Electronic Supplementary Material (ESI) for New Journal of Chemistry.

Supporting Informations

University of Angers, MOLTECH-Anjou UMR CNRS 6200, SCL group, 2 boulevard Lavoisier, 49045 Angers cedex

E-mail: frederic.gohier@univ-angers.fr

Contents

Instrumentation and Chemicals

Characterization of the isolated products was carried out in $CDCl_3$ or $(CD_3)_2SO$ at 25°C. Chemical shifts are reported in ppm relative to the solvent residual value: δ=7.26 ($CDCl_3$), 2.50 ($(CD_3)_2SO$) for ^1H NMR and δ= 77.16 ($CDCl_3$), 39.52 ($(CD_3)_2SO$) for ^{13}C NMR. NMR spectra were recorded on a Bruker Avance 300 (^1H: 300.1 MHz, ^{13}C: 75.7 MHz, T = 300 K). Spectra were referenced against the internal NMR-solvent standard. Chemical shifts were expressed in parts per million (ppm) and were reported as s (singlet), d (doublet), t (triplet), m (multiplet) and coupling constants *J* were given in Hz. Mass spectra were recorded under EI mode on a VG-Autospec mass spectrometer. The main peaks are described according to m/z. The peak corresponding to molecular mass is expressed as ($M^{+\bullet}$). Analytical grade solvents were used.

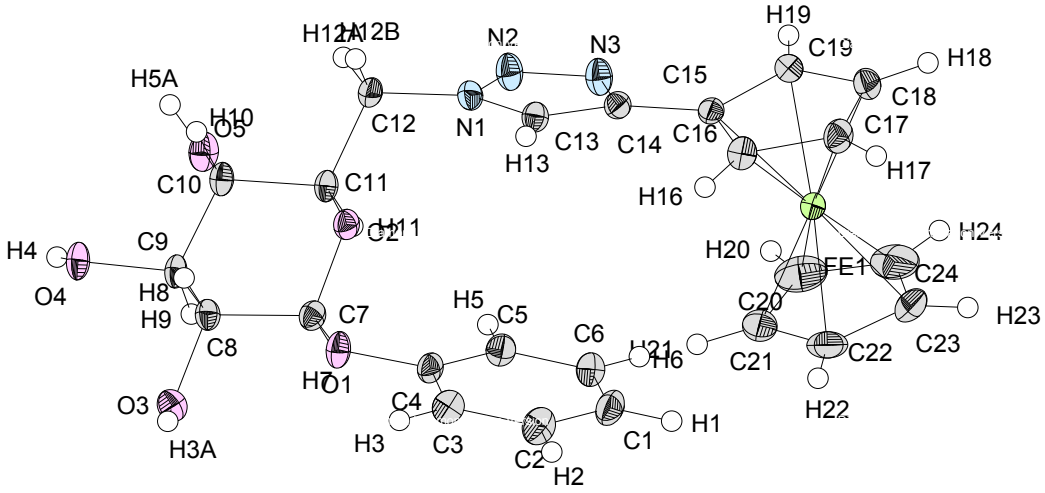
Structure refinement and crystal data

X-ray single-crystal diffraction data were collected at 150K on an Agilent SuperNova diffractometer equipped with Atlas CCD detector and micro-focus Cu-K_α radiation (λ = 1.54184 Å). The structure were solved by direct methods, expanded and refined on F^2 by full matrix least-squares techniques using SHELX programs (G. M. Sheldrick 2013-2016, SHELXS 2013/1 and SHELXL 2016/4). All non-H atoms were refined anisotropically and multiscan empirical absorption was corrected using CrysAlisPro program (CrysAlisPro, Agilent Technologies, V1.171.38.46, 2015). The H atoms were included in the calculation without refinement except for **15**, where water hydrogens were found by Fourier difference map then fixed.

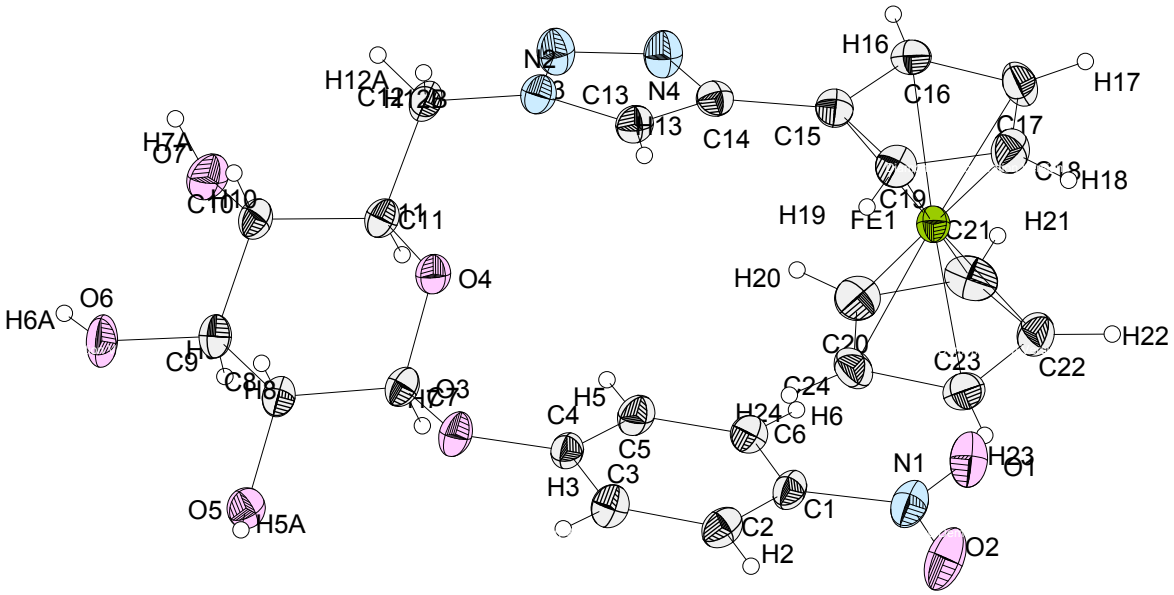
Table 1. Crystallographic data of **14**, **15**, **16**, **17** and **18**.

Crystal	**14 - FG204**	**15 - FAP057**	**16 - FG216**	**17 - FG206**	**18 - FAP068**
Formula	$C_{25}H_{29}FeN_3O_6$	$C_{25}H_{30}FeN_4O_9$	$C_{25}H_{28}FFeN_3O_6$	$C_{19}H_{23}FeN_3O_5$	$C_{24}H_{26}FeN_4O_5$
Molecular Weight	523.36	586.38	541.35	429.25	506.34
Temperature (K)	150	150	150	150	150
Crystal system	Orthorhombic	Orthorhombic	Monoclinic	Monoclinic	Orthorhombic
Space group	P 21 21 21	P 21 21 21	P 21	C 2	P 21 21 21
a (Å)	7.2219(2)	7.3870(1)	7.9733(2)	48.634(6)	5.6460(1)
b (Å)	8.0816(2)	9.4146(2)	7.1751(2)	5.7205(3)	11.2718(2)
c (Å)	39.3464(9)	36.6094(6)	20.4219(6)	13.3273(9)	34.7200(5)
β (°)	90	90	90.648(2)	95.447(8)	90
V ($Å^3$)	2296.4(1)	2546.02(8)	1168.25(6)	3691.1(5)	2209.60(6)
Z	4	4	2	8	4
Crystal color	Yellow	Yellow	Yellow	Yellow	Orange
Crystal size (mm^3)	0.13x0.03x0.02	0.37x0.16x0.08	0.21x0.09x0.04	0.30x0.12x0.03	0.28x0.21x0.09
D_c (g cm^{-3})	1.514	1.530	1.539	1.545	1.522
F(000)	1096	1224	564	1792	1056
μ (mm^{-1})	5.680	5.294	5.668	6.884	5.863
Transmission (min/max)	0.7717/1.000	0.5570/1.000	0.8054/1.000	0.5716/1.000	0.3999/1.000
θ (min/max) (°)	4.495/72.681	4.832/76.382	4.330/72.533	3.331/72.669	2.545/76.354
Data collected	8346	7709	4410	10919	10576
Data unique	4400	4688	3224	5598	4534
Data observed	4262	4526	3080	5010	4394
R (int)	0.0296	0.0339	0.0274	0.0495	0.0350
Parameters	321	360	330	513	311
R_1 [I > 2σ(I)]	0.0299	0.0311	0.0377	0.0510	0.0324
wR_2 [I > 2σ(I)]	0.0711	0.0760	0.0920	0.1354	0.0816
R_1 [all data]	0.0315	0.0336	0.0402	0.0560	0.0351
wR_2 [all data]	0.0718	0.0794	0.0939	0.1422	0.0853
GOF	1.061	1.025	1.084	1.026	1.070
Absolute structure parameter	0.002(2)	-0.007(3)	0.004(6)	-0.022(5)	-0.006(3)
Largest diff. peak/hole (e A^{-3})	0.310/-0.341	0.323/-0.301	0.488/-0.251	0.854/-0.519	0.341/-0.360
CCDC number	1896743	1896741	1896751	1896744	1896742

ORTEP representation 14



ORTEP representation 15



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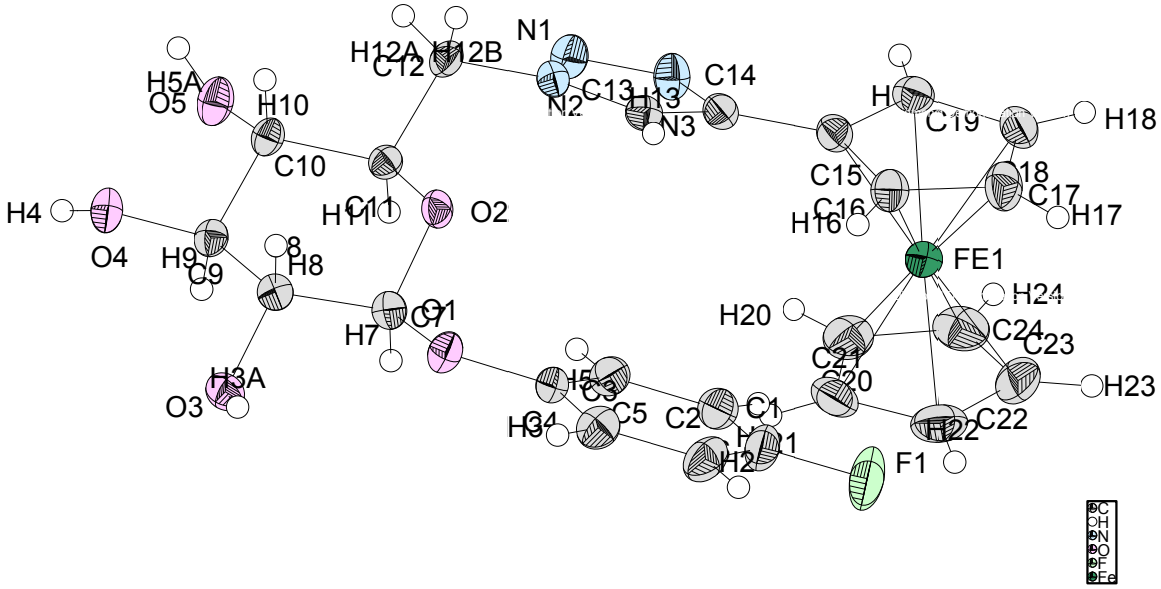

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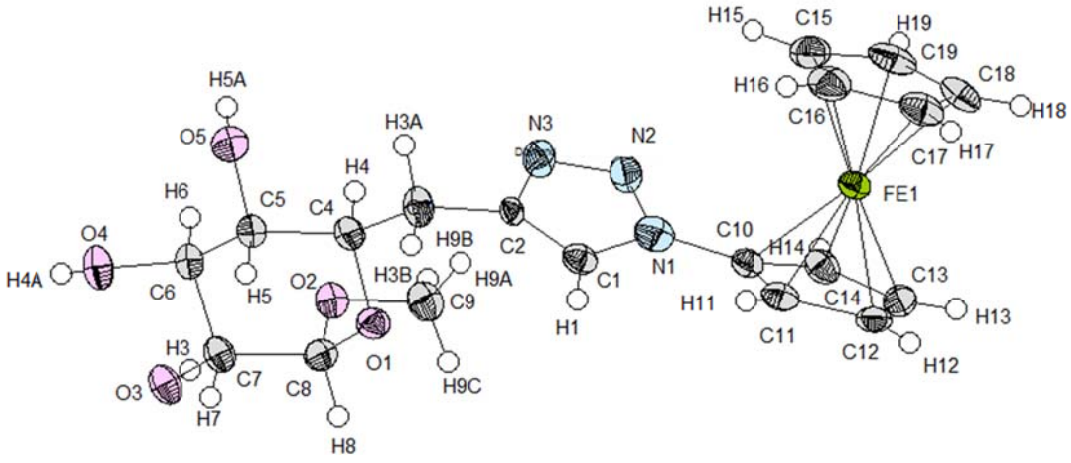

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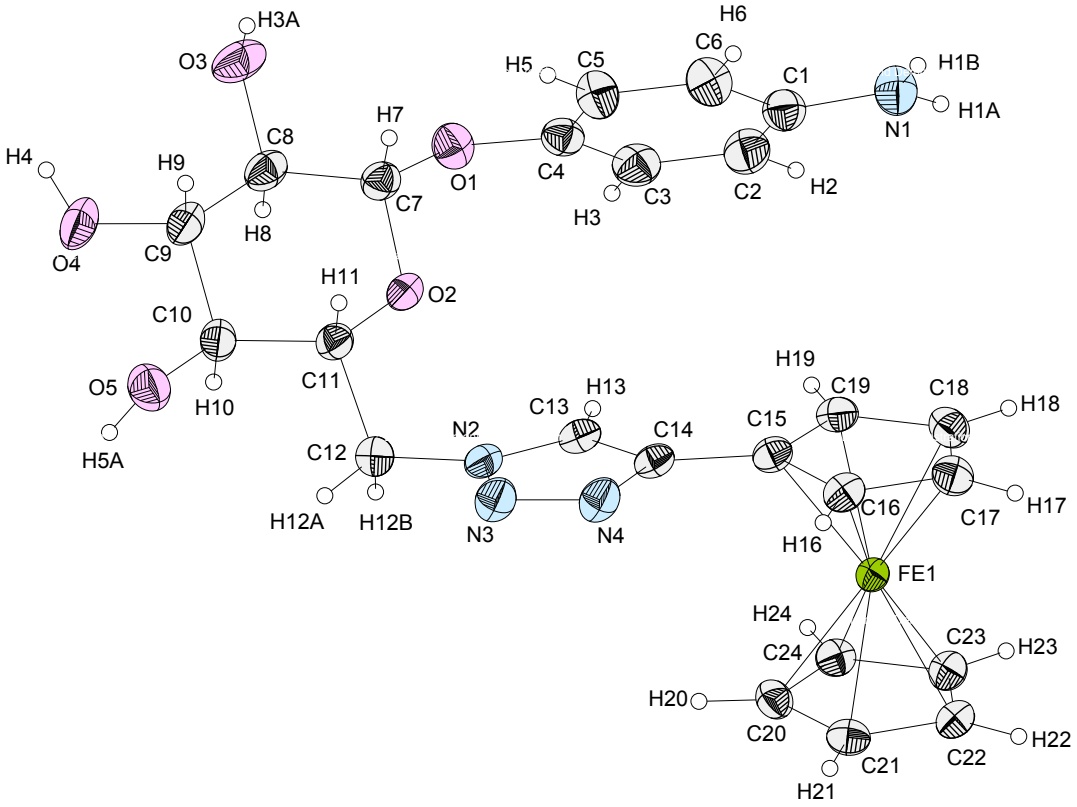

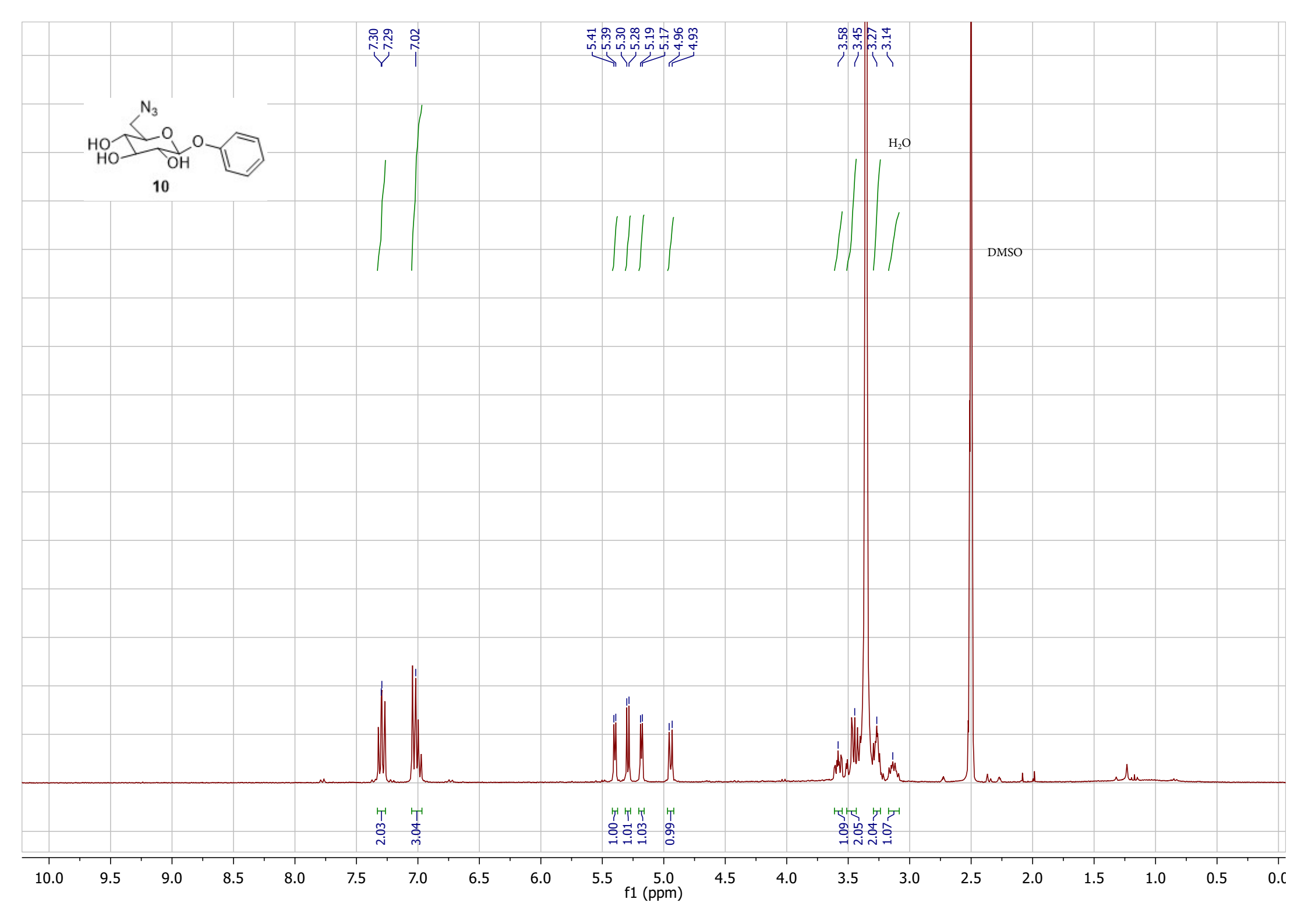
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5.30
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5.19
5.17
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4.93
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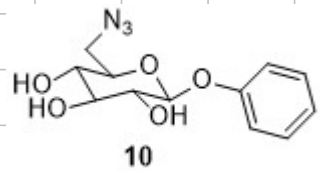
3.45
3.27
3.14
H2O
DMSO
N3
HO
HO
O
O
OH
10
2.03
3.04
1.00
1.01
1.03
0.99
1.09
2.05
2.04
1.07
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

FG 220 c.12.fid



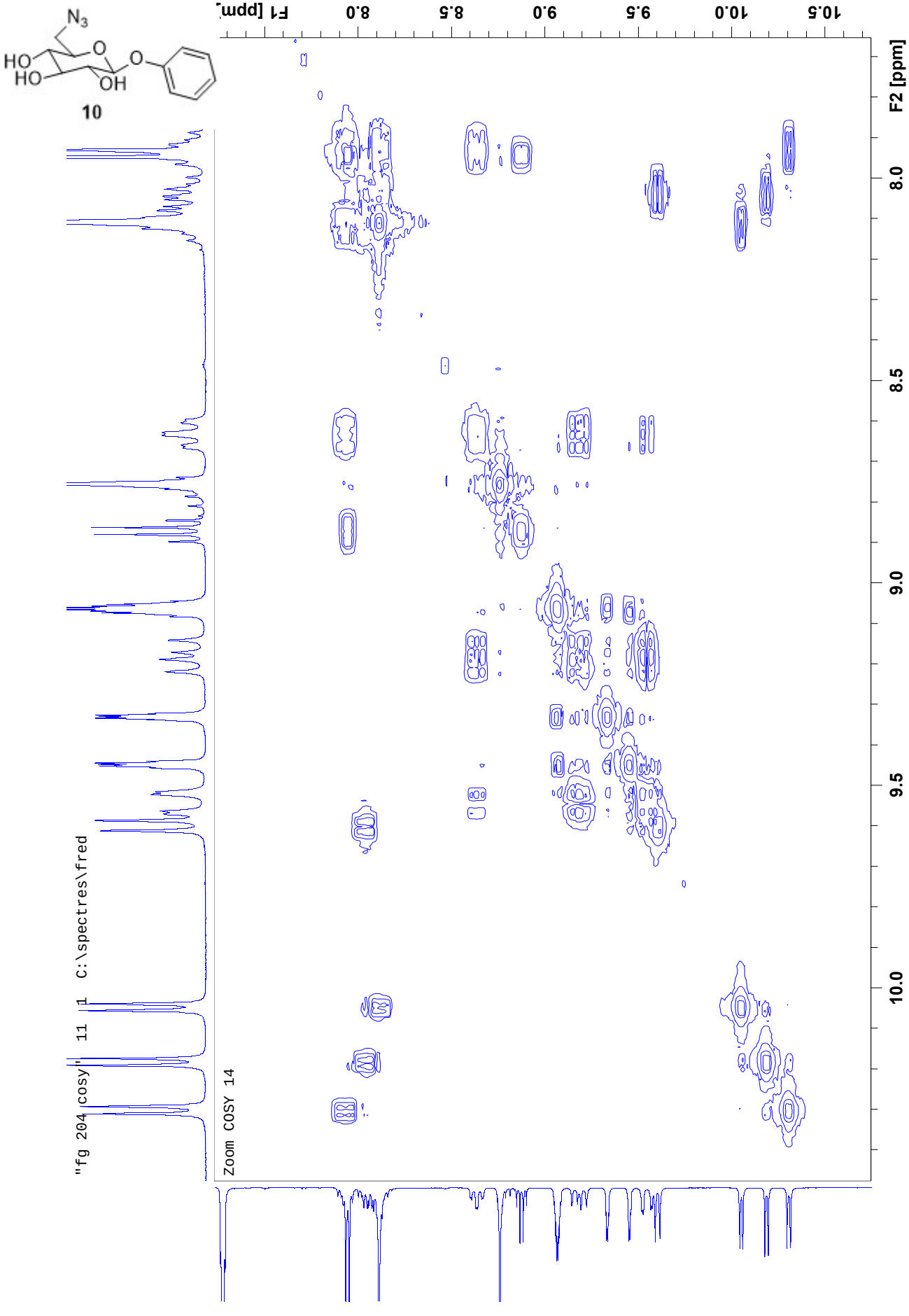
10

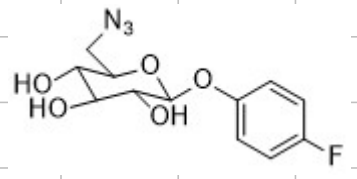
157.2
129.4
122.0
116.3
100.2
76.1
75.1
73.2
70.6
51.4

240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

f1 (ppm)

10


N3
HO
HO
O
O
OH
10
"fg 204 cosy" 11 1 C:\spectres\fred
Zoom COSY 14
F1 [ppm]
F2 [ppm]
10.5
10.0
9.5
9.0
8.5
8.0



12

7.12
7.06
5.43
5.42
5.30
5.29
5.20
5.19
4.91
4.90
3.58
3.46
3.27
3.26
3.14
3.14
3.13
3.13

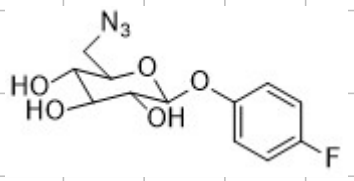
DMSO

2.03
2.02
0.97
0.98
1.00
0.97
1.05
2.05
2.04
0.97

9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5

f1 (ppm)

12



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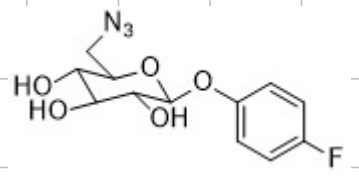
153.48
153.45
118.01
117.90
115.93
115.62
100.82
76.03
75.14
73.14
70.62
51.36

300000
280000
260000
240000
220000
200000
180000
160000
140000
120000
100000
80000
60000
40000
20000
0
-20000

240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

f1 (ppm)

12



-122.12

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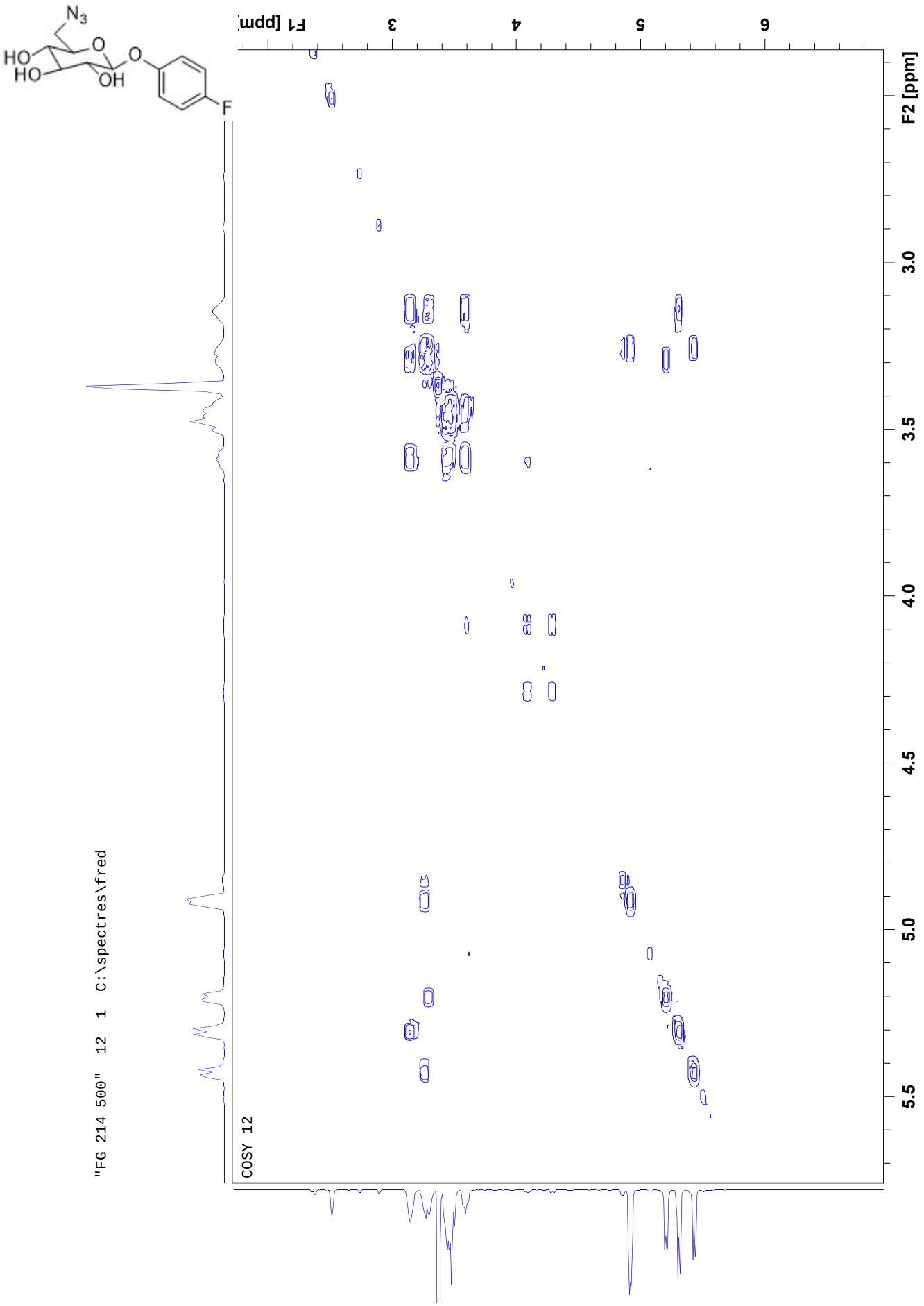
f1 (ppm)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HO
O
O
OH
F
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6
5
4
3
F2 [ppm]
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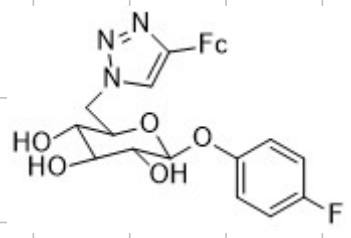
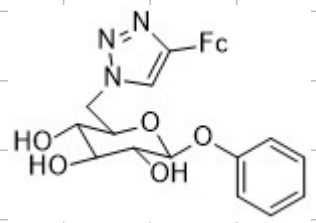
3.5
4.0
4.5
5.0
5.5
COSY 12
"FG 214 500" 12 1 C:\spectres\fred


N
N
Fc
O
HO
HO
OH
O
F

14

7.95
7.15
7.12
7.10
6.92
6.78
6.75
5.53
5.51
5.41
5.39
5.27
5.25
4.84
4.81
4.68
4.56
4.45
4.42
4.40
4.37
4.29
3.98
3.86
3.30
3.16
DMSO
MeOH
MeOH
1.01
2.08
1.09
2.05
1.00
1.02
1.01
1.01
1.02
1.05
0.99
1.09
2.03
4.98
1.04
0.94
1.05
0.98
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.
f1 (ppm)

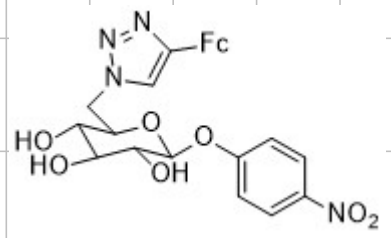
14



157.1
145.0
129.2
121.9
121.5
116.2
100.3
76.1
74.3
73.1
71.3
69.2
69.2
68.1
66.4
66.3
50.9

240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

f1 (ppm)



15

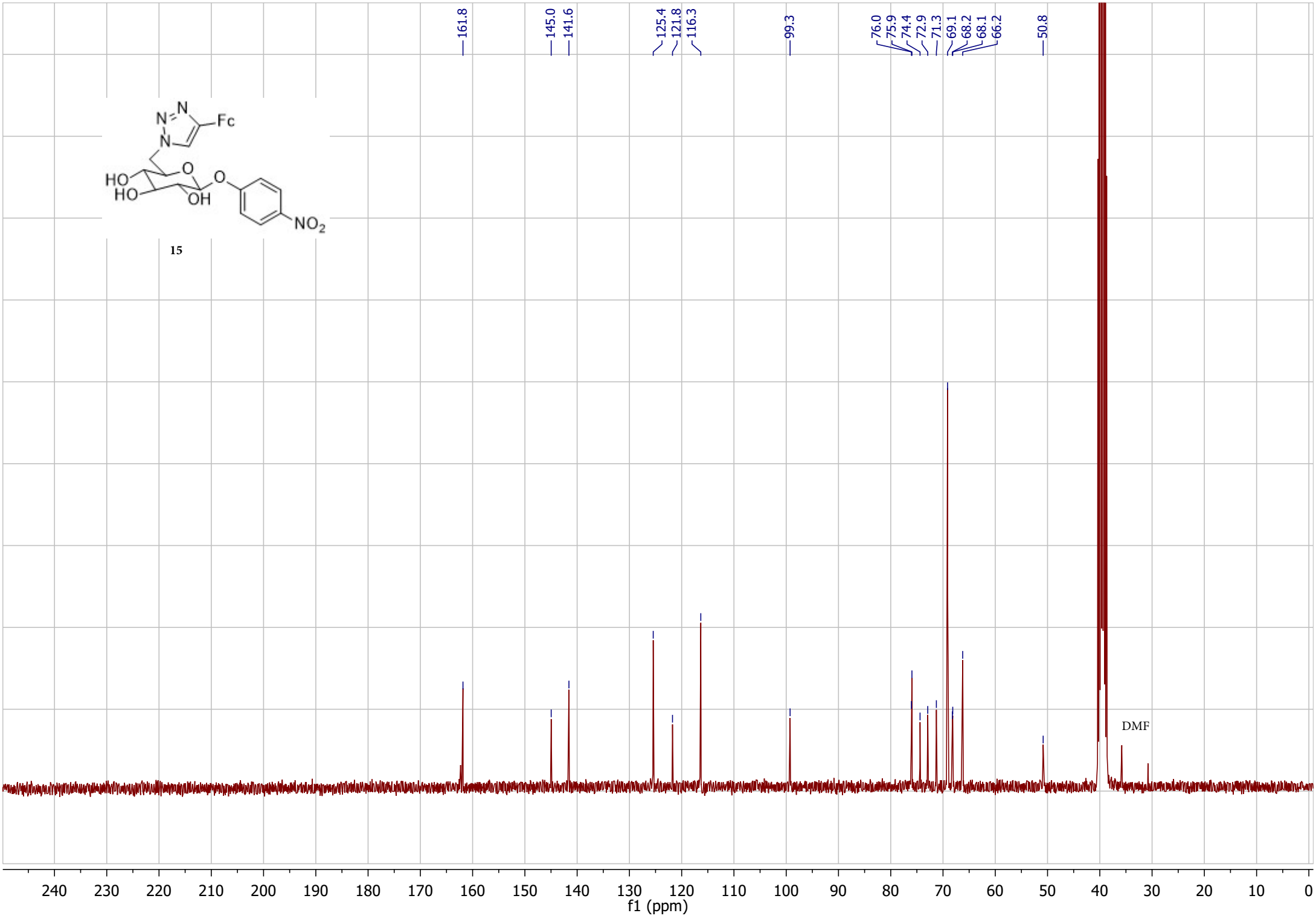
DMSO

DMF

f1 (ppm)

N
N
Fc
HO
HO
O
OH
O
NO2

15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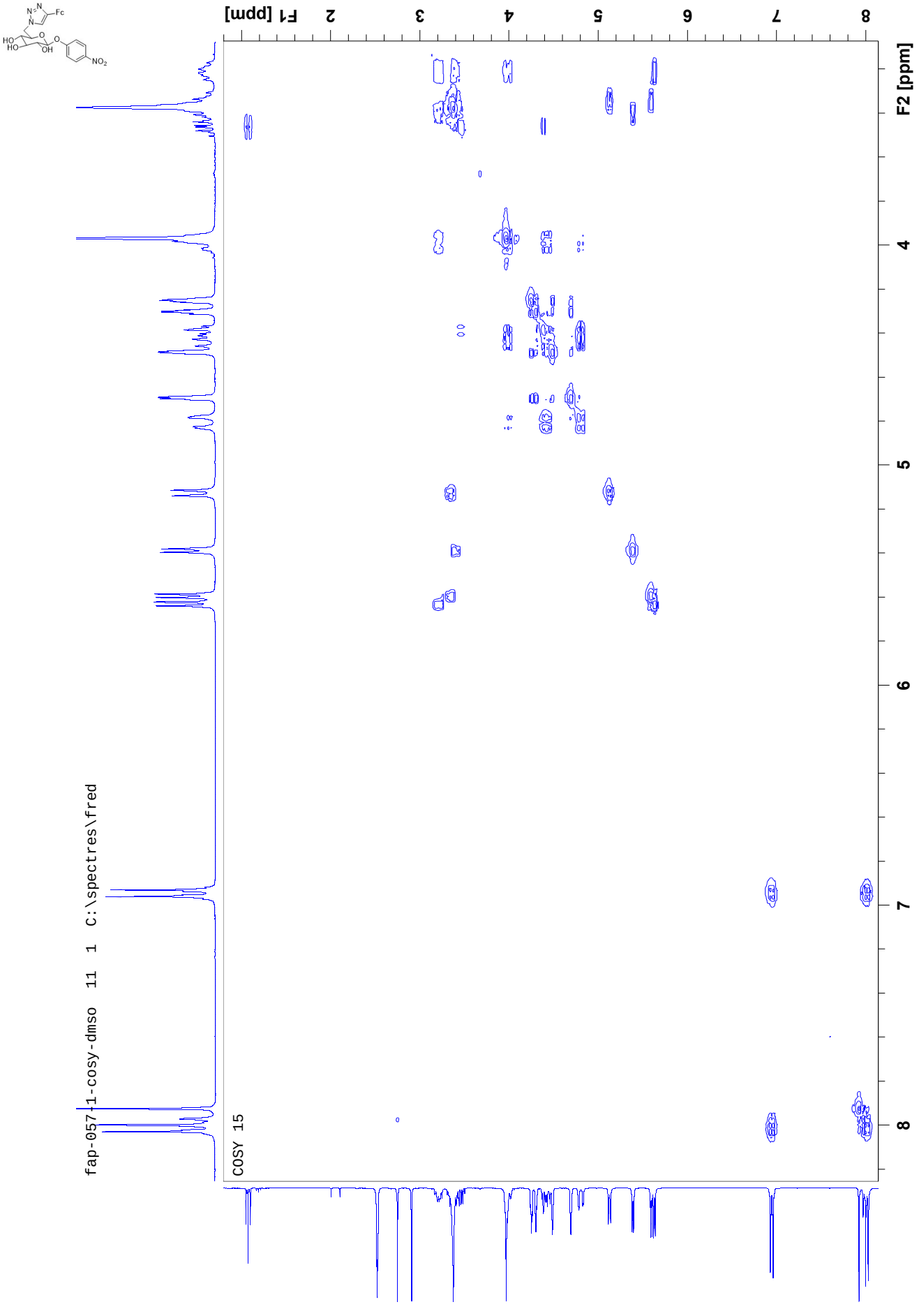
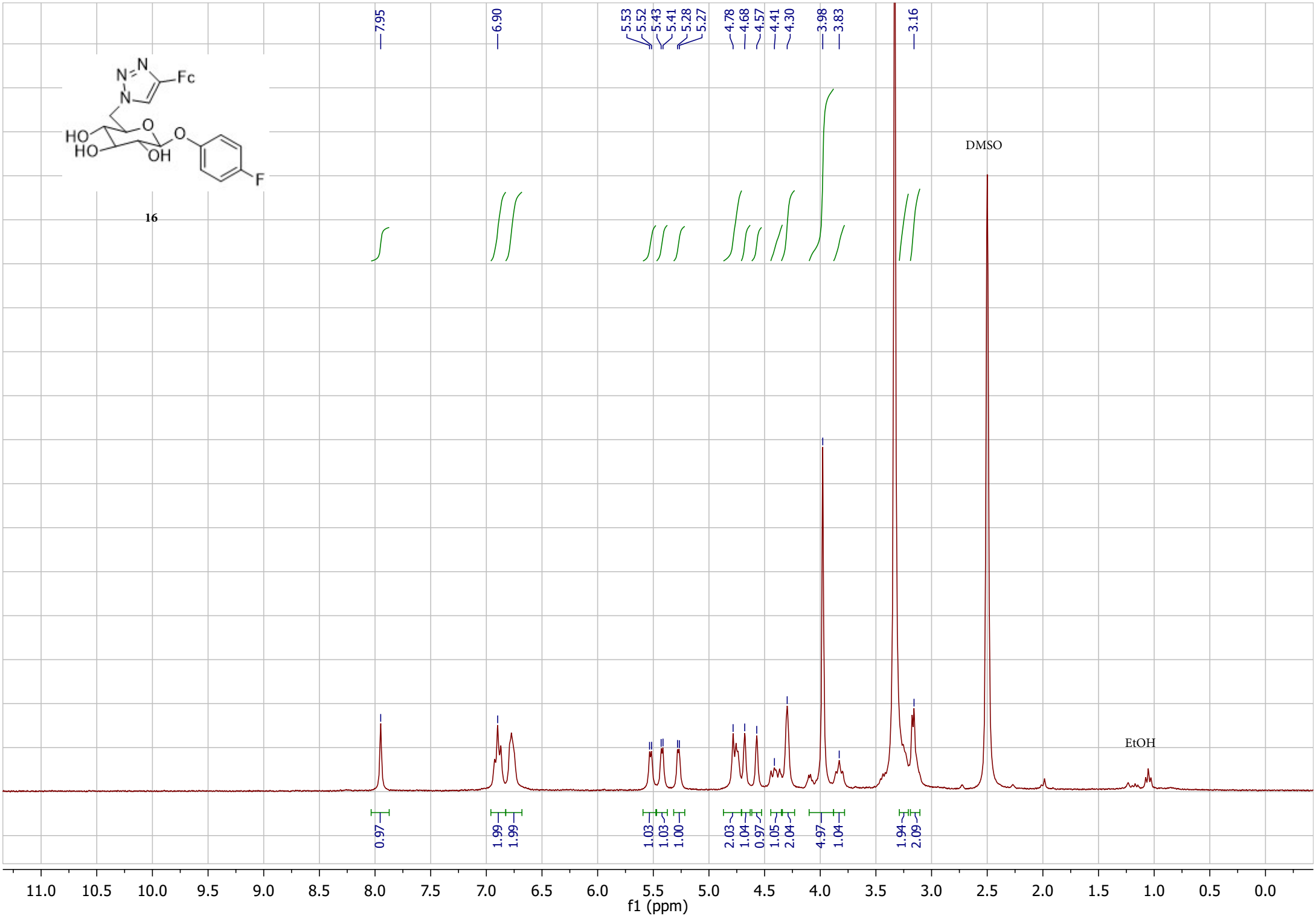
125.4
121.8
116.3
99.3
76.0
75.9
74.4
72.9
71.3
69.1
68.2
68.1
66.2
50.8
DMF
240
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


F1 [ppm]
F2 [ppm]
COSY 15
fap-057-1-cosy-dmso 11 1 C:\spectres\fred

N
N
Fc
HO
HO
O
O
OH
F

16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4.30
3.98
3.83
3.16
DMSO
EtOH
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1.99
1.99
1.03
1.03
1.00
2.03
1.04
0.97
1.05
2.04
4.97
1.04
1.94
2.09
11.0
10.5
10.0
9.5
9.0
8.5
8.0
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6.5
6.0
5.5
5.0
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4.0
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3.0
2.5
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1.5
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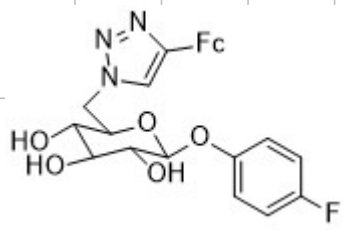
0.0
f1 (ppm)

16

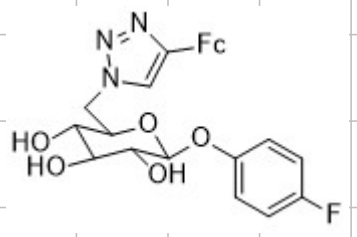


158.8
155.6
153.3
153.3
145.0
121.7
117.9
117.8
115.8
115.5
100.8
76.1
76.1
74.3
73.1
71.3
69.2
68.2
68.2
66.3
66.3
50.9

MeOH

240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

f1 (ppm)



16

-121.92

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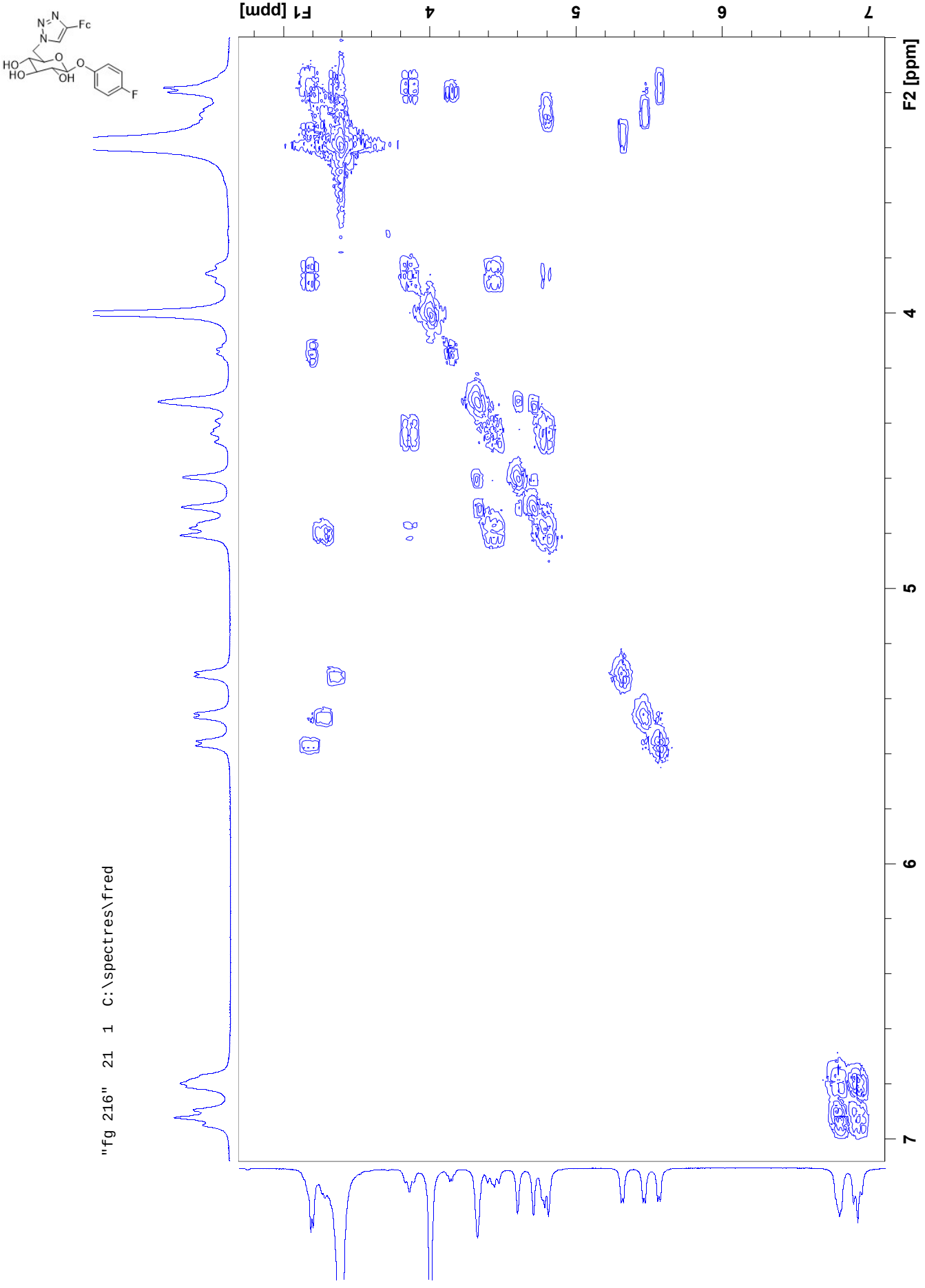
f1 (ppm)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6
5
4
F2 [ppm]
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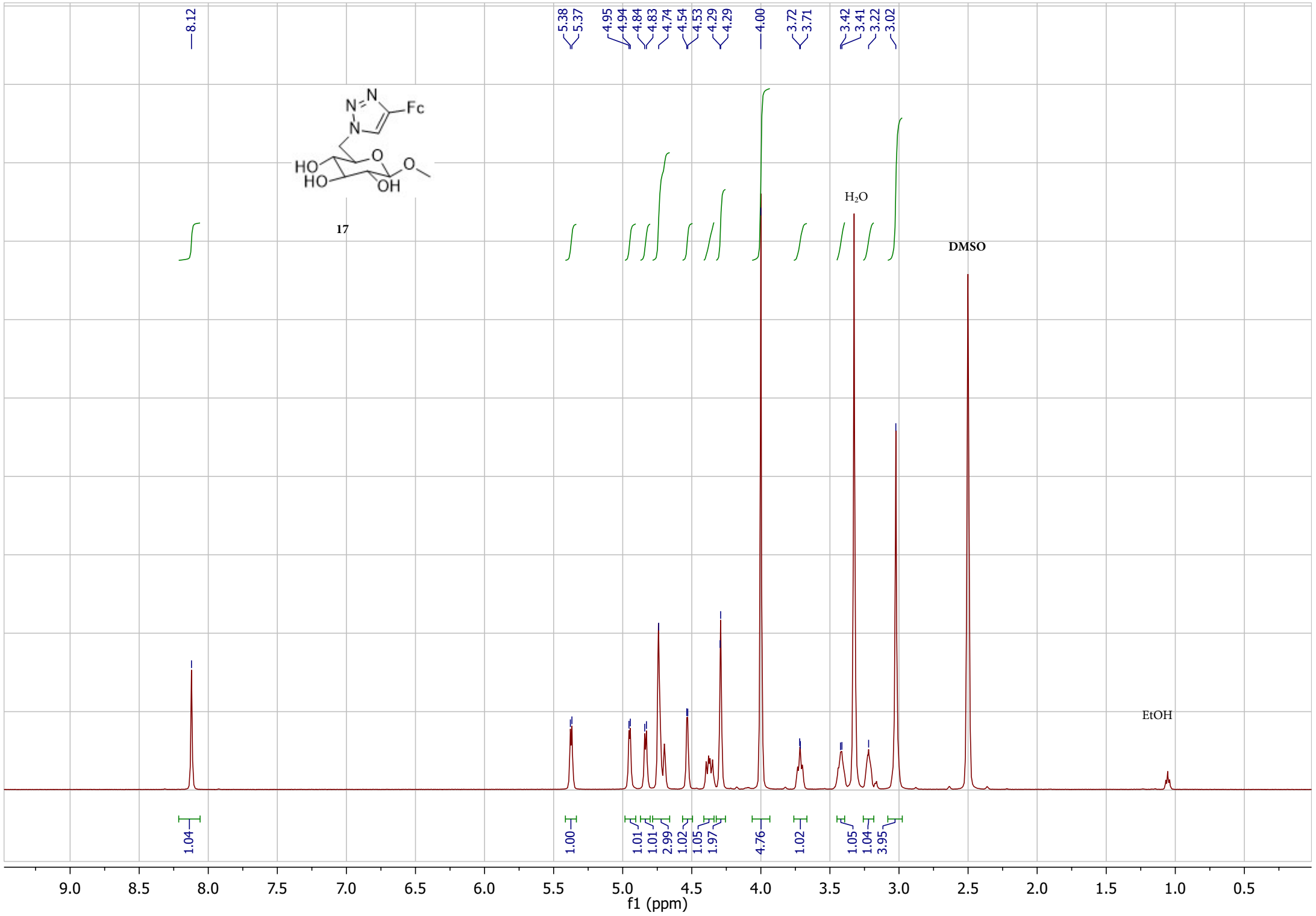
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5.37
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4.94
4.84
4.83
4.74
4.54
4.53
4.29
4.29
4.00
3.72
3.71
3.42
3.41
3.22
3.02
Fc
HO
HO
OH
O
O
17
H2O
DMSO
EtOH
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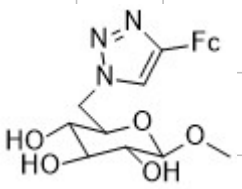
1.00
1.01
1.01
2.99
1.02
1.05
1.97
4.76
1.02
1.05
1.04
3.95
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

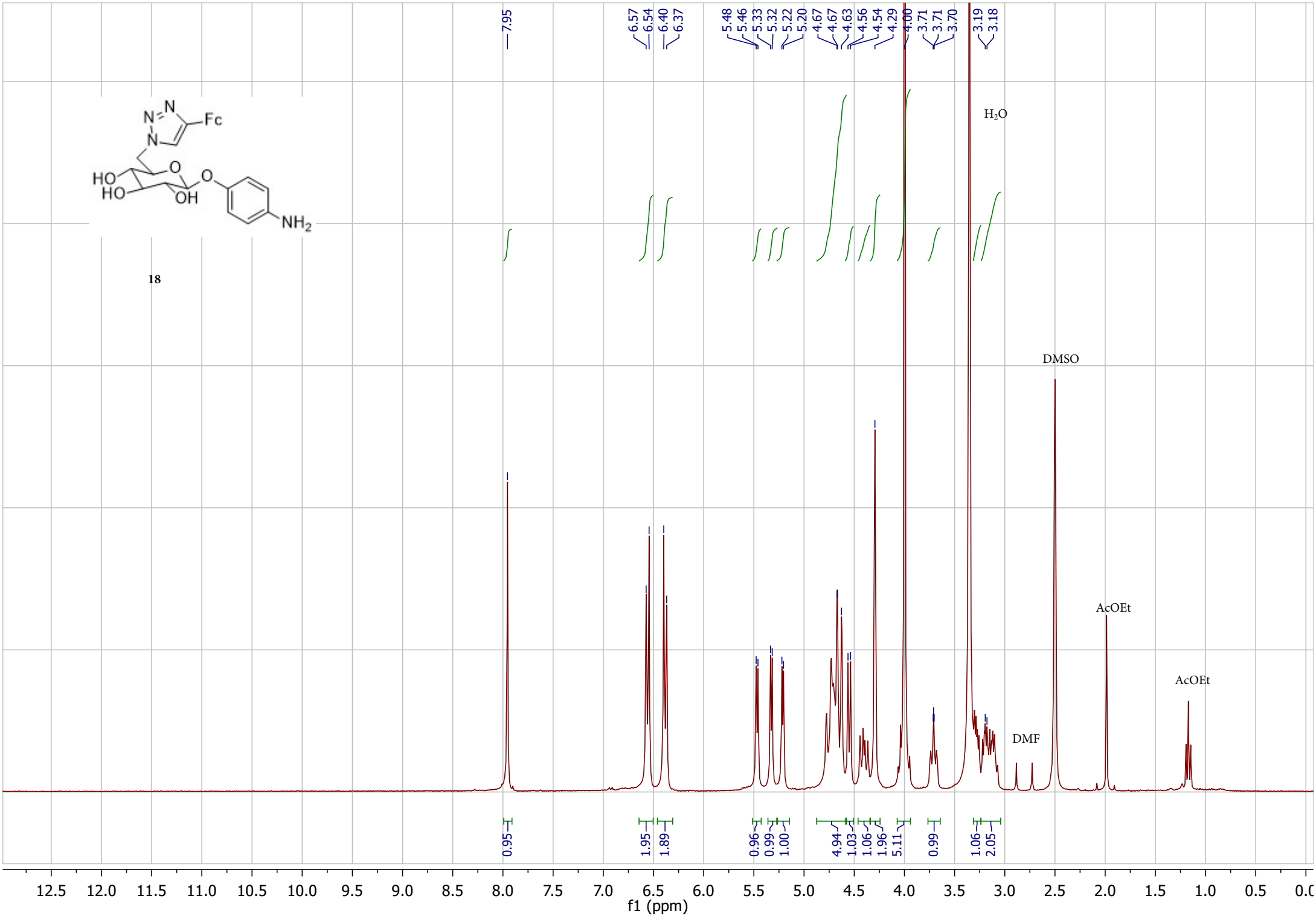
17



145.1
121.3
99.7
76.1
73.1
71.8
71.7
70.6
69.2
68.3
66.2
54.1
51.0

240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

f1 (ppm)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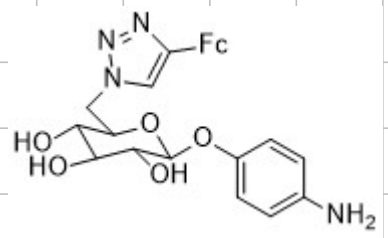
5.22
5.20
4.67
4.67
4.63
4.56
4.54
4.29
4.00
3.71
3.71
3.70
3.19
3.18
H2O
DMSO
AcOEt
AcOEt
DMF
0.95
1.95
1.89
0.96
0.99
1.00
4.94
1.03
1.06
1.96
5.11
0.99
1.06
2.05
12.5
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

18

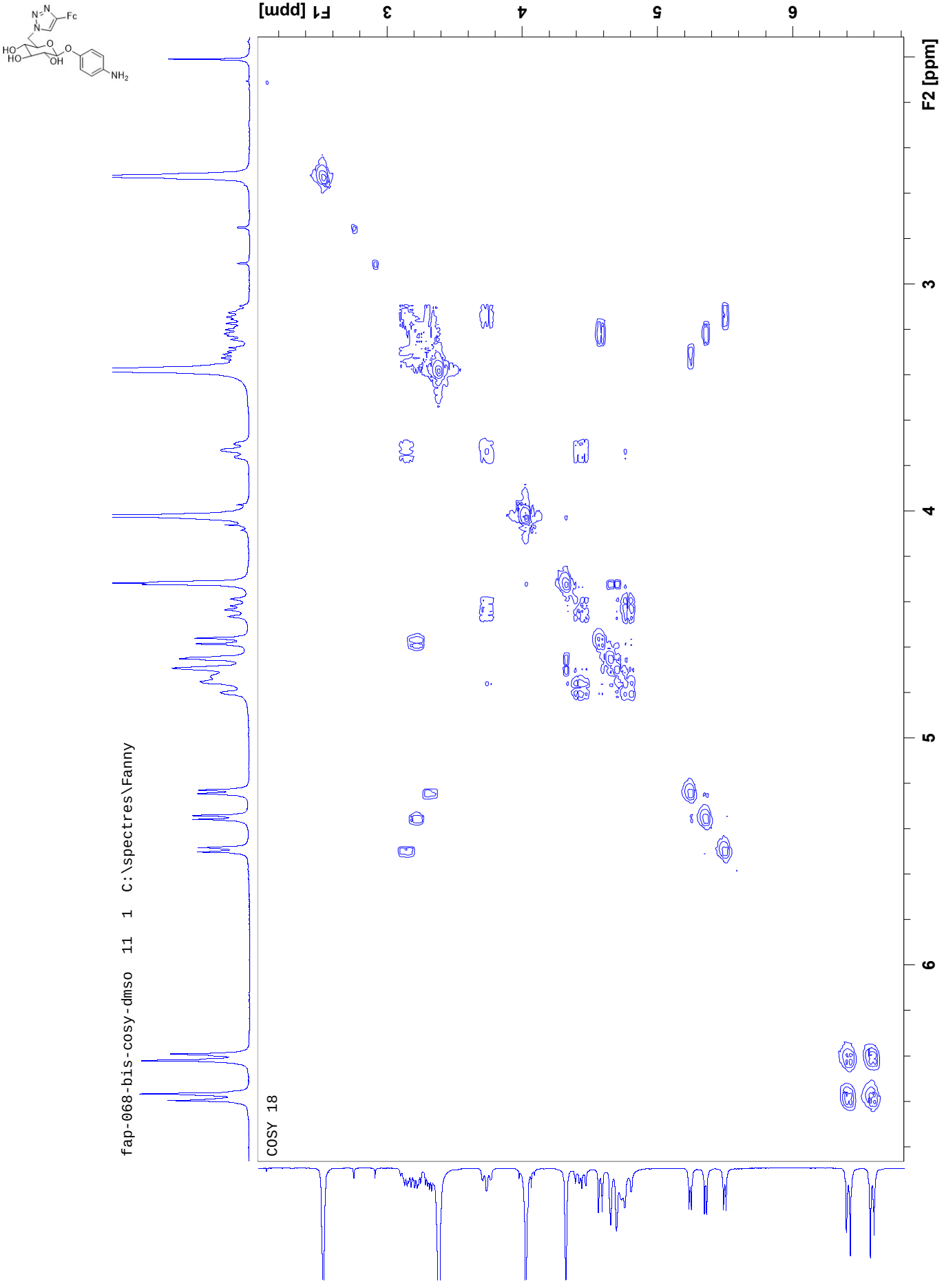
18



148.4
145.1
143.8
121.5
118.0
114.4
102.3
76.1
76.0
74.3
73.2
71.3
69.2
68.2
68.2
66.5
66.4
50.9

240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

f1 (ppm)


F1 [ppm]
3
4
5
6
F2 [ppm]
3
4
5
6
COSY 18
fap-068-bis-cosy-dmso 11 1 C:\spectres\Fanny
Fc
HO
HO
OH
O
O
NH2