Supporting Information

Synthesis and oligomerization of cysteinyl nucleosides

Miklós Bege[a,b,c], Ilona Bereczki[a], Dénes J. Molnár[a], Máté Kicsák[a], Krisztina Pénzes-Daku[d], Zsuzsanna Bereczky[d], Györgyi Ferenc[e], Lajos Kovács[f], Pál Herczegh[a] and Anikó Borbás[a,*]

[a] Department of Pharmaceutical Chemistry, University of Debrecen, Debrecen, H-4032, Hungary

[b] Doctoral School of Pharmaceutical Sciences, University of Debrecen, Debrecen, H-4032, Hungary

[c] MTA-DE Molecular Recognition and Interaction Research Group, University of Debrecen, Debrecen, H-4032, Hungary

[d] Division of Clinical Laboratory Science, University of Debrecen, Debrecen, H-4032, Hungary

[e] Nucleic Acid Synthesis Laboratory, Biological Research Center, Szeged, H-6726, Hungary

[f] Nucleic Acids Laboratory, Department of Medicinal Chemistry, University of Szeged, H-6720 Szeged, Hungary

Table of contents

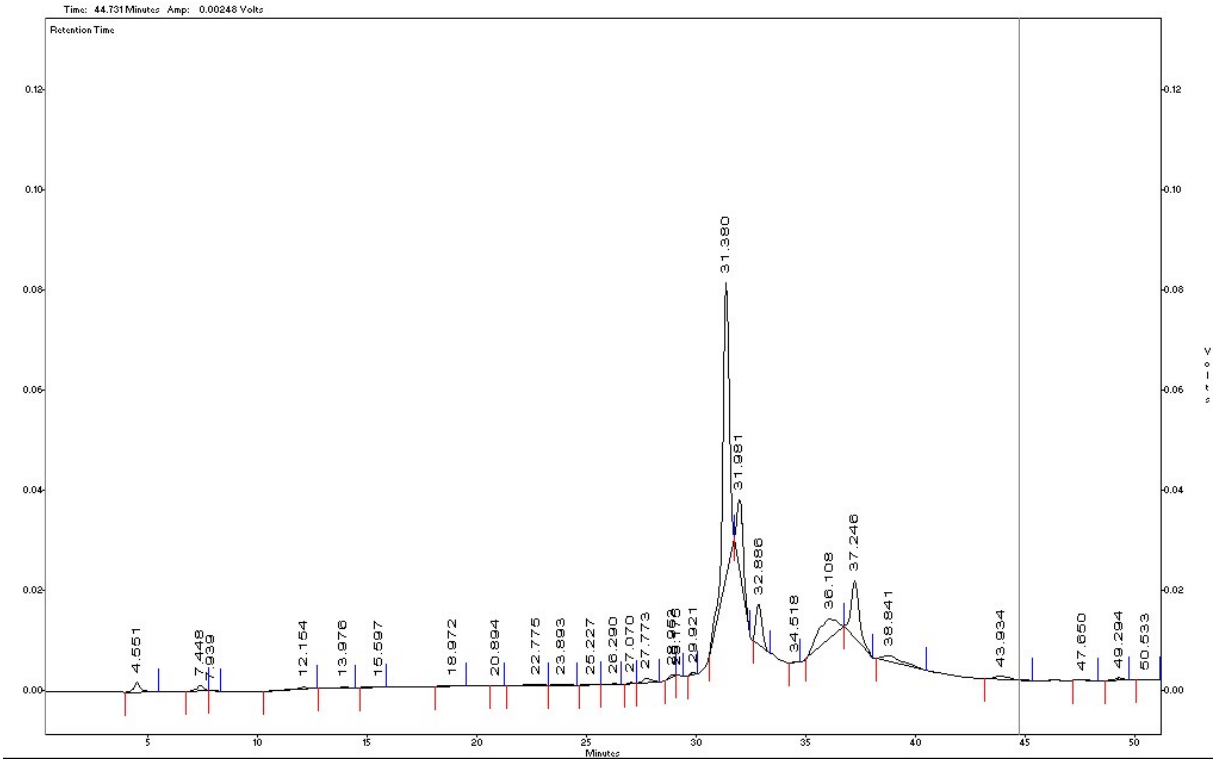


Figure S1: HPLC chromatogram of crude **13**. Jupiter C18, 300 Å, 250 × 4.6 mm column packing; detection: 260 nm; flow rate: 1.0 mL min-1; eluents: A: 0.1% TFA in water : acetonitrile = 99:1., B: 0.1% TFA in water:acetonitrile=50:50; gradient: 0–50% (v/v) B in A during 50 min.

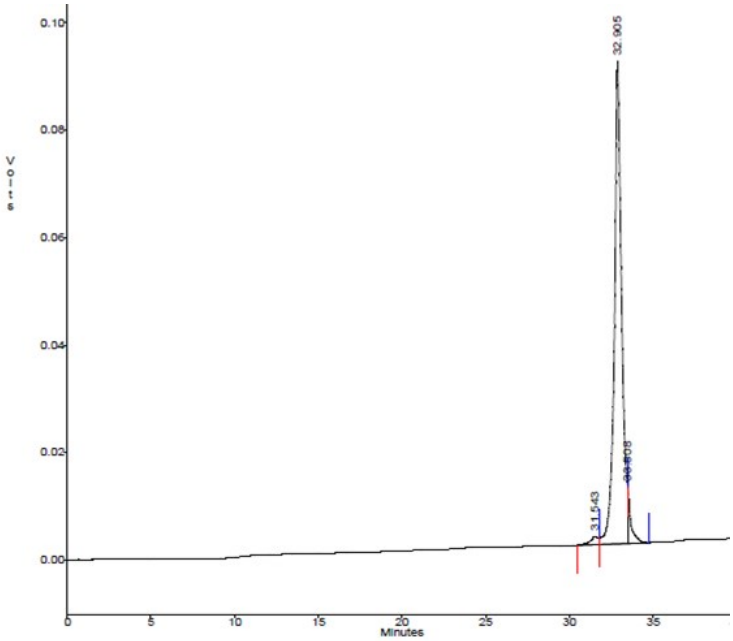
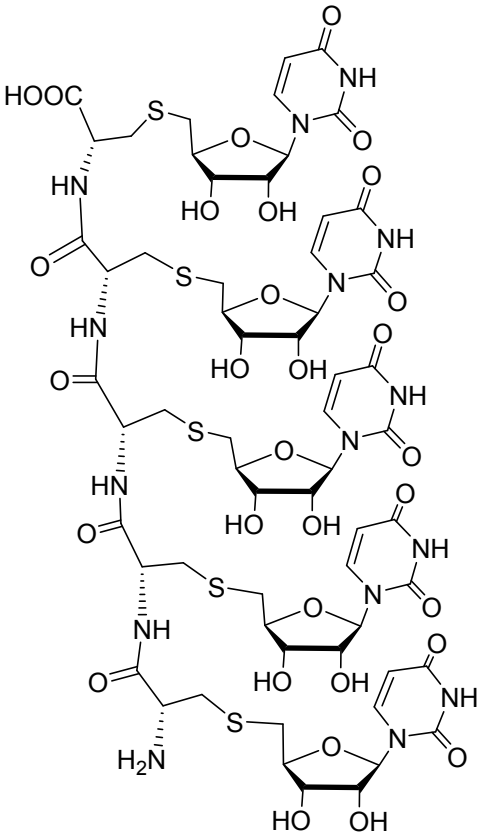


Figure S2: HPLC chromatogram of the purified **13.** Jupiter C18, 300 Å, 250 × 4.6 mm column packing; detection: 260 nm; flow rate: 1.0 mL min-1; eluents: A: 0.1% TFA in water : acetonitrile = 99:1., B: 0.1% TFA in water:acetonitrile=50:50; gradient: 0–50% (v/v) B in A during 50 min.



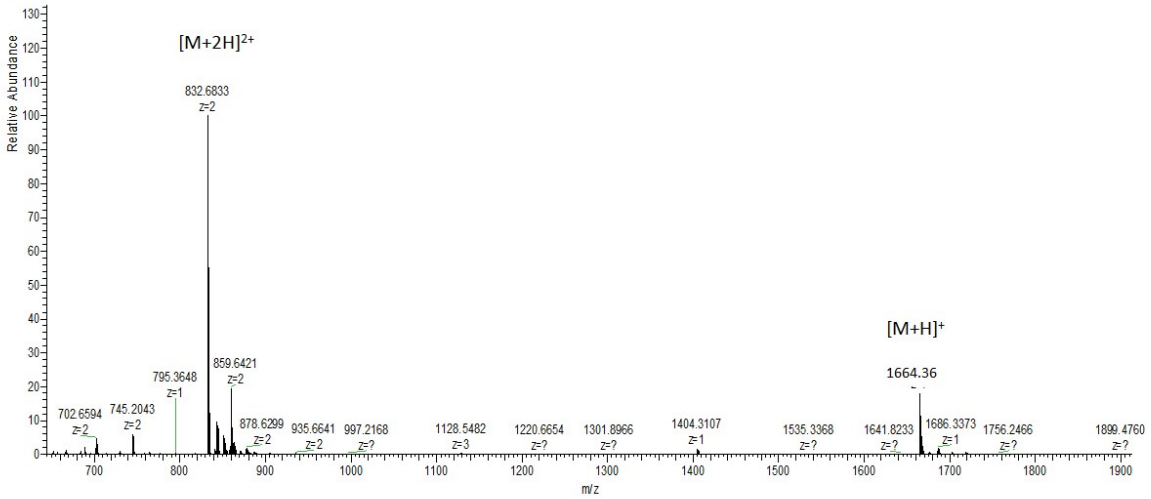
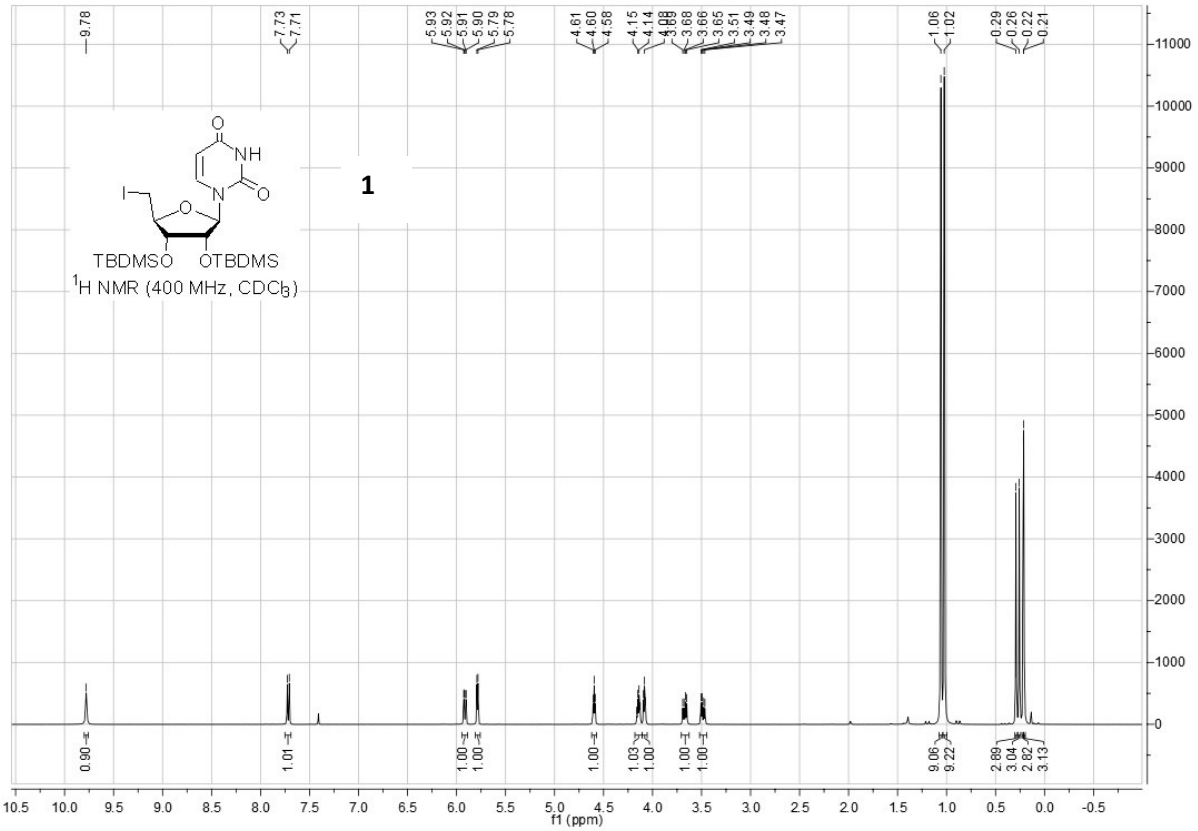
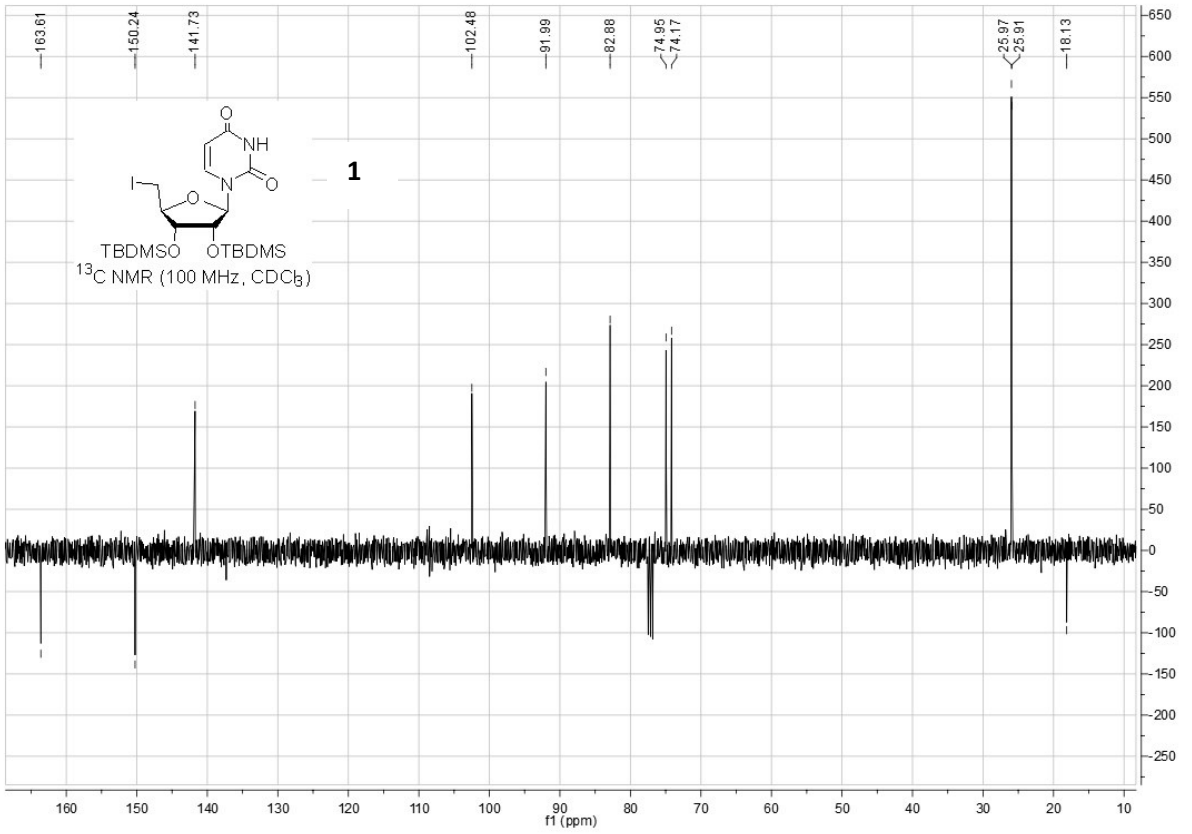
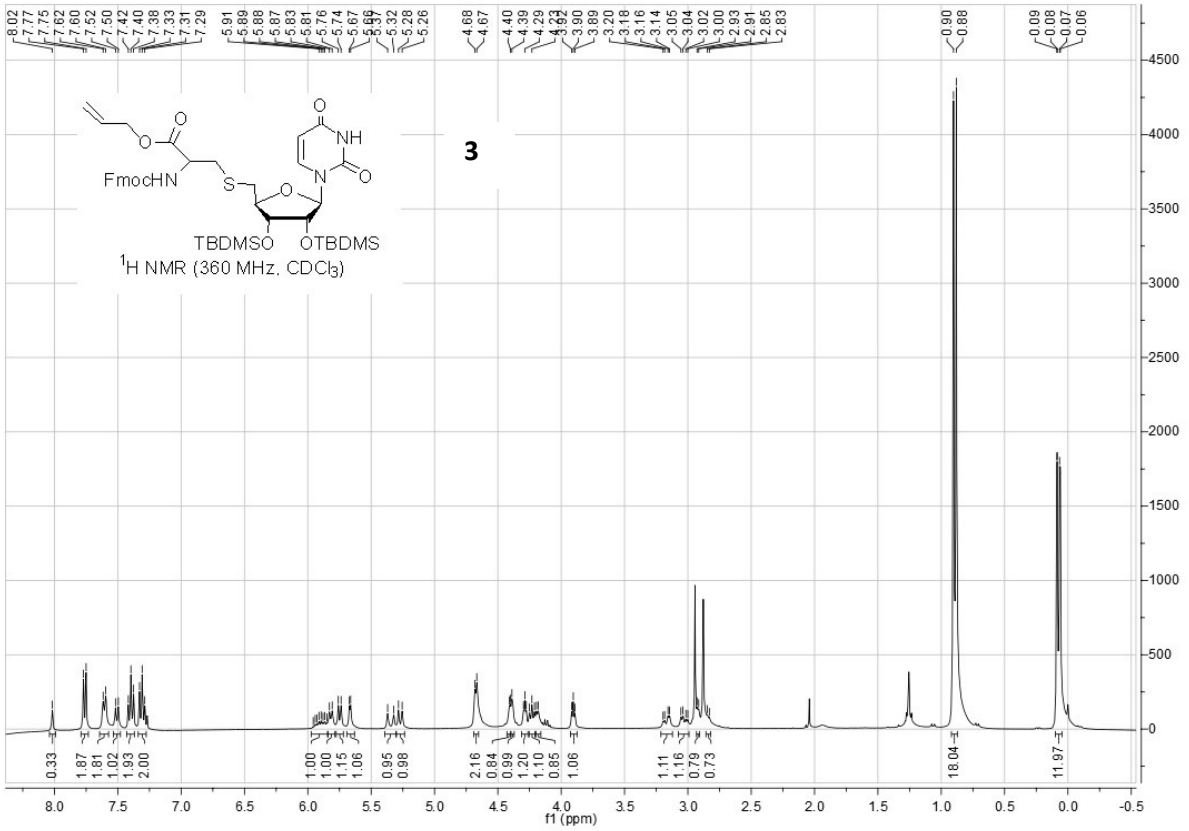


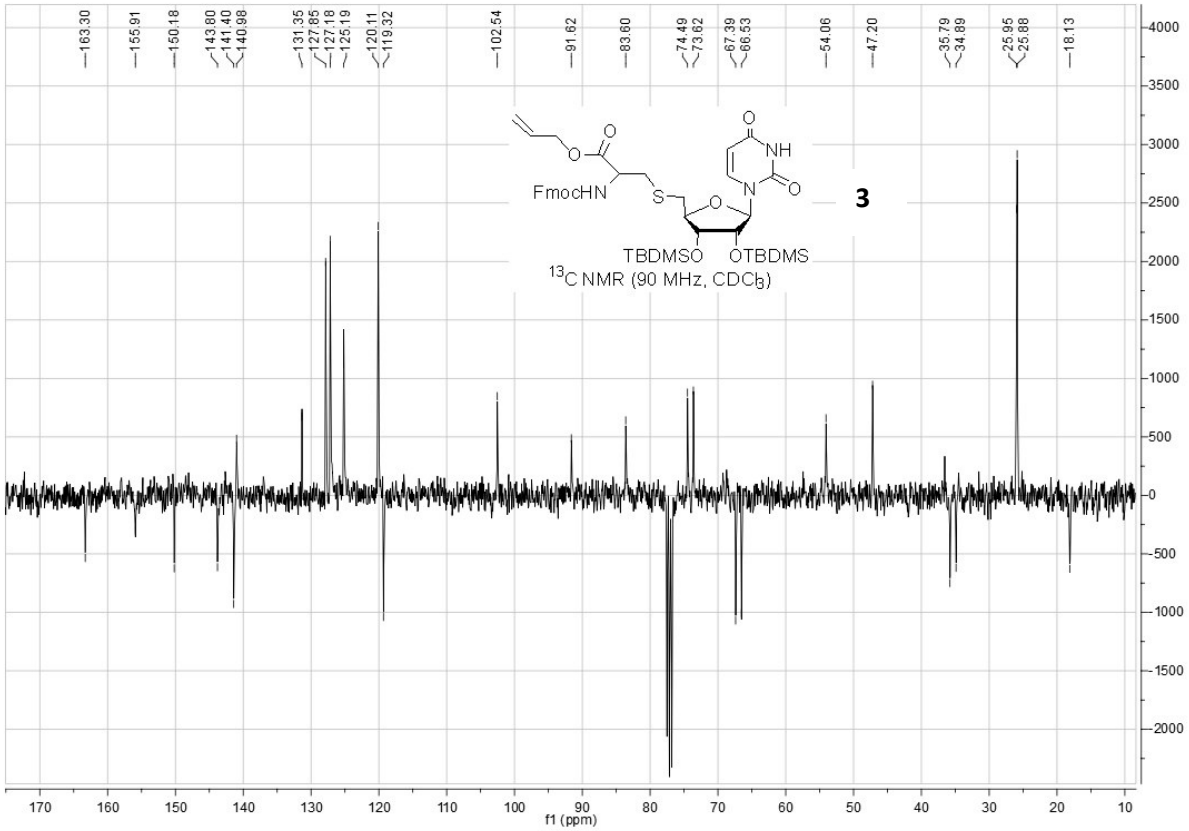
Figure S3: ESI mass spectrum of the purified compound **13**.

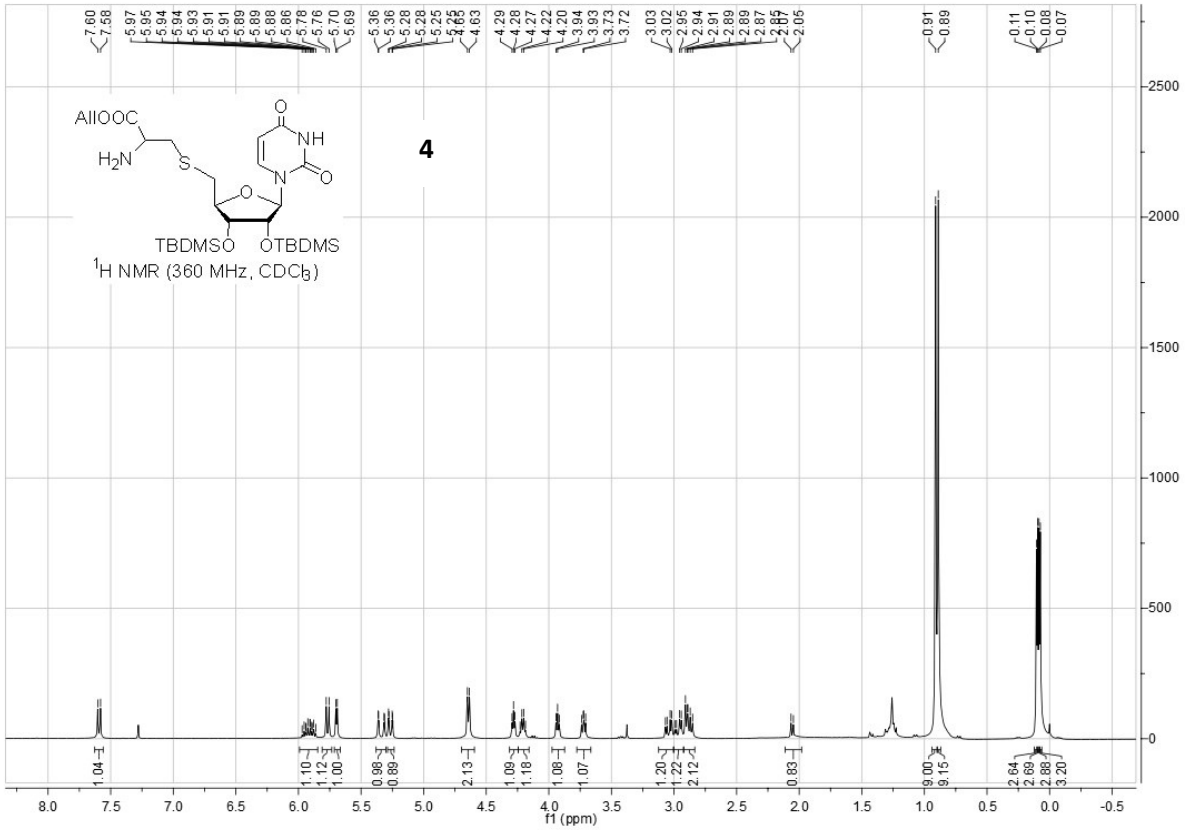
^{1}H and ^{13}C NMR spectra of the compounds

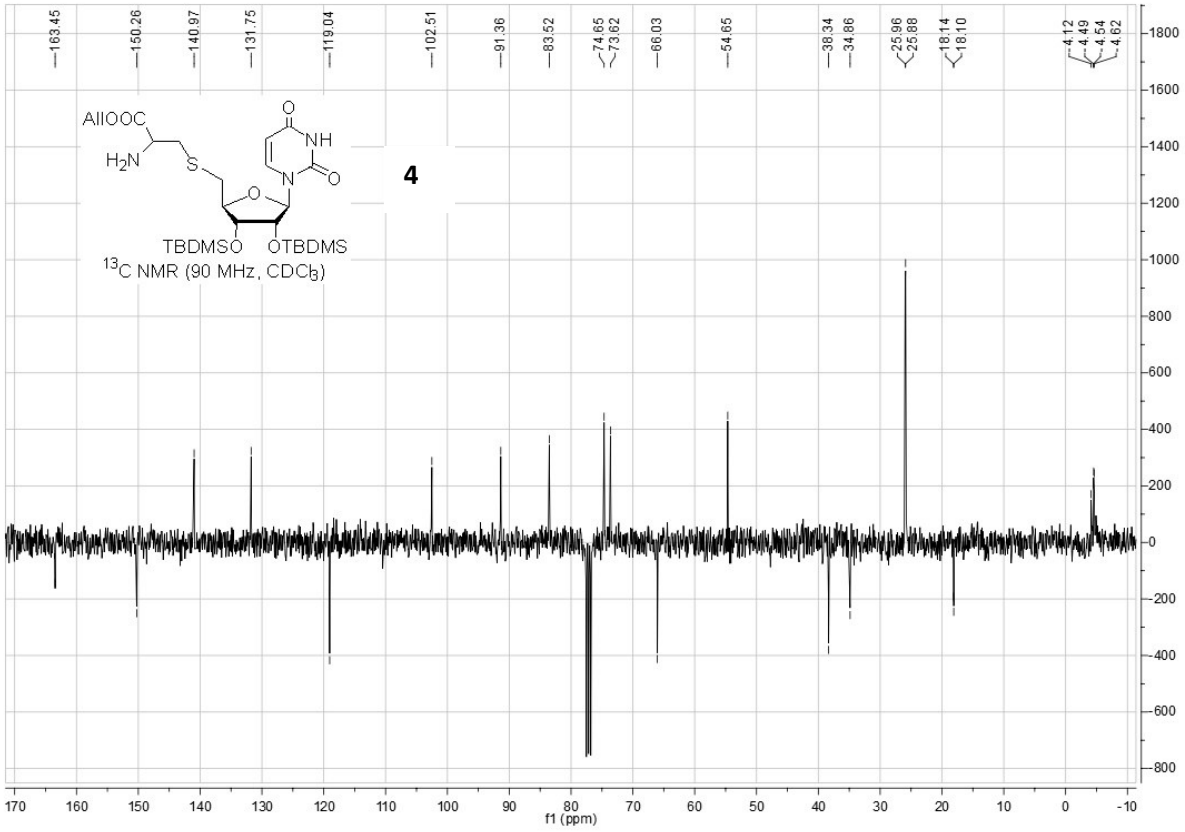


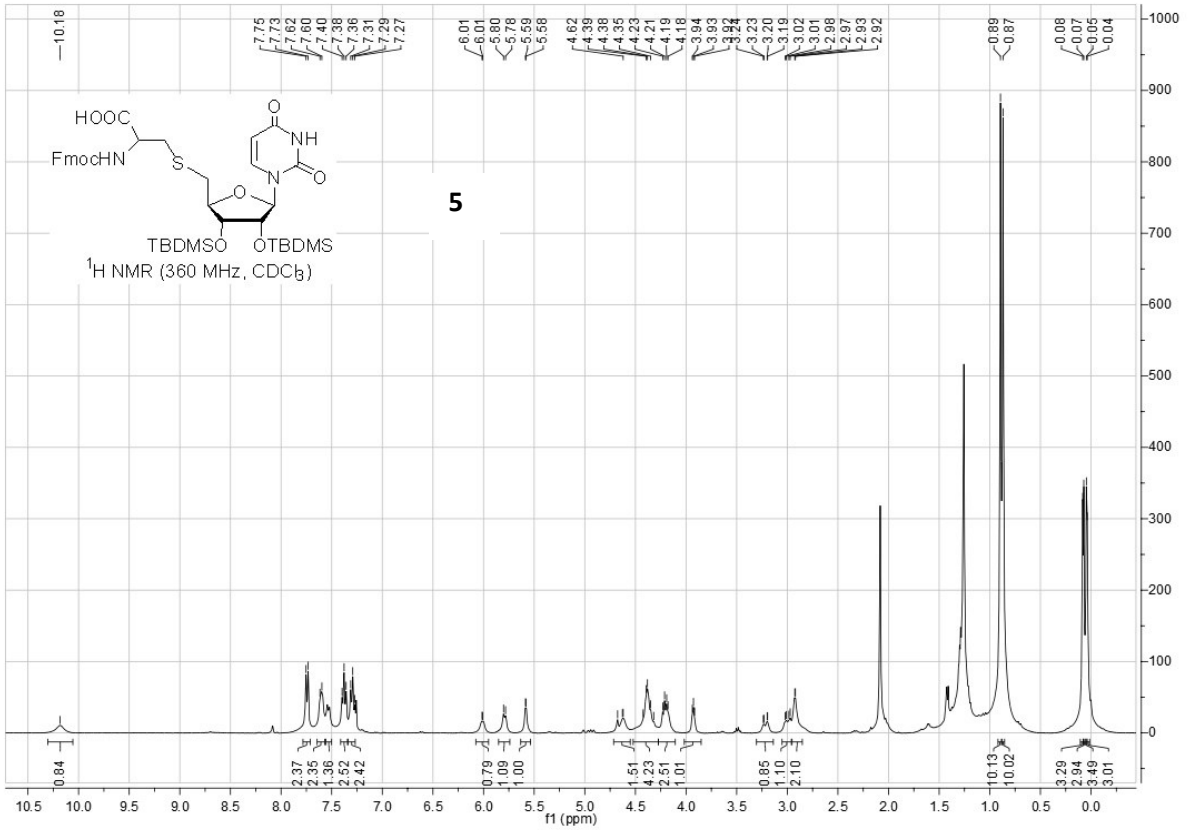


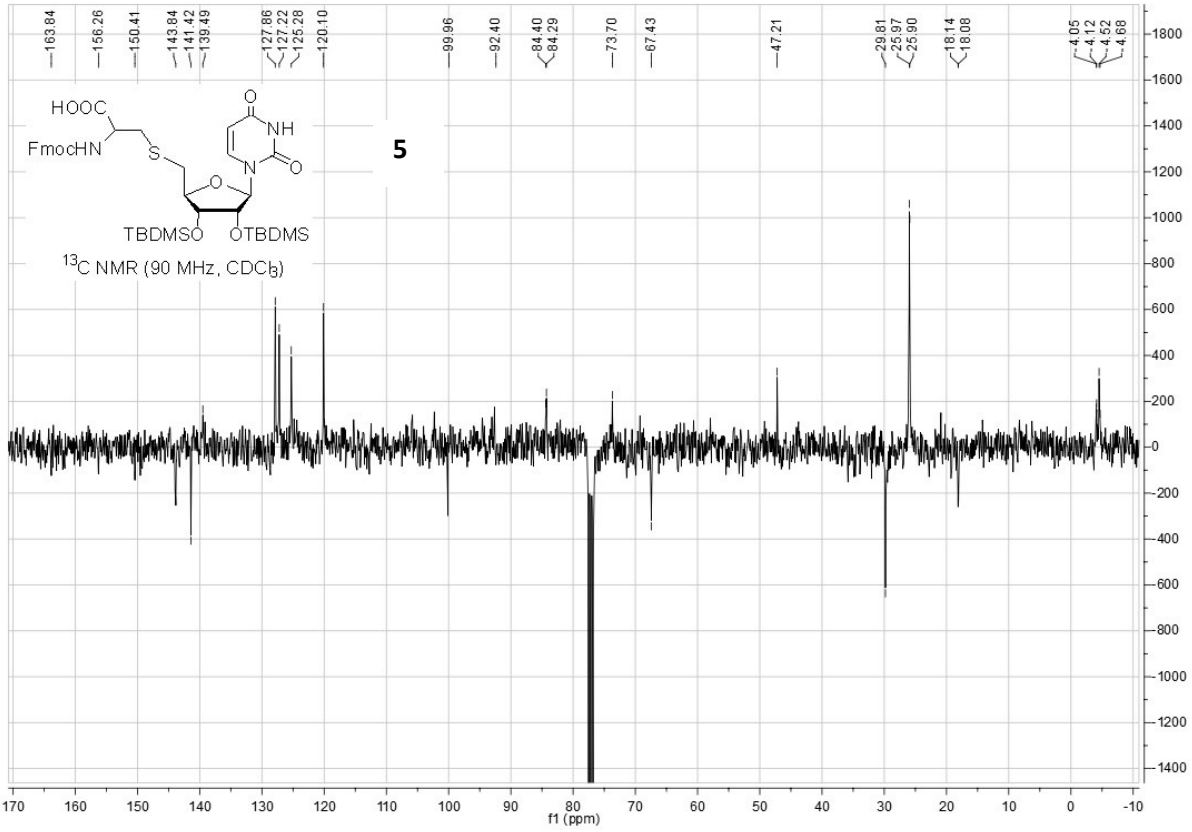

3
FmocHN
TBDMSO
OTBDMS
^{1}H NMR (360 MHz, CDCl$_3$)
f1 (ppm)

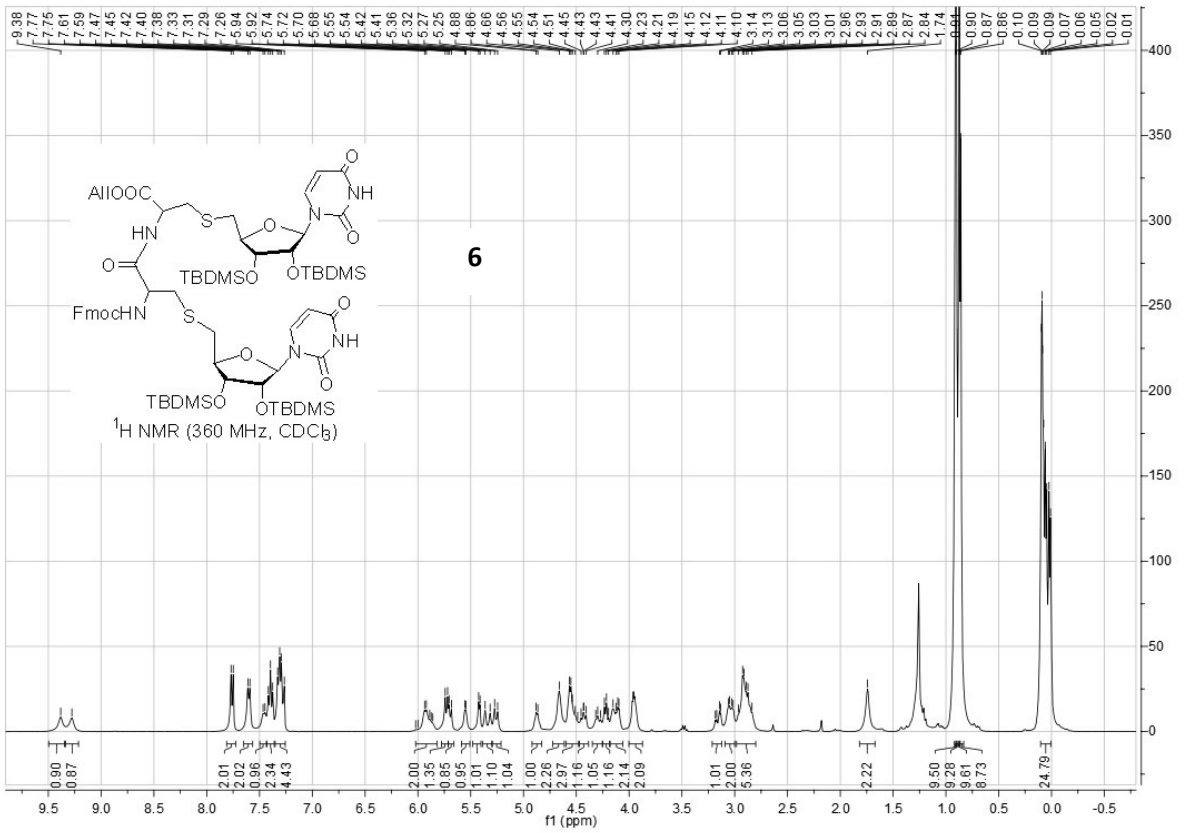

3
FmocHN
TBDMSO
OTBDMS
^{13}C NMR (90 MHz, CDCl$_3$)
f1 (ppm)

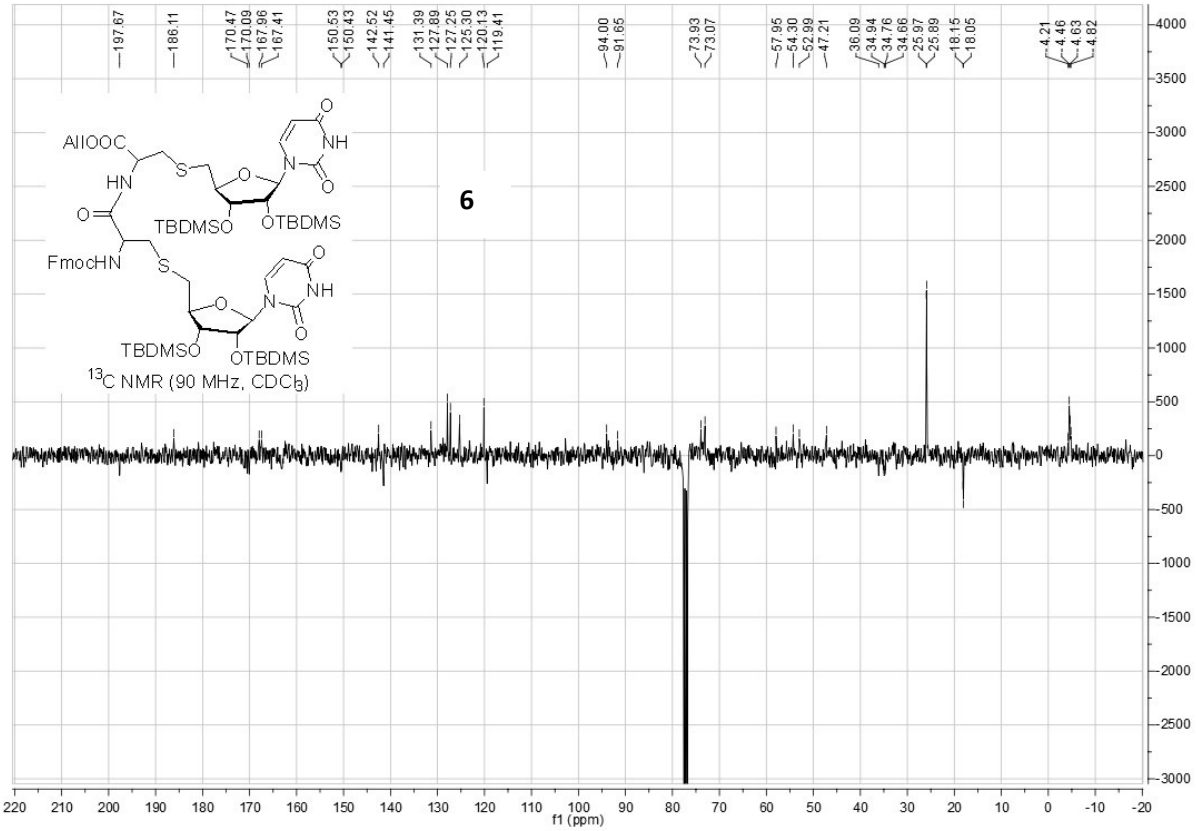

AllOOC
H2N
S
O
NH
N
O
TBDMSO
OTBDMS
4
1H NMR (360 MHz, CDCl3)
f1 (ppm)

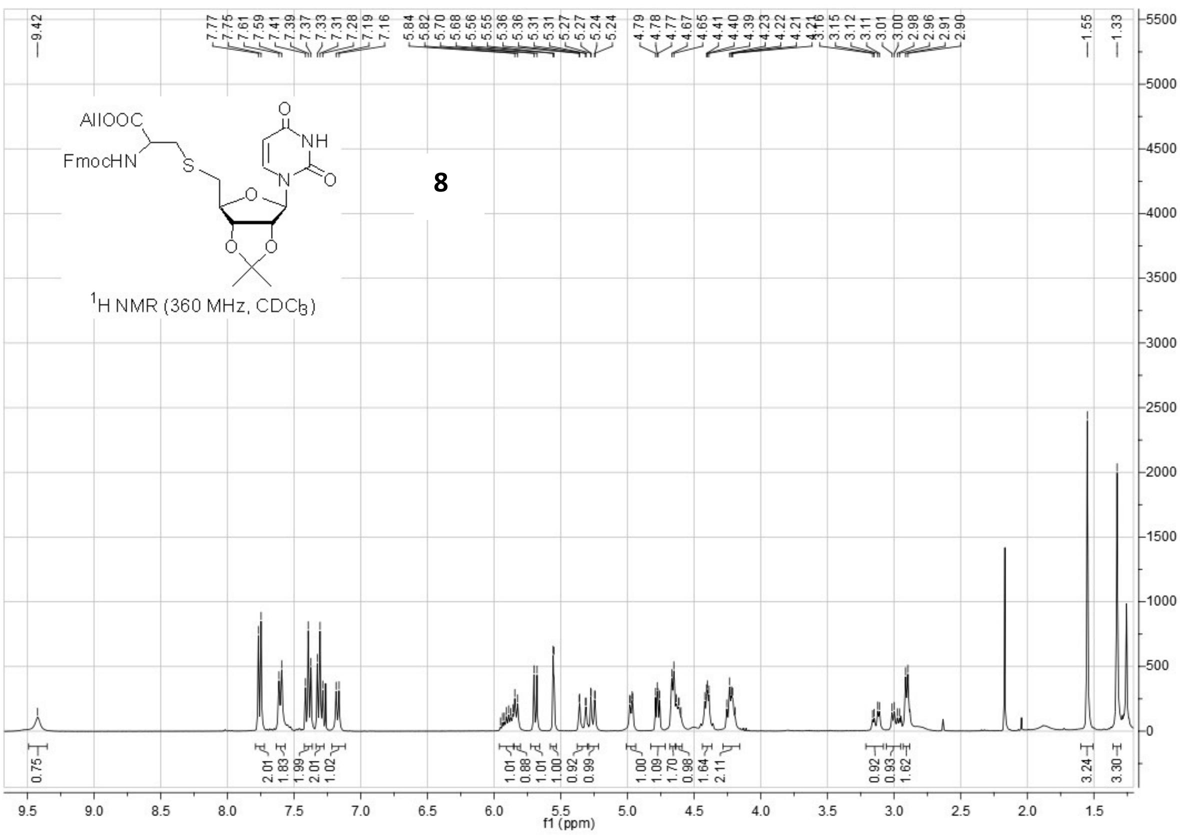

AllOOC
H2N
S
O
NH
N
O
TBDMSO
OTBDMS
4
13C NMR (90 MHz, CDCl3)
f1 (ppm)

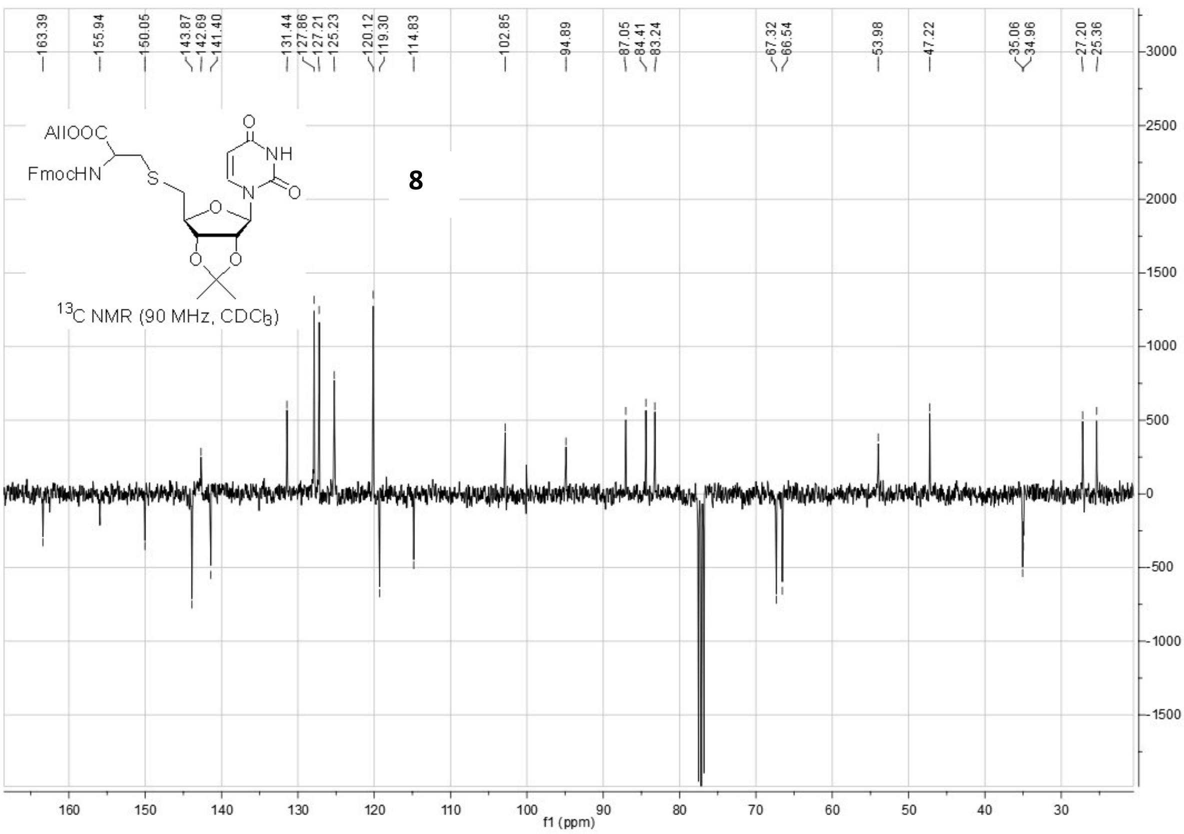

HOOC
FmocHN
S
O
NH
N
O
TBDMSO
OTBDMS
5
^{1}H NMR (360 MHz, CDCl$_3$)
f1 (ppm)

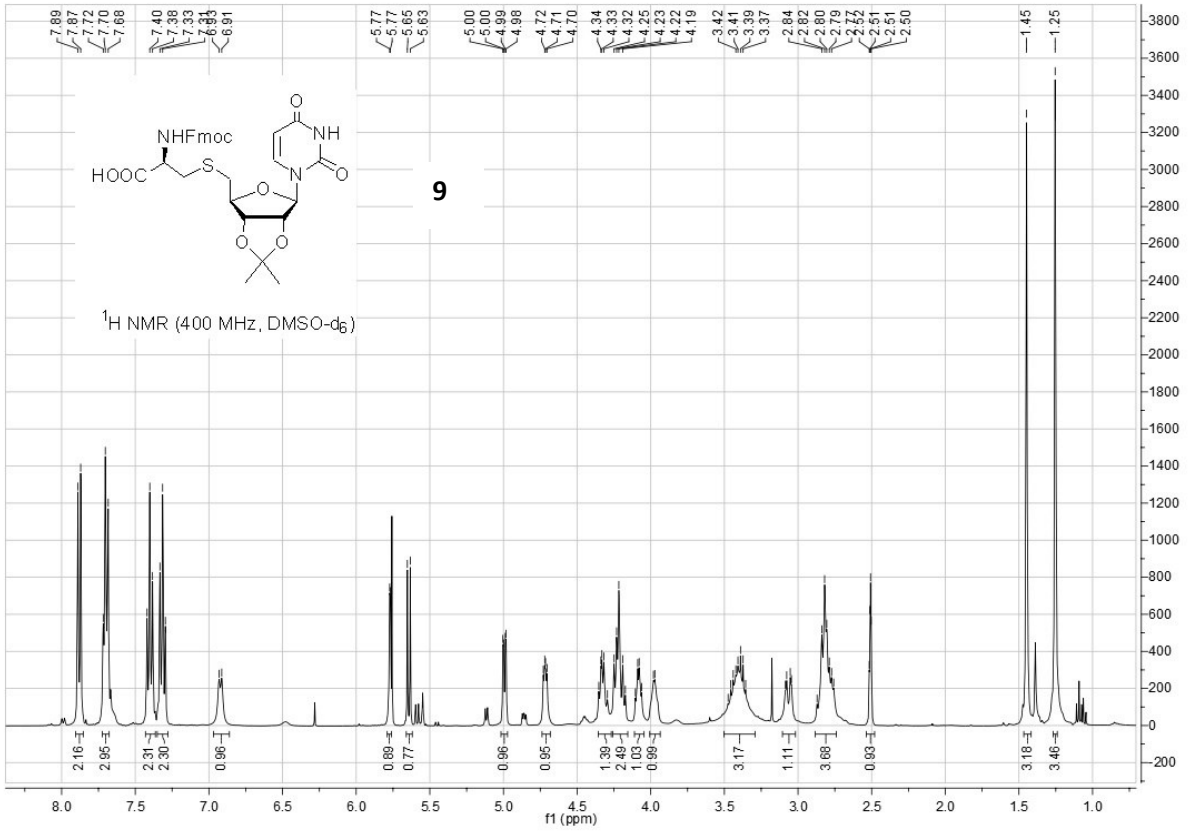

HOOC
FmocHN
S
O
NH
N
O
TBDMSO
OTBDMS
5
^{13}C NMR (90 MHz, CDCl$_3$)
f1 (ppm)

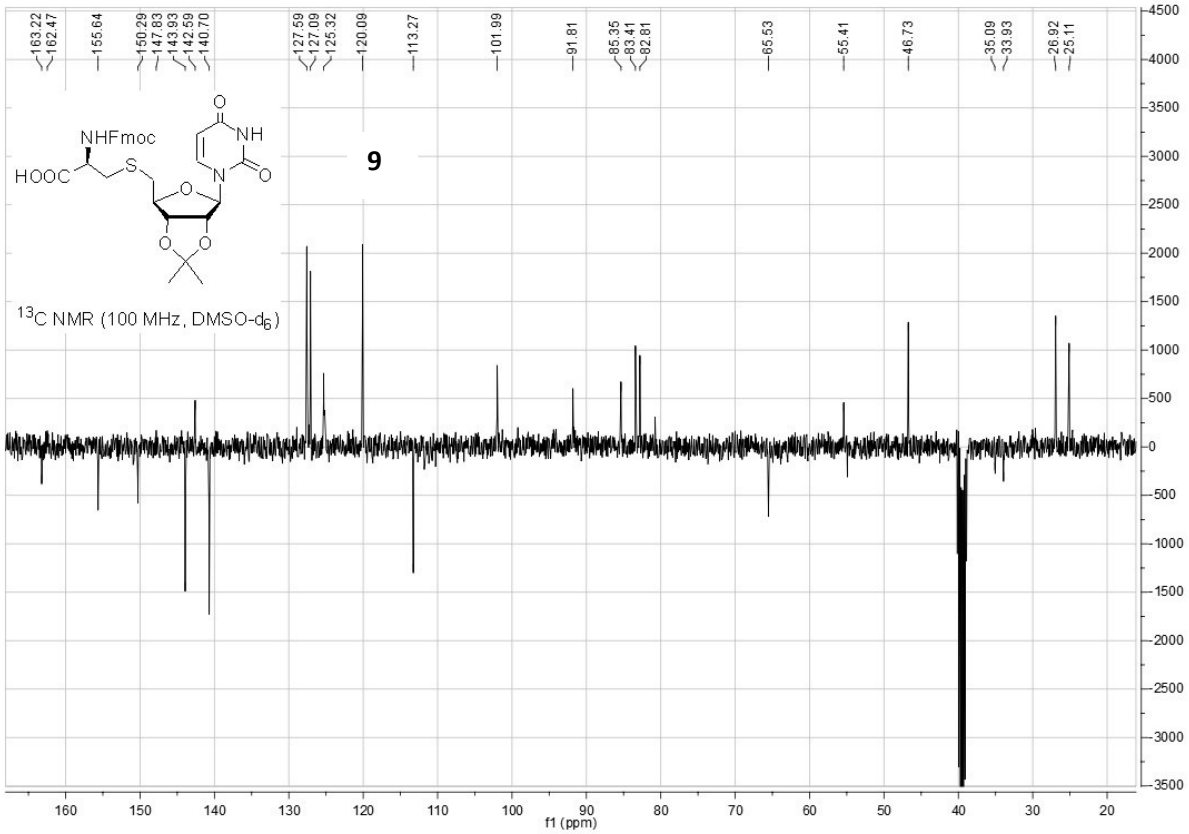

AllOOC
HN
TBDMSO
OTBDMS
FmocHN
TBDMSO
OTBDMS
6
^{1}H NMR (360 MHz, CDCl$_3$)

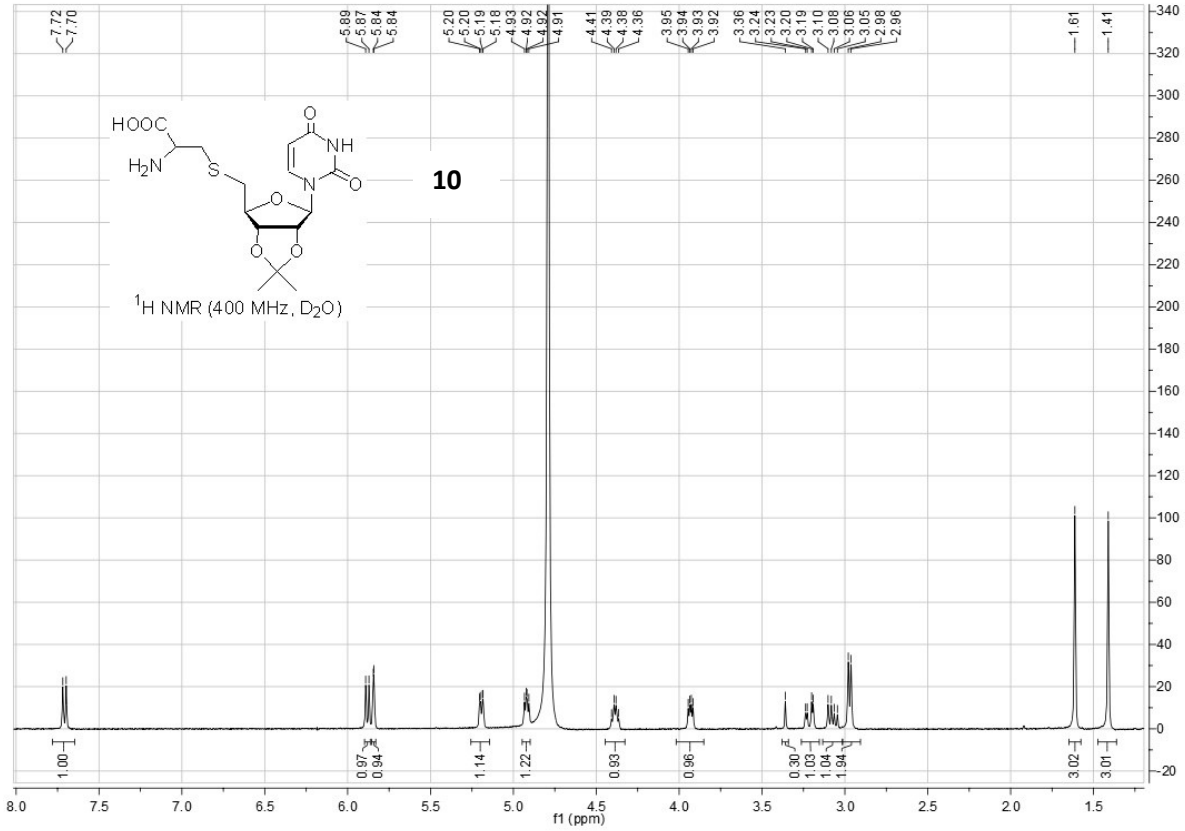

AllOOC
HN
TBDMSO
OTBDMS
FmocHN
TBDMSO
OTBDMS
6
^{13}C NMR (90 MHz, CDCl$_3$)

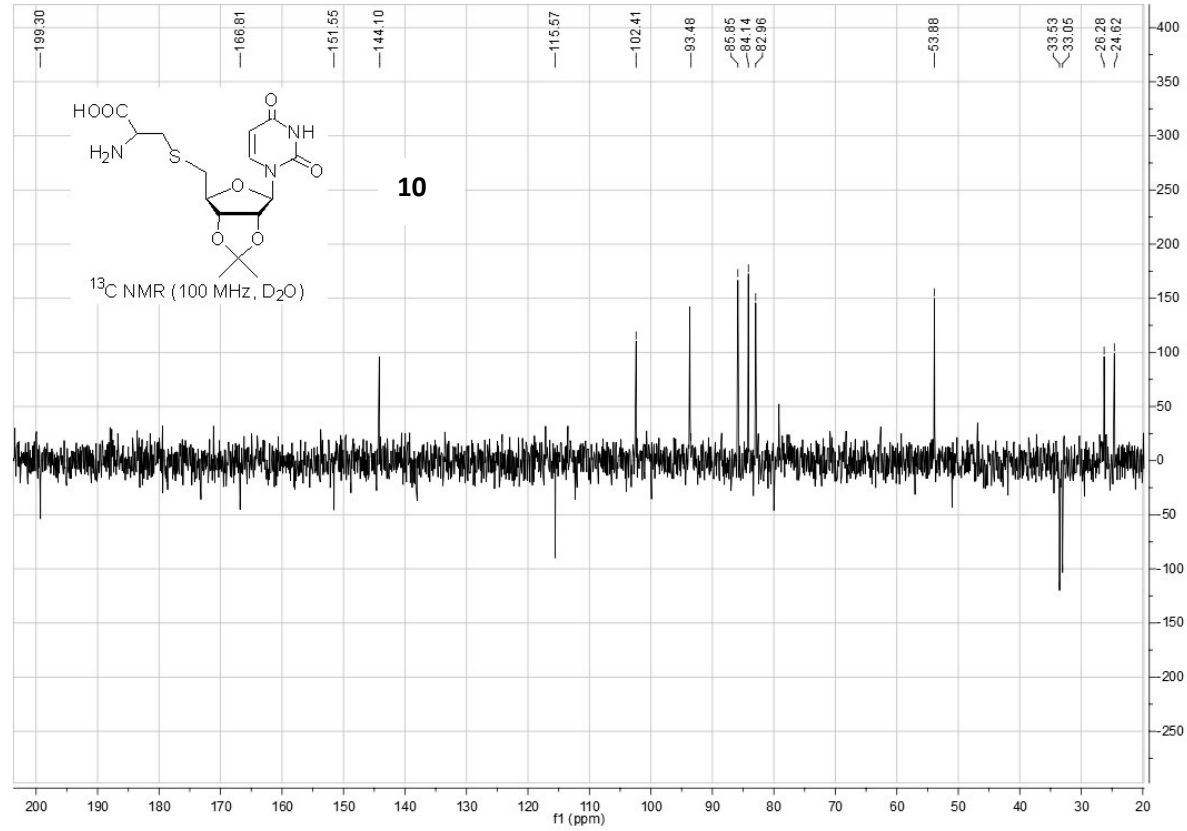

9.42
7.77 7.75 7.61 7.59 7.41 7.39 7.37 7.33 7.31 7.28 7.19 7.16
5.84 5.82 5.70 5.68 5.56 5.55 5.36 5.36 5.31 5.31 5.27 5.27 5.24 5.24
4.79 4.78 4.77 4.67 4.65 4.41 4.40 4.39 4.23 4.22 4.21 4.21
3.16 3.15 3.12 3.11 3.01 3.00 2.98 2.96 2.91 2.90
1.55
1.33
AllOOC
FmocHN
S
O
NH
N
8
1H NMR (360 MHz, CDCl3)
0.75
2.01 1.83 1.99 2.01 1.02
1.01 0.88 1.01 1.00 0.92 0.99
1.00 1.09 1.70 0.98 1.64 2.11
0.92 0.93 1.62
3.24
3.30
f1 (ppm)

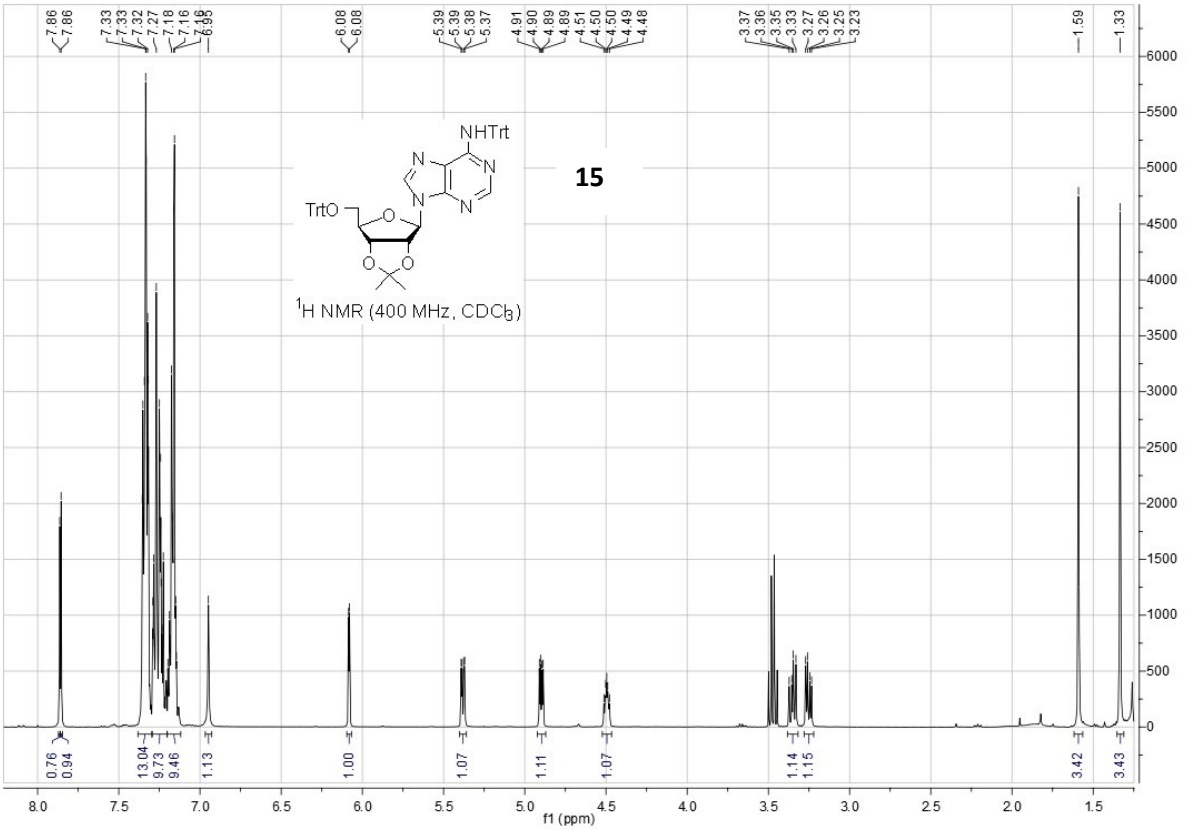

163.39
155.94
150.05
143.87 142.69 141.40
131.44 127.86 127.21 125.23
120.12 119.30
114.83
102.85
94.89
87.05 84.41 83.24
67.32 66.54
53.98
47.22
35.06 34.96
27.20 25.36
AllOOC
FmocHN
S
O
NH
N
8
13C NMR (90 MHz, CDCl3)
f1 (ppm)

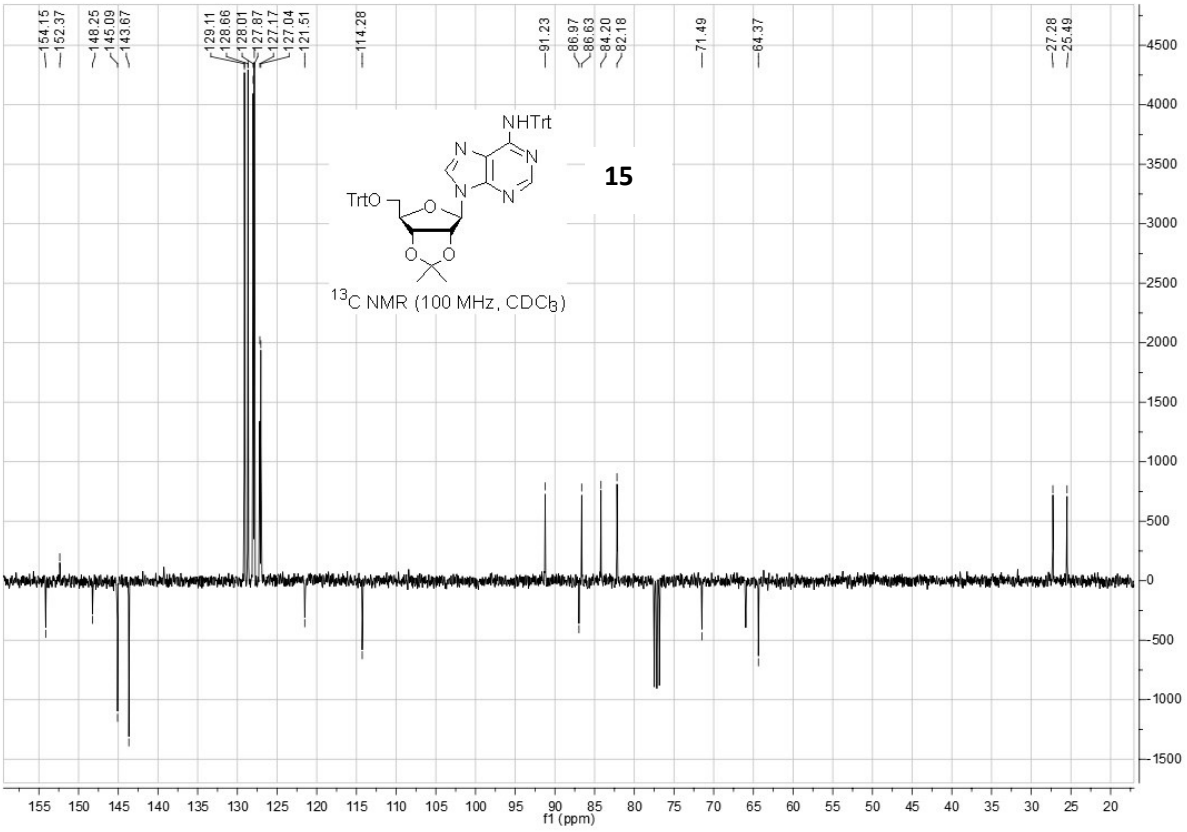

NHFmoc
HOOC
NH
O
O
S
N
O
O
O
9
^{1}H NMR (400 MHz, DMSO-d$_6$)
f1 (ppm)

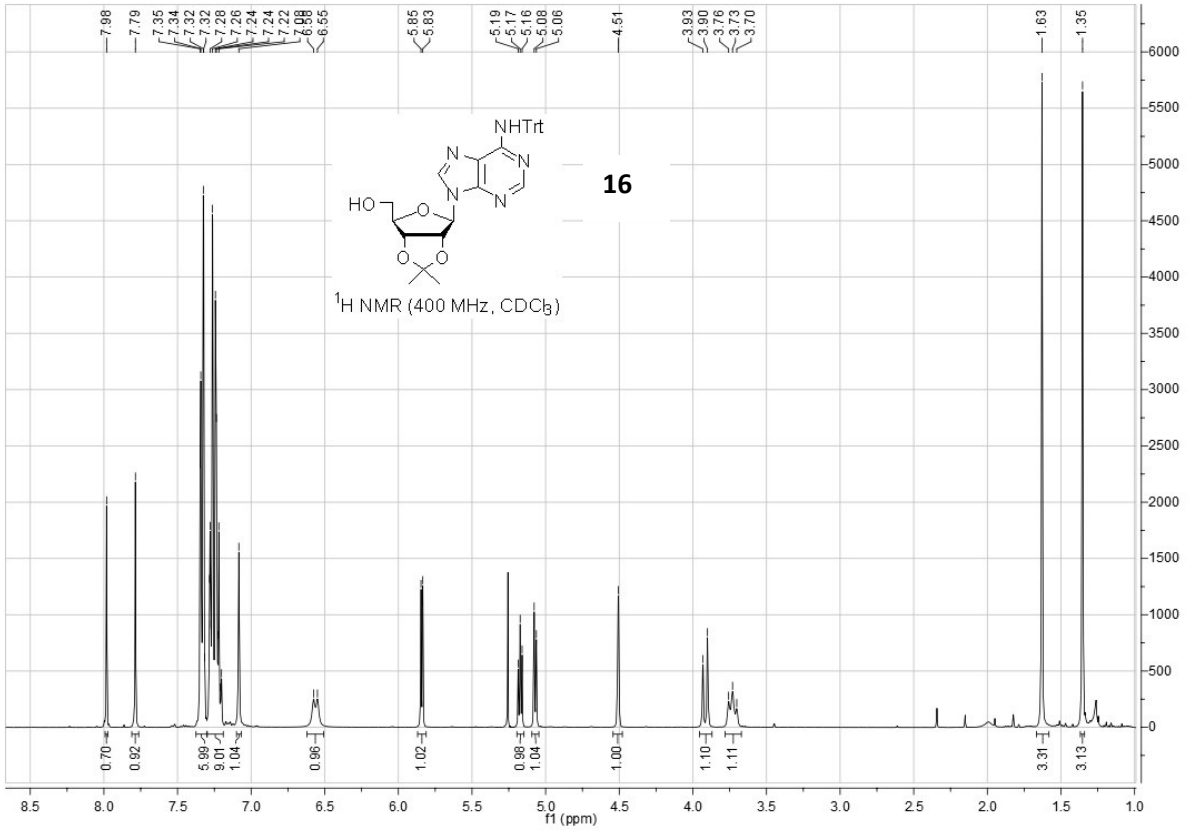

NHFmoc
HOOC
NH
O
O
S
N
O
O
O
9
^{13}C NMR (100 MHz, DMSO-d$_6$)
f1 (ppm)

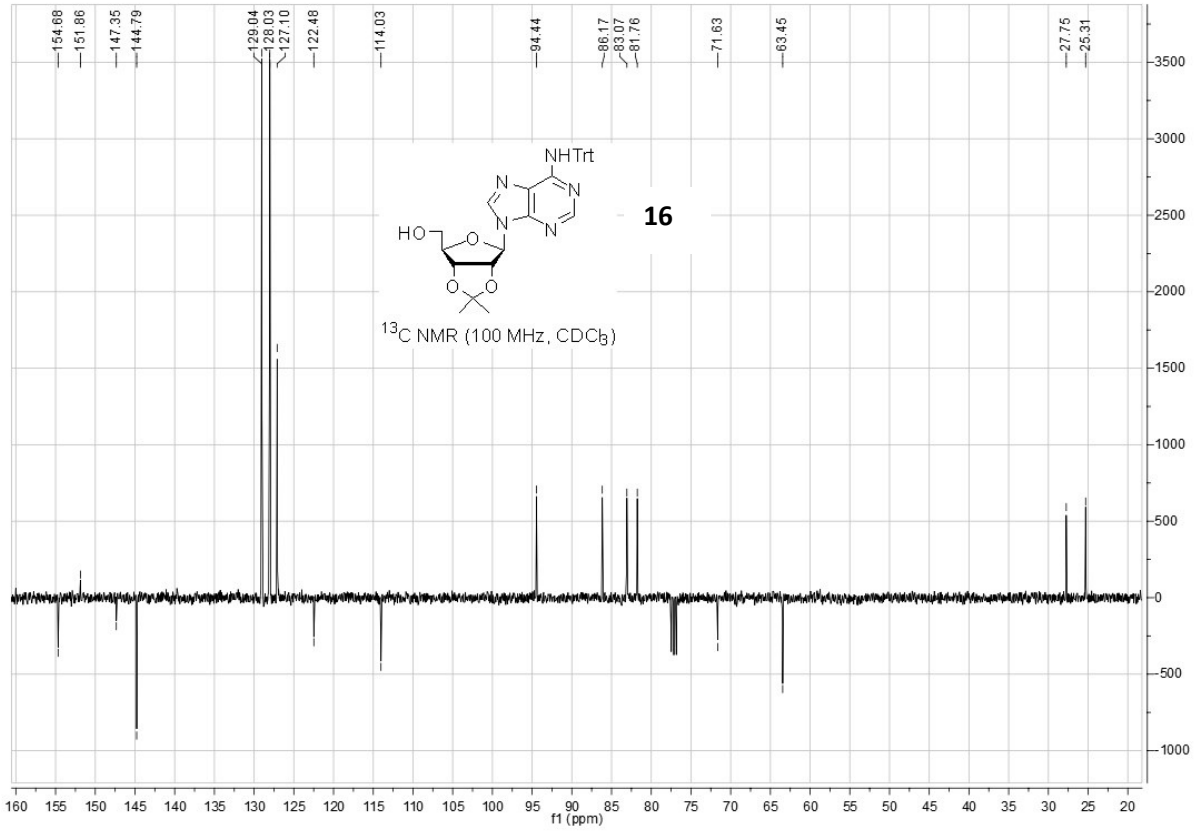

10
^{1}H NMR (400 MHz, D_2O)
f1 (ppm)

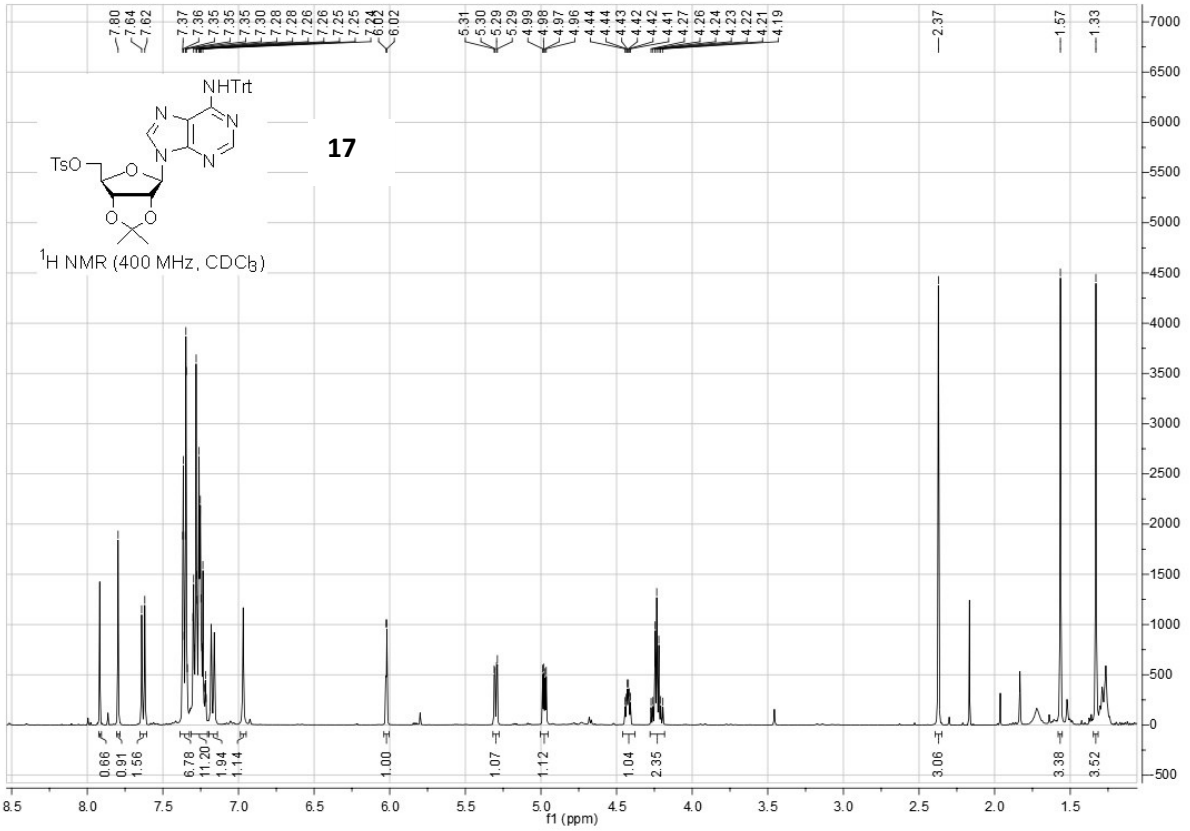

10
^{13}C NMR (100 MHz, D_2O)
f1 (ppm)

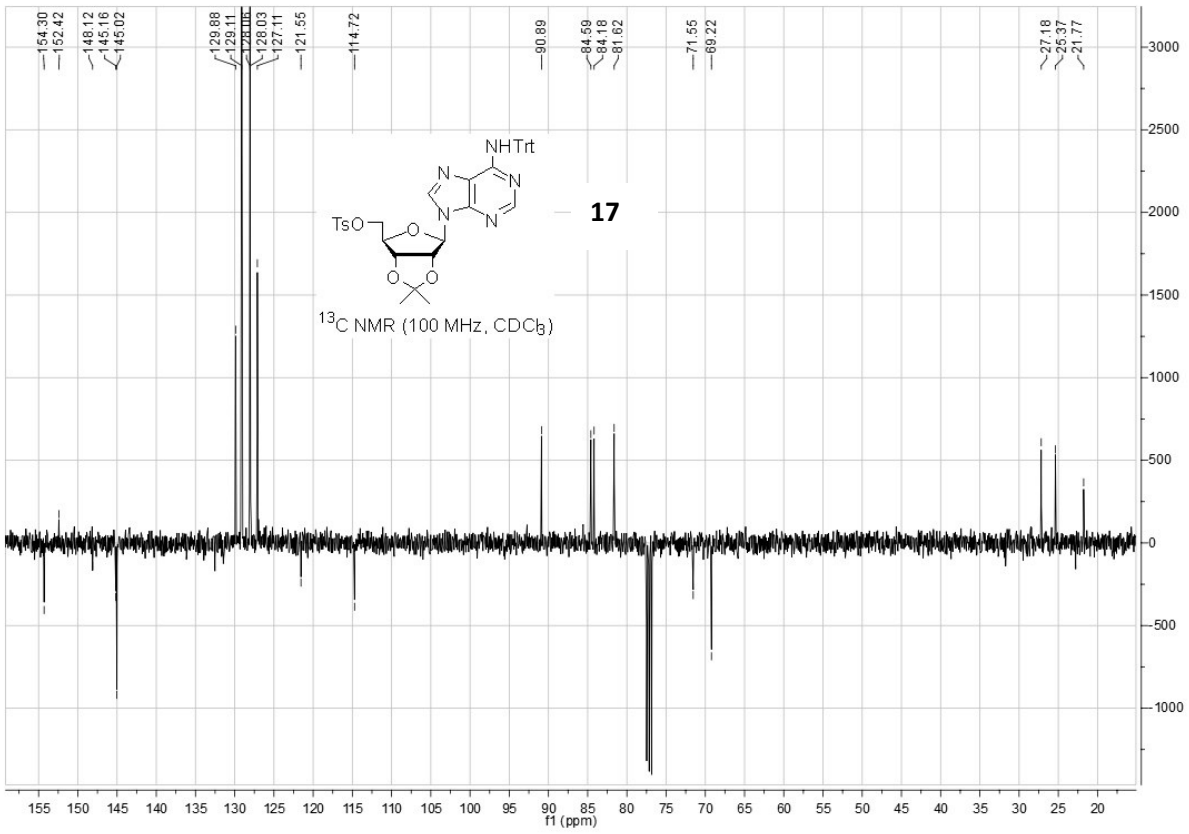

NHTrt
TrtO
15
^{1}H NMR (400 MHz, CDCl$_3$)

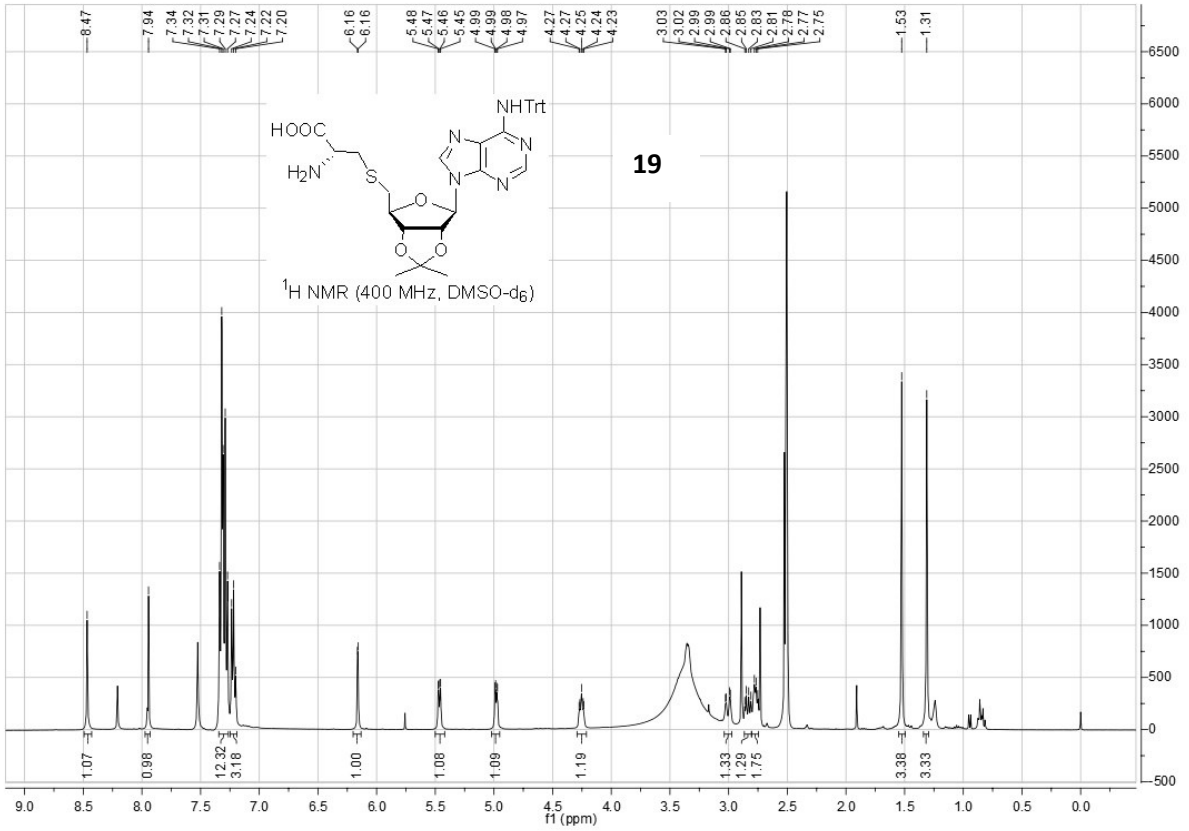

NHTrt
TrtO
15
^{13}C NMR (100 MHz, CDCl$_3$)

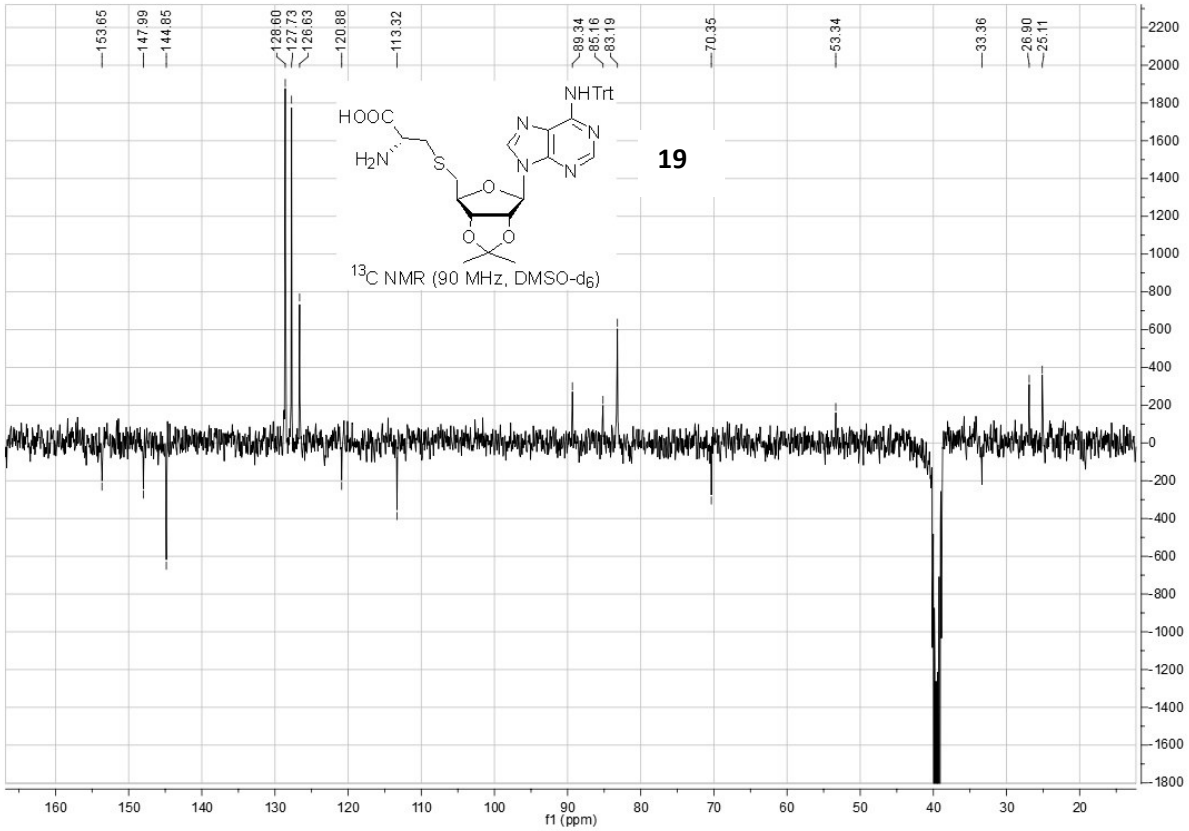

7.98
7.79
7.35
7.34
7.32
7.32
7.28
7.26
7.24
7.24
7.22
6.98
6.55
5.85
5.83
5.19
5.17
5.16
5.08
5.06
4.51
3.93
3.90
3.76
3.73
3.70
1.63
1.35
NHTrt
HO
16
^{1}H NMR (400 MHz, CDCl$_3$)
0.70
0.92
5.99
9.01
1.04
0.96
1.02
0.98
1.04
1.00
1.10
1.11
3.31
3.13
f1 (ppm)

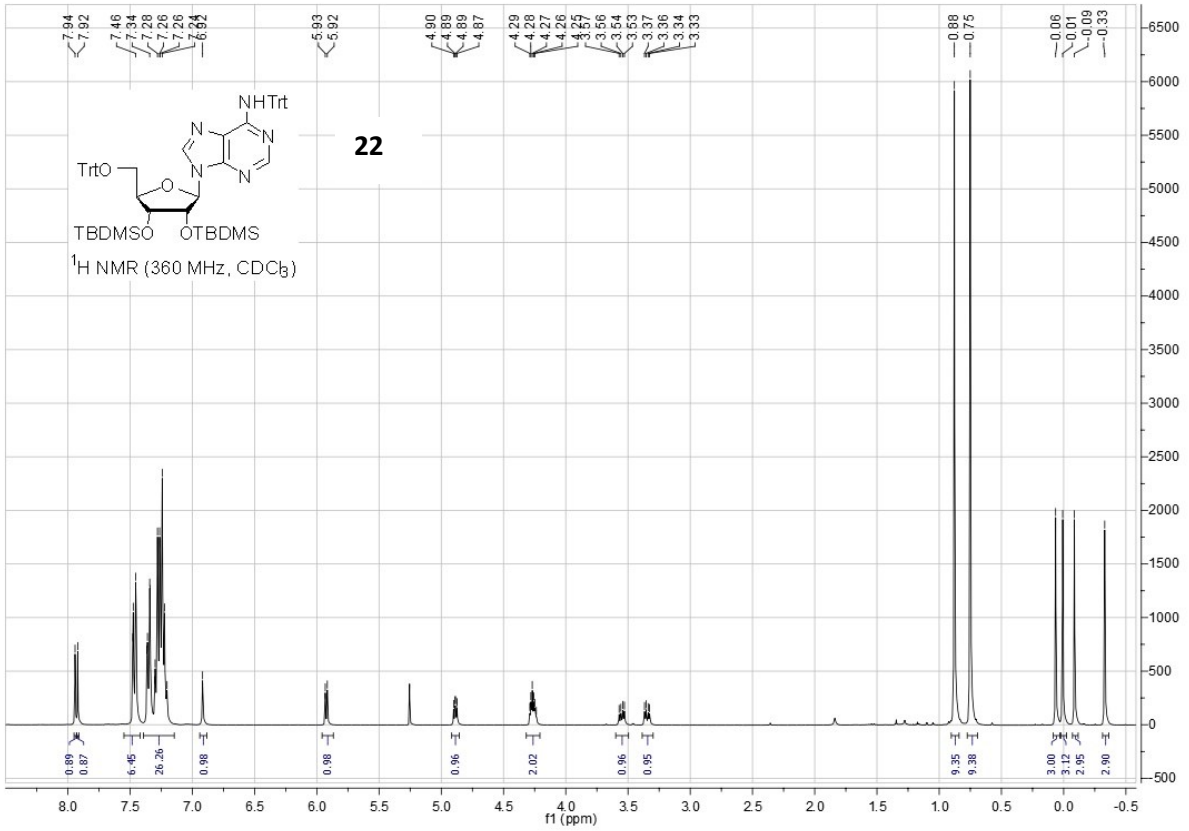

154.68
151.86
147.35
144.79
129.04
128.03
127.10
122.48
114.03
94.44
86.17
83.07
81.76
71.63
63.45
27.75
25.31
NHTrt
HO
16
^{13}C NMR (100 MHz, CDCl$_3$)
f1 (ppm)

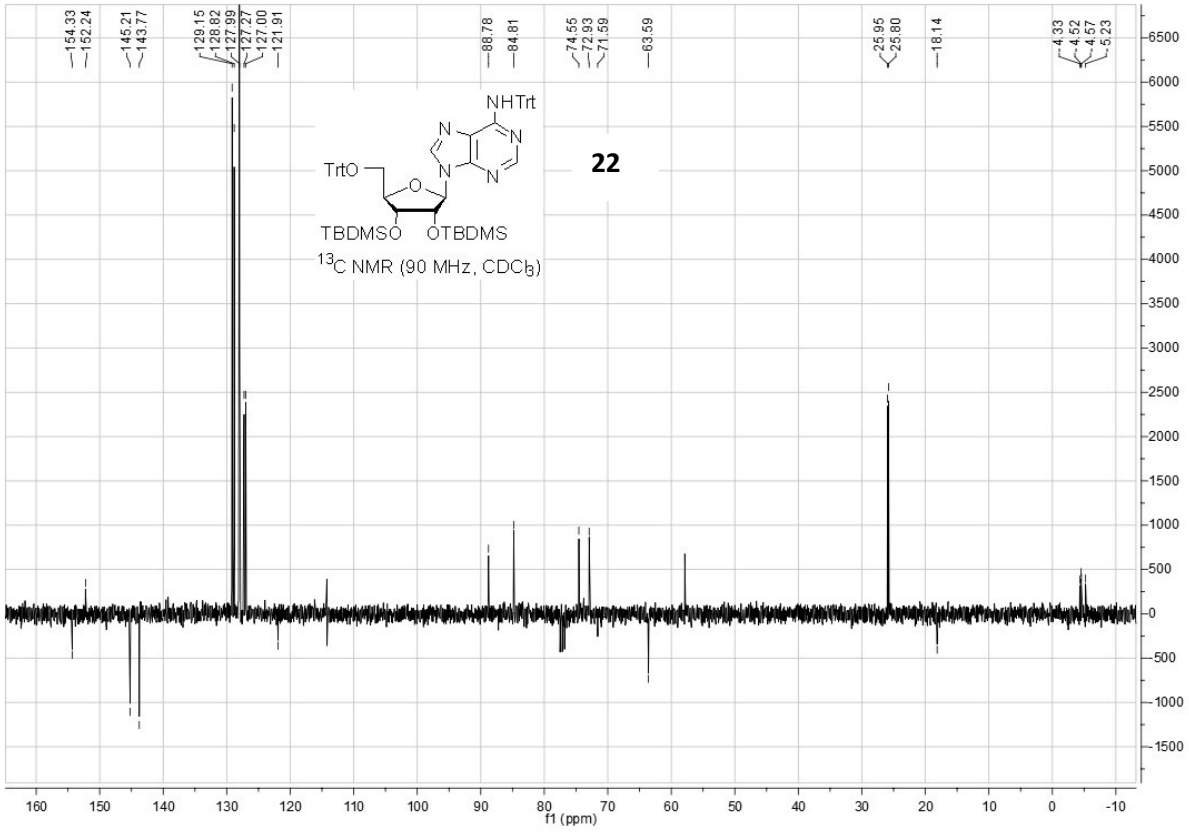

NHTrt
TsO
17
^{1}H NMR (400 MHz, CDCl$_3$)
f1 (ppm)

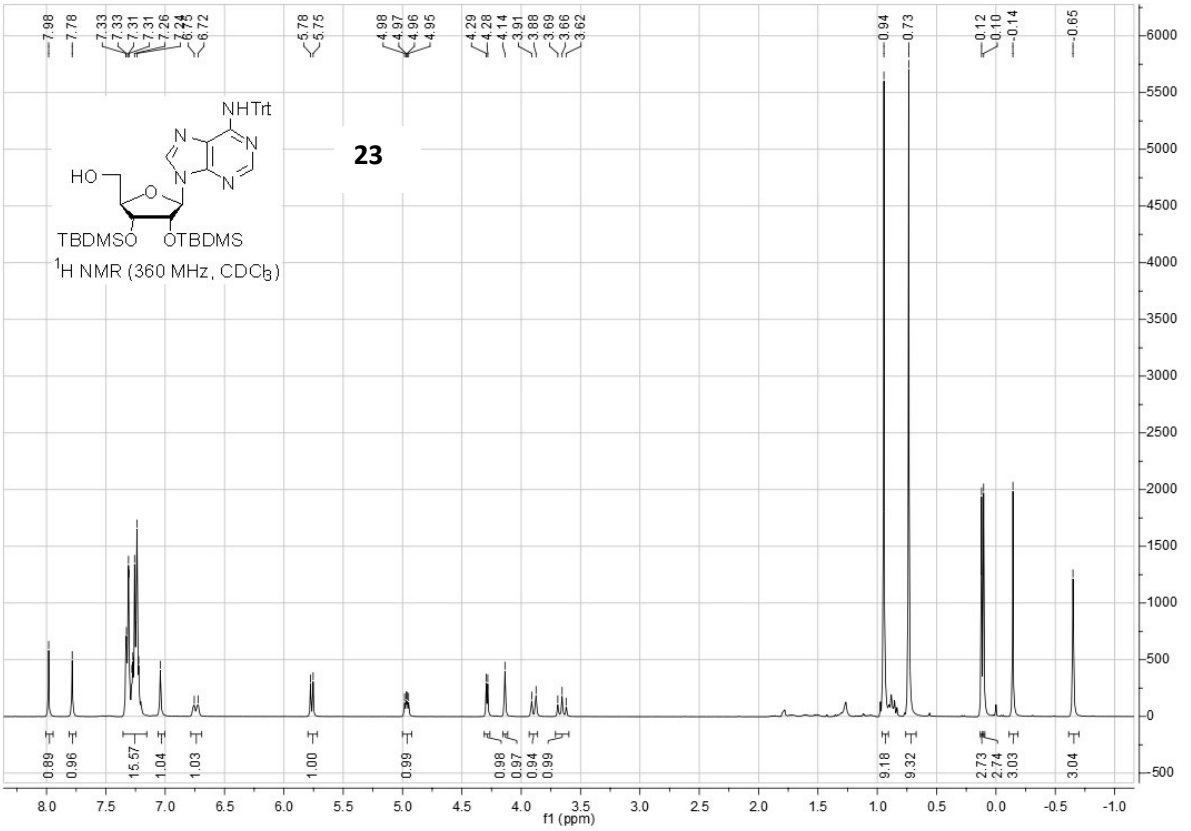

NHTrt
TsO
17
^{13}C NMR (100 MHz, CDCl$_3$)
f1 (ppm)

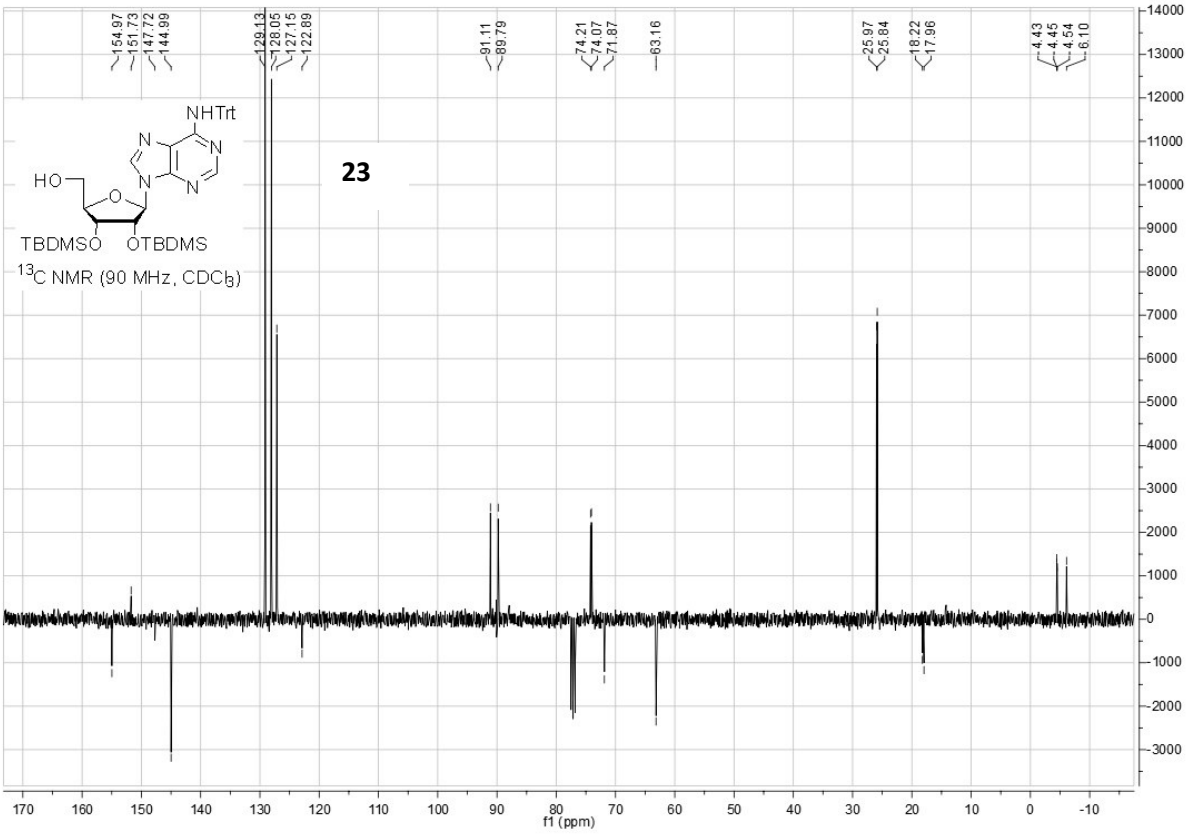

19
^{1}H NMR (400 MHz, DMSO-d$_6$)
f1 (ppm)

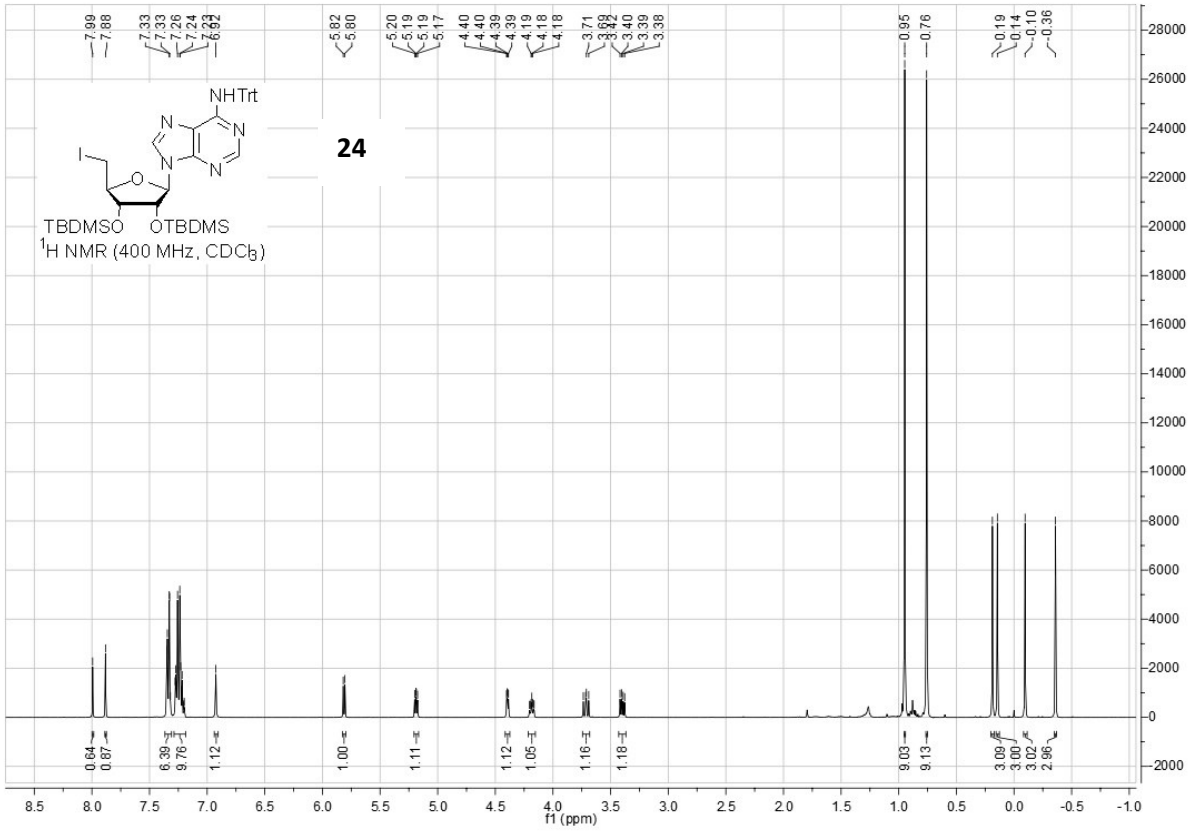

19
^{13}C NMR (90 MHz, DMSO-d$_6$)
f1 (ppm)

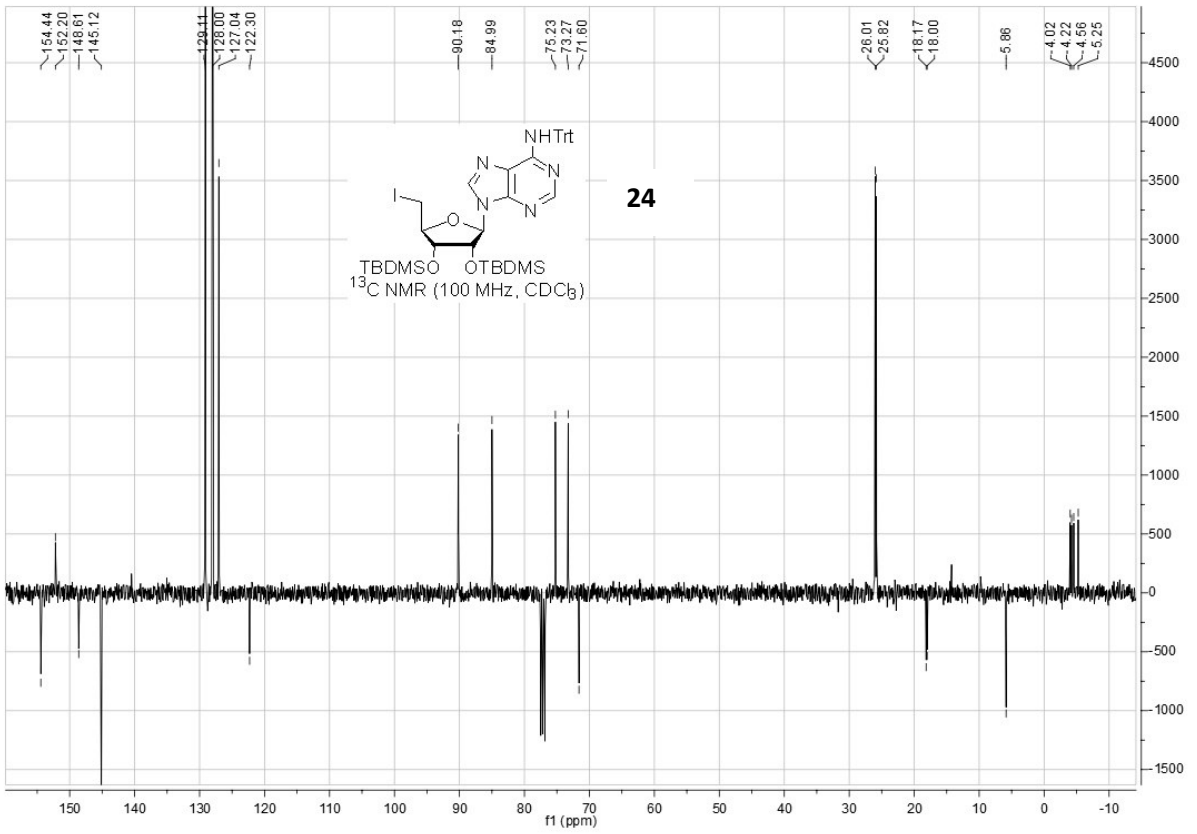

NHTrt
TrtO
TBDMSO
OTBDMS
22
^{1}H NMR (360 MHz, CDCl$_3$)
f1 (ppm)

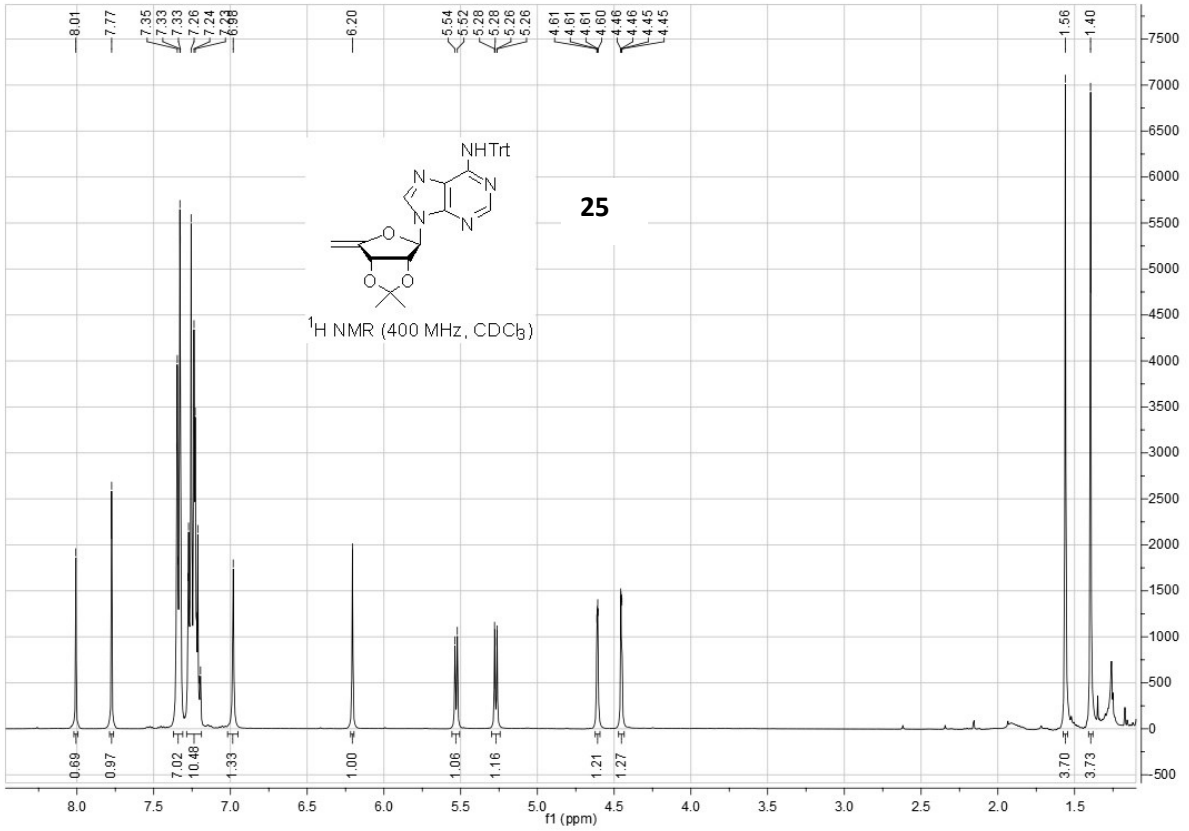

NHTrt
TrtO
TBDMSO
OTBDMS
22
^{13}C NMR (90 MHz, CDCl$_3$)
f1 (ppm)

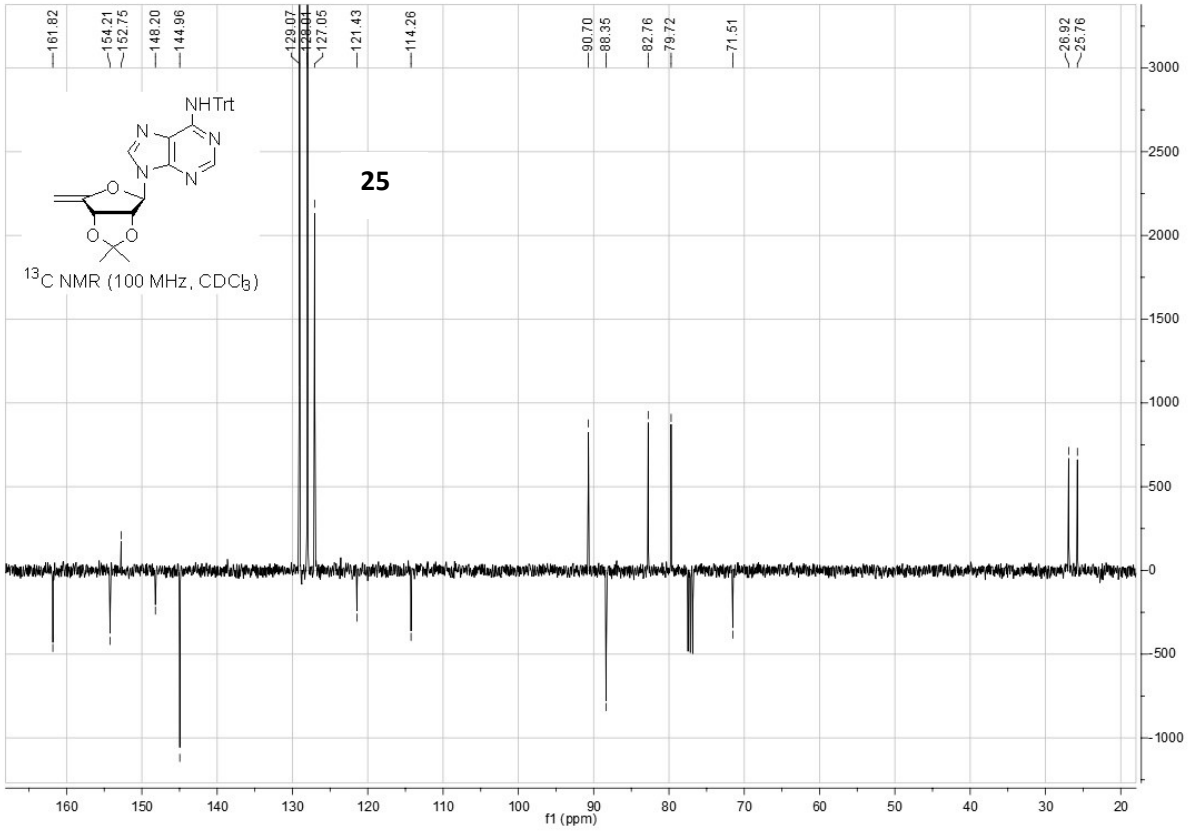

23
NHTrt
HO
TBDMSO
OTBDMS
^{1}H NMR (360 MHz, CDCl$_3$)
f1 (ppm)

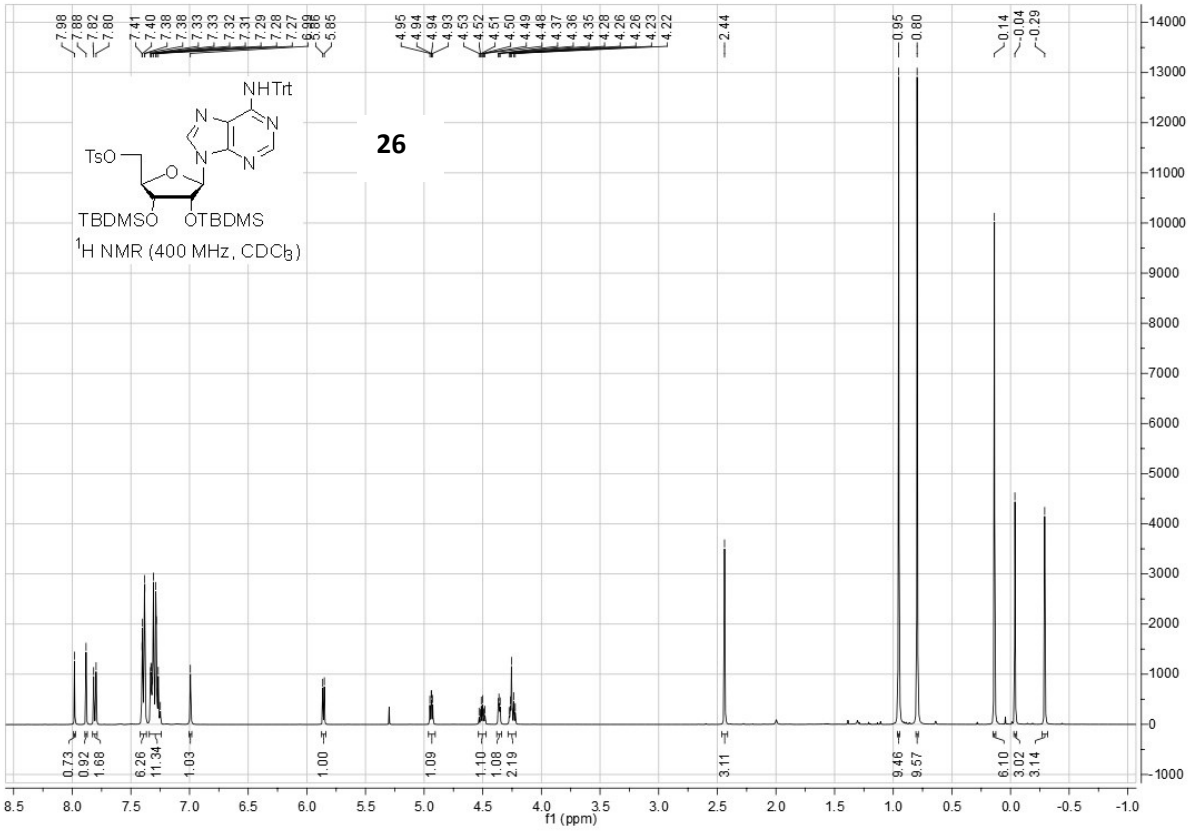

23
NHTrt
HO
TBDMSO
OTBDMS
^{13}C NMR (90 MHz, CDCl$_3$)
f1 (ppm)

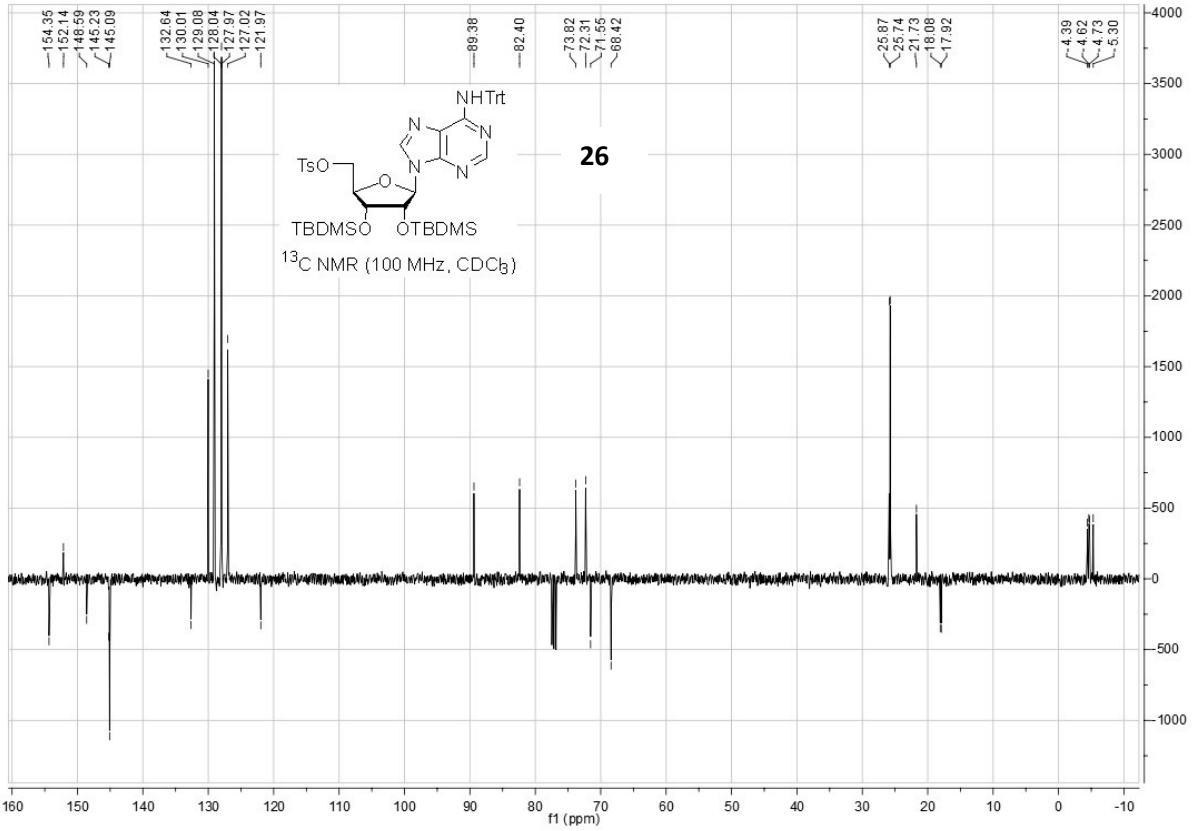

NHTrt
TBDMSO OTBDMS
24
^{1}H NMR (400 MHz, CDCl$_3$)
f1 (ppm)

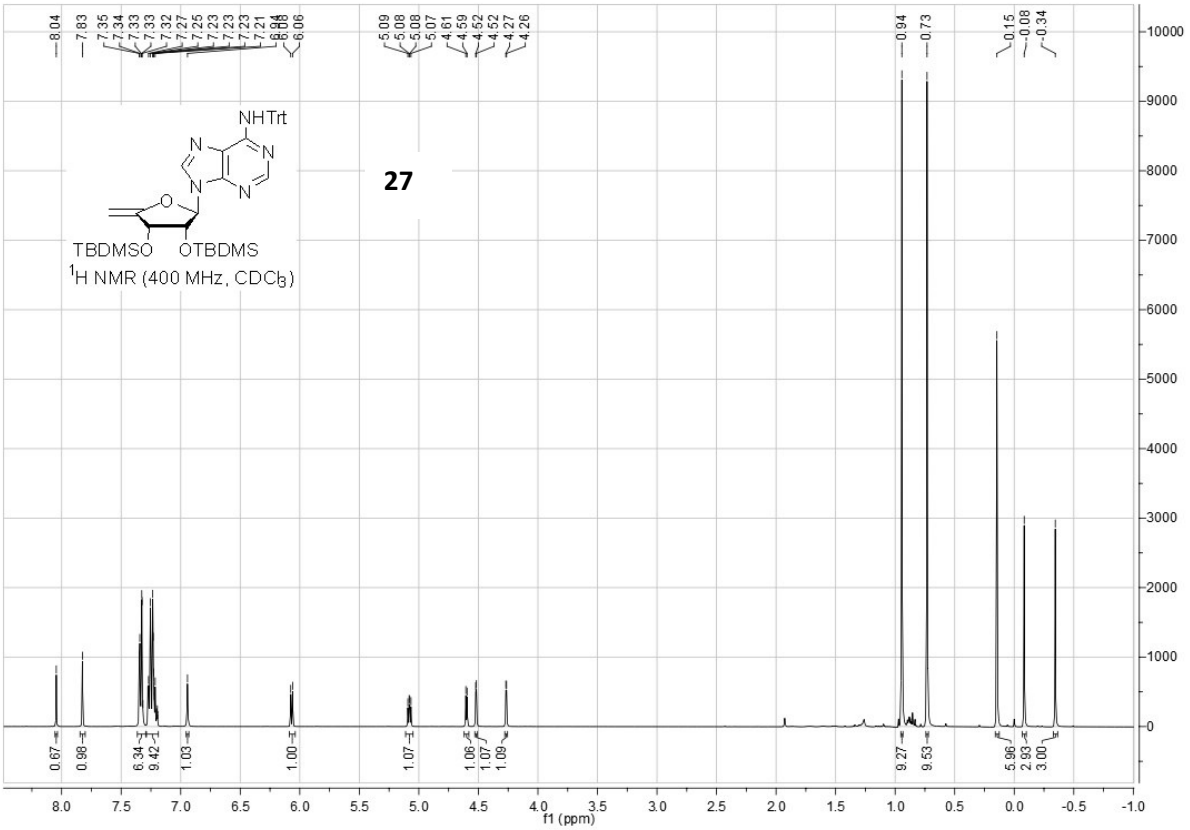

NHTrt
TBDMSO OTBDMS
24
^{13}C NMR (100 MHz, CDCl$_3$)
f1 (ppm)

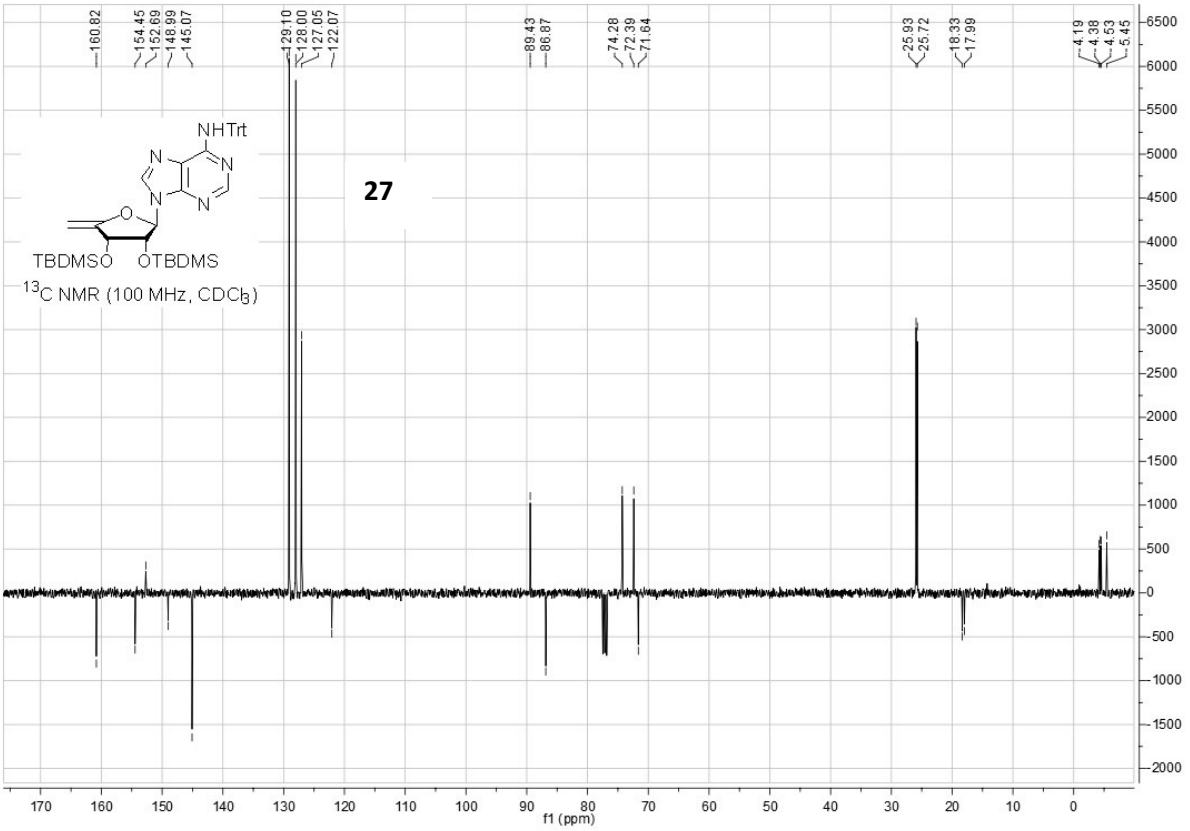

8.01
7.77
7.35
7.33
7.33
7.26
7.24
7.23
6.98
6.20
5.54
5.52
5.28
5.28
5.26
5.26
4.61
4.61
4.61
4.60
4.46
4.46
4.45
4.45
1.56
1.40
NHTrt
25
^{1}H NMR (400 MHz, CDCl$_3$)
0.69
0.97
7.02
10.48
1.33
1.00
1.06
1.16
1.21
1.27
3.70
3.73
f1 (ppm)

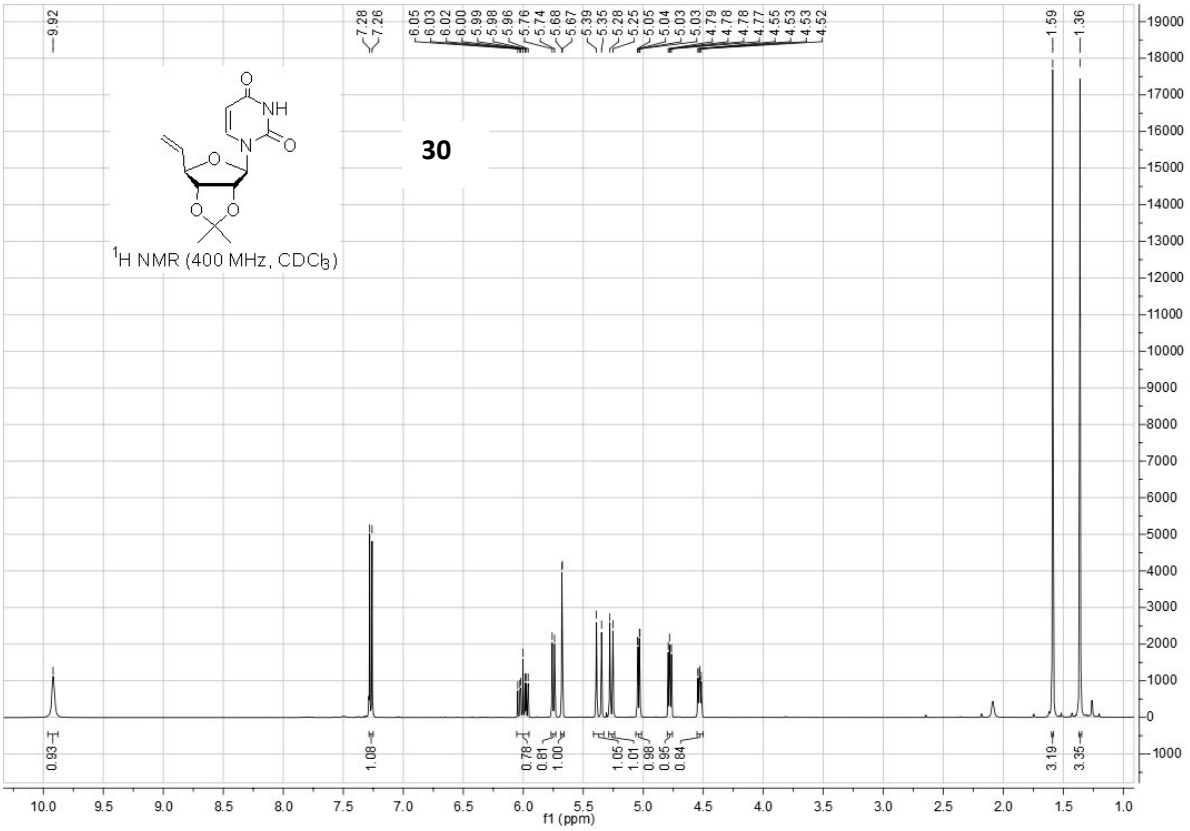

161.82
154.21
152.75
148.20
144.96
129.07
128.01
127.05
121.43
114.26
90.70
88.35
82.76
79.72
71.51
26.92
25.76
NHTrt
25
^{13}C NMR (100 MHz, CDCl$_3$)
f1 (ppm)

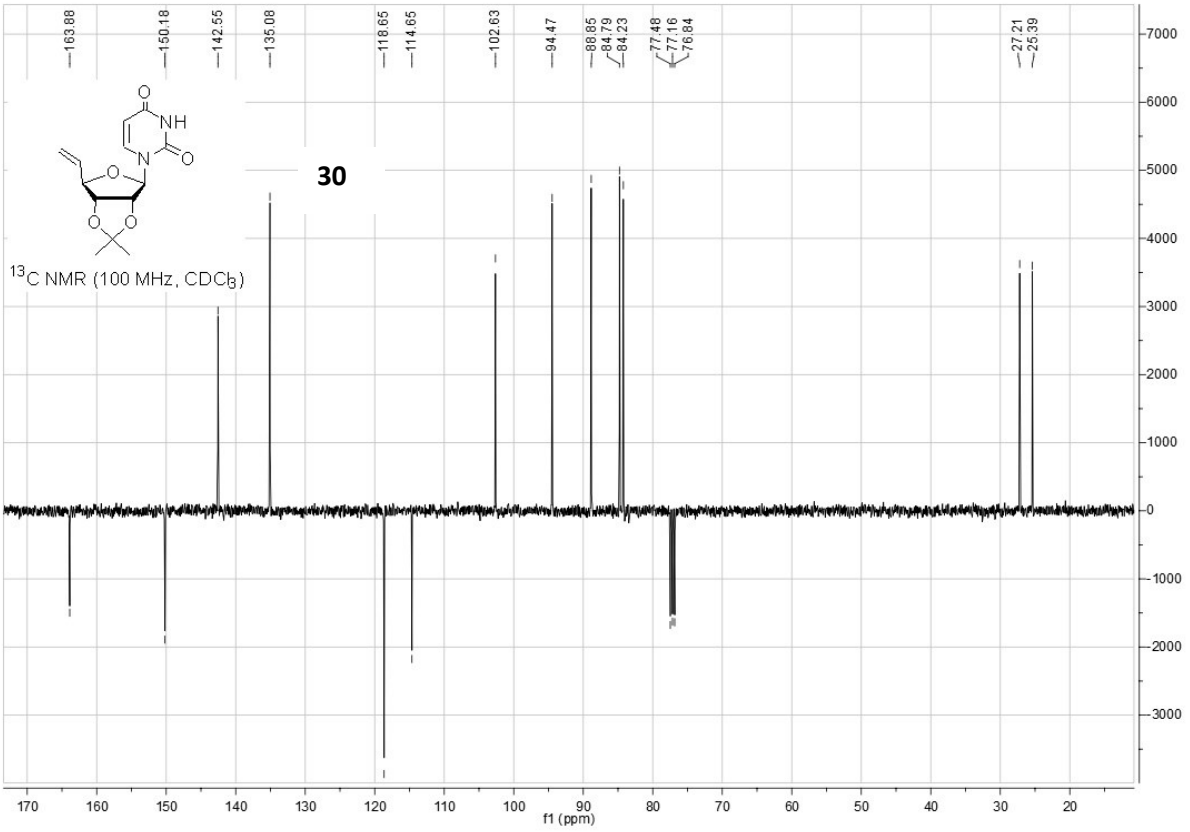

NHTrt
TsO
TBDMSO
OTBDMS
26
^{1}H NMR (400 MHz, CDCl$_3$)
f1 (ppm)

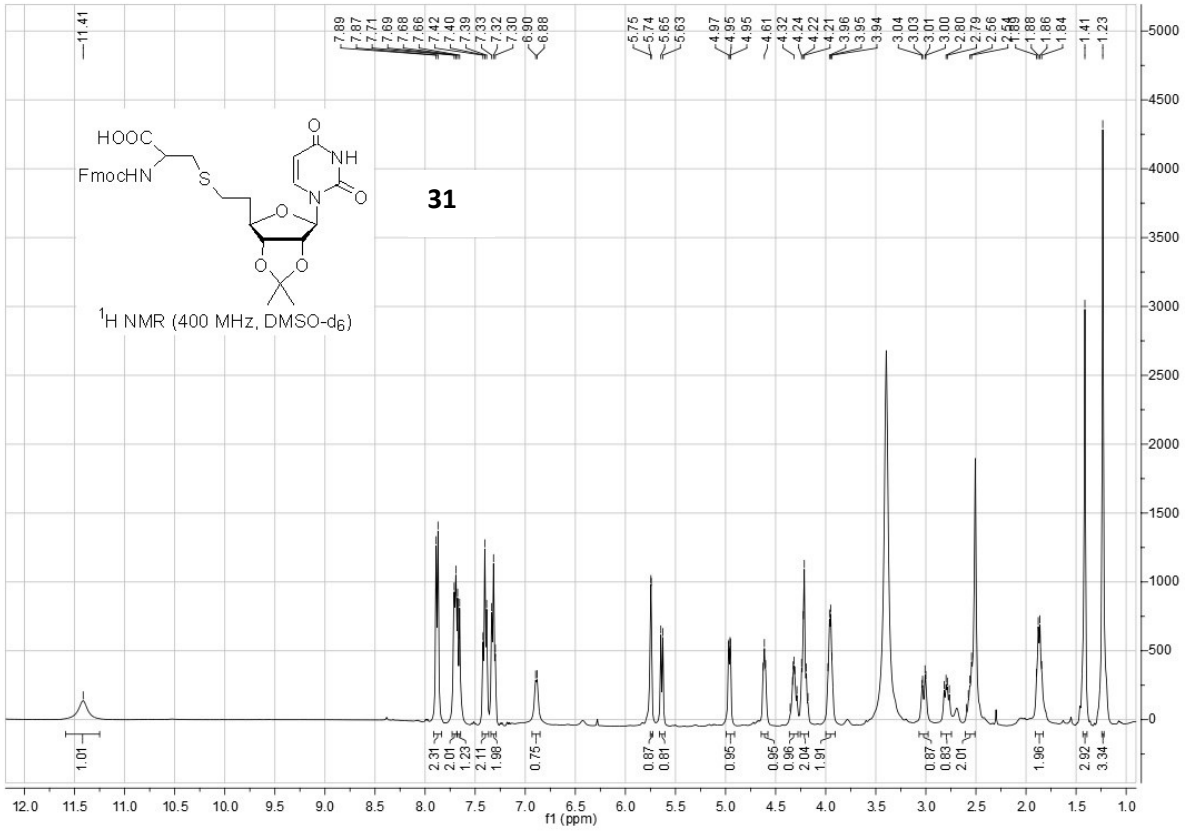

NHTrt
TsO
TBDMSO
OTBDMS
26
^{13}C NMR (100 MHz, CDCl$_3$)
f1 (ppm)

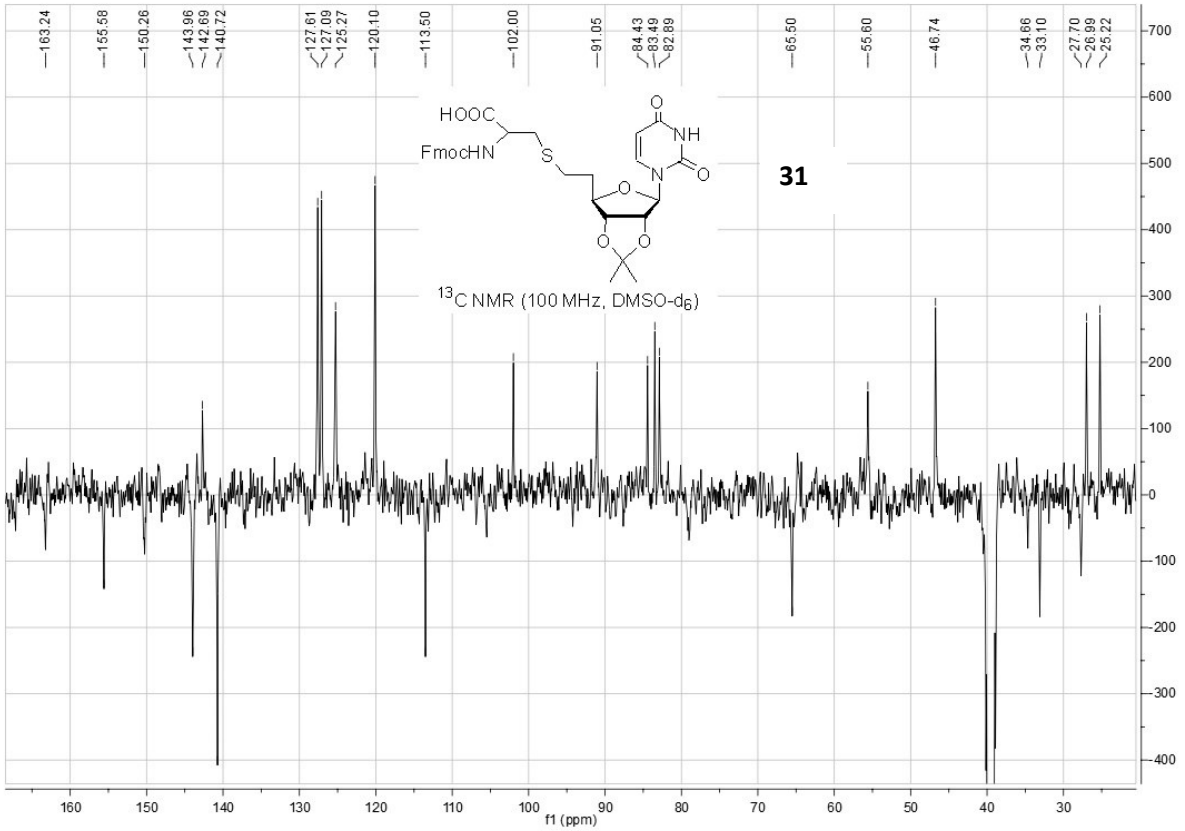

8.04
7.83
7.35
7.34
7.33
7.33
7.32
7.27
7.25
7.23
7.23
7.23
7.21
6.08
6.06
5.09
5.08
5.08
5.07
4.61
4.59
4.52
4.52
4.27
4.26
0.94
0.73
0.15
0.08
0.34
NHTrt
TBDMSO
OTBDMS
27
^{1}H NMR (400 MHz, CDCl$_3$)
0.67
0.98
6.34
9.42
1.03
1.00
1.07
1.06
1.07
1.09
9.27
9.53
5.96
2.93
3.00
f1 (ppm)

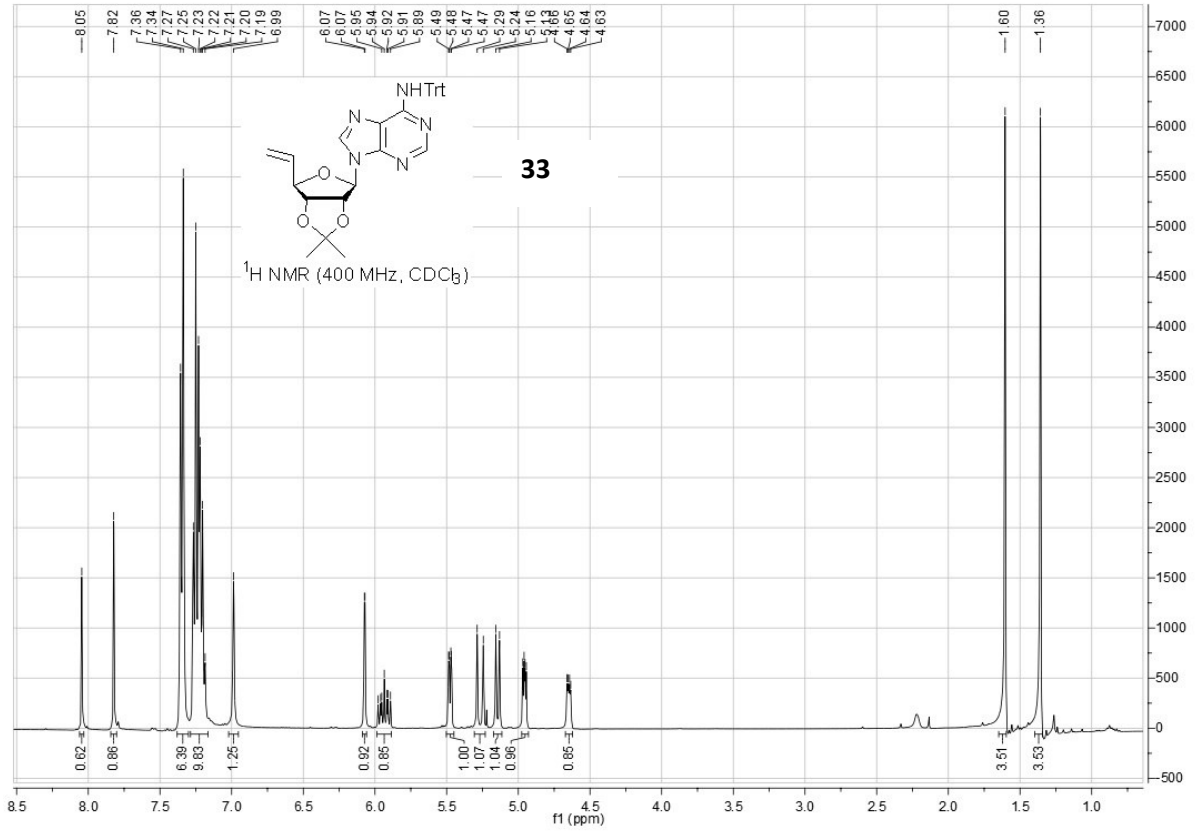

160.82
154.45
152.69
148.99
145.07
129.10
128.00
127.05
122.07
89.43
86.87
74.28
72.39
71.64
25.93
25.72
18.33
17.99
4.19
4.38
4.53
5.45
NHTrt
TBDMSO
OTBDMS
27
^{13}C NMR (100 MHz, CDCl$_3$)
f1 (ppm)

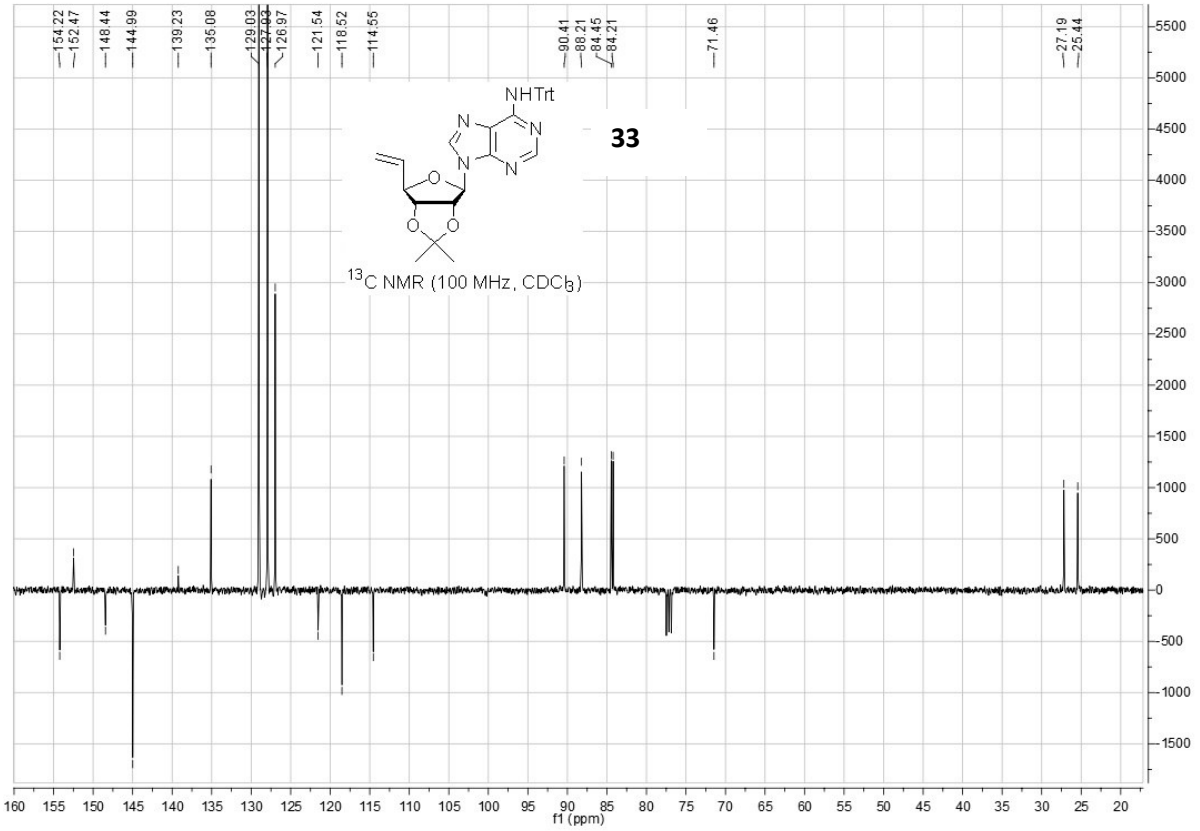

30
^{1}H NMR (400 MHz, CDCl$_3$)
f1 (ppm)

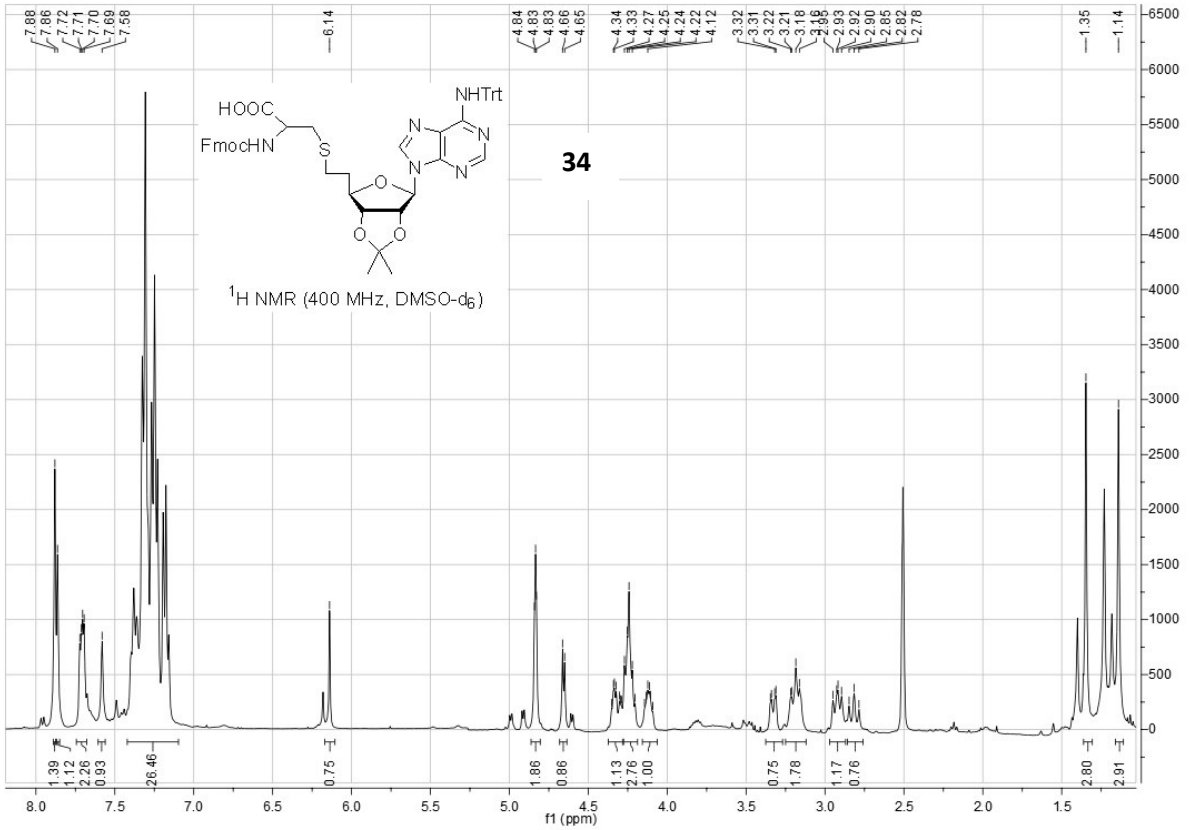

30
^{13}C NMR (100 MHz, CDCl$_3$)
f1 (ppm)

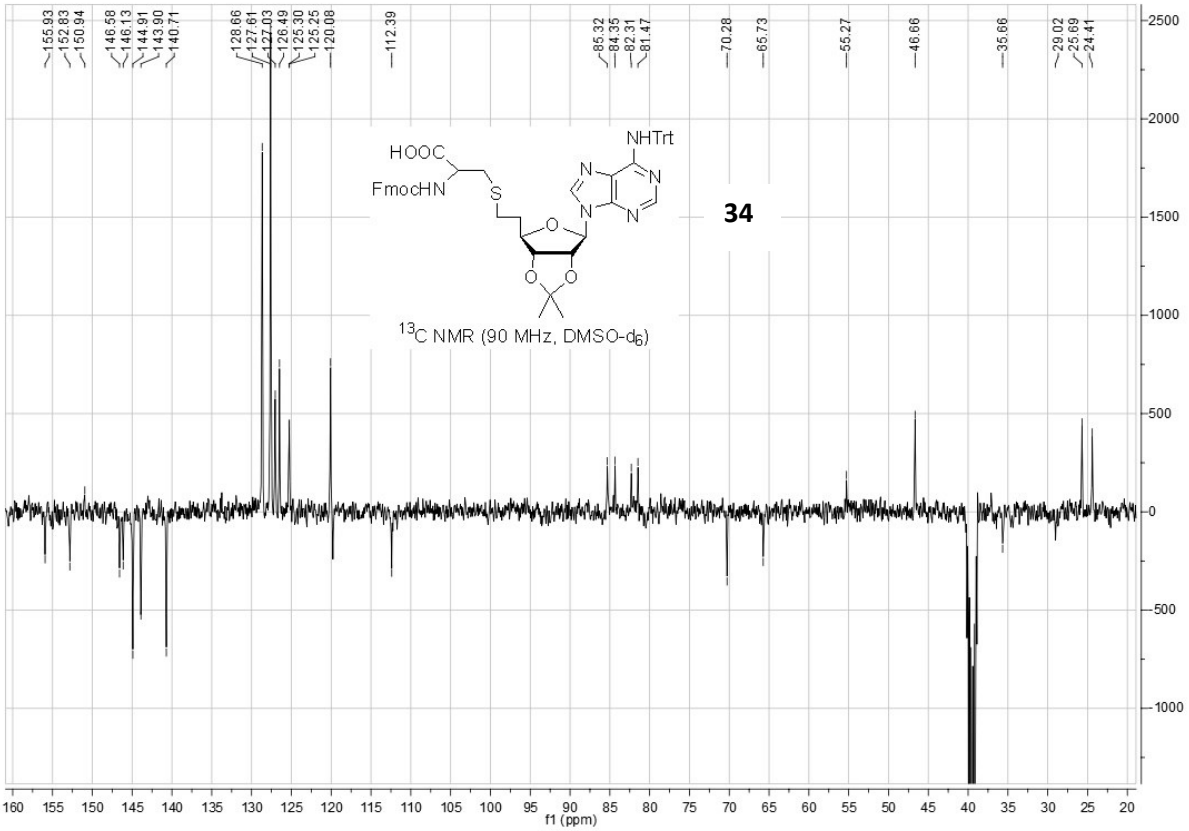

HOOC
FmocHN
S
NH
O
N
O
O
O
O
31
^{1}H NMR (400 MHz, DMSO-d$_6$)
f1 (ppm)

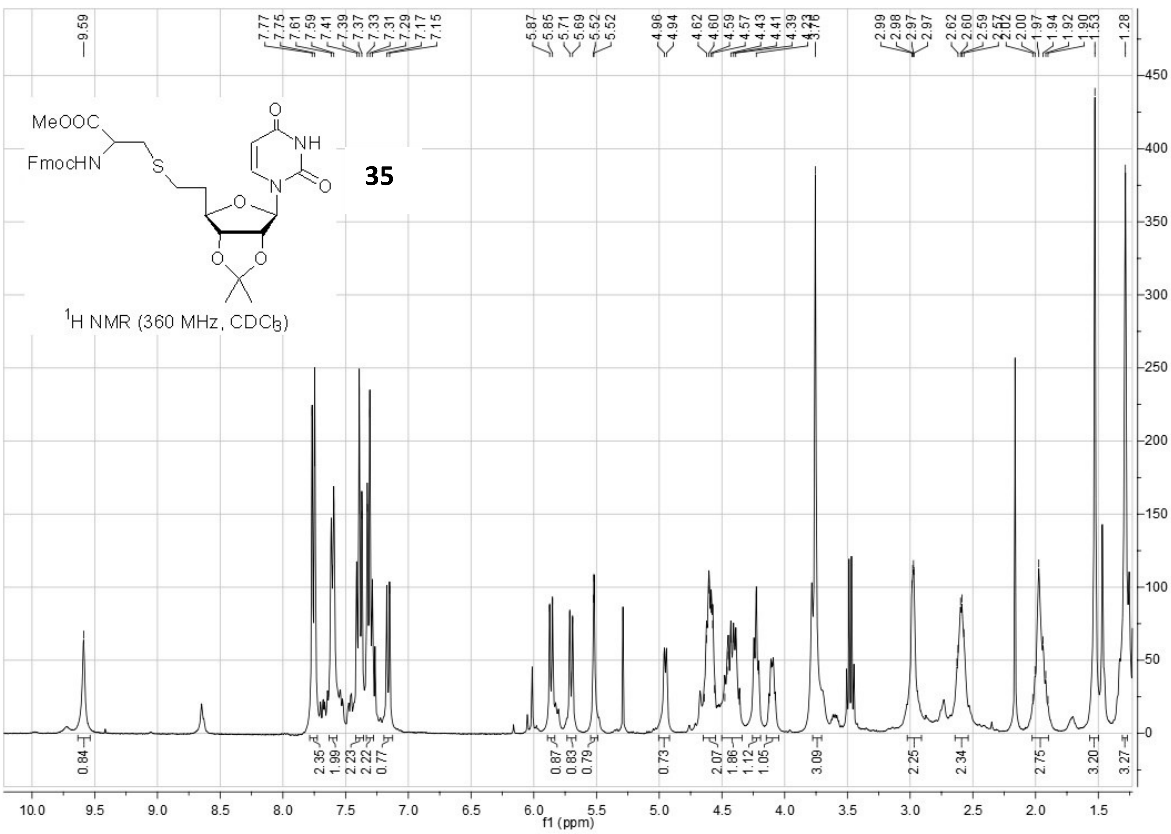

HOOC
FmocHN
S
NH
O
N
O
O
O
O
31
^{13}C NMR (100 MHz, DMSO-d$_6$)
f1 (ppm)

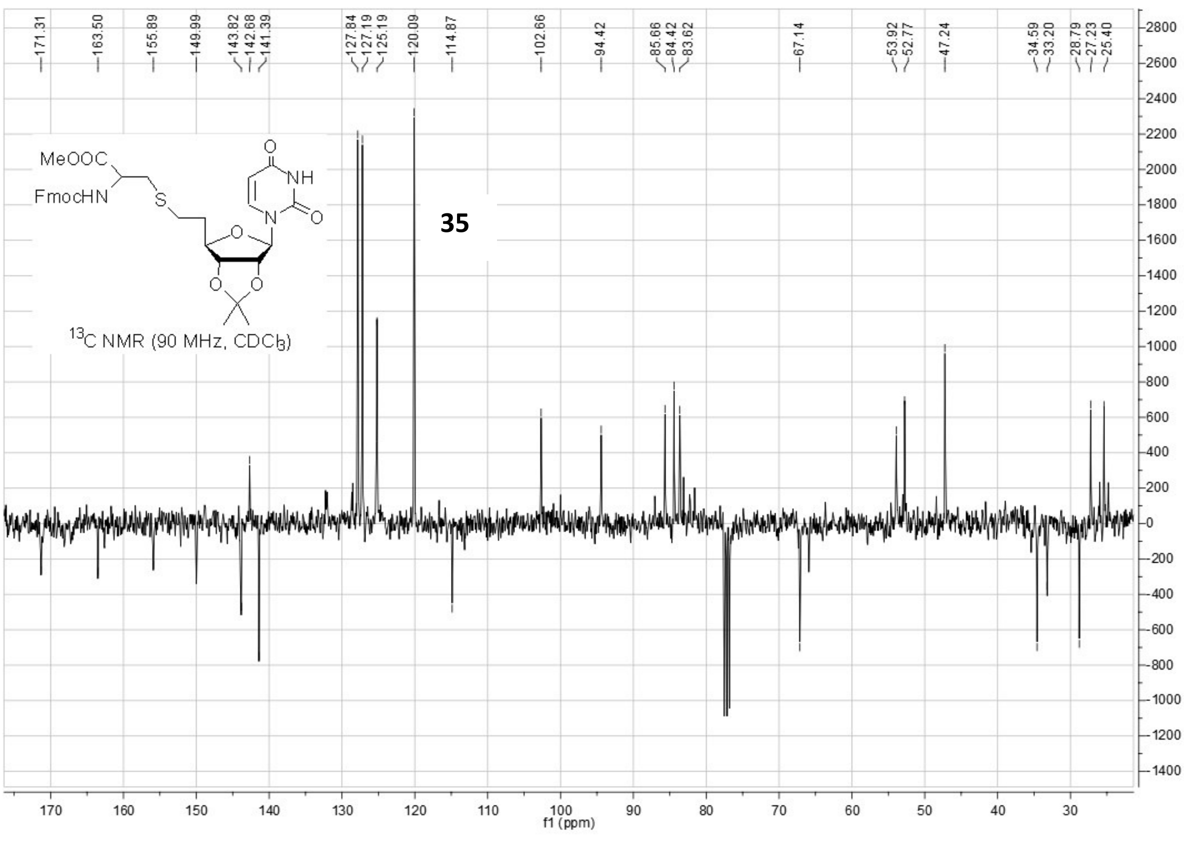

8.05
7.82
7.36
7.34
7.27
7.25
7.23
7.22
7.21
7.20
7.19
6.99
6.07
6.07
5.95
5.94
5.92
5.91
5.89
5.49
5.48
5.47
5.47
5.29
5.24
5.16
4.65
4.64
4.63
1.60
1.36
NHTrt
33
^{1}H NMR (400 MHz, CDCl$_3$)
0.62
0.86
6.39
9.83
1.25
0.92
0.85
1.00
1.07
1.04
0.96
0.85
3.51
3.53
f1 (ppm)

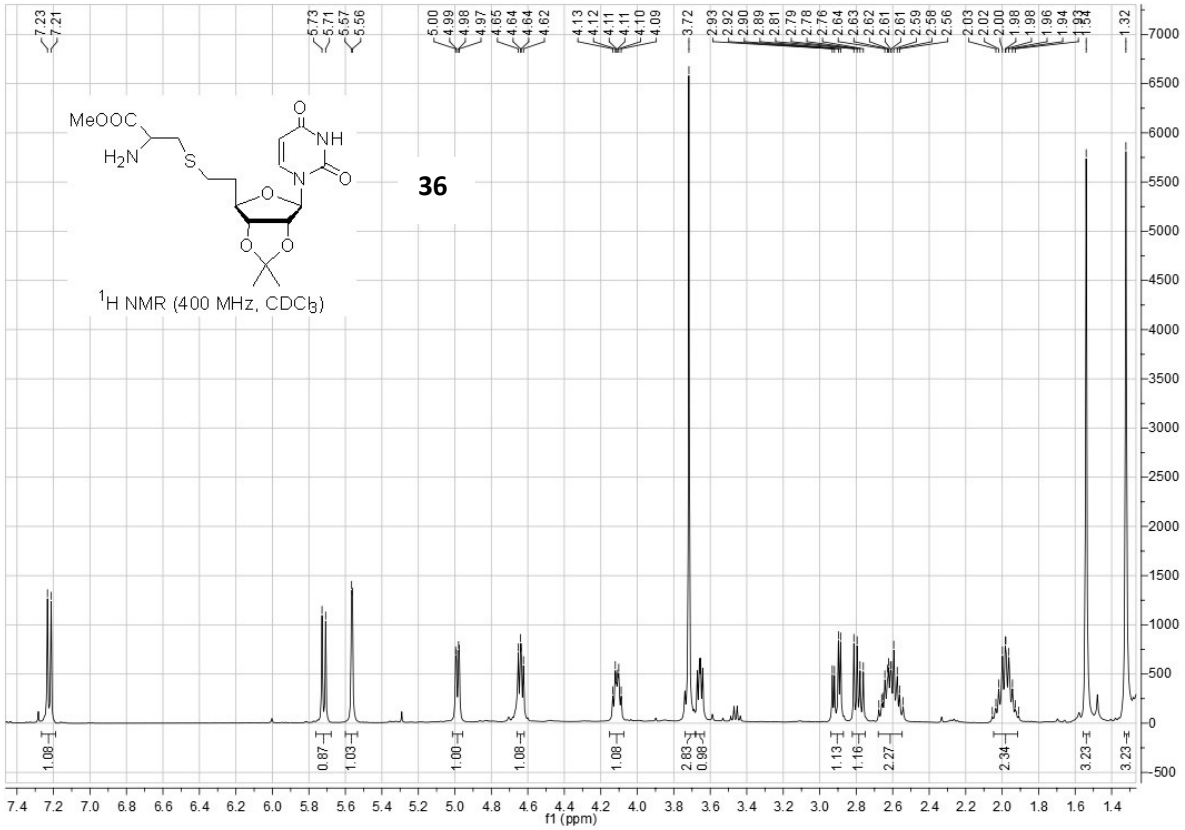

154.22
152.47
148.44
144.99
139.23
135.08
129.03
127.03
126.97
121.54
118.52
114.55
90.41
88.21
84.45
84.21
71.46
27.19
25.44
NHTrt
33
^{13}C NMR (100 MHz, CDCl$_3$)
f1 (ppm)

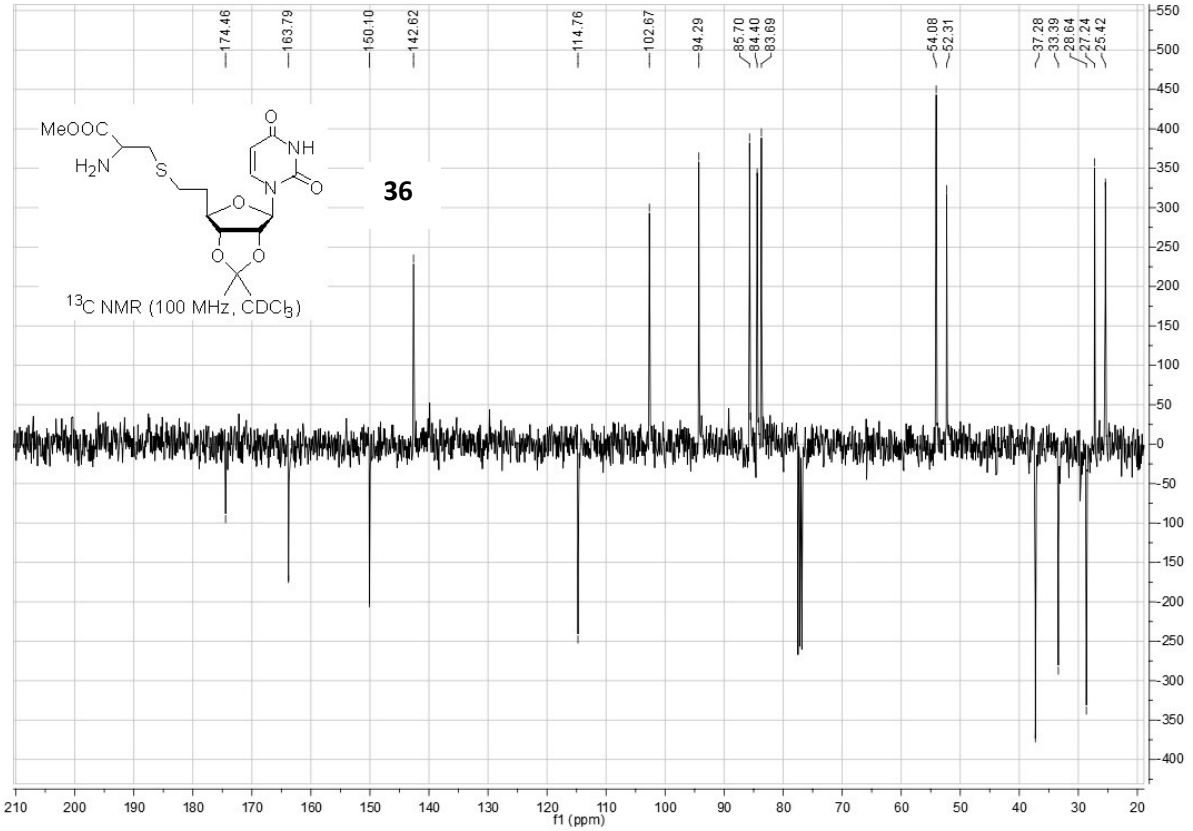

HOOC
FmocHN
NHTrt
34
^{1}H NMR (400 MHz, DMSO-d$_6$)
f1 (ppm)

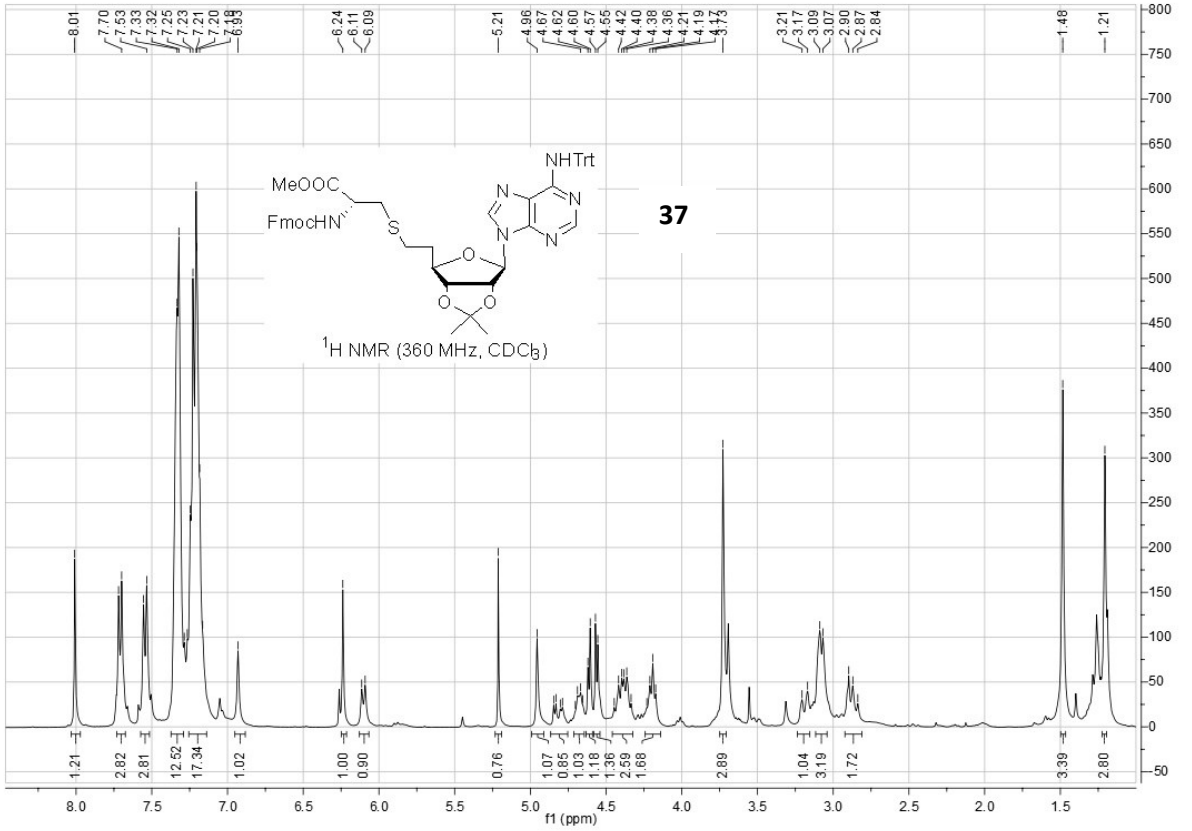

HOOC
FmocHN
NHTrt
34
^{13}C NMR (90 MHz, DMSO-d$_6$)
f1 (ppm)

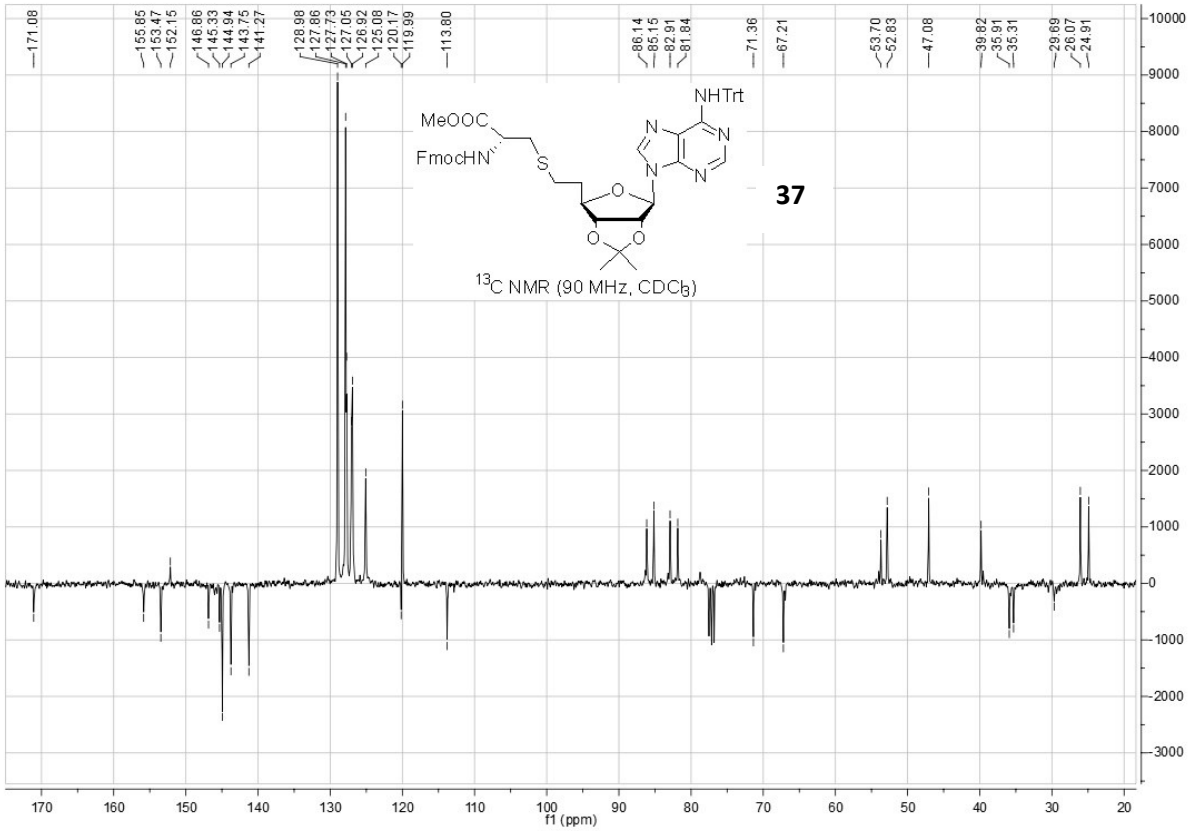

MeOOC
FmocHN
S
O
NH
N
O
O
O
O
35
^{1}H NMR (360 MHz, CDCl$_3$)
f1 (ppm)

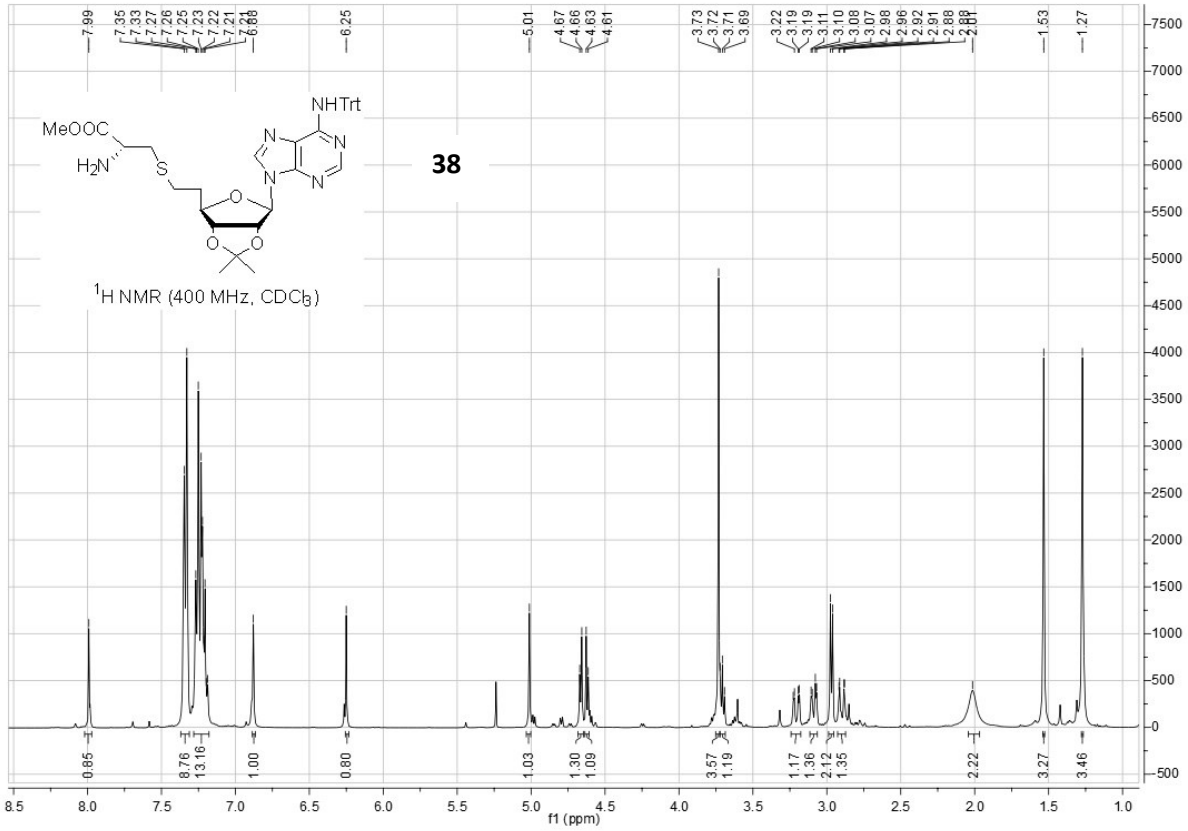

MeOOC
FmocHN
S
O
NH
N
O
O
O
O
35
^{13}C NMR (90 MHz, CDCl$_3$)
f1 (ppm)

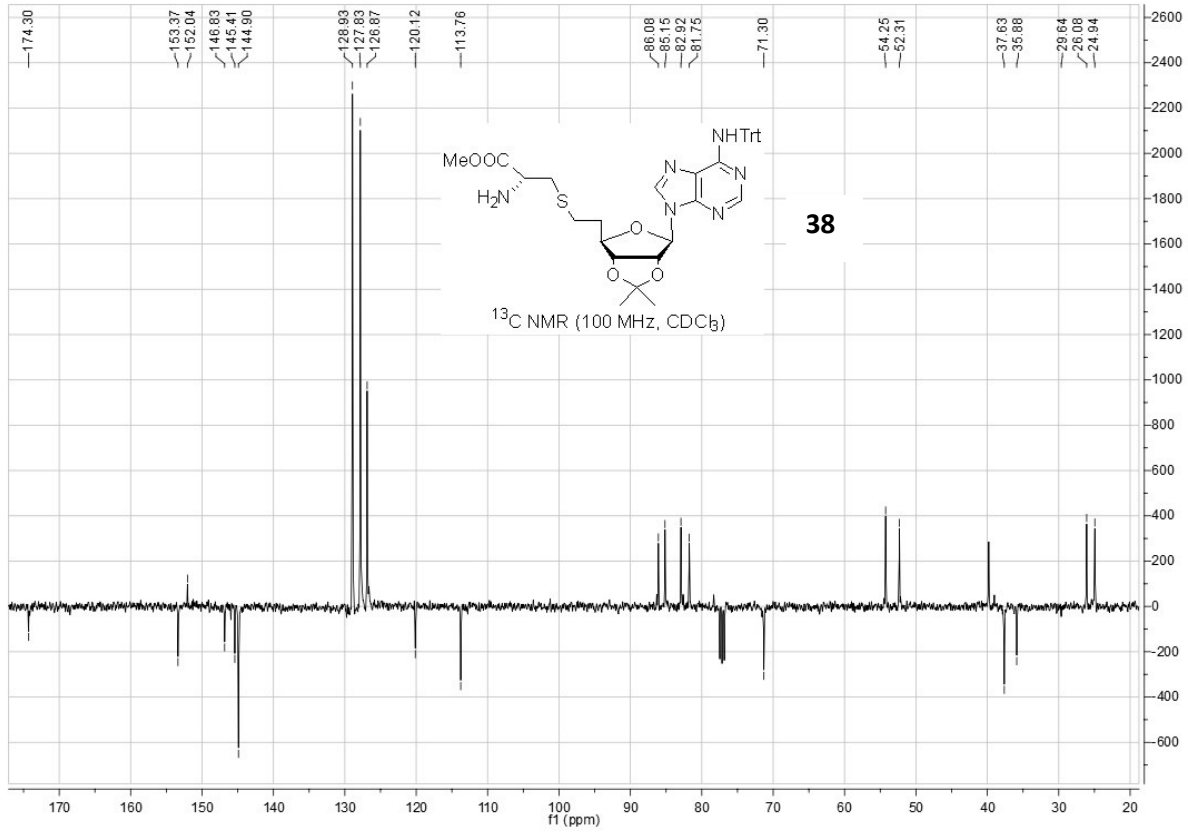

36
^{1}H NMR (400 MHz, CDCl$_3$)
f1 (ppm)

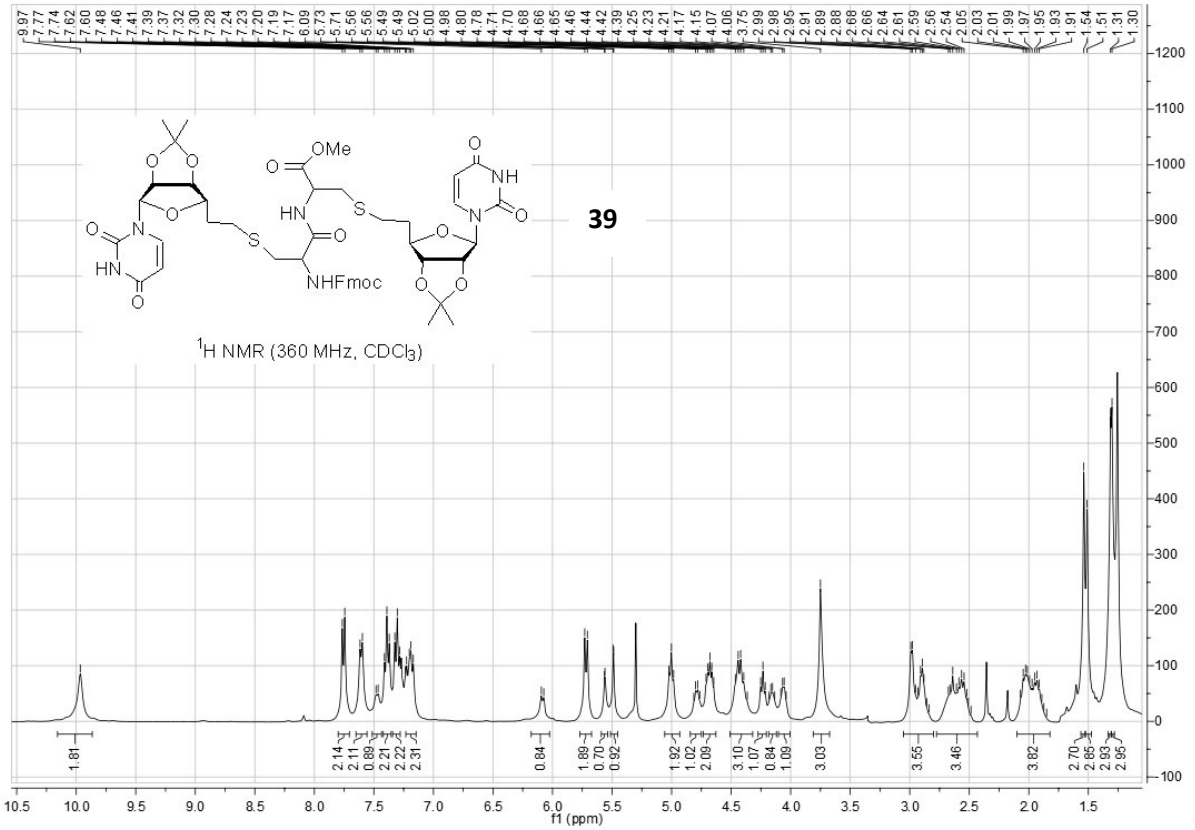

36
^{13}C NMR (100 MHz, CDCl$_3$)
f1 (ppm)

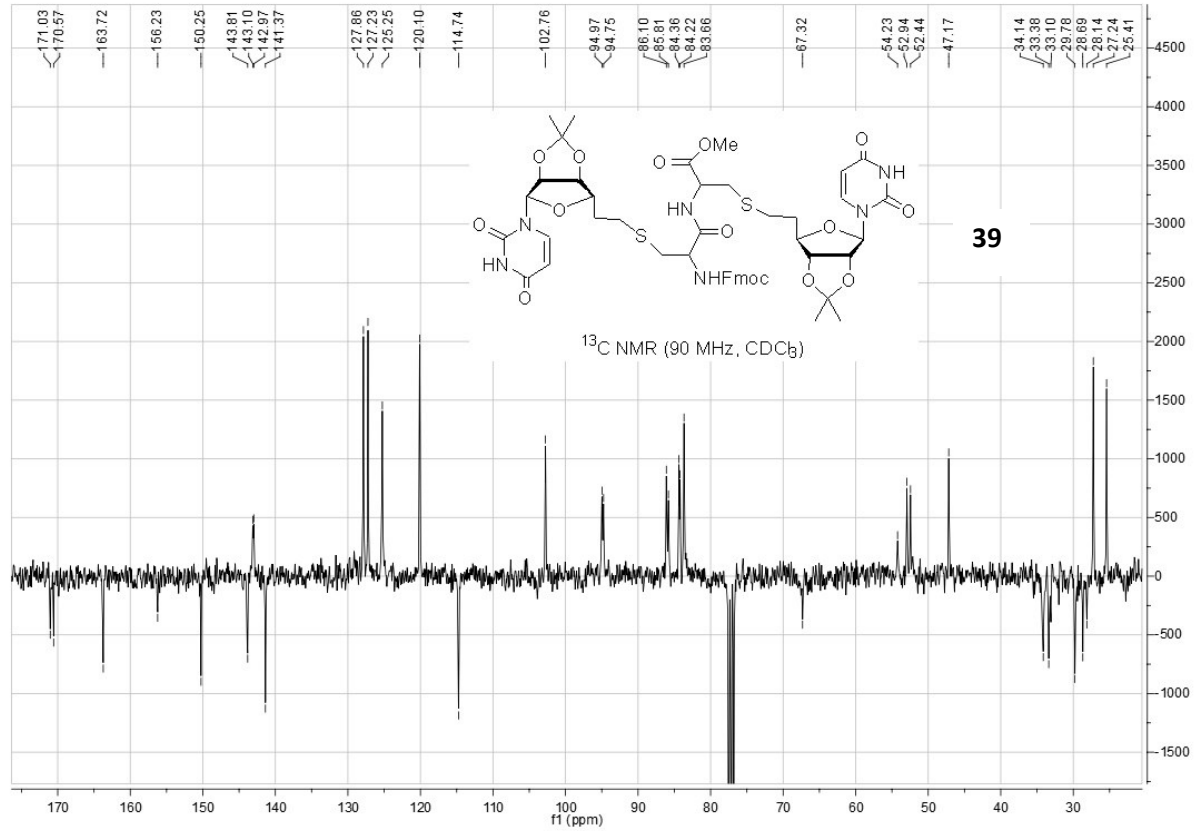

MeOOC
FmocHN
NHTrt
37
^{1}H NMR (360 MHz, CDCl$_3$)

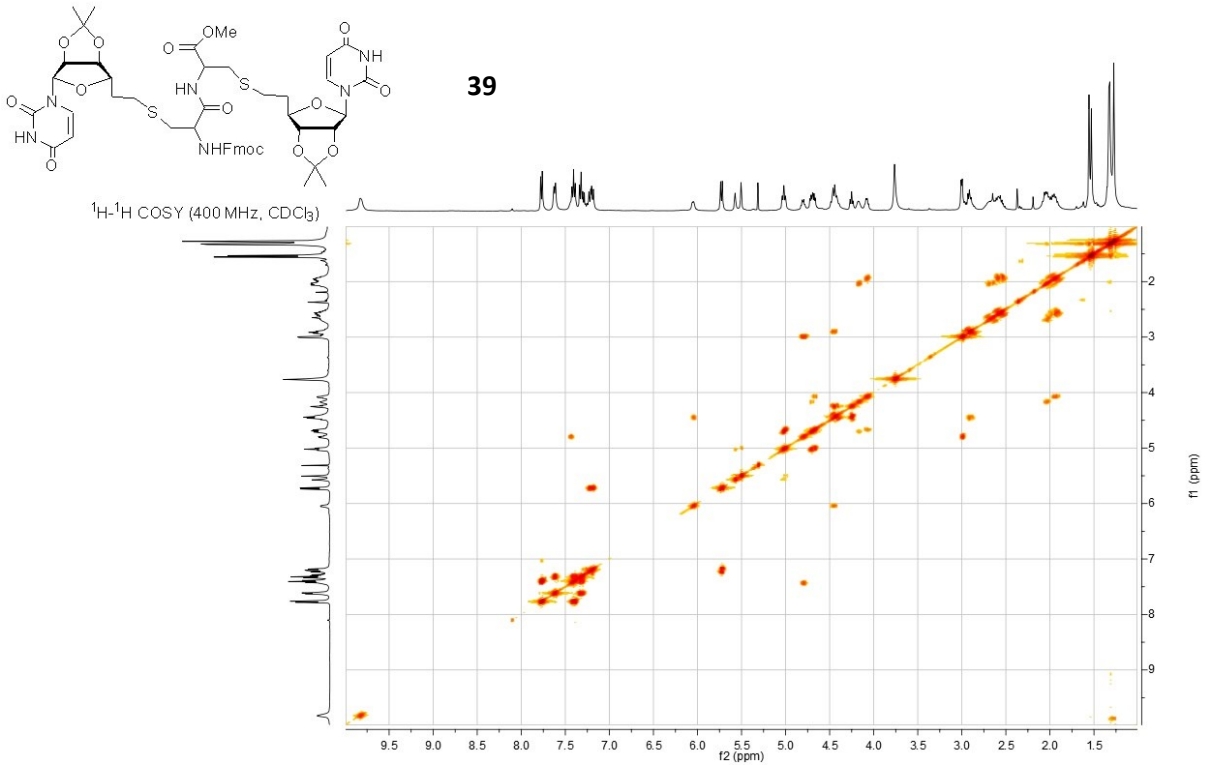

MeOOC
FmocHN
NHTrt
37
^{13}C NMR (90 MHz, CDCl$_3$)

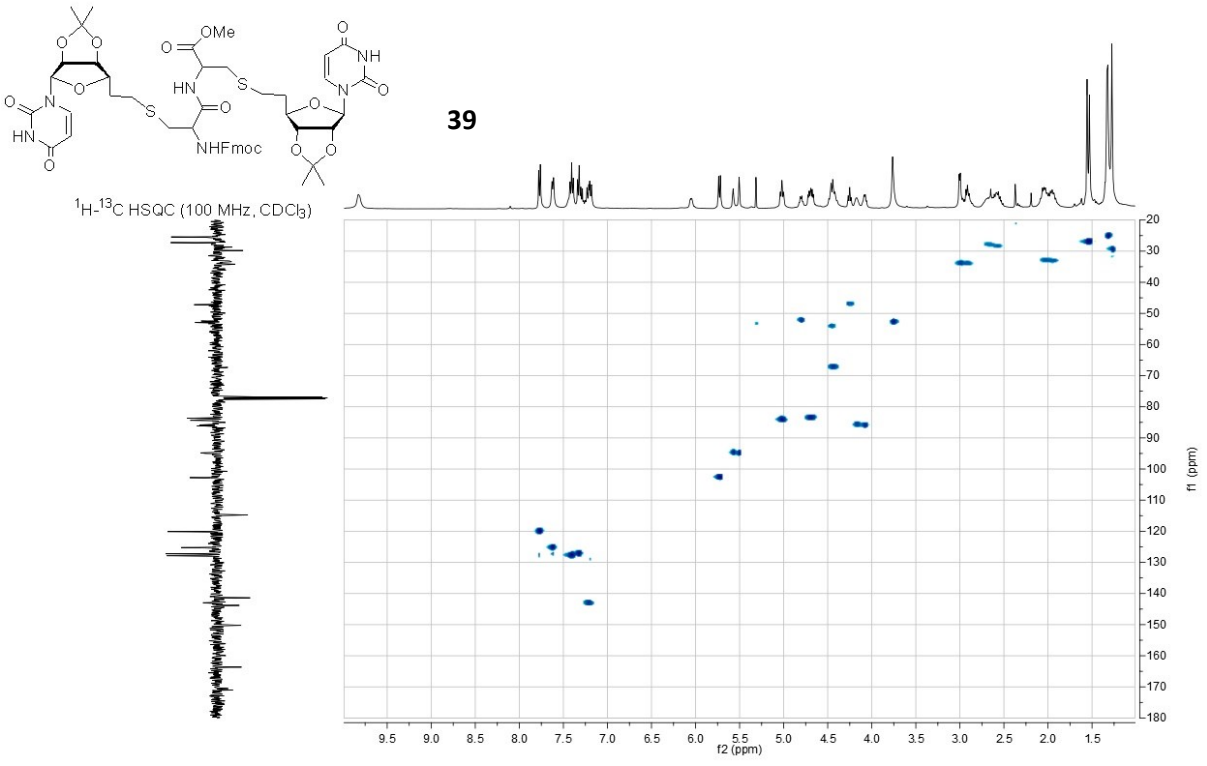

38
^{1}H NMR (400 MHz, CDCl$_3$)
f1 (ppm)

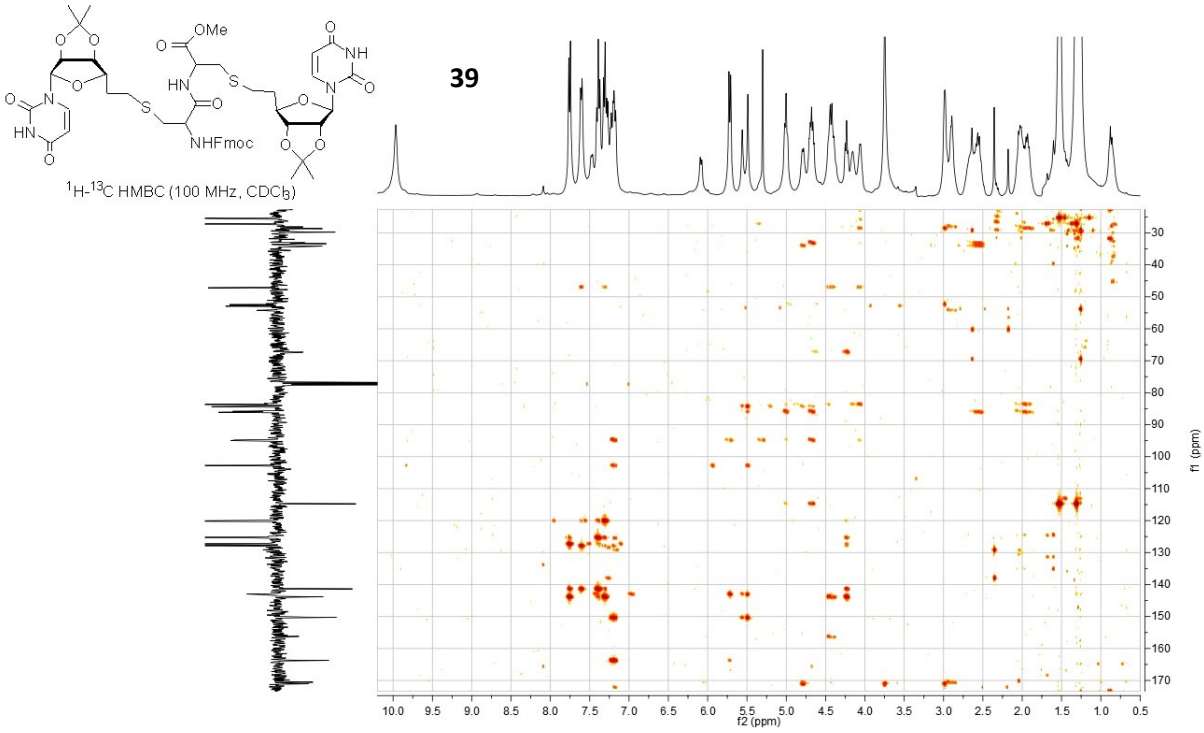

38
^{13}C NMR (100 MHz, CDCl$_3$)
f1 (ppm)

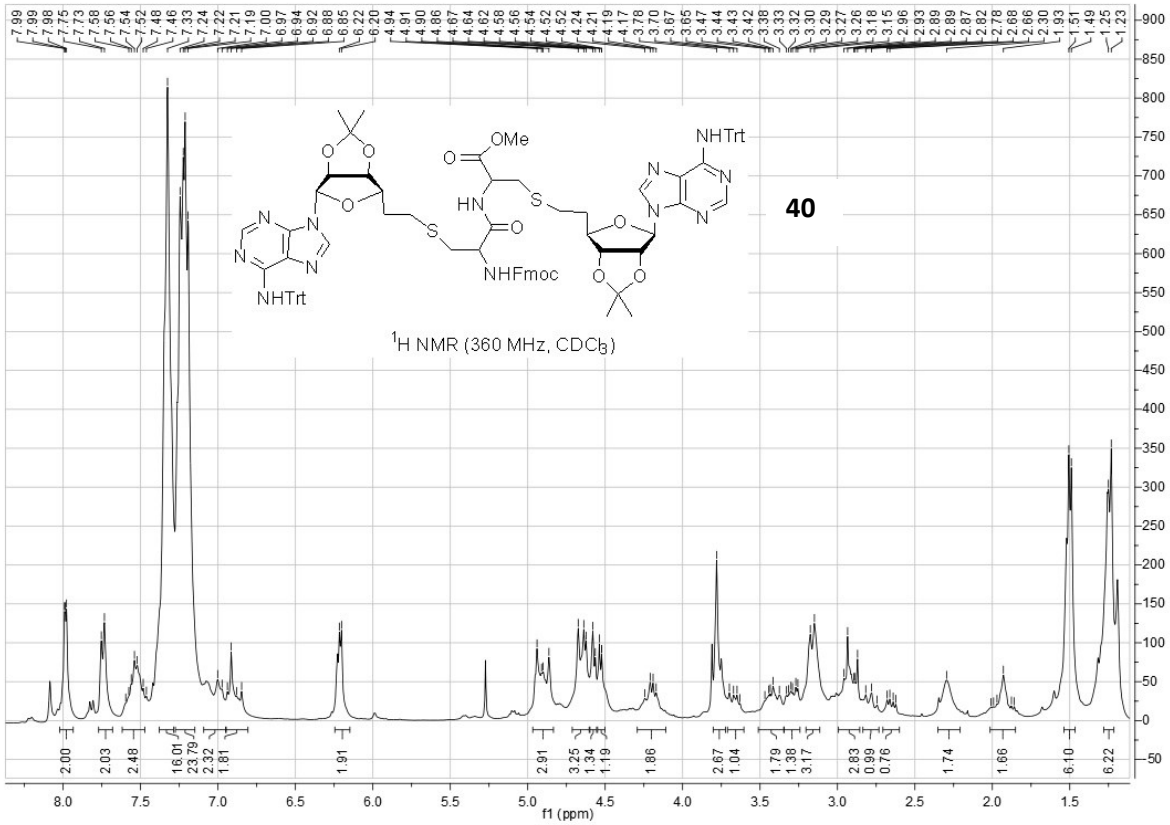

39
^{1}H NMR (360 MHz, CDCl$_3$)
f1 (ppm)

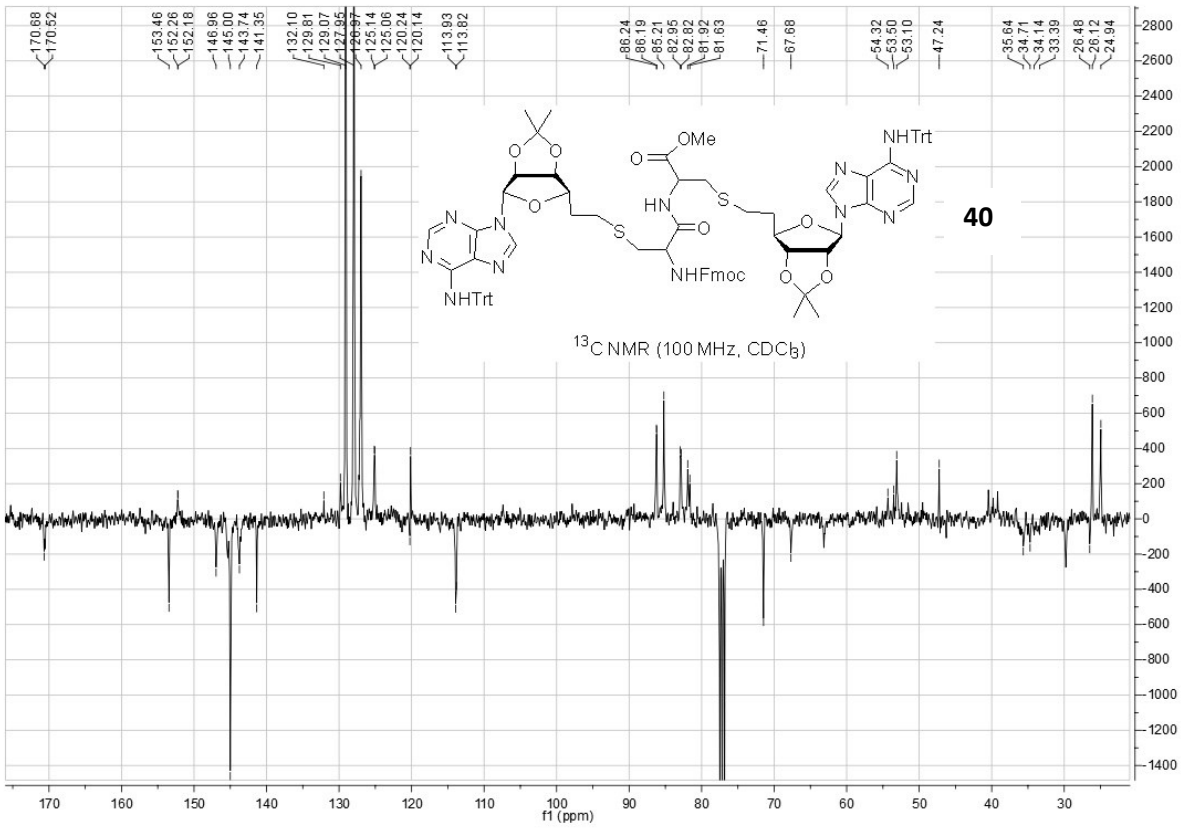

39
^{13}C NMR (90 MHz, CDCl$_3$)
f1 (ppm)

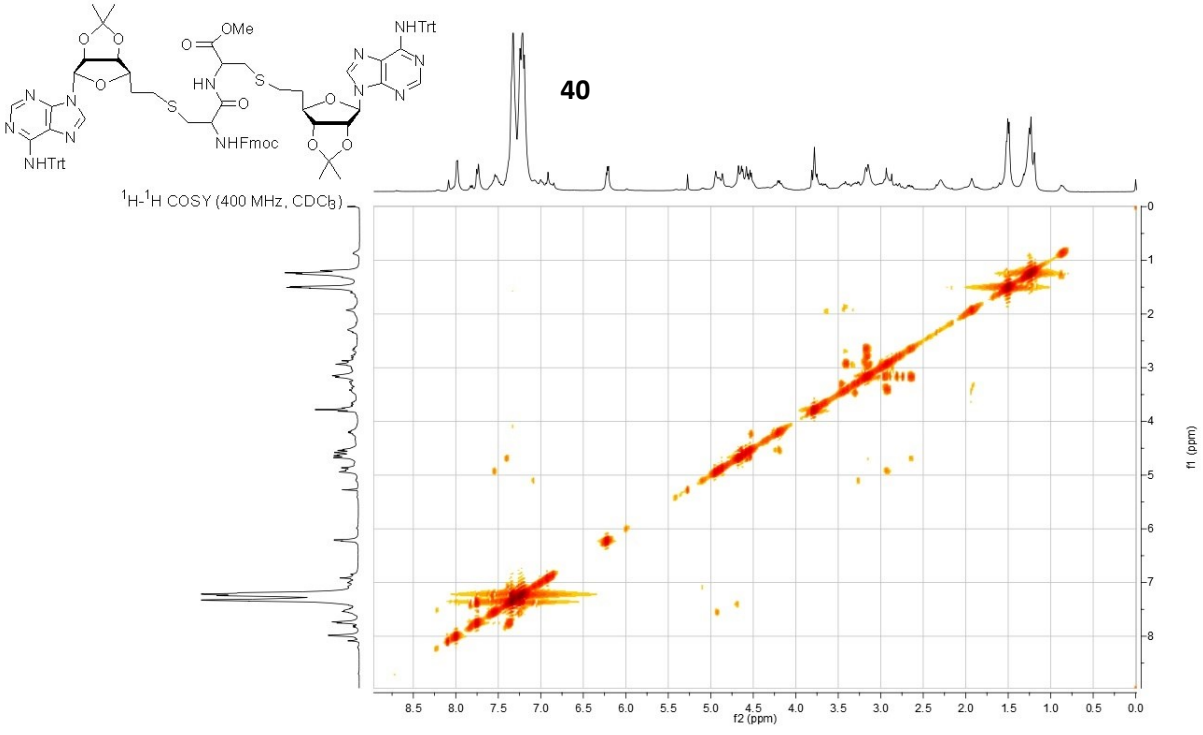

39
^{1}H-^{1}H COSY (400 MHz, CDCl$_3$)
f2 (ppm)
f1 (ppm)

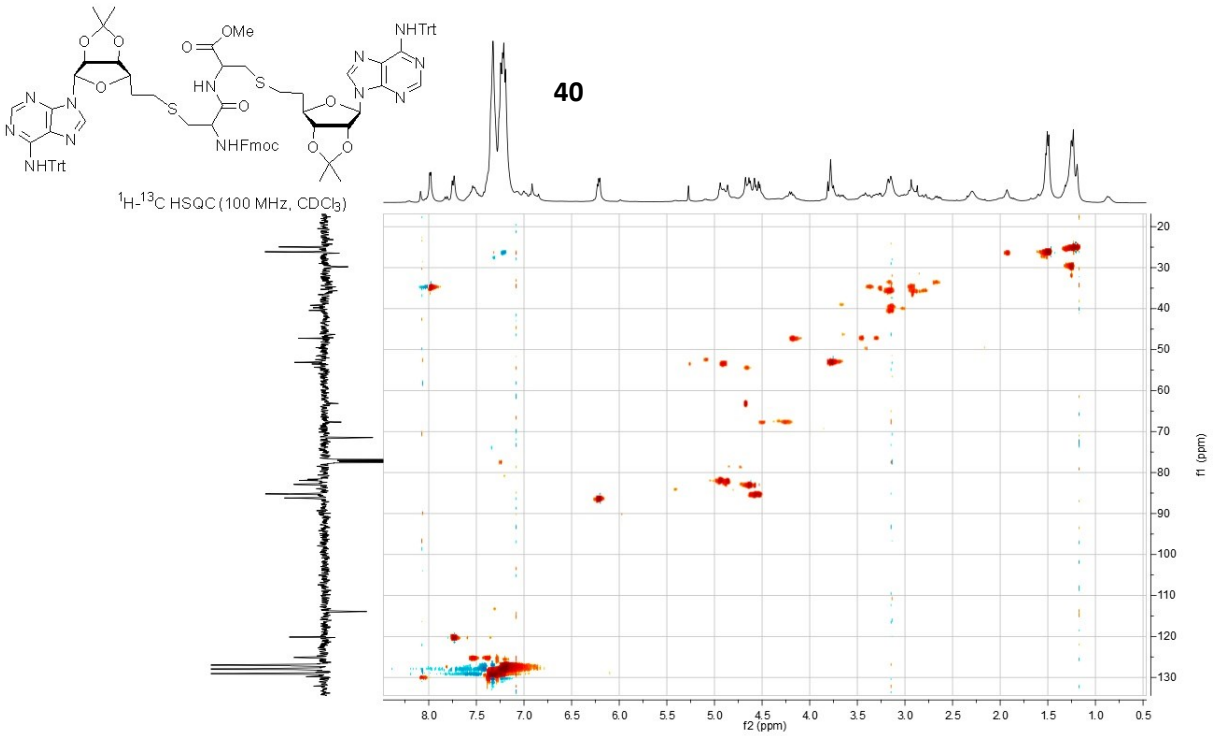

39
^{1}H-^{13}C HSQC (100 MHz, CDCl$_3$)
f2 (ppm)
f1 (ppm)

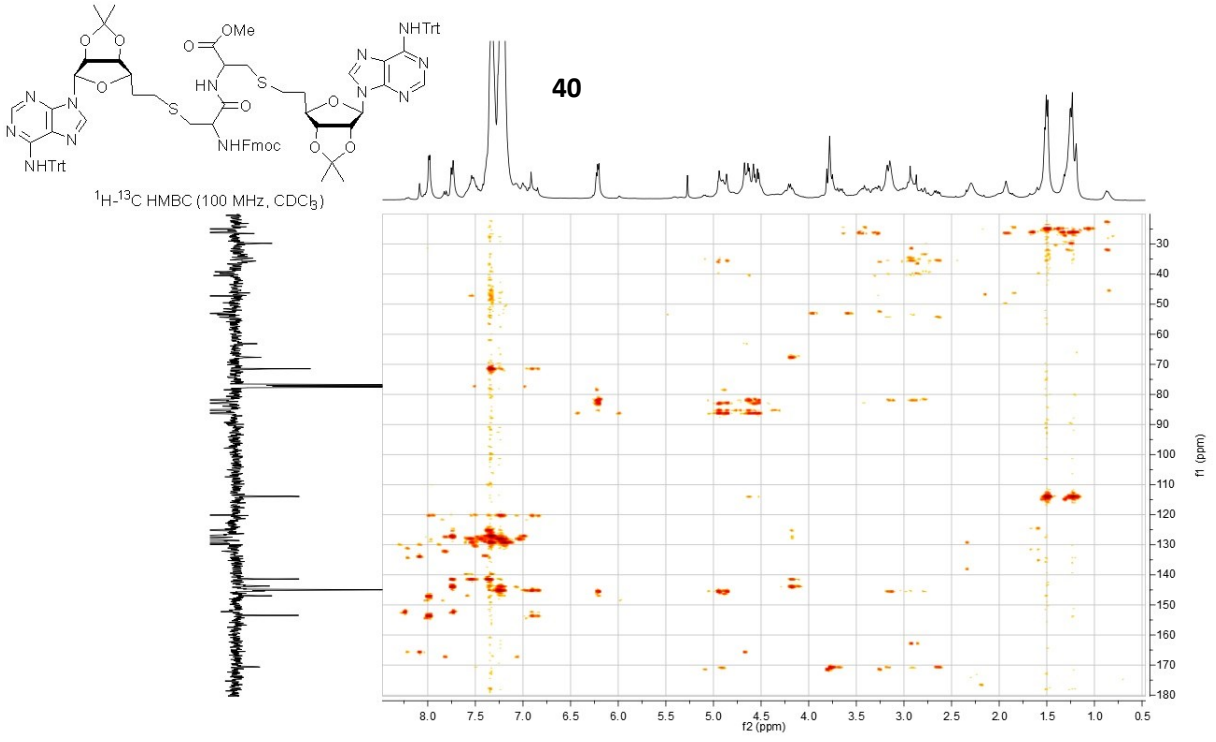

39
^{1}H-^{13}C HMBC (100 MHz, CDCl$_3$)
f2 (ppm)
f1 (ppm)


40
^{1}H NMR (360 MHz, CDCl$_3$)
f1 (ppm)


170.68
170.52
153.46
152.26
152.18
146.96
145.00
143.74
141.35
132.10
129.81
129.07
127.85
126.97
125.14
125.06
120.24
120.14
113.93
113.82
86.24
86.19
85.21
82.95
82.82
81.92
81.63
71.46
67.68
54.32
53.50
53.10
47.24
35.64
34.71
34.14
33.39
26.48
26.12
24.94
OMe
NHTrt
NHFmoc
40
13C NMR (100 MHz, CDCl3)
f1 (ppm)


OMe
NHTrt
NHFmoc
40
1H-1H COSY (400 MHz, CDCl3)
f1 (ppm)
f2 (ppm)


OMe
NHTrt
HN
S
NHFmoc
NHTrt
40
^{1}H-^{13}C HSQC (100 MHz, CDCl$_3$)
f2 (ppm)
f1 (ppm)


OMe
NHTrt
HN
S
NHFmoc
NHTrt
40
^{1}H-^{13}C HMBC (100 MHz, CDCl$_3$)
f2 (ppm)
f1 (ppm)