BEILSTEIN JOURNAL OF ORGANIC CHEMISTRY

Supporting Information

for

Synthesis of multiply fluorinated *N*-acetyl-D-glucosamine and D-galactosamine analogs via the corresponding deoxyfluorinated glucosazide and galactosazide phenyl thioglycosides

Vojtěch Hamala, Lucie Červenková Šťastná, Martin Kurfiřt, Petra Cuřínová, Martin Dračínský and Jindřich Karban

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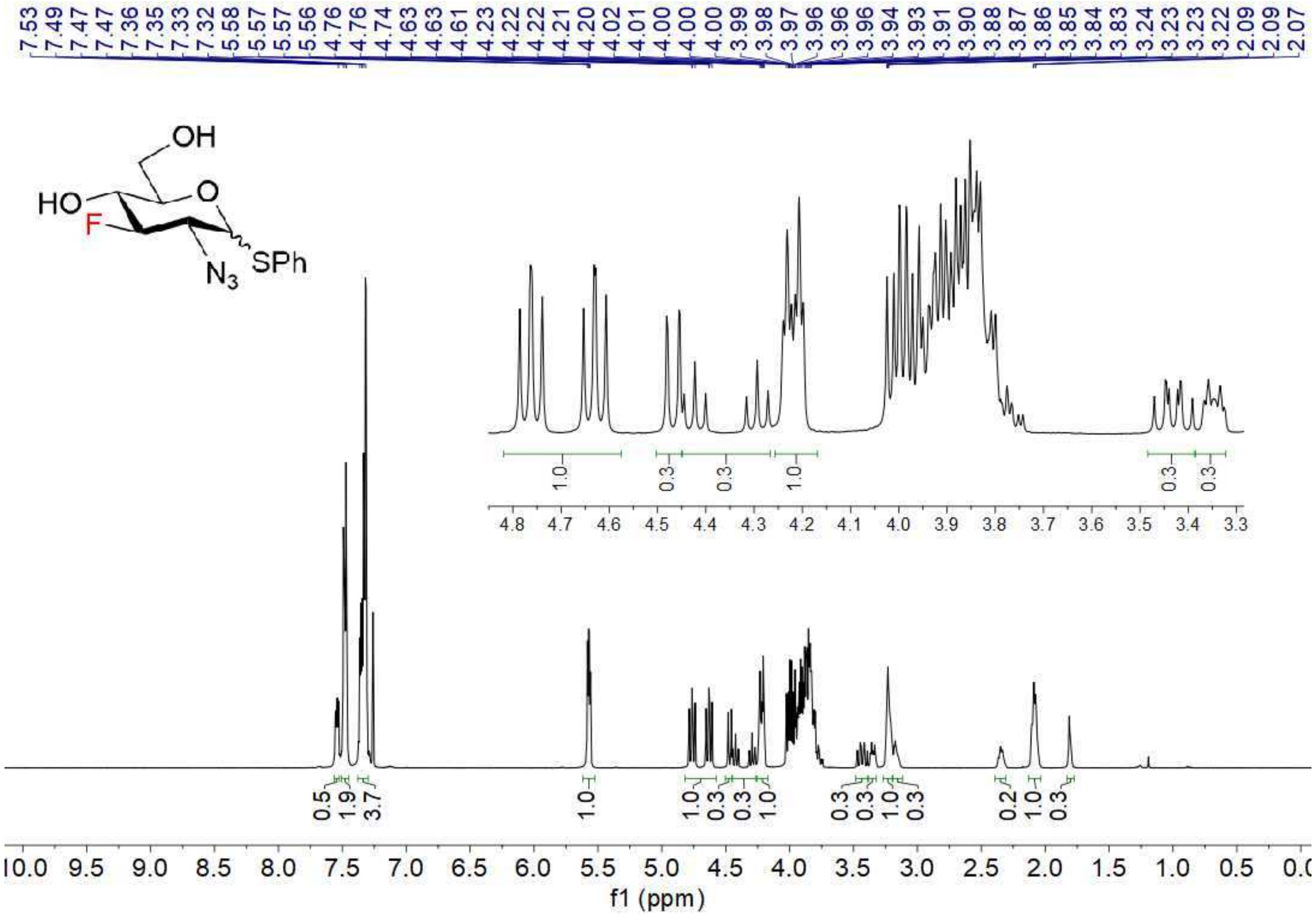
Copies of ^{1}H, ^{13}C, ^{19}F, and 2D NMR spectra for new compounds

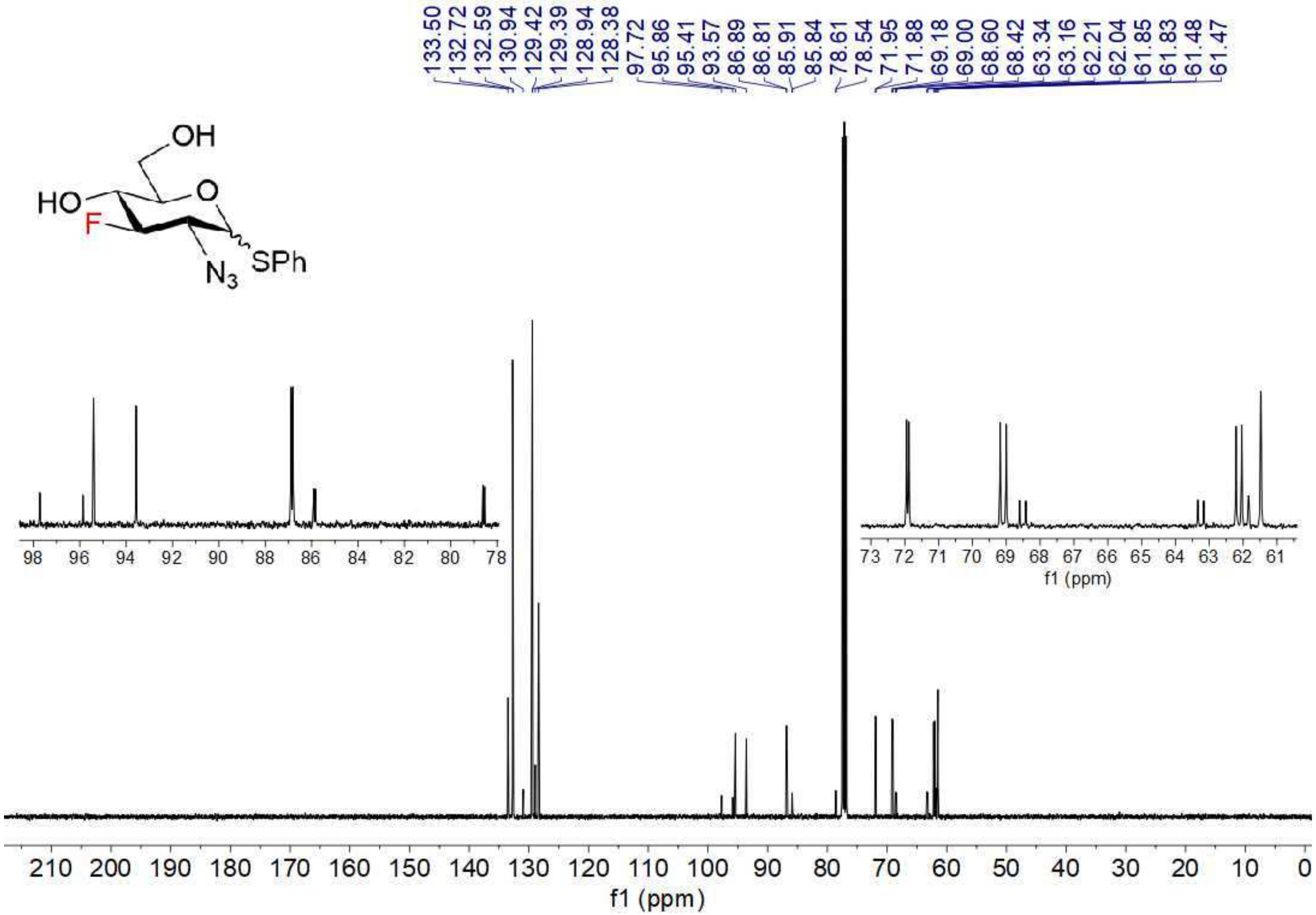
Table of Contents

NMR COMPOUND 18

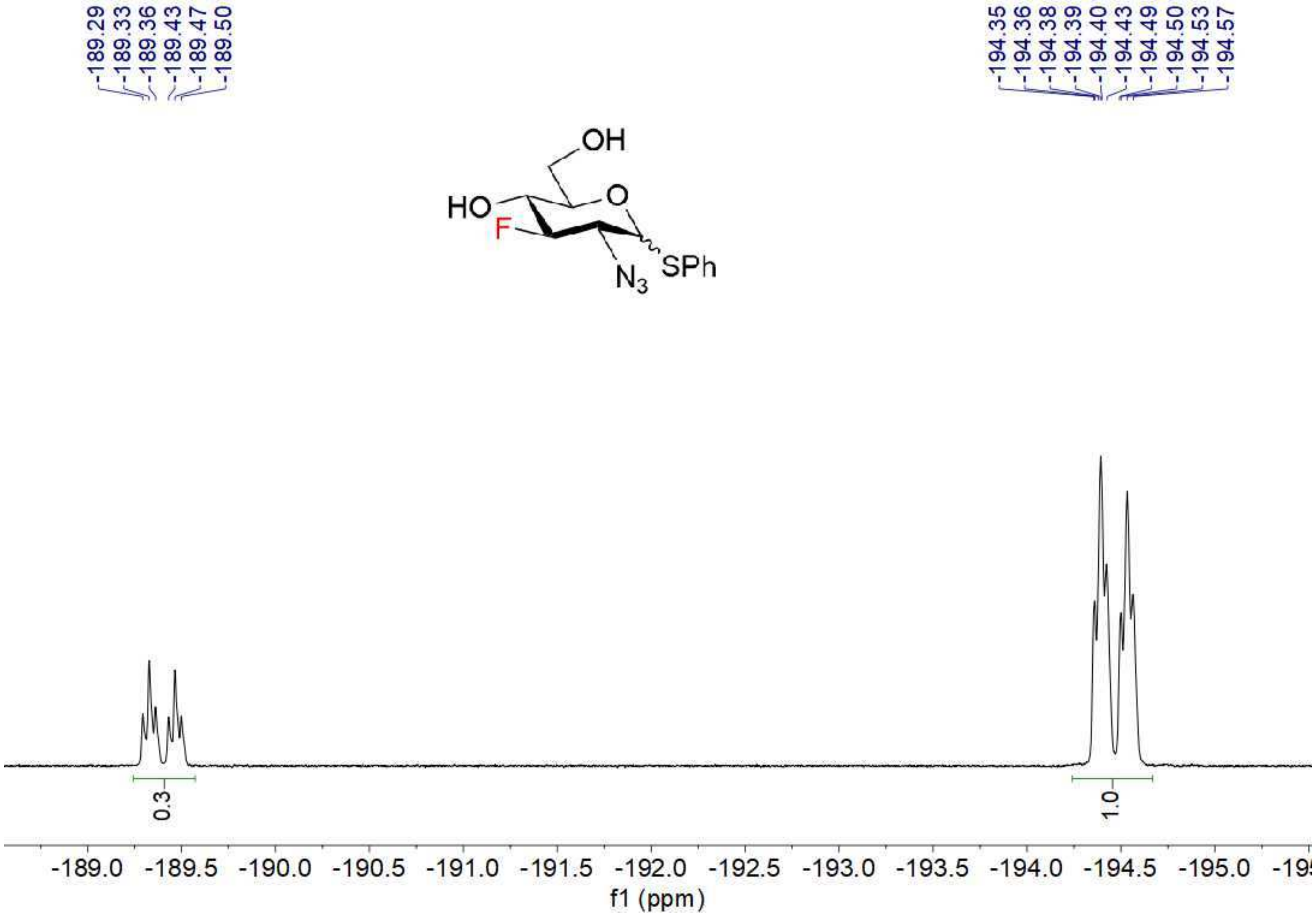
^{1}H NMR (400 MHz, $CDCl_3$) 18 (α/β ca. 10/3)



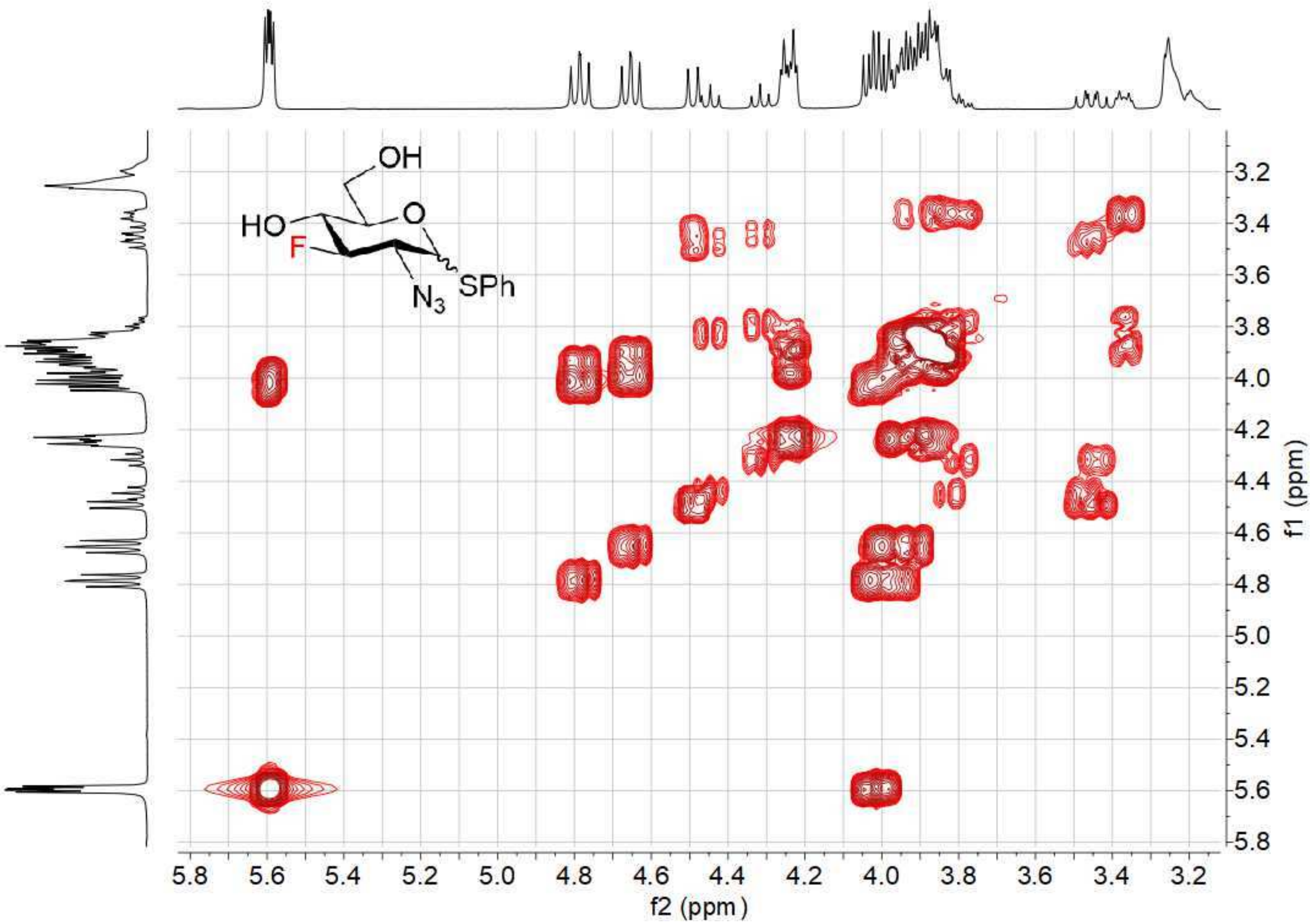
^{13}C NMR (100 MHz, CDCl$_3$) 18



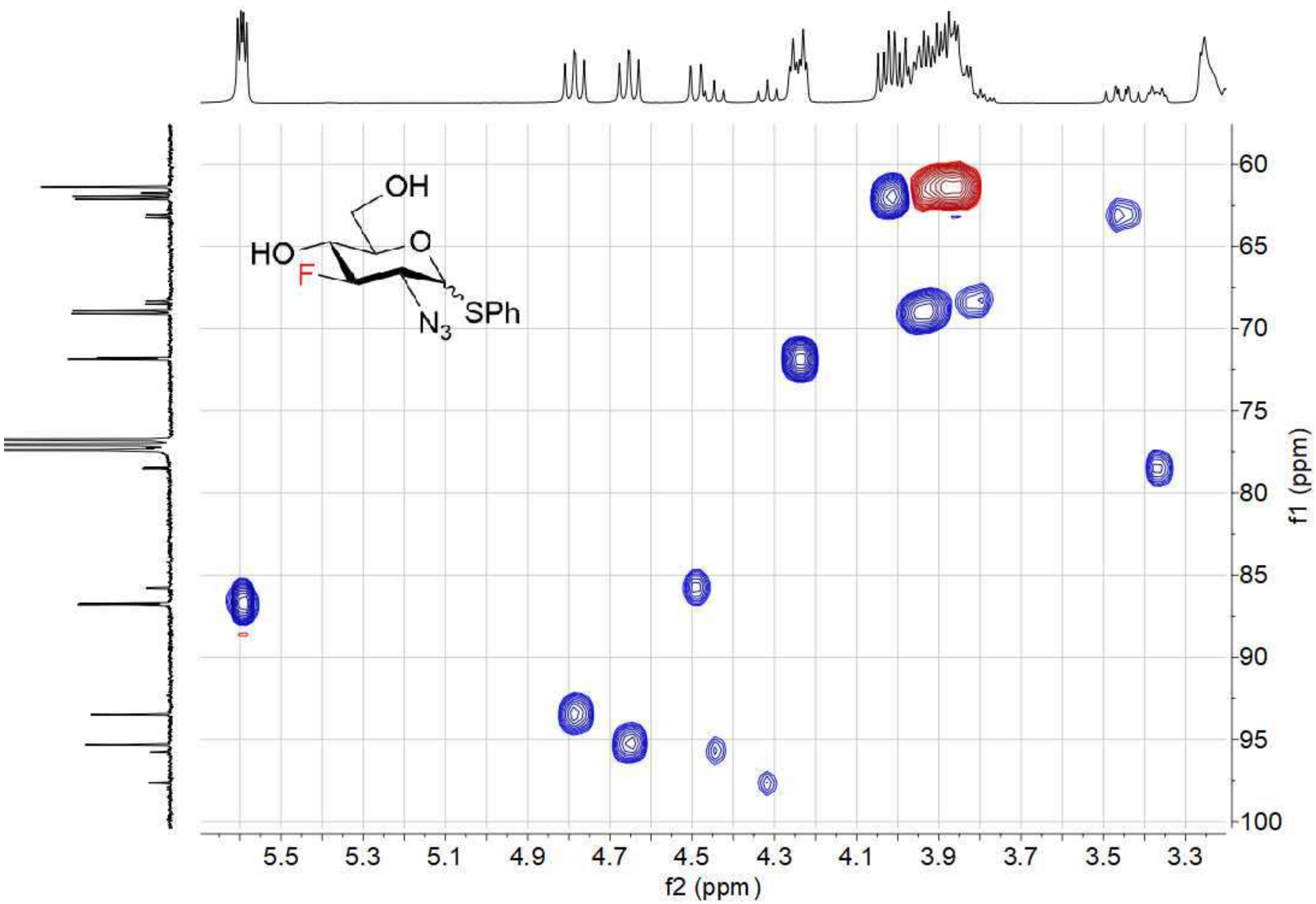
^{19}F NMR (376 MHz, $CDCl_3$) 18 (α/β ca. 10/3)



^{1}H-^{1}H COSY 18

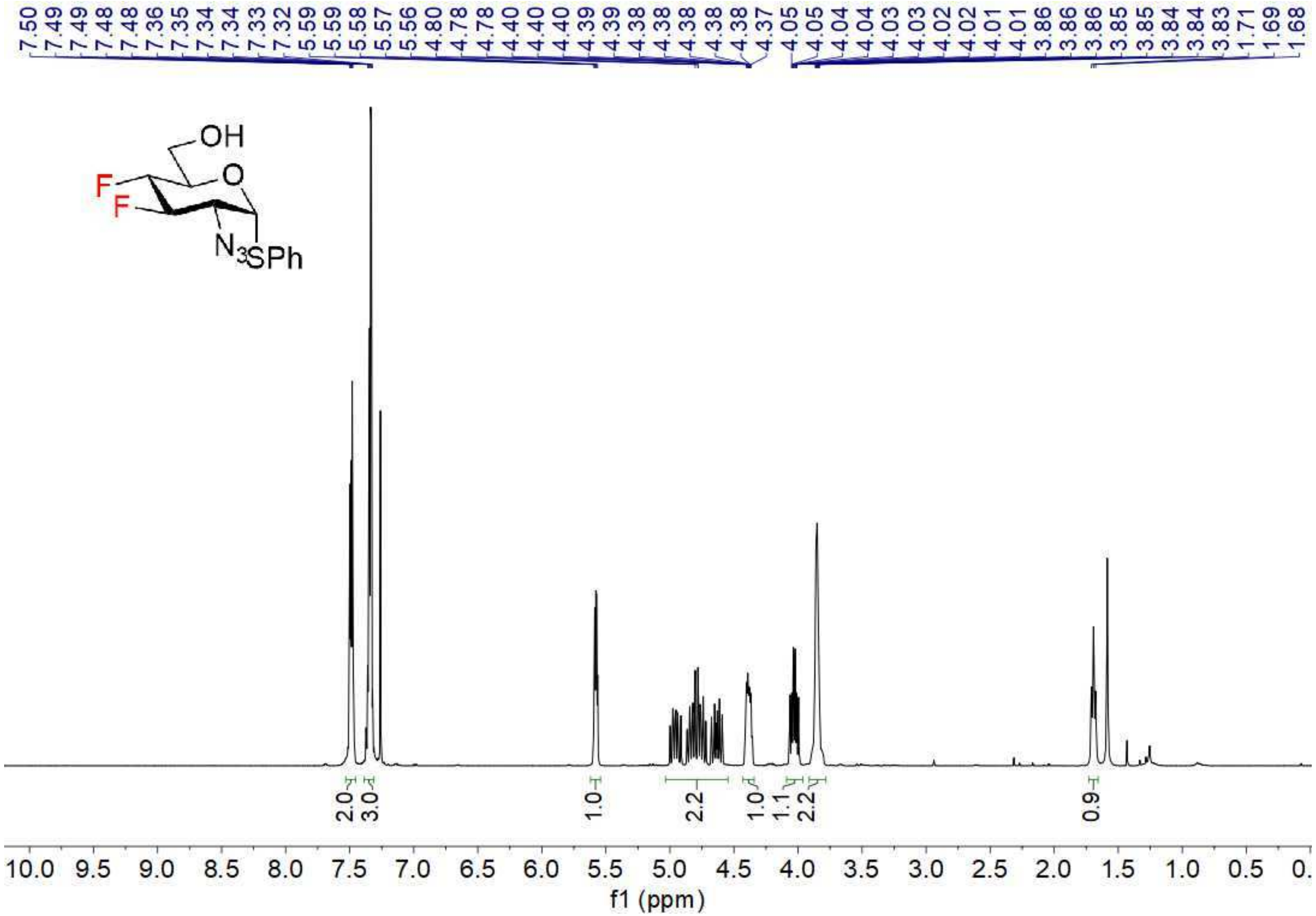


^{1}H-^{13}C HSQC 18

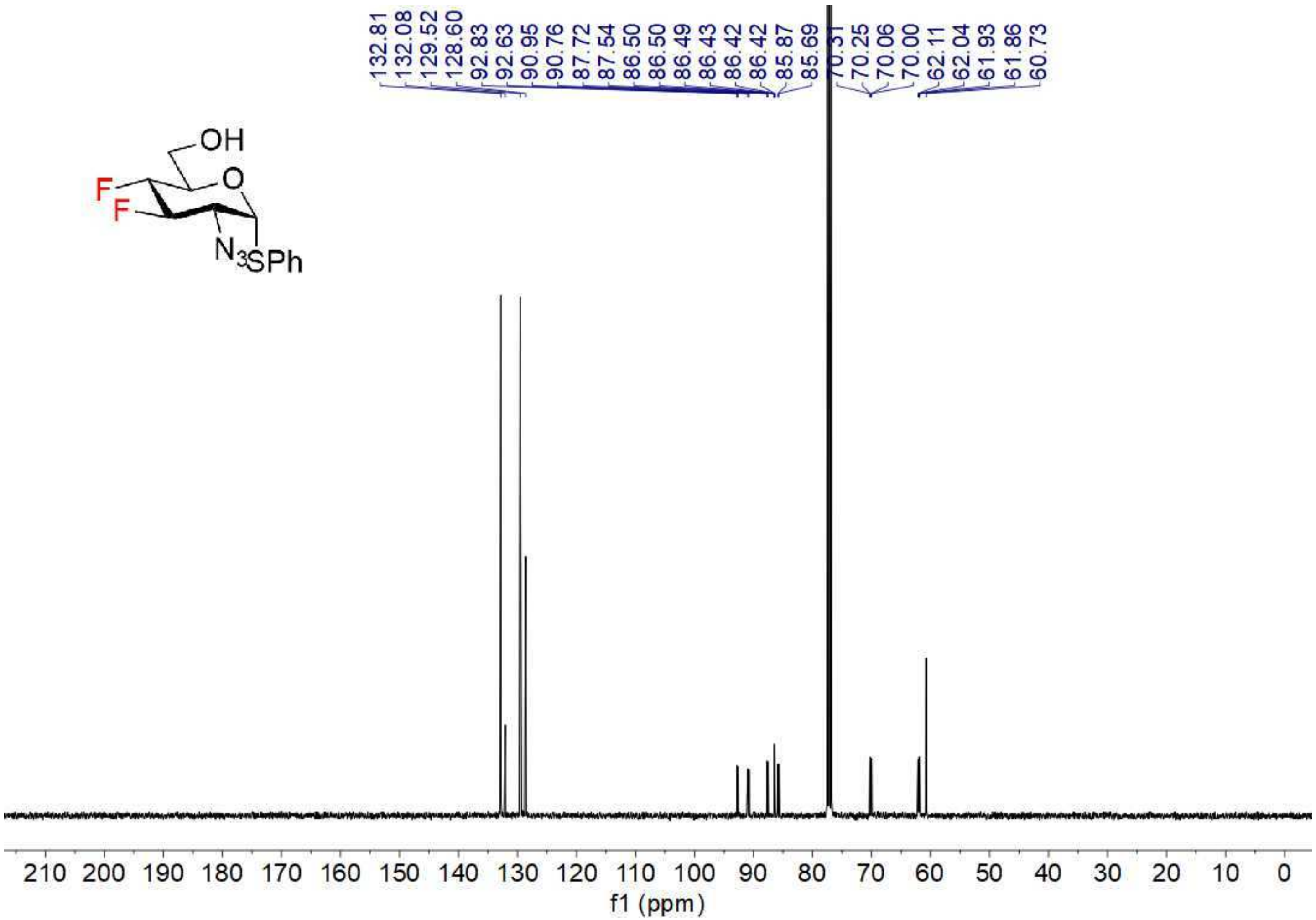


NMR COMPOUND α-19

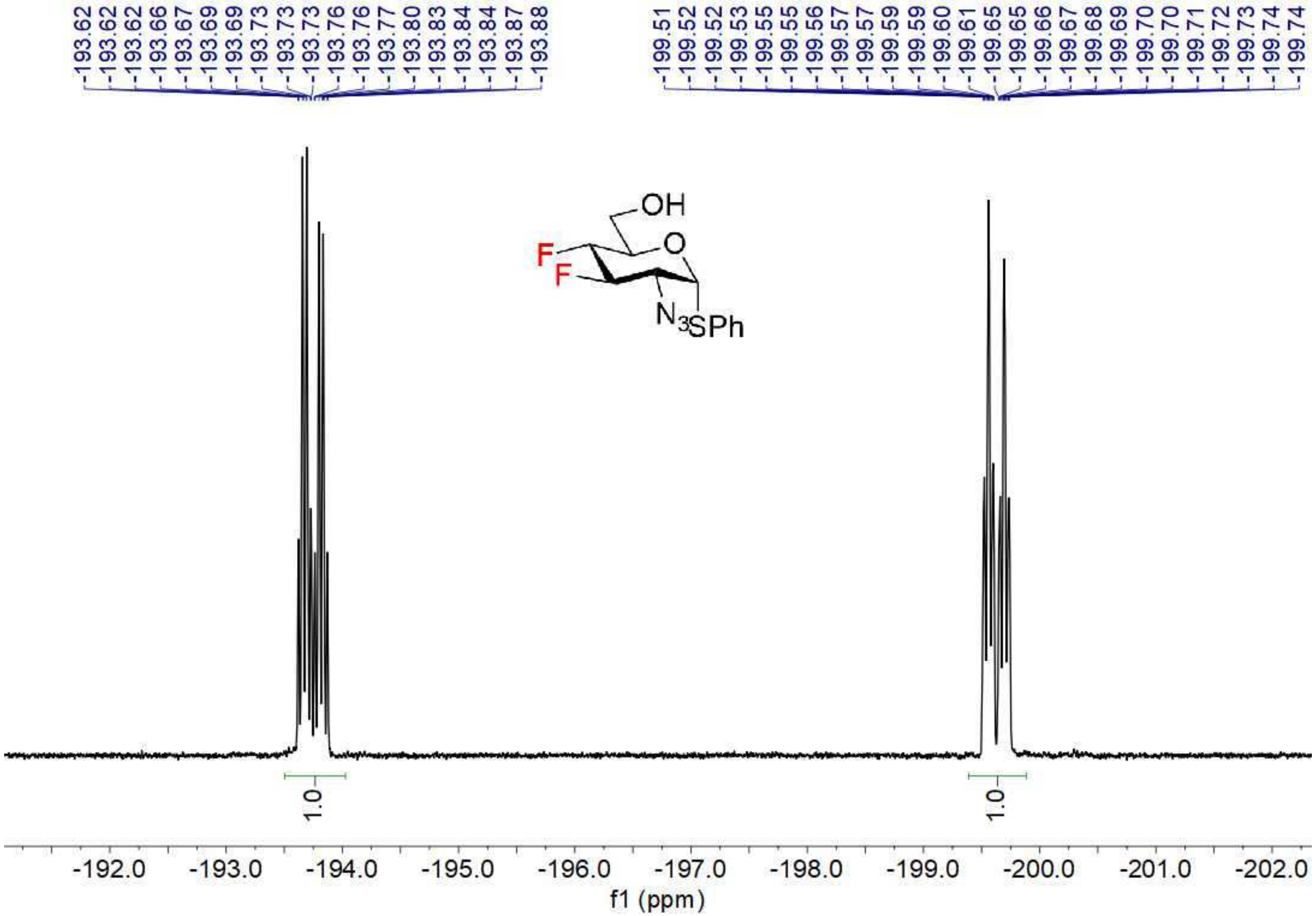
^{1}H NMR (400 MHz, $CDCl_3$) α-19



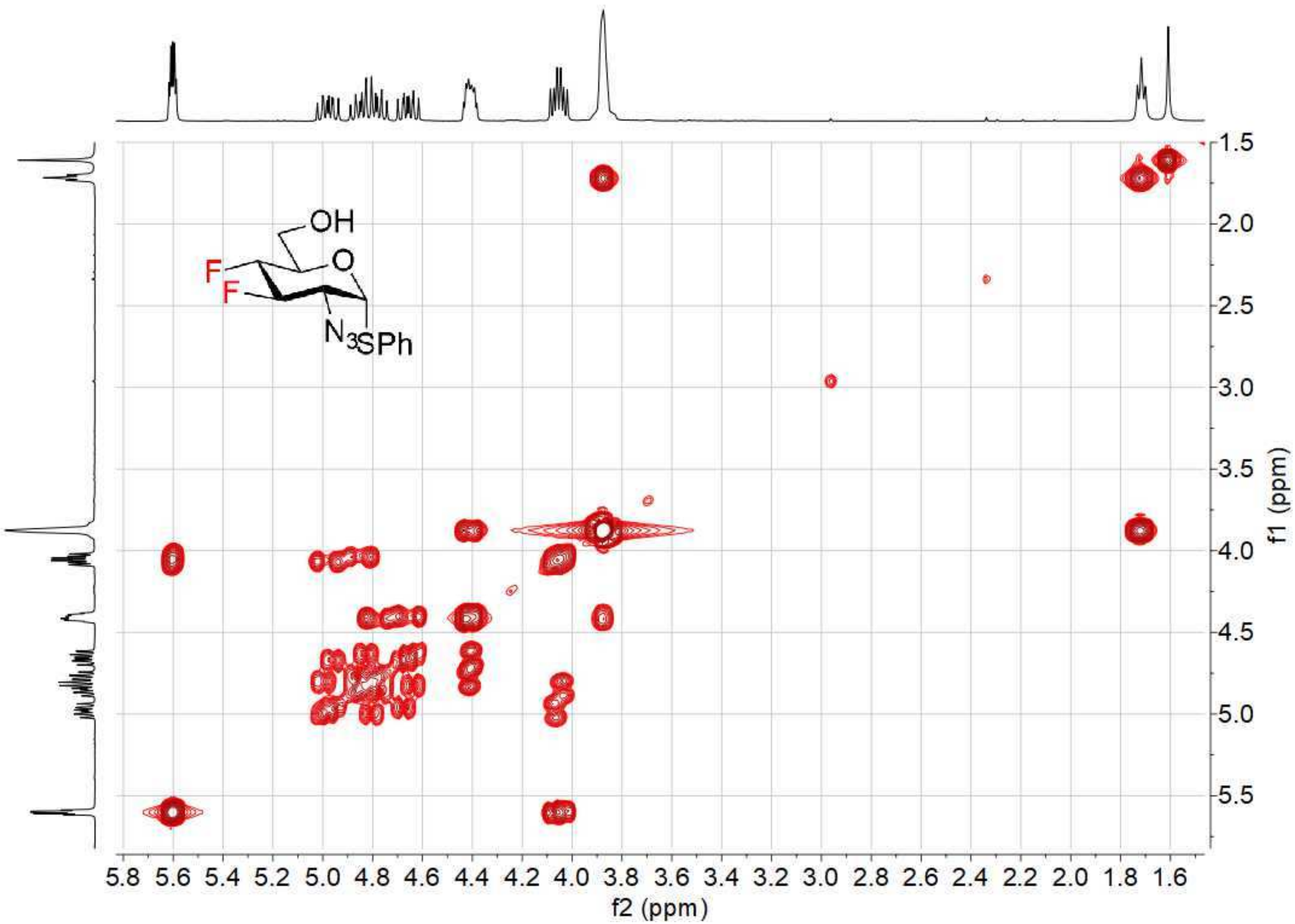
^{13}C NMR (100 MHz, $CDCl_3$) α-19



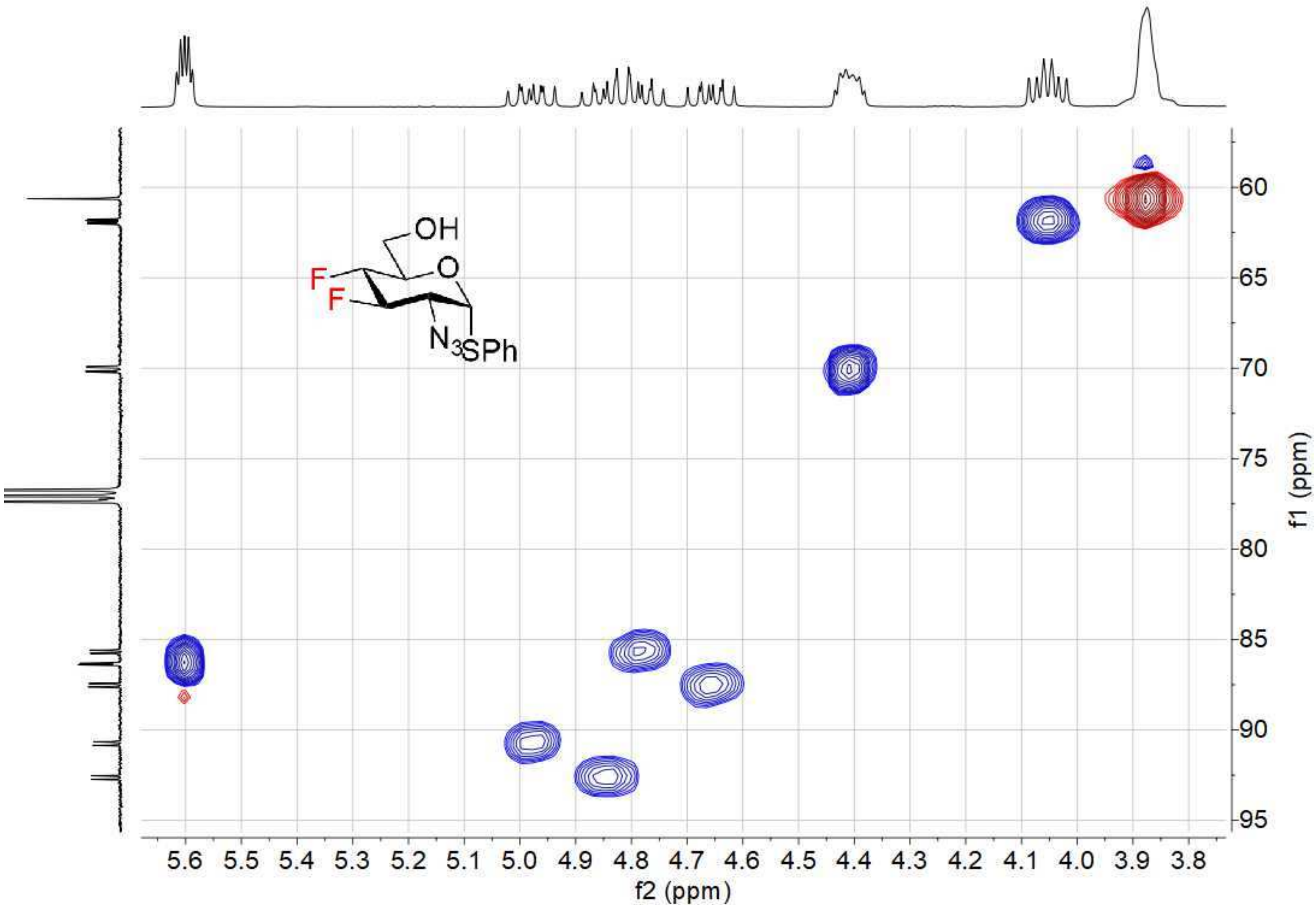
^{19}F NMR (376 MHz, $CDCl_3$) α-19



^{1}H-^{1}H COSY α-19



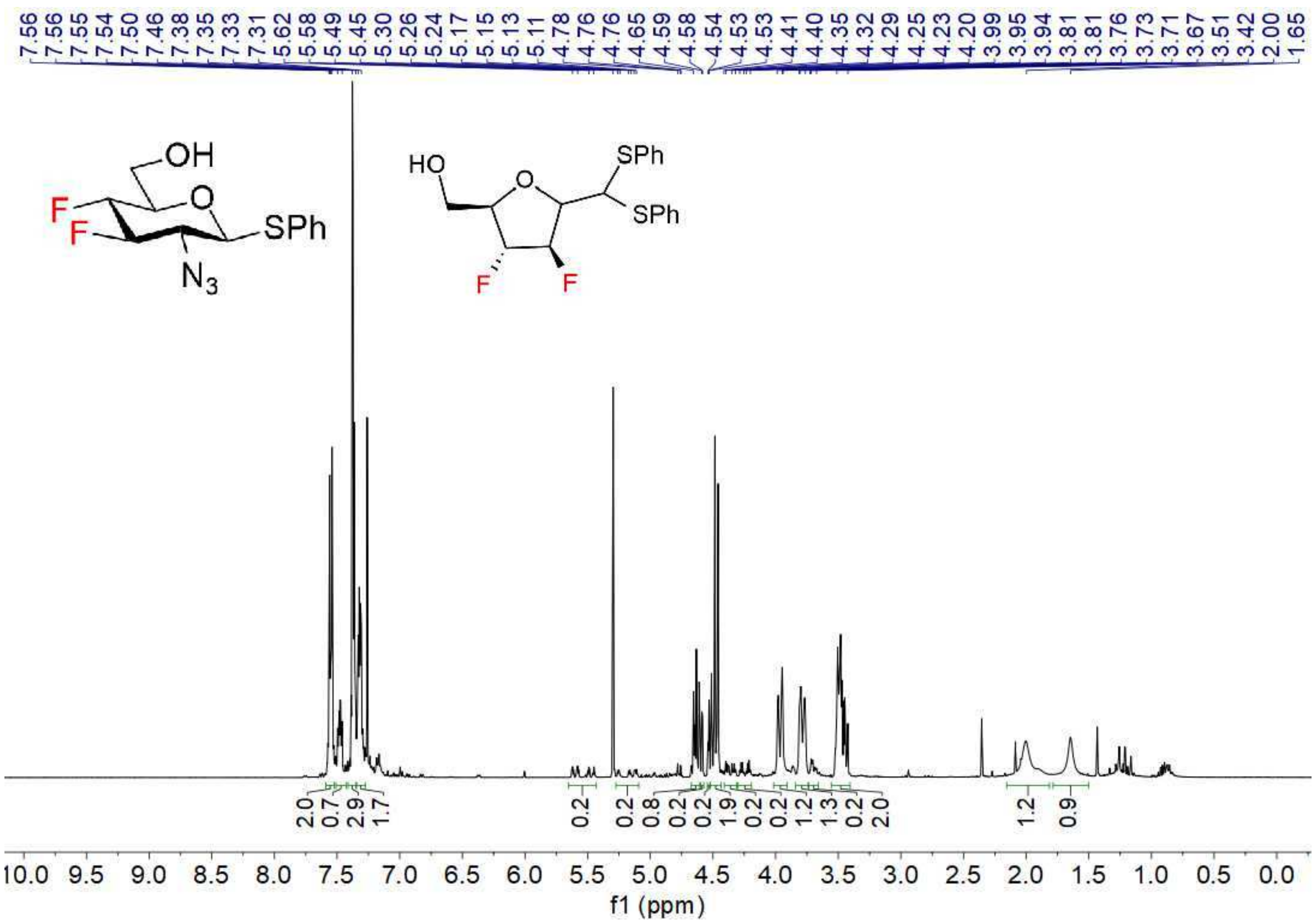
^{1}H-^{13}C HSQC α-19



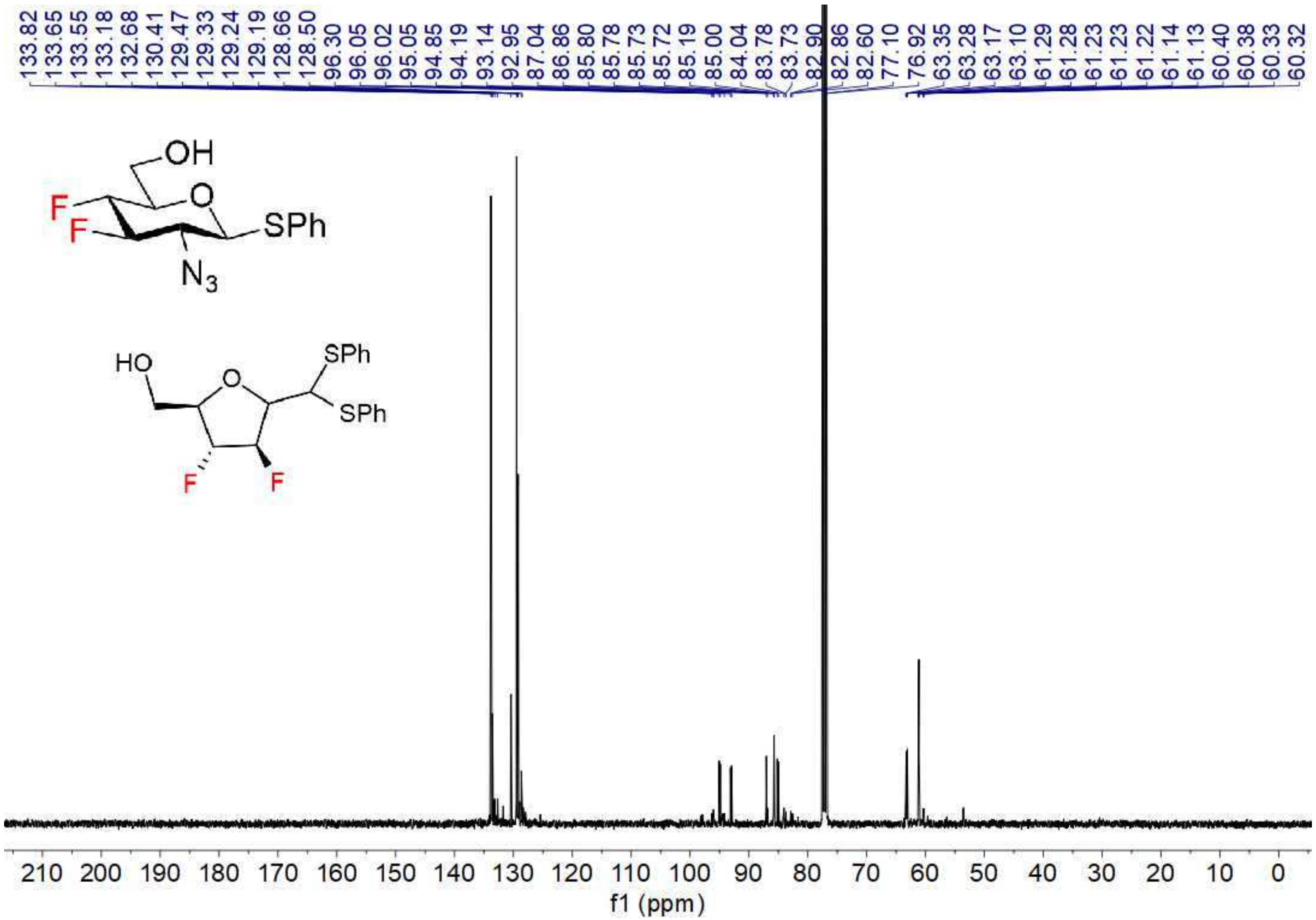
NMR COMPOUND β-19

(ca 16% of 2,3-dideoxy-2,3-difluoro-*C*-α- or β-D-arabinofuranosyl-formaldehyde diphenyl dithioacetal S1)

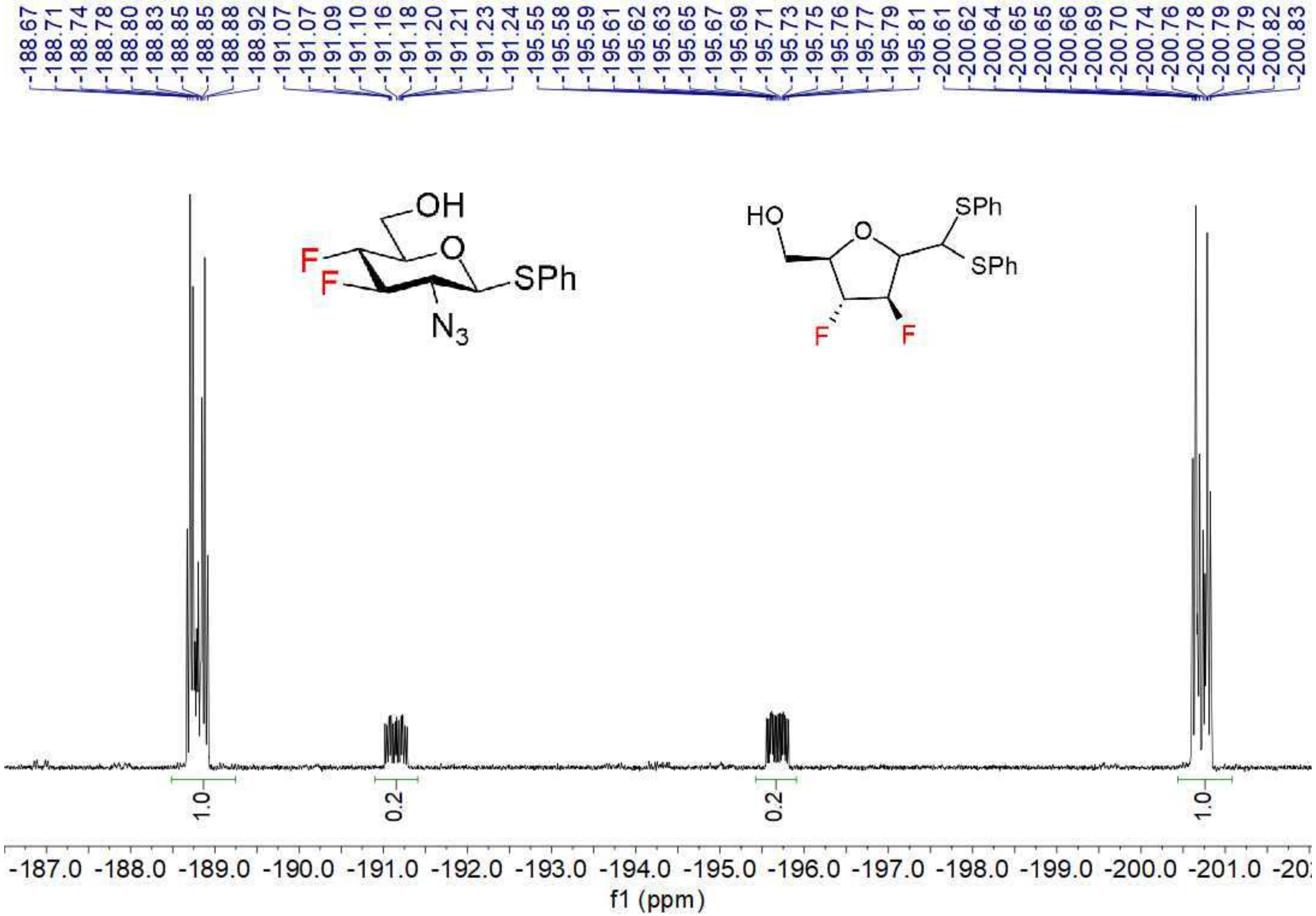
^{1}H NMR (400 MHz, $CDCl_3$) β-19 (and ca 16% S1)



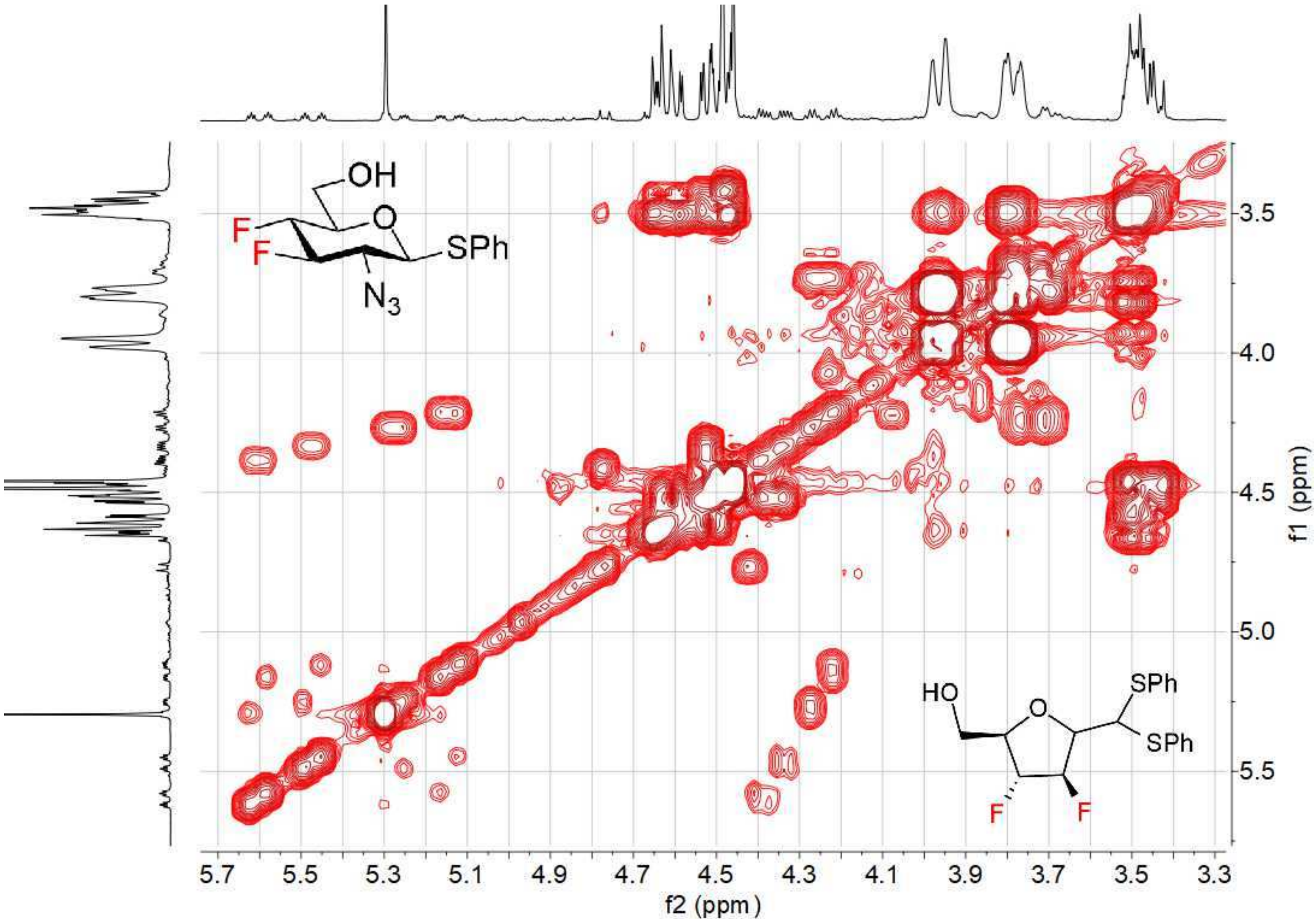
^{13}C NMR (100 MHz, CDCl$_3$) β-19 (and ca 16% S1)



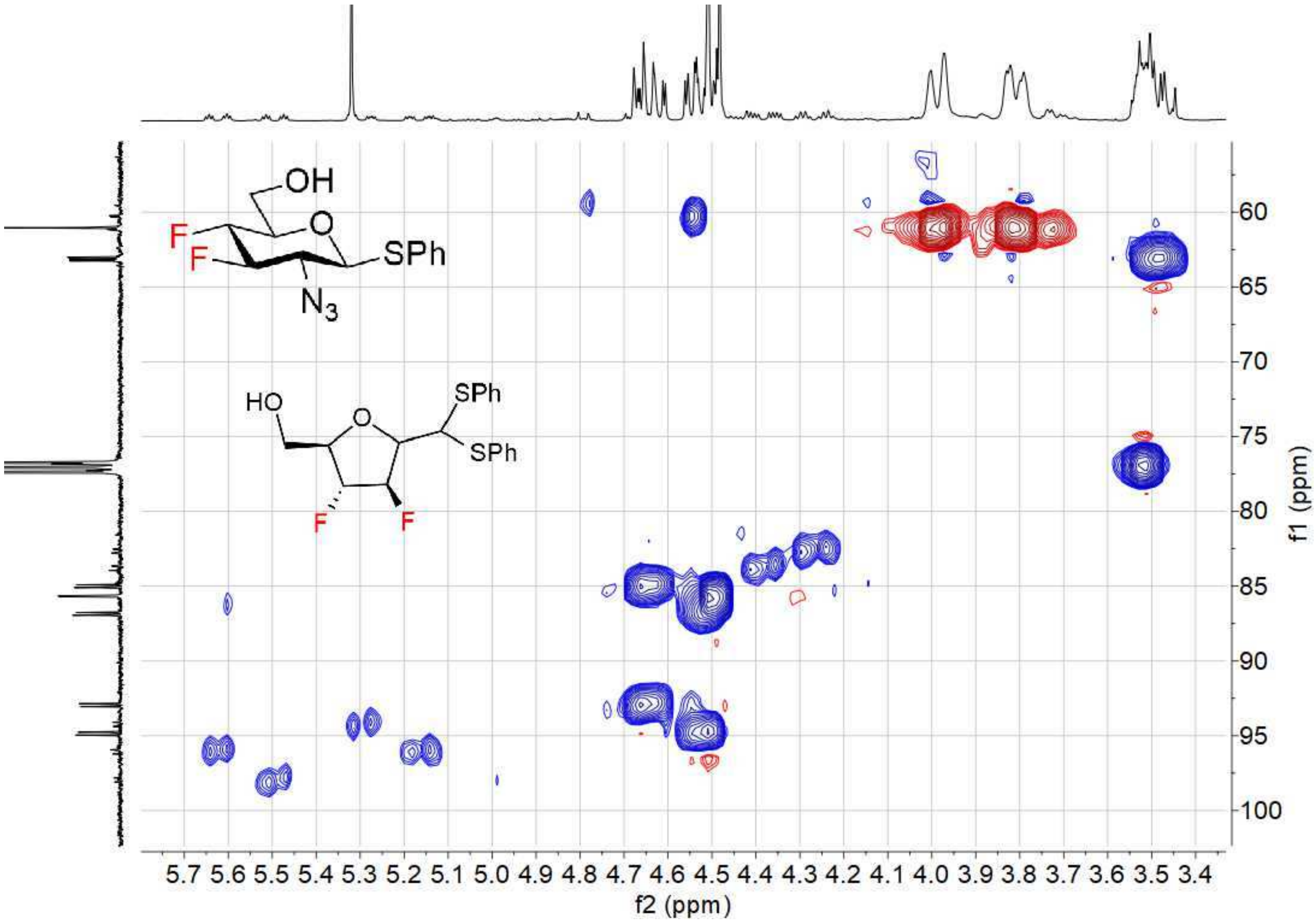
^{19}F NMR (376 MHz, CDCl$_3$) β-19 (and ca 16% S1)



^{1}H-^{1}H COSY β-19

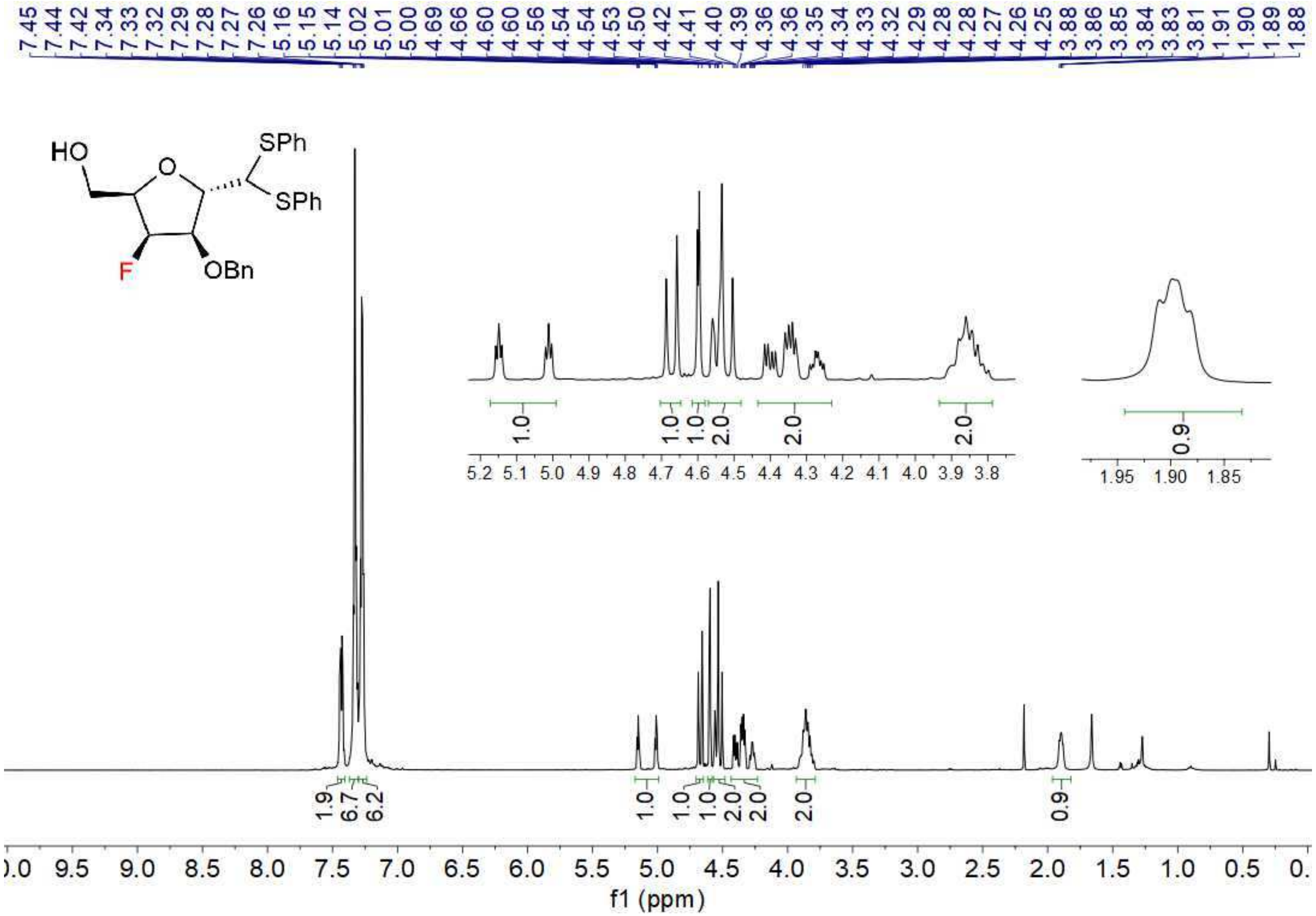


^{1}H-^{13}C HSQC β-19

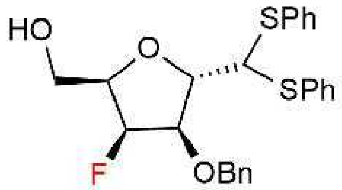


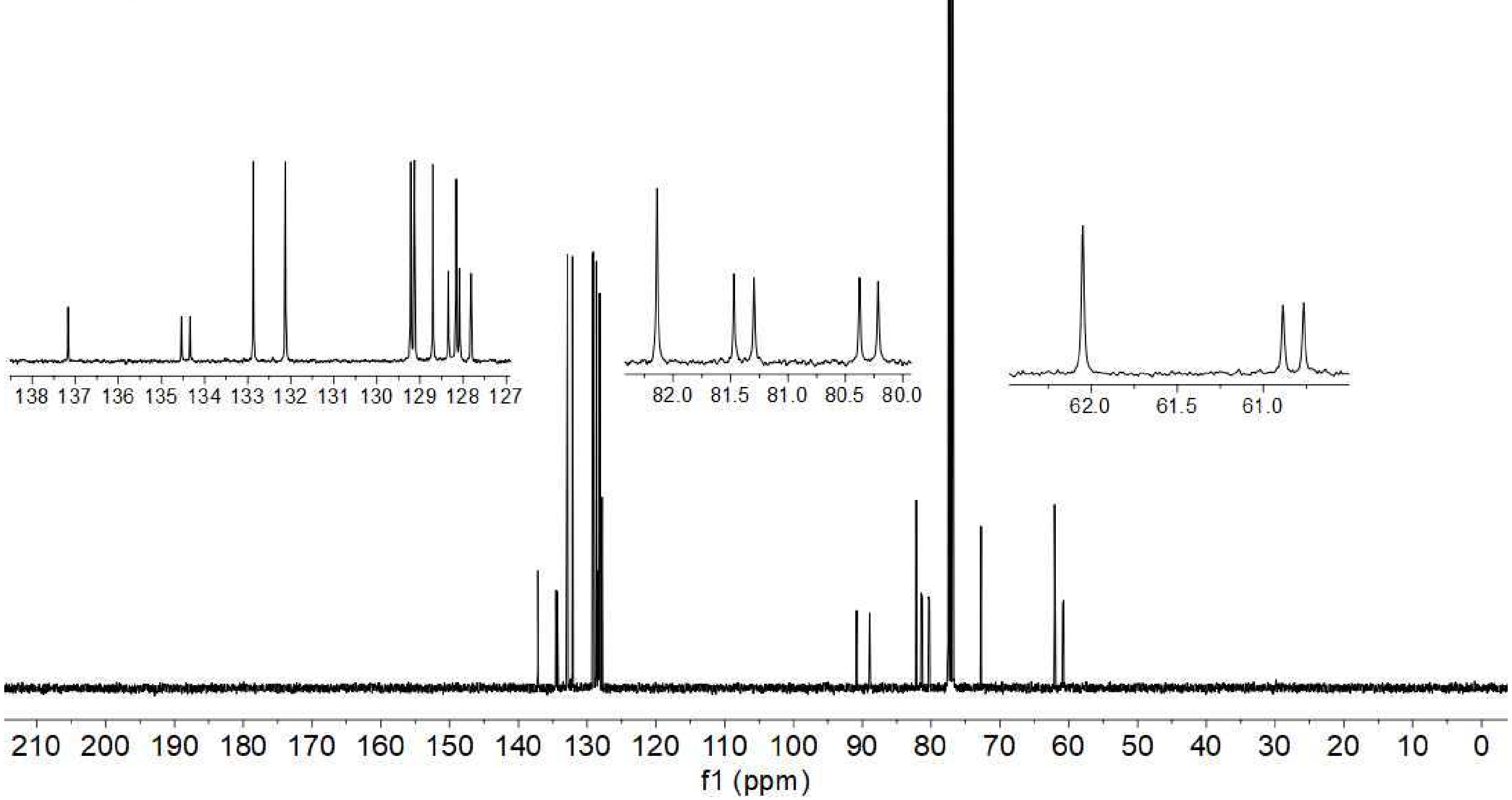
NMR COMPOUND 20

^{1}H NMR (400 MHz, CDCl$_3$) 20

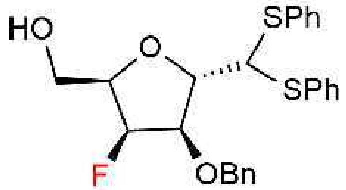


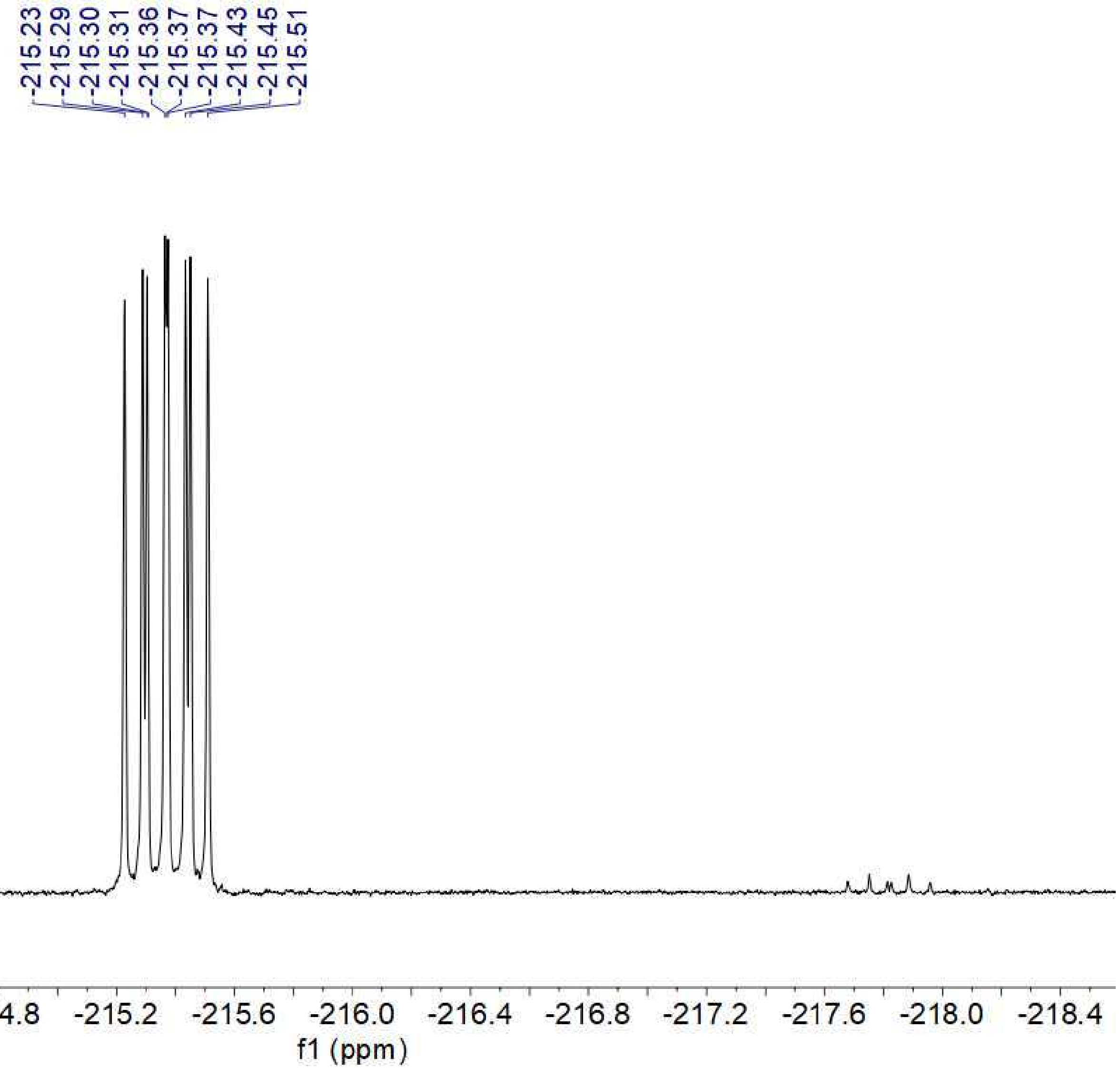
^{13}C NMR (100 MHz, CDCl$_3$) 20



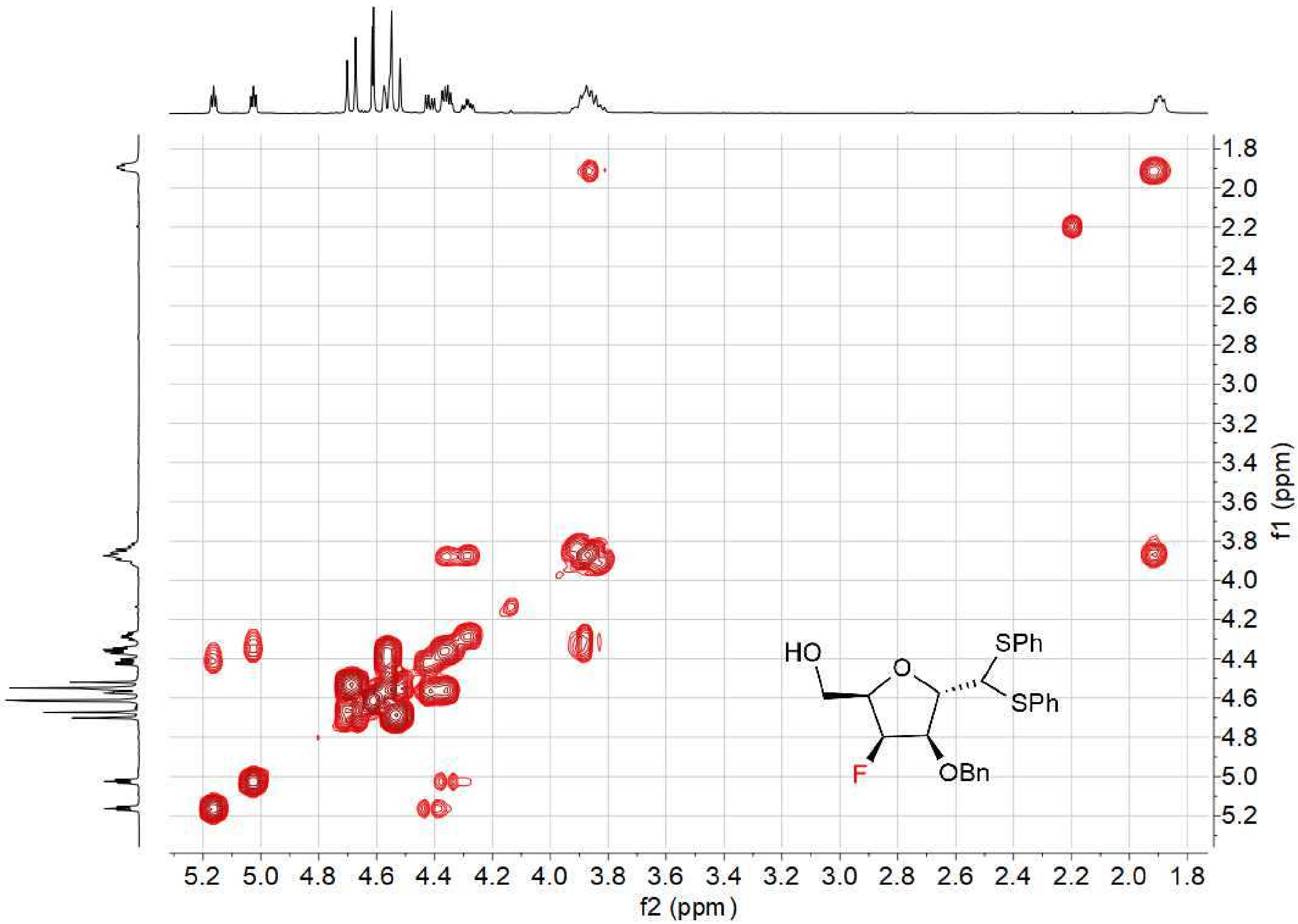


^{19}F NMR (376 MHz, CDCl$_3$) 20

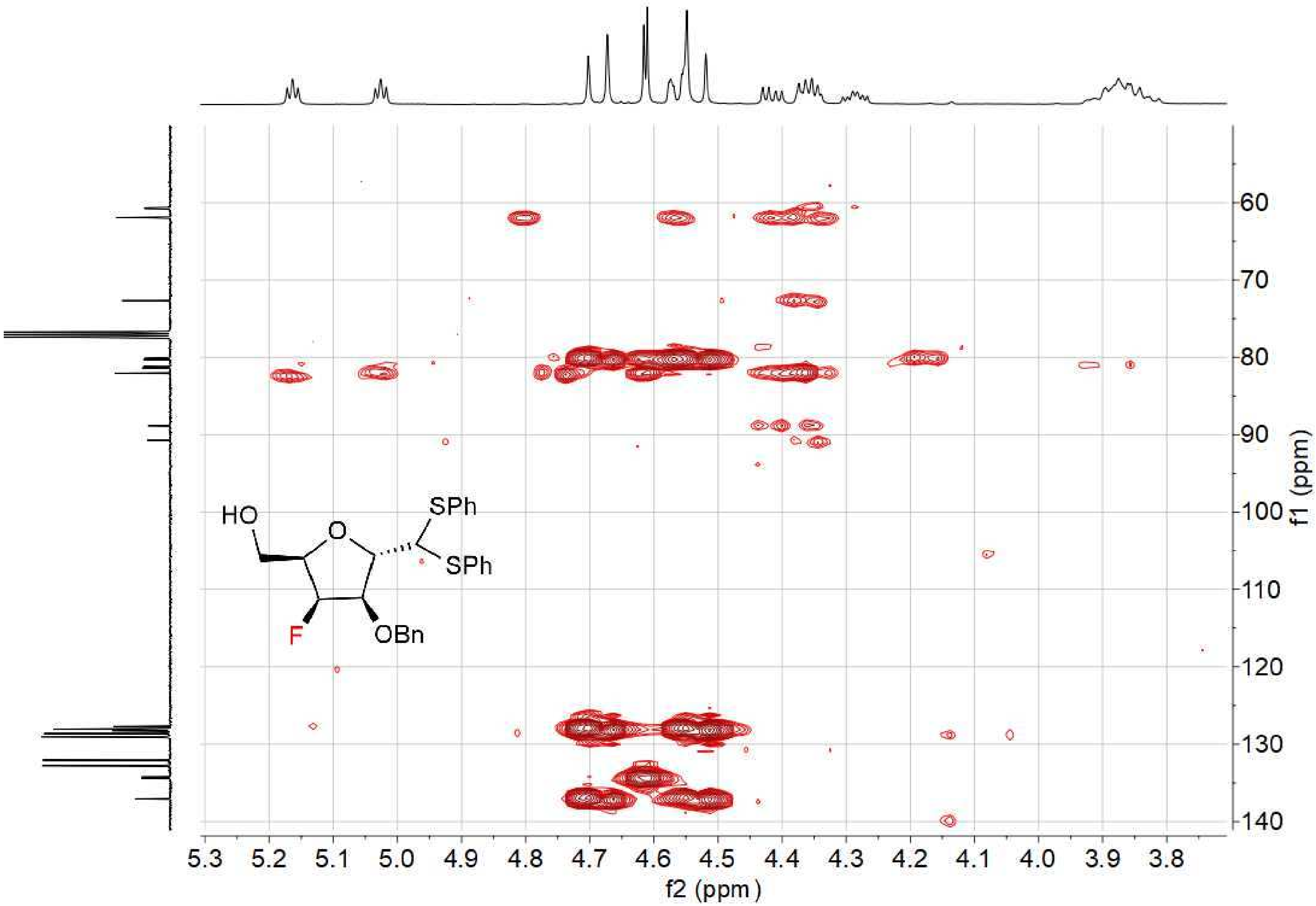




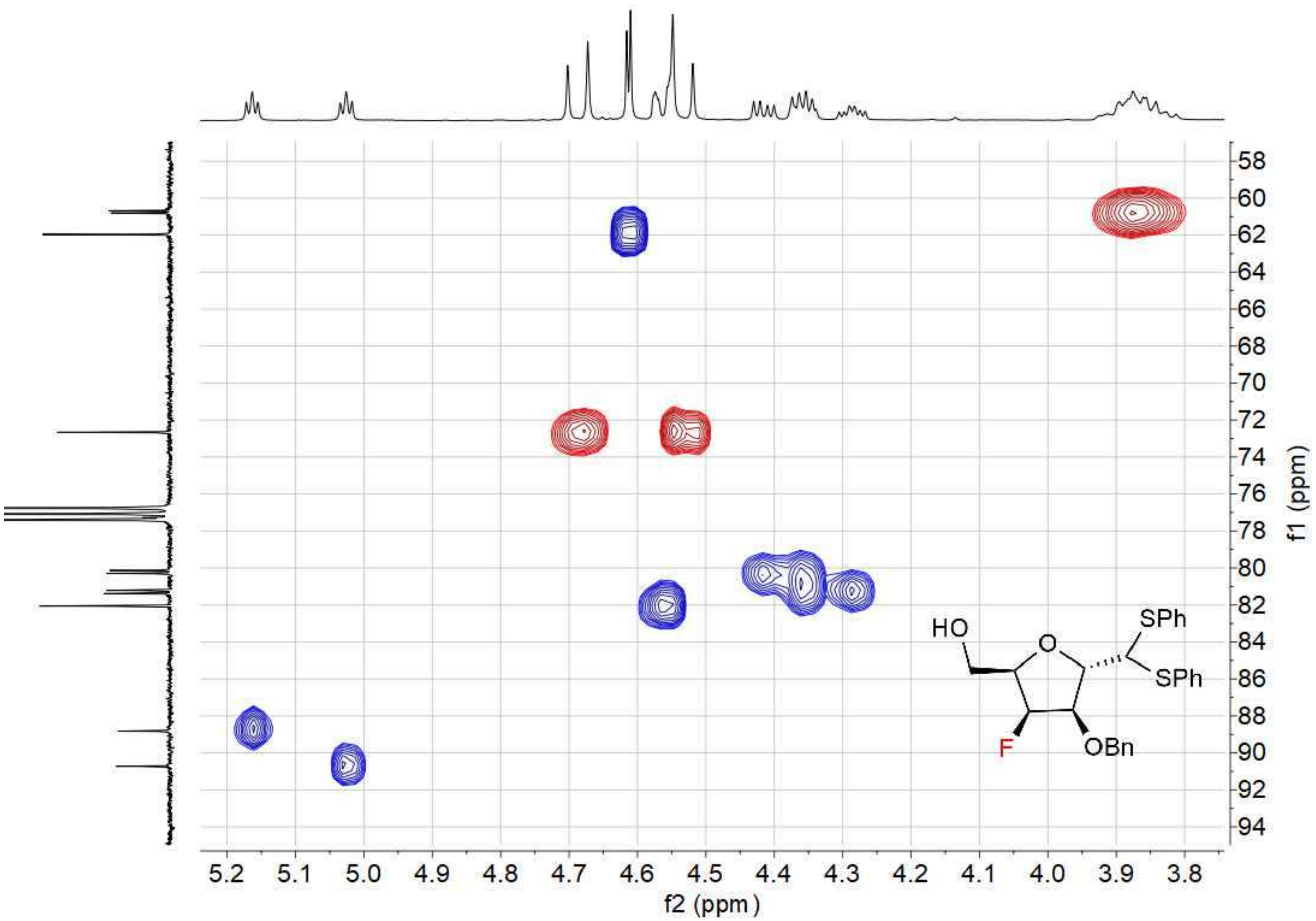
^1H-^1H COSY 20



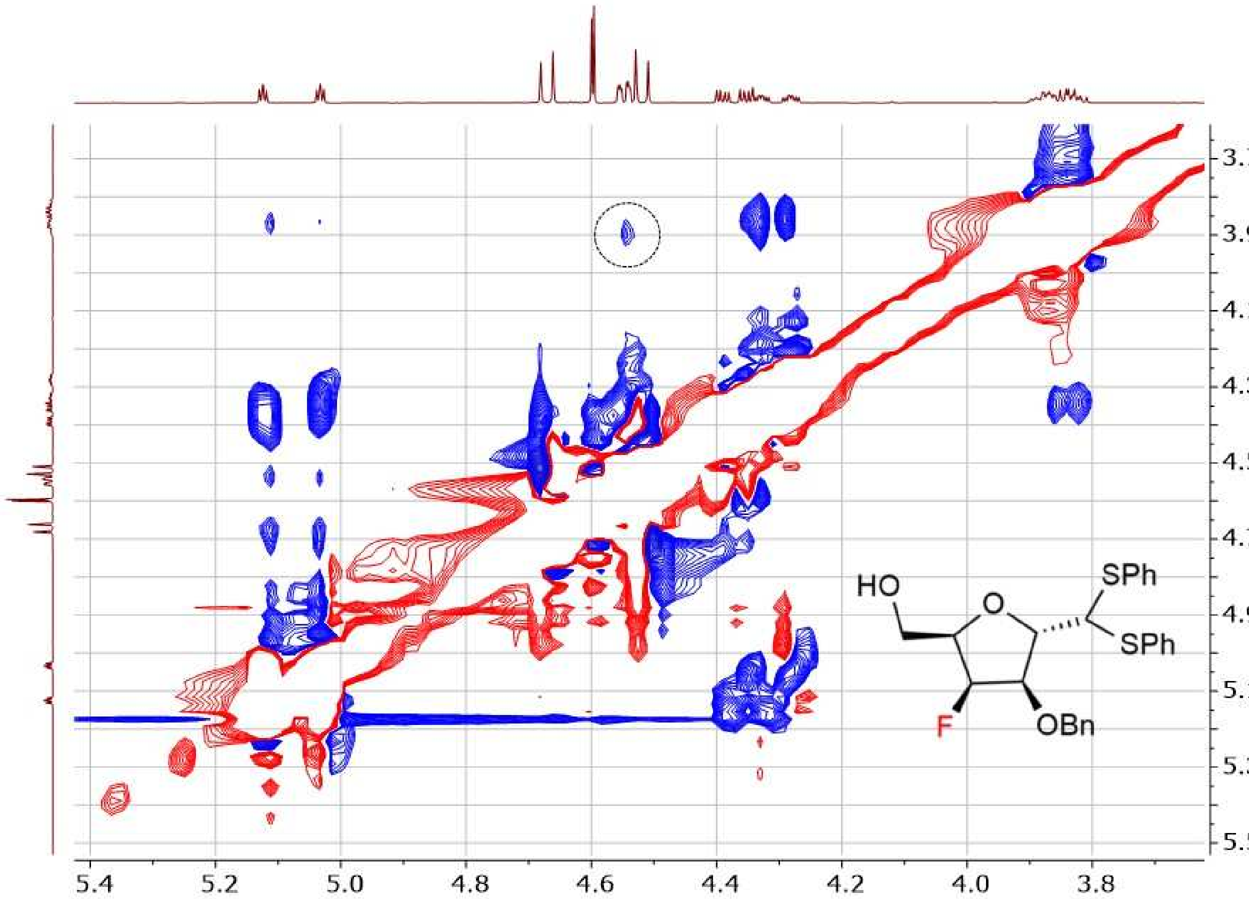
^{1}H-^{13}C HMBC 20



^{1}H-^{13}C HSQC 20

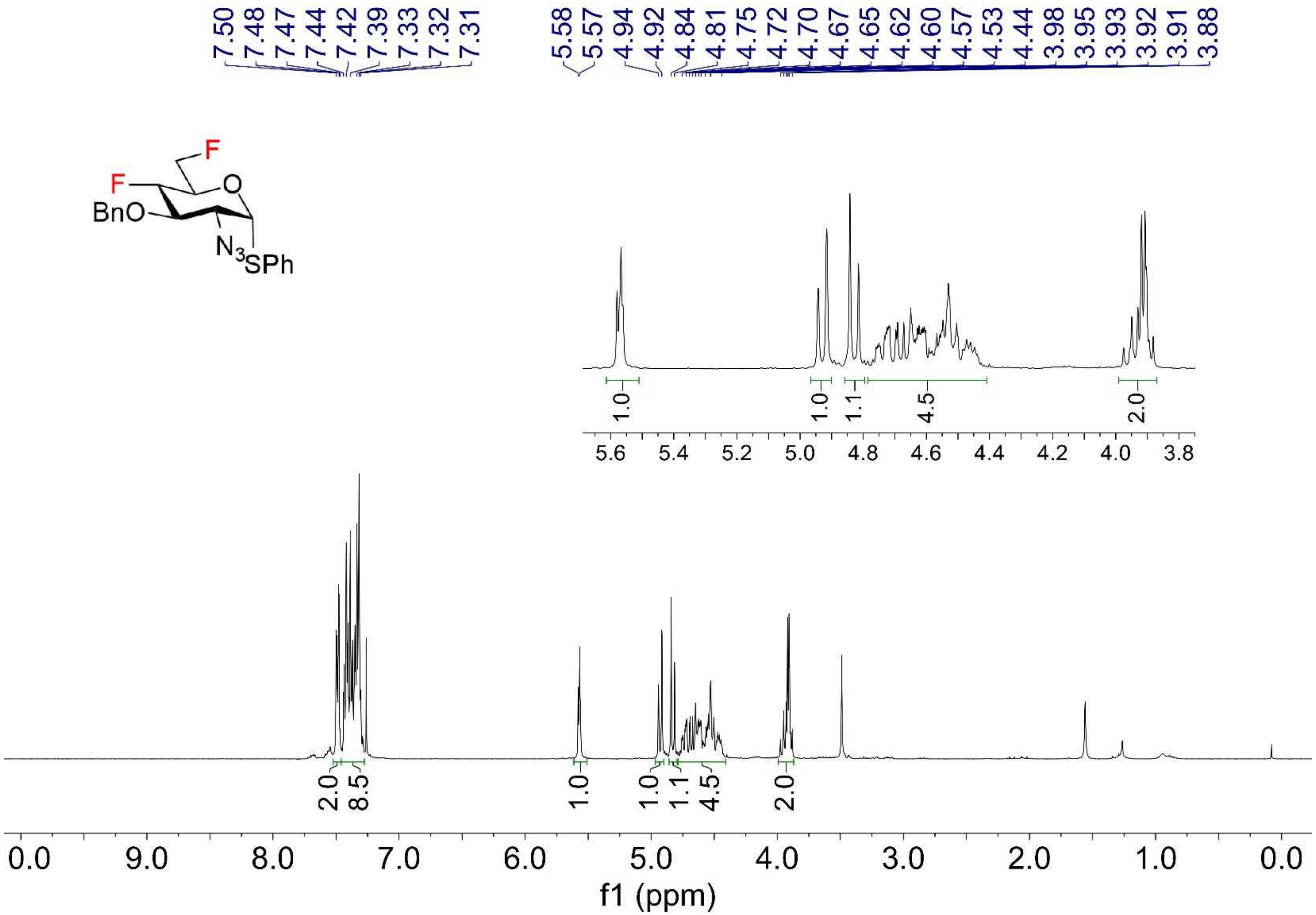


^{1}H-^{3}H ROESY 20

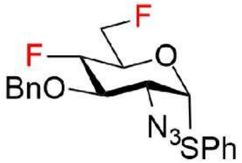


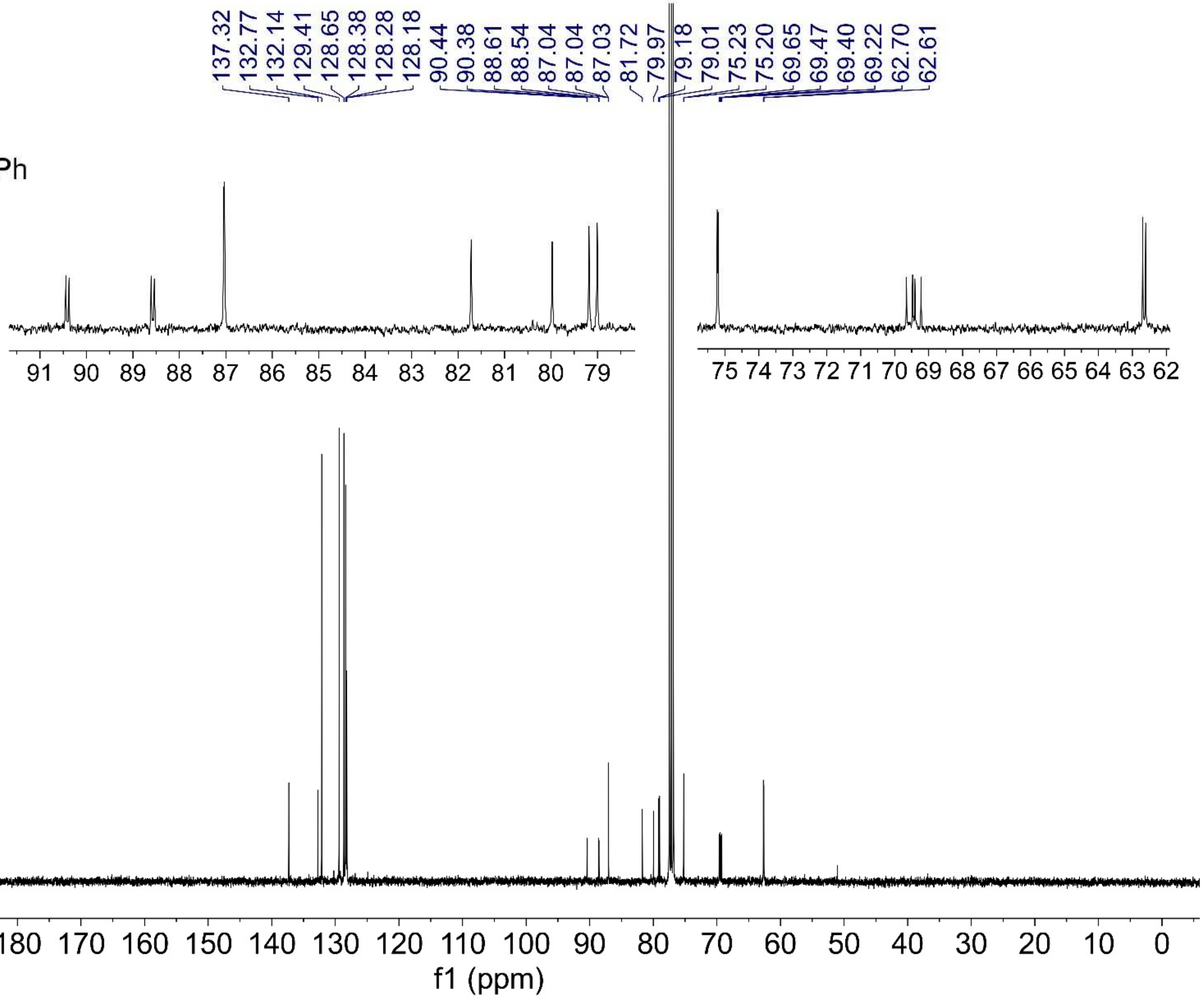
NMR COMPOUND α-23

^{1}H NMR (400 MHz, $CDCl_3$) α-23

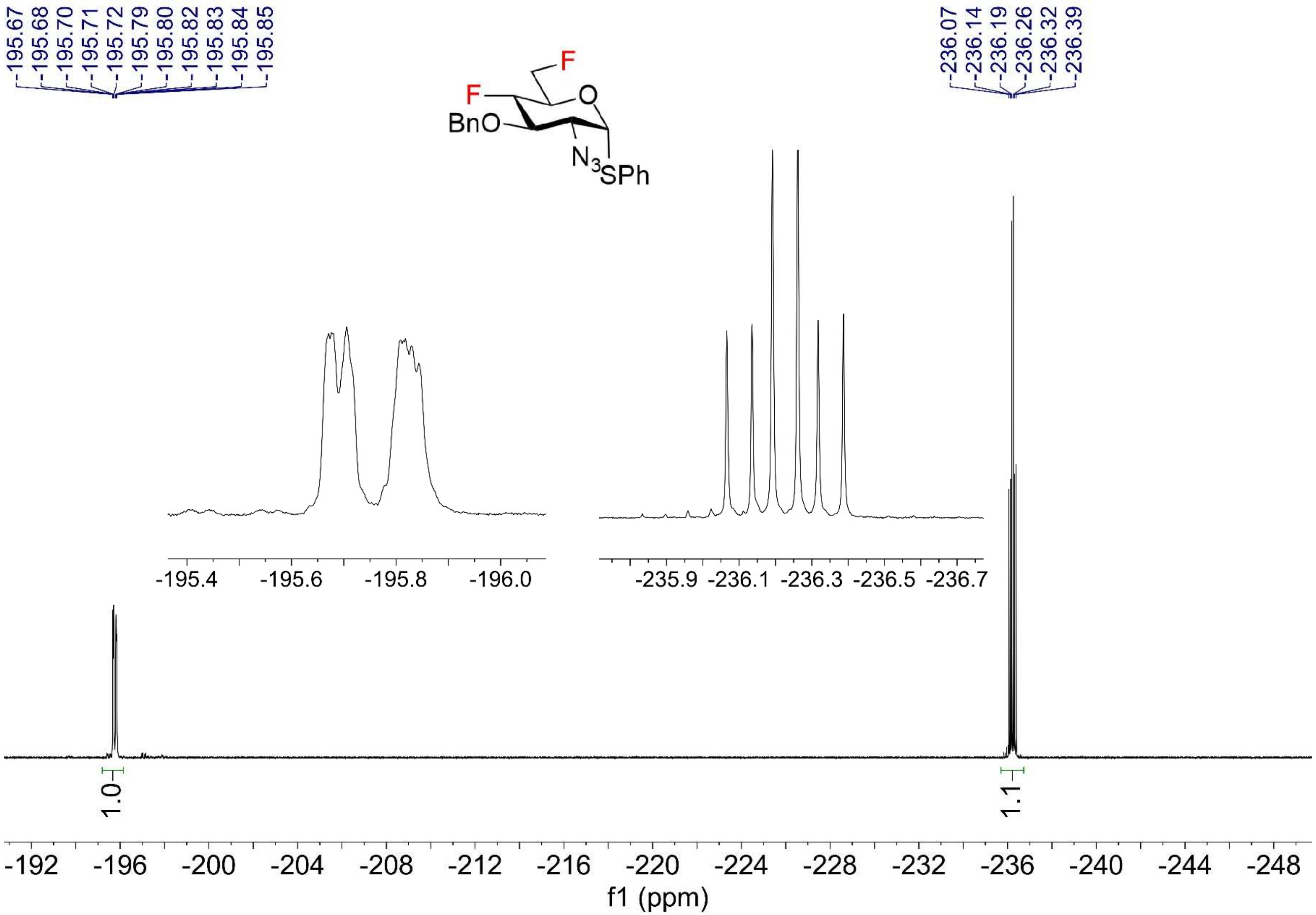


^{13}C NMR (100 MHz, CDCl$_3$) α-23

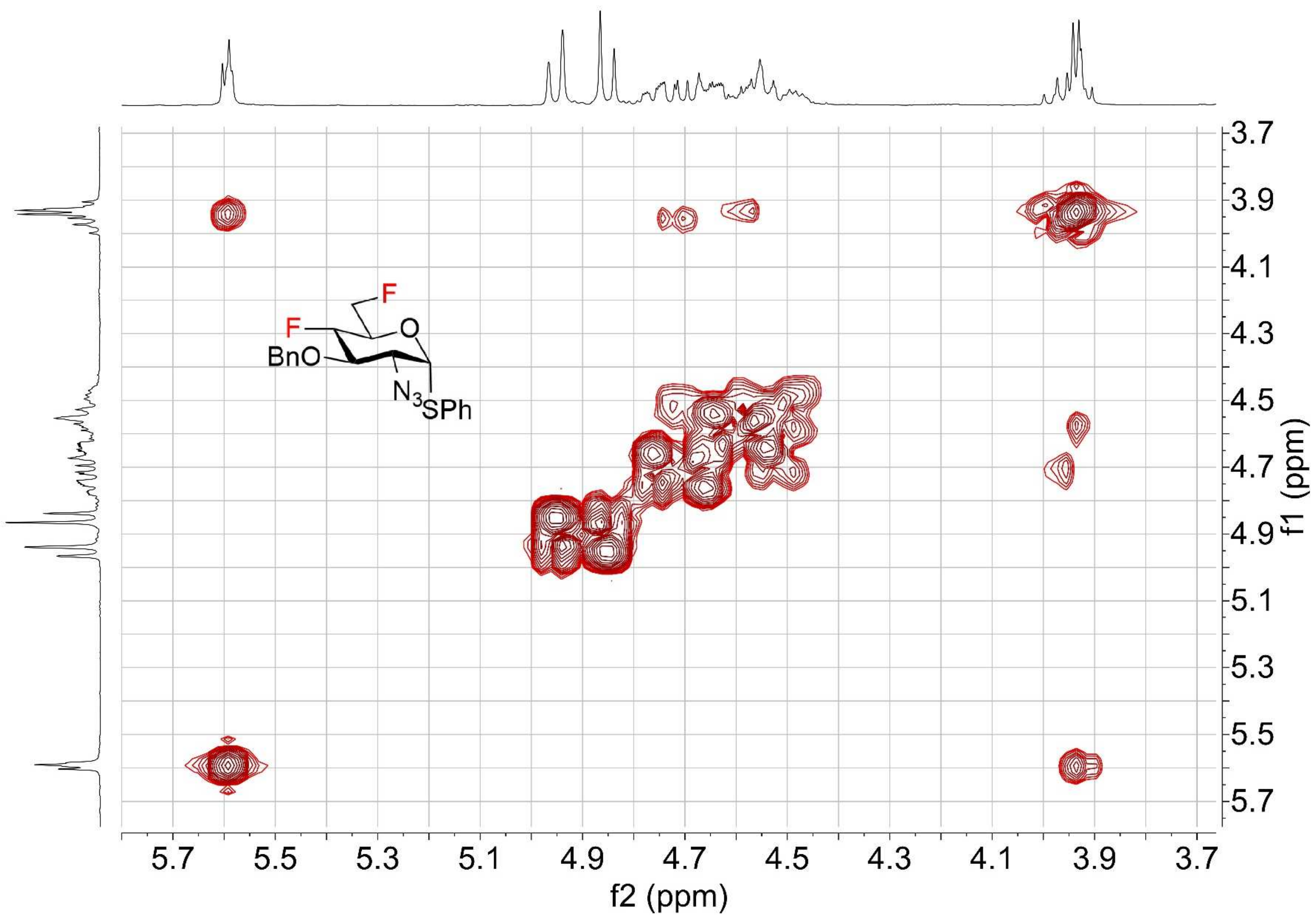




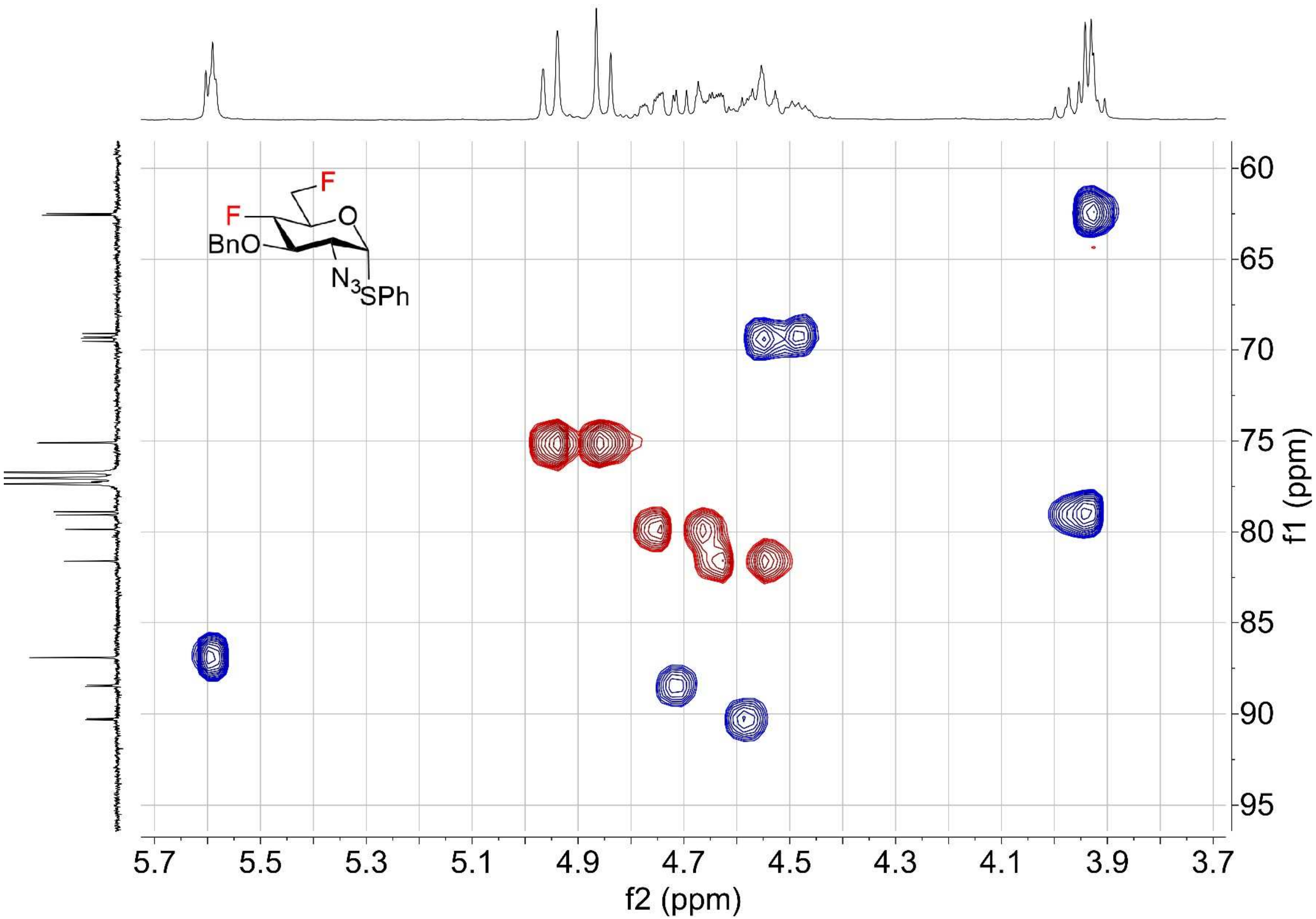
^{19}F NMR (376 MHz, CDCl$_3$) α-23



^{1}H-^{1}H COSY α-23

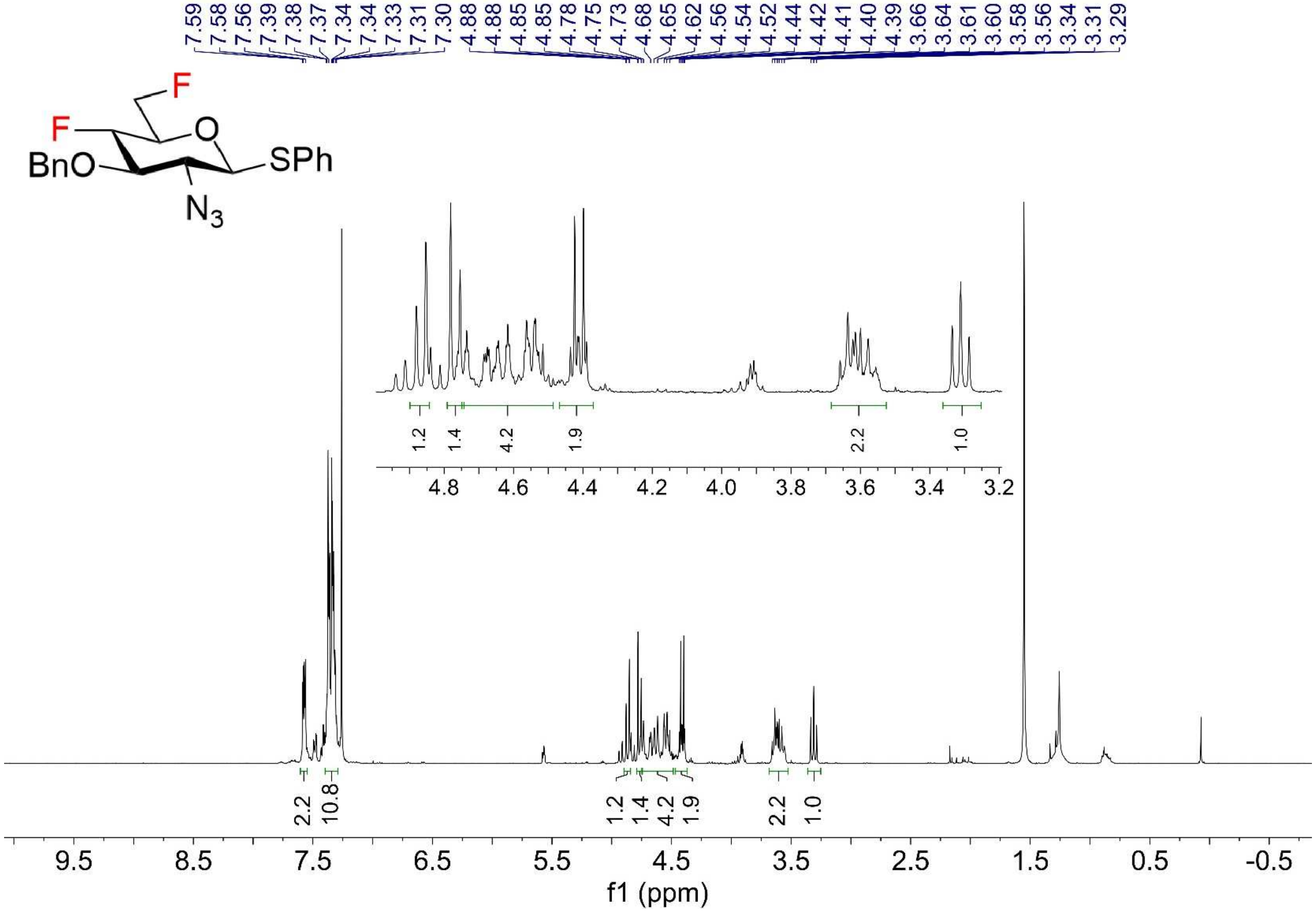


^{1}H-^{13}C HSQC α-23

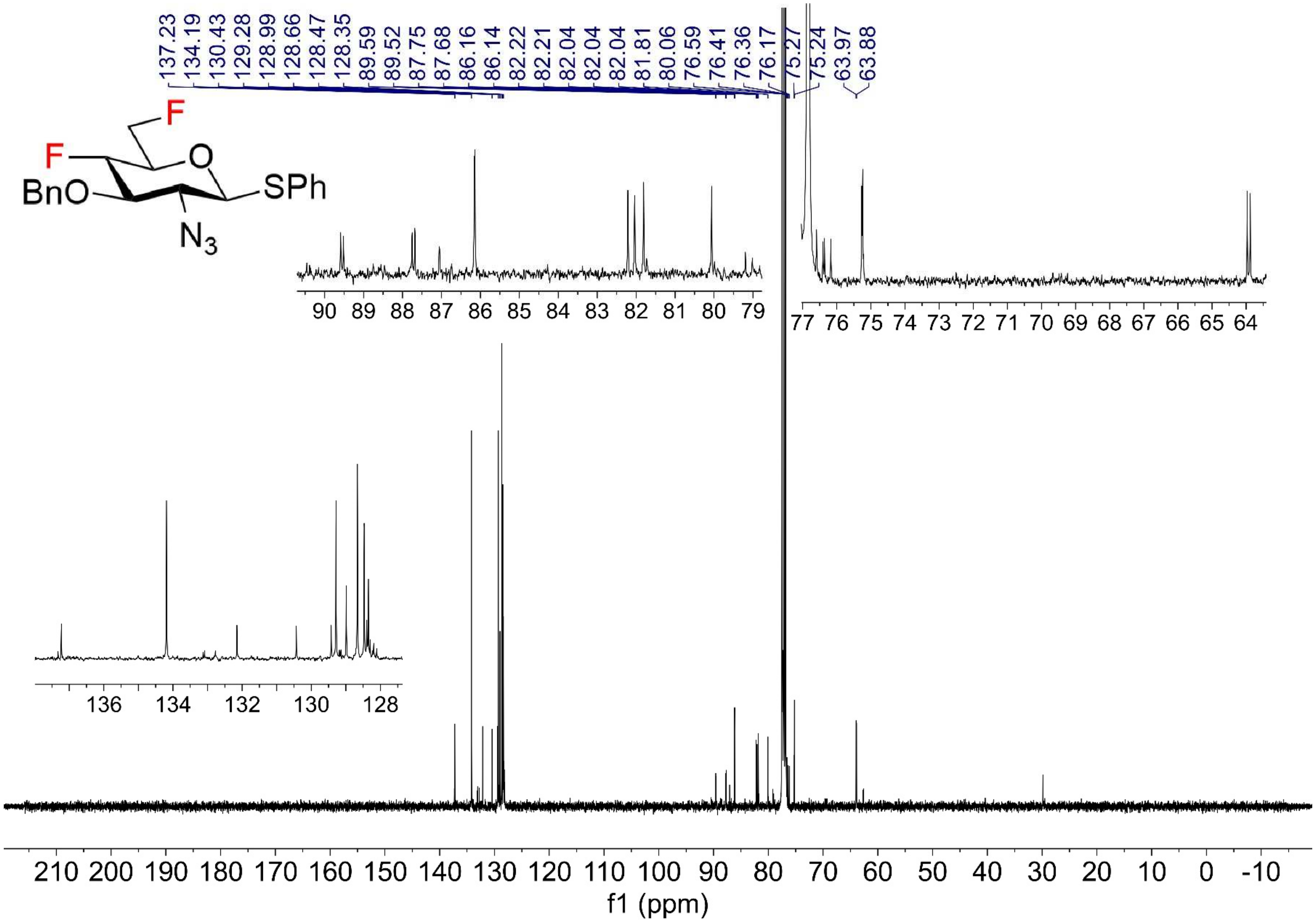


NMR COMPOUND β-23

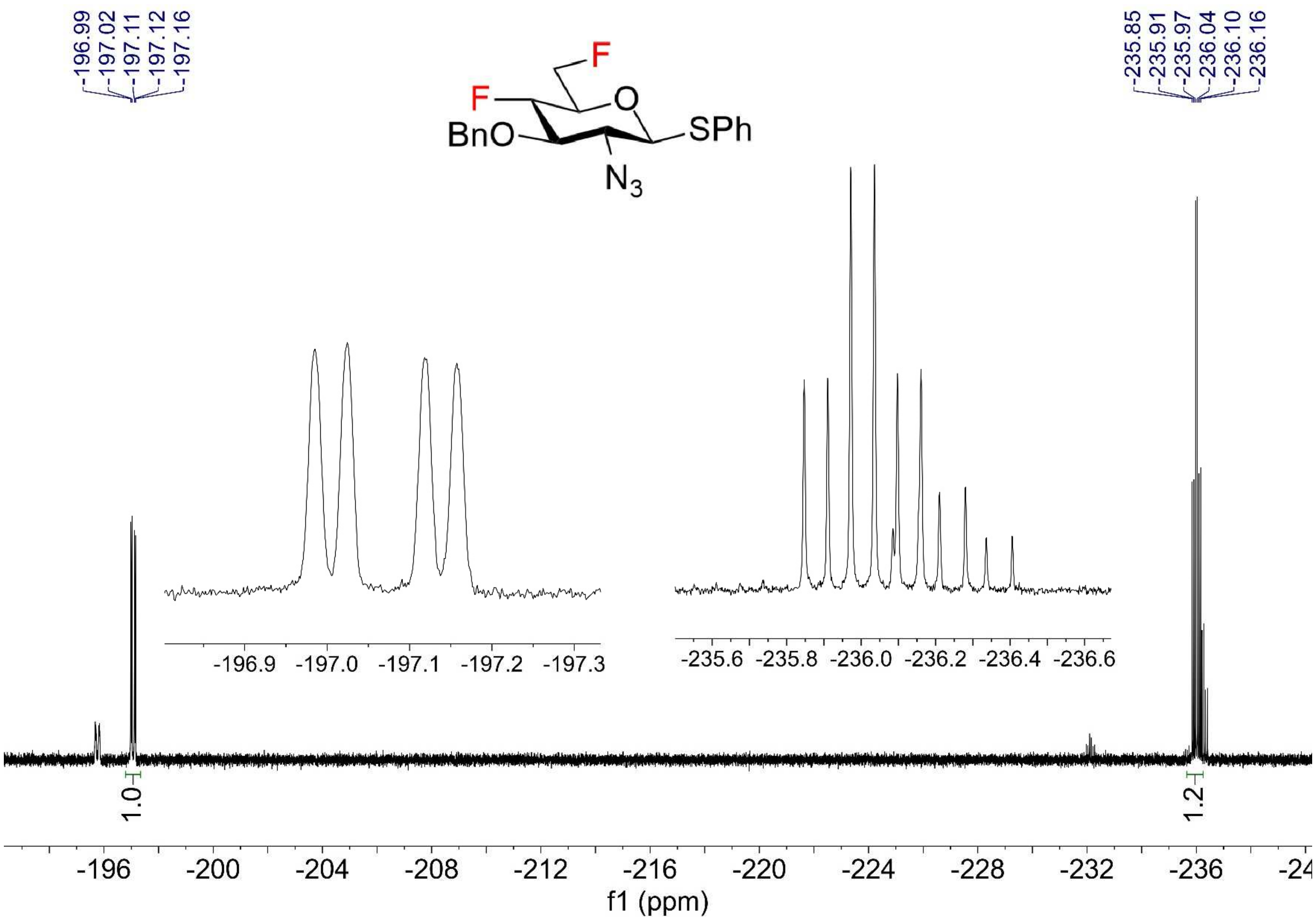
^{1}H NMR (400 MHz, $CDCl_3$) β-23



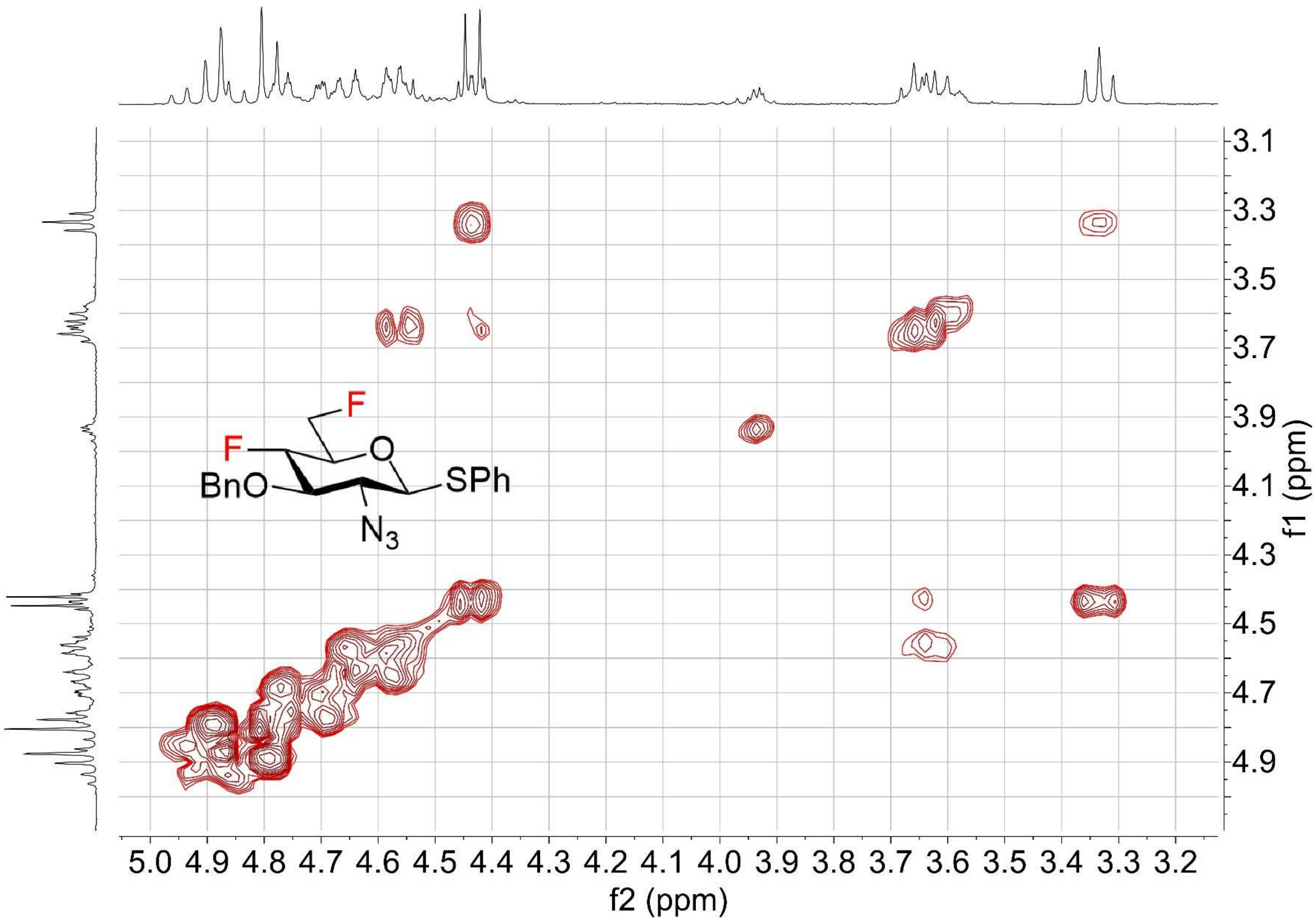
^{13}C NMR (100 MHz, CDCl$_3$) β-23



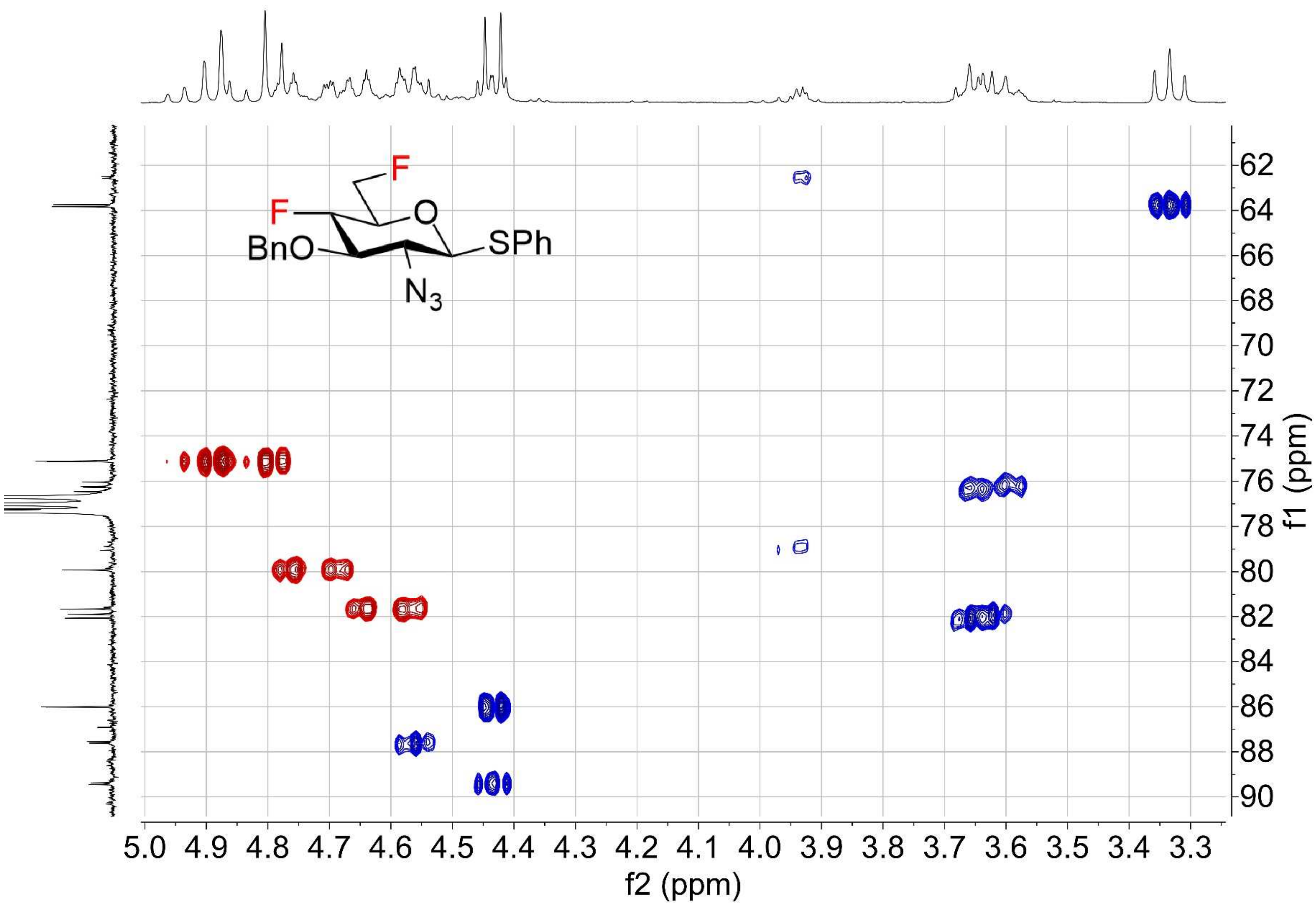
^{19}F NMR (376 MHz, CDCl$_3$) β-23



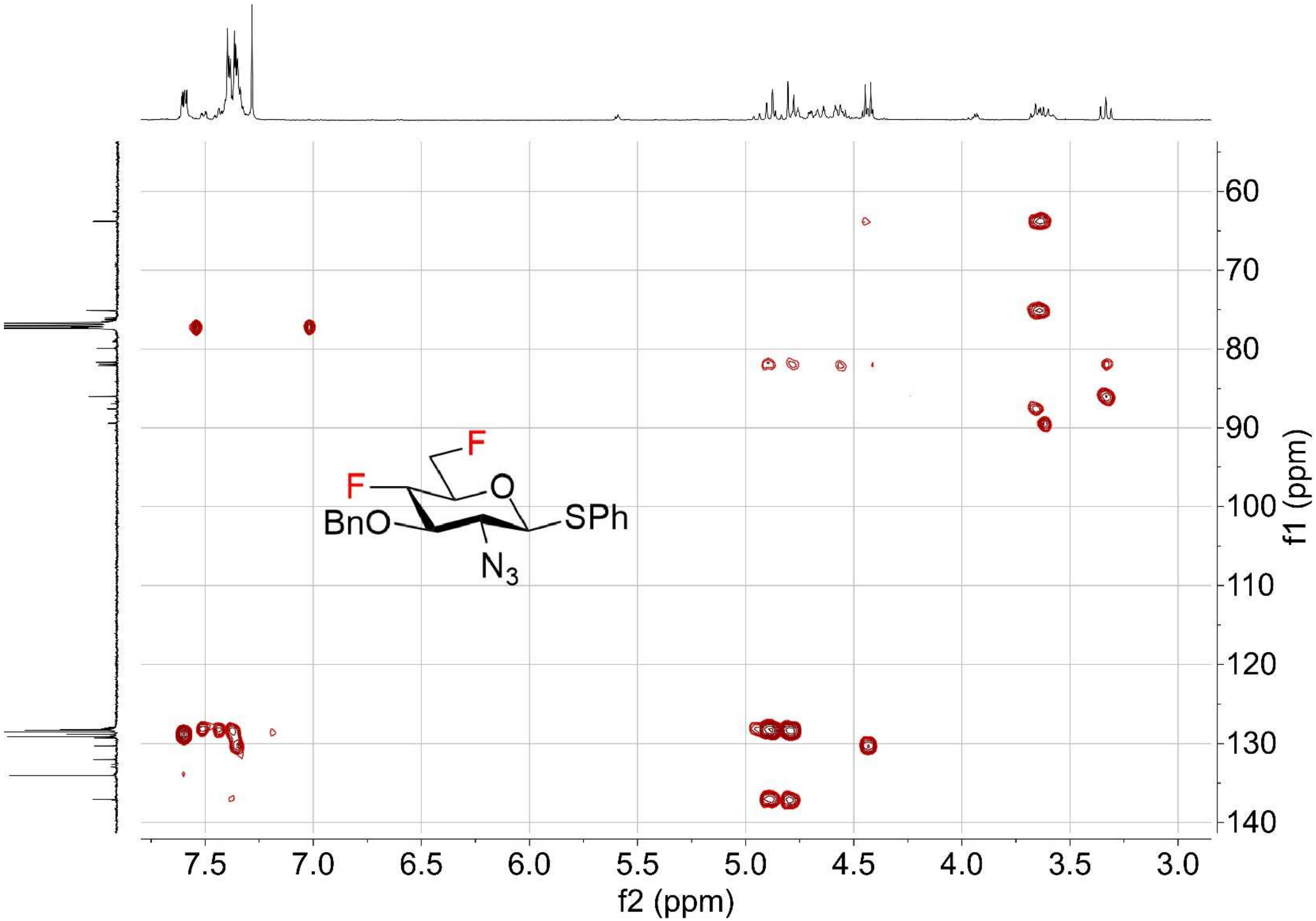
^1H-^1H COSY β-23



^{1}H-^{13}C HSQC β-23

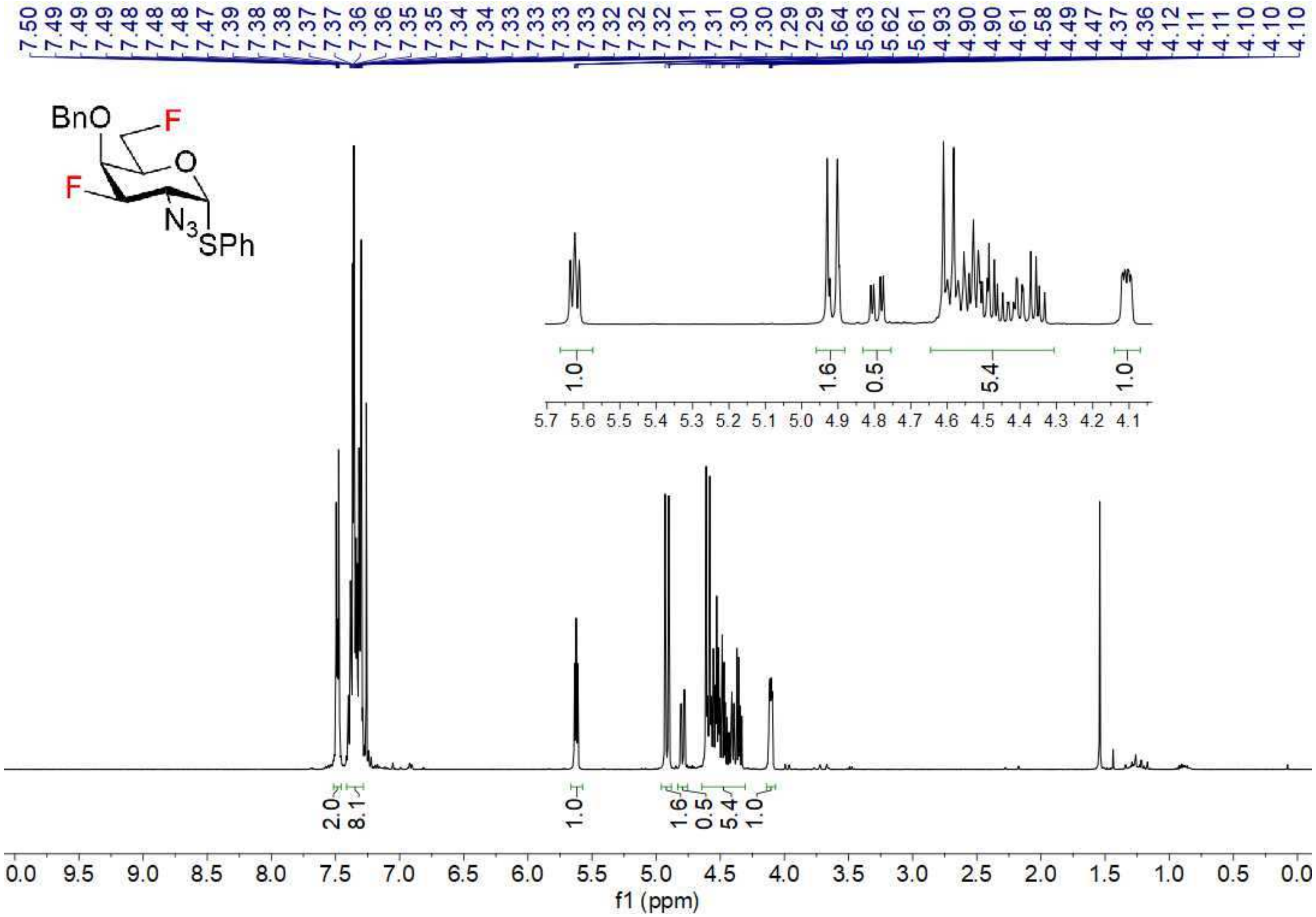


^{1}H-^{13}C HMBC β-23

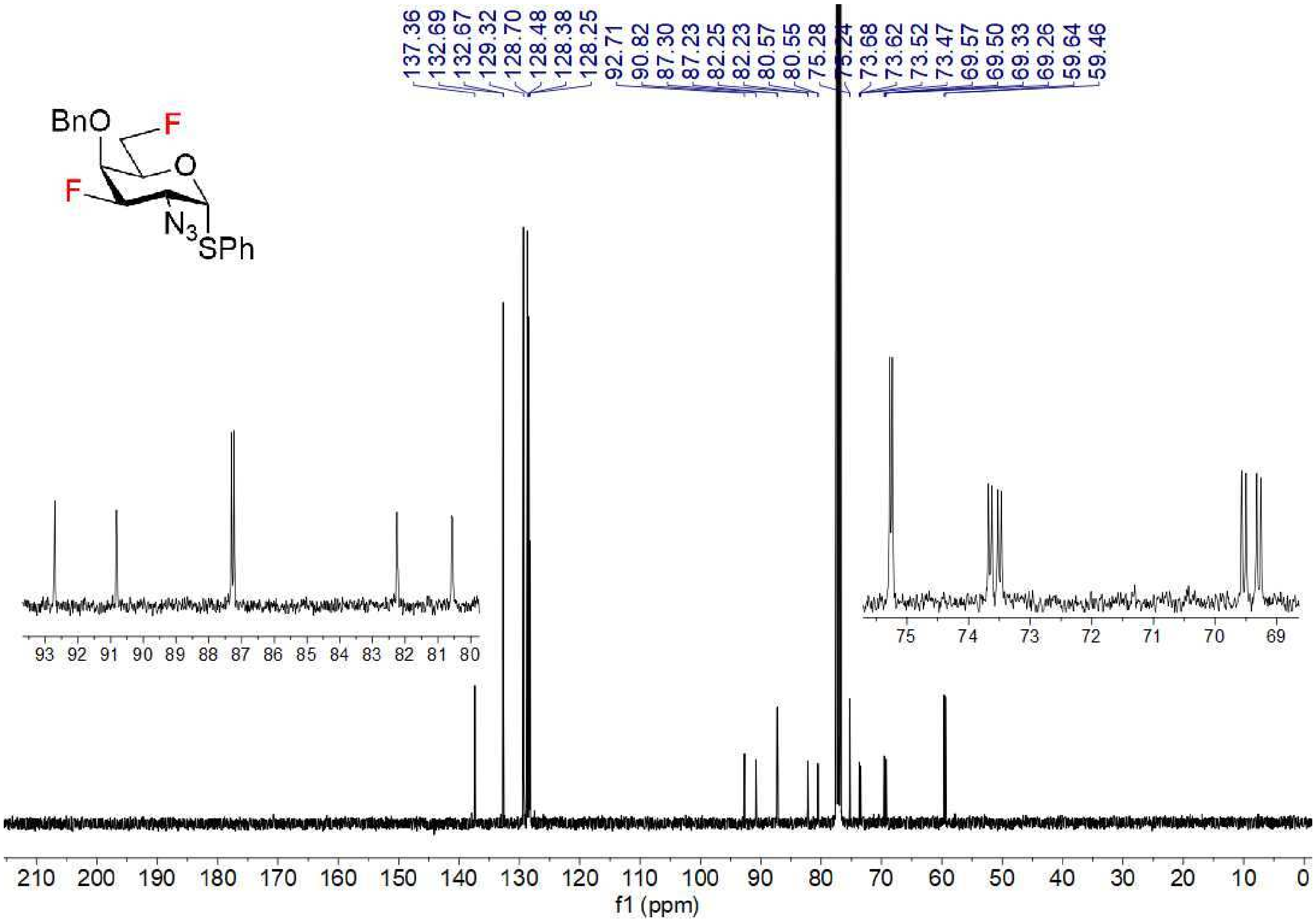


NMR COMPOUND α-24

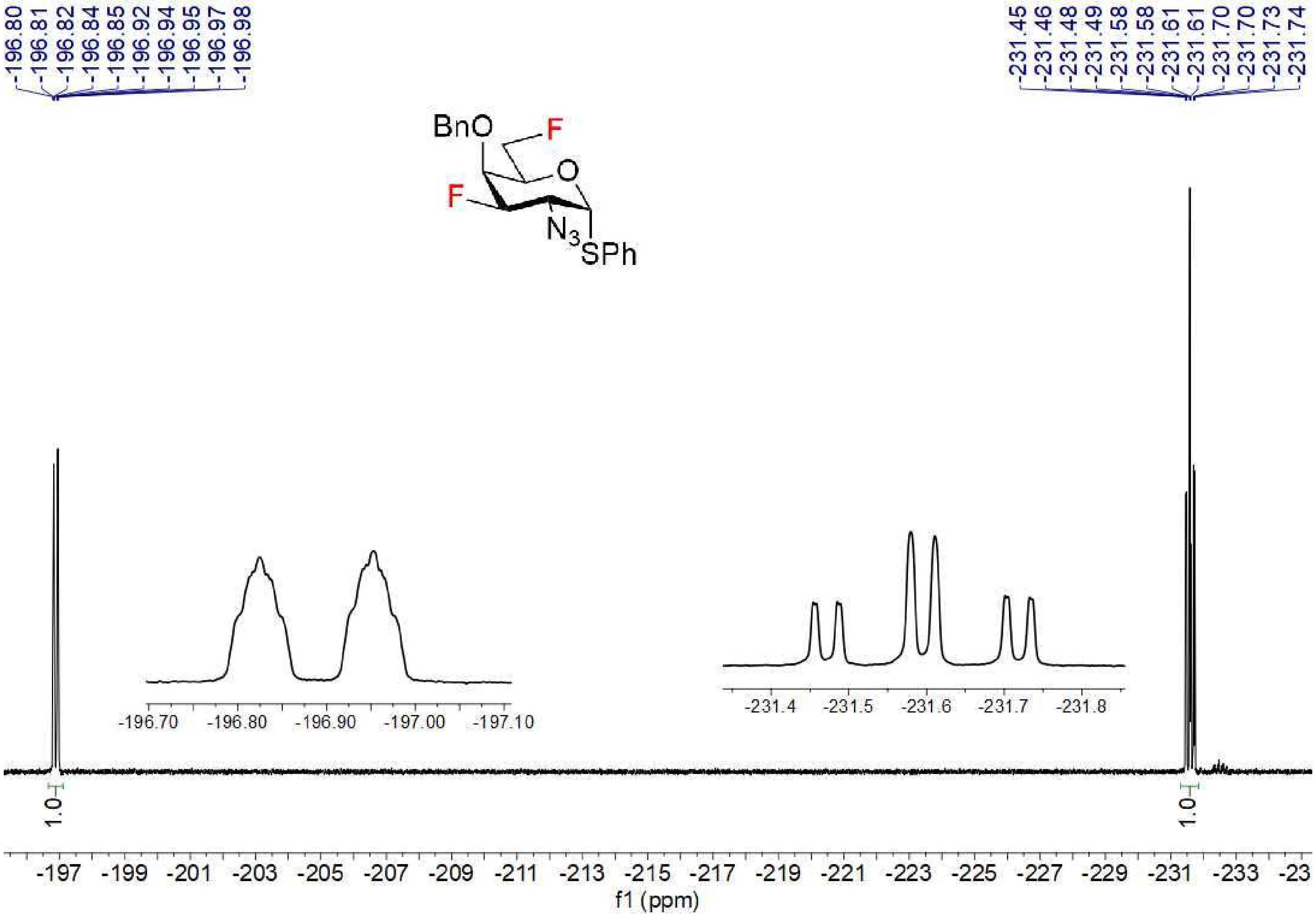
^{1}H NMR (400 MHz, $CDCl_3$) α-24



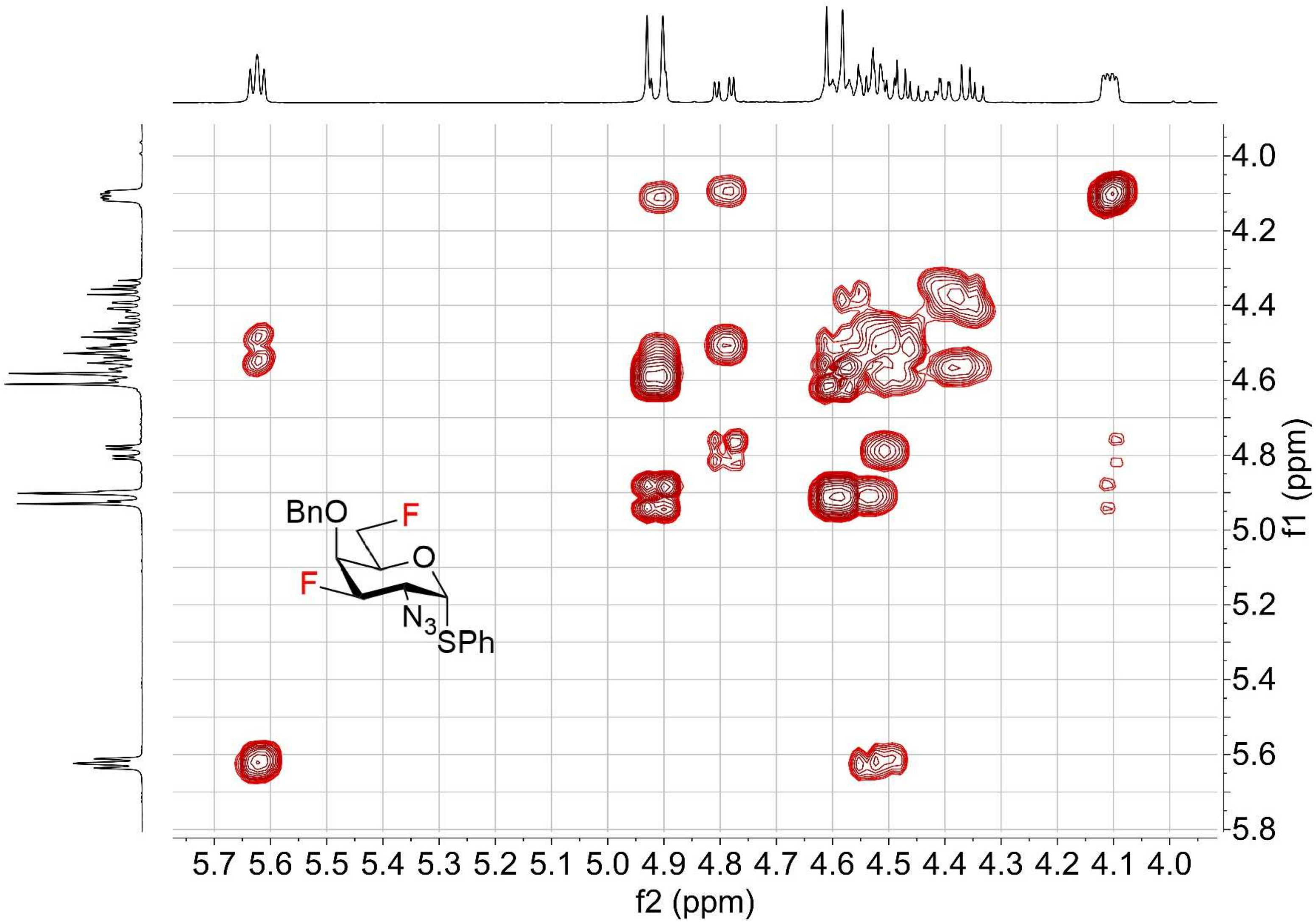
^{13}C NMR (100 MHz, CDCl$_3$) α-24



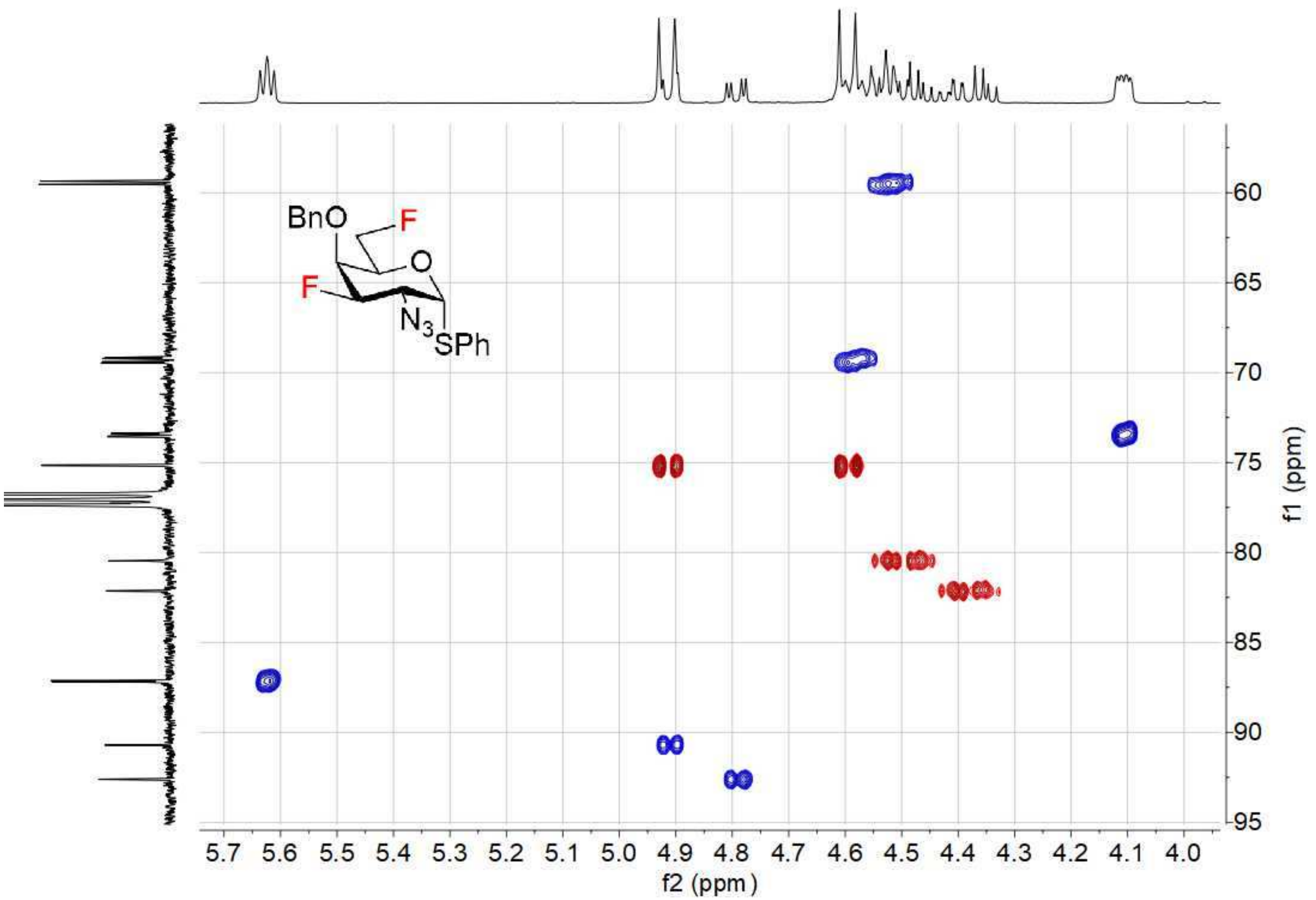
[19]F NMR (376 MHz, CDCl$_3$) α-24



^{1}H-^{1}H COSY α-24

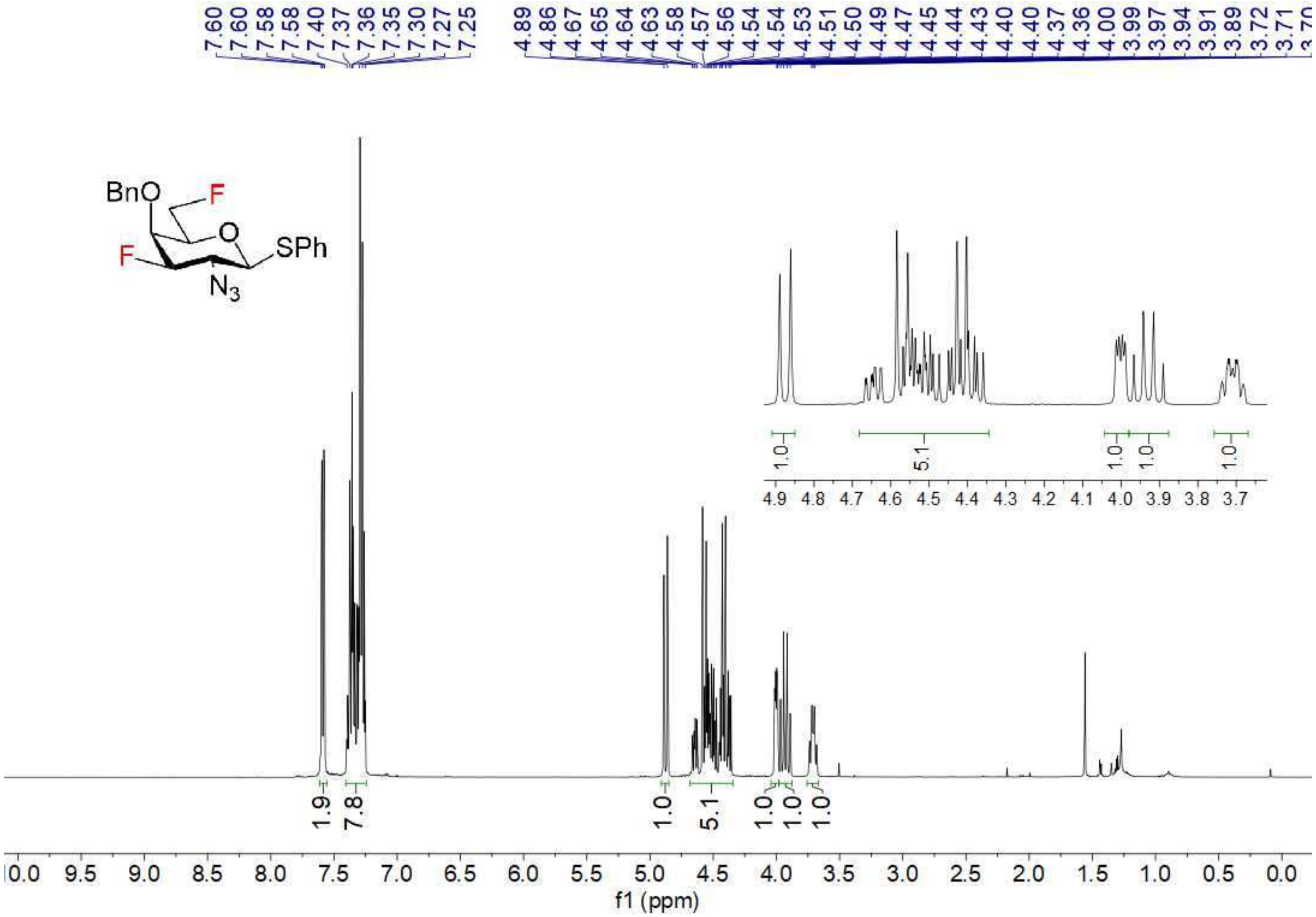


[1]H-[13]C HSQC α-24

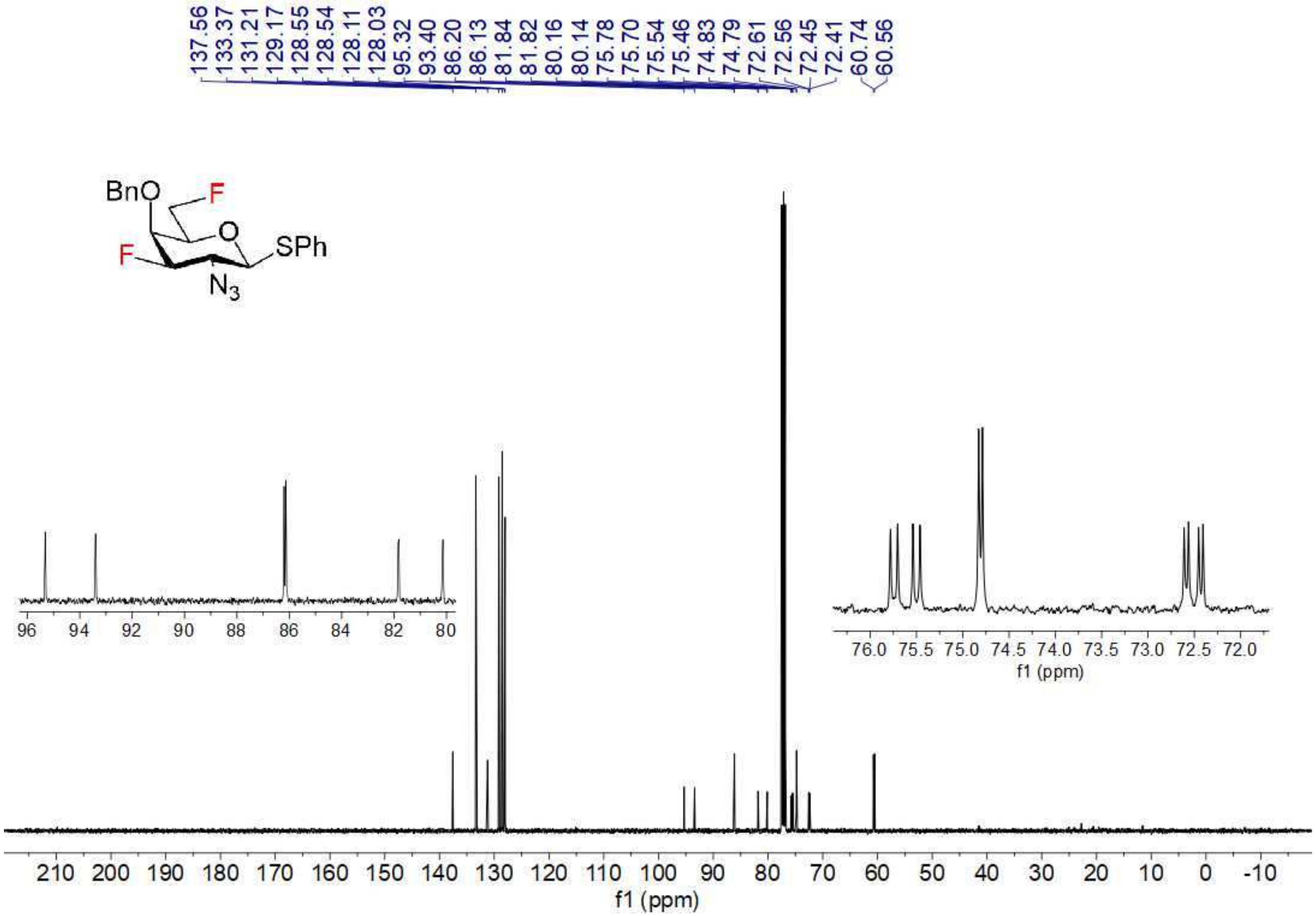


NMR COMPOUND β-24

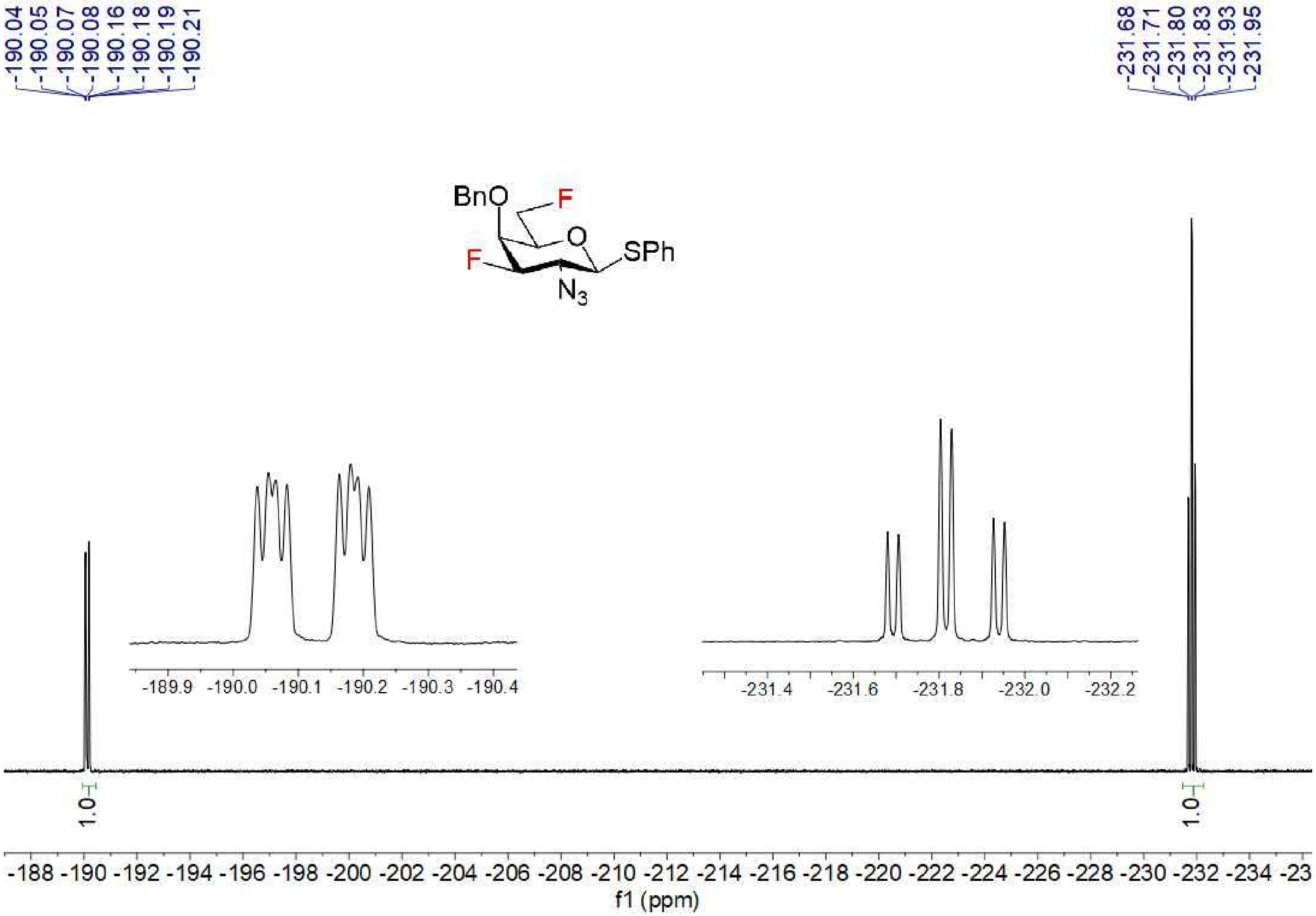
^{1}H NMR (400 MHz, $CDCl_3$) β-24



^{13}C NMR (100 MHz, CDCl$_3$) β-24



^{19}F NMR (376 MHz, CDCl$_3$) β-24



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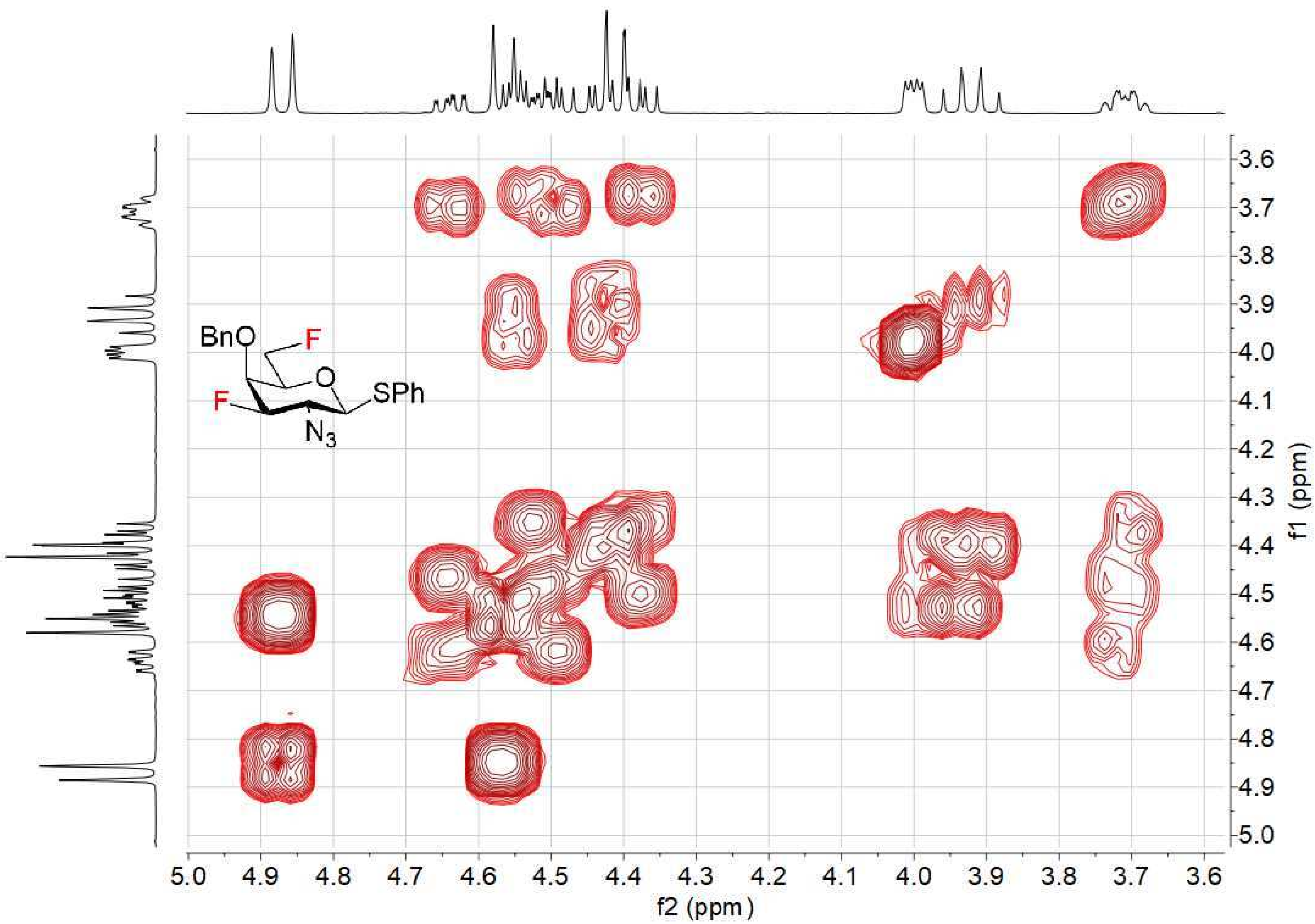

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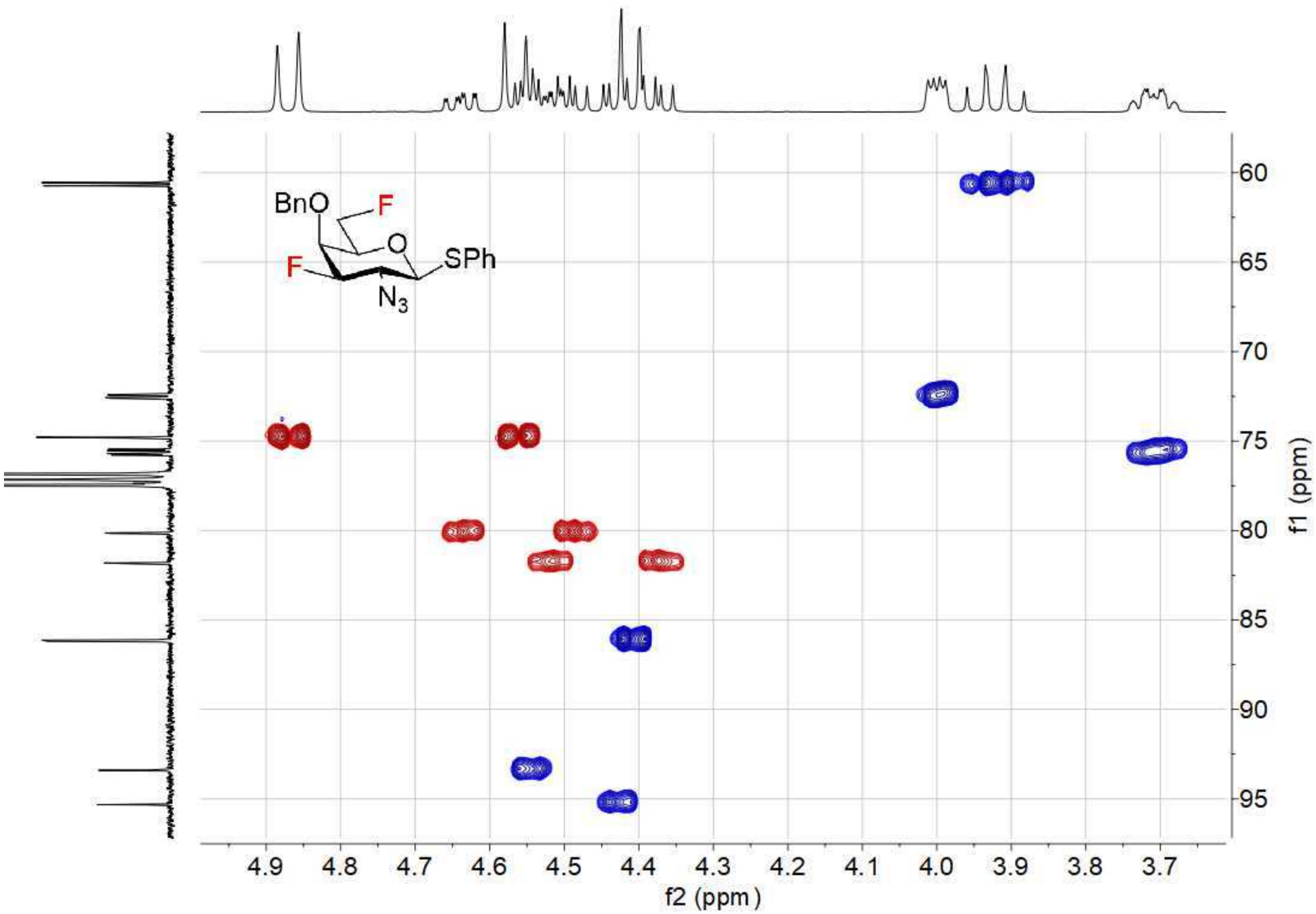
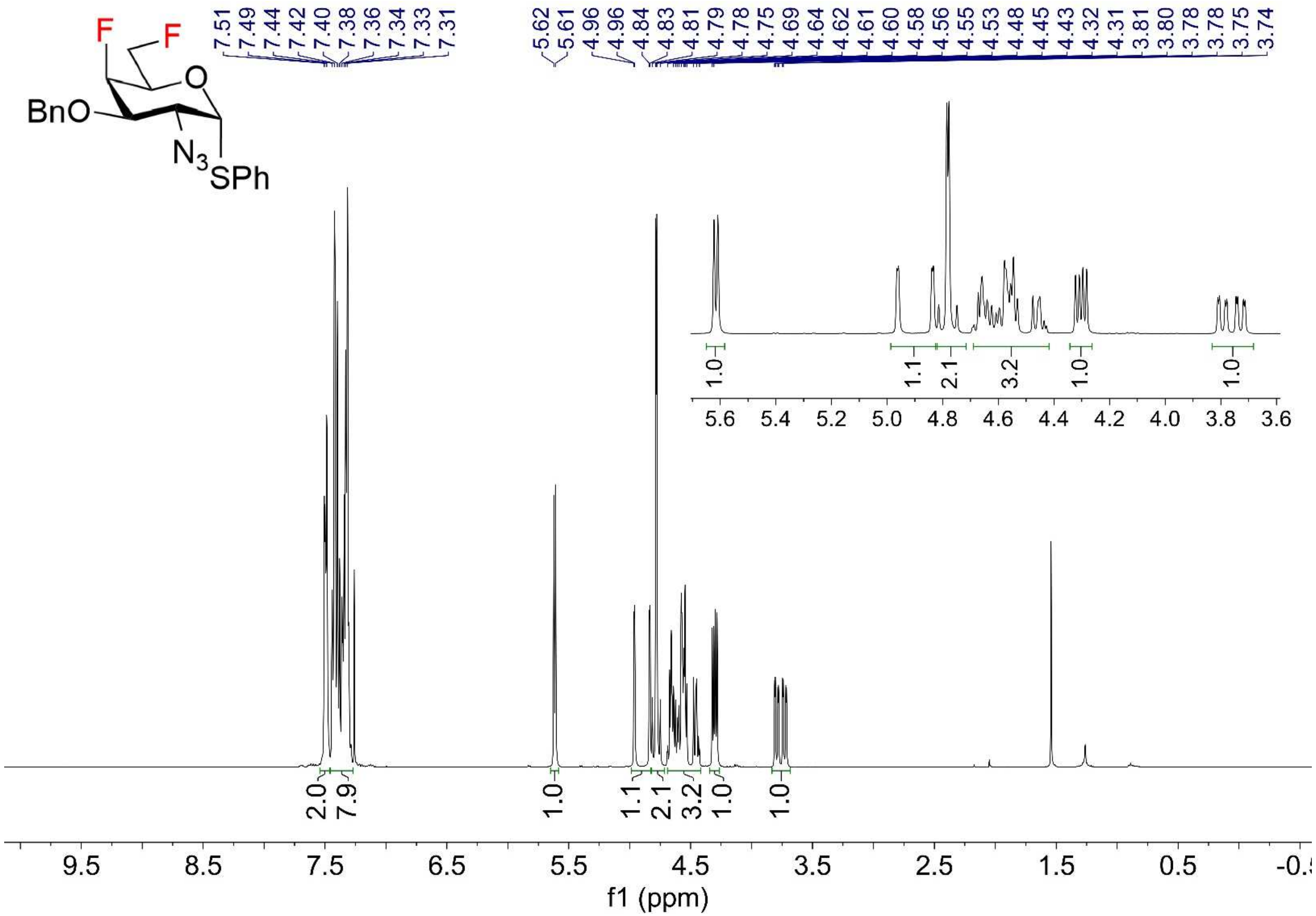
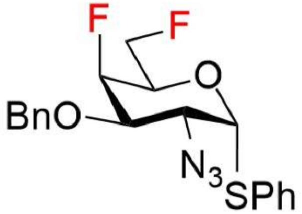


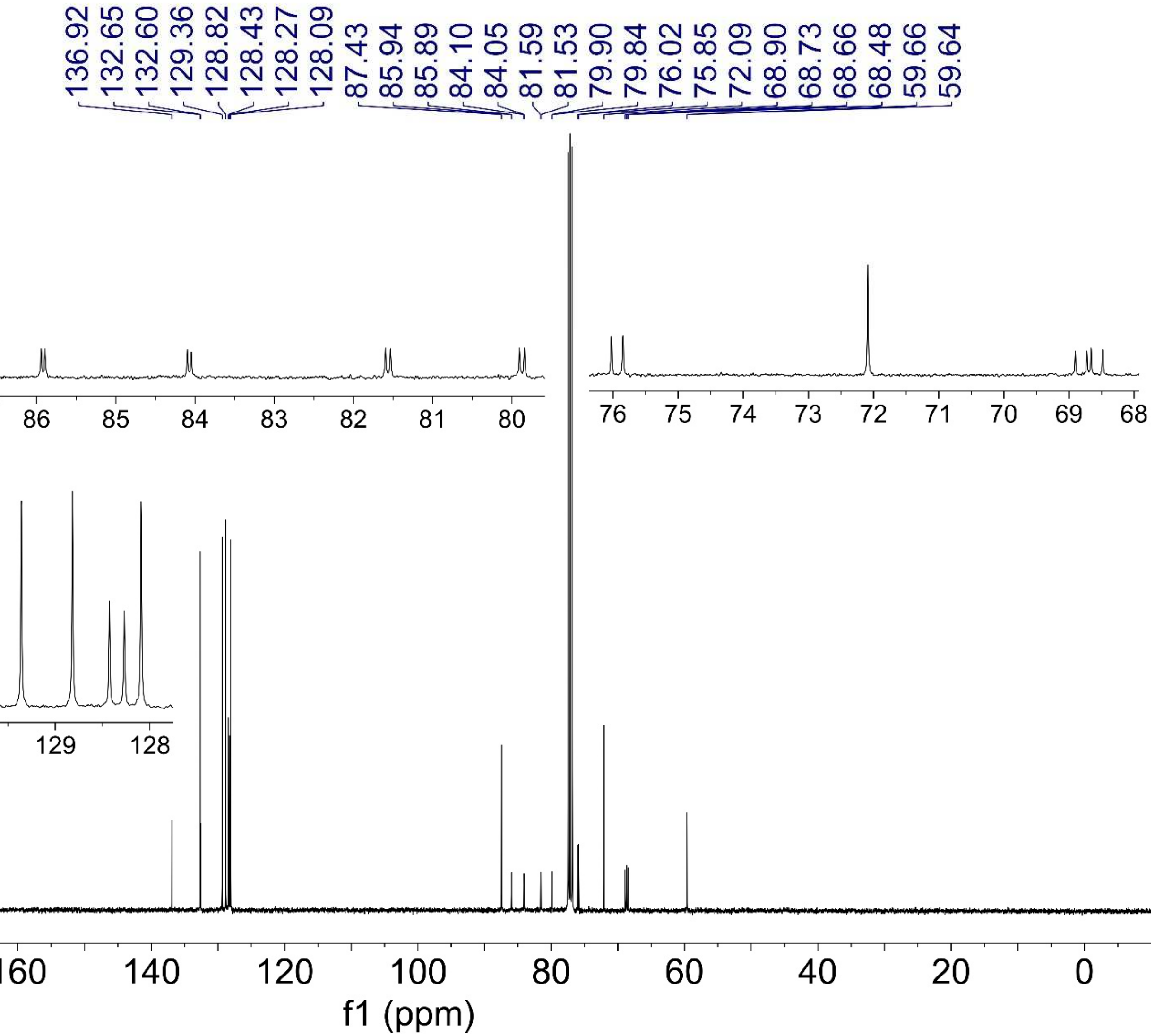
NMR COMPOUND 25

^{1}H NMR (400 MHz, $CDCl_3$) 25

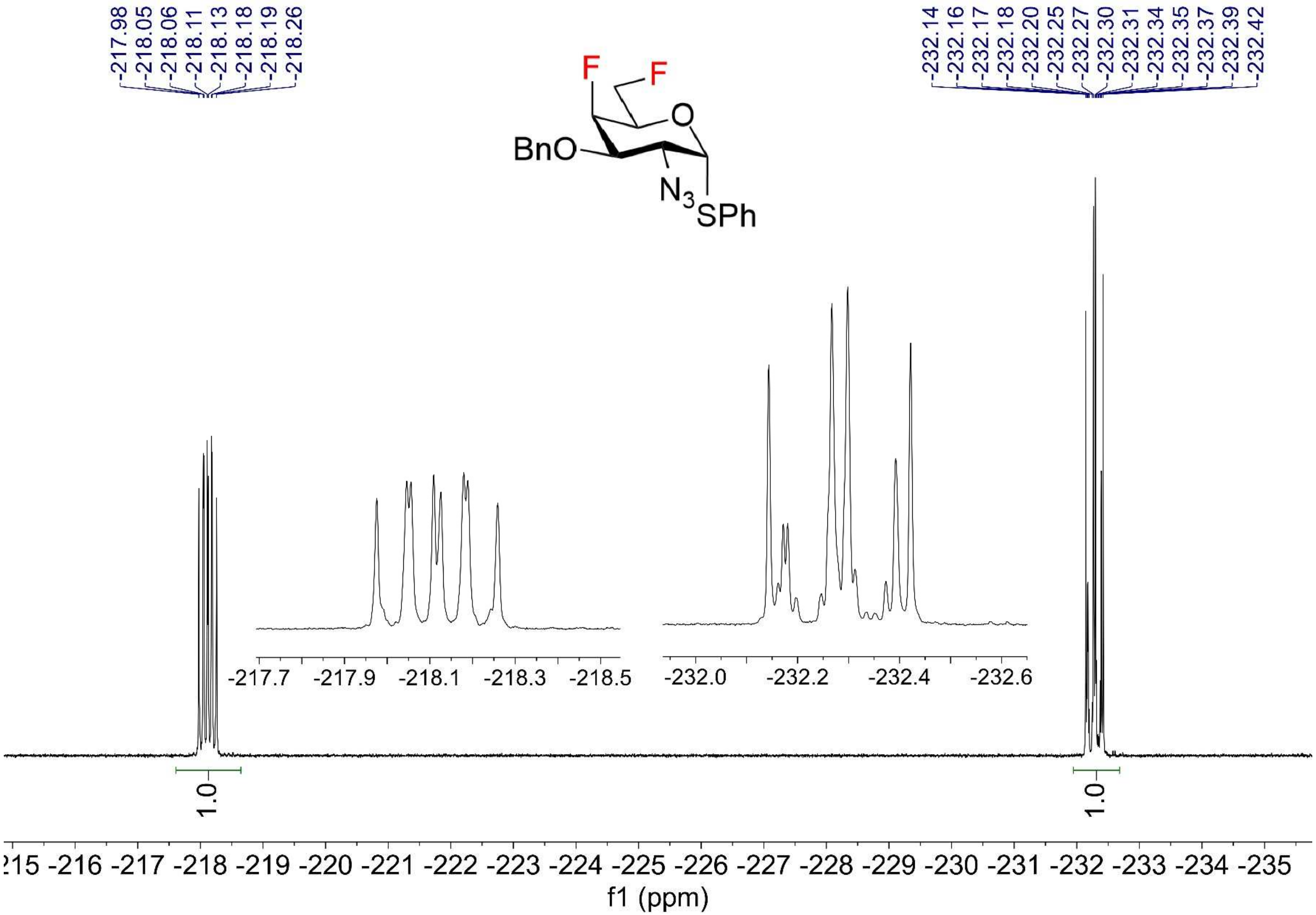


^{13}C NMR (100 MHz, $CDCl_3$) 25





^{19}F NMR (376 MHz, CDCl$_3$) 25



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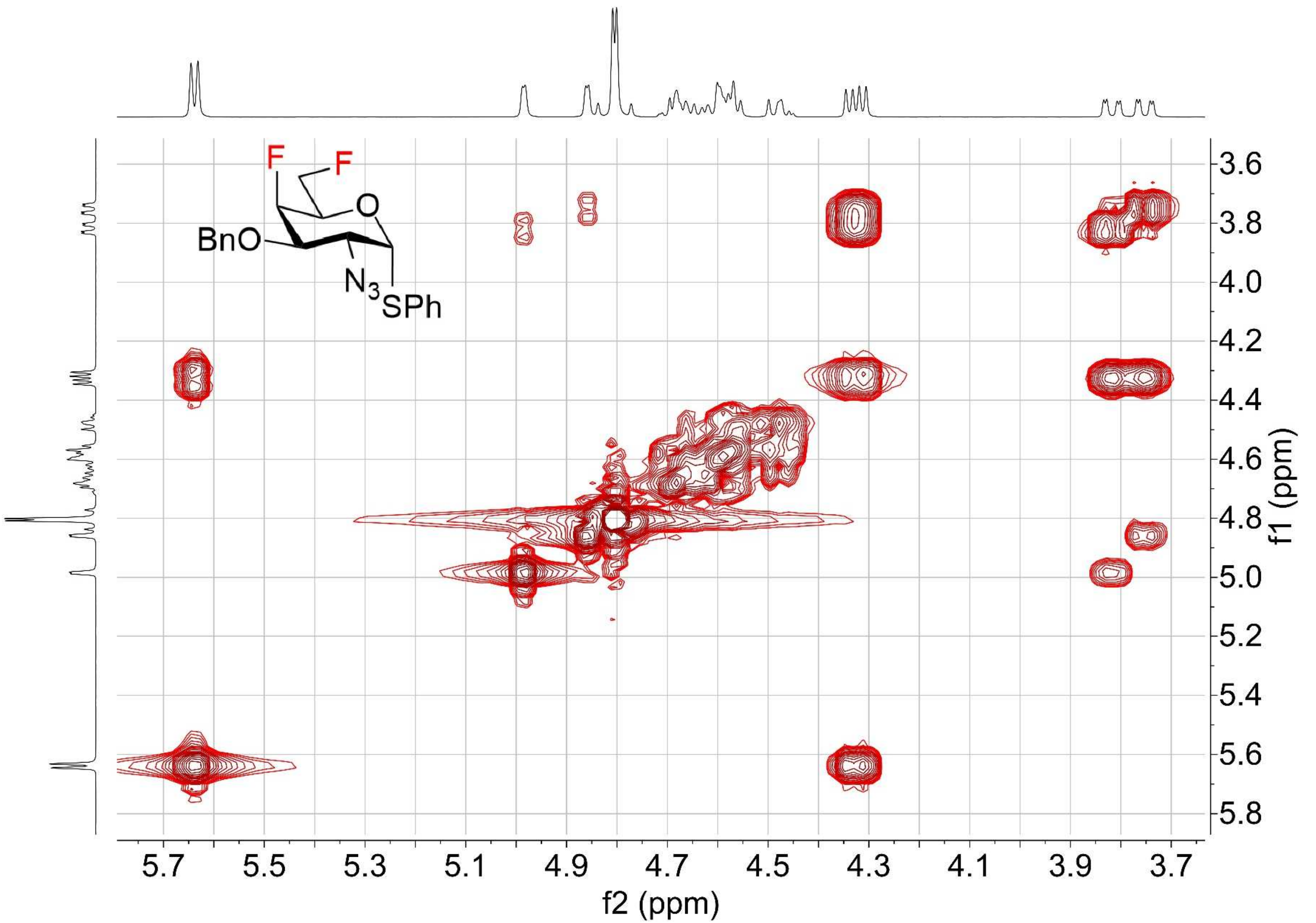
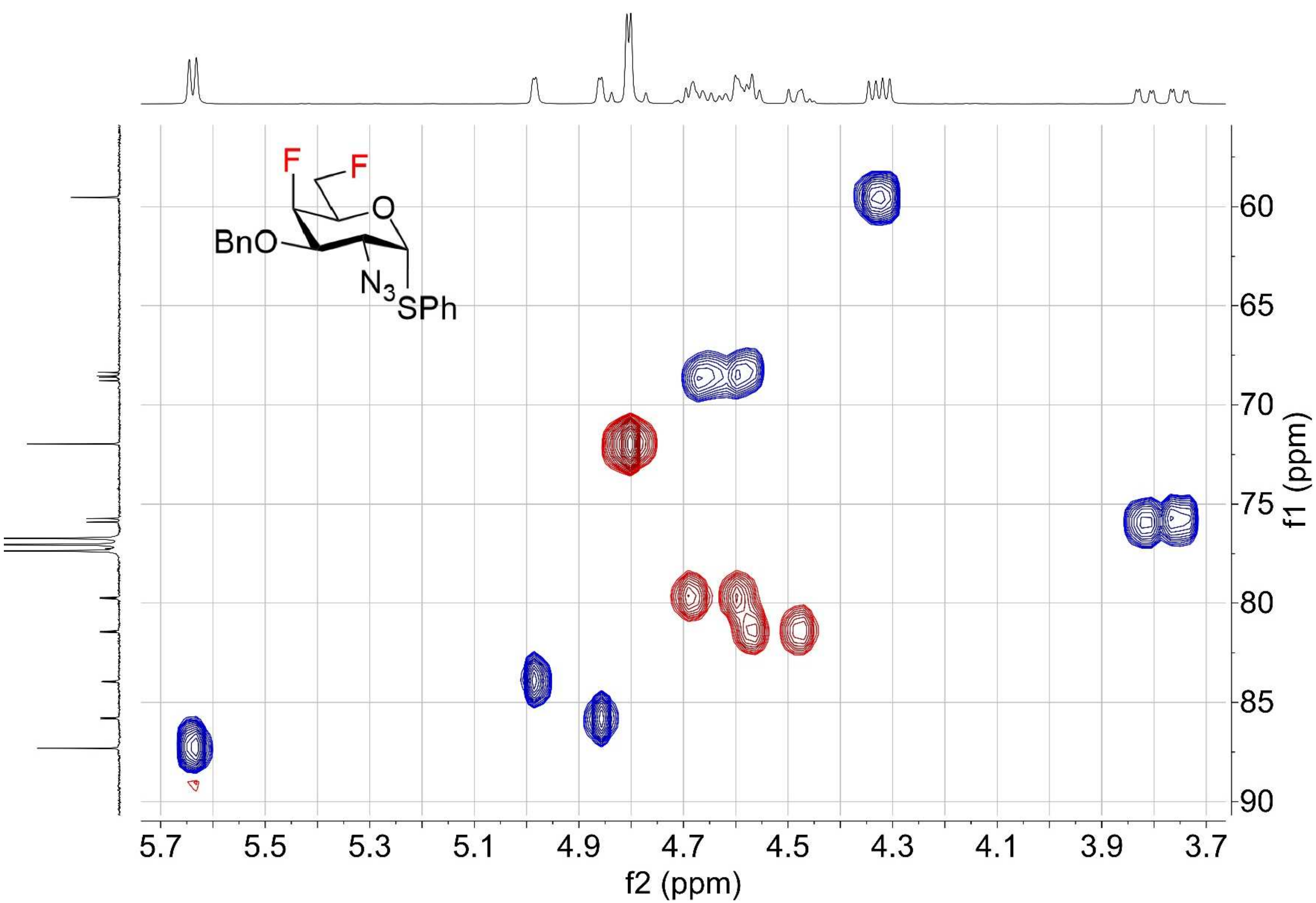


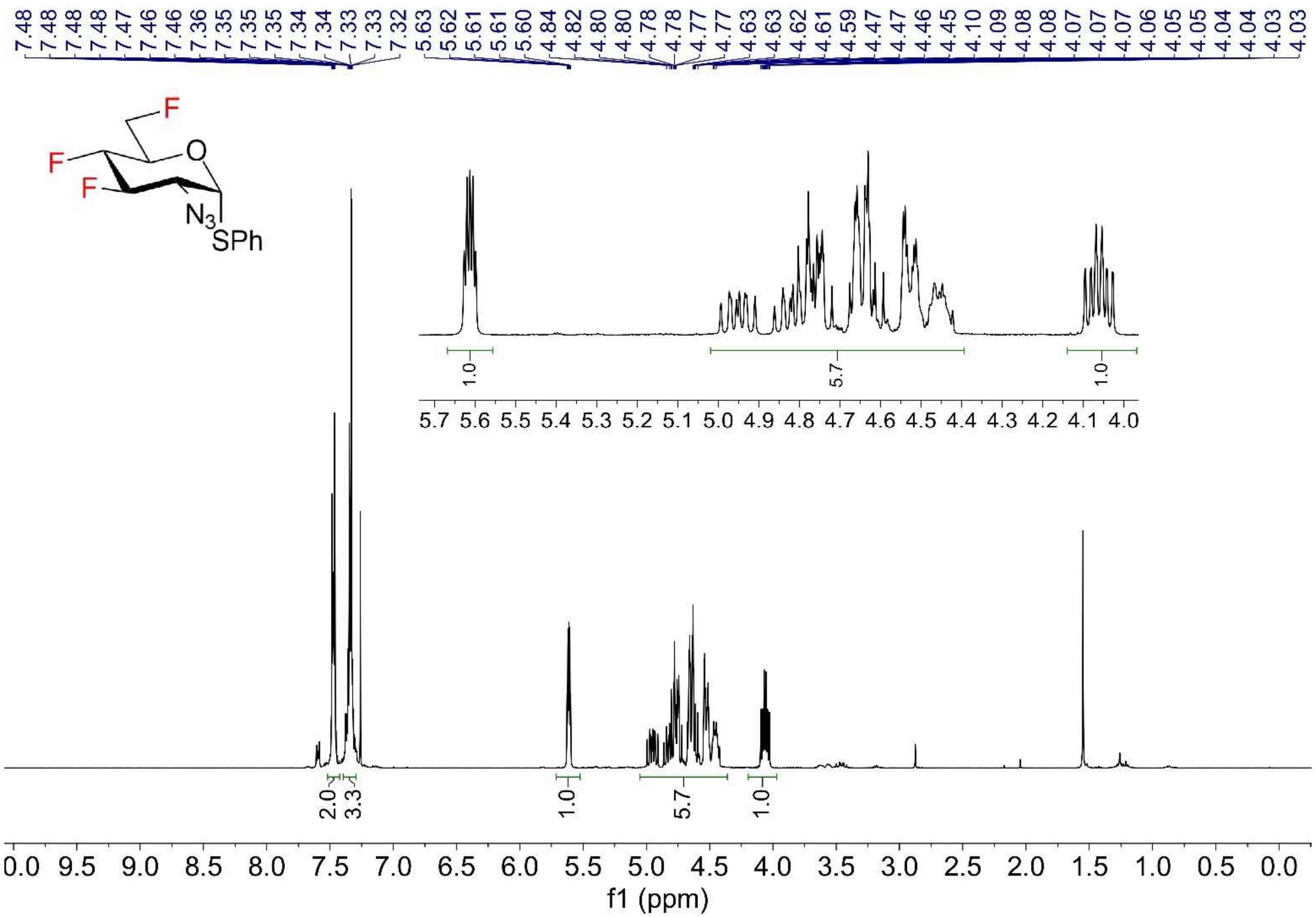
F
F
O
BnO
N3
SPh
3.6
3.8
4.0
4.2
4.4
4.6
4.8
5.0
5.2
5.4
5.6
5.8
f1 (ppm)
5.7
5.5
5.3
5.1
4.9
4.7
4.5
4.3
4.1
3.9
3.7
f2 (ppm)

^{1}H-^{13}C HSQC 25

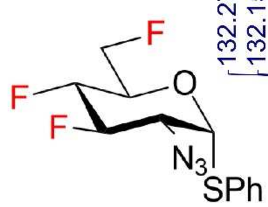


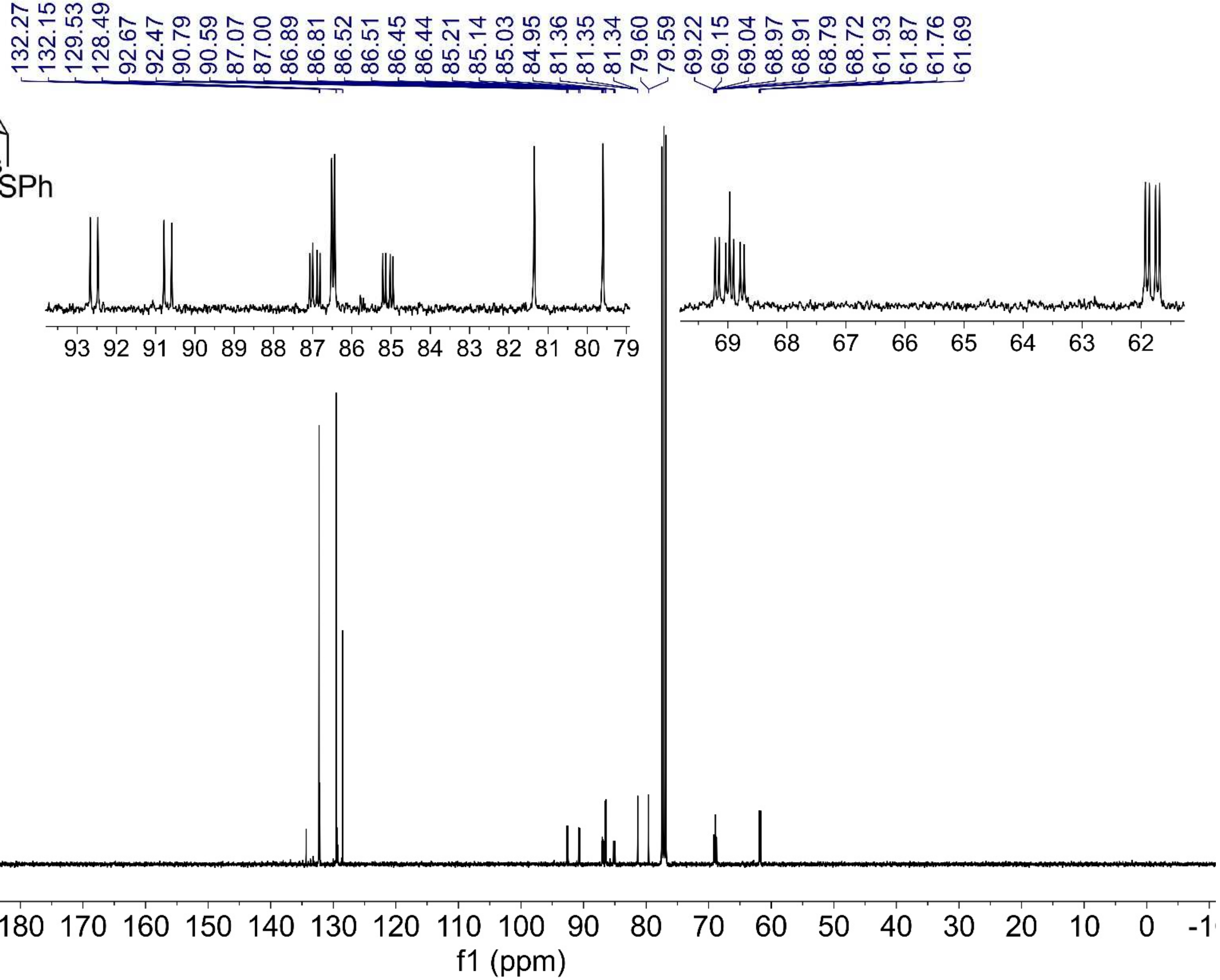
NMR COMPOUND 26

^{1}H NMR (400 MHz, CDCl$_3$) 26

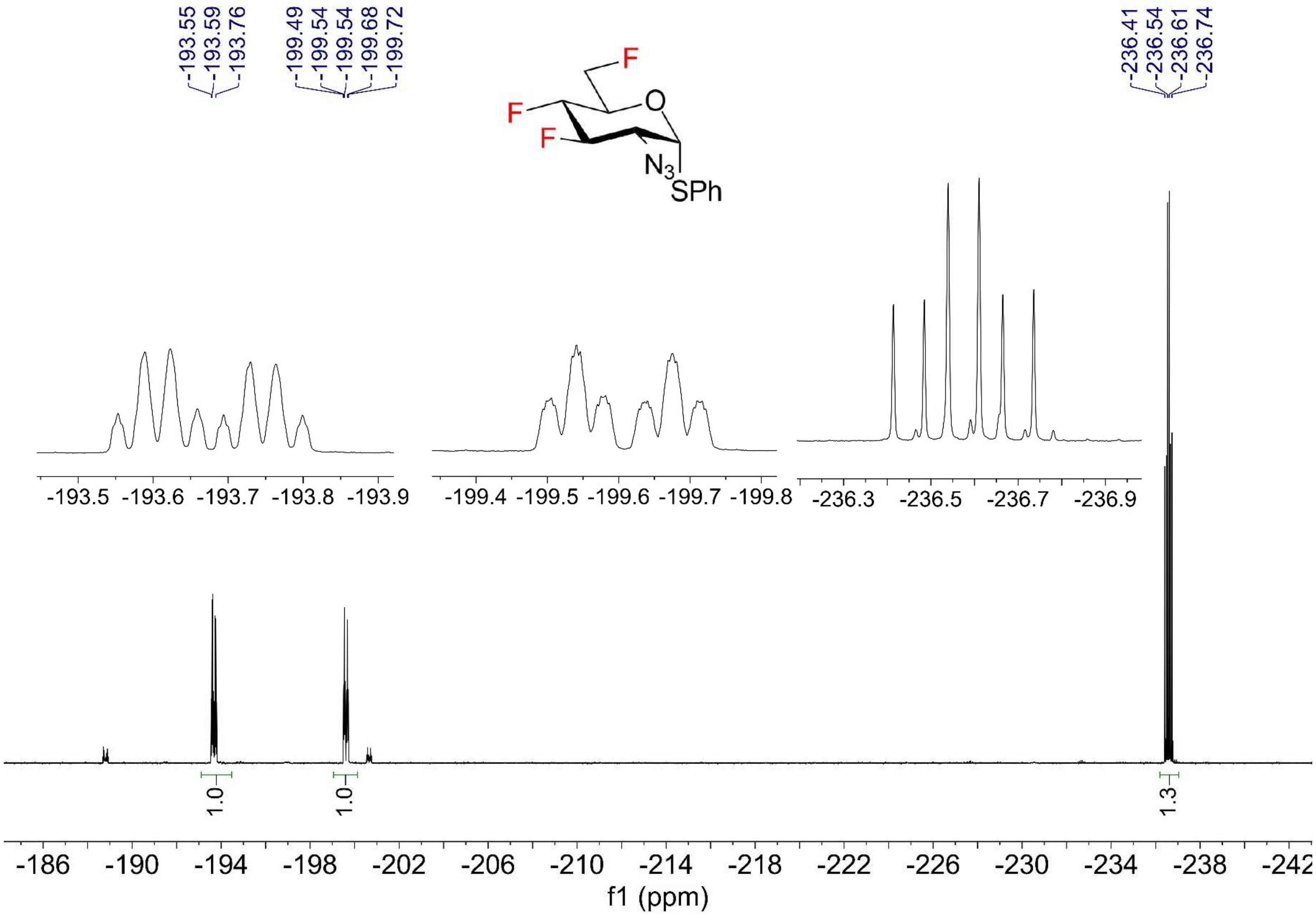


^{13}C NMR (100 MHz, CDCl$_3$) 26

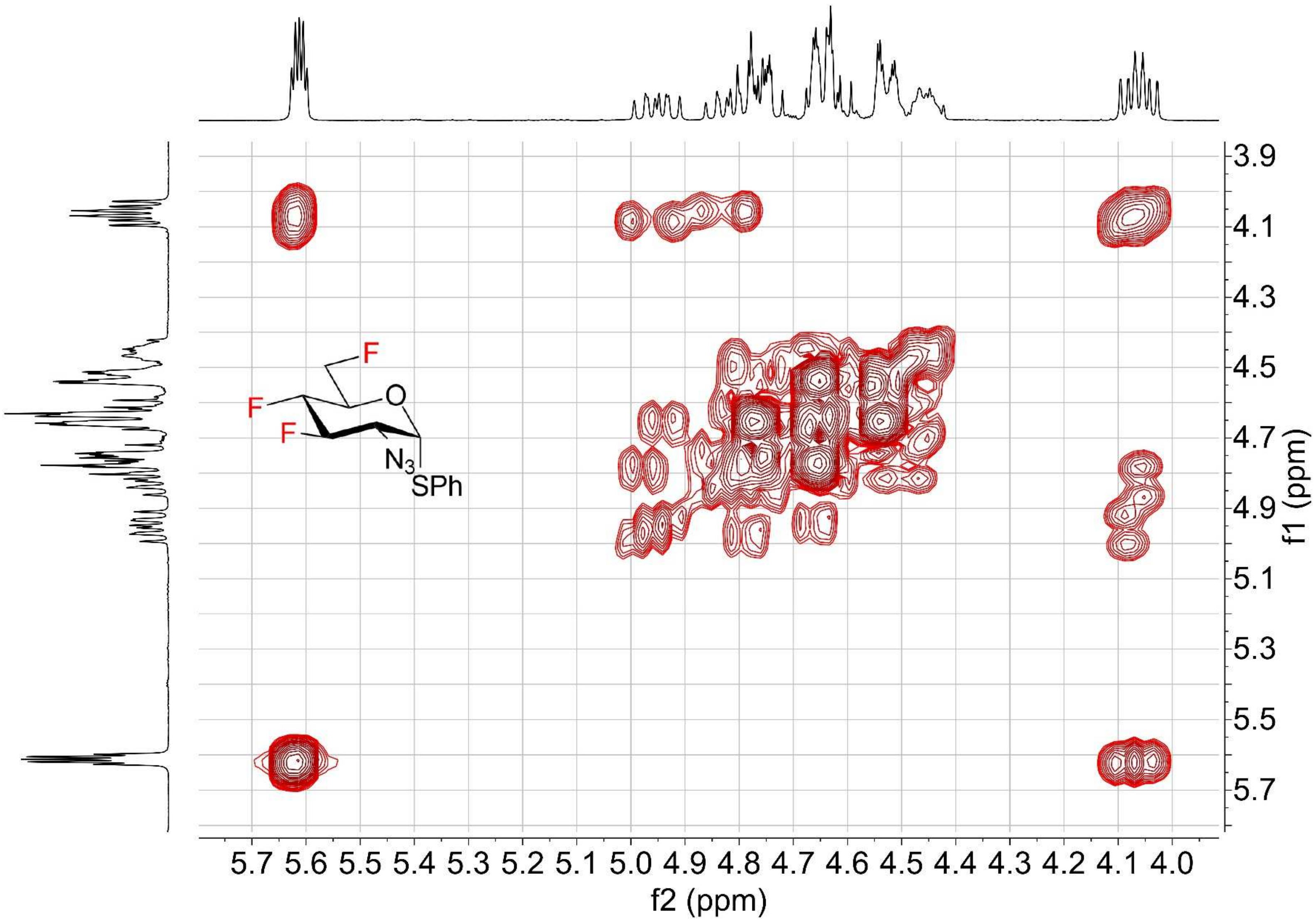




^{19}F NMR (376 MHz, $CDCl_3$) 26

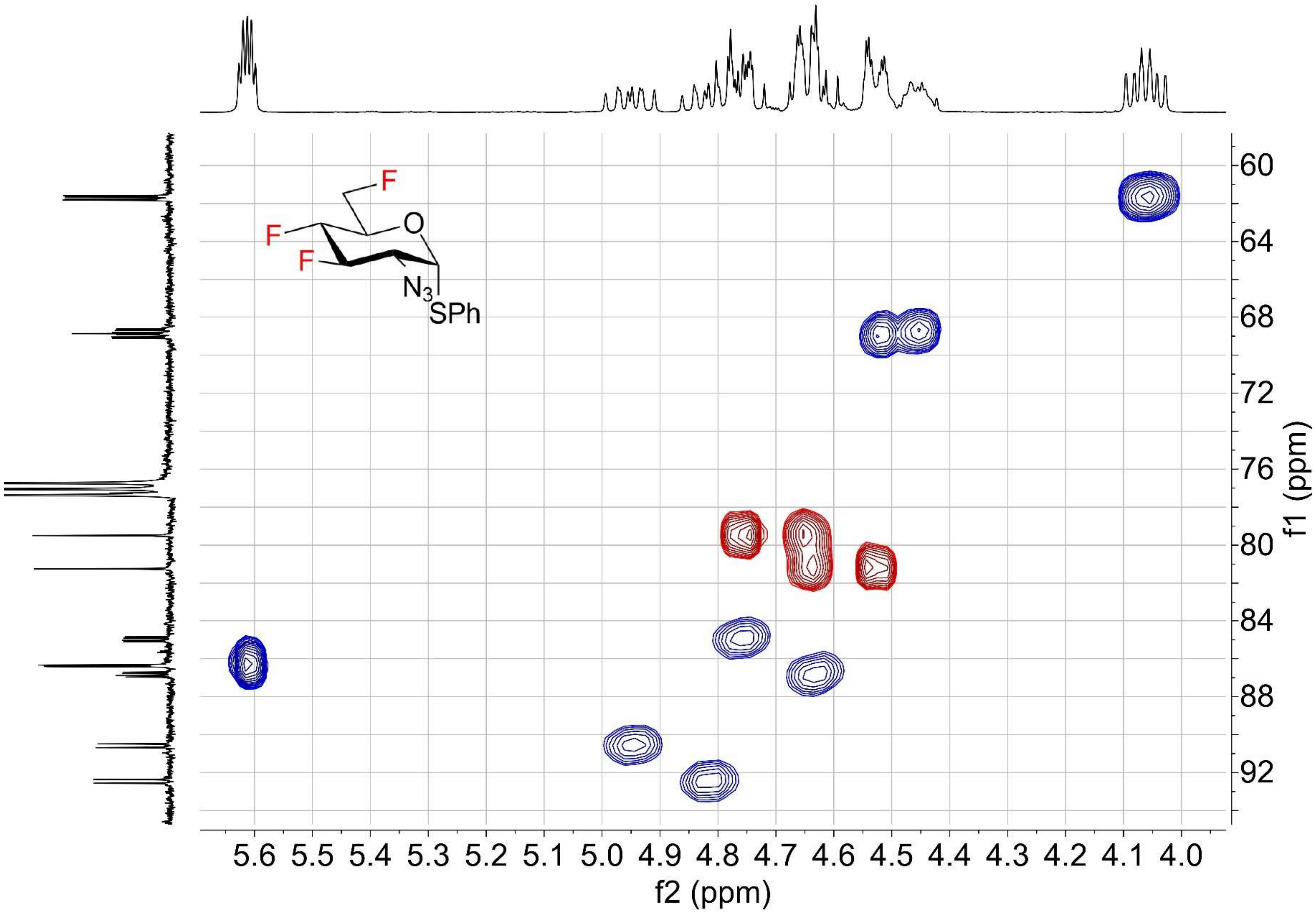


[1]H-[1]H COSY 26



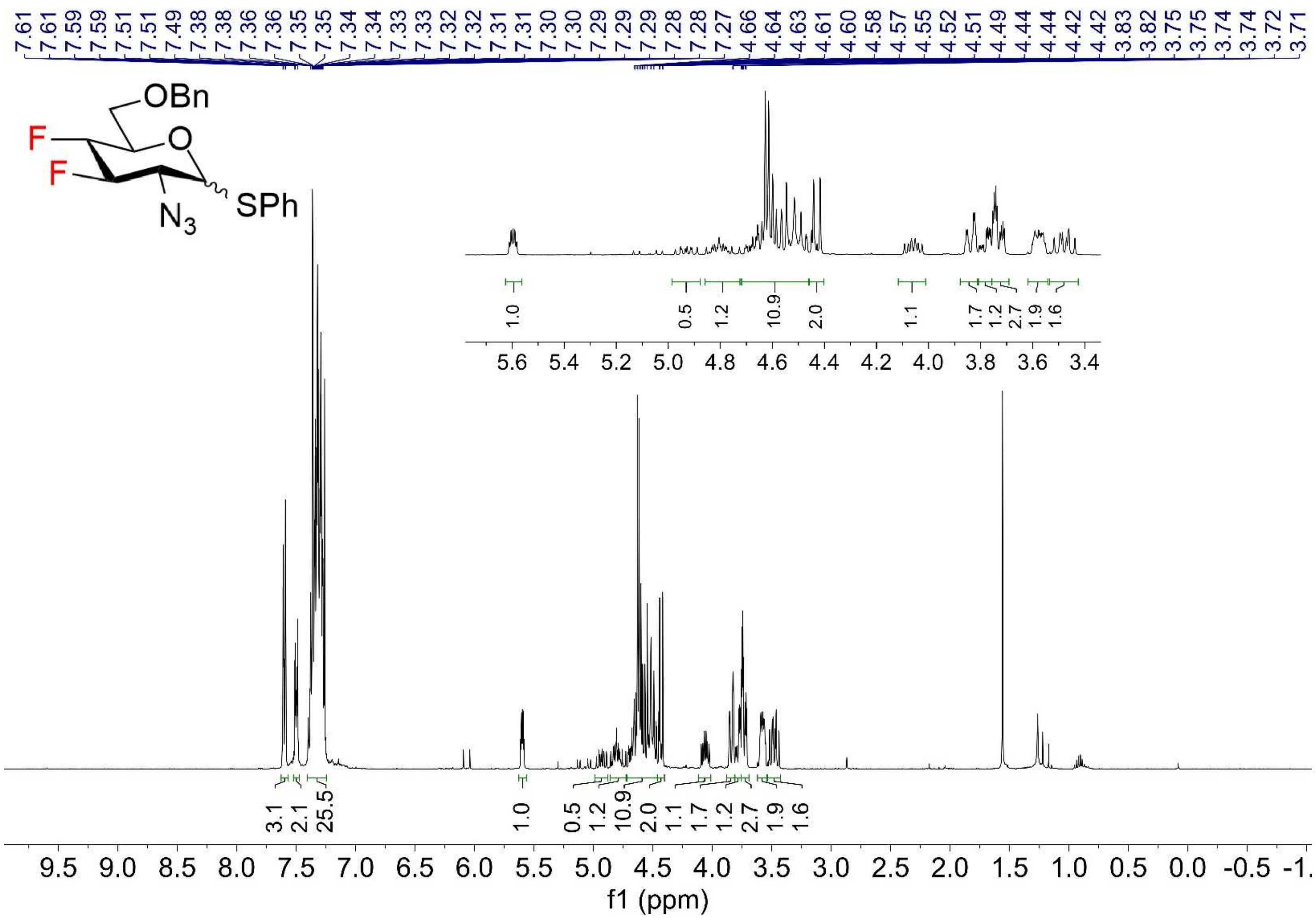
F
F
O
F
N3
SPh
5.7 5.6 5.5 5.4 5.3 5.2 5.1 5.0 4.9 4.8 4.7 4.6 4.5 4.4 4.3 4.2 4.1 4.0
f2 (ppm)
3.9
4.1
4.3
4.5
4.7
4.9
5.1
5.3
5.5
5.7
f1 (ppm)

^{1}H-^{13}C HSQC 26

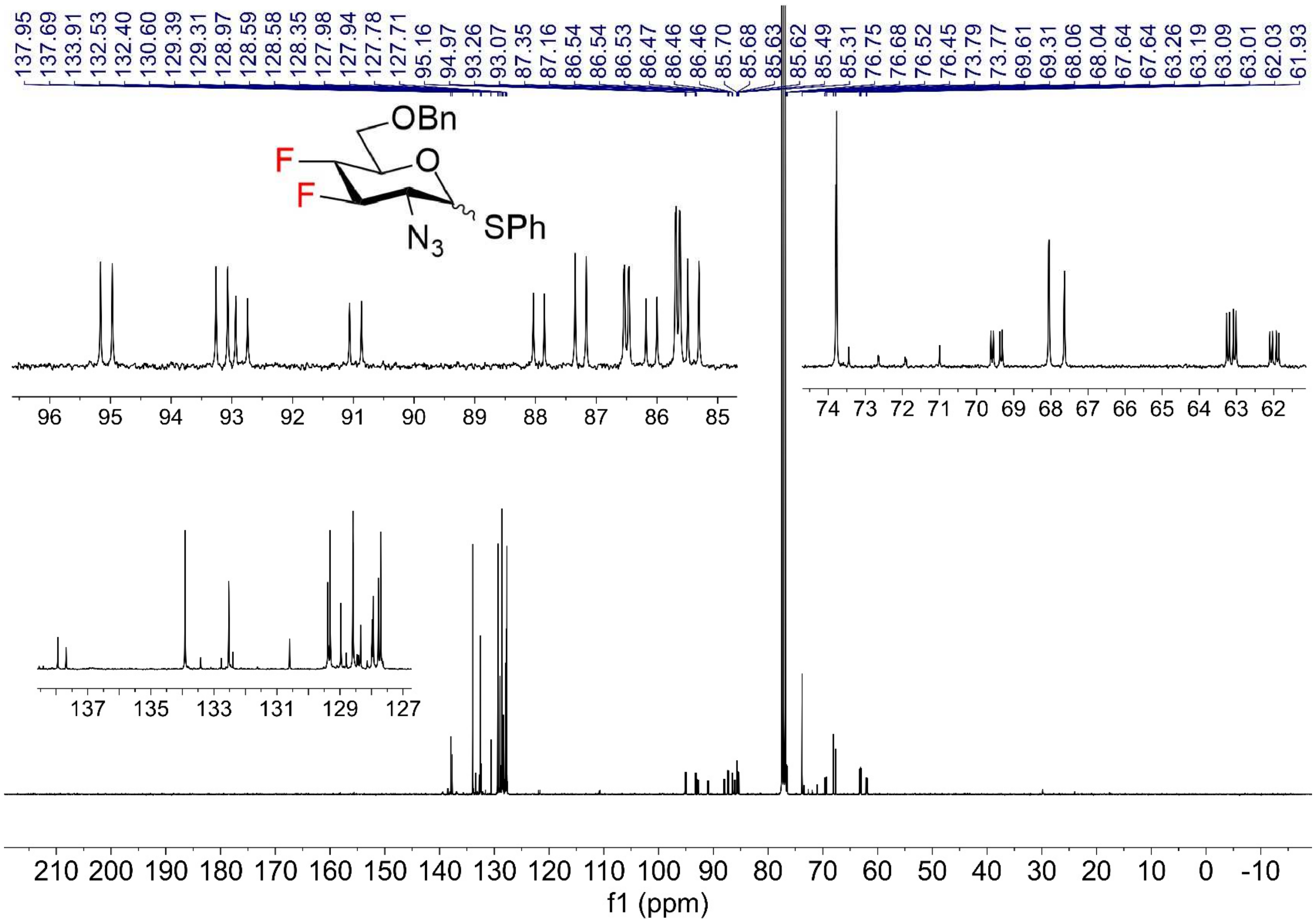


NMR COMPOUND 31

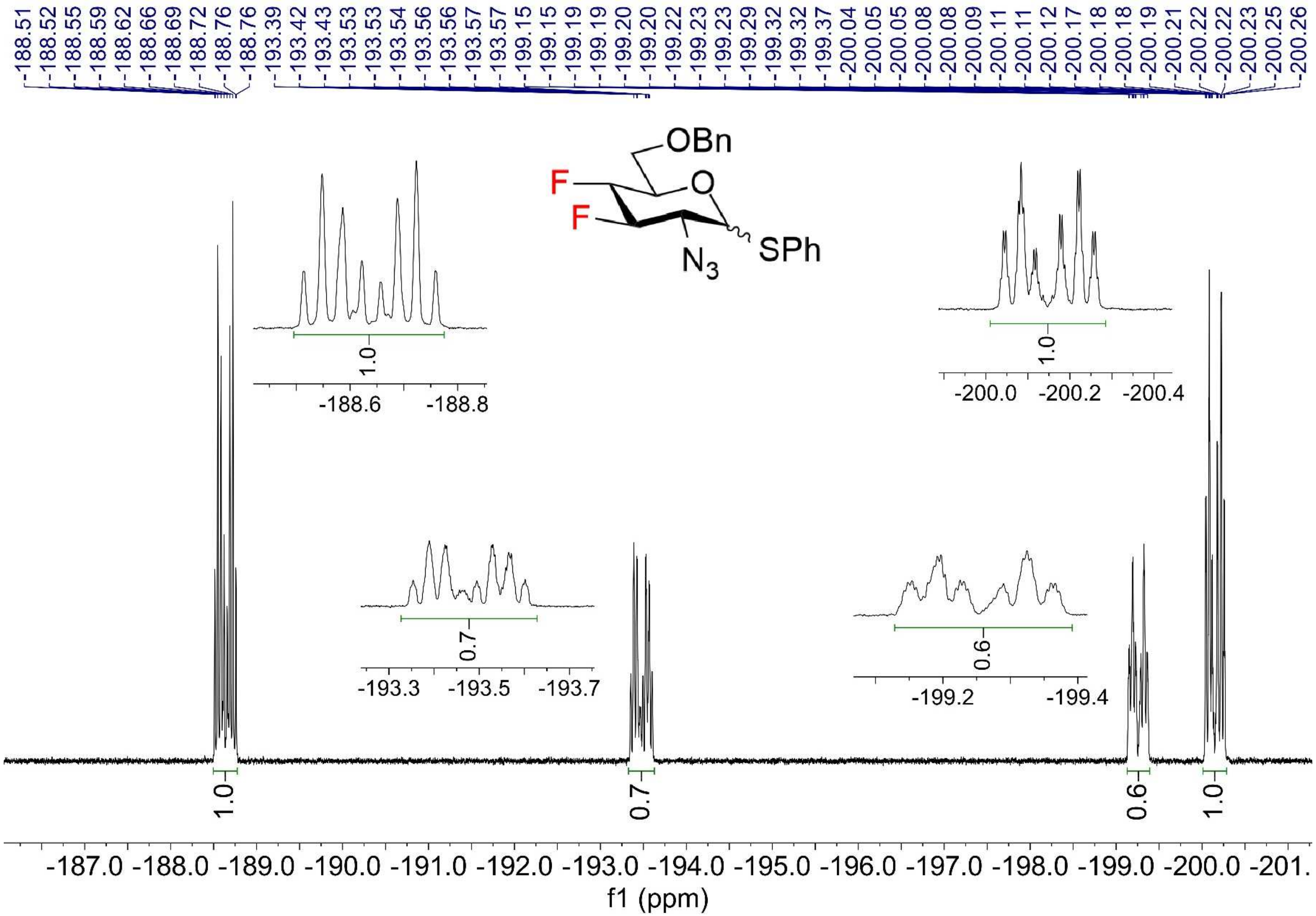
^{1}H NMR (400 MHz, $CDCl_3$) 31



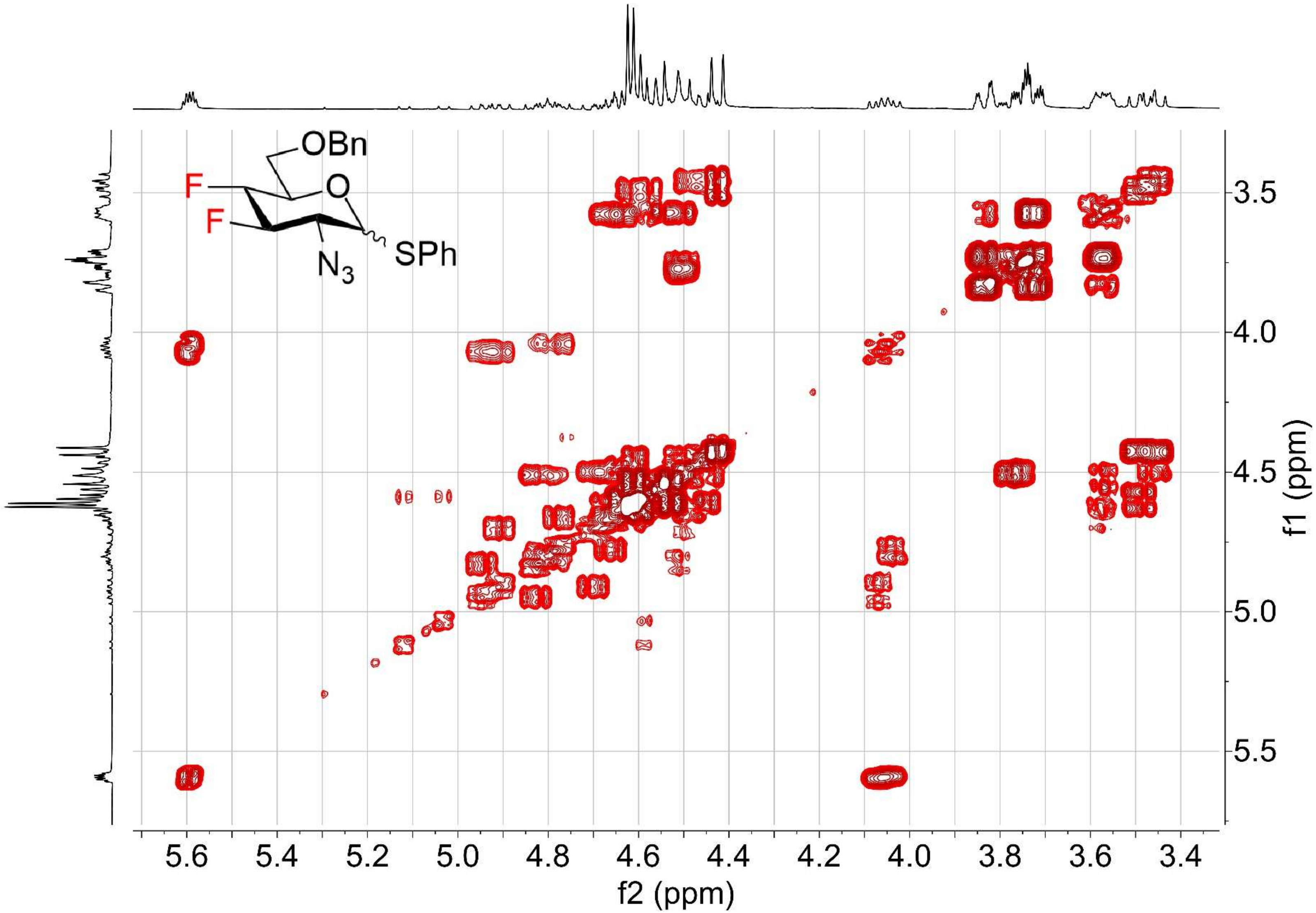
^{13}C NMR (100 MHz, CDCl$_3$) 31



^{19}F NMR (376 MHz, CDCl$_3$) 31

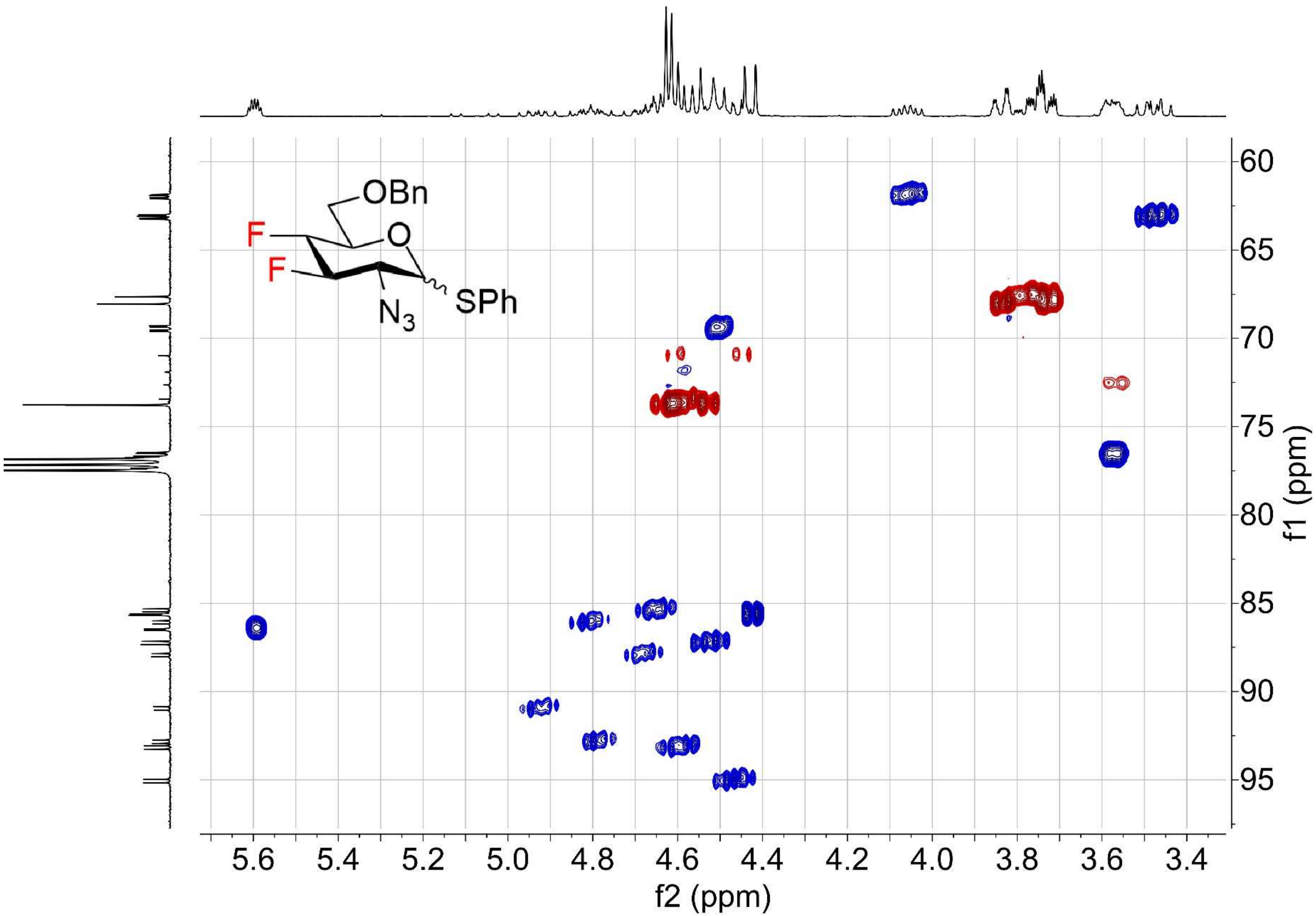


^{1}H-^{1}H COSY 31

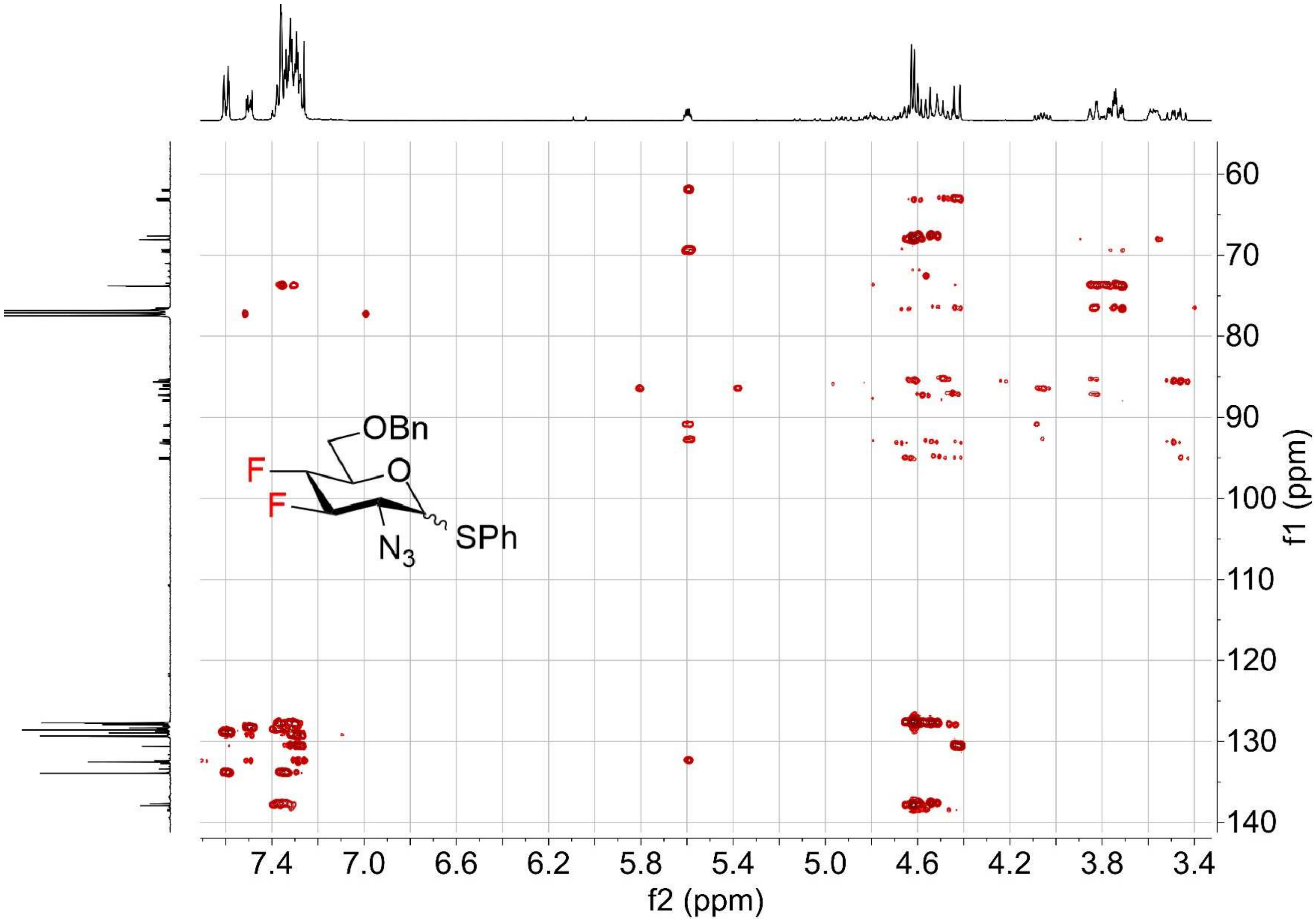


OBn
F
F
O
SPh
N_3
5.6
5.4
5.2
5.0
4.8
4.6
4.4
4.2
4.0
3.8
3.6
3.4
f2 (ppm)
3.5
4.0
4.5
5.0
5.5
f1 (ppm)

^{1}H-^{13}C HSQC 31

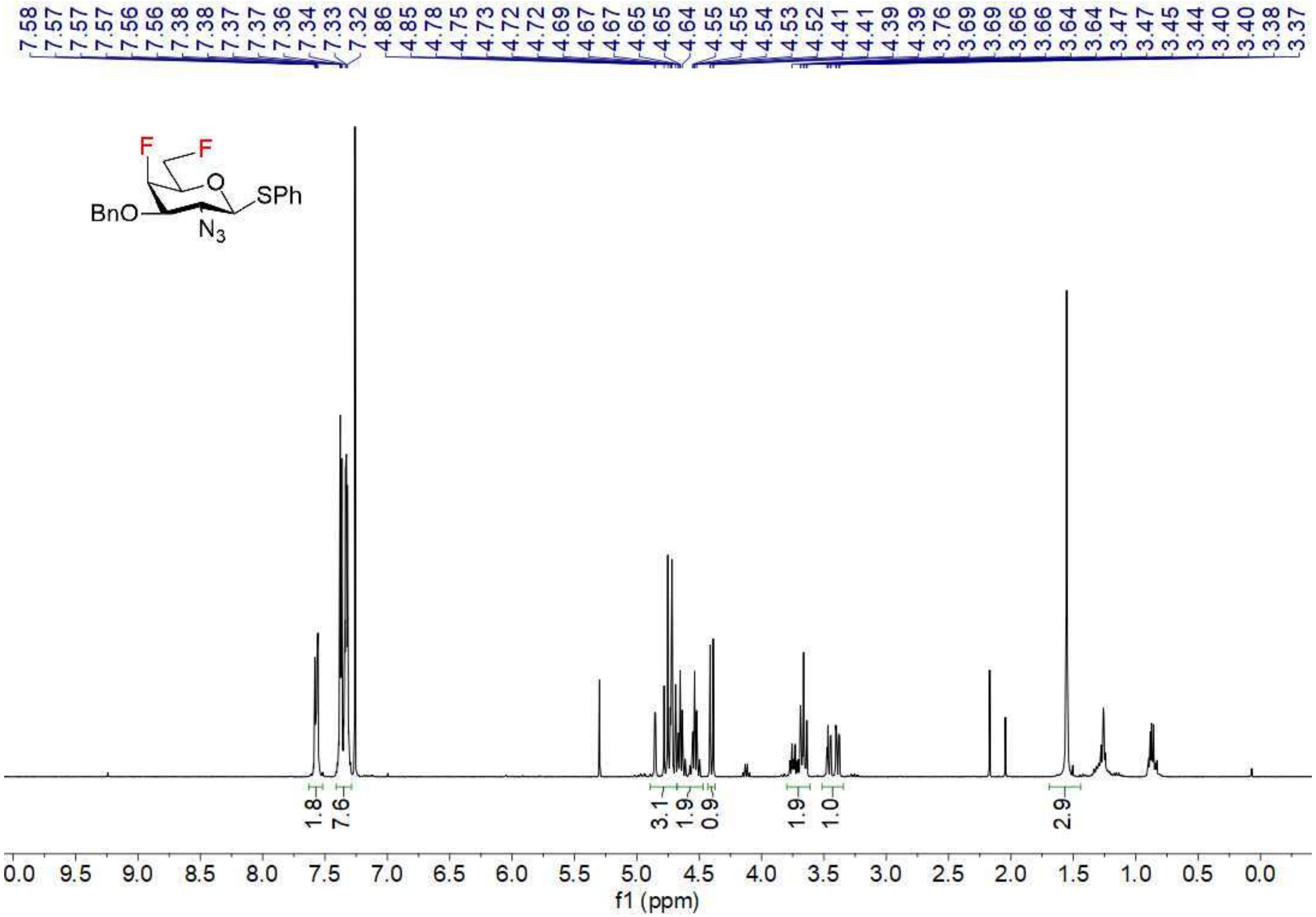


^1H-^{13}C HMBC 31

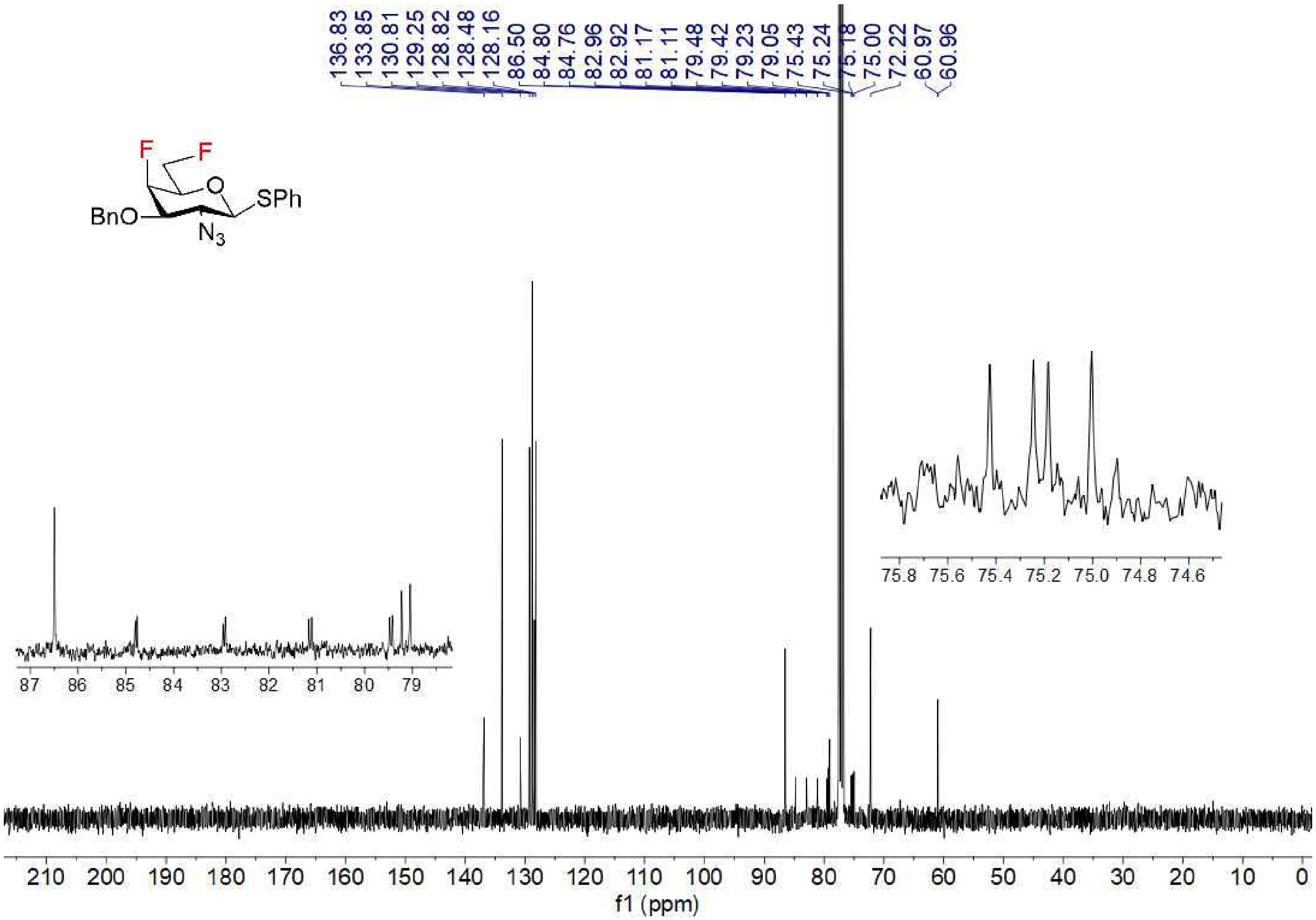


NMR COMPOUND β-25

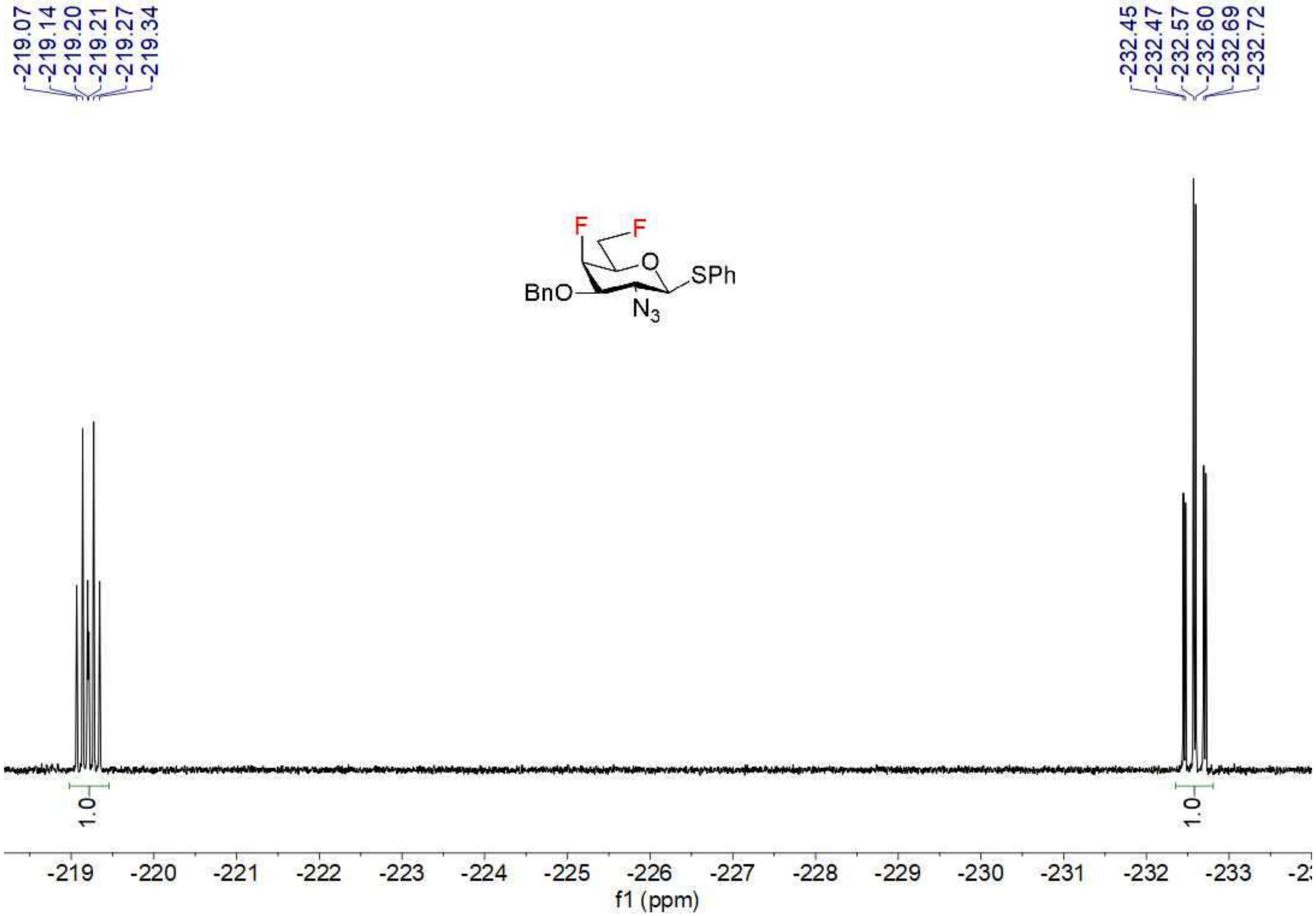
^{1}H NMR (400 MHz, $CDCl_3$) β-25



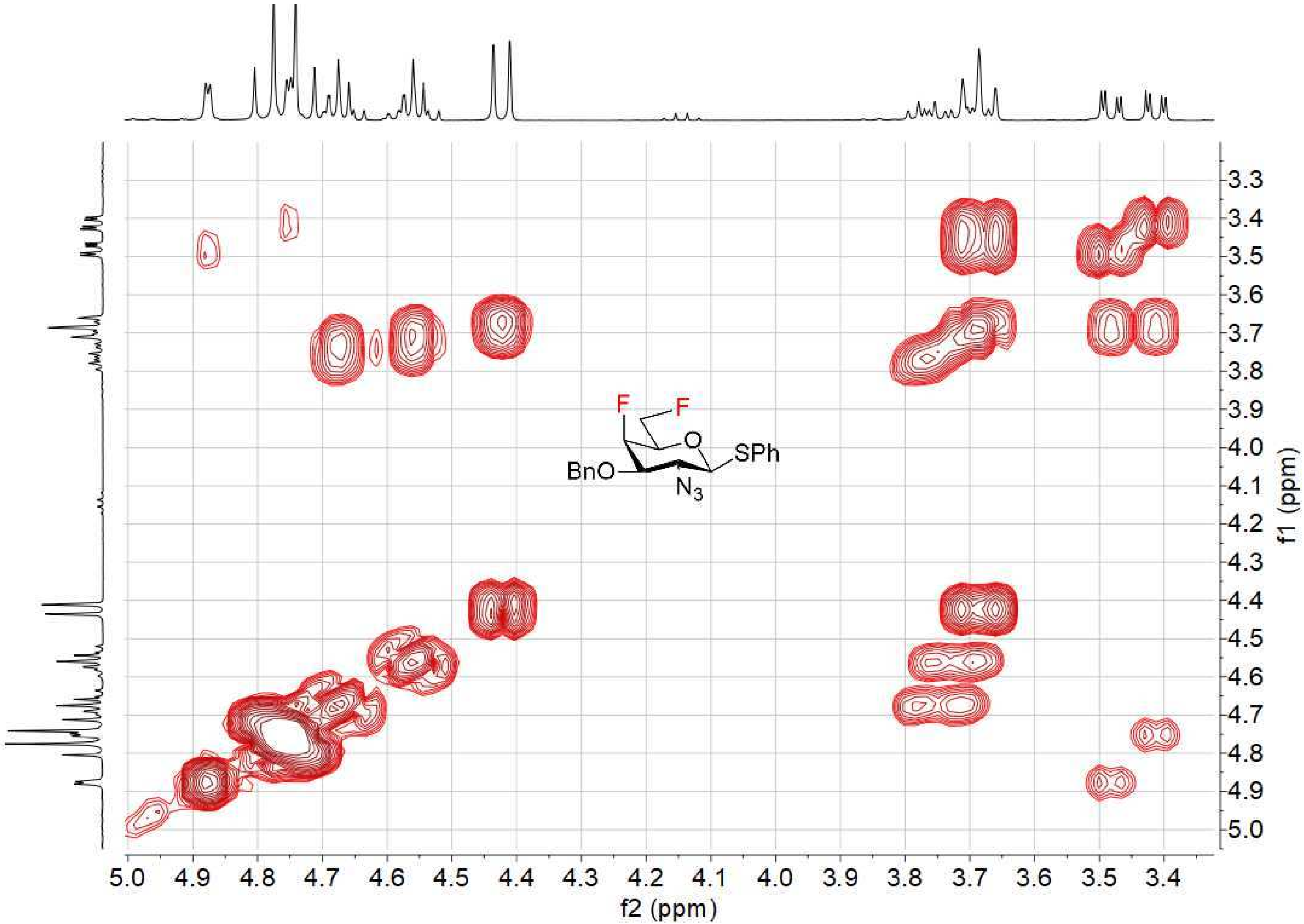
^{13}C NMR (100 MHz, CDCl$_3$) β-**25**



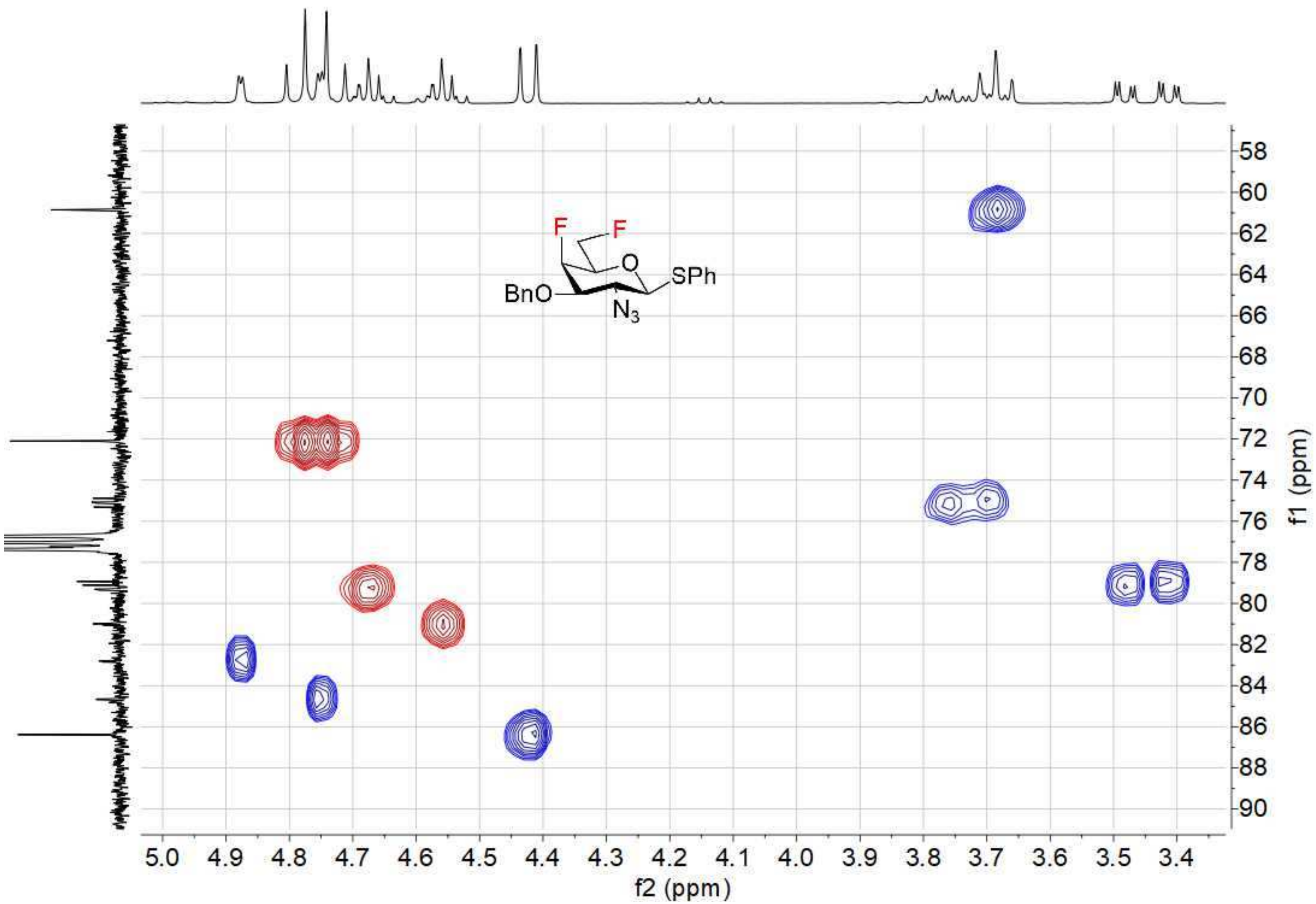
^{19}F NMR (376 MHz, $CDCl_3$) β-25



^{1}H-^{1}H COSY β-25

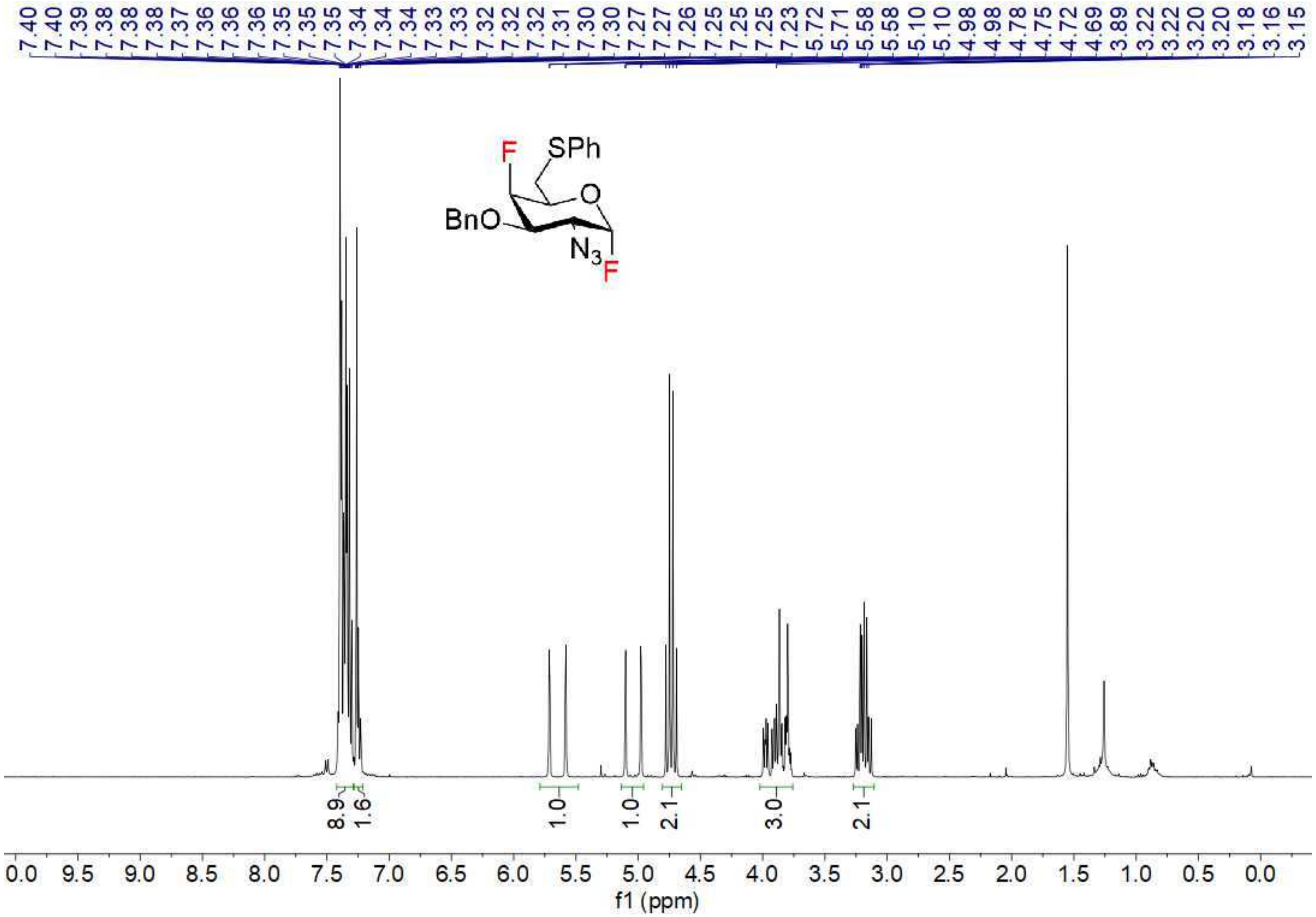


^{1}H-^{13}C HSQC β-25

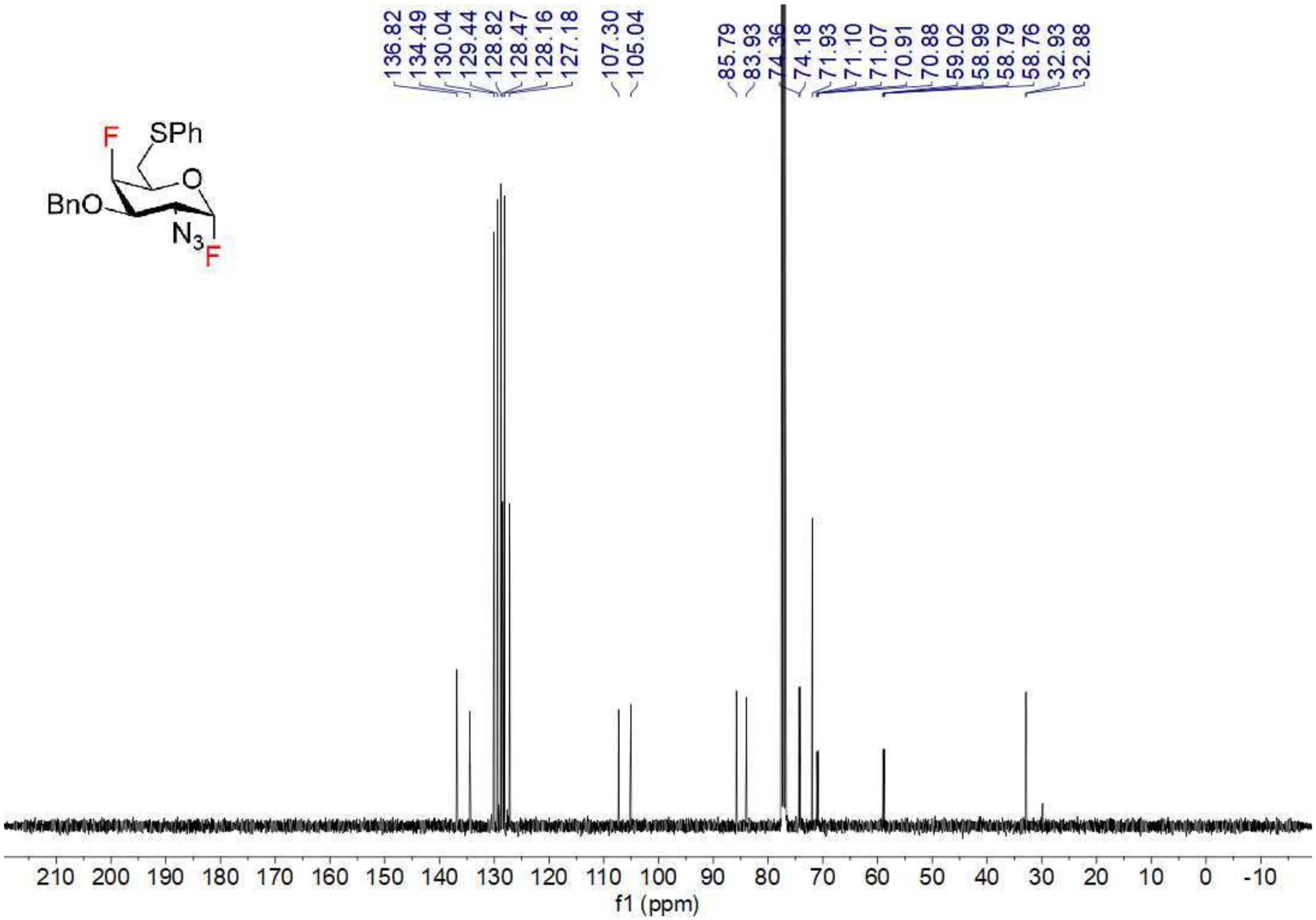


NMR COMPOUND 32

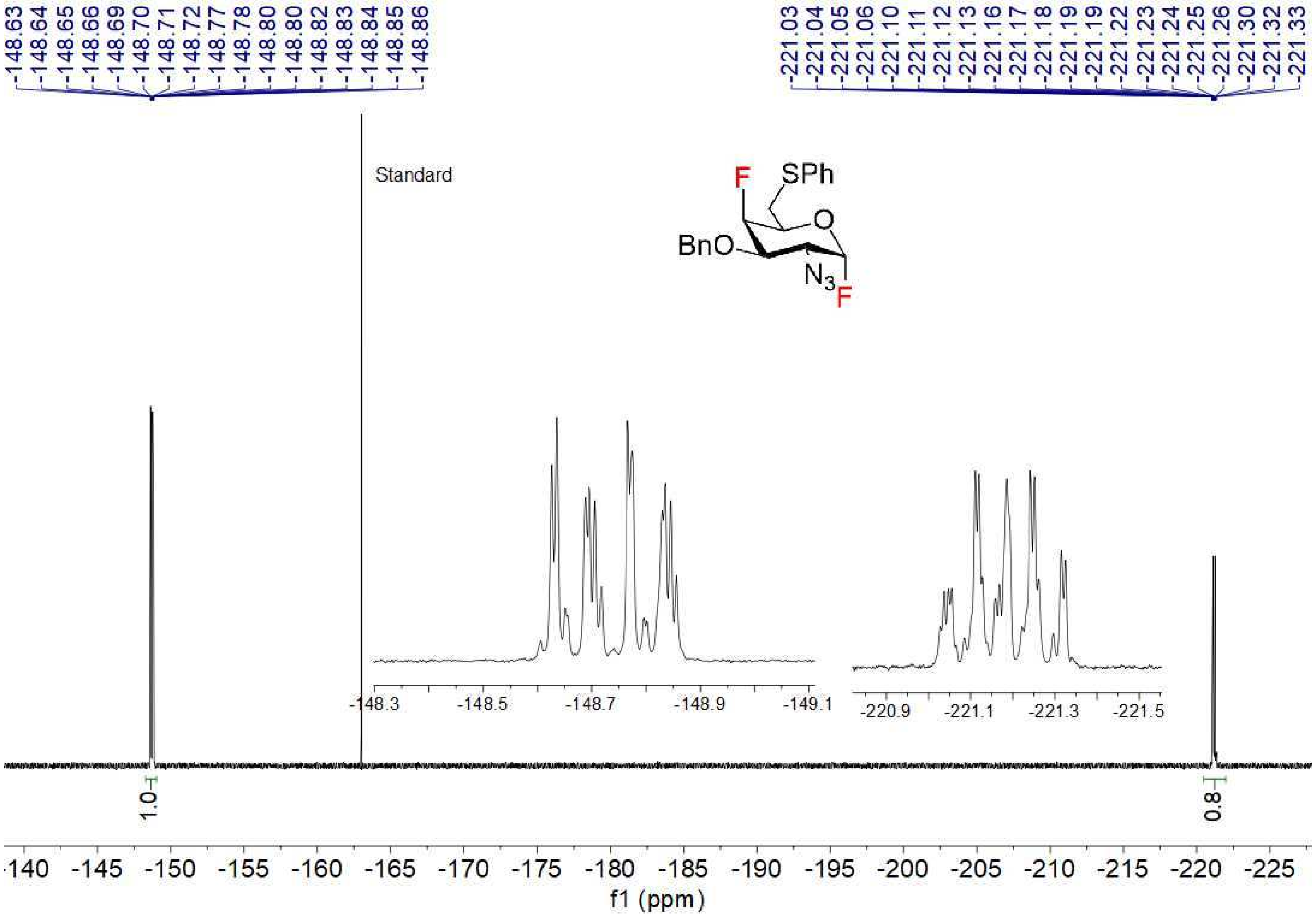
^{1}H NMR (400 MHz, CDCl$_3$) 32



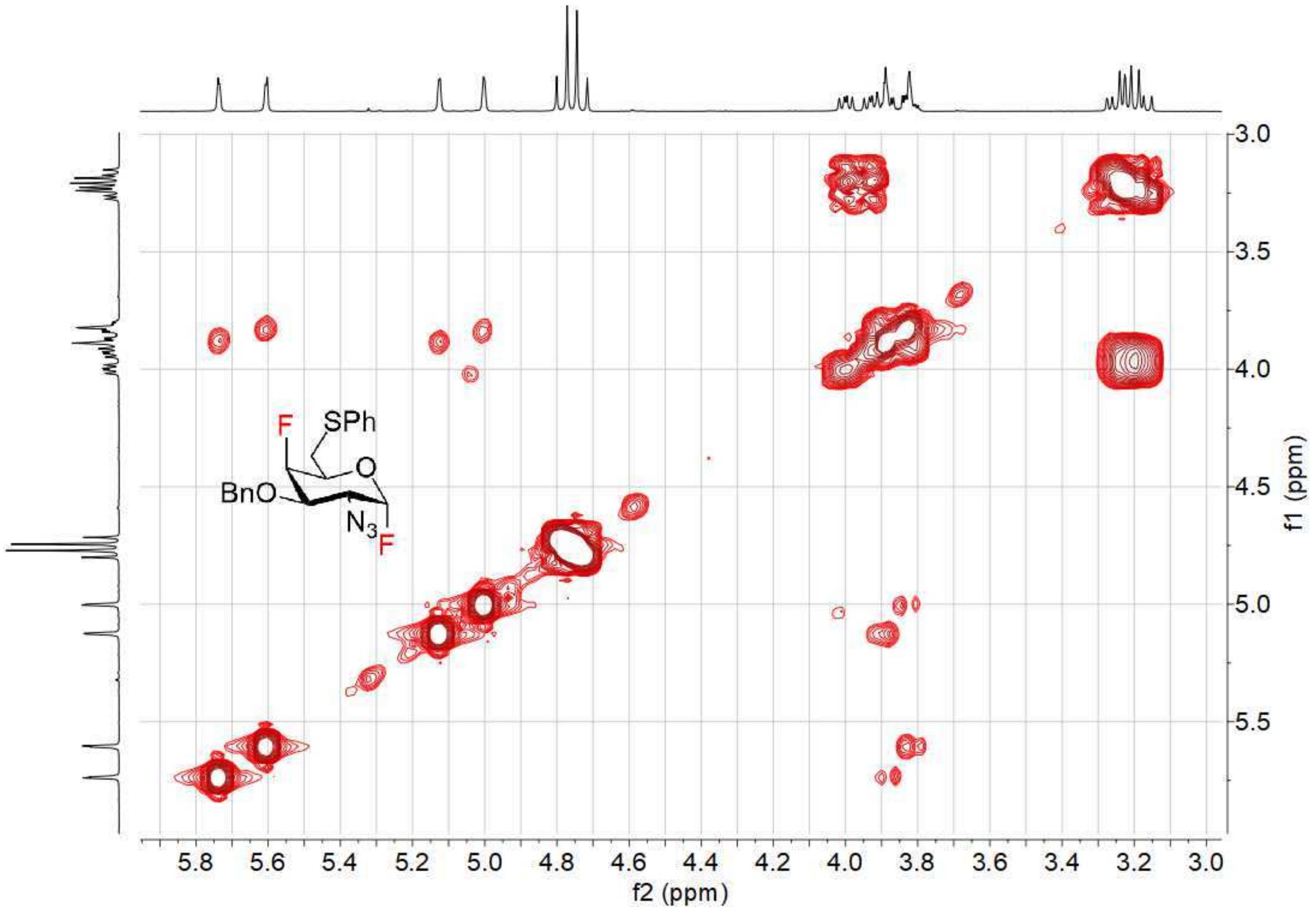
[13]C NMR (100 MHz, CDCl$_3$) 32



[19]F NMR (376 MHz, $CDCl_3$) 32

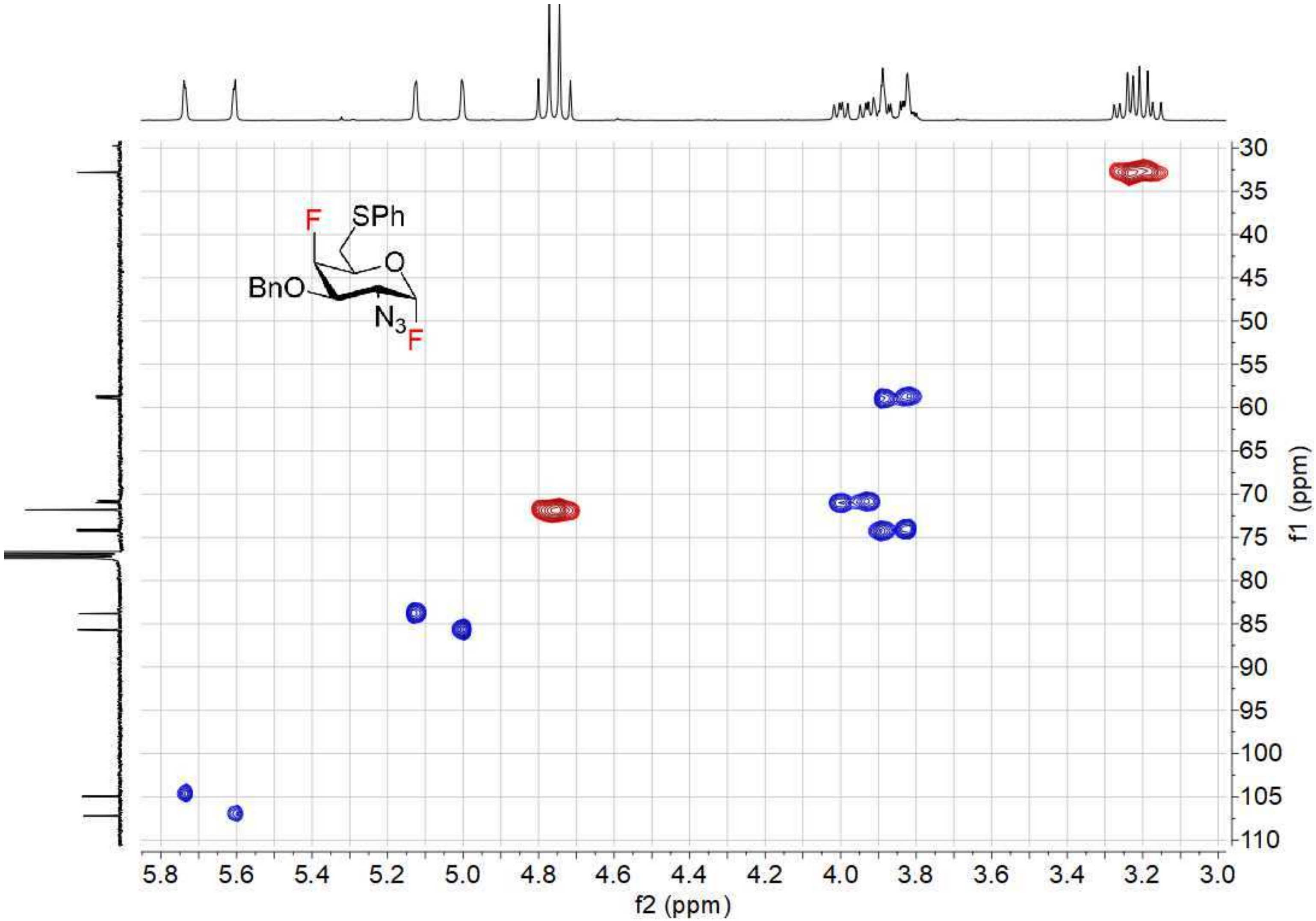


^{1}H-^{1}H COSY 32



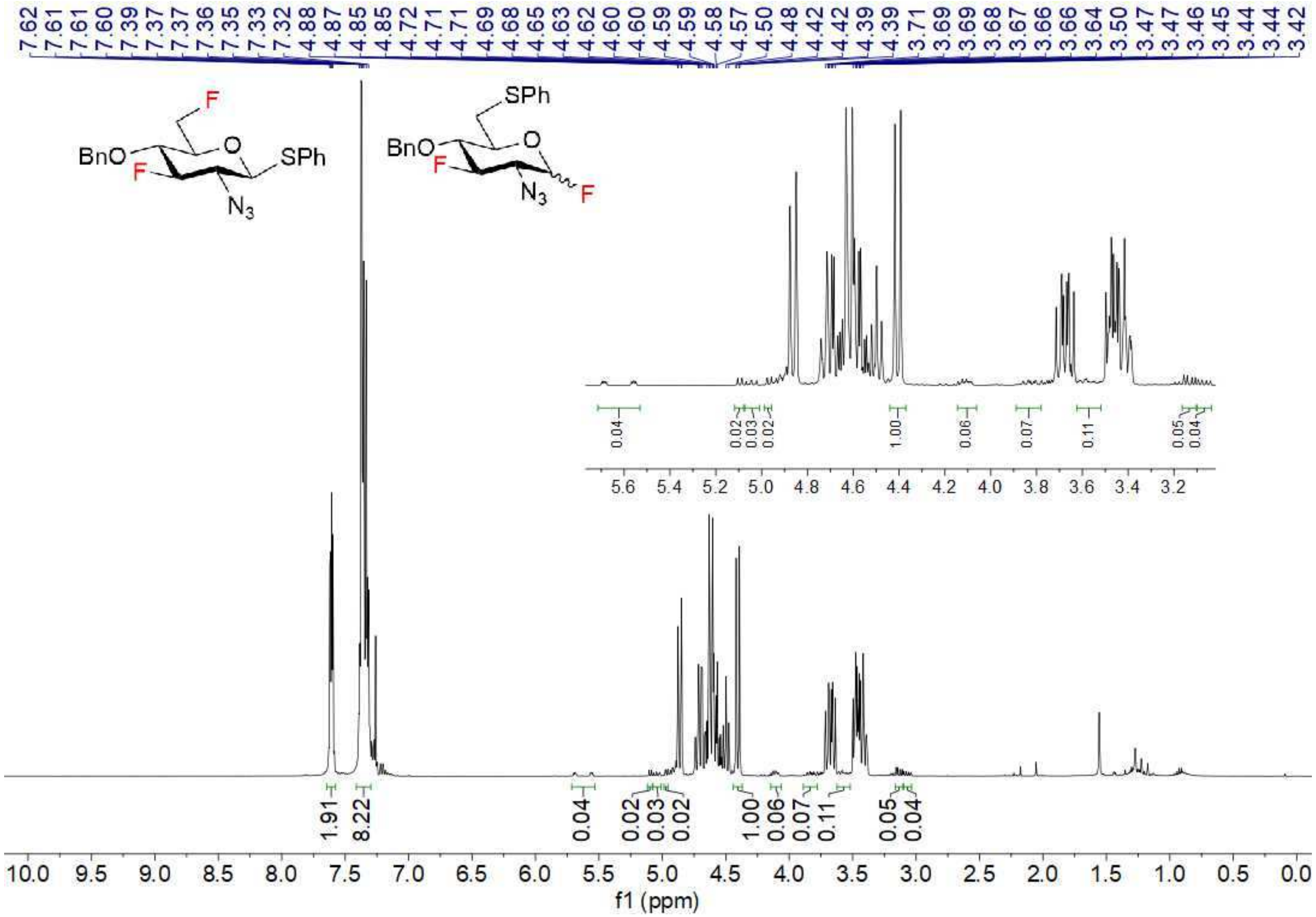
SPh
F
O
BnO
N3
F
3.0
3.5
4.0
4.5
5.0
5.5
f1 (ppm)
5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 4.0 3.8 3.6 3.4 3.2 3.0
f2 (ppm)

^{1}H-^{13}C HSQC 32

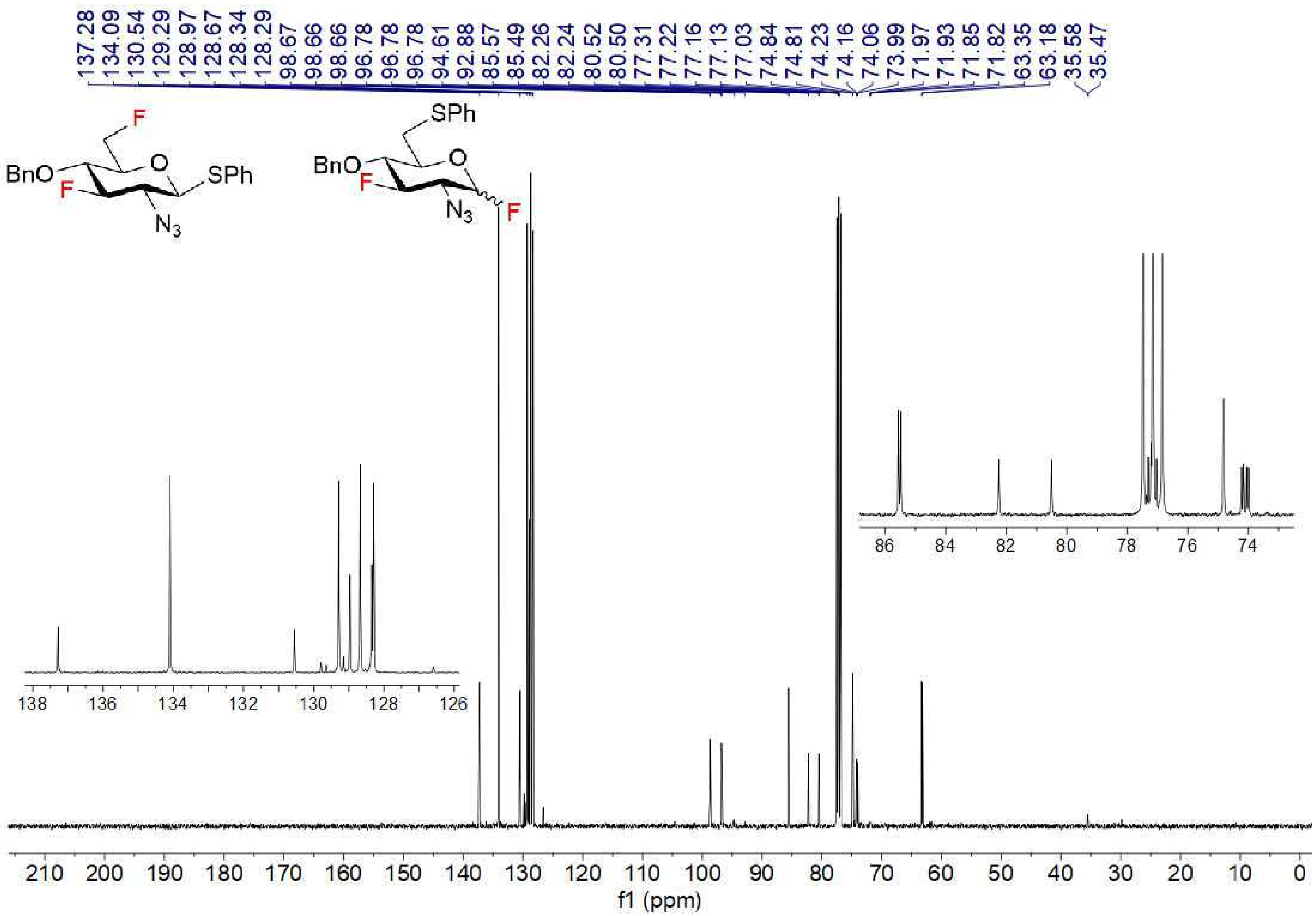


NMR COMPOUNDS β-22 and 33

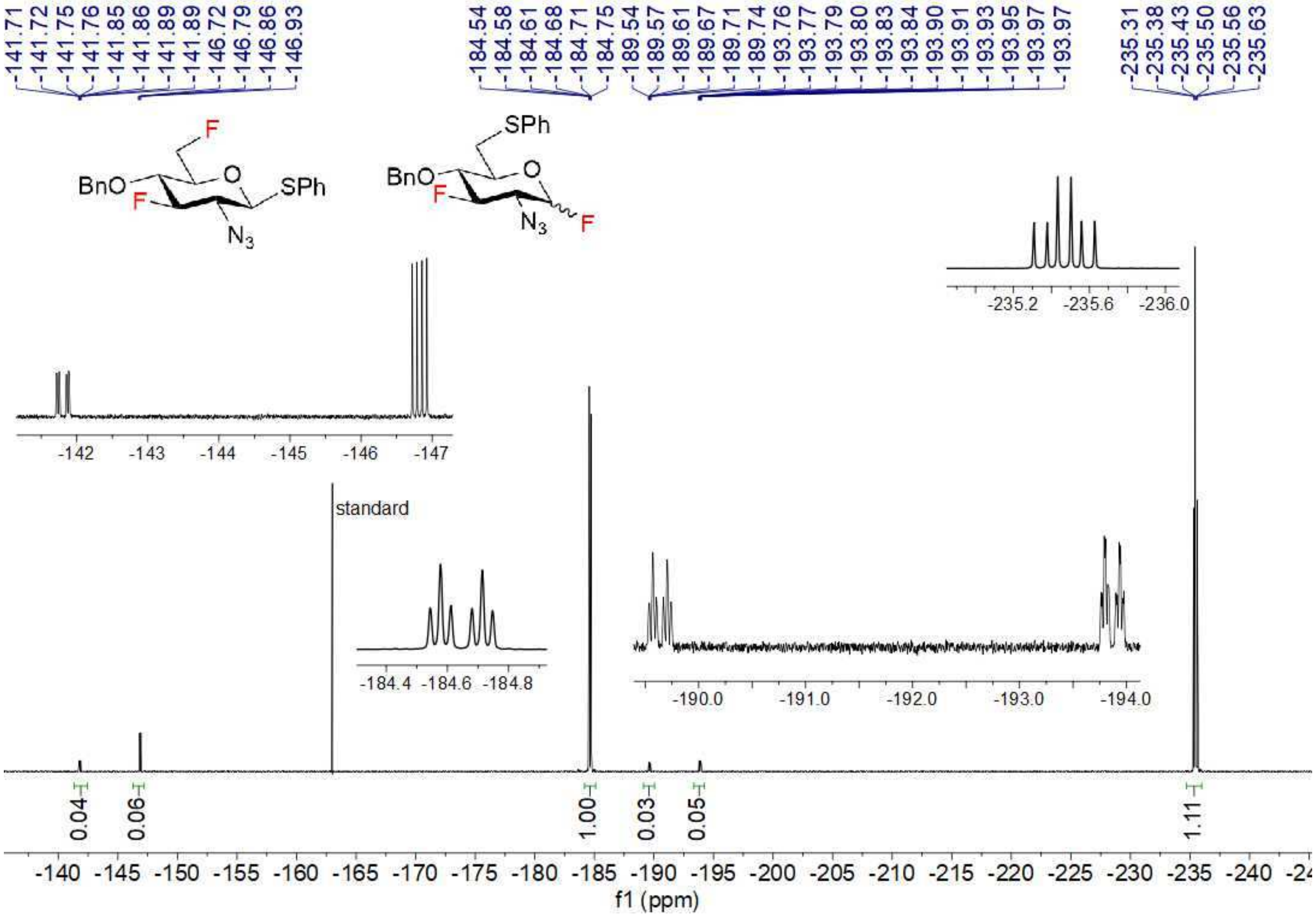
[1]H NMR (400 MHz, CDCl$_3$) β-22 (ca. 9% of 33)



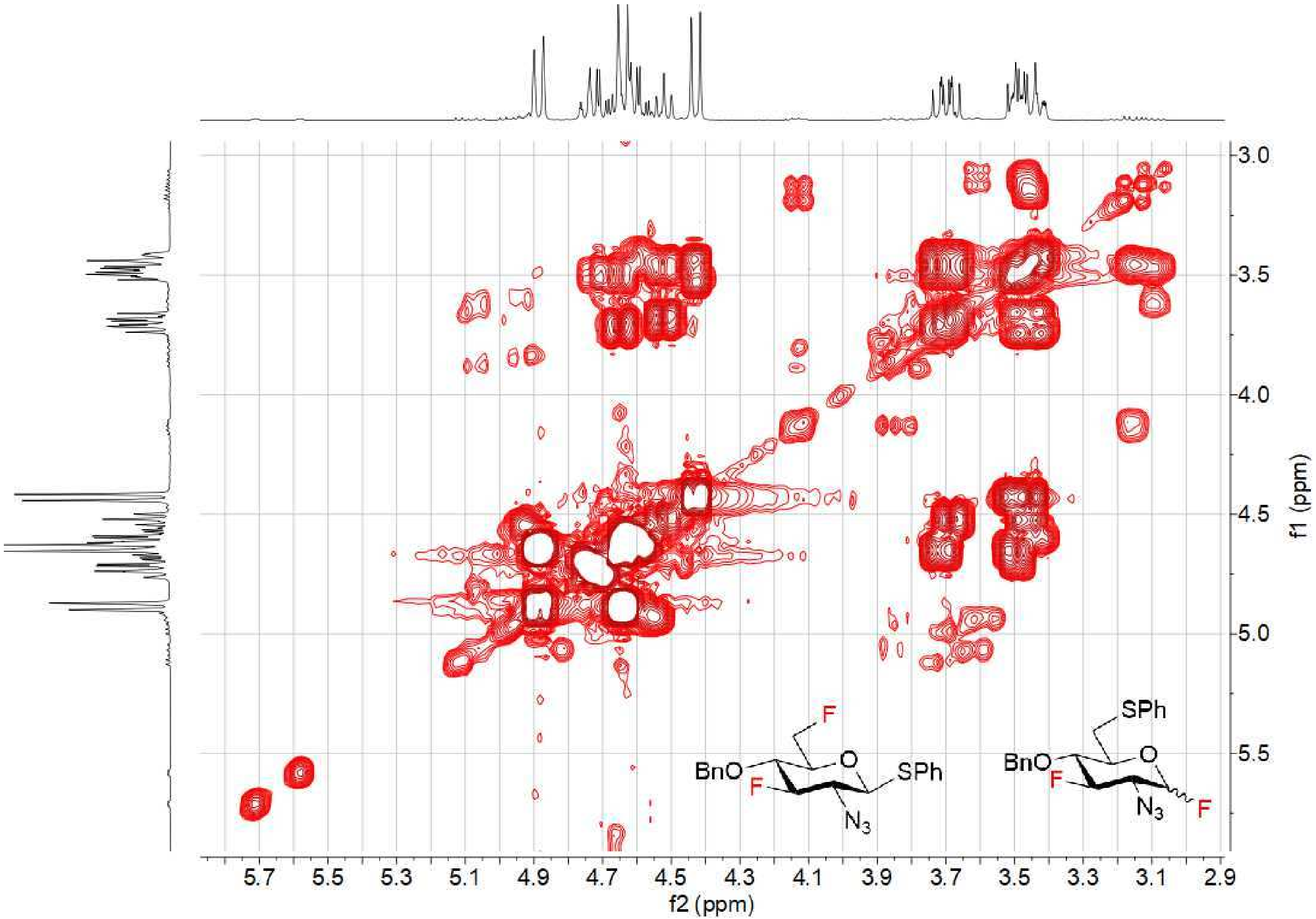
^{13}C NMR (100 MHz, $CDCl_3$) β-22 (ca. 9% of 33)



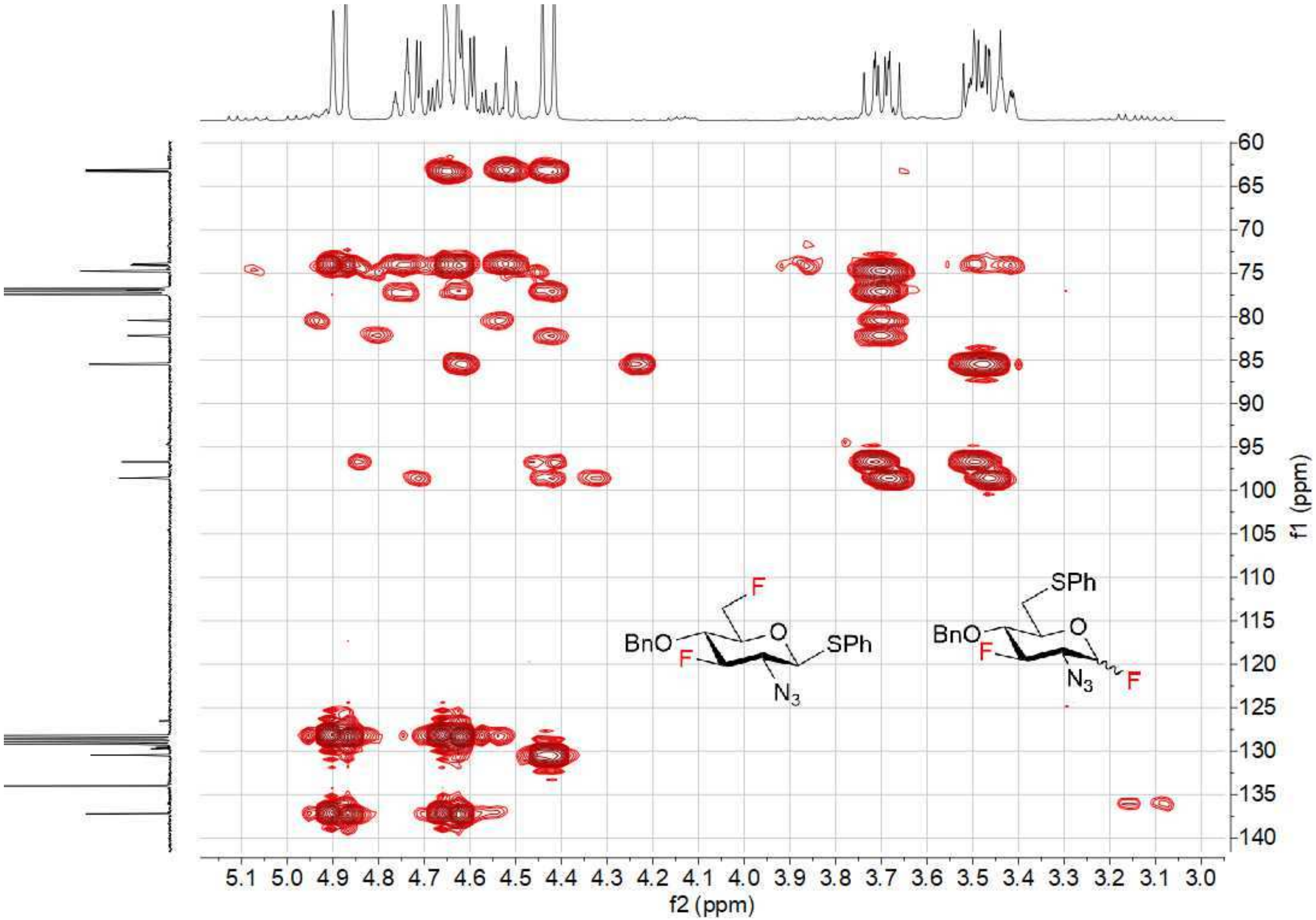
^{19}F NMR (376 MHz, CDCl$_3$) β-22 (ca. 5% of α-33 and 4% of β-33)



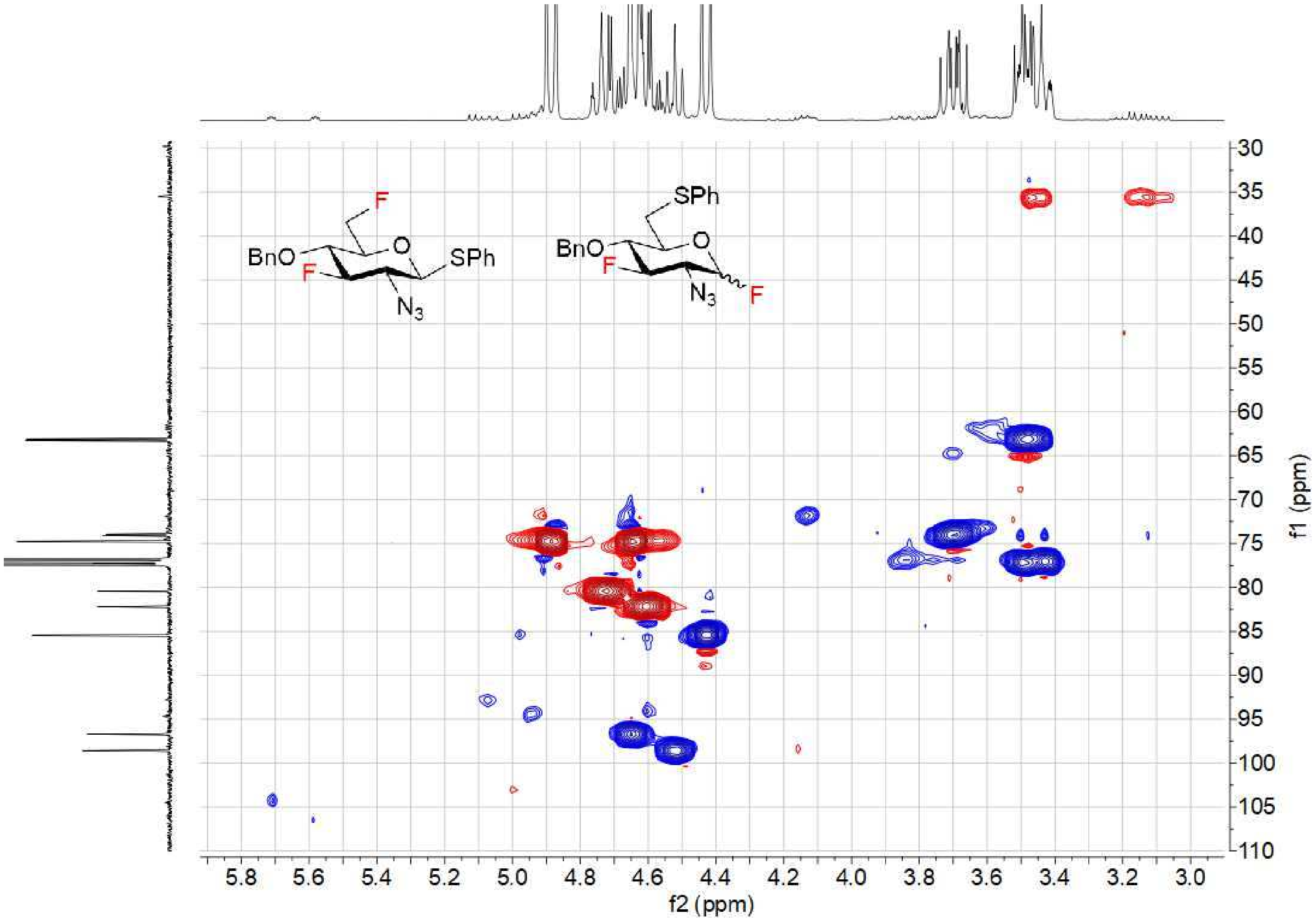
^{1}H-^{1}H COSY β-22 (ca. 9% of 33)



^{1}H-^{13}C HMBC β-22 (ca. 9% of 33)

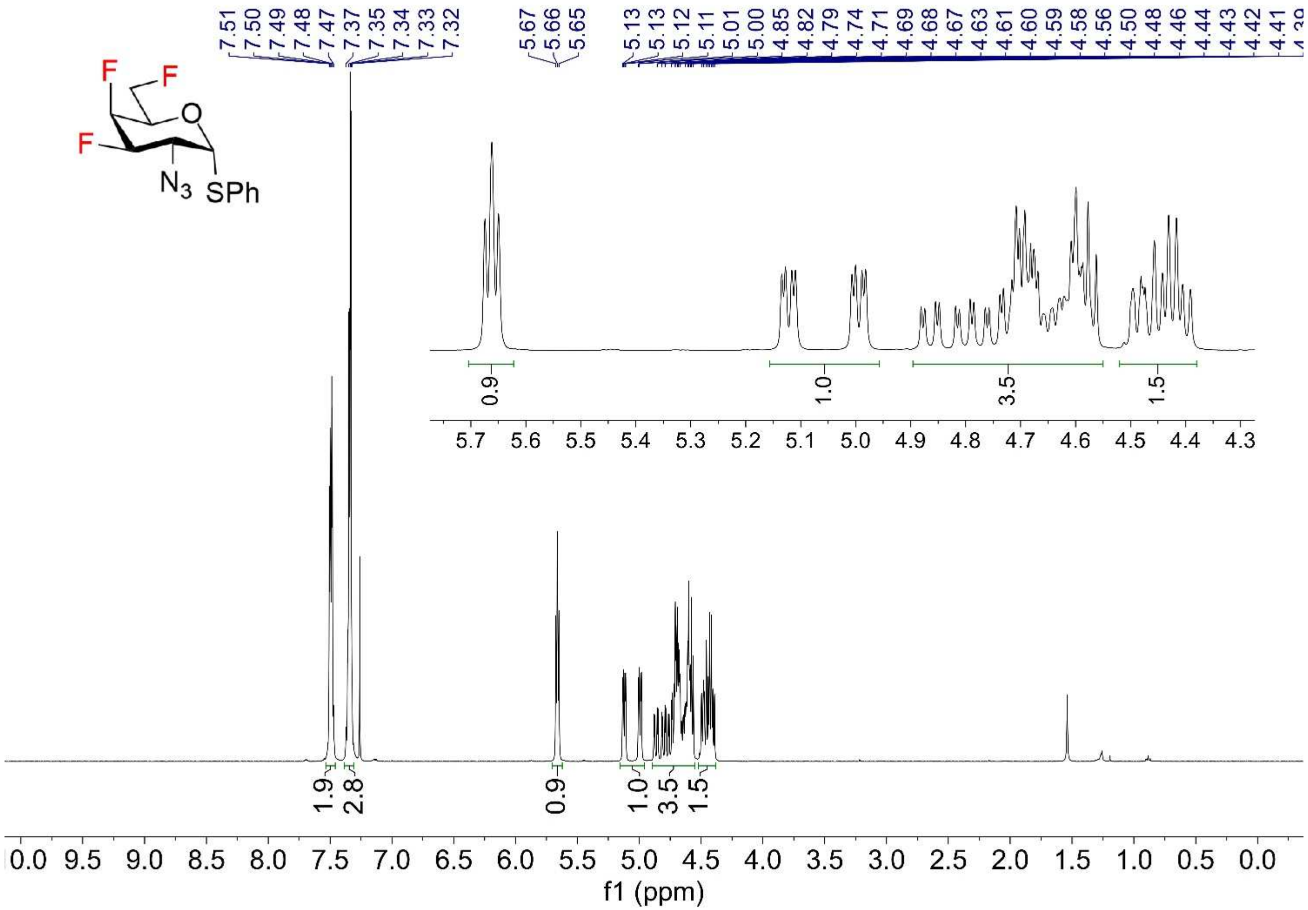


^{1}H-^{13}C HSQC β-22 (ca. 9% of 33)

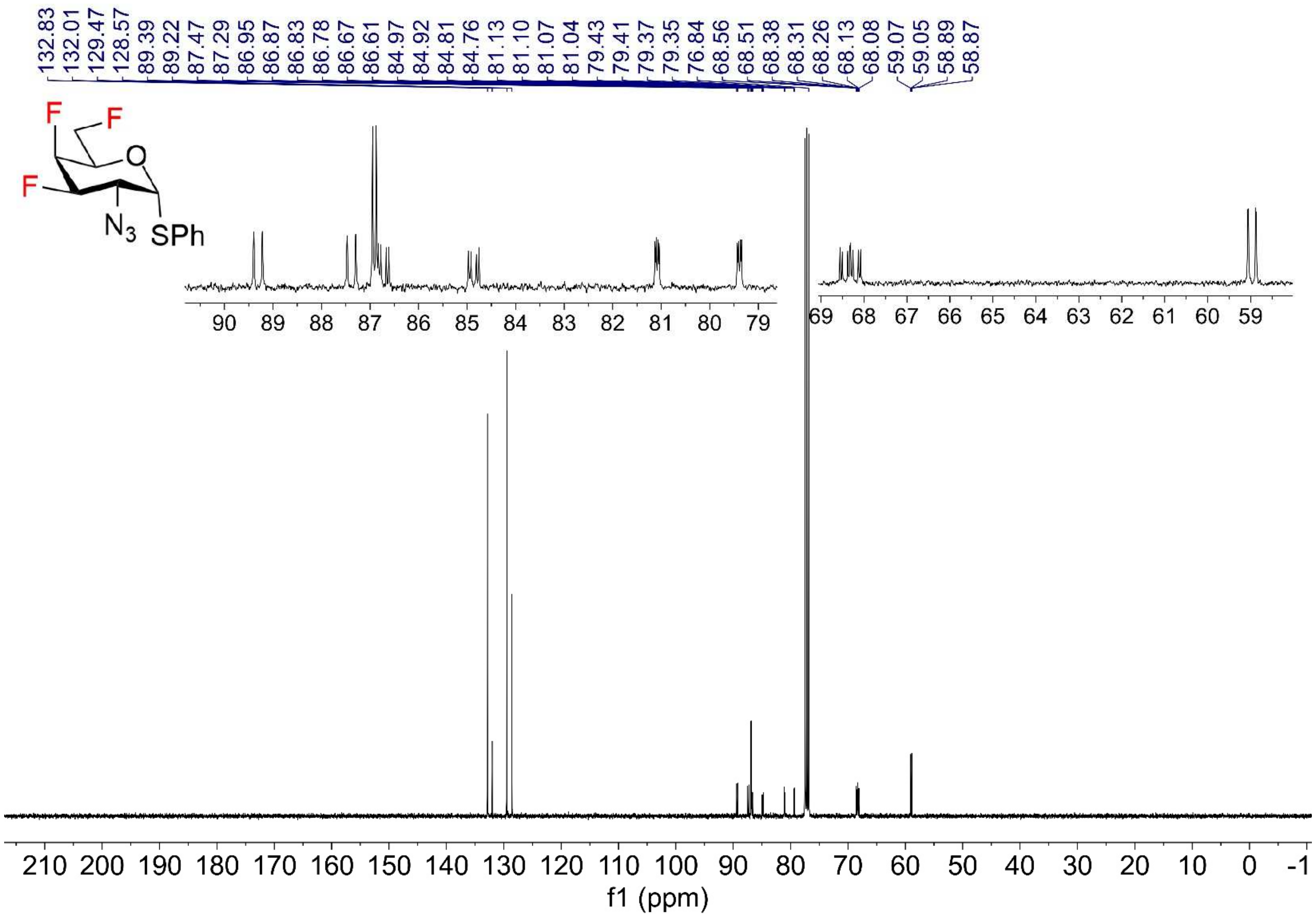


NMR COMPOUND α-34

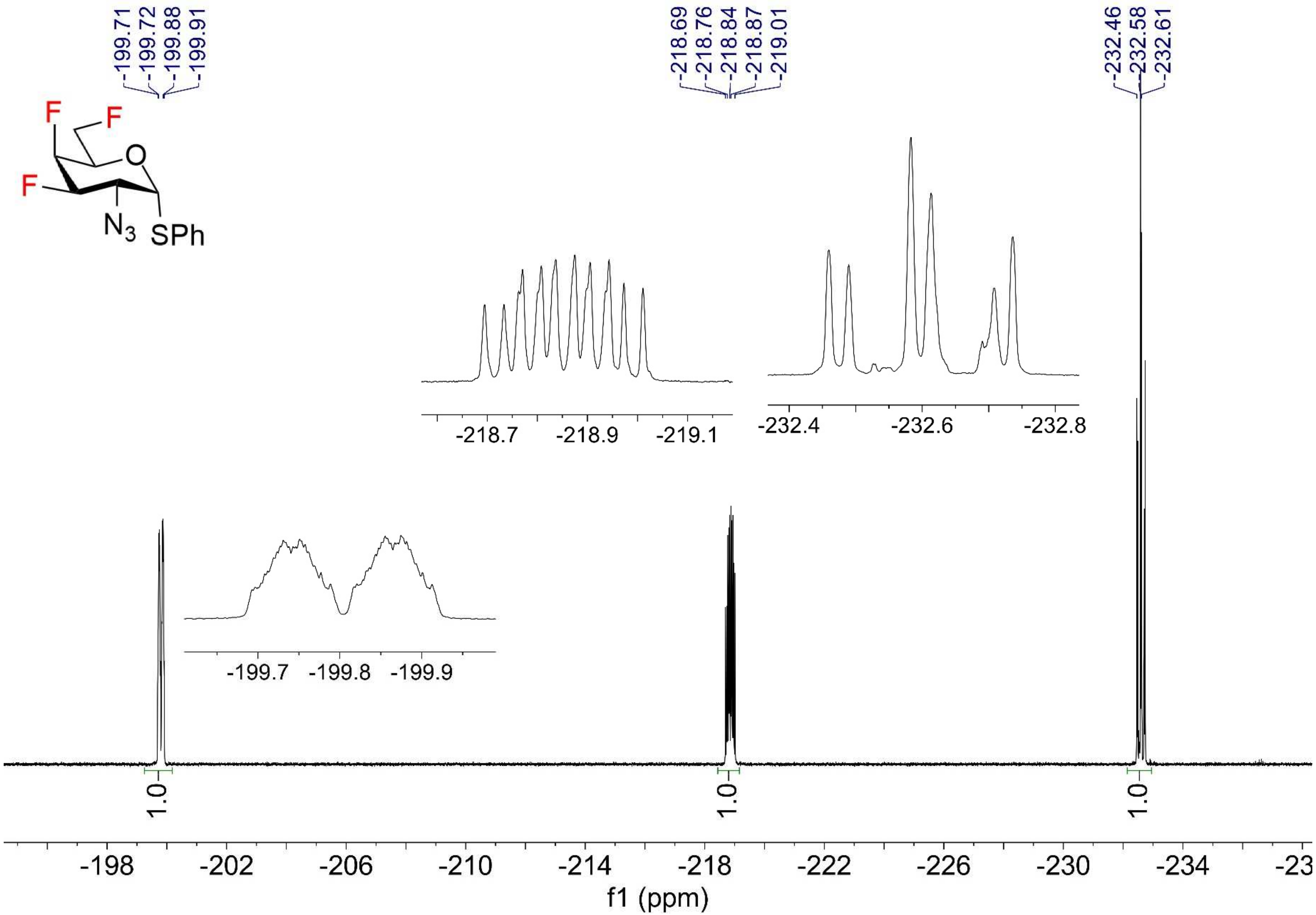
^{1}H NMR (400 MHz, $CDCl_3$) α-34



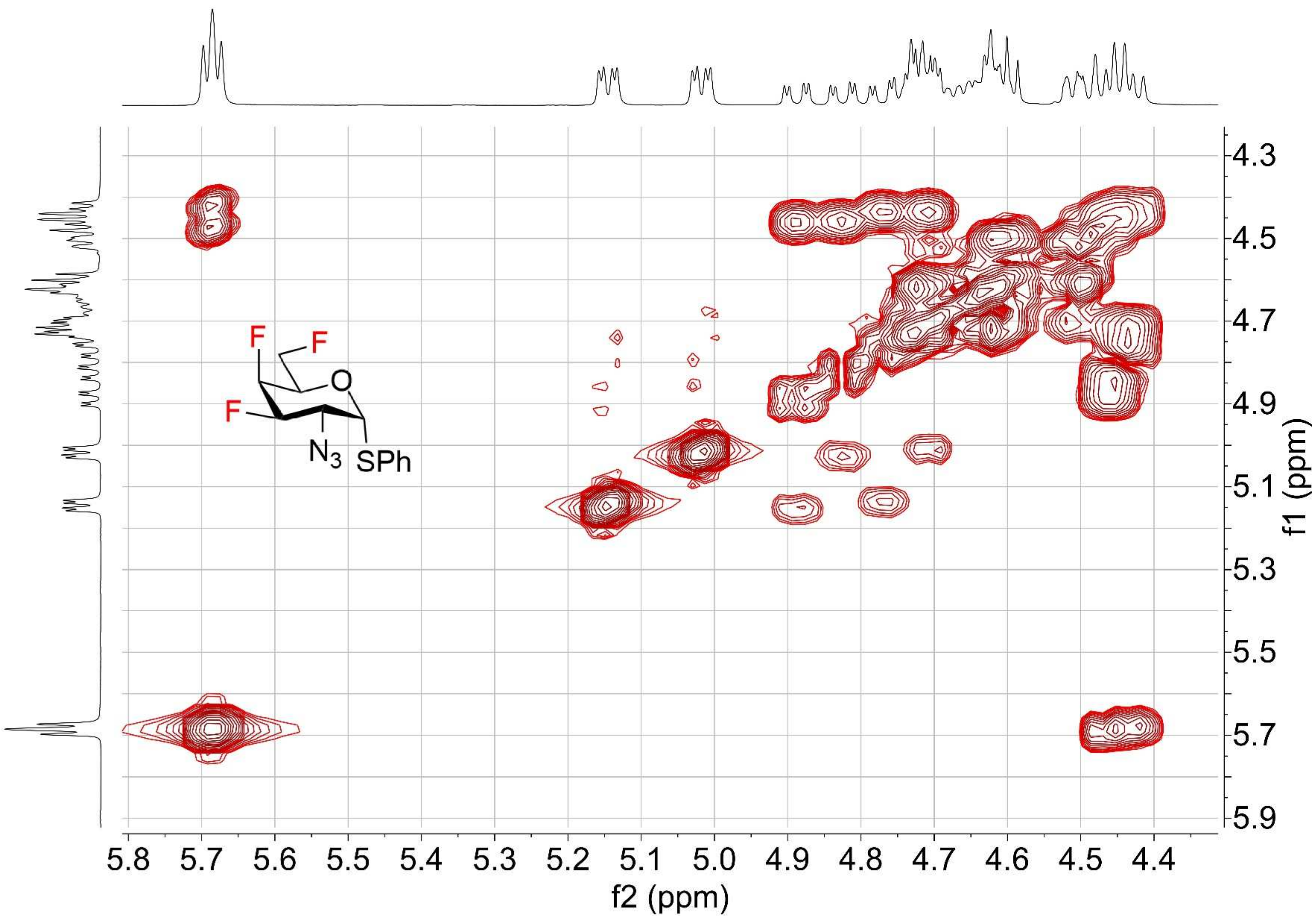
^{13}C NMR (100 MHz, CDCl$_3$) α-34



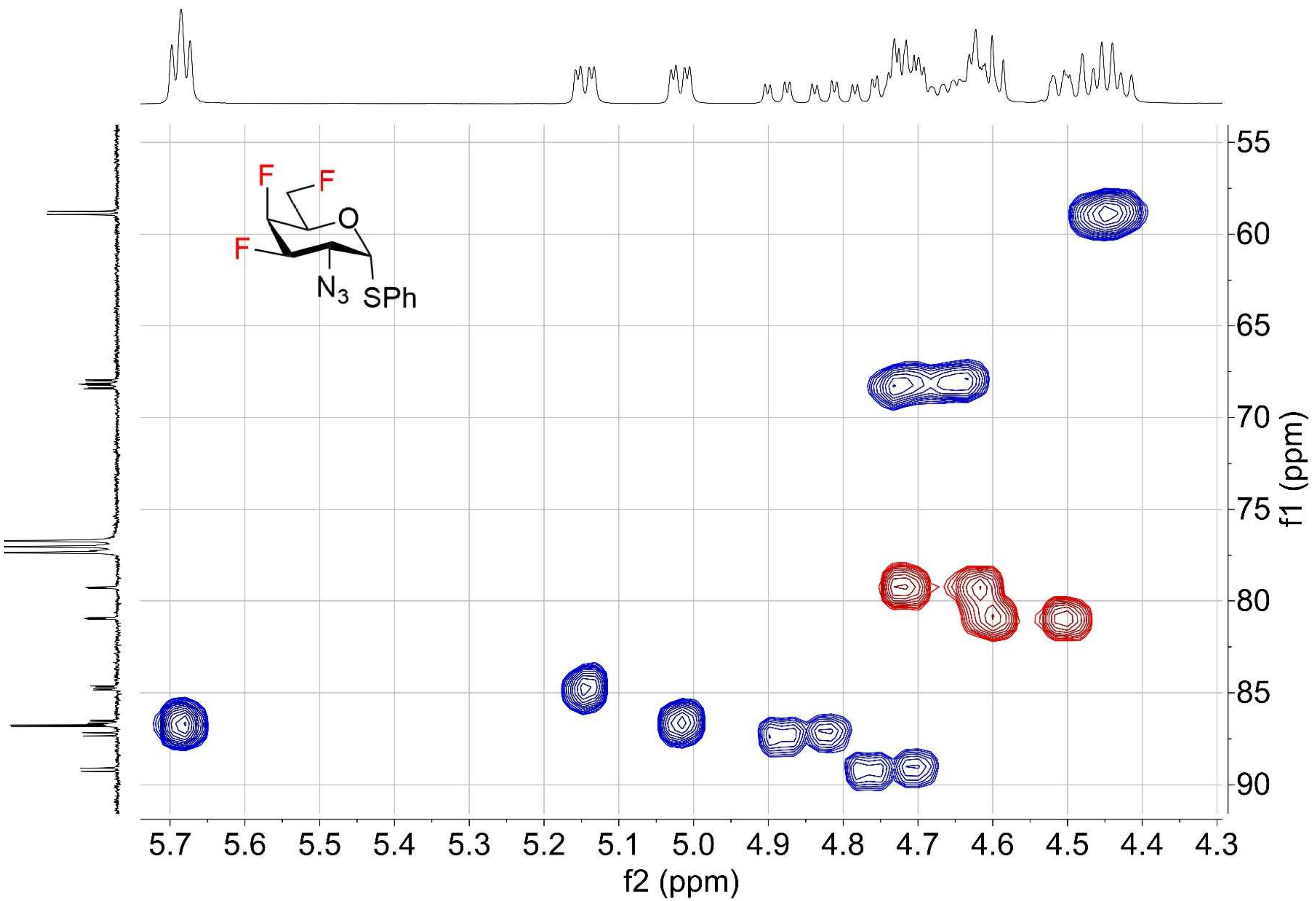
^{19}F NMR (376 MHz, CDCl$_3$) α-34



^{1}H-^{1}H COSY α-34

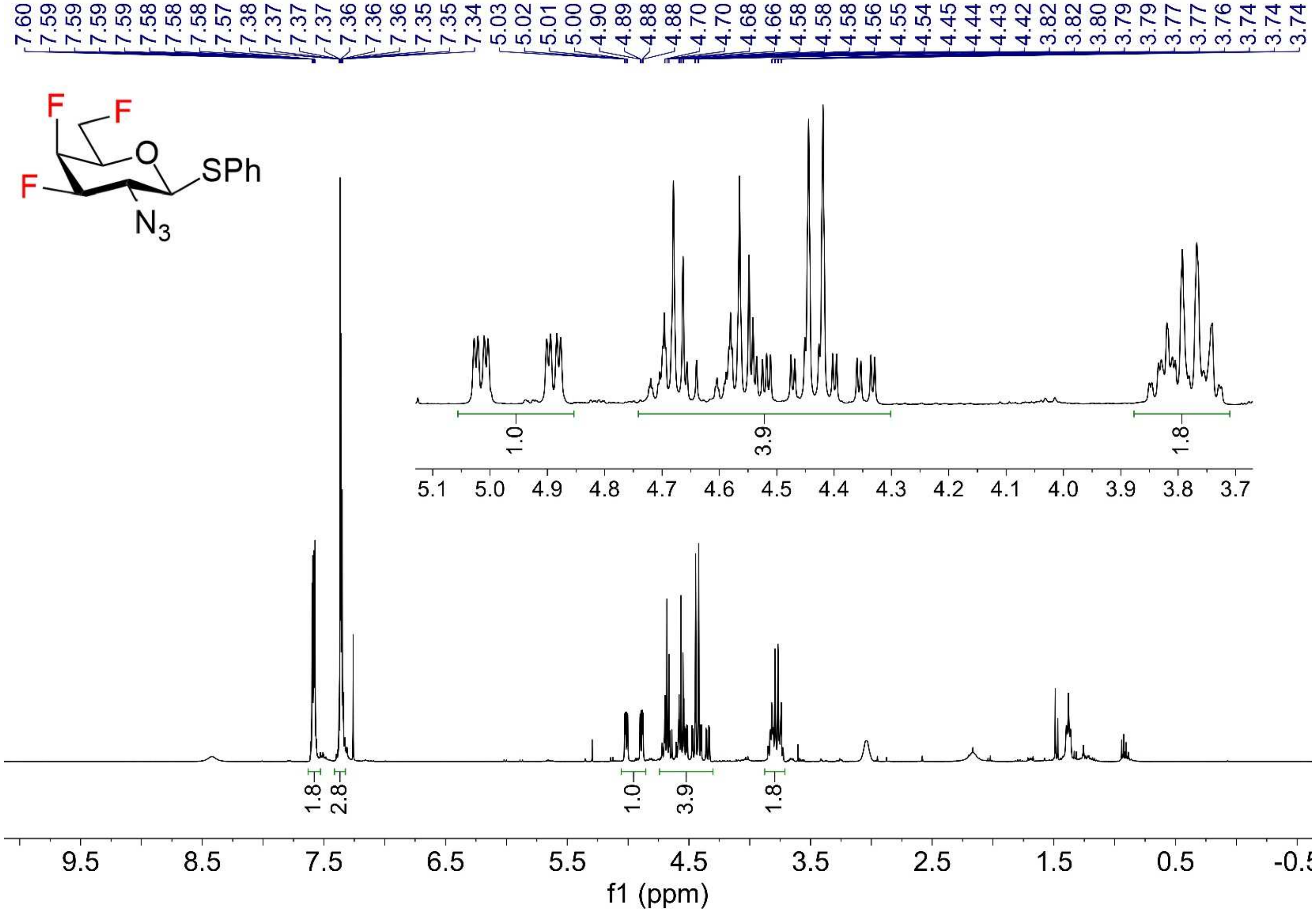


^{1}H-^{13}C HSQC α-34

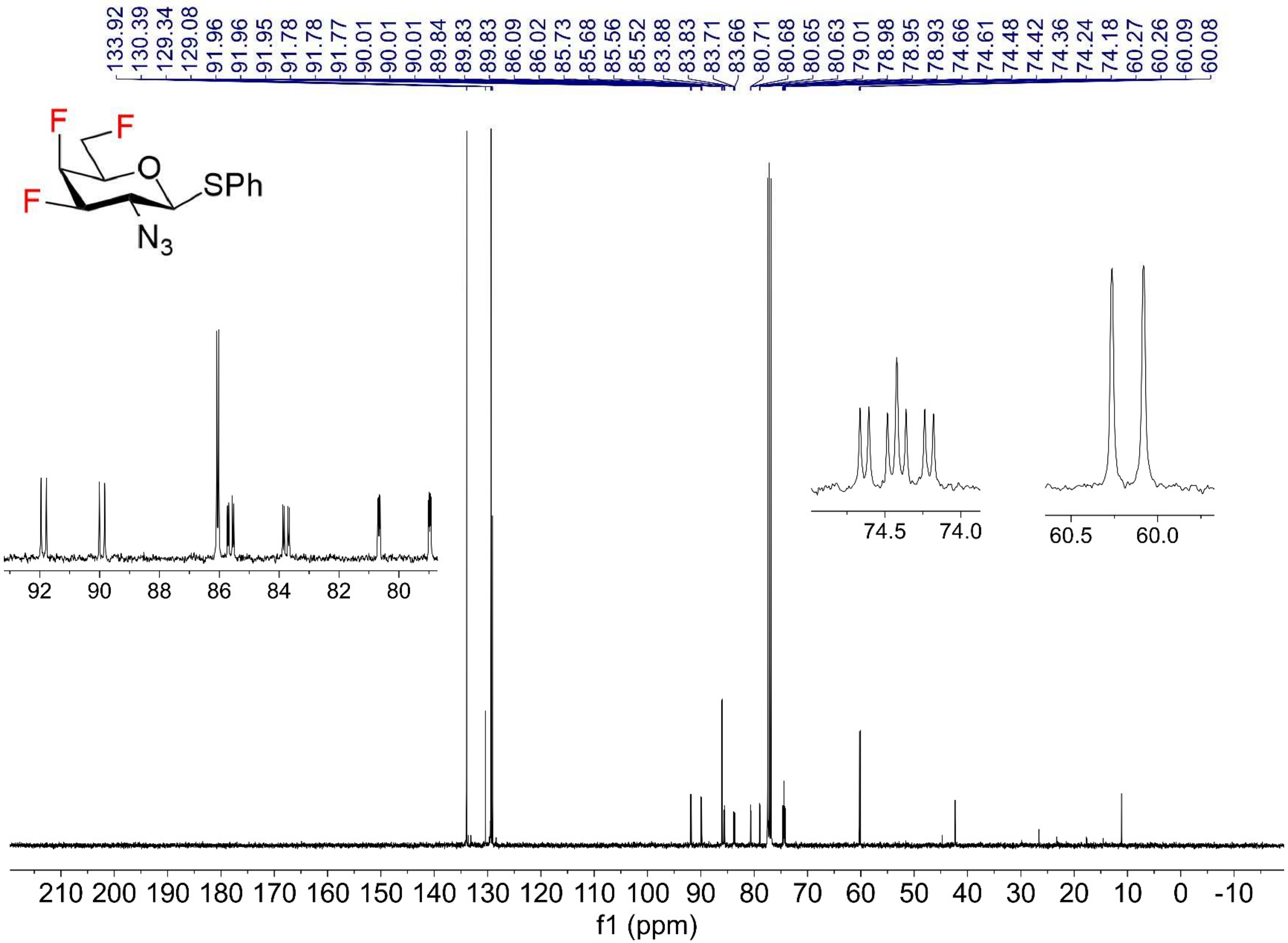


NMR COMPOUND β-34

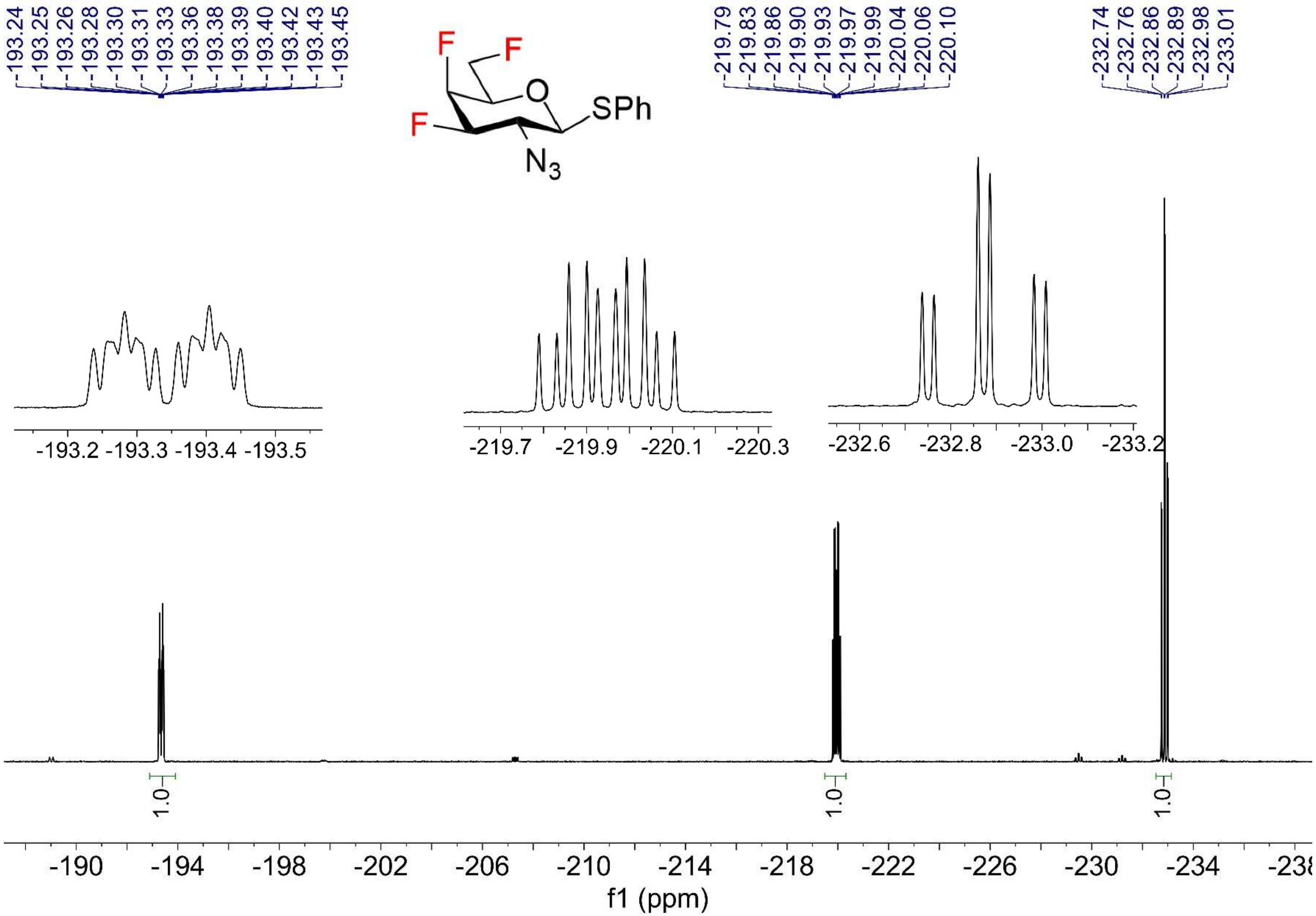
^{1}H NMR (400 MHz, $CDCl_3$) β-34



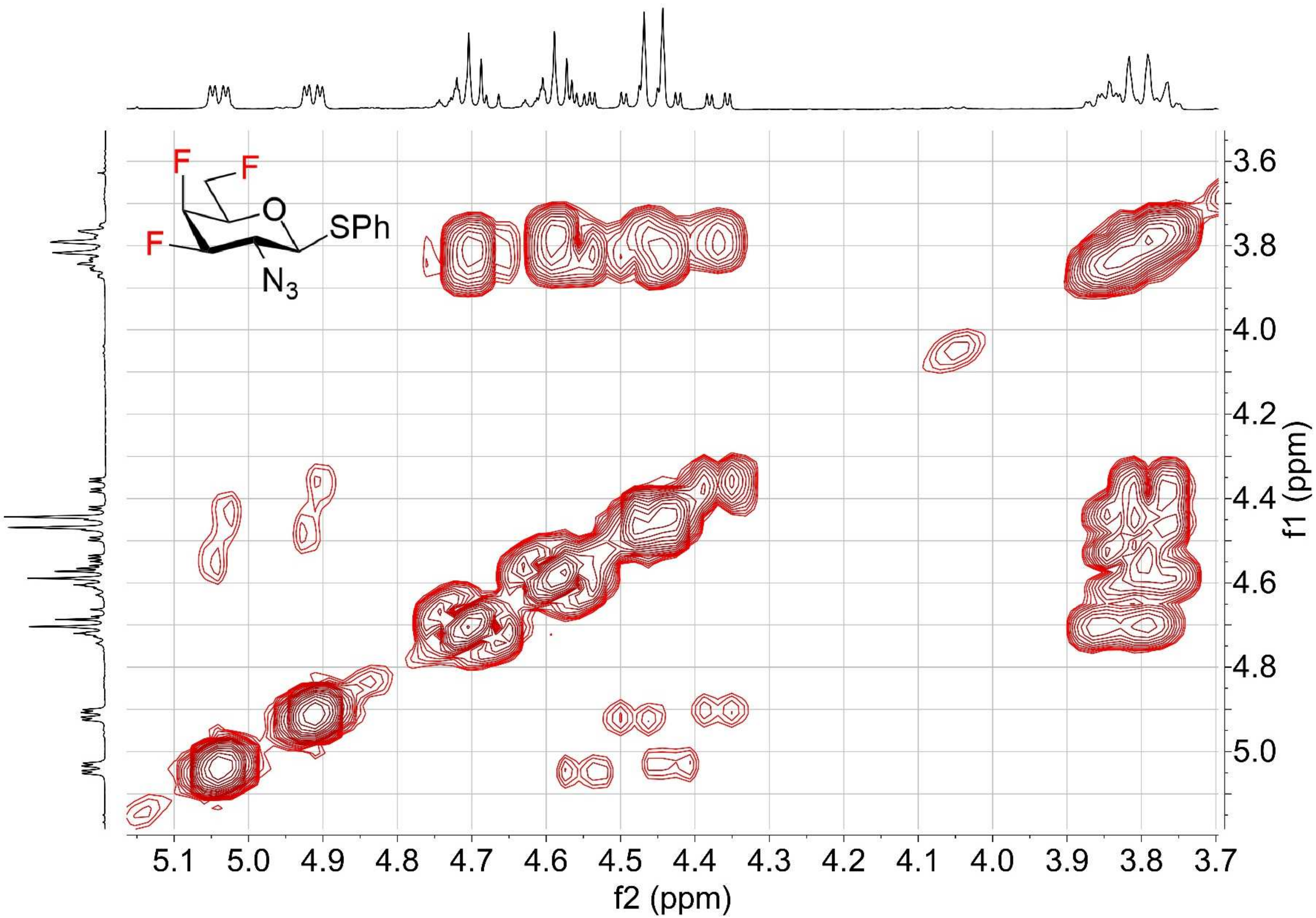
^{13}C NMR (100 MHz, CDCl$_3$) β-34



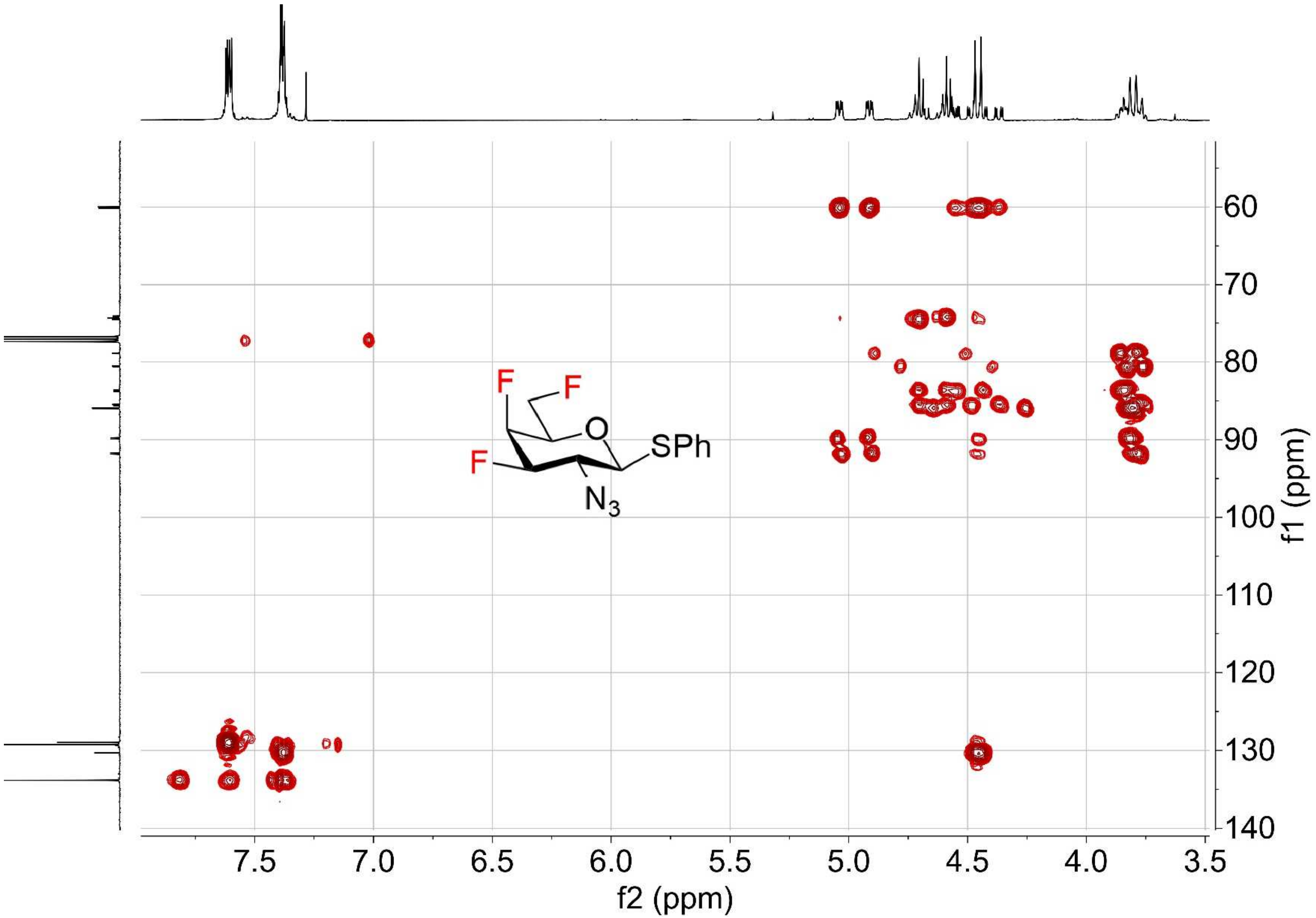
[19]F NMR (376 MHz, CDCl$_3$) β-34



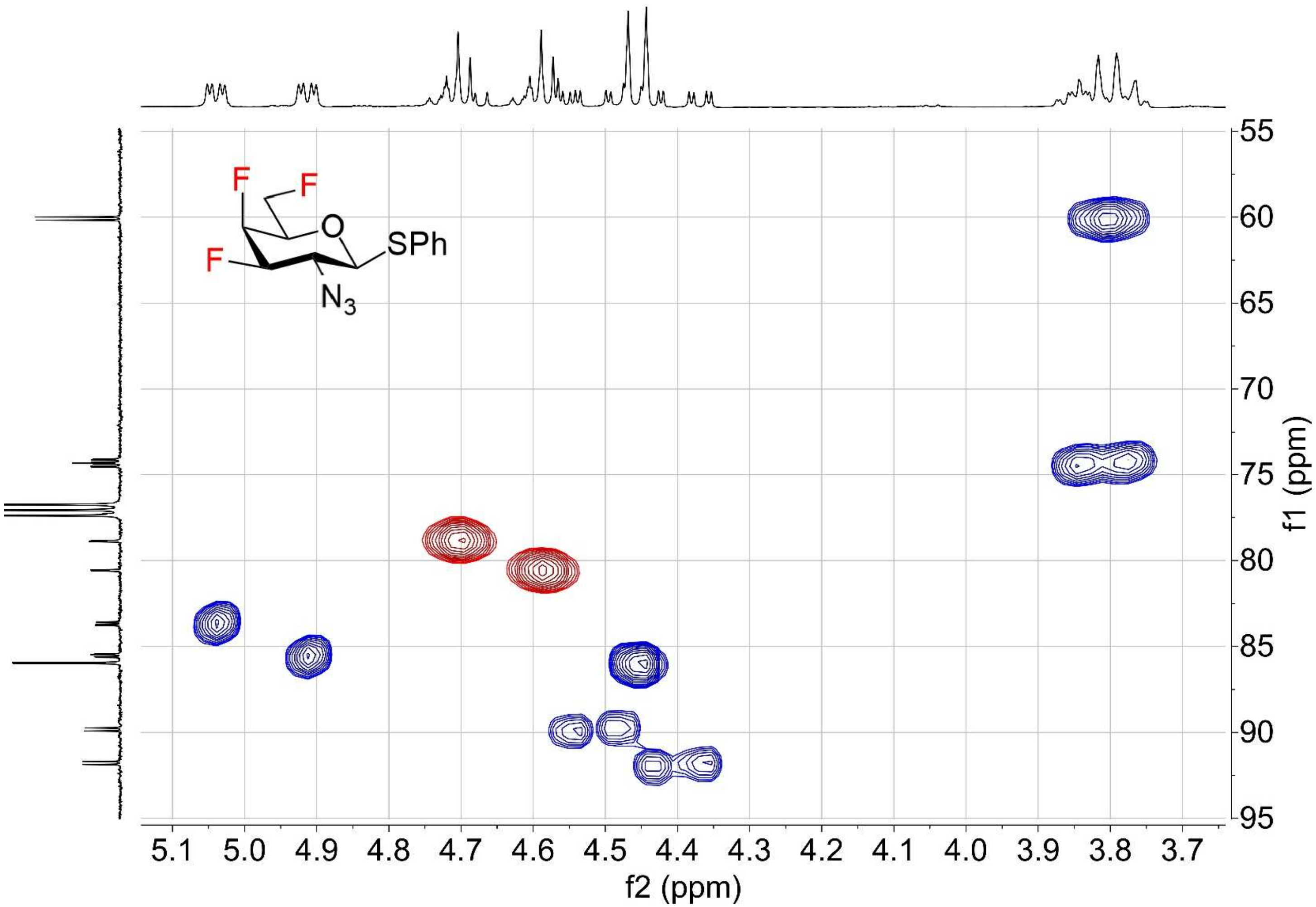
^{1}H-^{1}H COSY β-34



^{1}H-^{13}C HMBC β-34

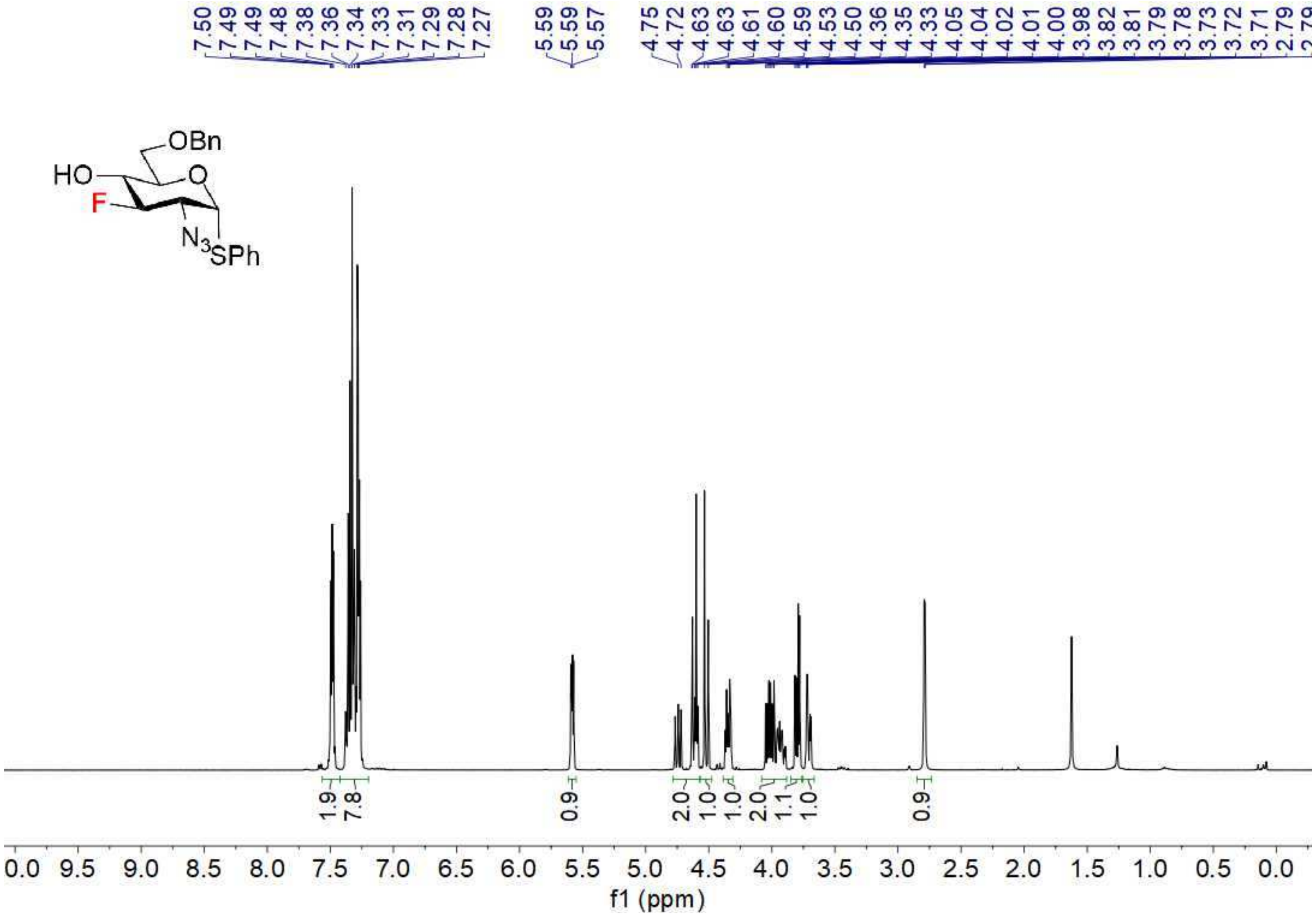


^1H-^{13}C HSQC β-34

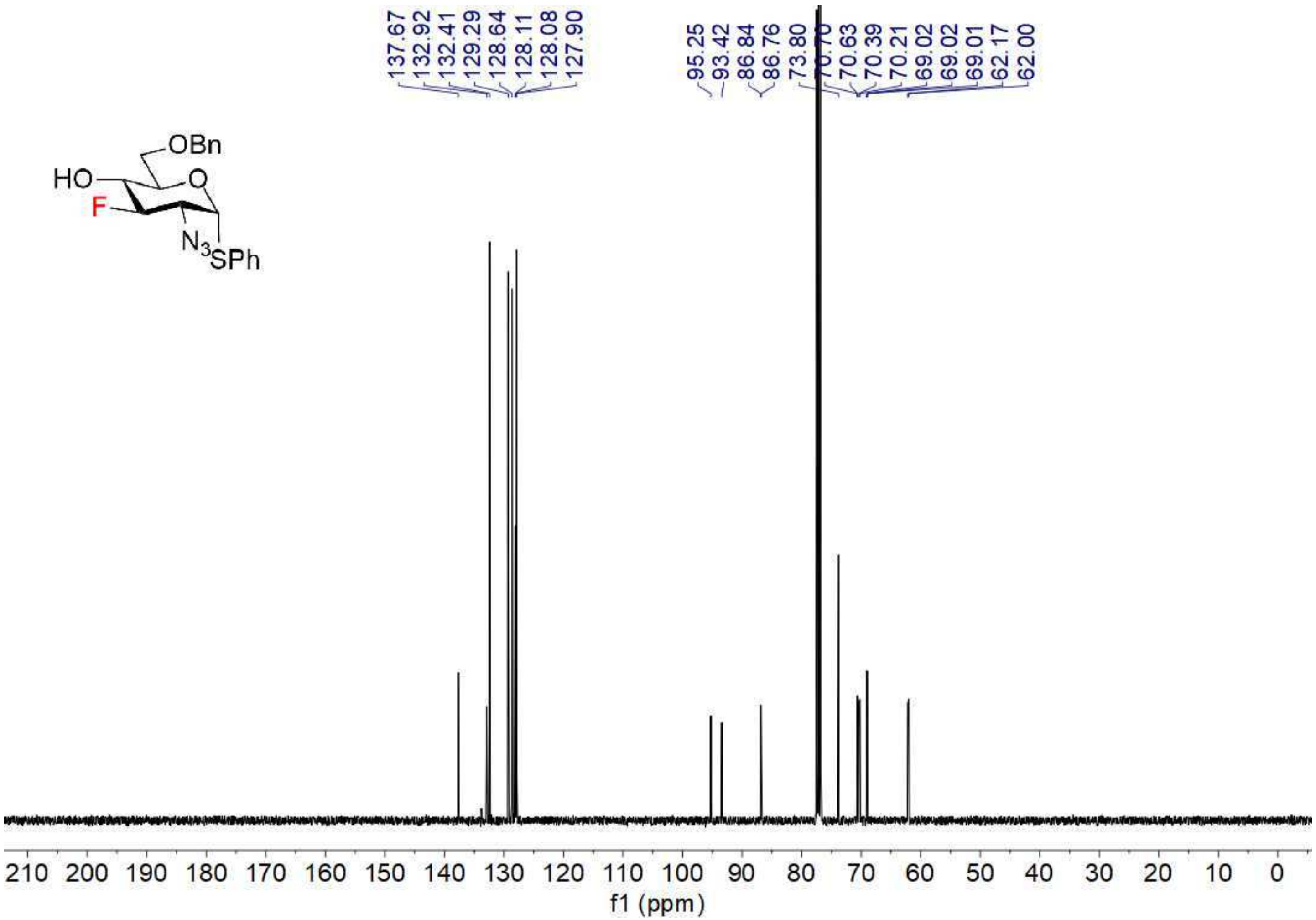


NMR COMPOUND α-35

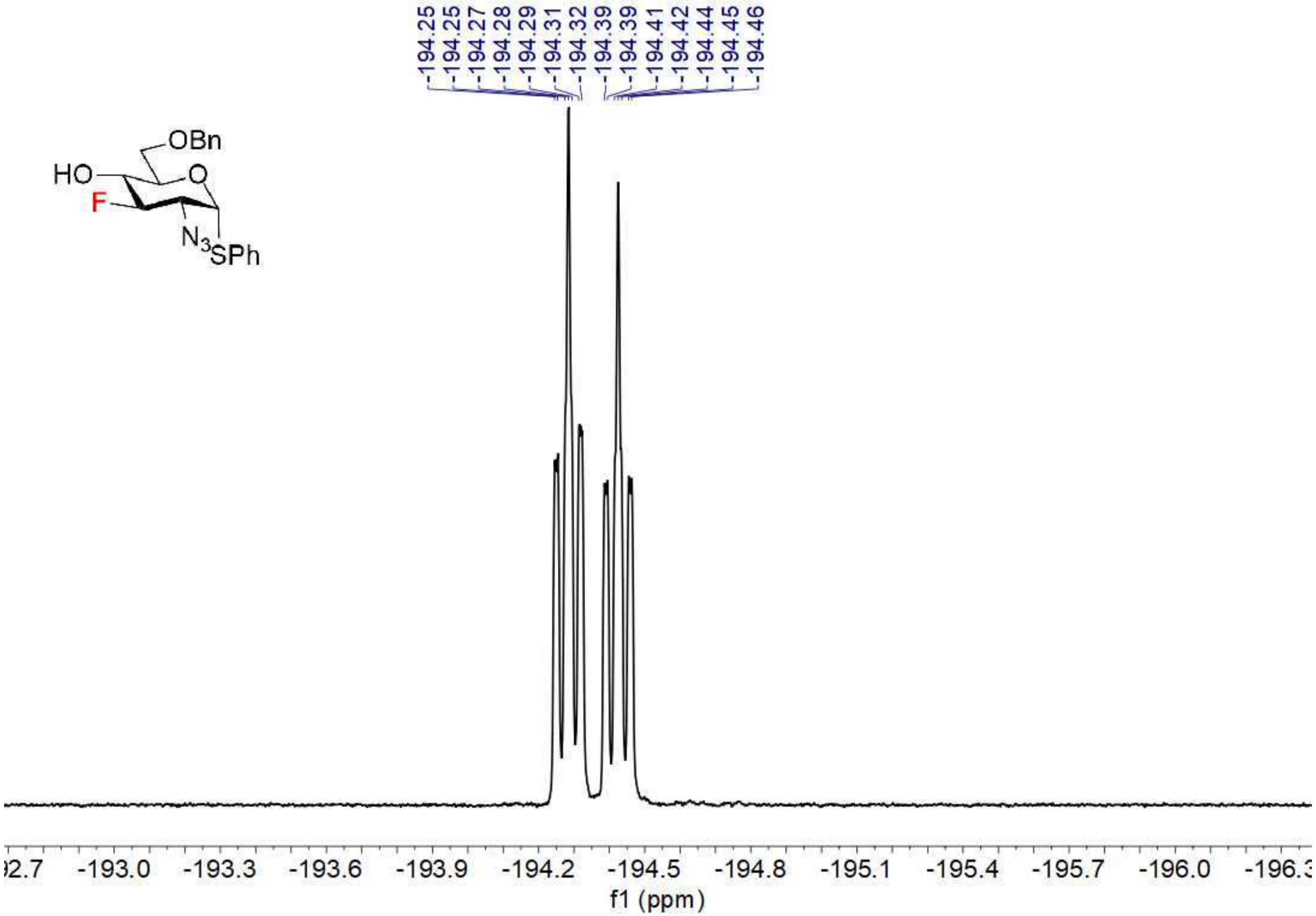
[1]H NMR (400 MHz, CDCl₃) α-35



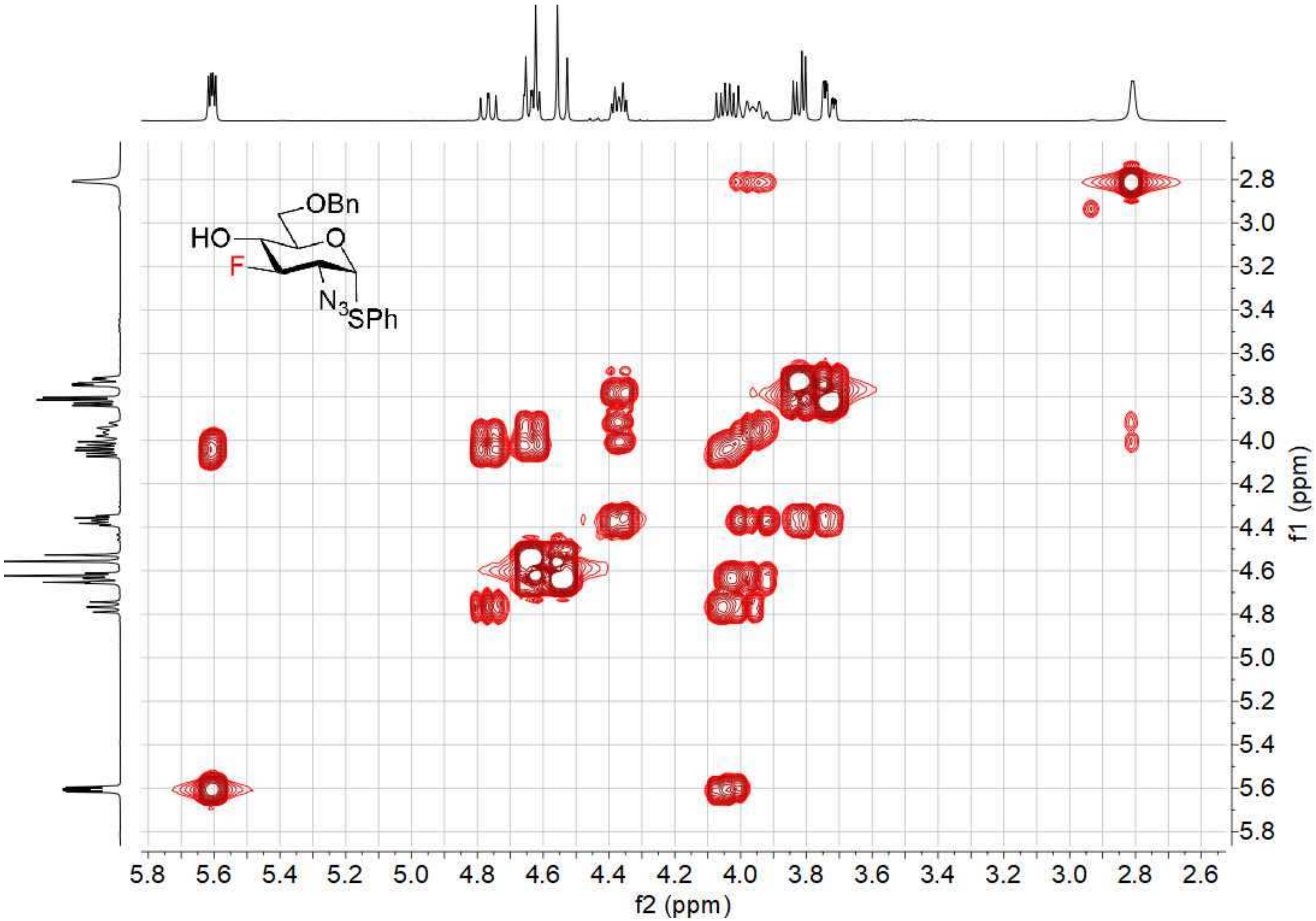
^{13}C NMR (100 MHz, CDCl$_3$) α-35



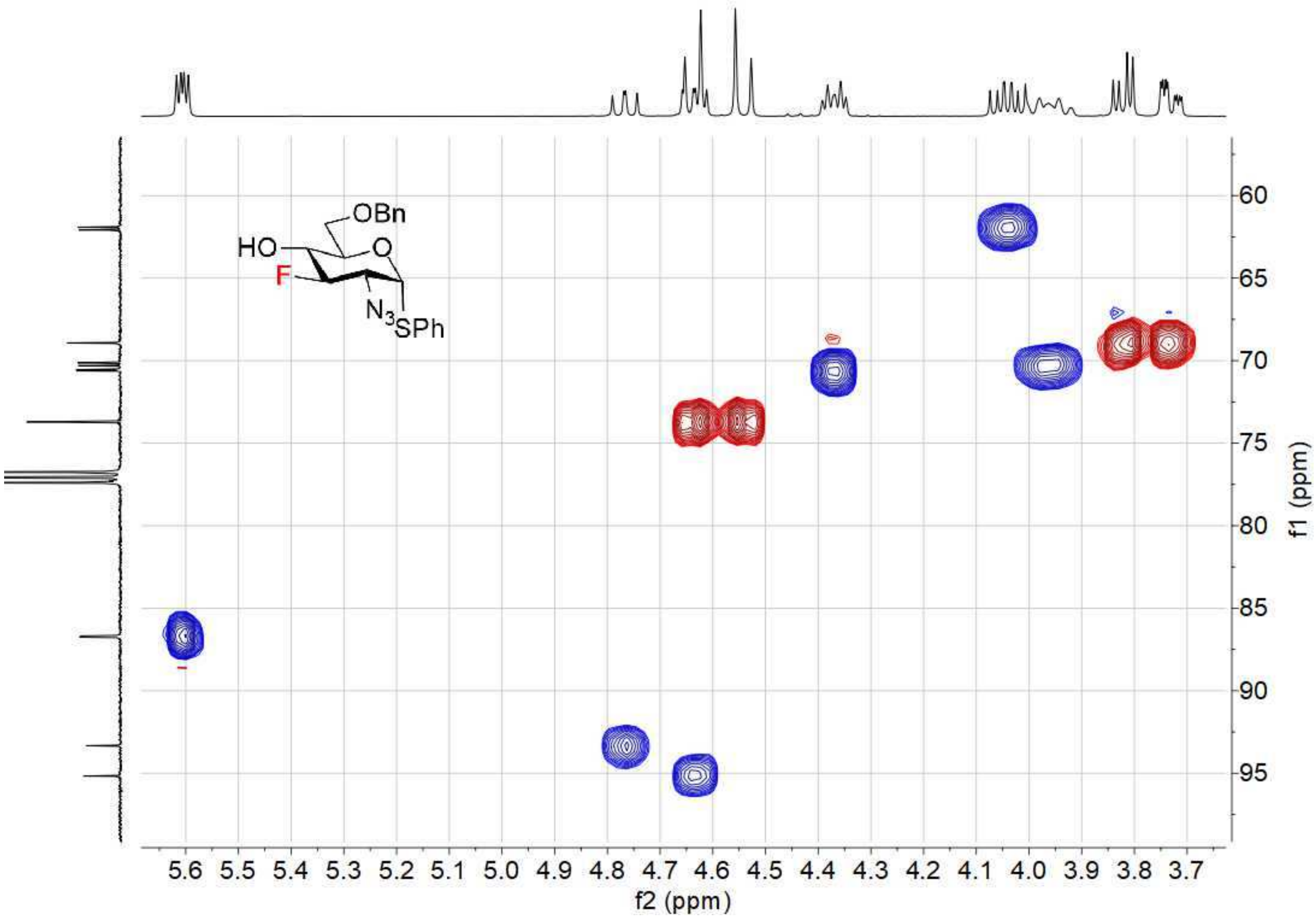
[19]F NMR (376 MHz, $CDCl_3$) α-35



^{1}H-^{1}H COSY α-35

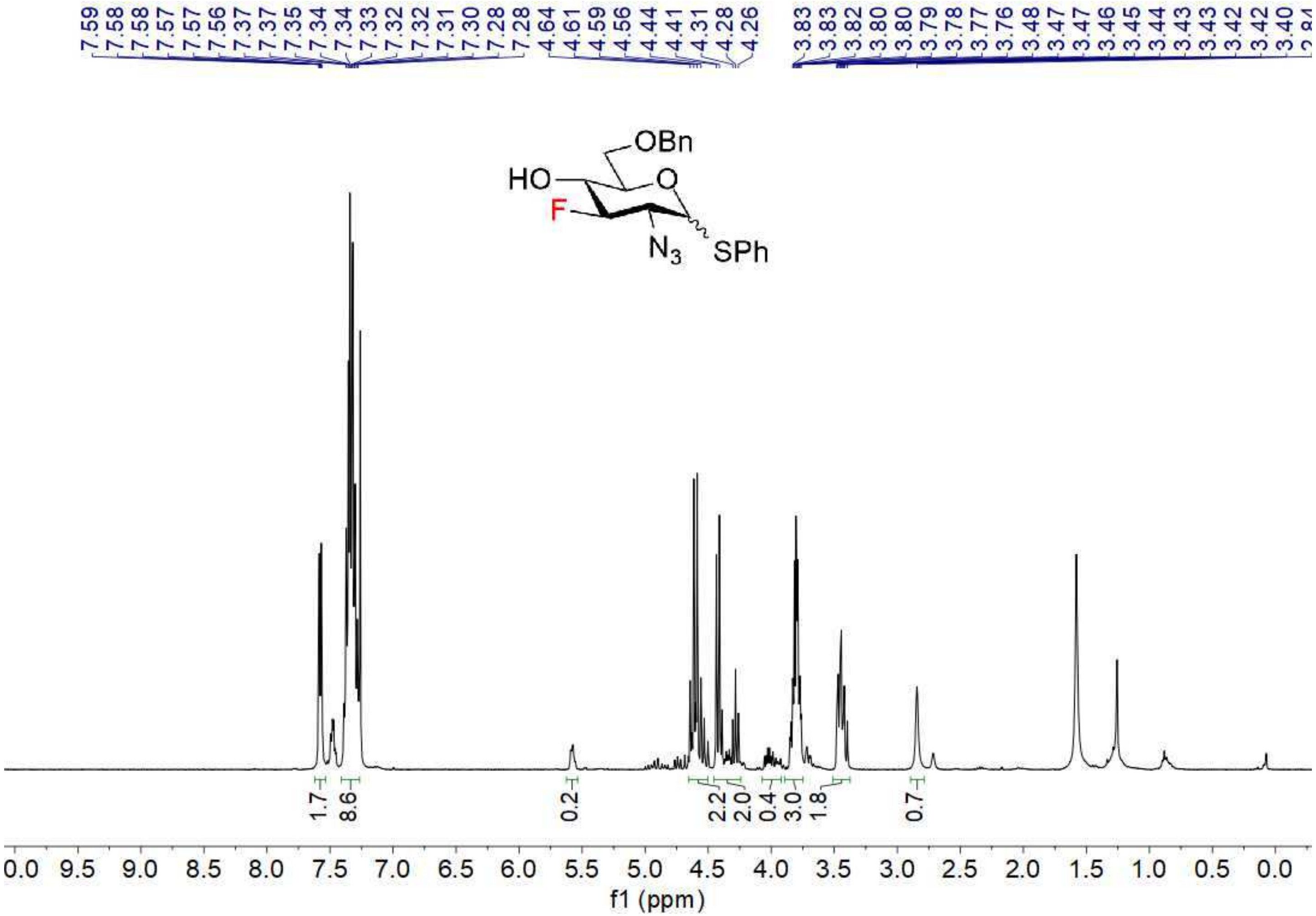


^{1}H-^{13}C HSQC α-35

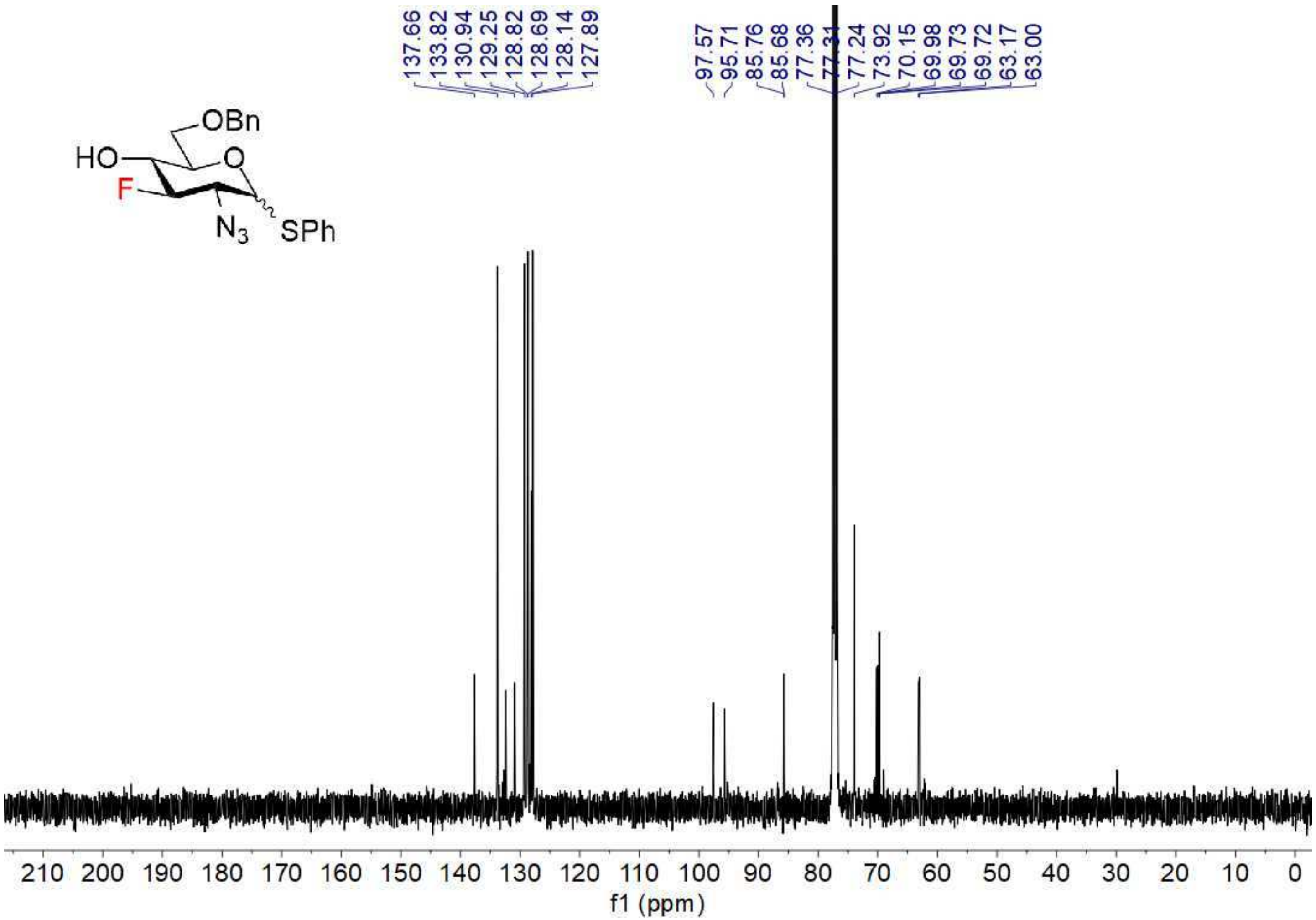


NMR COMPOUND 35 (α/β ca. 1/5)

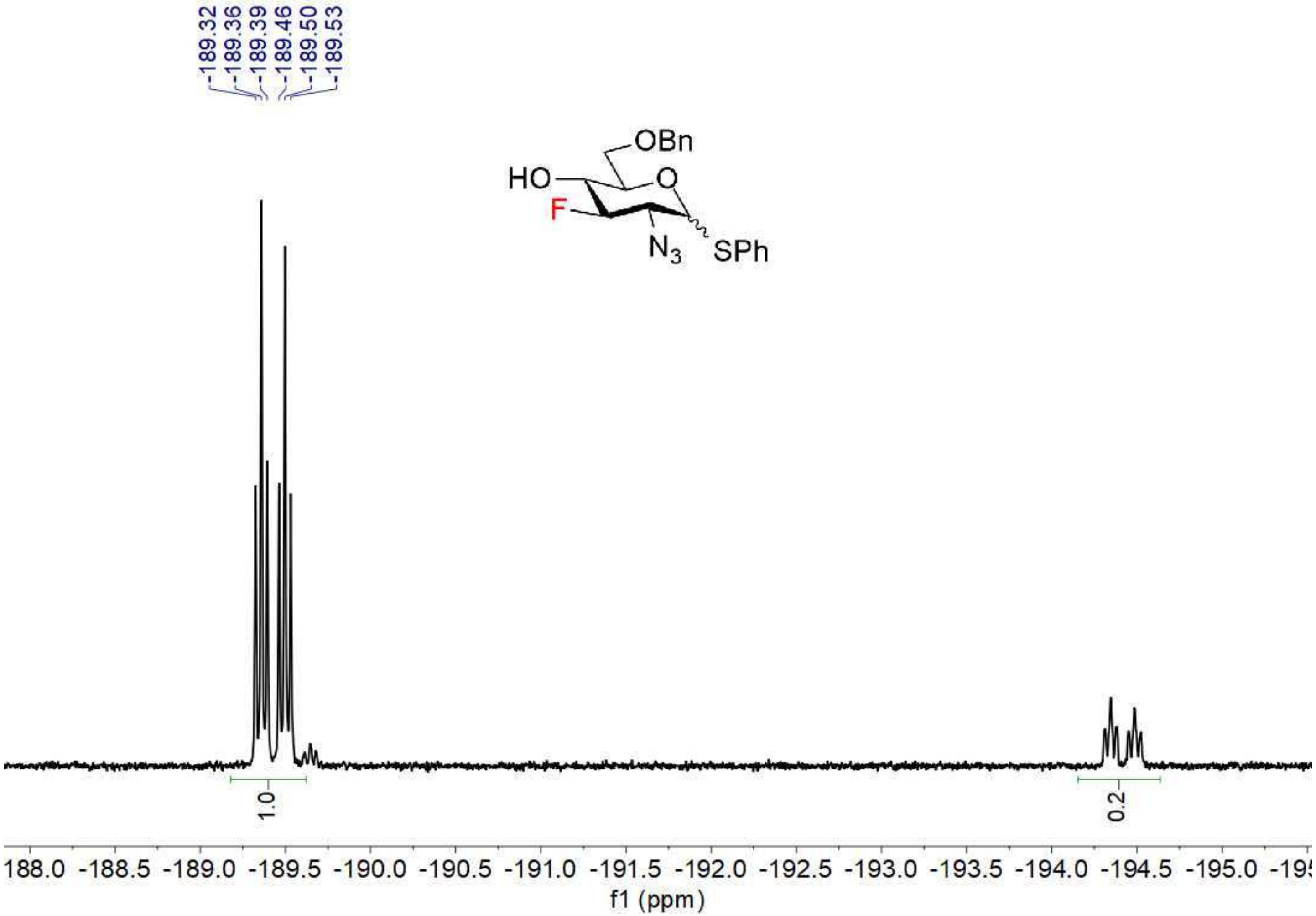
[1]H NMR (400 MHz, $CDCl_3$) 35 (α/β ca. 1/5)



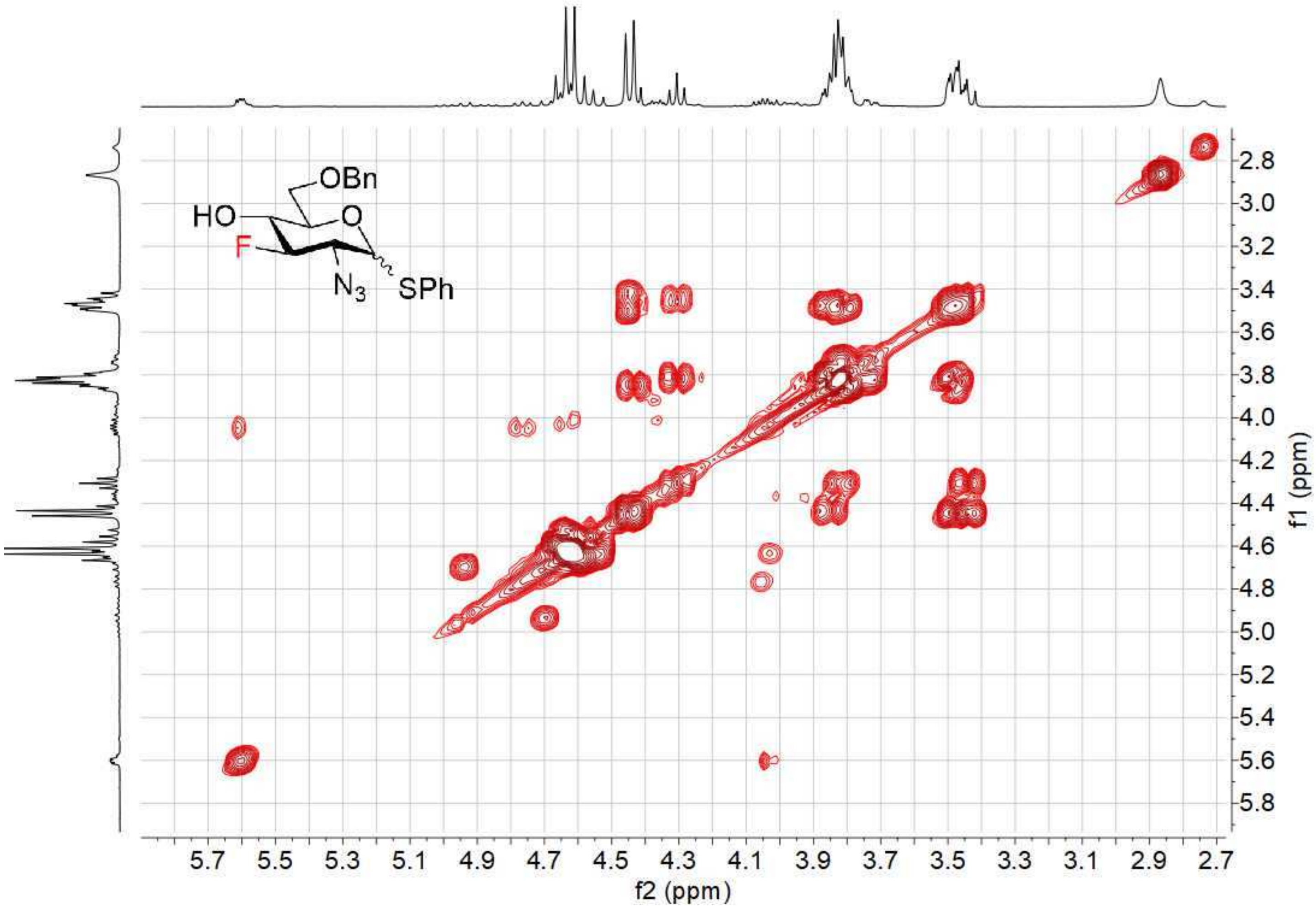
^{13}C NMR (100 MHz, $CDCl_3$) 35 (α/β ca. 1/5)



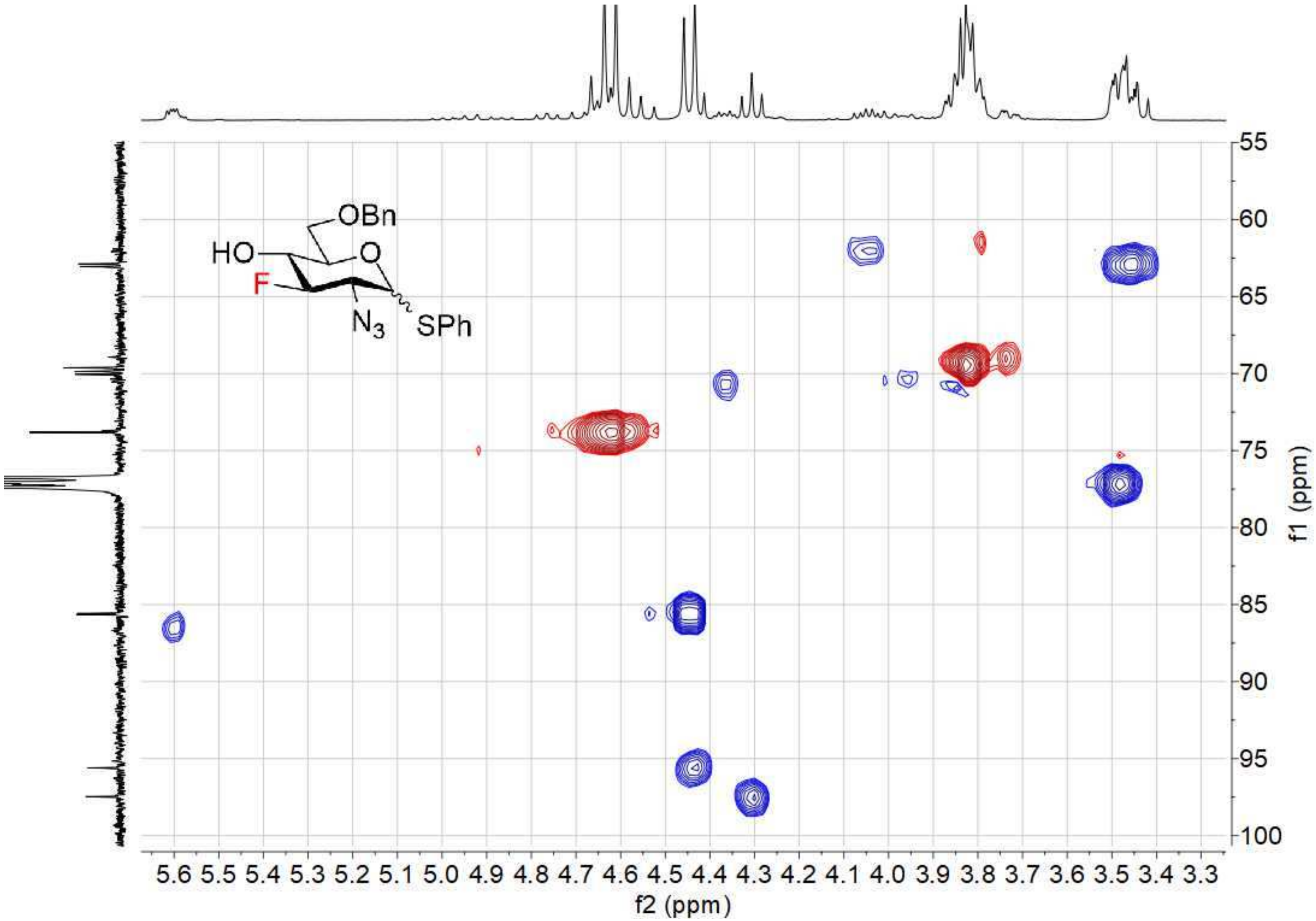
^{19}F NMR (376 MHz, CDCl$_3$) 35 (α/β ca. 1/5)



[1]H-[1]H COSY 35 (α/β ca. 1/5)

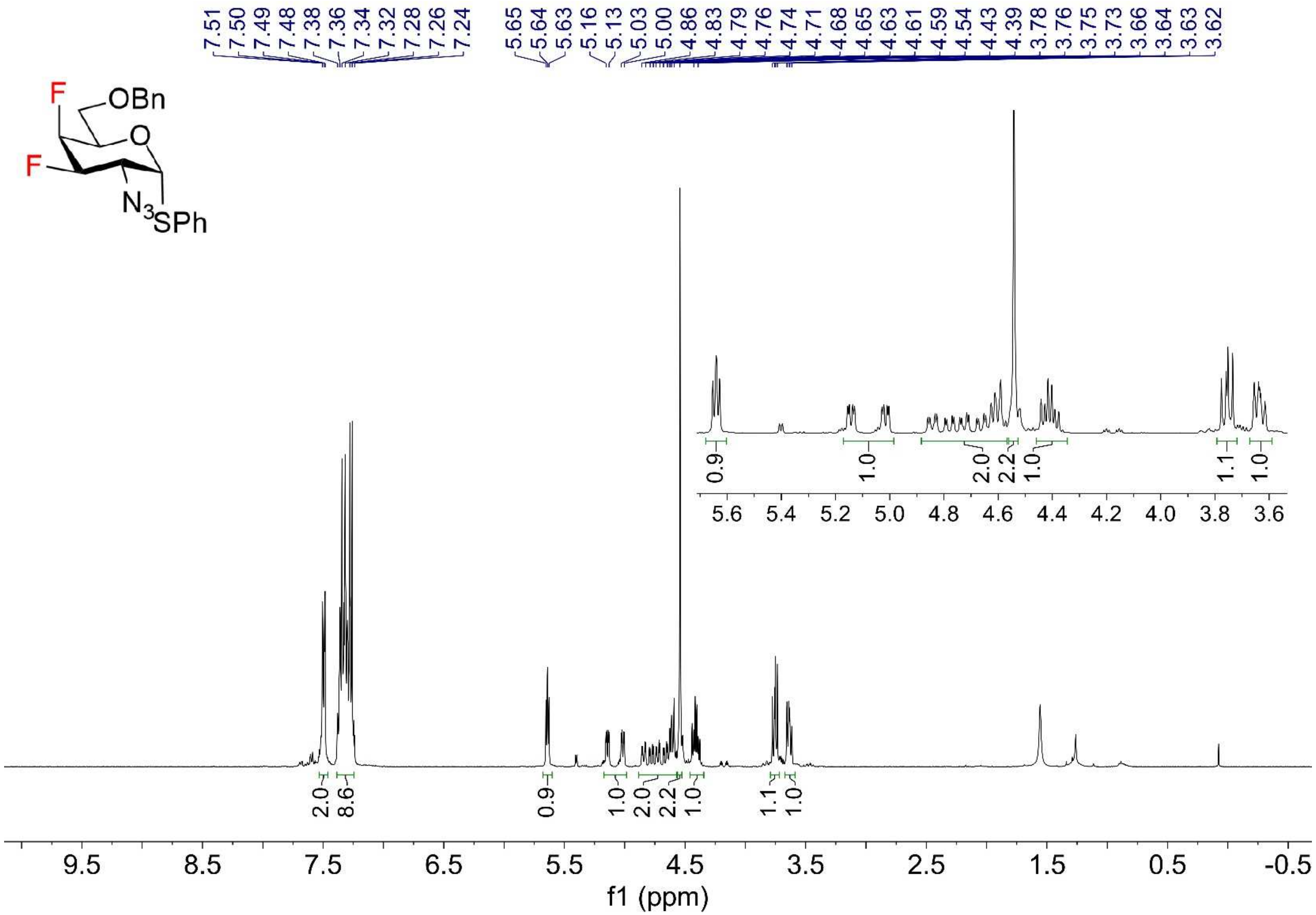


^{1}H-^{13}C HSQC 35 (α/β ca. 1/5)

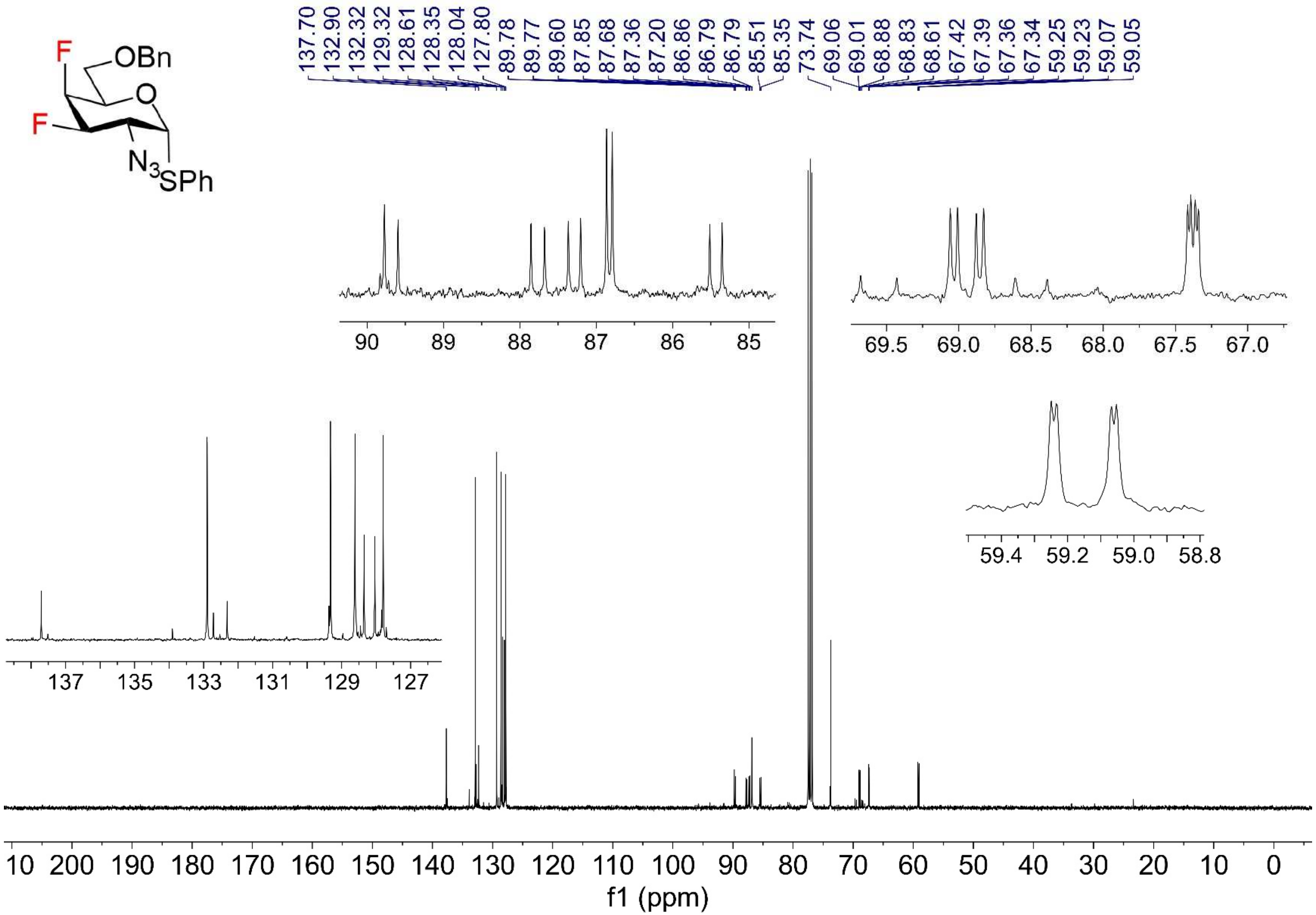


NMR COMPOUND α-36

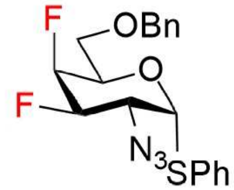
[1]H NMR (400 MHz, CDCl$_3$) α-36

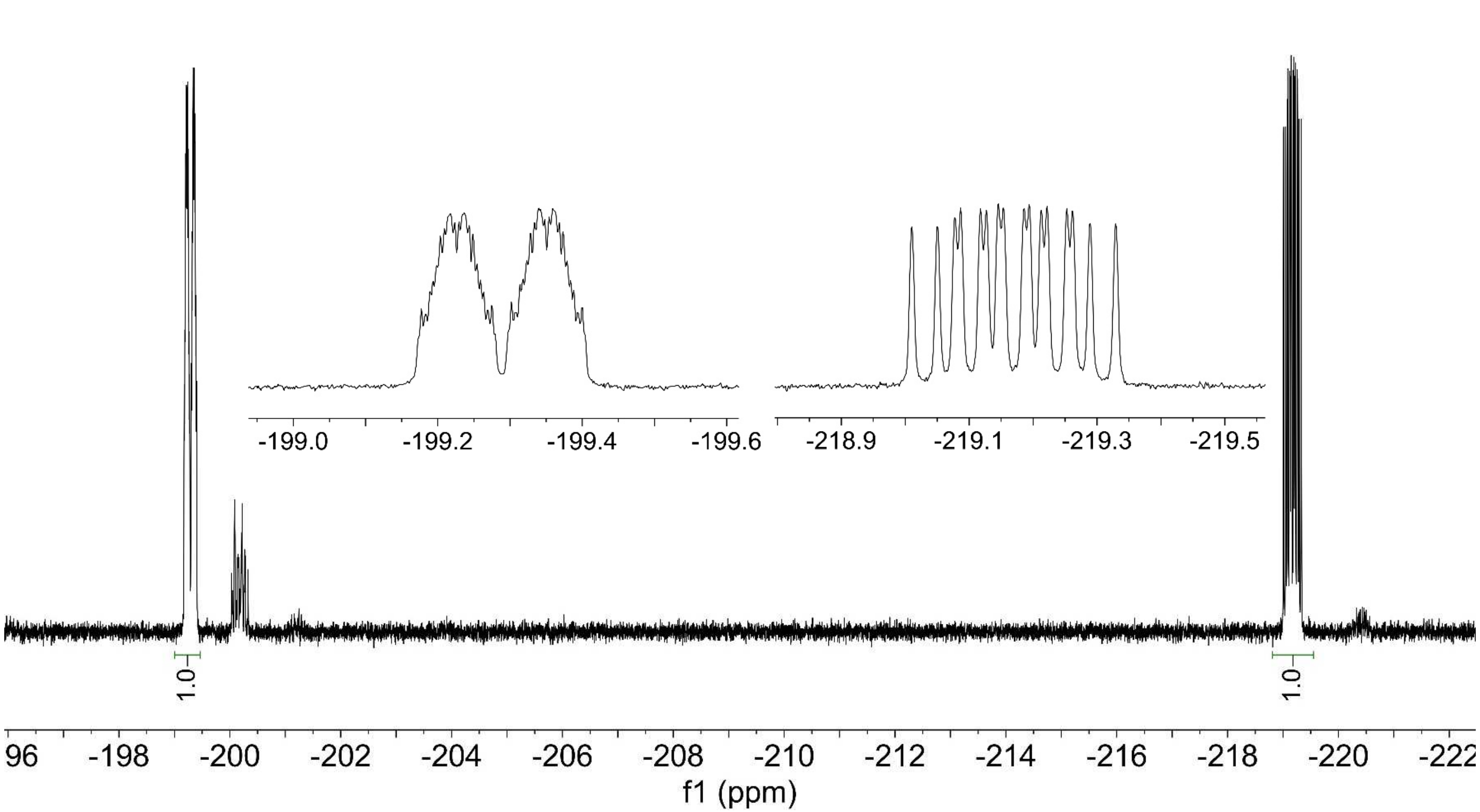


^{13}C NMR (100 MHz, CDCl$_3$) α-36

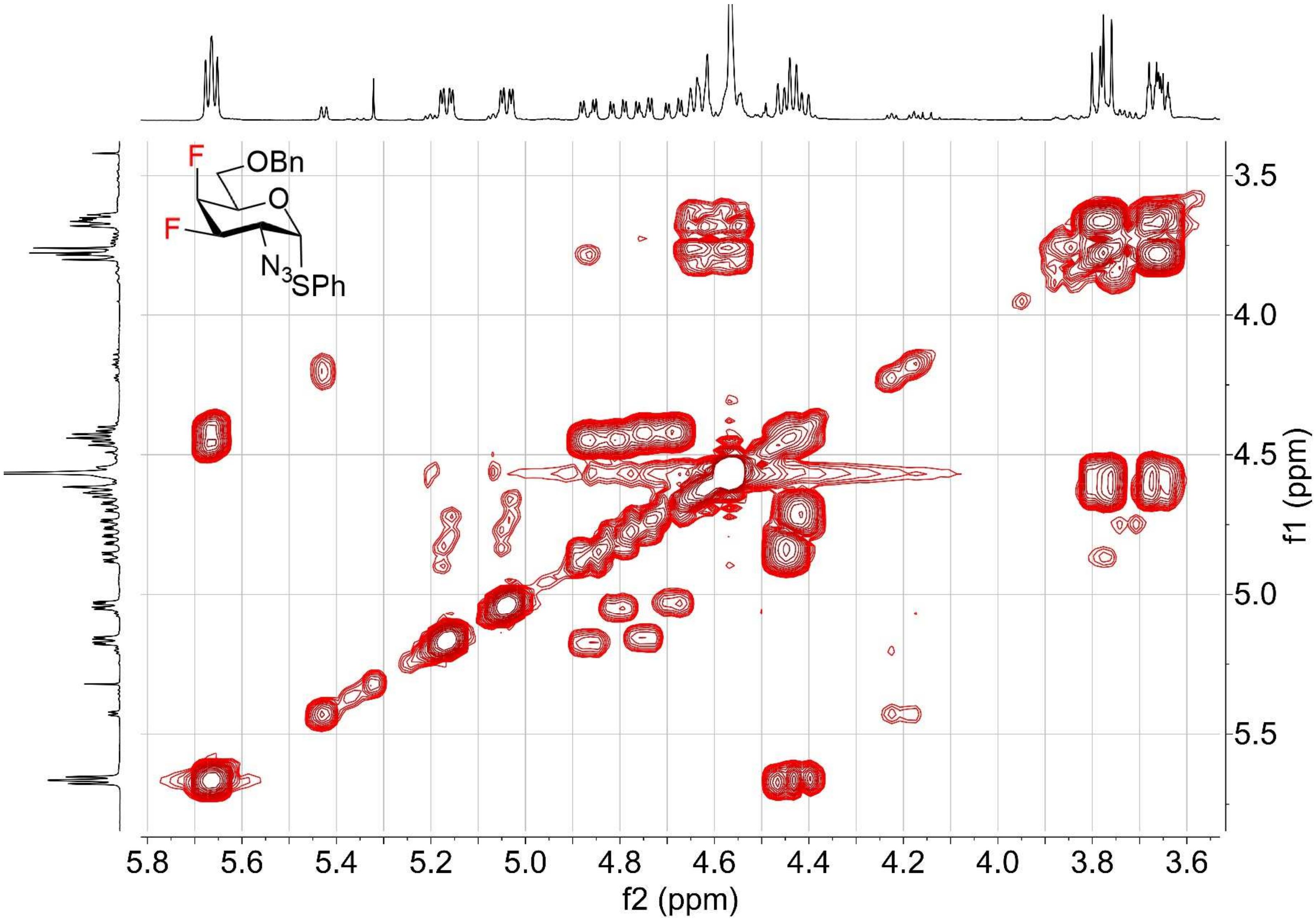


^{19}F NMR (376 MHz, CDCl$_3$) α-36

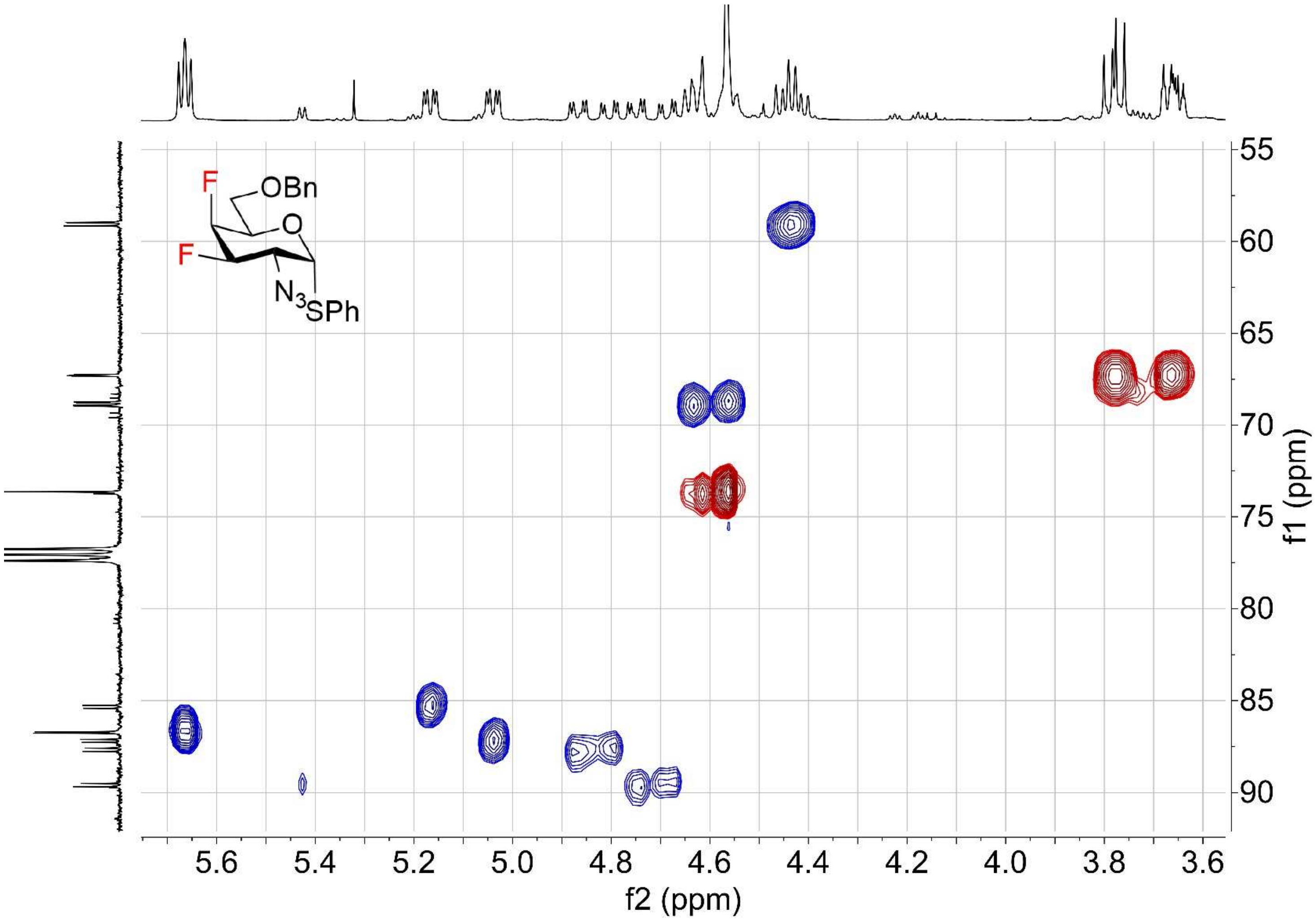




^{1}H-^{1}H COSY α-36

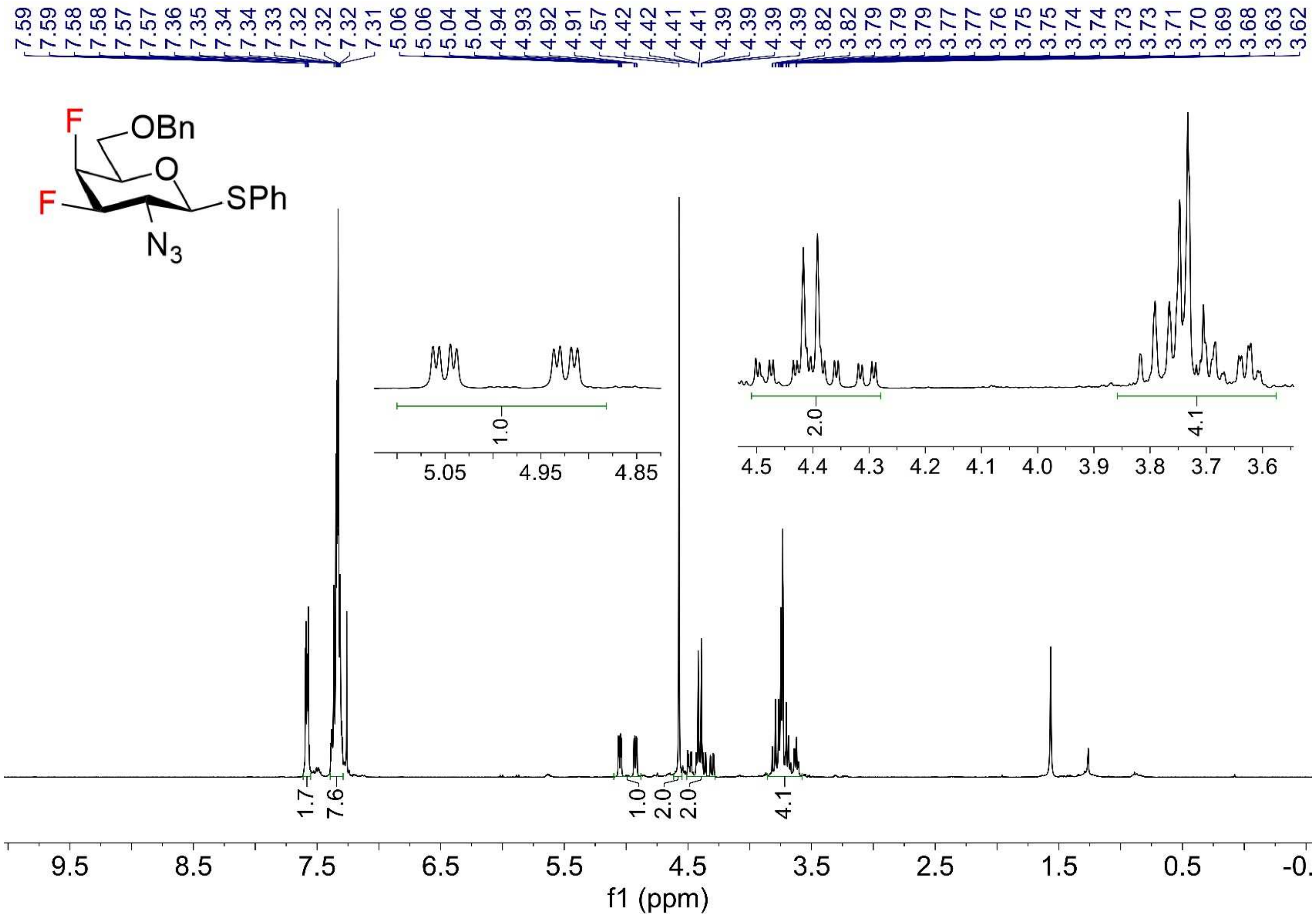


^{1}H-^{13}C HSQC α-36

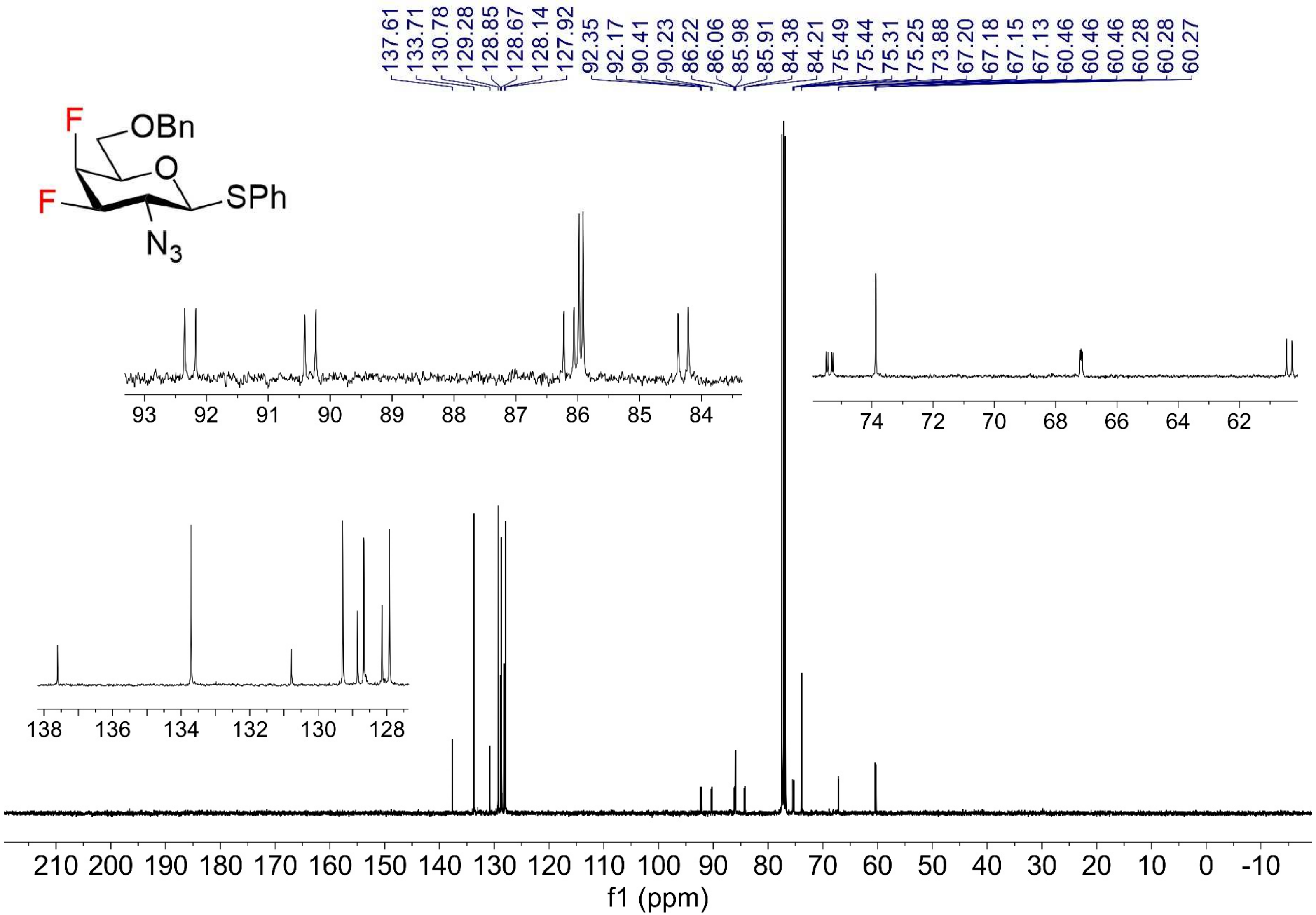


NMR COMPOUND β-36

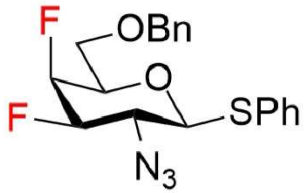
^{1}H NMR (400 MHz, $CDCl_3$) β-38

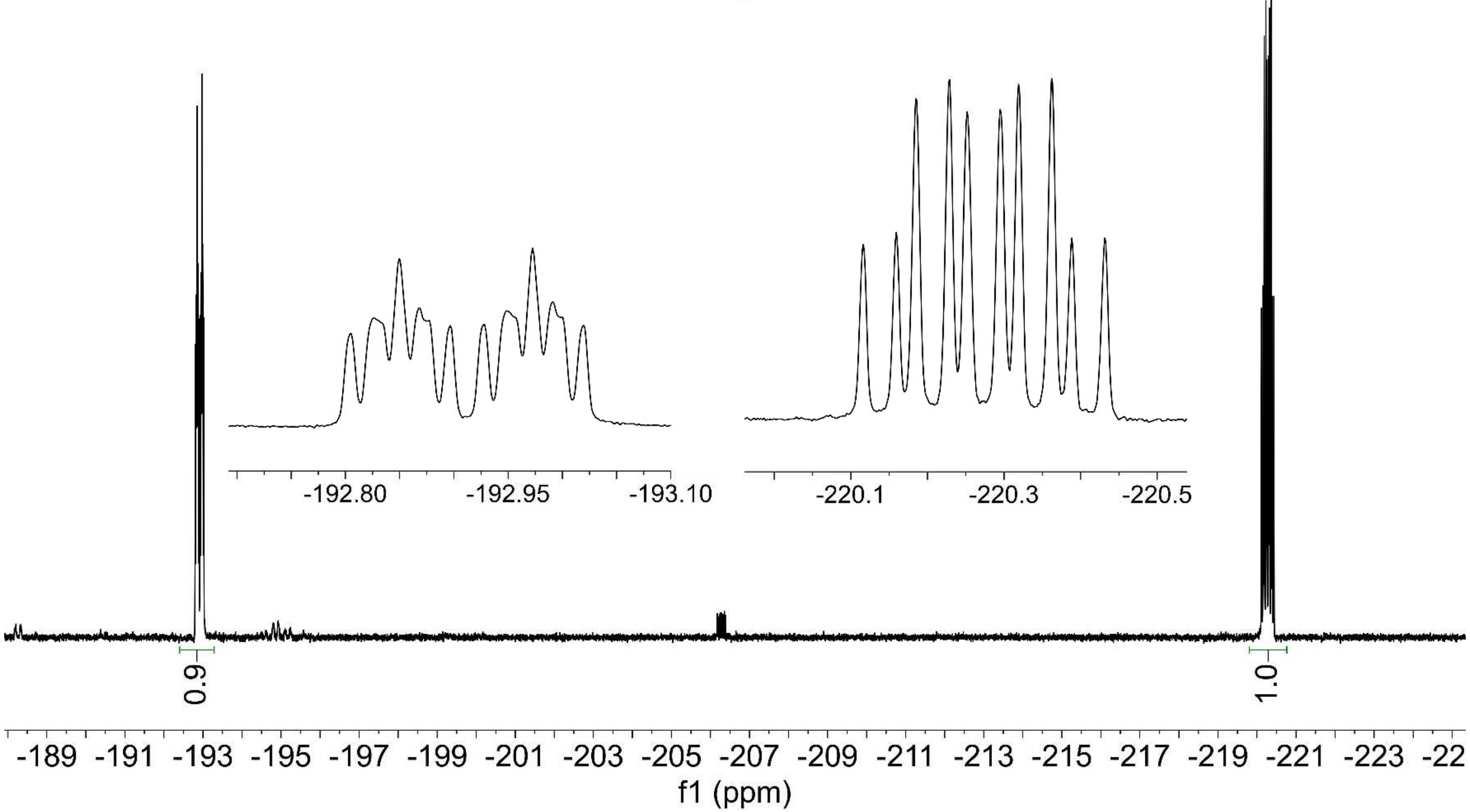


^{13}C NMR (100 MHz, $CDCl_3$) β-36

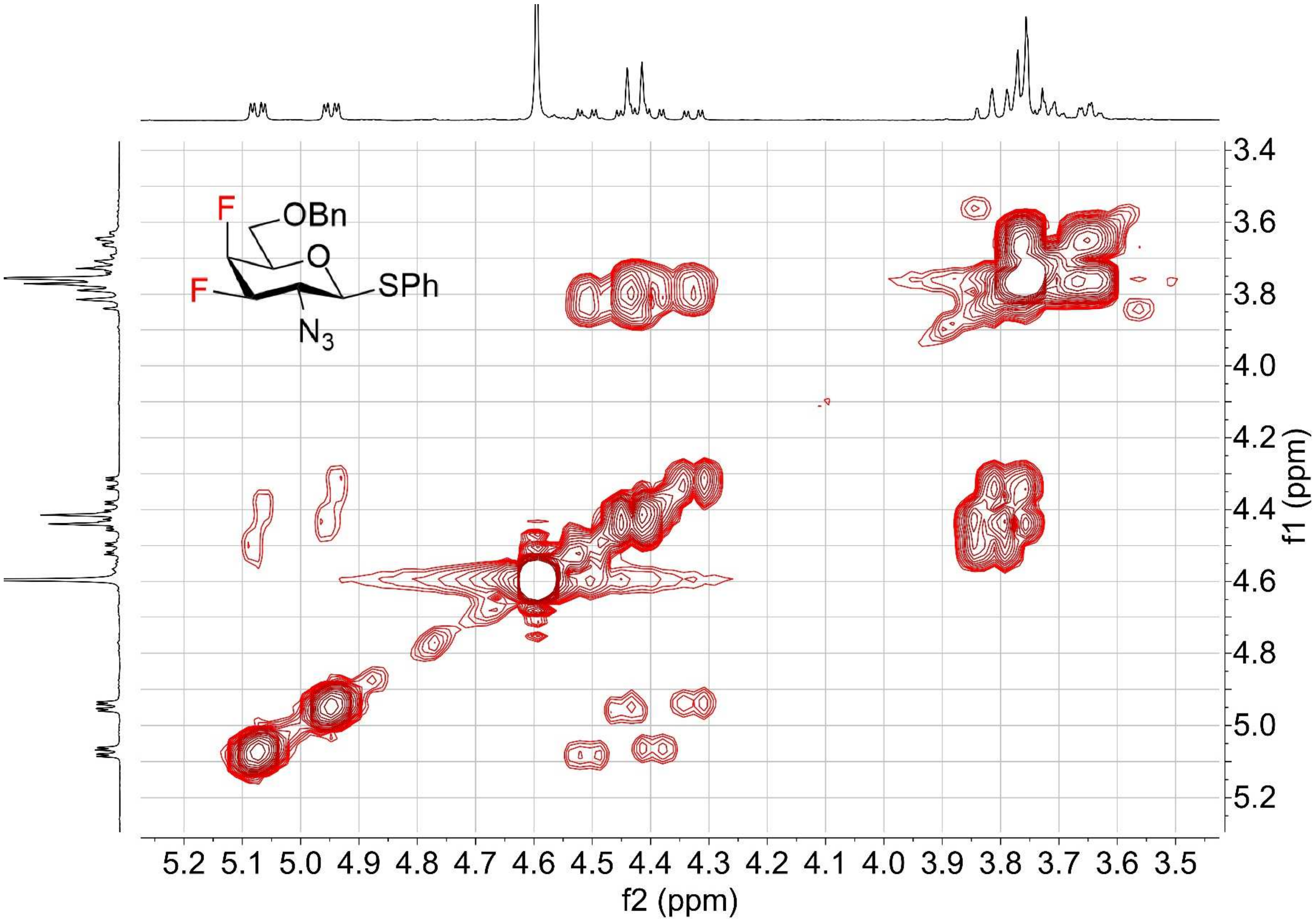


^{19}F NMR (376 MHz, $CDCl_3$) β-36

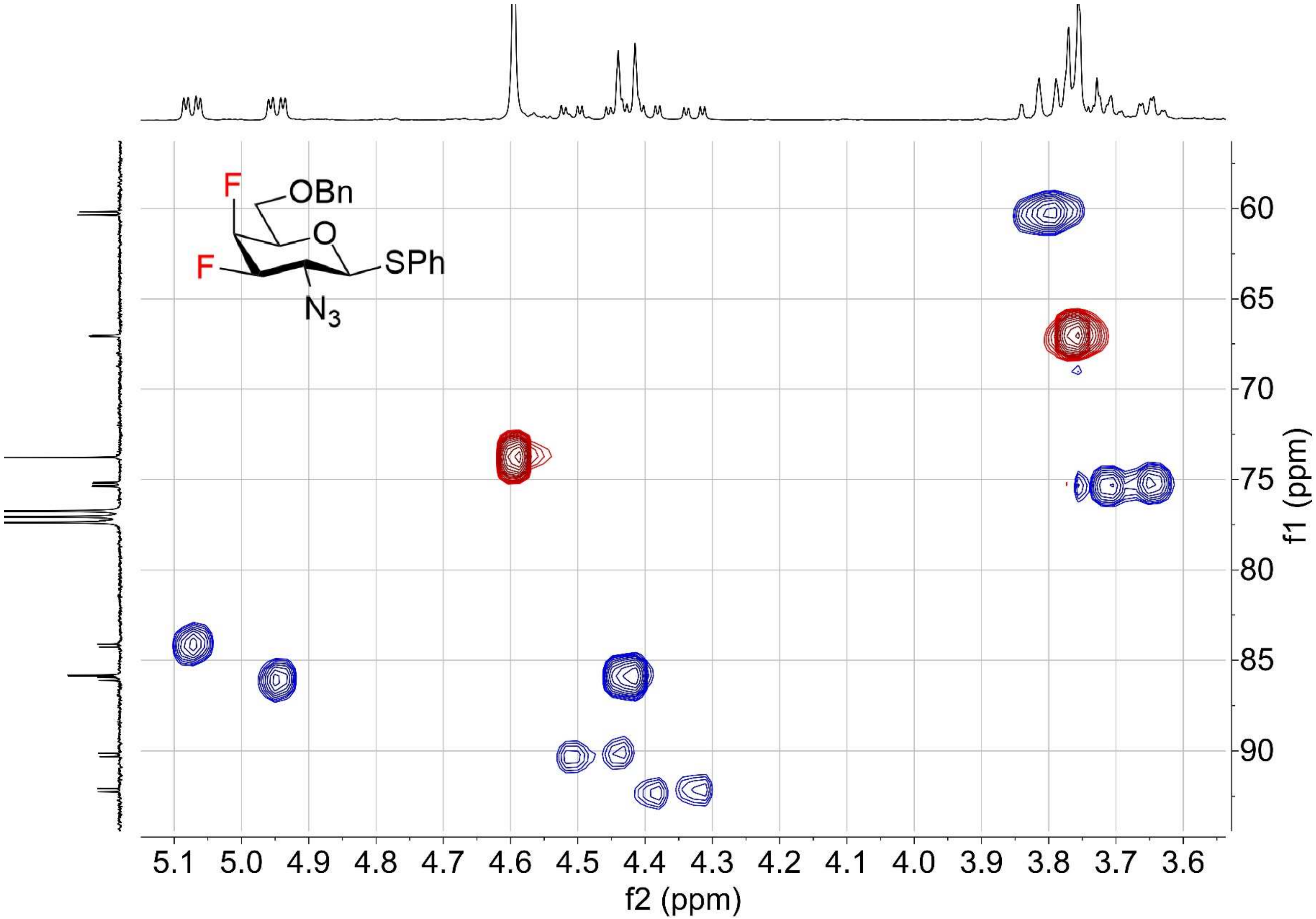




[1]H-[1]H COSY β-36

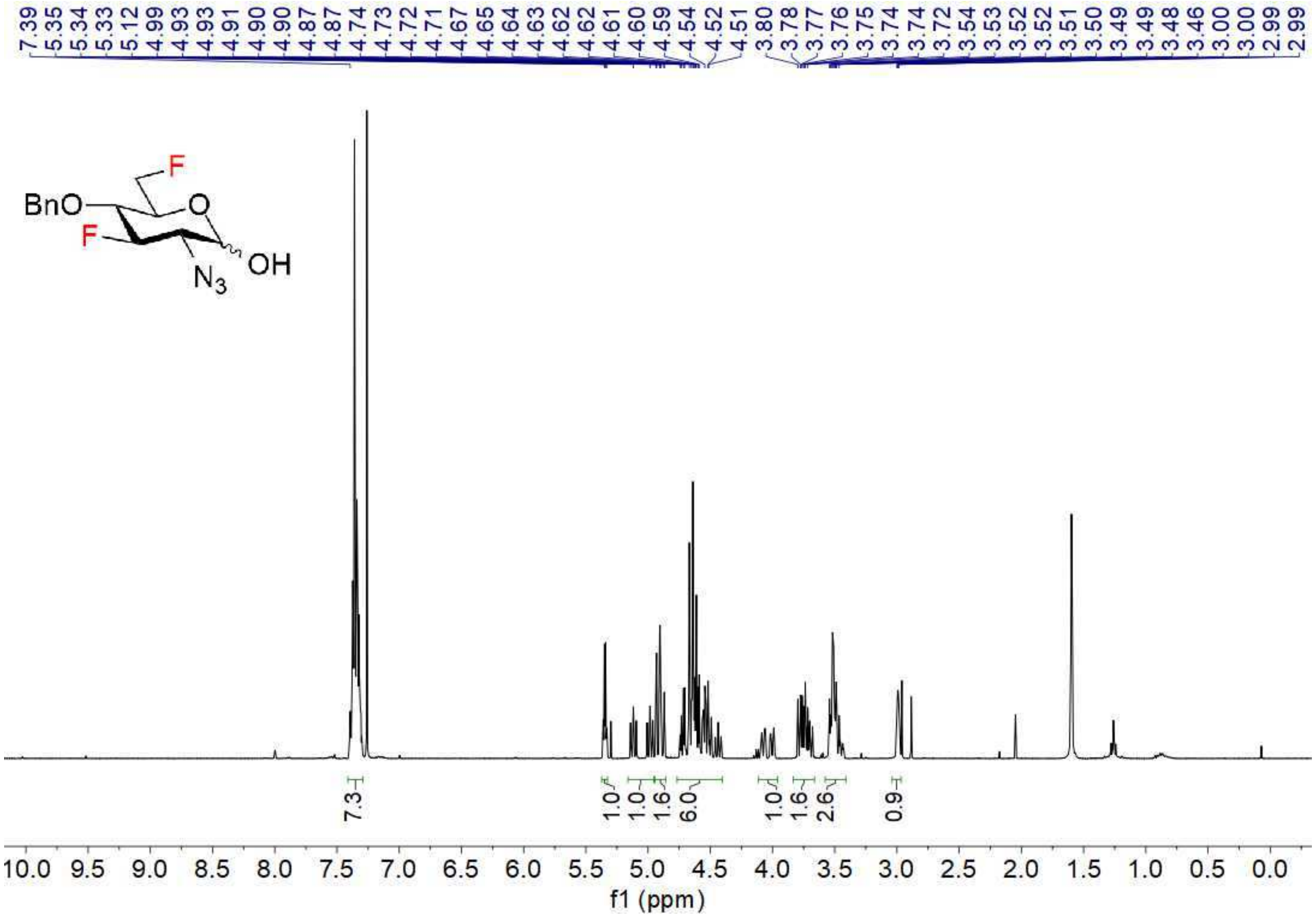


^{1}H-^{13}C HSQC β-36

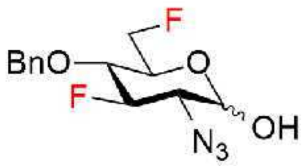


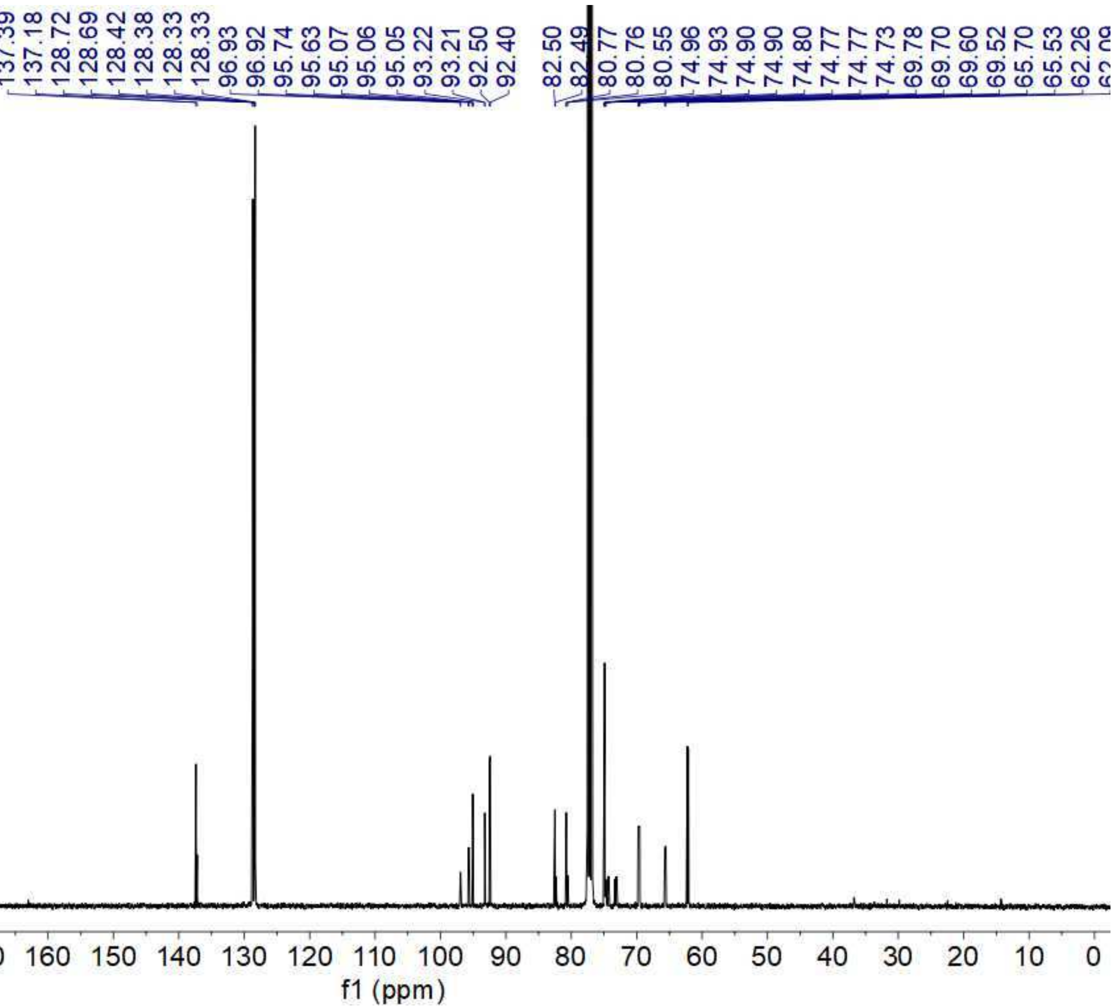
NMR COMPOUND 37

^{1}H NMR (400 MHz, $CDCl_3$) 37 (α/β ca. 5/3)

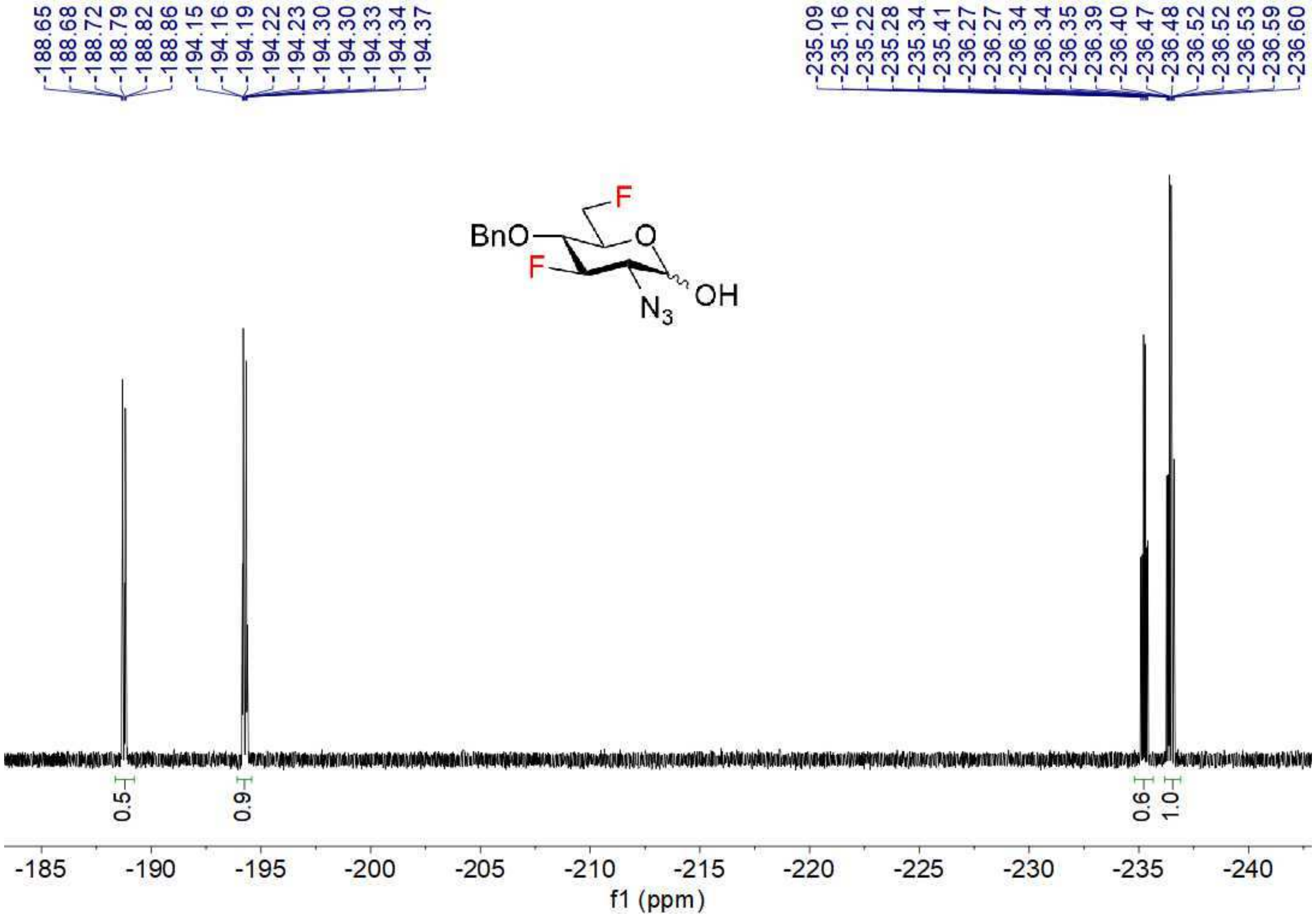


^{13}C NMR (100 MHz, CDCl$_3$) 37

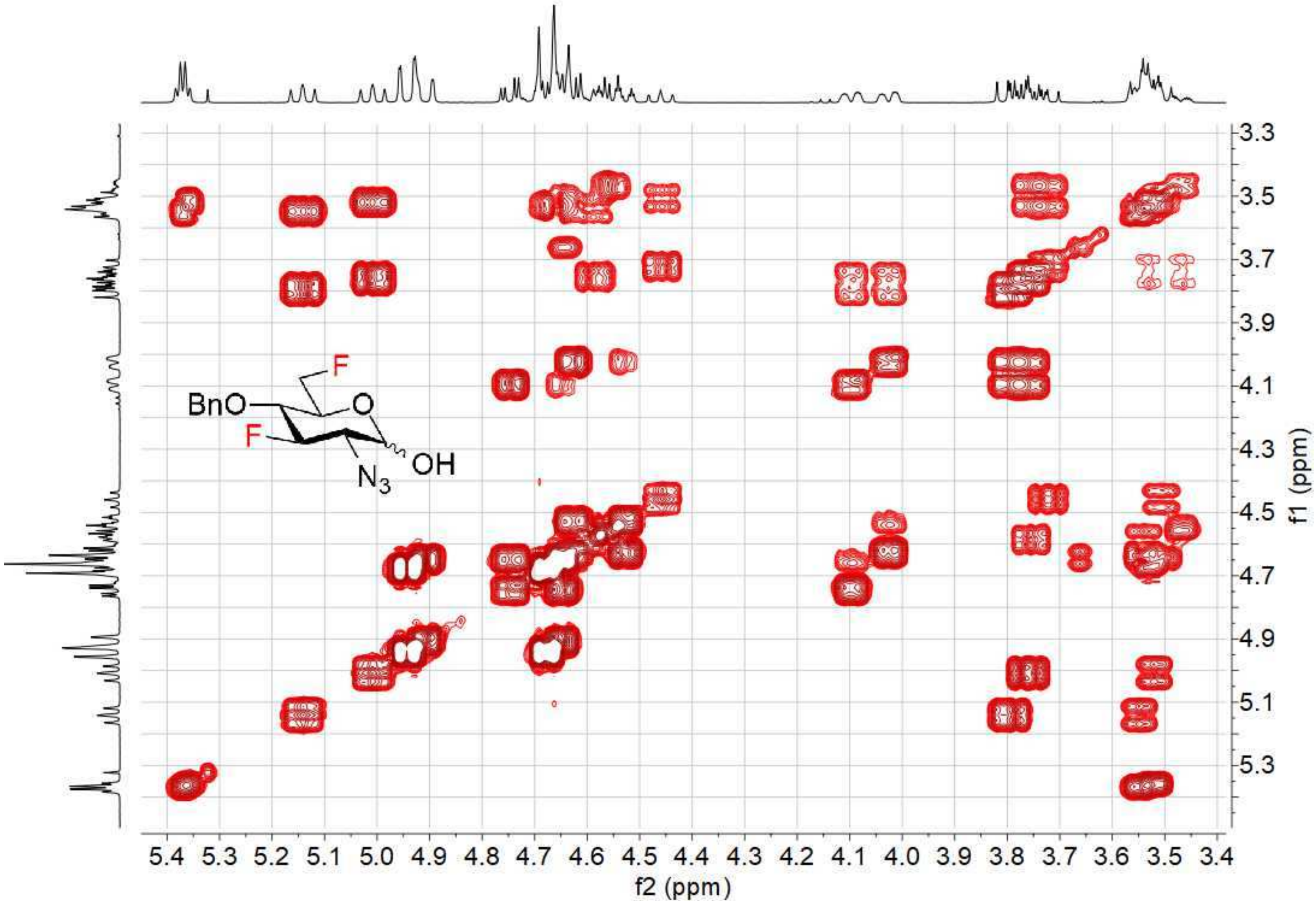




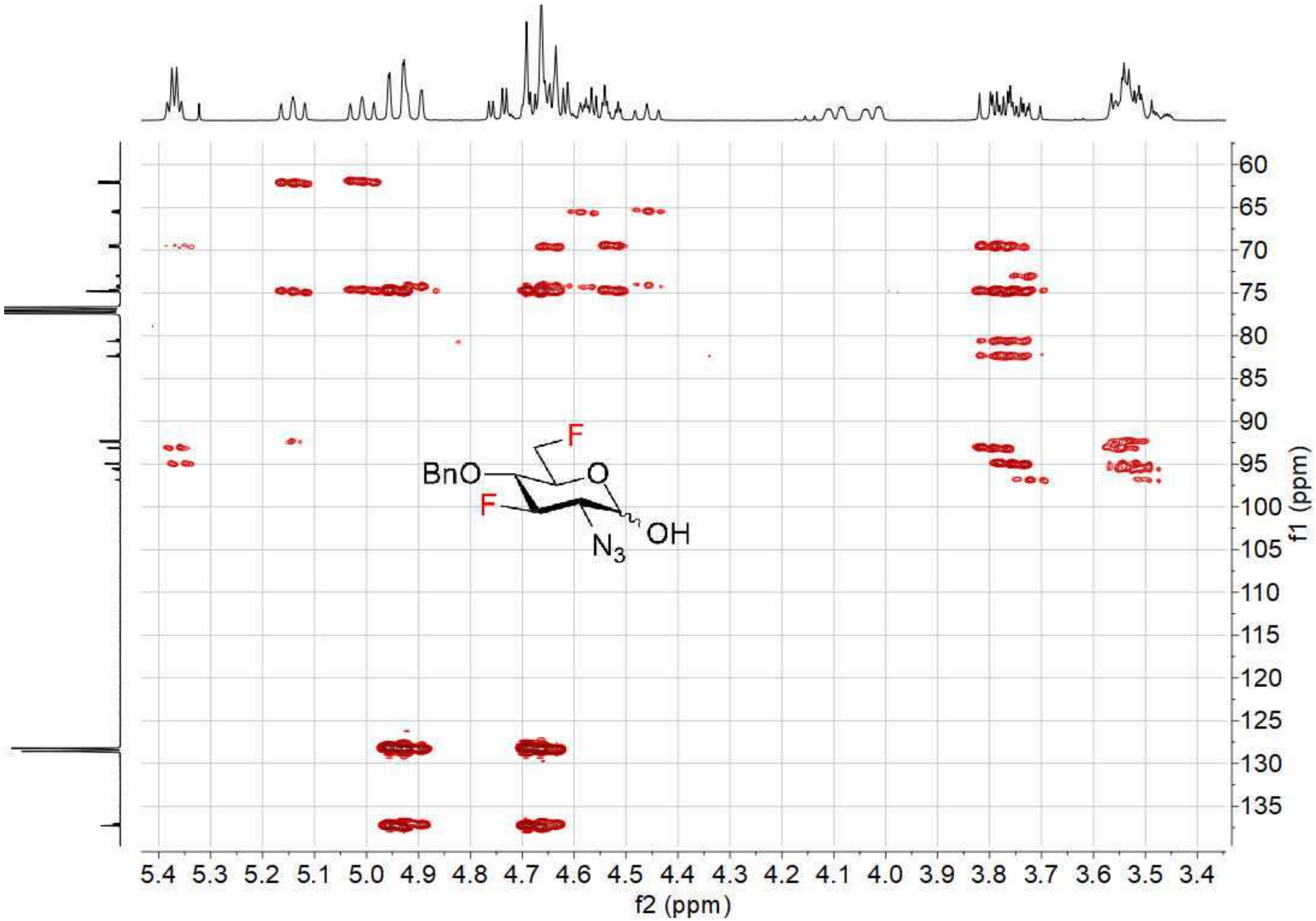
^{19}F NMR (376 MHz, $CDCl_3$) 37 (α/β ca. 5/3)



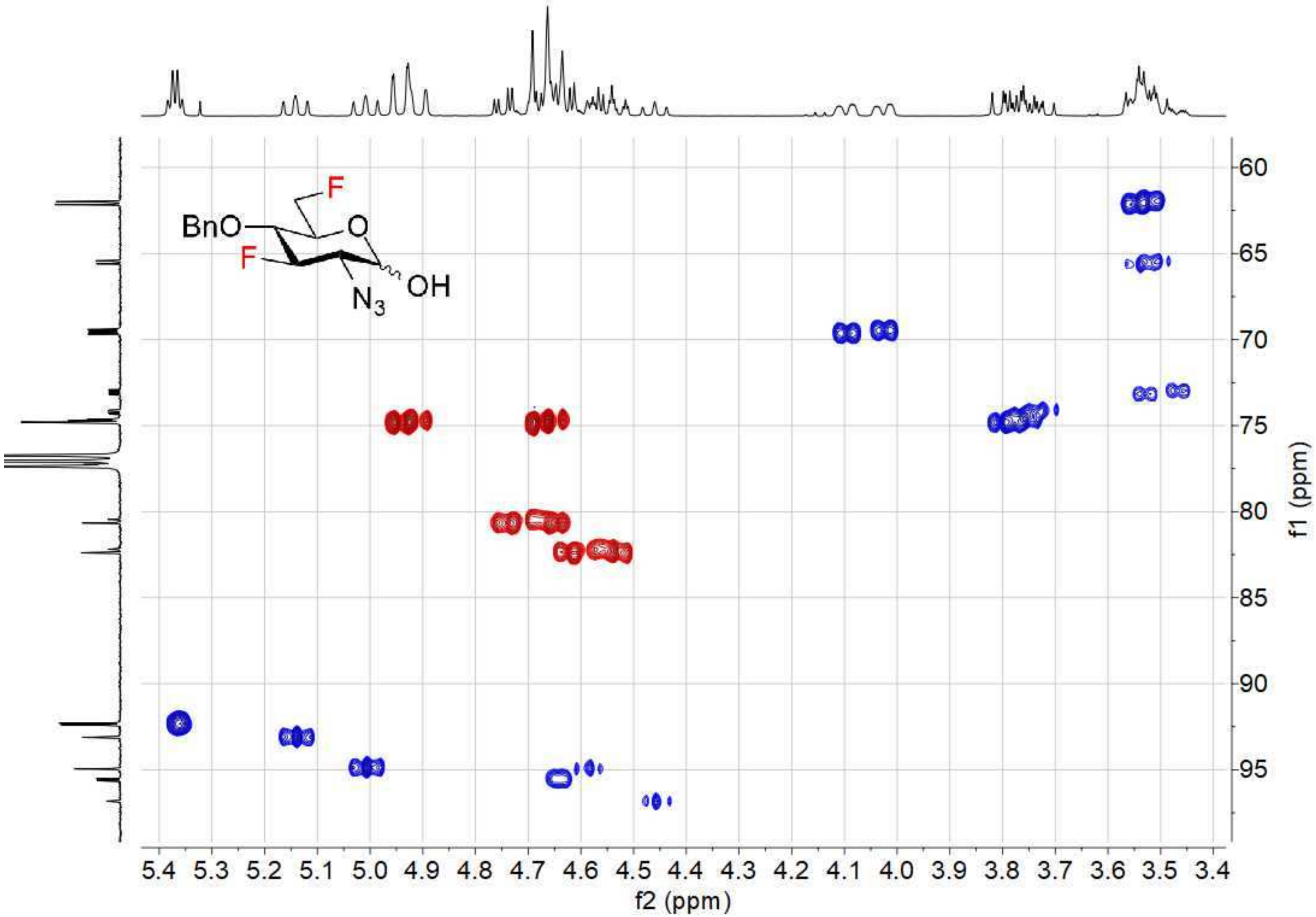
^{1}H-^{1}H COSY 37



[1]H-[13]C HMBC 37

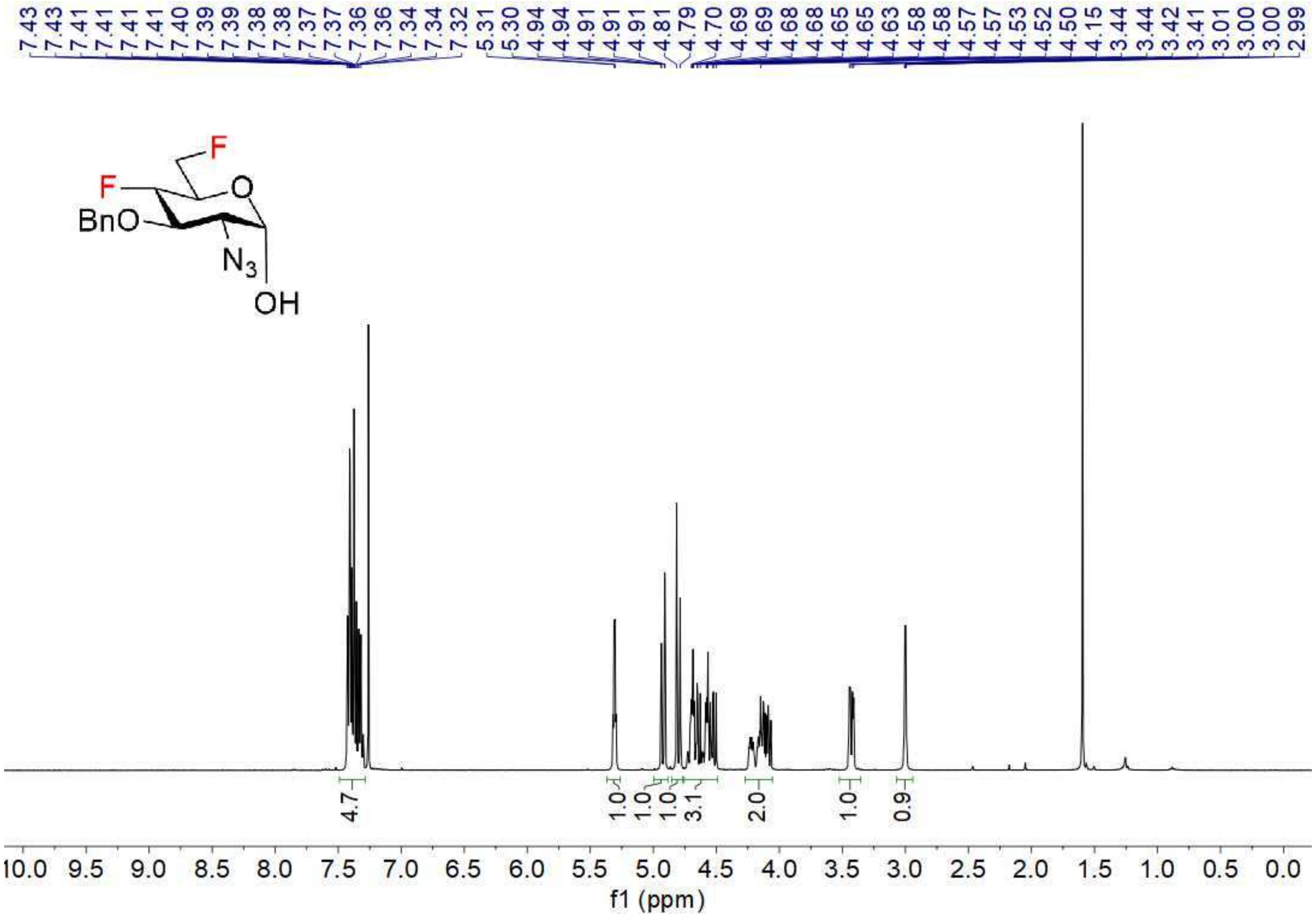


^{1}H-^{13}C HSQC 37

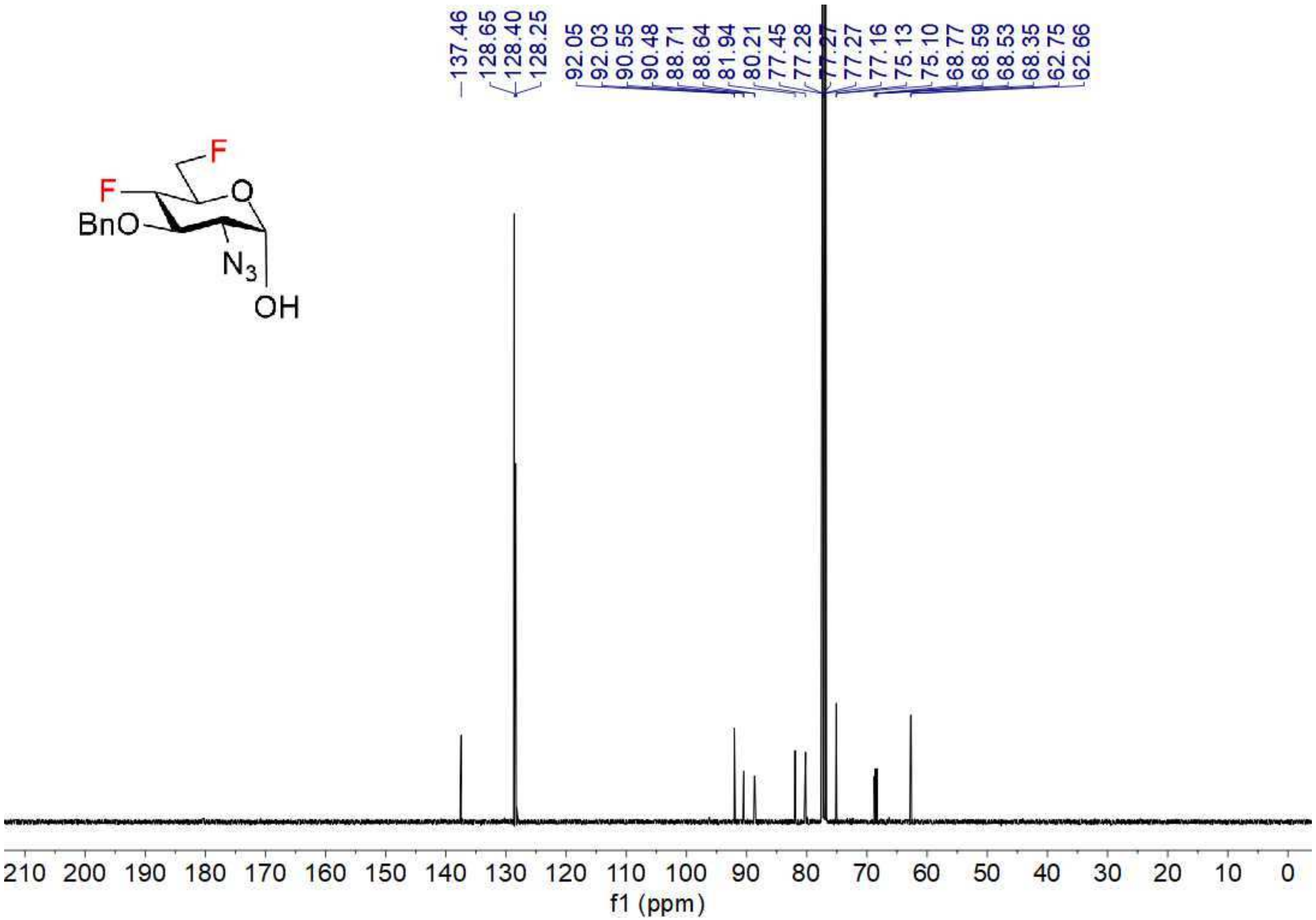


NMR COMPOUND 38

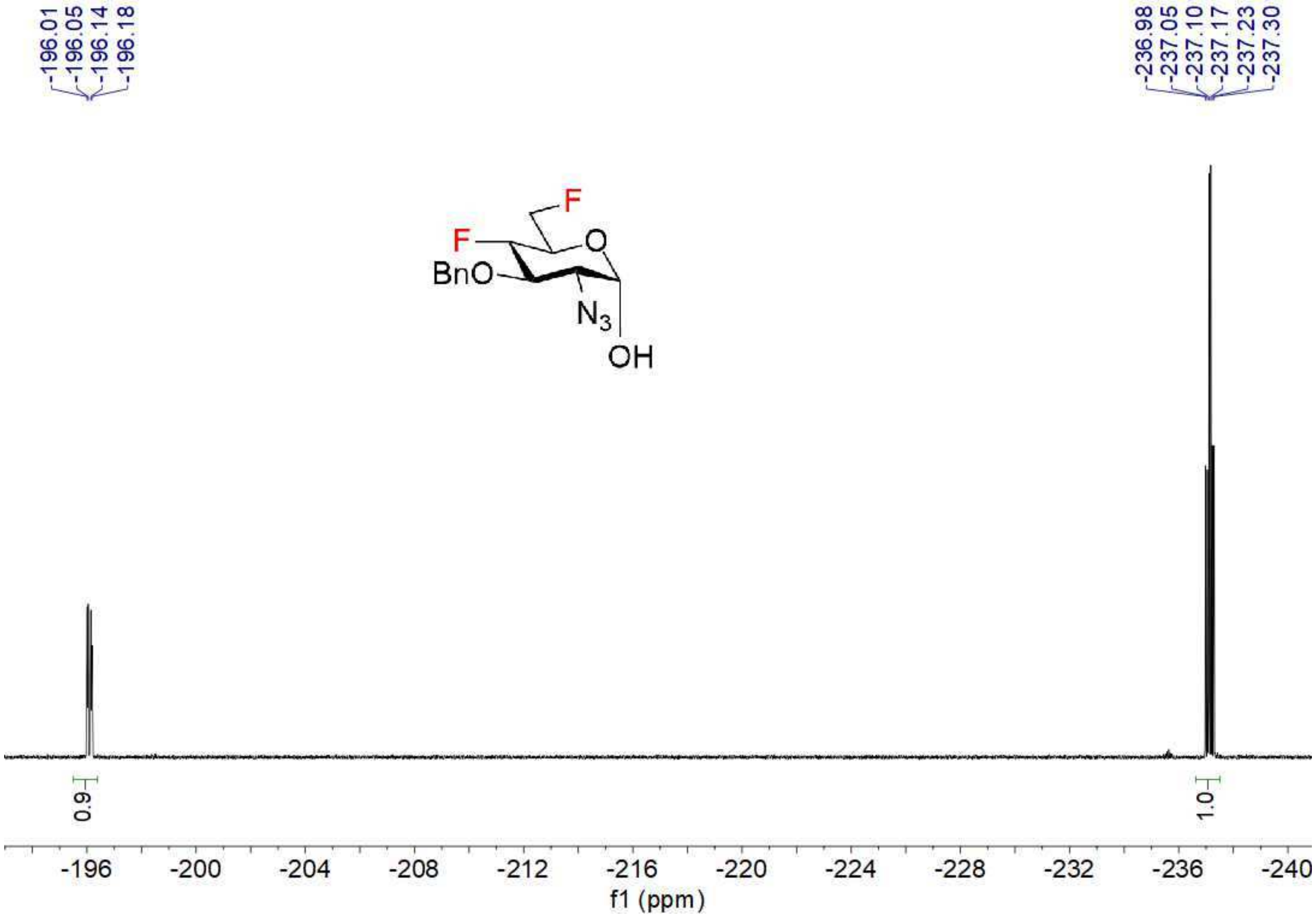
^{1}H NMR (400 MHz, $CDCl_3$) 38



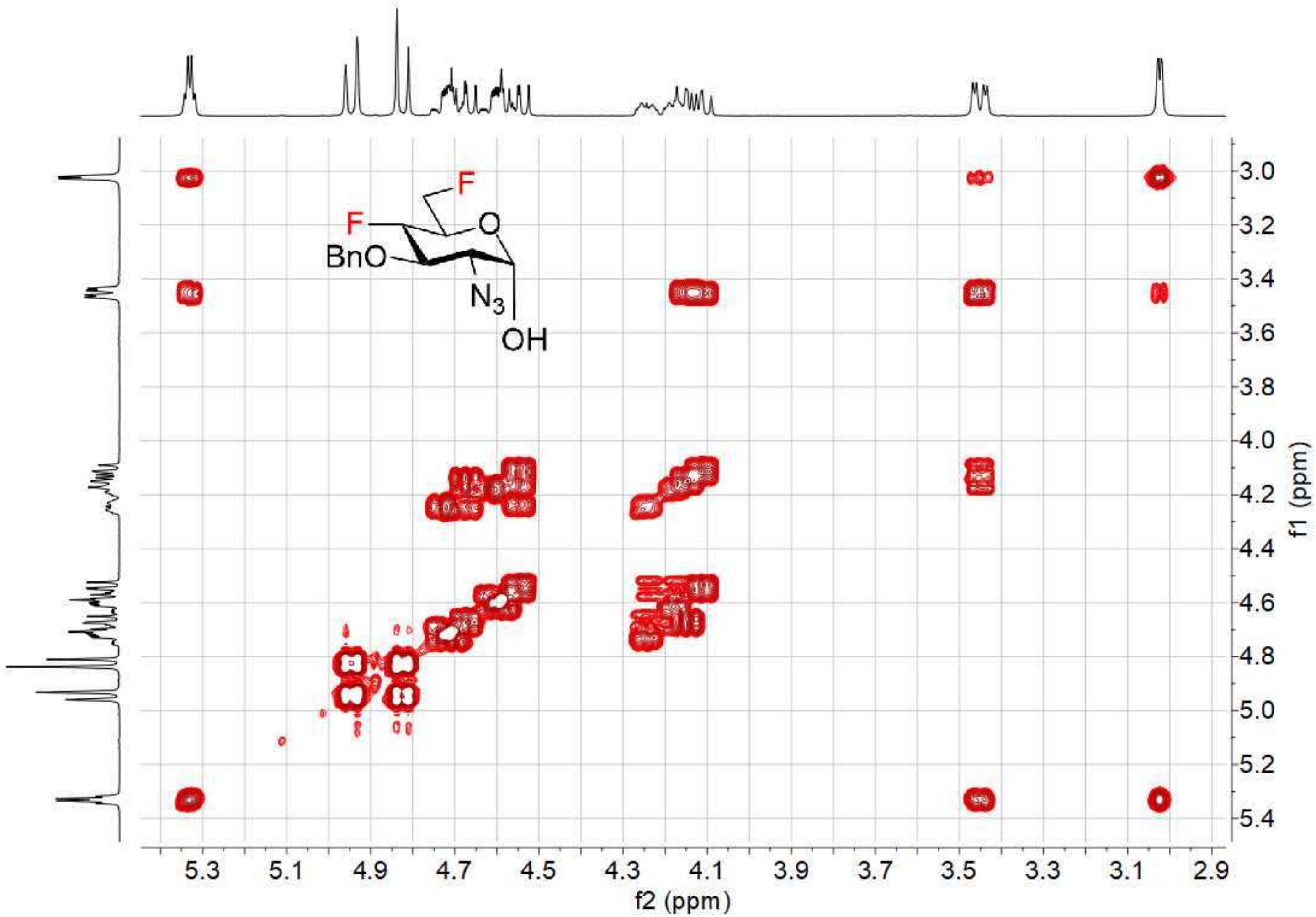
^{13}C NMR (100 MHz, $CDCl_3$) 38



[19]F NMR (376 MHz, $CDCl_3$) 38



^{1}H-^{1}H COSY 38


F
F
O
BnO
N3
OH
f1 (ppm)
f2 (ppm)
5.3
5.1
4.9
4.7
4.5
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4.1
3.9
3.7
3.5
3.3
3.1
2.9
3.0
3.2
3.4
3.6
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4.8
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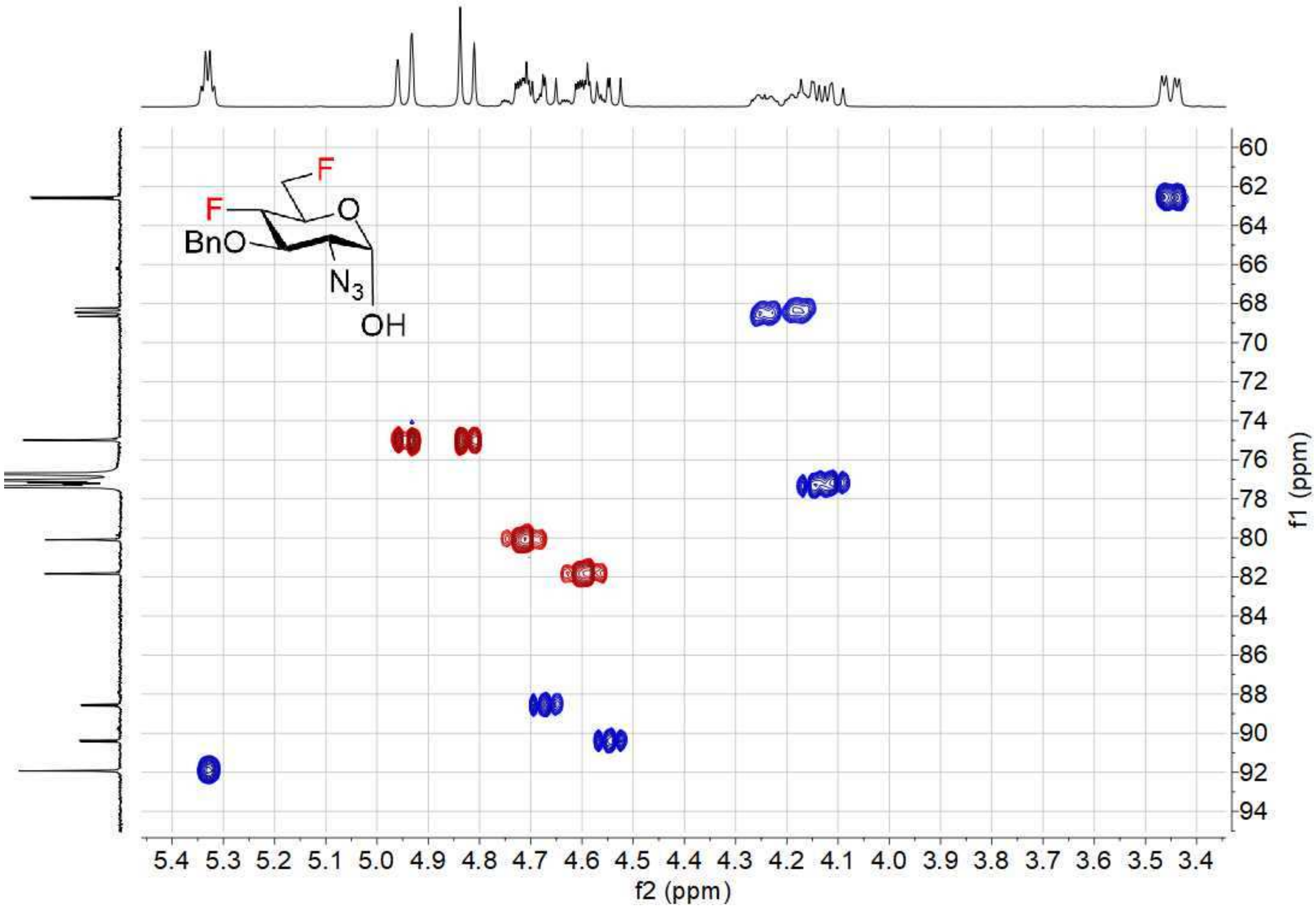
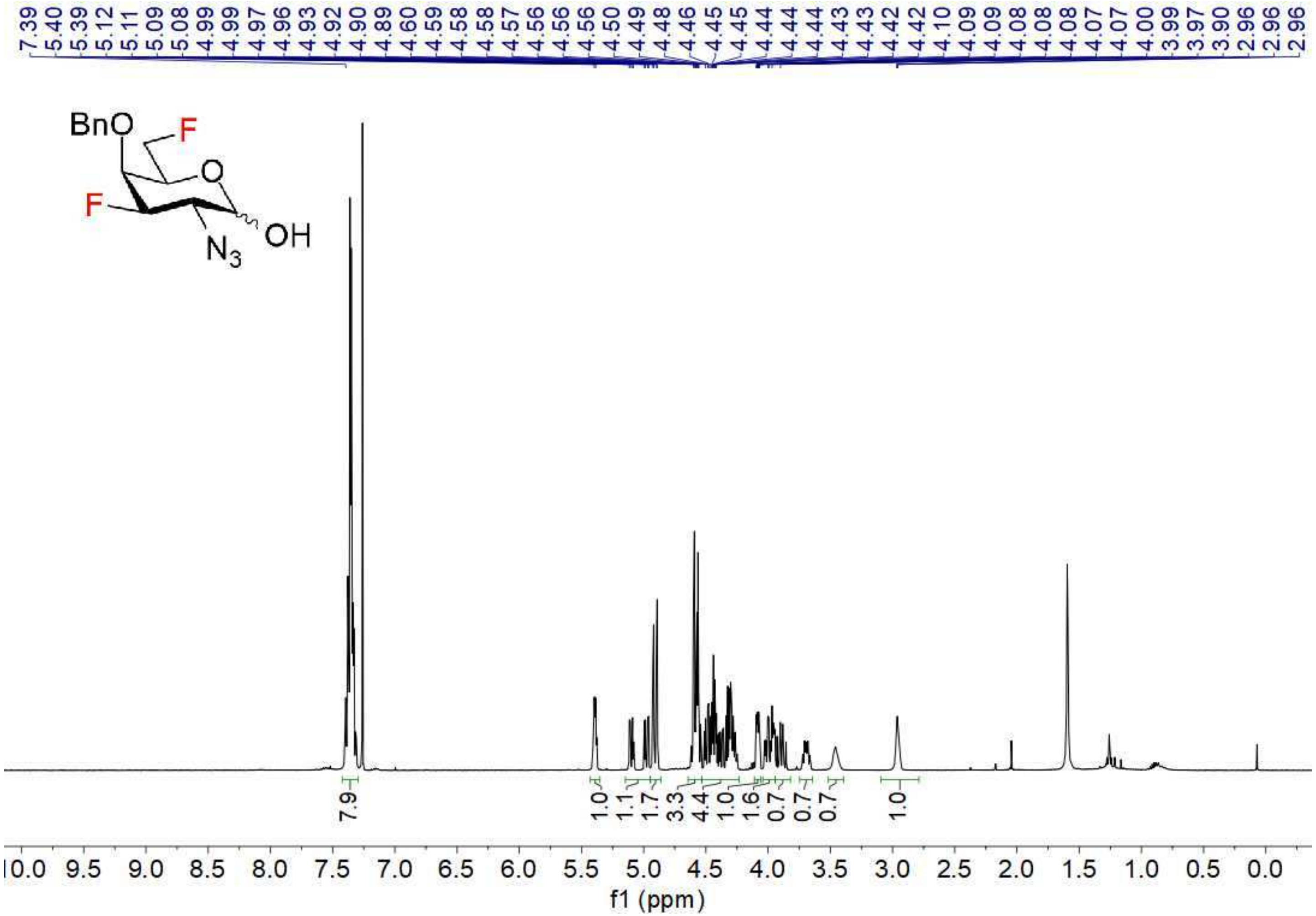
5.2
5.4

^{1}H-^{13}C HSQC 38

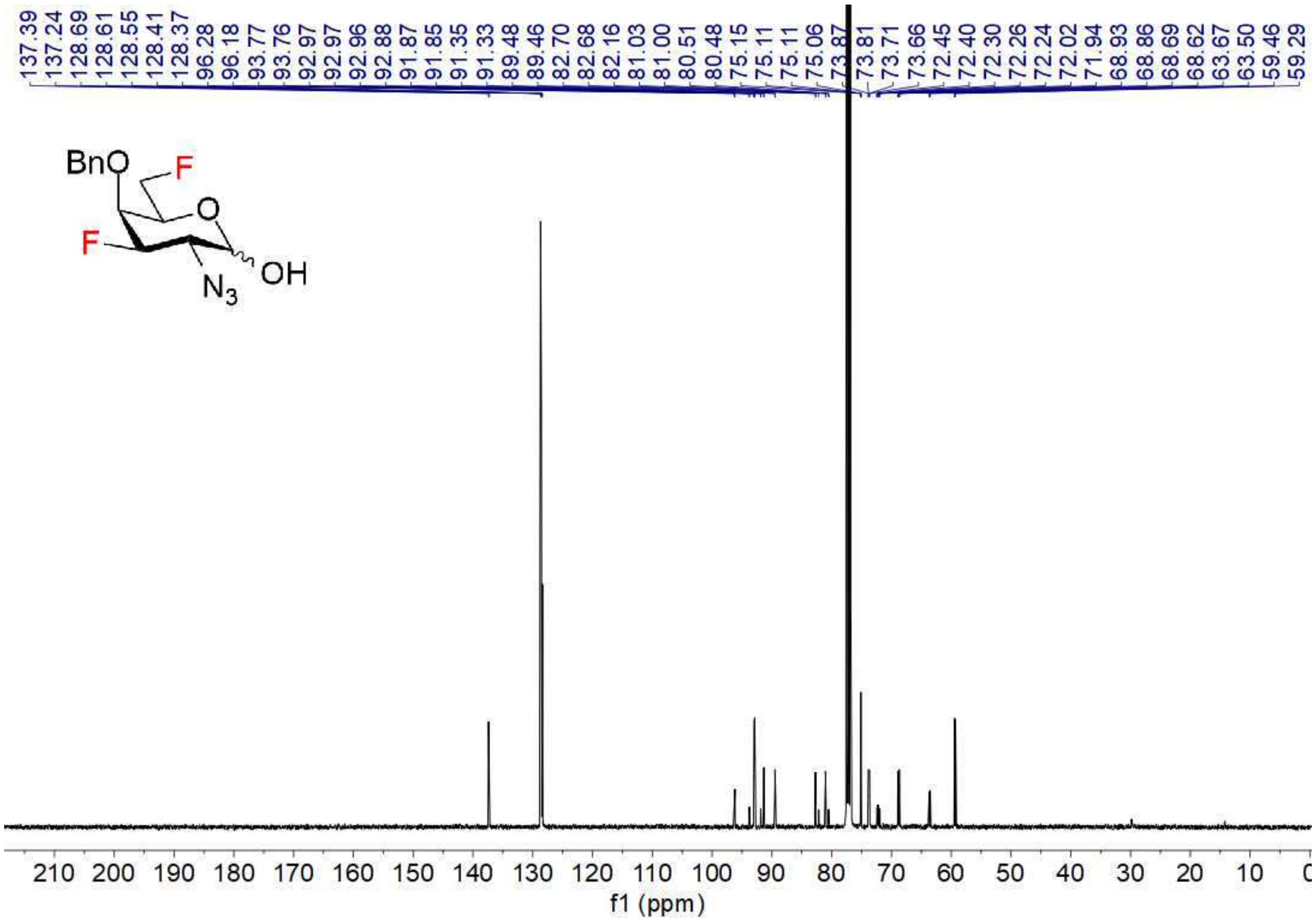


NMR COMPOUND 39

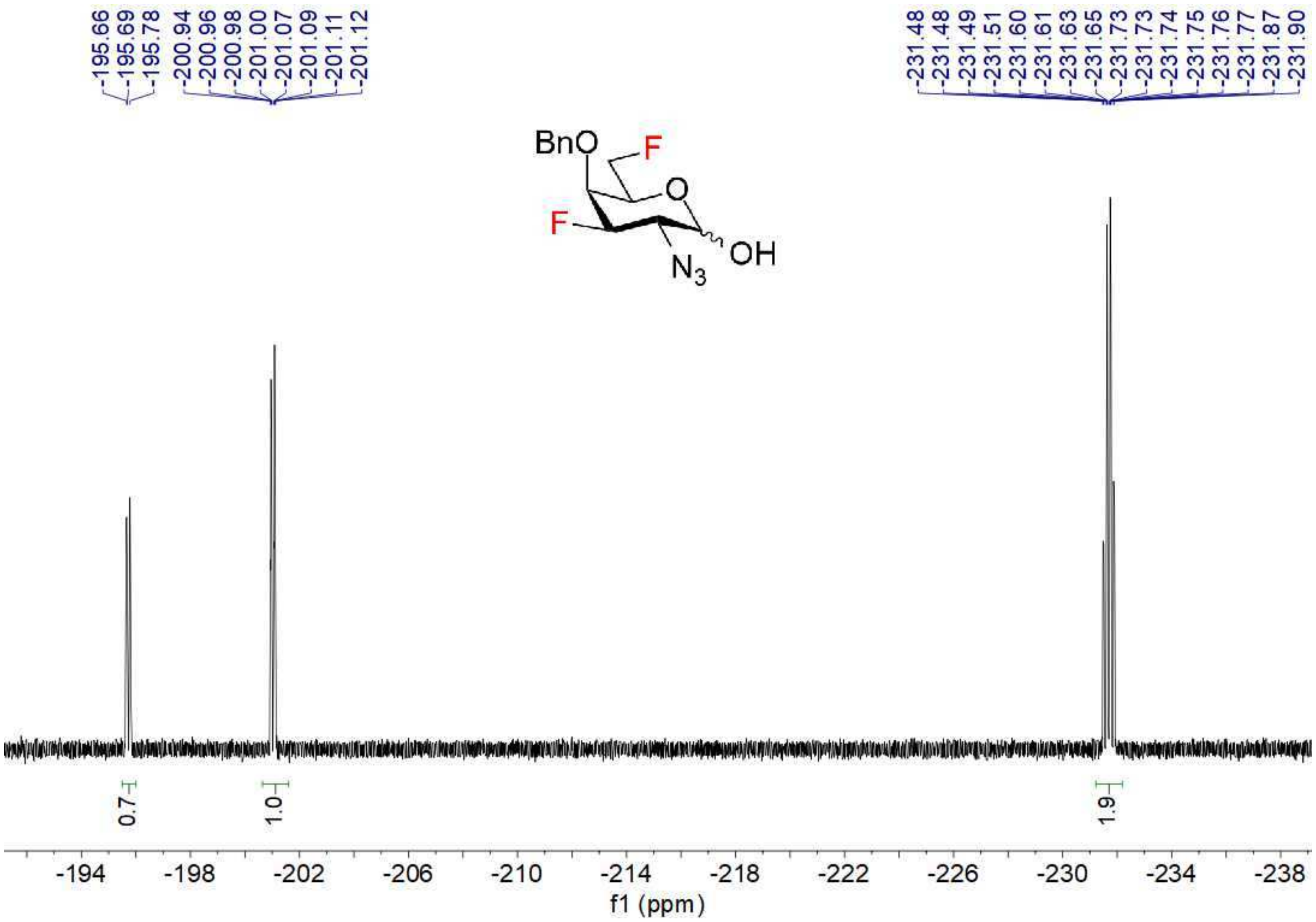
^{1}H NMR (400 MHz, $CDCl_3$) 39 (ca. 40% β-anomer)



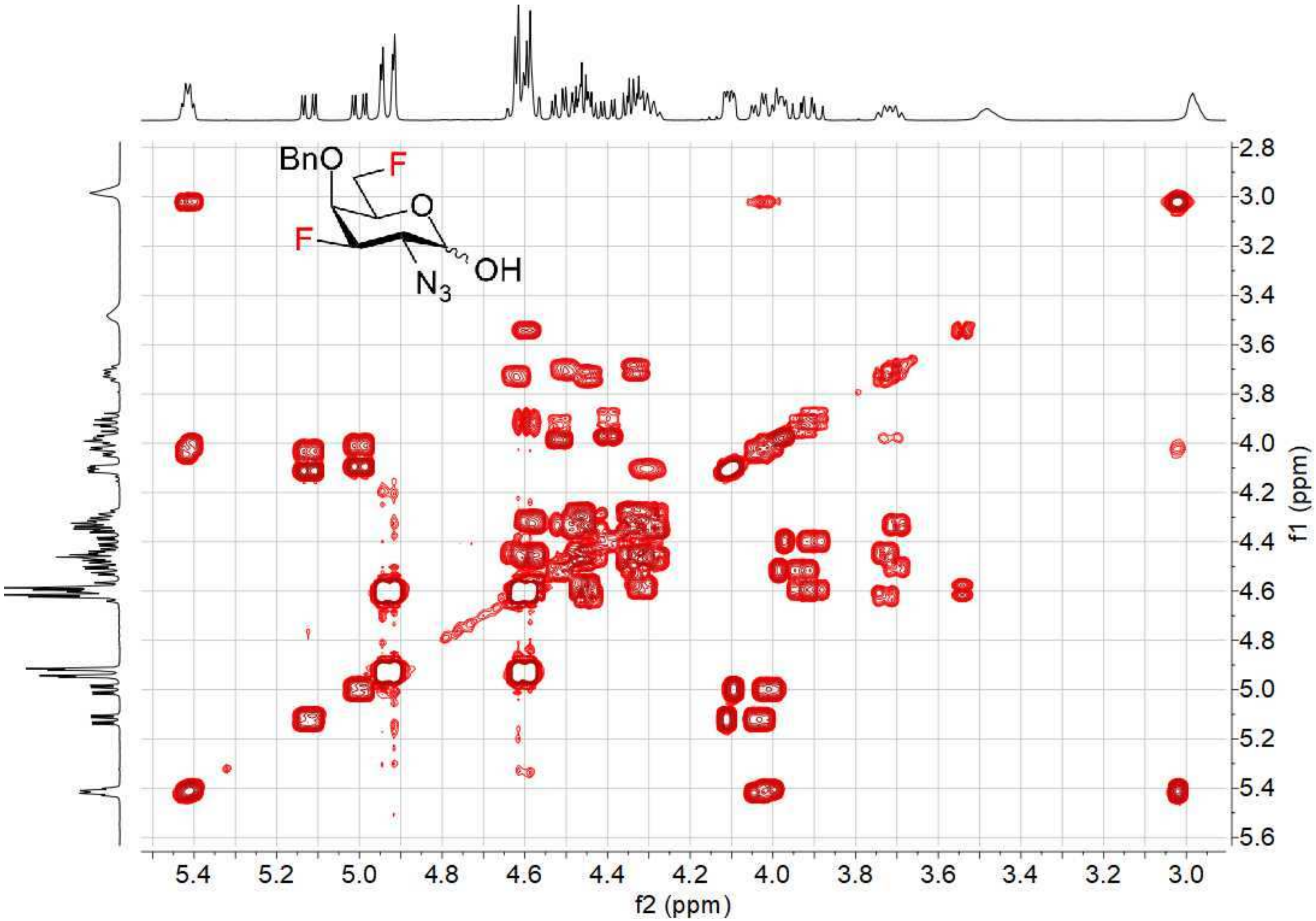
^{13}C NMR (100 MHz, $CDCl_3$) 39



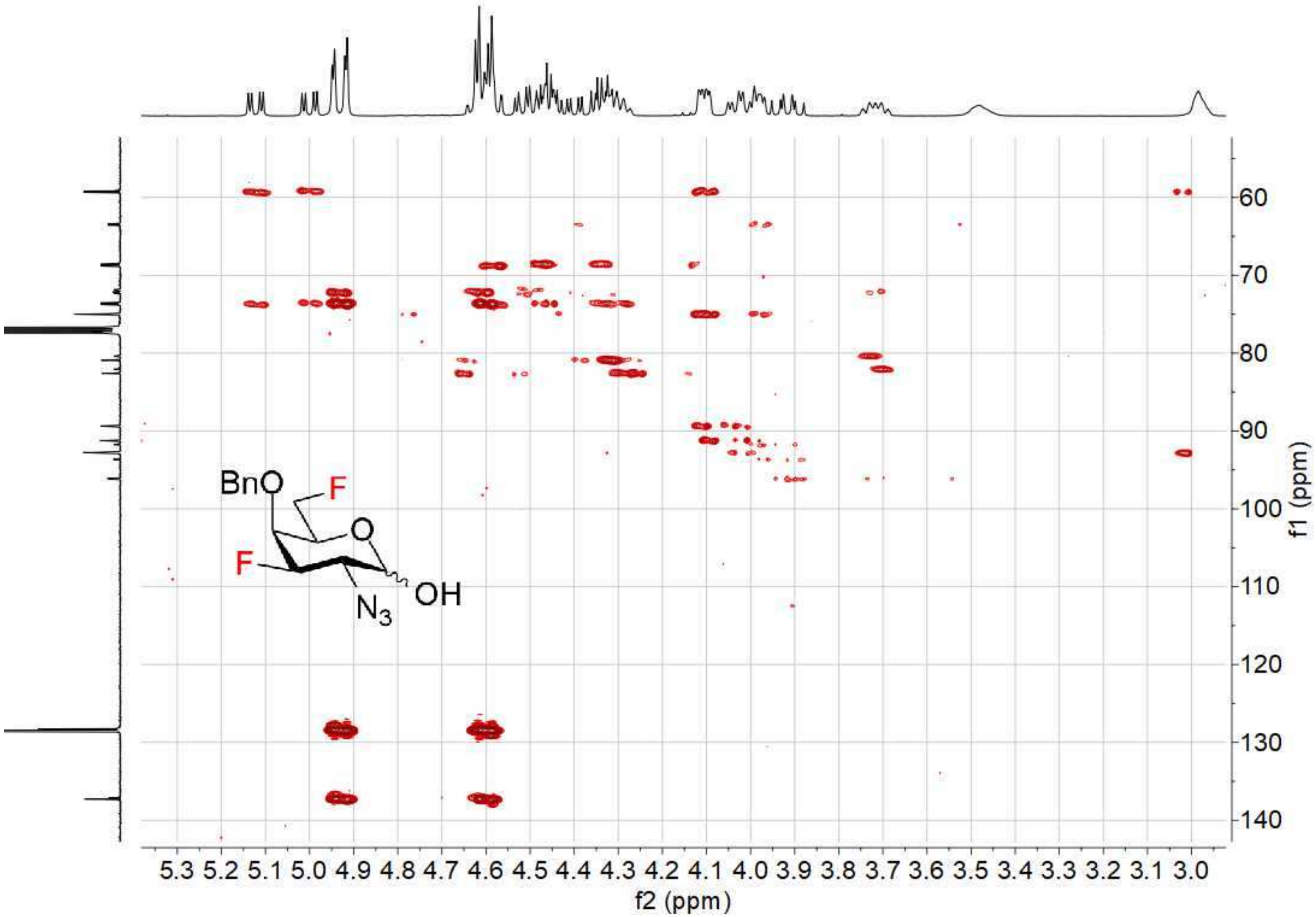
^{19}F NMR (376 MHz, $CDCl_3$) 39 (ca. 40% β-anomer)



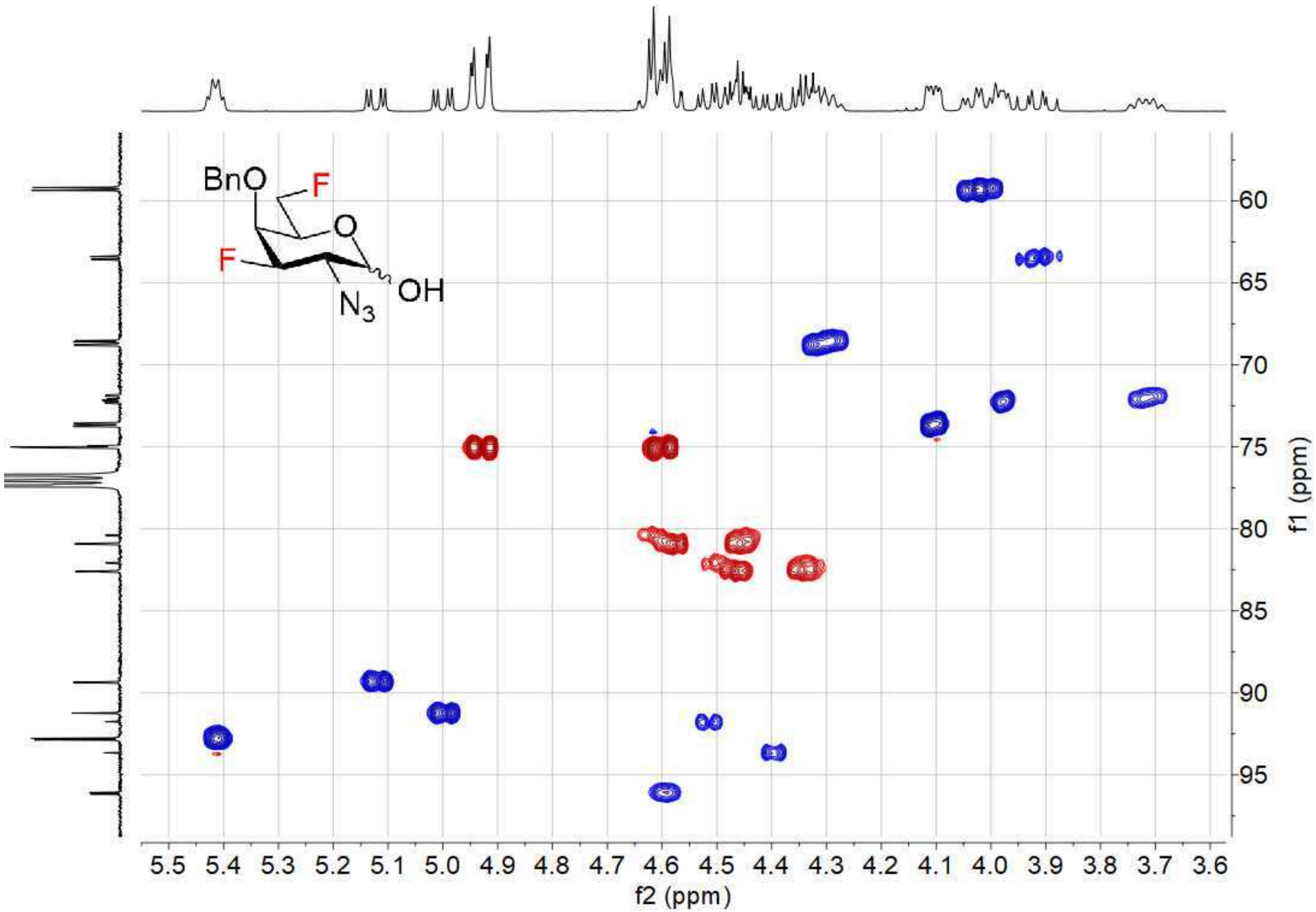
[1]H-[1]H COSY 39



^1H-^{13}C HMBC 39

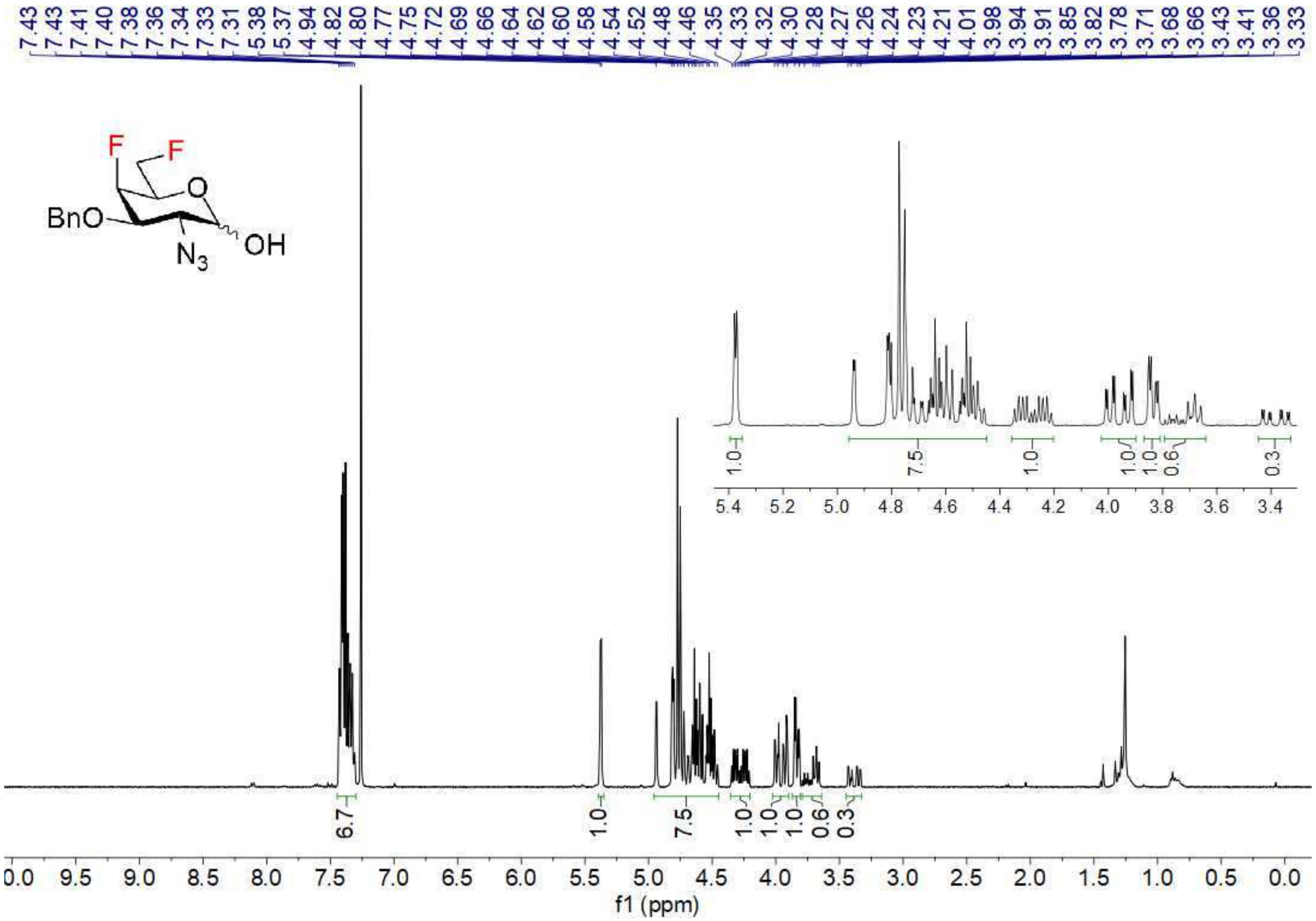


^{1}H-^{13}C HSQC 39

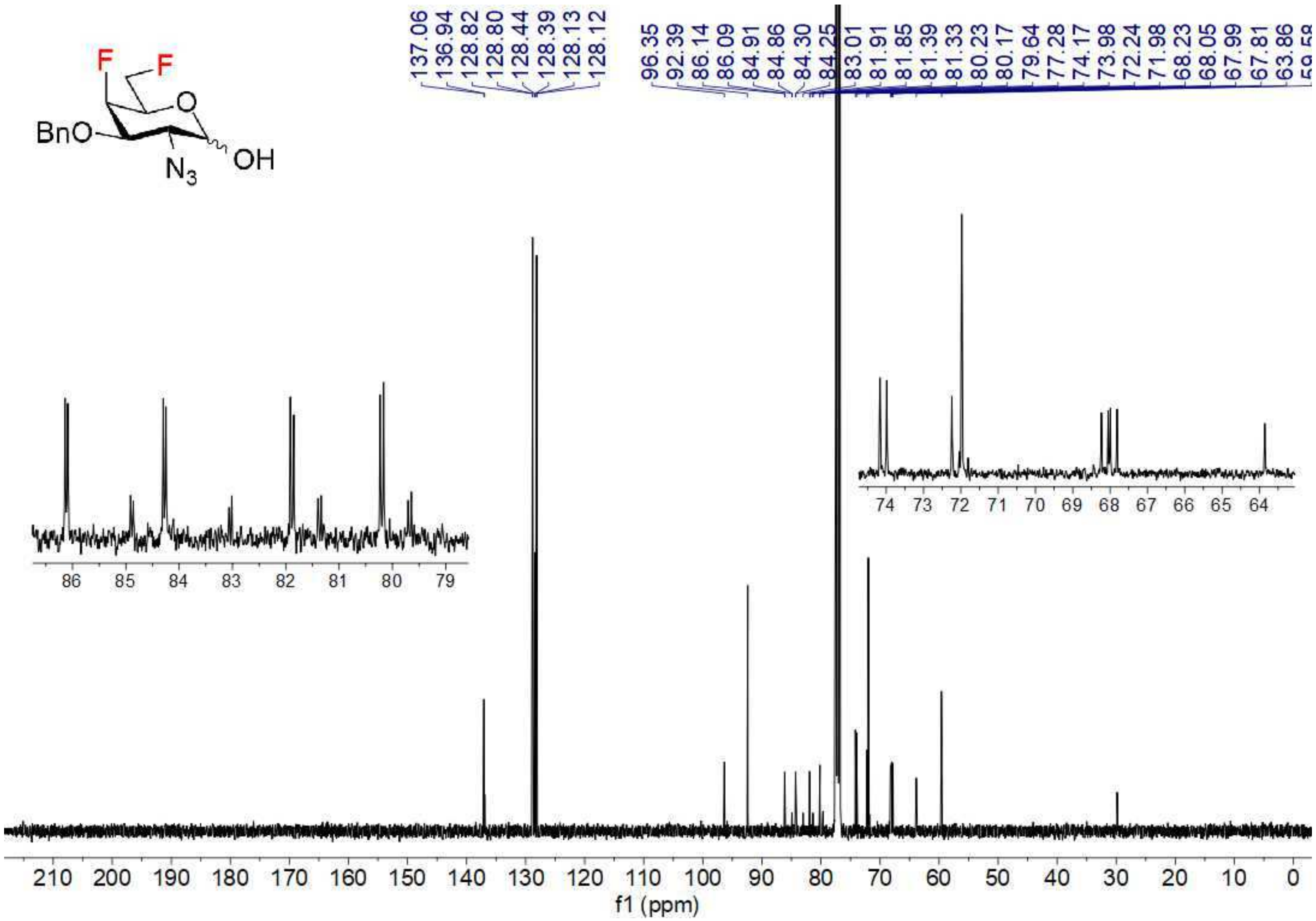


NMR COMPOUND 40

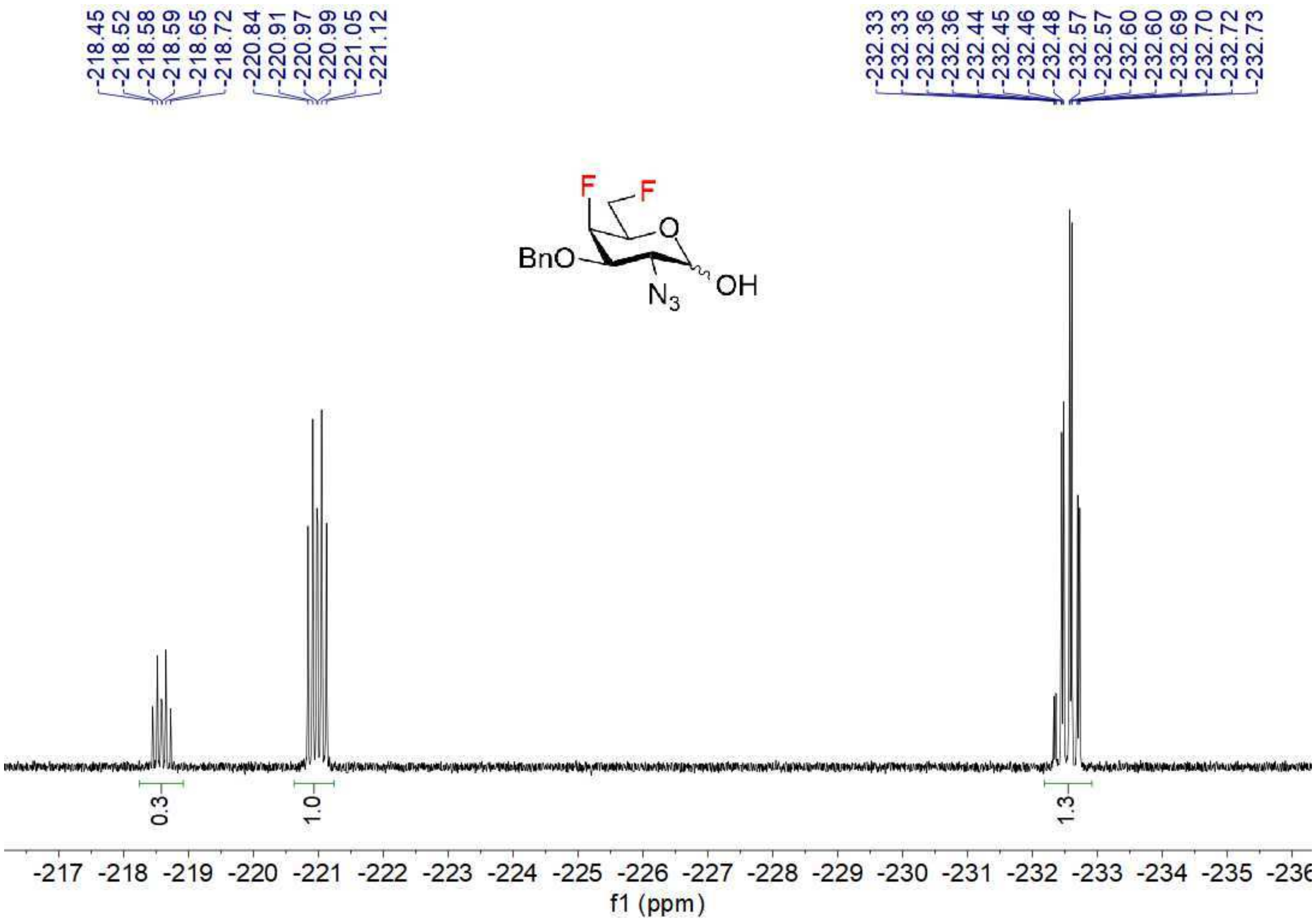
^{1}H NMR (400 MHz, CDCl$_3$) 40 (α/β ca. 10/3)



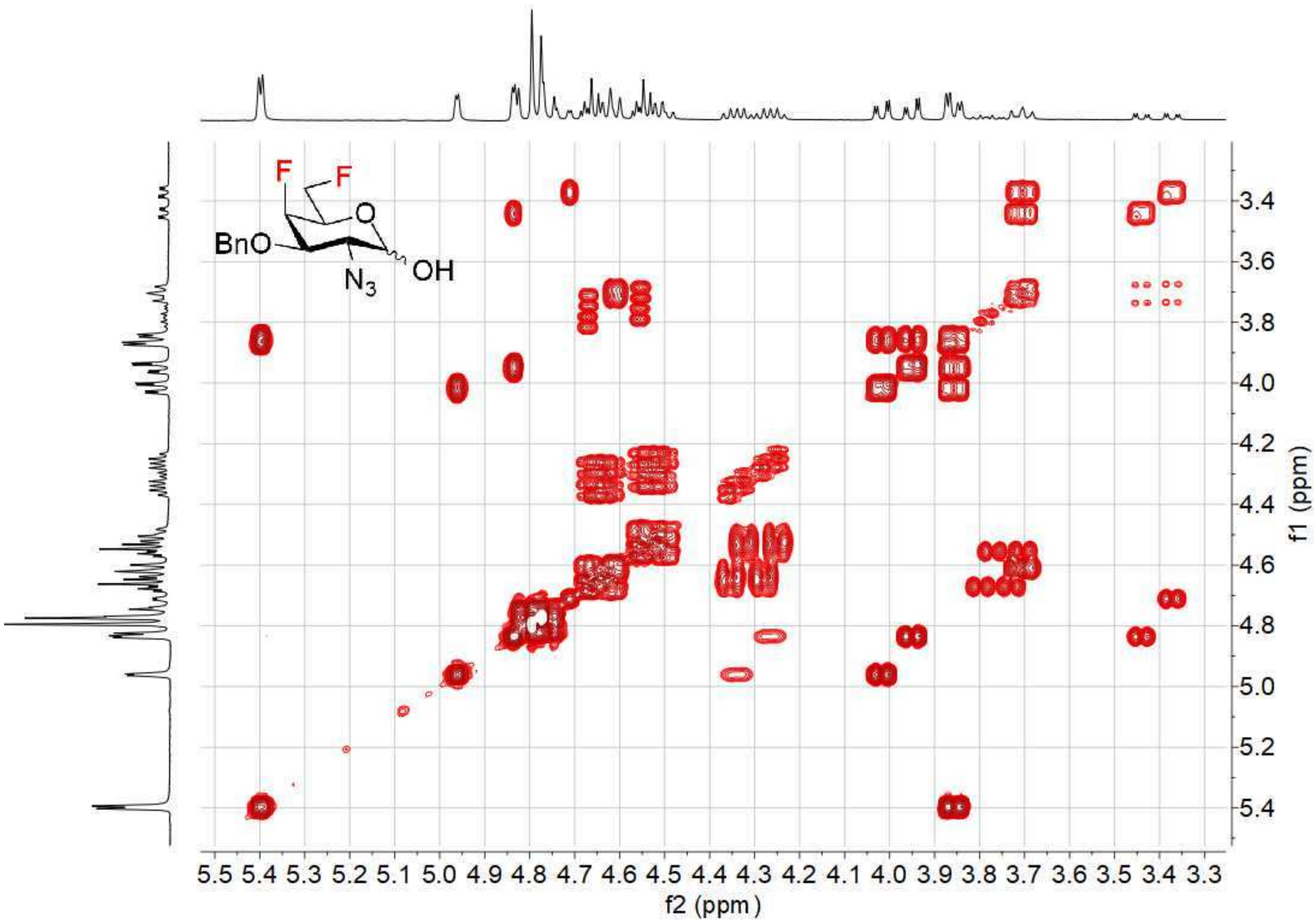
[13]C NMR (100 MHz, $CDCl_3$) 40



[19]F NMR (376 MHz, CDCl$_3$) 40 (α/β ca. 10/3)

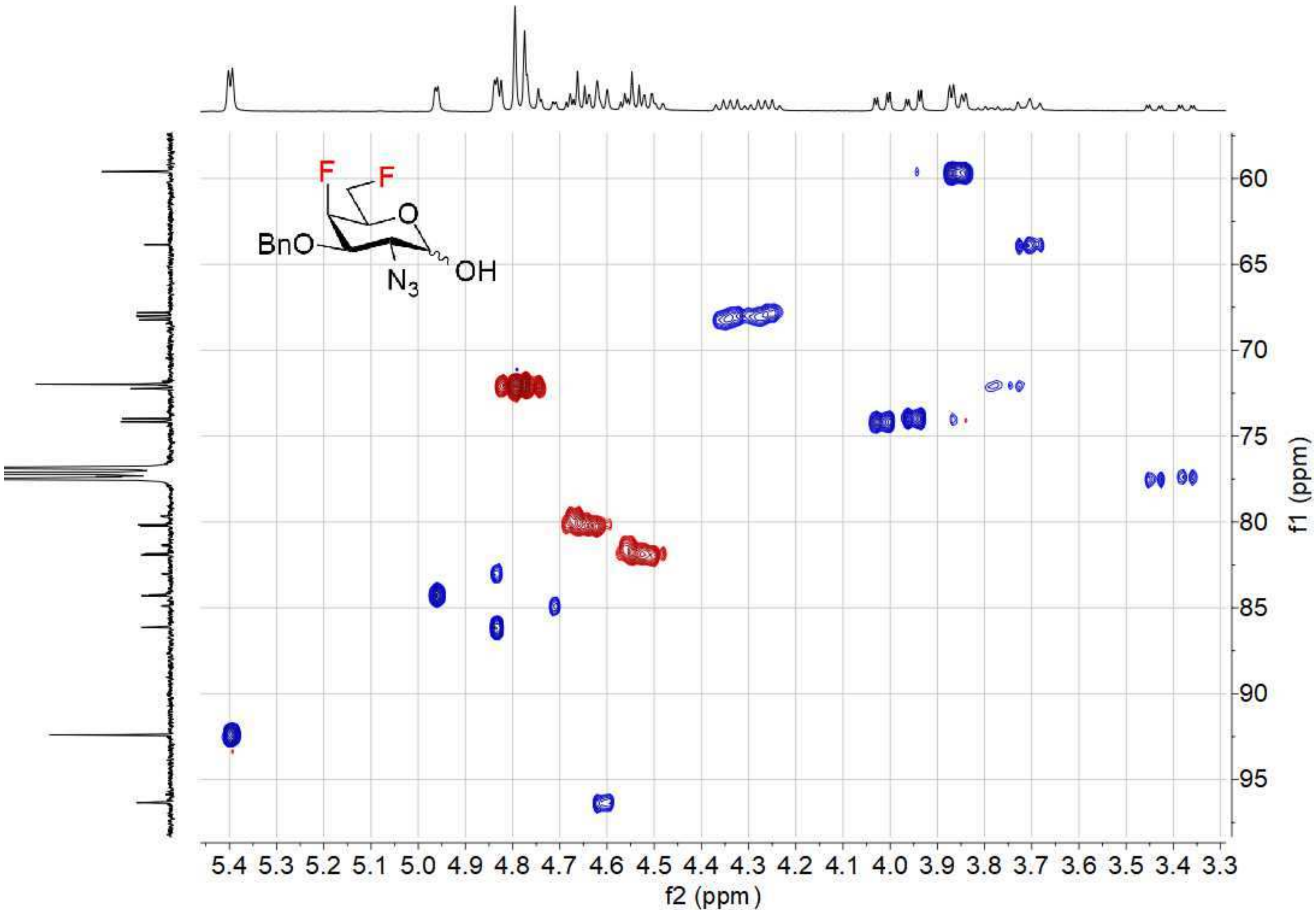


^{1}H-^{1}H COSY 40



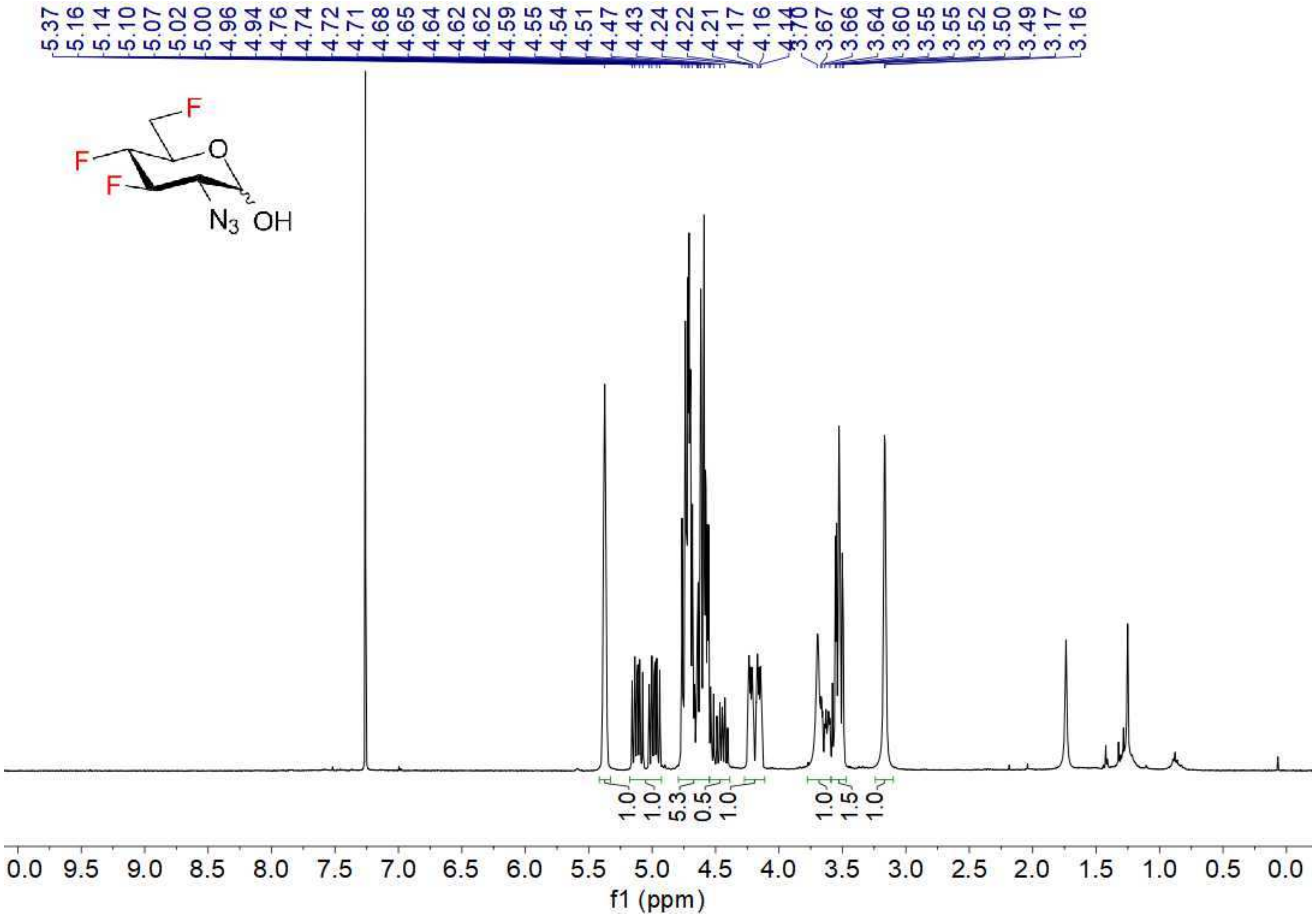
F
F
BnO
O
N3
OH
f1 (ppm)
f2 (ppm)
3.4
3.6
3.8
4.0
4.2
4.4
4.6
4.8
5.0
5.2
5.4
5.5 5.4 5.3 5.2 5.1 5.0 4.9 4.8 4.7 4.6 4.5 4.4 4.3 4.2 4.1 4.0 3.9 3.8 3.7 3.6 3.5 3.4 3.3

^{1}H-^{13}C HSQC 40

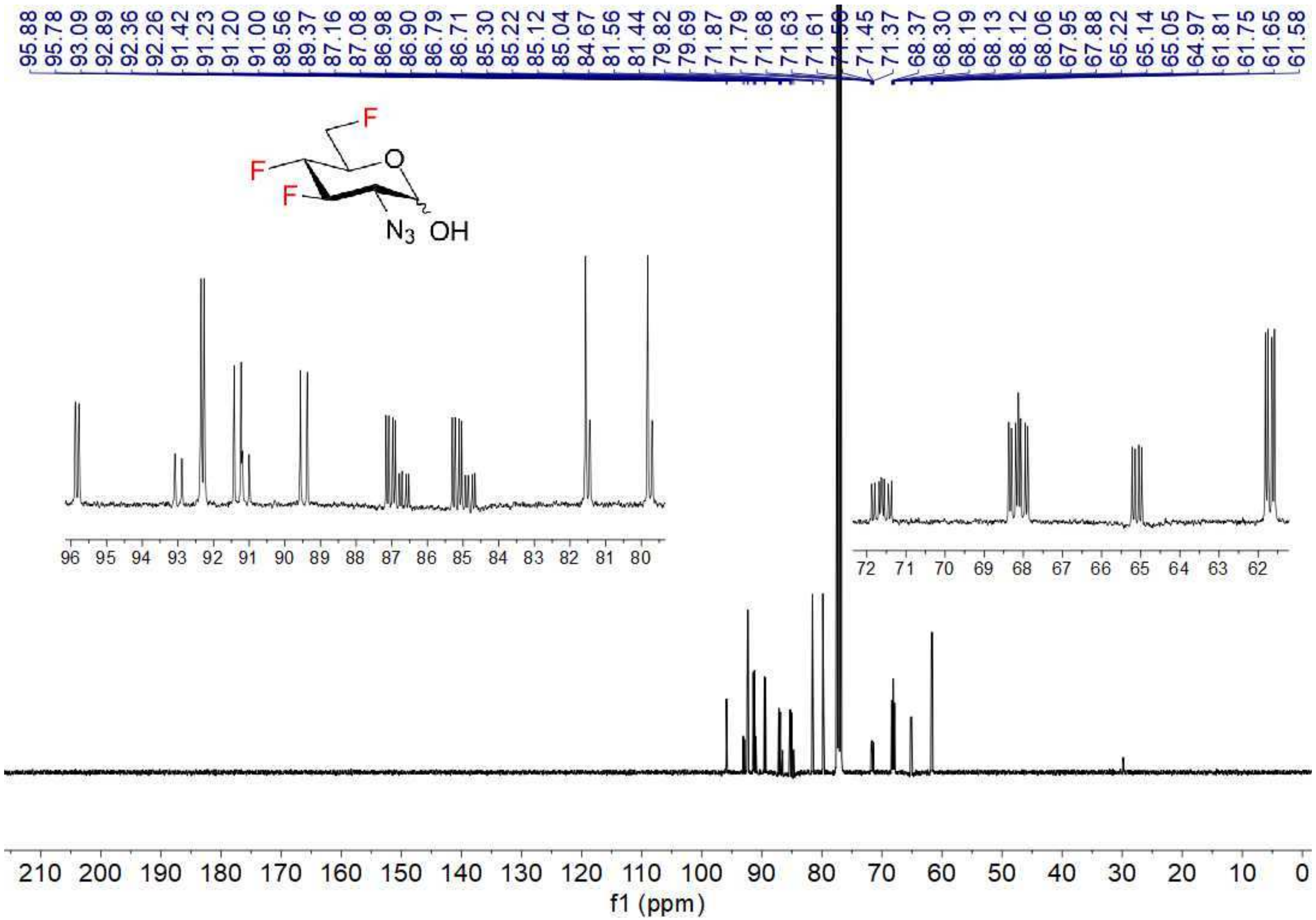


NMR COMPOUND 41

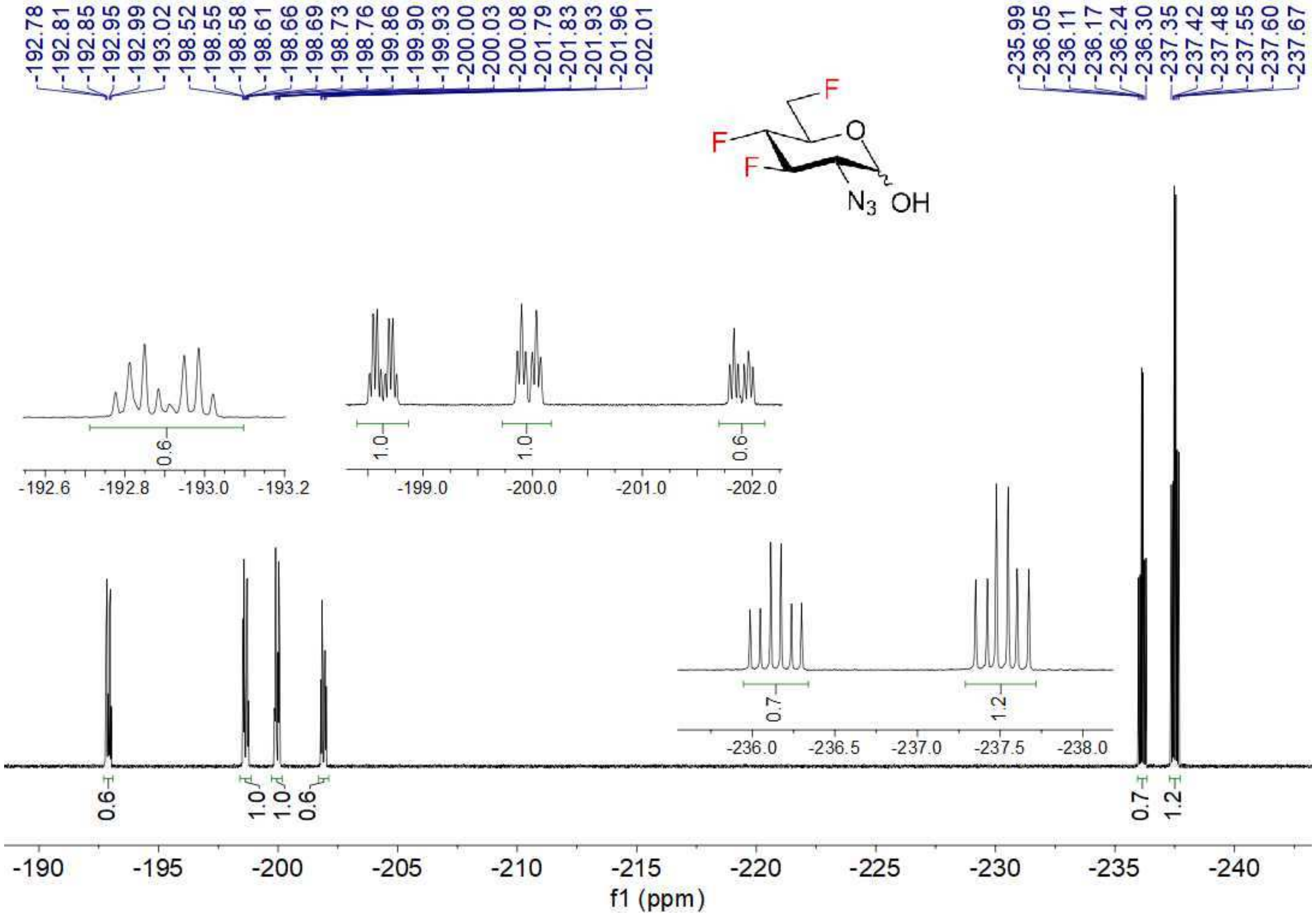
^{1}H NMR (400 MHz, $CDCl_3$) 41 (α/β ca. 2/1)



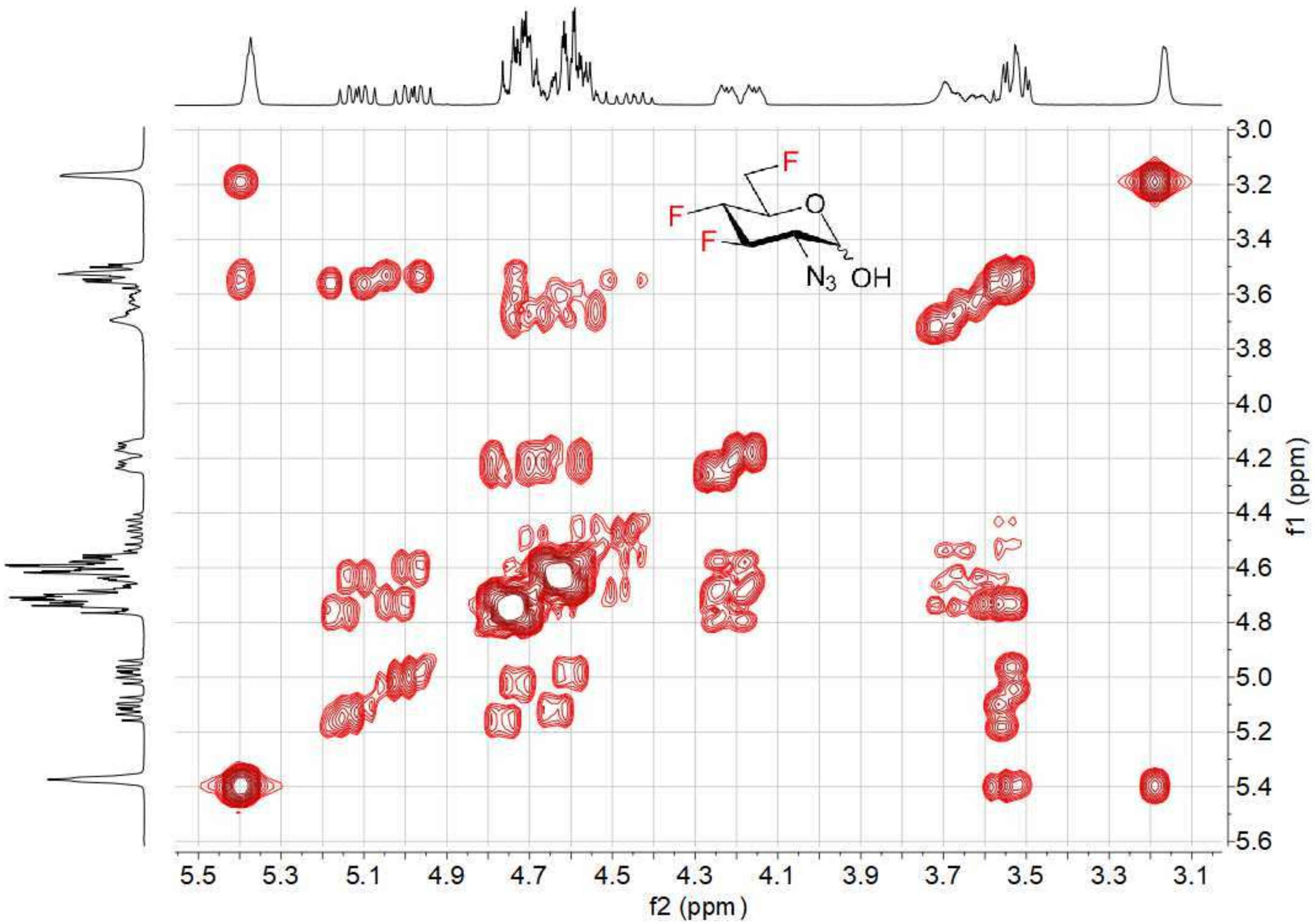
^{13}C NMR (100 MHz, CDCl$_3$) 41



^{19}F NMR (376 MHz, CDCl$_3$) 41 (α/β ca. 2/1)

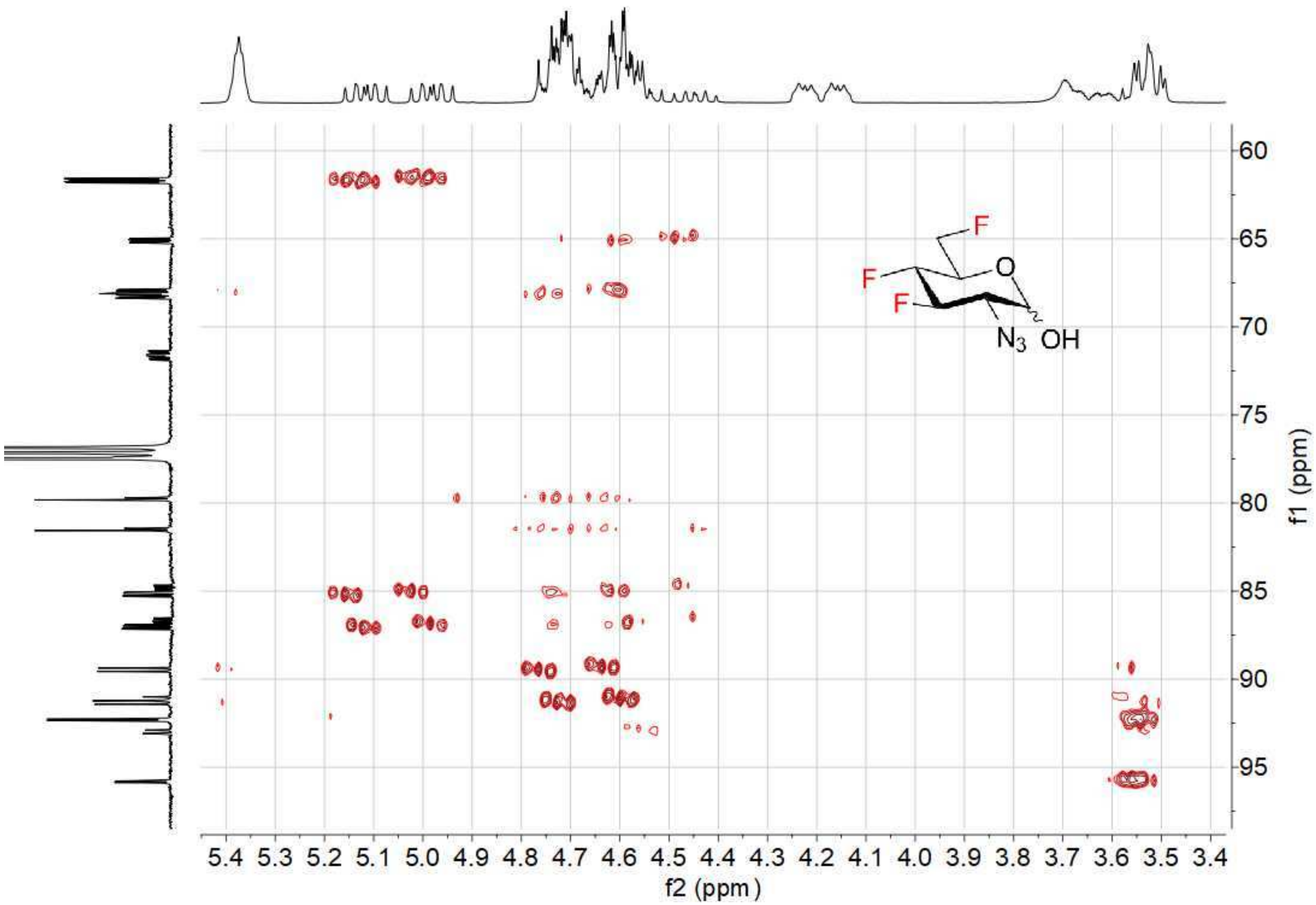


[1]H-[1]H COSY 41

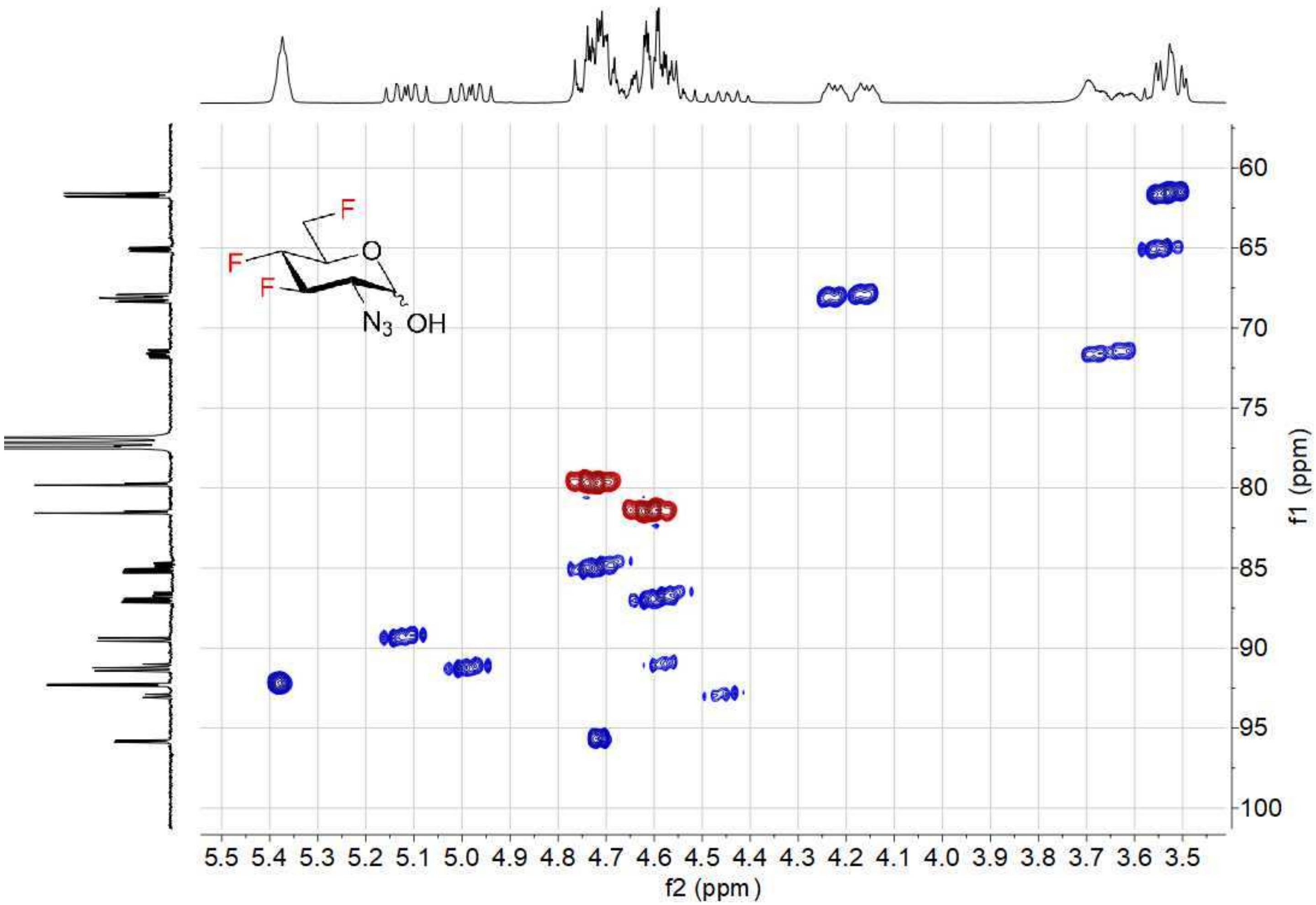


F
O
F
F
N3
OH
3.0
3.2
3.4
3.6
3.8
4.0
4.2
4.4
4.6
4.8
5.0
5.2
5.4
5.6
f1 (ppm)
5.5
5.3
5.1
4.9
4.7
4.5
4.3
4.1
3.9
3.7
3.5
3.3
3.1
f2 (ppm)

^{1}H-^{13}C HMBC 41

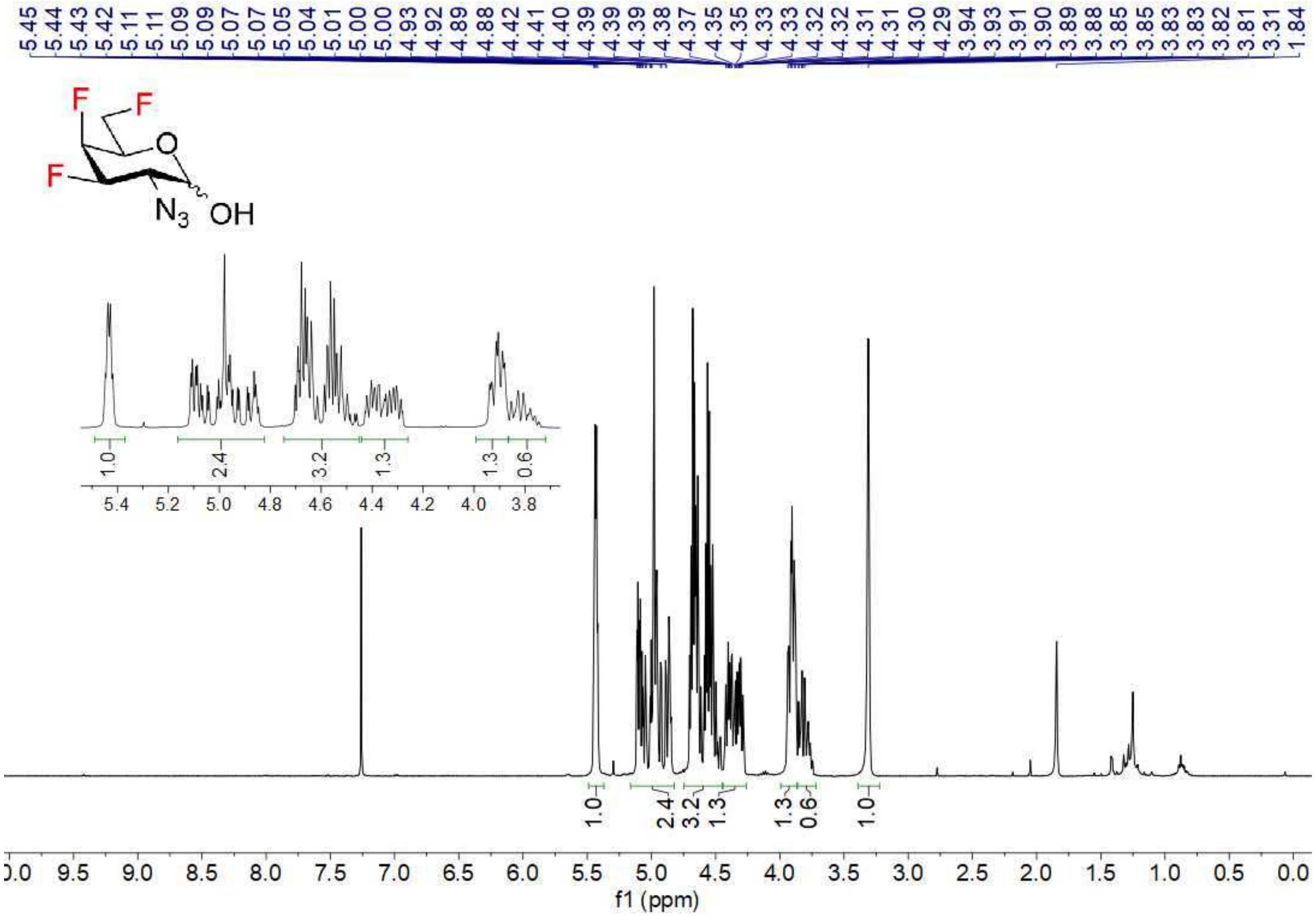


[1]H-[13]C HSQC 41

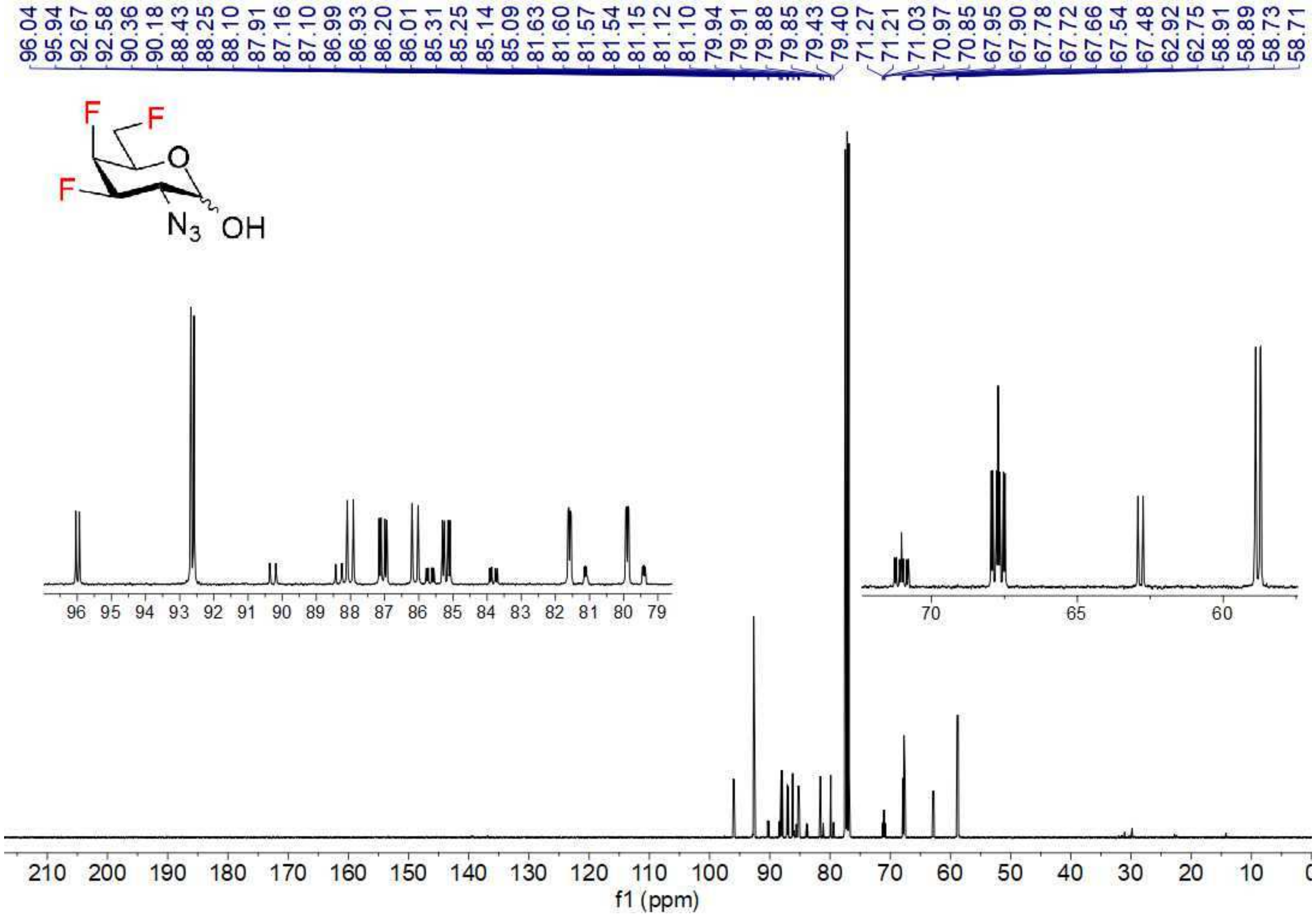


NMR COMPOUND 42

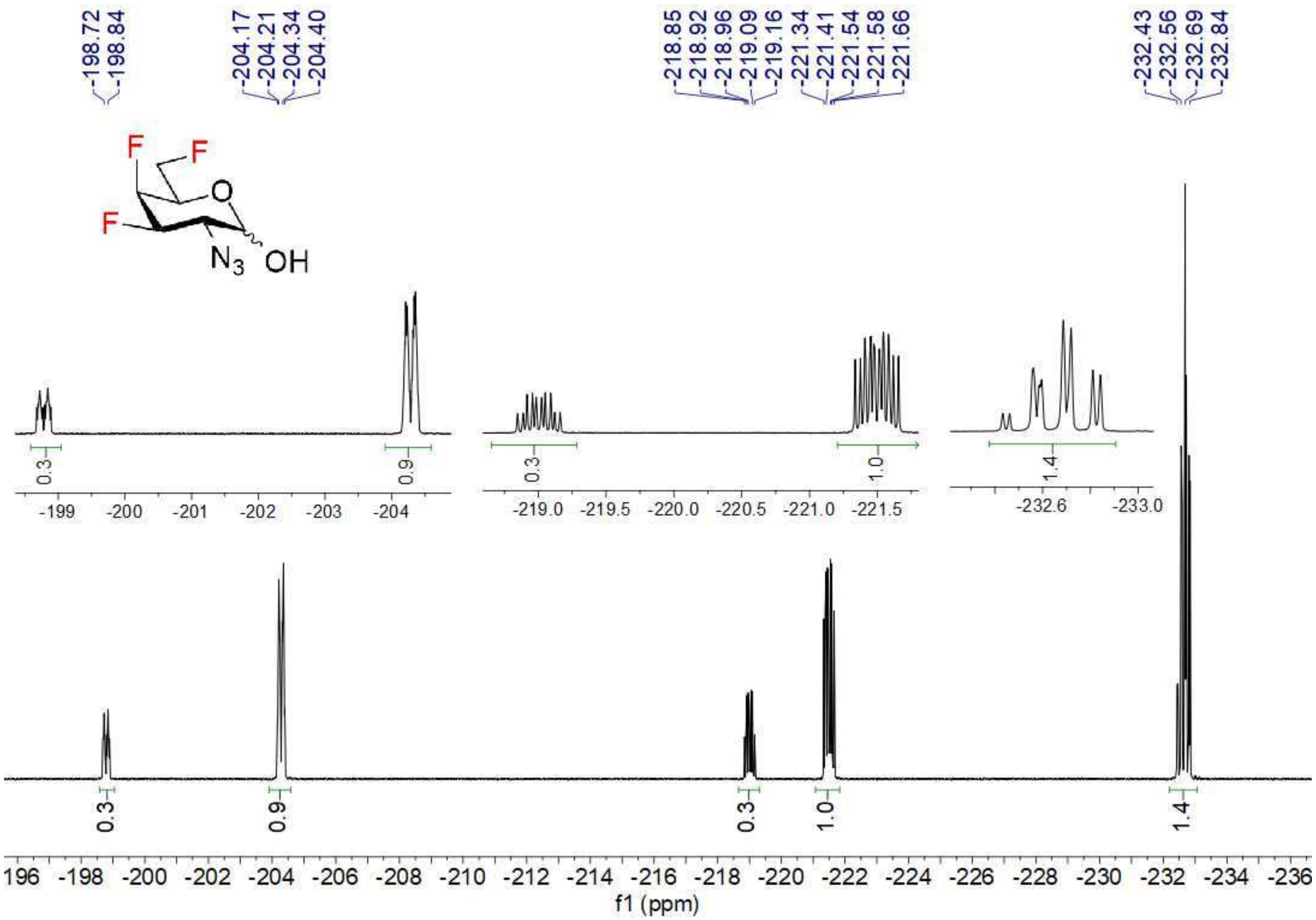
^{1}H NMR (400 MHz, $CDCl_3$) 42 (α/β ca. 10/3)



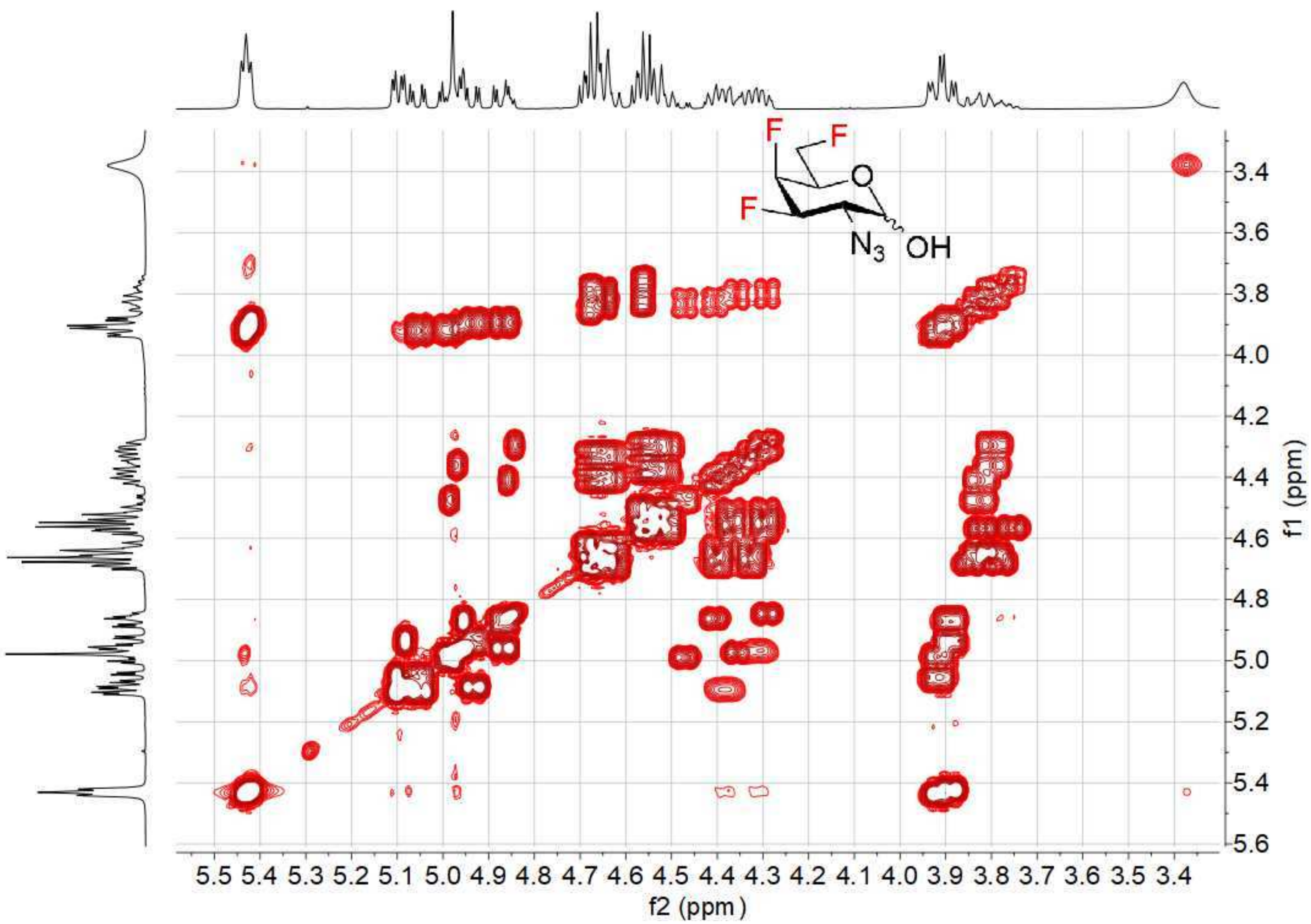
^{13}C NMR (100 MHz, CDCl$_3$) 42



^{19}F NMR (376 MHz, CDCl$_3$) 42 (α/β ca. 10/3)

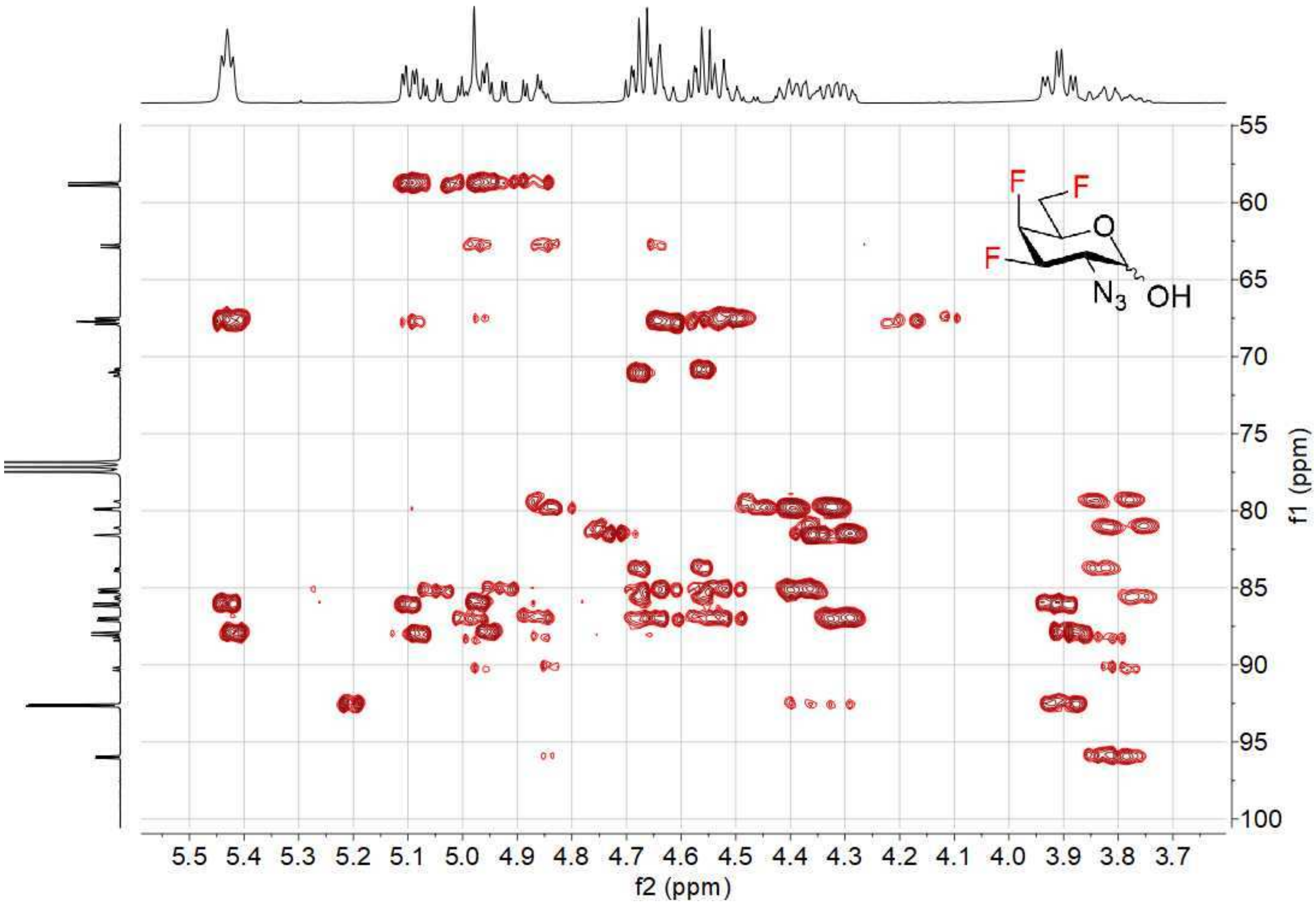


^{1}H-^{1}H COSY 42

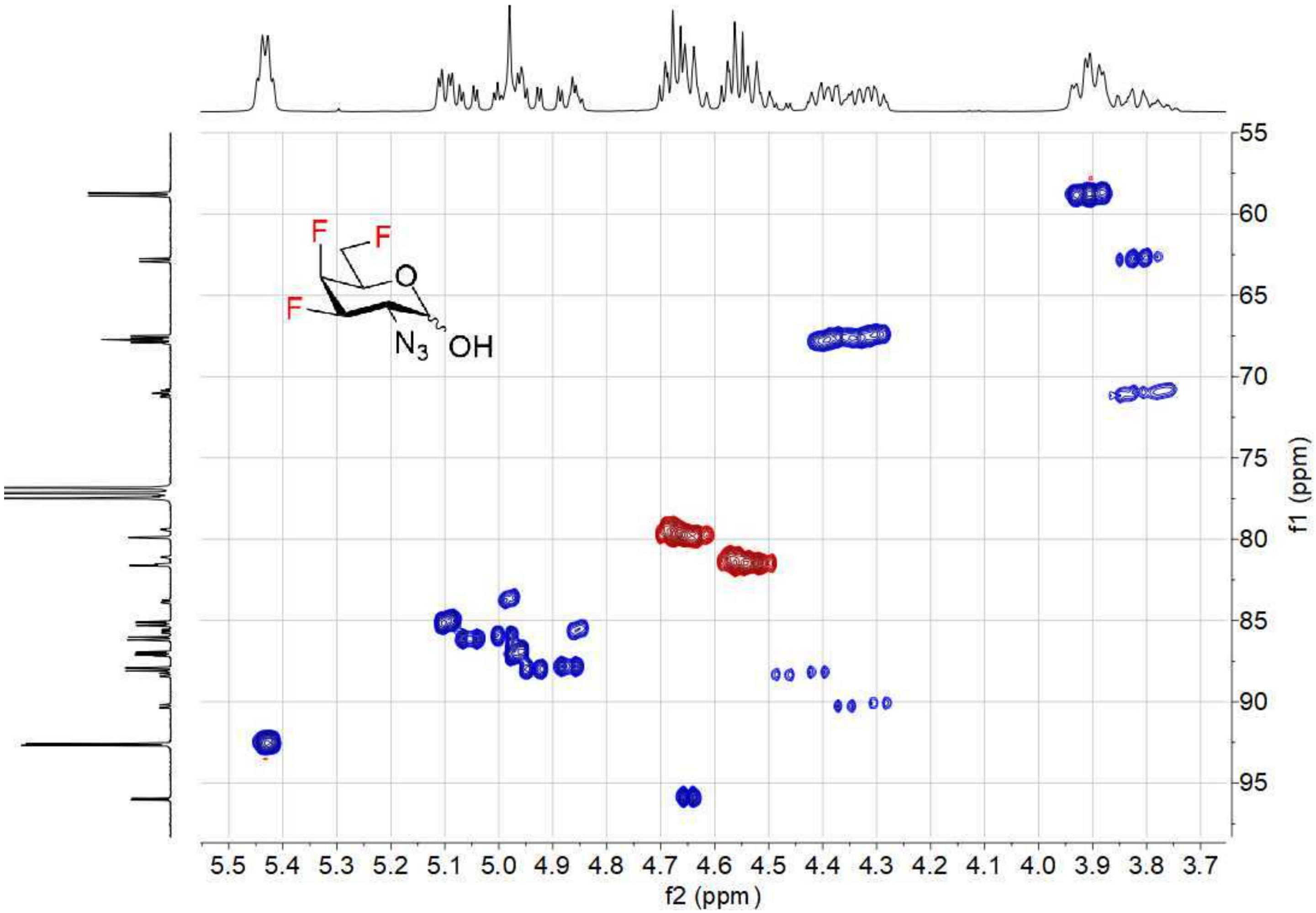


F
F
F
O
N3
OH
5.5 5.4 5.3 5.2 5.1 5.0 4.9 4.8 4.7 4.6 4.5 4.4 4.3 4.2 4.1 4.0 3.9 3.8 3.7 3.6 3.5 3.4
f2 (ppm)
3.4
3.6
3.8
4.0
4.2
4.4
4.6
4.8
5.0
5.2
5.4
5.6
f1 (ppm)

^{1}H-^{13}C HMBC 42

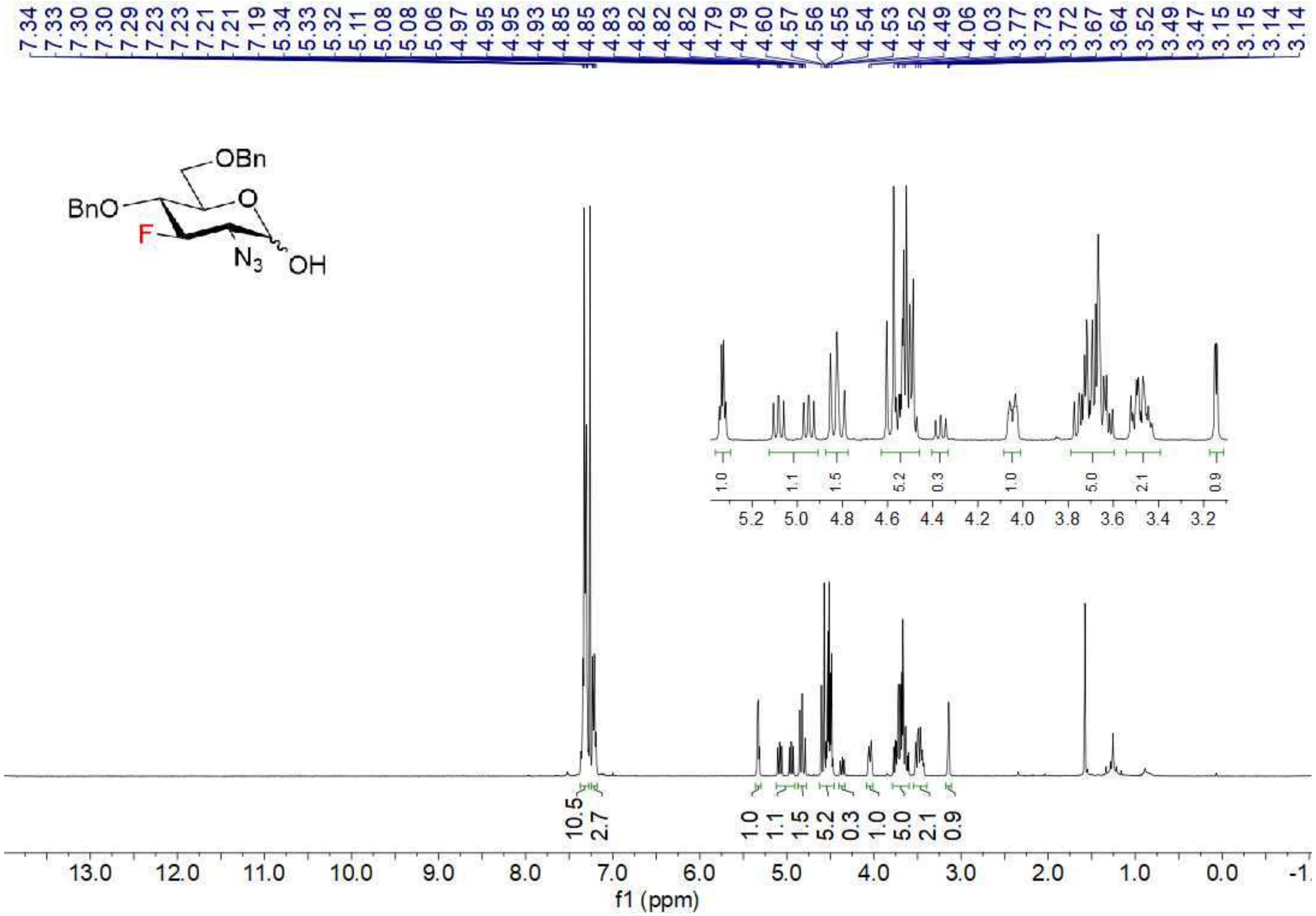


^{1}H-^{13}C HSQC 42

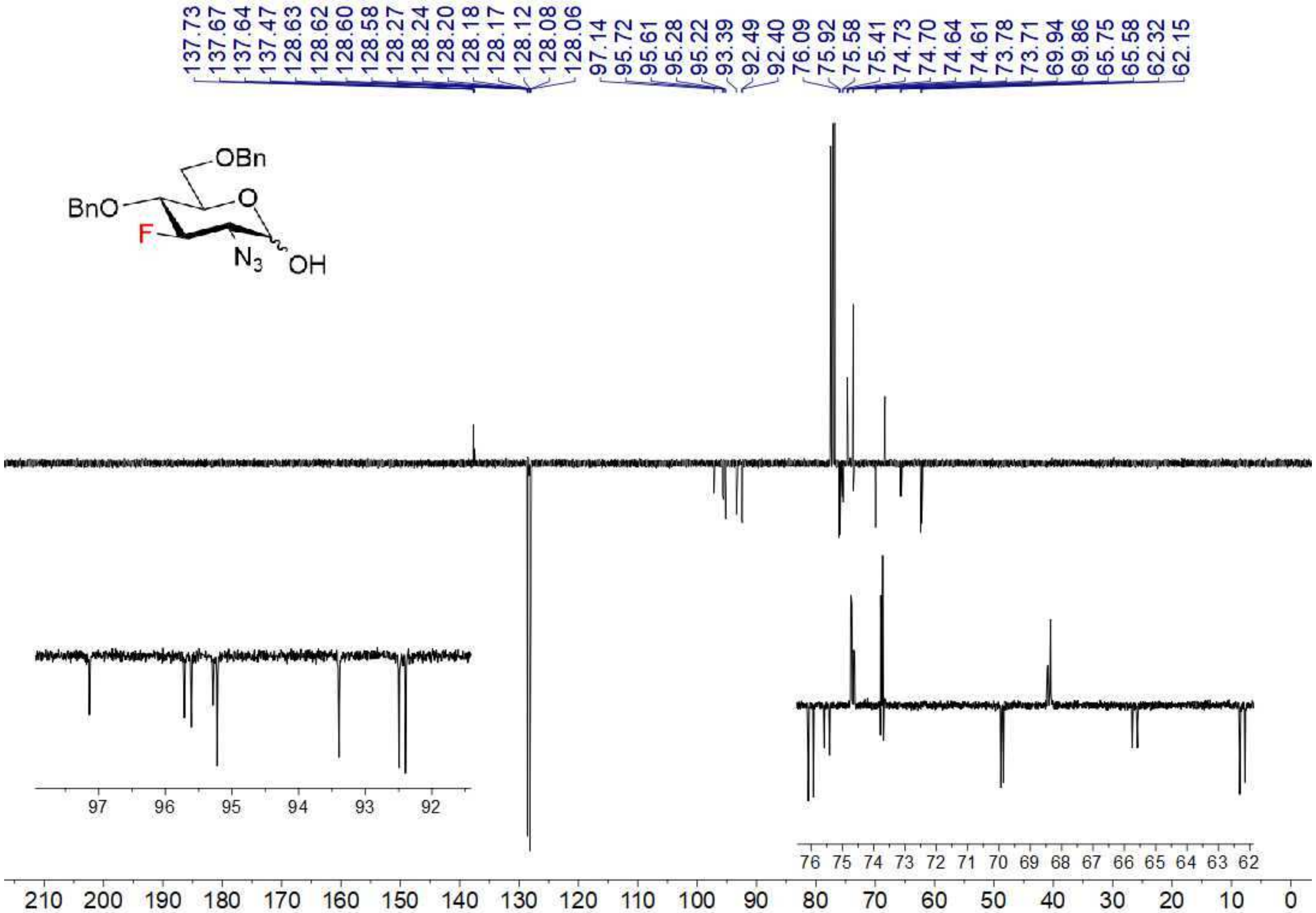


NMR COMPOUND 43

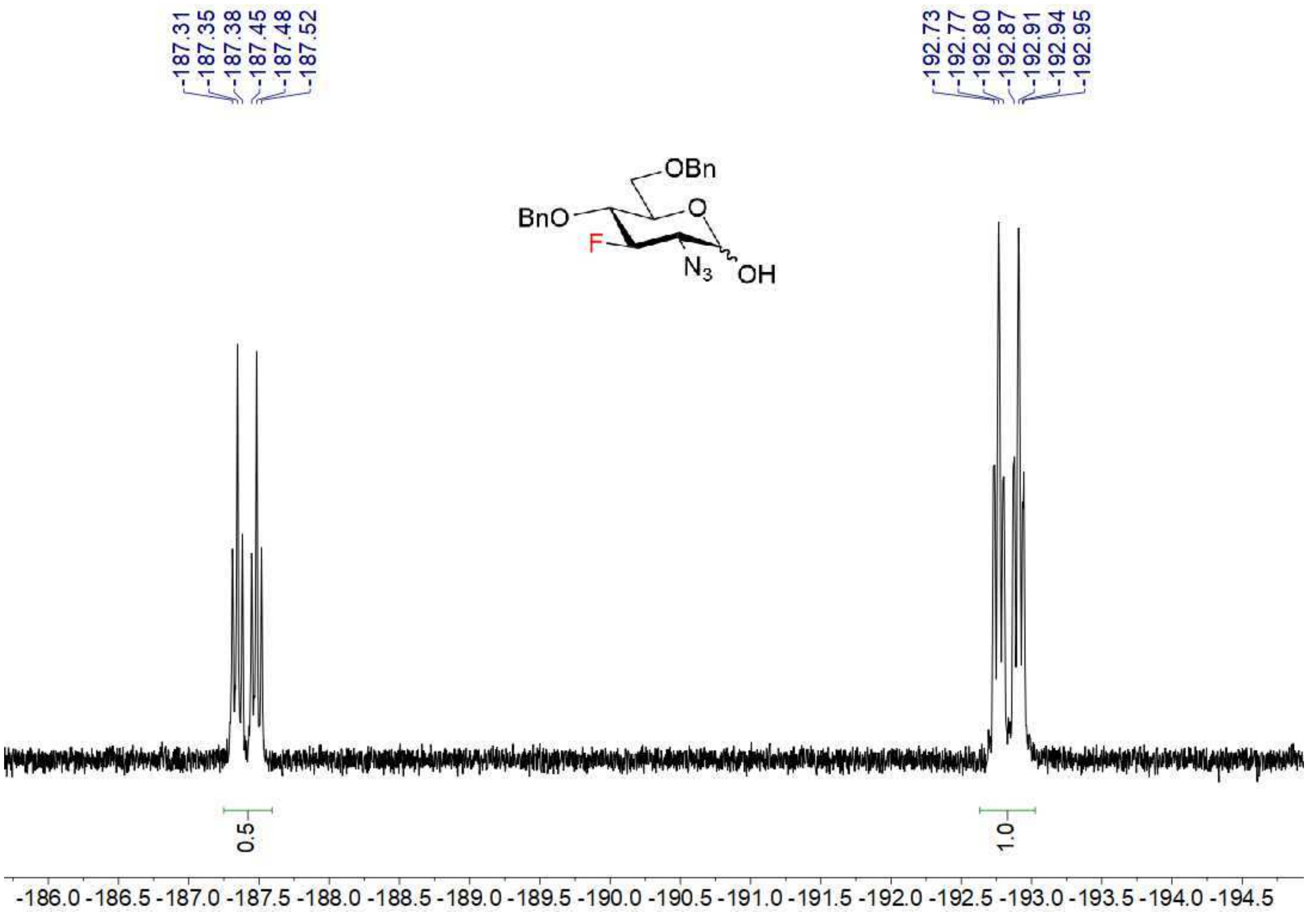
^{1}H NMR (400 MHz, $CDCl_3$) 43 (α/β ca. 2/1)



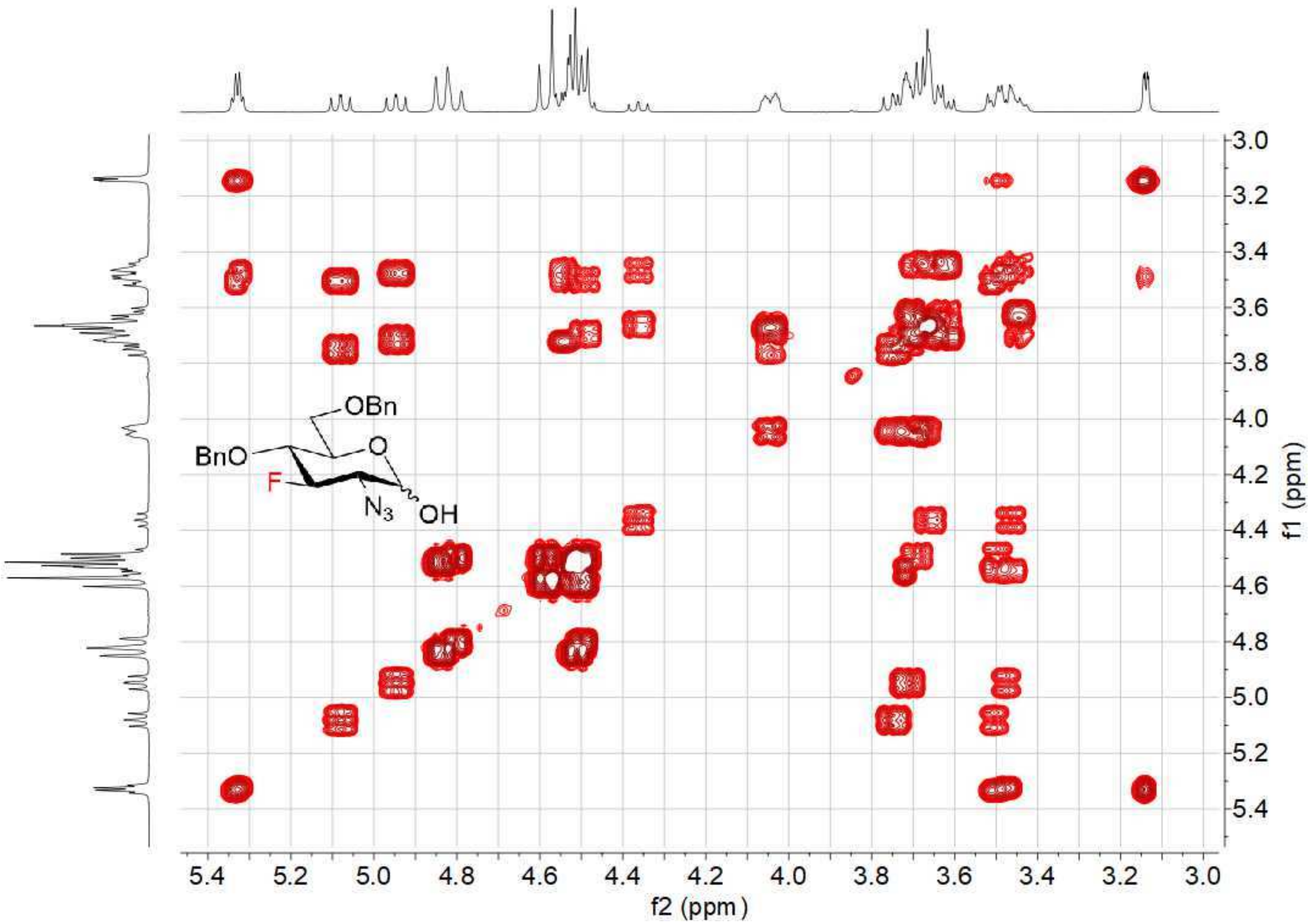
^{13}C APT NMR (100 MHz, CDCl$_3$) 43



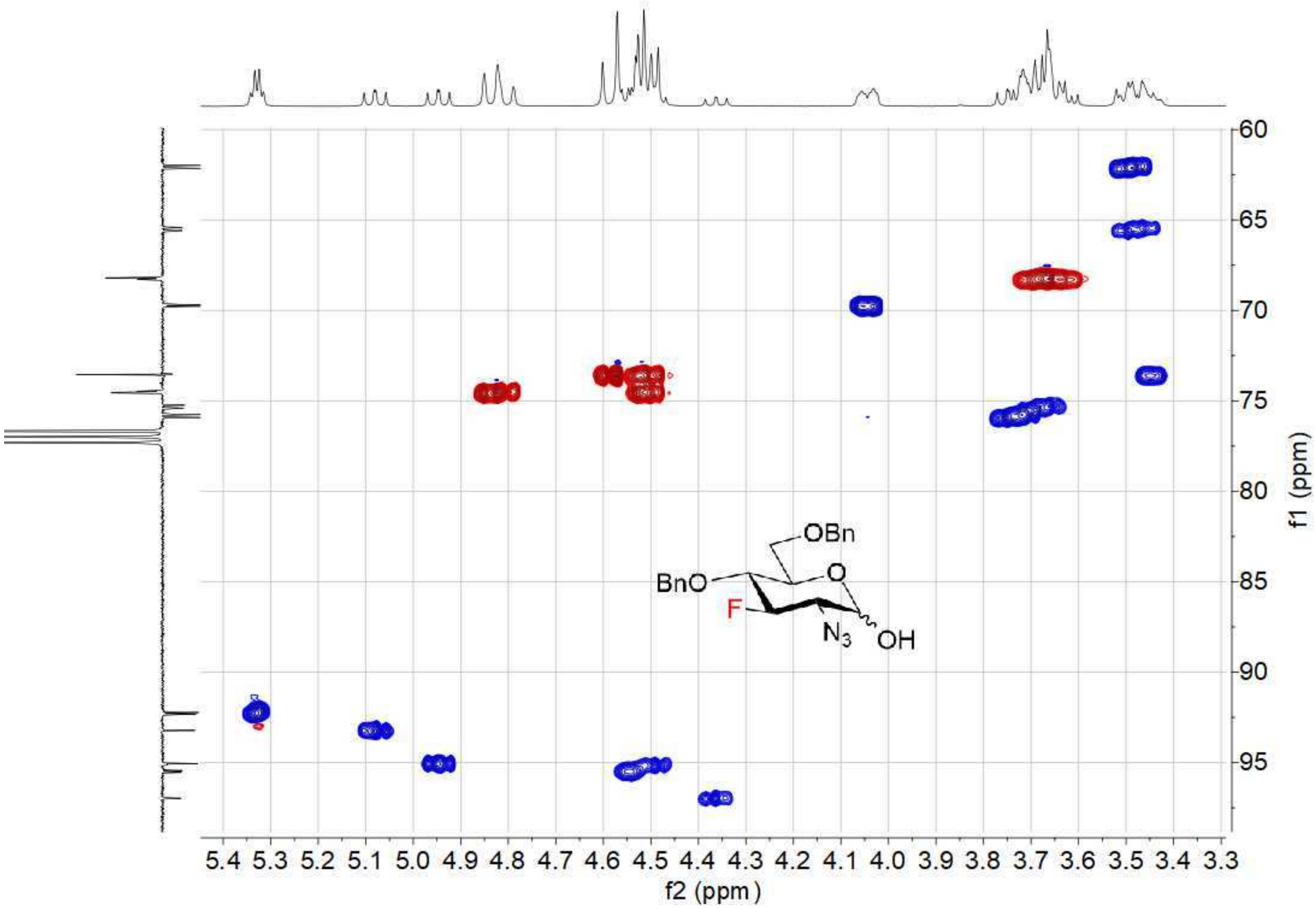
^{19}F NMR (376 MHz, $CDCl_3$) 43 (α/β ca. 2/1)



^1H-^1H COSY 43

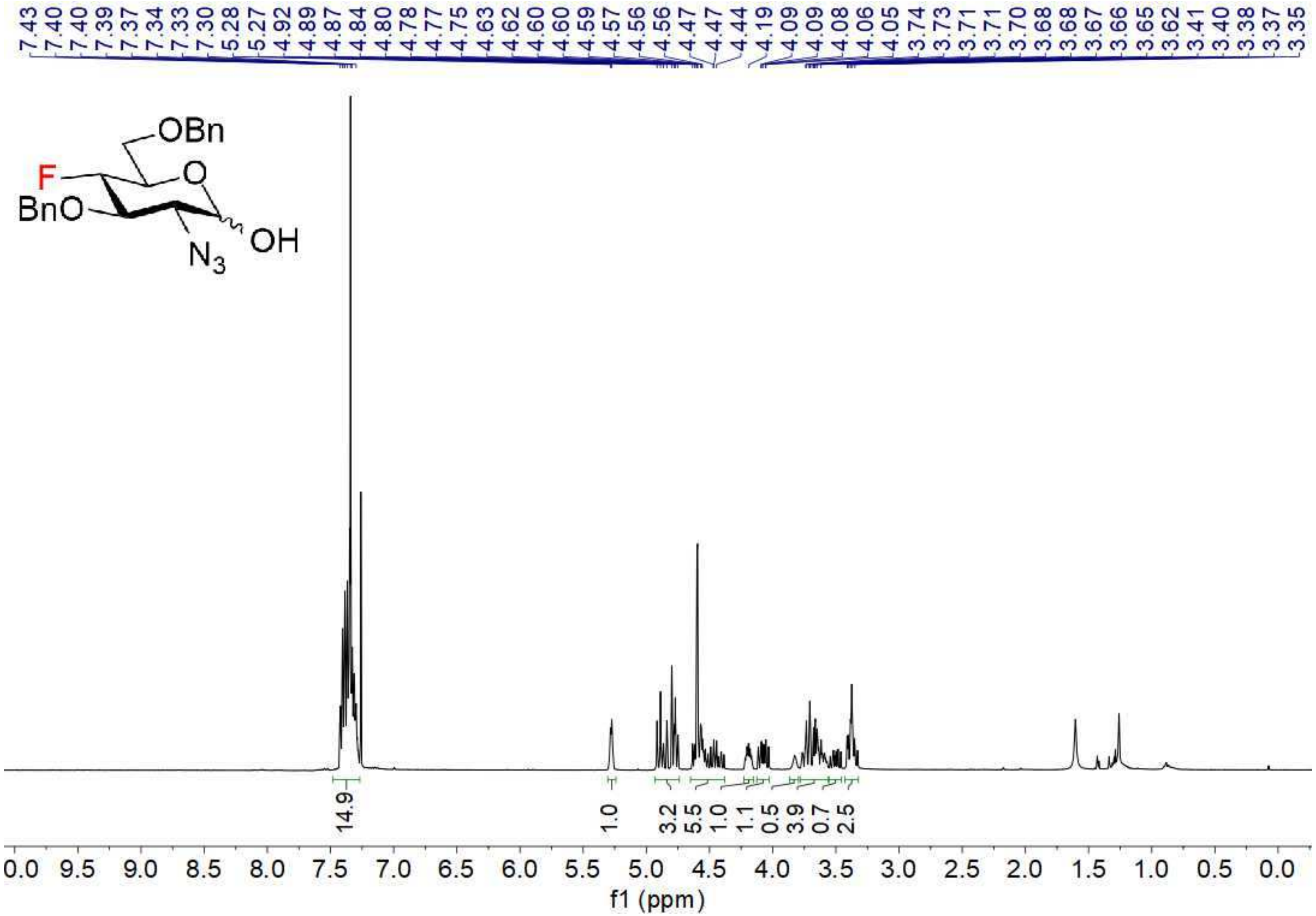


^{1}H-^{13}C HSQC 43

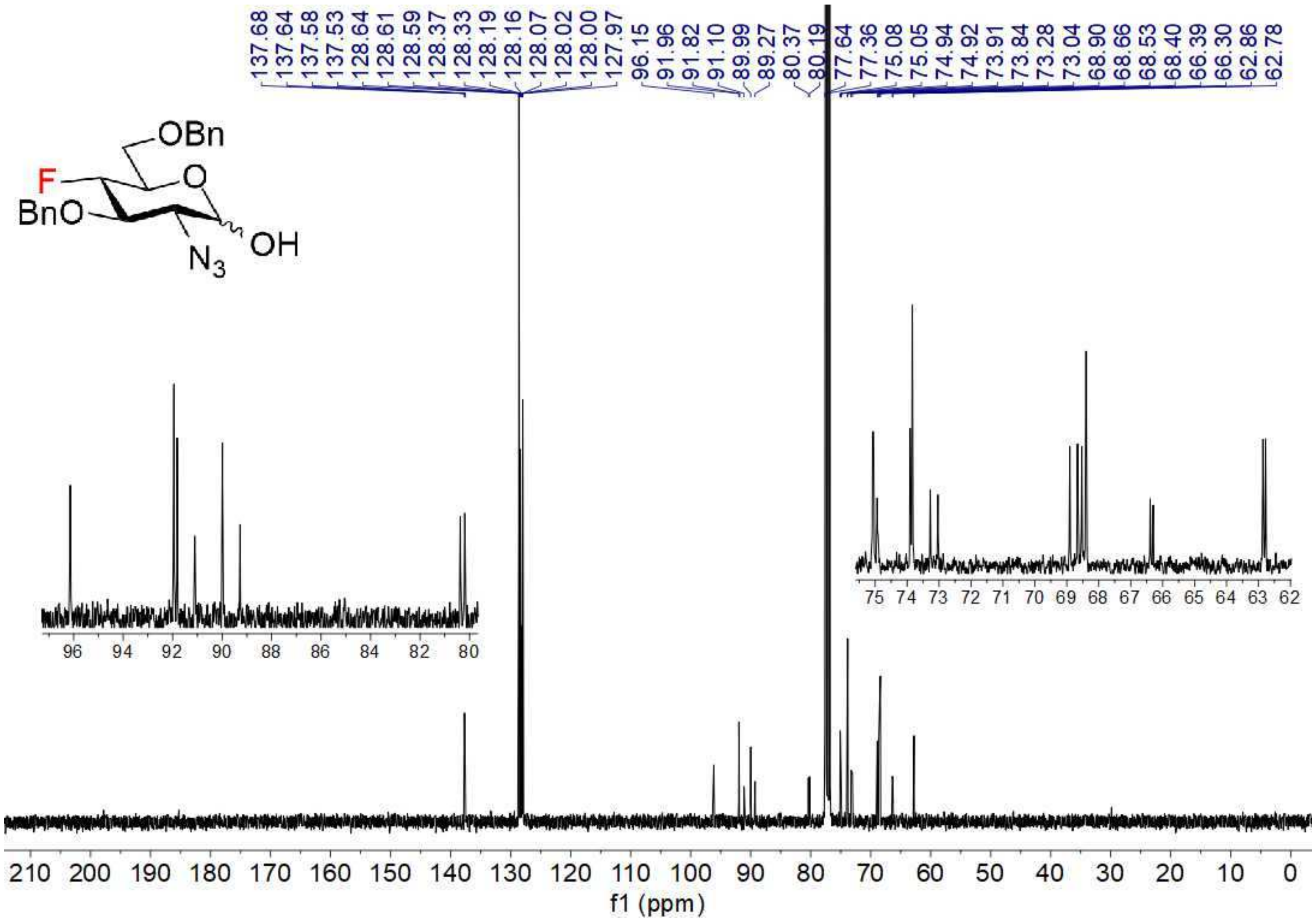


NMR COMPOUND 44

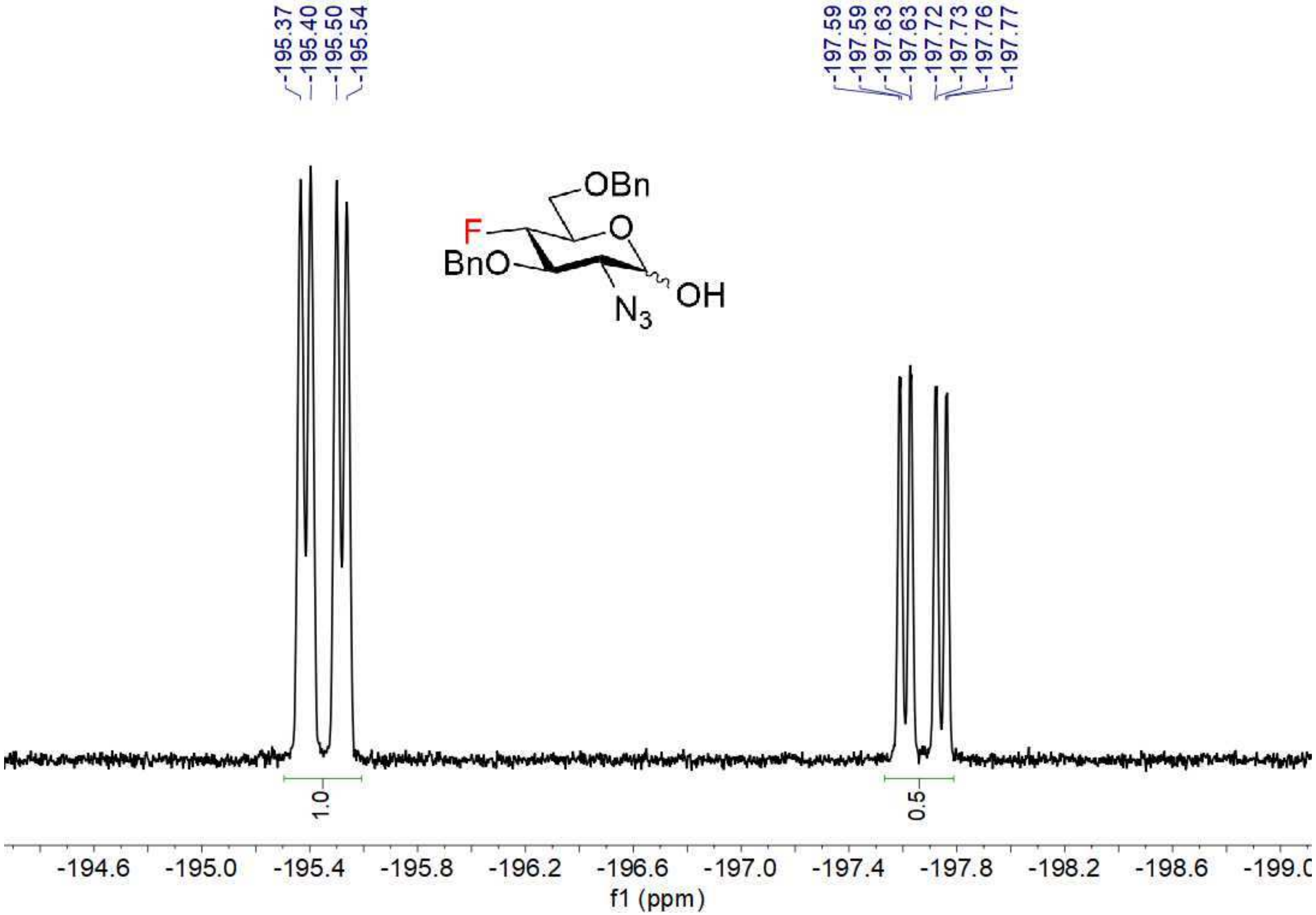
^{1}H NMR (400 MHz, CDCl$_3$) 44 (α/β ca. 2/1)



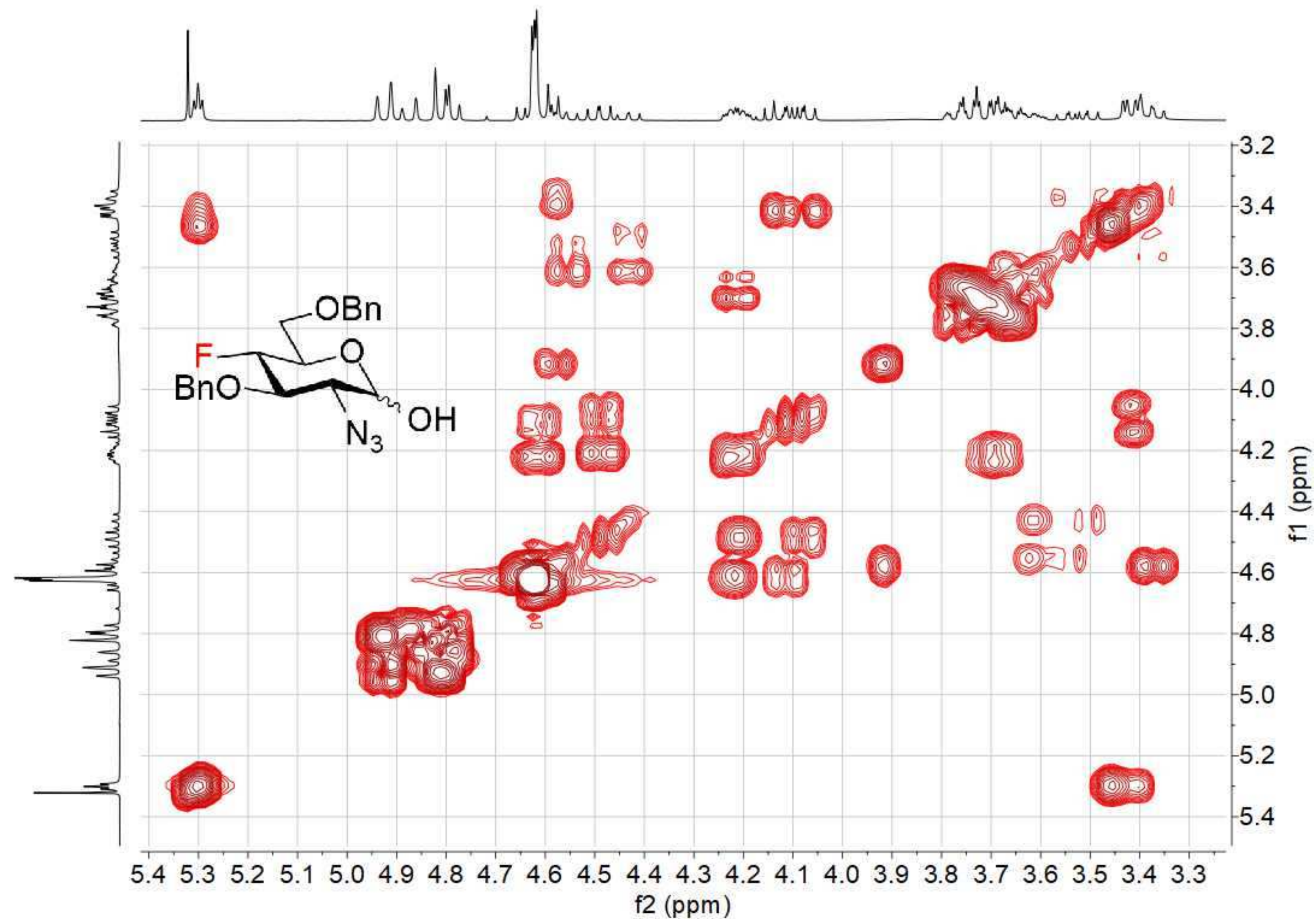
^{13}C NMR (100 MHz, $CDCl_3$) 44



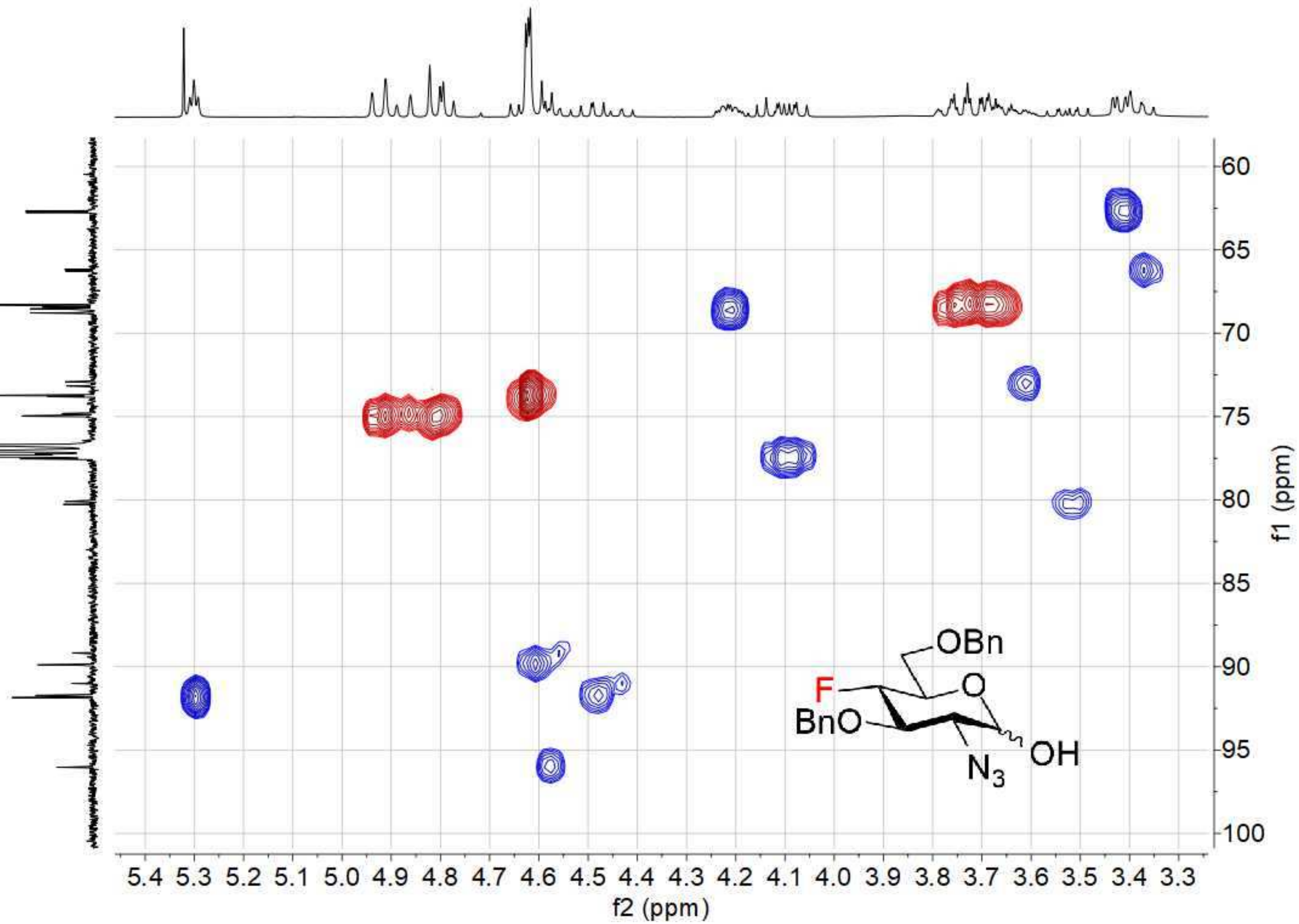
[19]F NMR (376 MHz, CDCl$_3$) 44 (α/β ca. 2/1)



^{1}H-^{1}H COSY 44

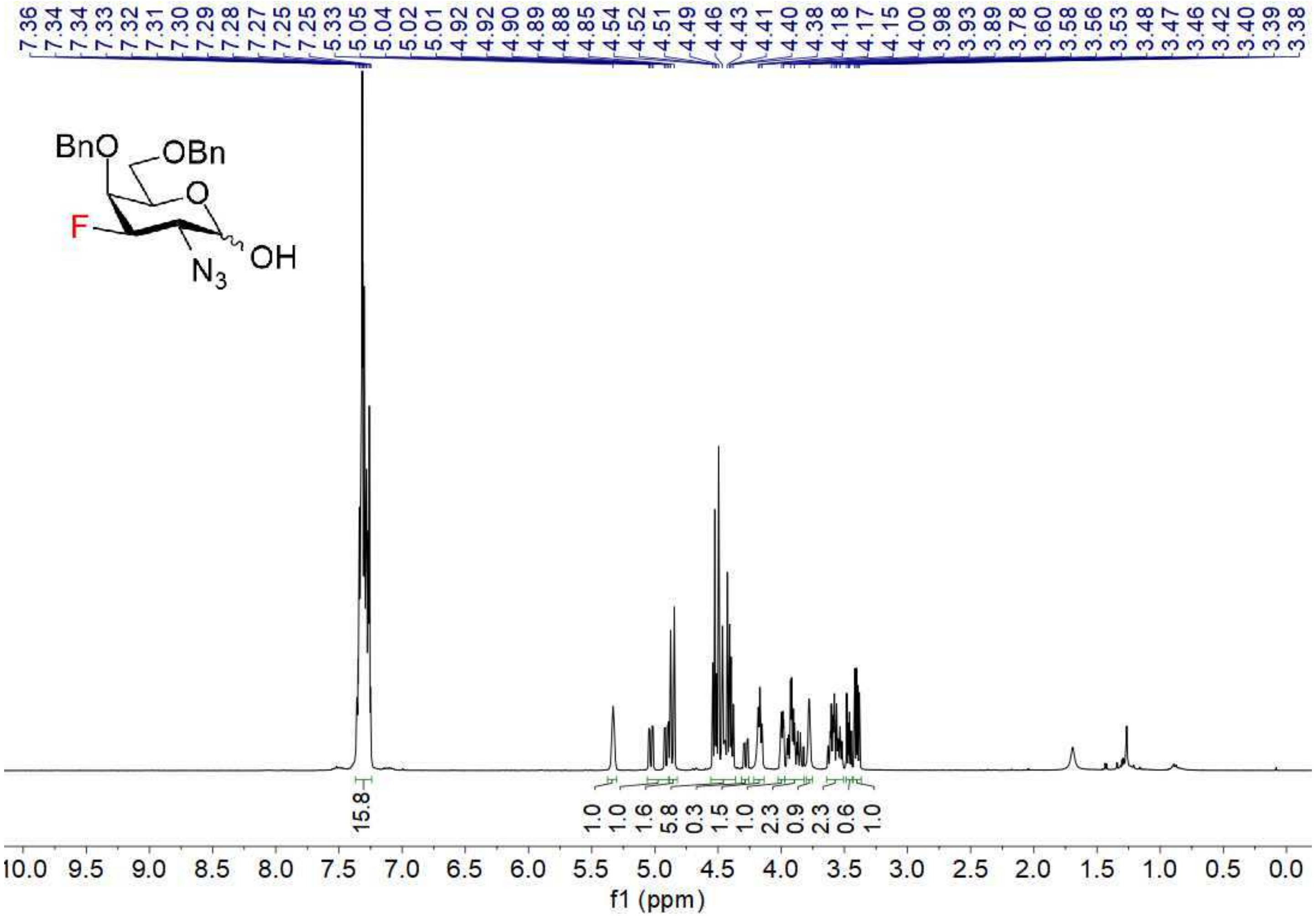


^{1}H-^{13}C HSQC 44

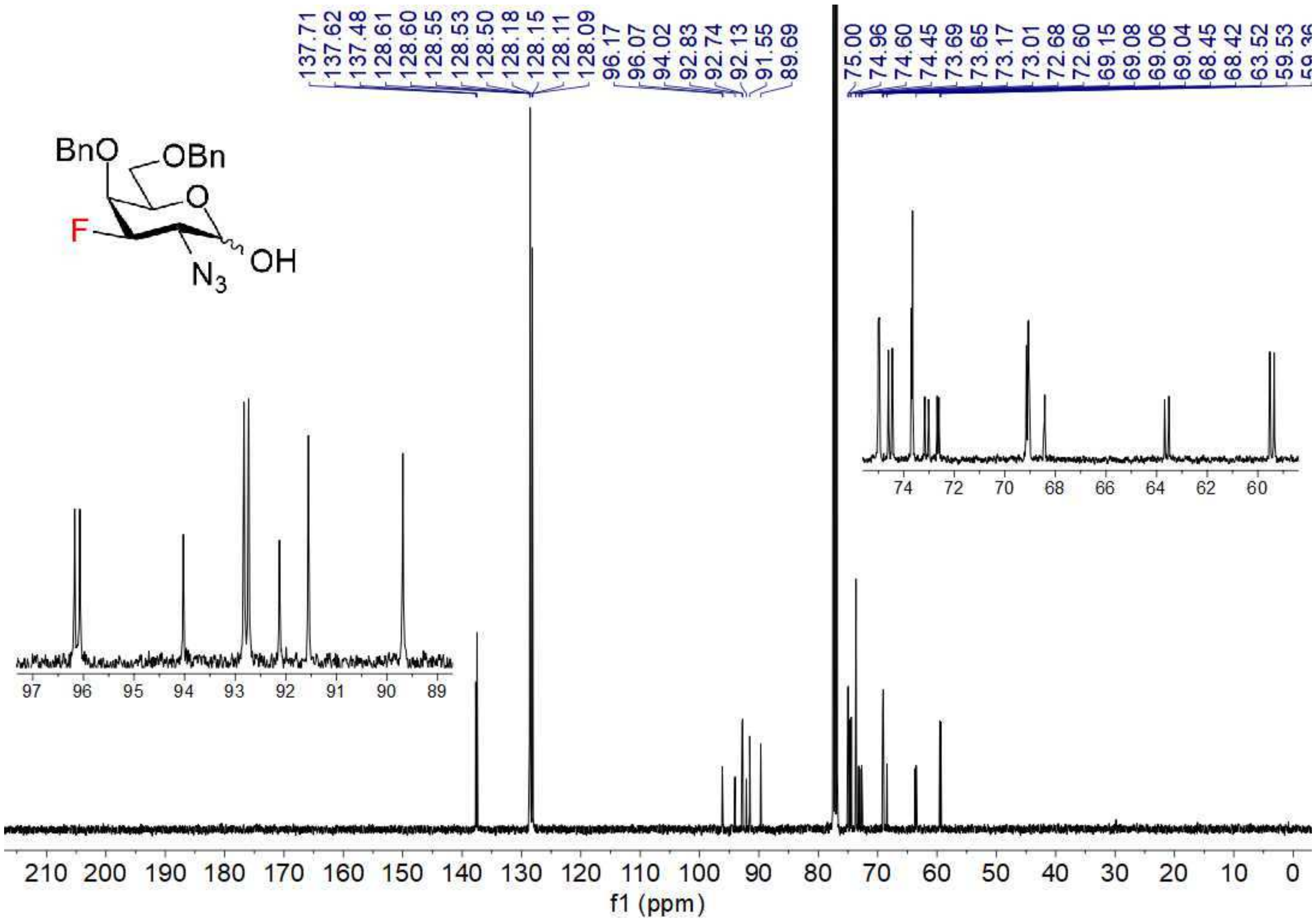


NMR COMPOUND 45

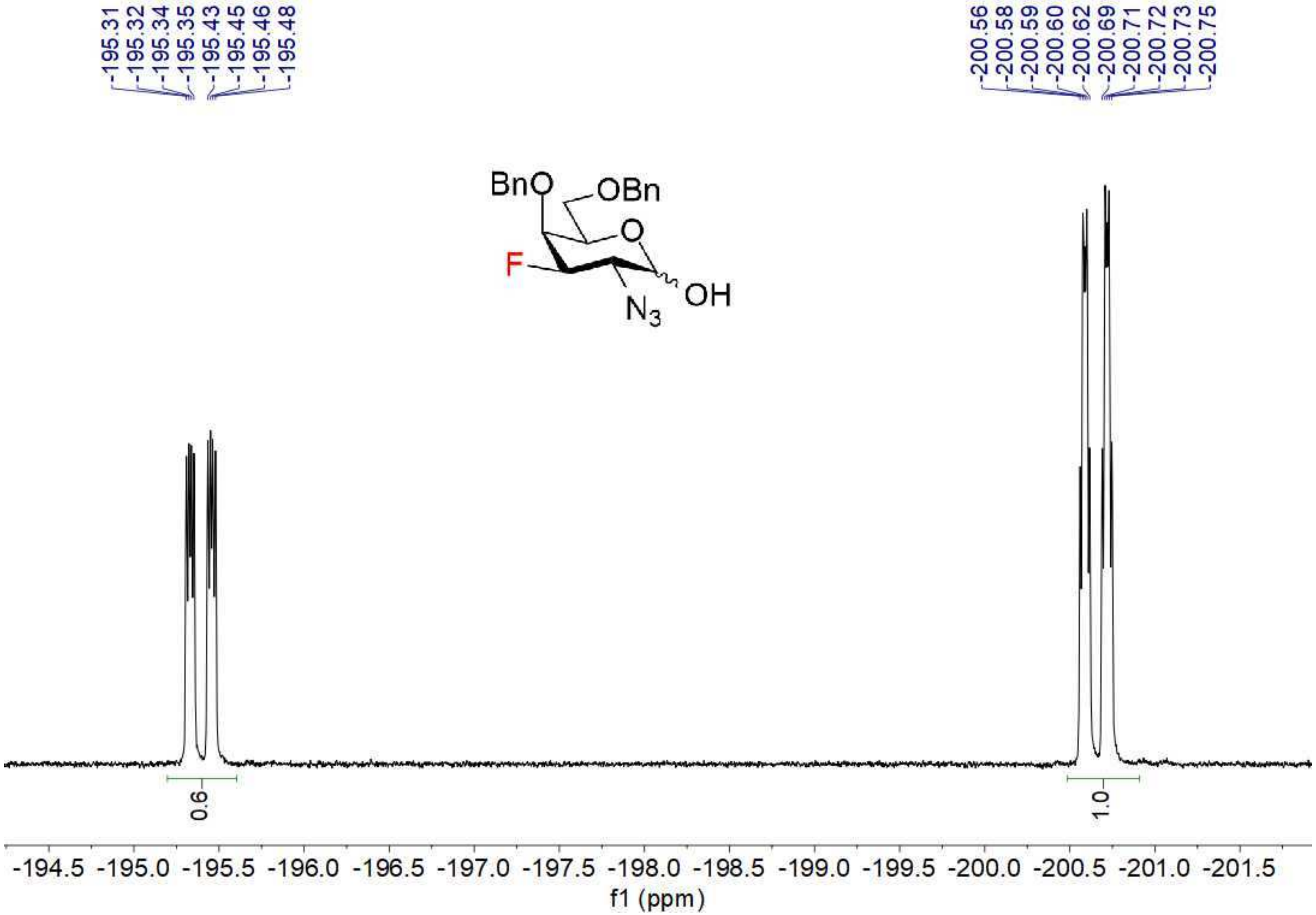
[1]H NMR (400 MHz, $CDCl_3$) 45 (α/β ca. 5/3)



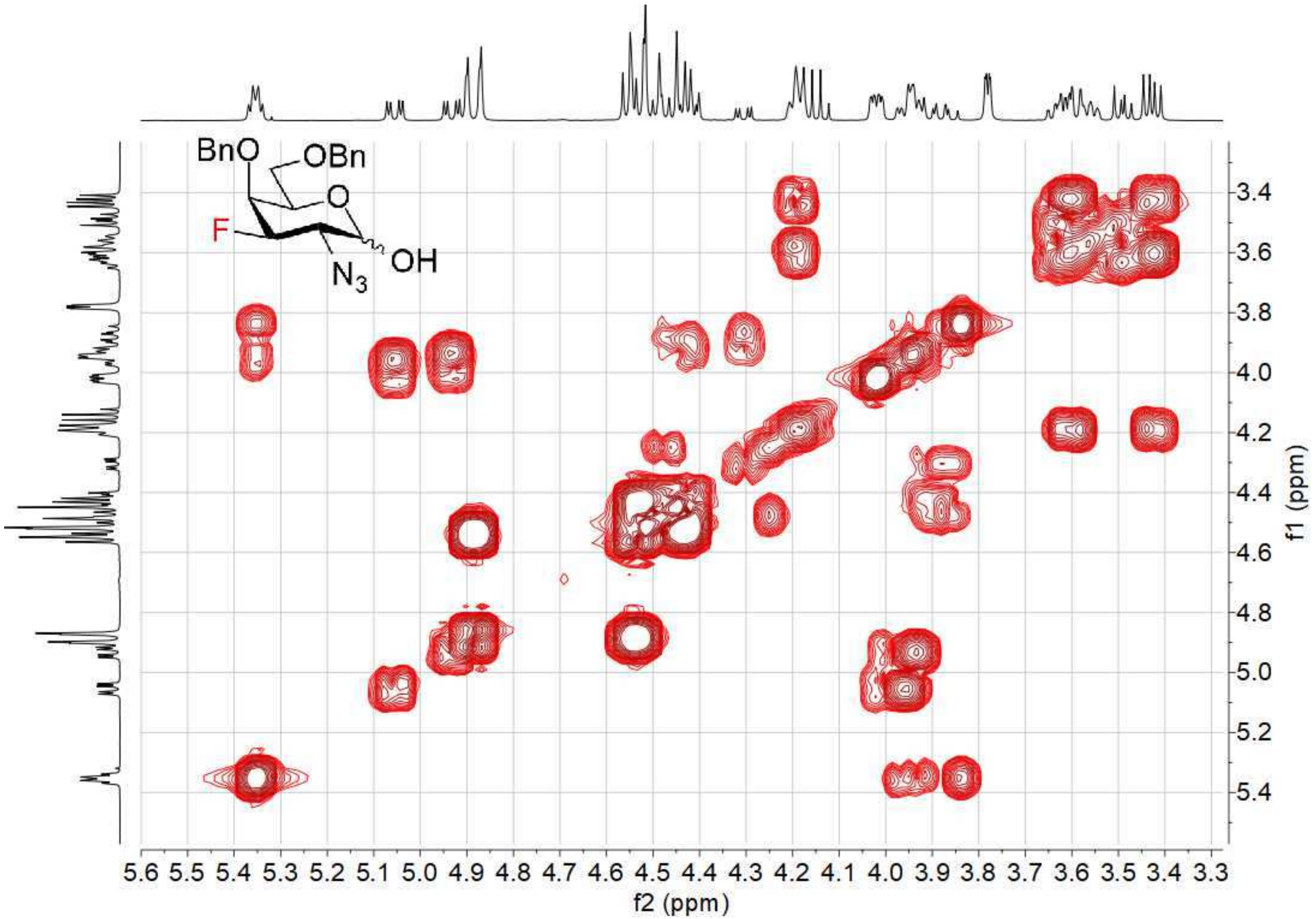
[13]C NMR (100 MHz, $CDCl_3$) 45



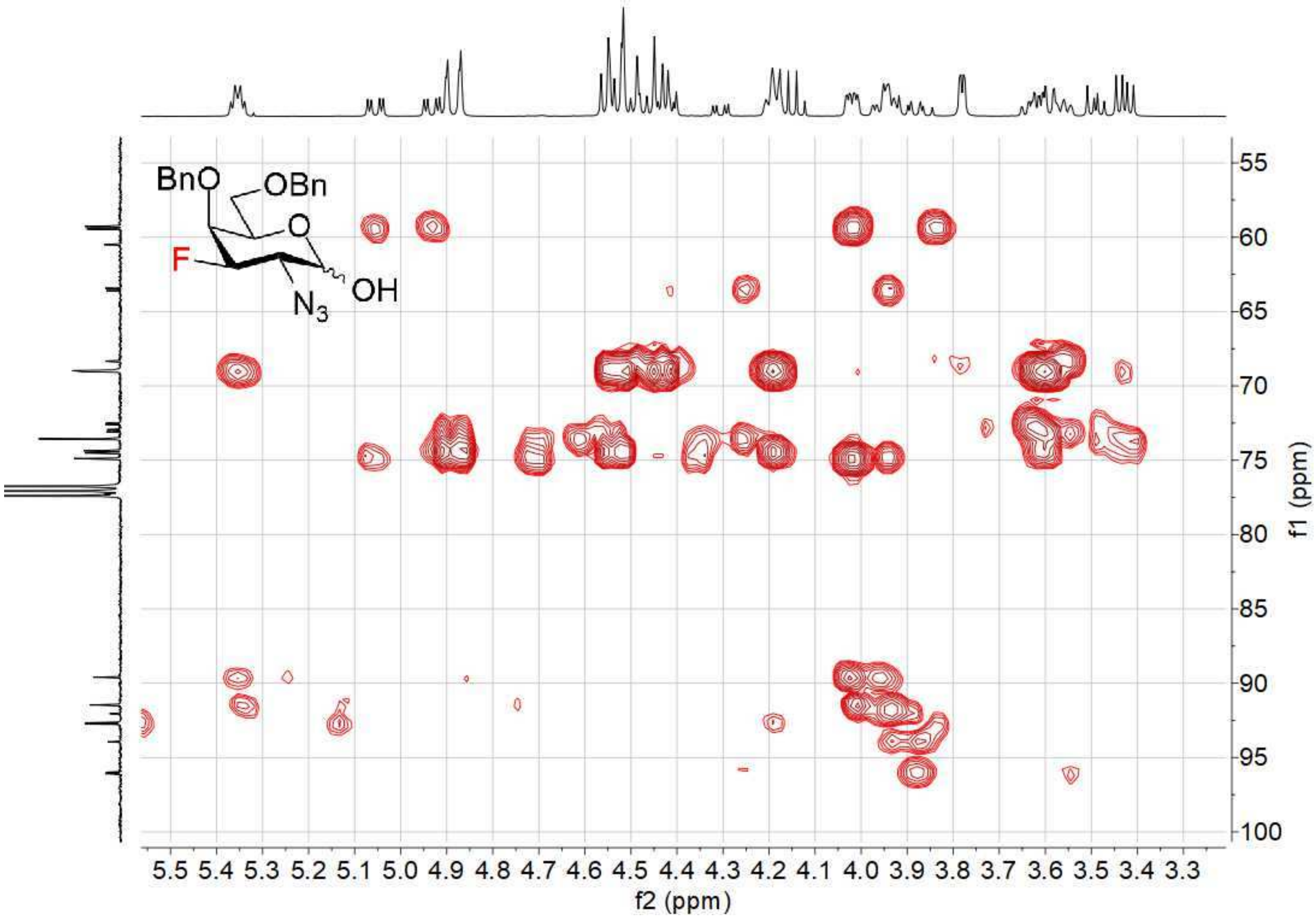
^{19}F NMR (376 MHz, CDCl$_3$) 45 (α/β ca. 5/3)



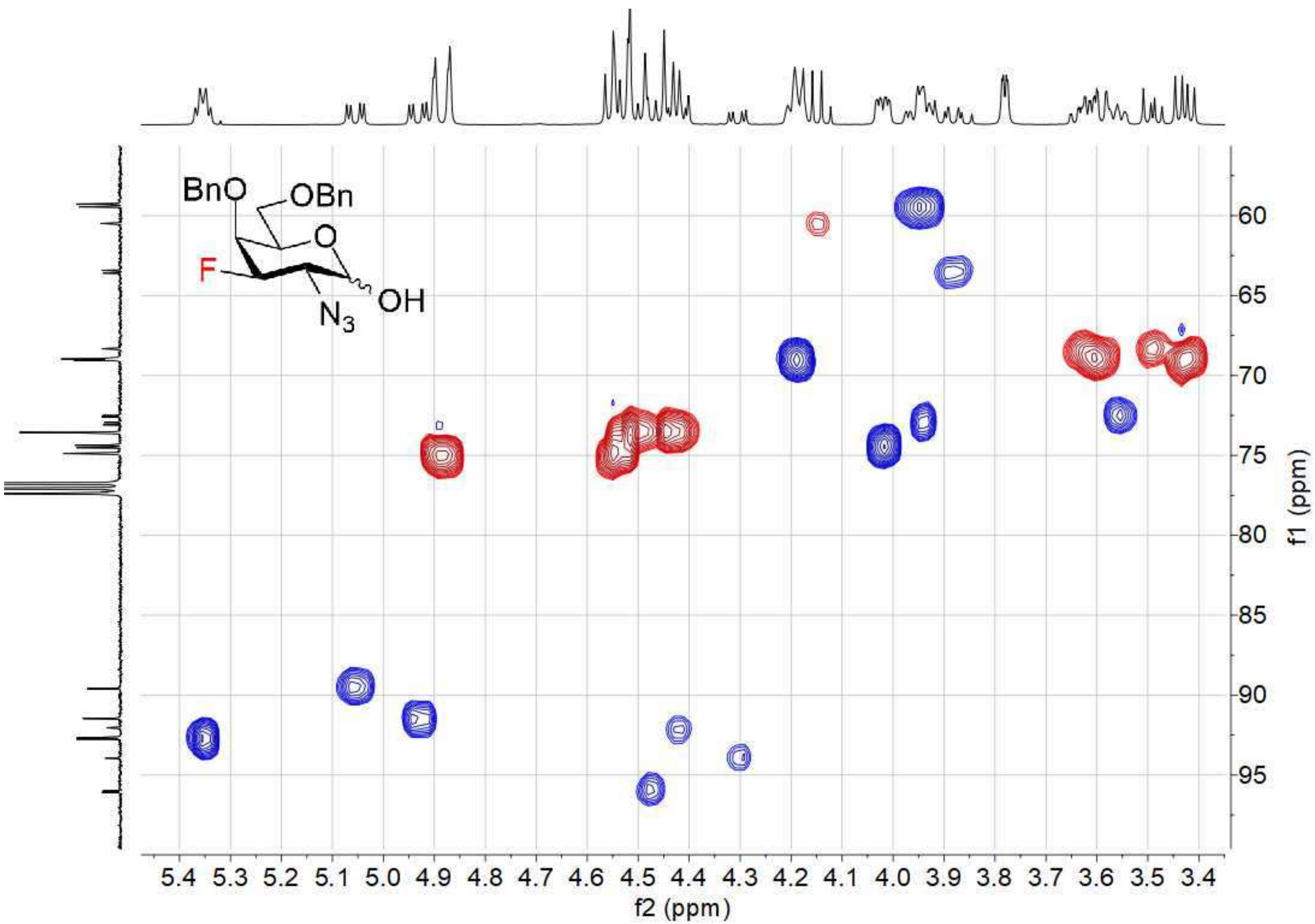
[1]H-[1]H COSY 45


BnO
OBn
O
F
N3
OH
5.6 5.5 5.4 5.3 5.2 5.1 5.0 4.9 4.8 4.7 4.6 4.5 4.4 4.3 4.2 4.1 4.0 3.9 3.8 3.7 3.6 3.5 3.4 3.3
f2 (ppm)
3.4
3.6
3.8
4.0
4.2
4.4
4.6
4.8
5.0
5.2
5.4
f1 (ppm)

^{1}H-^{13}C HMBC 45

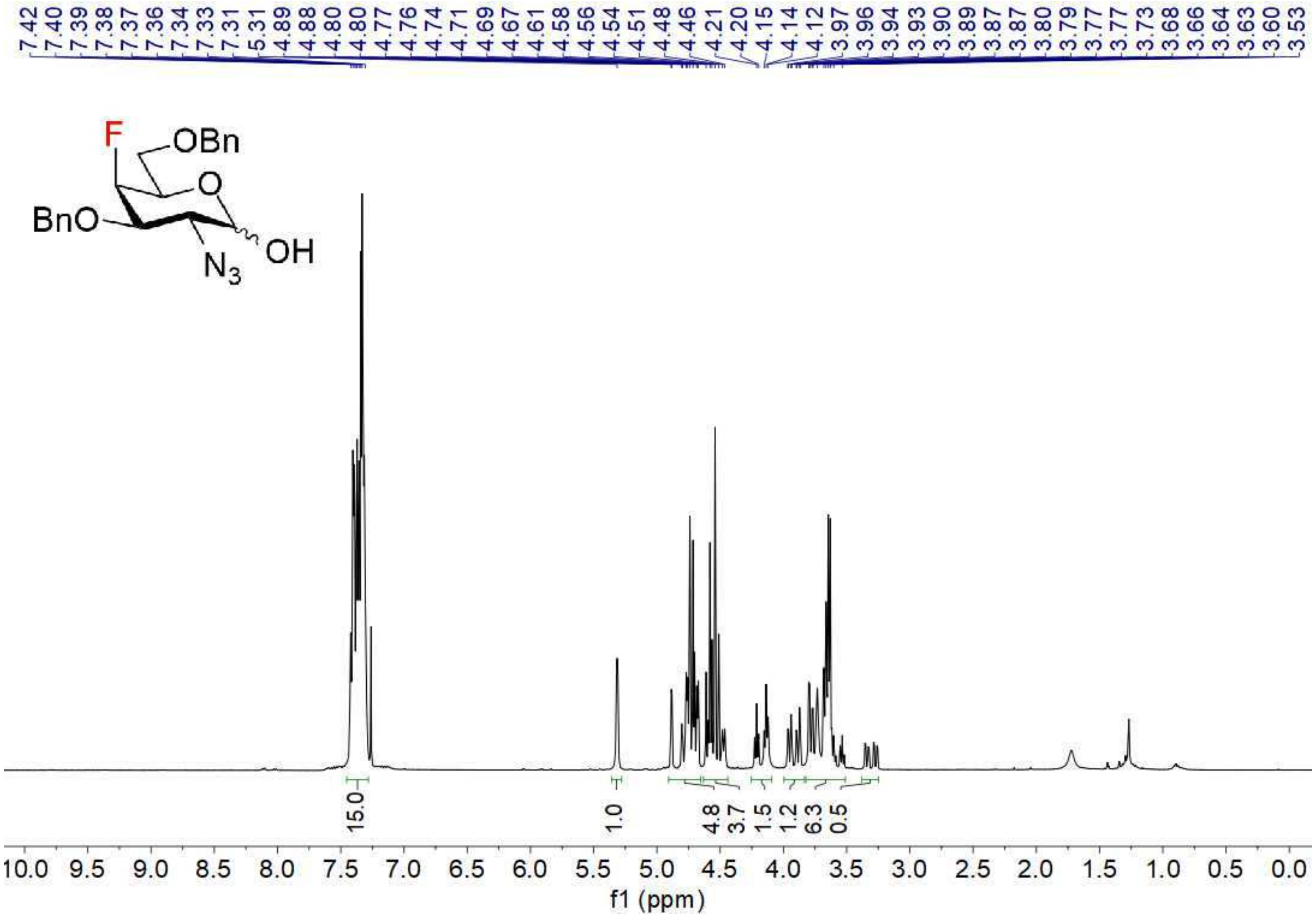


^{1}H-^{13}C HSQC 45

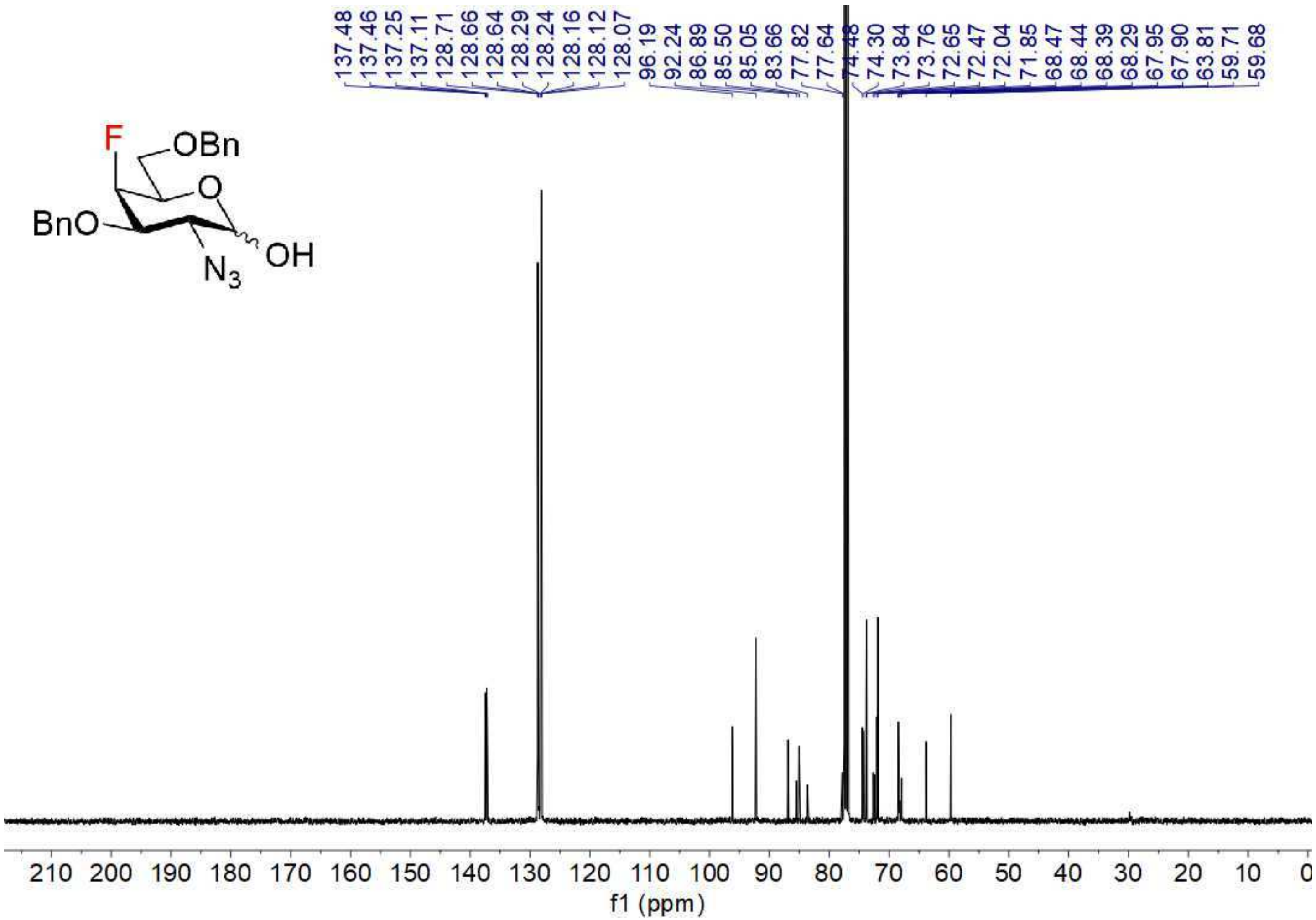


NMR COMPOUND 46

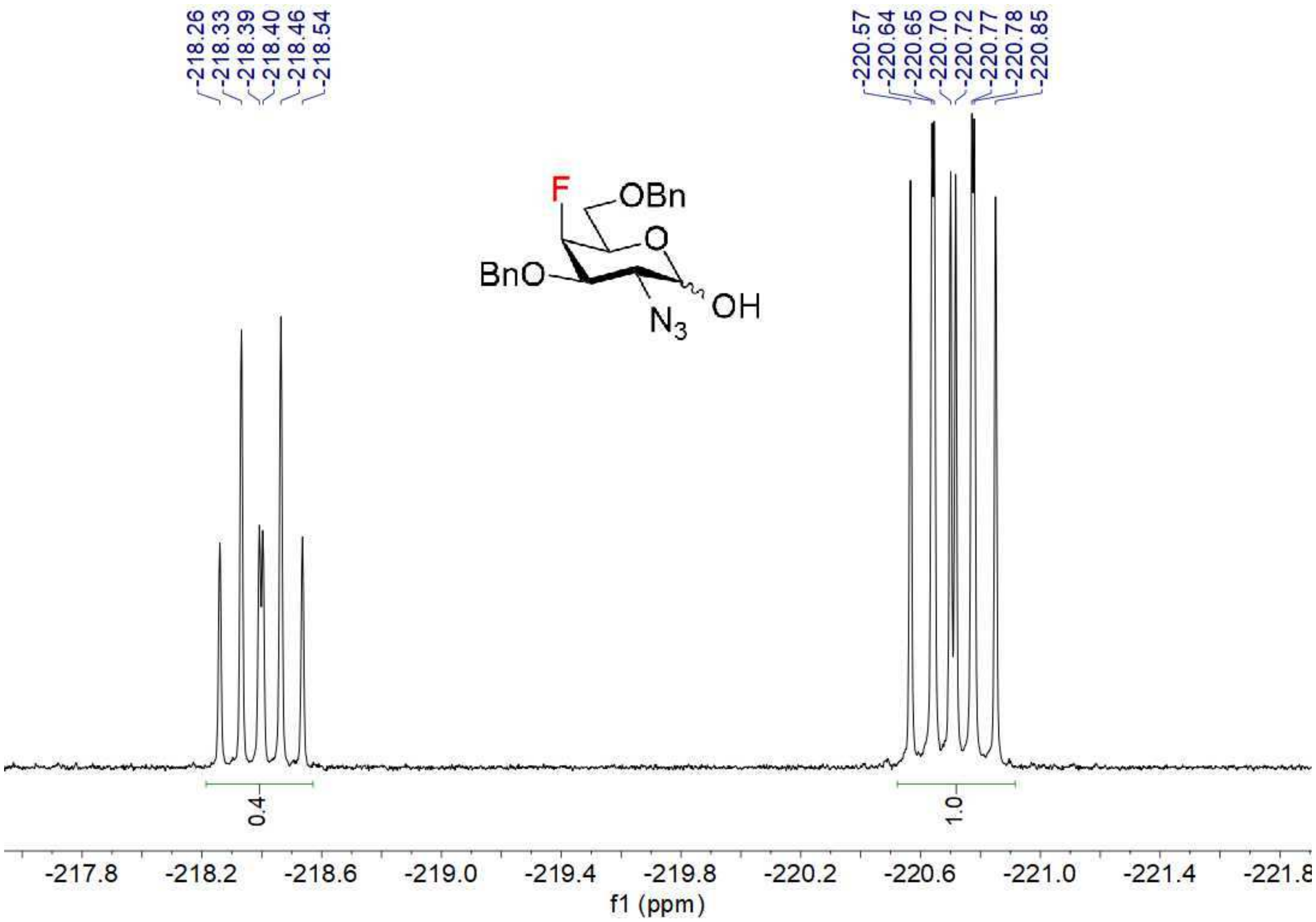
^{1}H NMR (400 MHz, $CDCl_3$) 46 (α/β ca. 5/2)



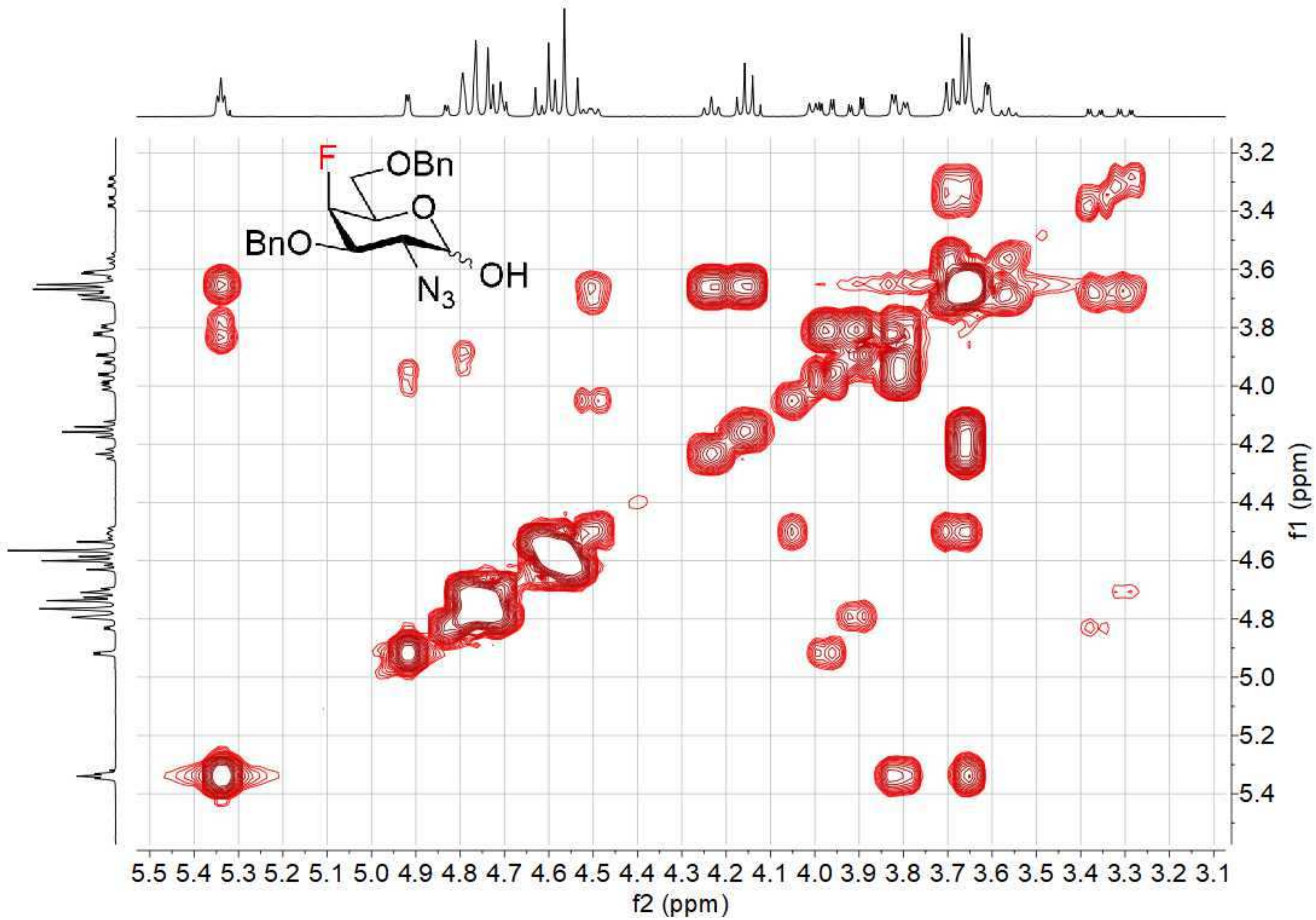
^{13}C NMR (100 MHz, CDCl$_3$) 46



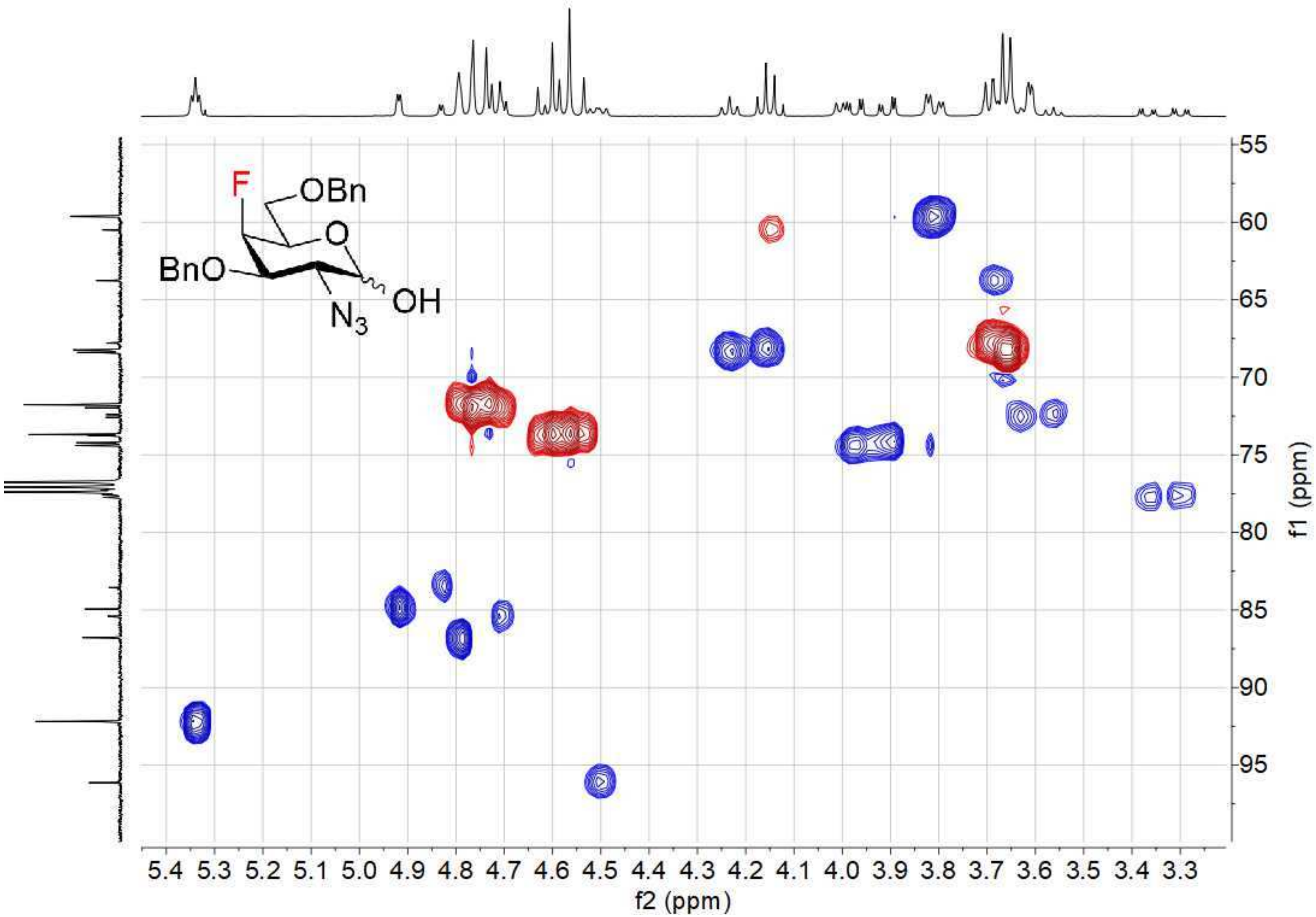
^{19}F NMR (376 MHz, CDCl$_3$) 46 (α/β ca. 5/2)



[1]H-[1]H COSY 46

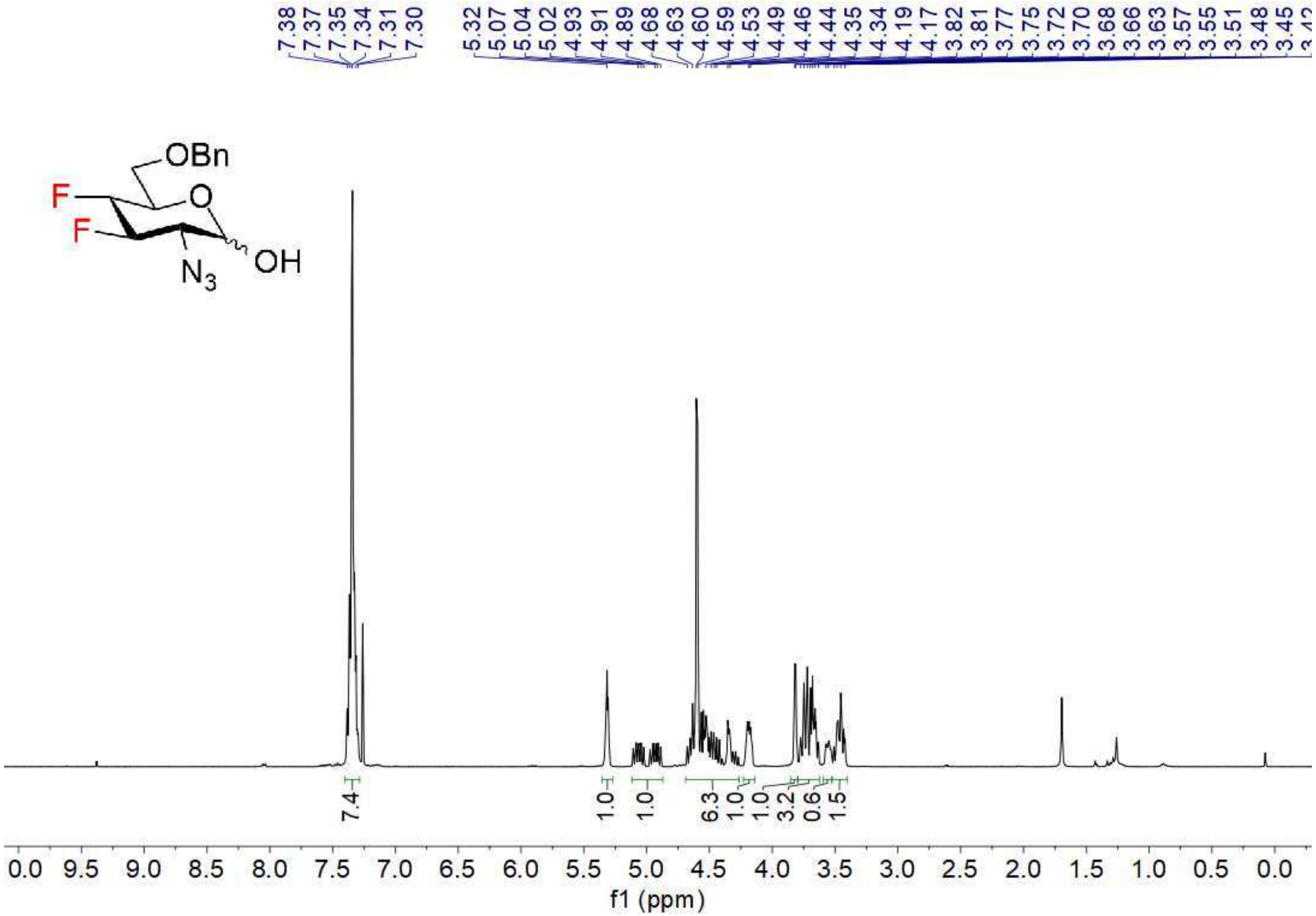

F
OBn
O
BnO
OH
N3
f1 (ppm)
f2 (ppm)
5.5 5.4 5.3 5.2 5.1 5.0 4.9 4.8 4.7 4.6 4.5 4.4 4.3 4.2 4.1 4.0 3.9 3.8 3.7 3.6 3.5 3.4 3.3 3.2 3.1
3.2 3.4 3.6 3.8 4.0 4.2 4.4 4.6 4.8 5.0 5.2 5.4

[1]H-[13]C HSQC 46

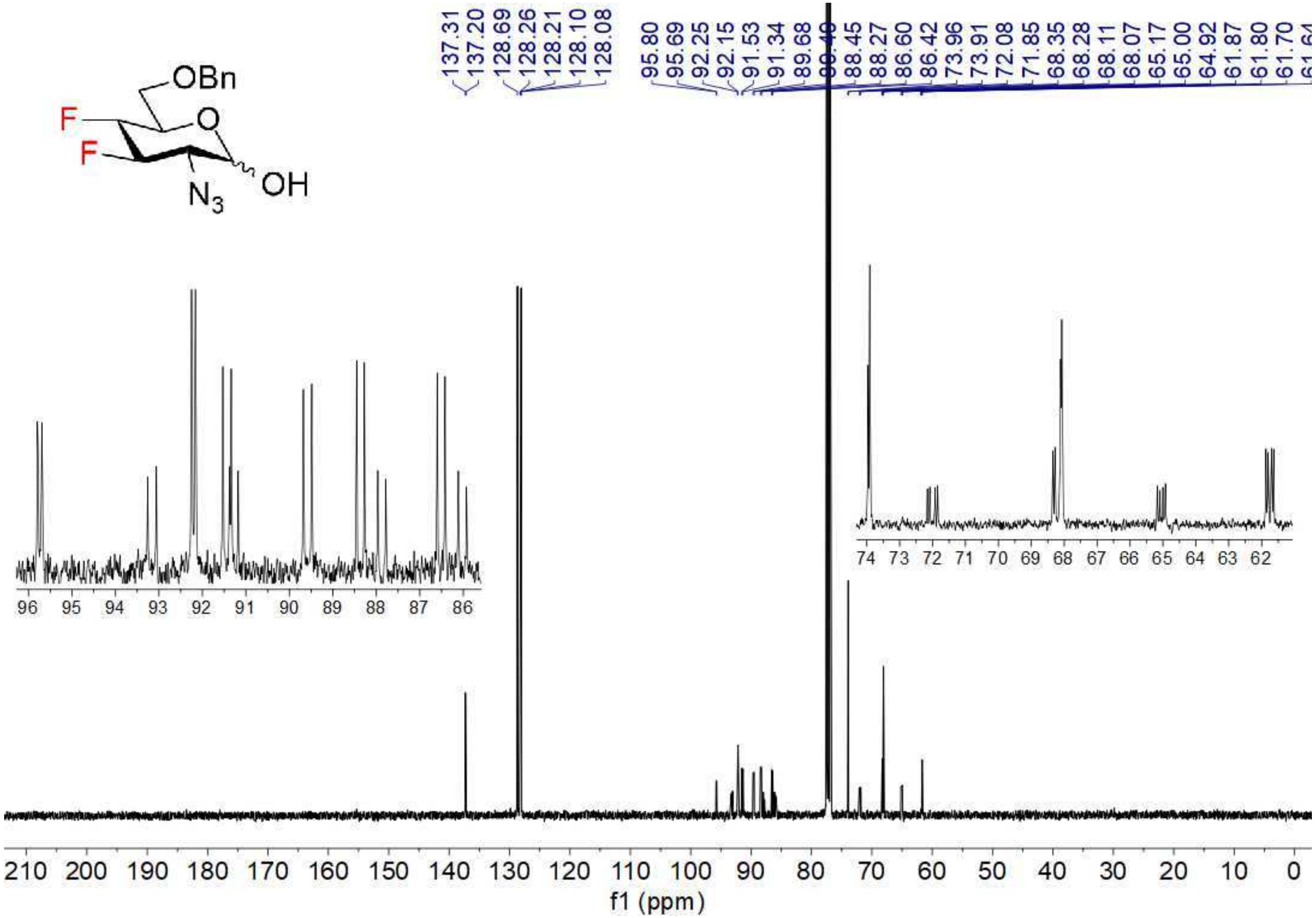


NMR COMPOUND 47

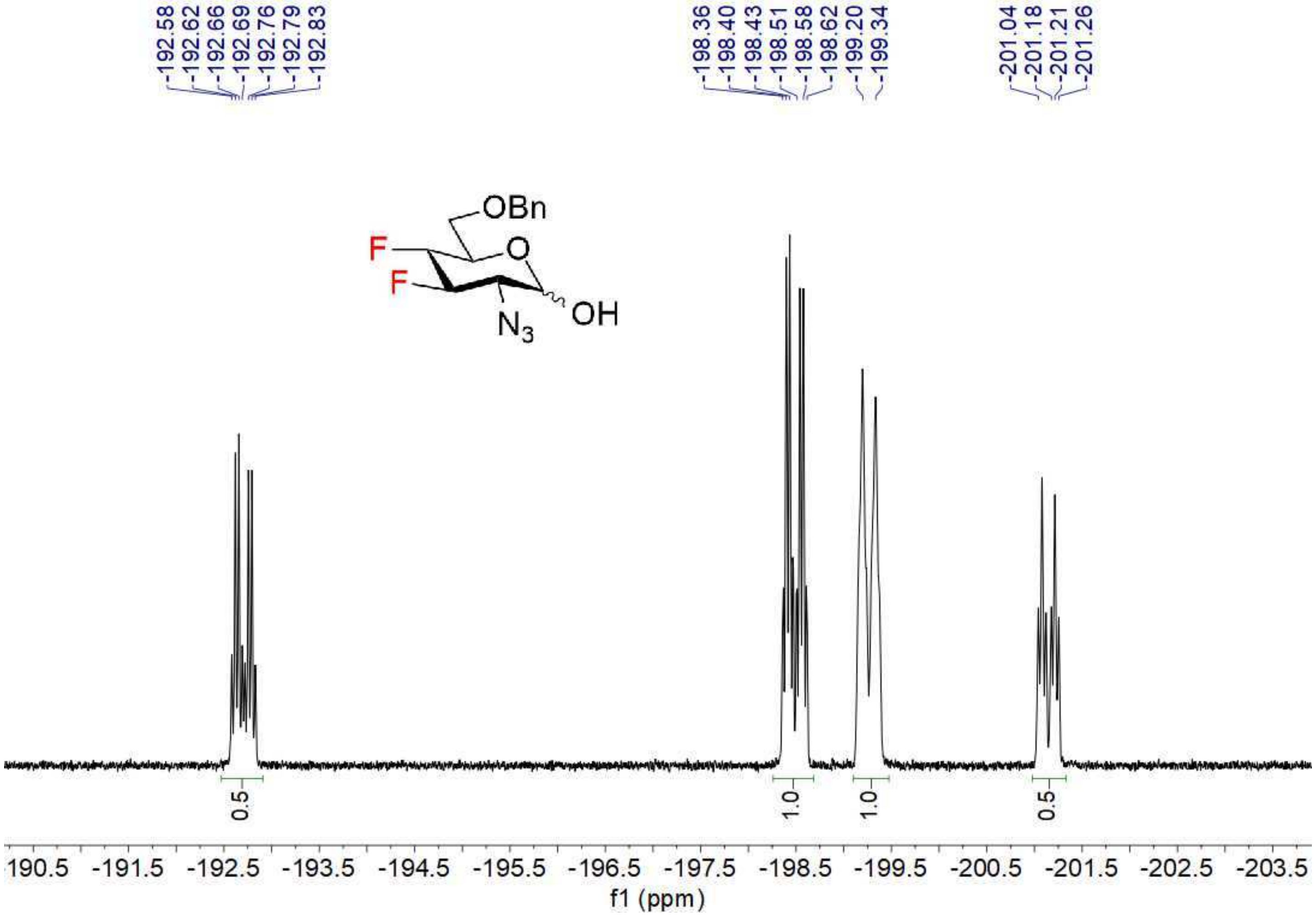
^{1}H NMR (400 MHz, $CDCl_3$) 47 (α/β ca. 2/1)



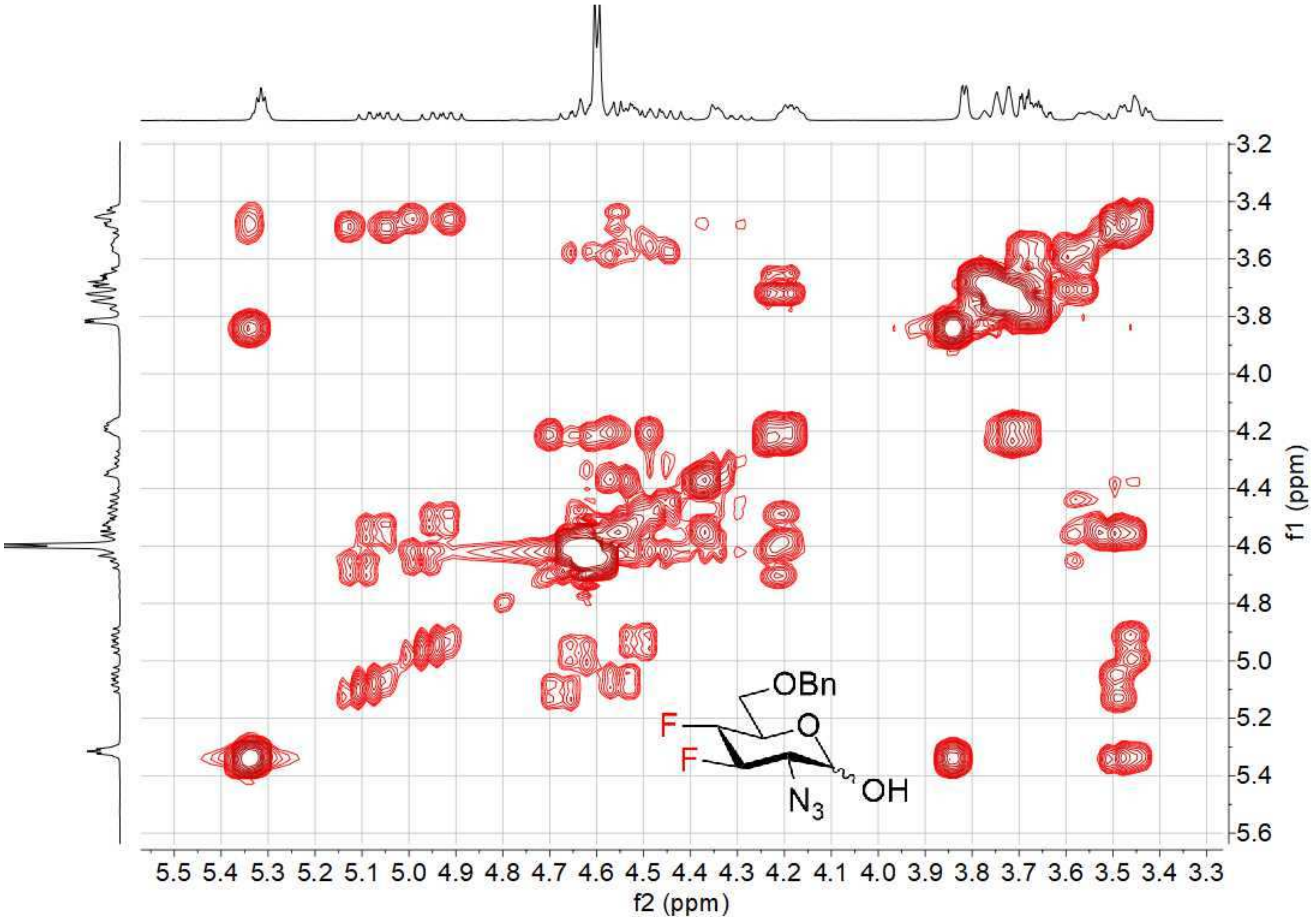
^{13}C NMR (100 MHz, $CDCl_3$) 47



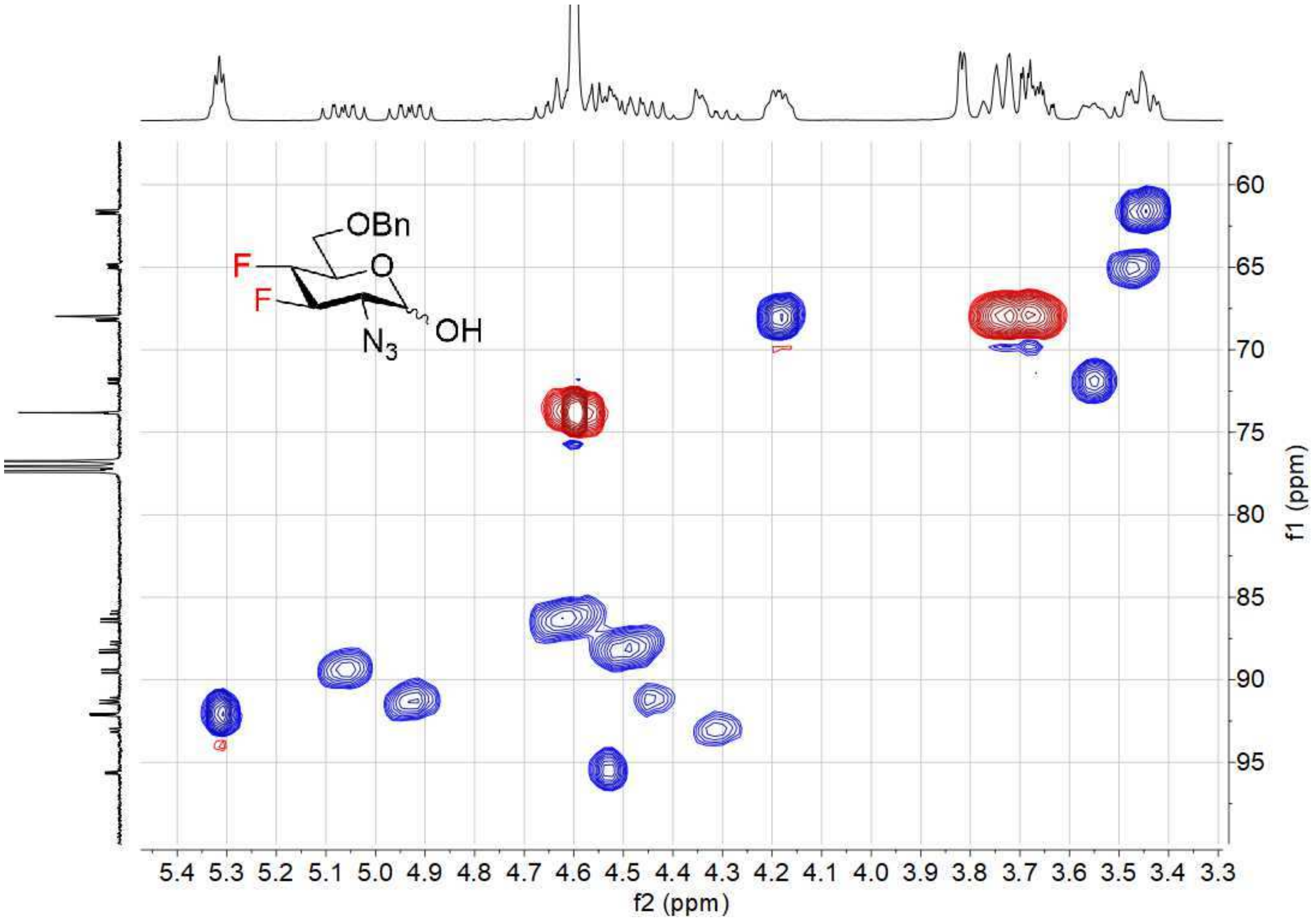
[19]F NMR (376 MHz, CDCl$_3$) 47 (α/β ca. 2/1)



^{1}H-^{1}H COSY 47

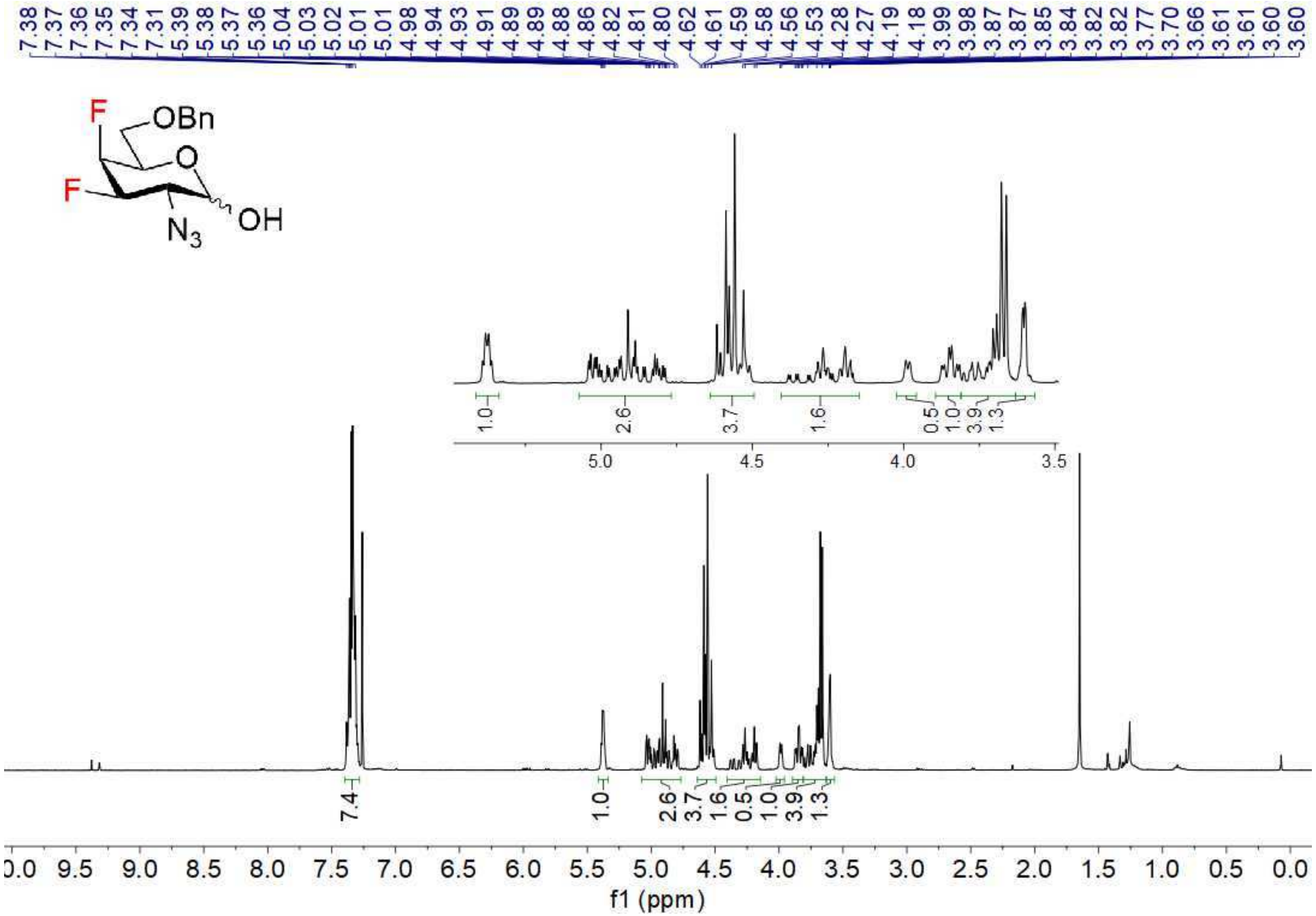


^{1}H-^{13}C HSQC 47

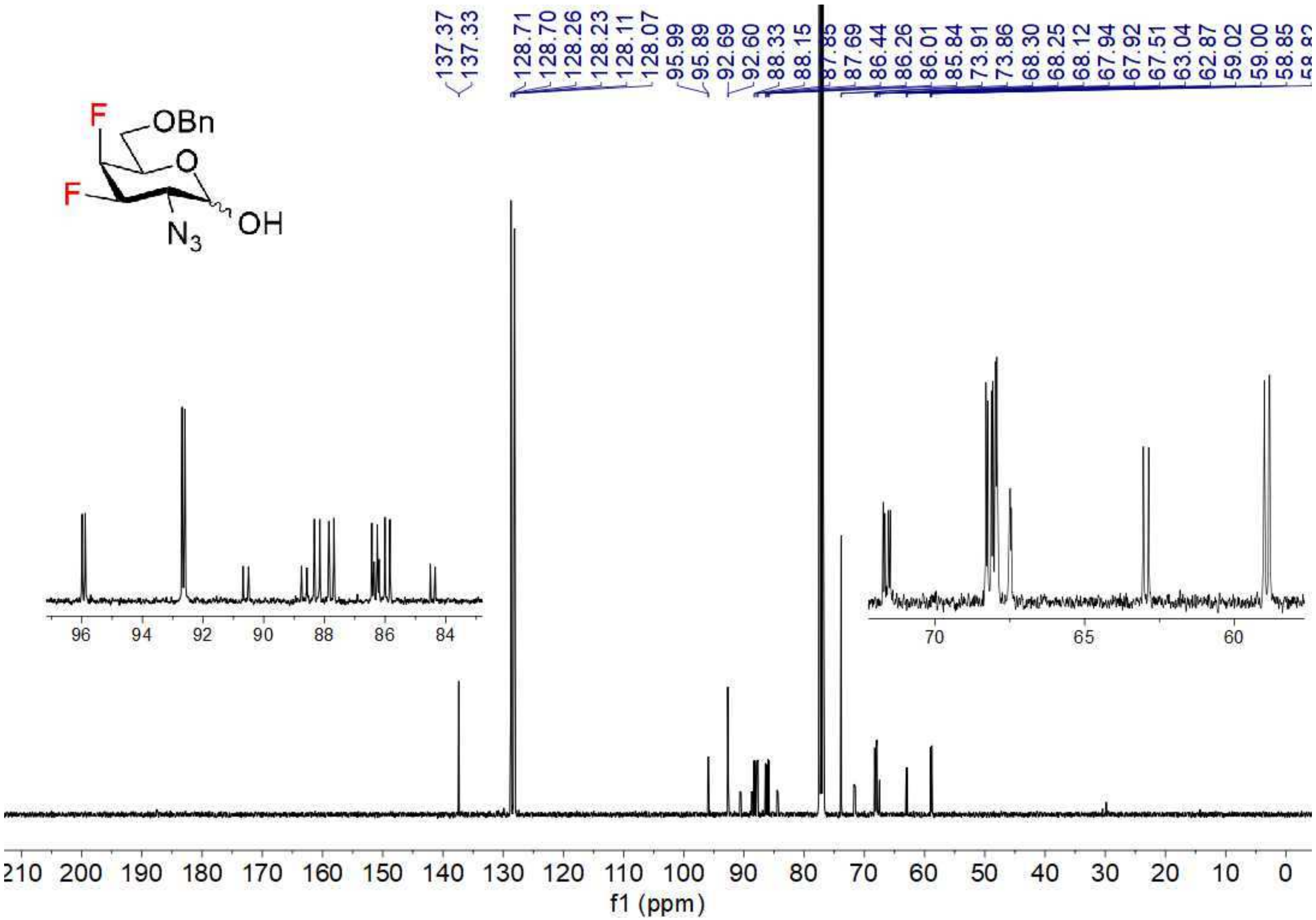


NMR COMPOUND 48

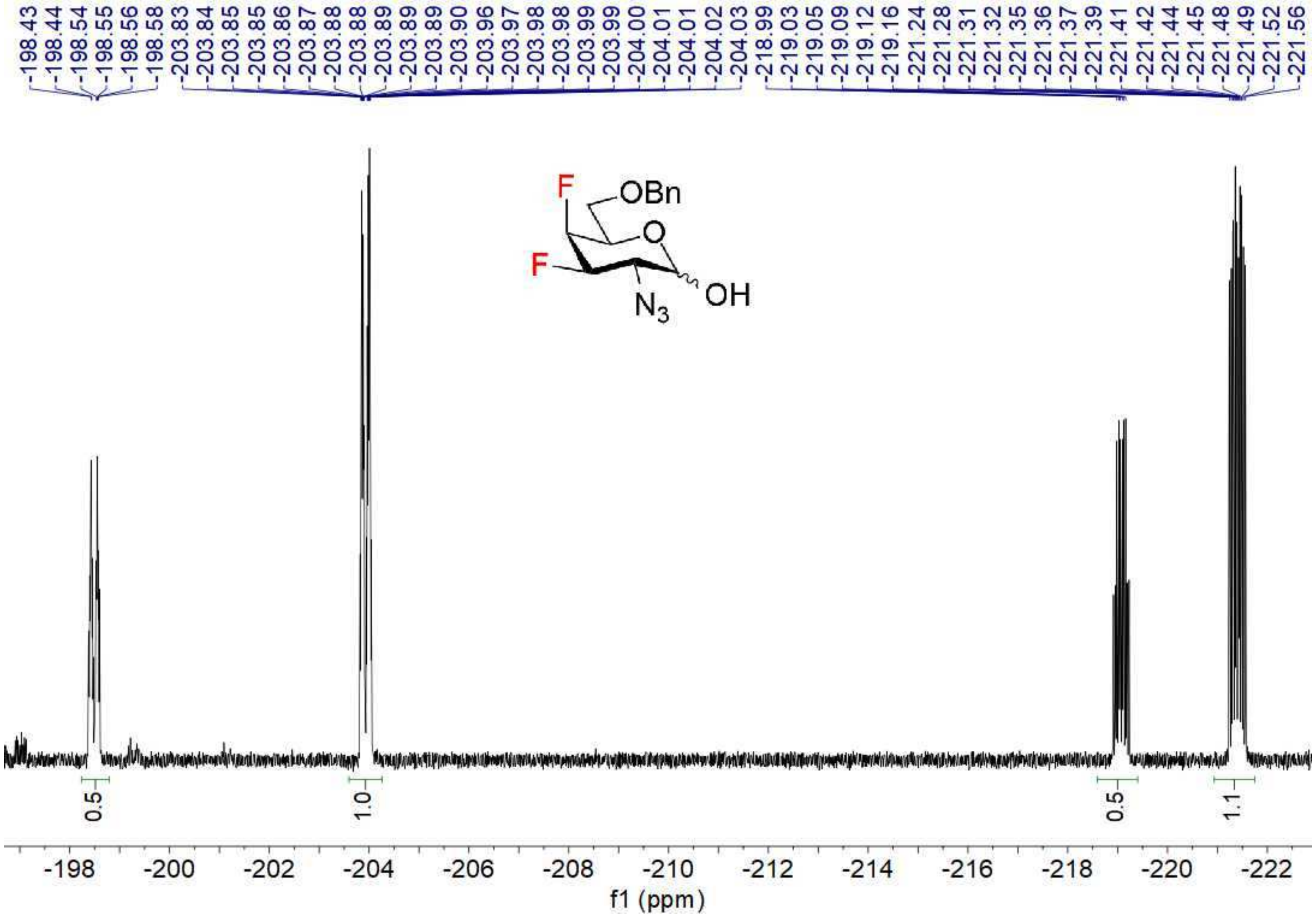
^{1}H NMR (400 MHz, $CDCl_3$) 48 (α/β ca. 2/1)



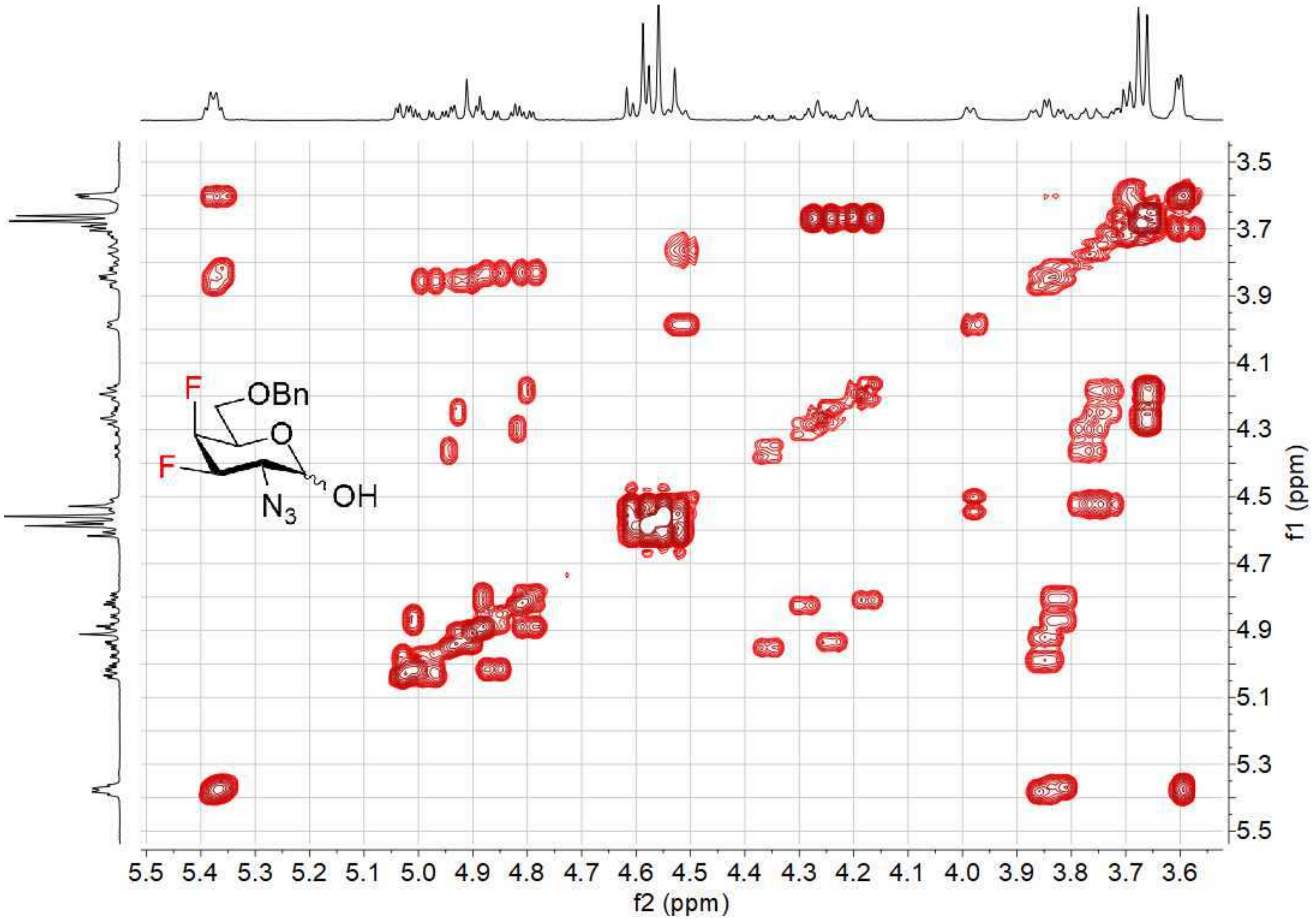
^{13}C NMR (100 MHz, CDCl$_3$) 48



^{19}F NMR (376 MHz, CDCl$_3$) 48 (α/β ca. 2/1)

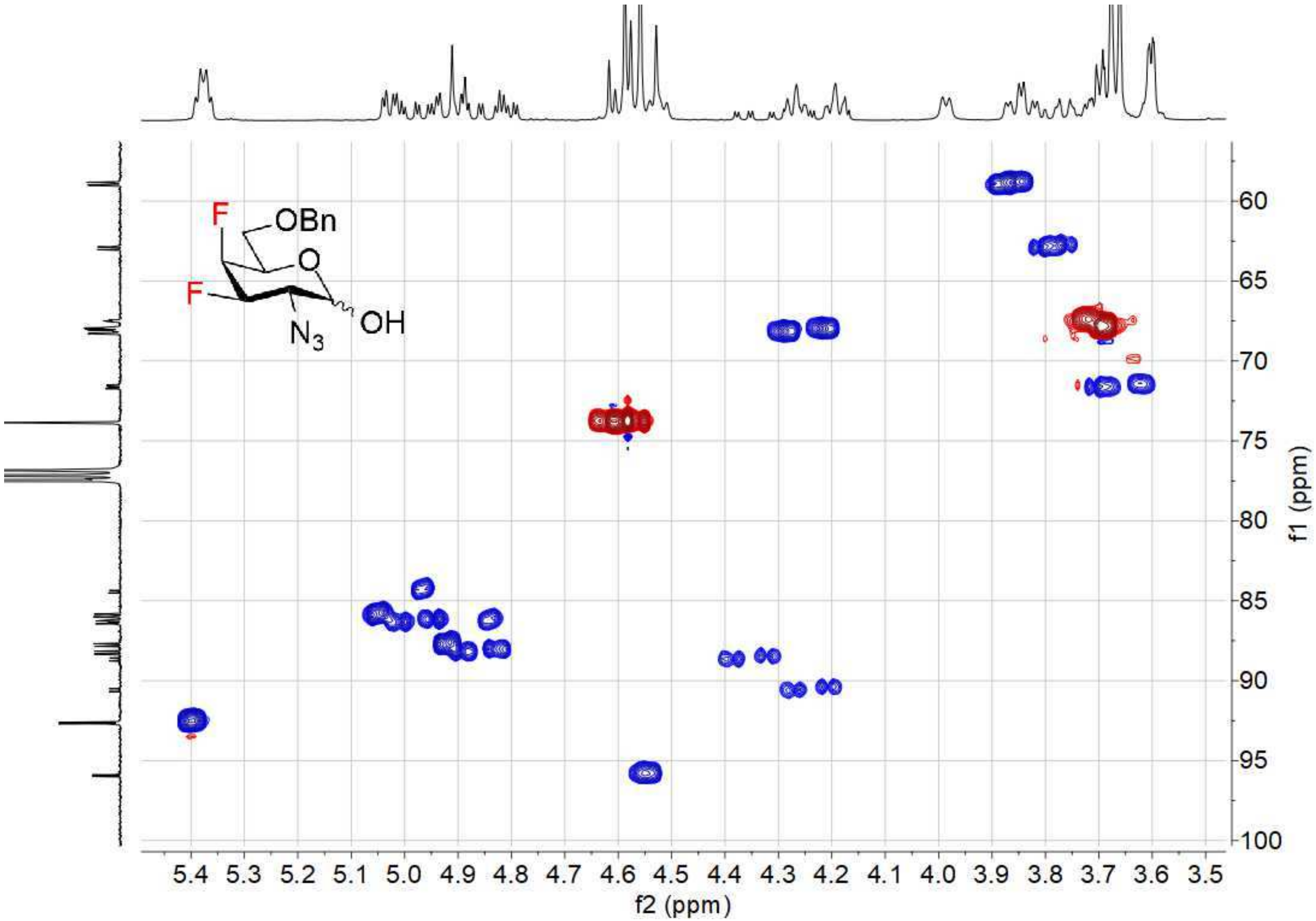


[1]H-[1]H COSY 48



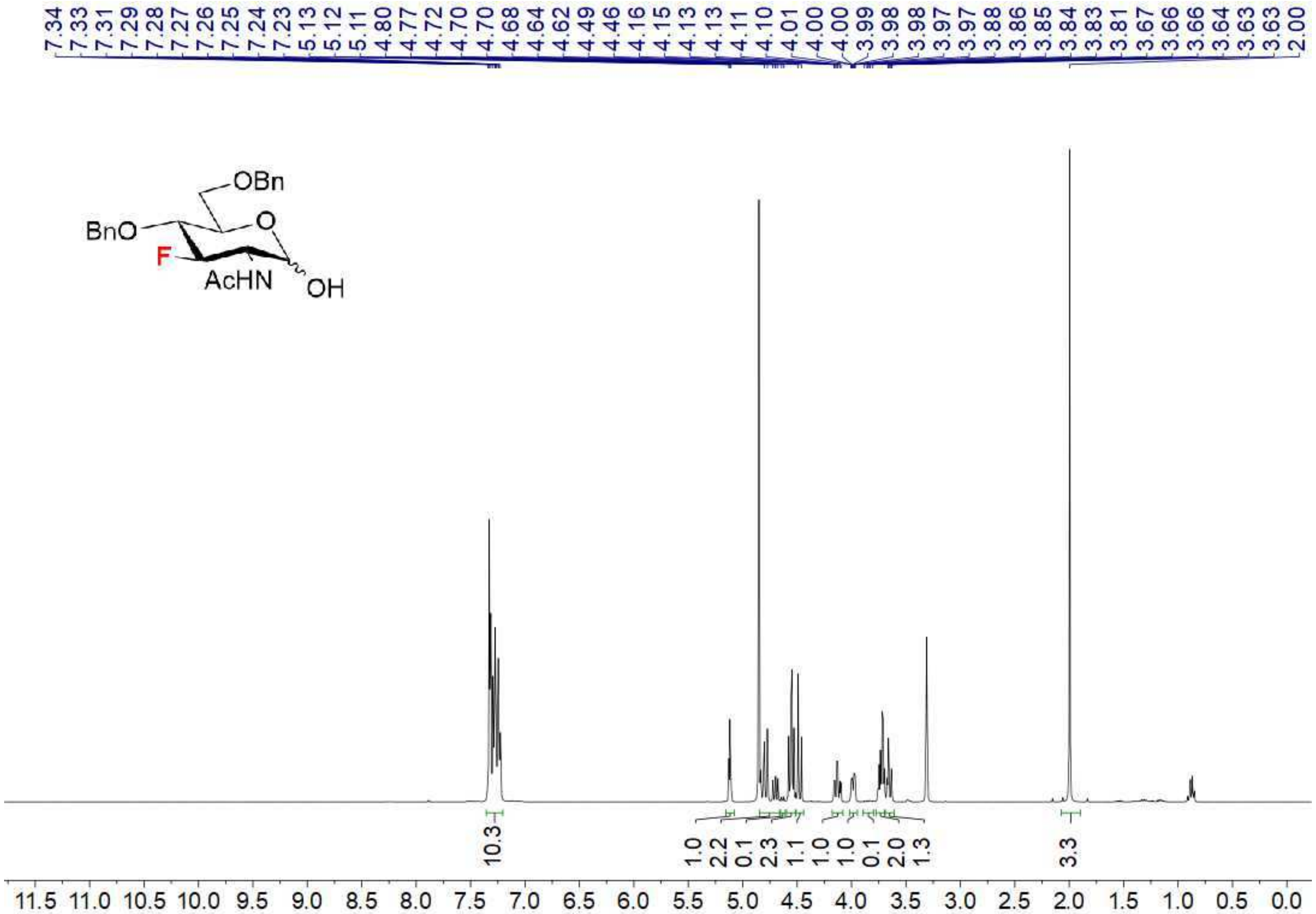
F
OBn
O
F
N3
OH
5.5 5.4 5.3 5.2 5.1 5.0 4.9 4.8 4.7 4.6 4.5 4.4 4.3 4.2 4.1 4.0 3.9 3.8 3.7 3.6
f2 (ppm)
3.5 3.7 3.9 4.1 4.3 4.5 4.7 4.9 5.1 5.3 5.5
f1 (ppm)

^{1}H-^{13}C HSQC 48

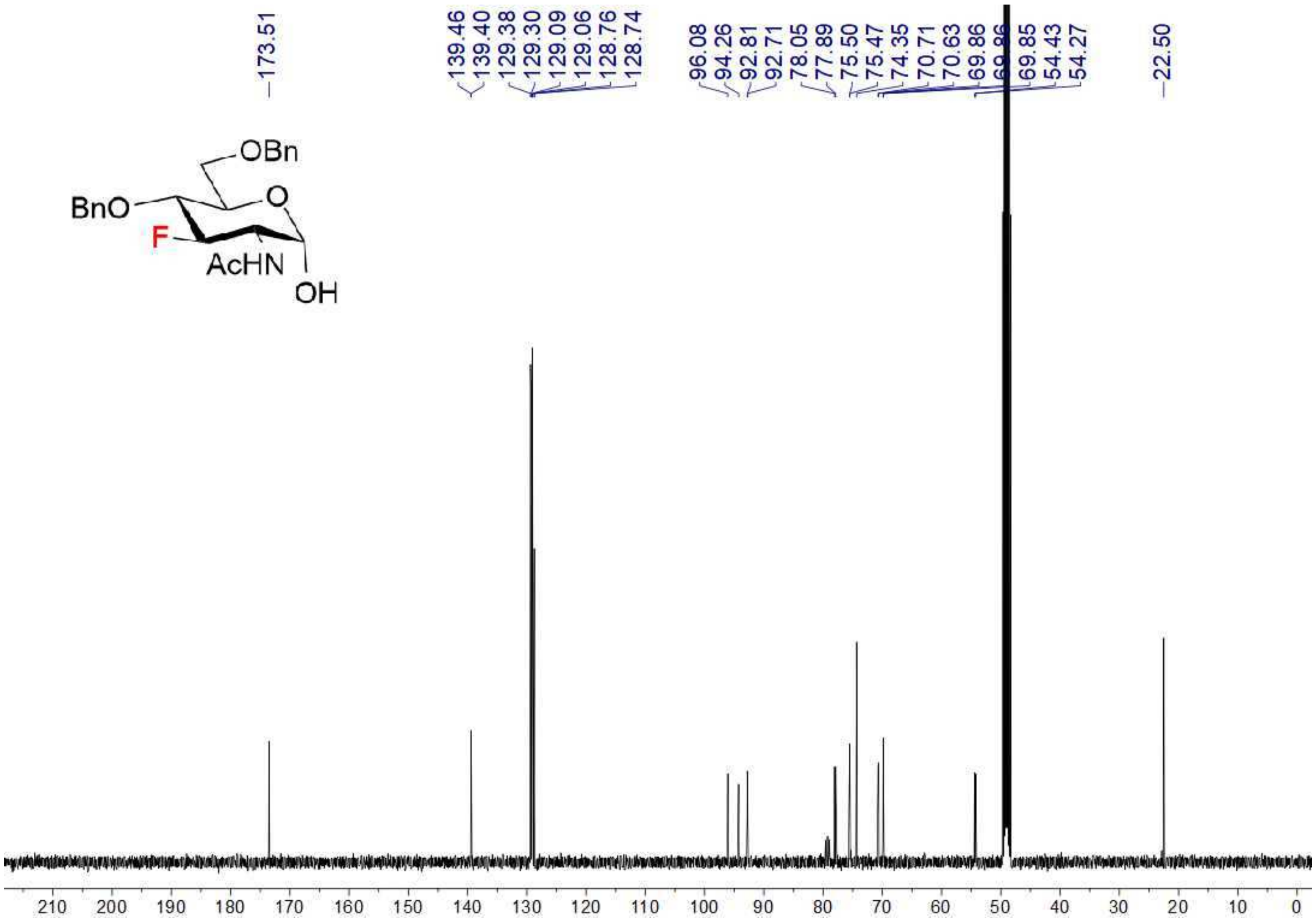


NMR COMPOUND 49

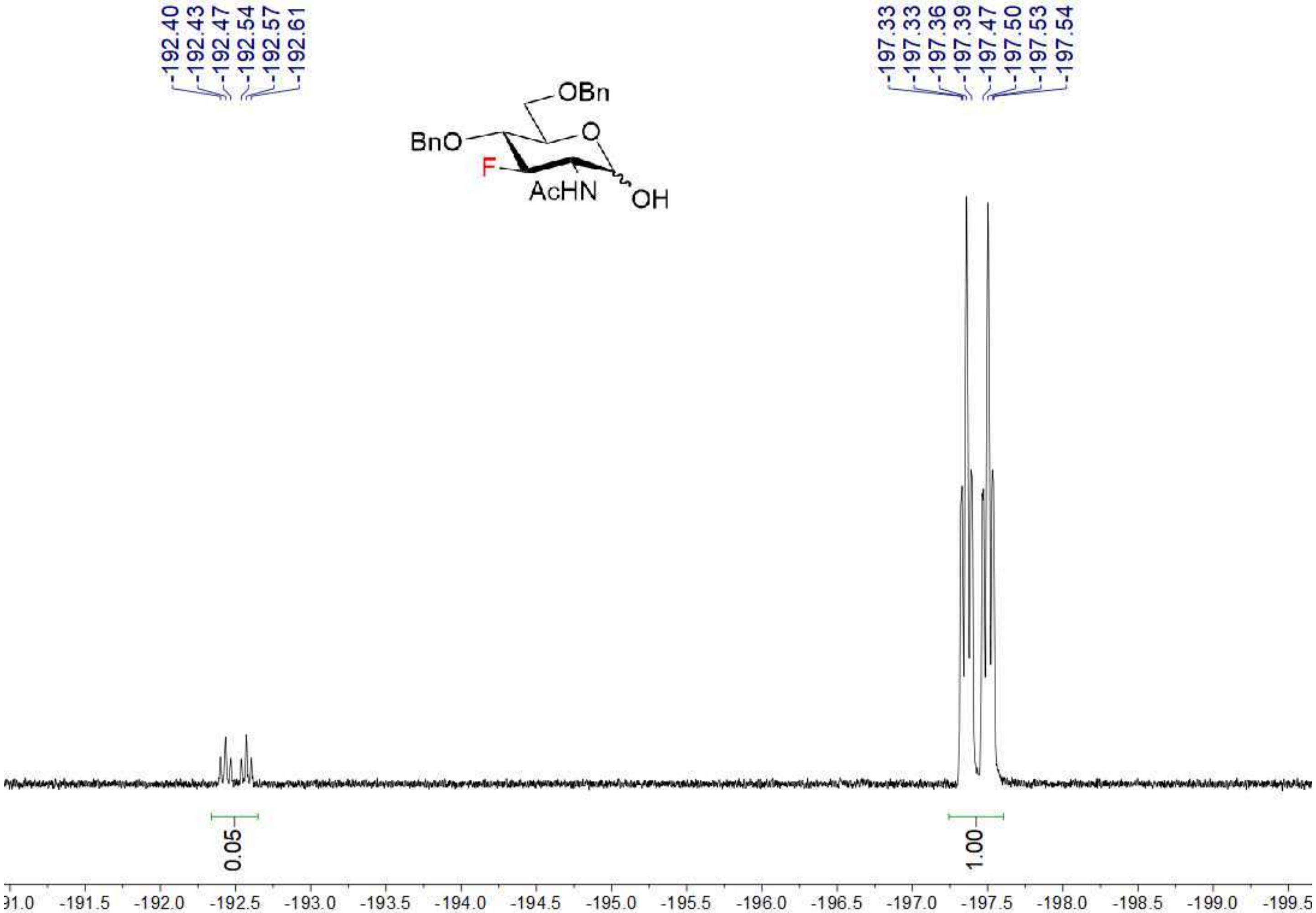
^{1}H NMR (400 MHz, CD_3OD) 49 (ca. 5% β-anomer)



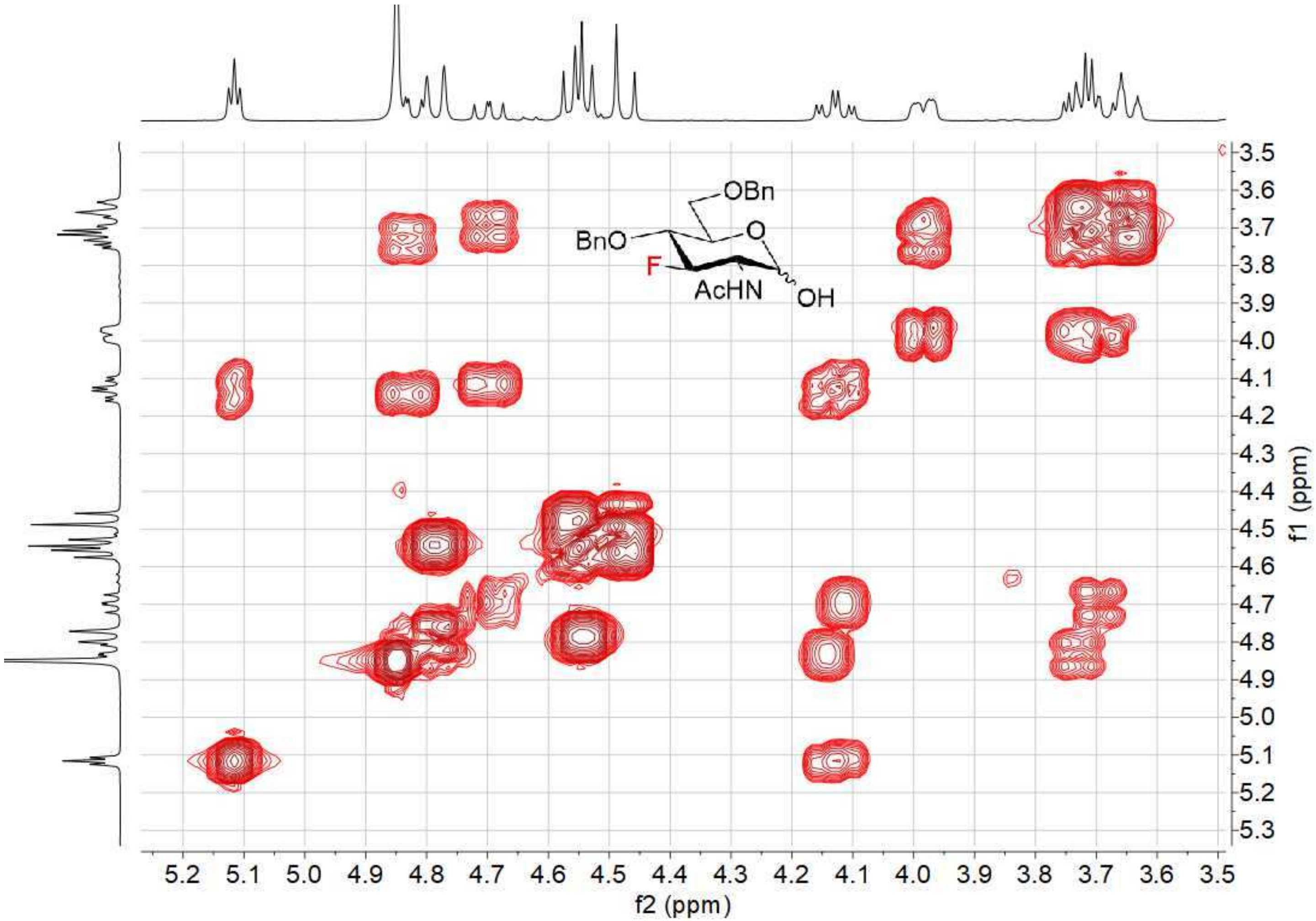
^{13}C NMR (100 MHz, CD_3OD) 49



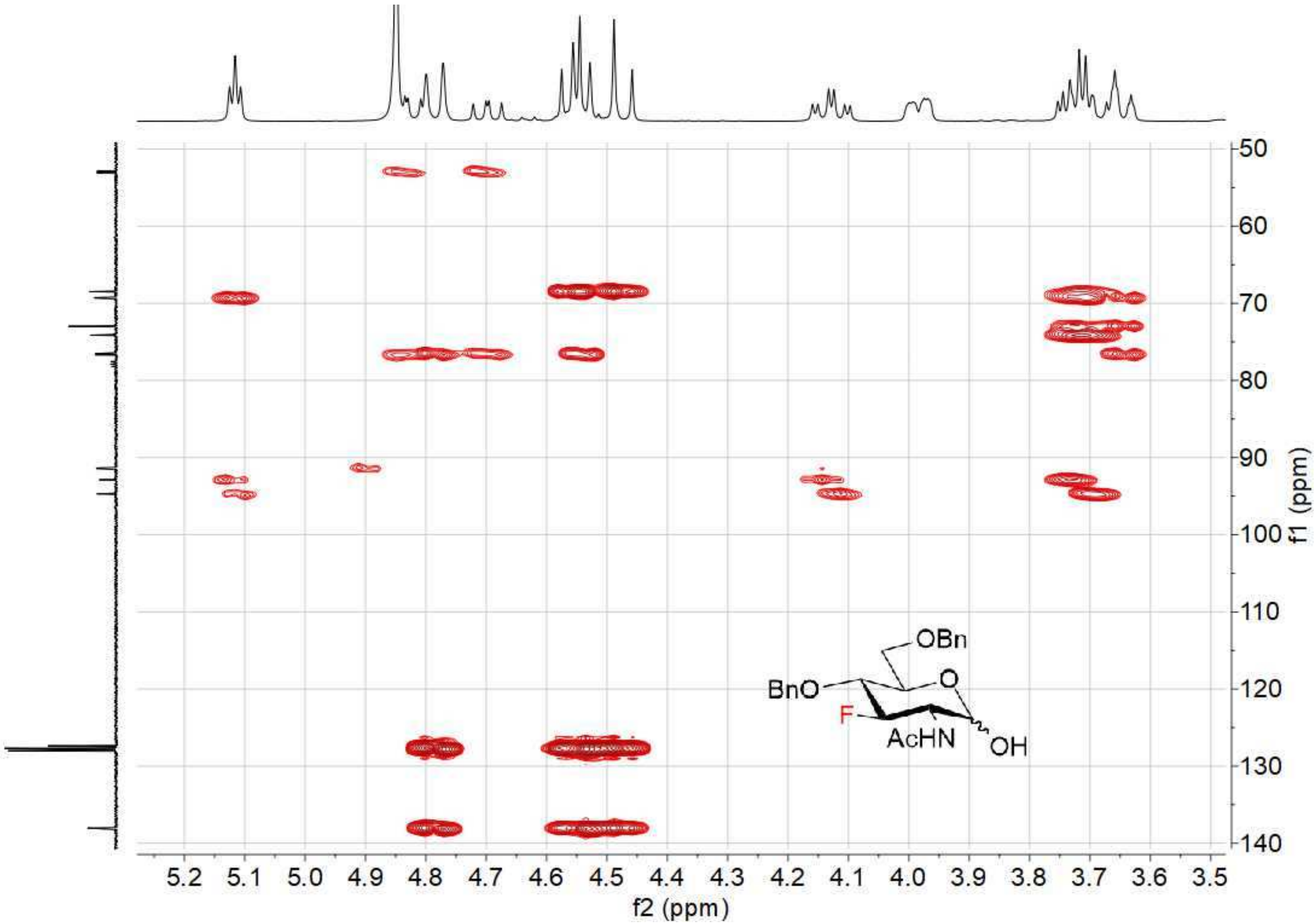
[19]F NMR (376 MHz, CD$_3$OD) 49 (ca. 5% β-anomer)



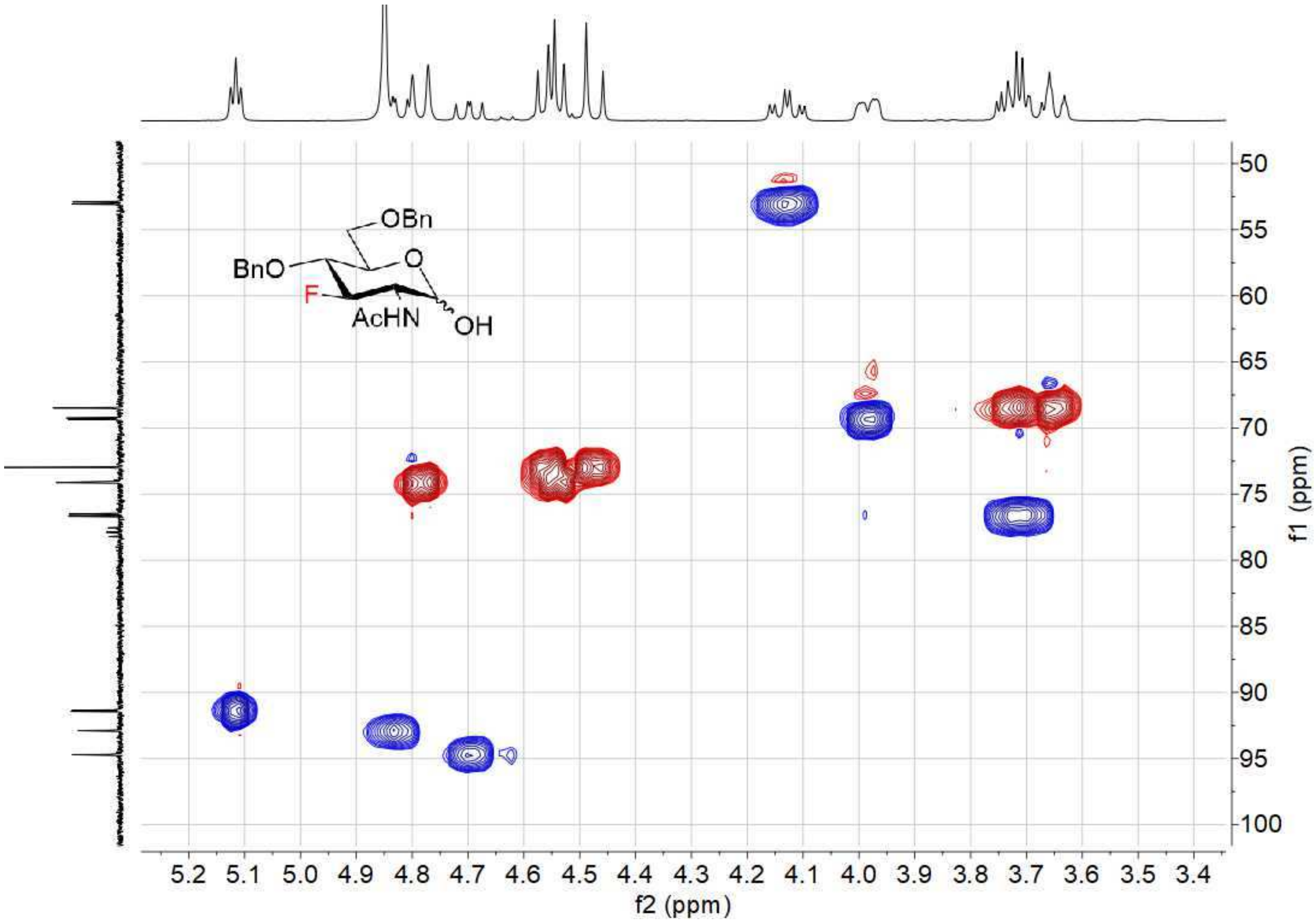
^{1}H-^{1}H COSY 49



^{1}H-^{13}C HMBC 49

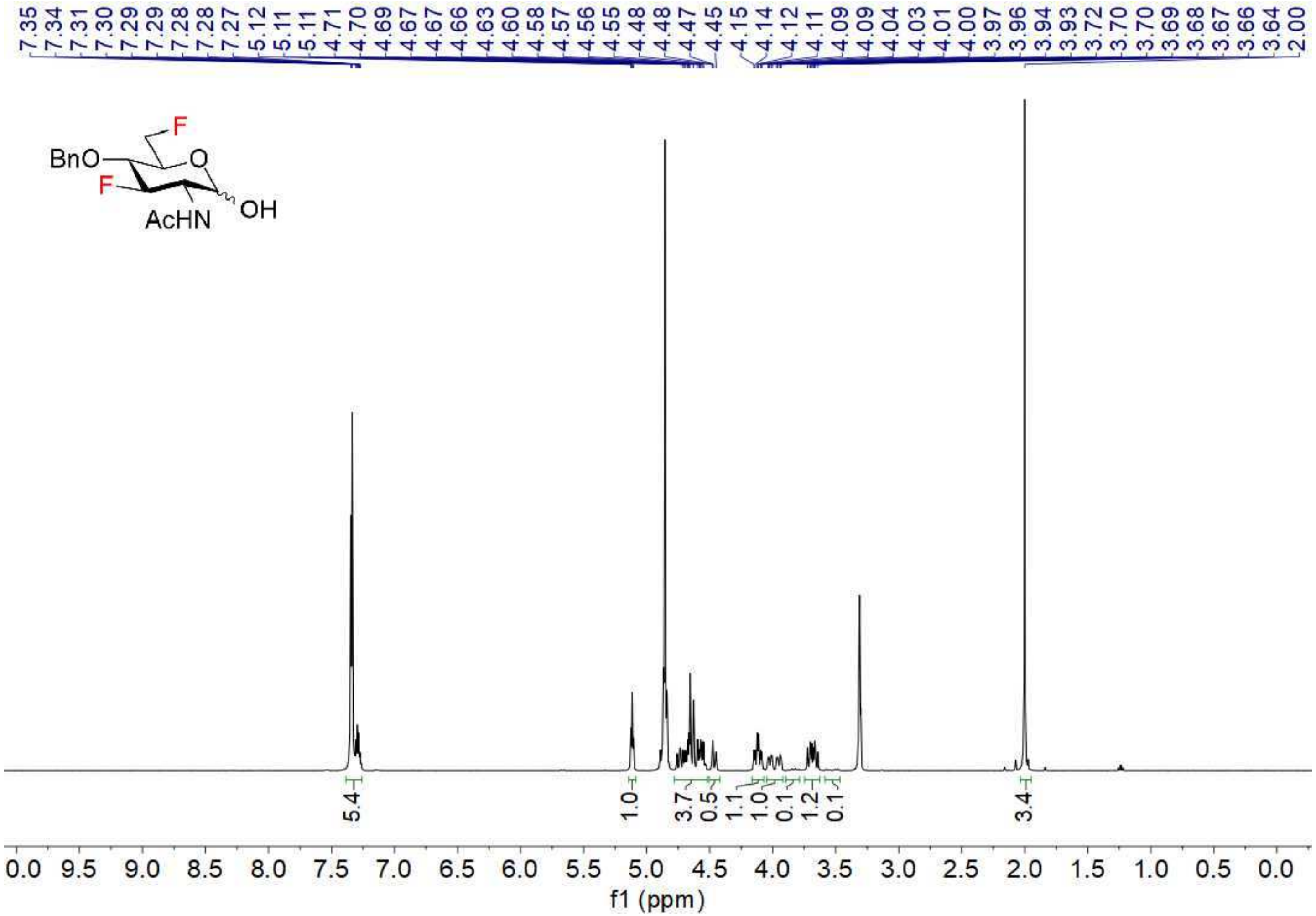


^{1}H-^{13}C HSQC 49

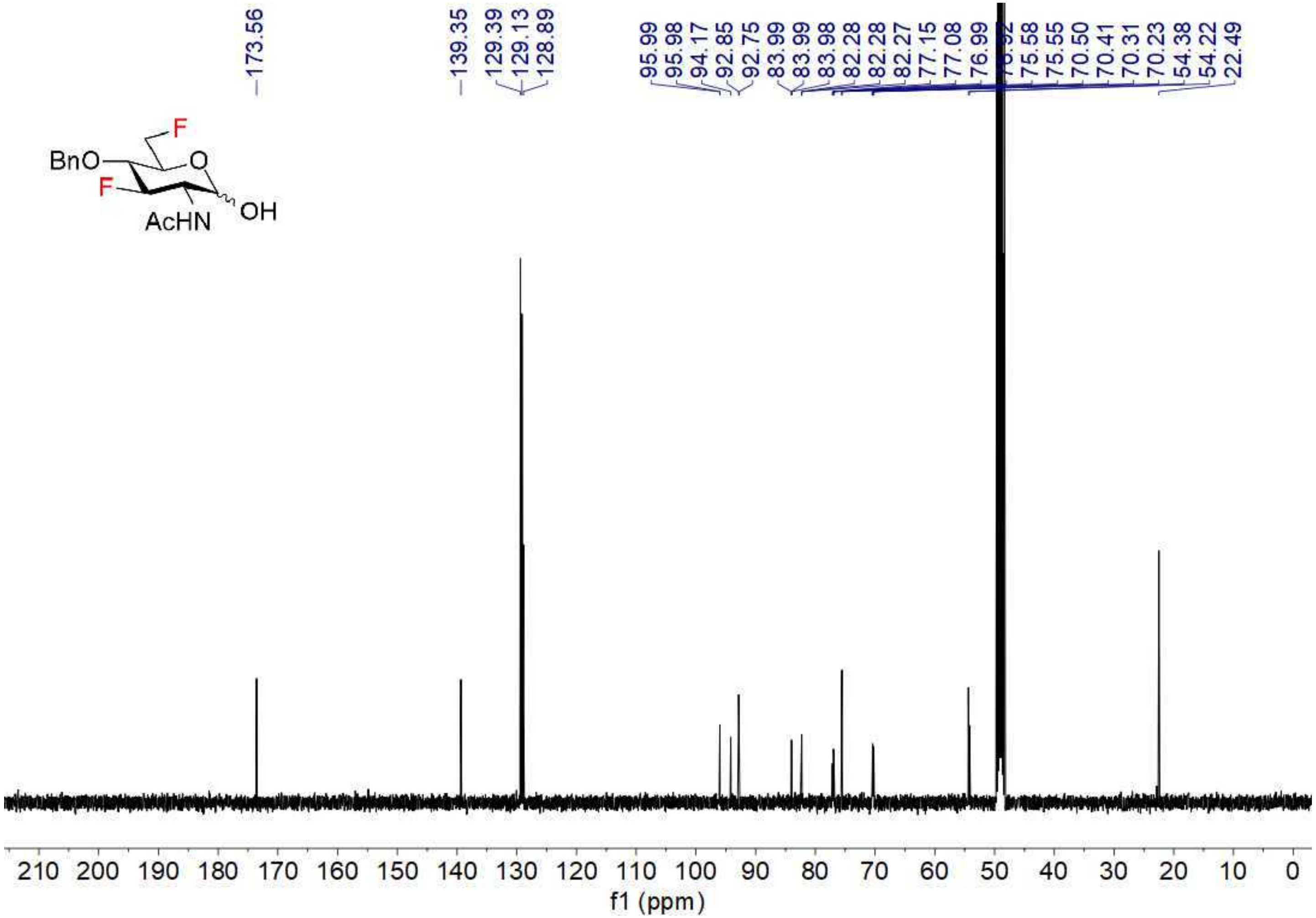


NMR COMPOUND 50

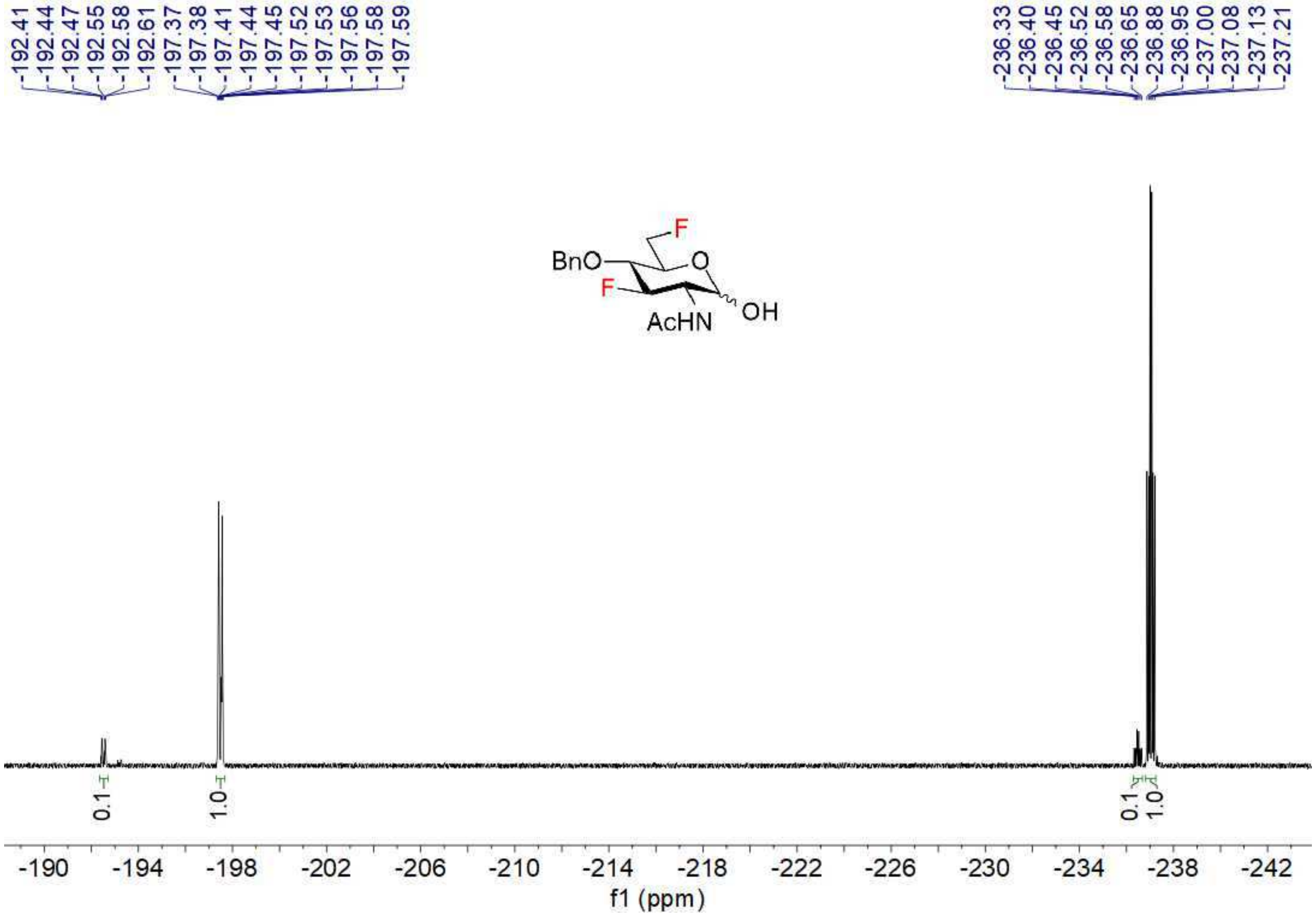
^{1}H NMR (400 MHz, CD_3OD) 50 (α/β ca. 10/1)



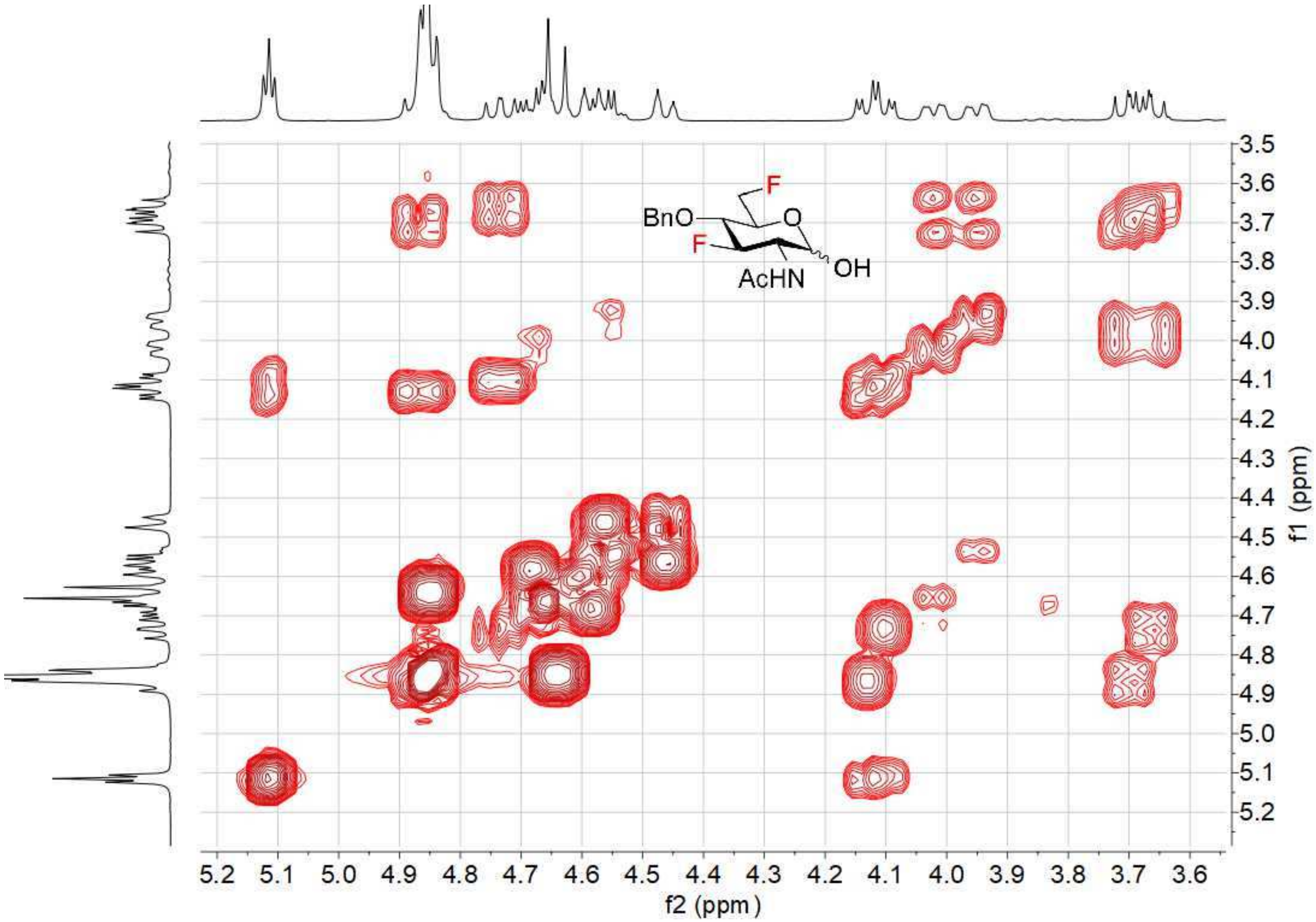
^{13}C NMR (100 MHz, CD_3OD) 50



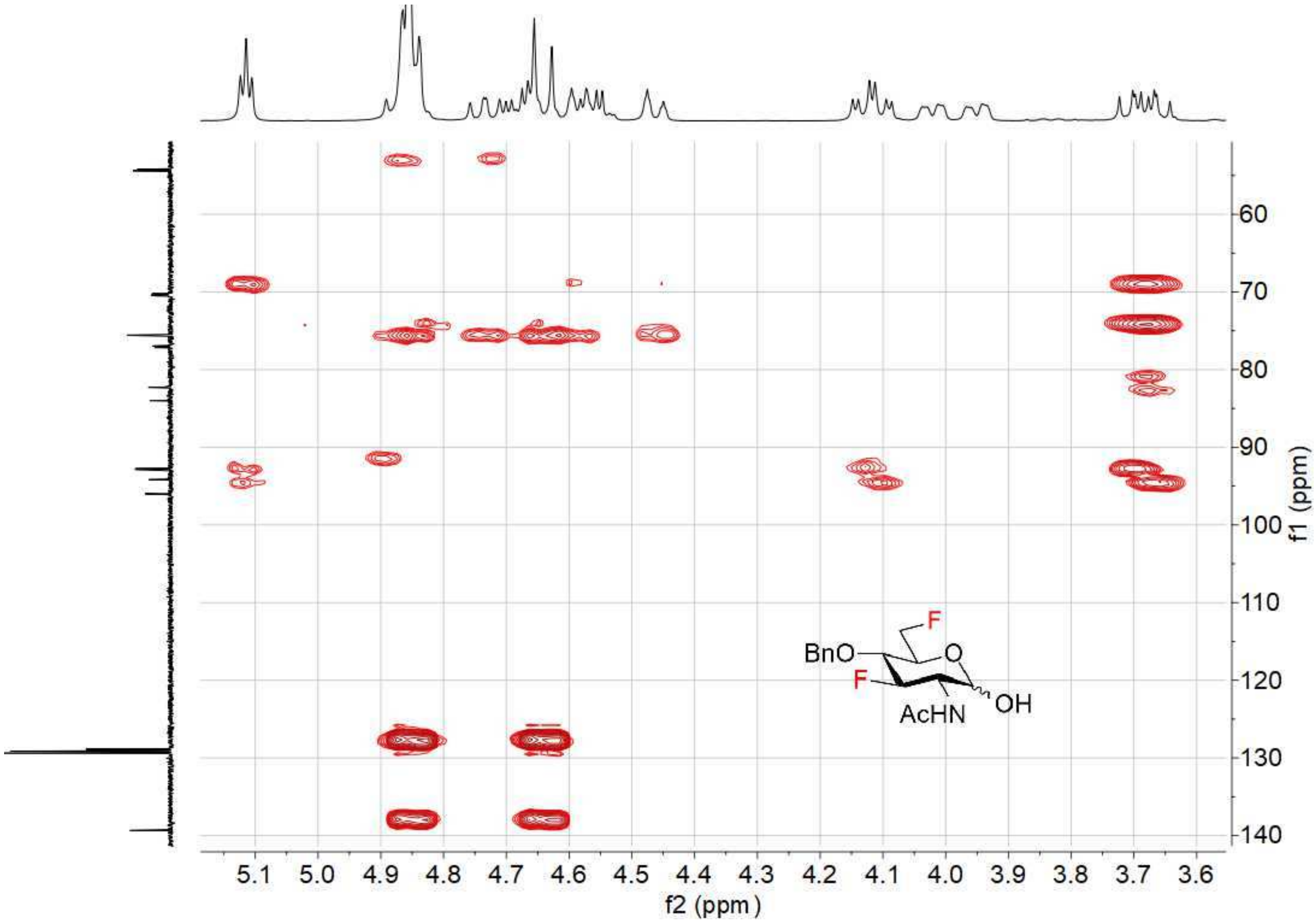
^{19}F NMR (376 MHz, CD_3OD) 50 (α/β ca. 10/1)



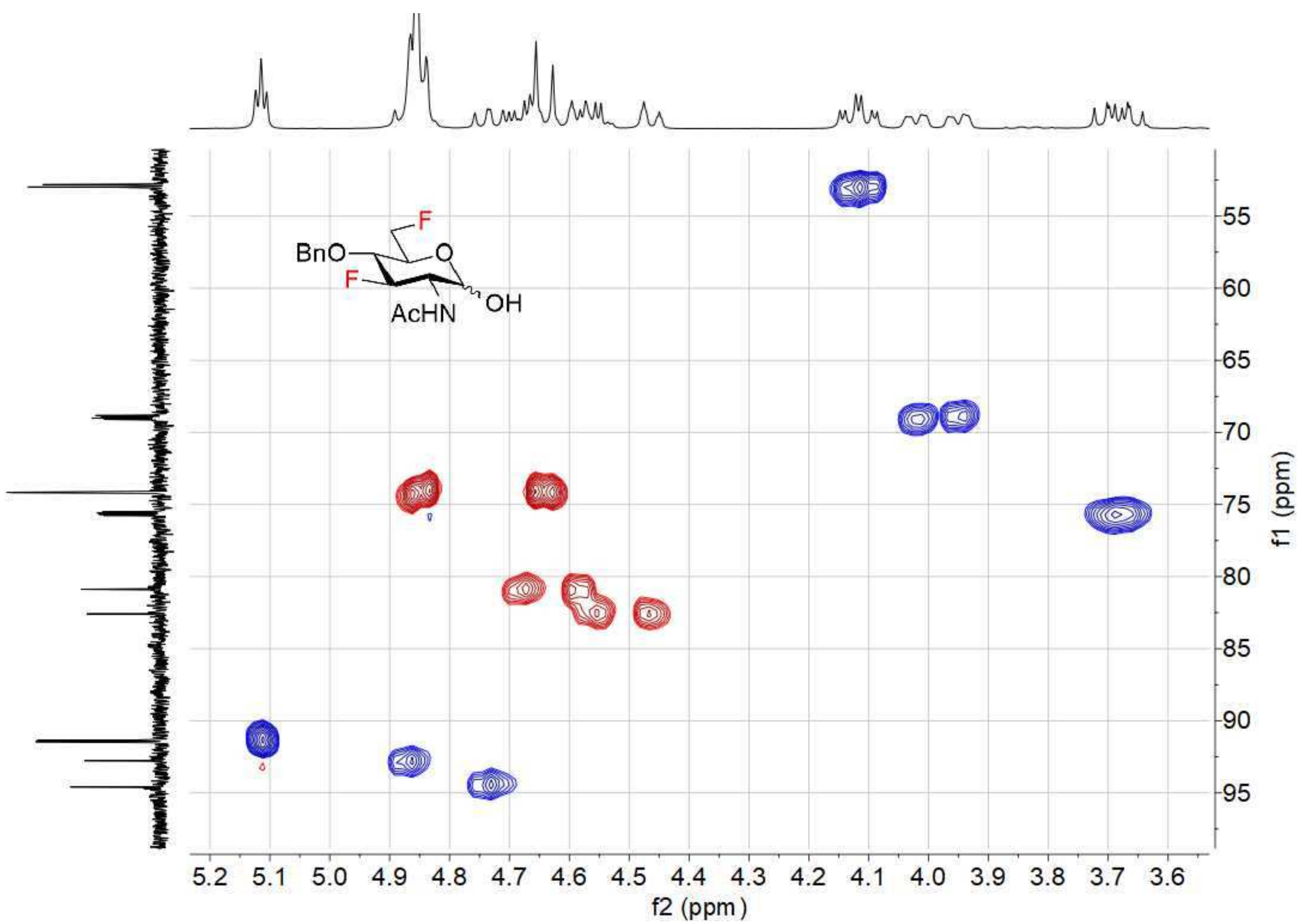
^{1}H-^{1}H COSY 50



^{1}H-^{13}C HMBC 50

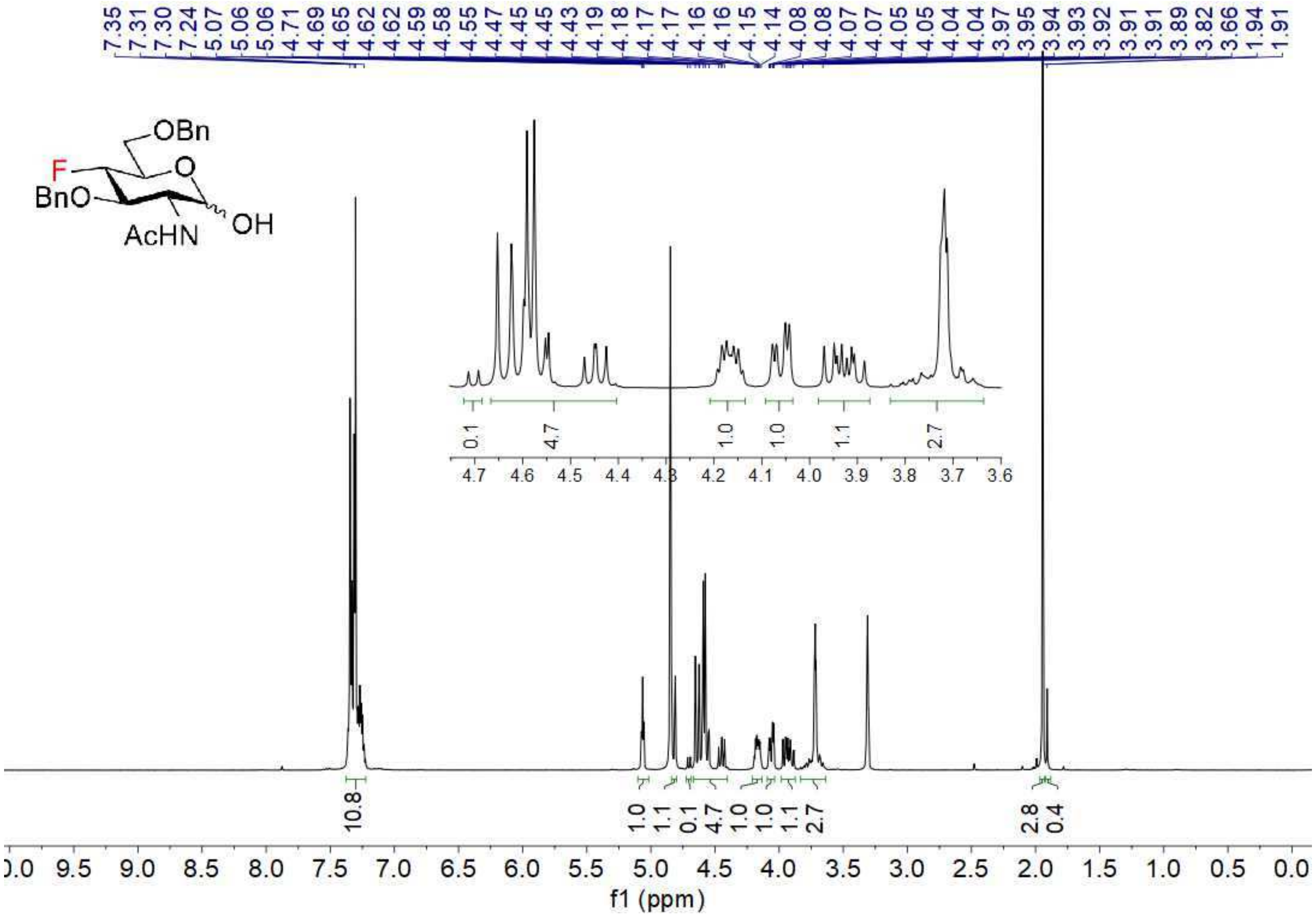


^{1}H-^{13}C HSQC 50

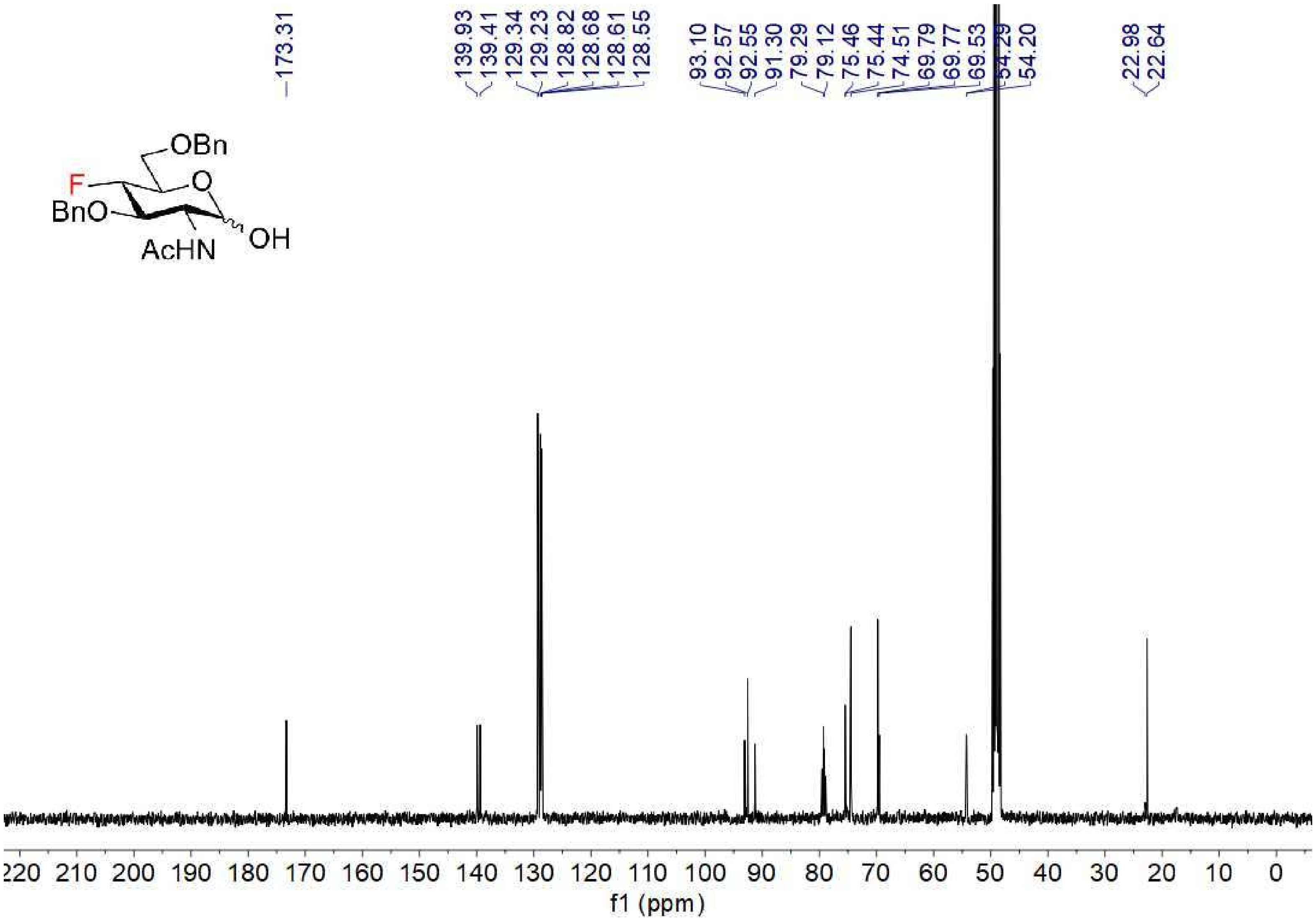


NMR COMPOUND 51

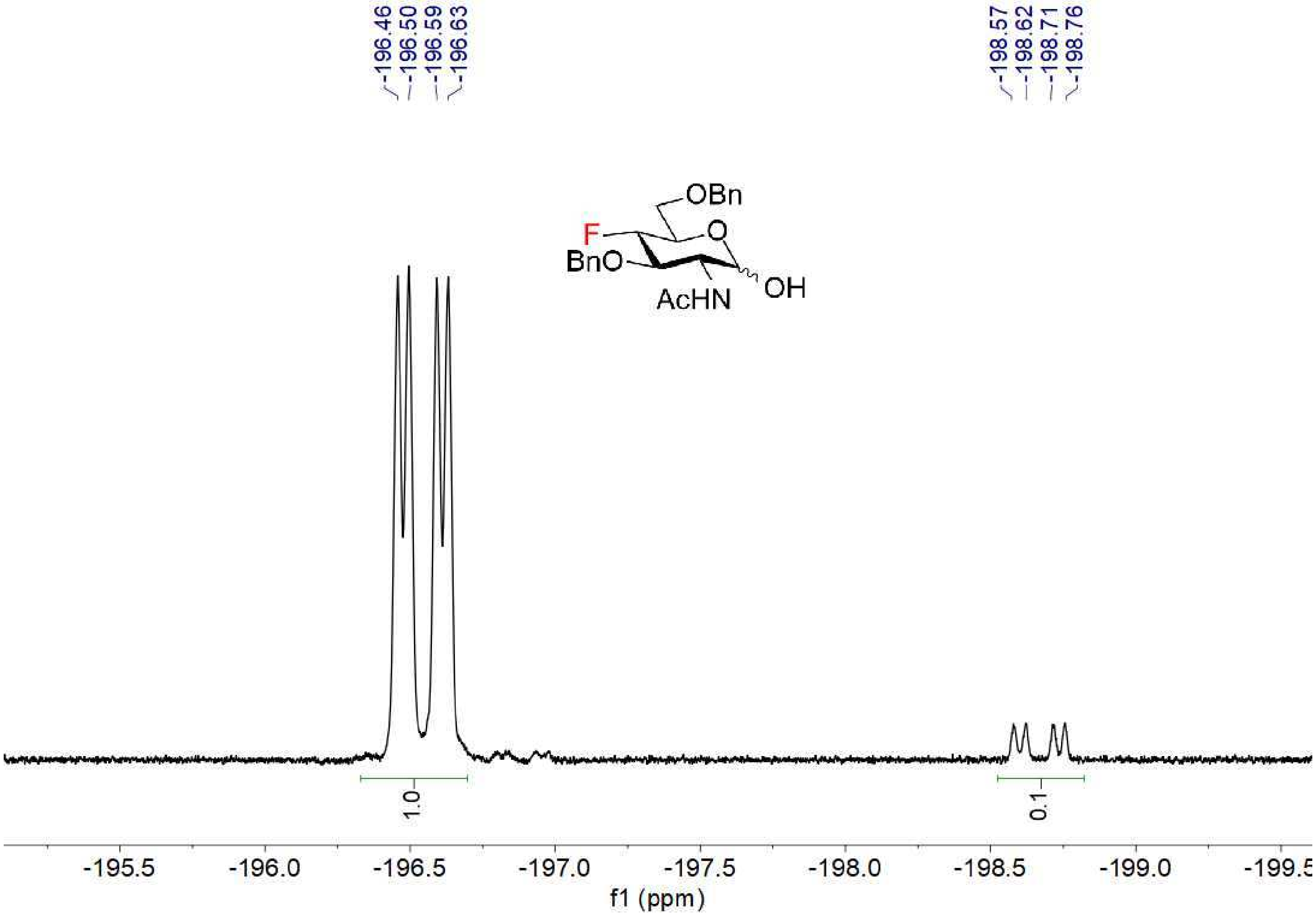
^{1}H NMR (400 MHz, CD_3OD) 51 (α/β ca. 10/1)



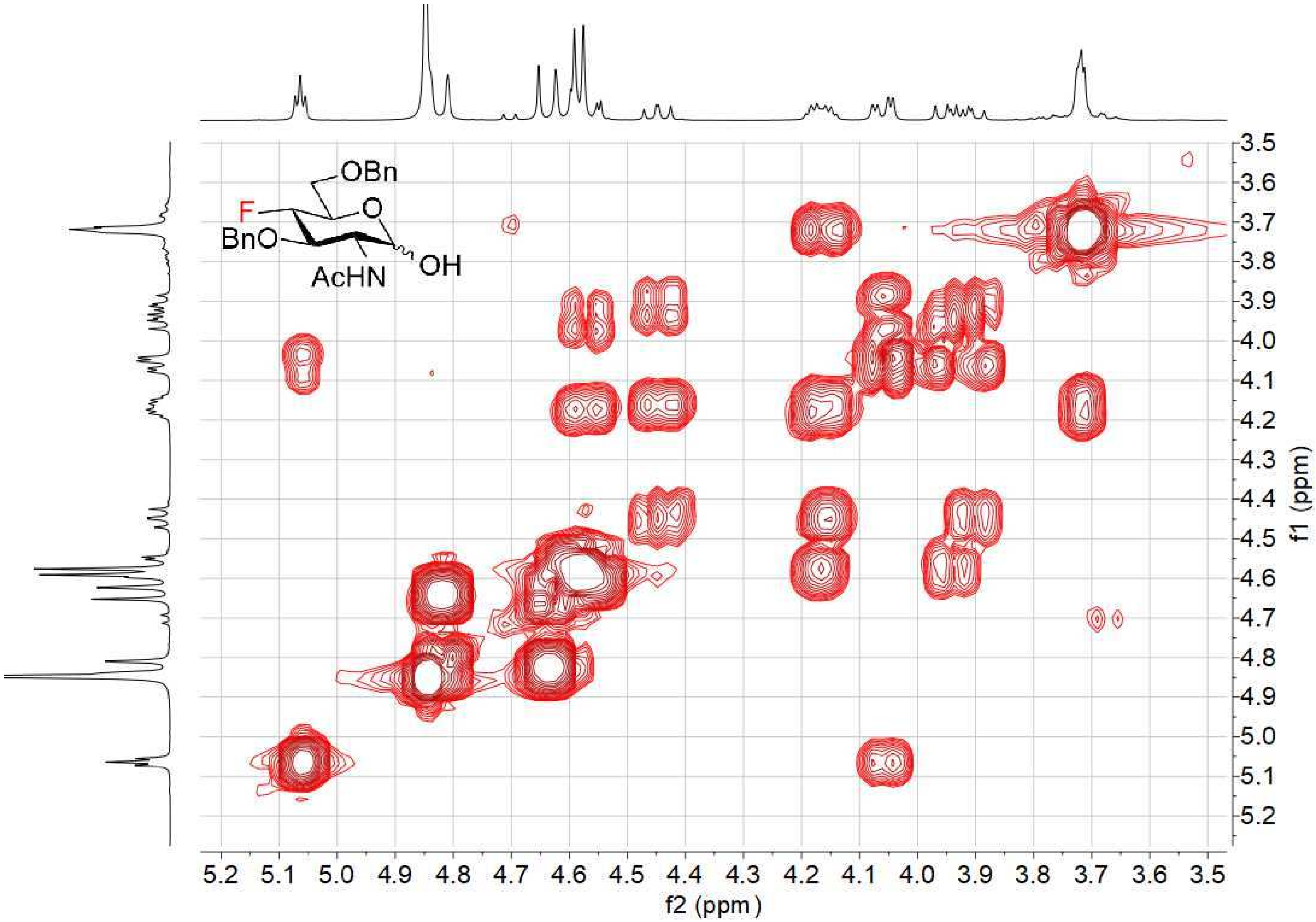
^{13}C NMR (100 MHz, CD_3OD) 51



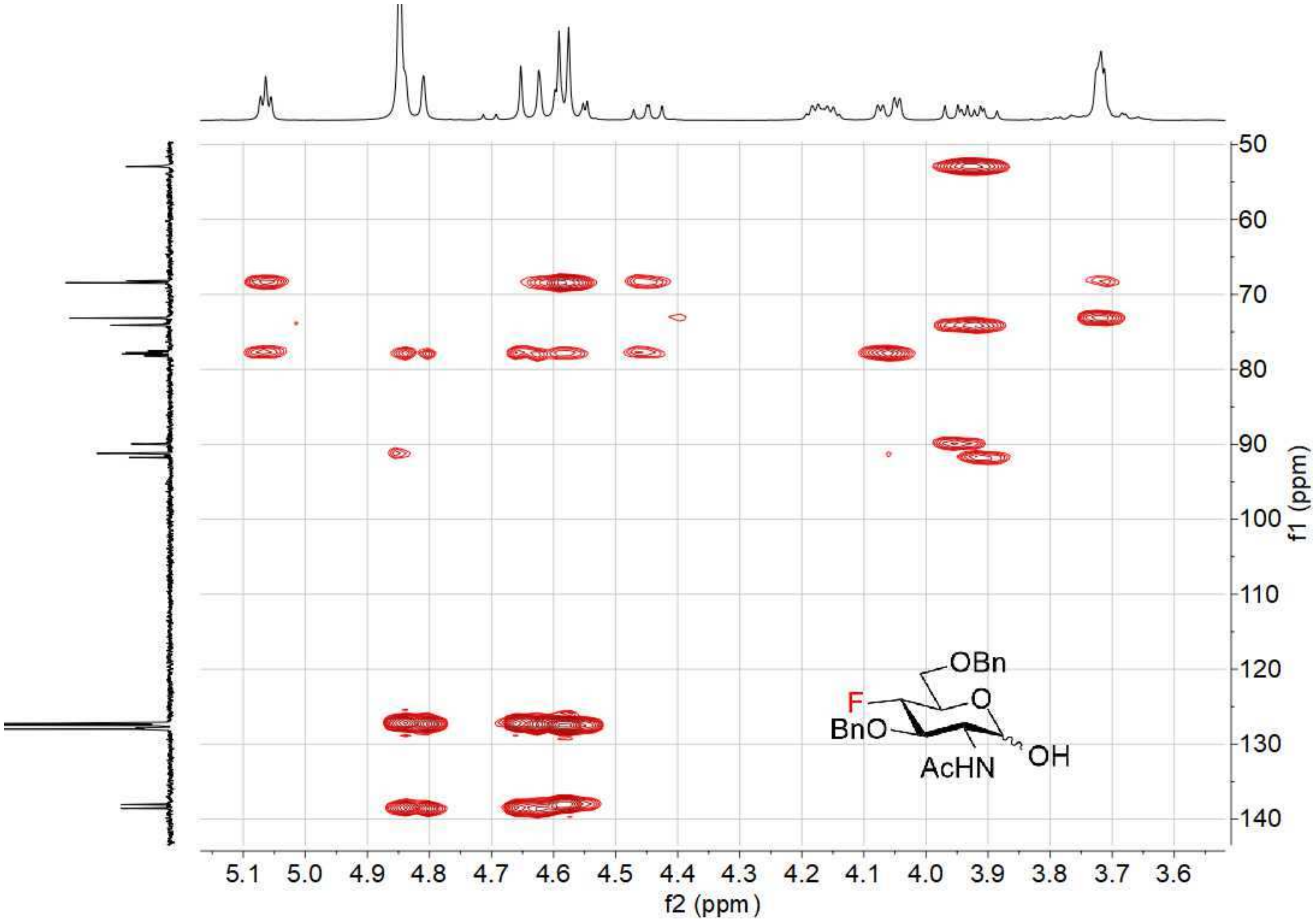
^{19}F NMR (376 MHz, CD_3OD) 51 (α/β ca. 10/1)



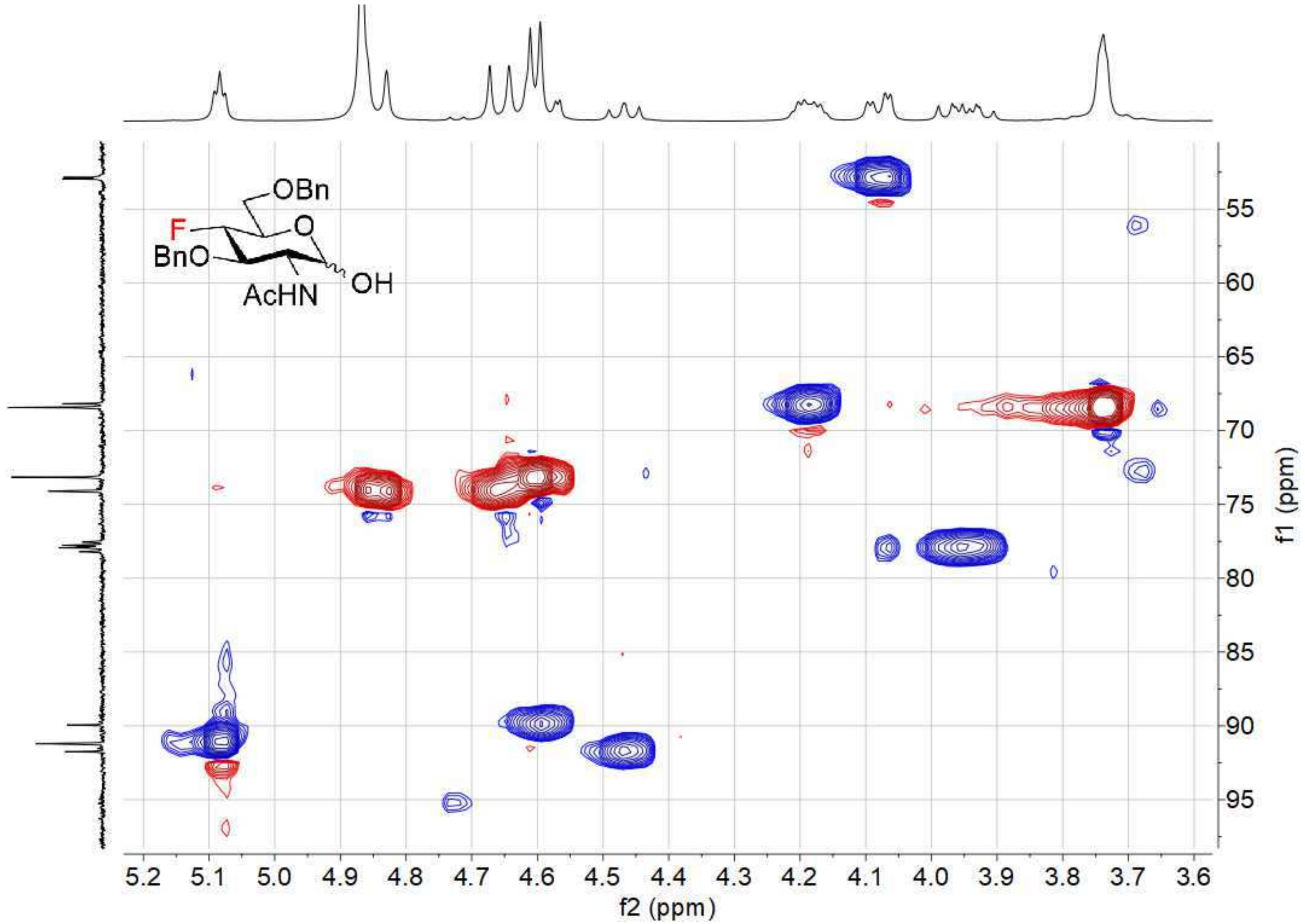
[1]H-[1]H COSY 51



^{1}H-^{13}C HMBC 51

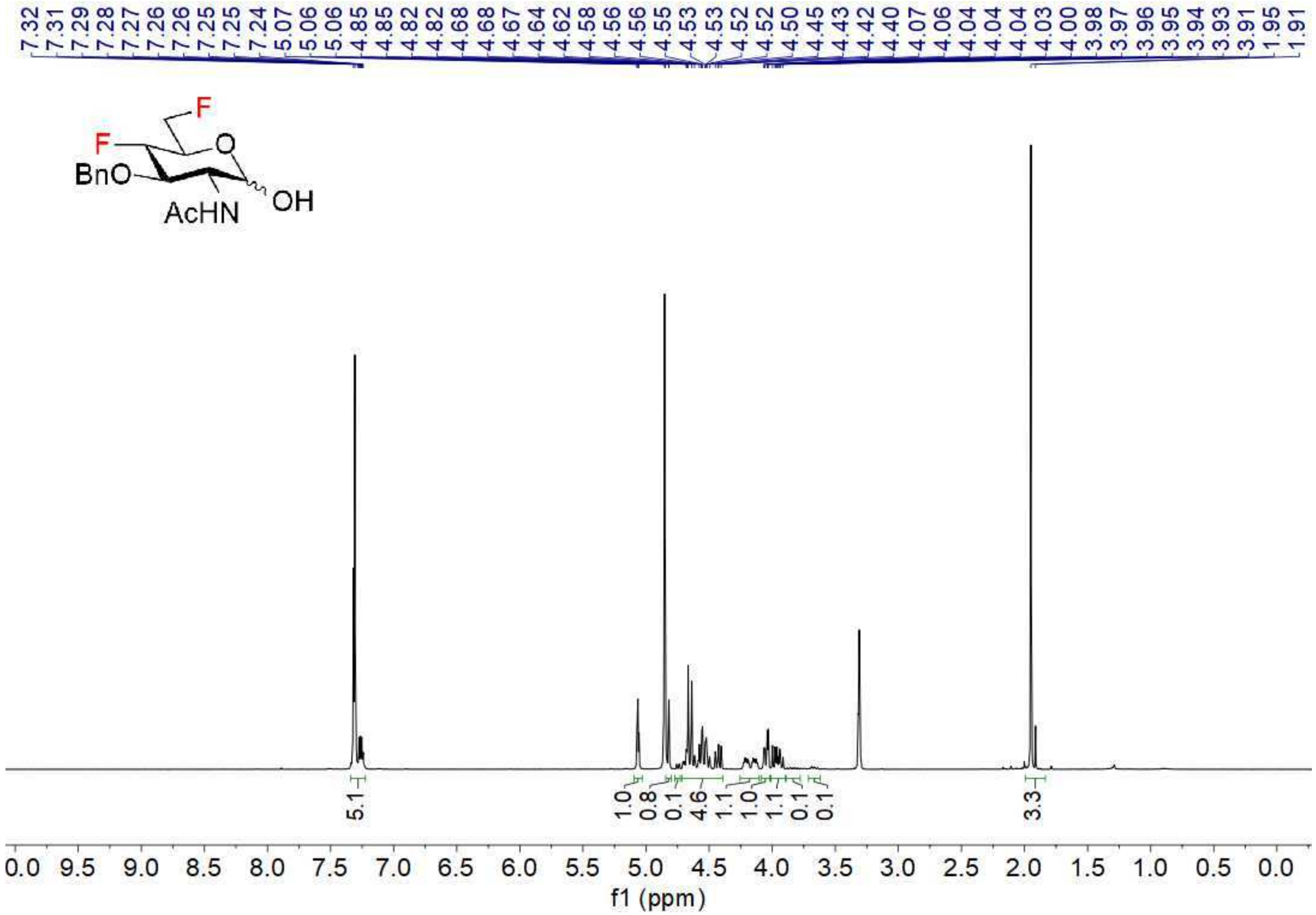


^{1}H-^{13}C HSQC 51

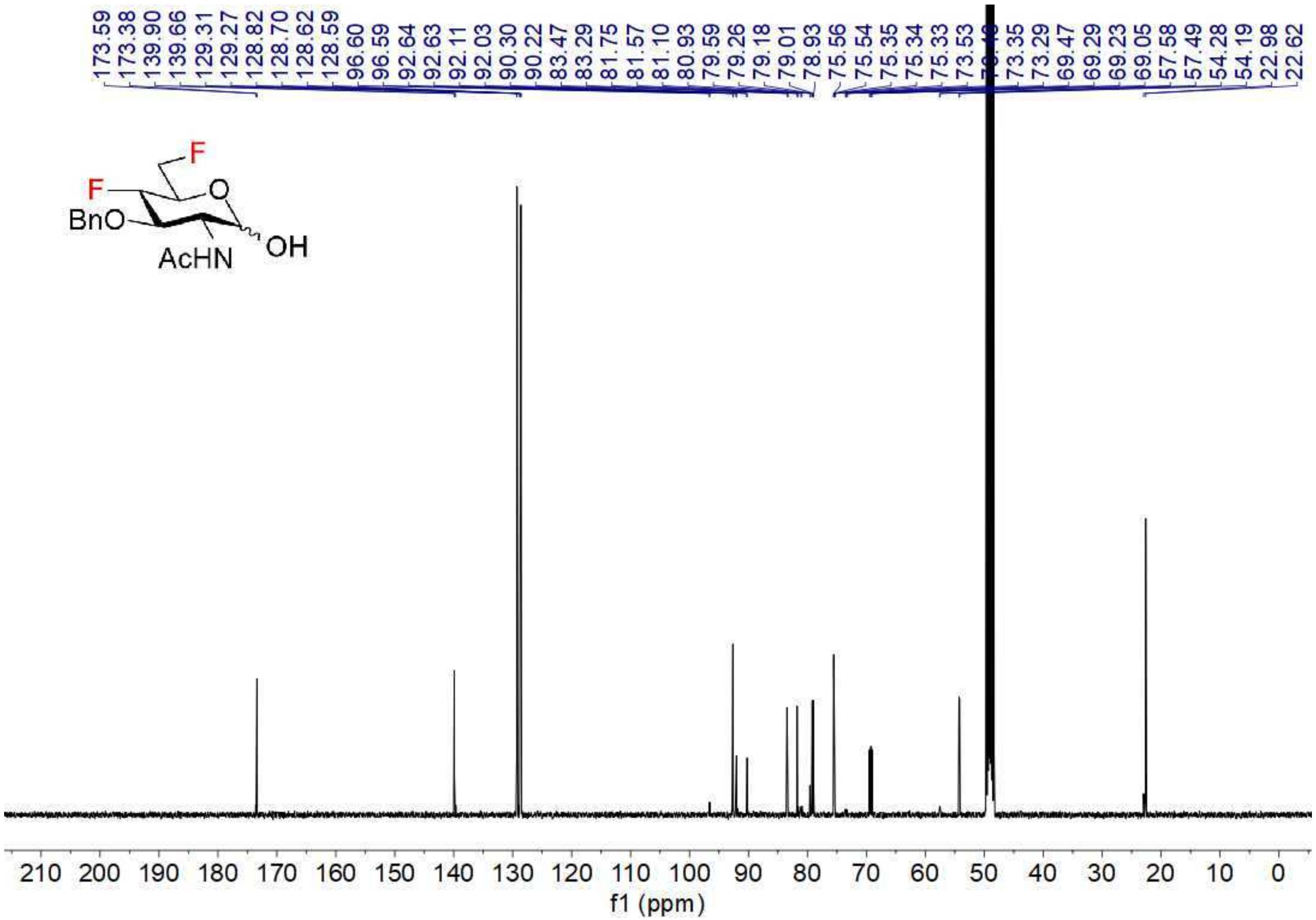


NMR COMPOUND 52

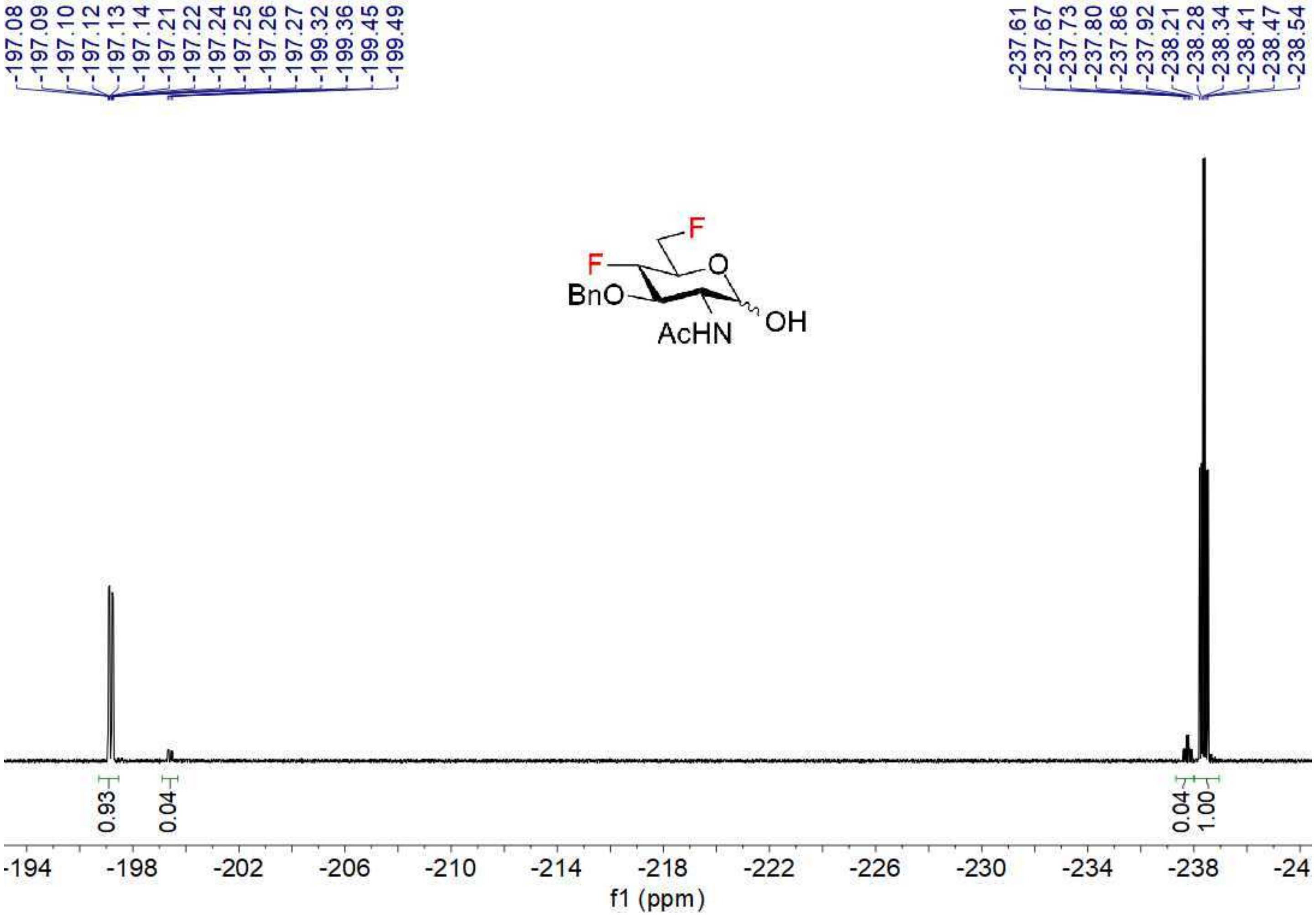
[1]H NMR (400 MHz, CD_3OD) 52 (ca. 4% β-anomer)



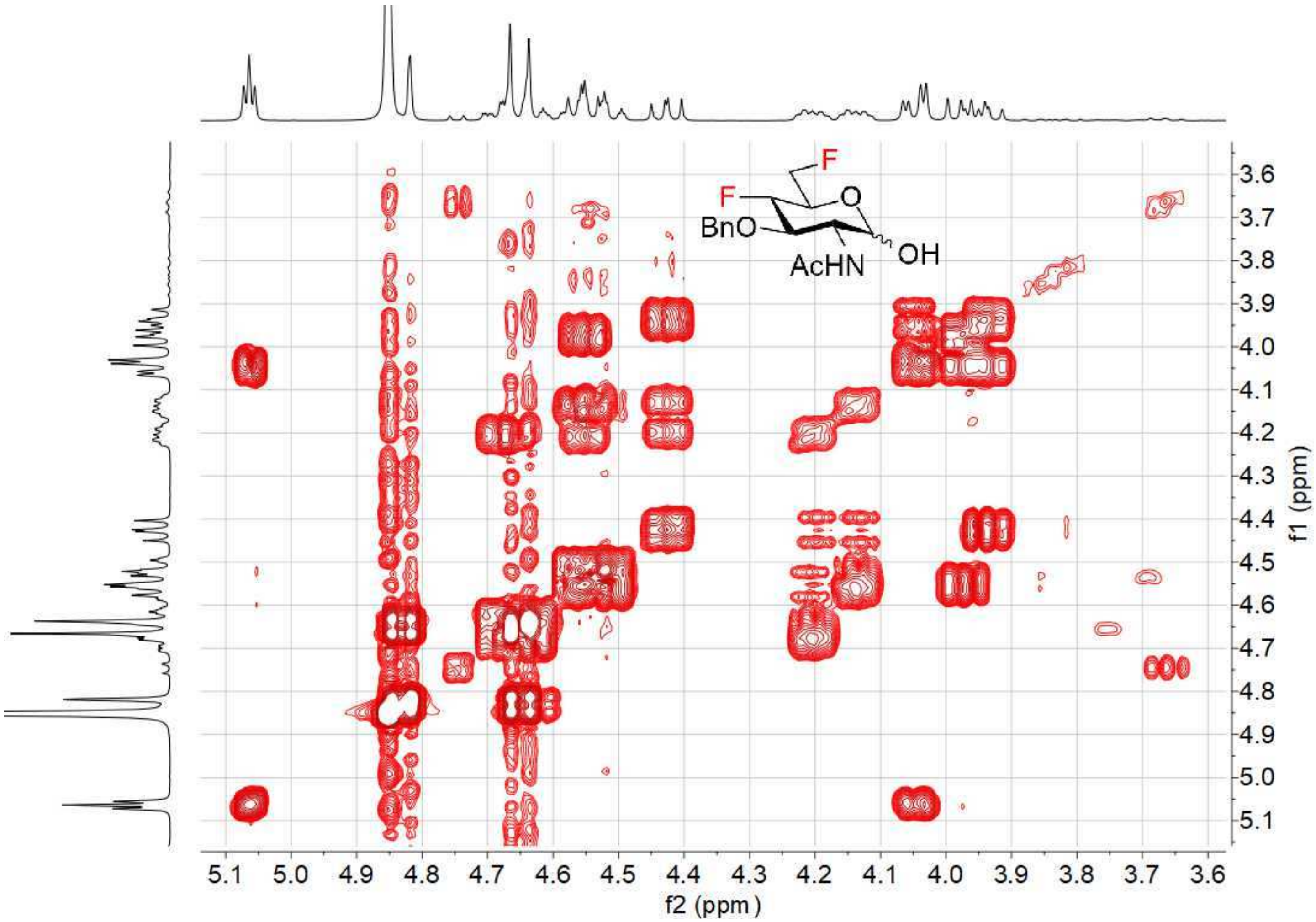
^{13}C NMR (100 MHz, CD_3OD) 52



^{19}F NMR (376 MHz, CD_3OD) 52 (ca. 4% β-anomer)

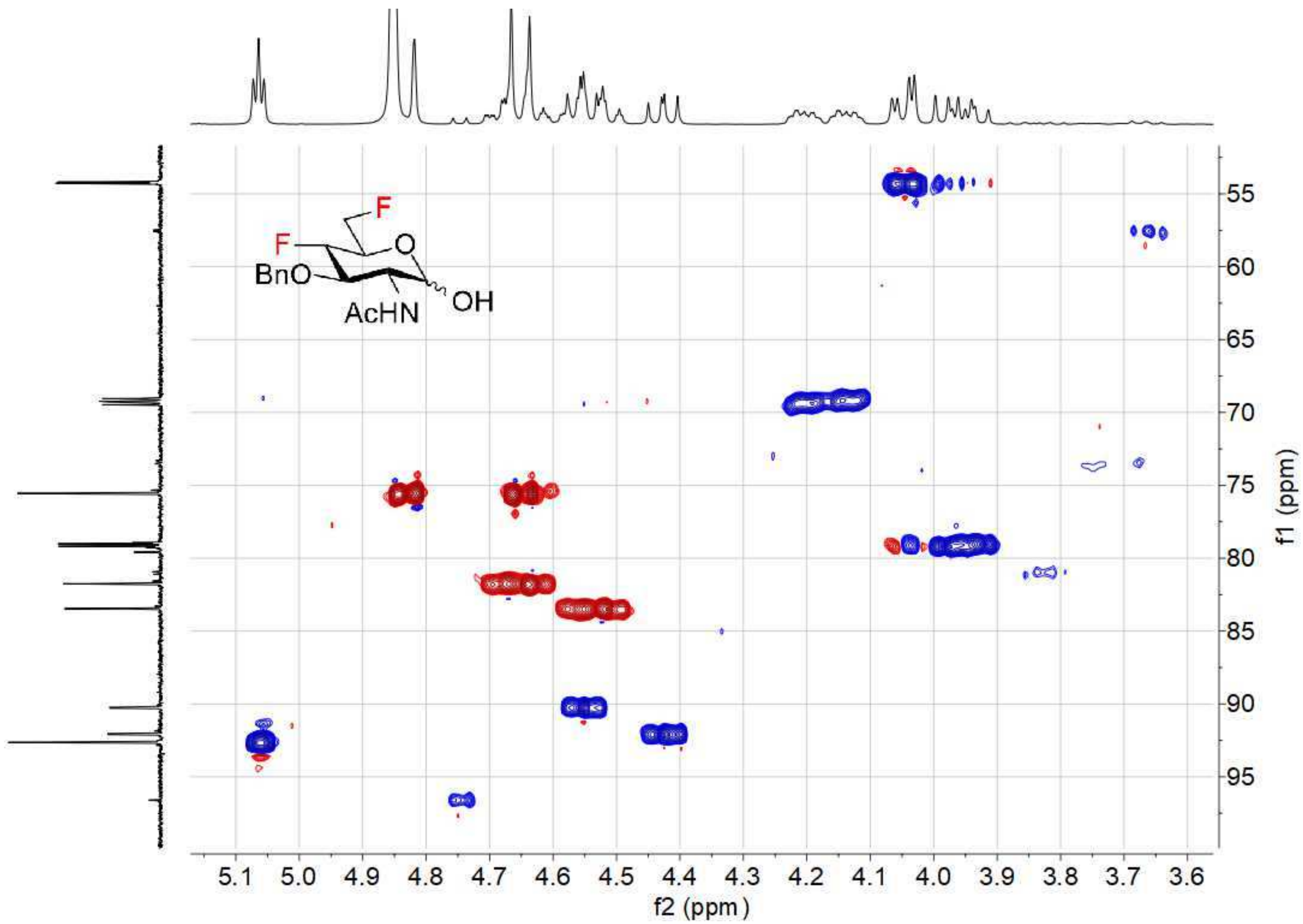


^{1}H-^{1}H COSY 52



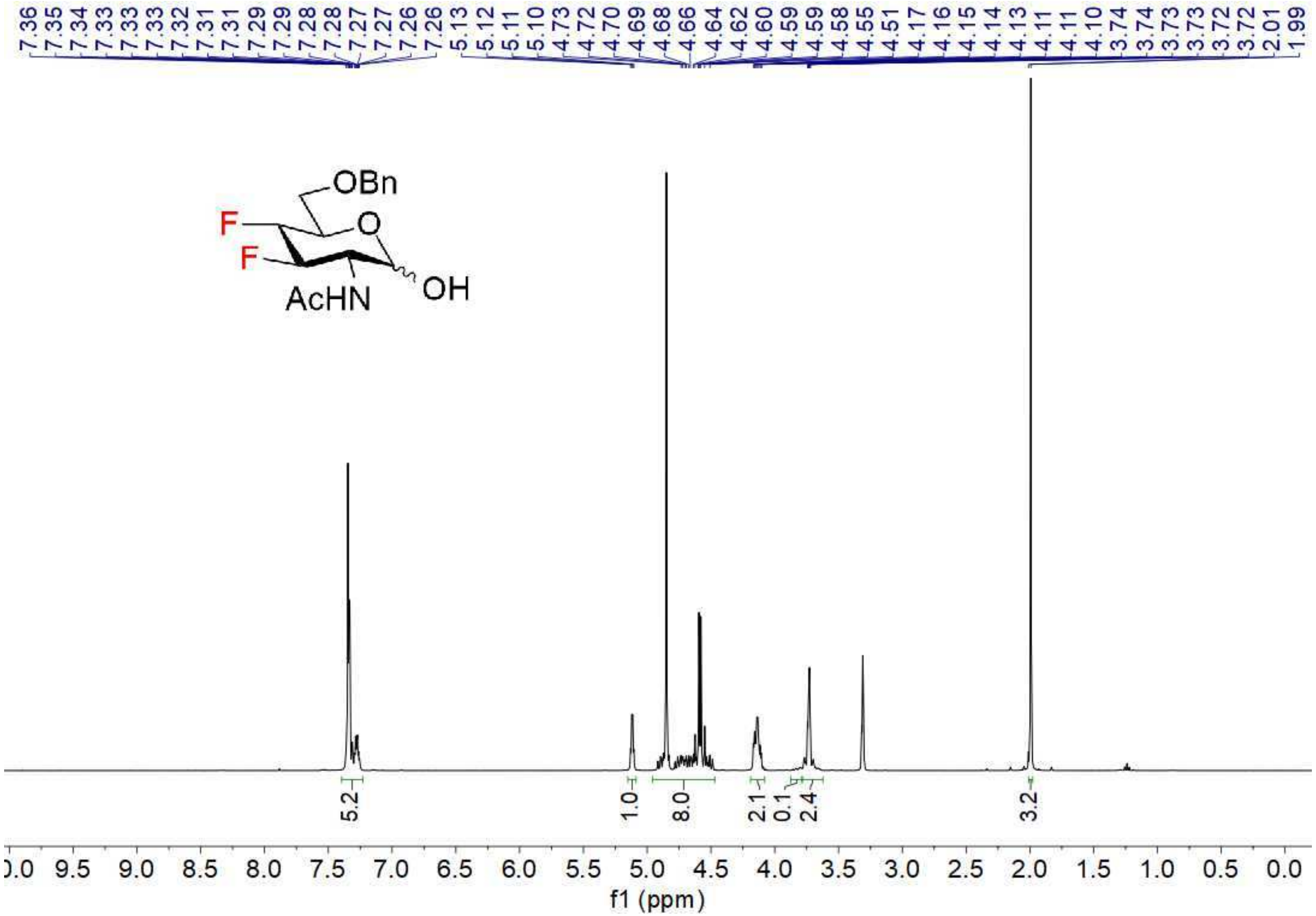
F
F
O
BnO
OH
AcHN
5.1 5.0 4.9 4.8 4.7 4.6 4.5 4.4 4.3 4.2 4.1 4.0 3.9 3.8 3.7 3.6
f2 (ppm)
3.6 3.7 3.8 3.9 4.0 4.1 4.2 4.3 4.4 4.5 4.6 4.7 4.8 4.9 5.0 5.1
f1 (ppm)

^{1}H-^{13}C HSQC 52

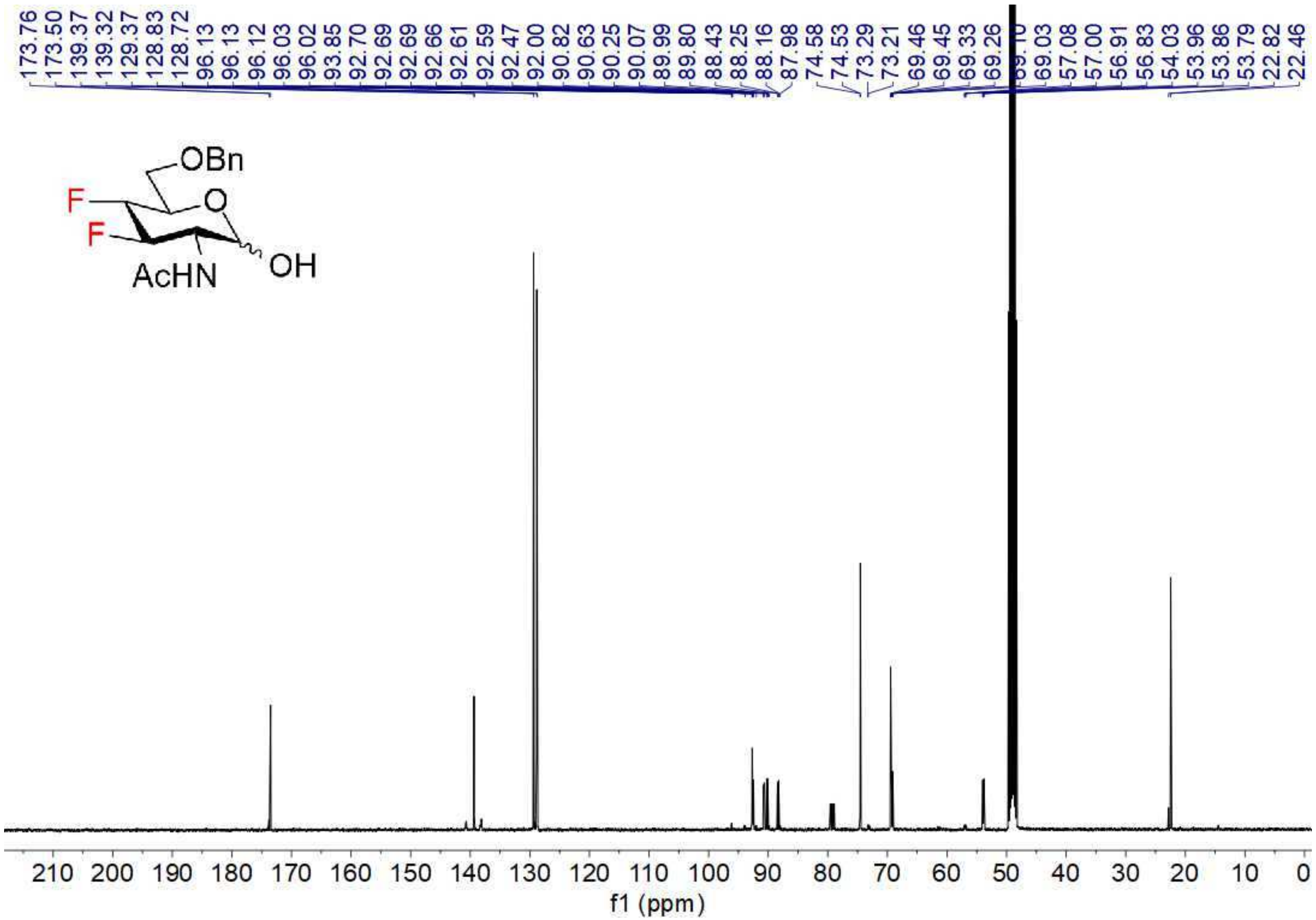


NMR COMPOUND 53

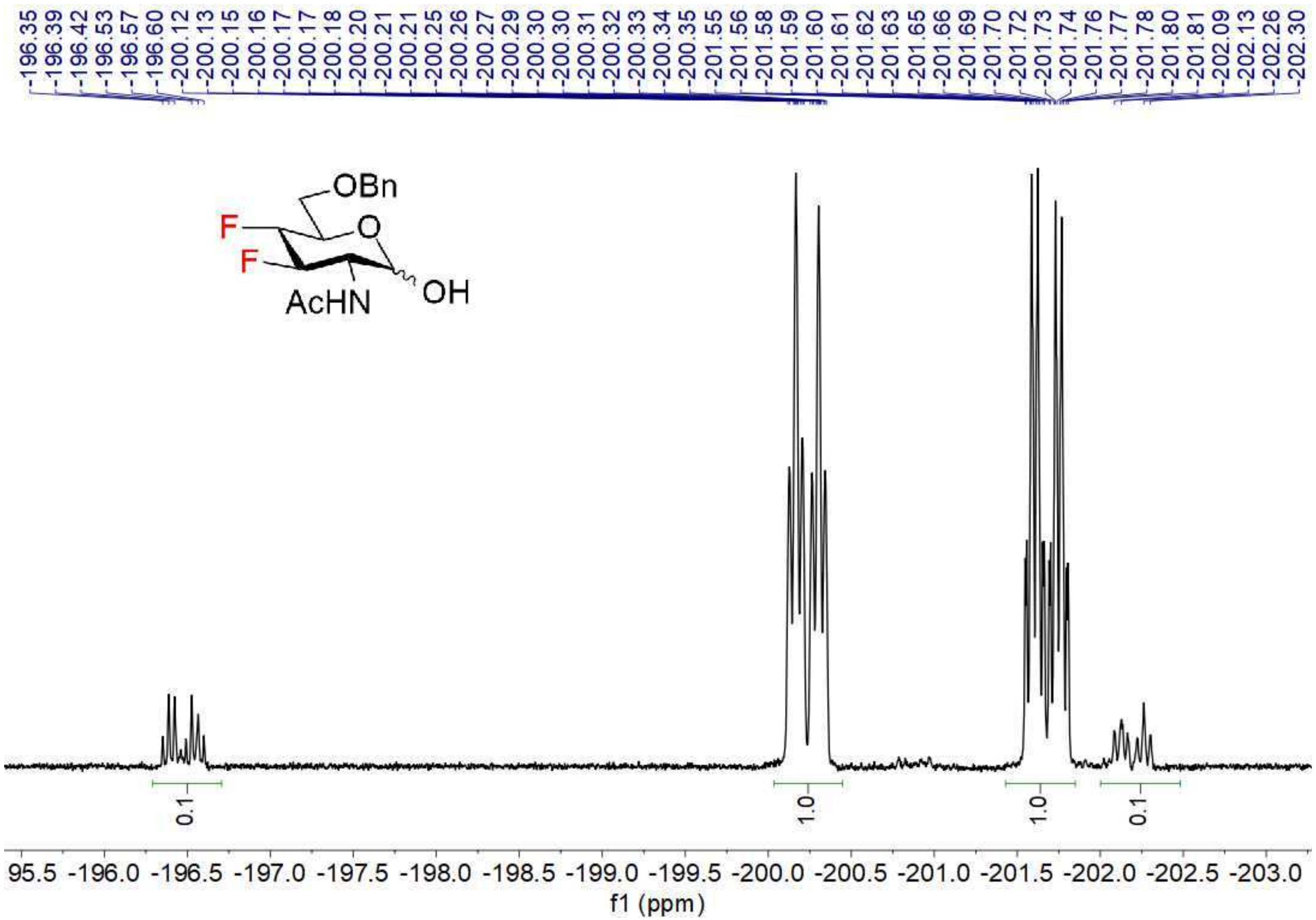
^{1}H NMR (400 MHz, CD_3OD) 53 (α/β ca. 10/1)



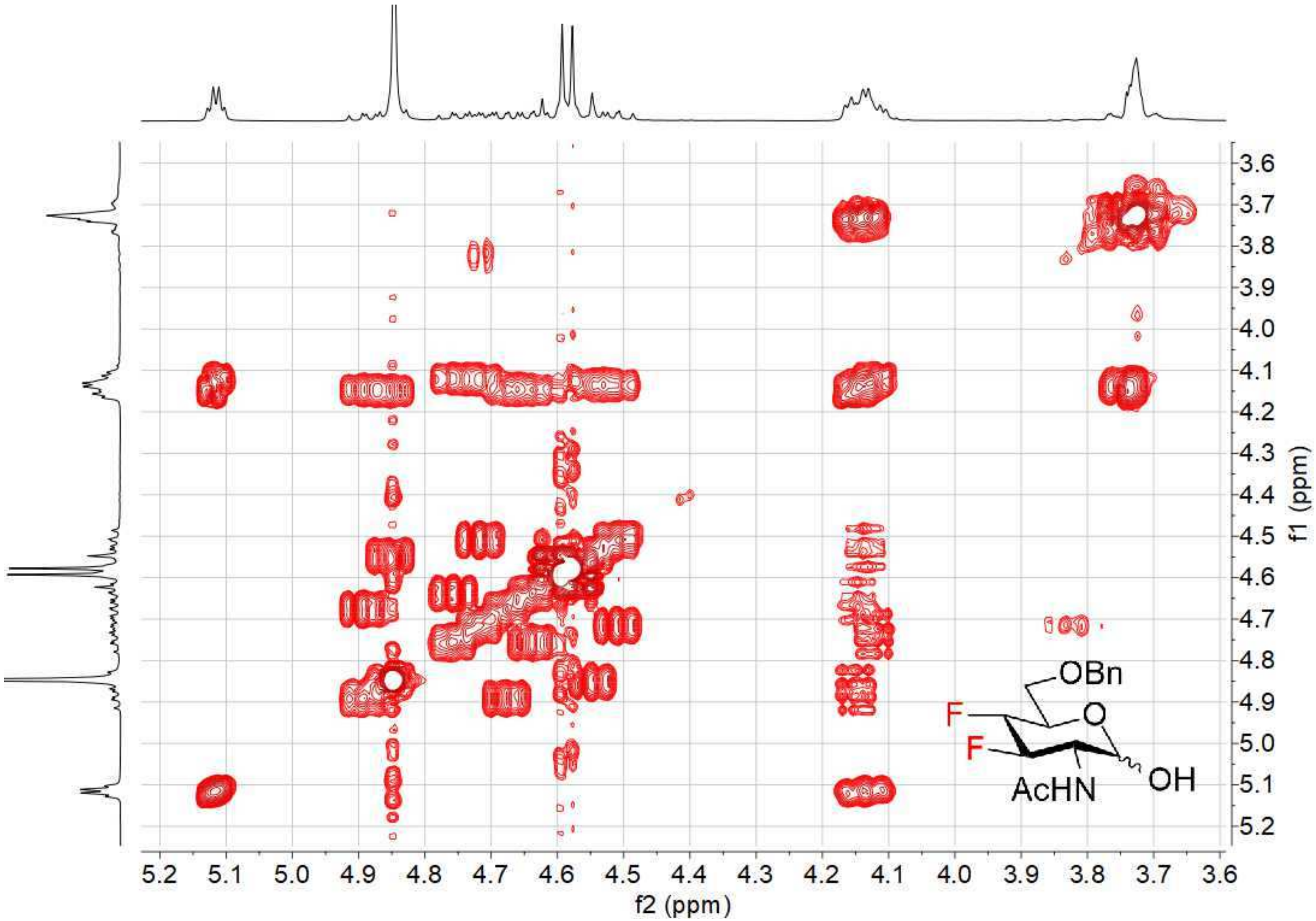
[13]C NMR (100 MHz, CD$_3$OD) 53



^{19}F NMR (376 MHz, CD_3OD) 53 (α/β ca. 10/1)

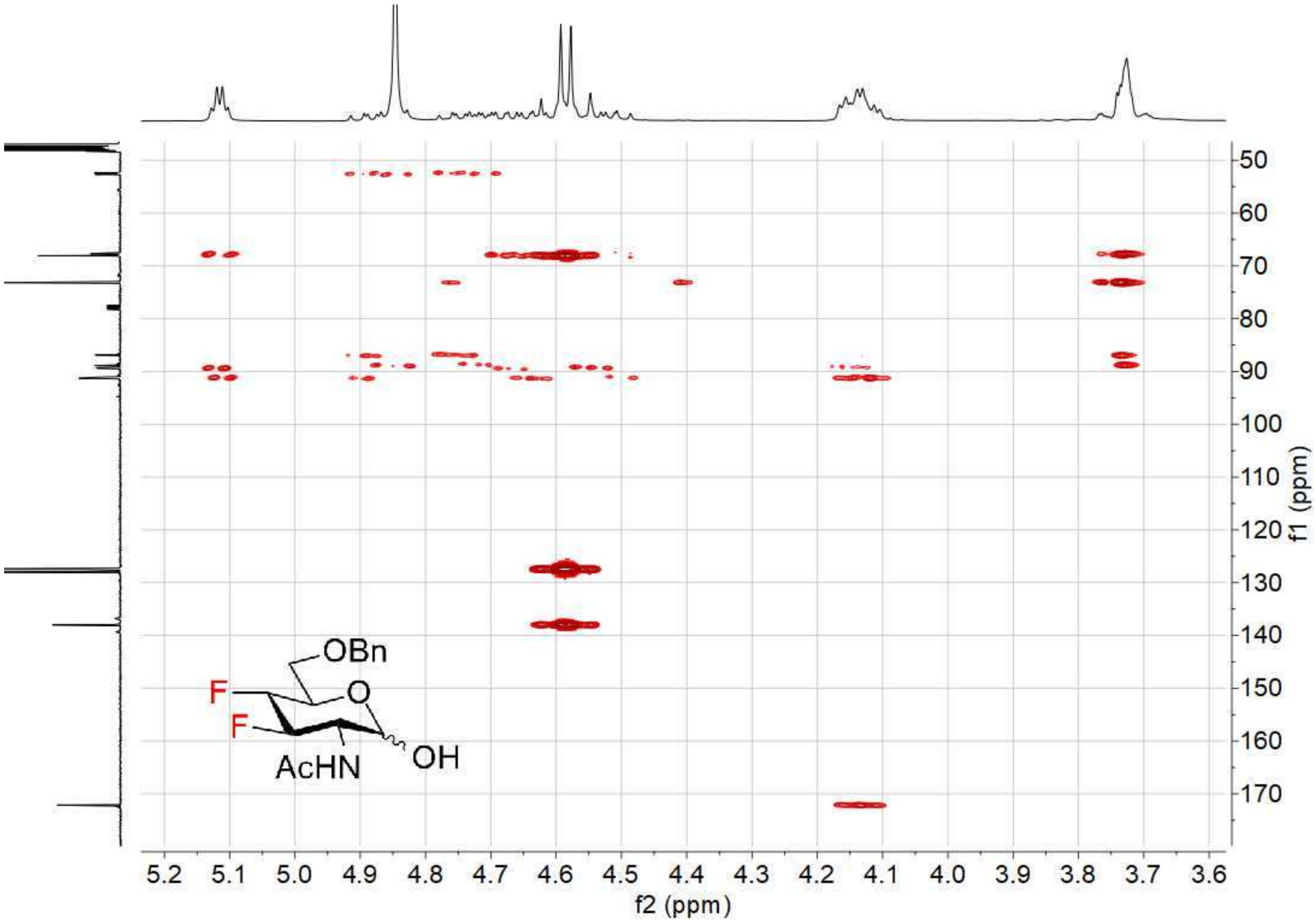


^{1}H-^{1}H COSY 53

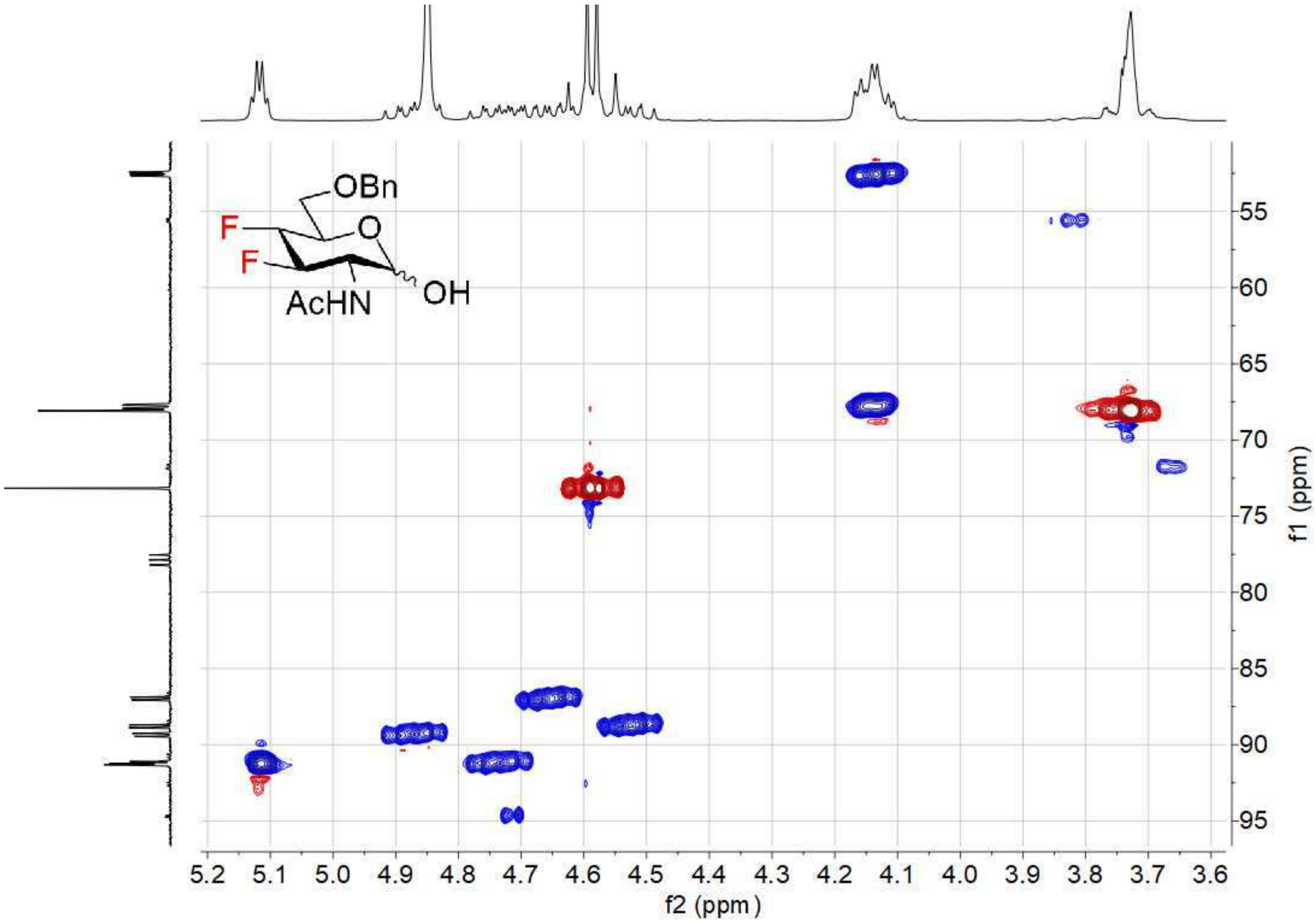


5.2 5.1 5.0 4.9 4.8 4.7 4.6 4.5 4.4 4.3 4.2 4.1 4.0 3.9 3.8 3.7 3.6
f2 (ppm)
3.6 3.7 3.8 3.9 4.0 4.1 4.2 4.3 4.4 4.5 4.6 4.7 4.8 4.9 5.0 5.1 5.2
f1 (ppm)
OBn
F
F
O
AcHN
OH

^{1}H-^{13}C HMBC 53

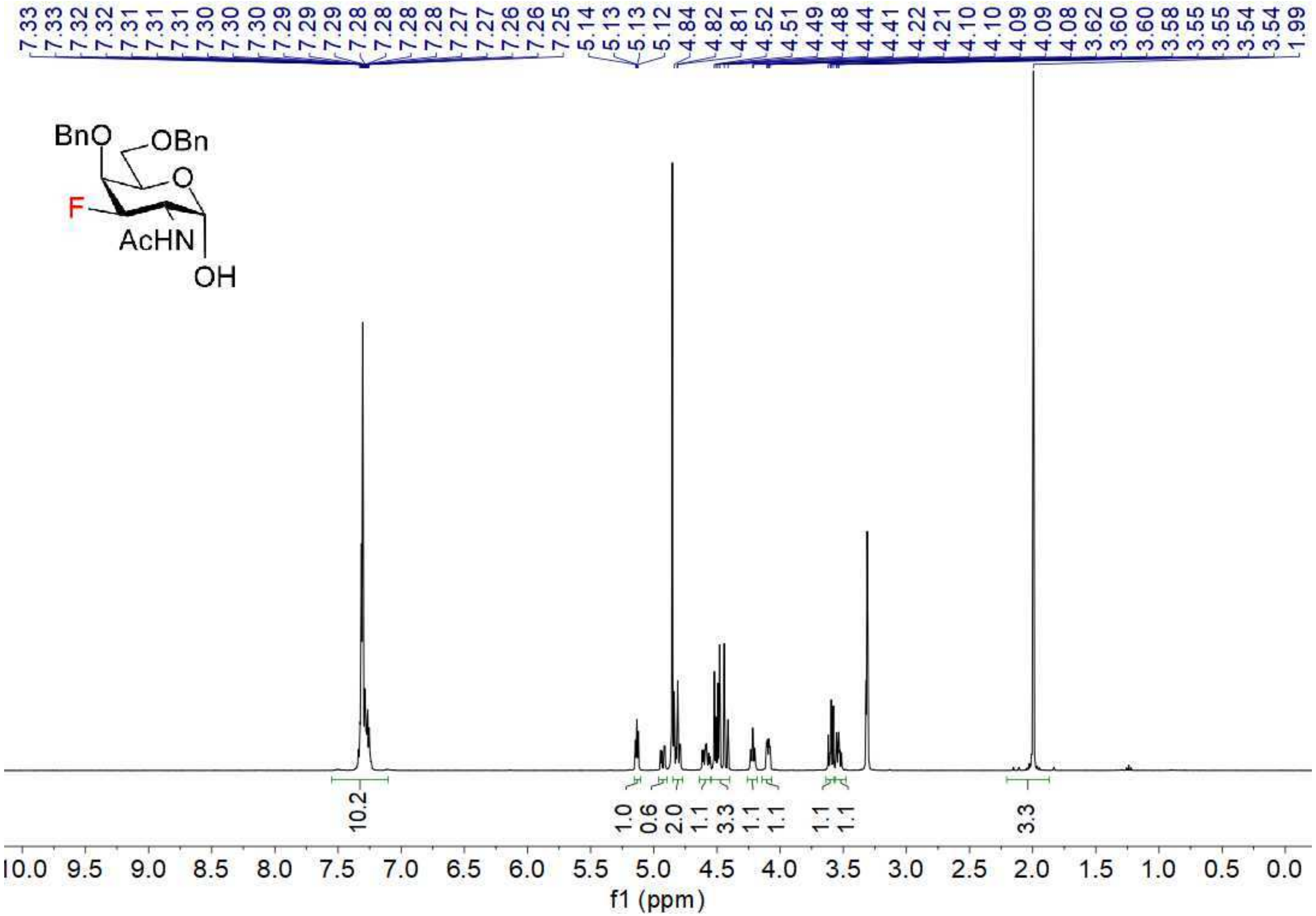


^{1}H-^{13}C HSQC 53

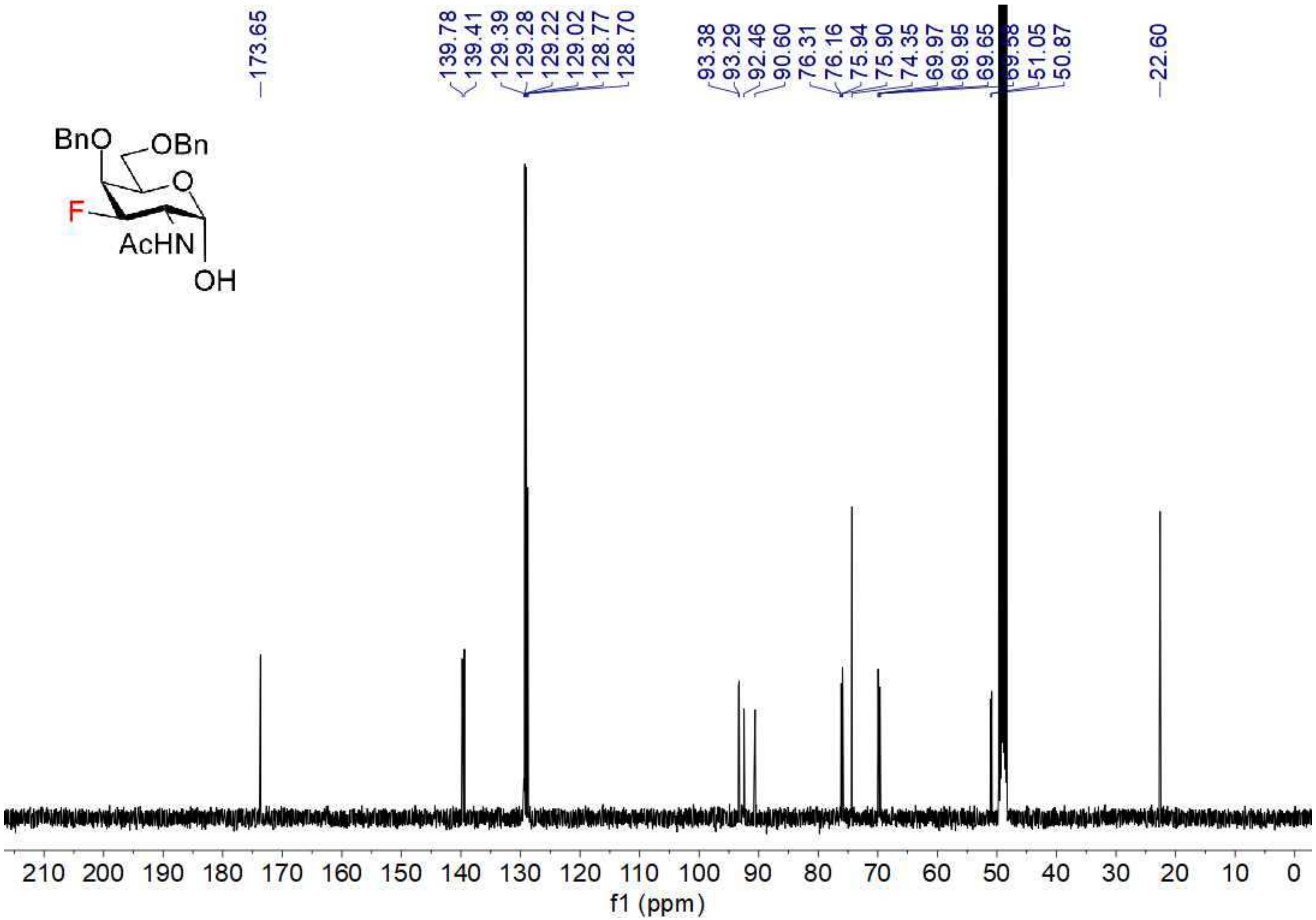


NMR COMPOUND 54

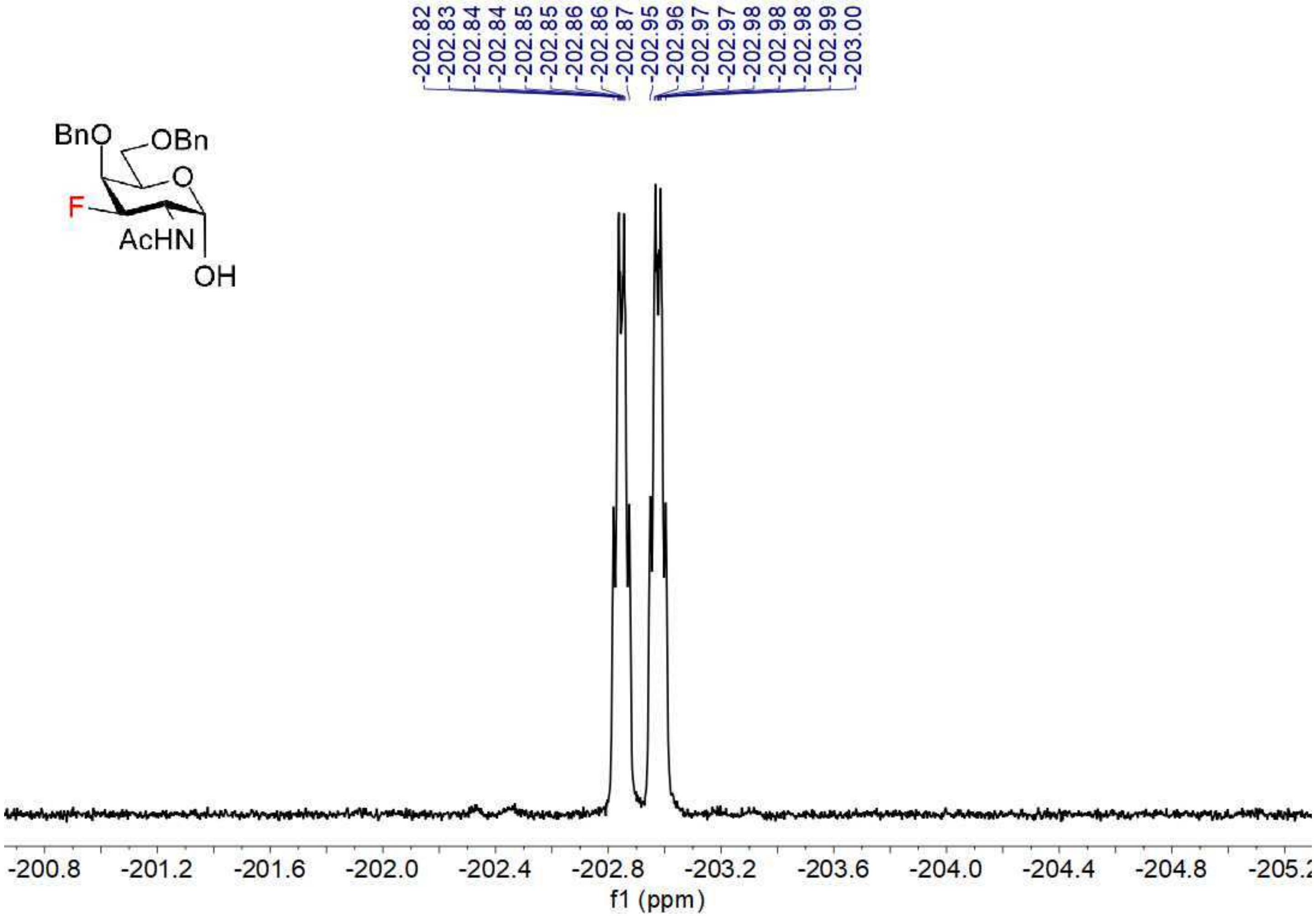
^{1}H NMR (400 MHz, CD_3OD) 54



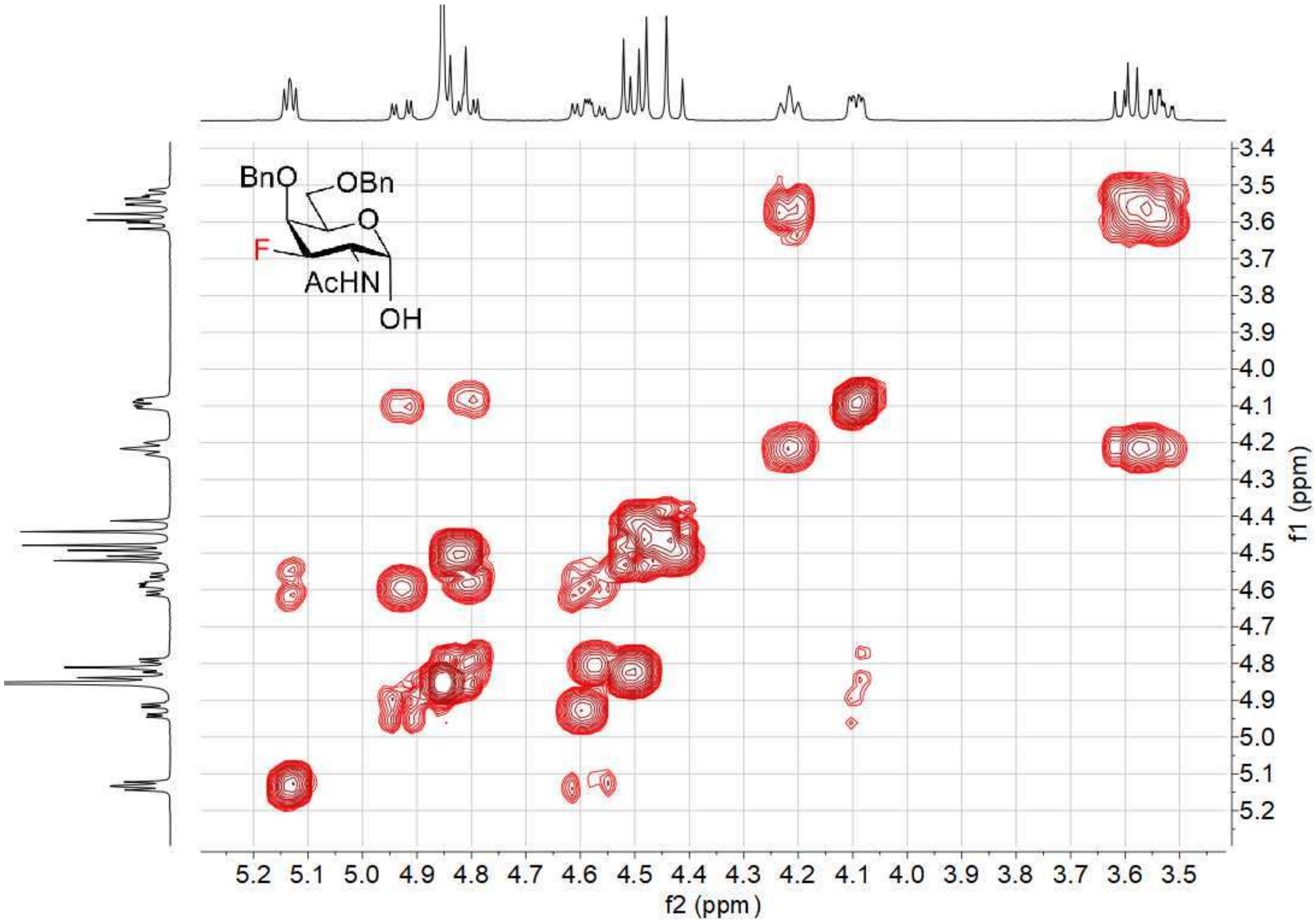
[13]C NMR (100 MHz, CD_3OD) 54



^{19}F NMR (376 MHz, CD_3OD) 54

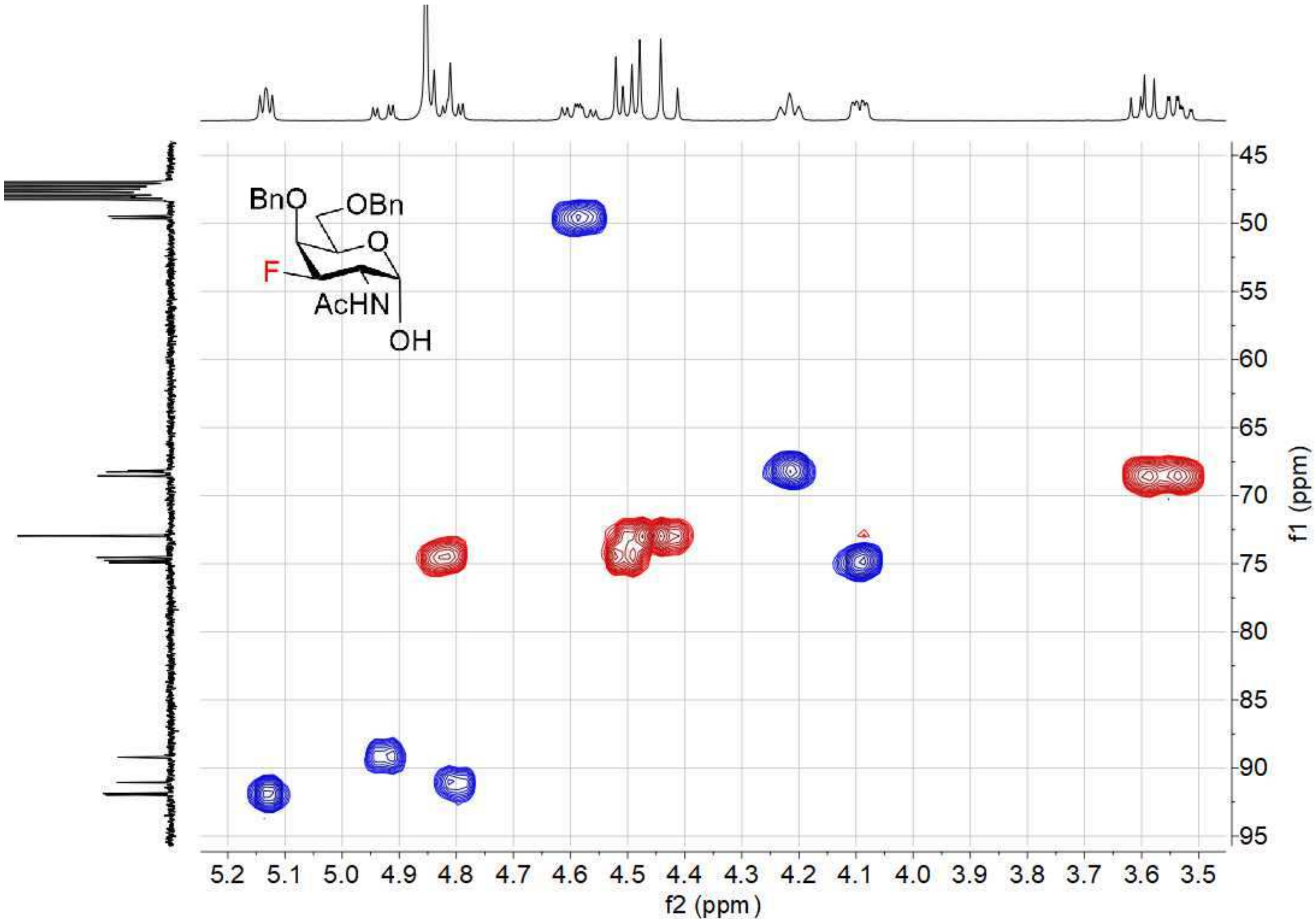


[1]H-[1]H COSY 54



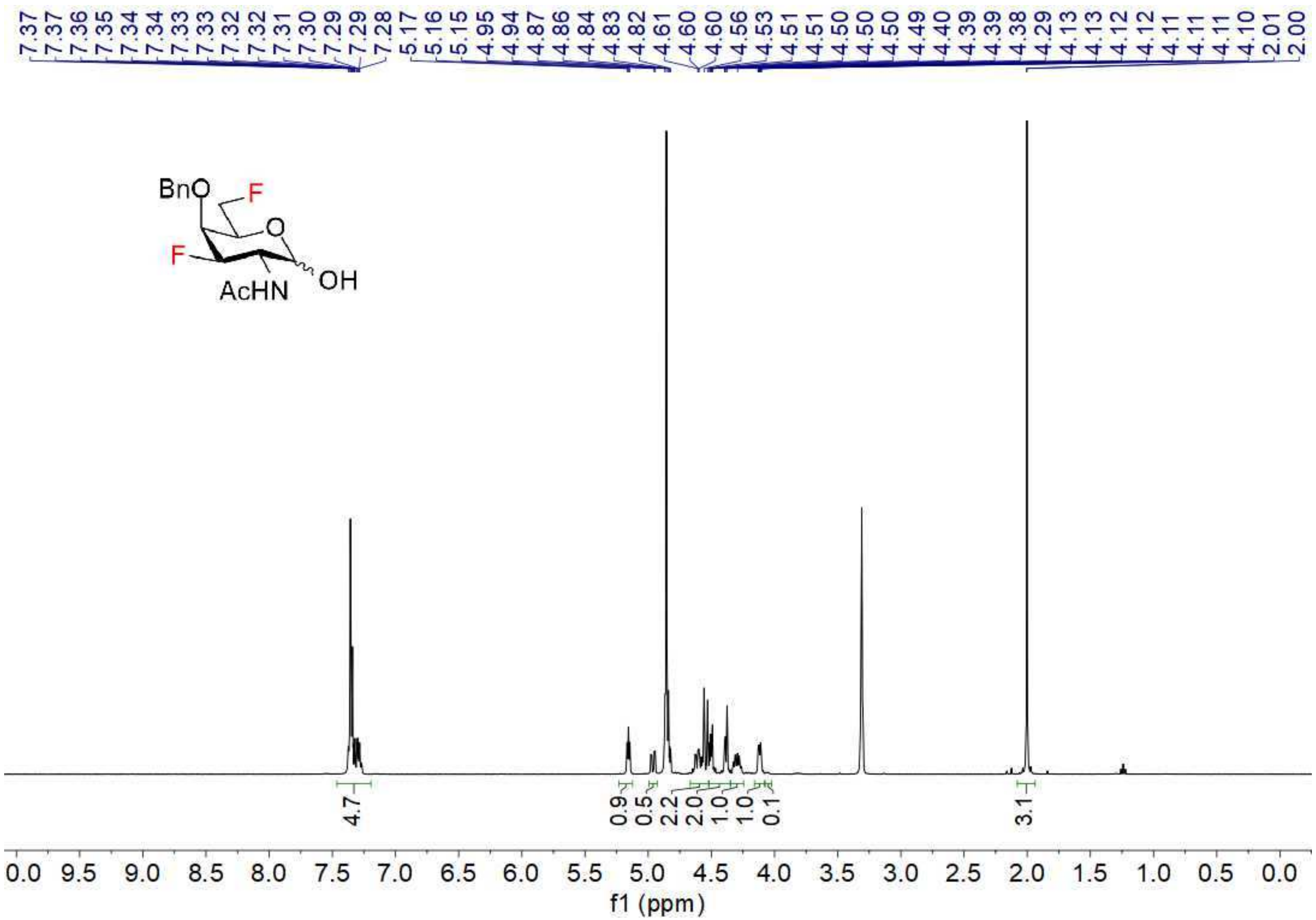
BnO
OBn
O
F
AcHN
OH
5.2 5.1 5.0 4.9 4.8 4.7 4.6 4.5 4.4 4.3 4.2 4.1 4.0 3.9 3.8 3.7 3.6 3.5
f2 (ppm)
3.4 3.5 3.6 3.7 3.8 3.9 4.0 4.1 4.2 4.3 4.4 4.5 4.6 4.7 4.8 4.9 5.0 5.1 5.2
f1 (ppm)

^{1}H-^{13}C HSQC 54

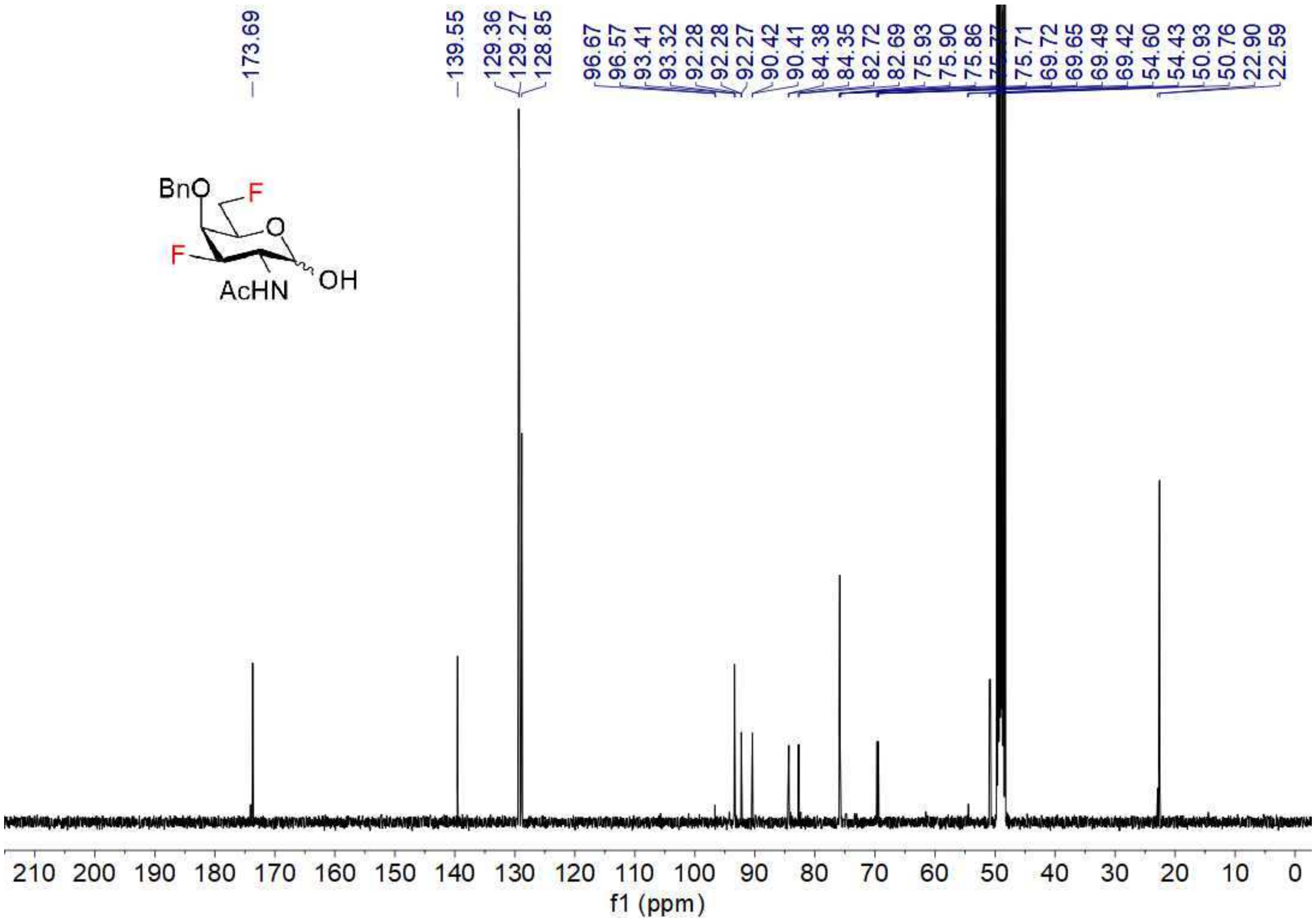


NMR COMPOUND 55

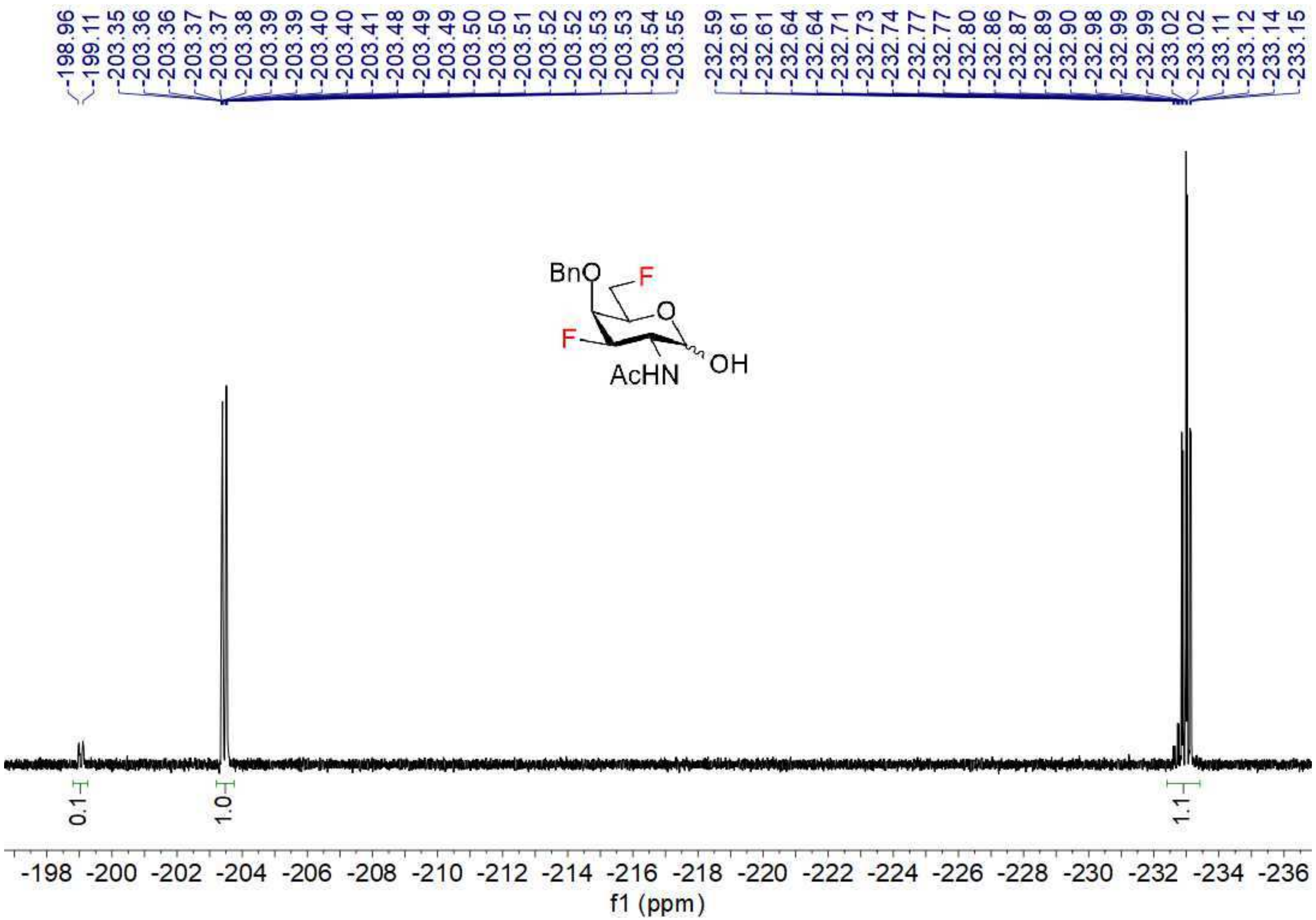
^{1}H NMR (400 MHz, CD_3OD) 55 (α/β ca. 10/1)



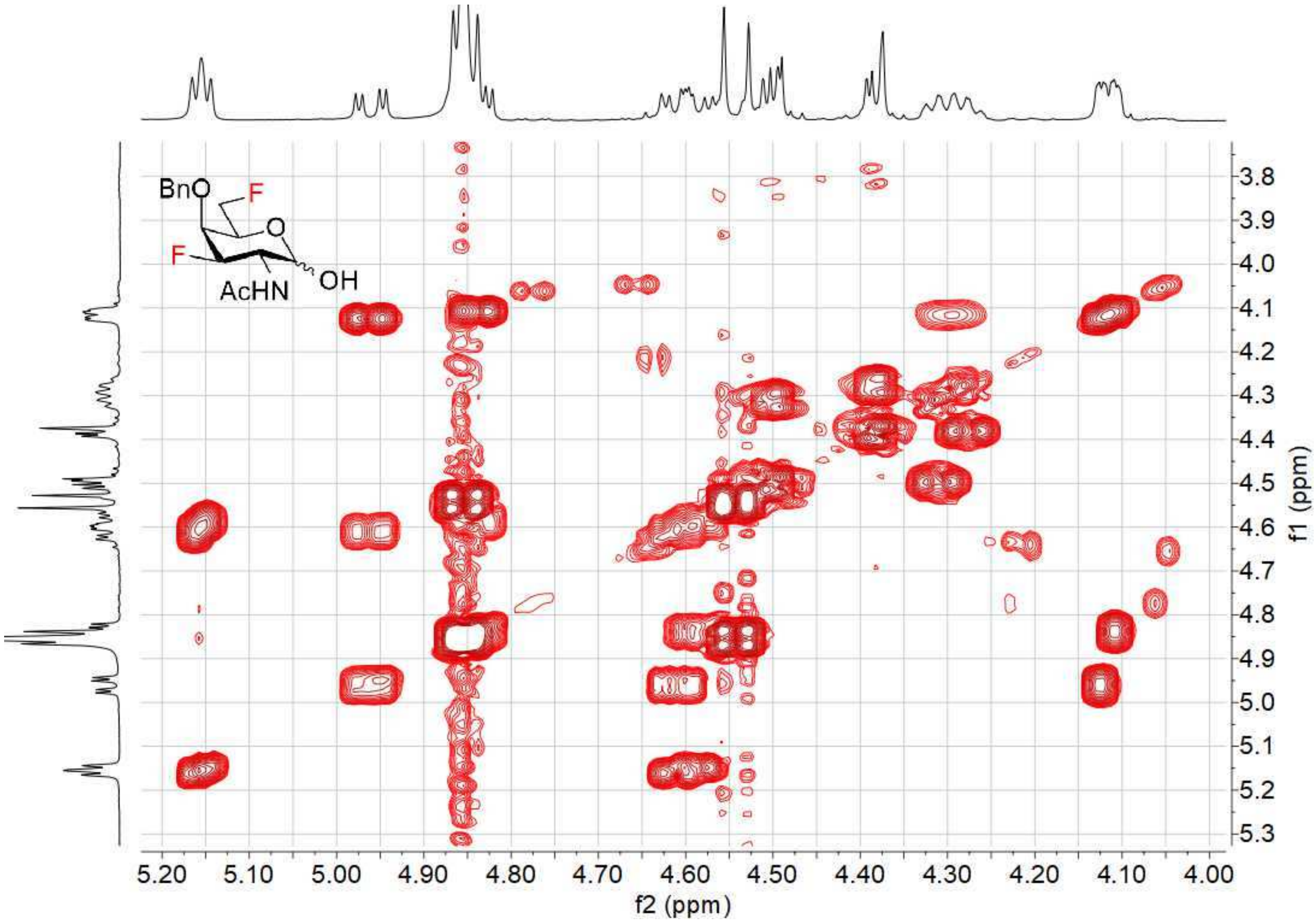
[13]C NMR (100 MHz, CD$_3$OD) 55



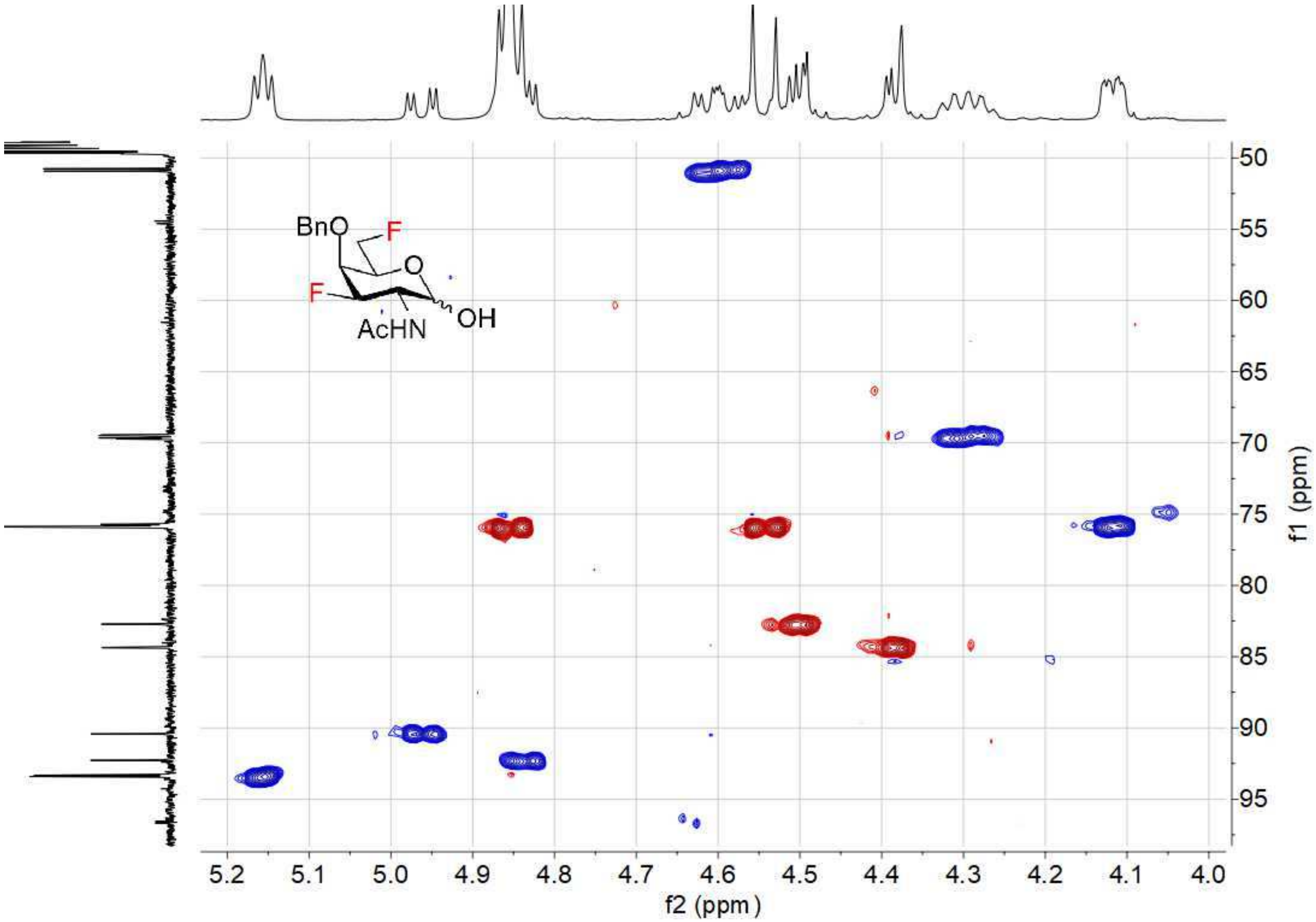
^{19}F NMR (376 MHz, CD_3OD) 55 (α/β ca. 10/1)



^{1}H-^{1}H COSY 55

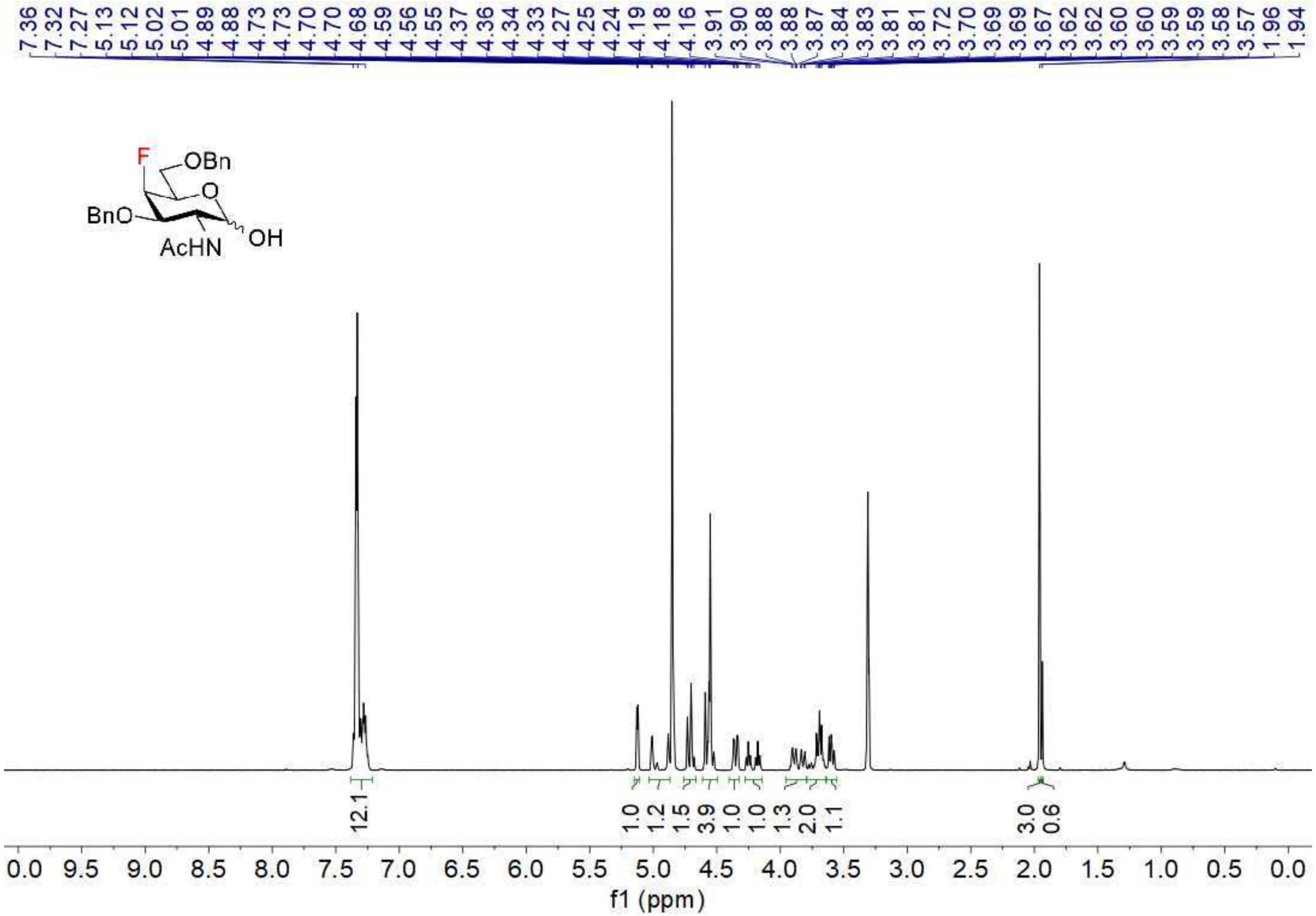

BnO
F
O
F
OH
AcHN
f1 (ppm)
f2 (ppm)
5.20 5.10 5.00 4.90 4.80 4.70 4.60 4.50 4.40 4.30 4.20 4.10 4.00
3.8 3.9 4.0 4.1 4.2 4.3 4.4 4.5 4.6 4.7 4.8 4.9 5.0 5.1 5.2 5.3

[1]H-[13]C HSQC 55

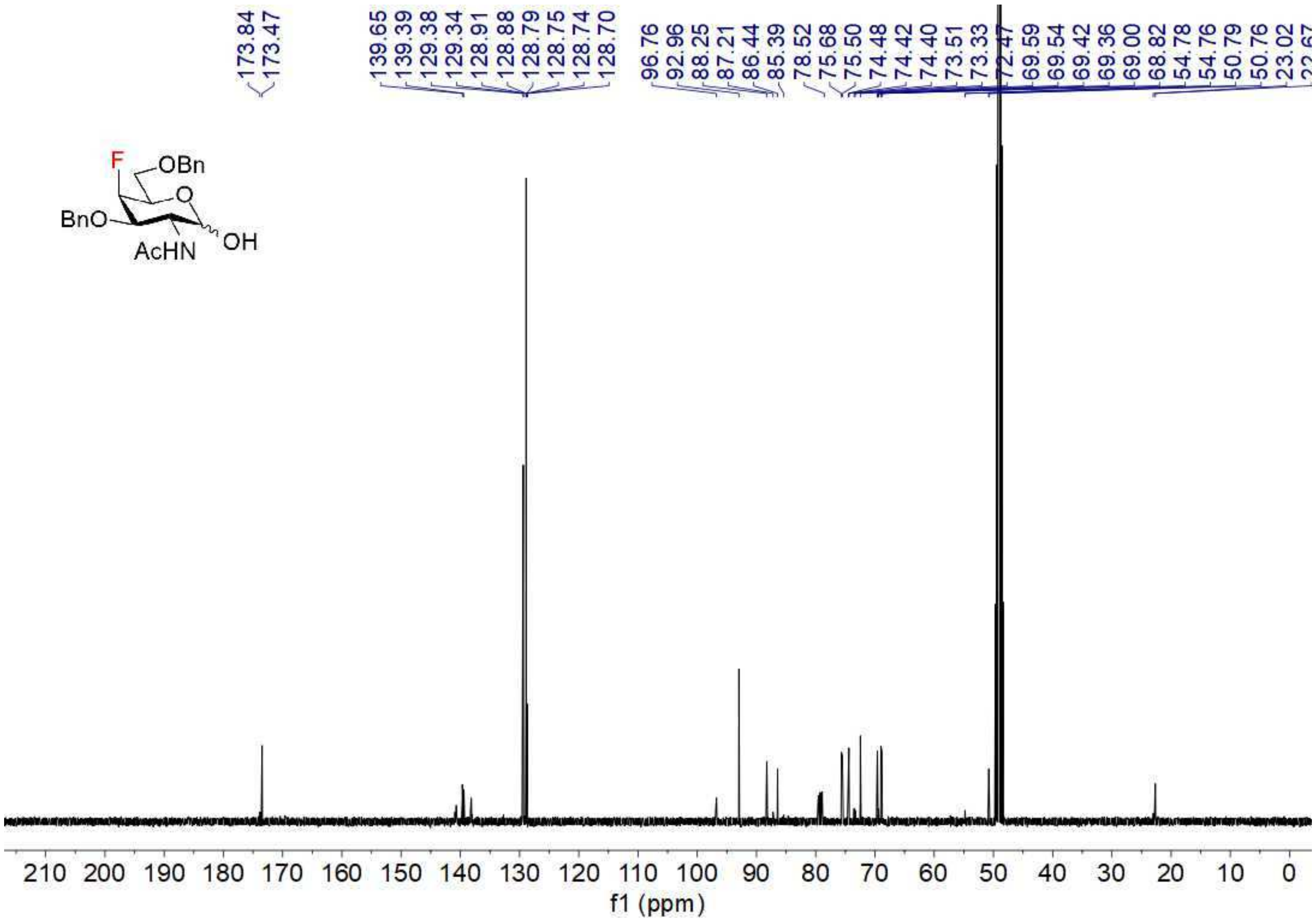


NMR COMPOUND 56

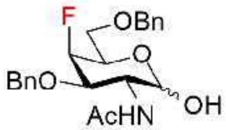
^{1}H NMR (400 MHz, CD_3OD) 56 (α/β ca. 5/1)

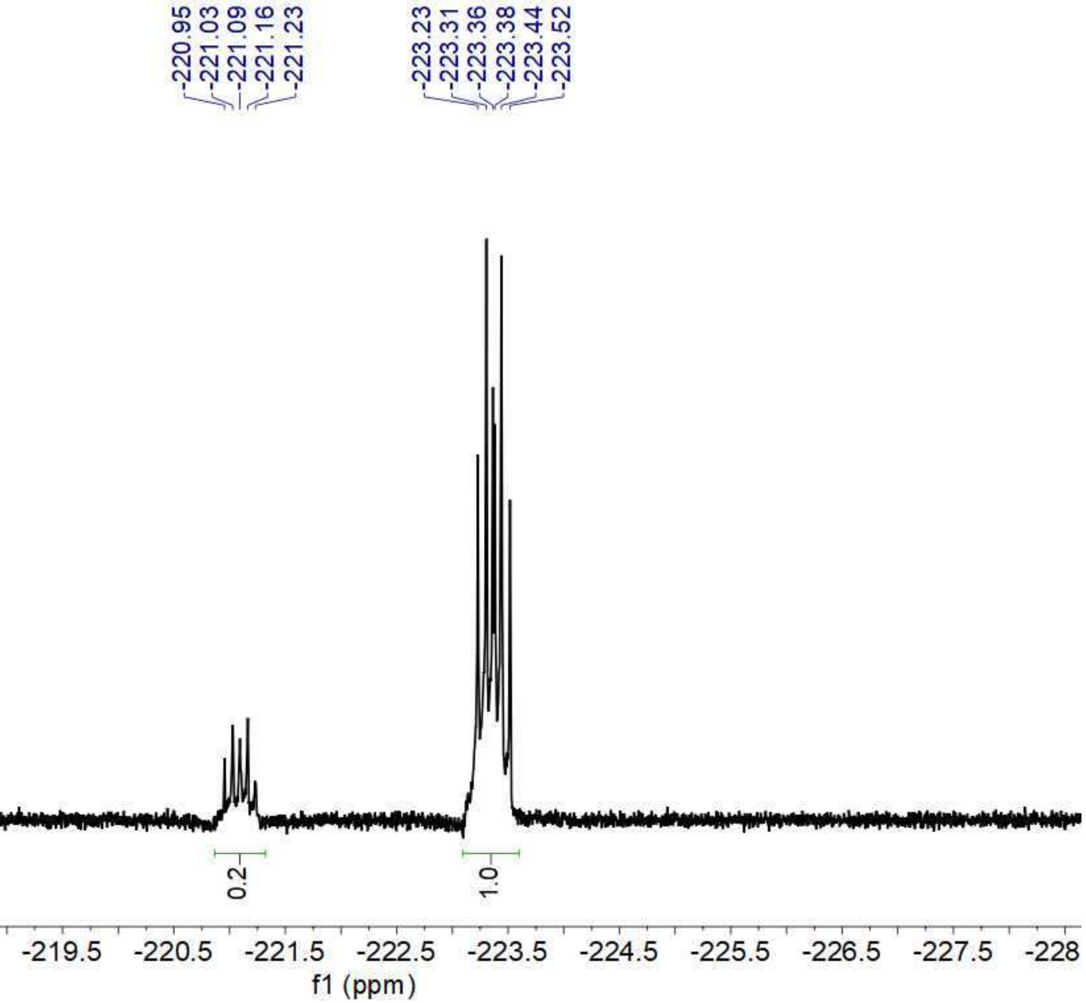


^{13}C NMR (100 MHz, CD_3OD) 56

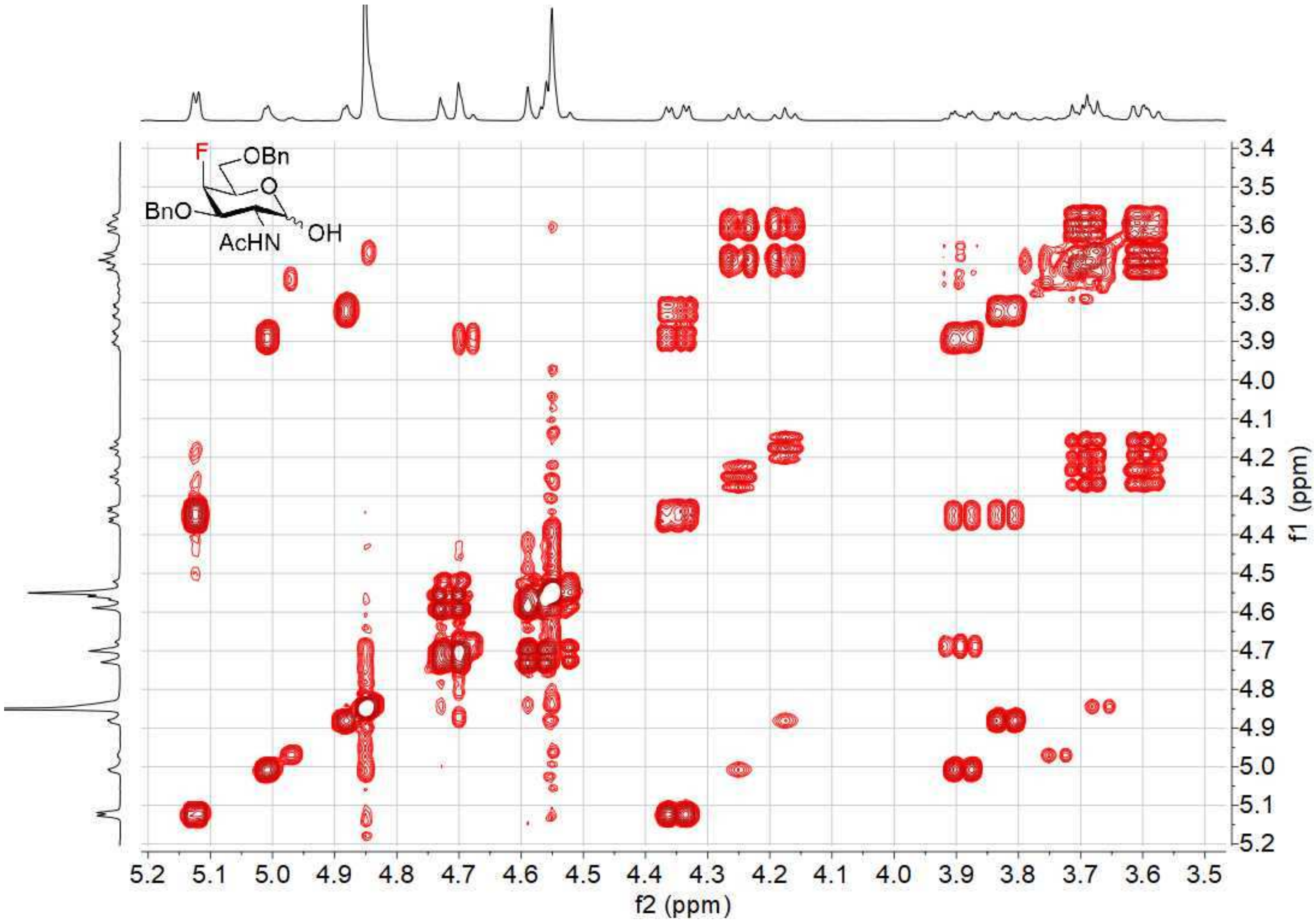


^{19}F NMR (376 MHz, CD_3OD) 56 (α/β ca. 5/1)

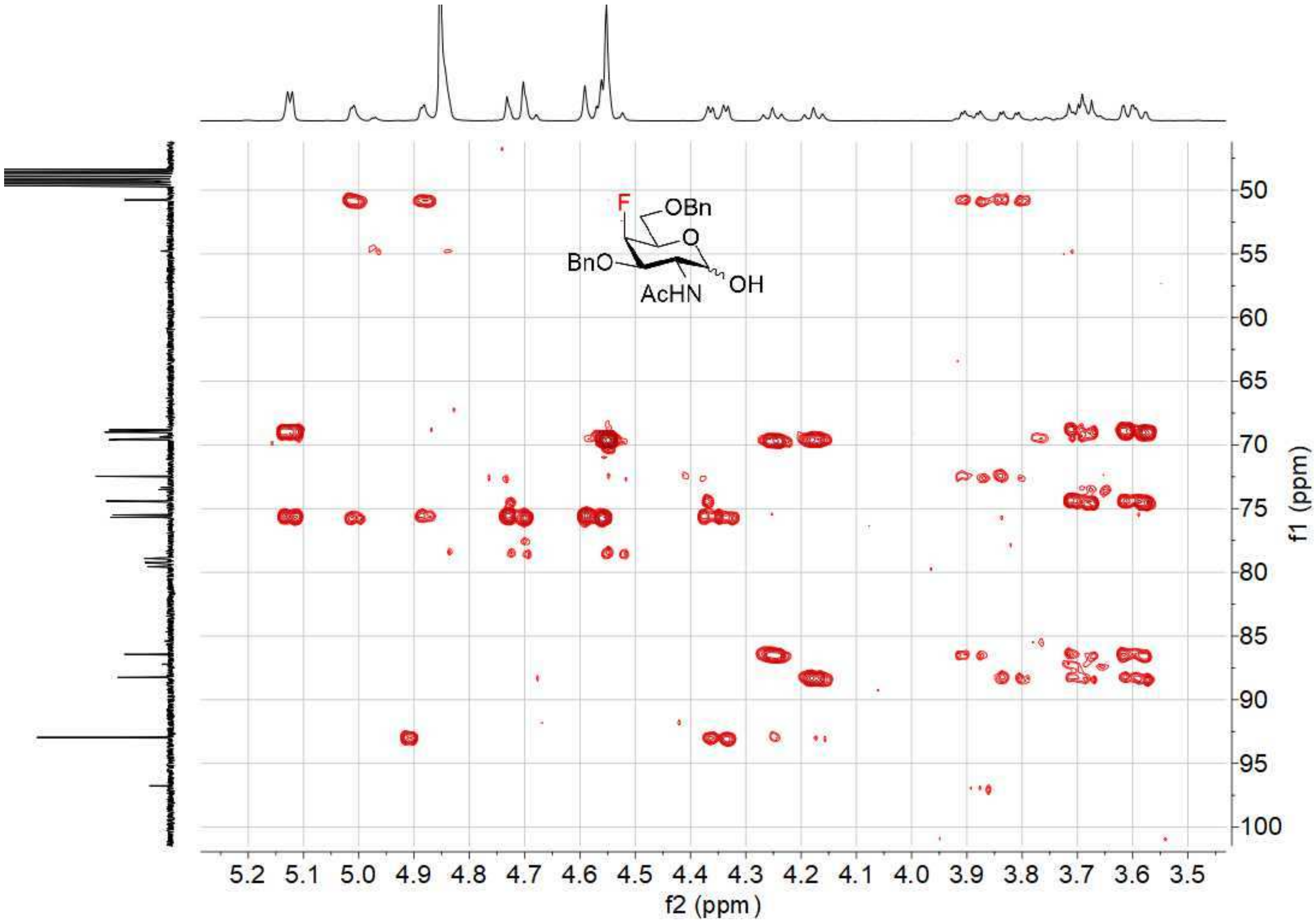




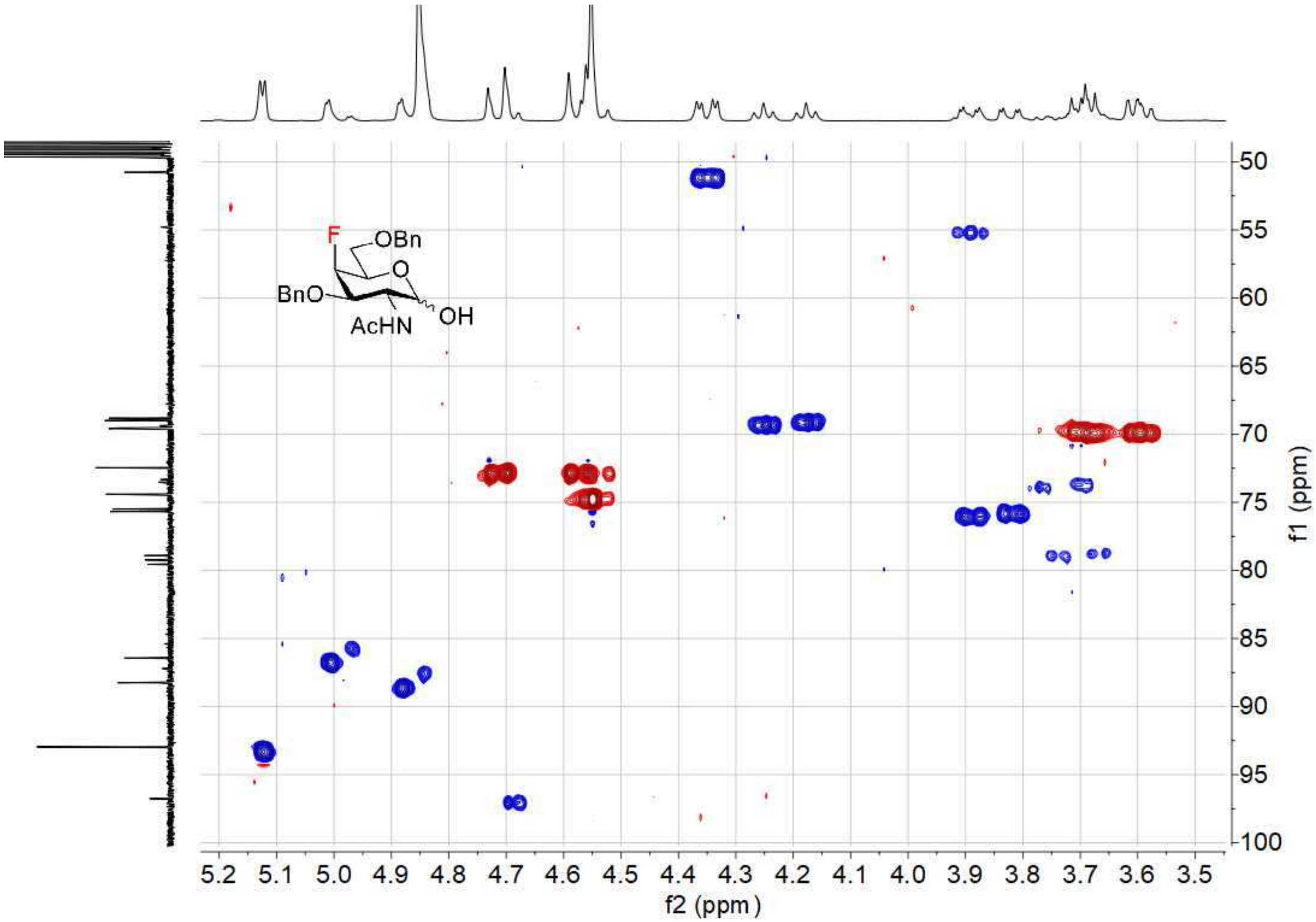
^{1}H-^{1}H COSY 56


F
OBn
O
BnO
OH
AcHN
f2 (ppm)
f1 (ppm)
5.2 5.1 5.0 4.9 4.8 4.7 4.6 4.5 4.4 4.3 4.2 4.1 4.0 3.9 3.8 3.7 3.6 3.5
3.4 3.5 3.6 3.7 3.8 3.9 4.0 4.1 4.2 4.3 4.4 4.5 4.6 4.7 4.8 4.9 5.0 5.1 5.2

[1]H-[13]C HMBC 56

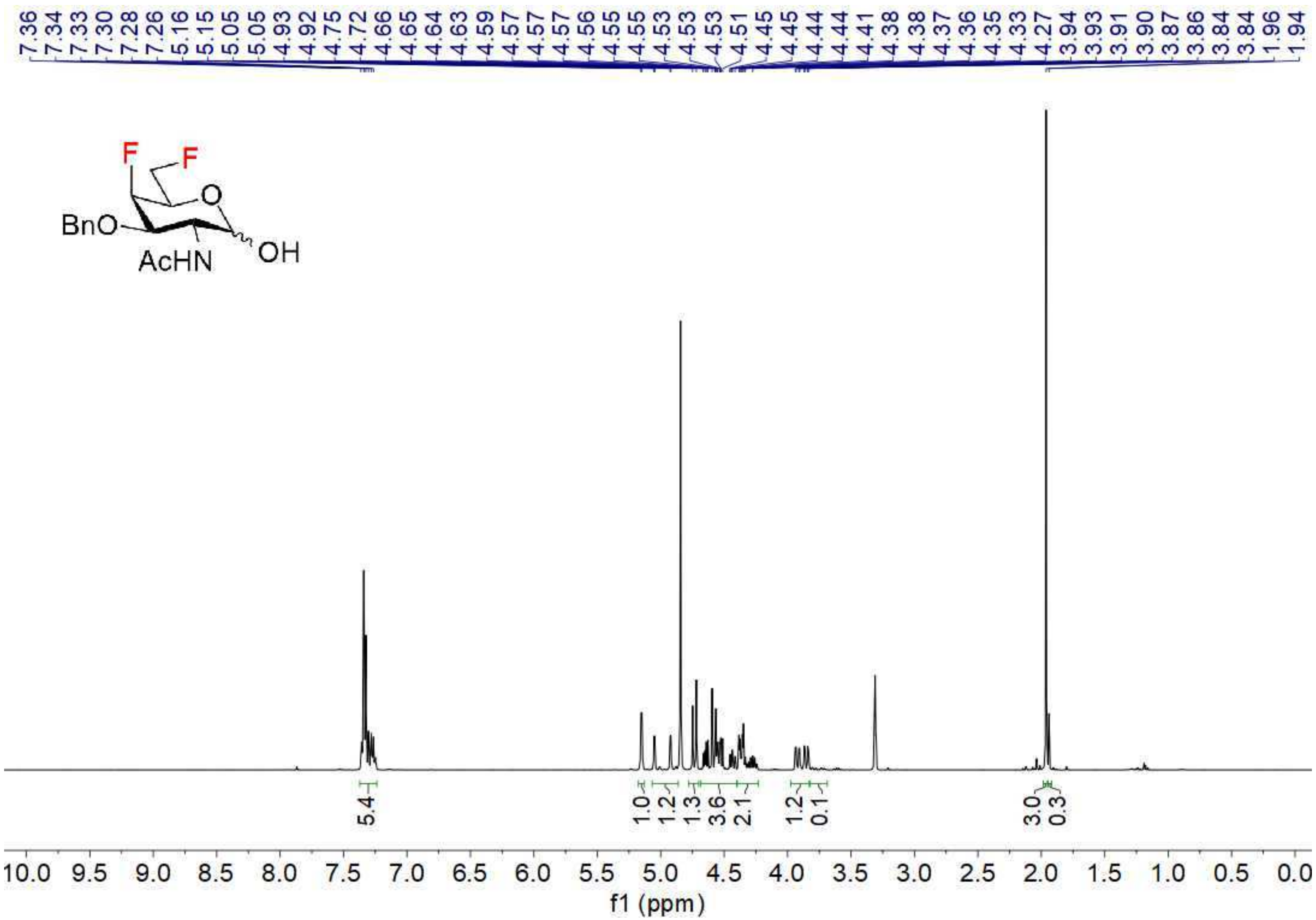


^1H-^{13}C HSQC 56

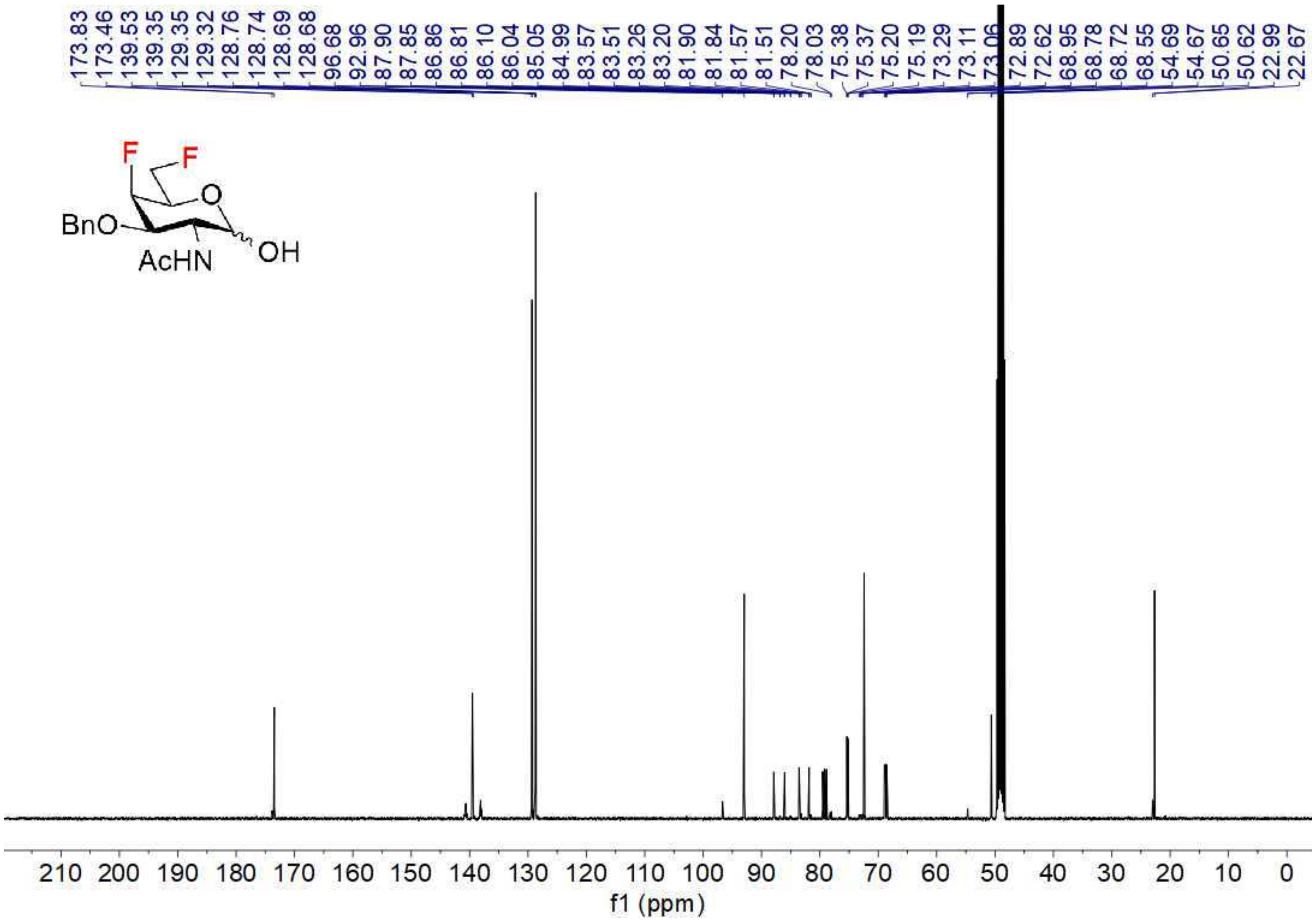


NMR COMPOUND 57

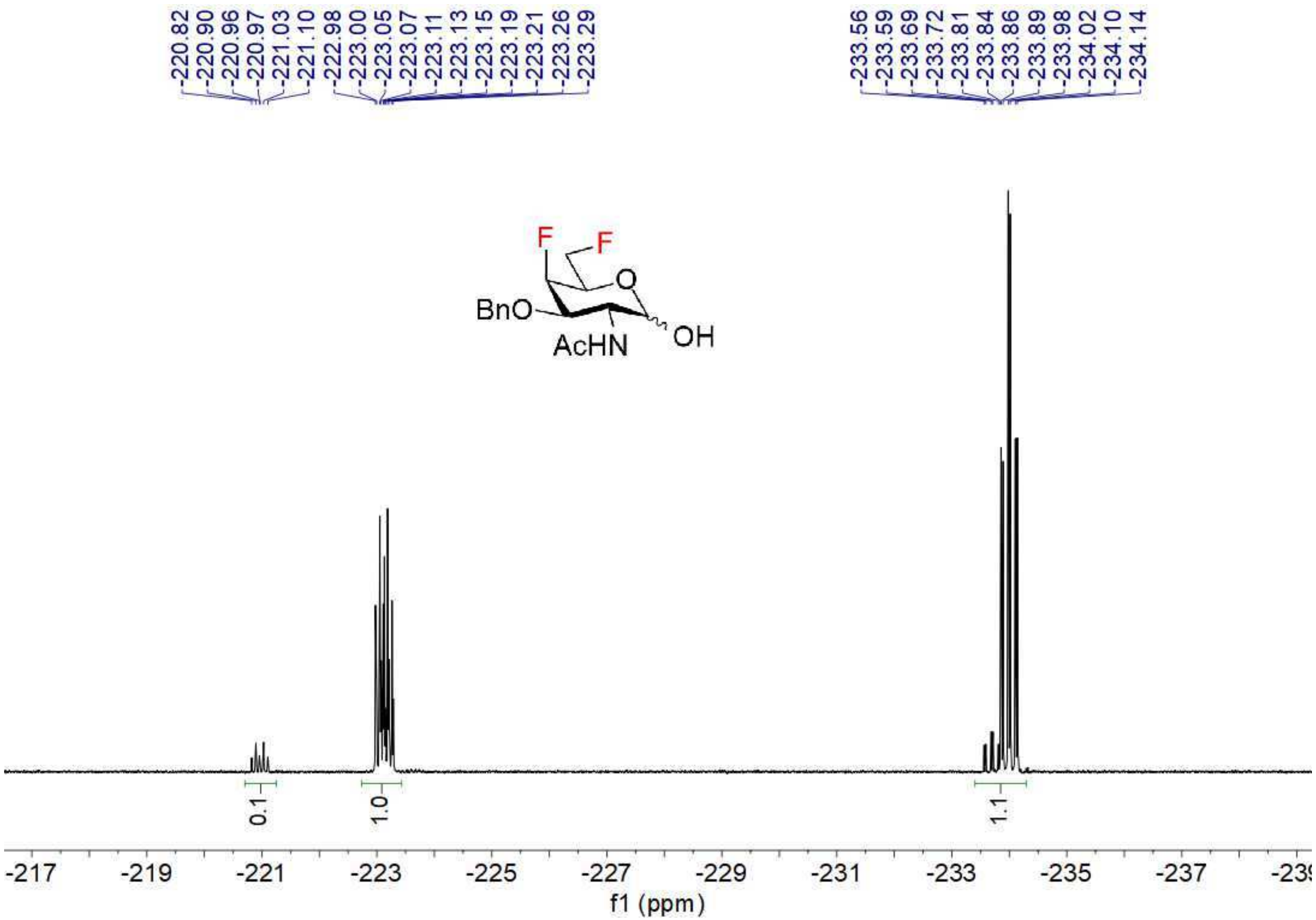
^{1}H NMR (400 MHz, CD_3OD) 57 (α/β ca. 10/1)



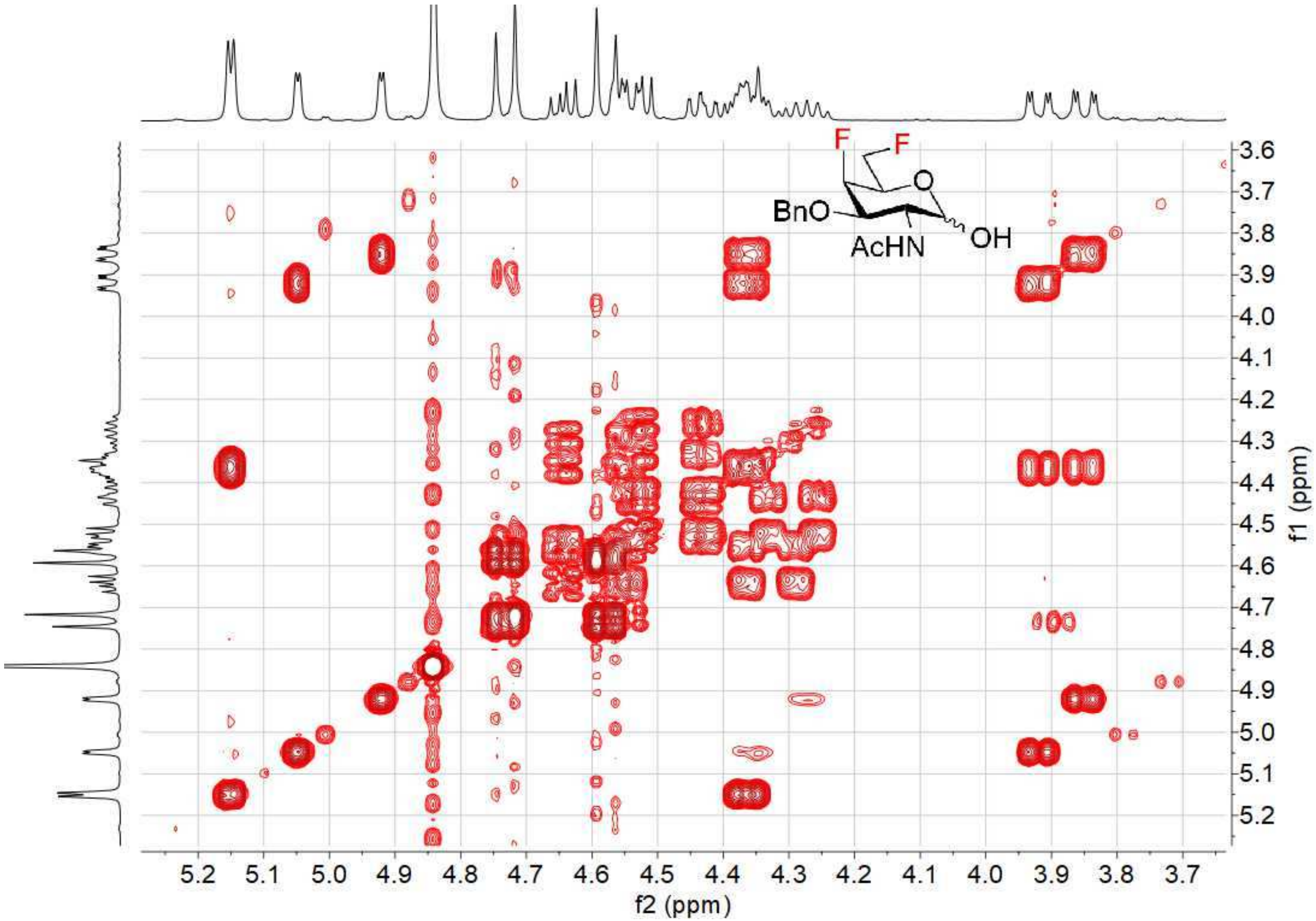
^{13}C NMR (100 MHz, CD_3OD) 57



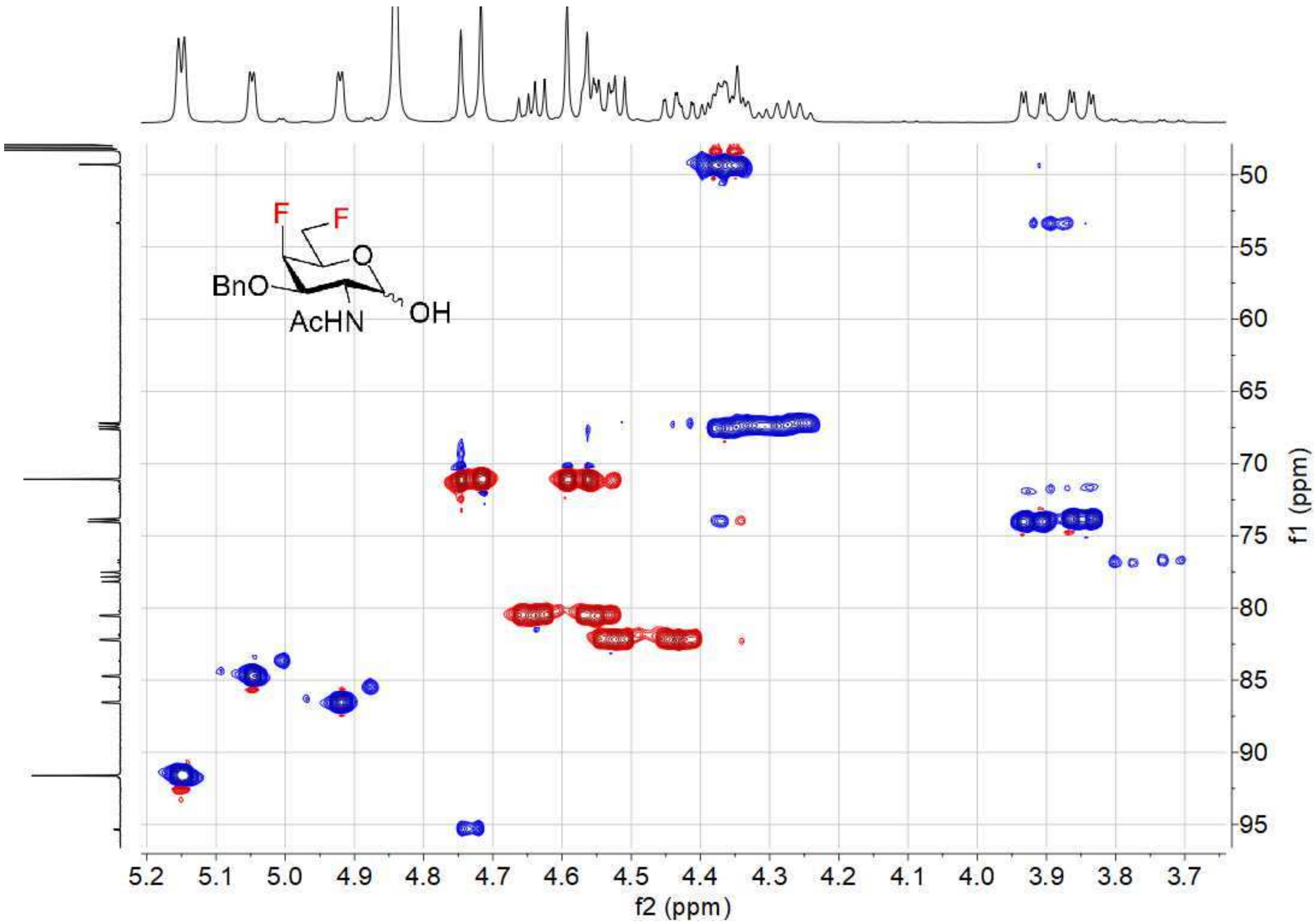
^{19}F NMR (376 MHz, CD_3OD) 57 (α/β ca. 10/1)



^{1}H-^{1}H COSY NMR 57

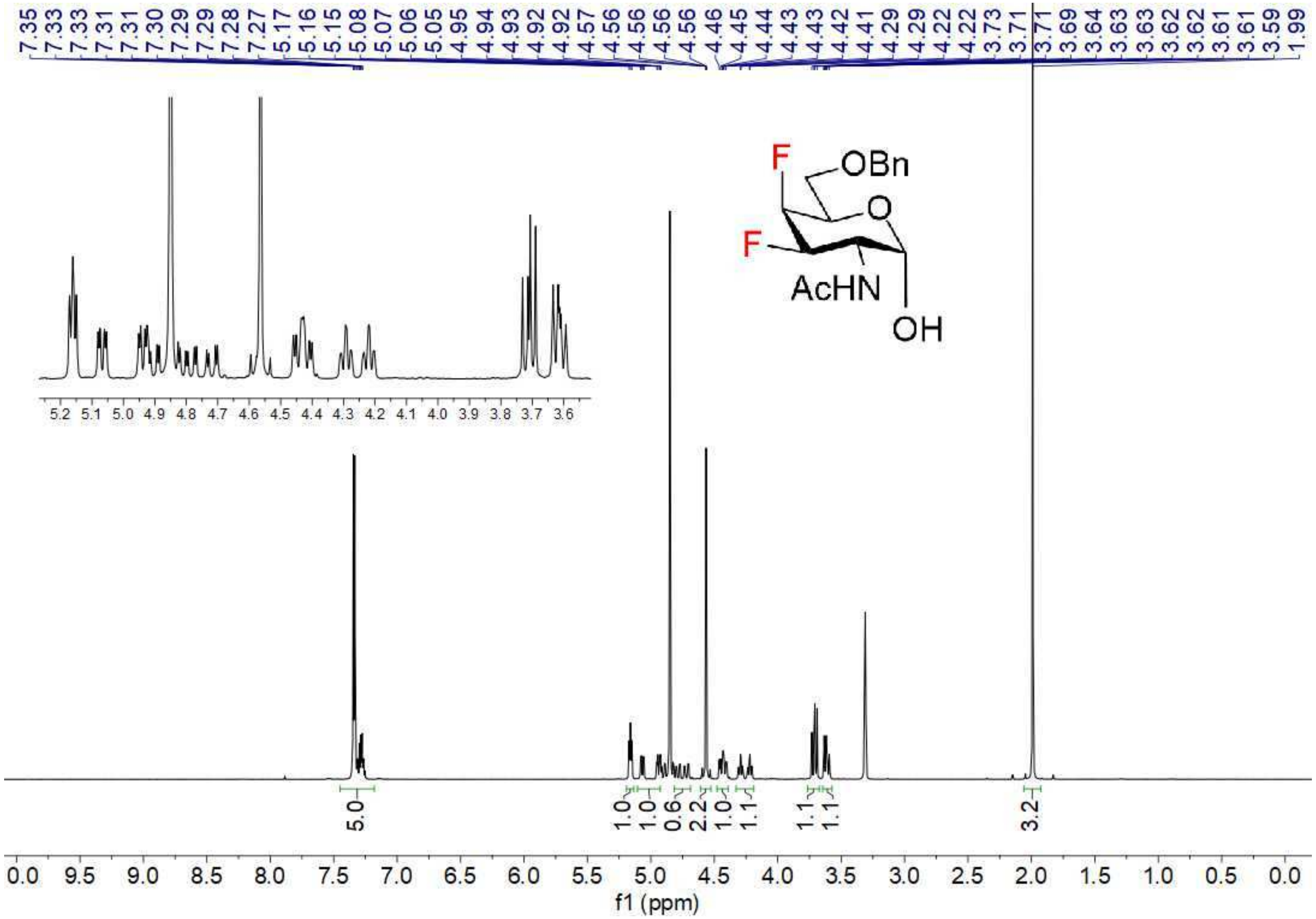


[1]H-[13]C HSQC NMR 57

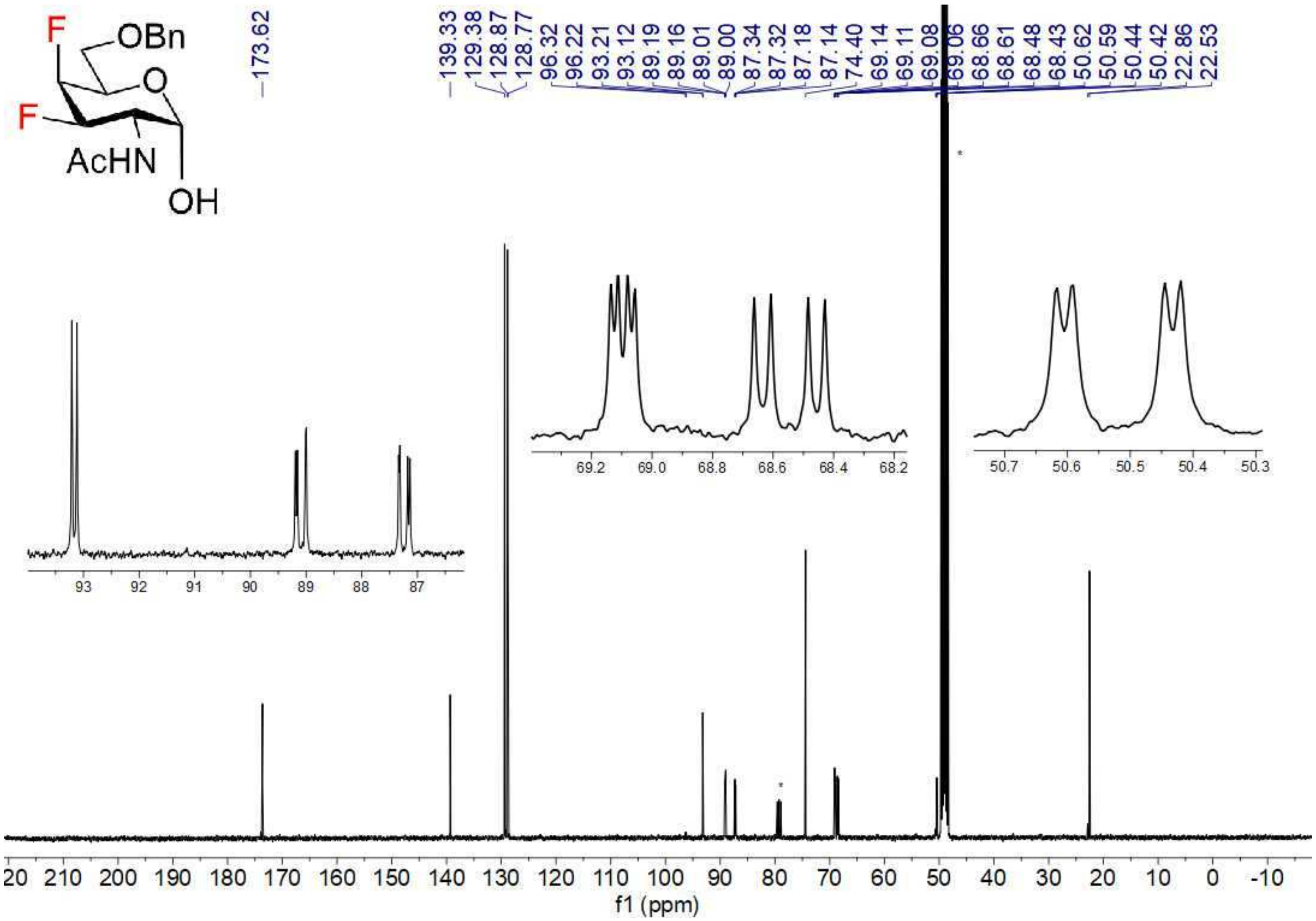


NMR COMPOUND 58

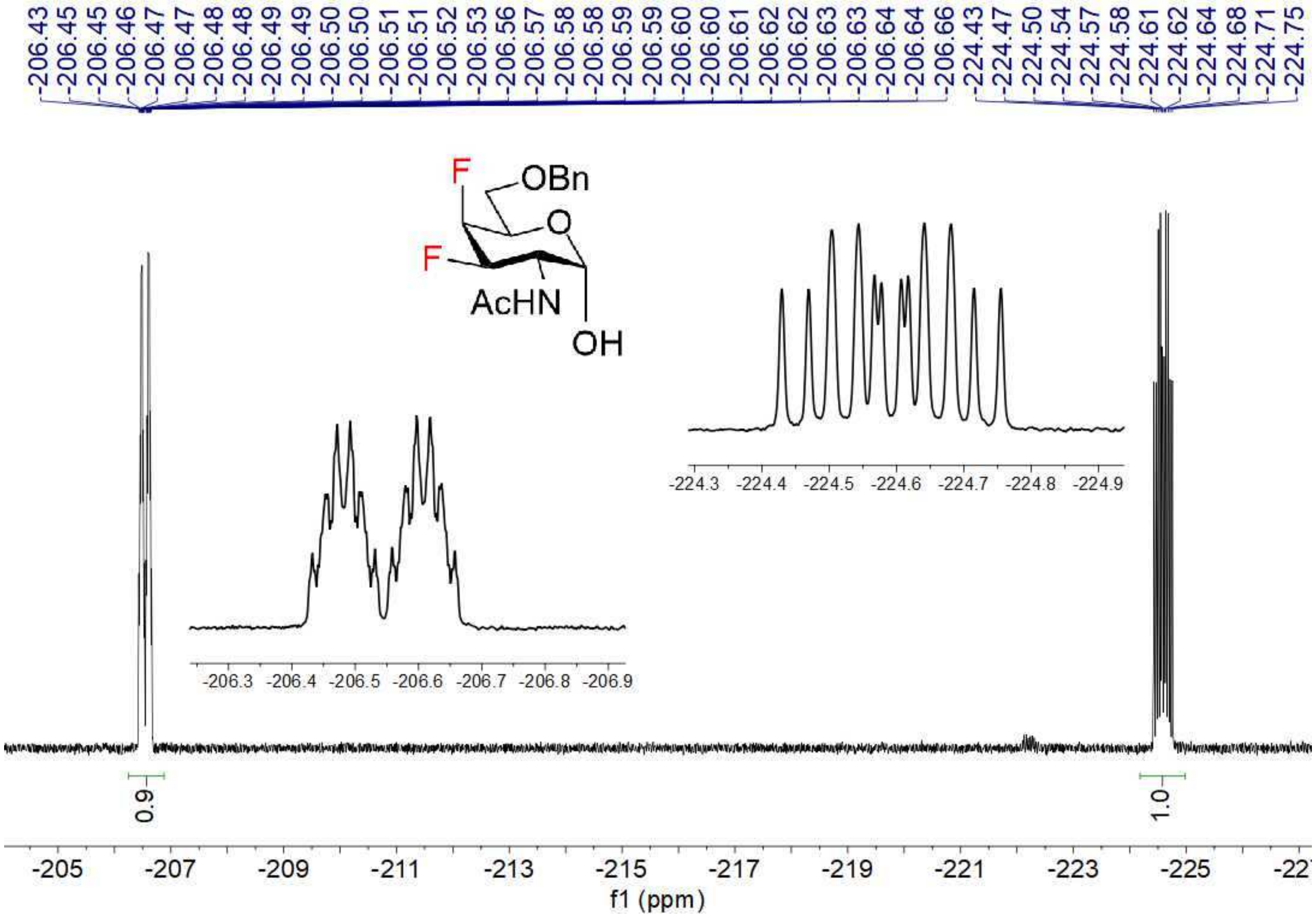
^{1}H NMR (400 MHz, CD_3OD) 58



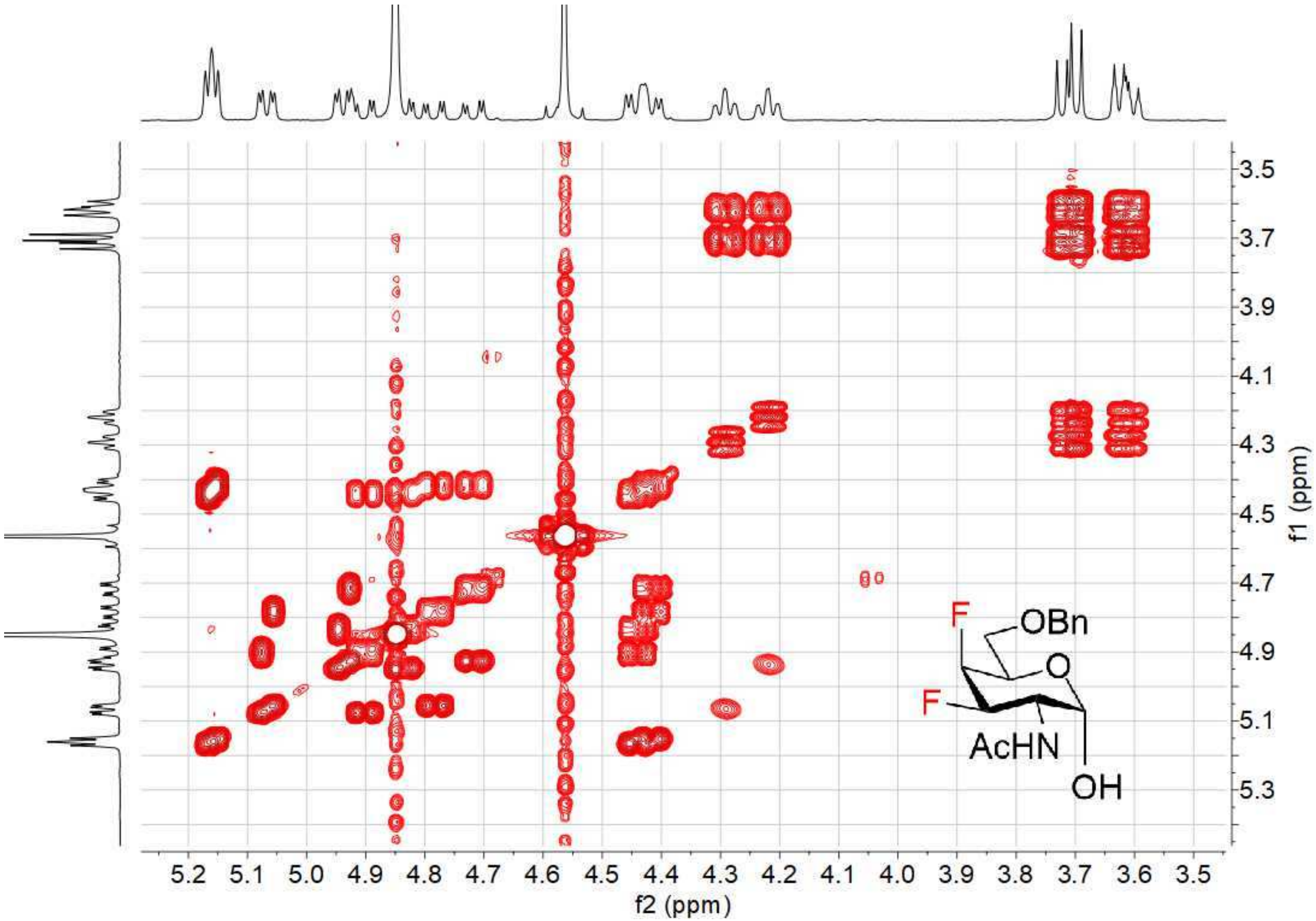
[13]C NMR (100 MHz, CD_3OD) 58



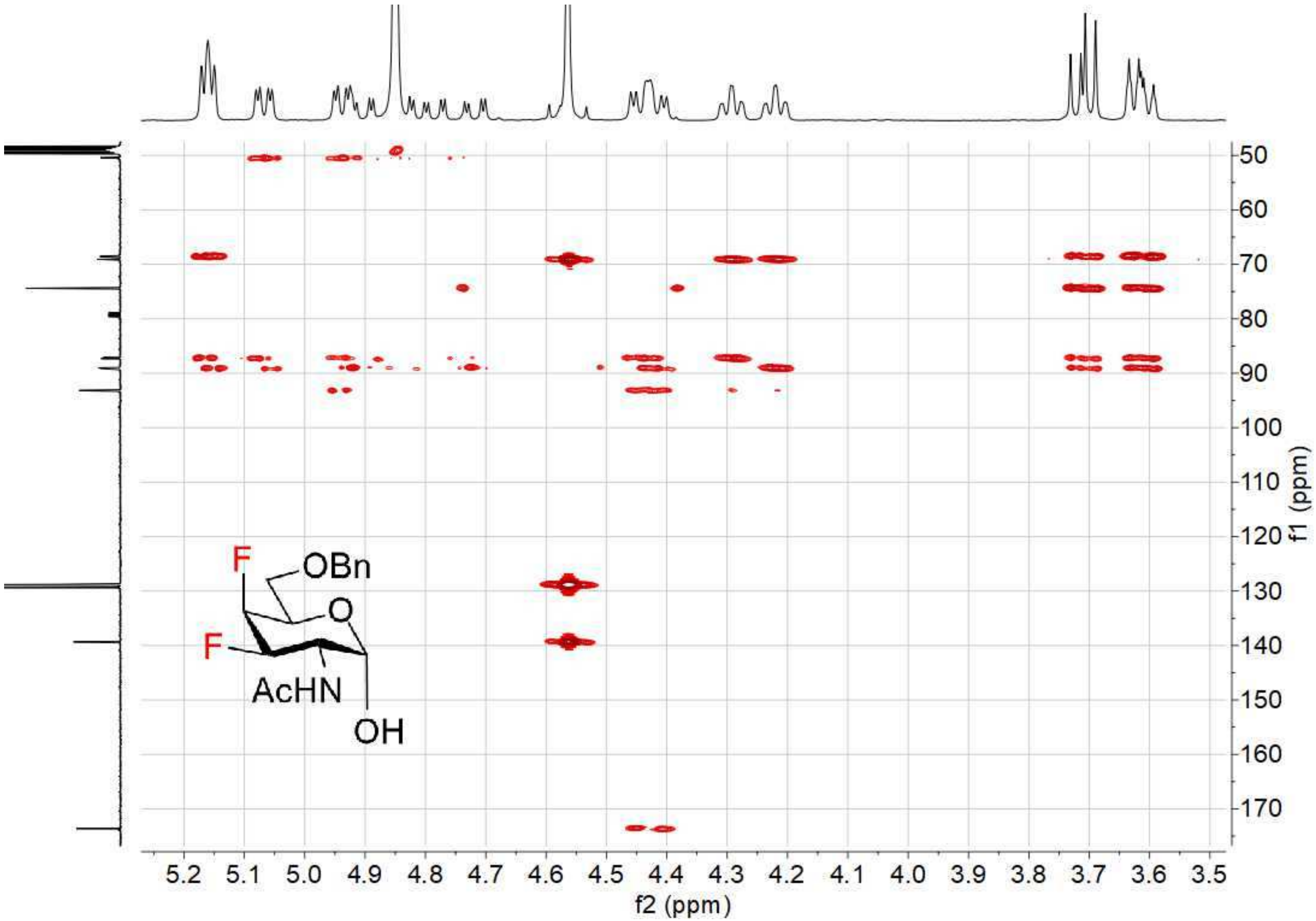
^{19}F NMR (376 MHz, CD_3OD) 58



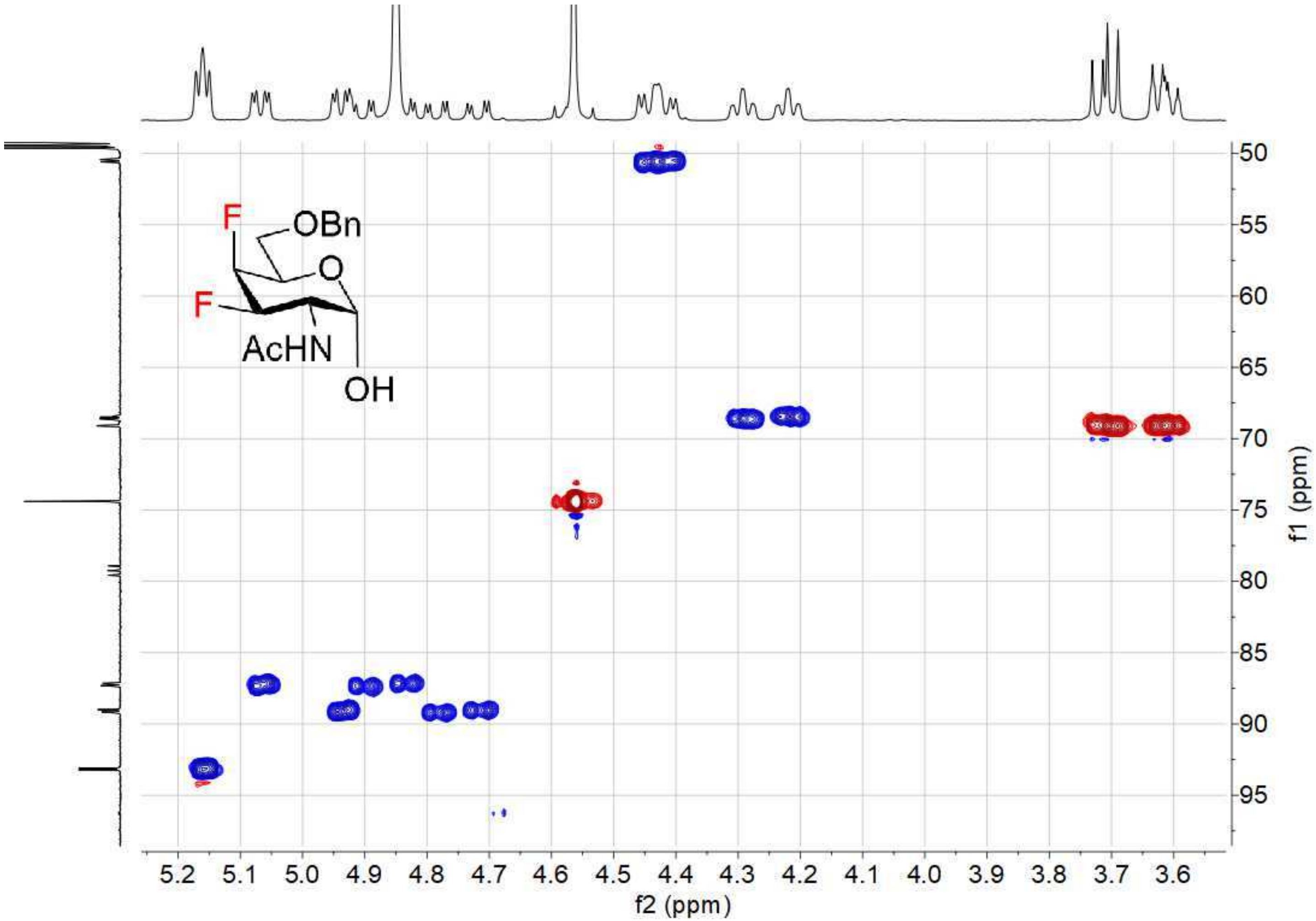
^{1}H-^{1}H COSY NMR 58



[1]H-[13]C HMBC 58

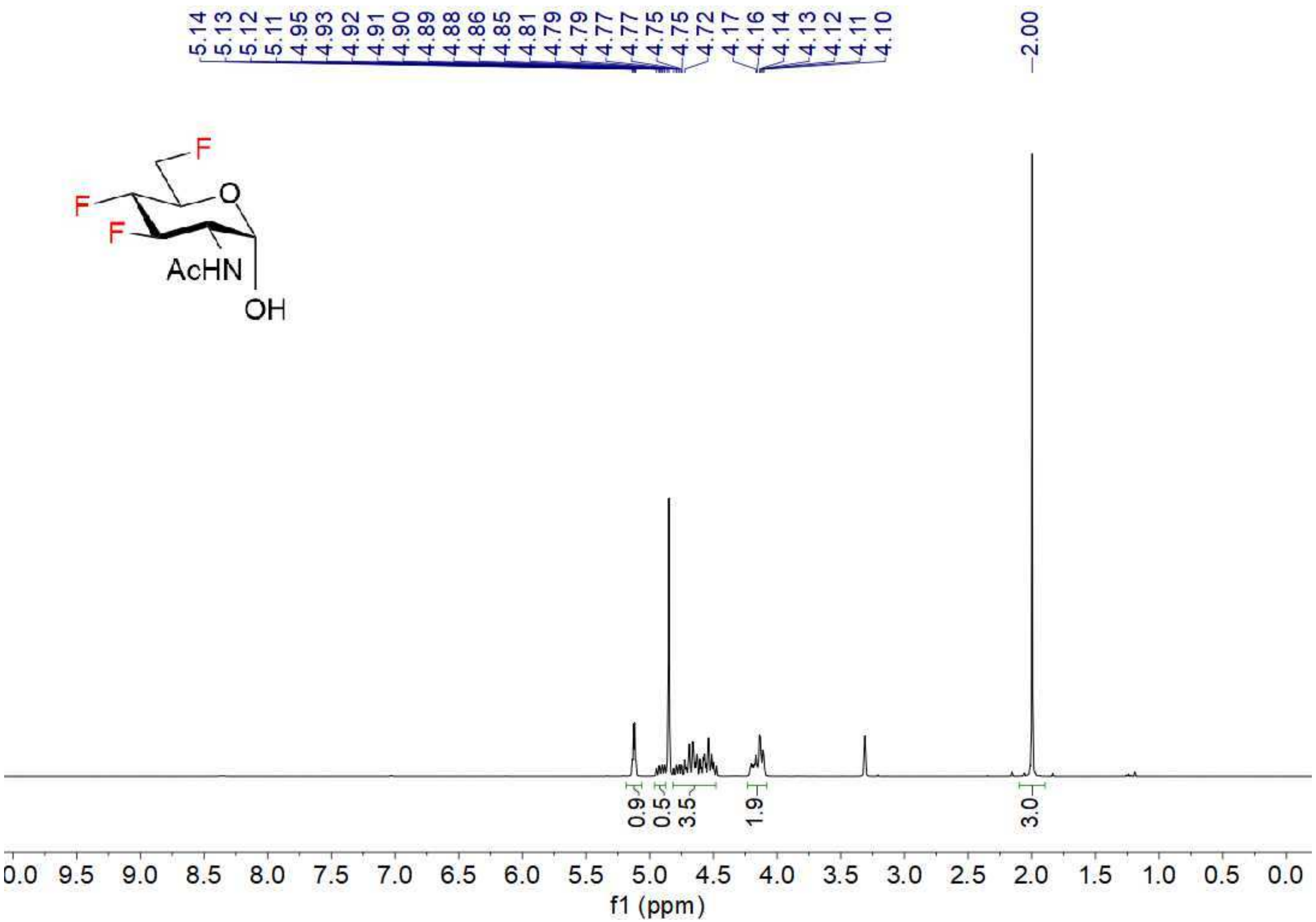


^{1}H-^{13}C HSQC 58

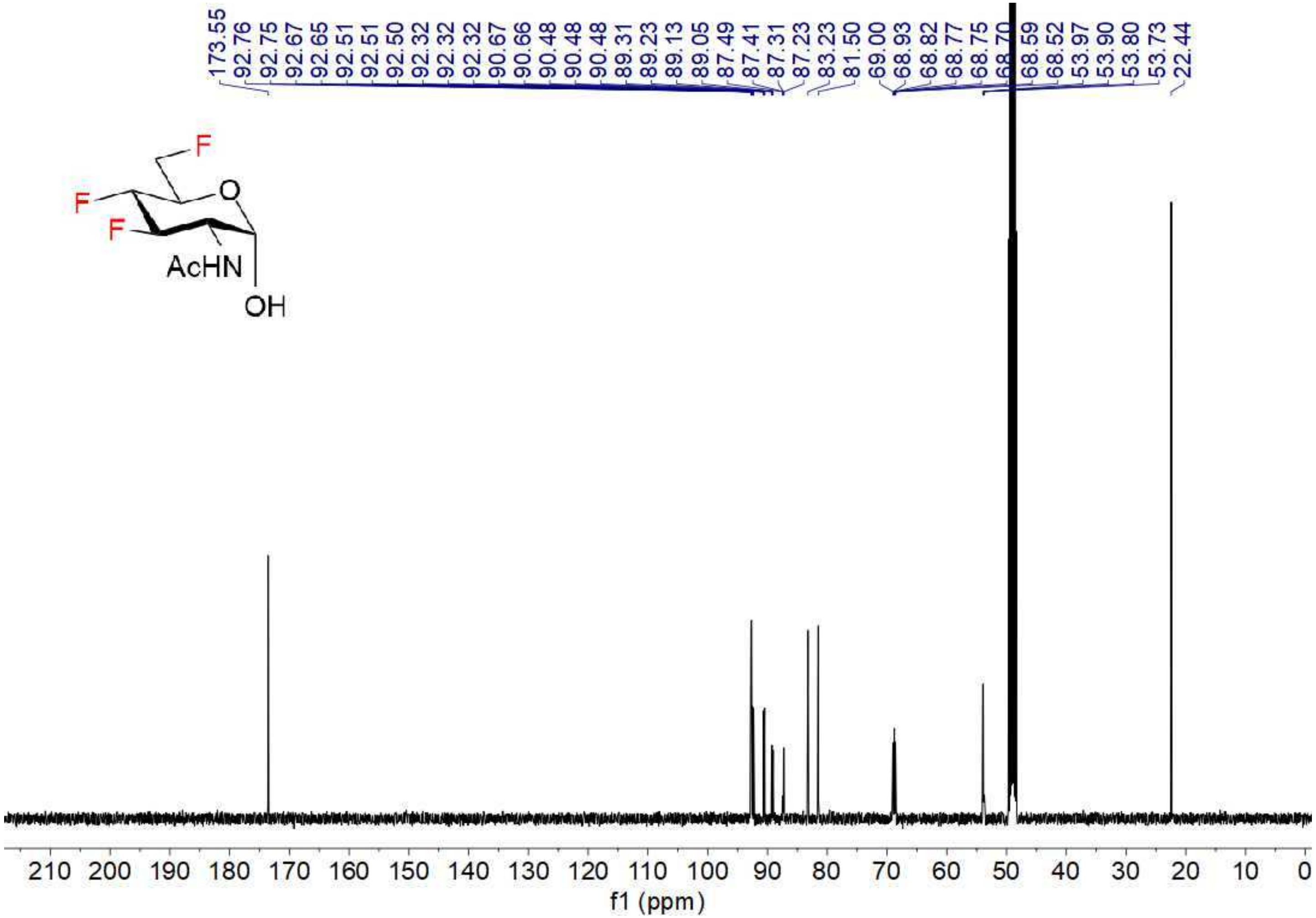


NMR COMPOUND 59

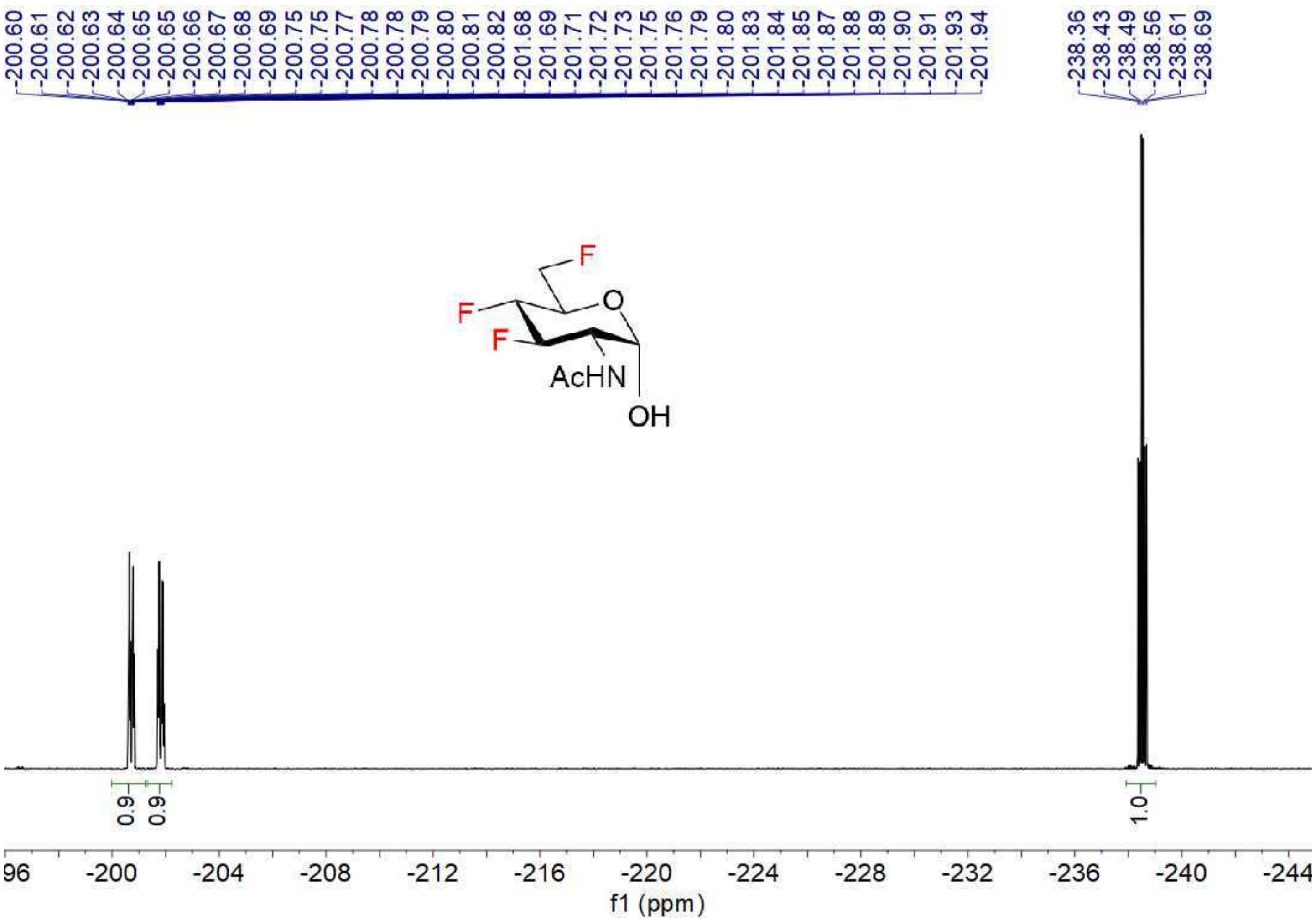
^{1}H NMR (400 MHz, CD_3OD) 59



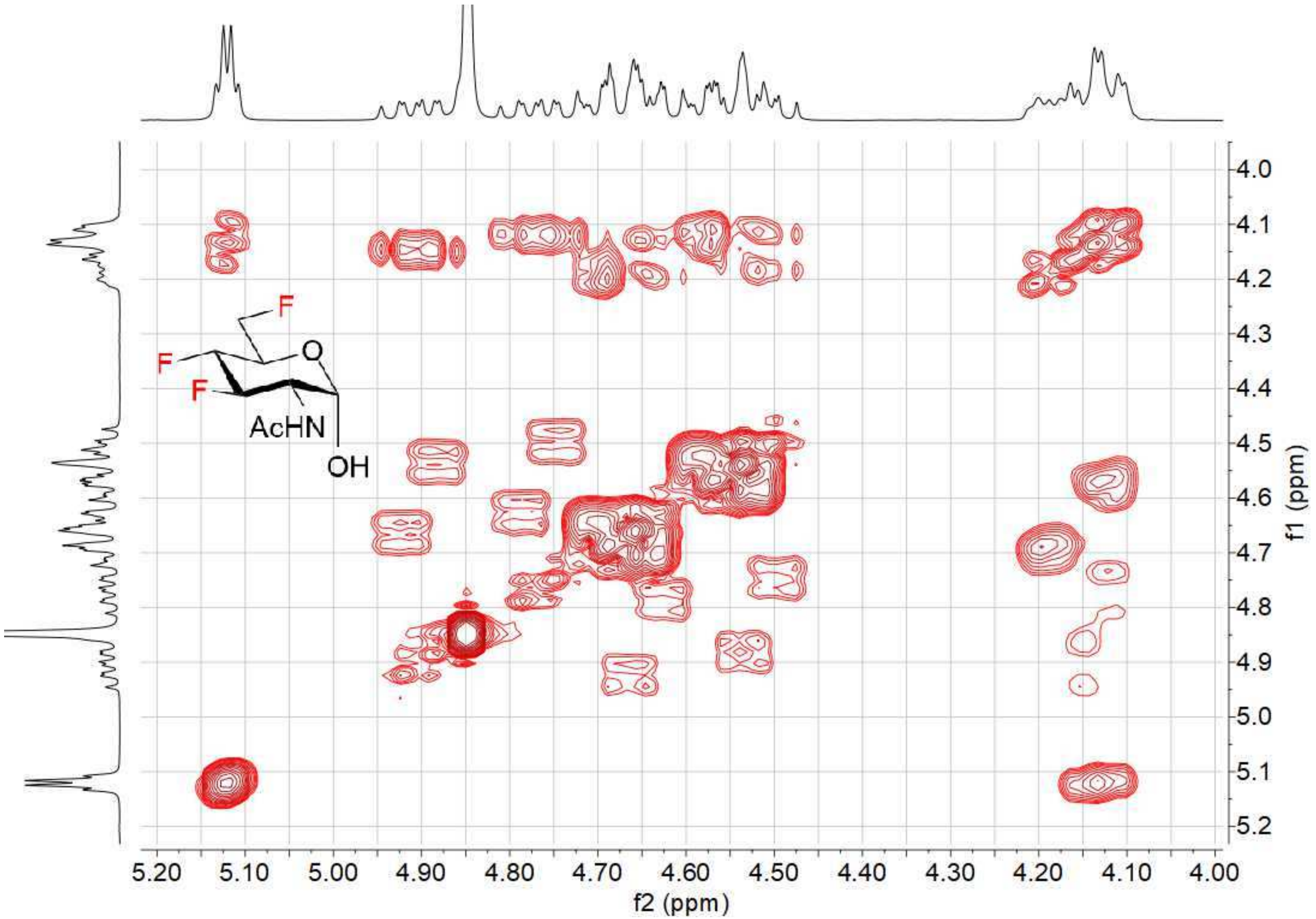
^{13}C NMR (100 MHz, CD_3OD) **59**



^{19}F NMR (376 MHz, CD_3OD) 59

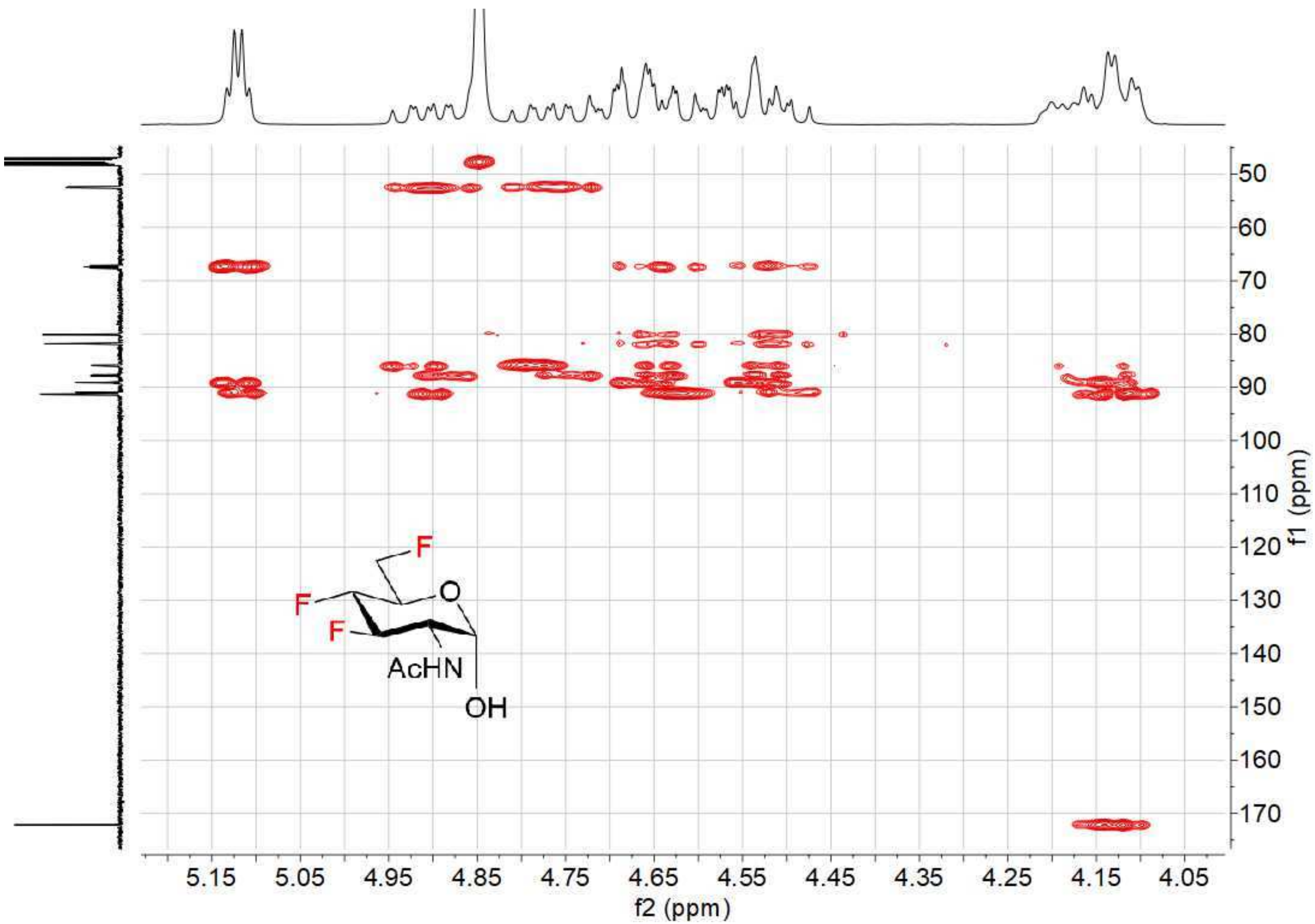


^{1}H-^{1}H COSY 59

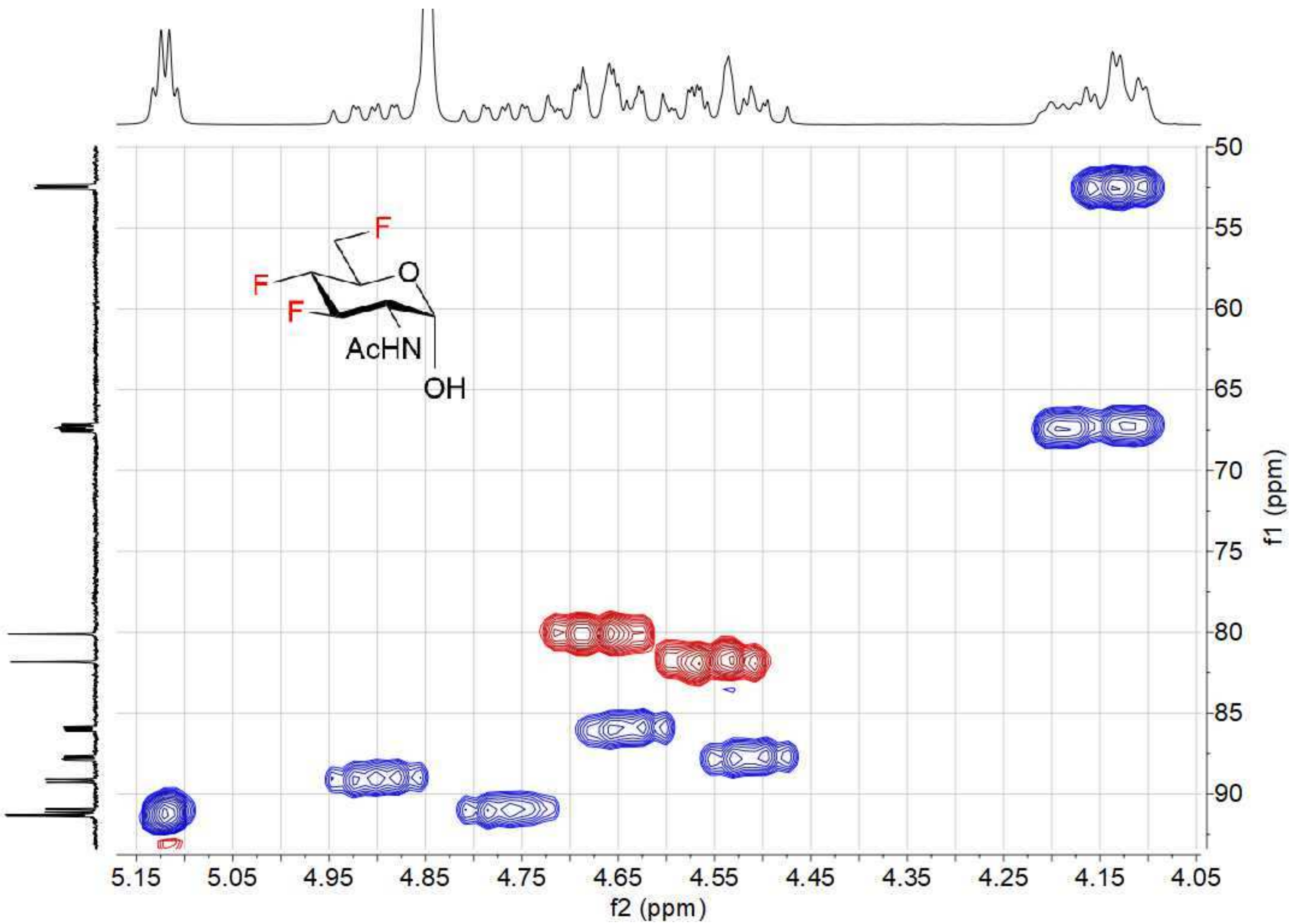


F
F
F
O
AcHN
OH
5.20 5.10 5.00 4.90 4.80 4.70 4.60 4.50 4.40 4.30 4.20 4.10 4.00
f2 (ppm)
4.0 4.1 4.2 4.3 4.4 4.5 4.6 4.7 4.8 4.9 5.0 5.1 5.2
f1 (ppm)

^{1}H-^{13}C HMBC 59

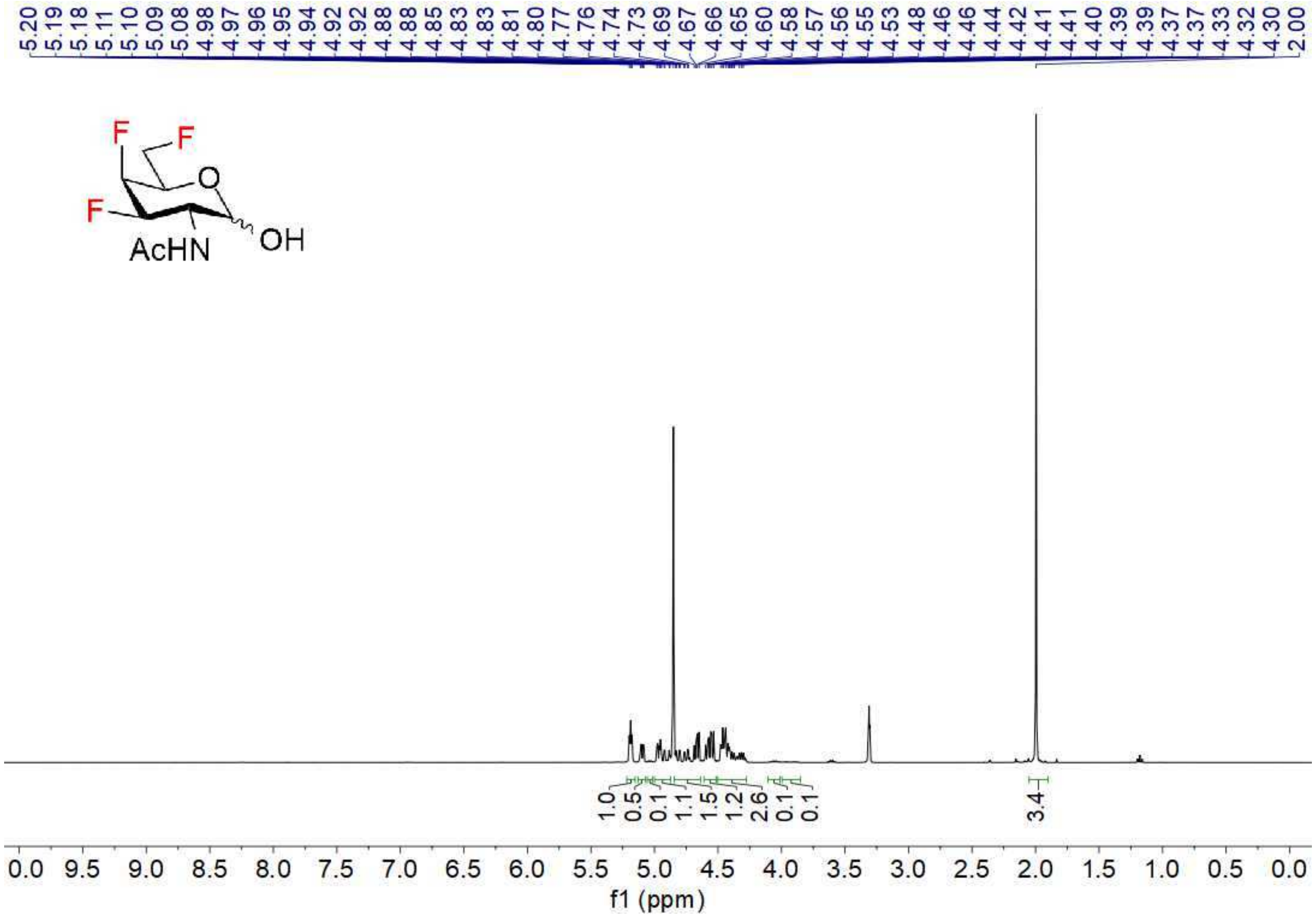


^{1}H-^{13}C HSQC 59

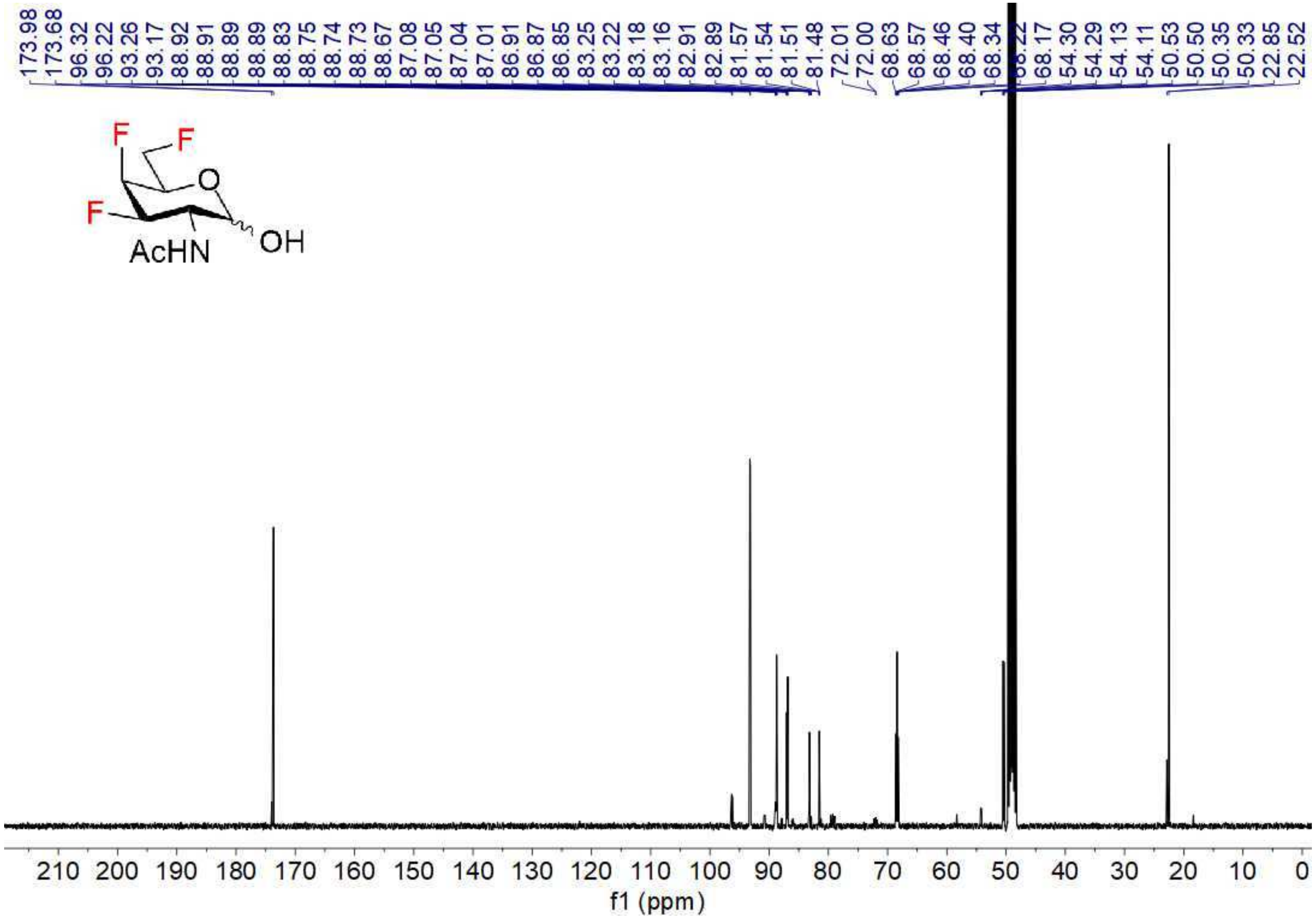


NMR COMPOUND 60

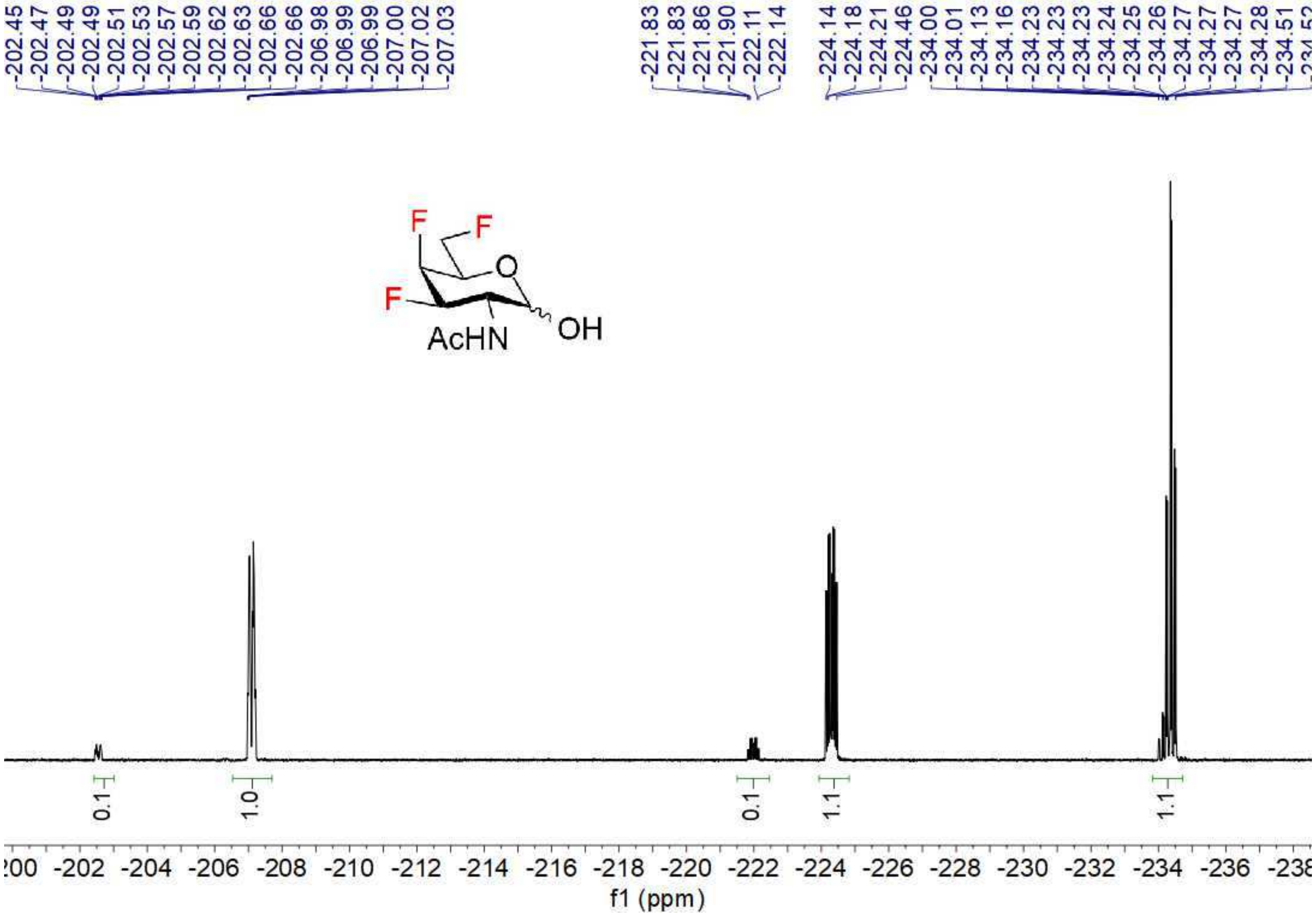
^{1}H NMR (400 MHz, CD_3OD) 60 (α/β ca. 10/1)



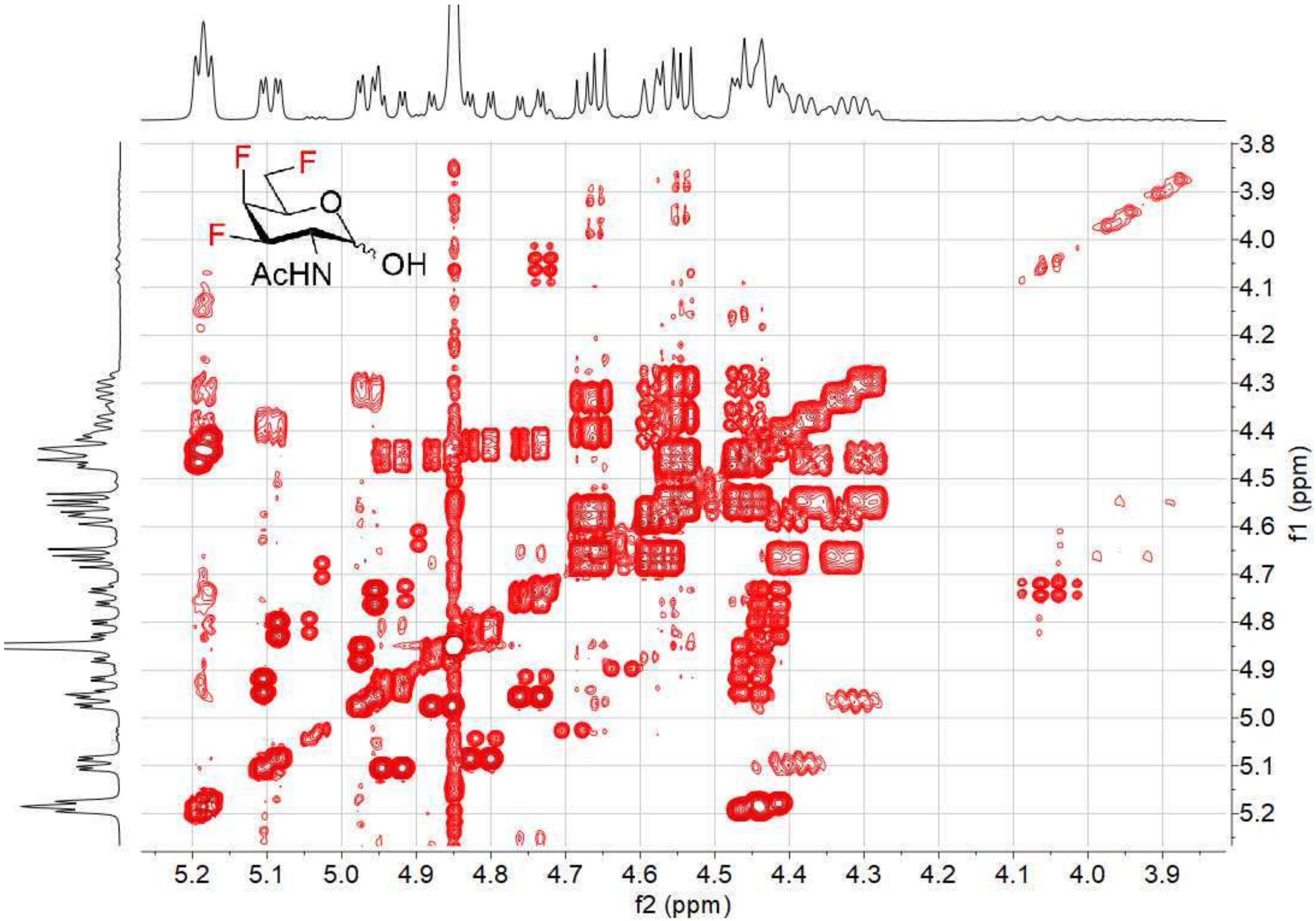
[13]C NMR (100 MHz, CD_3OD) 60



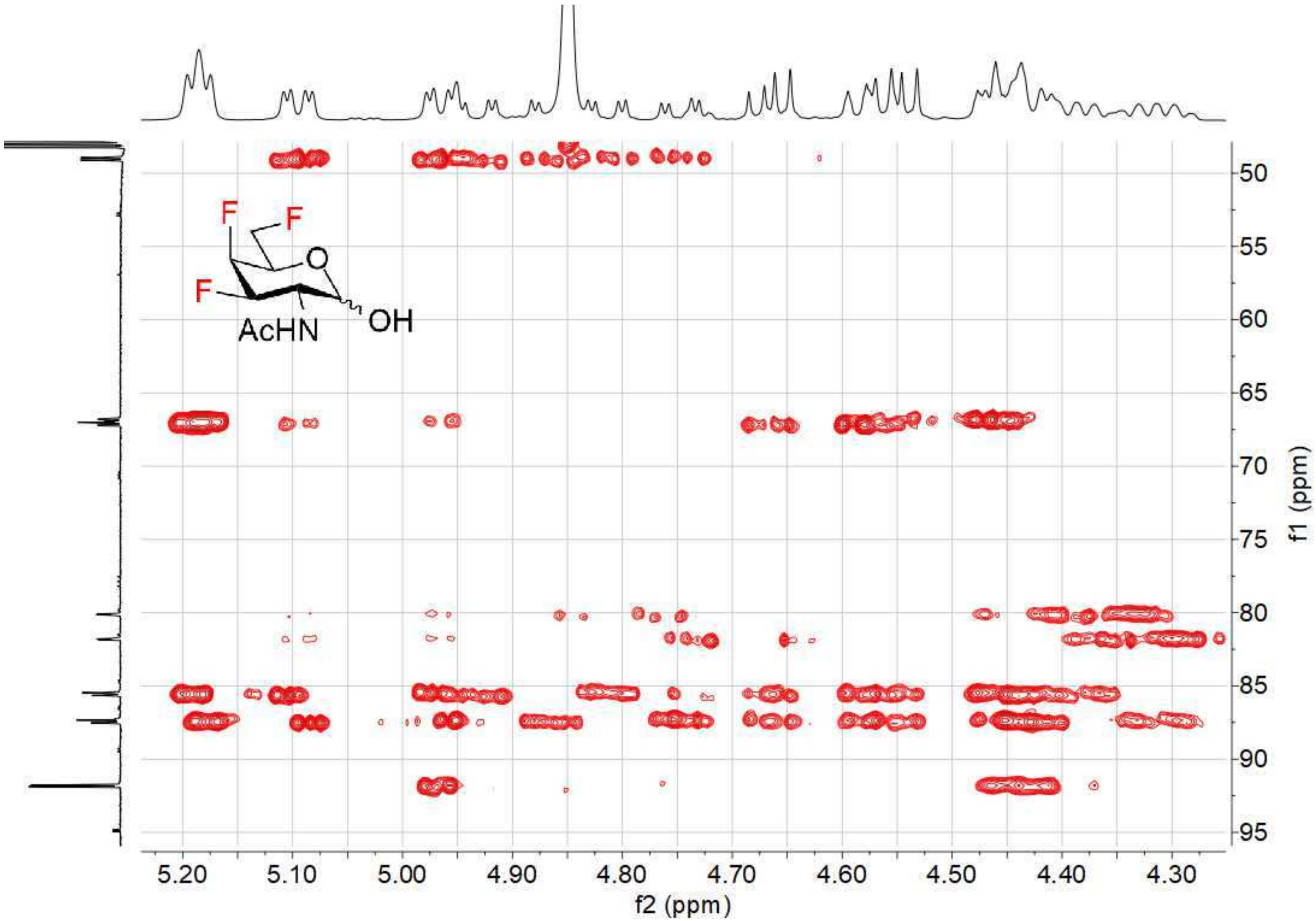
^{19}F NMR (376 MHz, CD_3OD) 60 (α/β ca. 10/1)



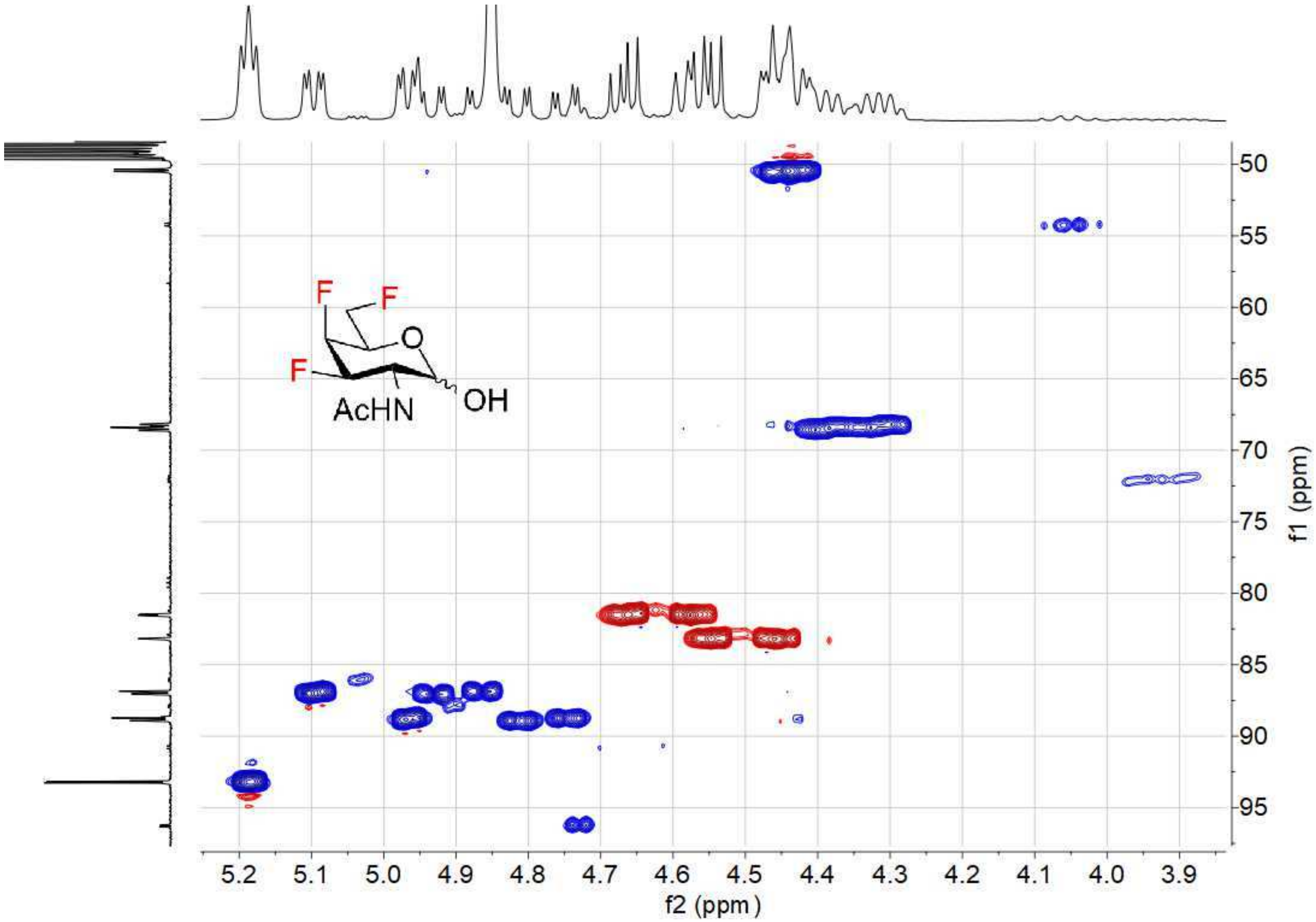
^{1}H-^{1}H COSY 60


F
F
F
O
AcHN
OH
5.2
5.1
5.0
4.9
4.8
4.7
4.6
4.5
4.4
4.3
4.2
4.1
4.0
3.9
f2 (ppm)
3.8
3.9
4.0
4.1
4.2
4.3
4.4
4.5
4.6
4.7
4.8
4.9
5.0
5.1
5.2
f1 (ppm)

^{1}H-^{13}C HMBC 60

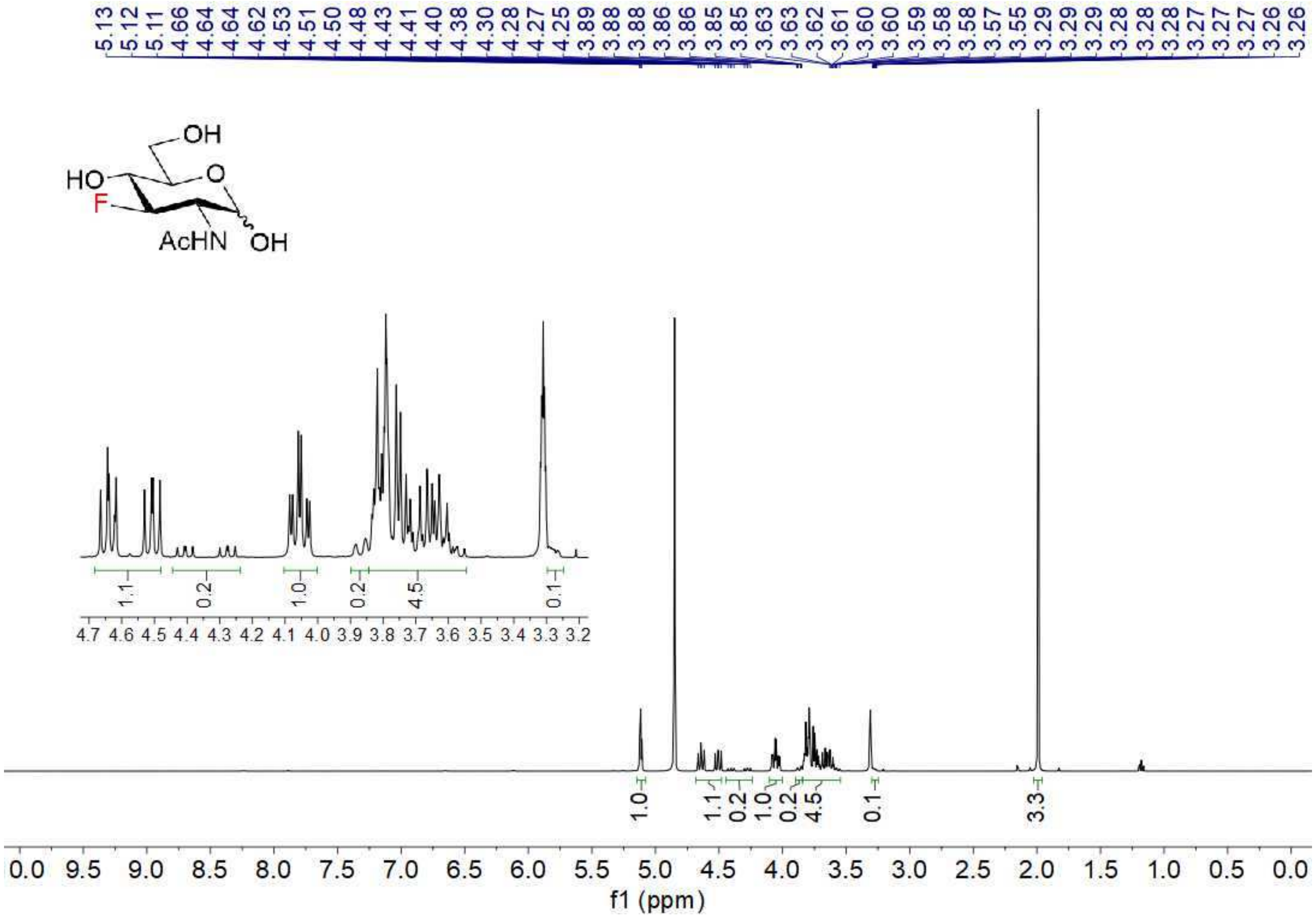


^{1}H-^{13}C HSQC 60

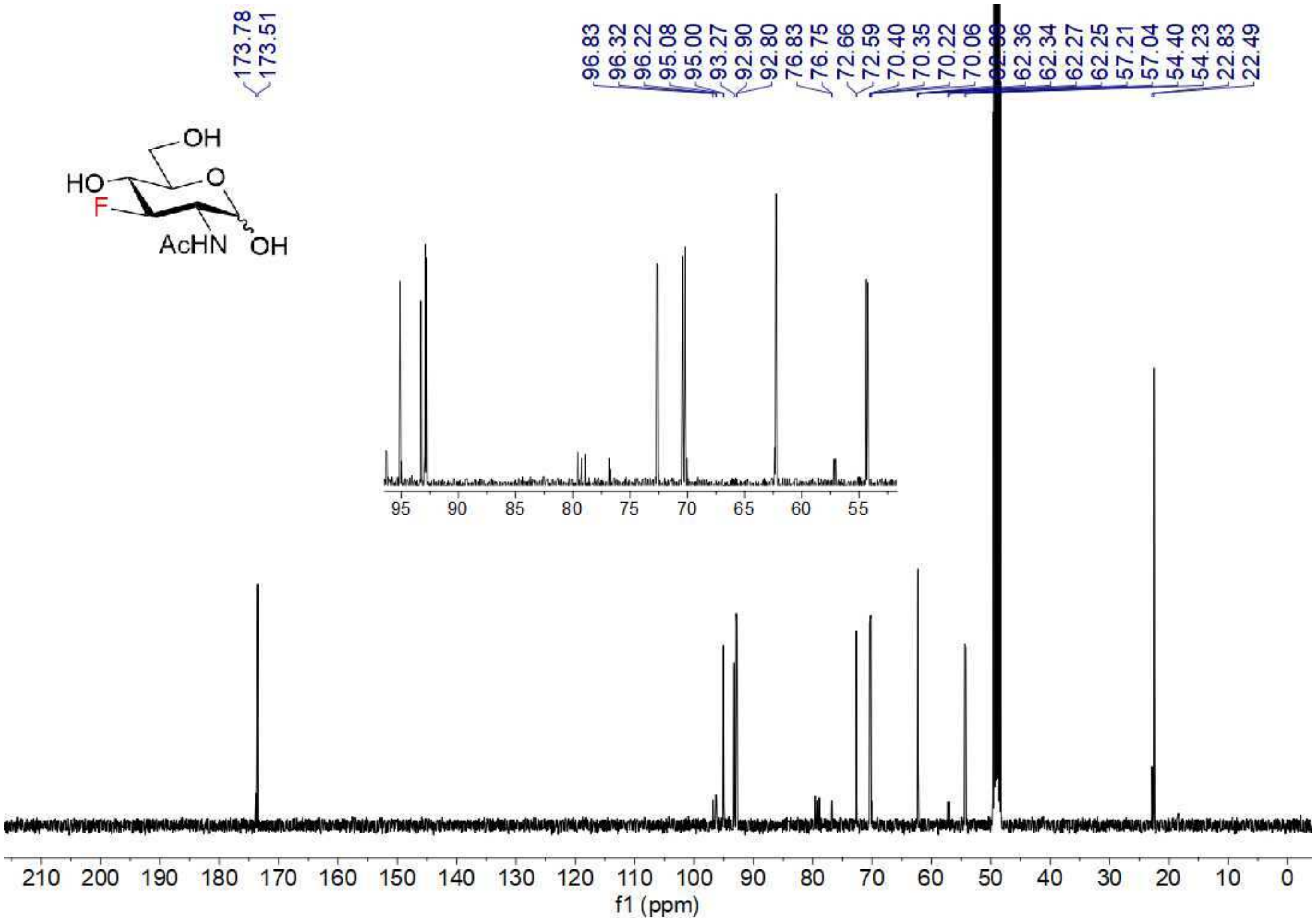


NMR COMPOUND 61

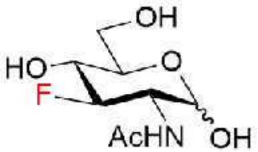
^{1}H NMR (400 MHz, CD_3OD) 61 (α/β ca. 10/1)

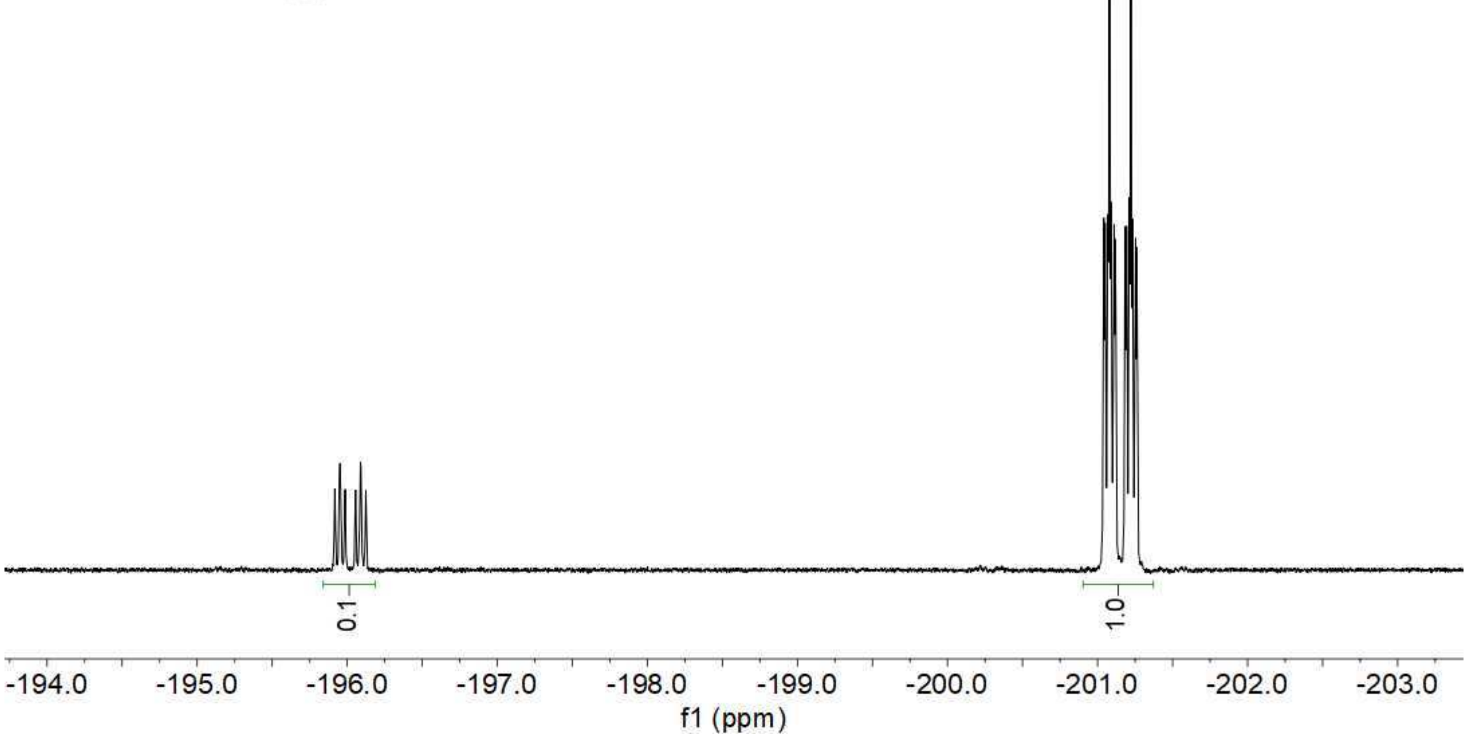


^{13}C NMR (100 MHz, CD_3OD) 61

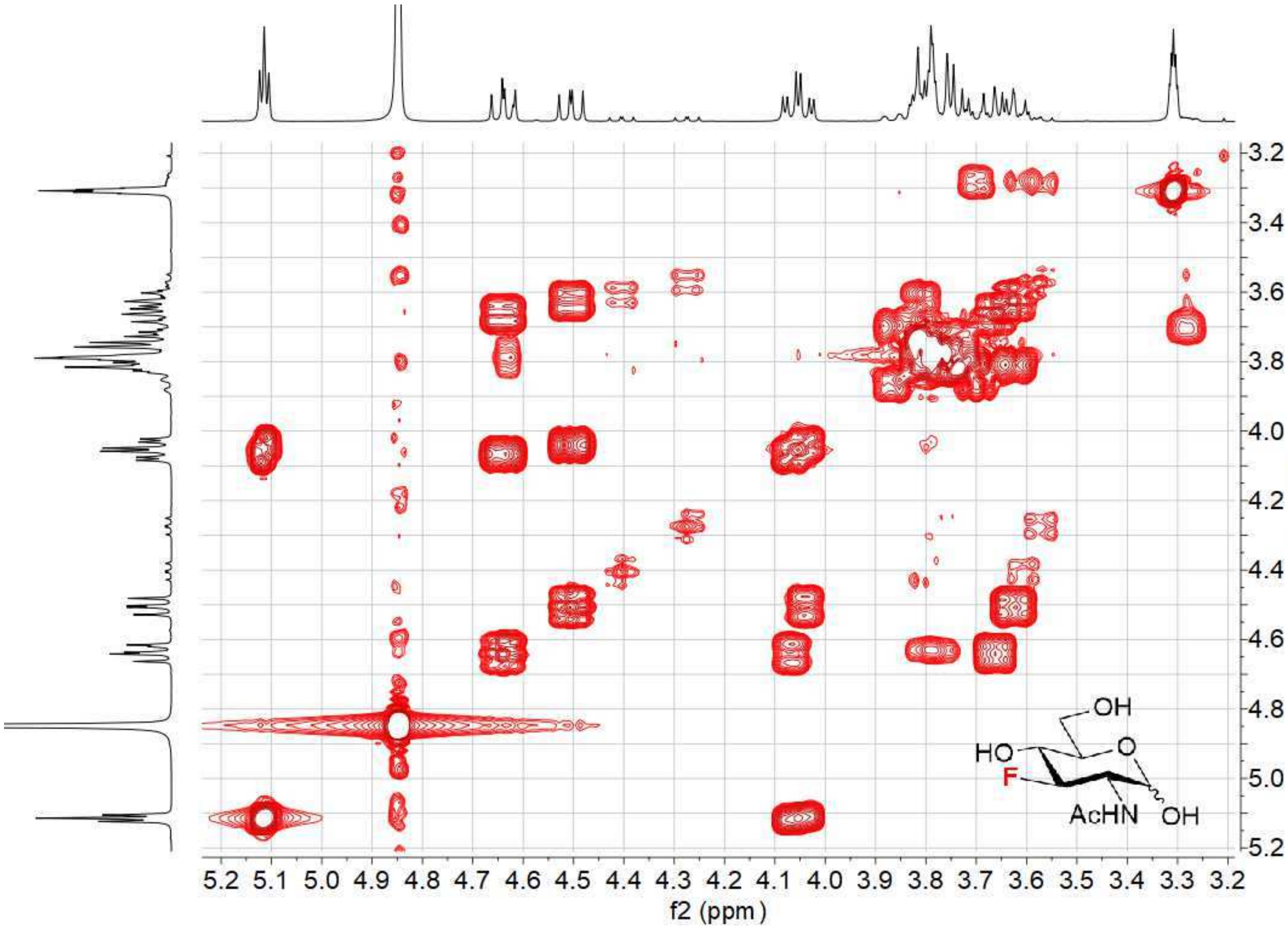


^{19}F NMR (376 MHz, CD_3OD) 61 (α/β ca. 10/1)

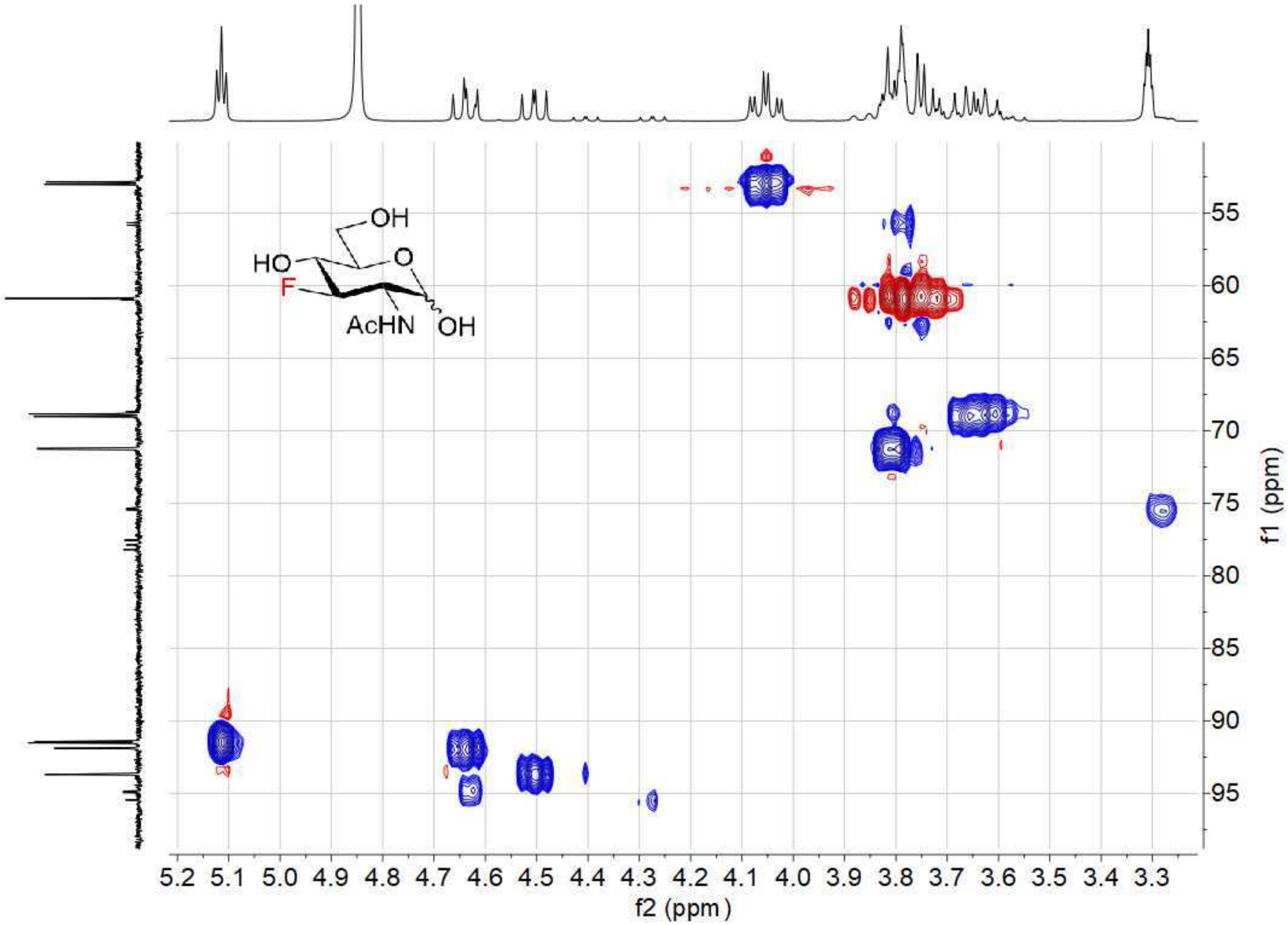




^{1}H-^{1}H COSY 61

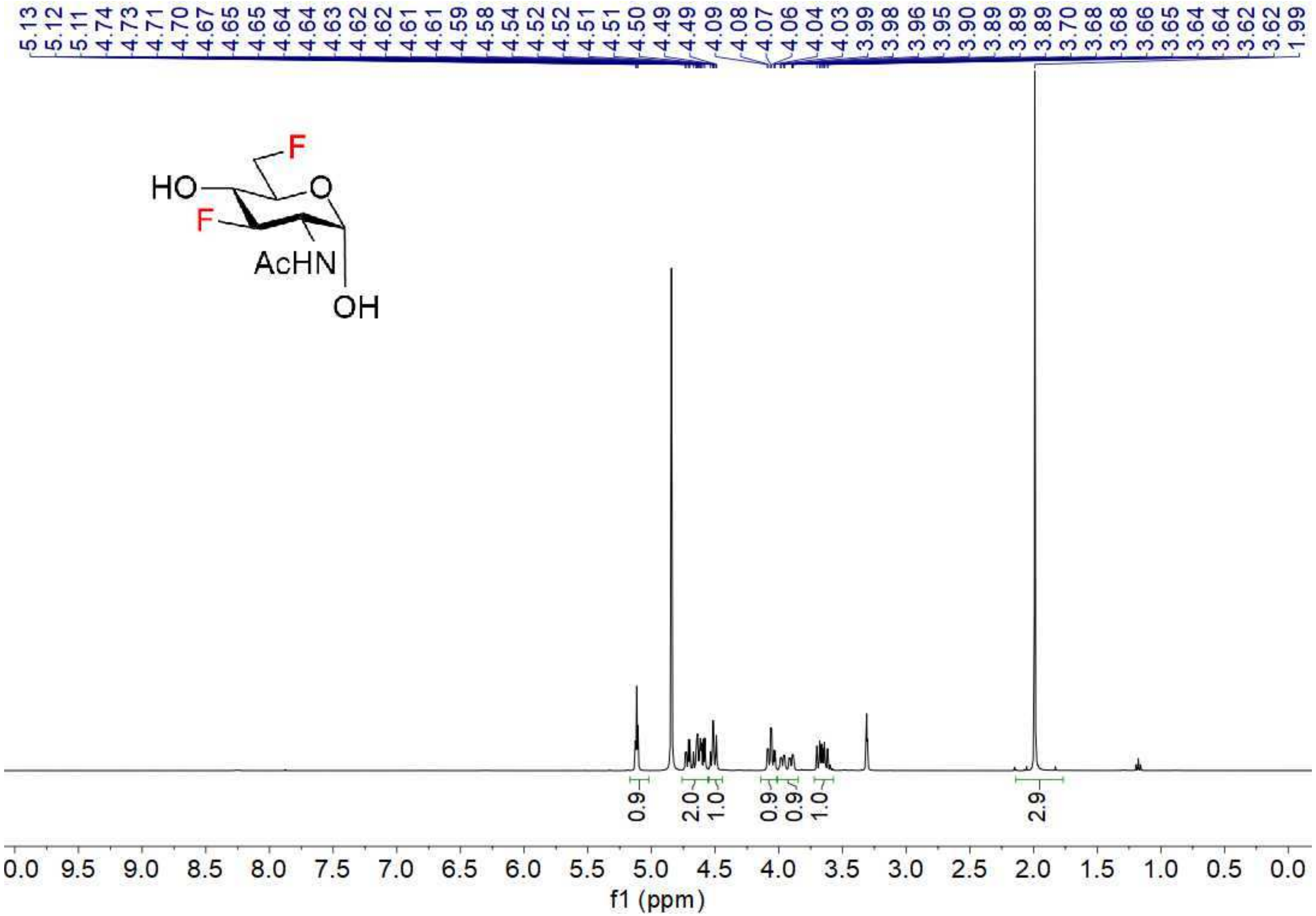


^{1}H-^{13}C HSQC 61

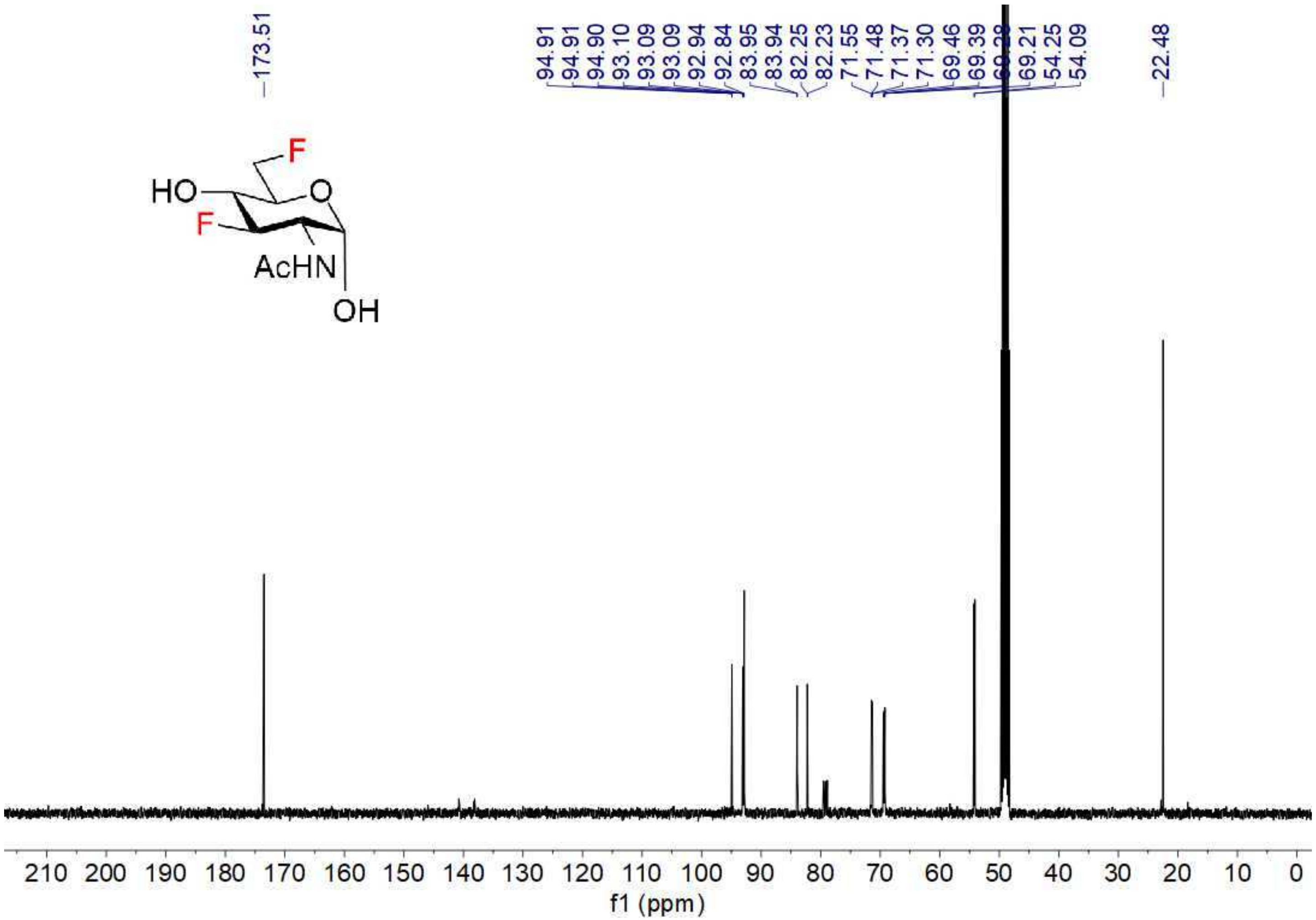


NMR COMPOUND 62

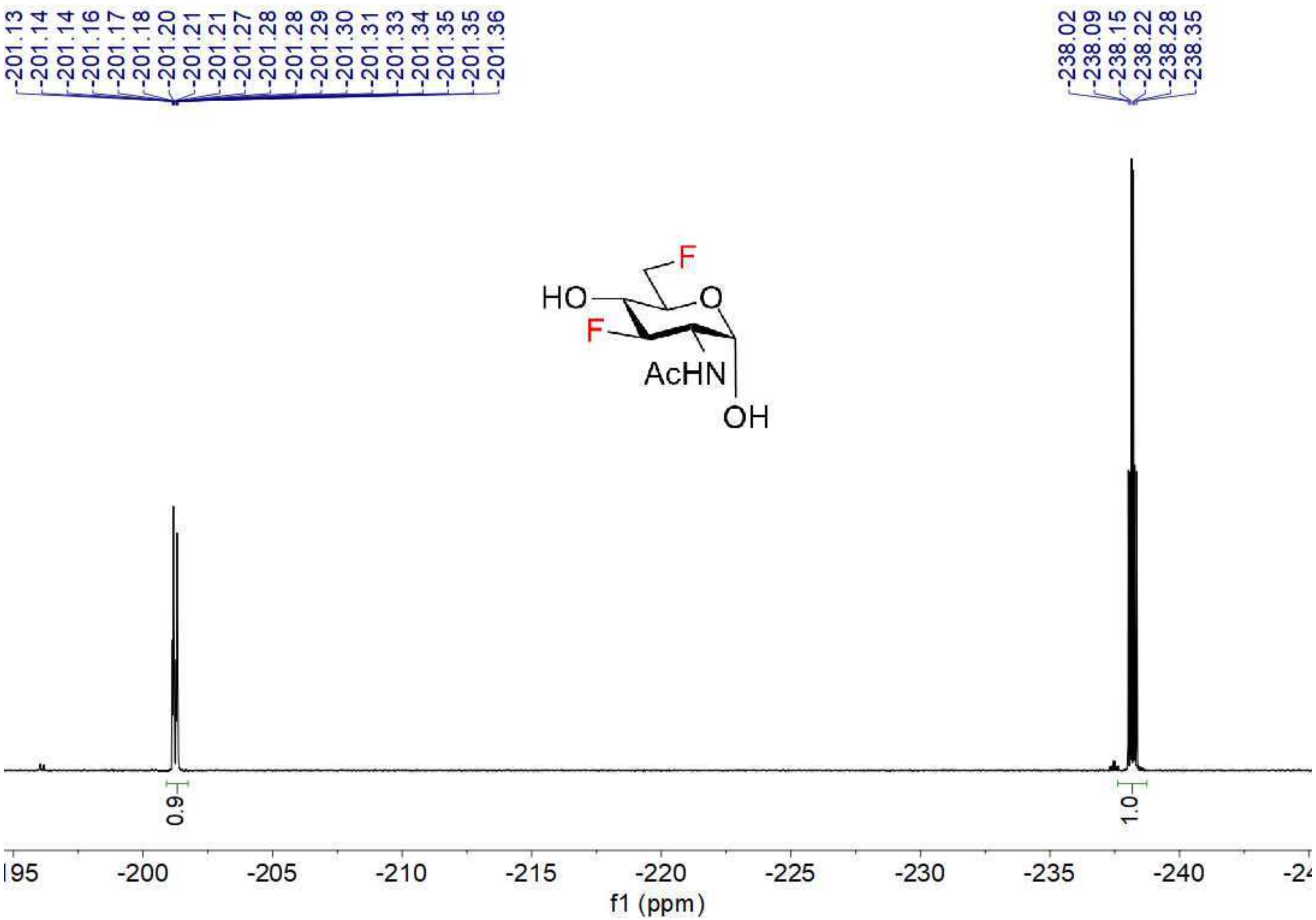
^{1}H NMR (400 MHz, CD_3OD) 62



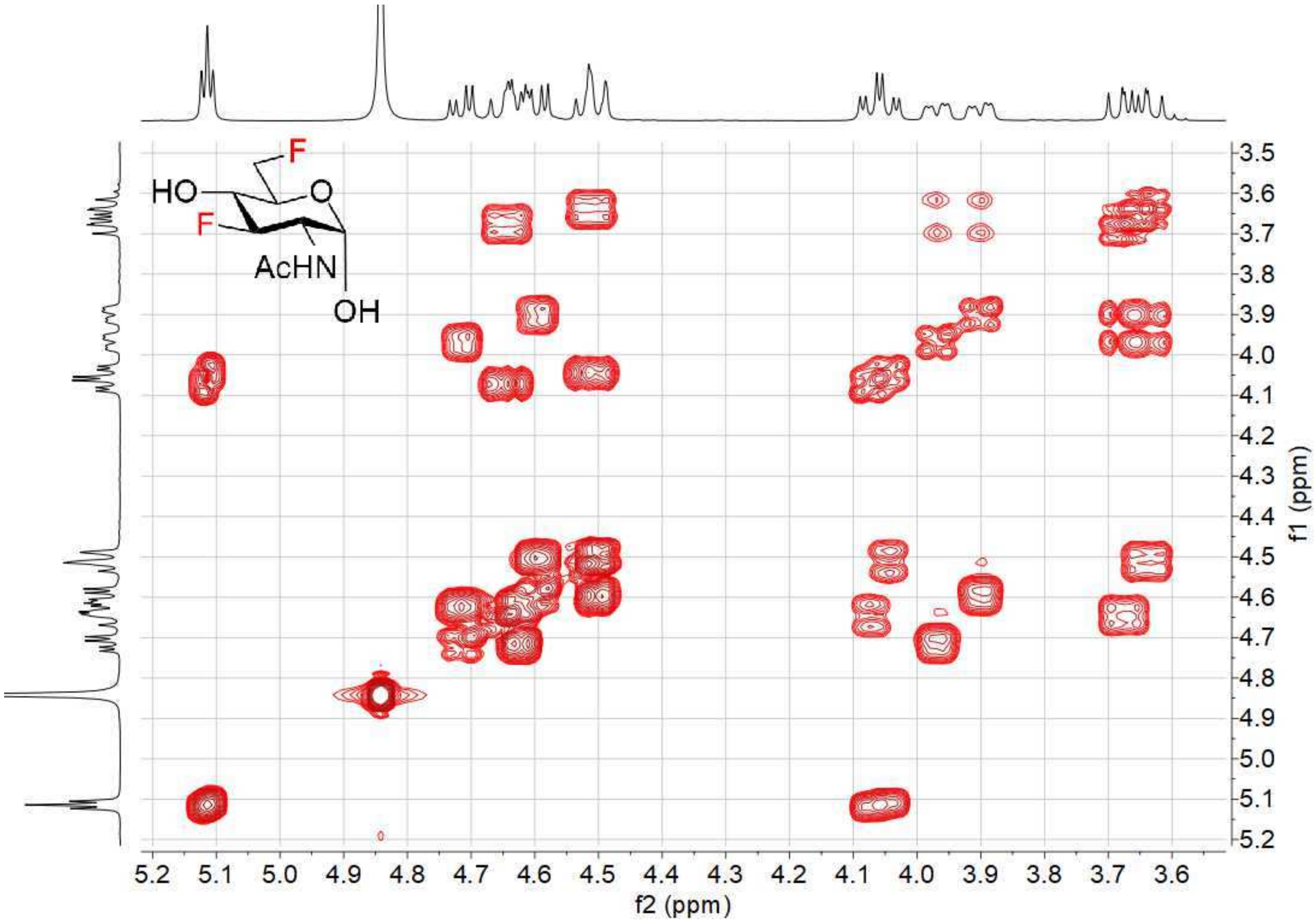
^{13}C NMR (100 MHz, CD_3OD) 62



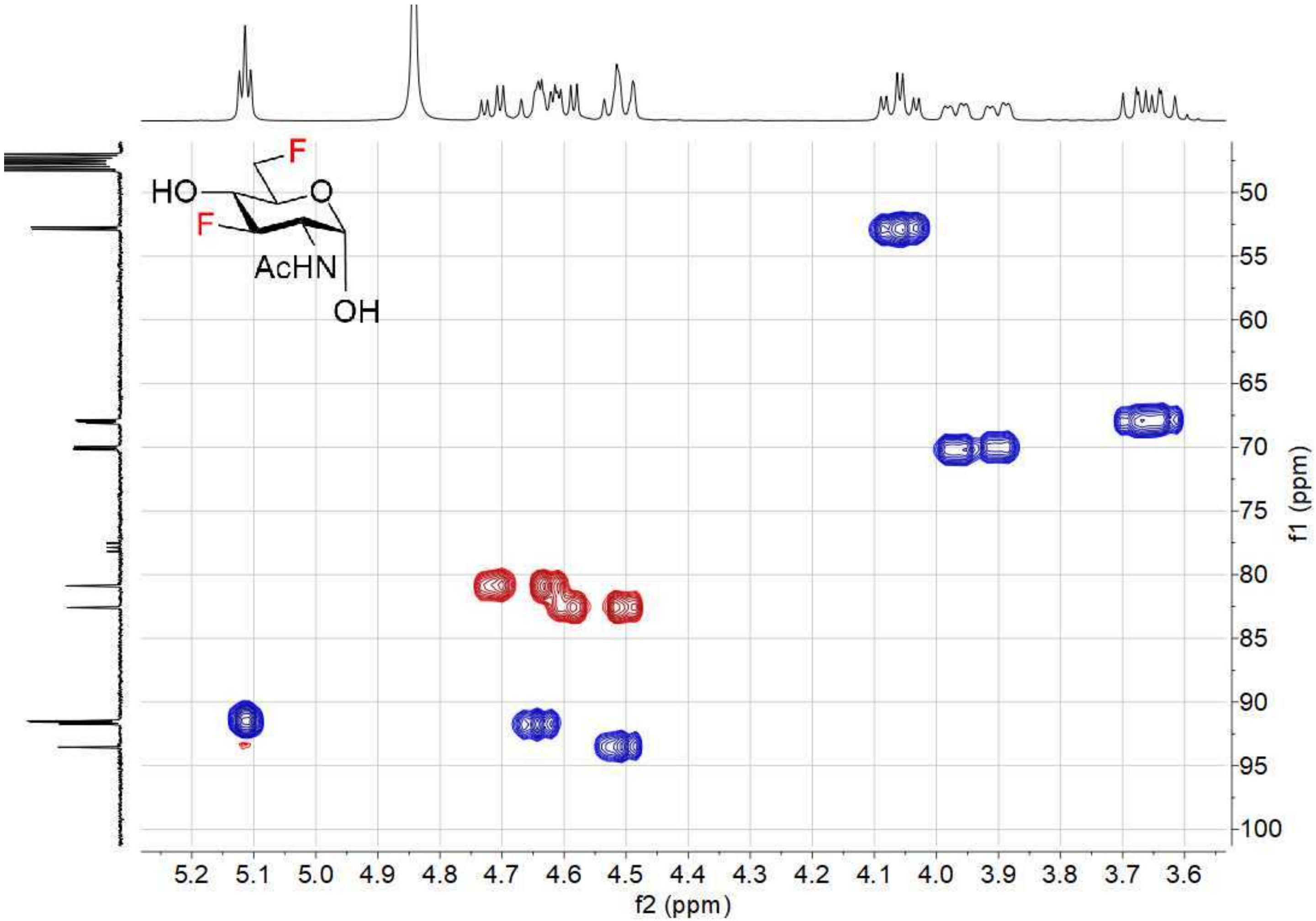
^{19}F NMR (376 MHz, CD$_3$OD) 62



^{1}H-^{1}H COSY 62

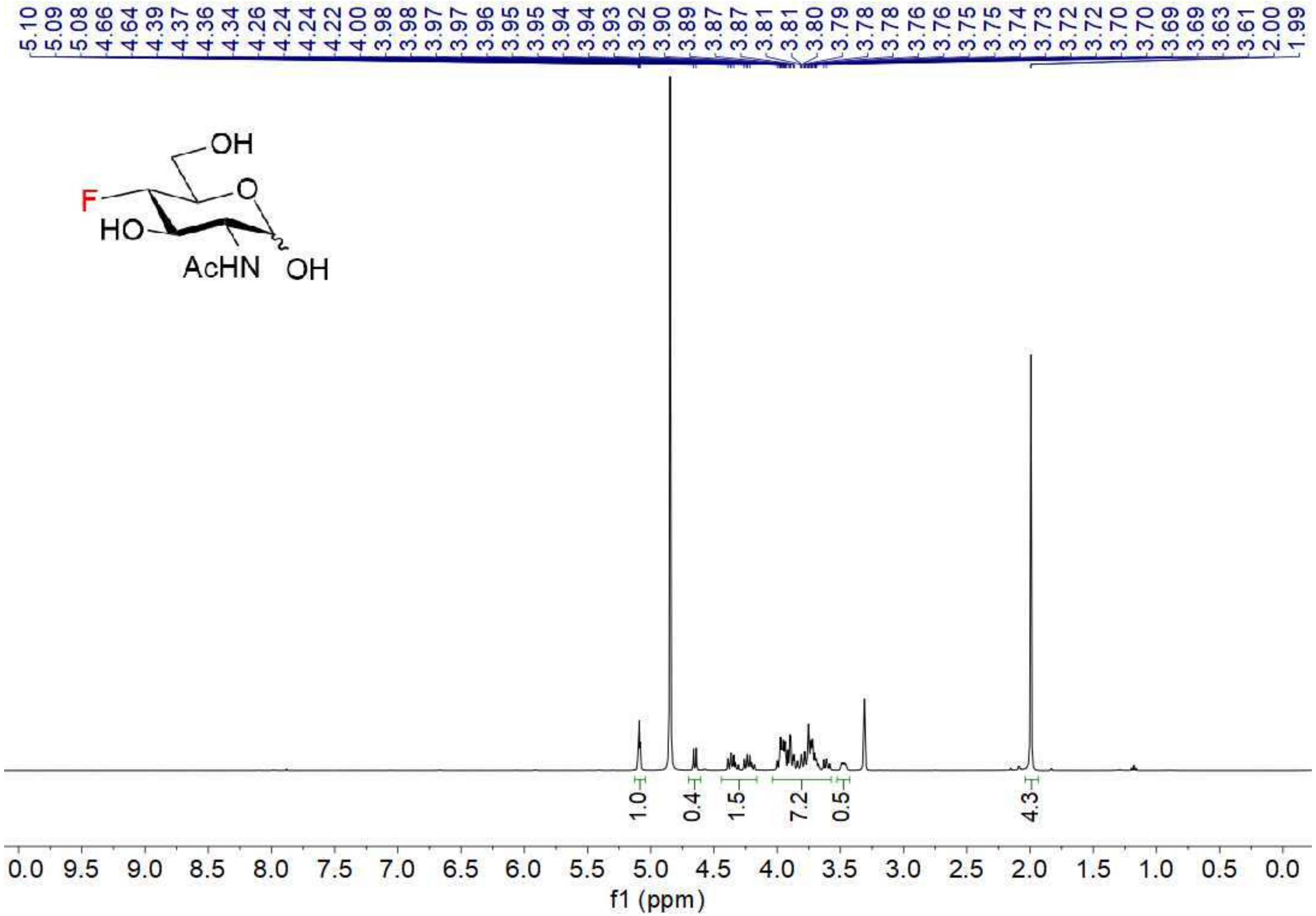

HO
F
F
O
AcHN
OH
f2 (ppm)
f1 (ppm)
5.2 5.1 5.0 4.9 4.8 4.7 4.6 4.5 4.4 4.3 4.2 4.1 4.0 3.9 3.8 3.7 3.6
3.5 3.6 3.7 3.8 3.9 4.0 4.1 4.2 4.3 4.4 4.5 4.6 4.7 4.8 4.9 5.0 5.1 5.2

^{1}H-^{13}C HSQC 62

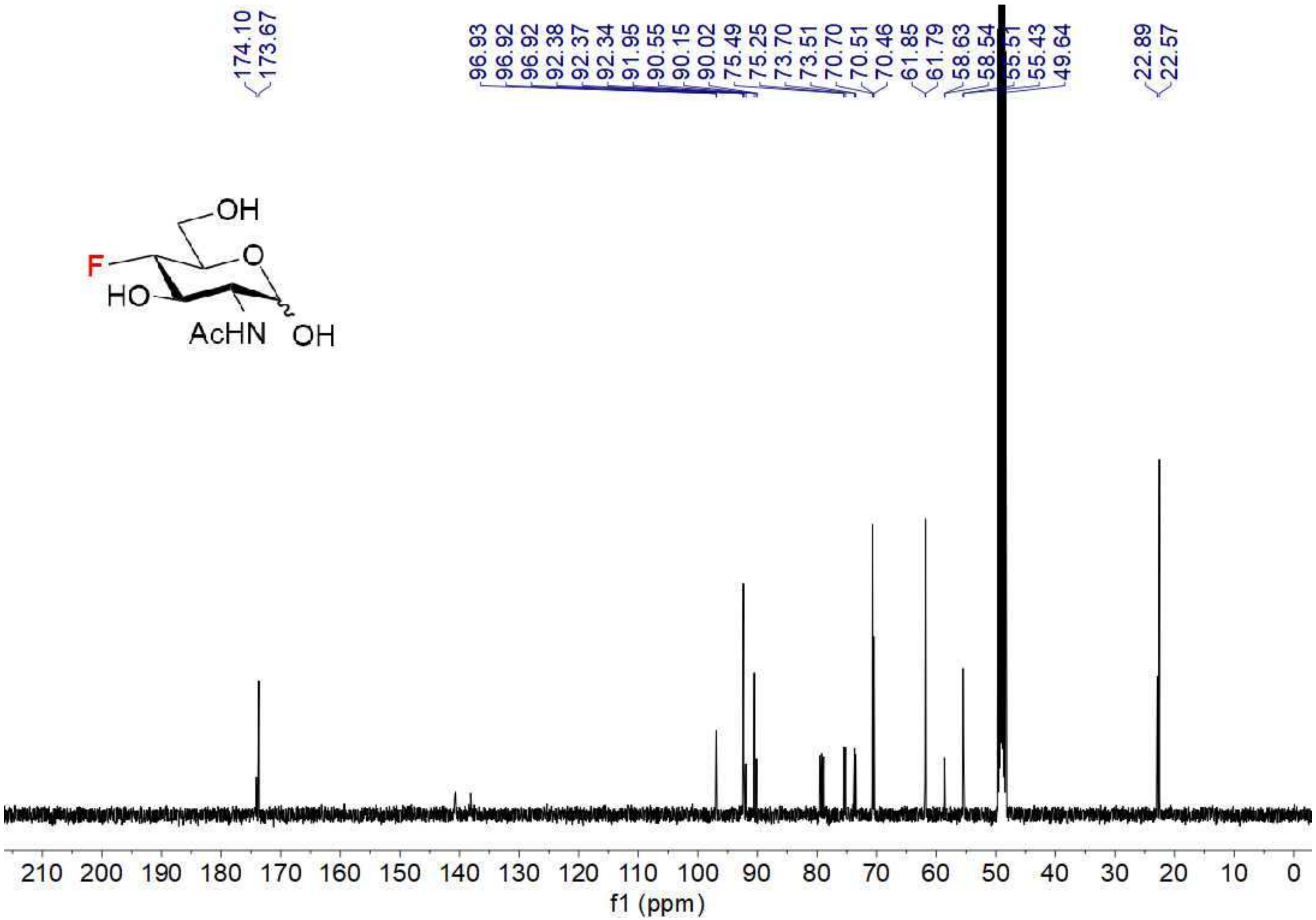


NMR COMPOUND 63

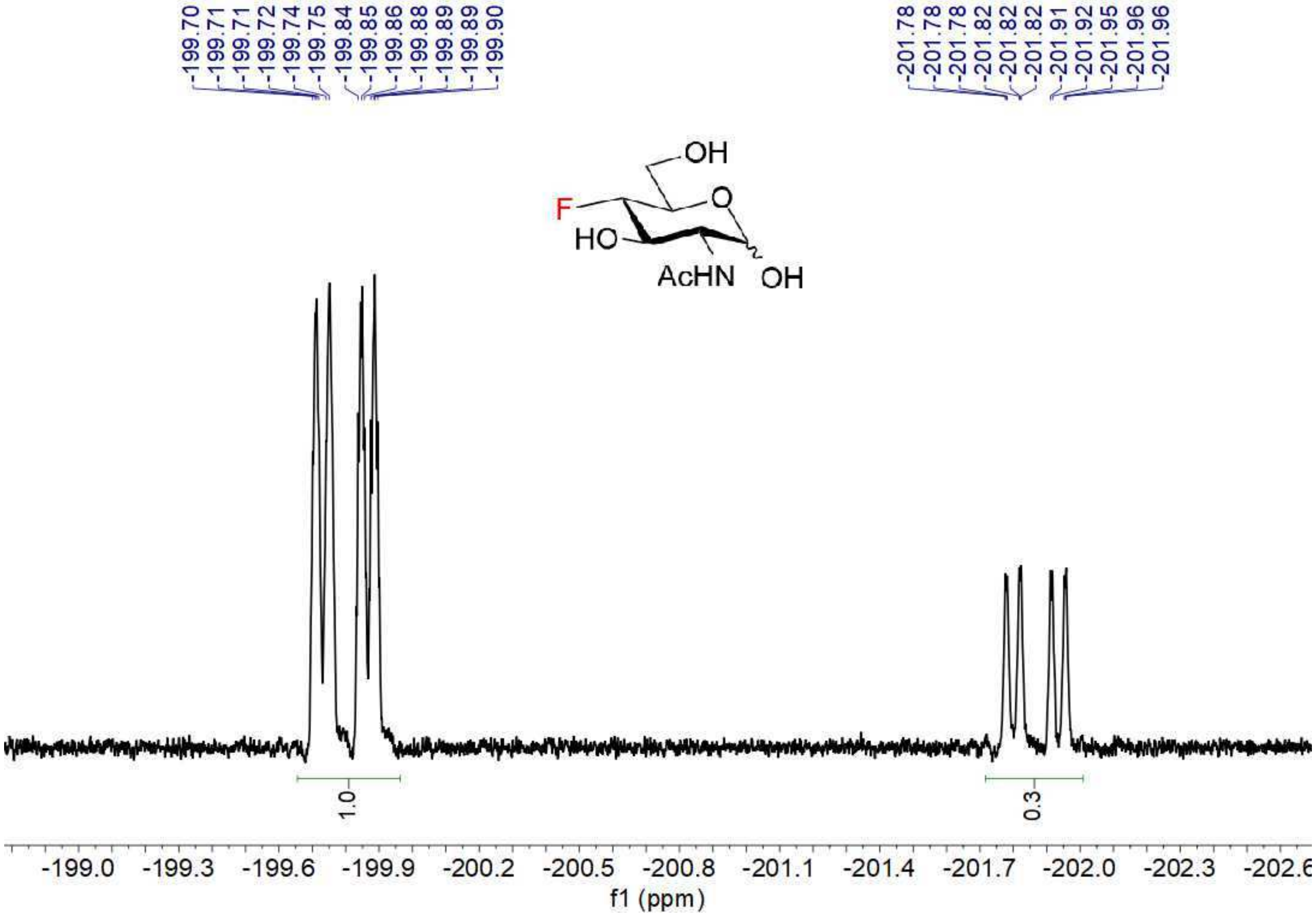
[1]H NMR (400 MHz, CD_3OD) 63 (α/β ca. 10/3)



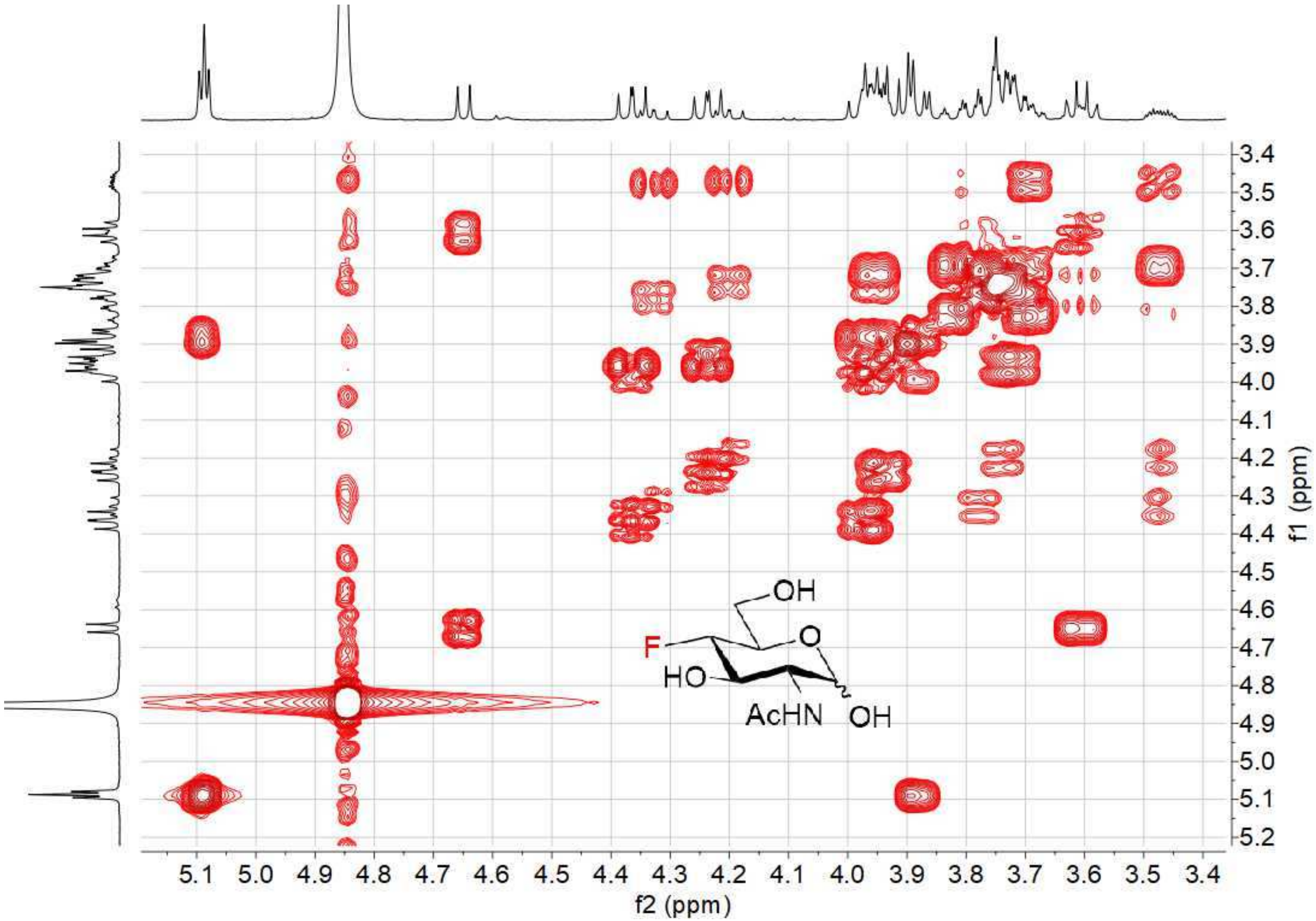
^{13}C NMR (100 MHz, CD_3OD) 63



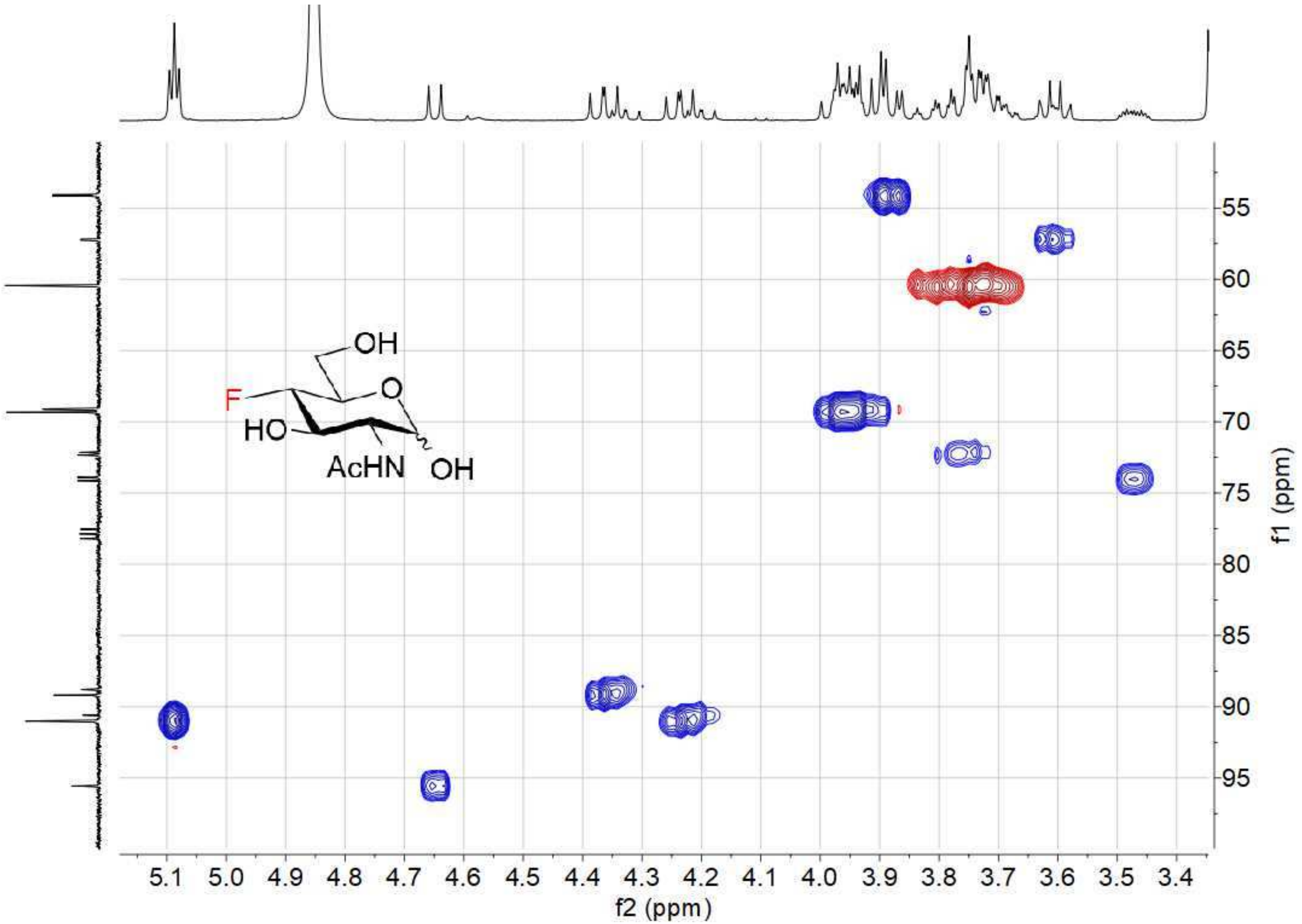
^{19}F NMR (376 MHz, CD$_3$OD) 63 (α/β ca. 10/3)



^{1}H-^{1}H COSY 63

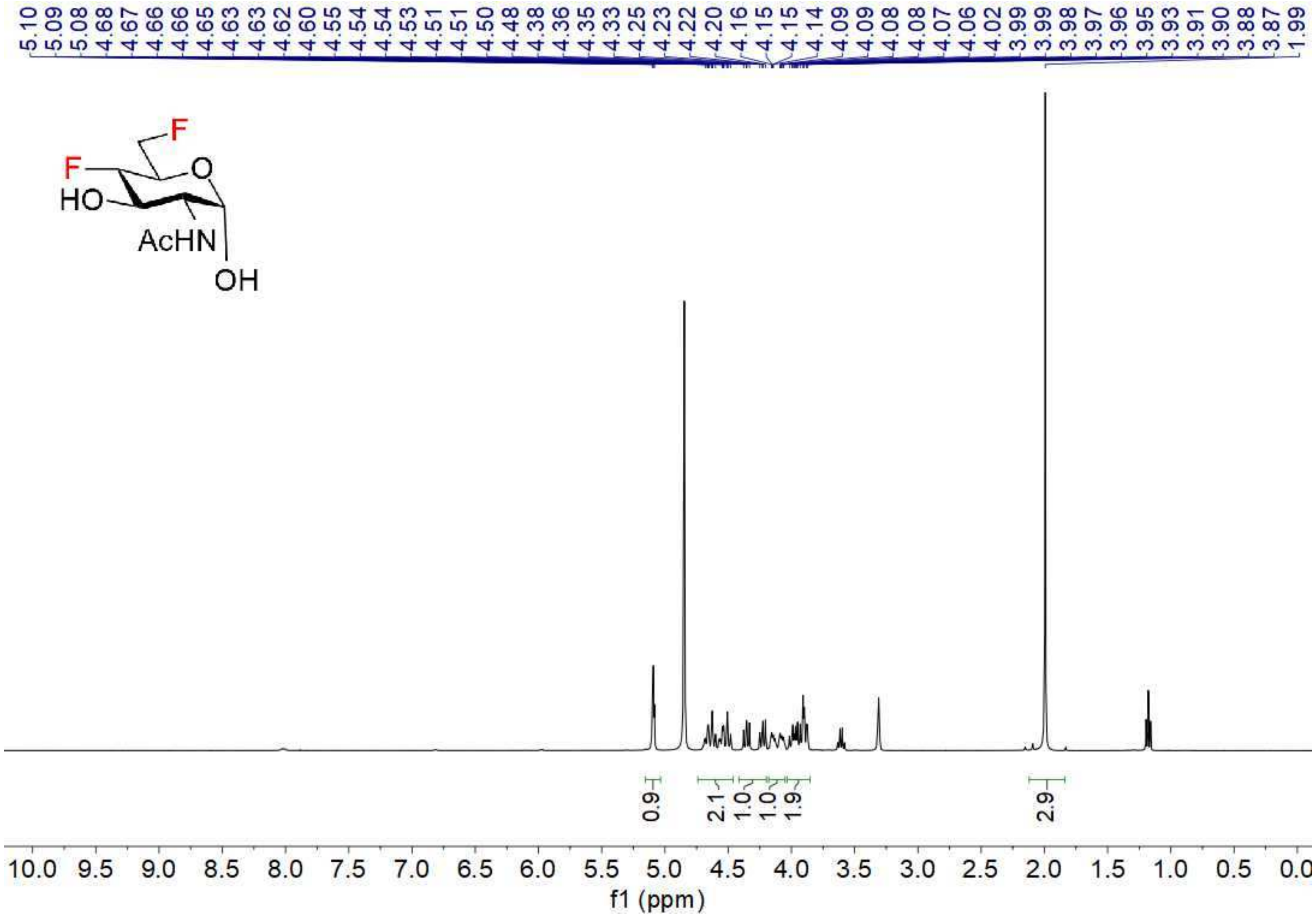


^{1}H-^{13}C HSQC 63

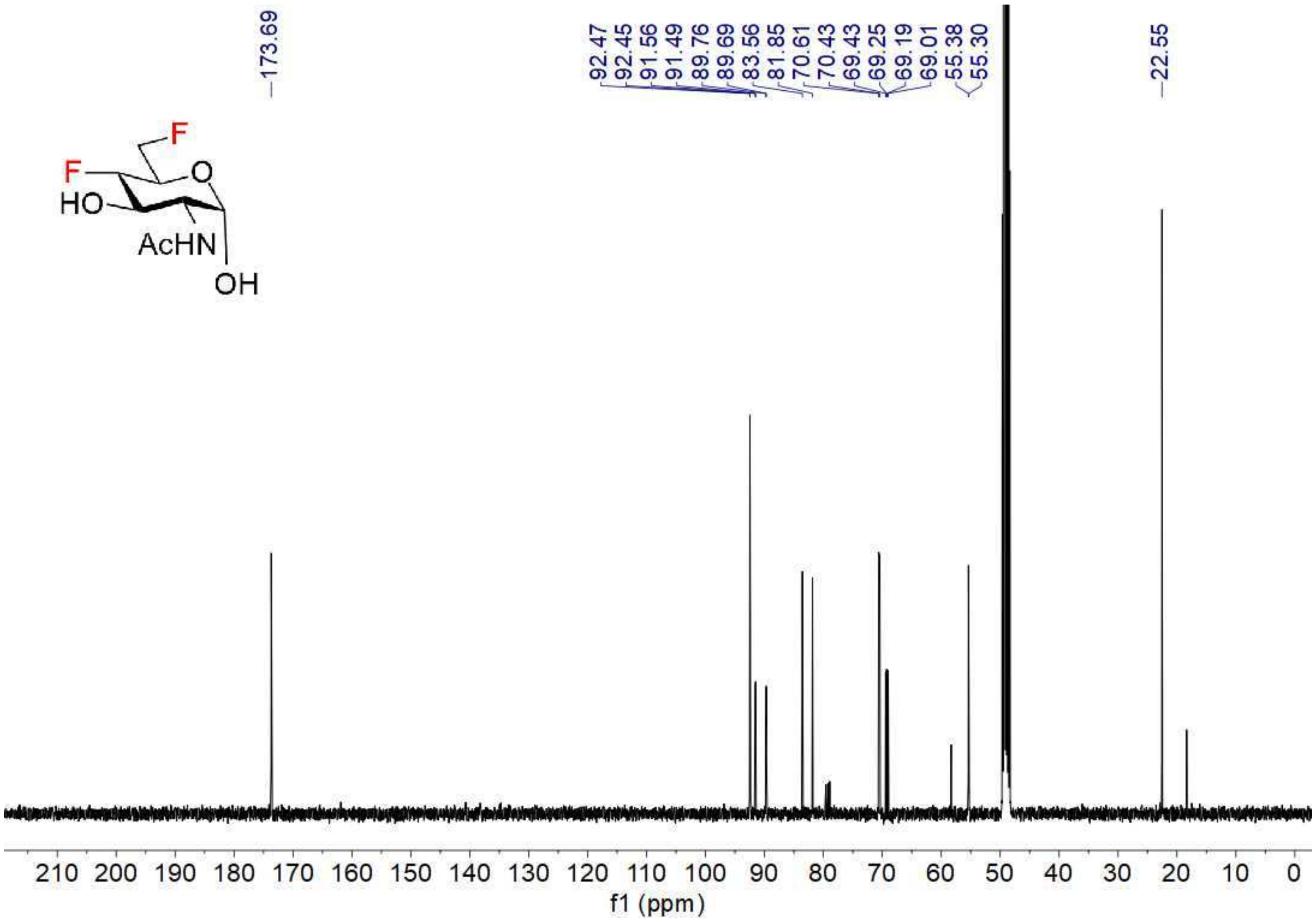


NMR COMPOUND 64

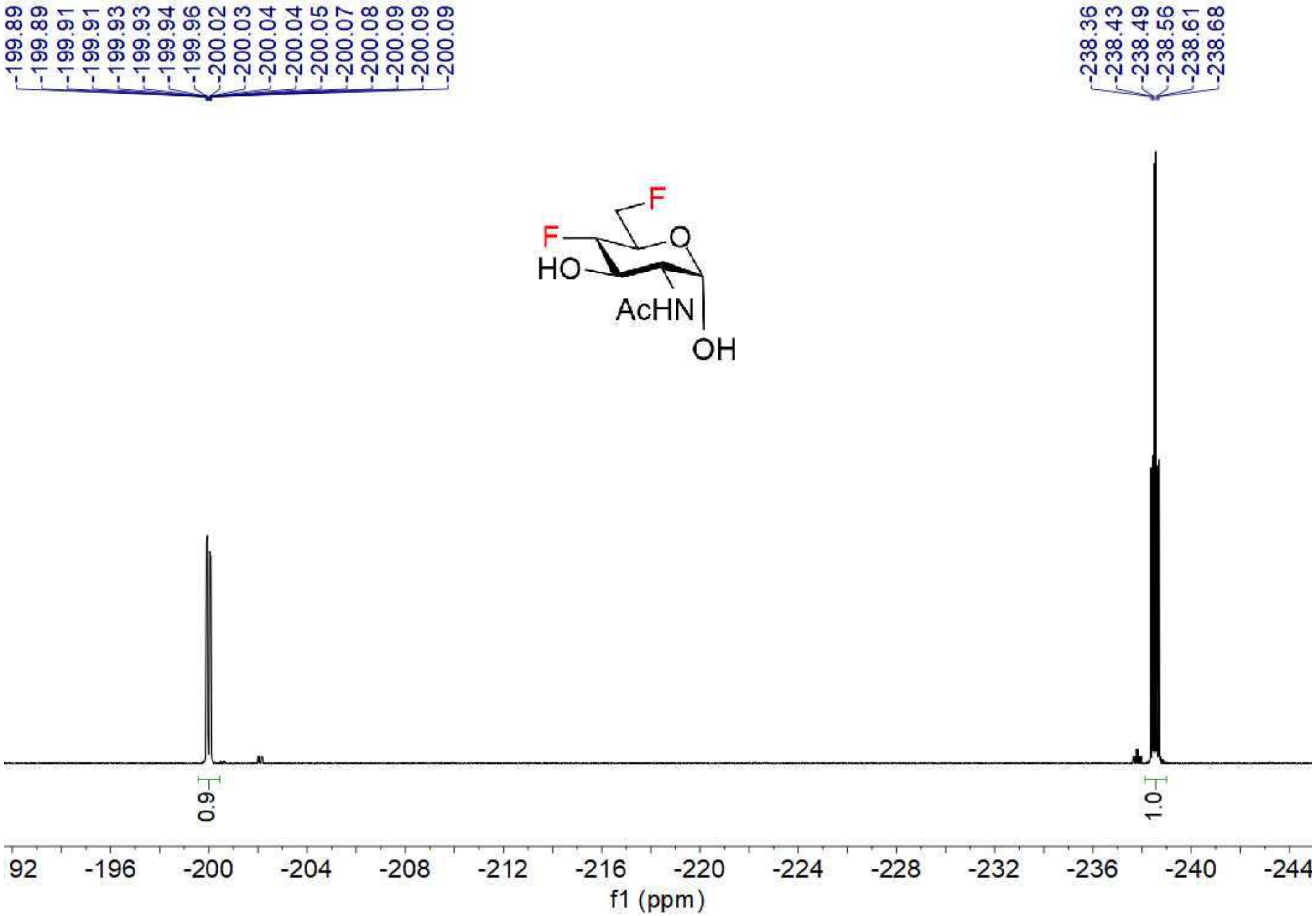
^{1}H NMR (400 MHz, CD_3OD) 64



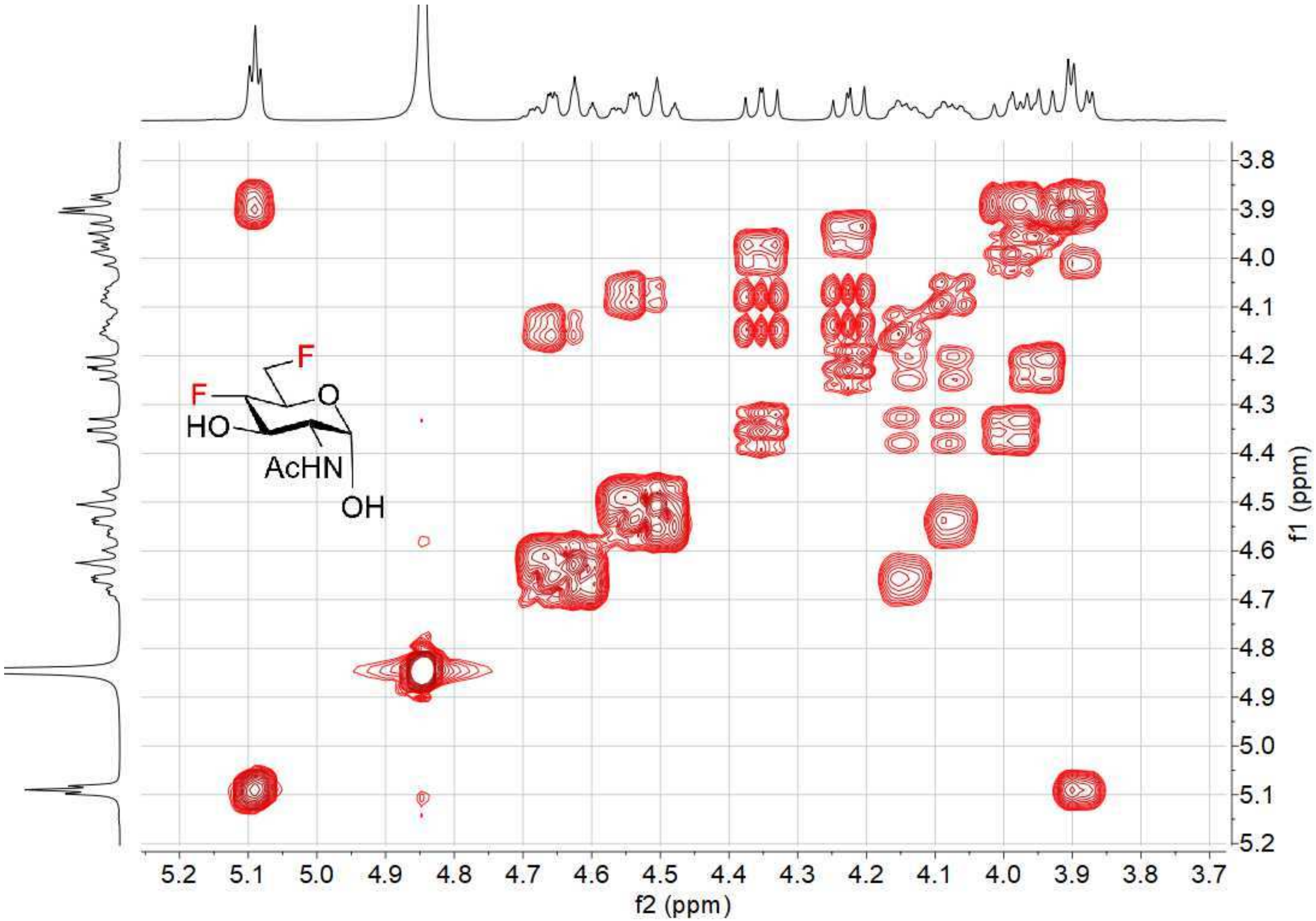
^{13}C NMR (100 MHz, CD_3OD) 64



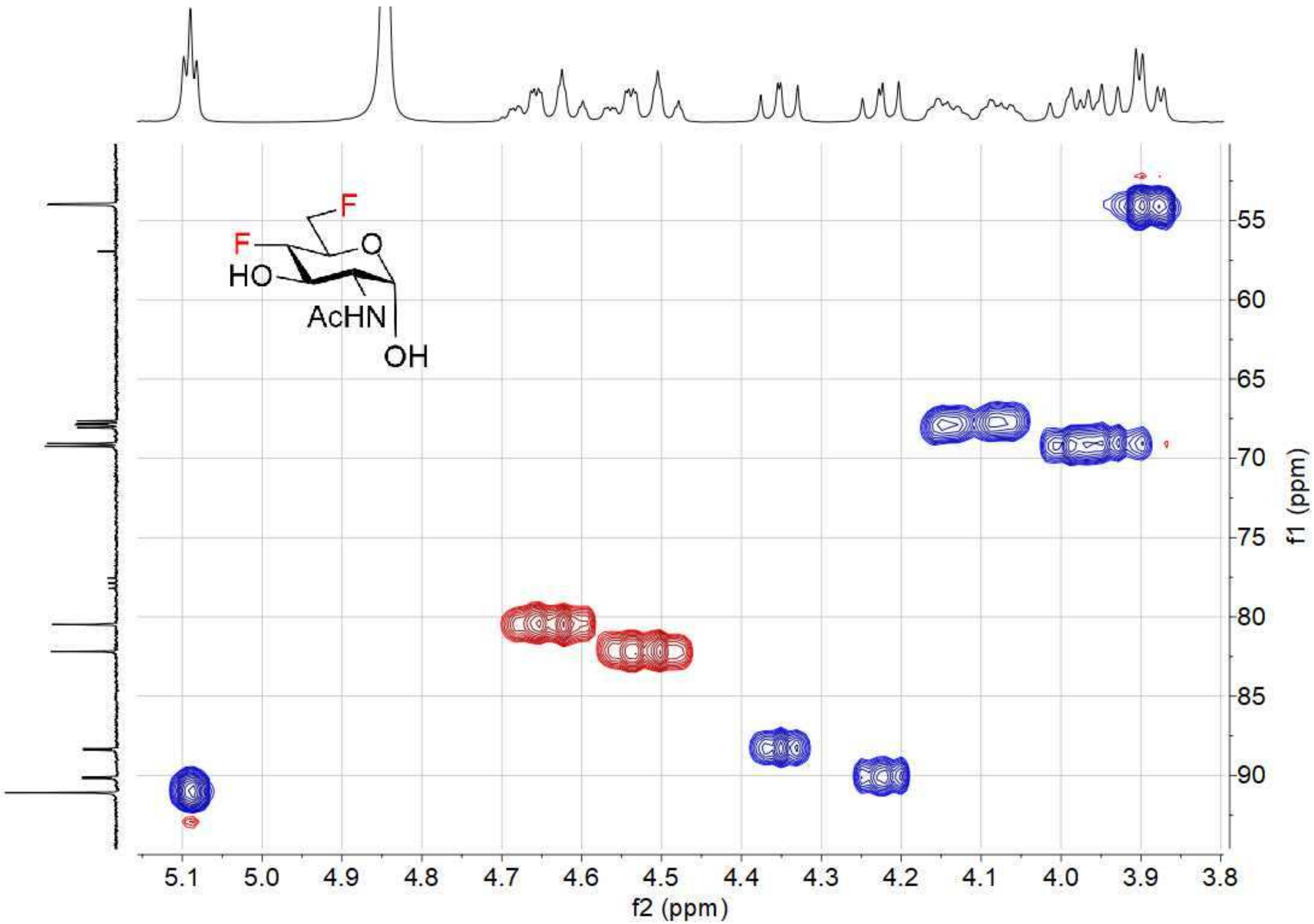
[19]F NMR (376 MHz, CD_3OD) 64



^{1}H-^{1}H COSY 64

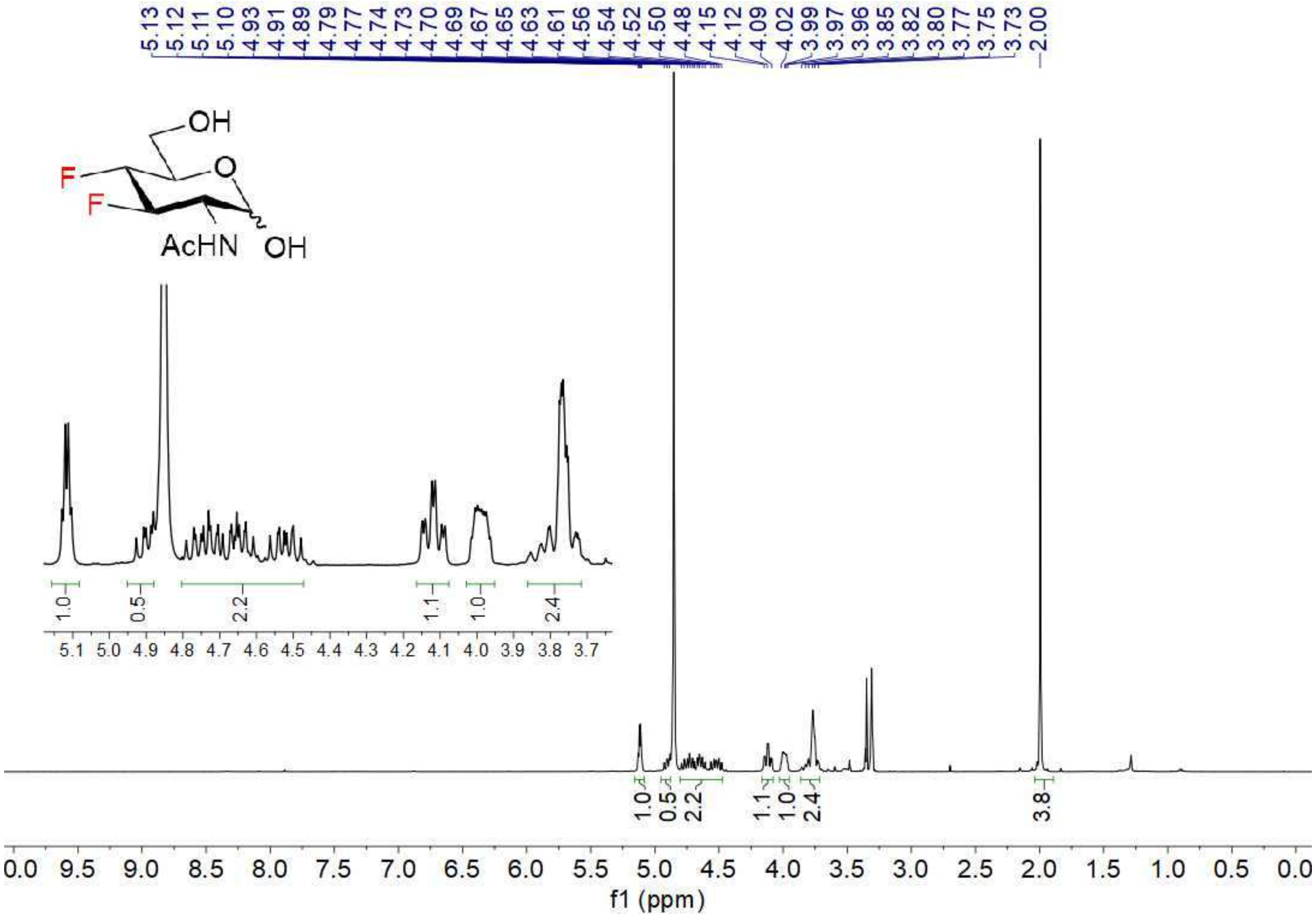

F
F
O
HO
AcHN
OH
5.2
5.1
5.0
4.9
4.8
4.7
4.6
4.5
4.4
4.3
4.2
4.1
4.0
3.9
3.8
3.7
f2 (ppm)
f1 (ppm)

^{1}H-^{13}C HSQC 64

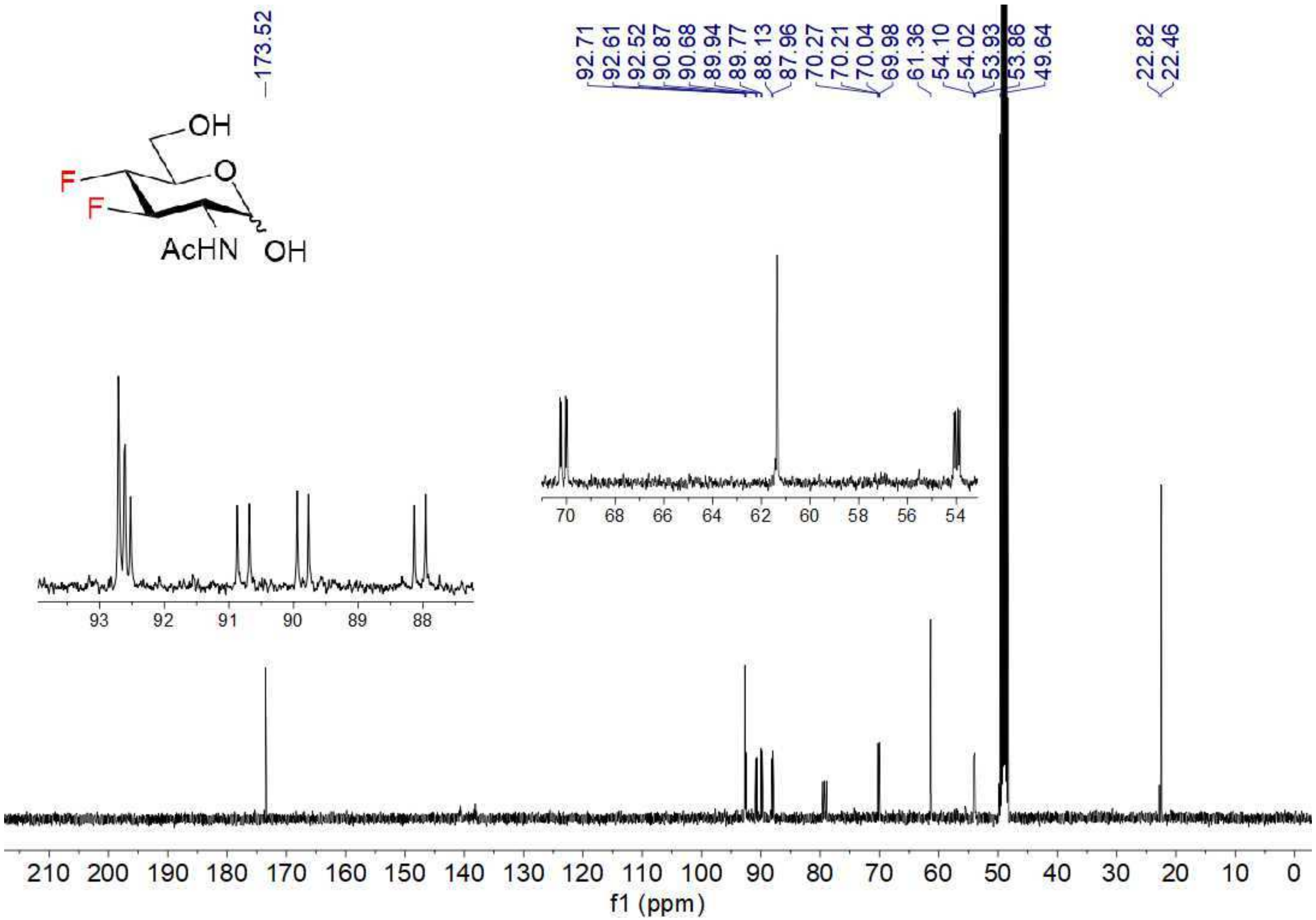


NMR COMPOUND 65

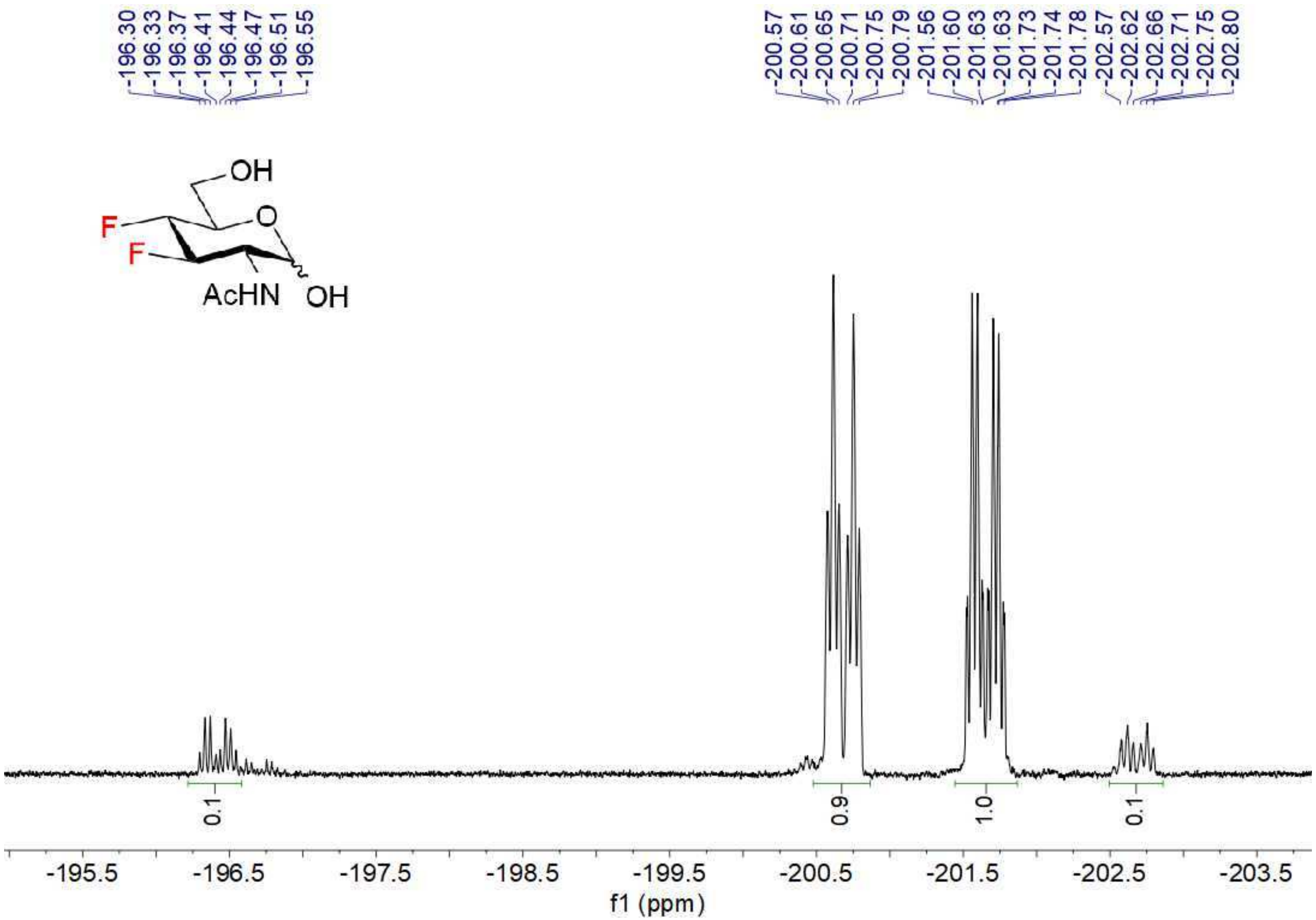
^{1}H NMR (400 MHz, CD_3OD) 65 (α/β ca. 10/1)



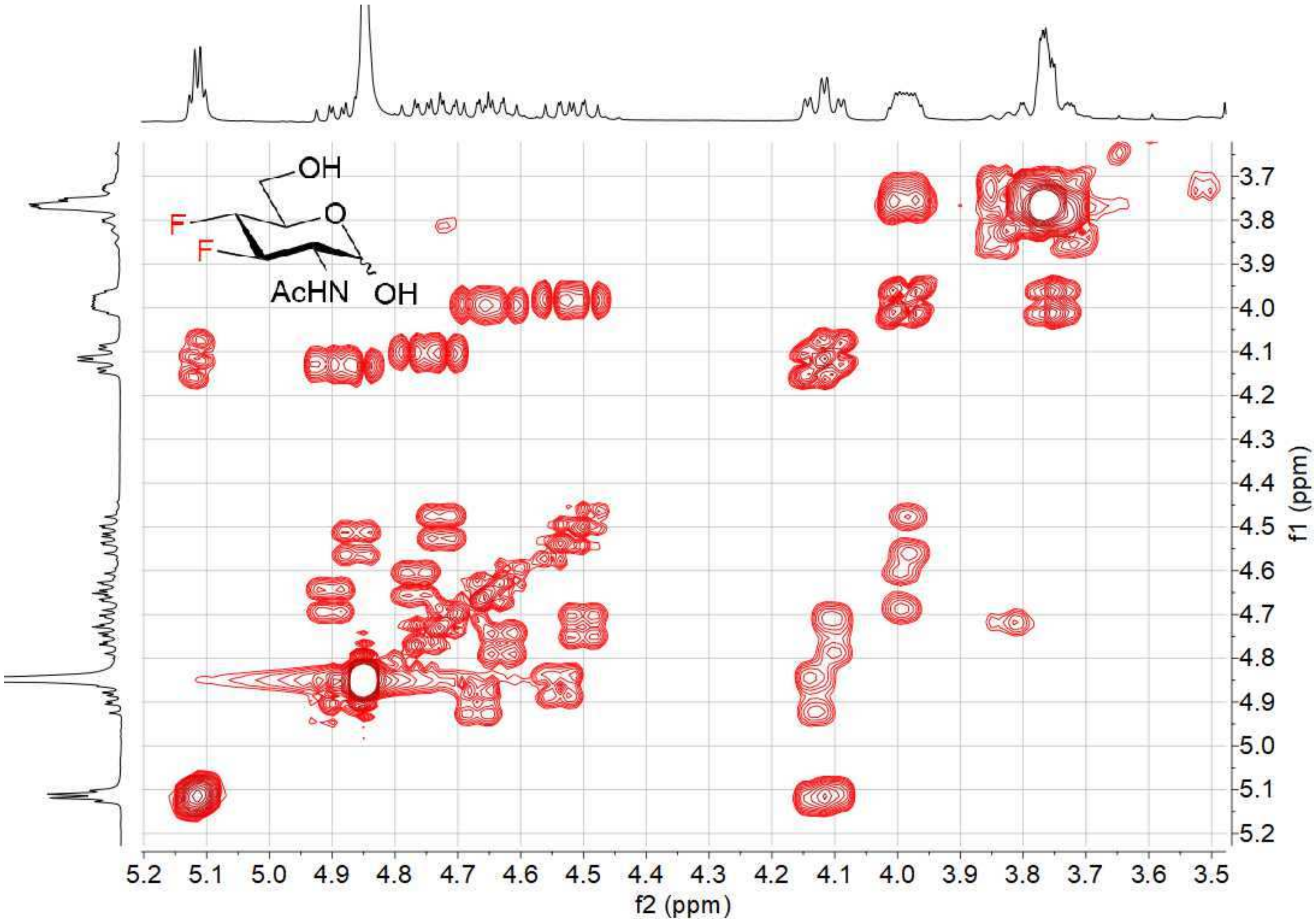
^{13}C NMR (100 MHz, CD$_3$OD) 65



^{19}F NMR (376 MHz, CD$_3$OD) 65 (α/β ca. 10/1)

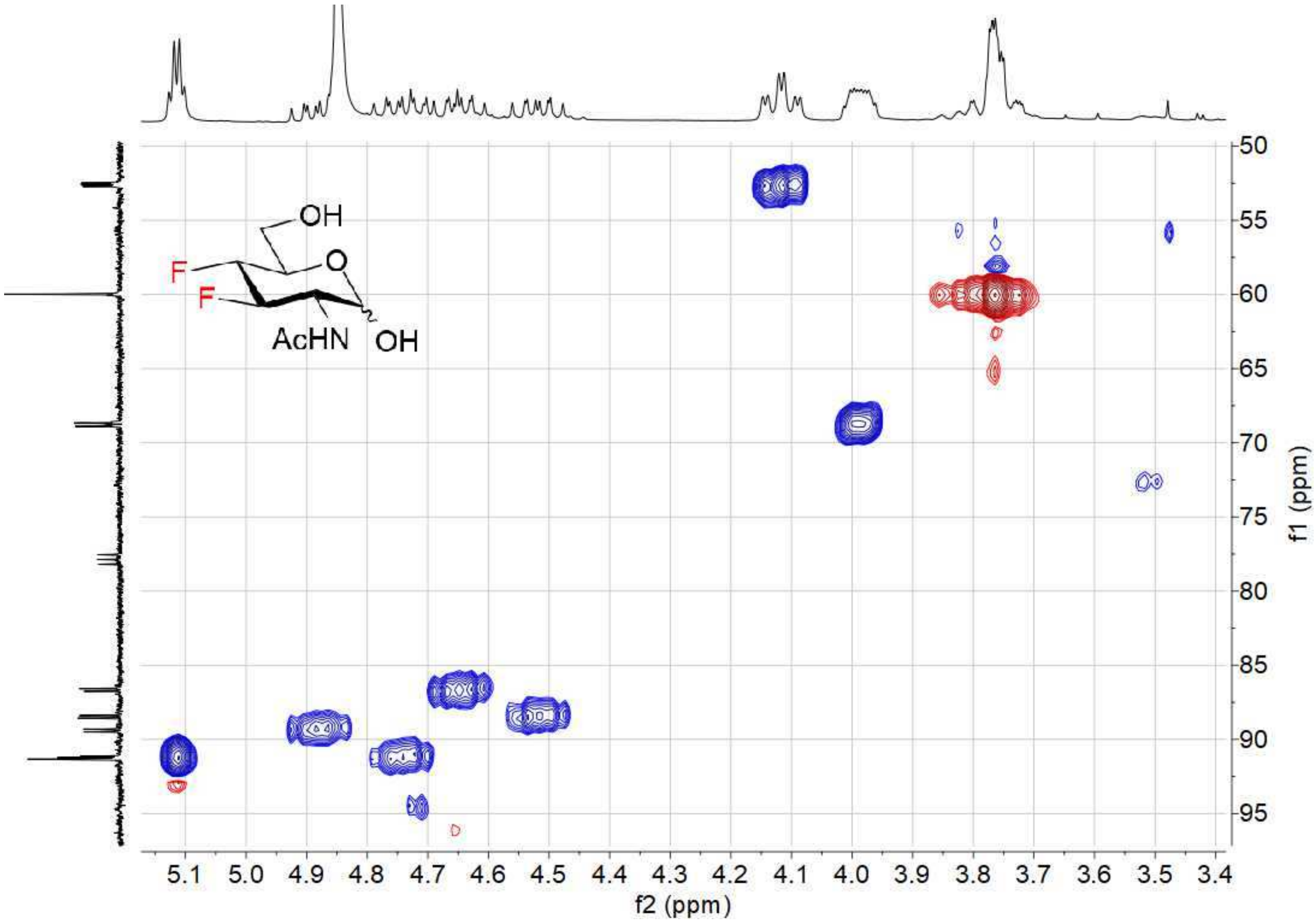


^{1}H-^{1}H COSY 65



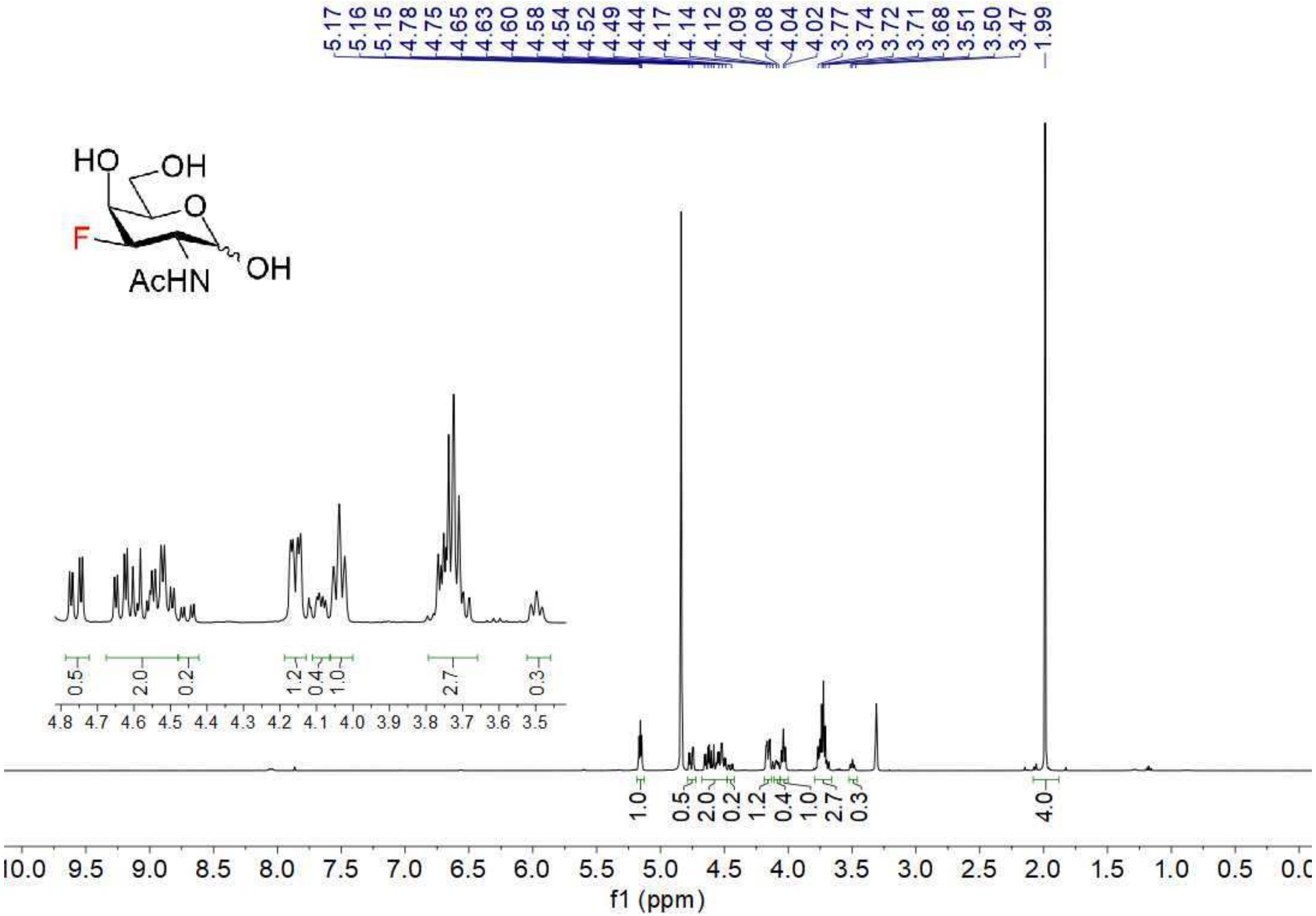
OH
O
F
F
AcHN
OH
3.7
3.8
3.9
4.0
4.1
4.2
4.3
4.4
4.5
4.6
4.7
4.8
4.9
5.0
5.1
5.2
f1 (ppm)
5.2 5.1 5.0 4.9 4.8 4.7 4.6 4.5 4.4 4.3 4.2 4.1 4.0 3.9 3.8 3.7 3.6 3.5
f2 (ppm)

$^{1}H-^{13}C$ HSQC 65

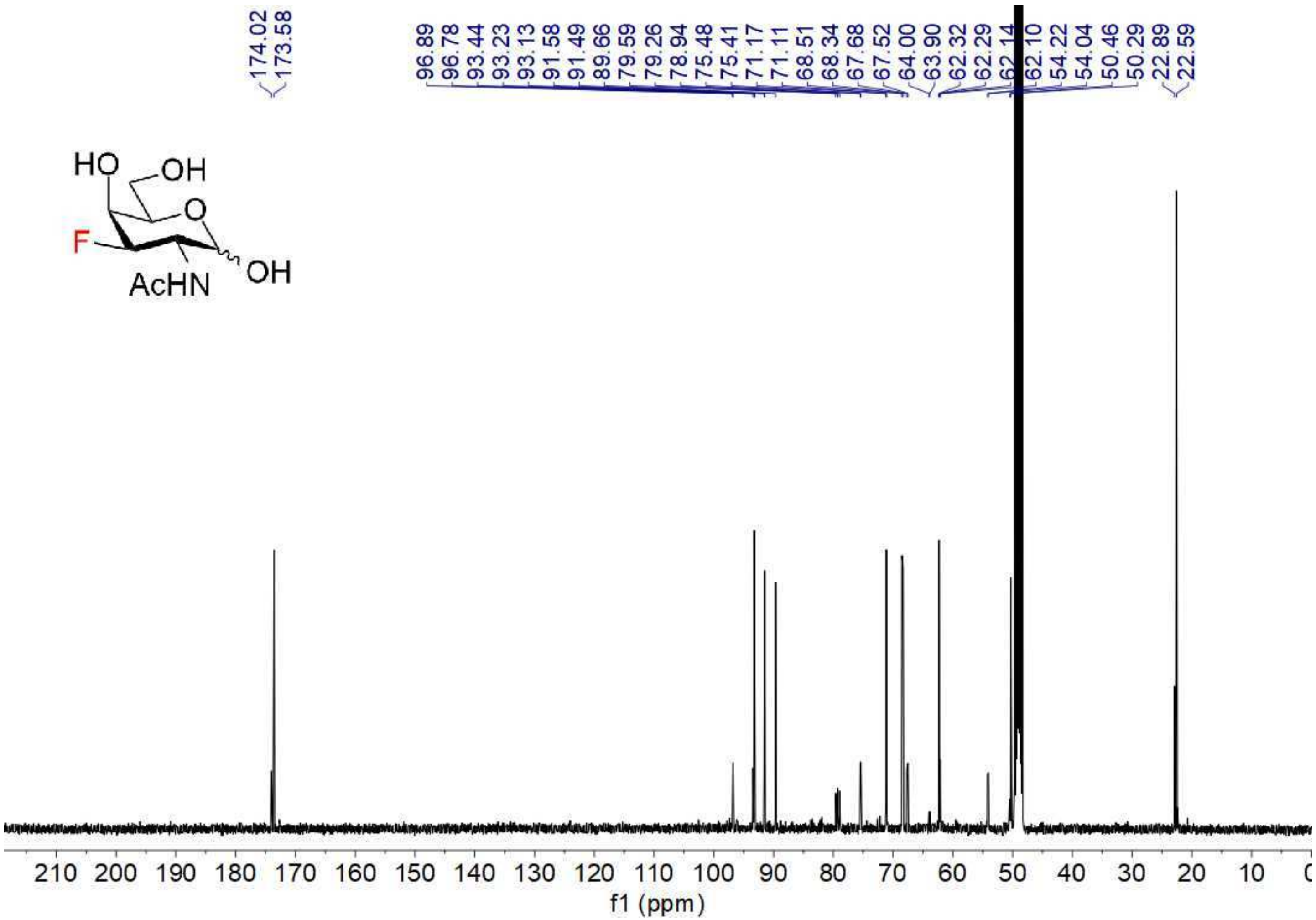


NMR COMPOUND 66

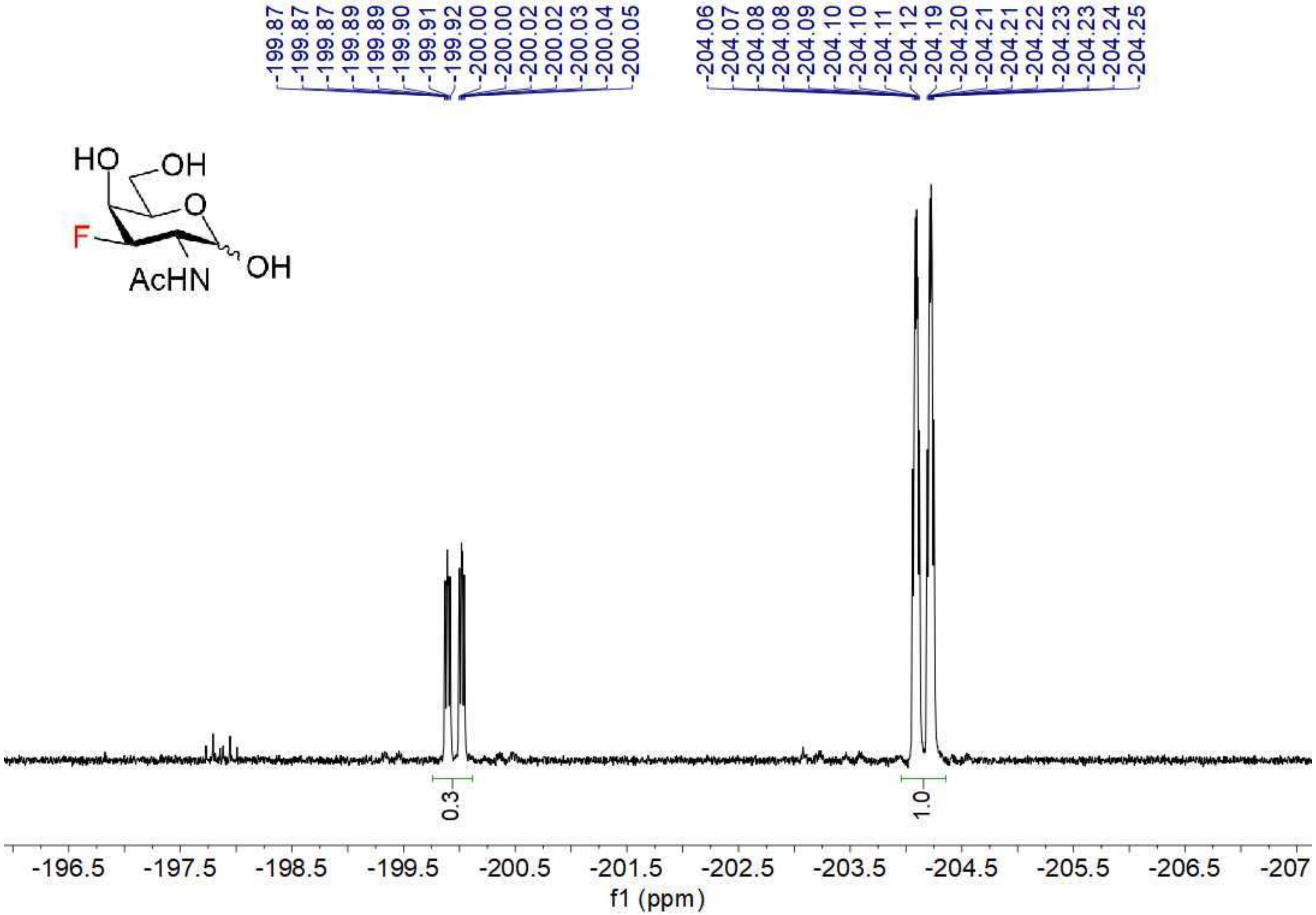
[1]H NMR (400 MHz, CD_3OD) 66 (α/β ca. 10/3)



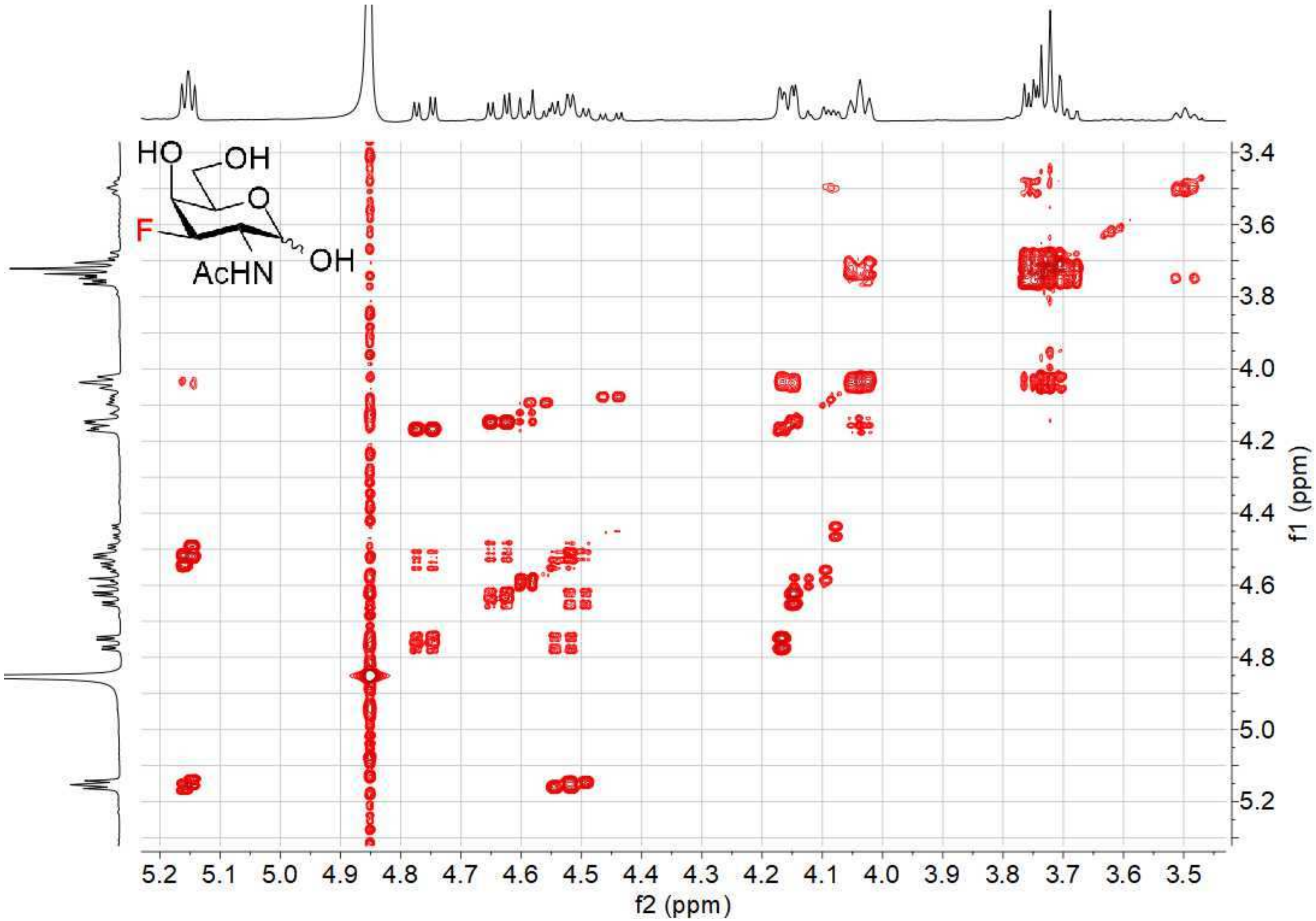
[13]C NMR (100 MHz, CD_3OD) 66



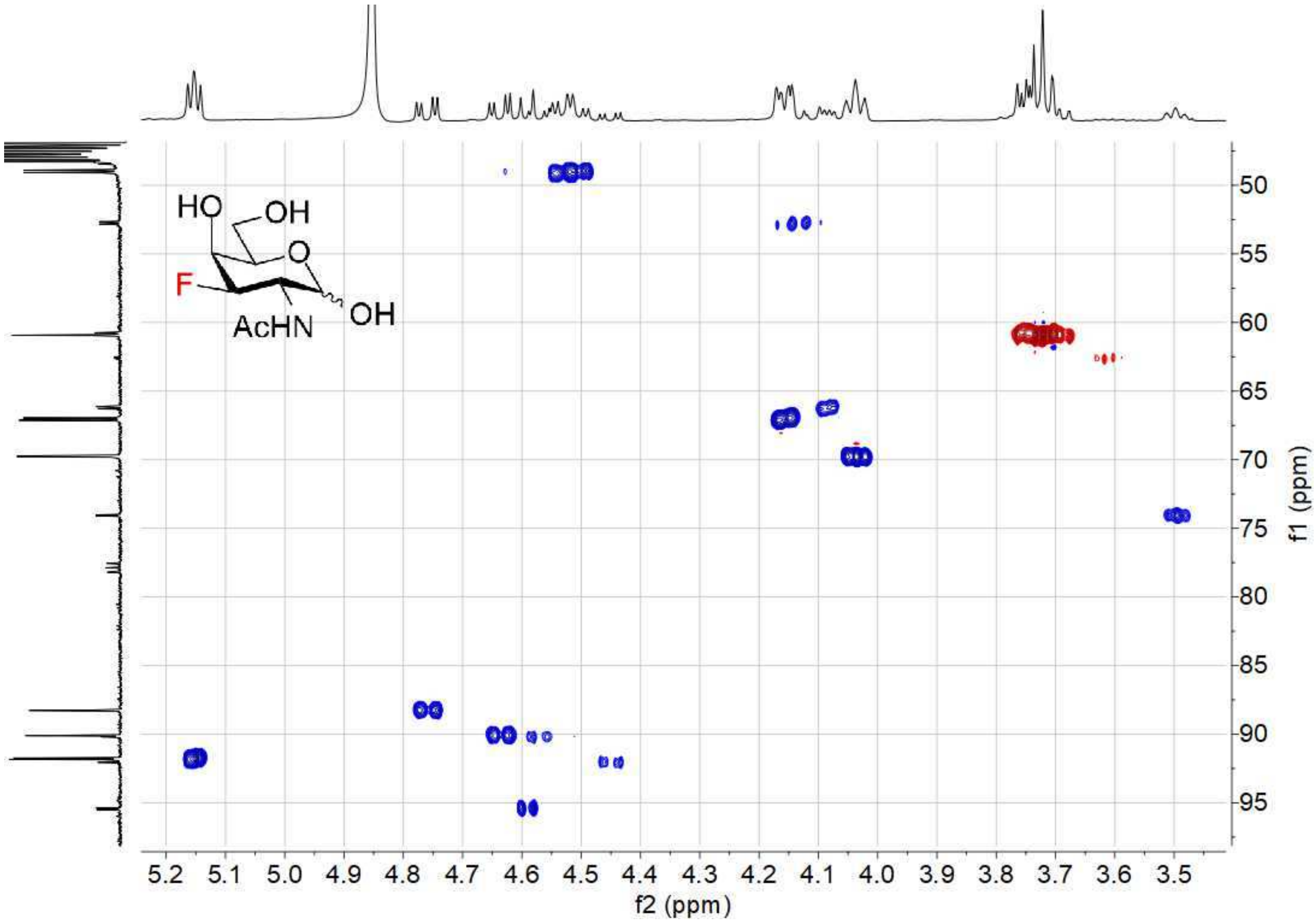
[19]F NMR (376 MHz, CD_3OD) 66 (α/β ca. 10/3)



^{1}H-^{1}H COSY NMR 66

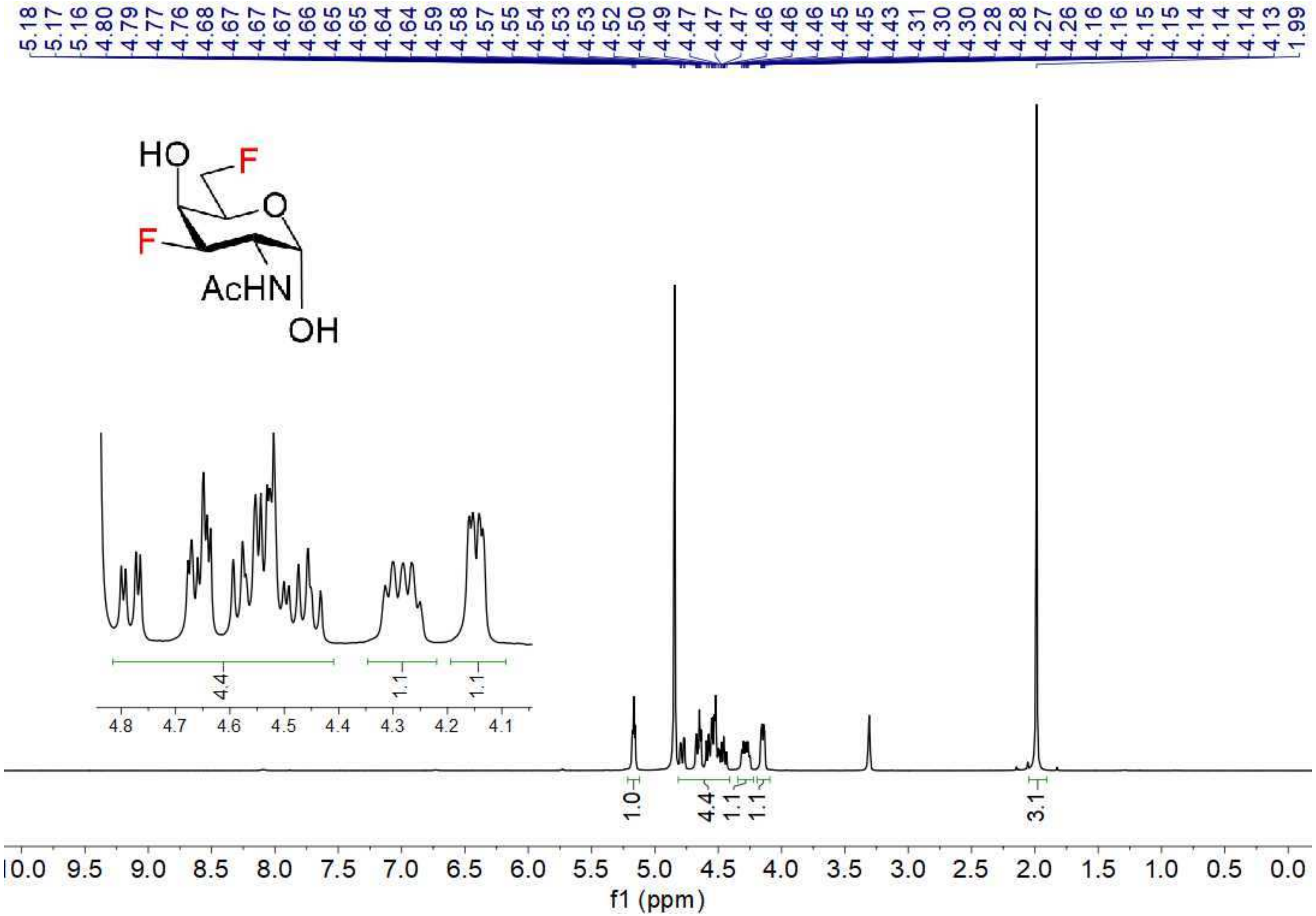


^{1}H-^{13}C HSQC NMR 66

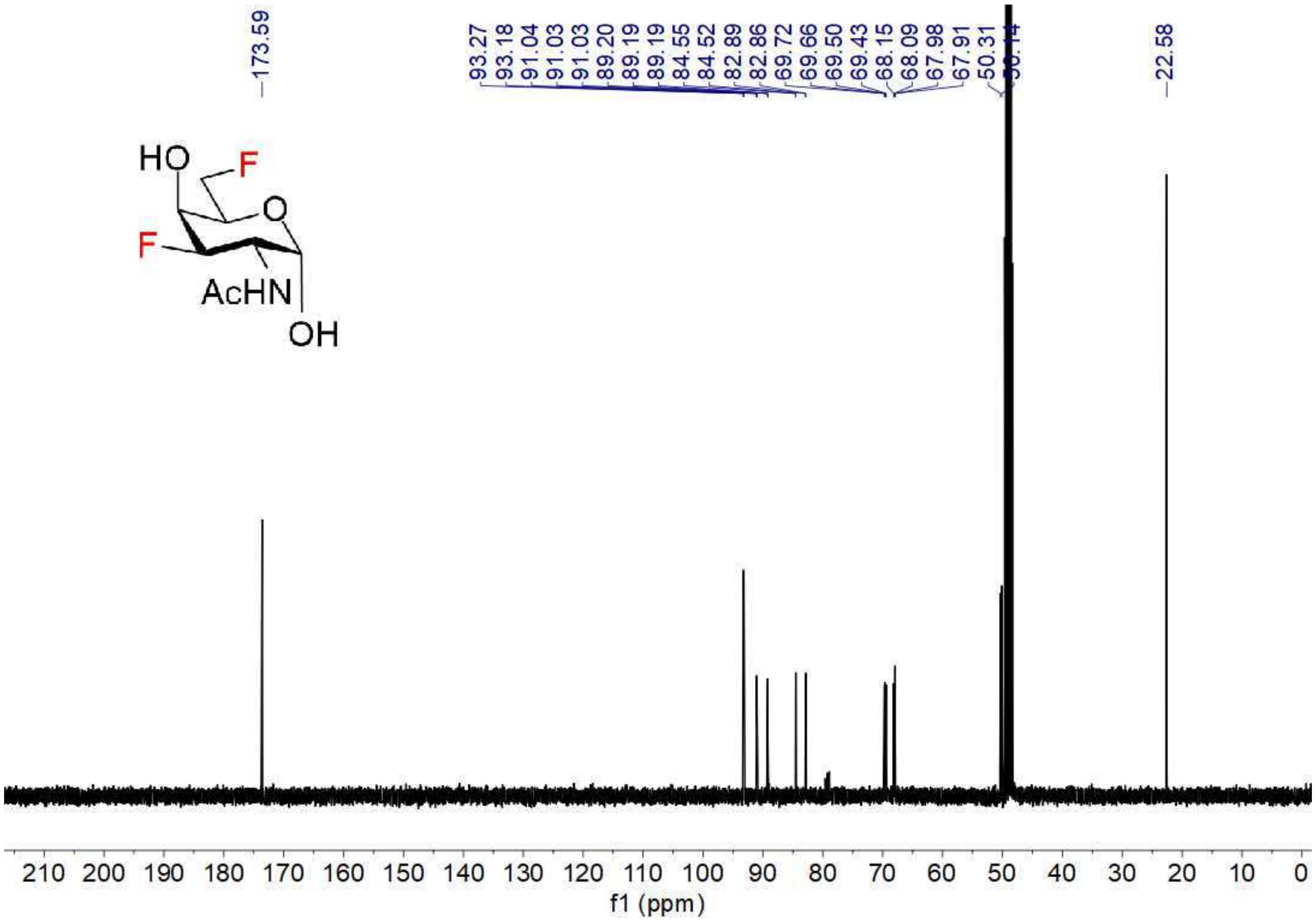


NMR COMPOUND 67

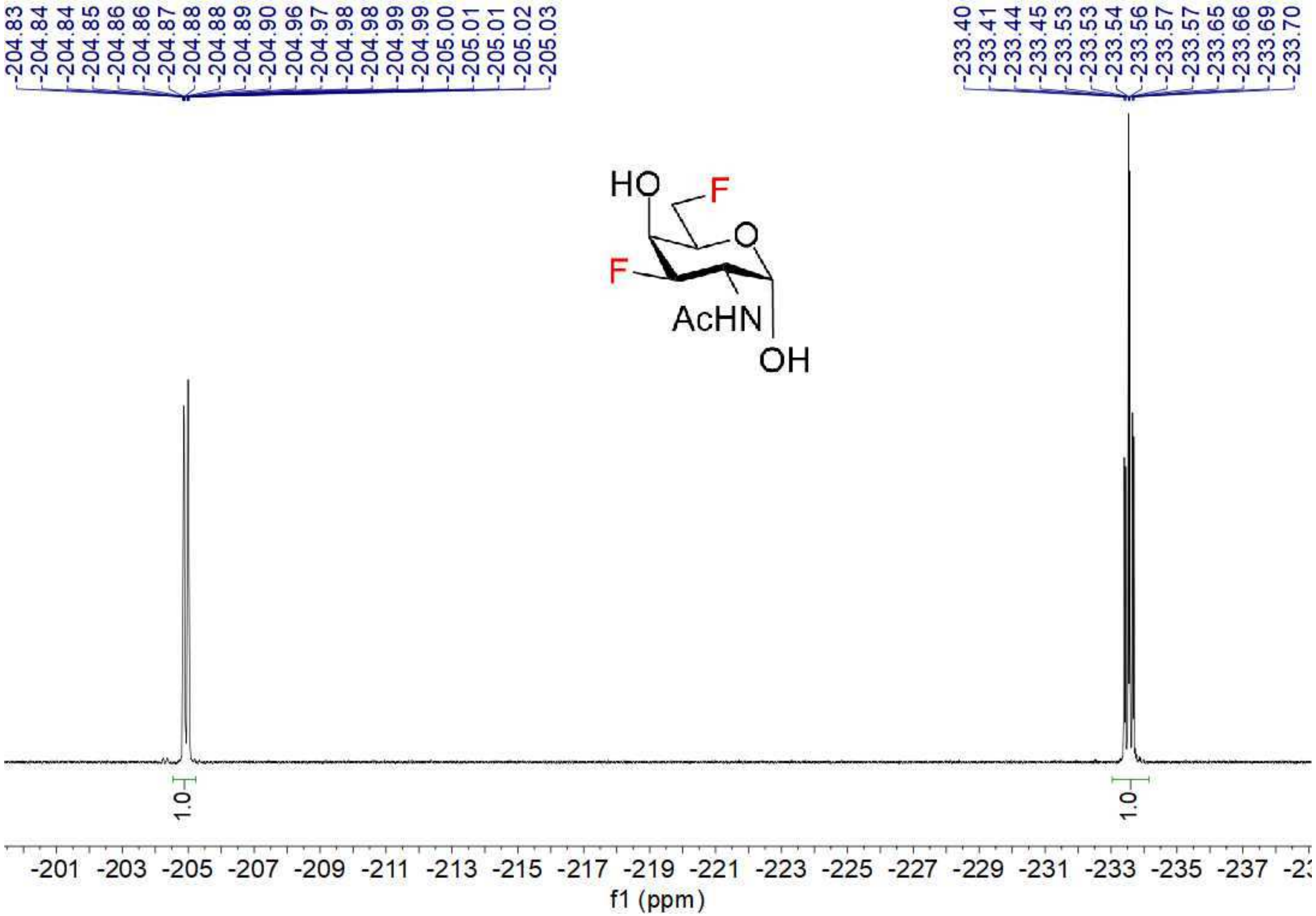
^{1}H NMR (400 MHz, CD_3OD) 67



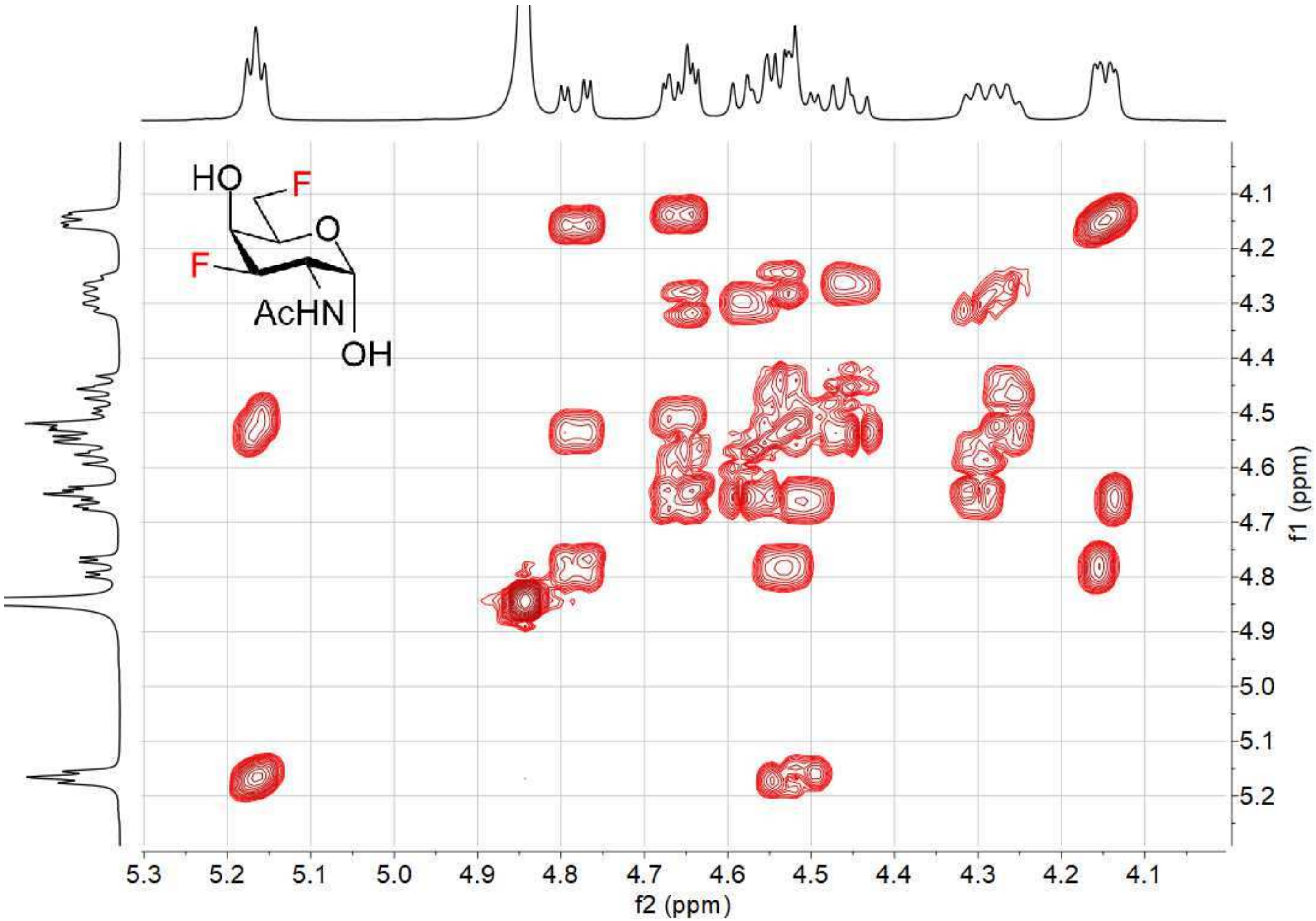
^{13}C NMR (100 MHz, CD_3OD) 67



^{19}F NMR (376 MHz, CD_3OD) 67

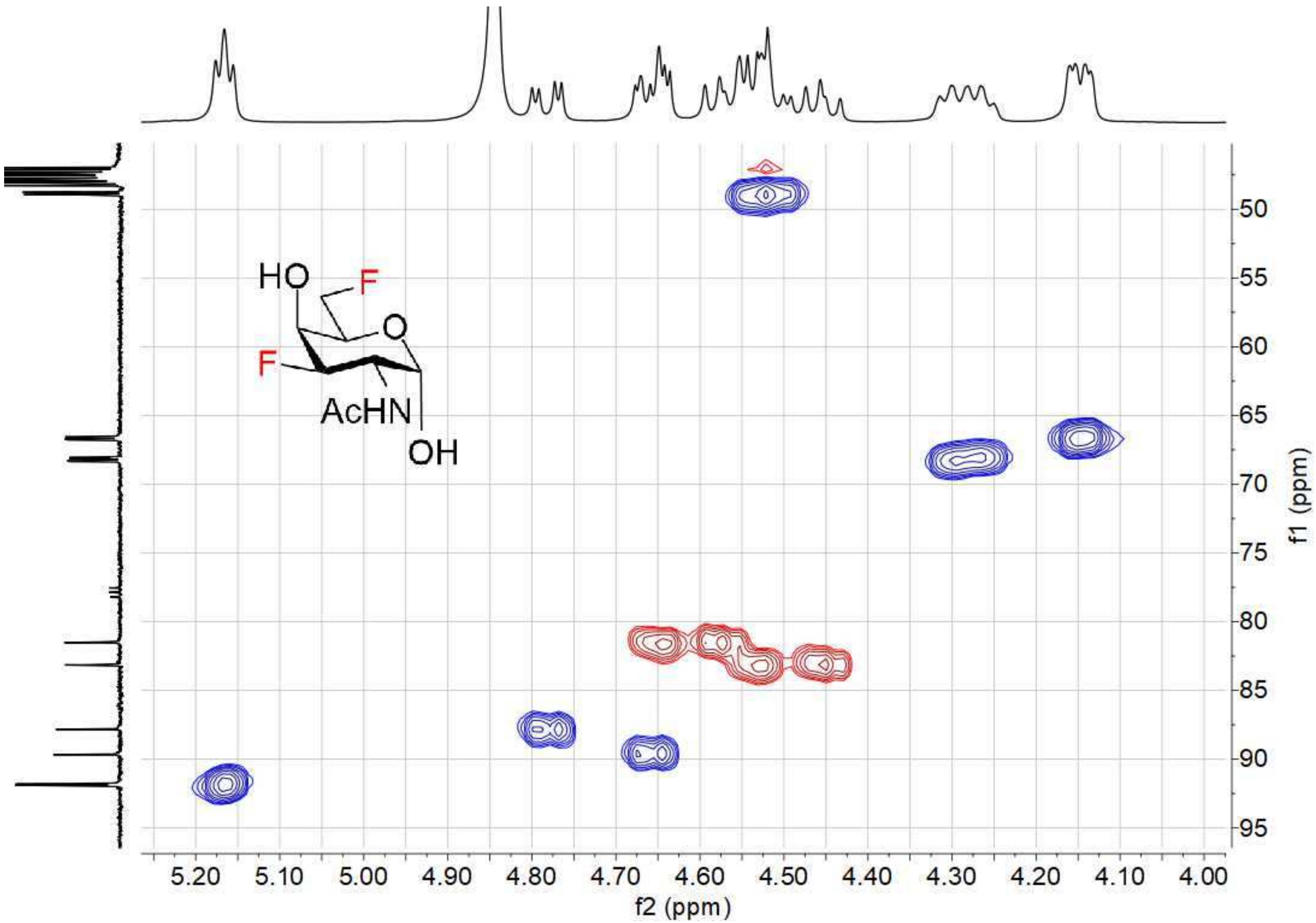


^{1}H-^{1}H COSY 67



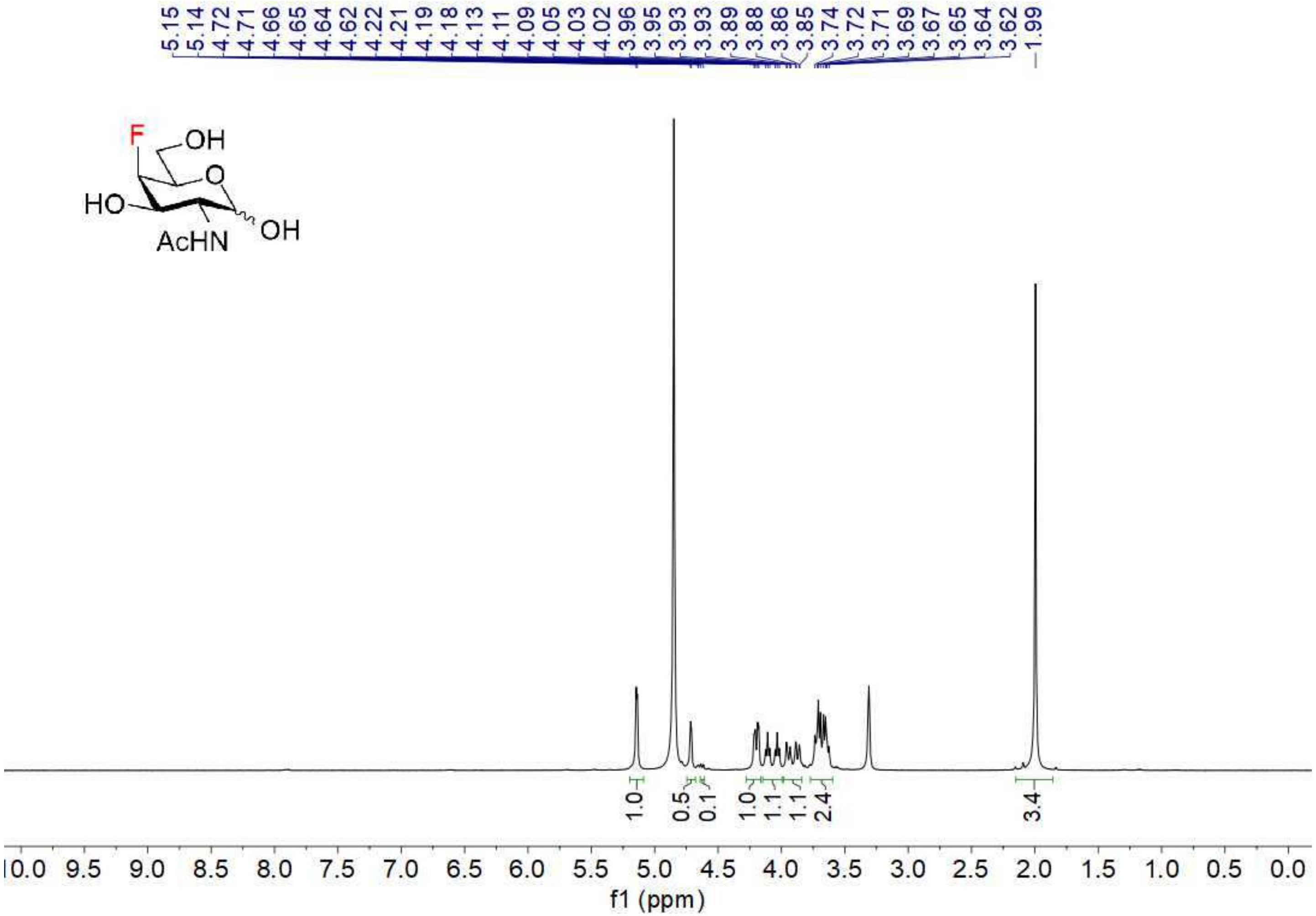
HO
F
O
F
AcHN
OH
5.3
5.2
5.1
5.0
4.9
4.8
4.7
4.6
4.5
4.4
4.3
4.2
4.1
f2 (ppm)
4.1
4.2
4.3
4.4
4.5
4.6
4.7
4.8
4.9
5.0
5.1
5.2
f1 (ppm)

^{1}H-^{13}C HSQC 67

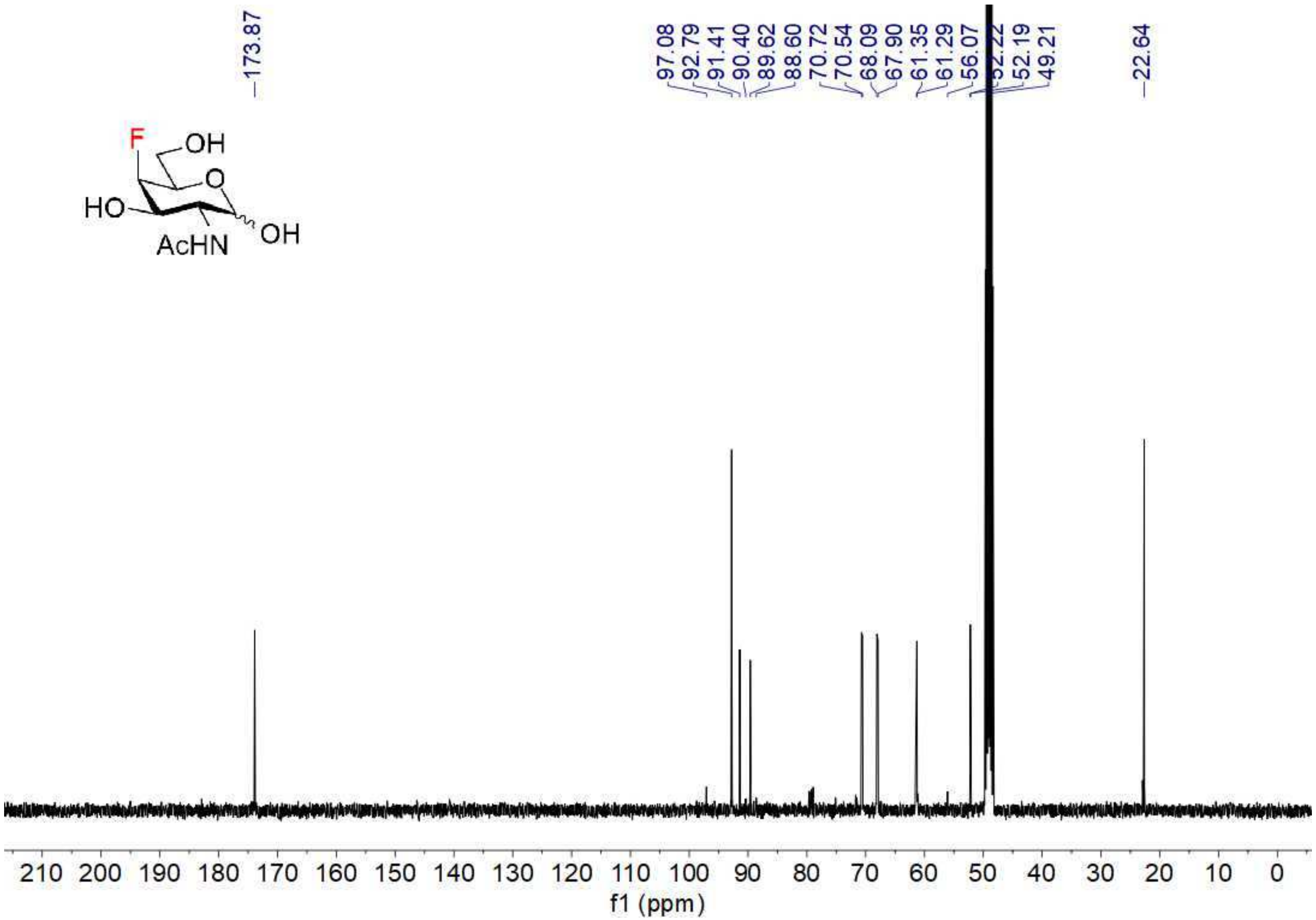


NMR COMPOUND 68

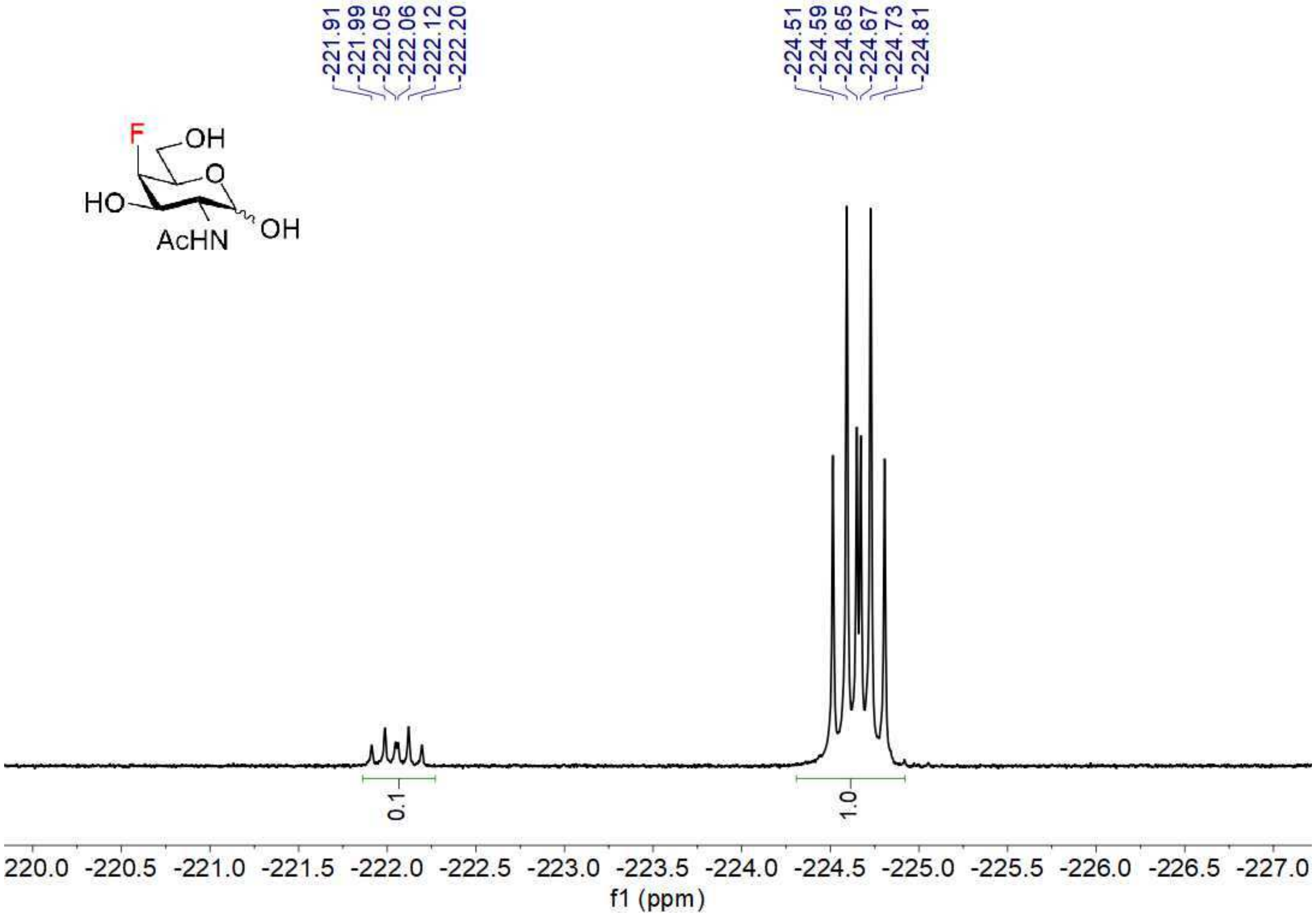
^{1}H NMR (400 MHz, CD_3OD) 68 (α/β ca. 10/1)



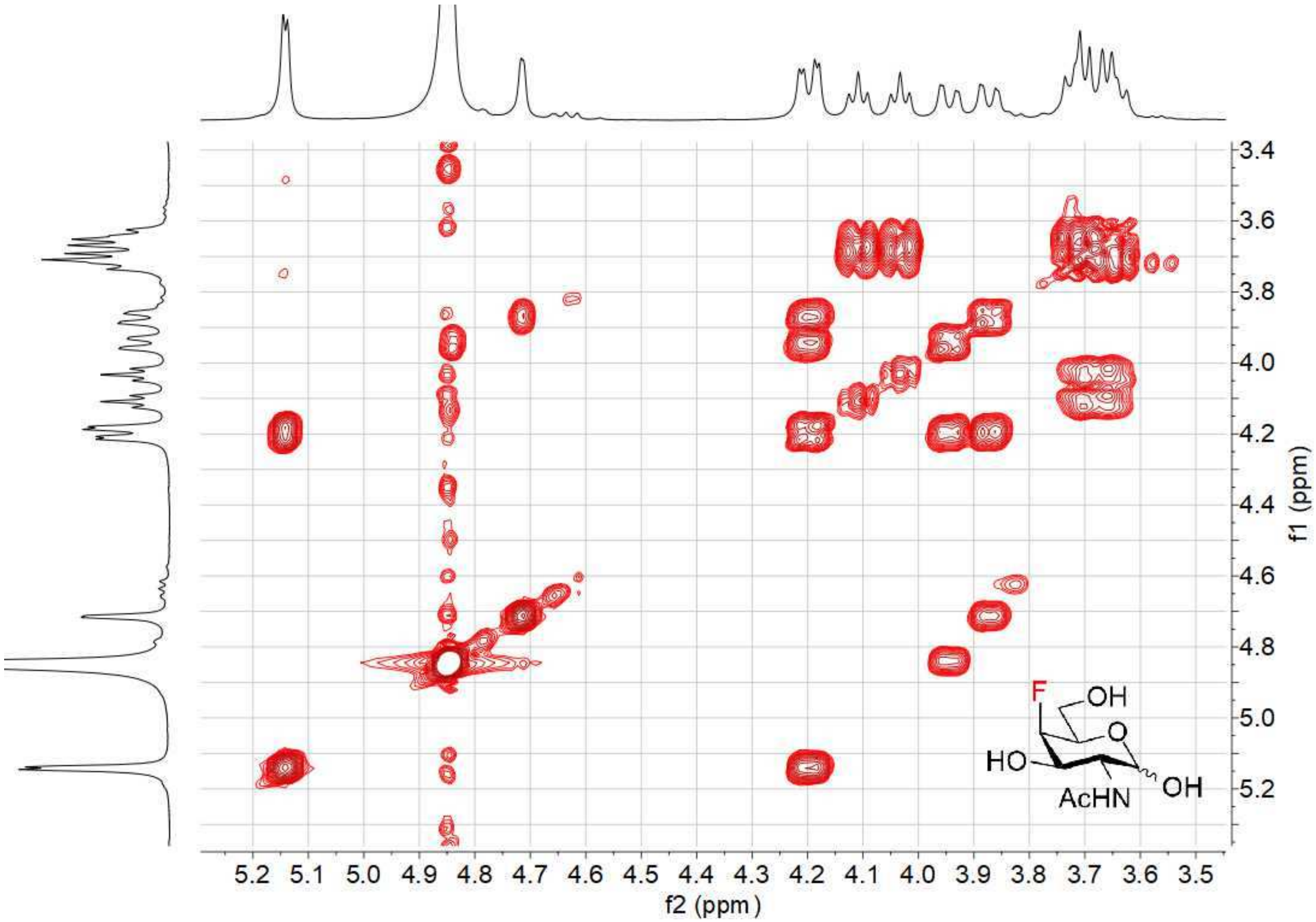
^{13}C NMR (100 MHz, CD_3OD) 68



^{19}F NMR (376 MHz, CD_3OD) 68 (α/β ca. 10/1)

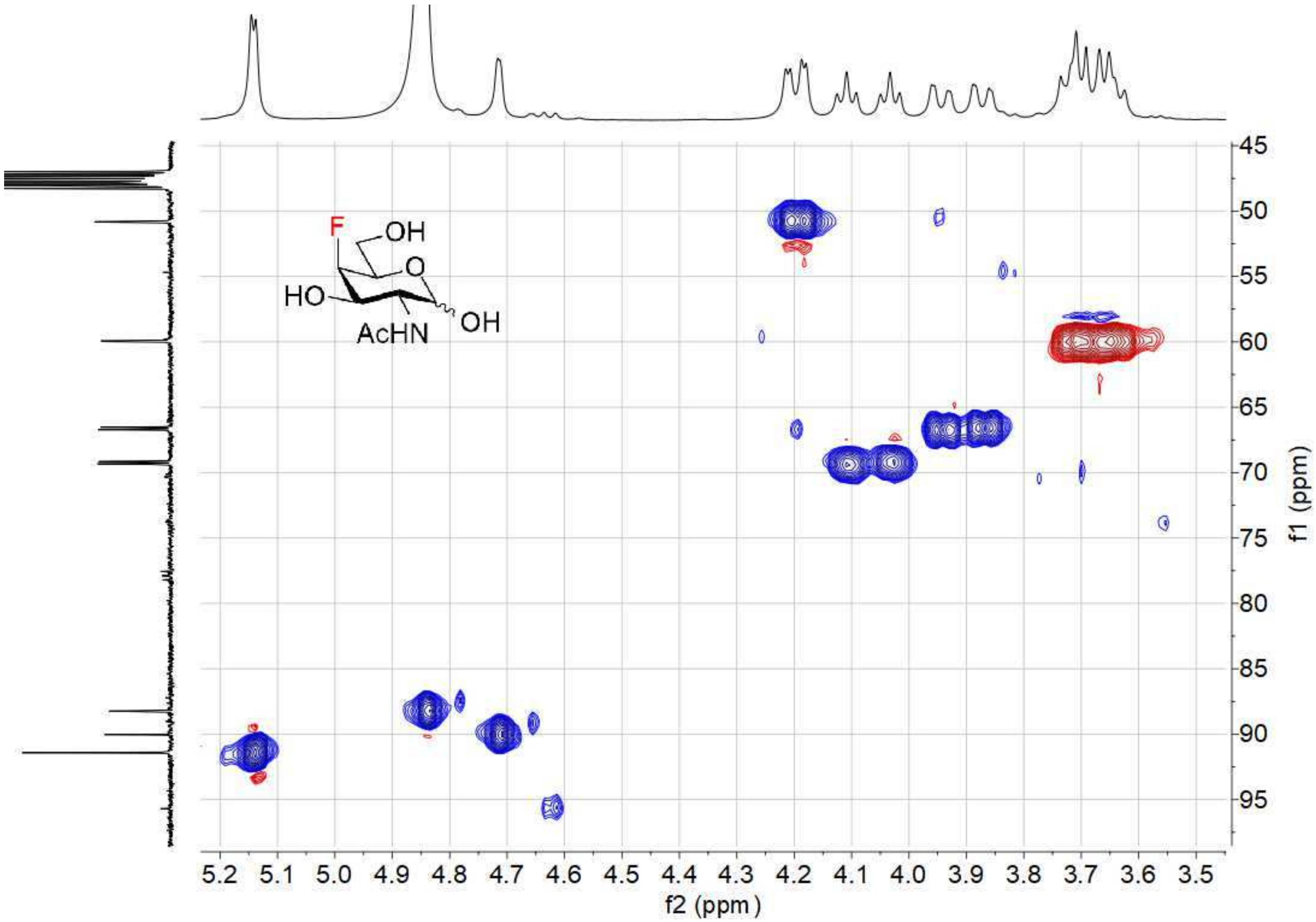


[1]H-[1]H COSY 68



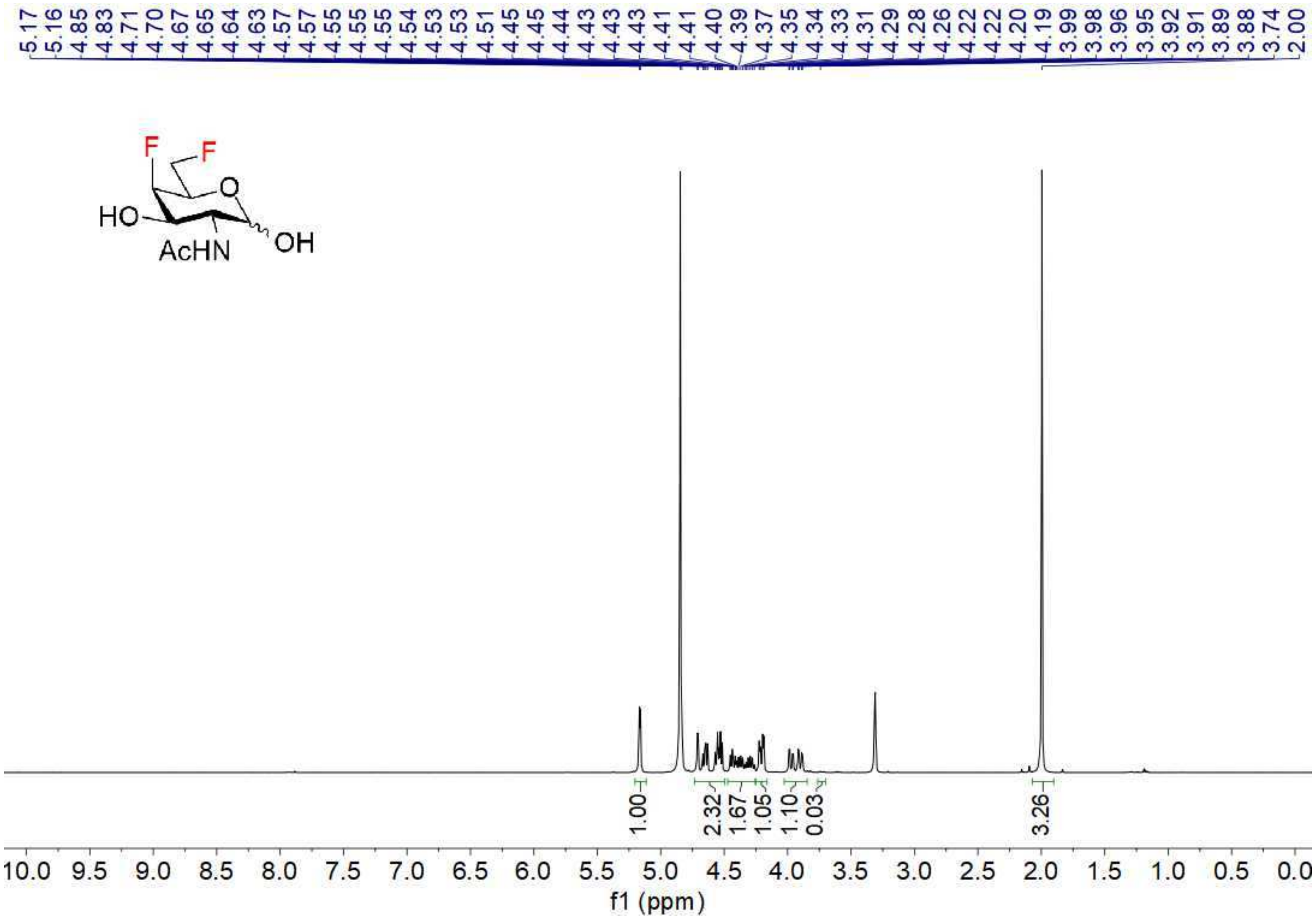
5.2 5.1 5.0 4.9 4.8 4.7 4.6 4.5 4.4 4.3 4.2 4.1 4.0 3.9 3.8 3.7 3.6 3.5
f2 (ppm)
3.4 3.6 3.8 4.0 4.2 4.4 4.6 4.8 5.0 5.2
f1 (ppm)
F
OH
O
HO
AcHN
OH

^{1}H-^{13}C HSQC 68

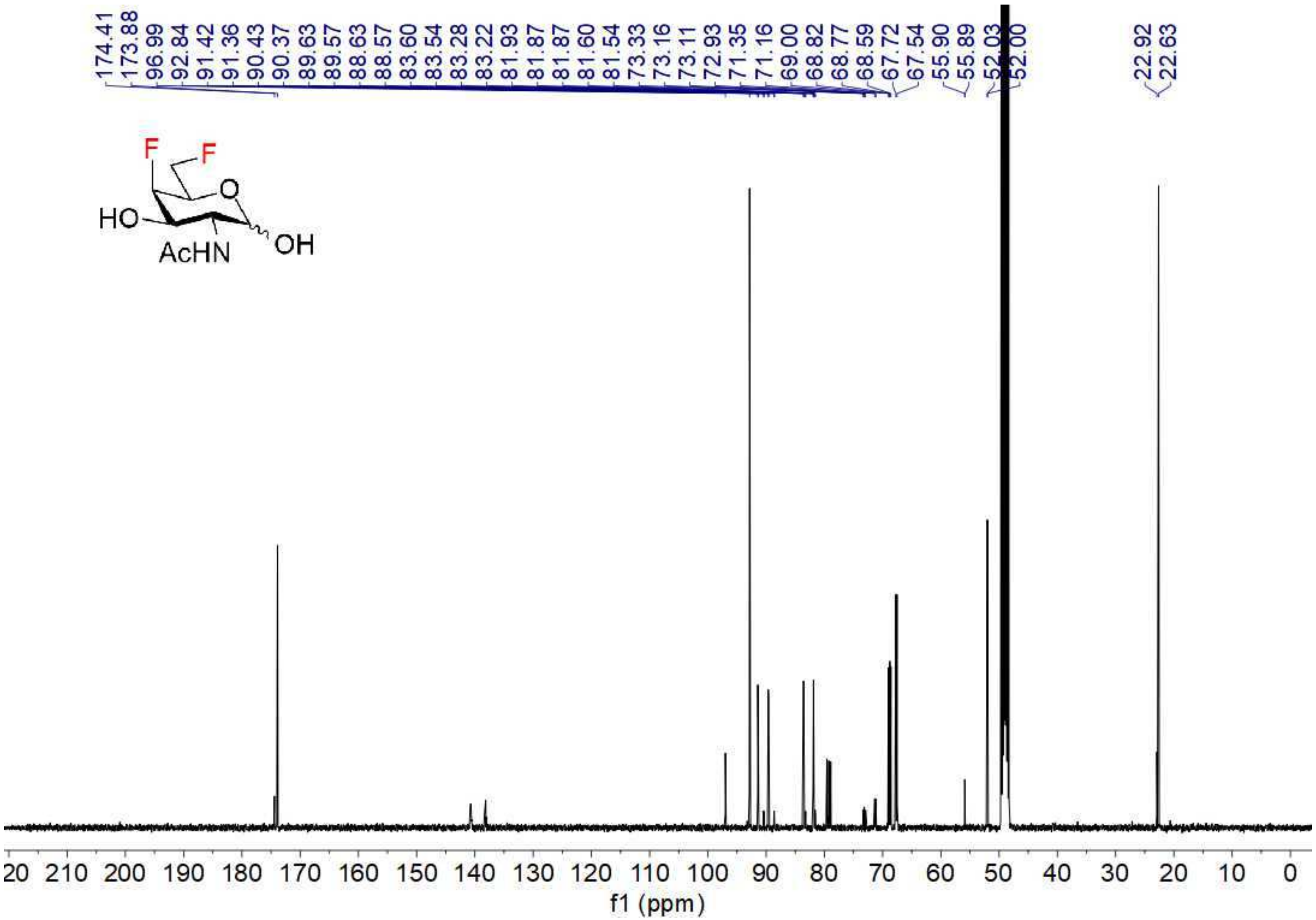


NMR COMPOUND 69

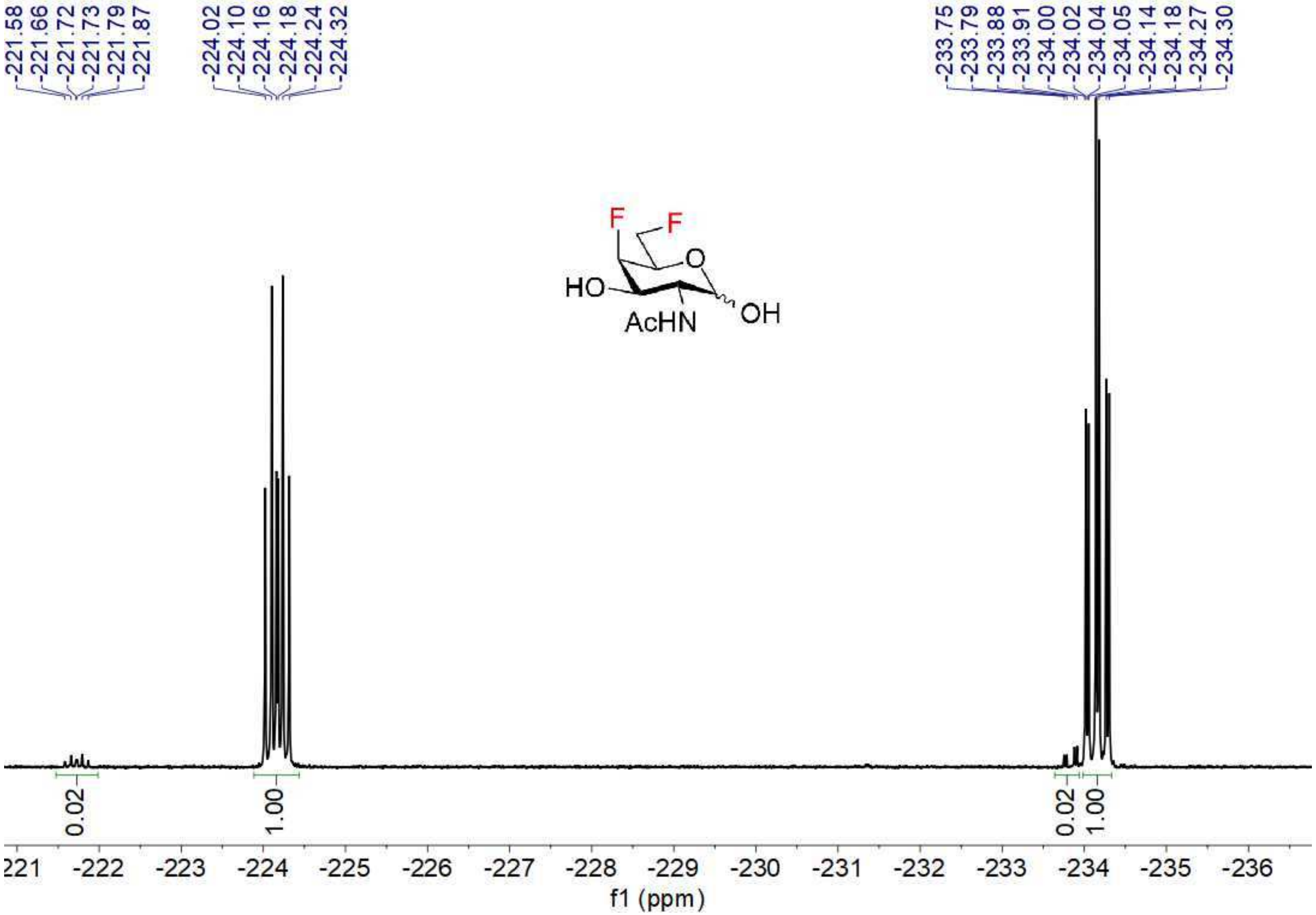
^{1}H NMR (400 MHz, CD_3OD) 69 (ca. 2-3% β-anomer)



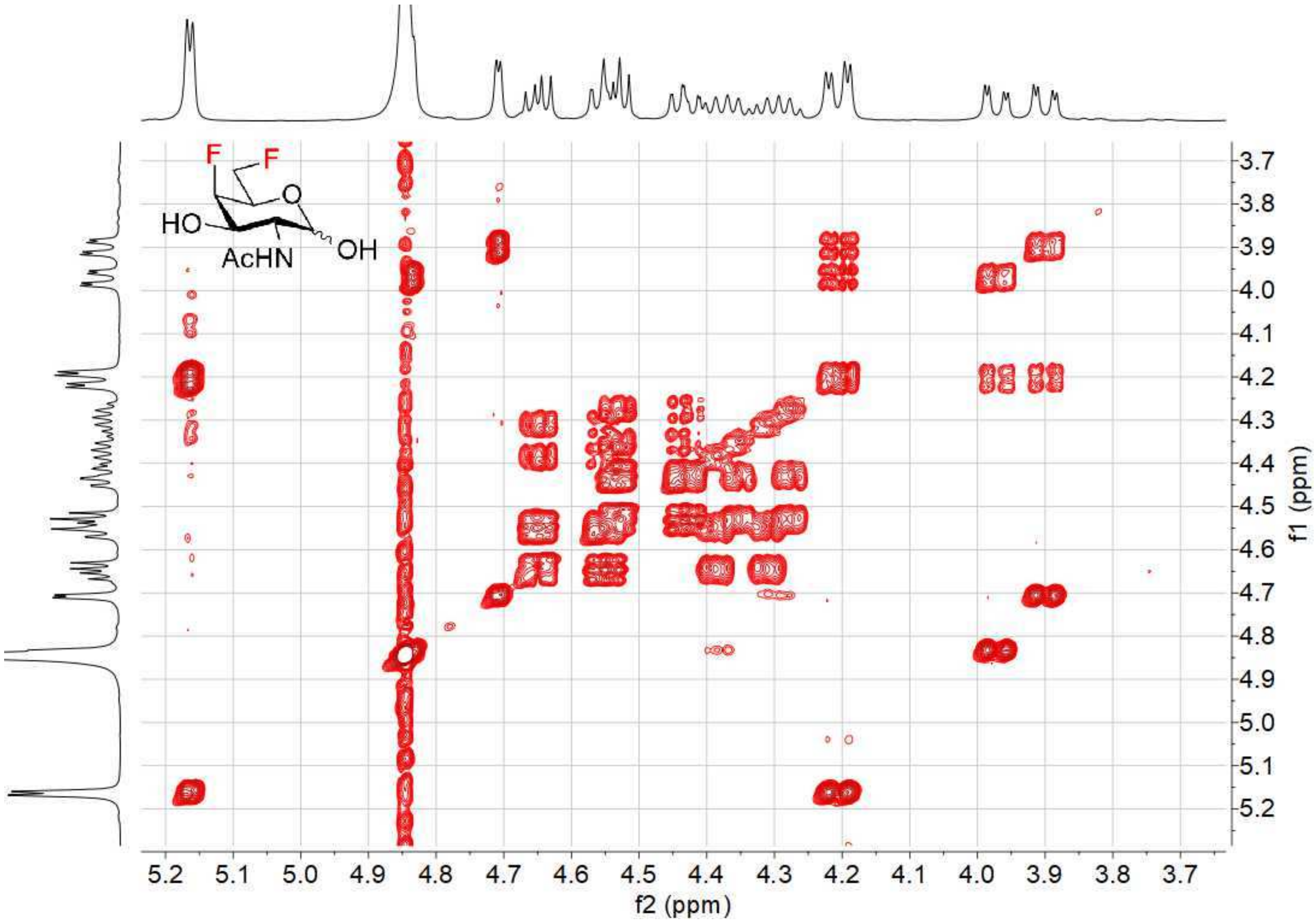
^{13}C NMR (100 MHz, CD_3OD) 69



[19]F NMR (376 MHz, CD_3OD) 69 (ca. 2-3% β-anomer)

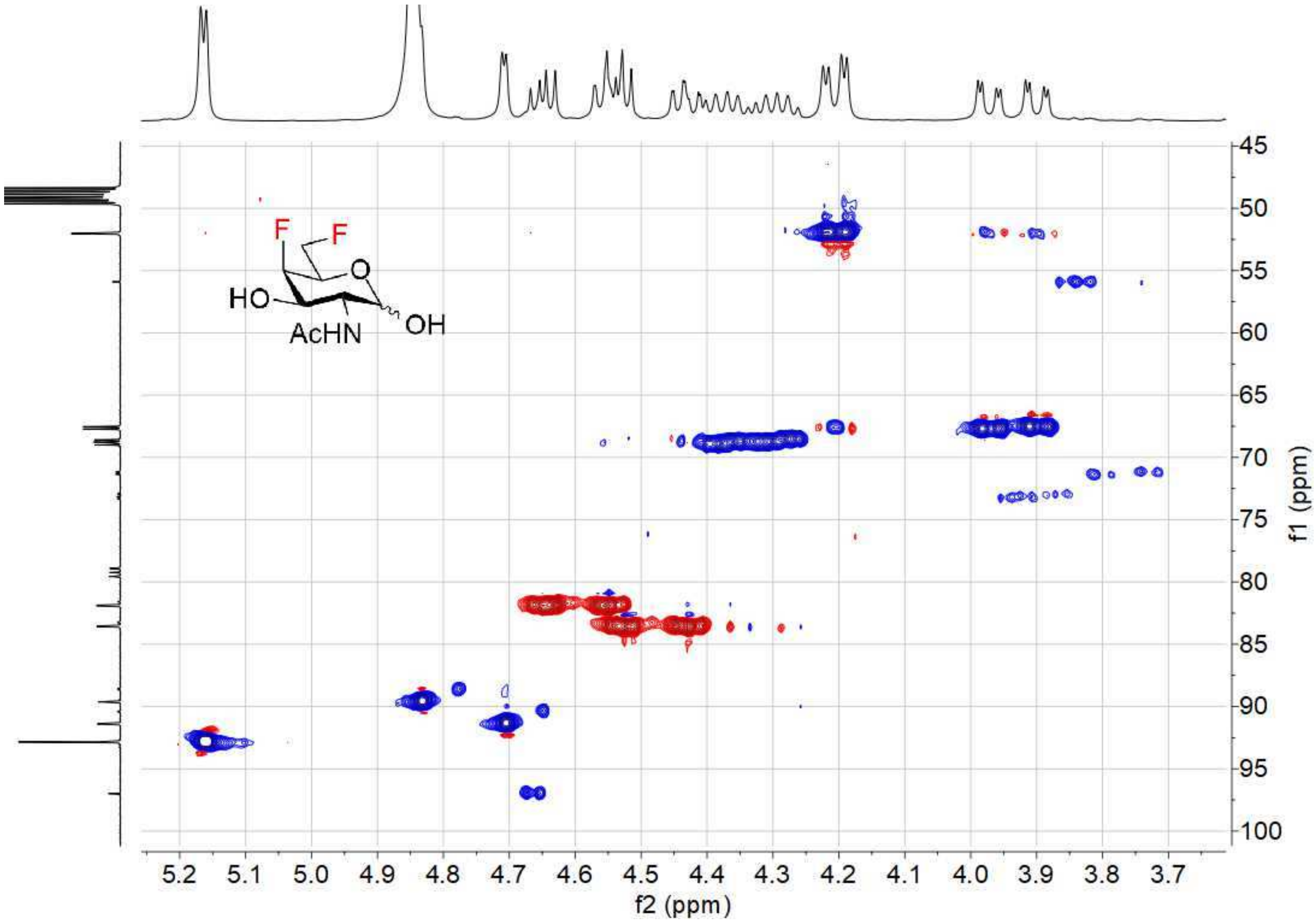


[1]H-[1]H COSY 69



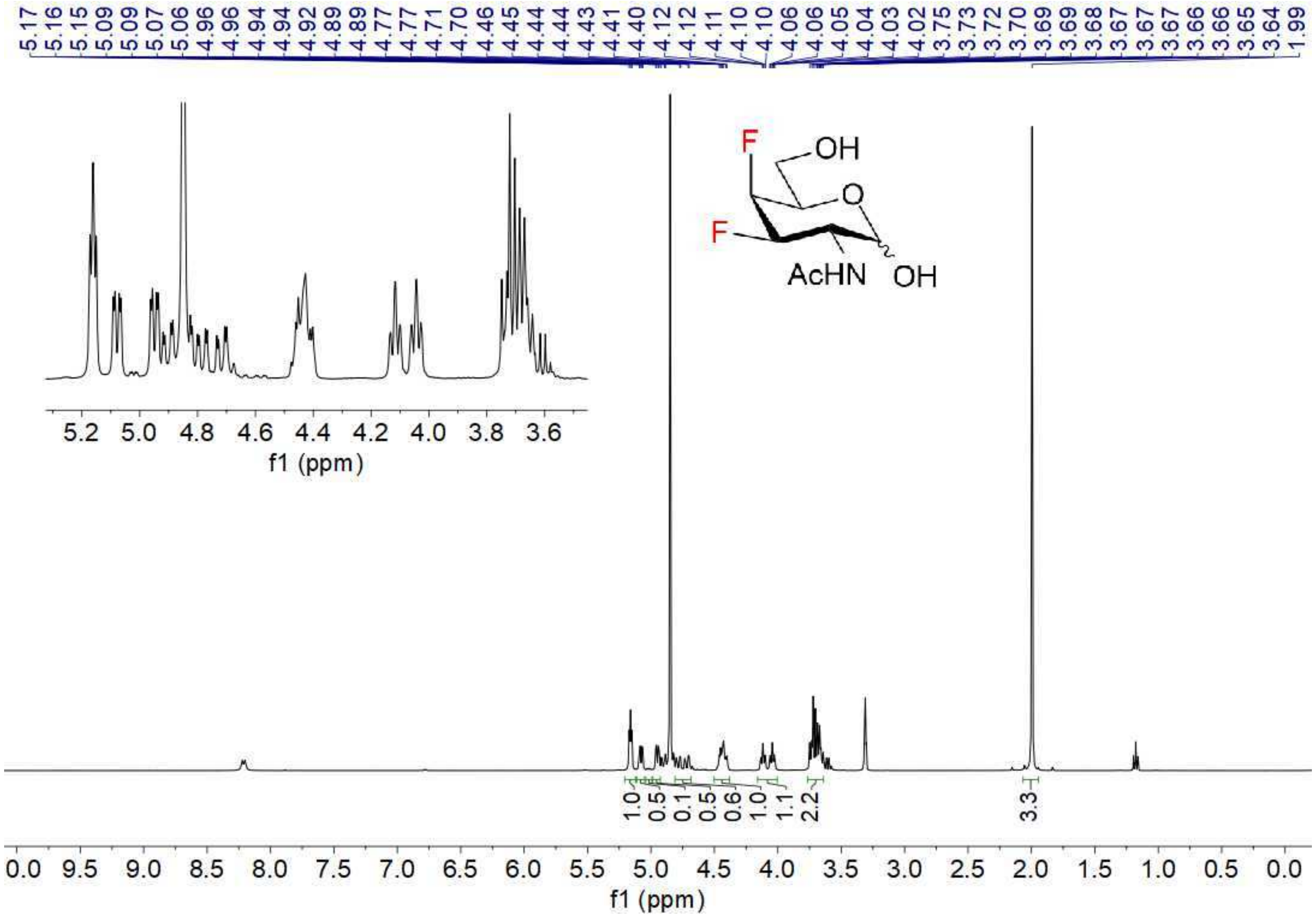
F
F
O
HO
AcHN
OH
5.2
5.1
5.0
4.9
4.8
4.7
4.6
4.5
4.4
4.3
4.2
4.1
4.0
3.9
3.8
3.7
f2 (ppm)
f1 (ppm)

^{1}H-^{13}C HSQC 69

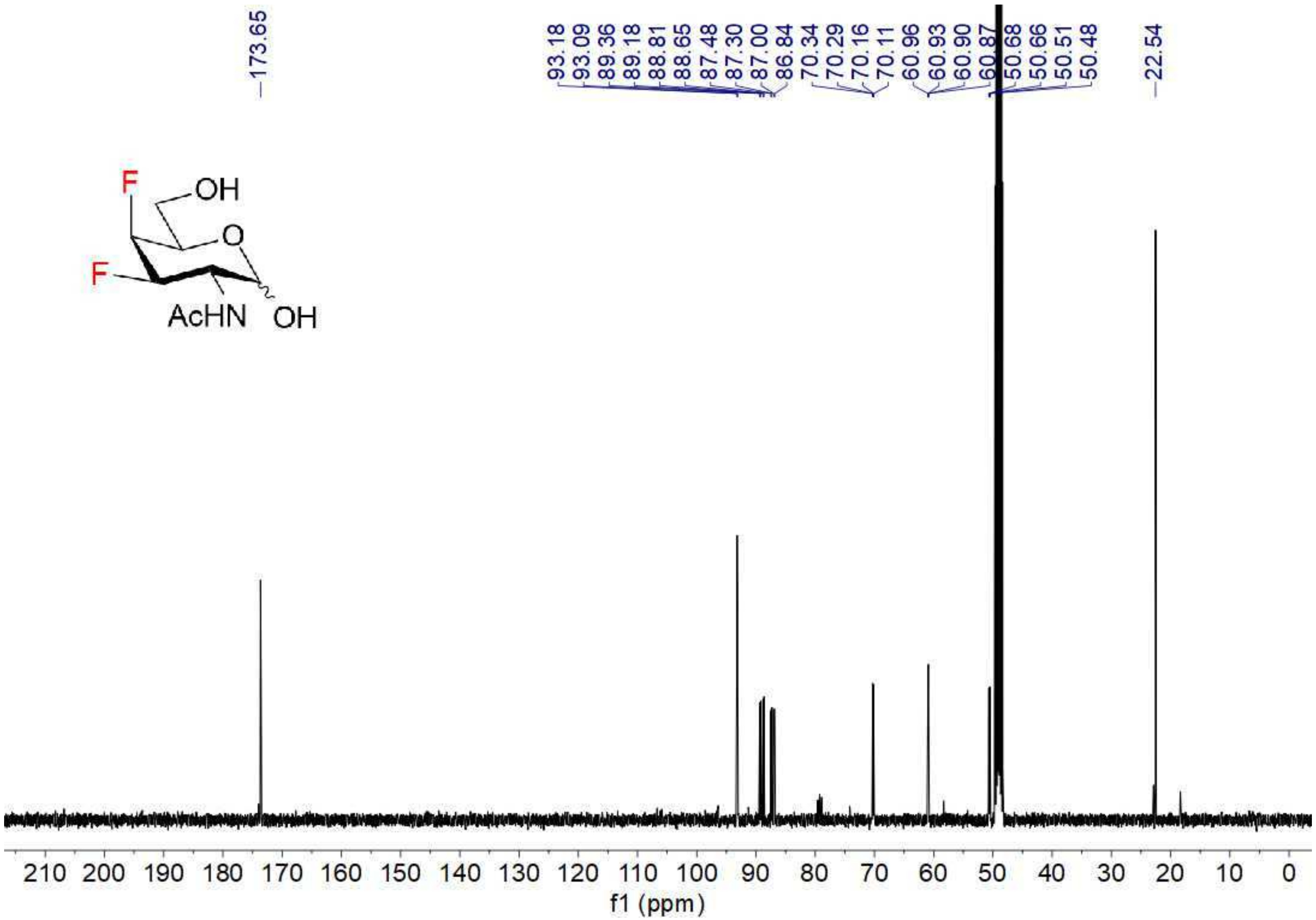


NMR COMPOUND 70

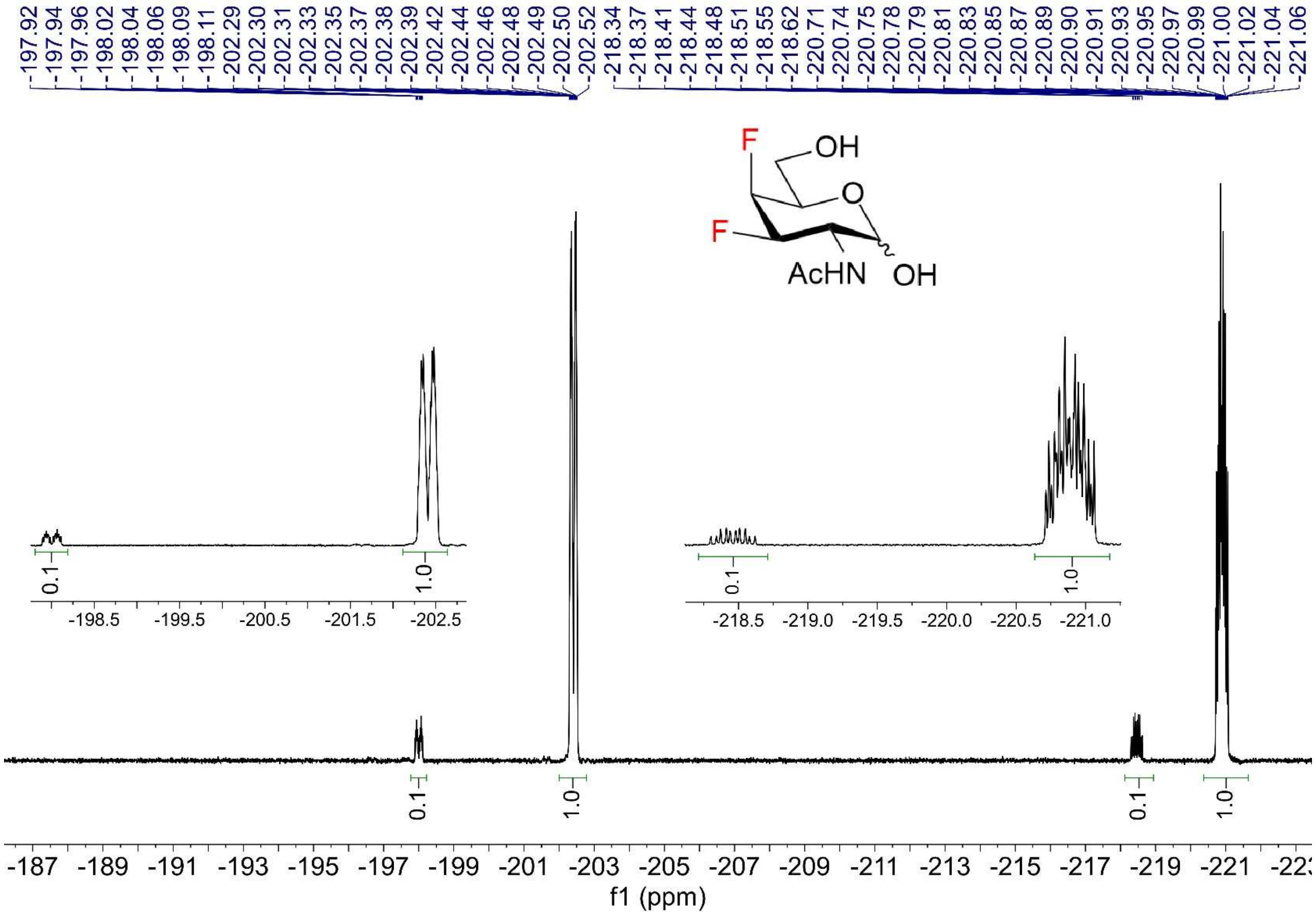
^{1}H NMR (400 MHz, CD_3OD) 70 (α/β ca. 10/1)



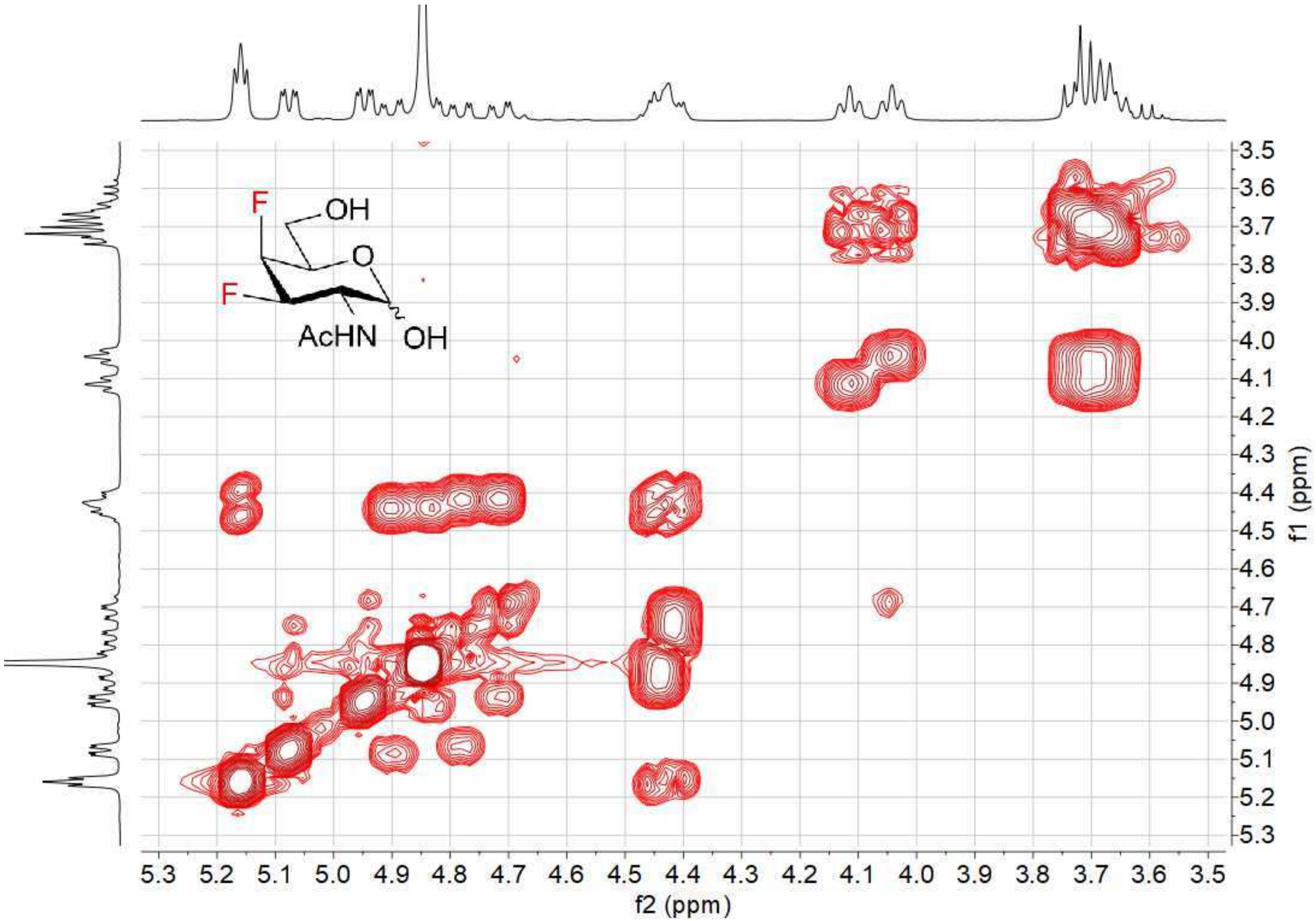
^{13}C NMR (100 MHz, CD_3OD) 70



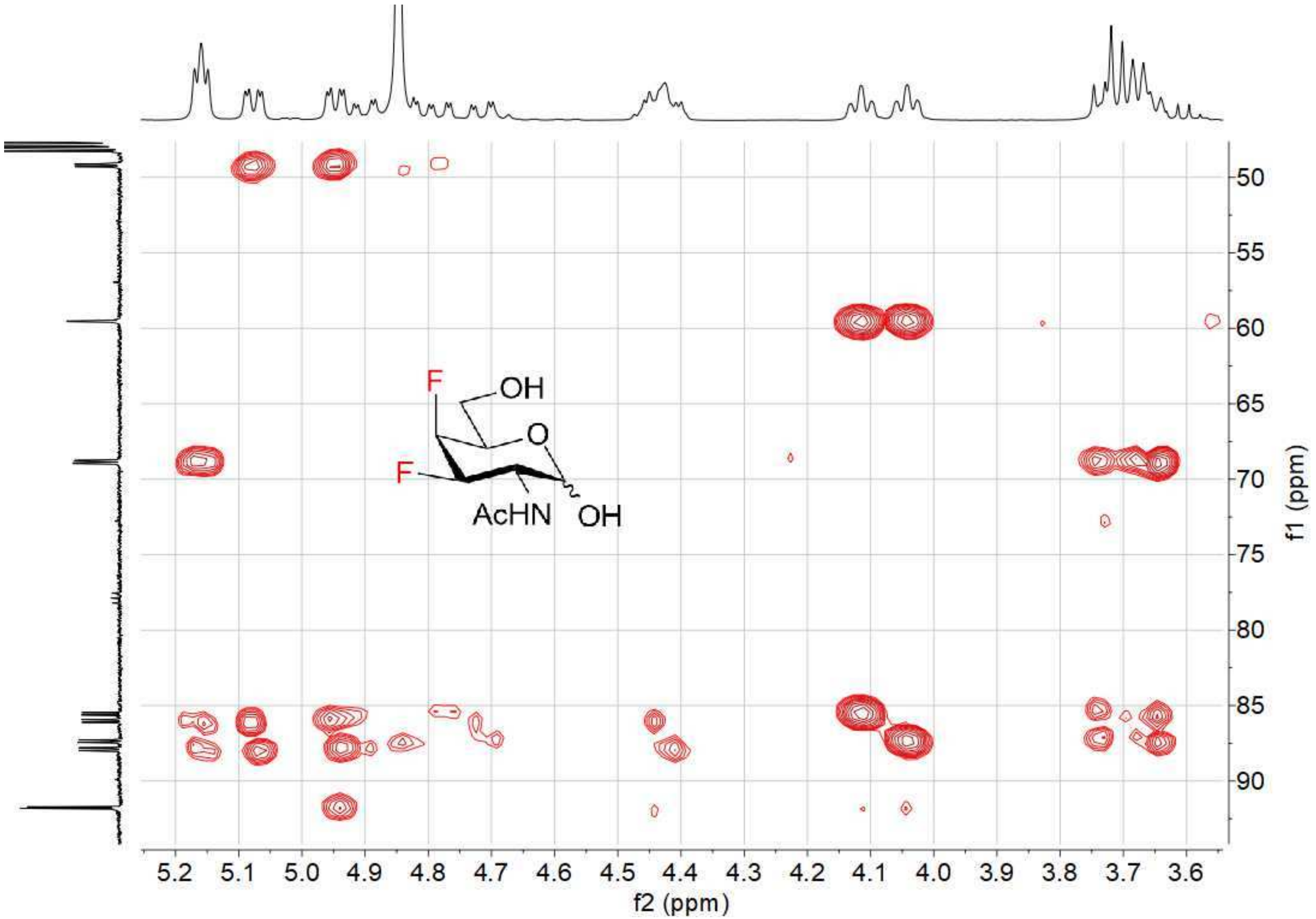
^{19}F NMR (376 MHz, CD$_3$OD) 70 (α/β ca. 10/1)



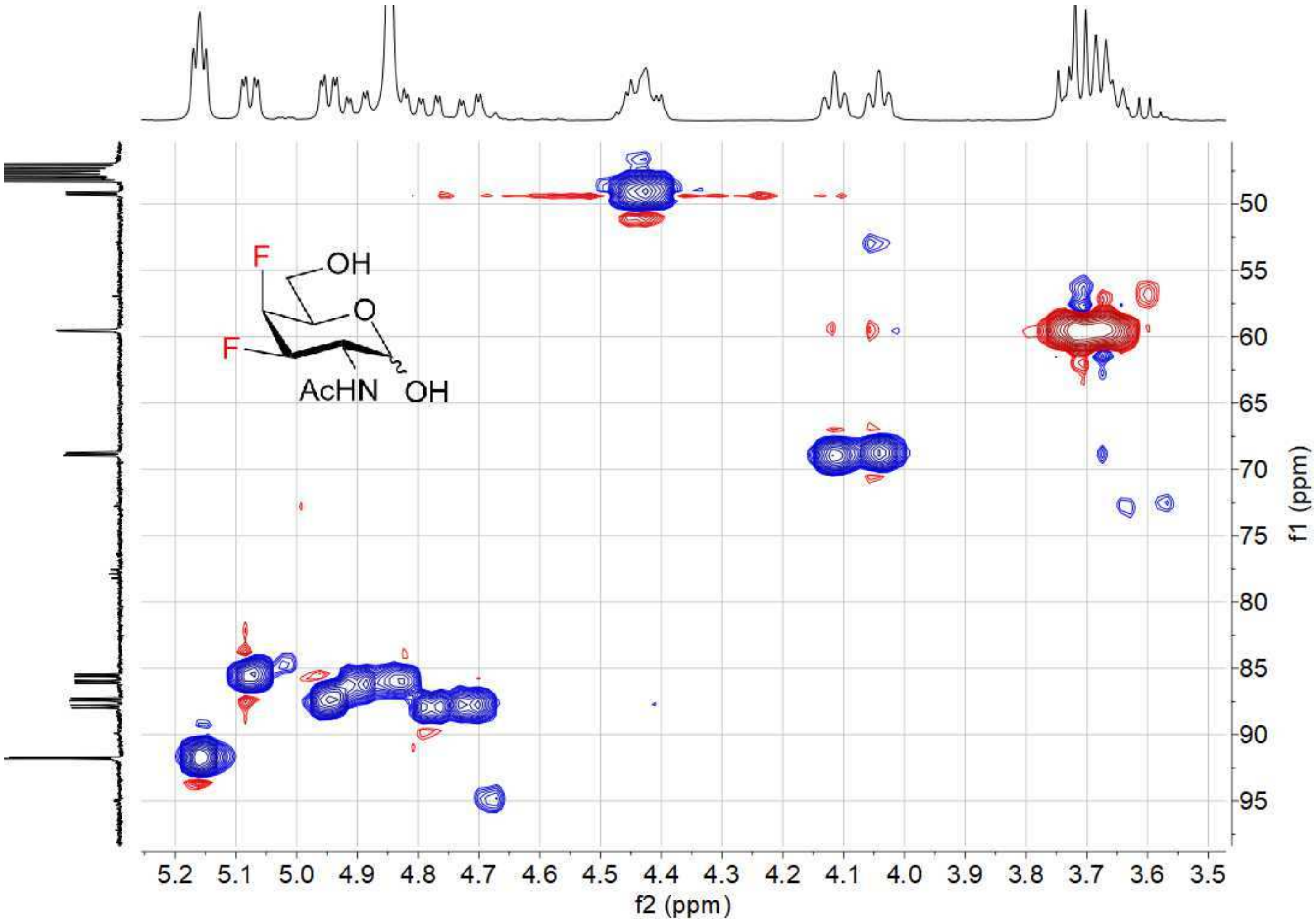
^{1}H-^{1}H COSY 70



^{1}H-^{13}C HMBC 70

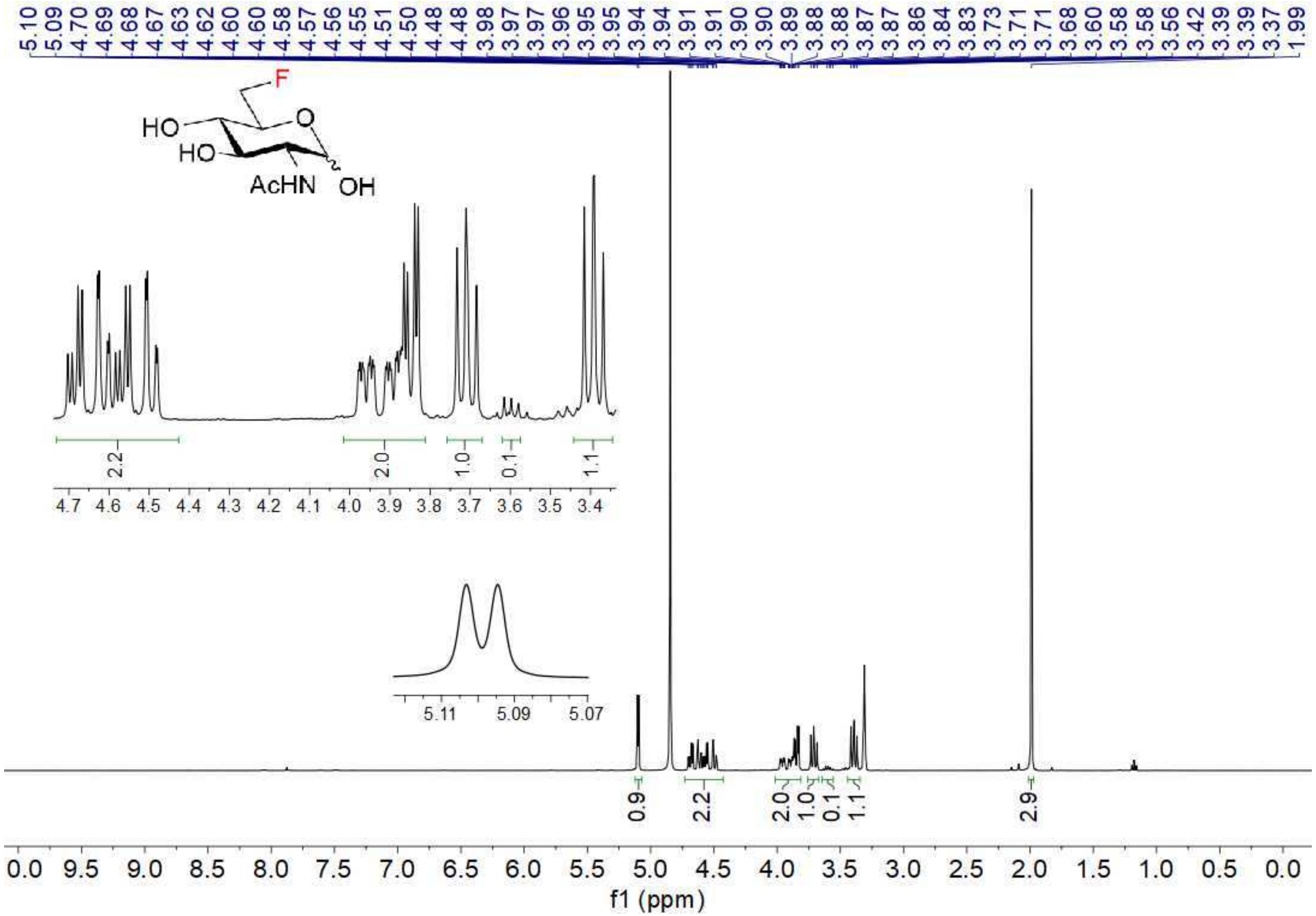


^{1}H-^{13}C HSQC 70

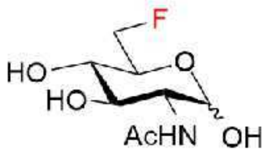


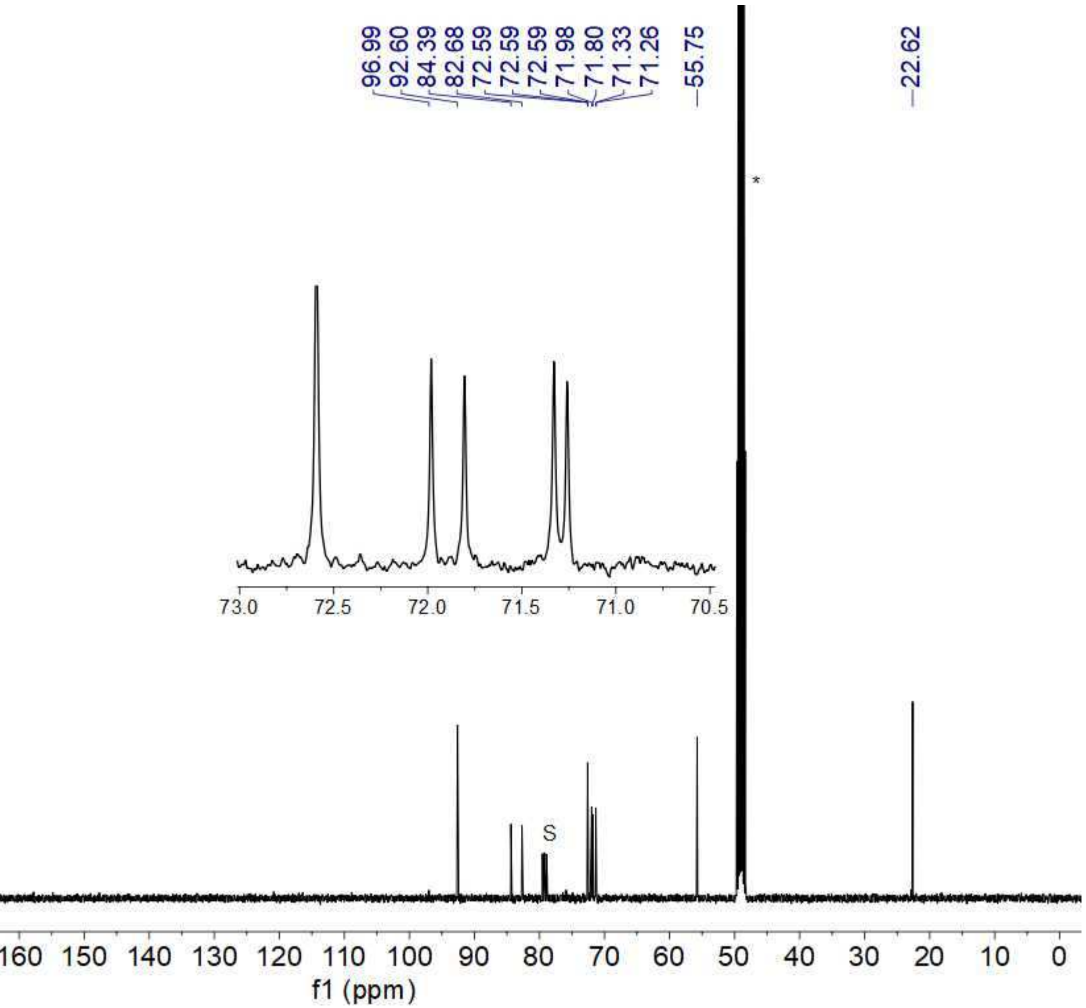
NMR COMPOUND 71

[1]H NMR (400 MHz, CD_3OD) 71 (ca. 8% β-anomer)

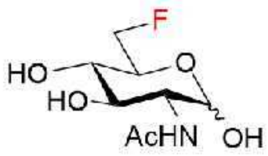


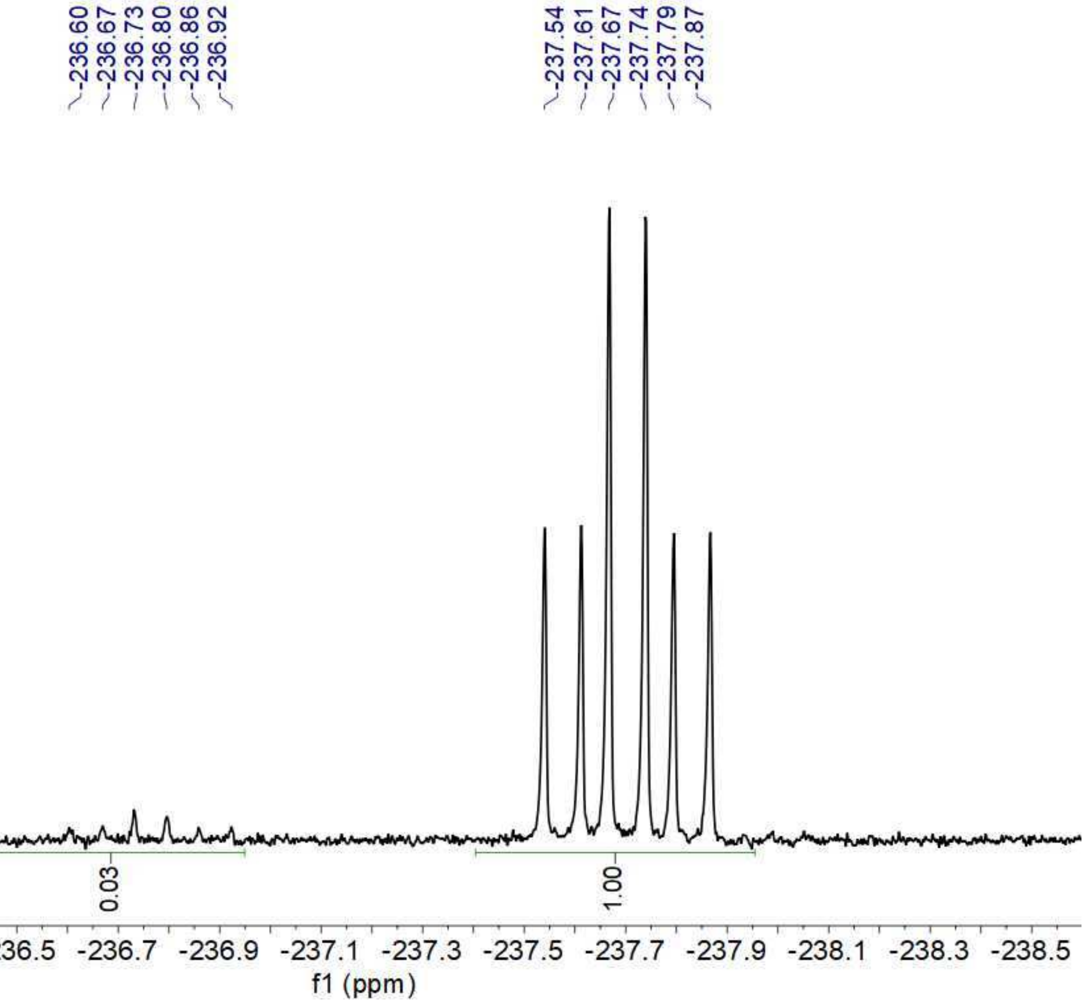
^{13}C NMR (100 MHz, CD_3OD) 71





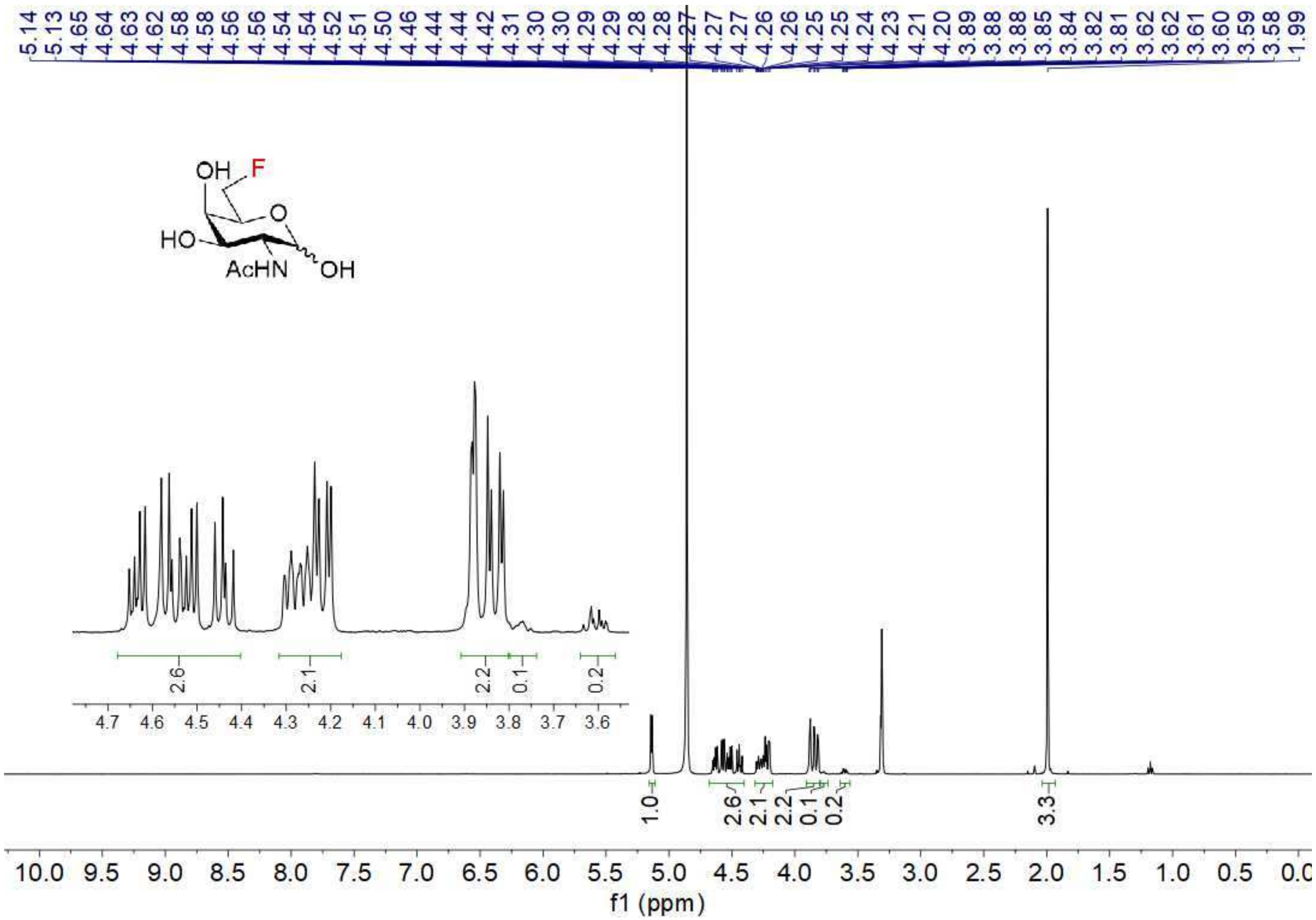
^{19}F NMR (376 MHz, CD_3OD) 71 (ca. 3% β-anomer)



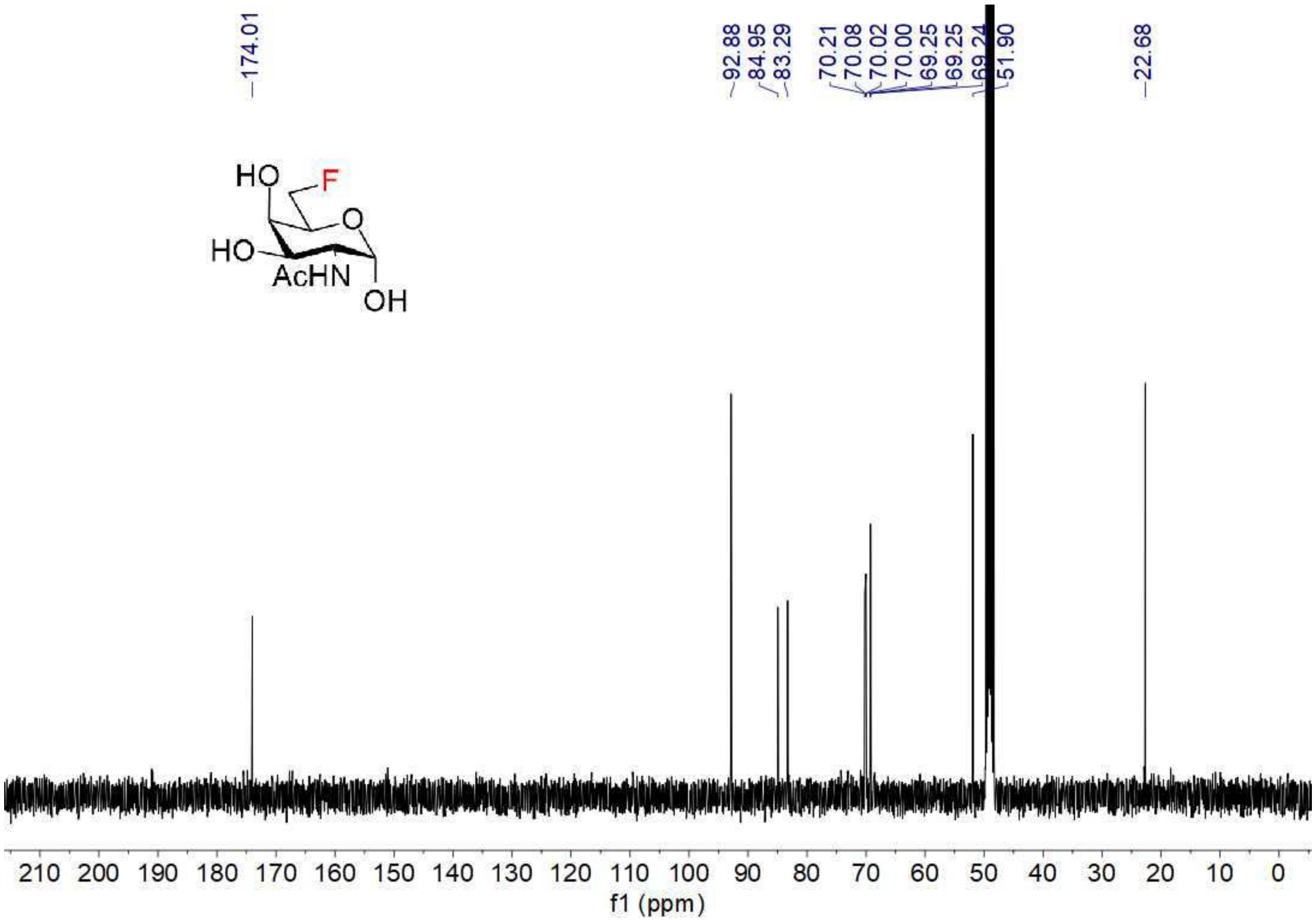


NMR COMPOUND 72

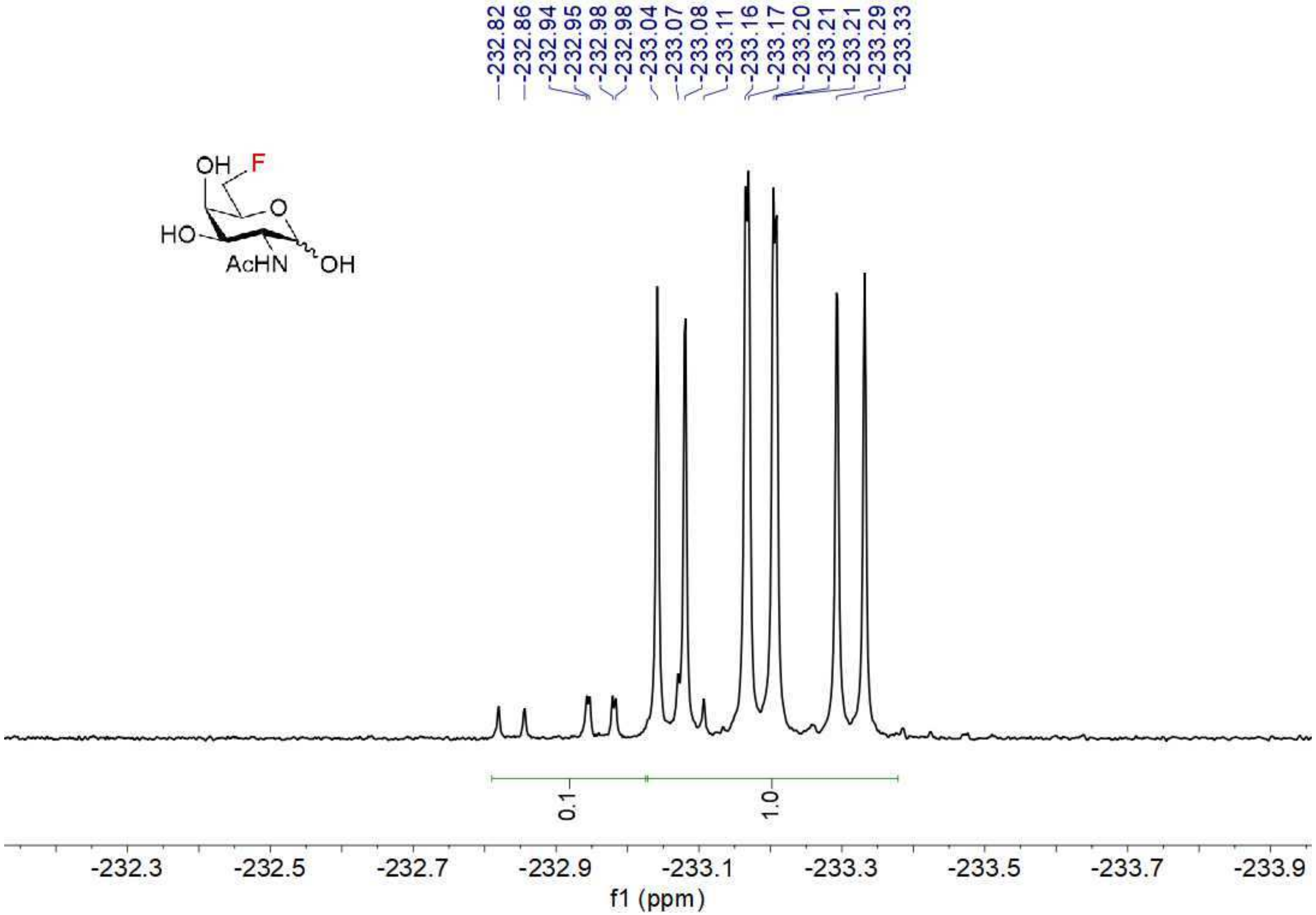
^{1}H NMR (400 MHz, CD_3OD) 72 (α/β ca. 10/1)



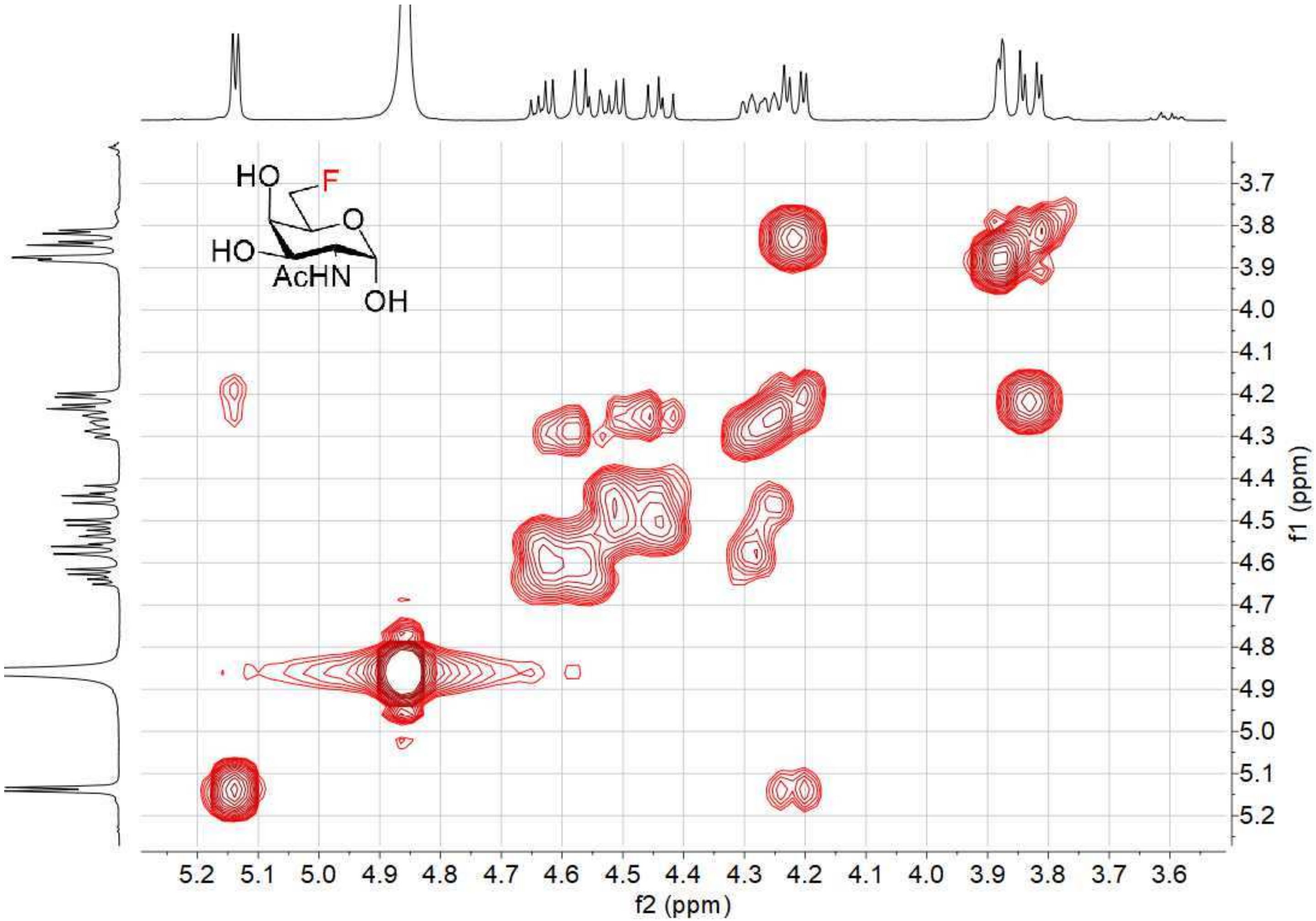
[13]C NMR (100 MHz, CD_3OD) 72



[19]F NMR (376 MHz, CD$_3$OD) 72 (α/β ca. 10/1)



[1]H-[1]H COSY 72



HO
F
O
HO
AcHN
OH
5.2 5.1 5.0 4.9 4.8 4.7 4.6 4.5 4.4 4.3 4.2 4.1 4.0 3.9 3.8 3.7 3.6
f2 (ppm)
3.7 3.8 3.9 4.0 4.1 4.2 4.3 4.4 4.5 4.6 4.7 4.8 4.9 5.0 5.1 5.2
f1 (ppm)

^{1}H-^{13}C HSQC 72