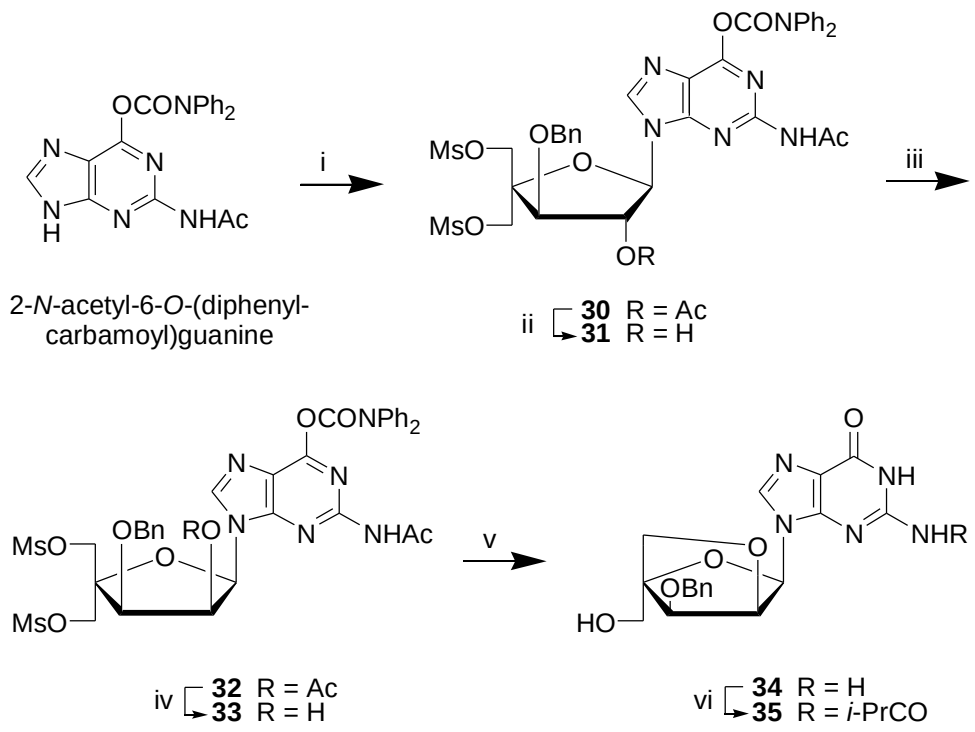
Supporting Information

Synthesis of the Guanine α-L-LNA Nucleoside 35 from Furanose 26

Synthesis of the guanine α-L-LNA nucleoside **35** is outlined in Scheme S1. Direct coupling of a carbohydrate derivative with guanine-type bases often produces N7/N9 isomeric mixtures that are difficult to separate.[1] In order to avoid the formation of the N7-regioisomer, we applied a slightly modified procedure[2] for the regioselective synthesis of the N9-regioisomeric guanine α-L-LNA nucleosides. 2-*N*-Acetyl-6-*O*-(diphenylcarbamoyl)guanine[2b] was silylated using *N*,*O*-bis(trimethylsilyl)acetamide in anhydrous 1,2-dichloroethane. The silylated guanine base was then coupled with the furanose **26** in toluene at 80 °C with trimethylsilyltriflate as Lewis acid affording nucleoside **30** in 59% yield. Selective deacylation of the 2'-*O*-acetyl group to give intermediate **31** in 95% yield was accomplished using half-saturated methanolic ammonia. Subsequent reaction with trifluoromethanesulfonic anhydride in a mixture of pyridine and dichloromethane afforded the 2'-*O*-triflate intermediate according to analytical TLC. Without purification, this intermediate was immediately reacted with potassium acetate in the presence of 18-crown-6 in toluene to yield the C2'-epimeric nucleoside **32** in 46% yield. The 2'-*O*-acetyl group was chemoselectively deacylated using half saturated methanolic ammonia giving compound **33** in 96% yield. Ring closure of nucleoside **33** was performed using sodium hydride in THF at 0 °C. The remaining 5'-*O*-mesyl group was subsequently substituted with an acetate group by reaction with potassium acetate in the presence of 18-crown-6 in dioxane. The crude product obtained was then directly subjected to treatment with saturated methanolic ammonia at room temperature to give nucleoside **34**

in 35% yield from **33**. The C2 exocyclic amino group of **34** was protected using a transient protection protocol (*O*-trimethylsilylation with chlorotrimethylsilane in pyridine, 2-*N*-protection with isobutyric anhydride, and desilylation) to give nucleoside **35** in 72% yield. Attempted debenzylation of **35** with ammonium formate and Pd/C gave a mixture of several products (according to analytical TLC and NMR spectroscopy) which in our hands proved impossible to separate.

Scheme S1.[a] Synthesis of the α-L-LNA Guanine Nucleoside **35**



[a] *Reagents, conditions and yields:* (i) a) BSA, DCE, 80 °C; b) **26**, TMSOTf, toluene, 80 °C (59%); (ii) half-sat. NH_3 in MeOH, 0 °C (95%); (iii) a) Tf_2O, pyridine, CH_2Cl_2, -30 °C; b) KOAc, 18-crown-6, toluene, 80 °C (46%); (iv) half-sat. NH_3 in MeOH, 0 °C (96%); (v) a)

NaH, THF, 0 ^{o}C; b) KOAc, 18-crown-6, dioxane, 80 ^{o}C; c) sat. NH_3 in MeOH, rt (35%); (vi) a) TMSCl, pyridine, rt; b) $(i\text{-PrCO})_2O$, pyridine, rt (72%).

References

1. Imazawa, M.; Eckstein, F. *J. Org. Chem.* **1978**, *43*, 3044.

2. (a) Zou, R.; Robins, M. J. *Can. J. Chem.* **1987**, *65*, 1436. (b) Robins, M. J.; Zou, R.; Guo, Z.; Wnuk, S. F. *J. Org. Chem.* **1996**, *61*, 9207.

Experimental Procedures for Conversion of Furanose 26 into Nucleoside 35

9-(2-*O*-Acetyl-3-*O*-benzyl-5-*O*-methanesulfonyl-4-*C*-methanesulfonyloxymethyl-*α*-L-*threo*-pentofuranosyl)-2-*N*-acetyl-6-*O*-(diphenylcarbamoyl)guanine (30). *N,O*-Bis(trimethylsilyl)acetamide (2.74 mL, 11.2 mmol) was added to a suspension of 2-*N*-acetyl-6-*O*-(diphenylcarbamoyl)guanine[2b] (3.29 g, 8.48 mmol) in anhydrous 1,2-dichloroethane (280 mL), and stirring was continued at 80 °C for 15 min. The clear solution was evaporated to dryness under reduced pressure, the residue was dissolved in anhydrous toluene (60 mL), and TMS-triflate (1.53 mL, 8.47 mmol) followed by a solution of furanose **26** (2.88 g, 5.65 mmol) in anhydrous toluene (60 mL) were added. The mixture was stirred at 80 °C for 1 h and then cooled to room temperature whereupon EtOAc (100 mL) was added. The resulting mixture was washed successively with a saturated aqueous solution of $NaHCO_3$ (80 mL) and brine (80 mL), dried (Na_2SO_4), and evaporated to dryness under reduced pressure. The residue was purified by silica gel column chromatography using MeOH/CH_2Cl_2 (0-5% MeOH, v/v) as eluent yielding nucleoside **30** (2.77 g, 59%) as

a clear oil. FAB-MS *m/z* 839 $[M + H]^+$; ^{1}H NMR ($CDCl_3$) δ 8.17 (s, 1H), 8.01 (s, 1H), 7.37-7.14 (m, 15H), 6.17 (s, 1H), 5.81 (s, 1H), 4.40 (m, 7H), 2.92 (s, 3H), 2.91 (s, 3H), 2.39 (s, 3H), 2.07 (s, 3H); ^{13}C NMR ($CDCl_3$) δ 195.5, 169.4, 156.3, 154.3, 152.3, 150.2, 141.9, 135.8, 129.5, 129.4, 129.2, 128.8, 128.6, 128.4, 128.4, 127.4, 127.2, 127.0, 120.9, 87.6, 86.0, 81.2, 79.2, 67.3, 65.5, 37.7, 37.6, 25.1, 20.6.

2-*N*-Acetyl-9-(3-*O*-benzyl-5-*O*-methanesulfonyl-4-*C*-(methanesulfonyloxymethyl)-*α*-L-*threo*-pentofuranosyl)-6-*O*-(diphenylcarbamoyl)guanine (31). To a solution of nucleoside **30** (3.62 g, 4.32 mmol) in MeOH (25 mL) was added a saturated solution of NH_3 in MeOH (25 mL). The solution was stirred at 0 °C for 30 min and evaporated to dryness. The residue was coevaporated with toluene (2 x 20 mL) and purified by silica gel column chromatography using MeOH/CH_2Cl_2 (0-5% MeOH, v/v) as eluent yielding nucleoside **31** (3.29 g, 95%) as a white foam. FAB-MS *m/z* 797 $[M + H]^+$; ^{1}H NMR ($CDCl_3$) δ 8.61 (s, 1H), 8.21 (s, 1H), 7.44-7.22 (m, 15H), 6.21 (s, 1H), 6.08 (d, 1H, *J* 5.7 Hz), 4.97-4.26 (m, 8H), 2.98 (s, 3H, Ms), 2.83 (s, 3H, Ms), 2.07 (s, 3H, NHAc); ^{13}C NMR ($CDCl_3$) δ 168.9, 155.9, 153.9, 151.1, 150.5, 142.3, 137.0, 129.2, 129.0, 128.7, 128.5, 128.4, 128.2, 128.1, 127.5, 127.4, 127.1, 126.9, 121.0, 89.3, 83.2, 82.5, 79.6, 73.2, 68.1, 37.5, 37.3, 24.8.

9-(2-*O*-Acetyl-3-*O*-benzyl-5-*O*-methanesulfonyl-4-*C*-(methanesulfonyloxymethyl)-*α*-L-*erythro*-pentofuranosyl)-2-*N*-acetyl-6-*O*-(diphenylcarbamoyl)guanine (32). Nucleoside **31** (1.20 g, 1.51 mmol) was coevaporated with anhydrous pyridine (2 x 10 mL), and the residue was dissolved in a mixture of anhydrous CH_2Cl_2 (30 mL) and anhydrous pyridine (6 mL). The reaction mixture was cooled to -30 °C and trifluoromethanesulfonic

anhydride (0.68 mL, 4.07 mmol) was added dropwise under stirring. After 2 h, the mixture was diluted by addition of CH_2Cl_2 (100 mL), and washing was performed using a saturated aqueous solution of $NaHCO_3$ (100 mL). The separated organic phase was dried (Na_2SO_4) and evaporated to dryness under reduced pressure. The residue was coevaporated using anhydrous toluene (2 x 20 mL) and dissolved in anhydrous toluene (45 mL). KOAc (0.74 g, 2.53 mmol) and 18-crown-6 (1.4 g, 5.27 mmol) were added and the reaction mixture was stirred for 40 min at 80 °C. The reaction mixture was evaporated to dryness under reduced pressure, and the residue was purified by silica gel column chromatography using MeOH/CH_2Cl_2 (0-5% MeOH, v/v) as eluent affording nucleoside **32** (0.59 g, 46%) as a yellowish oil. FAB-MS *m/z* 839 $[M + H]^+$; ^{1}H NMR ($CDCl_3$) δ 8.44 (s, 1H), 8.26 (s, 1H), 7.44-7.25 (m, 15H), 6.60 (d, 1H, *J* 4.1 Hz), 5.69 (m, 1H), 4.80-4.30 (m, 7H), 3.04 (s, 3H), 2.99 (s, 3H), 2.48 (s, 3H), 2.02 (s, 3H); ^{13}C NMR ($CDCl_3$) δ 187.2, 168.2, 151.1, 154.7, 152.3, 150.2, 143.2, 141.6, 136.0, 129.2, 128.8, 128.7, 128.4, 127.3, 127.2, 127.0, 126.6, 126.4, 120.2, 82.5, 82.2, 78.4, 74.7, 70.5, 68.1, 67.7, 37.9, 37.7, 25.1, 20.4.

2-*N*-Acetyl-9-(3-*O*-benzyl-5-*O*-methanesulfonyl-4-*C*-(methanesulfonyloxymethyl)-*α*-L-*erythro*-pentofuranosyl)-6-*O*-(diphenylcarbamoyl)guanine (33). To a stirred solution of nucleoside **32** (1.50 g, 1.79 mmol) in MeOH (28 mL) was added a saturated solution of NH_3 in MeOH (28 mL) at 0 °C. The solution was stirred at 0 °C for 30 min and then evaporated to dryness under reduced pressure. The residue was coevaporated with toluene (2 x 10 mL) and purified by silica gel column chromatography using MeOH/CH_2Cl_2 (0-5% MeOH, v/v) as eluent yielding nucleoside **33** (1.38 g, 96%) as a yellowish solid material which was used in the next step without further purification. FAB-

MS *m/z* 797 $[M + H]^+$; ^1H NMR ($(CD_3)_2SO$) δ 10.72 (s, 1H, D_2O exchangable), 8.53 (s, 1H), 7.52-7.31 (m, 15H), 6.44 (d, 1H, *J* 4.3 Hz), 6.19 (d, 1H, D_2O exchangable, *J* 5.1 Hz), 4.80-4.40 (m, 8H), 3.26 (s, 3H), 3.25 (s, 3H), 2.20 (s, 3H); ^{13}C NMR ($(CD_3)_2SO$) δ 168.2, 154.7, 152.3, 150.2, 145.1, 141.7, 137.6, 129.6, 128.5, 128.0, 127.9, 127.4, 127.1, 127.0, 119.6, 83.9, 82.1, 79.5, 72.6, 69.1, 68.9, 68.9, 37.1, 36.9, 24.6.

(1*R*,3*R*,4*S*,7*R*)-7-Benzyloxy-3-(guanin-9-yl)-1-hydroxymethyl-2,5-dioxabicyclo[2.2.1]heptane (34). Nucleoside **33** (0.57 g, 0.72 mmol) was coevaporated with anhydrous toluene (2 x 10 mL) and dissolved in anhydrous THF (18 mL). The solution was cooled to 0 °C and NaH (31 mg, 0.79 mmol) was added. The reaction mixture was stirred at 0 °C for 1h and then evaporated to dryness under reduced pressure. The residue was dissoved in dichloromethane (50 mL), and extraction was performed using a saturated aqueous solution of $NaHCO_3$ (20 mL). The separated organic phase was dried (Na_2SO_4) and evaporated to dryness under reduced pressure. The residue was dissolved in 1,4-dioxane (18 mL) whereupon KOAc (0.35 g, 3.60 mmol) and 18-crown-6 (0.38 g, 1.44 mmol) were added. The reaction mixture was stirred for 18 h at 80 °C and then evaporated to dryness under reduced pressure. A saturated solution of NH_3 in MeOH (25 mL) was added and stirring was continued for 24 h at room temperature. The reaction mixture was evaporated to dryness under reduced pressure, and the residue was coevaporated with toluene (2 x 20 mL) and purified by silica gel column chromatography using MeOH/AcOEt (0-15% MeOH, v/v) as eluent affording nucleoside **34** (96 mg, 35%) as a white solid material. FAB-MS *m/z* 386 $[M +H]^+$; ^1H NMR ($(CD_3)_2SO$) δ 7.89 (s, 1H), 7.39-7.30 (m, 8H), 6.06 (s, 1H), 4.68 (d, 2H, *J* 3.0 Hz), 4.63 (s, 1H), 4.36 (s, 1H), 4.07-3.99 (m, 2H),

3.70-3.55 (m, 3H); ^{13}C NMR ($(CD_3)_2SO$) δ 174.3, 155.5, 151.4, 137.9, 134.3, 128.3, 127.6, 127.5, 89.4, 82.9, 79.6, 76.7, 73.0, 71.0, 69.4.

(1*R*,3*R*,4*S*,7*R*)-7-Benzyloxy-1-hydroxymethyl-3-(2-*N*-isobutyrylguanin-9-yl)-2,5-dioxabicyclo[2.2.1]heptane (35). To nucleoside **34** (0.14 g, 0.36 mmol) dissolved in anhydrous pyridine (4 mL) was added chlorotrimethylsilane (0.45 mL, 3.58 mmol). After stirring for 15 min, isobutyric anhydride (0.59 mL, 3.58 mmol) was added and the mixture was stirred for 3 h at room temperature. The mixture was subsequently cooled to 0 ^{o}C and a saturated aqueous solution of $NaHCO_3$ (10 mL) was added. The mixture was stirred for 15 min at room temperature and then extracted with AcOEt (15 mL). The separated organic phase was washed with brine (10 mL), dried (Na_2SO_4) and evaporated to dryness under reduced pressure. The residue was coevaporated with toluene (2 x 10 mL) and purified by silica gel column chromatography using MeOH/AcOEt (0-15%, v/v) as eluent yielding nucleoside **35** (0.12 g, 72%) as a white solid material. FAB-MS *m/z* 456 $[M + H]^+$; ^{1}H NMR (CD_3OD) δ 8.05 (s, 1H), 7.36 (s, 1H), 7.32-7.20 (m, 6H), 6.04 (s, 1H), 4.60 (s, 2H), 4.47 (s, 1H), 4.23 (s, 1H), 4.06 (d, 1H, *J* 8.4 Hz), 3.91 (d, 1H, *J* 8.2 Hz), 3.78 (s, 2H), 3.52 (s, 1H), 2.51 (q, 1H, *J* 7.0 Hz), 1.1 (m, 6H); ^{13}C NMR (CD_3OD) δ 179.7, 155.6, 148.4, 147.8, 137.5, 136.7, 128.1, 127.7, 127.2, 119.5, 89.8, 84.5, 79.0, 72.9, 71.8, 69.7, 57.0, 35.5, 18.3, 18.2.

Copies of ^{13}C NMR spectra of Compounds 7, 8, 11, 14, 16, 19, 20, 21, 22, 26, 27, 28, 29, 30, 31, 32, 34 and 35, copies of selected NOE spectra of compound 20, and copies of ^{31}P NMR spectra of compounds 9 and 23.

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B1061

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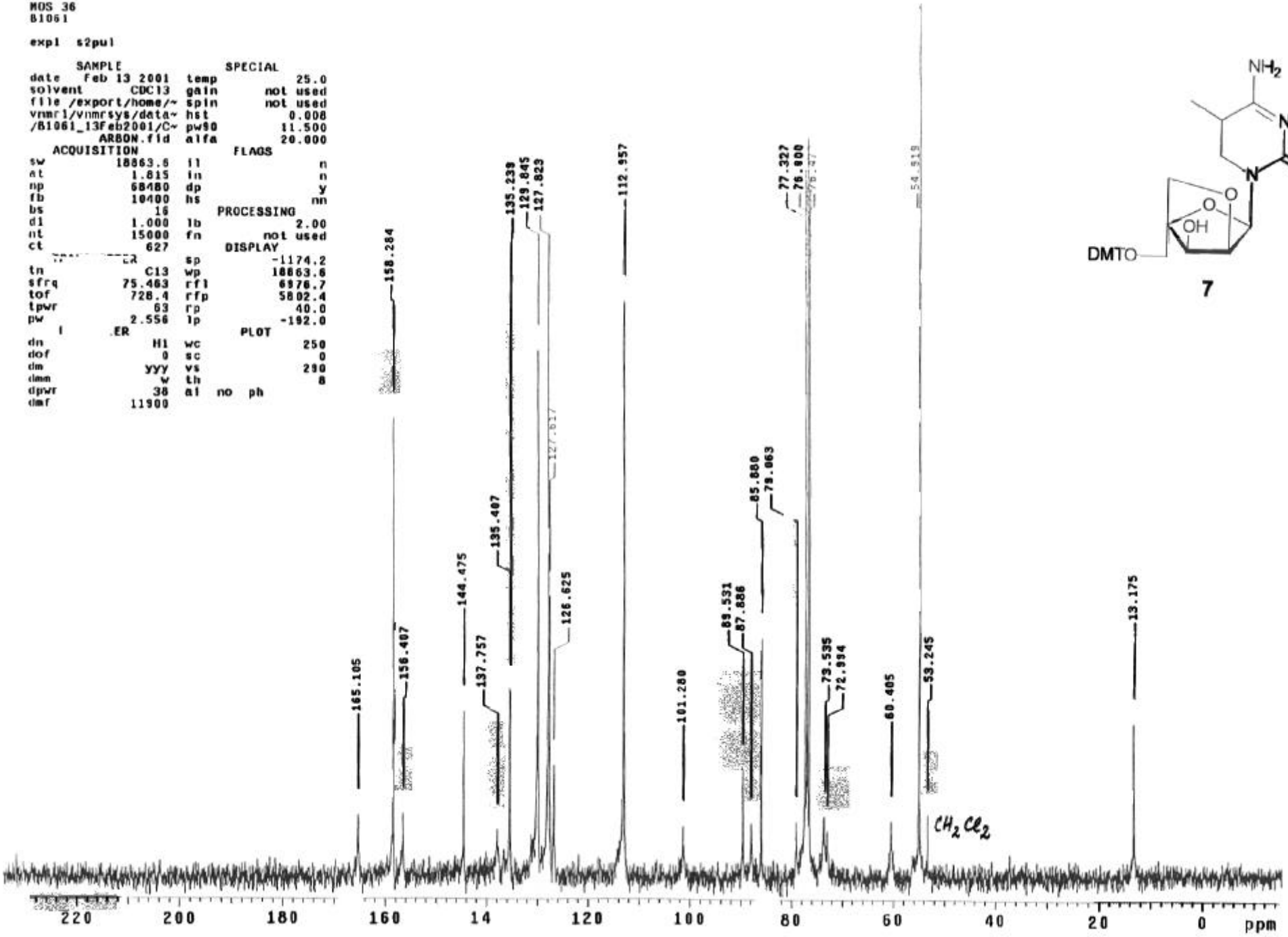

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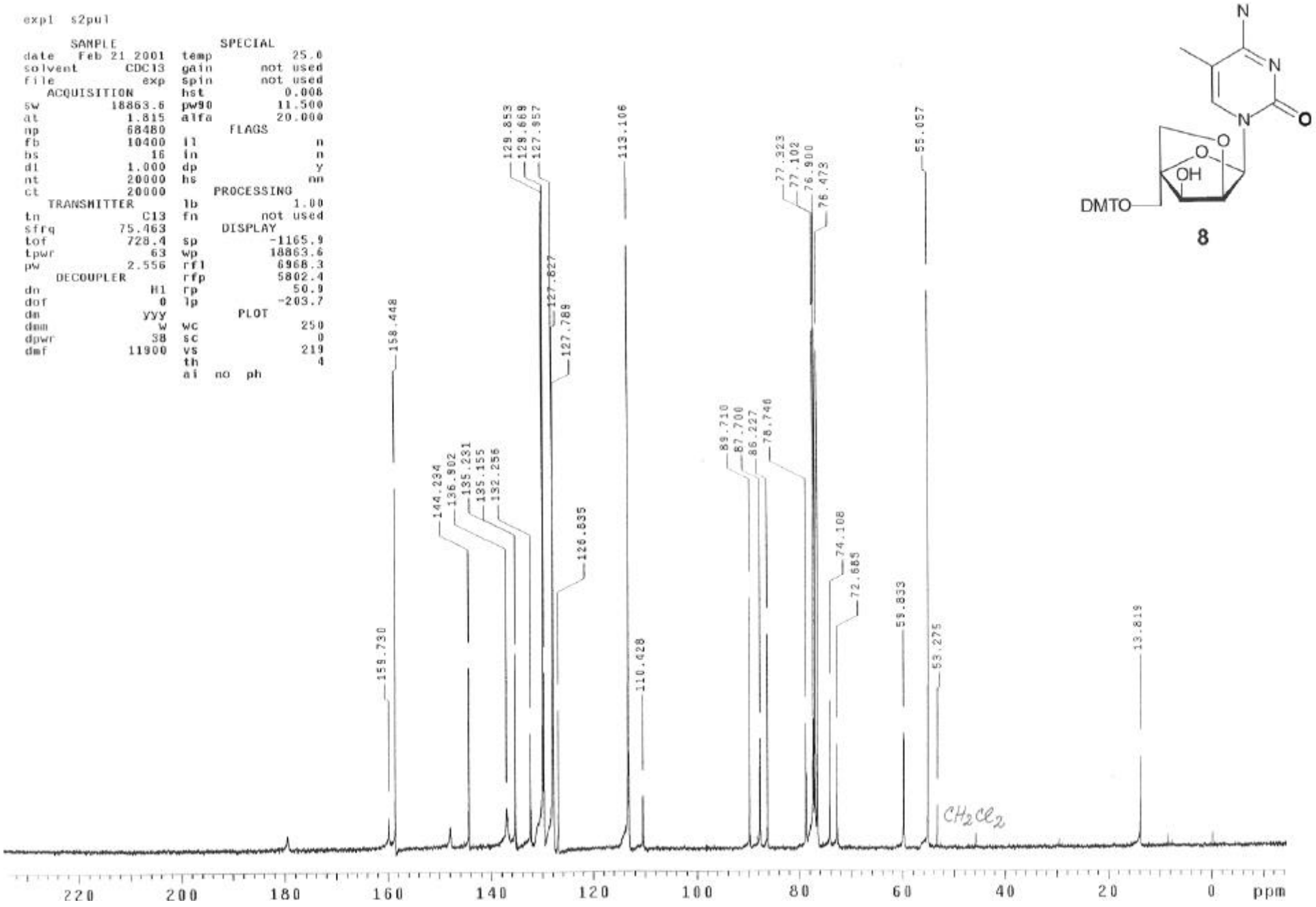

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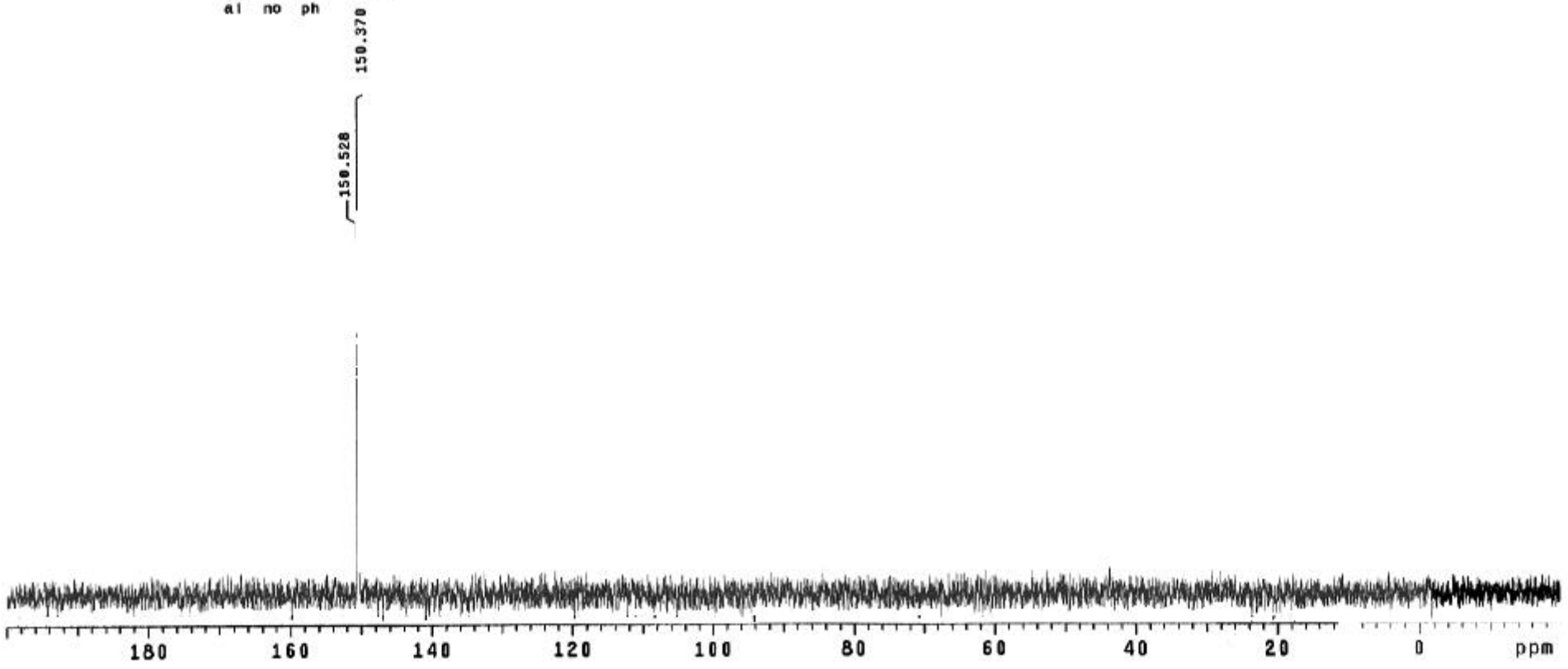

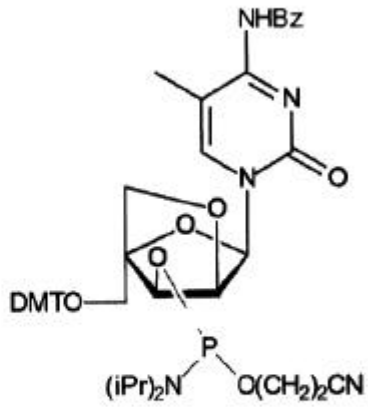

9

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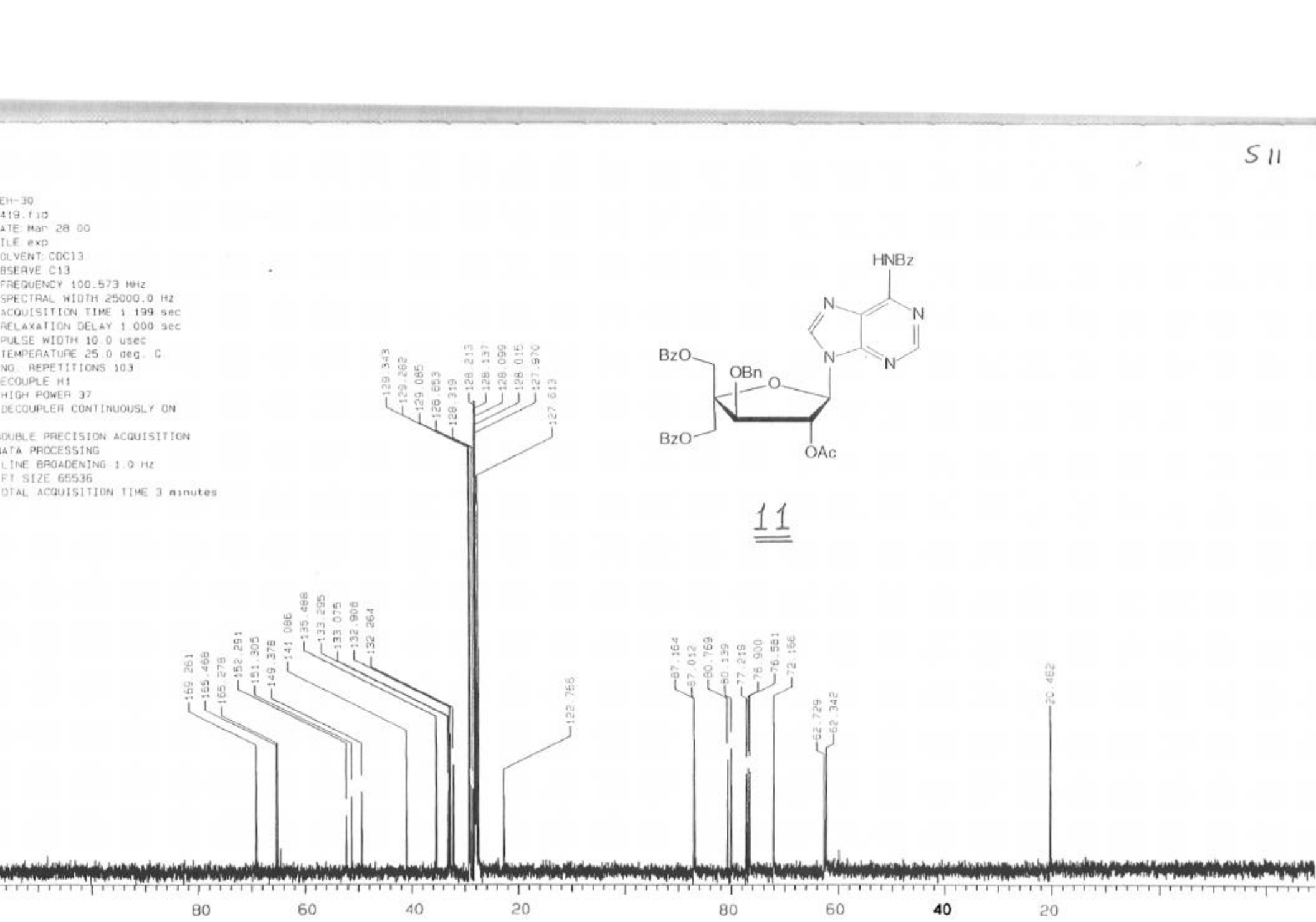

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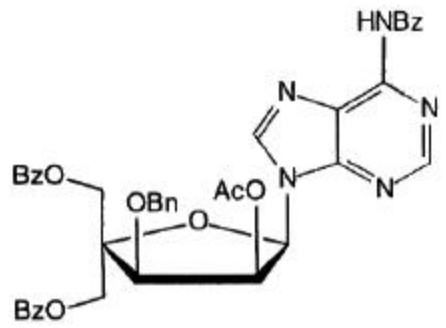

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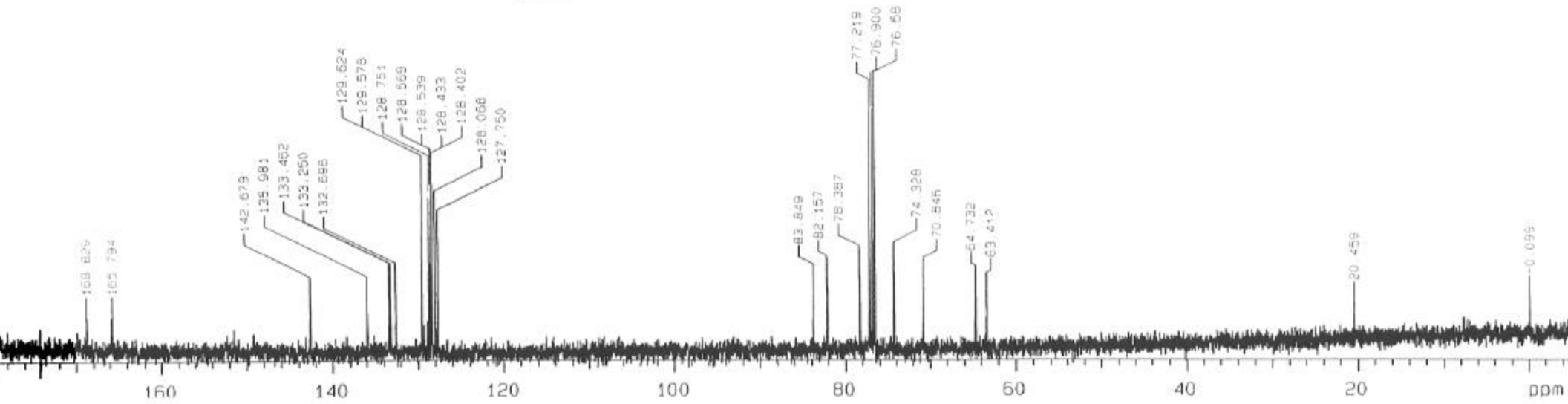

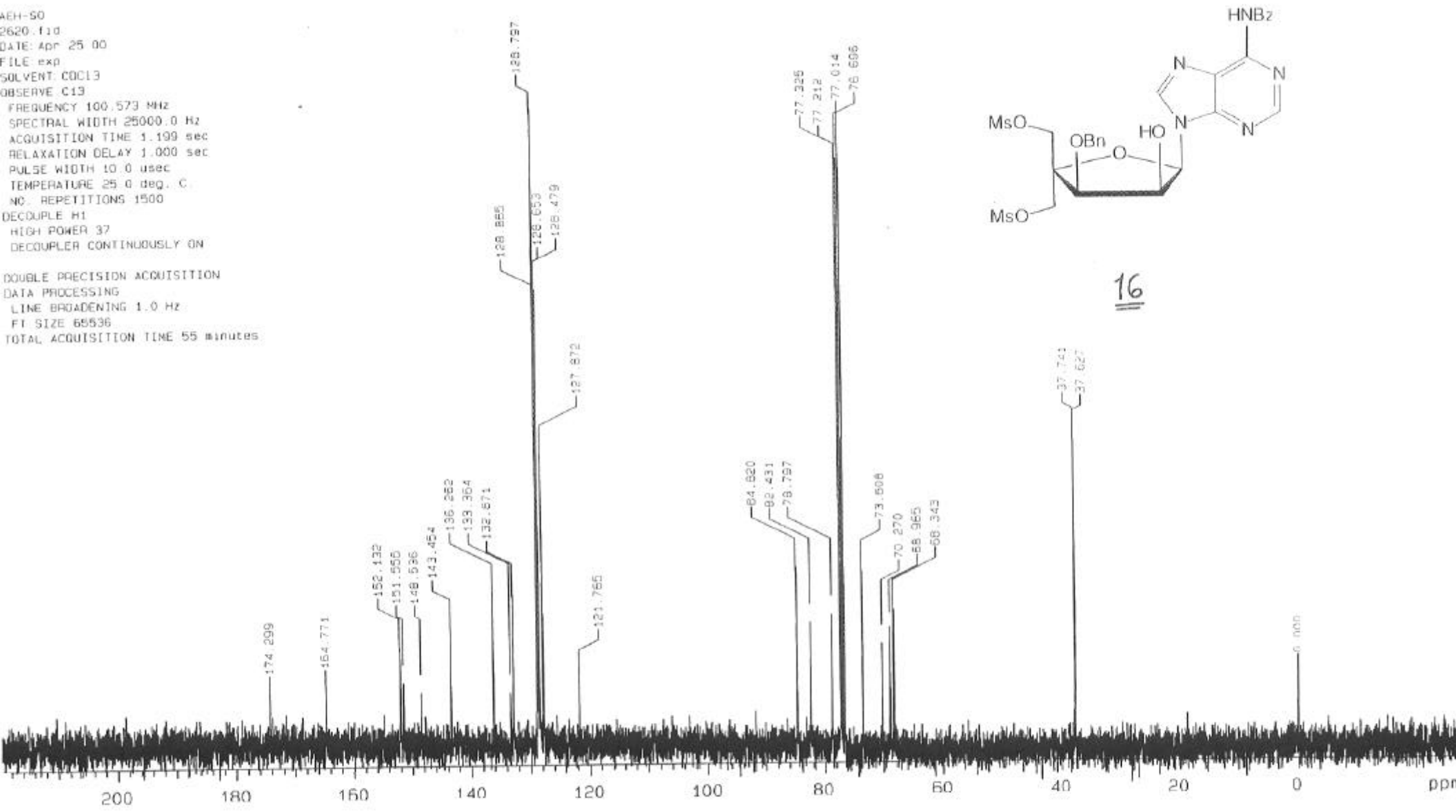
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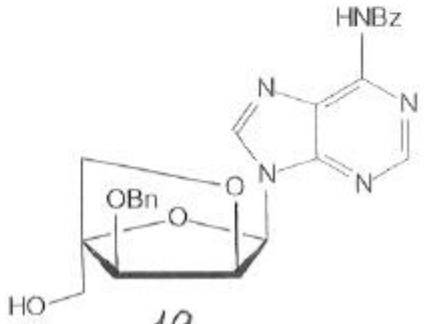

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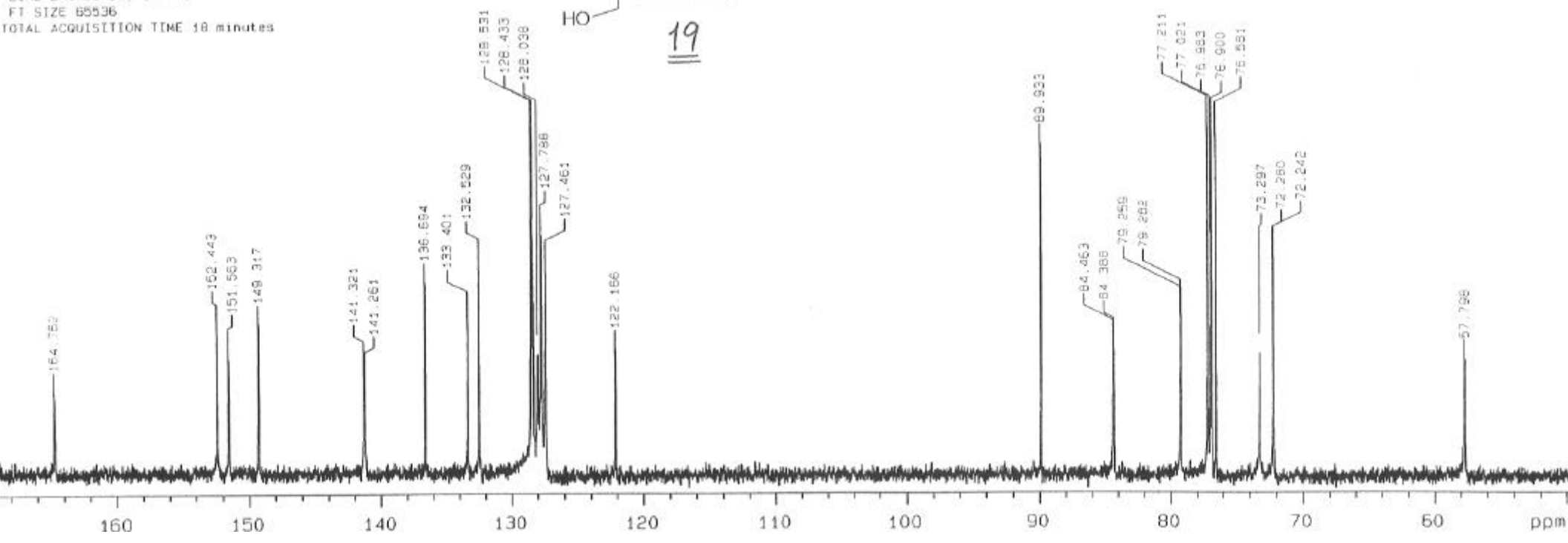

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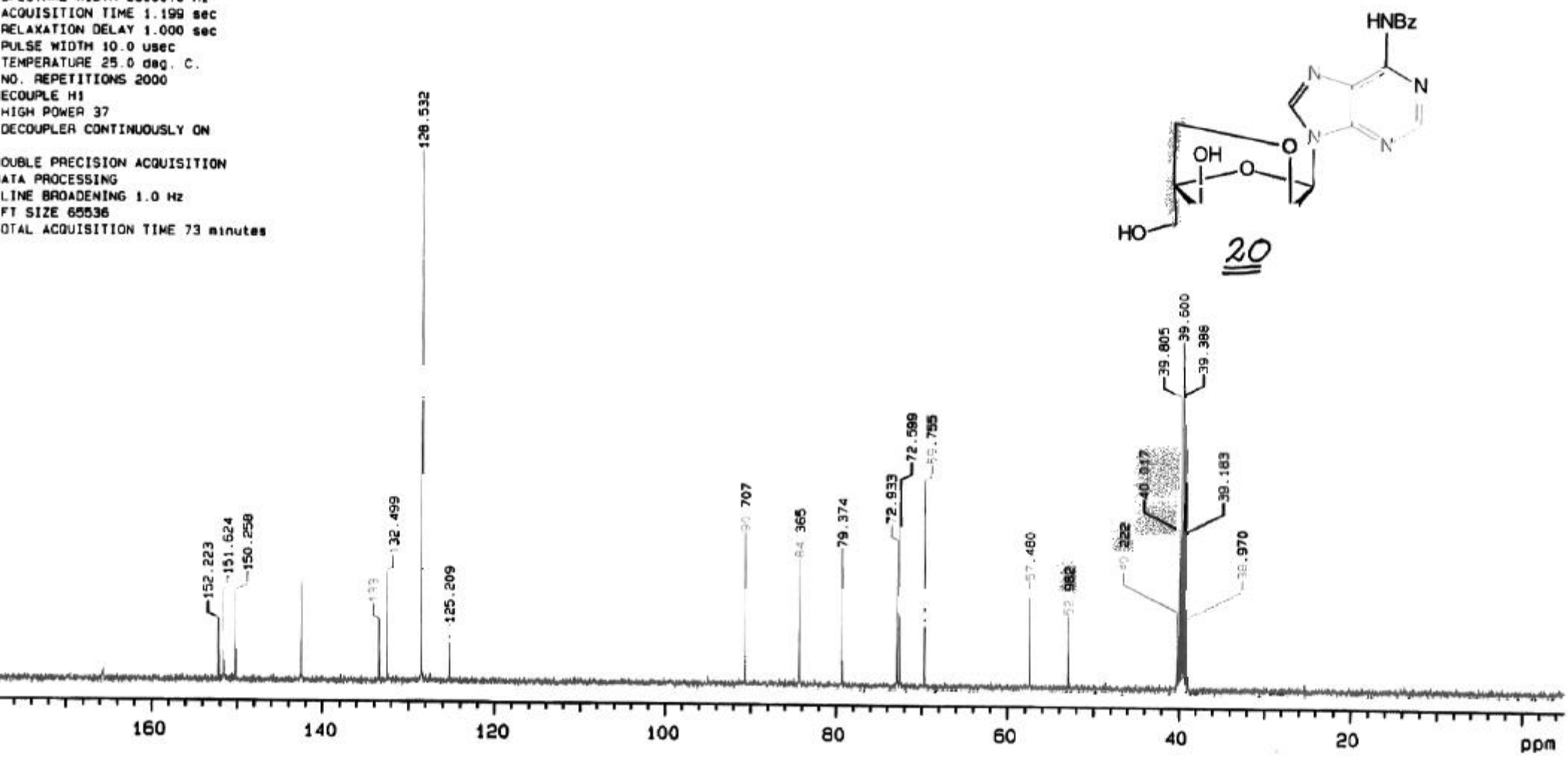
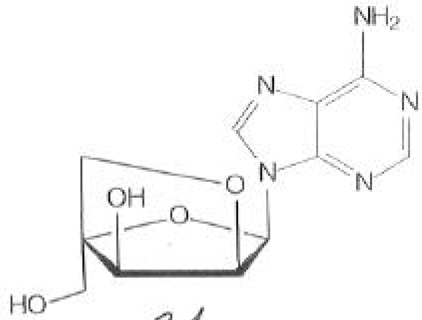
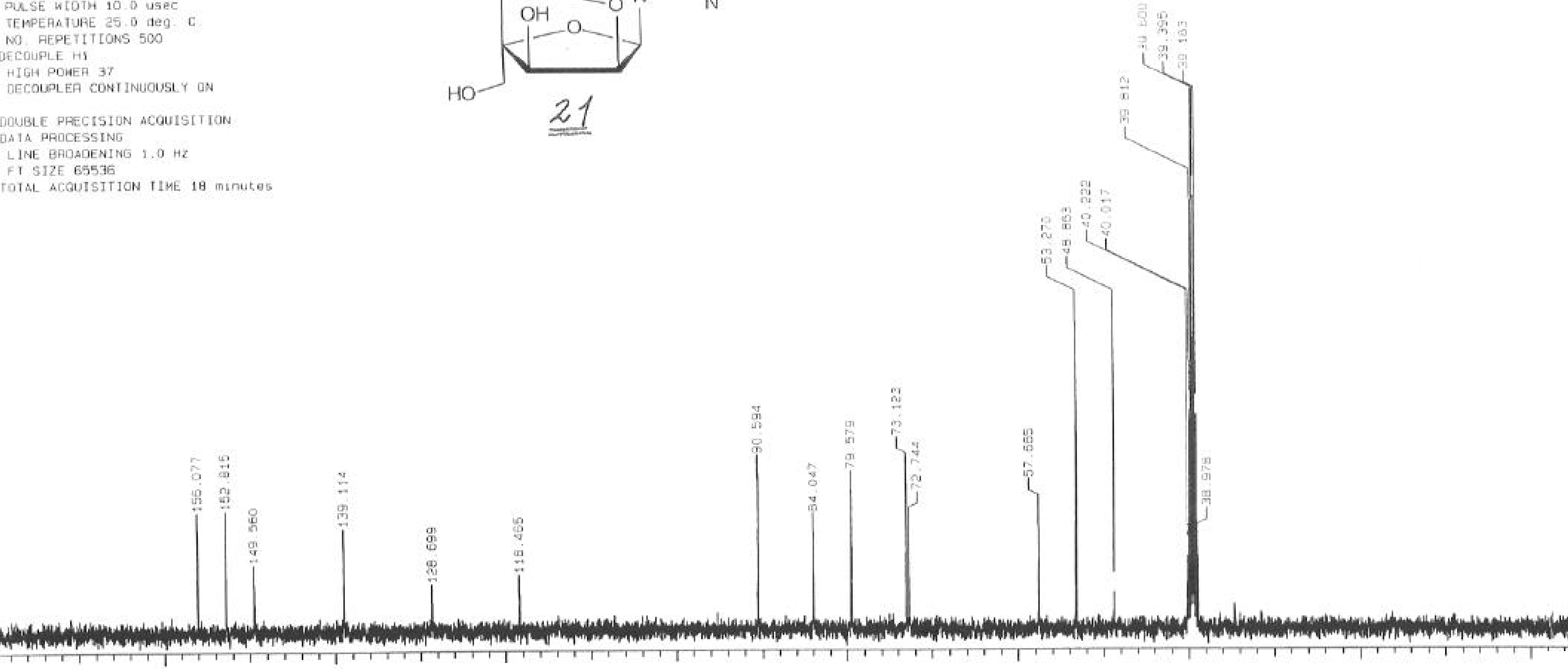
OTAL ACQUISITION TIME 73 minutes



EH 58
017.fid
ATE: Jun 16 00
ILE: exp
OLVENT: DMSO
BSERVE C13
FREQUENCY 100.574 MHz
SPECTRAL WIDTH 25000.0 Hz
ACQUISITION TIME 1.199 sec
RELAXATION DELAY 1.000 sec
PULSE WIDTH 10.0 usec
TEMPERATURE 25.0 deg. C
NO. REPETITIONS 500
ECOUPLE H1
HIGH POWER 37
DECOUPLER CONTINUOUSLY ON
DOUBLE PRECISION ACQUISITION
DATA PROCESSING
LINE BROADENING 1.0 Hz
FT SIZE 65536
TOTAL ACQUISITION TIME 18 minutes



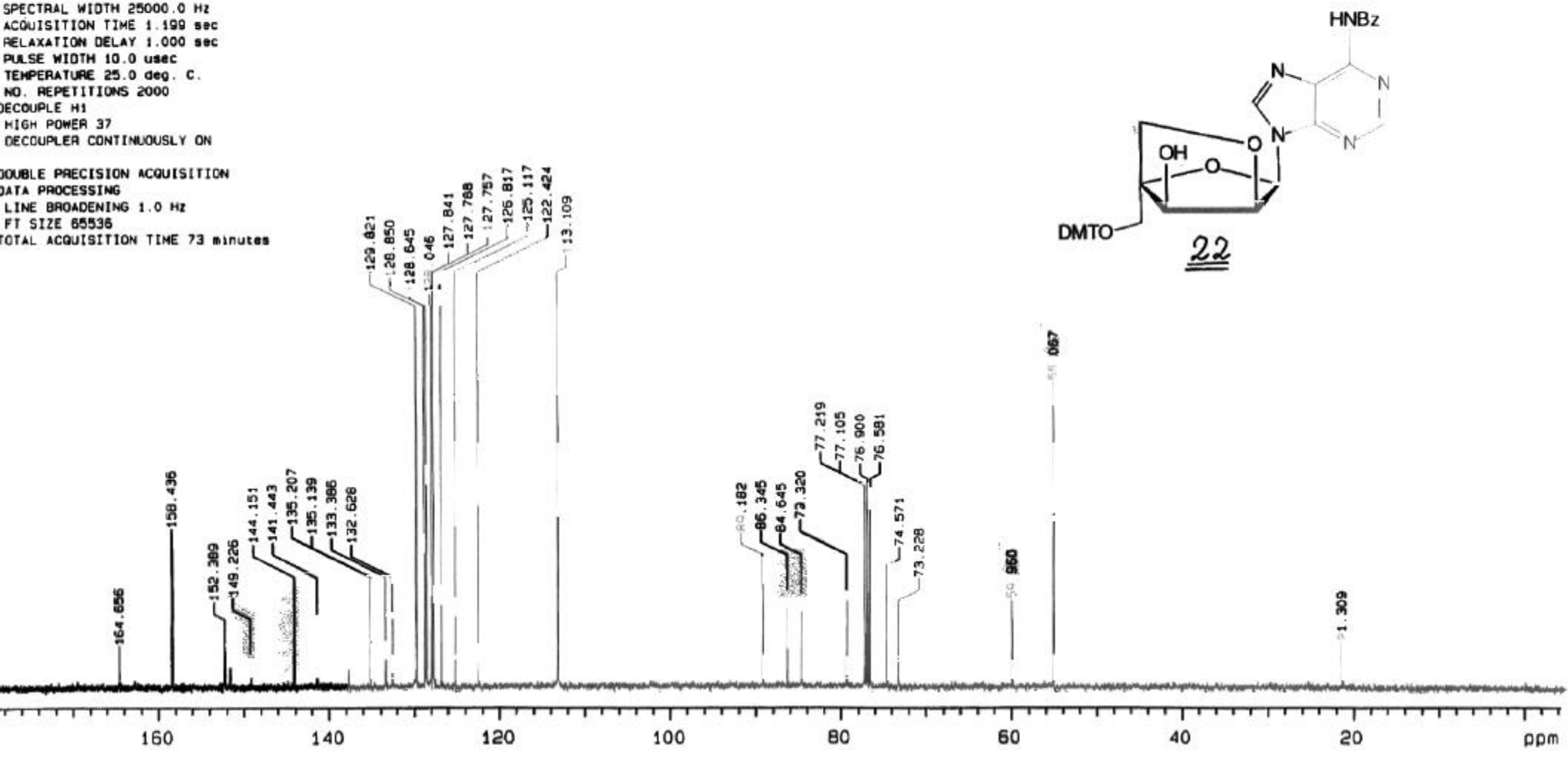
21



/

S6
EH-5b
993.fid
ATE: Jun 14 00
ILE: exp
OLVENT: CDCl3
BSERVE C13
FREQUENCY 100.573 MHz
SPECTRAL WIDTH 25000.0 Hz
ACQUISITION TIME 1.199 sec
RELAXATION DELAY 1.000 sec
PULSE WIDTH 10.0 usec
TEMPERATURE 25.0 deg. C.
NO. REPETITIONS 2000
DECOUPLE H1
HIGH POWER 37
DECOUPLER CONTINUOUSLY ON

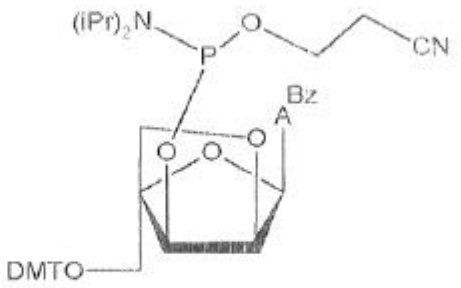
OUBLE PRECISION ACQUISITION
ATA PROCESSING
LINE BROADENING 1.0 Hz
FT SIZE 65536
OTAL ACQUISITION TIME 73 minutes



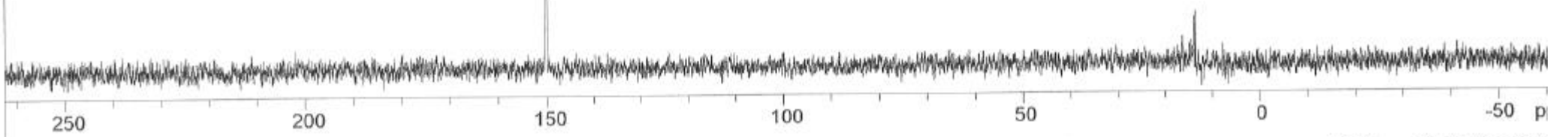
22

Interpolated Peak Listing

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	22385	35848K	98.67	18247.35	150.195
2	22426	6486K	17.85	18222.45	149.990



23



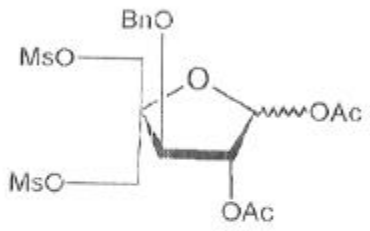
tbr f10O£ÎÇÓU§Ç□□àÇ□!

USER: DATE: Nov 23 2

F1: 121.491	F2: 300.092	SW1: 40000		OF1: 11910.0		PTSId: 65536
EX: s2pul		PW: 10.0 usec	PD: 1.0 sec	NA: 512	LB: 5.0	WinNuts - $TBR1123.J

Interpolated Peak Listing

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	14435	3591K	10.26	12793.46	169.528
2	14446	3981K	11.38	12790.19	169.485
3	14550	3059K	8.74	12760.46	169.091
4	23104	4246K	12.14	10311.73	136.642
5	23129	3075K	8.79	10304.42	136.546
6	25208	17455K	49.89	9709.33	128.660
7	25221	14648K	41.87	9705.49	128.609
8	25267	10452K	29.86	9692.53	128.437
9	25307	9013K	25.76	9680.99	128.284
10	25367	27944K	79.87	9663.72	128.055
11	32903	4663K	13.33	7506.24	99.466
12	34793	5194K	14.85	6965.17	92.297
13	36321	4874K	13.93	6527.74	86.500
14	37287	6294K	17.99	6251.20	82.836
15	37646	5492K	15.70	6148.39	81.473
16	37731	5077K	14.50	6124.03	81.150
17	38096	3636K	10.39	6019.54	79.766
18	38714	32712K	93.50	5842.69	77.422
19	38826	33004K	94.34	5810.81	77.000
20	38938	32009K	91.49	5778.52	76.572
21	39006	6603K	18.87	5759.17	76.316
22	39697	6683K	19.10	5561.45	73.696
23	39946	5285K	15.11	5490.12	72.750
24	40984	7214K	20.62	5192.98	68.813
25	41041	4842K	13.84	5176.55	68.595
26	41231	7609K	21.75	5122.30	67.876
27	41534	4545K	12.99	5035.45	66.725
28	49124	7281K	20.81	2862.55	37.932
29	49171	8723K	24.93	2849.13	37.754
30	49180	7462K	21.33	2844.26	37.690
31	49234	6263K	17.90	2831.01	37.514
32	53578	5094K	14.56	1587.41	21.035
33	53603	5175K	14.79	1580.22	20.940
34	53658	4117K	11.77	1564.41	20.730
35	53750	4649K	13.29	1538.25	20.384
36	58860	5021K	14.35	75.13	0.996



26

gga gw7

F1: 75.465	F2: 300.091	SW1: 18762		OF1: 7545.0		PTS1d: 65536
EX: s2pul		PW: 8.8 usec	PD: 1.2 sec	NA: 1024	LB: 1.0	

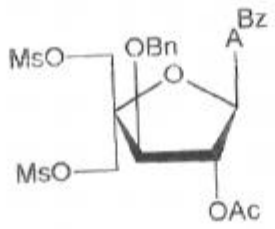
USER: DATE: Sep 15 2000

WinNuts - $gga_gw7.fid

S20

Interpolated Peak Listing

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	14439	10977K	19.96	12789.50	169.476
2	15739	6558K	11.93	12417.40	164.545
3	18811	16442K	29.90	11537.86	152.890
4	19154	4771K	8.68	11439.77	151.590
5	19664	7352K	13.37	11293.73	149.655
6	21888	15553K	28.28	10656.87	141.216
7	23354	11553K	21.01	10237.41	135.658
8	23940	5945K	10.81	10069.39	133.431
9	24103	16082K	29.25	10022.75	132.813
10	25153	42747K	77.74	9722.28	128.831
11	25172	55563K	101.04	9716.82	128.759
12	25196	29668K	53.95	9710.03	128.669
13	25211	6663K	12.12	9705.70	128.612
14	25247	53183K	96.71	9695.27	128.474
15	25270	3244K	5.90	9688.67	128.386
16	25370	3271K	5.95	9660.02	128.007
17	25422	37564K	68.31	9645.22	127.810
18	26701	3665K	6.66	9278.98	122.957
19	36103	14564K	26.49	6587.53	87.292
20	36450	16359K	29.75	6487.94	85.973
21	37696	19121K	34.77	6131.27	81.246
22	38184	18106K	32.93	5991.77	79.398
23	38703	19978K	36.33	5842.96	77.426
24	38761	7763K	14.12	5826.47	77.207
25	38814	21256K	38.65	5811.26	77.006
26	38926	20389K	37.08	5779.36	76.583
27	39792	17062K	31.03	5531.37	73.297
28	41358	12497K	22.73	5082.98	67.355
29	41852	15340K	27.90	4941.43	65.480
30	49184	27432K	49.88	2842.51	37.667
31	49202	25747K	46.82	2837.39	37.599
32	53667	15764K	28.67	1559.04	20.659



27

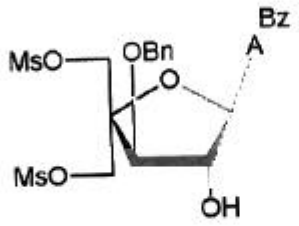
tbr a10 USER: DATE: Dec 22 200

F1: 75.465	F2: 300.091	SW1: 18762		OF1: 7542.2		PTS1d: 65536
EX: s2pul		PW: 8.8 usec	PD: 1.2 sec	NA: 1024	LB: 1.0	WinNuts - $tbr1222.a10

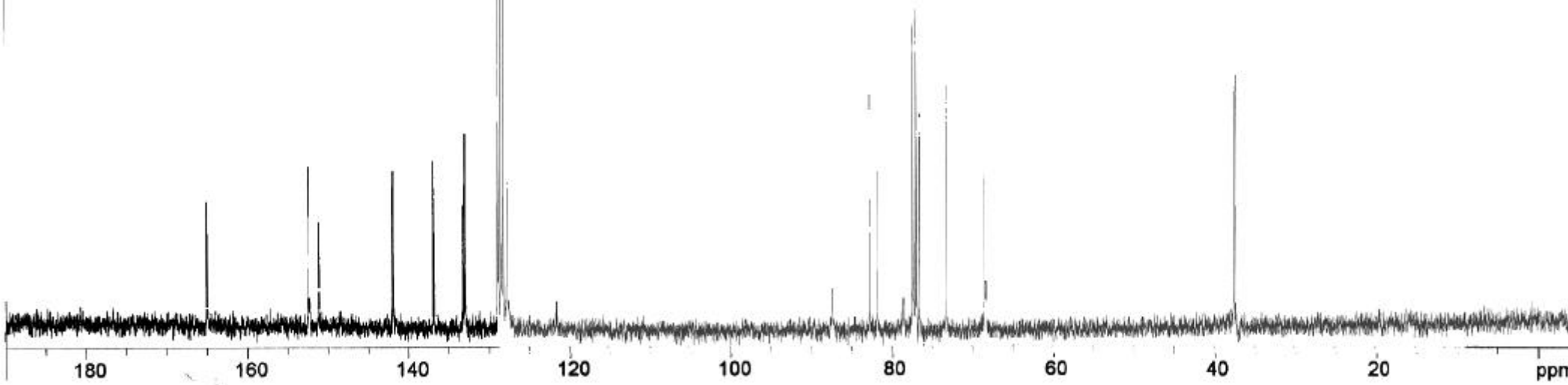
S21

Interpolated Peak Listing

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	15611	10394K	13.22	12455.07	165.044
2	18938	13280K	16.89	11502.63	152.423
3	19272	8660K	11.01	11407.01	151.156
4	19977	11558K	14.70	11204.99	148.479
5	21694	12957K	16.48	10713.48	141.966
6	23028	13960K	17.75	10331.58	136.905
7	23989	10038K	12.77	10056.39	133.259
8	24048	15909K	20.23	10039.47	133.035
9	25125	42218K	53.69	9731.18	128.949
10	25211	62125K	79.01	9706.67	128.625
11	25251	4129K	5.25	9695.16	128.472
12	25294	76972K	97.89	9682.77	128.308
13	25438	38355K	48.78	9641.81	127.765
14	27037	7646K	9.72	9183.84	121.697
15	36074	16017K	20.37	6596.61	87.413
16	37287	23378K	29.73	6249.40	82.812
17	37548	19112K	24.31	6174.63	81.821
18	38398	15828K	20.13	5931.54	78.600
19	38707	25160K	32.00	5843.03	77.427
20	38765	11336K	14.42	5826.23	77.204
21	38819	26473K	33.67	5810.81	77.000
22	38930	25940K	32.99	5779.02	76.579
23	39813	20079K	25.54	5526.35	73.231
24	41060	12675K	16.12	5169.36	68.500
25	41107	14716K	18.72	5155.77	68.320
26	49191	33423K	42.51	2841.52	37.653
27	49257	30003K	38.16	2822.53	37.402



28



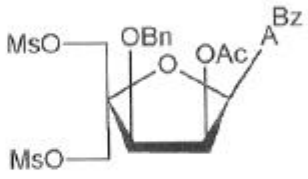
tbr 112 USER: DATE: Dec 22 200

F1: 75.465	F2: 300.091	SW1: 18762		OF1: 7543.2		PTS1d: 65536
EX: s2pul		PW: 8.8 usec	PD: 1.2 sec	NA: 1024	LB: 1.0	

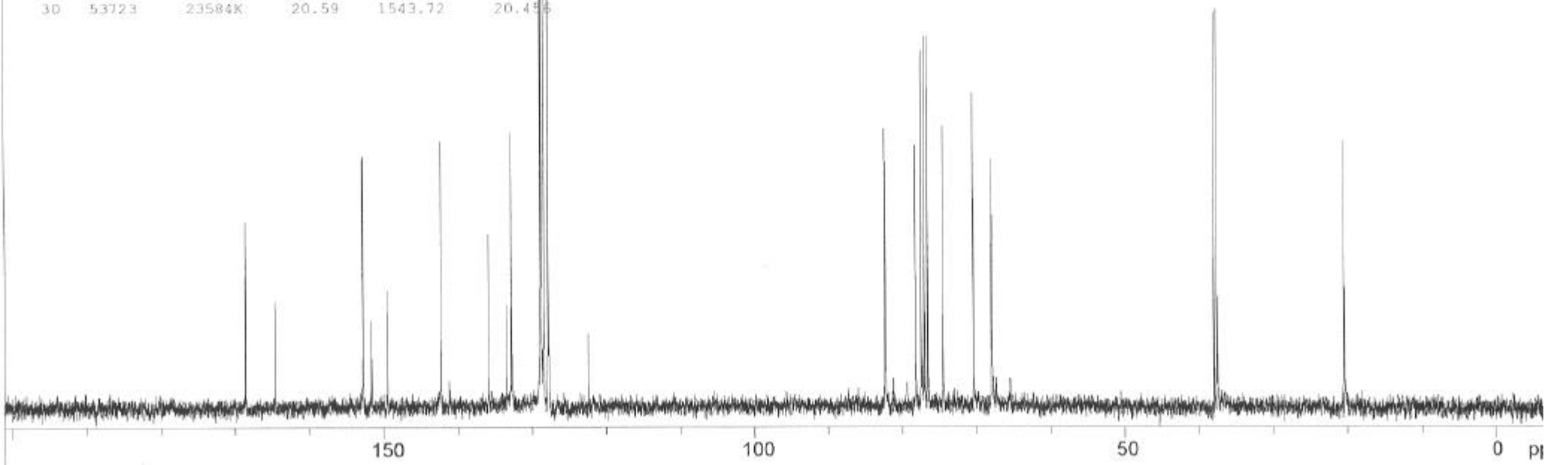
S 22

Interpolated Peak Listing

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	14668	16557K	14.45	12724.67	168.617
2	15726	9131K	7.97	12421.60	164.601
3	18836	21781K	19.01	11531.40	152.804
4	19140	7387K	6.45	11444.22	151.649
5	19708	10007K	8.73	11281.61	149.494
6	21595	23039K	20.11	10741.61	142.339
7	23289	15039K	13.13	10256.43	135.910
8	23951	8905K	7.77	10066.94	133.399
9	24105	23905K	20.87	10022.97	132.816
10	25160	114318K	99.79	9720.94	128.814
11	25177	41962K	36.63	9715.94	128.747
12	25197	7640K	6.67	9710.22	128.672
13	25247	8193K	7.15	9696.07	128.484
14	25270	69166K	60.37	9689.31	128.395
15	25416	55951K	48.84	9647.62	127.842
16	26846	6357K	5.55	9238.30	122.410
17	37399	24499K	21.39	6217.00	82.382
18	37434	21247K	18.55	6207.13	82.252
19	38494	22889K	19.98	5903.53	78.229
20	38704	31143K	27.18	5843.48	77.436
21	38762	15882K	13.86	5826.96	77.214
22	38816	32373K	28.26	5811.32	77.007
23	38927	32403K	28.28	5779.55	76.586
24	39462	24486K	21.37	5626.38	74.556
25	40547	27501K	24.01	5315.77	70.440
26	41187	21389K	18.67	5132.75	68.015
27	41226	16607K	14.50	5121.40	67.865
28	49088	34209K	29.86	2870.79	38.041
29	49180	34629K	30.23	2844.45	37.692
30	53723	23584K	20.59	1543.72	20.456



29

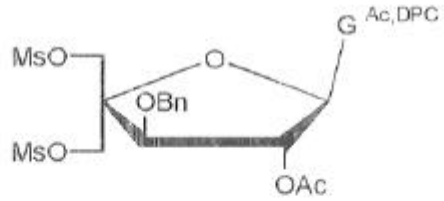


tbr 122 USER: DATE: Jan 9 2

F1: 75.465	F2: 300.091	SW1: 18762		OF1: 7542.9		PTS1d: 65536
EX: s2pul		PW: 8.8 usec	PD: 1.2 sec	NA: 1024	LB: 1.0	WinNuts - $tbr0109.1

Interpolated Peak Listing

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	7577	3873K	6.14	14756.53	195.541
2	14471	13525K	21.45	12782.86	169.388
3	17913	6797K	10.78	11797.49	156.330
4	18430	8181K	12.98	11649.41	154.368
5	18985	7929K	12.58	11490.55	152.263
6	19532	4056K	6.43	11334.14	150.190
7	21705	14497K	22.99	10711.95	141.916
8	23336	13005K	20.63	10244.97	135.758
9	24984	7353K	11.66	9773.07	129.504
10	24993	4567K	7.24	9770.47	129.470
11	25064	27884K	44.22	9750.25	129.202
12	25180	55075K	87.35	9717.06	128.762
13	25212	26444K	41.94	9707.89	128.641
14	25263	54260K	86.06	9693.26	128.447
15	25278	7797K	12.37	9688.90	128.389
16	25531	4575K	7.26	9616.57	127.431
17	25595	4700K	7.45	9598.18	127.187
18	25639	4429K	7.02	9585.74	127.022
19	27258	4162K	6.60	9122.14	120.879
20	32768	-4117100	-6.53	7544.66	99.975
21	36027	12614K	20.01	6611.67	87.612
22	36448	12511K	19.84	6491.18	86.016
23	37709	18854K	29.90	6130.26	81.233
24	38250	13495K	21.40	5975.44	79.182
25	38713	55729K	88.39	5842.66	77.422
26	38825	59355K	94.14	5810.81	77.000
27	38937	58681K	93.07	5778.56	76.573
28	39788	16152K	25.62	5534.95	73.344
29	41370	10748K	17.05	5082.00	67.342
30	41857	14969K	23.74	4942.62	65.495
31	49181	22063K	34.99	2845.85	37.711
32	49197	27481K	43.59	2841.46	37.653
33	49222	3812K	6.05	2834.09	37.555
34	52509	15012K	23.81	1893.25	25.088
35	53683	16001K	25.38	1557.20	20.635
36	58860	8083K	12.82	74.84	0.992



S23

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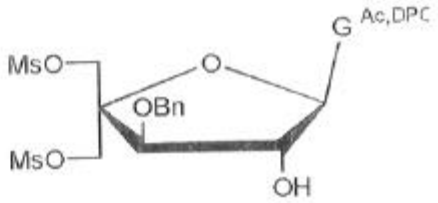
gga 136					USER:	DATE: Sep 25 20
F1: 75.465	F2: 300.091	SW1: 18762		OF1: 7544.7		PTS1d: 65536
EX: s2pul		PW: 8.8 usec	PD: 1.2 sec	NA: 1024	LB: 1.0	WinNuts - $GGA0925.1

Interpolated Peak Listing

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	14585	1865K	4.50	12758.12	169.060
2	18015	7375K	18.12	11776.21	156.048
3	18543	4983K	12.24	11625.24	154.048
4	19272	6387K	15.69	11416.42	151.281
5	19438	4302K	10.57	11368.90	150.651
6	21597	6168K	15.16	10750.77	142.460
7	21797	2765K	6.80	10693.41	141.700
8	22988	9466K	23.26	10352.61	137.184
9	24823	1905K	4.68	9827.12	130.221
10	24908	1809K	4.45	9802.93	129.900
11	24948	1945K	4.78	9791.37	129.747
12	25049	16240K	39.91	9762.39	129.363
13	25184	3126K	7.68	9723.83	128.852
14	25229	40428K	99.35	9710.95	128.681
15	25272	3204K	7.87	9698.67	128.519
16	25332	17941K	44.09	9681.59	128.292
17	25353	39163K	96.24	9675.62	128.213
18	25548	2471K	6.07	9619.77	127.473
19	25590	2431K	5.97	9607.79	127.314
20	25618	2431K	5.97	9599.54	127.205
21	25660	1841K	4.53	9587.73	127.049
22	26136	-1009222	-4.45	9451.47	125.243
23	27213	4838K	11.89	9142.89	121.154
24	35613	6661K	16.37	6738.11	89.288
25	37195	9980K	24.52	6285.28	83.287
26	37358	8862K	21.78	6238.71	82.670
27	38134	4327K	10.63	6016.35	79.724
28	38703	11984K	29.45	5853.46	77.565
29	38761	10394K	25.54	5836.95	77.346
30	38814	12880K	31.65	5821.78	77.145
31	38926	12734K	31.29	5789.87	76.722
32	39819	9910K	24.35	5534.13	73.334
33	41153	11862K	29.15	5152.16	68.272
34	49218	21395K	52.58	2843.23	37.676
35	49276	18970K	46.62	2826.66	37.457
36	49301	2163K	5.32	2819.45	37.361
37	49368	1815K	4.46	2800.40	37.108
38	52564	12035K	29.58	1885.43	24.984



S 24

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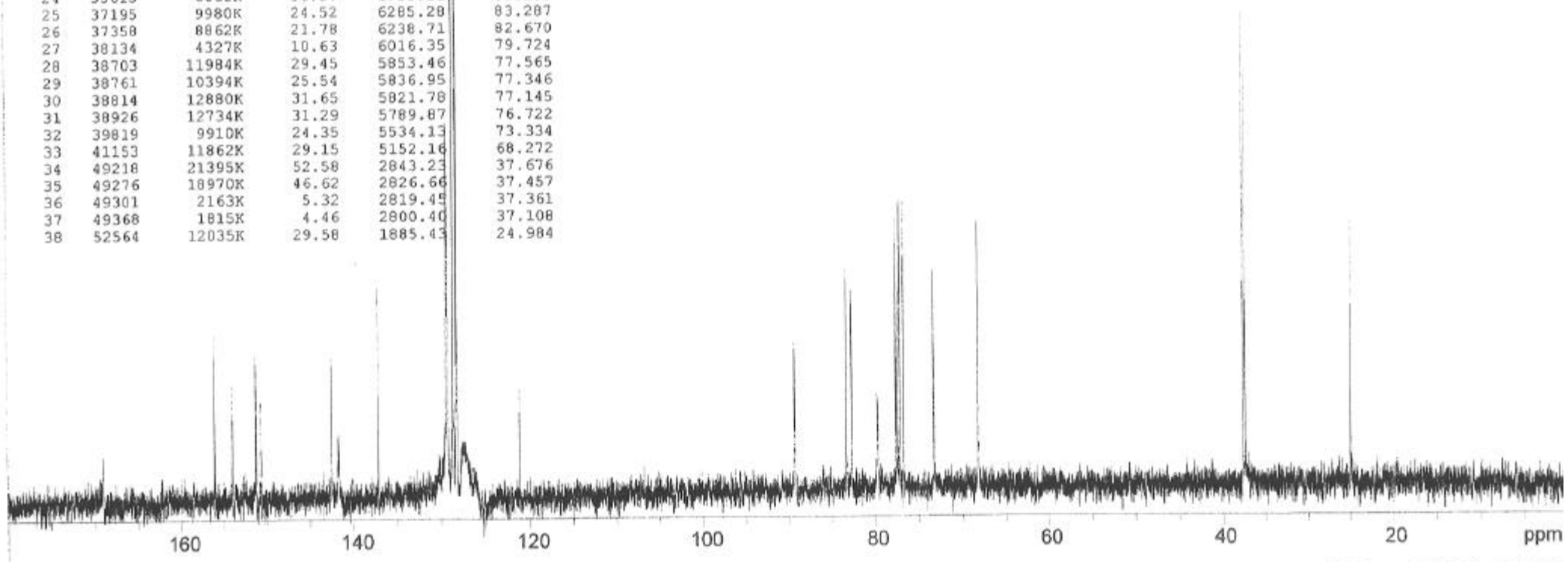
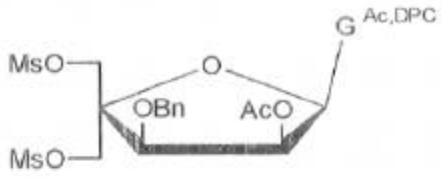


gga 017						USER:	DATE: Jan 24 2001
F1: 75.465	F2: 300.091	SW1: 18762		OF1: 7552.7		PTS1d: 65536	
EX: s2pul		PW: 8.8 usec	PD: 1.2 sec	NA: 256	LB: 1.0		WinNuts - $gga0124.017

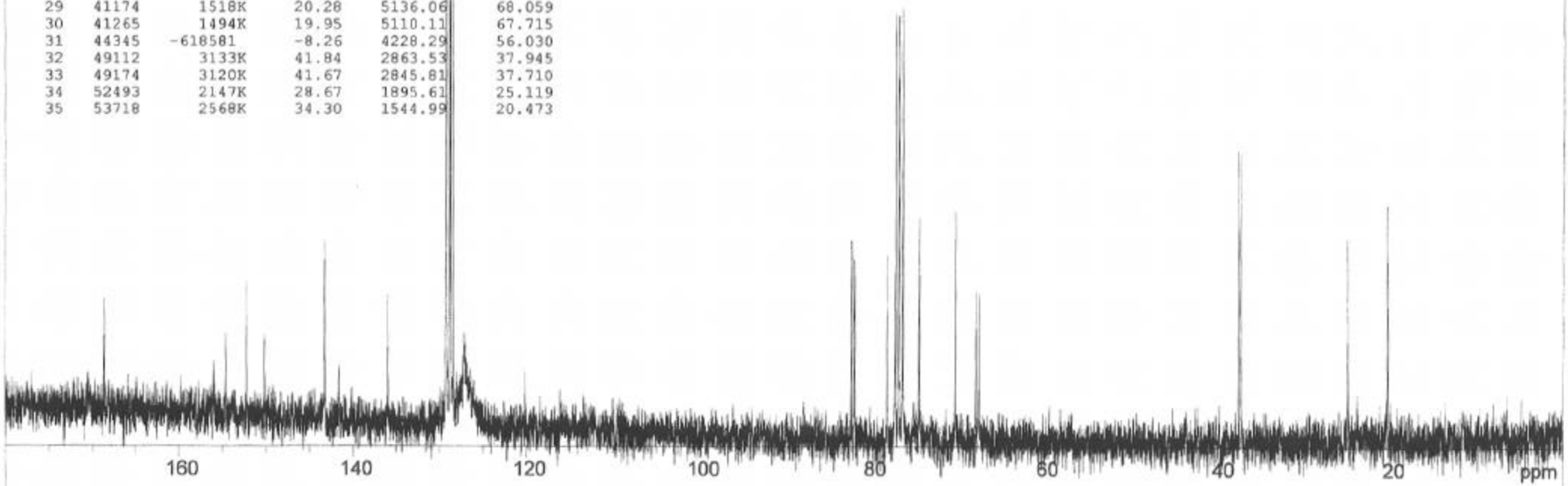
Interpolated Peak Listing

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	14651	1468K	19.61	12729.36	168.679
2	17974	741147	9.90	11777.89	156.071
3	18337	1039K	13.88	11673.95	154.693
4	18972	1625K	21.70	11492.18	152.285
5	19524	1034K	13.82	11334.16	150.191
6	21361	2114K	28.23	10808.35	143.223
7	21787	698890	9.33	10686.35	141.607
8	23274	1485K	19.83	10260.57	135.964
9	25063	5083K	67.88	9748.56	129.180
10	25167	6930K	92.56	9718.63	128.783
11	25186	4832K	64.53	9713.20	128.711
12	25270	6935K	92.63	9689.35	128.395
13	25561	801122	10.70	9605.86	127.289
14	25593	1057K	14.12	9596.68	127.167
15	25643	1025K	13.68	9582.50	126.979
16	25661	756524	10.10	9577.37	126.911
17	25745	614756	8.21	9553.35	126.593
18	25800	628269	8.39	9537.40	126.382
19	27434	636687	8.50	9069.73	120.184
20	37379	2100K	28.04	6222.52	82.456
21	37458	1872K	25.00	6200.13	82.159
22	38453	1955K	26.12	5915.21	78.383
23	38707	4701K	62.79	5842.36	77.418
24	38818	4665K	62.30	5810.73	76.999
25	38930	4707K	62.87	5778.47	76.571
26	39425	2368K	31.63	5636.98	74.697
27	40107	-624486	-8.34	5441.72	72.109
28	40522	2443K	32.63	5322.76	70.533
29	41174	1518K	20.28	5136.06	68.059
30	41265	1494K	19.95	5110.11	67.715
31	44345	-618581	-8.26	4228.29	56.030
32	49112	3133K	41.84	2863.53	37.945
33	49174	3120K	41.67	2845.81	37.710
34	52493	2147K	28.67	1895.61	25.119
35	53718	2568K	34.30	1544.99	20.473

S25



32



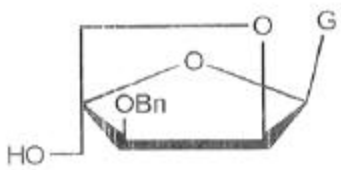
gga 003

USER: DATE: Nov 6 2000

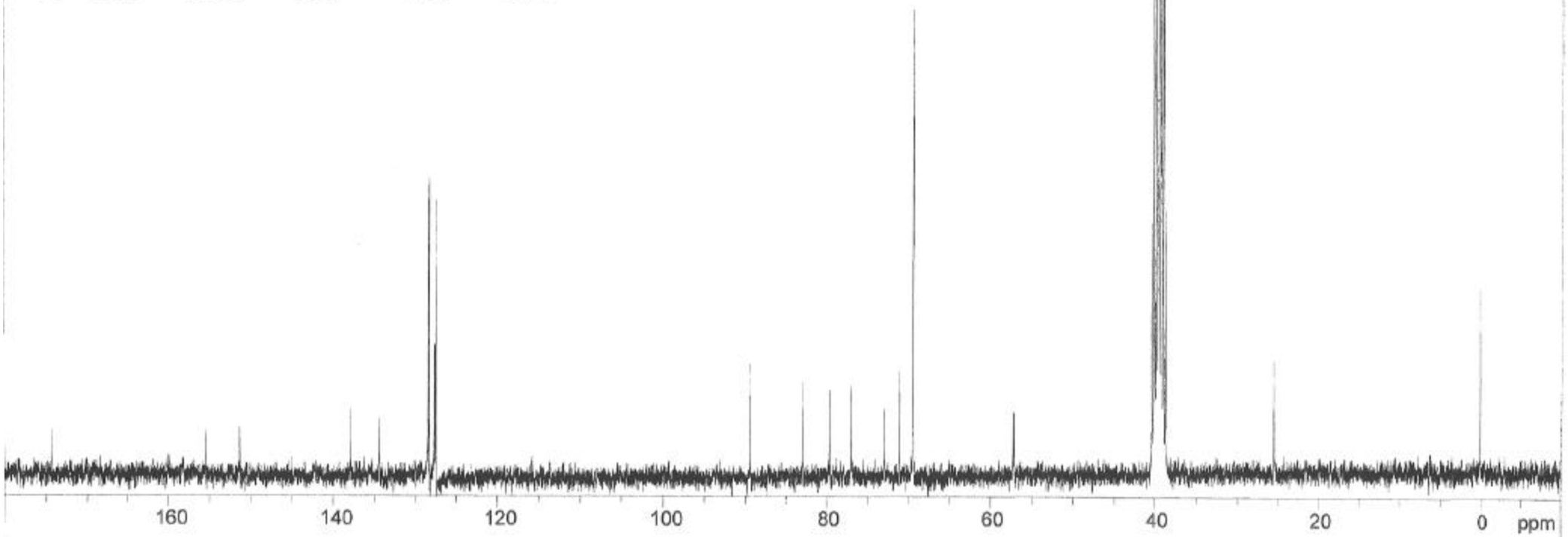
F1: 75.465	F2: 300.091	SW1: 18762		OF1: 7542.7		PTS1d: 65536
EX: s2pul		PW: 8.8 usec	PD: 1.2 sec	NA: 256	LB: 1.0	WinNuts - $gga1106.003

Interpolated Peak Listing

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	13042	5048K	2.37	13156.89	174.343
2	18010	5108K	2.39	11734.67	155.497
3	19104	5390K	2.53	11421.69	151.350
4	22655	7713K	3.61	10404.88	137.876
5	23595	6370K	2.98	10135.81	134.311
6	25183	34638K	16.23	9681.16	128.286
7	25367	15115K	7.08	9628.56	127.589
8	25407	32186K	15.08	9617.05	127.436
9	35429	13155K	6.16	6747.89	89.417
10	37141	10737K	5.03	6257.76	82.922
11	38022	9688K	4.54	6005.48	79.579
12	38737	10287K	4.82	5800.94	76.869
13	39763	7606K	3.56	5507.11	72.975
14	40276	12052K	5.65	5360.33	71.030
15	40707	53416K	25.03	5236.84	69.394
16	43935	7358K	3.45	4312.75	57.149
17	48367	28909K	13.55	3043.96	40.336
18	48441	88136K	41.30	3022.85	40.056
19	48514	180502K	84.58	3001.91	39.779
20	48587	211169K	98.95	2980.89	39.500
21	48661	179952K	84.33	2959.78	39.220
22	48734	90723K	42.51	2939.03	38.945
23	48807	30582K	14.33	2917.93	38.666
24	52292	13400K	6.28	1920.23	25.445
25	58978	22286K	10.44	6.18	0.082



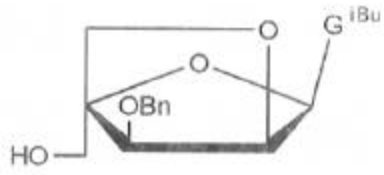
34



gga 011						USER: DATE: Dec 9 2000
F1: 75.465	F2: 300.092	SW1: 18762		OF1: 7509.8		PTS1d: 65536
EX: s2pul		PW: 8.8 usec	PD: 1.2 sec	NA: 8192	LB: 1.0	WinNuts - $gga1210.011

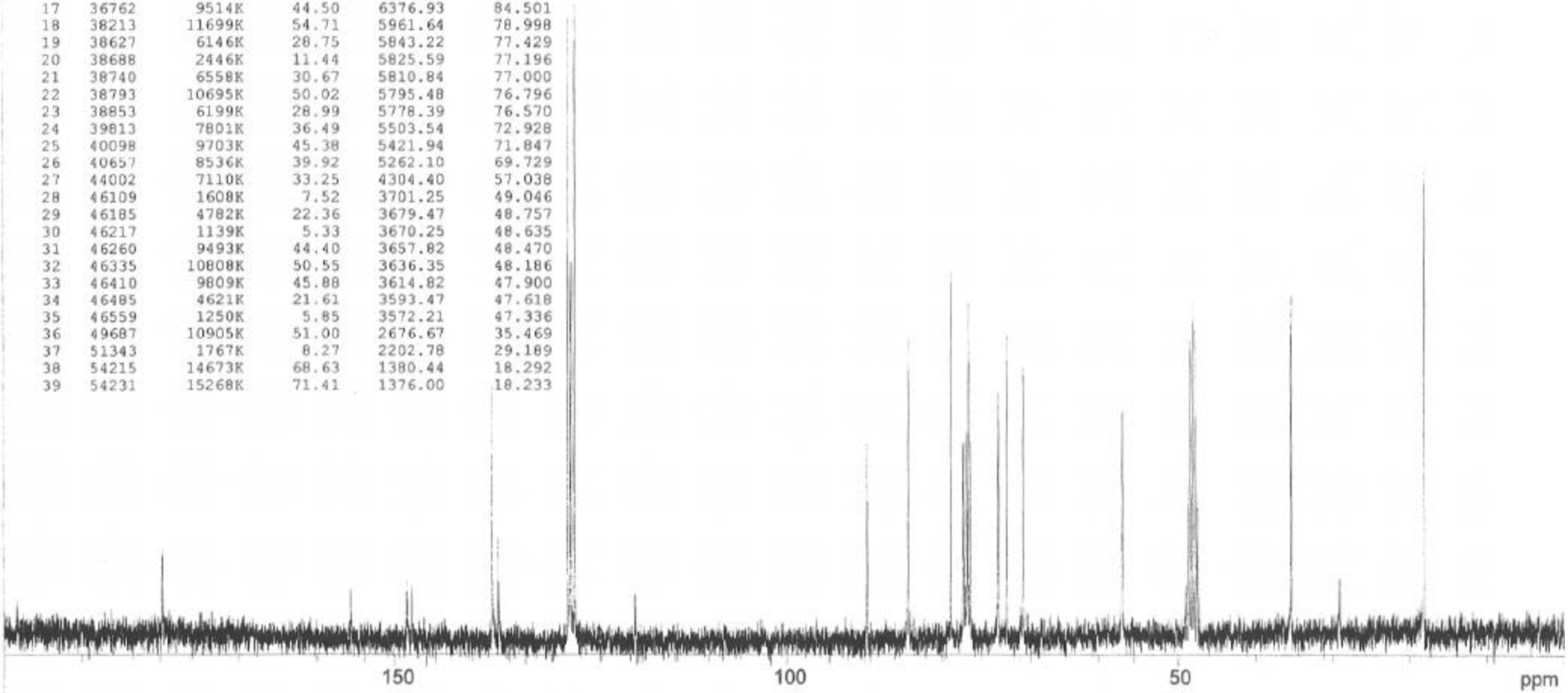
Interpolated Peak Listing

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	6756	1143K	5.34	14967.36	198.334
2	11668	2770K	12.96	13561.01	179.698
3	18027	1561K	7.30	11740.47	155.574
4	19916	1722K	8.05	11199.73	148.409
5	19938	1097K	5.13	11193.39	148.325
6	20088	1552K	7.26	11150.48	147.756
7	20584	-1127520	-5.27	11008.67	145.877
8	22780	7921K	37.05	10379.76	137.543
9	22991	3120K	14.59	10319.34	136.743
10	25273	20120K	94.10	9666.17	128.087
11	25383	11992K	56.09	9634.55	127.668
12	25505	20832K	97.43	9599.72	127.207
13	27545	1242K	5.81	9015.63	119.467
14	27605	-1170728	-5.48	8998.51	119.240
15	32133	-1116913	-5.22	7702.21	102.063
16	35377	6285K	29.40	6773.49	89.756
17	36762	9514K	44.50	6376.93	84.501
18	38213	11699K	54.71	5961.64	78.998
19	38627	6146K	28.75	5843.22	77.429
20	38688	2446K	11.44	5825.59	77.196
21	38740	6558K	30.67	5810.84	77.000
22	38793	10695K	50.02	5795.48	76.796
23	38853	6199K	28.99	5778.39	76.570
24	39813	7801K	36.49	5503.54	72.928
25	40098	9703K	45.38	5421.94	71.847
26	40657	8536K	39.92	5262.10	69.729
27	44002	7110K	33.25	4304.40	57.038
28	46109	1608K	7.52	3701.25	49.046
29	46185	4782K	22.36	3679.47	48.757
30	46217	1139K	5.33	3670.25	48.635
31	46260	9493K	44.40	3657.82	48.470
32	46335	10808K	50.55	3636.35	48.186
33	46410	9809K	45.88	3614.82	47.900
34	46485	4621K	21.61	3593.47	47.618
35	46559	1250K	5.85	3572.21	47.336
36	49687	10905K	51.00	2676.67	35.469
37	51343	1767K	8.27	2202.78	29.189
38	54215	14673K	68.63	1380.44	18.292
39	54231	15268K	71.41	1376.00	18.233



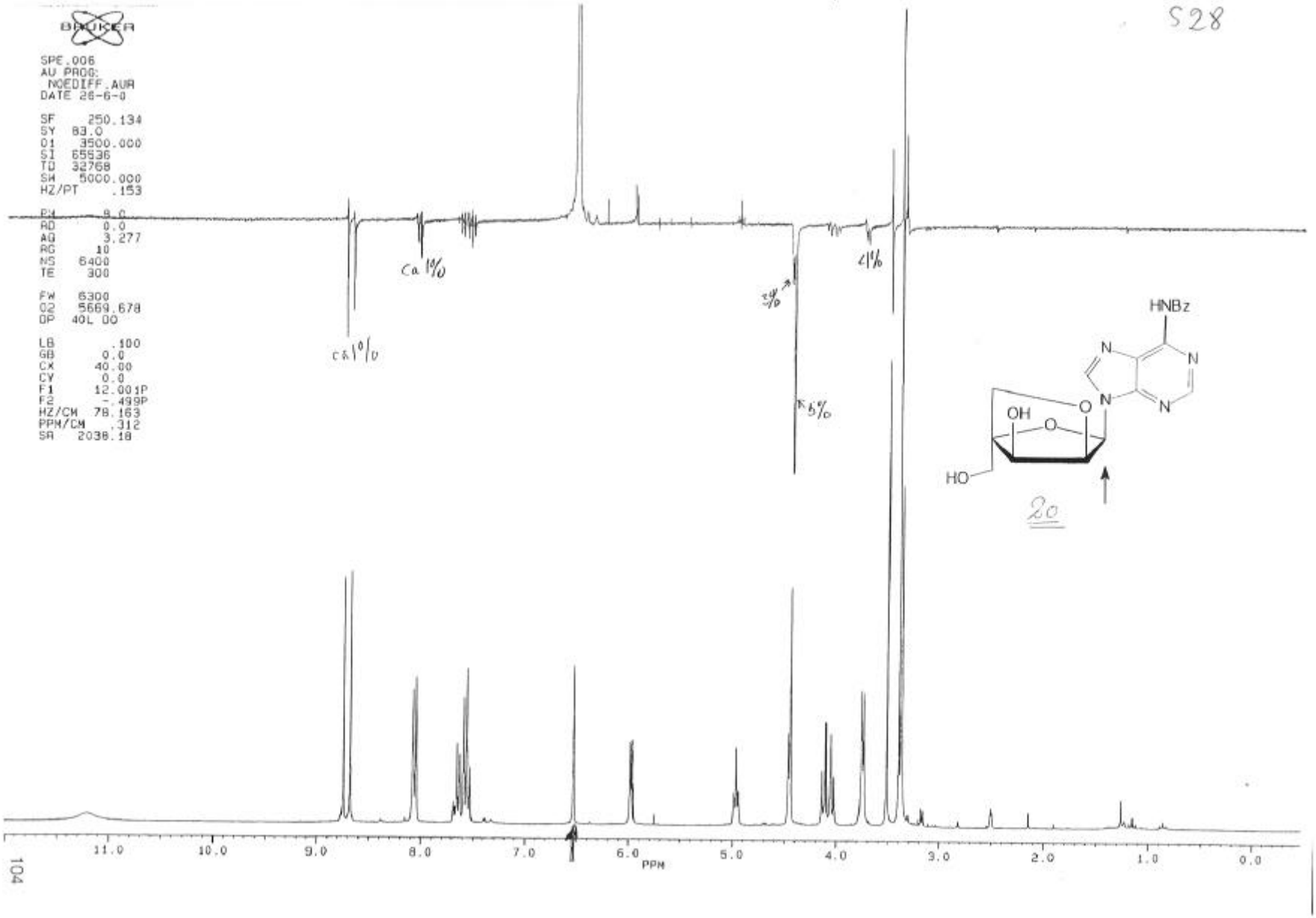
S27

35



gga 014 USER: DATE: Dec 19 2000

F1: 75.465	F2: 300.092	SW1: 18762		OF1: 7520.5		PTS1d: 65536
EX: s2pul		PW: 8.8 usec	PD: 1.2 sec	NA: 1024	LB: 1.0	WinNuts - $gga1219.014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SPE.006
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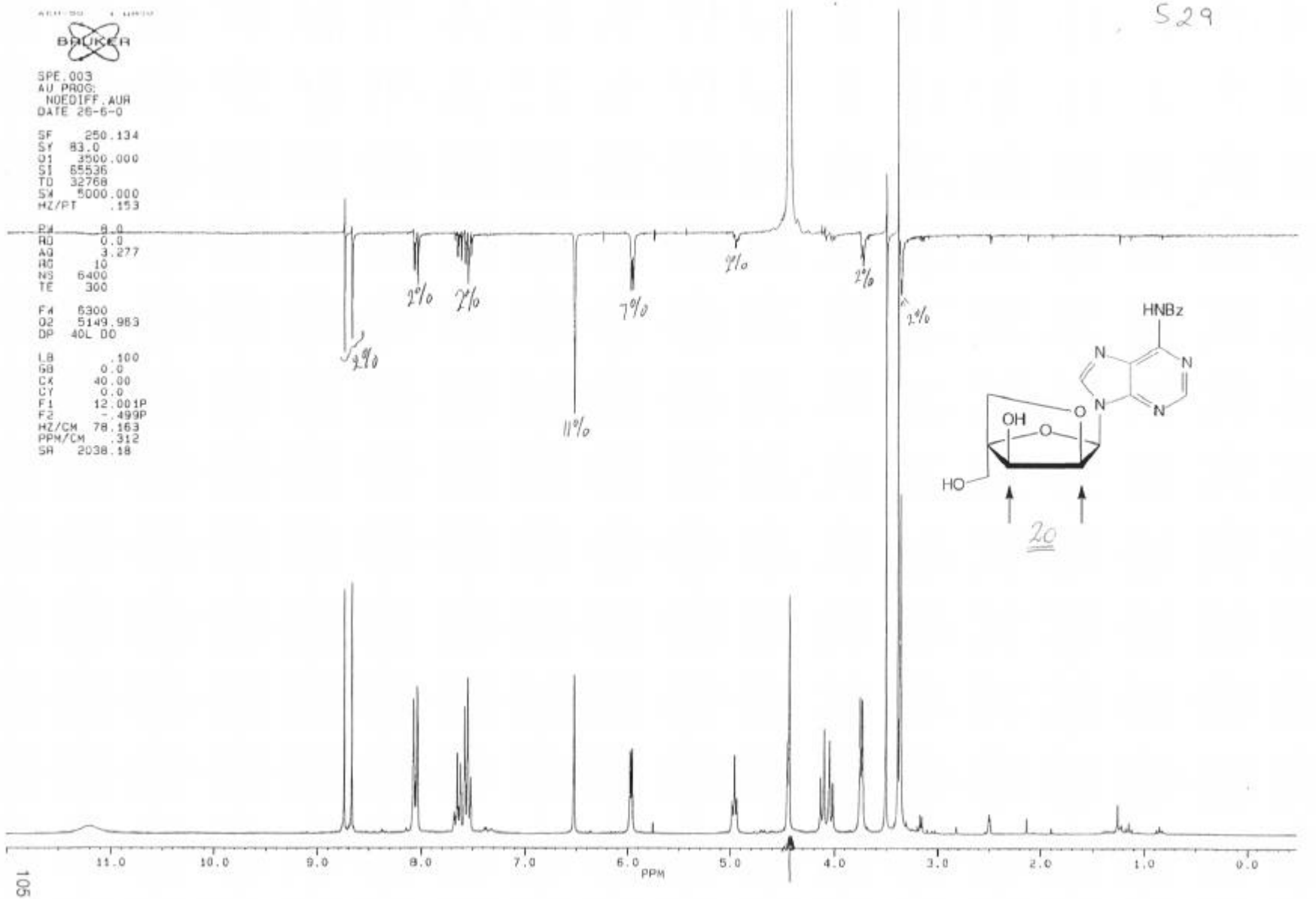
DATE 26-6-0
SF 250.134
SY 83.0
O1 3500.000
SI 65536
TD 32768
SH 5000.000
HZ/PT .153
PW 9.0
RD 0.0
AG 3.277
RG 10
NS 6400
TE 300
FW 6300
O2 5669.678
OP 40L 00
LB .100
GB 0.0
CX 40.00
CV 0.0
F1 12.001P
F2 -.499P
HZ/CM 78.163
PPM/CM .312
SR 2038.18
ca 1%
ca 1%
5%
4%
HNBz
N
N
N
N
O
OH
O
HO
20
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
PPM
104


S29
BRUKER
SPE.003
AU PROG:
NOEDIFF.AUR
DATE 26-6-0
SF 250.134
SW 83.0
O1 3500.000
SI 65536
TD 32768
SW 5000.000
HZ/PT .153
PW 8.0
RD 0.0
AQ 3.277
RG 10
NS 6400
TE 300
F4 6300
O2 5149.983
DP 40L DD
LB .100
GB 0.0
CX 40.00
CY 0.0
F1 12.001P
F2 -.499P
HZ/CM 78.163
PPM/CM .312
SR 2038.18
2%
2%
2%
11%
7%
2%
2%
2%
HNBz
N
N
N
N
O
OH
O
HO
20
11.0
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7.0
6.0
PPM
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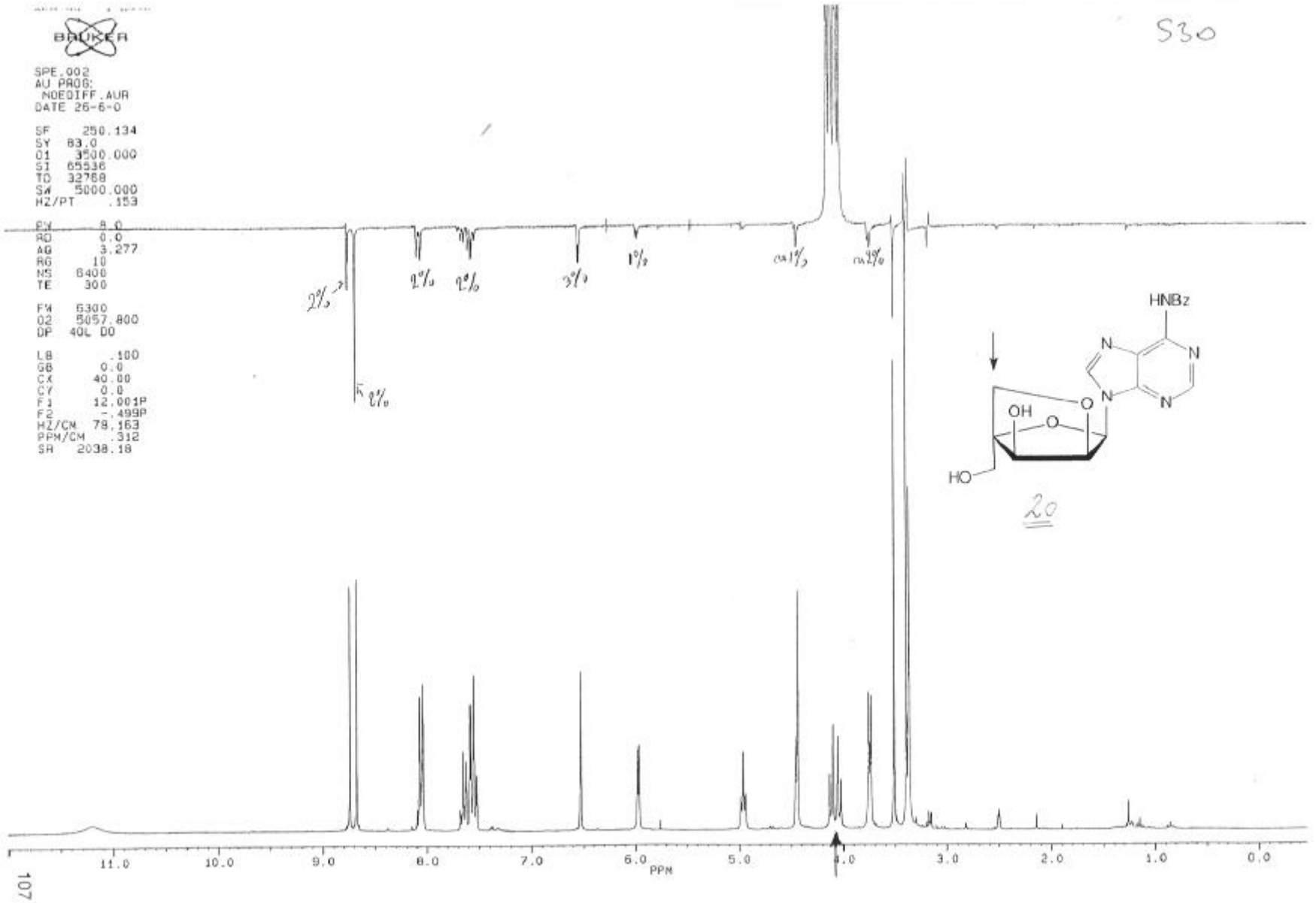
3.0
2.0
1.0
0.0
105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DATE 26-6-0
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SY 83.0
O1 3500.000
SI 65536
TD 32768
SW 5000.000
HZ/PT .153
PW 8.0
RD 0.0
AG 3.277
RG 10
NS 6400
TE 300
FW 6300
O2 5057.800
DP 40L DO
LB .100
GB 0.0
CX 40.00
CY 0.0
F1 12.001P
F2 -.499P
HZ/CM 78.163
PPM/CM .312
SR 2038.18
2%
9%
2%
2%
3%
1%
ca1%
ca2%
HNBz
N
N
N
N
O
OH
O
HO
20
11.0
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7.0
6.0
PPM
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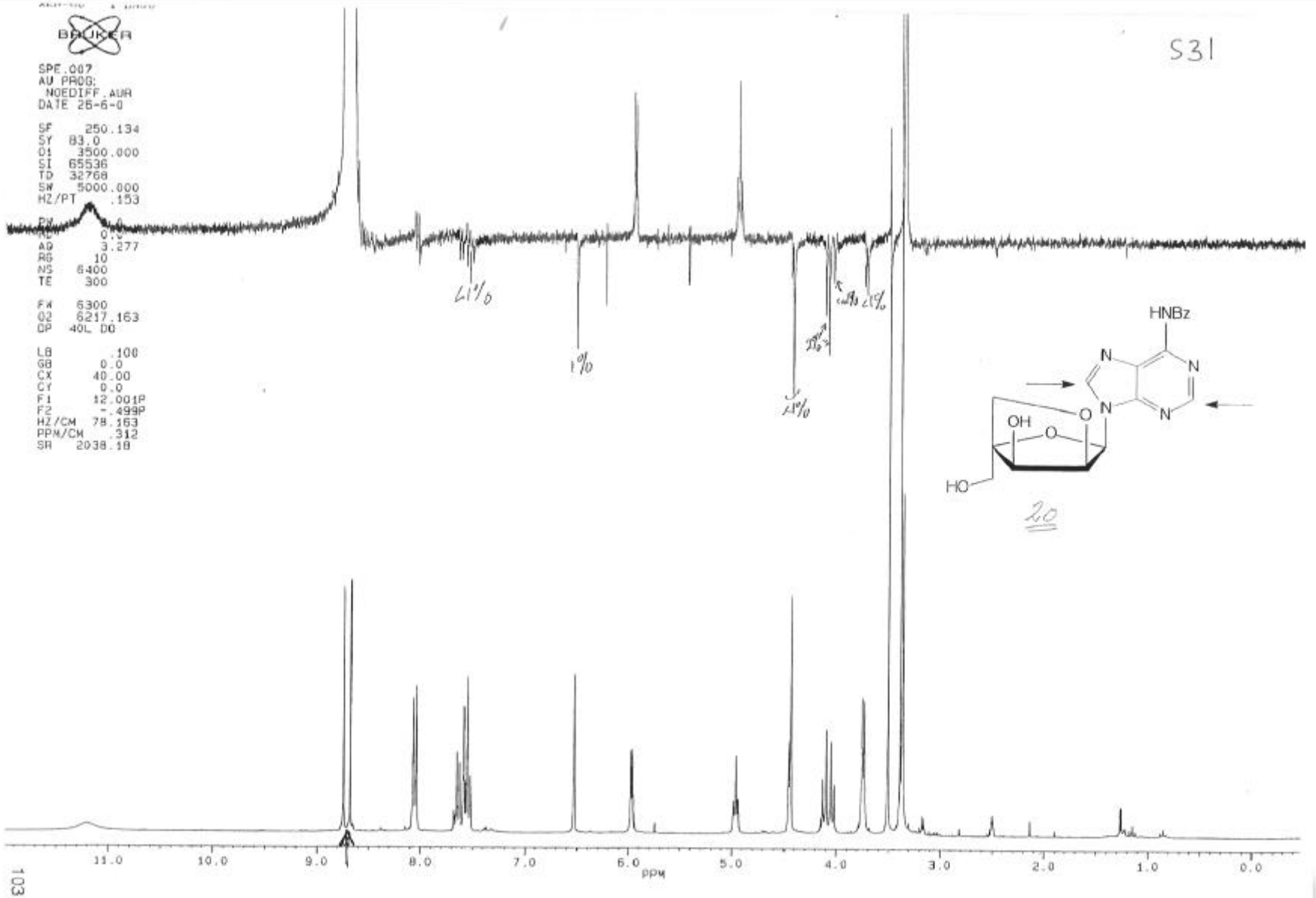
3.0
2.0
1.0
0.0
107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DATE 25-6-0
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SI 65536
TD 32768
SW 5000.000
HZ/PT .153
AQ 3.277
RG 10
NS 6400
TE 300
FW 6300
O2 6217.163
DP 40L D0
LB .100
GB 0.0
CX 40.00
CY 0.0
F1 12.001P
F2 -.499P
HZ/CM 78.163
PPM/CM .312
SR 2038.18
HNBz
N
N
N
N
O
OH
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10.0
9.0
8.0
7.0
6.0
5.0
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3.0
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1.0
0.0
ppm

20