Supporting Information

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

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

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[†] The authors equally contributed to this work

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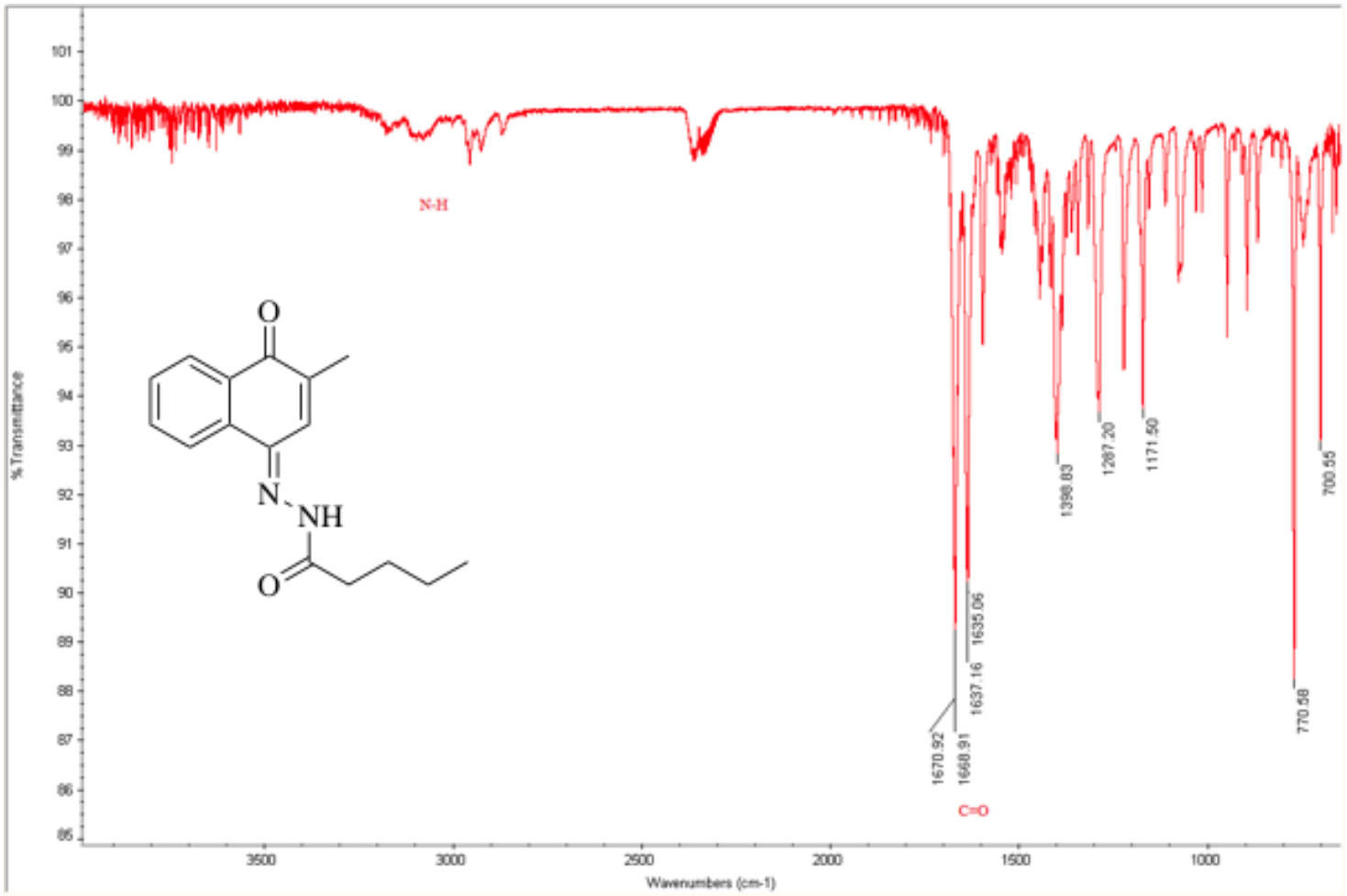


Figure S1. IR spectrum of compound **6a**

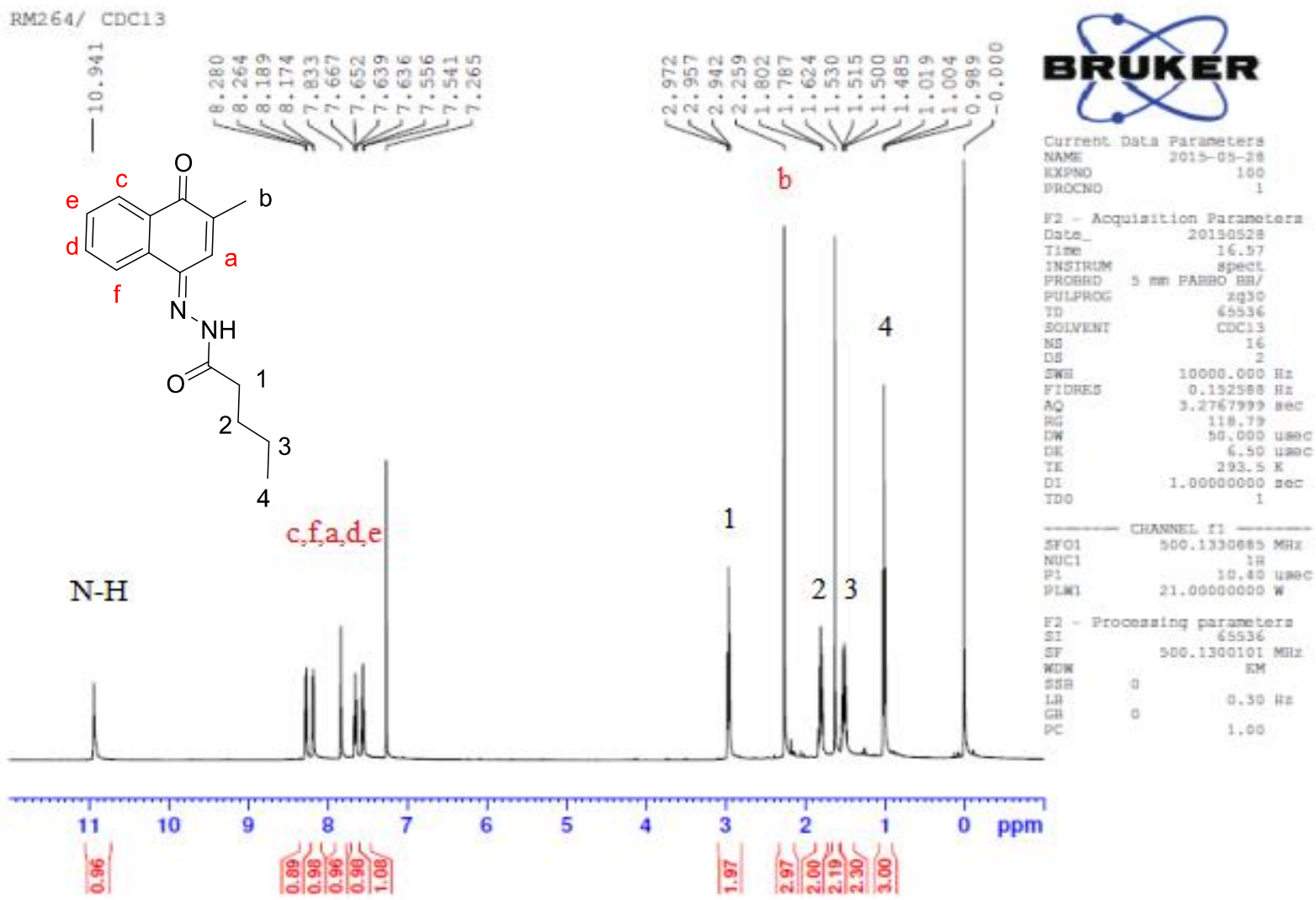


Figure S2. ^{1}H NMR ($CDCl_3$, 500 MHz) spectrum of compound **6a**

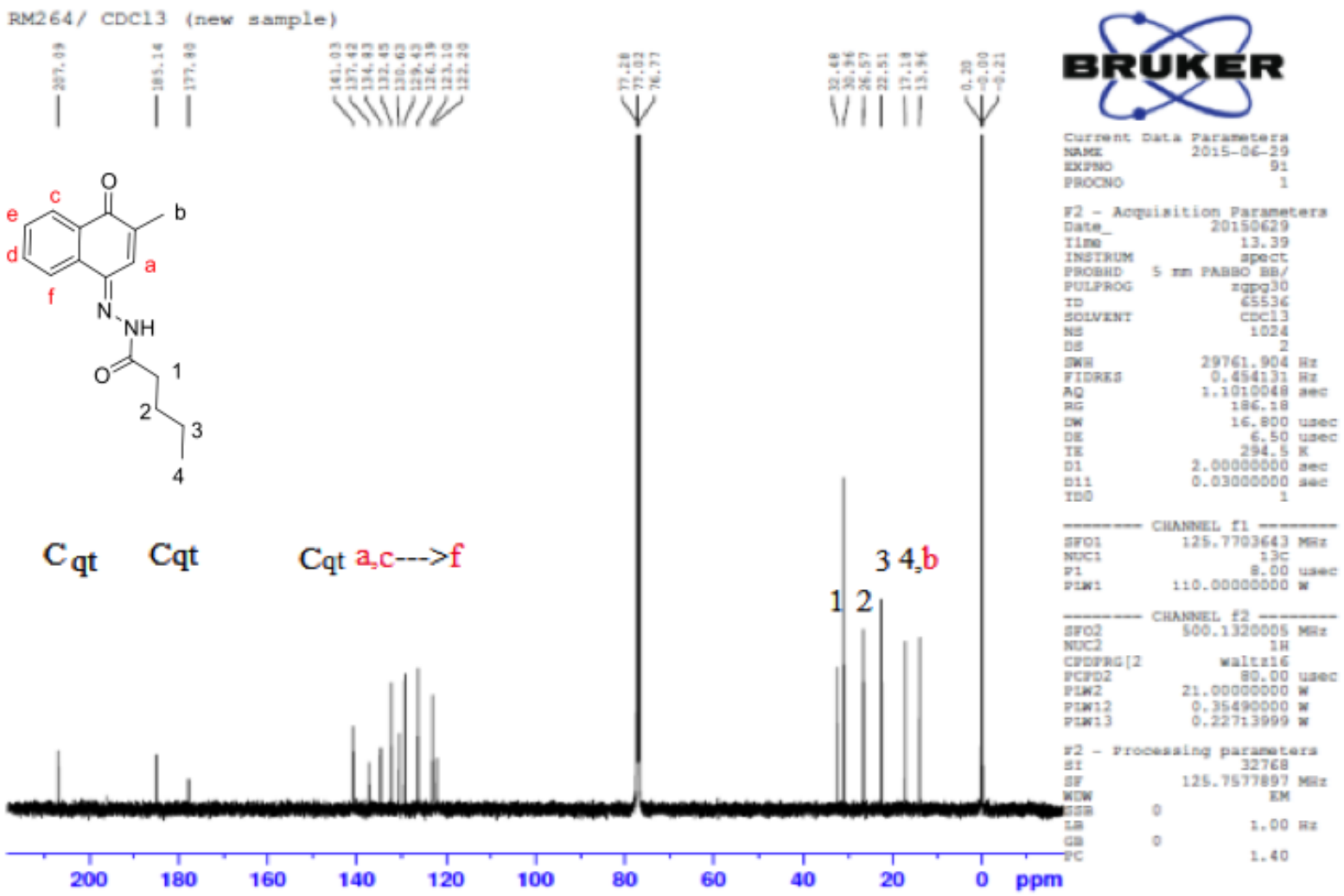


Figure S3. ^{13}C NMR ($CDCl_3$, 100 MHz) spectrum of compound **6a**

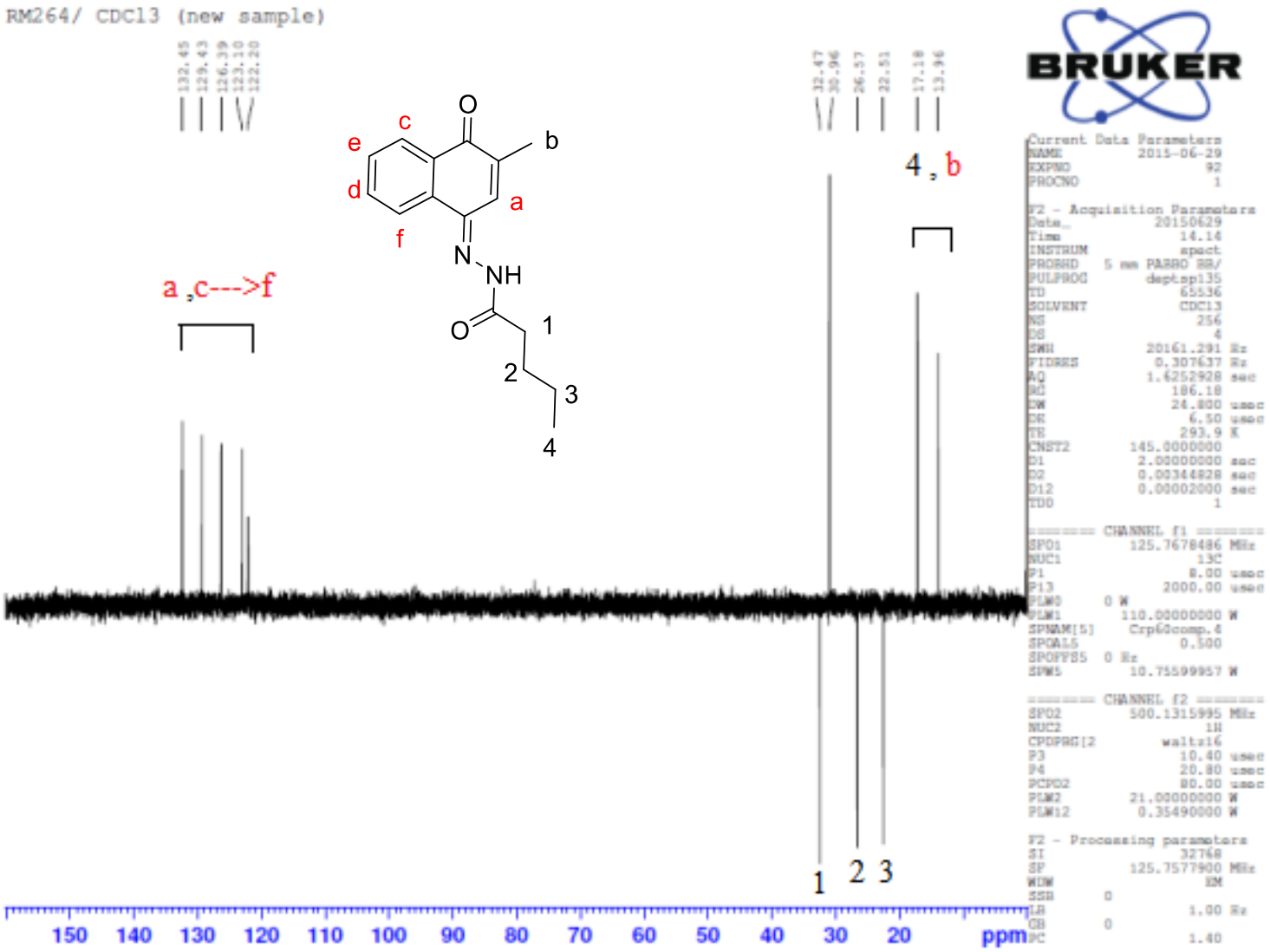
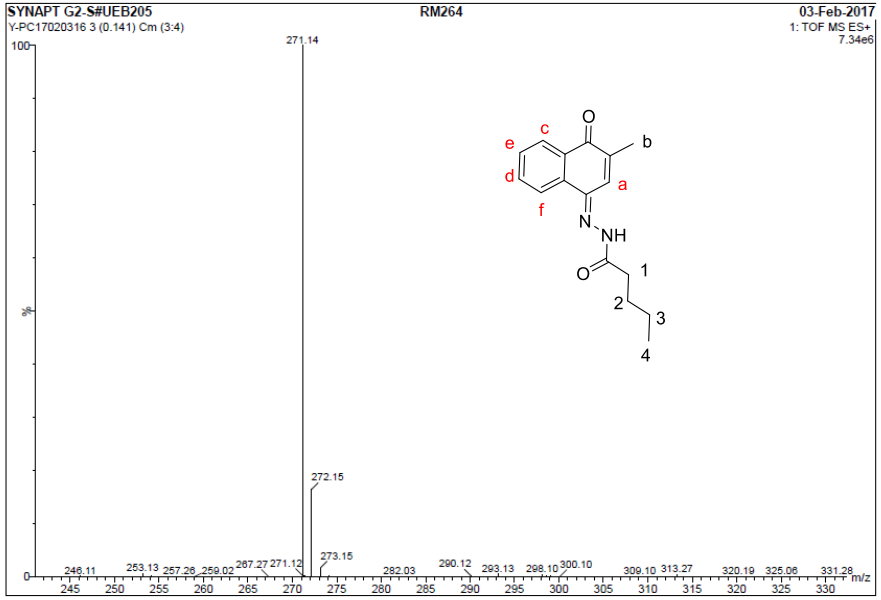


Figure S4. DEPT NMR ($CDCl_3$, 100 MHz) spectrum of compound **6a**



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
467 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 G2-S#UEB205 RM264 03-Feb-2017
Y-PC17020316 3 (0.141) Cm (3:4) 1: TOF MS ES+ 7.34e+006

Minimum:			-1.5					
Maximum:		1.0	1.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
271.1445	271.1447	-0.2	-0.7	8.5	2414.2	0.000	100.00	C16 H19 N2 O2
	271.1452	-0.7	-2.6	1.5	2430.0	15.760	0.00	C H15 N14 O3

Figure S5. HRMS spectrum of compound **6a**

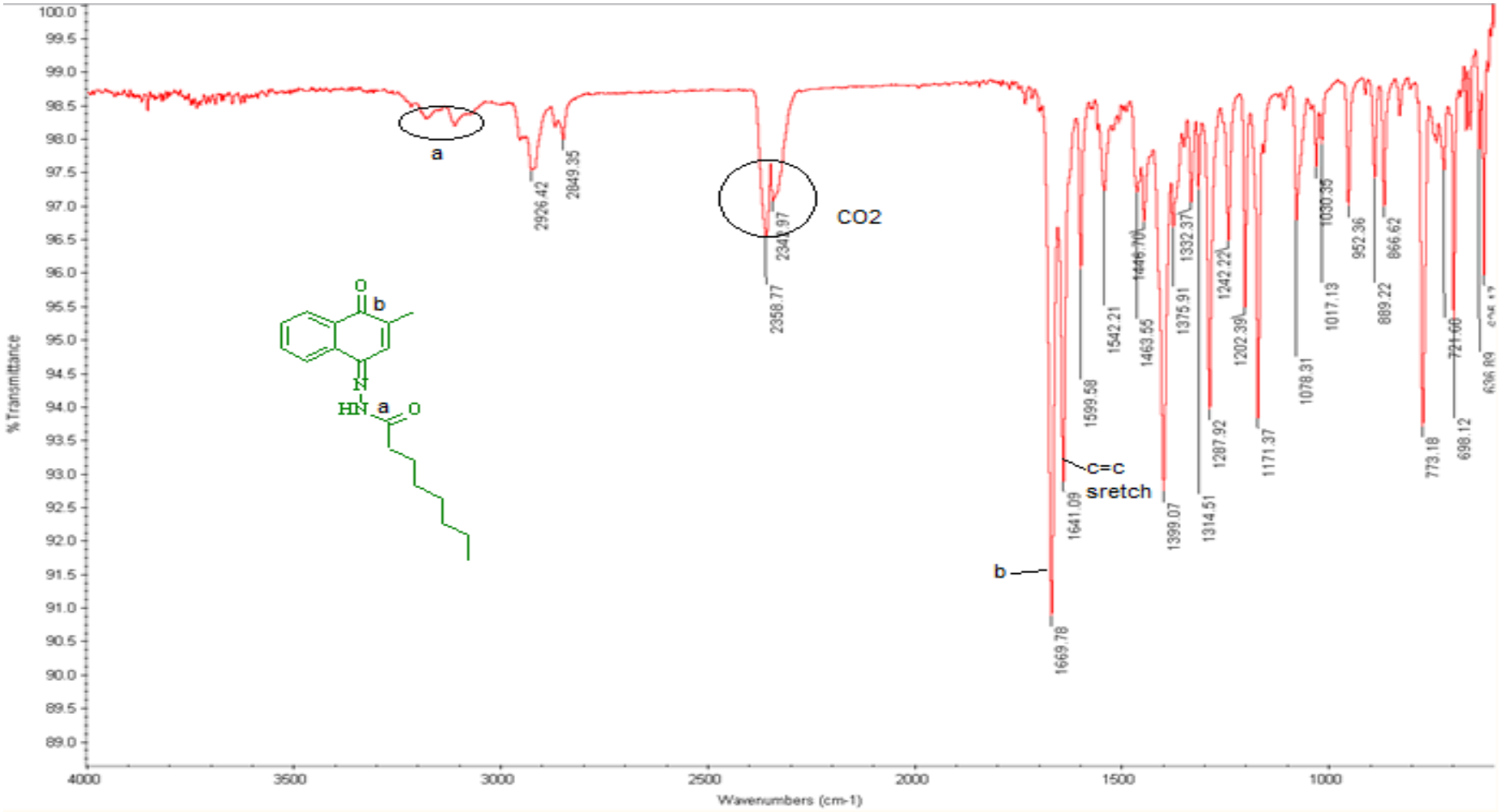


Figure S6. IR spectrum of compound **6b**

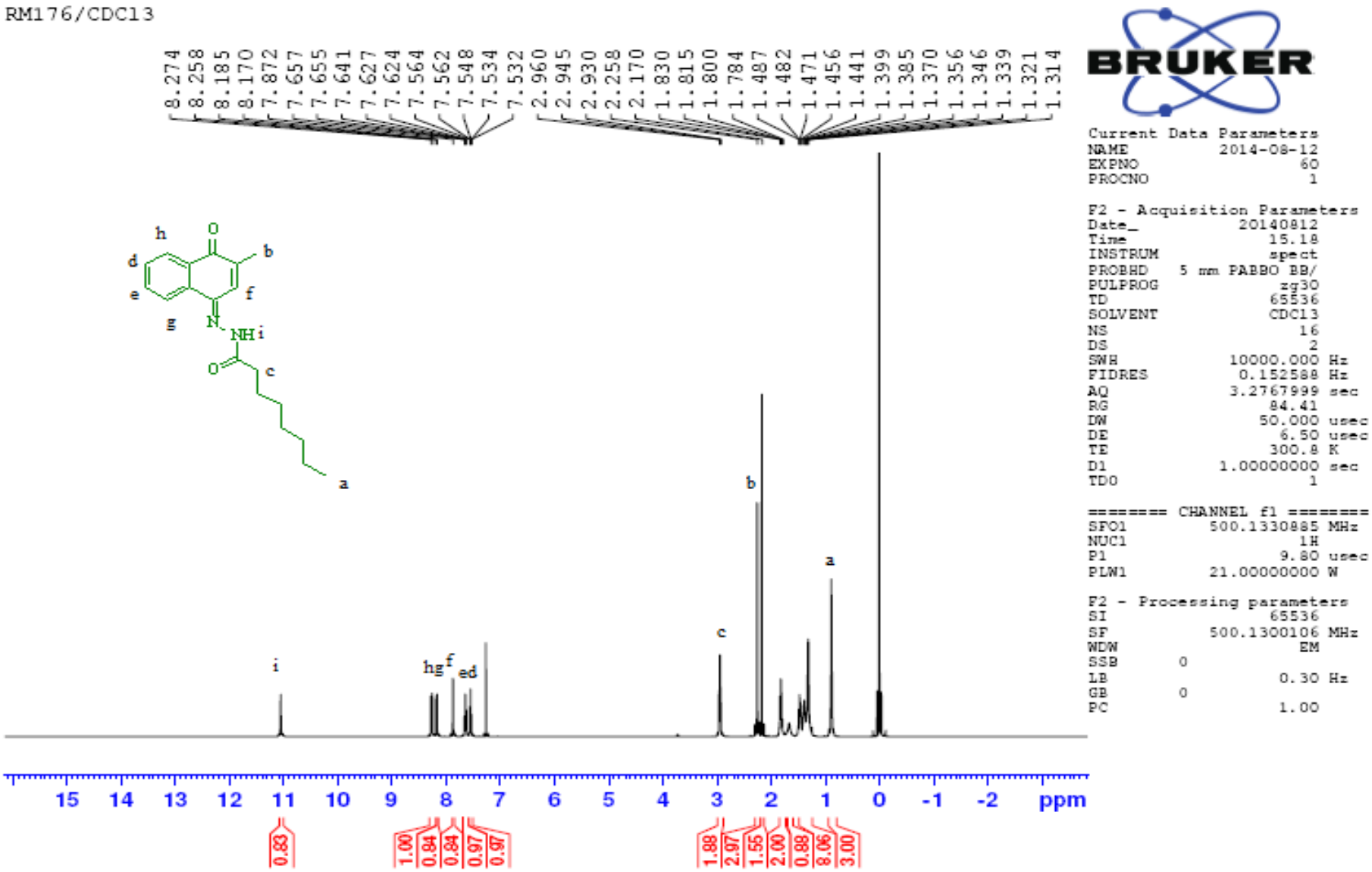


Figure S7. ^{1}H NMR ($CDCl_3$, 500 MHz) spectrum of compound **6b**

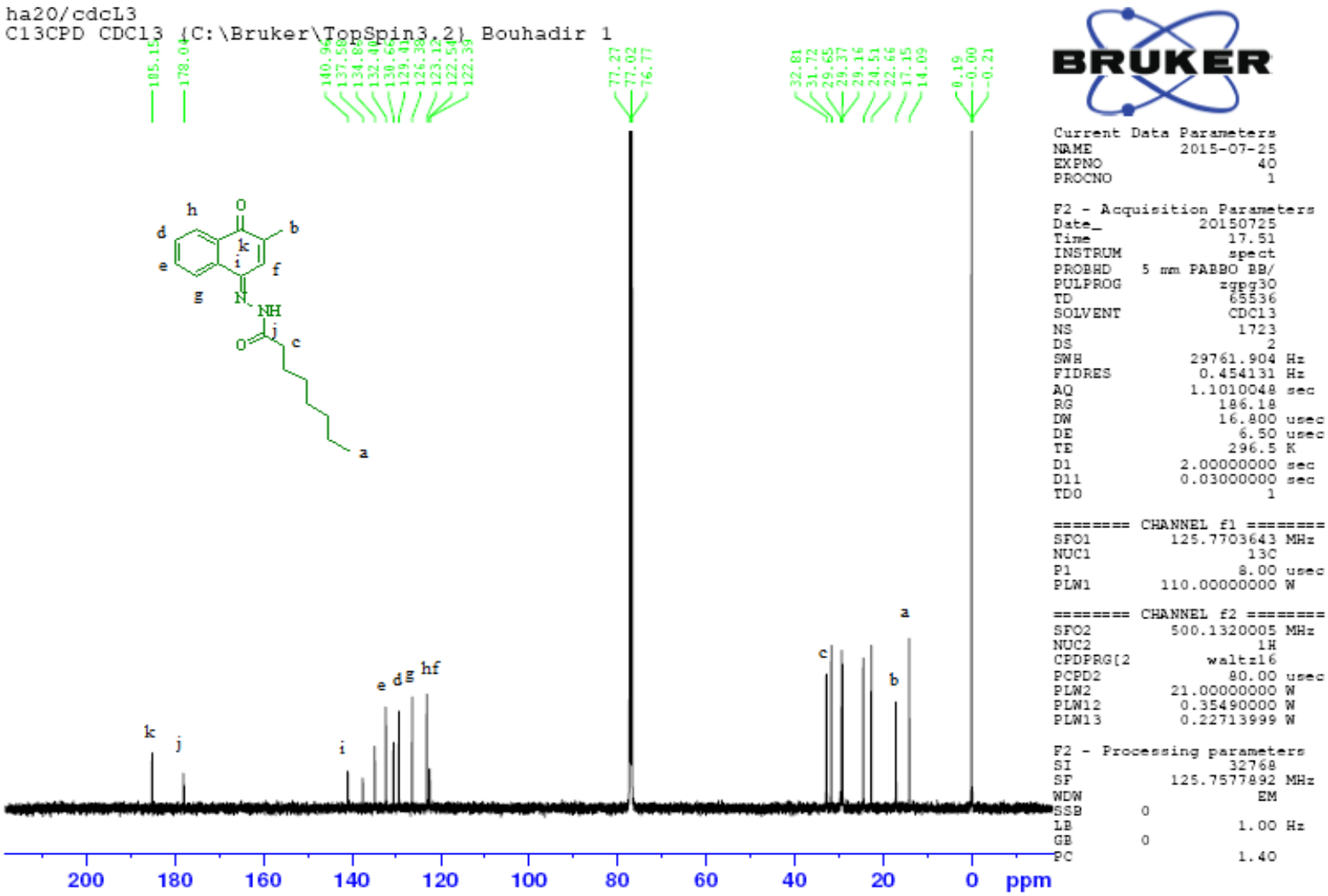


Figure S8. ^{13}C NMR ($CDCl_3$, 100 MHz) spectrum of compound **6b**

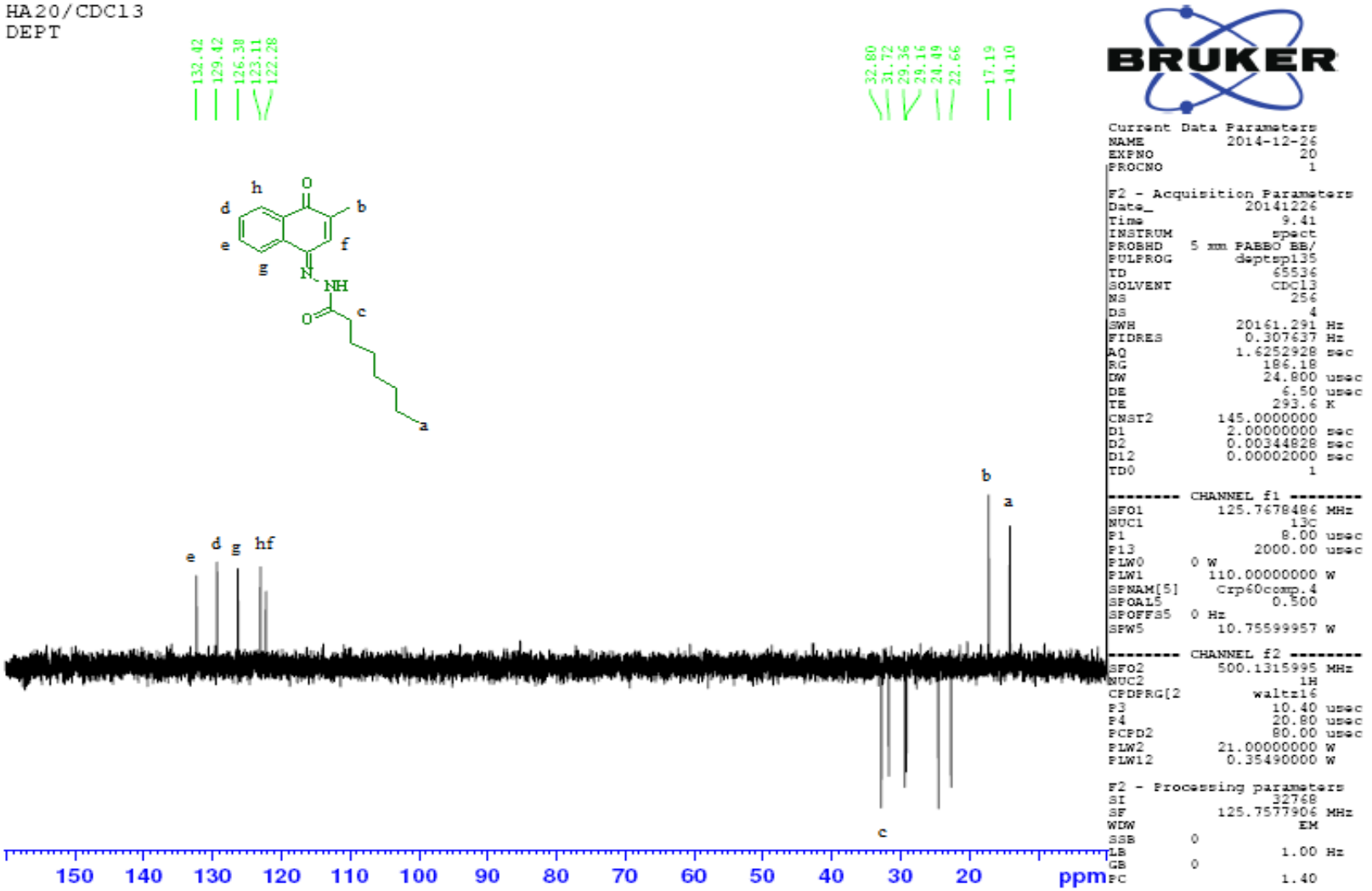


Figure S9. DEPT NMR ($CDCl_3$, 100 MHz) spectrum of compound **6b**

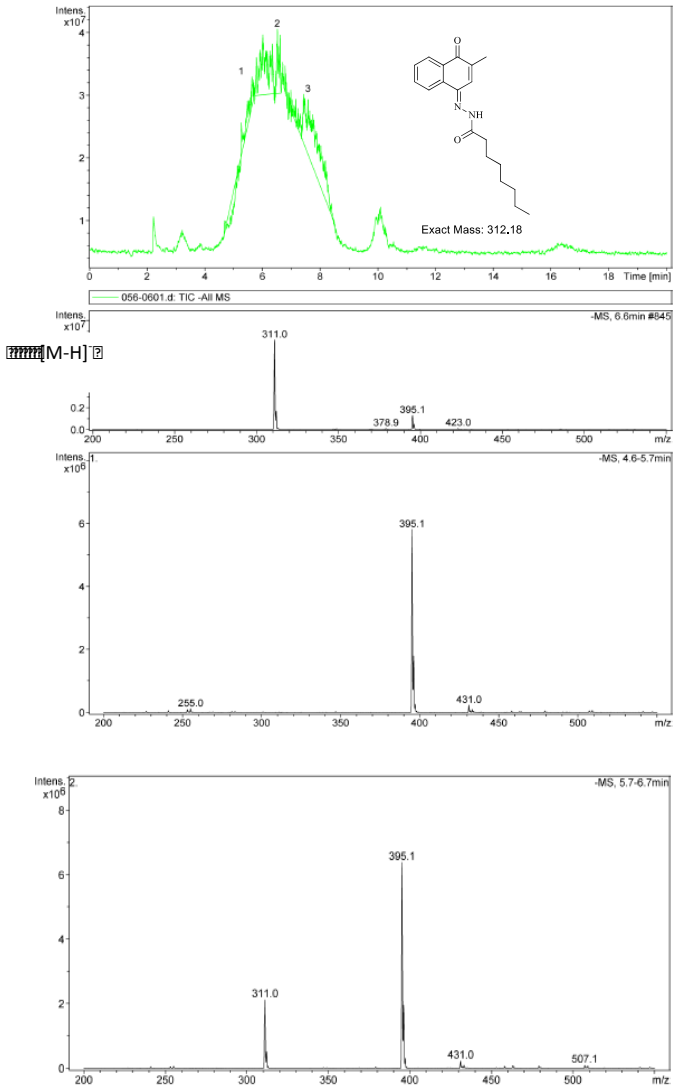
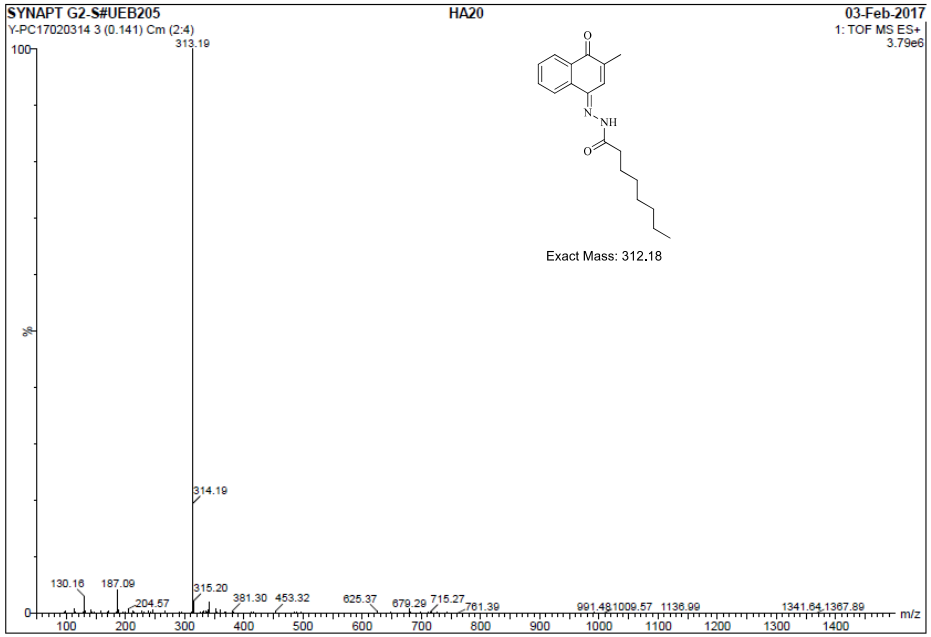


Figure S10. LC-MS spectrum of compound **6b**



Elemental Composition Report Page 1

Single Mass Analysis
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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

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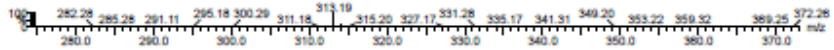
3.79e+006



Minimum:				-1.5				
Maximum:		1.0	1.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
313.1915	313.1916	-0.1	-0.3	8.5	2014.2	0.000	100.00	C19 H25 N2 O2
	313.1921	-0.6	-1.9	1.5	2028.7	14.547	0.00	C4 H21 N14 O3

Figure S11. HRMS spectrum of compound **6b**

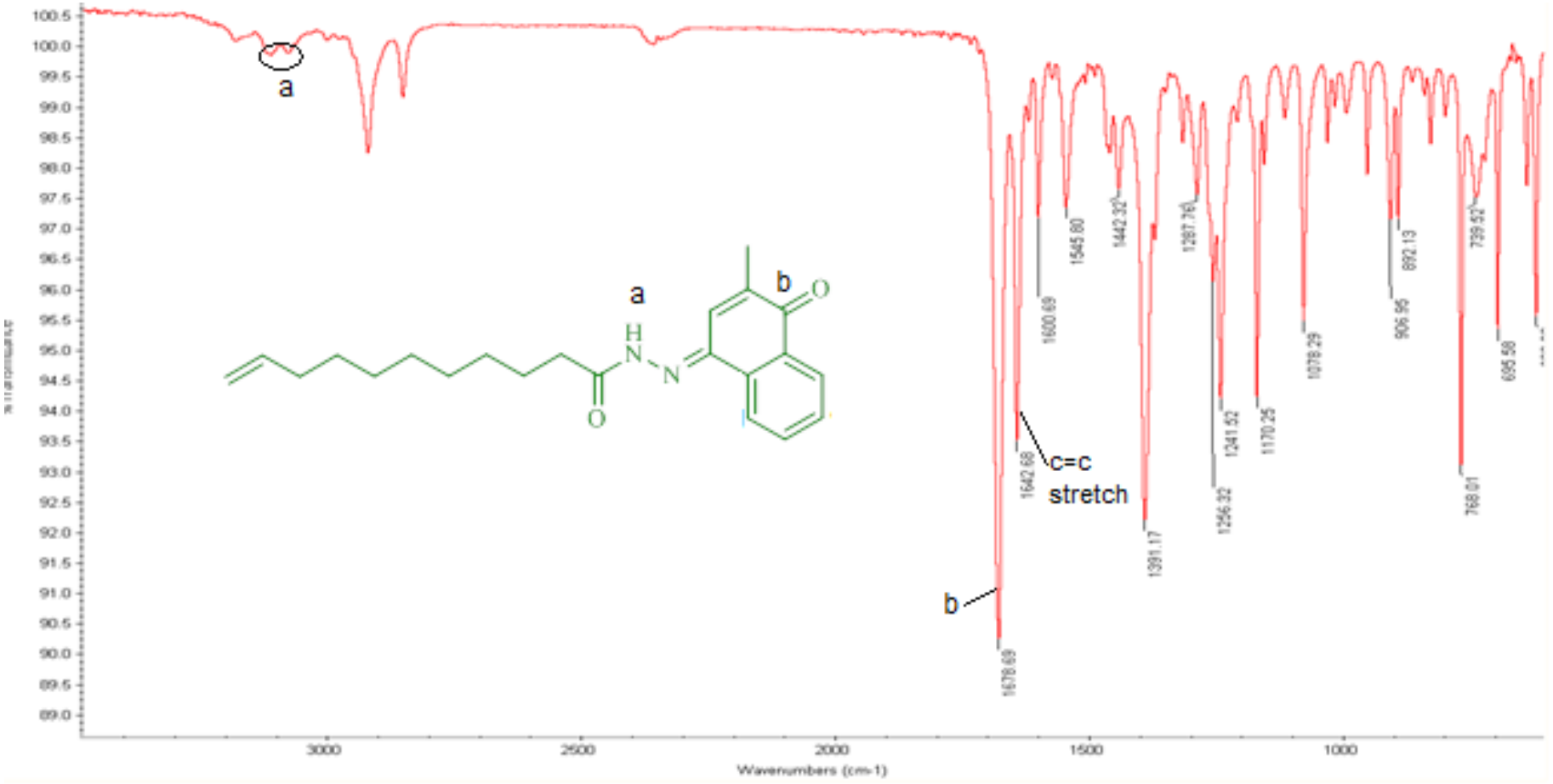


Figure S12. IR spectrum of compound **6c**

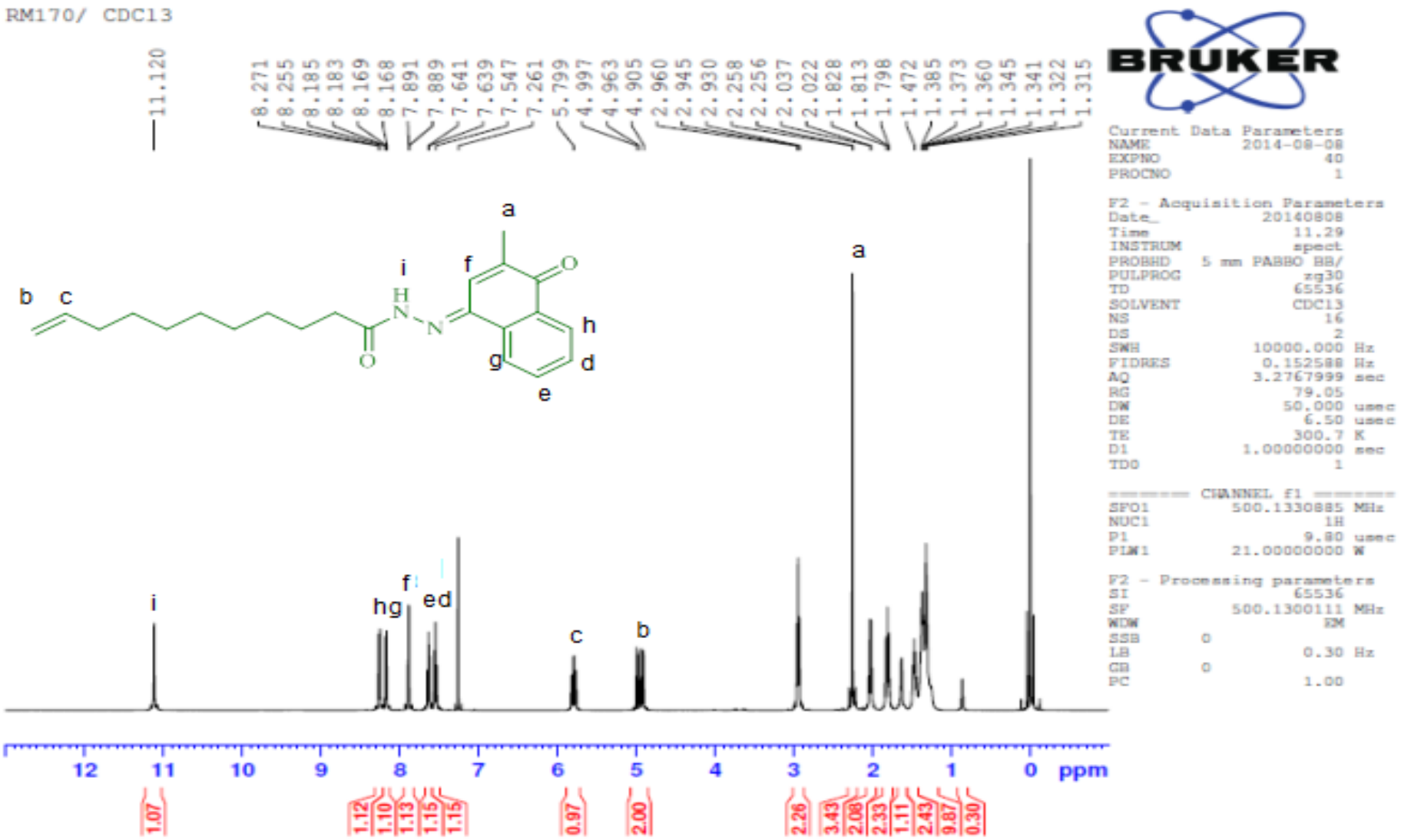


Figure S13. ^{1}H NMR ($CDCl_3$, 500 MHz) spectrum of compound **6c**

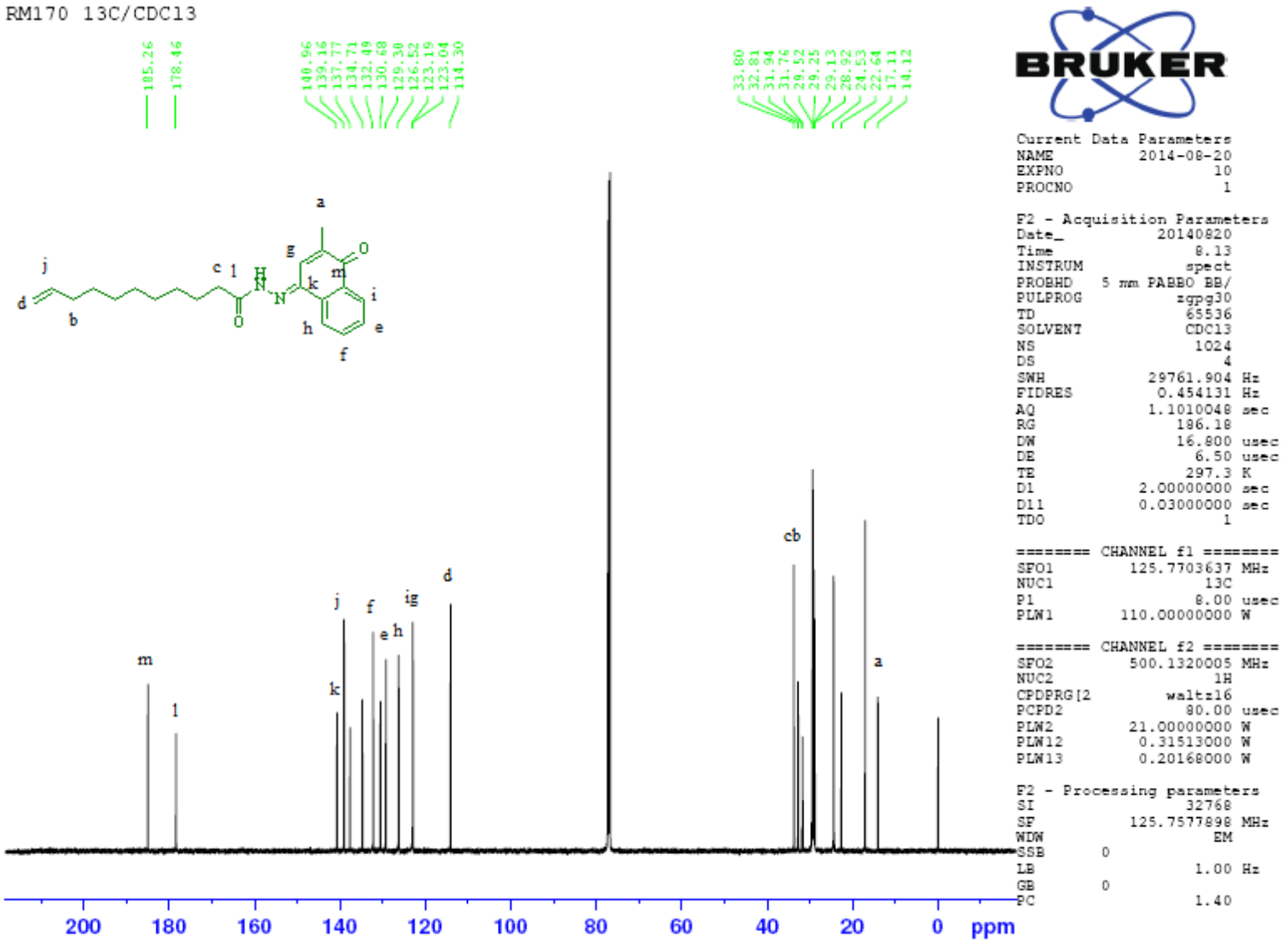
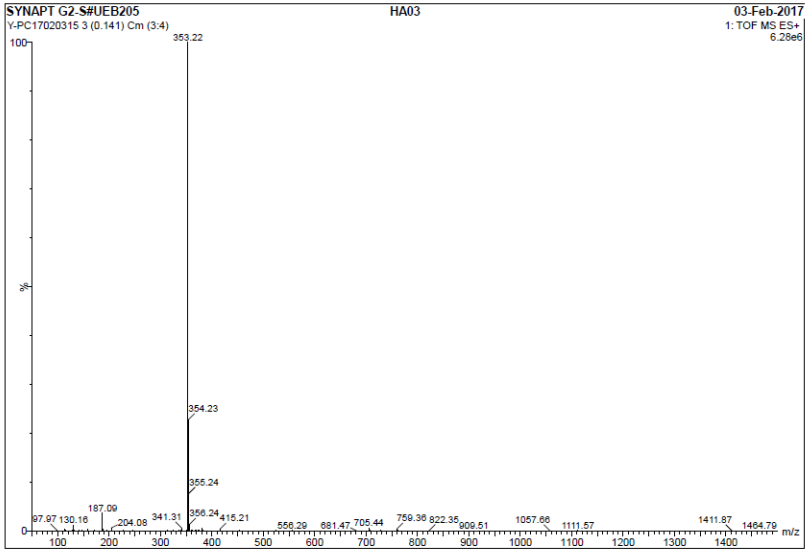


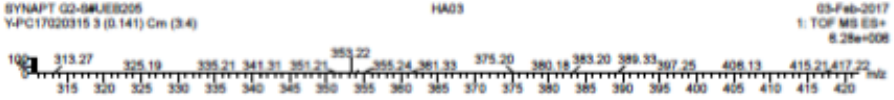
Figure S14. ^{13}C NMR ($CDCl_3$, 100 MHz) spectrum of compound **6c**



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



Minimum:				-1.5				
Maximum:		1.0	1.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
353.2230	353.2229	0.1	0.3	9.5	2033.0	0.022	97.78	C22 H29 N2 O2
	353.2234	-0.4	-1.1	2.5	2036.8	3.809	2.22	C7 H25 N14 O3

Figure S15. HRMS spectrum of compound **6b**

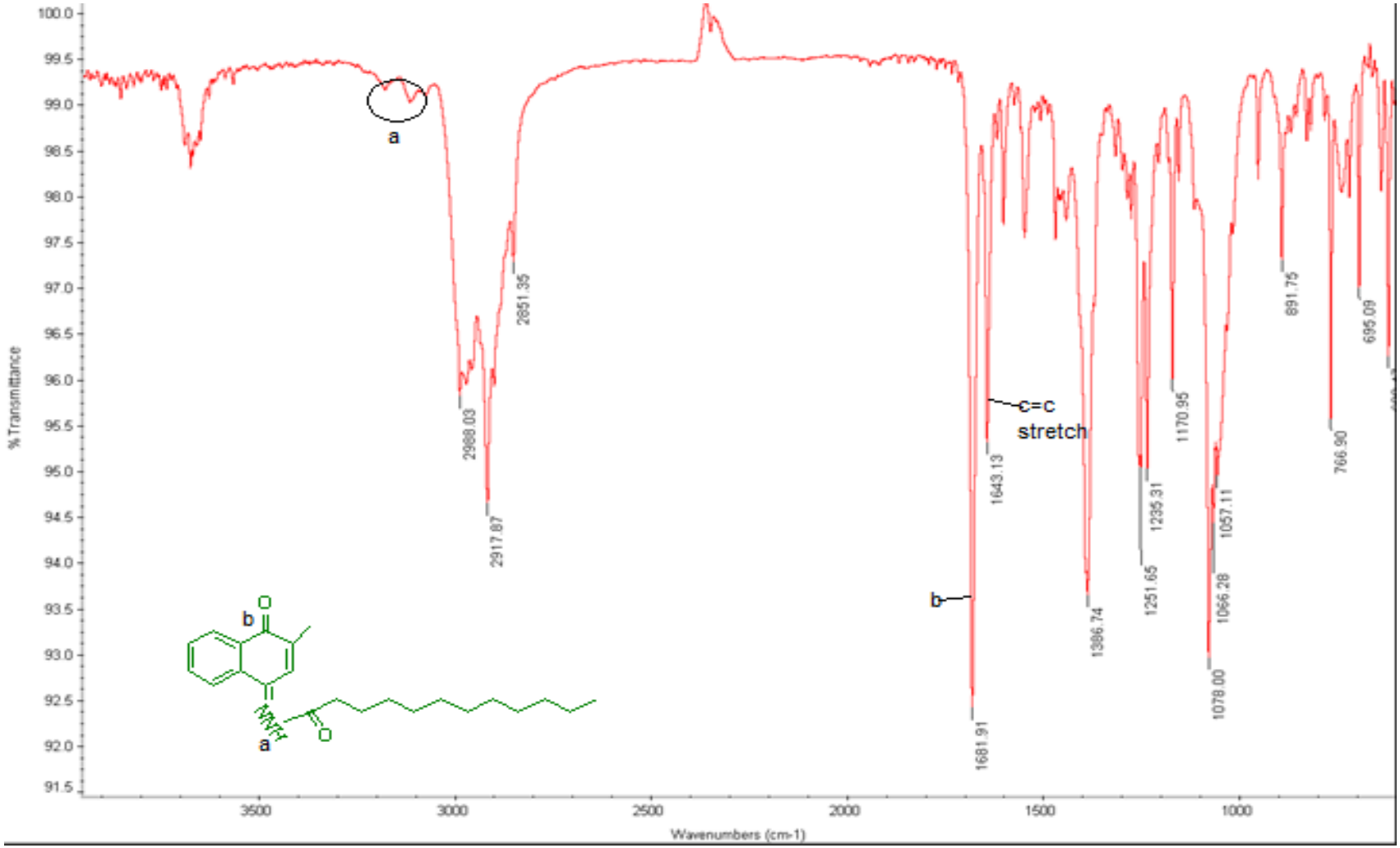


Figure S16. IR spectrum of compound **6d**

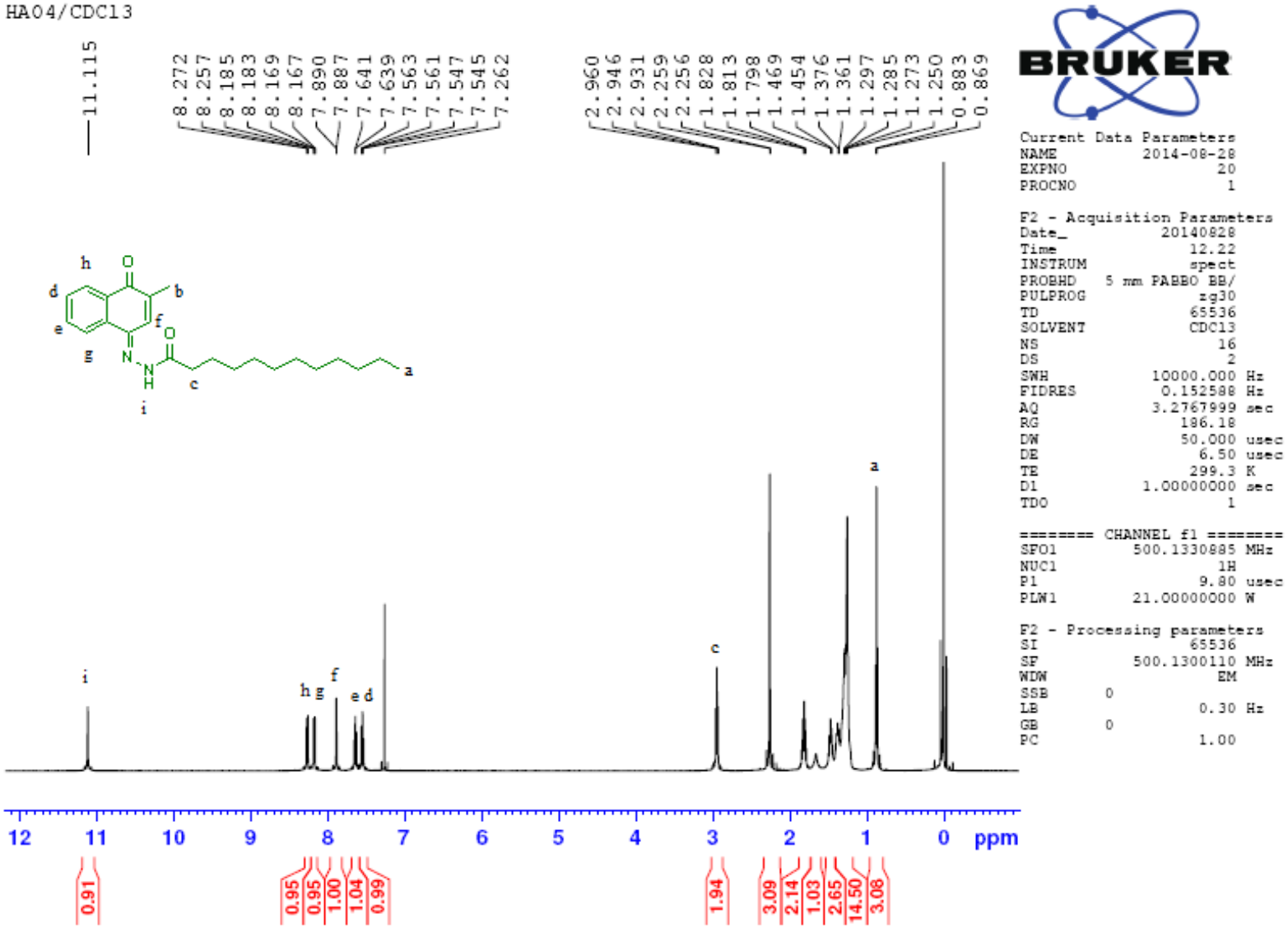


Figure S17. ^{1}H NMR ($CDCl_3$, 500 MHz) spectrum of compound **6d**

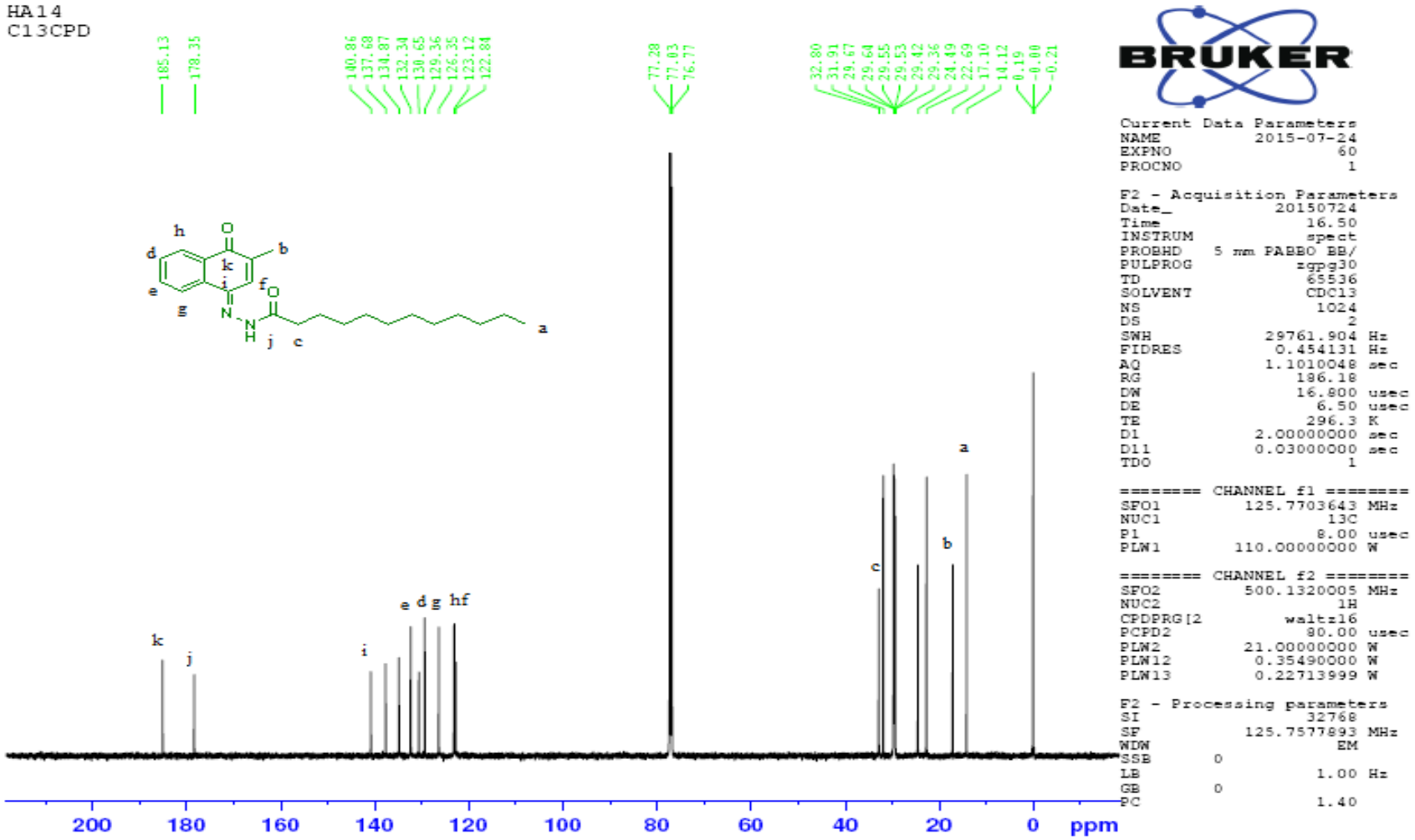


Figure S18. ^{13}C NMR ($CDCl_3$, 100 MHz) spectrum of compound **6d**

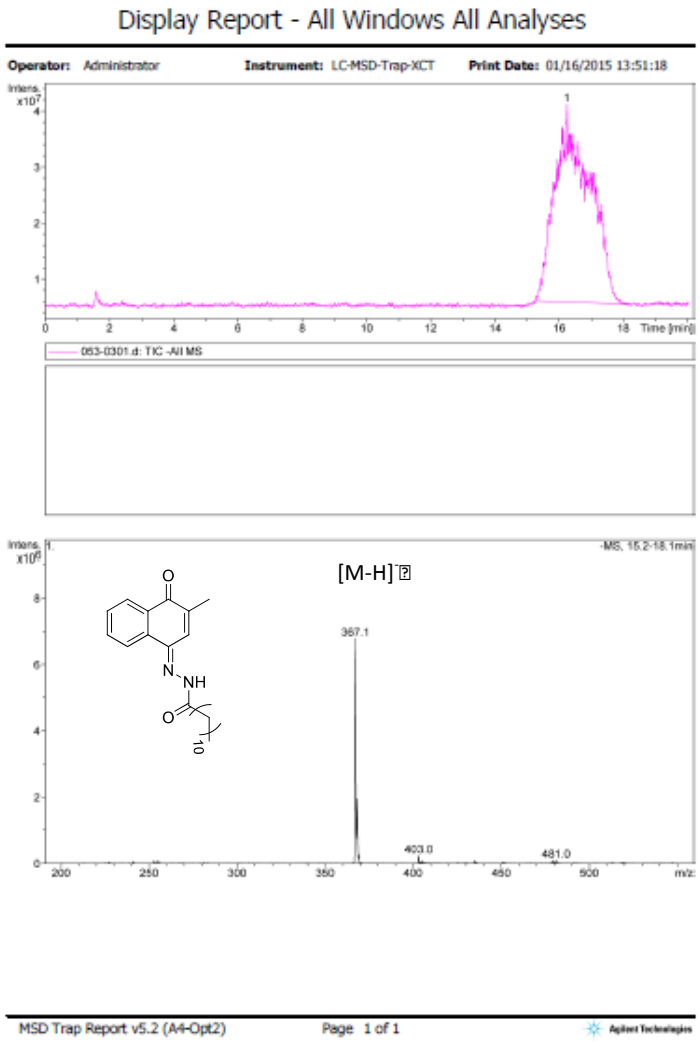
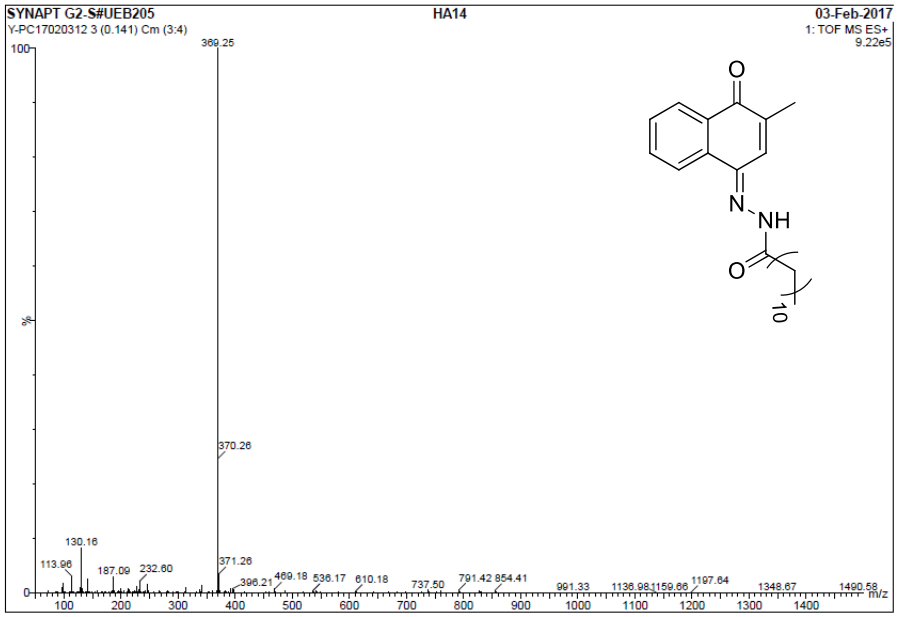
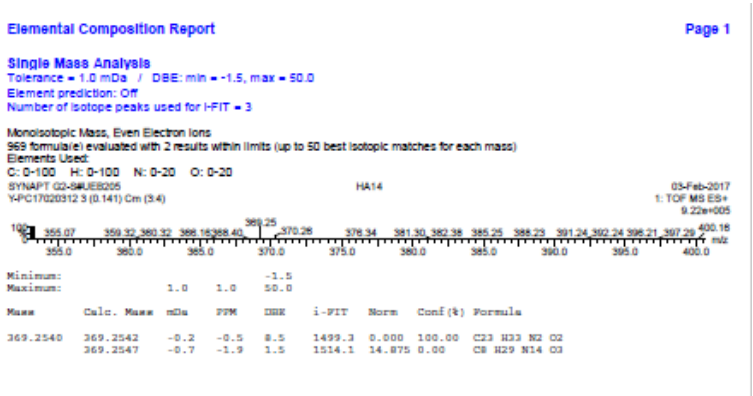


Figure S19. LC-MS spectrum of compound **6d**





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
969 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 G2-S#UEB205 HA14 03-Feb-2017
Y-PC17020312 3 (0.141) Cm (3:4) 1: TOF MS ES+ 9.22e+005

	Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
Minimum:					-1.5				
Maximum:			1.0	1.0	50.0				
	369.2540	369.2542	-0.2	-0.5	8.5	1499.3	0.000	100.00	C23 H33 N2 O2
		369.2547	-0.7	-1.9	1.5	1514.1	14.875	0.00	C8 H29 N14 O3

Figure S20. HRMS spectrum of compound **6d**

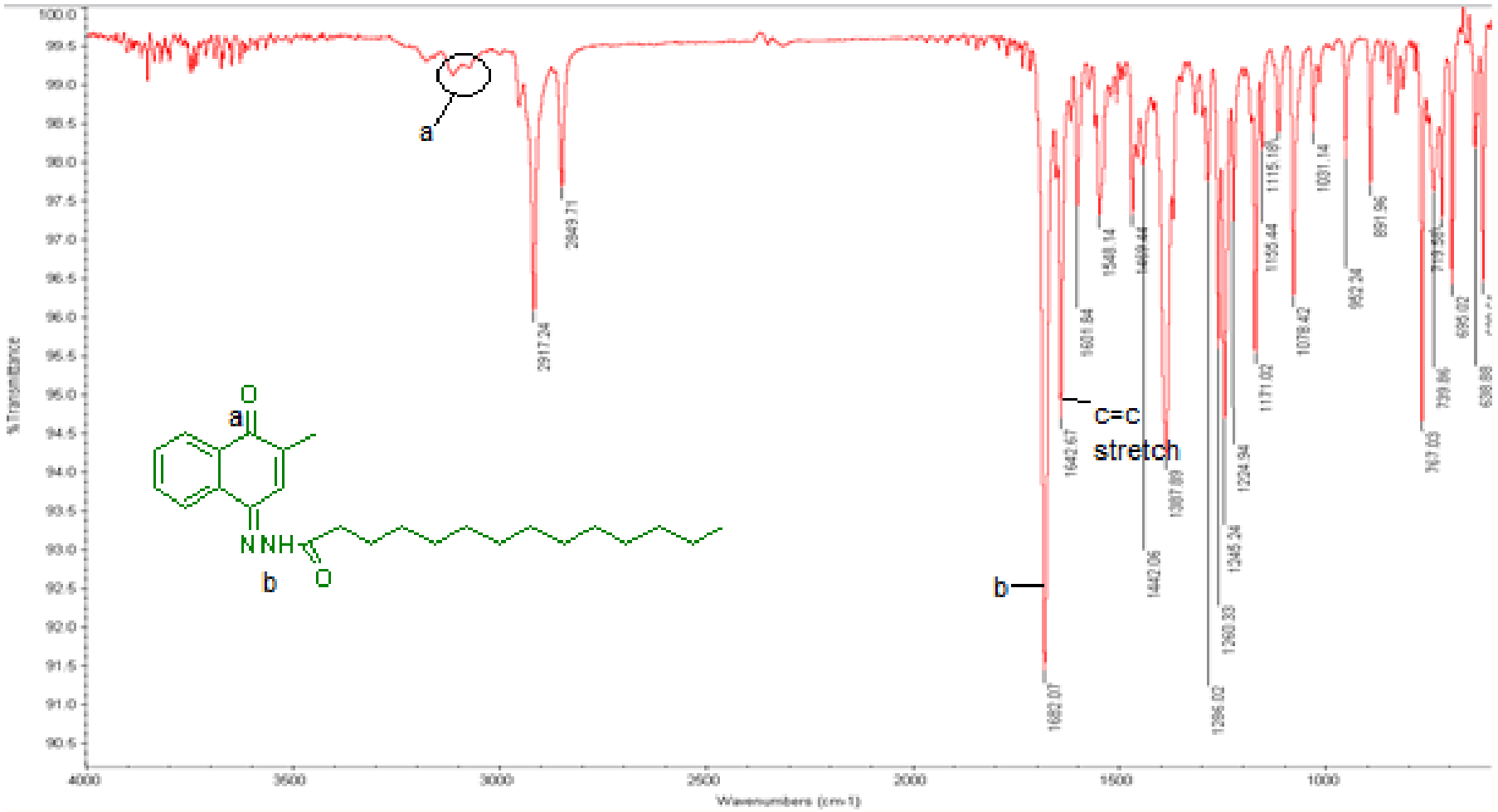


Figure S21. IR spectrum of compound **6e**

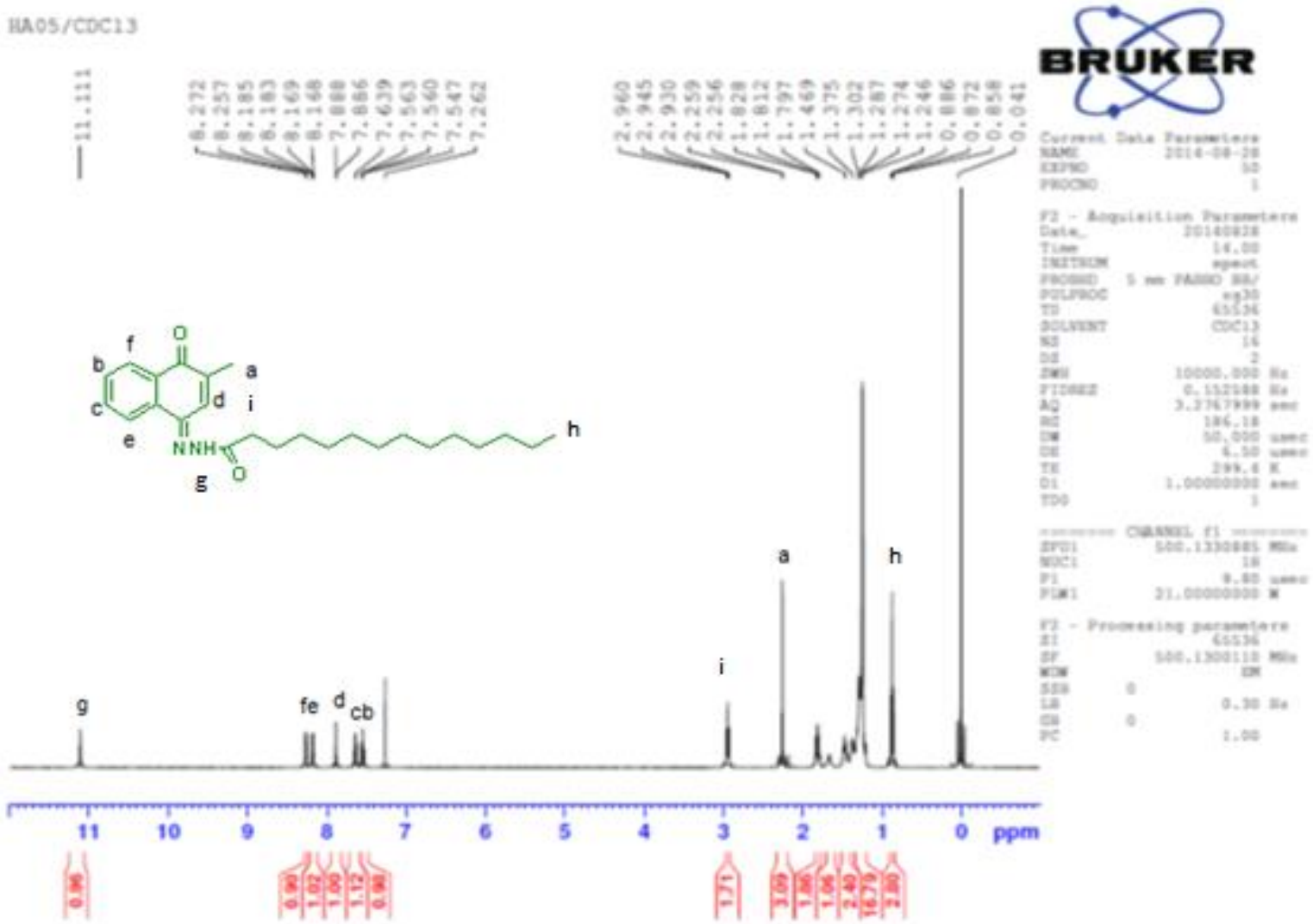


Figure S22. ^{1}H NMR ($CDCl_3$, 500 MHz) spectrum of compound **6e**

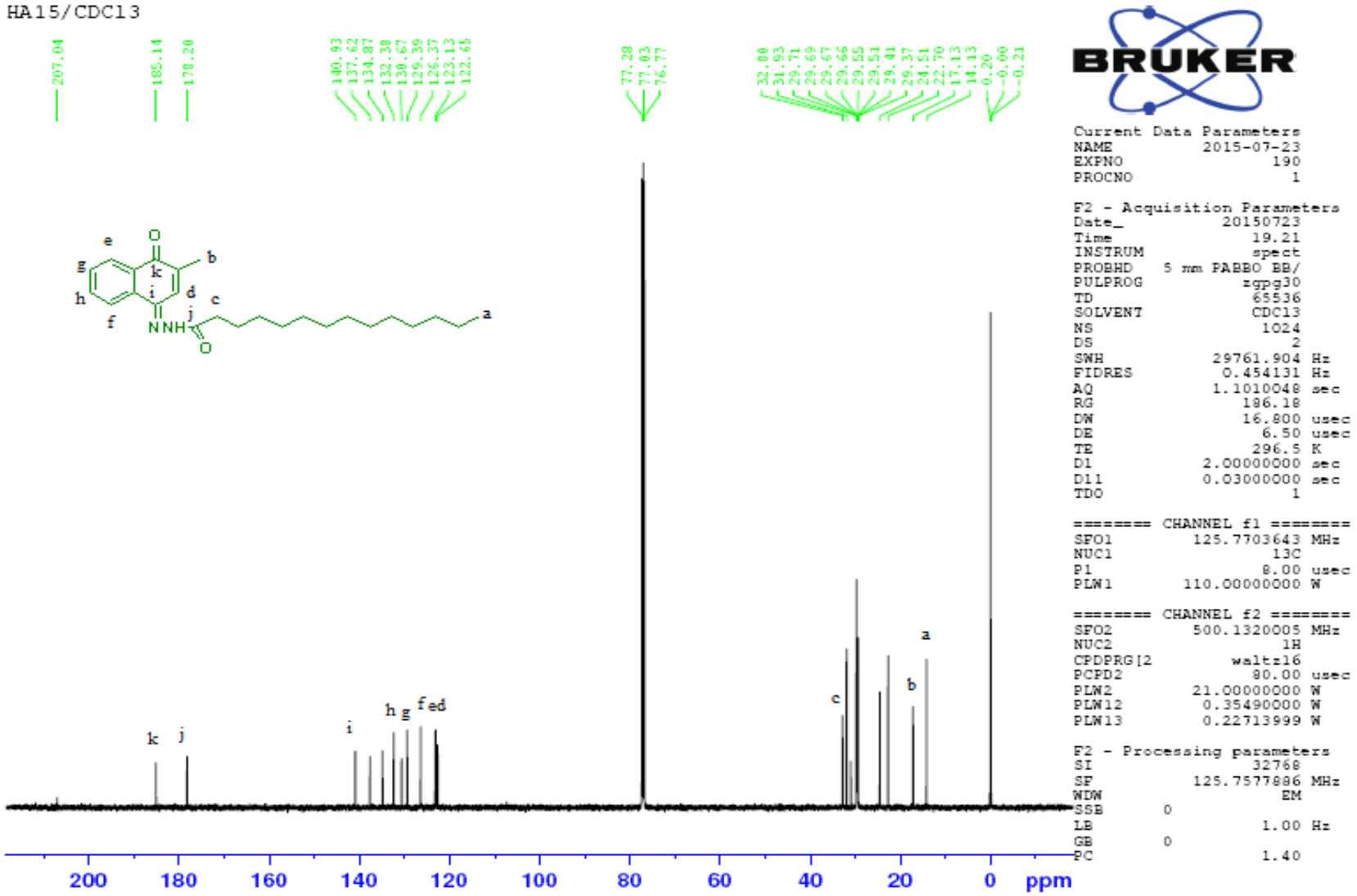


Figure S23. ^{13}C NMR ($CDCl_3$, 100 MHz) spectrum of compound **6e**

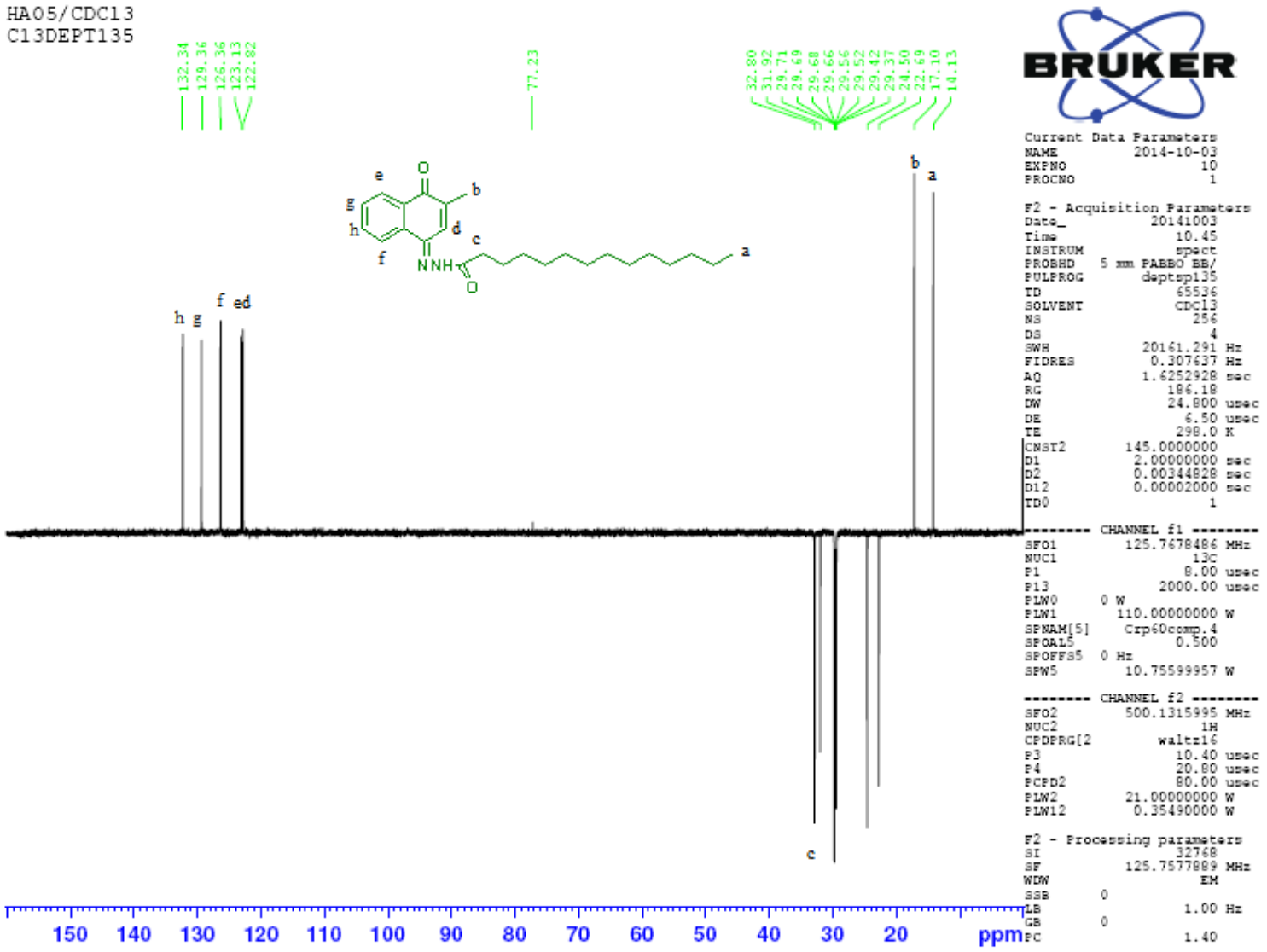
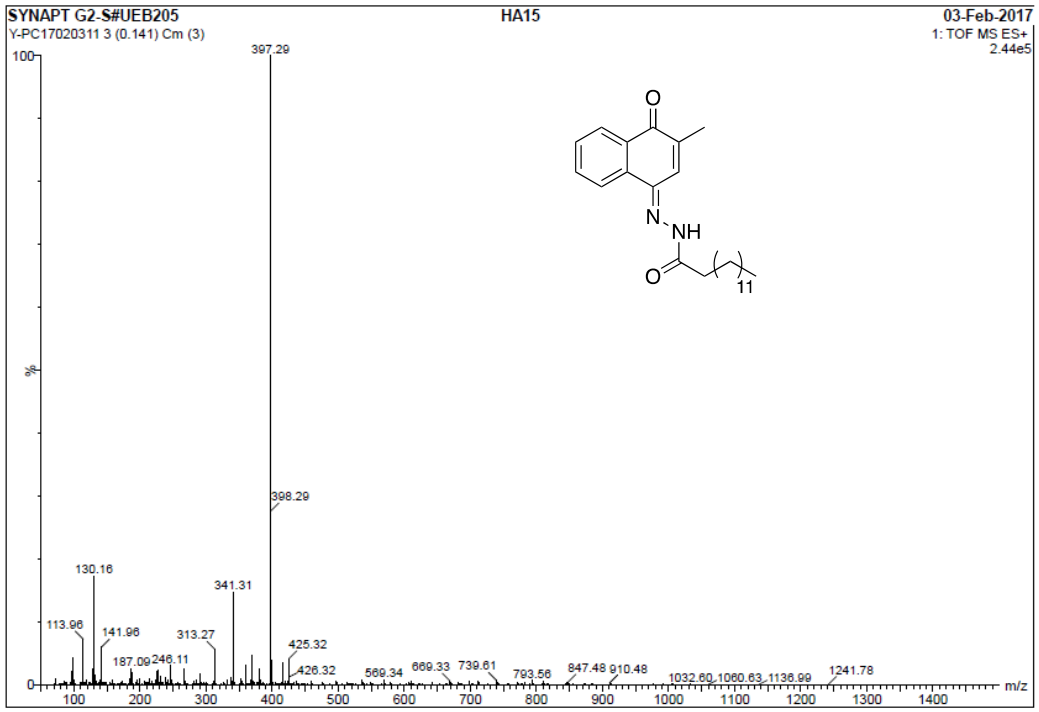


Figure S24. DEPT NMR ($CDCl_3$, 100 MHz) spectrum of compound **6e**



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

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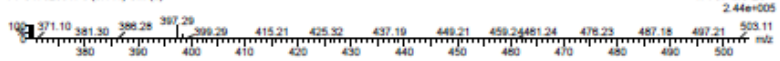
2.44e+005



Minimum:				-1.5				
Maximum:		1.0	1.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
397.2852	397.2855	-0.3	-0.8	8.5	1021.8	0.000	100.00	C25 H37 N2 O2
	397.2860	-0.8	-2.0	1.5	1036.6	14.744	0.00	C10 H33 N14 O3

Figure S25. HRMS spectrum of compound **6e**

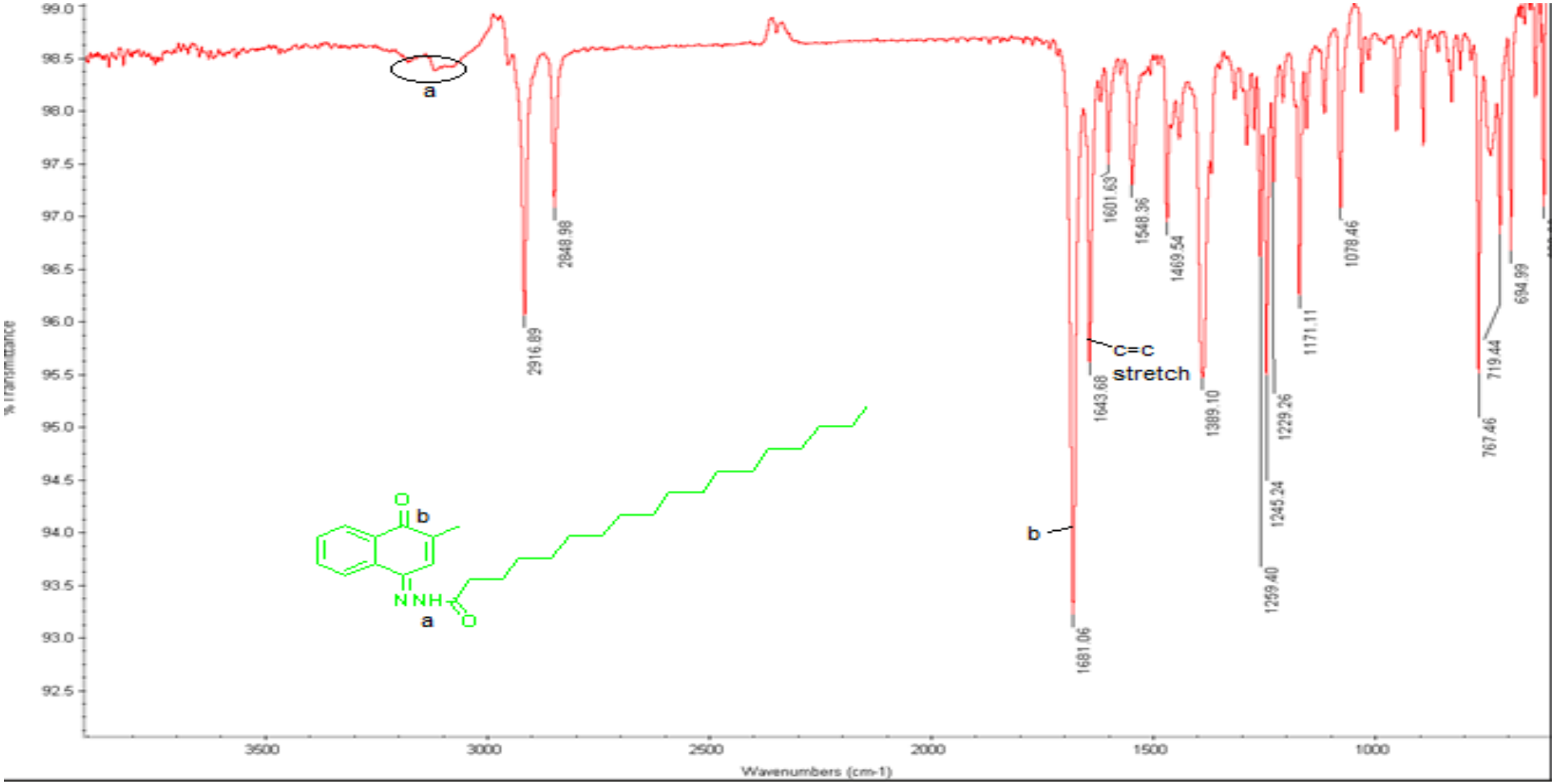


Figure S26. IR spectrum of compound **6f**

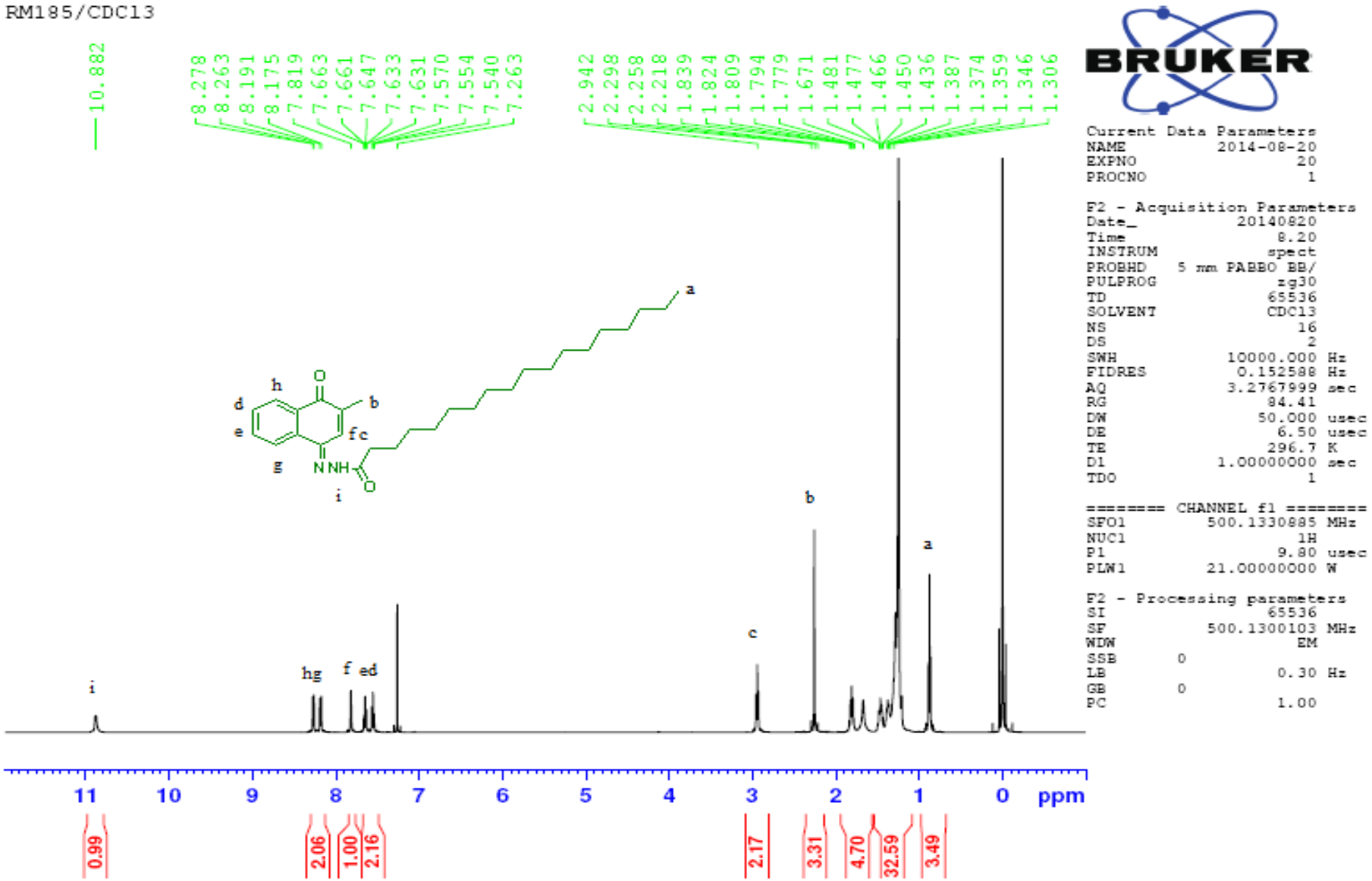


Figure S27. ^{1}H NMR ($CDCl_3$, 500 MHz) spectrum of compound **6f**

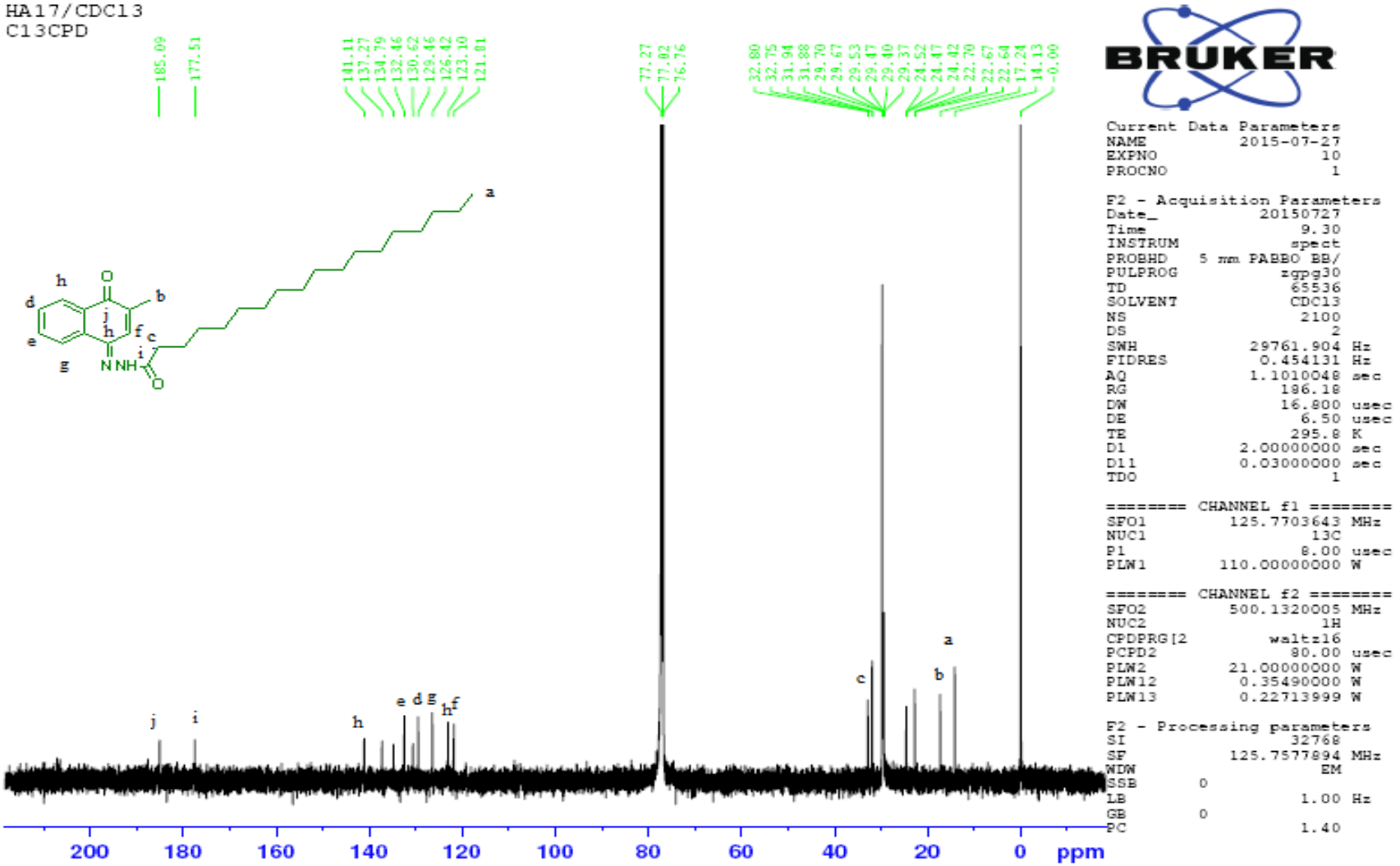


Figure S28. ^{13}C NMR ($CDCl_3$, 100 MHz) spectrum of compound **6f**

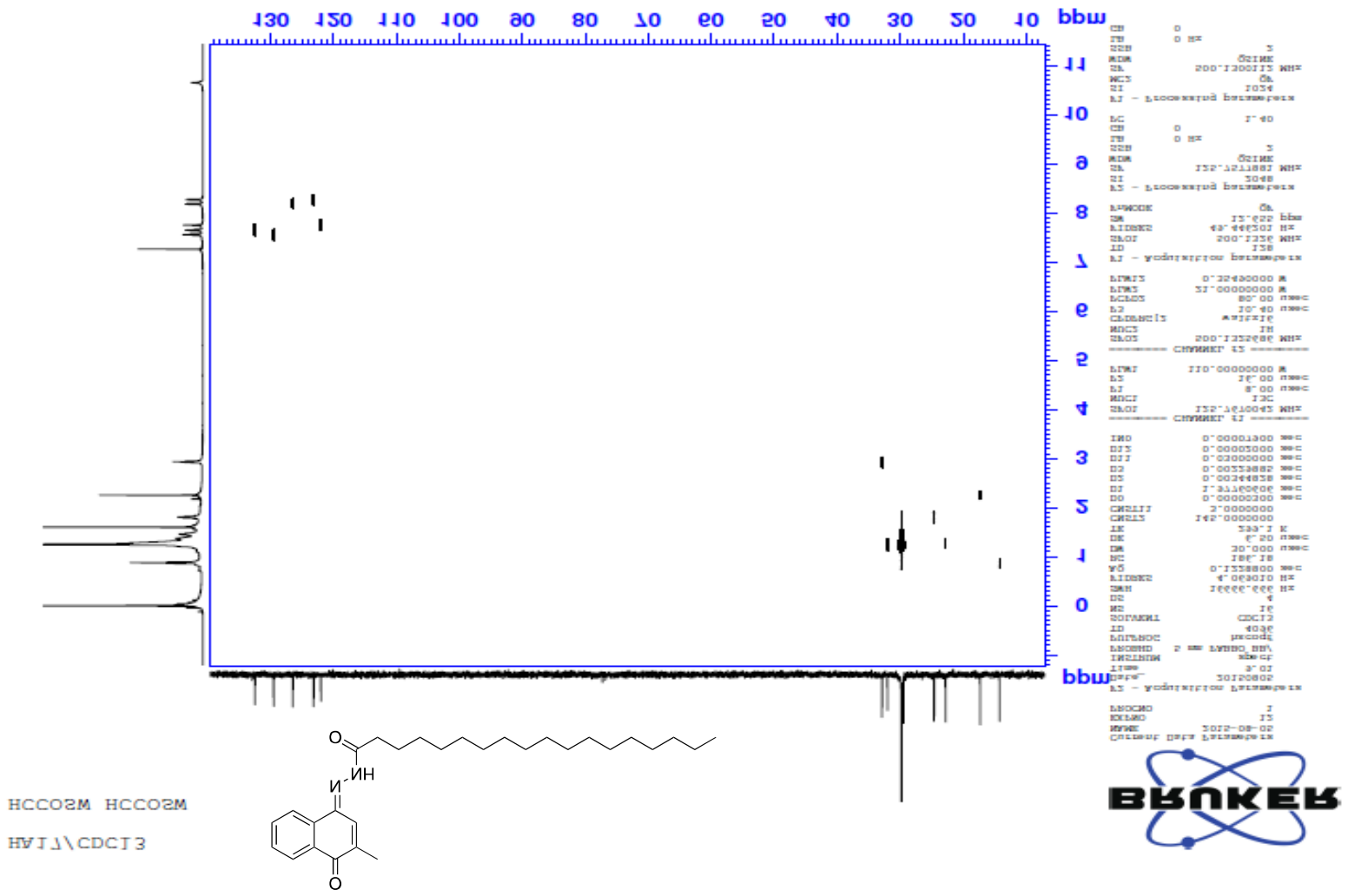


Figure S29. HMQC 2D-NMR spectrum of compound **6f**

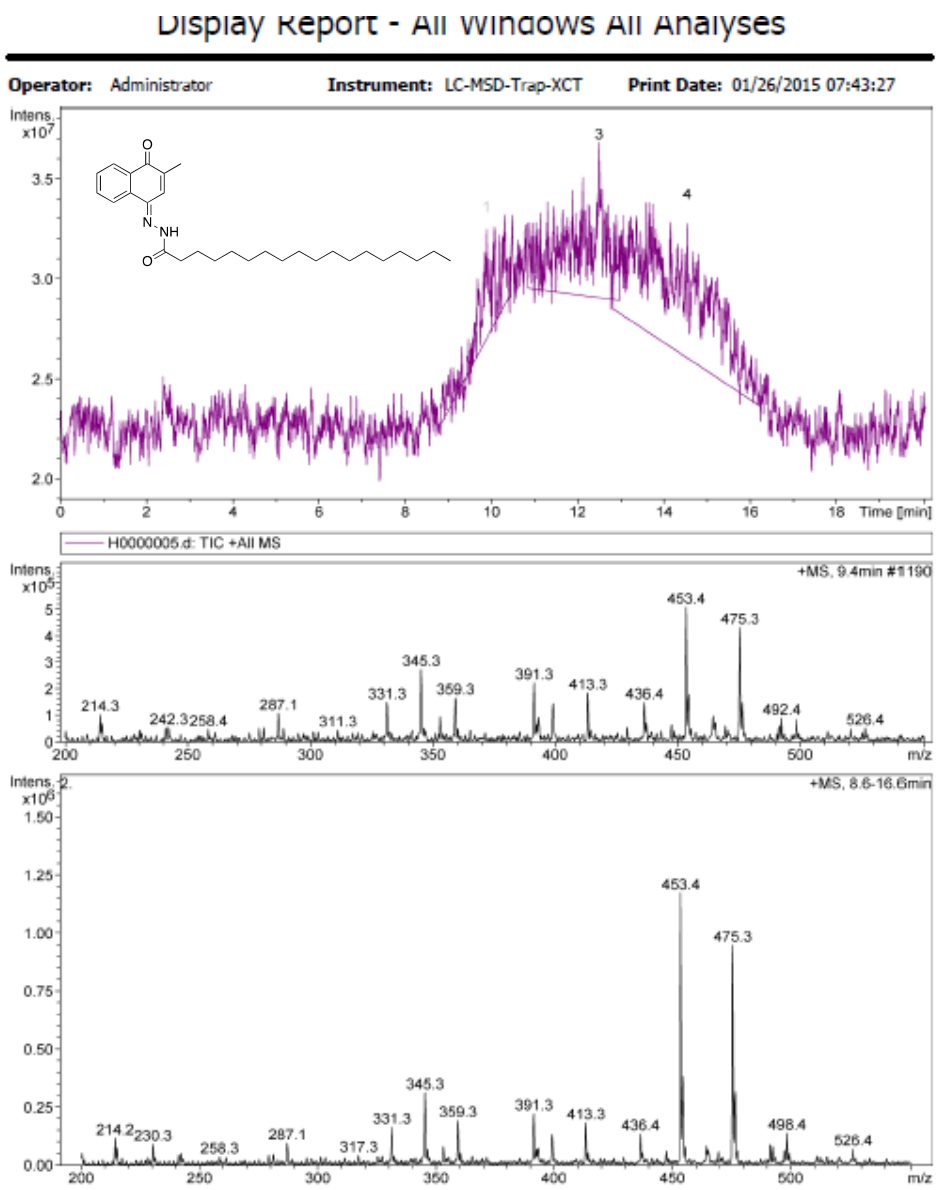
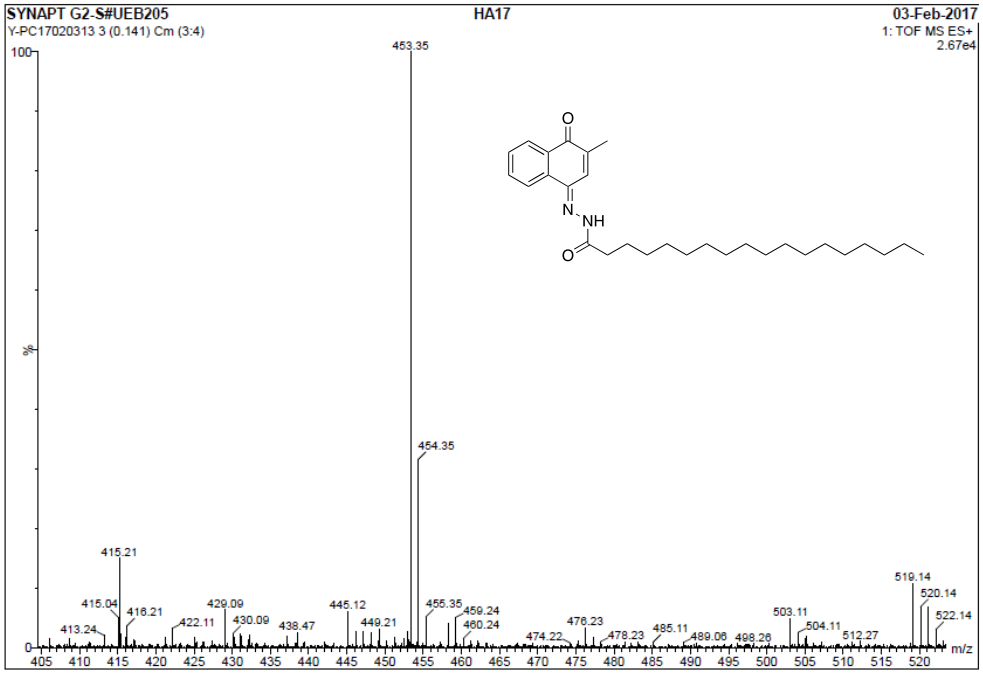


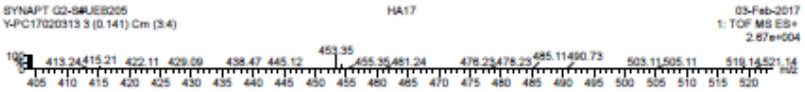
Figure S30. LC-MS spectrum of compound **6f**



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
1455 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



Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
Minimum:				-1.5				
Maximum:		1.0	1.0	50.0				
453.3480	453.3481	-0.1	-0.2	8.5	592.6	0.000	99.98	C29 H45 N2 O2
	453.3486	-0.6	-1.3	1.5	600.9	8.348	0.02	C14 H41 N14 O3

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

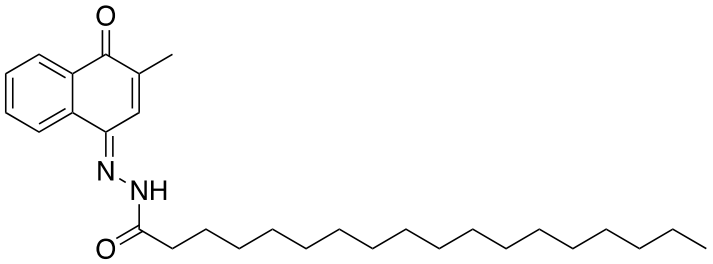


Figure S32. Elemental analysis of compound **6f**

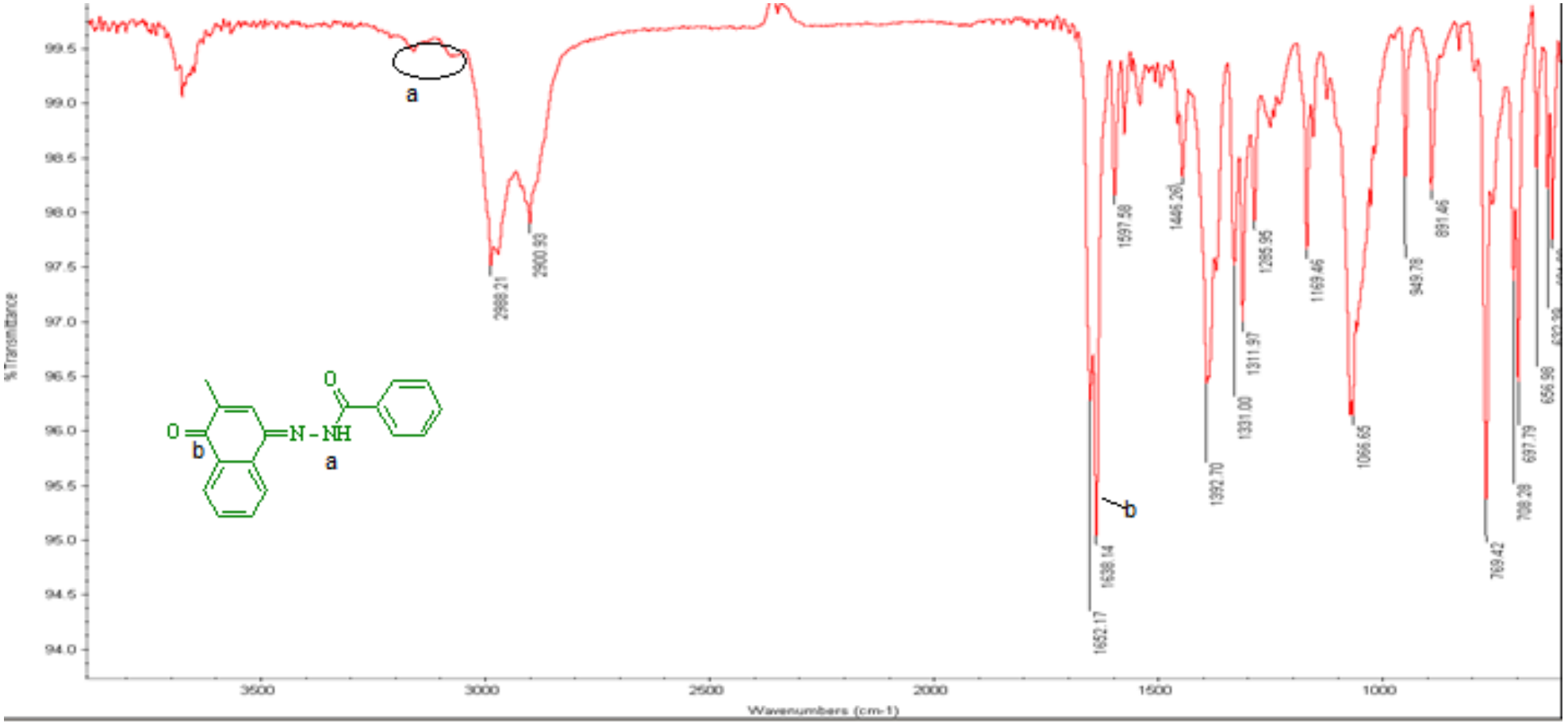


Figure S33. IR spectrum of compound **6g**

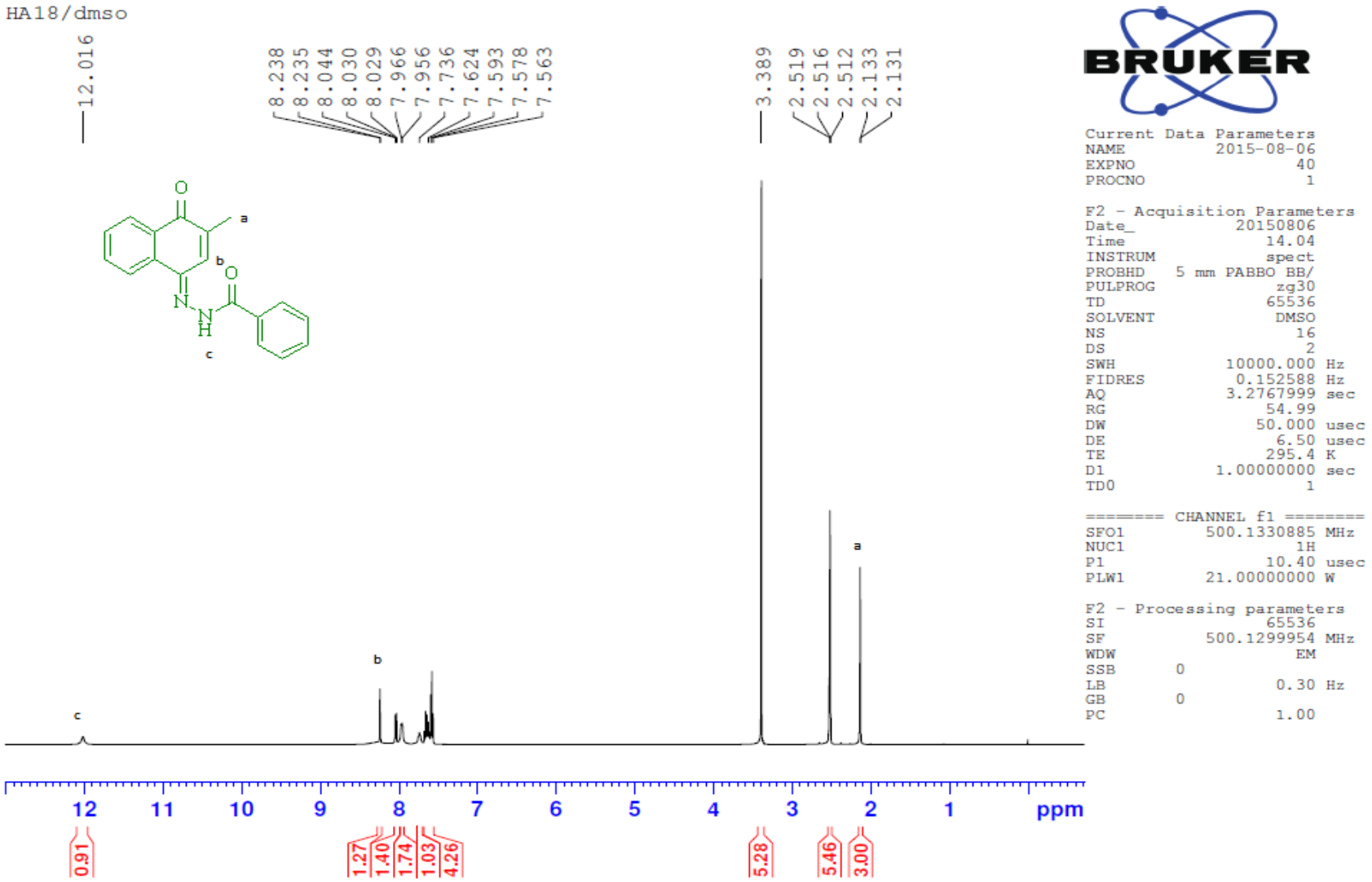


Figure S34. ^{1}H NMR (DMSO, 500 MHz) spectrum of compound **6g**

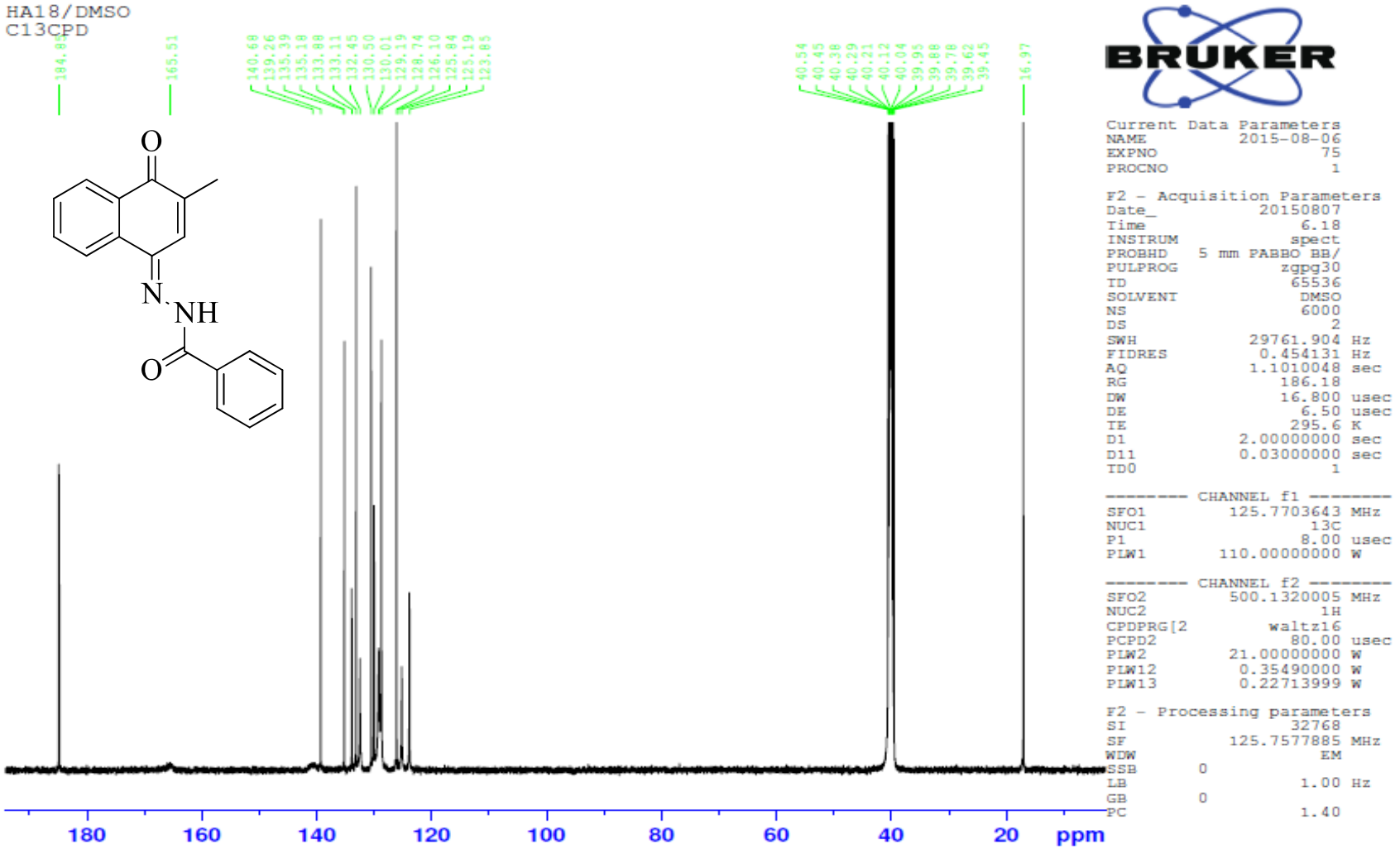


Figure S35. ^{13}C NMR (DMSO, 100 MHz) spectrum of compound **6g**

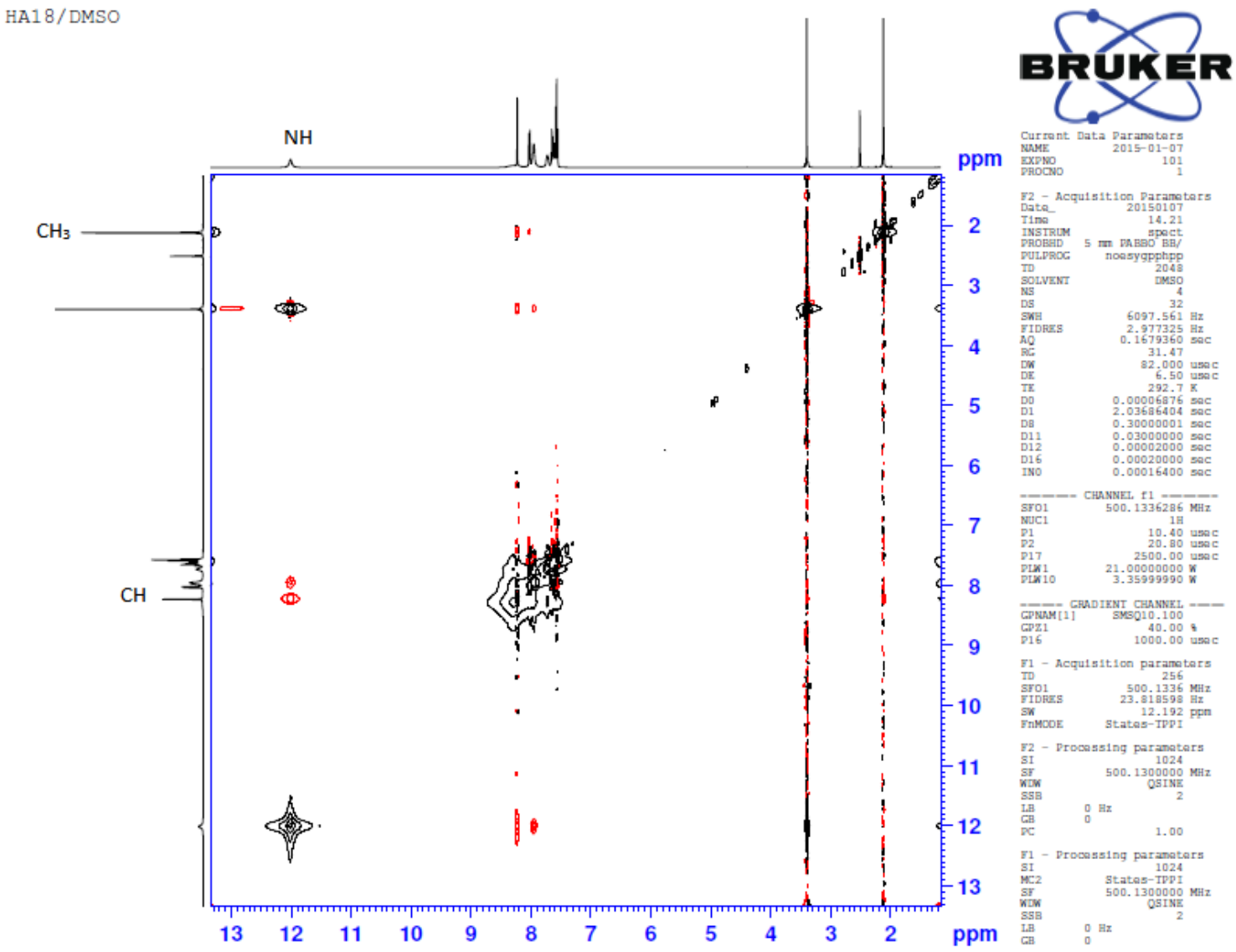


Figure S36. NOESY 2D-NMR spectrum of compound **6g**

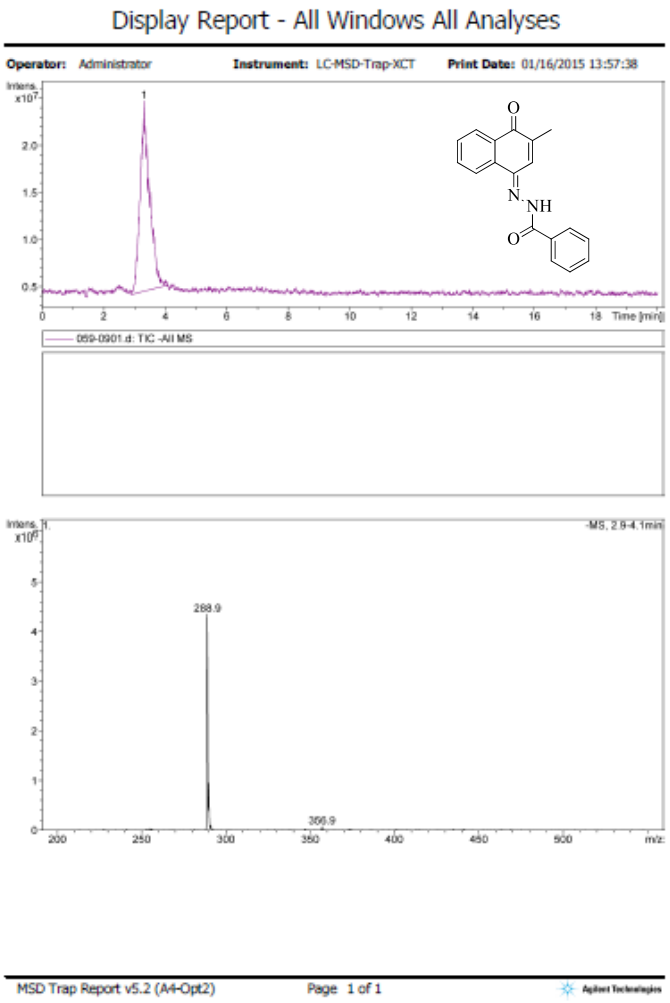
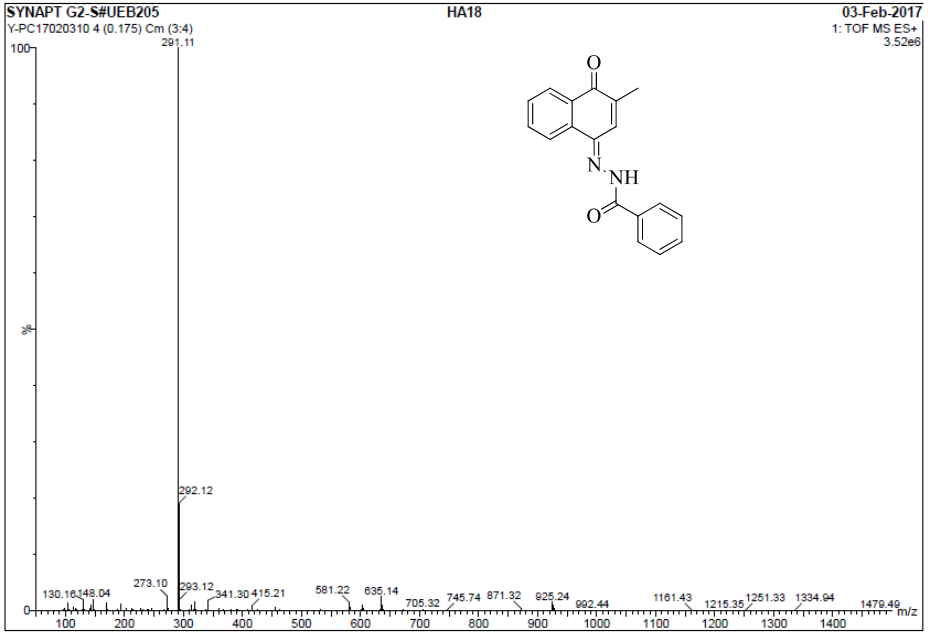


Figure S37. LC-MS spectrum of compound **6g**

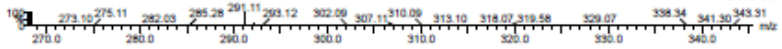


Elemental Composition Report Page 1

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

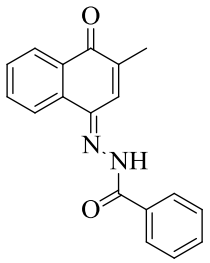
Monoisotopic Mass, Even Electron Ions
561 formula(e) evaluated with 3 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 0-100 H: 0-100 N: 0-20 O: 0-20

SYNAPT G2-S#UEB205 HA18 03-Feb-2017
Y-PC17020310 4 (0.175) Cm (3:4) 1: TOF MS ES+
3.53e+006



Minimum:				-1.5				
Maximum:		1.0	1.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
291.1132	291.1134	-0.2	-0.7	12.5	2039.8	0.000	100.00	C18 H15 N2 O2
	291.1139	-0.7	-2.4	5.5	2051.4	11.518	0.00	C3 H11 N14 O3
	291.1125	0.7	2.4	0.5	2052.3	12.426	0.00	C2 H15 N10 O7

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

Figure S39. Elemental analysis of compound **6g**

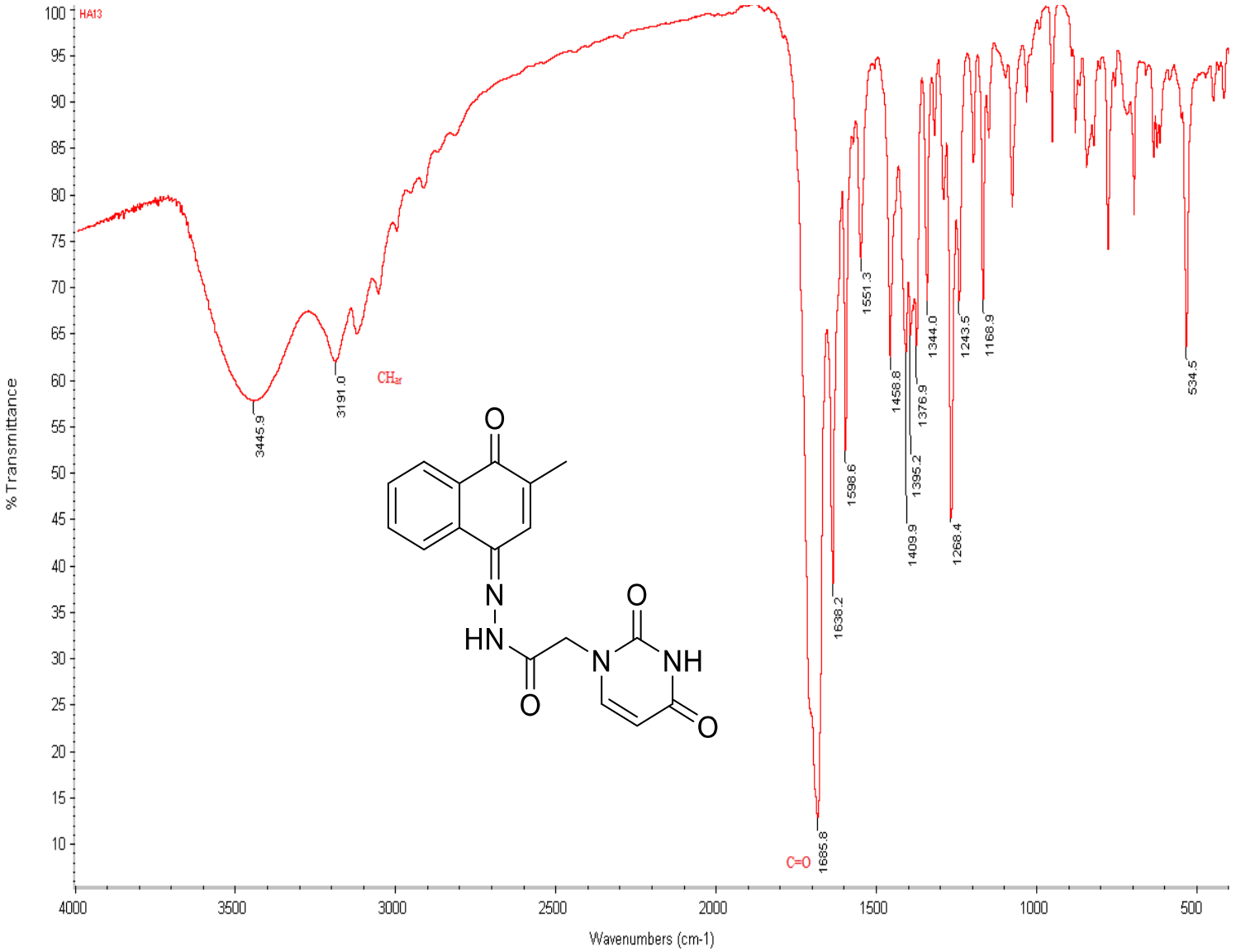


Figure S40. IR spectrum of compound **6h**

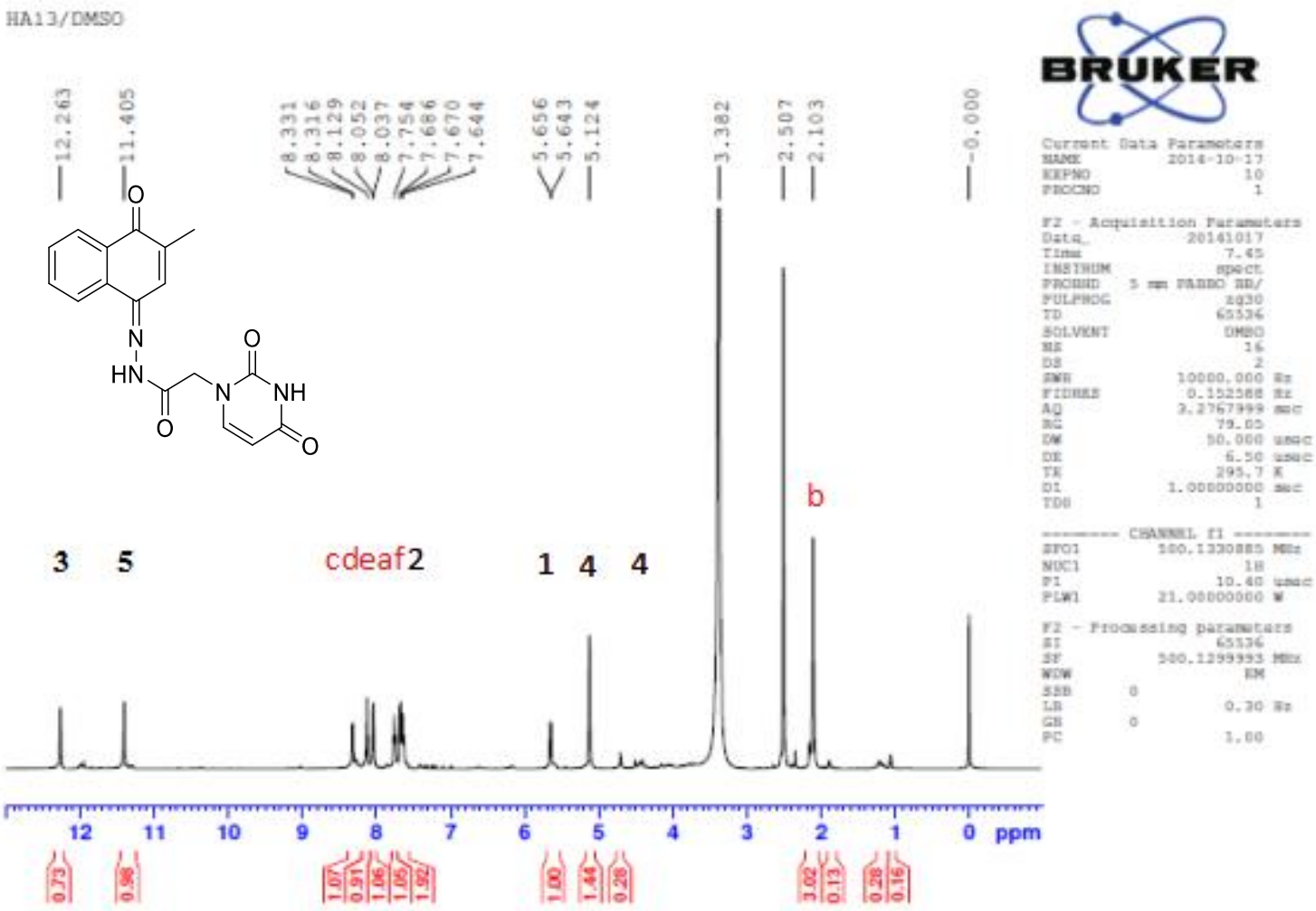


Figure S41. ^{1}H NMR (DMSO, 500 MHz) spectrum of compound **6h**

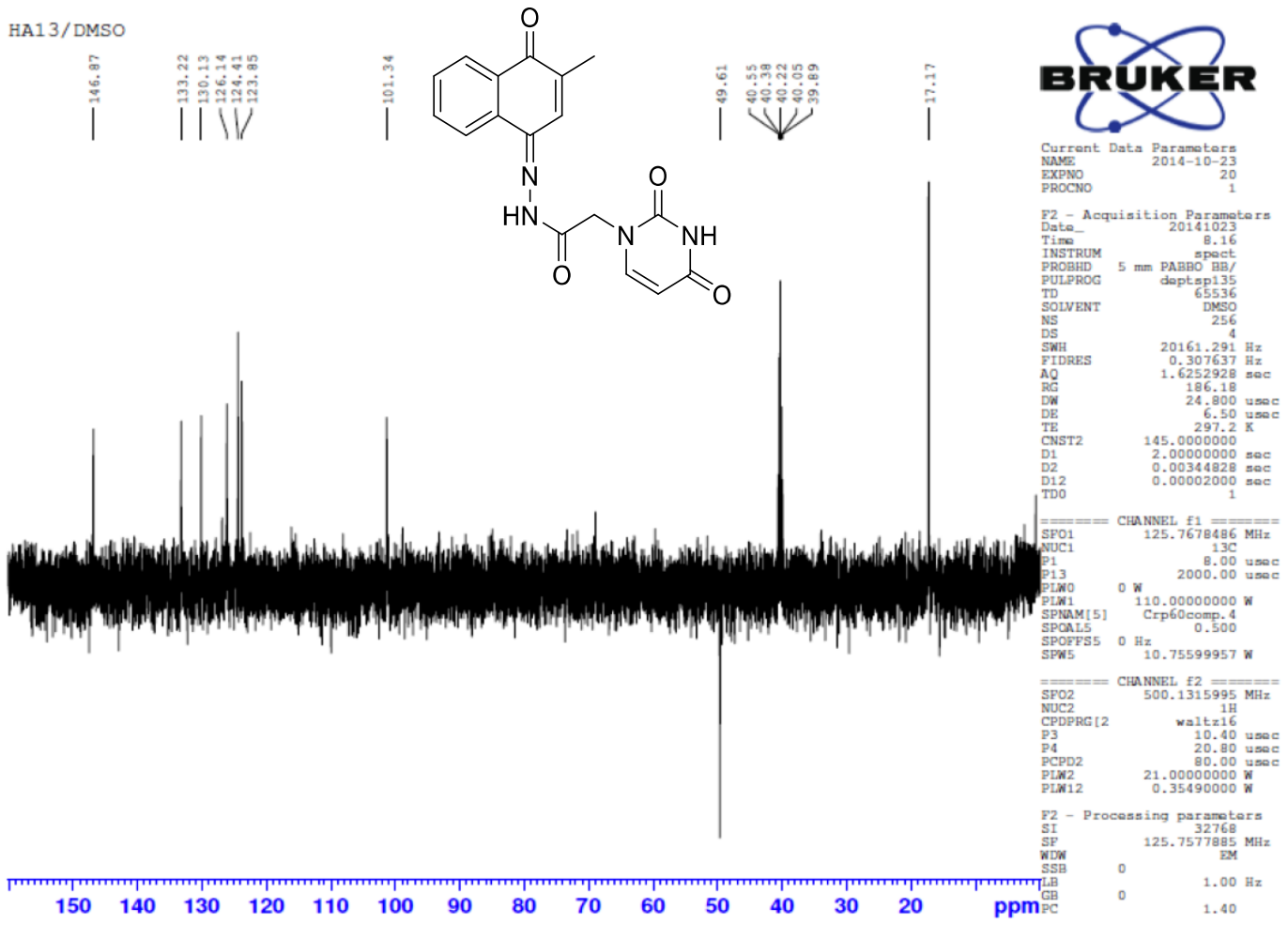


Figure S42. DEPT NMR (DMSO, 100 MHz) spectrum of compound **6h**

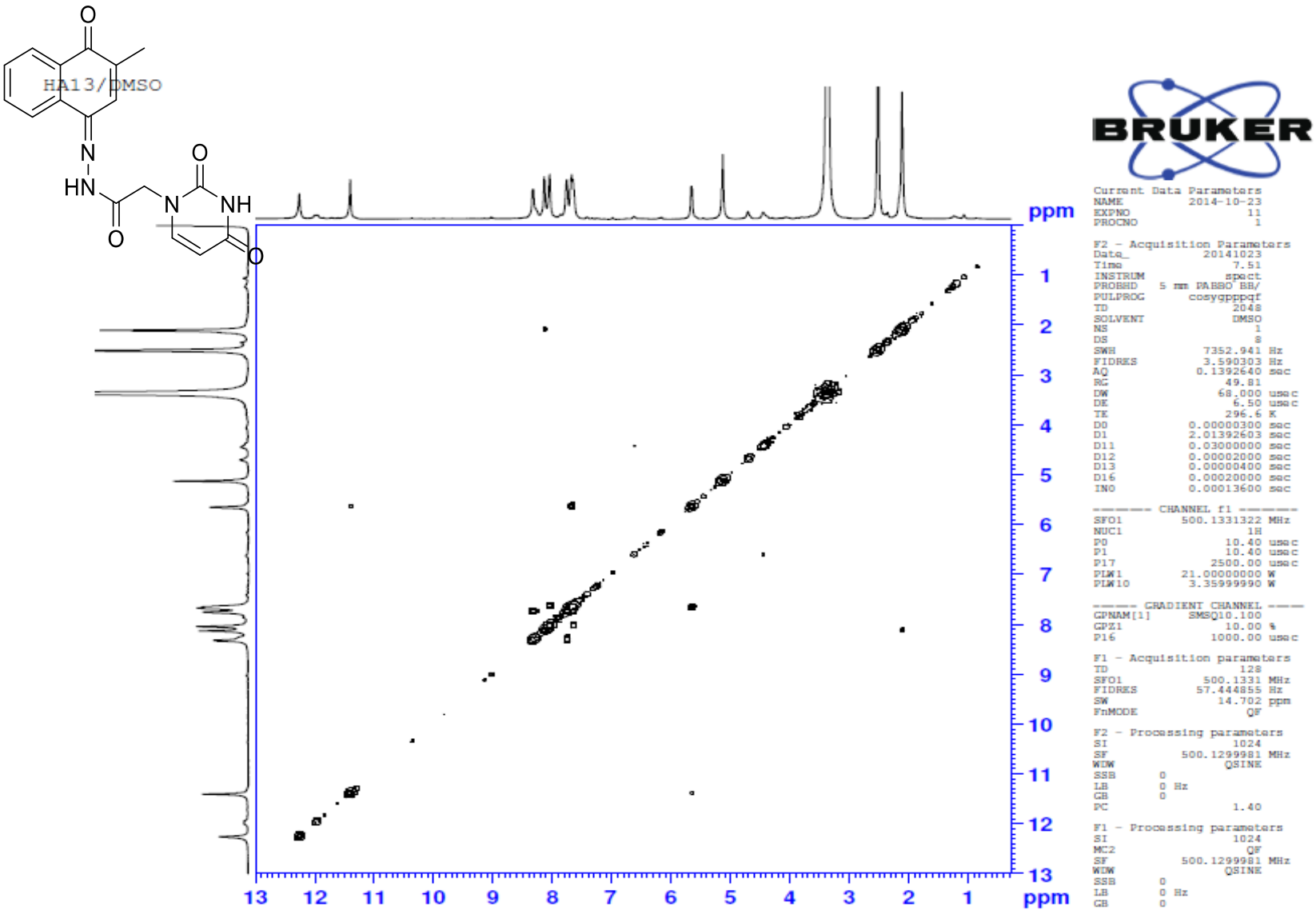
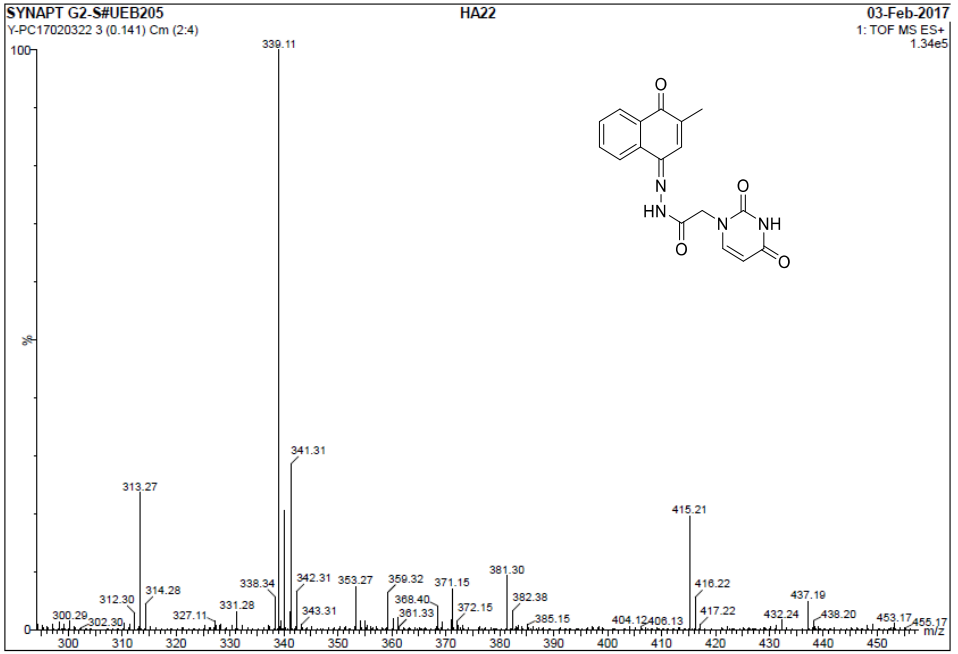


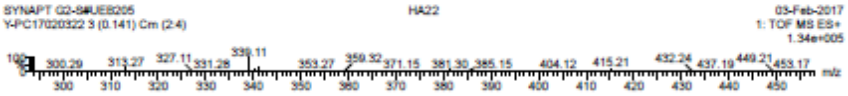
Figure S43. COSY 2D-NMR spectrum of compound **6h**



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



Minimum:				-1.5				
Maximum:		1.0	1.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
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	5.5	1207.8	16.603	0.00	C2 H11 N16 O5
	339.1085	0.9	2.7	0.5	1208.7	17.521	0.00	C H15 N12 O9

Figure S44. HRMS spectrum of compound **6h**

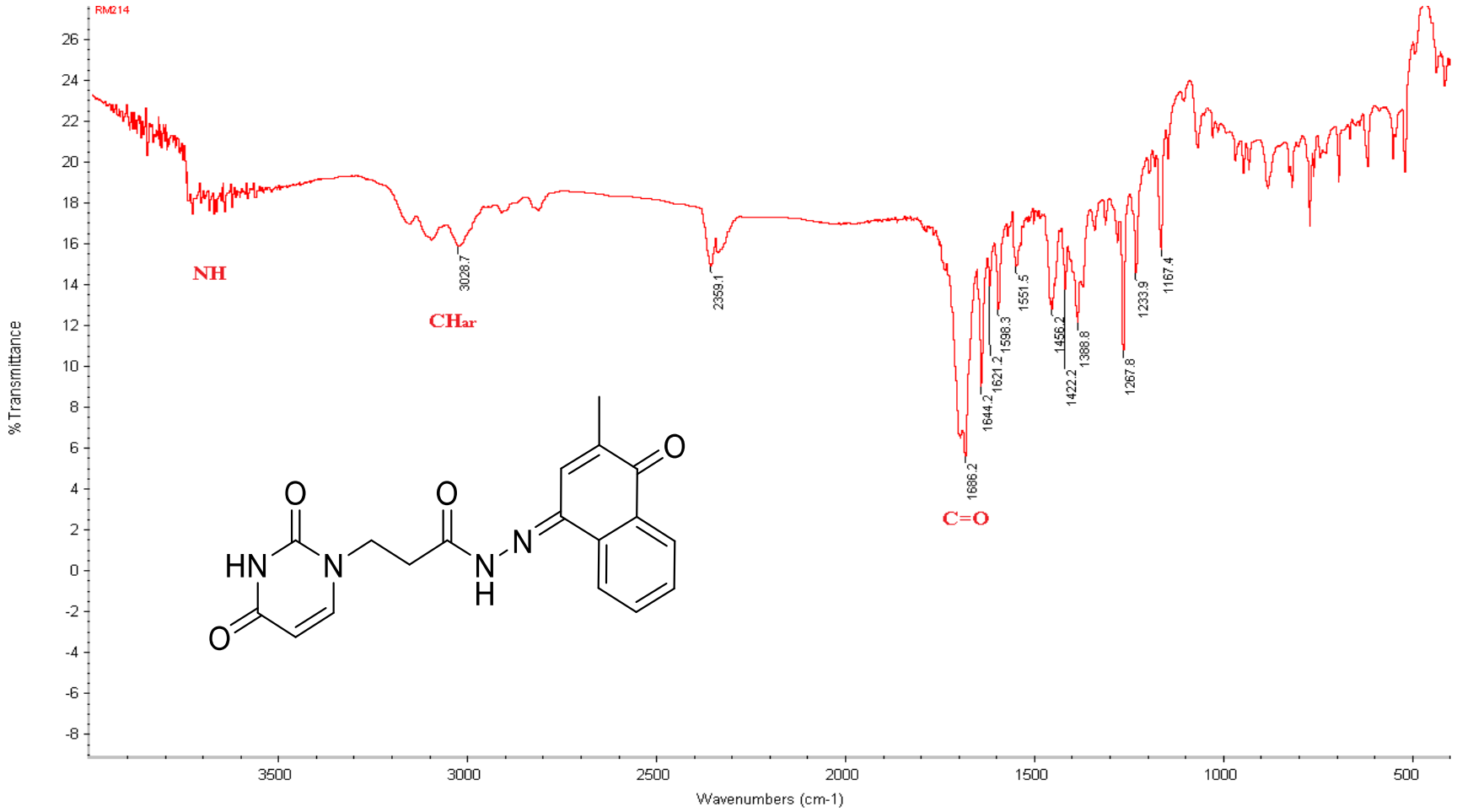


Figure S45. IR spectrum of compound **6i**

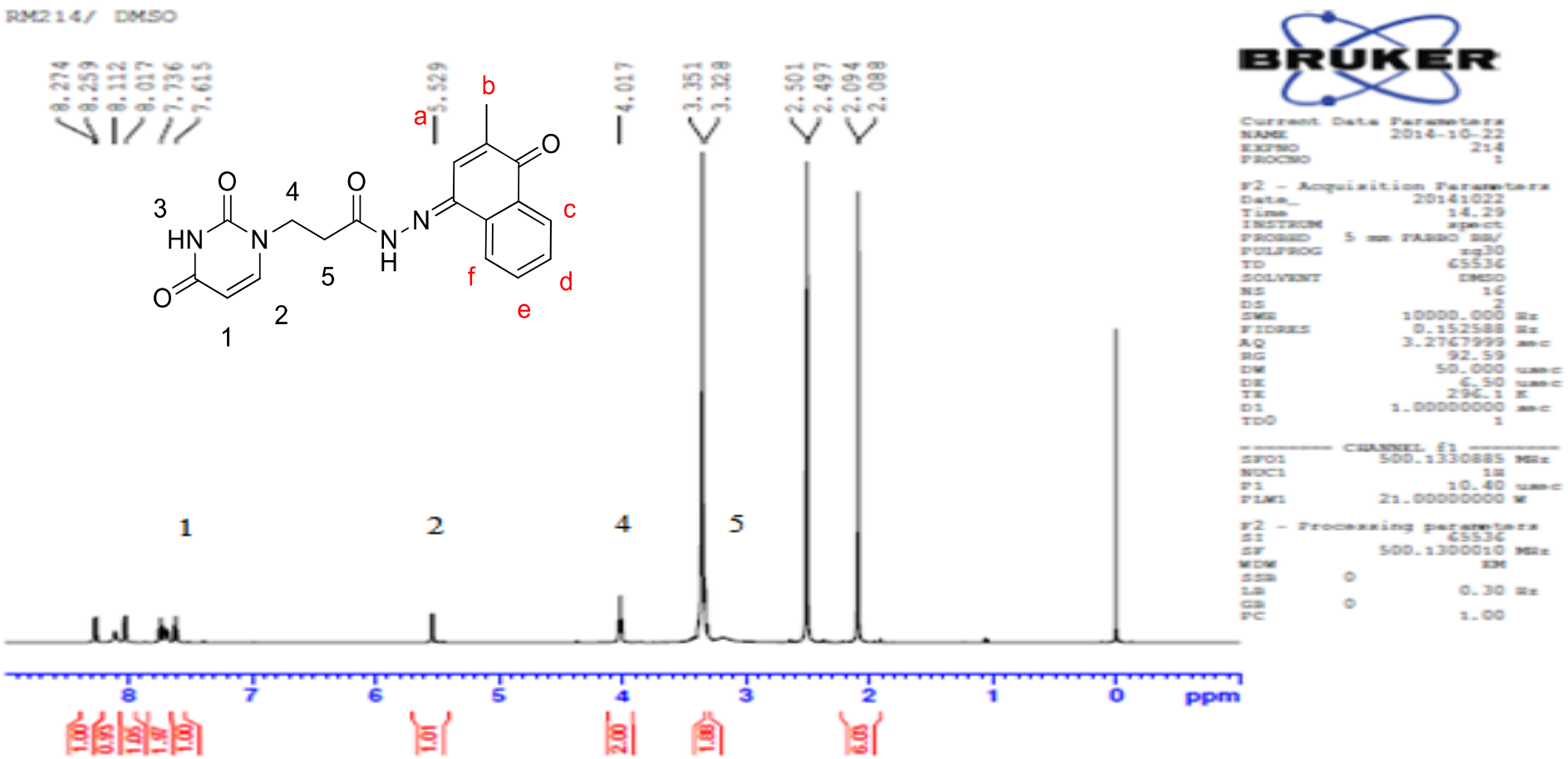


Figure S46. [1]H NMR (DMSO, 500 MHz) spectrum of compound **6i**

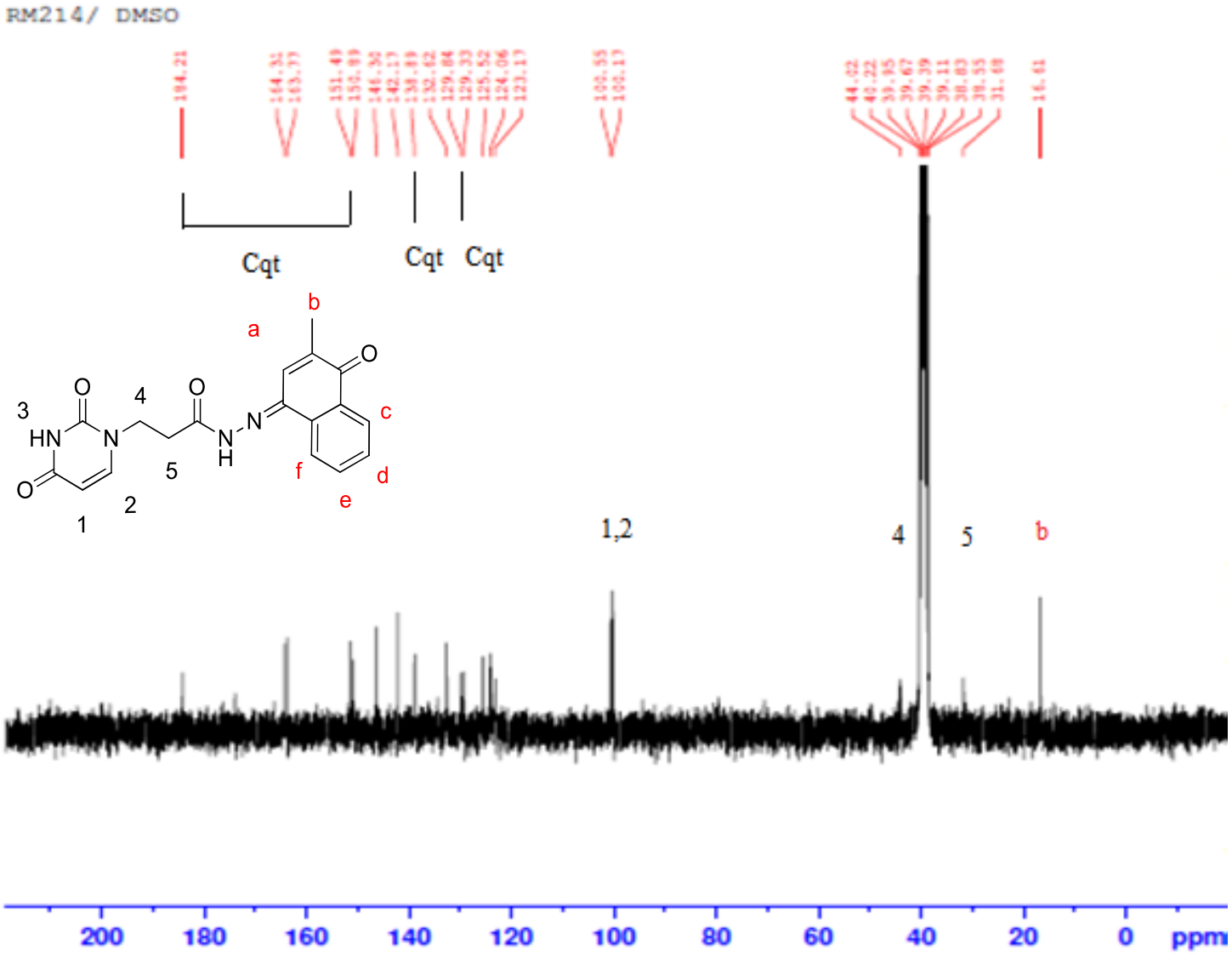
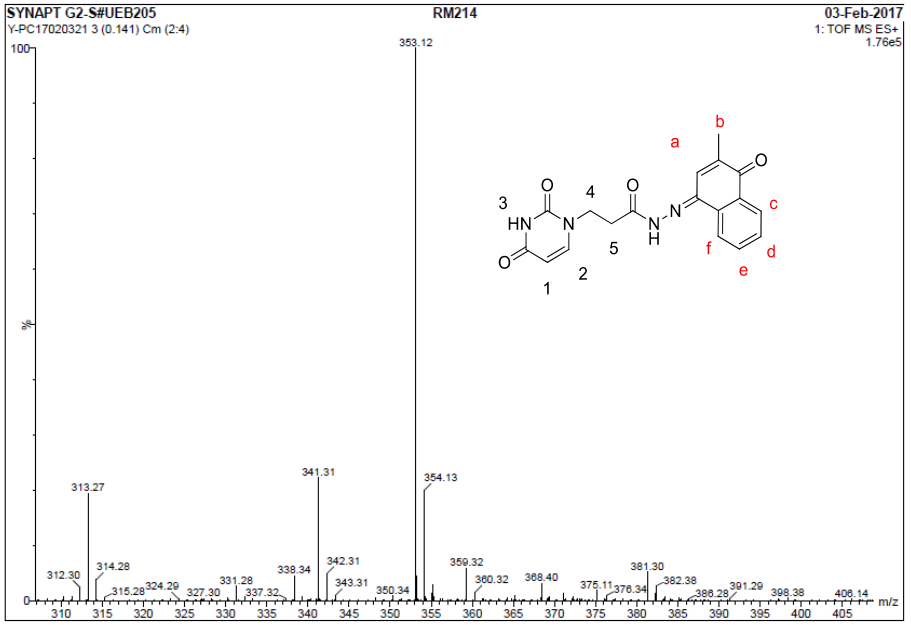


Figure S47. ^{13}C NMR (DMSO, 100 MHz) spectrum of compound **6i**

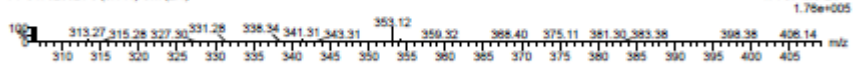


Elemental Composition Report **Page 1**

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
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-S#UEB205 RM214 03-Feb-2017
Y-PC17020321 3 (0.141) Cm (2:4) 1: TOF MS ES+ 1.76e+005



Minimum:				-1.5				
Maximum:		1.0	1.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
353.1249	353.1250	-0.1	-0.3	12.5	1123.2	0.000	100.00	C18 H17 N4 O4
	353.1255	-0.6	-1.7	5.5	1137.1	13.978	0.00	C3 H13 N16 O5
	353.1241	0.8	2.3	0.5	1137.9	14.793	0.00	C2 H17 N12 O9

Figure S48. HRMS spectrum of compound **6i**

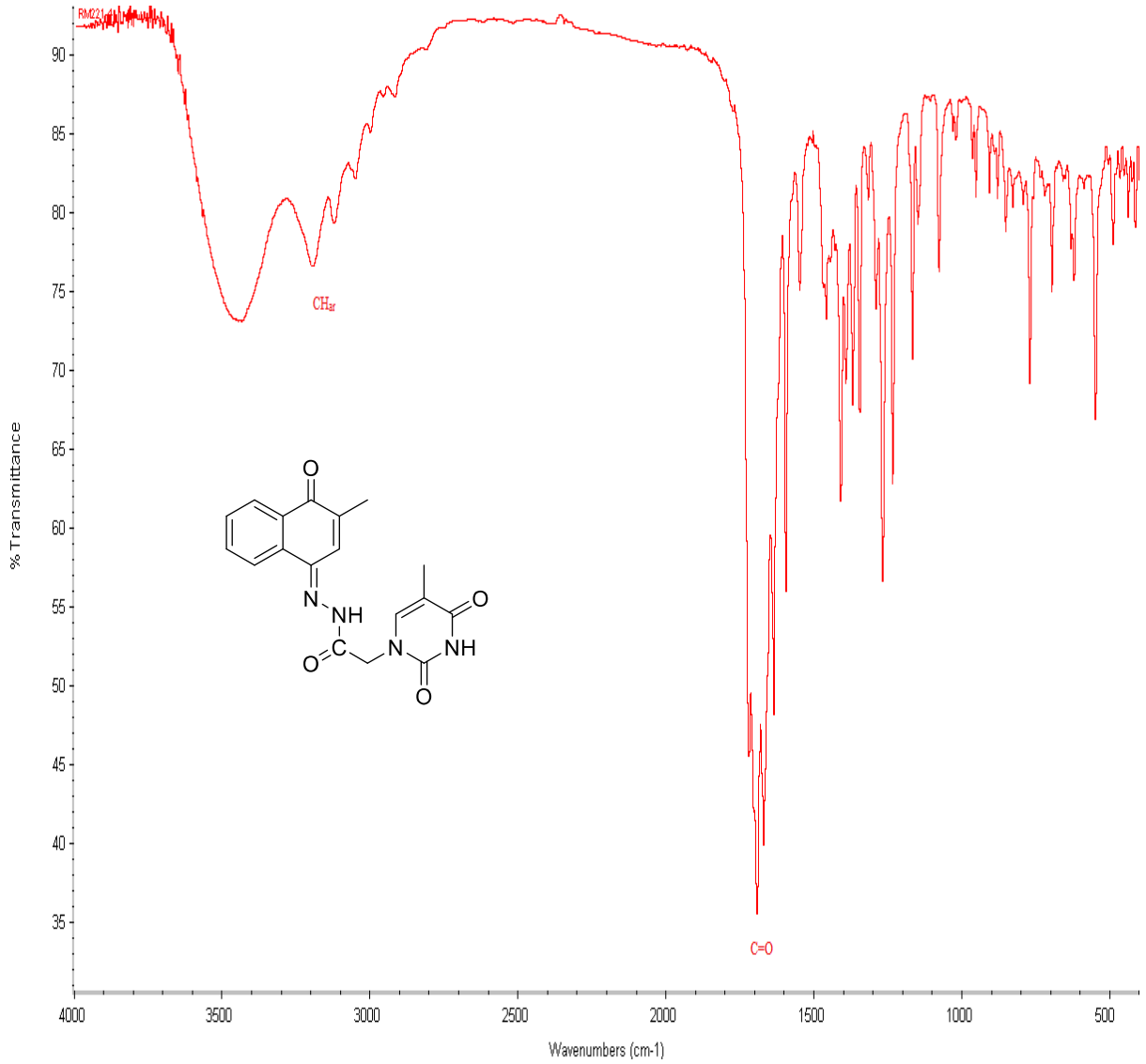


Figure S49. IR spectrum of compound **6j**

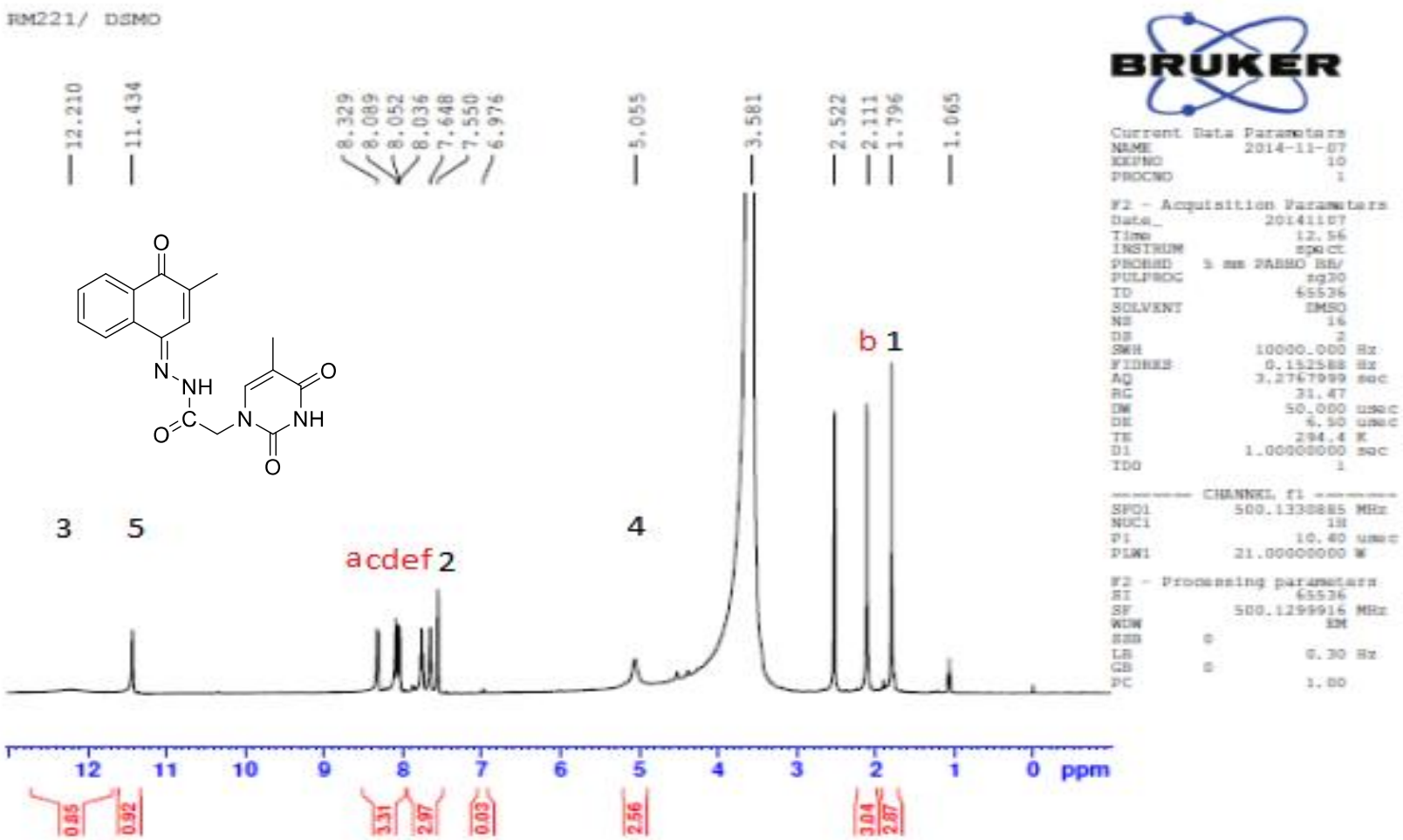


Figure S50. ^{1}H NMR (DMSO, 500 MHz) spectrum of compound **6j**

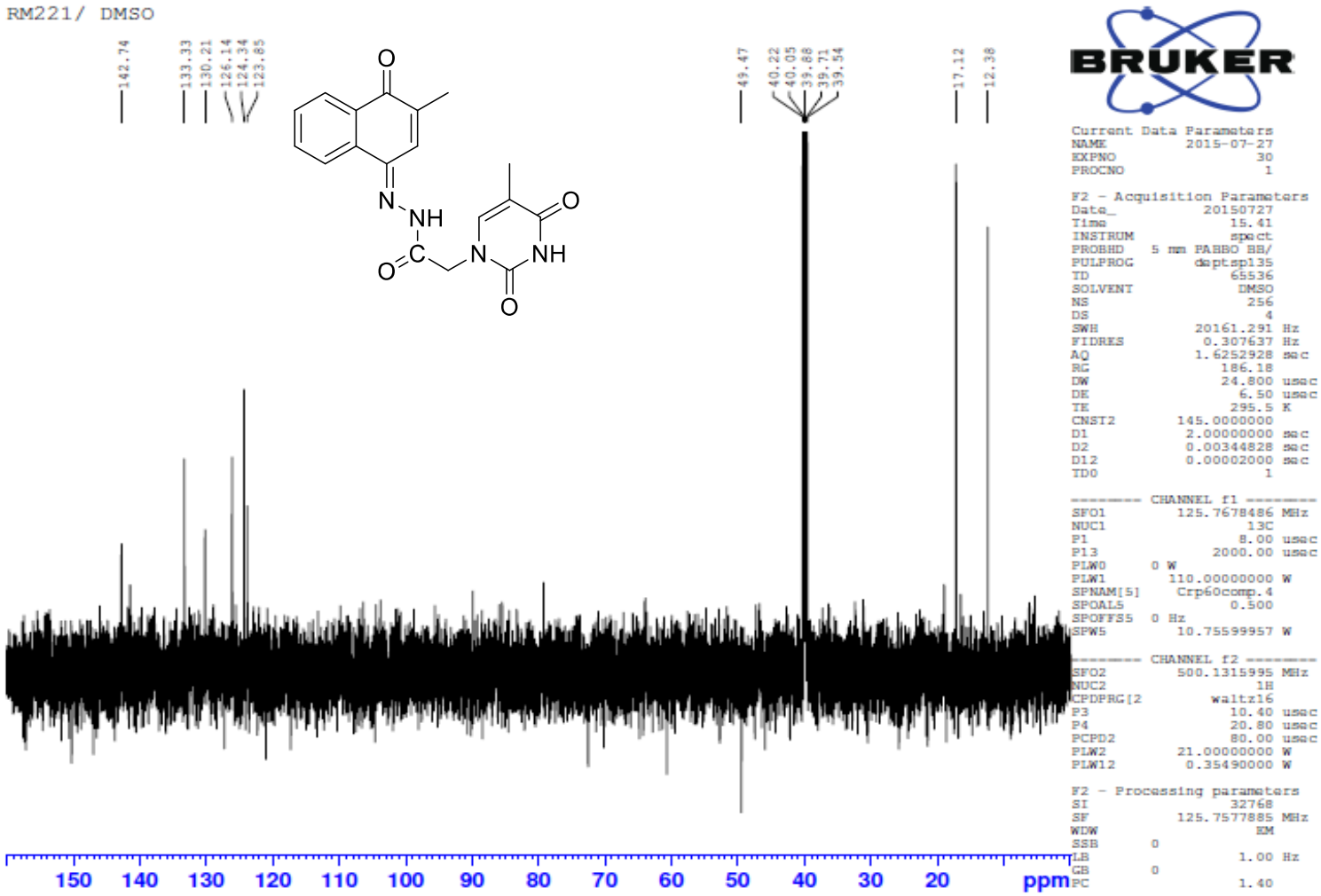


Figure S51. DEPT NMR (DMSO, 100 MHz) spectrum of compound **6j**

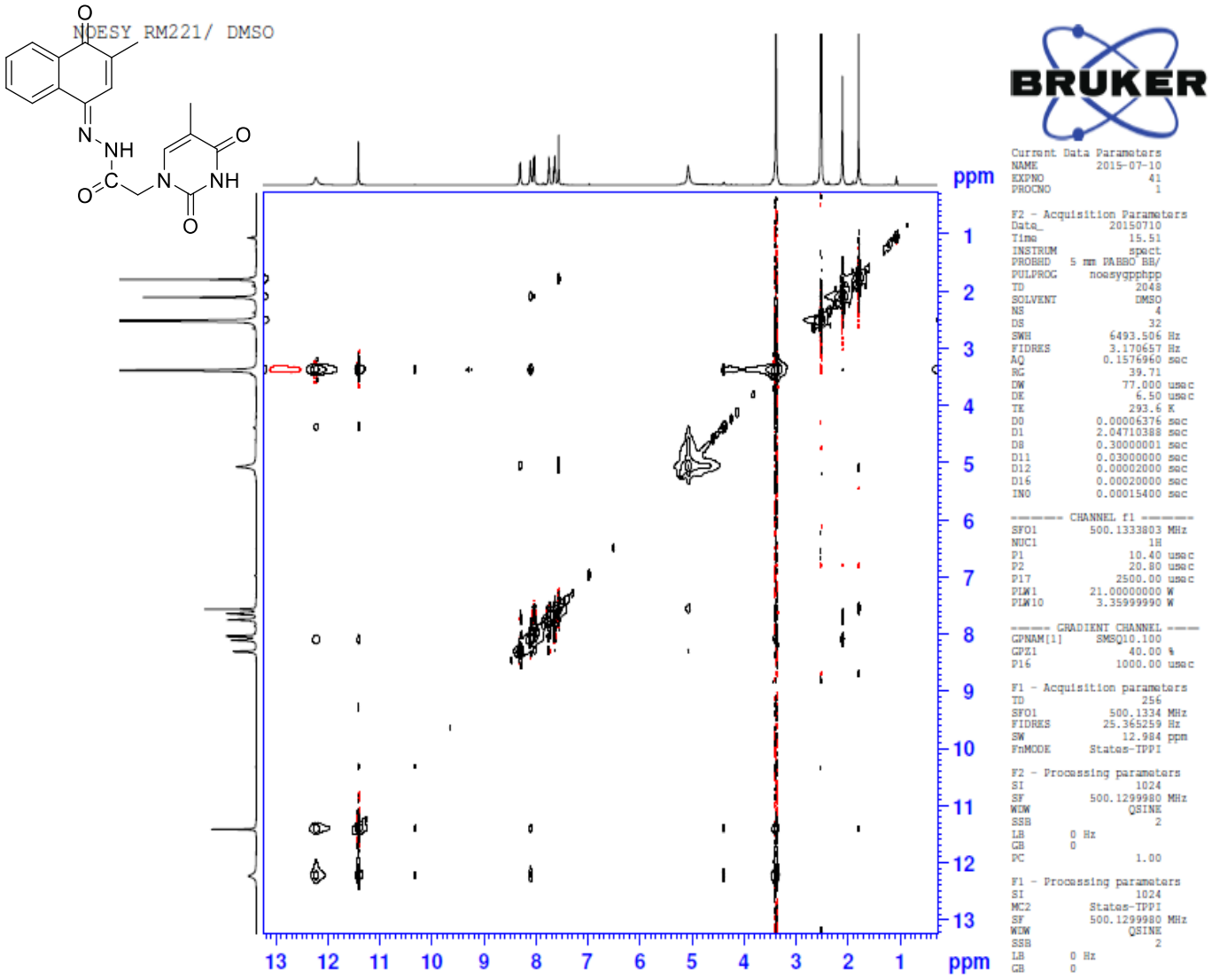
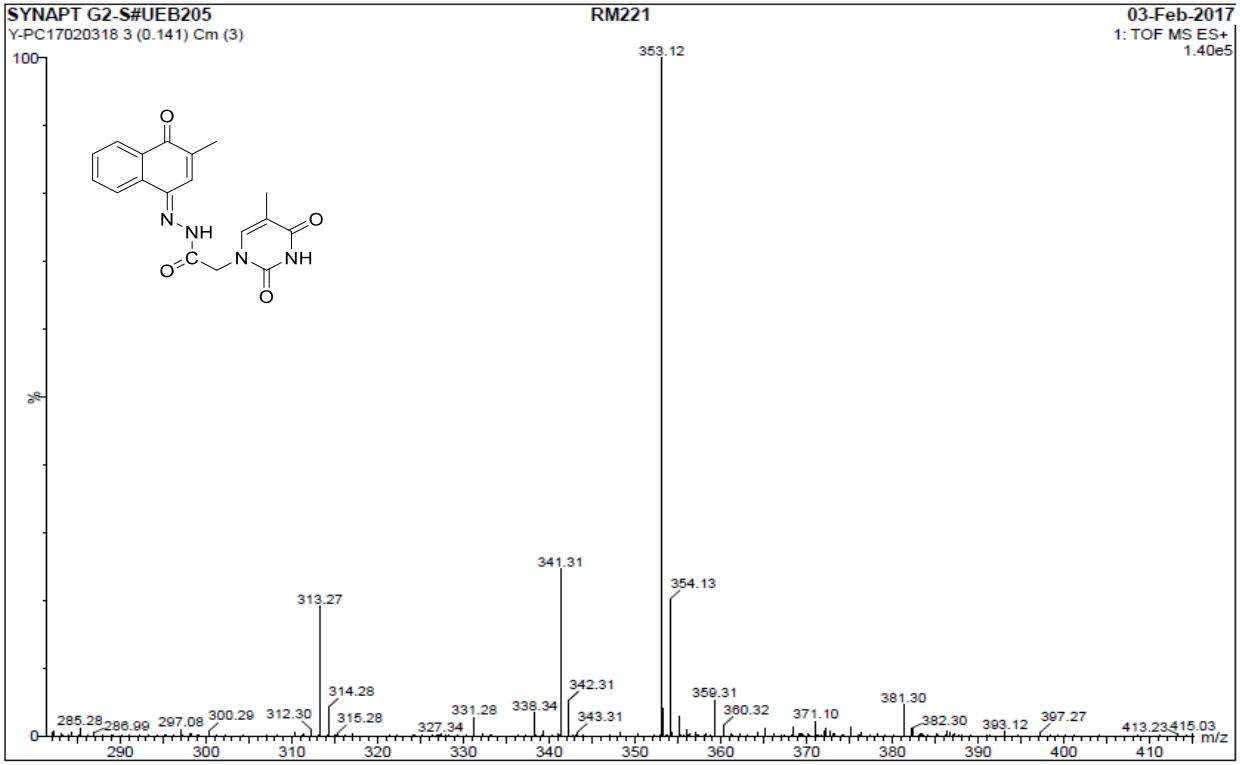
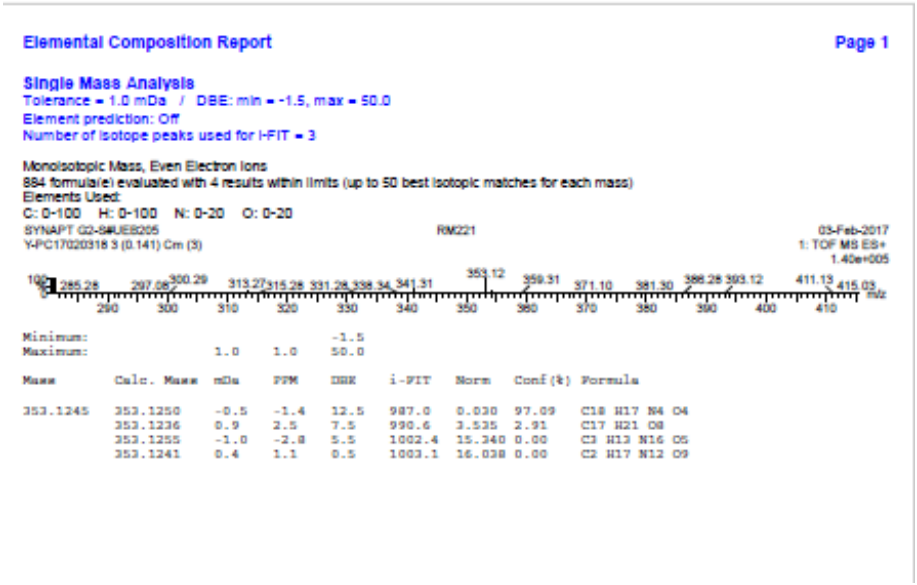


Figure S52. NOESY 2D-NMR spectrum of compound **6j**


SYNAPT G2-S#UEB205
RM221
03-Feb-2017
Y-PC17020318 3 (0.141) Cm (3)
1: TOF MS ES+
1.40e5
100
%
0
353.12
341.31
313.27
354.13
314.28
342.31
359.31
360.32
381.30
331.28
338.34
343.31
371.10
382.30
393.12
397.27
285.28
286.99
297.08
300.29
312.30
315.28
327.34
413.23
415.03
290
300
310
320
330
340
350
360
370
380
390
400
410
m/z



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

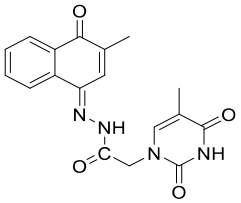
Monoisotopic Mass, Even Electron Ions
884 formula(e) evaluated with 4 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 0-100 H: 0-100 N: 0-20 O: 0-20

SYNAPT G2-S#UES205 RM221 03-Feb-2017
Y-PC17020318 3 (0.141) Cm (3) 1: TOF MS ES+
1.40e+005

285.28 297.08 300.29 313.27 315.28 331.28 338.34 341.31 353.12 359.31 371.10 381.30 386.28 393.12 411.13 415.03 m/z

Minimum:				-1.5				
Maximum:		1.0	1.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
353.1245	353.1250	-0.5	-1.4	12.5	987.0	0.030	97.09	C18 H17 N4 O4
	353.1236	0.9	2.5	7.5	990.6	3.535	2.91	C17 H21 O8
	353.1255	-1.0	-2.8	5.5	1002.4	15.340	0.00	C3 H13 N16 O5
	353.1241	0.4	1.1	0.5	1003.1	16.038	0.00	C2 H17 N12 O9

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**

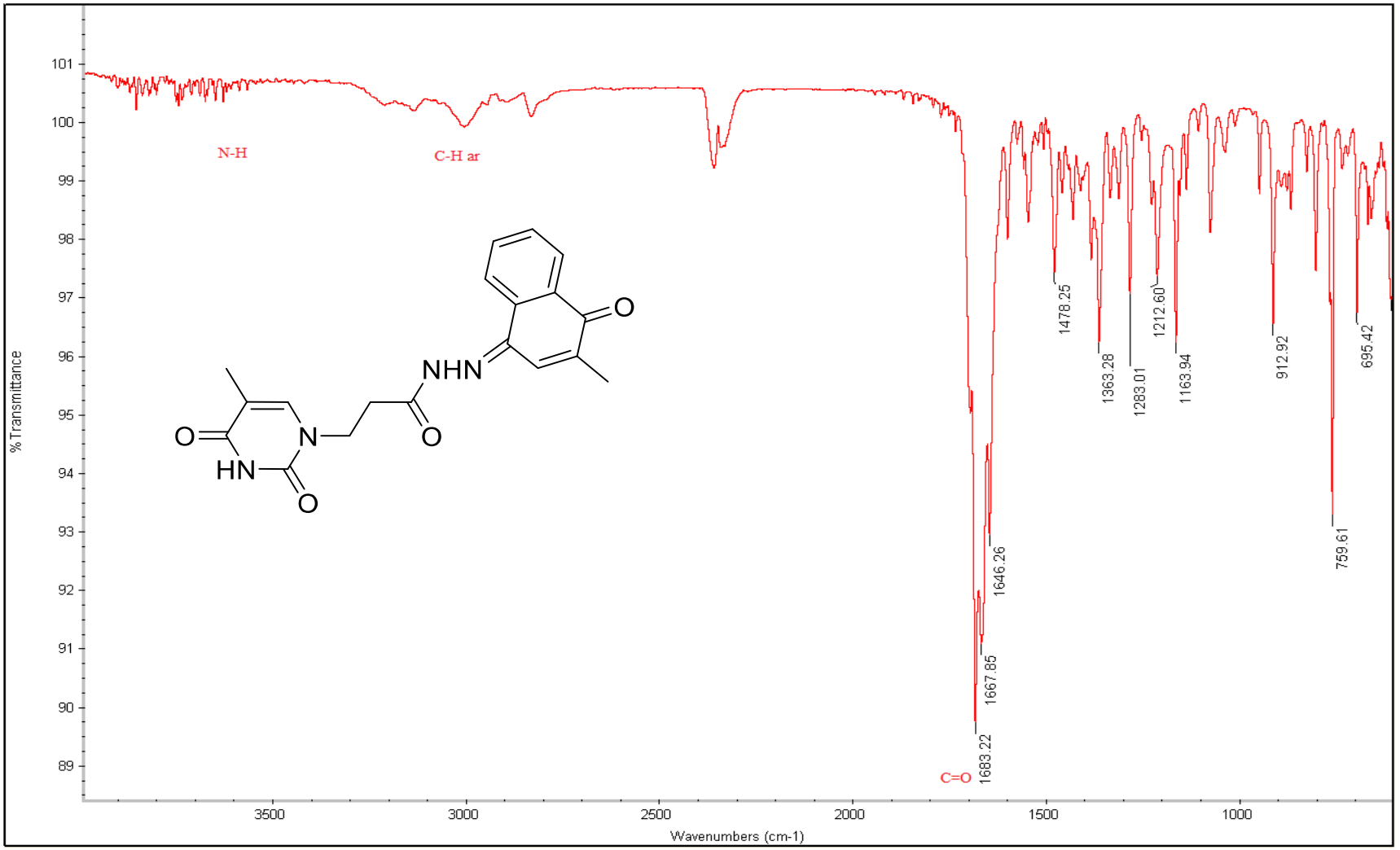


Figure S55. IR spectrum of compound **6k**

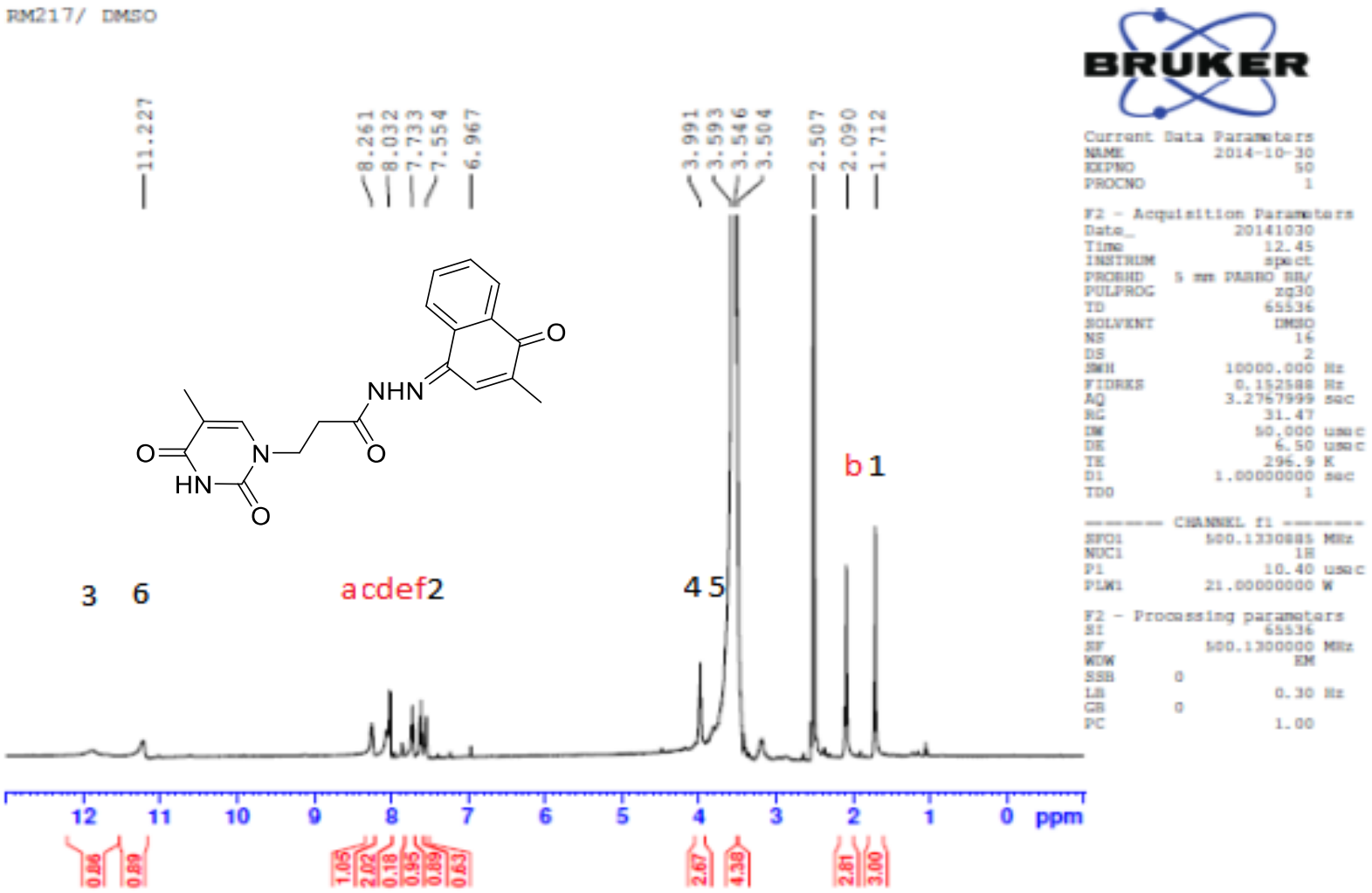


Figure S56. ^{1}H NMR (DMSO, 500 MHz) spectrum of compound **6k**

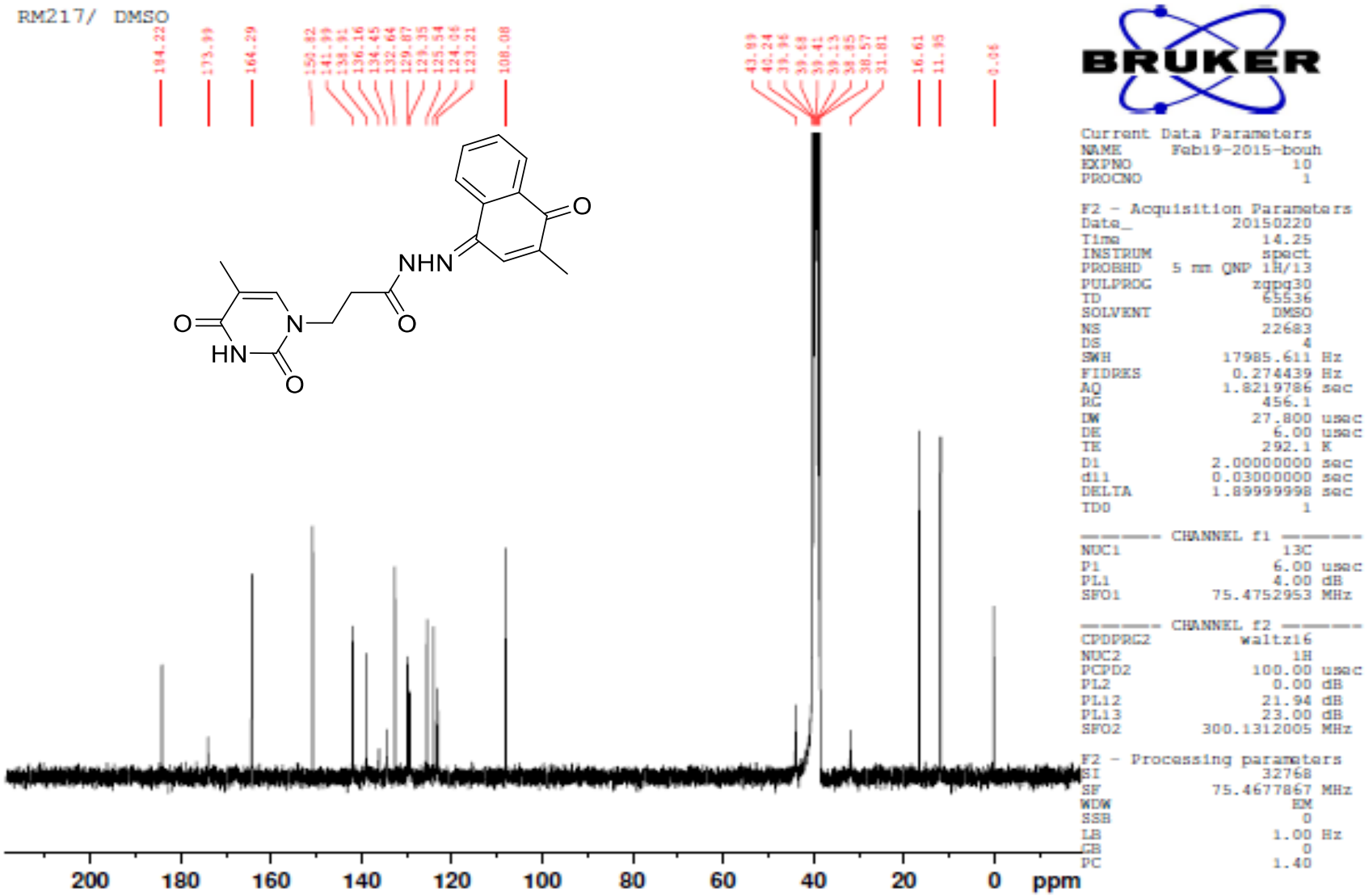


Figure S57. ^{13}C NMR (DMSO, 100 MHz) spectrum of compound **6k**

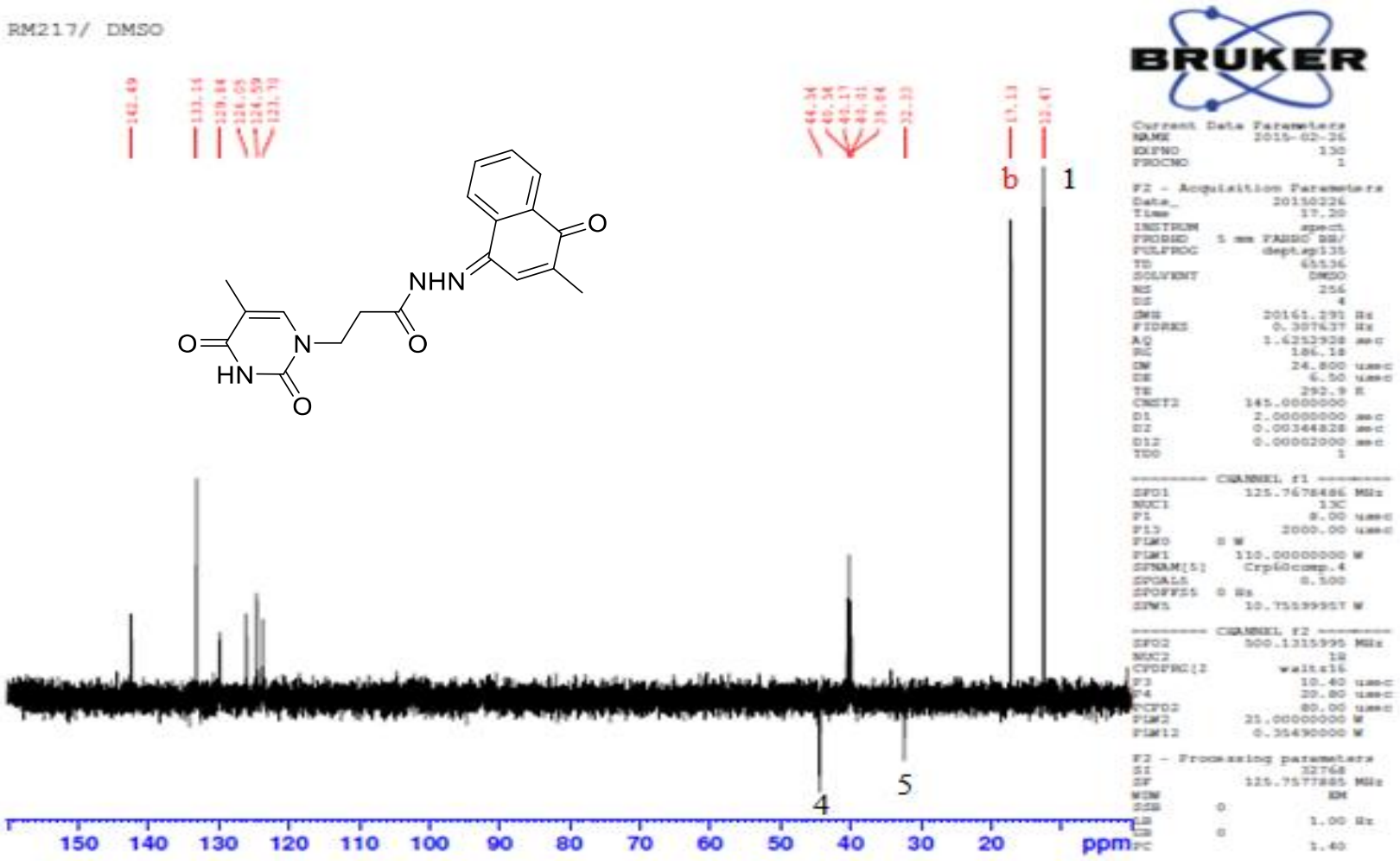


Figure S58. DEPT NMR (DMSO, 100 MHz) spectrum of compound **6k**

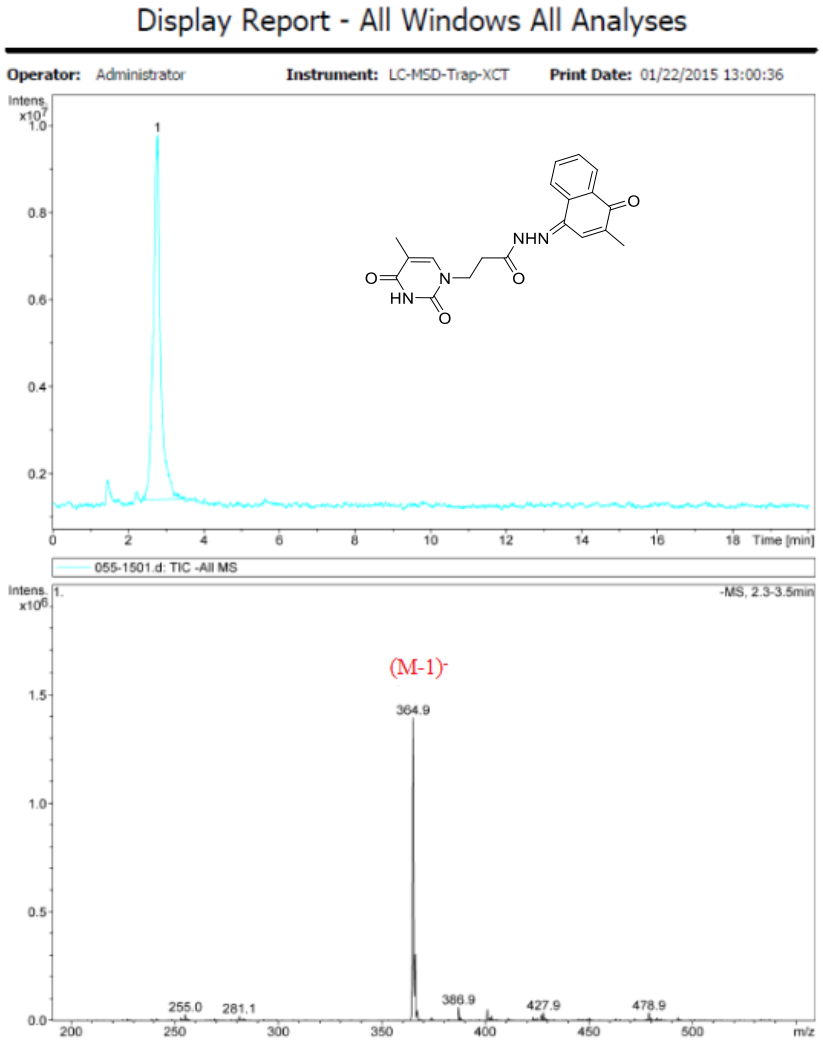
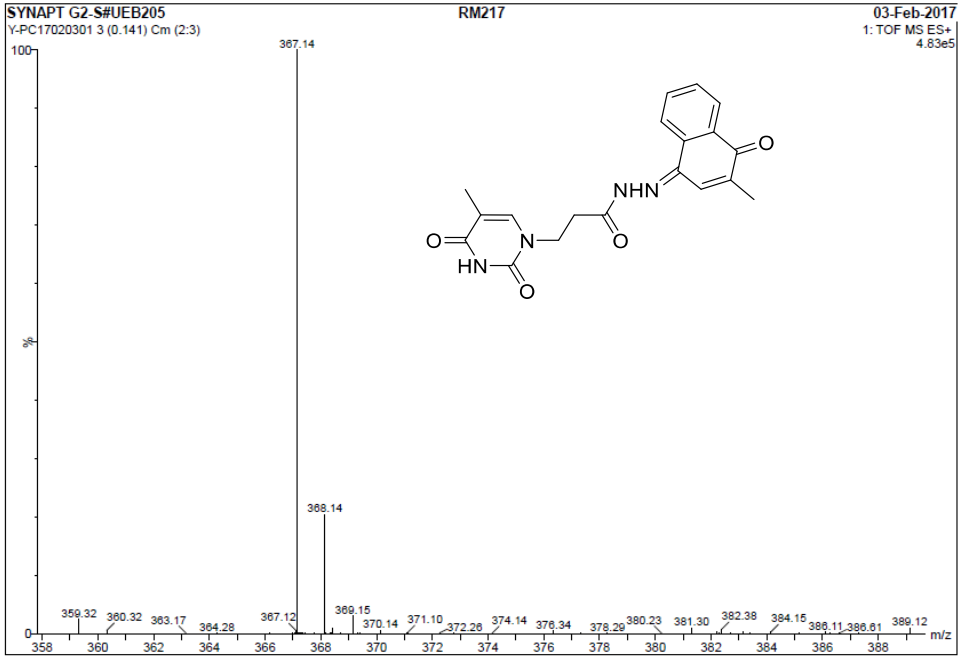


Figure S59. LC-MS spectrum of compound **6k**



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
964 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-S#UEB205 RM217 03-Feb-2017
Y-PC17020301 3 (0.141) Cm (2:3) 1: TOF MS ES+ 4.83e+005

Minimum:				-1.5				
Maximum:		1.0	1.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
367.1407	367.1406	0.1	0.3	12.5	1317.2	0.000	100.00	C19 H19 N4 O4
	367.1411	-0.4	-1.1	5.5	1329.2	12.047	0.00	C4 H15 N16 O5
	367.1398	0.9	2.5	0.5	1330.0	12.797	0.00	C3 H19 N12 O9

Figure S60. HRMS spectrum of compound **6k**

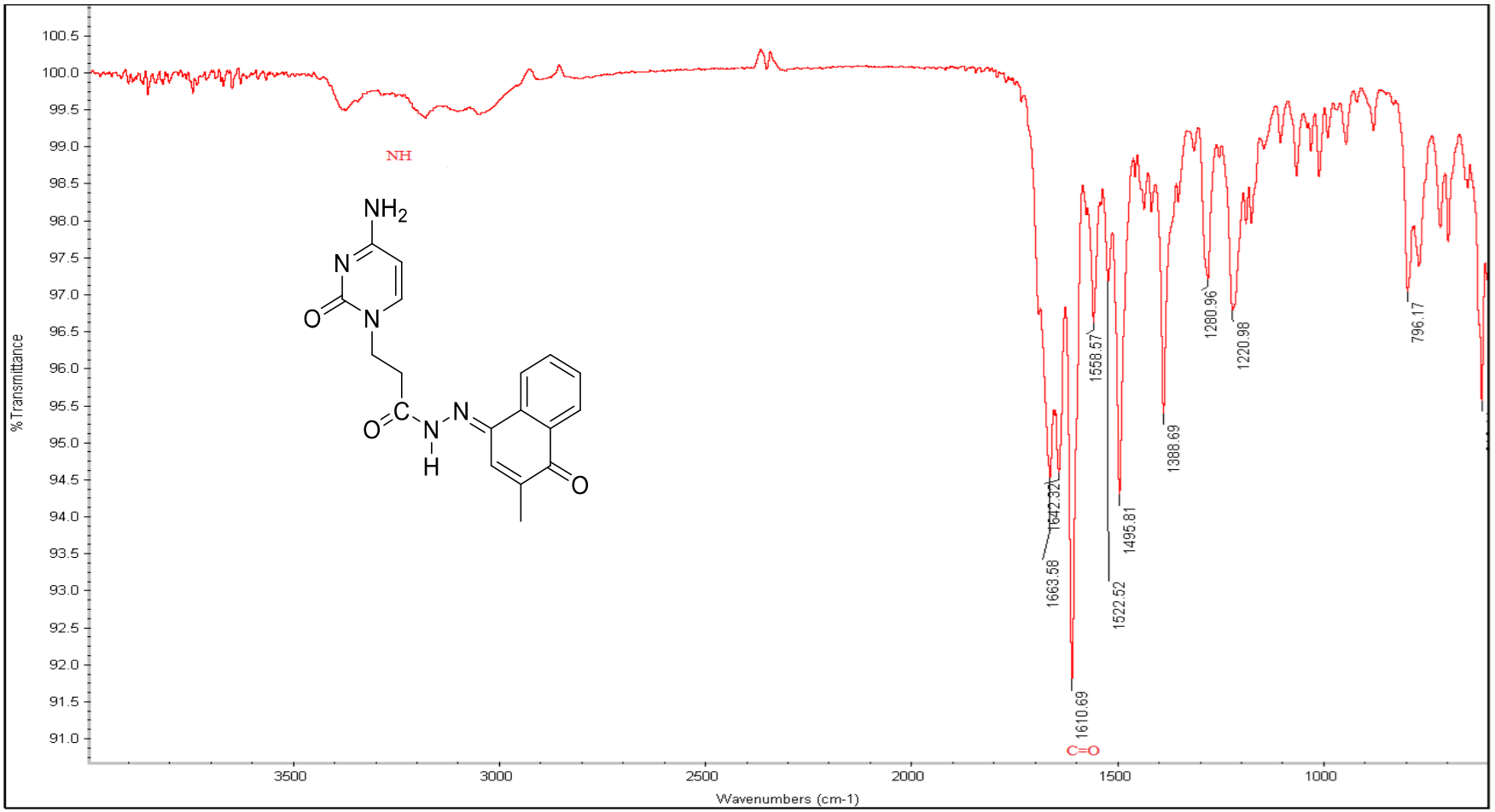


Figure S61. IR spectrum of compound **6l**

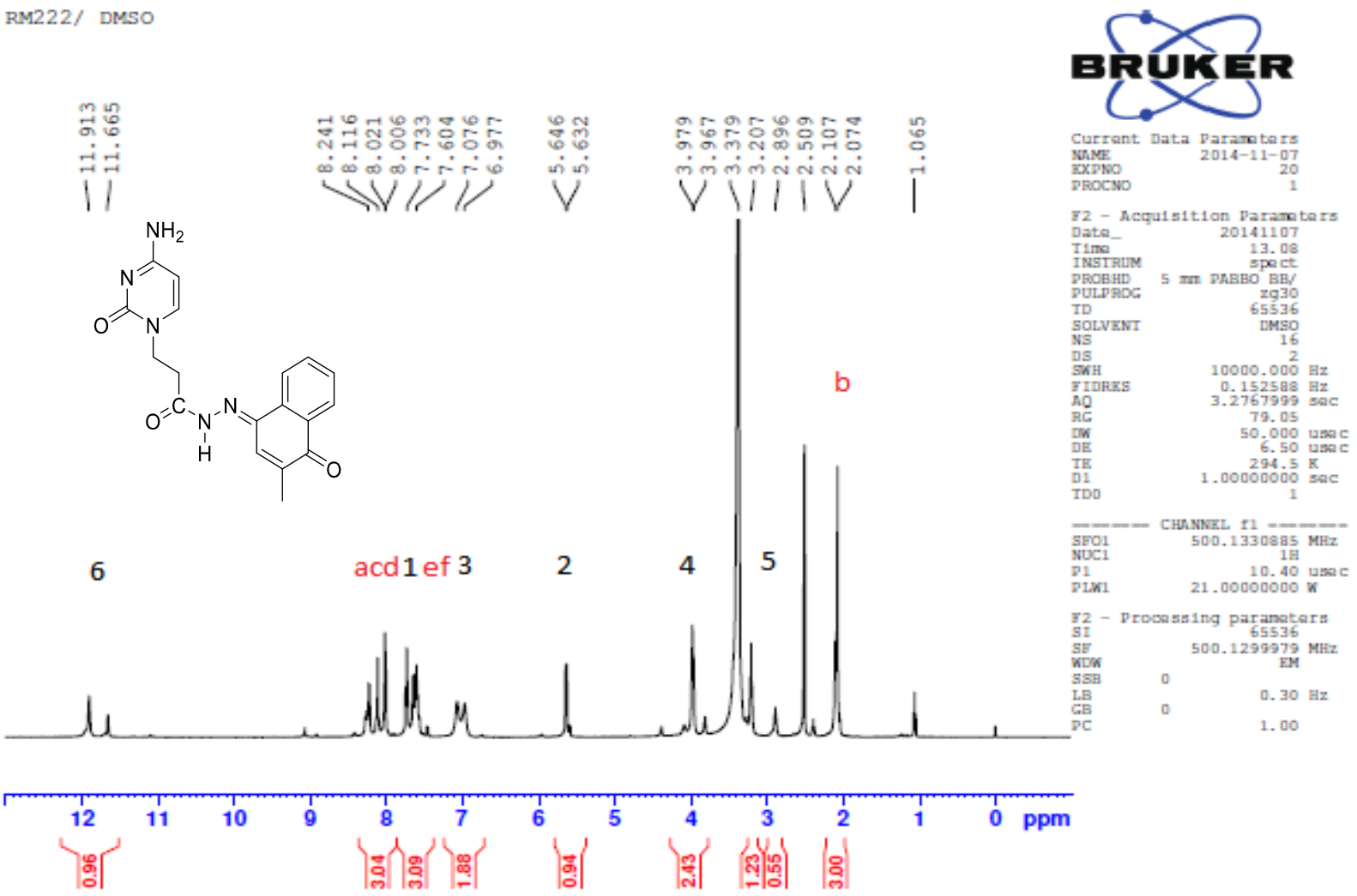


Figure S62. ^{1}H NMR (DMSO, 500 MHz) spectrum of compound **6l**

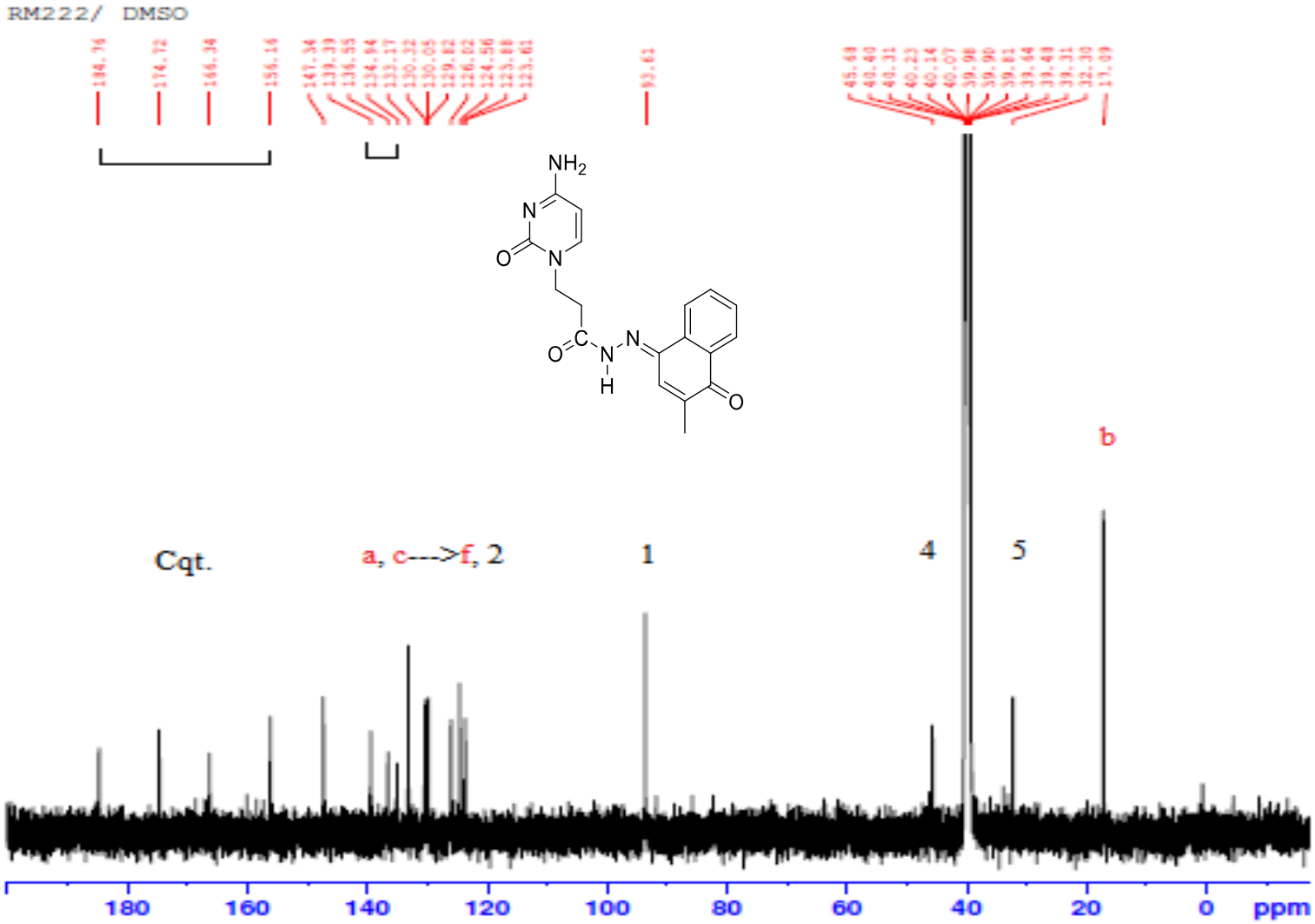


Figure S63. ^{13}C NMR (DMSO, 100 MHz) spectrum of compound **6l**

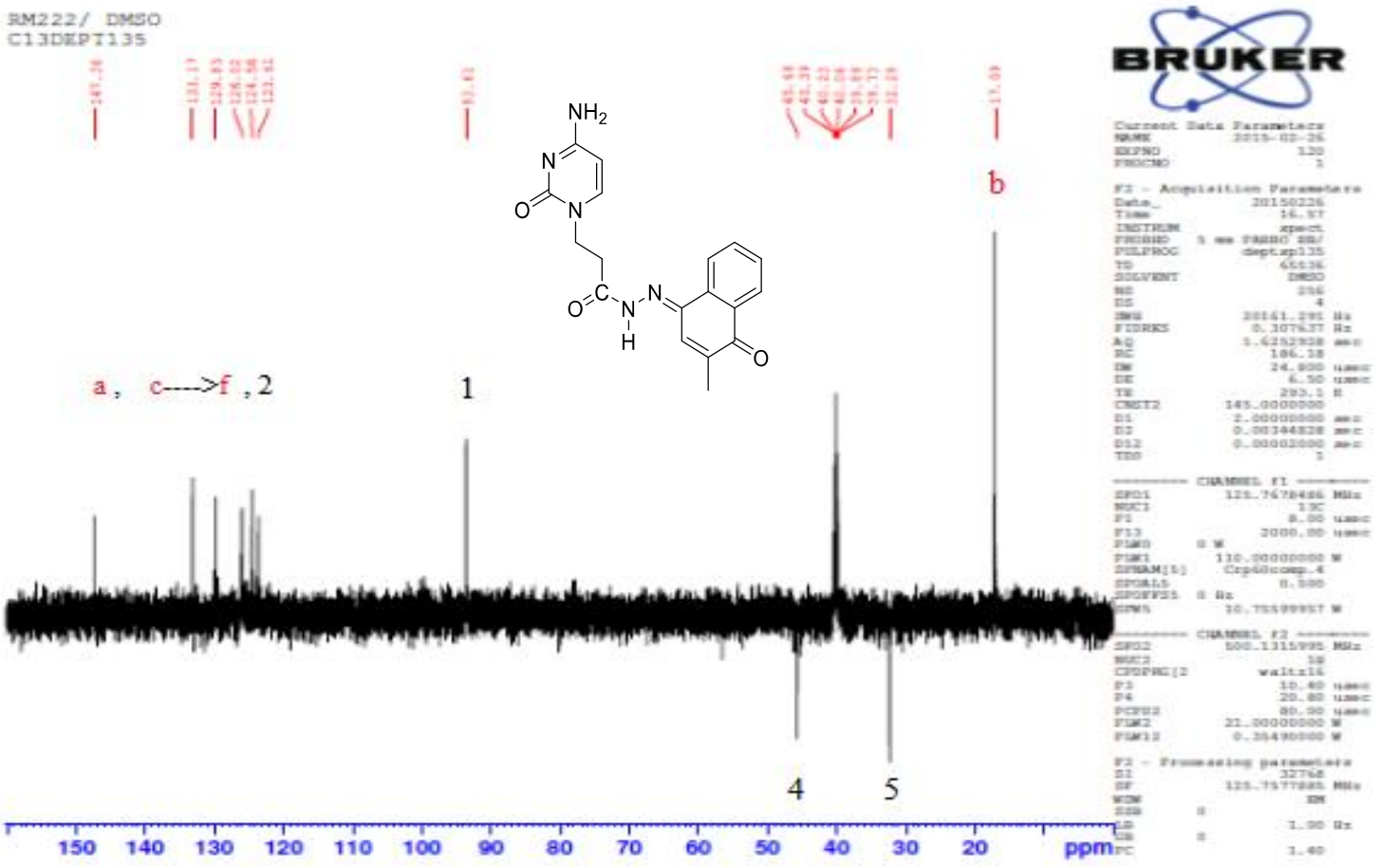
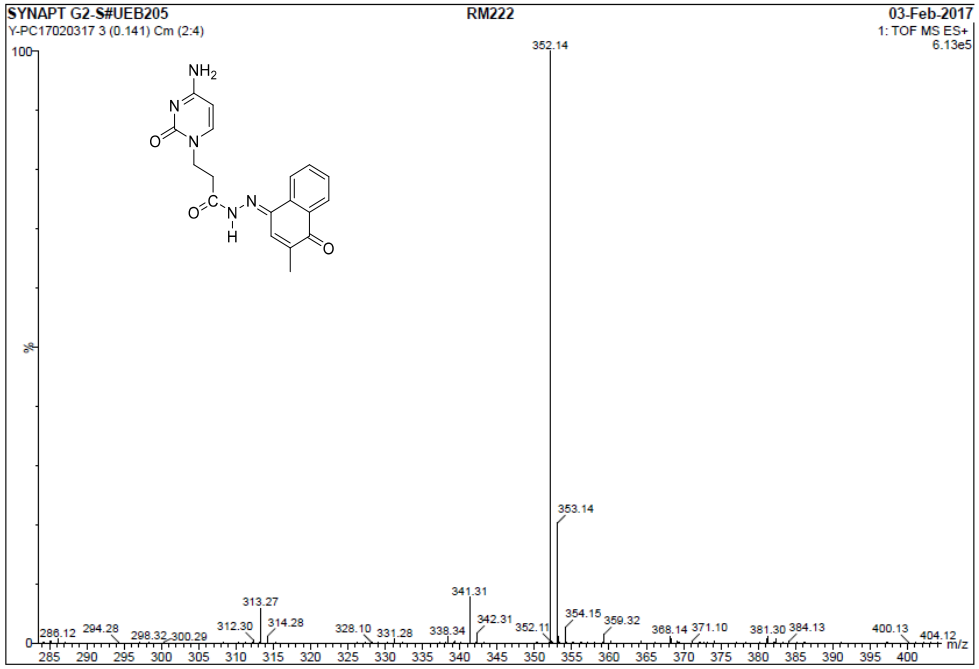
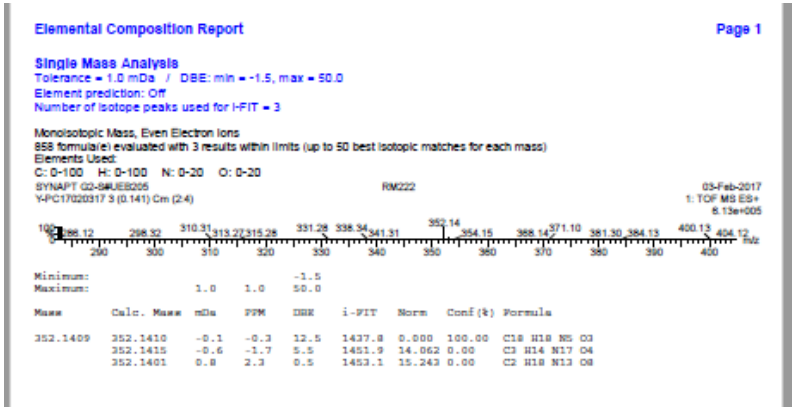


Figure S64. DEPT NMR (DMSO, 100 MHz) spectrum of compound **6l**





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
858 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-S#UEB205 RM222 03-Feb-2017
Y-PC17020317 3 (0.141) Cm (2:4) 1: TOF MS ES+ 6.13e+005

Minimum:				-1.5				
Maximum:		1.0	1.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
352.1409	352.1410	-0.1	-0.3	12.5	1437.8	0.000	100.00	C18 H18 N5 O3
	352.1415	-0.6	-1.7	5.5	1451.9	14.062	0.00	C3 H14 N17 O4
	352.1401	0.8	2.3	0.5	1453.1	15.243	0.00	C2 H18 N13 O8

Figure S65. HRMS spectrum of compound **6l**

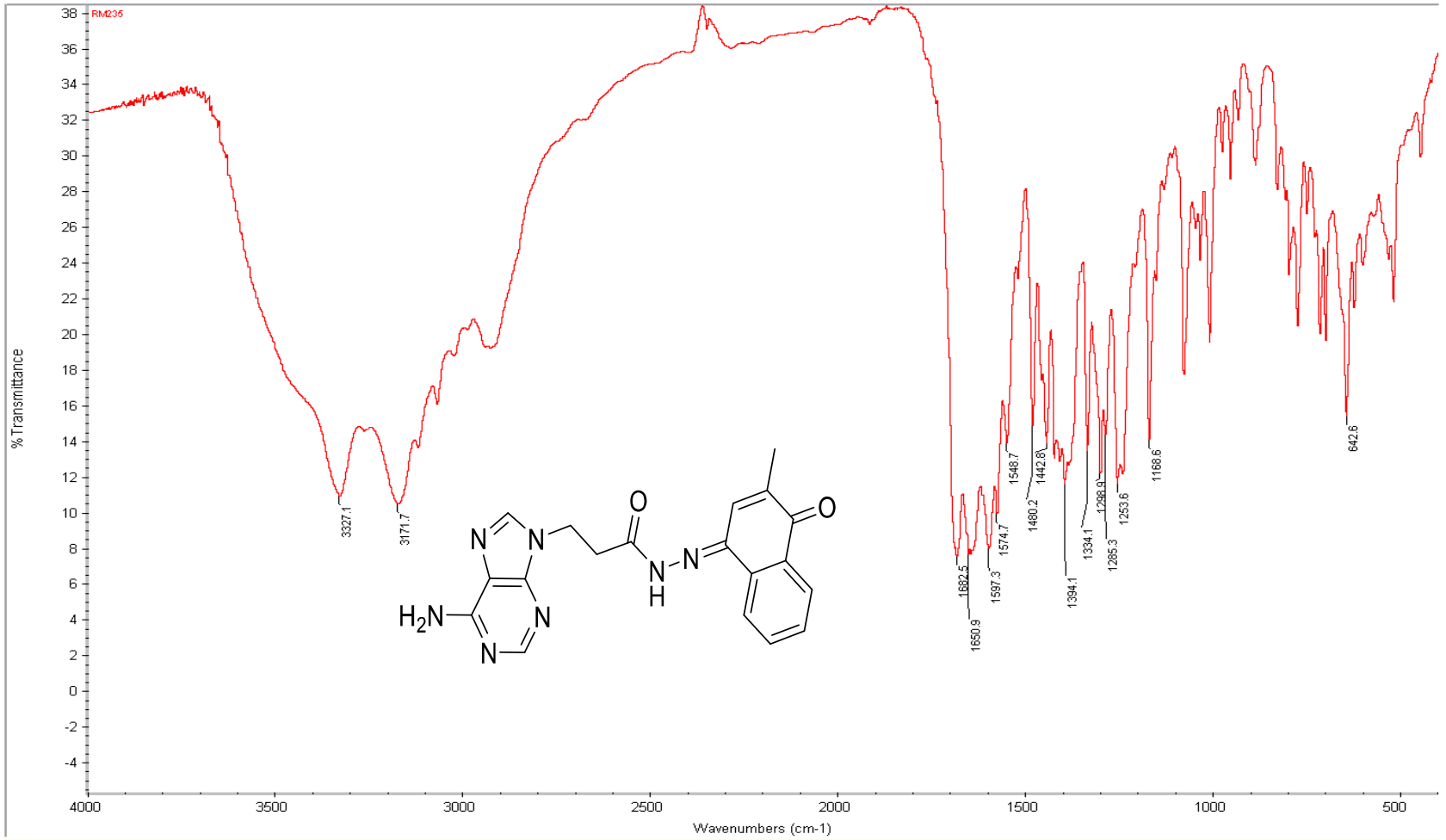


Figure S66. IR spectrum of compound **6m**

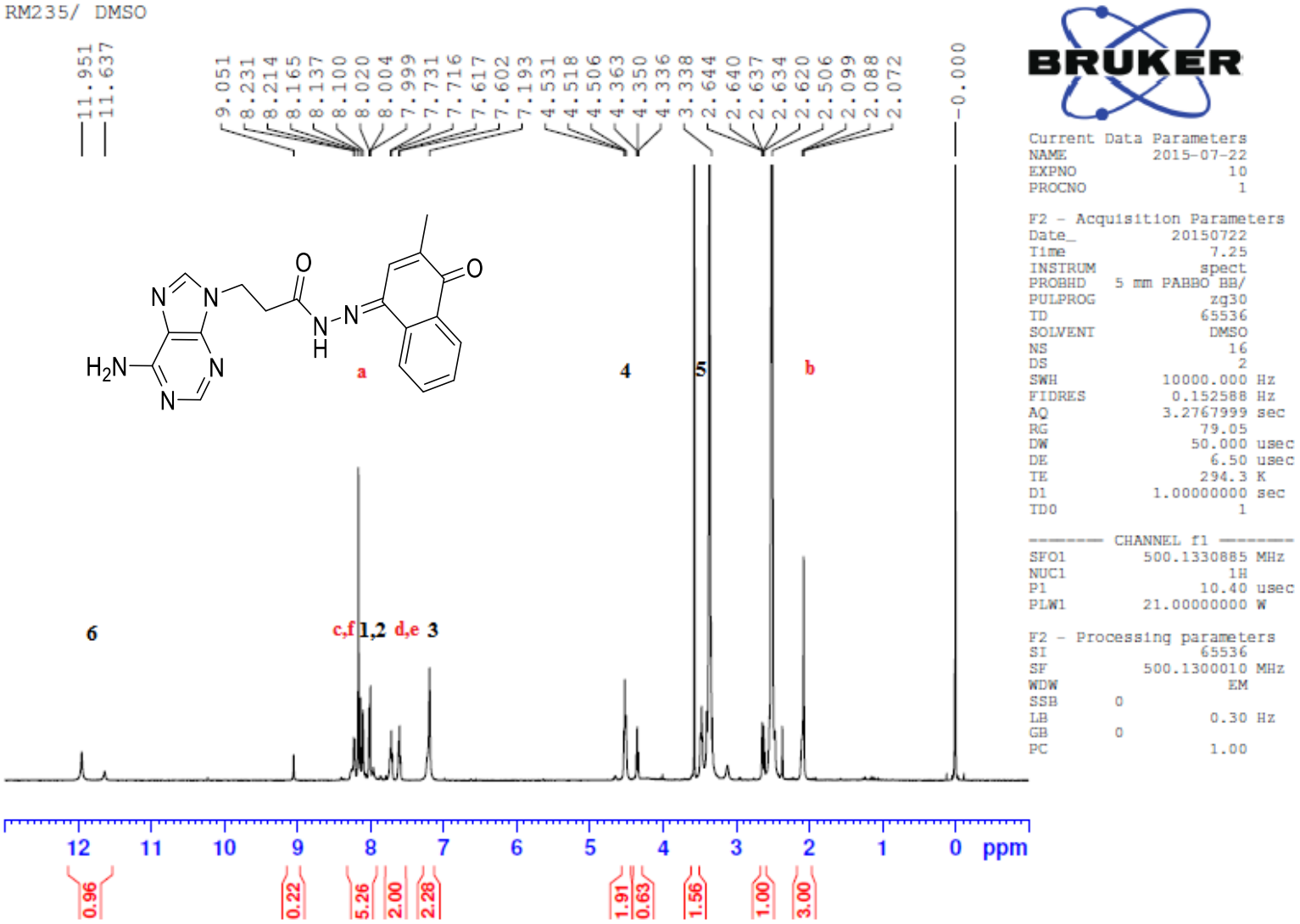


Figure S67. ^{1}H NMR (DMSO, 500 MHz) spectrum of compound **6m**

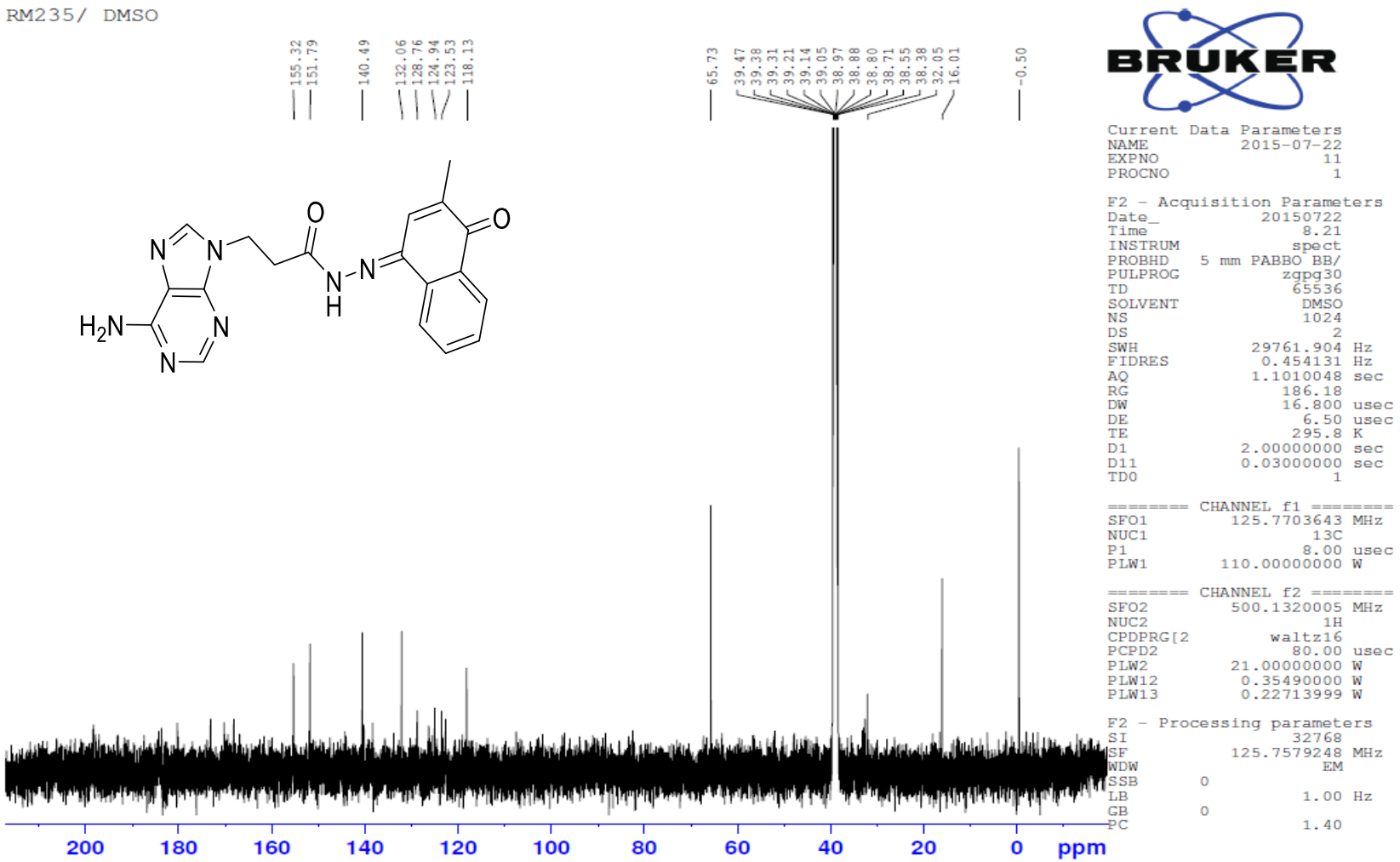


Figure S68. ^{13}C NMR (DMSO, 100 MHz) spectrum of compound **6m**

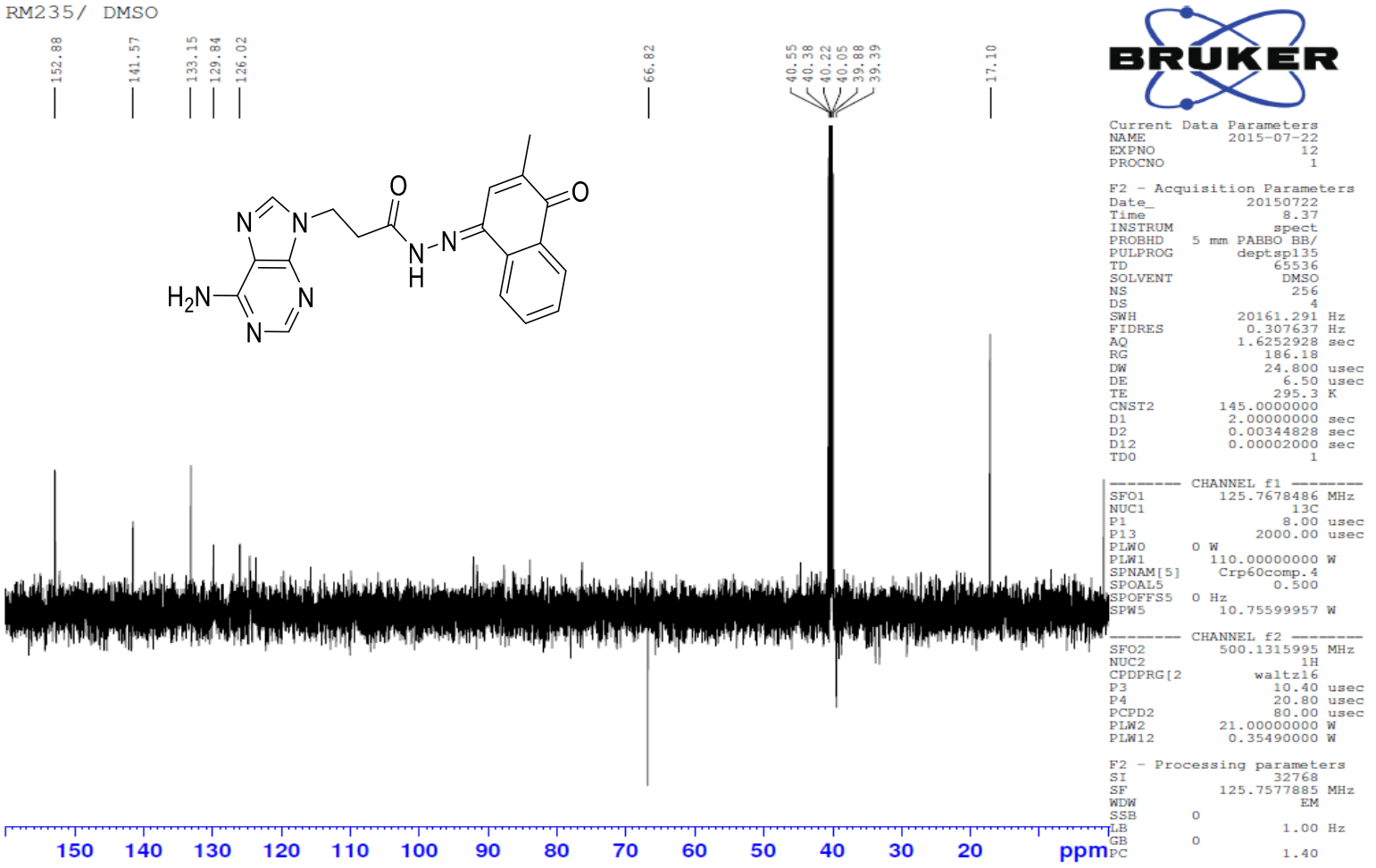


Figure S69. DEPT NMR (DMSO, 100 MHz) spectrum of compound **6m**

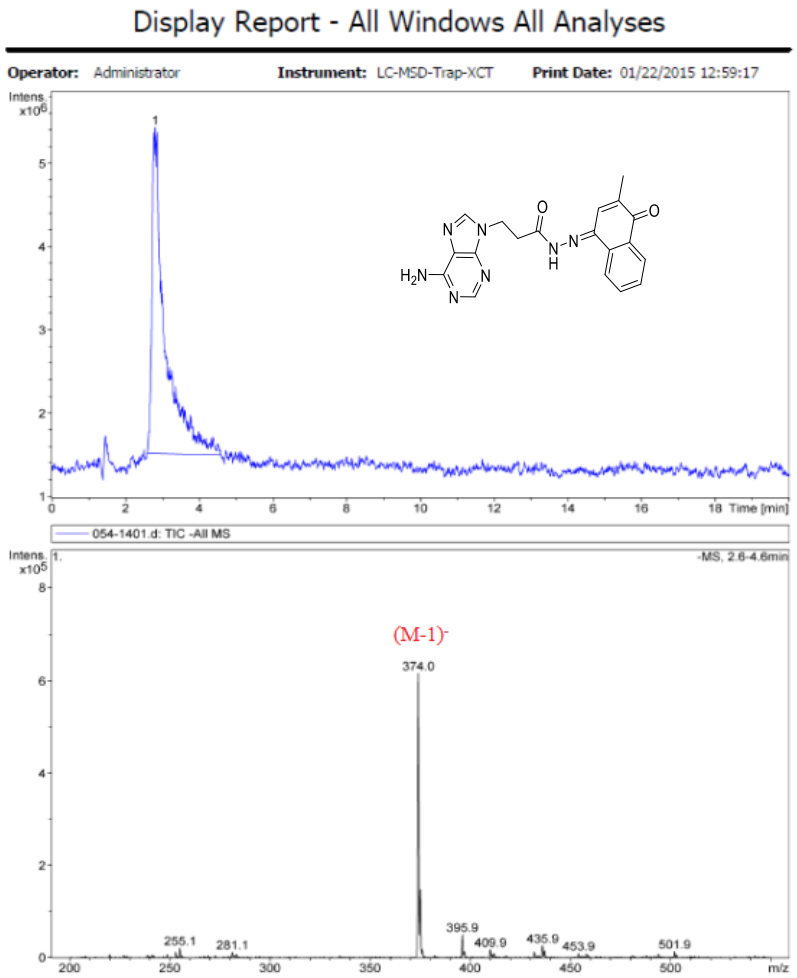
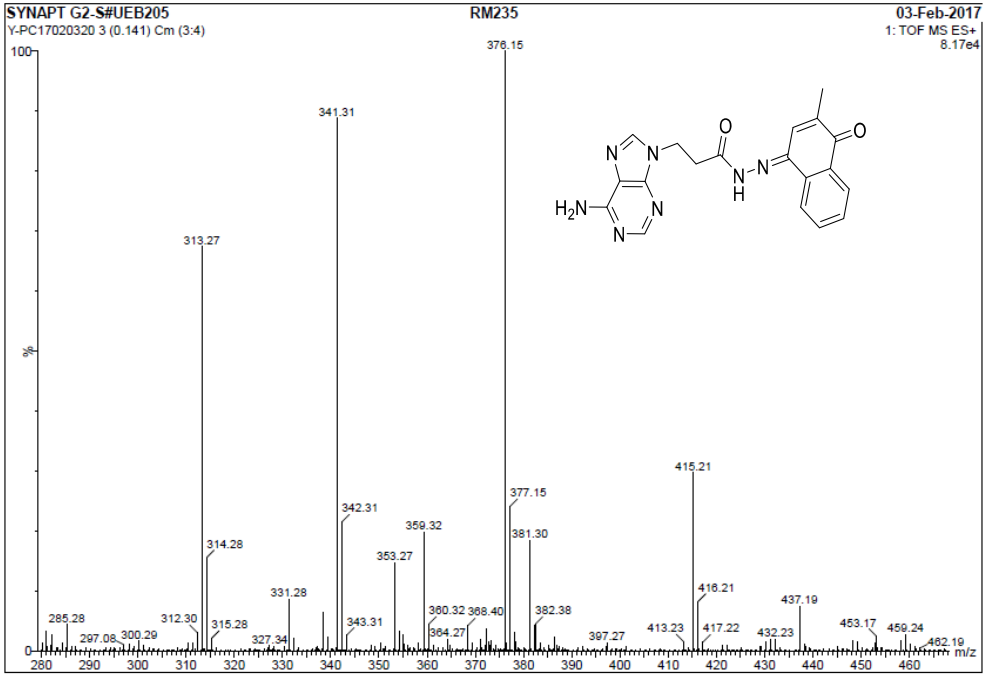


Figure S70. LC-MS spectrum of compound **6m**



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
993 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-S#UEB205 RM235 03-Feb-2017
Y-PC17020320 3 (0.141) Cm (3:4) 1: TOF MS ES+ 8.17e+004

Minimum:				-1.5				
Maximum:		1.0	1.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
376.1523	376.1522	0.1	0.3	14.5	919.4	0.000	100.00	C19 H18 N7 O2
	376.1527	-0.4	-1.1	7.5	936.6	17.273	0.00	C4 H14 N19 O3
	376.1514	0.9	2.4	2.5	936.9	17.526	0.00	C3 H18 N15 O7

Figure S71. HRMS spectrum of compound **6m**