GRAD 2 50-90 ACN.M
Last changed : 4/30/2014 1:53:57 AM by ERICAP
Sample Info : GJC-V-13



Data File C:\Chem32\1\Data\Graham\GJC-V-13.D
Sample Name: GJC-V-13



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Area Percent Report

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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	4.849	BB	0.1382	12.45995	1.36834	1.5457
2	5.641	BV	0.1086	10.24514	1.48218	1.2709
3	5.879	VB	0.1290	762.45325	91.70485	94.5827
4	12.474	BB	0.1841	20.96515	1.77175	2.6007

Totals : 806.12348 96.32713

Data File C:\Chem32\1\Data\Graham\GJC-V-13.D
Sample Name: GJC-V-13

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.178	BV	0.0536	51.60966	15.20893	1.2928
2	1.280	VB	0.0746	25.34420	4.87942	0.6348
3	1.704	BB	0.0665	27.20108	6.28689	0.6814
4	2.416	BB	0.0847	13.26962	2.39170	0.3324
5	3.042	VB	0.1074	7.05246	1.01067	0.1767
6	4.857	BV	0.1306	47.85615	5.44164	1.1987
7	5.641	BV	0.1079	34.24988	5.00033	0.8579
8	5.879	VB	0.1292	3635.32129	436.37903	91.0600
9	8.641	BB	0.1629	14.30626	1.31573	0.3584
10	12.473	BB	0.1842	58.22163	4.91606	1.4584
11	13.300	BB	0.1826	38.84506	3.31729	0.9730
12	19.327	BB	0.2101	14.89887	1.09970	0.3732
13	24.208	BB	0.2316	24.04900	1.63520	0.6024
Totals :				3992.22515	488.88258	

Signal 3: DAD1 D, Sig=230,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	0.506	BB	0.0787	15.42358	2.77905	2.4868
2	1.537	BV	0.0641	132.29343	29.63005	21.3301
3	1.676	VV	0.1006	103.62721	13.64454	16.7082
4	1.825	VB	0.0872	55.19541	8.80080	8.8993
5	2.133	BB	0.0493	245.69960	76.93481	39.6149
6	2.823	BB	0.3034	48.53992	2.42646	7.8262
7	11.098	BB	0.1477	19.44036	1.83414	3.1344
Totals :				620.21952	136.04986	

Signal 4: DAD1 E, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.879	VB	0.1290	486.09088	58.48824	96.5257
2	12.475	BB	0.1839	17.49597	1.48068	3.4743
Totals :				503.58685	59.96891	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.499	BB	0.0456	5.39560	1.97074	2.4470
2	1.587	BV	0.0475	12.08468	3.96990	5.4805
3	1.856	VV	0.2138	119.71537	7.35942	54.2922
4	2.120	VB	0.0443	63.14766	22.83095	28.6381
5	2.202	BB	0.1044	20.15869	2.65987	9.1422
Totals :				220.50201	38.79087	

Signal 6: DAD1 G, Sig=300,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.859	BB	0.1188	13.92960	1.78911	1.3344
2	5.642	BV	0.1052	9.17704	1.35145	0.8791
3	5.879	VB	0.1289	990.40204	119.21412	94.8744
4	12.473	BB	0.1847	18.17884	1.52887	1.7414
5	13.302	BB	0.1843	12.22162	1.03113	1.1708
Totals :				1043.90914	124.91468	

Signal 7: DAD1 H, Sig=320,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.860	BB	0.1160	18.51048	2.45365	1.2532
2	5.642	BV	0.1050	12.41302	1.83236	0.8404
3	5.879	VB	0.1289	1415.74805	170.39006	95.8489
4	12.475	BB	0.1856	14.12392	1.16336	0.9562
5	13.301	BB	0.1873	16.26711	1.36198	1.1013
Totals :				1477.06257	177.20141	

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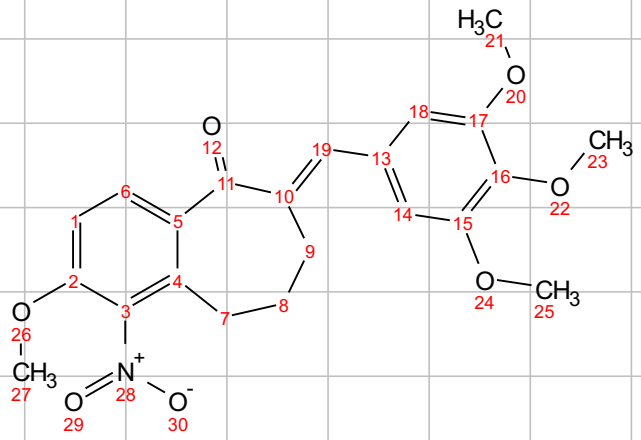
*** End of Report ***

GJC-V-23.10.fid

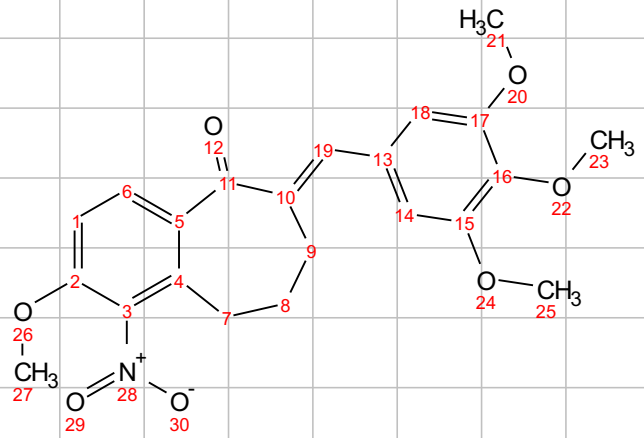
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.92 (d, *J* = 8.5 Hz, 1H), 7.79 (s, 1H), 7.05 (d, *J* = 8.6 Hz, 1H), 6.75 (s, 2H), 3.99 (s, 3H), 3.92 (s, 9H), 2.82 (t, *J* = 6.6 Hz, 2H), 2.68 (t, *J* = 6.6 Hz, 2H), 2.15 (p, *J* = 6.8 Hz, 2H).



2k

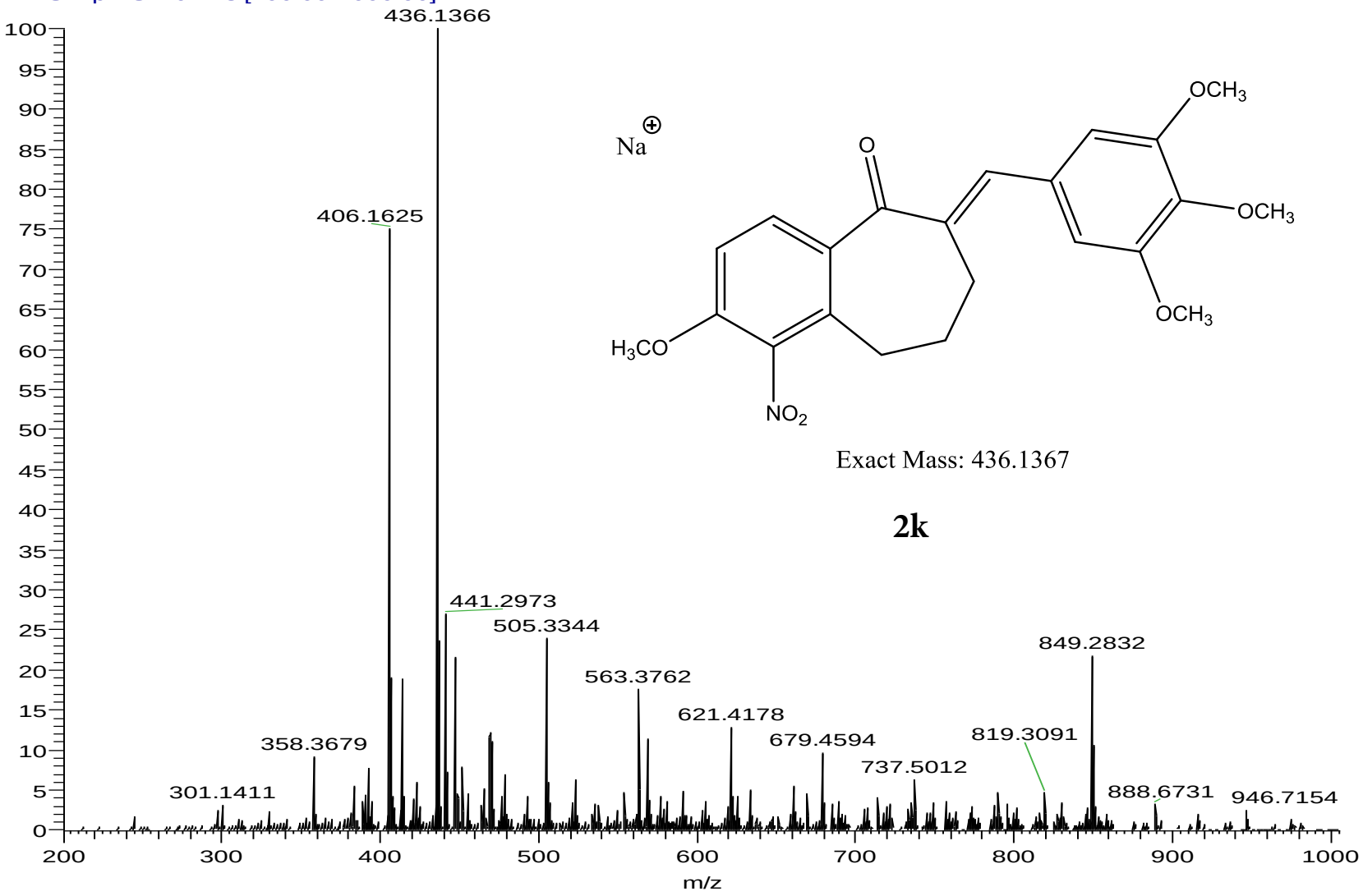
GJC-V-23.11.fid

^{13}C NMR (151 MHz, $CDCl_3$) δ 206.98, 153.49, 153.26, 141.03, 139.27, 139.01, 132.68, 132.41, 130.71, 110.57, 106.89, 61.00, 56.62, 56.20, 26.38, 26.06, 25.03.



2k

GJC-V-23_+ESI #2-13 RT: 0.01-0.10 AV: 12 NL: 7.15E6
T: FTMS + p ESI Full ms [200.00-1000.00]



Exact Mass: 436.1367

2k

Data File C:\Chem32\1\Data\Graham\GJC-V-23.D
Sample Name: GJC-V-23

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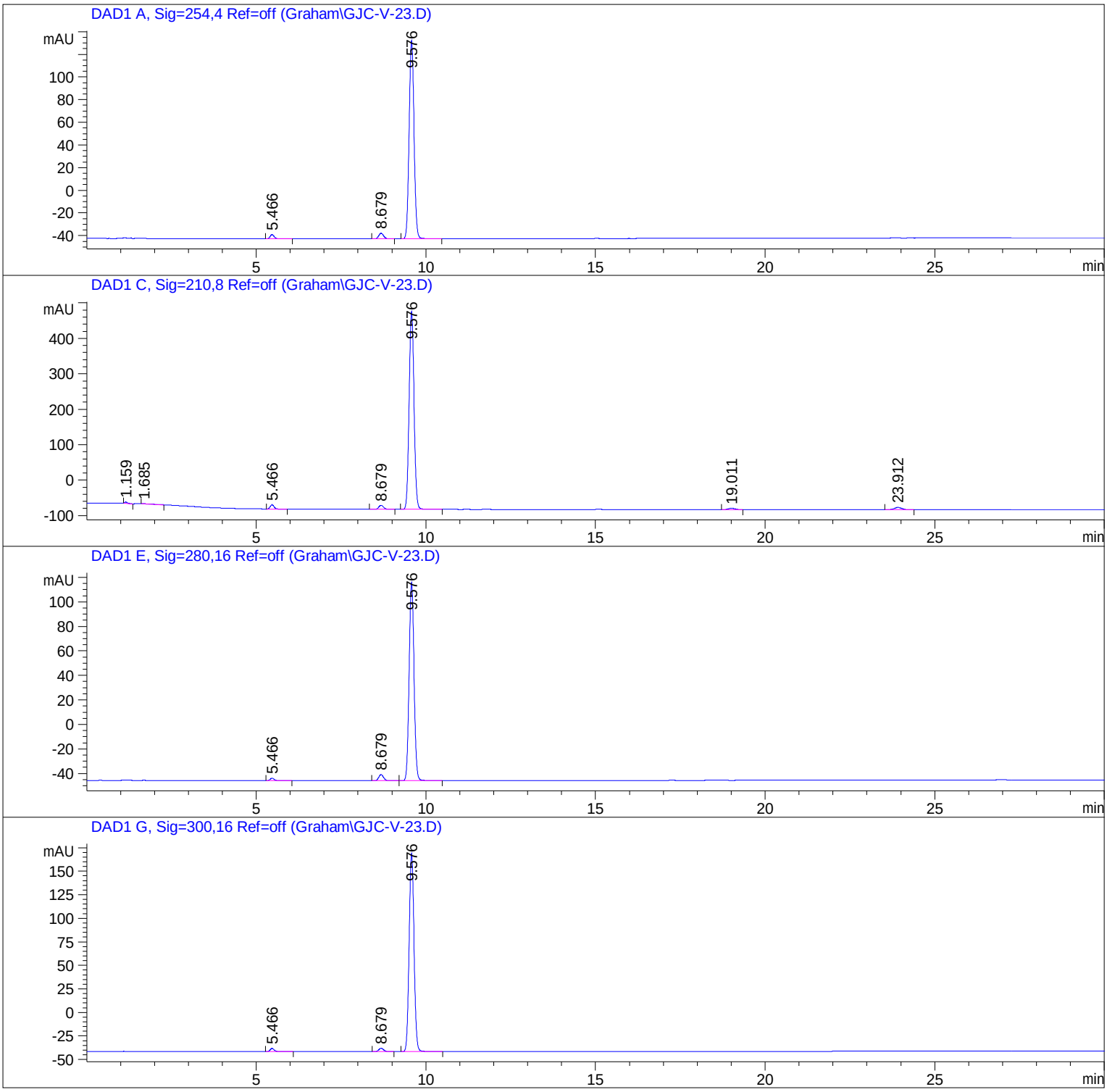
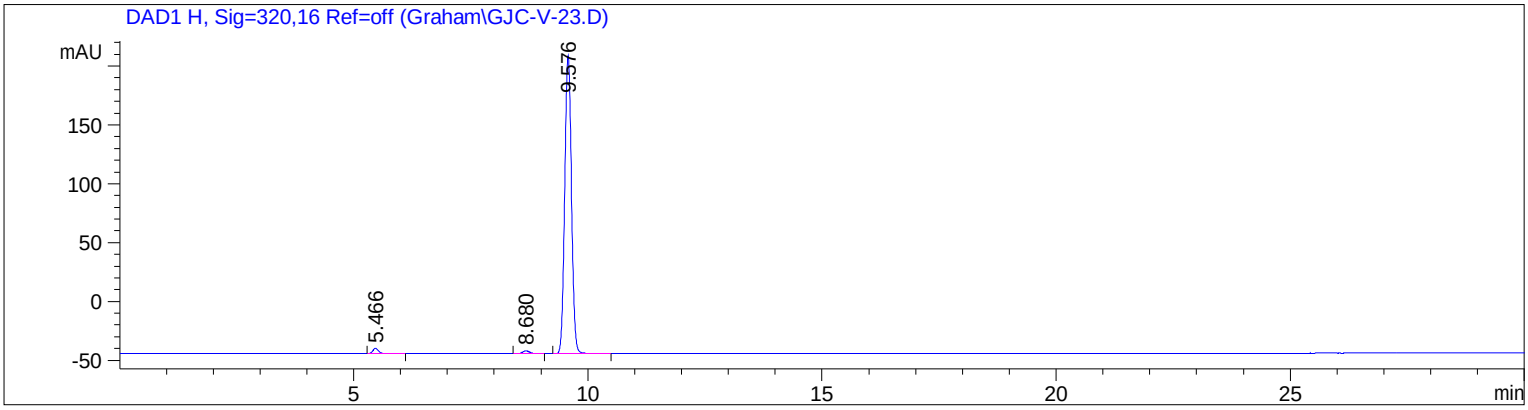
Sample Operator : SYSTEM
Acq. Instrument : 1200 HPLC Location : 1
Injection Date : 9/17/2018 9:18:54 AM
Inj Volume : No inj
Method : C:\CHEM32\1\METHODS\GRAD 2 50-90 ACN.M
Last changed : 4/30/2014 1:53:57 AM by ERICAP
Sample Info : GJC-V-23



Data File C:\Chem32\1\Data\Graham\GJC-V-23.D
Sample Name: GJC-V-23



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Area Percent Report

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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount: : 1.00000 [ng/ul] (not used in calc.)
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	5.466	BB	0.1260	29.46091	3.65507	1.6151
2	8.679	BB	0.1504	46.85326	4.86342	2.5687
3	9.576	BB	0.1565	1747.72717	174.95860	95.8162
Totals :				1824.04134	183.47709	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.159	BB	0.1027	31.65245	4.46615	0.5287
2	1.685	BB	0.1069	8.58727	1.10171	0.1434
3	5.466	BB	0.1238	97.24998	12.08947	1.6244
4	8.679	BB	0.1505	111.34425	11.54910	1.8598
5	9.576	BB	0.1567	5590.34766	558.67175	93.3778
6	19.011	BB	0.2133	54.21047	4.01961	0.9055
7	23.912	BB	0.2355	93.41354	6.14022	1.5603
Totals :				5986.80562	598.03802	

Data File C:\Chem32\1\Data\Graham\GJC-V-23.D
Sample Name: GJC-V-23

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.466	BB	0.1242	16.29162	2.01851	0.9694
2	8.679	BB	0.1502	47.54305	4.94169	2.8290
3	9.576	BB	0.1566	1616.71216	161.82237	96.2016
Totals :				1680.54683	168.78258	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.466	BB	0.1265	27.24419	3.36390	1.2582
2	8.679	BB	0.1503	34.04884	3.53687	1.5724
3	9.576	BB	0.1566	2104.09570	210.61261	97.1694
Totals :				2165.38873	217.51337	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.466	BB	0.1264	36.78322	4.54569	1.4202
2	8.680	BB	0.1511	23.06023	2.37809	0.8903
3	9.576	BB	0.1566	2530.20117	253.26511	97.6895
Totals :				2590.04462	260.18889	

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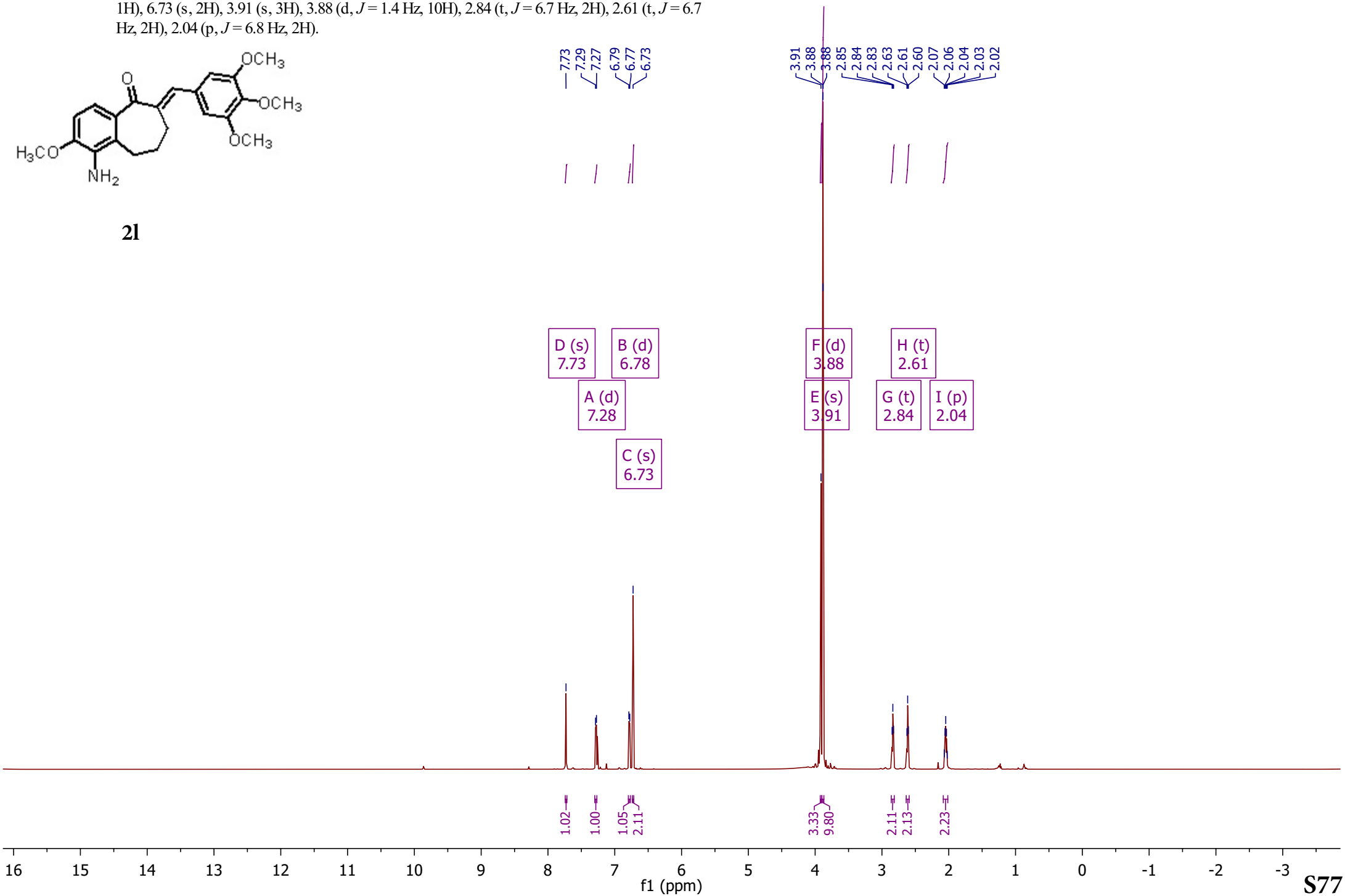
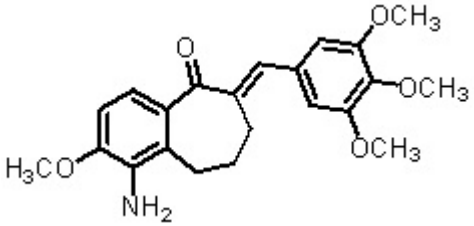
*** End of Report ***

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.73 (s, 1H), 7.28 (d, *J* = 8.4 Hz, 1H), 6.78 (d, *J* = 8.4 Hz, 1H), 6.73 (s, 2H), 3.91 (s, 3H), 3.88 (d, *J* = 1.4 Hz, 10H), 2.84 (t, *J* = 6.7 Hz, 2H), 2.61 (t, *J* = 6.7 Hz, 2H), 2.04 (p, *J* = 6.8 Hz, 2H).

2l



^{13}C NMR (151 MHz, $CDCl_3$) δ 197.29, 153.15, 150.89, 138.51, 137.72, 137.64, 132.72, 132.12, 131.45, 124.22, 120.75, 108.05, 106.77, 60.97, 56.17, 55.74, 25.52, 25.38, 24.64.



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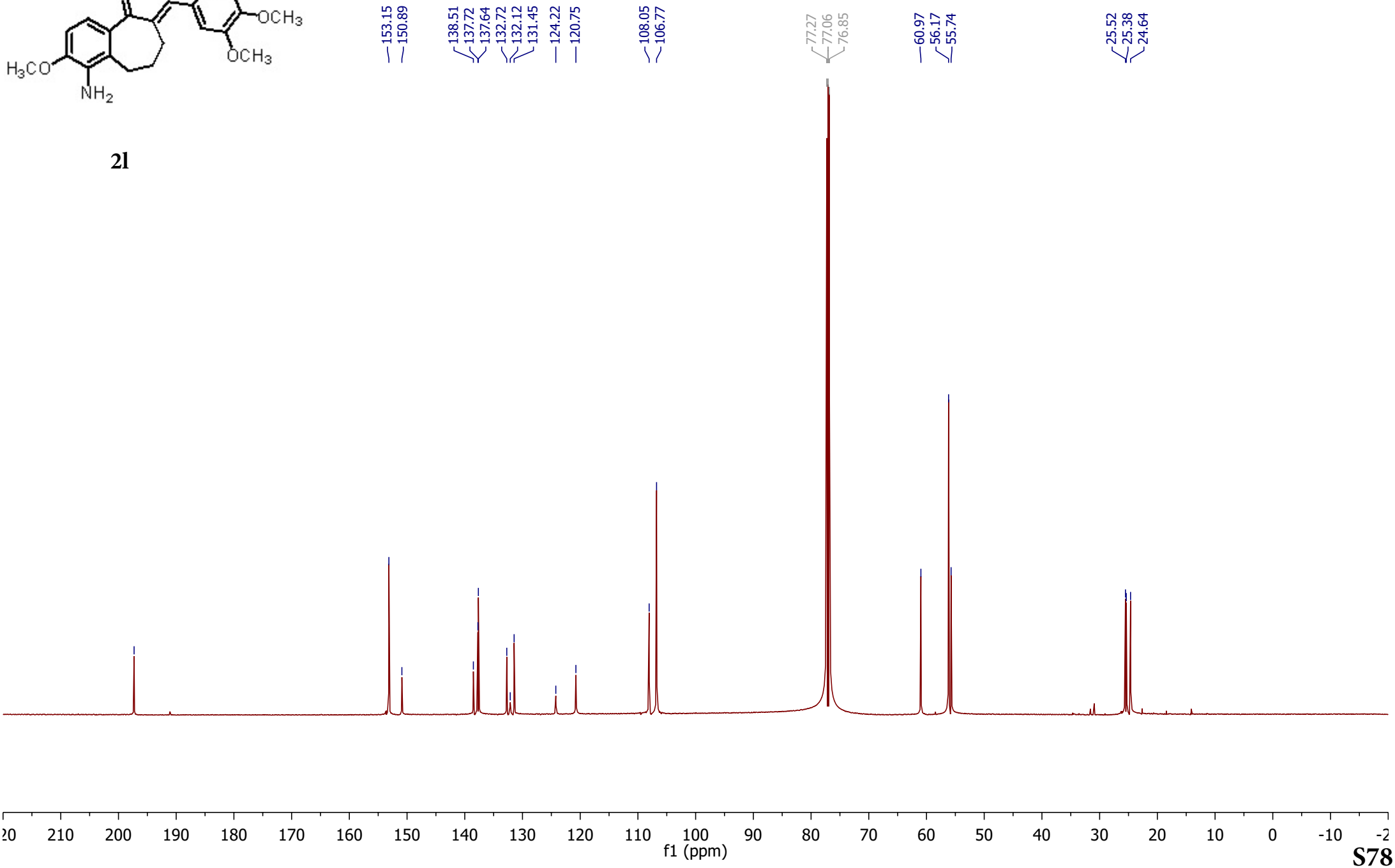

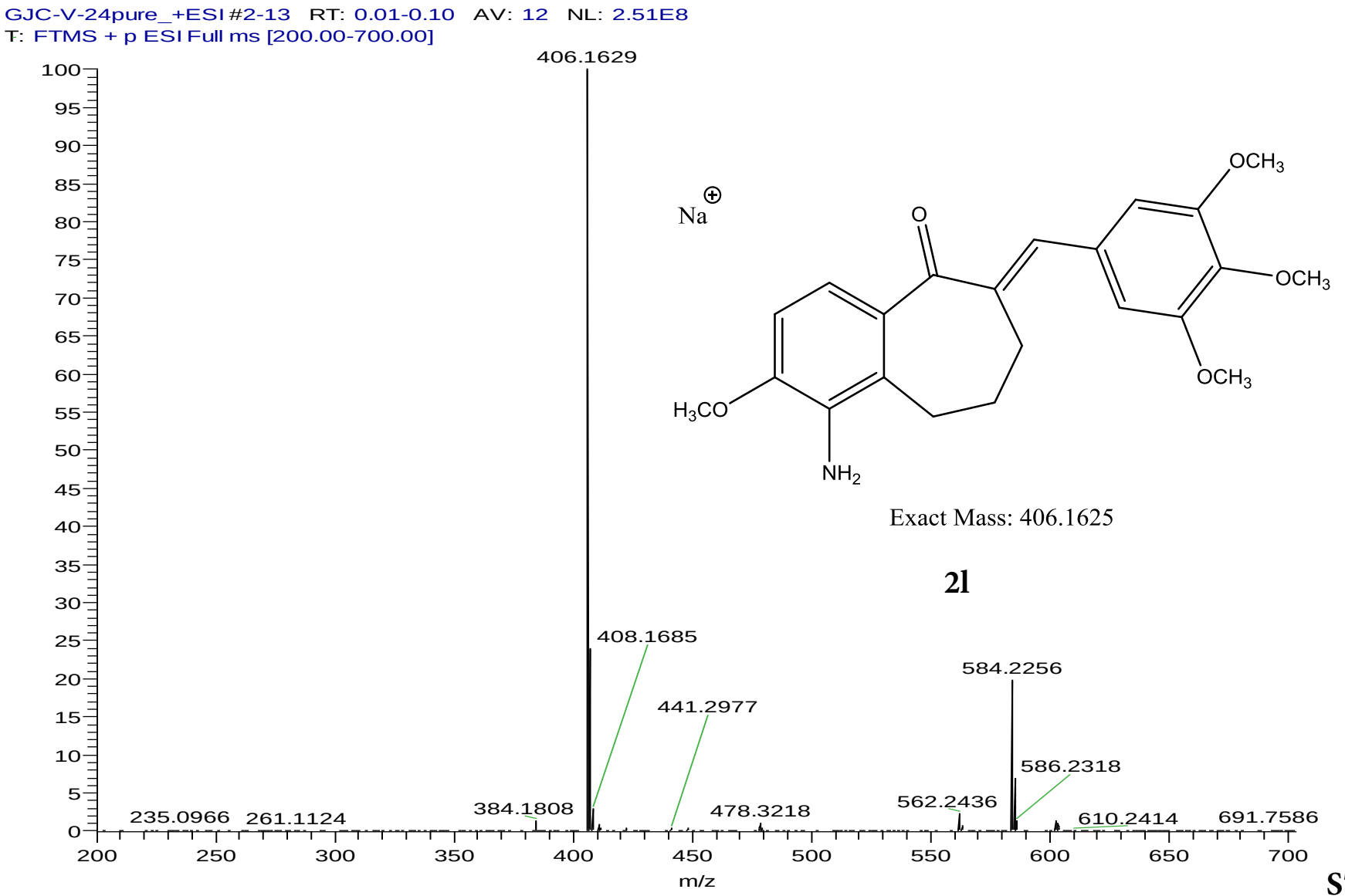

Exact Mass: 406.1625

2l

Data File C:\Chem32\1\Data\Graham\GJC-V-24.D
Sample Name: GJC-V-24

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Inj Volume : No inj
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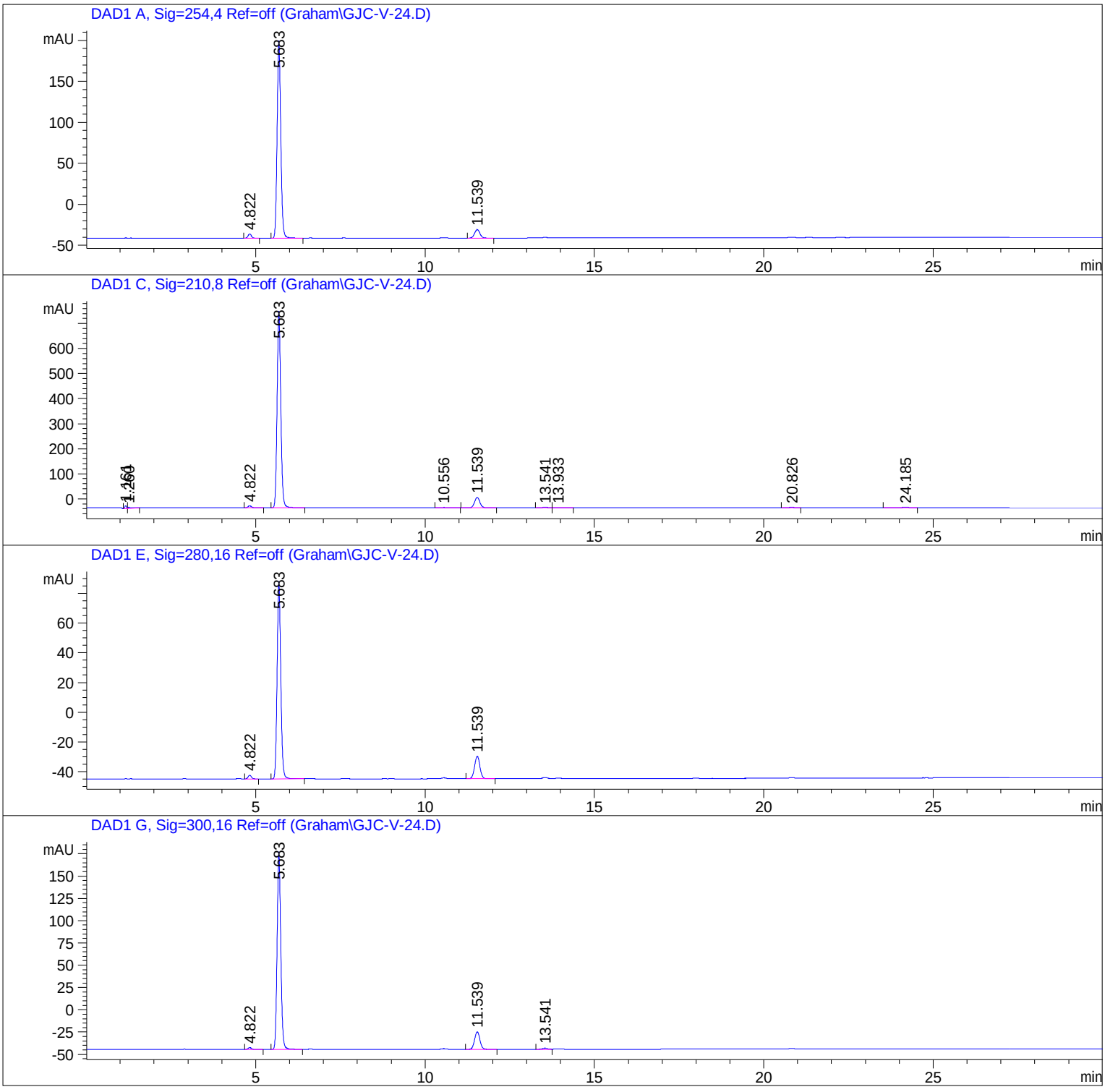
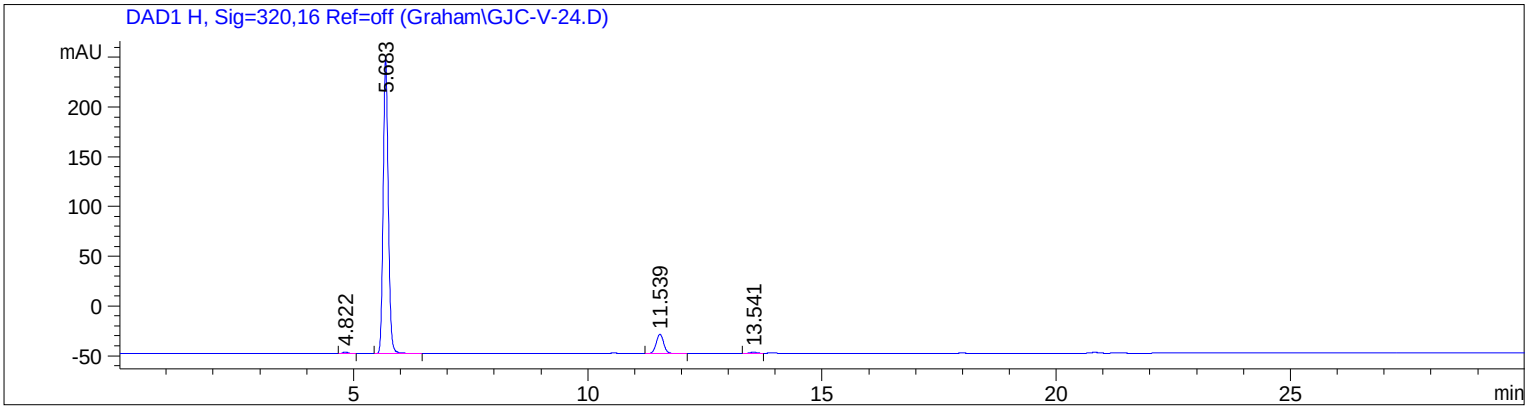
Last changed : 4/30/2014 1:53:57 AM by ERICAP
Sample Info : GJC-V-24



Data File C:\Chem32\1\Data\Graham\GJC-V-24.D
Sample Name: GJC-V-24



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Area Percent Report

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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	4.822	BB	0.1089	36.32634	5.23641	1.8147
2	5.683	BB	0.1196	1852.85059	241.15771	92.5626
3	11.539	BB	0.1699	112.55093	10.26814	5.6227
Totals :				2001.72786	256.66227	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.161	BV	0.0519	39.35026	11.50135	0.5834
2	1.260	VB	0.1305	37.51794	3.69421	0.5562
3	4.822	BB	0.1093	53.79057	7.71610	0.7974
4	5.683	BB	0.1201	6071.85010	786.52057	90.0136
5	10.556	BB	0.1659	13.30080	1.23265	0.1972
6	11.539	BB	0.1721	452.61765	41.22018	6.7099
7	13.541	BV	0.1608	23.49886	2.26855	0.3484
8	13.933	VB	0.1927	12.93293	1.02841	0.1917
9	20.826	BB	0.1722	17.17011	1.53900	0.2545
10	24.185	BB	0.2341	23.45098	1.55344	0.3477
Totals :				6745.48019	858.27445	

Data File C:\Chem32\1\Data\Graham\GJC-V-24.D
Sample Name: GJC-V-24

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.822	BB	0.1086	17.36822	2.51328	1.4417
2	5.683	BB	0.1197	1021.80127	132.88614	84.8190
3	11.539	BB	0.1719	165.51494	15.09705	13.7393
Totals :				1204.68443	150.49647	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.822	BB	0.1090	14.28299	2.05690	0.7342
2	5.683	BB	0.1197	1705.28369	221.87492	87.6558
3	11.539	BB	0.1720	213.89748	19.49762	10.9949
4	13.541	BB	0.1563	11.96817	1.20101	0.6152
Totals :				1945.43233	244.63045	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.822	BB	0.1087	11.57703	1.67318	0.4563
2	5.683	BB	0.1197	2301.58008	299.38916	90.7060
3	11.539	BB	0.1720	212.27731	19.34487	8.3659
4	13.541	BB	0.1563	11.97176	1.20110	0.4718
Totals :				2537.40619	321.60831	

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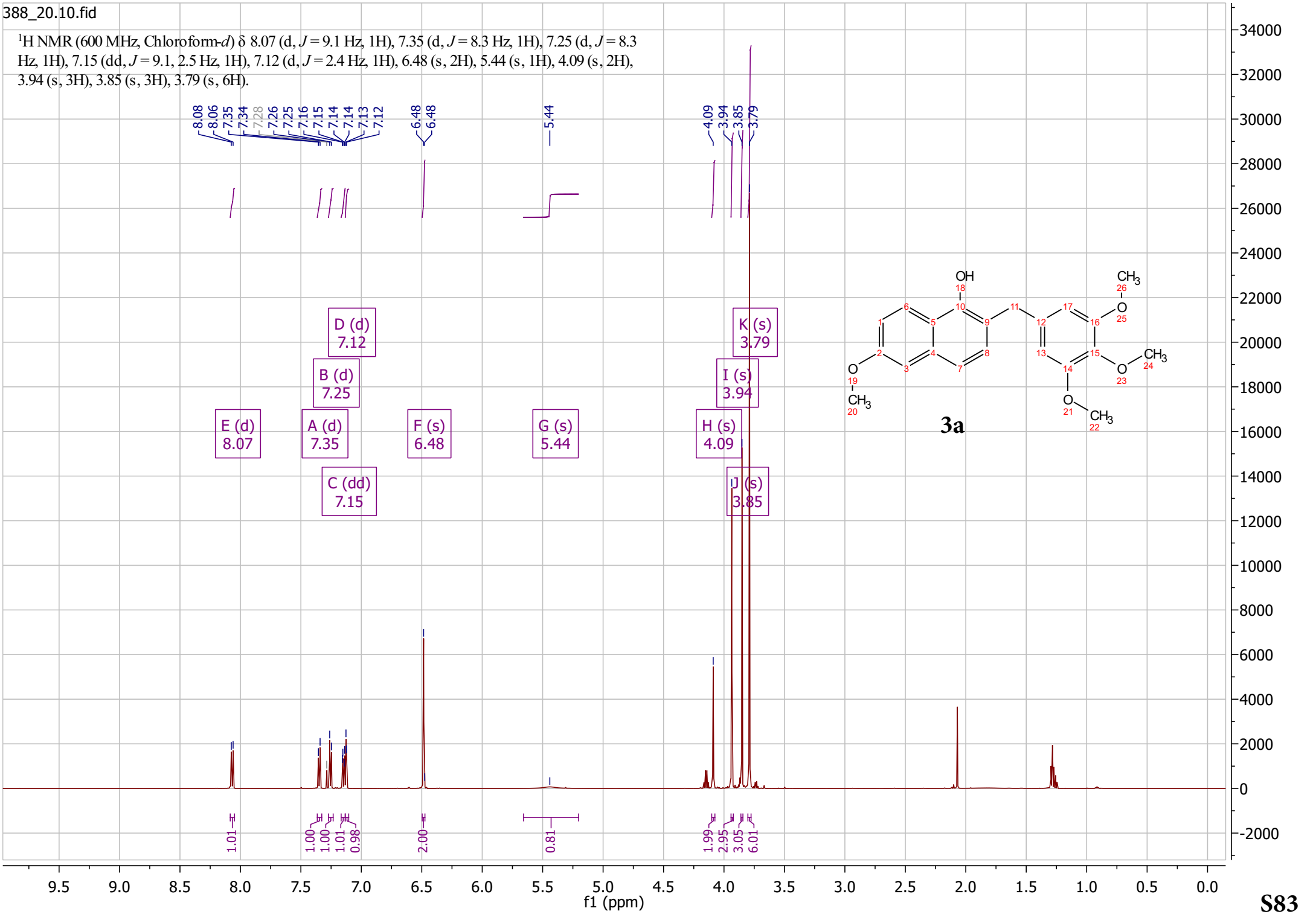
*** End of Report ***

388_20.10.fid

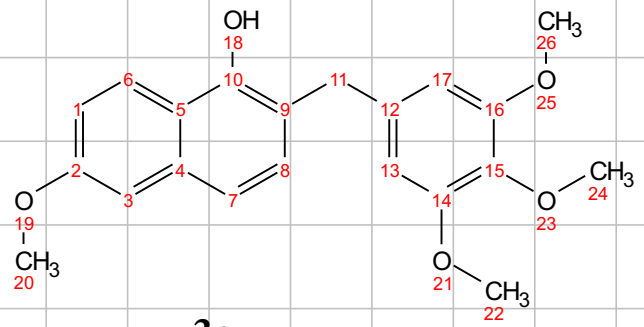
^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.07 (d, *J* = 9.1 Hz, 1H), 7.35 (d, *J* = 8.3 Hz, 1H), 7.25 (d, *J* = 8.3 Hz, 1H), 7.15 (dd, *J* = 9.1, 2.5 Hz, 1H), 7.12 (d, *J* = 2.4 Hz, 1H), 6.48 (s, 2H), 5.44 (s, 1H), 4.09 (s, 2H), 3.94 (s, 3H), 3.85 (s, 3H), 3.79 (s, 6H).



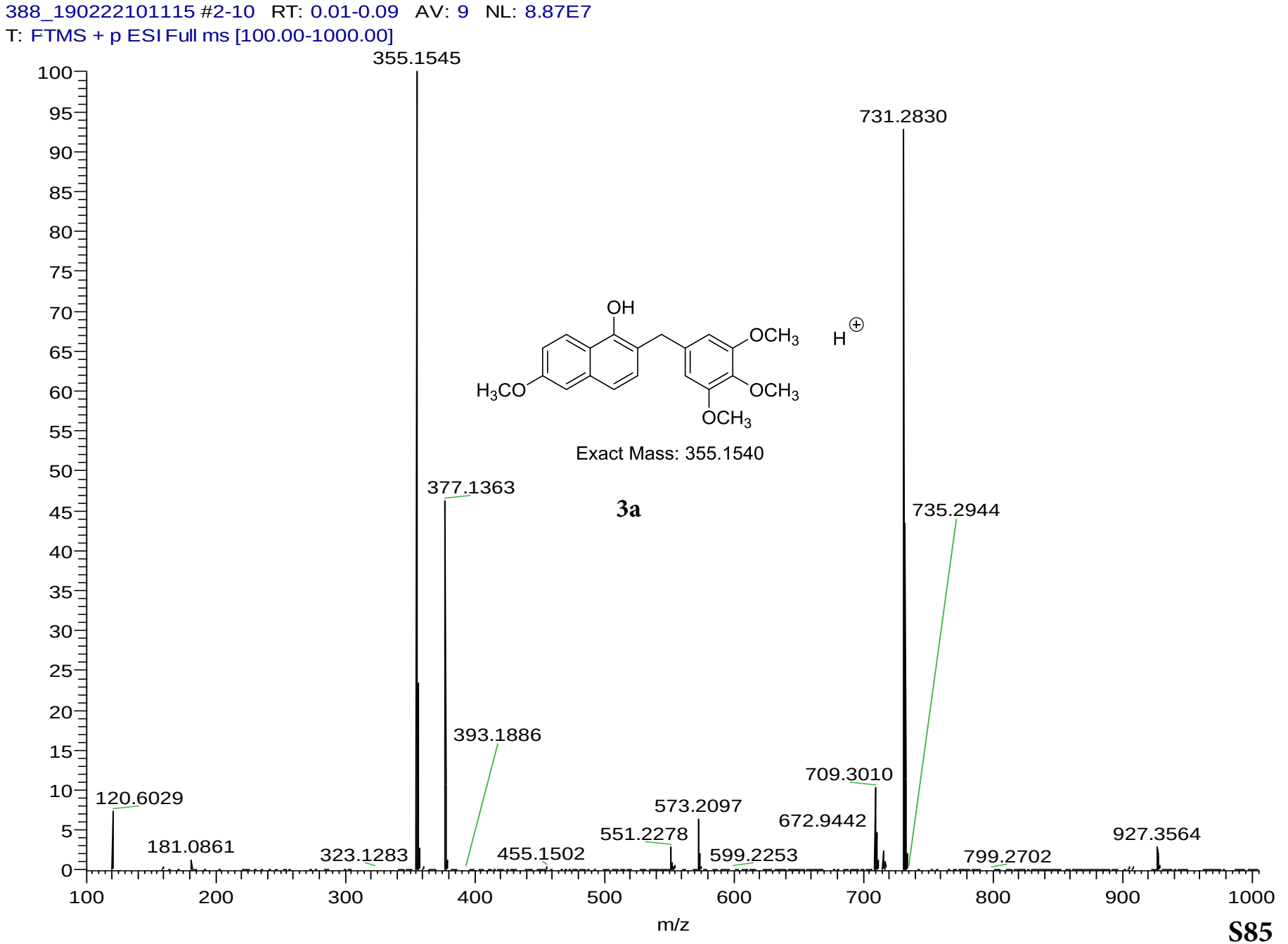
3a

388_20_CARBON_001
388_20

^{13}C NMR (126 MHz, cdcl$_3$) δ 157.70, 153.54, 149.50, 136.60, 135.29, 135.15, 129.47, 123.04, 120.22, 119.30, 117.92, 117.70, 105.65, 105.38, 60.87, 56.05, 55.27, 36.99.



3a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Exact Mass: 355.1540
3a
m/z

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Sample Name: 388

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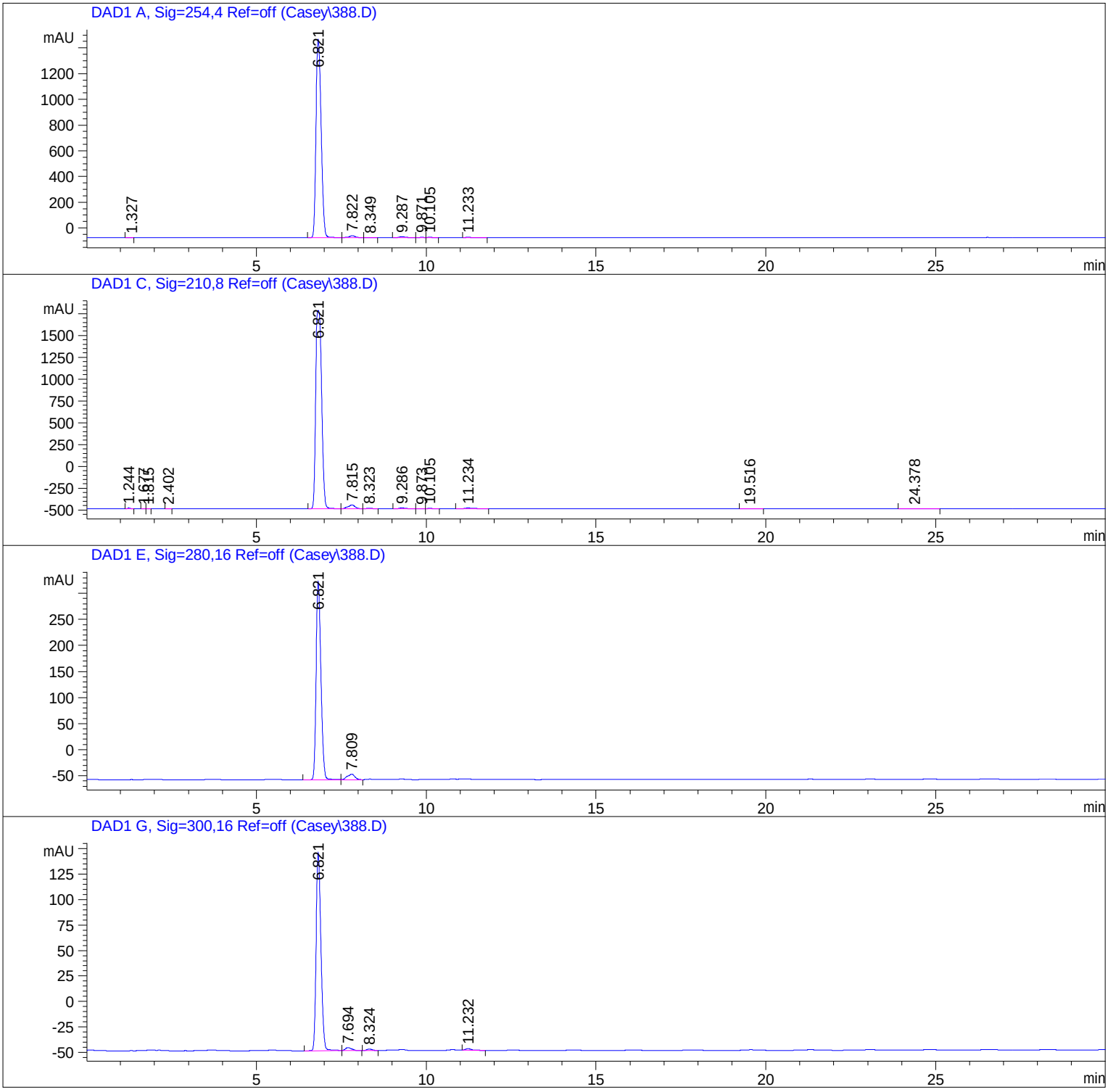
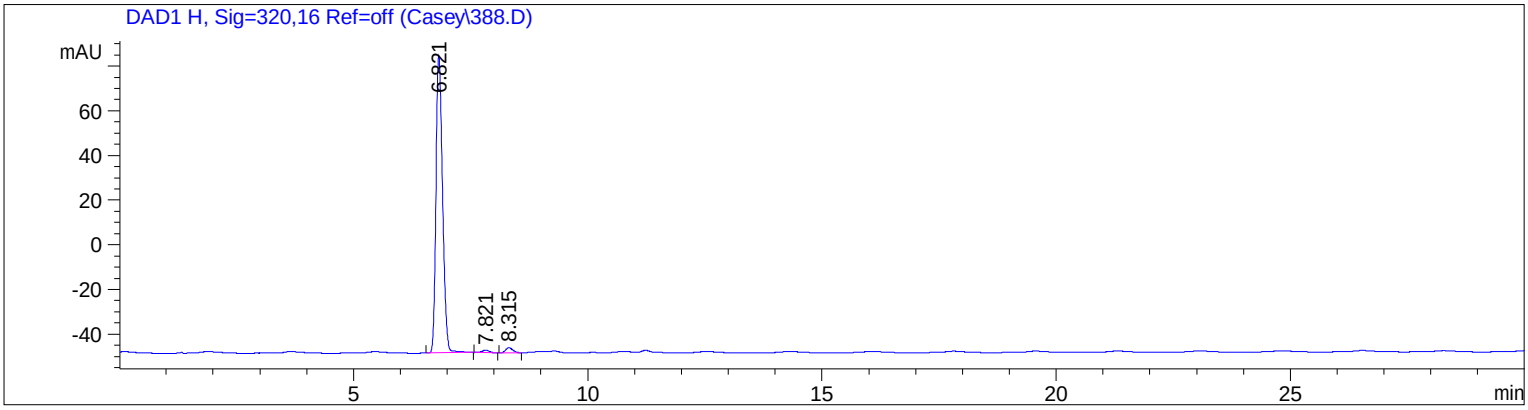
Method : C:\CHEM32\1\METHODS\GRAD 2 50-90 ACN.M
Last changed : 4/30/2014 1:53:57 AM by ERICAP



Data File C:\Chem32\1\Data\Casey\388.D
Sample Name: 388



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Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount: : 1.00000 [ng/ul] (not used in calc.)
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	1.327	BB	0.0738	5.66940	1.10691	0.0341
2	6.821	BV	0.1693	1.63038e4	1542.74524	97.9839
3	7.822	VB	0.1831	178.49548	14.55577	1.0727
4	8.349	BB	0.1806	12.12921	1.08340	0.0729
5	9.287	BB	0.1842	81.28567	6.76178	0.4885
6	9.871	BV	0.1546	17.51337	1.72290	0.1053
7	10.105	VB	0.1696	21.67645	1.92178	0.1303
8	11.233	BB	0.1822	18.68954	1.53368	0.1123

Totals : 1.66393e4 1571.43146

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.244	BV	0.0795	42.79947	7.86610	0.1495
2	1.677	BV	0.0732	9.35360	2.05018	0.0327
3	1.815	VB	0.0753	5.16686	1.09038	0.0180
4	2.402	BB	0.0794	8.76913	1.72260	0.0306
5	6.821	BV	0.1933	2.75210e4	2271.99487	96.1279
6	7.815	VV	0.2027	631.03979	44.69862	2.2042
7	8.323	VB	0.1844	53.60891	4.58698	0.1873
8	9.286	BB	0.1832	110.19287	9.10461	0.3849
9	9.873	BV	0.1552	18.75301	1.89960	0.0655
10	10.105	VB	0.1737	33.31054	2.95030	0.1164

Data File C:\Chem32\1\Data\Casey\388.D
Sample Name: 388

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
11	11.234	BB	0.2202	131.50397	8.60933	0.4593
12	19.516	BB	0.2384	23.76127	1.53699	0.0830
13	24.378	BB	0.2811	40.29131	2.10654	0.1407
Totals :				2.86295e4	2360.21712	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.821	BV	0.1522	3726.99829	380.75418	95.8223
2	7.809	VB	0.2149	162.49236	10.71655	4.1777
Totals :				3889.49065	391.47073	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.821	BV	0.1527	1915.24451	194.88666	96.1489
2	7.694	VB	0.1976	41.68980	3.08558	2.0929
3	8.324	BB	0.1783	17.93777	1.58126	0.9005
4	11.232	BB	0.1757	17.08527	1.44736	0.8577
Totals :				1991.95734	201.00085	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.821	BB	0.1524	1307.19165	133.26089	97.1100
2	7.821	BB	0.1693	13.80997	1.22717	1.0259
3	8.315	BB	0.1708	25.09184	2.30910	1.8640
Totals :				1346.09346	136.79716	

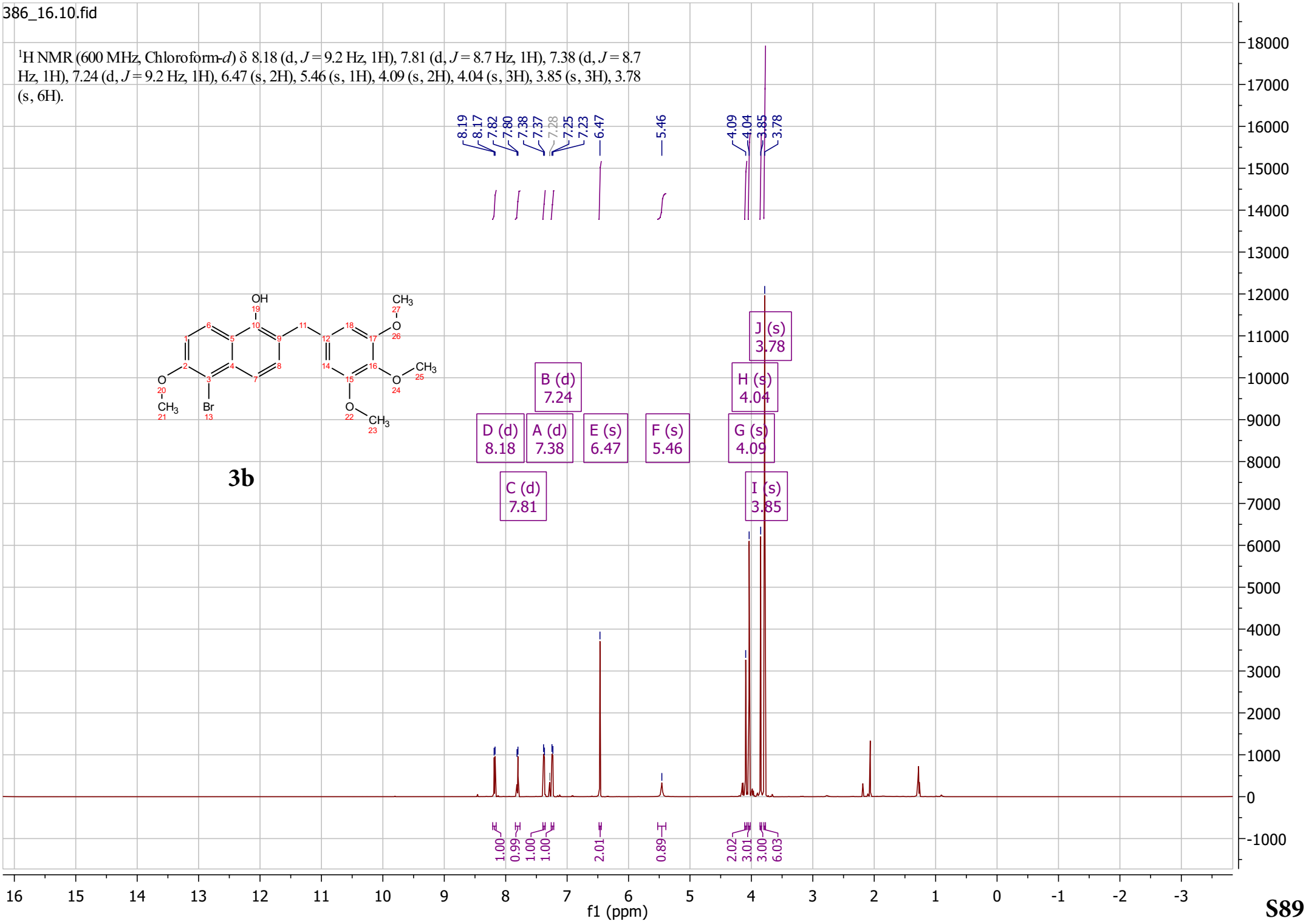
==========

*** End of Report ***

386_16.10.fid

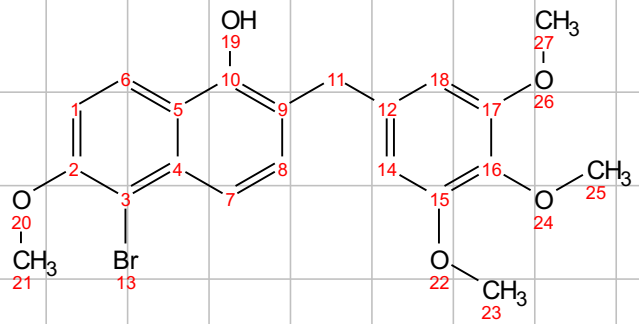
^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.18 (d, *J* = 9.2 Hz, 1H), 7.81 (d, *J* = 8.7 Hz, 1H), 7.38 (d, *J* = 8.7 Hz, 1H), 7.24 (d, *J* = 9.2 Hz, 1H), 6.47 (s, 2H), 5.46 (s, 1H), 4.09 (s, 2H), 4.04 (s, 3H), 3.85 (s, 3H), 3.78 (s, 6H).

3b



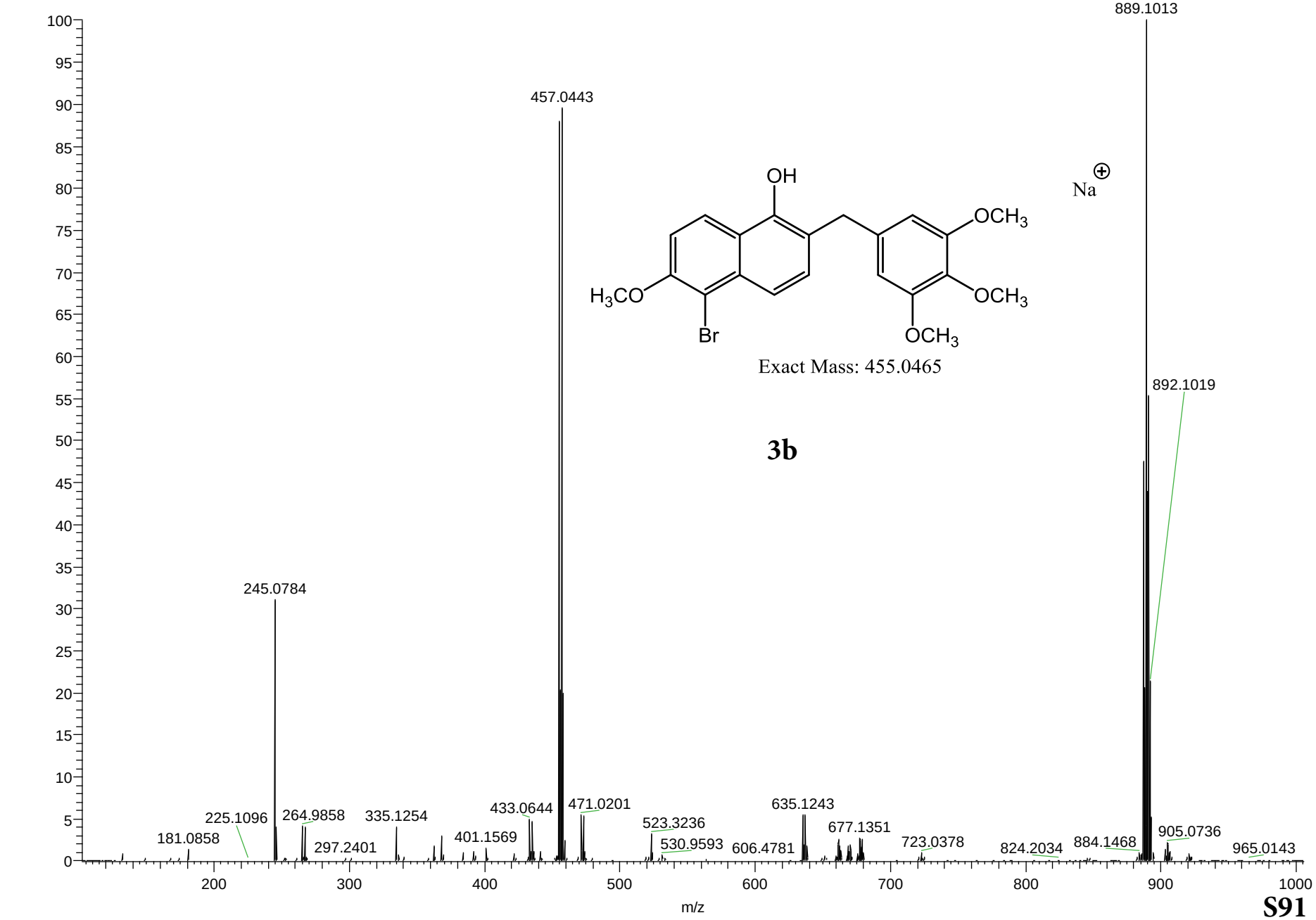
386_16_CARBON_001
386_16

^{13}C NMR (126 MHz, cdcl$_3$) δ 153.87, 153.64, 149.50, 136.76, 134.66, 133.62, 130.81, 122.76, 121.43, 118.65, 118.00, 112.65, 108.30, 105.33, 60.87, 56.98, 56.08, 37.07.



3b

386_+ESI #2-14 RT: 0.01-0.11 AV: 13 NL: 7.02E7
T: FTMS + p ESI Full ms [100.00-1000.00]



Exact Mass: 455.0465

3b

Data File C:\Chem32\1\Data\Casey\386.D
Sample Name: 386

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Acq. Instrument : 1200 HPLC Location : 1
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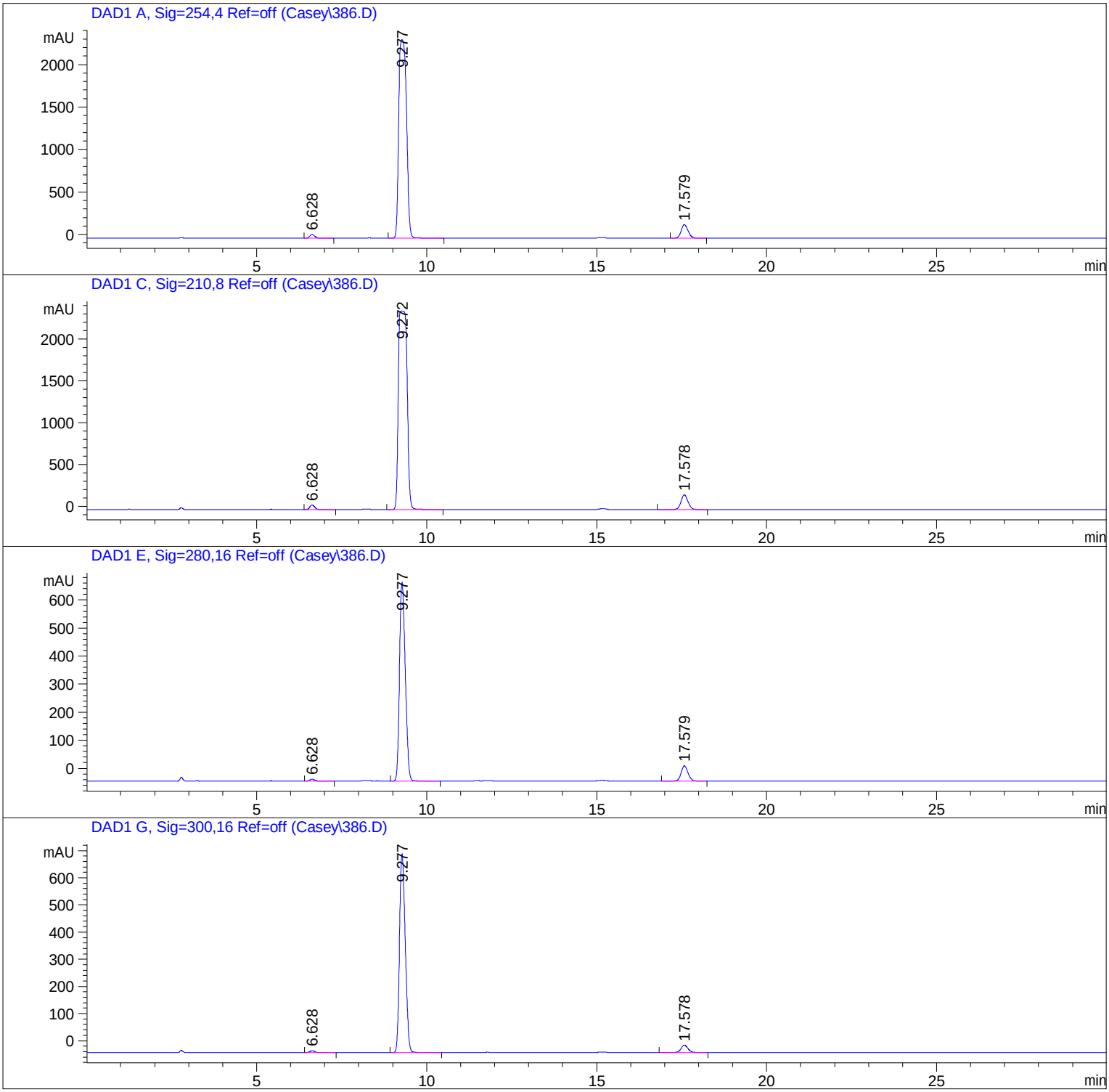
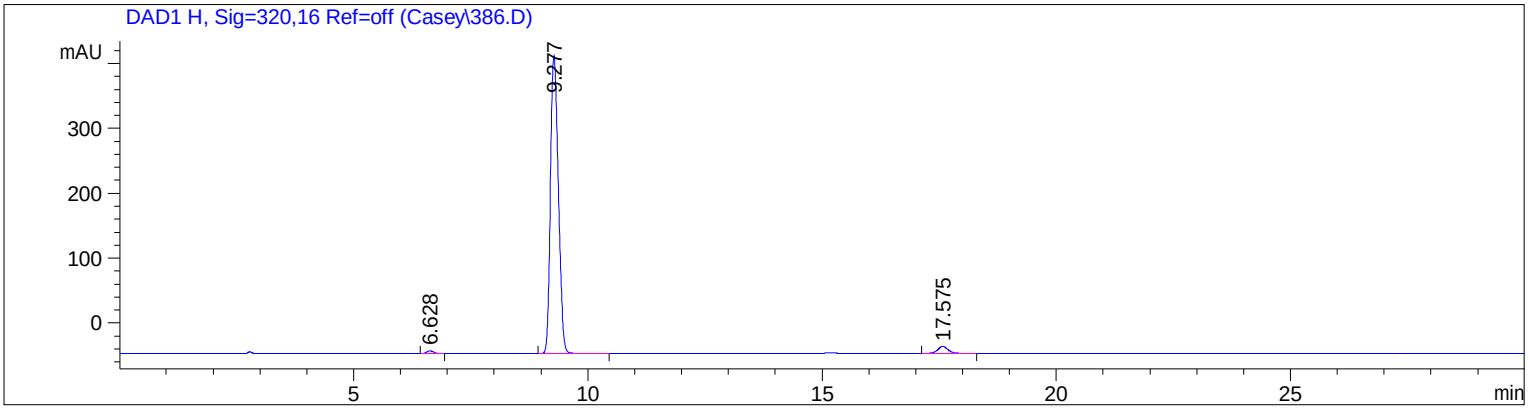
Inj Volume : No inj
Acq. Method : C:\CHEM32\1\METHODS\GRAD 2 50-90 ACN.M
Last changed : 4/30/2014 1:53:57 AM by ERICAP
Analysis Method : C:\CHEM32\1\METHODS\RT-ACNWASH 2.M
Last changed : 7/9/2015 2:27:22 PM by Blake
Method Info : General Column Wash Method

Additional Info : Peak(s) manually integrated



Data File C:\Chem32\1\Data\Casey\386.D
Sample Name: 386



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Area Percent Report

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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount: : 1.00000 [ng/ul] (not used in calc.)
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	6.628	BB	0.1540	433.52261	43.58640	1.0949
2	9.277	BB	0.2604	3.69235e4	2336.57349	93.2509
3	17.579	BB	0.2165	2238.82104	160.76668	5.6542
Totals :				3.95958e4	2540.92656	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.628	BB	0.1549	567.55383	56.64471	1.2536
2	9.272	BV	0.2892	4.21166e4	2371.83789	93.0283
3	17.578	BB	0.2232	2588.74756	178.50819	5.7181
Totals :				4.52729e4	2606.99080	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.628	BB	0.1559	68.22257	6.75187	0.7445
2	9.277	BB	0.1833	8330.97461	707.65771	90.9153

Data File C:\Chem32\1\Data\Casey\386.D
Sample Name: 386

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
3	17.579	BB	0.2183	764.25000	54.25961	8.3402
Totals :				9163.44718	768.66919	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.628	BB	0.1542	67.52934	6.77785	0.7461
2	9.277	BB	0.1830	8586.25293	731.17389	94.8637
3	17.578	BB	0.2231	397.36887	27.41063	4.3903
Totals :				9051.15114	765.36237	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.628	BB	0.1517	41.28418	4.23686	0.7368
2	9.277	BB	0.1829	5391.39795	459.33972	96.2257
3	17.575	BB	0.2338	170.18208	11.04479	3.0374
Totals :				5602.86421	474.62137	

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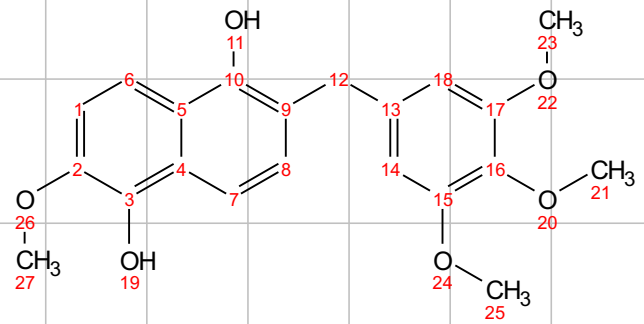
*** End of Report ***

CJM-VI-413_14.10.fid

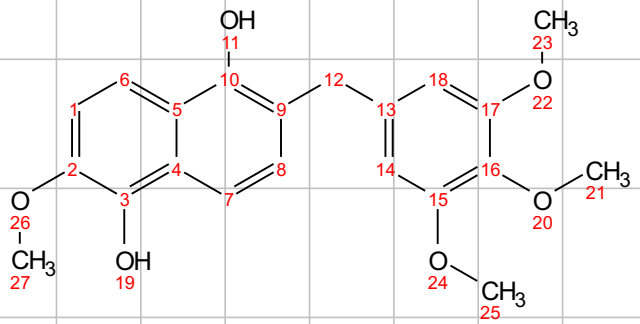
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.75 (d, *J* = 8.6 Hz, 1H), 7.68 (d, *J* = 9.1 Hz, 1H), 7.27 (d, 1H), 7.26 (d, *J* = 9.1 Hz, 1H), 6.49 (s, 2H), 6.03 (s, 1H), 5.22 (s, 1H), 4.10 (s, 2H), 4.02 (s, 3H), 3.85 (s, 3H), 3.80 (s, 6H).



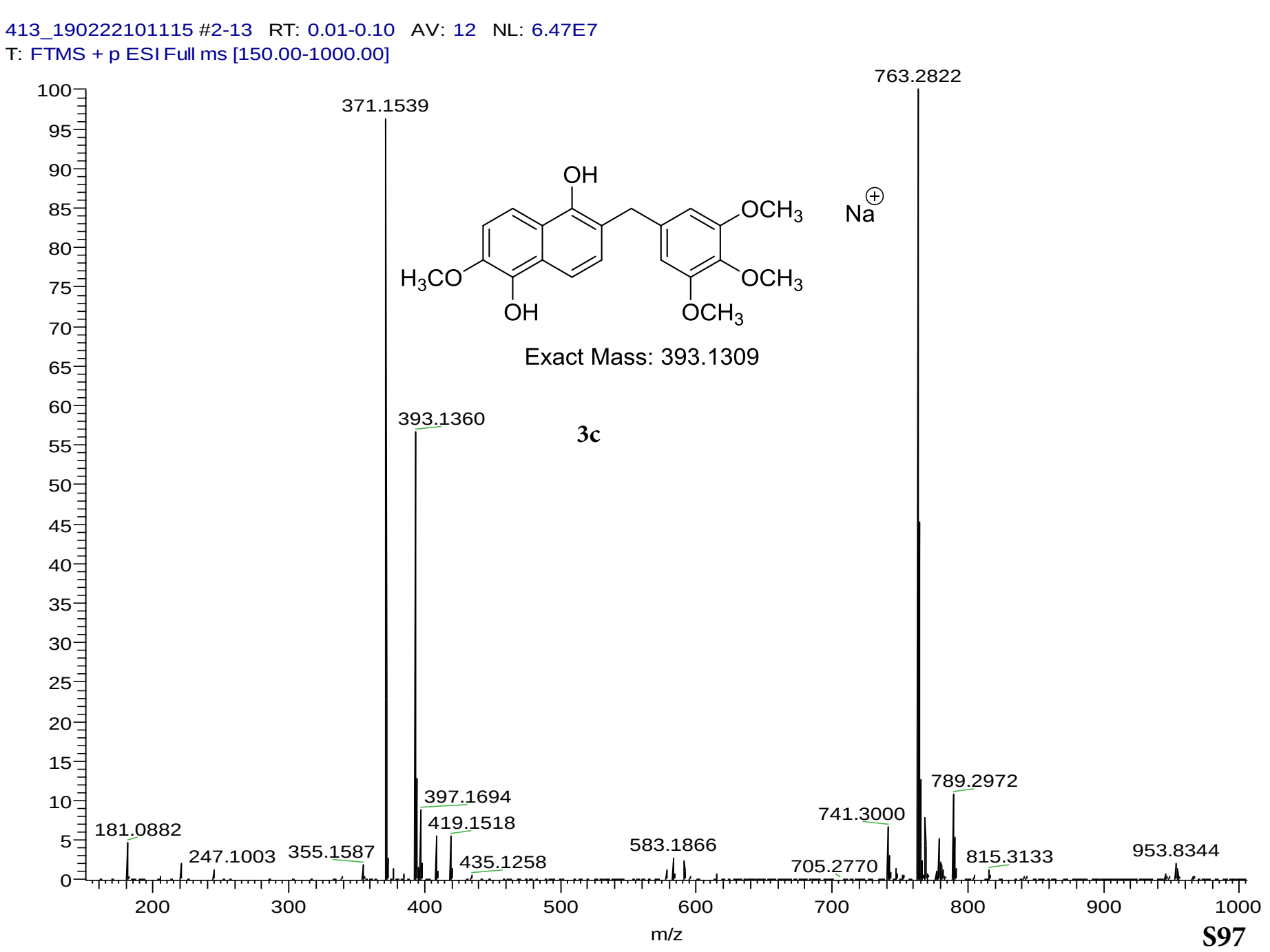
3c

CJM-VI-413_14.11.fid

^{13}C NMR (151 MHz, $CDCl_3$) δ 153.63, 149.10, 141.45, 139.64, 136.74, 135.07, 128.63, 124.50, 121.17, 117.85, 113.82, 113.03, 112.50, 105.38, 60.87, 57.10, 56.10, 37.22.



3c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371.1539
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OCH3
Na
H3CO
OCH3
OH
OCH3
Exact Mass: 393.1309
393.1360
3c
397.1694
419.1518
789.2972
741.3000
181.0882
247.1003
355.1587
435.1258
583.1866
705.2770
815.3133
953.8344
100
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
200
300
400
500
600
700
800
900
1000
m/z

Data File C:\Chem32\1\Data\Casey\413.D
Sample Name: 413

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Sample Operator : SYSTEM
Acq. Instrument : 1200 HPLC                    Location :   1
Injection Date  : 12/13/2018 4:35:36 PM
                                               Inj Volume : No inj
Acq. Method     : C:\CHEM32\1\METHODS\GRAD 2 50-90 ACN.M
Last changed    : 12/13/2018 2:18:35 PM by SYSTEM
                  (modified after loading)
Analysis Method : C:\Chem32\1\Methods\GRAD 2 30-90 ACN.M
Last changed    : 4/2/2014 4:04:33 PM by ERICA P
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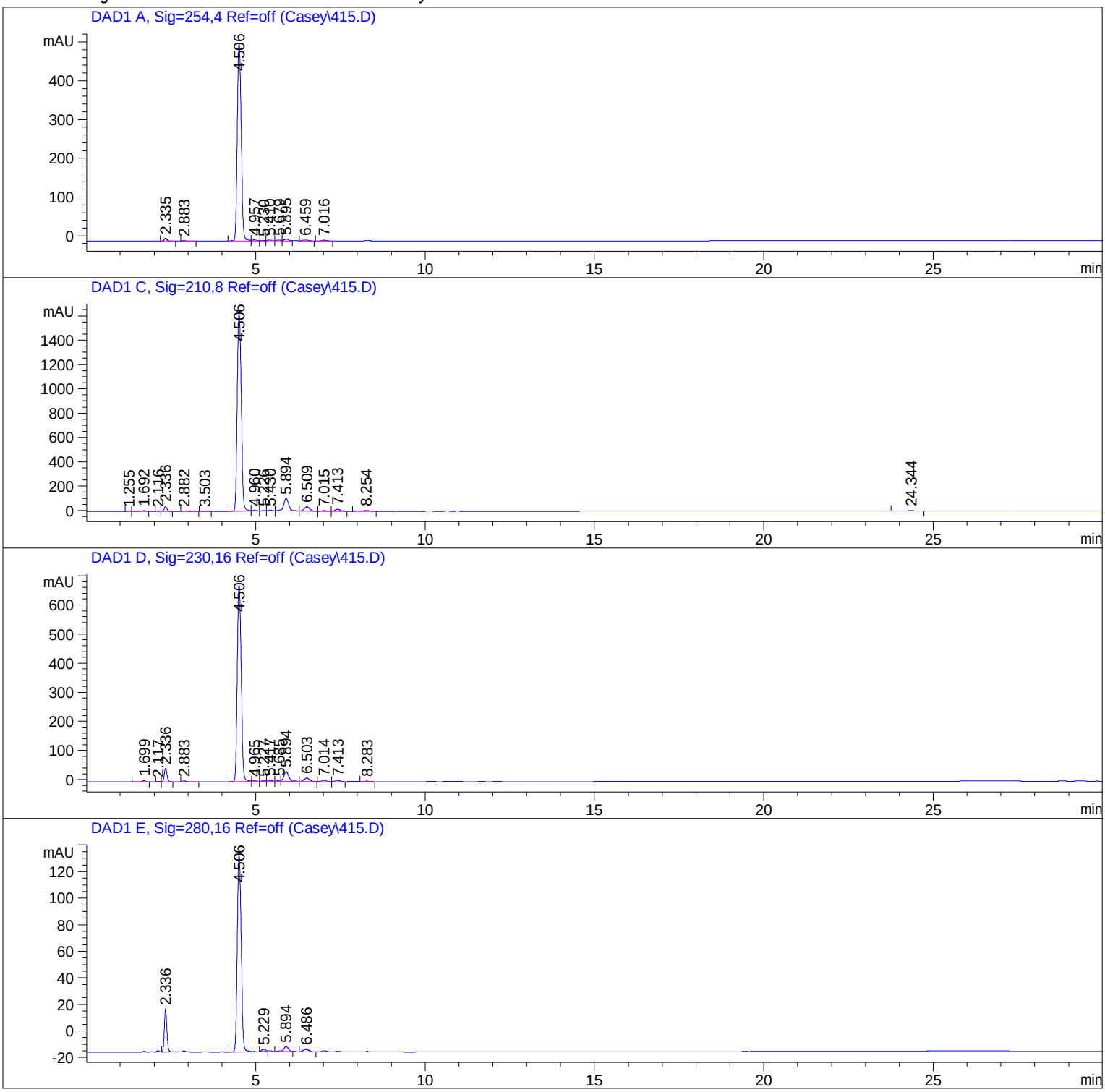
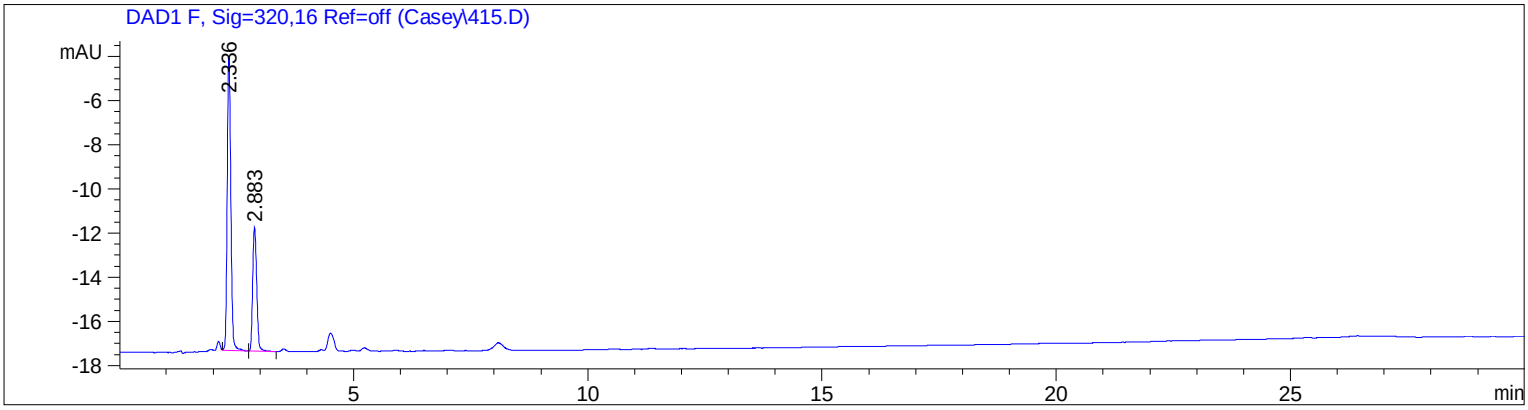


Data File C:\Chem32\1\Data\Casey\413.D
Sample Name: 413



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Area Percent Report

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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount: : 1.00000 [ng/ul] (not used in calc.)
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	2.335	BB	0.0864	40.36160	7.31525	0.9475
2	2.883	BB	0.0905	5.81169	1.01894	0.1364
3	4.506	BV	0.1269	4066.91528	510.60535	95.4732
4	4.957	VB	0.1354	21.81107	2.56115	0.5120
5	5.230	BV	0.1110	7.84239	1.10287	0.1841
6	5.410	VB	0.1318	17.56228	2.09553	0.4123
7	5.679	BV	0.1280	17.25841	2.14259	0.4052
8	5.895	VV	0.1481	39.42757	4.17746	0.9256
9	6.459	BB	0.1774	25.72060	2.31832	0.6038
10	7.016	BB	0.1573	17.03463	1.66608	0.3999

Totals : 4259.74551 535.00354

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.255	BV	0.0732	8.98394	1.83386	0.0568
2	1.692	VB	0.1149	51.83678	6.24023	0.3275
3	2.116	VV	0.0798	9.51839	1.85735	0.0601
4	2.336	VB	0.0844	225.22386	42.10829	1.4228
5	2.882	BB	0.1010	27.46944	4.16226	0.1735
6	3.503	BB	0.1565	10.42461	1.00921	0.0659
7	4.506	BV	0.1321	1.33919e4	1625.72400	84.5993
8	4.960	VB	0.1373	65.06580	7.64234	0.4110

Data File C:\Chem32\1\Data\Casey\413.D
Sample Name: 413

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
9	5.226	BV	0.1133	35.57837	4.86340	0.2248
10	5.430	VV	0.1483	83.05911	8.78724	0.5247
11	5.894	VV	0.1713	1158.25635	104.48417	7.3170
12	6.509	VB	0.1796	423.79041	36.45633	2.6772
13	7.015	BV	0.1570	48.97085	4.96838	0.3094
14	7.413	VB	0.1667	167.58316	16.20480	1.0587
15	8.254	BB	0.2341	88.28725	5.84887	0.5577
16	24.344	BB	0.2689	33.84544	1.94566	0.2138
Totals :				1.58298e4	1874.13638	

Signal 3: DAD1 D, Sig=230,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.699	BB	0.0904	24.94578	4.01840	0.3895
2	2.117	BV	0.0798	5.76358	1.12462	0.0900
3	2.336	VV	0.0829	241.05840	46.15130	3.7638
4	2.883	BB	0.1031	20.85190	3.07579	0.3256
5	4.506	BV	0.1272	5431.01807	679.65100	84.7982
6	4.965	VB	0.1386	20.74427	2.40613	0.3239
7	5.227	BV	0.1118	17.73738	2.46890	0.2769
8	5.417	VB	0.1350	34.50676	4.06643	0.5388
9	5.685	BV	0.0996	23.81562	3.67533	0.3718
10	5.894	VB	0.1621	349.85504	33.98846	5.4625
11	6.503	BB	0.1766	136.62740	11.84486	2.1333
12	7.014	BB	0.1584	39.55059	3.96565	0.6175
13	7.413	BB	0.1640	39.94331	3.95069	0.6237
14	8.283	BB	0.2057	18.22395	1.45917	0.2845
Totals :				6404.64204	801.84671	

Signal 4: DAD1 E, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.336	VB	0.0825	164.58078	31.74378	11.5805
2	4.506	BB	0.1266	1181.62817	148.81528	83.1433
3	5.229	BB	0.1162	11.14974	1.58339	0.7845
4	5.894	BB	0.1597	40.28735	3.86091	2.8348
5	6.486	BB	0.1822	23.54794	2.01643	1.6569
Totals :				1421.19398	188.01979	

Data File C:\Chem32\1\Data\Casey\413.D
Sample Name: 413

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

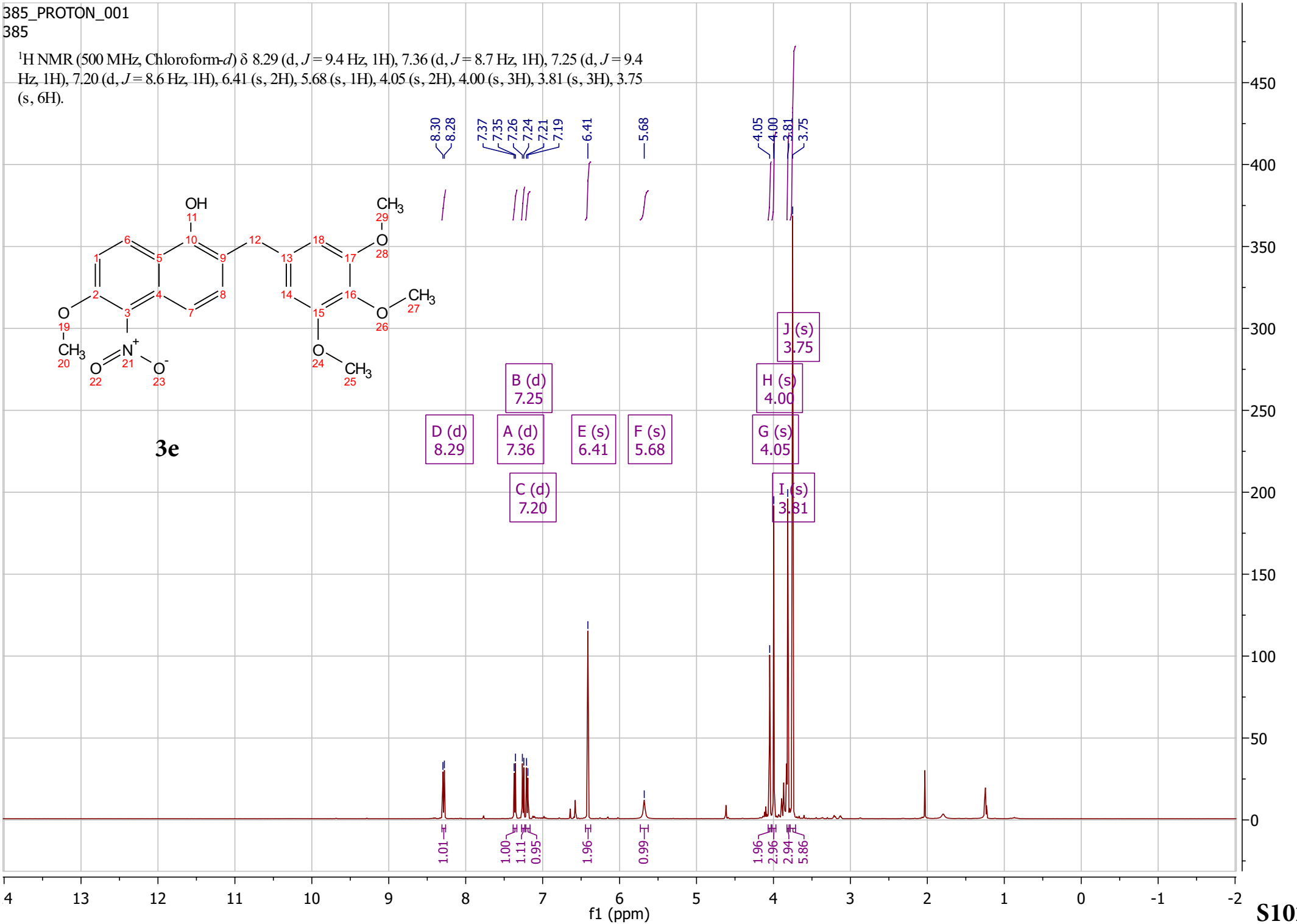
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.336	VB	0.0834	70.39255	13.37653	68.0252
2	2.883	BB	0.0928	33.08754	5.61431	31.9748
Totals :				103.48009	18.99084	

*** End of Report ***

385_PROTON_001
385

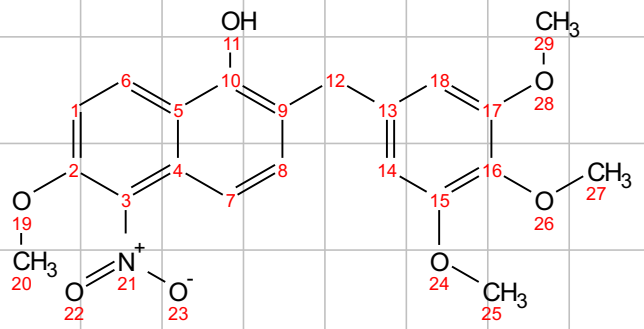
^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.29 (d, *J* = 9.4 Hz, 1H), 7.36 (d, *J* = 8.7 Hz, 1H), 7.25 (d, *J* = 9.4 Hz, 1H), 7.20 (d, *J* = 8.6 Hz, 1H), 6.41 (s, 2H), 5.68 (s, 1H), 4.05 (s, 2H), 4.00 (s, 3H), 3.81 (s, 3H), 3.75 (s, 6H).

3e

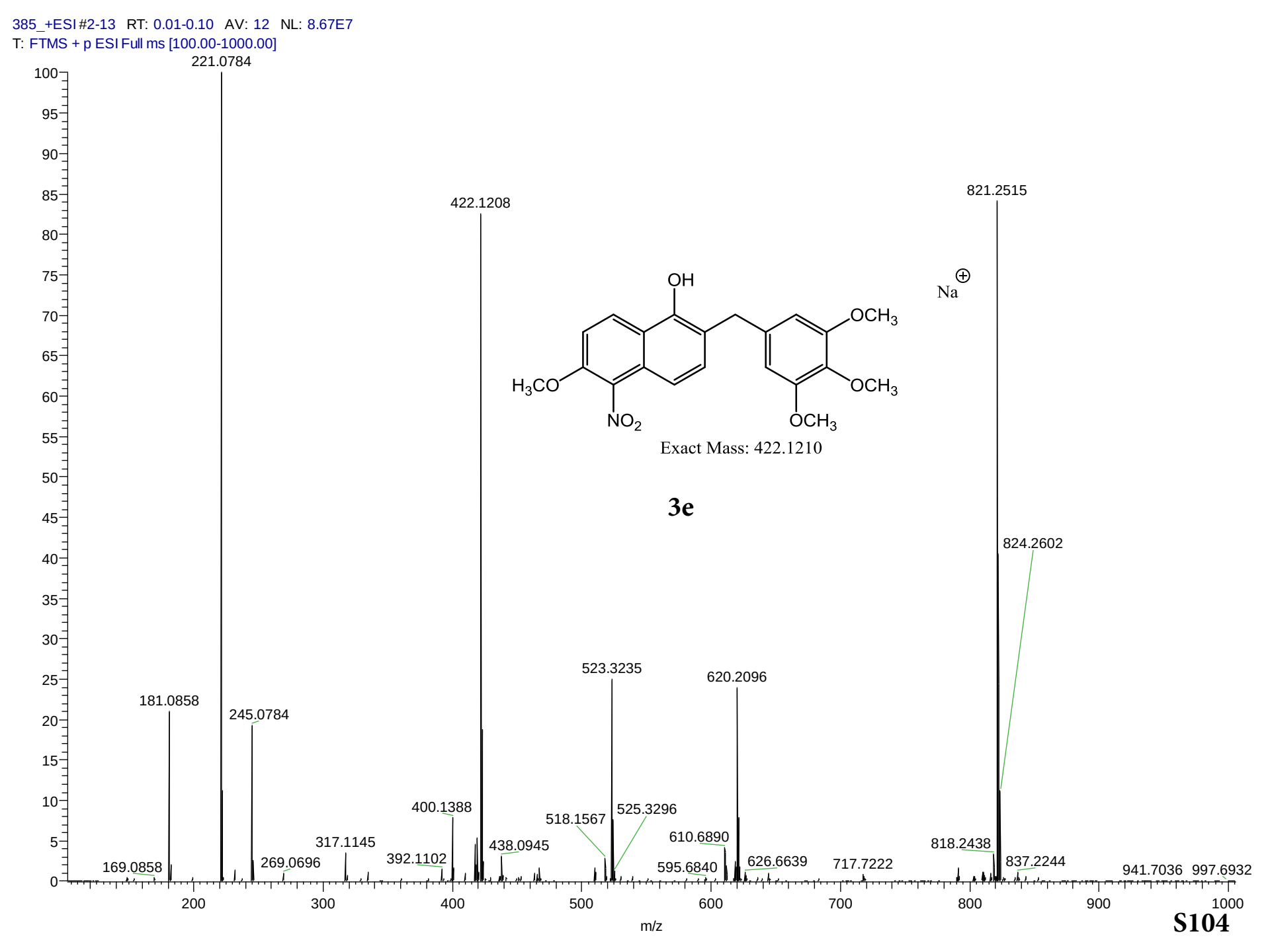


385_CARBON_001
385

^{13}C NMR (126 MHz, cdcl$_3$) δ 153.68, 149.79, 148.68, 136.83, 135.73, 134.22, 132.38, 126.37, 126.16, 120.14, 118.91, 112.61, 112.08, 105.30, 60.88, 56.97, 56.09, 37.00.



3e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OCH3
H3CO
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Exact Mass: 422.1210
m/z

3e

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Sample Name: 385

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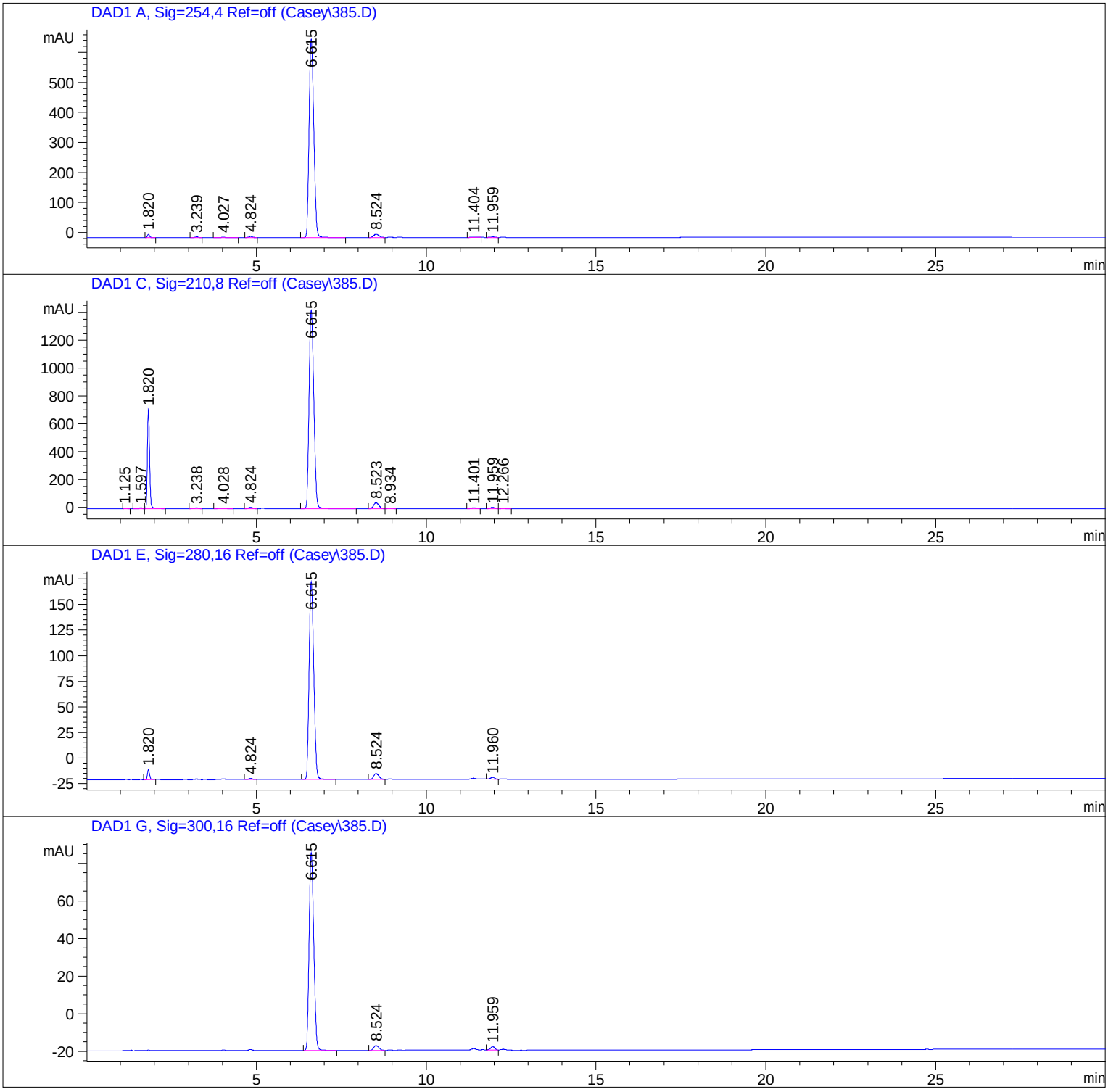
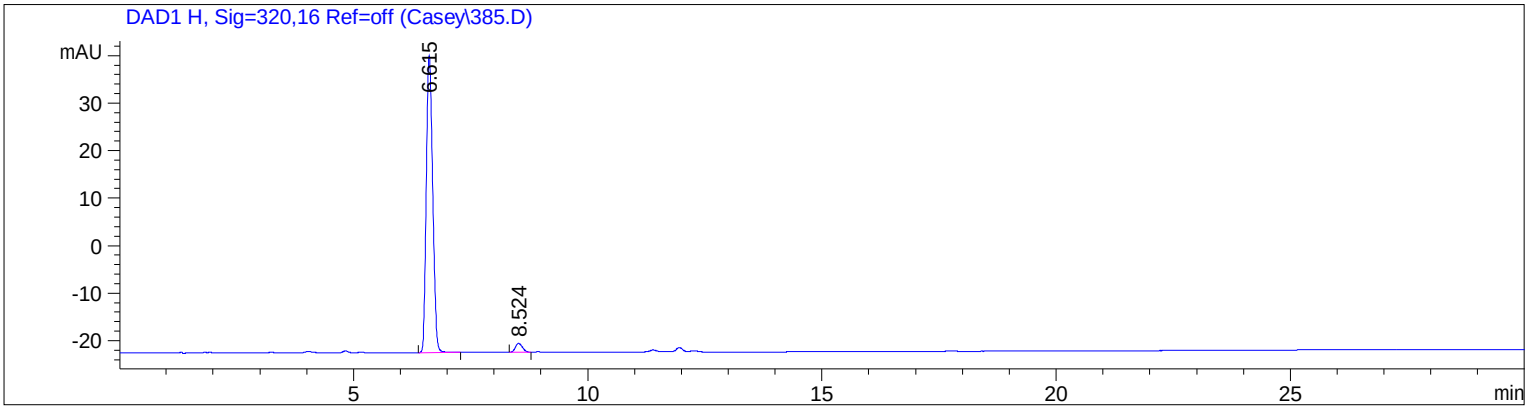
Sample Operator : SYSTEM
Acq. Instrument : 1200 HPLC Location : 1
Injection Date : 10/29/2018 10:22:42 PM
Inj Volume : No inj
Method : C:\CHEM32\1\METHODS\GRAD 2 50-90 ACN.M
Last changed : 4/30/2014 1:53:57 AM by ERICAP



Data File C:\Chem32\1\Data\Casey\385.D
Sample Name: 385



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Area Percent Report

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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount: : 1.00000 [ng/ul] (not used in calc.)
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	1.820	BB	0.0687	48.75412	11.21139	0.7507
2	3.239	BB	0.0983	16.75282	2.70335	0.2580
3	4.027	BB	0.1724	12.59255	1.00377	0.1939
4	4.824	BB	0.1228	30.22308	3.88203	0.4654
5	6.615	BB	0.1480	6242.88916	662.12317	96.1286
6	8.524	BB	0.1638	114.22340	10.94326	1.7588
7	11.404	BB	0.1501	12.50523	1.27847	0.1926
8	11.959	BB	0.1377	16.37278	1.87874	0.2521
Totals :				6494.31313	695.02418	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.125	BV	0.0970	14.09566	1.93520	0.0807
2	1.597	BV	0.0718	32.54264	6.79842	0.1864
3	1.820	VB	0.0697	3134.95337	707.89154	17.9540
4	3.238	BB	0.1132	42.90326	5.61276	0.2457
5	4.028	BB	0.1566	40.05943	3.57813	0.2294
6	4.824	BV	0.1224	66.60140	8.40714	0.3814
7	6.615	BB	0.1489	1.35164e4	1422.48596	77.4090
8	8.523	BV	0.1625	440.48975	42.66014	2.5227
9	8.934	VB	0.1517	24.95214	2.65446	0.1429
10	11.401	BB	0.1469	46.21439	4.95094	0.2647

Data File C:\Chem32\1\Data\Casey\385.D
Sample Name: 385

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
11	11.959	BB	0.1380	79.63085	9.10601	0.4561
12	12.266	BB	0.1580	22.17299	2.26890	0.1270
Totals :				1.74610e4	2218.34960	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.820	BB	0.0691	42.85547	9.78224	2.2112
2	4.824	BB	0.1203	8.85983	1.14441	0.4571
3	6.615	BB	0.1452	1811.47656	193.60114	93.4654
4	8.524	BB	0.1632	60.21402	5.79853	3.1068
5	11.960	BB	0.1375	14.71920	1.69151	0.7595
Totals :				1938.12507	212.01782	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.615	BB	0.1450	984.66321	105.38607	95.9480
2	8.524	BB	0.1609	26.48778	2.55579	2.5810
3	11.959	BB	0.1380	15.09552	1.72628	1.4709
Totals :				1026.24651	109.66814	

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

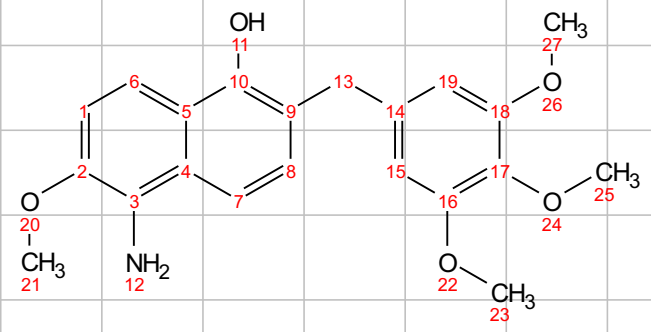
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.615	BB	0.1450	584.62366	62.58048	96.7953
2	8.524	BB	0.1629	19.35582	1.86856	3.2047
Totals :				603.97948	64.44904	

==========

*** End of Report ***

416_7.10.fid

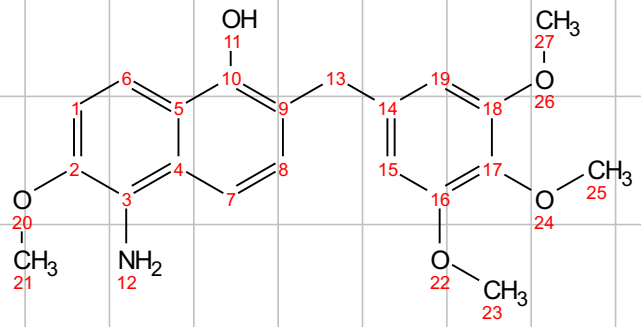
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.60 (d, *J* = 9.1 Hz, 1H), 7.34 (d, *J* = 8.6 Hz, 1H), 7.21 (dd, *J* = 10.9, 8.9 Hz, 2H), 6.47 (s, 2H), 5.67 (s, 1H), 4.27 (s, 2H), 4.08 (s, 2H), 3.96 (s, 3H), 3.84 (s, 3H), 3.77 (s, 6H).



3f

416_7.11.fid

^{13}C NMR (151 MHz, $CDCl_3$) δ 153.47, 149.60, 142.76, 136.55, 135.59, 129.47, 128.16, 124.46, 121.02, 117.71, 112.97, 112.73, 111.68, 105.48, 60.86, 56.63, 56.05, 36.74.



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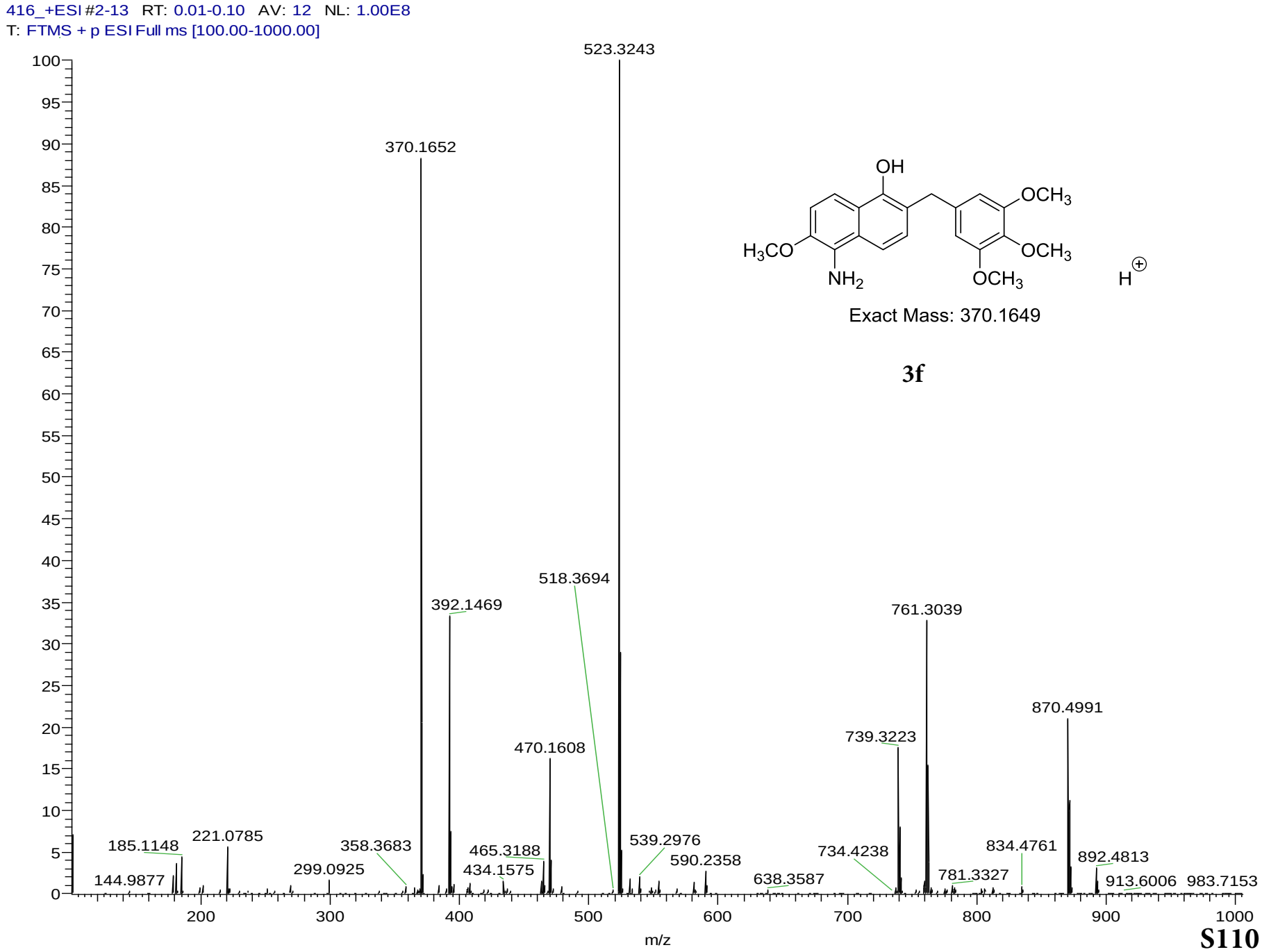
416_+ESI #2-13 RT: 0.01-0.10 AV: 12 NL: 1.00E8
T: FTMS + p ESI Full ms [100.00-1000.00]
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185.1148
221.0785
358.3683
465.3188
539.2976
590.2358
734.4238
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299.0925
434.1575
638.3587
781.3327
913.6006
983.7153
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OCH3
H3CO
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NH2
OCH3
H
Exact Mass: 370.1649
m/z

3f

Data File C:\Chem32\1\Data\Casey\CJM-VII-416.D
Sample Name: CJM-VII-416

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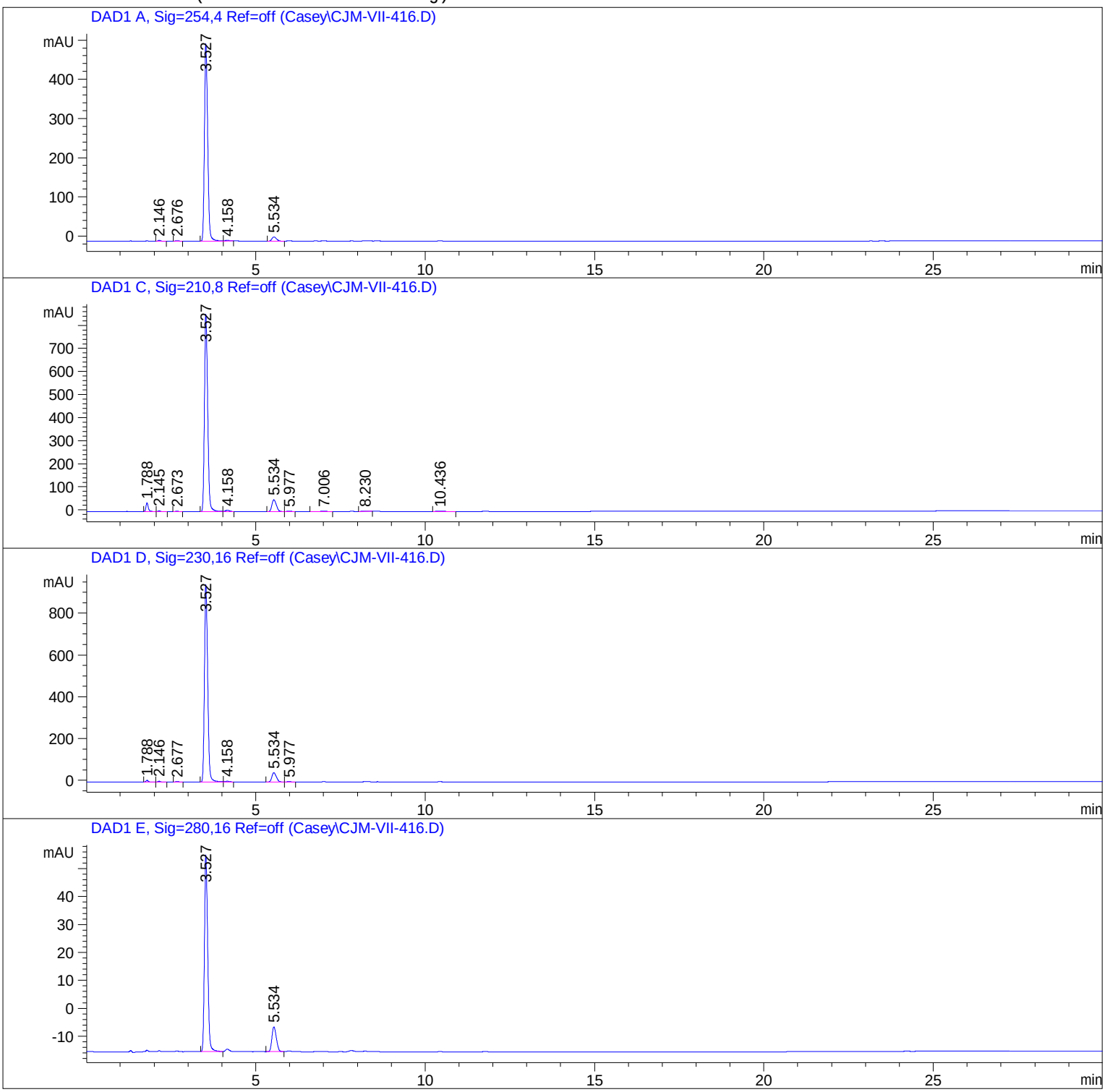
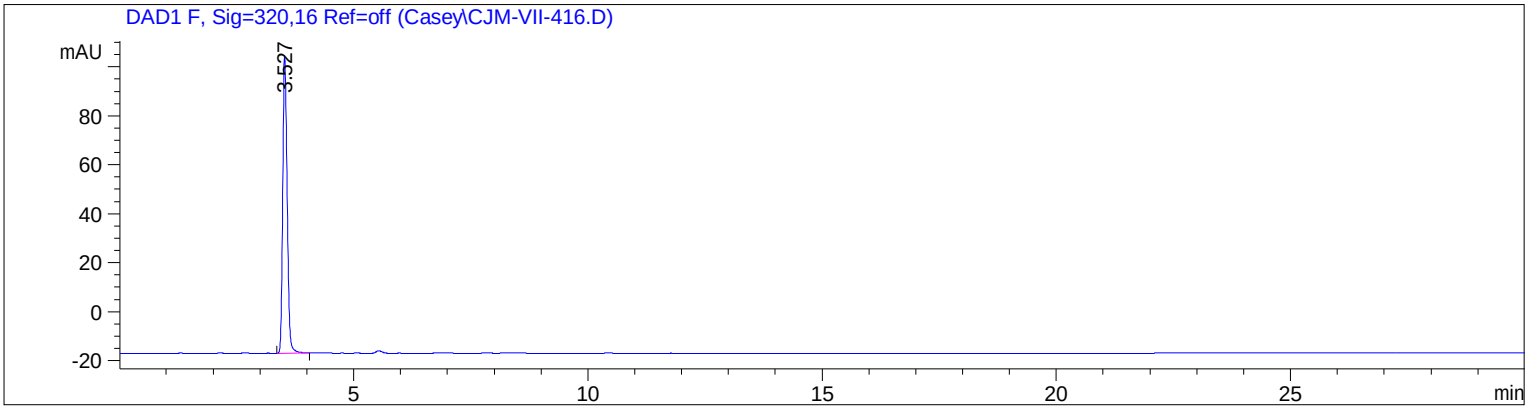
Sample Operator : SYSTEM
Acq. Instrument : 1200 HPLC Location : 1
Injection Date : 12/13/2018 3:02:38 PM
Inj Volume : No inj
Method : C:\CHEM32\1\METHODS\GRAD 2 50-90 ACN.M
Last changed : 12/13/2018 2:18:35 PM by SYSTEM
(modified after loading)



Data File C:\Chem32\1\Data\Casey\CJM-VII-416.D
Sample Name: CJM-VII-416



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Area Percent Report

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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount: : 1.00000 [ng/ul] (not used in calc.)
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	2.146	VB	0.0810	11.62124	2.22432	0.3248
2	2.676	BB	0.0910	7.81121	1.35938	0.2183
3	3.527	BV	0.1076	3445.81201	504.85614	96.3195
4	4.158	VB	0.1229	15.42586	1.97991	0.4312
5	5.534	BB	0.1432	96.81240	10.73545	2.7062
Totals :				3577.48273	521.15520	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.788	BV	0.0738	178.51828	37.30751	2.6955
2	2.145	VB	0.0832	17.99508	3.32414	0.2717
3	2.673	BB	0.0960	15.68292	2.53997	0.2368
4	3.527	BV	0.1074	5836.37598	857.74463	88.1265
5	4.158	VB	0.1173	44.19037	5.90879	0.6673
6	5.534	BV	0.1425	458.84863	51.20990	6.9284
7	5.977	VB	0.1393	12.27853	1.36005	0.1854
8	7.006	BB	0.2049	23.39492	1.63500	0.3533
9	8.230	BB	0.1566	18.55914	1.76580	0.2802
10	10.436	BB	0.2114	16.87946	1.25140	0.2549
Totals :				6622.72332	964.04719	

Data File C:\Chem32\1\Data\Casey\CJM-VII-416.D
Sample Name: CJM-VII-416

Signal 3: DAD1 D, Sig=230,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.788	BV	0.0742	38.65020	8.02876	0.5564
2	2.146	VB	0.0814	17.53037	3.32858	0.2524
3	2.677	BB	0.0922	14.84146	2.54028	0.2137
4	3.527	BV	0.1074	6444.05908	946.88269	92.7745
5	4.158	VB	0.1213	23.66982	3.02602	0.3408
6	5.534	BV	0.1427	397.55267	44.29490	5.7235
7	5.977	VB	0.1492	9.63199	1.02897	0.1387
Totals :				6945.93560	1009.13020	

Signal 4: DAD1 E, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.527	BB	0.1077	482.48431	70.62589	85.8845
2	5.534	BB	0.1400	79.29857	8.89349	14.1155
Totals :				561.78288	79.51938	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.527	BB	0.1069	822.84998	121.67978	100.0000
Totals :				822.84998	121.67978	

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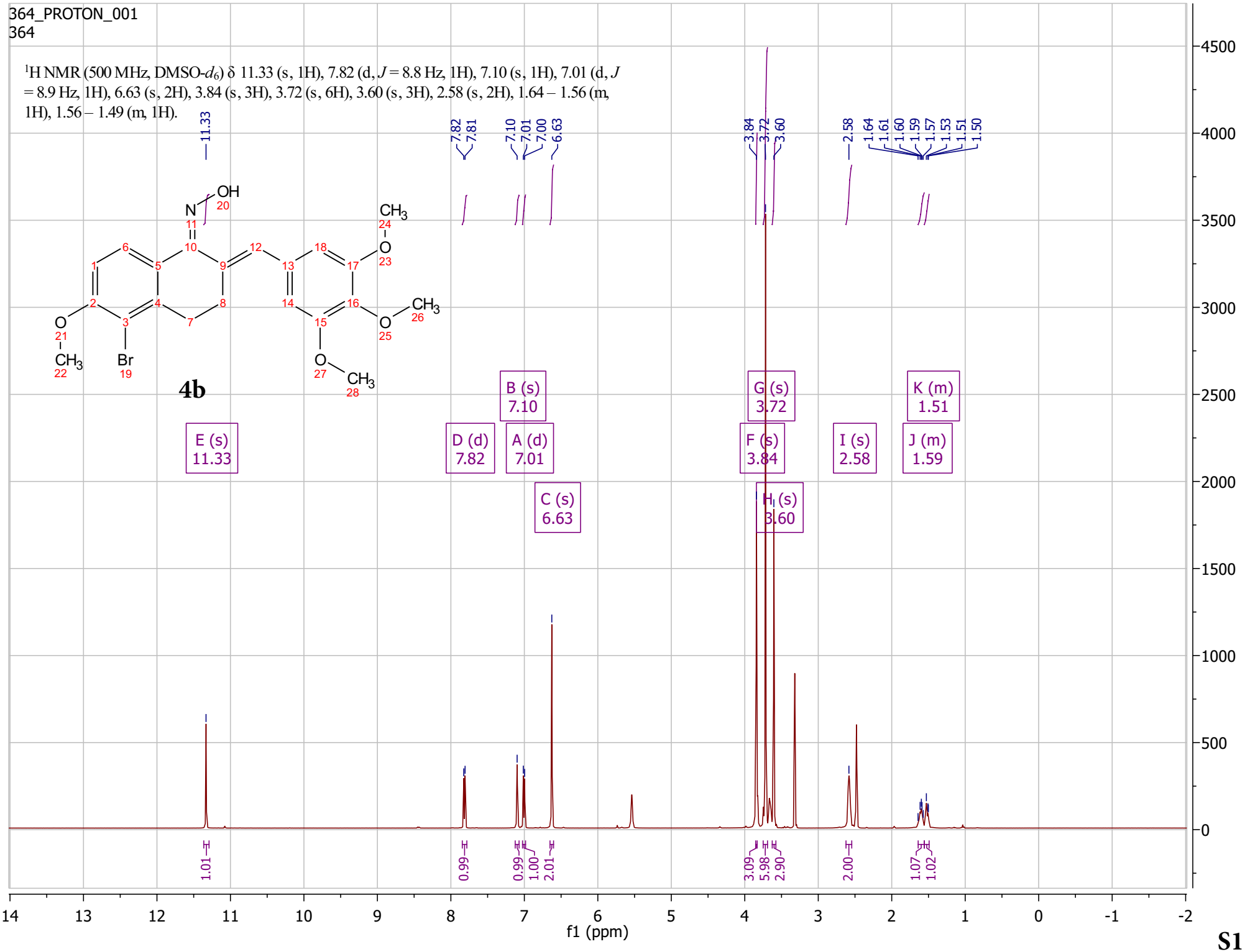
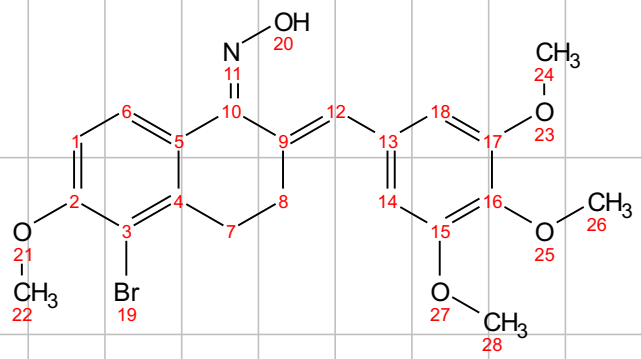
*** End of Report ***


364_PROTON_001
364
^{1}H NMR (500 MHz, DMSO-d_6) δ 11.33 (s, 1H), 7.82 (d, J = 8.8 Hz, 1H), 7.10 (s, 1H), 7.01 (d, J = 8.9 Hz, 1H), 6.63 (s, 2H), 3.84 (s, 3H), 3.72 (s, 6H), 3.60 (s, 3H), 2.58 (s, 2H), 1.64 – 1.56 (m, 1H), 1.56 – 1.49 (m, 1H).
4b
E (s) 11.33
D (d) 7.82
A (d) 7.01
B (s) 7.10
C (s) 6.63
F (s) 3.84
G (s) 3.72
H (s) 3.60
I (s) 2.58
J (m) 1.59
K (m) 1.51
f1 (ppm)

364_CARBON_001
364

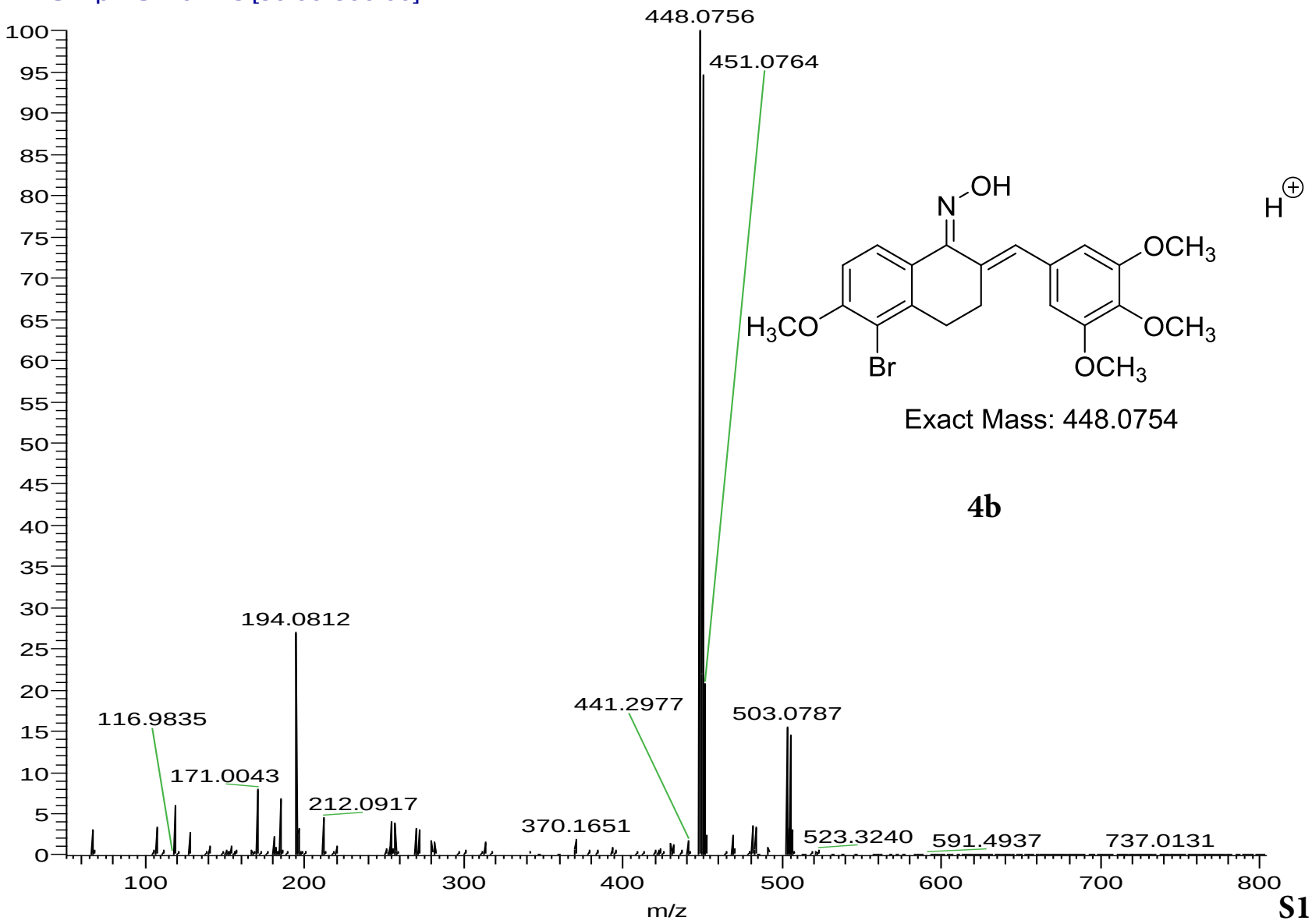
^{13}C NMR (126 MHz, dmso) δ 156.24, 154.21, 152.88, 138.52, 137.85, 136.77, 125.75, 125.34, 113.10, 110.91, 105.56, 66.24, 60.35, 56.77, 56.22, 33.65, 26.48, 22.95.



4b

CJM-VI-364

364_+ESI #2-12 RT: 0.01-0.09 AV: 11 NL: 2.28E7
T: FTMS + p ESI Full ms [50.00-800.00]



Exact Mass: 448.0754

4b

Data File C:\Chem32\1\Data\Casey\CJM-VI-364 Sample Name: CJM-VI-364

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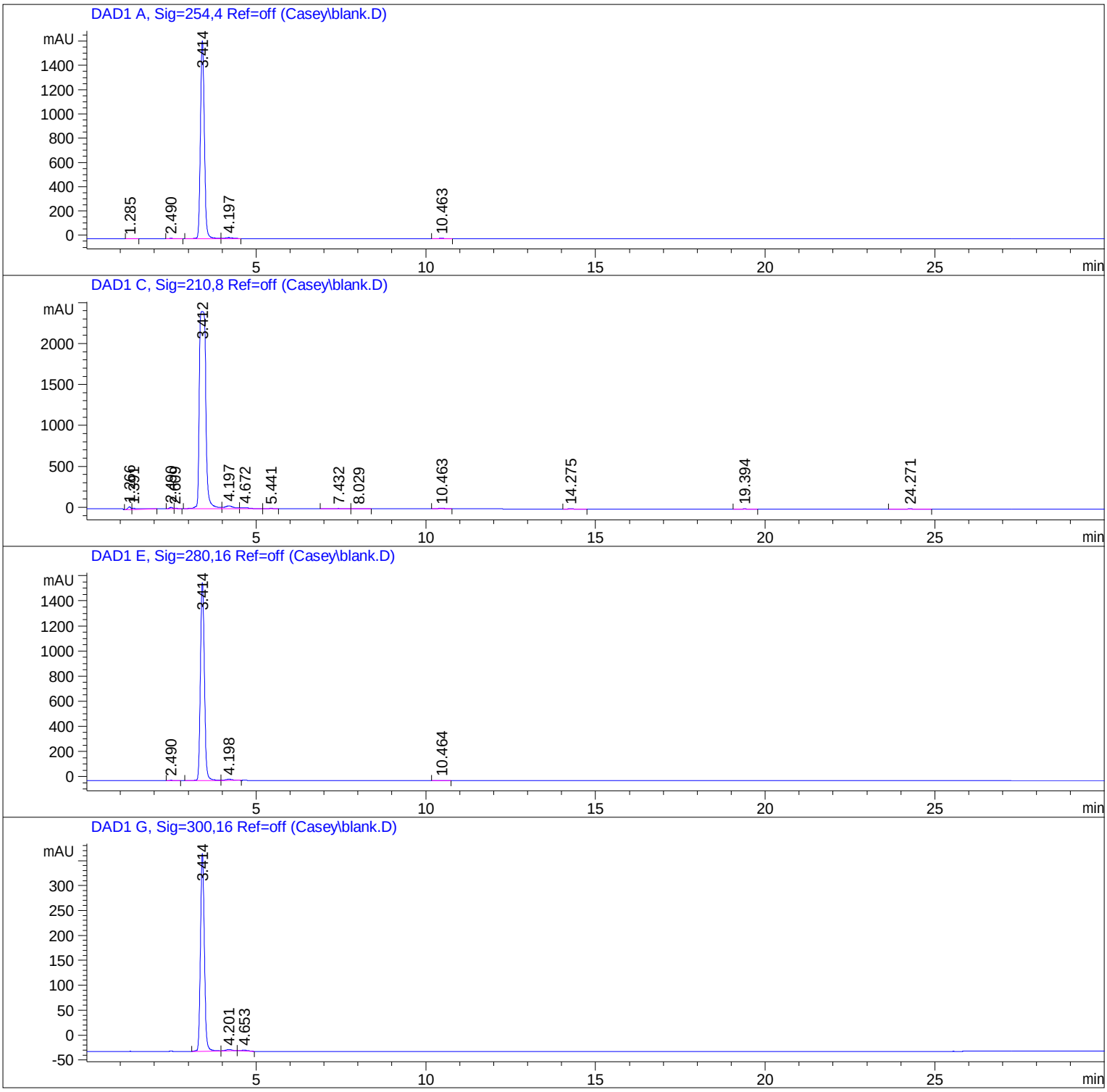
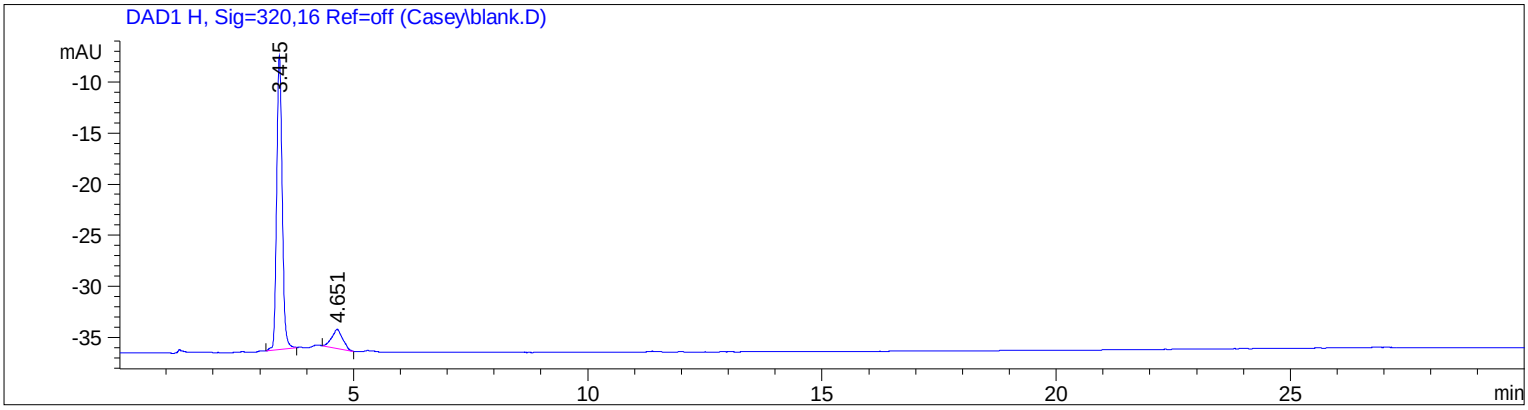
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : 1200 HPLC Location : 1
Injection Date : 7/25/2018 9:45:21 AM
Inj Volume : No inj
Method : C:\CHEM32\1\METHODS\GRAD 2 50-90 ACN.M
Last changed : 4/30/2014 1:53:57 AM by ERICAP
Sample Info : GRAD 2 50-90



Data File C:\Chem32\1\Data\Casey\CJM-VI-364 Sample Name: CJM-VI-364



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Area Percent Report

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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	1.285	BB	0.0947	9.18303	1.32830	0.0700
2	2.490	BB	0.1298	29.18906	3.21759	0.2225
3	3.414	BV	0.1223	1.29265e4	1634.76965	98.5261
4	4.197	VB	0.2184	123.55957	8.36227	0.9418
5	10.463	BB	0.1779	31.44169	2.73882	0.2396
Totals :				1.31199e4	1650.41663	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.266	BV	0.0846	199.93243	35.01984	0.6009
2	1.391	VB	0.2445	256.61960	12.99091	0.7713
3	2.490	BV	0.1019	98.47733	14.74831	0.2960
4	2.609	VB	0.0850	33.38246	5.99014	0.1003
5	3.412	BV	0.2109	3.15734e4	2408.82056	94.8932
6	4.197	VV	0.2615	611.47723	33.42126	1.8378
7	4.672	VB	0.2758	186.63203	9.90611	0.5609
8	5.441	BB	0.1389	27.98796	3.11140	0.0841
9	7.432	BB	0.2296	45.58840	2.77548	0.1370
10	8.029	BB	0.2526	22.81437	1.41107	0.0686
11	10.463	BB	0.1762	121.29078	10.54598	0.3645
12	14.275	BB	0.1944	12.46243	1.00655	0.0375
13	19.394	BB	0.2190	26.94321	1.90506	0.0810
14	24.271	BB	0.2590	55.54523	3.25591	0.1669

Data File C:\Chem32\1\Data\Casey\CJM-VI-364 Sample Name: CJM-VI-364

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
Totals :				3.32726e4	2544.90858	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.490	BB	0.1252	21.82883	2.51534	0.1693
2	3.414	BV	0.1259	1.27163e4	1580.23584	98.6479
3	4.198	VB	0.2206	134.32085	9.07922	1.0420
4	10.464	BB	0.1767	18.13908	1.57162	0.1407
Totals :				1.28906e4	1593.40202	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.414	BB	0.1213	3109.18774	397.33414	98.3588
2	4.201	BB	0.1842	29.10686	2.49292	0.9208
3	4.653	BB	0.2056	22.77155	1.62432	0.7204
Totals :				3161.06615	401.45137	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.415	BB	0.1241	232.37601	28.80913	88.6292
2	4.651	BB	0.2233	29.81300	1.85737	11.3708
Totals :				262.18901	30.66650	

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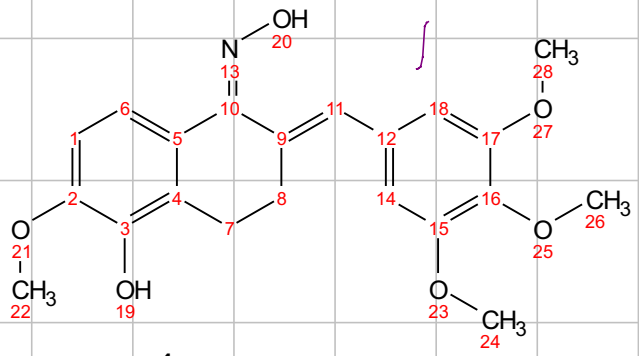
*** End of Report ***

370.10.fid

^{1}H NMR (600 MHz, DMSO-d_6) δ 11.13 (s, 1H), 8.60 (s, 1H), 7.32 (d, J = 8.6 Hz, 1H), 7.08 (s, 1H), 6.87 (d, J = 8.7 Hz, 1H), 6.65 (s, 2H), 3.81 (s, 3H), 3.77 (s, 6H), 3.65 (s, 3H), 2.54 (d, J = 5.2 Hz, 2H), 1.59 – 1.52 (m, 1H), 1.49 – 1.44 (m, 1H).



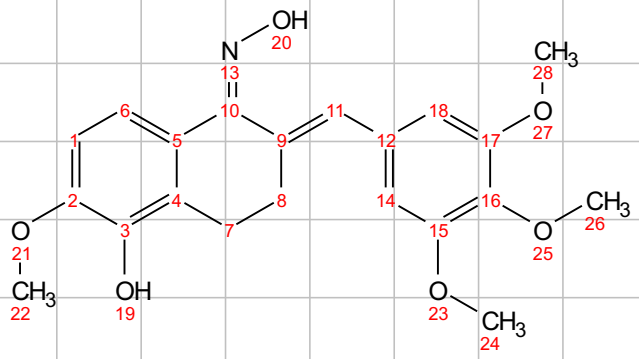
4c

E (s) 11.13
F (s) 8.60
A (d) 7.32
C (d) 6.87
B (s) 7.08
D (s) 6.65
G (s) 3.81
I (s) 3.65
H (s) 3.77
L (d) 2.54
J (m) 1.55
K (m) 1.47

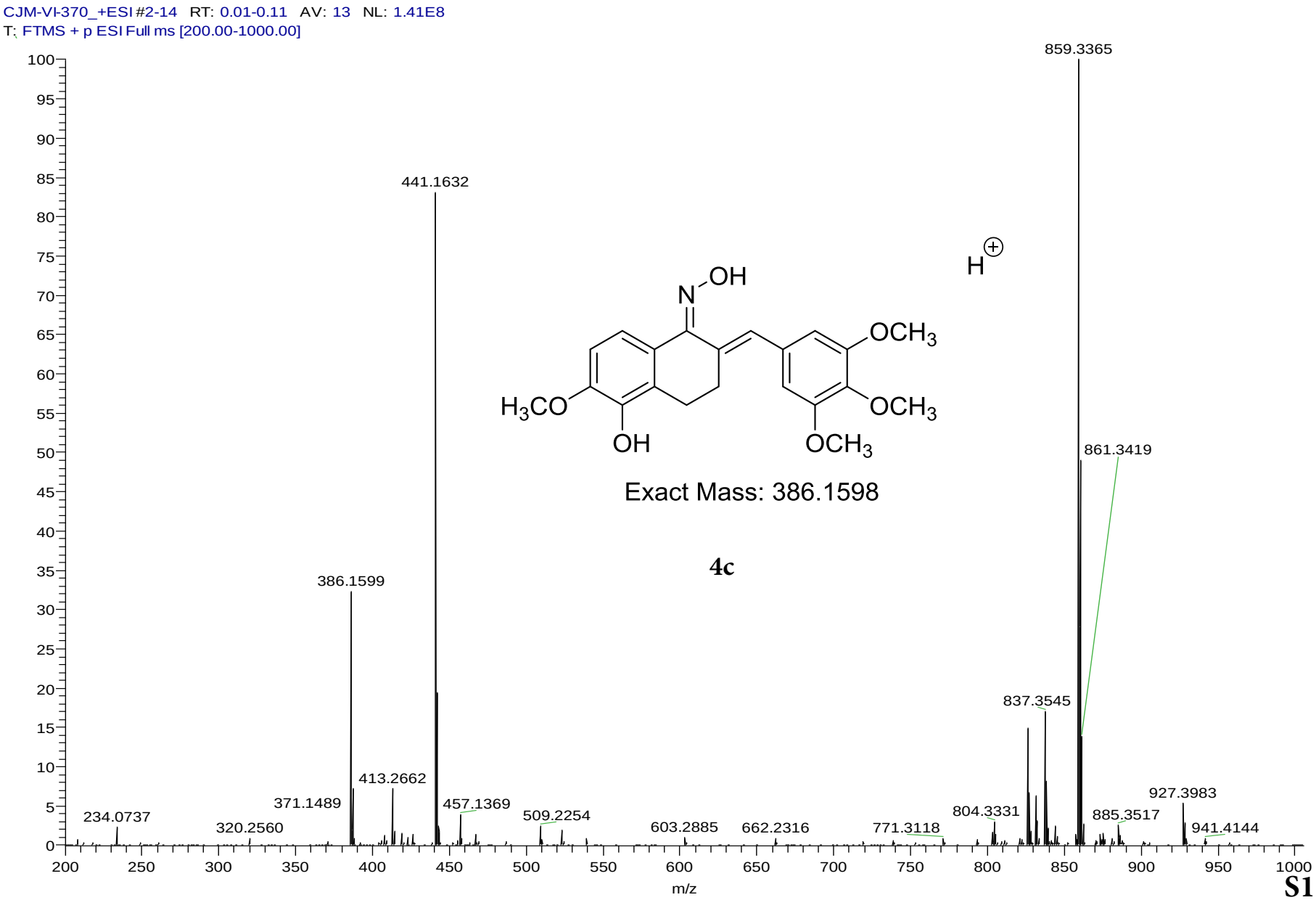
f1 (ppm)

370.11.fid

^{13}C NMR (151 MHz, DMSO) δ 154.98, 152.95, 147.75, 143.27, 138.31, 136.78, 125.60, 124.33, 105.60, 66.58, 60.40, 56.28, 33.86, 23.11, 19.24.



CJM-VI-370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457.1369
804.3331
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234.0737
509.2254
941.4144
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771.3118
m/z
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N
H
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H3CO
OH
OCH3
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Exact Mass: 386.1598
4c

Data File C:\Chem32\1\Data\Casey\370.D
Sample Name: 370

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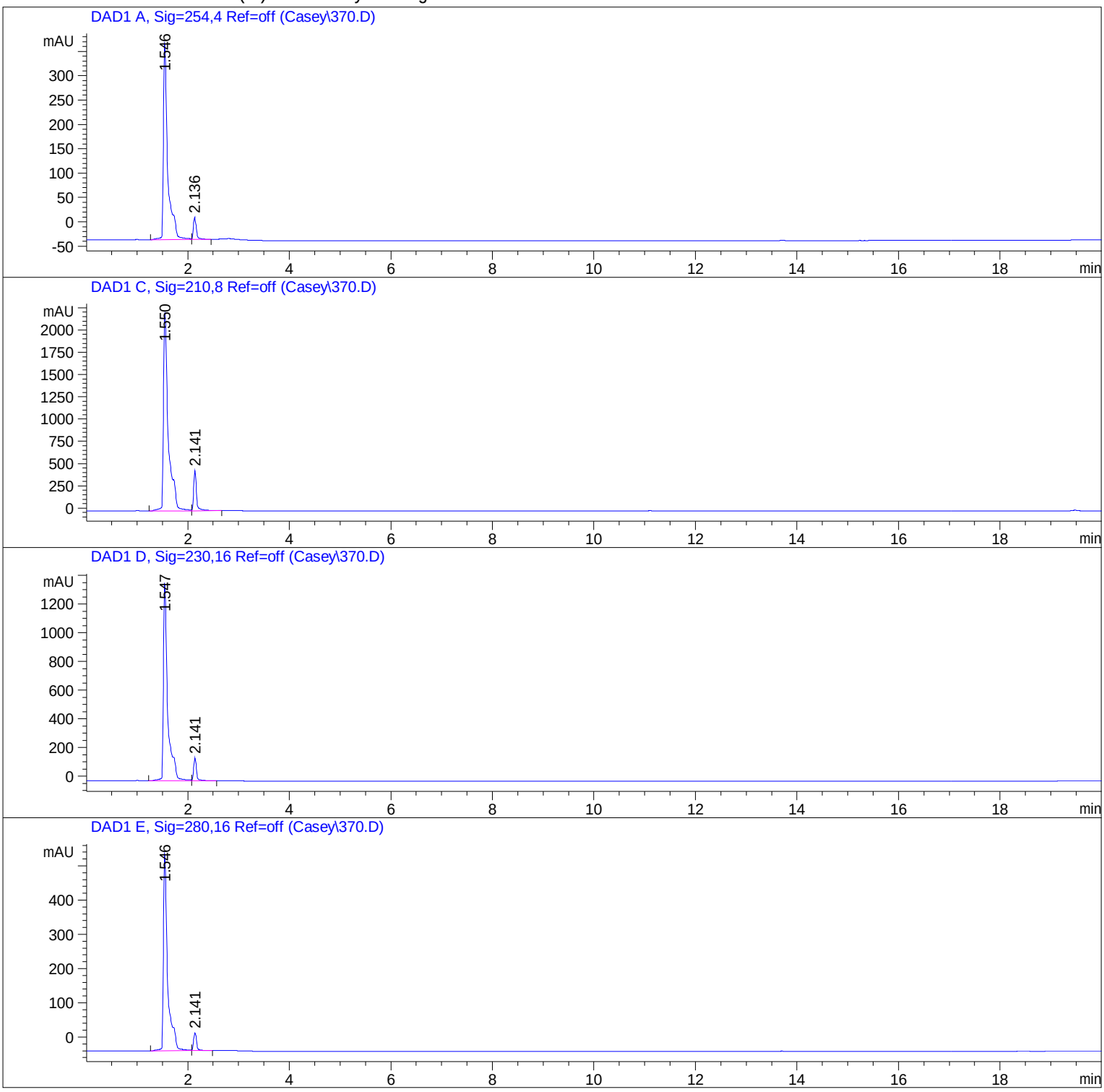
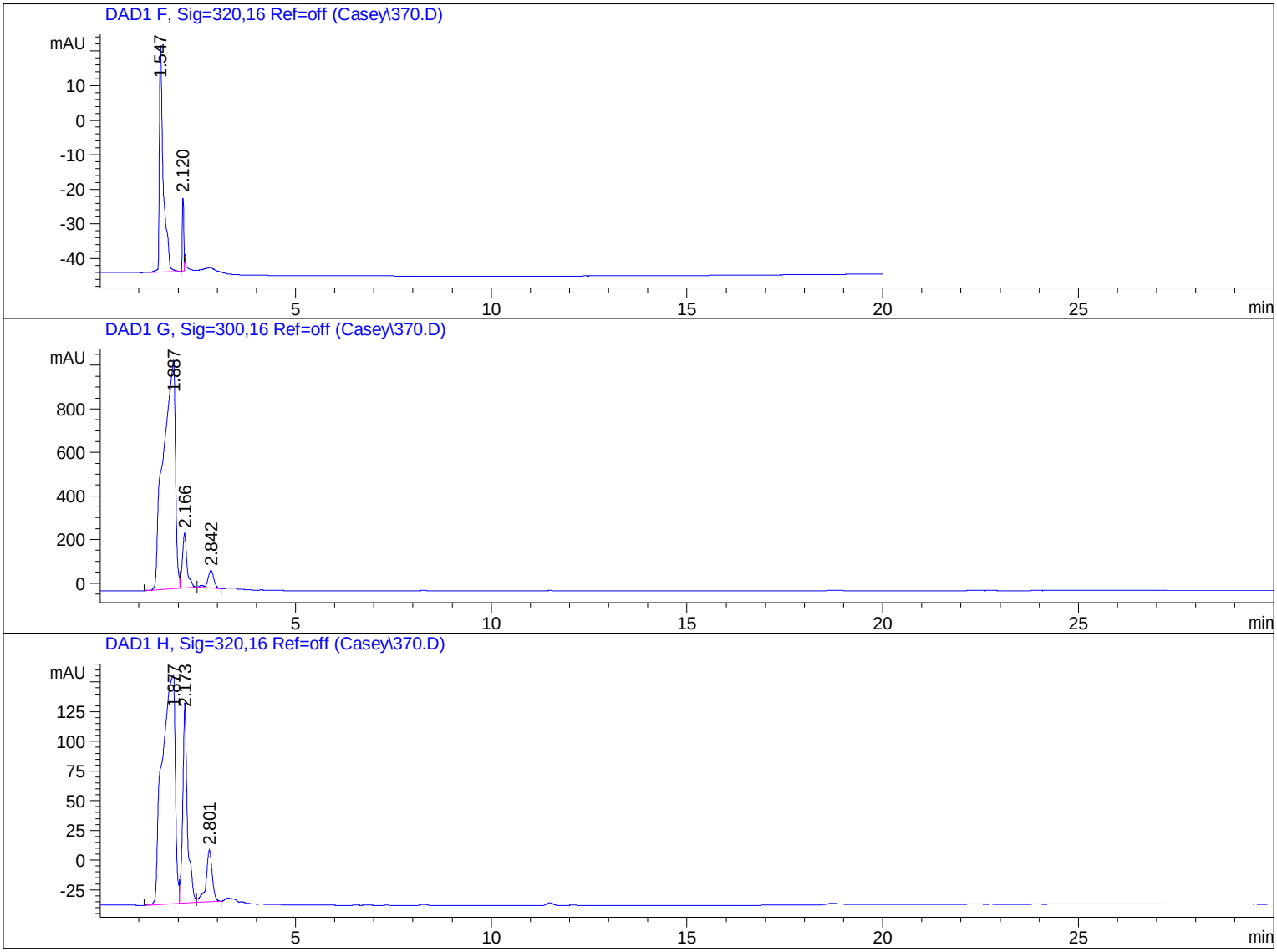
Sample Operator : SYSTEM
Acq. Instrument : 1200 HPLC Location : -
Injection Date : 8/28/2018 11:01:24 AM
Inj Volume : No inj
Acq. Method : C:\CHEM32\1\METHODS\GRAD 2 50-90 ACN.M
Last changed : 8/28/2018 10:14:14 AM by SYSTEM
(modified after loading)
Analysis Method : C:\Chem32\1\Methods\DEF_LC.M
Last changed : 6/22/2014 3:13:01 PM by SYSTEM
Additional Info : Peak(s) manually integrated



Data File C:\Chem32\1\Data\Casey\370.D
Sample Name: 370



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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	1.546	BV	0.0894	2411.40527	405.59781	92.9594
2	2.136	VB	0.0529	182.63727	45.42101	7.0406

Totals : 2594.04254 451.01882

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.550	BV R	0.0967	1.48922e4	2218.21313	89.3982
2	2.141	VB	0.0611	1766.06958	456.44412	10.6018
Totals :				1.66583e4	2674.65726	

Signal 3: DAD1 D, Sig=230,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.547	BV	0.0887	8153.61328	1384.08777	92.9038
2	2.141	VB	0.0598	622.79248	158.70169	7.0962
Totals :				8776.40576	1542.78946	

Signal 4: DAD1 E, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.546	BV	0.0884	3389.73926	577.89960	93.9447
2	2.141	VB	0.0546	218.49078	52.43055	6.0553
Totals :				3608.23004	630.33015	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.547	BB	0.1032	459.75562	66.03182	89.2748
2	2.120	BV	0.0420	55.23376	21.44827	10.7252
Totals :				514.98937	87.48009	

Signal 6: DAD1 G, Sig=300,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.887	BV	0.2678	2.22940e4	1049.82861	88.0038
2	2.166	VB	0.1251	2157.87866	253.97755	8.5181
3	2.842	VB R	0.1572	881.13116	81.25540	3.4782

Data File C:\Chem32\1\Data\Casey\370.D
Sample Name: 370

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
Totals :				2.53330e4	1385.06157	

Signal 7: DAD1 H, Sig=320,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.877	BV	0.2868	4442.33643	192.79108	69.6885
2	2.173	VV	0.1244	1454.13464	168.90517	22.8115
3	2.801	VB	0.1597	478.08810	43.68796	7.4999
Totals :				6374.55917	405.38420	

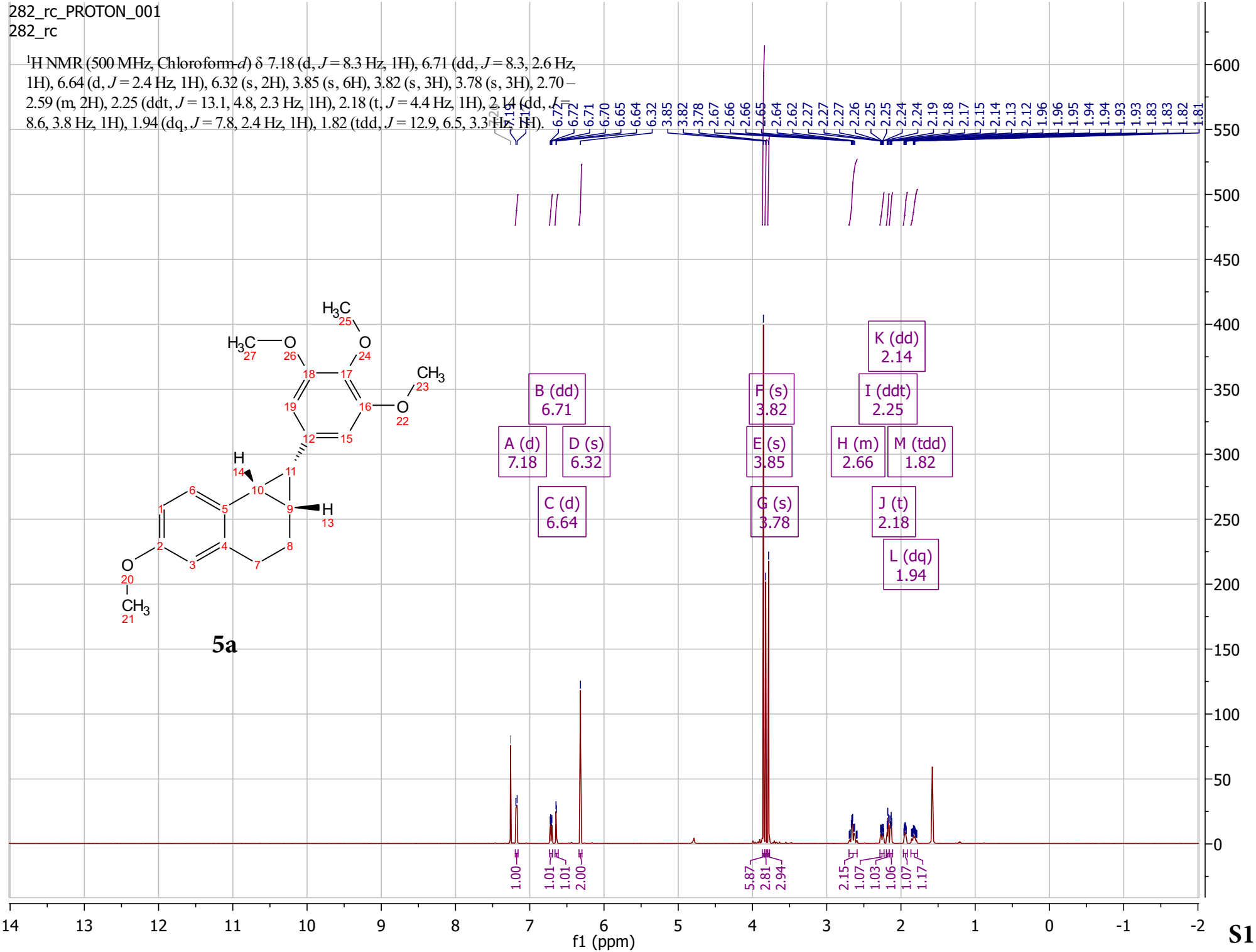
==========

*** End of Report ***

282_rc_PROTON_001
282_rc

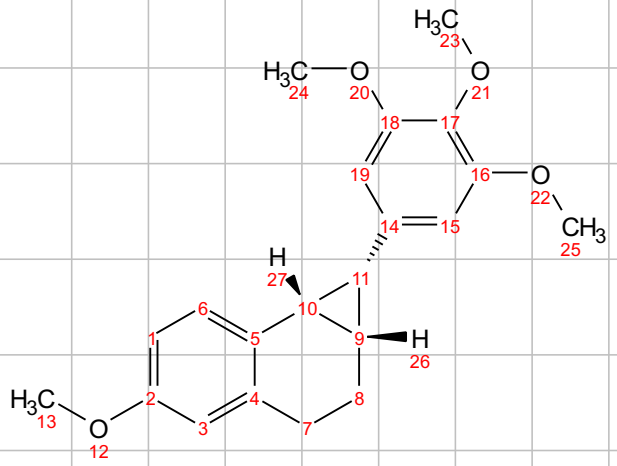
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.18 (d, *J* = 8.3 Hz, 1H), 6.71 (dd, *J* = 8.3, 2.6 Hz, 1H), 6.64 (d, *J* = 2.4 Hz, 1H), 6.32 (s, 2H), 3.85 (s, 6H), 3.82 (s, 3H), 3.78 (s, 3H), 2.70 – 2.59 (m, 2H), 2.25 (ddt, *J* = 13.1, 4.8, 2.3 Hz, 1H), 2.18 (t, *J* = 4.4 Hz, 1H), 2.14 (dd, *J* = 8.6, 3.8 Hz, 1H), 1.94 (dq, *J* = 7.8, 2.4 Hz, 1H), 1.82 (tdd, *J* = 12.9, 6.5, 3.3 Hz, 1H).

5a



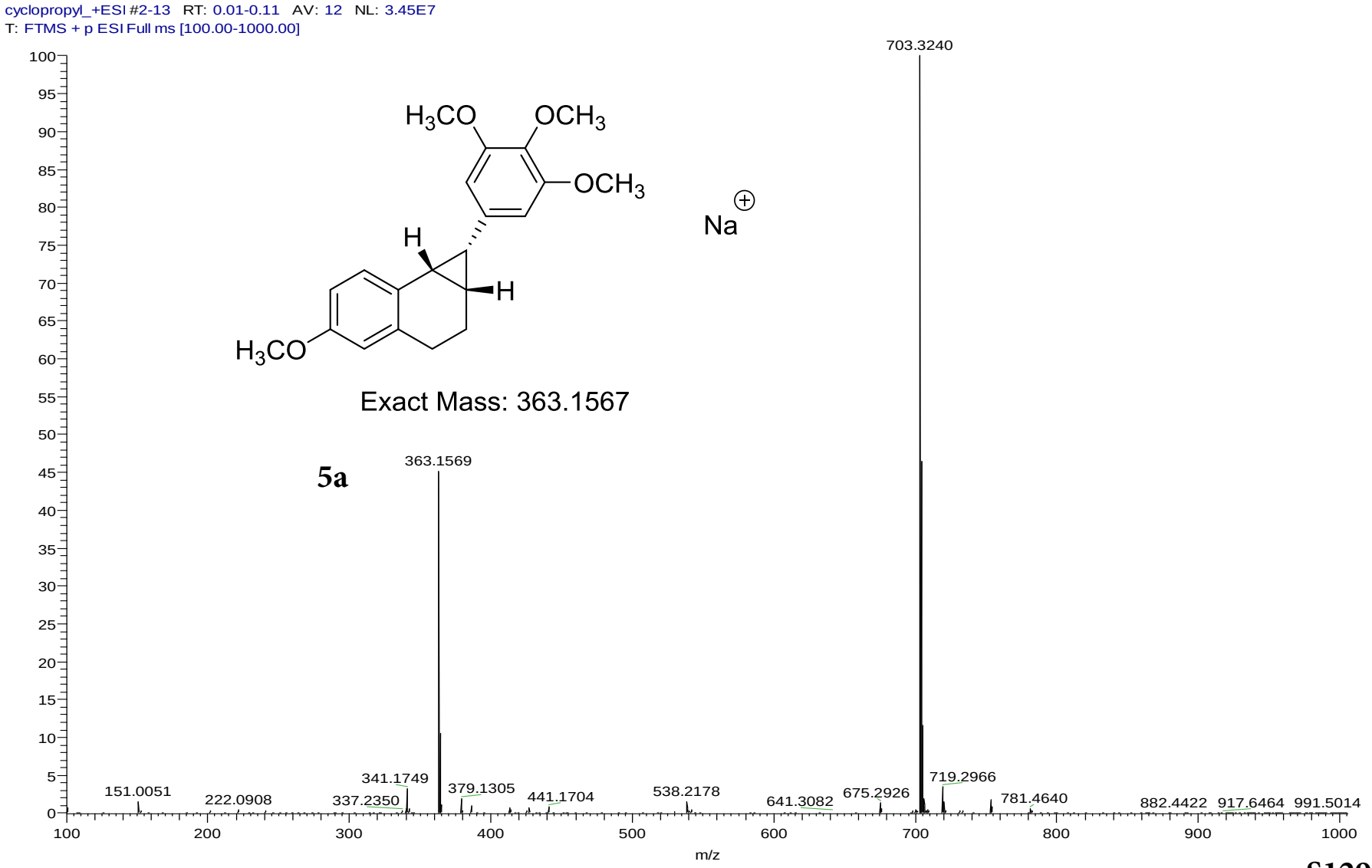
282_rc_CARBON_001
282_rc

^{13}C NMR (126 MHz, cdcl$_3$) δ 157.39, 153.23, 138.42, 134.75, 129.31, 129.19, 129.18, 114.29, 111.58, 109.99, 102.40, 97.28, 77.26, 77.00, 76.75, 60.91, 56.06, 55.31, 27.70, 26.28, 26.00, 24.53, 18.99.



5a

CJM-IV-282


cyclopropyl_+ESI #2-13 RT: 0.01-0.11 AV: 12 NL: 3.45E7
T: FTMS + p ESI Full ms [100.00-1000.00]
703.3240
H3CO
OCH3
OCH3
H
H
H3CO
Na
Exact Mass: 363.1567
5a
363.1569
151.0051
222.0908
337.2350
341.1749
379.1305
441.1704
538.2178
641.3082
675.2926
719.2966
781.4640
882.4422
917.6464
991.5014
m/z

Data File C:\Chem32\1\Data\Casey\CJM-IV-282.D
Sample Name: CJM-IV-282

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    Sample Operator : SYSTEM
    Acq. Instrument : 1200 HPLC                       Location :     -
    Injection Date  : 1/24/2018 1:53:58 PM
                                                   Inj Volume : No inj
    Acq. Method     : C:\CHEM32\1\METHODS\GRAD 2 50-90 ACN.M
    Last changed    : 4/30/2014 1:53:57 AM by ERICAP
    Analysis Method : C:\CHEM32\1\METHODS\RT-ACNWASH 2.M
    Last changed    : 1/24/2018 2:42:18 PM by SYSTEM
                      (modified after loading)
    Method Info     : General Column Wash Method

    ======================================================================
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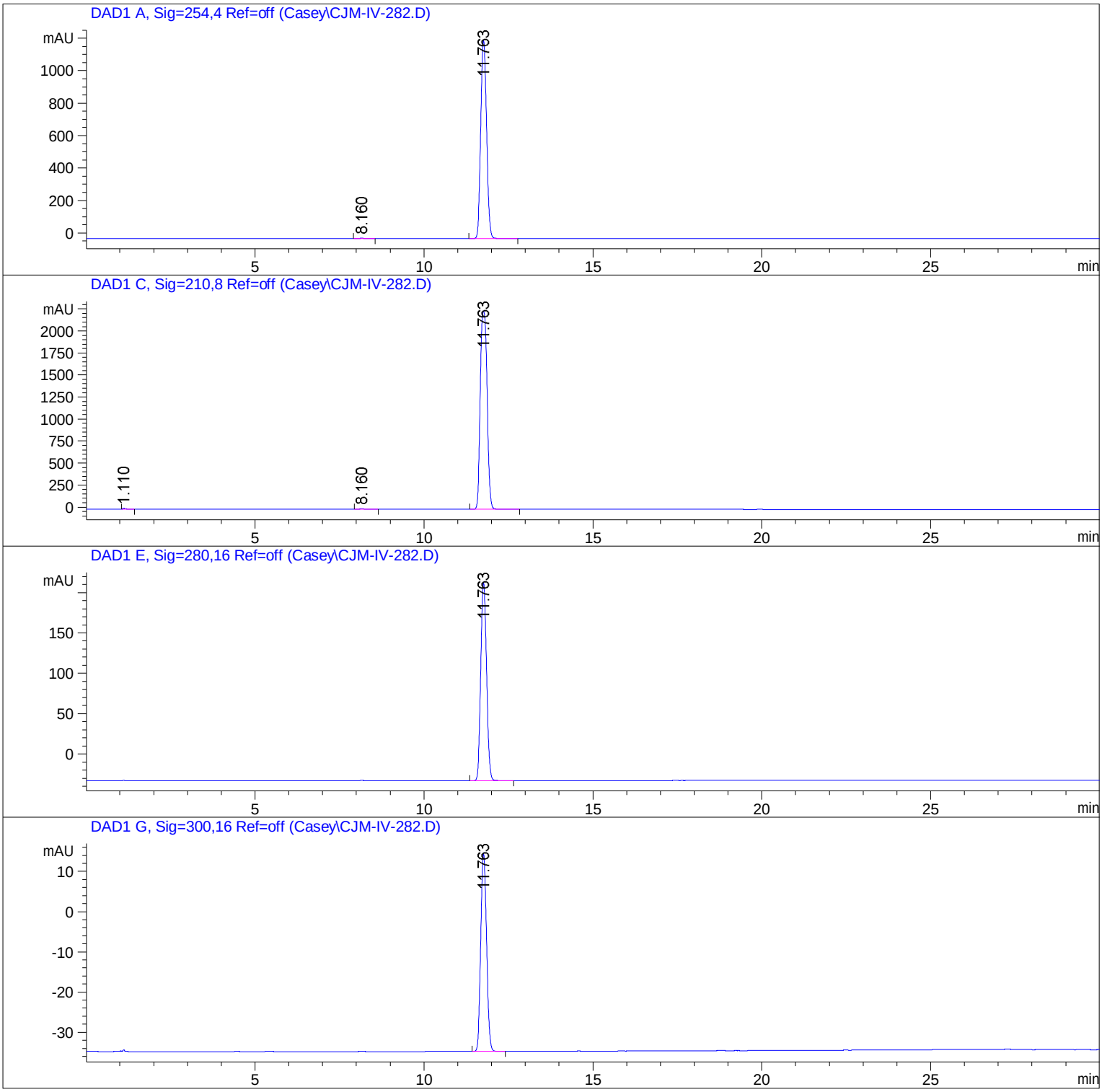

Data File C:\Chem32\1\Data\Casey\CJM-IV-282.D
Sample Name: CJM-IV-282

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Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : 1200 HPLC                    Location :     -
Injection Date  : 1/24/2018 1:53:58 PM
                                             Inj Volume : No inj
Acq. Method     : C:\CHEM32\1\METHODS\GRAD 2 50-90 ACN.M
Last changed    : 4/30/2014 1:53:57 AM by ERICAP
Analysis Method : C:\CHEM32\1\METHODS\RT-ACNWASH 2.M
Last changed    : 1/24/2018 2:42:18 PM by SYSTEM
                  (modified after loading)
Method Info     : General Column Wash Method
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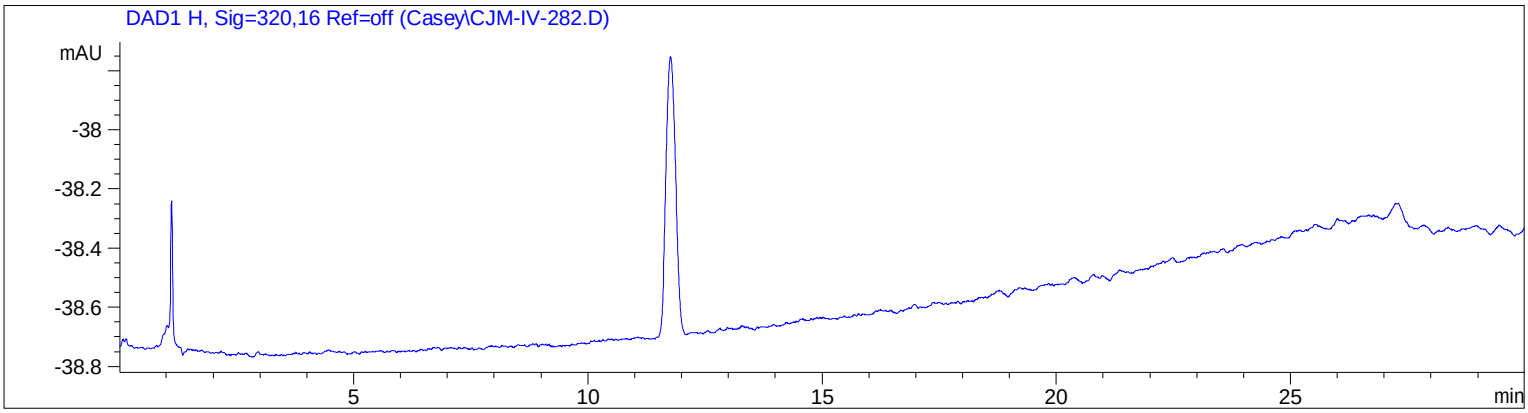

Area Percent Report

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Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not 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	8.160	BB	0.1542	16.60509	1.69643	0.1142
2	11.763	BB	0.1845	1.45259e4	1223.22546	99.8858
Totals :				1.45426e4	1224.92190	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.110	BB	0.0755	77.73724	14.74757	0.2486
2	8.160	BB	0.1544	45.65153	4.65519	0.1460
3	11.763	BB	0.2215	3.11424e4	2249.99146	99.6054

Data File C:\Chem32\1\Data\Casey\CJM-IV-282.D
Sample Name: CJM-IV-282

Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : 1200 HPLC Location : -
Injection Date : 1/24/2018 1:53:58 PM
Inj Volume : No inj
Acq. Method : C:\CHEM32\1\METHODS\GRAD 2 50-90 ACN.M
Last changed : 4/30/2014 1:53:57 AM by ERICAP
Analysis Method : C:\CHEM32\1\METHODS\RT-ACNWASH 2.M
Last changed : 1/24/2018 2:42:18 PM by SYSTEM
(modified after loading)
Method Info : General Column Wash Method

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
Totals :				3.12658e4	2269.39422	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.763	BB	0.1836	2898.56958	245.81544	100.0000
Totals :				2898.56958	245.81544	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.763	BB	0.1842	584.11859	49.29821	100.0000
Totals :				584.11859	49.29821	

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

Summed Peaks Report

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

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

Data File C:\Chem32\1\Data\Casey\CJM-IV-282.D
Sample Name: CJM-IV-282

```
    ======================================================================
    Acq. Operator   : SYSTEM
    Sample Operator : SYSTEM
    Acq. Instrument : 1200 HPLC                       Location :     -
    Injection Date  : 1/24/2018 1:53:58 PM
                                                  Inj Volume : No inj
    Acq. Method     : C:\CHEM32\1\METHODS\GRAD 2 50-90 ACN.M
    Last changed    : 4/30/2014 1:53:57 AM by ERICAP
    Analysis Method : C:\CHEM32\1\METHODS\RT-ACNWASH 2.M
    Last changed    : 1/24/2018 2:42:18 PM by SYSTEM
                      (modified after loading)
    Method Info     : General Column Wash Method

    ======================================================================

      Empty table.

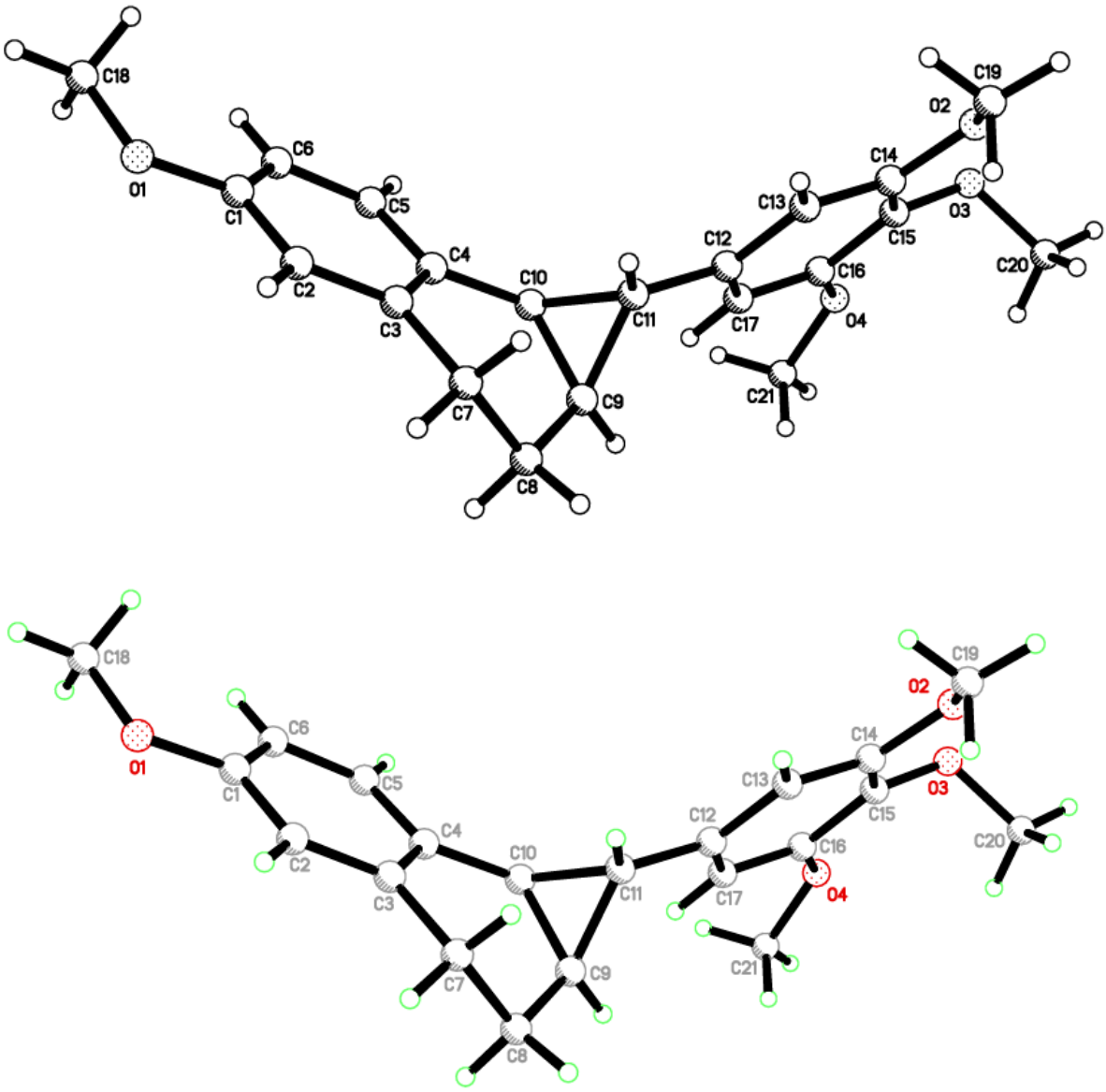
    Signal 3: DAD1 E, Sig=280,16 Ref=off
      Empty table.

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

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

    ======================================================================
                         Final Summed Peaks Report
    ======================================================================

    Signal 1: DAD1 A, Sig=254,4 Ref=off
    Signal 2: DAD1 C, Sig=210,8 Ref=off
    Signal 3: DAD1 E, Sig=280,16 Ref=off
    Signal 4: DAD1 G, Sig=300,16 Ref=off
    Signal 5: DAD1 H, Sig=320,16 Ref=off
                              *** End of Report ***
```



Compound 5a

X-ray crystal structure of **KGP518 (Compound 5a)**

X-ray Crystallographic Analysis:
X-ray crystallographic analysis of compound KGP518. Crystallographic data were collected on a crystal of KGP03 with dimensions 0.343 x 0.322 x 0.147 mm^3. Data were collected at 150(2) K on a Bruker X8 Apex using Mo KR radiation (λ = 0.71073 Å). The structure was solved by direct methods after correction of the data using SADABS. Crystallographic data and definement details for the complex mentioned herein is found in the Supporting

Information (Table S1-S5). All data were processed using the Bruker AXS SHELXTL software, version 6.10.

Table 5. Crystal data and structure refinement for KGP518.

Identification code	KGP518	
Empirical formula	C21 H24 O4	
Formula weight	340.40	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	a = 7.0800(2) Å	α= 90°.
	b = 8.7733(3) Å	β= 90°.
	c = 28.3213(8) Å	γ = 90°.
Volume	1759.18(9) $Å^3$	
Z	4	
Density (calculated)	1.285 Mg/m^3	
Absorption coefficient	0.088 mm^{-1}	
F(000)	728	
Crystal size	0.343 x 0.322 x 0.147 mm^3	
Theta range for data collection	2.731 to 28.290°.	
Index ranges	-9<=h<=9, -9<=k<=11, -37<=l<=37	
Reflections collected	14446	
Independent reflections	4355 [R(int) = 0.0309]	
Completeness to theta = 25.242°	99.8 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.921 and 0.905	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4355 / 0 / 230	
Goodness-of-fit on F^2	1.052	
Final R indices [I>2sigma(I)]	R1 = 0.0388, wR2 = 0.0832	
R indices (all data)	R1 = 0.0462, wR2 = 0.0863	

Absolute structure parameter	-0.2(3)
Extinction coefficient	n/a
Largest diff. peak and hole	0.217 and -0.178 e.Å^{-3}

Table 6. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for KGP518. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
O(4)	13424(2)	9775(1)	8800(1)	30(1)
O(3)	12147(2)	11286(1)	9554(1)	28(1)
O(2)	9310(2)	10195(2)	10070(1)	36(1)
O(1)	1114(2)	3351(2)	7720(1)	35(1)
C(4)	6220(3)	5058(2)	8220(1)	22(1)
C(3)	5368(3)	3766(2)	8424(1)	22(1)
C(2)	3669(3)	3242(2)	8242(1)	25(1)
C(6)	3631(3)	5243(2)	7661(1)	28(1)
C(5)	5348(3)	5752(2)	7837(1)	26(1)
C(1)	2793(3)	3981(2)	7867(1)	26(1)
C(10)	8023(3)	5663(2)	8409(1)	23(1)
C(13)	8727(3)	8130(2)	9508(1)	23(1)
C(17)	10881(2)	7901(2)	8853(1)	22(1)
C(12)	9300(3)	7379(2)	9098(1)	21(1)
C(16)	11845(3)	9186(2)	9011(1)	22(1)
C(15)	11245(3)	9970(2)	9413(1)	22(1)
C(11)	8214(3)	6018(2)	8938(1)	21(1)
C(14)	9712(3)	9410(2)	9667(1)	24(1)
C(7)	6321(3)	2969(2)	8833(1)	25(1)
C(9)	9195(3)	4654(2)	8722(1)	24(1)
C(8)	8478(3)	3052(2)	8804(1)	27(1)
C(21)	14425(3)	8820(2)	8481(1)	33(1)
C(20)	13798(3)	10989(2)	9833(1)	32(1)
C(19)	8015(3)	9524(3)	10393(1)	37(1)
C(18)	80(3)	4156(3)	7368(1)	41(1)

Table 7. Bond lengths [Å] and angles [°] for KGP518.

O(4)-C(16)	1.369(2)
O(4)-C(21)	1.421(2)
O(3)-C(15)	1.379(2)
O(3)-C(20)	1.435(2)
O(2)-C(14)	1.364(2)
O(2)-C(19)	1.423(2)
O(1)-C(1)	1.375(2)
O(1)-C(18)	1.424(3)
C(4)-C(5)	1.388(3)
C(4)-C(3)	1.408(2)
C(4)-C(10)	1.483(3)
C(3)-C(2)	1.387(3)
C(3)-C(7)	1.513(3)
C(2)-C(1)	1.391(3)
C(6)-C(1)	1.385(3)
C(6)-C(5)	1.388(3)
C(10)-C(9)	1.503(3)
C(10)-C(11)	1.538(2)
C(13)-C(14)	1.396(3)
C(13)-C(12)	1.397(2)
C(17)-C(16)	1.391(2)
C(17)-C(12)	1.395(3)
C(12)-C(11)	1.490(2)
C(16)-C(15)	1.398(2)
C(15)-C(14)	1.391(3)
C(11)-C(9)	1.513(2)
C(7)-C(8)	1.531(3)
C(9)-C(8)	1.513(3)

C(16)-O(4)-C(21)	117.50(14)
C(15)-O(3)-C(20)	112.62(14)
C(14)-O(2)-C(19)	117.66(15)
C(1)-O(1)-C(18)	117.16(17)
C(5)-C(4)-C(3)	118.89(17)

C(5)-C(4)-C(10)	120.51(16)
C(3)-C(4)-C(10)	120.60(16)
C(2)-C(3)-C(4)	119.10(17)
C(2)-C(3)-C(7)	121.22(16)
C(4)-C(3)-C(7)	119.69(16)
C(3)-C(2)-C(1)	121.05(17)
C(1)-C(6)-C(5)	118.74(18)
C(4)-C(5)-C(6)	121.96(18)
O(1)-C(1)-C(6)	124.37(18)
O(1)-C(1)-C(2)	115.39(17)
C(6)-C(1)-C(2)	120.22(17)
C(4)-C(10)-C(9)	118.53(15)
C(4)-C(10)-C(11)	120.04(15)
C(9)-C(10)-C(11)	59.67(12)
C(14)-C(13)-C(12)	120.15(17)
C(16)-C(17)-C(12)	119.98(16)
C(17)-C(12)-C(13)	119.51(16)
C(17)-C(12)-C(11)	121.80(16)
C(13)-C(12)-C(11)	118.68(16)
O(4)-C(16)-C(17)	124.47(16)
O(4)-C(16)-C(15)	114.74(15)
C(17)-C(16)-C(15)	120.79(16)
O(3)-C(15)-C(14)	120.49(16)
O(3)-C(15)-C(16)	120.50(16)
C(14)-C(15)-C(16)	119.01(16)
C(12)-C(11)-C(9)	121.28(16)
C(12)-C(11)-C(10)	120.18(15)
C(9)-C(11)-C(10)	59.03(11)
O(2)-C(14)-C(15)	114.67(16)
O(2)-C(14)-C(13)	124.85(17)
C(15)-C(14)-C(13)	120.48(17)
C(3)-C(7)-C(8)	112.44(16)
C(10)-C(9)-C(8)	116.91(16)
C(10)-C(9)-C(11)	61.30(11)
C(8)-C(9)-C(11)	121.25(16)
C(9)-C(8)-C(7)	112.77(15)

Symmetry transformations used to generate equivalent atoms:

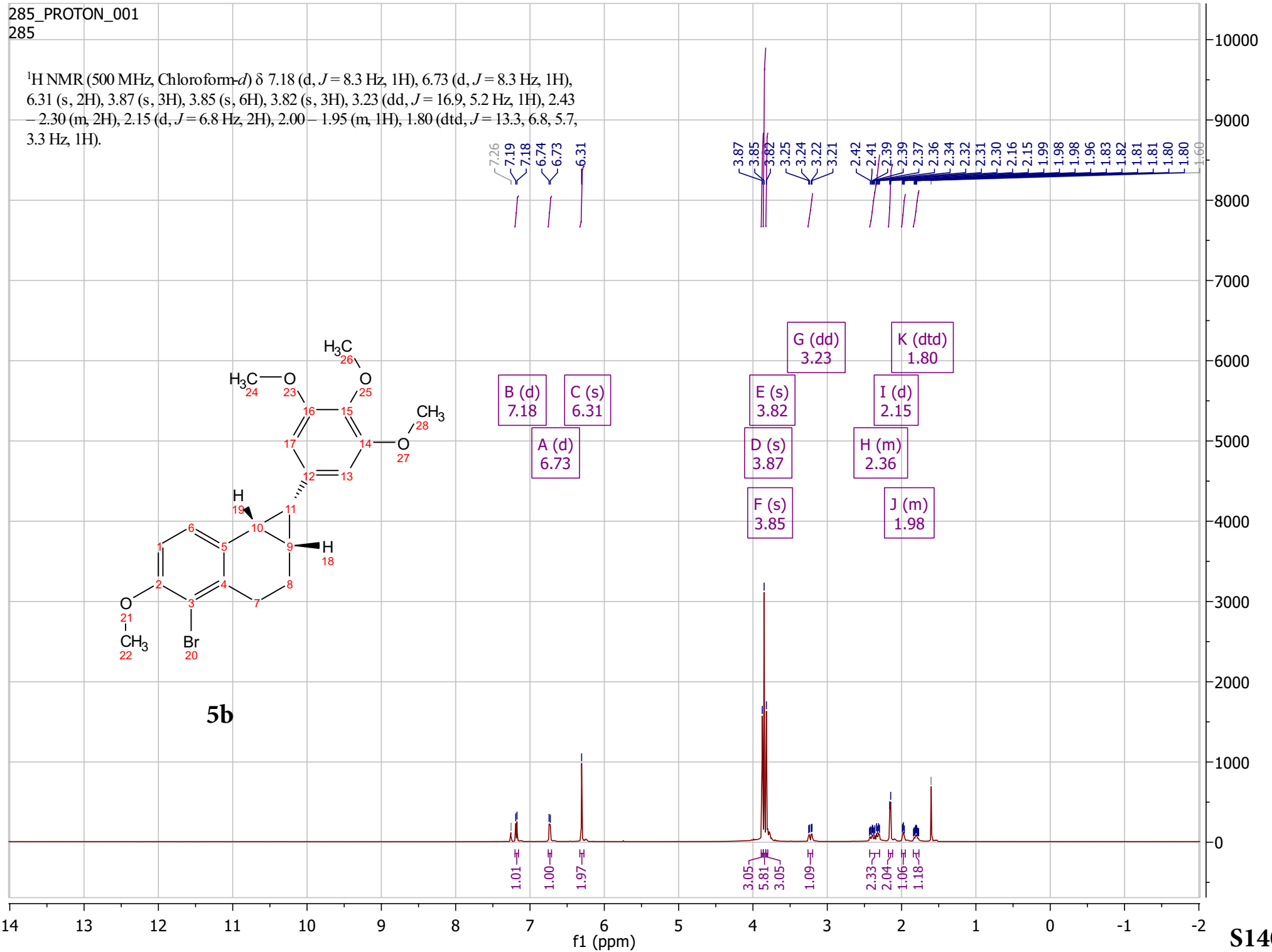
Table 8. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for KGP518. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U_{11} + ... + 2\ h\ k\ a^*\ b^*\ U_{12}\ ]$.

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(4)	33(1)	26(1)	30(1)	-5(1)	13(1)	-9(1)
O(3)	33(1)	17(1)	32(1)	-3(1)	2(1)	-5(1)
O(2)	44(1)	32(1)	34(1)	-15(1)	18(1)	-12(1)
O(1)	28(1)	41(1)	35(1)	-8(1)	-7(1)	-2(1)
C(4)	27(1)	19(1)	19(1)	-4(1)	1(1)	-2(1)
C(3)	26(1)	19(1)	20(1)	-4(1)	2(1)	1(1)
C(2)	28(1)	21(1)	26(1)	-4(1)	3(1)	-2(1)
C(6)	36(1)	28(1)	20(1)	-4(1)	-5(1)	5(1)
C(5)	37(1)	22(1)	20(1)	-1(1)	1(1)	-3(1)
C(1)	25(1)	28(1)	25(1)	-10(1)	-1(1)	0(1)
C(10)	28(1)	22(1)	19(1)	0(1)	2(1)	-6(1)
C(13)	22(1)	22(1)	23(1)	1(1)	3(1)	-1(1)
C(17)	27(1)	21(1)	18(1)	-1(1)	1(1)	1(1)
C(12)	23(1)	19(1)	20(1)	1(1)	-3(1)	0(1)
C(16)	24(1)	20(1)	22(1)	3(1)	2(1)	-1(1)
C(15)	26(1)	16(1)	26(1)	-1(1)	1(1)	-2(1)
C(11)	22(1)	20(1)	20(1)	0(1)	-1(1)	-2(1)
C(14)	28(1)	20(1)	24(1)	-3(1)	4(1)	1(1)
C(7)	31(1)	18(1)	26(1)	1(1)	-1(1)	-3(1)
C(9)	23(1)	23(1)	26(1)	-4(1)	0(1)	-1(1)
C(8)	30(1)	20(1)	32(1)	-2(1)	-3(1)	2(1)
C(21)	29(1)	39(1)	32(1)	-7(1)	8(1)	-4(1)
C(20)	38(1)	30(1)	28(1)	-1(1)	-4(1)	-9(1)
C(19)	32(1)	51(1)	27(1)	-12(1)	10(1)	-13(1)
C(18)	34(1)	55(1)	35(1)	-12(1)	-10(1)	4(1)

285_PROTON_001
285

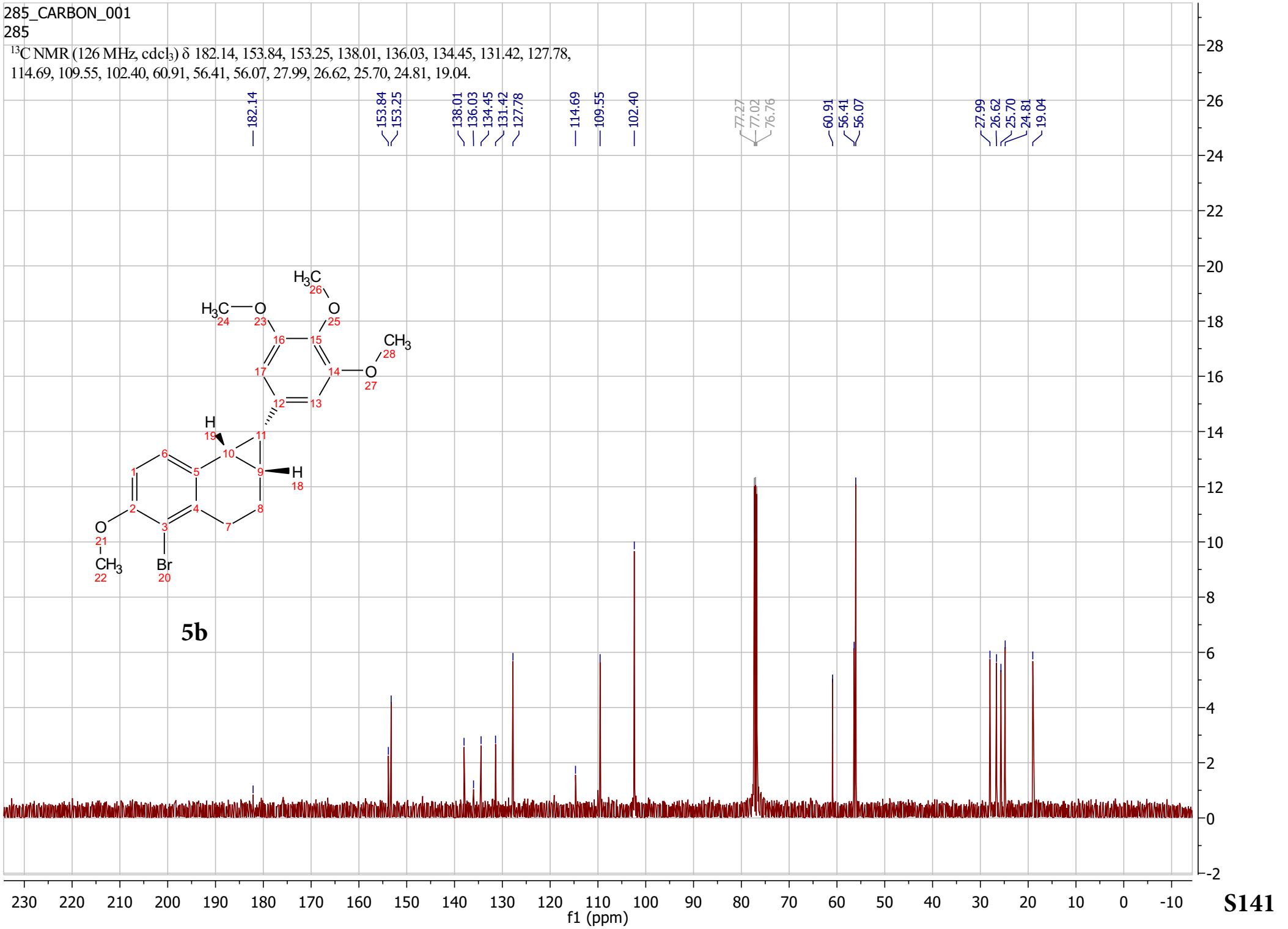
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.18 (d, *J* = 8.3 Hz, 1H), 6.73 (d, *J* = 8.3 Hz, 1H), 6.31 (s, 2H), 3.87 (s, 3H), 3.85 (s, 6H), 3.82 (s, 3H), 3.23 (dd, *J* = 16.9, 5.2 Hz, 1H), 2.43 – 2.30 (m, 2H), 2.15 (d, *J* = 6.8 Hz, 2H), 2.00 – 1.95 (m, 1H), 1.80 (dtd, *J* = 13.3, 6.8, 5.7, 3.3 Hz, 1H).

5b



285_CARBON_001
285

^{13}C NMR (126 MHz, cdcl$_3$) δ 182.14, 153.84, 153.25, 138.01, 136.03, 134.45, 131.42, 127.78, 114.69, 109.55, 102.40, 60.91, 56.41, 56.07, 27.99, 26.62, 25.70, 24.81, 19.04.



CJM-IV-285

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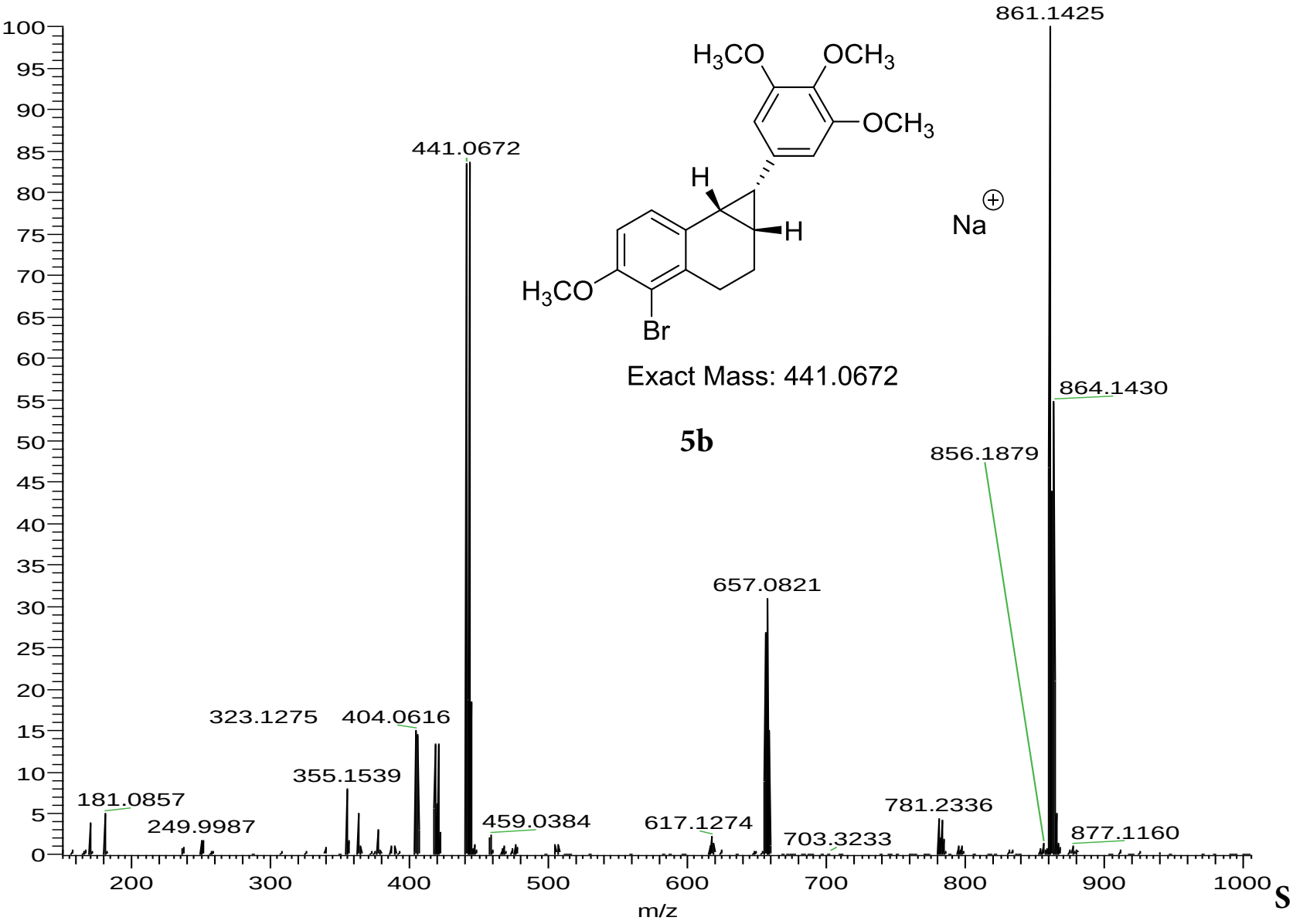
T: FTMS + p ESI Full ms [150.00-1000.00]



Exact Mass: 441.0672

5b

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Sample Name: CJM-IV-285

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Sample Operator : SYSTEM
Acq. Instrument : 1200 HPLC Location : 1
Injection Date : 1/27/2018 4:52:12 PM
Inj Volume : No inj
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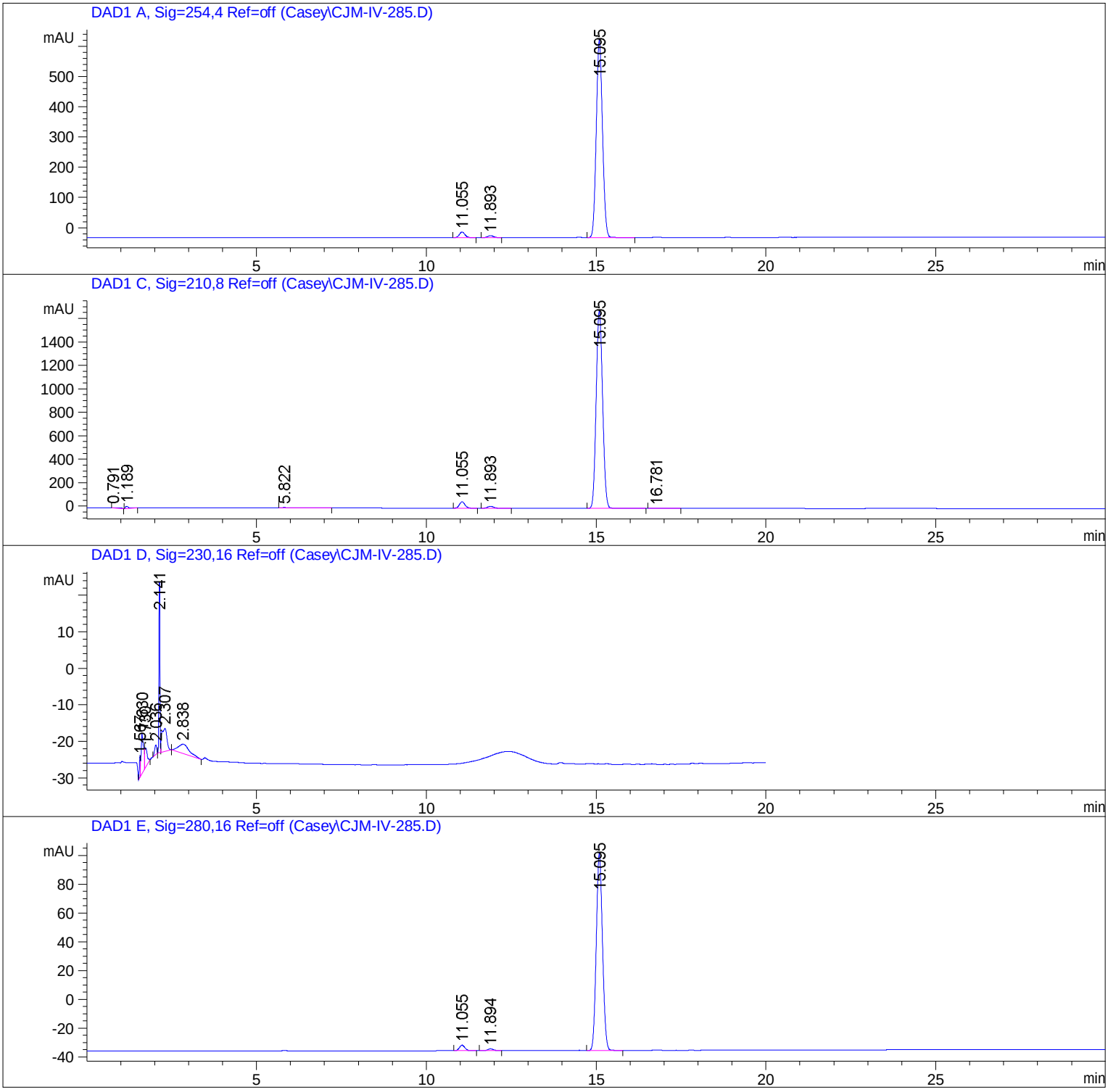
Last changed : 4/30/2014 1:53:57 AM by ERICAP



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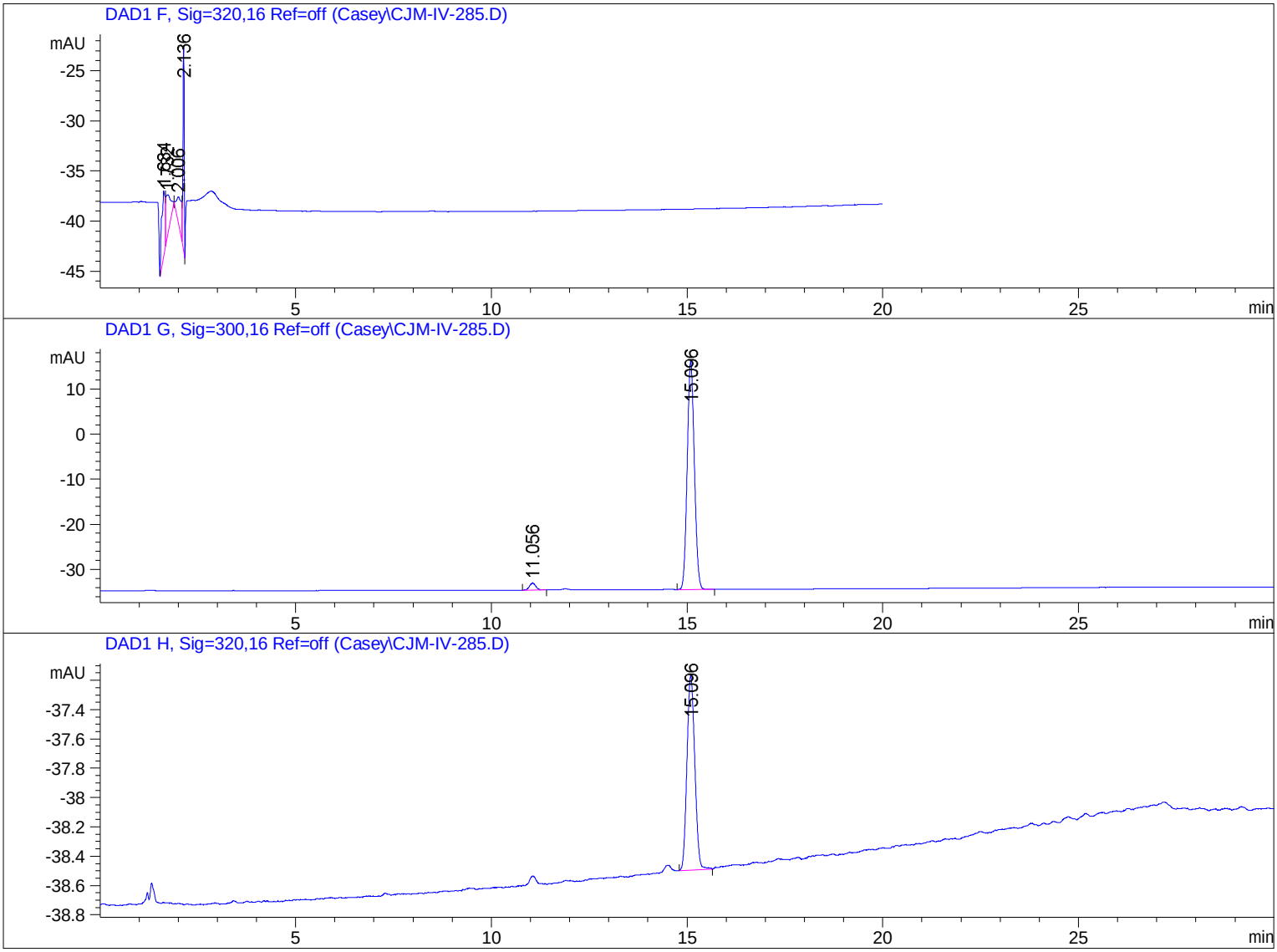

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Area Percent Report

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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	11.055	BB	0.1749	210.08453	18.72971	2.4362
2	11.893	BB	0.1828	74.49093	6.44665	0.8638
3	15.095	BB	0.2003	8339.04785	655.86804	96.7000

Totals : 8623.62331 681.04441

Data File C:\Chem32\1\Data\Casey\CJM-IV-285.D
Sample Name: CJM-IV-285

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	0.791	BB	0.4732	79.67748	2.03319	0.3420
2	1.189	BB	0.1178	151.20578	19.22491	0.6490
3	5.822	BB	0.1303	27.81019	3.30190	0.1194
4	11.055	BB	0.1747	615.49341	54.92379	2.6419
5	11.893	BB	0.1809	190.11765	16.43821	0.8161
6	15.095	BB	0.2077	2.22068e4	1686.06982	95.3195
7	16.781	BB	0.2436	26.13459	1.66034	0.1122
Totals :				2.32972e4	1783.65218	

Signal 3: DAD1 D, Sig=230,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.567	BV	0.0330	11.58737	5.56341	3.4214
2	1.630	VV	0.0714	55.89860	10.97168	16.5054
3	1.730	VB	0.0624	26.19165	5.41365	7.7337
4	2.036	BB	0.0672	10.79056	2.66830	3.1862
5	2.141	BV	0.0400	112.47144	46.91952	33.2099
6	2.307	VB	0.1439	64.20996	6.24658	18.9595
7	2.838	BB	0.3305	57.51886	2.51823	16.9838
Totals :				338.66843	80.30137	

Signal 4: DAD1 E, Sig=280,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.055	BB	0.1749	43.35928	3.86585	2.3994
2	11.894	BB	0.1821	15.00840	1.28665	0.8305
3	15.095	BB	0.2004	1748.72144	137.50285	96.7701
Totals :				1807.08912	142.65535	

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.634	BV	0.0864	42.63451	6.50628	28.0186
2	1.732	VB	0.1001	33.11727	4.10771	21.7641
3	2.006	BV	0.1286	25.21438	2.61142	16.5704
4	2.136	VB	0.0393	51.19877	20.40096	33.6469

Data File C:\Chem32\1\Data\Casey\CJM-IV-285.D
Sample Name: CJM-IV-285

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
Totals :				152.16494	33.62637	

Signal 6: DAD1 G, Sig=300,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.056	BB	0.1748	17.41896	1.55404	2.6238
2	15.096	BB	0.2004	646.47247	50.81461	97.3762
Totals :				663.89143	52.36864	

Signal 7: DAD1 H, Sig=320,16 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.096	BB	0.2186	18.55459	1.33155	100.0000
Totals :				18.55459	1.33155	

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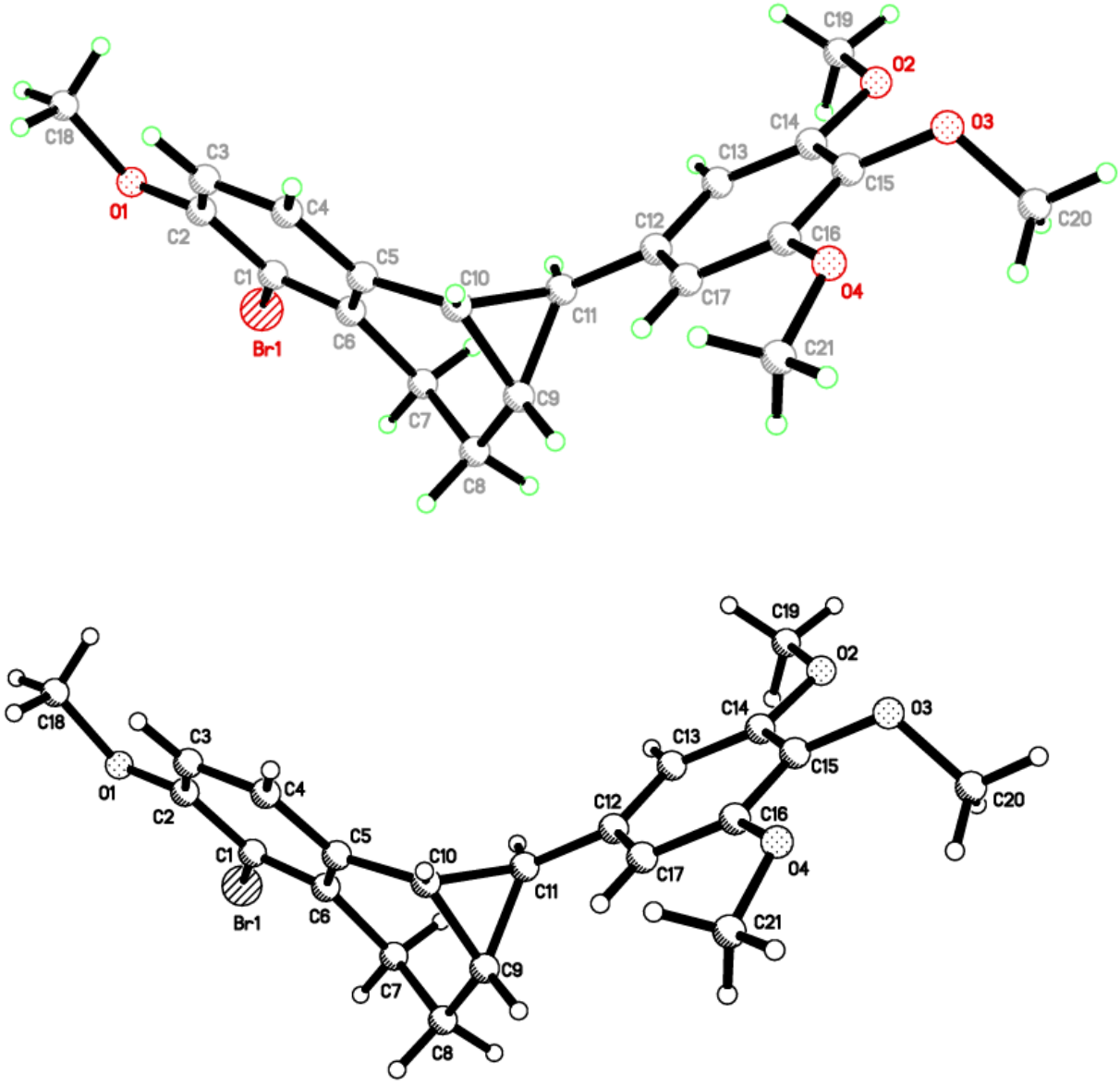
*** End of Report ***



Compound 5b

X-ray crystal structure of **KGP517 (Compound 5b)**

X-ray Crystallographic Analysis:
X-ray crystallographic analysis of compound KGP517. Crystallographic data were collected on a crystal of KGP517 with dimensions 0.213 x 0.164 x 0.067 mm^3. Data were collected at 150(2) K on a Bruker X8 Apex using Mo KR radiation (λ = 0.71073 Å). The structure was solved by direct methods after correction of the data using SADABS. Crystallographic data and definement details for the complex mentioned herein is found in the Supporting

Information (Table S1-S5). All data were processed using the Bruker AXS SHELXTL software, version 6.10.

Table 9. Crystal data and structure refinement for KGP517.

Identification code	KGP517	
Empirical formula	C21 H23 Br O4	
Formula weight	419.30	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	Pbca	
Unit cell dimensions	a = 13.7677(8) Å	α= 90°.
	b = 10.5053(9) Å	β= 90°.
	c = 25.8983(16) Å	γ = 90°.
Volume	3745.8(5) $Å^3$	
Z	8	
Density (calculated)	1.487 Mg/m^3	
Absorption coefficient	2.218 mm^{-1}	
F(000)	1728	
Crystal size	0.213 x 0.164 x 0.067 mm^3	
Theta range for data collection	2.562 to 26.425°.	
Index ranges	-17<=h<=17, -13<=k<=13, -32<=l<=32	
Reflections collected	30172	
Independent reflections	3829 [R(int) = 0.0560]	
Completeness to theta = 25.242°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.702 and 0.526	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3829 / 0 / 239	
Goodness-of-fit on F^2	1.076	
Final R indices [I>2sigma(I)]	R1 = 0.0366, wR2 = 0.0796	
R indices (all data)	R1 = 0.0498, wR2 = 0.0843	

Extinction coefficient	n/a
Largest diff. peak and hole	0.381 and -0.231 e.Å^{-3}

Table 10. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for KGP517. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
Br(1)	5378(1)	322(1)	6364(1)	31(1)
O(3)	-2544(1)	-804(2)	5596(1)	32(1)
O(4)	-2318(1)	1012(2)	6305(1)	32(1)
O(1)	5179(1)	-1023(2)	7342(1)	35(1)
O(2)	-1015(1)	-1917(2)	5143(1)	29(1)
C(1)	4227(2)	181(2)	6768(1)	20(1)
C(12)	249(2)	288(2)	6032(1)	20(1)
C(6)	3367(2)	743(2)	6605(1)	18(1)
C(14)	-814(2)	-1020(2)	5515(1)	22(1)
C(10)	1566(2)	1002(2)	6716(1)	22(1)
C(16)	-1480(2)	507(2)	6109(1)	23(1)
C(15)	-1619(2)	-439(2)	5737(1)	23(1)
C(4)	2593(2)	-113(3)	7366(1)	27(1)
C(8)	2503(2)	2534(2)	6133(1)	29(1)
C(9)	1538(2)	2005(2)	6303(1)	24(1)
C(5)	2522(2)	552(2)	6904(1)	19(1)
C(17)	-551(2)	880(2)	6258(1)	22(1)
C(2)	4294(2)	-513(2)	7225(1)	24(1)
C(3)	3471(2)	-625(2)	7531(1)	29(1)
C(7)	3320(2)	1554(2)	6121(1)	23(1)
C(11)	1258(2)	663(2)	6162(1)	20(1)
C(13)	115(2)	-663(2)	5665(1)	19(1)
C(19)	-212(2)	-2596(3)	4932(1)	29(1)
C(20)	-2977(2)	65(3)	5241(1)	40(1)
C(21)	-2239(2)	2153(3)	6599(1)	39(1)
C(18)	5225(2)	-1872(3)	7776(1)	38(1)

Table 11. Bond lengths [Å] and angles [°] for KGP517.

Br(1)-C(1)	1.903(2)
O(3)-C(15)	1.379(3)
O(3)-C(20)	1.426(3)
O(4)-C(16)	1.368(3)
O(4)-C(21)	1.424(3)
O(1)-C(2)	1.365(3)
O(1)-C(18)	1.437(3)
O(2)-C(14)	1.374(3)
O(2)-C(19)	1.425(3)
C(1)-C(6)	1.389(3)
C(1)-C(2)	1.393(3)
C(12)-C(13)	1.390(3)
C(12)-C(17)	1.394(3)
C(12)-C(11)	1.483(3)
C(6)-C(5)	1.412(3)
C(6)-C(7)	1.518(3)
C(14)-C(13)	1.389(3)
C(14)-C(15)	1.390(3)
C(10)-C(5)	1.481(3)
C(10)-C(9)	1.502(3)
C(10)-C(11)	1.538(3)
C(16)-C(17)	1.392(3)
C(16)-C(15)	1.397(3)
C(4)-C(5)	1.389(3)
C(4)-C(3)	1.390(4)
C(8)-C(9)	1.505(3)
C(8)-C(7)	1.525(3)
C(9)-C(11)	1.506(3)
C(2)-C(3)	1.388(4)

C(15)-O(3)-C(20)	112.2(2)
C(16)-O(4)-C(21)	117.5(2)
C(2)-O(1)-C(18)	117.2(2)
C(14)-O(2)-C(19)	117.14(18)

C(6)-C(1)-C(2)	122.4(2)
C(6)-C(1)-Br(1)	120.72(17)
C(2)-C(1)-Br(1)	116.84(18)
C(13)-C(12)-C(17)	120.1(2)
C(13)-C(12)-C(11)	118.1(2)
C(17)-C(12)-C(11)	121.7(2)
C(1)-C(6)-C(5)	118.5(2)
C(1)-C(6)-C(7)	121.6(2)
C(5)-C(6)-C(7)	119.9(2)
O(2)-C(14)-C(13)	124.5(2)
O(2)-C(14)-C(15)	115.5(2)
C(13)-C(14)-C(15)	119.9(2)
C(5)-C(10)-C(9)	118.7(2)
C(5)-C(10)-C(11)	118.53(19)
C(9)-C(10)-C(11)	59.39(15)
O(4)-C(16)-C(17)	124.2(2)
O(4)-C(16)-C(15)	114.6(2)
C(17)-C(16)-C(15)	121.2(2)
O(3)-C(15)-C(14)	120.3(2)
O(3)-C(15)-C(16)	120.5(2)
C(14)-C(15)-C(16)	119.2(2)
C(5)-C(4)-C(3)	121.3(2)
C(9)-C(8)-C(7)	114.0(2)
C(10)-C(9)-C(8)	116.4(2)
C(10)-C(9)-C(11)	61.49(15)
C(8)-C(9)-C(11)	120.0(2)
C(4)-C(5)-C(6)	119.0(2)
C(4)-C(5)-C(10)	120.5(2)
C(6)-C(5)-C(10)	120.4(2)
C(16)-C(17)-C(12)	119.0(2)
O(1)-C(2)-C(3)	124.6(2)
O(1)-C(2)-C(1)	117.0(2)
C(3)-C(2)-C(1)	118.4(2)
C(2)-C(3)-C(4)	120.2(2)
C(6)-C(7)-C(8)	113.1(2)
C(12)-C(11)-C(9)	122.9(2)

C(12)-C(11)-C(10)	122.1(2)
C(9)-C(11)-C(10)	59.12(15)
C(14)-C(13)-C(12)	120.6(2)

Symmetry transformations used to generate equivalent atoms:

Table 12. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for KGP517. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$.

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br(1)	21(1)	38(1)	33(1)	3(1)	7(1)	0(1)
O(3)	20(1)	36(1)	41(1)	-3(1)	-1(1)	-6(1)
O(4)	20(1)	40(1)	34(1)	-12(1)	4(1)	4(1)
O(1)	26(1)	38(1)	40(1)	16(1)	-8(1)	2(1)
O(2)	27(1)	25(1)	34(1)	-11(1)	-3(1)	-1(1)
C(1)	18(1)	20(1)	22(1)	-2(1)	4(1)	-5(1)
C(12)	22(1)	18(1)	20(1)	5(1)	-1(1)	3(1)
C(6)	22(1)	14(1)	18(1)	-2(1)	-2(1)	-7(1)
C(14)	27(1)	18(1)	20(1)	2(1)	0(1)	-2(1)
C(10)	21(1)	24(1)	21(1)	-5(1)	1(1)	0(1)
C(16)	21(1)	25(1)	22(1)	2(1)	4(1)	2(1)
C(15)	19(1)	25(1)	25(1)	3(1)	-1(1)	-5(1)
C(4)	24(1)	37(2)	20(1)	2(1)	2(1)	-9(1)
C(8)	33(1)	17(1)	38(1)	6(1)	-9(1)	-4(1)
C(9)	24(1)	16(1)	31(1)	-2(1)	-4(1)	3(1)
C(5)	19(1)	20(1)	18(1)	-3(1)	-1(1)	-3(1)
C(17)	24(1)	23(1)	20(1)	-2(1)	-1(1)	2(1)
C(2)	23(1)	22(1)	26(1)	4(1)	-7(1)	-5(1)
C(3)	28(1)	35(2)	23(1)	12(1)	-4(1)	-8(1)
C(7)	22(1)	24(1)	22(1)	6(1)	-1(1)	-6(1)
C(11)	20(1)	18(1)	23(1)	-2(1)	0(1)	1(1)
C(13)	19(1)	17(1)	21(1)	3(1)	-1(1)	2(1)
C(19)	37(1)	24(1)	27(1)	-5(1)	-1(1)	4(1)
C(20)	28(1)	62(2)	32(1)	-3(1)	-7(1)	-3(1)

C(21)	28(1)	41(2)	48(2)	-14(1)	6(1)	10(1)
C(18)	42(2)	33(2)	38(2)	12(1)	-13(1)	2(1)

Table 13. Inhibition of tubulin polymerization, percent inhibition of colchicine binding, and cytotoxicity of the target chalcone, naphthol, oxime, and cyclopropane analogues.

Compound	Inhibition of tubulin polymerization IC_{50} (μM) ± SD	% inhibition of colchicine binding ± SD	GI_{50} (μM) SRB assay			
			NCI-H460	DU-145	SK-OV-3	HUVEC
2a	> 20		22.4 ± 1.14	22.6 ± 1.31	40.3 ± 12	
2b	> 20		22.6 ± 2.04	27.6 ± 3.59	29.6 ± 1.99	
2c	> 20		6.44 ± 0.651	5.49 ± 1.23	6.29 ± 1.01	2.13003 ± 0.166543
2e	> 20		36.3 ± 1.52	37.6 ± 1.96	23.5 ± 1.25	
2f	> 20		46.4 ± 2.79	32.4 ± 7.64	> 135	> 135
2g	> 20		44.4 ± 9.68	57.2 ± 8.00	33.3 ± 5.37	
2h	> 20		6.57 ± 1.88	9.71 ± 0.697	12.0 ± 3.49	
2i	> 20		4.44 ± 0.562	9.91 ± 1.40	8.50 ± 0.911	2.29116 ± 0.147537
2j	> 20	14% ± 2 (5 μM)	0.635 ± 0.0685	0.456 ± 0.0207	0.941 ± 0.335	
2k	> 20		5.96 ± 0.465	5.94 ± 0.372	16.1 ± 2.69	
2l	> 20	0.34% ± 7 (5 μM)	0.733 ± 0.320	0.237 ± 0.0864	0.936 ± 0.636	24.76329 ± 2.484048
3a	17 ± 0.4		13.4 ± 0.812	8.84 ± 3.93	7.50 ± 0.955	2.87955 ± 0.067026
3b	> 20		5.92 ± 0.142	6.17 ± 0.0882	18.3 ± 1.08	
3c	> 20		24.4 ± 2.83	23.4 ± 1.22	21.9 ± 1.69	

3e	> 20		22.9 ± 1.89	18.6 ± 1.34	21.0 ± 0.22	
3f	> 20		65.2 ± 11.5	49.7 ± 6.14	16.4 ± 1.32	
4b	> 20		47.0 ± 4.19	50.1 ± 6.32	80.1 ± 2.68	
4c	> 20		22.8 ± 0.594	23.9 ± 1.97	38.2 ± 5.95	
5a	> 20	0% (5 μM)	38.8 ± 2.47	41.1 ± 3.04	54.5 ± 13.2	
5b	> 20	0% (5 μM)	59.2 ± 8.68	61.5 ± 7.15	108 ± 26.2	
CA4	1.2 ± 0.08	99% ± 1 (5 μM)	0.00500 ± 0.000359	0.00602 ± 0.000661	0.00506 ± 0.00145	
Doxorubicin			0.0547 ± 0.0190	0.1081 ± 0.0942	0.0875 ± 0.1145	2.02847 ± 0.150179
Paclitaxel			0.00165 ± 0.000326	0.00152 ± 0.000288	0.00136 ± 0.000359	0.00913 ± 0.000664