

Supporting data

Ternifolipyrons A-J: New cytotoxic α -pyrones from *Isodon ternifolius* (D. Don) Kudo

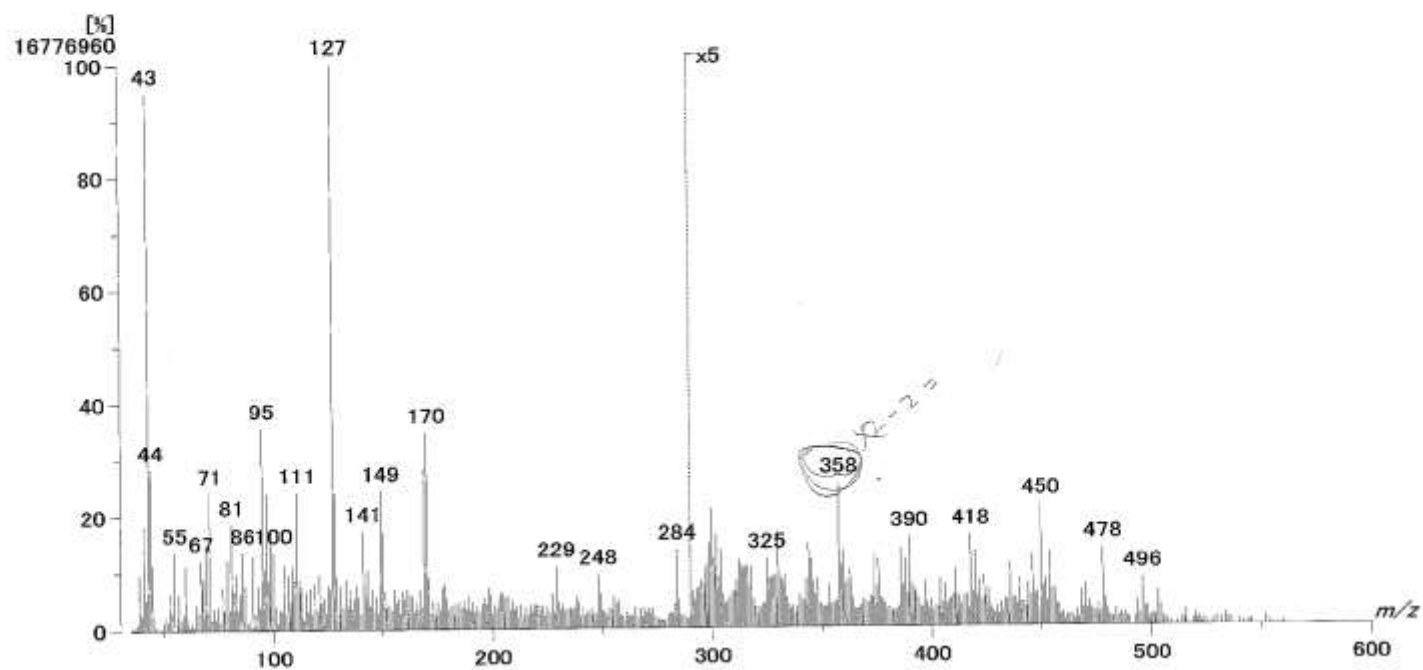
Abdelsamed I. Elshamy ^{a,*}, Tarik A. Mohamed ^c, Ningombam Swapana ^{b,d}, Yusuke Kasai^b, Masaaki Noji ^b, Thomas Efferth ^e, Hiroshi Imagawa ^c, Mohamed-Elamir F. Hegazy ^{c,e,**}, Akemi Umeyama ^{b,*}

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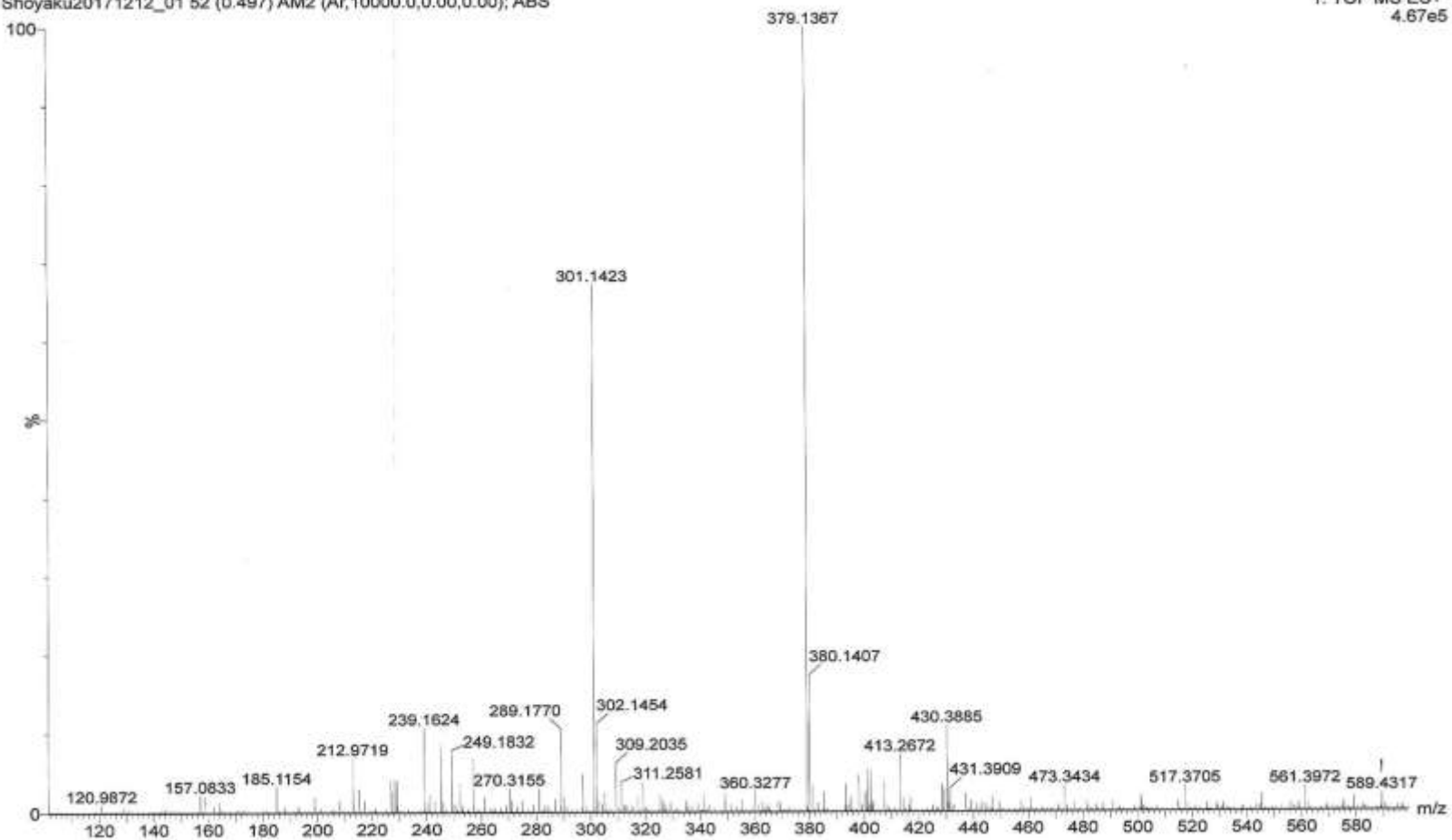
[Mass Spectrum]
Data : Umeyama-EL08-Dec-2017.004 Date : 08-Dec-2017 10:16
Sample : YGS-EtOAc-fr24-26-fr71-73
Note : MStation
Inlet : Direct Ion Mode : EI+
Spectrum Type : Normal Ion [MF-Linear]
RT : 1.97 min Scan# : 60
BP : m/z 127 Int. : 1599.98 (16776960)
Output m/z range : 35 to 600 Cut Level : 0.00 %



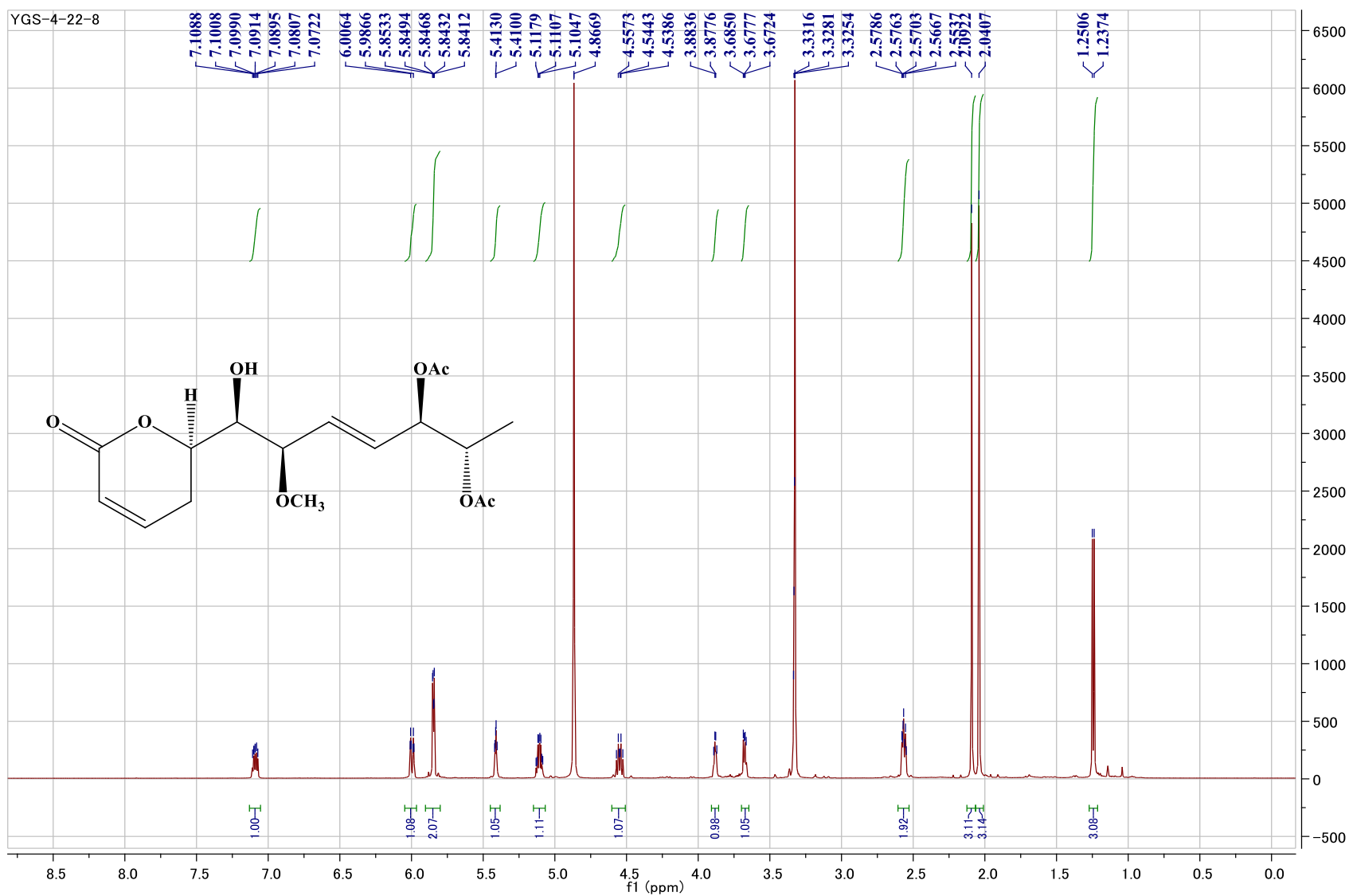
S1: LREIMS of 1

YGS-2-71-73 (Elshamy)
Shoyaku20171212_01 52 (0.497) AM2 (Ar,10000.0,0.00,0.00); ABS

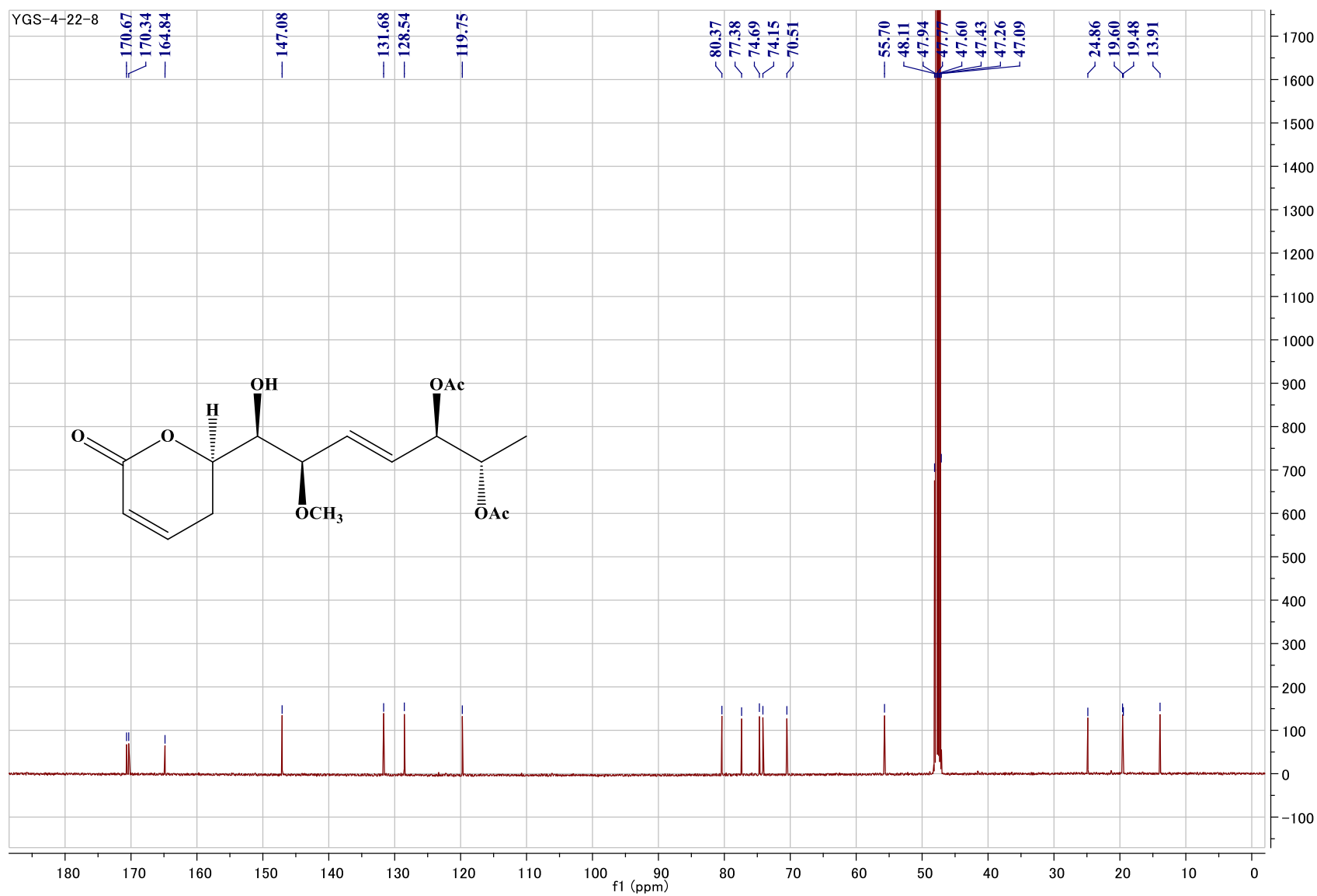
1: TOF MS ES+
4.67e5



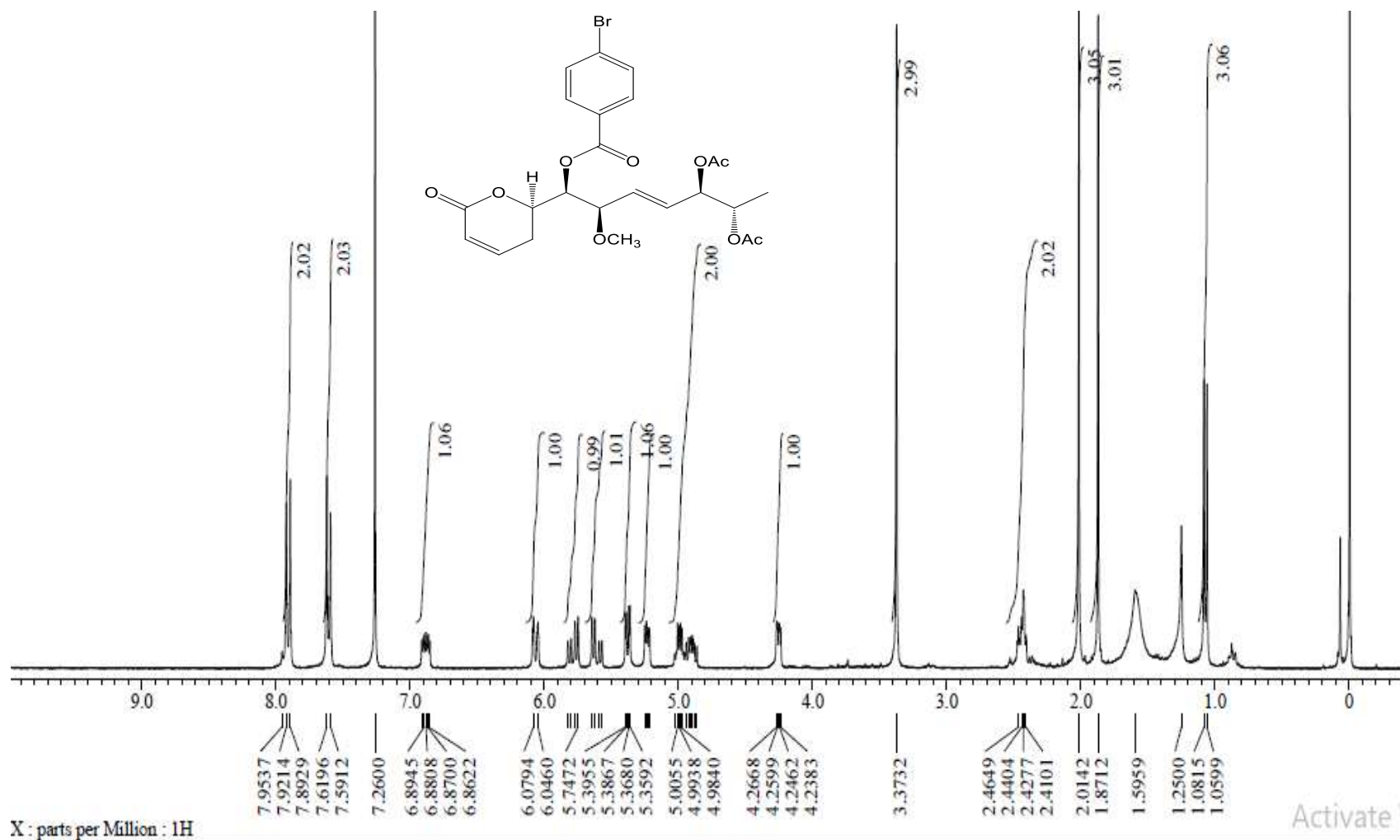
S2: TOESIMS of 1



S3: ^1H NMR of **1**

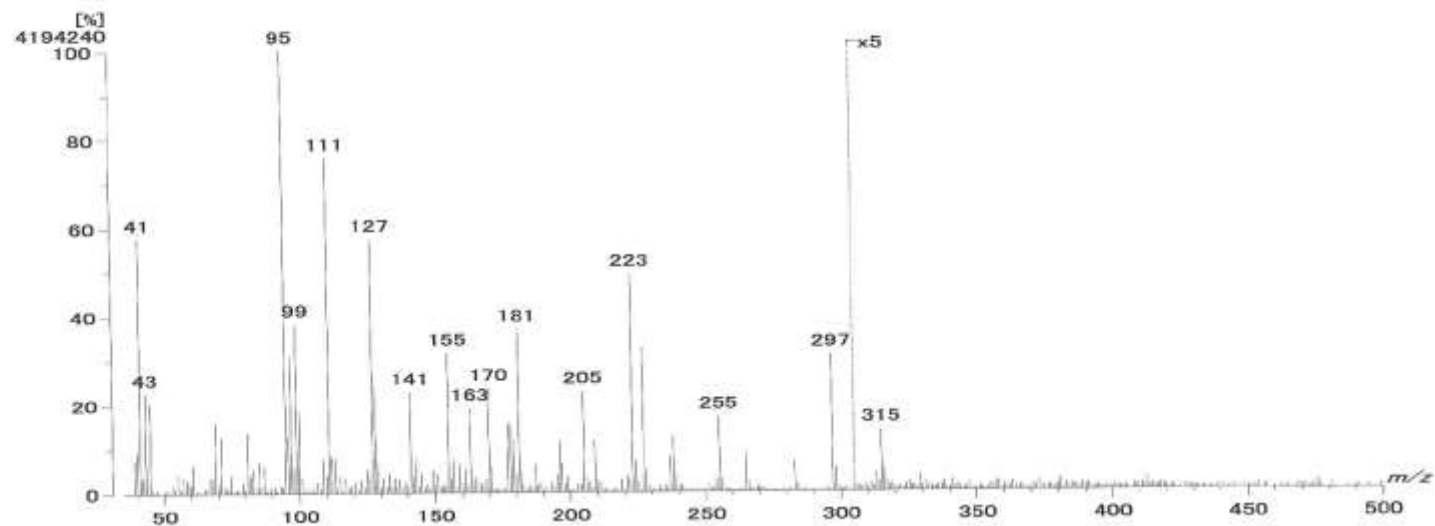


S4: ^{13}C NMR of 1



S5: ¹H NMR of (6*R*, 5'*R*, 6'*S*, 1'*R*, 2'*R*)-6-[5',6'-diacetyloxy-1'-(4-bromobenzoyl)oxy]-2'-methoxy-3*E*-heptenyl]-5,6-dihydro-2*H*-pyran-2-one

[Mass Spectrum]
 Data : Umeyama-CI.22-Feb-2018.003 Date : 22-Feb-2018 11:08
 Sample : YGS-4-22-4(CH4)
 Note : MStation
 Inlet : Direct Ion Mode : CI+
 Spectrum Type : Normal Ion [MF-Linear]
 RT : 1.28 min Scan# : 48
 BP : m/z 95 Int. : 399.99 (4194240)
 Output m/z range : 35 to 500 Cut Level : 0.00 %



S6: LRCIMS of 2

Data : Umeyama-CIHR.22-Feb-2018.001

Date : 22-Feb-2018 19:02

Instrument : MStation

Sample : YGS-4-22-4

Note : MStation

Inlet : Direct Ion Mode : CI+

RT : 1.17 min Scan# : 29

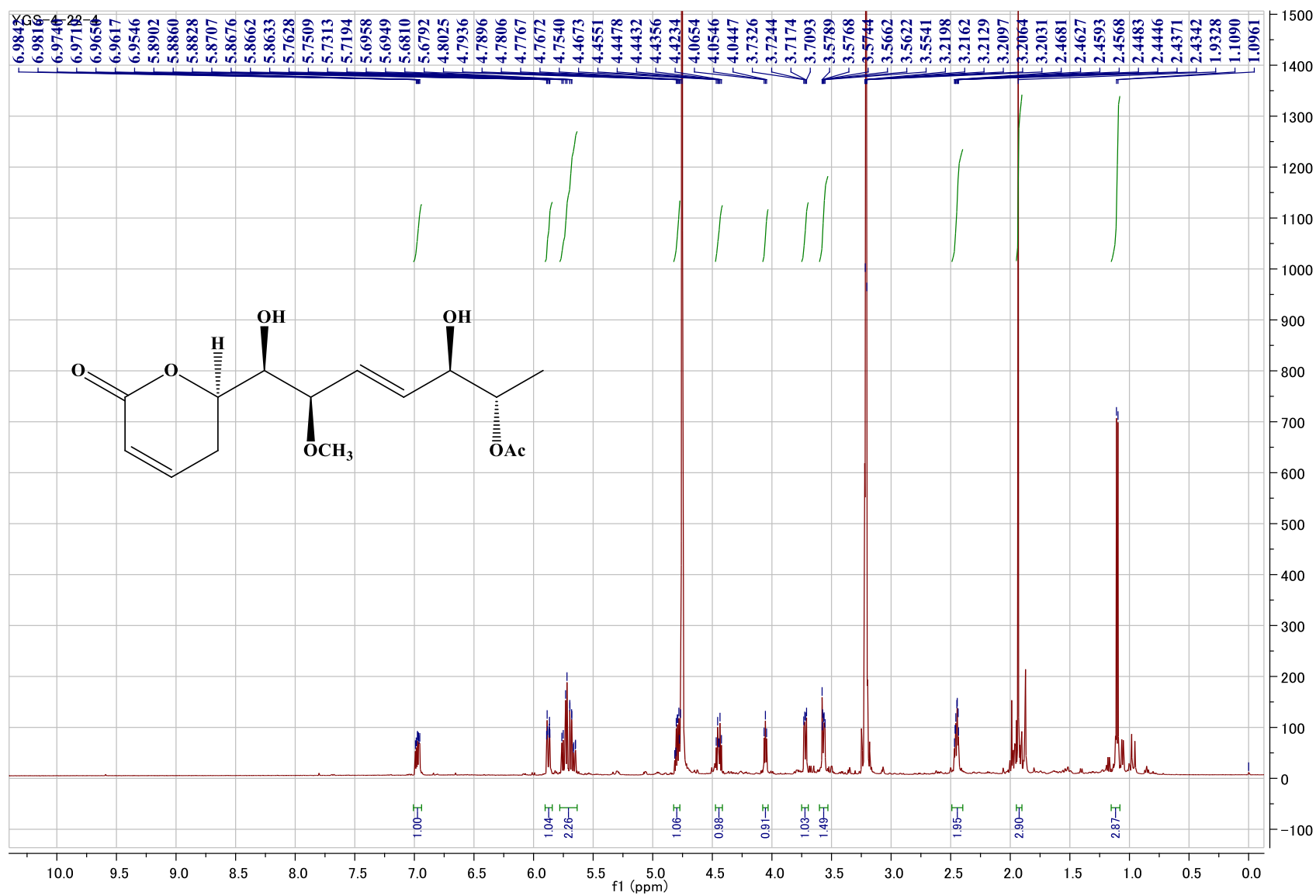
Elements : C 150/0, H 250/0, O 50/0

Mass Tolerance : 5mmu

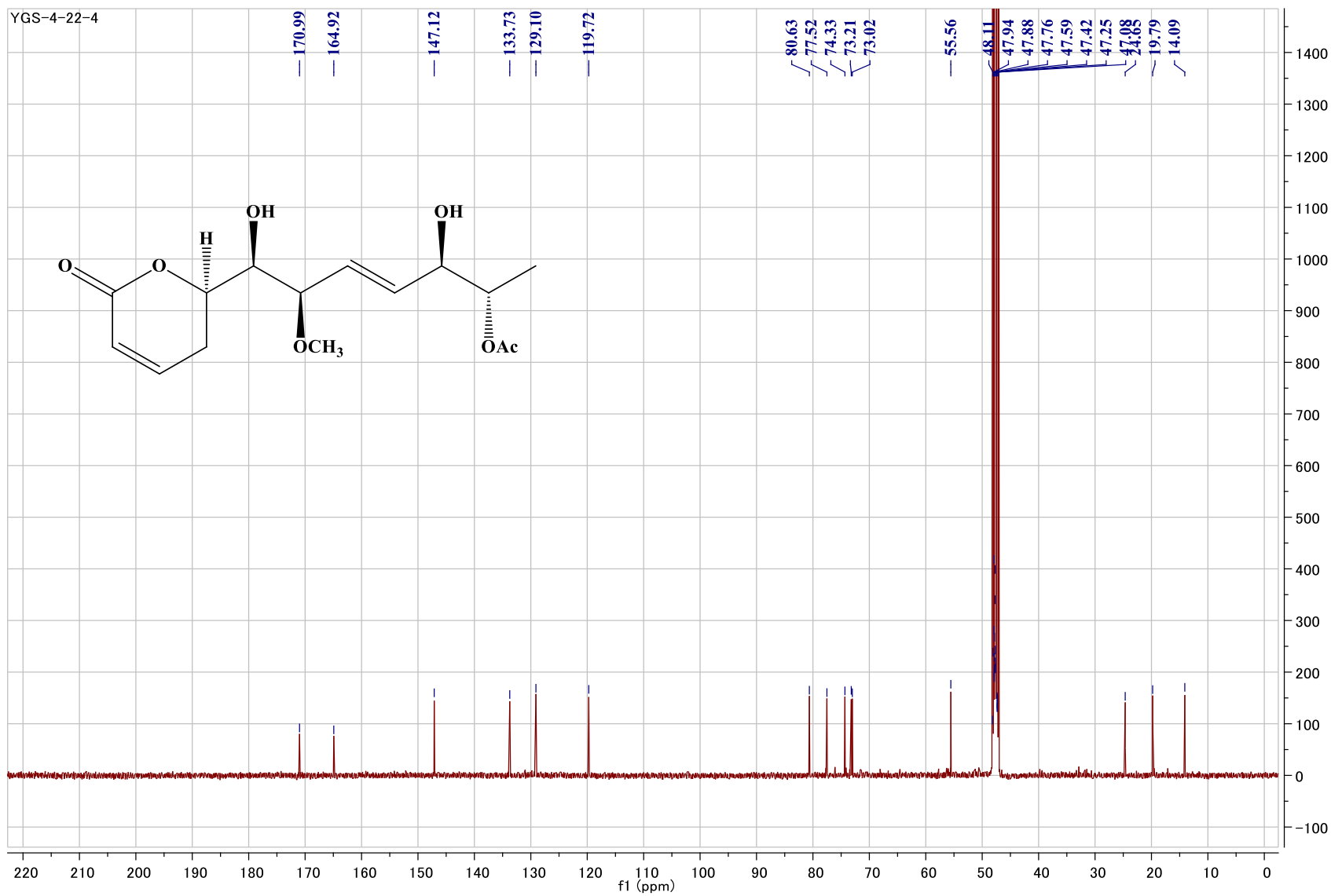
Unsaturation (U.S.) : 0.0 - 15.0

	Observed m/z	Int%	Err [ppm / mmu]	U.S. Composition
1	315.1453	10.33	+2.9 / +0.9	4.5 C15 H23 O7

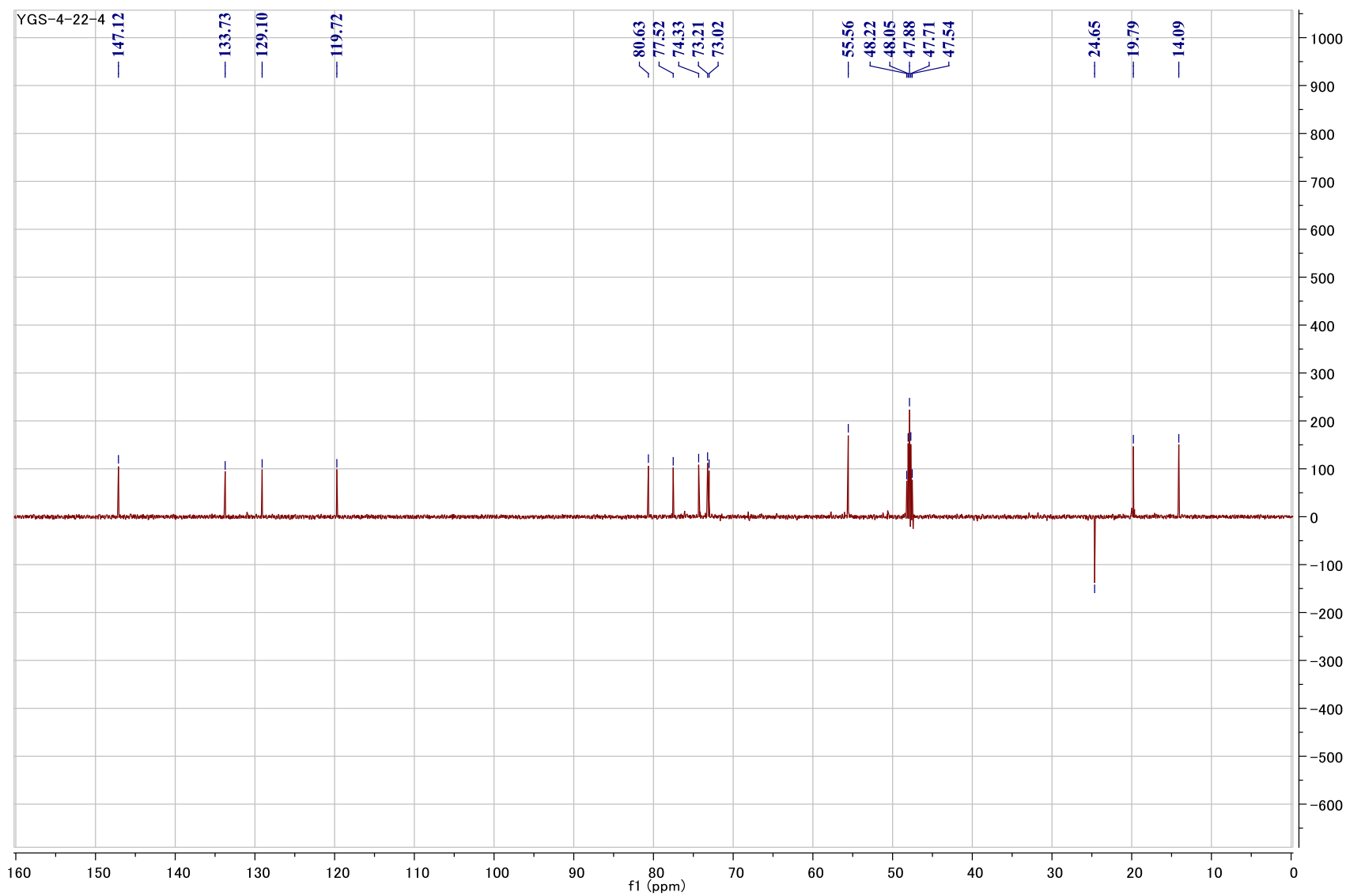
S7: HRCIMS of 2



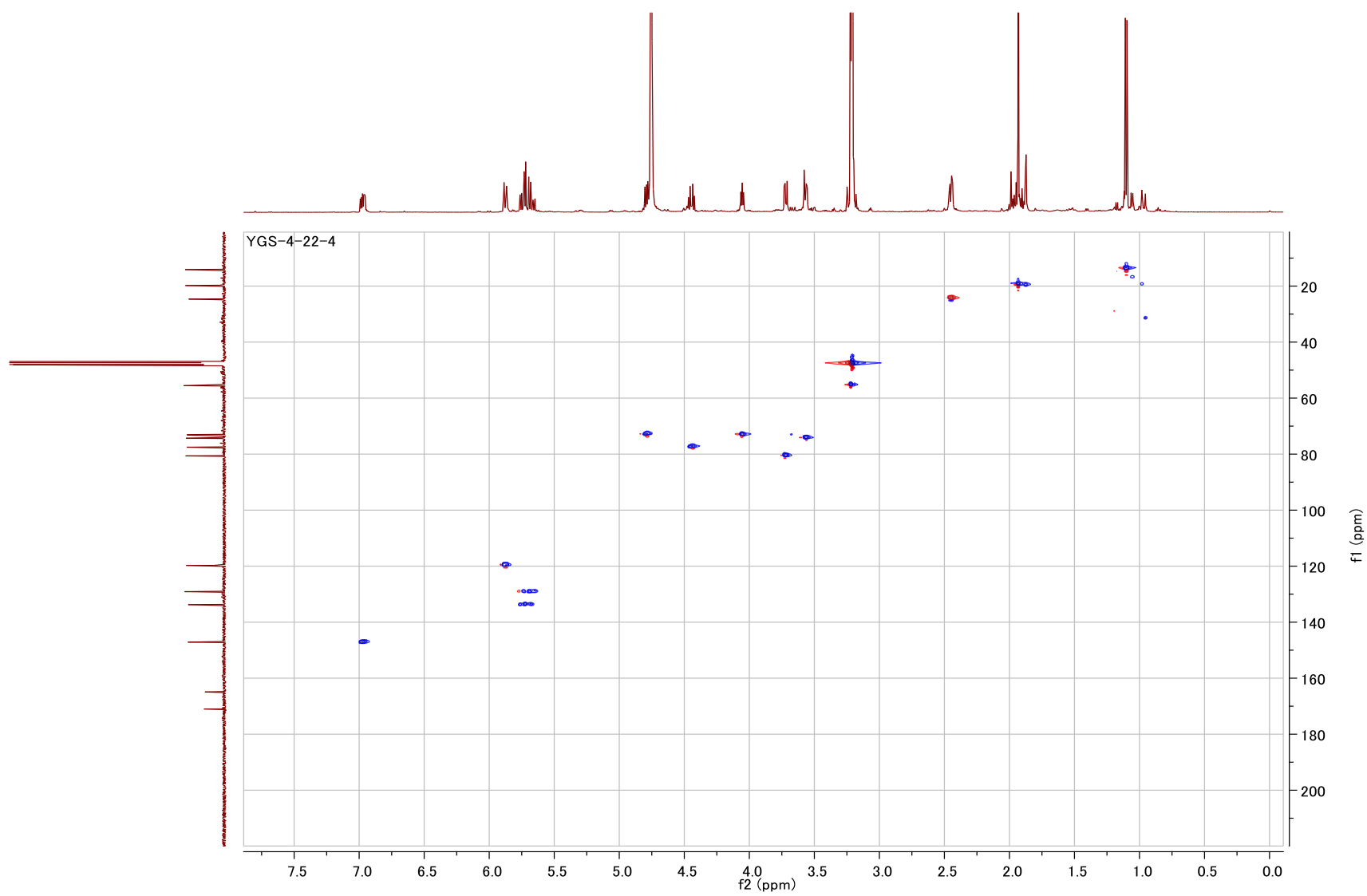
S8: ^1H NMR of 2



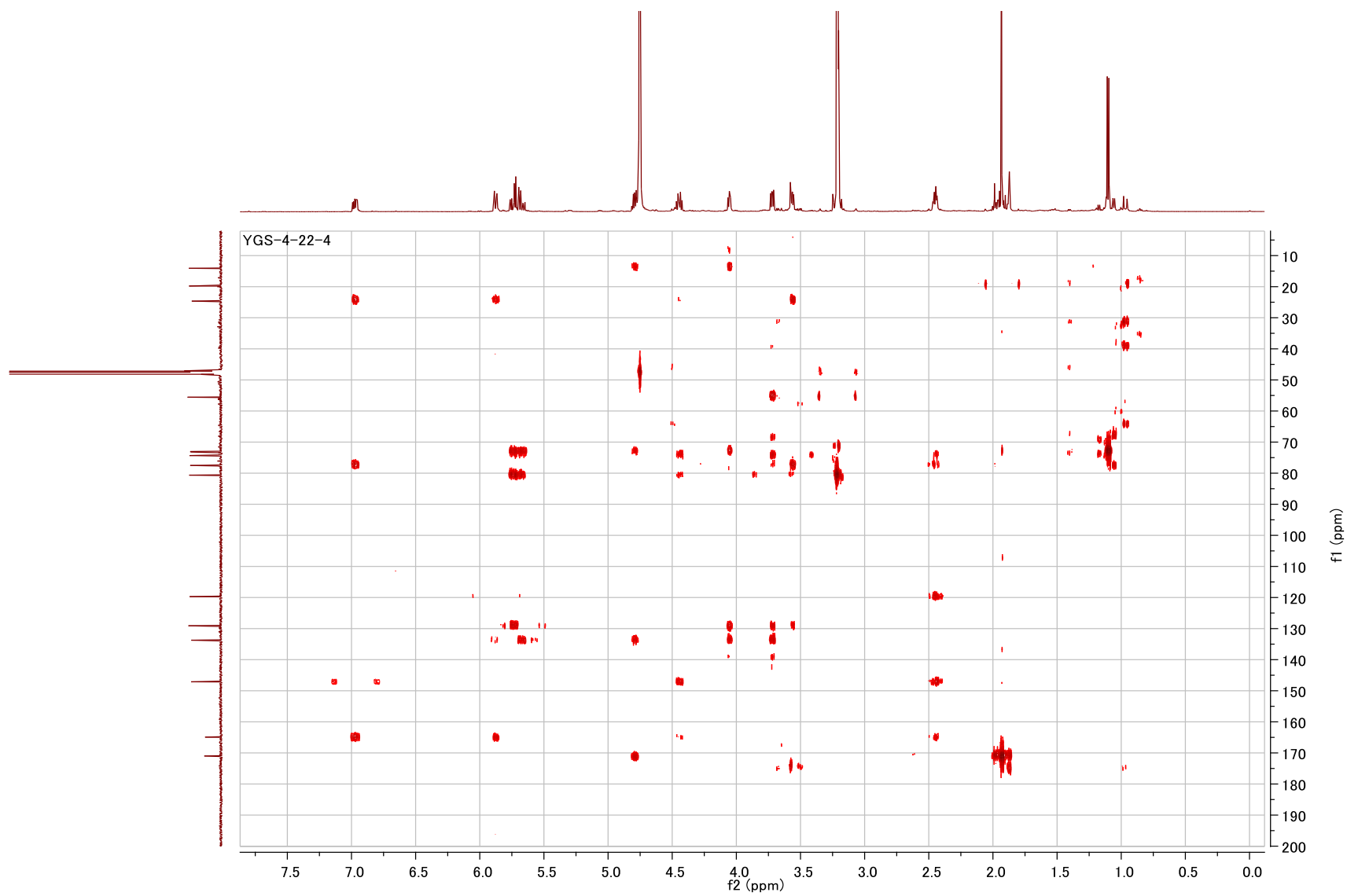
S9: ¹³C NMR of 2



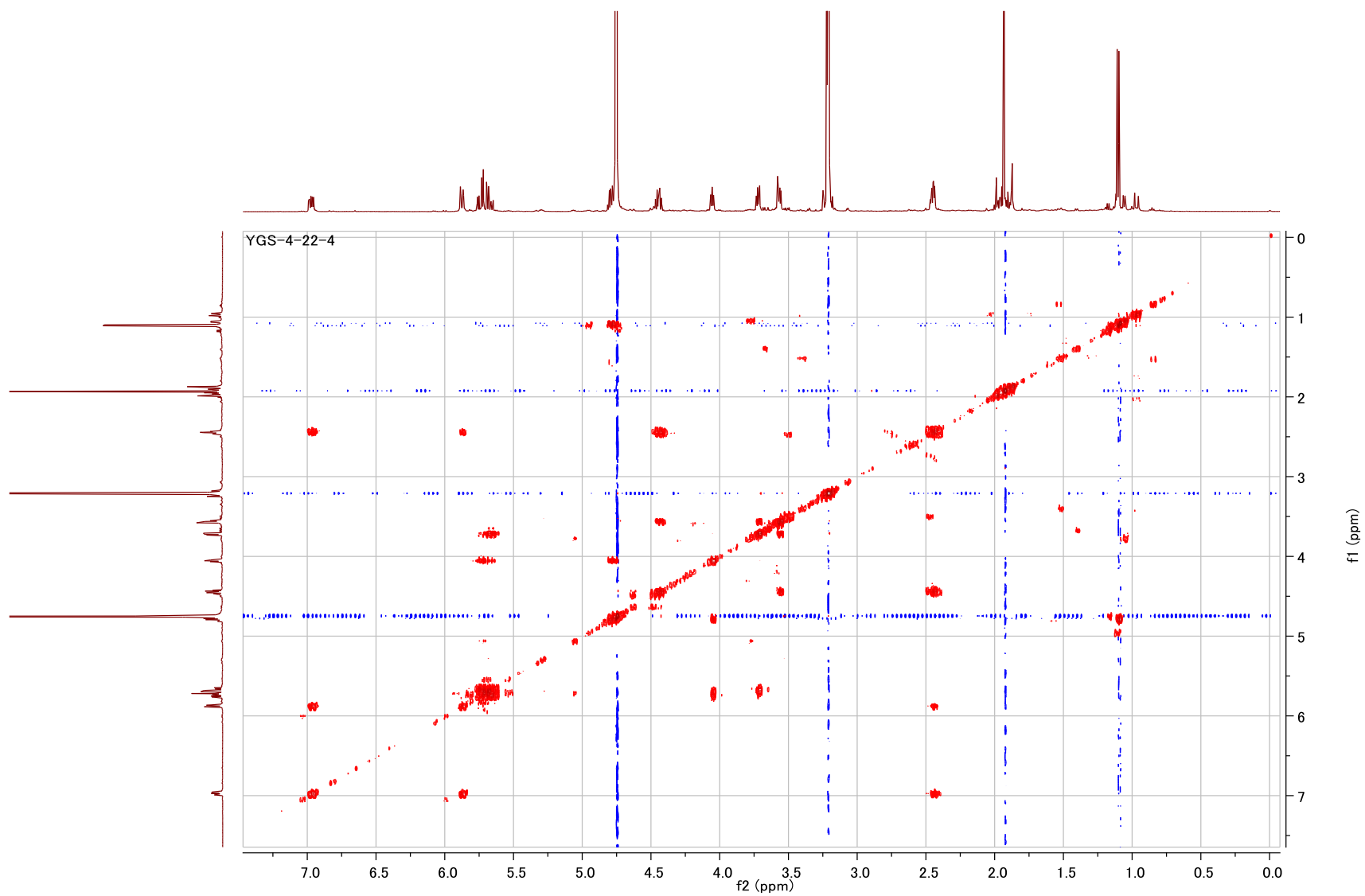
S10: DEPT-135 of 2



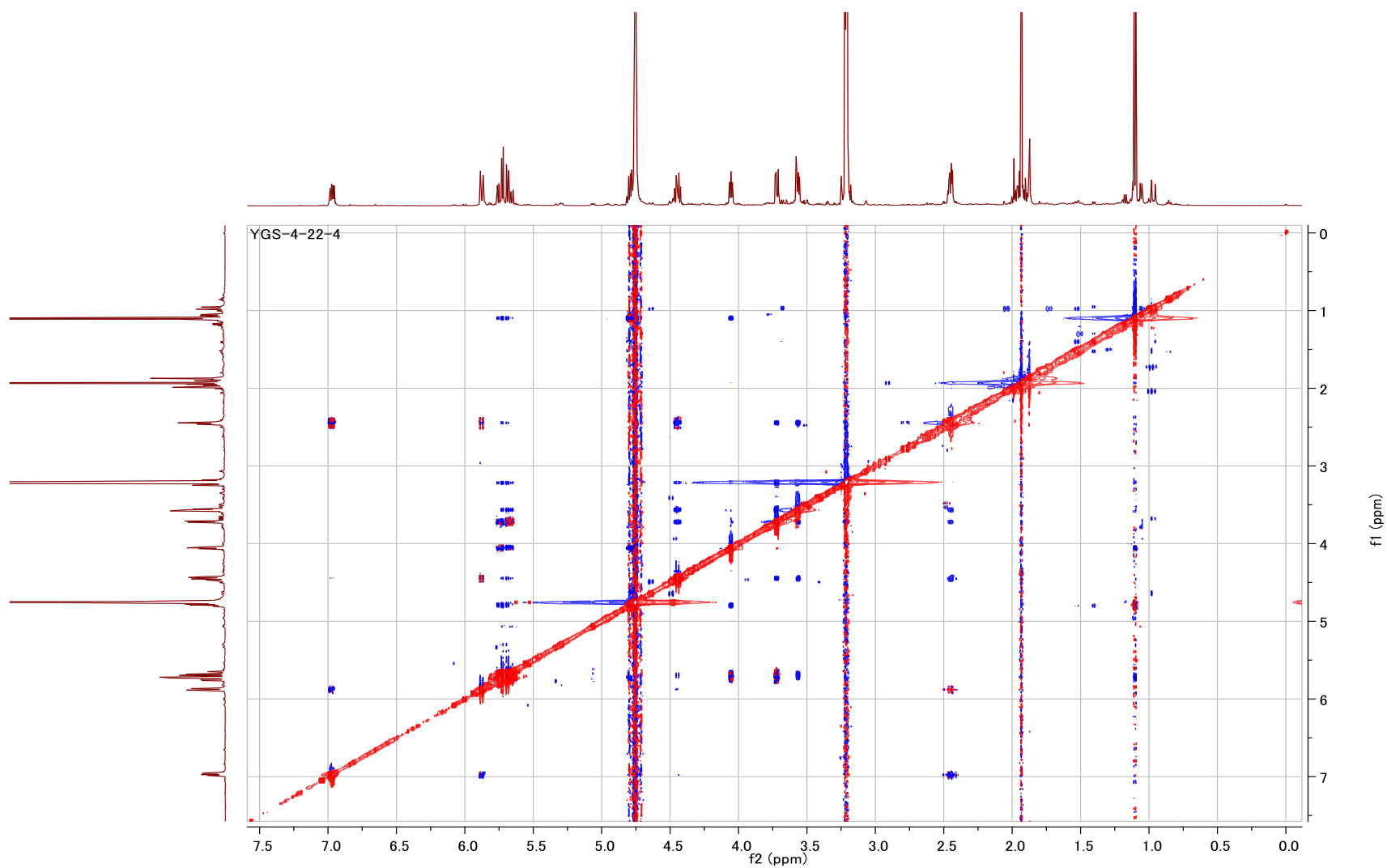
S11: HSQC of 2



S12: HMBC of 2

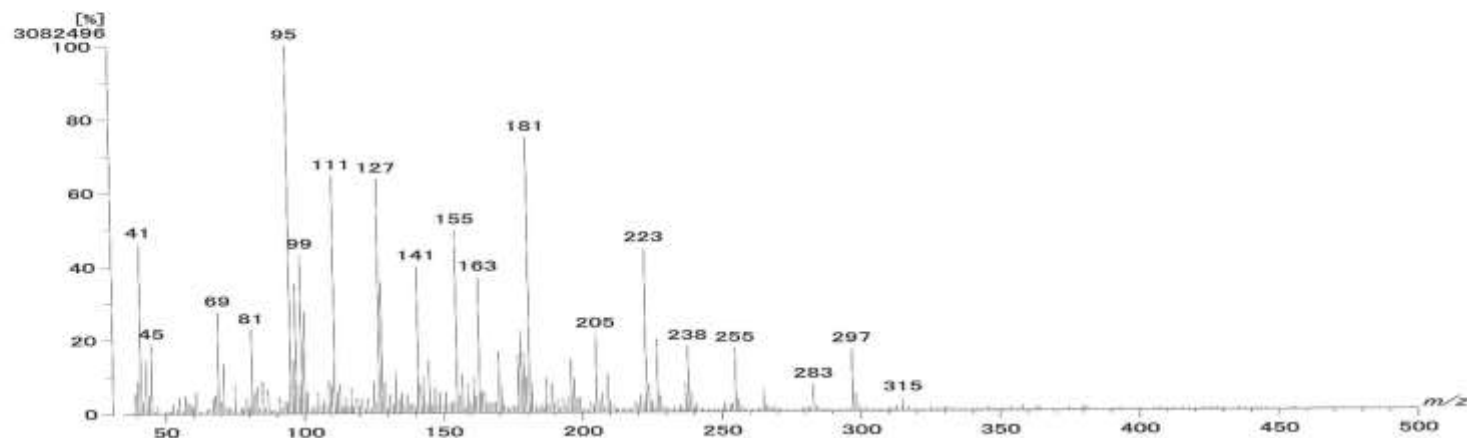


S13: ^1H ^1H COSY of 2



S14: NOESY of 2

[Mass Spectrum]
 Data : Umeyama-CI.22-Feb-2018.004 Date : 22-Feb-2018 18:21
 Sample : YGS-4-22-3(CH4)
 Note : MStation
 Inlet : Direct Ion Mode : CI+
 Spectrum Type : Normal Ion [MF=Linear]
 RT : 1.01 min Scan# : 38
 BP : m/z 95 Int : 293.97 (3082486)
 Output m/z range : 35 to 500 Cut Level : 0.00 %

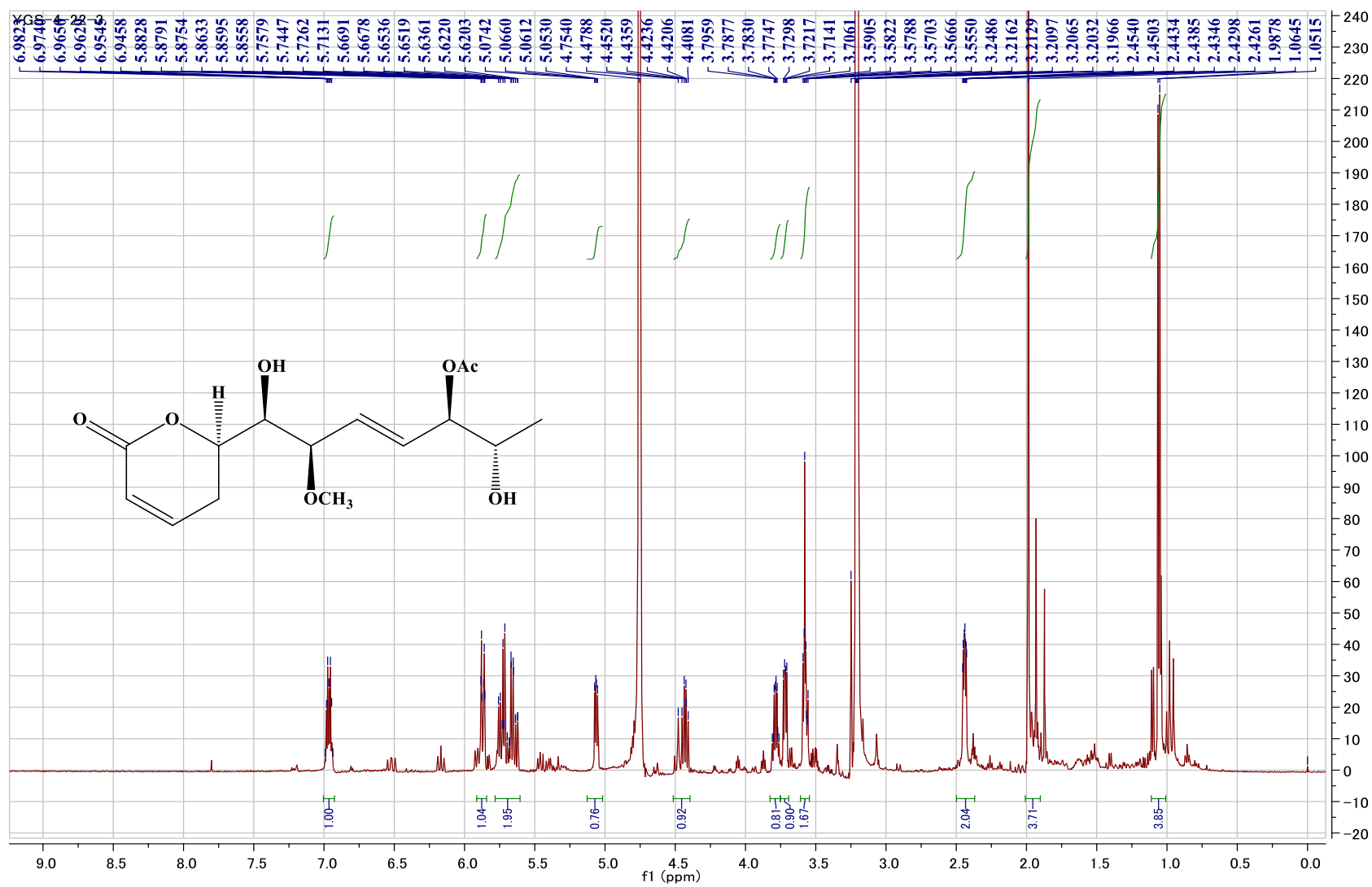


S15: LRCIMS of 3

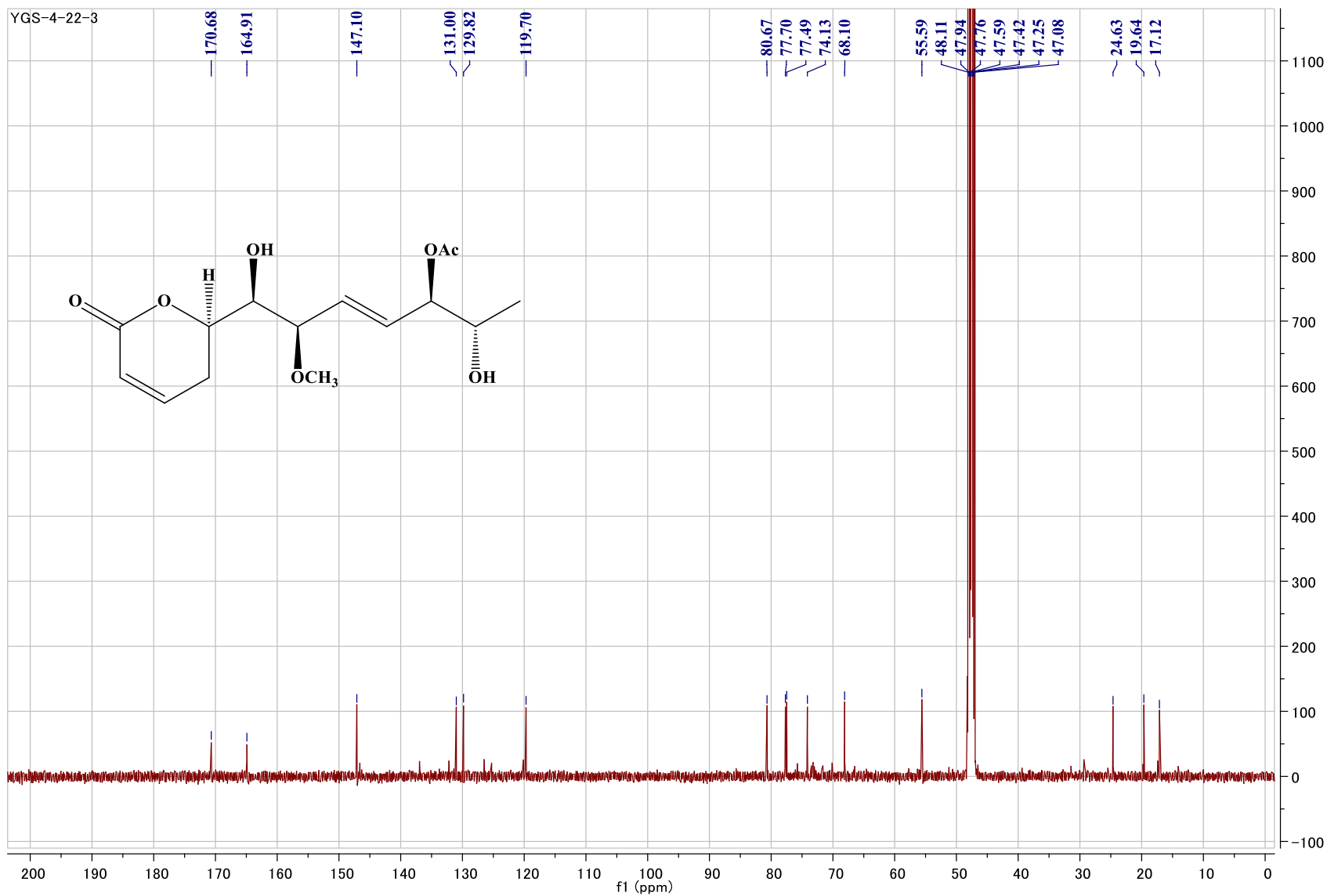
Data : Umeyama-CIHR.26-Feb-2018.003 Date : 26-Feb-2018 15:41
 Instrument : MStation
 Sample : YGS-4-22-3
 Note : MStation
 Inlet : Direct Ion Mode : CI+
 RT : 0.96 min Scan# : 24
 Elements : C 150/0, H 250/0, O 50/0
 Mass Tolerance : 5mmu
 Unsaturation (U.S.) : 0.0 – 15.0

	Observed m/z	Int%	Err [ppm / mmu]	U.S. Composition
1	315.1439	5.87	-1.5 / -0.5	4.5 C15 H23 O7

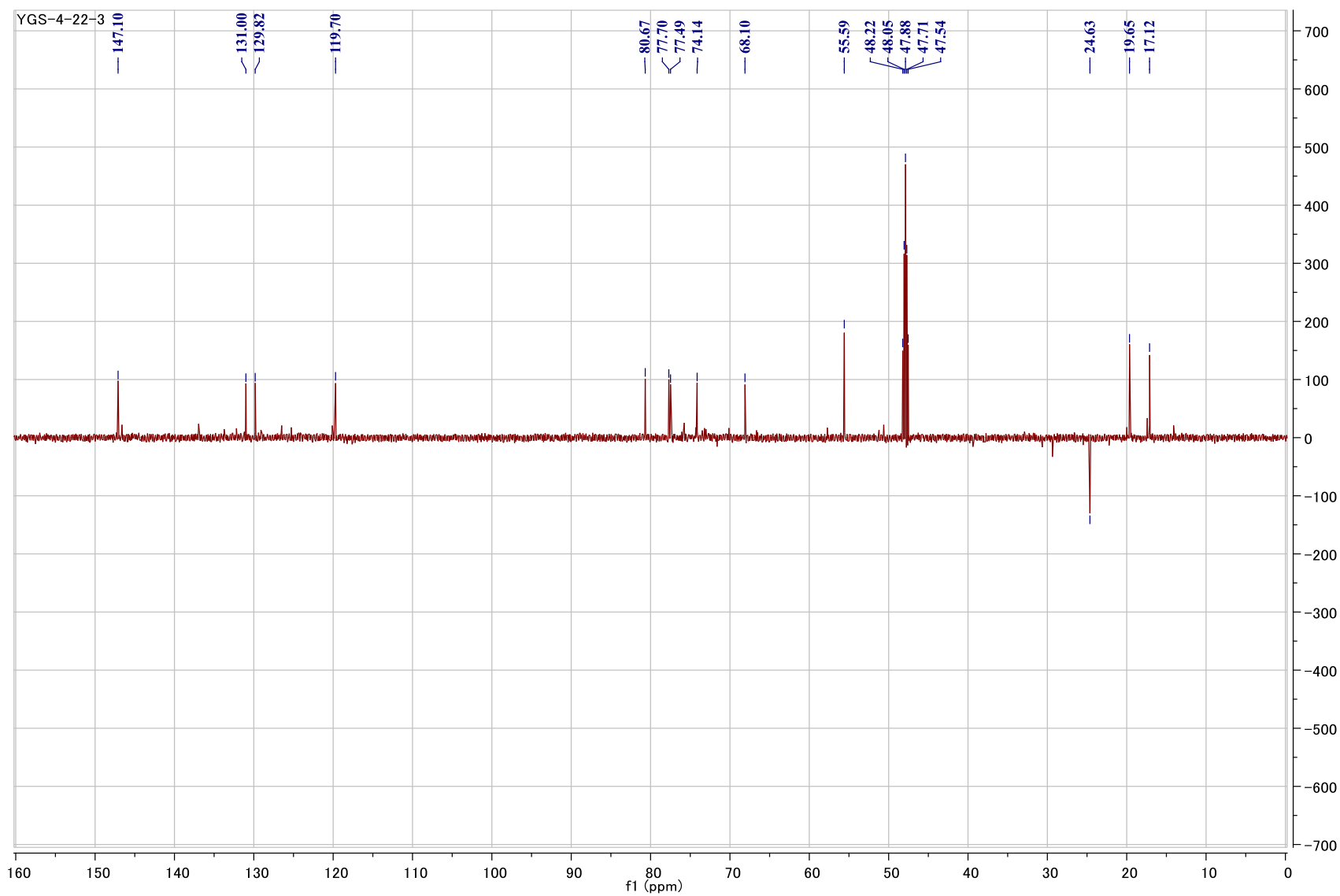
S16: HRCIMS of 3



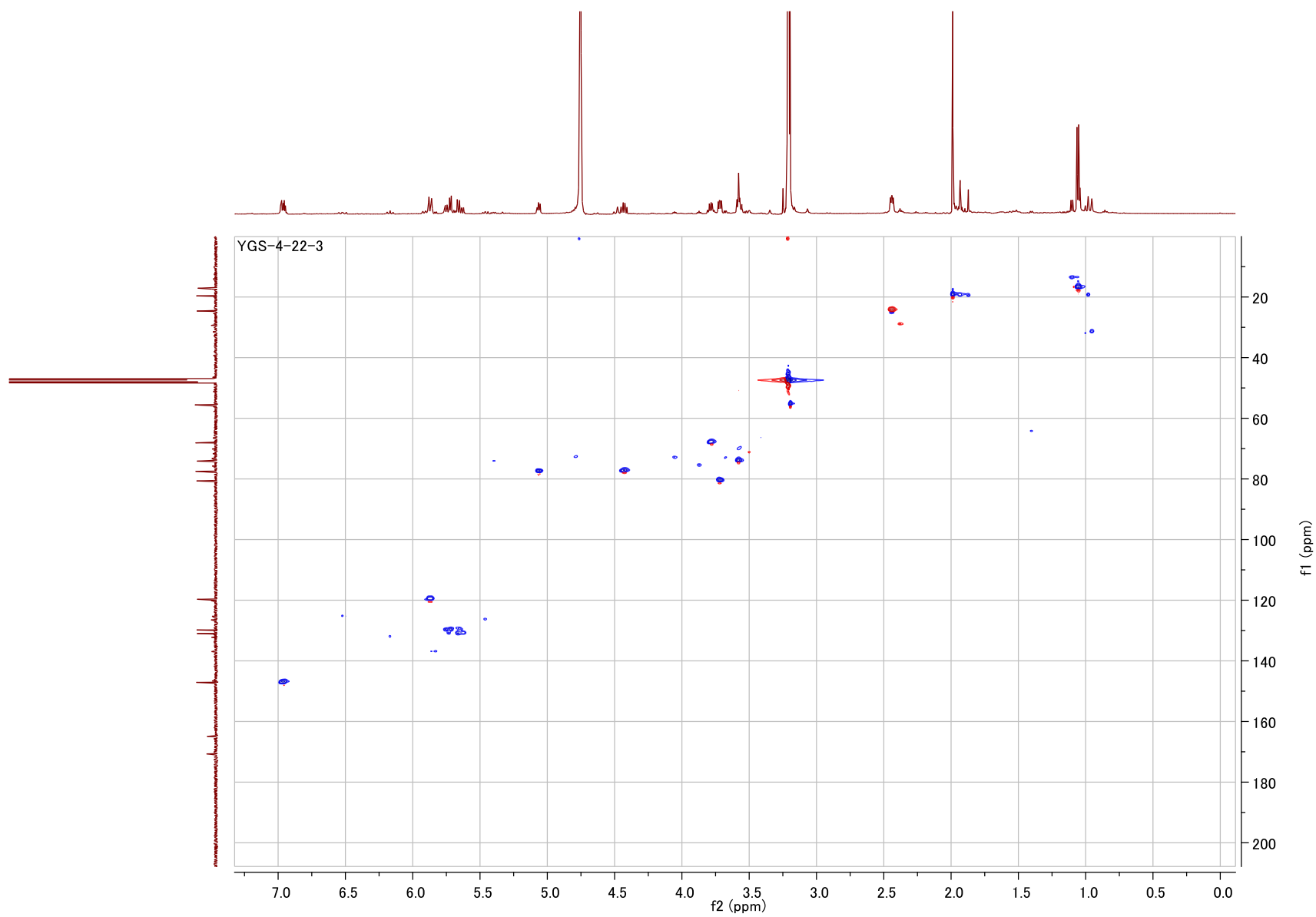
S17: ¹H NMR of 3



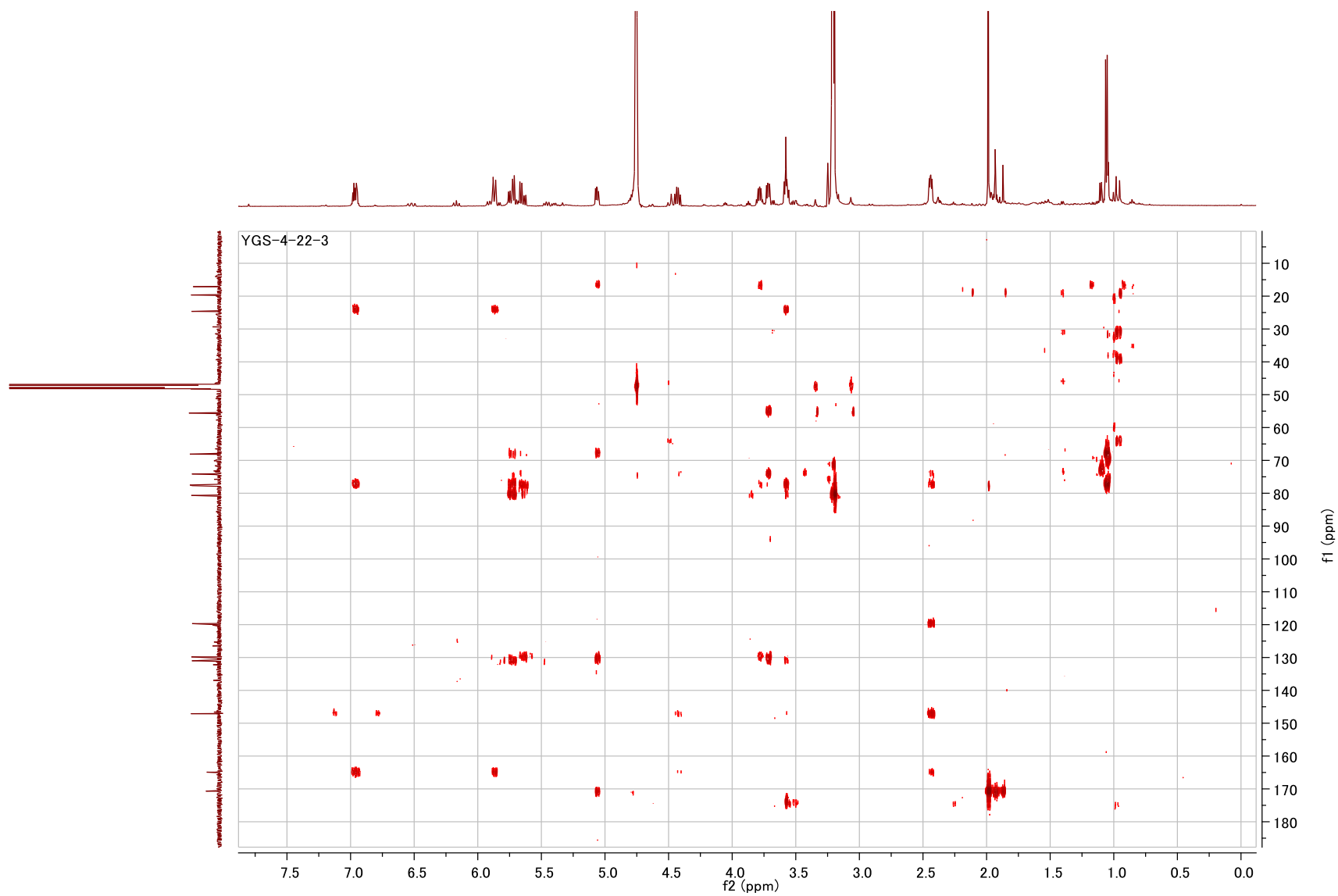
S18: ¹³C NMR of 3



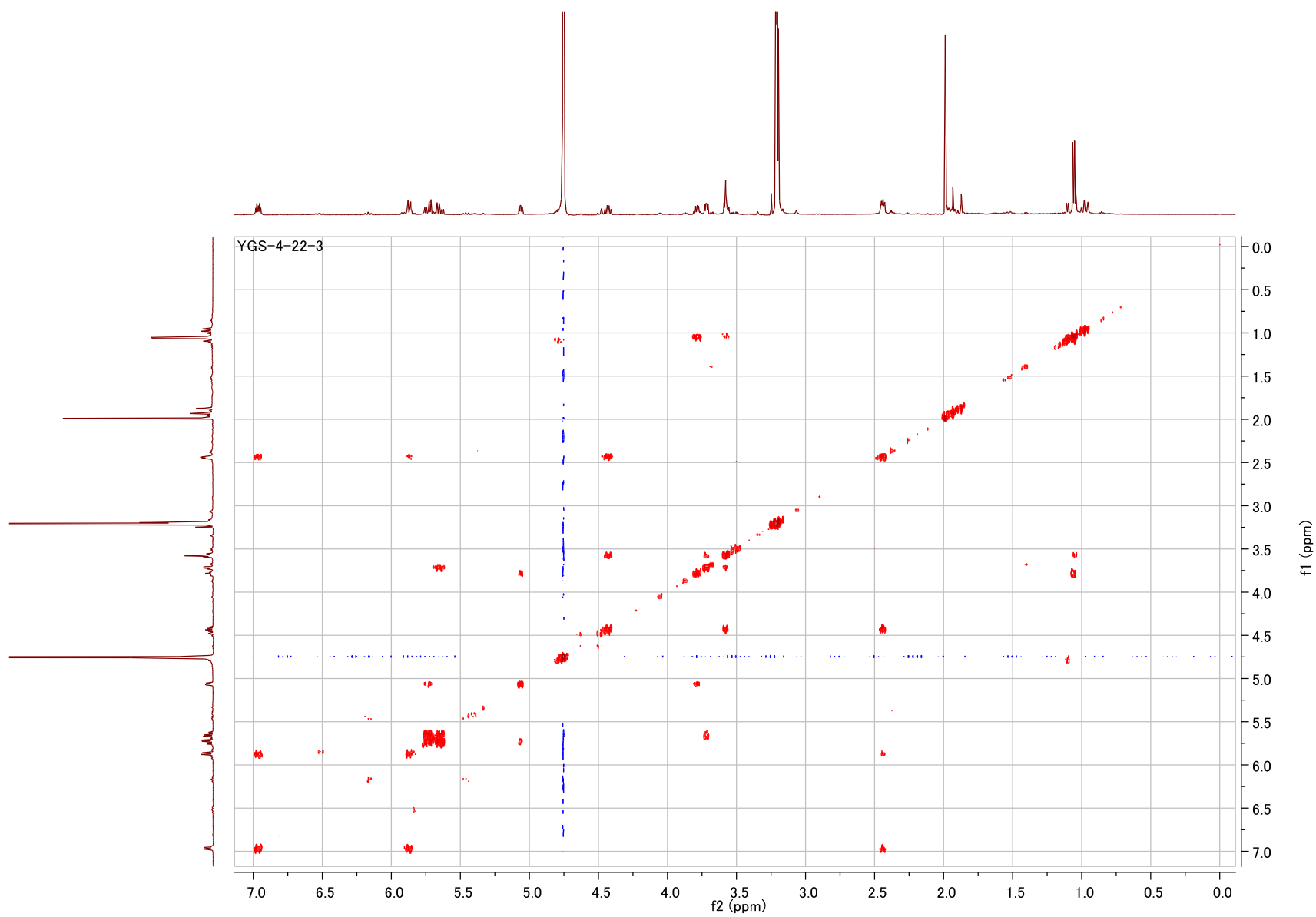
S19: DEPT-135 of 3



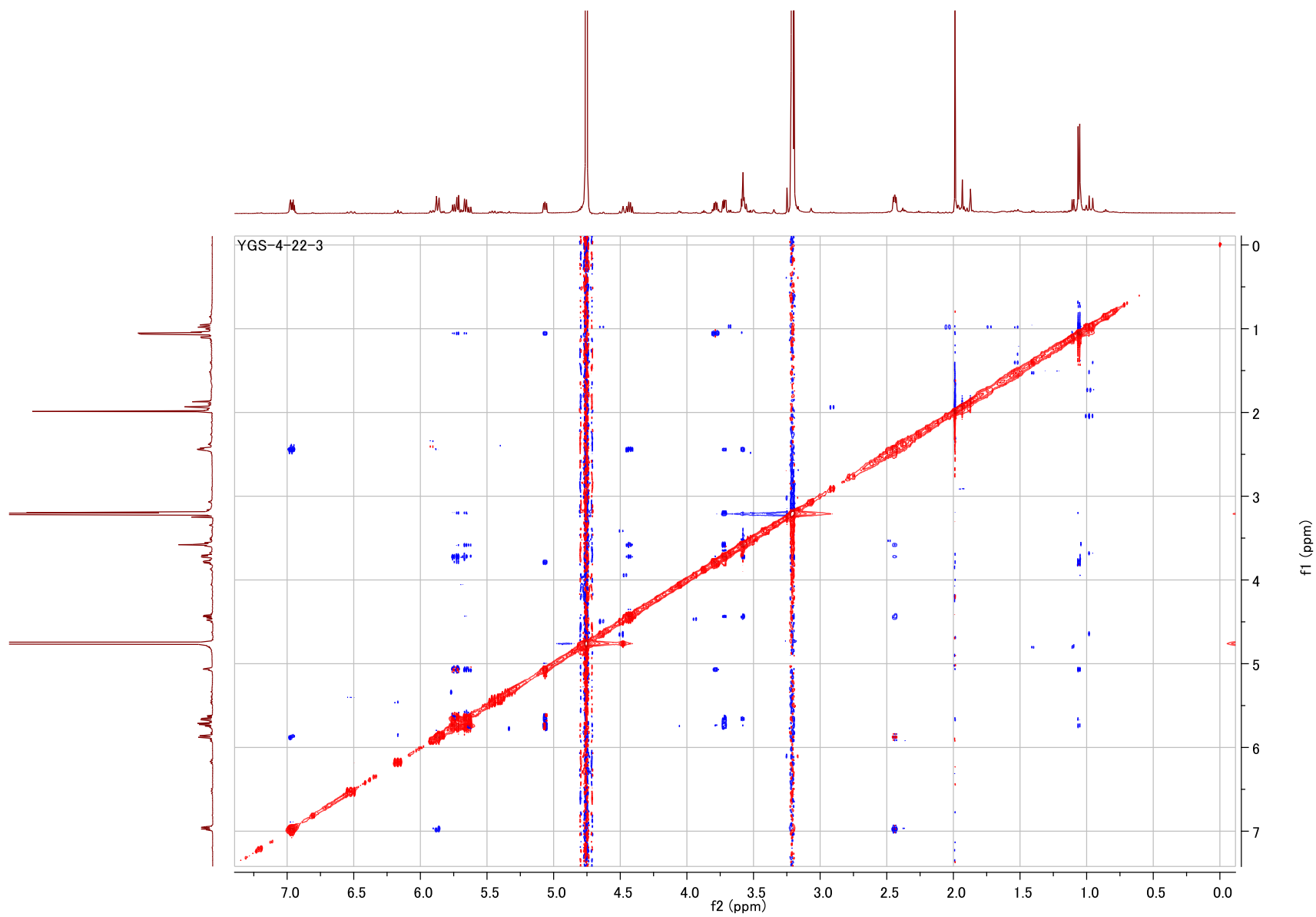
S20: HSQC of 3



S21: HMBC of 3

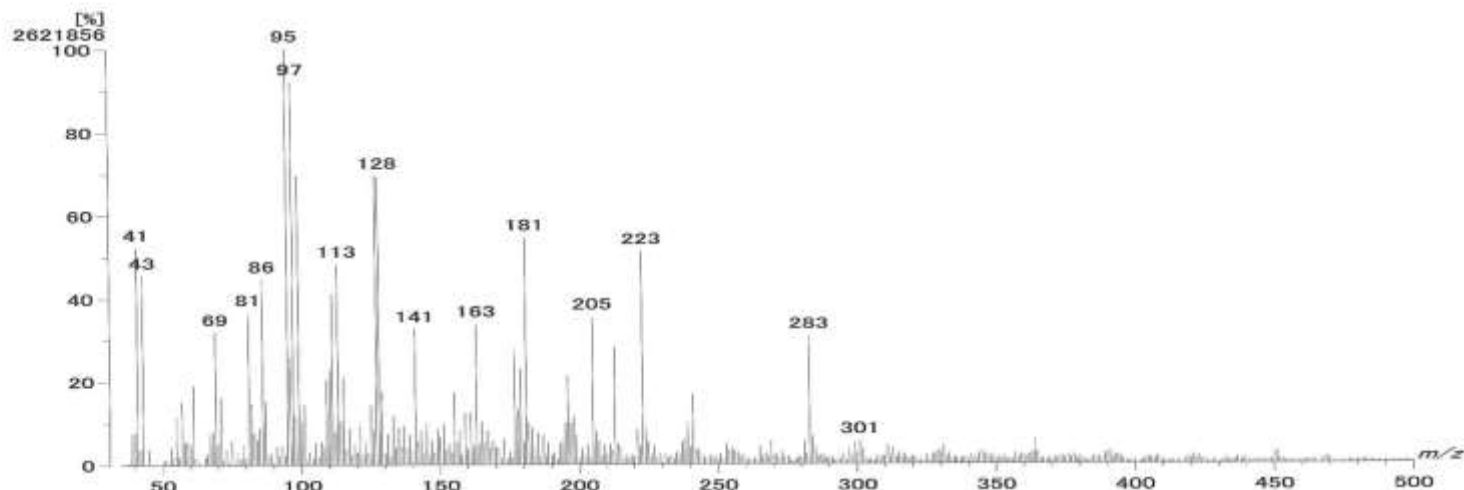


S22: ^1H ^1H COSY of **3**



S23: NOESY of 3

[Mass Spectrum]
 Data : Umeyama-GI26-Feb-2018.001 Date : 26-Feb-2018 13:33
 Sample : YGS-4-22-2(OH4)
 Note : MStation
 Inlet : Direct Ion Mode : CI+
 Spectrum Type : Normal Ion [MF-Linear]
 RT : 1.56 min Scan# : 58-k(12)[k=1.0]
 BP : m/z 95 Int. : 250.04 (2621856)
 Output m/z range : 35 to 500 Cut Level : 0.00 %

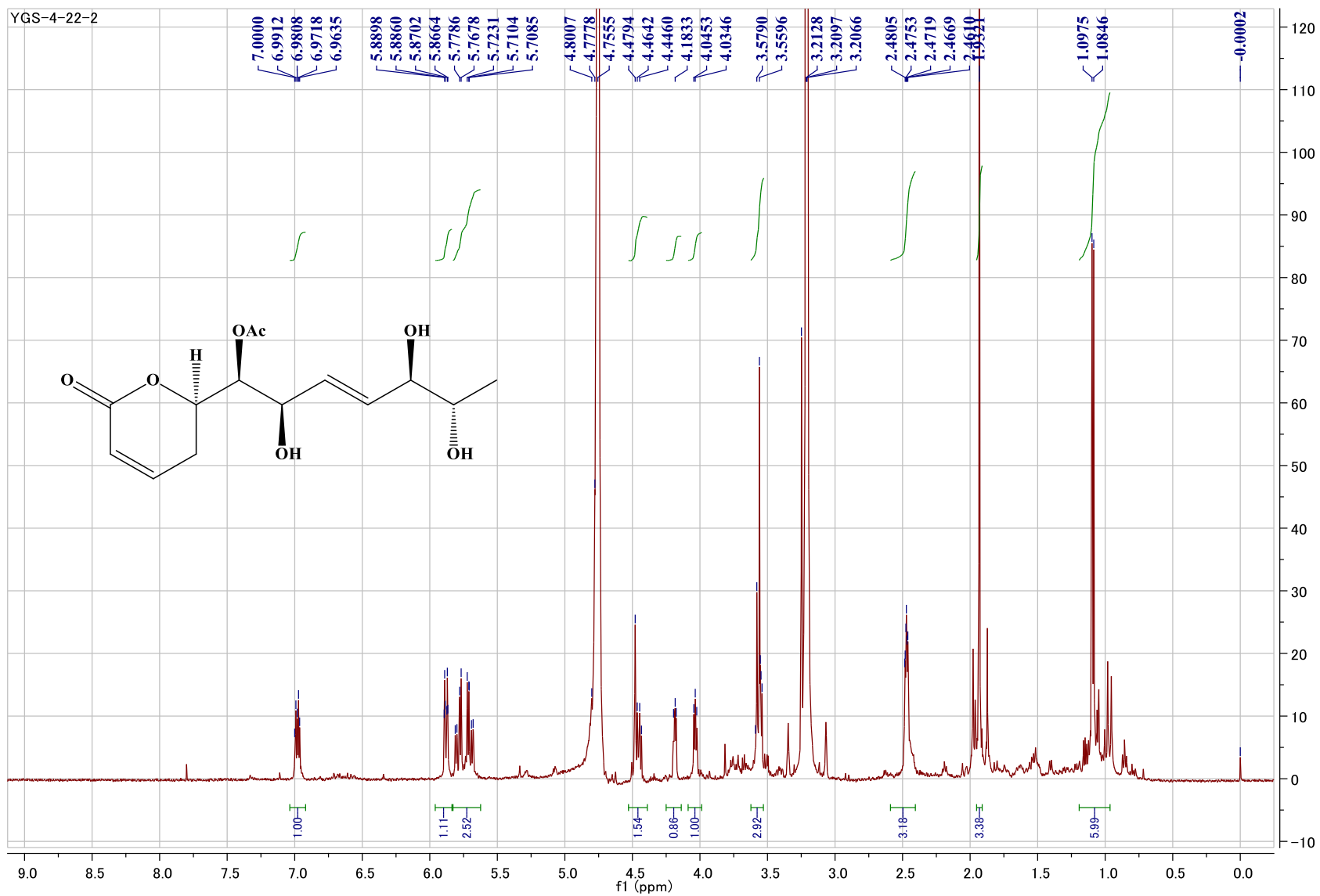


S24: LRCIMS of 4

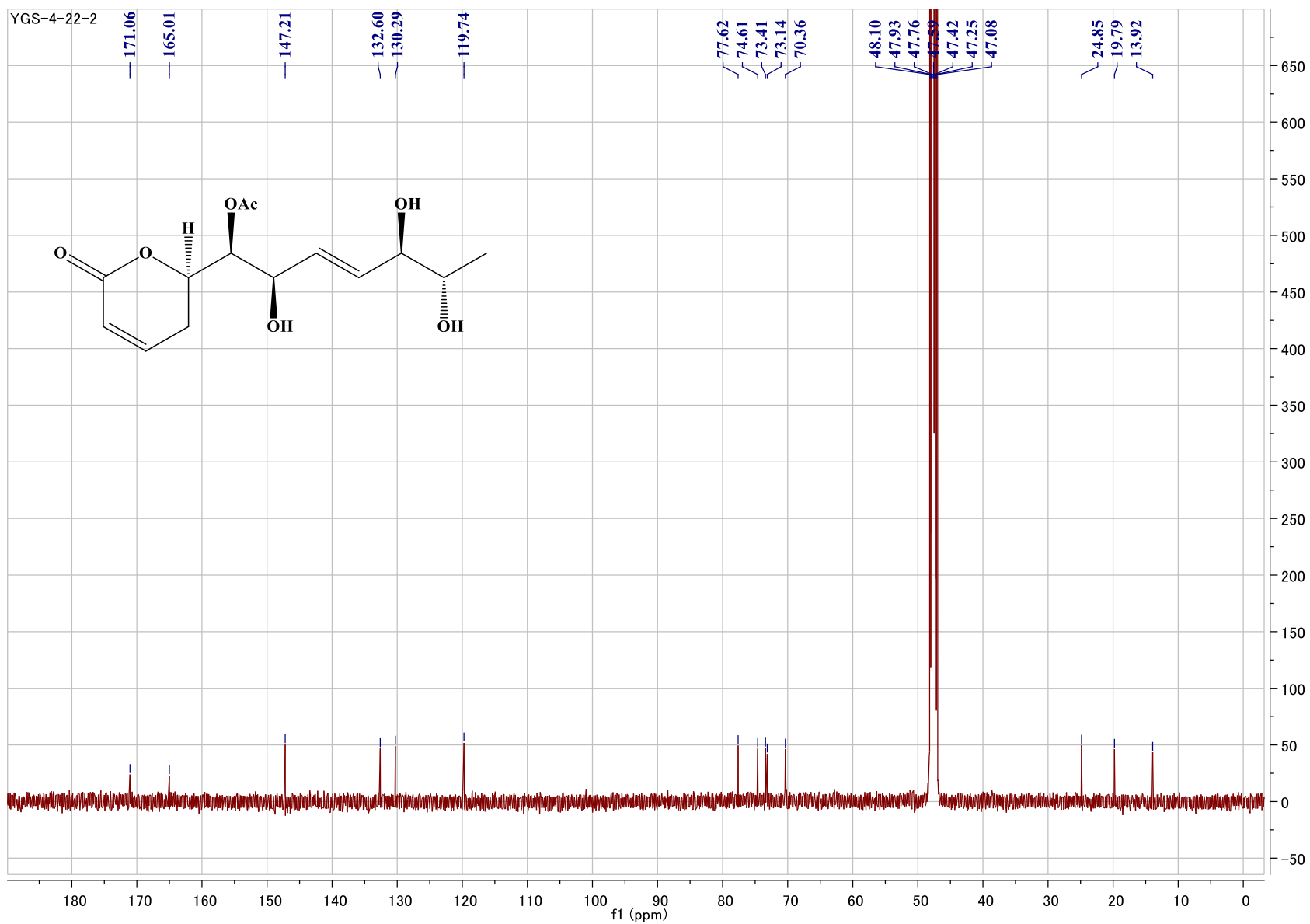
Data : Umeyama-CIHR.26-Feb-2018.002 Date : 26-Feb-2018 15:31
 Instrument : MStation
 Sample : YGS-4-22-2
 Note : MStation
 Inlet : Direct Ion Mode : CI+
 RT : 1.00 min Scan# : 25
 Elements : C 150/0, H 250/0, O 50/0
 Mass Tolerance : 5mmu
 Unsaturation (U.S.) : 0.0 - 15.0

	Observed m/z	Int %	Err [ppm / mmu]	U.S.	Composition
1	301.1265	2.59	+12.1 / +3.6	13.5	C21 H17 O2
2			-7.4 / -2.2	4.5	C14 H21 O7

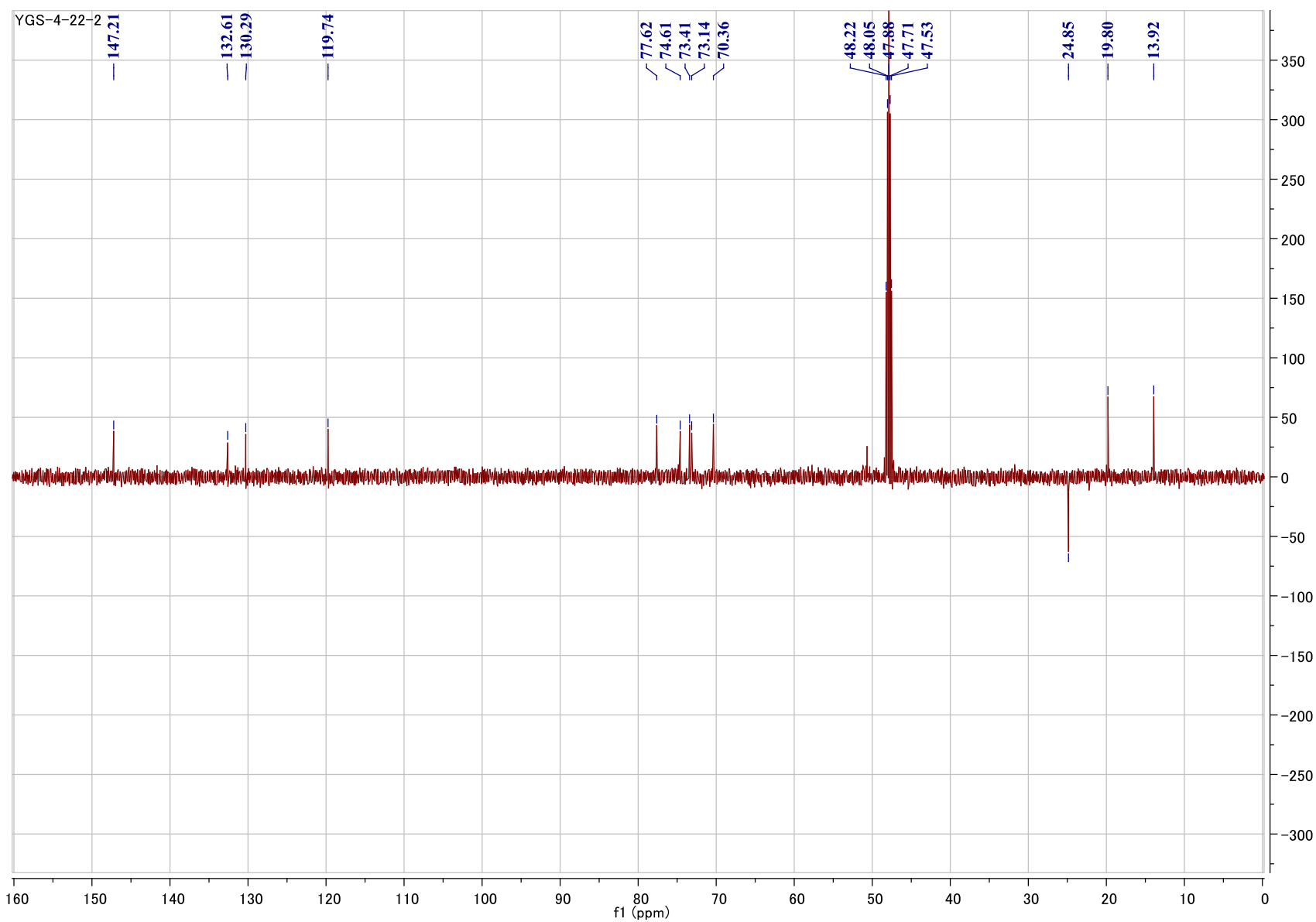
S25: HRCIMS of 4



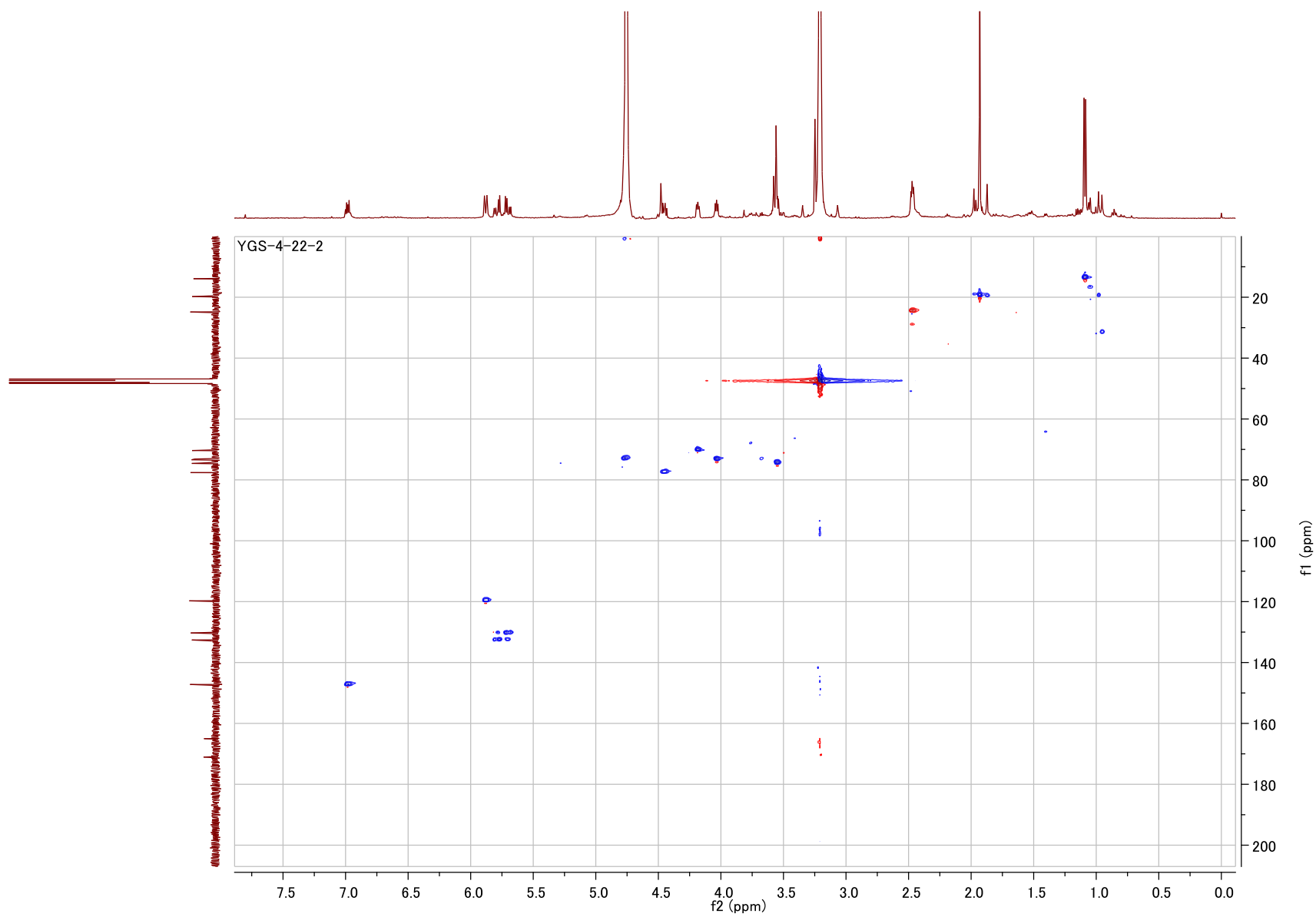
S26: ¹H NMR of 4



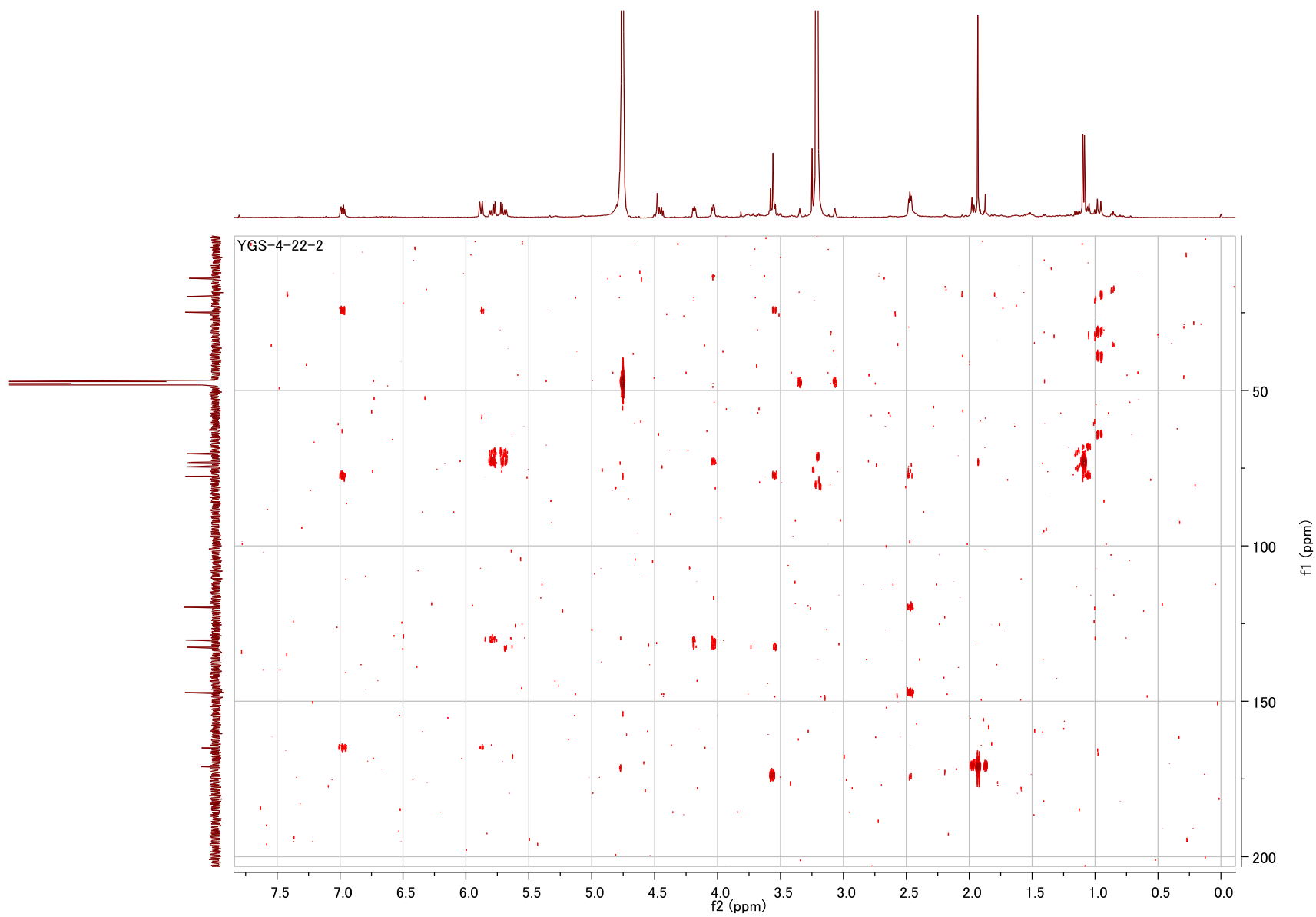
S27: ^{13}C NMR of 4



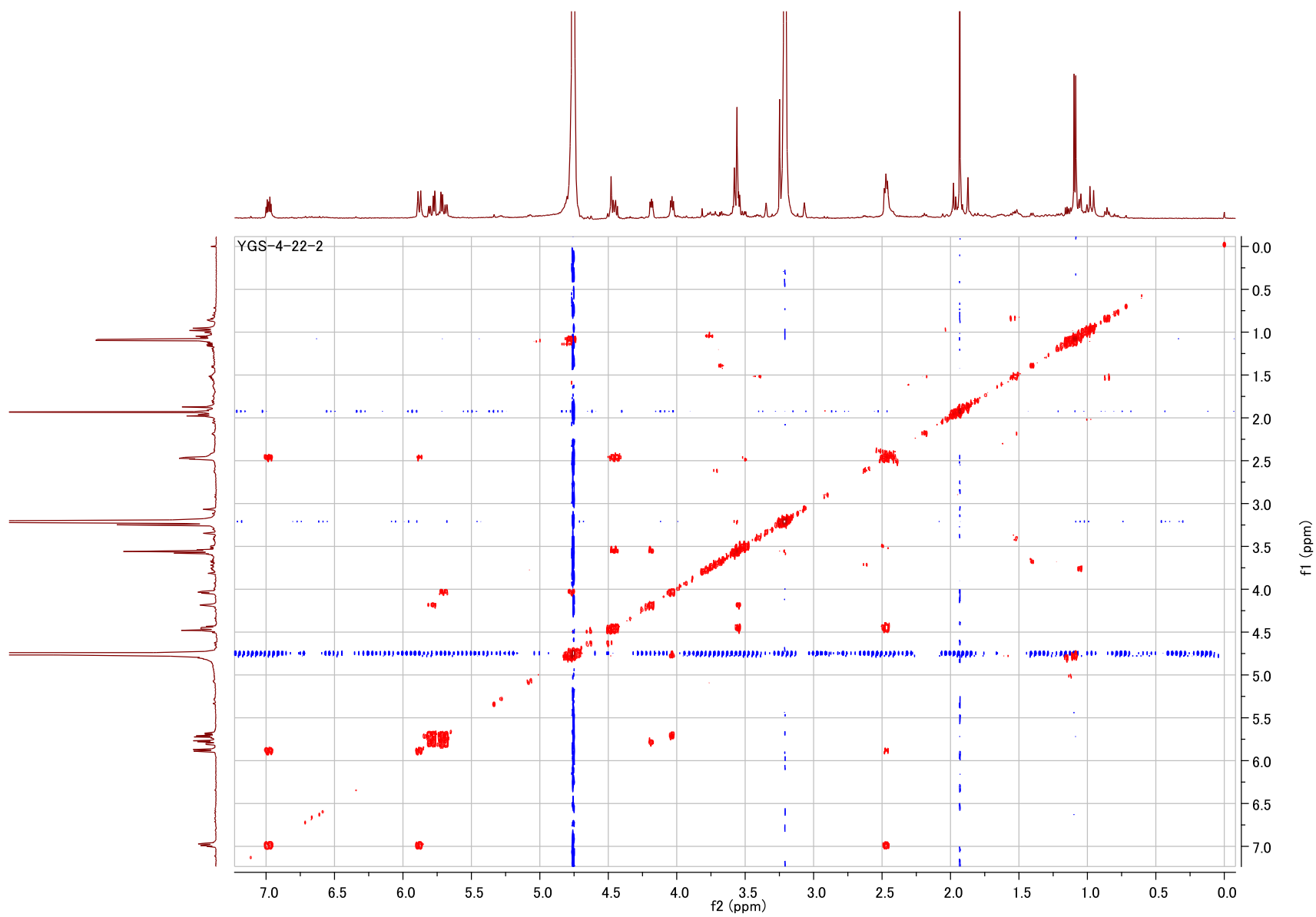
S28: DEPT-135 of 4



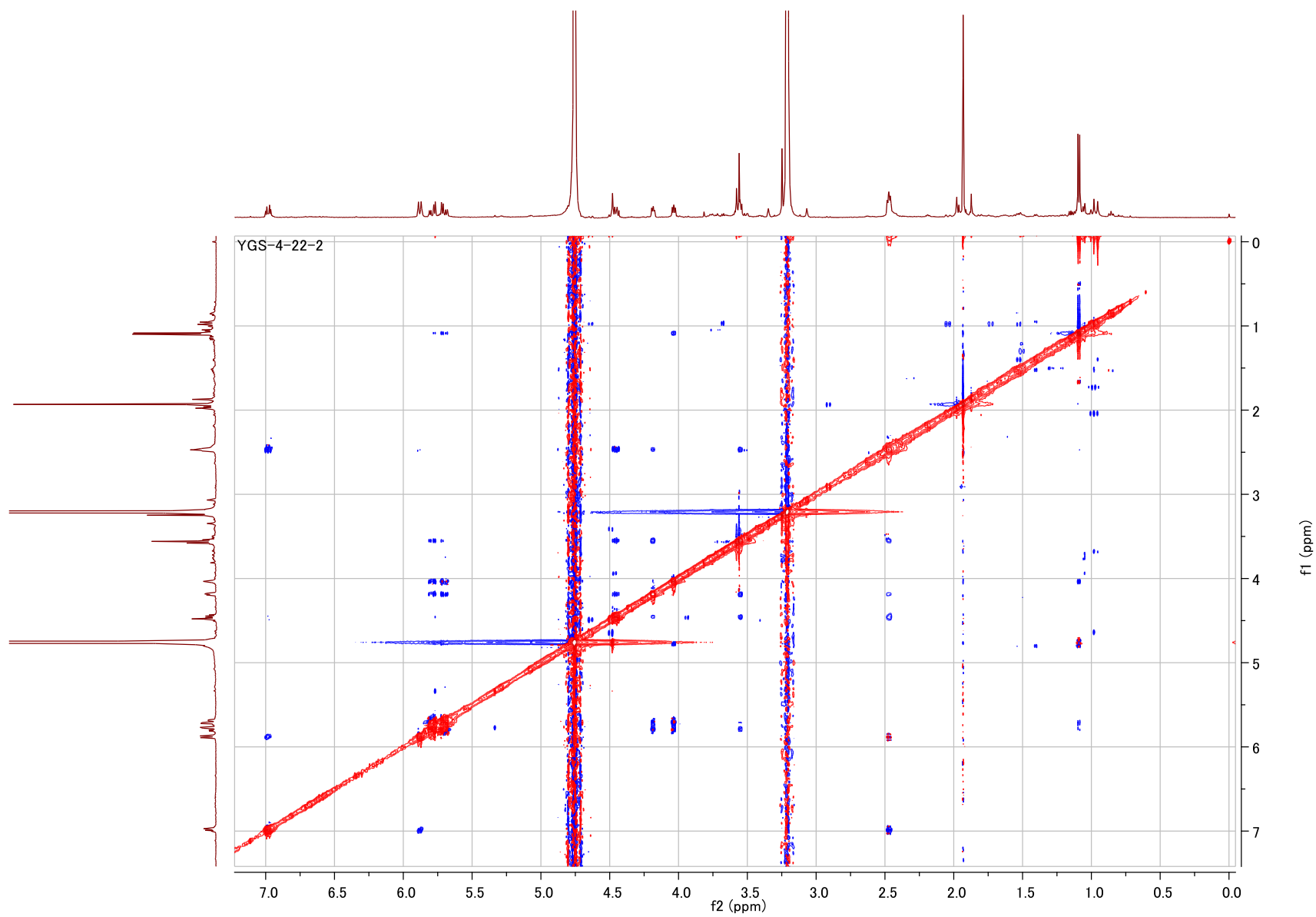
S29: HSQC of 4



S30: HMBC of 4

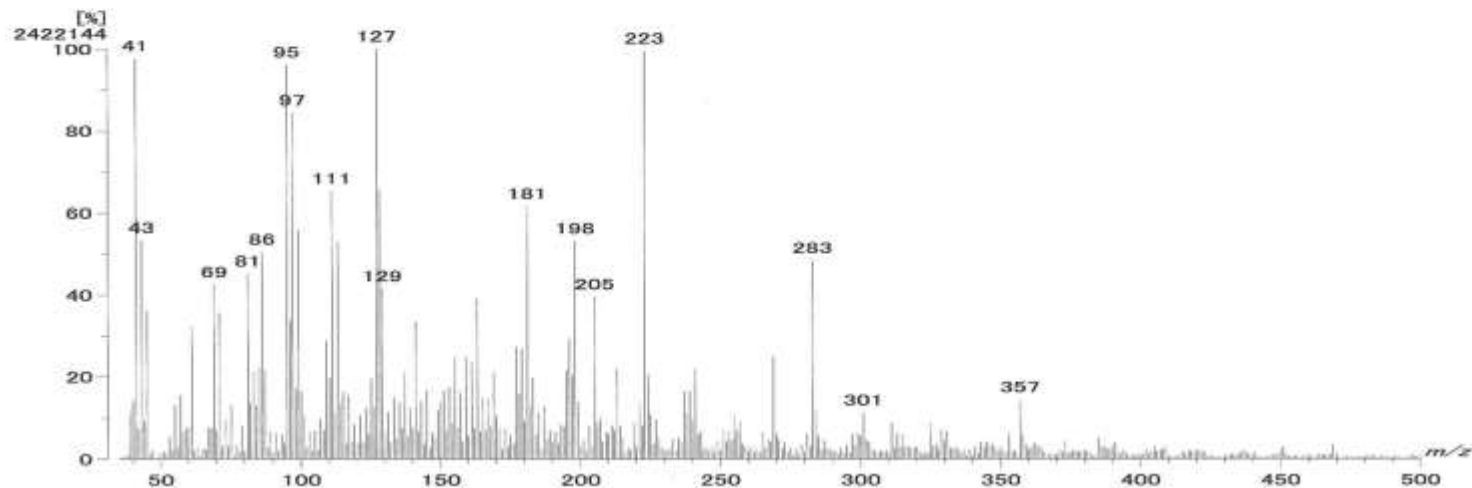


S31: ^1H ^1H COSY of 4



S32: NOESY of 4

[Mass Spectrum]
 Data : Umeyama-CL07-Mar-2018.001 Date : 07-Mar-2018 11:41
 Sample : YGS-4-22-1(CH4)
 Note : MStation
 Inlet : Direct Ion Mode : CI+
 Spectrum Type : Normal Ion [MF-Linear]
 RT : 1.45 min Scan# : 54
 BP : m/z 127 Int : 230.99 (2422144)
 Output m/z range : 35 to 500 Cut Level : 0.00 %

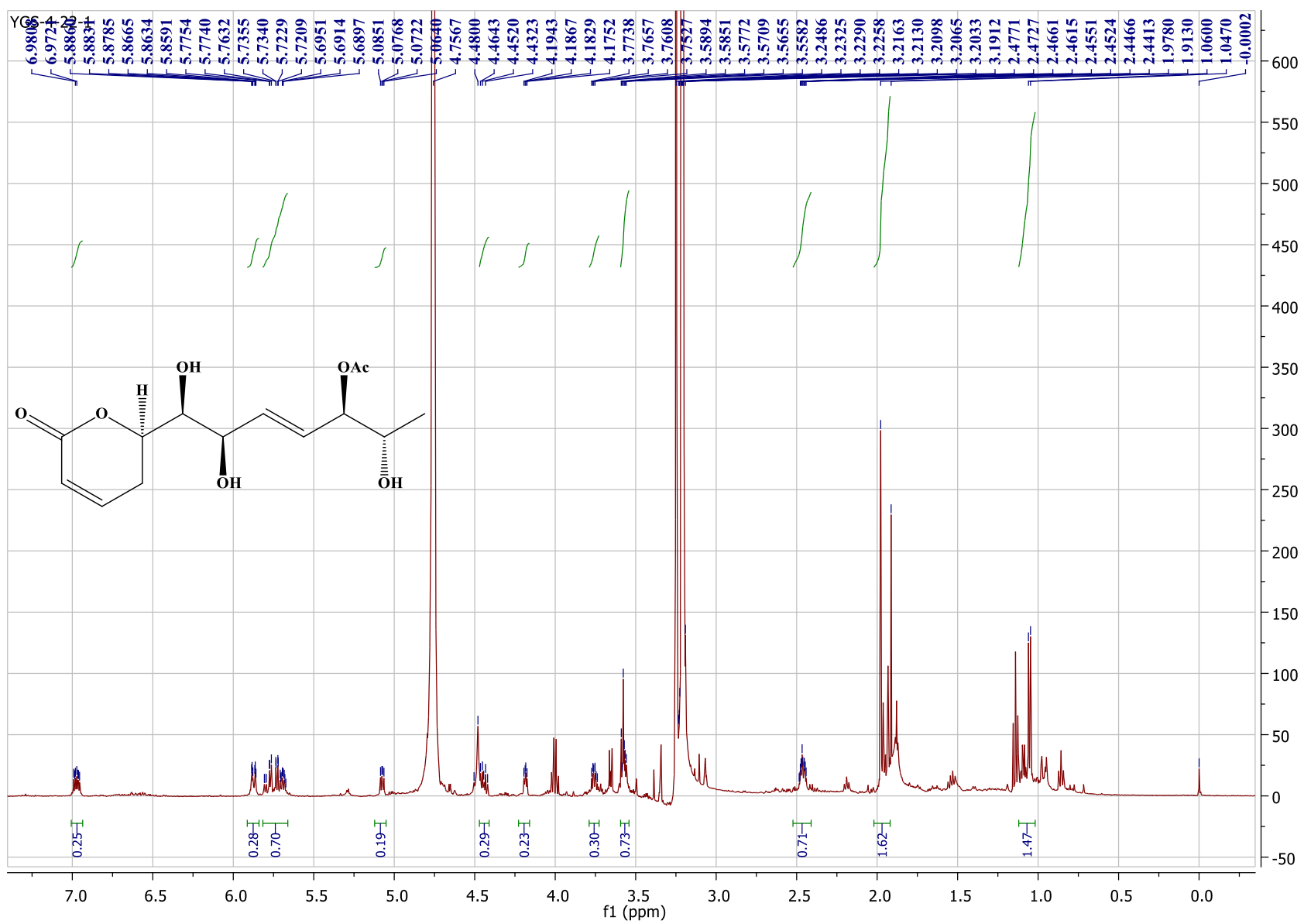


S33: LRCIMS of 5

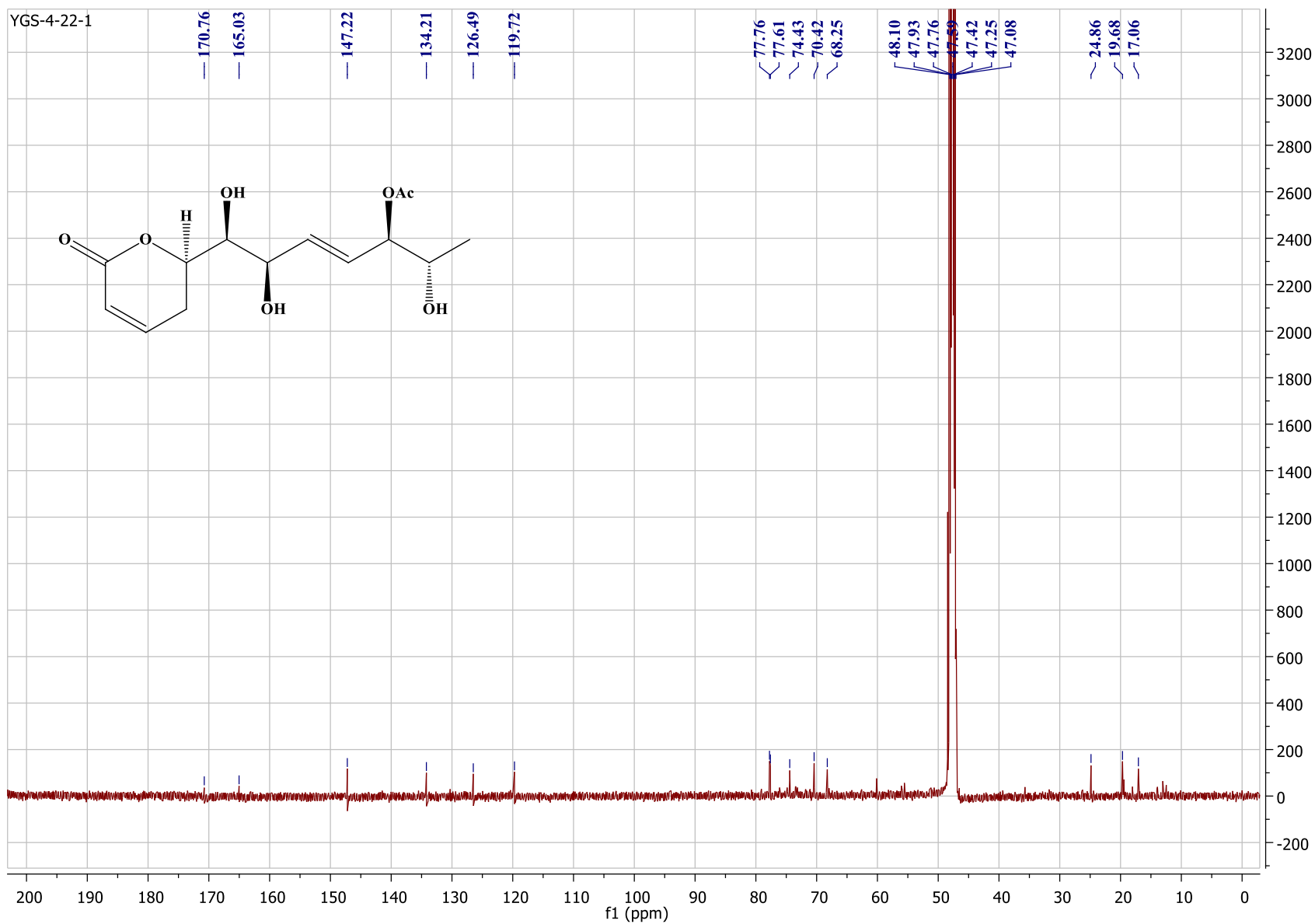
Data : Umeyama-CIHR.07-Mar-2018.002 Date : 07-Mar-2018 13:07
 Instrument : MStation
 Sample : YGS-4-22-1
 Note : MStation
 Inlet : Direct Ion Mode : CI+
 RT : 1.54 min Scan# : 38
 Elements : C 150/0, H 250/0, O 50/0
 Mass Tolerance : 5mmu
 Unsaturation (U.S.) : 0.0 - 15.0

	Observed m/z	Int%	Err [ppm / mmu]	U.S.	Composition
1	301.1269	13.63	+13.4 / +4.0	13.5	C21 H17 O2
2			-6.1 / -1.8	4.5	C14 H21 O7

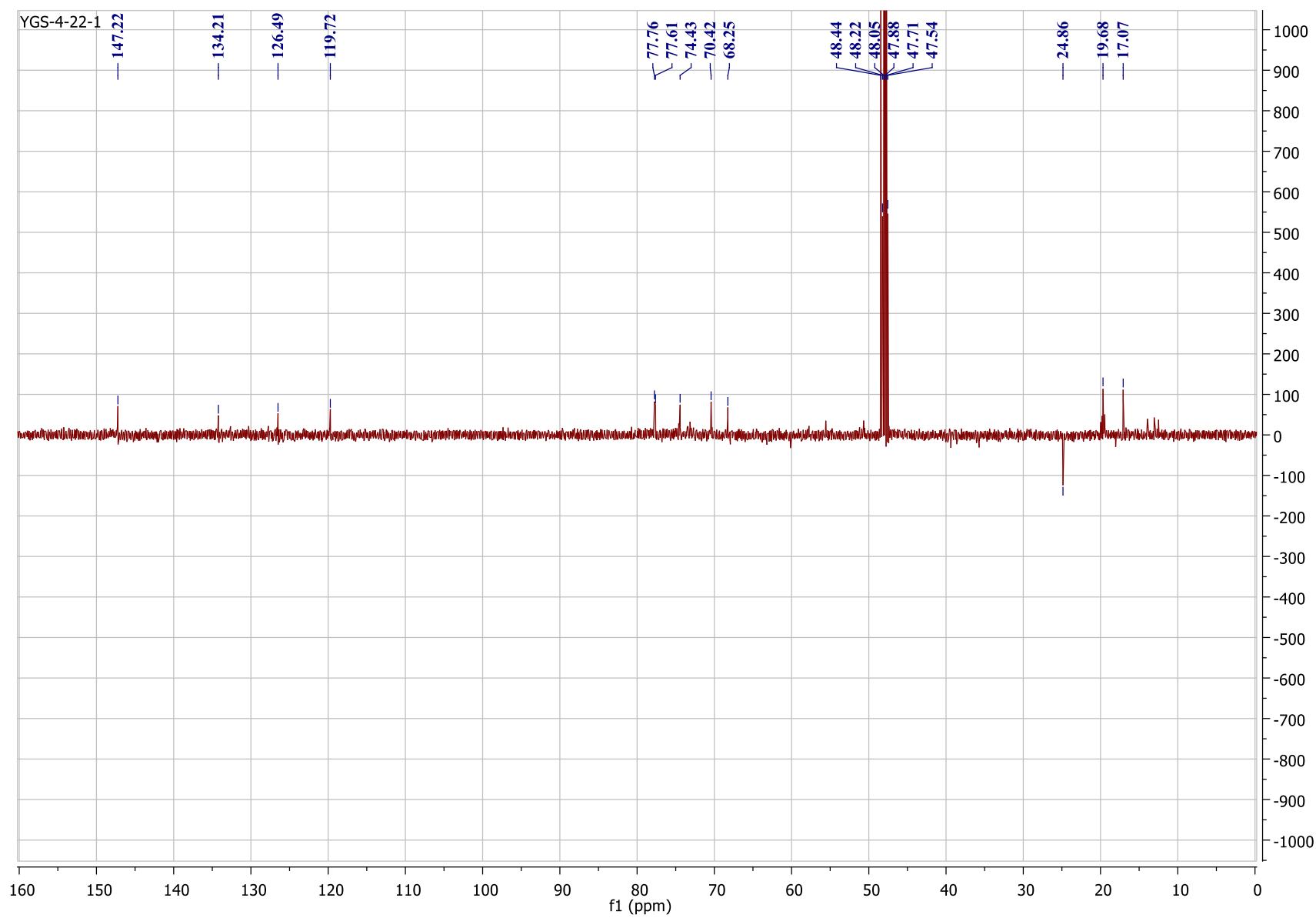
S34: HRCIMS of 5



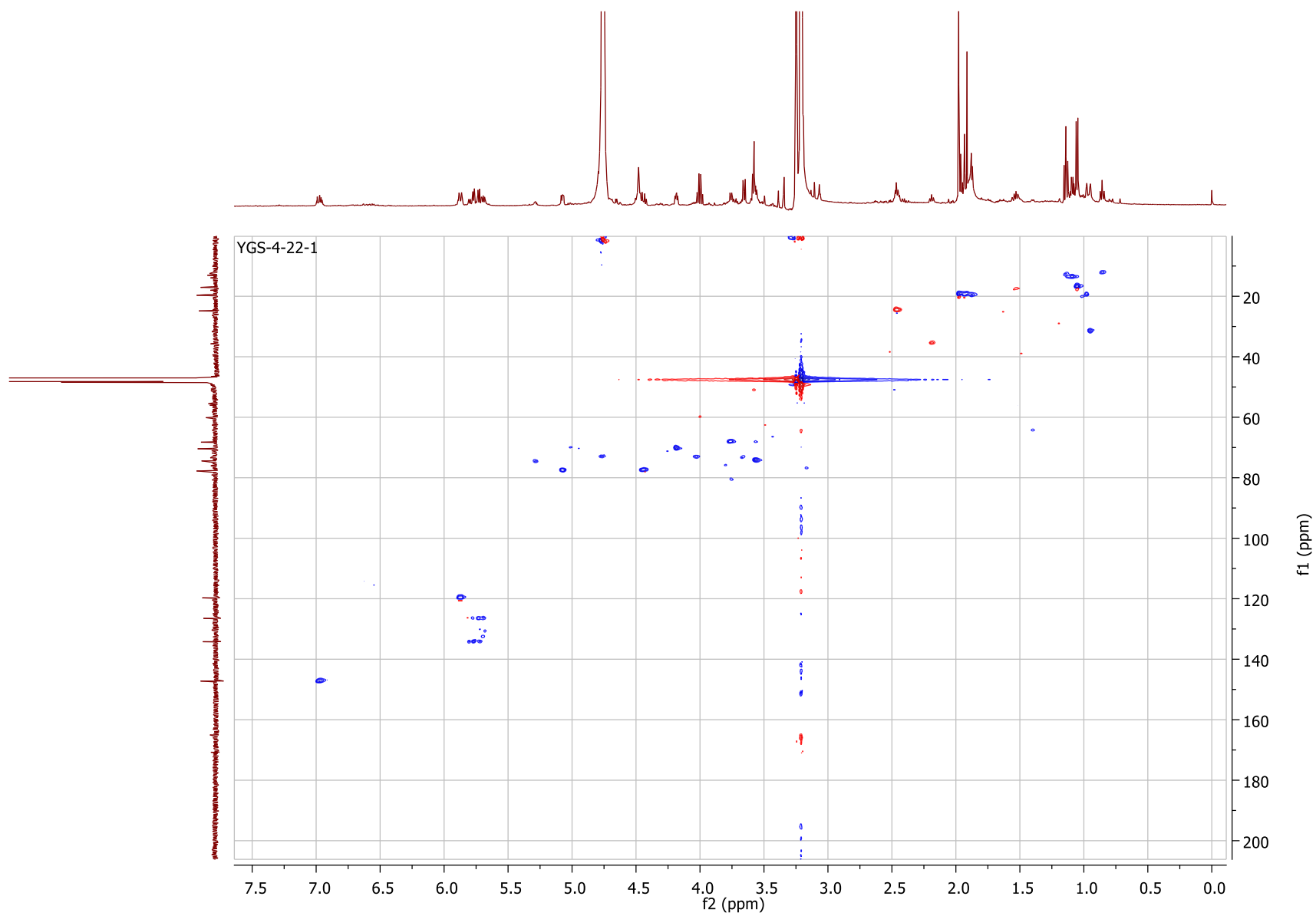
S35: ¹H NMR of 5



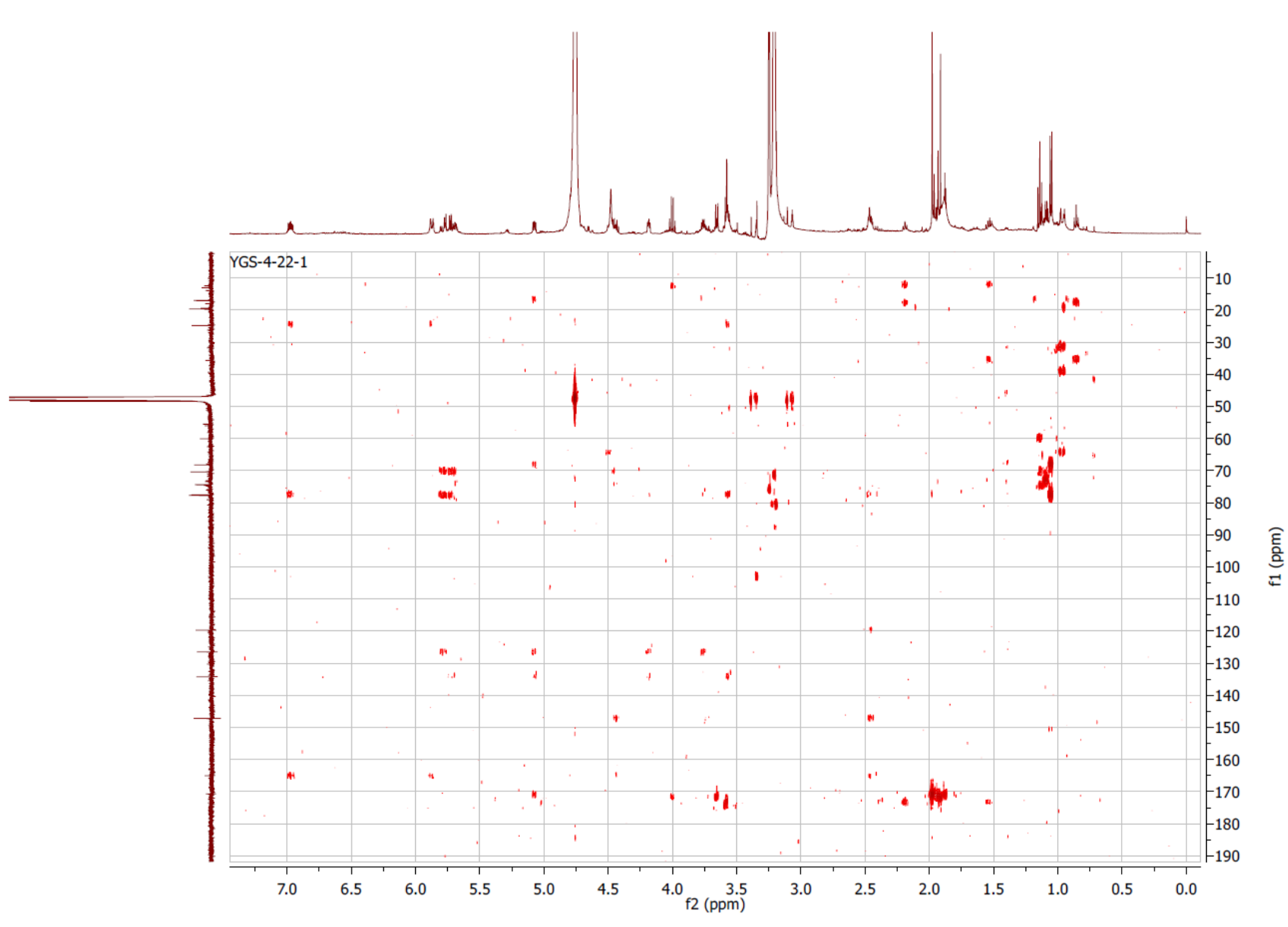
S36: ¹³C NMR of 5



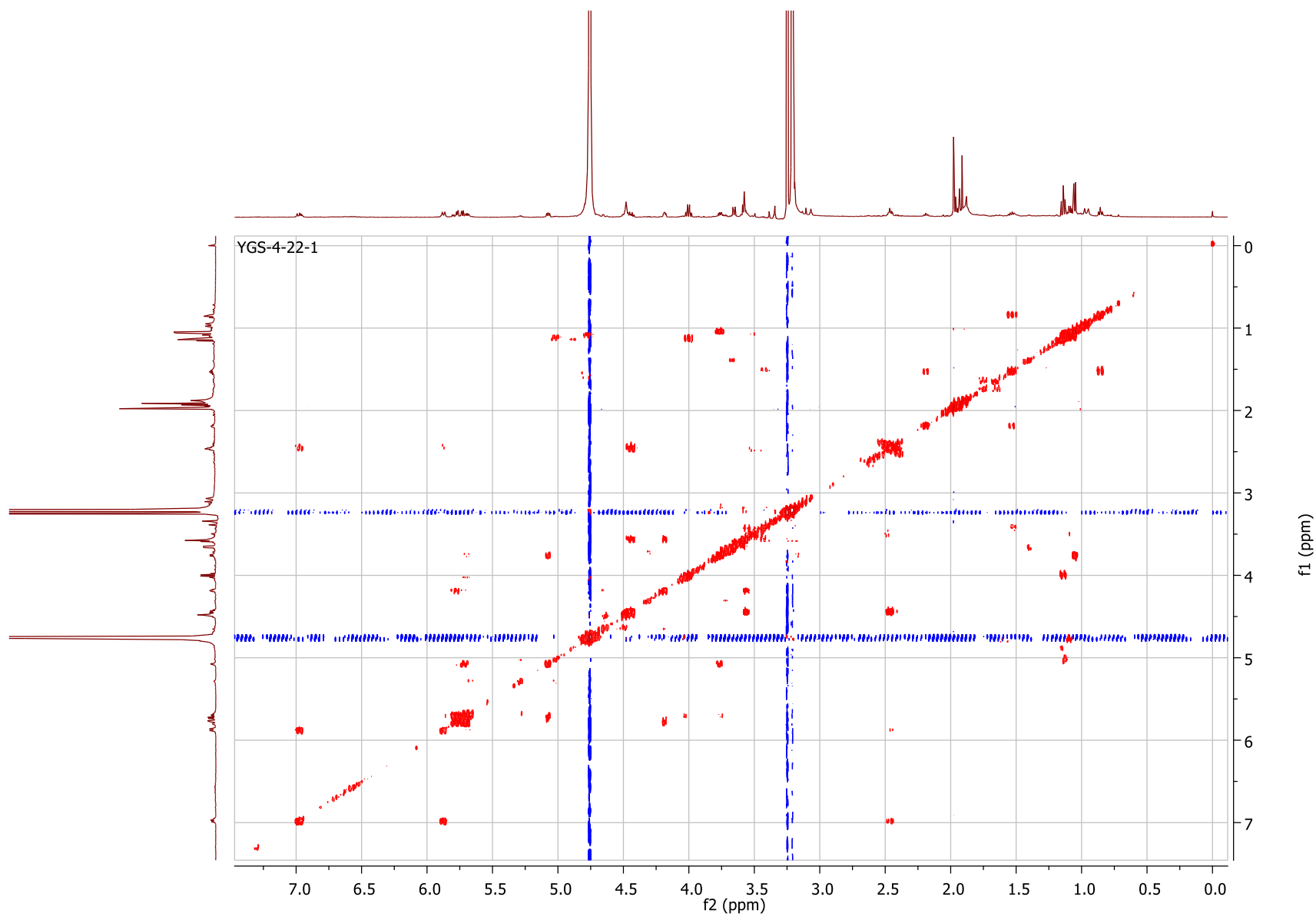
S37: DEPT-135 of 5



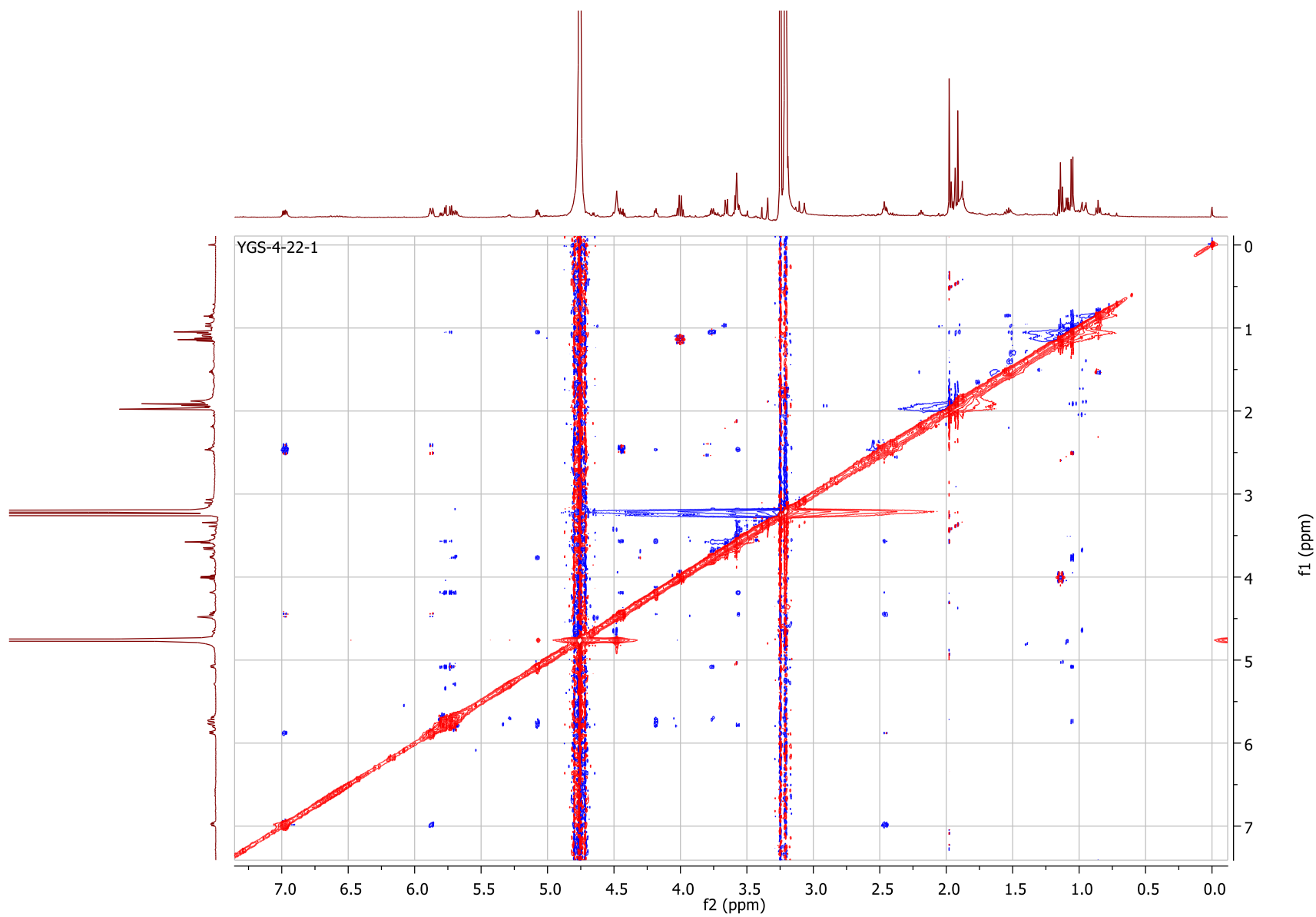
S38: HSQC of 5



S39: HMBC of 5

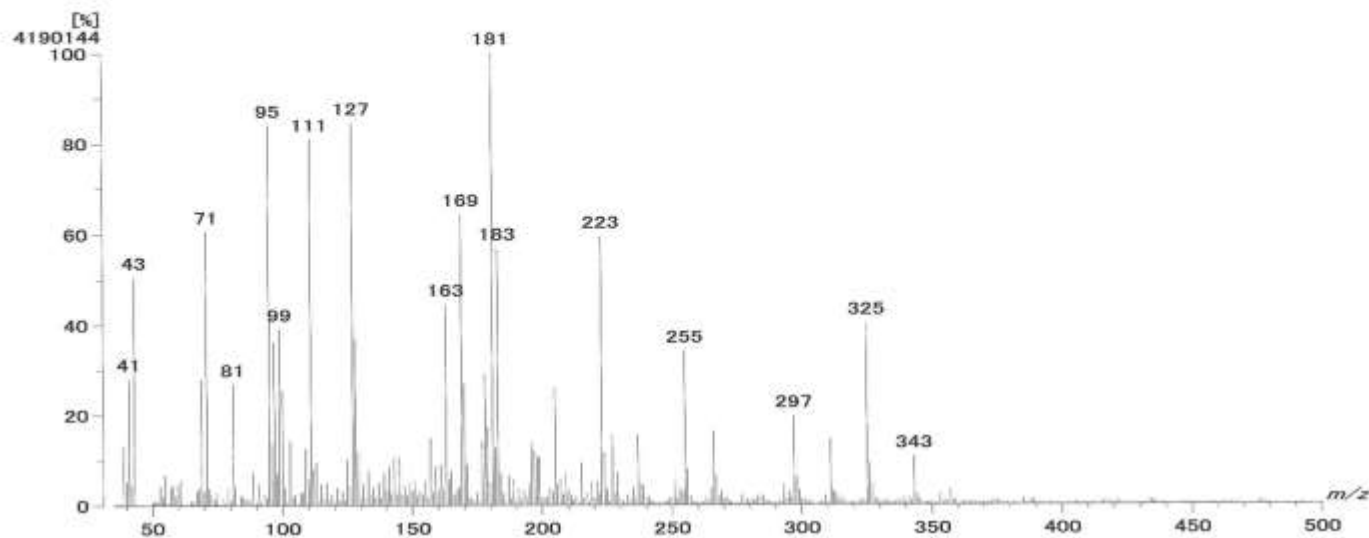


S40: ^1H ^1H COSY of 5



S41: NOESY of 5

[Mass Spectrum]
 Data : Umeyama-CL26-Feb-2018.003 Date : 26-Feb-2018 13:56
 Sample : YGS-4-22-9(CH4)
 Note : MStation
 Inlet : Direct Ion Mode : CI+
 Spectrum Type : Normal Ion [MF-Linear]
 RT : 1.20 min Scan# : 45-k(13)[k=1.0]
 BP : m/z 181 Int. : 399.60 (4190144)
 Output m/z range : 35 to 500 Cut Level : 0.00 %

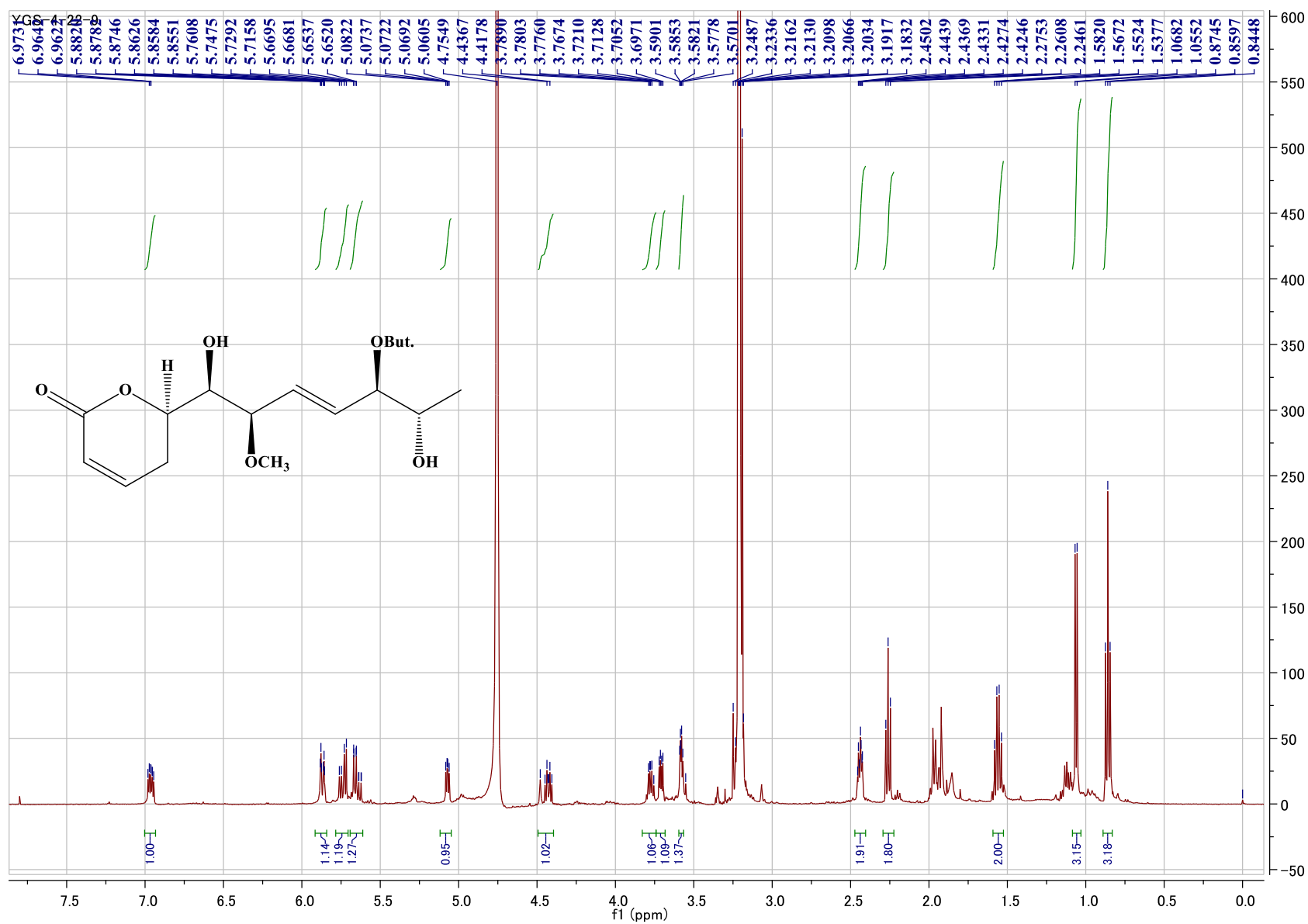


S42: LRCIMS of 6

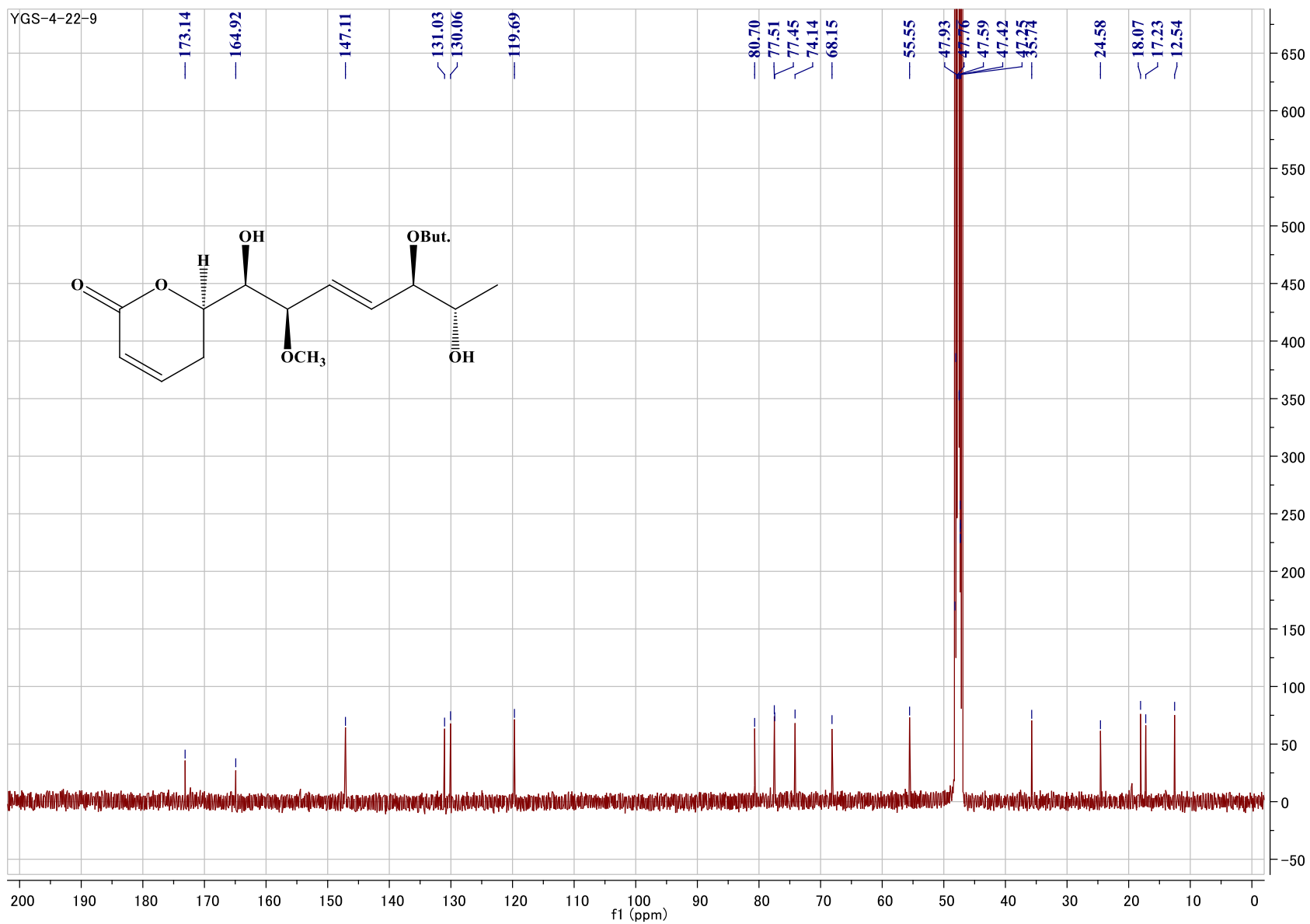
Data : Umeyama-CIHR.26-Feb-2018.004 Date : 26-Feb-2018 15:47
 Instrument : MStation
 Sample : YGS-4-22-9
 Note : MStation
 Inlet : Direct Ion Mode : CI+
 RT : 1.04 min Scan# : 27
 Elements : C 150/0, H 250/0, O 50/0
 Mass Tolerance : 5mmu
 Unsaturation (U.S.) : 0.0 - 15.0

	Observed m/z	Int%	Err [ppm / mmu]	U.S. Composition
1	343.1755	12.81	-0.5 / -0.2	4.5 C17 H27 O7

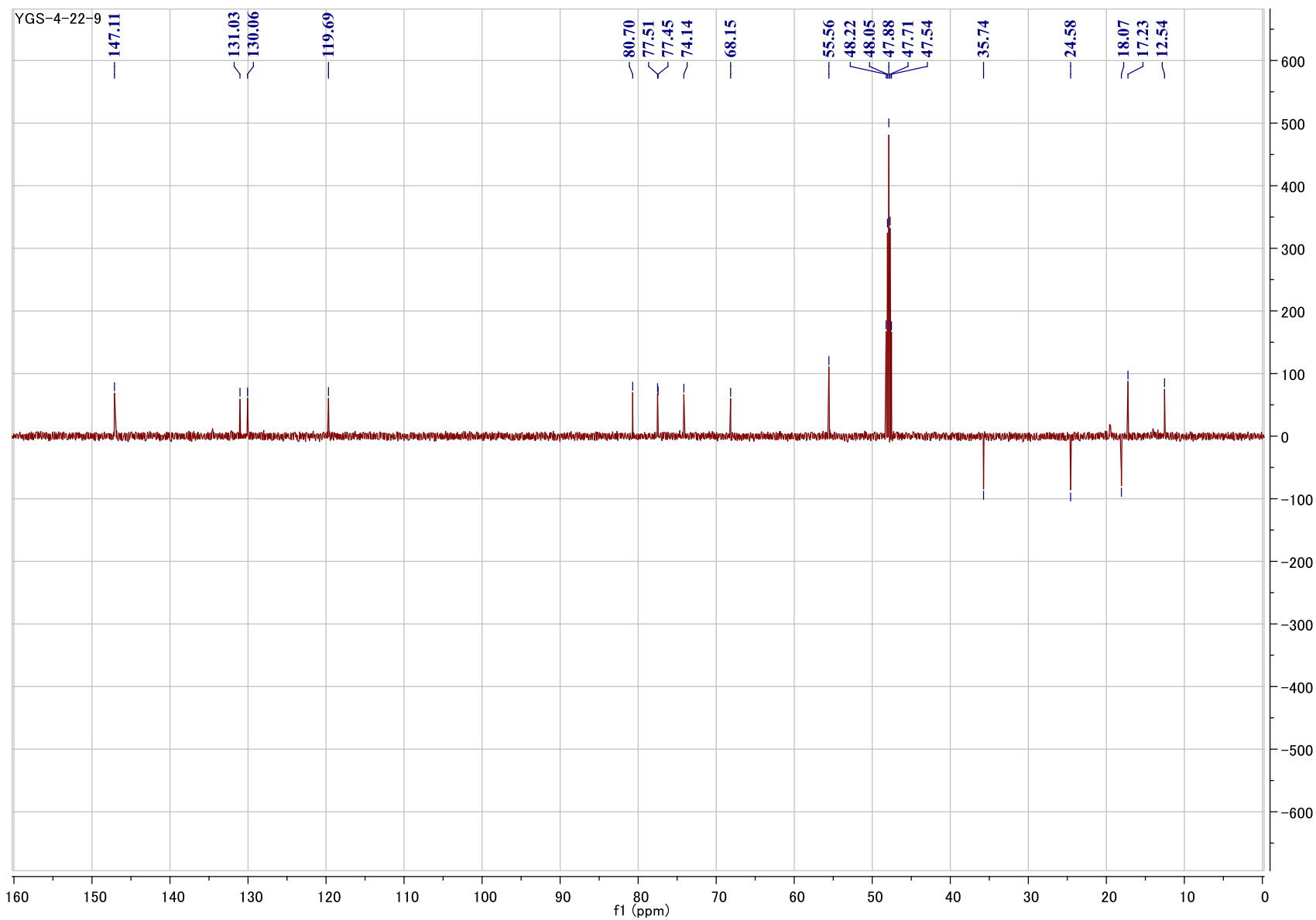
S43: HRCIMS of 6



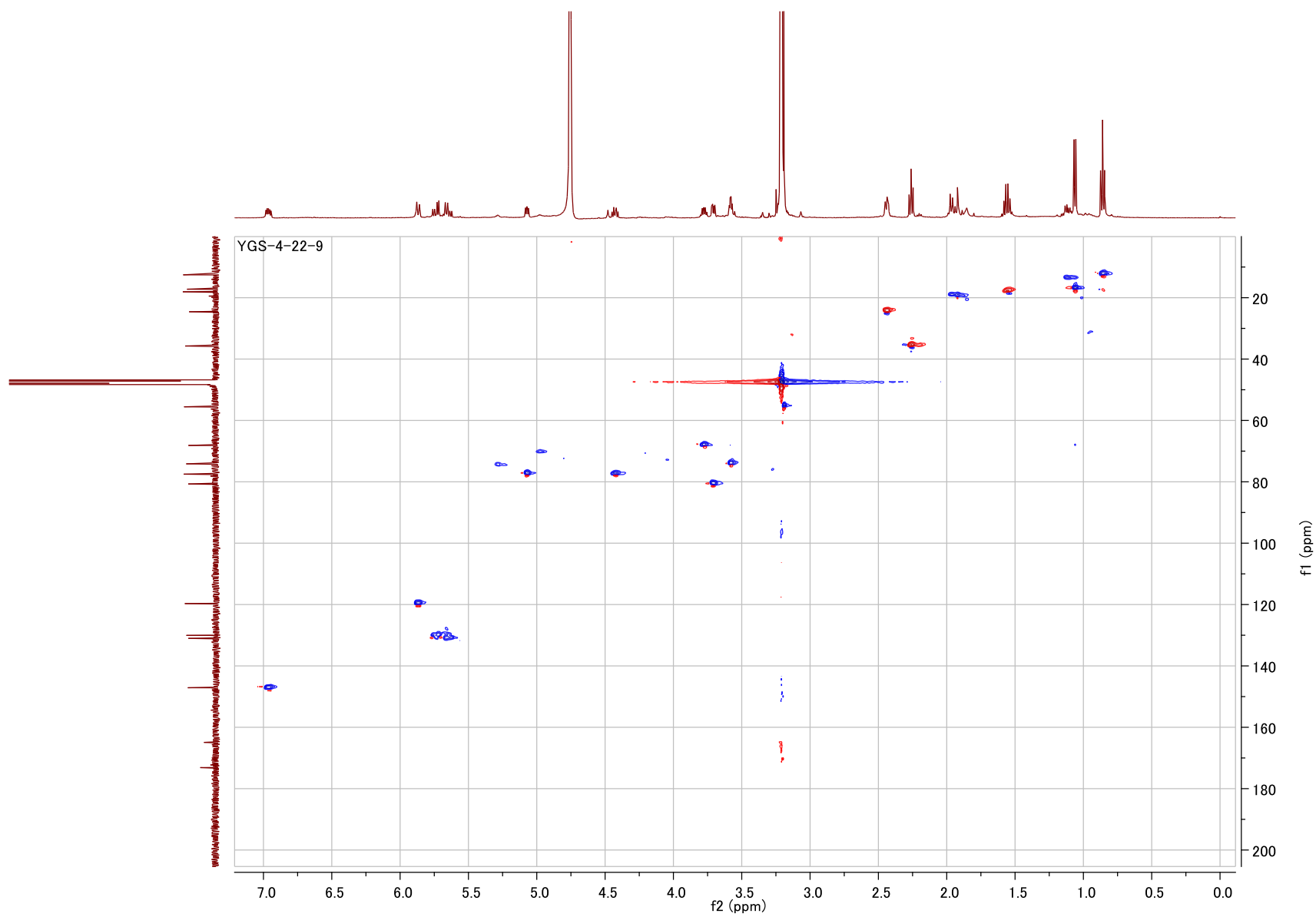
S44: ¹H NMR of 6



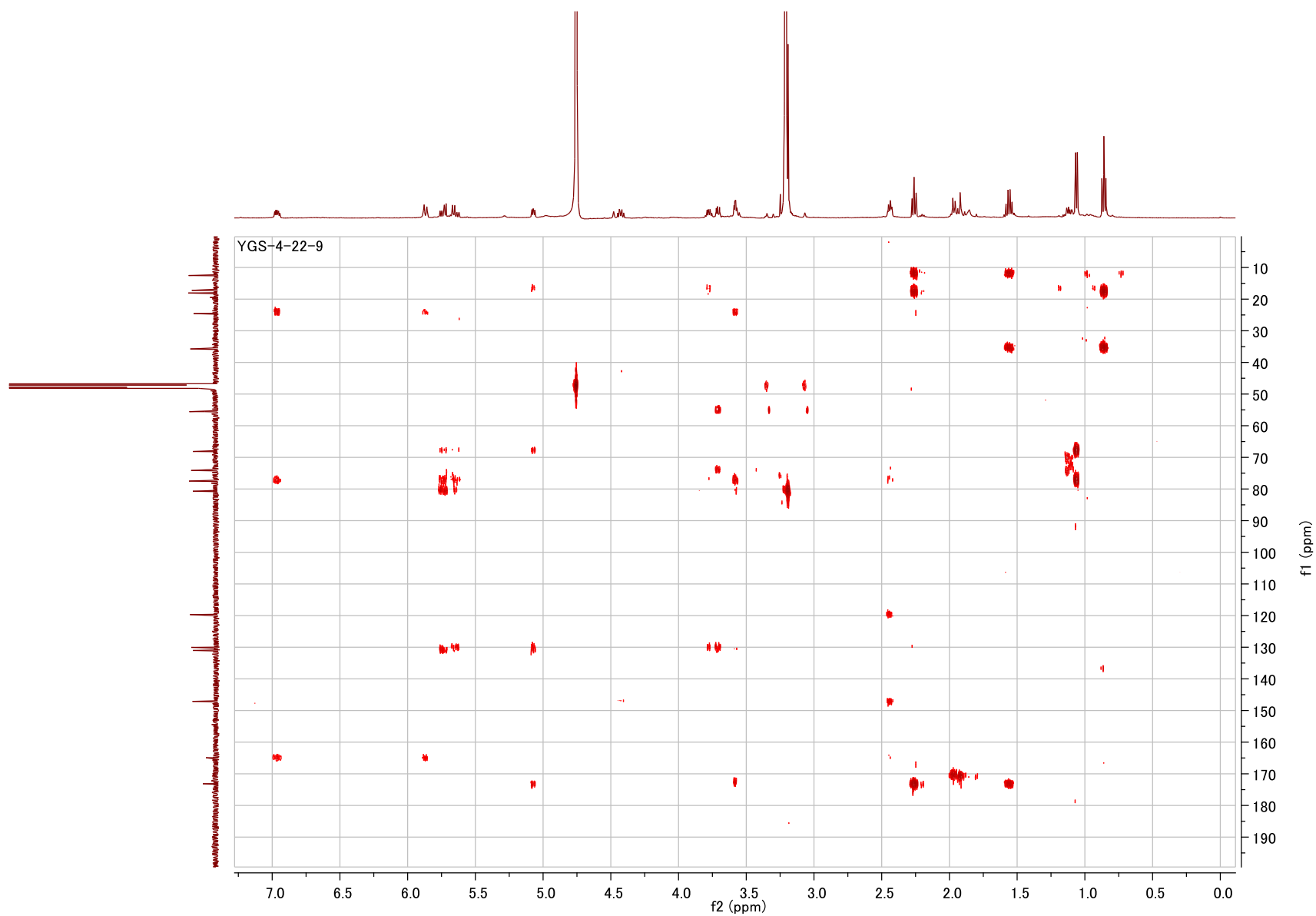
S45: ^{13}C NMR of 6



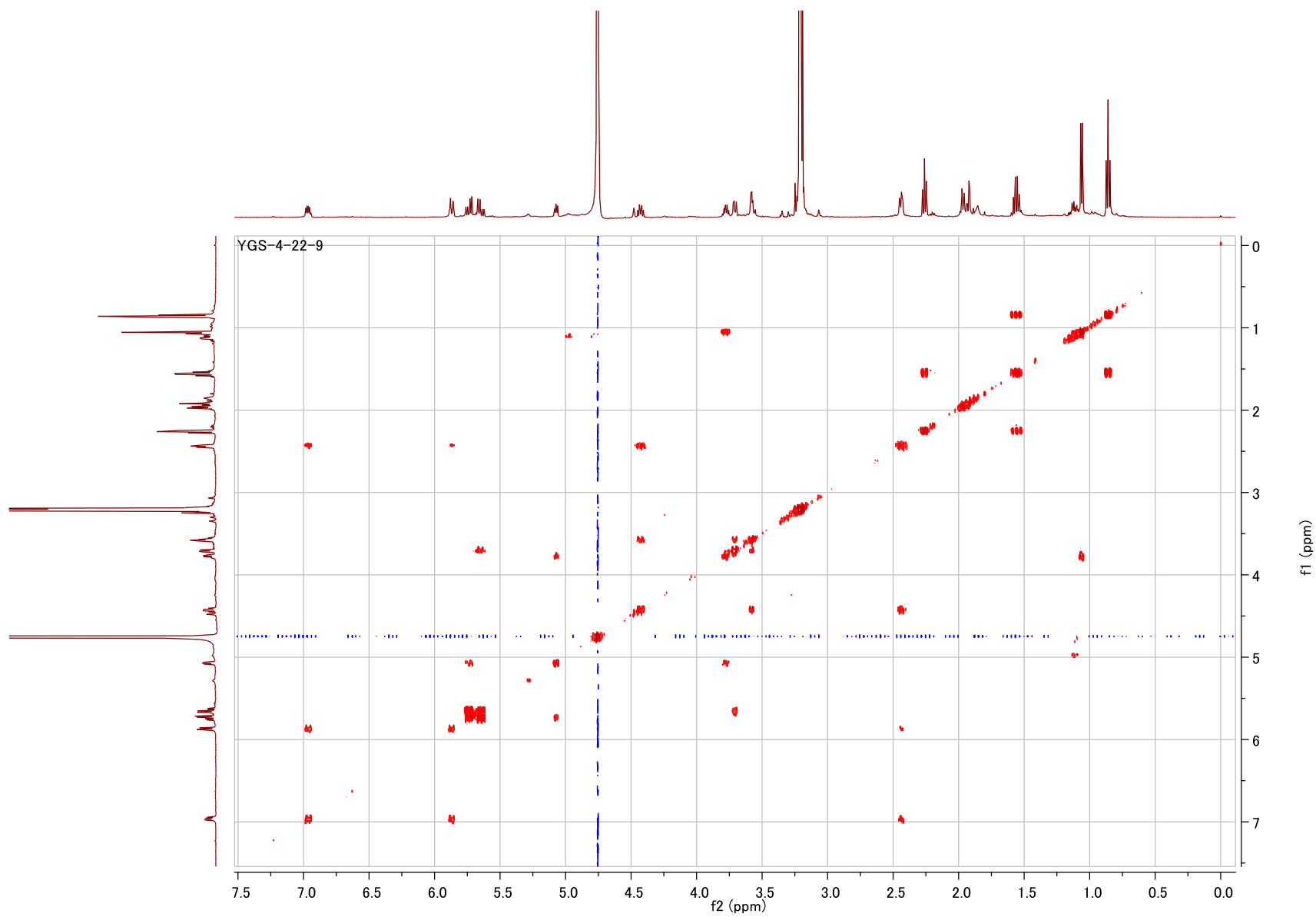
S46: DEPT-135 of **6**



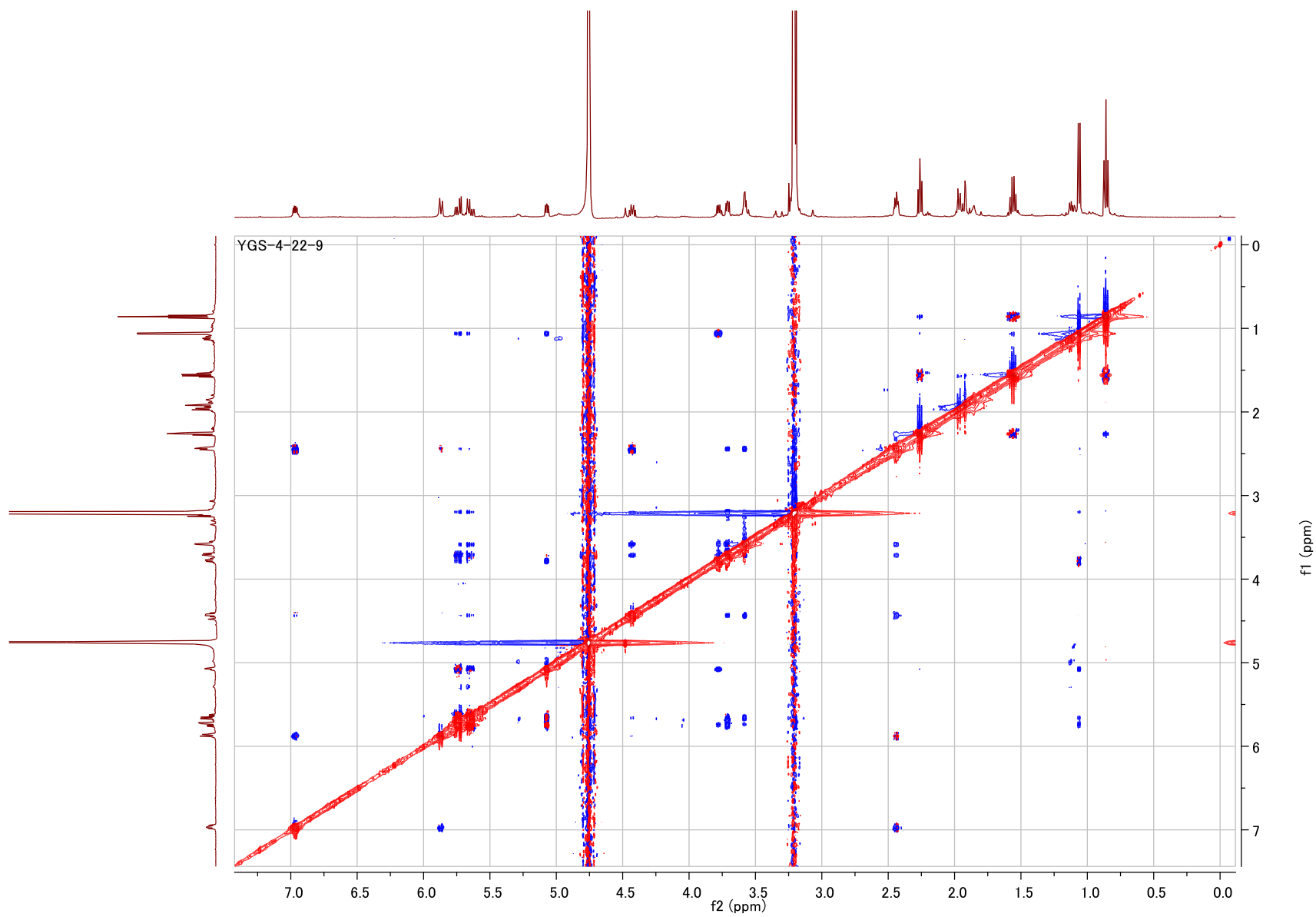
S47: HSQC of 6



S48: HMBC of 6

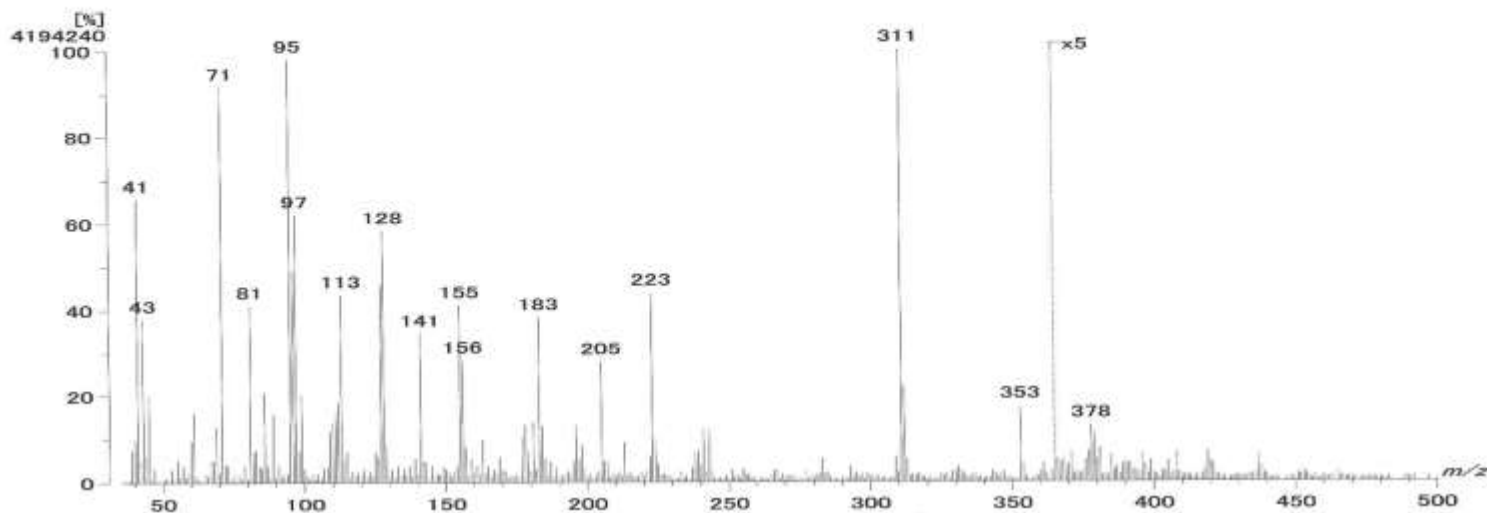


S49: ^1H ^1H COSY of **6**



S50: NOESY of 6

[Mass Spectrum]
 Data : Umeyama-CL15-Feb-2018.001 Date : 15-Feb-2018 10:36
 Sample : YGS-4-22-14(CH4)
 Note : MStation
 Inlet : Direct Ion Mode : CI+
 Spectrum Type : Normal Ion [MF-Linear]
 RT : 1.48 min Scan# : 55
 BP : m/z 311 Int. : 399.99 (4194240)
 Output m/z range : 35 to 500 Cut Level : 0.00 %

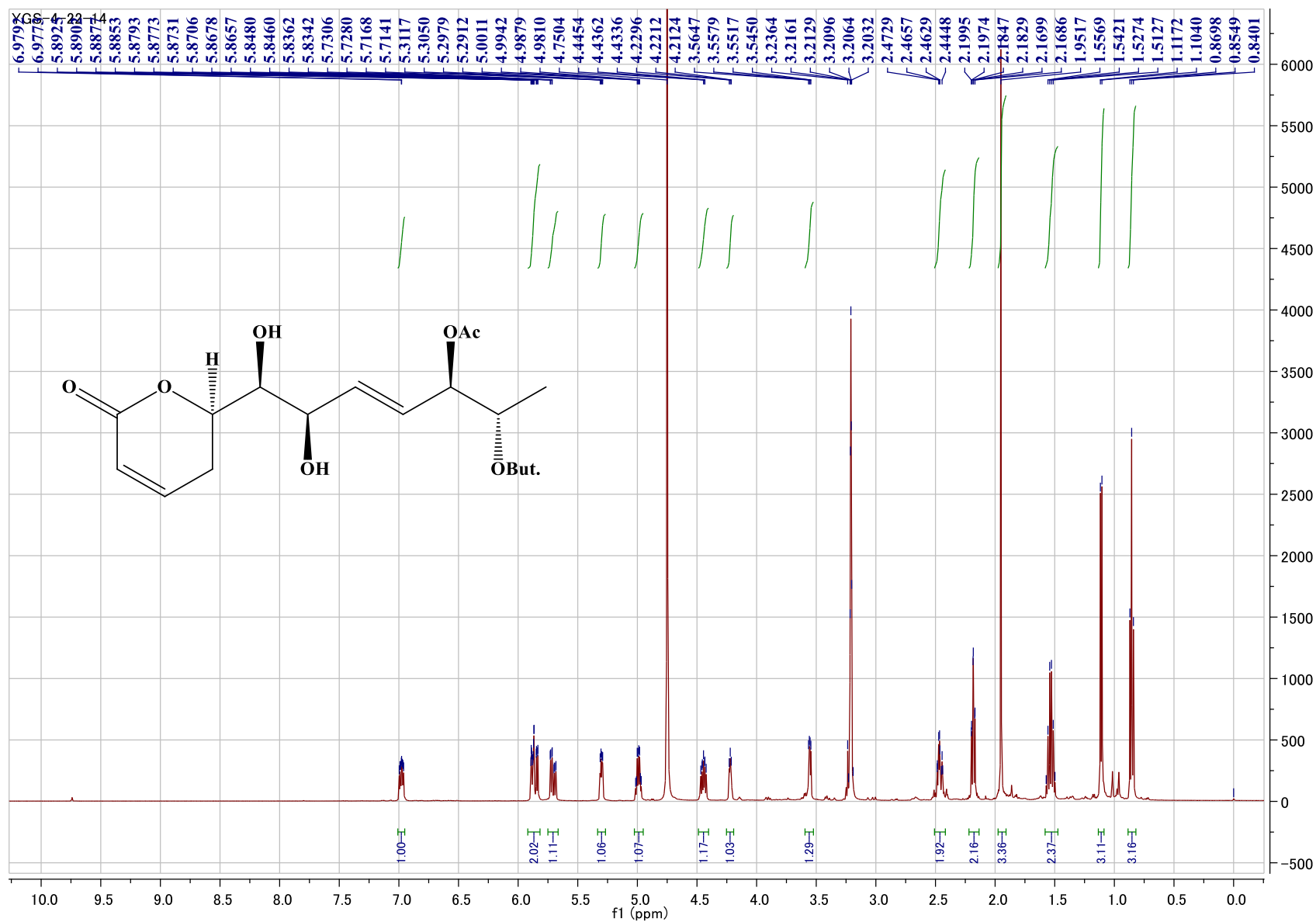


S51: LRCIMS of 7

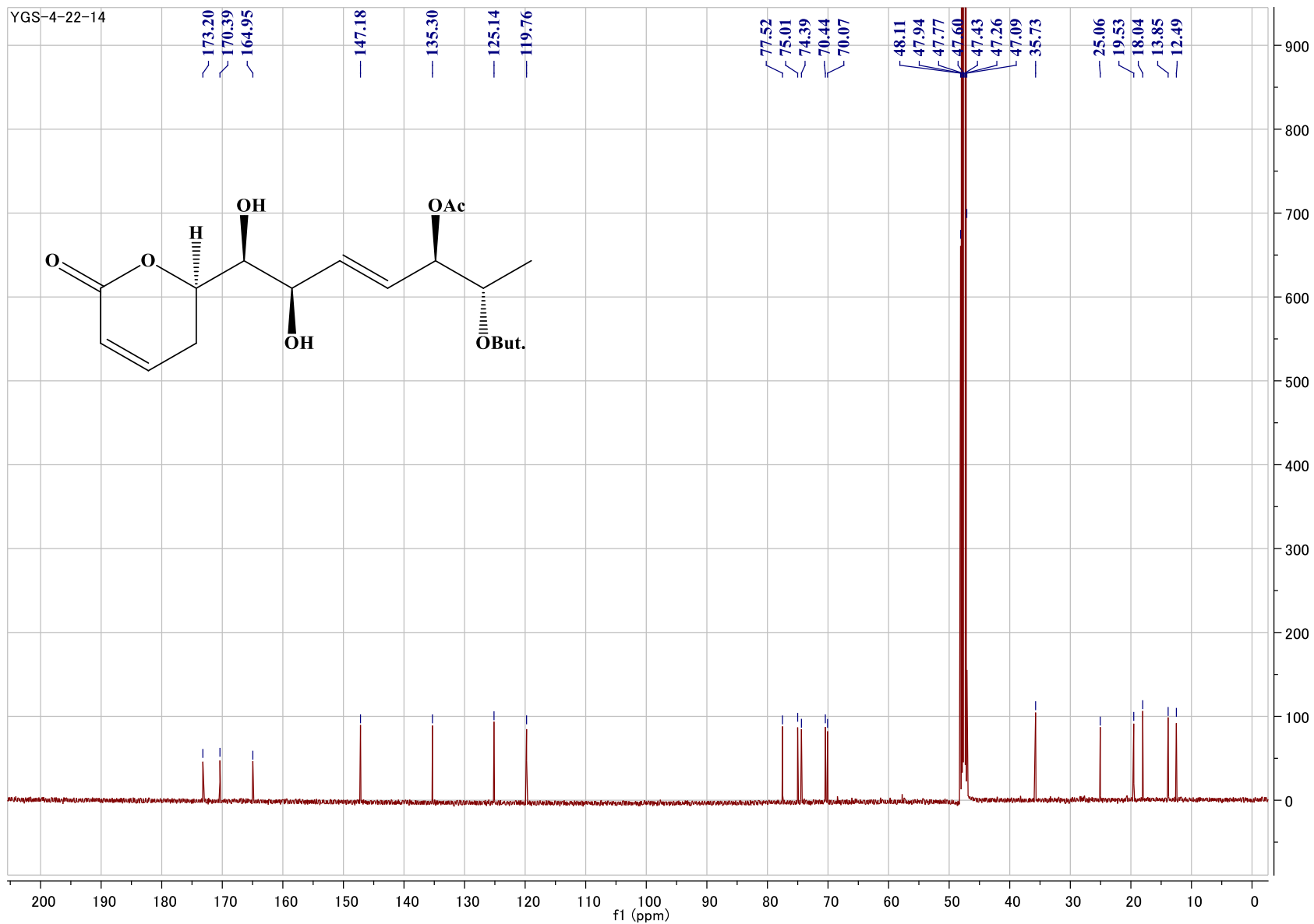
Data : Umeyama-CIHR.16-Feb-2018.001 Date : 16-Feb-2018 10:51
 Instrument : MStation
 Sample : YGS-4-22-14
 Note : MStation
 Inlet : Direct Ion Mode : CI+
 RT : 1.80 min Scan# : 46
 Elements : C 150/0, H 250/0, O 50/0
 Mass Tolerance : 5mmu
 Unsaturation (U.S.) : 0.0 - 15.0

	Observed m/z	Int%	Err [ppm / mmu]	U.S. Composition
1	371.1705	13.98	-0.3 / -0.1	5.5 C18 H27 O8

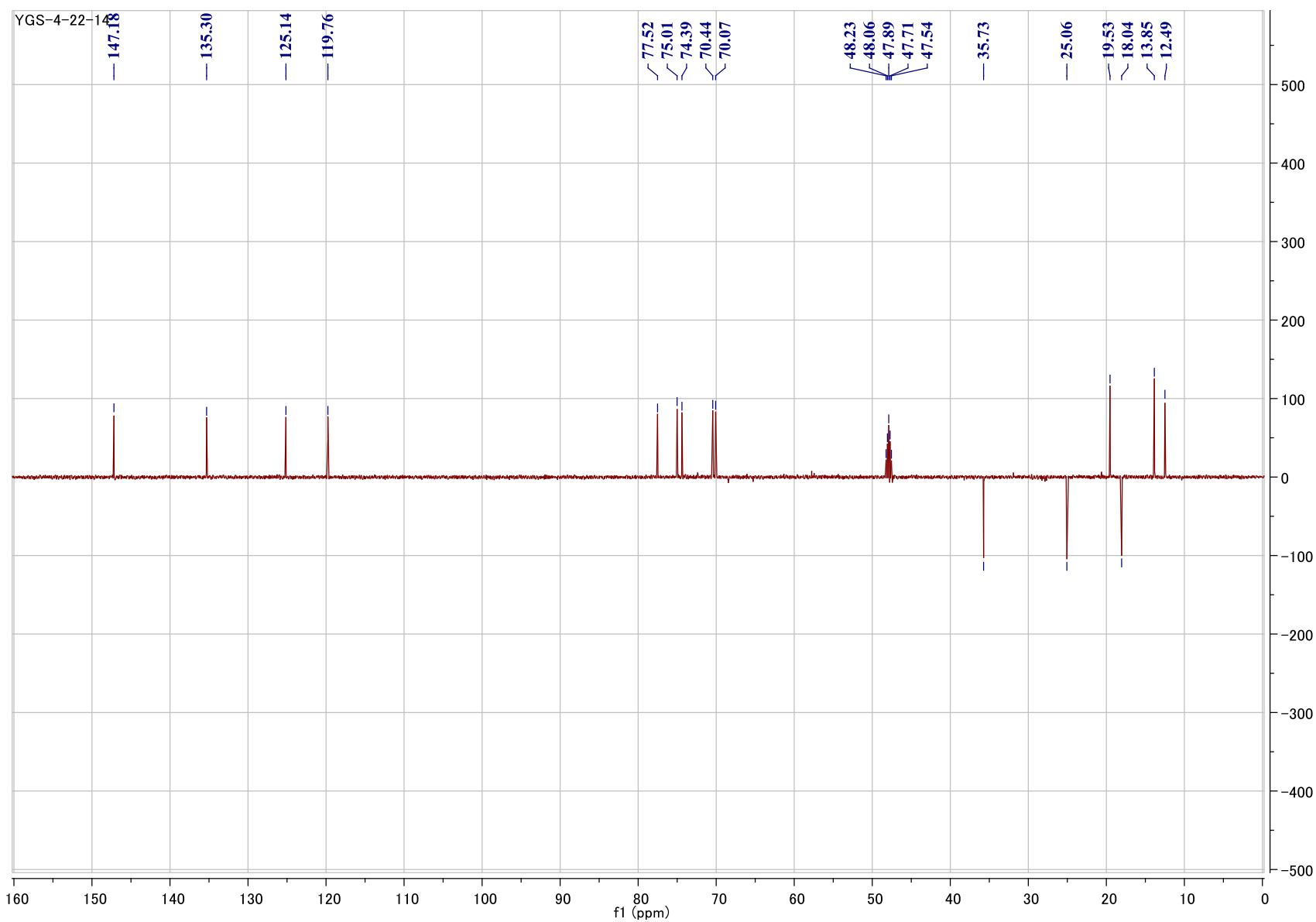
S52: HRCIMS of 7



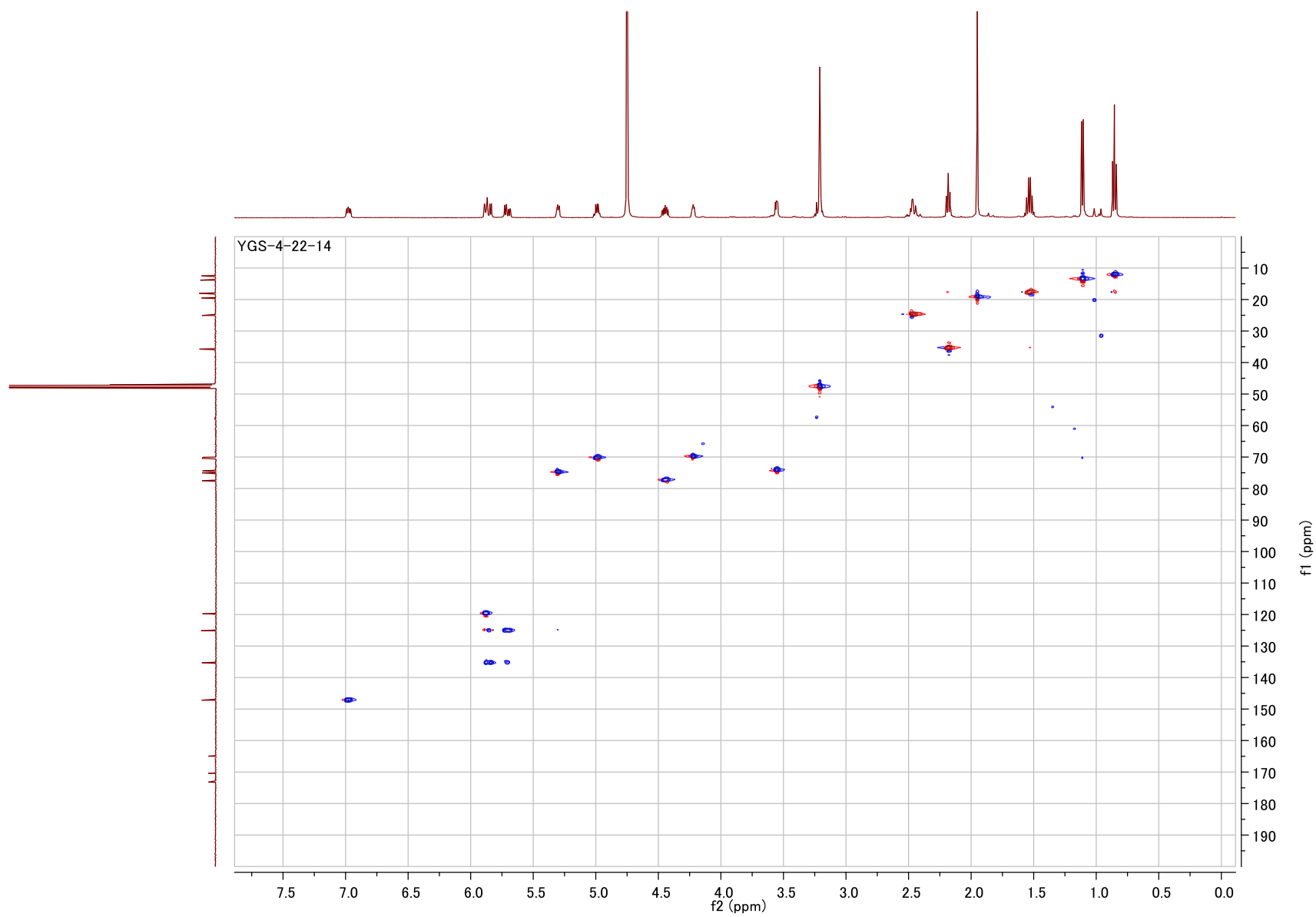
S53: ^1H NMR of 7



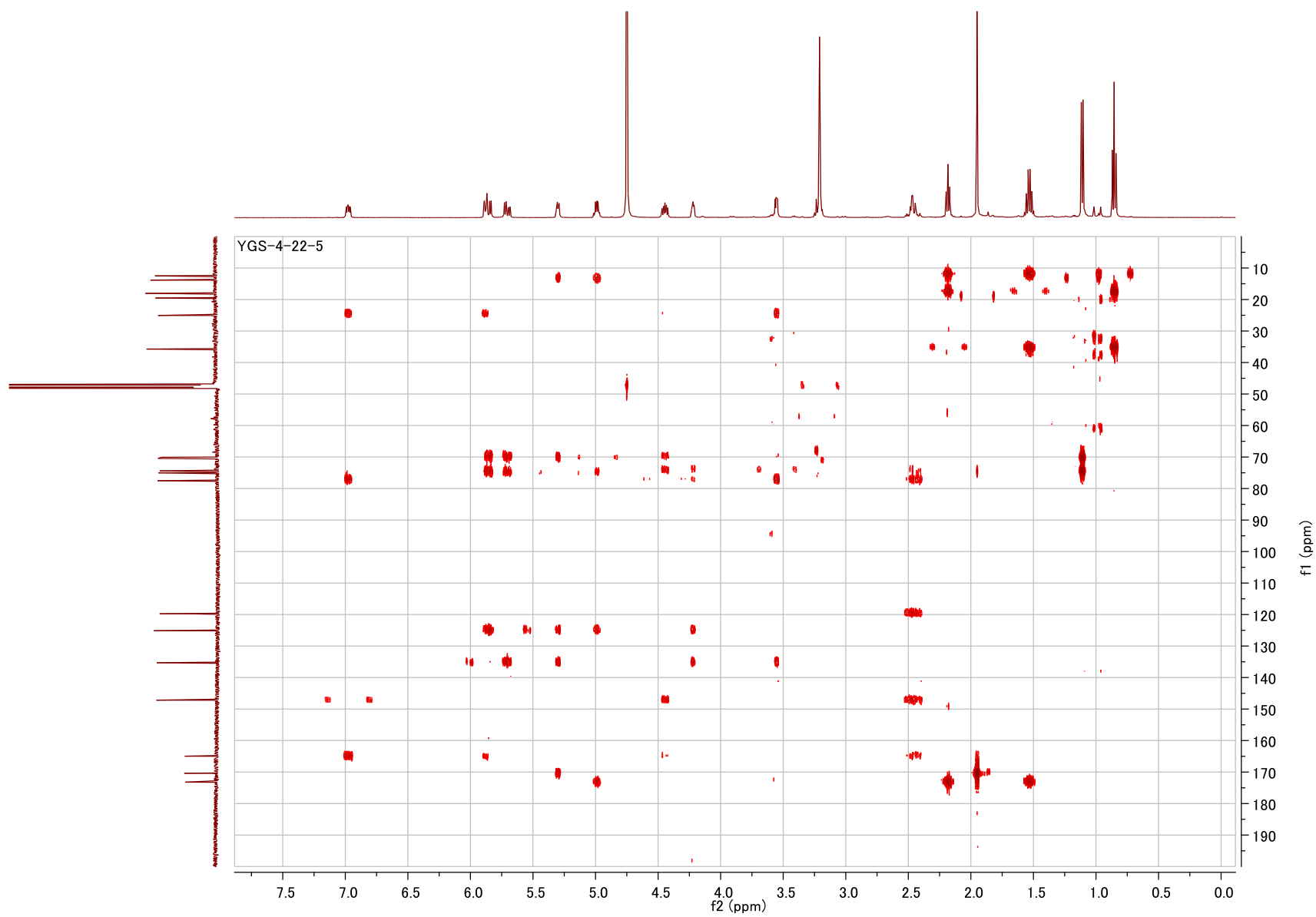
S54: ^{13}C NMR of 7



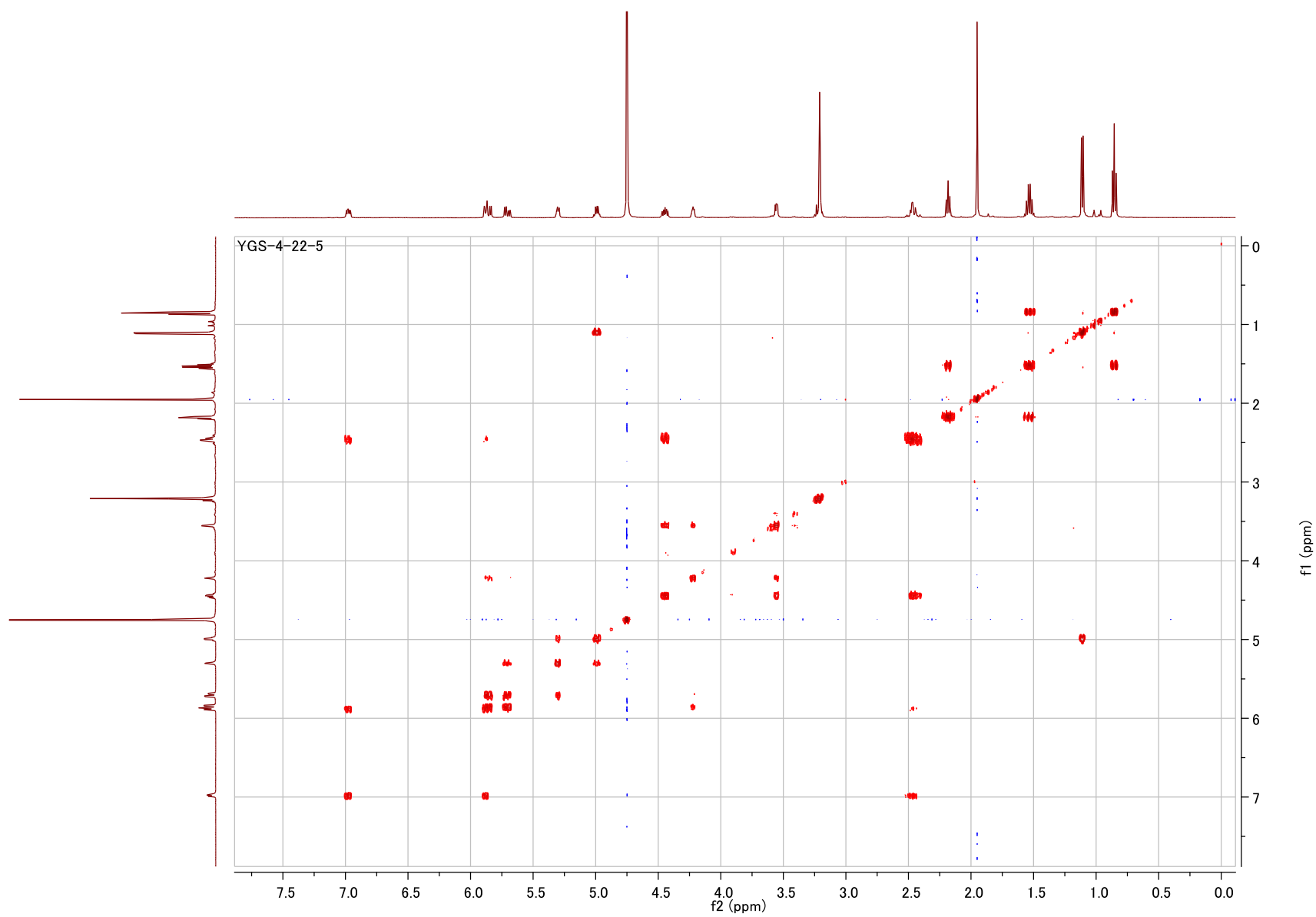
S55: DEPT-135 of 7



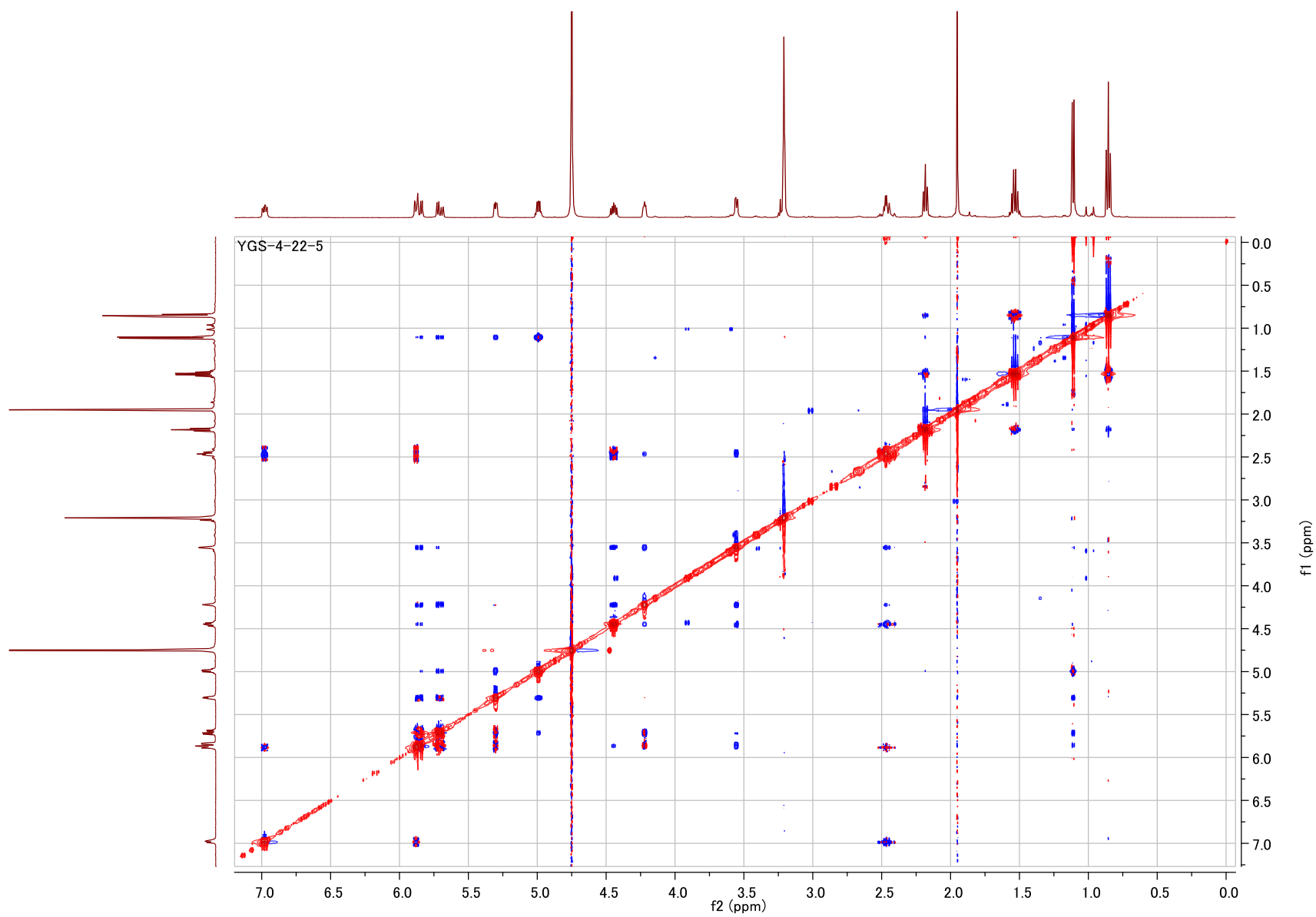
S56: HSQC of 7



S57: HMBC of 7



S58: ^1H ^1H COSY of 7

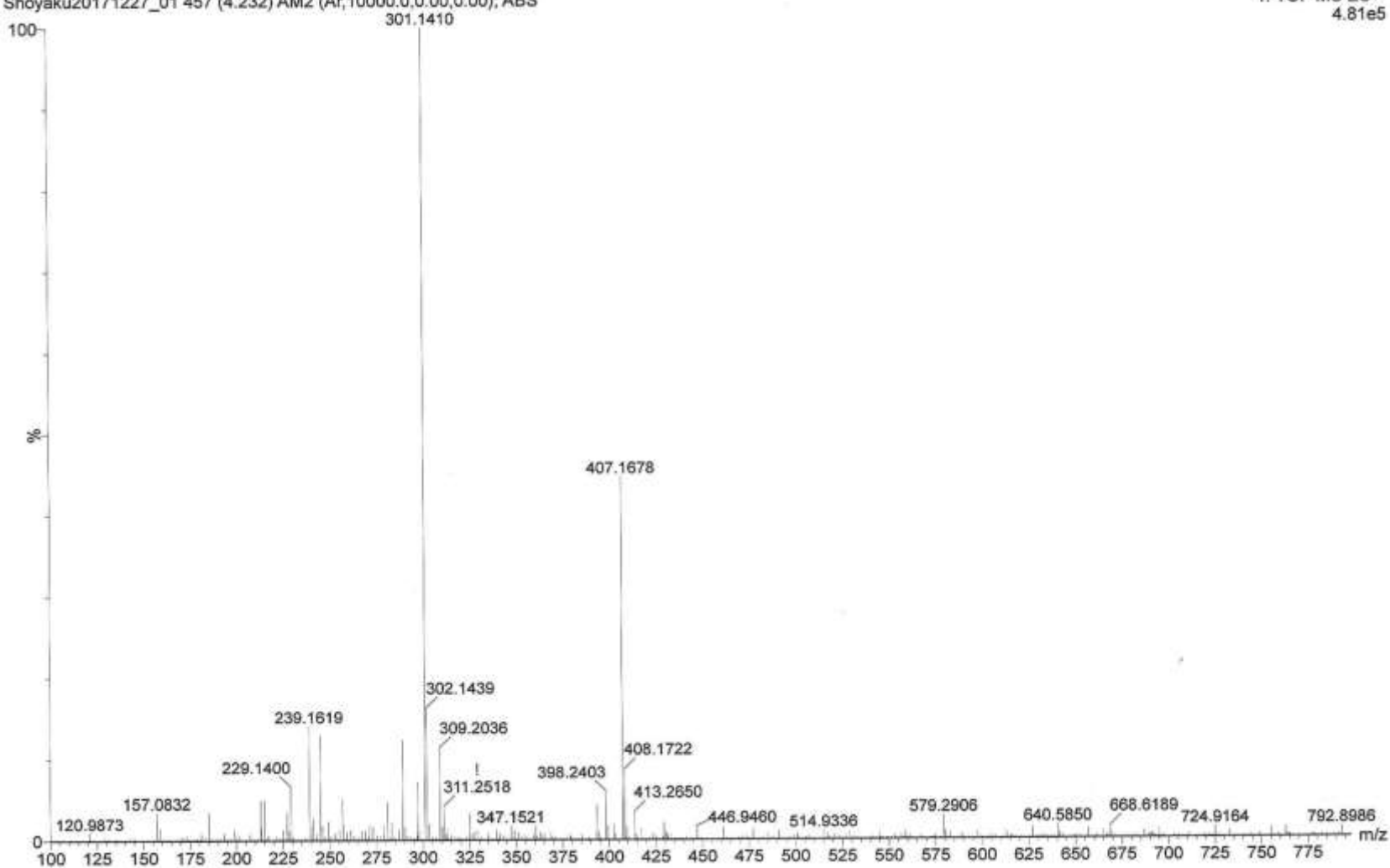


S59: NOESY of 7

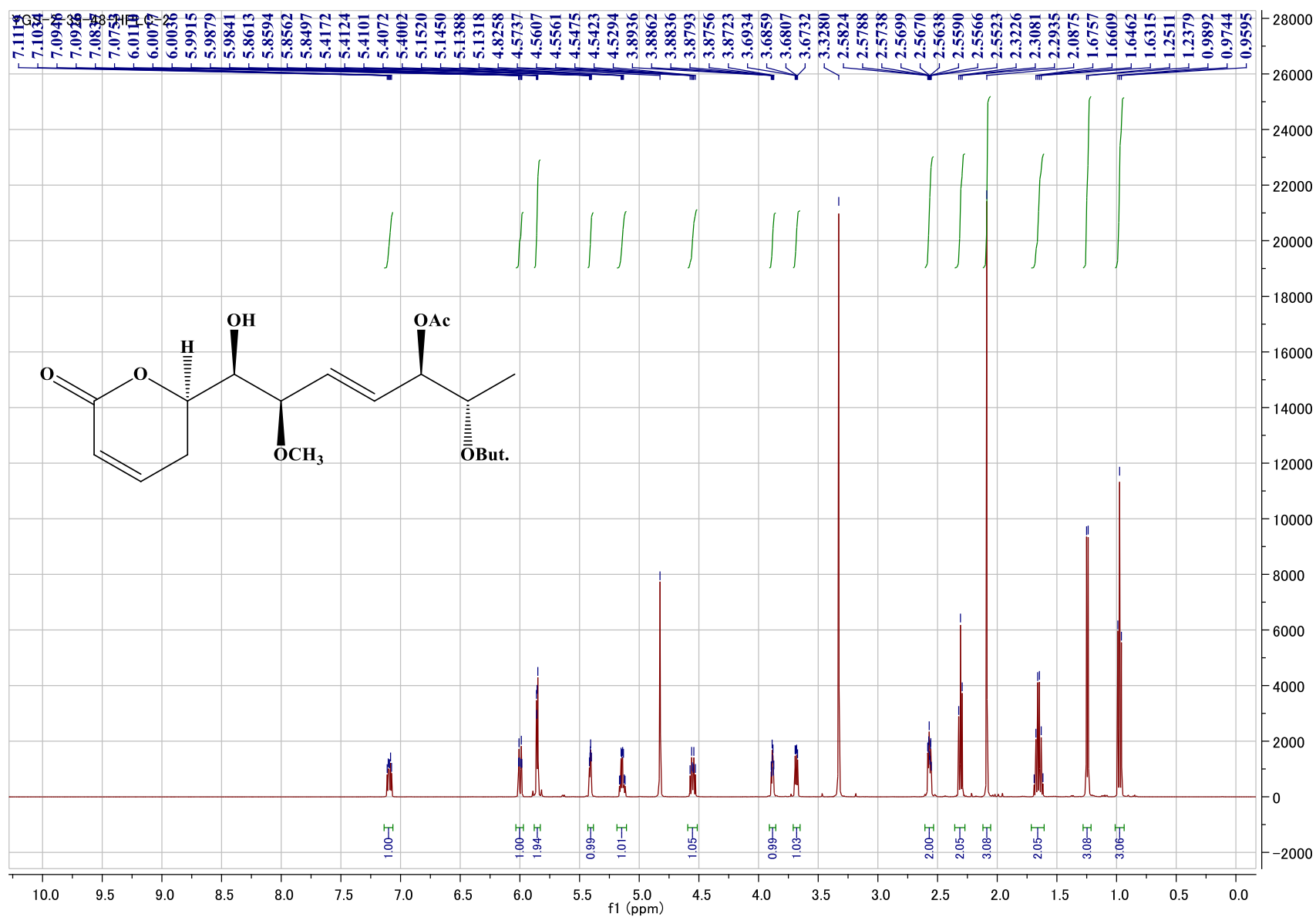
YGS-2-39-48-HPLC2 (Elshamy)

Shoyaku20171227_01 457 (4.232) AM2 (Ar,10000.0,0.00,0.00); ABS

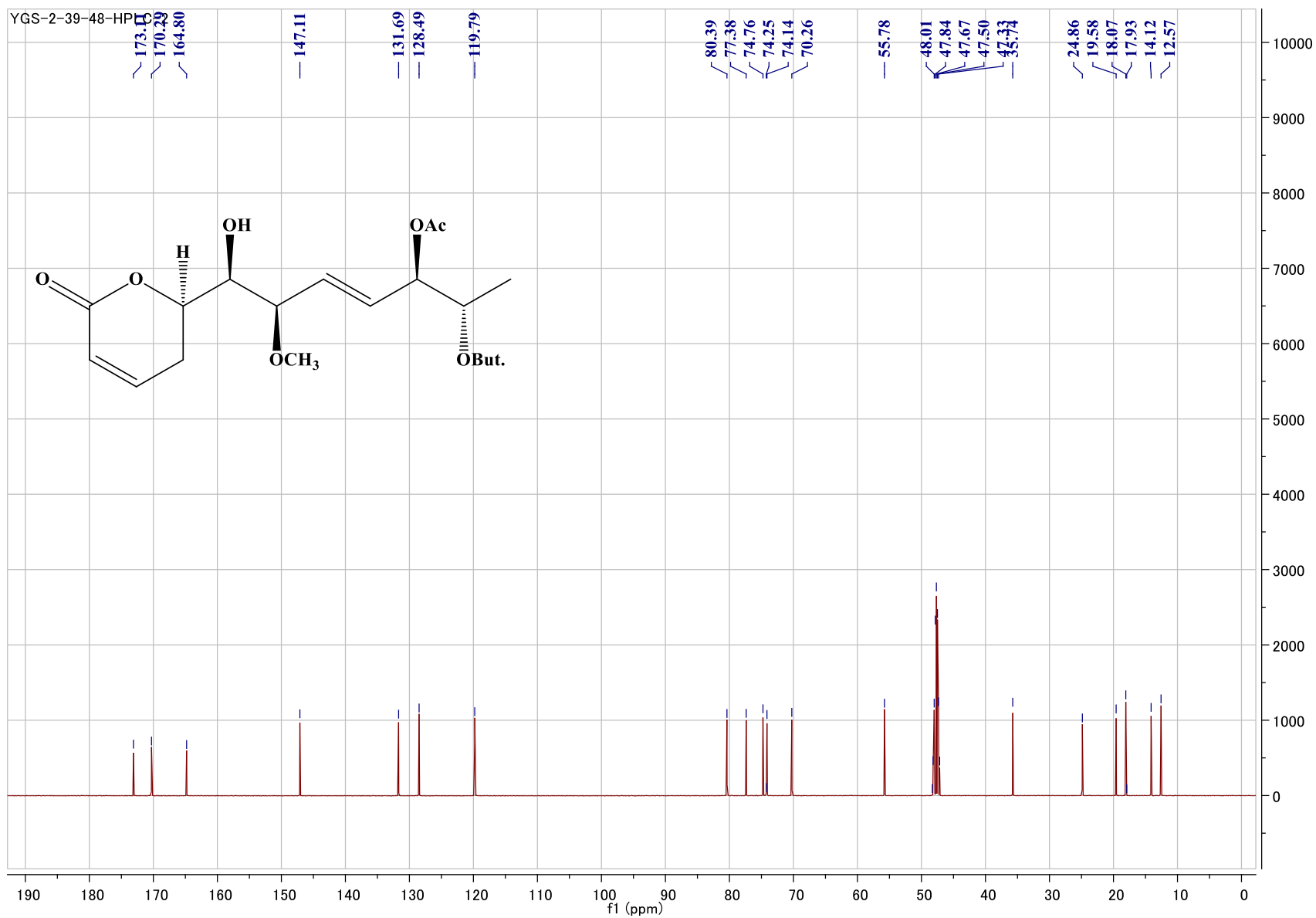
1: TOF MS ES+
4.81e5



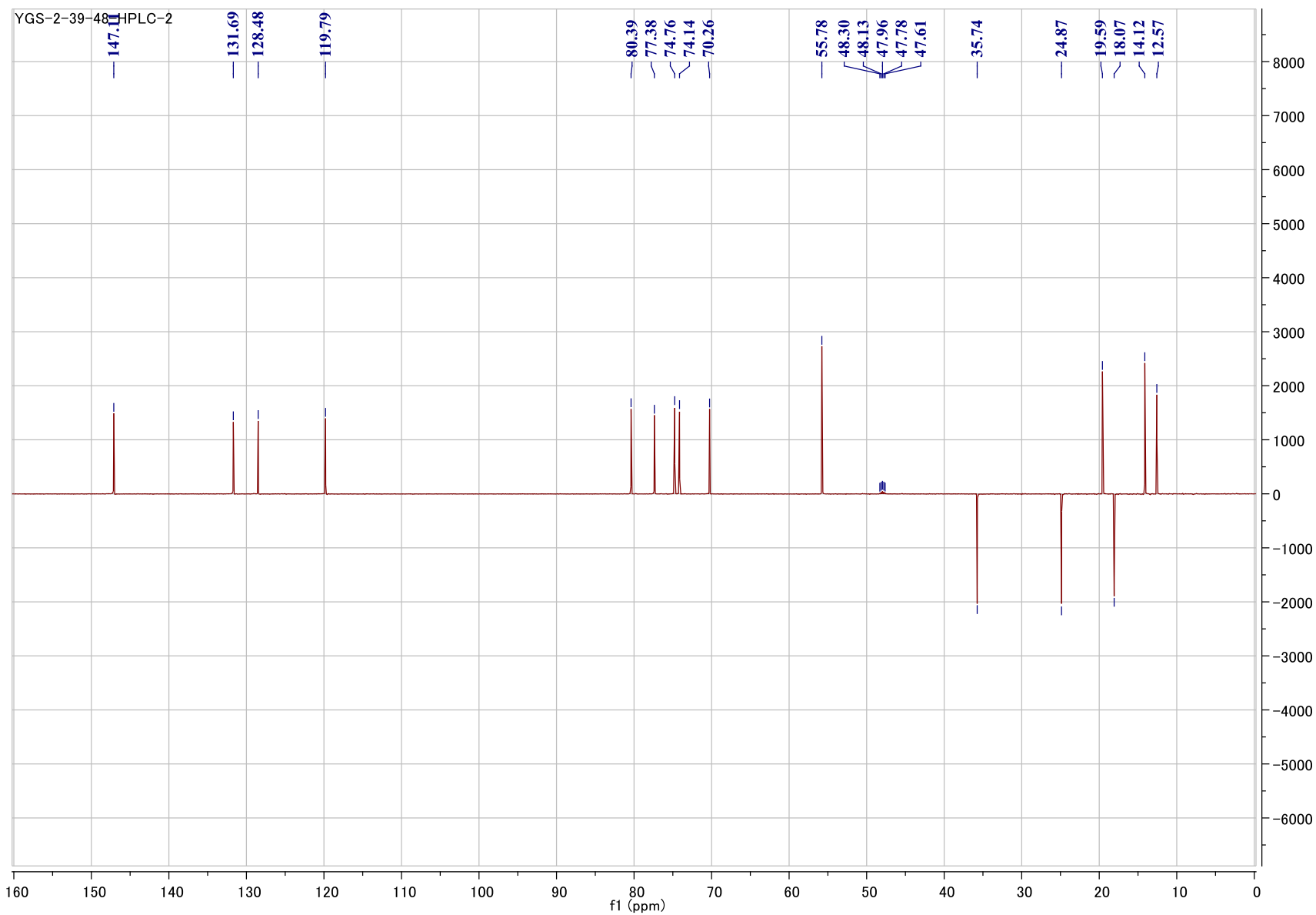
S60: TOFESIMS of 8



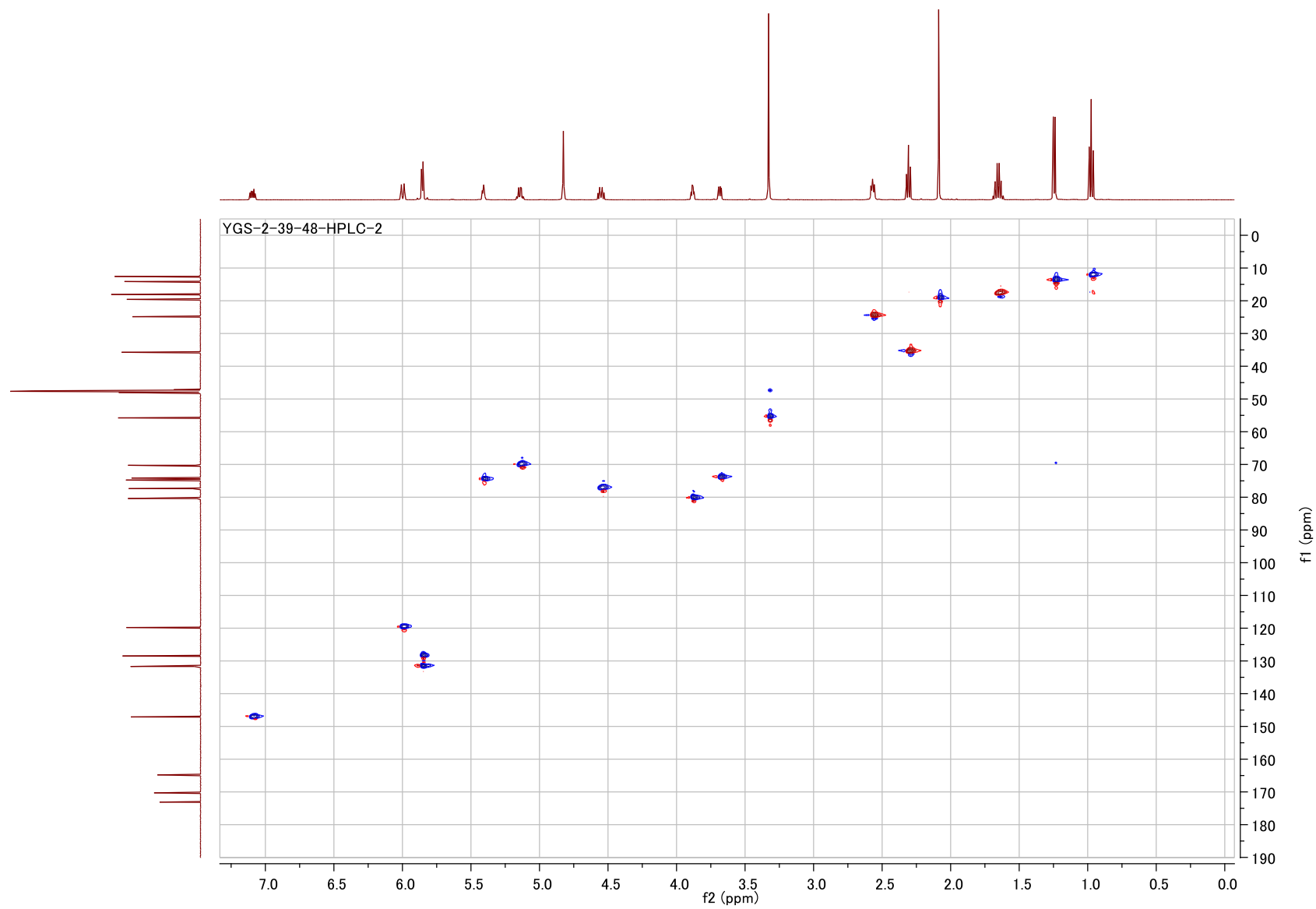
S61: ¹H NMR of 8



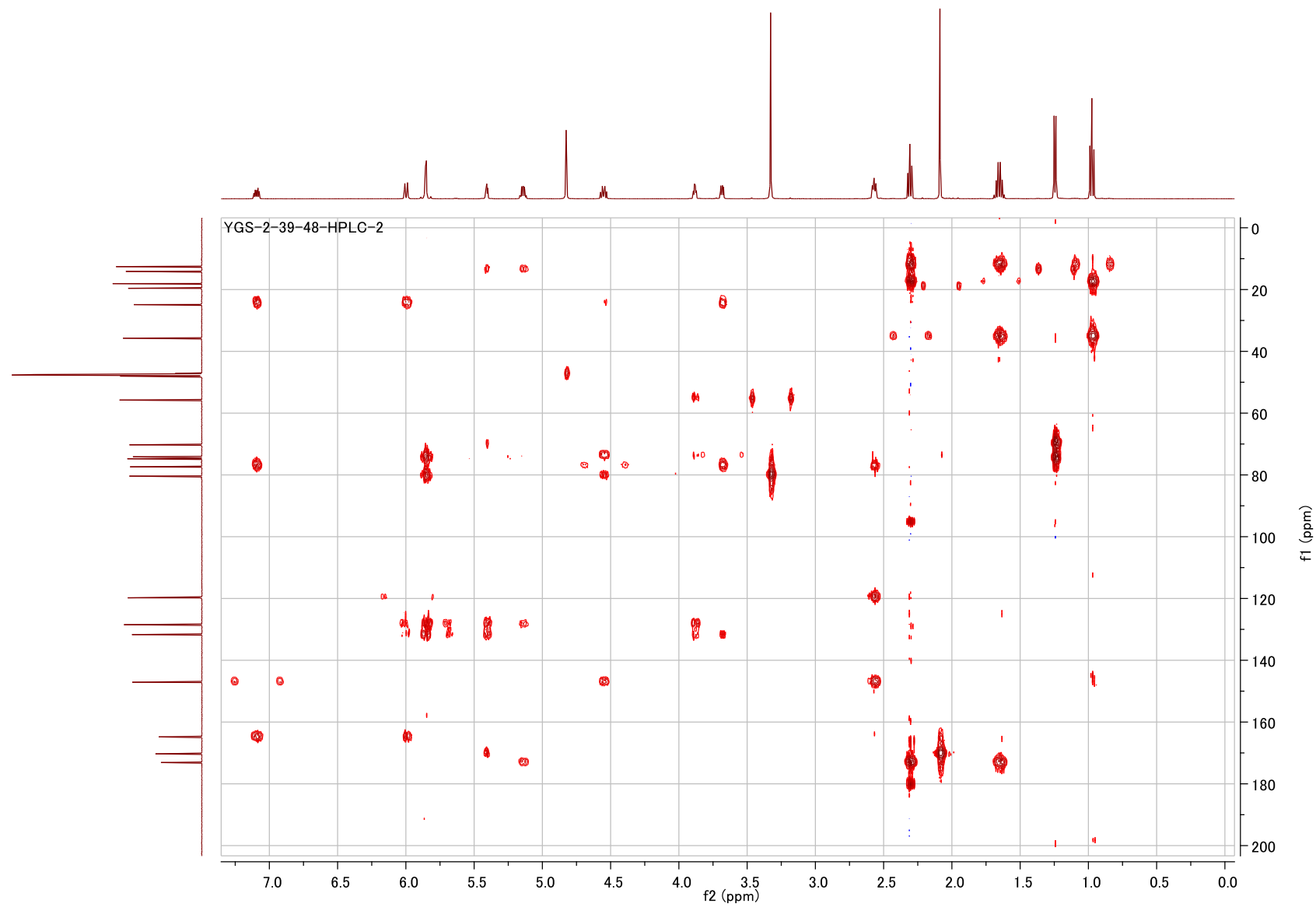
S62: ^{13}C NMR of 8



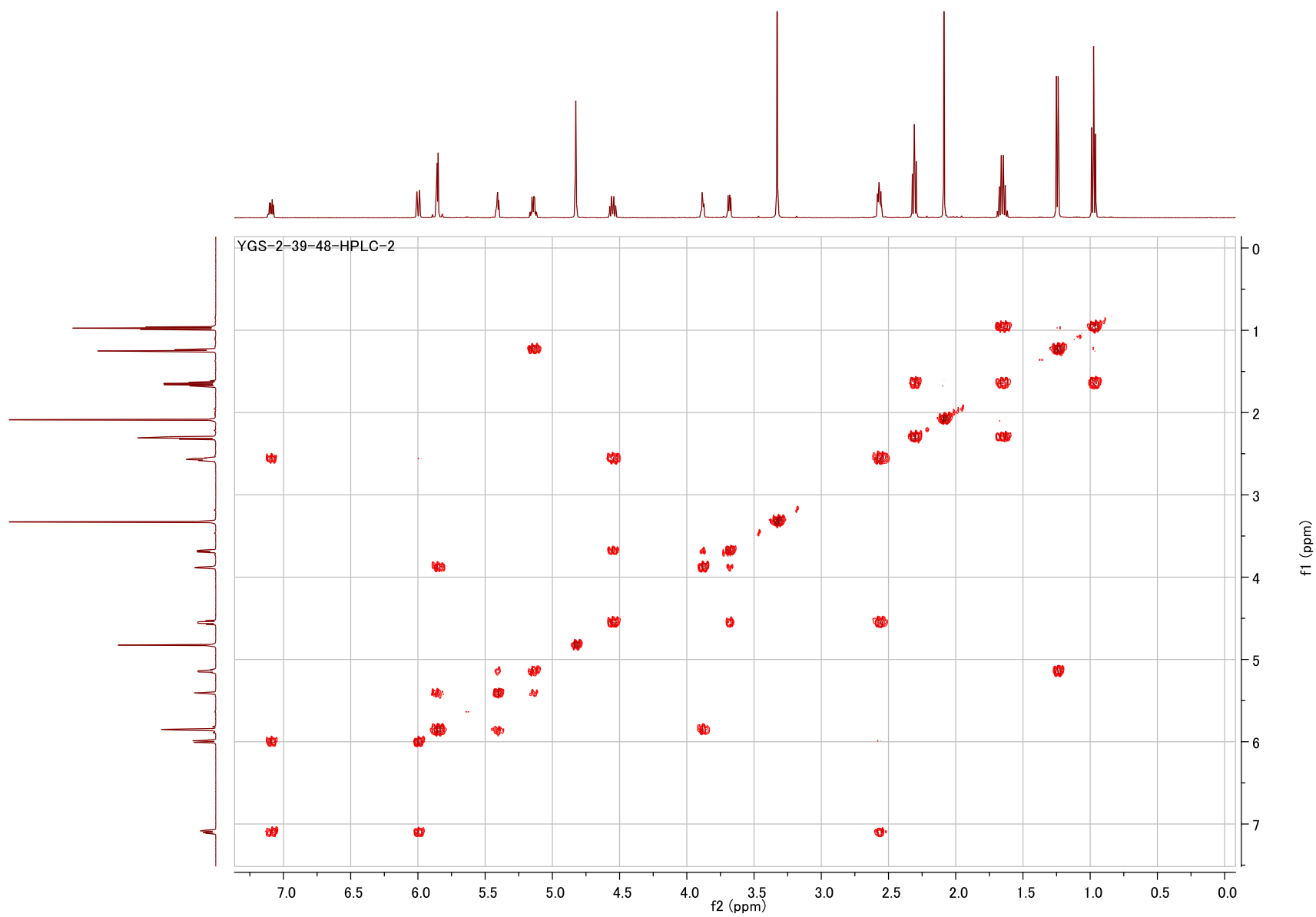
S63: DEPT-135 of 8



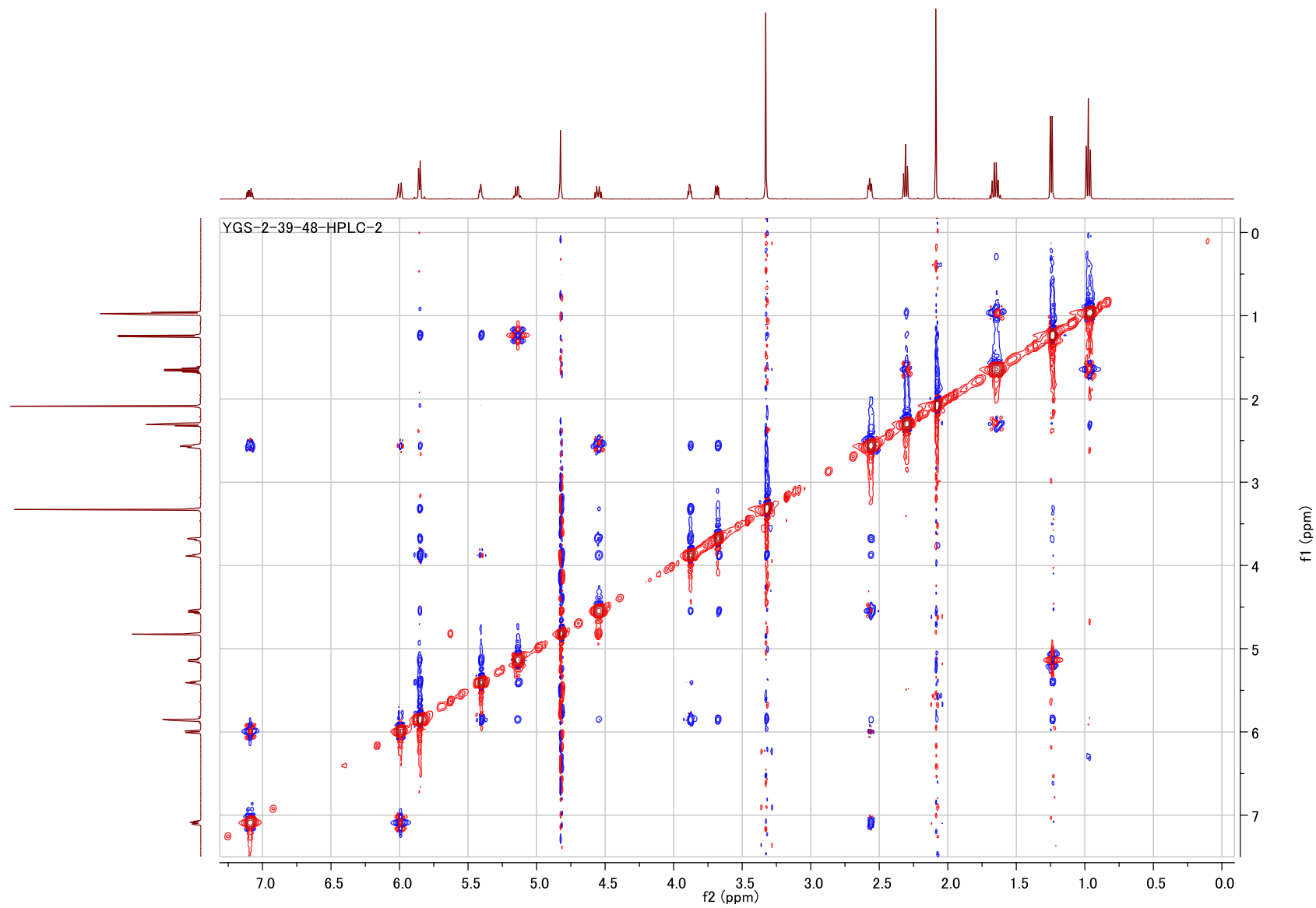
S64: HSQC of 8



S65: HMBC of **8**

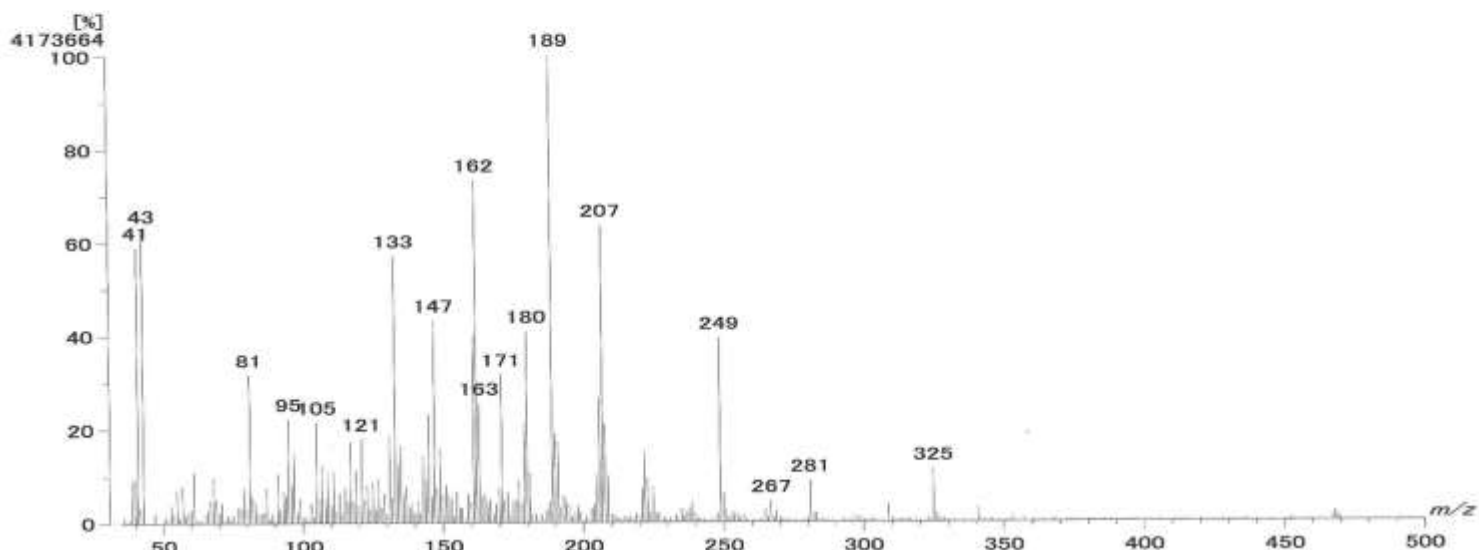


S66: ^1H ^1H COSY of **8**



S67: NOESY of 8

[Mass Spectrum]
 Data : Umeyama-CI.05-Mar-2018.003 Date : 05-Mar-2018 15:59
 Sample : YGS-4-22-7A(CH4)
 Note : MStation
 Inlet : Direct Ion Mode : CI+
 Spectrum Type : Normal Ion [MF-Linear]
 RT : 0.93 min Scan# : 35-k(12)[k=1.0]
 BP : m/z 189 Int. : 398.03 (4173664)
 Output m/z range : 35 to 500 Cut Level : 0.00 %

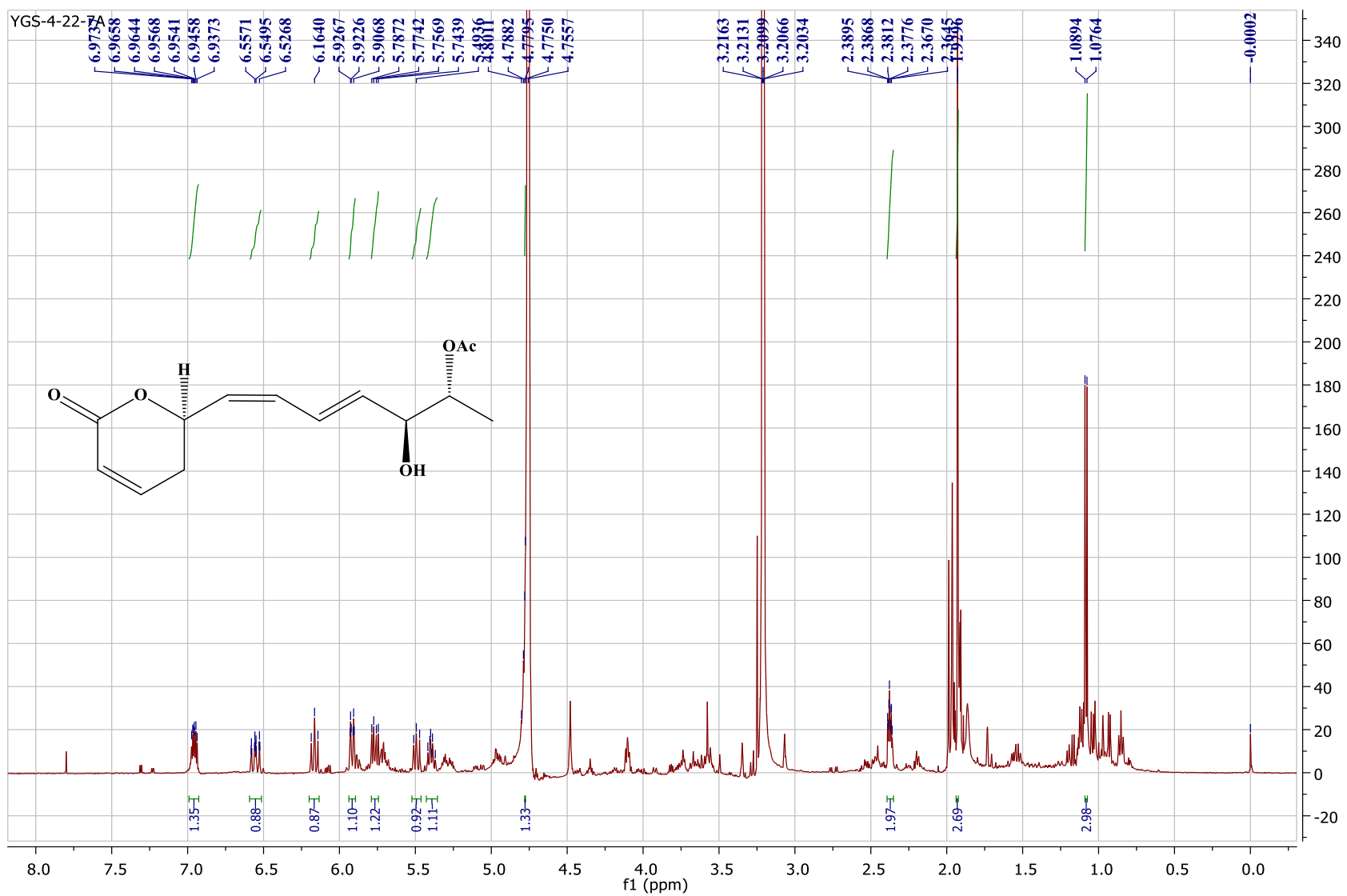


S68: LRCIMS of 9

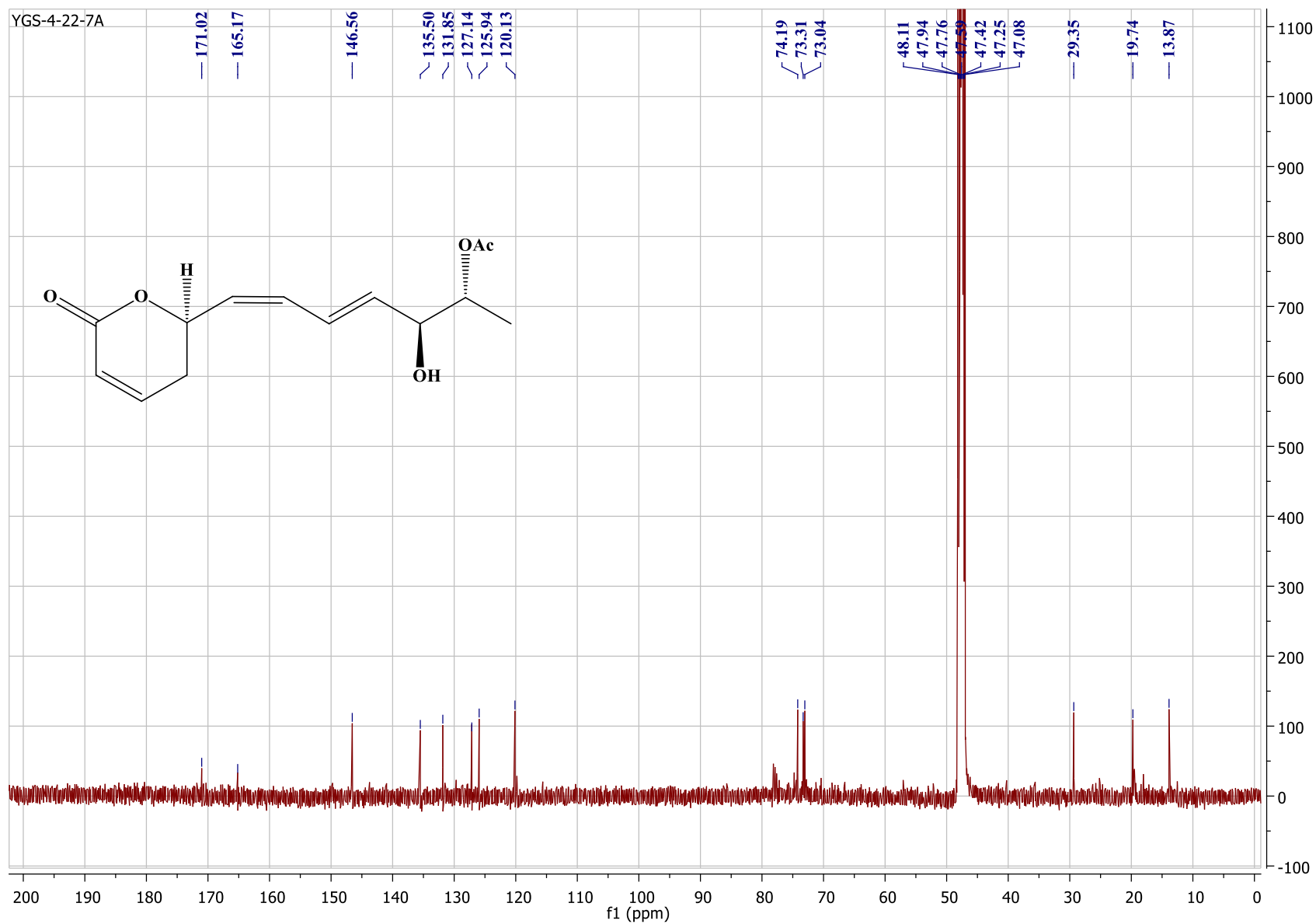
Data : Umeyama-CIHR.05-Mar-2018.001 Date : 05-Mar-2018 16:12
 Instrument : MStation
 Sample : YGS-4-22-7A
 Note : MStation
 Inlet : Direct Ion Mode : CI+
 RT : 1.22 min Scan# : 29
 Elements : C 150/0, H 250/0, O 50/0
 Mass Tolerance : 5mmu
 Unsaturation (U.S.) : 0.0 - 15.0

Observed m/z	Int%	Err [ppm / mmu]	U.S. Composition
1 267.1236	12.71	+1.3 / +0.4	5.5 C14 H19 O5

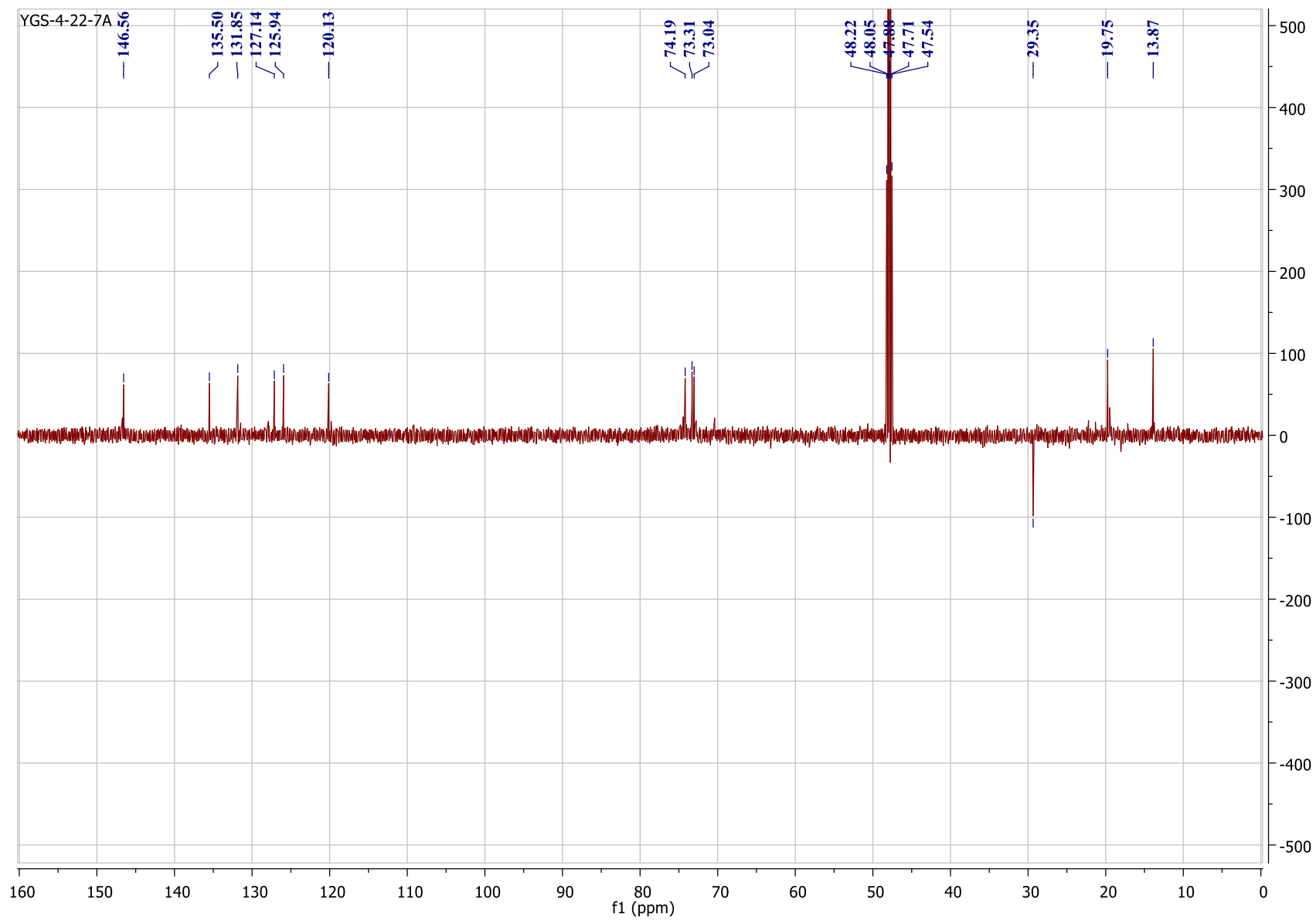
S69: HRCIMS of 9



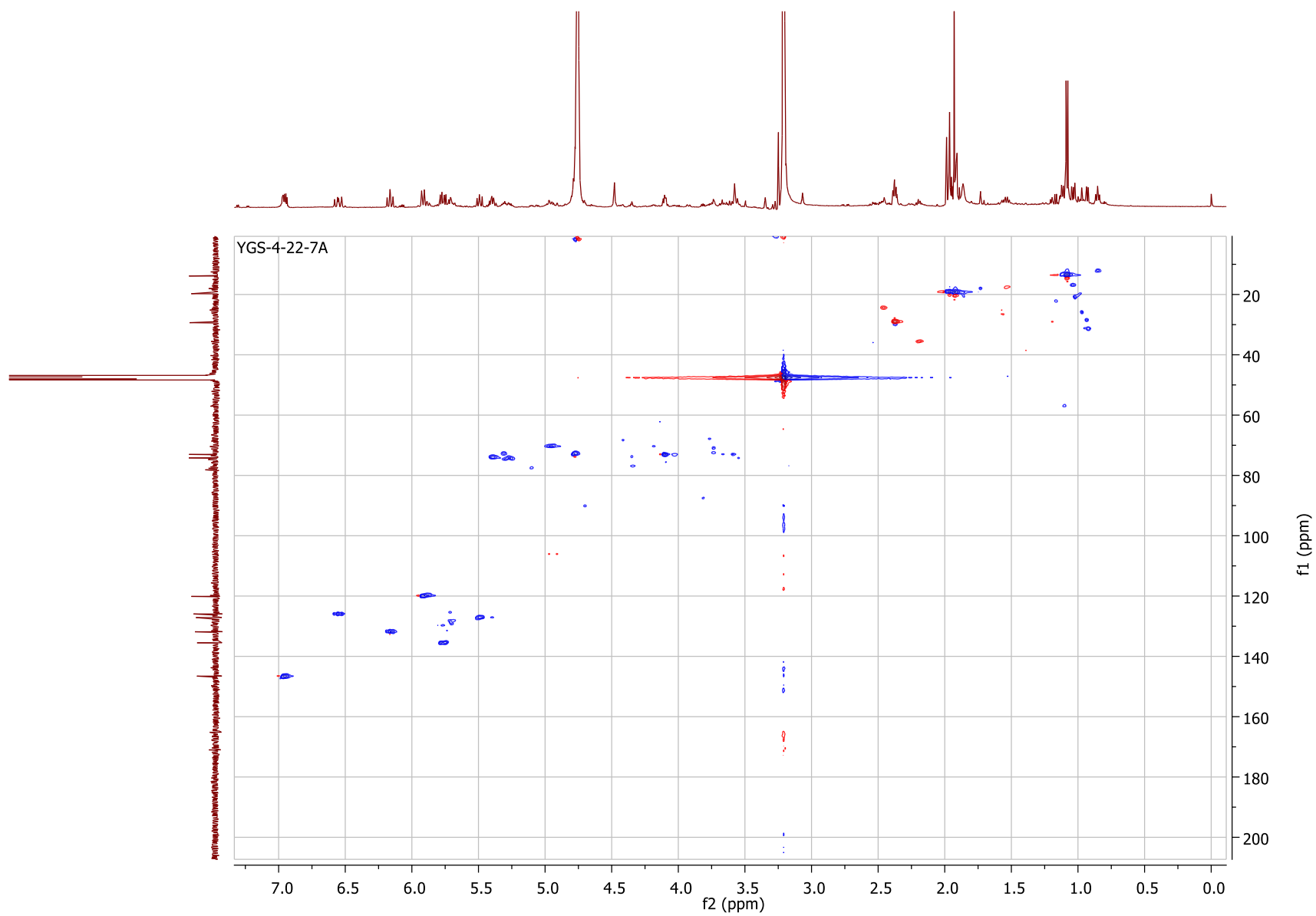
S70: ^1H NMR of 9



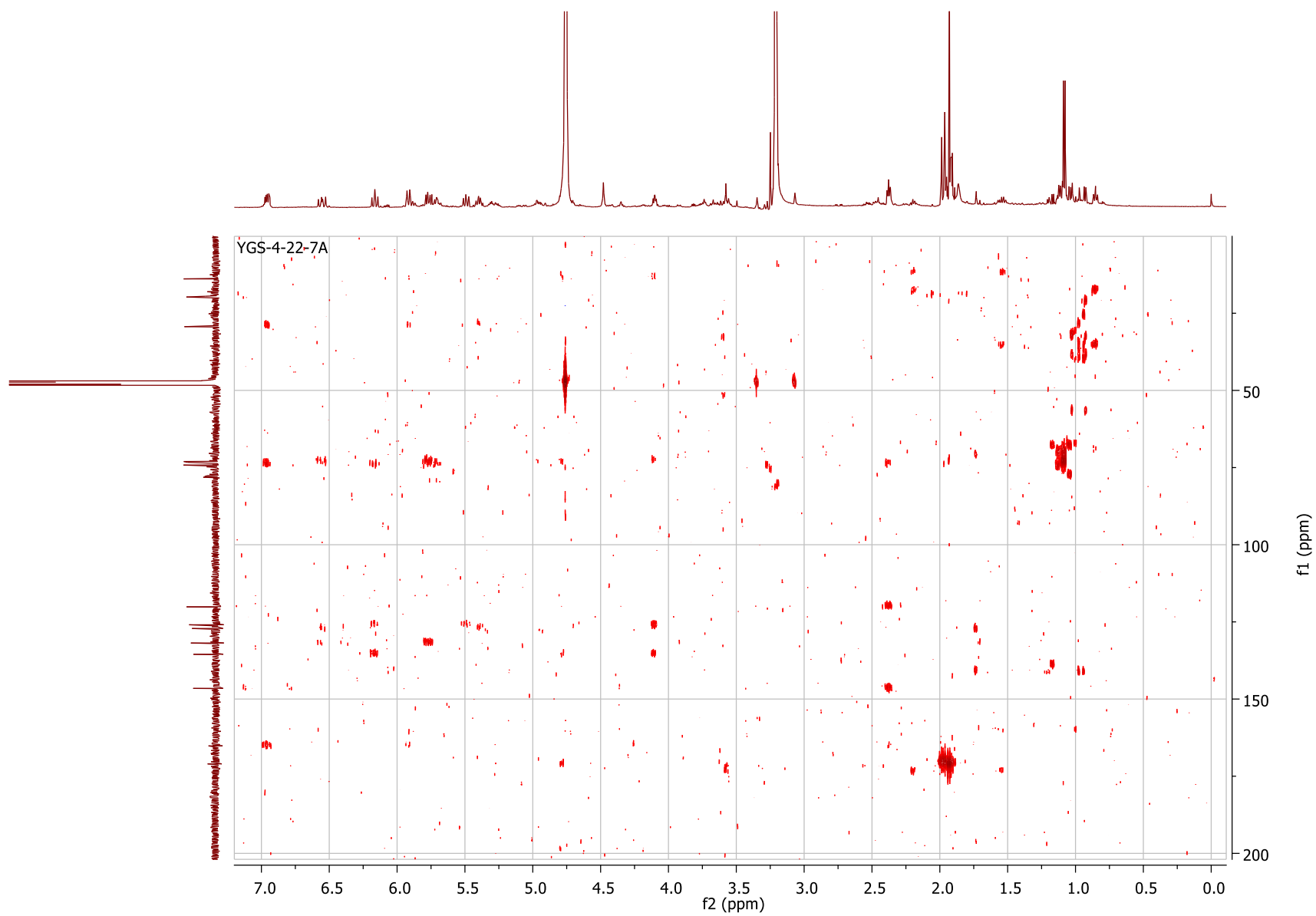
S71: ¹³C NMR of 9



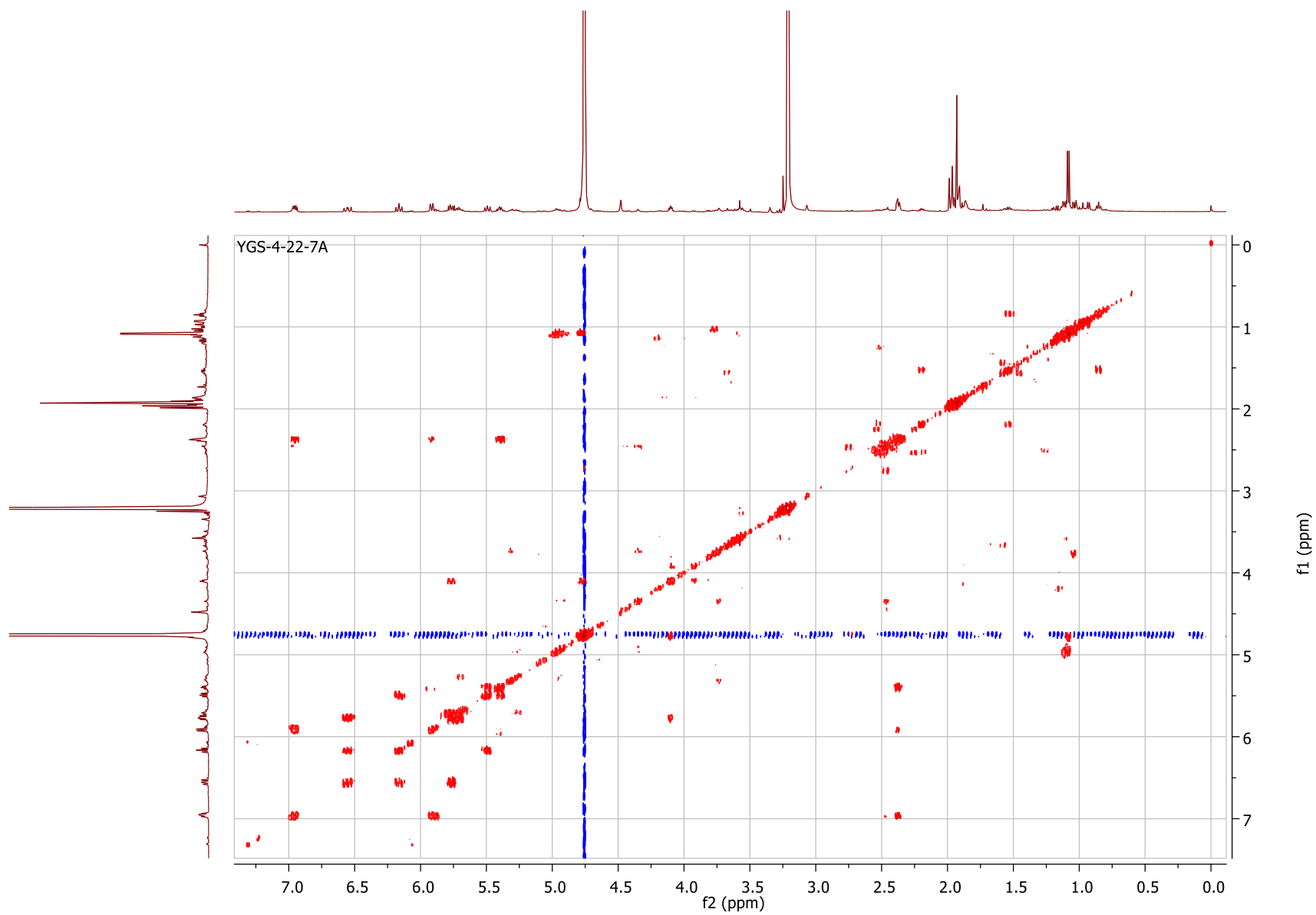
S72: DEPT-135 of 9



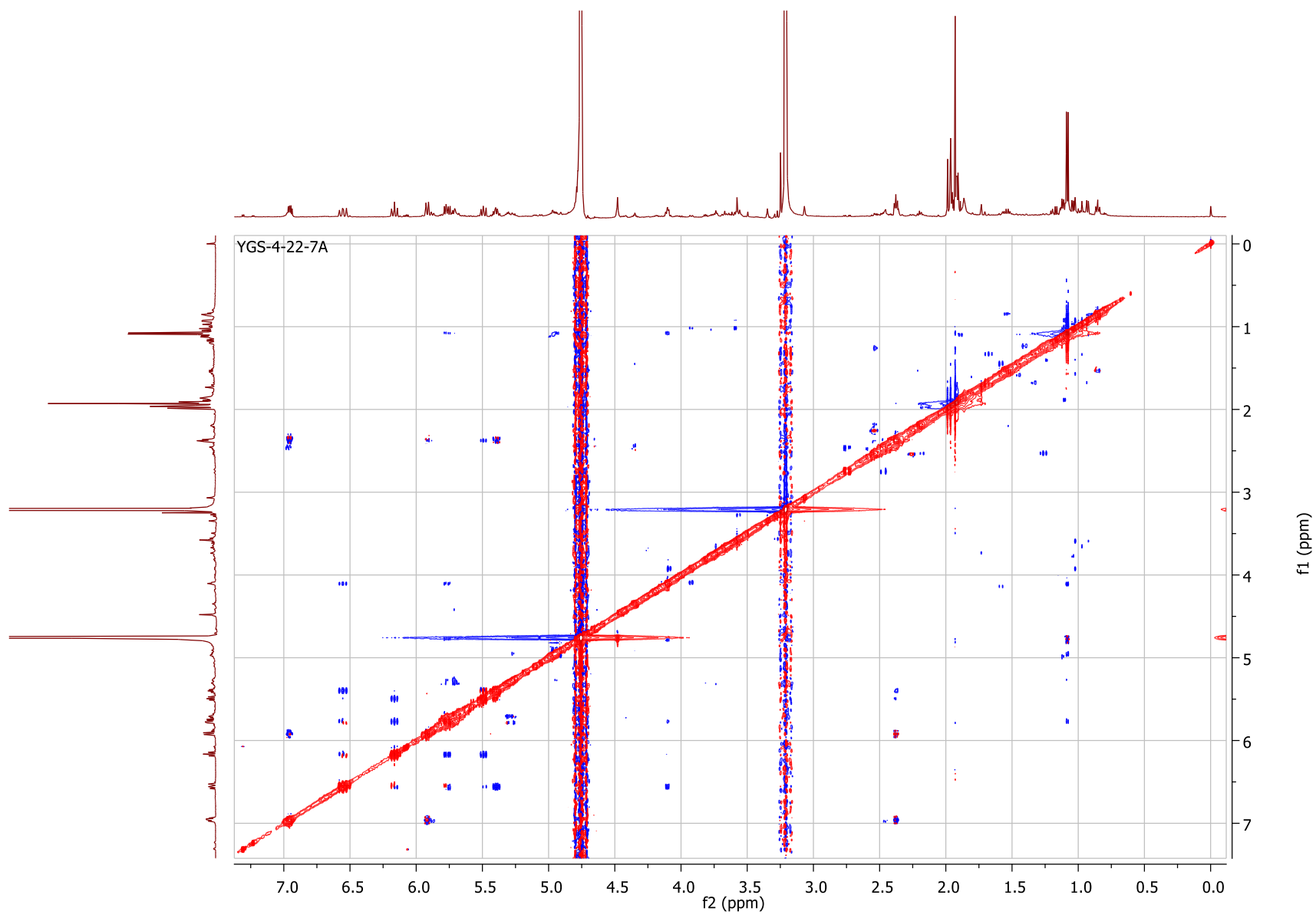
S73: HSQC of **9**



S74: HMBC of 9



S75: ^1H ^1H COSY of **9**

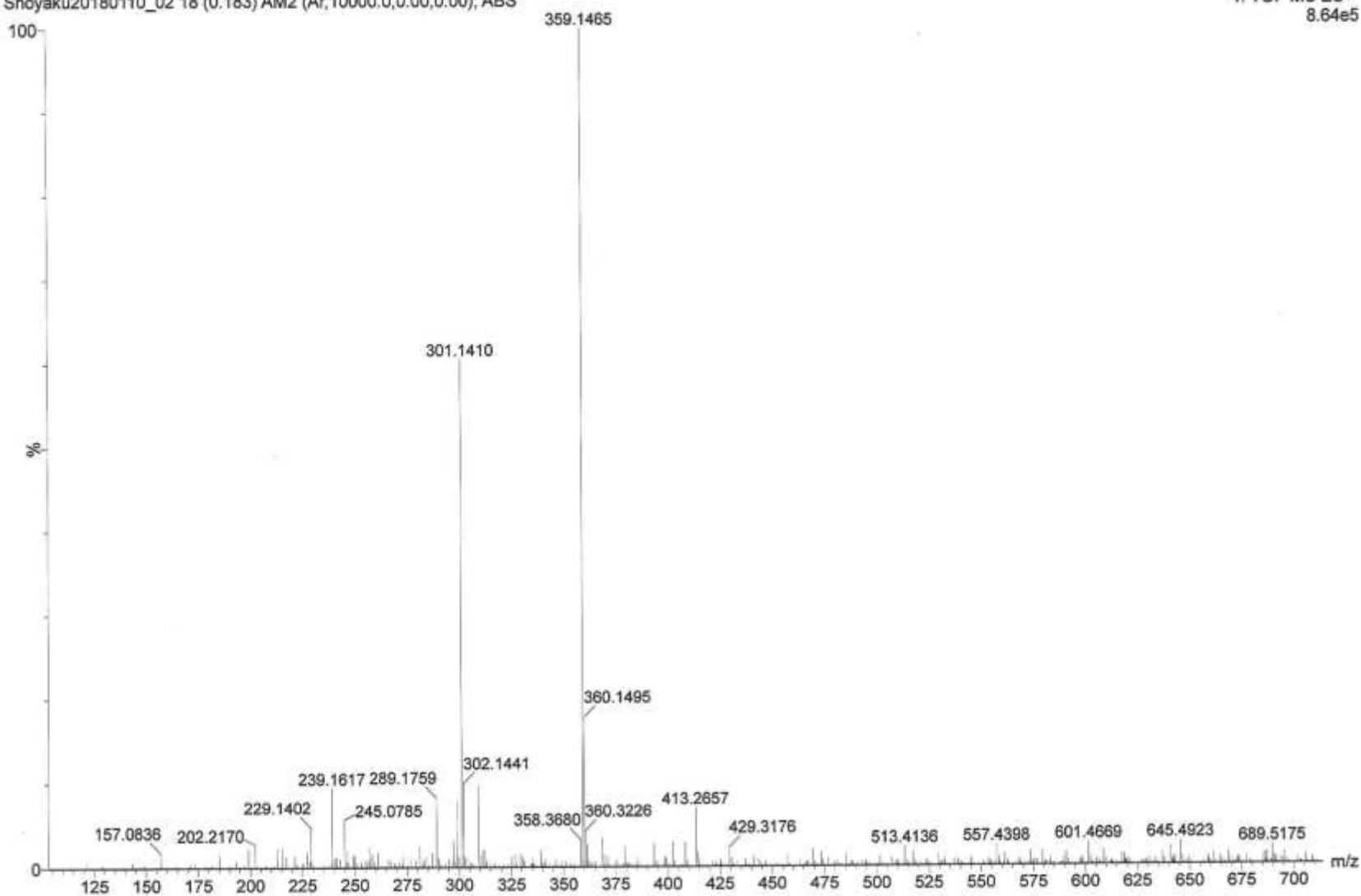


S76: NOESY of 9

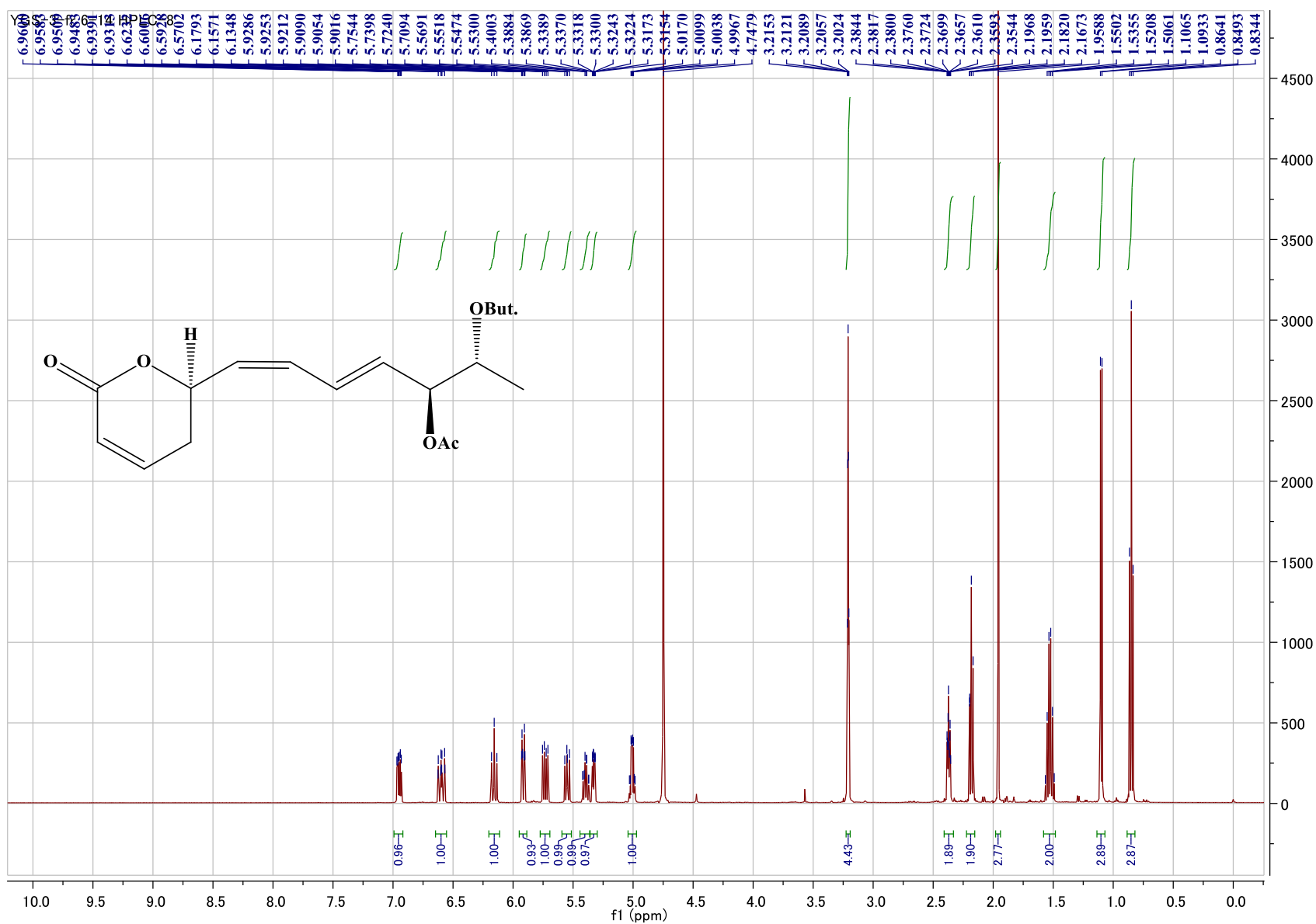
YGS-3 fr6-14 HPLC-8 (Elshamy)

Shoyaku20180110_02 18 (0.183) AM2 (Ar, 10000.0, 0.00, 0.00); ABS

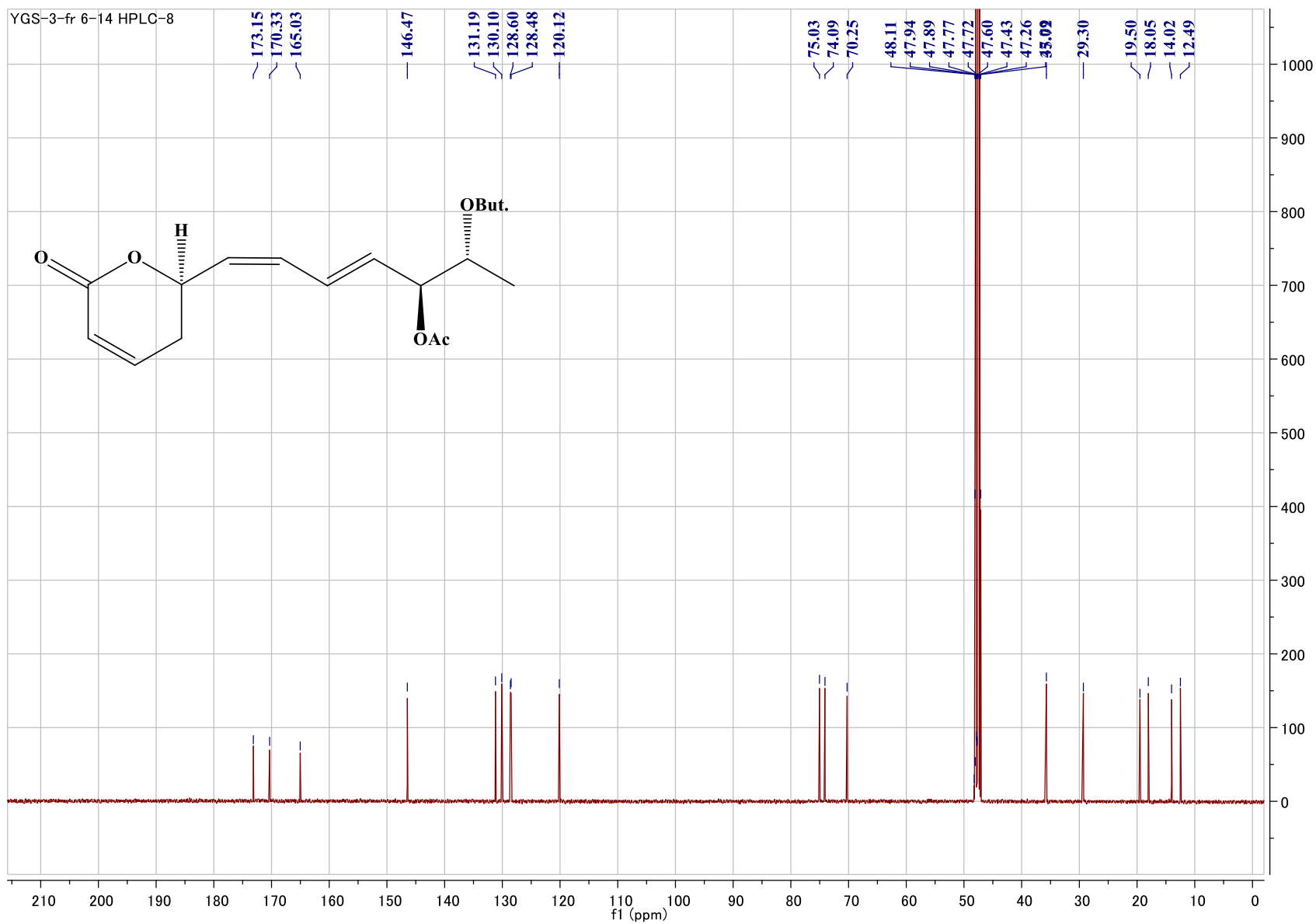
1: TOF MS ES+
8.64e5



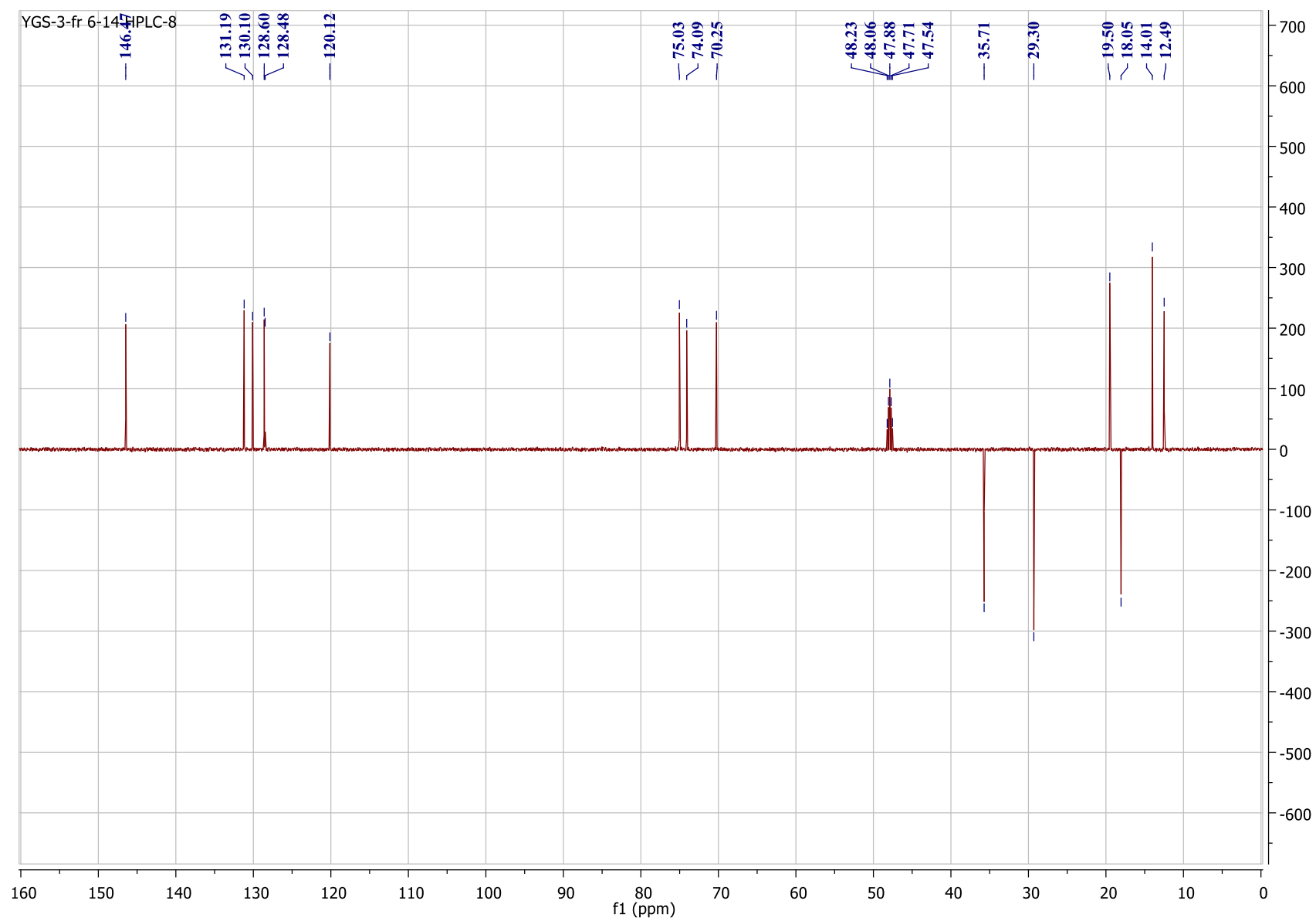
S77: TOFESIMS of 10



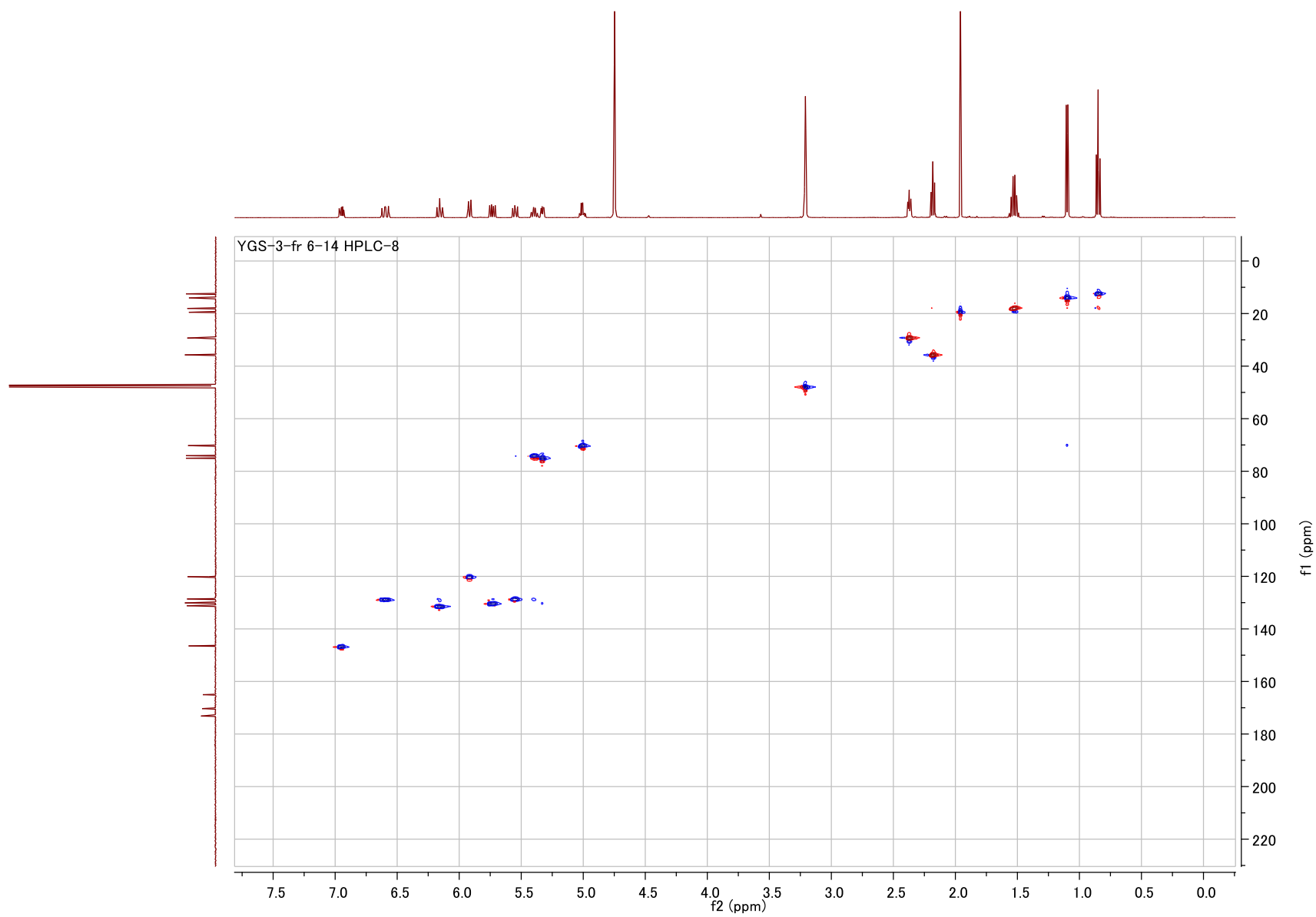
S78: ¹H NMR of 10



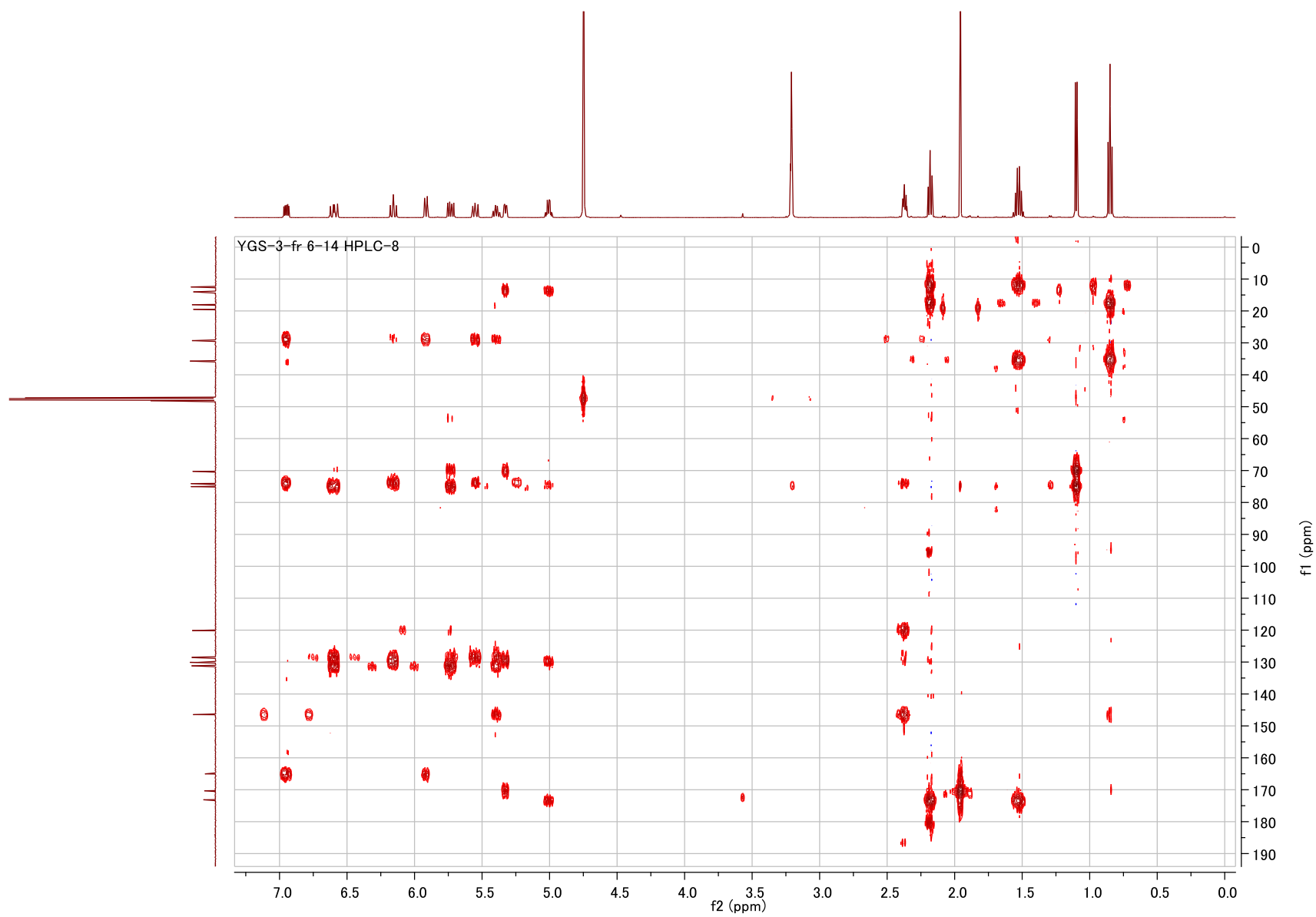
S79: ¹³C NMR of 10



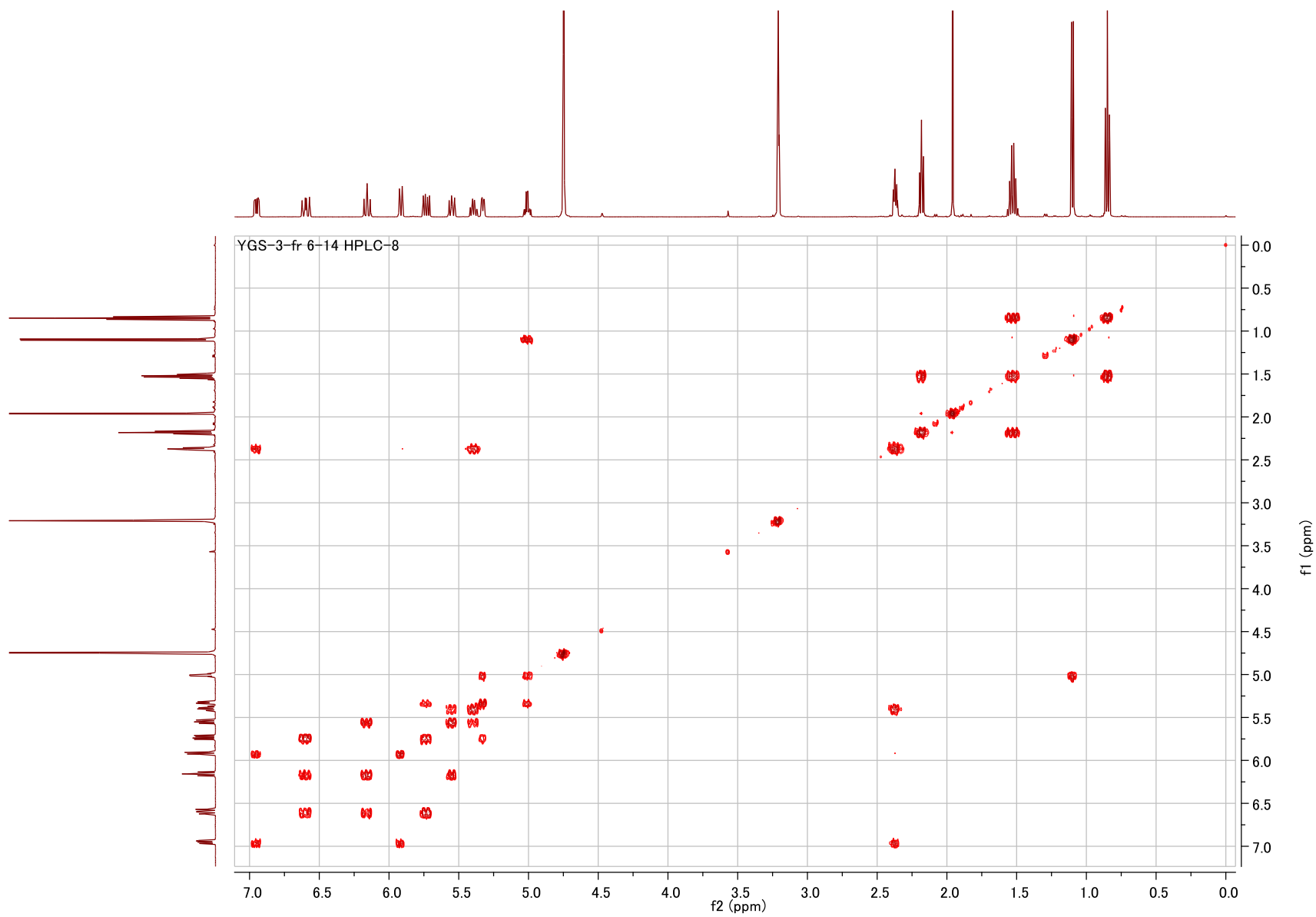
S80: DEPT-135 of 10



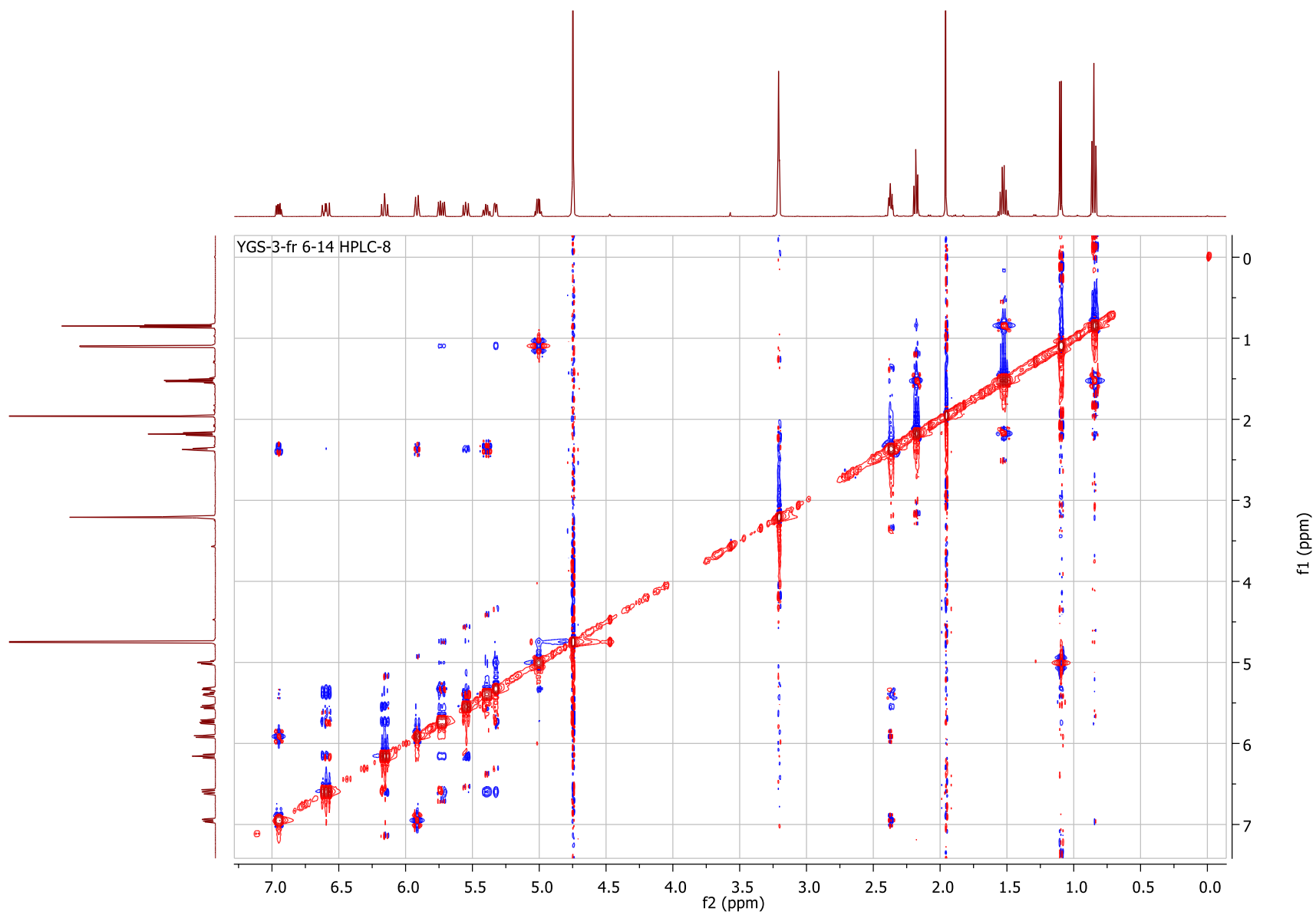
S81: HSQC of 10



S82: HMBC of 10

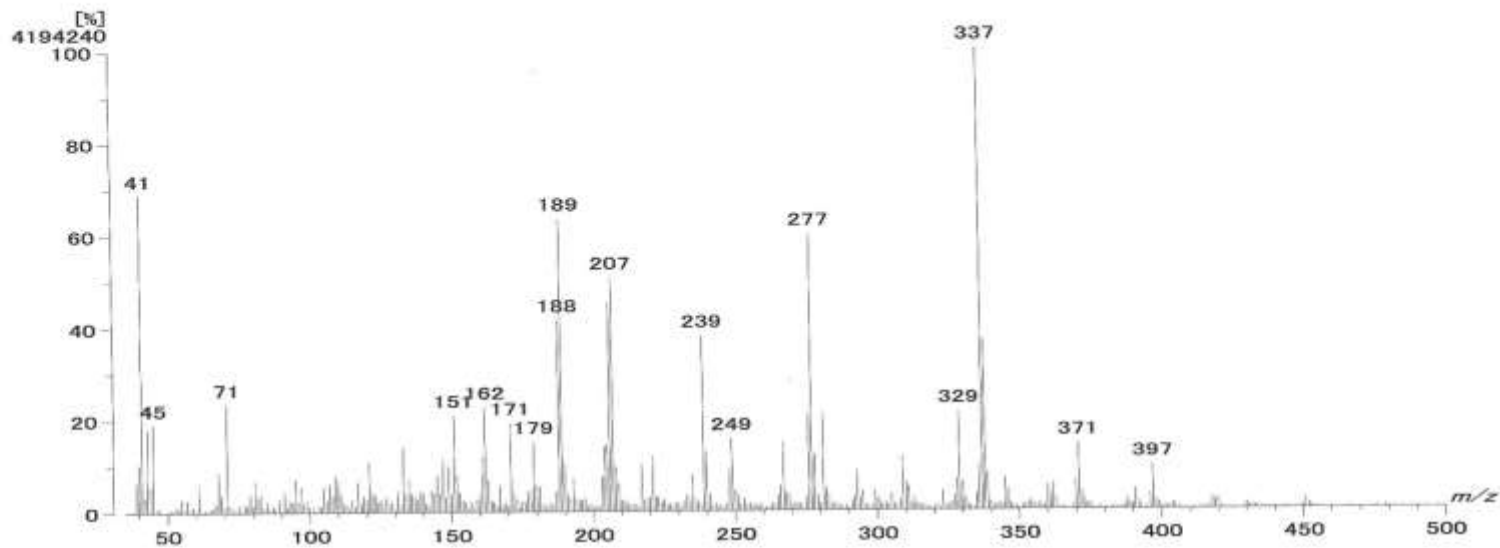


S83: ^1H ^1H COSY of **10**



S84: NOESY of 10

[Mass Spectrum]
 Data : Umeyama-CI.22-Feb-2018.001 Date : 22-Feb-2018 10:37
 Sample : YGS-6-5A(OH4)
 Note : MStation
 Inlet : Direct Ion Mode : CI+
 Spectrum Type : Normal Ion [MF-Linear]
 RT : 1.15 min Scan# : 43
 BP : m/z 337 Int. : 399.99 (4194240)
 Output m/z range : 35 to 500 Cut Level : 0.00 %

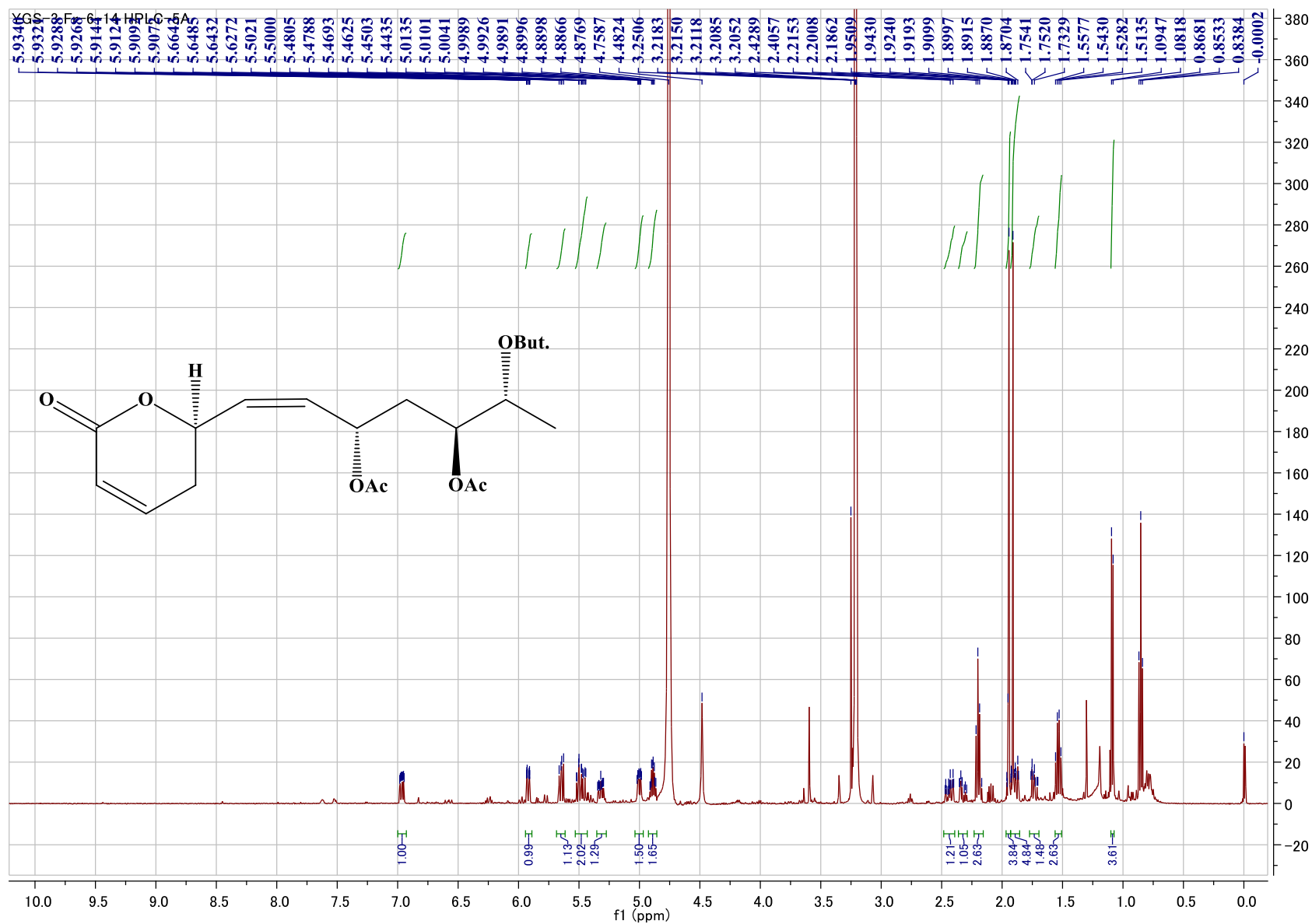


S85: LRCIMS of 11

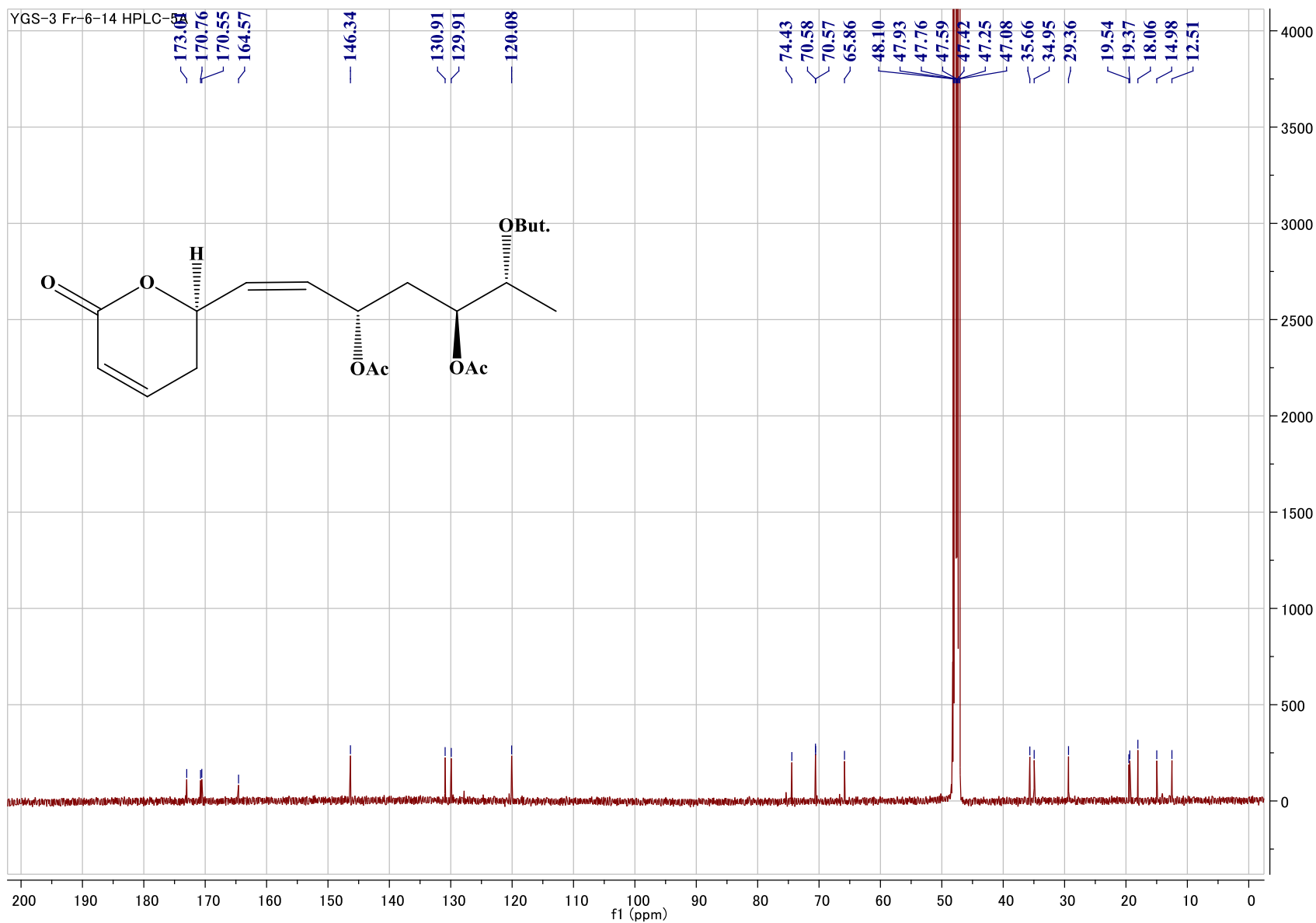
Data : Umeyama-CIHR.22-Feb-2018.003 Date : 22-Feb-2018 19:13
 Instrument : MStation
 Sample : YGS-6-5A
 Note : MStation
 Inlet : Direct Ion Mode : CI+
 RT : 0.54 min Scan# : 15
 Elements : C 150/0, H 250/0, O 50/0
 Mass Tolerance : 5mmu
 Unsaturation (U.S.) : 0.0 - 15.0

	Observed m/z	Int%	Err [ppm / mmu]	U.S. Composition
1	397.1871	2.06	+2.2 / +0.9	6.5 C20 H29 O8

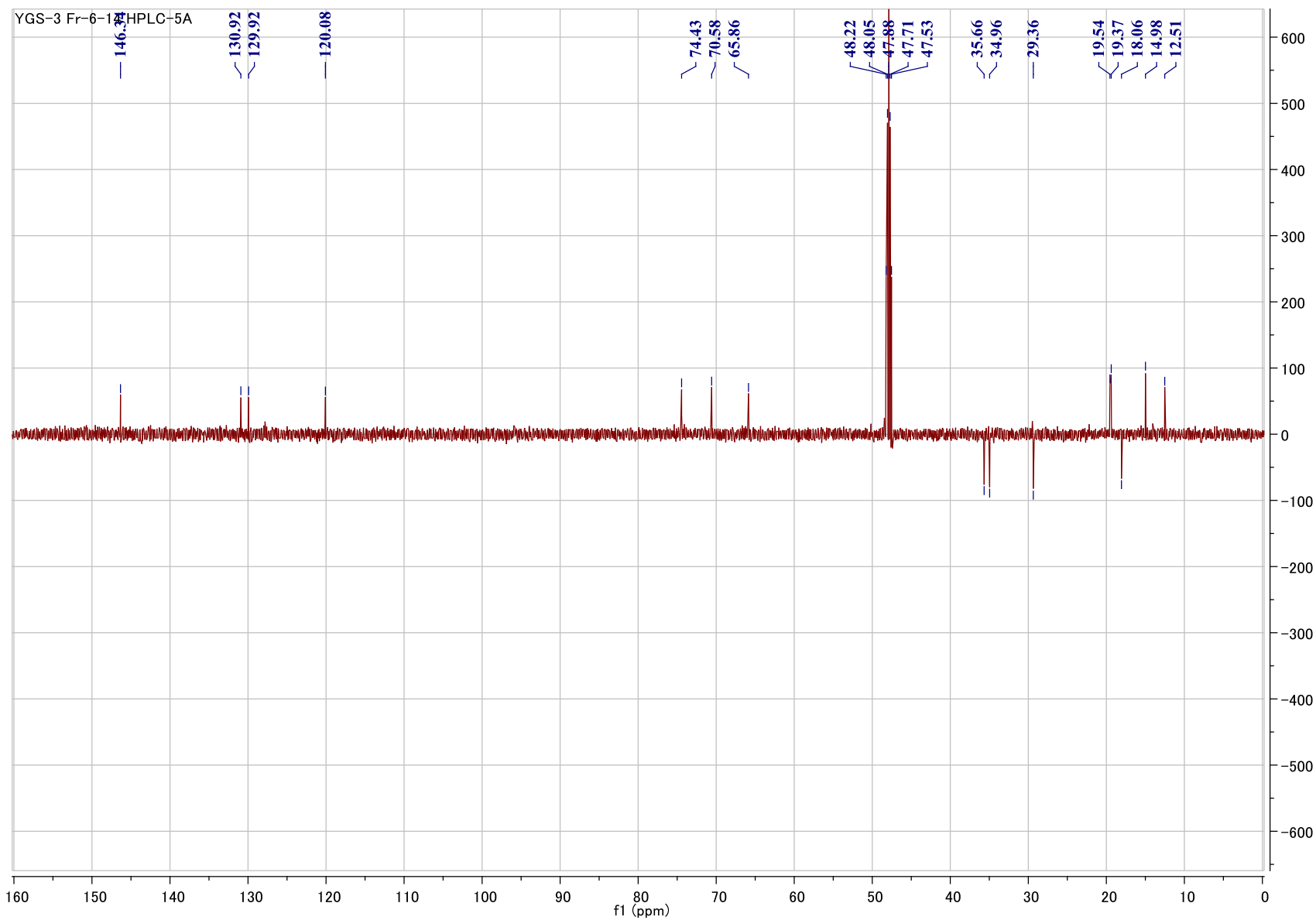
S86: HRCIMS of 11



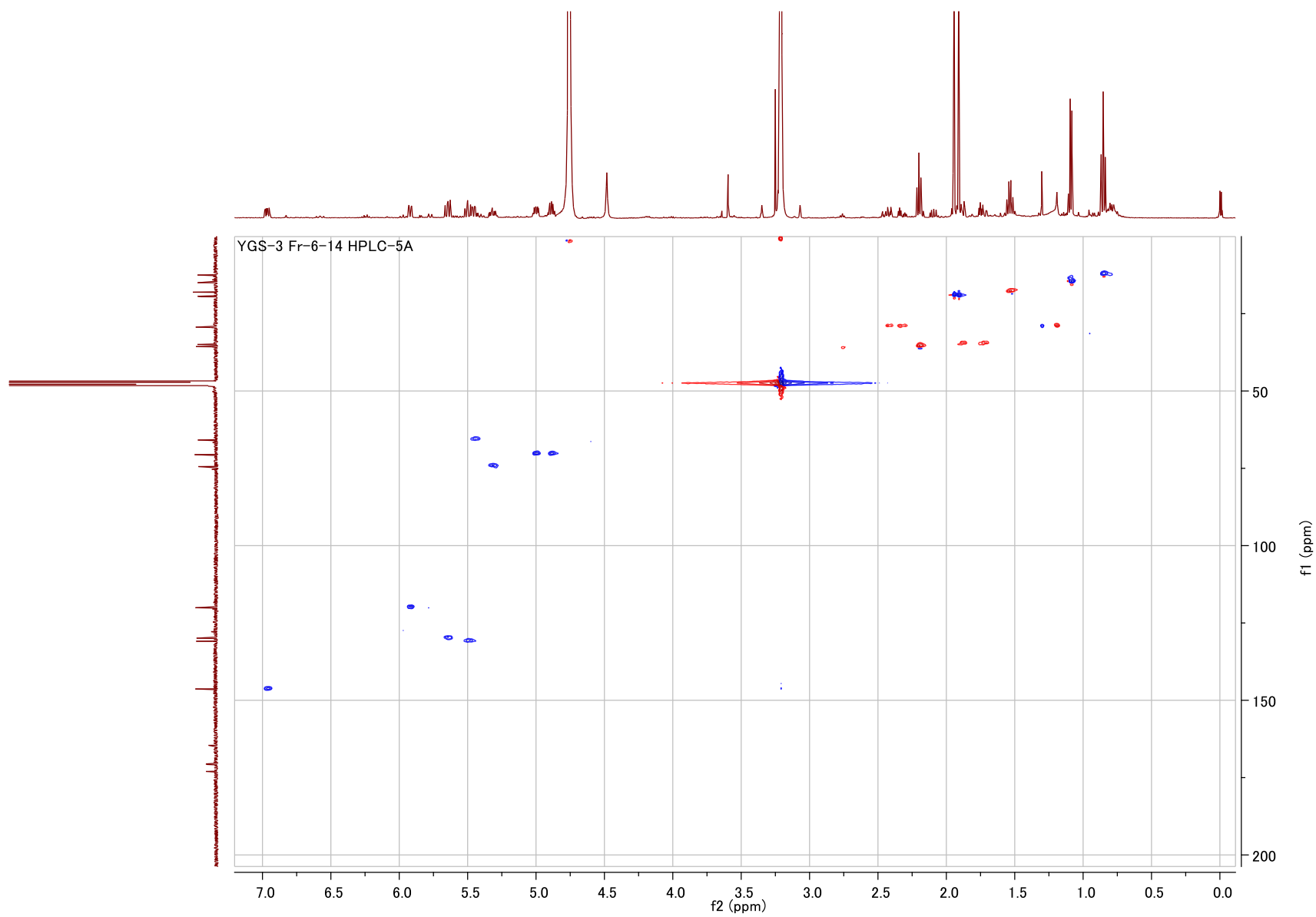
S87: ¹H NMR of 11



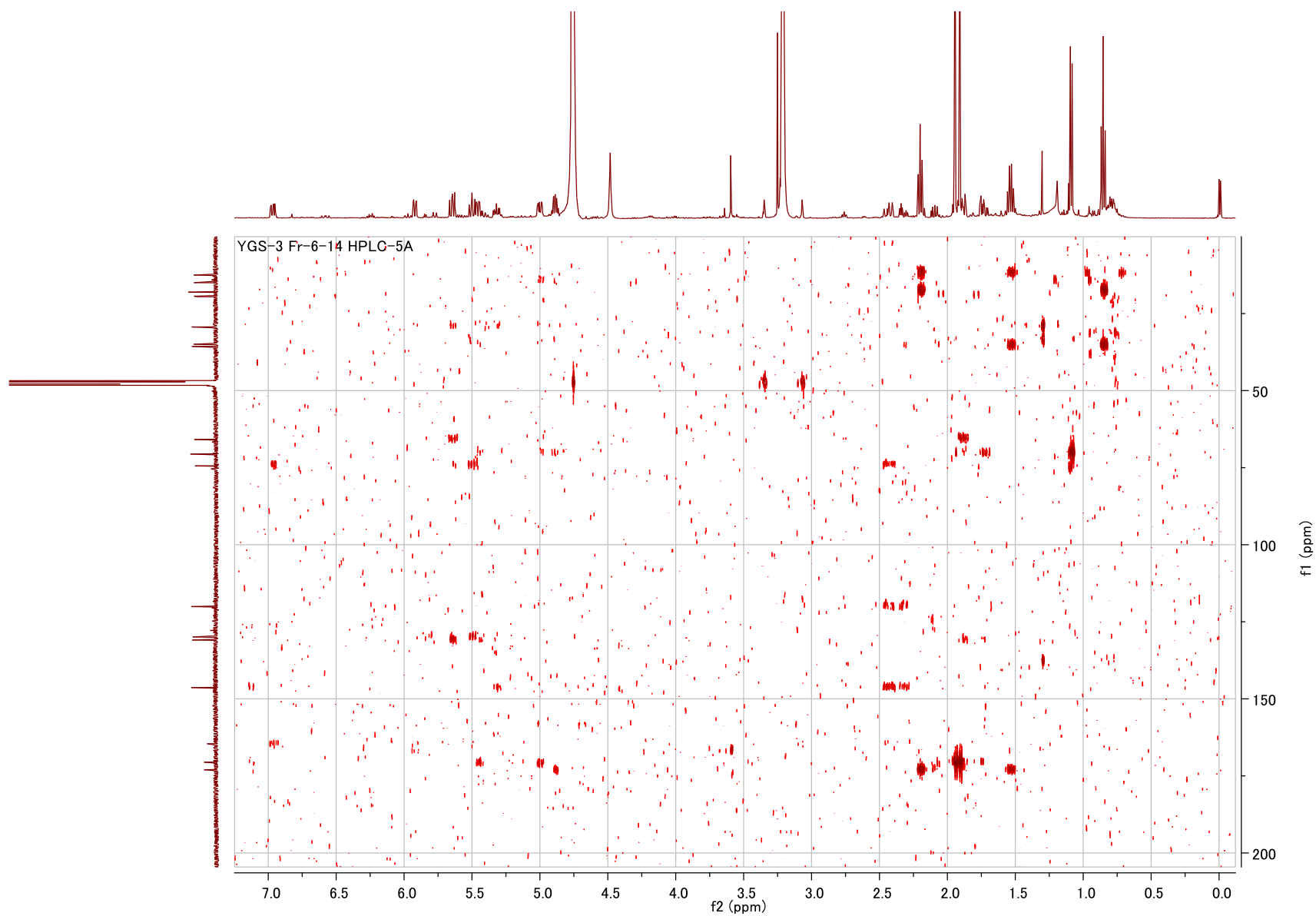
S88: ^{13}C NMR of 11



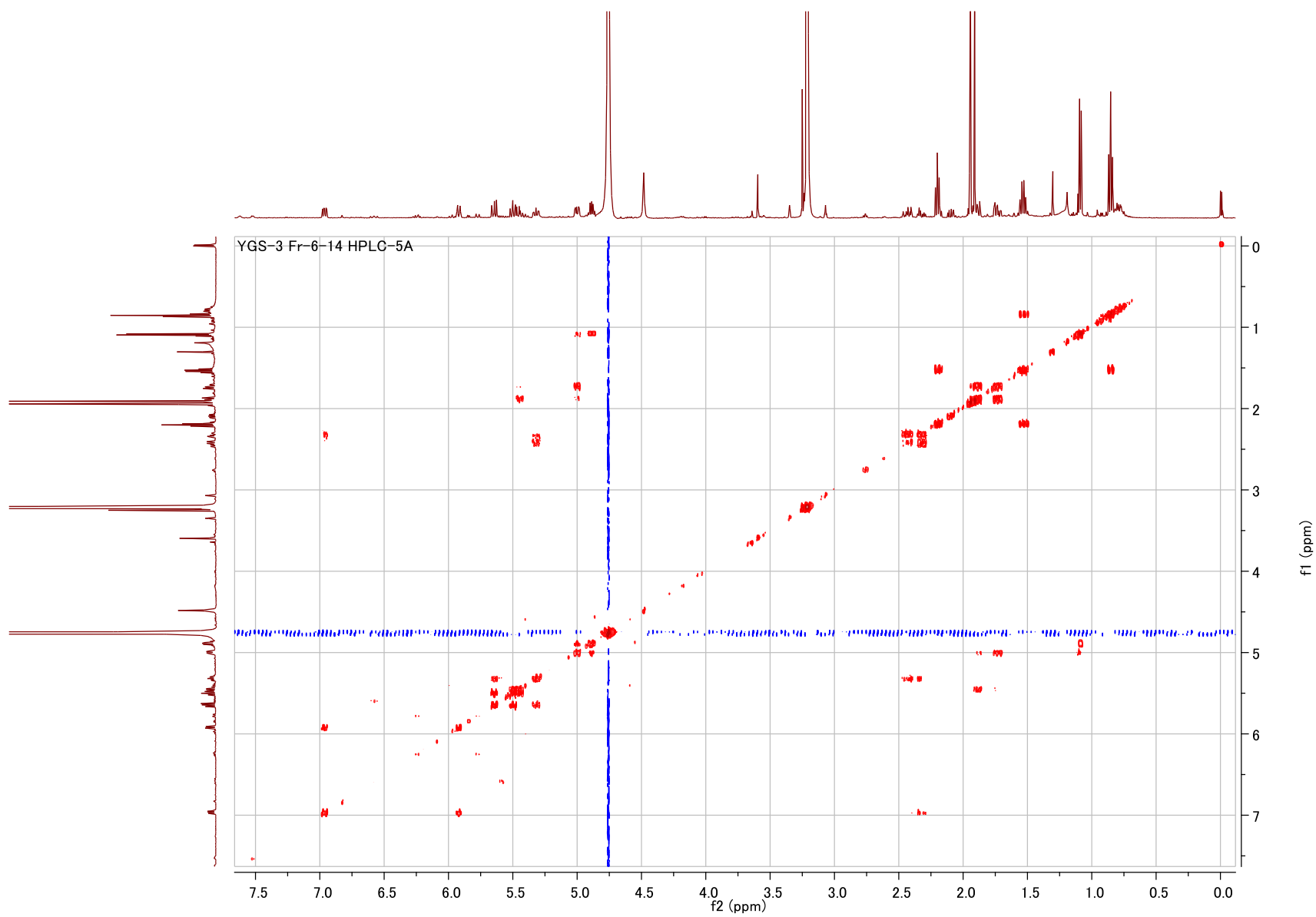
S89: DEPT-135 of 11



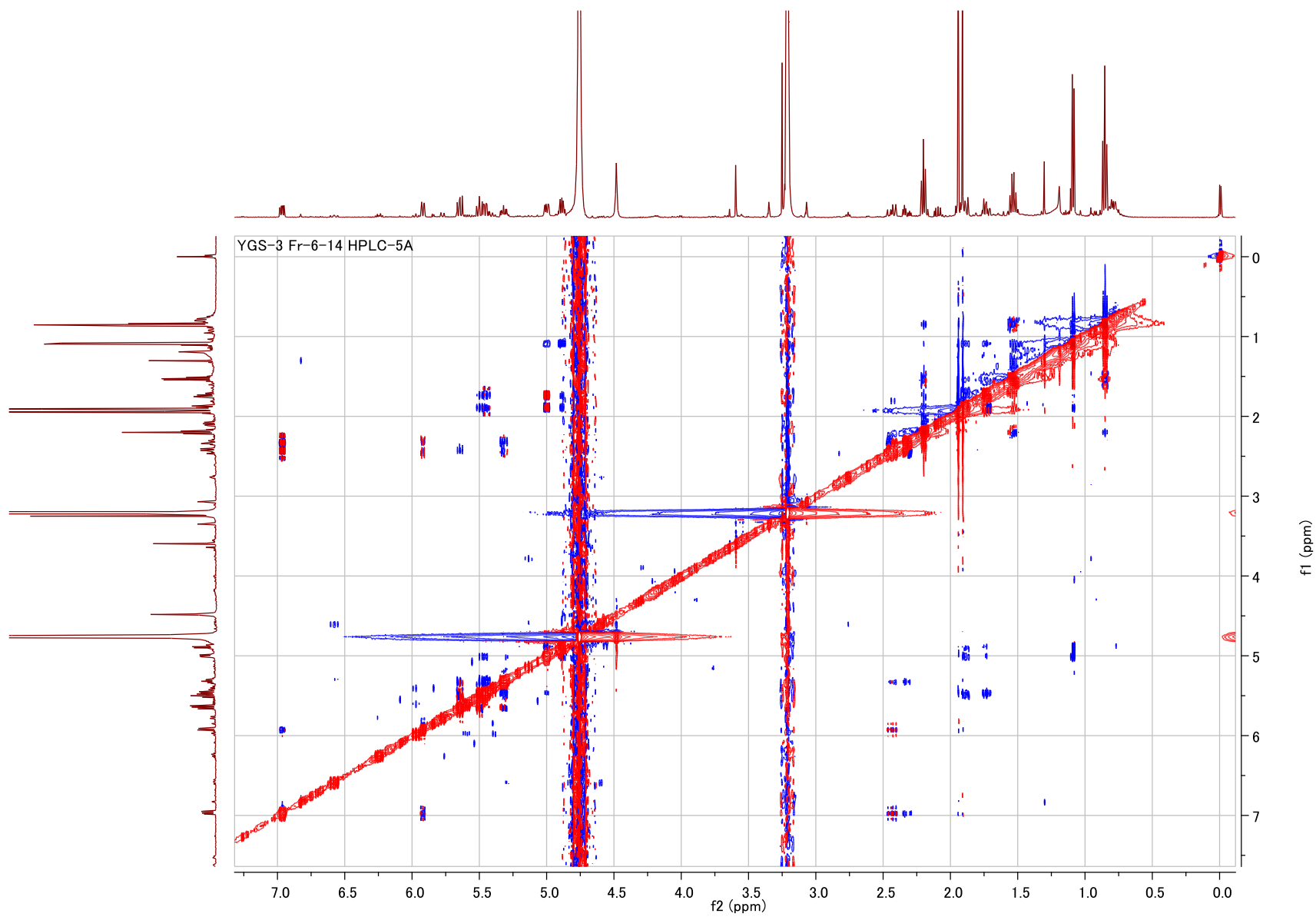
S90: HSQC of 11



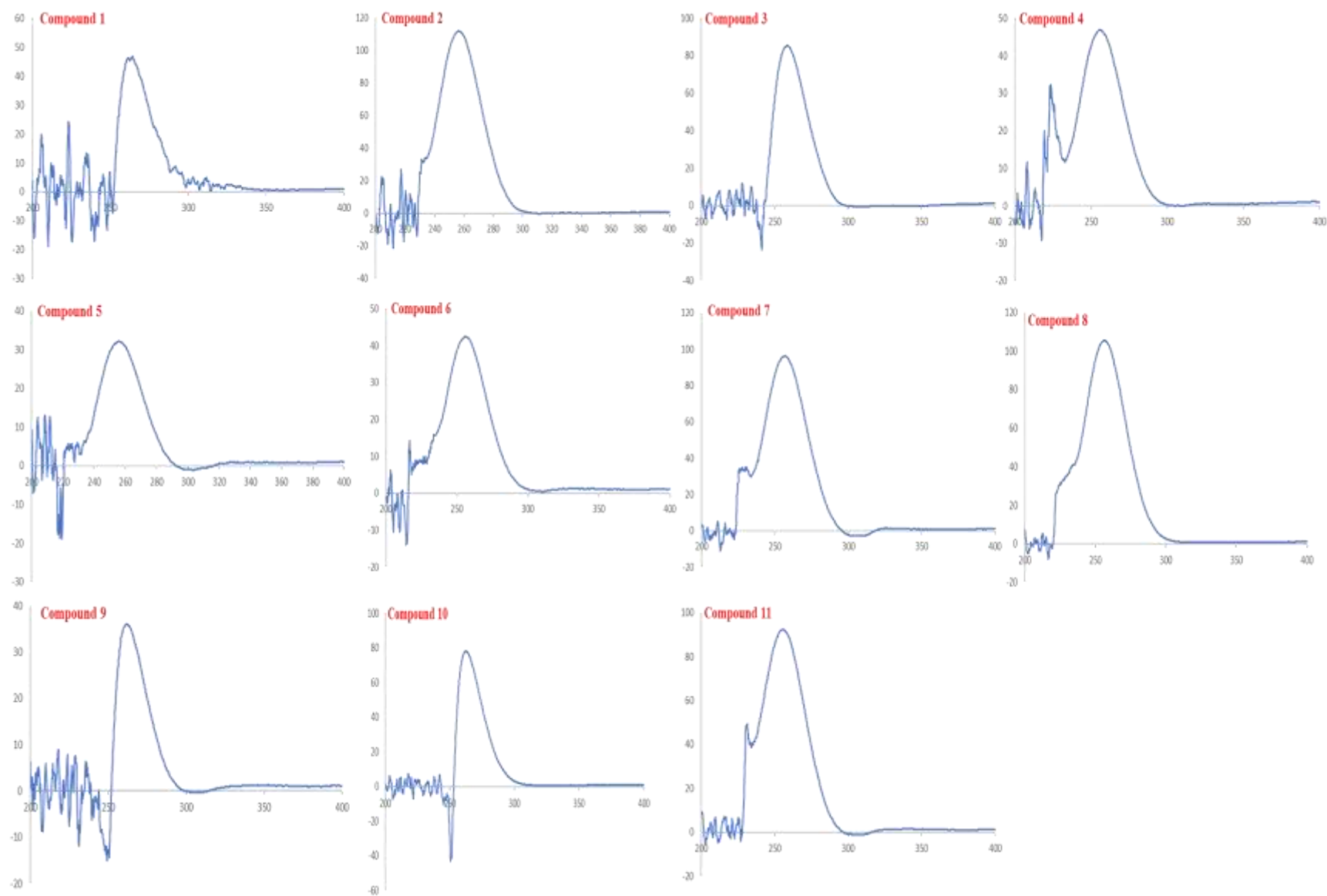
S91: HMBC of 11



S92: ^1H ^1H COSY of **11**



S93: NOESY of 11



S94: Experimental ECD of isolates 1-11