

Electronic supplementary information for

New *Securinega* alkaloids with anti-HIV activity from *Flueggea virosa*

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Table S1. NMR data comparison of alkaloid **3** with virosecurinine and flueggenine A in CDCl₃.

Figure Sa. Key 2D NMR correlations for alkaloids **5–8**.

Figure Sb. CD spectra for alkaloids **1, 2** and **4–9**.

Figure S1. ¹H NMR spectrum for flueggenine E (**1**) in CDCl₃.

Figure S2. ¹³C NMR spectrum for flueggenine E (**1**) in CDCl₃.

Figure S3. ¹H–¹H COSY spectrum for flueggenine E (**1**) in CDCl₃.

Figure S4. HSQC spectrum for flueggenine E (**1**) in CDCl₃.

Figure S5. HMBC spectrum for flueggenine E (**1**) in CDCl₃.

Figure S6. ROESY spectrum for flueggenine E (**1**) in CDCl₃.

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Figure S8. (+)-ESIMS spectrum for flueggenine E (**1**).

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Figure S10. ¹H NMR spectrum for flueggenine F (**2**) in CDCl₃.

Figure S11. ¹³C NMR spectrum for flueggenine F (**2**) in CDCl₃.

Figure S12. ¹H–¹H COSY spectrum for flueggenine F (**2**) in CDCl₃.

Figure S13. HSQC spectrum for flueggenine F (**2**) in CDCl₃.

Figure S14. HMBC spectrum for flueggenine F (**2**) in CDCl₃.

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Figure S16. IR spectrum for flueggenine F (**2**).

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Figure S19. ¹H NMR spectrum for flueggenine G (**3**) in CDCl₃.

Figure S20. ¹³C NMR spectrum for flueggenine G (**3**) in CDCl₃.

Figure S21. ¹H–¹H COSY spectrum for flueggenine G (**3**) in CDCl₃.

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Figure S22. HSQC spectrum for flueggenine G (**3**) in CDCl₃.
Figure S23. HMBC spectrum for flueggenine G (**3**) in CDCl₃.
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Figure S29. ¹³C NMR spectrum for flueggenine H (**4**) in CDCl₃ (5% CD₃OD).
Figure S30. ¹H-¹H COSY spectrum for flueggenine H (**4**) in CDCl₃ (5% CD₃OD).
Figure S31. HSQC spectrum for flueggenine H (**4**) in CDCl₃ (5% CD₃OD).
Figure S32. HMBC spectrum for flueggenine H (**4**) in CDCl₃ (5% CD₃OD).
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Figure S41. HMBC spectrum for flueggenine I (**5**) in CDCl₃.
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Figure S50. HMBC spectrum for flueggenine E (**6**) in CDCl₃.
Figure S51. ROESY spectrum for flueggenine E (**6**) in CDCl₃.
Figure S52. IR spectrum for flueggenine E (**6**).
Figure S53. (+)-ESIMS spectrum for flueggenine E (**6**).
Figure S54. (+)-HRESIMS spectrum for flueggenine E (**6**).

Figure S55. ¹H NMR spectrum for flueviroisine F (**7**) in CDCl₃.
Figure S56. ¹³C NMR spectrum for flueggenine F (**7**) in CDCl₃.
Figure S57. ¹H-¹H COSY spectrum for flueggenine F (**7**) in CDCl₃.
Figure S58. HSQC spectrum for flueggenine F (**7**) in CDCl₃.
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Figure S60. ROESY spectrum for flueggenine F (**7**) in CDCl₃.
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Figure S64. ^1H NMR spectrum for flueviroisine G (**8**) in CDCl_3 .
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Figure S70. IR spectrum for flueggenine G (**8**).
Figure S71. (+)-ESIMS spectrum for flueggenine G (**8**).
Figure S72. (+)-HRESIMS spectrum for flueggenine G (**8**).

Figure S73. ^1H NMR spectrum for flueviroisine H (**9**) in CDCl_3 .
Figure S74. ^{13}C NMR spectrum for flueggenine H (**9**) in CDCl_3 .
Figure S75. ^1H - ^1H COSY spectrum for flueggenine H (**9**) in CDCl_3 .
Figure S76. HSQC spectrum for flueggenine H (**9**) CDCl_3 .
Figure S77. HMBC spectrum for flueggenine H (**9**) in CDCl_3 .
Figure S78. ROESY spectrum for flueggenine H (**9**) in CDCl_3 .
Figure S79. IR spectrum for flueggenine H (**9**).
Figure S80. (+)-ESIMS spectrum for flueggenine H (**9**).
Figure S81. (+)-HRESIMS spectrum for flueggenine H (**9**).

Table S1. NMR data comparison of alkaloid **3** with virosecurinine and fluggenine A in CDCl₃.

No.	3		Virosecurinine^a		fluggenine A^b	
	δ_C	δ_H (multi., <i>J</i> in Hz)	δ_C	δ_H (multi., <i>J</i> in Hz)	δ_C	δ_H (multi., <i>J</i> in Hz)
2	63.2	2.04 (dd, 11.3, 2.6)	63.0	2.09 (dd, 11.0, 2.0)		
3	27.0	a 1.67 (m) b 1.57 (m)	27.2	a 1.65–1.48 (m) b 1.65–1.48 (m)		
4	24.3	a 1.88 (m) b 1.22 (m)	24.5	a 1.87 (m) b 1.26–1.17 (m)		
5	25.6	1.56–1.64 (m, 2H)	25.9	1.65–1.48 (m) 1.65–1.48 (m)		
6	48.9	a 2.99 (m) b 2.41 (m)	48.8	a 2.95 (dt, 10.5, 4.0) b 2.41 (m)		
7	58.5	3.85 (dd, 5.6, 4.1)	58.8	3.80 (t, 4.5)		
8	42.2	a 2.50 (dd, 9.1, 4.1) b 1.72 (d, 9.1)	42.3	a 2.49 (dd, 9.0, 4.0) b 1.76 (d, 9.0)		
9	89.7		89.5			
11	173.2		173.7			
12	103.6	5.56 (s)	105.1	5.53 (s)		
13	171.8		170.1			
14	134.7		121.4	6.58 (d, 9.0)		
15	135.4	6.67 (d, 5.6)	140.2	6.40 (dd, 9.5, 5.5)		
						dihydronorsecurinine moiety
2'	67.0	3.10 (dd, 8.7, 7.0)			66.7	3.12 (m)
3'	29.0 ^c	a 1.92 (m) b 1.79 (m)			29.0	a 1.90 (m) b 1.73 (m)
4'	26.6	a 1.96 (m) b 1.63 (m)			26.5	a 1.93 (m) b 1.69 (m)
5'	57.5	a 3.22 (m) b 2.60 (m)			57.6	a 3.25 (m) b 2.53 (m)
7'	65.0	3.01 (m)			64.3	3.20 (d, 5.6)
8'	35.8	a 2.59 (m) b 1.49 (d, 11.0)			35.4	a 2.61 (dd, 11.1, 5.6) b 1.50 (d, 11.1)
9'	91.9				91.7	
11'	172.6				172.6	
12'	110.2	5.67 (d, 2.1)			110.4	5.69 (d, 2.0)
13'	172.8				172.2	
14'	29.1 ^c	α 2.97 (m) β 2.66 (ddd, 15.0, 11.8, 2.1)			27.3	α 2.90 (dd, 15.5, 5.5) β 2.75 (ddd, 15.5, 12.1, 2.0)
15'	42.9	2.53 (m)			42.8	2.54 (m)

^a Wu, H. Y.; Zhou, J. Y. *Zhongguo Zhongyao Zazhi* **2004**, *29*, 535–536.

^b Zhang, H.; Zhang, C. R.; Zhu, K. K.; Gao, A. H.; Luo, C.; Li, J.; Yue, J. M. *Org. Lett.* **2013**, *15*, 120–123.

^c Interchangeable assignments.

Figure Sa. Key 2D NMR correlations for alkaloids **5–8**.

COSY —
HMBC ↷

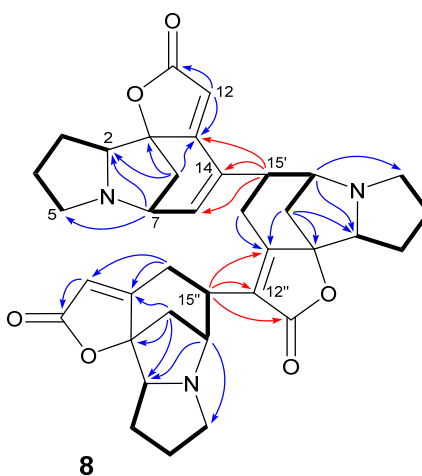
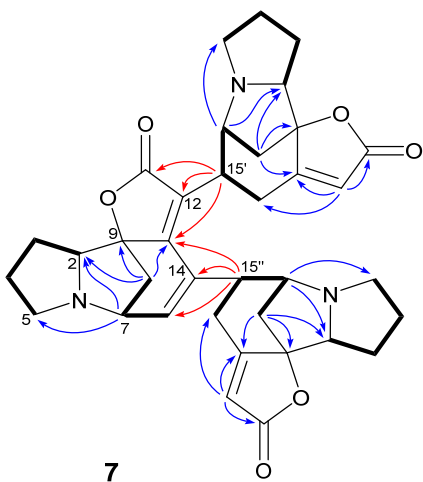
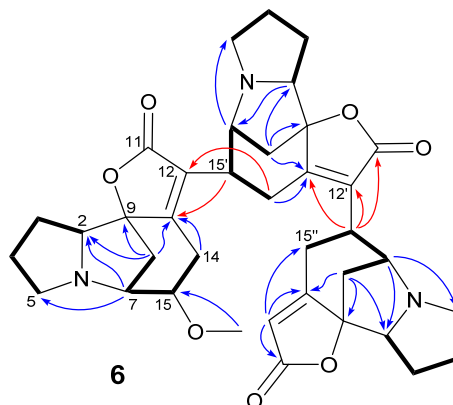
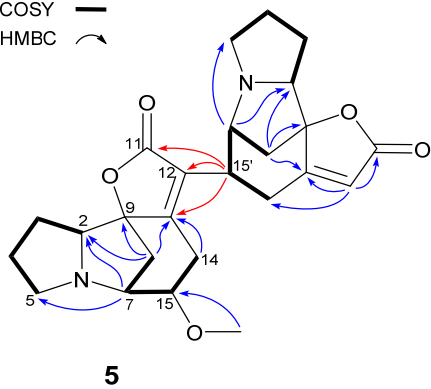
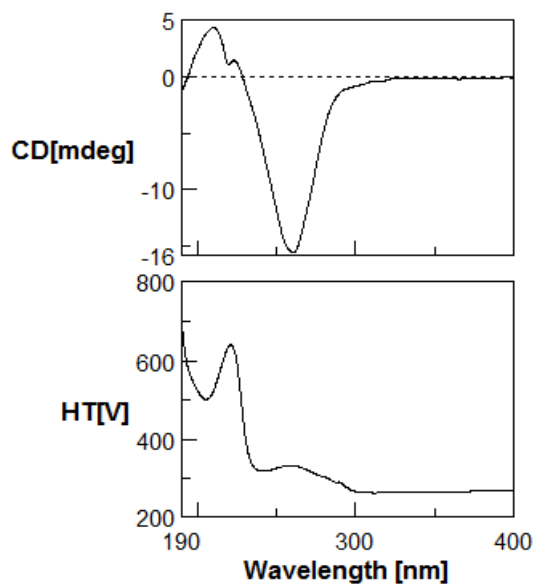
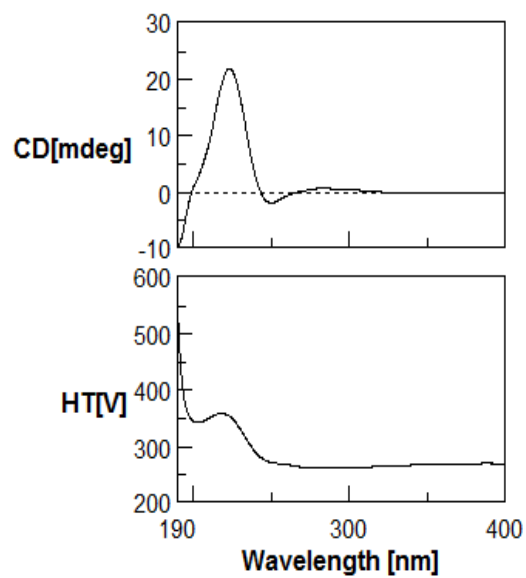


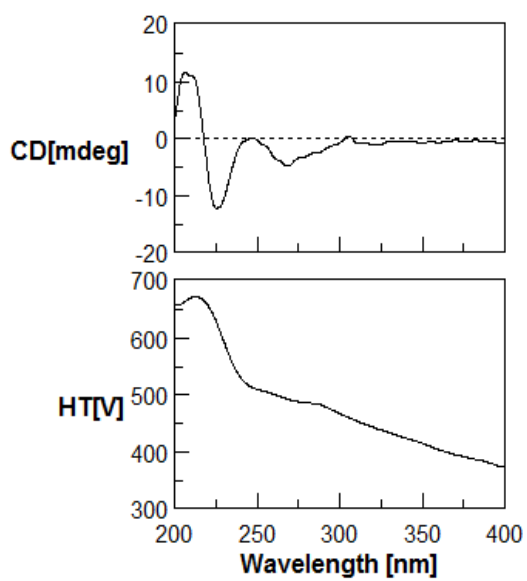
Figure Sb. CD spectra for alkaloids 1, 2 and 4-9.



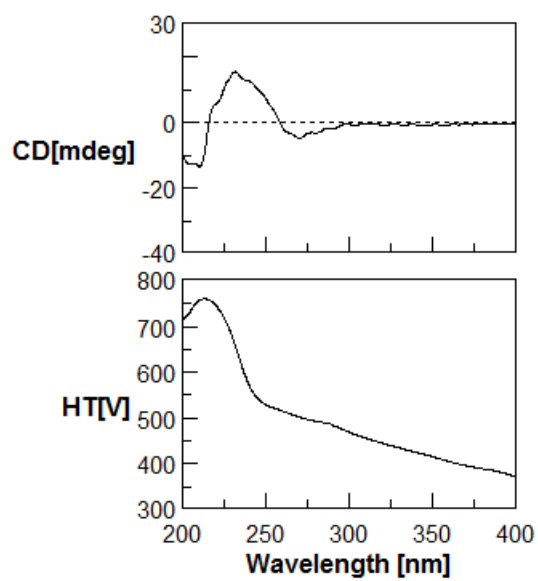
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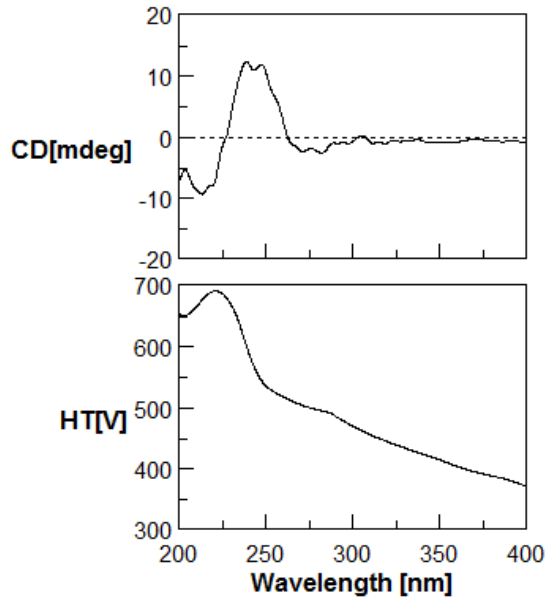
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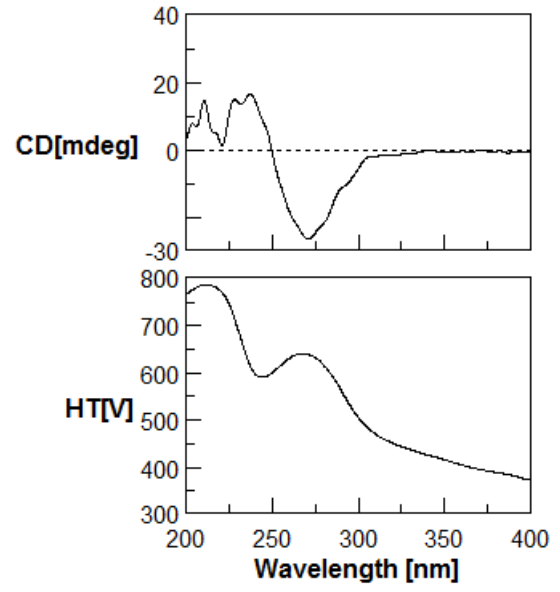
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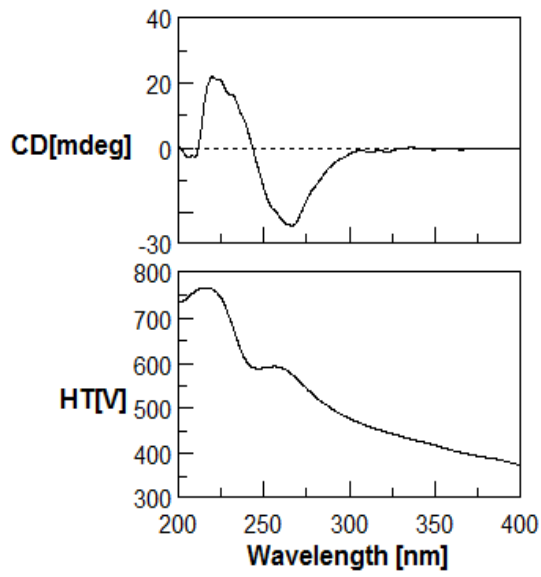
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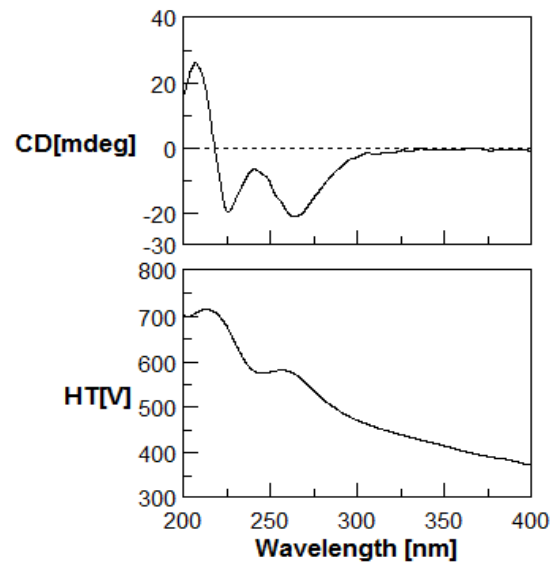
Alkaloid 6



Alkaloid 7



Alkaloid 8



Alkaloid 9

Figure S1. ^1H NMR spectrum for flueggeine E (**1**) in CDCl_3 .

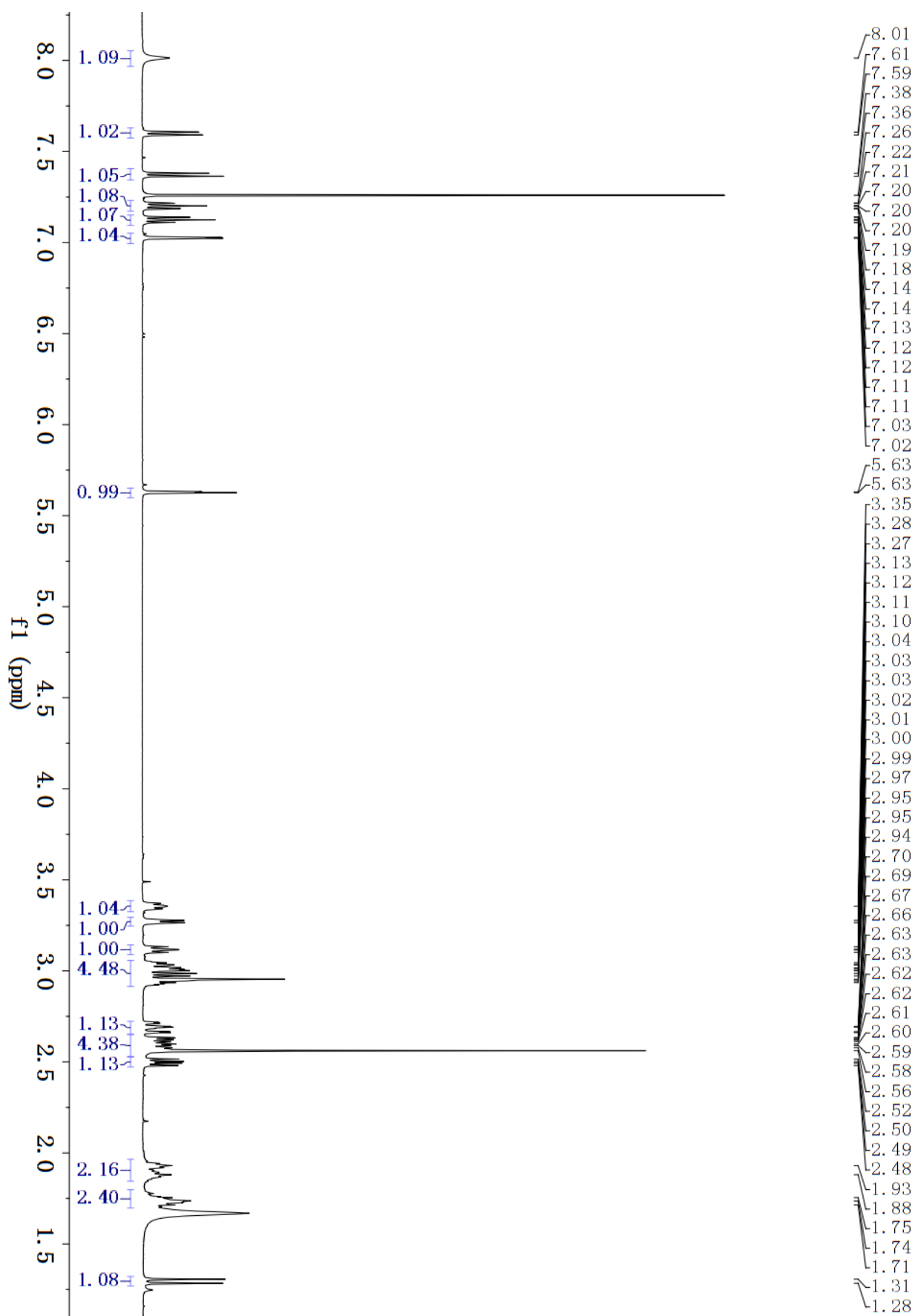


Figure S2. ^{13}C NMR spectrum for flueggeanine E (**1**) in CDCl_3 .

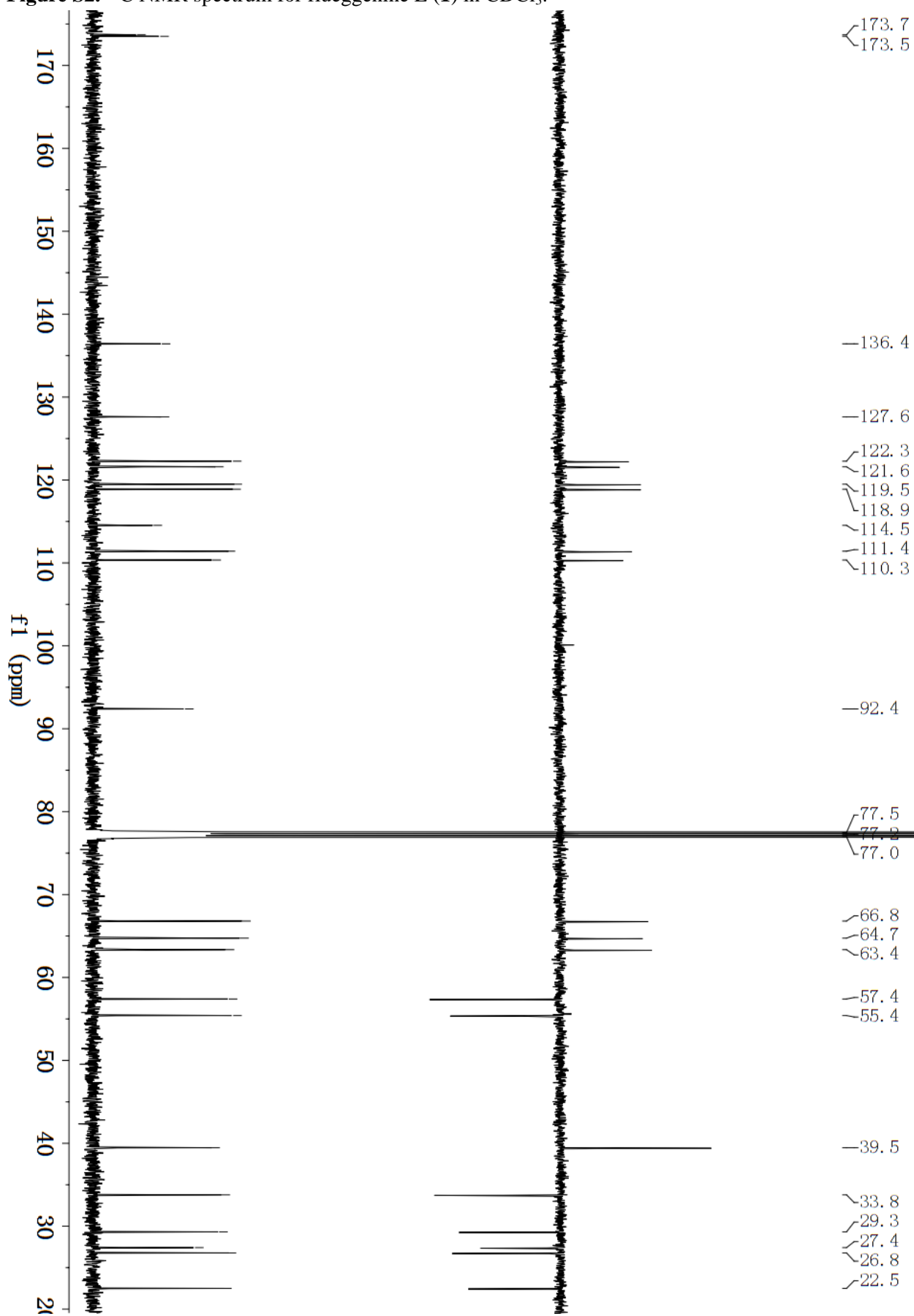


Figure S3. ^1H - ^1H COSY spectrum for flueggenine E (**1**) in CDCl_3 .

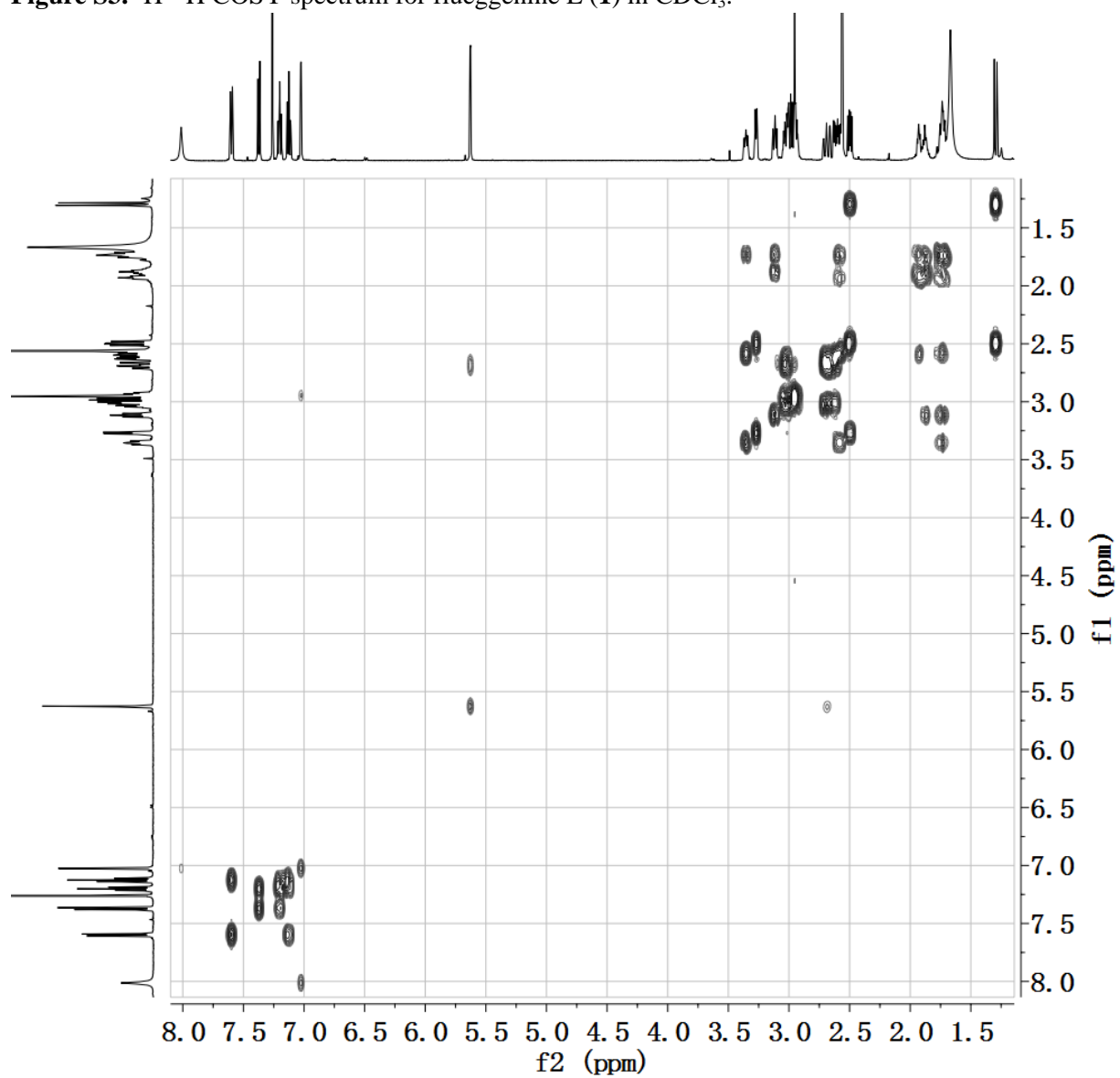


Figure S4. HSQC spectrum for flueggeine E (1) in CDCl₃.

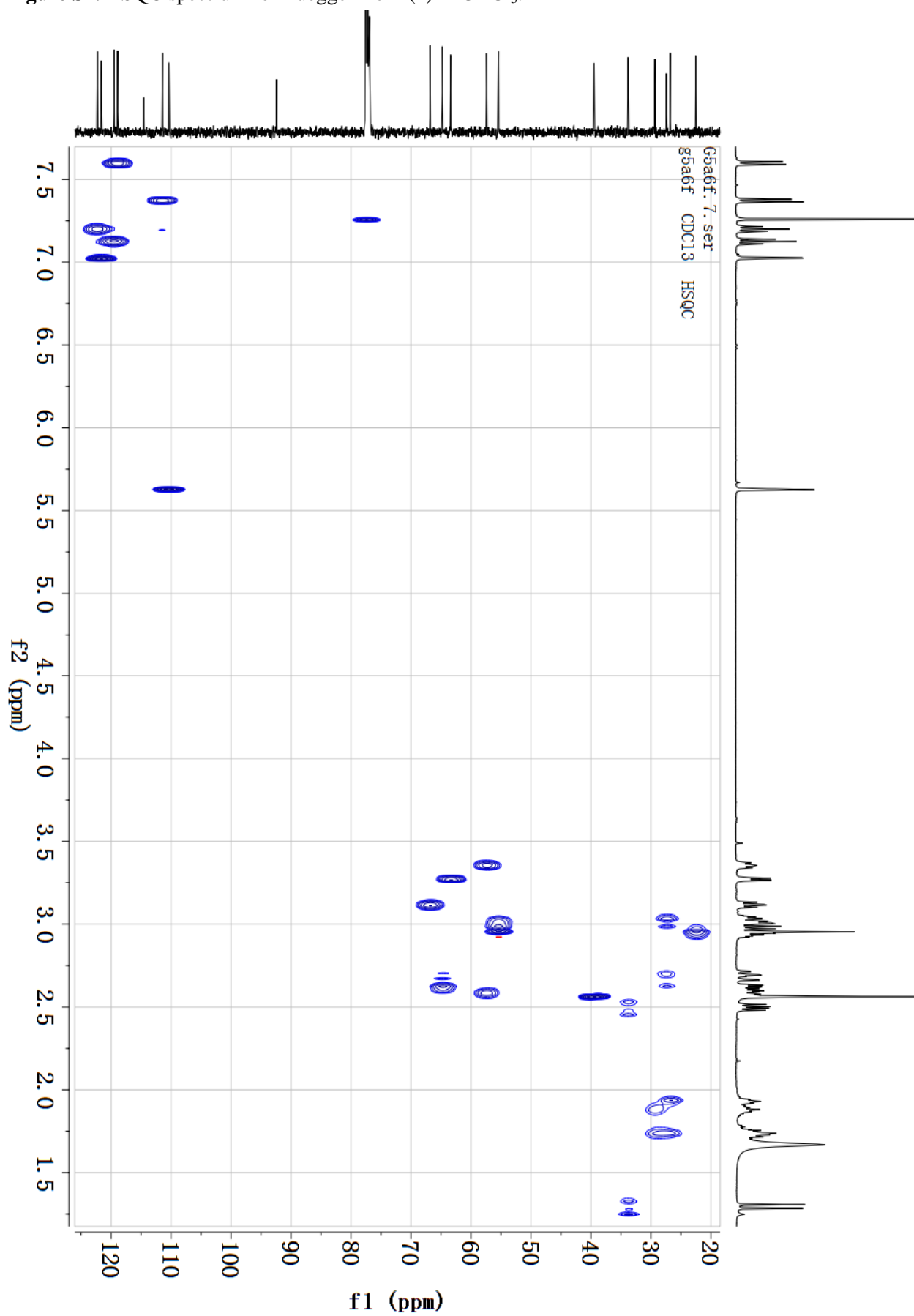


Figure S5. HMBC spectrum for flueggeanine E (1) in CDCl₃.

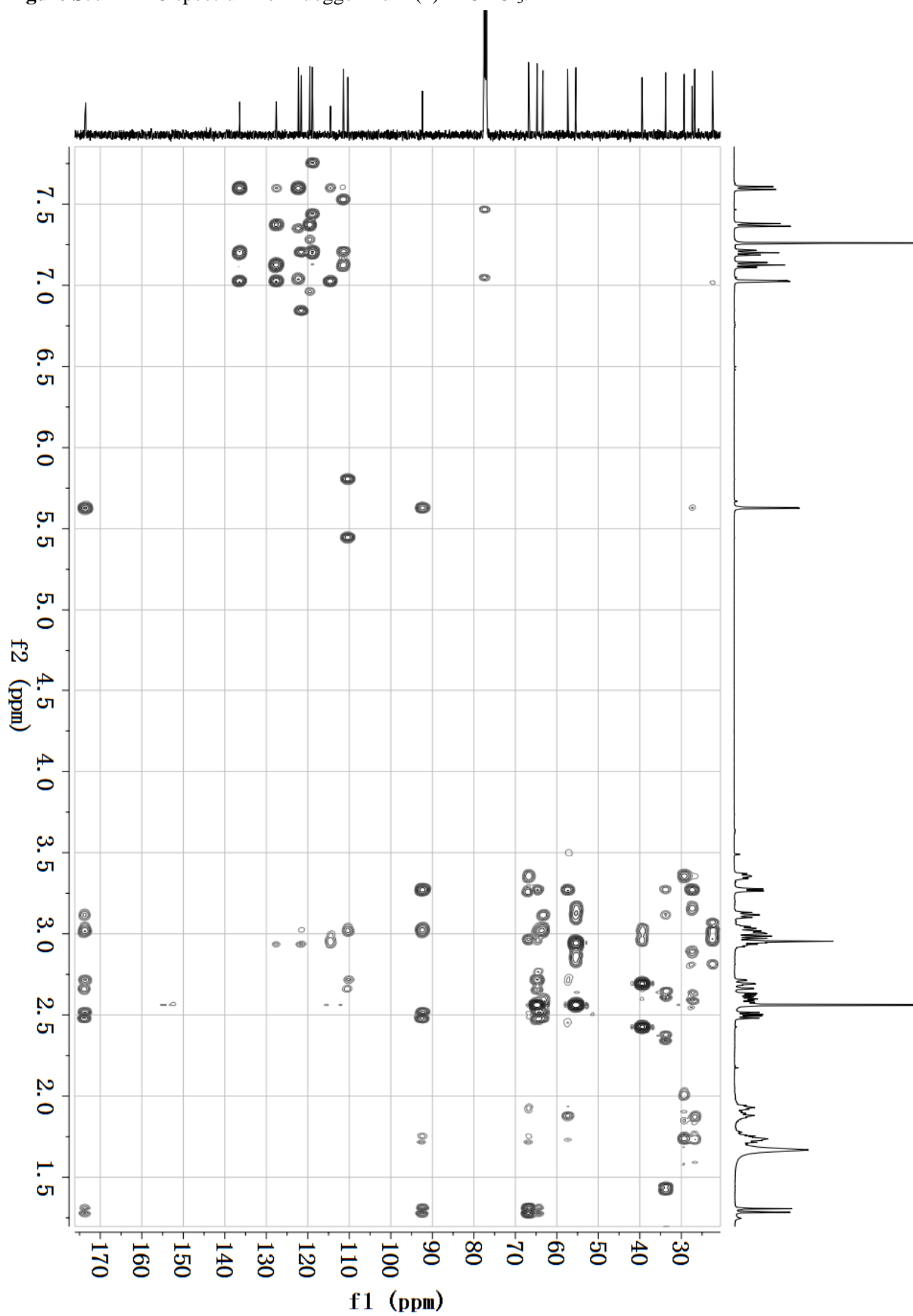


Figure S6. ROESY spectrum for flueggein E (1) in CDCl₃.

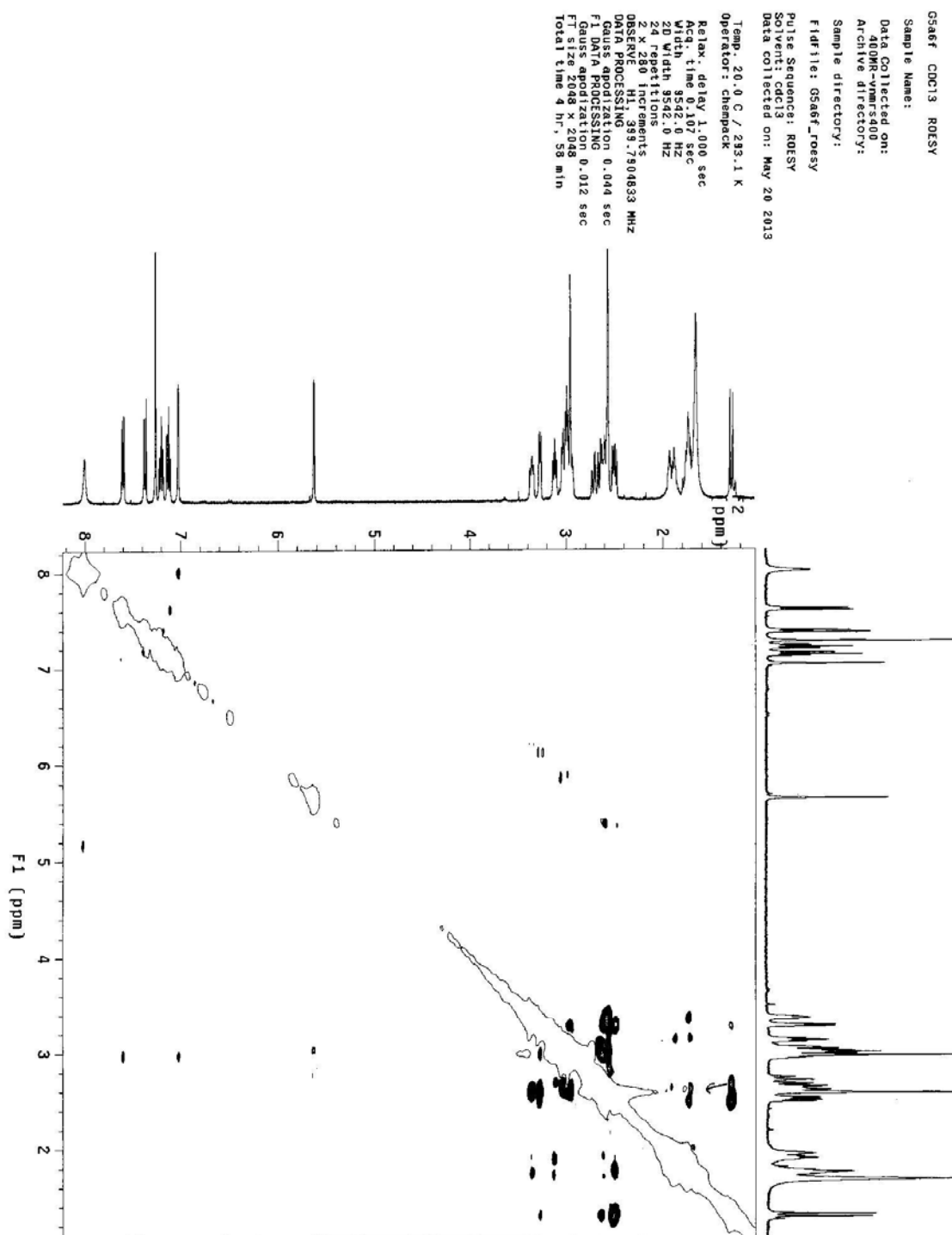


Figure S7. IR spectrum for flueggeine E (1).

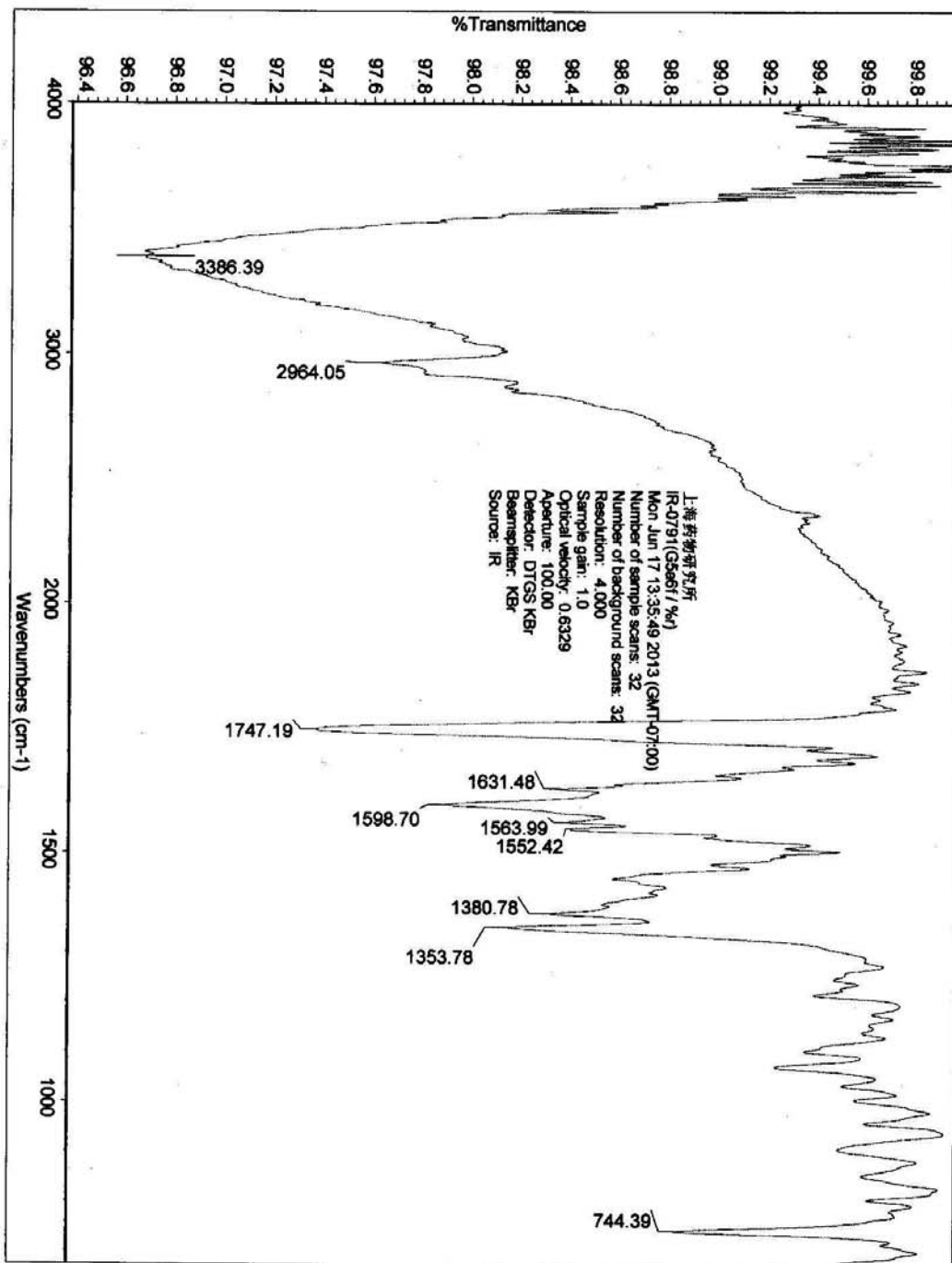


Figure S8. (+)-ESIMS spectrum for flueggeine E (1).

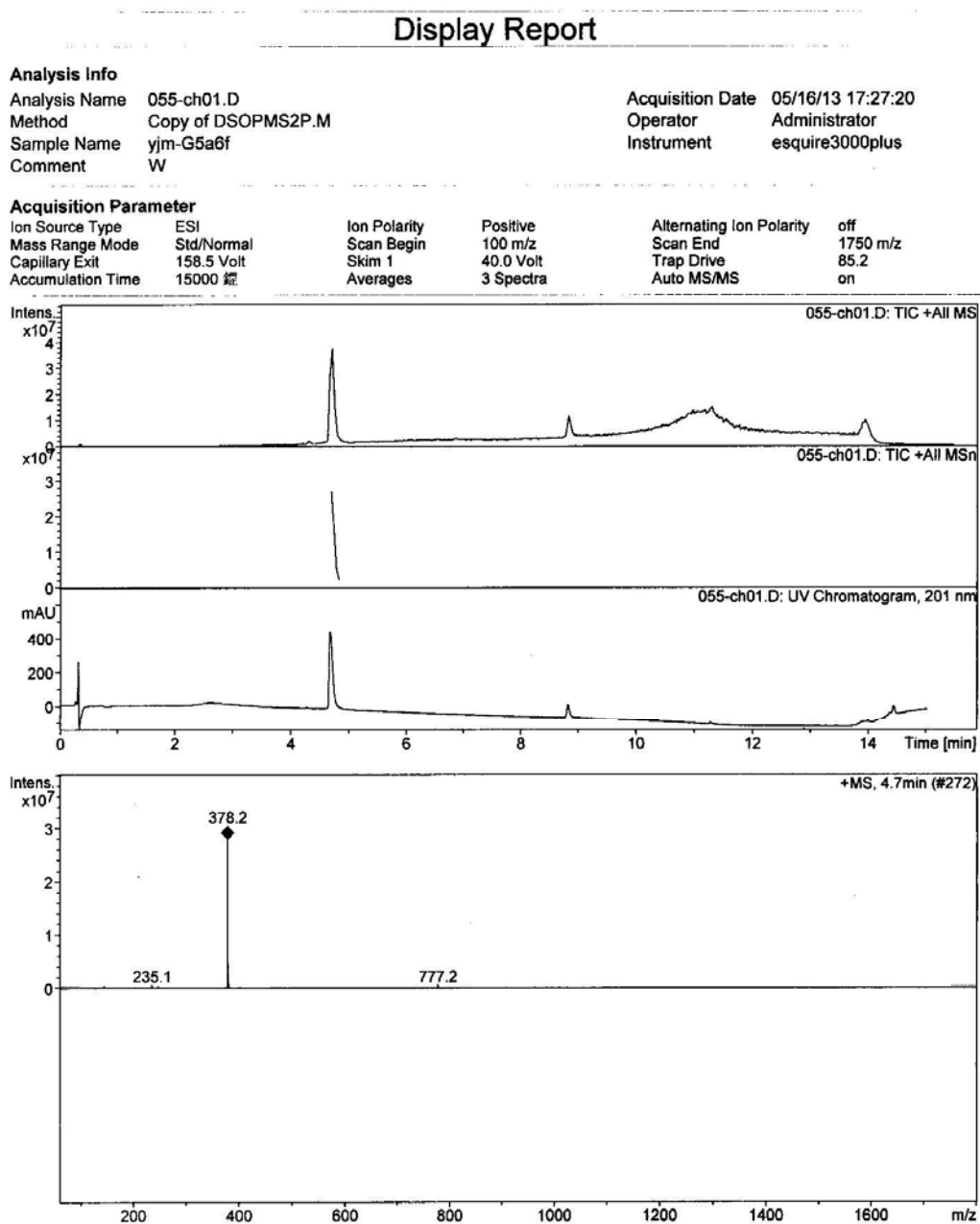


Figure S9. (+)-HRESIMS spectrum for flueggeine E (1).

Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
 Element prediction: Off
 Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

266 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 6-80 H: 2-120 N: 0-3 O: 0-20

G5a6f

LCT PXE KE324

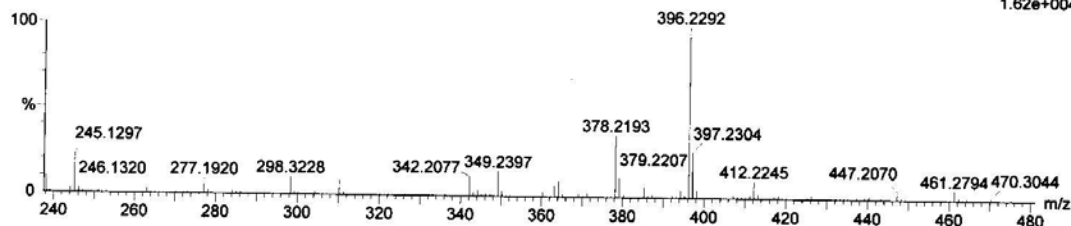
23-May-2013

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Maximum: 3.0 5.0 -1.5

Maximum: 3.0 5.0 50.0

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Figure S10. ^1H NMR spectrum for flueggeine F (**2**) in CDCl_3 .

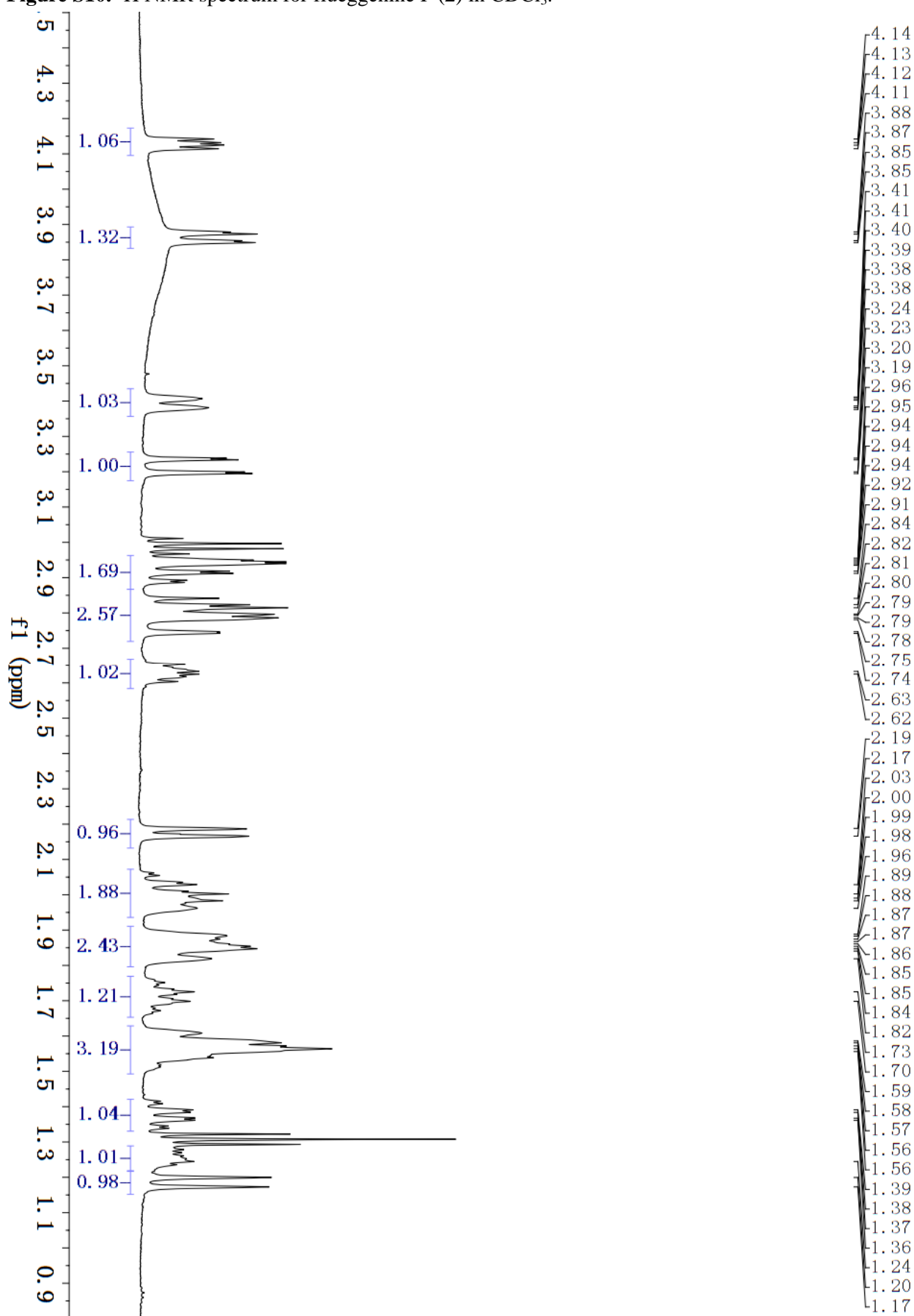


Figure S11. ^{13}C NMR spectrum for flueggeine F (2) in CDCl_3 .

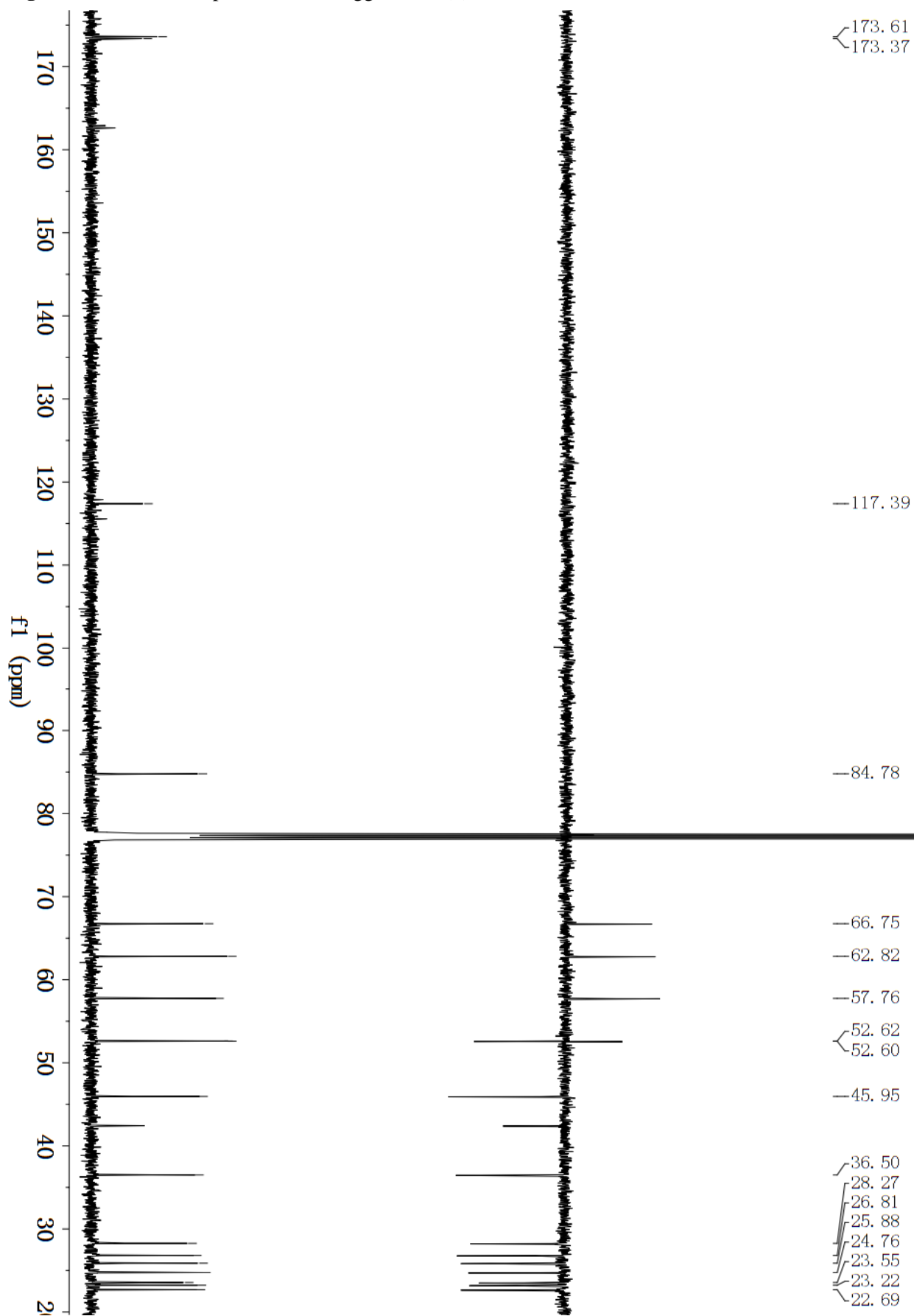


Figure S12. ^1H - ^1H COSY spectrum for flueggeanine F (**2**) in CDCl_3 .

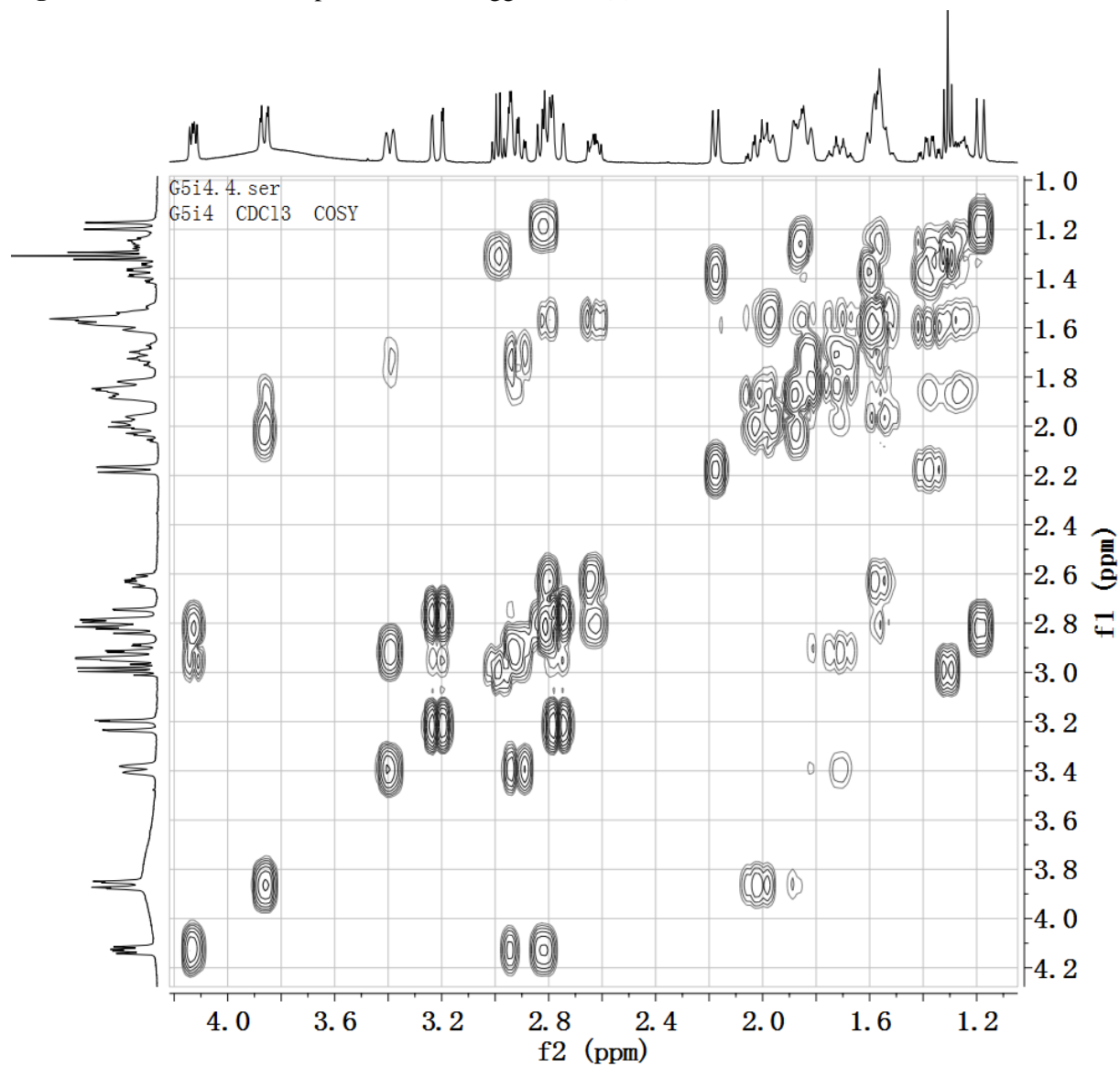


Figure S13. HSQC spectrum for flueggeine F (2) in CDCl₃.

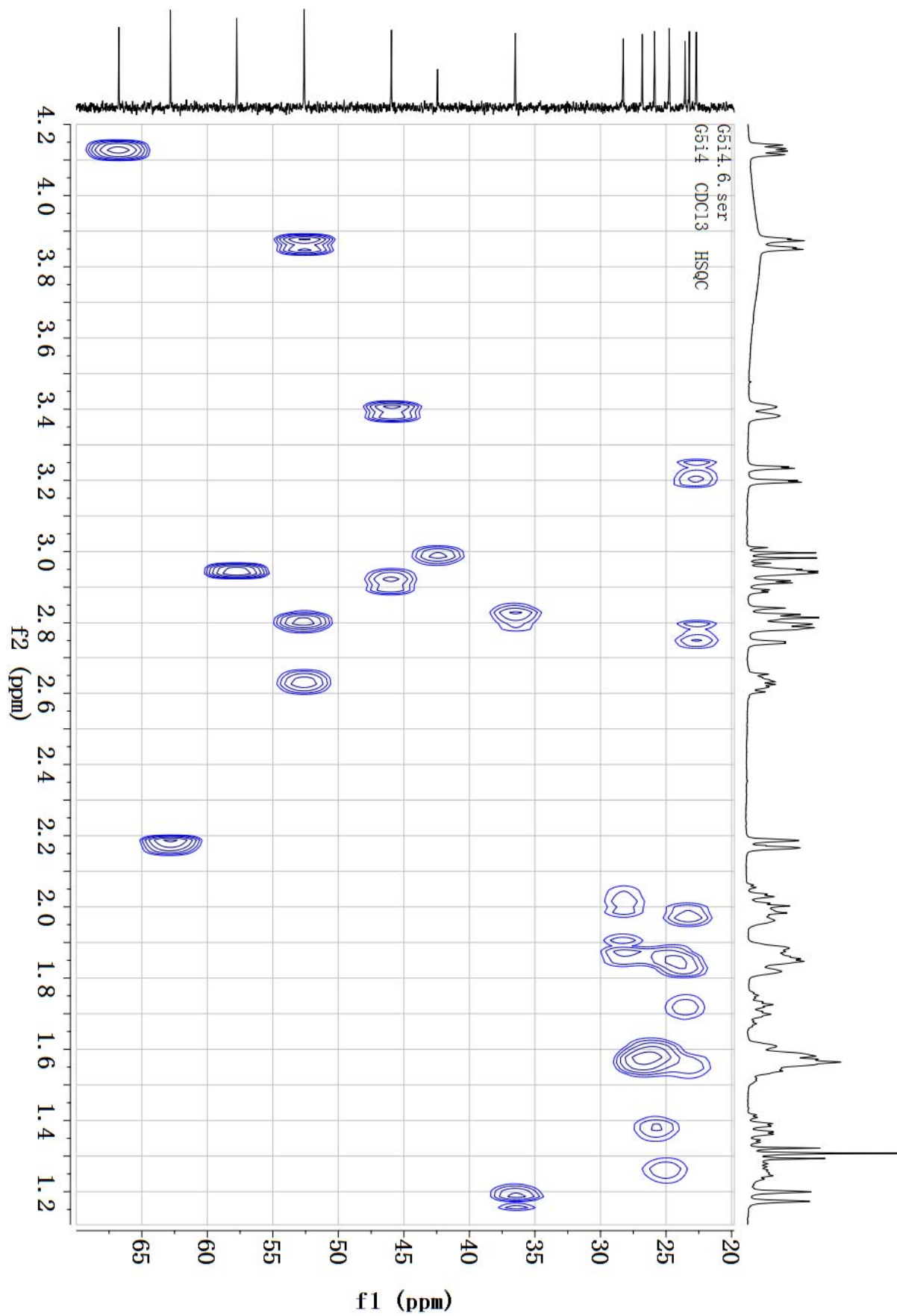


Figure S14. HMBC spectrum for flueggeanine F (2) in CDCl₃.

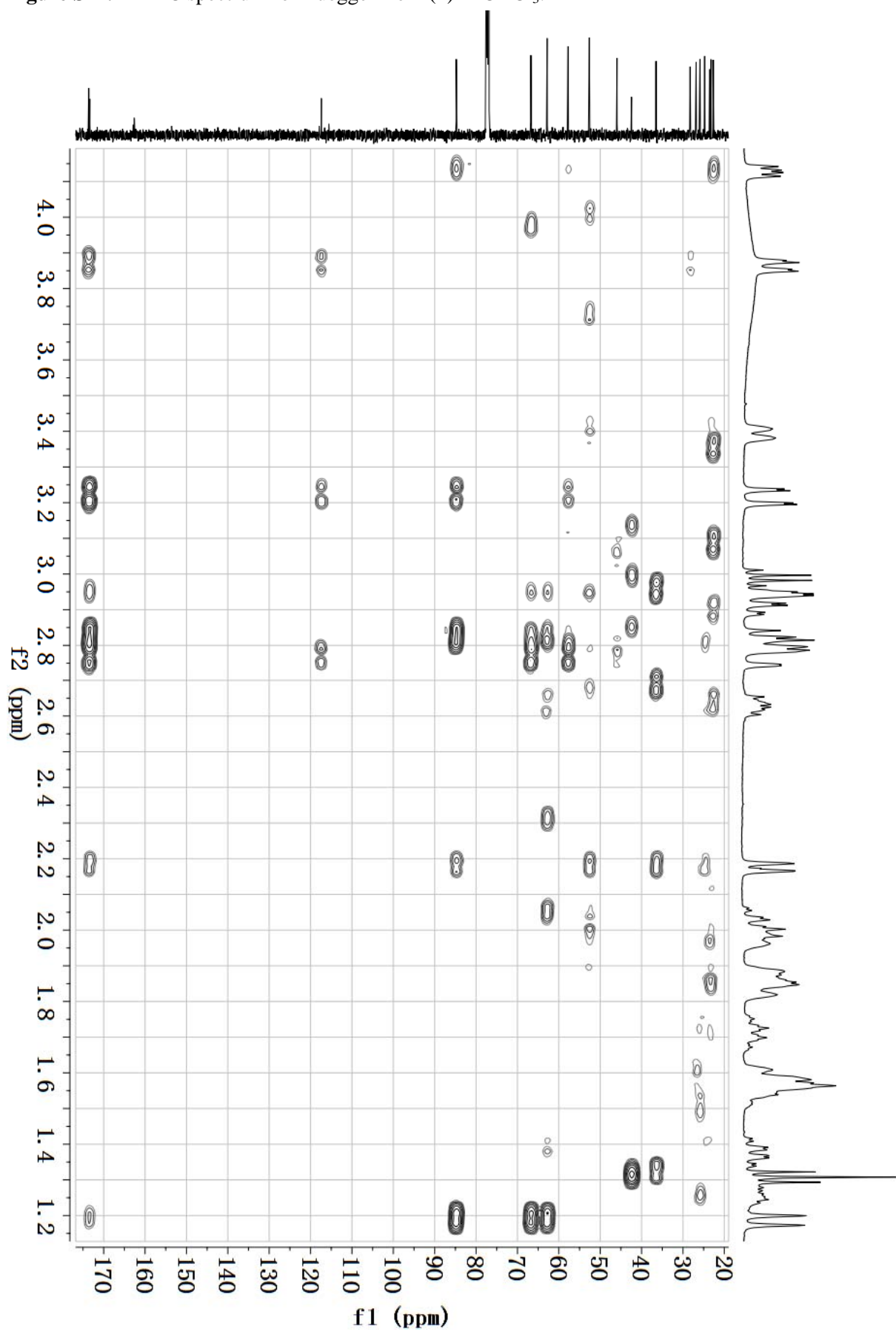


Figure S15. ROESY spectrum for flueggenine F (2) in CDCl₃.

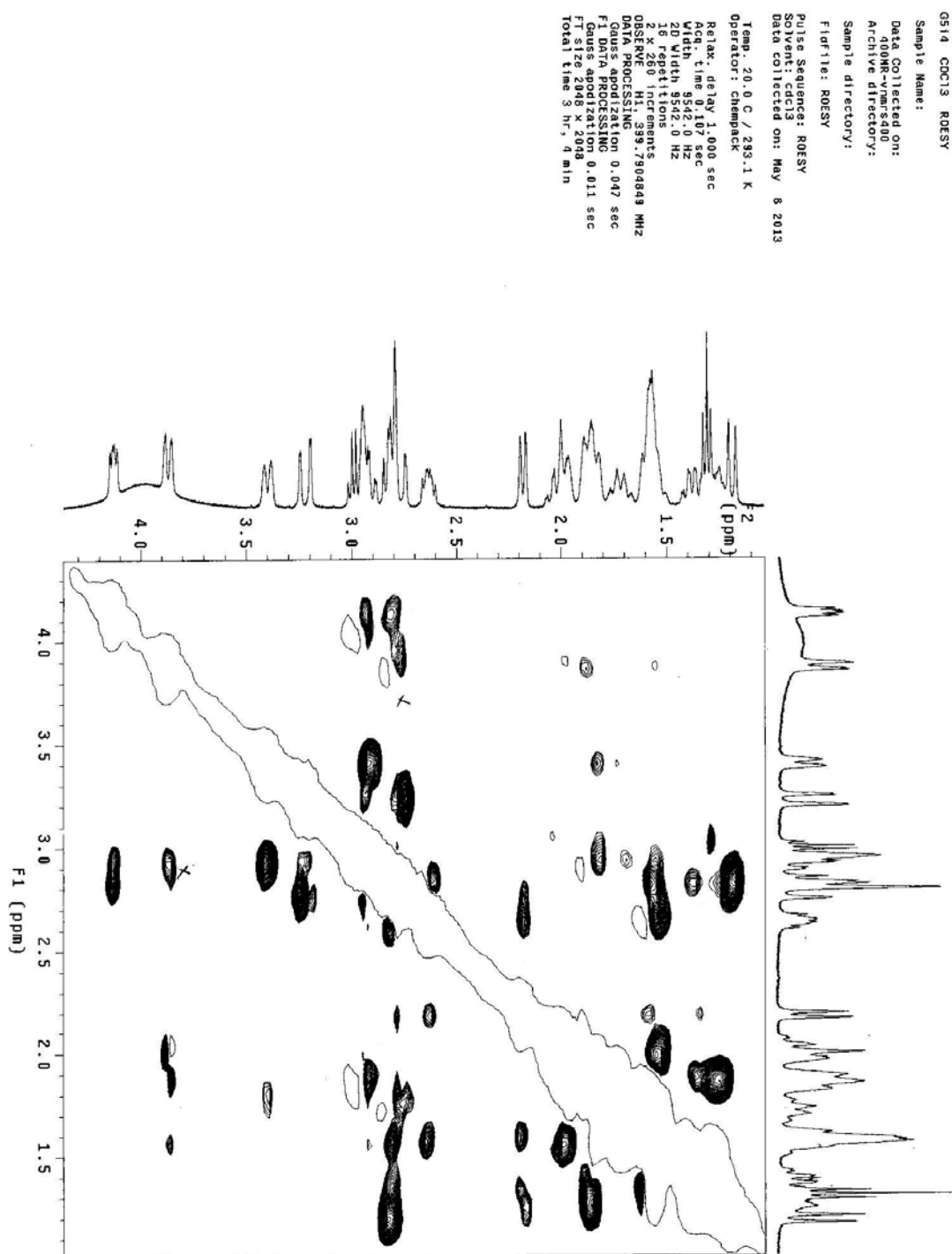


Figure S16. IR spectrum for flueggeine F (2).

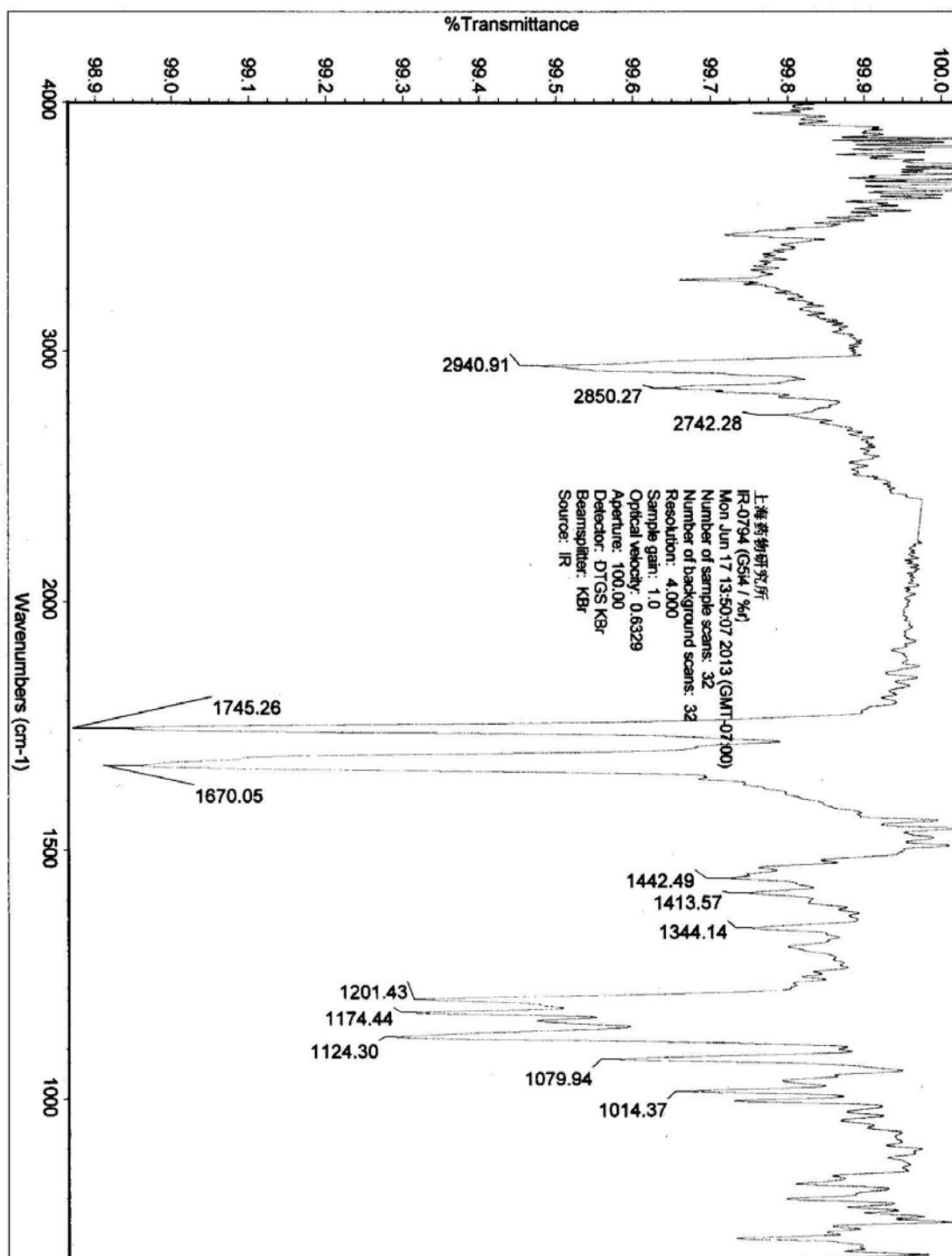


Figure S17. (+)-ESIMS spectrum for flueggeanine F (2).

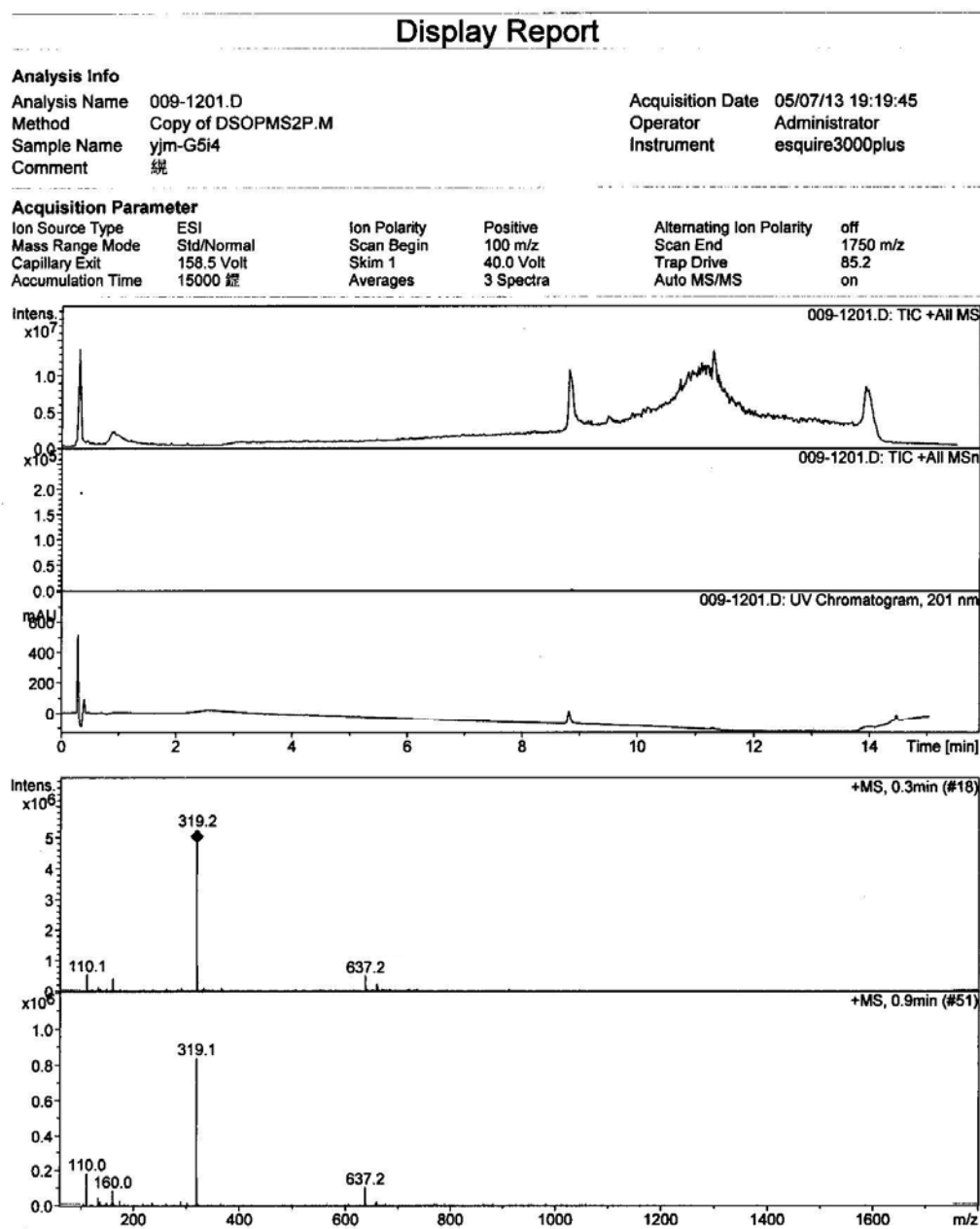


Figure S18. (+)-HRESIMS spectrum for flueggeine F (2).

Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

150 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 6-80 H: 2-120 N: 0-2 O: 0-20

G5i4

LCT PXE KE324

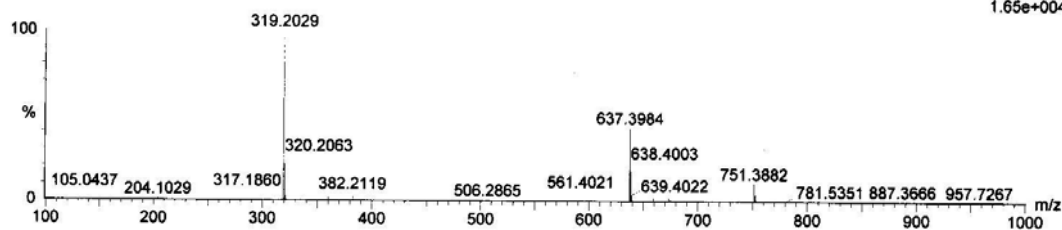
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Maximum: 3.0 5.0 -1.5 50.0

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Figure S19. ^1H NMR spectrum for flueggeine G (**3**) in CDCl_3 .

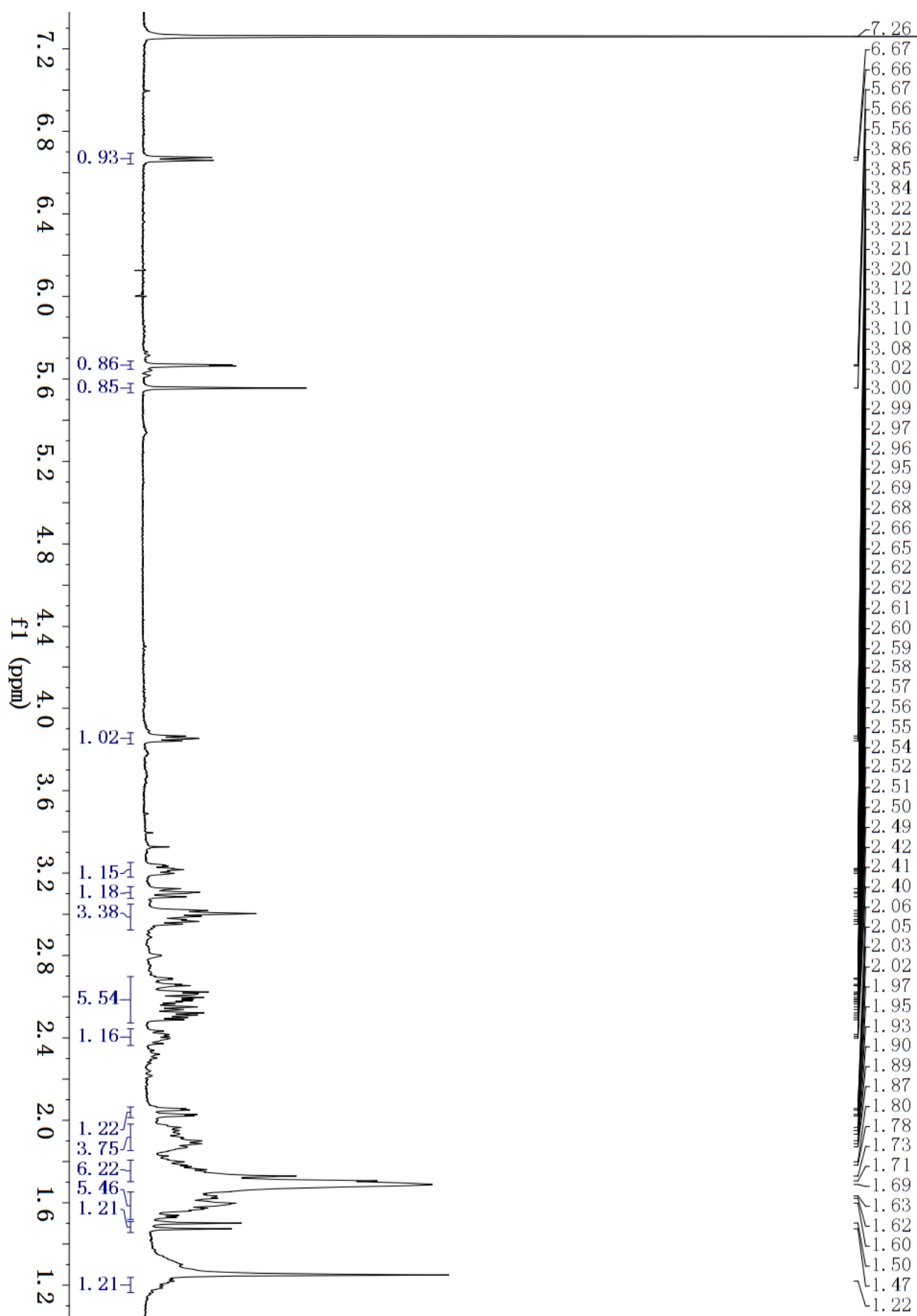


Figure S20. ^{13}C NMR spectrum for flueggenine G (3) in CDCl_3 .

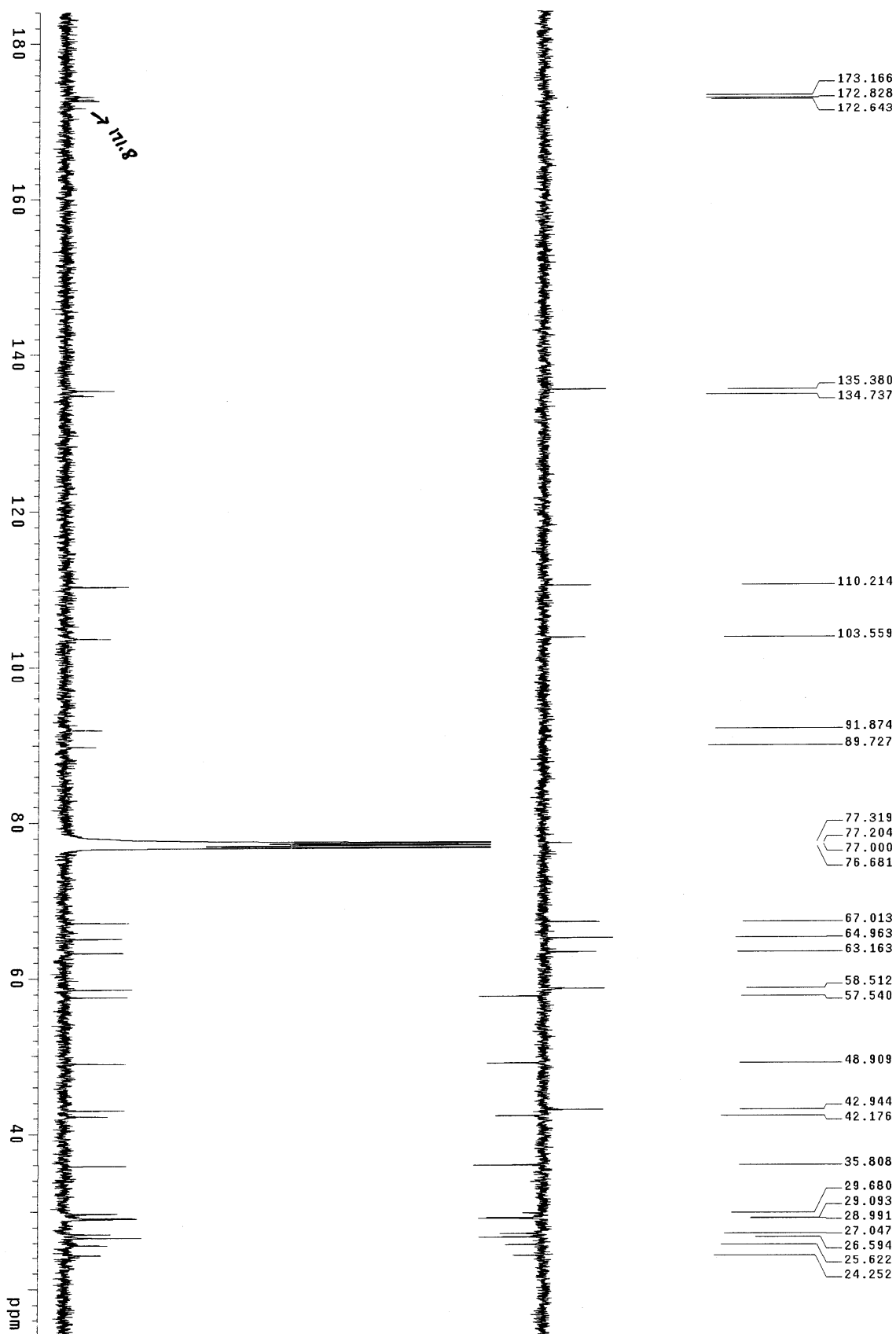


Figure S21. ^1H - ^1H COSY spectrum for flueggeine G (**3**) in CDCl_3 .

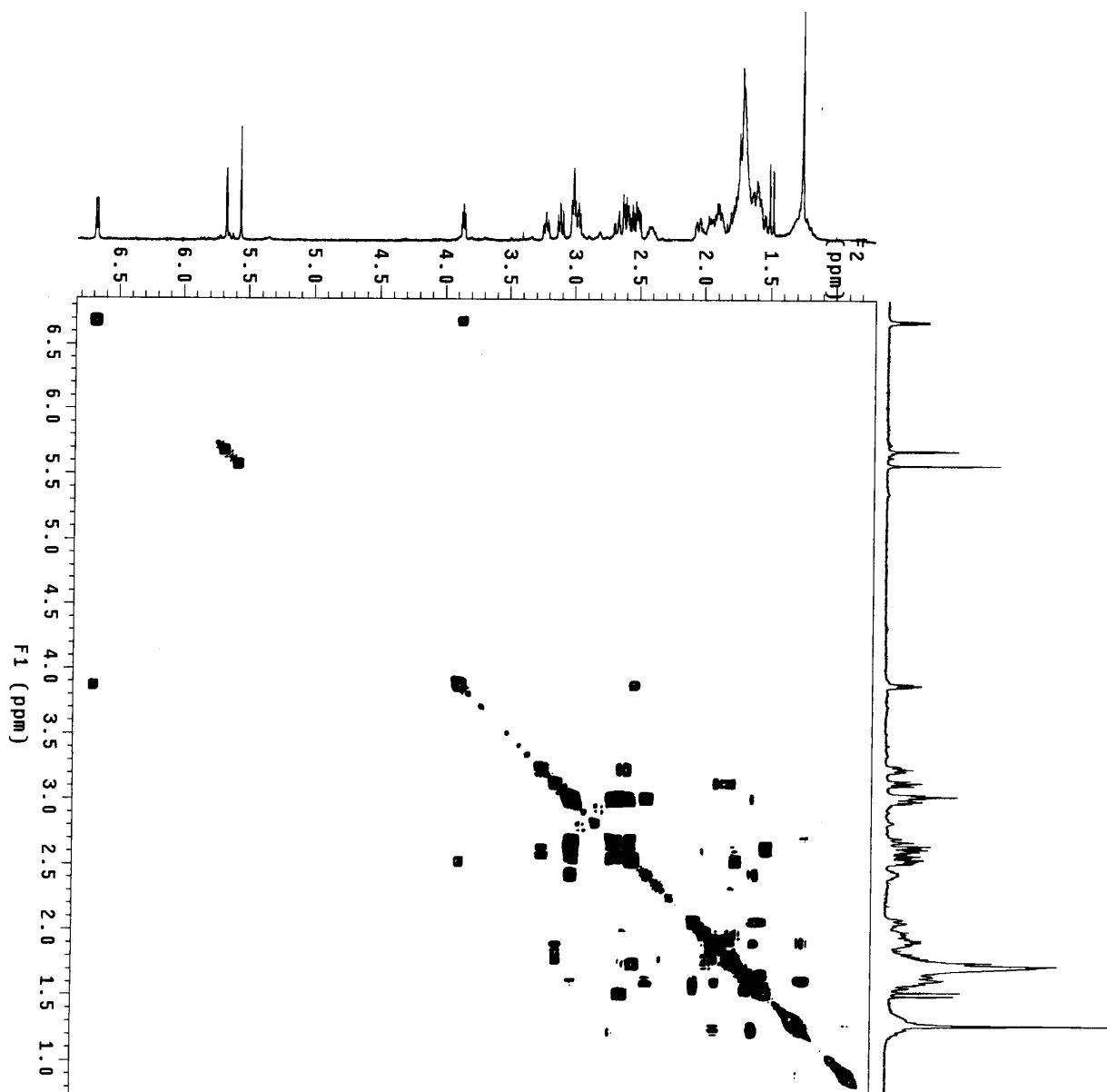
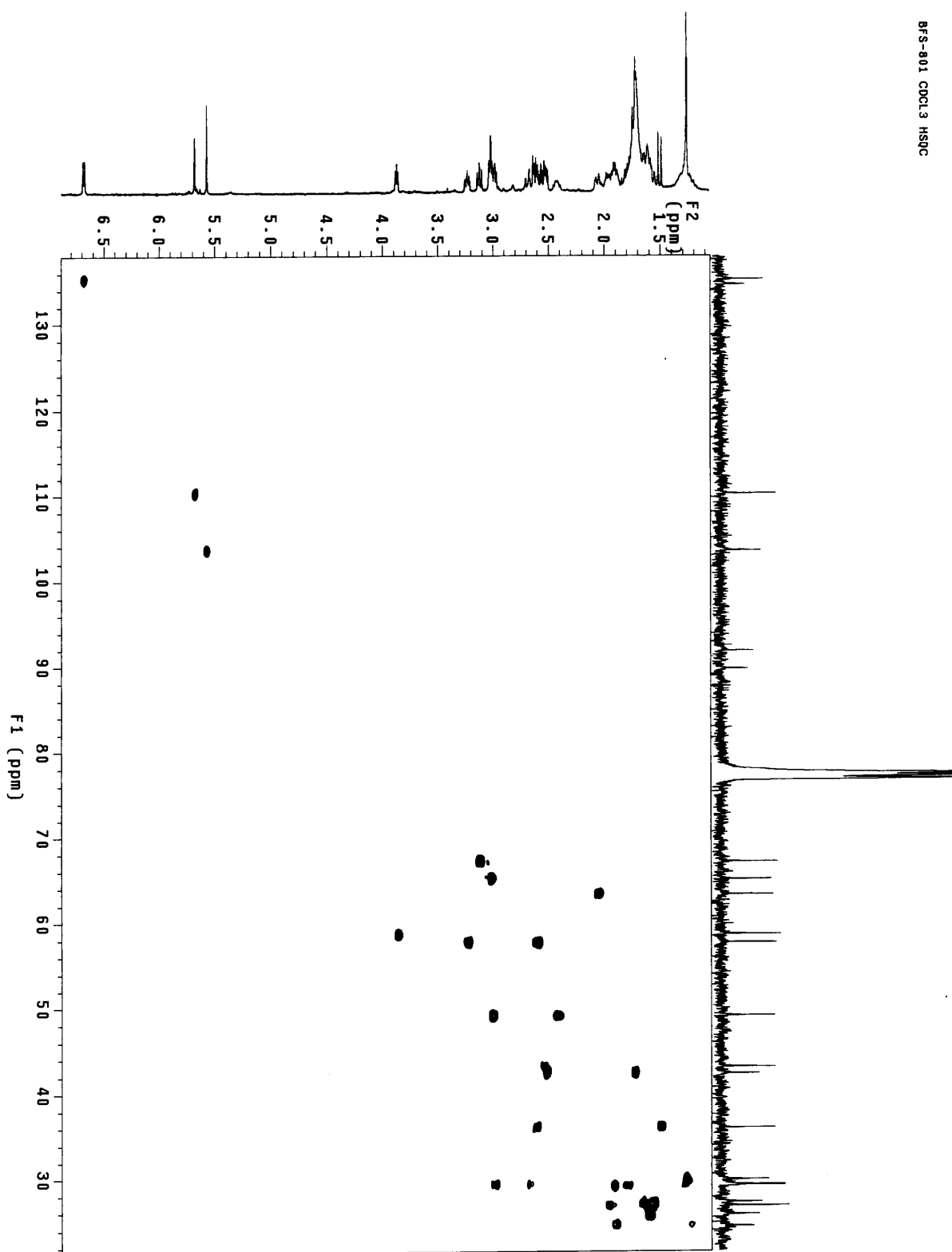
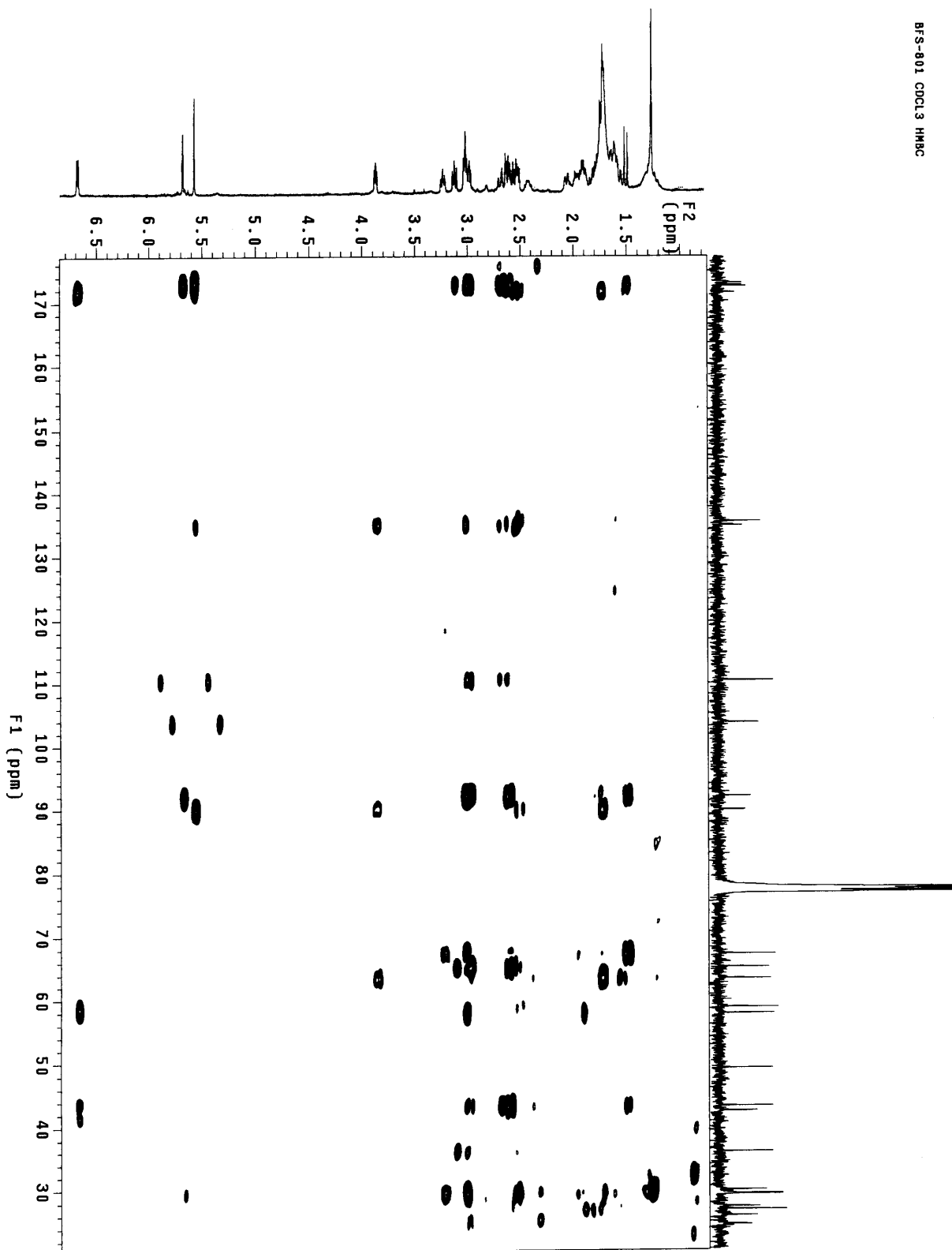


Figure S22. HSQC spectrum for flueggenine G (3) in CDCl₃.



BFS-801 CDCL3 HSQC

Figure S23. HMBC spectrum for flueggein G (3) in CDCl₃.



BFS-801 CDCl3 HMBC

Figure S24. ROESY spectrum for flueggenine G (3) in CDCl₃

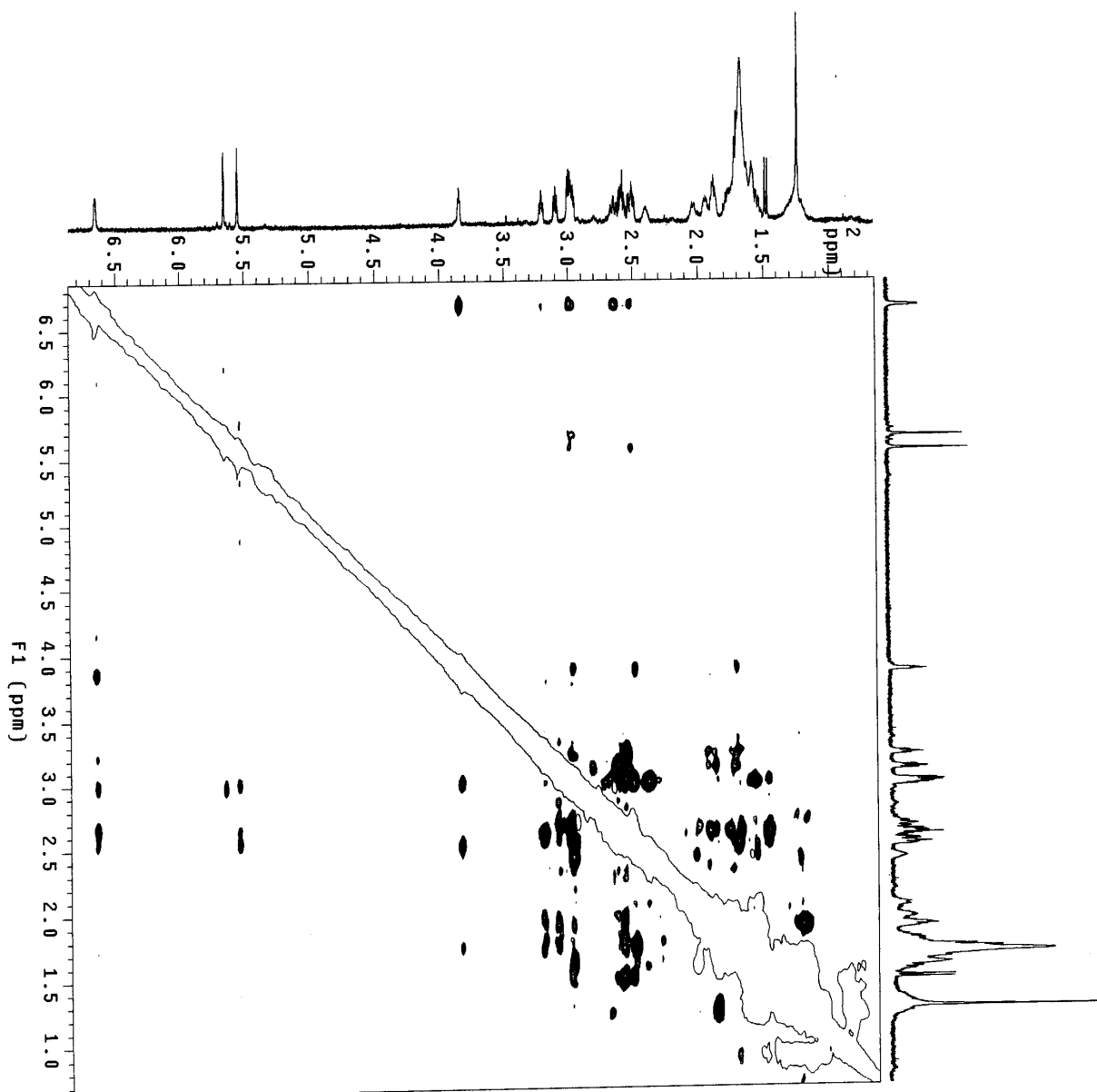


Figure S25. IR spectrum for flueggeine G (3).

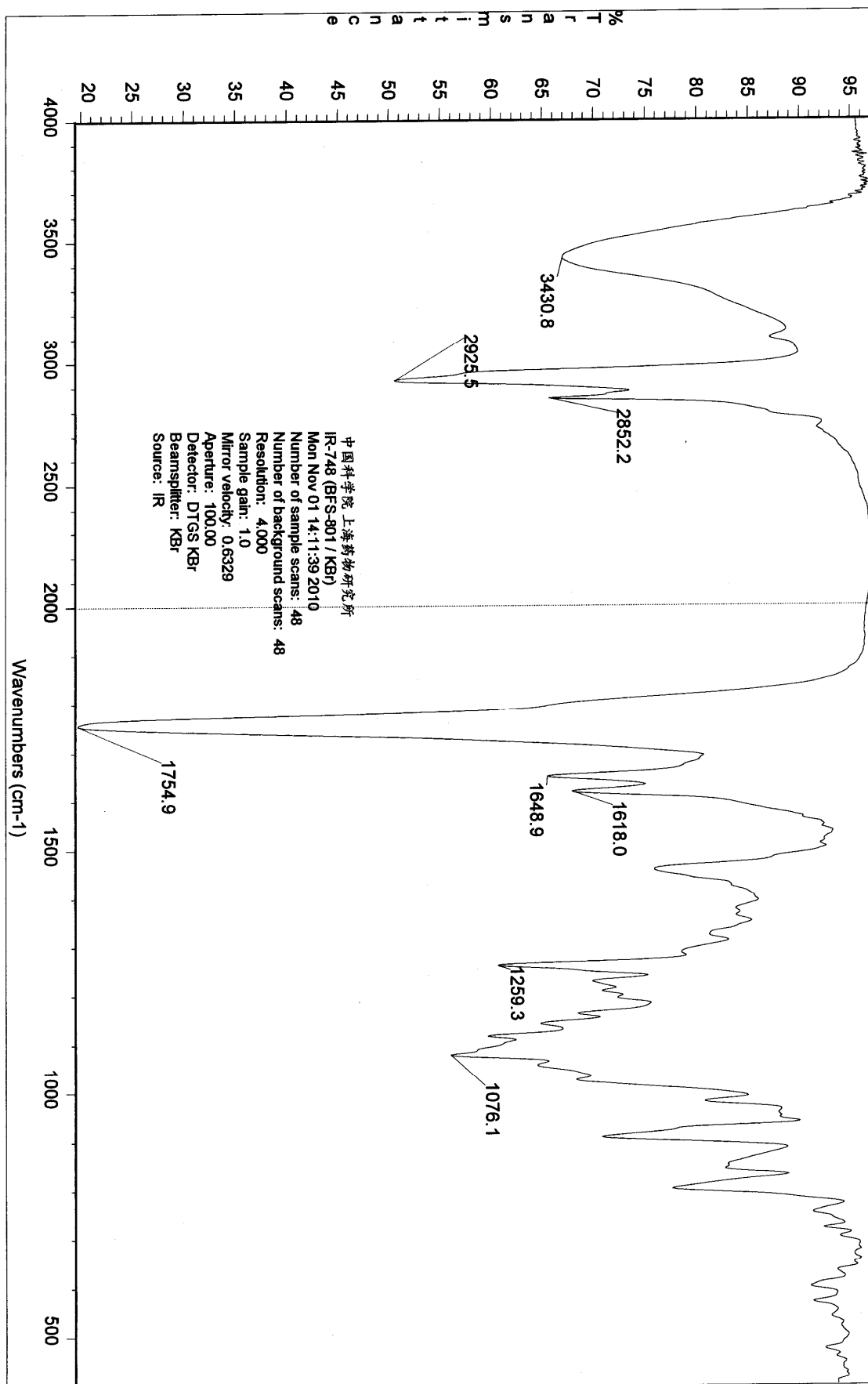


Figure S26. EIMS spectrum for flueggeine G (3).

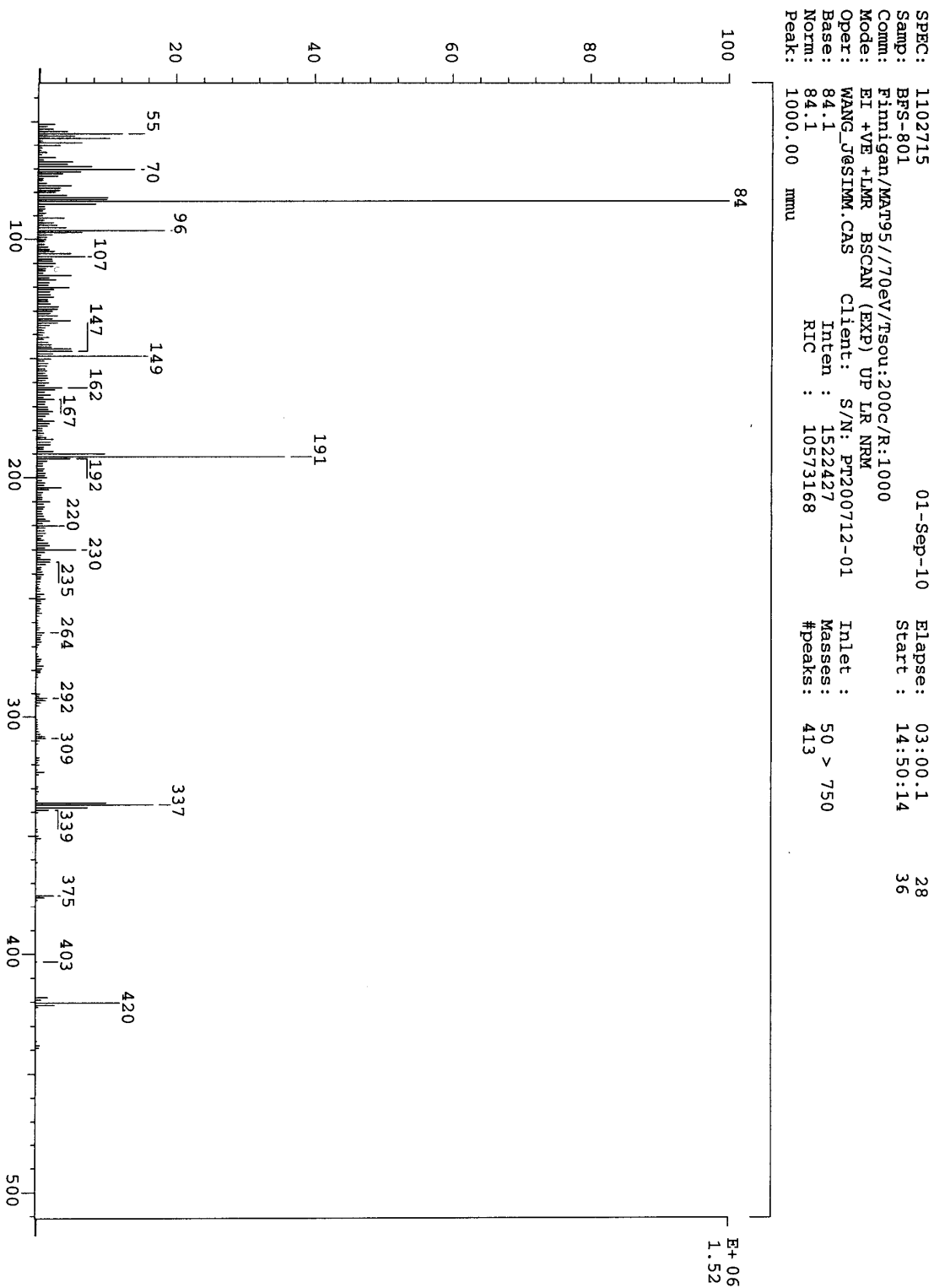


Figure S27. HREIMS spectrum for flueggeine G (3).

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 Limt: (0)
 : (533) C27.H100.N2.O5
 Peak: 1000.00 mmu R+D: -2.0 > 60.0
 Data: CMASS : converted

Mass	Intensity	%RA	%RIC	Delta	R+D	Composition
70.06583	* 37171	15.64	0.15	-0.2	1.5	C4.H8.N
71.08595	* 12883	5.42	0.05	0.1	0.5	C5.H11
77.03990	* 9041	3.80	0.04	-0.8	4.5	C6.H5
78.04795	* 9455	3.98	0.04	-1.0	4.0	C6.H6
82.06525	* 19679	8.28	0.08	0.4	2.5	C5.H8.N
					-2.3	-2.0 C2.H10.O3
83.07255	* 12469	5.25	0.05	0.9	2.0	C5.H9.N
83.08556	* 9219	3.88	0.04	0.5	1.5	C6.H11
84.08089	* 237627	100.00	0.98	0.4	1.5	C5.H10.N
85.08450	* 9041	3.80	0.04			
91.05436	* 8037	3.38	0.03	0.4	4.5	C7.H7
96.08174	* 40126	16.89	0.17	-0.4	2.5	C6.H10.N
106.0397	* 9100	3.83	0.04	-1.9	1.0	C2.H6.N2.O3
					2.1	5.0 C7.H6.O
107.0719	* 11582	4.87	0.05	-1.1	-0.5	C4.H11.O3
					1.6	4.0 C7.H9.N
115.0545	* 9573	4.03	0.04	0.3	6.5	C9.H7
146.0970	* 8096	3.41	0.03	0.0	5.5	C10.H12.N
					-2.7	1.0 C7.H14.O3
147.1043	* 7741	3.26	0.03	0.5	5.0	C10.H13.N
					-2.1	0.5 C7.H15.O3
149.0231	* 21452	9.03	0.09	0.8	6.5	C8.H5.O3
162.0912	* 7446	3.13	0.03	0.7	5.5	C10.H12.N.O
					-2.0	1.0 C7.H14.O4
190.0874	* 26593	11.19	0.11	-0.6	6.5	C11.H12.N.O2
191.0949	* 85985	36.18	0.36	-0.3	6.0	C11.H13.N.O2
					-2.9	1.5 C8.H15.O5
192.0991	* 8982	3.78	0.04	0.6	1.0	C8.H16.O5
230.1187	* 11110	4.68	0.05	-0.6	7.5	C14.H16.N.O2
258.9797	* 11346	4.77	0.05			
308.9754	* 11760	4.95	0.05			
336.1247	* 19679	8.28	0.08	-1.2	12.5	C20.H18.N.O4
					1.5	17.0 C23.H16.N2.O
337.1310	* 45858	19.30	0.19	0.5	12.0	C20.H19.N.O4
338.1350	* 18851	7.93	0.08			
358.9720	* 11110	4.68	0.05			
359.9829	* 7446	3.13	0.03	1.9	27.0	C26.O3
					-2.1	23.0 C21.N2.O5
367.9784	* 8037	3.38	0.03			
405.9799	* 12114	5.10	0.05	-2.2	27.5	C26.N.O5
420.2049	* 27716	11.66	0.11	0.0	13.0	C25.H28.N2.O4
420.9689	* 8273	3.48	0.03			

Figure S29. ^{13}C NMR spectrum for flueggein H (**4**) in CDCl_3 (5% CD_3OD).

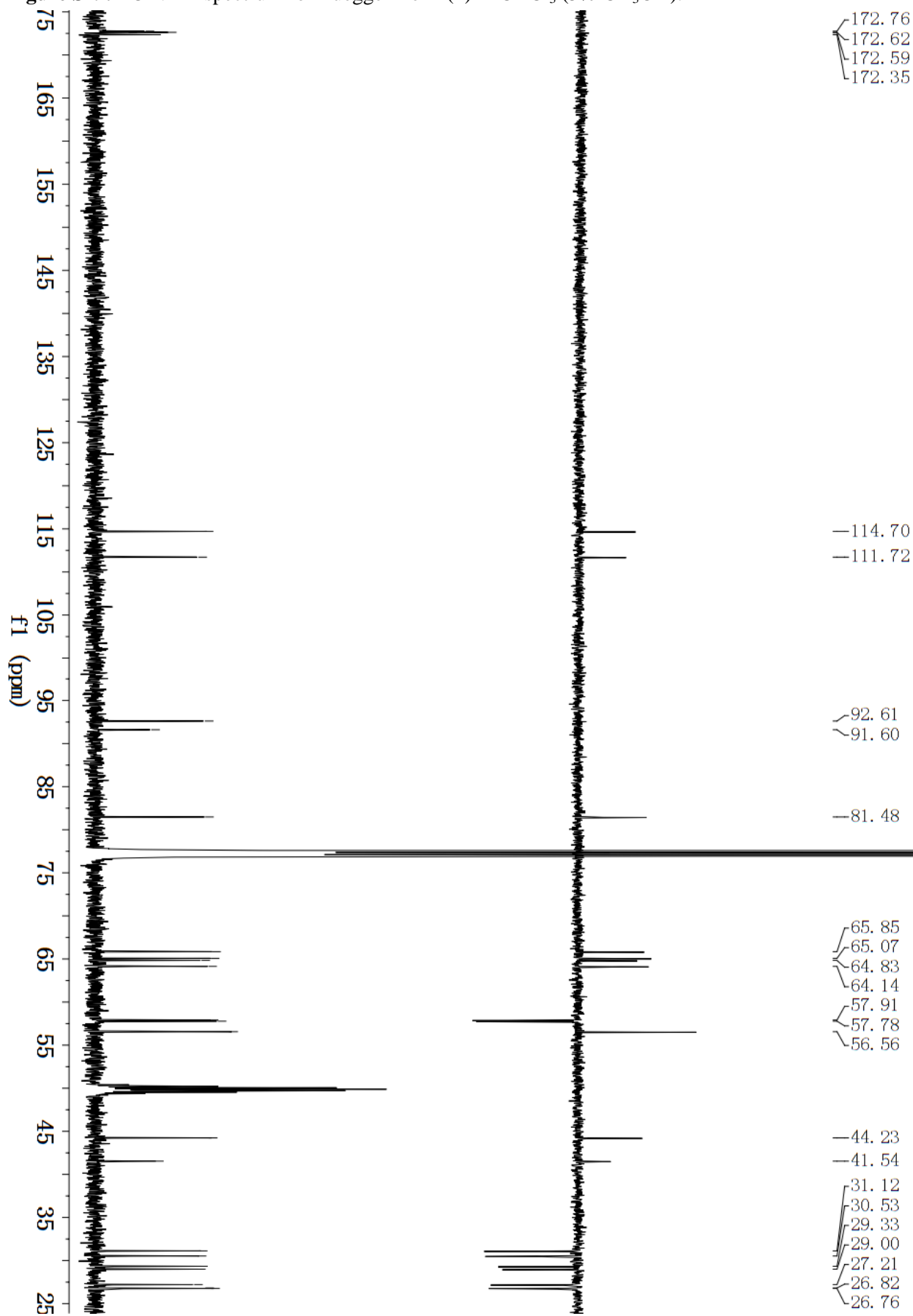


Figure S30. ^1H - ^1H COSY spectrum for flueggeine H (**4**) in CDCl_3 (5% CD_3OD).

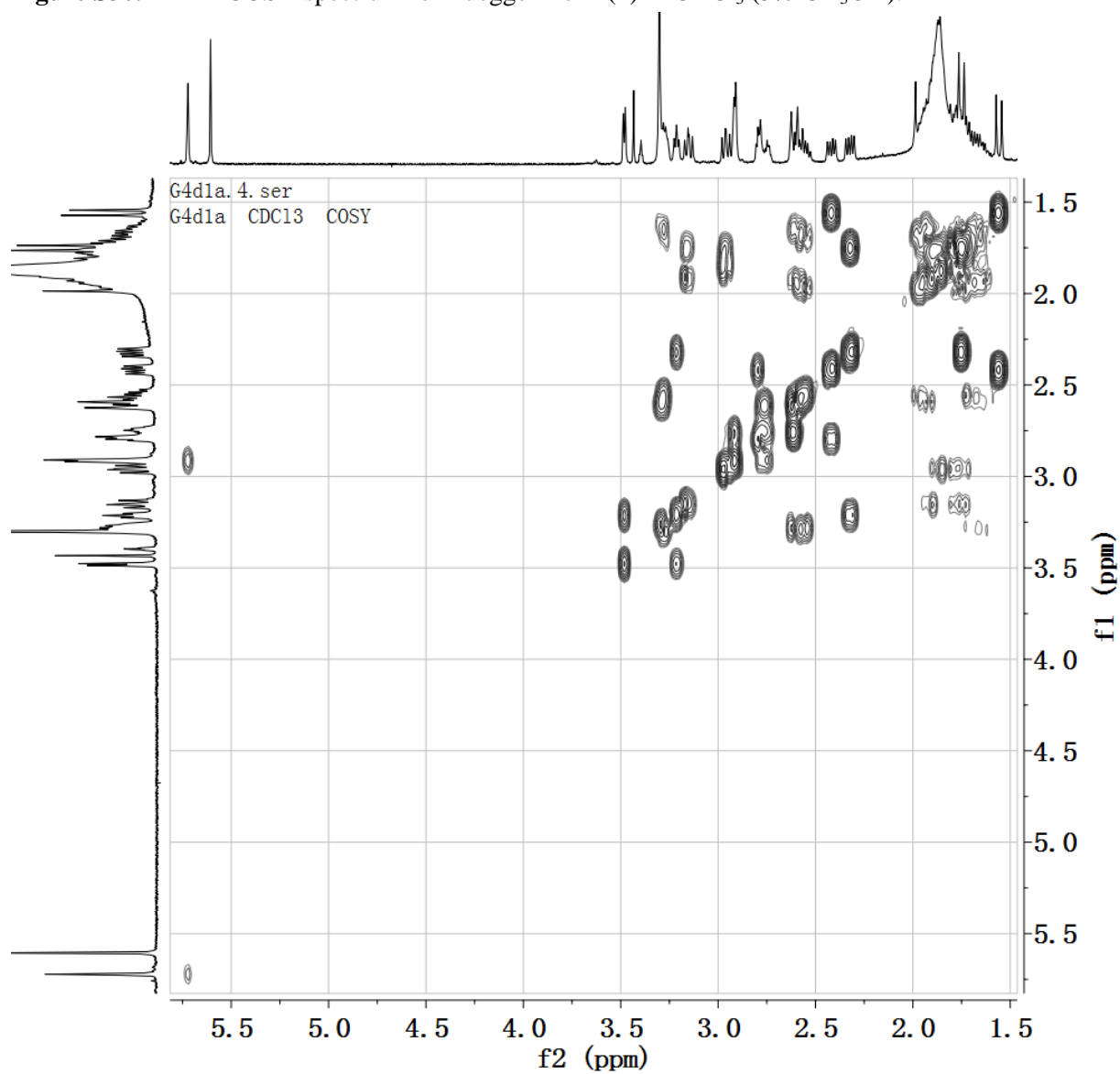


Figure S31. HSQC spectrum for flueggeine H (4) in CDCl₃ (5% CD₃OD).

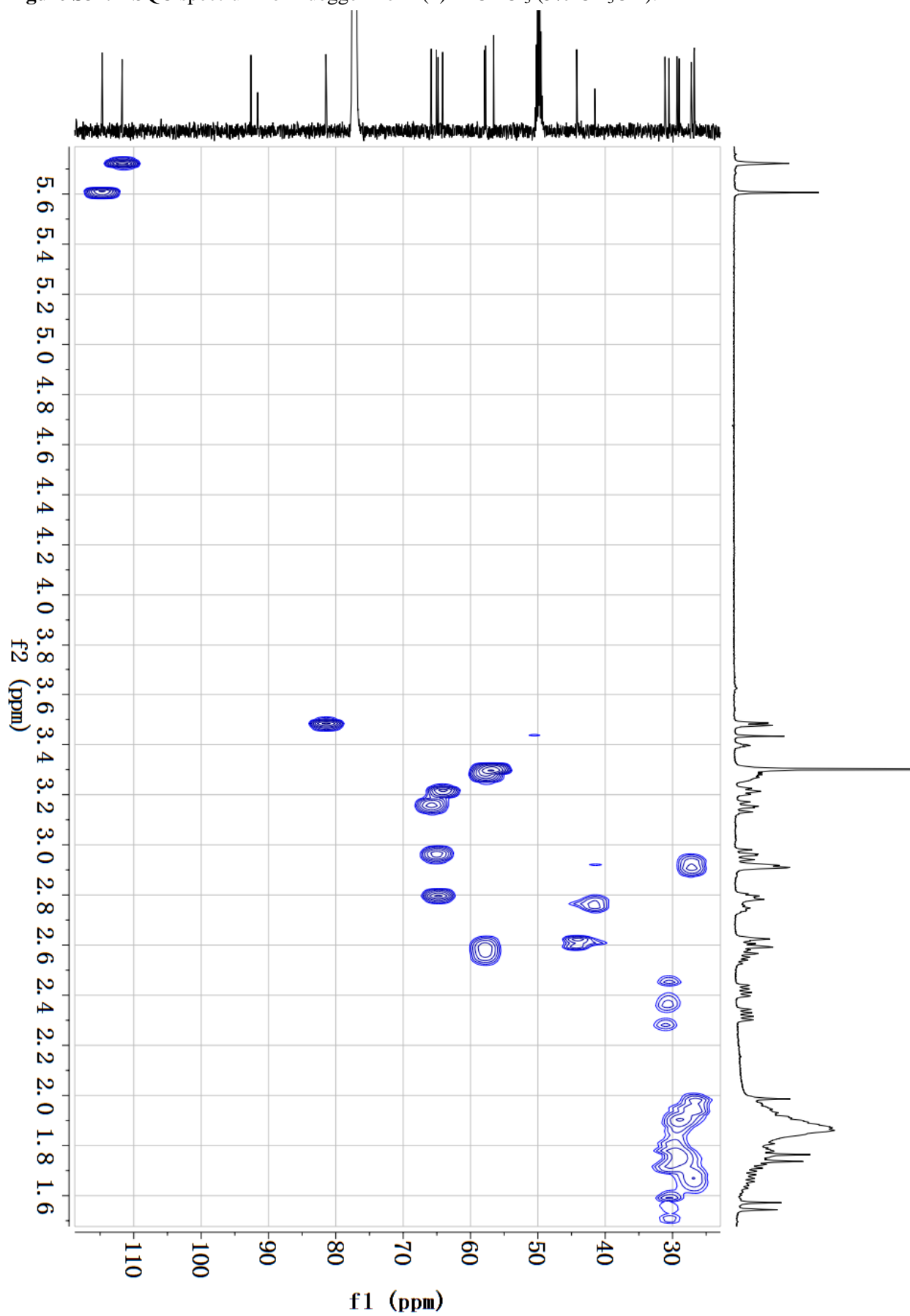


Figure S32. HMBC spectrum for flueggeine H (4) in CDCl₃ (5% CD₃OD).

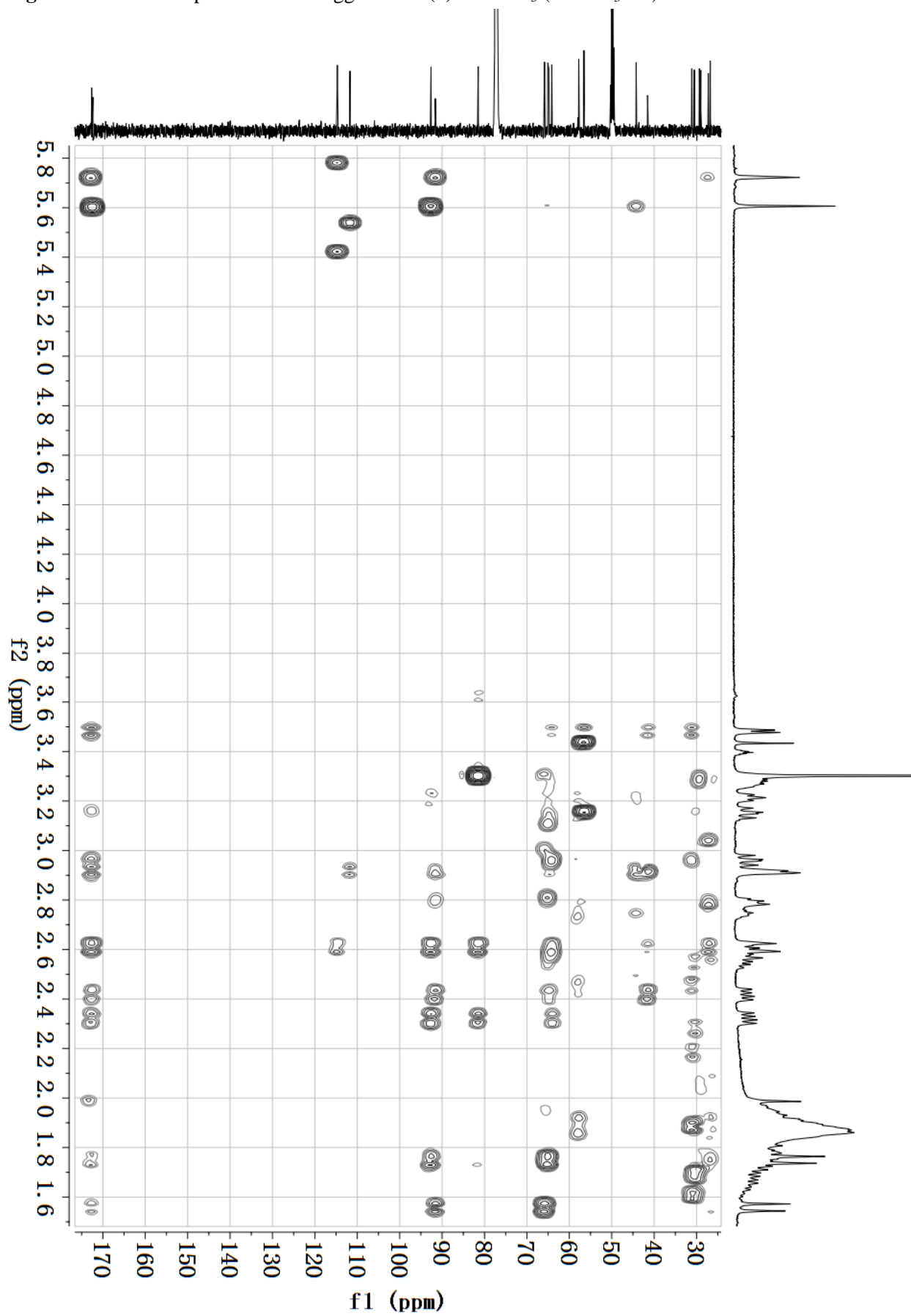


Figure S33. ROESY spectrum for flueggeine H (4) in CDCl₃ (5% CD₃OD).

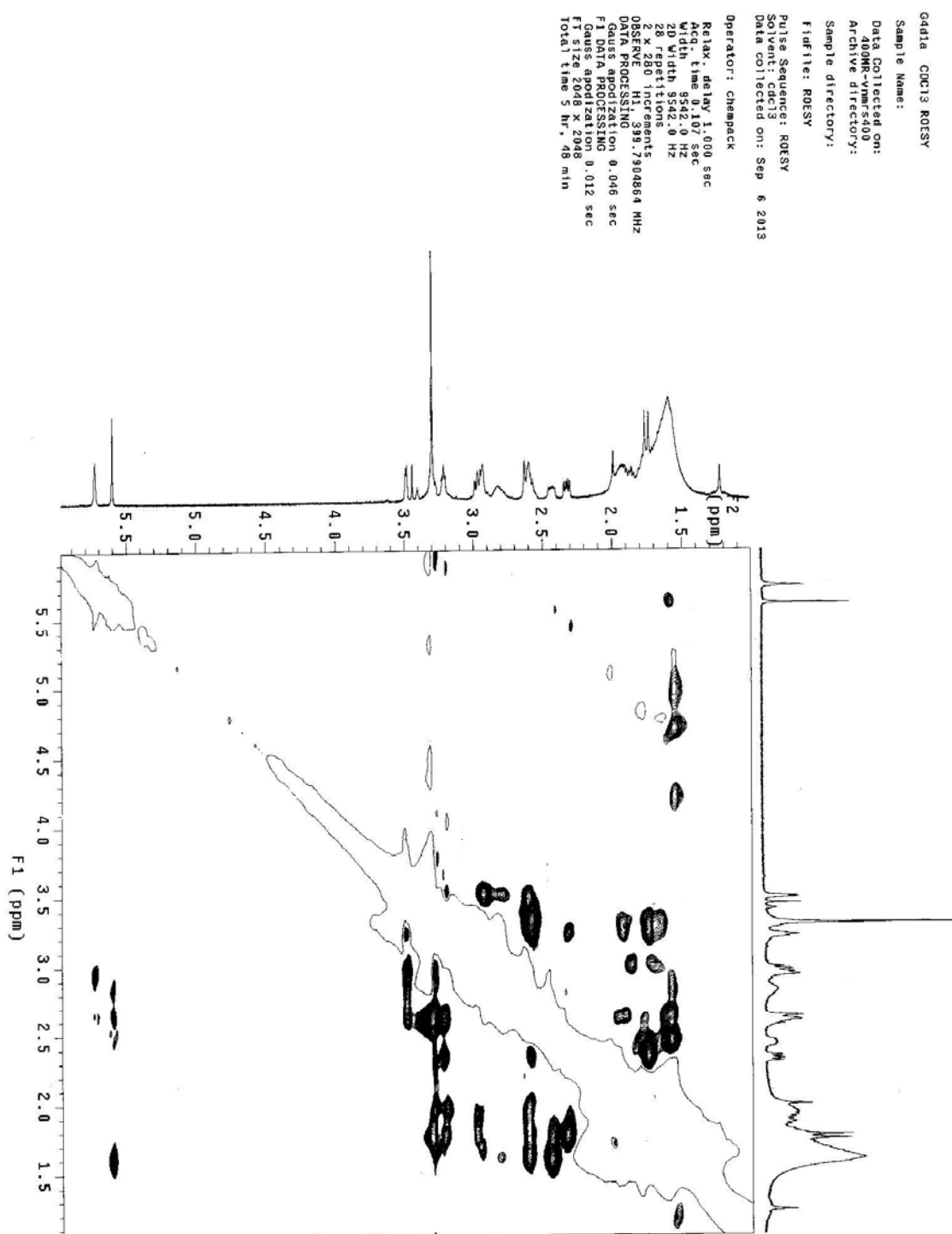


Figure S34. IR spectrum for flueggenine H (4).

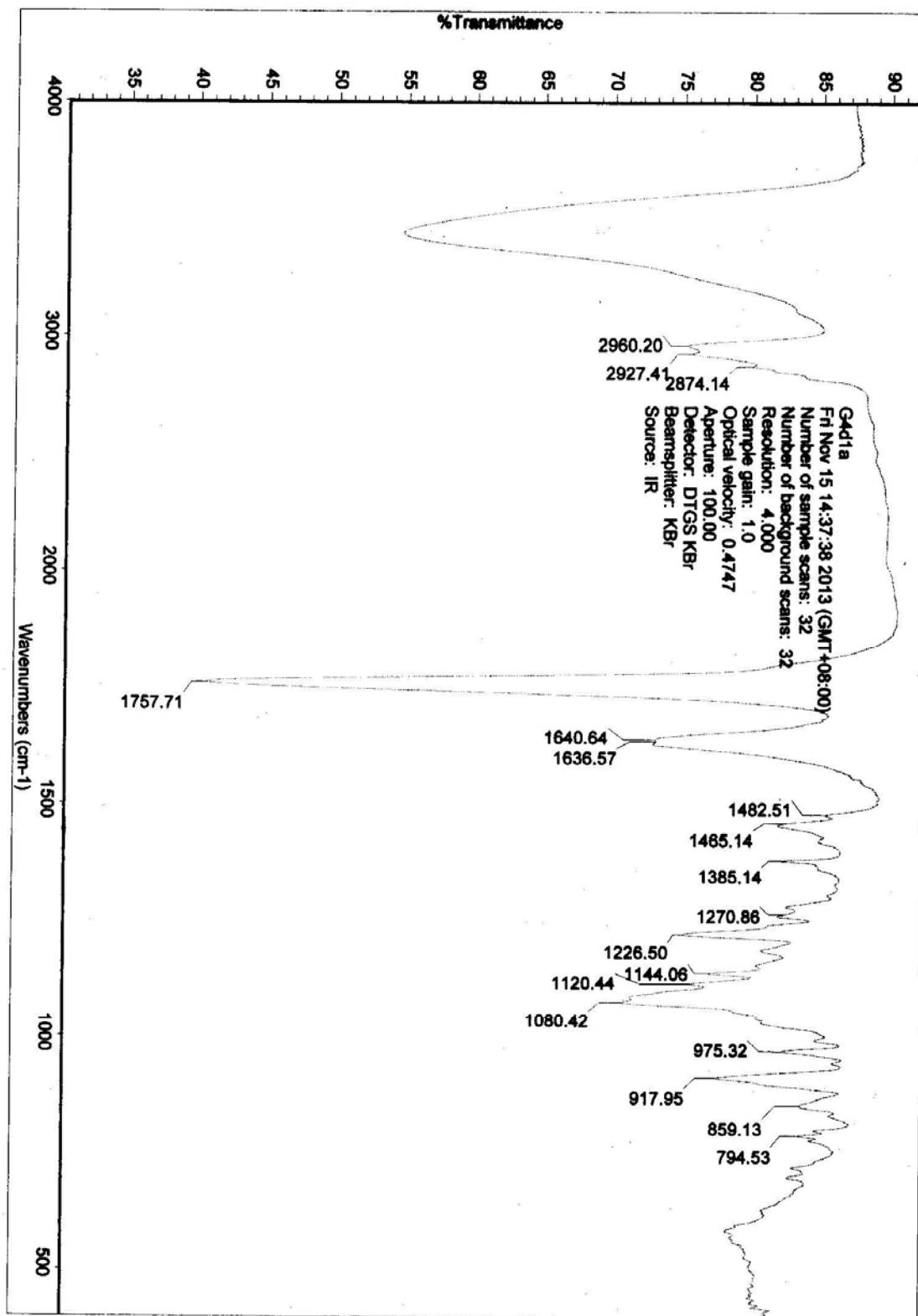


Figure S35. (+)-ESIMS spectrum for flueggeanine H (4).

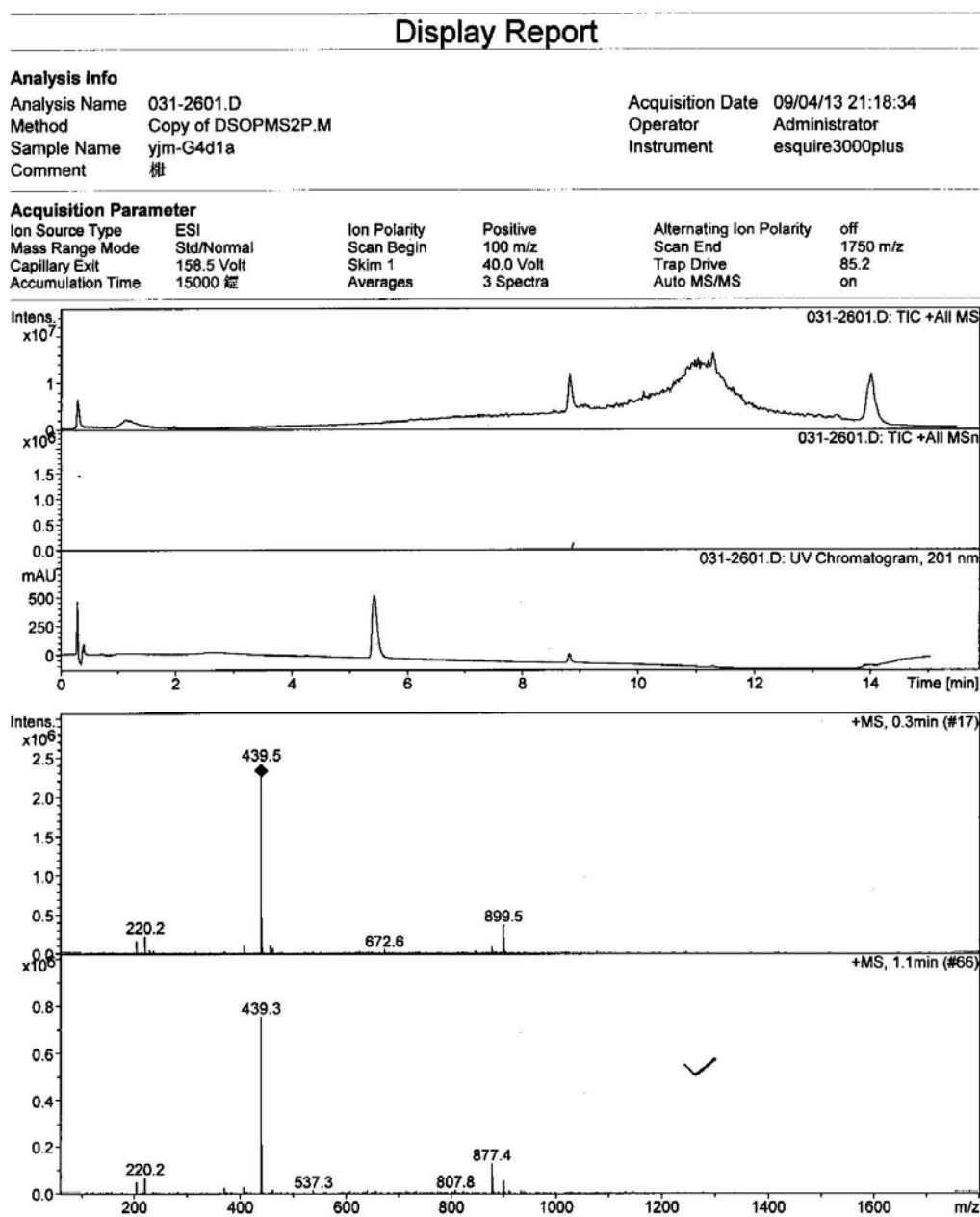


Figure S36. (+)-HRESIMS spectrum for flueggeine H (4).

Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

249 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 5-80 H: 2-120 N: 0-2 O: 0-20

G4d1a

LCT PXE KE324

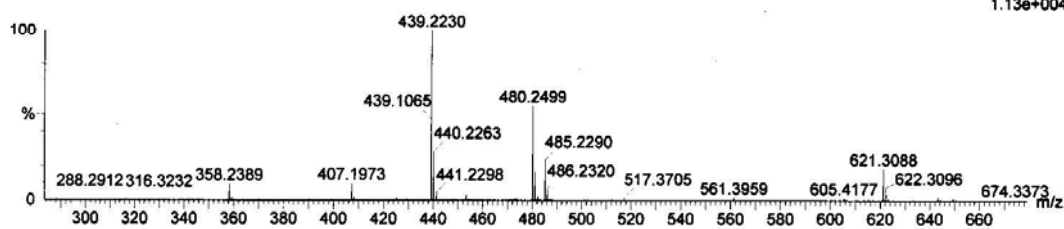
13-Sep-2013

15:12:40

G4d1a_0913 5 (0.107) AM2 (Ar,10000.0,0.00,1.00); ABS; Cm (5.21)

1: TOF MS ES+

1.13e+004



Minimum:

Maximum: 3.0 5.0 -1.5

Maximum: 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
439.2230	439.2233	-0.3	-0.7	11.5	114.2	0.0	C25 H31 N2 O5

Figure S37. ^1H NMR spectrum for flueggeine I (**5**) in CDCl_3 .

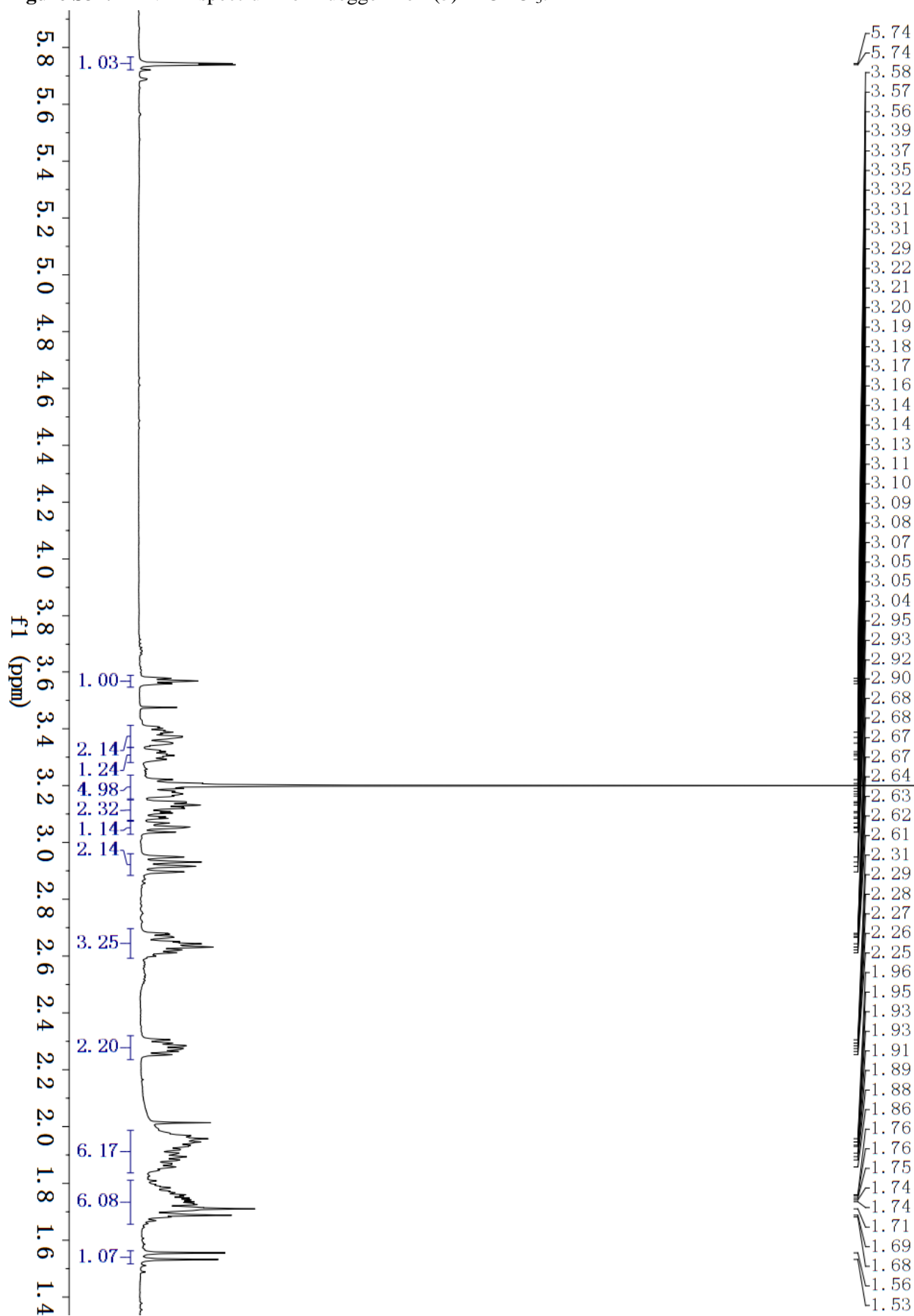


Figure S38. ^{13}C NMR spectrum for flueggein I (5) in CDCl_3 .

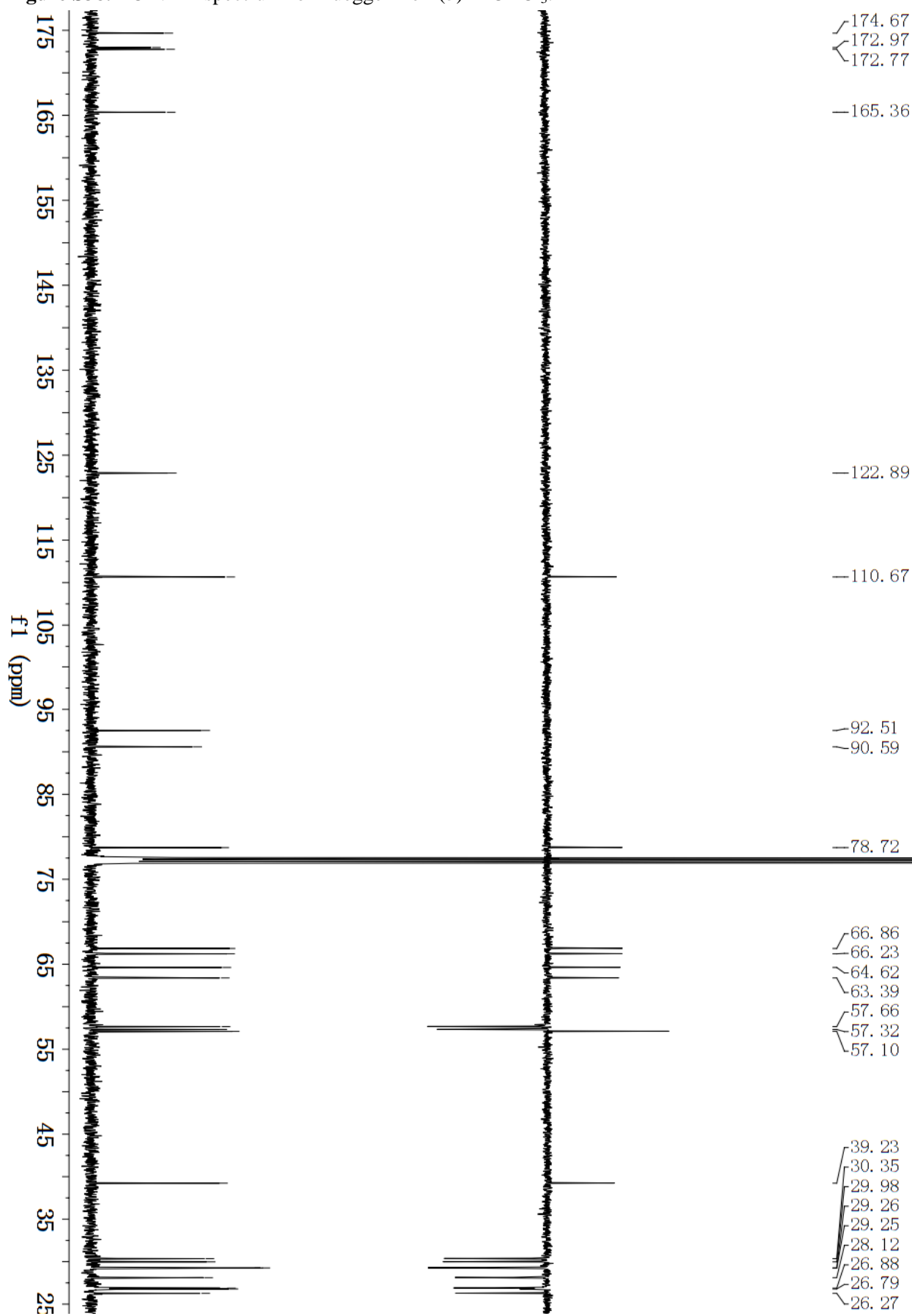


Figure S39. ^1H - ^1H COSY spectrum for flueggeine I (5) in CDCl_3 .

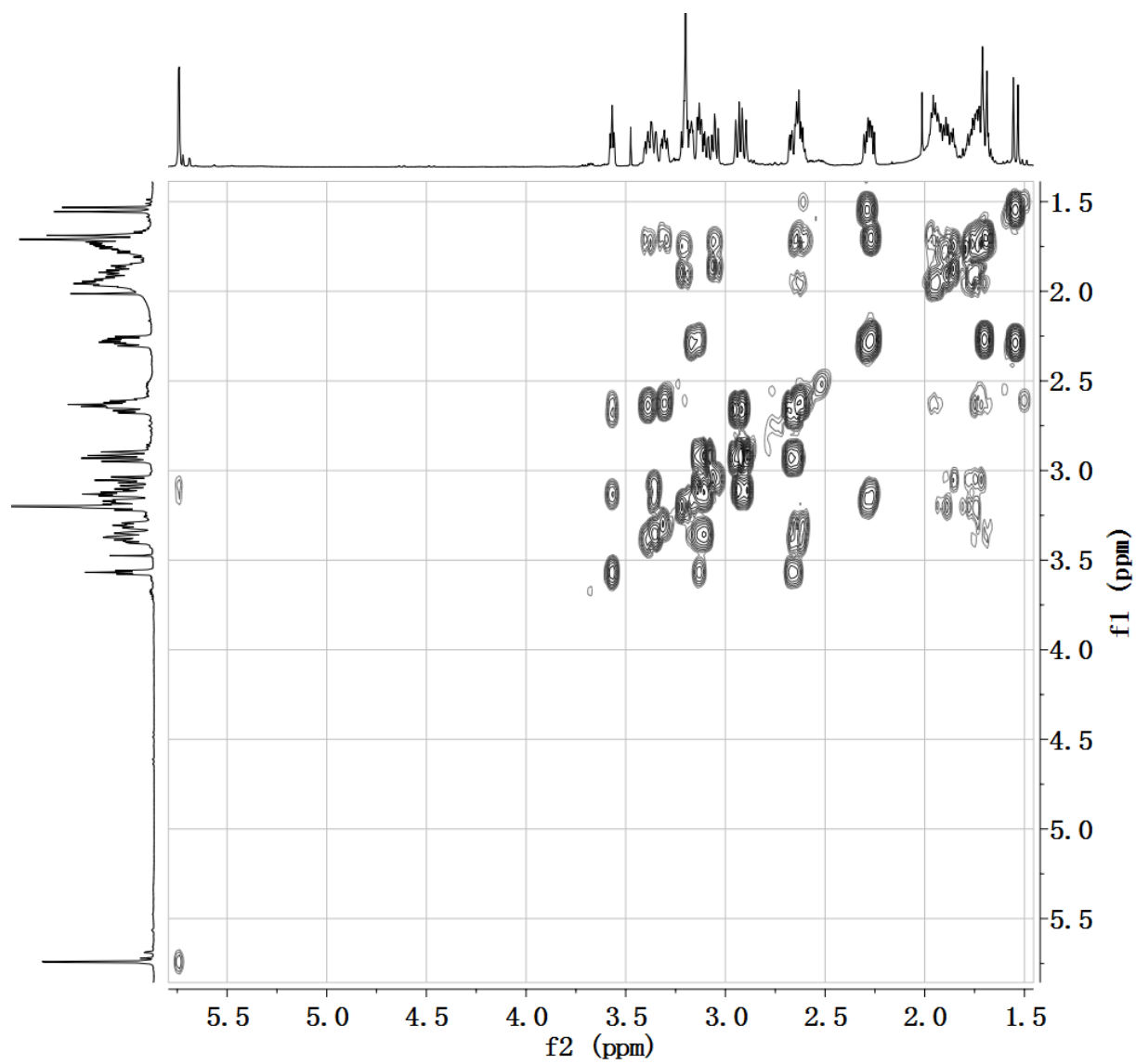


Figure S40. HSQC spectrum for flueggenine I (5) CDCl₃.

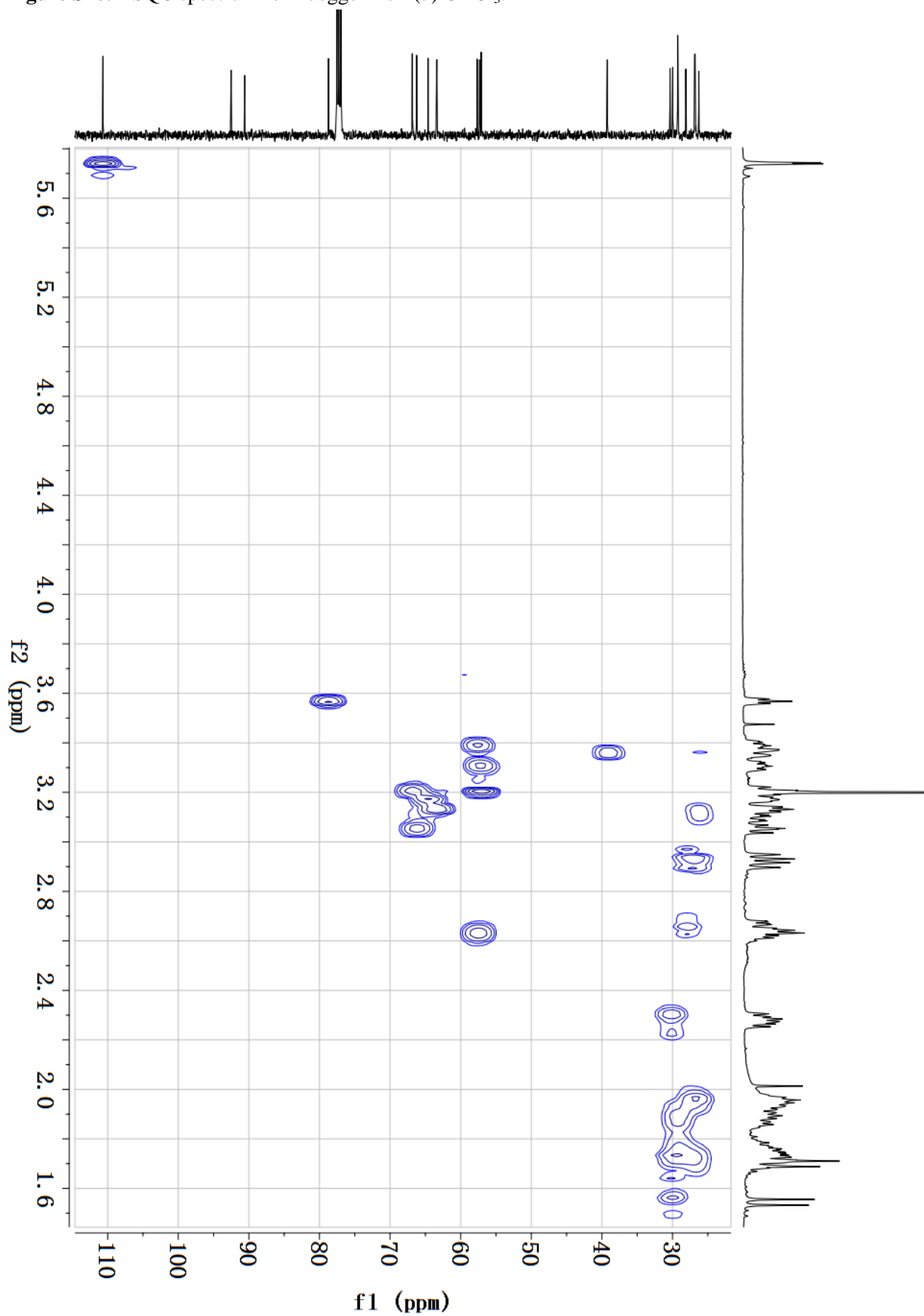


Figure S41. HMBC spectrum for flueggeine I (5) in CDCl₃.

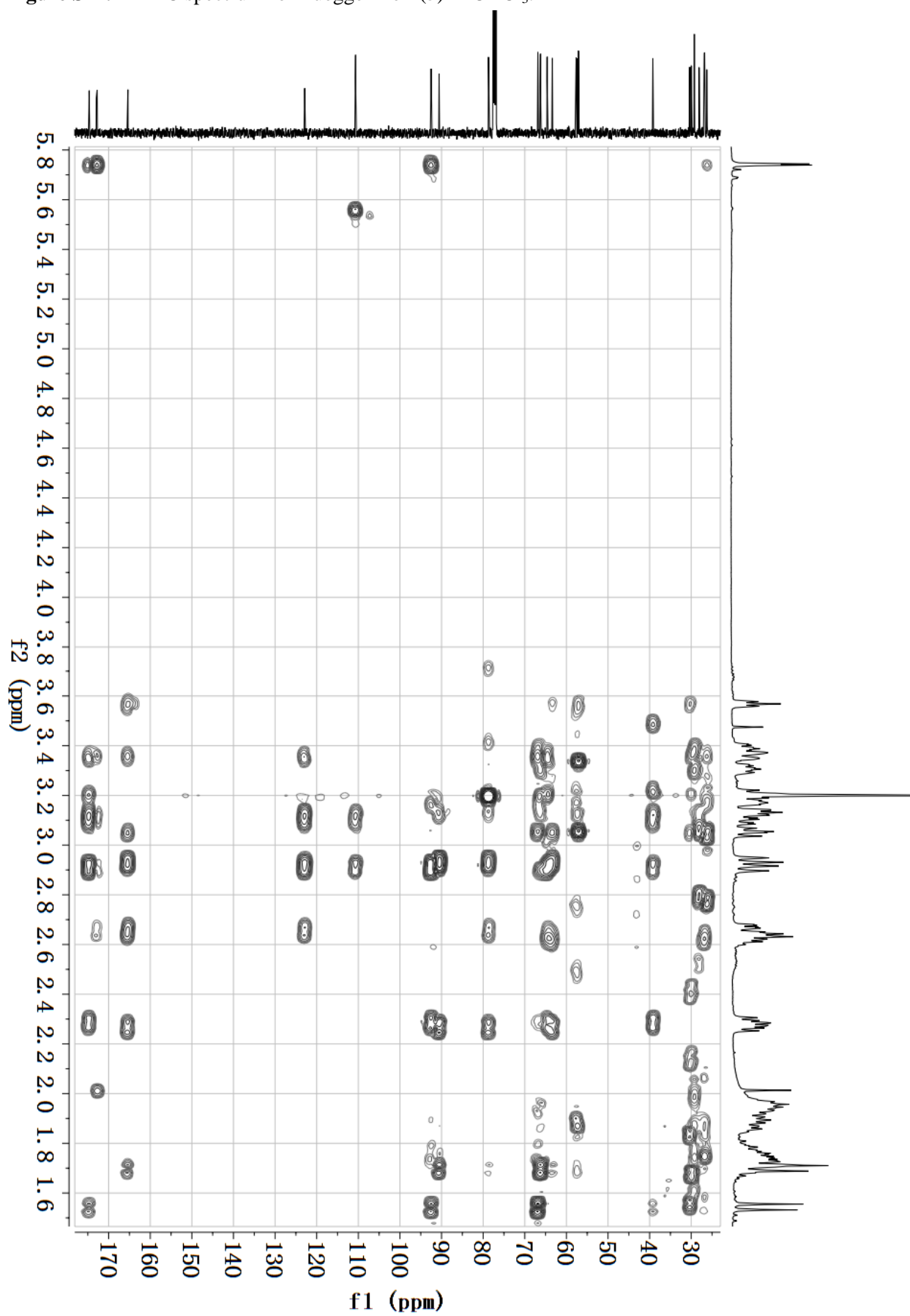


Figure S42. ROESY spectrum for flueggeine I (5) in CDCl₃.

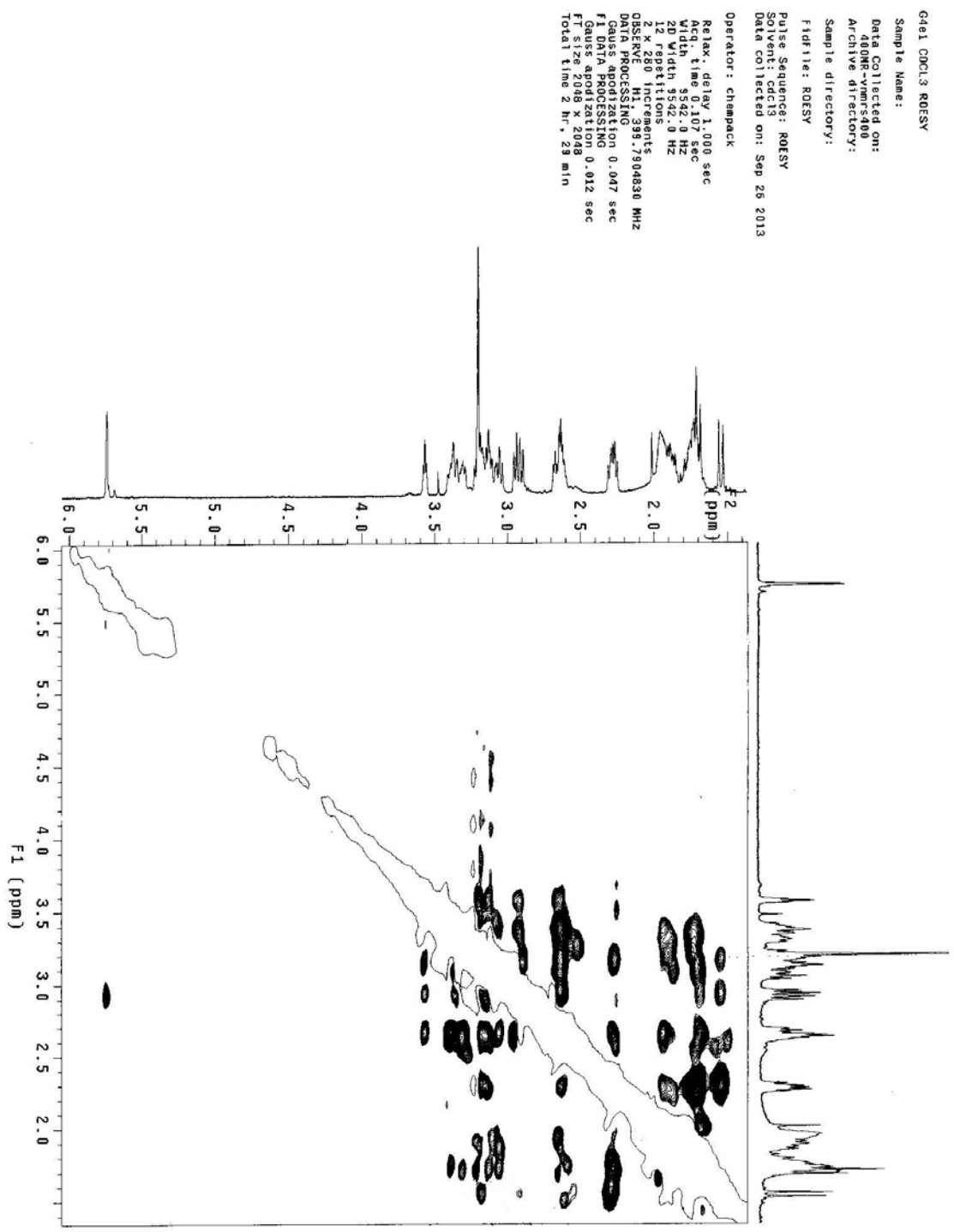


Figure S43. IR spectrum for flueggenine I (5).

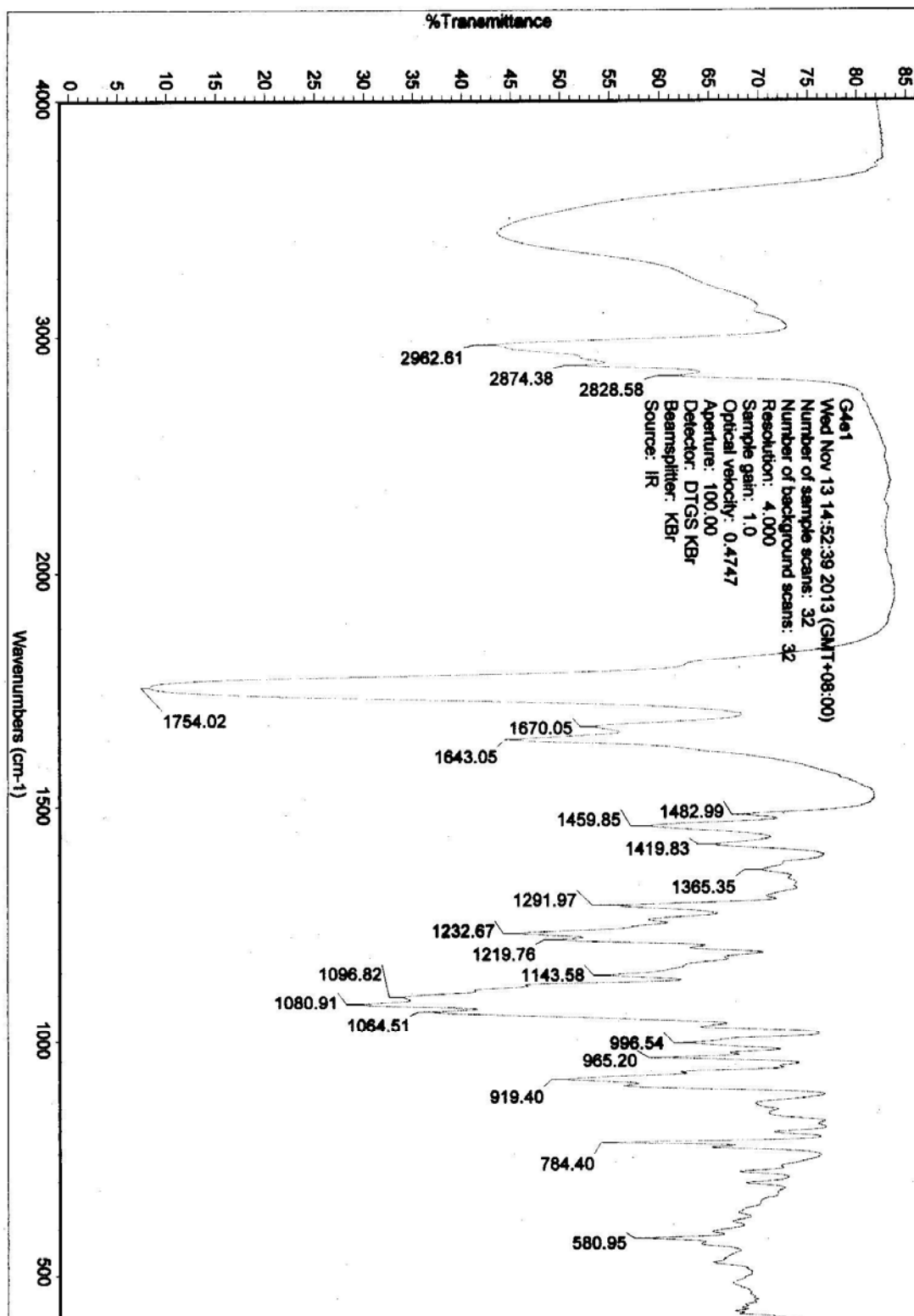


Figure S44. (+)-ESIMS spectrum for flueggeinone I (5).

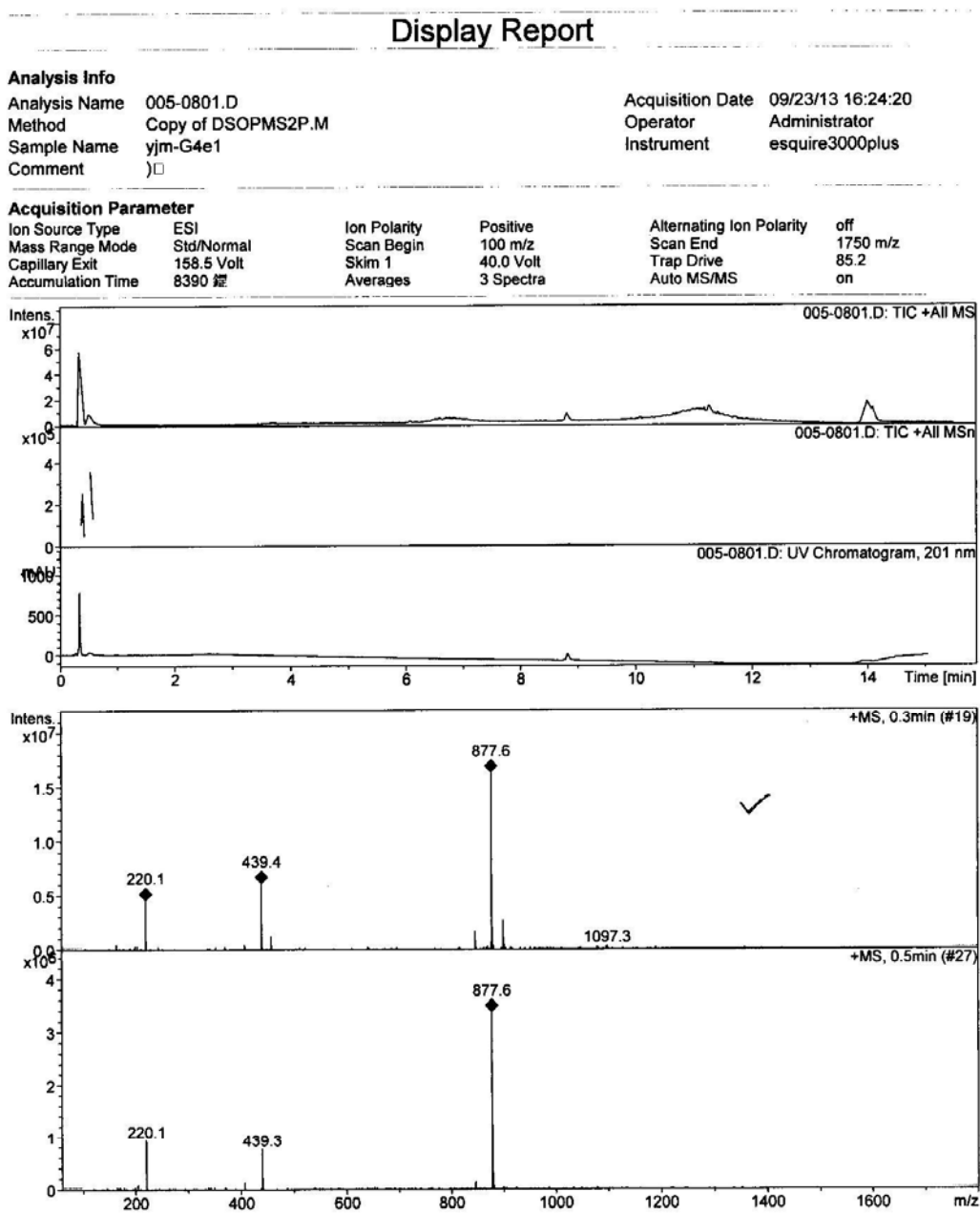


Figure S45. (+)-HRESIMS spectrum for flueggeine I (5).

Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

403 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 5-80 H: 2-120 N: 0-4 O: 0-20

G4ge1

LCT PXE KE324

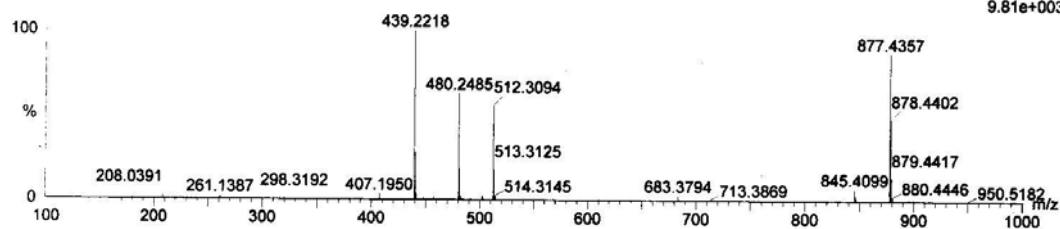
27-Sep-2013

10:22:18

1: TOF MS ES+

9.81e+003

G4e1_0927 27 (0.583) AM2 (Ar,10000.0,0.00,1.00); ABS; Cm (11:27)



Minimum: -1.5
Maximum: 3.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
439.2218	439.2233	-1.5	-3.4	11.5	81.6	0.0	C25 H31 N2 O5

Figure S46. ^1H NMR spectrum for flueviroisine E (**6**) in CDCl_3 .

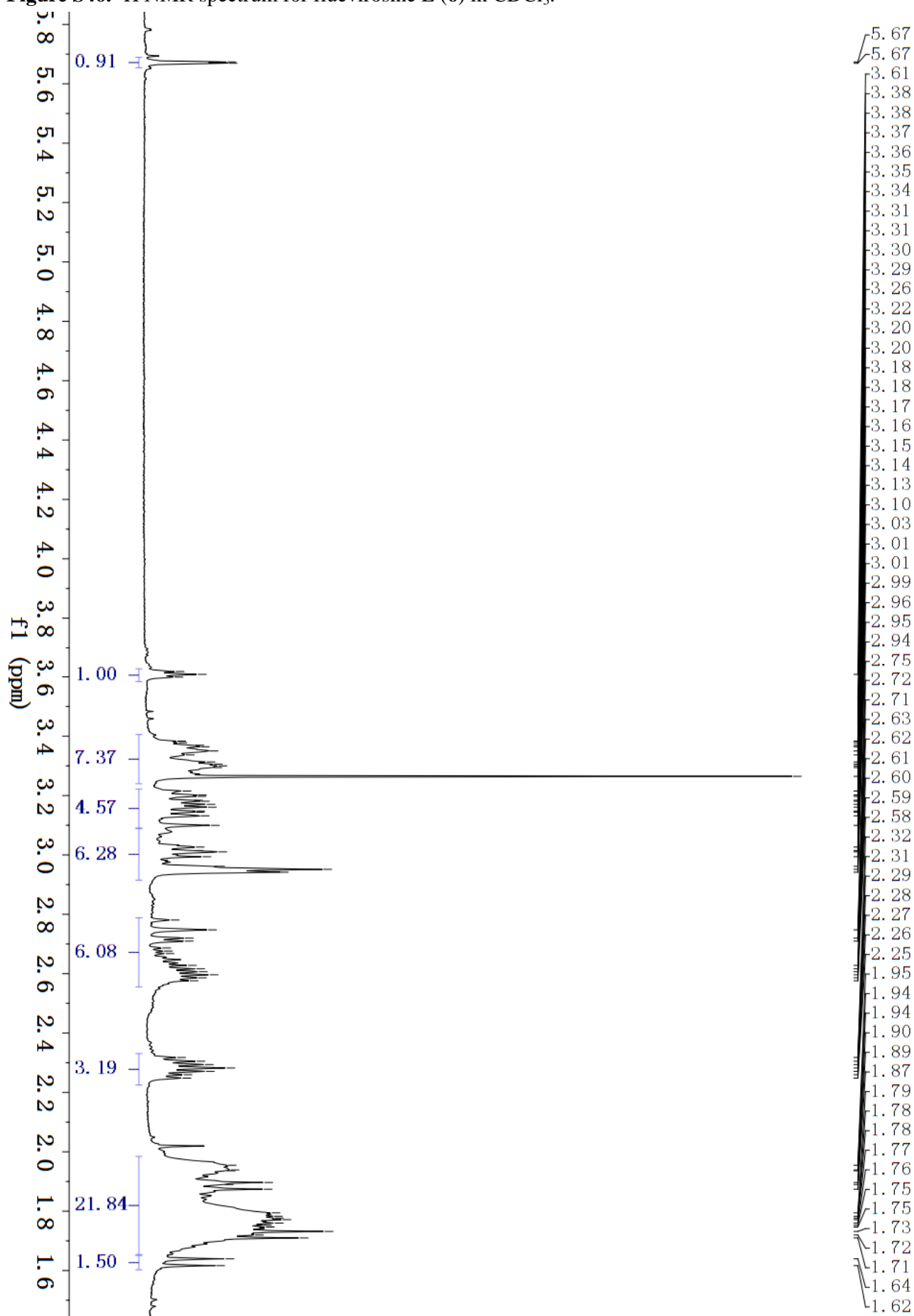


Figure S47. ^{13}C NMR spectrum for flueggein E (**6**) in CDCl_3 .

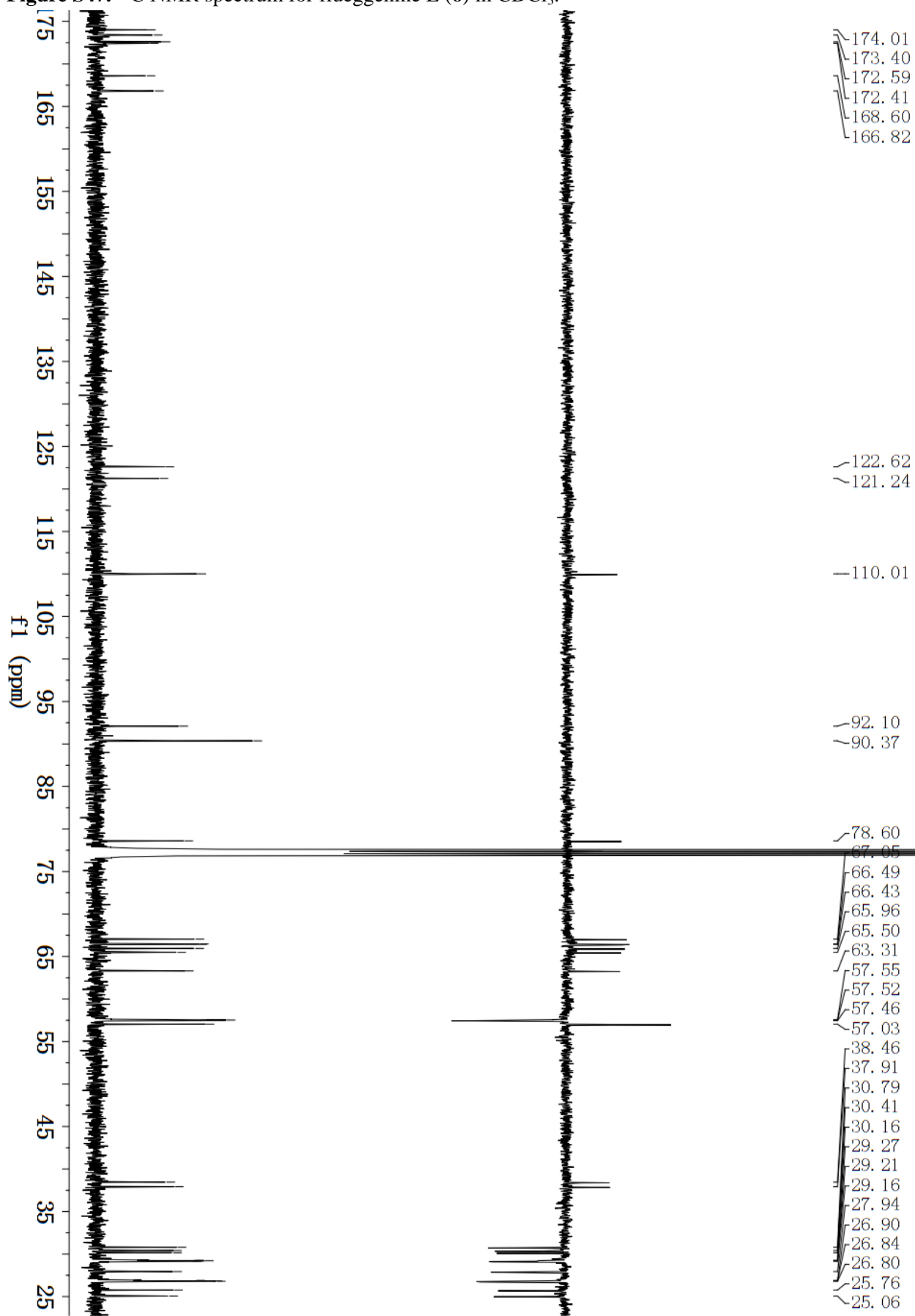


Figure S48. ^1H - ^1H COSY spectrum for flueggeine E (**6**) in CDCl_3 .

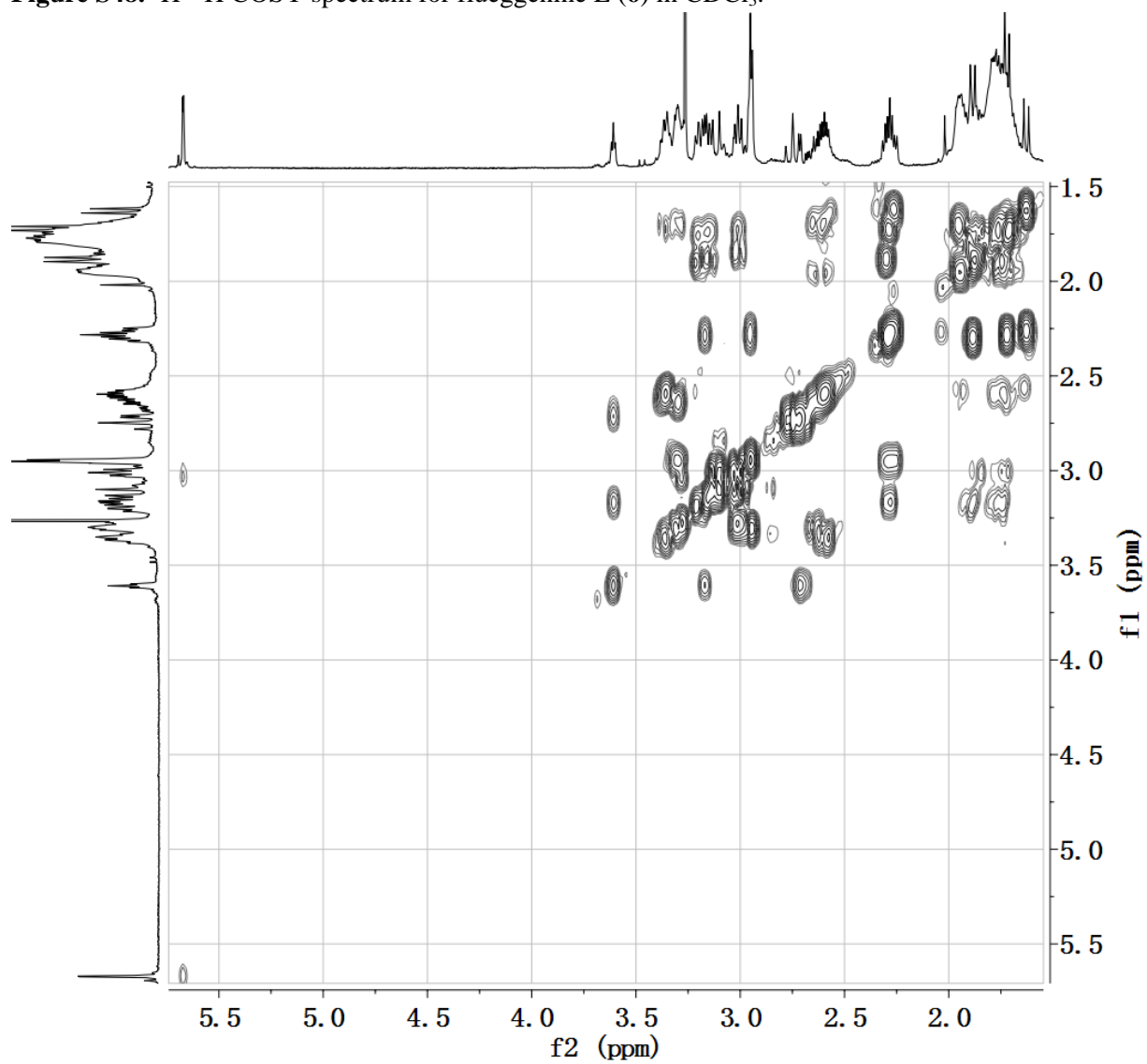


Figure S49. HSQC spectrum for flueggenine E (6) CDCl₃.

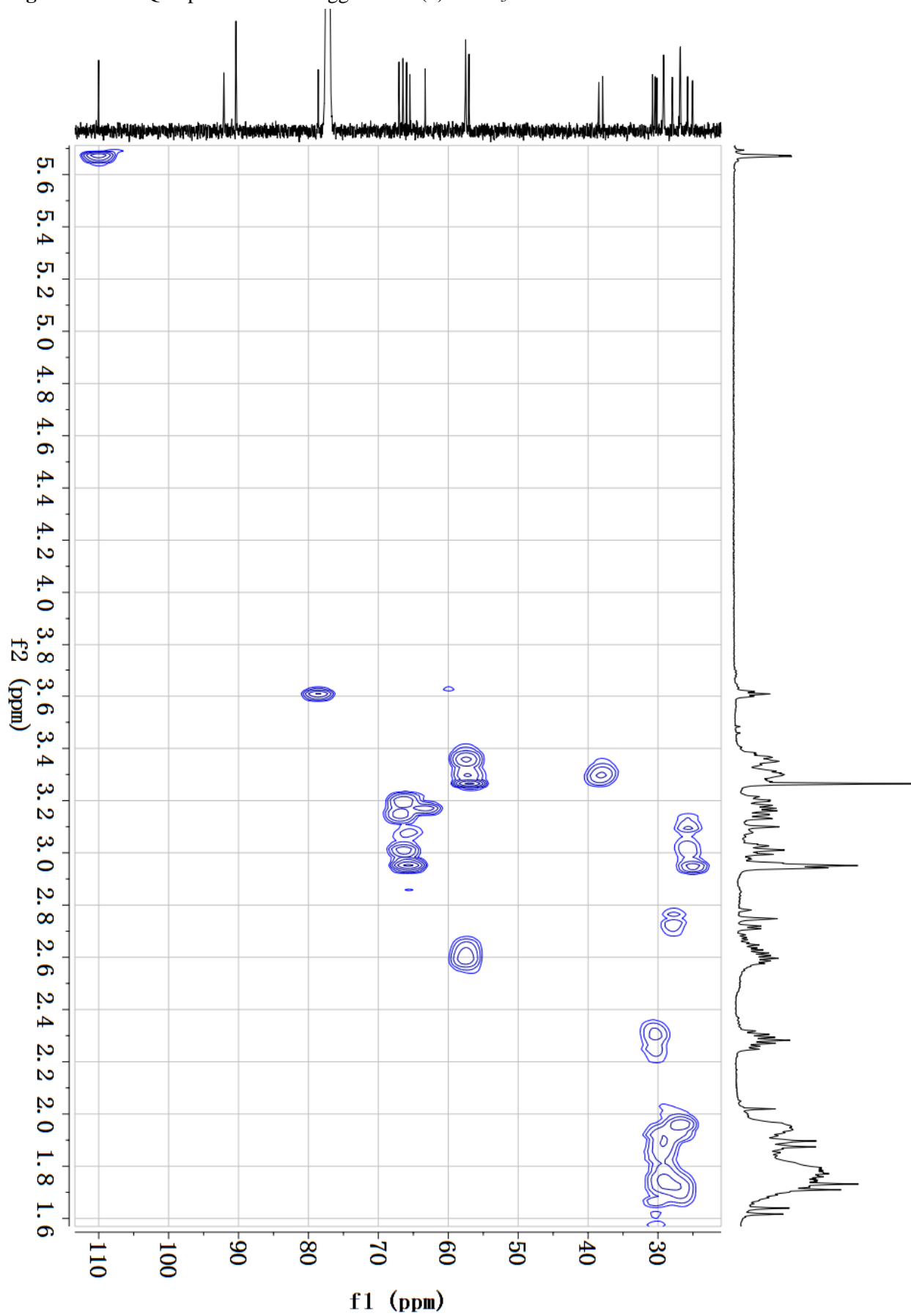


Figure S50. HMBC spectrum for flueggeine E (**6**) in CDCl₃.

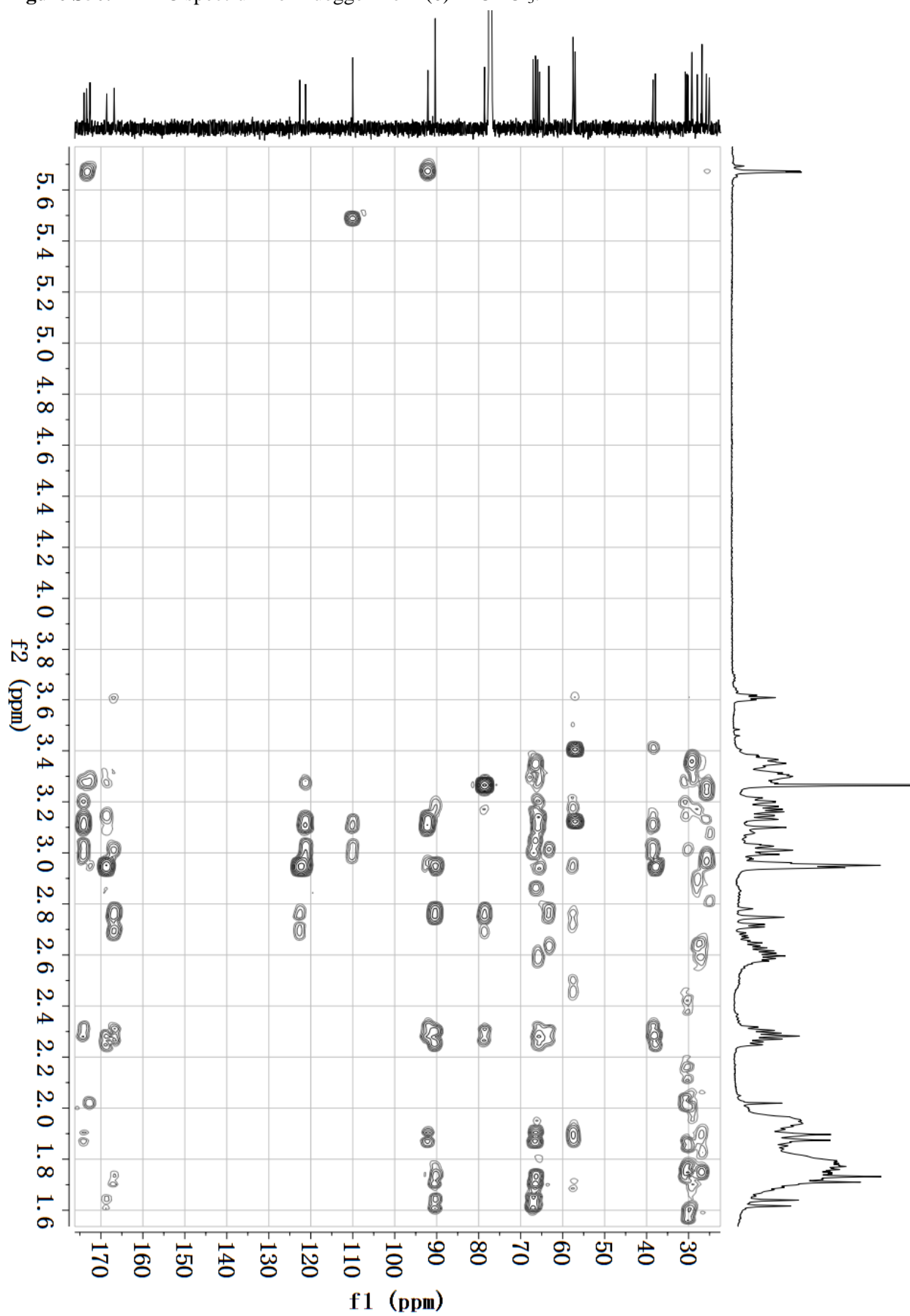


Figure S51. ROESY spectrum for flueggeine E (6) in CDCl₃.

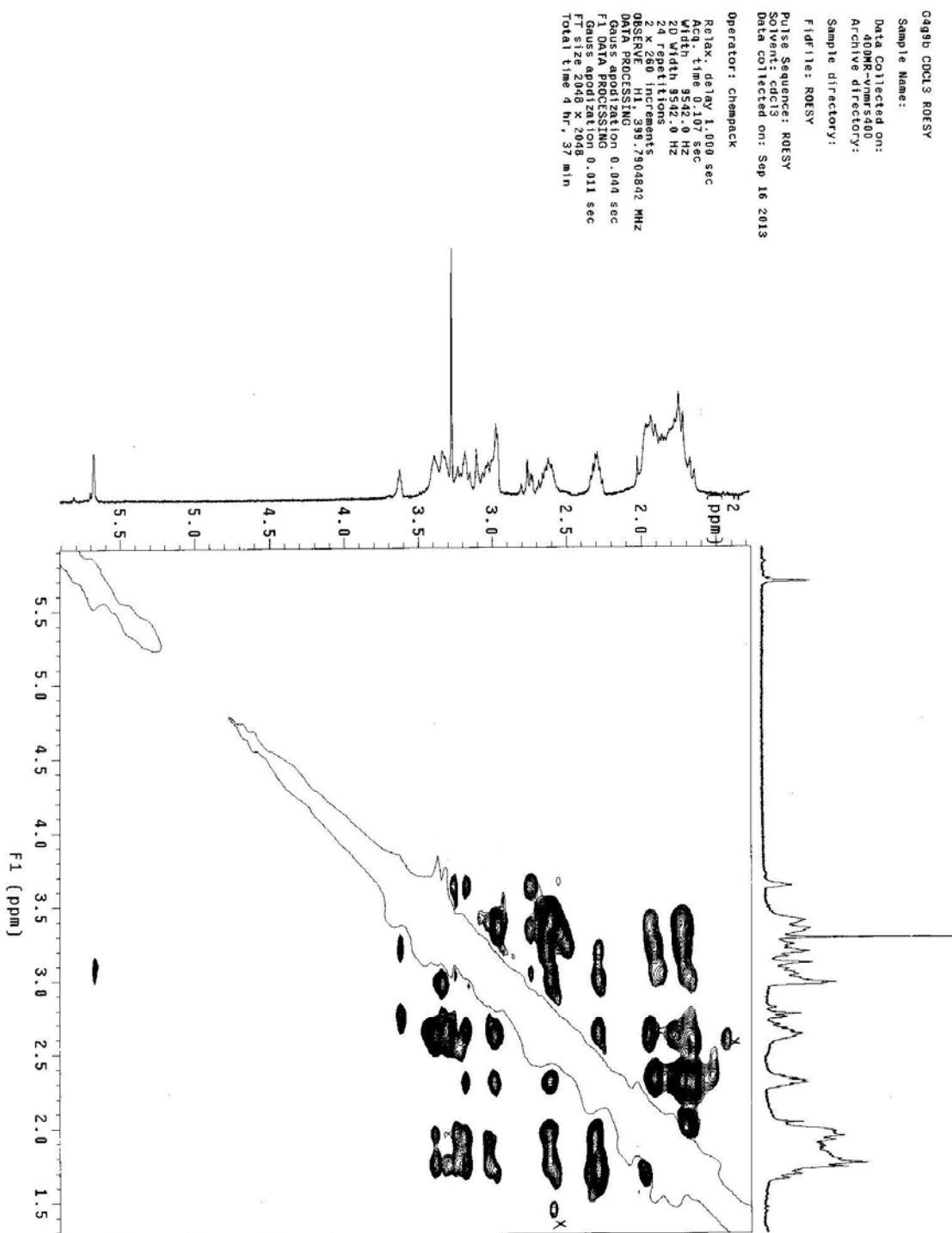


Figure S52. IR spectrum for flueggenine E (6).

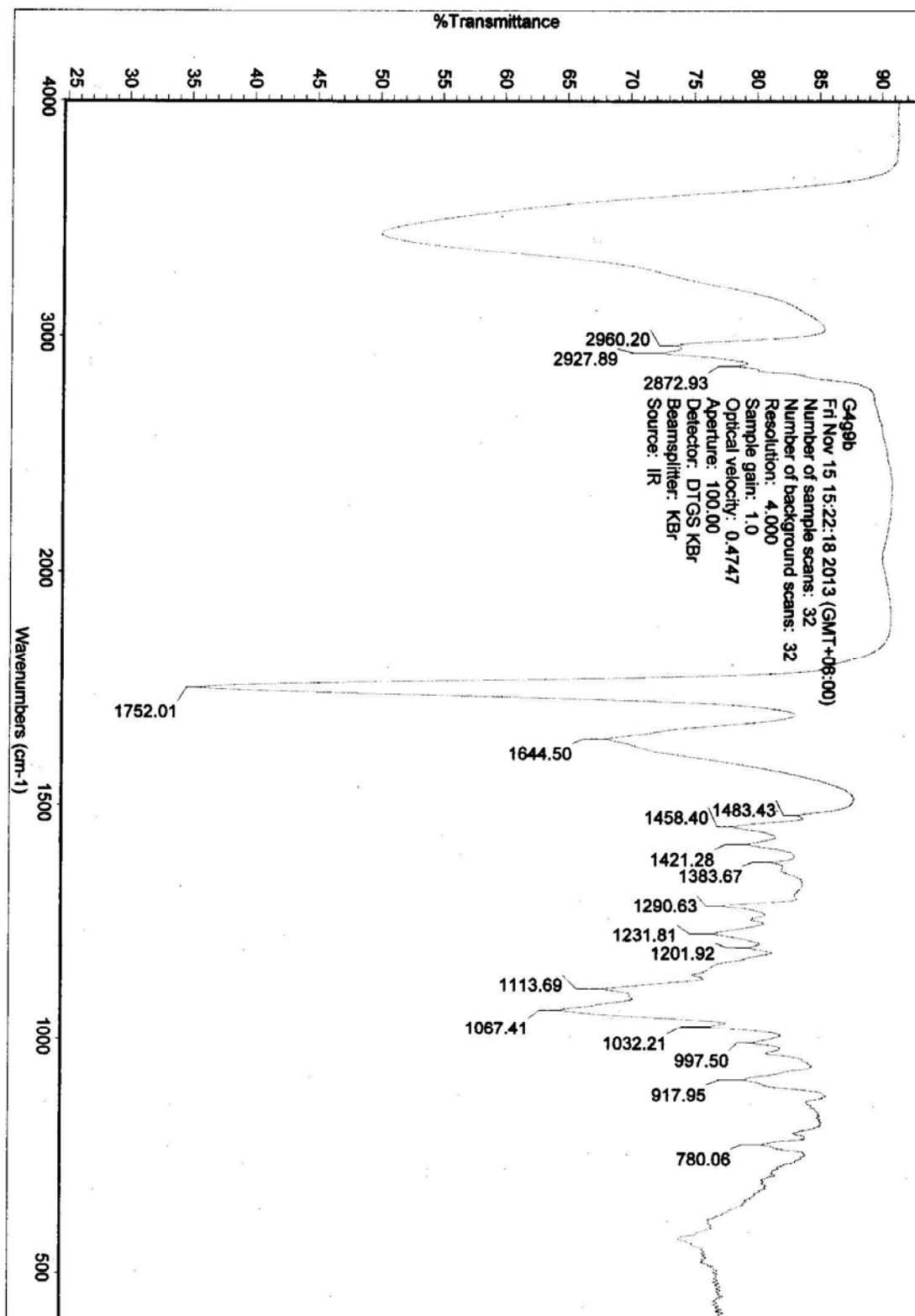


Figure S53. (+)-ESIMS spectrum for flueggeine E (6).

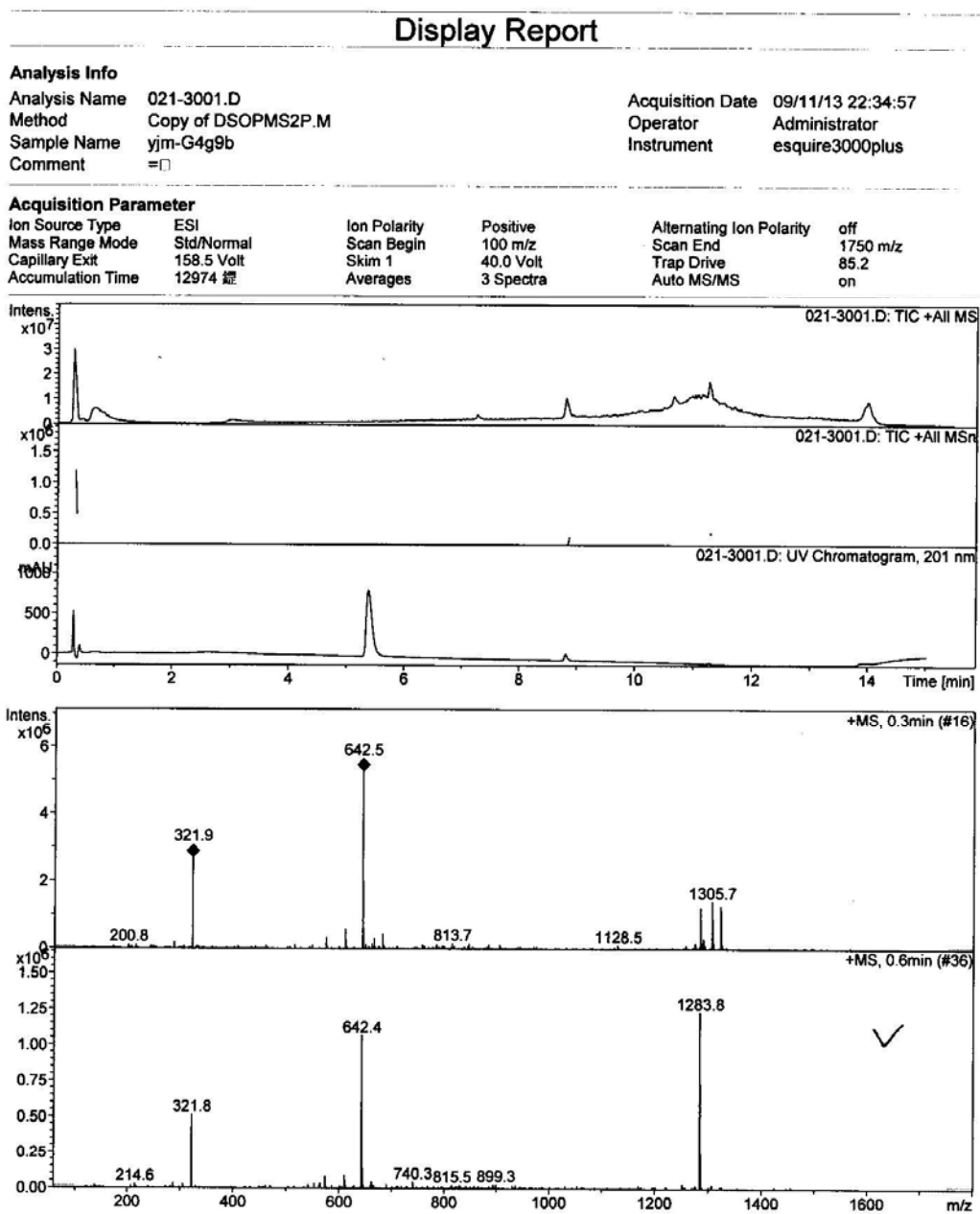


Figure S54. (+)-HRESIMS spectrum for flueggeine E (6).

Elemental Composition Report

Single Mass Analysis

Tolerance = 3.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

793 formula(e) evaluated with 3 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 5-80 H: 2-120 N: 0-5 O: 0-20

G4g9b

LCT PXE KE324

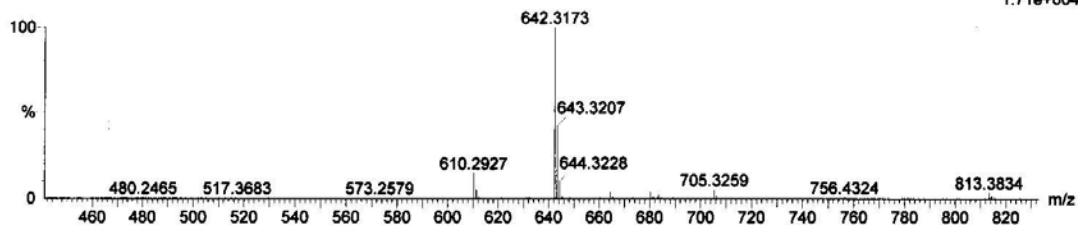
13-Sep-2013

15:18:05

G4g9b_0913 27 (0.565) AM2 (Ar,10000.0,0.00,1.00); ABS; Cm (26.42)

1: TOF MS ES+

1.71e+004



Minimum:

Maximum: 3.0 3.0 -1.5

50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
642.3173	642.3179	-0.6	-0.9	17.5	74.1	0.0	C37 H44 N3 O7
	642.3184	-1.1	-1.7	-0.5	83.0	8.9	C24 H52 N O18
	642.3161	1.2	1.9	30.5	83.3	9.2	C49 H40 N

Figure S55. ^1H NMR spectrum for flueviroisine F (**7**) in CDCl_3 .

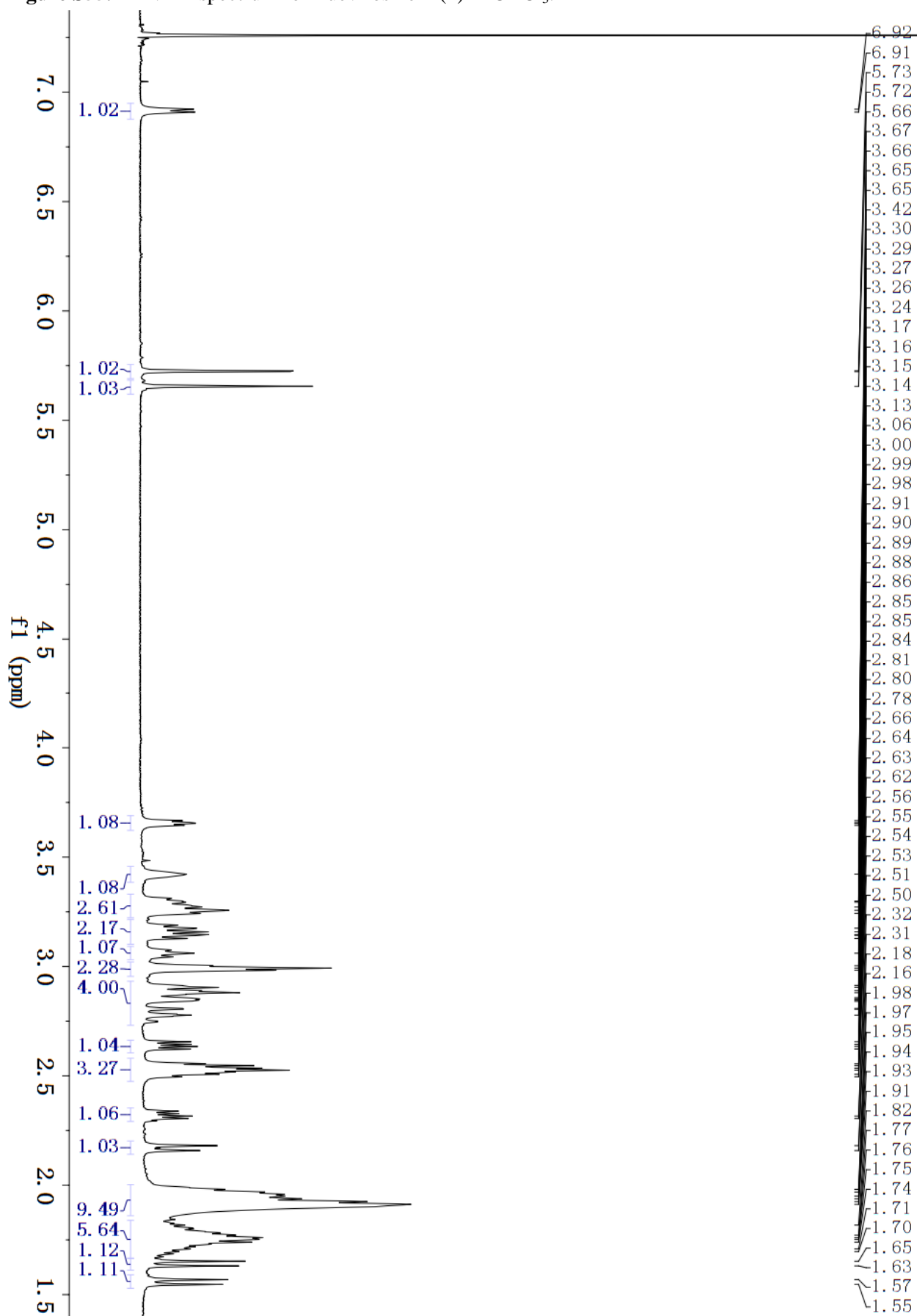


Figure S56. ^{13}C NMR spectrum for flueggein F (7) in CDCl_3 .

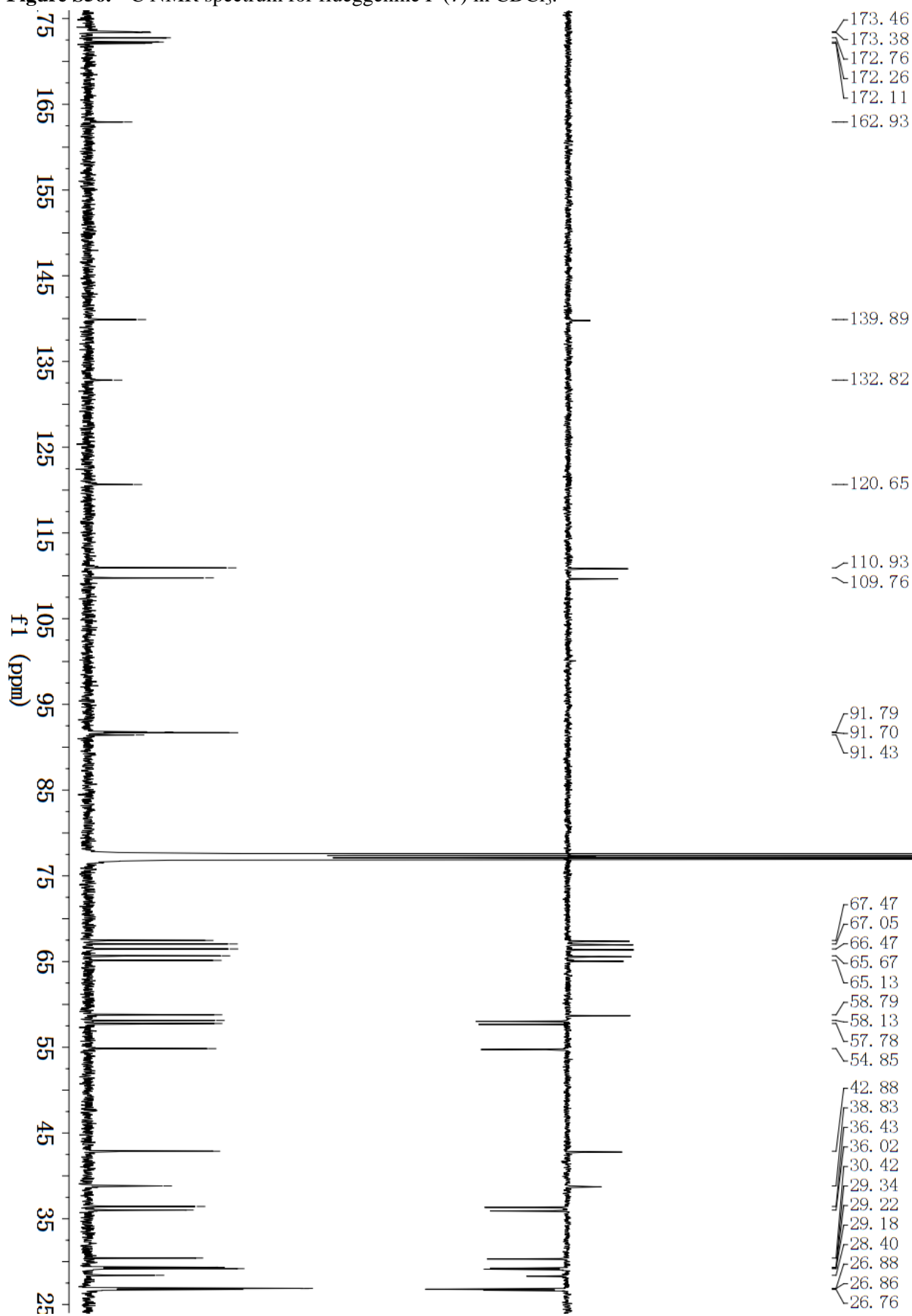


Figure S57. ^1H - ^1H COSY spectrum for flueggeine F (7) in CDCl_3 .

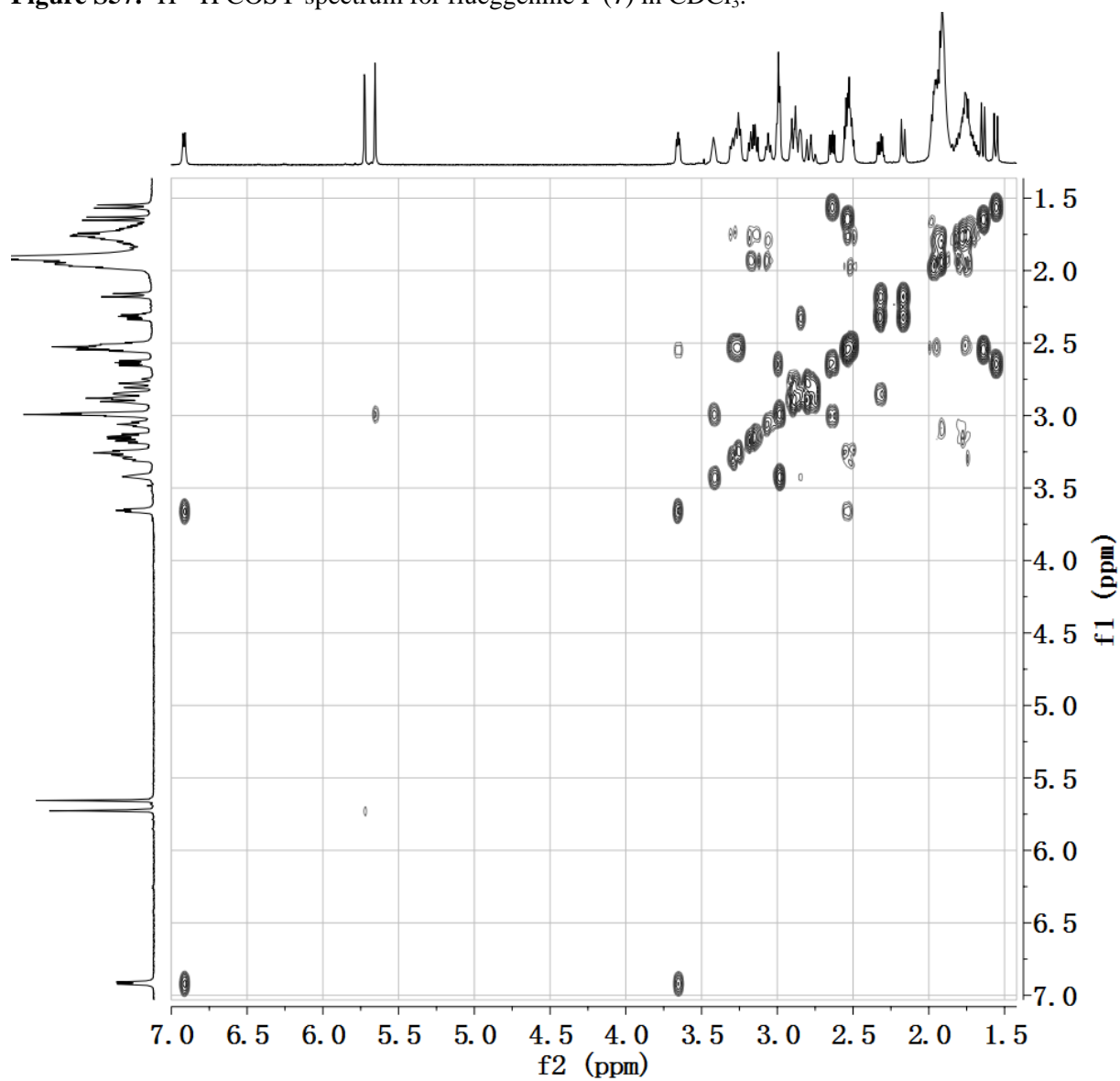


Figure S58. HSQC spectrum for flueggein F (7) CDCl_3 .

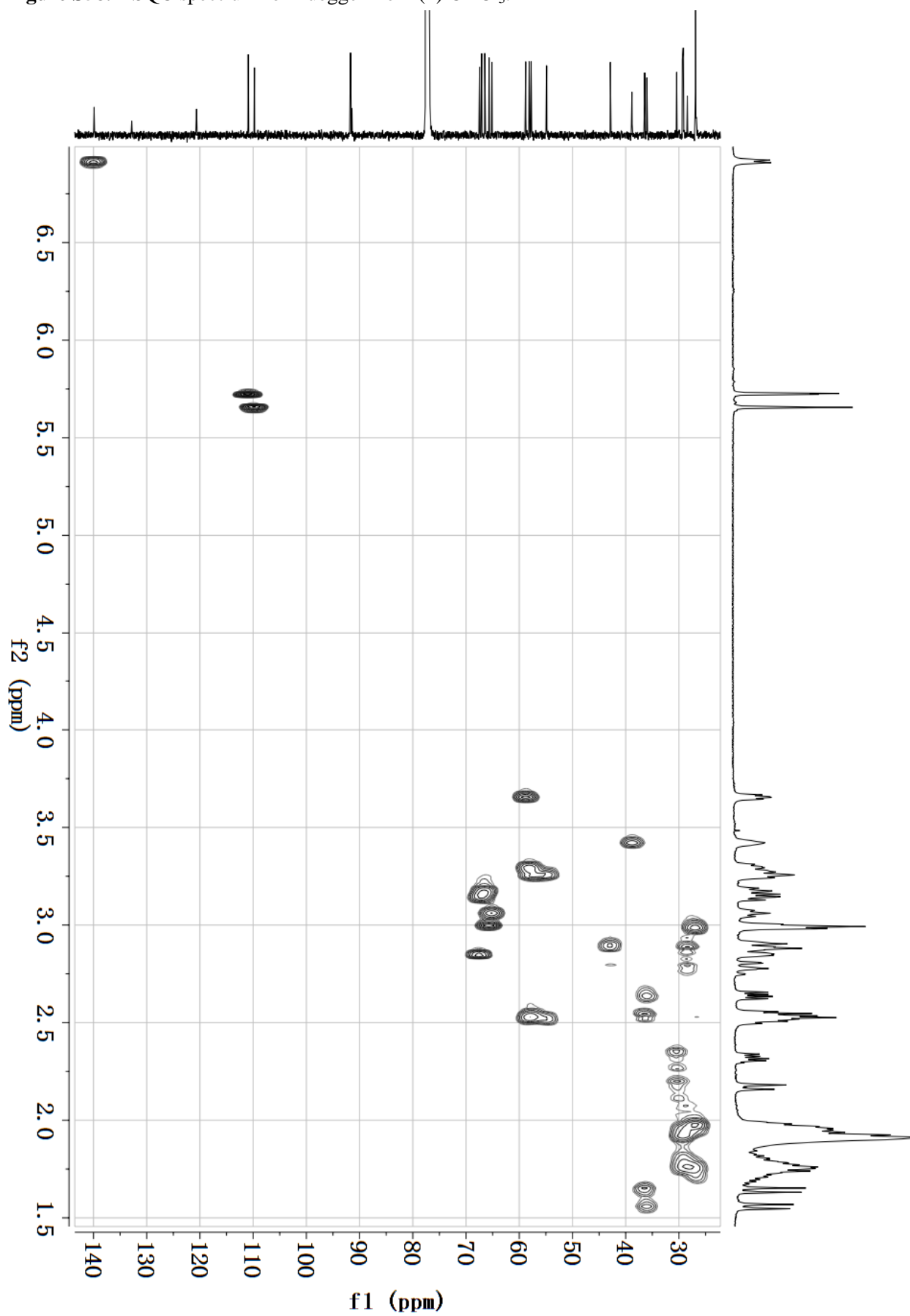


Figure S59. HMBC spectrum for flueggein F (7) in CDCl₃.

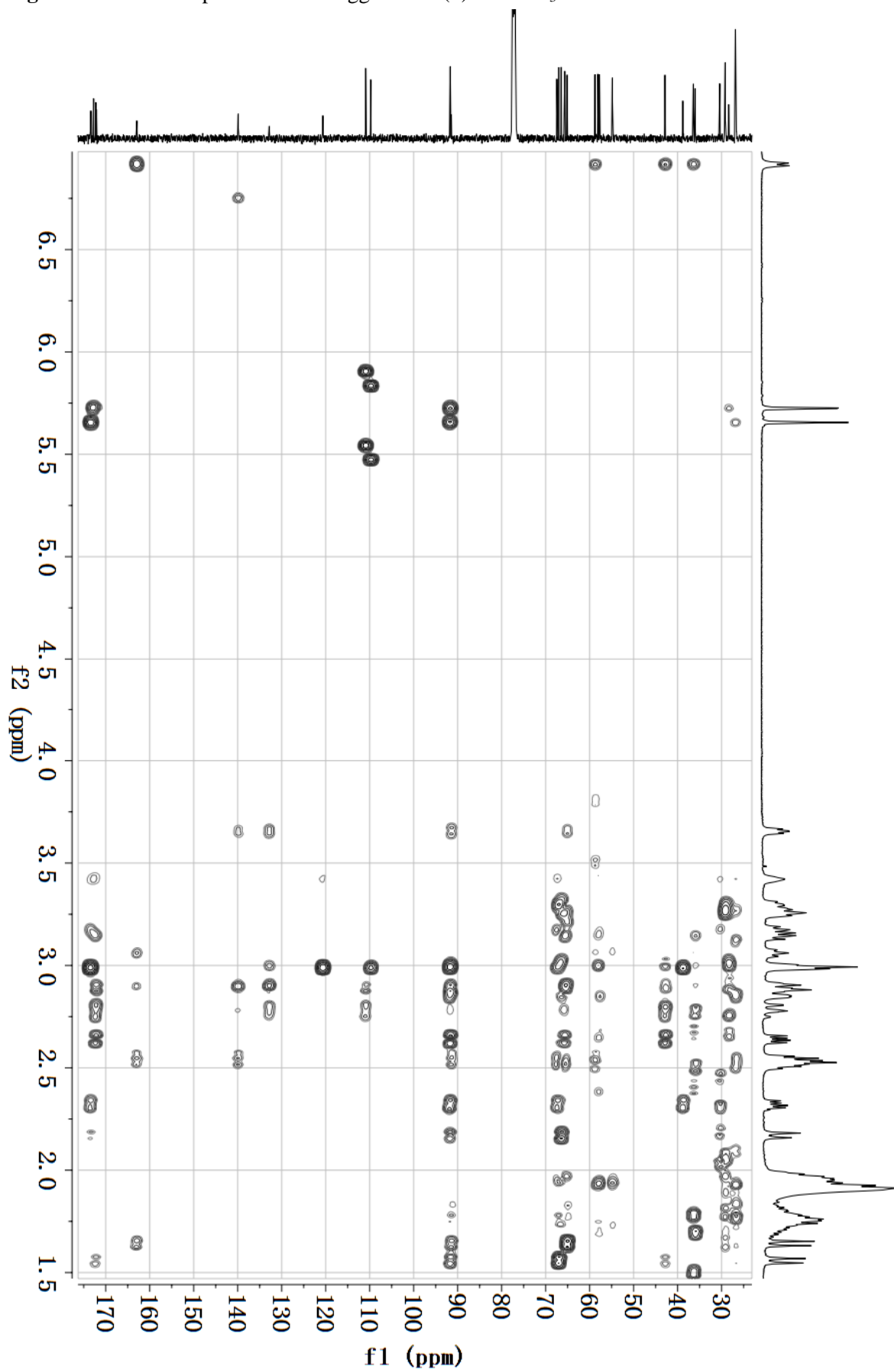


Figure S60. ROESY spectrum for flueggeine F (7) in CDCl₃.

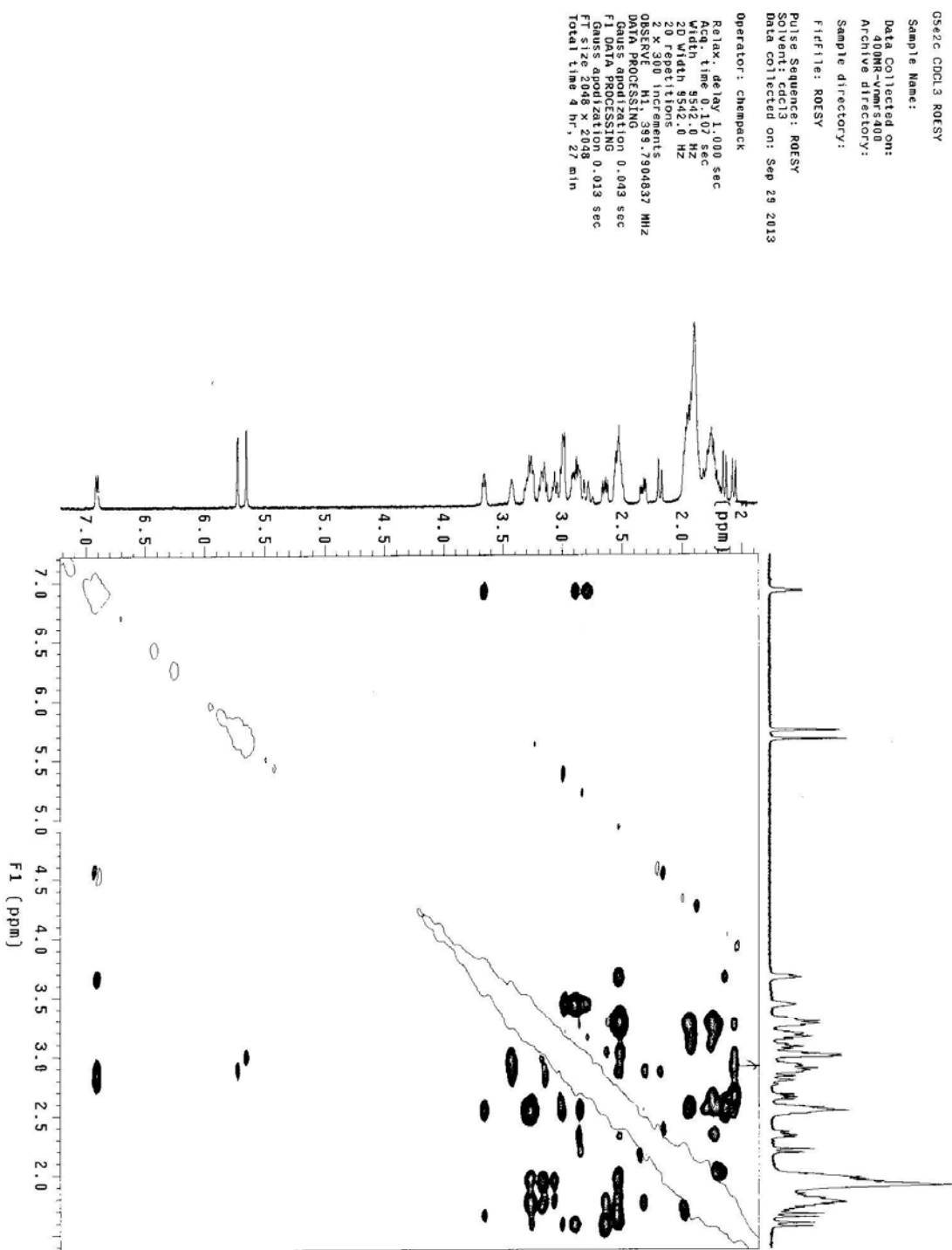


Figure S61. IR spectrum for flueggenine F (7).

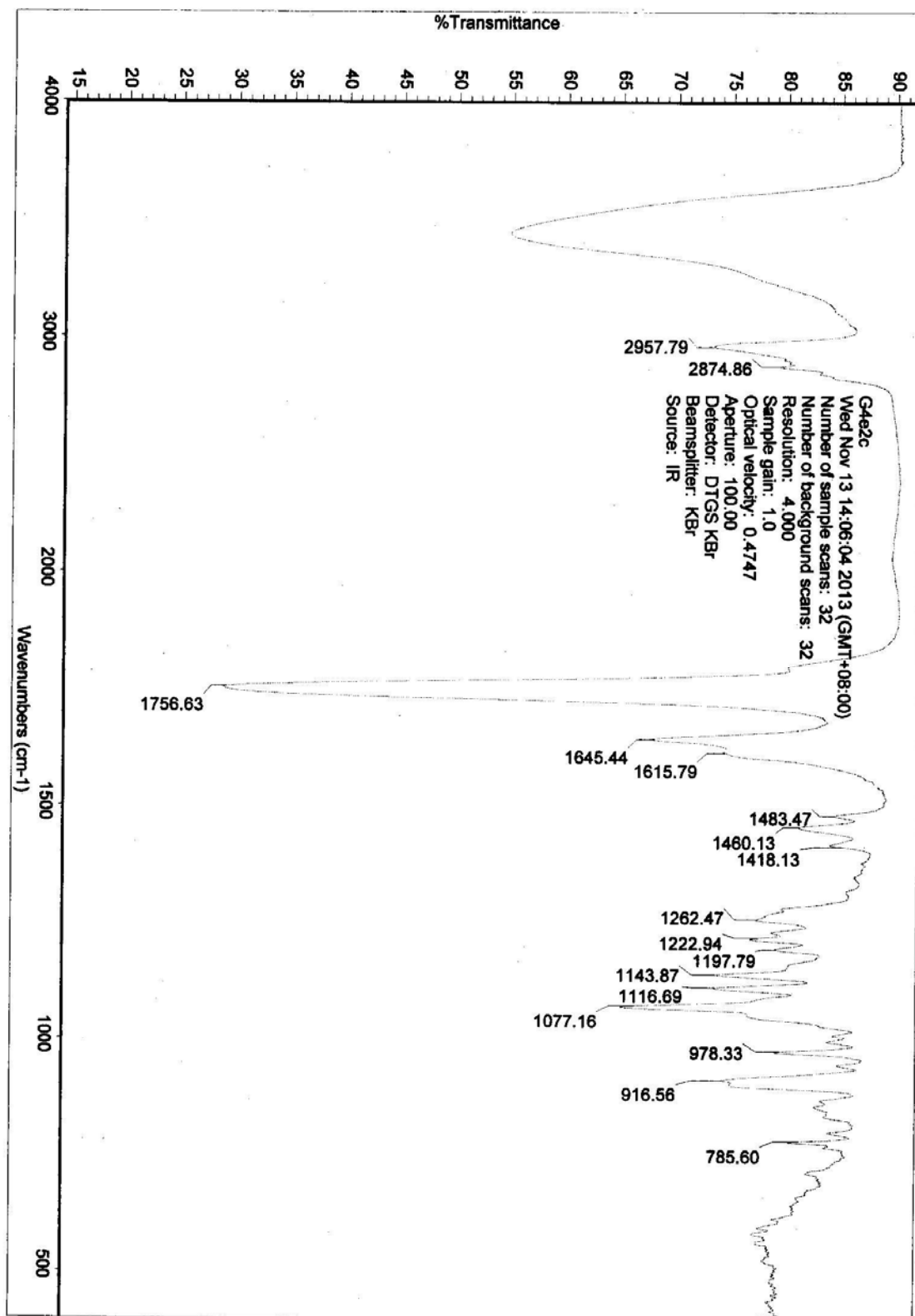


Figure S62. (+)-ESIMS spectrum for flueggeine F (7).

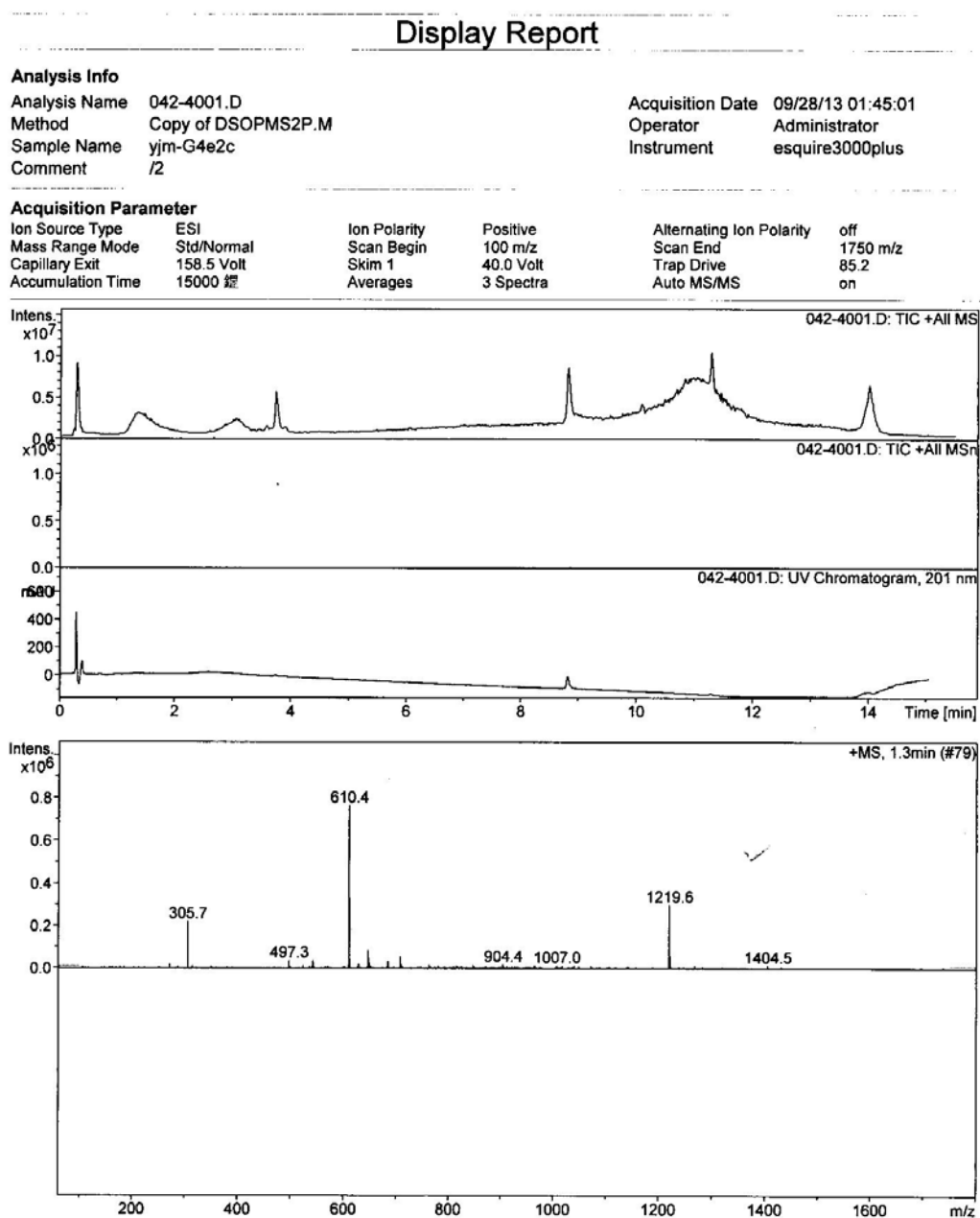


Figure S63. (+)-HRESIMS spectrum for flueggeine F (7).

Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

628 formula(e) evaluated with 3 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 5-80 H: 2-120 N: 0-4 O: 0-20

G4e2c

LCT PXE KE324

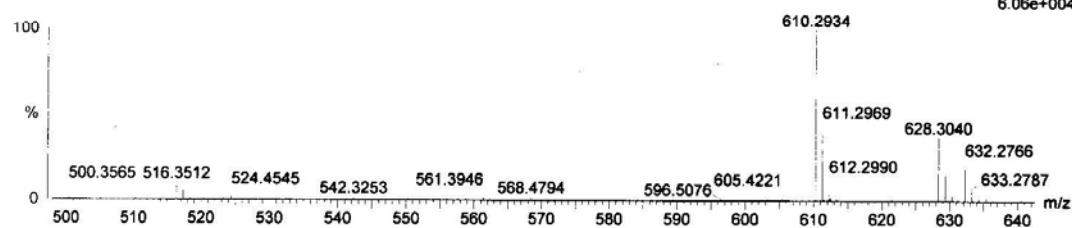
27-Sep-2013

10:01:36

1: TOF MS ES+

6.06e+004

G4e2c_0927 19 (0.422) AM2 (Ar,11000.0,0.00,0.70); ABS; Cm (15.62)



Minimum: -1.5
Maximum: 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
610.2934	610.2922	1.2	2.0	0.5	143.1	7.7	C23 H48 N O17
	610.2917	1.7	2.8	18.5	135.3	0.0	C36 H40 N3 O6 ✓
	610.2957	-2.3	-3.8	22.5	142.2	6.9	C41 H40 N O4

Figure S64. ^1H NMR spectrum for flueviroisine G (**8**) in CDCl_3 .

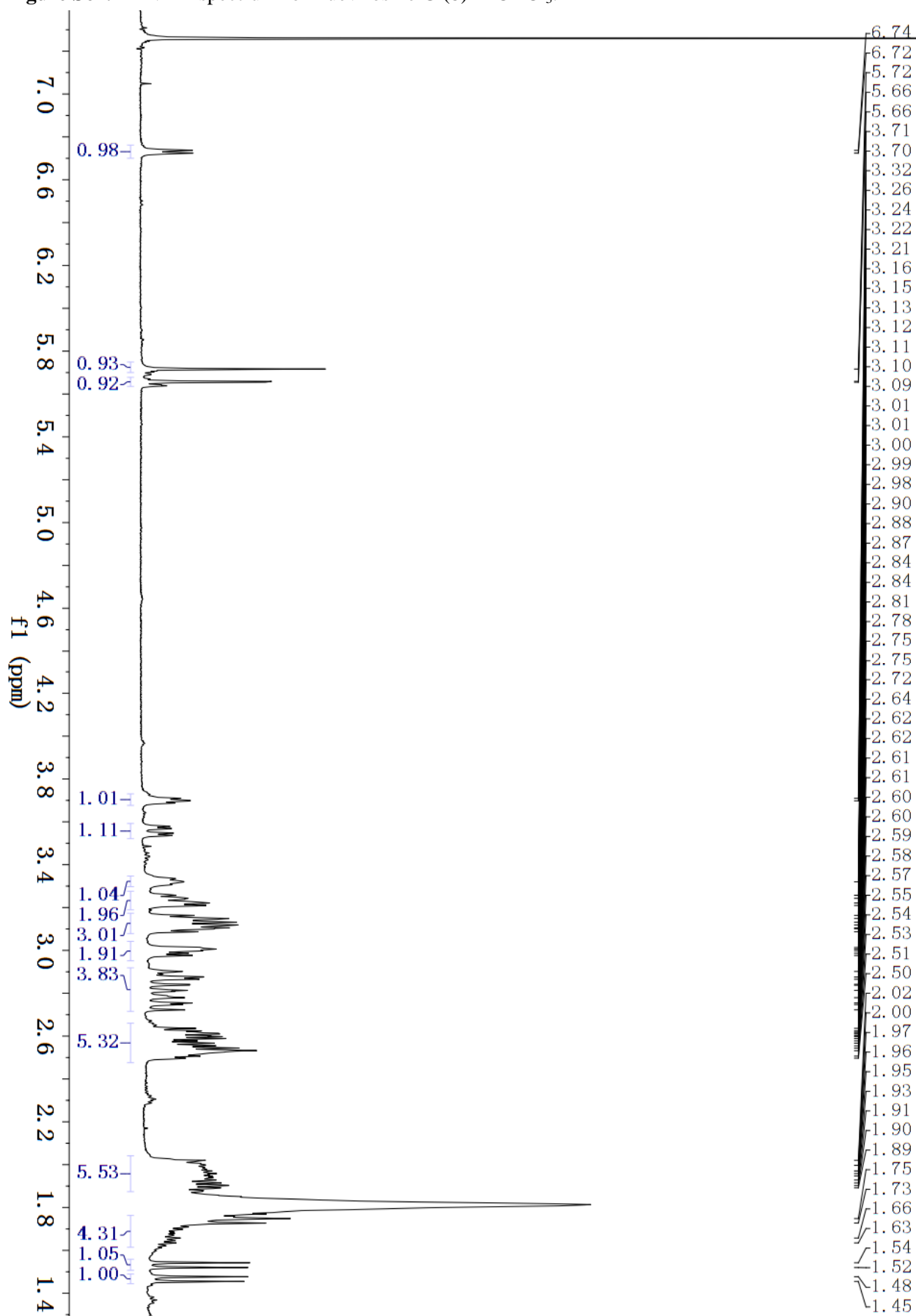


Figure S65. ^{13}C NMR spectrum for flueggenine G (**8**) in CDCl_3 .

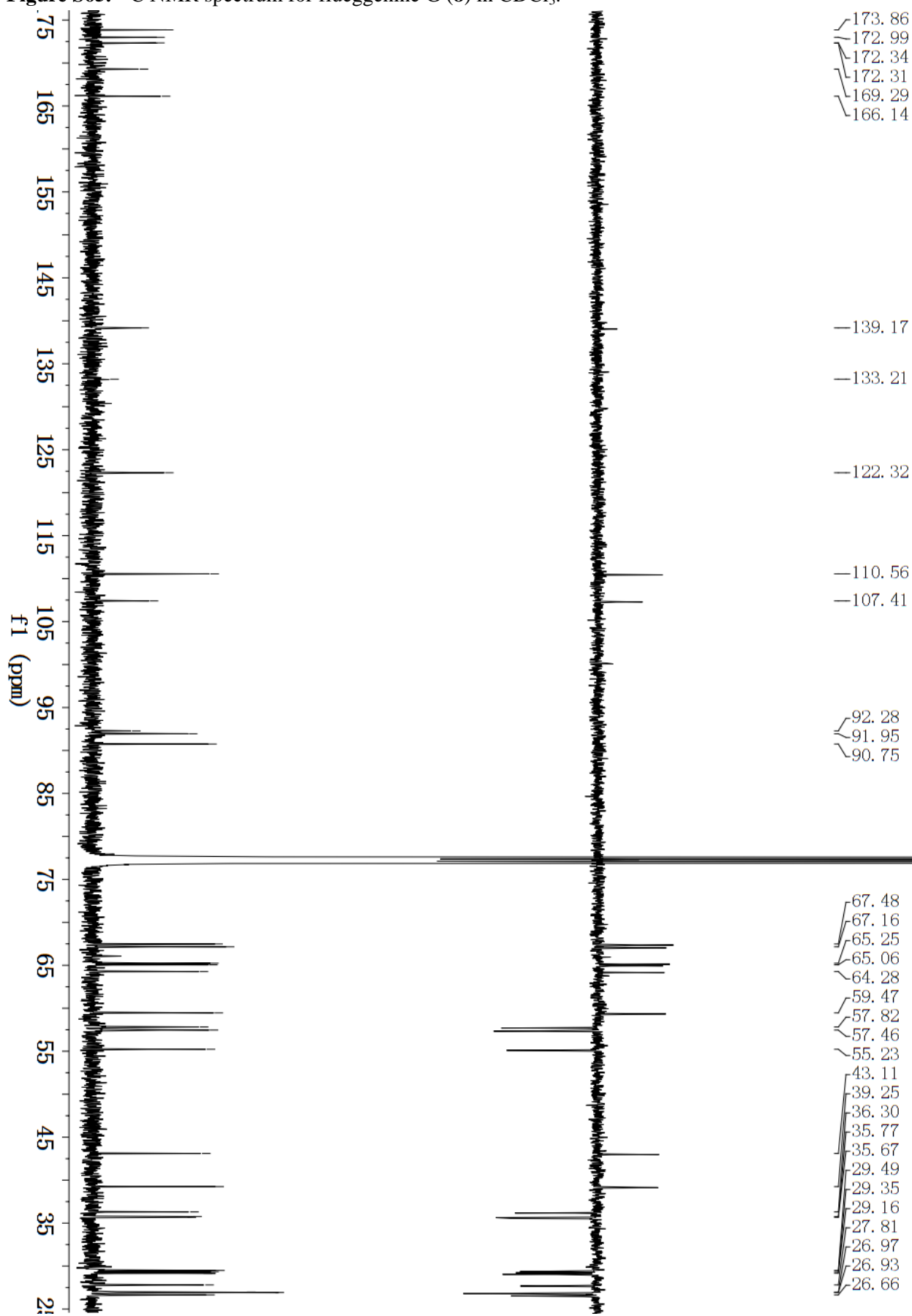


Figure S66. ^1H - ^1H COSY spectrum for flueggeine G (**8**) in CDCl_3 .

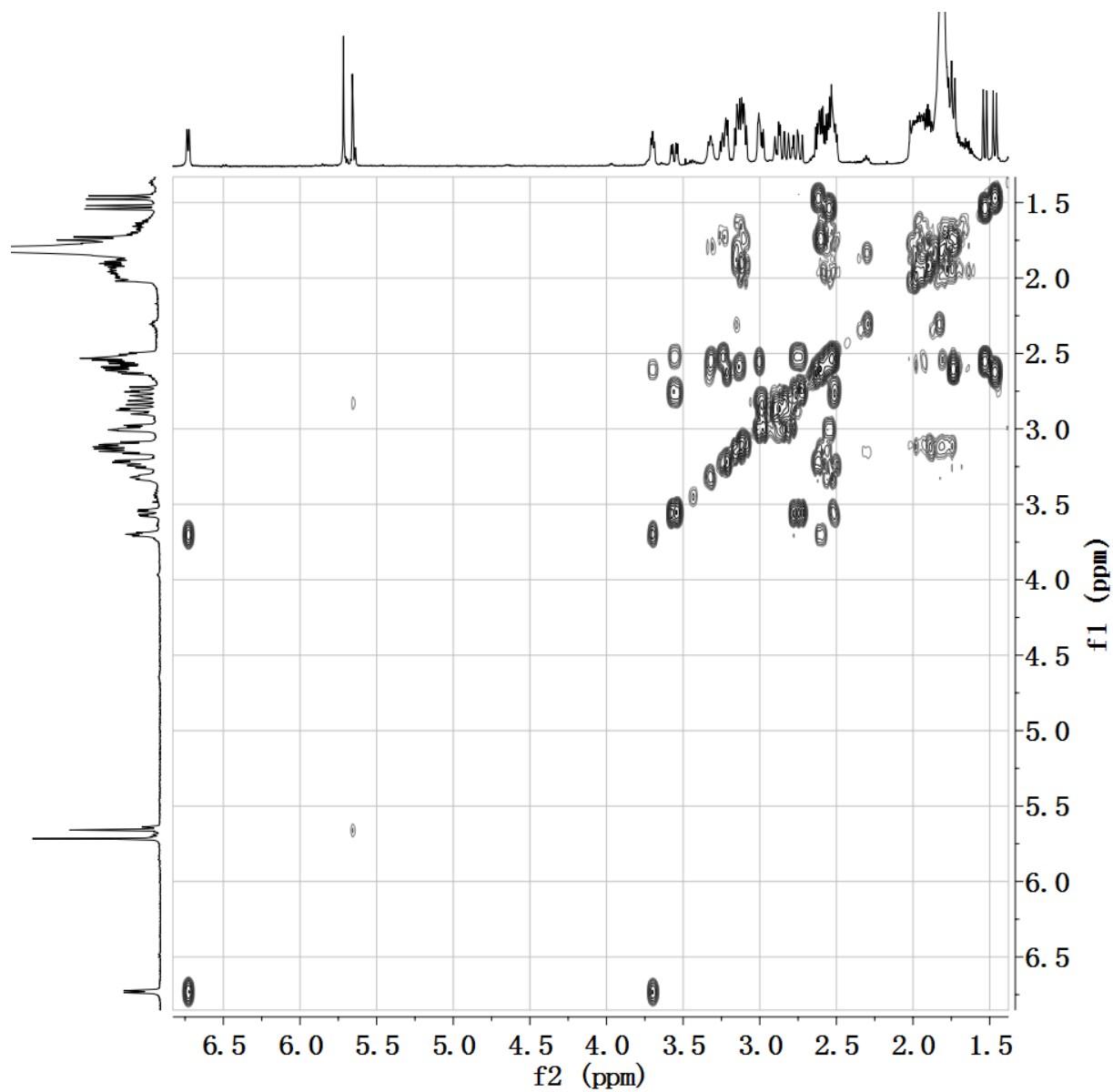


Figure S67. HSQC spectrum for flueggeine G (8) CDCl₃.

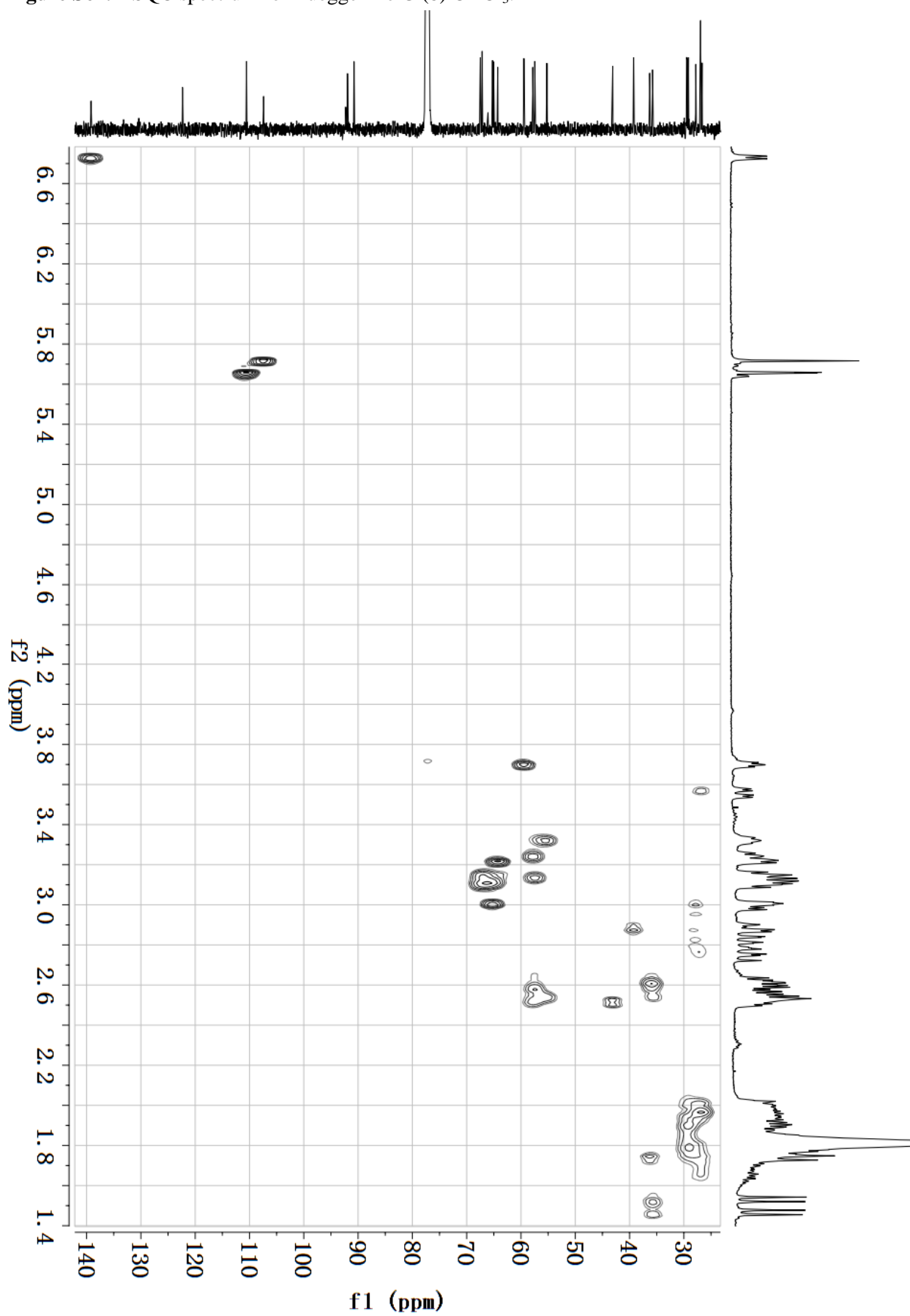


Figure S68. HMBC spectrum for flueggeine G (**8**) in CDCl₃.

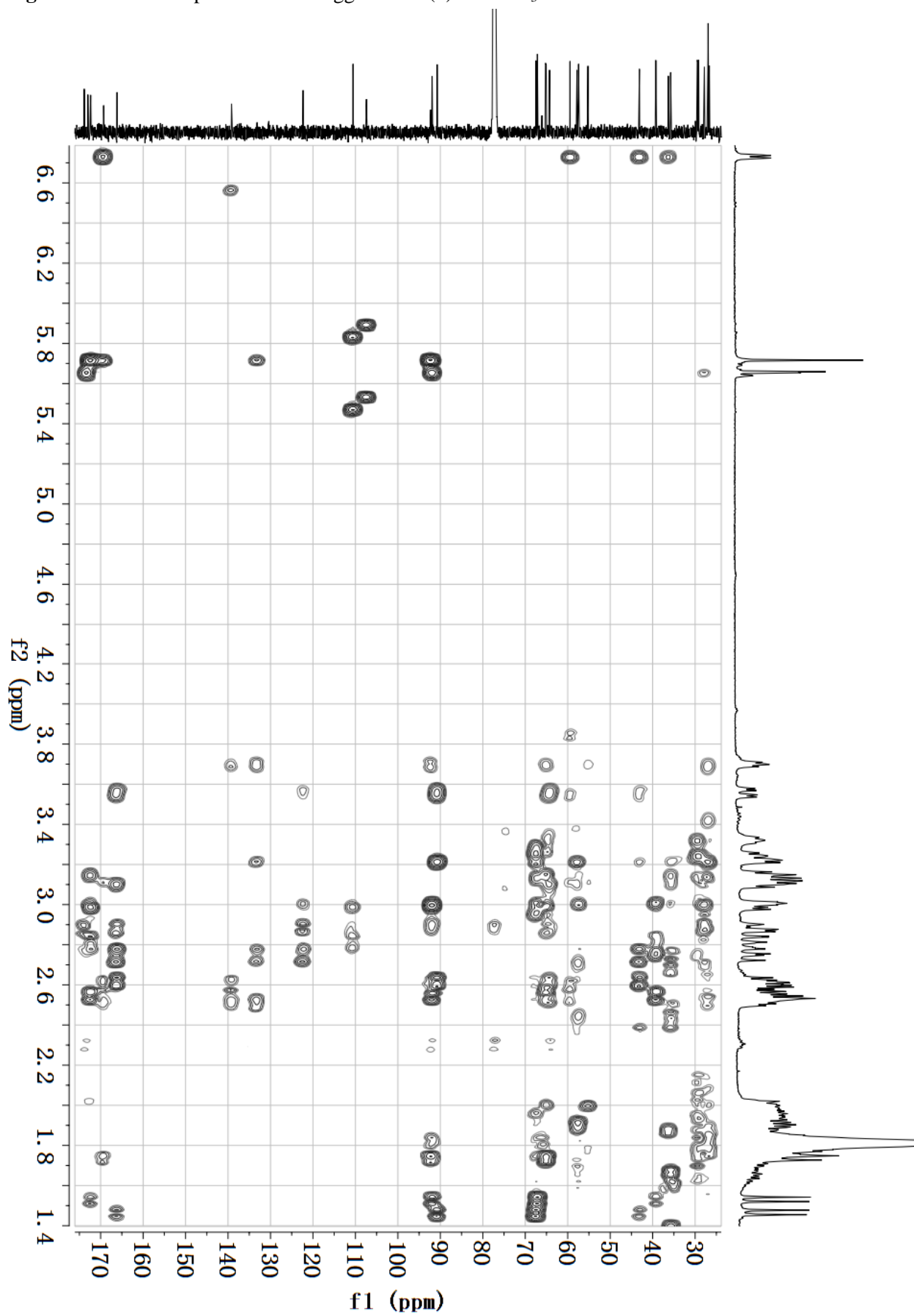


Figure S69. ROESY spectrum for flueggeanine G (8) in CDCl₃.

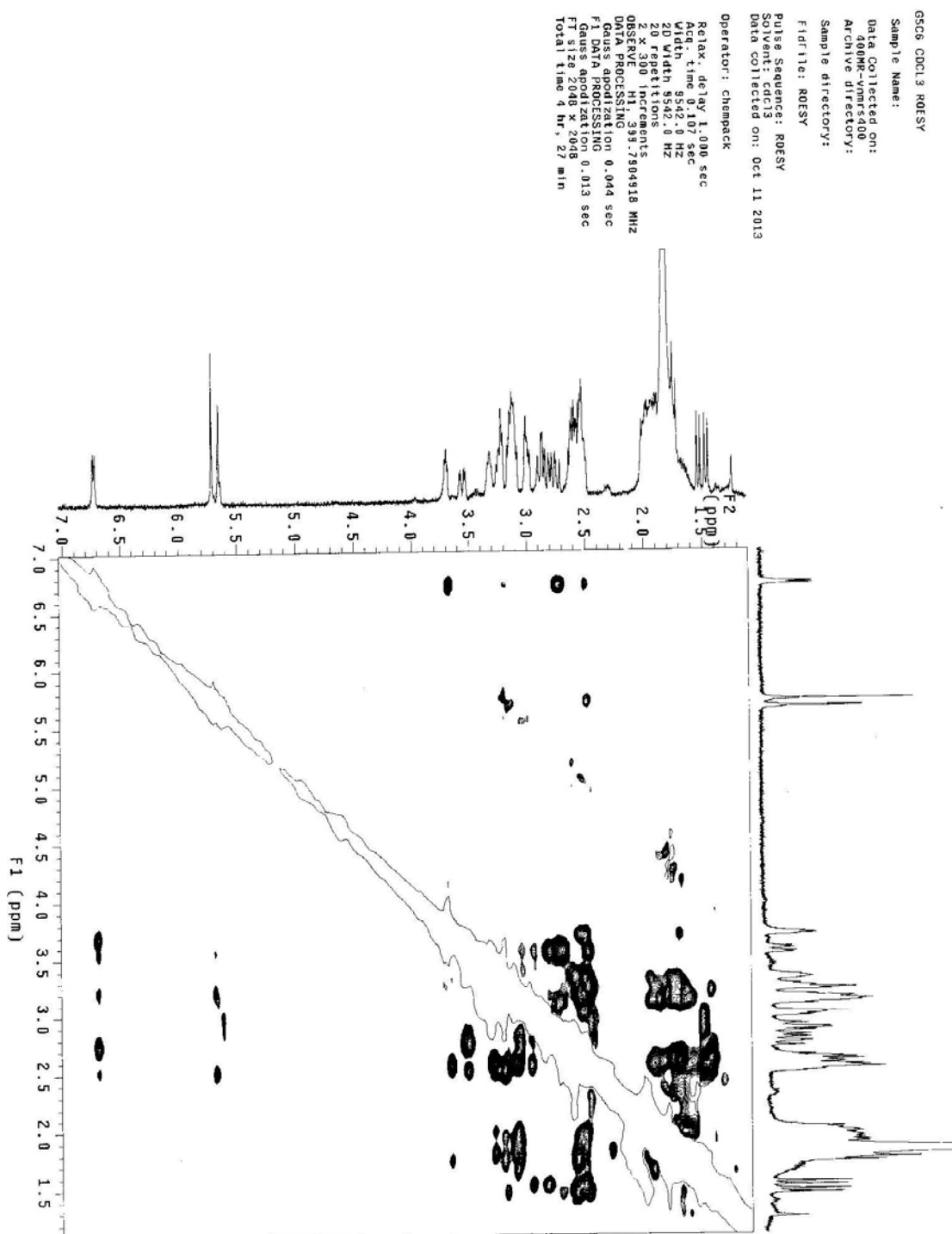


Figure S70. IR spectrum for flueggenine G (8).

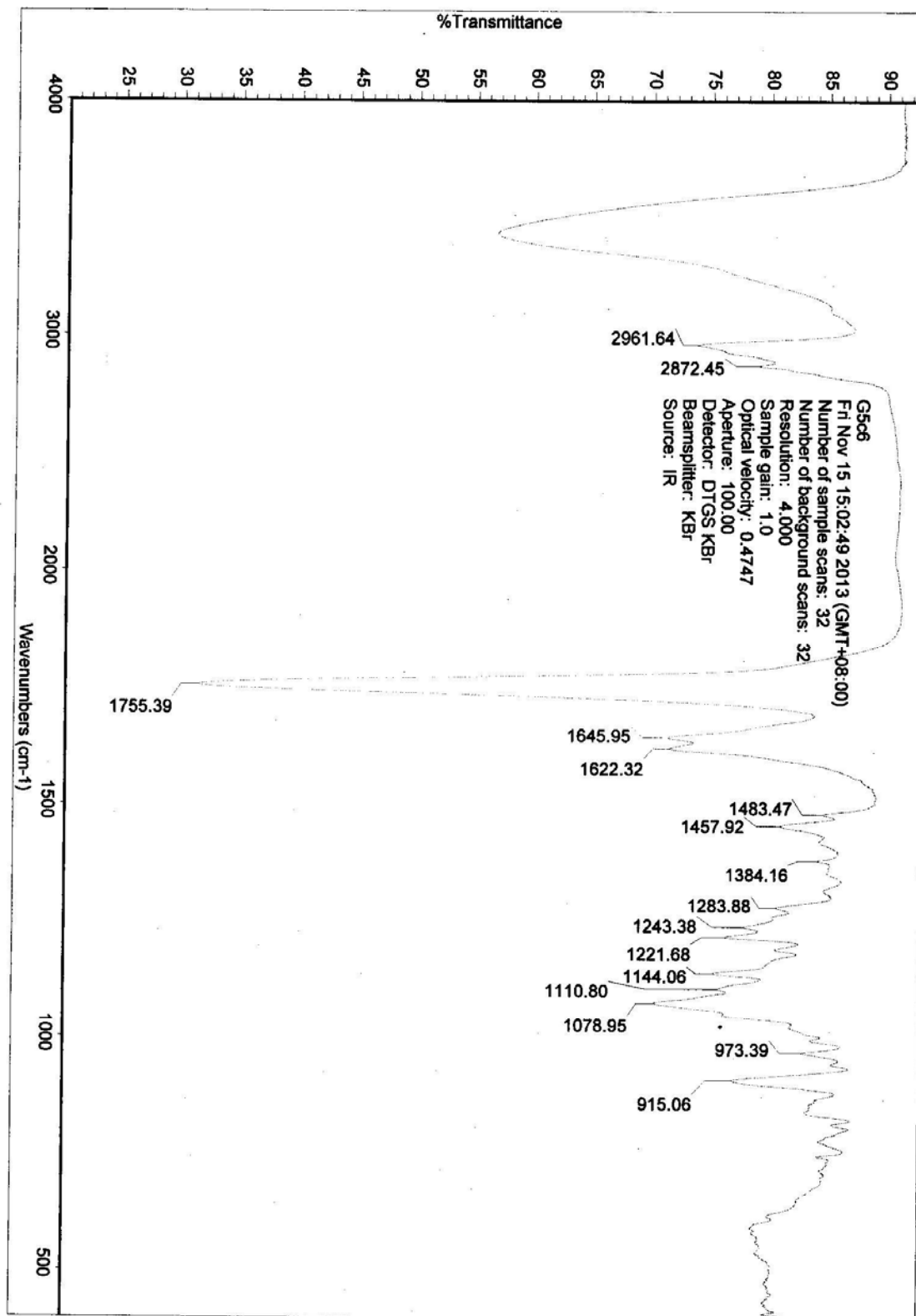


Figure S71. (+)-ESIMS spectrum for flueggeanine G (8).

Display Report

Analysis Info

Analysis Name	041-8601.D	Acquisition Date	10/16/13 08:22:31
Method	Copy of DSOPMS2P.M	Operator	Administrator
Sample Name	yjm-G5C6	Instrument	esquire3000plus
Comment	喘		

Acquisition Parameter

Ion Source Type	ESI	Ion Polarity	Positive	Alternating Ion Polarity	off
Mass Range Mode	Std/Normal	Scan Begin	100 m/z	Scan End	1750 m/z
Capillary Exit	158.5 Volt	Skim 1	40.0 Volt	Trap Drive	85.2
Accumulation Time	15000 罫	Averages	3 Spectra	Auto MS/MS	on

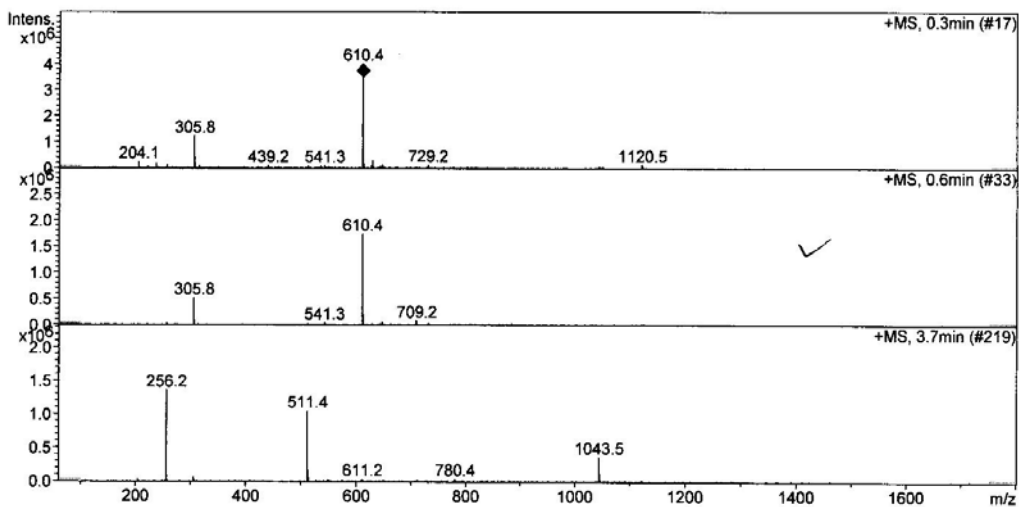
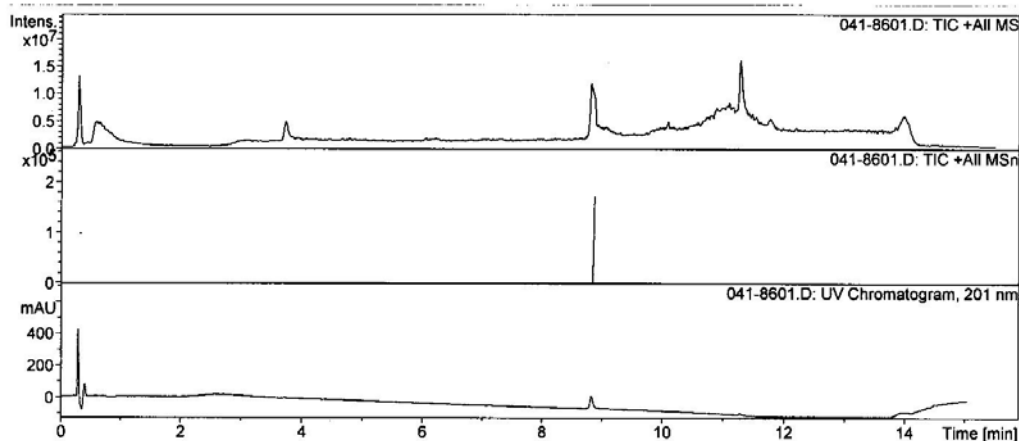


Figure S72. (+)-HRESIMS spectrum for flueggenine G (8).

Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

628 formula(e) evaluated with 2 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 5-80 H: 2-120 N: 0-4 O: 0-20

G5c6

LCT PXE KE324

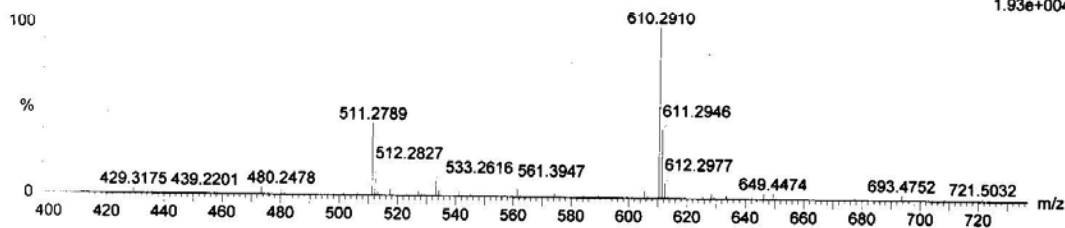
10-Oct-2013

16:25:50

1: TOF MS ES+

1.93e+004

G5c6_1010 24 (0.510) AM2 (Ar,10000.0,0.00,1.00); ABS; Cm (22:45)



Minimum: -1.5
Maximum: 3.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
610.2910	610.2917	-0.7	-1.1	18.5	89.9	0.0	C36 H40 N3 O6
	610.2922	-1.2	-2.0	0.5	96.7	6.8	C23 H48 N O17

Figure S73. ^1H NMR spectrum for flueviroisine H (**9**) in CDCl_3 .

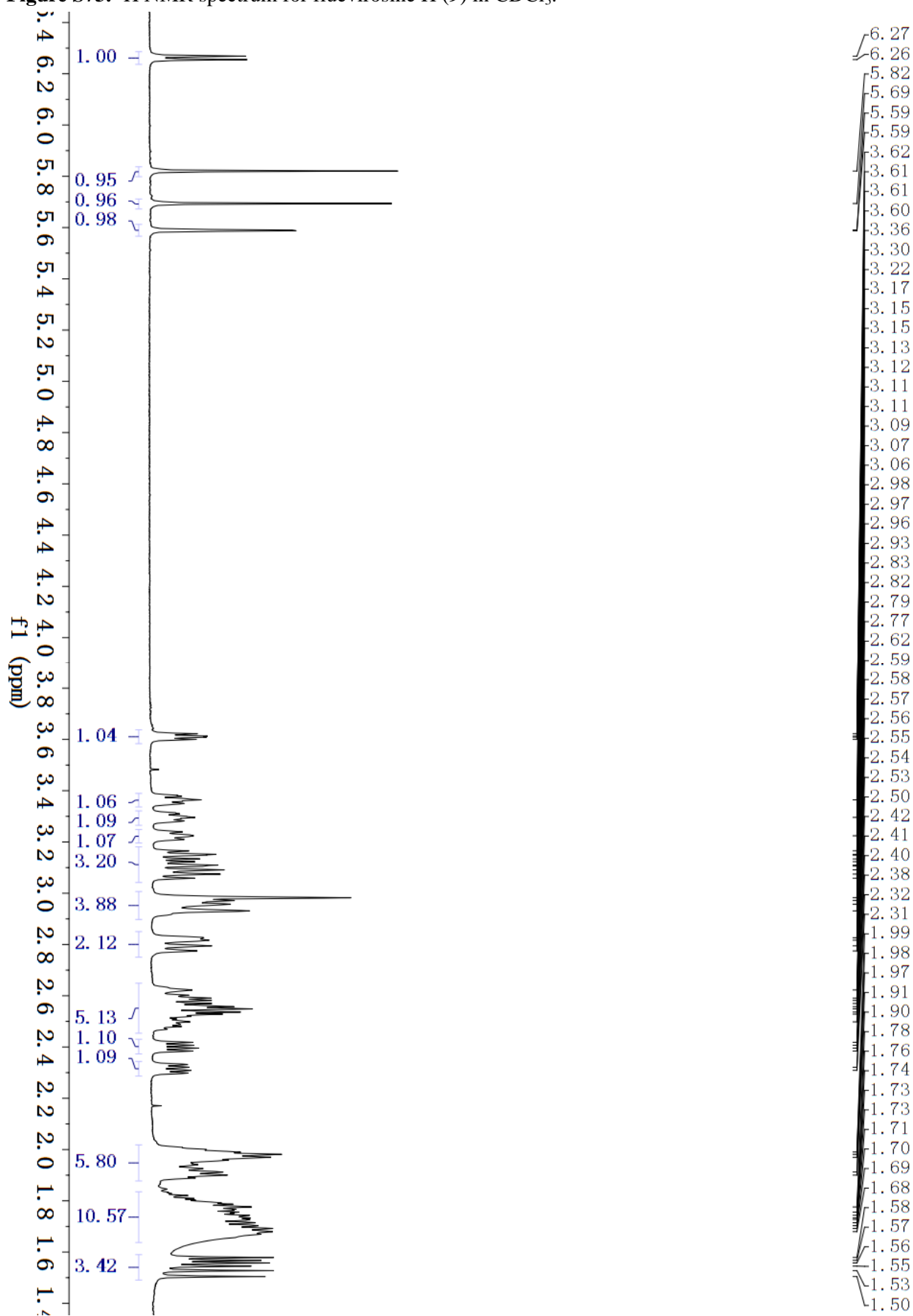


Figure S74. ^{13}C NMR spectrum for flueggeanine H (9) in CDCl_3 .

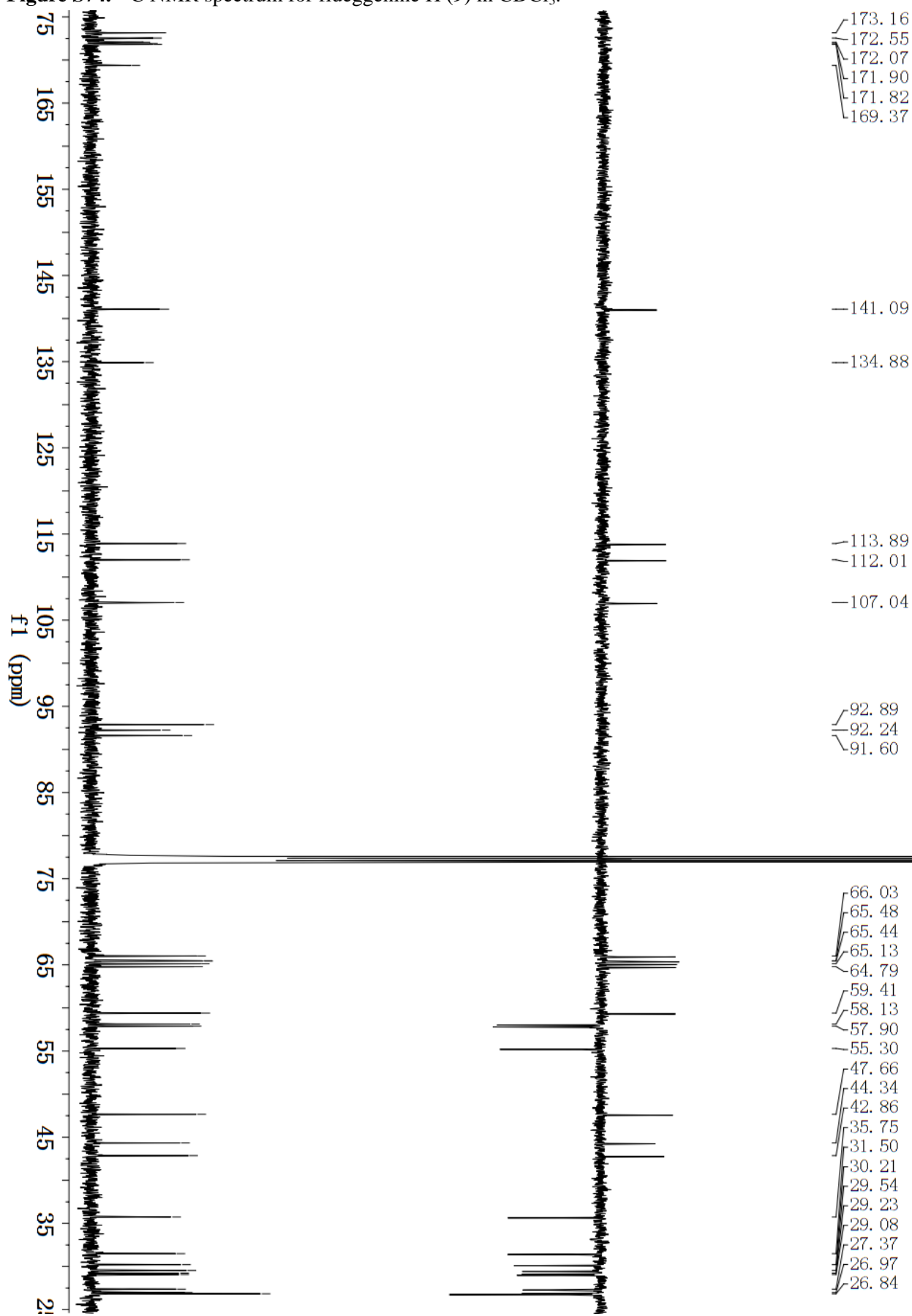


Figure S75. ^1H - ^1H COSY spectrum for flueggeanine H (9) in CDCl_3 .

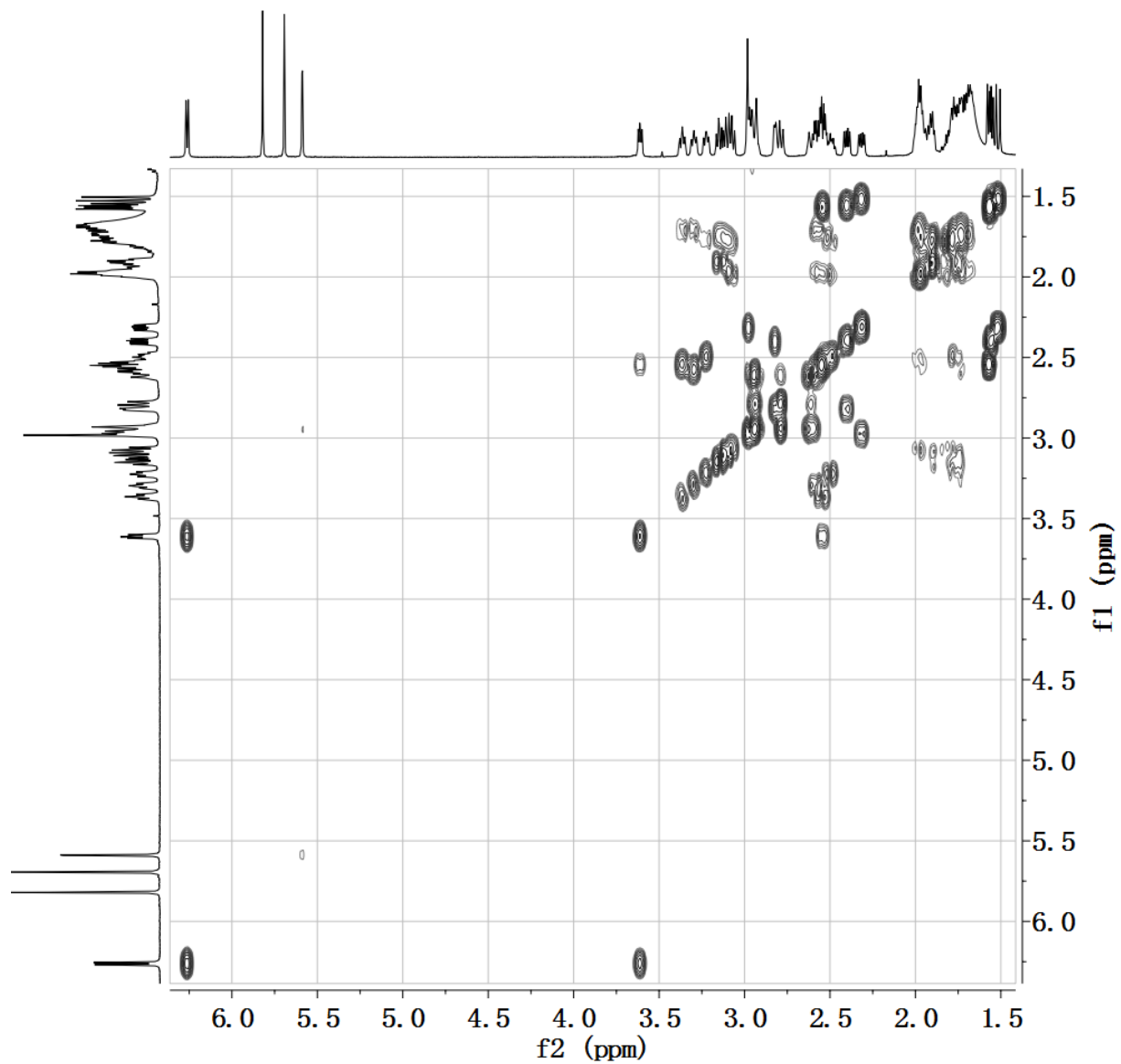


Figure S76. HSQC spectrum for flueggeine H (9) CDCl₃.

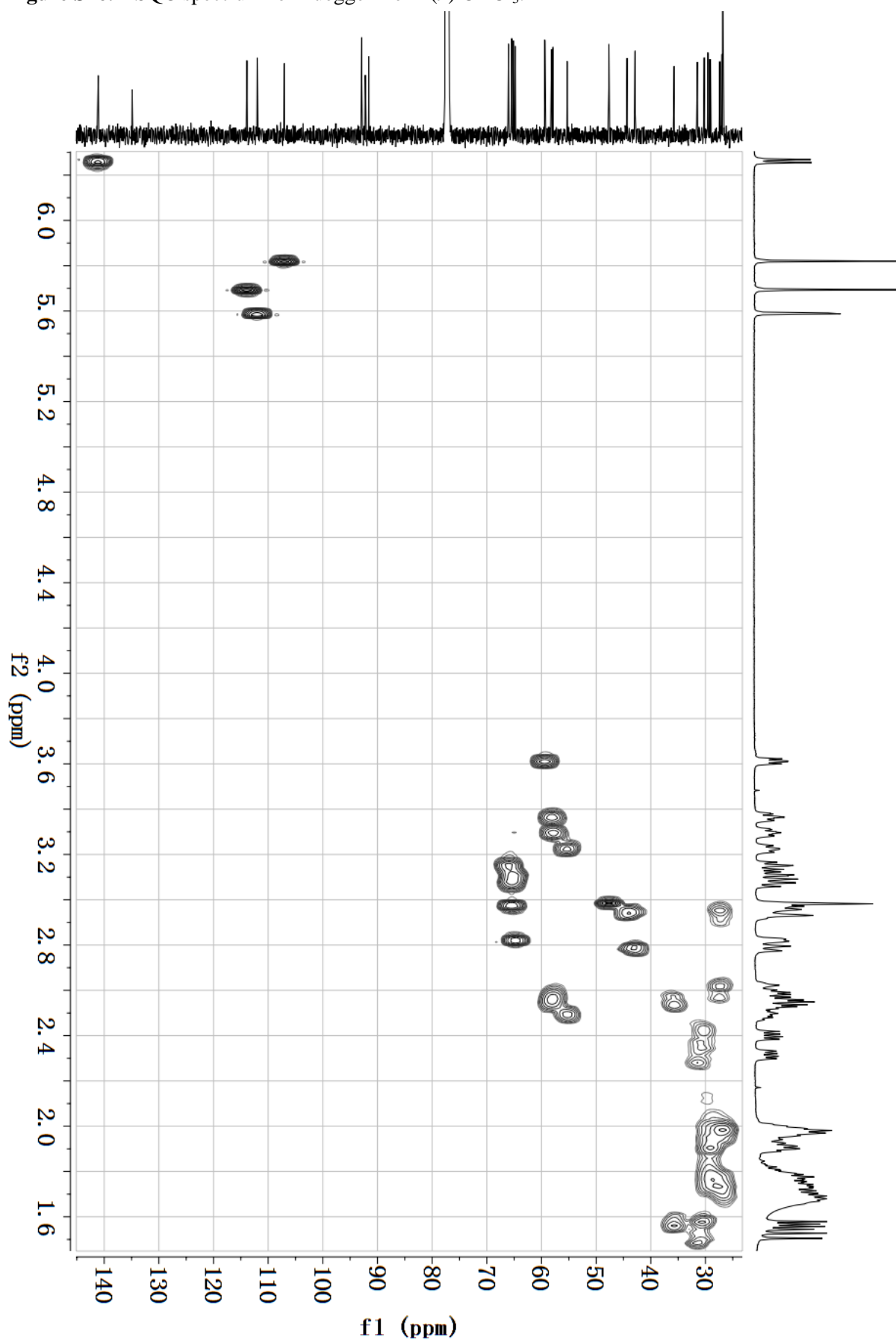


Figure S77. HMBC spectrum for flueggeine H (9) in CDCl₃.

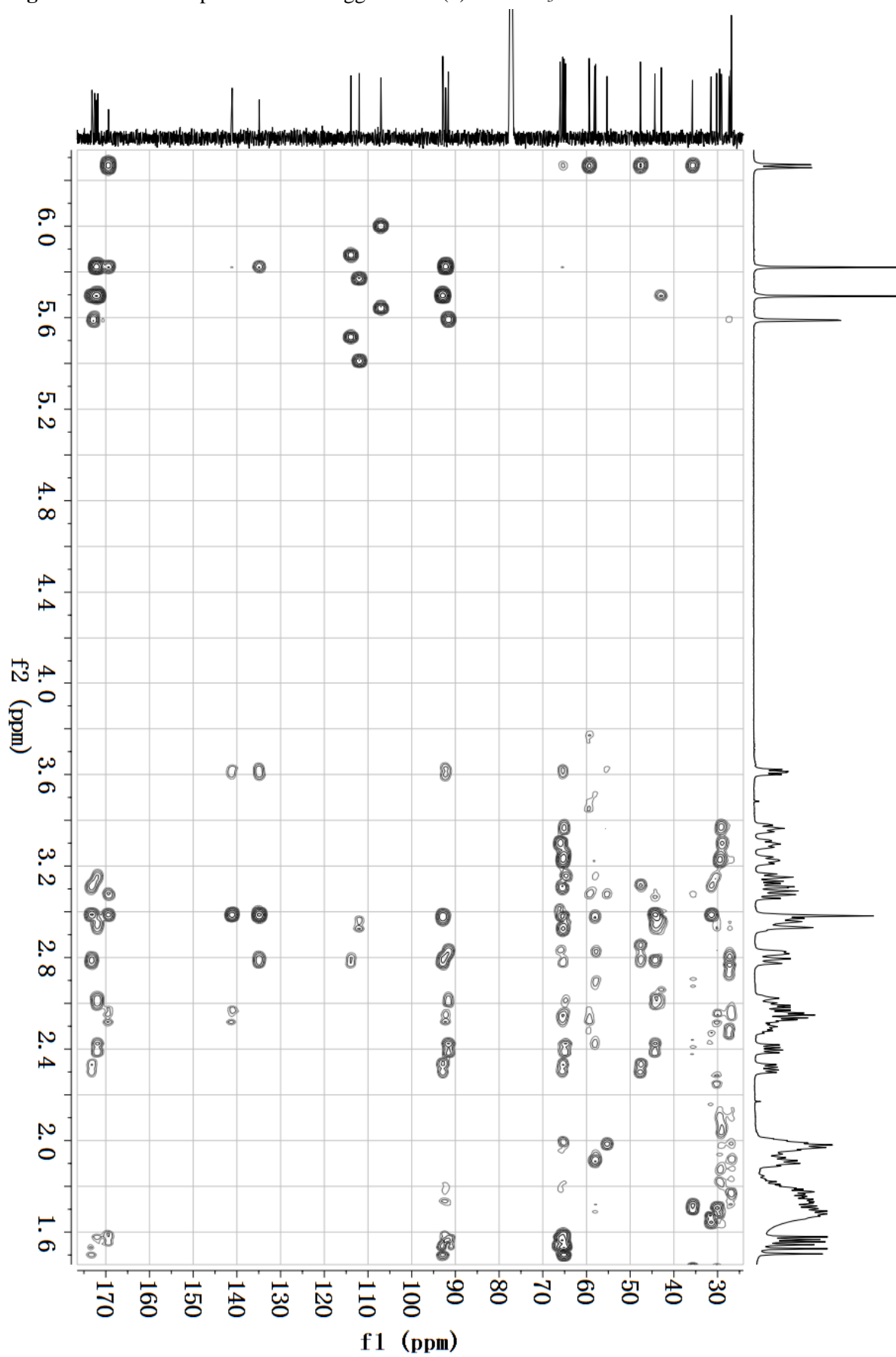


Figure S78. ROESY spectrum for flueggeine H (9) in CDCl₃.

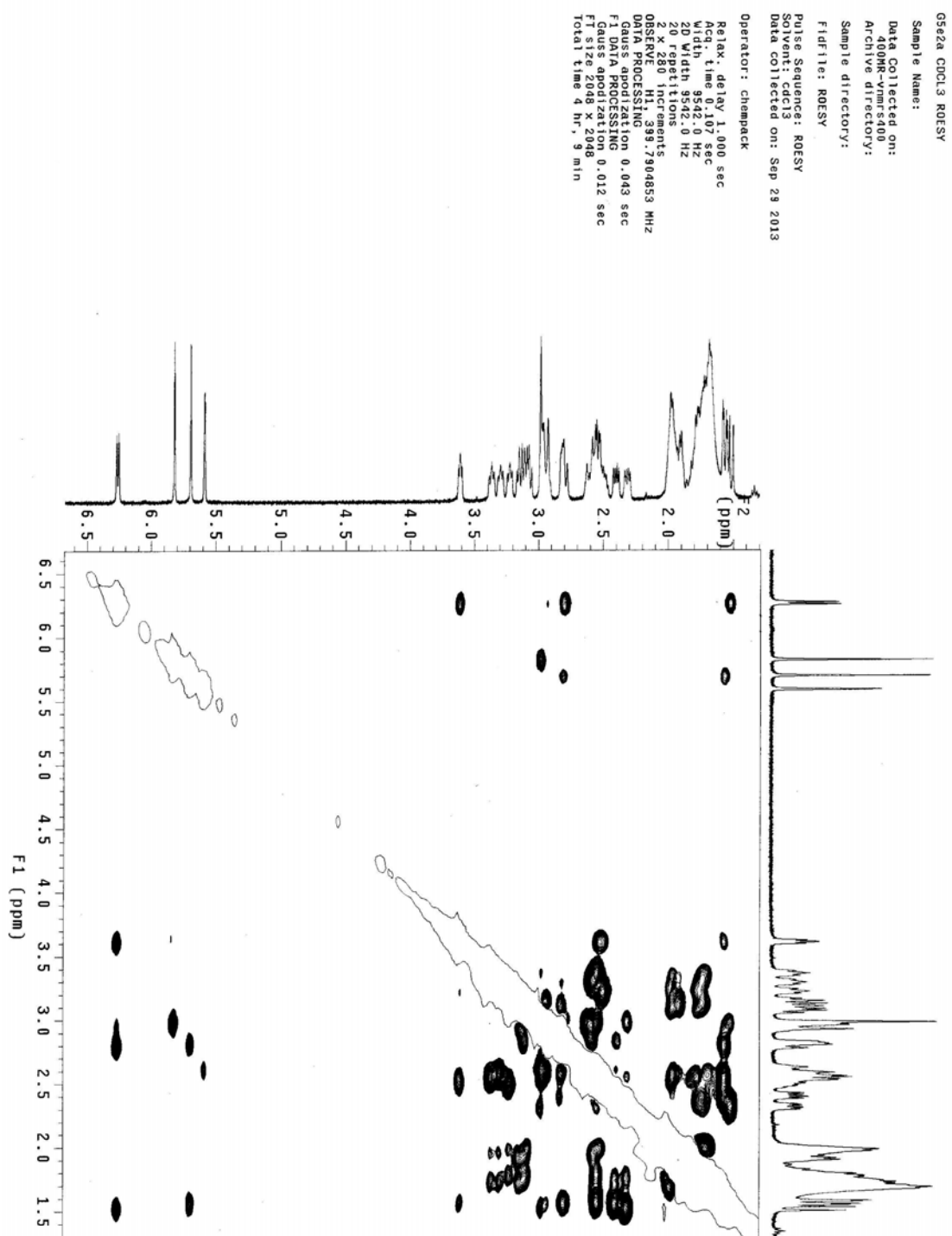


Figure S79. IR spectrum for flueggenine H (9).

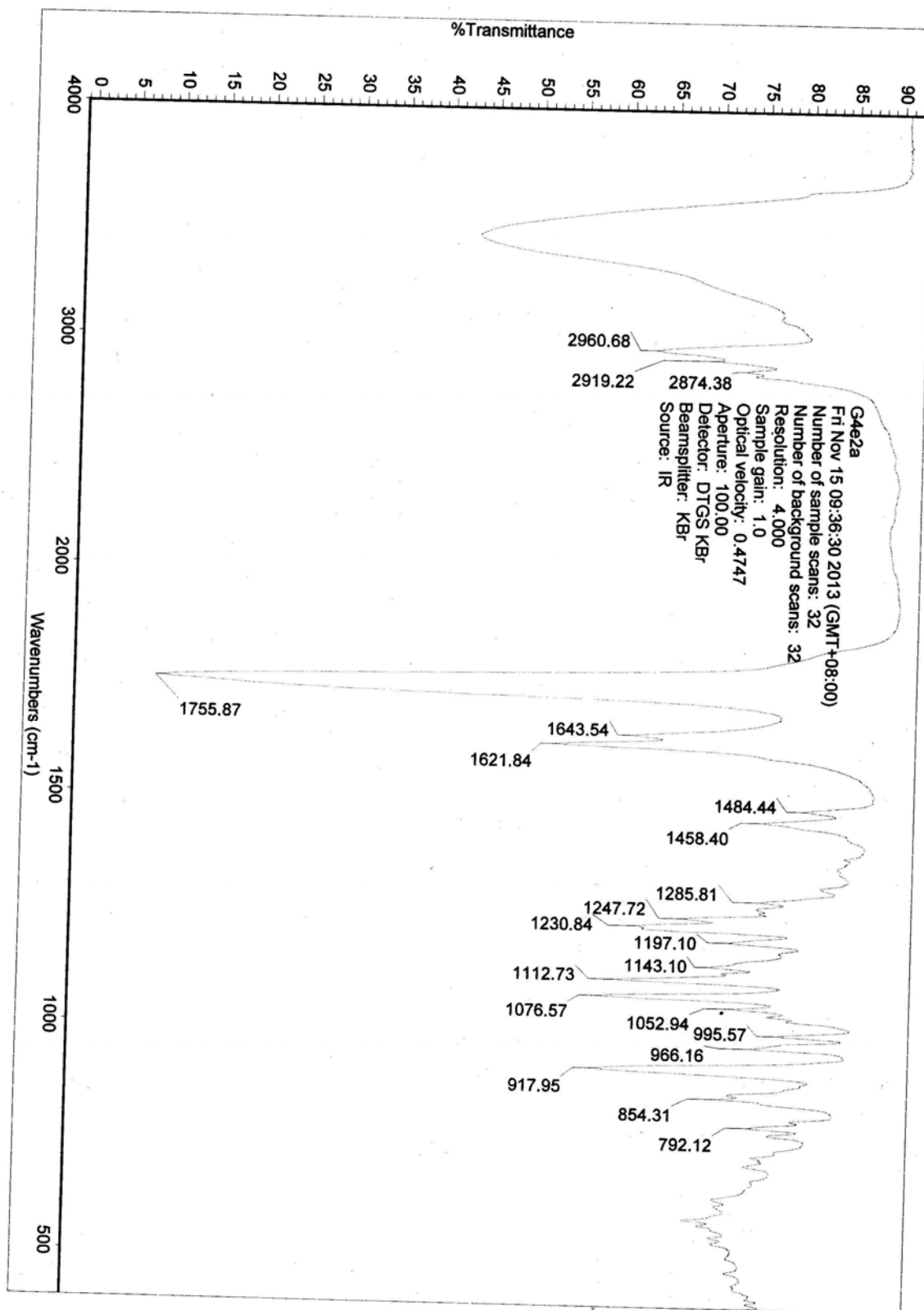


Figure S80. (+)-ESIMS spectrum for flueggeine H (9).

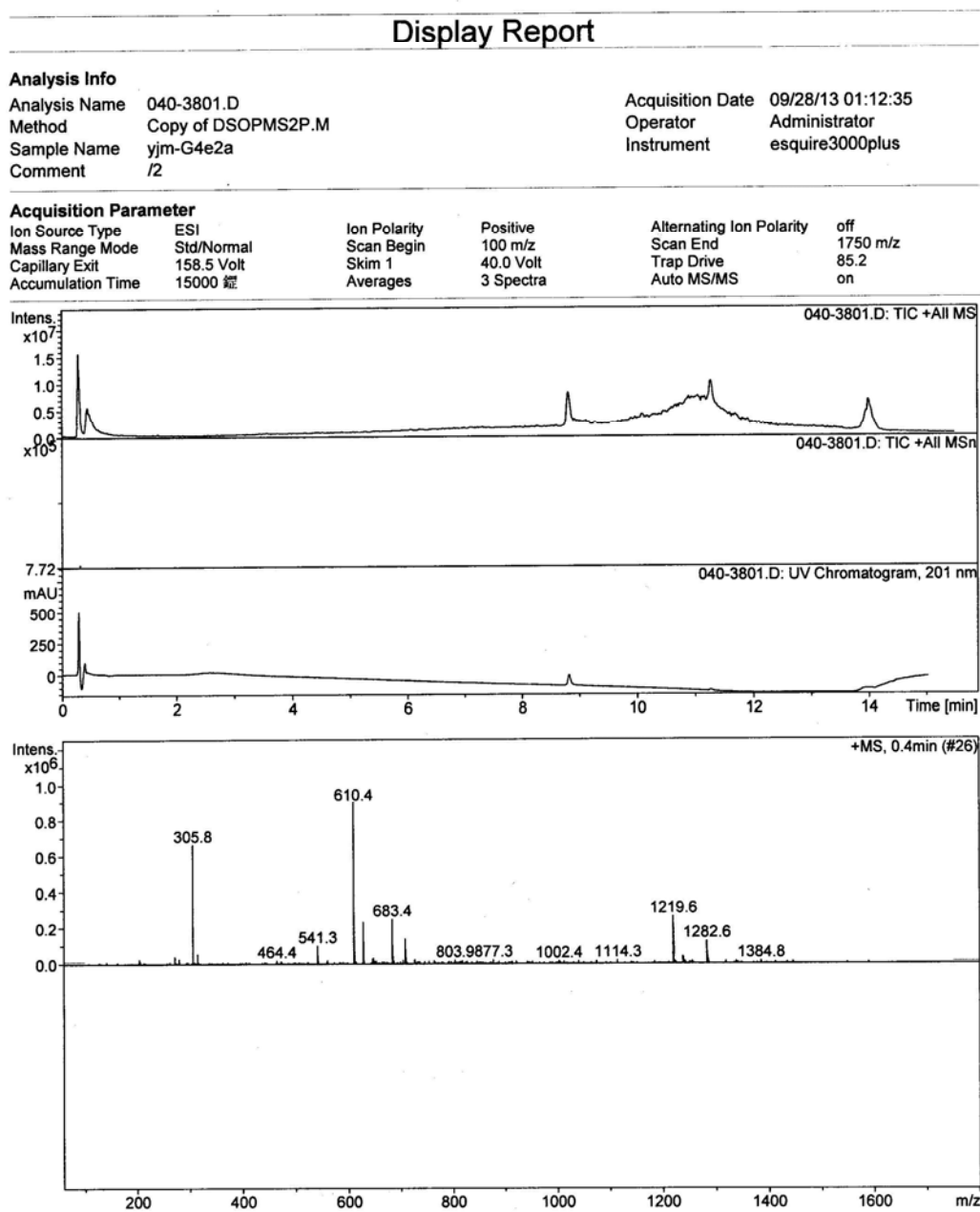


Figure S81. (+)-HRESIMS spectrum for fluegenine H (**9**).

Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

628 formula(e) evaluated with 2 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 5-80 H: 2-120 N: 0-4 O: 0-20

G4ge2a

LCT PXE KE324

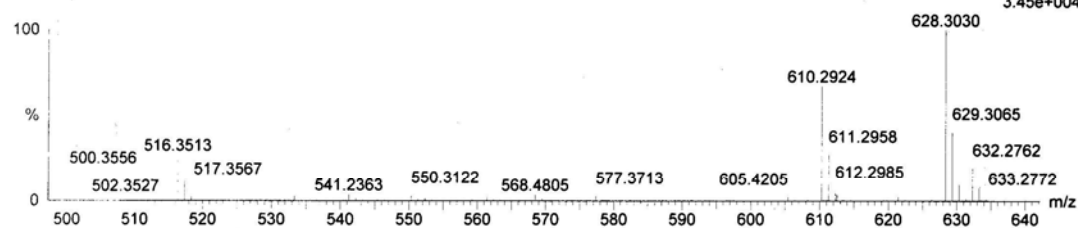
27-Sep-2013

10:18:25

1: TOF MS ES+

3.45e+004

G4e2a_0927 31 (0.670) AM2 (Ar,11000.0,0.00,0.70); ABS; Cm (13:32)



Minimum: -1.5
Maximum: 3.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
610.2924	610.2922	0.2	0.3	0.5	145.6	7.5	C23 H48 N O17
	610.2917	0.7	1.1	18.5	138.1	0.0	C36 H40 N3 O6 ✓

Figure S83. ^{13}C NMR spectrum for flueggein I (**10**) in CDCl_3 .

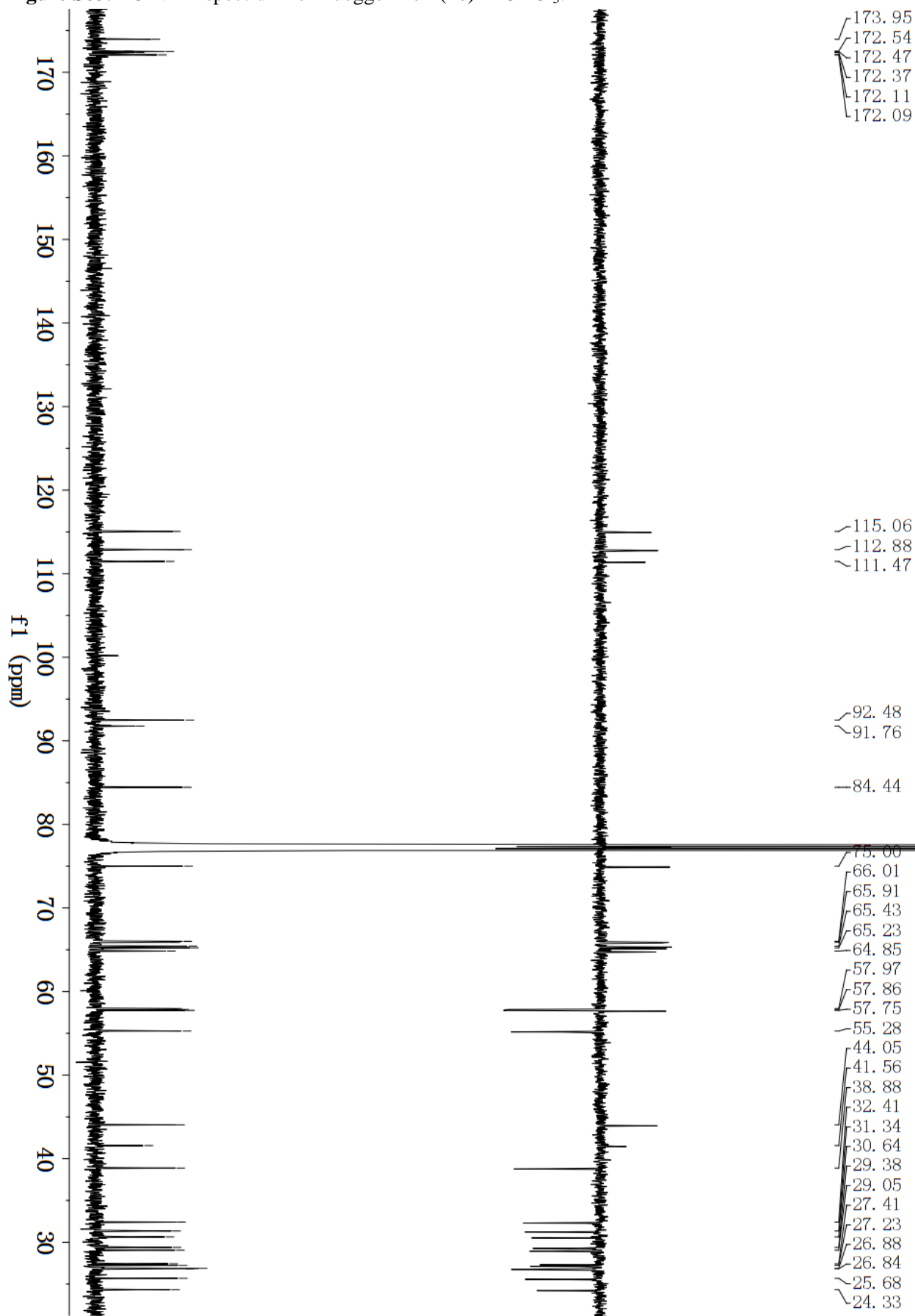


Figure S85. HSQC spectrum for flueggenine I (10) CDCl₃.

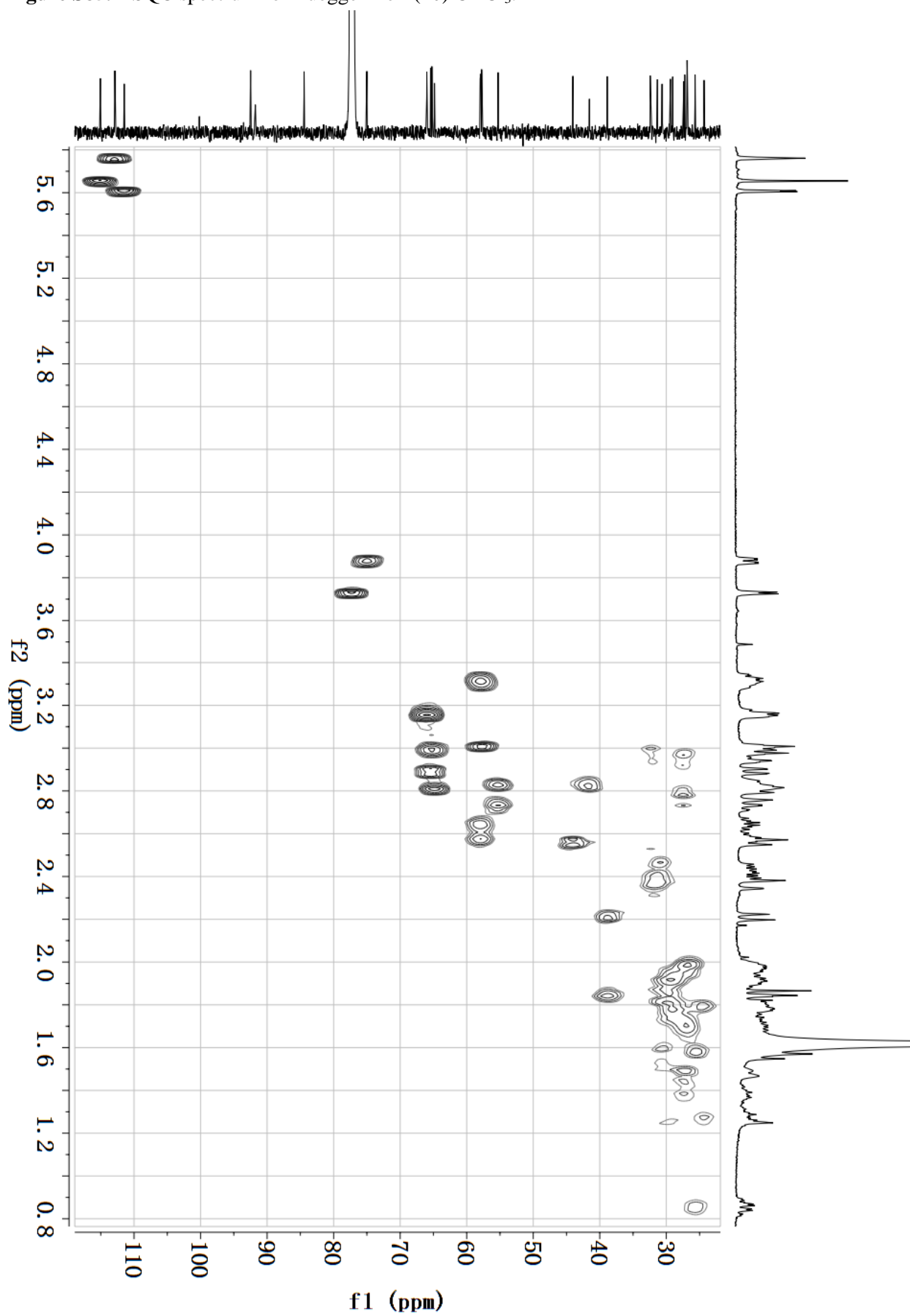


Figure S86. HMBC spectrum for flueggeine I (10) in CDCl₃.

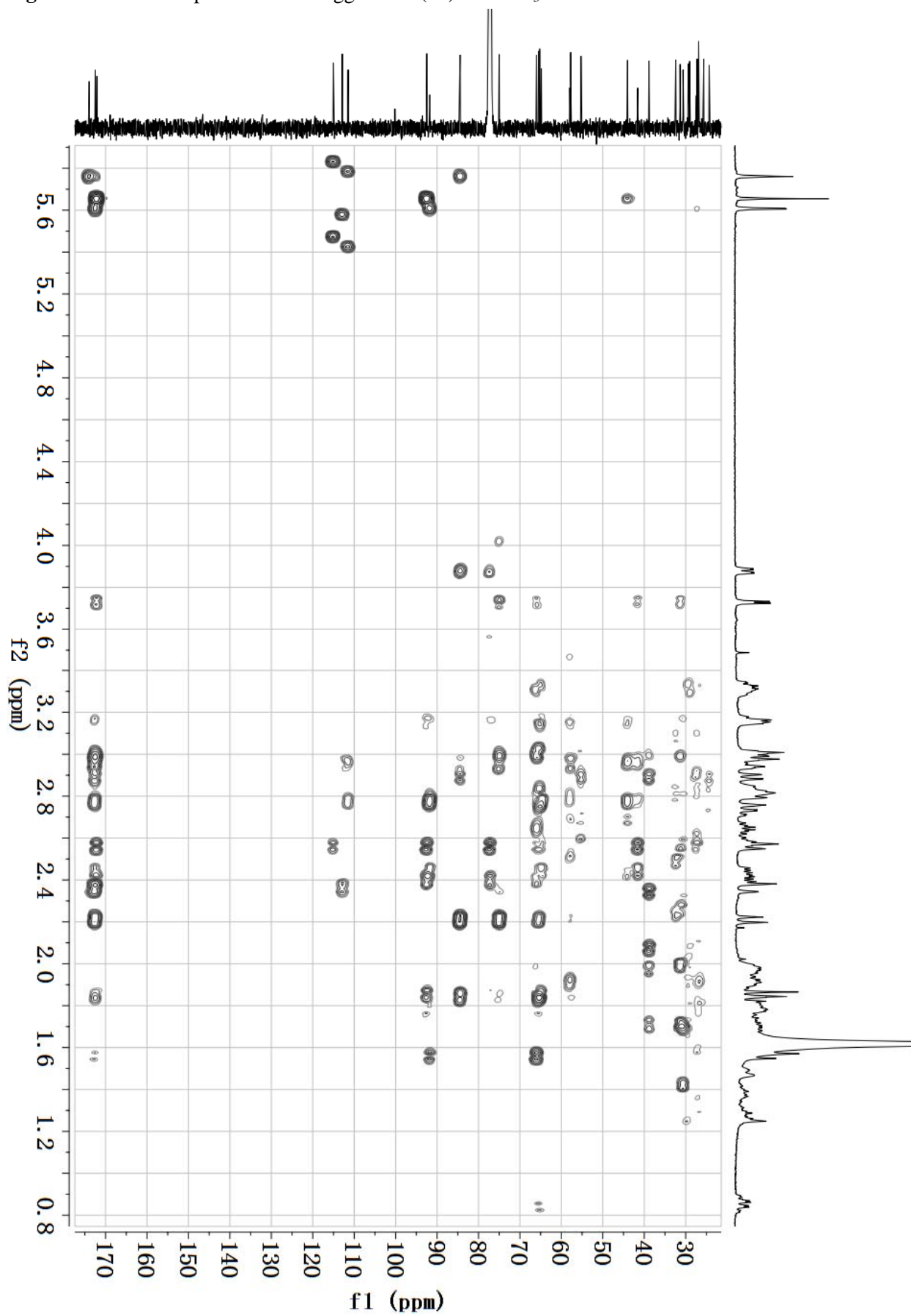


Figure S87. ROESY spectrum for flueggein I (10) in CDCl₃.

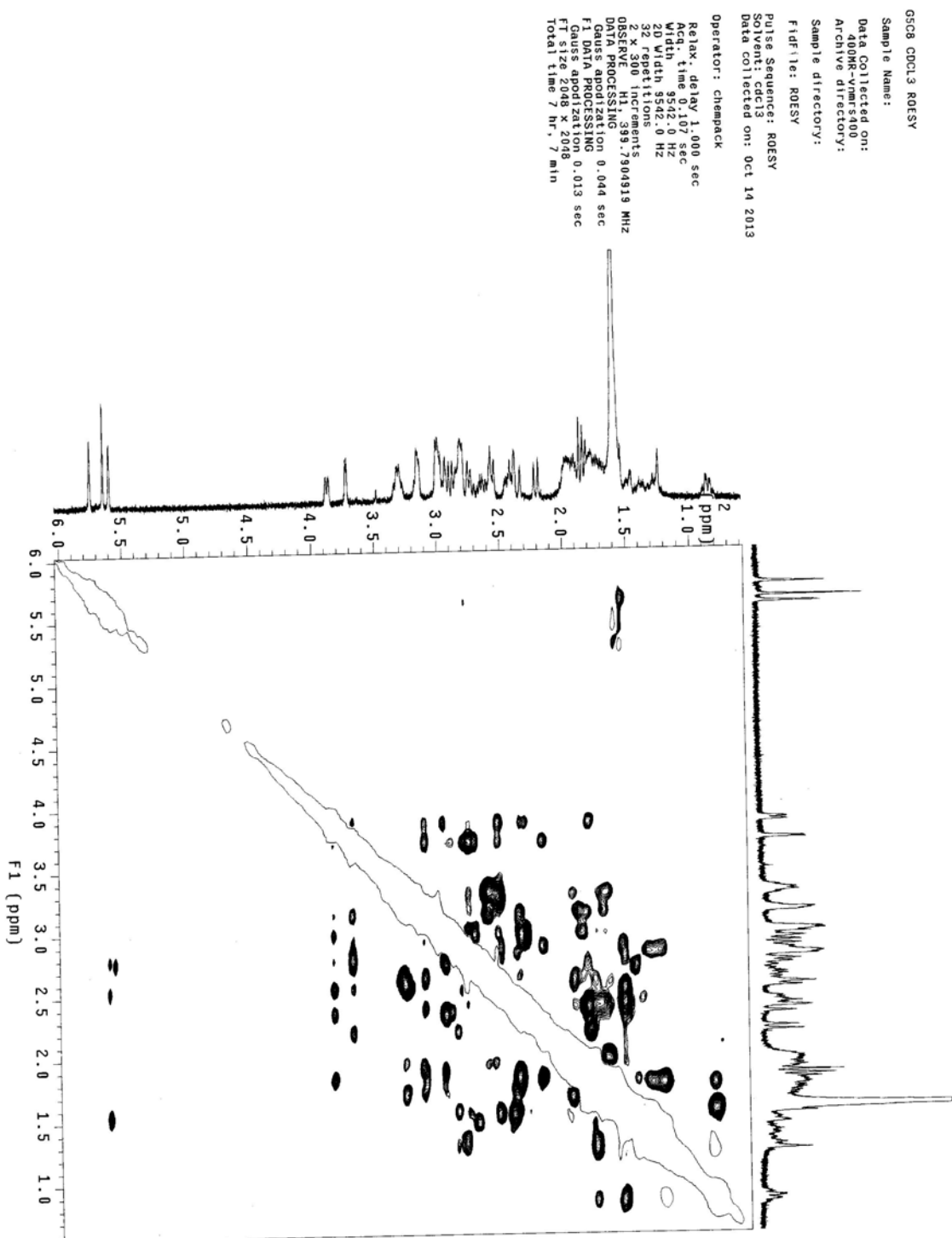


Figure S88. IR spectrum for flueggenine I (10).

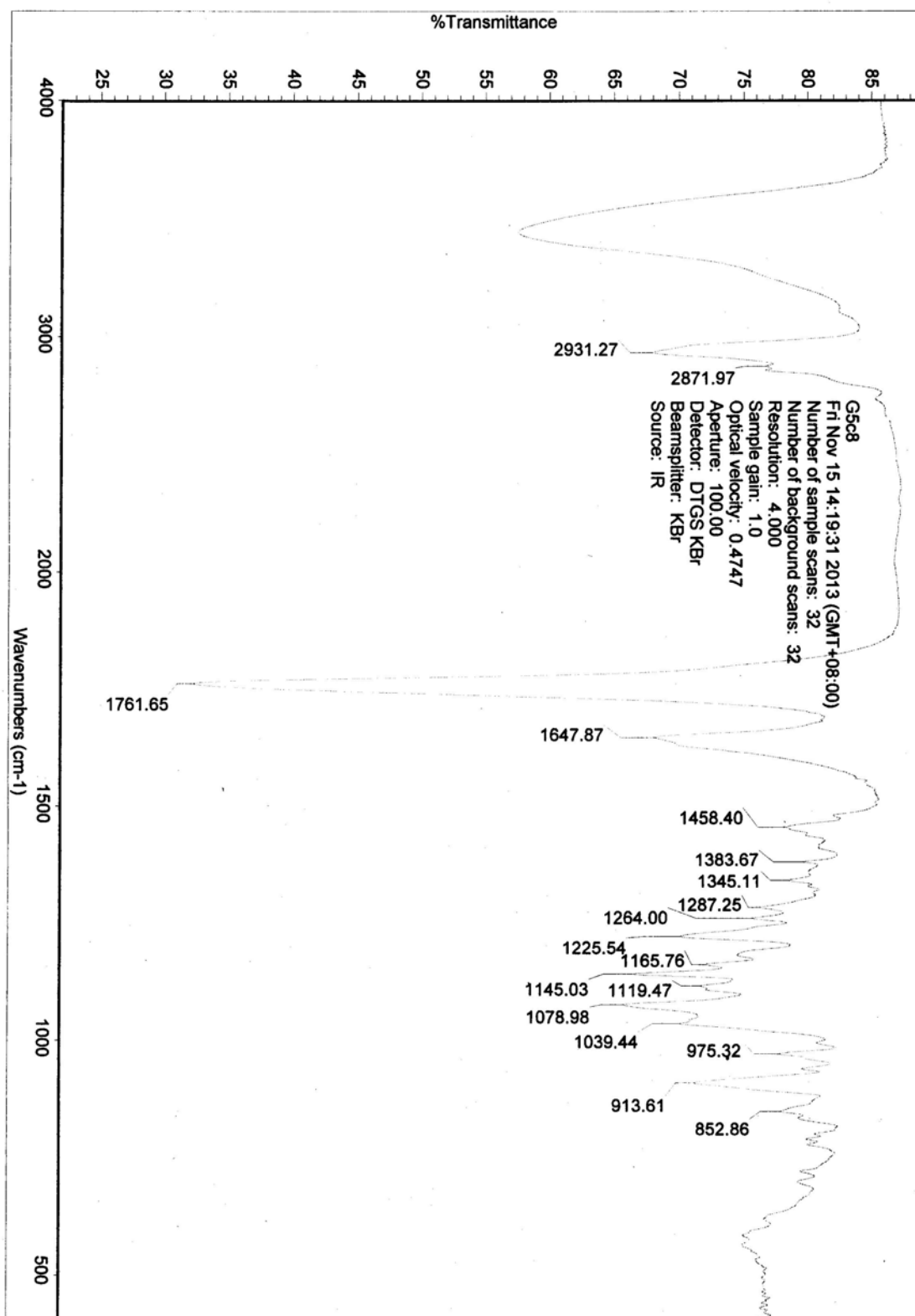


Figure S89. (+)-ESIMS spectrum for flueggein I (10).

Display Report

Analysis Info

Analysis Name 042-8701.D
Method Copy of DSOPMS2P.M
Sample Name yjm-G5C8
Comment 囁

Acquisition Date 10/16/13 08:38:45
Operator Administrator
Instrument esquire3000plus

Acquisition Parameter

Ion Source Type	ESI	Ion Polarity	Positive	Alternating Ion Polarity	off
Mass Range Mode	Std/Normal	Scan Begin	100 m/z	Scan End	1750 m/z
Capillary Exit	158.5 Volt	Skim 1	40.0 Volt	Trap Drive	85.2
Accumulation Time	15000 經	Averages	3 Spectra	Auto MS/MS	on

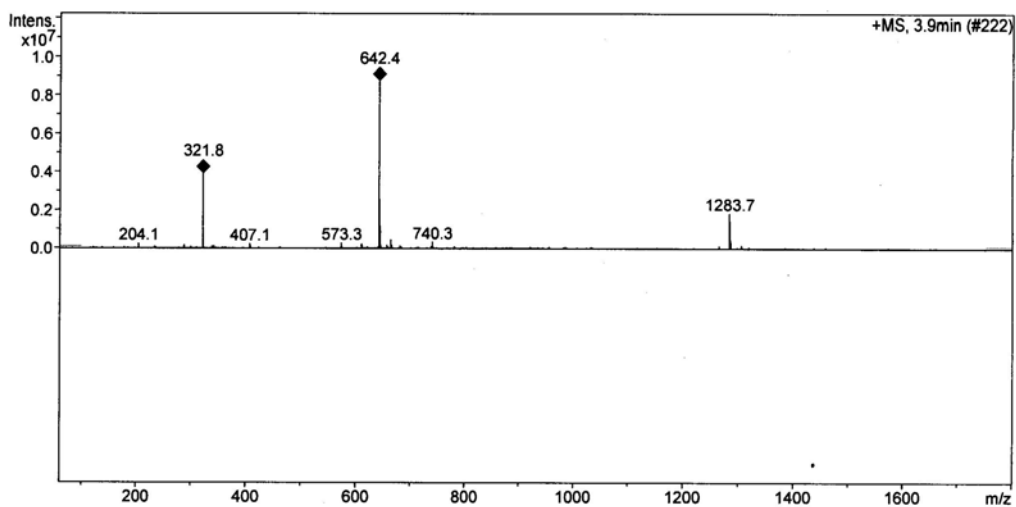
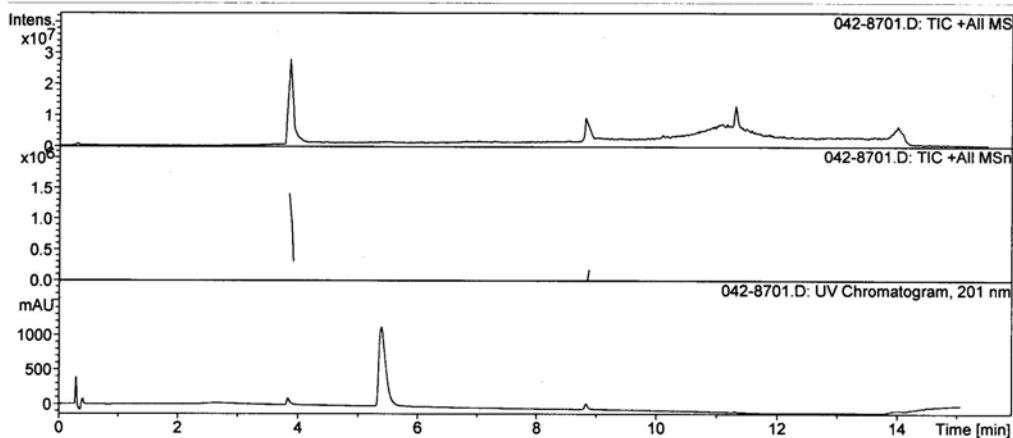


Figure S90. (+)-HRESIMS spectrum for flueggenine I (10).

Elemental Composition Report

Single Mass Analysis

Tolerance = 3.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

668 formula(e) evaluated with 2 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 5-80 H: 2-120 N: 0-4 O: 0-20

GEC8

LCT PXE KE324

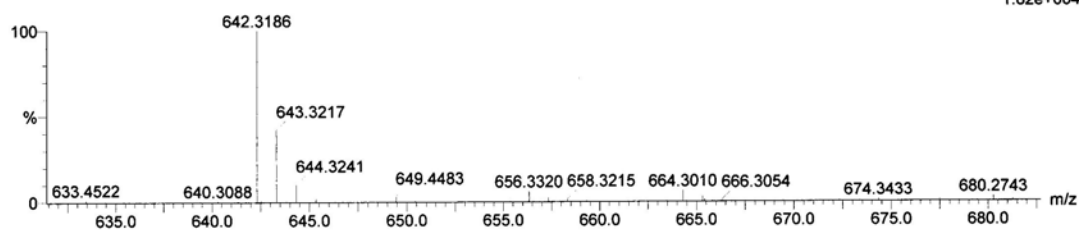
12-May-2015

14:18:17

1: TOF MS ES+

1.82e+004

GEC8_0512 39 (0.847) AM2 (Ar,10000.0,0.00,1.00); ABS; Cm (29:43)



Minimum: -1.5
Maximum: 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
642.3186	642.3184	0.2	0.3	-0.5	101.6	8.5	C24 H52 N O18
	642.3179	0.7	1.1	17.5	93.1	0.0	C37 H44 N3 O7