Supporting Information

Structure-Activity Relationship of Truncated 2,8-Disubstituted-adenosine Derivatives as Dual A_{2A}/A_3 Adenosine Receptor Antagonists and their Cancer Immunotherapeutic Activity

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Additional *in vitro* data and procedures

Table S1. The cAMP functional assay of $A_{2A}AR$ with 100 nM of full agonist CGS21680

Compound	% inhibition, $A_{2A}AR$ cAMP functional assay
5a	53.47%
5d	63.82%
5g	12.31%

Table S2. The result of *in vitro* CYP inhibition assay

Compound	% inhibition at 10 μM				
	CYP1A2	CYP2C9	CYP2C19	CYP2D6	CYP3A4
5d	14%	4%	6%	3%	0%

Table S3. The result of *in vitro* hERG inhibition assay

Compound	% inhibition		
	1 μM	3 μM	10 μM
5d	13%	18%	31%

Cyclic AMP Accumulation Assay with CGS-21680. Human adenosine receptor functional experiments were carried out in $A_{2A}AR$-HEK293 cell line for $hA_{2A}AR$. The cells are seeded on the 96 well culture plate (NUNC 136101) with stimulation buffer (1X HBSS, Gibco 14025092; 7.5% BSA stabilizer; 250 mM IBMX, Sigma I5879; 1 M HEPES). 10 μM of test compound and 100 nM of CGS-21680 (**26**) are added, then the cells are centrifuged at 500 rpm for 3 min, followed by incubated at 30 °C for 30 min. After incubation, the amount of cAMP is determined using LANCE Ultra cAMP detection kit (Perkin Elmer TRF0262).

In vitro CYP inhibition assay. Human liver microsomes (0.25 mg/mL), 0.1 M phosphate buffer (pH 7.4), a cocktail of five coenzyme substrates (Phenacetin 50 μM, Diclofenac 10 μM, S-mephenytoin 100 μM, Dextromethorphan 5 μM, Midazolam 2.5 μM), and test compound (10 μM

concentration) was pre-incubated for 5 min at 37 °C. NADPH generation system solution was added and incubated for 15 min at 37 °C. In order to terminate the reaction, acetonitrile solution containing an internal standard (Terfenadine) was added and centrifuged for 5 min (14,000× g rpm, 4 °C). The supernatant was injected into the LC-MS/MS system to analyze the metabolites of the substrates simultaneously. Metabolites of each substrate produced during the reaction were analyzed using the Shimadzu Nexera XR system and TSQ vantage (Thermo). Kinetex C18 column (2.1 × 100 mm, 2.6 μm particle size; Phenomenex, USA) was used for HPLC. The mobile phase used contained 0.1% formic acid in distilled water (A) and 0.1% formic acid containing acetonitrile (B). The generated metabolites were quantified using MRM (Multiple Reaction Monitoring) and Xcalibur (version 1.6.1) was used for data analysis.[1,2]

In vitro hERG assay. CHO cells stably expressing hERG potassium channels from Sophion Biosciences were used for this test. The cells were cultured in a humidified and air-controlled (5% CO_2) incubator at 37 °C. The CHO cells which were at least two days after plating and more than 75% confluent would be used for experiments. Before testing, cells were harvested using TrypLE and resuspended in the physiological solution at the room temperature. The physiological solution and external solution were prepared at least one month. The intracellular solution was prepared in batches aliquoted, and stored at 4 °C until used. Test compounds were dissolved in 100% DMSO at 10 mM to obtain stock solutions for different test concentrations. Then the stock solutions were further diluted into external solution to achieve final concentrations for testing. Visual check for precipitation was conducted before testing. Final DMSO concentration in extracellular solution was not more than 0.30% for the test compounds. hERG SyncroPatch(384PE) assay was conducted at room temperature. The Setup, Prime Chip, Catch and Seal Cells, Amplifier Settings, Voltage and Application Protocols were established with Biomek Software (Nanion). One addition of 40 μL of the vehicle was applied, followed by 300s for a baseline period. Then the doses of the compounds were added with 40 μL. The exposure of test compound at each concentration was no less than 300s. The recording for the whole process had to pass the quality control or the well was abandoned and the compound was retested, all automatically set by PatchControl. Three concentrations (1.00 μM, 3.00 μM, and 10.00 μM) were tested for each compound. Minimum 2 replicates per concentration were obtained.

Single-Crystal X-ray crystallography data of compounds 11b, 22a and 22b.

Compound **11b**, **22a** or **22b** (~10 mg) was dissolved in ethyl acetate and dichloromethane (1.0 mL, 1:4) in a glass vial. After staying at room temperature, the single crystals of **11b, 22a** or **22b** were obtained. X-ray diffraction data of **11b** (CCDC No: 2164196), **22a** (CCDC No: 2164194), and **22b 22a** (CCDC No: 2258518) were obtained on a SuperNova, Dual, Cu at zero, AtlasS2 diffractometer. Using Olex2[3], the structure was solved with the ShelXT[4] structure solution program using Intrinsic Phasing and refined with the ShelXL[4] refinement package using Least Squares minimization.

*Single-Crystal X-ray crystallography of compound **11b***

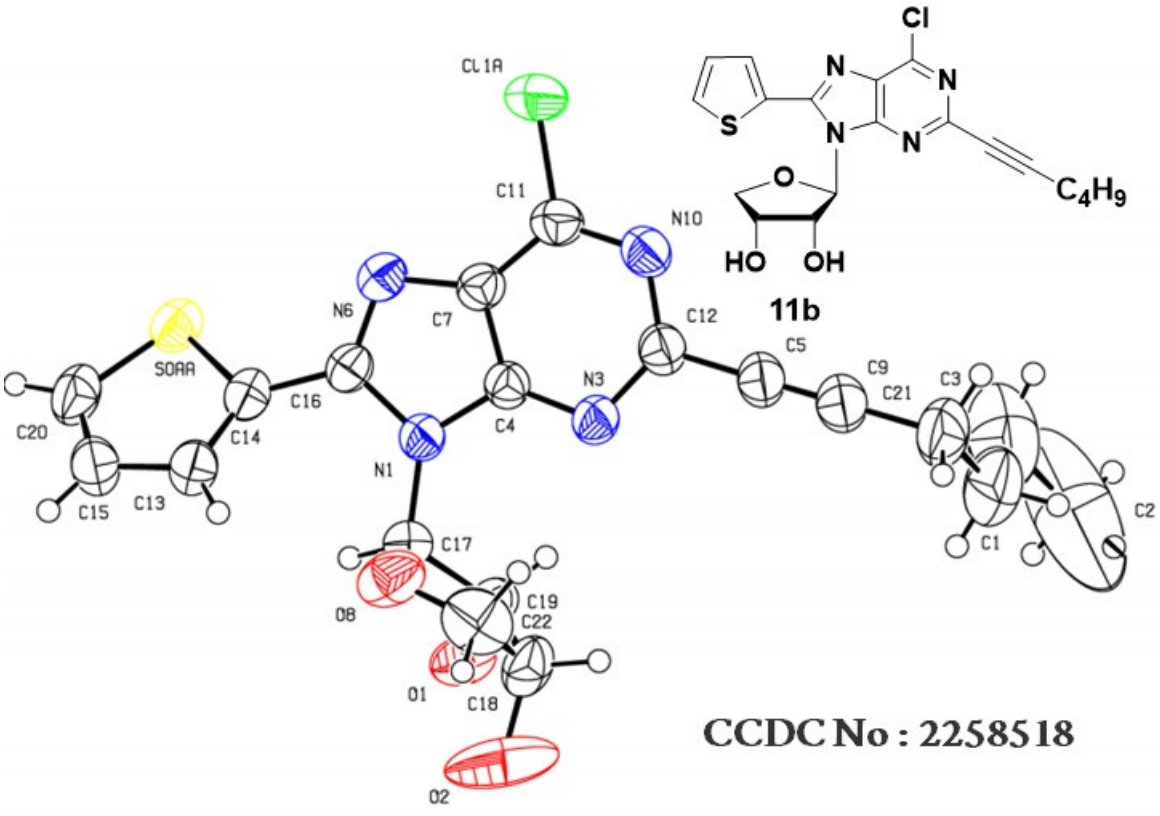

Figure S1. ORTEP diagram of compound **11b** showing thermal ellipsoid at 50% probability.

Table S4. Crystal data and structure refinement for **11b**

Empirical formula	$C_{19}H_{19}ClN_4O_3S$
Formula weight	418.89
Temperature/K	293(1)
Crystal system	monoclinic
Space group	C2
a/Å	25.4206(7)
b/Å	7.3860(2)
c/Å	10.6375(3)
α/°	90
β/°	96.378(2)
γ/°	90
Volume/Å^3	1984.90(10)
Z	4
ρ_{calc}g/cm^3	1.402
μ/mm^{-1}	2.929
F(000)	872.0
Crystal size/mm^3	0.563 × 0.19 × 0.087
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	6.998 to 147.472
Index ranges	-31 ≤ h ≤ 26, -8 ≤ k ≤ 8, -13 ≤ l ≤ 12
Reflections collected	3649
Independent reflections	2927 [R_{int} = 0.0113, R_{sigma} = 0.0187]
Data/restraints/parameters	2927/1/256
Goodness-of-fit on F^2	1.036

Final R indexes [I>=2σ (I)]	R_1 = 0.0469, wR_2 = 0.1323
Final R indexes [all data]	R_1 = 0.0483, wR_2 = 0.1343
Largest diff. peak/hole / e Å^{-3}	0.36/-0.19

Single-Crystal X-ray crystallography of compound ***22a***

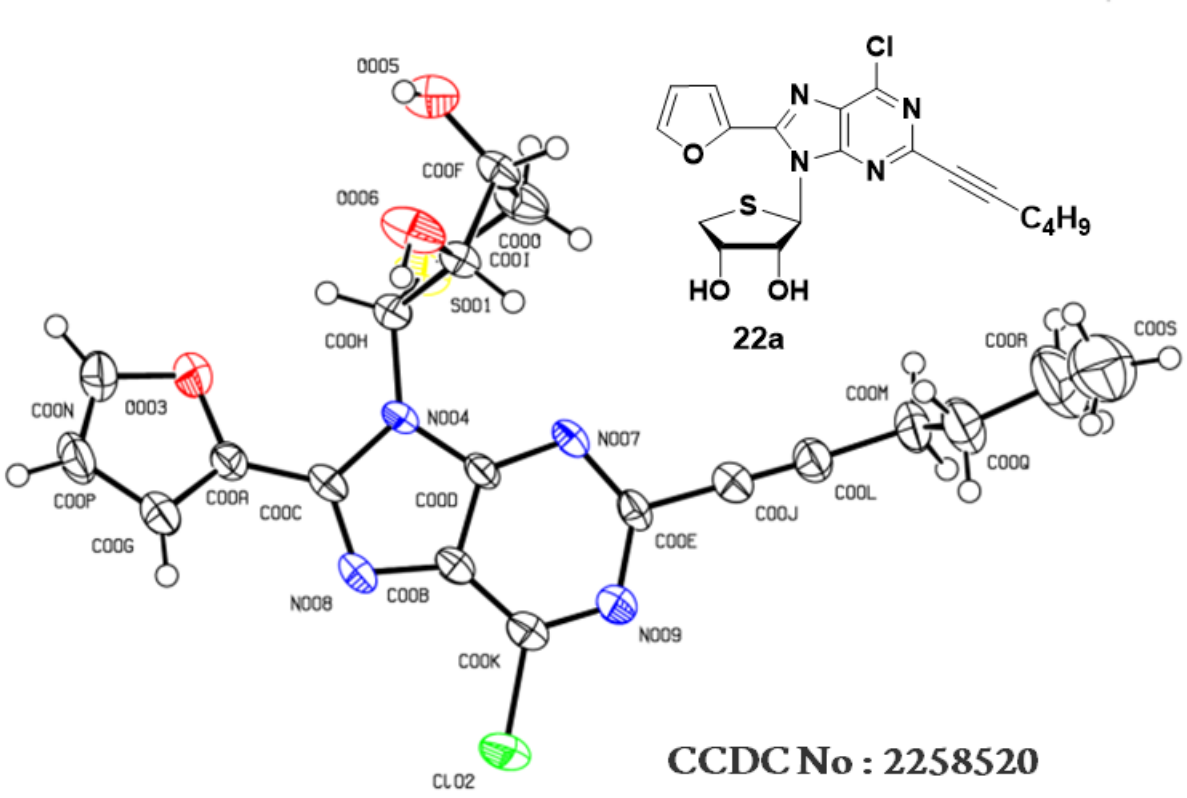

Figure S2. ORTEP diagram of compound 22a showing thermal ellipsoid at 50% probability.

Table S5. Crystal data and structure refinement for **22a**

Empirical formula	$C_{19}H_{19}ClN_4O_3S$
Formula weight	418.89
Temperature/K	293(2)
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	7.1613(18)
b/Å	10.7229(15)
c/Å	25.009(4)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	1920.5(6)
Z	4
ρ_{calc}g/cm^3	1.449
μ/mm^{-1}	0.337
F(000)	872.0
Crystal size/mm^3	0.262 × 0.114 × 0.075
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	5.004 to 58.788
Index ranges	-8 ≤ h ≤ 8, -13 ≤ k ≤ 14, -25 ≤ l ≤ 34
Reflections collected	11286
Independent reflections	4489 [R_{int} = 0.0733, R_{sigma} = 0.1095]

Data/restraints/parameters	4489/0/256
Goodness-of-fit on F^2	1.073
Final R indexes [I>=2σ (I)]	R_1 = 0.0743, wR_2 = 0.1247
Final R indexes [all data]	R_1 = 0.1233, wR_2 = 0.1459
Largest diff. peak/hole / e $Å^{-3}$	0.42/-0.31

Single-Crystal X-ray crystallography of compound ***22b***

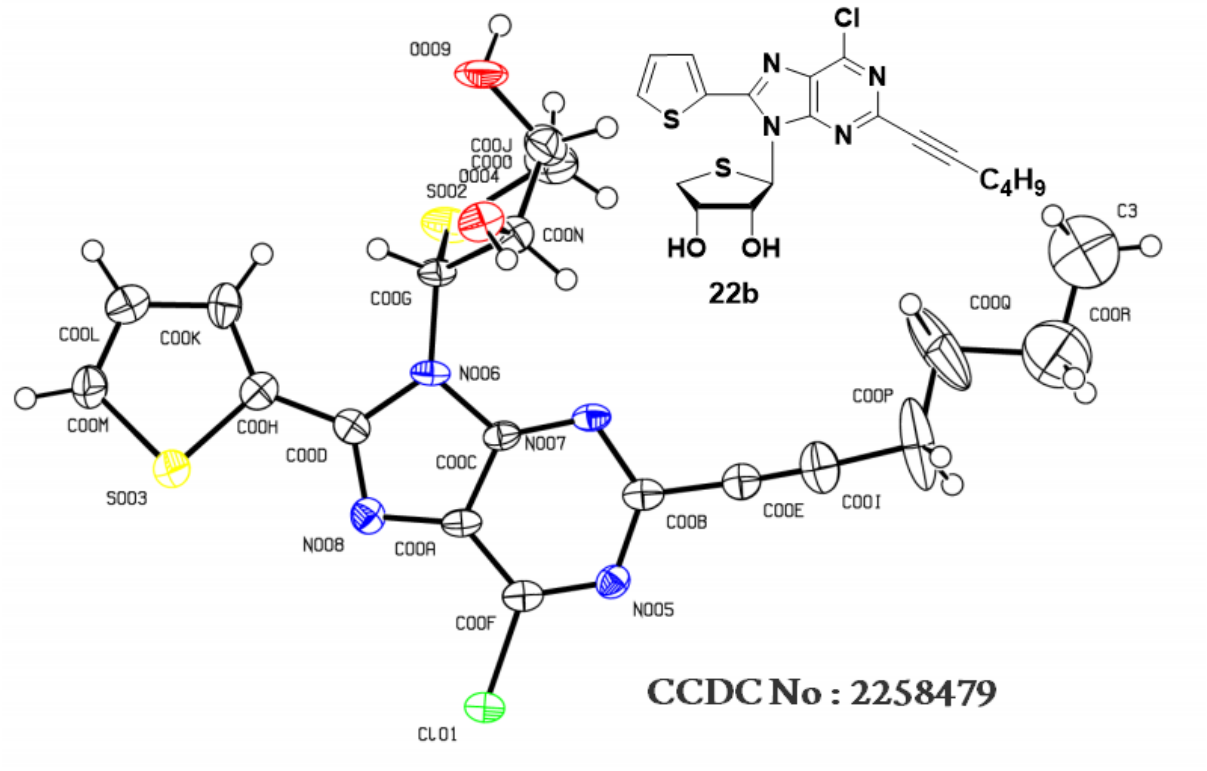

Figure S3. ORTEP diagram of compound 22b showing thermal ellipsoid at 50% probability.

Table S6. Crystal data and structure refinement for **22b**

Empirical formula	$C_{19}H_{19}ClN_4O_2S_2$
Formula weight	434.97
Temperature/K	111.0(8)
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	7.0735(7)
b/Å	24.677(4)
c/Å	12.1455(13)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	2120.0(4)
Z	4
ρ_{calc}g/cm^3	1.3627
μ/mm^{-1}	0.399
F(000)	905.9
Crystal size/mm^3	0.466 × 0.394 × 0.229
Radiation	Mo Kα (λ = 0.71073)
2Θ range for data collection/°	4.7 to 59.38
Index ranges	-8 ≤ h ≤ 9, -34 ≤ k ≤ 29, -15 ≤ l ≤ 16

Reflections collected	23486
Independent reflections	5357 [R_{int} = 0.0955, R_{sigma} = 0.0926]
Data/restraints/parameters	5357/12/255
Goodness-of-fit on F^2	1.038
Final R indexes [I>=2σ (I)]	R_1 = 0.0902, wR_2 = 0.2143
Final R indexes [all data]	R_1 = 0.1161, wR_2 = 0.2353
Largest diff. peak/hole / e $Å^{-3}$	1.00/-0.90

Reference

(1) Kim, M.-J.; Kim, H.; Cha, I.-J.; Park, J.-S.; Shon, J.-H.; Liu, K.-H.; Shin, J.-G. High-throughput screening of inhibitory potential of nine cytochrome P450 enzymes *in vitro* using liquid chromatography/tandem mass spectrometry. *Rapid Commun. Mass Spectrom.* **2005**, 19, 2651–2658.

(2) Kerns, E.H.; Di, L. (Eds.) *Chapter 32—CYP Inhibition Methods. Drug-like Properties: Concepts, Structure Design and Methods*, Academic Press: San Diego, CA, USA, 2008, pp. 360–3

(3) Dolomanov , O. V.; Bourhis , L. J.; Gildea , R. J; Howard, J. A. K.; Puschmann , H. J. *Appl. Cryst.* **2009**, *42*, 339-341..

(4) Sheldrick , G. M. *Acta Cryst.* **2015**, *A71*, 3-8.

(5) Sheldrick , G. M. *Acta Cryst.* **2015**, *C71*, 3-8.

HPLC data of the representative compounds

HPLC data of the compound ***5d***

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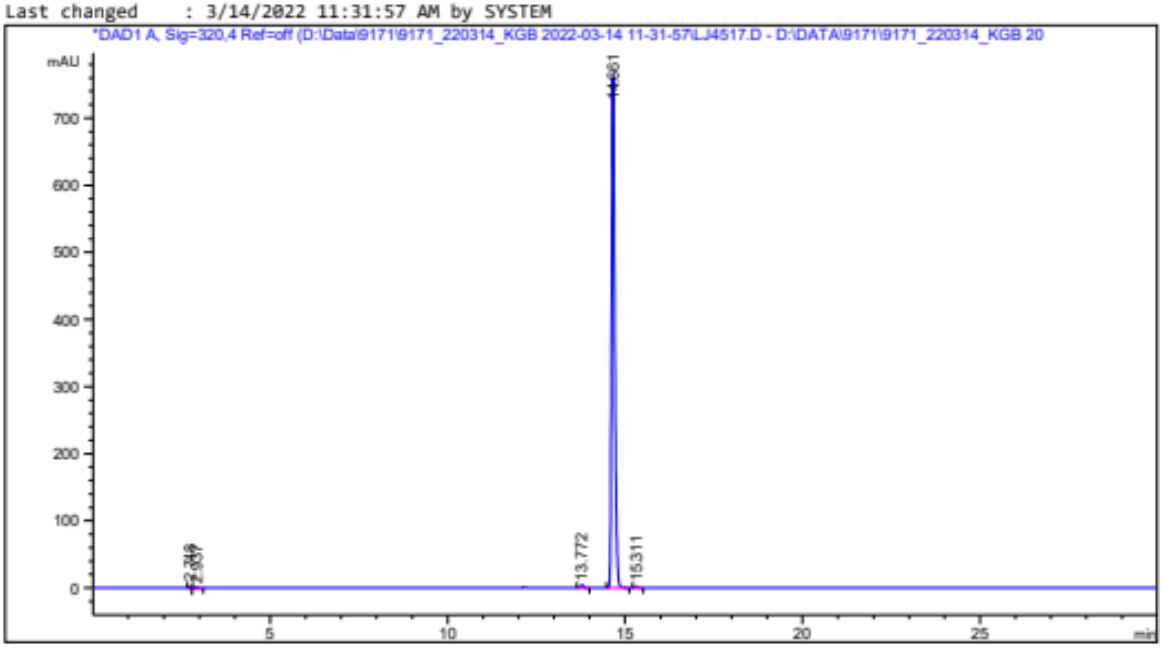


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   1   2.748 BB    0.0527   10.08669    3.04008   0.2170
   2   2.937 BB    0.1353   15.64708    1.53052   0.3366
   3  13.772 BB    0.0918   22.83916    3.81585   0.4913
   4  14.661 BB    0.0903 4589.68164  761.54034  98.7254
   5  15.311 BB    0.0935   10.68061    1.74166   0.2297

Totals :                  4648.93519  771.66846
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HPLC data of the compound **5g**

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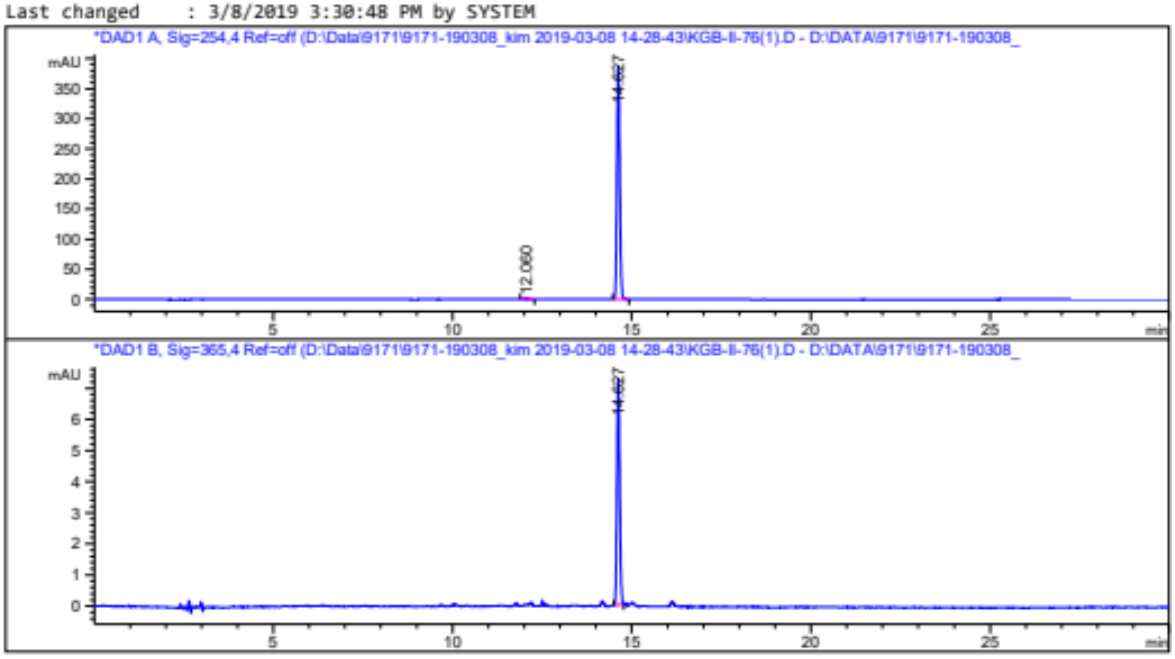


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Use Multiplier & Dilution Factor with ISTDs


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   1  12.060 BB    0.0941   10.40172    1.63749   0.5011
   2  14.627 BB    0.0821 2065.25195  387.94006  99.4989

Totals :                  2075.65367  389.57755
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HPLC data of the compound ***5h***

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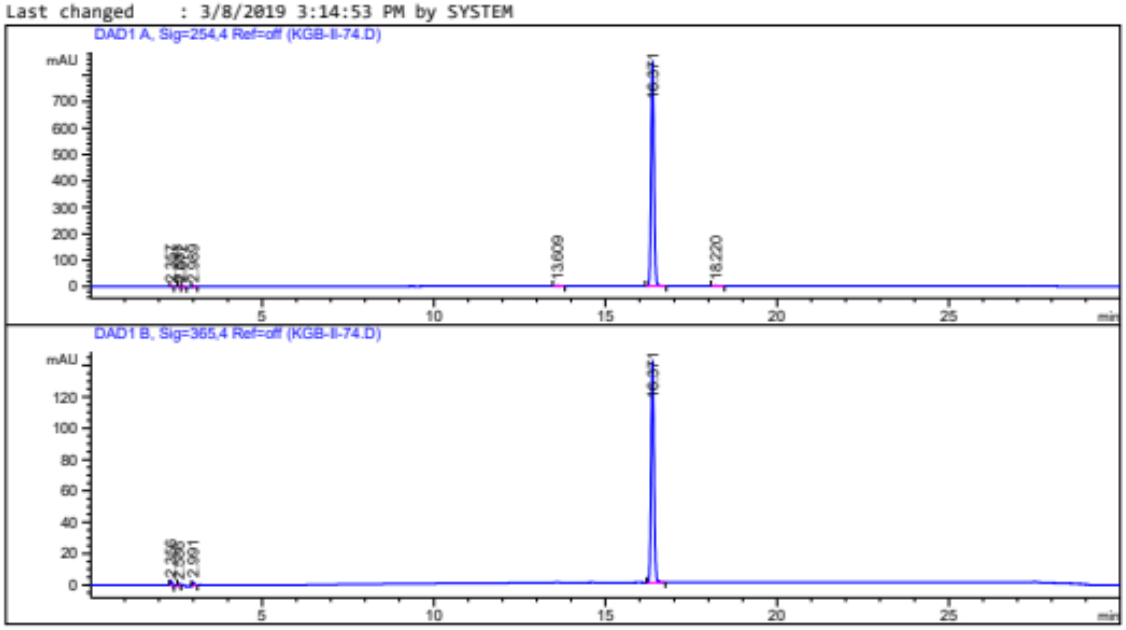


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Use Multiplier & Dilution Factor with ISTDs


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   1   2.357 BB    0.0506    9.57488    2.89289   0.1960
   2   2.587 VV    0.0674    5.21559    1.13854   0.1067
   3   2.672 VB    0.0747    5.29344    1.05193   0.1083
   4   2.989 BB    0.0503   10.31255    3.13893   0.2111
   5  13.609 BB    0.0929   10.03435    1.56261   0.2054
   6  16.371 BB    0.0867 4836.85352  845.49213  98.9963
   7  18.220 BB    0.0974    8.60644    1.33185   0.1761

Totals :                  4885.89077  856.60887
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HPLC data of the compound ***5i***

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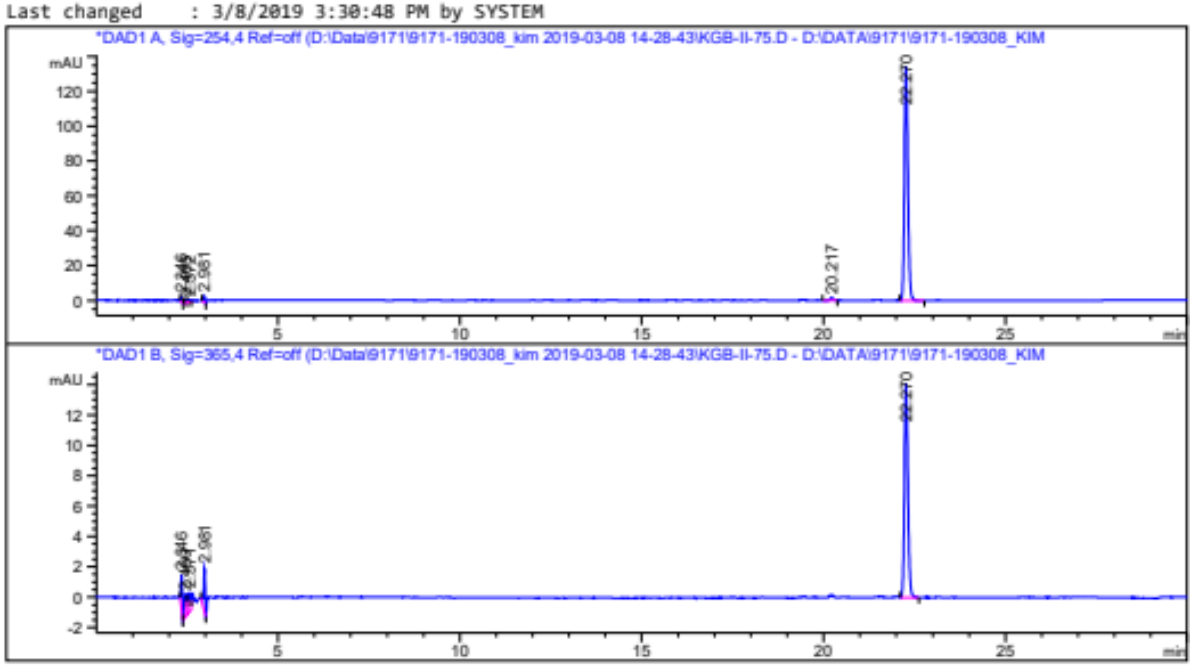


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       1   2.346 BB    0.0398    6.28328    2.64020   0.6640
       2   2.463 BV    0.0803    7.73419    1.60179   0.8173
       3   2.572 VV    0.0785    7.98579    1.35988   0.8439
       4   2.981 BB    0.0444   10.16790    3.66342   1.0745
       5  20.217 BB    0.0996    8.04234    1.24146   0.8499
       6  22.270 BB    0.1046  906.08051  134.56239  95.7504
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HPLC data of the compound ***5j***

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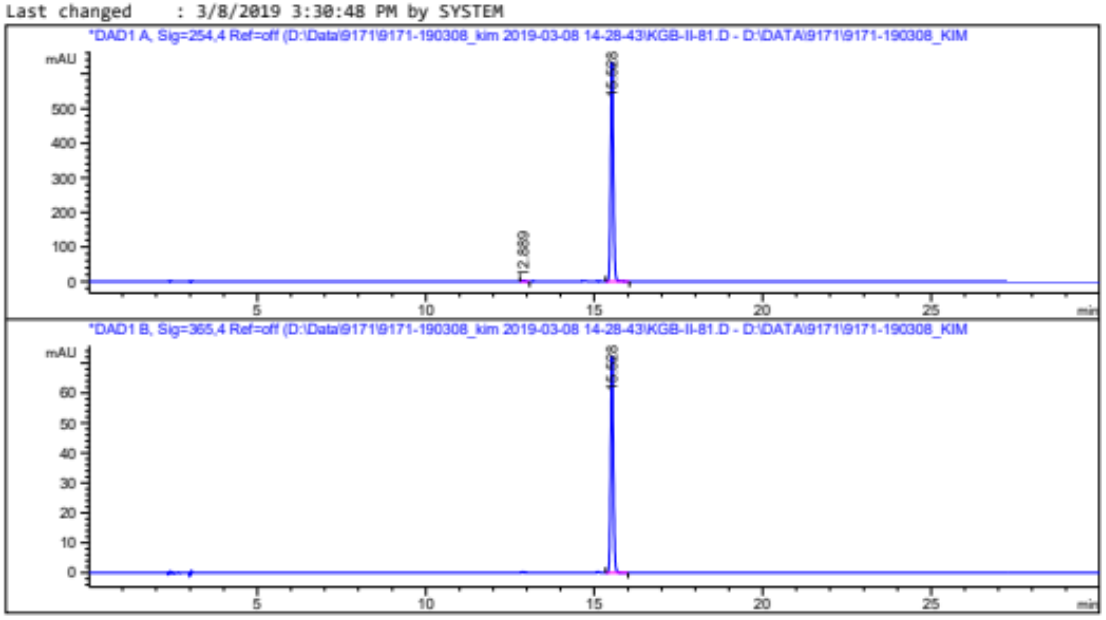


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Totals :                  3464.39143  638.42874
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HPLC data of the compound ***5k***

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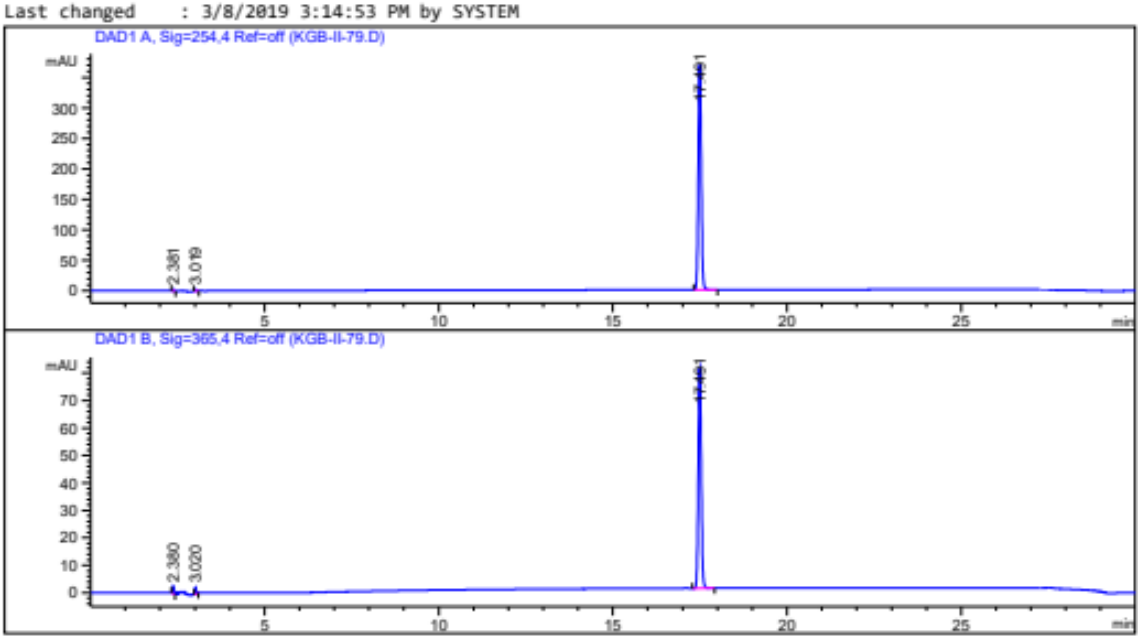


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Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


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   1   2.381 BB    0.0486    9.12424    3.07989   0.4171
   2   3.019 BB    0.0474    9.00926    2.97110   0.4118
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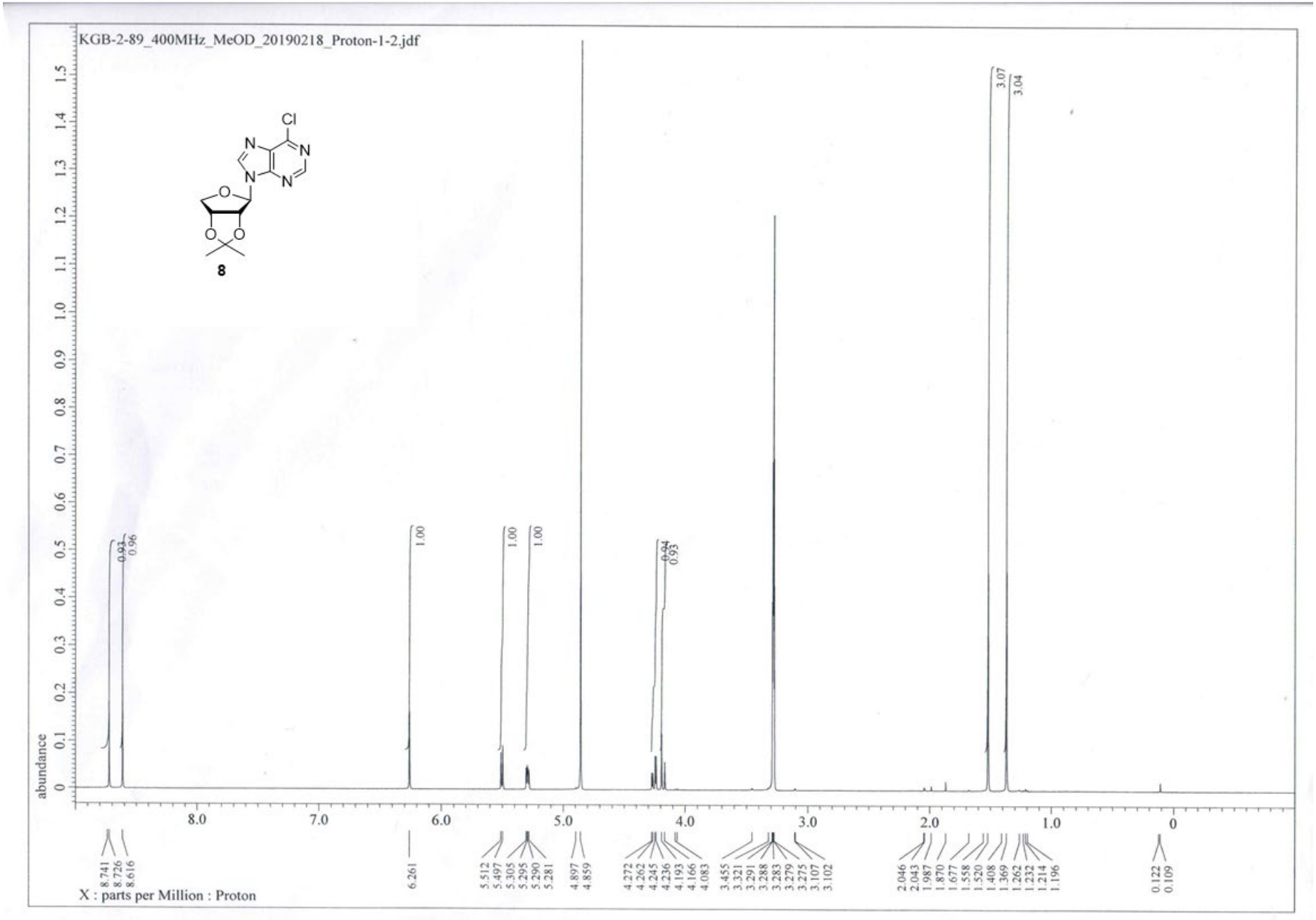
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Cl
N
N
N
N
O
O
O
8
abundance
X : parts per Million : Proton
8.741
8.726
8.616
6.261
5.512
5.497
5.305
5.295
5.290
5.281
4.897
4.859
4.272
4.262
4.245
4.236
4.193
4.166
4.083
3.455
3.321
3.291
3.288
3.283
3.279
3.275
3.107
3.102
2.046
2.043
1.987
1.870
1.677
1.558
1.520
1.408
1.369
1.262
1.232
1.214
1.196
0.122
0.109
3.07
3.04
1.00
1.00
1.00
0.94
0.93
0.93
0.96

HXY-4-77-1

exp5 std13c

SAMPLE		DEC. & VT	
date	Mar 22 2011	dfrq	400.046
solvent	CDCl3	dn	H1
file	exp	dpwr	41
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sfrq	100.601	dm	yyy
tn	C13	dmm	w
at	0.916	dmf	10900
np	46076	dseq	
sw	25157.2	dres	1.0
fb	not used	homo	n
bs	4	temp	25.0
tpwr	55	PROCESSING	
pw	10.1	lb	2.00
d1	2.000	wtfile	
tof	1043.3	proc	ft
nt	1500	fn	not used
ct	240	math	f
alock	n		
gain	not used	werr	
FLAGS		wexp	
il	n	wbs	
in	n	wnt	
dp	y		
hs	nn		
DISPLAY			
sp	-1091.8		
wp	19238.7		
vs	264		
sc	0		
wc	250		
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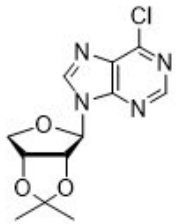
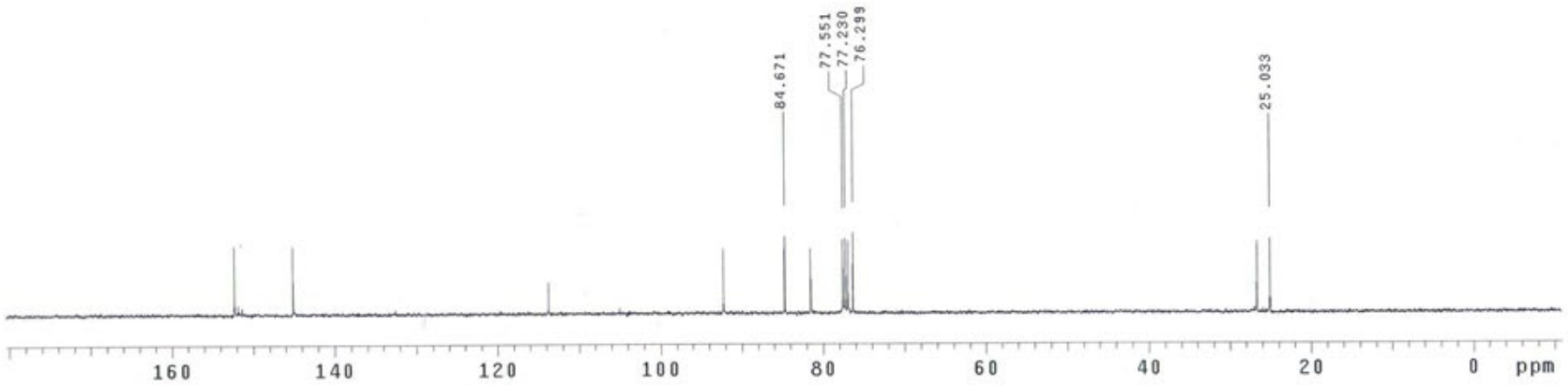
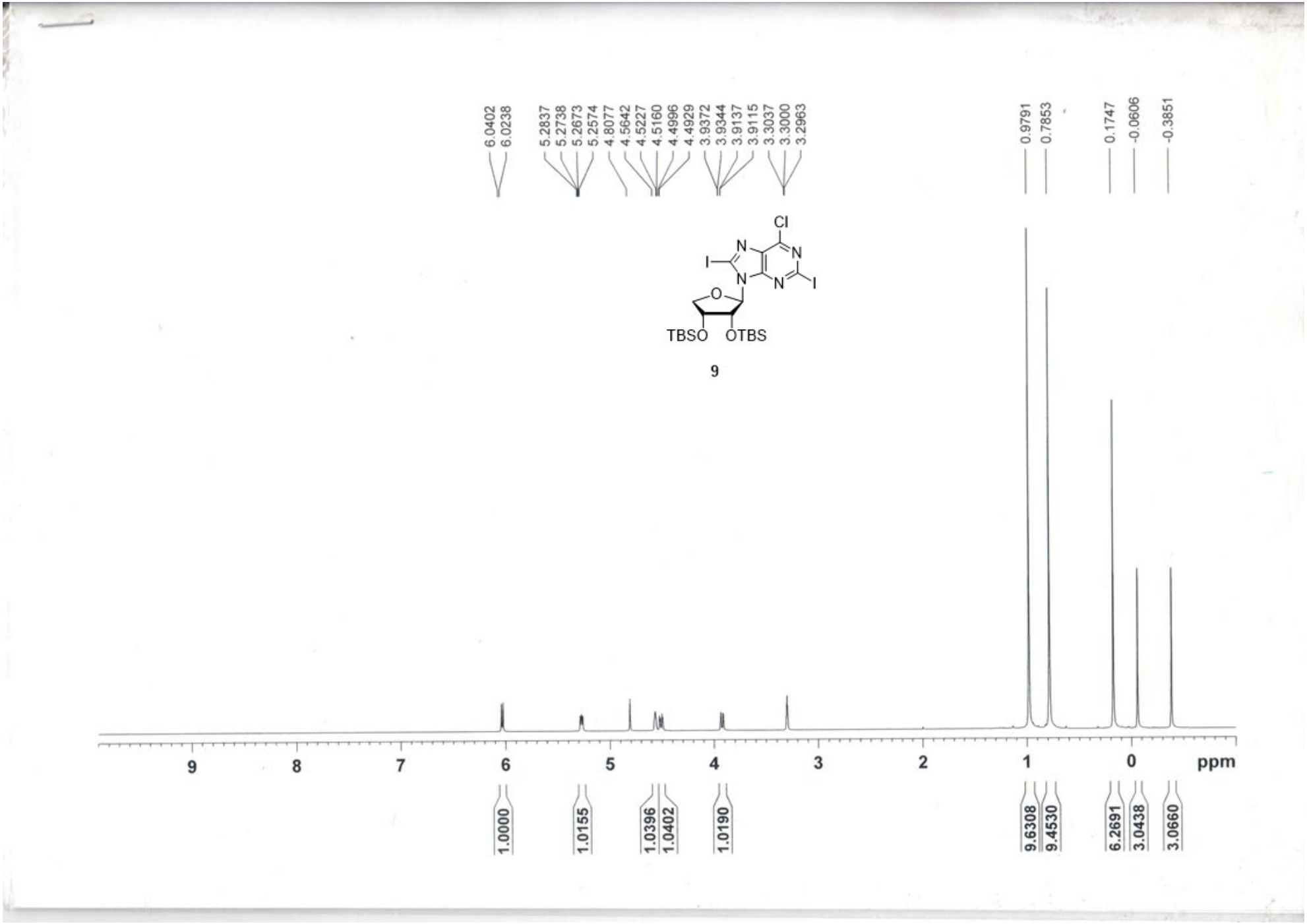
is	41.78		
rfl	9786.4		
rfp	7768.6		
th	12		
ins	1.000		
ai ph			



8

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5.2673
5.2574
4.8077
4.5642
4.5227
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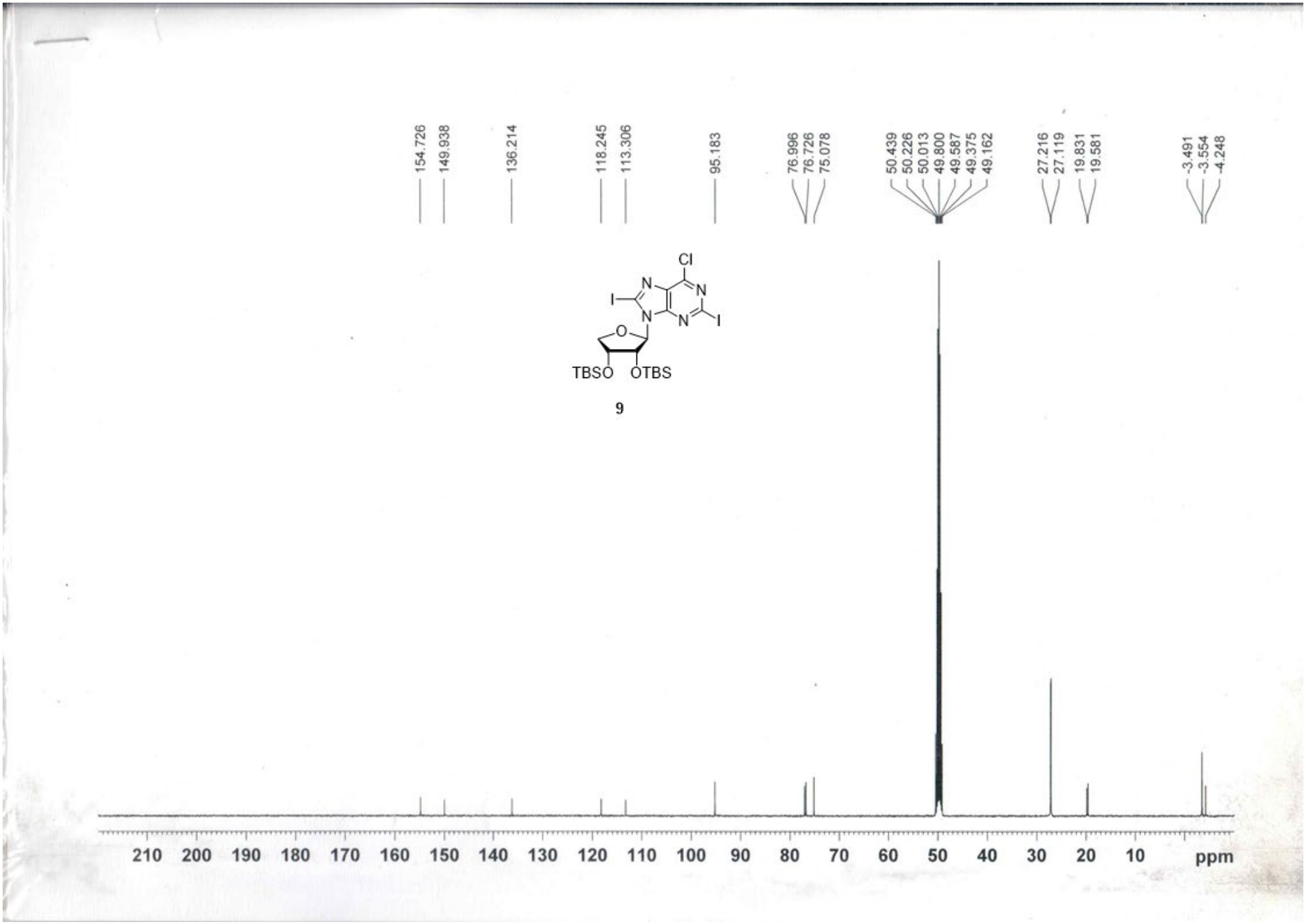
4.4996
4.4929
3.9372
3.9344
3.9137
3.9115
3.3037
3.3000
3.2963
0.9791
0.7853
0.1747
-0.0606
-0.3851
Cl
N
I
N
N
N
I
O
TBSO
OTBS
9
9
8
7
6
5
4
3
2
1
0
ppm
1.0000
1.0155
1.0396
1.0402
1.0190
9.6308
9.4530
6.2691
3.0438
3.0660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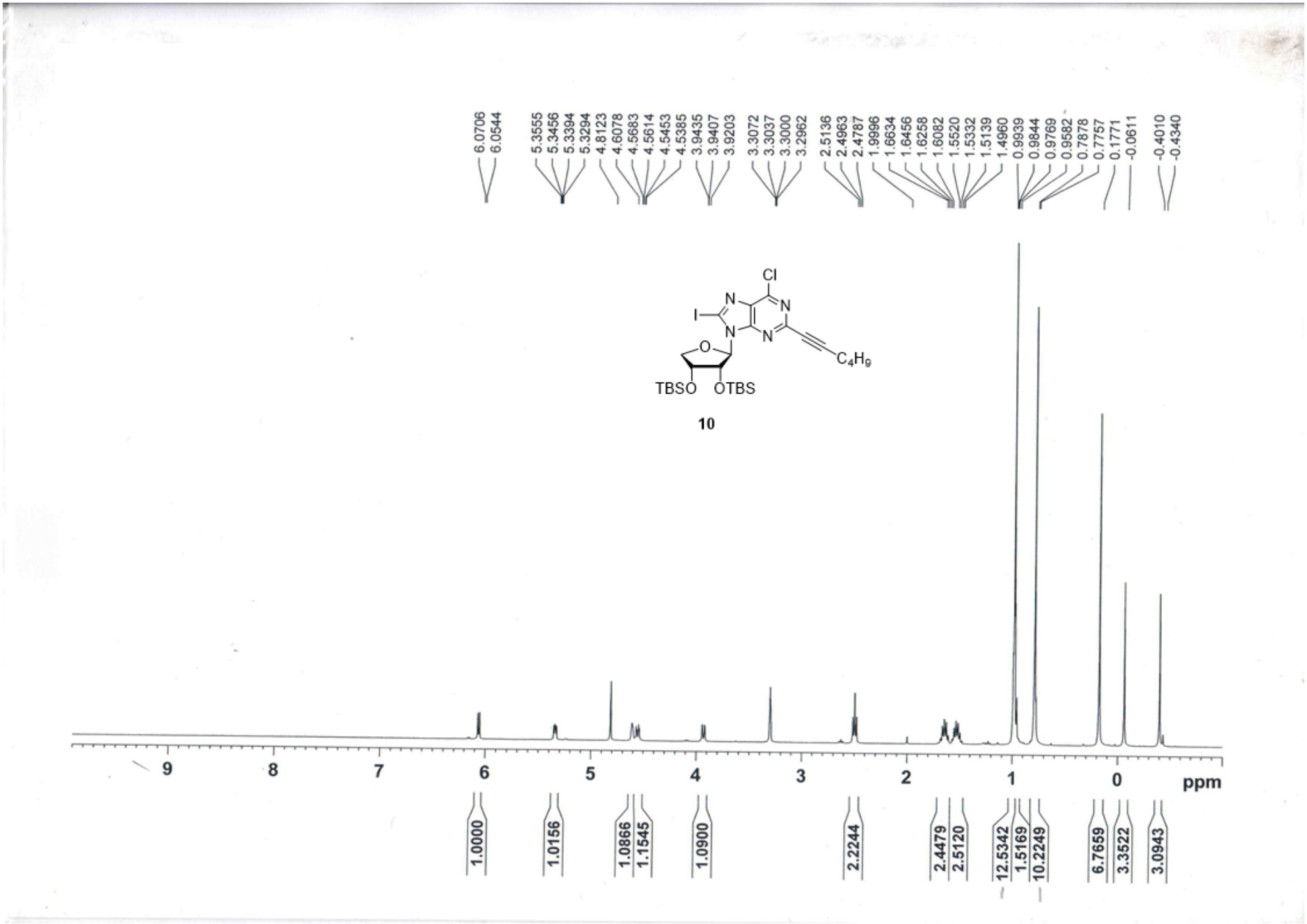
136.214
118.245
113.306
95.183
76.996
76.726
75.078
50.439
50.226
50.013
49.800
49.587
49.375
49.162
27.216
27.119
19.831
19.581
-3.491
-3.554
-4.248
Cl
N
N
I
N
N
I
O
TBSO
OTBS
9
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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6.0544
5.3555
5.3456
5.3394
5.3294
4.8123
4.6078
4.5683
4.5614
4.5453
4.5385
3.9435
3.9407
3.9203
3.3072
3.3037
3.3000
3.2962
2.5136
2.4963
2.4787
1.9996
1.6634
1.6456
1.6258
1.6082
1.5520
1.5332
1.5139
1.4960
0.9939
0.9844
0.9769
0.9582
0.7878
0.7757
0.1771
-0.0611
-0.4010
-0.4340
Cl
N
N
I
N
N
O
C4H9
TBSO
OTBS
10
9
8
7
6
5
4
3
2
1
0
ppm
1.0000
1.0156
1.0866
1.1545
1.0900
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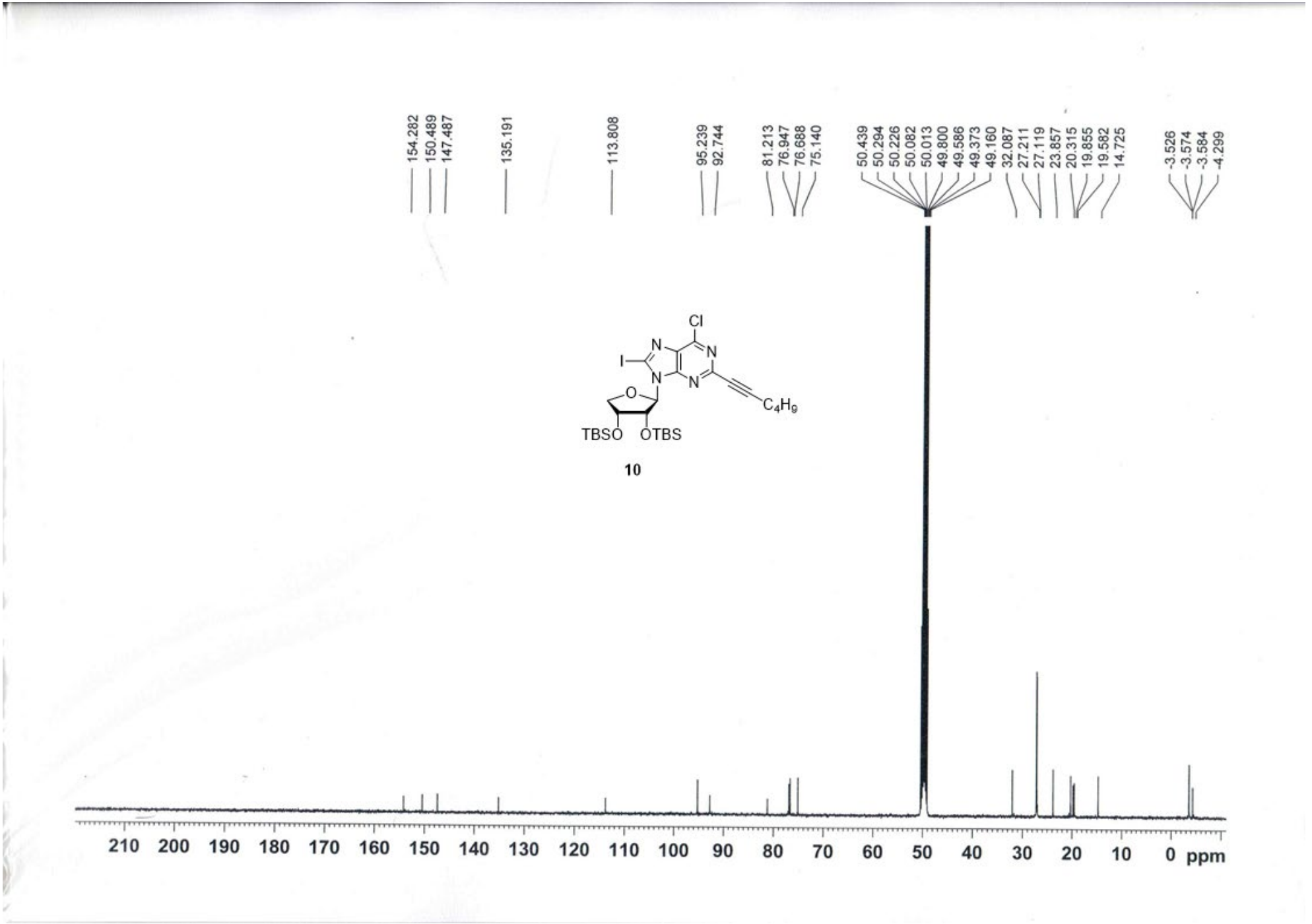
2.4479
2.5120
12.5342
1.5169
10.2249
6.7659
3.3522
3.0943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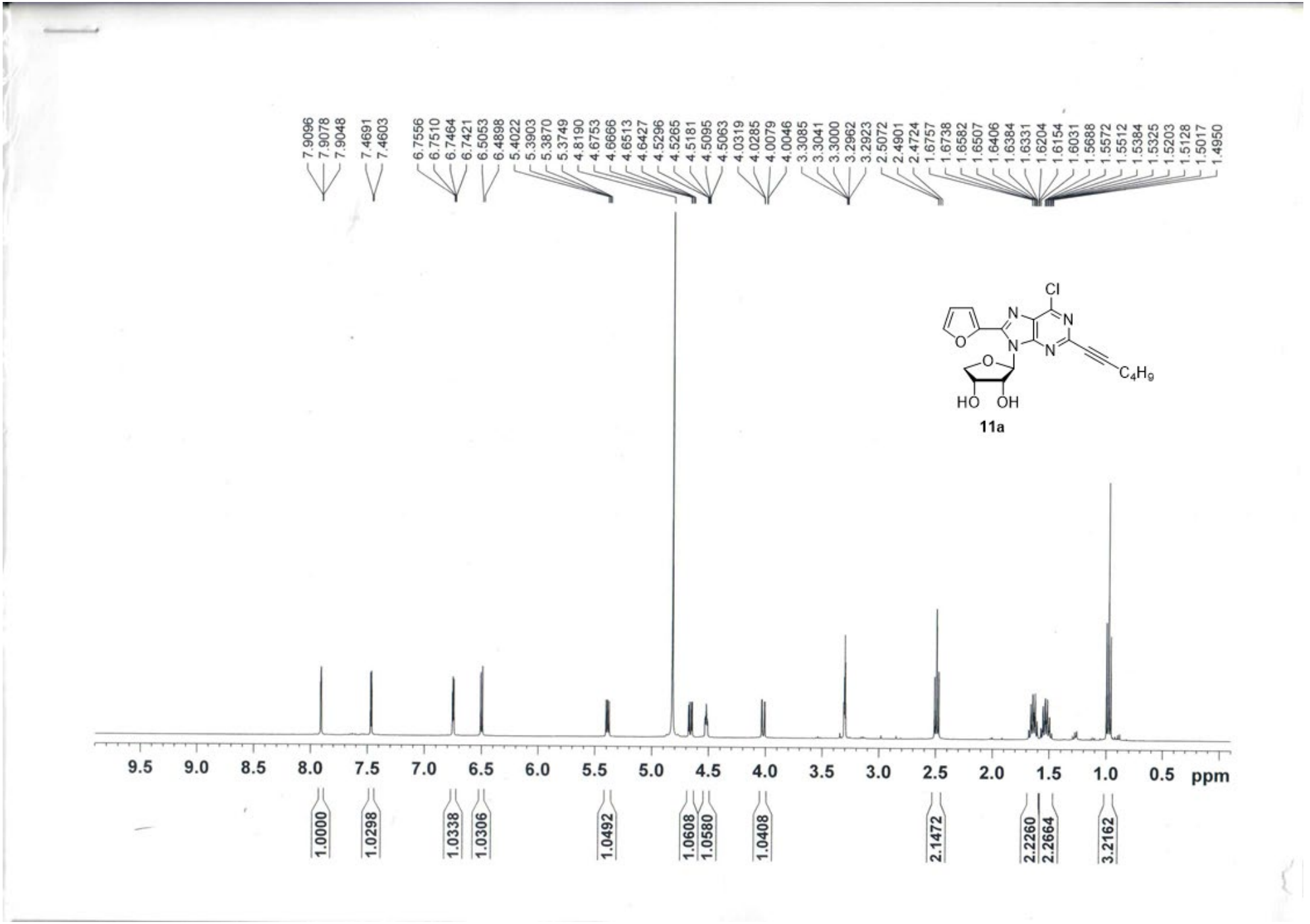
150.489
147.487
135.191
113.808
95.239
92.744
81.213
76.947
76.688
75.140
50.439
50.294
50.226
50.082
50.013
49.800
49.586
49.373
49.160
32.087
27.211
27.119
23.857
20.315
19.855
19.582
14.725
-3.526
-3.574
-3.584
-4.299
Cl
I
N
N
N
N
O
C4H9
TBSO
OTBS
10
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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7.9078
7.9048
7.4691
7.4603
6.7556
6.7510
6.7464
6.7421
6.5053
6.4898
5.4022
5.3903
5.3870
5.3749
4.8190
4.6753
4.6666
4.6513
4.6427
4.5296
4.5265
4.5181
4.5095
4.5063
4.0319
4.0285
4.0079
4.0046
3.3085
3.3041
3.3000
3.2962
3.2923
2.5072
2.4901
2.4724
1.6757
1.6738
1.6582
1.6507
1.6406
1.6384
1.6331
1.6204
1.6154
1.6031
1.5688
1.5572
1.5512
1.5384
1.5325
1.5203
1.5128
1.5017
1.4950
Cl
N
N
N
N
O
O
HO
OH
C4H9
11a
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
ppm
1.0000
1.0298
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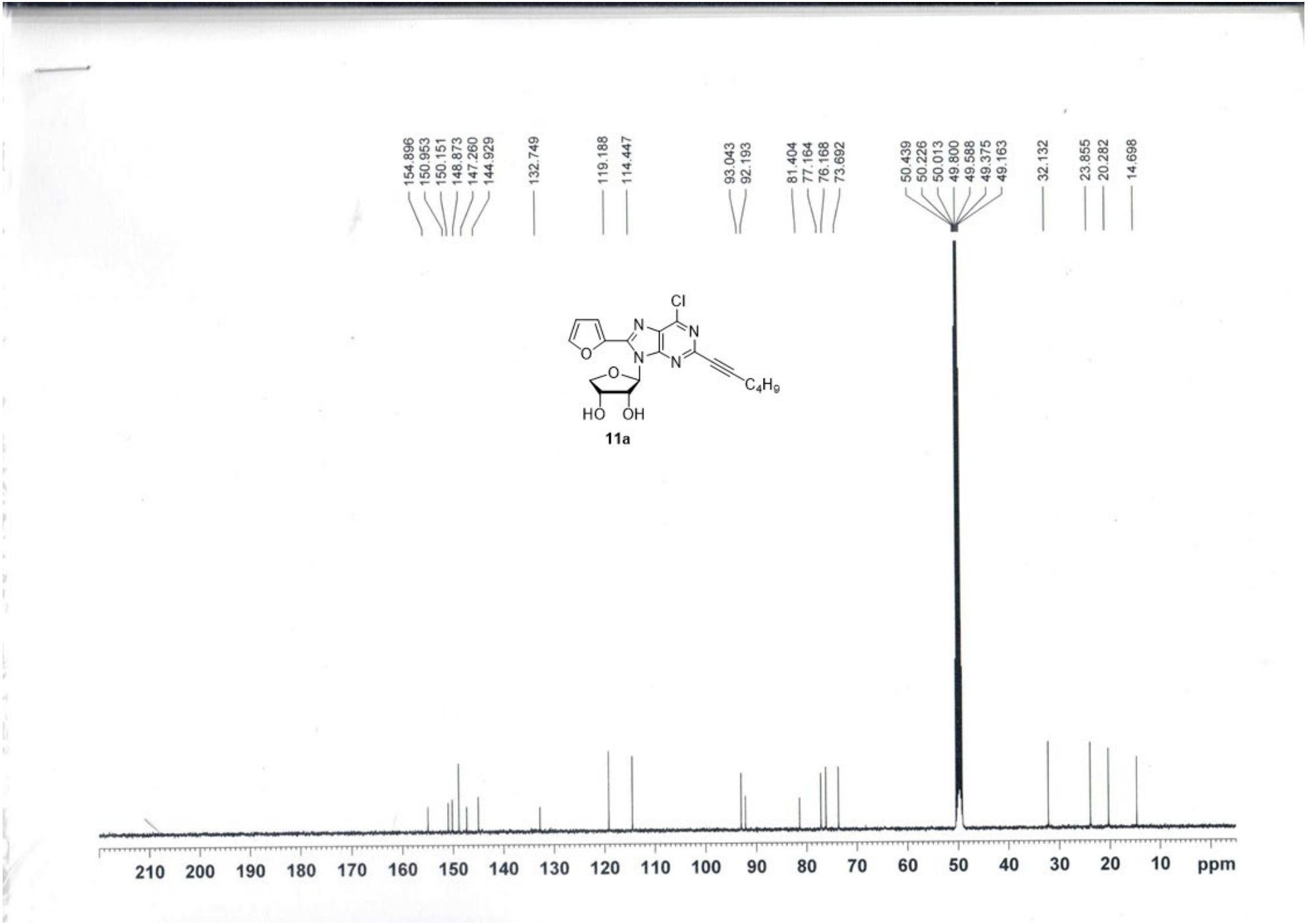
1.0306
1.0492
1.0608
1.0580
1.0408
2.1472
2.2260
2.2664
3.2162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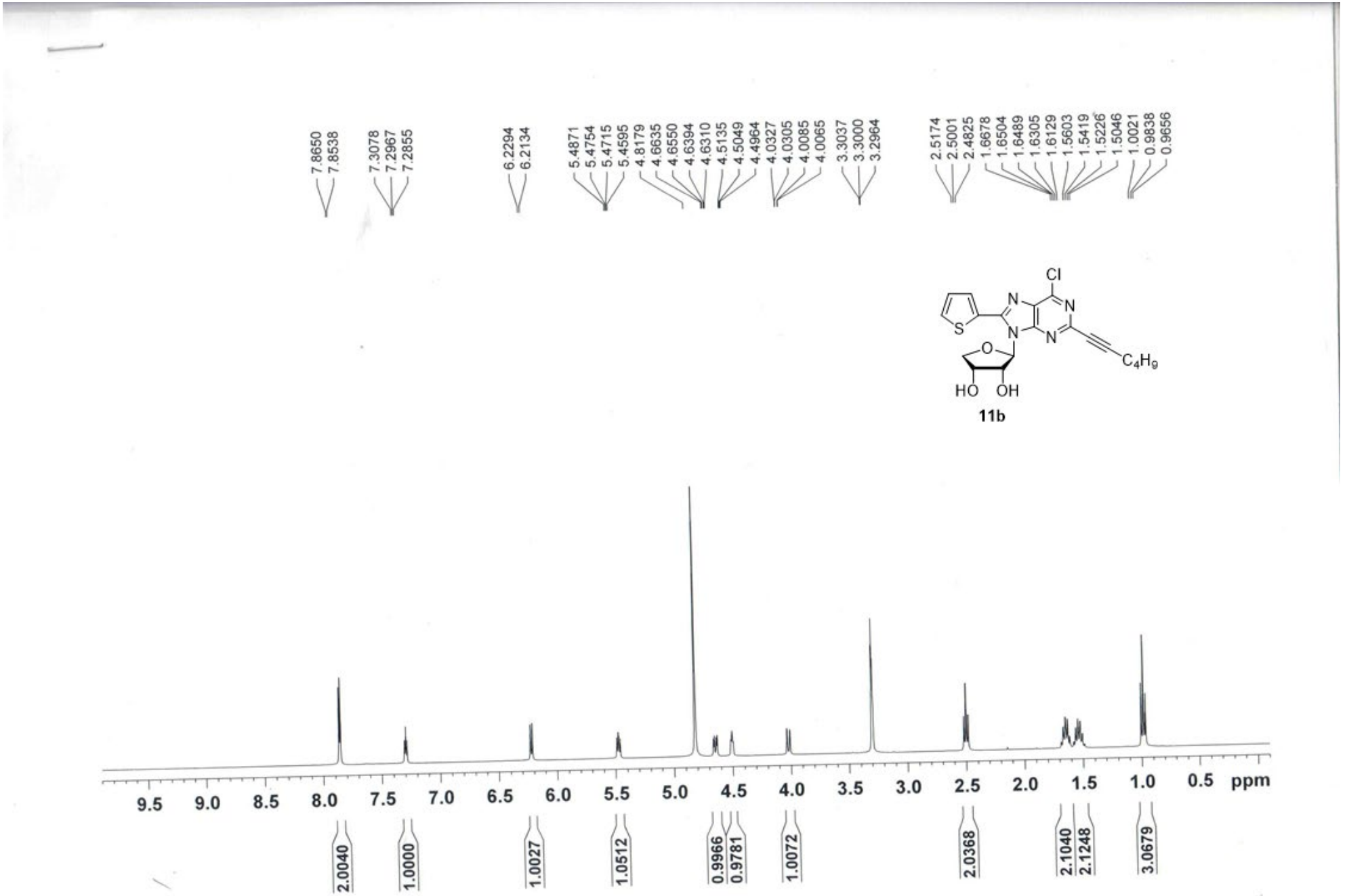
150.953
150.151
148.873
147.260
144.929
132.749
119.188
114.447
93.043
92.193
81.404
77.164
76.168
73.692
50.439
50.226
50.013
49.800
49.588
49.375
49.163
32.132
23.855
20.282
14.698
Cl
N
N
N
N
O
O
HO
OH
C_4H_9
11a
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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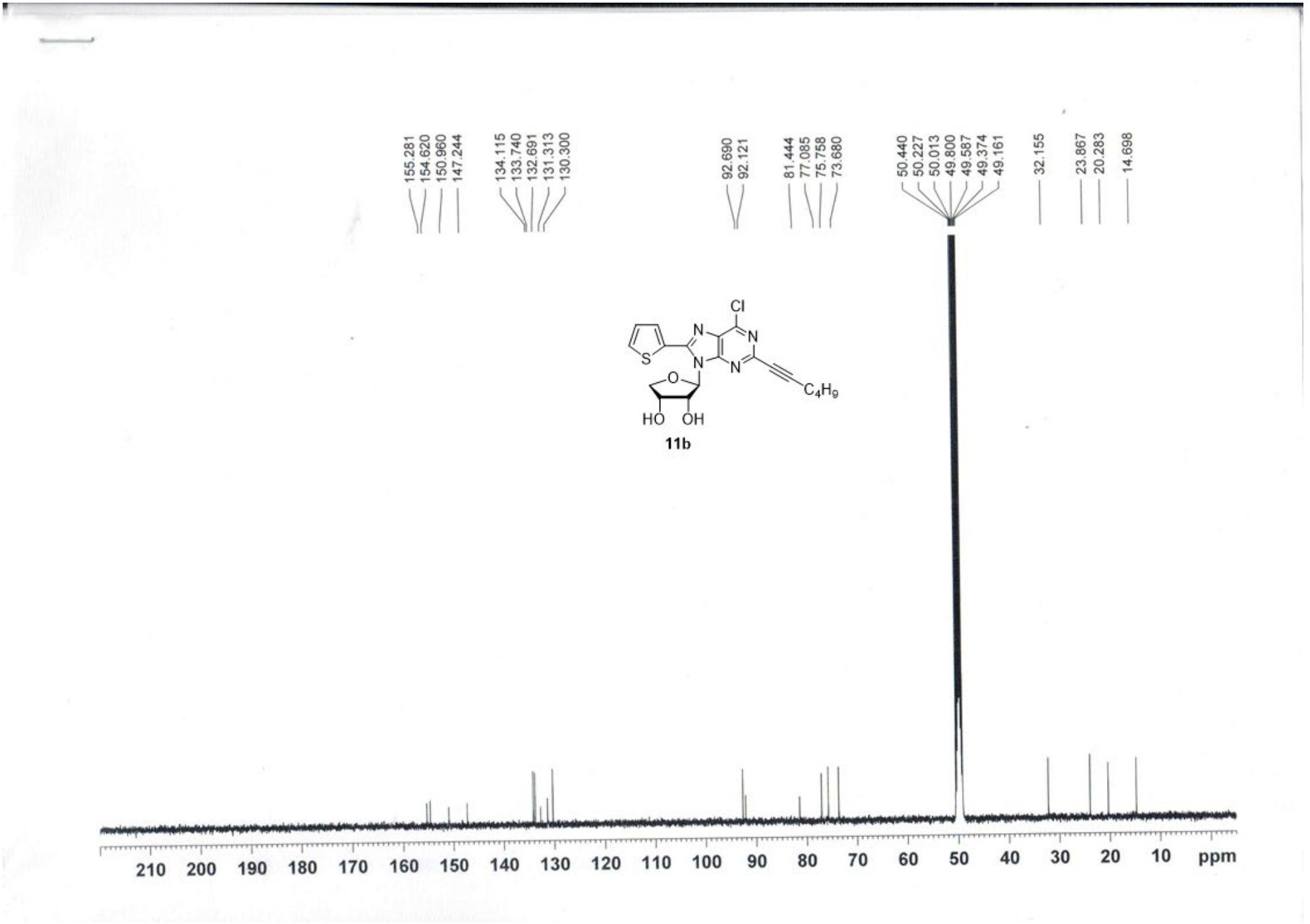
7.8538
7.3078
7.2967
7.2855
6.2294
6.2134
5.4871
5.4754
5.4715
5.4595
4.8179
4.6635
4.6550
4.6394
4.6310
4.5135
4.5049
4.4964
4.0327
4.0305
4.0085
4.0065
3.3037
3.3000
3.2964
2.5174
2.5001
2.4825
1.6678
1.6504
1.6489
1.6305
1.6129
1.5603
1.5419
1.5226
1.5046
1.0021
0.9838
0.9656
Cl
N
N
N
N
S
O
HO
OH
C4H9
11b
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
ppm
2.0040
1.0000
1.0027
1.0512
0.9966
0.9781
1.0072
2.0368
2.1040
2.1248
3.0679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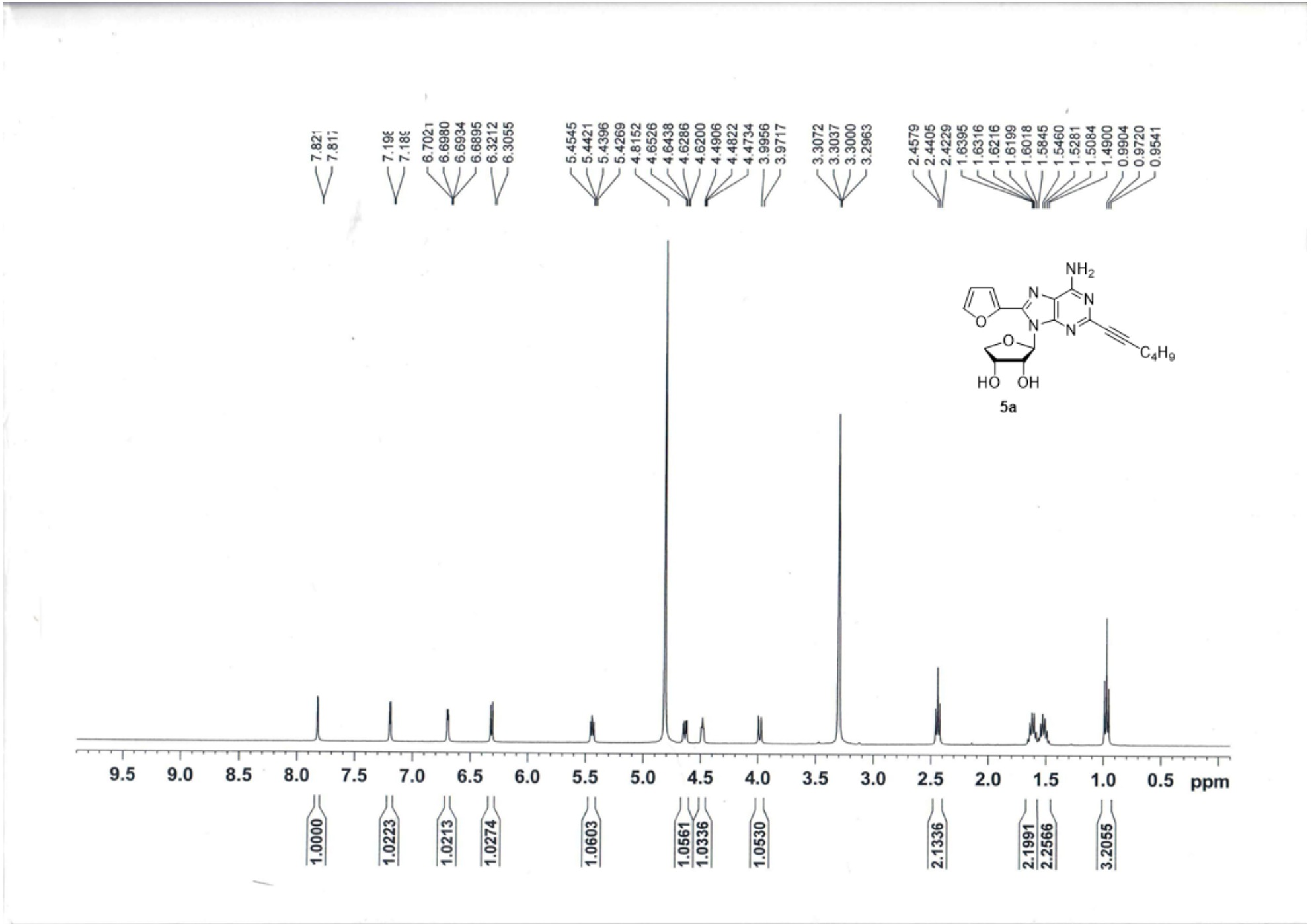
154.620
150.960
147.244
134.115
133.740
132.691
131.313
130.300
92.690
92.121
81.444
77.085
75.758
73.680
50.440
50.227
50.013
49.800
49.587
49.374
49.161
32.155
23.867
20.283
14.698
Cl
N
N
N
N
S
O
HO
OH
C_4H_9
11b
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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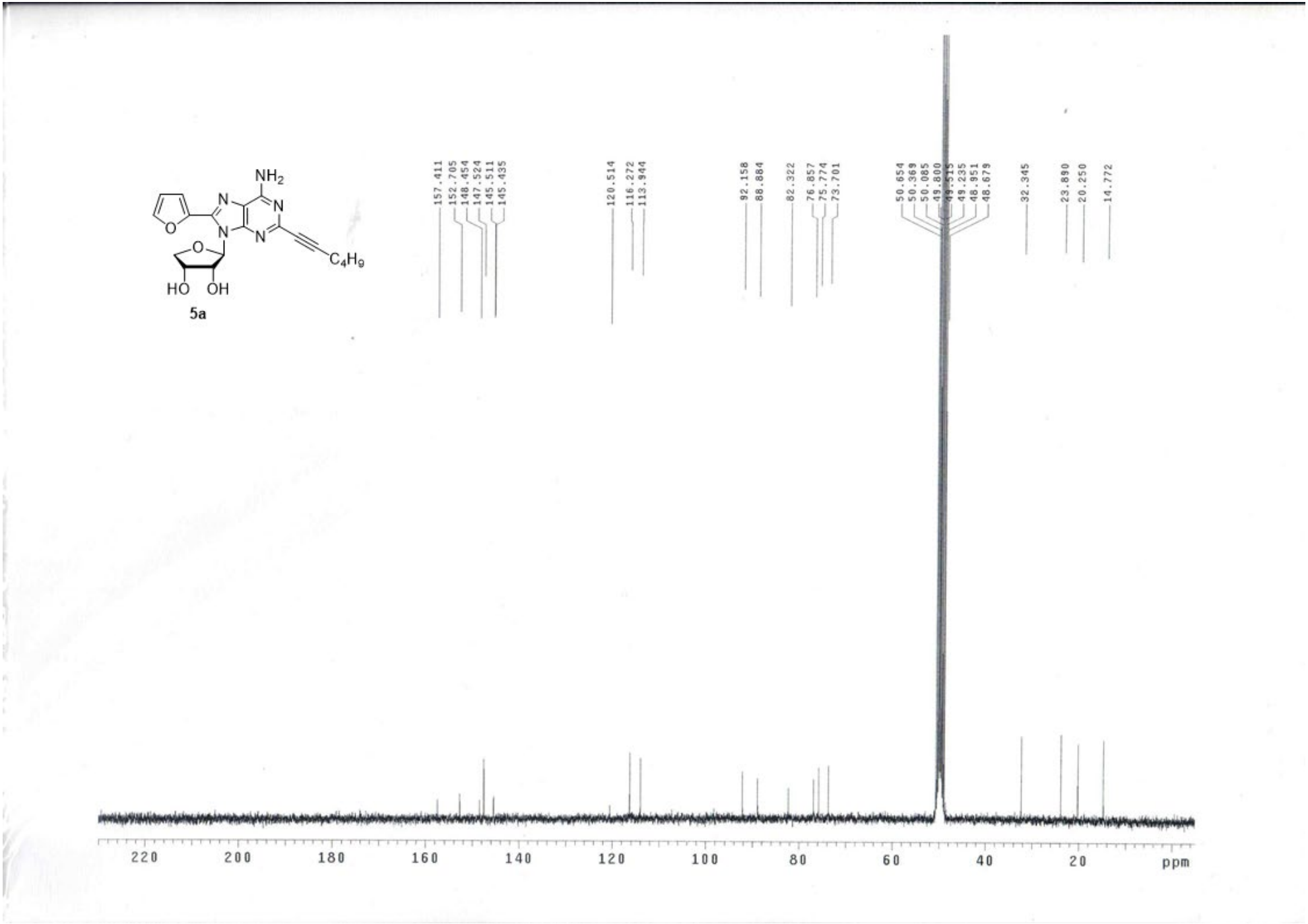
7.811
7.198
7.185
6.7021
6.6980
6.6934
6.6895
6.3212
6.3055
5.4545
5.4421
5.4396
5.4269
4.8152
4.6526
4.6438
4.6286
4.6200
4.4906
4.4822
4.4734
3.9956
3.9717
3.3072
3.3037
3.3000
3.2963
2.4579
2.4405
2.4229
1.6395
1.6316
1.6216
1.6199
1.6018
1.5845
1.5460
1.5281
1.5084
1.4900
0.9904
0.9720
0.9541
NH2
N
N
N
N
O
O
HO
OH
C4H9
5a
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
ppm
1.0000
1.0223
1.0213
1.0274
1.0603
1.0561
1.0336
1.0530
2.1336
2.1991
2.2566
3.2055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N
N
N
N
O
O
HO
OH
C4H9
5a
157.411
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148.454
147.524
145.511
145.435
120.514
116.272
113.944
92.158
88.884
82.322
76.857
75.774
73.701
50.654
50.369
50.085
49.800
49.515
49.235
48.951
48.679
32.345
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20.250
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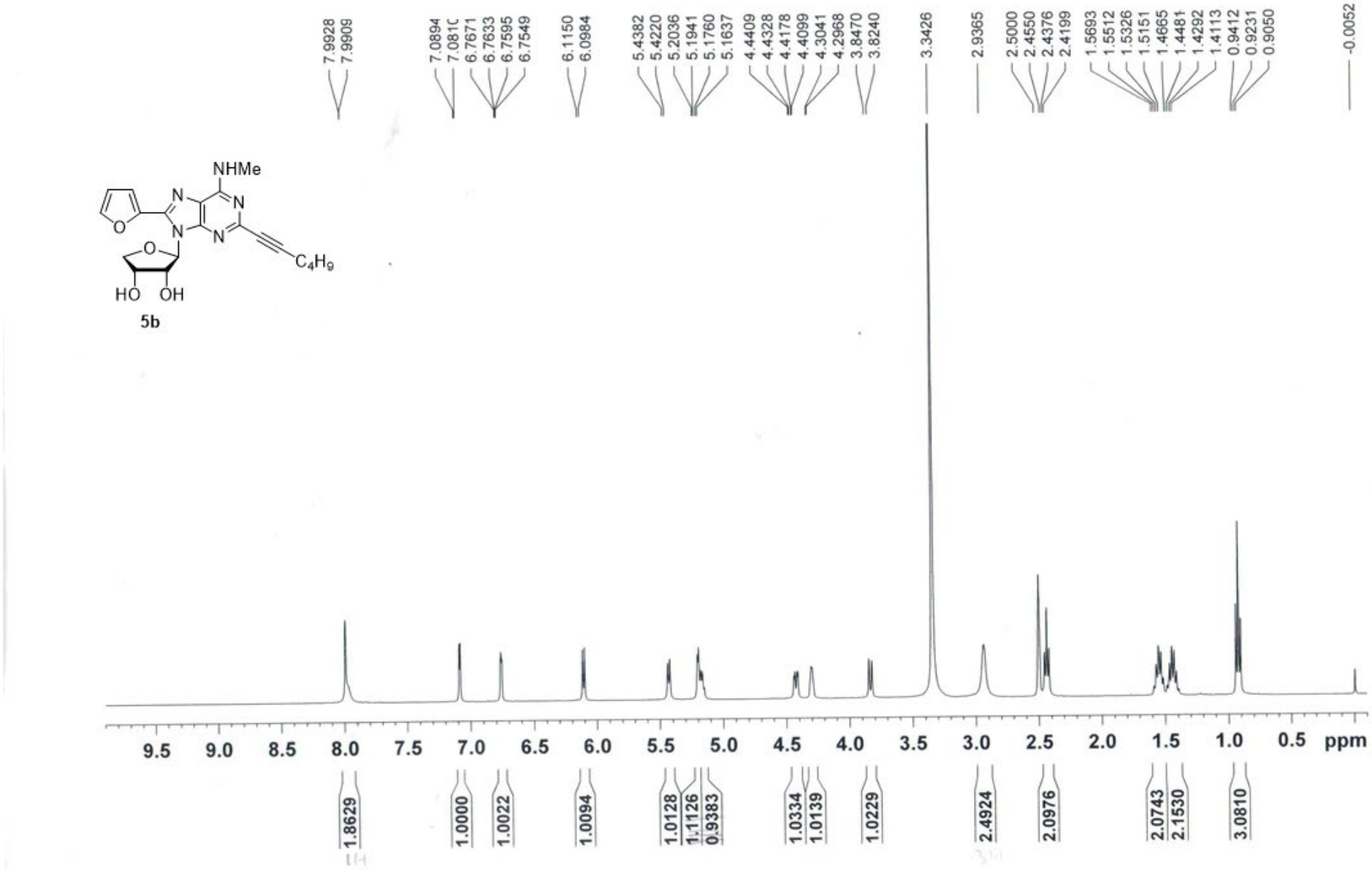
220
200
180
160
140
120
100
80
60
40
20
ppm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6.7595
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6.1150
6.0984
5.4382
5.4220
5.2036
5.1941
5.1760
5.1637
4.4409
4.4328
4.4178
4.4099
4.3041
4.2968
3.8470
3.8240
3.3426
2.9365
2.5000
2.4550
2.4376
2.4199
1.5693
1.5512
1.5326
1.5151
1.4665
1.4481
1.4292
1.4113
0.9412
0.9231
0.9050
-0.0052
NHMe
N
N
N
N
O
O
HO
OH
C4H9
5b
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
ppm
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1.0000
1.0022
1.0094
1.0128
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1.0334
1.0139
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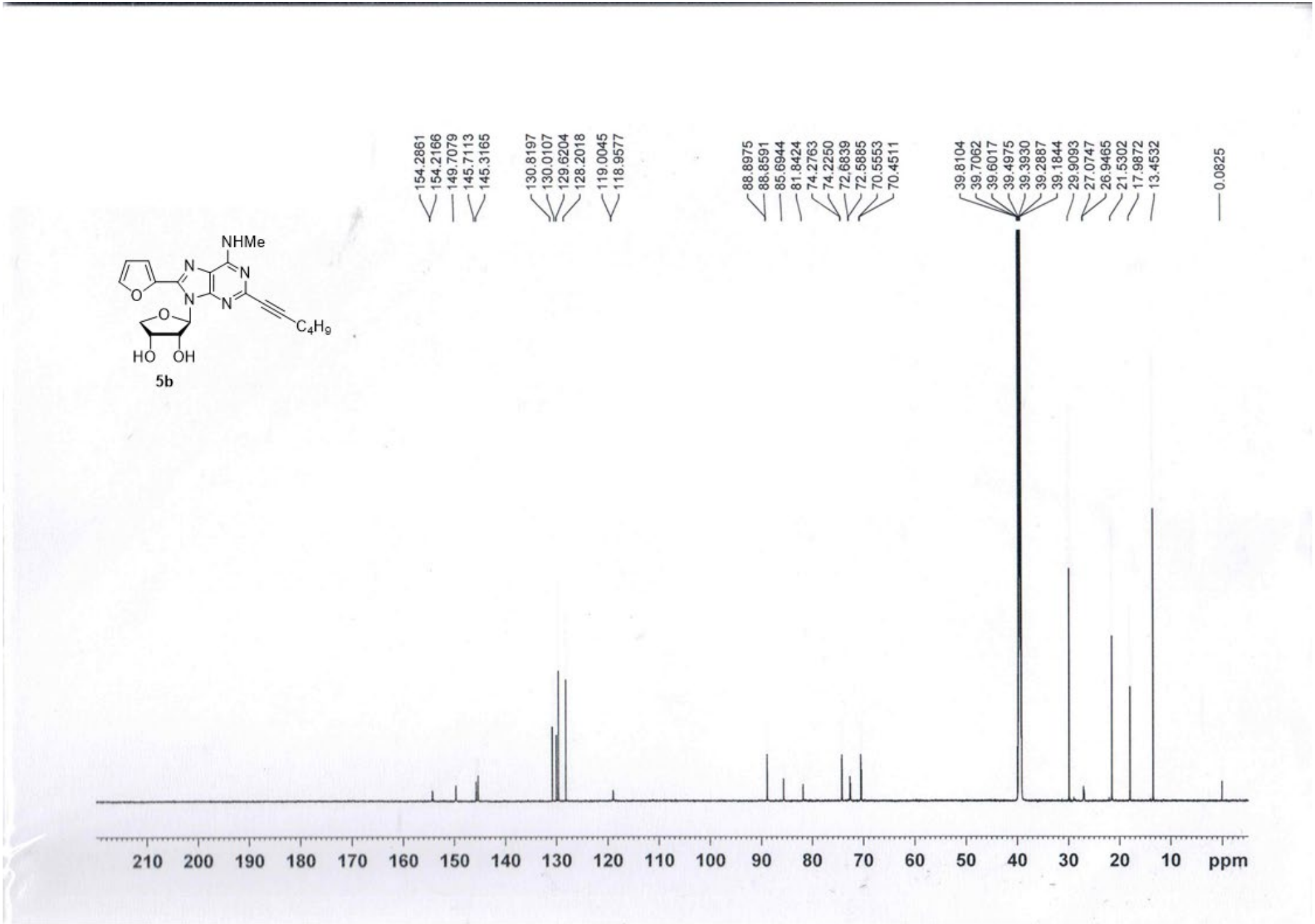
2.4924
2.0976
2.0743
2.1530
3.0810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130.0107
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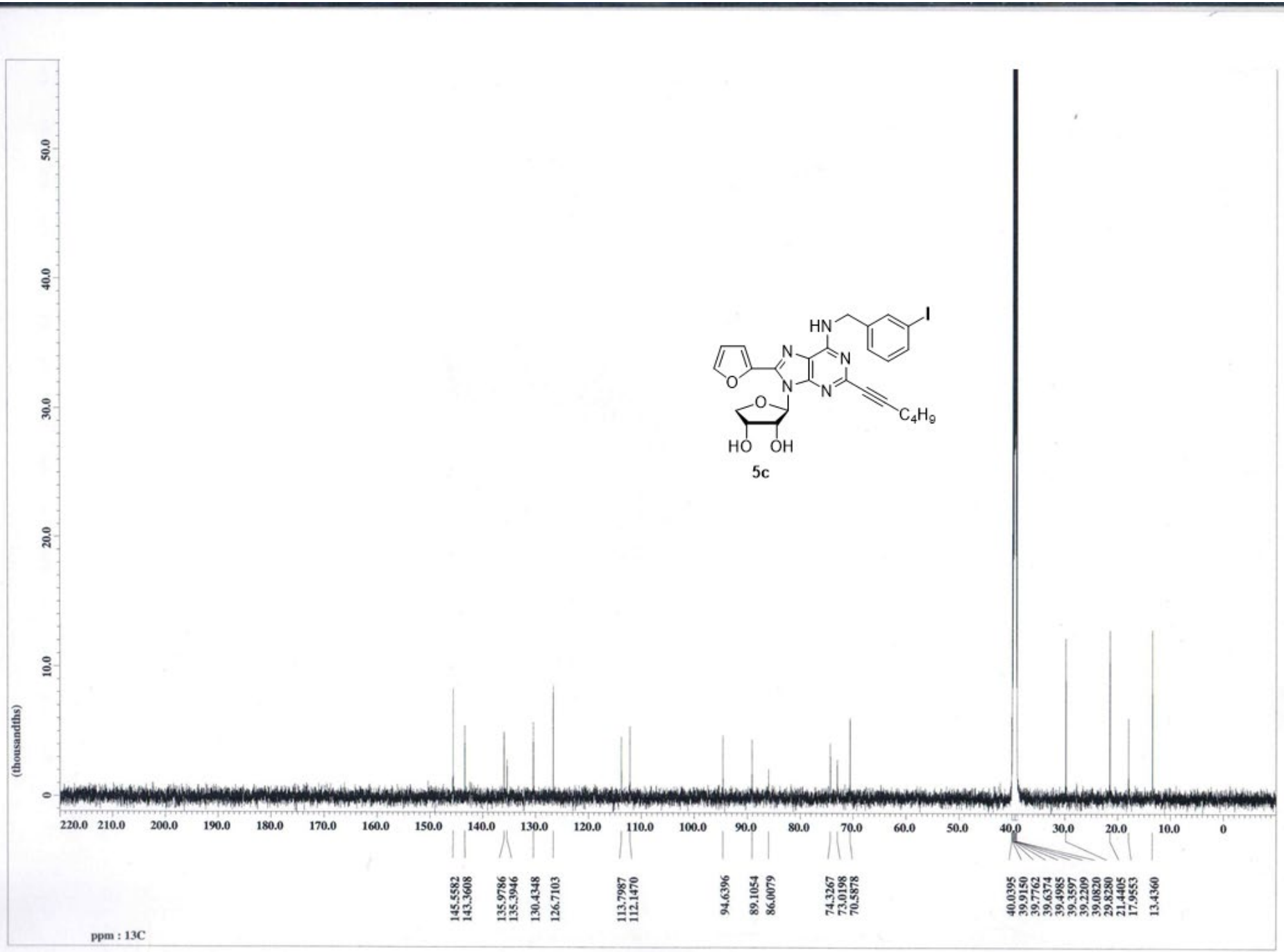
128.2018
119.0045
118.9577
88.8975
88.8591
85.6944
81.8424
74.2763
74.2250
72.6839
72.5885
70.5553
70.4511
39.8104
39.7062
39.6017
39.4975
39.3930
39.2887
39.1844
29.9093
27.0747
26.9465
21.5302
17.9872
13.4532
0.0825
NHMe
N
N
N
N
O
O
HO
OH
C4H9
5b
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


4.854
3.318
3.314
3.310
3.306
3.302
HN
I
N
N
N
N
O
O
HO
OH
C_4H_9
5c
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.07
0.73
0.86
0.75
0.93
0.74
0.87
1.01
1.49
1.01
0.99
1.00
1.67
1.72
1.66
1.95


HN
I
N
N
N
N
O
O
HO
OH
C_4H_9
5c
(thousandths)
50.0
40.0
30.0
20.0
10.0
0
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
145.5582
143.3608
135.9786
135.3946
130.4348
126.7103
113.7987
112.1470
94.6396
89.1054
86.0079
74.3267
73.0198
70.5878
40.0395
39.9150
39.7762
39.6374
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39.2209
39.0820
29.8280
21.4405
17.9553
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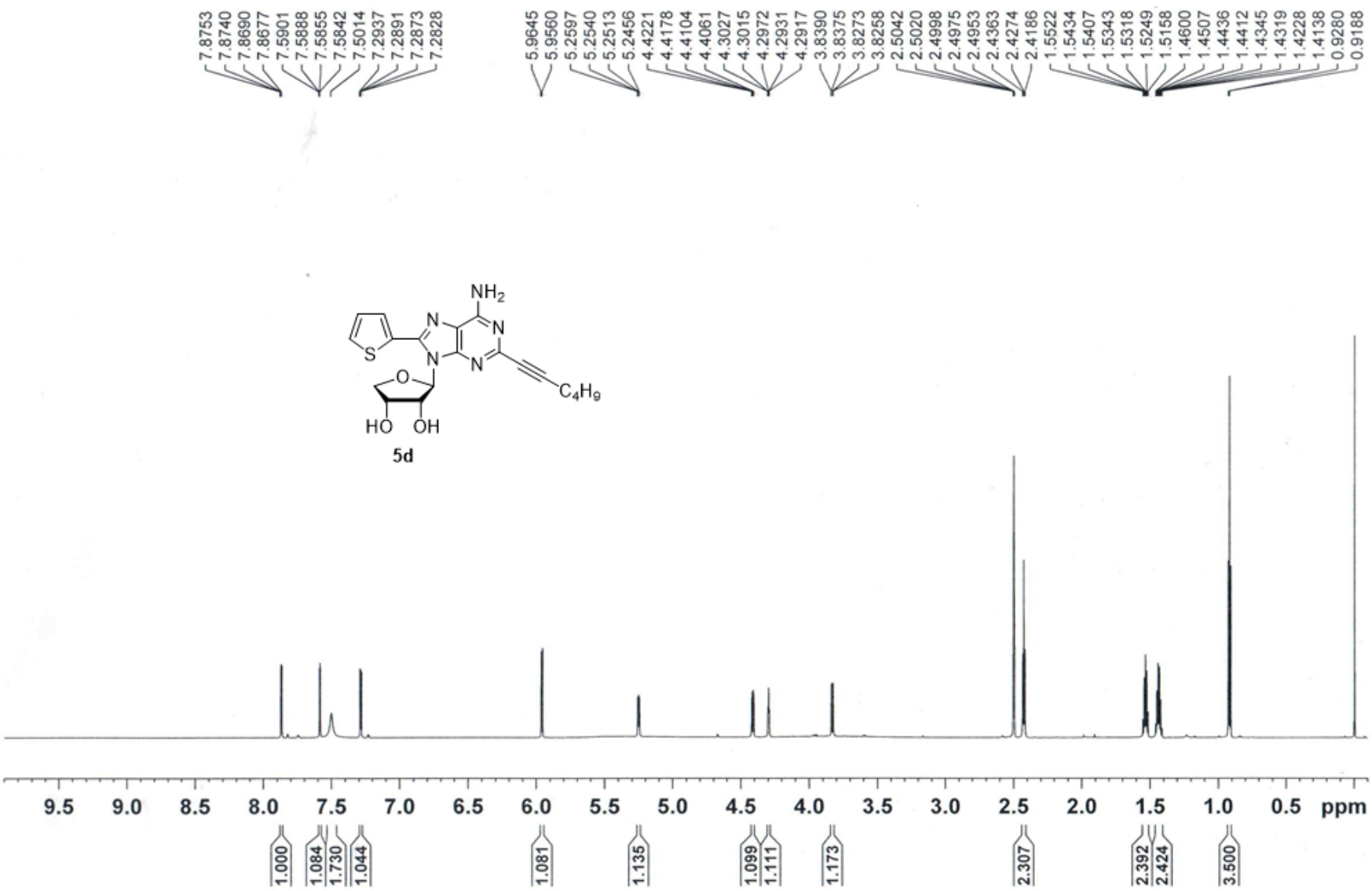
ppm : 13C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7.5888
7.5855
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7.2891
7.2873
7.2828
5.9645
5.9560
5.2597
5.2540
5.2513
5.2456
4.4221
4.4178
4.4104
4.4061
4.3027
4.3015
4.2972
4.2931
4.2917
3.8390
3.8375
3.8273
3.8258
2.5042
2.5020
2.4998
2.4975
2.4953
2.4363
2.4274
2.4186
1.5522
1.5434
1.5407
1.5343
1.5318
1.5249
1.5158
1.4600
1.4507
1.4436
1.4412
1.4345
1.4319
1.4228
1.4138
0.9280
0.9188
NH2
N
N
N
N
S
O
HO
OH
C4H9
5d
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
ppm
1.000
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1.730
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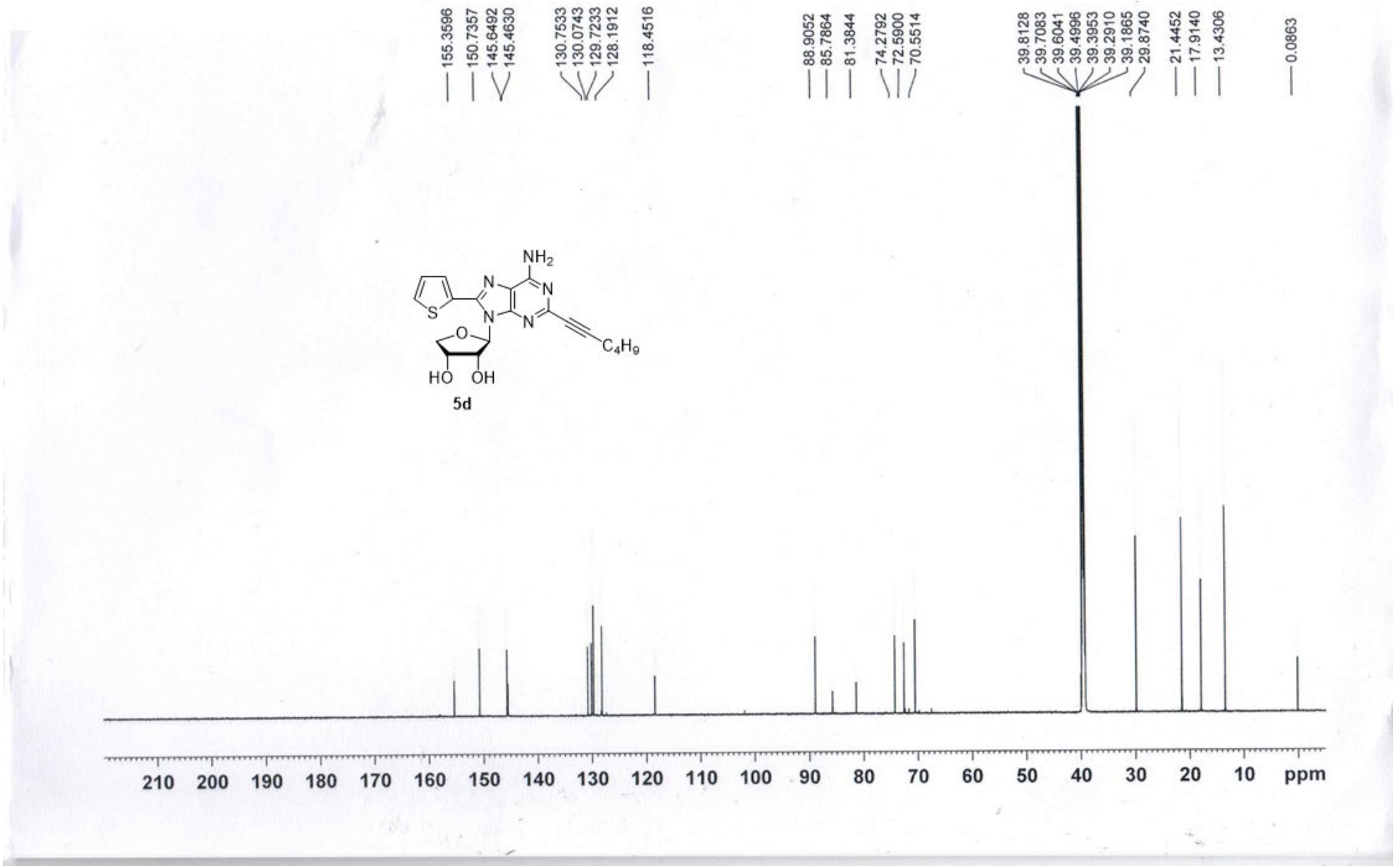
1.081
1.135
1.099
1.111
1.173
2.307
2.392
2.424
3.500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145.4630
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130.0743
129.7233
128.1912
118.4516
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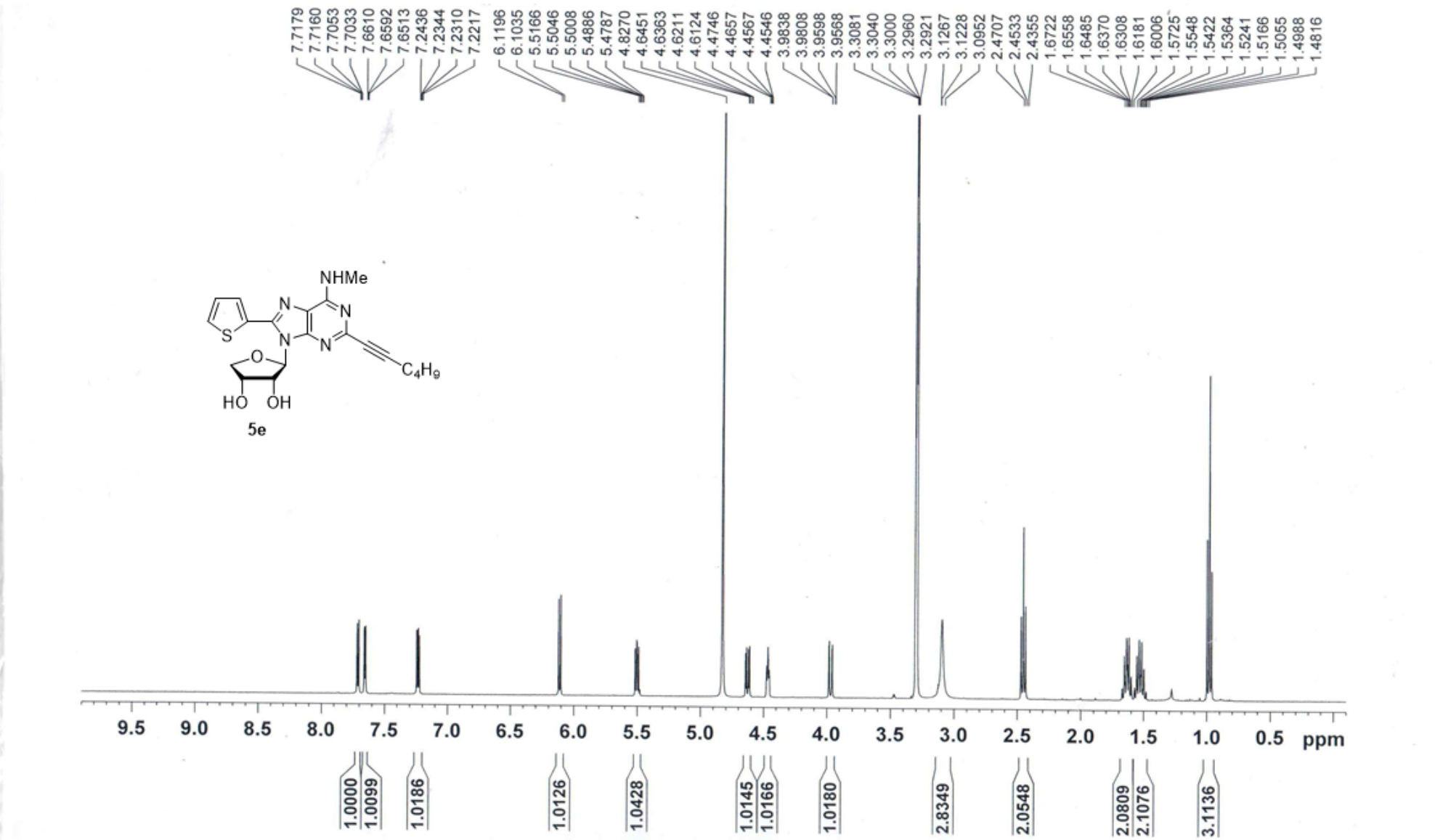
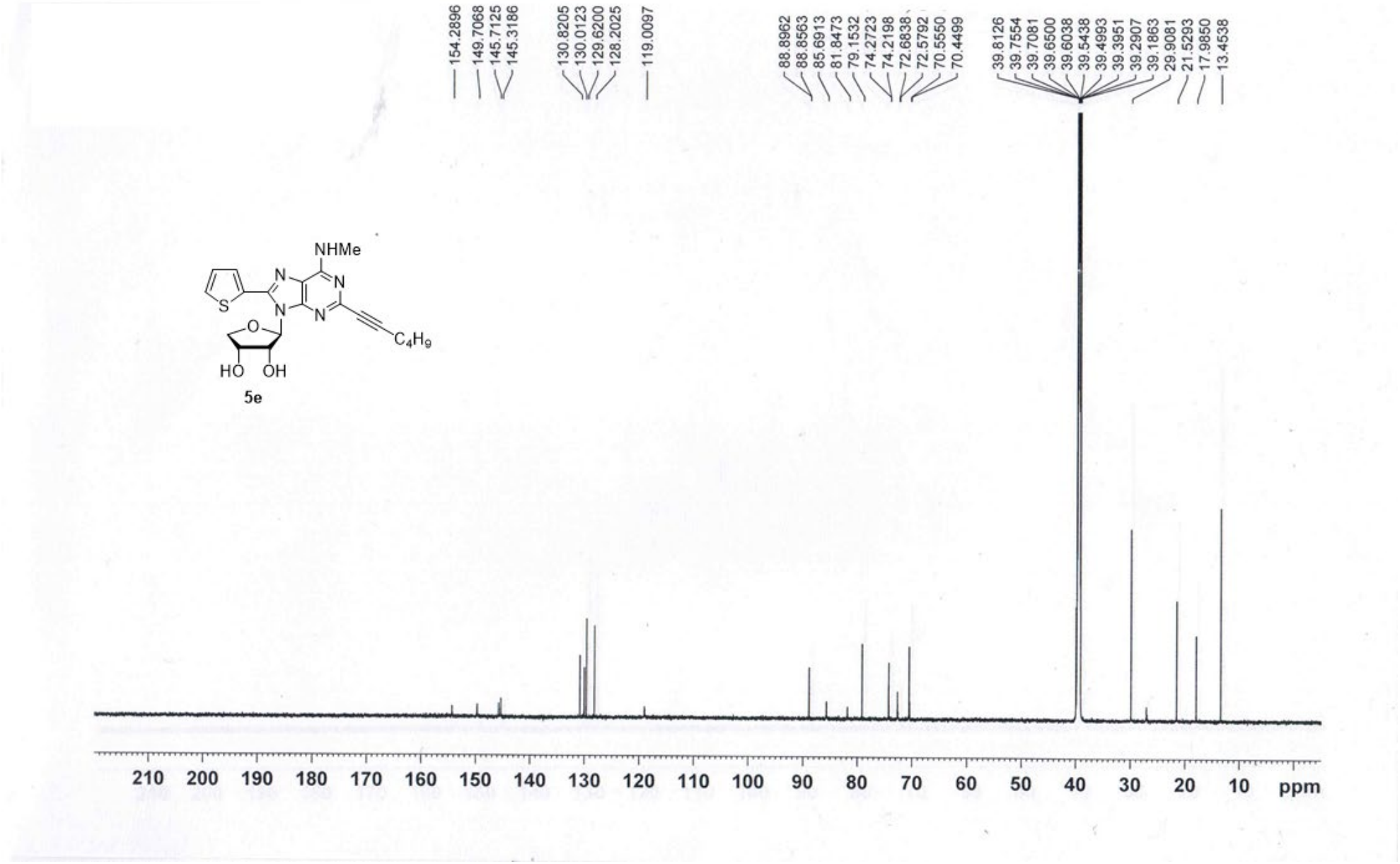
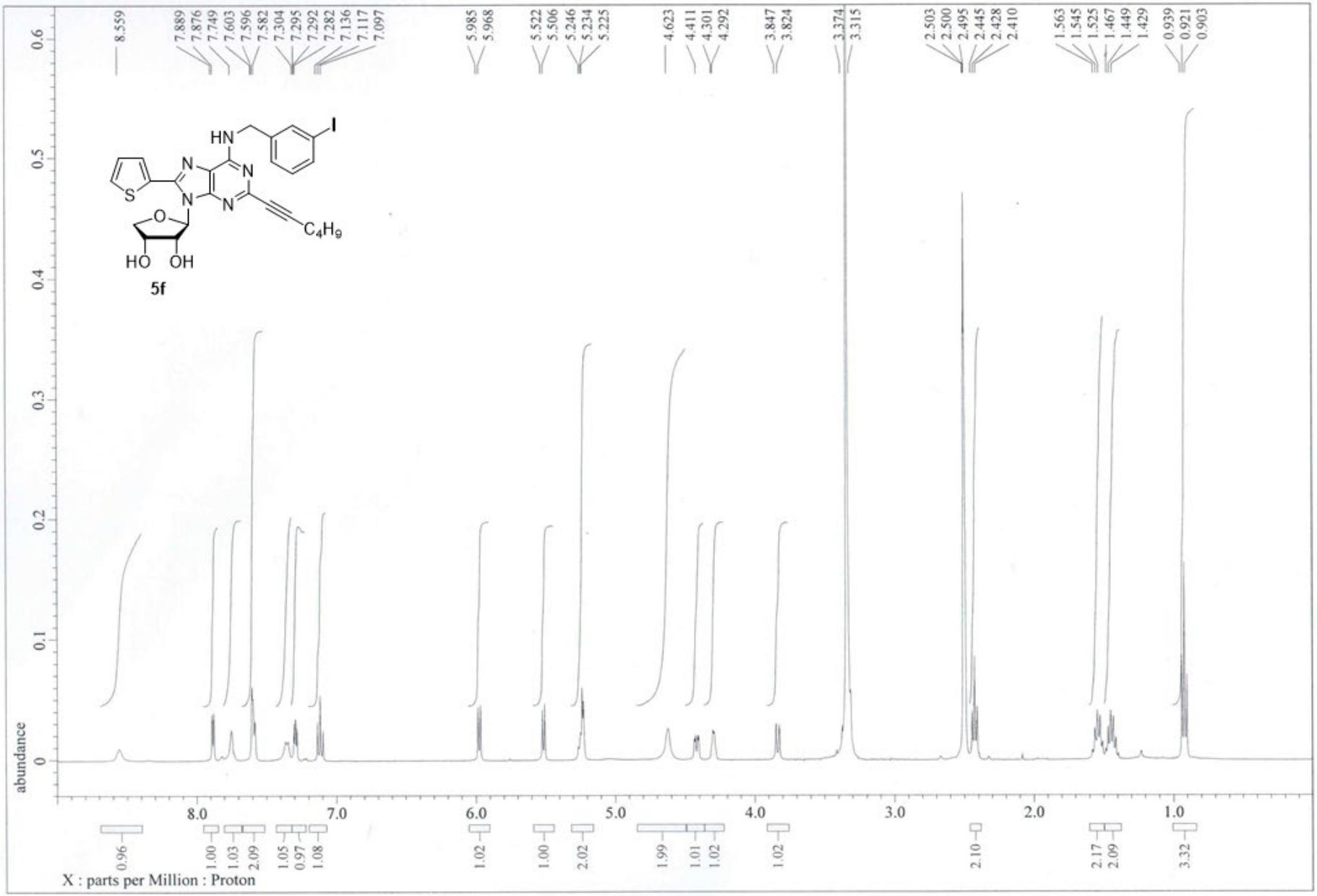
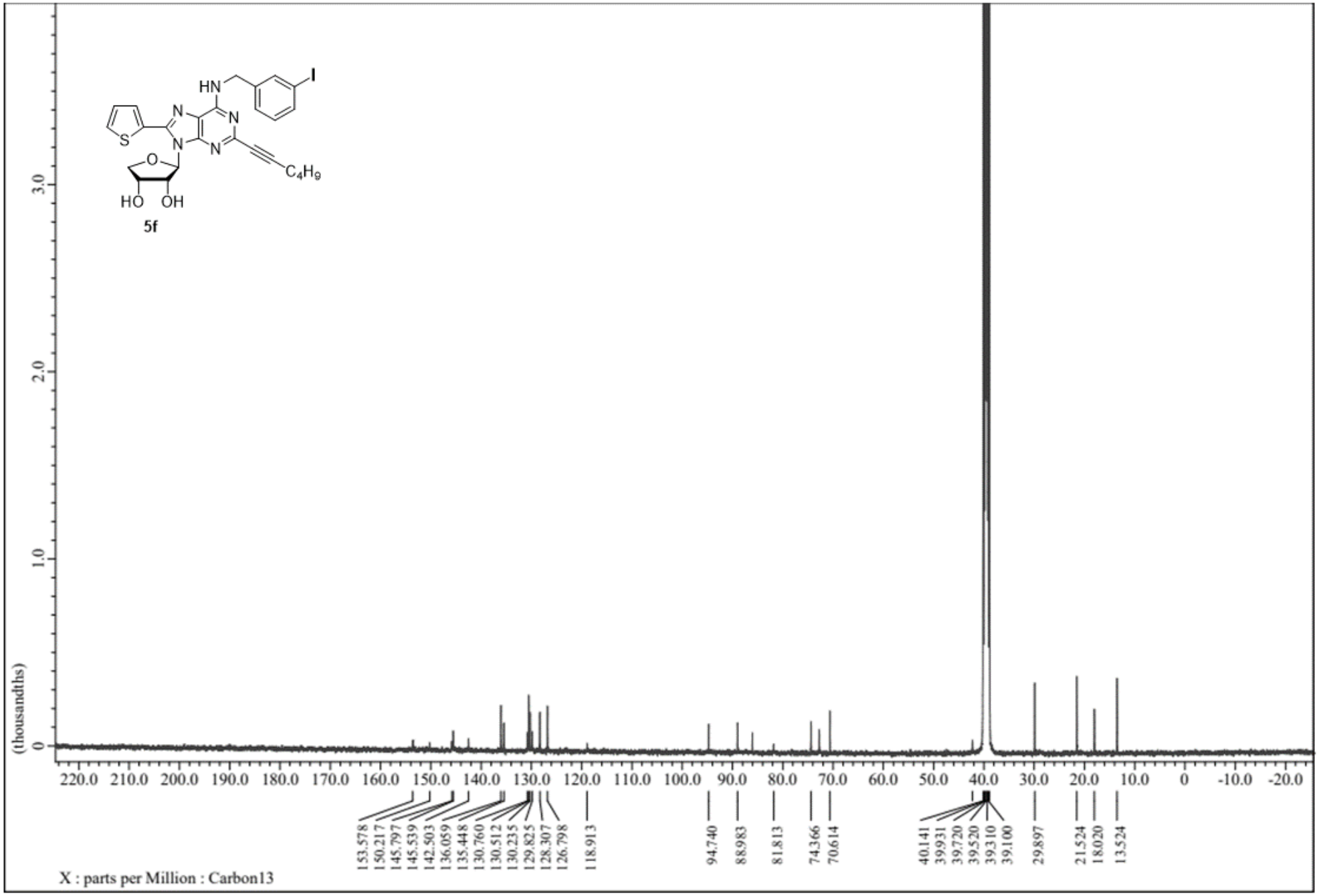
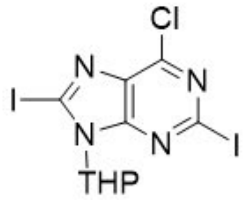
85.7864
81.3844
74.2792
72.5900
70.5514
39.8128
39.7083
39.6041
39.4996
39.3953
39.2910
39.1865
29.8740
21.4452
17.9140
13.4306
0.0863
NH2
N
N
N
N
S
O
HO
OH
C4H9
5d
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


7.7179
7.7160
7.7053
7.7033
7.6610
7.6592
7.6513
7.2436
7.2344
7.2310
7.2217
6.1196
6.1035
5.5166
5.5046
5.5008
5.4886
5.4787
4.8270
4.6451
4.6363
4.6211
4.6124
4.4746
4.4657
4.4567
4.4546
3.9838
3.9808
3.9598
3.9568
3.3081
3.3040
3.3000
3.2960
3.2921
3.1267
3.1228
3.0952
2.4707
2.4533
2.4355
1.6722
1.6558
1.6485
1.6370
1.6308
1.6181
1.6006
1.5725
1.5548
1.5422
1.5364
1.5241
1.5166
1.5055
1.4988
1.4816
NHMe
N
N
N
N
S
O
HO
OH
C_4H_9
5e
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
ppm
1.0000
1.0099
1.0186
1.0126
1.0428
1.0145
1.0166
1.0180
2.8349
2.0548
2.0809
2.1076
3.1136

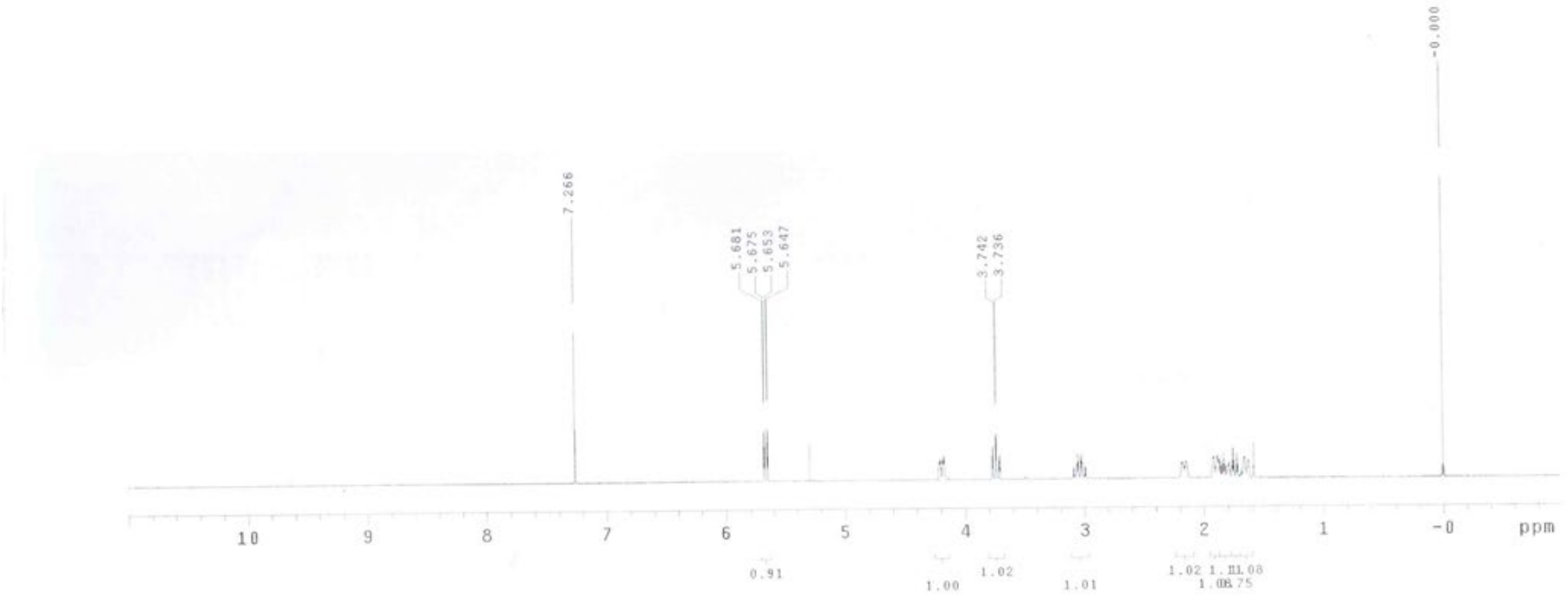

154.2896
149.7068
145.7125
145.3186
130.8205
130.0123
129.6200
128.2025
119.0097
88.8962
88.8563
85.6913
81.8473
79.1532
74.2723
74.2198
72.6838
72.5792
70.5550
70.4499
39.8126
39.7554
39.7081
39.6500
39.6038
39.5438
39.4993
39.3951
39.2907
39.1863
29.9081
21.5293
17.9850
13.4538
NHMe
N
N
N
N
S
O
HO
OH
C4H9
5e
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

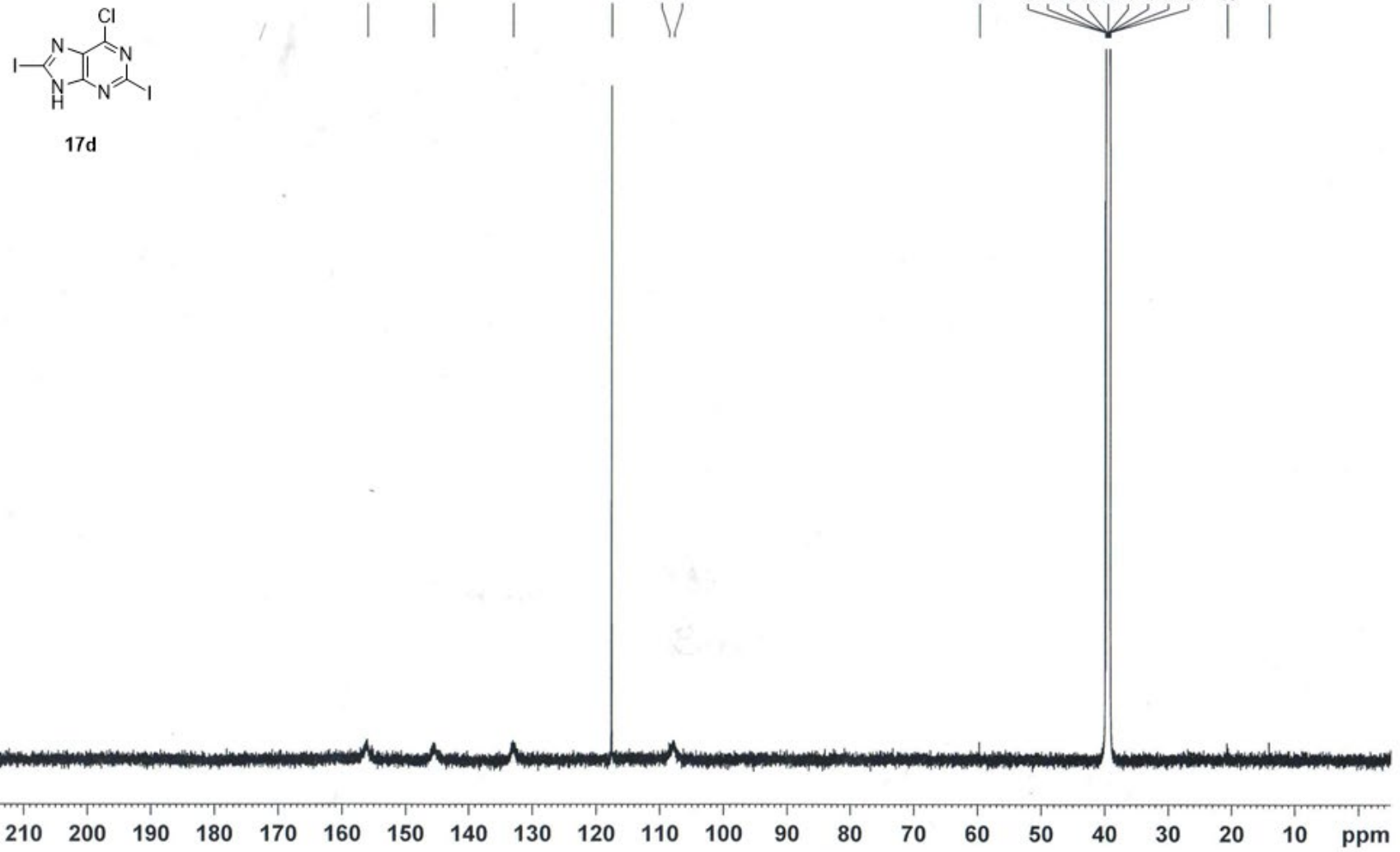

8.559
7.889
7.876
7.749
7.603
7.596
7.582
7.304
7.295
7.292
7.282
7.136
7.117
7.097
5.985
5.968
5.522
5.506
5.246
5.234
5.225
4.623
4.411
4.301
4.292
3.847
3.824
3.374
3.315
2.503
2.500
2.495
2.445
2.428
2.410
1.563
1.545
1.525
1.467
1.449
1.429
0.939
0.921
0.903
HN
I
N
N
N
N
S
O
HO
OH
C4H9
5f
abundance
0
0.1
0.2
0.3
0.4
0.5
0.6
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.96
1.00
1.03
2.09
1.05
0.97
1.08
1.02
1.00
2.02
1.99
1.01
1.02
1.02
2.10
2.17
2.09
3.32
X : parts per Million : Proton

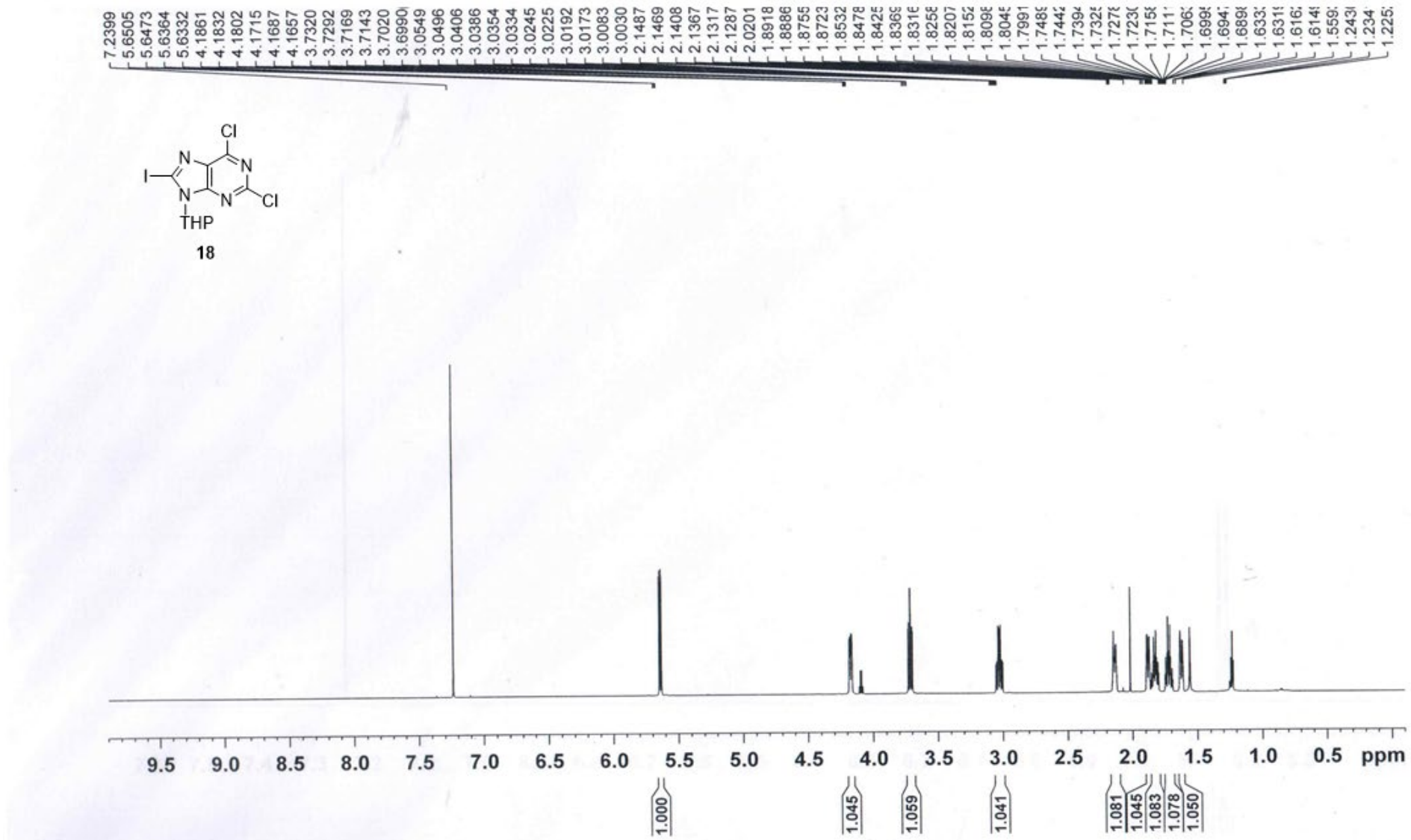

HN
I
N
N
N
N
S
O
HO
OH
C_4H_9
5f
(thousandths)
3.0
2.0
1.0
0
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
153.578
150.217
145.797
145.539
142.503
136.059
135.448
130.760
130.512
130.235
129.825
128.307
126.798
118.913
94.740
88.983
81.813
74.366
70.614
40.141
39.931
39.720
39.520
39.310
39.100
29.897
21.524
18.020
13.524
X : parts per Million : Carbon13

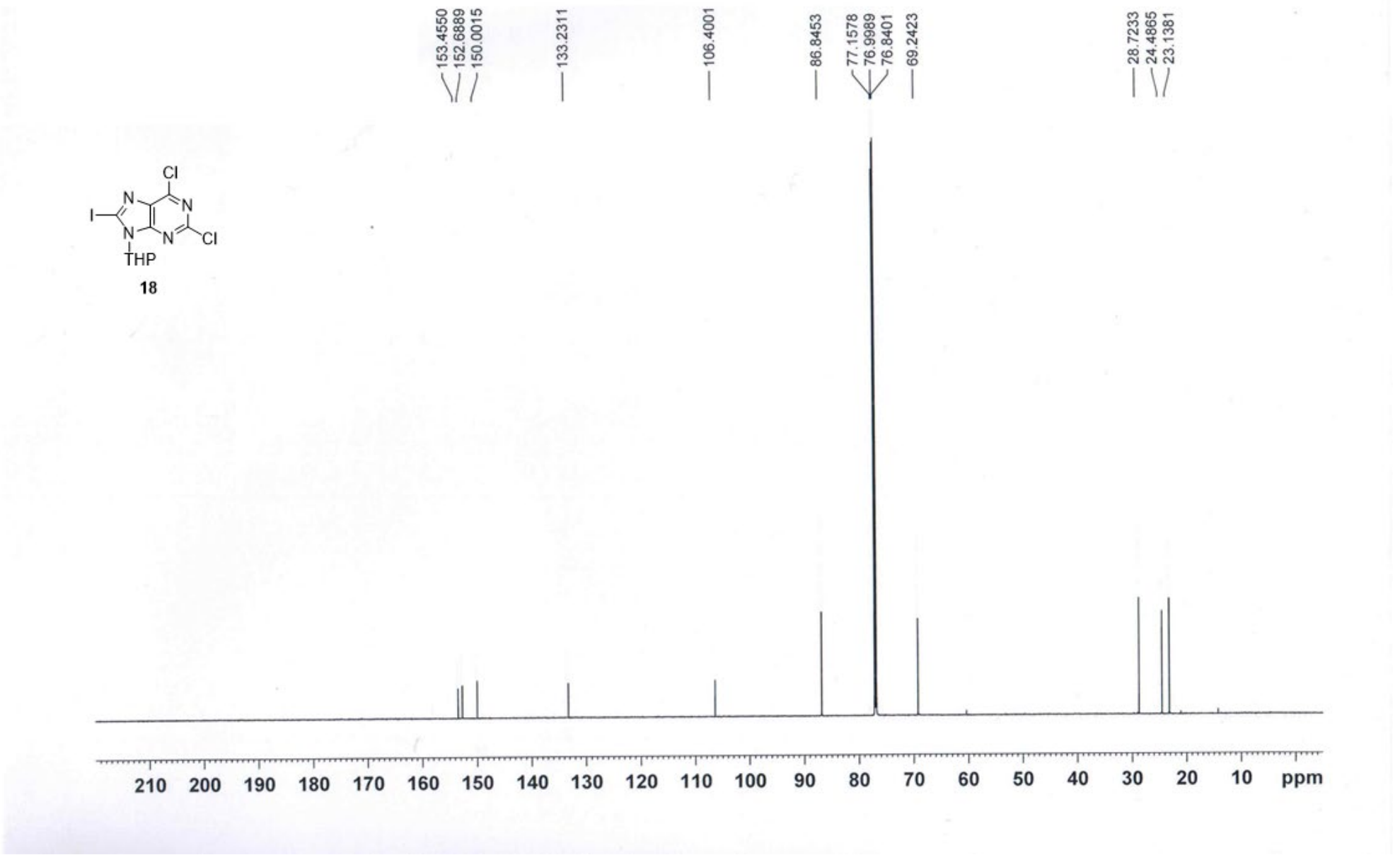

Cl
N
N
I
N
N
I
THP

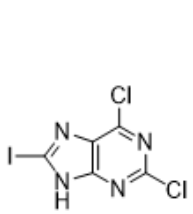
17c


7.266
5.681
5.675
5.653
5.647
3.742
3.736
0.000
10
9
8
7
6
5
4
3
2
1
0
ppm
0.91
1.00
1.02
1.01
1.02
1.11
1.08
1.08
1.75


155.9773
145.5938
133.0028
117.6236
108.5564
107.7838
59.7287
39.9652
39.8604
39.8112
39.7070
39.6026
39.4982
39.3939
39.2894
39.1852
20.7460
14.0662
Cl
N
N
I
N
H
N
I
17d
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

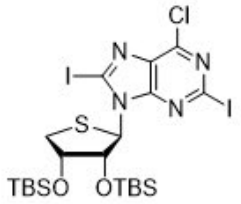

7.2399
5.6505
5.6473
5.6364
5.6332
4.1861
4.1832
4.1802
4.1715
4.1687
4.1657
3.7320
3.7292
3.7169
3.7143
3.7020
3.0549
3.0496
3.0406
3.0386
3.0354
3.0334
3.0245
3.0225
3.0192
3.0173
3.0083
3.0030
2.1487
2.1469
2.1408
2.1367
2.1317
2.1287
2.0201
1.8918
1.8886
1.8755
1.8723
1.8532
1.8478
1.8425
1.8365
1.8316
1.8258
1.8207
1.8152
1.8098
1.8045
1.7991
1.7485
1.7442
1.7394
1.7325
1.7278
1.7230
1.7158
1.7111
1.7065
1.6998
1.6947
1.6898
1.6331
1.6165
1.6148
1.5593
1.2431
1.2341
1.2253
Cl
N
N
I
N
N
Cl
THP
18
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
ppm
1.000
1.045
1.059
1.041
1.081
1.045
1.083
1.078
1.050


153.4550
152.6889
150.0015
133.2311
106.4001
86.8453
77.1578
76.9989
76.8401
69.2423
28.7233
24.4865
23.1381
Cl
N
N
I
N
N
Cl
THP
18
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


Cl
N
N
I
N
H
N
Cl

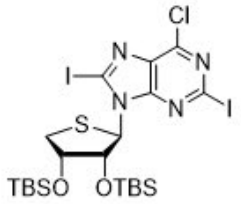
19

152.6754
147.0034
134.2622
115.1913
50.1182
50.0118
49.9054
49.7991
49.6929
49.5867
49.4802
48.6917
10.0535
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


Cl
N
N
I
N
N
I
S
TBSO
OTBS

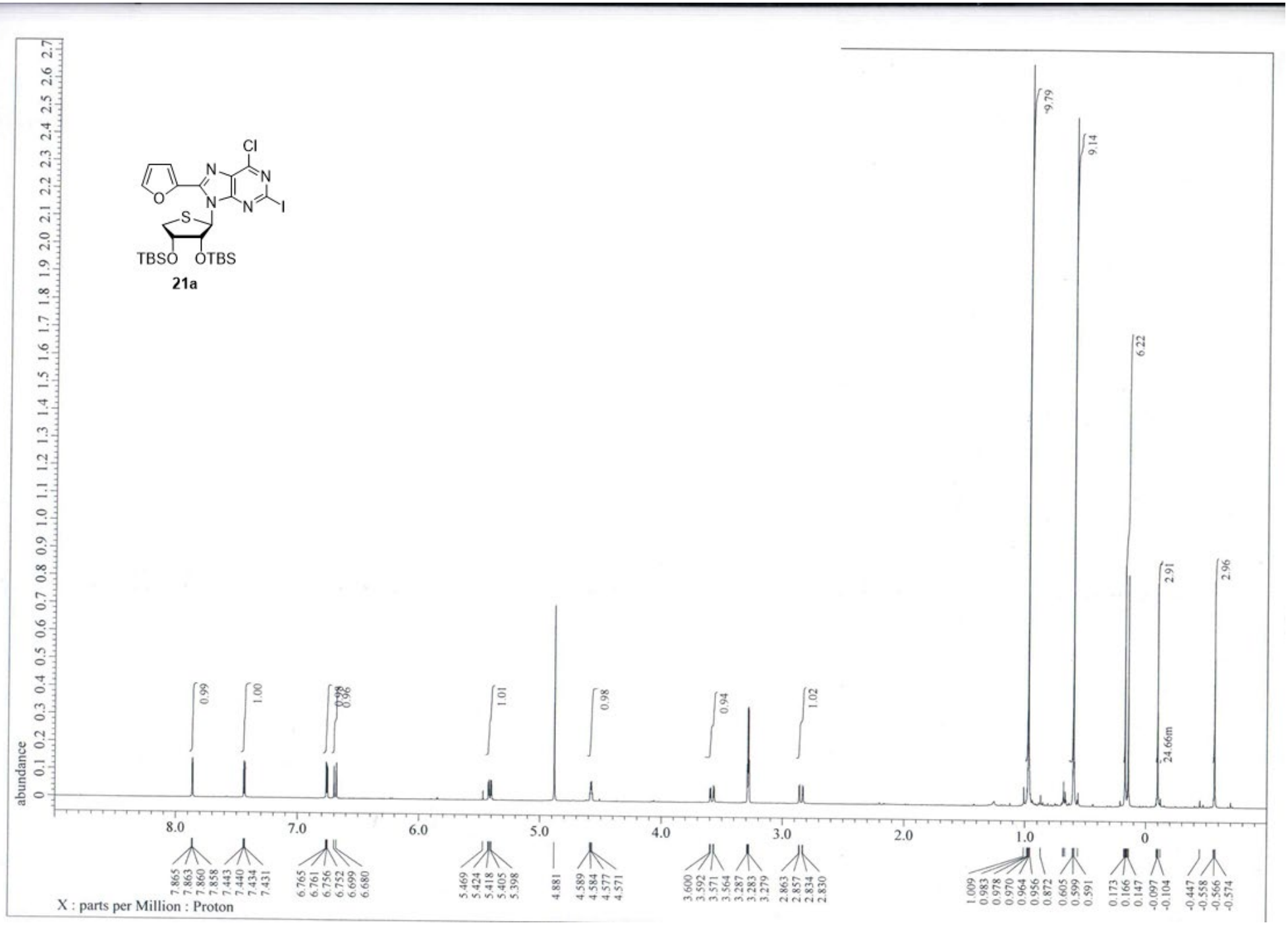
20

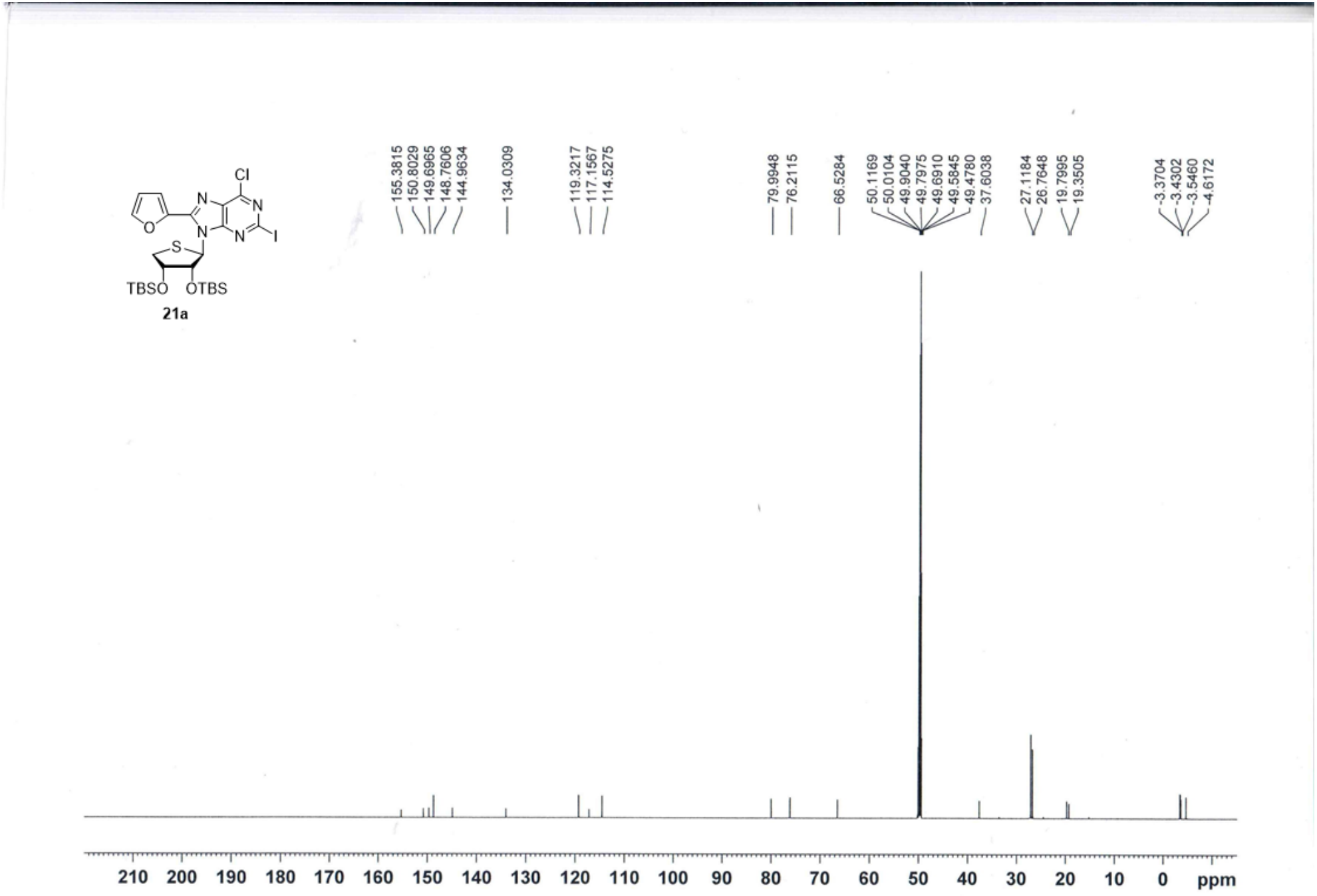
0.889
0.743
0.174
0.167
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
0.78
0.90
0.95
1.00
1.26
2.19
7.77
7.76
4.98
2.35
2.34

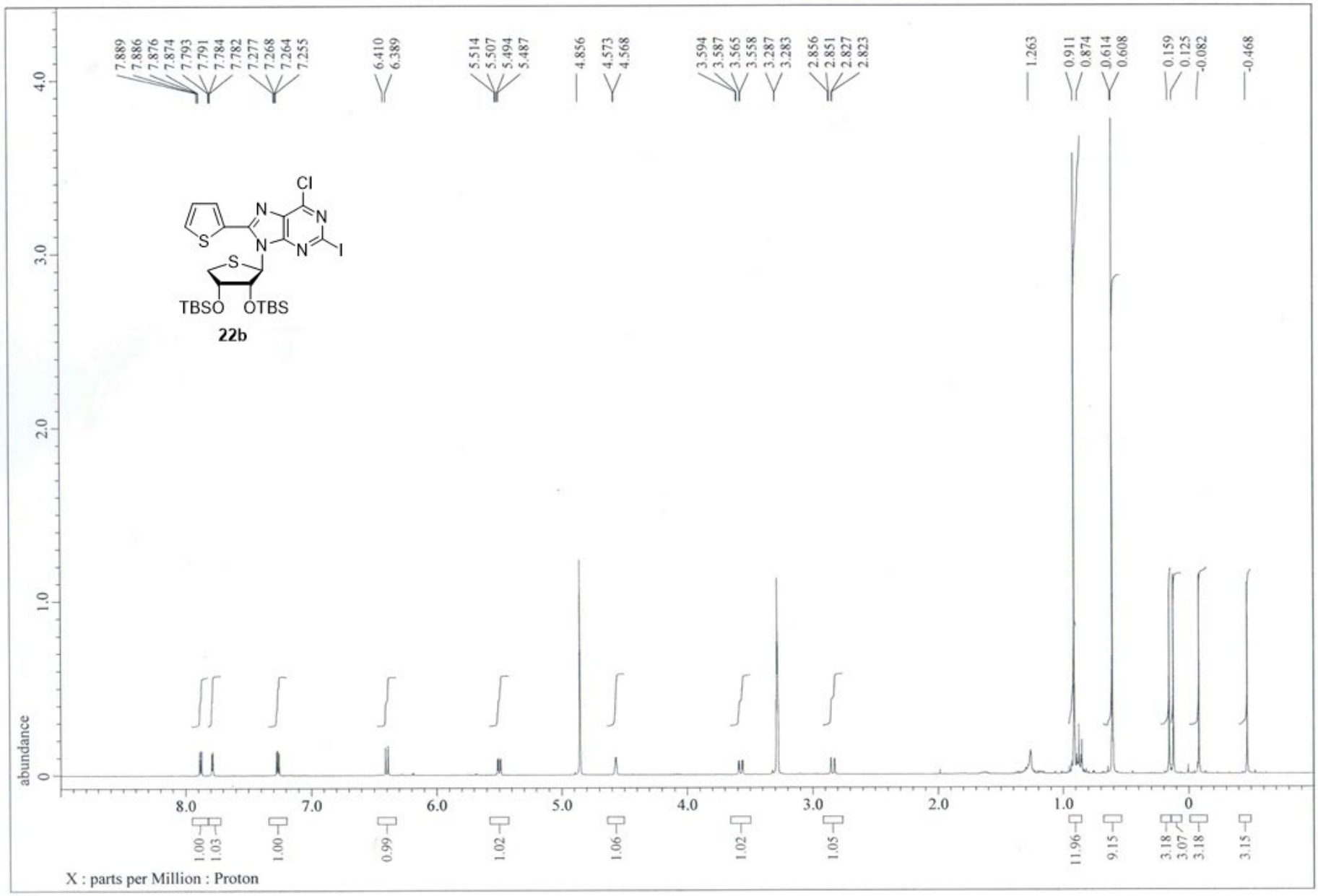

Cl
N
N
I
N
N
I
S
TBSO
OTBS

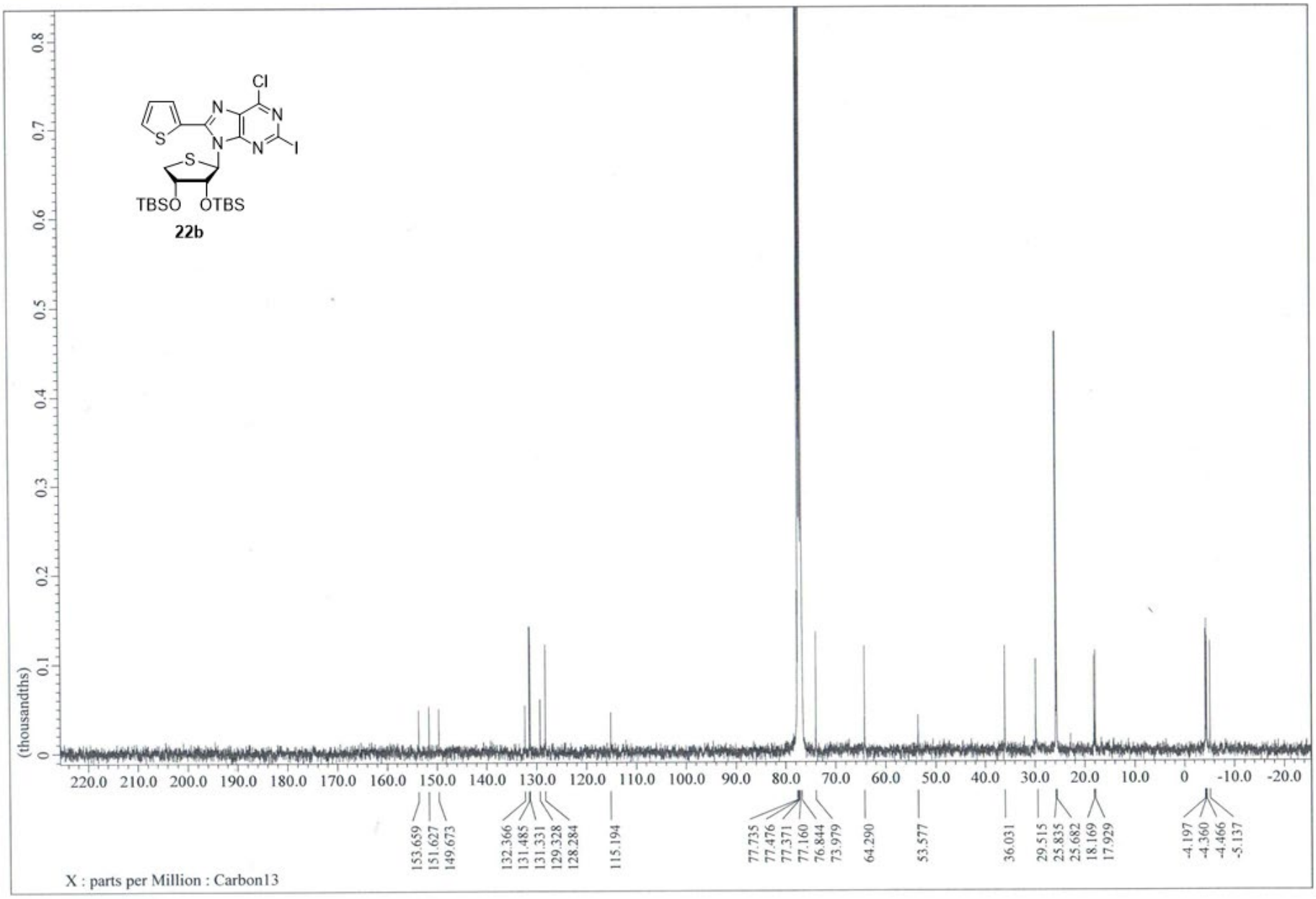
20

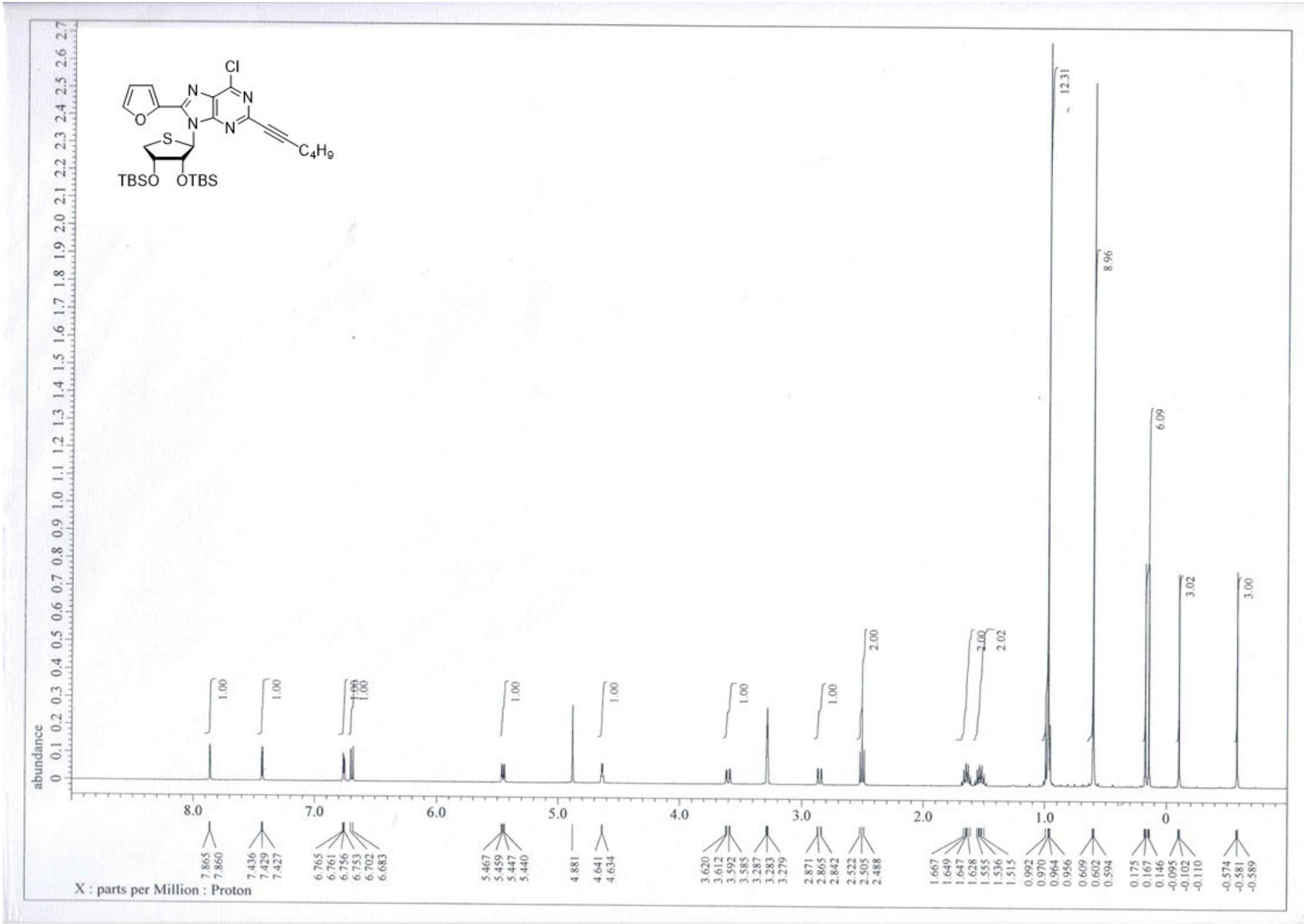
49.268
49.050
48.840
26.452
26.219
49.478
48.630
75.445
220
200
180
160
140
120
100
80
60
40
20
0
ppm

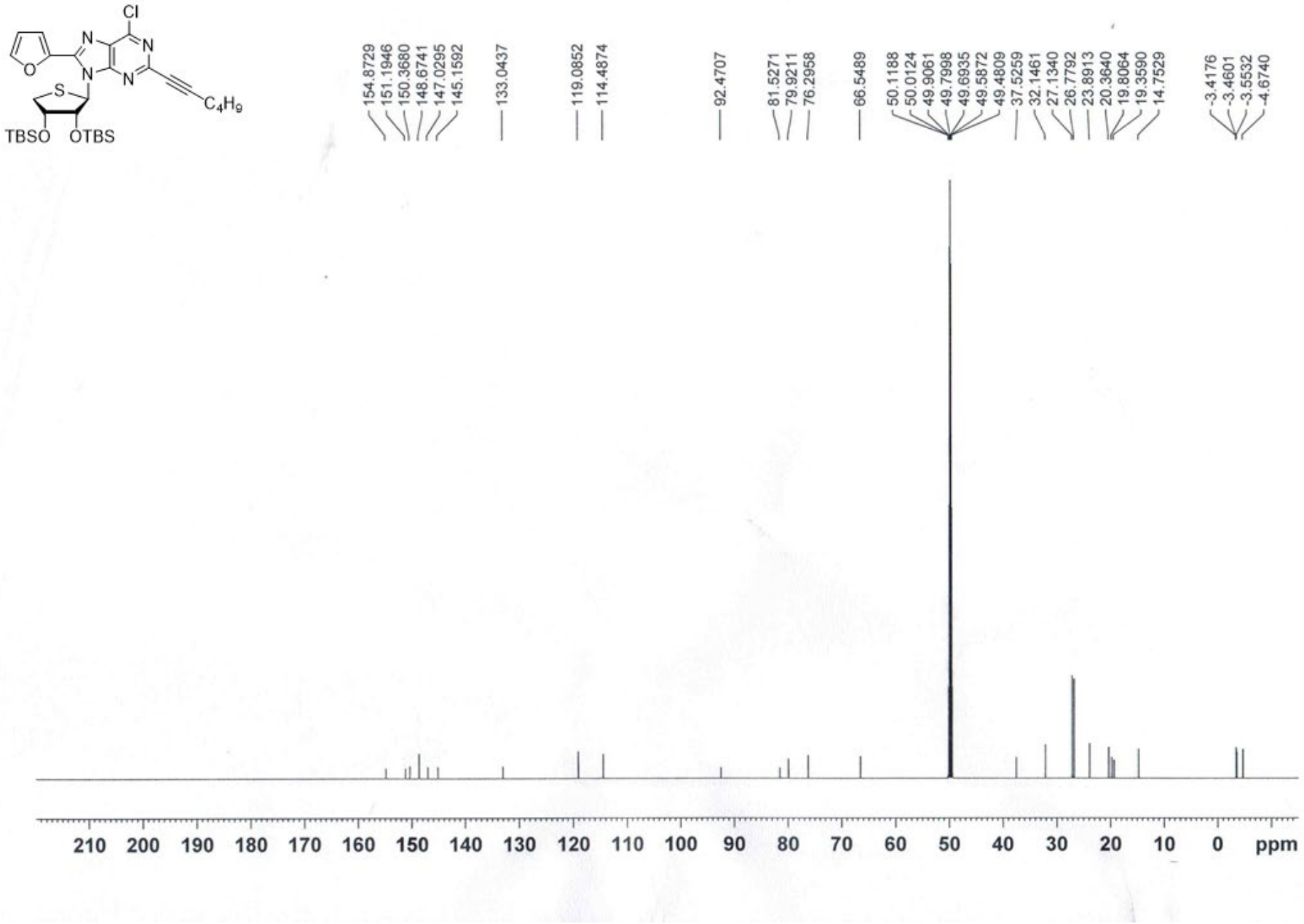

Cl
N
N
N
N
I
O
S
TBSO
OTBS
21a
abundance
X : parts per Million : Proton
9.79
9.14
6.22
2.91
2.96
24.66m
0.99
1.00
0.98
0.96
1.01
0.98
0.94
1.02

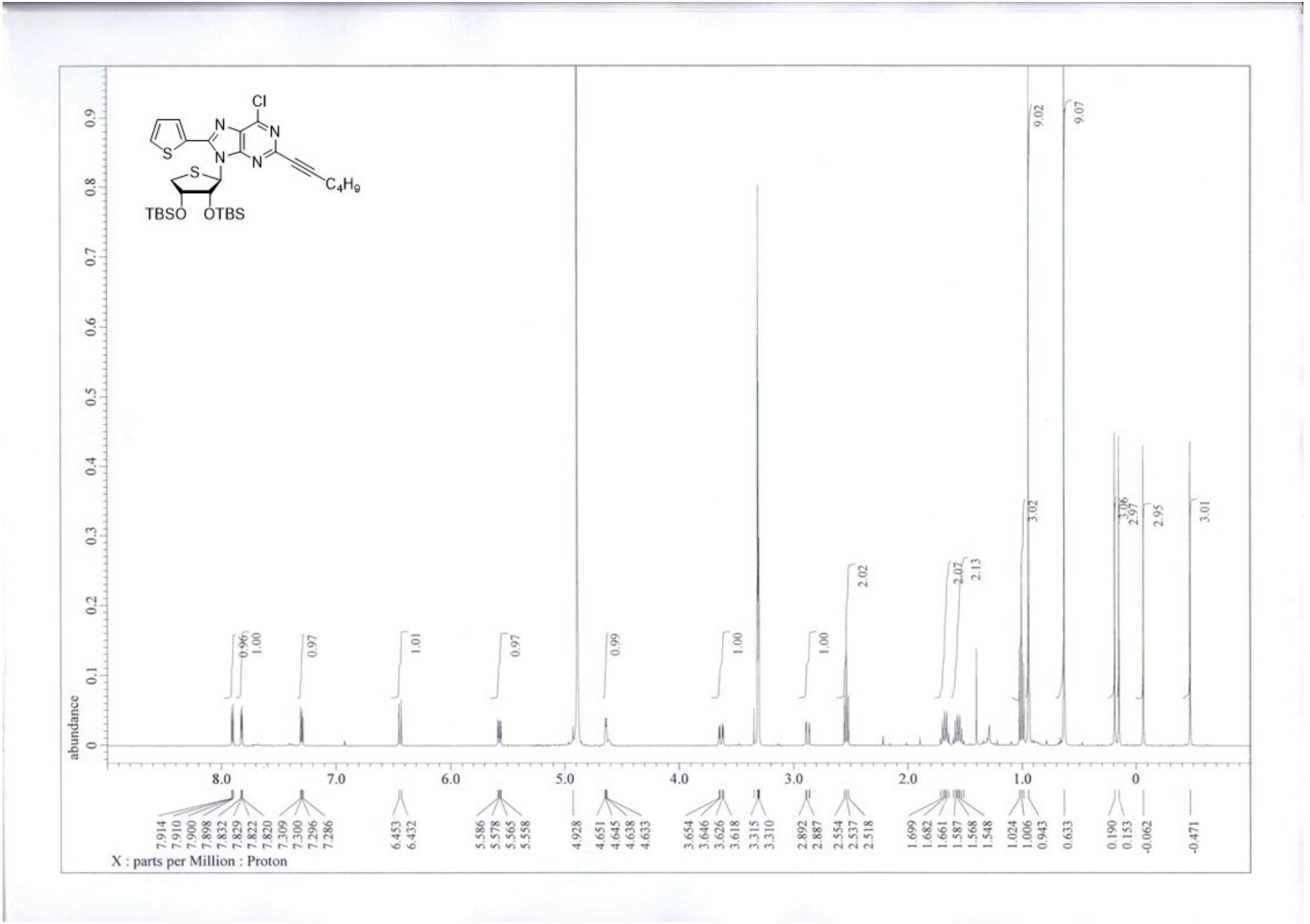

Cl
N
N
O
N
N
I
S
TBSO
OTBS
21a
155.3815
150.8029
149.6965
148.7606
144.9634
134.0309
119.3217
117.1567
114.5275
79.9948
76.2115
66.5284
50.1169
50.0104
49.9040
49.7975
49.6910
49.5845
49.4780
37.6038
27.1184
26.7648
19.7995
19.3505
-3.3704
-3.4302
-3.5460
-4.6172
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

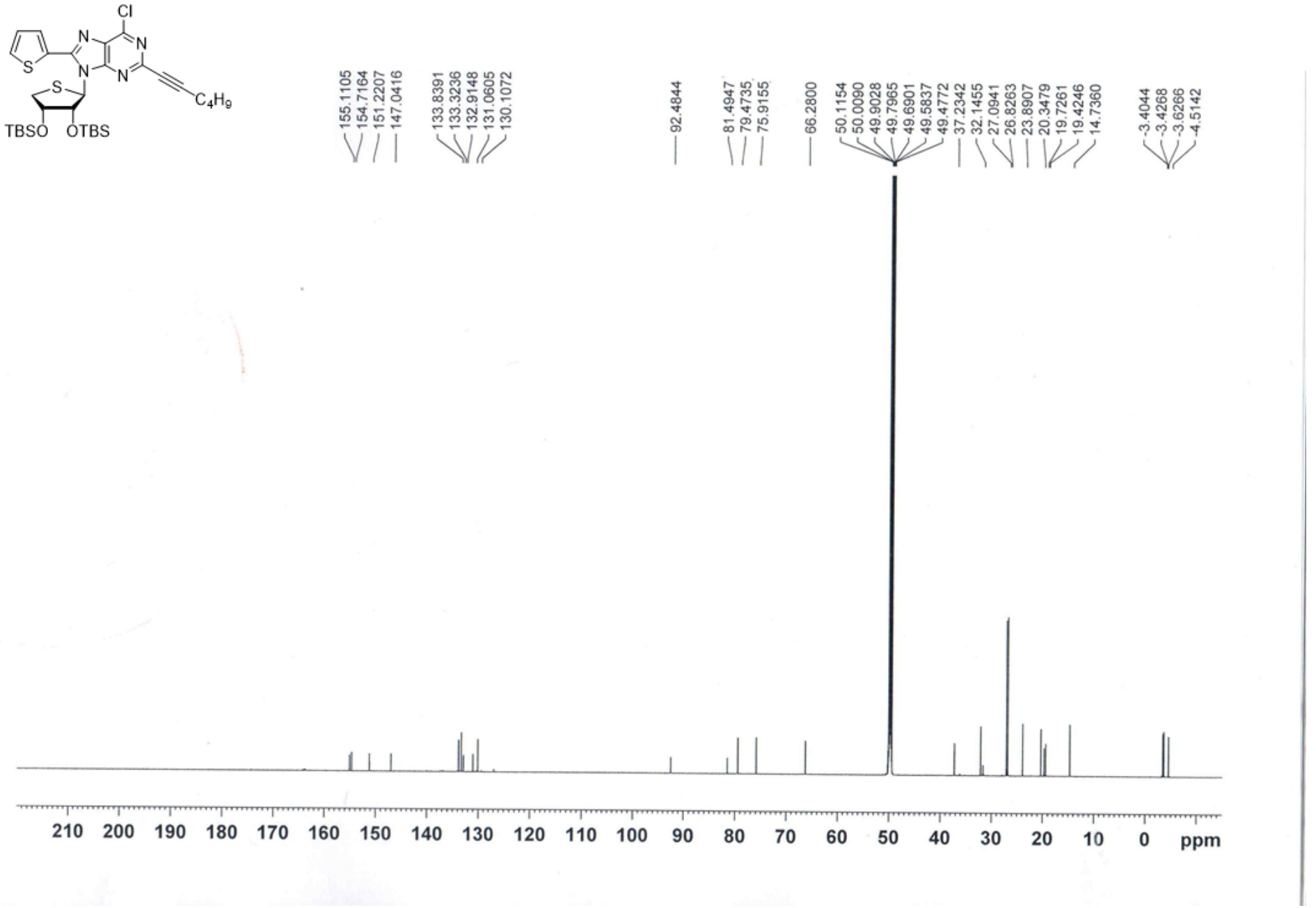

7.889
7.886
7.876
7.874
7.793
7.791
7.784
7.782
7.277
7.268
7.264
7.255
6.410
6.389
5.514
5.507
5.494
5.487
4.856
4.573
4.568
3.594
3.587
3.565
3.558
3.287
3.283
2.856
2.851
2.827
2.823
1.263
0.911
0.874
0.614
0.608
0.159
0.125
-0.082
-0.468
Cl
N
N
N
N
S
S
I
TBSO
OTBS
22b
abundance
4.0
3.0
2.0
1.0
0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
1.00
1.03
1.00
0.99
1.02
1.06
1.02
1.05
11.96
9.15
3.18
3.07
3.18
3.15
X : parts per Million : Proton

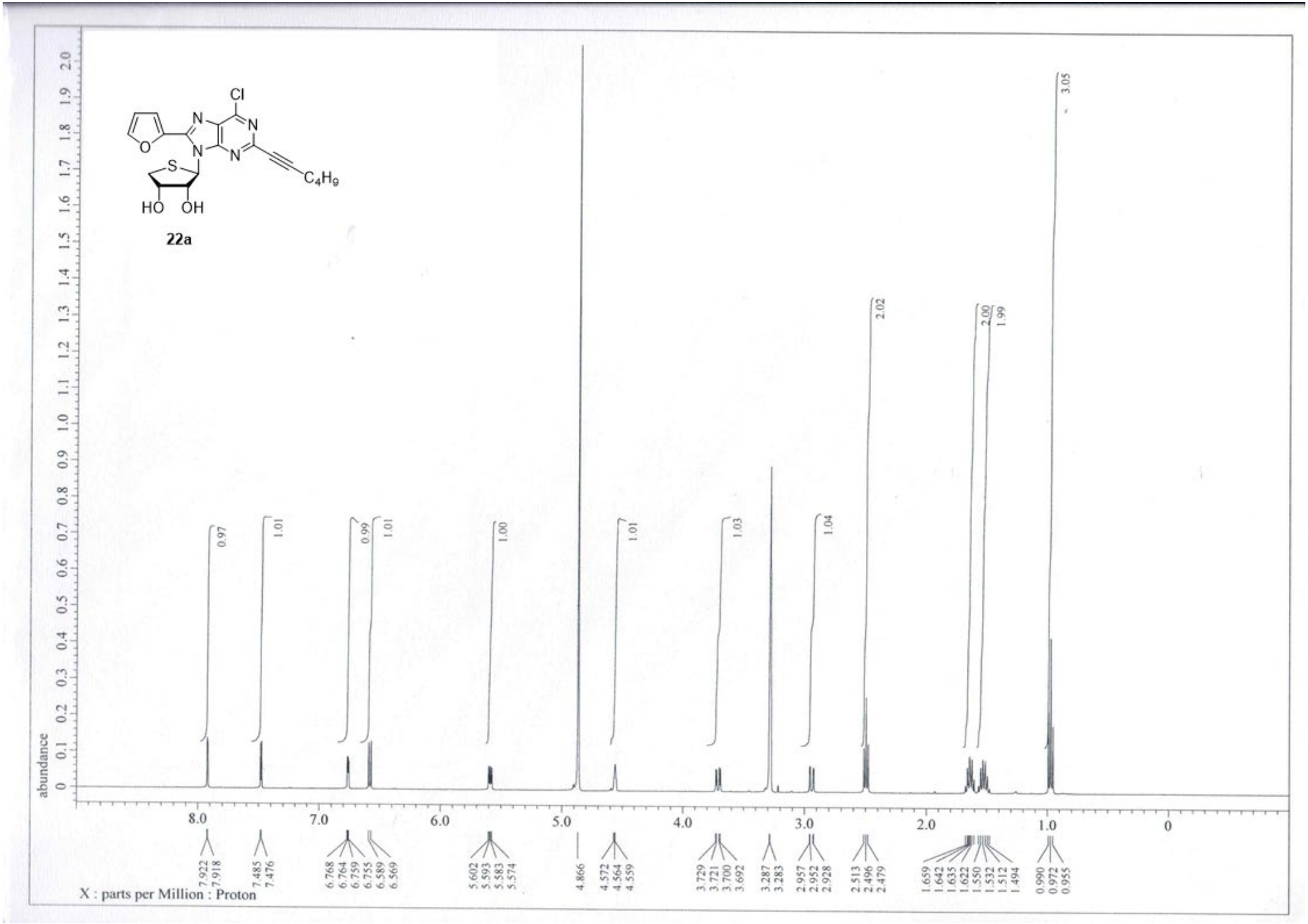

Cl
N
N
S
N
N
I
S
TBSO
OTBS
22b
(thousandths)
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
153.659
151.627
149.673
132.366
131.485
131.331
129.328
128.284
115.194
77.735
77.476
77.371
77.160
76.844
73.979
64.290
53.577
36.031
29.515
25.835
25.682
18.169
17.929
-4.197
-4.360
-4.466
-5.137
X : parts per Million : Carbon13

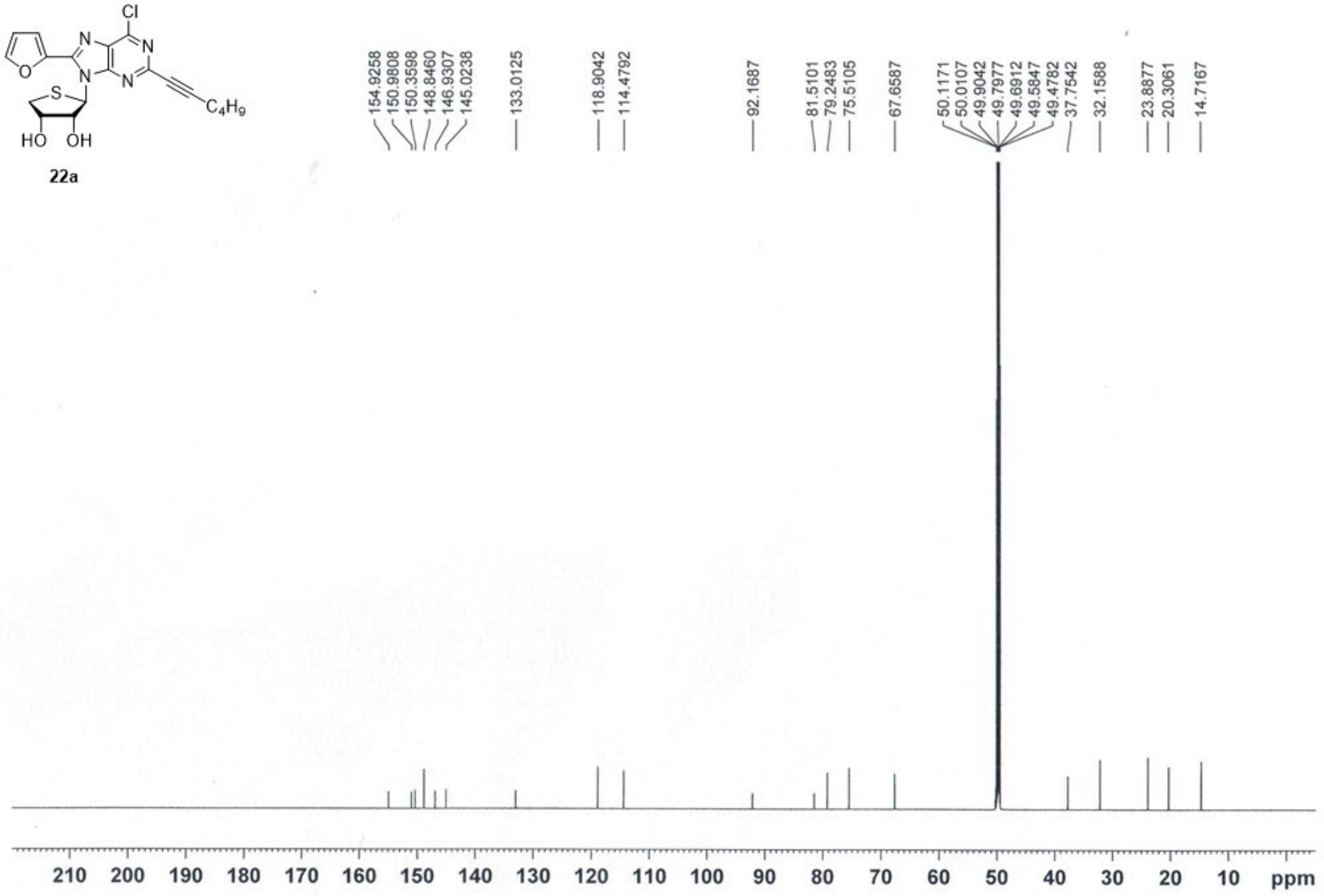

Cl
N
N
N
N
O
S
C_4H_9
TBSO
OTBS
abundance
X : parts per Million : Proton
12.31
8.96
6.09
3.02
3.00
2.02
2.00
1.00

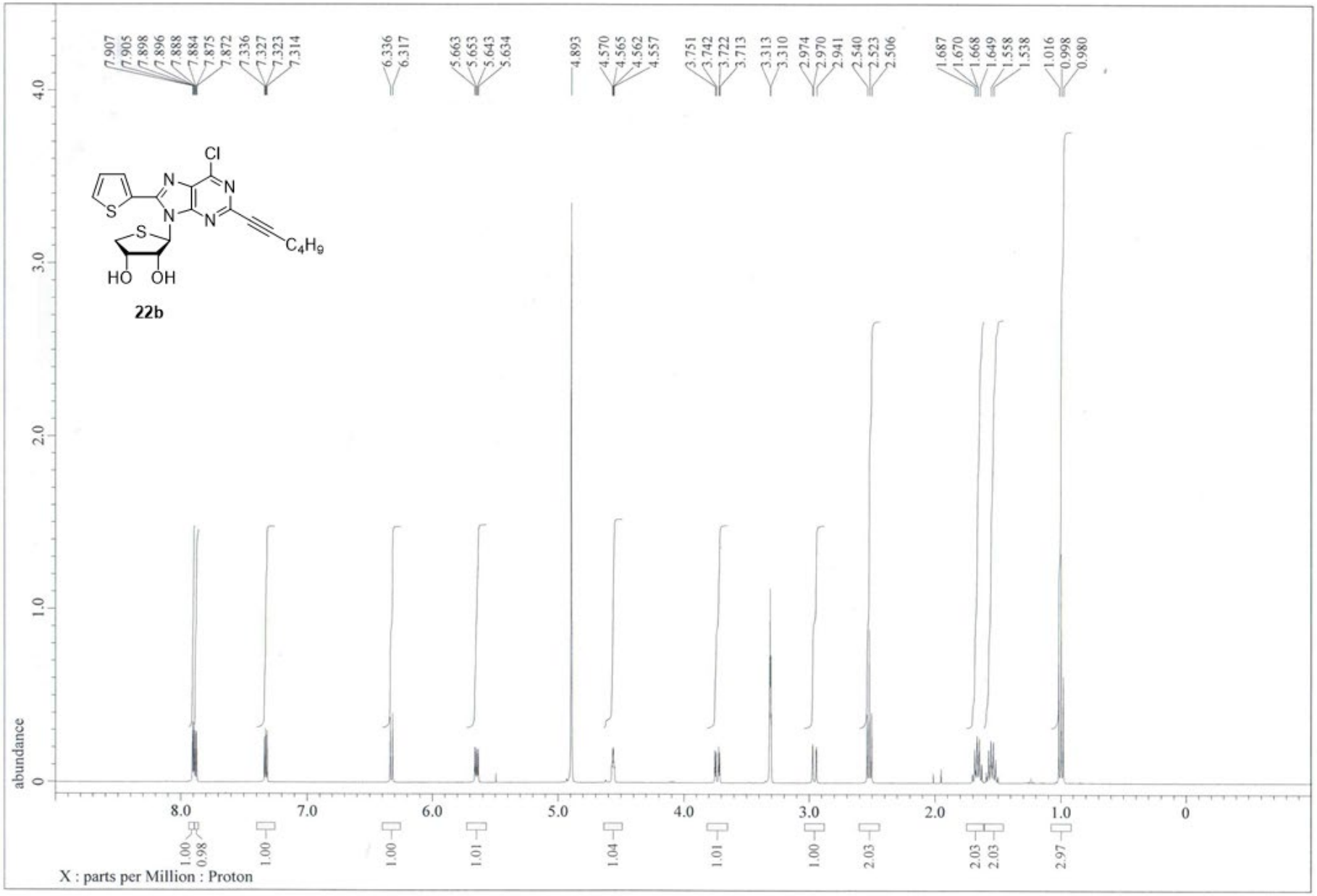

Cl
N
N
N
N
O
S
C4H9
TBSO
OTBS
154.8729
151.1946
150.3680
148.6741
147.0295
145.1592
133.0437
119.0852
114.4874
92.4707
81.5271
79.9211
76.2958
66.5489
50.1188
50.0124
49.9061
49.7998
49.6935
49.5872
49.4809
37.5259
32.1461
27.1340
26.7792
23.8913
20.3640
19.8064
19.3590
14.7529
-3.4176
-3.4601
-3.5532
-4.6740
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

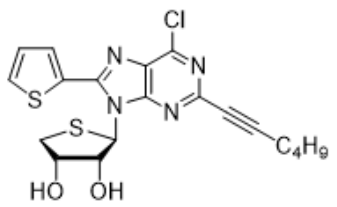

Cl
N
N
N
N
S
S
C_4H_9
TBSO
OTBS
abundance
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
0.96
1.00
0.97
1.01
0.97
0.99
1.00
1.00
2.02
2.07
2.13
3.02
9.02
9.07
3.06
2.97
2.95
3.01
7.914
7.910
7.900
7.898
7.832
7.829
7.822
7.820
7.309
7.300
7.296
7.286
6.453
6.432
5.586
5.578
5.565
5.558
4.928
4.651
4.645
4.638
4.633
3.654
3.646
3.626
3.618
3.315
3.310
2.892
2.887
2.554
2.537
2.518
1.699
1.682
1.661
1.587
1.568
1.548
1.024
1.006
0.943
0.633
0.190
0.153
-0.062
-0.471
X : parts per Million : Proton


Cl
N
N
N
N
S
S
C_4H_9
TBSO
OTBS
155.1105
154.7164
151.2207
147.0416
133.8391
133.3236
132.9148
131.0605
130.1072
92.4844
81.4947
79.4735
75.9155
66.2800
50.1154
50.0090
49.9028
49.7965
49.6901
49.5837
49.4772
37.2342
32.1455
27.0941
26.8263
23.8907
20.3479
19.7261
19.4246
14.7360
-3.4044
-3.4268
-3.6266
-4.5142
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


Cl
N
N
N
N
O
S
HO
OH
C_4H_9
22a
abundance
X : parts per Million : Proton
0.97
1.01
0.99
1.01
1.00
1.01
1.03
1.04
2.02
2.00
1.99
3.05
7.922
7.918
7.485
7.476
6.768
6.764
6.759
6.755
6.589
6.569
5.602
5.593
5.583
5.574
4.866
4.572
4.564
4.559
3.729
3.721
3.700
3.692
3.287
3.283
2.957
2.952
2.928
2.513
2.496
2.479
1.659
1.642
1.635
1.622
1.550
1.532
1.512
1.494
0.990
0.972
0.955

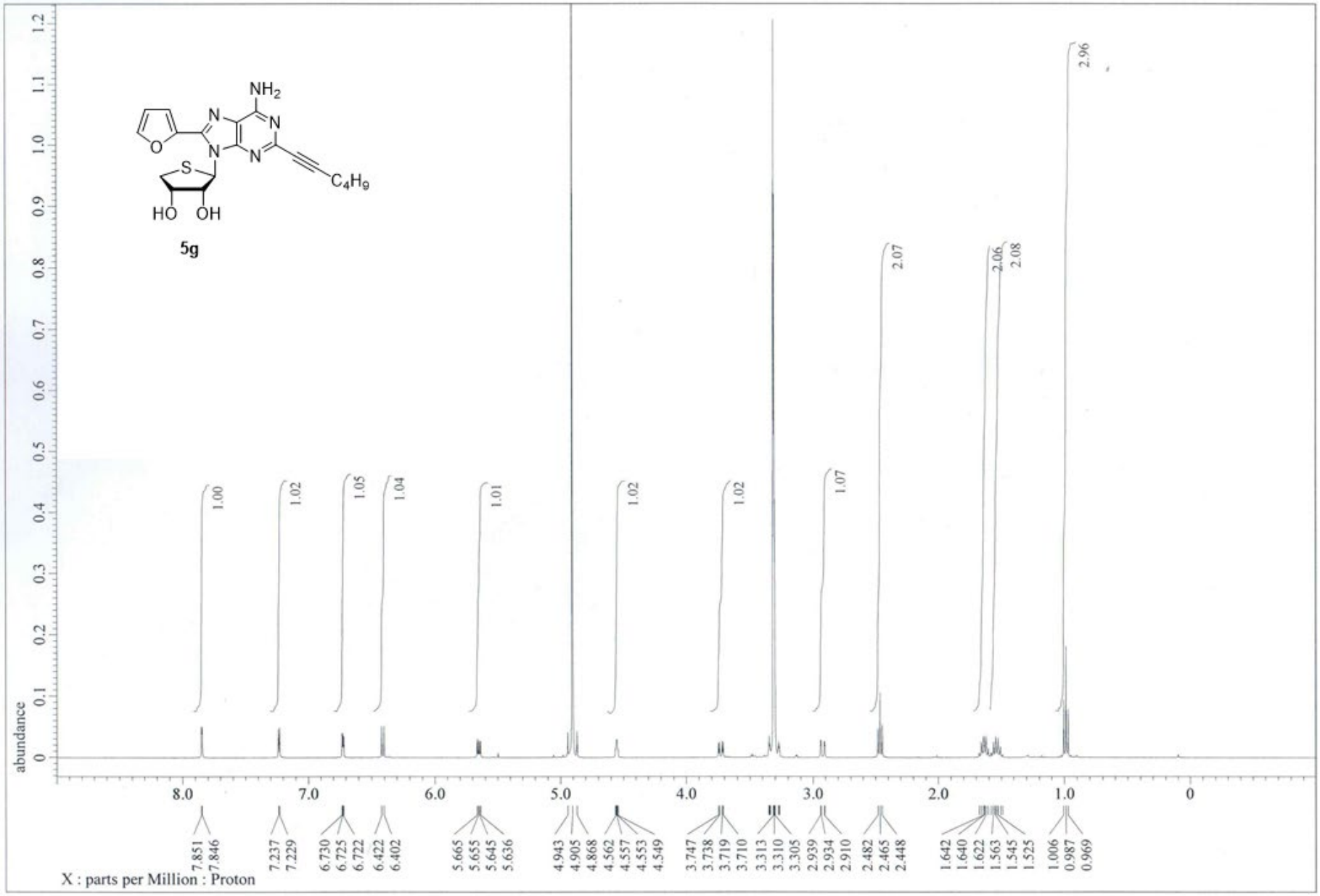

Cl
N
N
N
N
O
S
HO
OH
C_4H_9
22a
154.9258
150.9808
150.3598
148.8460
146.9307
145.0238
133.0125
118.9042
114.4792
92.1687
81.5101
79.2483
75.5105
67.6587
50.1171
50.0107
49.9042
49.7977
49.6912
49.5847
49.4782
37.7542
32.1588
23.8877
20.3061
14.7167
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

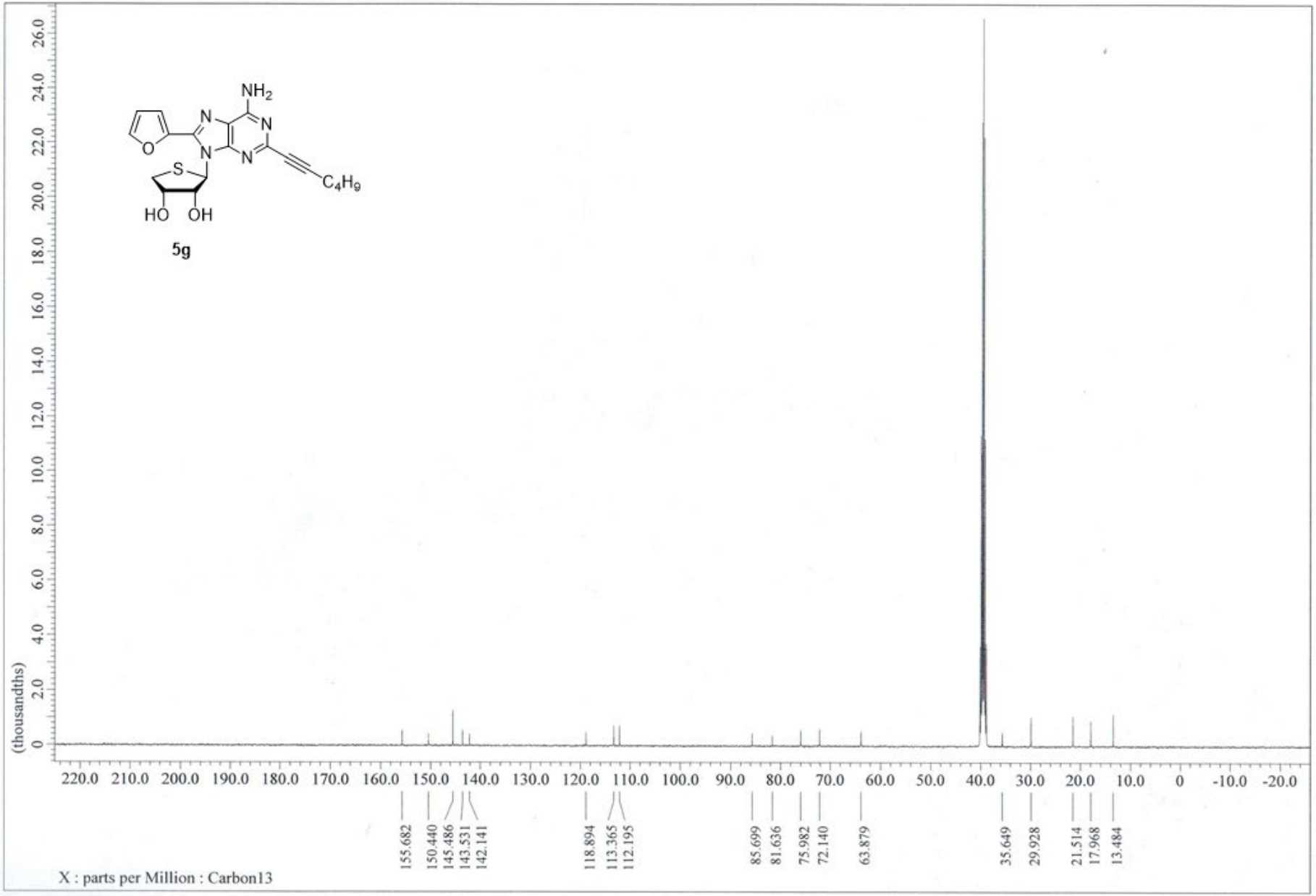

7.907
7.905
7.898
7.896
7.888
7.884
7.875
7.872
7.336
7.327
7.323
7.314
6.336
6.317
5.663
5.653
5.643
5.634
4.893
4.570
4.565
4.562
4.557
3.751
3.742
3.722
3.713
3.313
3.310
2.974
2.970
2.941
2.540
2.523
2.506
1.687
1.670
1.668
1.649
1.558
1.538
1.016
0.998
0.980
Cl
N
N
N
N
S
S
HO
OH
C_4H_9
22b
abundance
4.0
3.0
2.0
1.0
0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
1.00
0.98
1.00
1.00
1.01
1.04
1.01
1.00
2.03
2.03
2.03
2.97
X : parts per Million : Proton

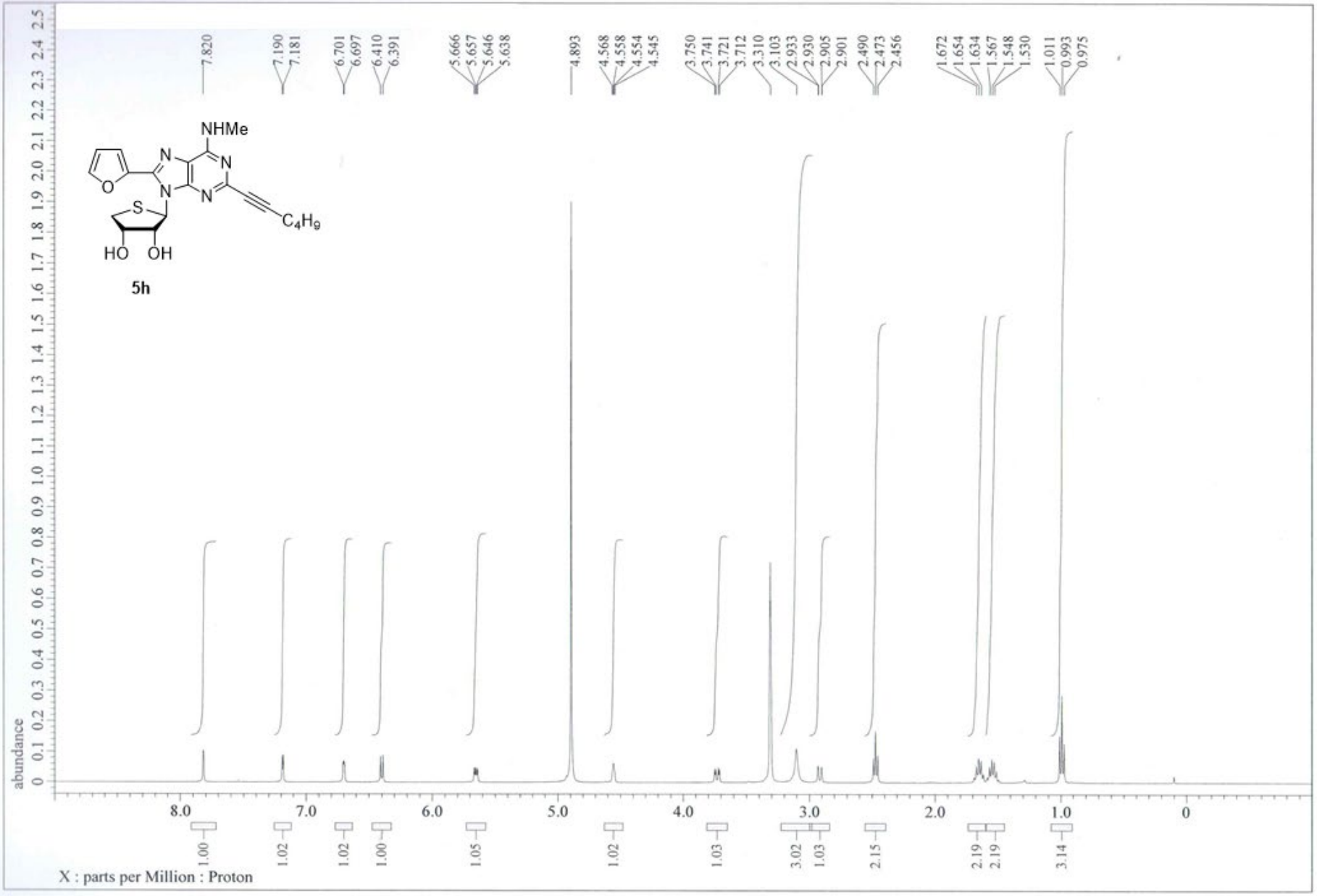

Cl
N
N
N
S
S
C_4H_9
HO
OH

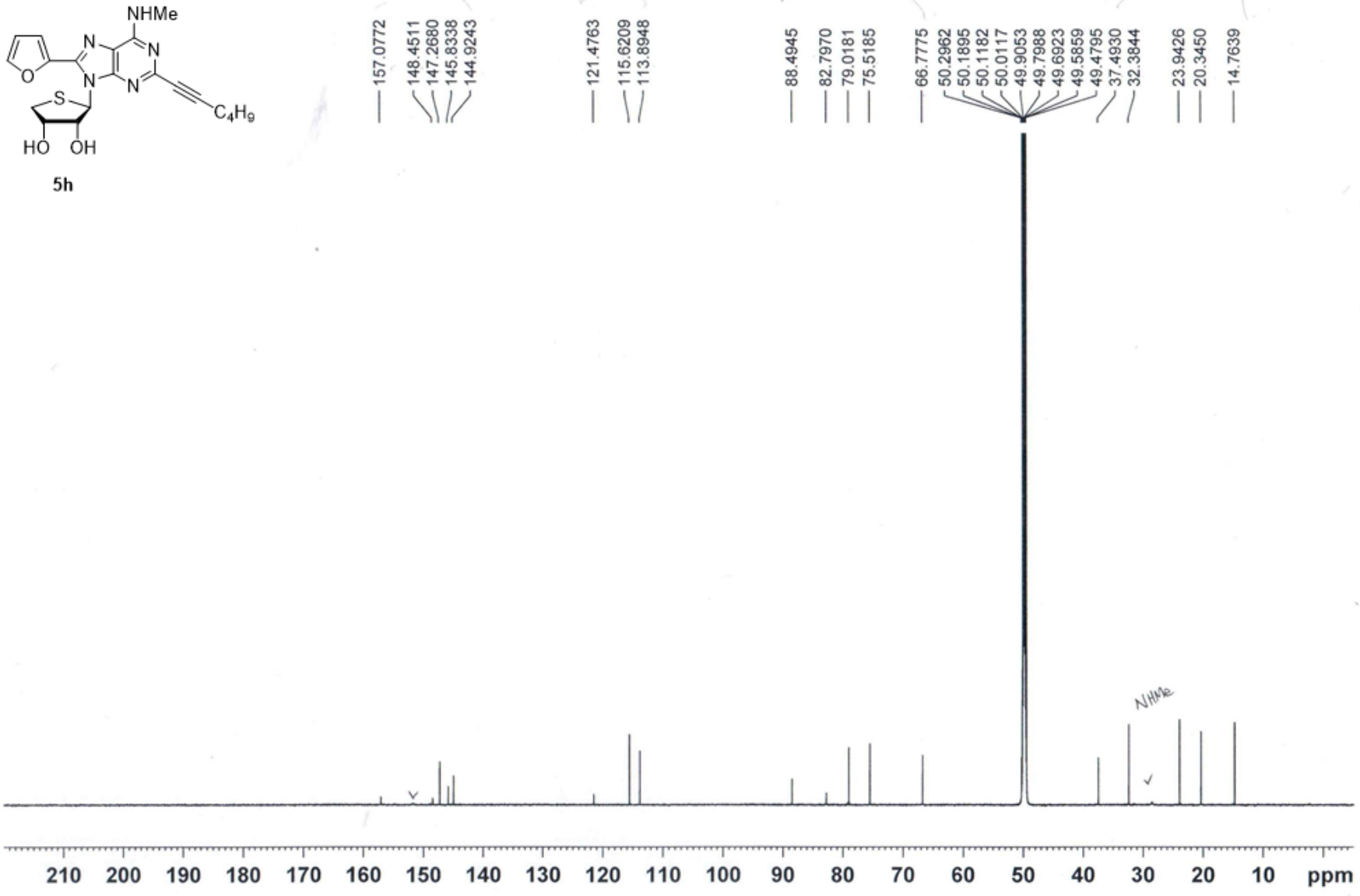
22b

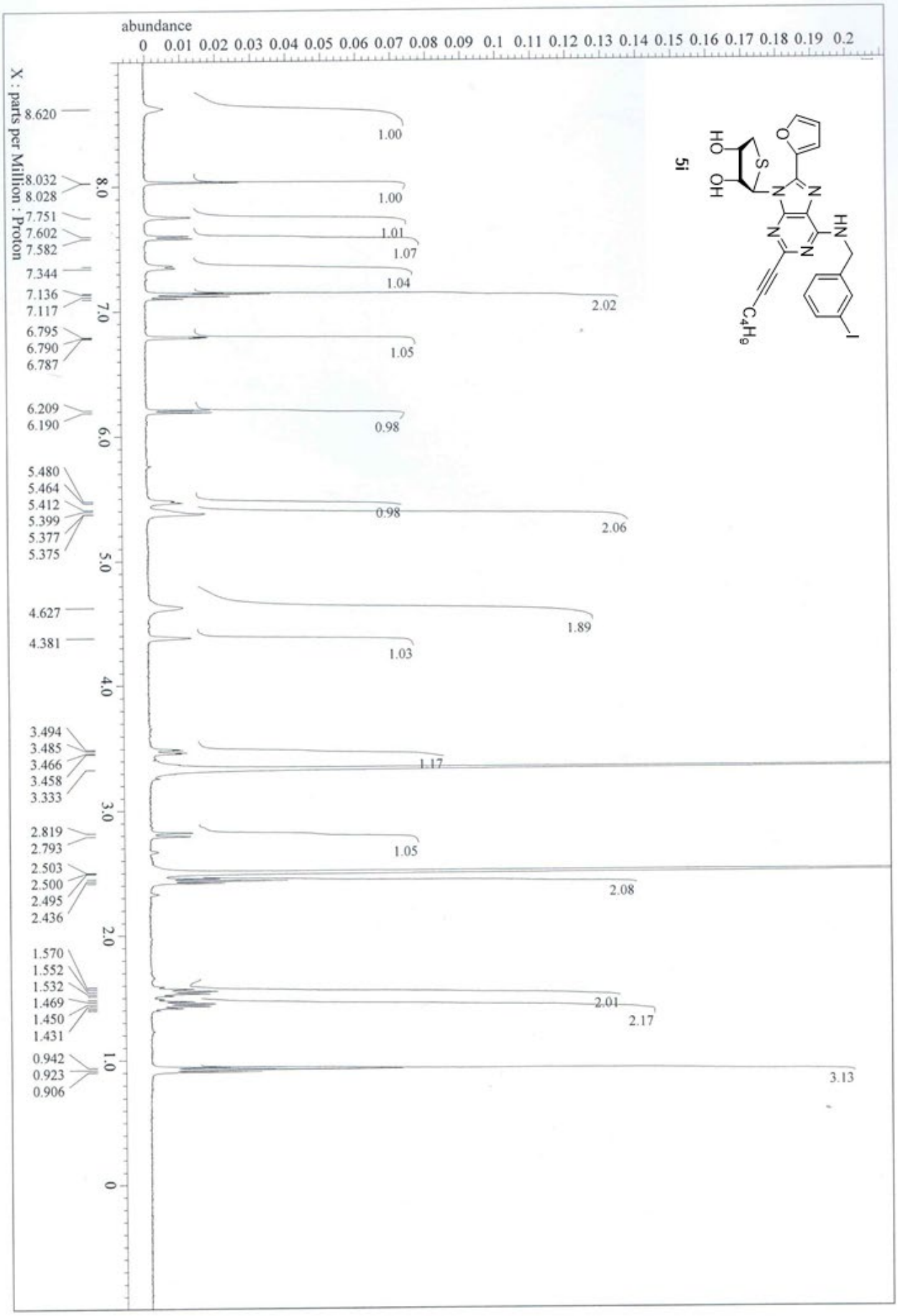
155.2142
154.5775
150.9496
146.8798
133.6566
133.4472
132.9050
131.2441
130.3466
92.1183
81.5243
78.8956
75.3056
67.5129
50.2951
50.1886
50.1169
50.0105
49.9040
49.7976
49.6911
49.5845
49.4780
37.6214
32.1587
23.8926
20.3157
14.7224
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

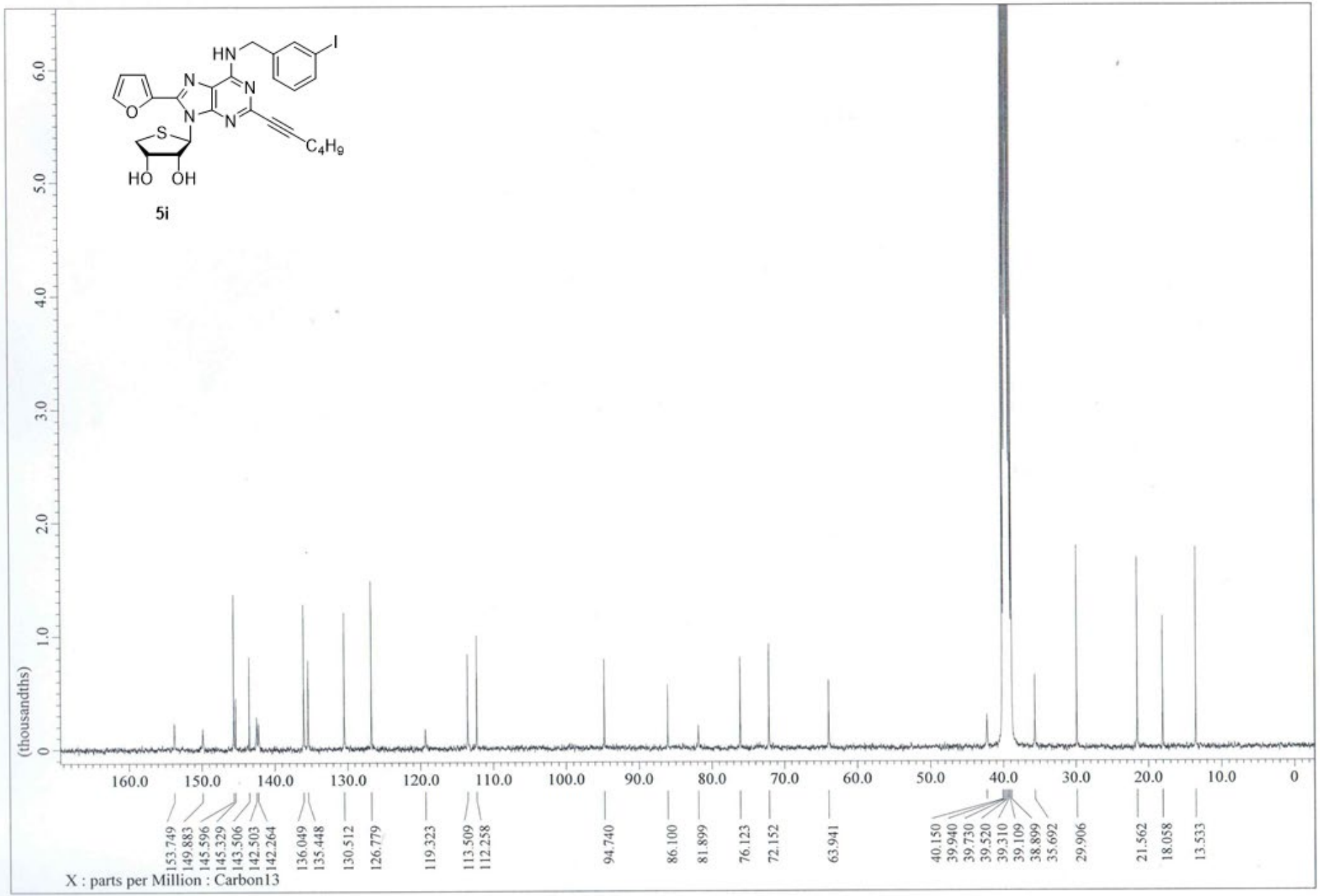

NH2
N
N
N
N
O
S
C4H9
HO
OH
5g
abundance
X : parts per Million : Proton
1.00
1.02
1.05
1.04
1.01
1.02
1.02
1.07
2.07
2.06
2.08
2.96
7.851
7.846
7.237
7.229
6.730
6.725
6.722
6.422
6.402
5.665
5.655
5.645
5.636
4.943
4.905
4.868
4.562
4.557
4.553
4.549
3.747
3.738
3.719
3.710
3.313
3.310
3.305
2.939
2.934
2.910
2.482
2.465
2.448
1.642
1.640
1.622
1.563
1.545
1.525
1.006
0.987
0.969

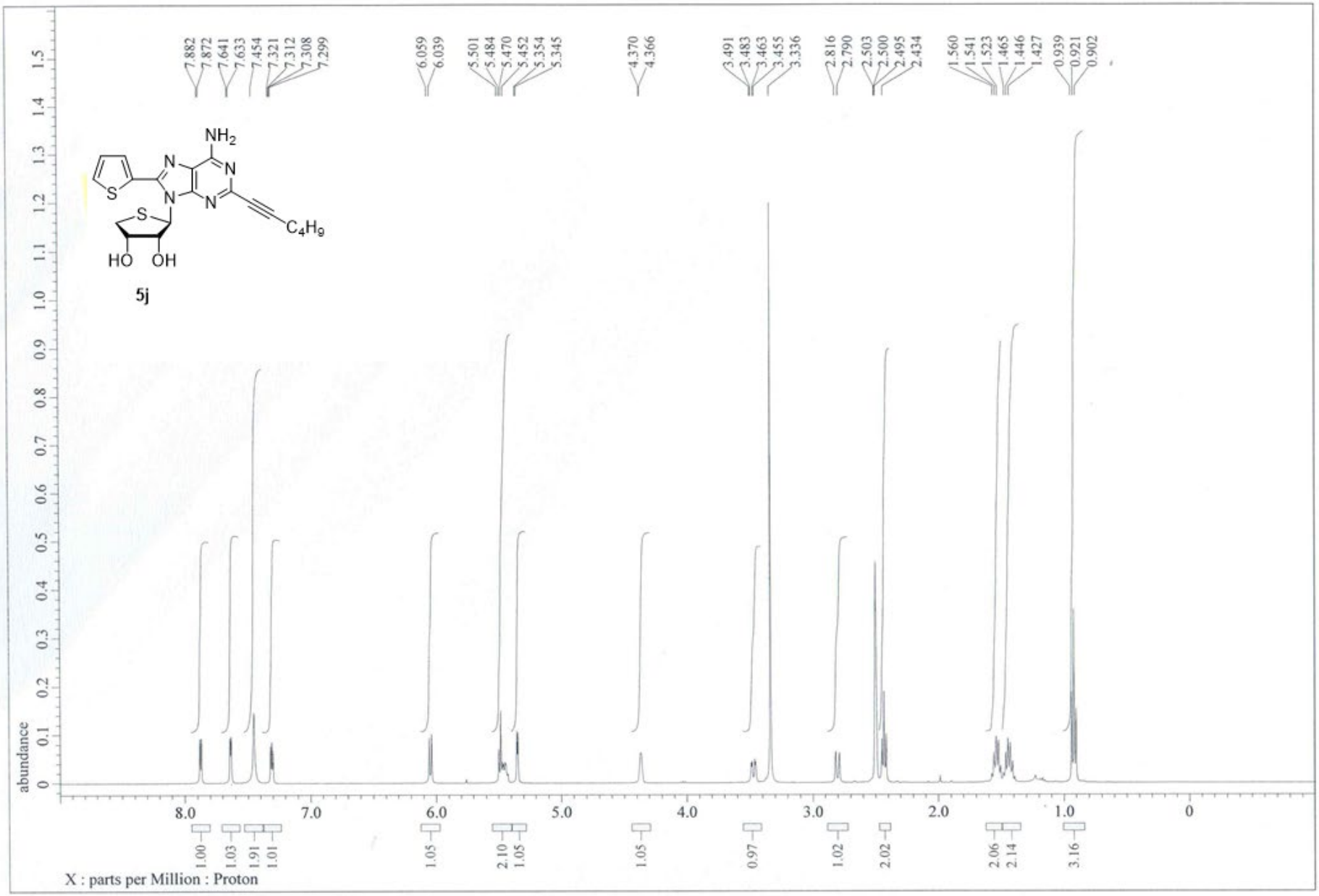

NH2
N
N
N
N
O
S
HO
OH
C4H9
5g
(thousandths)
26.0
24.0
22.0
20.0
18.0
16.0
14.0
12.0
10.0
8.0
6.0
4.0
2.0
0
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
155.682
150.440
145.486
143.531
142.141
118.894
113.365
112.195
85.699
81.636
75.982
72.140
63.879
35.649
29.928
21.514
17.968
13.484
X : parts per Million : Carbon13

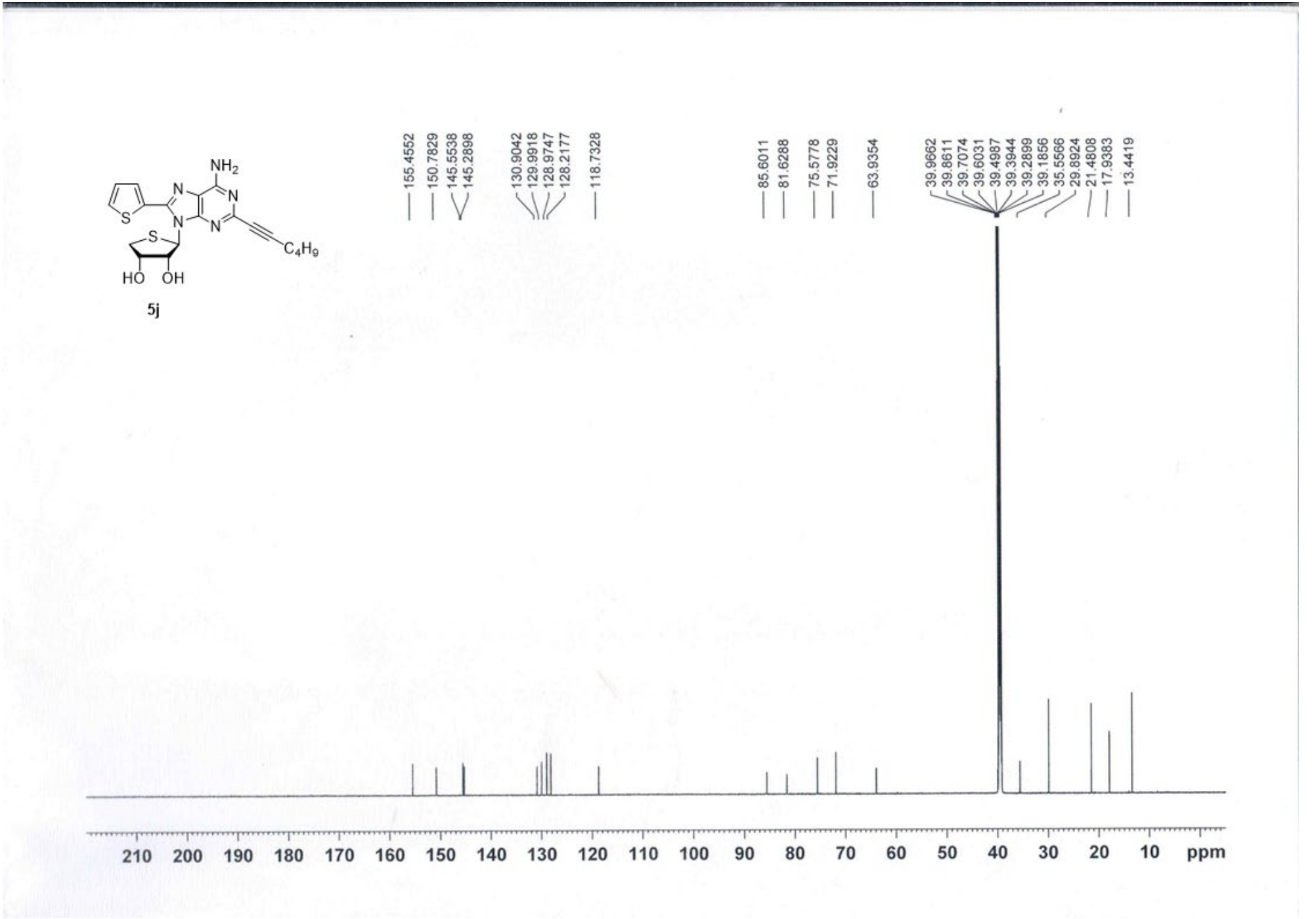

7.820
7.190
7.181
6.701
6.697
6.410
6.391
5.666
5.657
5.646
5.638
4.893
4.568
4.558
4.554
4.545
3.750
3.741
3.721
3.712
3.310
3.103
2.933
2.930
2.905
2.901
2.490
2.473
2.456
1.672
1.654
1.634
1.567
1.548
1.530
1.011
0.993
0.975
NHMe
N
N
N
N
O
S
HO
OH
C4H9
5h
abundance
X : parts per Million : Proton
1.00
1.02
1.02
1.00
1.05
1.02
1.03
3.02
1.03
2.15
2.19
2.19
3.14

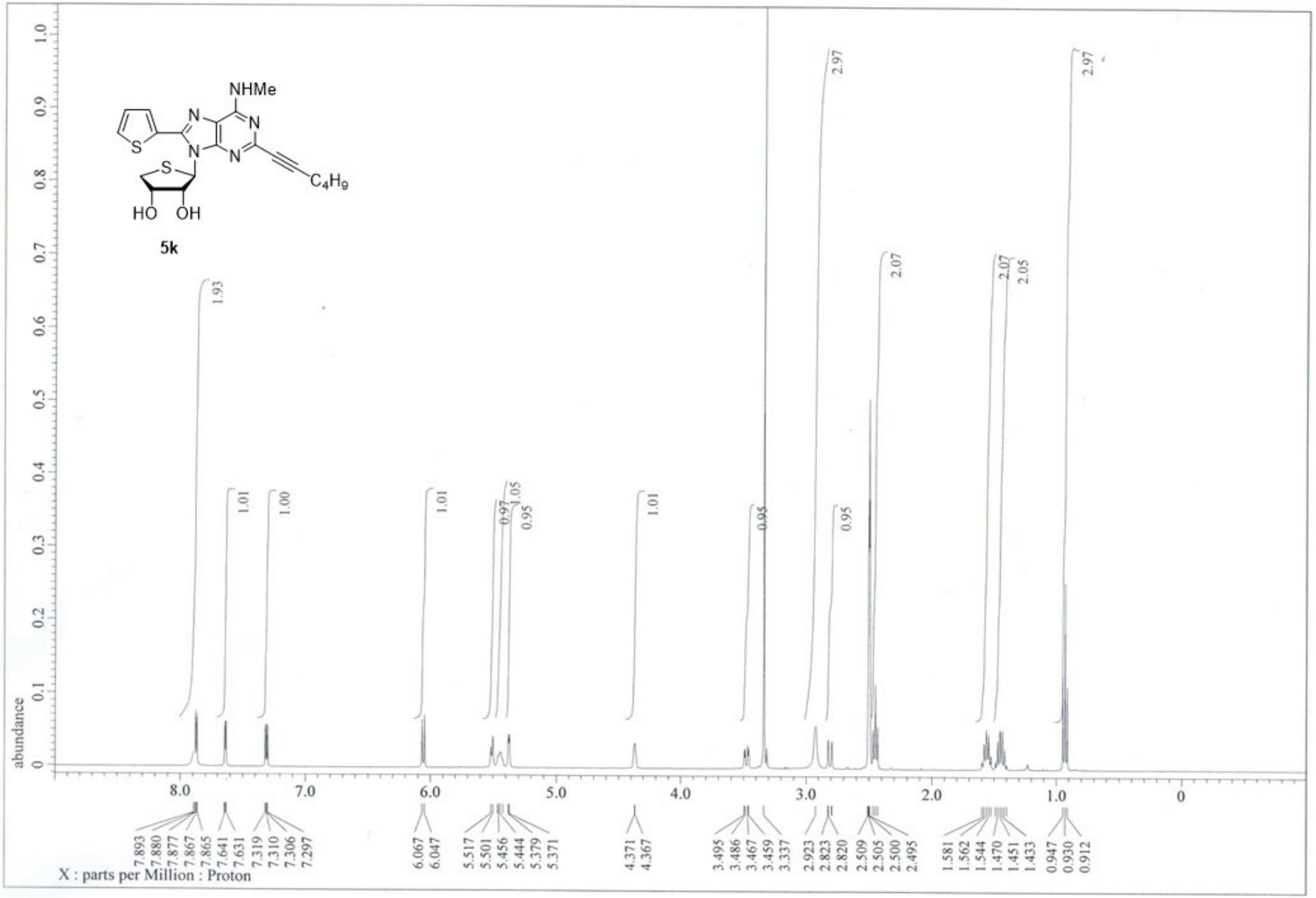

NHMe
N
N
N
N
O
S
HO
OH
C_4H_9
5h
157.0772
148.4511
147.2680
145.8338
144.9243
121.4763
115.6209
113.8948
88.4945
82.7970
79.0181
75.5185
66.7775
50.2962
50.1895
50.1182
50.0117
49.9053
49.7988
49.6923
49.5859
49.4795
37.4930
32.3844
23.9426
20.3450
14.7639
NHMe
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

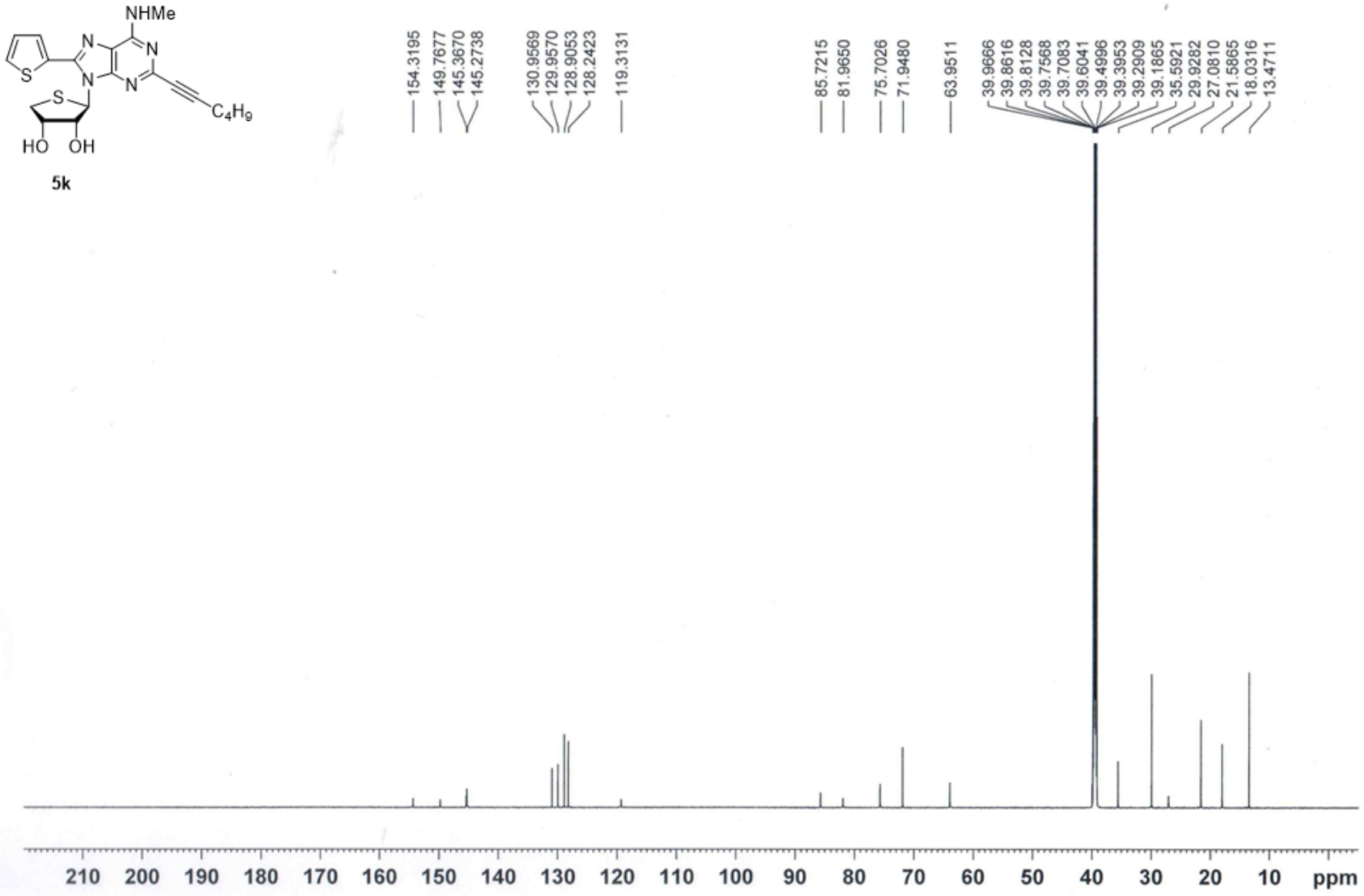

abundance
0 0.01 0.02 0.03 0.04 0.05 0.06 0.07 0.08 0.09 0.1 0.11 0.12 0.13 0.14 0.15 0.16 0.17 0.18 0.19 0.2
X : parts per Million : Proton
8.620
8.032
8.028
7.751
7.602
7.582
7.344
7.136
7.117
6.795
6.790
6.787
6.209
6.190
5.480
5.464
5.412
5.399
5.377
5.375
4.627
4.381
3.494
3.485
3.466
3.458
3.333
2.819
2.793
2.503
2.500
2.495
2.436
1.570
1.552
1.532
1.469
1.450
1.431
0.942
0.923
0.906
1.00
1.00
1.01
1.07
1.04
2.02
1.05
0.98
0.98
2.06
1.89
1.03
1.17
1.05
2.08
2.01
2.17
3.13
5i
HO
OH
S
O
N
HN
I
C4H9

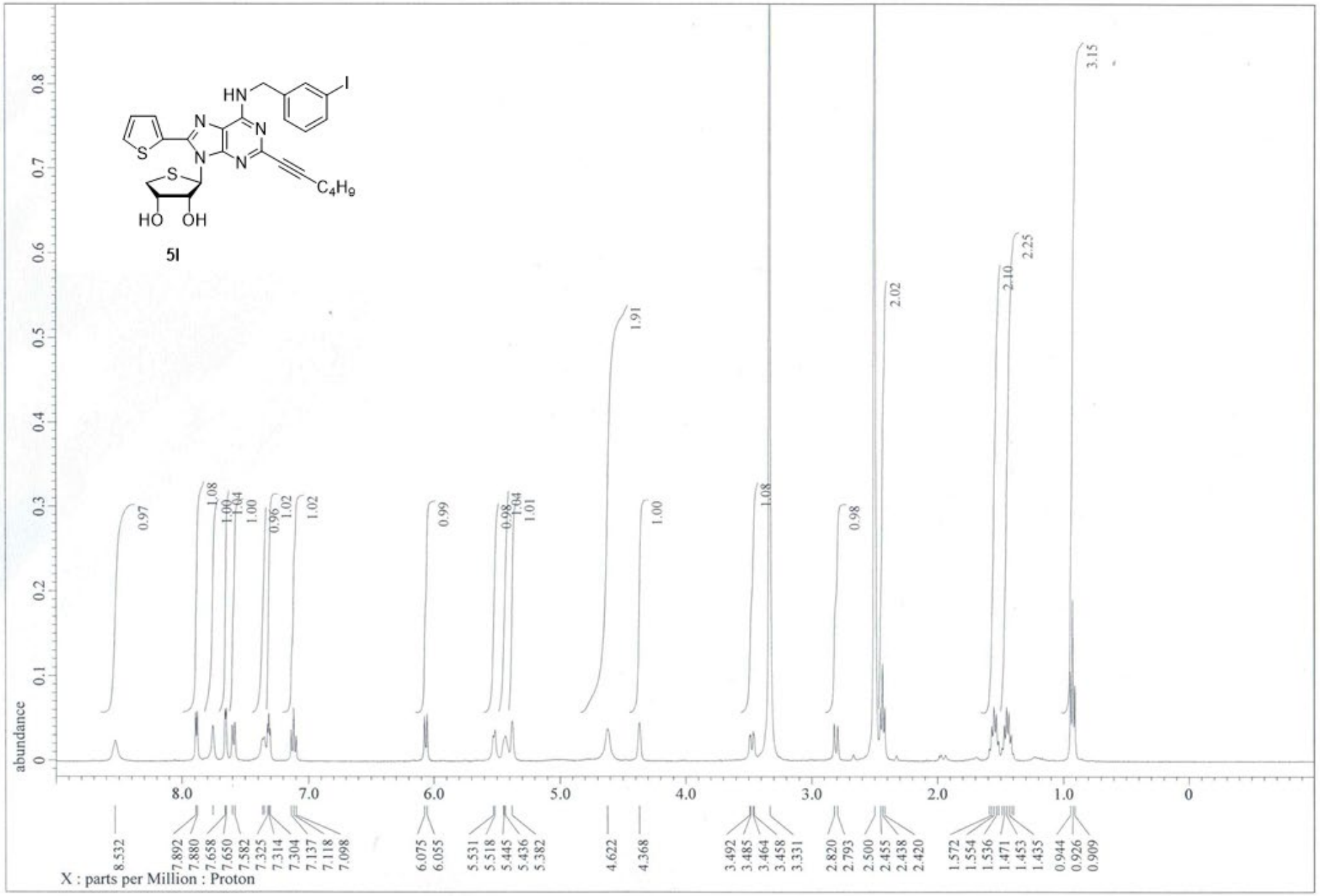

HN
I
N
N
N
N
O
S
C_4H_9
HO
OH
5i
(thousandths)
6.0
5.0
4.0
3.0
2.0
1.0
0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
153.749
149.883
145.596
145.329
143.506
142.503
142.264
136.049
135.448
130.512
126.779
119.323
113.509
112.258
94.740
86.100
81.899
76.123
72.152
63.941
40.150
39.940
39.730
39.520
39.310
39.109
38.899
35.692
29.906
21.562
18.058
13.533
X : parts per Million : Carbon13

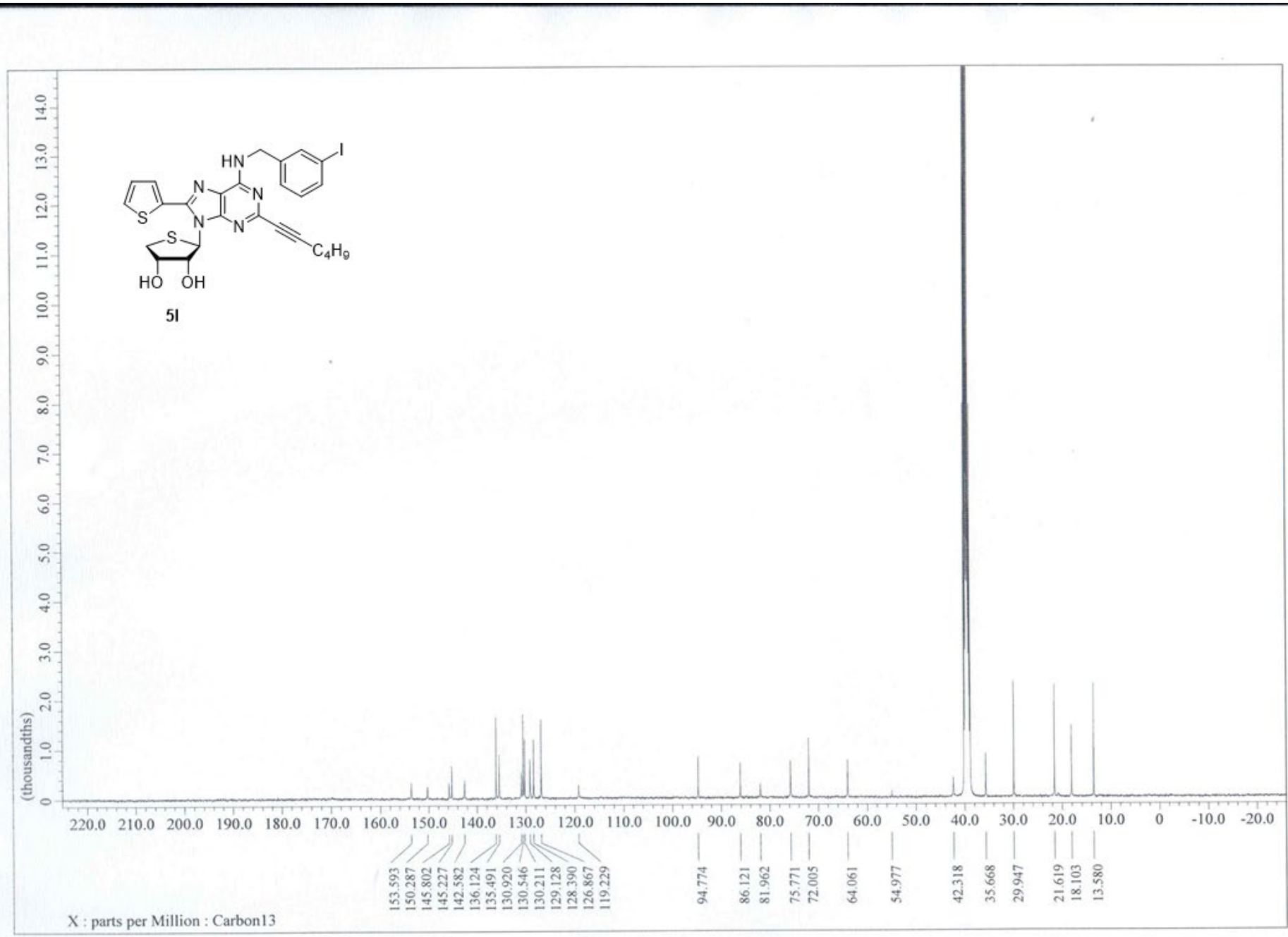

7.882
7.872
7.641
7.633
7.454
7.321
7.312
7.308
7.299
6.059
6.039
5.501
5.484
5.470
5.452
5.354
5.345
4.370
4.366
3.491
3.483
3.463
3.455
3.336
2.816
2.790
2.503
2.500
2.495
2.434
1.560
1.541
1.523
1.465
1.446
1.427
0.939
0.921
0.902
NH2
N
N
N
N
S
S
C4H9
HO
OH
5j
abundance
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
1.00
1.03
1.91
1.01
1.05
2.10
1.05
1.05
0.97
1.02
2.02
2.06
2.14
3.16
X : parts per Million : Proton

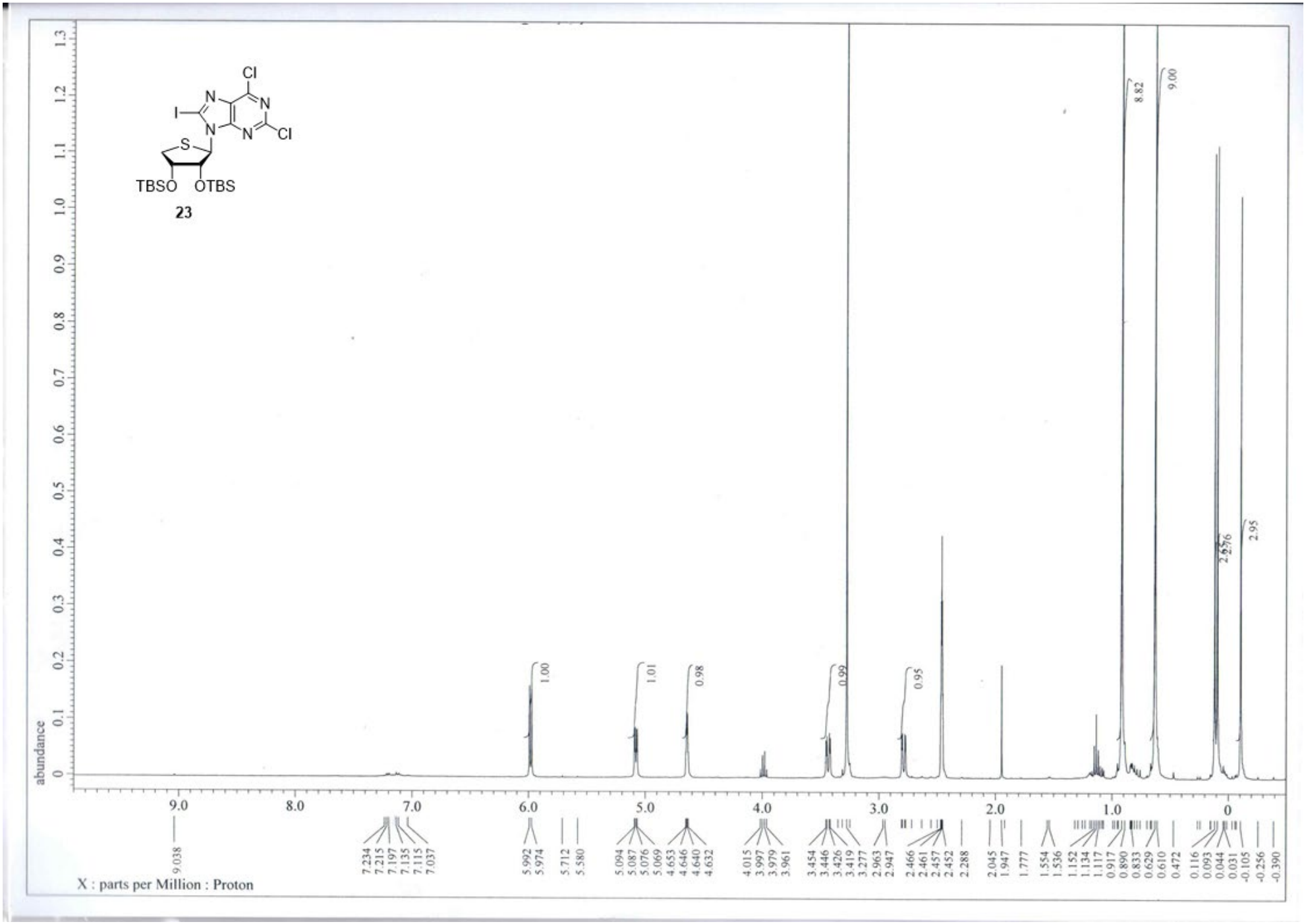

NH2
N
N
N
N
S
S
C4H9
HO
OH
5j
155.4552
150.7829
145.5538
145.2898
130.9042
129.9918
128.9747
128.2177
118.7328
85.6011
81.6288
75.5778
71.9229
63.9354
39.9662
39.8611
39.7074
39.6031
39.4987
39.3944
39.2899
39.1856
35.5566
29.8924
21.4808
17.9383
13.4419
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

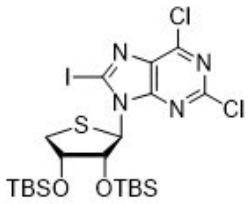

NHMe
N
N
N
N
S
S
C4H9
HO
OH
5k
abundance
X : parts per Million : Proton
1.93
1.01
1.00
1.01
0.97
1.05
0.95
1.01
0.95
0.95
2.97
2.07
2.07
2.05
2.97
2.97
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.893
7.880
7.877
7.867
7.865
7.641
7.631
7.319
7.310
7.306
7.297
6.067
6.047
5.517
5.501
5.456
5.444
5.379
5.371
4.371
4.367
3.495
3.486
3.467
3.459
3.337
2.923
2.823
2.820
2.509
2.505
2.500
2.495
1.581
1.562
1.544
1.470
1.451
1.433
0.947
0.930
0.912


NHMe
N
N
N
N
S
S
C_4H_9
HO
OH
5k
154.3195
149.7677
145.3670
145.2738
130.9569
129.9570
128.9053
128.2423
119.3131
85.7215
81.9650
75.7026
71.9480
63.9511
39.9666
39.8616
39.8128
39.7568
39.7083
39.6041
39.4996
39.3953
39.2909
39.1865
35.5921
29.9282
27.0810
21.5865
18.0316
13.4711
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

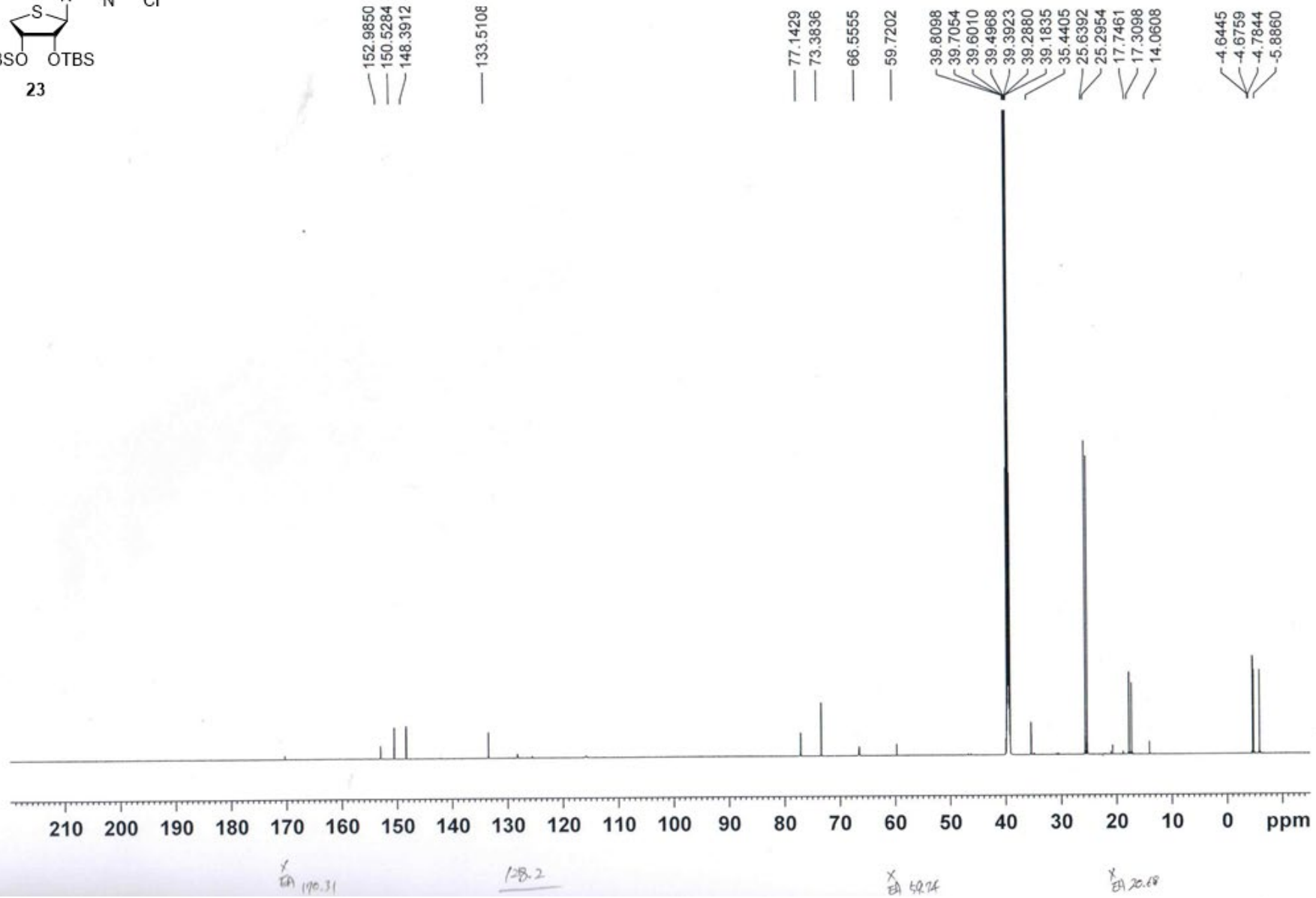

HN
I
N
N
N
N
S
S
C4H9
HO
OH
5l
abundance
X : parts per Million : Proton
8.532
7.892
7.880
7.658
7.650
7.582
7.325
7.314
7.304
7.137
7.118
7.098
6.075
6.055
5.531
5.518
5.445
5.436
5.382
4.622
4.368
3.492
3.485
3.464
3.458
3.331
2.820
2.793
2.500
2.455
2.438
2.420
1.572
1.554
1.536
1.471
1.453
1.435
0.944
0.926
0.909

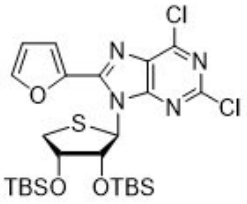

HN
I
N
N
N
N
S
S
C_4H_9
HO
OH
5l
(thousandths)
14.0
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
153.593
150.287
145.802
145.227
142.582
136.124
135.491
130.920
130.546
130.211
129.128
128.390
126.867
119.229
94.774
86.121
81.962
75.771
72.005
64.061
54.977
42.318
35.668
29.947
21.619
18.103
13.580
X : parts per Million : Carbon13


Cl
N
N
I
N
N
Cl
S
TBSO
OTBS
23
abundance
X : parts per Million : Proton
1.00
1.01
0.98
0.99
0.95
8.82
9.00
2.95
9.038
7.234
7.215
7.197
7.135
7.115
7.037
5.992
5.974
5.712
5.580
5.094
5.087
5.076
5.069
4.653
4.646
4.640
4.632
4.015
3.997
3.979
3.961
3.454
3.446
3.426
3.419
3.277
2.963
2.947
2.466
2.461
2.457
2.452
2.288
2.045
1.947
1.777
1.554
1.536
1.152
1.134
1.117
0.917
0.890
0.833
0.629
0.610
0.472
0.116
0.093
0.044
0.031
-0.105
-0.256
-0.390

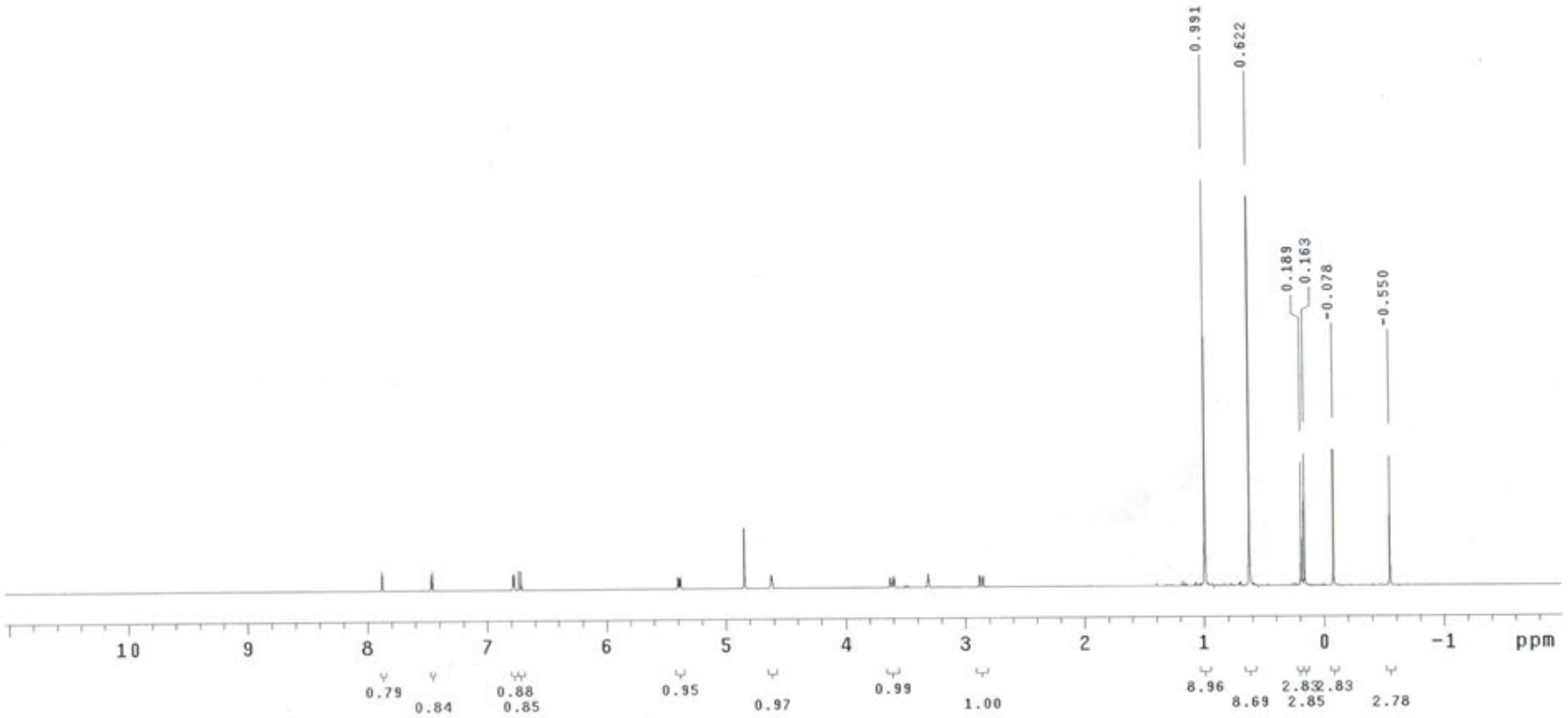

Cl
N
N
I
N
N
Cl
S
TBSO
OTBS

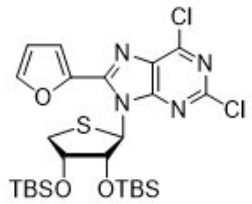
23


152.9850
150.5284
148.3912
133.5108
77.1429
73.3836
66.5555
59.7202
39.8098
39.7054
39.6010
39.4968
39.3923
39.2880
39.1835
35.4405
25.6392
25.2954
17.7461
17.3098
14.0608
-4.6445
-4.6759
-4.7844
-5.8860
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

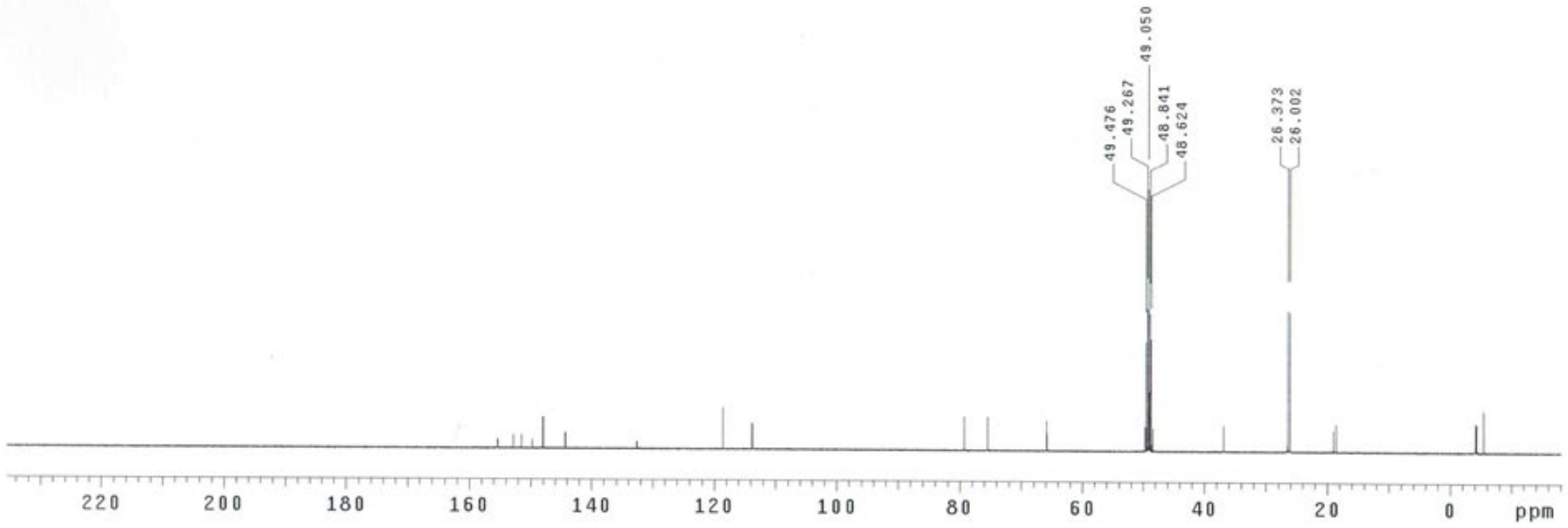

Cl
N
N
O
N
N
Cl
S
TBSO
OTBS

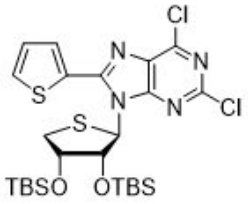
24a


0.991
0.622
0.189
0.163
-0.078
-0.550
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
0.79
0.84
0.88
0.85
0.95
0.97
0.99
1.00
8.96
8.69
2.83
2.85
2.83
2.78

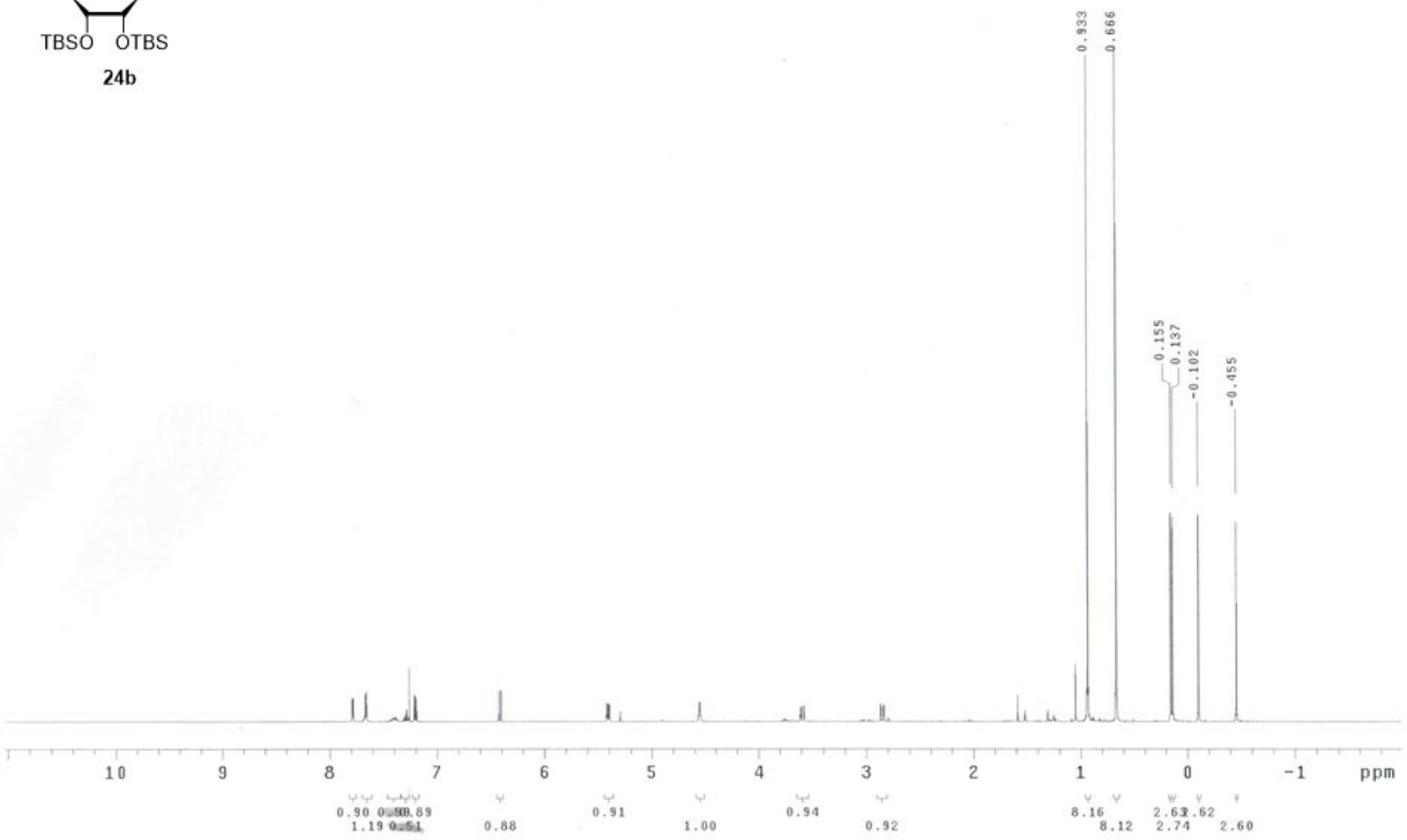

Cl
N
N
O
N
N
Cl
S
TBSO
OTBS

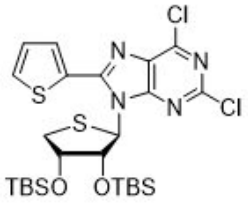
24a


49.476
49.267
49.050
48.841
48.624
26.373
26.002
220
200
180
160
140
120
100
80
60
40
20
0
ppm

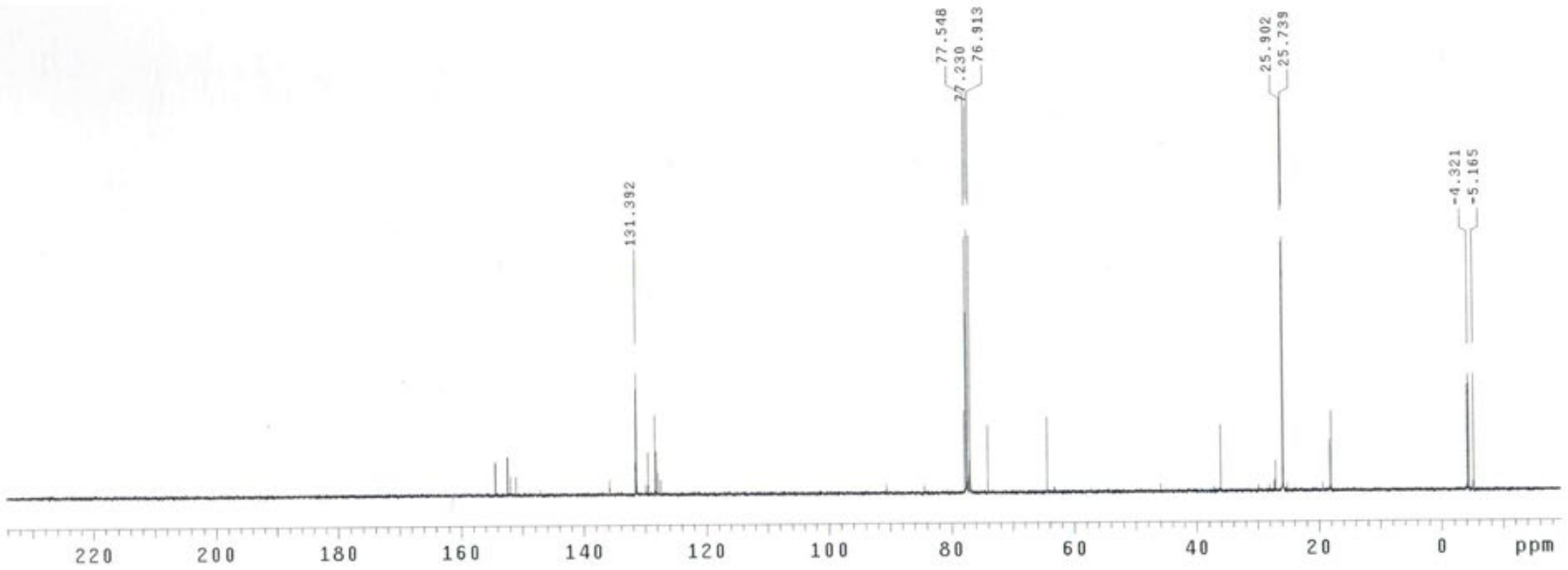

Cl
N
N
S
N
N
Cl
S
TBSO
OTBS

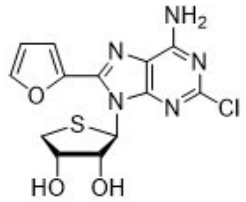
24b


0.933
0.666
0.155
0.137
-0.102
-0.455
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
0.90
0.89
1.19
0.51
0.88
0.91
1.00
0.94
0.92
8.16
8.12
2.63
2.62
2.74
2.60

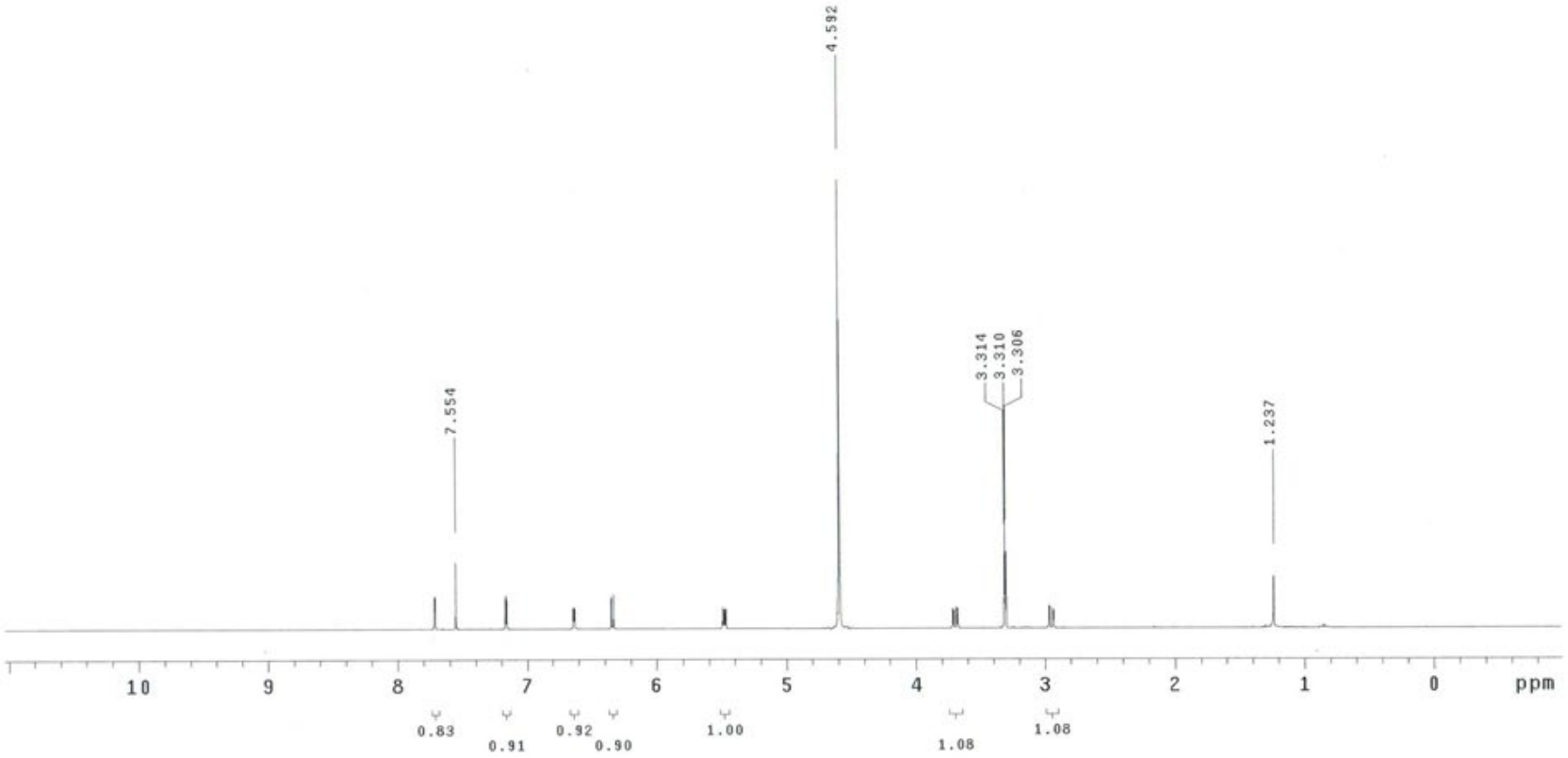

Cl
N
N
S
N
N
Cl
S
TBSO
OTBS

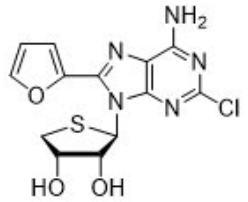
24b


77.548
77.230
76.913
25.902
25.739
-4.321
-5.165
131.392
220
200
180
160
140
120
100
80
60
40
20
0
ppm

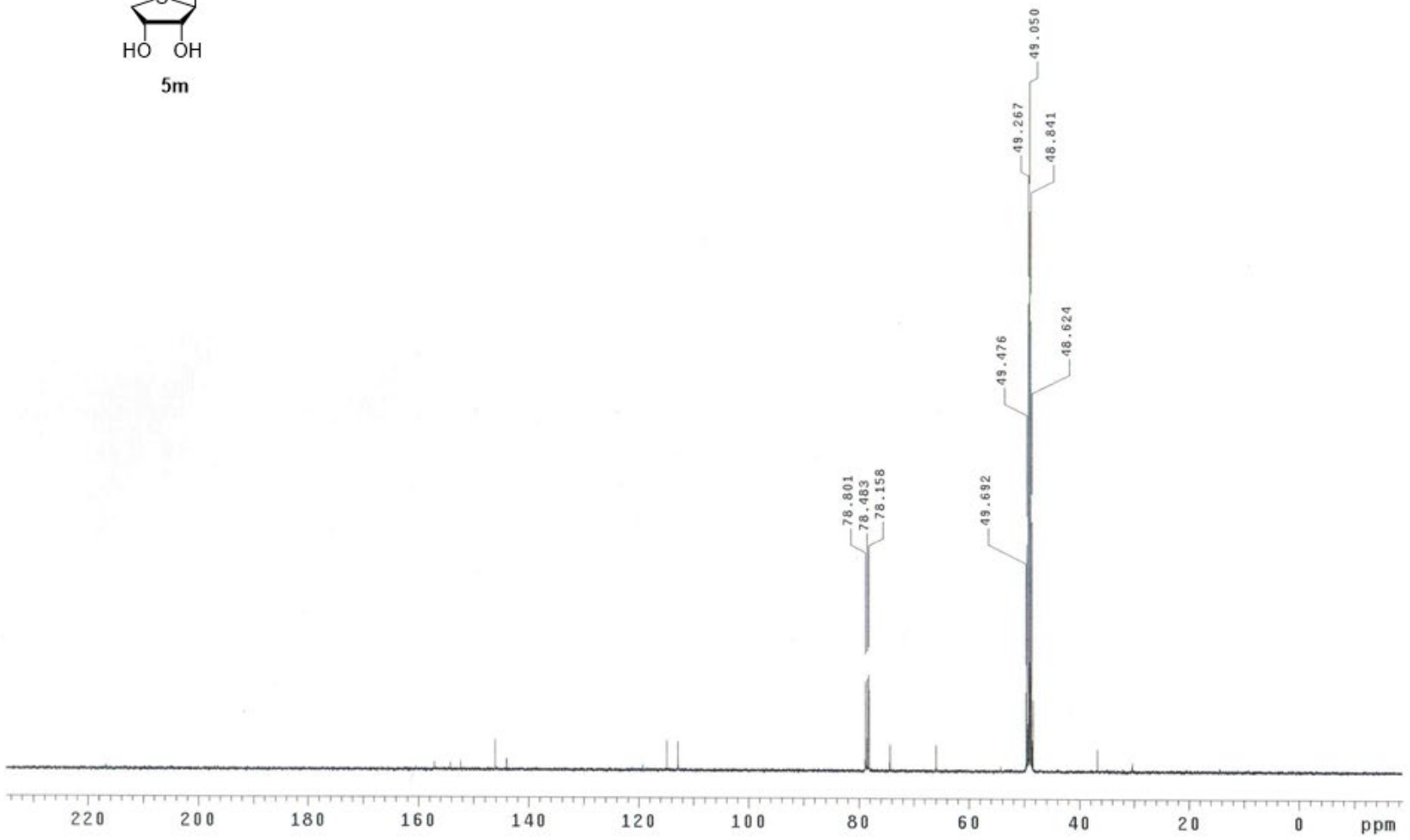

NH2
N
N
O
N
N
Cl
S
HO
OH

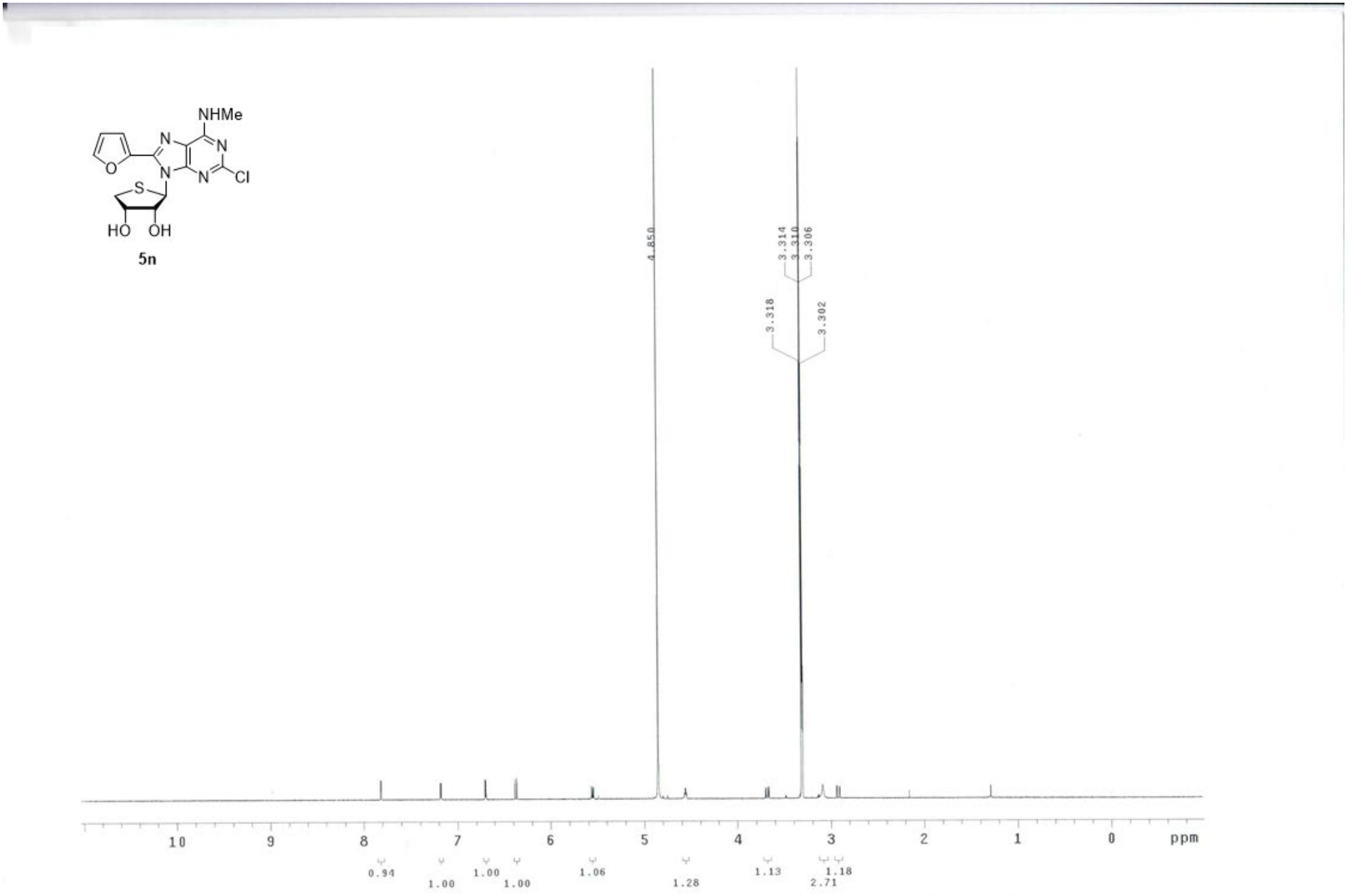
5m

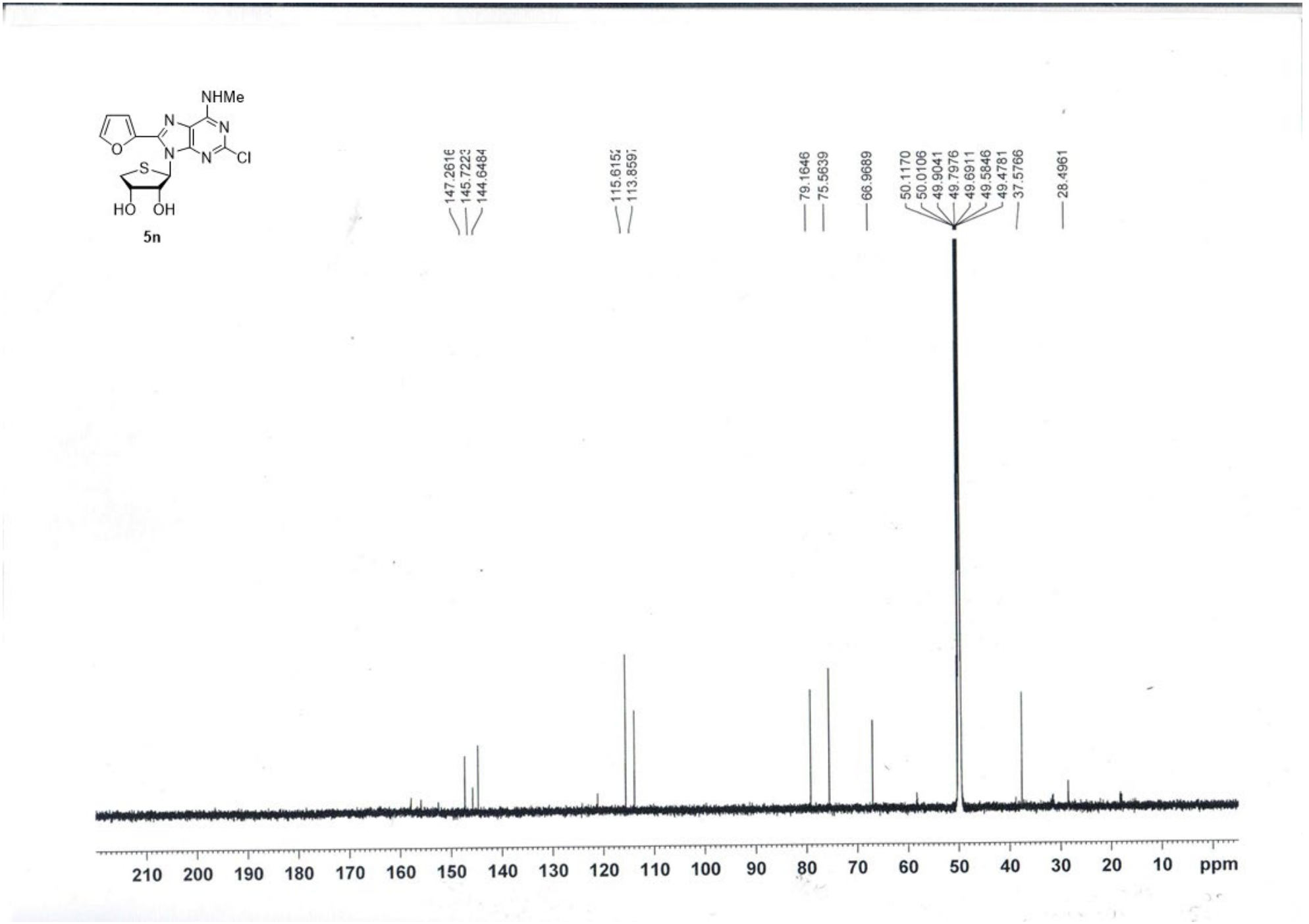

4.592
7.554
3.314
3.310
3.306
1.237
10
9
8
7
6
5
4
3
2
1
0
ppm
0.83
0.91
0.92
0.90
1.00
1.08
1.08

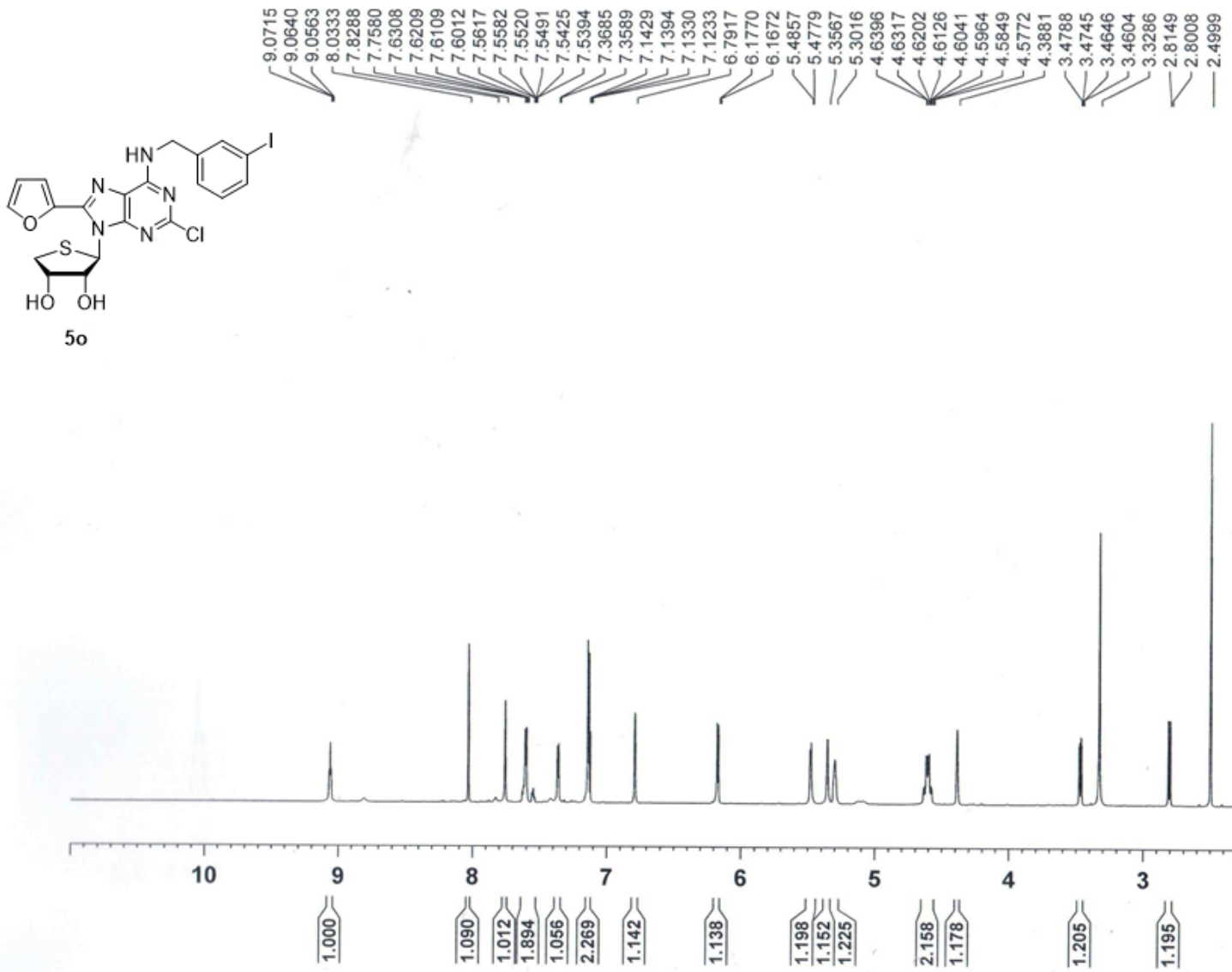

NH2
N
N
O
N
N
Cl
S
HO
OH

5m

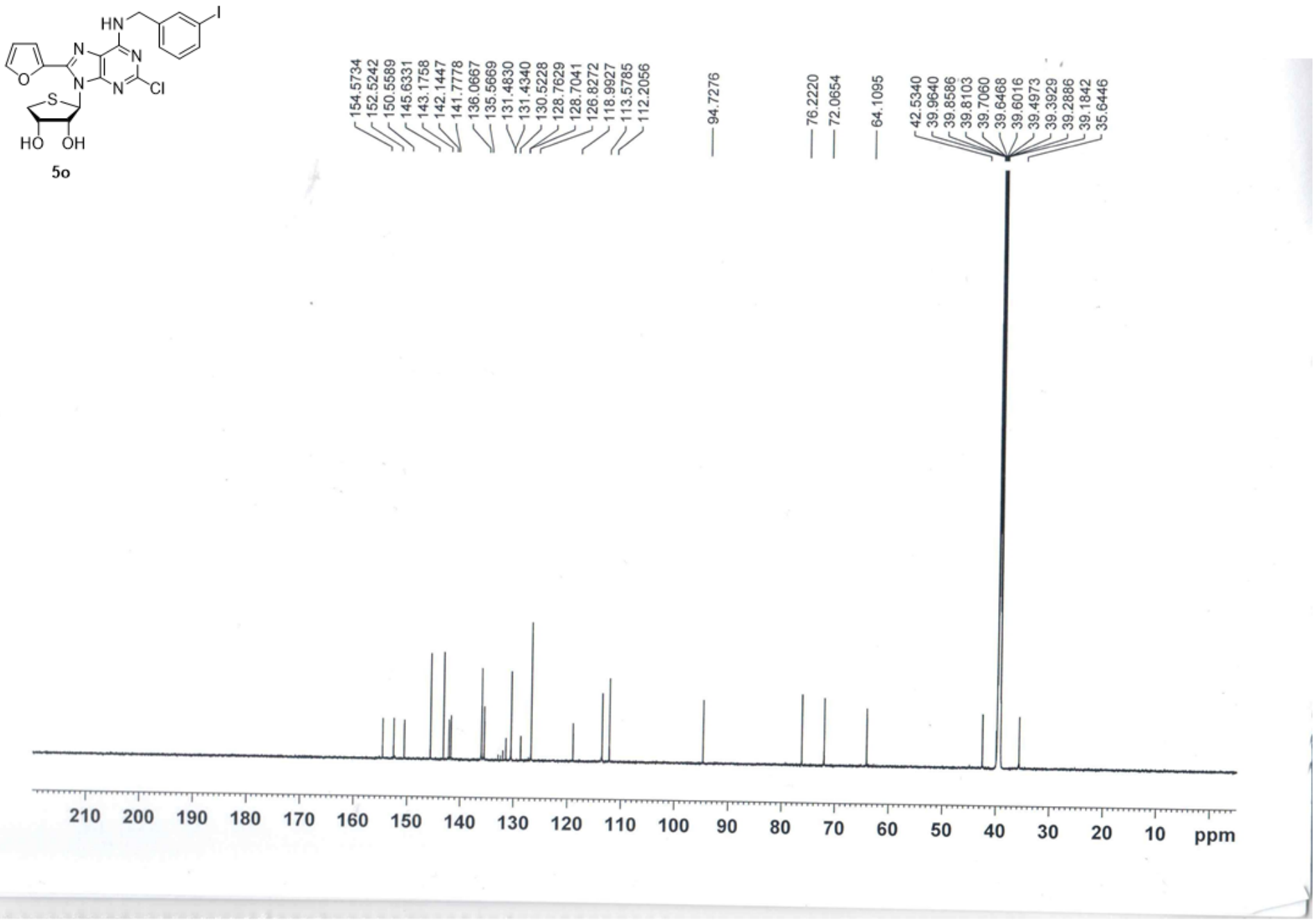

49.050
49.267
48.841
48.624
49.476
49.692
78.801
78.483
78.158
220
200
180
160
140
120
100
80
60
40
20
0
ppm

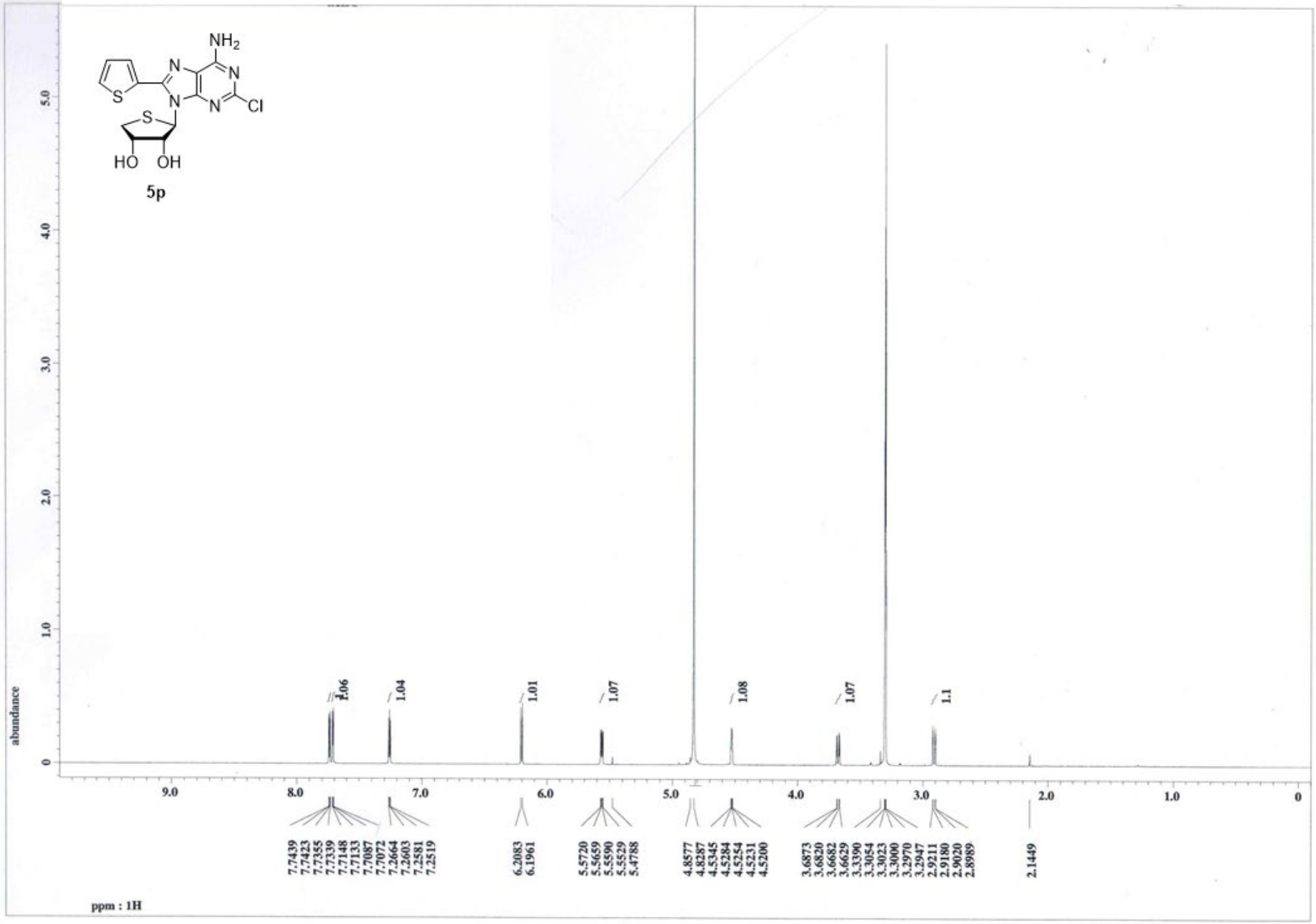

NHMe
N
N
N
N
Cl
O
S
HO
OH
5n
4.850
3.318
3.314
3.310
3.306
3.302
10
9
8
7
6
5
4
3
2
1
0
ppm
0.94
1.00
1.00
1.00
1.06
1.28
1.13
2.71
1.18

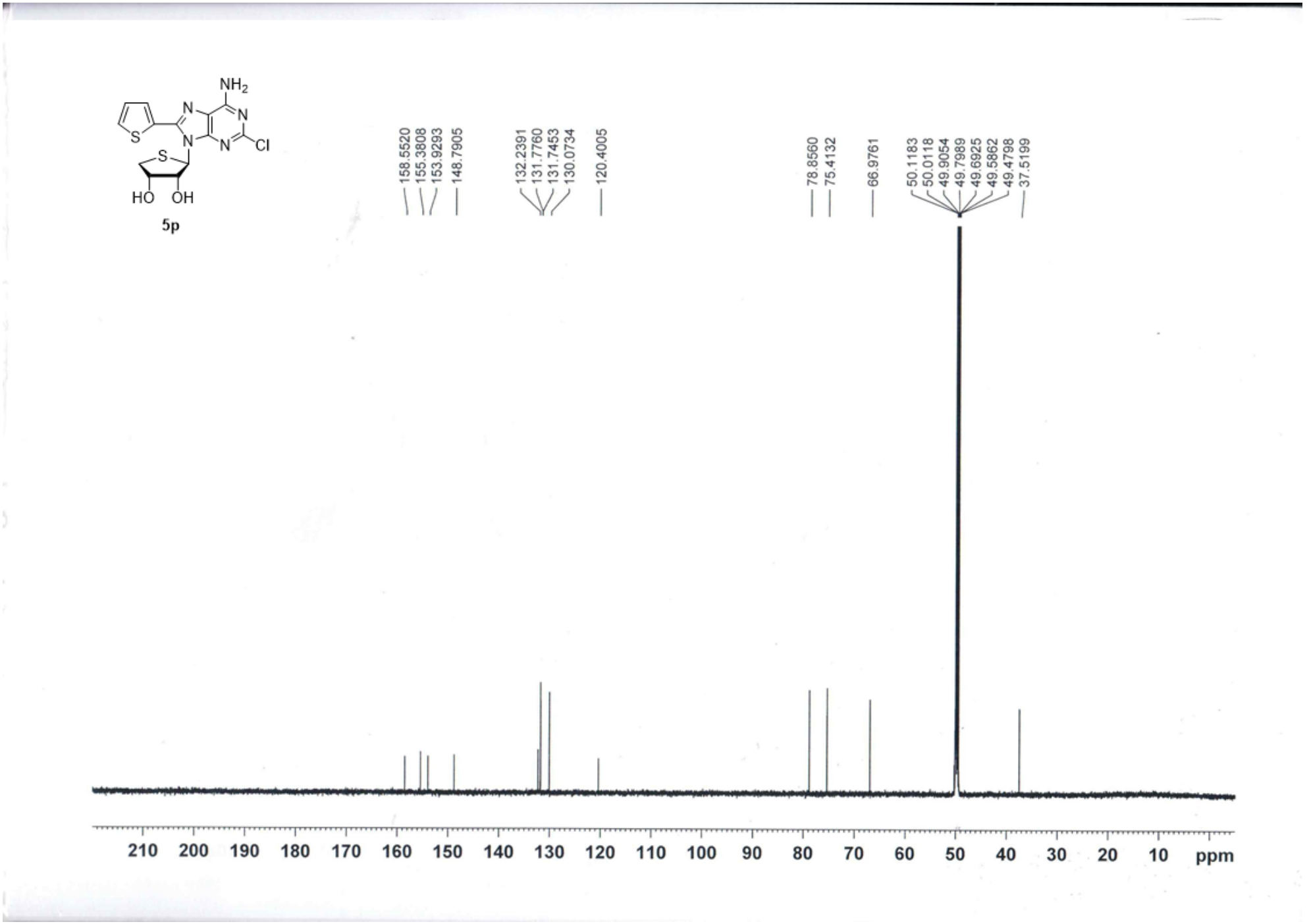

NHMe
N
N
N
N
Cl
O
S
HO
OH
5n
147.2616
145.7223
144.6484
115.6152
113.8591
79.1646
75.5639
66.9689
50.1170
50.0106
49.9041
49.7976
49.6911
49.5846
49.4781
37.5766
28.4961
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

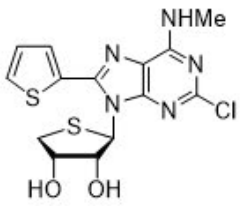

9.0715
9.0640
9.0563
8.0333
7.8288
7.7580
7.6308
7.6209
7.6109
7.6012
7.5617
7.5582
7.5520
7.5491
7.5425
7.5394
7.3685
7.3589
7.1429
7.1394
7.1330
7.1233
6.7917
6.1770
6.1672
5.4857
5.4779
5.3567
5.3016
4.6396
4.6317
4.6202
4.6126
4.6041
4.5964
4.5849
4.5772
4.3881
3.4788
3.4745
3.4646
3.4604
3.3286
2.8149
2.8008
2.4999
HN
I
N
N
N
N
Cl
O
S
HO
OH
10
9
8
7
6
5
4
3
2
1
ppm
1.000
1.090
1.012
1.894
1.056
2.269
1.142
1.138
1.198
1.152
1.225
2.158
1.178
1.205
1.195

5o


HN
I
N
N
N
N
Cl
O
S
HO
OH
5o
154.5734
152.5242
150.5589
145.6331
143.1758
142.1447
141.7778
136.0667
135.5669
131.4830
131.4340
130.5228
128.7629
128.7041
126.8272
118.9927
113.5785
112.2056
94.7276
76.2220
72.0654
64.1095
42.5340
39.9640
39.8586
39.8103
39.7060
39.6468
39.6016
39.4973
39.3929
39.2886
39.1842
35.6446
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

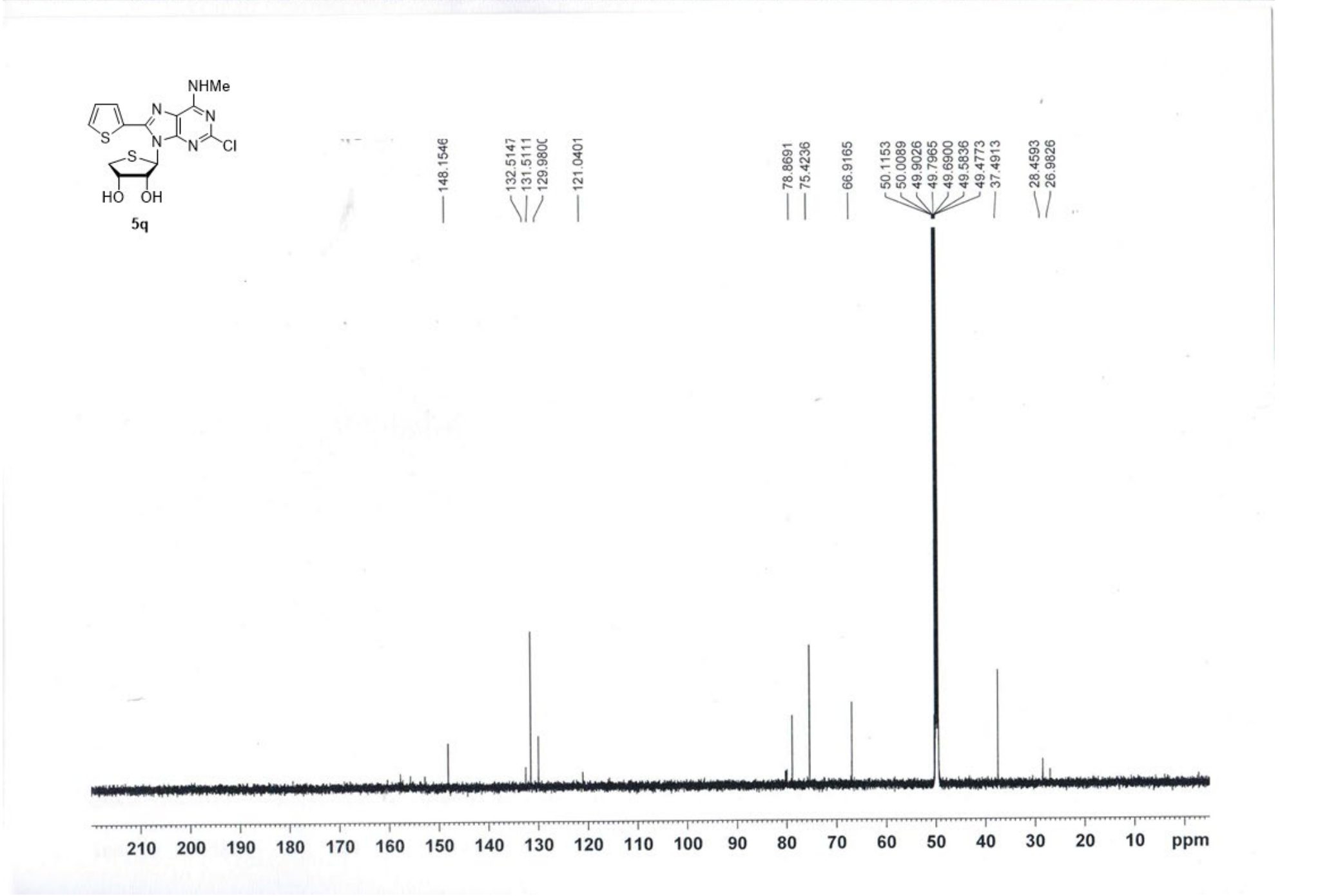

NH2
N
N
N
N
S
S
Cl
HO
OH
5p
abundance
ppm : 1H
1.06
1.04
1.01
1.07
1.08
1.07
1.1
7.7439
7.7423
7.7355
7.7339
7.7148
7.7133
7.7087
7.7072
7.2664
7.2603
7.2581
7.2519
6.2083
6.1961
5.5720
5.5659
5.5590
5.5529
5.4788
4.8577
4.8287
4.5345
4.5284
4.5254
4.5231
4.5200
3.6873
3.6820
3.6682
3.6629
3.3390
3.3054
3.3023
3.3000
3.2970
3.2947
2.9211
2.9180
2.9020
2.8989
2.1449

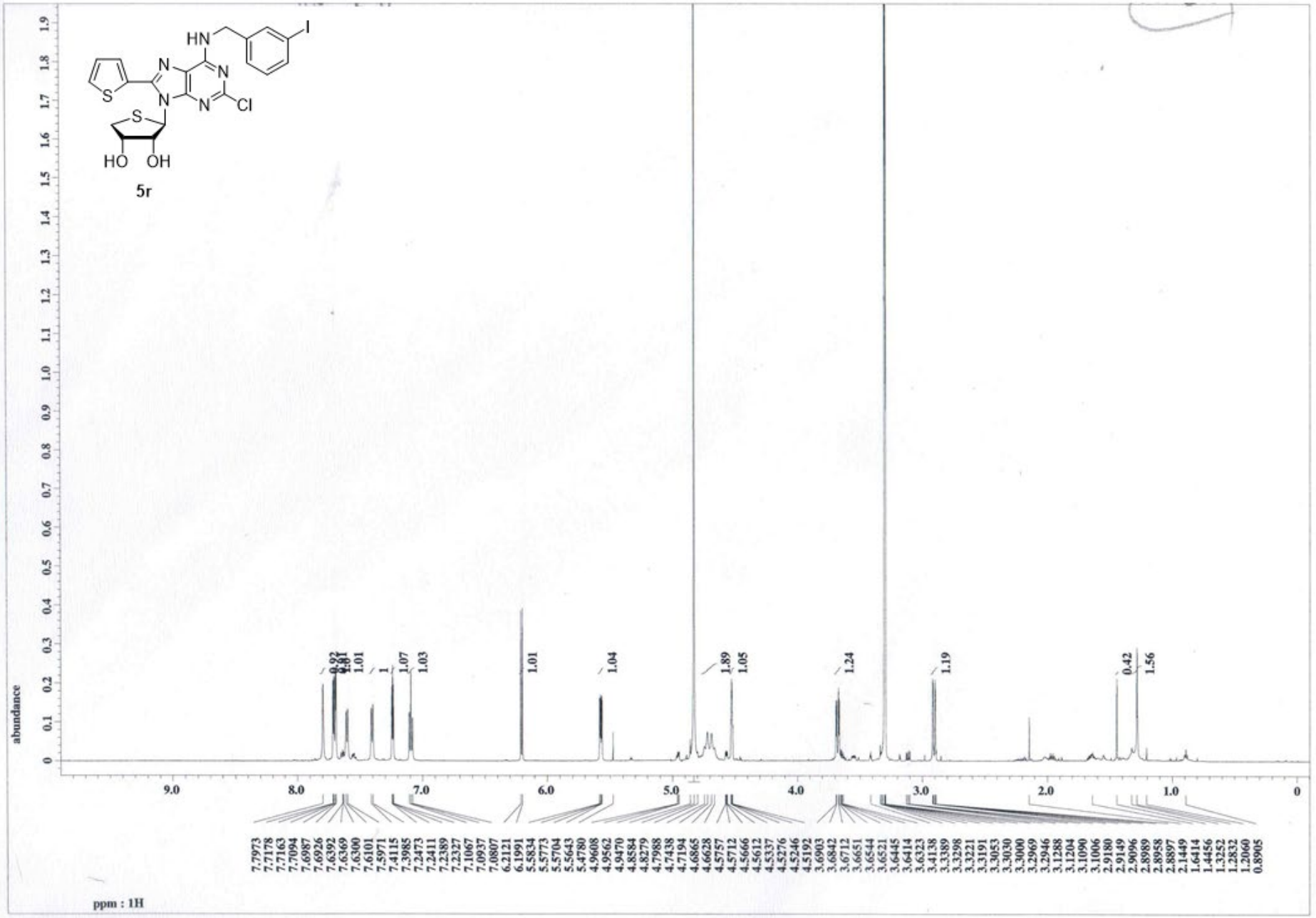

NH2
N
N
N
N
Cl
S
S
HO
OH
5p
158.5520
155.3808
153.9293
148.7905
132.2391
131.7760
131.7453
130.0734
120.4005
78.8560
75.4132
66.9761
50.1183
50.0118
49.9054
49.7989
49.6925
49.5862
49.4798
37.5199
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

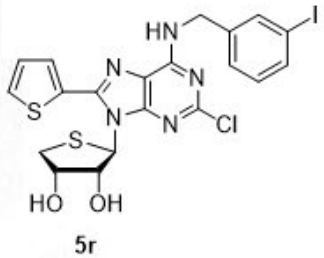

NHMe
N
N
S
N
N
Cl
S
HO
OH

5q

4.852
3.310
3.314
3.306
3.302
10
9
8
7
6
5
4
3
2
1
0
ppm
1.89
0.98
0.97
1.00
1.05
1.05
2.80
1.19

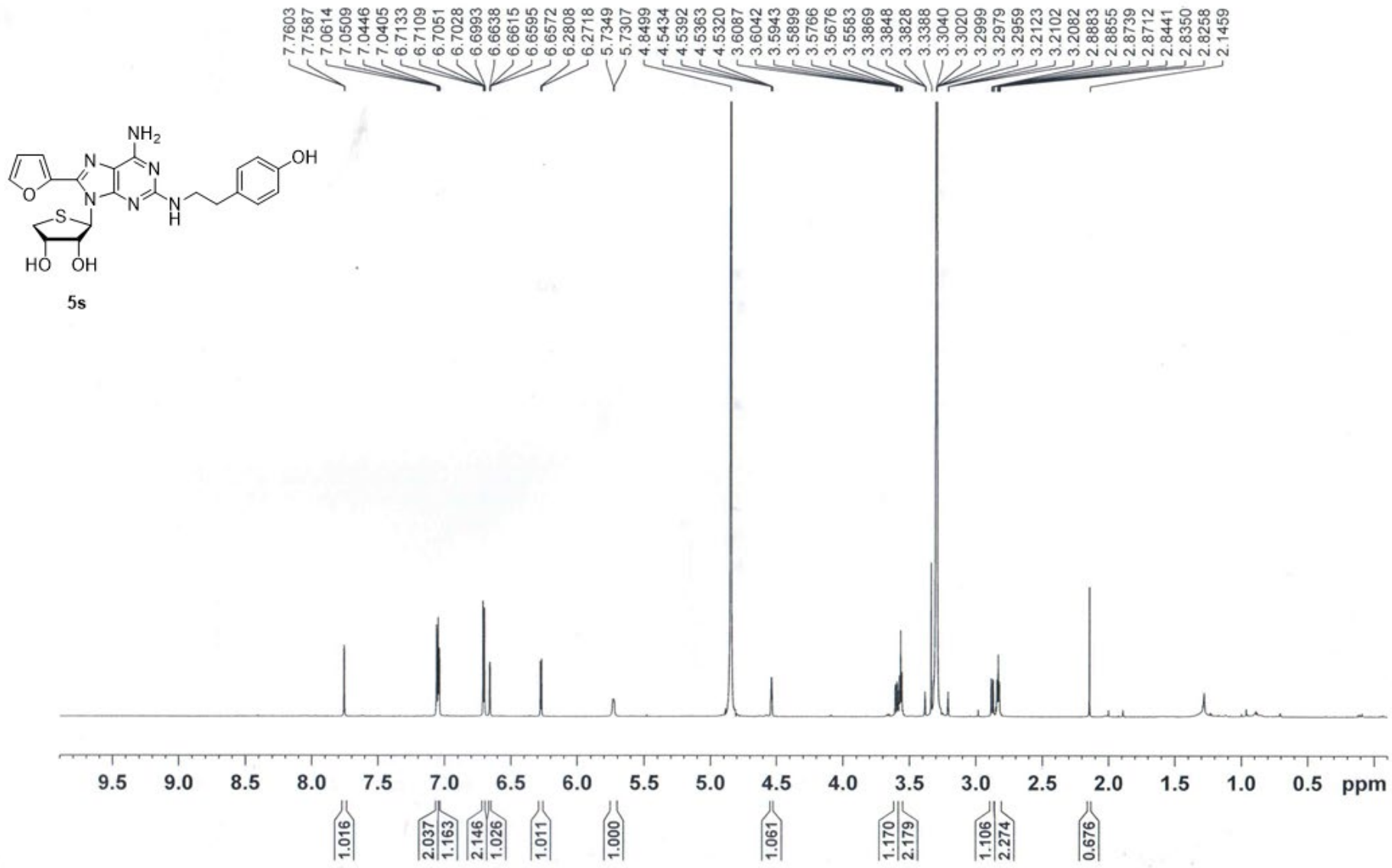

NHMe
N
N
S
N
N
Cl
S
HO
OH
5q
148.1546
132.5147
131.5111
129.980
121.0401
78.8691
75.4236
66.9165
50.1153
50.0089
49.9026
49.7965
49.6900
49.5836
49.4773
37.4913
28.4593
26.9826
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

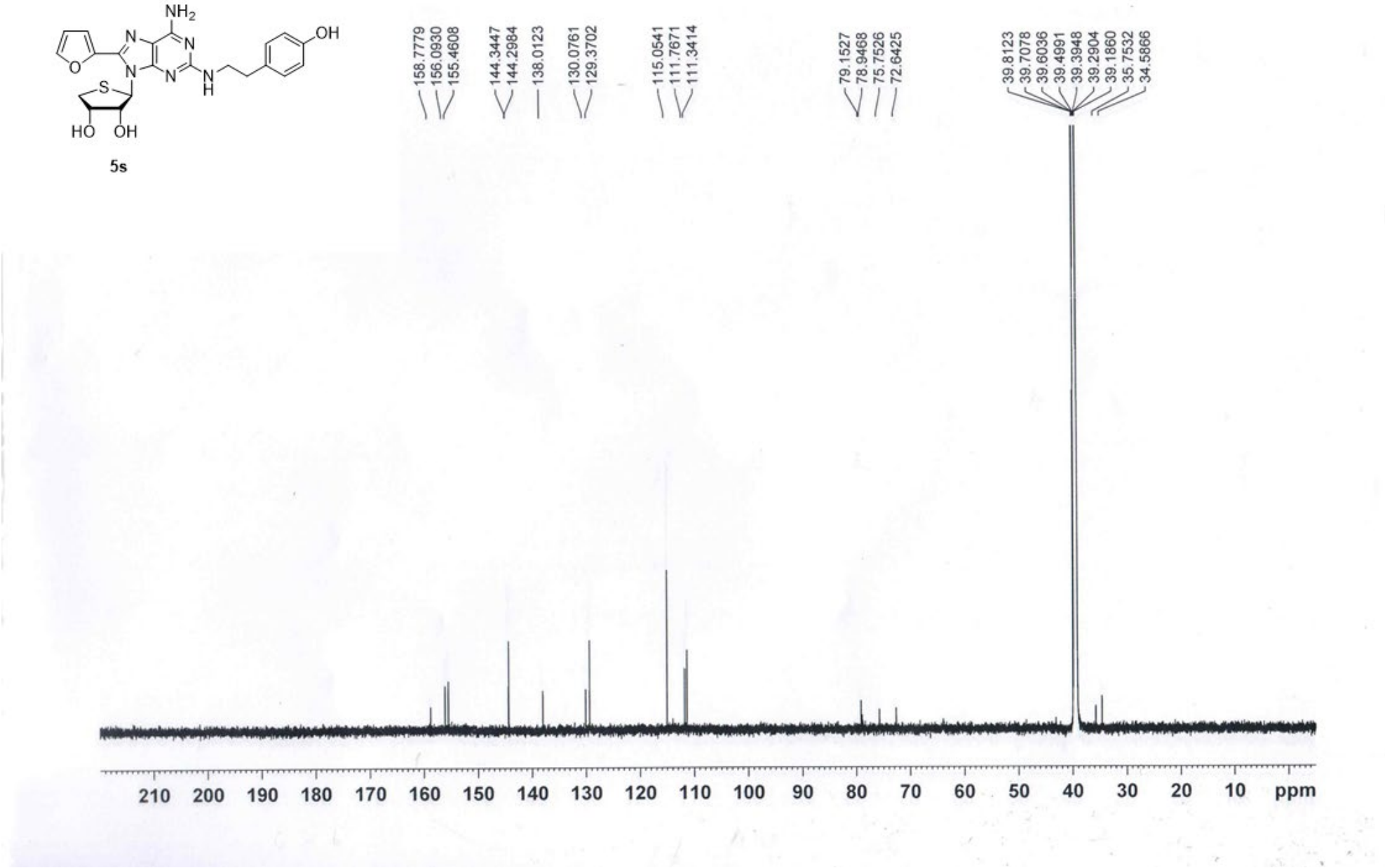

HN
I
N
N
N
N
Cl
S
S
HO
OH
5r
abundance
ppm : 1H


HN
I
N
N
S
N
N
Cl
S
HO
OH

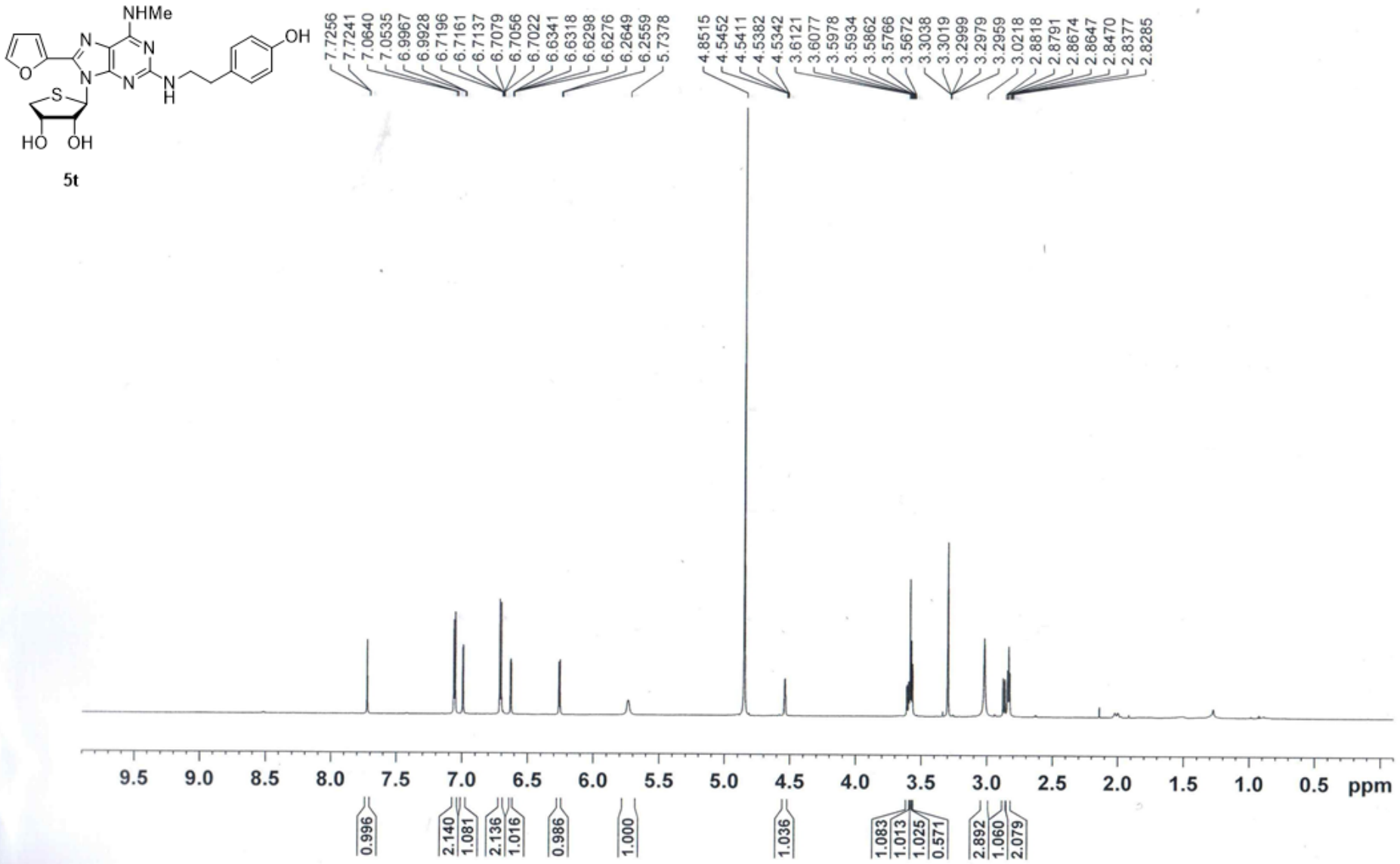
5r

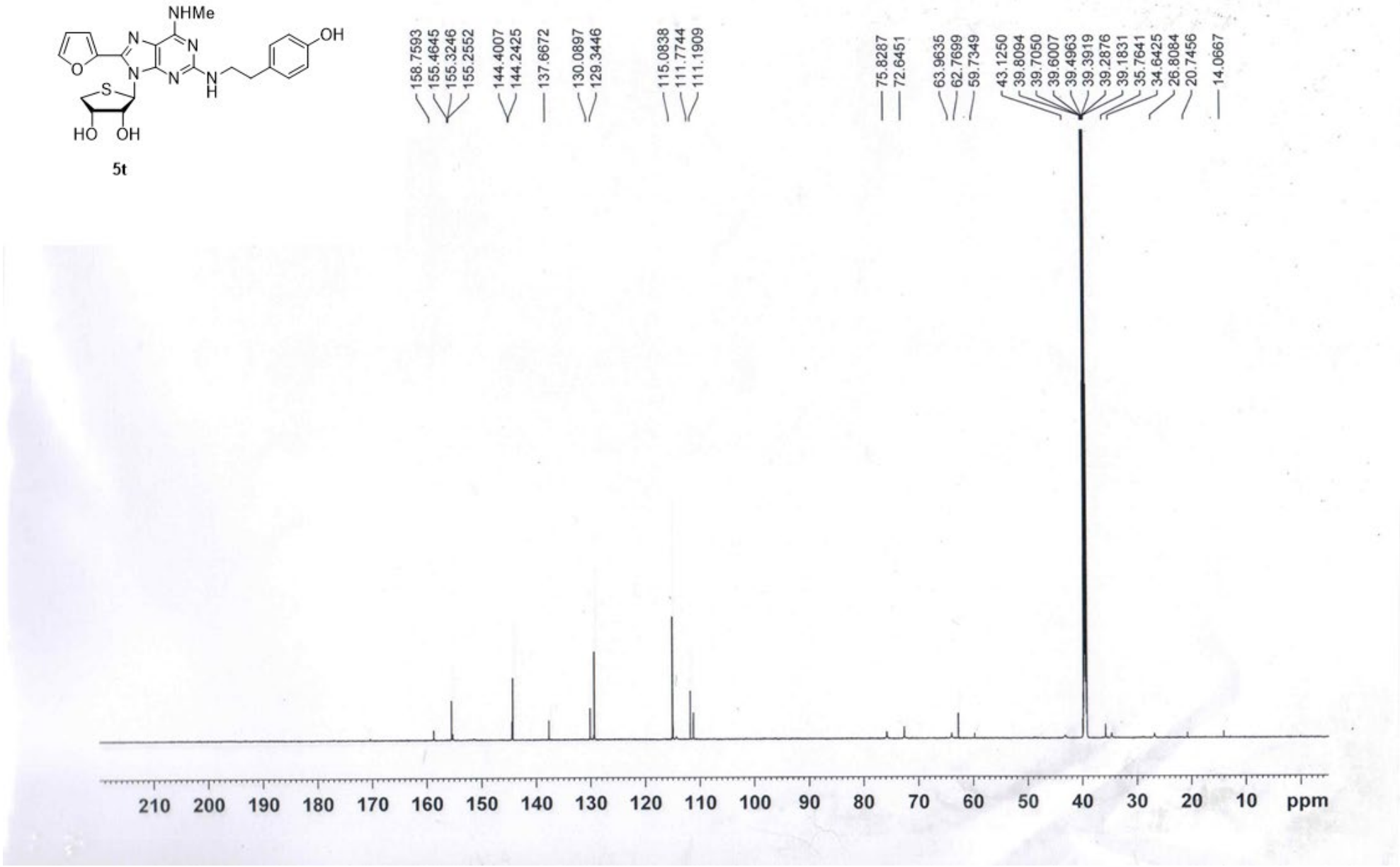
154.4050
152.3587
150.9335
145.6376
141.8221
136.1064
135.5629
130.5240
130.4604
130.2371
129.2225
128.2906
126.8643
118.8381
94.7142
89.7718
83.8409
75.9109
71.8950
64.1363
59.8797
42.5572
39.8118
39.7074
39.6031
39.4986
39.3944
39.2899
39.1856
36.7529
35.6027
27.8087
26.7230
24.6784
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

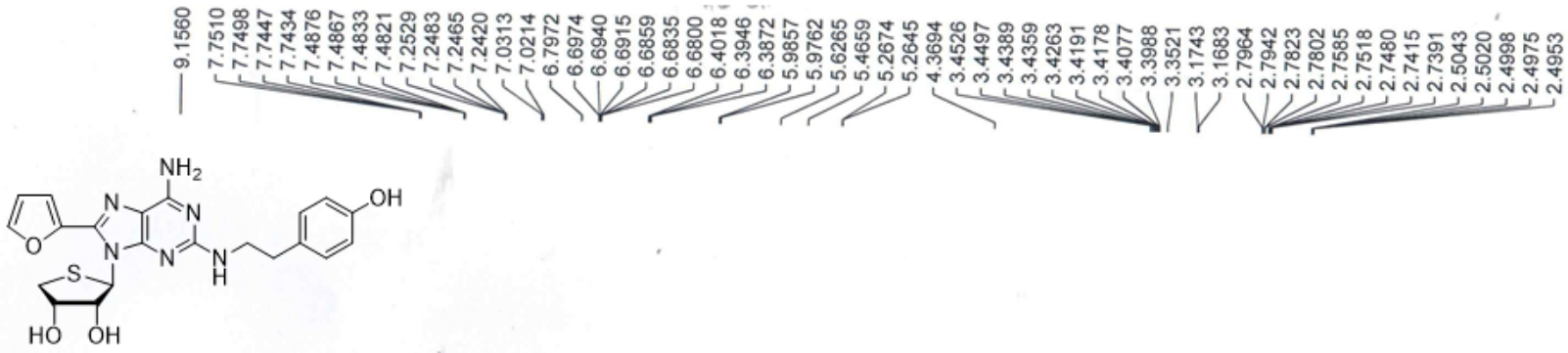

7.7603
7.7587
7.0614
7.0509
7.0446
7.0405
6.7133
6.7109
6.7051
6.7028
6.6993
6.6638
6.6615
6.6595
6.6572
6.2808
6.2718
5.7349
5.7307
4.8499
4.5434
4.5392
4.5363
4.5320
3.6087
3.6042
3.5943
3.5899
3.5766
3.5676
3.5583
3.3869
3.3848
3.3828
3.3388
3.3040
3.3020
3.2999
3.2979
3.2959
3.2123
3.2102
3.2082
2.8883
2.8855
2.8739
2.8712
2.8441
2.8350
2.8258
2.1459
NH2
OH
N
N
N
N
O
S
N
H
HO
OH
5s
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
ppm
1.016
2.037
1.163
2.146
1.026
1.011
1.000
1.061
1.170
2.179
1.106
2.274
0.676


NH2
OH
N
N
N
N
O
S
NH
HO
OH
158.7779
156.0930
155.4608
144.3447
144.2984
138.0123
130.0761
129.3702
115.0541
111.7671
111.3414
79.1527
78.9468
75.7526
72.6425
39.8123
39.7078
39.6036
39.4991
39.3948
39.2904
39.1860
35.7532
34.5866
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

5s

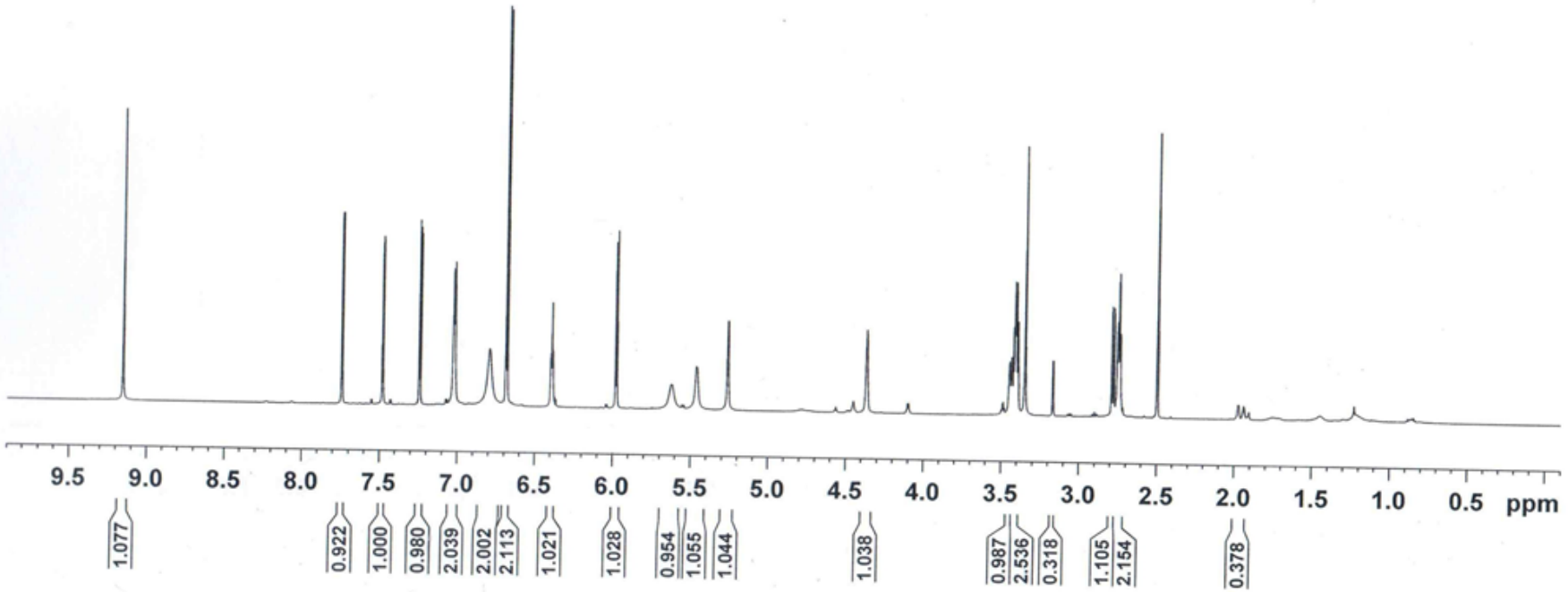

NHMe
OH
N
N
N
N
O
S
N
H
HO
OH
5t
7.7256
7.7241
7.0640
7.0535
6.9967
6.9928
6.7196
6.7161
6.7137
6.7079
6.7056
6.7022
6.6341
6.6318
6.6298
6.6276
6.2649
6.2559
5.7378
4.8515
4.5452
4.5411
4.5382
4.5342
3.6121
3.6077
3.5978
3.5934
3.5862
3.5766
3.5672
3.3038
3.3019
3.2999
3.2979
3.2959
3.0218
2.8818
2.8791
2.8674
2.8647
2.8470
2.8377
2.8285
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
ppm
0.996
2.140
1.081
2.136
1.016
0.986
1.000
1.036
1.083
1.013
1.025
0.571
2.892
1.060
2.079


NHMe
OH
HO
OH
5t
158.7593
155.4645
155.3246
155.2552
144.4007
144.2425
137.6672
130.0897
129.3446
115.0838
111.7744
111.1909
75.8287
72.6451
63.9635
62.7699
59.7349
43.1250
39.8094
39.7050
39.6007
39.4963
39.3919
39.2876
39.1831
35.7641
34.6425
26.8084
20.7456
14.0667
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


9.1560
7.7510
7.7498
7.7447
7.7434
7.4876
7.4867
7.4833
7.4821
7.2529
7.2483
7.2465
7.2420
7.0313
7.0214
6.7972
6.6974
6.6940
6.6915
6.6859
6.6835
6.6800
6.4018
6.3946
6.3872
5.9857
5.9762
5.6265
5.4659
5.2674
5.2645
4.3694
3.4526
3.4497
3.4389
3.4359
3.4263
3.4191
3.4178
3.4077
3.3988
3.3521
3.1743
3.1683
2.7964
2.7942
2.7823
2.7802
2.7585
2.7518
2.7480
2.7415
2.7391
2.5043
2.5020
2.4998
2.4975
2.4953

NH2
N
N
N
N
H
O
S
OH
HO
OH

5u


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
ppm
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0.922
1.000
0.980
2.039
2.002
2.113
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1.044
1.038
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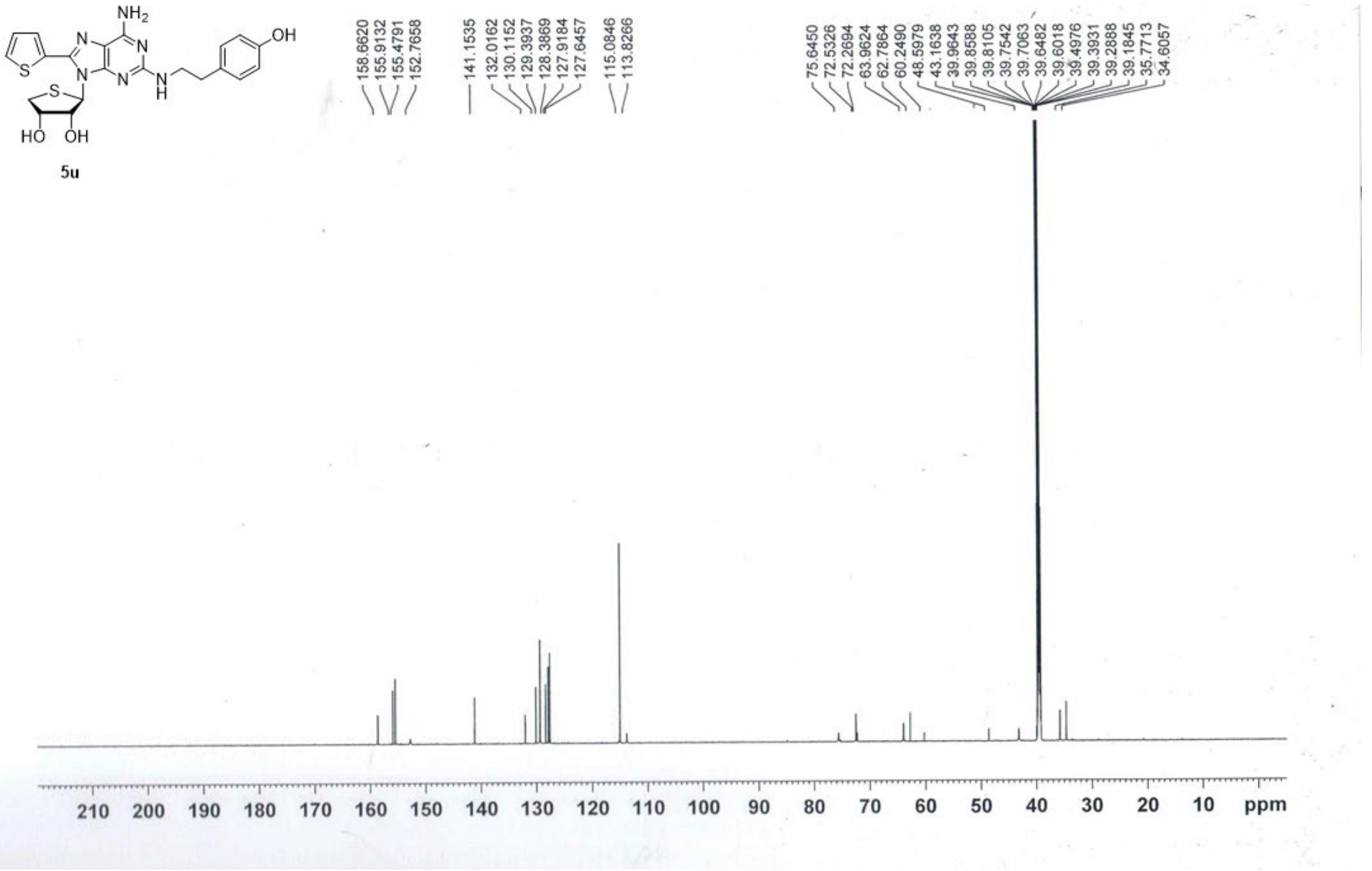
0.378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OH
S
N
N
N
N
H
S
HO
OH
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155.4791
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141.1535
132.0162
130.1152
129.3937
128.3869
127.9184
127.6457
115.0846
113.8266
75.6450
72.5326
72.2694
63.9624
62.7864
60.2490
48.5979
43.1638
39.9643
39.8588
39.8105
39.7542
39.7063
39.6482
39.6018
39.4976
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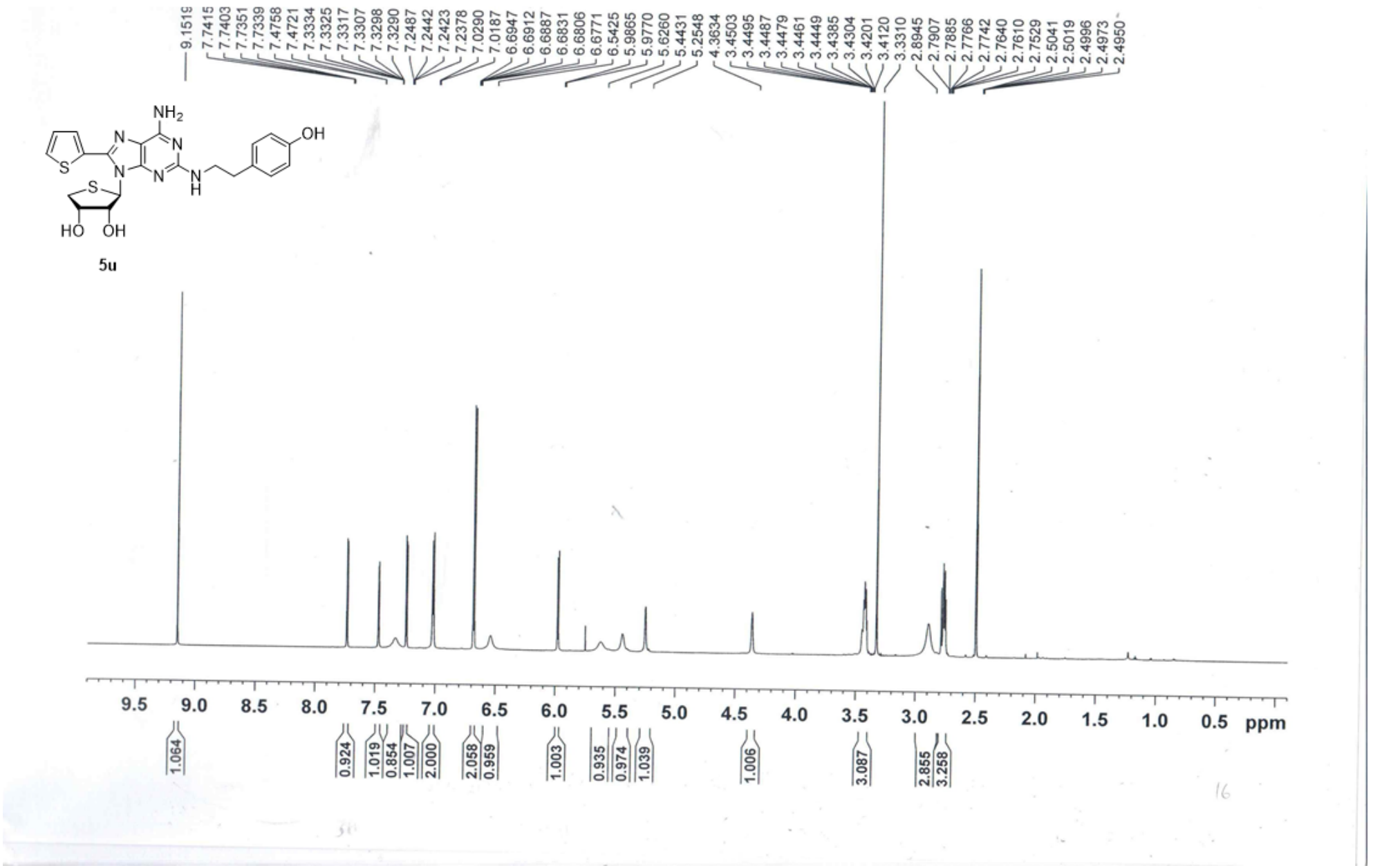
39.1845
35.7713
34.6057
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

5u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7.7339
7.4758
7.4721
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7.3325
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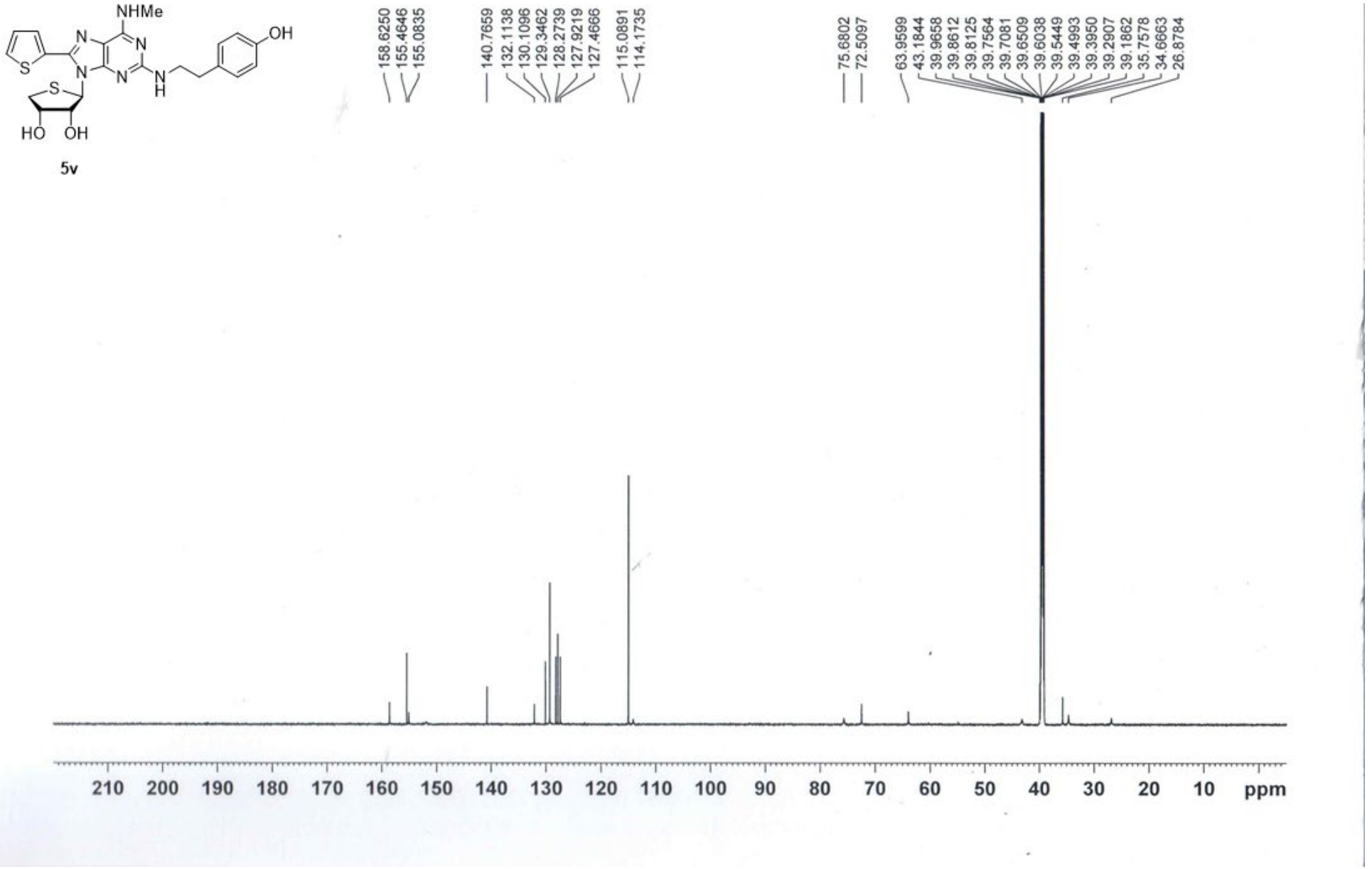
7.3307
7.3298
7.3290
7.2487
7.2442
7.2423
7.2378
7.0290
7.0187
6.6947
6.6912
6.6887
6.6831
6.6806
6.6771
6.5425
5.9865
5.9770
5.6260
5.4431
5.2548
4.3634
3.4503
3.4495
3.4487
3.4479
3.4461
3.4449
3.4385
3.4304
3.4201
3.4120
3.3310
2.8945
2.7907
2.7885
2.7766
2.7742
2.7640
2.7610
2.7529
2.5041
2.5019
2.4996
2.4973
2.4950
NH2
OH
N
N
N
N
S
S
NH
HO
OH
5u
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
ppm
1.064
0.924
1.019
0.854
1.007
2.000
2.058
0.959
1.003
0.935
0.974
1.039
1.006
3.087
2.855
3.258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S
N
N
N
N
H
S
HO
OH
5v
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132.1138
130.1096
129.3462
128.2739
127.9219
127.4666
115.0891
114.1735
75.6802
72.5097
63.9599
43.1844
39.9658
39.8612
39.8125
39.7564
39.7081
39.6509
39.6038
39.5449
39.4993
39.3950
39.2907
39.1862
35.7578
34.6663
26.8784
210
200
190
180
170
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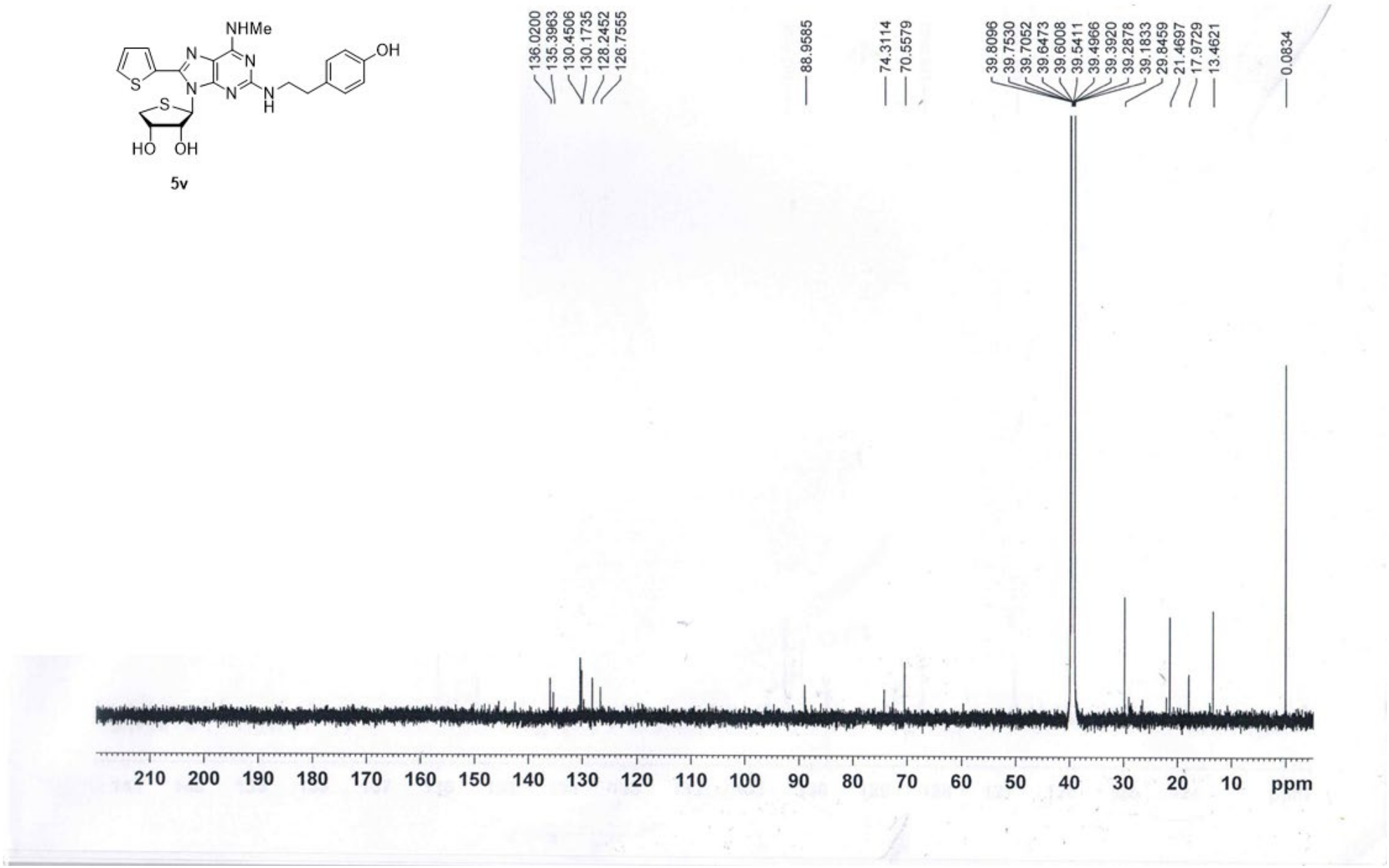
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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OH
S
N
N
N
N
S
N
H
HO
OH
5v
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130.1735
128.2452
126.7555
88.9585
74.3114
70.5579
39.8096
39.7530
39.7052
39.6473
39.6008
39.5411
39.4966
39.3920
39.2878
39.1833
29.8459
21.4697
17.9729
13.4621
0.0834
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm