Supporting Information

Discovery and Synthesis of Hydronaphthoquinones as Novel

Proteasome Inhibitors

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Sebti,^{†,§} and Harshani R. Lawrence^{*,†,‡}

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





LC-MS



S3

Empirical Formula Confirmation Report

Page 1 of 1

Sample Name: <u>YG1-066</u> Sample Location: <u>P1-A-01</u> Sample Id: <u>YG1-066</u> Operator: <u>EasyAccess</u> Data File Name: <u>D:\PE Sciex Data\Projects\chemist\07-08\Data\180708-NEG100_1000-01497.wiff</u> Acq Time: <u>July 18 2008, 07:25:11 AM</u> Mothod: <u>D:\TOF_Data\damethods\EASY ACESS2.ANM\efc.xml</u>

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





Friday, July 18, 2008

7:27:22 AM

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
 C:\EZChrom Elite\Enterprise\Projects\HTS Chemistry\Method\Yiyu\analytical\CH3CN 35 H2O

 C:\EZChrom Elite\Enterprise\Projects\HTS Chemistry\Data
 C:\EZChrom Elite\Enterprise\Projects\HTS Chemistry\Method\Yiyu\analytical\CH3CN 35 H2O

 35 Heo Go
 C:\EZChrom Ellecture

 Method:
 C:\EZChrom Ellecture

 65 TFA 0.1 Iml 40 min-postrun.met
 Acquired:

 Acquired:
 7/23/2010 2:47:47 PM

 Printed:
 7/23/2010 3:19:59 PM
 2: 254 nm, 4 Retention Time 75 75 50 - 50 À. N 25 11.840 18.707 21.147 8.053 25 15.587 0 0 15 Minutes 0 5 10 20 25 30

2: 254 nm, 4 nm Results

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

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

No items selected for this section

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

No items selected for this section

2. Compound 15b ¹H NMR



¹³C NMR



LC-MS



Wednesday, October 08, 2008

18:46:20 PM

HRMS

Page 1 of 1 Empirical Formula Confirmation Report Sample Name: YG1-159-1 Sample Location: P1-C-06 Sample Id: YG1-159-1 Operator: EasyAccess
Data File Name: D:YE Sciex Data\Projects\chemist\10-08\Data\231008-ESI_POS2-03571.wiff Acq Time: October 23 2008, 01:13:20 PM
Method: D:\TOF_Data\damethods\EASY ACESS2.ANM\efc.xml One or more scans have failed IRM. Review the data file for details. 025, 442.007 457.981 419.0 1.8e6 1.7e6 1.6e6 1.5e6 1.4e6 1.3e6 1.2e6 1.1e6 6.0e5 5.0e5 4.0e5 3.0e5 2.0e5 1.0e5 0.0 Merged XIC, Period# : 1 Experiment# : 1 0.264 to 0.458 min from 231008-ESI_POS2-03571.wiff Agile 420.0240 1.9e5 1.8e5 1.7e5 1.6e5 1.4e5 1.3e5 1.2e5 1.2e5 1.1e5 1.0e5 9.0e4 8.0e4 8.0e4 7.0e4 6.0e4 5.0e4 4.0e4 3.0e4 2.0e4 42 1.004 500 660 1.9e5 counts Max +TOF MS: 0.264 to 0.458 -ESI POS2-03571.wiff Agilent 1000 420.0240 1.9e5 1.8e5 1.7e5 1.5e5 1.4e5 1.4e5 1.2e5 1.2e5 1.0e5 9.0e4 8.0e4 7.0e4 6.0e4 5.0e4 3.0e4 442.0067 0096 422.02 457.9802 2.0e4 . 424 420 428 430 432 434 436 438 Mass Peak RT (min) Peak area Description Formula C16H13N5O3S3 Compound name 0.32 1.13325 E7 --419.01805
 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
 131203.64
 442.00727

 20152.54
 457.98121
 -1.36 [M+Na]+ 442.00667 -0.60229 457,98015 -2.30

-1.05493

Thursday, October 23, 2008

[M+K]+

13:15:32 PM

Area % Report

Page 1 of 1

 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

 Method:
 C:\EZChrom Elite\Enterprise\Projects\HTS Chemistry\Method\Yiyu\analytical\CH3CN 40 H2O

 60 TFA 0.1 1ml 40 min postrun.met

 Acquired:
 8/20/2010 12:19:49 PM

 Printed:
 11/2/2010 1:24:34 PM



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 ¹H NMR



S10

Mass List Report

Page 1 of 1

Sample#: YG1-159-2 Sample Location: P1-A-06 Sample Id: YG1-159-2 Operator: EasyAccess Data File Name: D:PE Sciex Data\Projects\chemist\10-08\Data\221008-NEG100_1000-03553.wiff Acq Time: October 22 2008, 05:11:12 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.



Wednesday, October 22, 2008

17:13:18 PM

Empirical Formula Confirmation Report

Sample Name: YG1-159-2 Sample Location: P1-C-01 Sample Id: YG1-159-2 Operator: EasyAccess
Data File Name: D:YPE Sciex Data/Projects/chemist/10-08/Data/231008-NEG100_1000-03566.wiff Acq Time: October 23 2008, 12:50:10
PM
Method: D:\TOF_Data/damethods/EASY ACESS2.ANM/lefc.xml

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



Merged XIC, Period#:1 Experiment#:1



Thursday, October 23, 2008

12:52:22 PM

Area % Report

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 Data File:
 C:\EZChrom Elite\Enterprise\Projects\HTS Chemistry\Data\Yiyu\proteasome\YG1-159-2 CH3CN

 40 H2O 60 TFA 0.1 Iml 40 min.met 8-16-2010 6-50-06 PM

 Method:
 C:\EZChrom Elite\Enterprise\Projects\HTS Chemistry\Method\Yiyu\analytical\CH3CN 40 H2O

 60 TFA 0.1 Iml 40 min postrun.met

 Acquired:
 8/16/2010 6:50:50 PM

 Printed:
 11/2/2010 1:23:20 PM

 300
 Retention Time

 200
 200



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



¹³C NMR



Mass List Report Page 1 of 1 Sample#: YG4-040-2 Sample Location: P1-B-03 Sample Id: YG4-040-2 Operator: EasyAccess Data File Name: D:YE Sciex Data/Projects/chemist/08-10/Data/060810-ESI_POS2-15228.wiff Acq Time: August 06 2010, 04:42:36 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. 060810-ESI_POS2-15228.wif 1.306 1.2e6 1.106 1.006 9.005 8.005 173.07 173.07 7.0e5 173.07 173.07 6.0e5 5.0e5 4.0e5 3.0e5 2.0e5 1.0e5 0.0 1.2 0.3 0.4 0.9 1.0 Time 0.5 0.6 0.7 0.2 0.8 Experiment# : Average of all experiments Period# : Average of all periods Peak# Experiment# Time Area Most Abundant Masses/scan 0.29 2.71028 E6 406.00700 1 1 Max. 2.1e4 count 501 min from 060810-ESI POS2-15228.wiff Agiler 2.1e4 2.0e4 406.0070 1.8e4 1.604 1.4e4 1.204 1.0e4 8000.0 6000.0 4000.0 2000.0 LL Lul 0.0 1000 m/z amu ------1200 1600 800 1400 Retention Time : 0.29 min Peak#:1 Experiment# : 1

Friday, August 06, 2010

16:44:44 PM

HRMS

Sample N Data File I	ame: YG4 Name: D;	1-040-2 Sample PE Sciex Data	e Location: P	1-C-01 Sample Id: emist\08-10\Data\0	YG4-040-2 Op 060810-ESI_PC	erator: EasyAc 0S2-15235.wiff	cess Acq Time: <u>Augu</u>	st 06 2010, 05	:04:36 PM
lethod: E	HATOF_D	ata\damethods	EASY ACE	SS2.ANM\etc.xml					
ne or n	nore sca	ans have fail	led IRM. R	leview the data	file for deta	ils.			
Merg	ed XIC a	t : 203.508, 405	.002, 406.010	0, 427.992, 443.966	811.012				Max. 2.8e5 cp
2.80	6	406.01							
2.4e	6 -								
2.20	6 -								
1.8e	5 -								
1.6e	6								
1.40	5-								
1.0e	5 -								
8.0e	4-								
6.0e	4-								
2.0e	4-								
0.	0 0.1	0.2 0.3	0.4 0.6	0.6 0.7 0.8	0.9 1.0 Time, mir	1.1 1.2 1.3	1.4 1.5	1.6 1.7 1.8	3 1.9 2.0
lerged)	(IC, Peri	od#:1 Exp	eriment# :	1					
-									
## +TC	F MS: 0.3	241 to 0.348 mi	n from 06081	0-ESI_POS2-15235	wiff Agilent			Ma	. 5.3e4 count
5.3e	4	4	106.0075						
5.0e	4								
4.50									
10									
4.00	4								
4.0e 3.5e 3.0e	4								
4.0e 3.5e 3.0e 2.5e	.4 .4 .17	3.0768							
4.0e 3.5e 3.0e 2.5e 2.0e	.4 .4 .4 .4 .7	3.0768							
4.0e 3.5e 3.0e 2.5e 2.0e	.4 .4 .4 .4 .4	3.0768							
4.0e 3.5e 3.0e 2.5e 2.0e 1.5e	.4 .4 .4 .4 .4 .4	3.0768							
4.0e 3.5e 3.0e 2.5e 2.0e 1.5e 1.0e 5000.	.4 .4 .4 .4 .4 .4 .4 .4 .4 .4 .4 .4 .4	3.0768							
4.0e 3.5e 3.0e 2.5e 1.5e 1.0e 5000.	.4 .4 .4 .4 .4 .4 .4 .4 .4 .4 .4 .4 .4	3.0768	400			200 12	20 1400	1600	180
4.0e 3.5e 3.0e 2.5e 1.5e 1.0e 6000 0.	4 4 17: 4 4 4 17: 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4	3.0768 	400	660 est PO32 1225	100 110 m/z amu m/z amu wif Aquie vi			1600 Maj	180 x 5 3e4 count
4.0e 3.5e 3.0e 2.5e 2.0e 1.5e 5.000 0. 0.	4 17: 4 7: 4 7: 4 7: 4 7: 4 7: 4 7: 4 7: 4	3.0768 	400 6 from 06081	605 8 	100 110 m/z amu m/z amu	12 ¹	20 1400	160D Ma	180 x. 5.3e4 count
4.0e 3.5e 2.5e 2.0e 1.5e 5.000 0. 0. 0. 0. 0. 0. 0. 0. 0. 0. 0. 0.	4 17: 4	3.0768 3.0768 4. t. j. t. j.	400 0 from 06081	600 -ESL POS2-16236	óð næmu mæmu wiff Agilent	200 12	00 1400	1600 Ma	150 4. 5.394 count
4.0e 3.5e 3.0e 2.5e 1.5e 1.0e 6000. 0 0 85.0e 5.0e 4.5e	4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4	3. pree 	400 n from 26081		00 10 m/z smu wiff Agilent	000 12	DÖ 1400	1600 Ma	180 2 5 3e4 count
4.0e 3.5e 3.0e 2.6e 2.0e 1.6e 5.000 0. 0. 5.3e 5.0e 4.5e 4.0e	4 4 17: 4	3.0768 3.0768 200 241 10.0.348 mi	400 n from 06081	600 0 600 0 0.ESLPOS2-16235	000 10 m/z anu s wiff Agile nt	,00 12	30 1400	1600 Mai	−10α v: 5.3e4 count
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4.0a 3.6a 2.6a 2.0a 1.6a 5.0a 6.0a 4.6a 4.6a 3.6a 3.6a 2.6a 2.6a 2.6a		3. 0768 4. L. J. L. L. J. J. 200 241 10 0. 348 mi .0076	480 0 from 06081	600 0 600 0 6-E91_POS2-16236	ob r/z anu wif Agilent	12	5 <u>0</u> 1400	1600 Ma	180 6.334 count
4.0a 3.6e 2.0a 1.0a 6000 0 0 1.0a 5.0a 4.0a 3.6e 3.6e 3.6e 2.6a 2.6a 1.6e		3. p768 	400 100 rom 06081	600 s	ob 10 m/z amu .wiff Agilant	200 12	00 1400	1600 Mar	10C
4.00 3.60 2.00 1.60 1.60 6.00 0 4.60 6.00 4.60 4.60 2.60 2.60 2.60 1.60 1.60 0 0 0		3. p768	400 10 from 08081	600 s	oo in the second	12	00 1400	1600 Mer	180 2 5 3#4 court
4.00 3.66 2.66 2.00 1.66 1.00 0 0 0 1.66 6.00 0 0 0 0 0 0 0 0 0 0 0 0		3.0768 200 201 to 0.348 mi .0076	460 500	600 e	000 102 smu m/z smu s.wiff Agilent	500 12 0 0 640 660	00 1400 680 760 720	1600 Mai 740 760	100
4.00 3.66 2.60 2.60 1.65 1.65 6.00 0.0 0.0 0.0 0.0 0.0 0.0 0.	A 4 177	3.0768 200 241 10 0.348 mi .0076	400 400 400 400 400 500	620 640 660 (top 10 m/z amu s wiff Agilent	000 12 0 0 640 660	90 1400 880 700 720	1600 Me 740 760	180 x 6 3#4 count 760 800
4.00 3.66 3.00 2.50 1.55 1.00 6.000 0. 4.56 4.56 4.56 3.06 4.56 4.56 3.06 4.56 4.56 4.56 4.56 4.56 4.56 5.00 0. 0. 0. 0. 0. 0. 0. 0. 0.	4 177	200 241 to 0.346 mi 20075	460 660	600 0000000000000000000000000000000000	são edo e20 m/z amu	0 ••• e40 e60	90 1400 880 700 720	1600 M≊ 740 780	180 • 6.3=4 count • 780 800
4.0× 3.0× 2.6× 2.0× 1.5× 1.0× 5.0× 0.0× 0.0× 0.0× 0.0× 0.0× 0.0× 0	4 4 177	3.0768 200 201 10 0.348 ml .0075 .0075	460 500 anne Mas 405.00	600 signal signa		500 12 0 640 660 1 Description 6	50 1400 660 700 720	1600 Mai	180 2 6 3 4 4 court
4.00 3.60 2.60 2.60 1.50 1.50 0 0 0 0 0 0 0 0 0 0 0 0 0	4 4 177 4	3.0768 200 201 200 201 to 0.348 mt .0076 .0076	460 500 460 500 ame Mas 405.00	<u>s Peak RT (mi</u>	550 edo e20 m/z amu self Agilent selo edo e21 m/z amu n) Peak area 28 1.05080 E	0 . e40 e60 Description 6	00 1400 680 700 720	1600 Mar 740 780	180 2 6 3 # 4 count
4.00 3.66 3.66 2.66 2.66 1.66 1.66 1.66 5.00 0 0 0 0 0 0 0 0 0 0 0 0	4 4 177 4 17	3. 0768	460 500 me Mass 405.00	620 640 660 (s Peak RT (mi 240 0.	iob in/2 and iswif Agilent in/2 and iswif Agilent in/2 and in/2 and in/2 and	2 600 12 6 600 660 6 Error (ppm)	eao 700 720	1600 Mar 740 780	180 2 6 3 4 6 our
4.00 3.60 3.00 2.60 2.00 1.50 1.50 0.00	4 4 177	200 200 201 201 201 201 201 201	460 600 from 06081 460 600 ame Mas 405.00185 405.00185	620 640 560 f S Peak RT (mi 240 0. Measured Mass 404.99629 dots 0754	iob n/z smu m/z smu n/z smu sao edo edo n) Peak area n/z smu zs 1.05080 E E Error (mDa) -5.59865 -2.54470	bo 12 a Description 6 Error (ppm) -13.73 -5.28	00 1400 080 700 720	740 7 ⁸ 0	100 x 6 344 court

Friday, August 06, 2010

17:06:50 PM

Area % Report

 Data File:
 C:\EZChrom Elite\Enterprise\Projects\HTS Chemistry\Data\Yiyu\proteasome\YG4-040-2 CH3CN

 35 H2O 65 TFA 0.1 Iml 30 min.met 8-9-2010 3-16-19 PM

 Method:
 C:\EZChrom Elite\Enterprise\Projects\HTS Chemistry\Method\untitled.met

 Assuring
 %00010 2:17:02 PM

 Acquired: Printed: 8/9/2010 3:17:03 PM 10/29/2010 4:48:12 PM 1: 254 nm. 4 r Retention Time 150 150 100 100 Ě N 50 20.413 50 9.547 0 0 0 5 10 15 Minutes 20 25 30 1: 254 nm, 4 nm Results Retention Time Area Area % Height Height % 98.90 1.10 9.547 2841826 96.42 150553 20.413 105595 3.58 1678

Totals				
	2947421	100.00	152231	100.00

Page 1 of 1



S18



Tuesday, May 24, 2011

13:47:45 PM

Empirical Formula Confirmation Report

Page 1 of 1

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.





 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

Page 1 of 1 Area % Report Data File: C:\EZChrom Elite\Enterprise\Projects\HTS Chemistry\Data\Yiyu\proteasome\YG4-112-1 CH3CN 50 H2O 50 TFA 0.1 Iml 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 Retention Time 100 100 ž ž 50 50 3.360 14.520 0 0 0 5 10 15 20 Minutes 25 30 35 40 1: 254 nm, 4 nm Results Retention Time Area % Area Height Height % 3.360 14.520 29592 3429846 0.86 2.61 3388 99.14 126423 97.39 Totals 3459438 100.00 129811 100.00

S21



S22

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Mass List Report
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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 9.2e6 cp YG4-112-21-221010-ESI 083 494 8.5e6 8.5e6 7.5e6 7.5e6 6.5e6 6.0e6 5.5e6 4.5e6 4.5e6 3.5e6 3.5e6 3.5e6 2.5e6 1.5e6 1.5e6 5.0e5 0.0 0.2 0.3 0.4 0.6 0.0 0.7 0.8 0.0 1.8 1.9 Tim e. mi Experiment# : Average of all experiments Period# : Average of all periods Most Abundant Masses/scan Peak# Experiment# Time Агеа 1 0.31 3.84290 E7 102.12778 1 Max. 2.4e5 cou in from YG4-112-21-2210 269 to 0.653 02 1278 2.405 2.265 2.005 1.8e5 1.6e5 1.4e5 494.0737 1.205

800

Retention Time : 0.31 min

1000

m/z a

1200

1400

1600

Friday, October 22, 2010

1.0e5 8.0e4 6.0e4 4.0e4 2.0e4

Peak#:1

Experiment# : 1

12:35:25 PM



Friday, October 22, 2010

26985.03 516.05708

7303.21 987.14299

516.05637

987.13984

-0.71237

-3.15505

-1.38

-3.20

[M+H]+

[M+Na]+ [2M+H]+

13:58:41 PM

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
 C:\EZChrom Elite\Enterprise\Projects\HTS Chemistry\Method\untitled.met

 Method:
 C:\EZChrom Elite\Enterprise\Projects\HTS Chemistry\Data\Yiyu\proteasome\YG4-112-2 CH3CN

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



S26

LC-MS

Sample#: YG4-040-5 Sample Location: P1-B-07. Sample Id: YG4-040-5. Operator: EasyAccess Data File Name: D:VPE Sciex Data/Projects/chemist/08-10/Data/060810-ESI_POS2-15232.wiff. Acg Time: August 05:2010, 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

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

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







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+H1+	359946.57	492.07947	492.07620	-3.27546	-6.66	
[M+Nal+	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\prote asome\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 3000 3000 Retention Time 2000 2000 È È 1000 1000 21.253 16.000 3.360 5.560 160 0 0 0 5 10 15 20 25 30 Minutes

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 ¹H NMR



¹³C NMR



Mass List Report

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Sample#: YG4-019- Sample Location: P1-E-03 Sample Id: YG4-019-2 Operator: EasyAccess Data File Name: D:\PE Sciex Data\Projects\chemist\07-10\Data180710-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.



Friday, July 16, 2010

16:40:34 PM



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

Species	Abundance (counts)	lon 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

Area % Report

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Retention 1 mile	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



S34

Mass List Report

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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



Friday, October 22, 2010

12:38:33 PM

Empirical Formula Confirmation Report

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Sample Name: YG4-112-3 Sample Location: P1-D-03 Sample Id: YG4-112-3 Operator: EasyAccess Data File Name: D:VPE Sciex Data\Projects\Yiyu Ge110-10\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







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

Species	Abundance (counts)	lon 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



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



Figure 1: A, chemical shift (in CDCl₃, 400 MHz) of 3-H in compound 8a (upper) and 9a (lower); B, chemical shift (in CDCl₃, 400 MHz) of 3-H in compound 12 (upper) and 13a (lower). These ¹H NMRs indicate coupling intermediate 8a and 12 with thiophenesulfonamide proceeded regioselectively at the 4-carbonyl of the naphthoquinone ring.