## **Supporting Information**

## Org. Commun. 10:4 (2017) 259-272

# Synthesis and biological assessment of novel acylhydrazone derivatives of 2-methyl-1,4naphthoquinone

Kamal Bouhadir,<sup>1†</sup> Hala Atallah,<sup>1,2†</sup> Rana Mezher,<sup>1,2</sup> Maamoun Fatfat,<sup>3</sup> Hala Gali-Muhtasib<sup>3</sup> and Jomana Elaridi<sup>\*2</sup>

<sup>1</sup> Department of Chemistry, Faculty of Arts and Sciences, American University of Beirut, Beirut, 11-0236, Lebanon

<sup>2</sup> Department of Natural Sciences, School of Arts and Sciences, Lebanese American University, Beirut, 1102-2801, Lebanon

<sup>3</sup> Department of Biology, and Department of Anatomy, Cell Biology and Physiological Sciences, American University of Beirut, Beirut, 11-0236, Lebanon

<sup>†</sup> The authors equally contributed to this work

Table of contents	Page
Figure S1 IR spectrum of 6a	4
Figure S2. <sup>13</sup> H NMR spectrum of <b>6a</b>	5
Figure S3. <sup>13</sup> C NMR spectrum of <b>6a</b>	6
Figure S4. DEPT NMR spectrum of <b>6a</b>	7
Figure S5. HRMS spectrum of <b>6a</b>	8
Figure S6 IR spectrum of compound <b>6b</b>	9
Figure S7. <sup>13</sup> H NMR spectrum of <b>6b</b>	10
Figure S8. <sup>13</sup> C NMR spectrum of <b>6b</b>	11

Figure S9. DEPT NMR spectrum of <b>6b</b>	12
Figure S10. LC-MS spectrum of <b>6b</b>	13
Figure S11. HRMS spectrum of <b>6b</b>	14
Figure S12. IR spectrum of <b>6c</b>	15
Figure S13. <sup>13</sup> H NMR spectrum of <b>6c</b>	16
Figure S14. <sup>13</sup> C NMR spectrum of <b>6c</b>	17
Figure S15. HRMS spectrum of 6c	18
Figure S16. IR spectrum of 6d	19
Figure S17. <sup>13</sup> H NMR spectrum of <b>6d</b>	20
Figure S18. <sup>13</sup> C NMR spectrum of <b>6d</b>	21
Figure S19. LC-MS spectrum of 6d	22
Figure S20. HRMS spectrum of 6d	23
Figure S21. IR spectrum of <b>6e</b>	24
Figure S22. <sup>13</sup> H NMR spectrum of <b>6e</b>	25
Figure S23. <sup>13</sup> C NMR spectrum of <b>6e</b>	26
Figure S24. DEPT NMR spectrum of <b>6e</b>	27
Figure S25. HRMS spectrum of 6e	28
Figure S26. IR spectrum of <b>6f</b>	29
Figure S27. <sup>13</sup> H NMR spectrum of <b>6f</b>	30
Figure S28. <sup>13</sup> C NMR spectrum of <b>6f</b>	31
Figure S29. HMQC 2D-NMR spectrum of 6f	32
Figure S30. LC-MS spectrum of 6f	33
Figure S31. HRMS spectrum of <b>6f</b>	34
Figure S32. Elemental analysis of <b>6f</b>	35
Figure S33. IR spectrum of <b>6g</b>	36
Figure S34. <sup>13</sup> H NMR spectrum of <b>6g</b>	37
Figure S35. <sup>13</sup> C NMR spectrum of <b>6g</b>	38
Figure S36. NOESY 2D-NMR spectrum of <b>6g</b>	39
Figure S37. LC-MS spectrum of <b>6g</b>	40
Figure S38. HRMS spectrum of <b>6g</b>	41
Figure S39. Elemental analysis of <b>6g</b>	42
Figure S40. IR spectrum of <b>6h</b>	43
Figure S41. <sup>13</sup> H NMR spectrum of <b>6h</b>	44
Figure S42. DEPT NMR spectrum of <b>6h</b>	45
Figure S43. COSY NMR spectrum of 6h	46
Figure S44. HRMS spectrum of <b>6h</b>	47

Figure S45. IR spectrum of <b>6i</b>	48
Figure S46. <sup>13</sup> H NMR spectrum of <b>6i</b>	49
Figure S47. <sup>13</sup> C NMR spectrum of <b>6i</b>	50
Figure S48. HRMS spectrum of 6i	51
Figure S49. IR spectrum of <b>6</b> j	52
Figure S50. <sup>13</sup> H NMR spectrum of <b>6</b> j	53
Figure S51. DEPT NMR spectrum of 6j	54
Figure S52. NOESY NMR spectrum of 6j	55
Figure S53. HRMS spectrum of 6j	56
Figure S54. Elemental analysis of <b>6</b> j	57
Figure S55. IR spectrum of <b>6k</b>	58
Figure S56. <sup>13</sup> H NMR spectrum of <b>6k</b>	59
Figure S57. <sup>13</sup> C NMR spectrum of <b>6k</b>	60
Figure S58. DEPT NMR spectrum of <b>6k</b>	61
Figure S59. LC-MS spectrum of <b>6k</b>	62
Figure S60. HRMS spectrum of <b>6k</b>	63
Figure S61. IR spectrum of <b>6</b>	64
Figure S62. <sup>13</sup> H NMR spectrum of <b>6</b> I	65
Figure S63. <sup>13</sup> C NMR spectrum of <b>6</b> l	66
Figure S64. DEPT NMR spectrum of 61	67
Figure S65. HRMS spectrum of 6l	68
Figure S66. IR spectrum of <b>6m</b>	69
Figure S67. <sup>13</sup> H NMR spectrum of <b>6m</b>	70
Figure S68. <sup>13</sup> C NMR spectrum of <b>6m</b>	71
Figure S69. DEPT NMR spectrum of 6m	72
Figure S70. LC-MS spectrum of 6m	73
Figure S71. HRMS spectrum of 6m	74



Figure S1. IR spectrum of compound 6a



Figure S2. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz) spectrum of compound 6a



Figure S3. <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) spectrum of compound 6a



Figure S4. DEPT NMR (CDCl<sub>3</sub>, 100 MHz) spectrum of compound 6a



Elemental Compositio	n Report				Page 1
Single Mass Analysis Tolerance = 1.0 mDa / 1 Element prediction: Off Number of isotope peaks	DBE: min = -1.5, used for I-FIT = 3	max = 50.0			
Monoisotopic Mass, Even El 467 formula(e) evaluated wit Elements Used: C: 0-100 H: 0-100 N: 0 SYNAPT G2-84UEB205 Y-PC17020316 3 (0.141) Cm (3	ectron lons h 2 results within li h-20 O: 0-20 (4)	mits (up to 50 best is	otopic matches for e W264	ach mass)	03-Feb-2017 1: TOF MS ES+ 7.34=008
198 248.11 253.13 250.0	257.28 287.27 <sup>27</sup> 280.0 270	0 280.0	290.12 293.13 296.1 290.0 SC	0.300.10 313.27 0.0 310.0	320.19 325.08 331.28 320.0 330.0
Minimum: Maximum:	1.0 1.0	-1.5			
Mass Calc. Mass	mDa PPM	DRE 1-FIT	Norm Conf(%)	Formula	
271.1445 271.1447 271.1452	-0.2 -0.7 -0.7 -2.6	8.5 2414.2 1.5 2430.0	0.000 100.00 15.760 0.00	C16 H19 N2 O2 C H15 N14 O3	

Figure S5. HRMS spectrum of compound 6a



Figure S6. IR spectrum of compound 6b

RM176/CDC13



Figure S7. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz) spectrum of compound 6b



Figure S8. <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) spectrum of compound 6b

11



Figure S9. DEPT NMR (CDCl<sub>3</sub>, 100 MHz) spectrum of compound 6b



Figure S10. LC-MS spectrum of compound 6b



Elemental Composition Report

Page 1

Single Mass Analysis Tolerance = 1.0 mDa / DBE: min = -1.5, max = 50.0 Element prediction: Off Number of isotope peaks used for I-FIT = 3 Monoisotopic Mass, Even Electron Ions 664 formula(e) evaluated with 2 results within limits (up to 50 best isotopic matches for each mass) Elements Used: C: 0-100 H: 0-100 N: 0-20 O: 0-20 SYNAPT 02-SHUEB205 03-Feb-2017 HA20 Y-PC17020314 3 (0.141) Cm (2.4) 1: TOF MS ES+ 3.79e+008 100 282.28 285.28 291.11 295.18 300.29 311.18 315.20 327.17 331.28 335.17 341.31 349.20 353.22 359.52 389.25 372.28 280.0 290.0 300.0 310.0 320.0 390.0 340.0 350.0 380.0 370.0 Minimum: -1.5 1.0 1.0 50.0 Maximum: Calc. Mass mDa FFM DBR i-FIT Norm Conf(%) Formula Мани 313.1915 313.1916 -0.1 -0.3 0.5 2014.2 0.000 100.00 C19 H25 N2 02 313.1921 -0.6 -1.9 1.5 2020.7 14.547 0.00 C4 H21 N14 03

Figure S11. HRMS spectrum of compound 6b



Figure S12. IR spectrum of compound 6c



Figure S13. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz) spectrum of compound 6c



Figure S14. <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) spectrum of compound 6c



#### Elemental Composition Report

Page 1

Single Mass Analysis Tolerance = 1.0 mDa / DBE: min = -1.5, max = 50.0 Element prediction: Off Number of isotope peaks used for I-FIT = 3

Monoisotopic Mass, Even Electron Ions 883 formula(e) evaluated with 2 results within limits (up to 50 best isotopic matches for each mass) Elements Used: C: 0-100 H: 0-100 N: 0-20 O: 0-20 BYNAPT 02-BHUEB205 HADS 03-Feb-2017 Y-PC17020315 3 (0.141) Cm (3:4) 1: TOF MS ES+ 6.25e+008 102 313.27 325.19 335.21 341.31 351.21 355.24 361.33 375.20 380.16 383.20 389.33 307.25 406.13 415.21 147.22 315 320 325 330 325 340 345 350 355 360 365 370 375 380 385 390 395 400 455 410 415 420 Minimum: -1.5 1.0 1.0 50.0 Maximum: Calc. Mass mDa FFM DHE i-FIT Norm Conf(%) Formula Мани 353.2230 353.2229 0.1 0.3 9.5 2033.0 0.022 97.78 C22 H29 N2 02 353.2224 -0.4 -1.1 2.5 2036.8 3.009 2.22 C7 H25 N14 03

Figure S15. HRMS spectrum of compound 6b



Figure S16. IR spectrum of compound 6d



Figure S17. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz) spectrum of compound 6d



Figure S18. <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) spectrum of compound 6d

Display Report - All Windows All Analyses



Figure S19. LC-MS spectrum of compound 6d



Elemental Composition	on Report		Page 1
Single Mass Analysis Tolerance = 1.0 mDa / 1 Element prediction: Off Number of isotope peaks	DBE: min = -1.5, max = 50 used for I-FIT = 3	.0	
Monoisotopic Mass, Even El 969 formula(e) evaluated wit Elements Used: C: 0-100 H: 0-100 N: 0	ectron lons th 2 results within limits (up to 0-20 O: 0-20	50 best isotopic matches for ea	ach mass)
SYNAPT 02-SIUE8205 Y-PC17020312 3 (0.141) Cm (3	(4)	HA14	03-Feb-2017 1: TOF MS ES+ 9.22#+005
108 355.07 359.32.38 355.0 380.0	0.32 368.16368.40 370.2 365.0 370.0	28 378.34 381.30,382.38 375.0 380.0	385.25 388.23 391.24 392.24 396.21 397.29 400.16 mlz 385.0 390.0 395.0 400.0
Minimum: Muximum:	-1.5 1.0 1.0 50.0		
Mass Calc. Mass	mDa PPM DBR	i-WIT Norm Conf(%)	Formula
369.2540 369.2542 369.2547	-0.2 -0.5 0.5 -0.7 -1.9 1.5	1499.3 0.000 100.00 1514.1 14.875 0.00	C23 H33 N2 O2 C8 H29 N14 O3

Figure S20. HRMS spectrum of compound 6d



Figure S21. IR spectrum of compound 6e



Figure S22. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz) spectrum of compound 6e



Figure S23. <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) spectrum of compound 6e



Figure S24. DEPT NMR (CDCl<sub>3</sub>, 100 MHz) spectrum of compound 6e



Elemental Composition Report

Page 1

Single Mass Analysis Tolerance = 1.0 mDa / DBE: min = -1.5, max = 50.0 Element prediction: Off Number of isotope peaks used for I-FIT = 3 Monoisotopic Mass, Even Electron Ions 1130 formula(e) evaluated with 2 results within limits (up to 50 best isotopic matches for each mass) Elements Used: C: 0-100 H: 0-100 N: 0-20 O: 0-20 03-Feb-2017 1: TOF MS ES+ 2.44e+005 SYNAPT G2-S#UEB205 Y-PC17020311 3 (0.141) Cm (3) HA15 102 371.10 381.30 388.28 397 29 380 390 400 425.32 497.21 503.11 500 mlz 437.19 440 415.21 449.21 459.24481.24 478.23 487.18 410 430 470 450 460 480 490 Minimum: Maximum: -1.5 Мани Calc. Mass mDa PPM. DBE i-FIT Norm Conf(%) Formula 297.2855 -0.3 -0.8 8.5 1021.8 0.000 100.00 C25 H37 N2 02 297.2860 -0.8 -2.0 1.5 1036.6 14.744 0.00 C10 H33 N14 03 397.2852 397.2855

Figure S25. HRMS spectrum of compound 6e



Figure S26. IR spectrum of compound 6f



Figure S27. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz) spectrum of compound 6f



Figure S28. <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) spectrum of compound 6f



Figure S29. HMQC 2D-NMR spectrum of compound 6f



### Display Report - All Windows All Analyses

Figure S30. LC-MS spectrum of compound 6f



Figure S31. HRMS spectrum of compound 6f

71 HA17	76.521	9.791	6.193
72 HA17	76.934	9.761	6.207
Valeur moyenne	76.728	9.776	6.200
Déviation (abs.)	0.292	0.021	0.010
Delta [%]	0.414	0.029	0.014

Figure S32. Elemental analysis of compound 6f



Figure S33. IR spectrum of compound 6g


Figure S34. <sup>1</sup>H NMR (DMSO, 500 MHz) spectrum of compound 6g



Figure S35. <sup>13</sup>C NMR (DMSO, 100 MHz) spectrum of compound 6g



Figure S36. NOESY 2D-NMR spectrum of compound 6g



Figure S37. LC-MS spectrum of compound 6g



Elemental	I Compositio	n Repo	rt							Page 1
Single Ma Tolerance - Element pro Number of	ediction: Off	BE: min	i = -1.5, r I-FIT = 3	nax = 50	1.0					
Monoisotopio 561 formulai Elements Us C: 0-100	c Mass, Even Ele le) evaluated with led: H: 0-100 N: 0	ctron ion 3 results -20 O:	s s within lin 0-20	nits (up to	50 best iso	topic mat	ches for ea	ch mass)		
SYNAPT 02- Y-PC1702031	S#UEB205 0.4 (0.175) Cm (3-	6)			۲	A18				03-Feb-2017 1: TOF MS ES+ 3.53e+008
198 <u>27</u> 270.0	3.10 <sup>275.11</sup> 282. 280.0	03 285.2	290.0	93.12 3	02.09 307.1	10.09 3 310.0	13.10 318	107_319.58 320.0	329.07 330.0	338.34 341.30 343.31 340.0 m/z
Minimum: Maximum:		1.0	1.0	-1.5 50.0						
Мани	Culc. Mass	nDu	228	DRE	i-FIT	Norm	Conf(≹)	Formula		
291.1132	291.1134 291.1139 291.1125	-0.2 -0.7 0.7	-0.7 -2.4 2.4	12.5 5.5 0.5	2039.8 2051.4 2052.3	0.000 11.518 12.426	100.00 0.00 0.00	C10 H15 N2 C3 H11 N14 C2 H15 N10	02 03 07	

Figure S38. HRMS spectrum of compound 6g

	N° Nom	C [%]	H [%]	N [%]
/	65 HA18	74.266	4.879	9.707
	66 HA18	74.421	4.876	9.661
	Valeur moyenne	74.343	4.878	9.684
	Déviation (abs.)	0.110	0.002	0.033
	Delta [%]	0.155	0.003	0.046
				the second se

Figure S39. Elemental analysis of compound 6g



Figure S40. IR spectrum of compound 6h



Figure S41. <sup>1</sup>H NMR (DMSO, 500 MHz) spectrum of compound 6h



Figure S42. DEPT NMR (DMSO, 100 MHz) spectrum of compound 6h



Figure S43. COSY 2D-NMR spectrum of compound 6h



# Elemental Composition Report

Page 1

### Single Mass Analysis Tolerance = 1.0 mDa / DBE: min = -1.5, max = 50.0

Element prediction: Off Number of isotope peaks used for I-FIT = 3 Monoisotopic Mass, Even Electron Ions 810 formula(e) evaluated with 3 results within limits (up to 50 best isotopic matches for each mass) Elements Used: C: 0-100 H: 0-100 N: 0-20 O: 0-20 03-Feb-2017 1: TOF MS ES+ SYNAPT 02-SI/UEB205 HA22 Y-PC17020322 3 (0.141) Cm (2:4) 1.34e+005 300.29 313.27 327.11 300 310 320 330 339.11 15 404.12 415.21 432.24 437.19 449.21 453.17 400 410 420 430 440 450 353.27 359.32 371.15 381.30 385.15 380 370 390 380 350 Minimum: Maximum: -1.5 1.0 1.0 Calc. Mass mDa 225 DEK i-FIT Norm Conf(%) Formula Мани 339.1094 339.1093 0.1 0.3 12.5 1191.2 0.000 100.00 C17 H15 N4 O4 339.1098 -0.4 -1.2 339.1085 0.9 2.7 5.5 1207.8 16.603 0.00 C2 H11 N16 OS 0.5 1208.7 17.521 0.00 C H15 N12 O9

Figure S44. HRMS spectrum of compound 6h



Figure S45. IR spectrum of compound 6i

RM214/ DMSO



Figure S46. <sup>1</sup>H NMR (DMSO, 500 MHz) spectrum of compound 6i





Figure S47. <sup>13</sup>C NMR (DMSO, 100 MHz) spectrum of compound 6i



# Elemental Composition Report

### Page 1

1.76e+005

405

Single Mass Analysis Tolerance = 1.0 mDa / DBE: min = -1.5, max = 50.0 Element prediction: Off Number of isotope peaks used for I-FIT = 3

### Monoisotopic Mass, Even Electron Ions

884 formula(e) evaluated with 3 results within limits (up to 50 best isotopic matches for each mass) Elements Used: C: 0-100 H: 0-100 N: 0-20 O: 0-20 SYNAPT G2-SHUEB205 03-Feb-2017 1: TOF MS ES+ RM214 Y-PC17020321 3 (0.141) Cm (2.4) 313.27,315.28 527.50 510 515 520 525 550 535 540 545 55 350 355 353.12 368.40 375.11 381.30 383.38 98.38 406.14 m/z 198 398.38 370 375 400 -1.5 50.0 Minimum: Maximum: 1.0 1.0 Calc. Mass mDa PPM DBR i-FIT Norm Conf(%) Formula Мани

353.1249	353,1250 353,1255 353,1241	-0.1 -0.6 0.8	-0.3 -1.7 2.3	12.5 5.5 0.5	1123.2 1137.1 1137.9	0.000 13.978 14.793	100.00 0.00 0.00	C10 H17 N4 C3 H13 N16 C2 H17 N12	

Figure S48. HRMS spectrum of compound 6i



Figure S49. IR spectrum of compound 6j



Figure S50. <sup>1</sup>H NMR (DMSO, 500 MHz) spectrum of compound 6j



Figure S51. DEPT NMR (DMSO, 100 MHz) spectrum of compound 6j



Figure S52. NOESY 2D-NMR spectrum of compound 6j



	i Composit	on Repo	nτ									Pag
Single Ma	as Analysi											
Tolerance -	•1.0 mDa /	DBE: mir	n = -1.5, i	max = 50	.0							
Element pr	ediction: Off											
Number of	isotope peak	used for	I-FIT = 3									
Monologiani	Mass Even	lectron los										
884 formula	<ul> <li>evaluated v</li> </ul>	th 4 result	rs s within ile	nits (up to	50 best iso	topic mat	thes for ea	ch mass)				
Elements Us	ed:			Contra Contra Contra								
	U-0-100 N	0-20 O	0-20									
C: 0-100	H. 0-100 N.											
C: 0-100 SYNAPT 02-	BUEB205				FO	M221						03-Feb-3
C: 0-100 SYNAPT G2- Y-PC1702031	SIUEB205 8 3 (0.141) Cm	30			FØ	W221						03-Feb-3 1: TOF MS E
C: 0-100 SYNAPT G2- Y-PC1702031	B 3 (0.141) Cm	3)			FO	W221						03-Feb-3 1: TOF MS E 1.40er
C: D-100 SYNAPT G2- Y-PC1702031	84UEB205 8 3 (0.141) Cm	3)	Water on a		RU	353.12	359.31			386.28	393.12	03-Feb-3 1: TOF MS 8 1.40er 411.13
C: 0-100 SYNAPT G2- Y-PC1702031	297.08 <sup>300</sup>	3) 29 313.1	27315.28 3	31.28,338.	R8 34, 341, 31	W221 353.12	359.31	371.10	381.30	388.28	393.12	03-Feb-3 1: TOF MS I 1.40er 411.13 415.0
C: 0-100 SYNAPT G2- Y-PC1702031	297.08 <sup>300</sup> 290 300	3) 29 313.2 310	27 <u>315.28 3</u> 320	31.28.338 330	R0 34, 341,31 340	353.12 350	359.31 380	<u>371.10</u> 370	381.30 380	386.28 390	393.12	03-Feb-3 1: TOF MS 8 1.40er 411.13 415.0 0 410
C: 0-100 SYNAPT 02- Y-PC1702031	297.08 <sup>300</sup> 290 300	3) 29 313.2 310	27 <u>315.28 3</u> 320	31.28.338 330	R0 34, 341, 31 1111, 1111, 111 340	353.12 350	359.31 380	371.10 370	381.30 380	386.28	303.12 400	03-Feb- 1: TOF MS 0 1.40er 411.13 415.0 0 410
C: 0-100 SYNAPT G2- Y-PC1702031 102 285.28 Minimum: Munimum: Munimum:	297.08 <sup>300</sup> 290 300	3) 29 3132 310 1.0	27 <u>315 28 3</u> 320 1.0	31.28,338. 330 -1.5 50.0	R0 34, 341,31 340 340	353.12 350	359.31 380	371.10 370	381.30 380	388.28 390	393.12 111111 400	03-Feb-3 1: TOF MS 0 1.40er 411.13 415.0 0 410
C: D-100 SYNAPT G2- Y-PC1702031 10 285 28 285 285 28 285 285 28 285 285 28 285 285 28 285 285 285 285 285 285 285 285 285 285	Calc. Max	3) 29 3133 310 1.0 * mDu	27 <u>315.28</u> 3 320 1.0 77M	31.28.338. 330 -1.5 50.0 DBM	R9 34 341.31 340 i-FIT	353.12 350 Norm	359.31 380 Conf (%)	371.10 370 Pormula	381.30 380	388.28	393.12 40	03-Feb- 1: TOF MS: 1.40er 411.13 415.0 0 410
C: D-100 SYNAPT 02- Y-PC1702031 1 285.28 Minimum: Maximum: Maxe 253.1245	н. 0-100 М. S#UEB205 8 3 (0.141) Ст 290 300 Calc. Маж 253,1250	3) 29 3133 310 1.0 mDa -0.5	27315.28 3 320 1.0 77M -1.4	31.28.338. 330 -1.5 50.0 DBH 12.5	R9 34,341,31 340 i-PIT 987.0	353.12 350 Norm 0.030	359.31 380 Conf (%) 97.09	S70 Formula	381.30 380	388.28	393.12 40	03-Feb-2 1: TOF MS ( 1.40er 411.13 415 ( 0 410
C: D-100 SYNAPT G2- Y-PC1702031 10 285.28 Minimum: Muximum: Muximum: Muxem	Calc. Mas 253.1250	3) 29 313.3 310 1.0 s mDa -0.5 0.9	27315.28 3 320 1.0 77M -1.4 2.5	31.28.338. 330 -1.5 50.0 DBH 12.5 7.5	R9 34,341,31 340 i-PIT 987.0 990.6	353.12 350 Norm 0.030 3.535	359.31 380 Conf(%) 97.09 2.91	271.10 270 Pormula C18 H17 C17 H21	381.30 380 7 N4 C	386.28 390	303.12 400	03-Feb-3 1: TOF MS 8 1.40er 411.13 415 ( 0 410
C: 0-100 SYNAPT 02- Y-PC1702031 10 28528 28528 Minimum: Maximum: Maximum: Maxes 253.1245	Calc. Max 253.1250 253.1250	3) 29 313,2 310 1.0 s mDa -0.5 0.9 -1.0	27315 28 3 320 1.0 77M -1.4 2.5 -2.8	31 28 338 330 -1.5 50.0 DBH 12.5 7.5 5.5	R9 34, 341,31 340 i-PIT 987.0 990.6 1002.4	8221 353.12 350 Norm 0.030 3.535 15.340	359.31 380 Conf (%) 97.09 2.91 0.00	S71.10 S70 Pormula C18 H17 C17 H21 C17 H21 C1 H13	381.30 380 7 N4 0 N16 0	388.28	303.12 40	05-Feb- 1: TOF MSI 1.40er 411.13 415. 0 410

# Figure S53. HRMS spectrum of compound 6j



N° Nom	C [%]	H [%]	N [%]
53 RM221	61.464	4.579	15.775
54 RM221	61.021	4.690	15.618
Valeur moyenne	61.242	4.634	15.697
Déviation (abs.)	0.313	0.078	0.111
Delta [%]	0.442	0.111	0.157
		-	

Figure S54. Elemental analysis of compound 6j



Figure S55. IR spectrum of compound 6k



Figure S56. <sup>1</sup>H NMR (DMSO, 500 MHz) spectrum of compound 6k



Figure S57. <sup>13</sup>C NMR (DMSO, 100 MHz) spectrum of compound 6k



Figure S58. DEPT NMR (DMSO, 100 MHz) spectrum of compound 6k



Display Report - All Windows All Analyses

Figure S59. LC-MS spectrum of compound 6k



### Elemental Composition Report

### Page 1

03-Feb-2017 1: TOF MS ES+

4.83e+005

Single Mass Analysis Tolerance = 1.0 mDa / DBE: min = -1.5, max = 50.0 Element prediction: Off Number of isotope peaks used for I-FIT = 3

# Monoisolopic Mass, Even Electron Ions 954 formular(e) evaluated with 3 results within limits (up to 50 best isotopic matches for each mass) Elements Used: C: 0-100 H: 0-100 W: 0-200 C: 0-100 SYNAPT C2-AWLEB205 RM217 YV-PC17020301 3 (0.141) Cm (2.3) RM214 100 359.32 364.28 386.16 <sup>387,14</sup> 370.14 372.26 376.34 378.29 381.30 <sup>382,28</sup> 386 357.5 360.0 382.5 395.5 370.0 372.5 375.0 377.5 380.0 382.5 386.0

Figure S60. HRMS spectrum of compound 6k



Figure S61. IR spectrum of compound 61





Figure S62. <sup>1</sup>H NMR (DMSO, 500 MHz) spectrum of compound 6l



**Figure S63.** <sup>13</sup>C NMR (DMSO, 100 MHz) spectrum of compound **6** 



Figure S64. DEPT NMR (DMSO, 100 MHz) spectrum of compound 6l



	Compositi	on repo	n.						Рад
Single Ma	as Analysis								
Tolerance -	1.0 mDa /	DBE: min	1 = -1.5, 1	max = 50	.0				
Element pr	ediction: Off								
Number of	isotope peaks	used for	I-FIT = 3						
Monoisotopi	c Mass, Even B	lectron lor	15						
858 formular	e) evaluated w	rm 3 result	s wienin ili	nits (up to	SU DEST ISC	topic mat	ches for ea	(cn mass)	
C: 0-100	H: 0-100 N-	0-20 O	0-20						
SYNAPT CO.	SMI IFR205				R	M222			03-Eeb-2
Y-PC1702031	7 3 (0.141) Cm (	2.4)							1: TOF MS E
									6.13e+
100 100 10	208 22	310.31.919	17115-10	331.28	338.34.041	35	2.14	389 14371.10 391 30 394 5	400.13 404 5
200.12	290.32	Va12	20010-20			****	1. 224.12	300.147 301.30_304.1	in function i
0 <del></del> 0									
2	0 300	310	320	330	340	350	360	370 380 3	400
21 Zinimum:	0 300	310	320	330	340	350	360	370 380 Si	400
21 Minimum: Maximum:	0 300	310	320	-1.5 50.0	340	350	360	370 380 31	400 400
2 Minimum: Maximum:	0 300	310	320	-1.5 50.0	340	350	360	s70 580 S	80 400
о <del>трин</del> 21 Minimum: Махітит: Мажи	O SOO Calc. Mas	310 1.0 # mDu	320 1.0 FFM	330 -1.5 50.0 DBR	340 i-PIT	350 Norm	360 Conf (%)	370 380 31 Formula	400
Minimum: Maximum: Maxw 252.1409	0 300 Calc. Mass 352,1410	310 1.0 = mDu -0.1	320 1.0 PPM -0.3	330 -1.5 50.0 DBR 12.5	340 i-FIT 1437.0	350 Norm	360 Conf(%)	370 380 31 Formula	io 400
Minimum: Maximum: Mass 252.1409	0 300 Calc. Mas 352.1410 352.1415	310 1.0 # mDu -0.1 -0.6	320 1.0 FFM -0.3 -1.7	330 -1.5 50.0 DBR 12.5 5.5	340 i-FIT 1437.8 1451.9	350 Norm 0.000 14.062	360 Conf(%) 100.00 0.00	370 380 3 Formula C18 H18 N5 03 C3 H14 N17 04	io i 460
Minimum: Maximum: Mass 352.1409	Calc. Mas 352.1410 352.1401	310 1.0 s mDa -0.1 -0.6 0.8	320 1.0 PPM -0.3 -1.7 2.3	330 -1.5 50.0 DBR 12.5 5.5 0.5	340 i-FIT 1437.0 1451.9 1453.1	350 Norm 0.000 14.062 15.243	360 Conf(%) 100.00 0.00 0.00	370 380 31 Pormula C18 H18 N5 C0 C3 H14 N17 C4 C2 H18 N13 C6	io i 460

Figure S65. HRMS spectrum of compound 61



Figure S66. IR spectrum of compound 6m

RM235/ DMSO



Figure S67. <sup>1</sup>H NMR (DMSO, 500 MHz) spectrum of compound 6m



Figure S68. <sup>13</sup>C NMR (DMSO, 100 MHz) spectrum of compound 6m



Figure S69. DEPT NMR (DMSO, 100 MHz) spectrum of compound 6m
Display Report - All Windows All Analyses



Figure S70. LC-MS spectrum of compound 6m



Elemental	I Compositio	n Repo	rt						Page 1
Single Ma Tolerance – Element pre Number of I Monoisotopic 993 formula(i Elements Us	88 Analysis 1.0 mDa / D ediction: Off sotope peaks ( c Mass, Even Ele e) evaluated with ed:	DBE: min used for actron lon h 3 result	n = -1.5, n I-FIT = 3 Is swithin lin	nax = 50 hts (up to	.0 50 best is	otopic mat	thes for ea	ch mass)	
C: 0-100 H SYNAPT G2-5 Y-PC1702032	0 H: 0-100 N: 0-20 O: 0-20 7 (22-44/EE205 FM235 200320 3 (0.141) Cm (3.4)								03-Feb-2017 1: TOF MS ES+ 8.17e+004
108 285.28 280 28	300.29 313 10 300 310	27 11 11 11 11 11 11 11 11 11 11 11 11 11	331.28 330 340	in mining 350	359.32 380 37		30 397.27 390 40	415.21 432.23 437.19 0 410 420 430 440	453.17 459.24482.19 0 450 480
Minimum: Maximum:		1.0	1.0	-1.5 50.0					
Мани	Calc. Mass	mDa.	228	DBX	i-FIT	Norm	Conf(≹)	Vormula	
376.1523	376.1522 376.1527 376.1514	0.1 -0.4 0.9	0.3 -1.1 2.4	14.5 7.5 2.5	919.4 936.6 936.9	0.000 17.273 17.526	100.00 0.00 0.00	C19 H18 N7 O2 C4 H14 N19 O3 C3 H18 N15 O7	

Figure S71. HRMS spectrum of compound 6m