Supporting Information

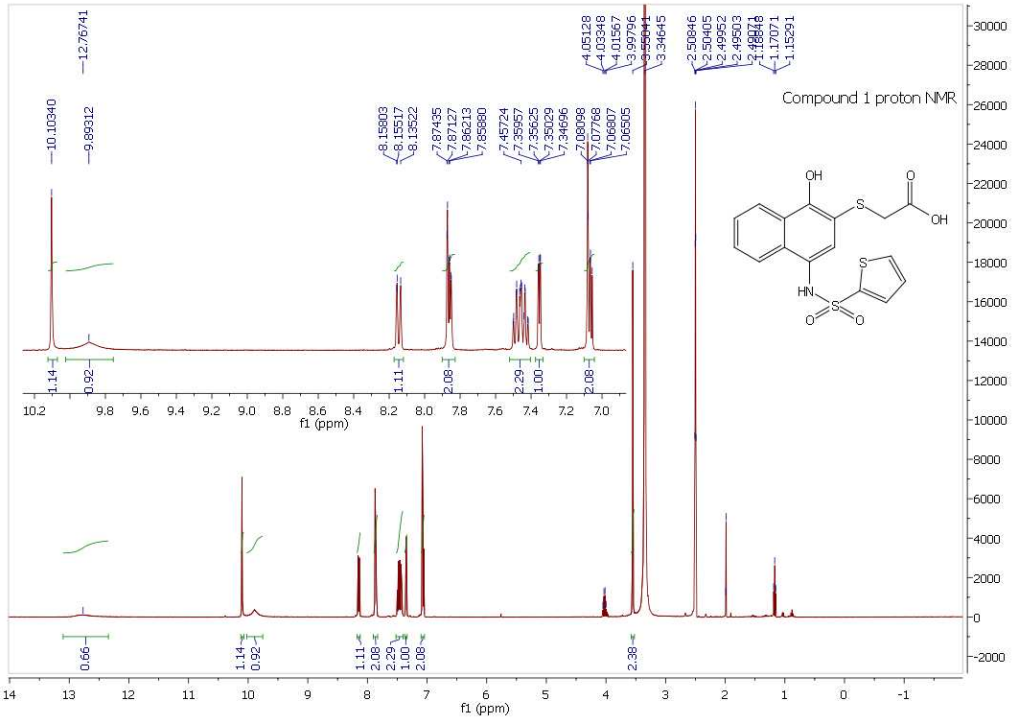
Discovery and Synthesis of Hydronaphthoquinones as Novel Proteasome Inhibitors

Yiyu Ge,[†, ‡, ⊺] Aslamuzzaman Kazi,[†, ⊺] Frank Marsilio,[‡] Yunting Luo,[‡] Sanjula Jain[†], Wesley Brooks,[‡] Kenyon G. Daniel,[‡] Wayne C. Guida,[†, ‡, ∥, §] Saïd M. Sebti,[†, §] and Harshani R. Lawrence*[, †, ‡]

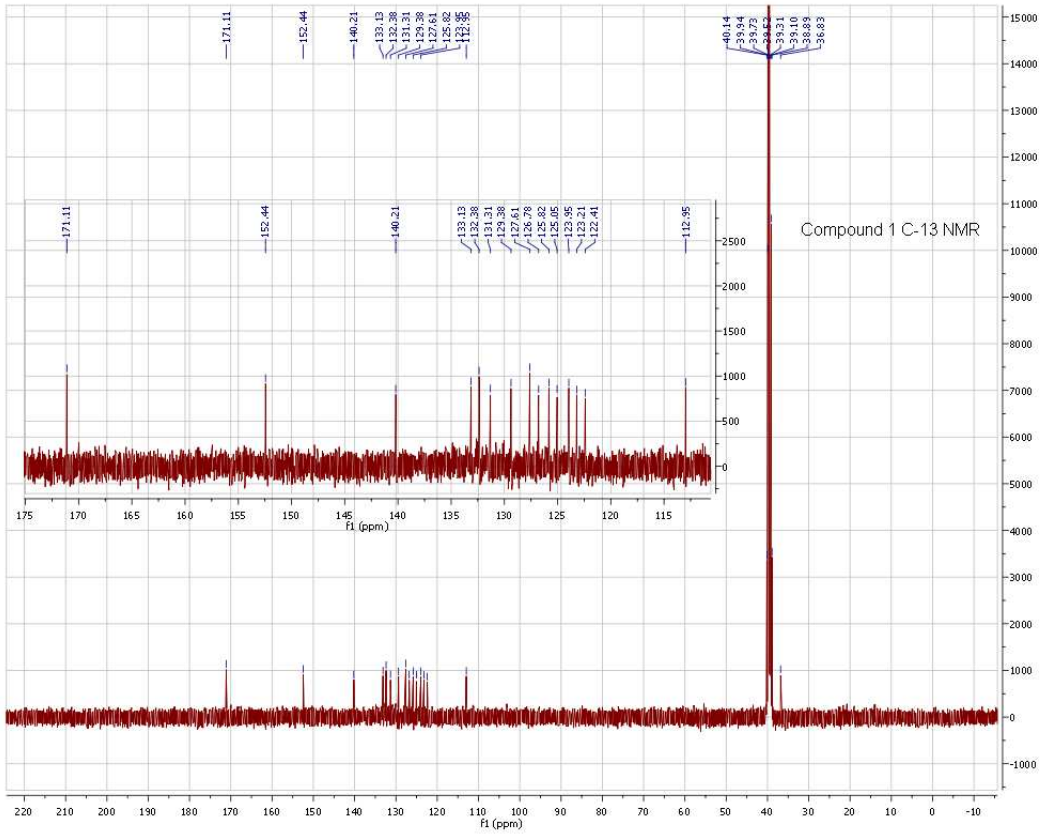
Contents

1. Compound 1:

^{1}H NMR



^{13}C NMR



LC-MS

Sample#: YG1-066 Sample Location: P1-B-02 Sample Id: YG1-066 Operator: EasyAccess
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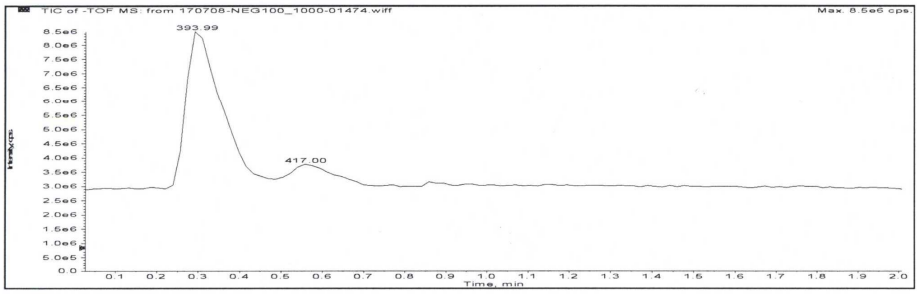

Period# : Average of all periods **Experiment# : Average of all experiments**

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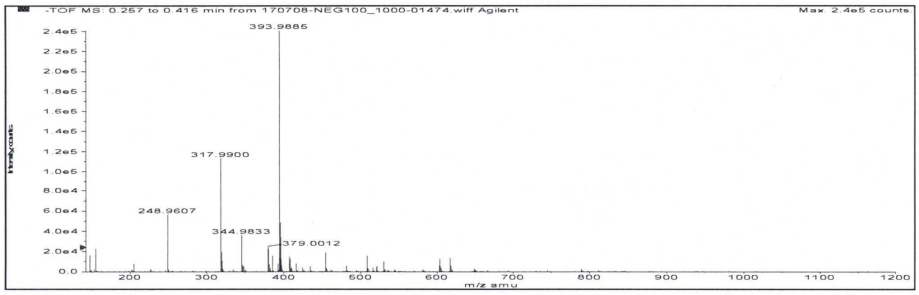

Peak# : 1 Experiment# : 1 Retention Time : 0.30 min

HRMS

Empirical Formula Confirmation Report Page 1 of 1

Sample Name: YG1-066 Sample Location: P1-A-01 Sample Id: YG1-066 Operator: EasyAccess
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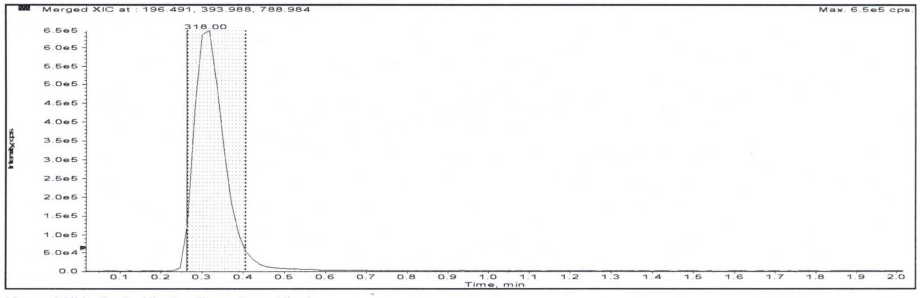
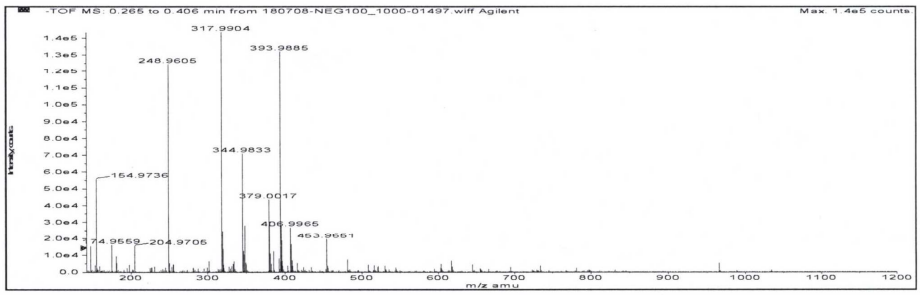
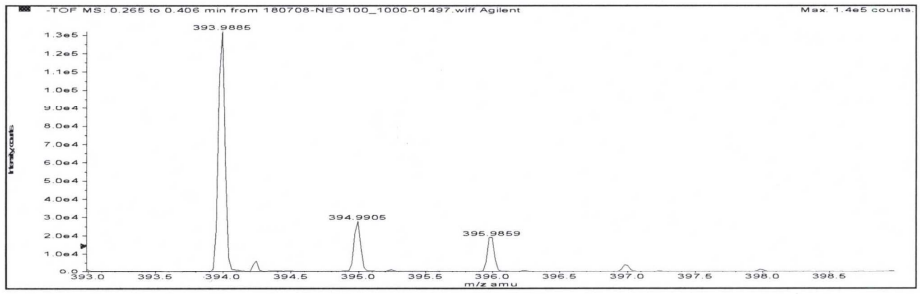


Merged XIC, Period# : 1 Experiment# : 1





Formula	Compound name	Mass	Peak RT (min)	Peak area	Description
C16H13NO5S3	--	394.99558	0.31	3.23556 E6	--

Species	Abundance (counts)	Ion Mass	Measured Mass	Error (mDa)	Error (ppm)	Ret. Time Error (min)
[M-H]-	135068.67	393.98831	393.98854	0.23500	0.60	--

Friday, July 18, 2008 7:27:22 AM

HPLC

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\HTS Chemistry\Data\Yiyu\proteasome\YG1-066 CH3CN 35 H2O 65 TFA 0.1 1ml 30 min.met 7-23-2010 2-45-30 PM.dat
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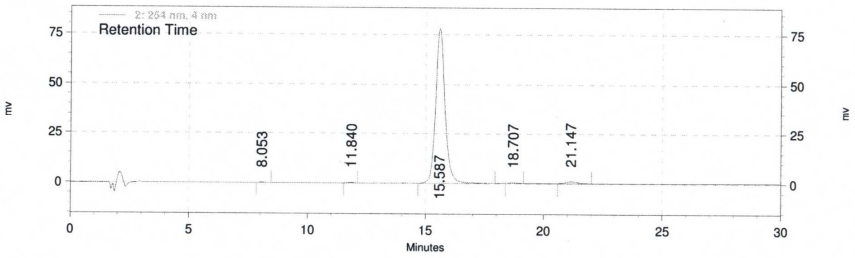
Printed: 7/23/2010 3:19:59 PM



2: 254 nm, 4 nm Results

Retention Time	Area	Area %
8.053	4427	0.20
11.840	4423	0.20
15.587	2149247	97.63
18.707	7808	0.35
21.147	35511	1.61
Totals	2201416	100.00

---< General Method Parameters >----------

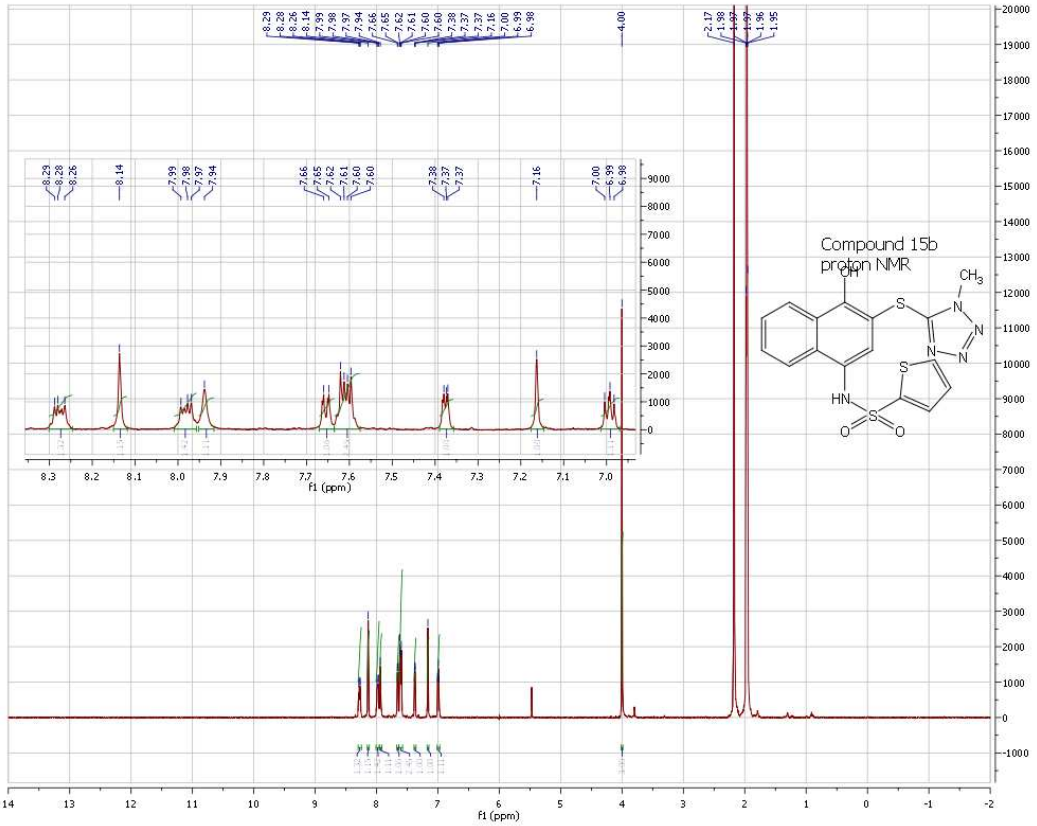
No items selected for this section

---< 2: 254 nm, 4 nm >----------

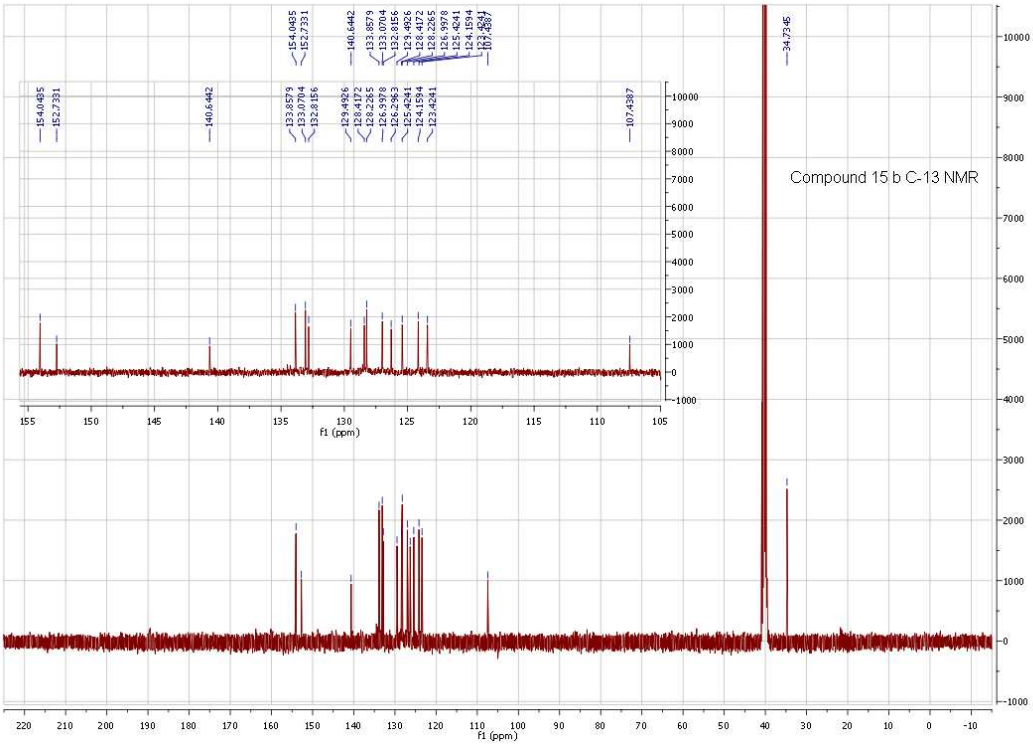
No items selected for this section

2. Compound 15b

^{1}H NMR



^{13}C NMR



LC-MS

Sample#: YG1-159-1 Sample Location: P1-B-02 Sample Id: YG1-159-1 Operator: EasyAccess
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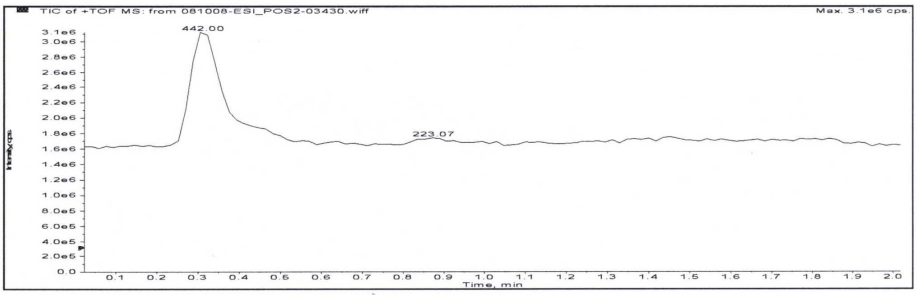

Period# : Average of all periods **Experiment# : Average of all experiments**

Peak#	Experiment#	Time	Area	Most Abundant Masses/scan
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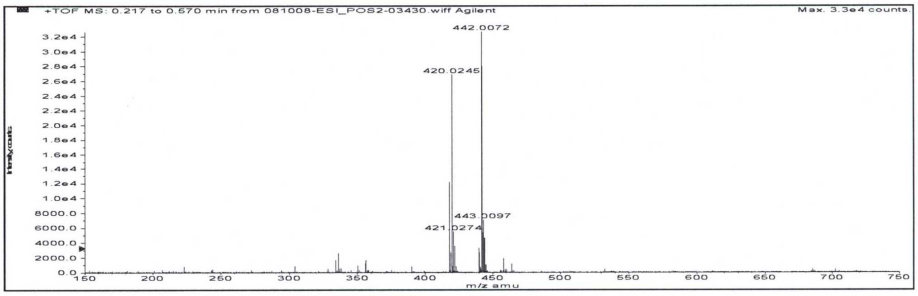

Peak# : 1 Experiment# : 1 Retention Time : 0.31 min

HRMS

Empirical Formula Confirmation Report

Sample Name: YG1-159-1 Sample Location: P1-C-06 Sample Id: YG1-159-1 Operator: EasyAccess
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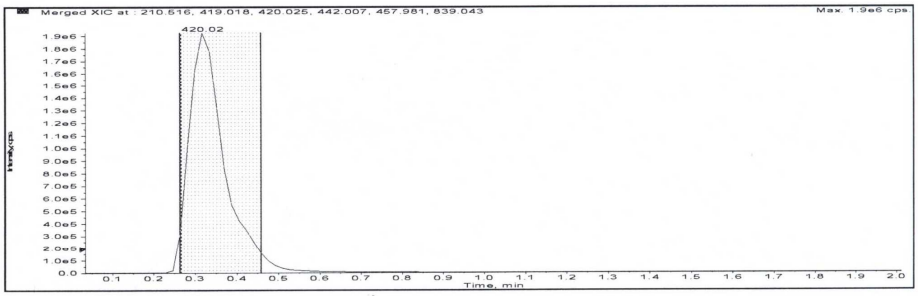

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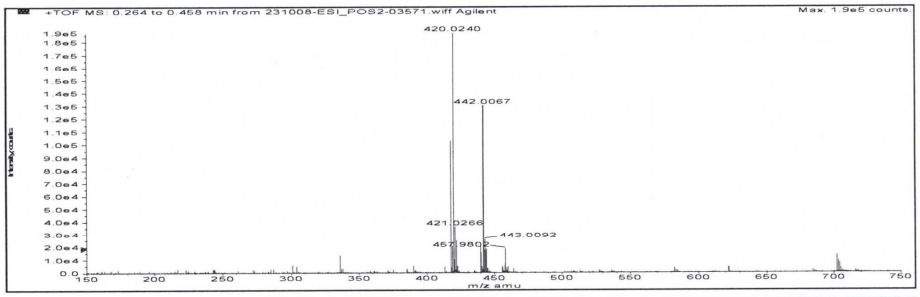

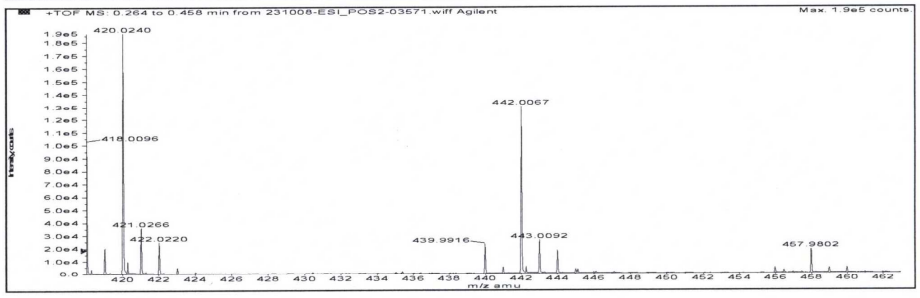

Formula	Compound name	Mass	Peak RT (min)	Peak area	Description
C16H13N5O3S3	--	419.01805	0.32	1.13325 E7	--

Species	Abundance (counts)	Ion Mass	Measured Mass	Error (mDa)	Error (ppm)	Ret. Time Error (min)
M+	20825.27	419.01750	419.01239	-5.11698	-12.21	--
[M+H]+	186981.04	420.02533	420.02397	-1.35572	-3.23	--
[M+Na]+	131203.64	442.00727	442.00667	-0.60229	-1.36	--
[M+K]+	20152.54	457.98121	457.98015	-1.05493	-2.30	--

Thursday, October 23, 2008 13:15:32 PM

HPLC

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\HTS Chemistry\Data\Yiyu\proteasome\YG1-159-1 CH3CN 40 H2O 60 TFA 0.1 1ml 40 min.met 8-20-2010 12-17-32 PM.dat
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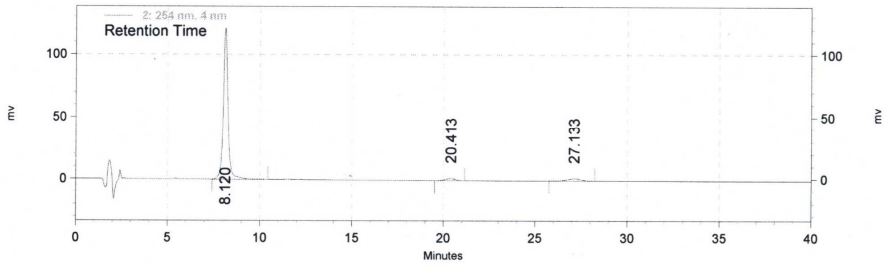
Printed: 11/2/2010 1:24:34 PM

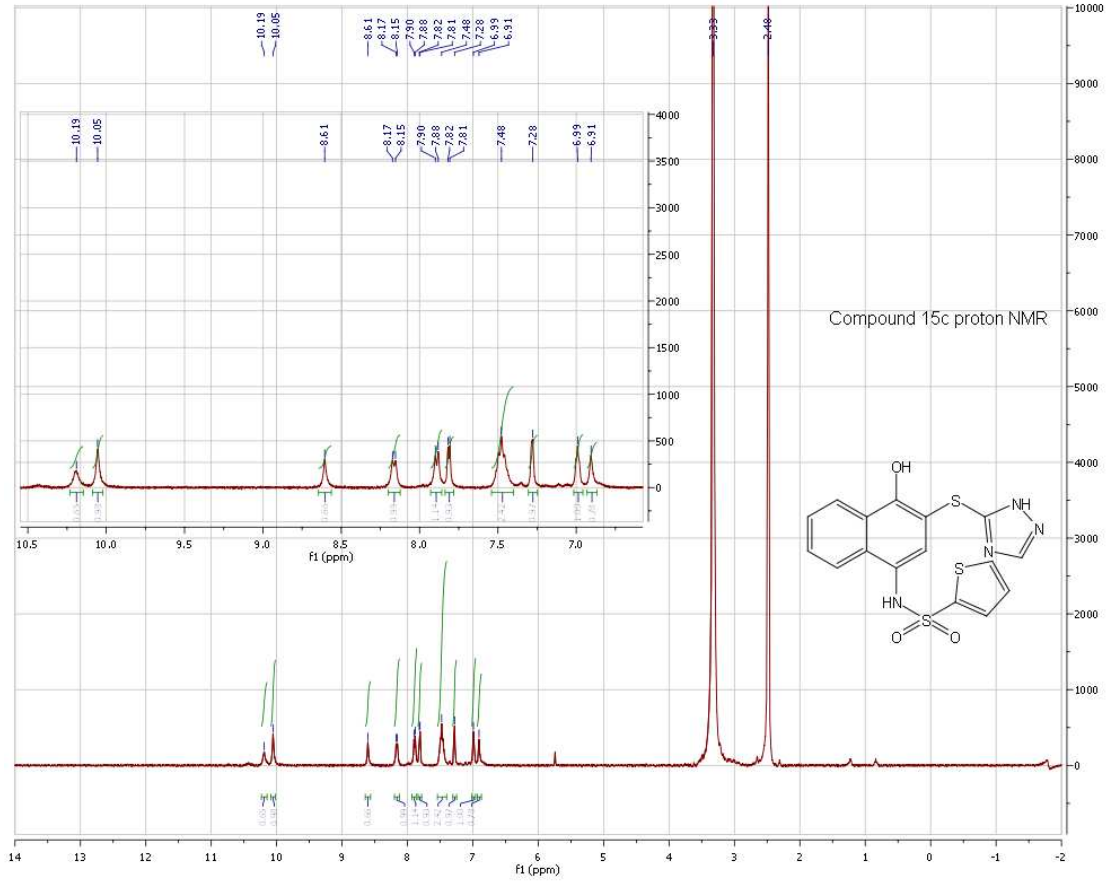


2: 254 nm, 4 nm Results

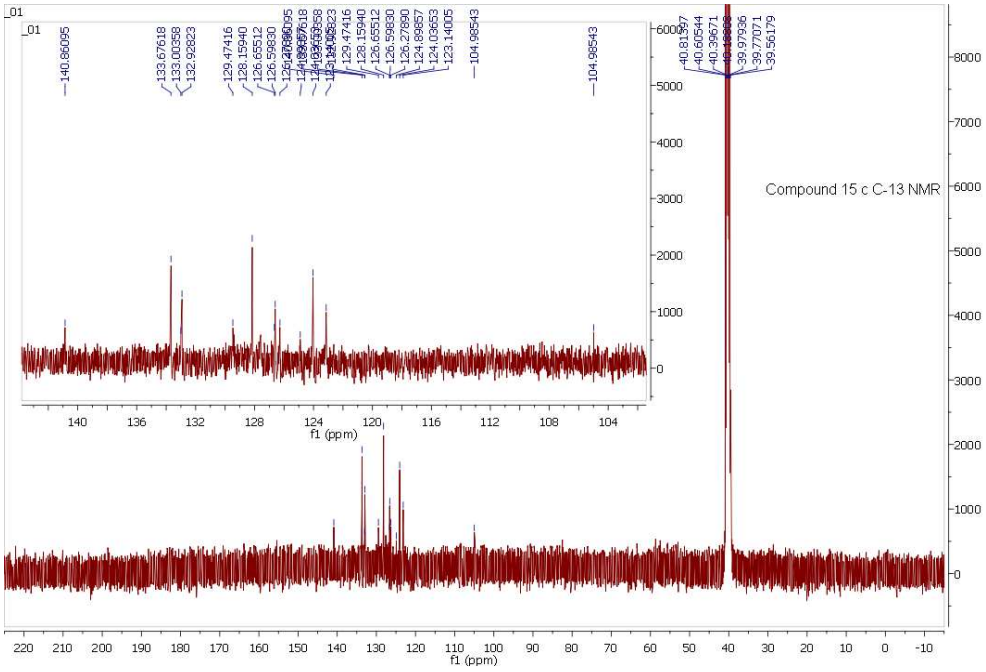
Retention Time	Area	Area %
8.120	2148498	93.14
20.413	62418	2.71
27.133	95754	4.15
Totals	2306670	100.00

3. Compound 15c

^{1}H NMR



^{13}C NMR



LC-MS

Sample#: YG1-159-2 Sample Location: P1-A-06 Sample Id: YG1-159-2 Operator: EasyAccess
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Method: D:\TOF_Data\damethods\EASY ACESS1.ANM\mass_list.xml

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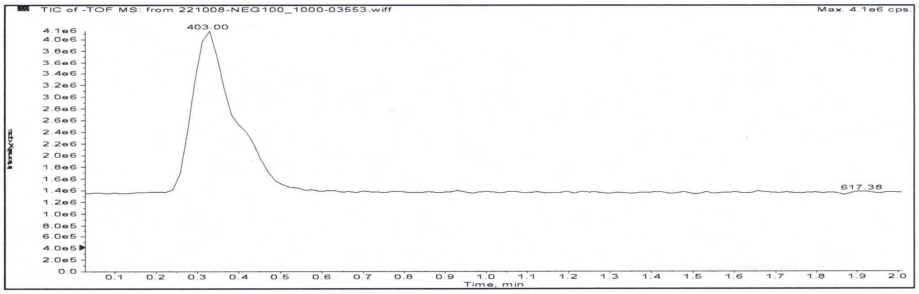

Period# : Average of all periods **Experiment# : Average of all experiments**

Peak#	Experiment#	Time	Area	Most Abundant Masses/scan
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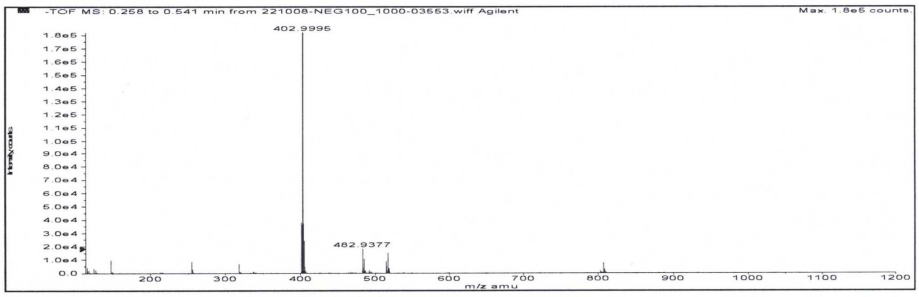

Peak# : 1 Experiment# : 1 Retention Time : 0.33 min

Empirical Formula Confirmation Report

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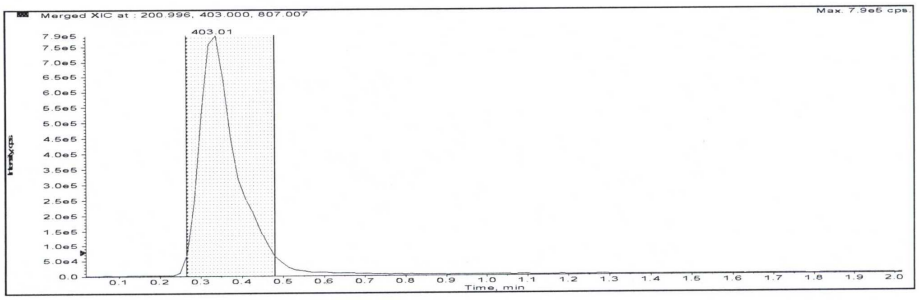
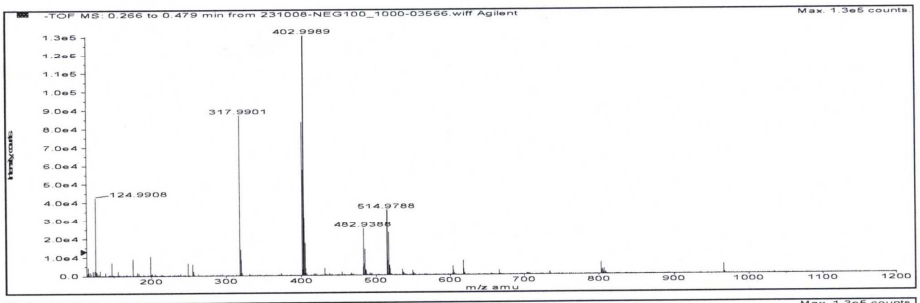
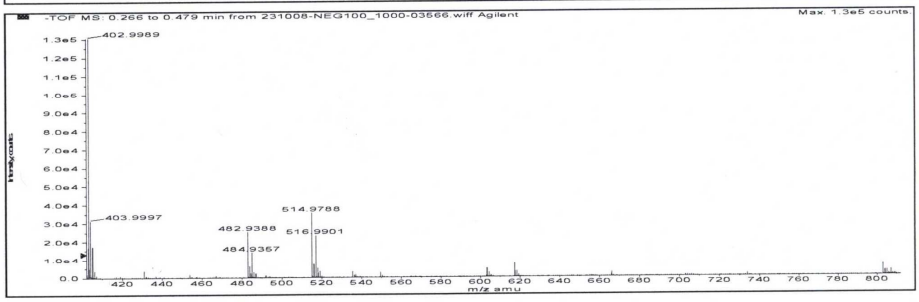


Merged XIC, Period# : 1 Experiment# : 1





Formula	Compound name	Mass	Peak RT (min)	Peak area	Description
C16H12N4O3S3	--	404.00715	0.33	4.93637 E6	--

Species	Abundance (counts)	Ion Mass	Measured Mass	Error (mDa)	Error (ppm)	Ret. Time Error (min)
[M-H]-	135976.83	402.99988	402.99892	-0.95878	-2.38	--
[2M-H]-	2926.85	807.00703	807.00339	-3.63637	-4.51	--

Thursday, October 23, 2008 12:52:22 PM

HPLC

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\HTS Chemistry\Data\Yiyu\proteasome\YG1-159-2 CH3CN 40 H2O 60 TFA 0.1 1ml 40 min.met 8-16-2010 6-50-06 PM
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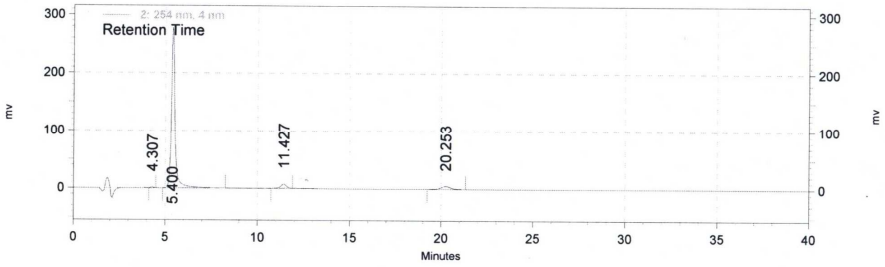
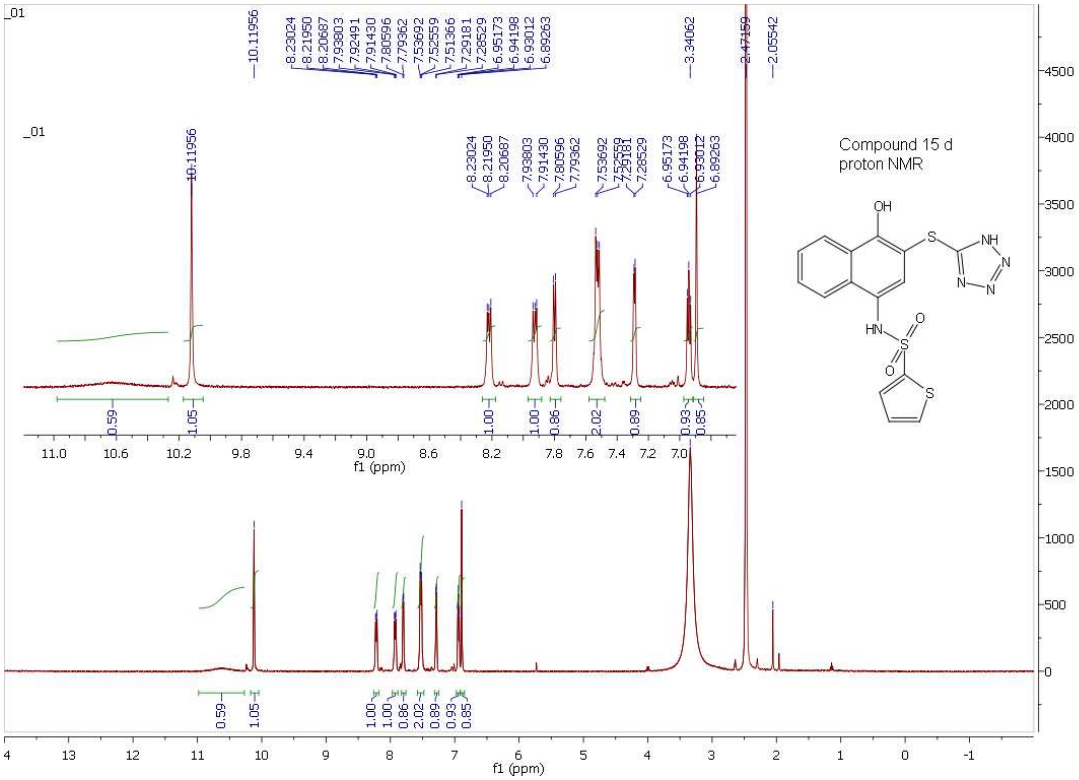


2: 254 nm, 4 nm Results

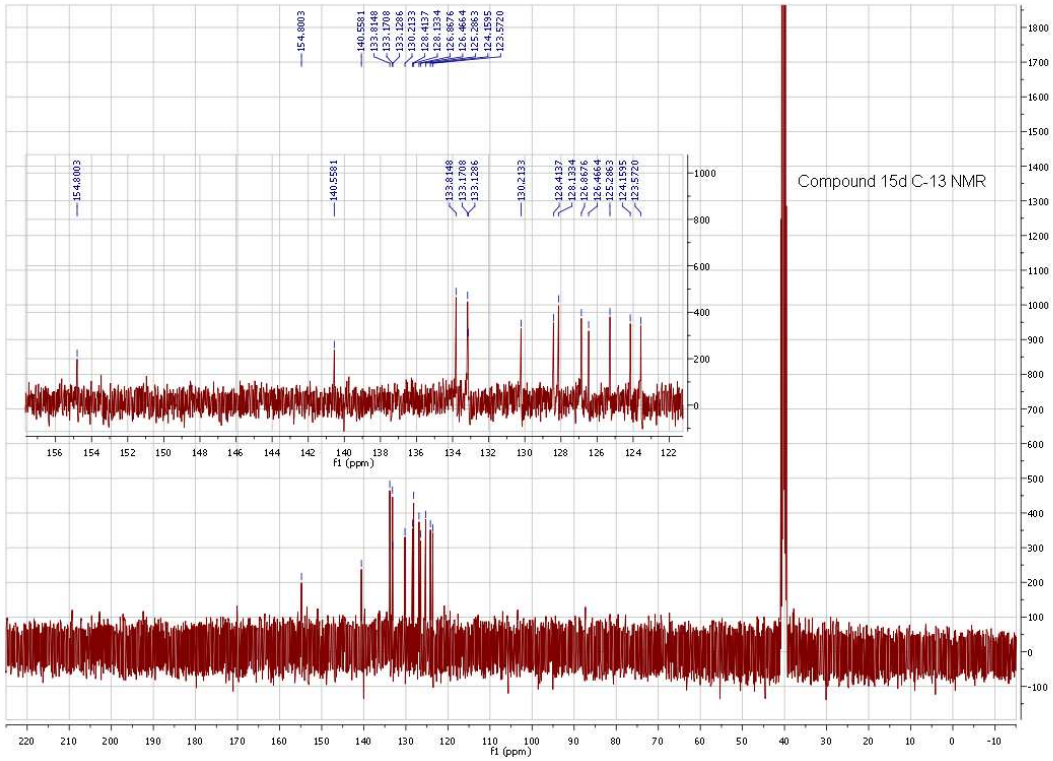
Retention Time	Area	Area %
4.307	15723	0.38
5.400	3817846	91.71
11.427	149246	3.59
20.253	180116	4.33
Totals	4162931	100.00

4. Compound 15d

^{1}H NMR



^{13}C NMR



LC-MS

Mass List Report

Sample#: YG4-040-2 Sample Location: P1-B-03 Sample Id: YG4-040-2 Operator: EasyAccess
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Method: D:\TOF_Data\damethods\EASY ACESS1.ANM\mass_list.xml

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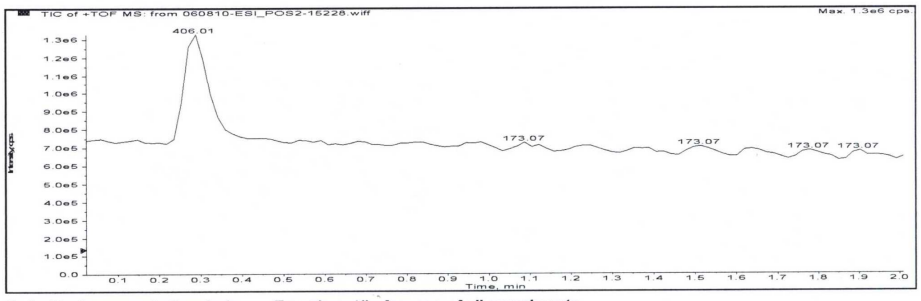

Period# : Average of all periods **Experiment# : Average of all experiments**

Peak#	Experiment#	Time	Area	Most Abundant Masses/scan
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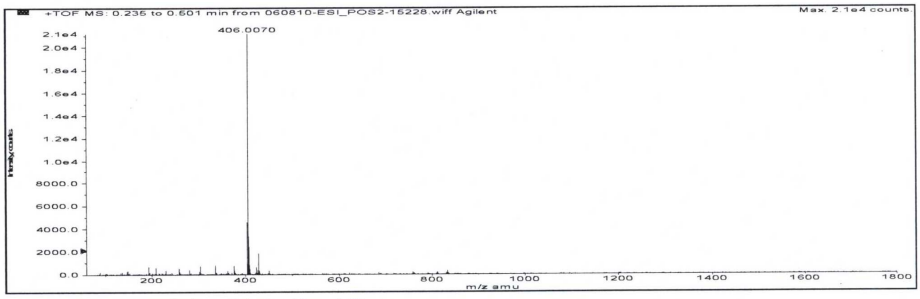

Peak# : 1 Experiment# : 1 Retention Time : 0.29 min

Friday, August 06, 2010 16:44:44 PM

HRMS

Empirical Formula Confirmation Report Page 1 of 1

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Data File Name: D:\PE Sciex Data\Projects\chemist\08-10\Data\060810-ESI_POS2-15235.wiff Acq Time: August 06 2010, 05:04:36 PM
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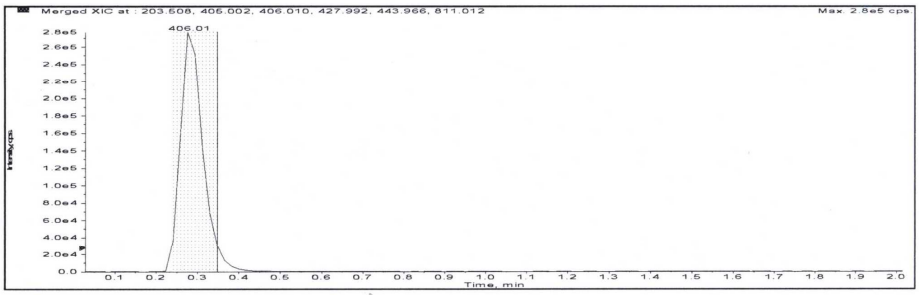
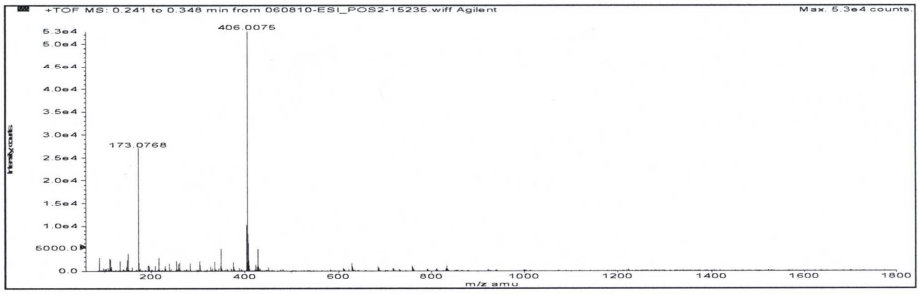
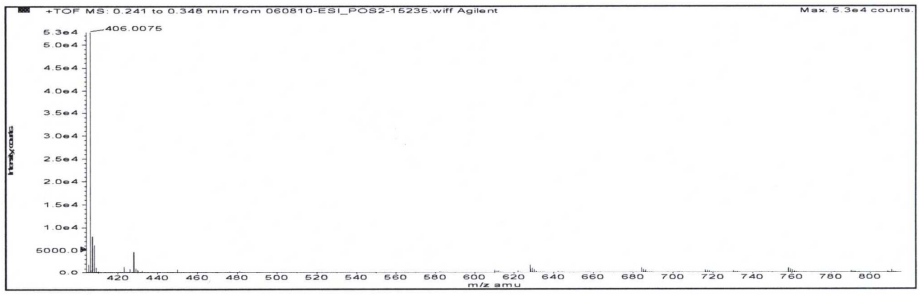


Merged XIC, Period# : 1 Experiment# : 1





Formula	Compound name	Mass	Peak RT (min)	Peak area	Description
C15H11N5O3S3	--	405.00240	0.28	1.05080 E6	--

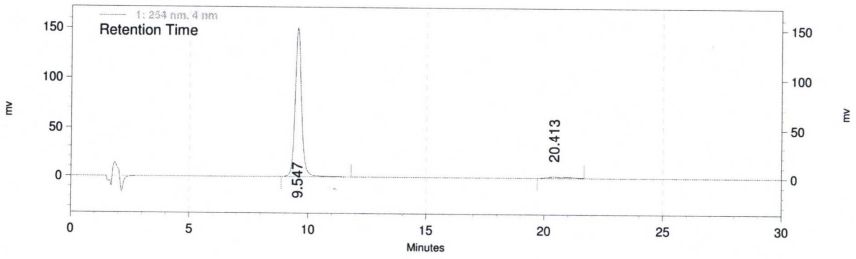
Species	Abundance (counts)	Ion Mass	Measured Mass	Error (mDa)	Error (ppm)	Ret. Time Error (min)
M+	1919.27	405.00185	404.99629	-5.55965	-13.73	--
[M+H]+	52837.78	406.00968	406.00754	-2.14179	-5.28	--
[M+Na]+	4772.00	427.99162	427.98902	-2.60337	-6.08	--

Friday, August 06, 2010 17:06:50 PM

HPLC

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\HTS Chemistry\Data\Yiyu\proteasome\YG4-040-2 CH3CN 35 H2O 65 TFA 0.1 1ml 30 min.met 8-9-2010 3-16-19 PM
Method: C:\EZChrom Elite\Enterprise\Projects\HTS Chemistry\Method\untitled.met
Acquired: 8/9/2010 3:17:03 PM
Printed: 10/29/2010 4:48:12 PM

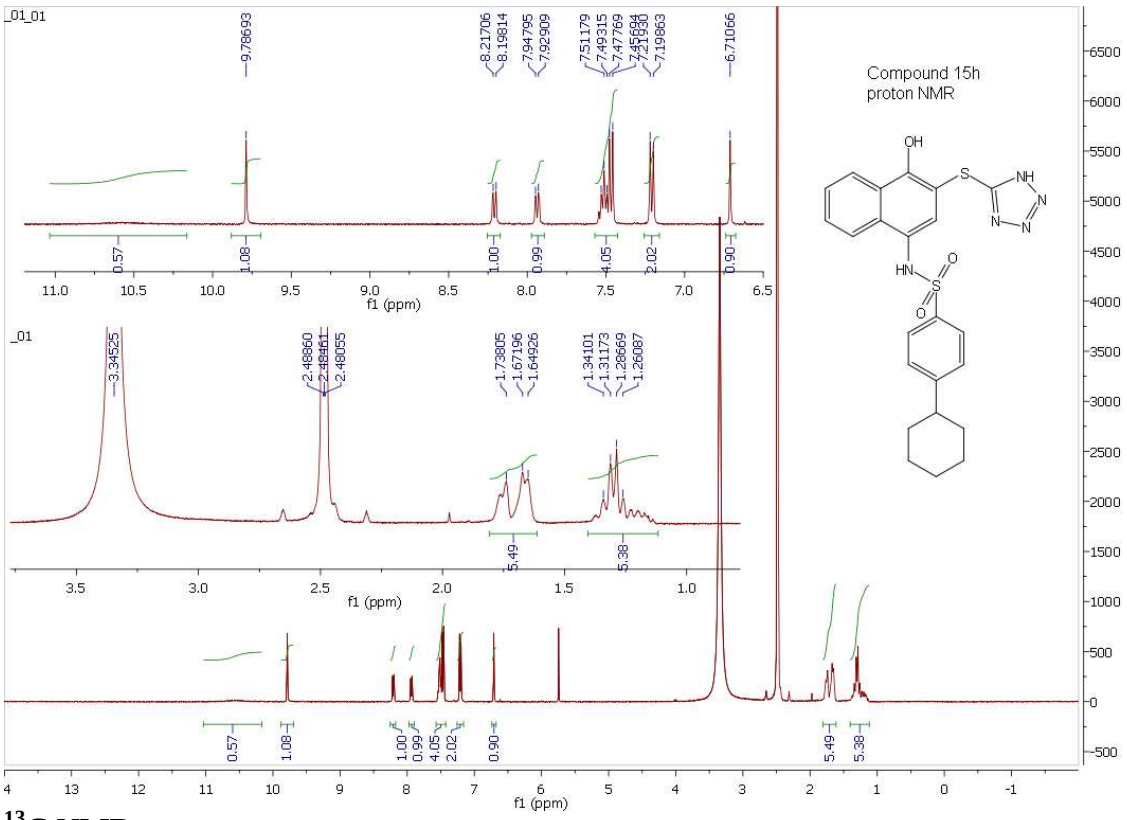


1: 254 nm, 4 nm
Results

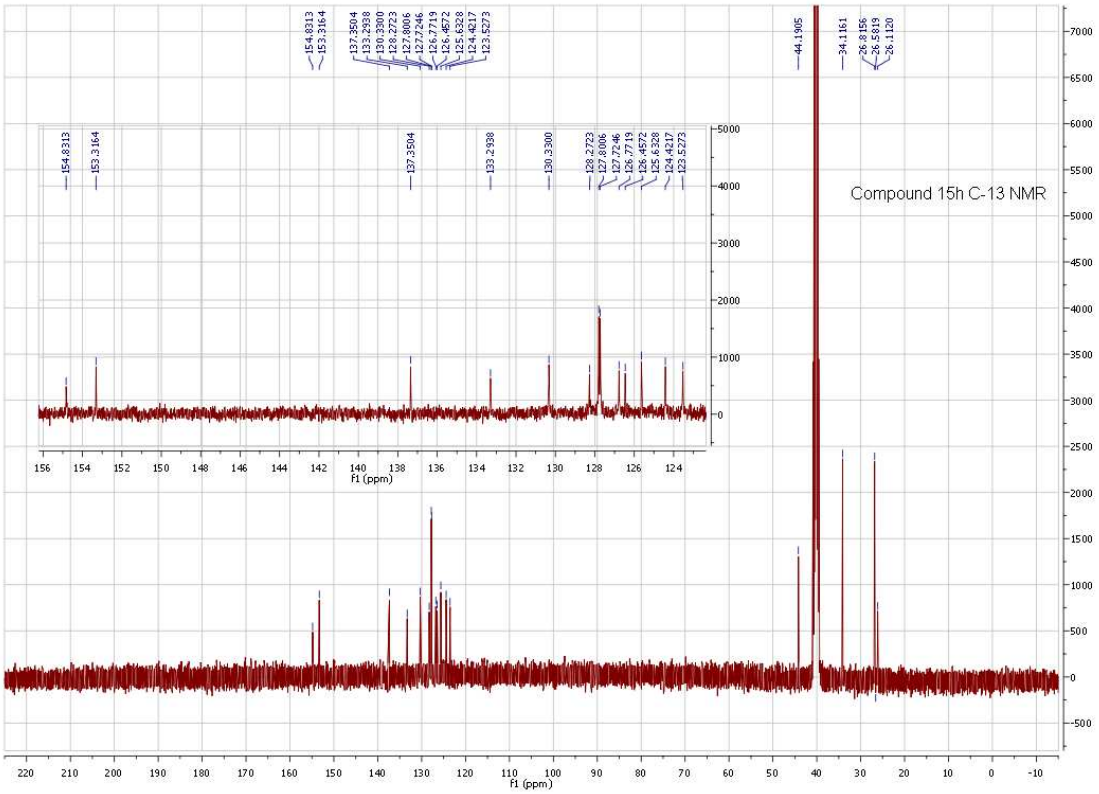
Retention Time	Area	Area %	Height	Height %
9.547	2841826	96.42	150553	98.90
20.413	105595	3.58	1678	1.10
Totals	2947421	100.00	152231	100.00

5. Compound 15h

^{1}H NMR



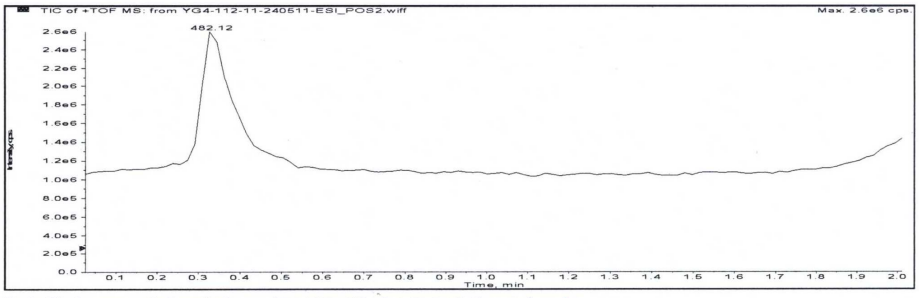
^{13}C NMR



LC-MS

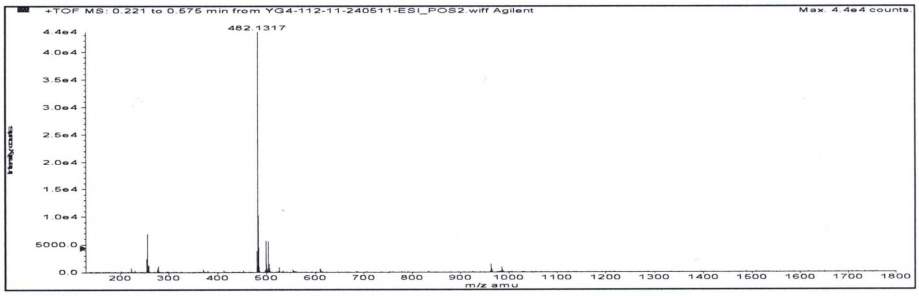
Sample#: YG4-112-1 Sample Location: P1-C-06 Sample Id: YG4-112-1 Operator: EasyAccess
Data File Name: D:\PE Sciex Data\Projects\Yiyu Ge\05-11\Data\YG4-112-11-240511-ESI_POS2.wiff Acq Time: May 24 2011, 01:45:35 PM
Method: D:\TOF_Data\damethods\EASY ACESS1.ANM\mass_list.xml

One or more scans have failed IRM. Review the data file for details.



Period# : Average of all periods **Experiment# : Average of all experiments**

Peak#	Experiment#	Time	Area	Most Abundant Masses/scan
1	1	0.33	8.58794 E6	482.13168



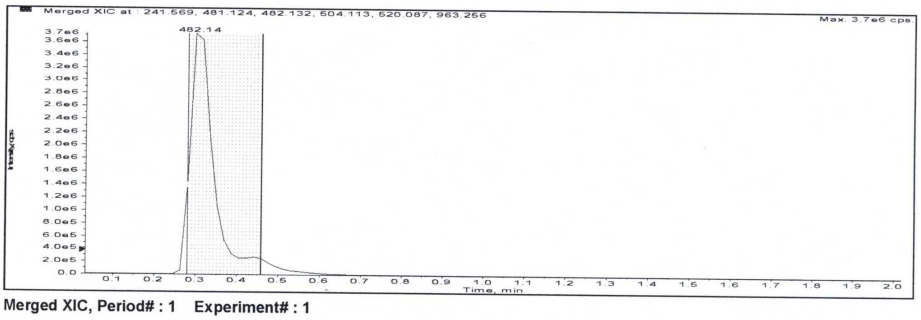
Peak# : 1 Experiment# : 1 Retention Time : 0.33 min

HRMS

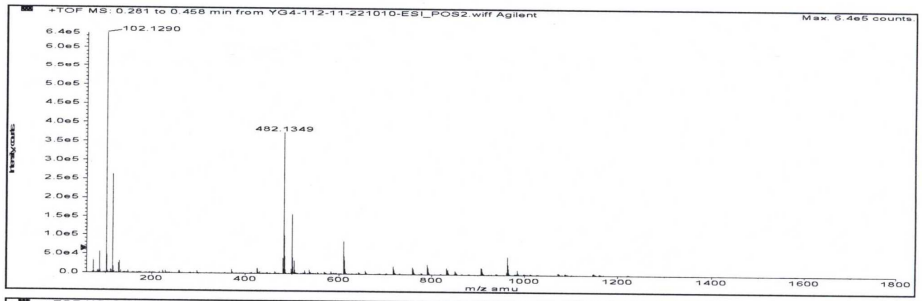
Empirical Formula Confirmation Report

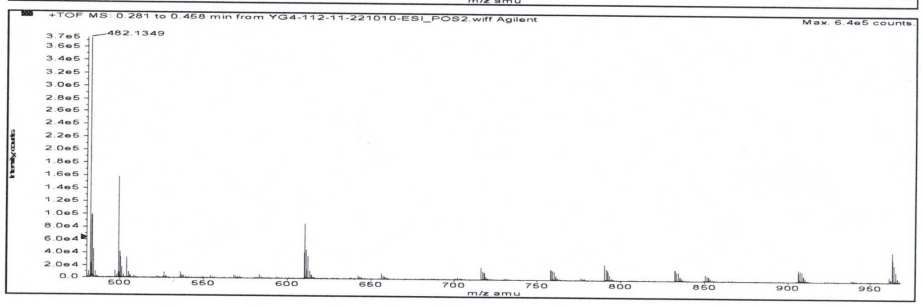
Sample Name: YG4-112-1 Sample Location: P1-D-01 Sample Id: YG4-112-1 Operator: EasyAccess
Data File Name: D:\PE Sciex Data\Projects\Yiyu Ge\10-10\Data\YG4-112-11-221010-ESI_POS2.wiff Acq Time: October 22 2010, 01:53:10 PM
Method: D:\TOF_Data\damethods\EASY ACESS2.ANM\efc.xml

One or more scans have failed IRM. Review the data file for details.



Merged XIC, Period# : 1 Experiment# : 1





Formula	Compound name	Mass	Peak RT (min)	Peak area	Description
C23H23N5O3S2	--	481.12423	0.31	1.50053 E7	--

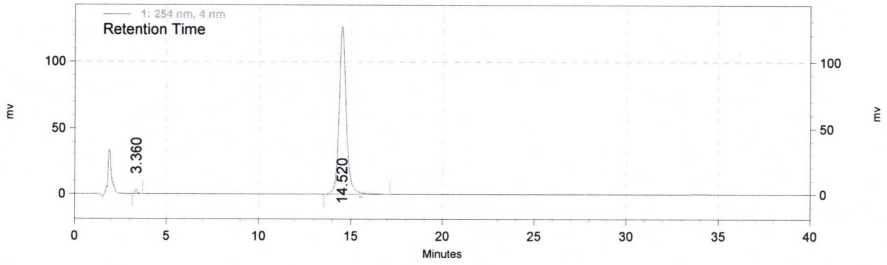
Species	Abundance (counts)	Ion Mass	Measured Mass	Error (mDa)	Error (ppm)	Ret. Time Error (min)
M+	11209.37	481.12368	481.12463	0.94722	1.97	--
[M+H]+	390907.19	482.13151	482.13487	3.36201	6.97	--
[M+Na]+	33888.36	504.11345	504.11685	3.40078	6.75	--
[2M+H]+	48438.21	963.25574	963.26119	5.44751	5.66	--

Friday, October 22, 2010 13:55:23 PM

HPLC

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\HTS Chemistry\Data\Yiyu\proteasome\YG4-112-1 CH3CN 50 H2O 50 TFA 0.1 1ml 40 min.met 11-1-2010 9-39-31 PM
Method: C:\EZChrom Elite\Enterprise\Projects\HTS Chemistry\Method\untitled.met
Acquired: 11/1/2010 9:40:17 PM
Printed: 11/2/2010 8:59:34 AM

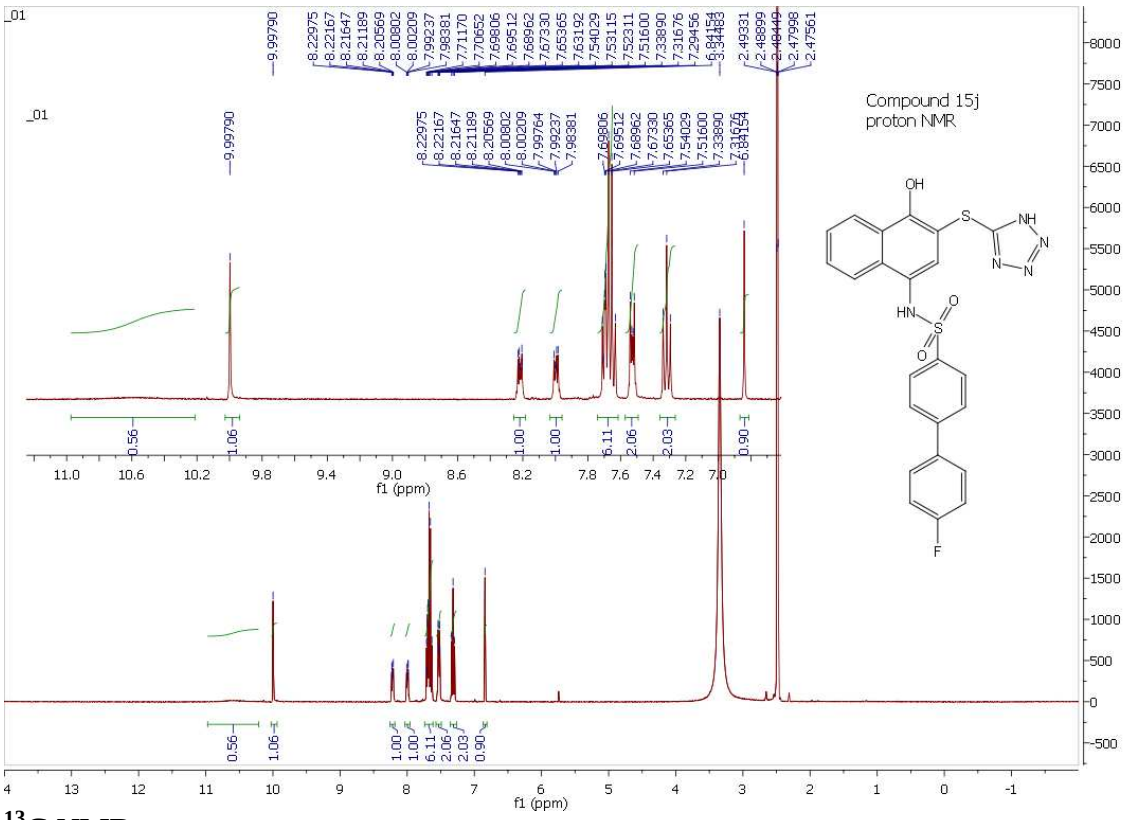


1: 254 nm, 4 nm
Results

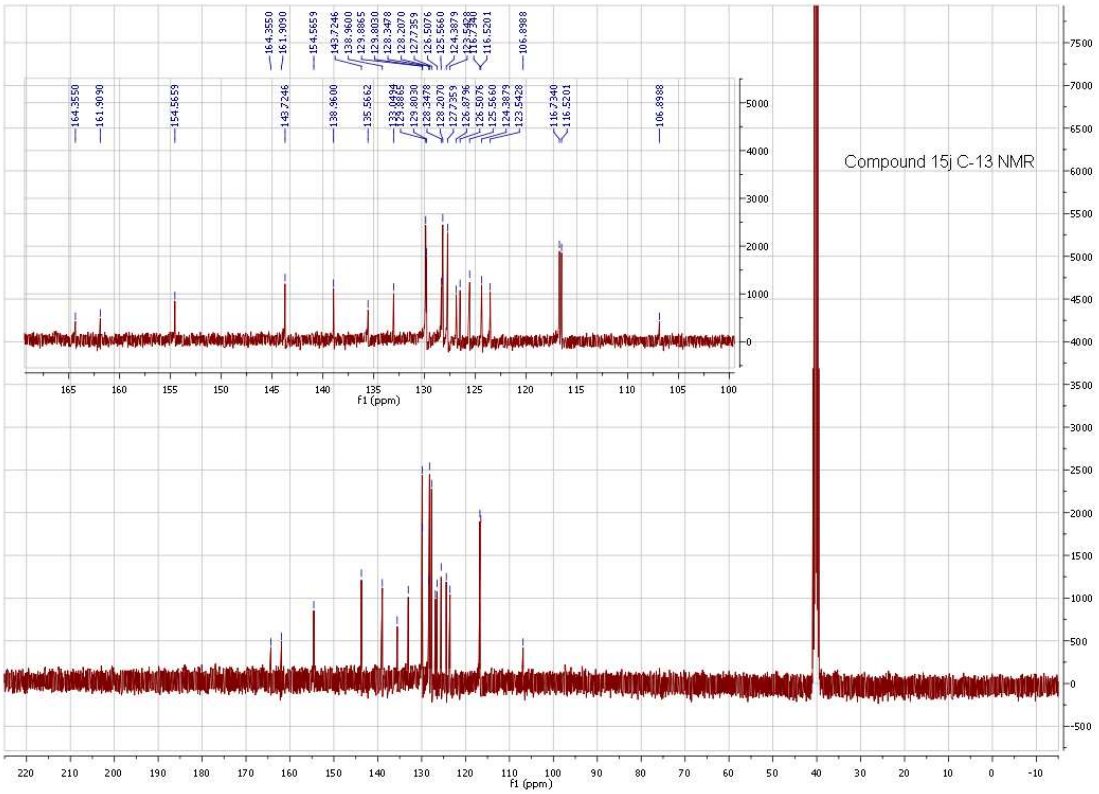
Retention Time	Area	Area %	Height	Height %
3.360	29592	0.86	3388	2.61
14.520	3429846	99.14	126423	97.39
Totals	3459438	100.00	129811	100.00

6. Compound 15j

^{1}H NMR



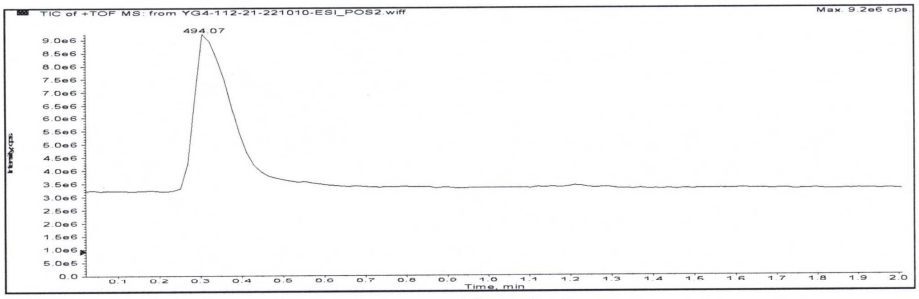
^{13}C NMR



LC-MS

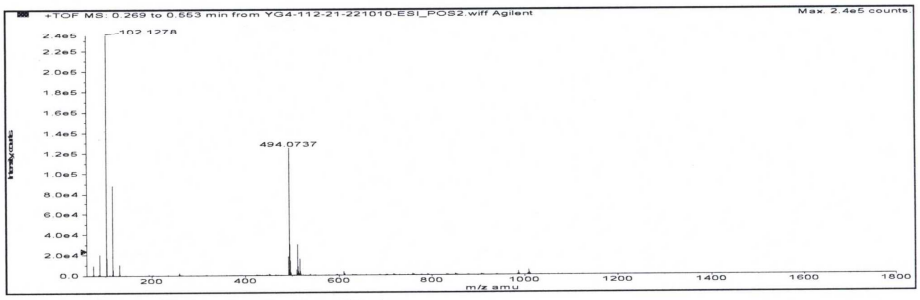
Mass List Report

Sample#: YG4-112-2 Sample Location: P1-B-01 Sample Id: YG4-112-2 Operator: EasyAccess
Data File Name: D:\PE Sciex Data\Projects\Yiyu Ge\10-10\Data\YG4-112-21-221010-ESI_POS2.wiff Acq Time: October 22 2010, 12:33:18 PM
Method: D:\TOF_Data\damethods\EASY ACESS1.ANM\mass_list.xml



Period# : Average of all periods **Experiment# : Average of all experiments**

Peak#	Experiment#	Time	Area	Most Abundant Masses/scan
1	1	0.31	3.84290 E7	102.12778



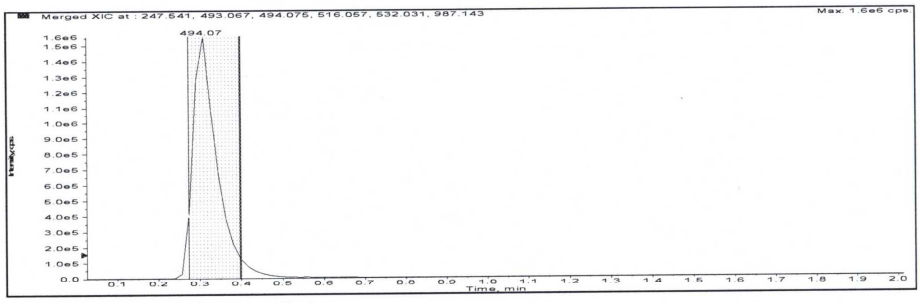
Peak# : 1 Experiment# : 1 Retention Time : 0.31 min

Friday, October 22, 2010 12:35:25 PM

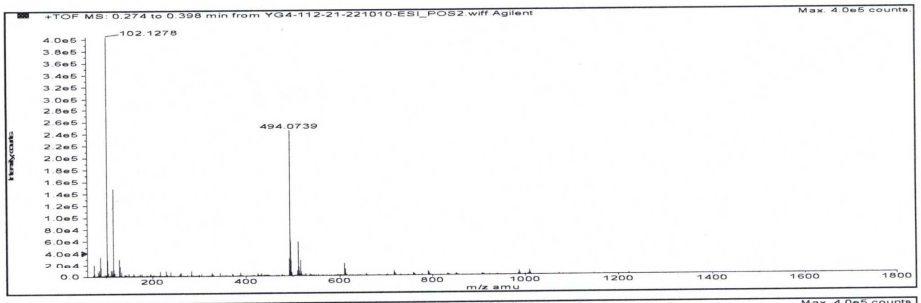
HRMS

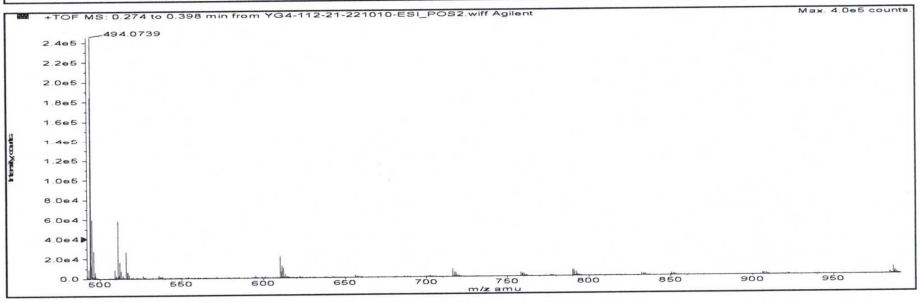
Empirical Formula Confirmation Report

Sample Name: YG4-112-2 Sample Location: P1-D-02 Sample Id: YG4-112-2 Operator: EasyAccess
Data File Name: D:\PE Sciex Data\Projects\Yiyu Ge\10-10\Data\YG4-112-21-221010-ESI_POS2.wiff Acq Time: October 22 2010, 01:56:27 PM
Method: D:\TOF_Data\damethods\EASY ACESS2.ANM\efc.xml



Merged XIC, Period# : 1 Experiment# : 1





Formula	Compound name	Mass	Peak RT (min)	Peak area	Description
C23H16FN5O3S2	--	493.06786	0.31	6.26626 E6	--

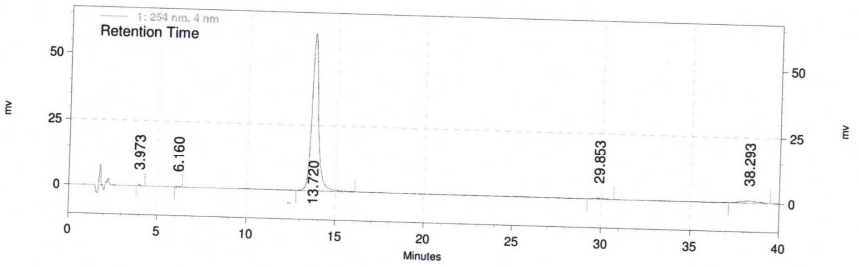
Species	Abundance (counts)	Ion Mass	Measured Mass	Error (mDa)	Error (ppm)	Ret. Time Error (min)
M+	9307.48	493.06731	493.06116	-6.14645	-12.47	--
[M+H]+	246536.55	494.07514	494.07394	-1.19645	-2.42	--
[M+Na]+	26985.03	516.05708	516.05637	-0.71237	-1.38	--
[2M+H]+	7303.21	987.14299	987.13984	-3.15505	-3.20	--

Friday, October 22, 2010 13:58:41 PM

HPLC

Area % Report Page 1 of 1

Data File: C:\EZChrom Elite\Enterprise\Projects\HTS Chemistry\Data\Yiyu\proteasome\YG4-112-2 CH3CN 45 H2O 55 TFA 0.1 1ml 40 min.met 10-27-2010 2-55-33 AM
Method: C:\EZChrom Elite\Enterprise\Projects\HTS Chemistry\Method\untitled.met
Acquired: 10/27/2010 2:56:14 AM
Printed: 10/29/2010 4:36:47 PM

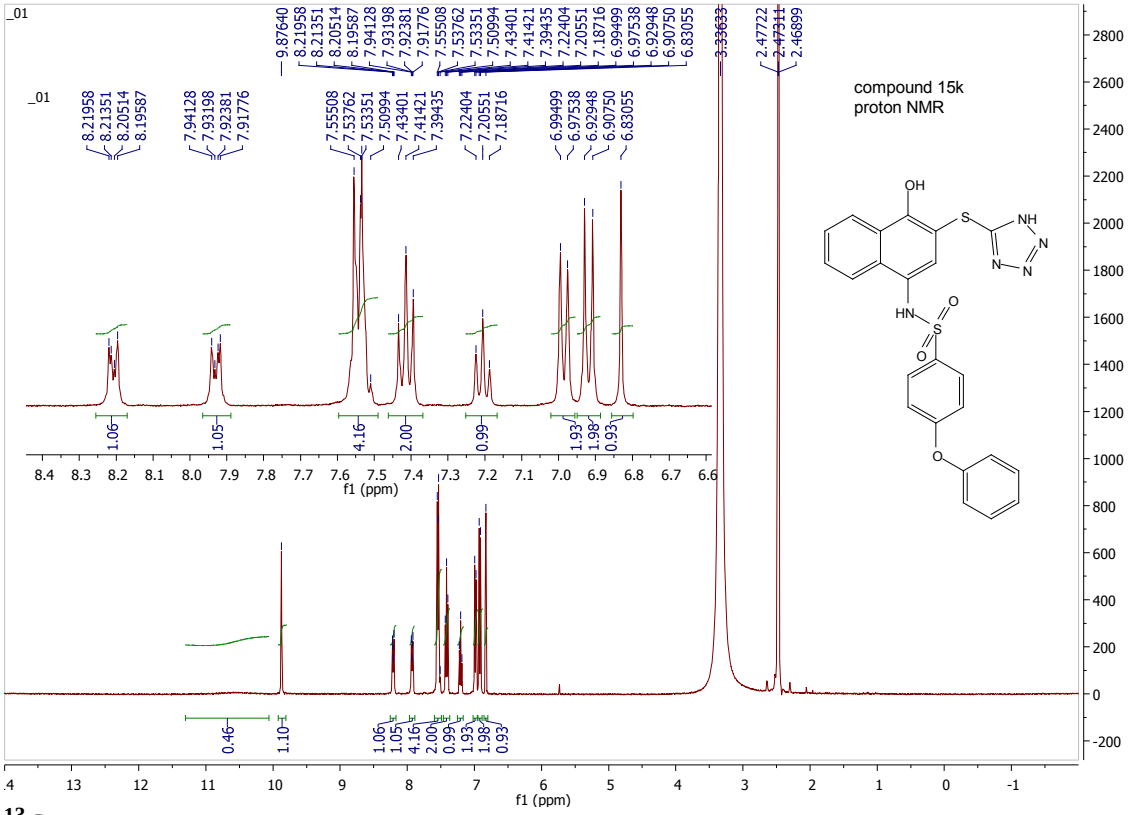


1: 254 nm, 4 nm
Results

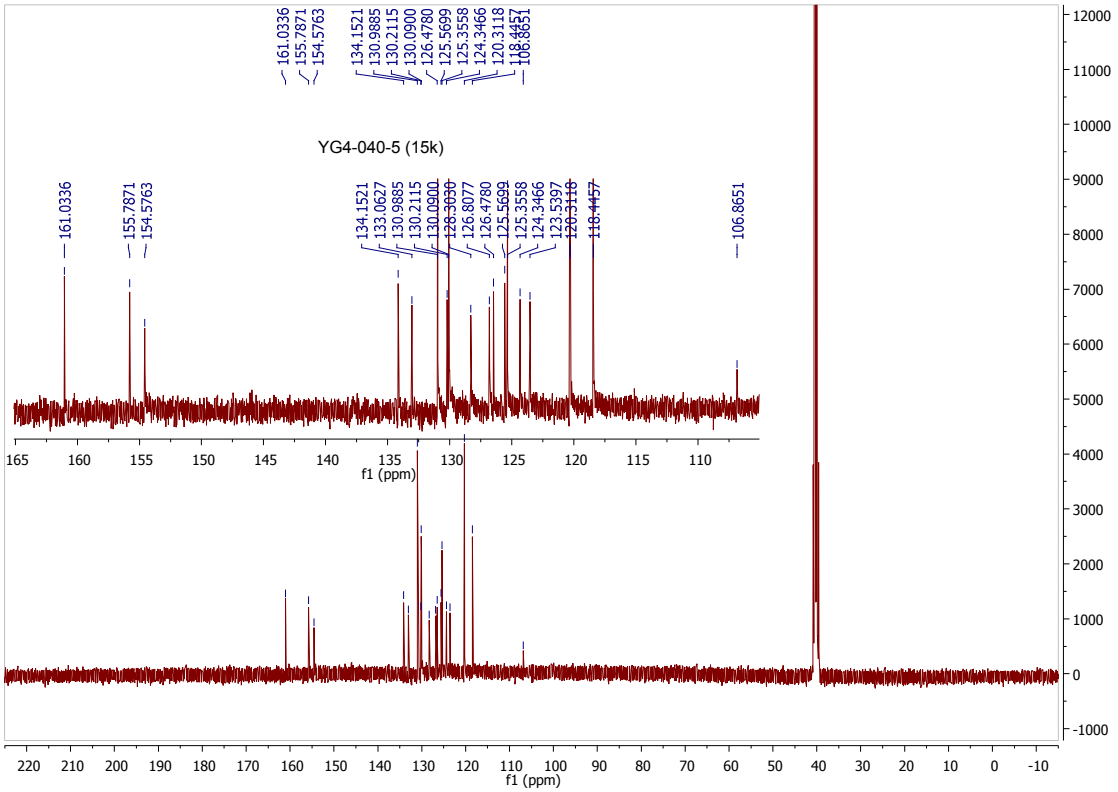
Retention Time	Area	Area %	Height	Height %
3.973	5146	0.31	567	0.92
6.160	3531	0.21	317	0.52
13.720	1585545	95.36	59413	96.66
29.853	15522	0.93	366	0.60
38.293	52990	3.19	802	1.30
Totals	1662734	100.00	61465	100.00

7. Compound 15k

^{1}H NMR



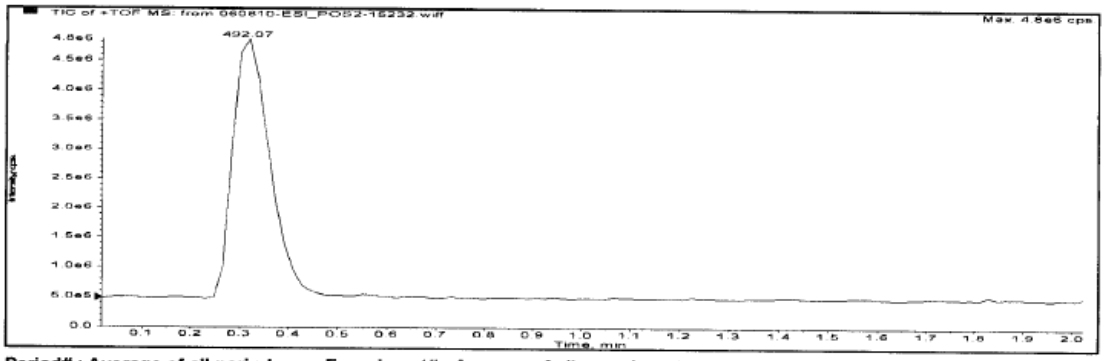
^{13}C NMR



LC-MS

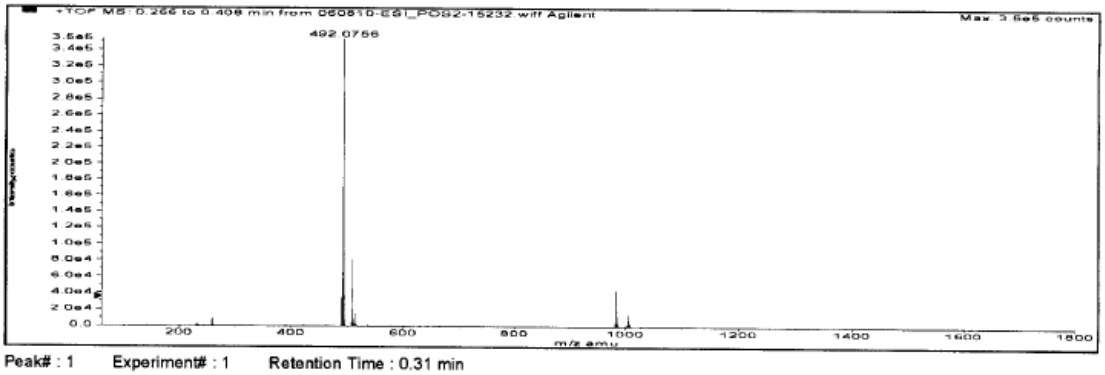
Sample#: YG4-040-5 Sample Location: P1-B-07 Sample Id: YG4-040-5 Operator: EasyAccess
Data File Name: D:\PE Sciex Data\Projects\chemist\08-10\Data\060810-ESI_POS2-15232.wiff Acq Time: August 06 2010, 04:55:10 PM
Method: D:\TOF_Data\damethods\EASY ACESS1.ANM\mass_list.xml

One or more scans have failed IRM. Review the data file for details.



Period# : Average of all periods **Experiment# : Average of all experiments**

Peak#	Experiment#	Time	Area	Most Abundant Masses/scan
1	1	0.31	2.29417 E7	492.07558

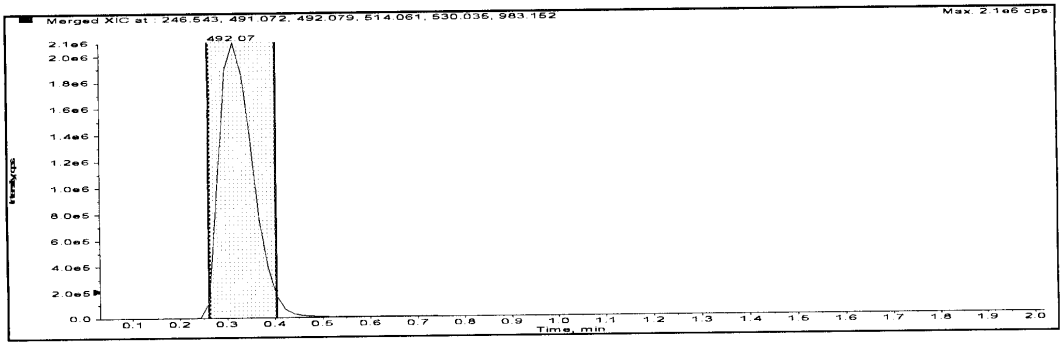


Peak# : 1 **Experiment# : 1** **Retention Time : 0.31 min**

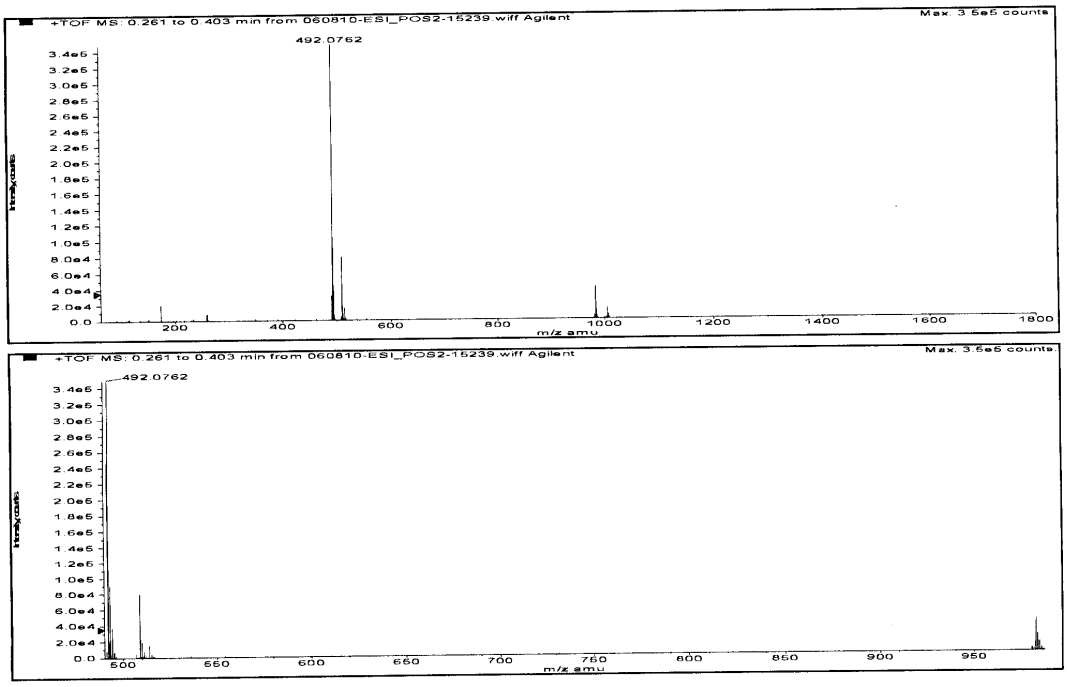
HRMS

Sample Name: YG4-040-5 Sample Location: P1-C-05 Sample Id: YG4-040-5 Operator: EasyAccess
Data File Name: D:\PE Sciex Data\Projects\chemist\08-10\Data\060810-ESI_POS2-15239.wiff Acq Time: August 06 2010, 05:17:39 PM
Method: D:\TOF_Data\damethods\EASY ACESS2.ANM\efc.xml

One or more scans have failed IRM. Review the data file for details.



Merged XIC, Period# : 1 Experiment# : 1



Formula	Compound name	Mass	Peak RT (min)	Peak area	Description
C23H17N5O4S2	--	491.07220	0.31	1.03613 E7	--

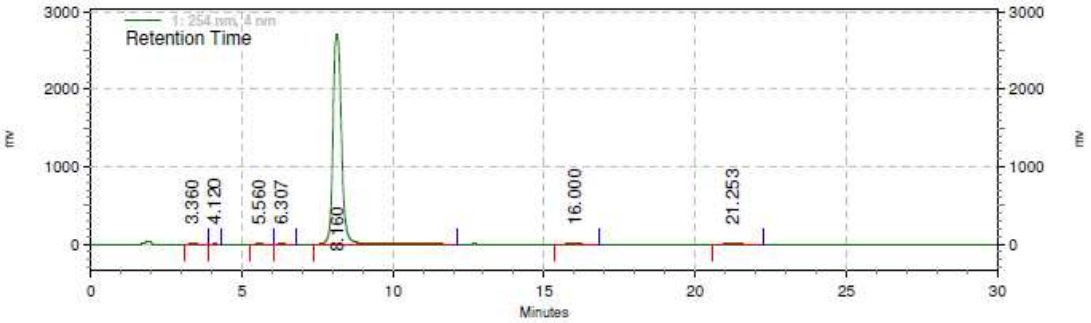
Species	Abundance (counts)	Ion Mass	Measured Mass	Error (mDa)	Error (ppm)	Ret. Time Error (min)
M+	8287.65	491.07165	491.06368	-7.96960	-16.23	--
[M+H]+	359946.57	492.07947	492.07620	-3.27546	-6.66	--
[M+Na]+	15508.62	514.06142	514.05876	-2.66056	-5.18	--
[2M+H]+	41565.94	983.15167	983.14487	-6.79518	-6.91	--

Friday, August 06, 2010 17:19:52 PM

HPLC

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\HTS Chemistry\Data\Yiyu\proteasome\YG4-040-5 CH3CN 50 H2O 50 TFA 0.1 1ml 30 min.met 8-9-2010 11-00-23 PM
Method: C:\EZChrom Elite\Enterprise\Projects\HTS Chemistry\Method\untitled.met
Acquired: 8/9/2010 11:01:10 PM
Printed: 10/29/2010 4:44:04 PM

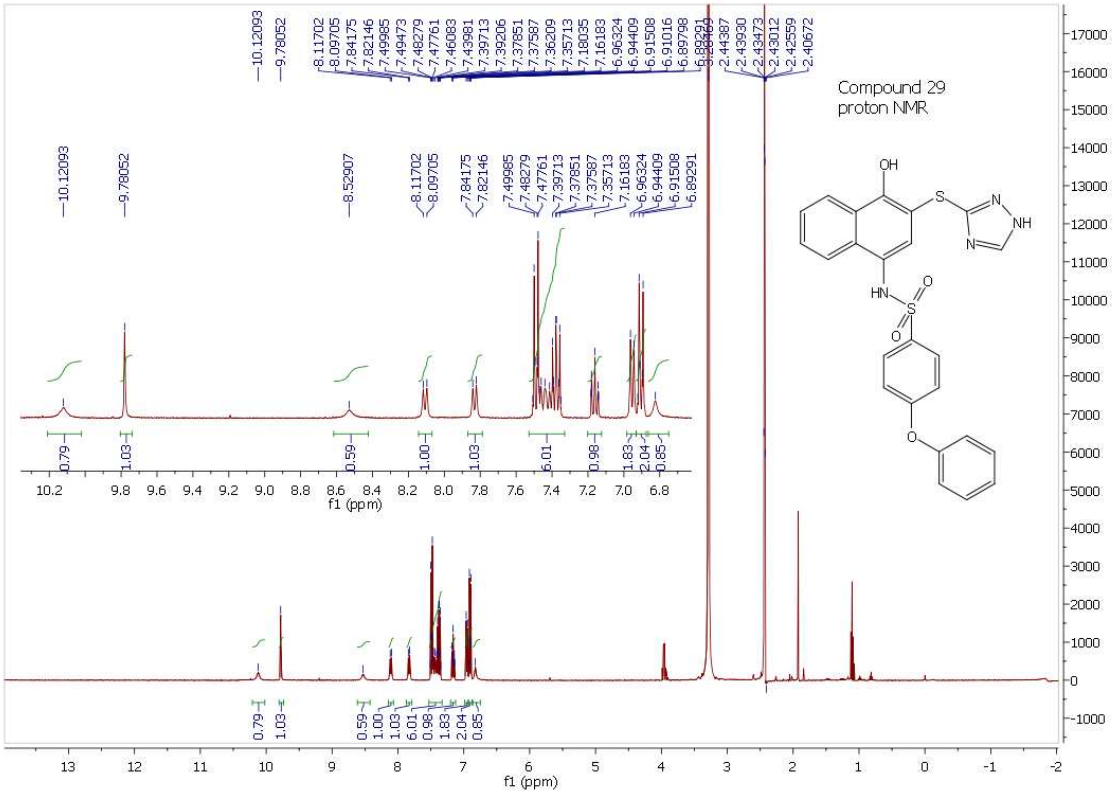


1: 254 nm, 4 nm
Results

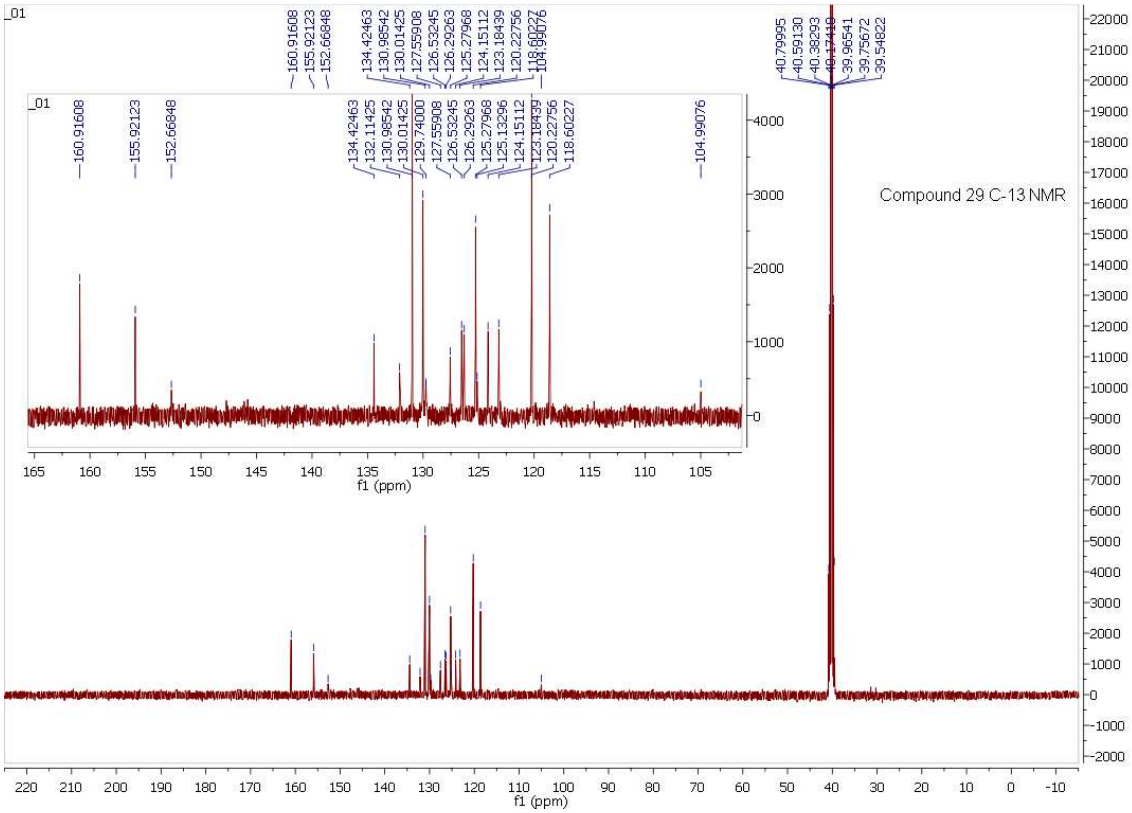
Retention Time	Area	Area %	Height	Height %
3.360	56218	0.11	5992	0.22
4.120	10924	0.02	1198	0.04
5.560	29549	0.06	2021	0.07
6.307	25592	0.05	2008	0.07
8.160	52618247	99.50	2700174	99.43
16.000	75004	0.14	2599	0.10
21.253	67909	0.13	1757	0.06
Totals	52883443	100.00	2715749	100.00

8. Compound 29

^{1}H NMR



^{13}C NMR

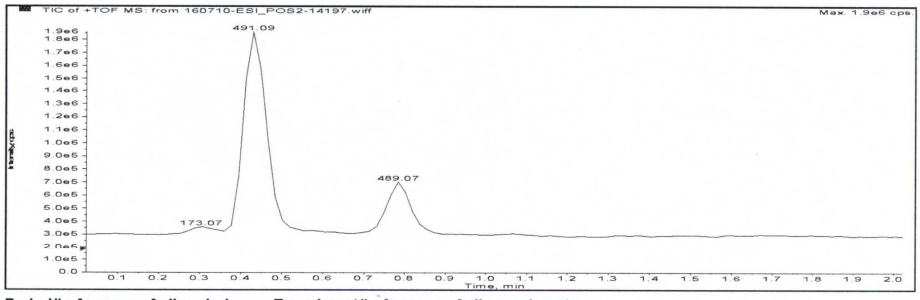


LC-MS

Mass List Report

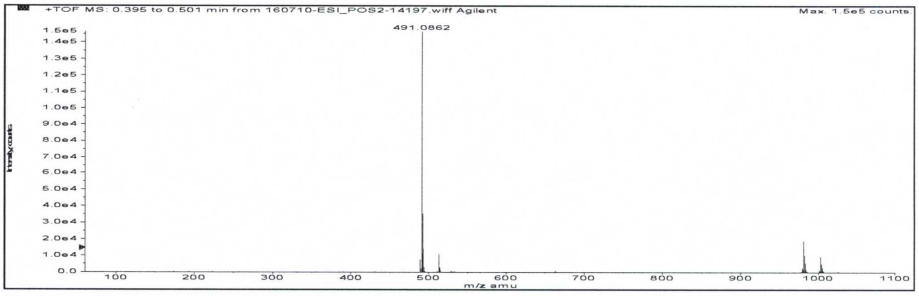
Sample#: YG4-019-2 Sample Location: P1-E-03 Sample Id: YG4-019-2 Operator: EasyAccess
Data File Name: D:\PE Sciex Data\Projects\chemist\07-10\Data\160710-ESI_POS2-14197.wiff Acq Time: July 16 2010, 04:38:27 PM
Method: D:\TOF_Data\damethods\EASY ACESS1.ANM\mass_list.xml

One or more scans have failed IRM. Review the data file for details.



Period# : Average of all periods **Experiment# : Average of all experiments**

Peak#	Experiment#	Time	Area	Most Abundant Masses/scan
1	1	0.43	6.31694 E6	491.08617



Peak# : 1 Experiment# : 1 Retention Time : 0.43 min

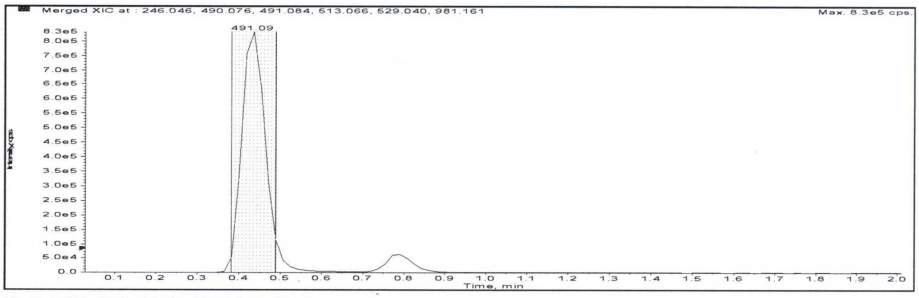
Friday, July 16, 2010 16:40:34 PM

HRMS

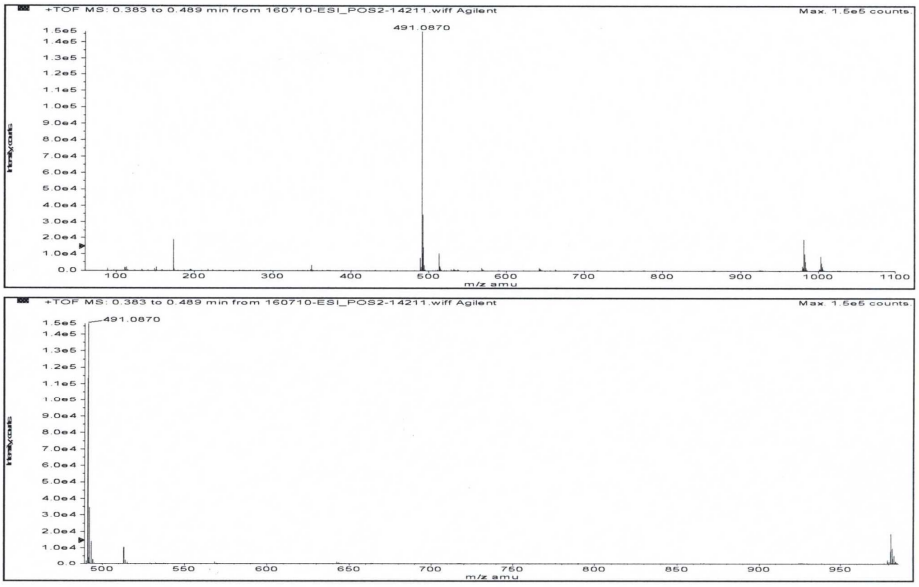
Empirical Formula Confirmation Report

Sample Name: YG4-019-1 Sample Location: P1-A-09 Sample Id: YG4-019-1 Operator: EasyAccess
Data File Name: D:\PE Sciex Data\Projects\chemist\07-10\Data\160710-ESI_POS2-14211.wiff Acq Time: July 16 2010, 05:44:33 PM
Method: D:\TOF_Data\damethods\EASY ACESS2.ANM\efc.xml

One or more scans have failed IRM. Review the data file for details.



Merged XIC, Period# : 1 Experiment# : 1



Formula	Compound name	Mass	Peak RT (min)	Peak area	Description
C24H18N4O4S2	--	490.07695	0.43	3.28869 E6	--

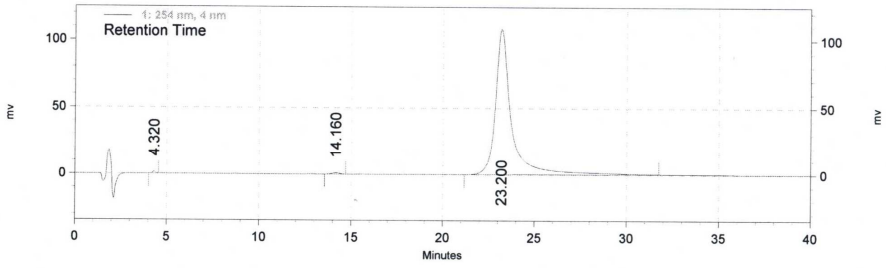
Species	Abundance (counts)	Ion Mass	Measured Mass	Error (mDa)	Error (ppm)	Ret. Time Error (min)
M+	2189.12	490.07640	490.07464	-1.76096	-3.59	--
[M+H]+	148950.13	491.08422	491.08697	2.74642	5.59	--
[M+Na]+	10451.45	513.06617	513.06970	3.52869	6.88	--
[2M+H]+	18871.08	981.16117	981.16489	3.72259	3.79	--

Friday, July 16, 2010 17:46:45 PM

HPLC

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\HTS Chemistry\Data\Yiyu\proteasome\YG4-019-1 CH3CN 40 H2O 60 TFA 0.1 1ml 40 min.met 8-18-2010 2-47-17 AM
Method: C:\EZChrom Elite\Enterprise\Projects\HTS Chemistry\Method\untitled.met
Acquired: 8/18/2010 2:47:59 AM
Printed: 10/29/2010 5:02:13 PM

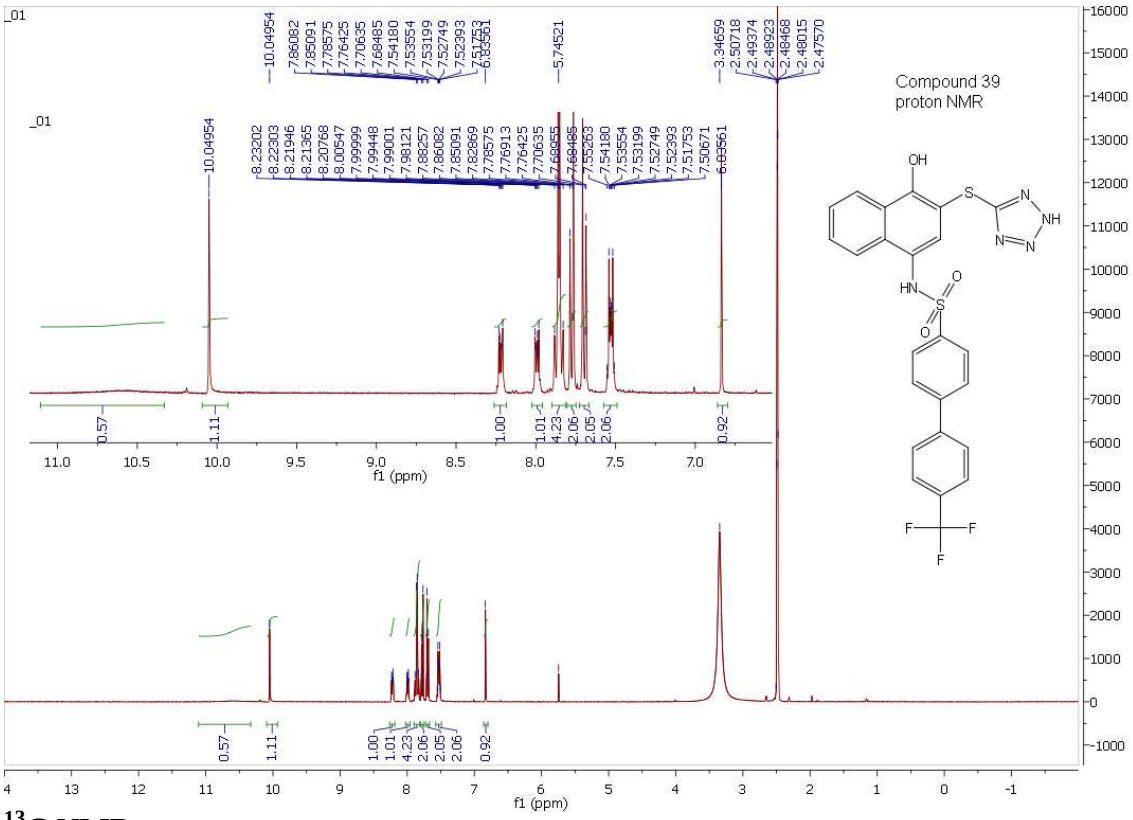


1: 254 nm, 4 nm
Results

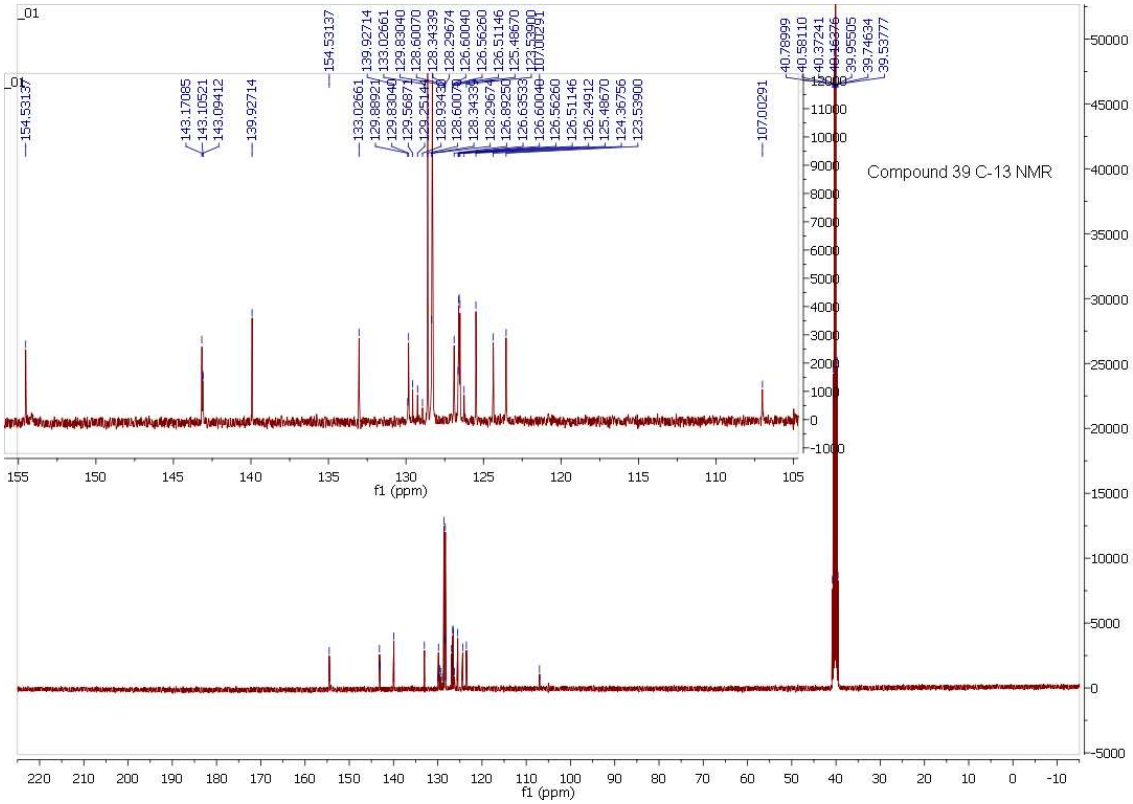
Retention Time	Area	Area %	Height	Height %
4.320	15514	0.23	1684	1.51
14.160	27804	0.41	1060	0.95
23.200	6692262	99.36	108660	97.54
Totals	6735580	100.00	111404	100.00

9. Compound 39

^{1}H NMR

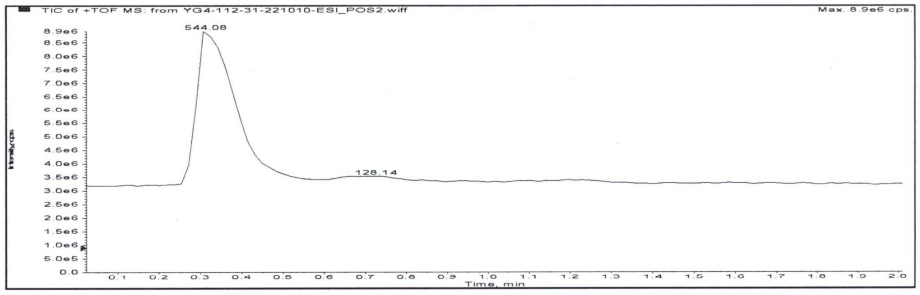


^{13}C NMR



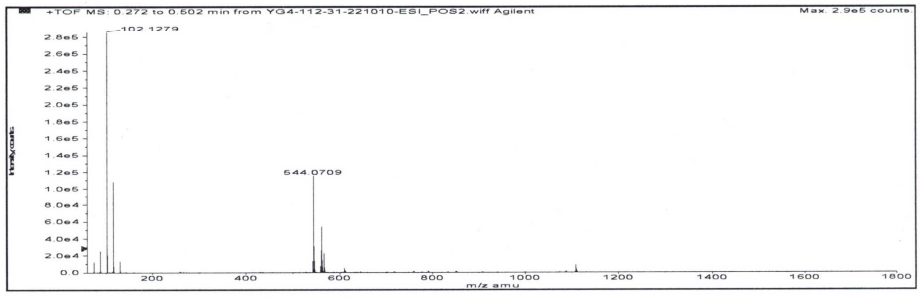
LC-MS

Sample#: YG4-112-3 Sample Location: P1-B-02 Sample Id: YG4-112-3 Operator: EasyAccess
Data File Name: D:\PE Sciex Data\Projects\Yiyu Ge\10-10\Data\YG4-112-31-221010-ESI_POS2.wiff Acq Time: October 22 2010, 12:36:27 PM
Method: D:\TOF_Data\damethods\EASY ACESS1.ANM\mass_list.xml



Period# : Average of all periods **Experiment# : Average of all experiments**

Peak#	Experiment#	Time	Area	Most Abundant Masses/scan
1	1	0.31	3.87168 E7	102.12791

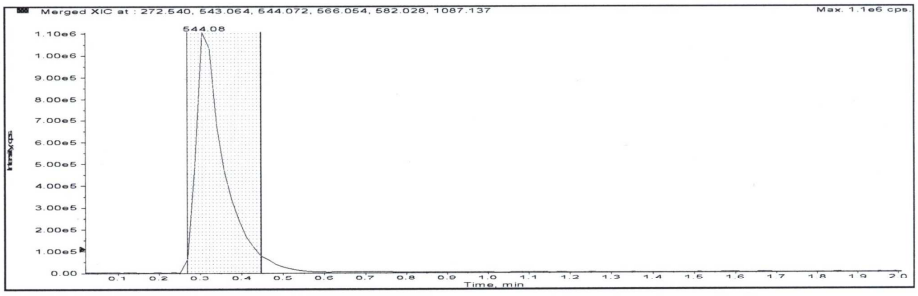


Peak# : 1 **Experiment# : 1** **Retention Time : 0.31 min**

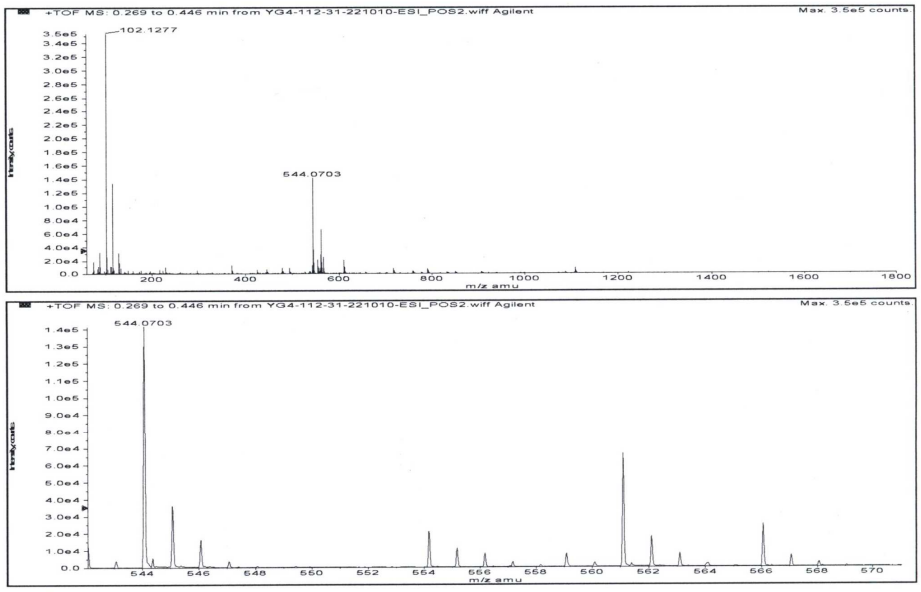
HRMS

Empirical Formula Confirmation Report

Sample Name: YG4-112-3 Sample Location: P1-D-03 Sample Id: YG4-112-3 Operator: EasyAccess
Data File Name: D:\PE Sciex Data\Projects\Yiyu Ge\10-10\Data\YG4-112-31-221010-ESI_POS2.wiff Acq Time: October 22 2010, 01:59:43 PM
Method: D:\TOF_Data\damethods\EASY ACESS2.ANM\efc.xml



Merged XIC, Period# : 1 Experiment# : 1



Formula	Compound name	Mass	Peak RT (min)	Peak area	Description
C24H16F3N5O3S2	--	543.06467	0.31	5.20252 E6	--

Species	Abundance (counts)	Ion Mass	Measured Mass	Error (mDa)	Error (ppm)	Ret. Time Error (min)
M+	3959.35	543.06412	543.05716	-6.95692	-12.81	--
[M+H]+	143213.52	544.07194	544.07035	-1.59507	-2.93	--
[M+Na]+	25292.71	566.05389	566.05250	-1.38360	-2.44	--

Friday, October 22, 2010 14:01:57 PM

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\HTS Chemistry\Data\Yiyu\proteasome\YG4-112-3 CH3CN 50 H2O 50 TFA 0.1 1ml 30 min.met 10-27-2010 10-35-25 AM
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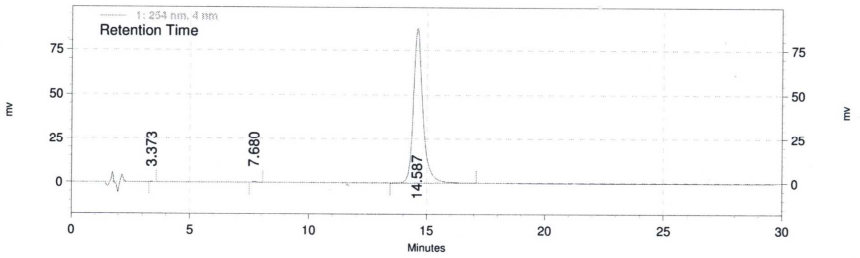
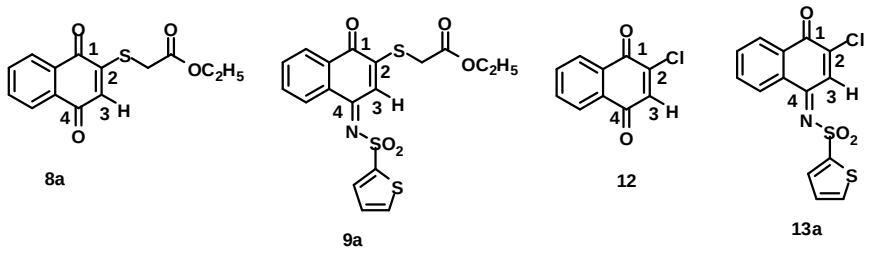
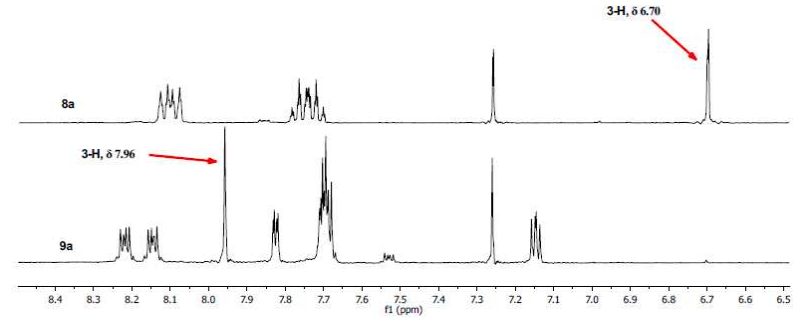


1: 254 nm, 4 nm
Results

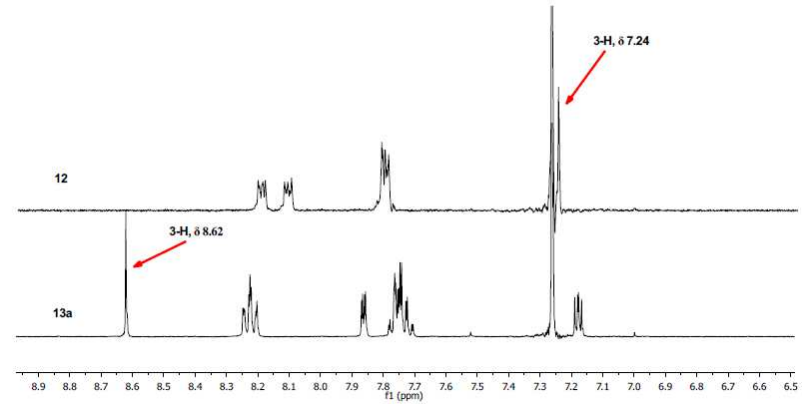
Retention Time	Area	Area %	Height	Height %
3.373	2888	0.12	392	0.44
7.680	7260	0.29	524	0.59
14.587	2464588	99.59	87415	98.96
Totals	2474736	100.00	88331	100.00

10. ^{1}H NMR analysis of intermediates **8a**, **9a**, **12** and **13a** to show the regioselectivity of coupling described in Scheme 2.





A



B

Figure 1: **A**, chemical shift (in $CDCl_3$, 400 MHz) of 3-H in compound **8a** (upper) and **9a** (lower); **B**, chemical shift (in $CDCl_3$, 400 MHz) of 3-H in compound **12** (upper) and **13a** (lower). These ^{1}H NMRs indicate coupling intermediate **8a** and **12** with thiophenesulfonamide proceeded regioselectively at the 4-carbonyl of the naphthoquinone ring.