Supporting Information

Discovery of imidazo[1,2-b]pyridazine-containing TAK1 kinase inhibitors with excellent activities against multiple myeloma

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Contents of Supporting Information:

Dose-response curves graphs for H929 and MPC-11 Cells…….S2

KinomeScan Profiling Table ……...S3

^{1}H-NMR ^{13}C-NMR spectra for all compounds…….S6

HPLC traces for final compounds…….S53

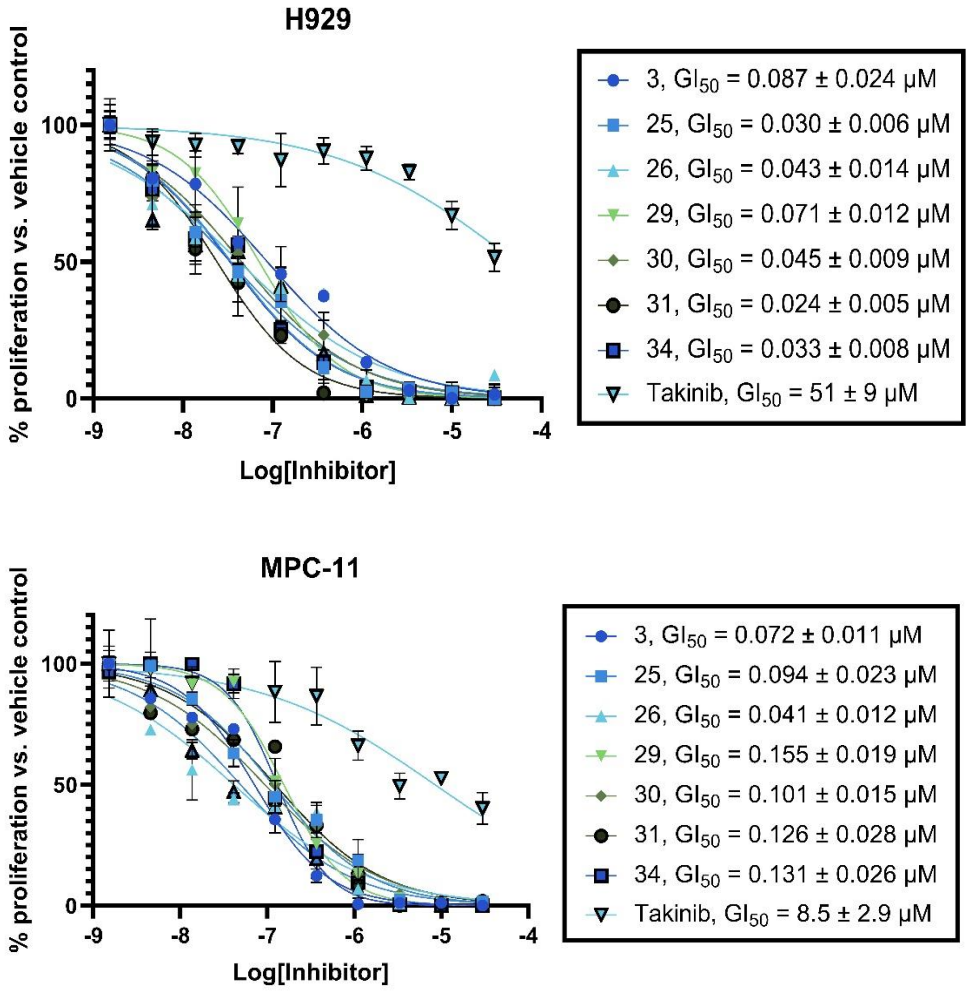


Figure S1. Dose-response curves MM cells were treated with varying concentrations of compound for 72 h. Data was fitted to a non-linear regression equation using GraphPad Prism 9.0 software. Each data point represents the mean and error bars represent the SEM of triplicates.

The KinomeScan profiling (Eurofins) for select kinases for Compound 26 is shown in Table S1. The KinomeScan technology utilizes active site-directed competition binding assay to quantitatively measure interactions between test compounds and kinases. For details, see:

• Fabian, M.A. *et al*. A small molecule-kinase interaction map for clinical kinase inhibitors. *Nat. Biotechnol.* **23**, 329-336 (2005).

• Karaman, M.W. *et al*. A quantitative analysis of kinase inhibitor selectivity. *Nat. Biotechnol*. 26, 127-132 (2008).

Table S1: KinomeScan profiling for compound **26**

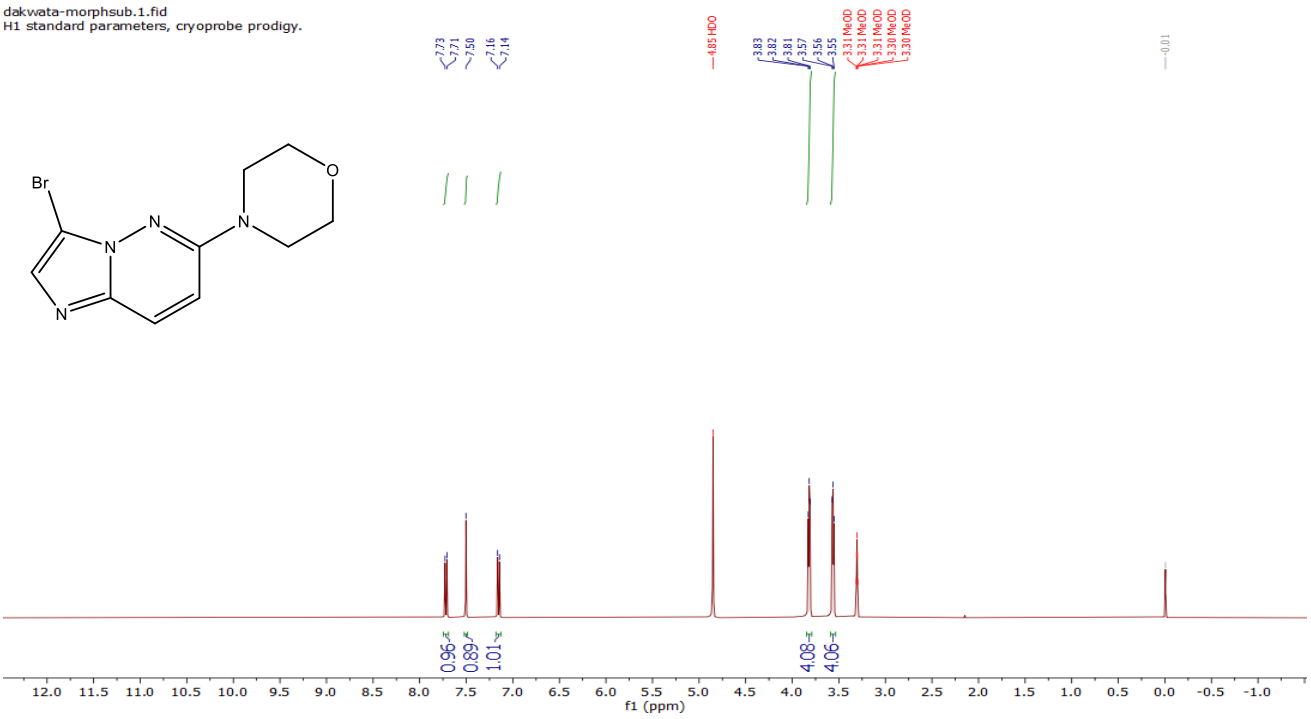
Kinase Target	% Ctrl (50 nM)	% Ctrl (500 nM)
ABL1	70	44
ACVR1B	100	100
CABC1	94	100
AKT1	98	100
AKT2	100	97
ALK	64	80
AURKA	87	85
AURKB	100	82
AXL	95	60
BMPR2	89	56
BRAF	91	100
BRAF	98	100
BTK	67	89
CDK19	90	13
CDK2	100	84
CDK3	100	90
CDK7	88	23
CDK9	79	78
CHEK1	91	100
CSF1R	100	100
DCLK1	84	100
EGFR	60	31
EPHA2	86	100
ERBB2	87	68
MAPK3	100	100
PTK2	98	100
FGFR2	100	100
FGFR3	100	100
FLT3	31	3

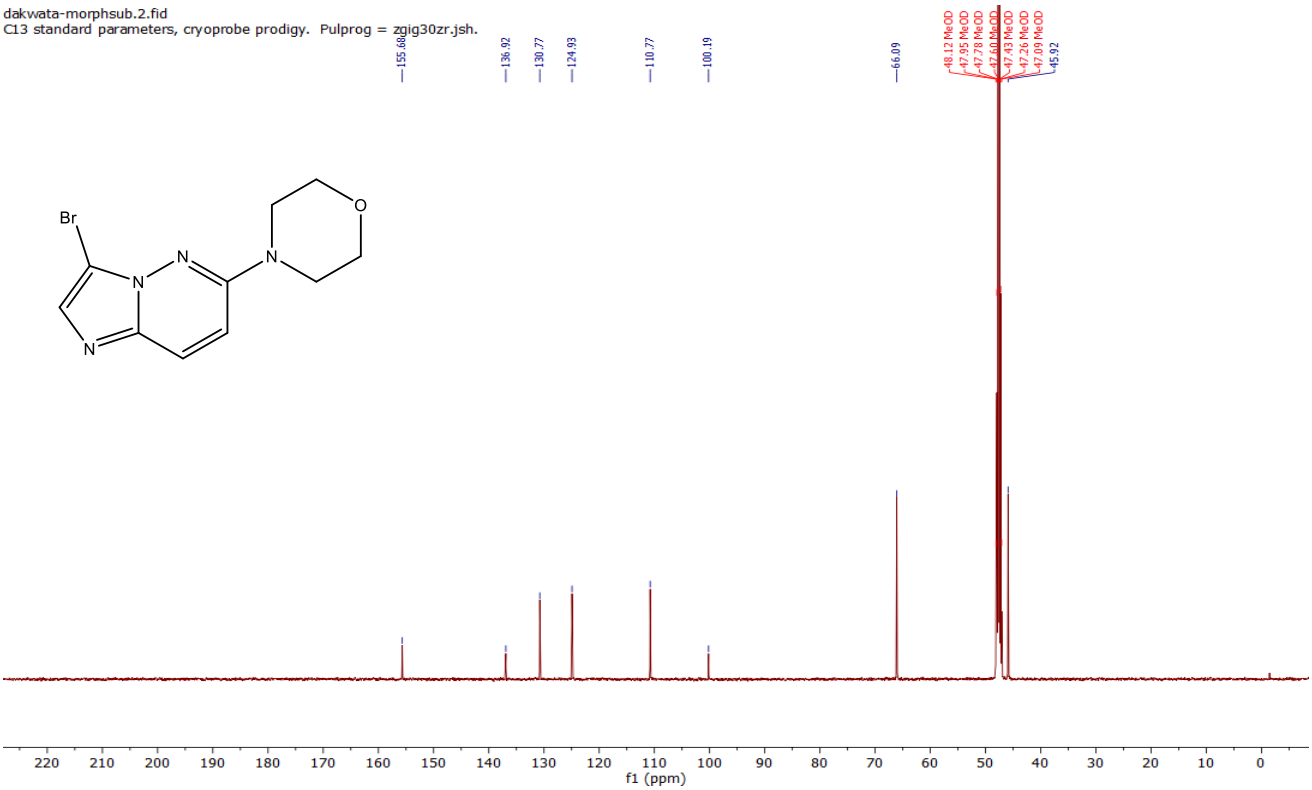
GSK3B	76	74
IGF1R	99	100
CHUK	86	85
IKBKB	88	89
INSR	100	81
JAK2	100	87
JAK3	76	11
MAPK8	80	54
MAPK9	80	50
MAPK10	85	52
KIT	96	50
STK11	72	94
MAP3K4	100	100
MAPKAPK2	62	89
MARK3	100	100
MAP2K1	80	100
MAP2K2	72	100
MET	89	80
MKNK1	92	94
MKNK2	75	45
MAP3K9	96	100
MAPK14	100	100
MAPK11	100	100
PAK1	100	98
PAK2	76	55
PAK4	90	83
CDK16	98	100
PDGFRA	81	76
PDGFRB	87	12
PDPK1	100	100
PIM1	66	19
PIM2	92	3.5
PIM3	81	33
PKAC alpha	88	100
PLK1	97	96
PLK3	78	98
PLK4	88	90
PRKCE	86	64
RAF1	100	100
RET	88	61
RIOK2	76	23

ROCK2	69	21
RPS6KA3	97	30
NUAK2	89	58
SRPK3	97	85
TGFBR1	100	100
TEK	99	100
TSSK1B	78	100
TYK2	88	100
ULK2	92	91
KDR	86	100
STK32C	85	100
ZAP70	100	100

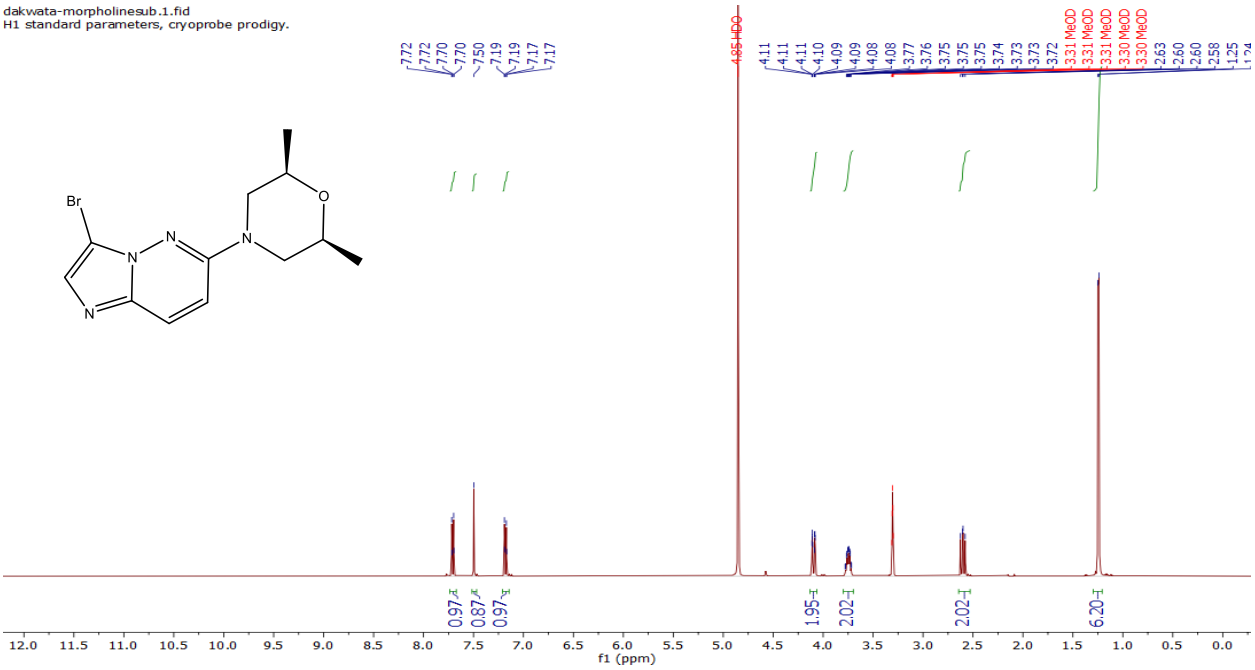
^{1}H-NMR ^{13}C-NMR spectra for all compounds

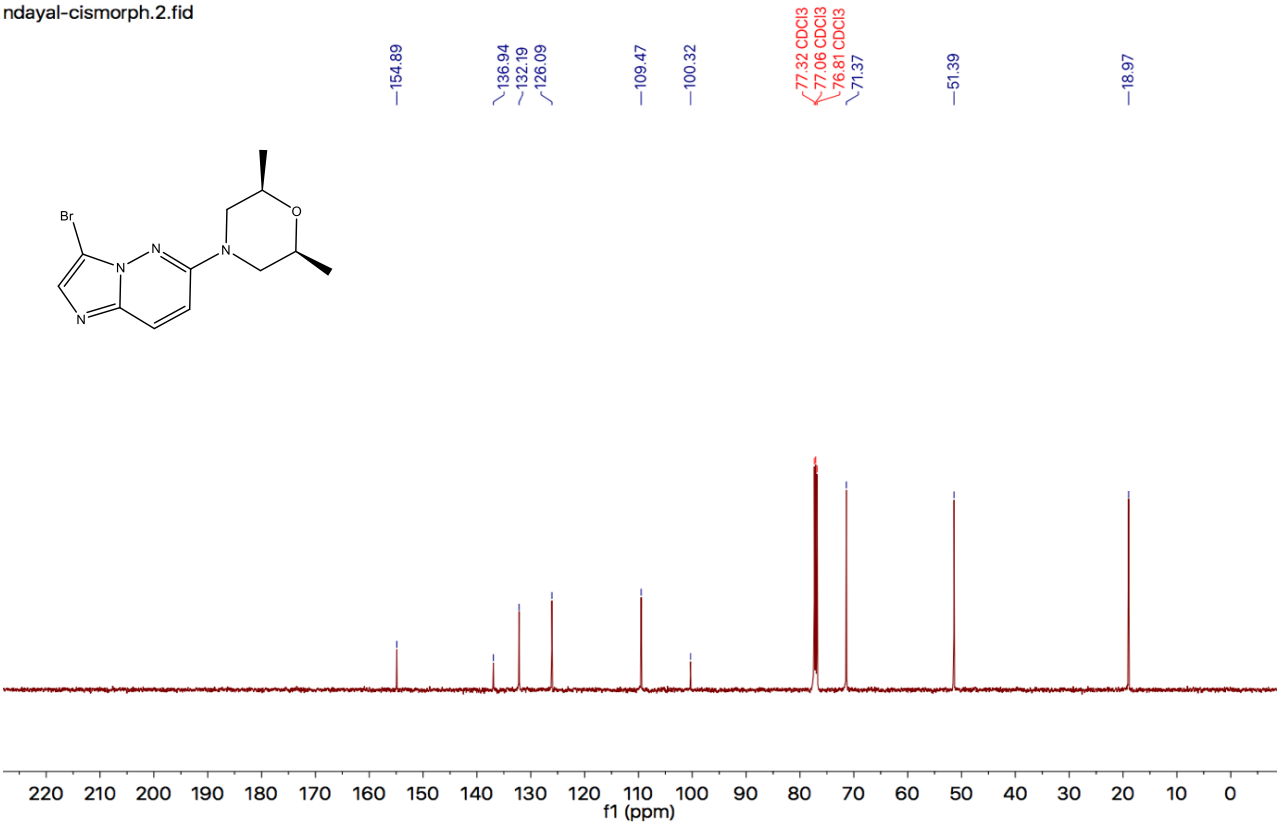
Compound **S1**





Compound **S2**


dakwata-morpholinesub.1.fid
H1 standard parameters, cryoprobe prodigy.
Br
N
N
N
O
N
7.72
7.72
7.70
7.70
7.50
7.19
7.19
7.17
7.17
4.85 HDO
4.11
4.11
4.11
4.10
4.09
4.09
4.08
4.08
3.77
3.76
3.75
3.75
3.75
3.74
3.73
3.73
3.72
3.31 MeOD
3.31 MeOD
3.31 MeOD
3.30 MeOD
3.30 MeOD
2.63
2.60
2.60
2.58
1.25
0.97
0.87
0.97
1.95
2.02
2.02
6.20
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)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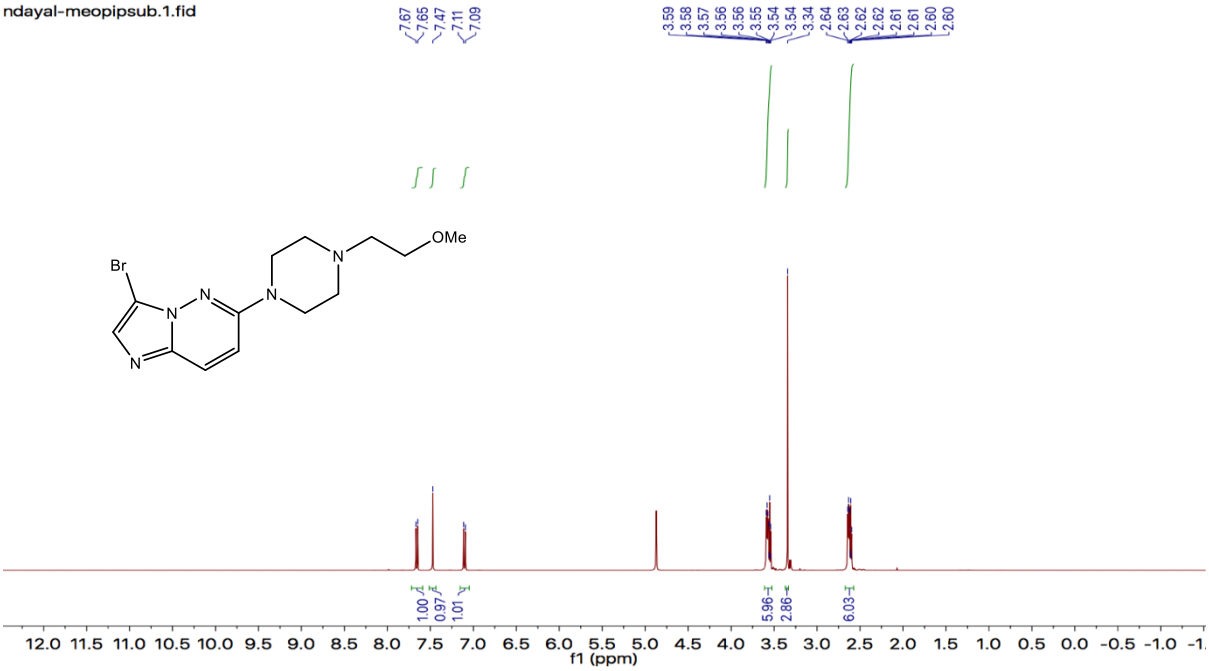
154.89
136.94
132.19
126.09
109.47
100.32
77.32 CDCl3
77.06 CDCl3
76.81 CDCl3
71.37
51.39
18.97
Br
N
N
N
O
N
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

Compound **S3**

ndayal-meopipsub.1.fid



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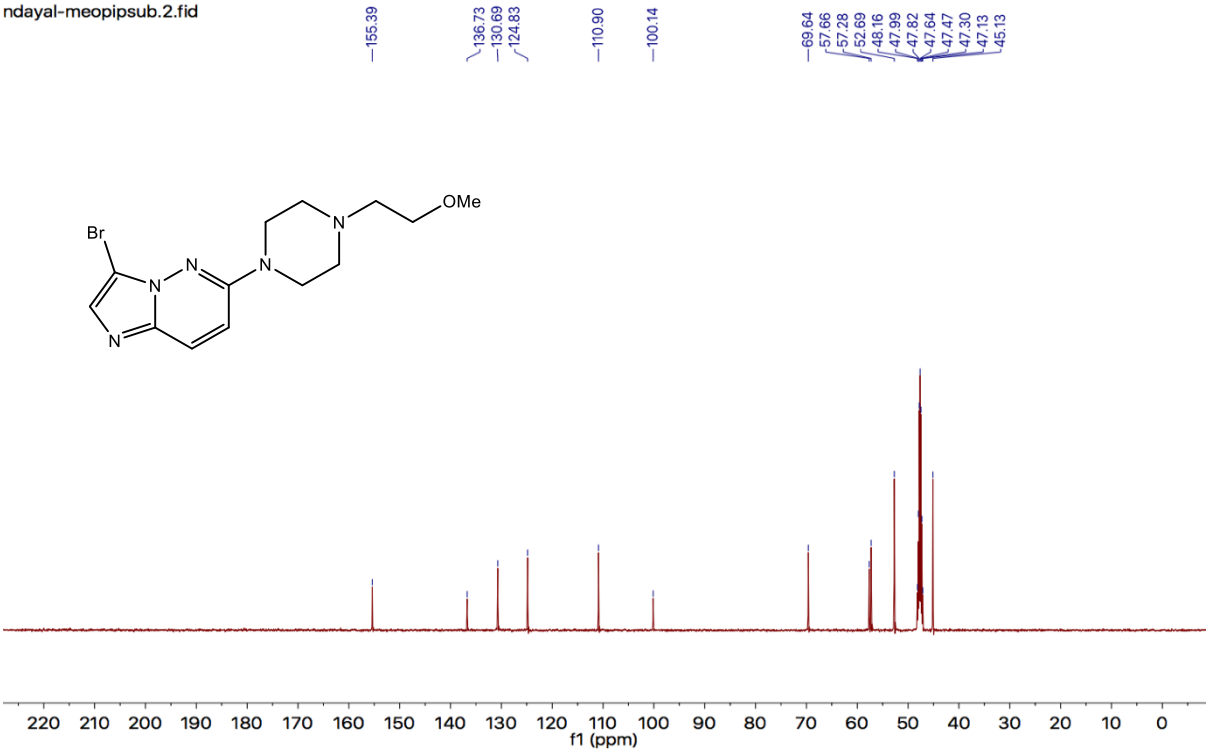
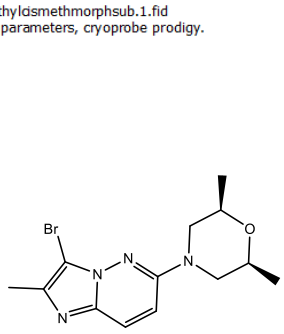


Compound **S4**

dakwata-methyldsmethmorphsub.1.fid
H1 standard parameters, cryoprobe prodigy.



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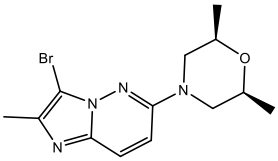

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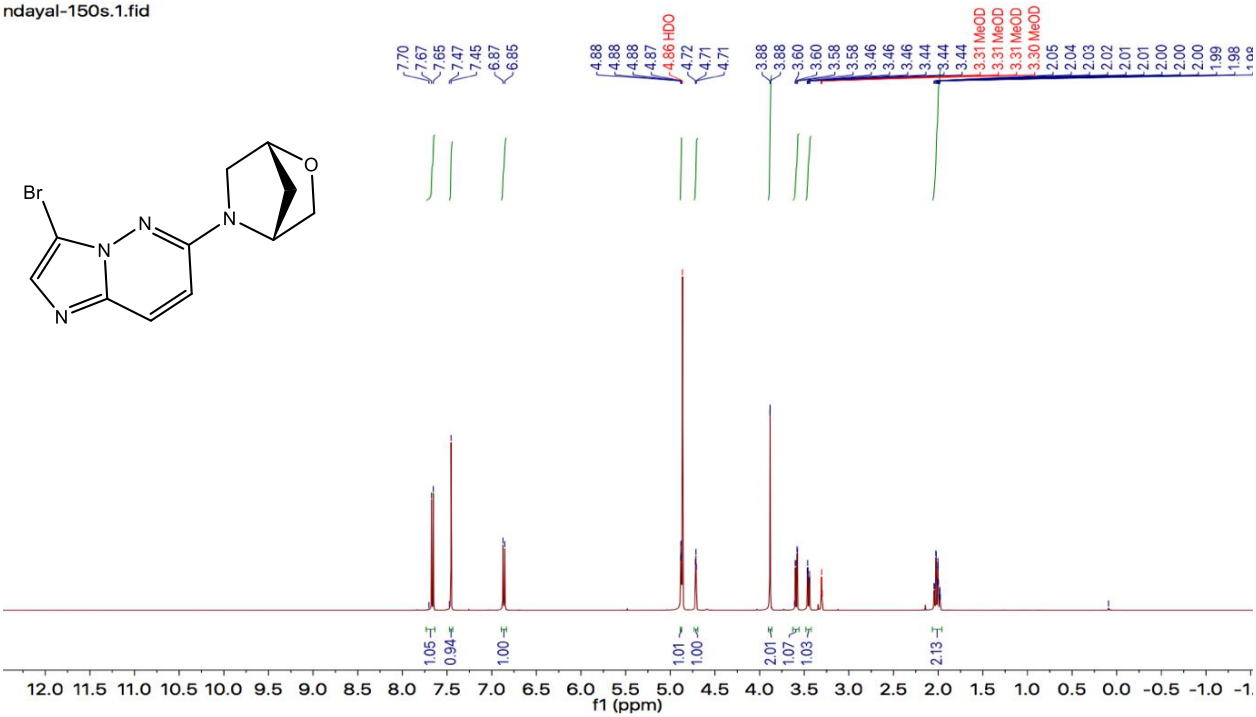

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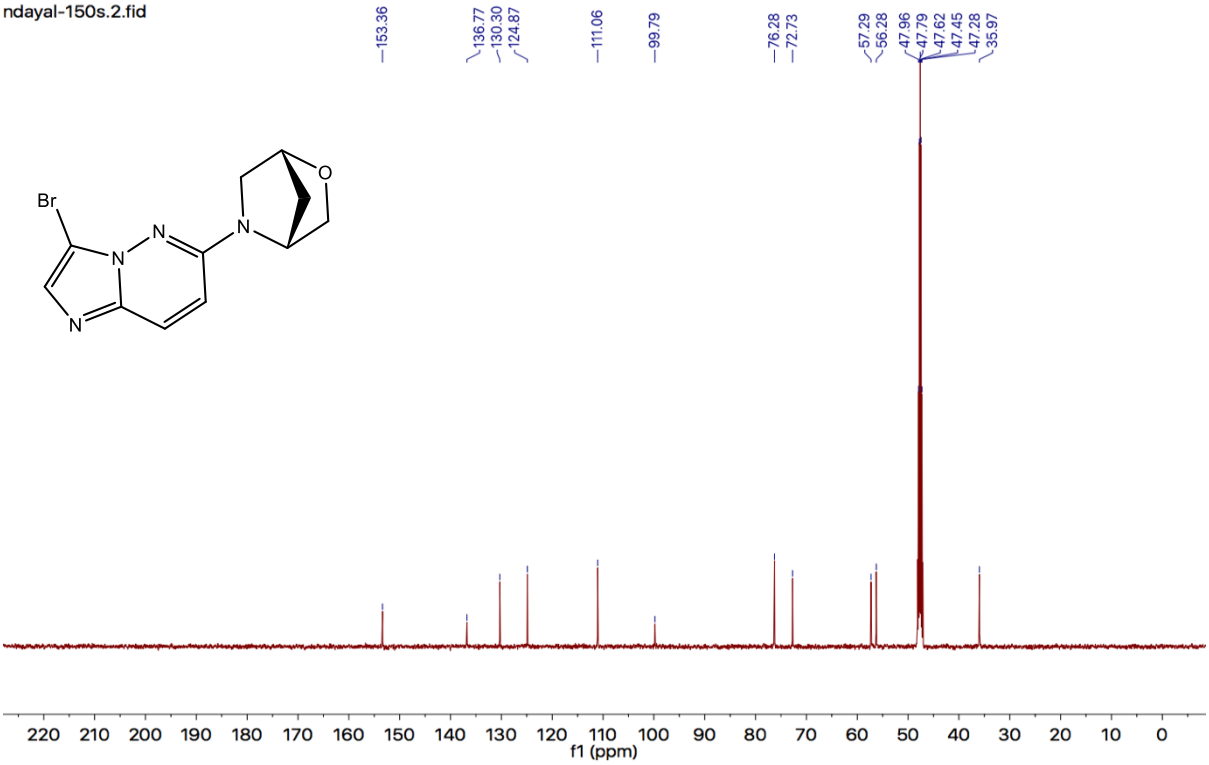

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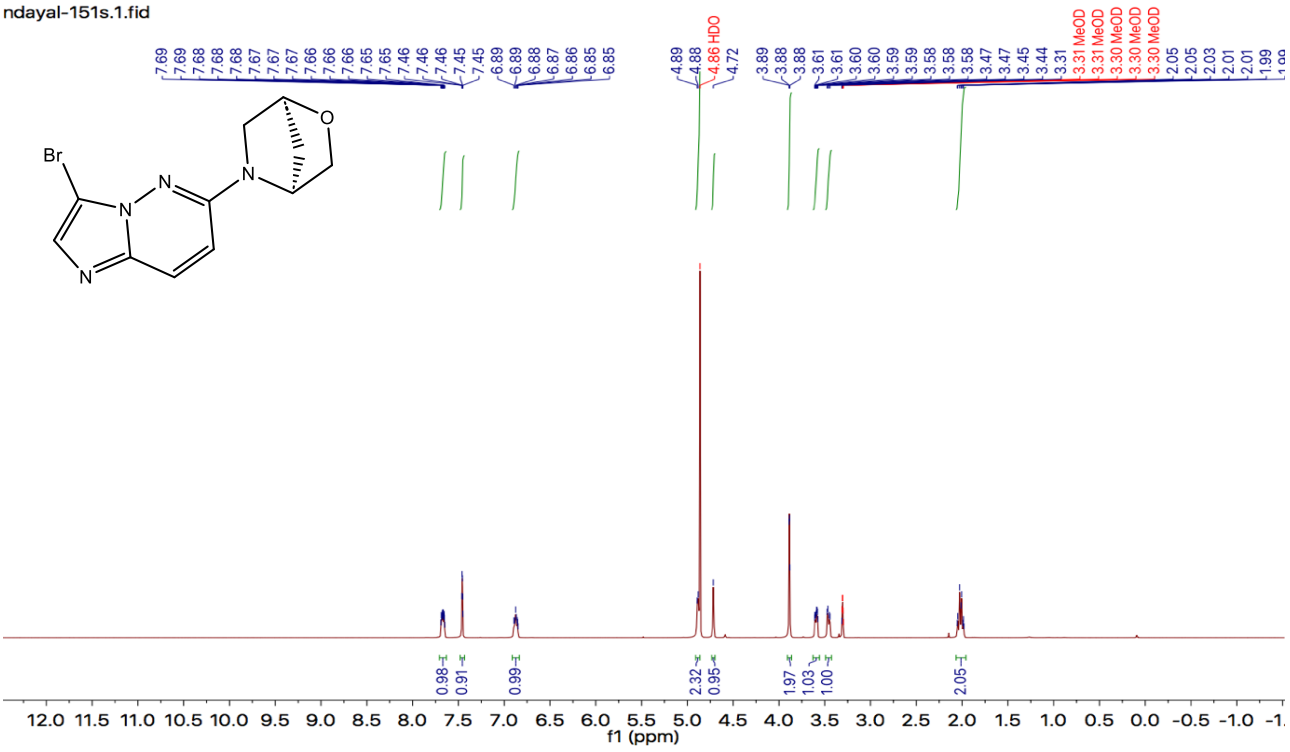
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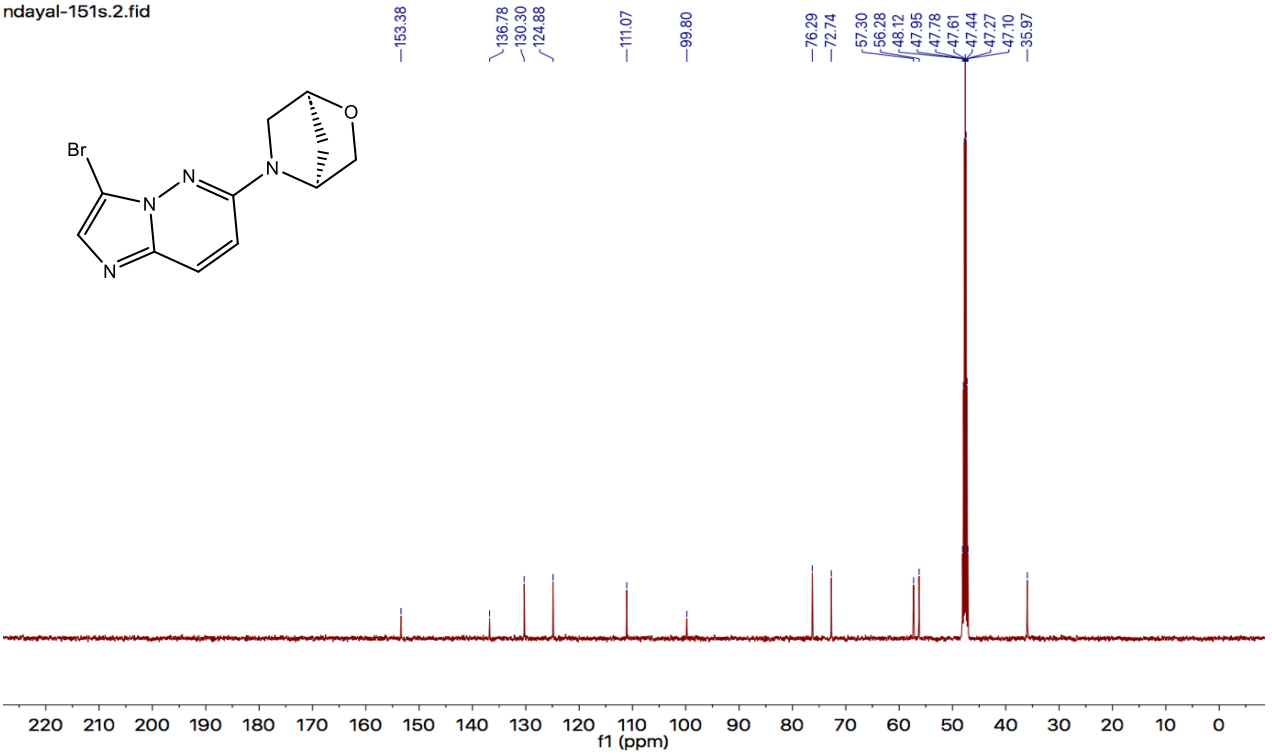
Br
N
N
N
O
f1 (ppm)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Br
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N
N
O
f1 (ppm)

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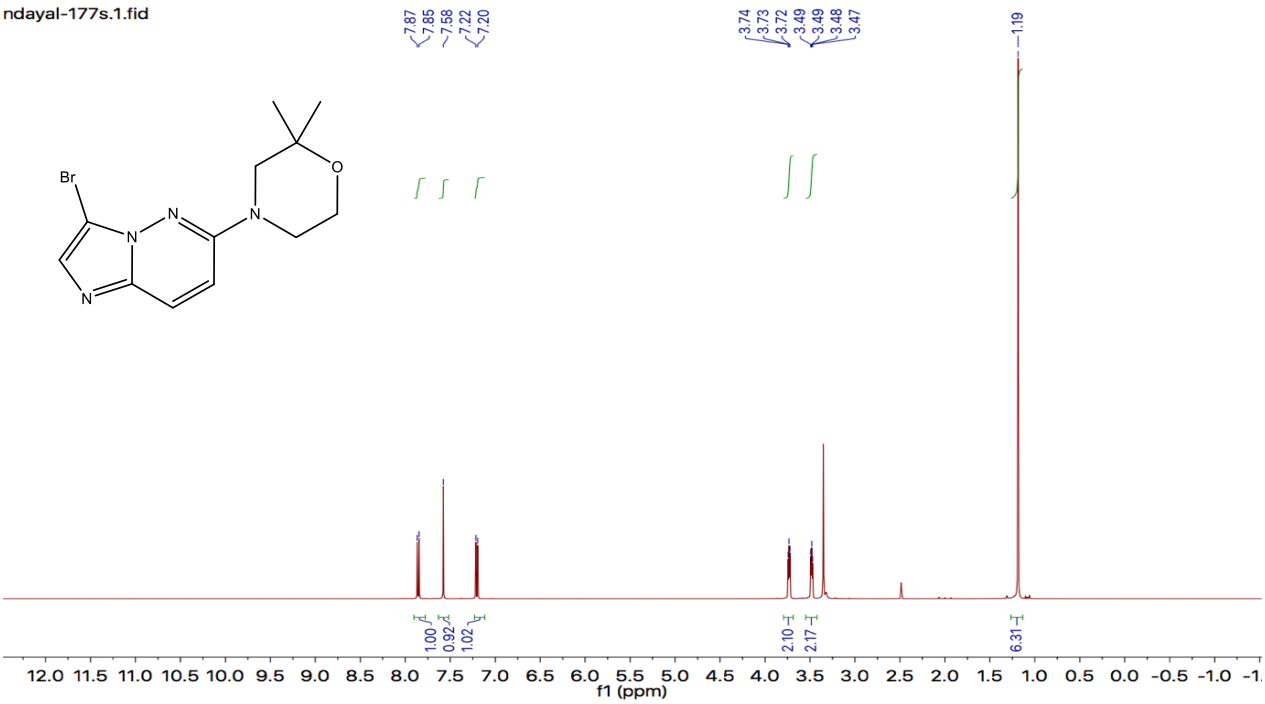
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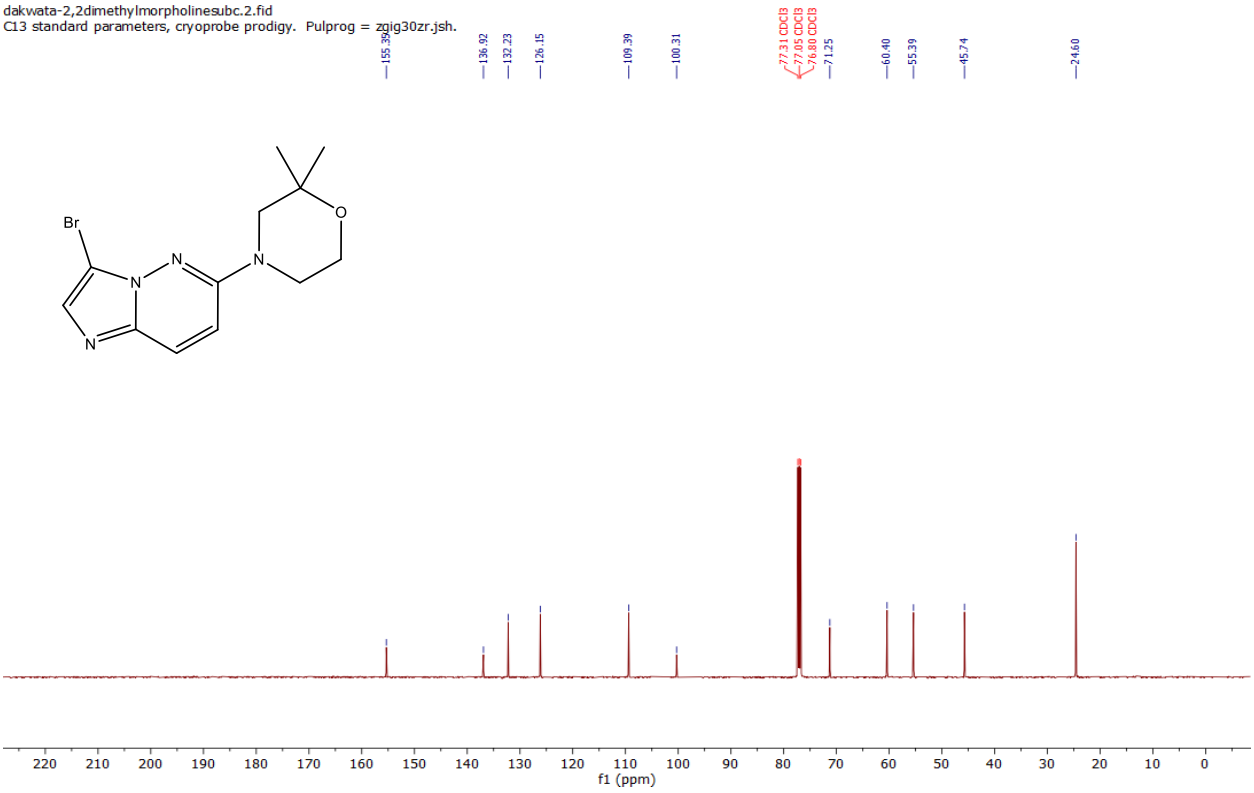
7.87
7.85
7.58
7.22
7.20
3.74
3.73
3.72
3.49
3.49
3.48
3.47
1.19
Br
N
N
N
O
1.00
0.92
1.02
2.10
2.17
6.31
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.
f1 (ppm)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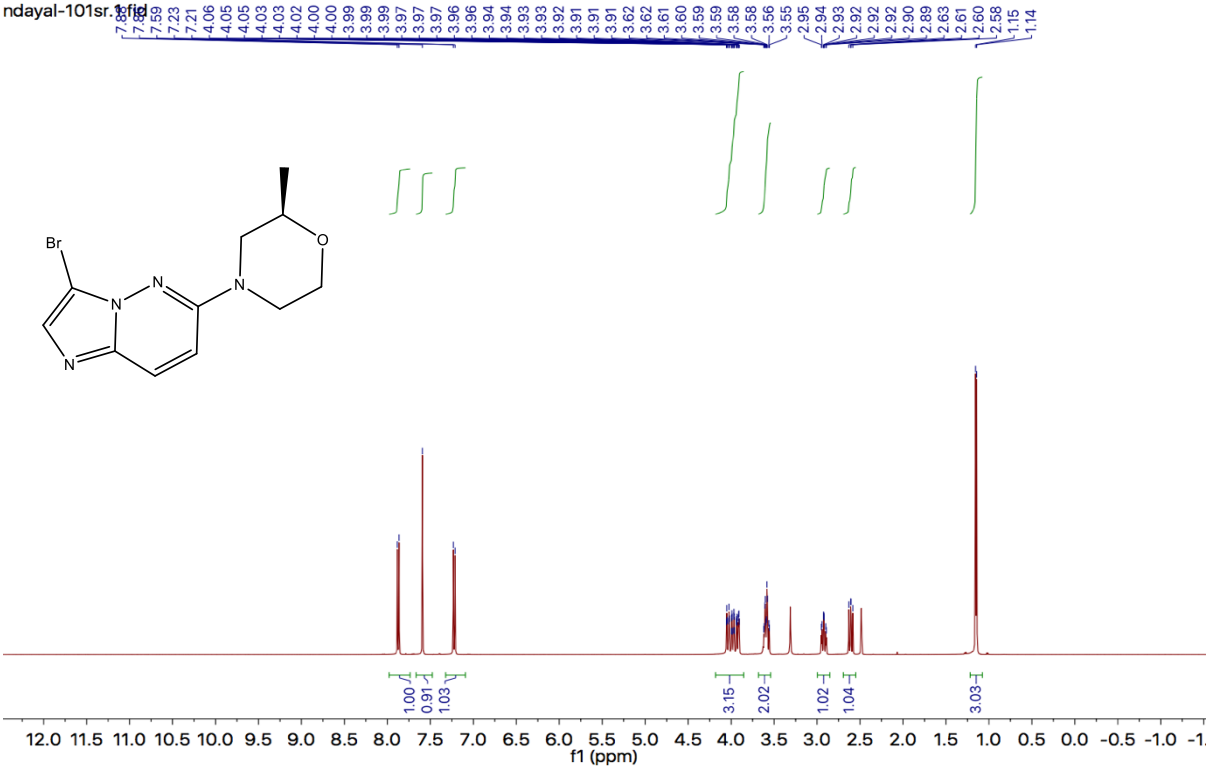
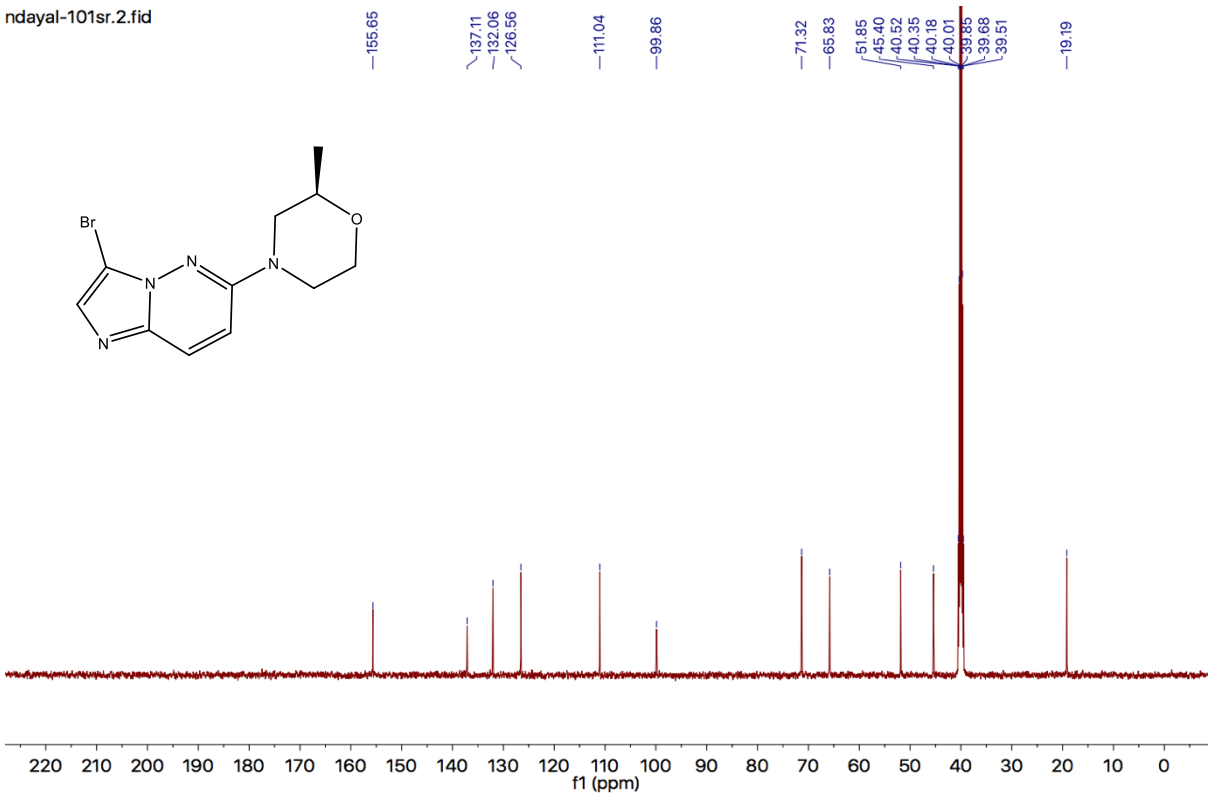
155.39
136.92
132.23
126.15
109.39
100.31
77.31 CDCl3
77.05 CDCl3
76.80 CDCl3
71.25
60.40
55.39
45.74
24.60
Br
N
N
N
O
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

Compound **S8**


ndayal-101sr.1.fid
Br
N
N
N
O
1.00
0.91
1.03
3.15
2.02
1.02
1.04
3.03
f1 (ppm)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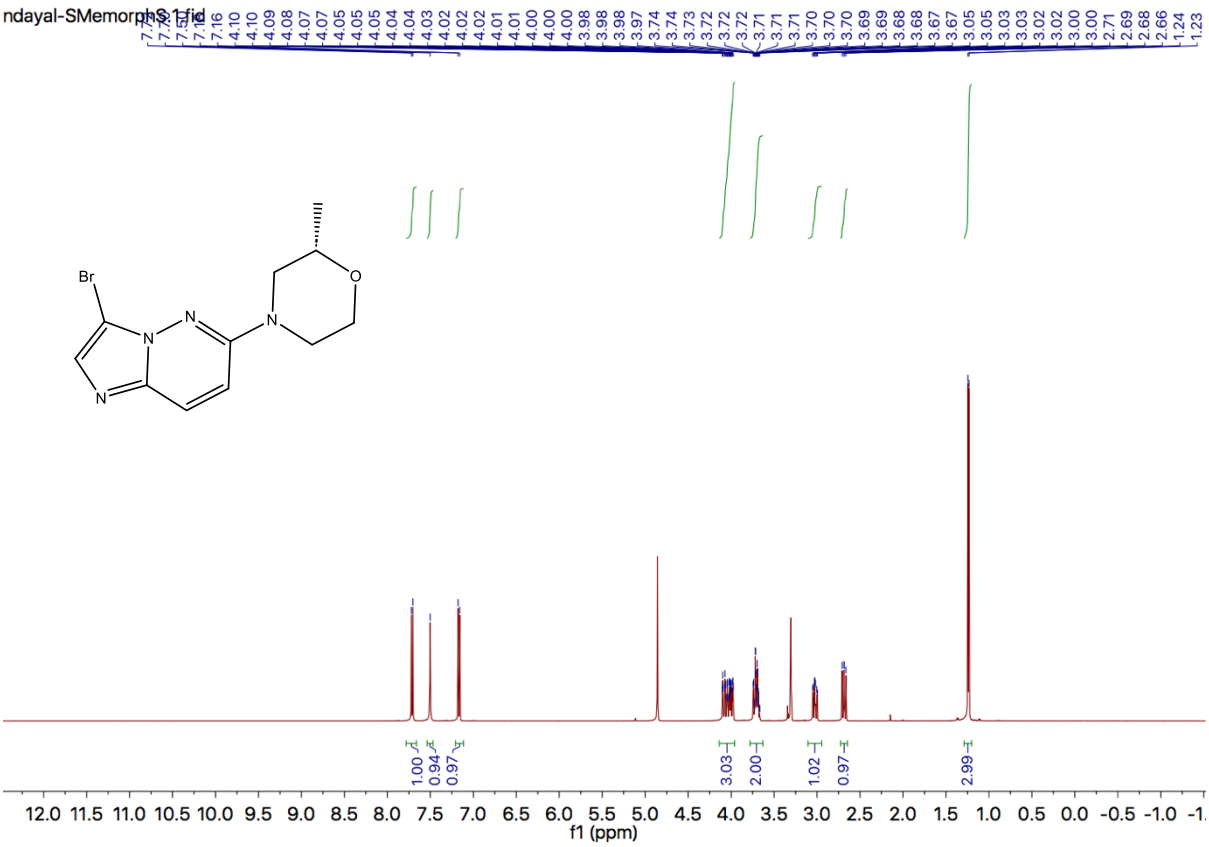
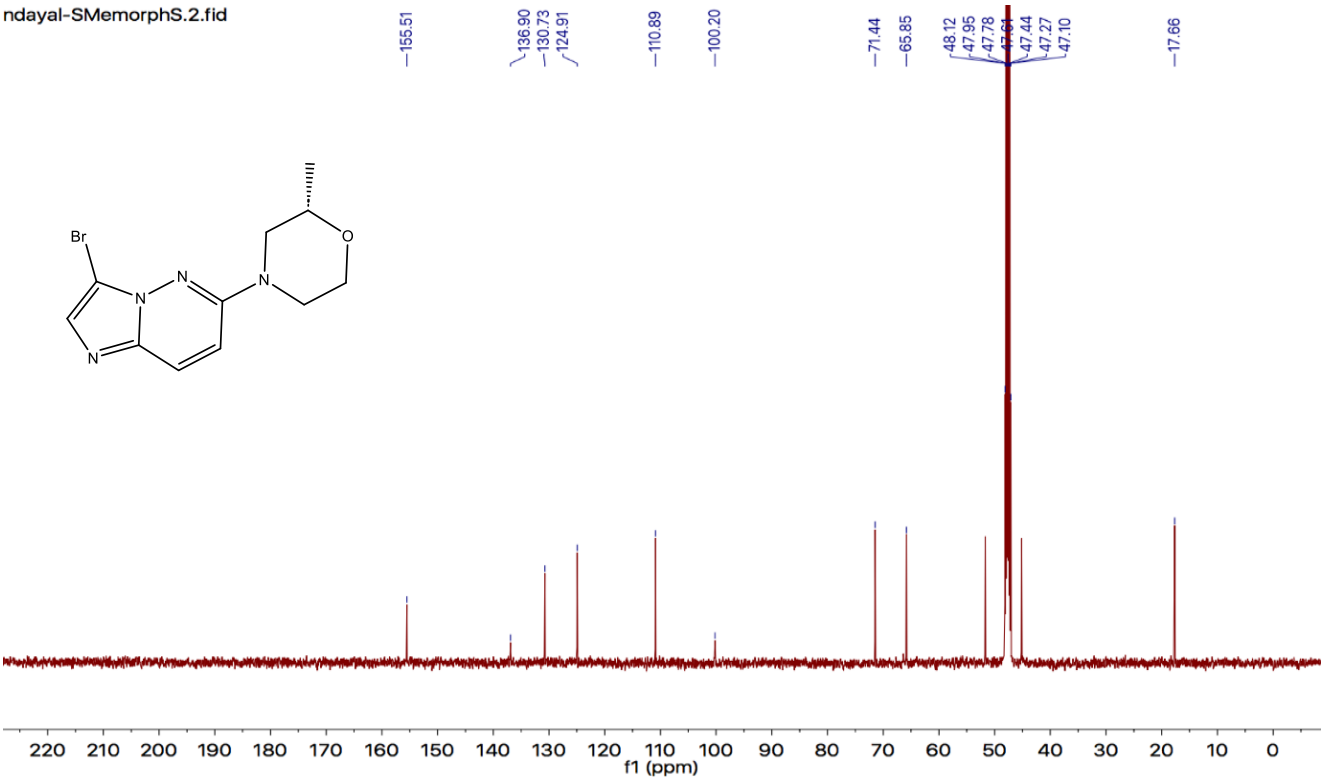
155.65
137.11
132.06
126.56
111.04
99.86
71.32
65.83
51.85
45.40
40.52
40.35
40.18
40.01
39.85
39.68
39.51
19.19
Br
N
N
N
N
O
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

Compound **S9**


ndayal-SMemorphS.1.fid
Br
O
N
N
N
N
1.00
0.94
0.97
3.03
2.00
1.02
0.97
2.99
f1 (ppm)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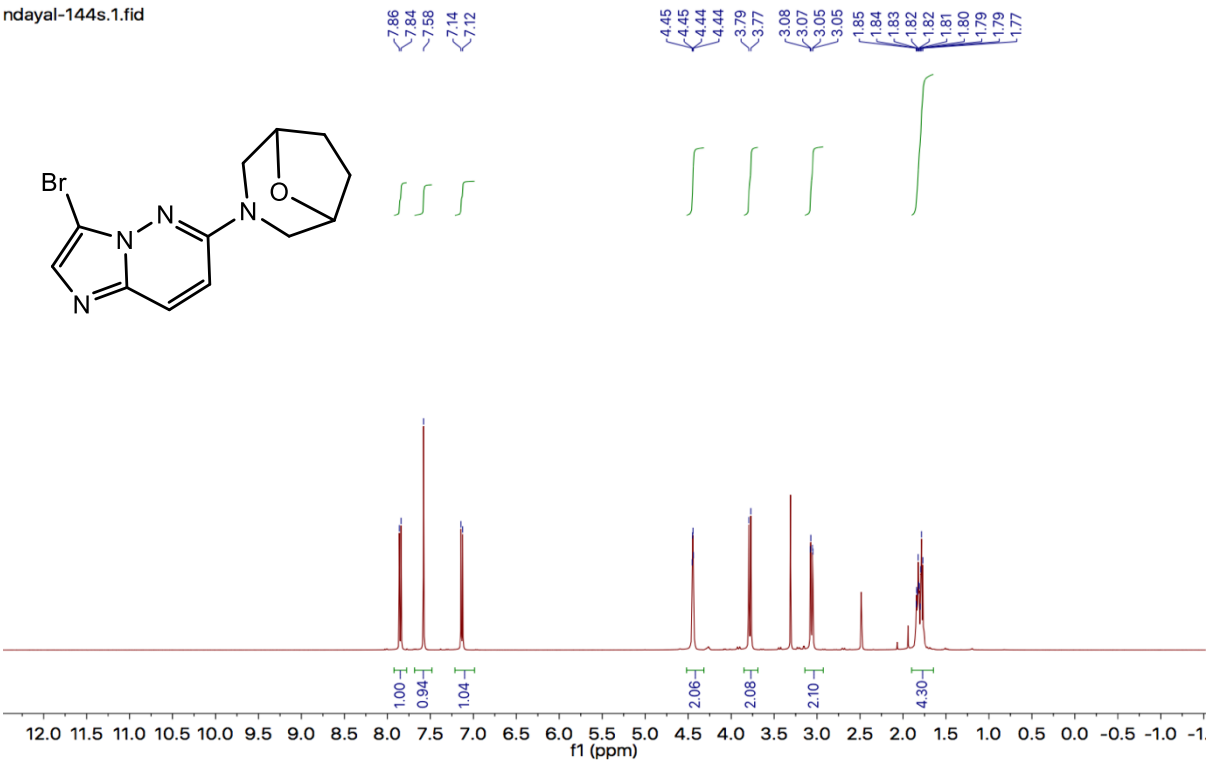
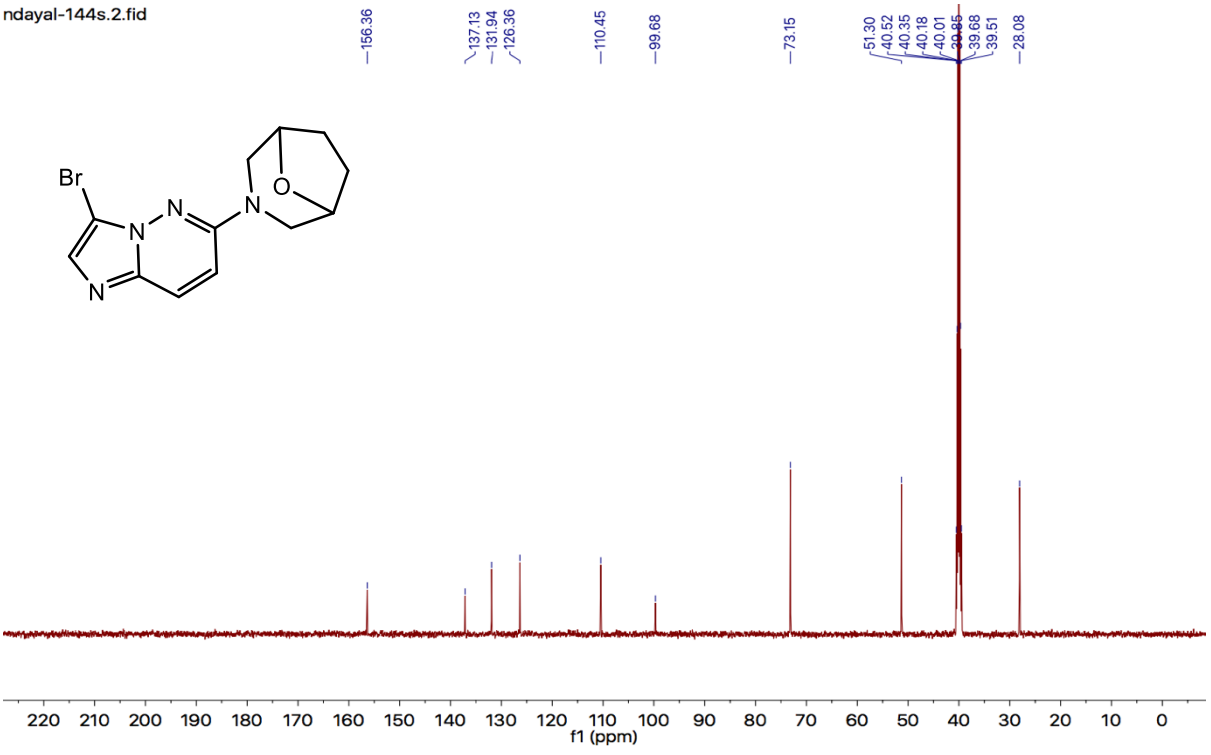
155.51
136.90
130.73
124.91
110.89
100.20
71.44
65.85
48.12
47.95
47.78
47.61
47.44
47.27
47.10
17.66
Br
O
N
N
N
N
f1 (ppm)

Compound **S10**





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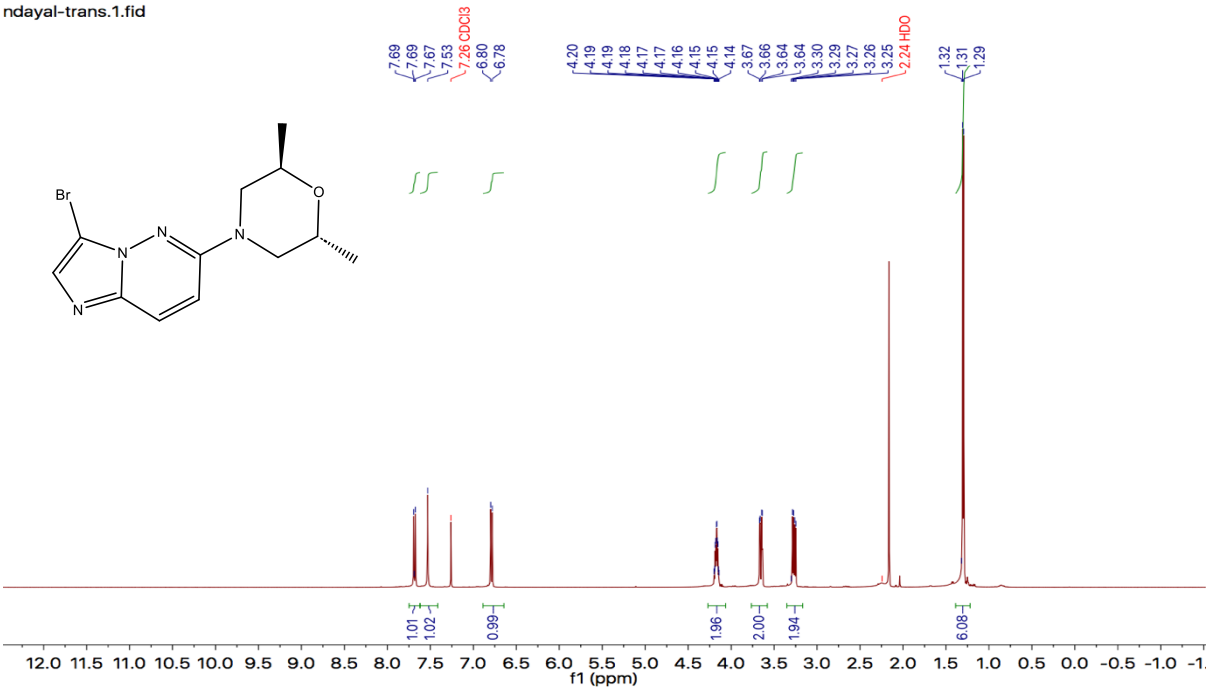

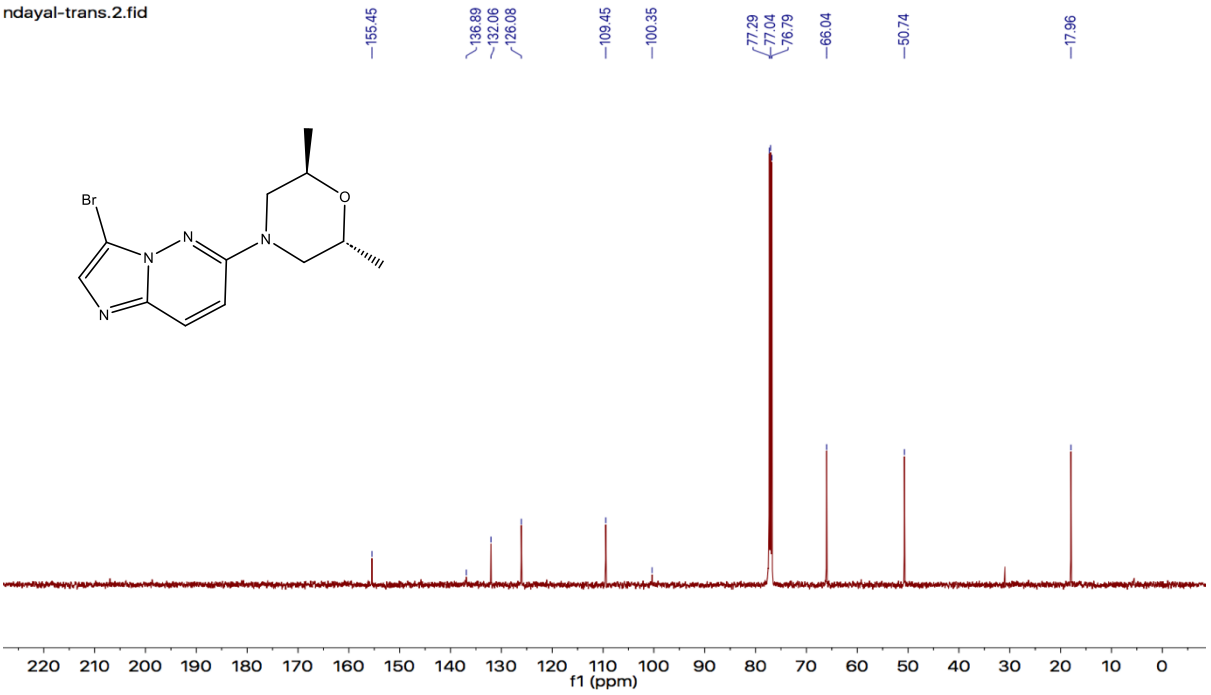

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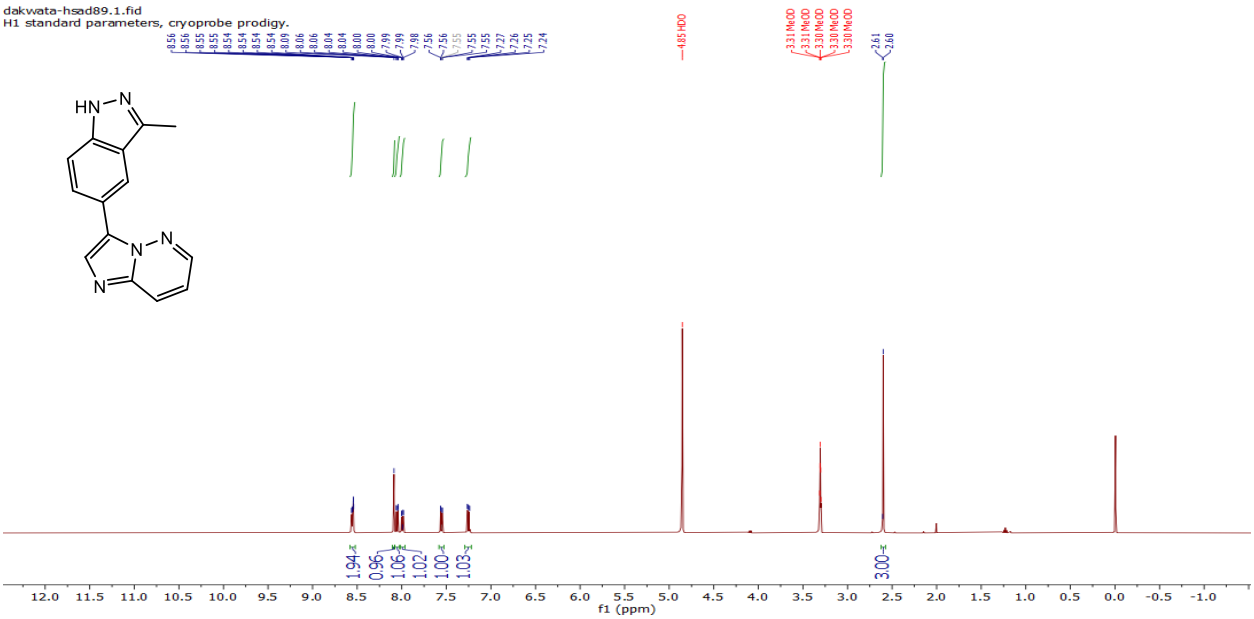
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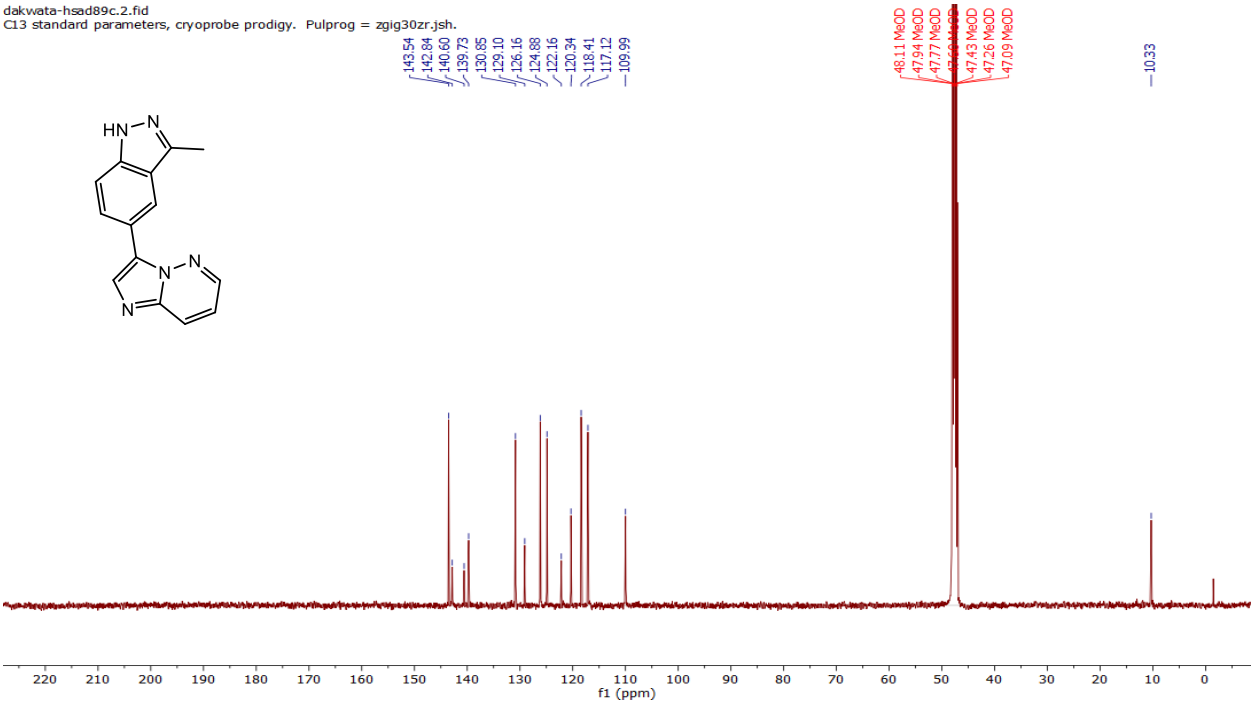
HN
N
N
N
N
4.85 HDO
3.31 MeOD
3.30 MeOD
2.61
2.60
1.94
0.96
1.06
1.02
1.00
1.03
3.00
f1 (ppm)


dakwata-hsad89c.2.fid
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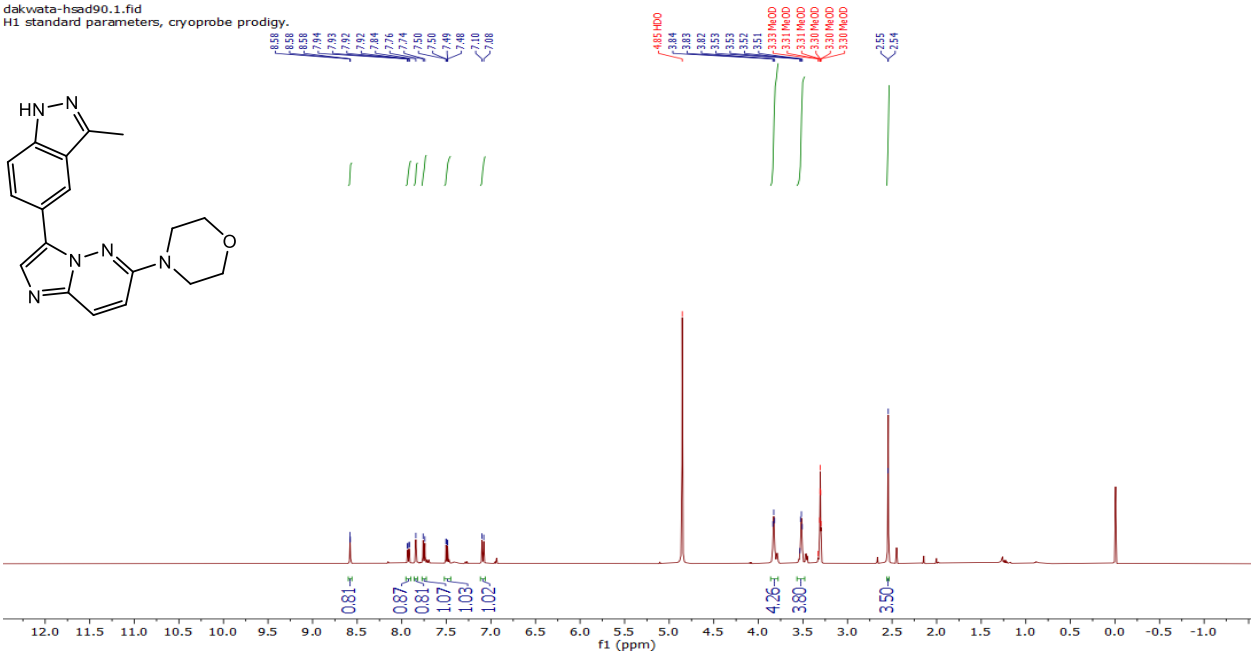
N
N
N
N
143.54
142.84
140.60
139.73
130.85
129.10
126.16
124.88
122.16
120.34
118.41
117.12
109.99
48.11 MeOD
47.94 MeOD
47.77 MeOD
47.43 MeOD
47.26 MeOD
47.09 MeOD
10.33
f1 (ppm)

Compound **2**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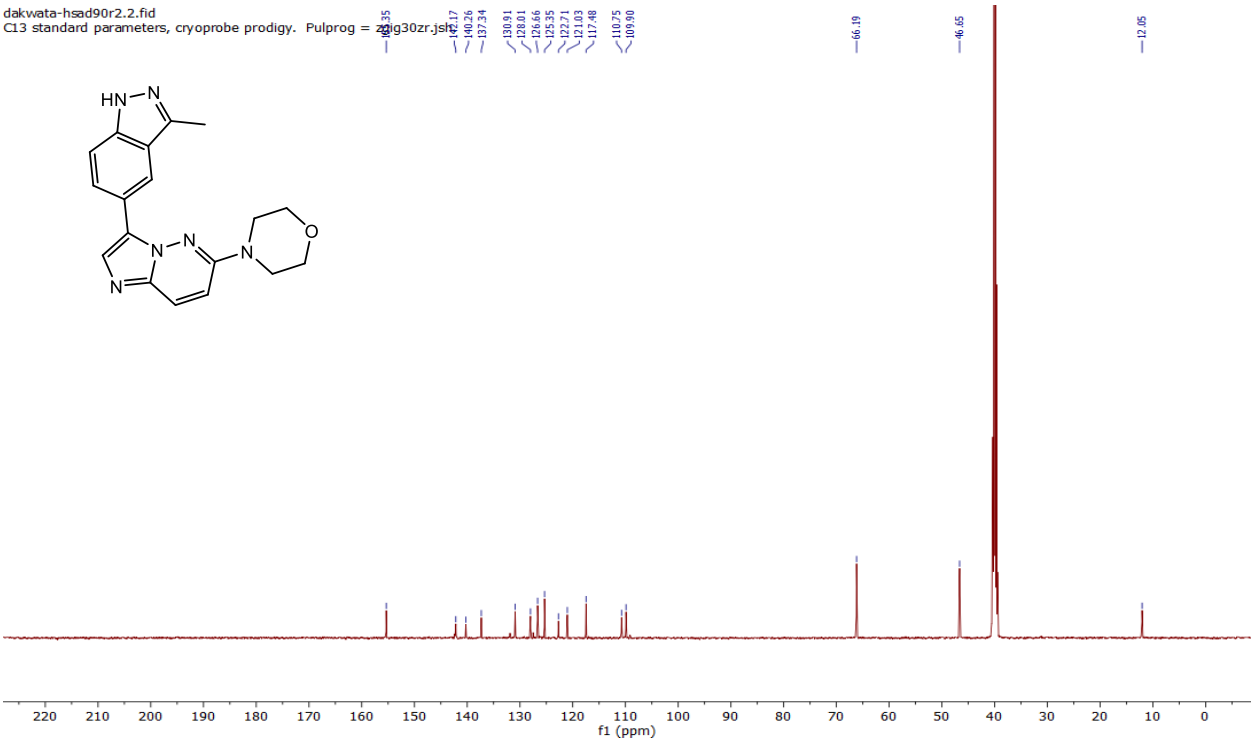
f1 (ppm)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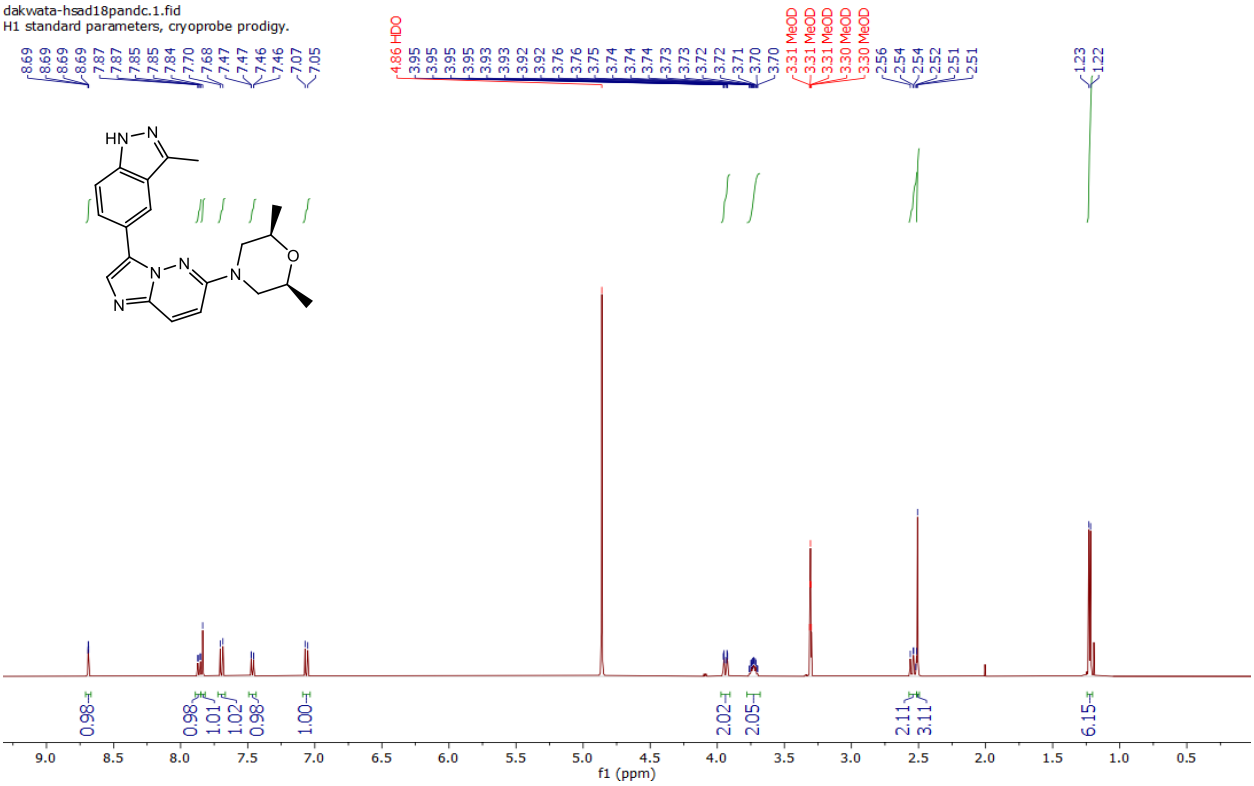
f1 (ppm)

Compound **3**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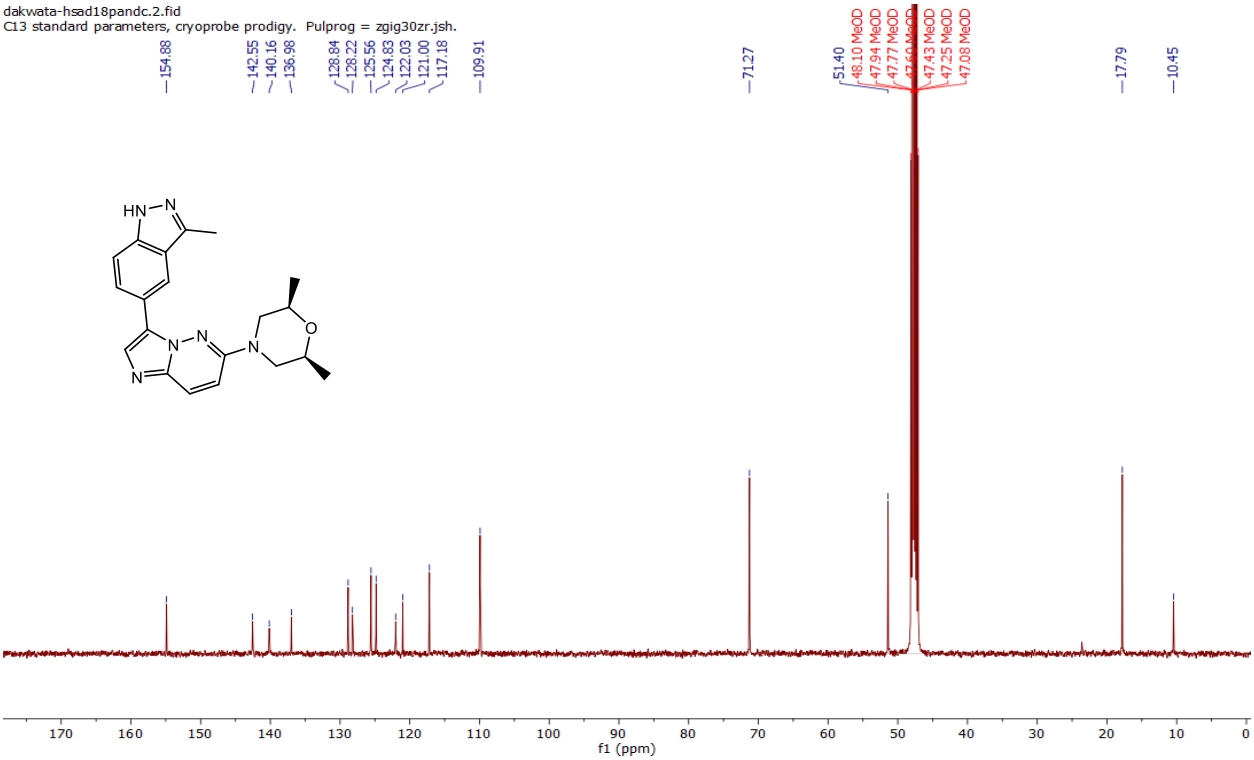
f1 (ppm)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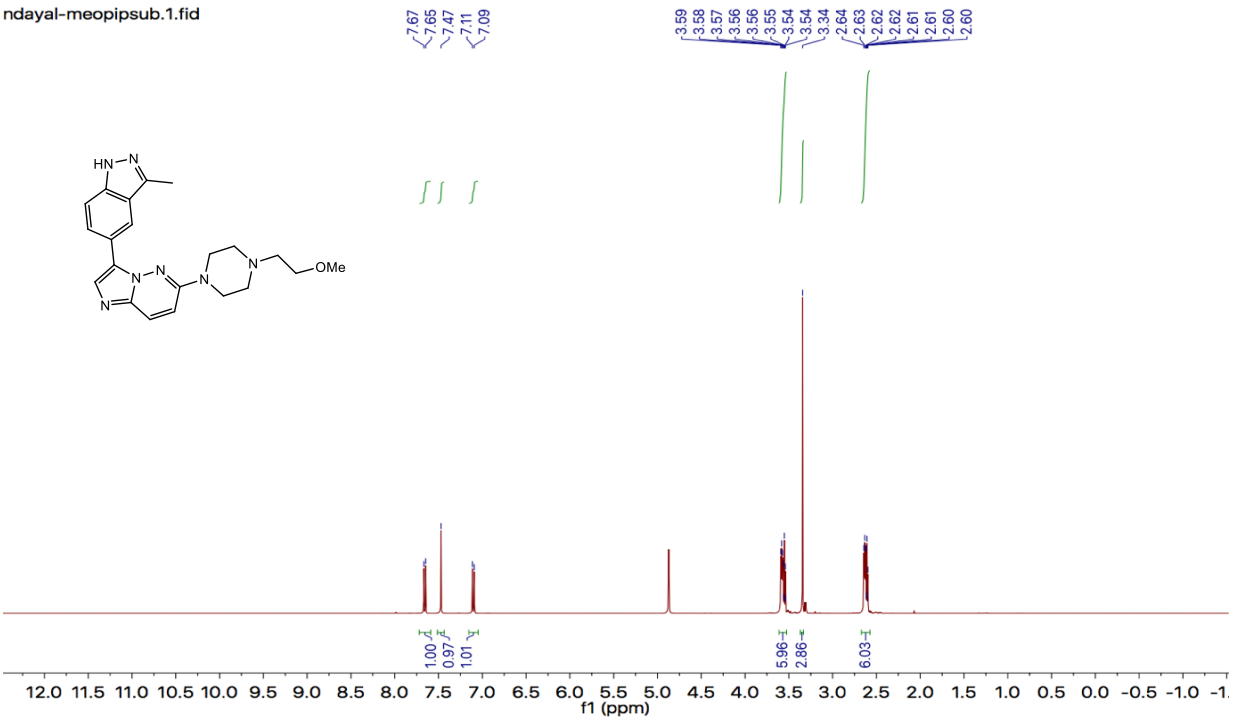
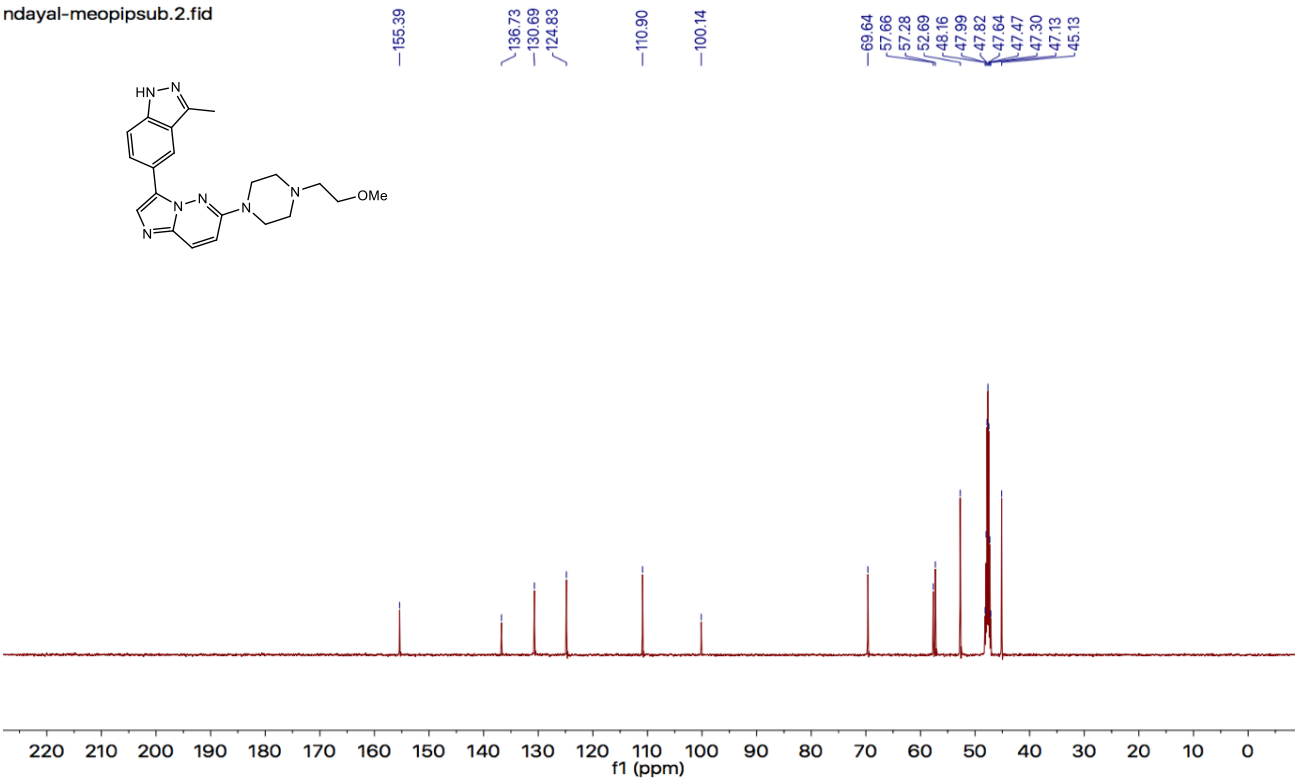
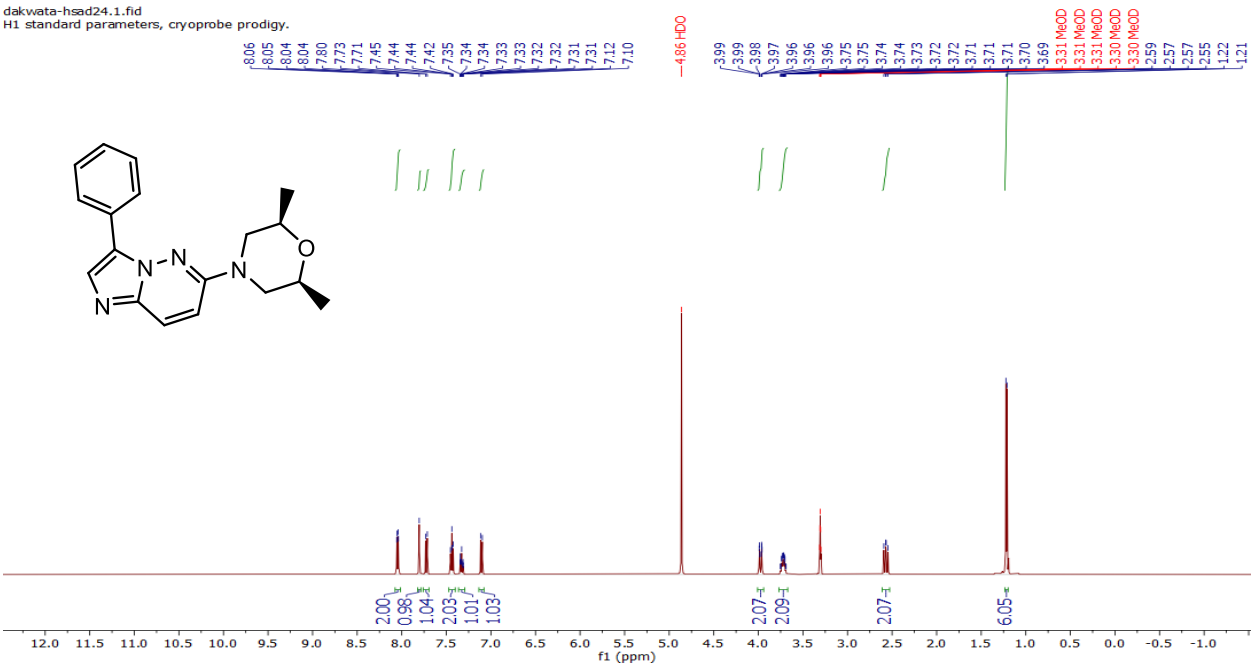
f1 (ppm)

Compound **4**


ndayal-meopipsub.1.fid
7.67
7.65
7.47
7.11
7.09
3.59
3.58
3.57
3.56
3.56
3.55
3.54
3.54
3.34
2.64
2.63
2.62
2.62
2.61
2.61
2.60
2.60
HN
N
N
N
N
N
N
OMe
1.00
0.97
1.01
5.96
2.86
6.03
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 -0.5 -1.0
f1 (ppm)


ndayal-meopipsub.2.fid
155.39
136.73
130.69
124.83
110.90
100.14
69.64
57.66
57.28
52.69
48.16
47.99
47.82
47.64
47.47
47.30
47.13
45.13
HN
N
N
N
N
N
OMe
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)

Compound **5**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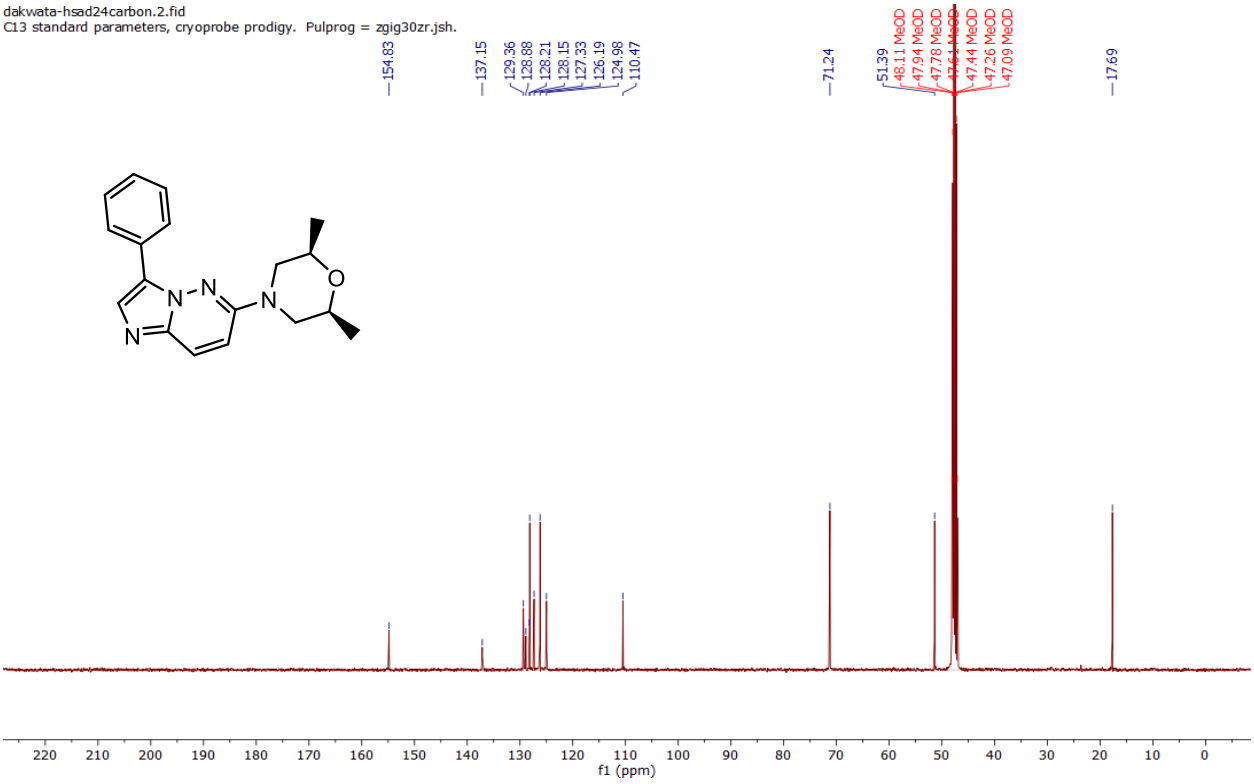
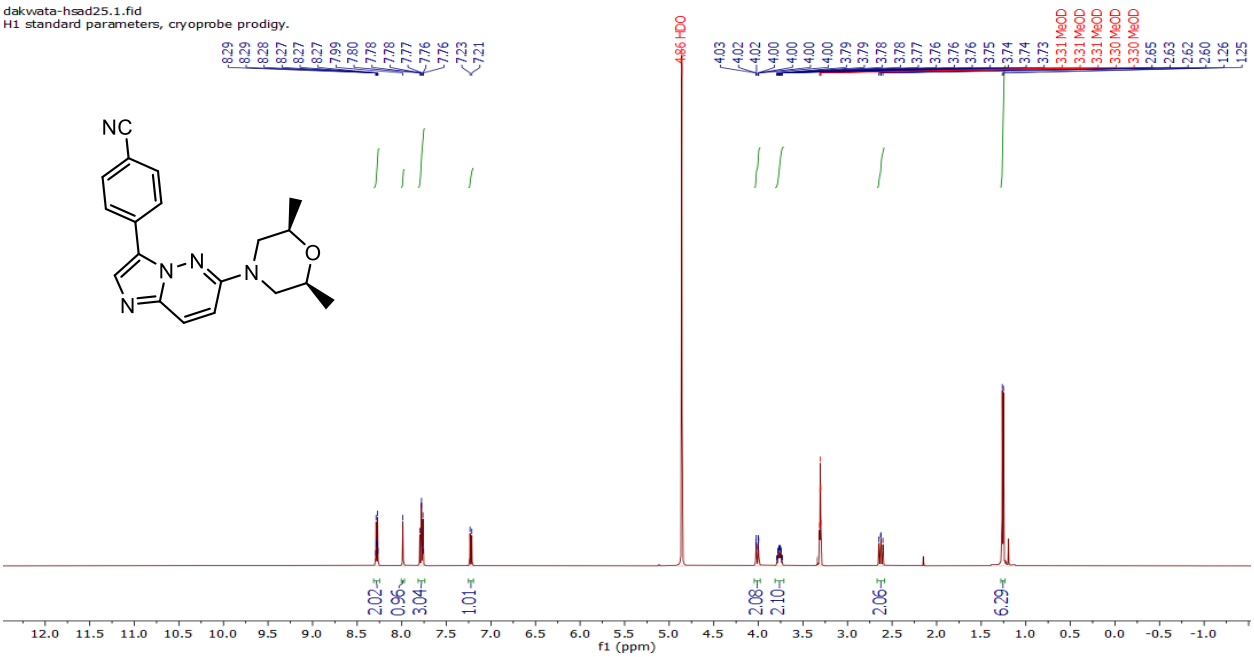
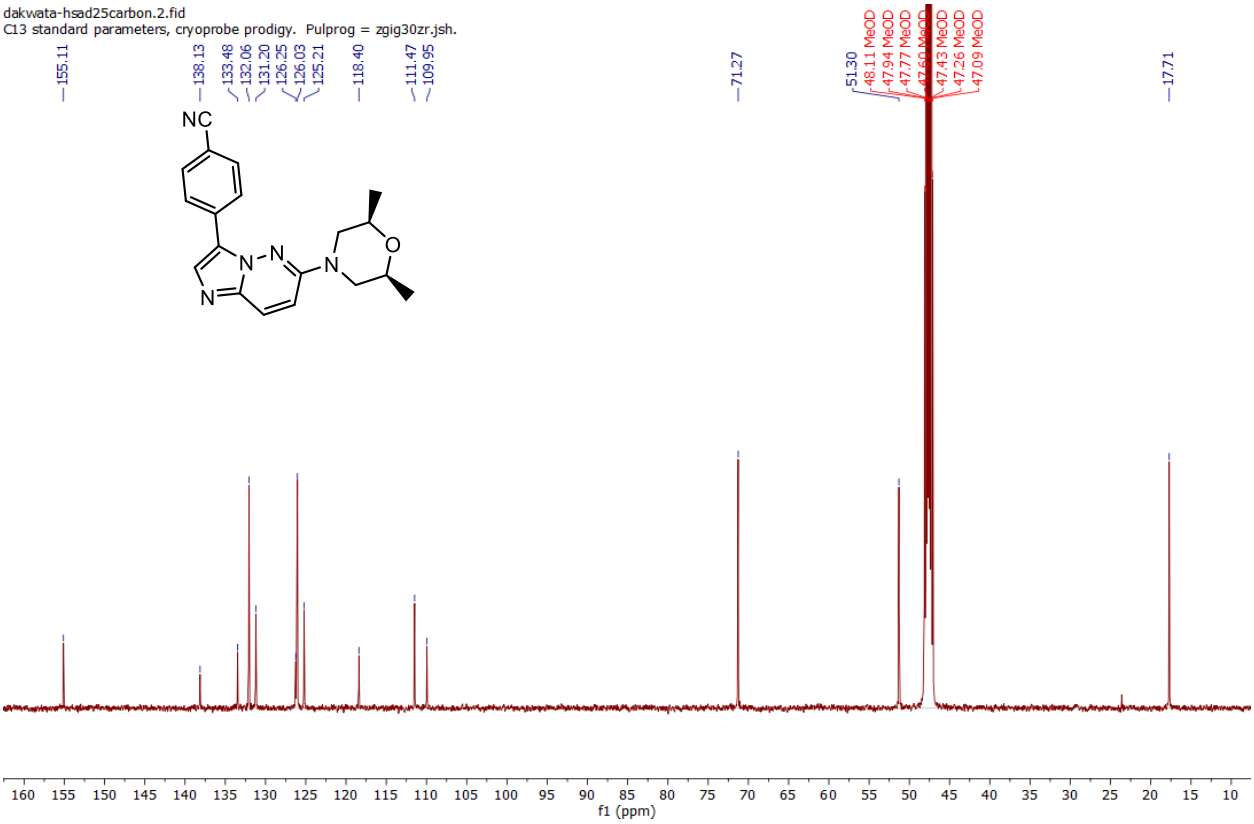
H1 standard parameters, cryoprobe prodigy.
f1 (ppm)

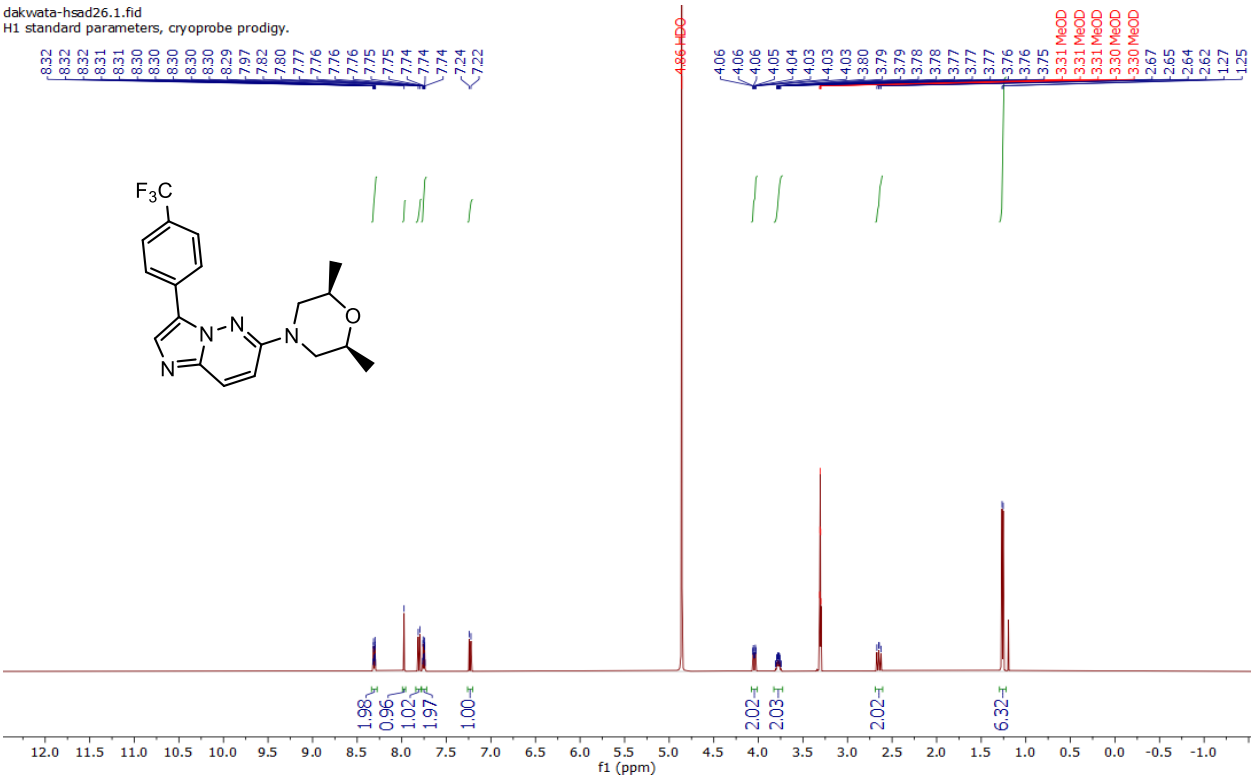

dakwata-hsad24carbon.2.fid
C13 standard parameters, cryoprobe prodigy. Pulprog = zgig30zr.jsh.
f1 (ppm)

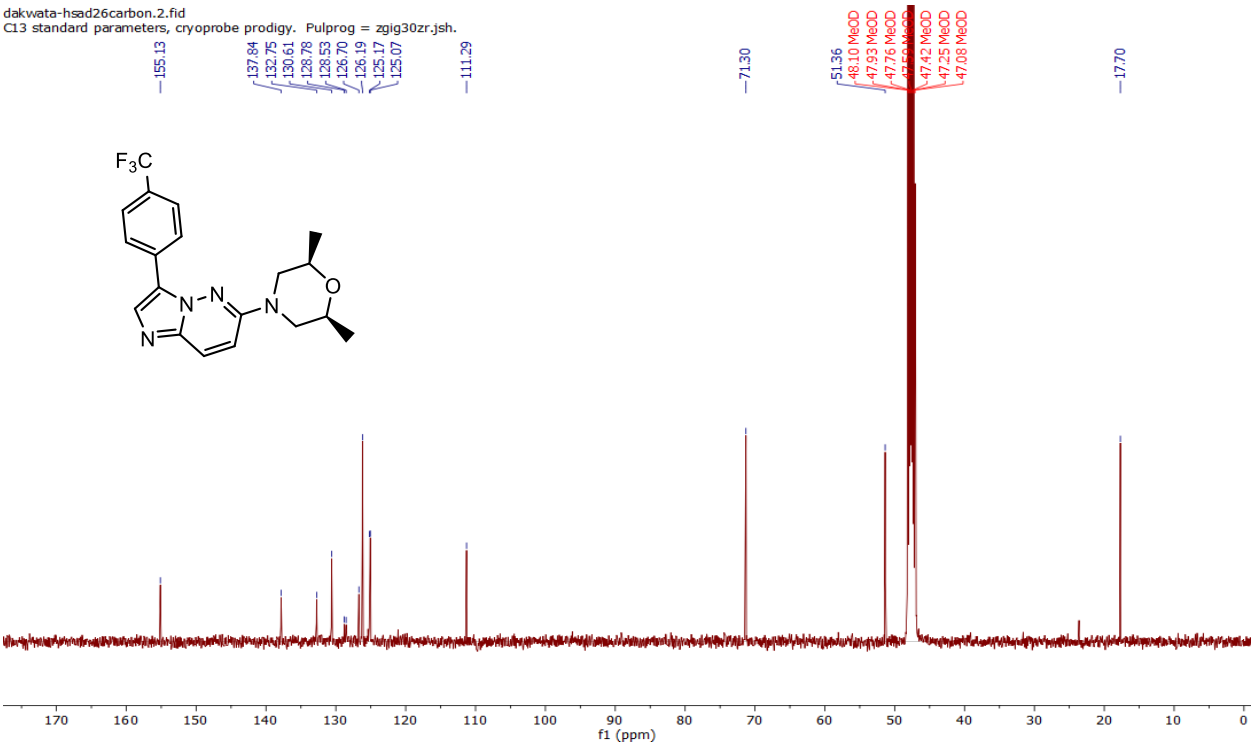
Compound **6**


dakwata-hsad25.1.fid
H1 standard parameters, cryoprobe prodigy.
NC
4.86 HDO
3.31 MeOD
3.30 MeOD
2.02
0.96
3.04
1.01
2.08
2.10
2.06
6.29
f1 (ppm)

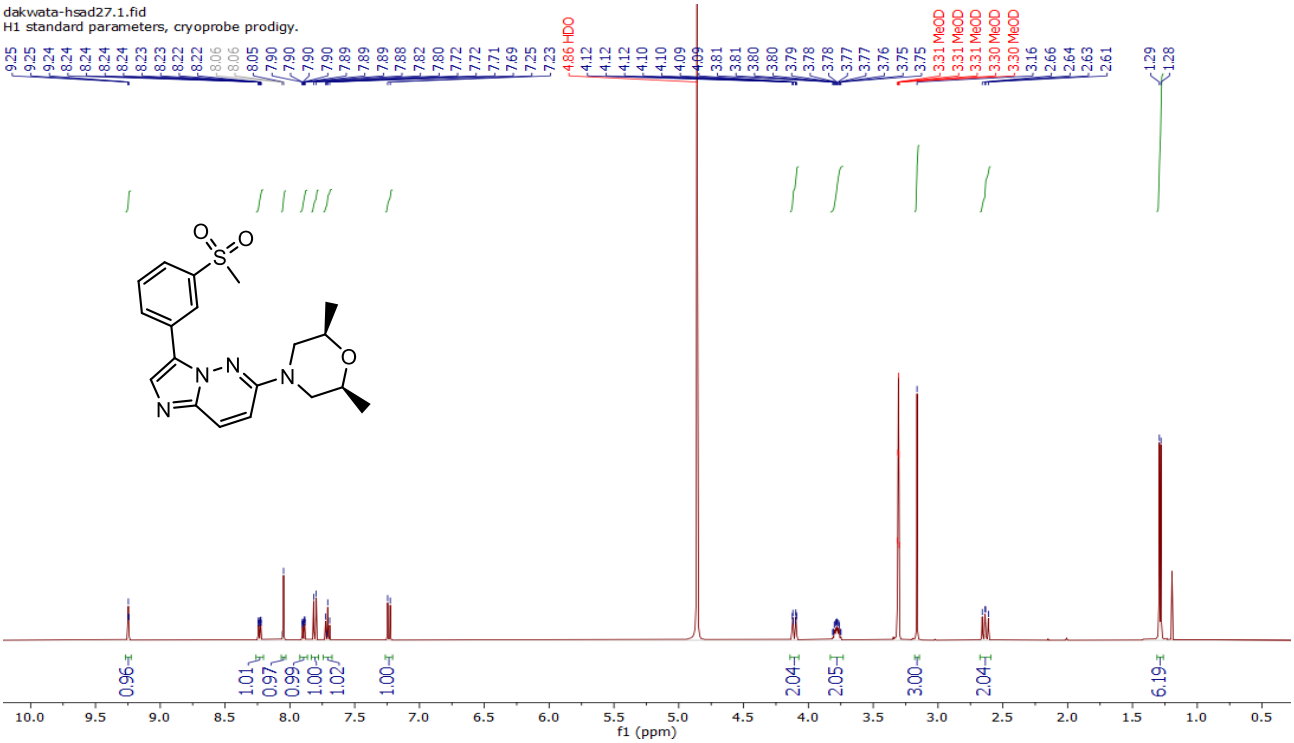

dakwata-hsad25carbon.2.fid
C13 standard parameters, cryoprobe prodigy. Pulprog = zgig30zr.jsh.
NC
155.11
138.13
133.48
132.06
131.20
126.25
126.03
125.21
118.40
111.47
109.95
71.27
51.30
48.11 MeOD
47.94 MeOD
47.77 MeOD
47.60 MeOD
47.43 MeOD
47.26 MeOD
47.09 MeOD
17.71
f1 (ppm)

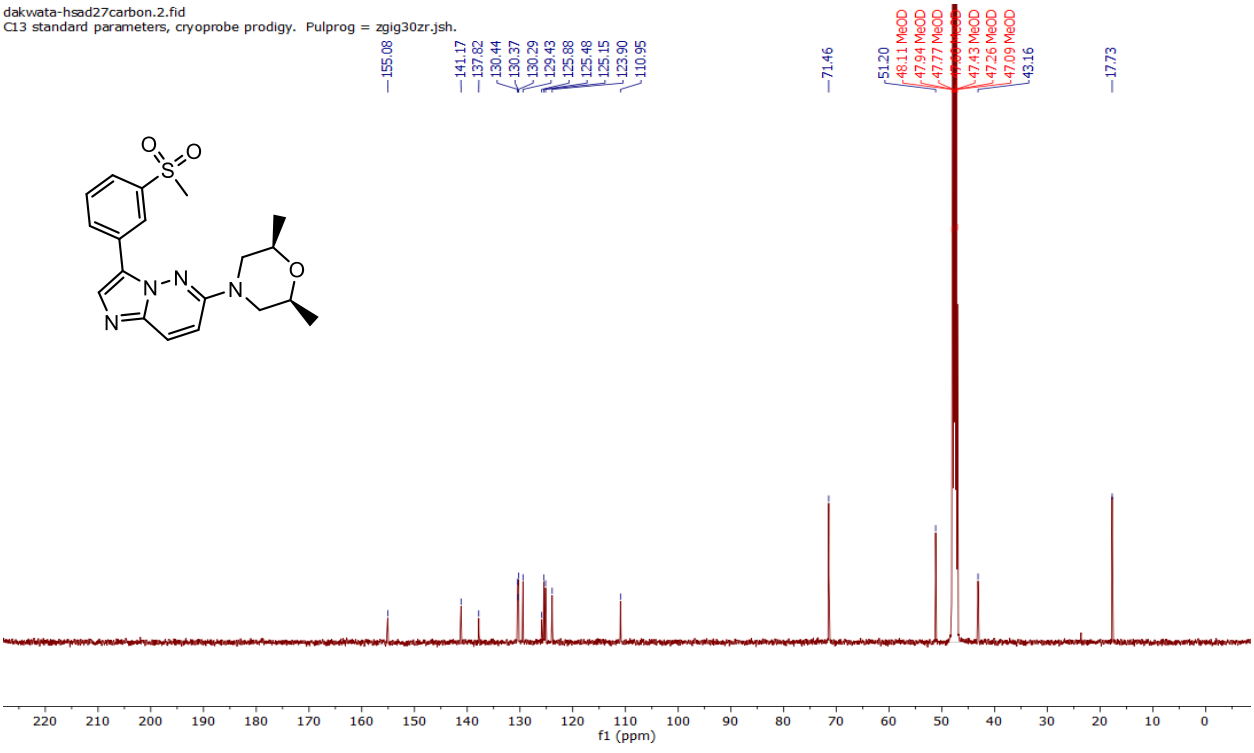
Compound **7**


dakwata-hsad26.1.fid
H1 standard parameters, cryoprobe prodigy.
F3C
f1 (ppm)

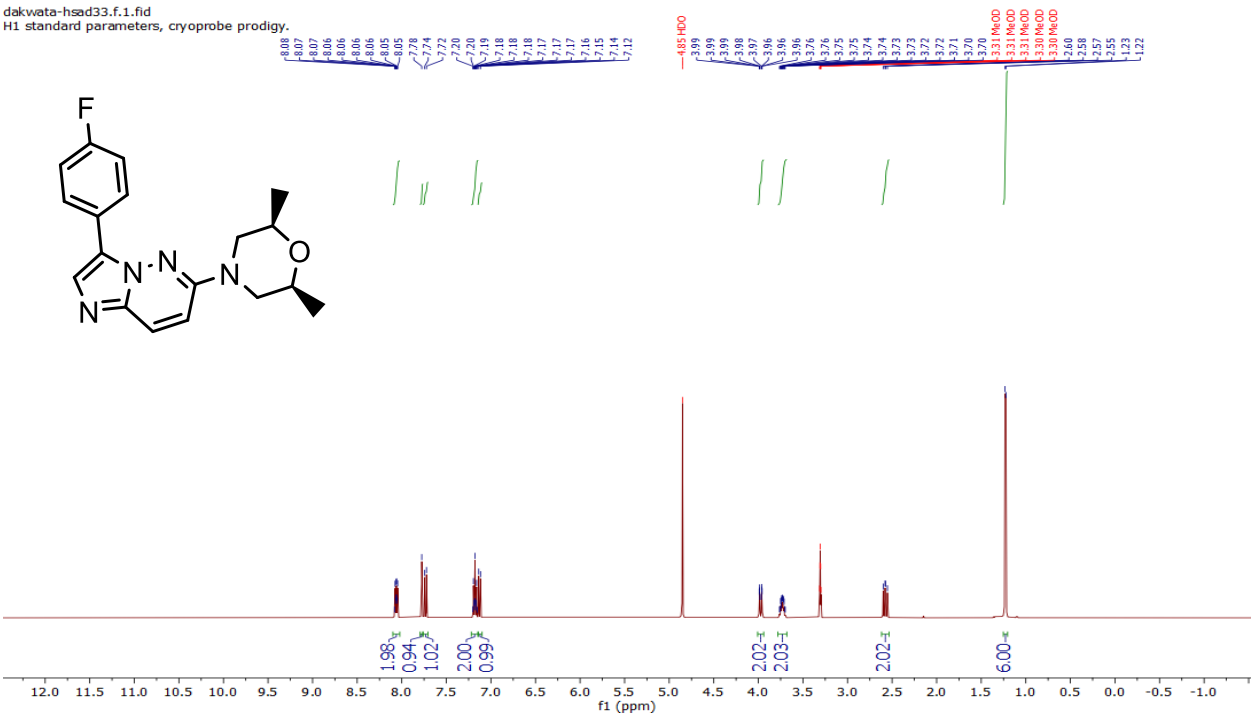

dakwata-hsad26carbon.2.fid
C13 standard parameters, cryoprobe prodigy. Pulprog = zgig30zr.jsh.
F3C
f1 (ppm)

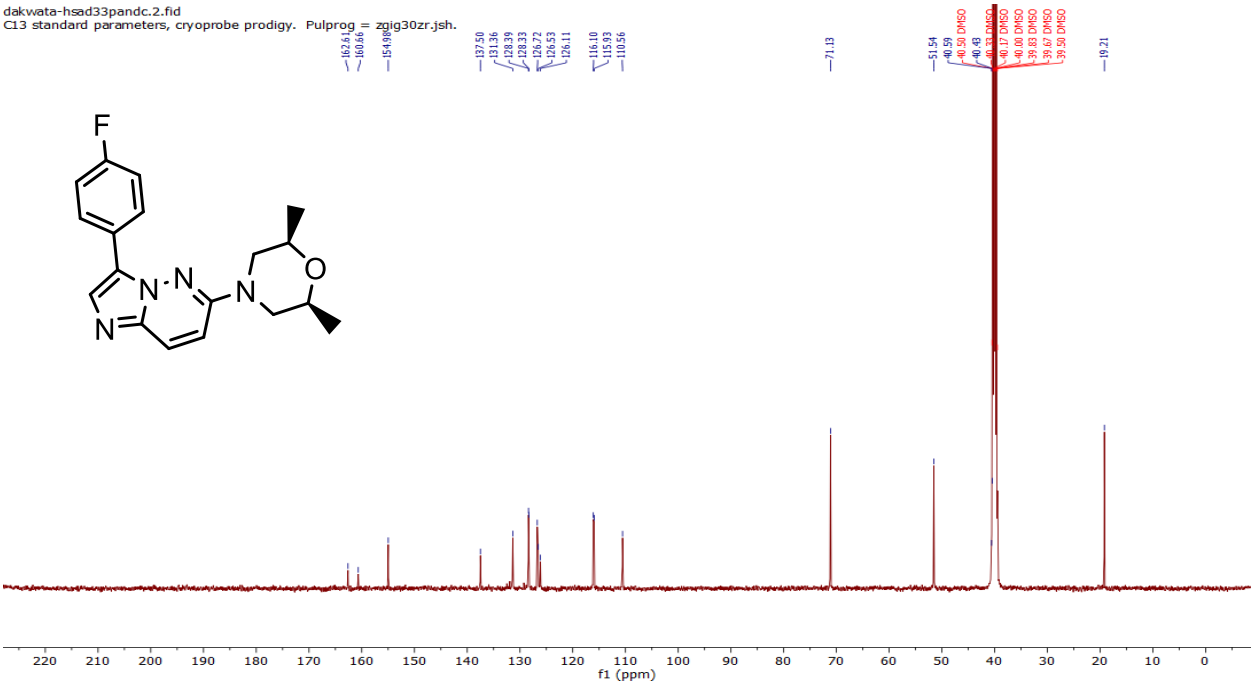
Compound **8**


dakwata-hsad27.1.fid
H1 standard parameters, cryoprobe prodigy.
f1 (ppm)

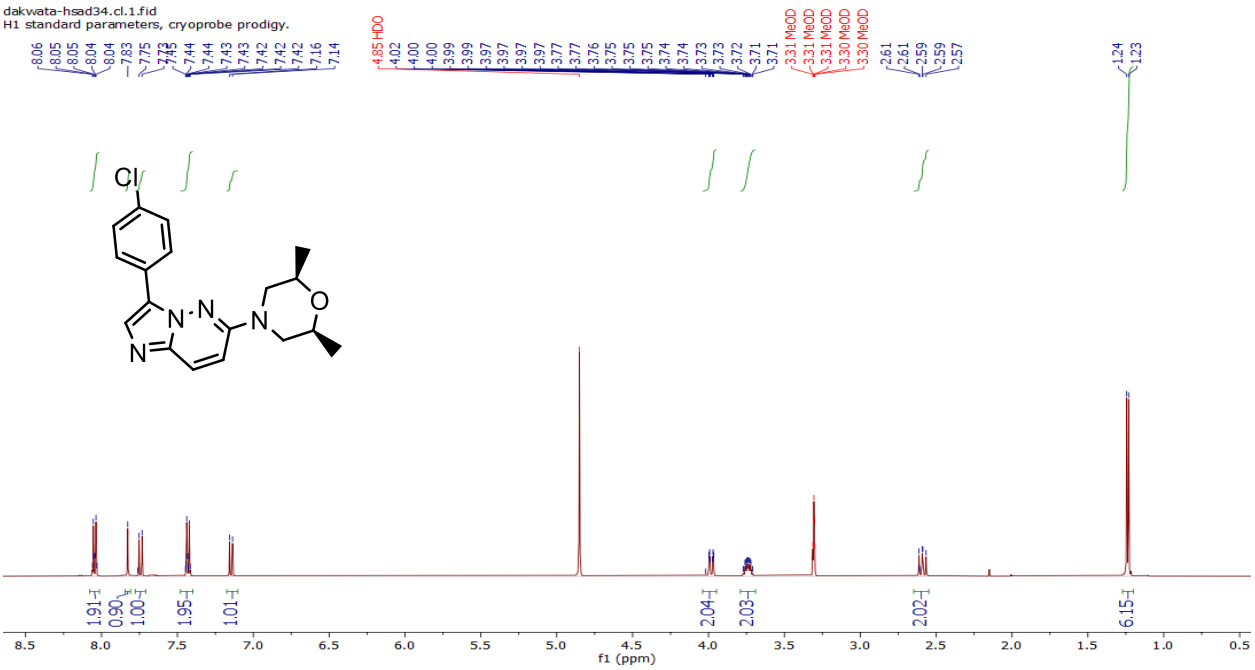

dakwata-hsad27carbon.2.fid
C13 standard parameters, cryoprobe prodigy. Pulprog = zgig30zr.jsh.
f1 (ppm)

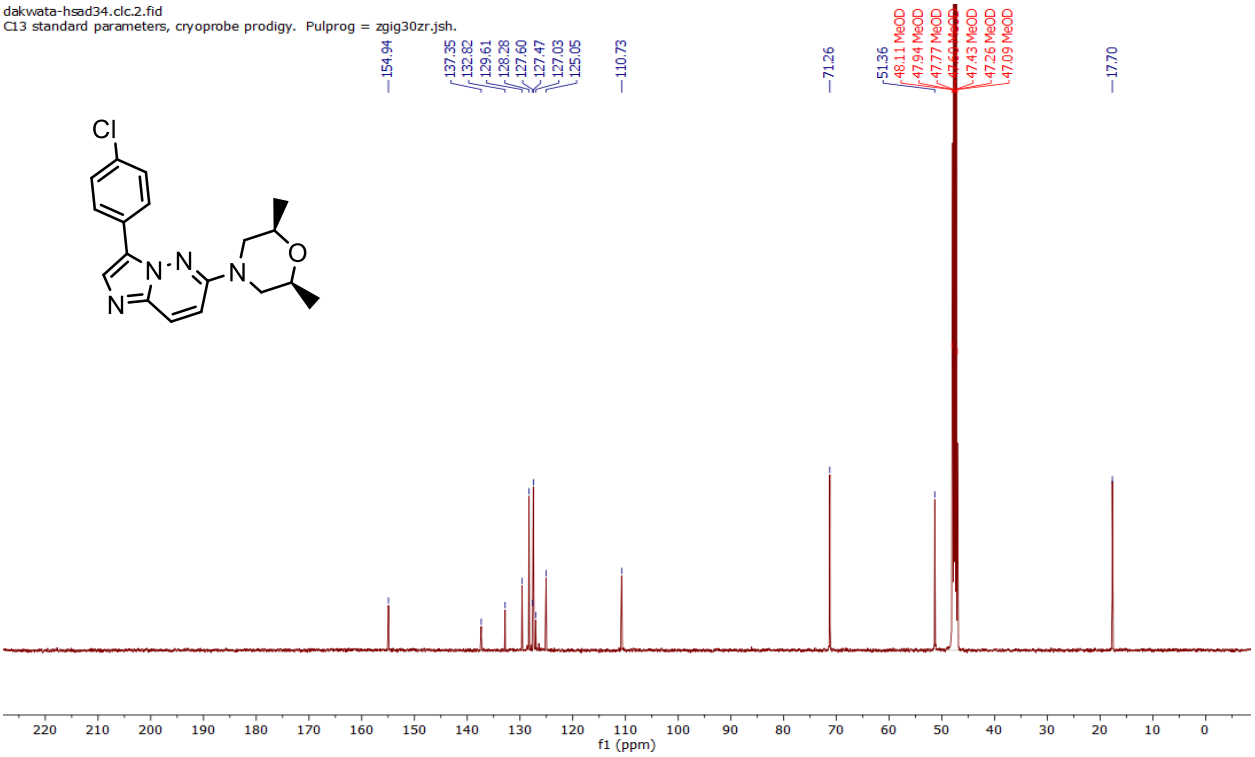
Compound **9**


dakwata-hsad33.f.1.fid
H1 standard parameters, cryoprobe prodigy.
F
N
N
N
O
1.98
0.94
1.02
2.00
0.99
2.02
2.03
2.02
6.00
f1 (ppm)

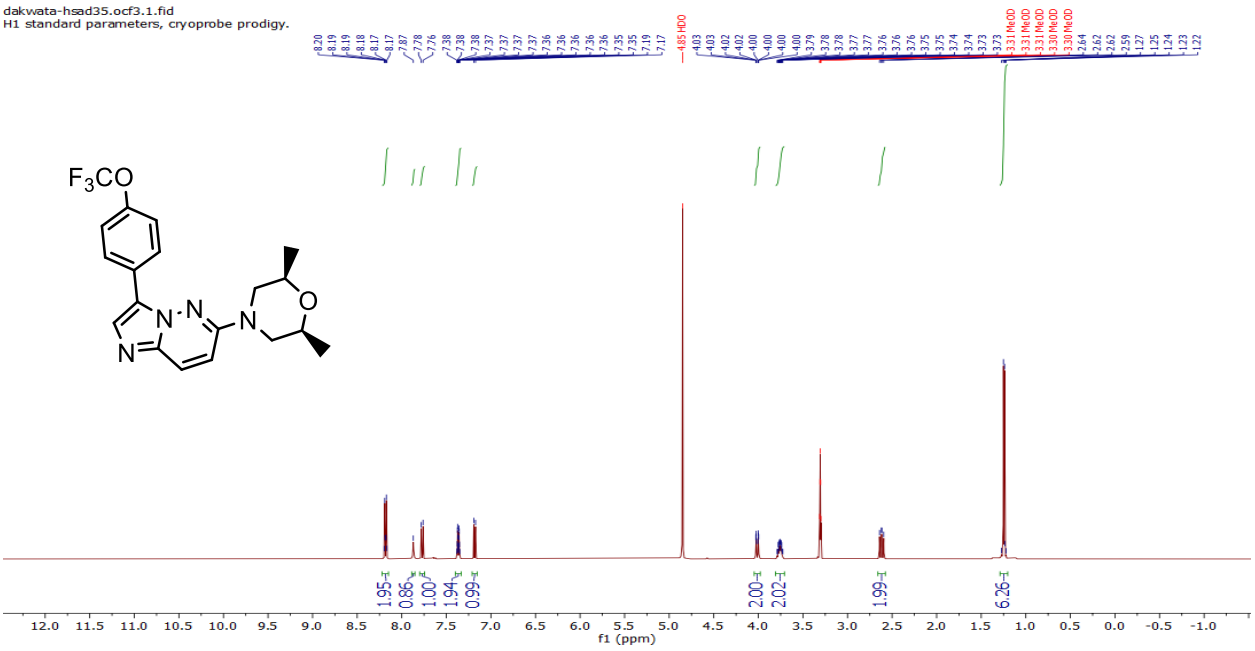

dakwata-hsad33pandc.2.fid
C13 standard parameters, cryoprobe prodigy. Pulprog = zgig30zr.jsh.
F
N
N
N
O
f1 (ppm)

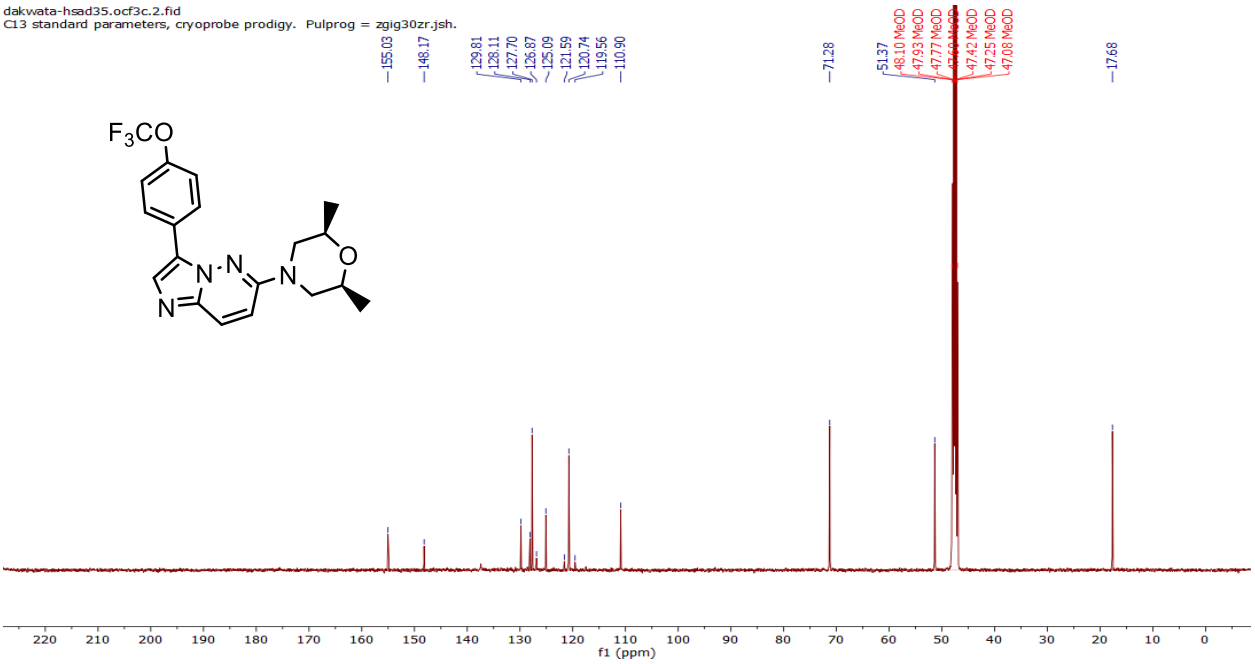
Compound **10**


dakwata-hsad34.cl.1.fid
H1 standard parameters, cryoprobe prodigy.
Cl
f1 (ppm)

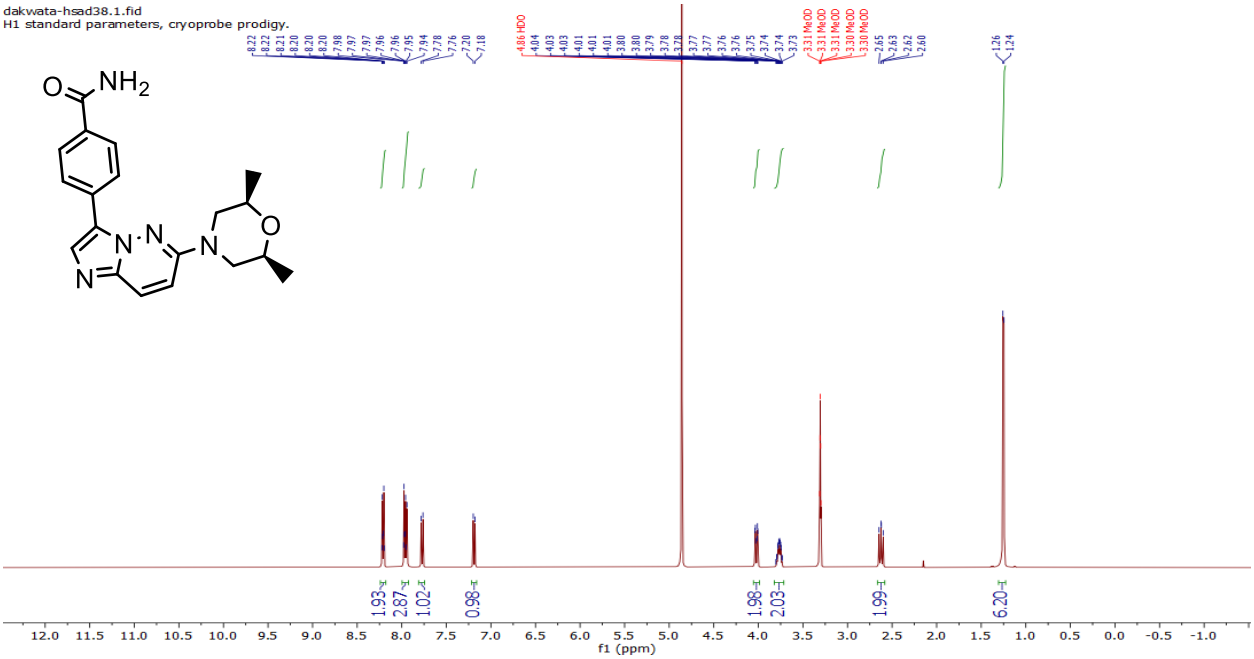

dakwata-hsad34.clc.2.fid
C13 standard parameters, cryoprobe prodigy. Pulprog = zgig30zr.jsh.
Cl
f1 (ppm)

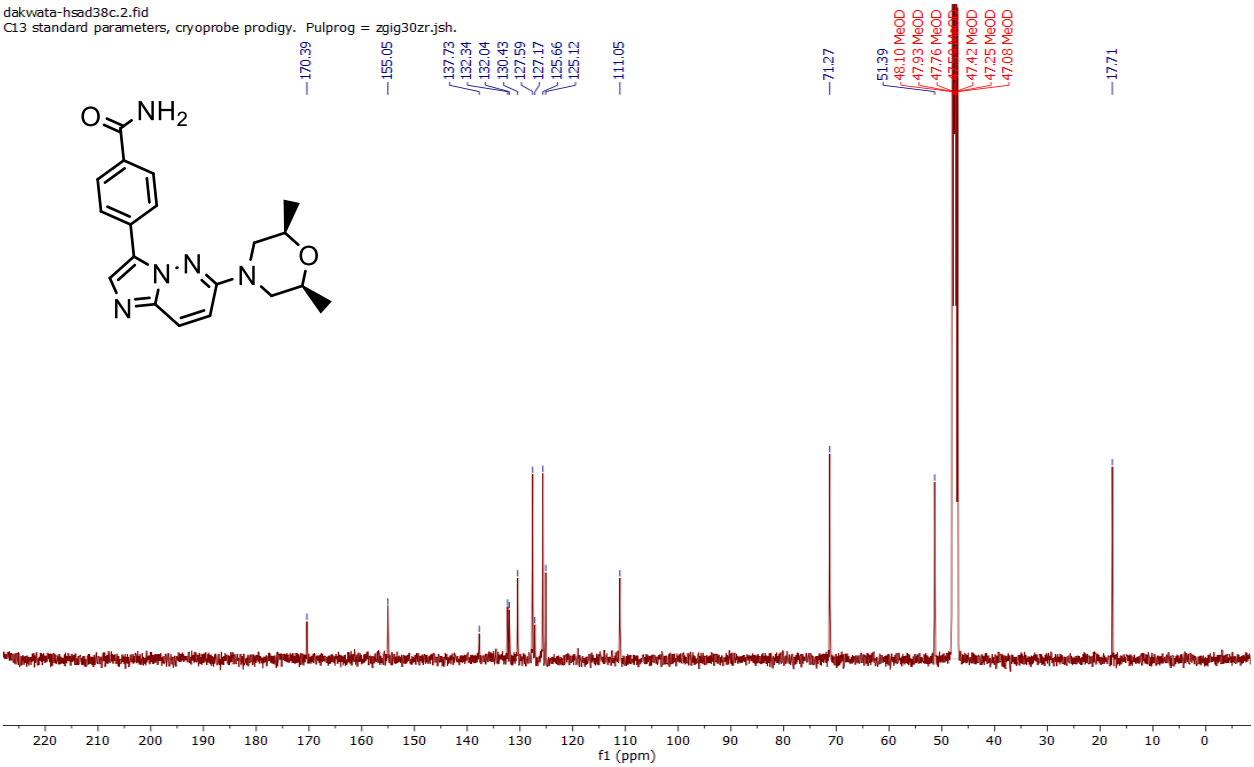
Compound **11**


dakwata-hsad35.ocf3.1.fid
H1 standard parameters, cryoprobe prodigy.
F3CO
f1 (ppm)

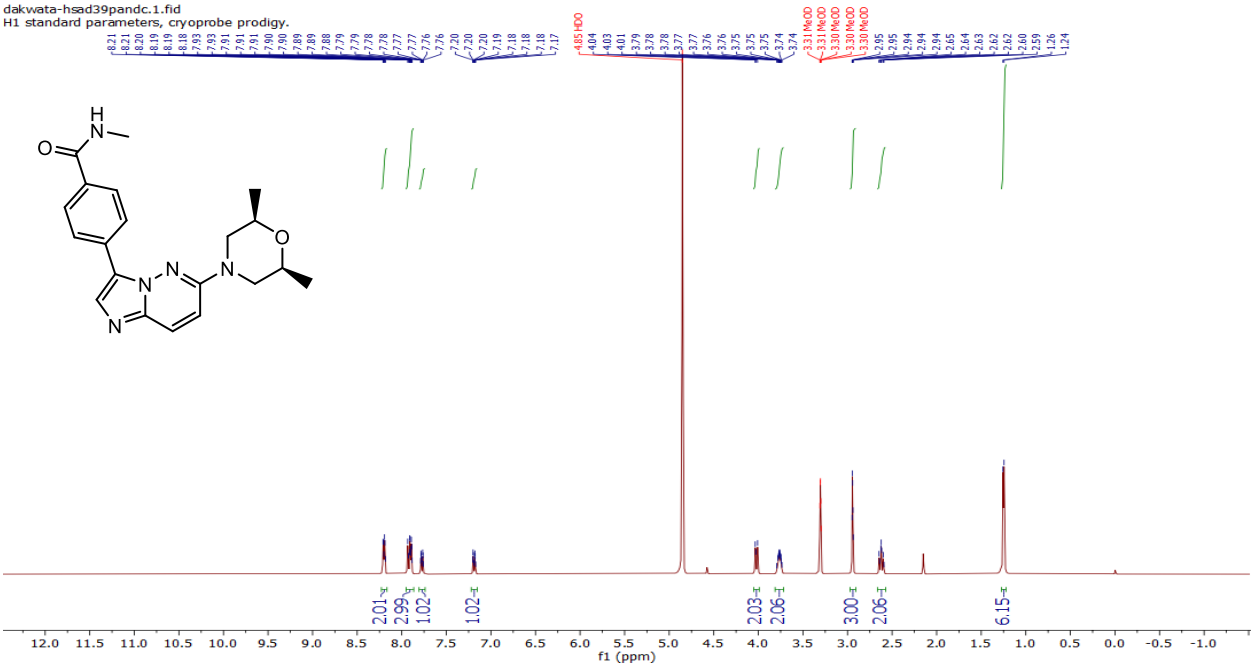

dakwata-hsad35.ocf3c.2.fid
C13 standard parameters, cryoprobe prodigy. Pulprog = zgig30zr.jsh.
F3CO
f1 (ppm)

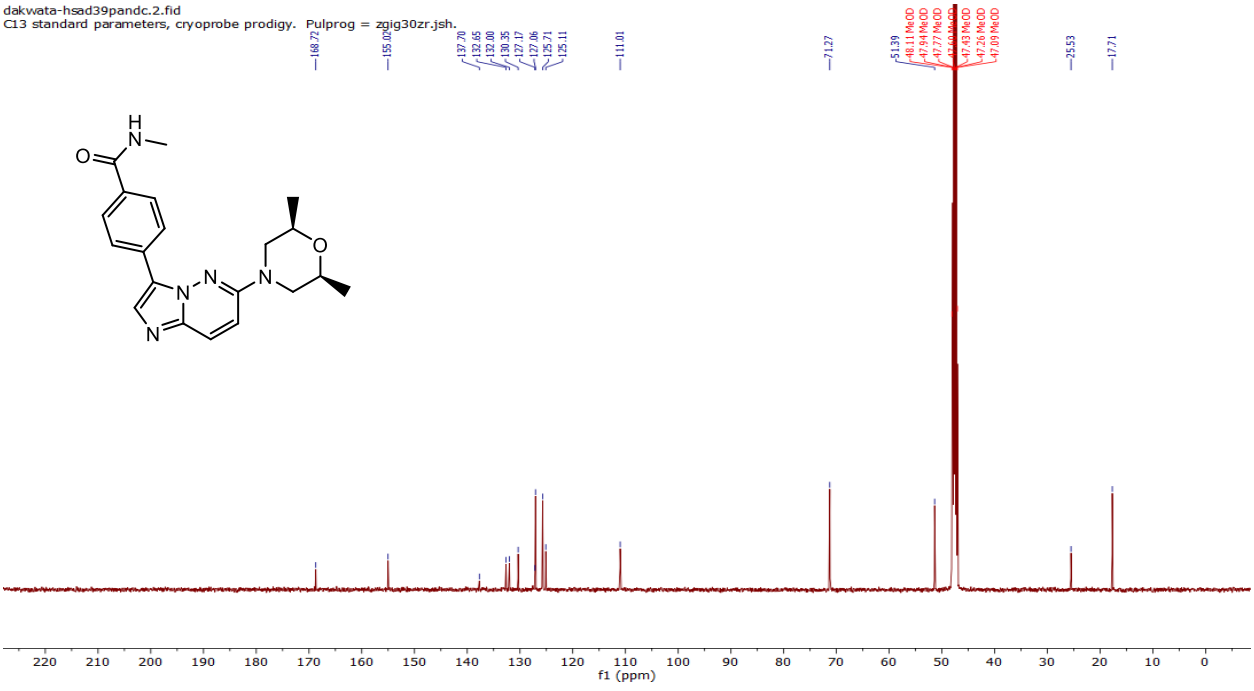
Compound **12**


dakwata-hsad38.1.fid
H1 standard parameters, cryoprobe prodigy.
f1 (ppm)

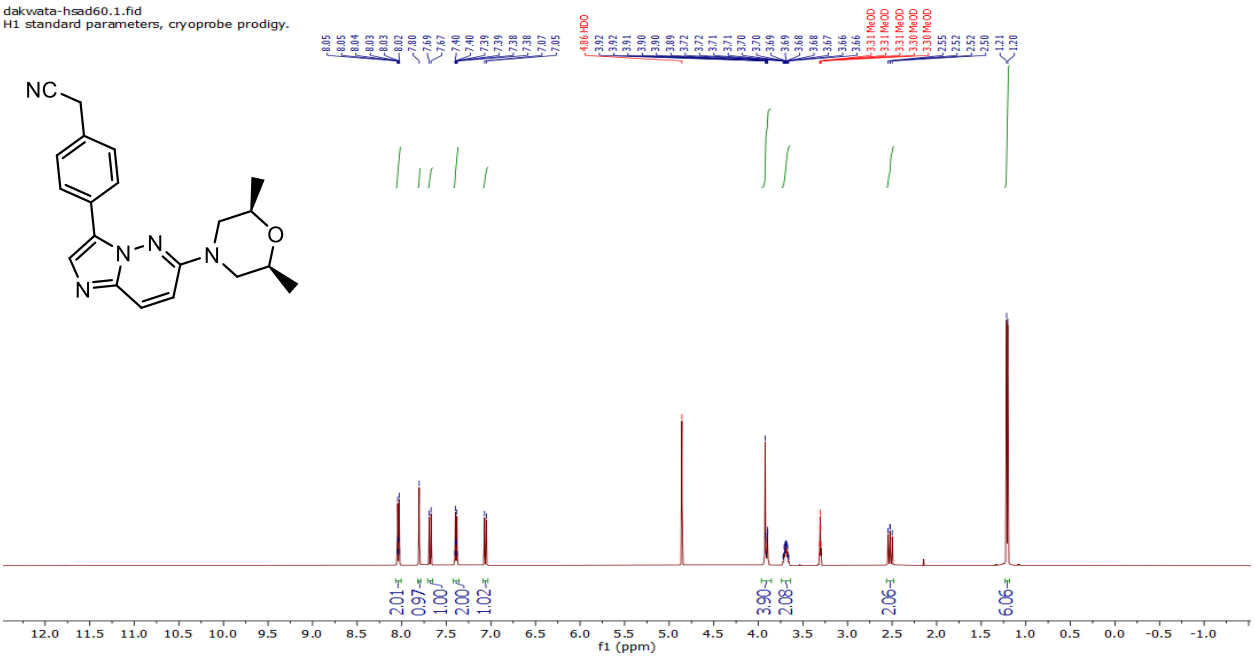

dakwata-hsad38c.2.fid
C13 standard parameters, cryoprobe prodigy. Pulprog = zgig30zr.jsh.
f1 (ppm)

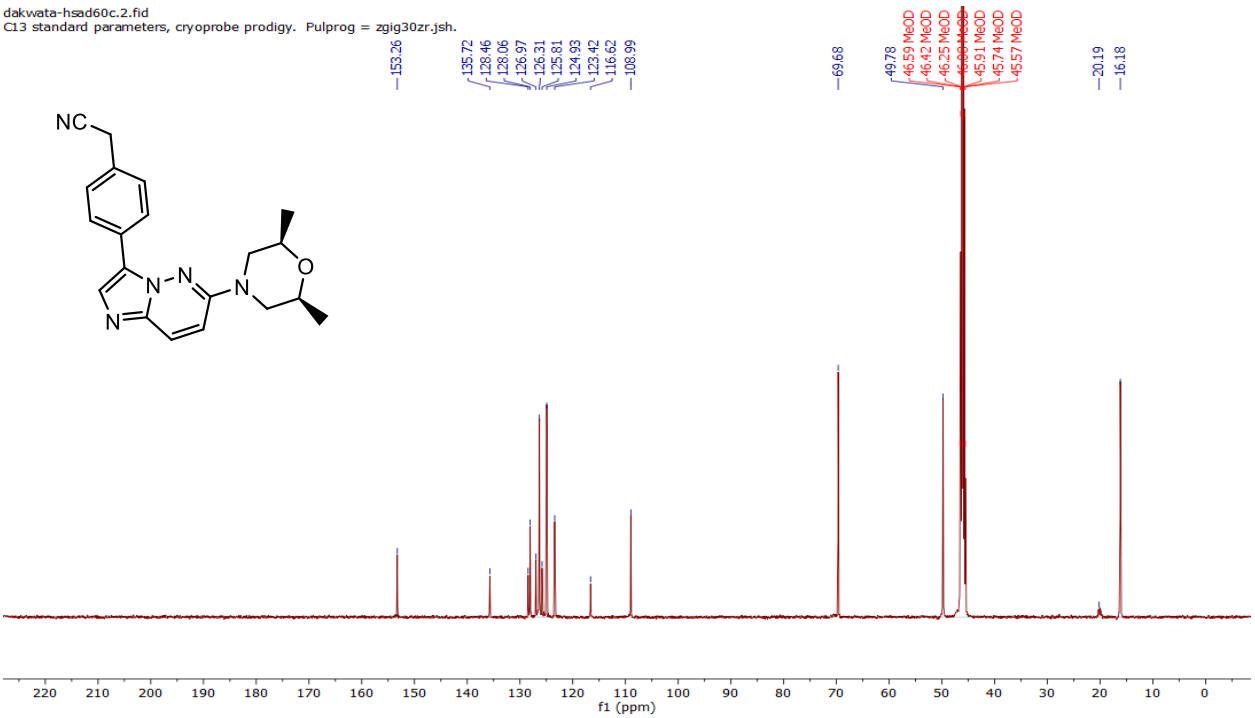
Compound **13**


dakwata-hsad39pandc.1.fid
H1 standard parameters, cryoprobe prodigy.
f1 (ppm)

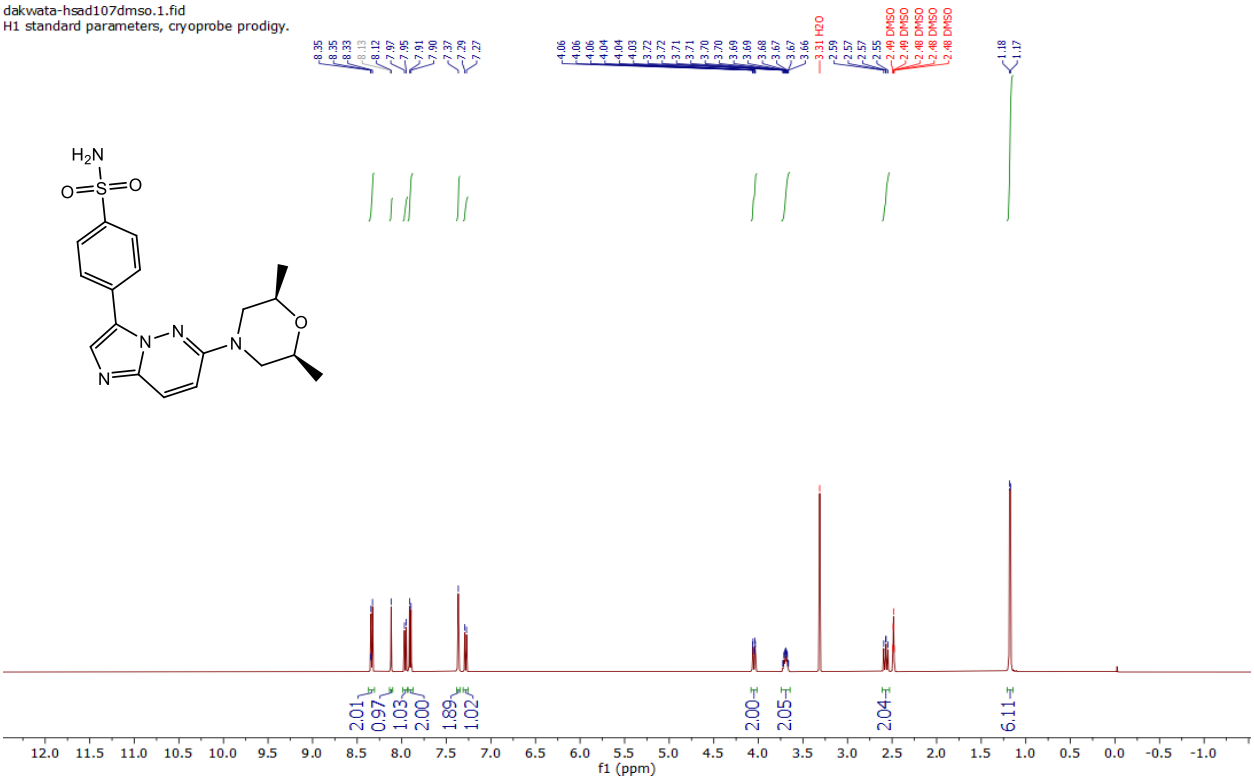

dakwata-hsad39pandc.2.fid
C13 standard parameters, cryoprobe prodigy. Pulprog = zgig30zr.jsh.
f1 (ppm)

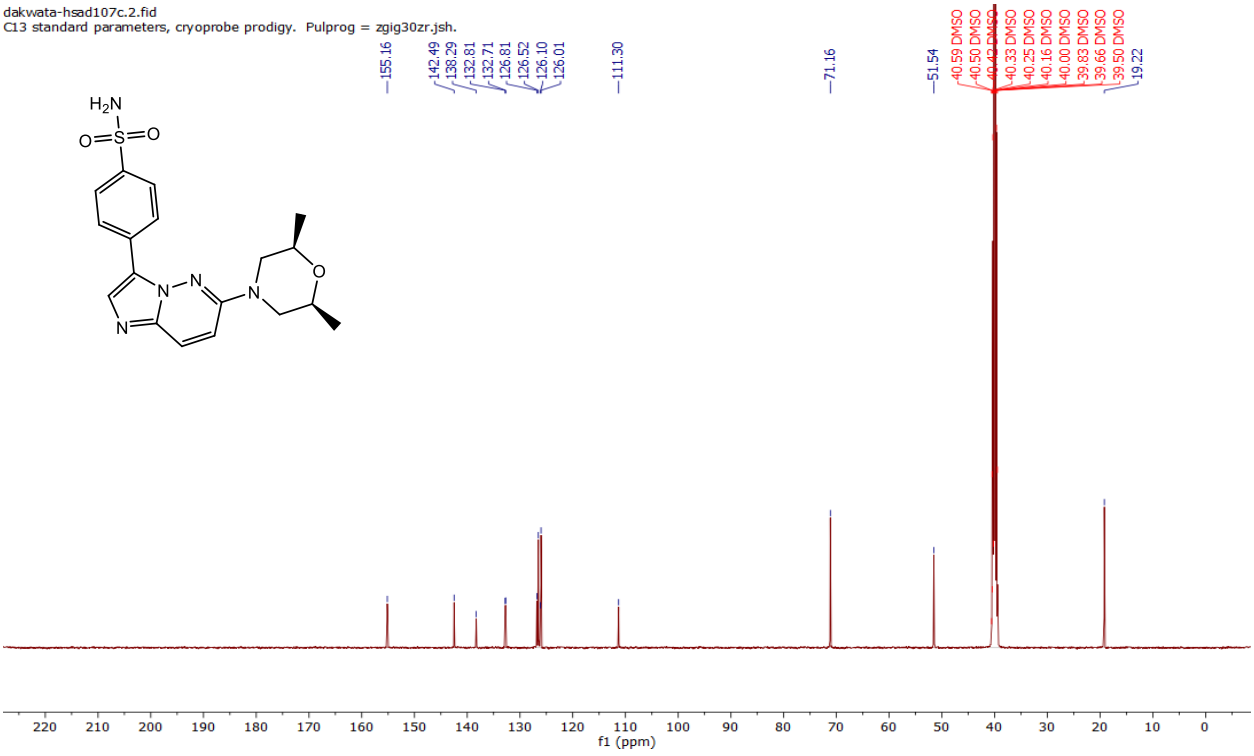
Compound **14**


dakwata-hsad60.1.fid
H1 standard parameters, cryoprobe prodigy.
NC
2.01
0.97
1.00
2.00
1.02
3.90
2.08
2.06
6.06
f1 (ppm)

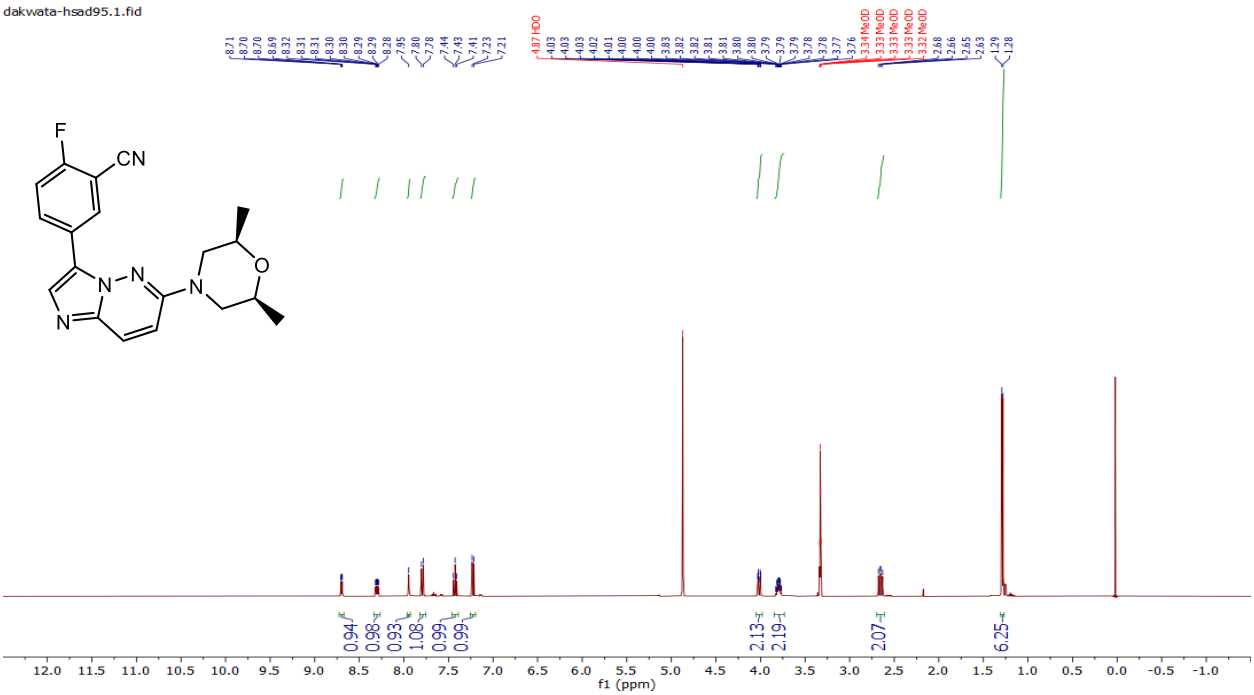

dakwata-hsad60c.2.fid
C13 standard parameters, cryoprobe prodigy. Pulprog = zgig30zr.jsh.
NC
153.26
135.72
128.46
128.06
126.97
126.31
125.81
124.93
123.42
116.62
108.99
69.68
49.78
46.59 MeOD
46.42 MeOD
46.25 MeOD
45.91 MeOD
45.74 MeOD
45.57 MeOD
20.19
16.18
f1 (ppm)

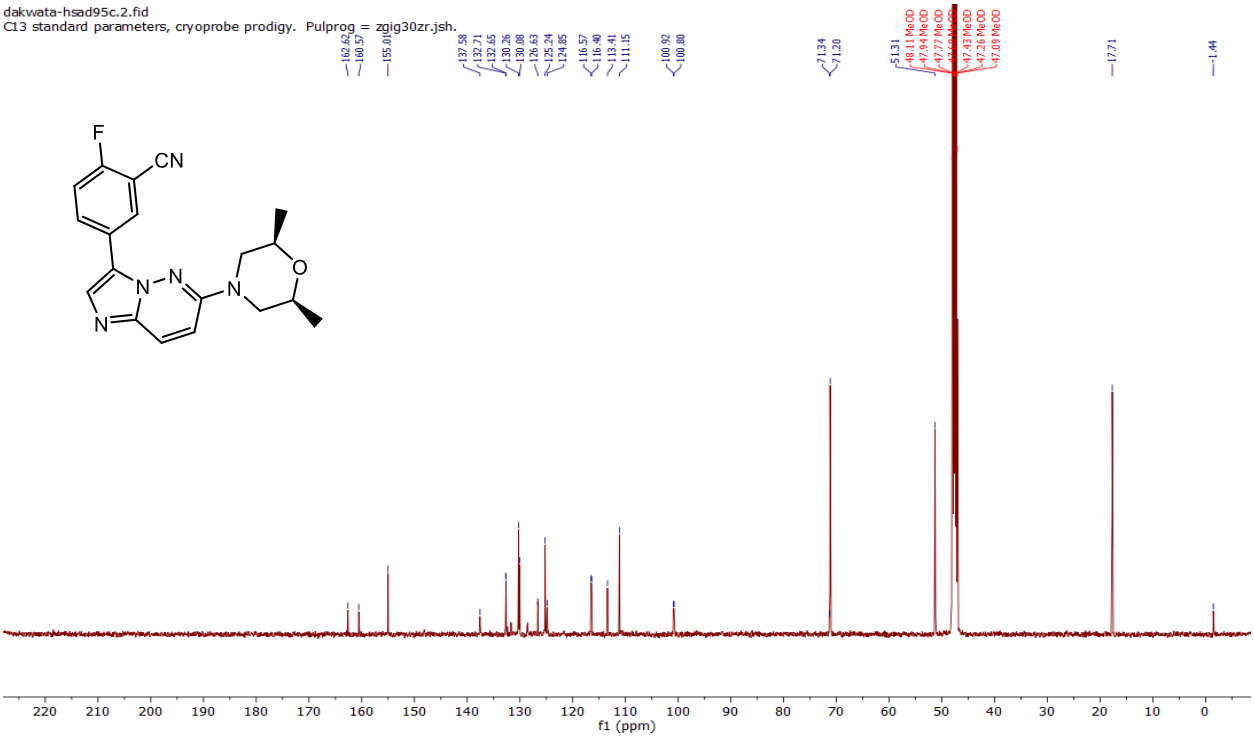
Compound **15**


dakwata-hsad107dmso.1.fid
H1 standard parameters, cryoprobe prodigy.
f1 (ppm)

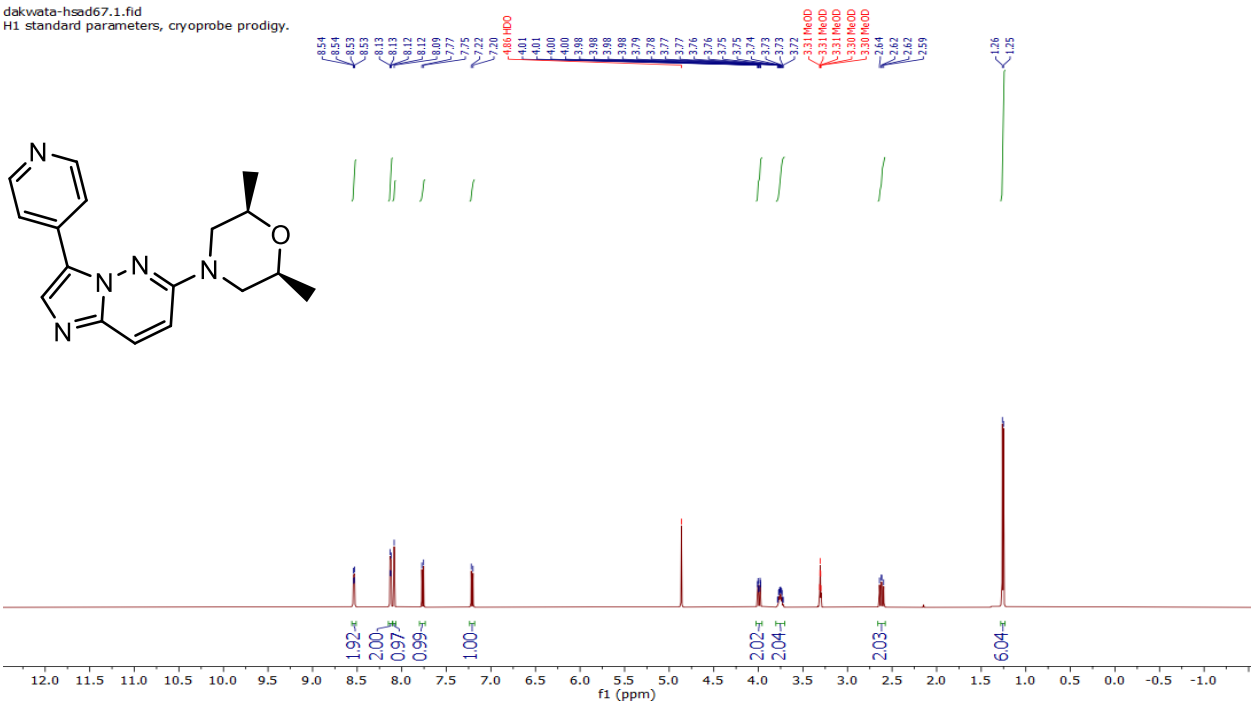

dakwata-hsad107c.2.fid
C13 standard parameters, cryoprobe prodigy. Pulprog = zgig30zr.jsh.
f1 (ppm)

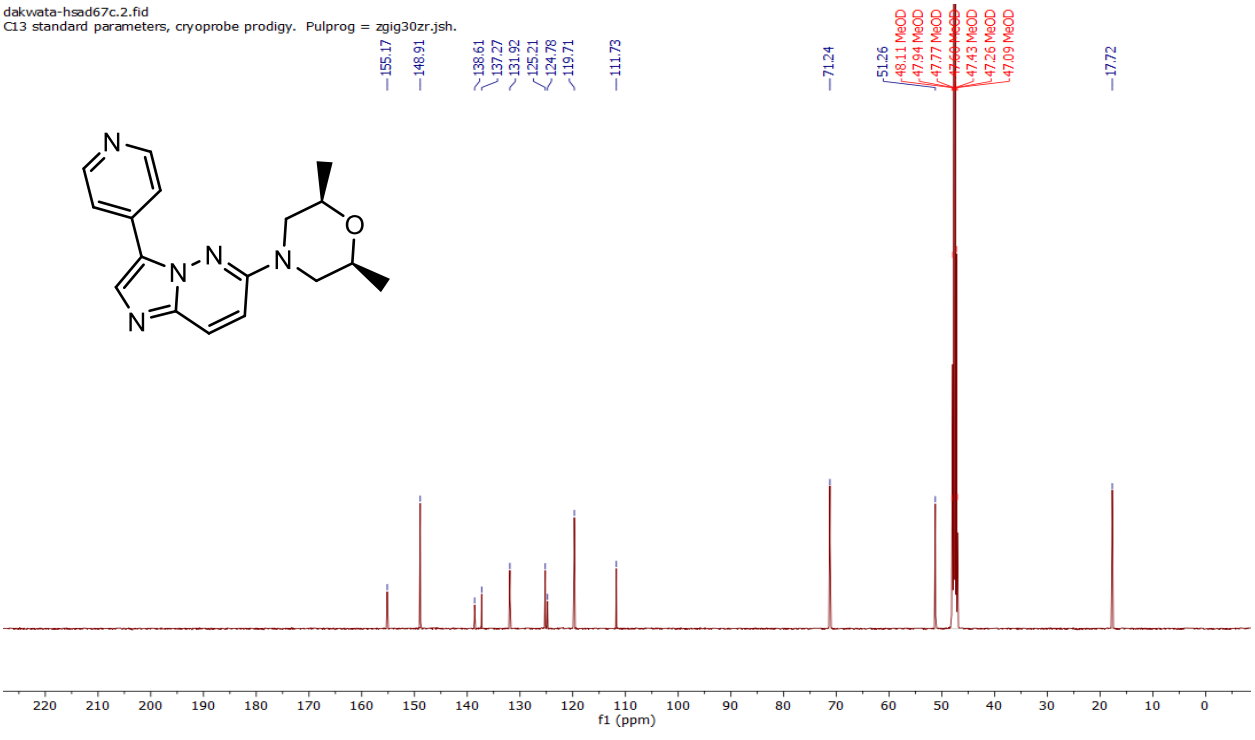
Compound **16**


dakwata-hsad95.1.fid
F
CN
N
N
N
O
f1 (ppm)

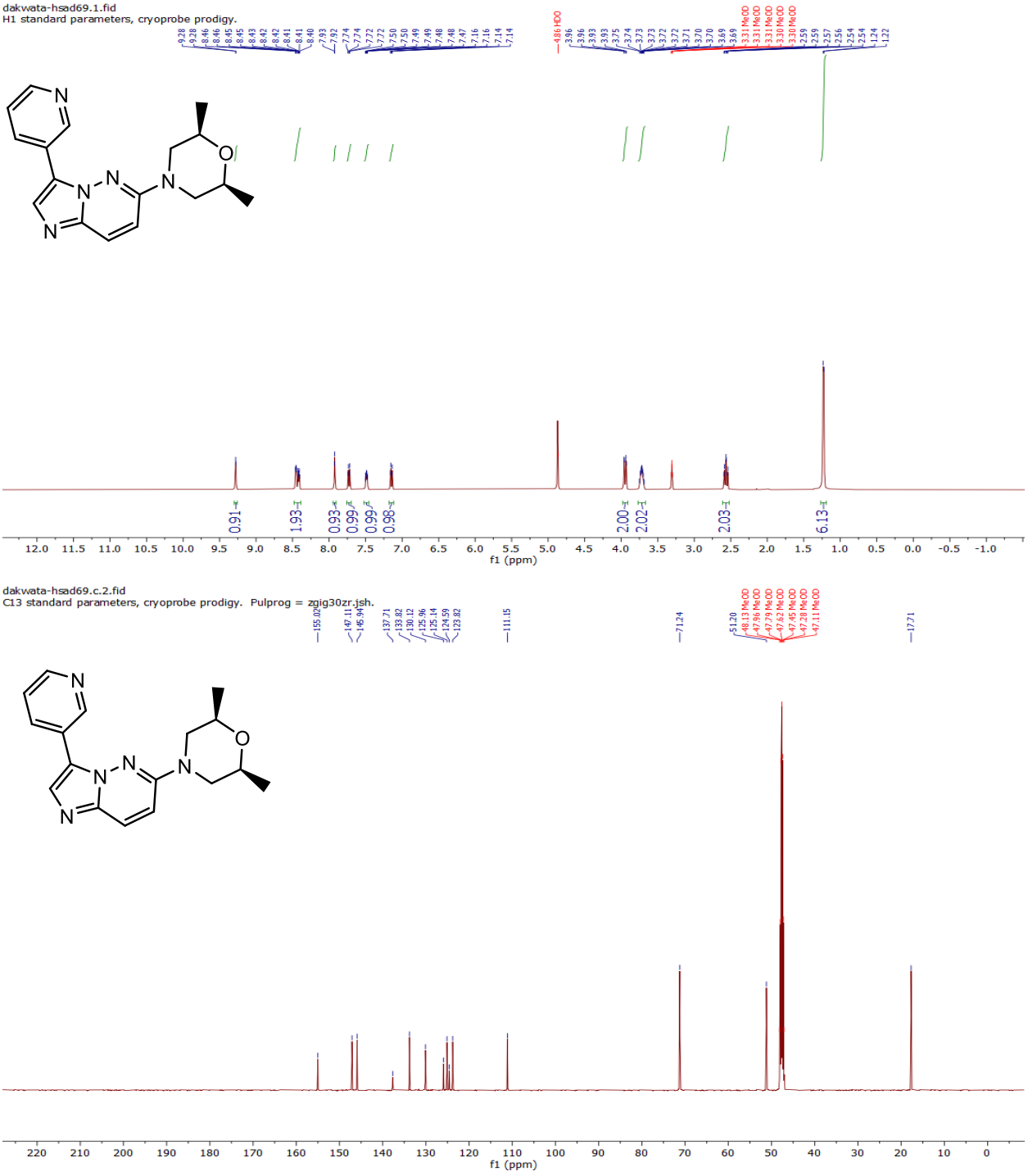

dakwata-hsad95c.2.fid
C13 standard parameters, cryoprobe prodigy. Pulprog = zgig30zr.jsh.
F
CN
N
N
N
O
f1 (ppm)

Compound **17**

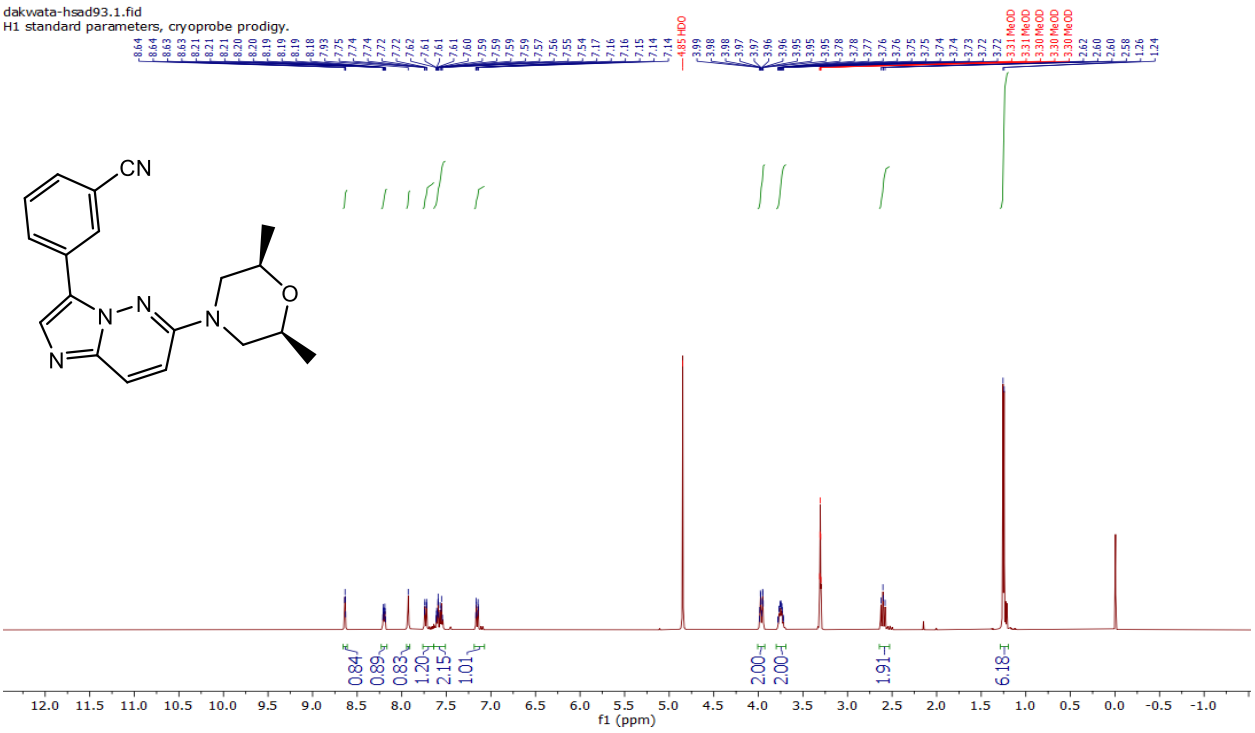

dakwata-hsad67.1.fid
H1 standard parameters, cryoprobe prodigy.
f1 (ppm)

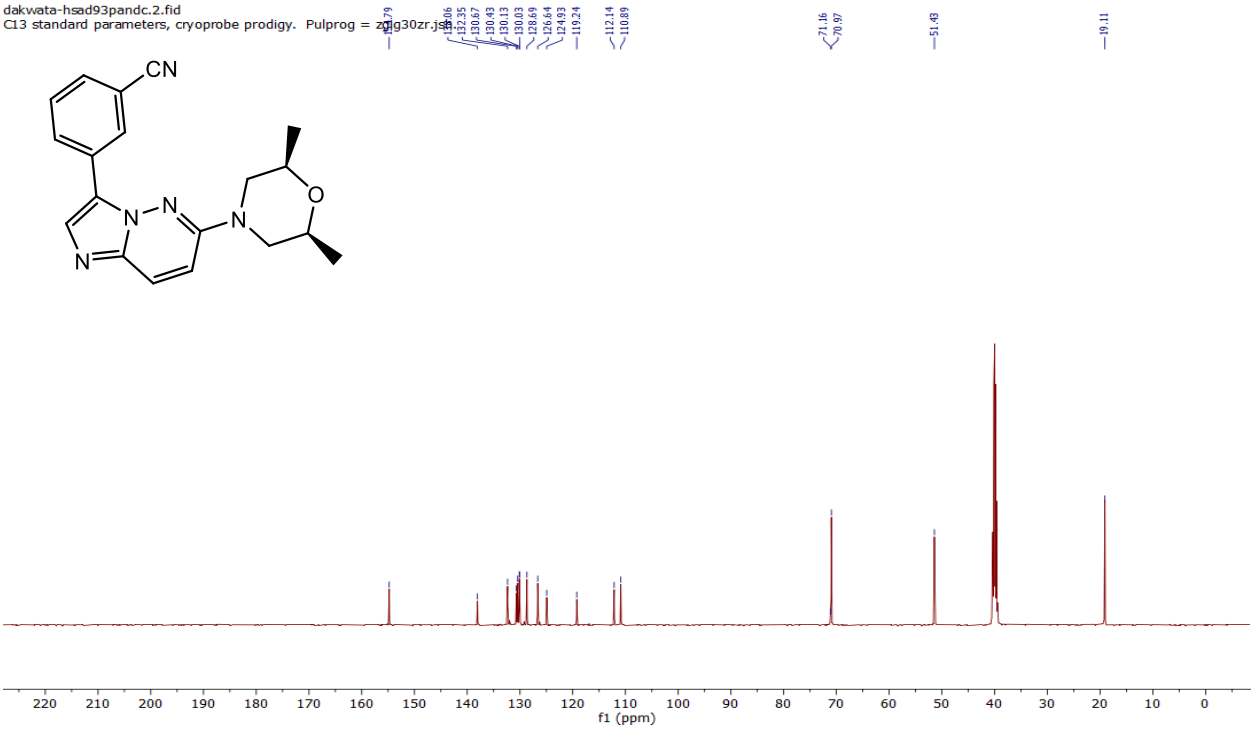

dakwata-hsad67c.2.fid
C13 standard parameters, cryoprobe prodigy. Pulprog = zgig30zr.jsh.
f1 (ppm)

Compound **18**

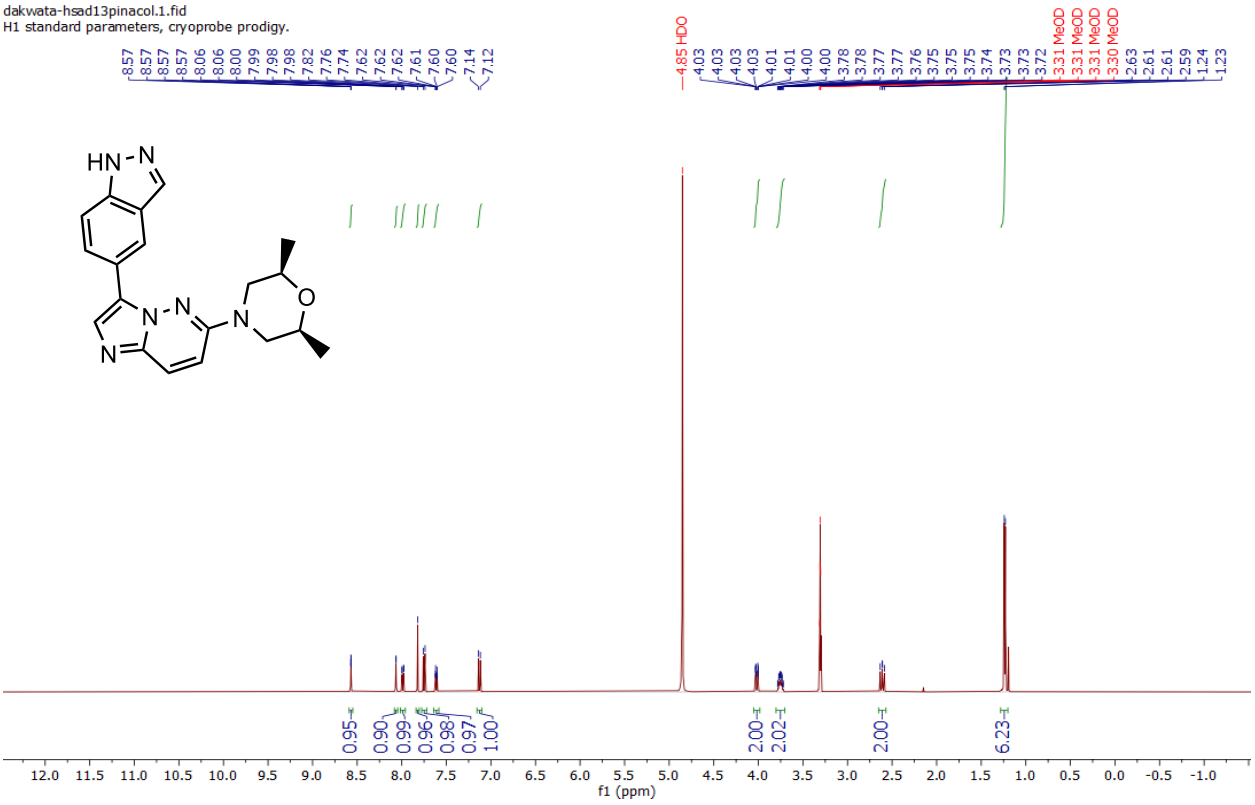

dakwata-hsad69.1.fid
H1 standard parameters, cryoprobe prodigy.
0.91
1.93
0.93
0.99
0.99
0.98
2.00
2.02
2.03
6.13
f1 (ppm)
dakwata-hsad69.c.2.fid
C13 standard parameters, cryoprobe prodigy. Pulprog = zgig30zr.jsh.
f1 (ppm)

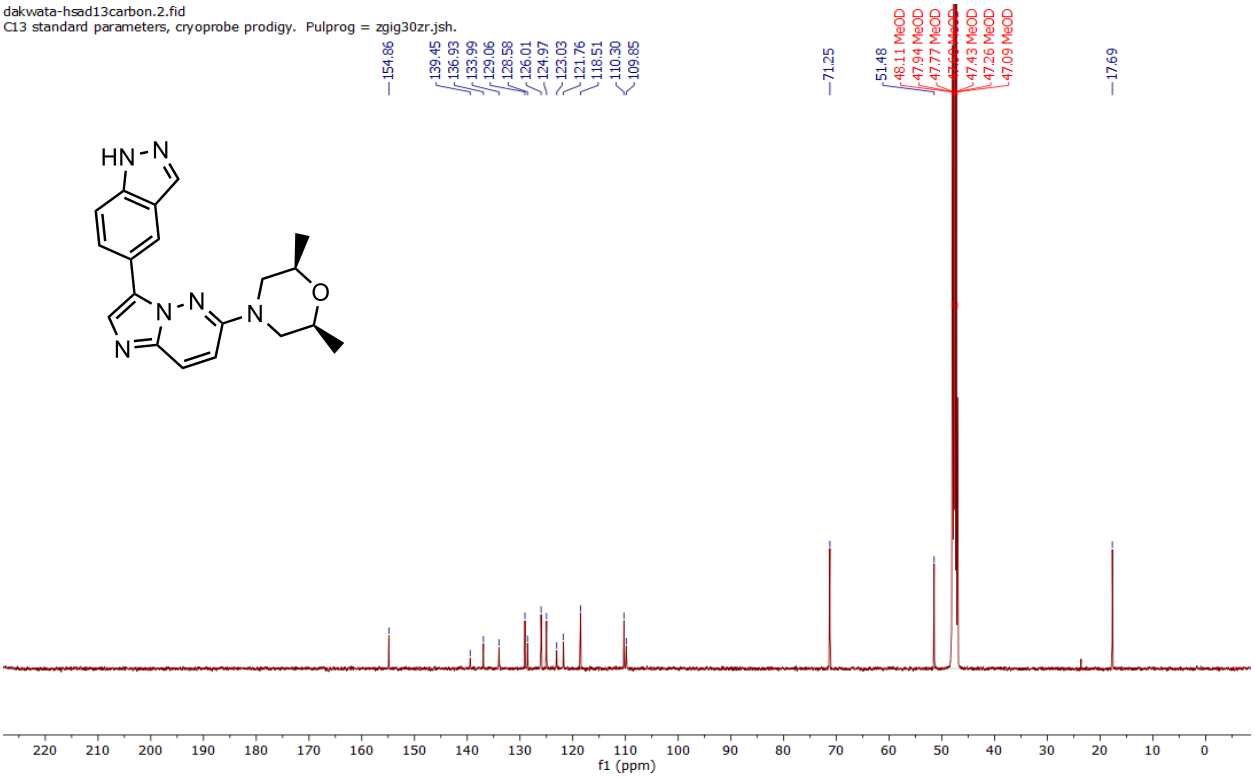
Compound **19**


dakwata-hsad93.1.fid
H1 standard parameters, cryoprobe prodigy.
CN
0.84
0.89
0.83
1.20
2.15
1.01
2.00
2.00
1.91
6.18
f1 (ppm)

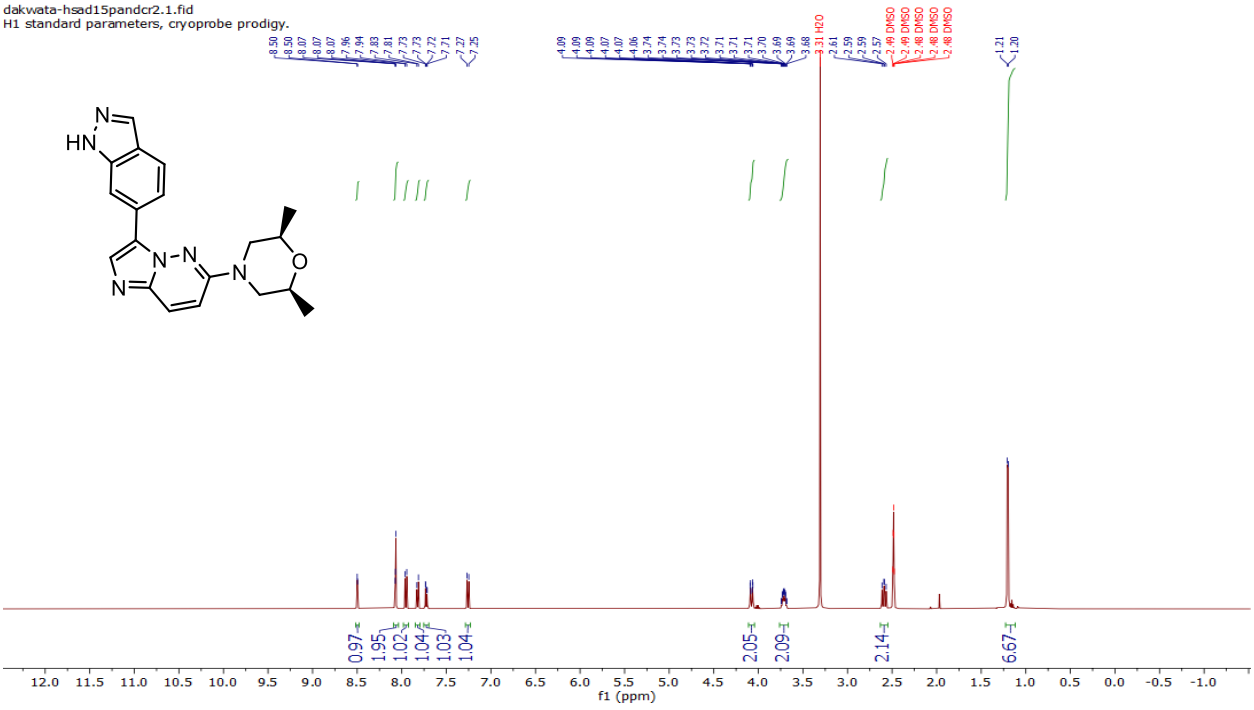

dakwata-hsad93pandc.2.fid
C13 standard parameters, cryoprobe prodigy. Pulprog = zgpg30zr.jsq
CN
f1 (ppm)

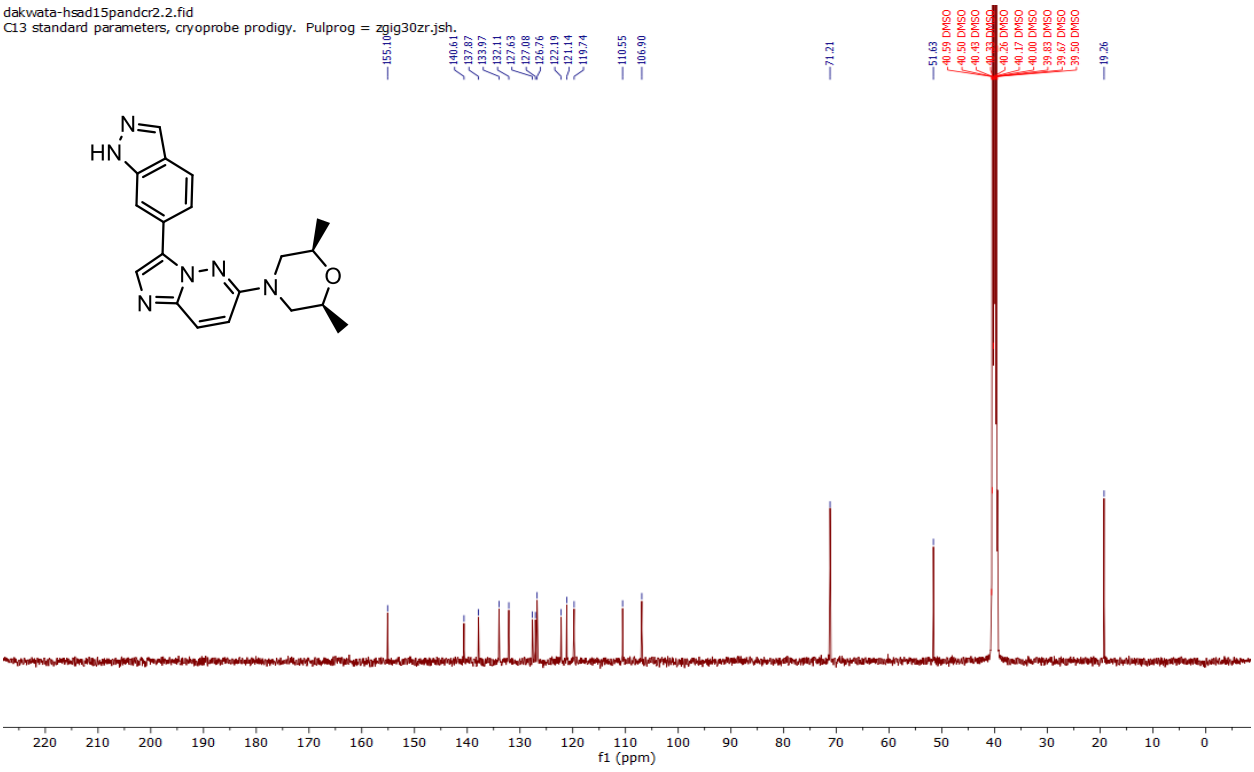
Compound **20**


dakwata-hsad13pinacol.1.fid
H1 standard parameters, cryoprobe prodigy.
HN-N
N-N
N
O
N
4.85 HDO
3.31 MeOD
3.31 MeOD
3.30 MeOD
0.95
0.90
0.99
0.96
0.98
0.97
1.00
2.00
2.02
2.00
6.23
f1 (ppm)


dakwata-hsad13carbon.2.fid
C13 standard parameters, cryoprobe prodigy. Pulprog = zgig30zr.jsh.
HN-N
N-N
N
O
N
154.86
139.45
136.93
133.99
129.06
128.58
126.01
124.97
123.03
121.76
118.51
110.30
109.85
71.25
51.48
48.11 MeOD
47.94 MeOD
47.77 MeOD
47.60 MeOD
47.43 MeOD
47.26 MeOD
47.09 MeOD
17.69
f1 (ppm)

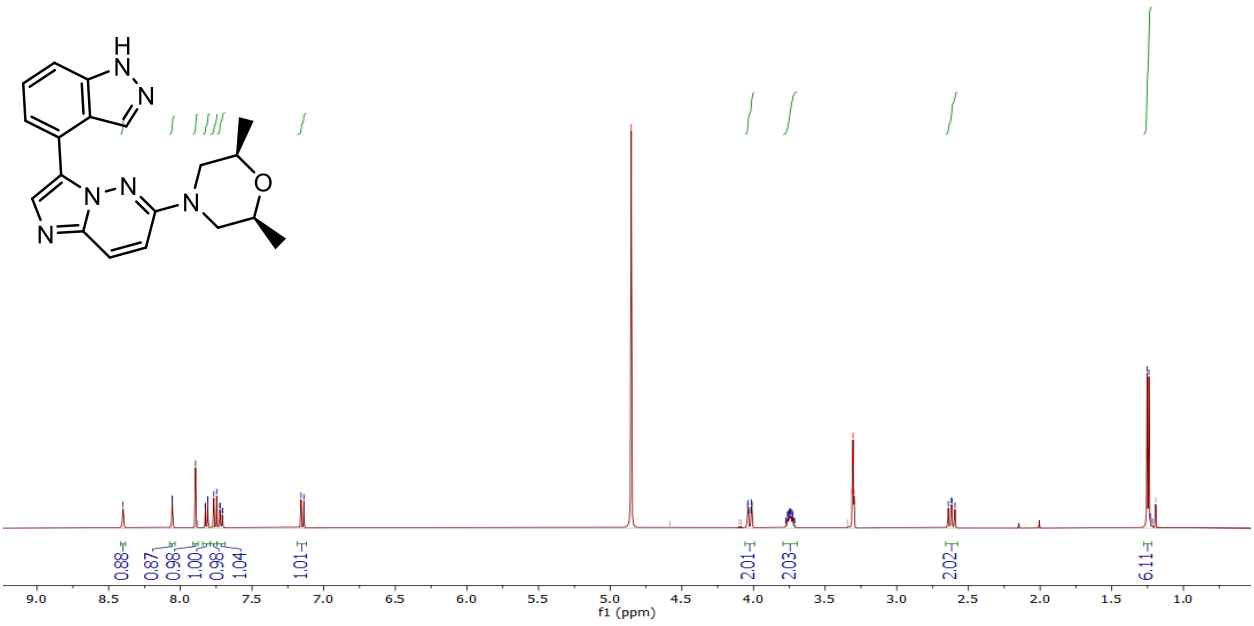
Compound **21**


dakwata-hsad15pandcr2.1.fid
H1 standard parameters, cryoprobe prodigy.
f1 (ppm)

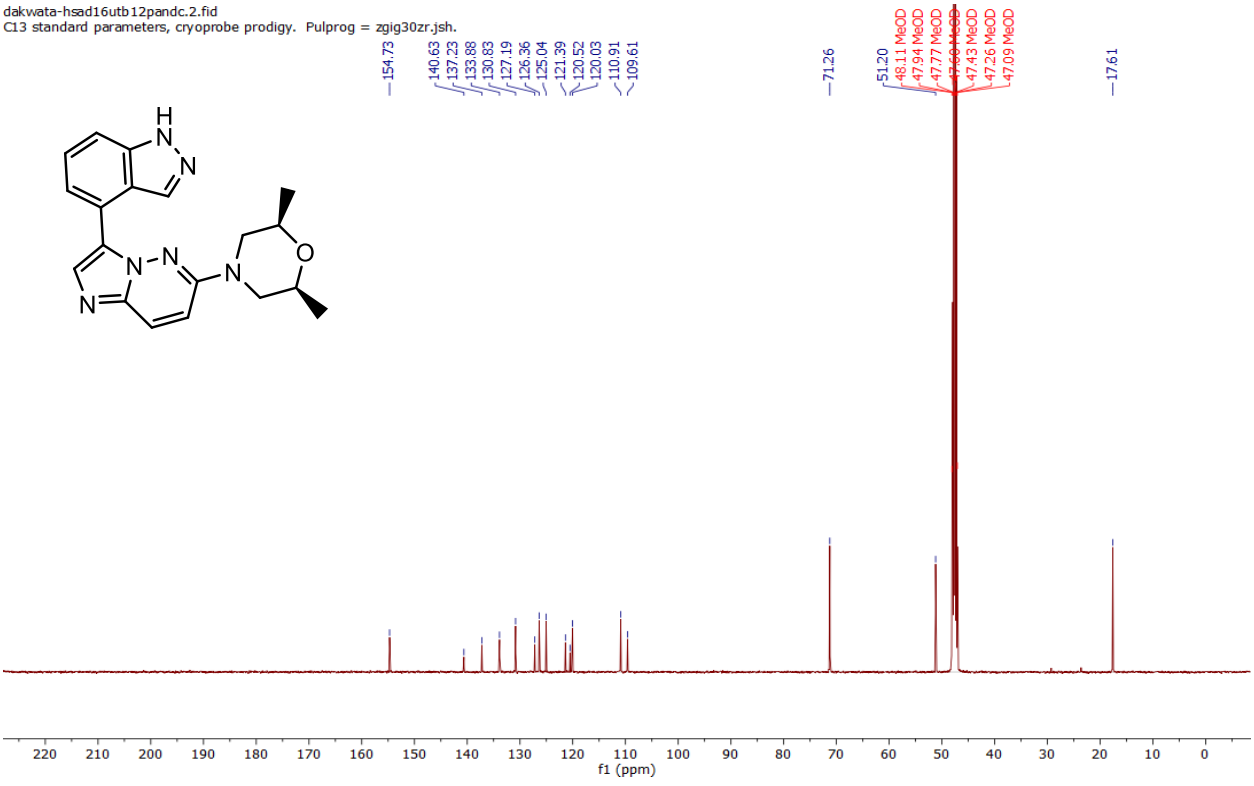

dakwata-hsad15pandcr2.2.fid
C13 standard parameters, cryoprobe prodigy. Pulprog = zgig30zr.jsh.
f1 (ppm)

Compound **22**

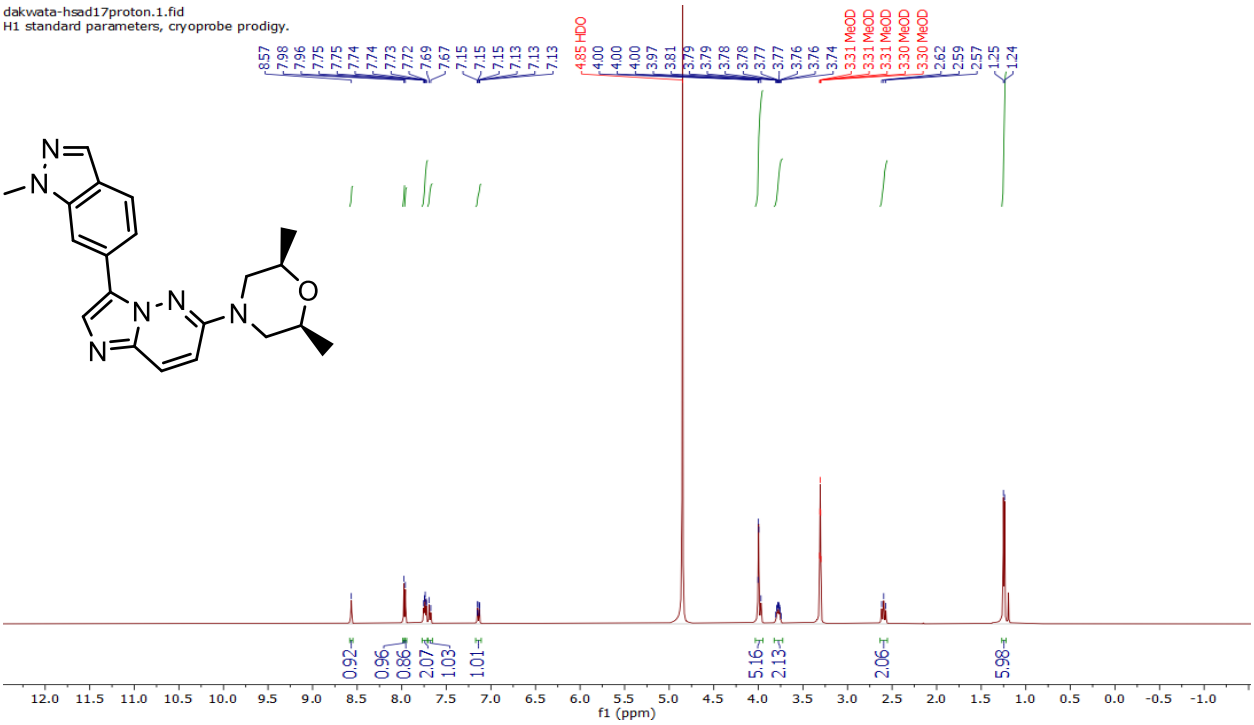
dakwata-hsad15pandc.1.fid
H1 standard parameters, cryoprobe prodigy.

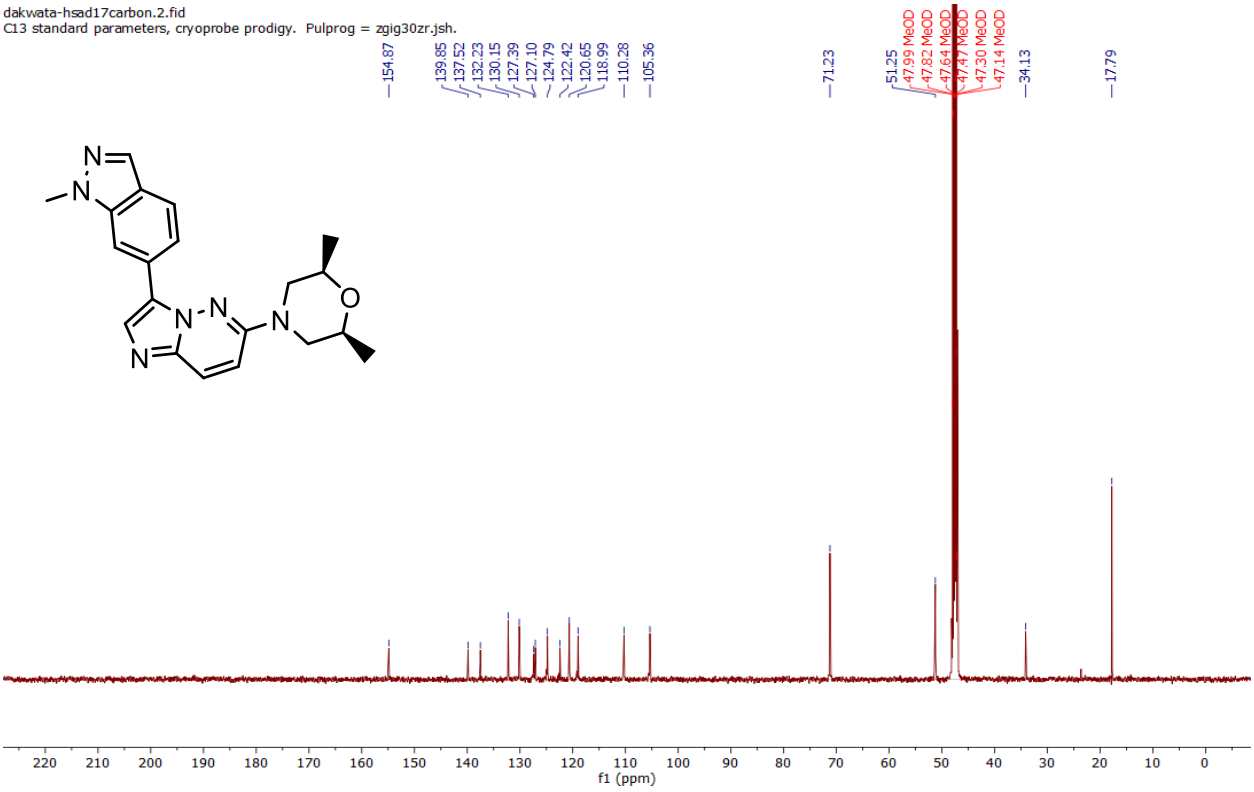


dakwata-hsad16utb12pandc.2.fid
C13 standard parameters, cryoprobe prodigy. Pulprog = zgig30zr.jsh.

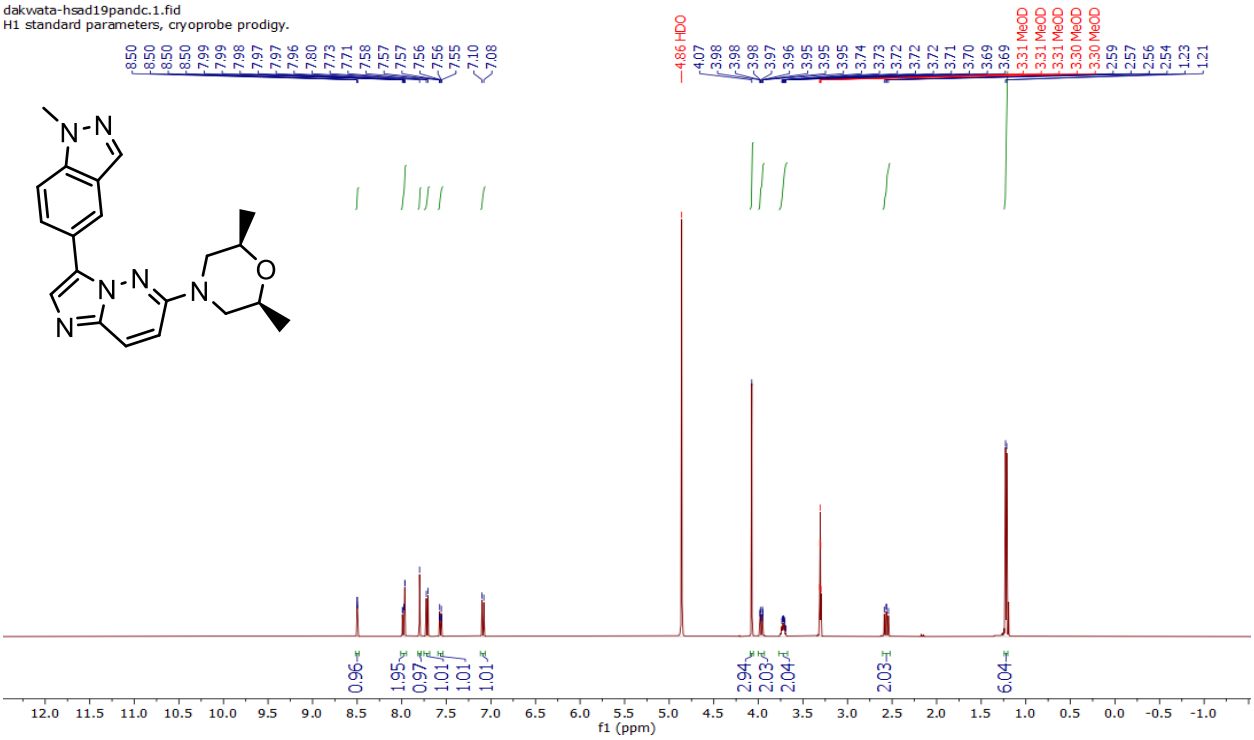


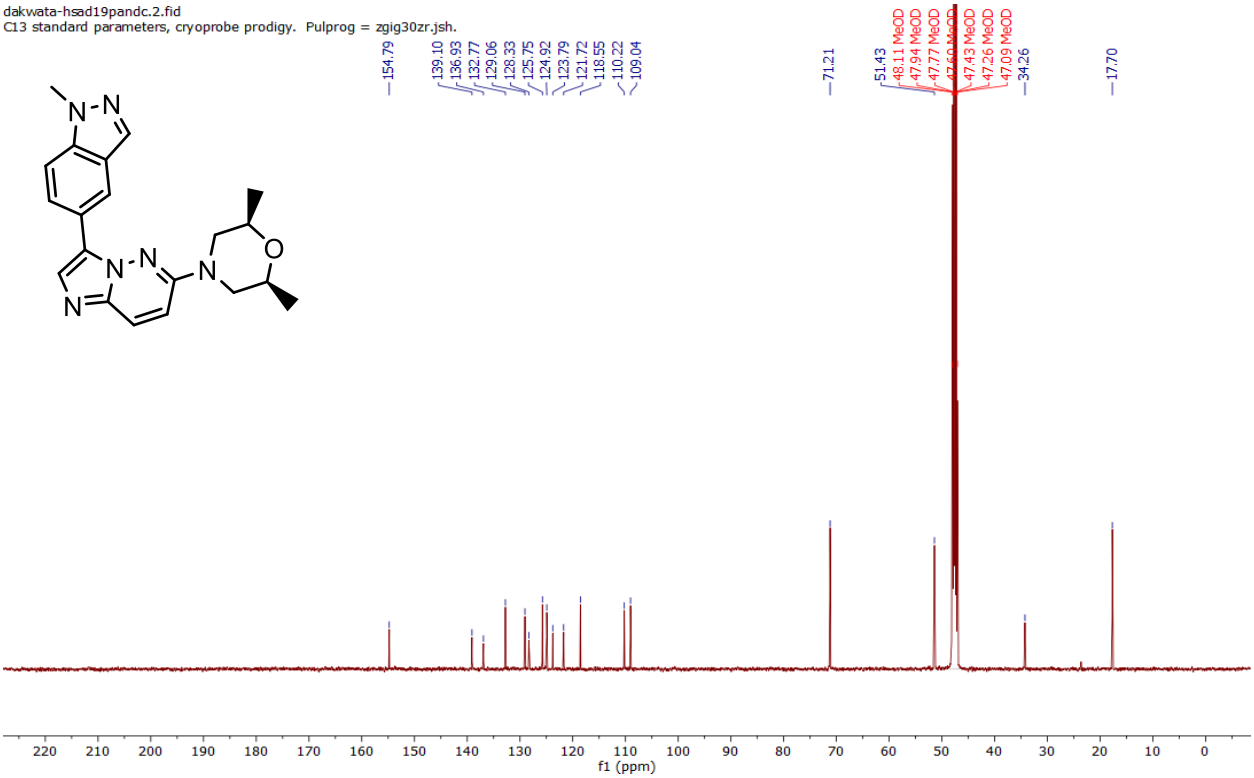
Compound **23**


dakwata-hsad17proton.1.fid
H1 standard parameters, cryoprobe prodigy.
f1 (ppm)

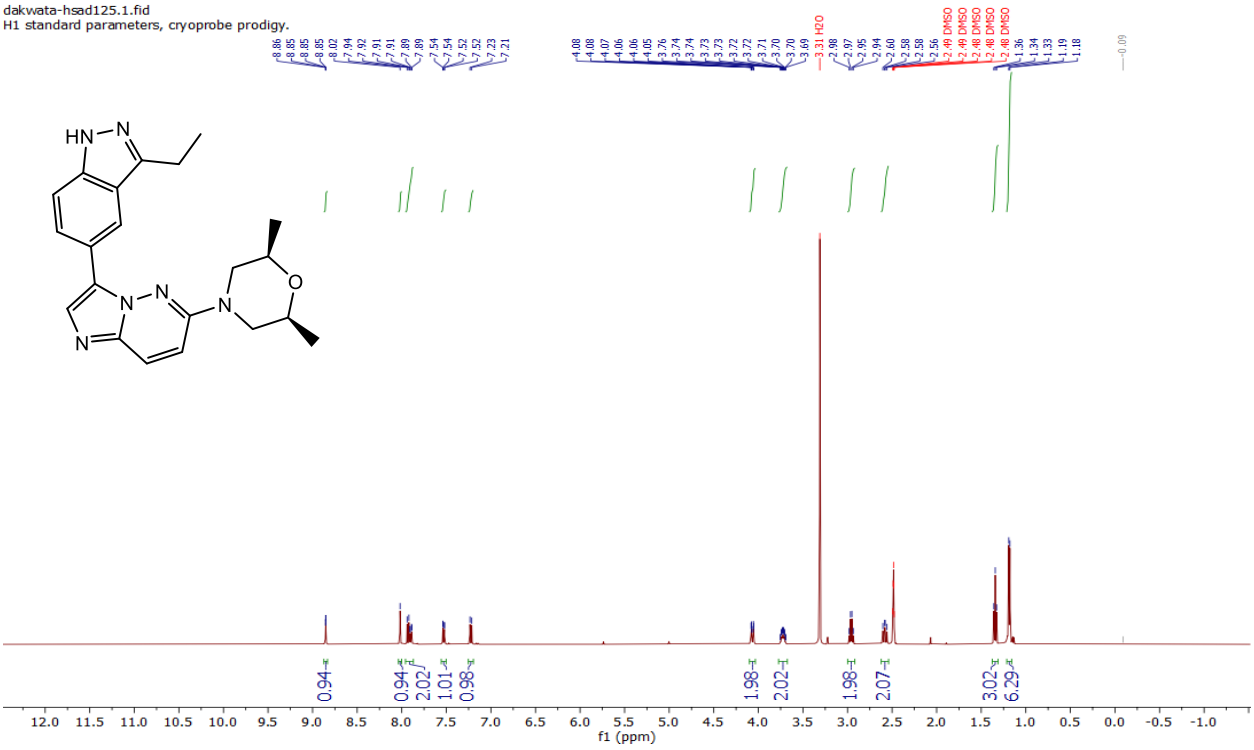

dakwata-hsad17carbon.2.fid
C13 standard parameters, cryoprobe prodigy. Pulprog = zgig30zr.jsh.
f1 (ppm)

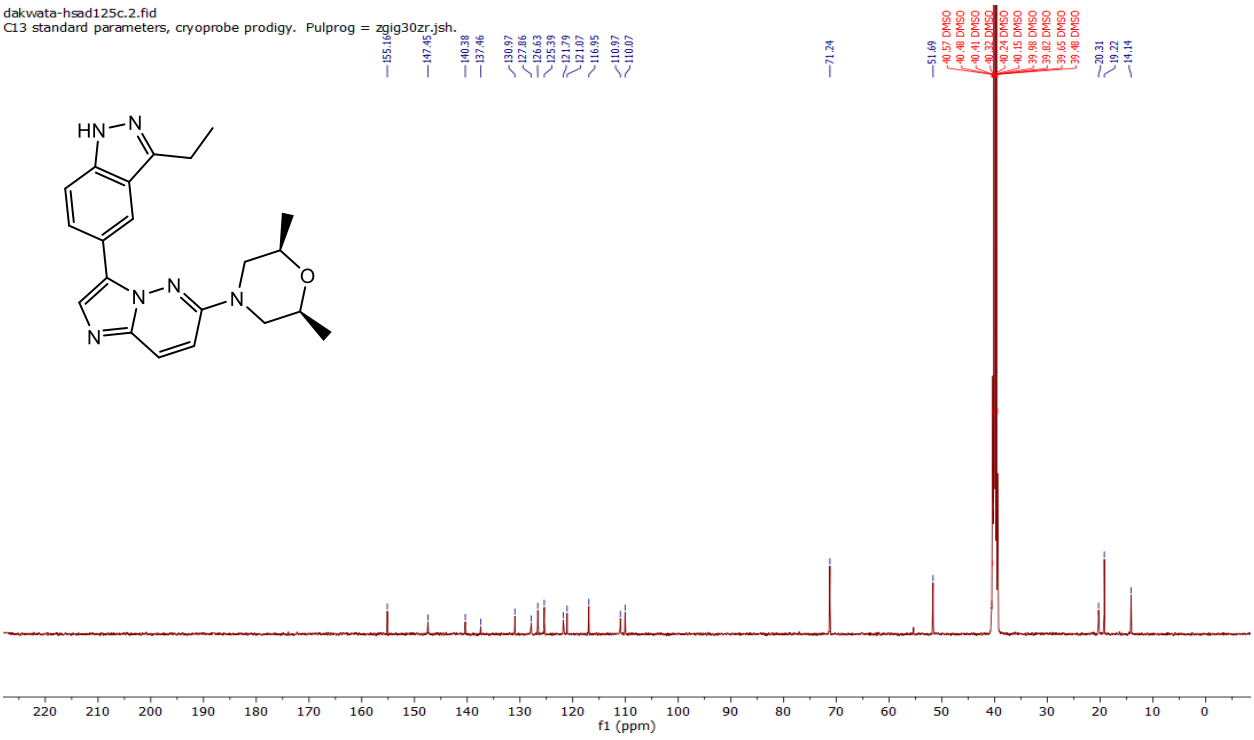
Compound **24**


dakwata-hsad19pandc.1.fid
H1 standard parameters, cryoprobe prodigy.
f1 (ppm)

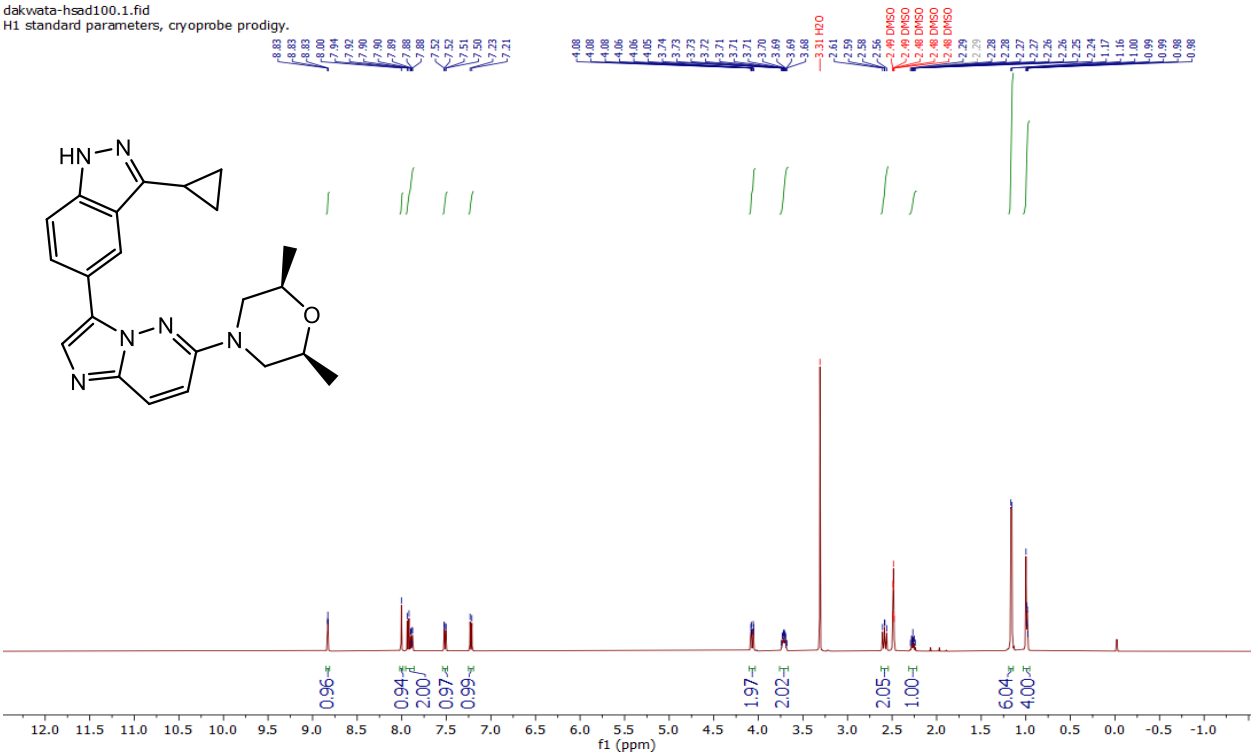

dakwata-hsad19pandc.2.fid
C13 standard parameters, cryoprobe prodigy. Pulprog = zgig30zr.jsh.
f1 (ppm)

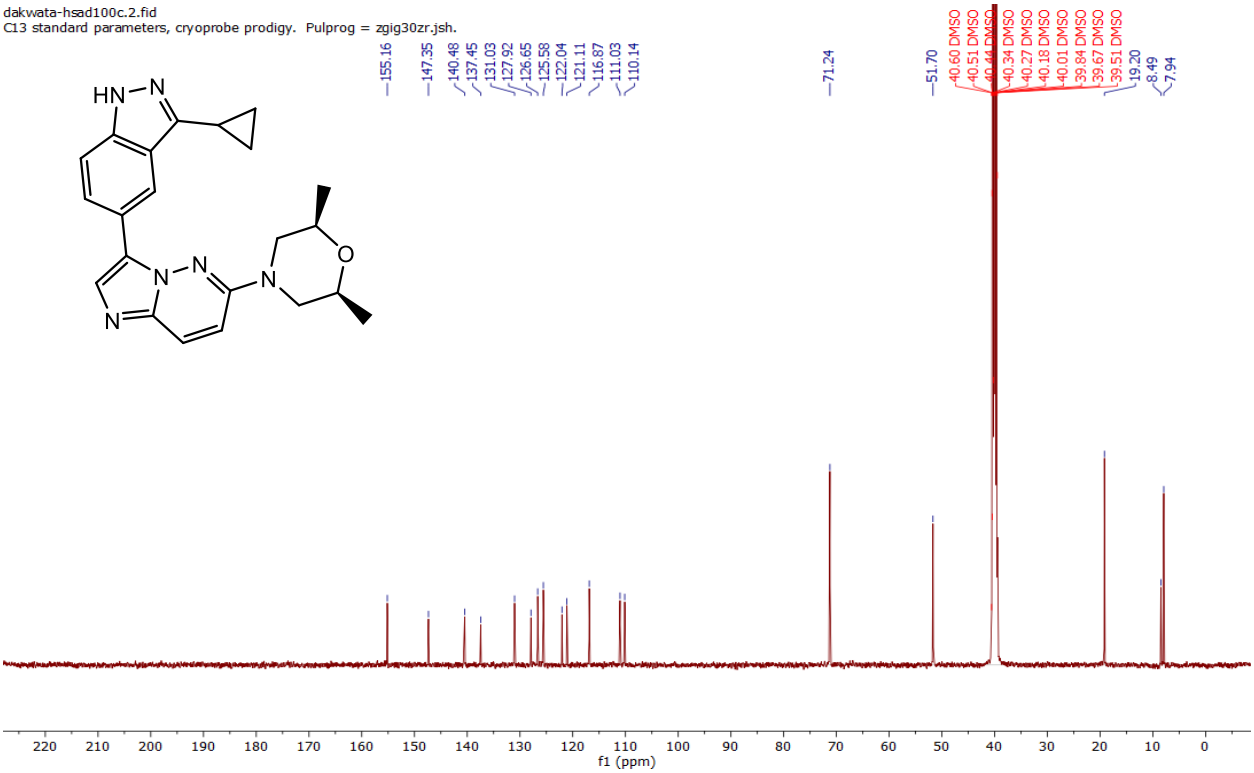
Compound **25**


dakwata-hsad125.1.fid
H1 standard parameters, cryoprobe prodigy.
f1 (ppm)

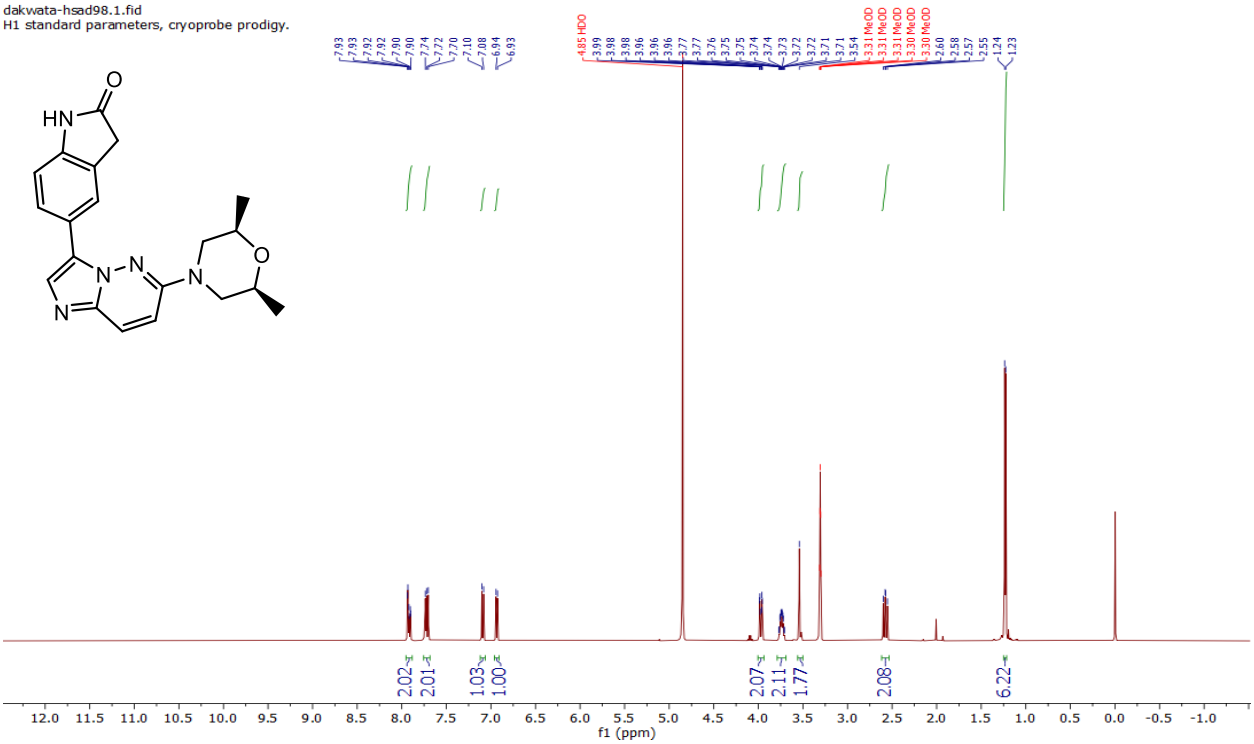

dakwata-hsad125c.2.fid
C13 standard parameters, cryoprobe prodigy. Pulprog = zgig30zr.jsh.
f1 (ppm)

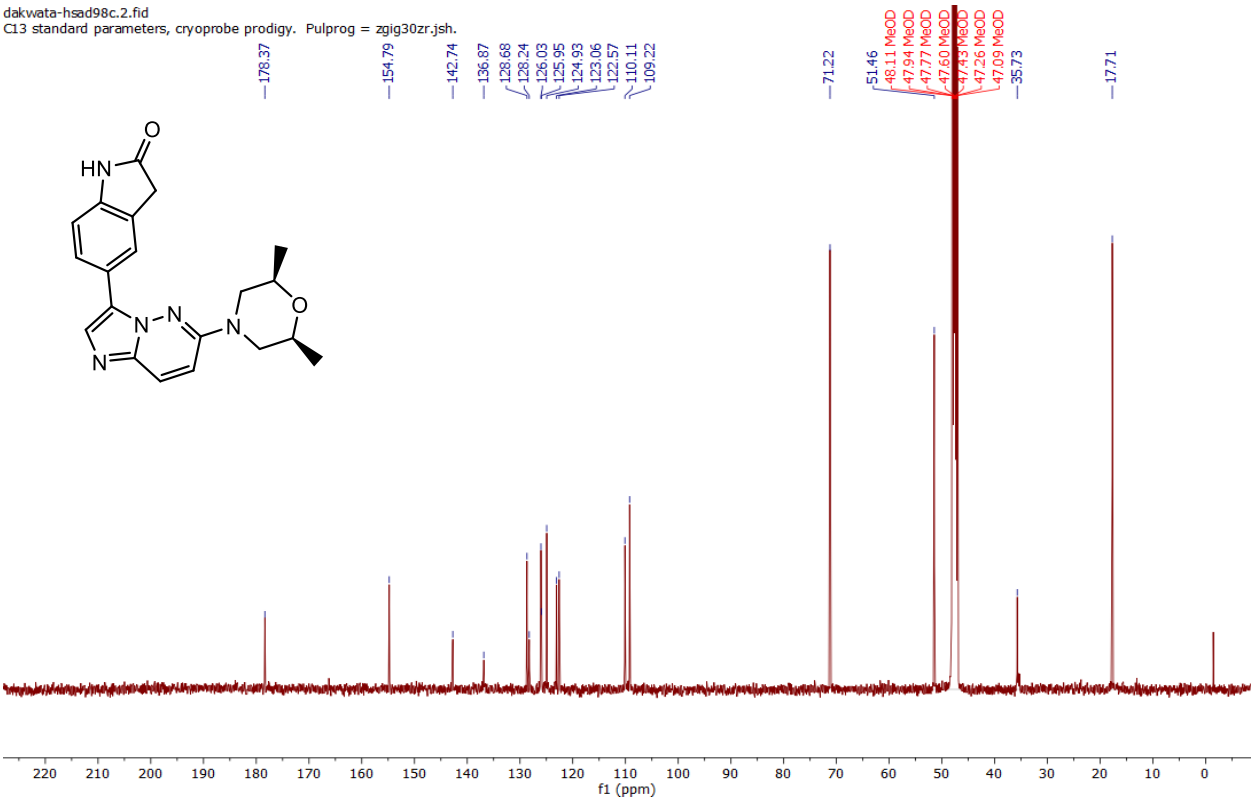
Compound **26**


dakwata-hsad100.1.fid
H1 standard parameters, cryoprobe prodigy.
HN
N
N
N
N
O
0.96
0.94
2.00
0.97
0.99
1.97
2.02
2.05
1.00
6.04
4.00
f1 (ppm)

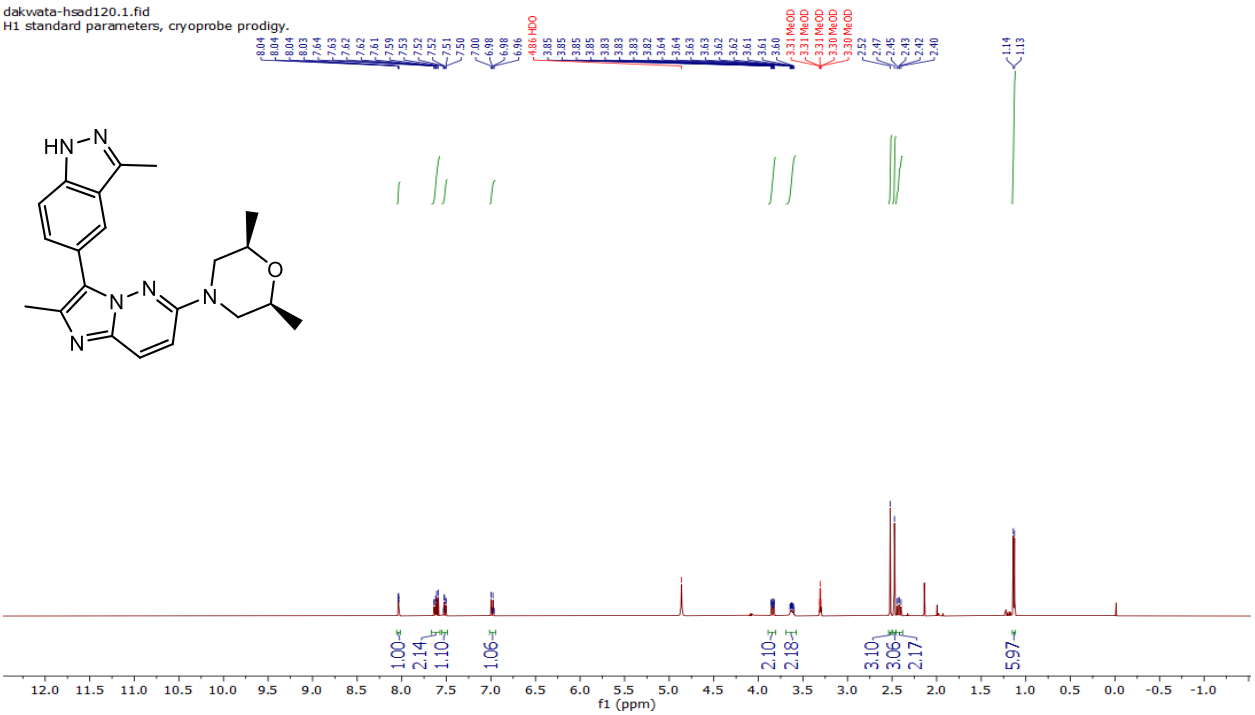

dakwata-hsad100c.2.fid
C13 standard parameters, cryoprobe prodigy. Pulprog = zgig30zr.jsh.
HN
N
N
N
N
O
155.16
147.35
140.48
137.45
131.03
127.92
126.65
125.58
122.04
121.11
116.87
111.03
110.14
71.24
51.70
40.60 DMSO
40.51 DMSO
40.44 DMSO
40.34 DMSO
40.27 DMSO
40.18 DMSO
40.01 DMSO
39.84 DMSO
39.67 DMSO
39.51 DMSO
19.20
8.49
7.94
f1 (ppm)

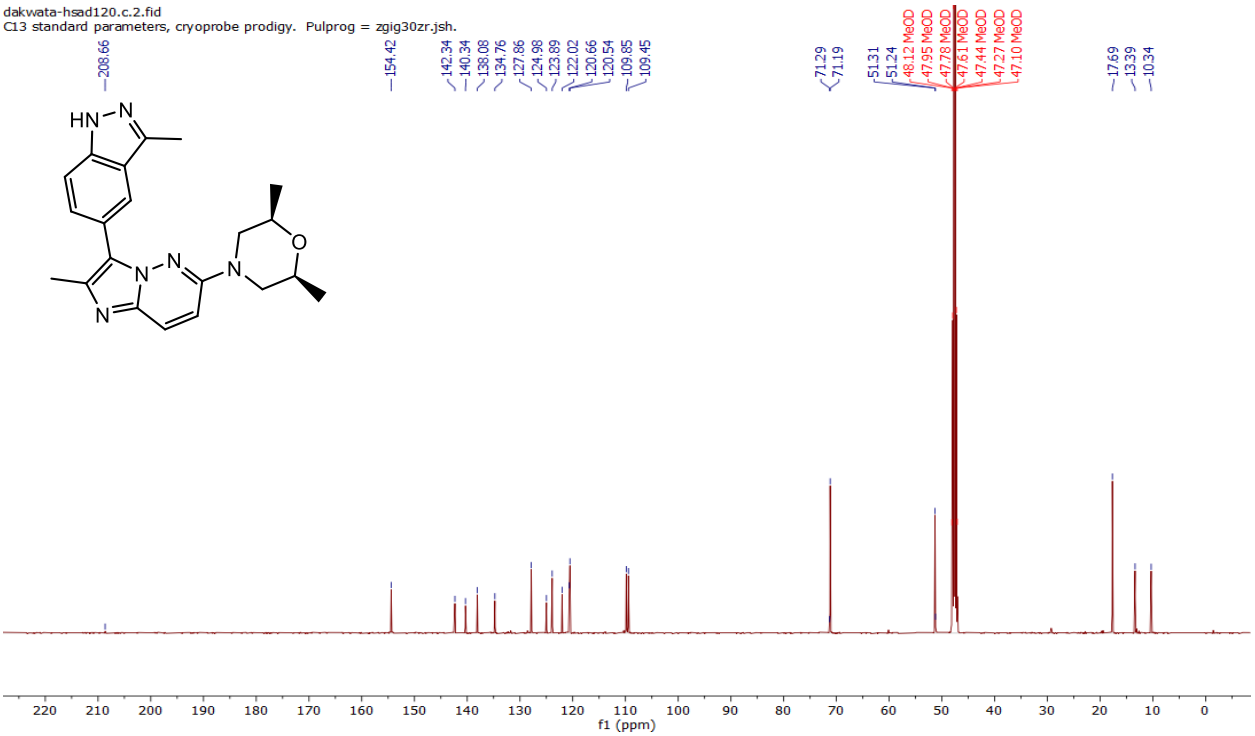
Compound **27**


dakwata-hsad98.1.fid
H1 standard parameters, cryoprobe prodigy.
HN
O
N
N
N
O
f1 (ppm)

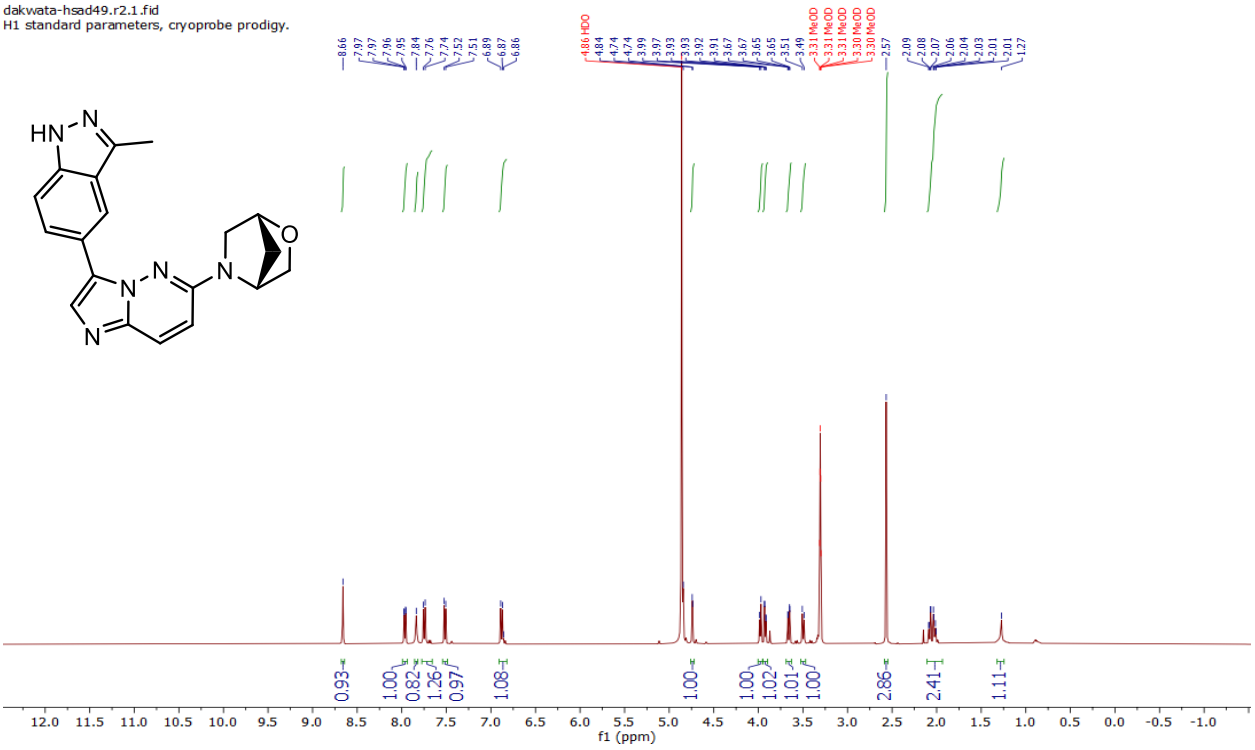

dakwata-hsad98c.2.fid
C13 standard parameters, cryoprobe prodigy. Pulprog = zgig30zr.jsh.
HN
O
N
N
N
O
f1 (ppm)

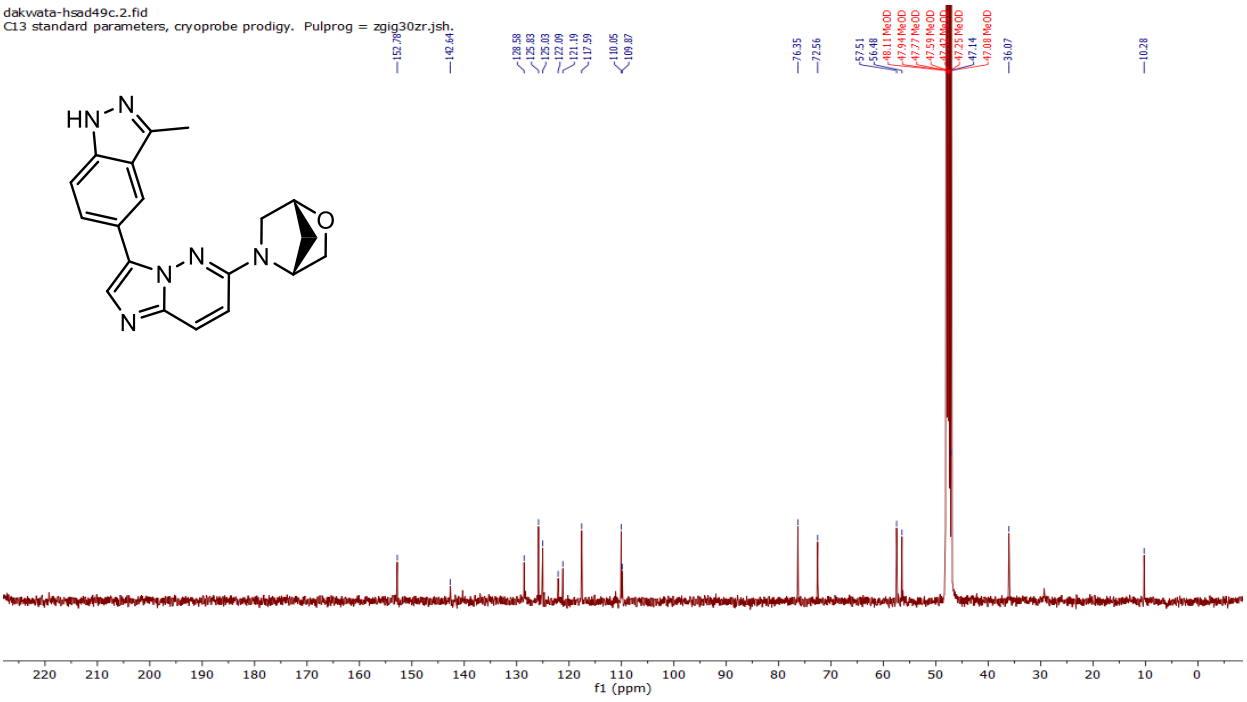
Compound **28**


dakwata-hsad120.1.fid
H1 standard parameters, cryoprobe prodigy.
f1 (ppm)

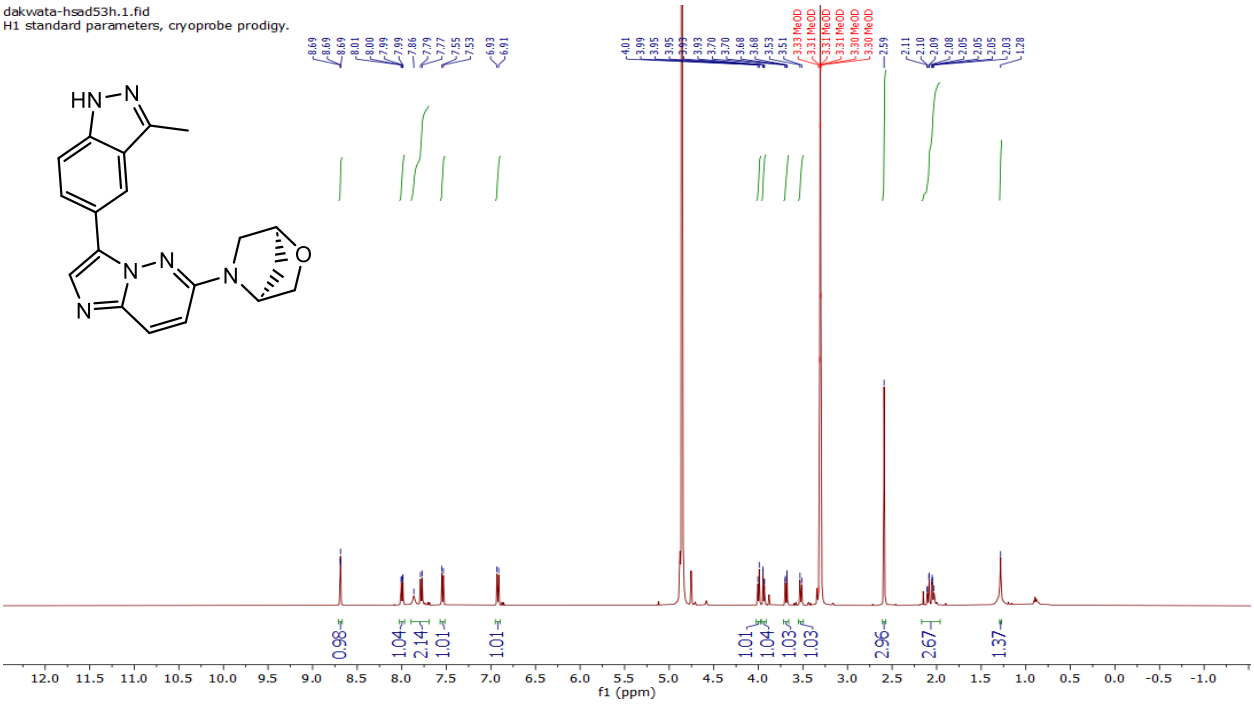

dakwata-hsad120.c.2.fid
C13 standard parameters, cryoprobe prodigy. Pulprog = zgig30zr.jsh.
f1 (ppm)

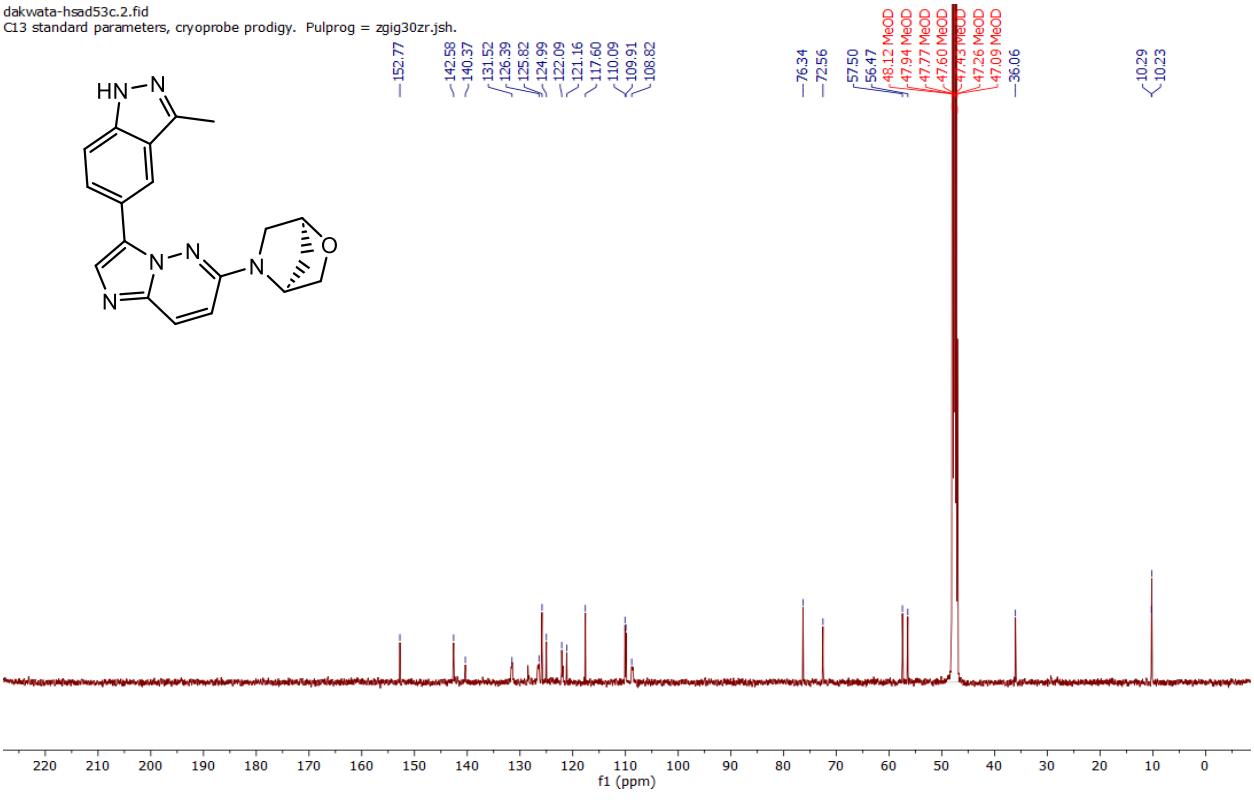
Compound **29**


dakwata-hsad49.r2.1.fid
H1 standard parameters, cryoprobe prodigy.
f1 (ppm)

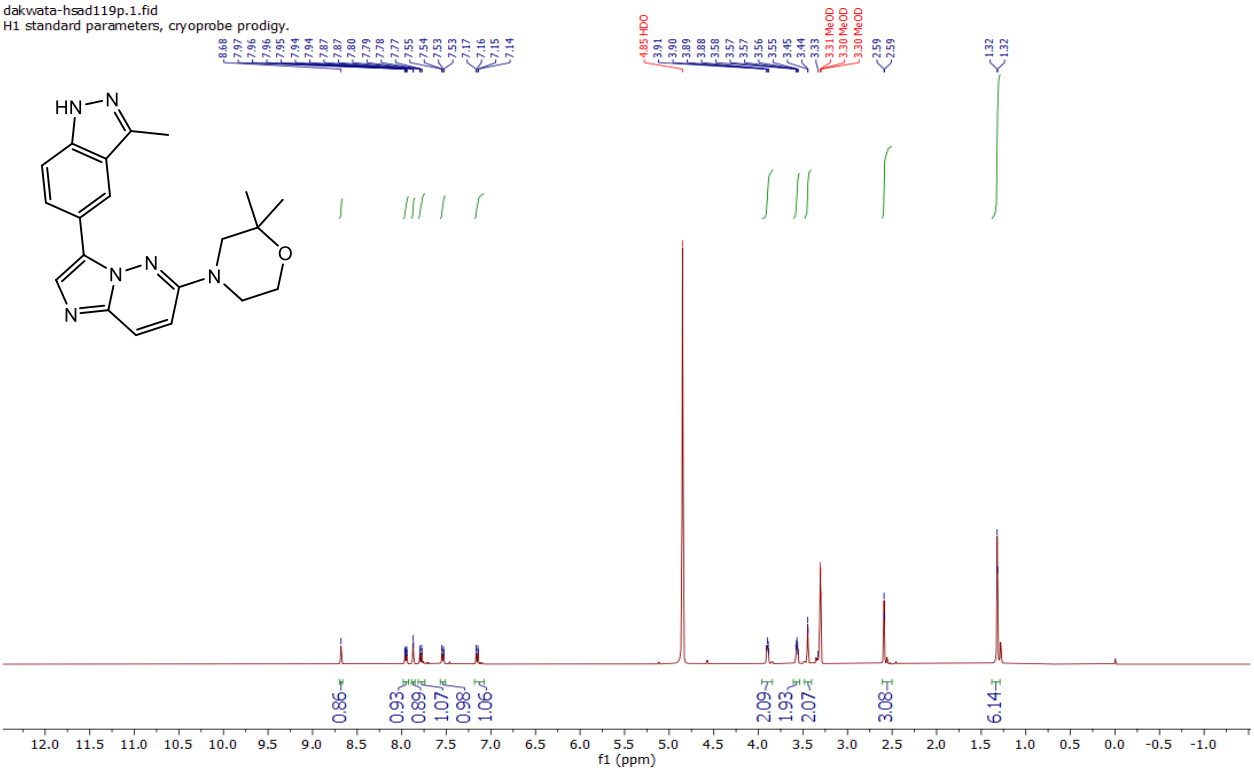

dakwata-hsad49c.2.fid
C13 standard parameters, cryoprobe prodigy. Pulprog = zgig30zr.jsh.
f1 (ppm)

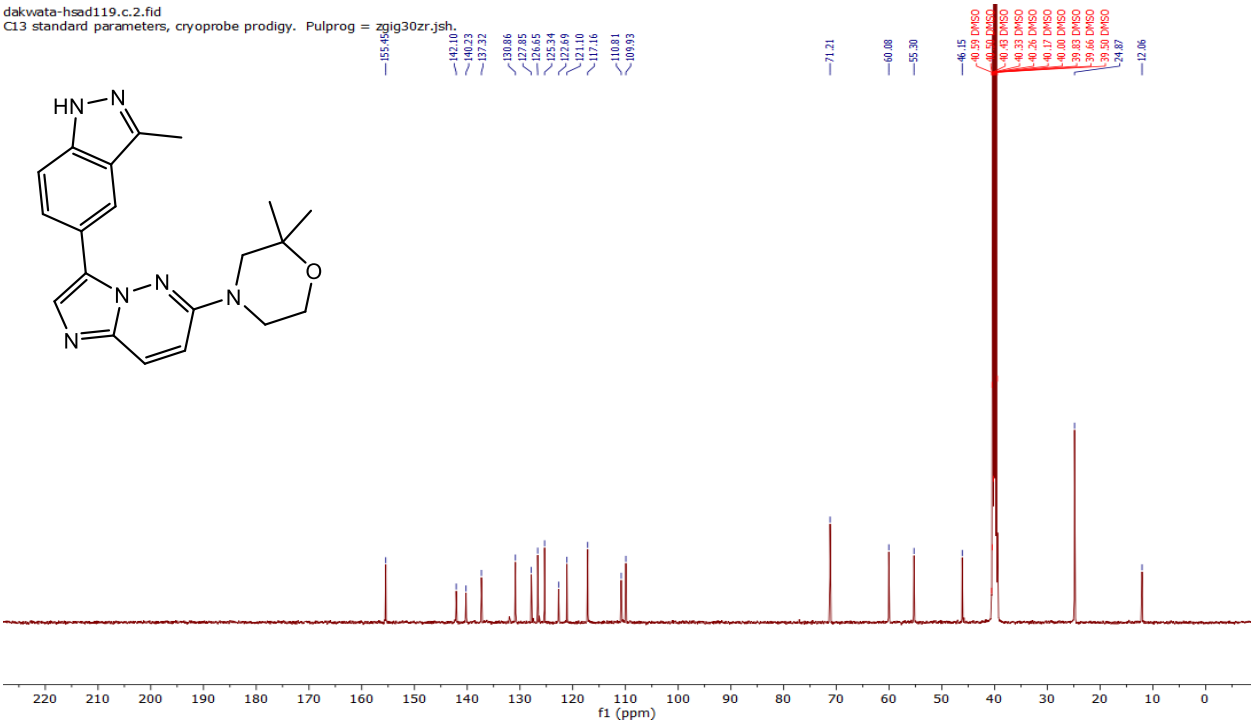
Compound **30**


dakwata-hsad53h.1.fid
H1 standard parameters, cryoprobe prodigy.
f1 (ppm)

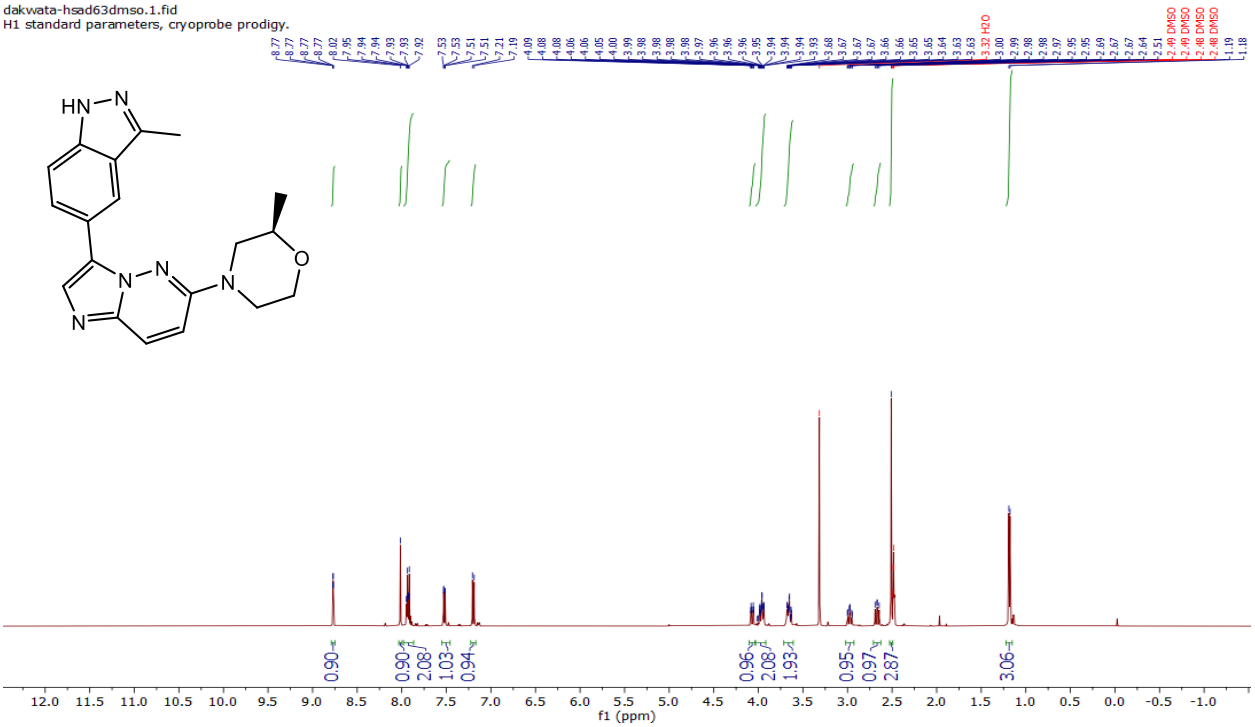

dakwata-hsad53c.2.fid
C13 standard parameters, cryoprobe prodigy. Pulprog = zgig30zr.jsh.
f1 (ppm)

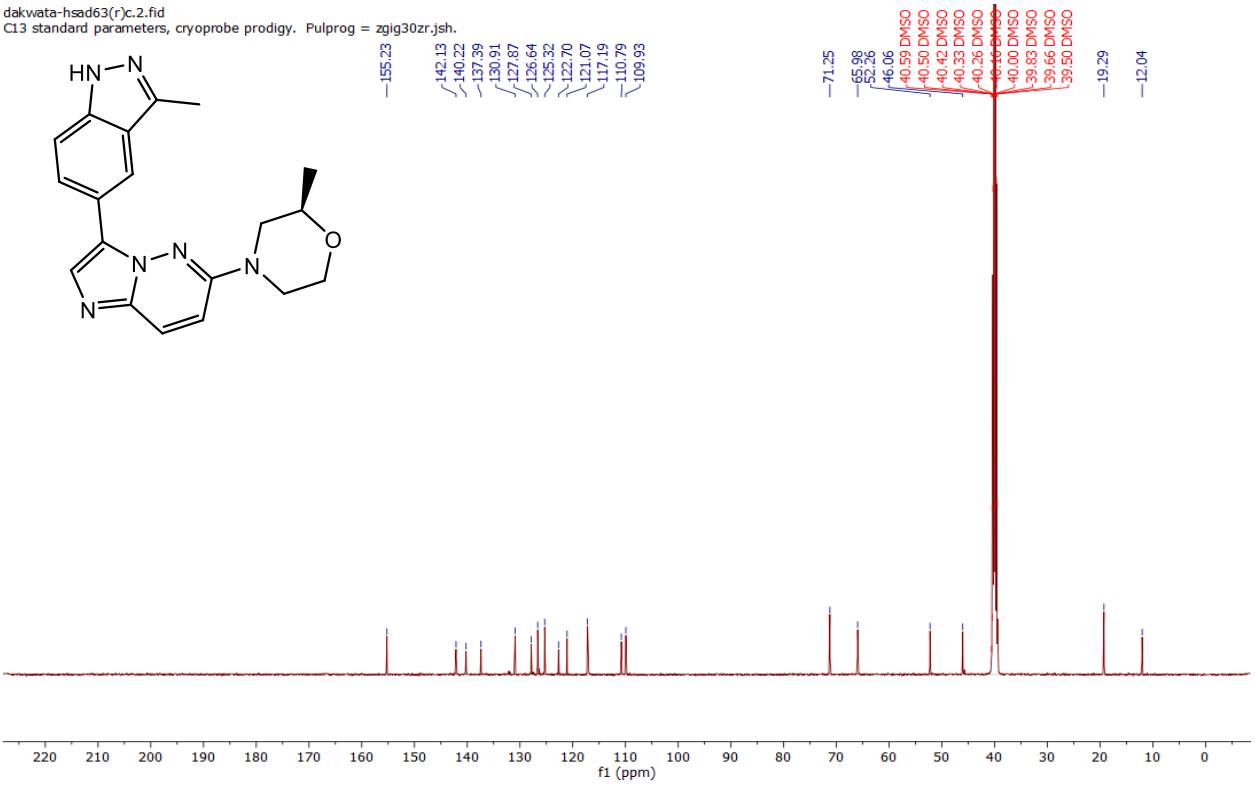
Compound **31**


dakwata-hsad119p.1.fid
H1 standard parameters, cryoprobe prodigy.
f1 (ppm)

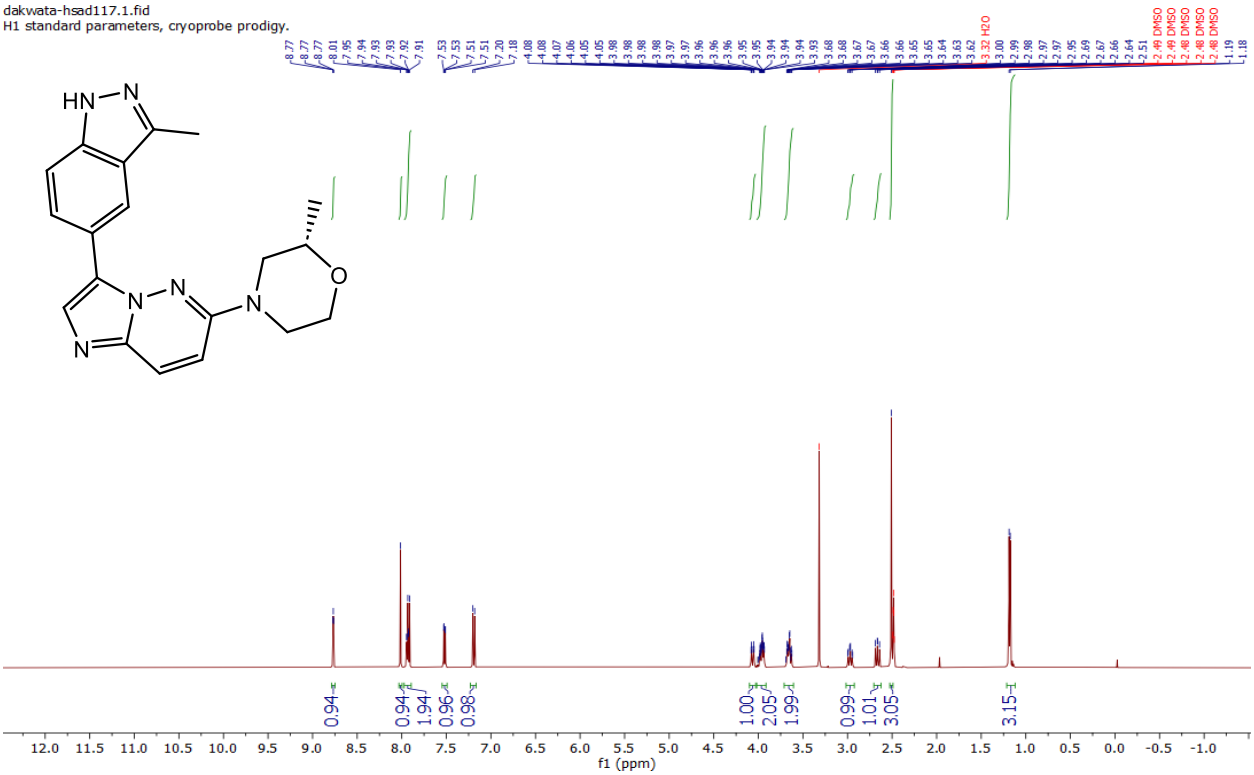

dakwata-hsad119.c.2.fid
C13 standard parameters, cryoprobe prodigy. Pulprog = zgig30zr.jsh.
f1 (ppm)

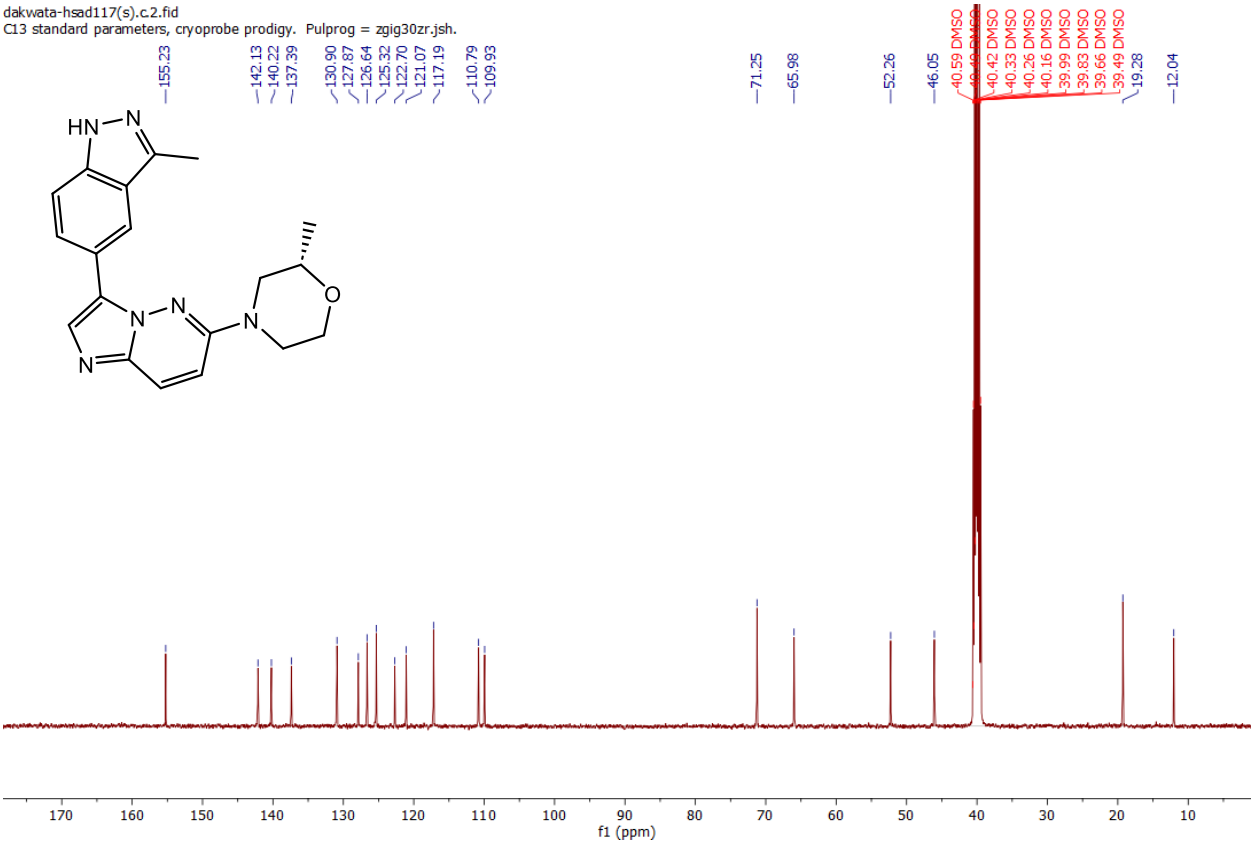
Compound **32**


dakwata-hsad63dmso.1.fid
H1 standard parameters, cryoprobe prodigy.
f1 (ppm)

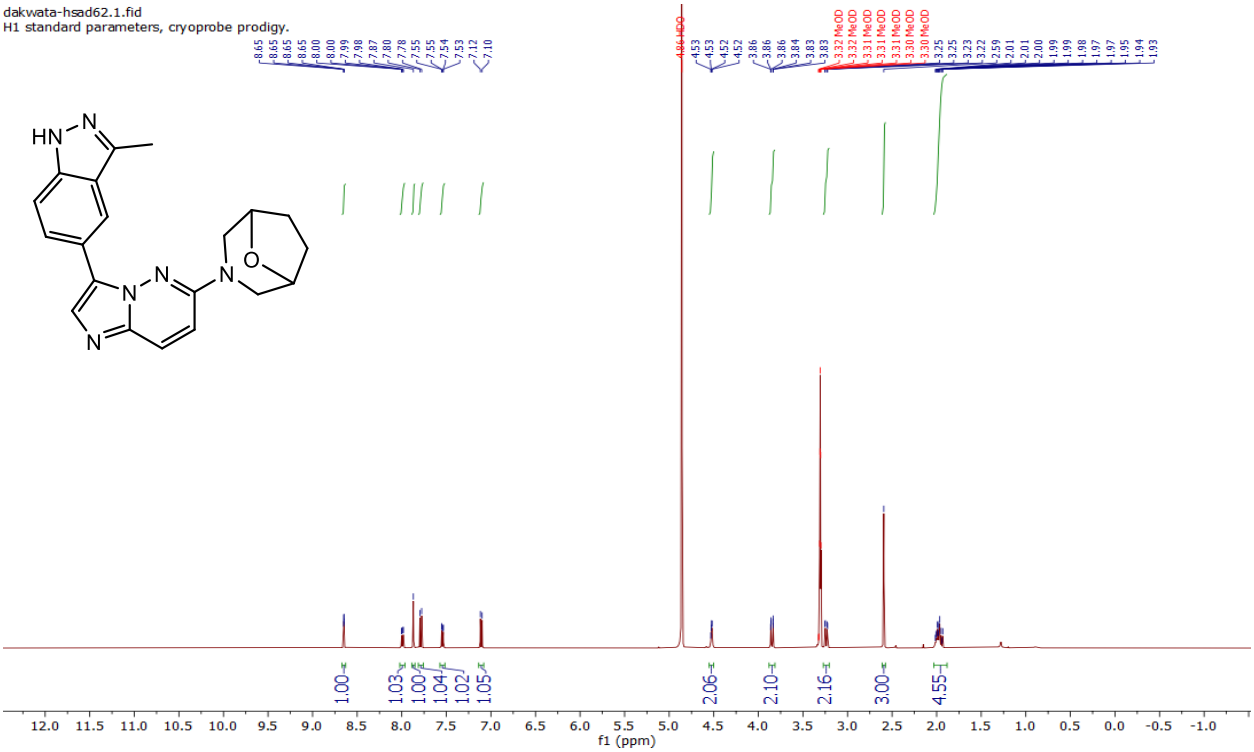

dakwata-hsad63(r)c.2.fid
C13 standard parameters, cryoprobe prodigy. Pulprog = zgig30zr.jsh.
f1 (ppm)

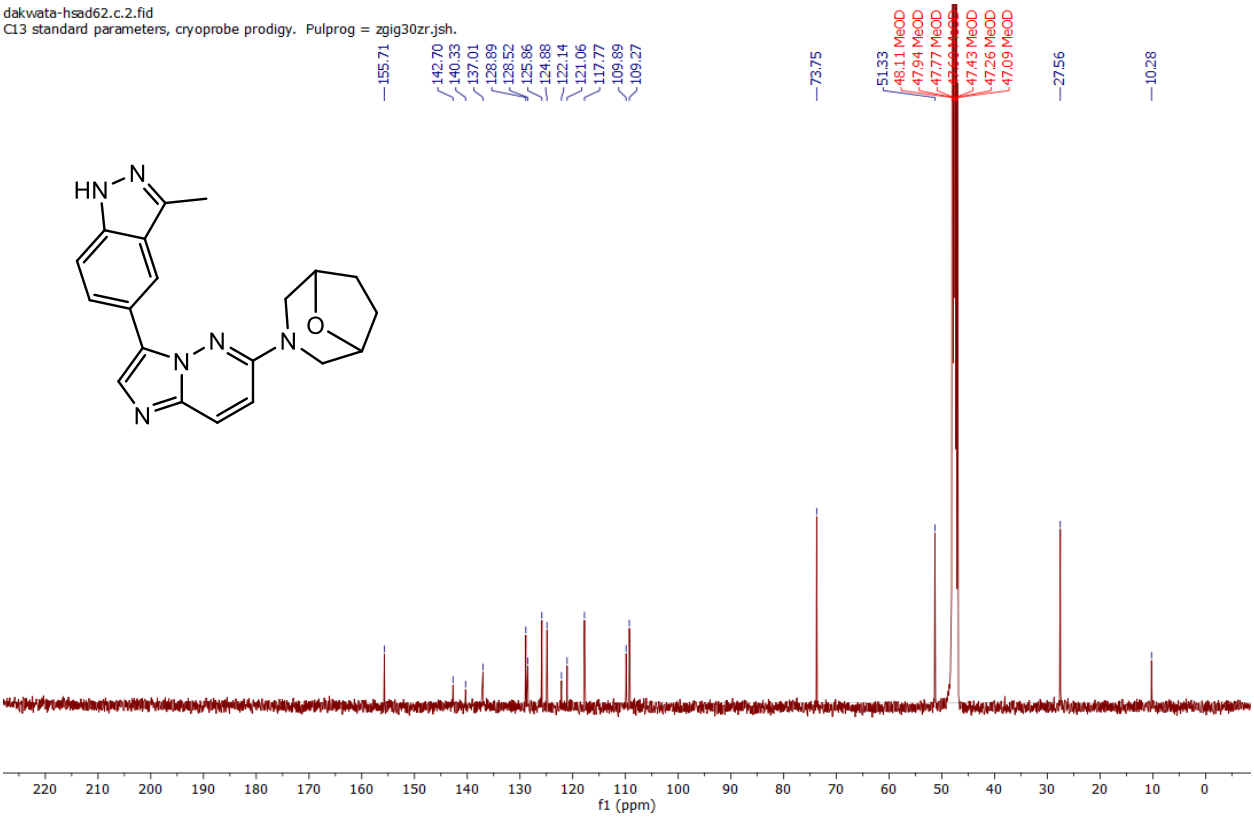
Compound **33**


dakwata-hsad117.1.fid
H1 standard parameters, cryoprobe prodigy.
f1 (ppm)

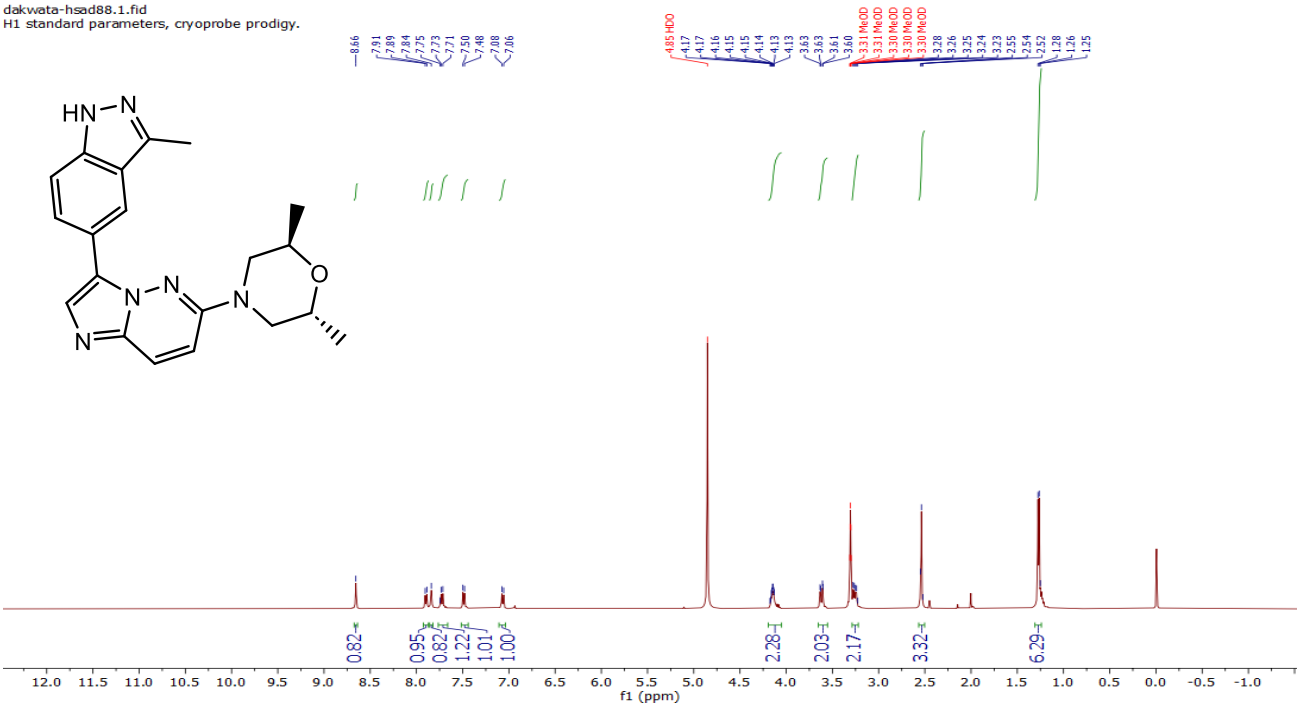

dakwata-hsad117(s).c.2.fid
C13 standard parameters, cryoprobe prodigy. Pulprog = zgig30zr.jsh.
f1 (ppm)

Compound **34**


dakwata-hsad62.1.fid
H1 standard parameters, cryoprobe prodigy.
f1 (ppm)


dakwata-hsad62.c.2.fid
C13 standard parameters, cryoprobe prodigy. Pulprog = zgig30zr.jsh.
f1 (ppm)

Compound **35**


dakwata-hsad88.1.fid
H1 standard parameters, cryoprobe prodigy.
f1 (ppm)

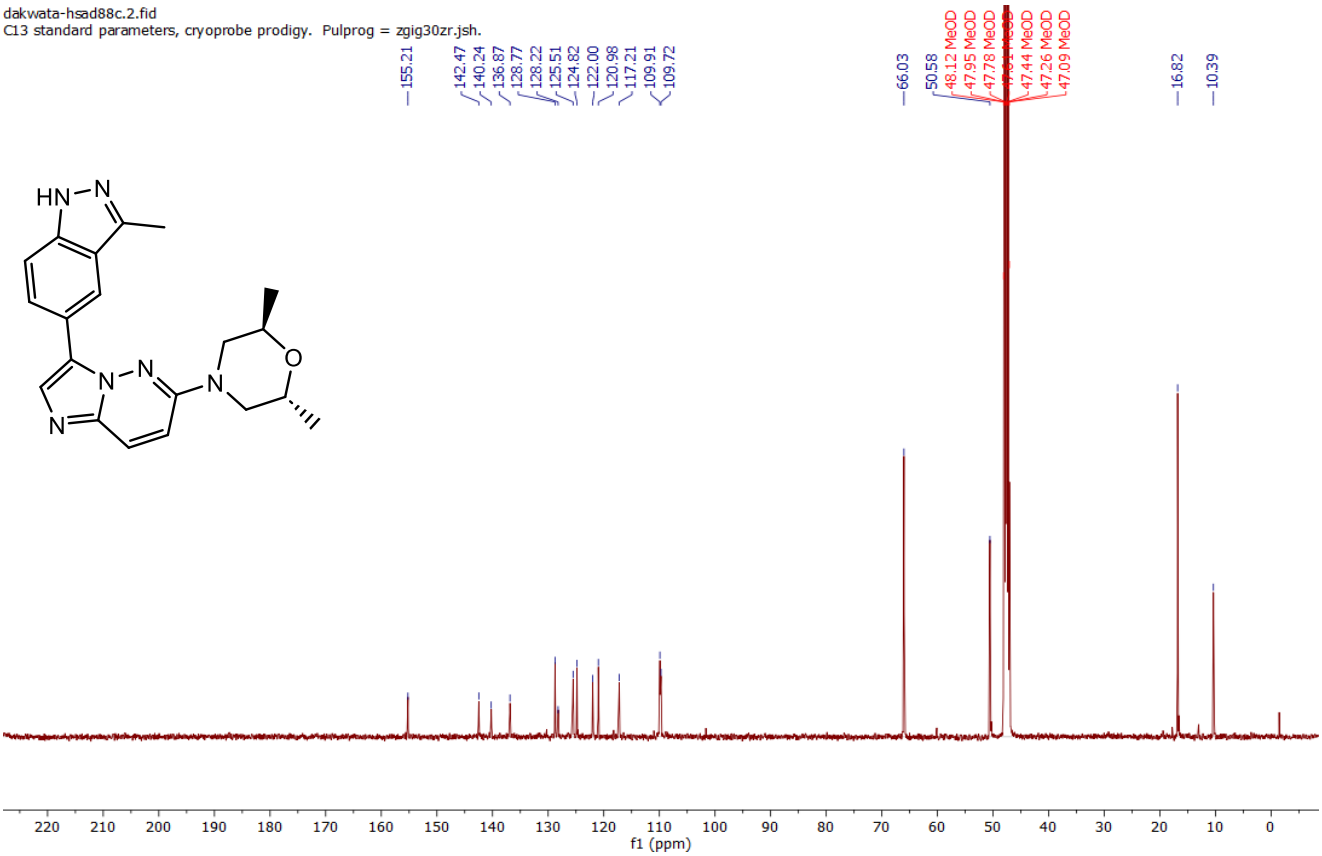

dakwata-hsad88c.2.fid
C13 standard parameters, cryoprobe prodigy. Pulprog = zgig30zr.jsh.
f1 (ppm)

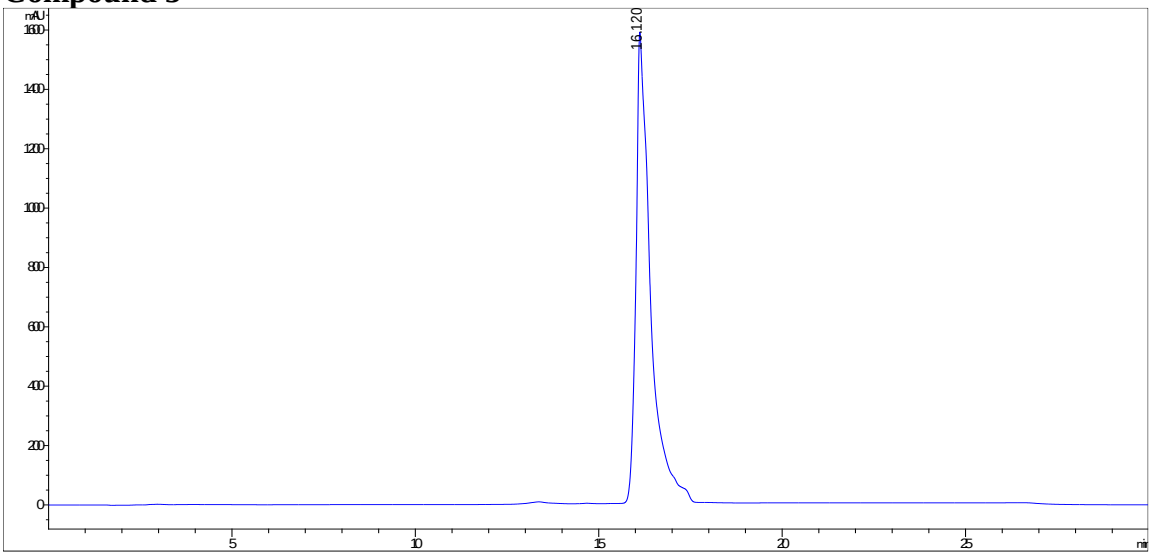
Table S1. HPLC purity

Compound	Retention time (min)	% Purity Area
3	16.1	99
25	17.0	98
26	17.1	97
29	14.8	95
30	15.8	97
31	16.2	99
32	15.8	99
33	15.9	98
34	15.6	98
35	15.3	98

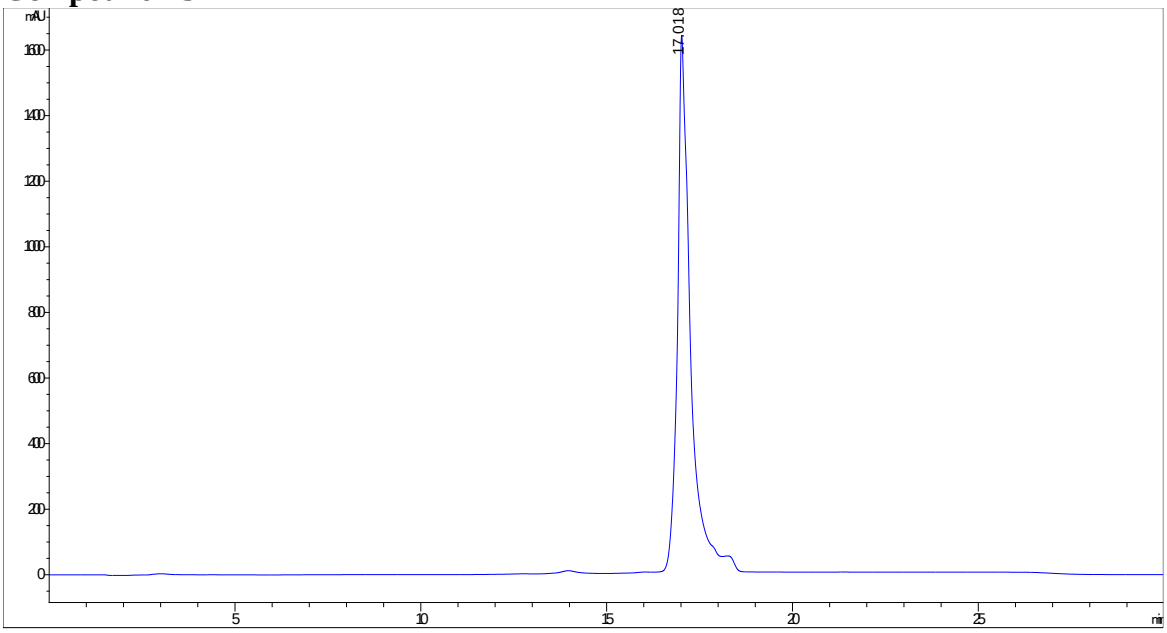
UV detection wavelength 260 nm, Agilent Eclipse instrument; C18 column 5C18-MS-II COSMOSIL (4.6ID × 250 mm); method: 0 → 10 min 50% D , 10 → 20 min 50 to 100% B, 20 →30 min 50% D (C: 0.1% NH4OH in H2O, D: MeOH),.

HPLC Chromatogram

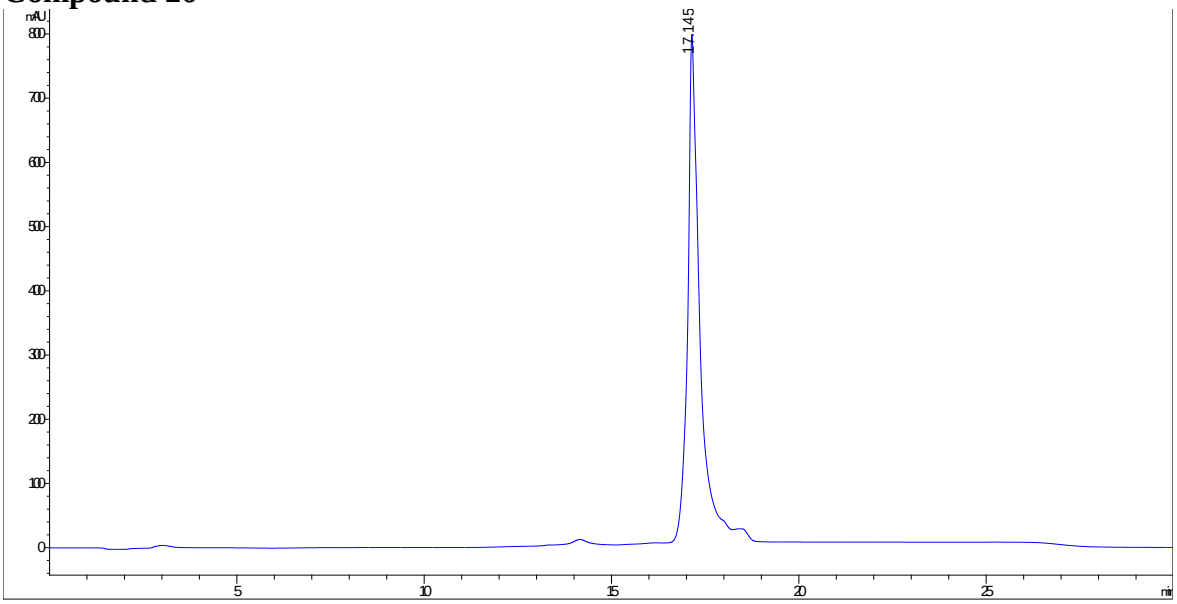
Compound 3



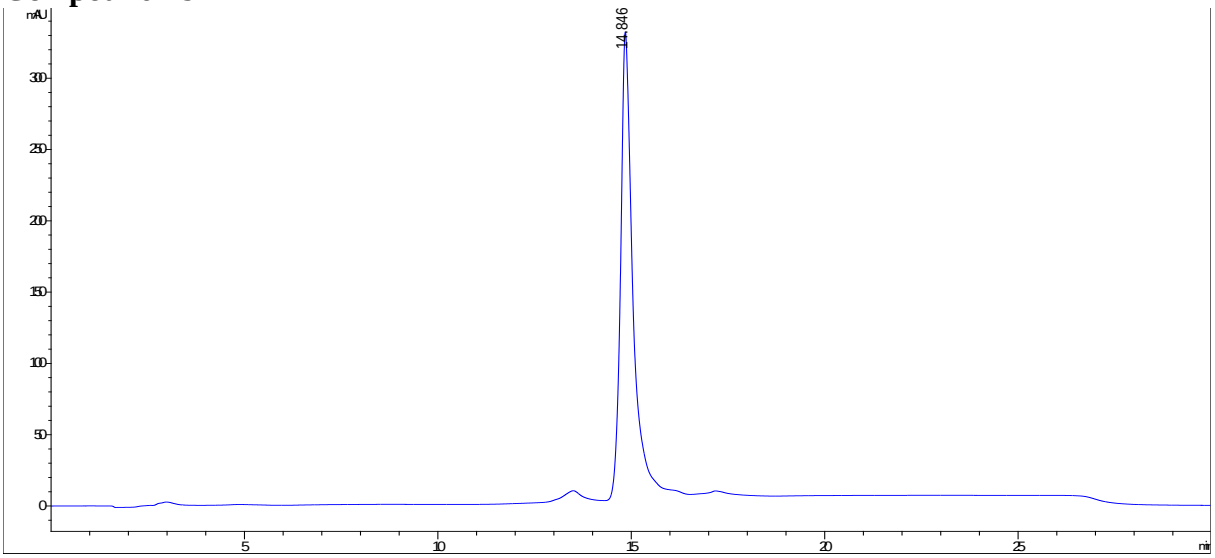
Compound 25



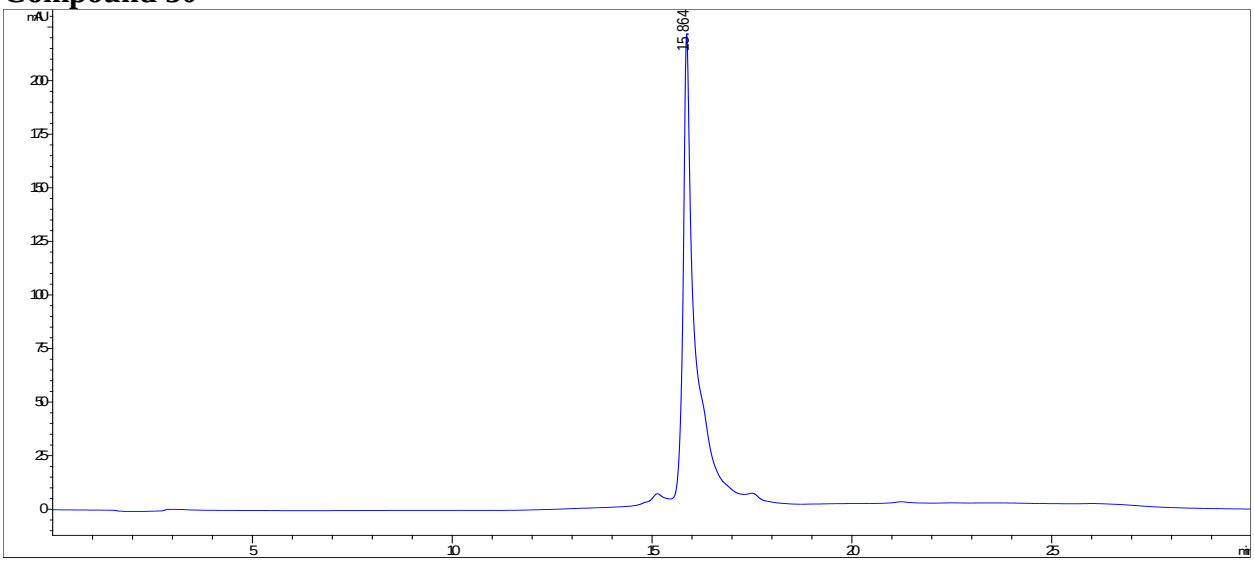
Compound 26



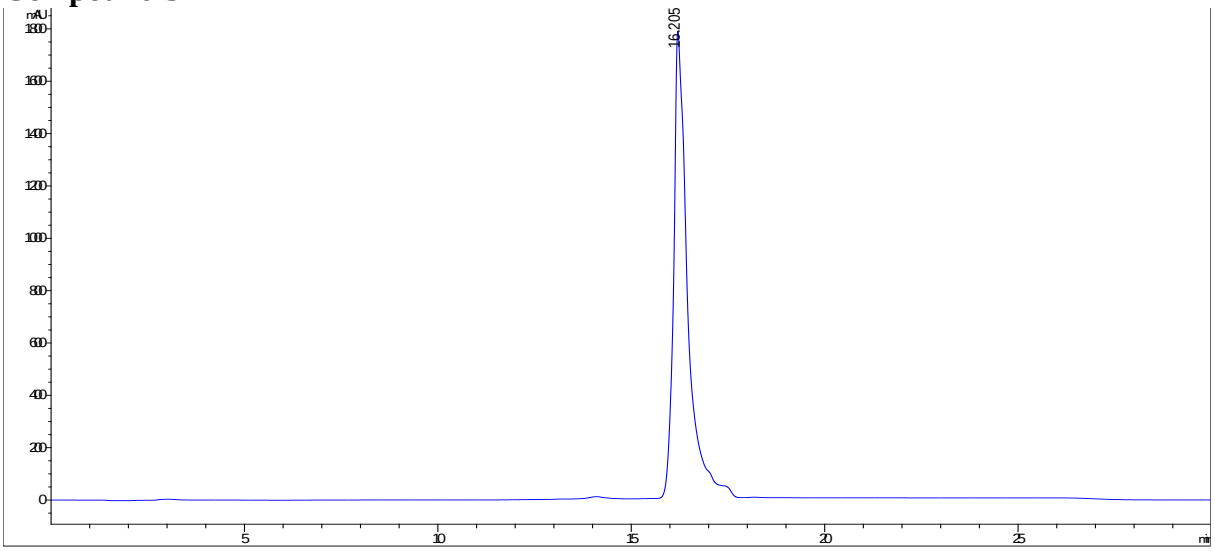
Compound 29



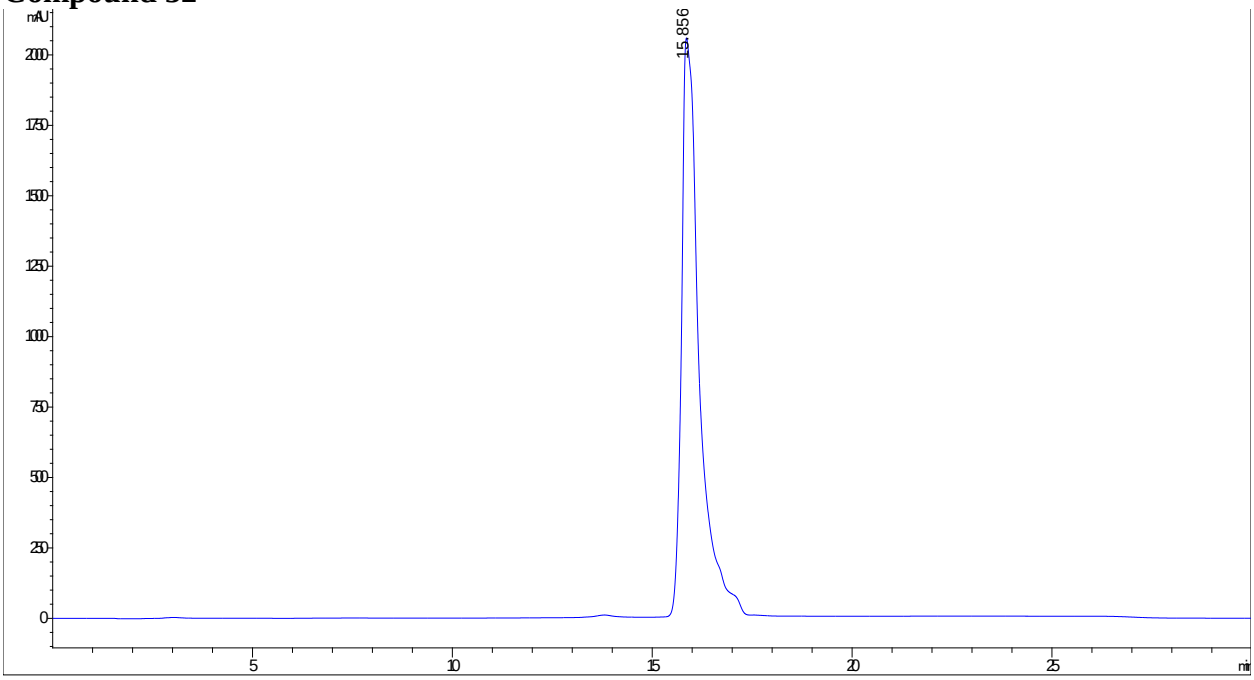
Compound 30



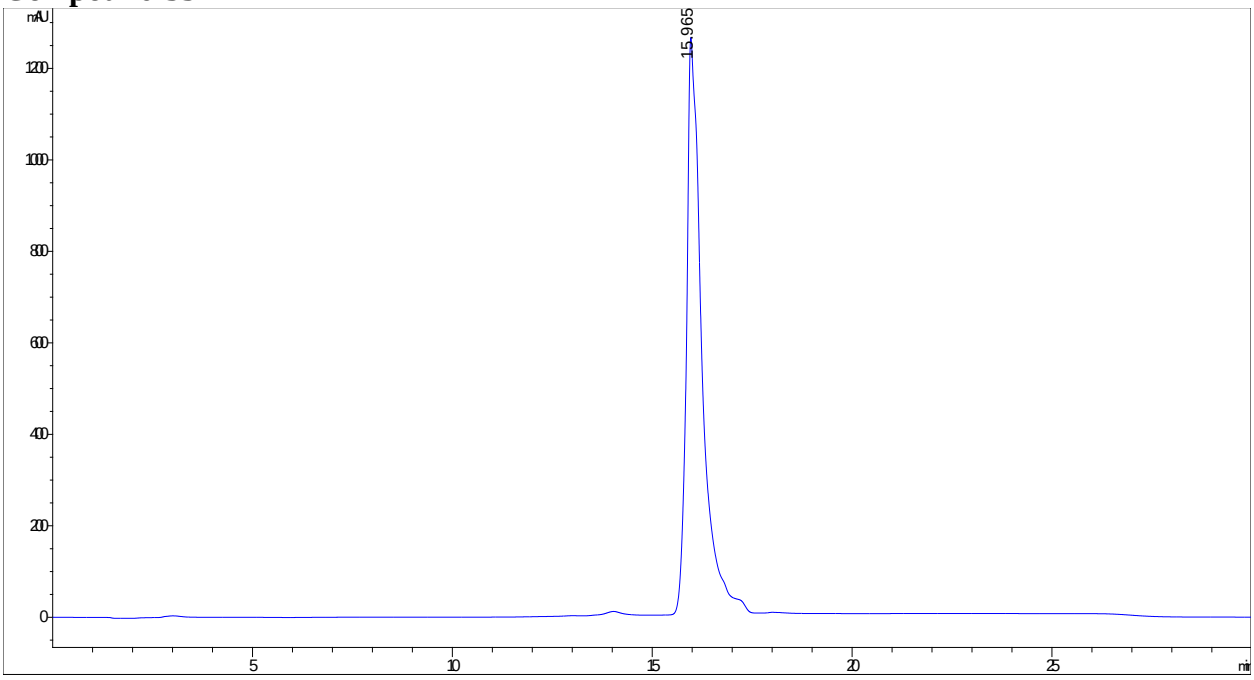
Compound 31



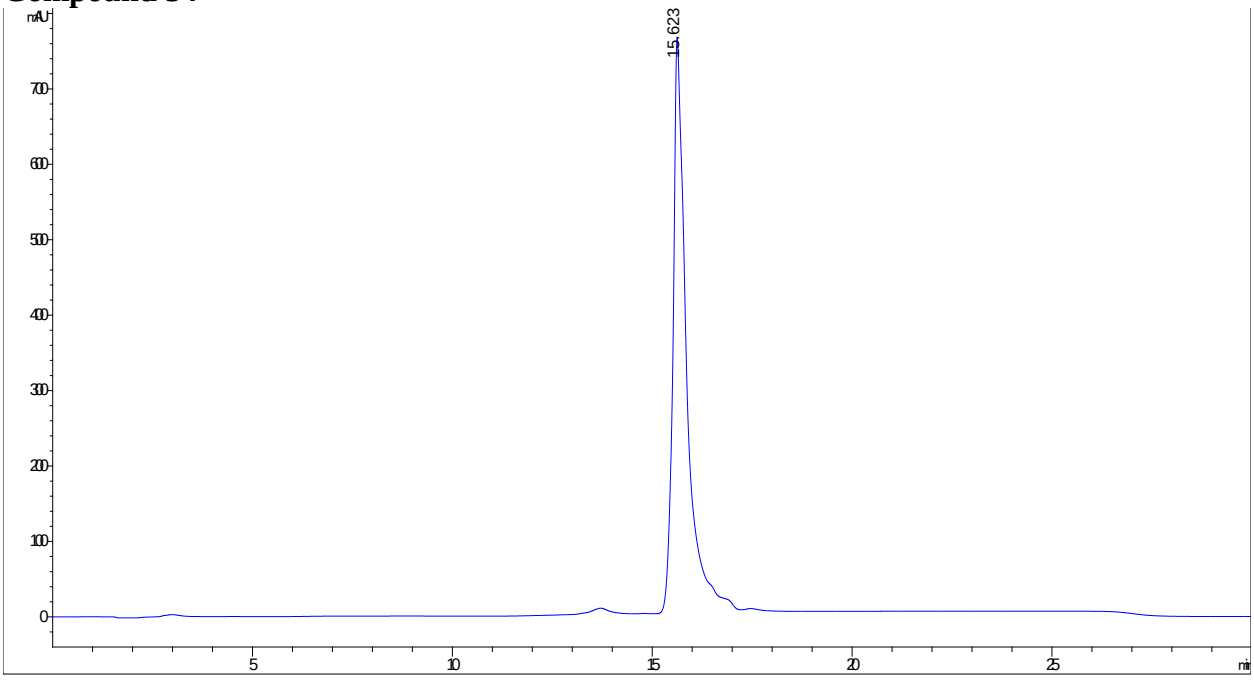
Compound 32



Compound 33



Compound 34



Compound 35