

Supporting Information

Monomers for Preparation of Amide Linked RNA: Asymmetric Synthesis of All Four Nucleoside 5'-Azido 3'-Carboxylic Acids

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Experimental Procedures

Methylene chloride, pyridine, acetonitrile and toluene are dried by refluxing with CaH_2 followed by distillation. Tetrahydrofuran was distilled from sodium/benzophenone ketyl. (1*S*, 2*R*, 5*S*)-Menthyl Glyoxylate was prepared following a literature procedure.¹ Reactions were carried out in oven (150 °C) dried glassware under an atmosphere of dry nitrogen. NMR spectra were recorded at ambient temperature. Chemical shifts for ^1H are given in ppm using internal standard (tetramethylsilane for spectra in CDCl_3) or the residual solvent peak (δ 3.31 ppm, for spectra in CD_3OD). Chemical shifts for ^{13}C are given in ppm using the residual solvent peak (δ 77.17 ppm, for spectra in CDCl_3 , and δ 49.00 ppm for spectra in CD_3OD). Thin layer chromatography (TLC) was performed on 0.25 mm silica gel 60-F₂₅₄ plates. Column chromatography was done on 230-400 mesh silica gel.

(2*R*, 3*R*)-3-[2-(*tert*-Butyldiphenylsilanoxy)ethyl]-2-hydroxy-4-pentenoic acid dimethylamide (6c). A solution of AlMe_3 in toluene (25.5 mL, 2 M, 51 mmol) was slowly added to a suspension of the dimethylamine hydrochloride (4.16 g, 51 mmol) in dry toluene (60 mL) at 0 °C. The mixture was warmed to room temperature and stirred for 2 h until gas evolution was ceased. This solution of the aluminum amide reagent was then added to a solution of (2*R*, 3*R*)-3-[2-(*tert*-butyldiphenylsilanoxy)ethyl]-2-hydroxy-4-pentenoic acid (1'*S*, 2'*R*, 5'*S*)-menthyl ester **6b** (8.74 g, 16.3 mmol) in dry toluene (25 mL). The mixture was heated for 1 day at 70 °C and for 1 day at 90 °C. CH_2Cl_2 (340 mL) was added and the mixture was poured into 0.1 N HCl (350 mL). The aqueous layer was

¹ Whitesell, J. K.; Lawrence, R. M.; Chen, H-H. *J. Org. Chem.* **1986**, *51*, 4779-4784.

extracted with CH₂Cl₂ (3 × 350 mL). The combined organic layers were dried (Na₂SO₄) and concentrated. The residue was purified by silica gel column chromatography (0.5-5% of isopropanol in CH₂Cl₂, stepwise gradient by 1%) to afford **6c** as oil. Yield: 5.47 g, 79%, TLC R_f = 0.17 CH₂Cl₂/isopropanol (100:1). NMR spectra revealed ~20% of unknown impurity which was also formed (~10%) in our previous synthesis from ethyl ester.² The impurity did not interfere with the next iodolactonization step and was completely removed afterward. ¹H NMR (CDCl₃, 300 MHz) δ 7.68-7.61 (m, 4H), 7.45-7.33 (m, 6H), 5.71-5.59 (m, 1H), 5.13-4.91 (m, 2H), 4.48 (d, *J* = 3.9 Hz, H), 3.88-3.56 (m, 2H), 2.98 (s, 6H), 2.75-2.62 (m, 1H), 1.95-1.73 (m, 2H), 1.05 (s, 9H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 173.2, 136.0, 135.6, 135.5, 133.94, 133.87, 129.7, 127.7, 127.7, 117.4, 70.8, 61.3, 42.9, 36.5, 36.0, 34.3, 27.0, 19.3.

(3*R*, 4*S*, 5*S*)-5-Azidomethyl -4-[2-(*tert*-butyldiphenylsilanoxy)ethyl] -3-hydroxy-2-dihydrofuranone (mixture of **11a and **11b**).** NaN₃ (2.6 g, 40.0 mmol) was added to the solution of (3*R*, 4*S*, 5*S*)-4-[2-(*tert*-butyldiphenylsilanoxy)ethyl]-3-hydroxy-5-iodomethyl-2-dihydrofuranone (4.6 g of a mixture of **5a** and **5b**, 8.78 mmol) in dry DMF (60 mL). The mixture was stirred for 23 h at 50 °C and concentrated in vacuum. The residue was purified by silica gel column chromatography (10-30% of ethyl acetate in hexanes, stepwise gradient by 10%) to afford a non-separable mixture of C4-C5 *trans* (**11a**) and *cis* (**11b**) azidolactones in a ratio of 4:1. Yield: 3.03 g, 78%, TLC R_f = 0.24 CH₂Cl₂/isopropanol (100:1). R 3430, 2107, 1781 cm⁻¹. ¹H NMR (CDCl₃, 300 MHz) major diastereomer δ 7.67-7.64 (m, 4H), 7.47-7.37 (m, 6H), 4.54-4.50 (m, 1H), 4.48-4.43 (m, 1H), 3.77-3.73 (m, 3H), 3.64-3.37 (m, 2H), 2.61-2.52 (m, 1H), 2.07-1.91 (m, 1H),

² Rozners, E.; Liu, Y. *Org. Lett.* **2003**, *5*, 181-184.

1.68-1.55 (m, 1H), 1.07 (s, 9H). ^{13}C NMR (CDCl_3 , 75.4 MHz) major diastereomer δ 176.2, 150.7, 135.8, 133.1, 133.0, 130.3, 128.2, 81.8, 69.1, 62.4, 53.0, 40.7, 27.1, 19.3.

5-Azido-3-[2-(*tert*-butyldiphenylsilanoxy)ethyl]-1,2-di-*O*-acetyl-3,5-dideoxy

ribofuranose (4). DIBAL-H (5.9 mL, 1.5 M in toluene, 8.91 mmol, 2.3 equiv.) was added to a stirred solution of (3*R*, 4*S*, 5*S*)-5-azidomethyl-4-[2-(*tert*-butyldiphenylsilanoxy)ethyl]-3-hydroxy-2-dihydrofuranone (mixture of **7a** and **7b**) (1.7 g, 3.87 mmol) in dry CH_2Cl_2 (14 mL) over 7 min at -78°C . After the mixture was stirred for 110 min at -78°C , saturated aqueous NH_4Cl (100 mL) was added to quench the reaction at -78°C . The solution was extracted with ethyl acetate (4×150 mL). The combined organic layers were dried (Na_2SO_4), concentrated, and co-evaporated with dry toluene (3×5 mL) to give crude diol (1.77 g) as a colorless oil that was used in the next step without further purification. The oil was dissolved in a mixture of acetic anhydride and pyridine (1:1, 20 mL). The mixture was stirred for 23 h at room temperature and concentrated. The residue was purified by silica gel column chromatography (10-20% of ethyl acetate in hexanes stepwise gradient by 10%) to afford **4** as oil. Yield: 1.73 g, 86% two steps. IR 2104, 1750 cm^{-1} . ^1H NMR showed the expected mixture of four diastereomers that was used in the next step without separation.

2'-*O*-Acetyl-5'-azido-3'-[2-(*tert*-butyldiphenylsilanoxy)ethyl]-3',5'-dideoxy-4-*N*-

propionylecytidine 12b. TMSCl (0.40 g, 3.68 mmol) was added to a solution of 2'-*O*-acetyl-5'-azido-3'-[2-(*tert*-butyldiphenylsilanoxy)ethyl]-3',5'-dideoxycytidine **12b'** (1.06 g, 1.84 mmol) in pyridine (20 mL). After stirring for 1 h, propionic anhydride (0.29 g, 2.2 mmol) was added. The mixture was stirred for 16 h, H_2O (1 mL) was added and the

mixture was stirred for another 20 min. The solution was concentrated, co-evaporated with toluene (3×8 mL) and purified by silica gel column chromatography (5-30% of isopropanol in hexanes, stepwise gradient by 5%) to afford **12b** as a single 3',4'-*trans* diastereomer. Yield: 0.71 g, 61%. TLC R_f = 0.34 CH_2Cl_2 /isopropanol (96:4), IR 2103, 1668 cm^{-1} . ^1H NMR (CDCl_3 , 300 MHz) δ 10.43 (s, 1H), 8.07 (d, J = 7.5 Hz, H), 7.64-7.59 (m, 4H), 7.52 (d, J = 7.5 Hz, H), 7.44-7.32 (m, 6H), 5.87 (s, 1H), 5.41 (d, J = 5.7 Hz, H), 4.13-4.07 (m, 1H), 3.86-3.80 (m, 1H), 3.76-3.61 (m, 2H), 3.57-3.51 (m, 1H), 2.67-2.49 (m, 3H), 2.04 (s, 3H), 1.63-1.47 (m, 2H), 1.15 (t, J = 7.5 Hz, 3H), 1.04 (s, 9H). ^{13}C NMR (CDCl_3 , 75.4 MHz) δ 175.0, 169.0, 163.3, 154.6, 144.2, 135.4, 133.4, 133.2, 129.8, 127.7, 96.8, 91.7, 82.8, 76.7, 61.3, 51.6, 38.2, 30.4, 27.3, 26.8, 20.5, 19.1, 8.6. MS (ESI) calculated for $\text{C}_{32}\text{H}_{40}\text{N}_6\text{O}_6\text{Si}$ 632.3, found $[\text{M}+\text{H}]^+$ 633.5.

2'-O-Acetyl-5'-azido-3'-(2-hydroxyethyl)-3',5'-dideoxy-4-N-propionylcytidine 13b.

Acetic acid (0.92 mL, 16 mmol) was added to 1M TBAF solution in THF (16 mL). To this solution of 1M TBAF/HOAc (1:1 mol/mol) was added 2'-O-acetyl-5'-azido-3'-[2-(*tert*-butyldiphenylsilanoxy)ethyl]-3',5'-dideoxy-4-N-propionylcytidine **12b'** (0.35g, 0.55mmol) dissolved in THF (200 mL). The reaction mixture was stirred for 5 h, diluted with CH_2Cl_2 (1200 mL) and applied to silica gel chromatography (1-9% of methanol in CH_2Cl_2 stepwise gradient by 2%) to give crude product 227 mg, which was further purified by another silica gel column (1-7% of methanol in CH_2Cl_2 , stepwise gradient by 2%) to afford **13b**. Yield: 217 mg, 99%. TLC R_f = 0.1 MeOH/ CH_2Cl_2 (3:97), IR 2103, 1645 cm^{-1} , ^1H NMR (CDCl_3 , 300 MHz) δ 9.93 (s, 1H), 8.17 (d, J = 7.8 Hz, H), 7.36 (d, J = 7.5 Hz, H), 5.89 (d, J = 5.1 Hz, H), 5.83 (s, 1H), 4.19-4.13 (m, 1H), 3.90-3.85 (m, 1H), 3.74-3.68 (m, 1H), 3.59-3.49 (m, 3H), 2.63-2.41 (m, 3H), 2.16 (s, 3H), 1.56-1.53 (m,

2H), 1.13 (t, J = 7.5 Hz, 3H). ^{13}C NMR (CDCl_3 , 75.4 MHz) δ 175.2, 169.6, 162.9, 155.0, 144.4, 96.7, 91.7, 83.3, 77.6, 59.8, 51.8, 38.3, 30.5, 27.2, 20.9, 8.7. MS (ESI) calculated for $\text{C}_{16}\text{H}_{22}\text{N}_6\text{O}_6+\text{Na}$ 317.2, found $[\text{M}+\text{Na}]^+$ 417.1.

2'-O-Acetyl-5'-azido-3'-(2-hydroxyethyl)-3',5'-dideoxy-6-N-benzoyladenine 13c.

Acetic acid (17 μL , 0.3 mmol) was added to 1M TBAF solution in THF (0.30 mL). To this solution of 1M TBAF/HOAc (1:1 mol/mol) was added 2'-O-acetyl-5'-azido-3'-[2-(*tert*-butyldiphenylsiloxy)ethyl]-3',5'-dideoxy-6-N-benzoyladenine **12c** (21 mg, 0.03mmol) dissolved in THF (12 mL). The reaction mixture was stirred for 4 h, diluted with ethyl acetate (60 mL) and applied to silica gel chromatography (1-5% of methanol in ethyl acetate, stepwise gradient by 2%) to give crude product 17 mg, which was further purified by another silica gel column (1-7% of methanol in ethyl acetate, stepwise gradient by 2%) to afford **13c**. Yield: 13 mg, 93%. TLC R_f = 0.53 CH_2Cl_2 /isopropanol (90:10), IR 2104, 1641 cm^{-1} . ^1H NMR (CDCl_3 , 300 MHz) δ 8.77 (s, H), 8.32 (s, H), 8.03 (d, J = 7.2 Hz, 2H), 7.62-7.49 (m, 3H), 6.14 (s, 1H), 5.86 (d, J = 4.5 Hz, 1H), 4.25-4.21 (m, 1H), 3.81-3.56 (m, 4H), 3.10-3.00 (m, 1H), 2.18 (s, 1H), 1.86-1.60 (m, 2H). ^{13}C NMR (CDCl_3 , 75.4 MHz) δ 170.1, 165.0, 152.8, 151.2, 150.6, 149.6, 141.8, 133.7, 132.9, 129.0, 128.1, 123.3, 89.6, 83.7, 78.2, 60.5, 52.1, 39.5, 27.4, 20.9. MS (ESI) calculated for $\text{C}_{21}\text{H}_{22}\text{N}_8\text{O}_5+\text{Na}$ 489.2, found $[\text{M}+\text{Na}]^+$ 489.1.

2'-O-Acetyl-5'-azido-3'-(2-hydroxyethyl)-3',5'-dideoxy-2-N-acetyl-6-O-diphenylcarbamoylguanosine 13d. Acetic acid (1.37 mL, 24 mmol) was added to 1M TBAF solution in THF (24 mL). To this solution of 1M TBAF/HOAc (1:1 mol/mol) was added 2'-O-acetyl-5'-azido-3'-[2-(*tert*-butyldiphenylsiloxy)ethyl]-3',5'-dideoxy-2-N-

acetyl-6-*O*-diphenylcarbamoylguanosine **12d** (0.707 g, 0.83 mmol) dissolved in THF (300 mL). The reaction mixture was stirred for 5 h, diluted with CH₂Cl₂ (700 mL) and applied to silica gel chromatography (2-10% of isopropanol in CH₂Cl₂, stepwise gradient by 2%) to afford **13d**. Yield: 0.498 g, 98%. TLC R_f = 0.22 MeOH/CH₂Cl₂ (3:97). IR 2104, 1741 cm⁻¹. ¹H NMR (CDCl₃, 300 MHz) δ 8.64 (s, H), 8.05(s, H), 7.44-7.25 (m, 10H), 5.85 (s, H), 5.83 (d, *J*= 5.7 Hz, 1H), 4.17-4.11 (m, 1H), 3.74-3.49 (m, 5H), 2.22(s, 3H), 2.14 (s, 3H), 1.91-1.80 (m, 1H), 1.66-1.56 (m, 1H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 170.3, 169.7, 156.1, 153.8, 151.6, 150.5, 143.9, 141.7, 129.2, 127.1, 121.6, 90.7, 84.4, 78.5, 60.0, 52.6, 39.3, 28.0, 24.8, 20.7. MS (ESI) calculated for C₂₉H₂₉N₉O₇+Na 638.2, found [M+Na]⁺ 638.1.

Dimer 17. 2-(6-chloro-1H-benzotriazole-1-yl)-1,1,3,3-tetramethyluronium hexafluorophosphate (HCTU) (16.8 mg, 0.04 mmol) and diisopropylethylamine (9.56 mg, 0.074 mmol) were added to the solution of 2'-*O*-acetyl-5'-azido-3'-carboxymethyl-3',5'-dideoxyuridine **3a** (13 mg, 0.037 mmol) in DMF (0.5 mL). The mixture was stirred for 1.5 h. Then, to this solution of activated ester in DMF was added a solution of 5'-amino-5'-deoxy-2',3'-*O*-isopropylideneuridine³ **16** (60 mg, 0.21 mmol) in DMF (0.5 mL). The mixture was stirred for 50 min, concentrated, and purified by silica gel column chromatography (2-10% of methanol in CH₂Cl₂, stepwise gradient by 2%) to afford dimer **17**. Yield: 19 mg, 83%. TLC R_f = 0.25 MeOH/CH₂Cl₂ (6:94). ¹H NMR (CDCl₃, 300 MHz) δ 10.26 (s, 1H), 9.48 (s, 1H), 7.54 (d, *J*= 8.1 Hz, 1H), 7.21 (d, *J*= 8.1 Hz, 1H), 7.01 (b, 1H), 5.86-5.78 (m, 3H), 5.55 (d, *J*= 6 Hz, 1H), 5.28 (s, 1H), 5.23-5.20 (m, 1H), 4.93-4.89 (m, 1H), 4.26-4.23(m, 1H), 4.16-4.13 (m, 1H), 3.94-3.85 (m, 1H), 3.83-3.77 (m,

1H), 3.65-3.60 (m, 1H), 3.35 (d, J = 14.4 Hz, 1H), 3.11-3.07 (m, 1H), 2.55-2.48 (m, 1H), 2.33-2.25 (m, 1H), 2.15 (s, 3H), 1.56 (s, 3H), 1.36 (s, 3H). MS (ESI) calculated for $C_{25}H_{30}N_8O_{11}$ 618.2, found $[M+H]^+$ 619.0.

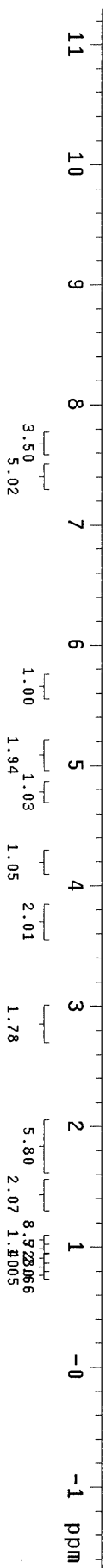
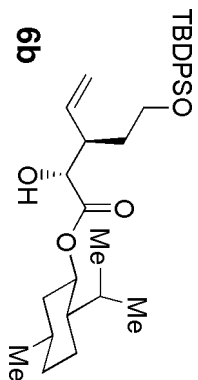
Dimer 18. A saturated solution of NH_3 in methanol (2 mL) was added to dimer **17** (3 mg, 0.0049 mmol). The solution was stirred for 20 min. It was concentrated and purified by preparative silica gel TLC eluting with 9% methanol in CH_2Cl_2 to afford **18**. Yield: 3 mg, >99%. TLC R_f = 0.35 MeOH/ CH_2Cl_2 (9:91). 1H NMR ($CDCl_3$, 300 MHz) δ 10.02 (s, 1H), 9.68 (s, 1H), 7.68 (d, J = 8.1 Hz, 1H), 7.22 (d, J = 8.1 Hz, 1H), 7.01 (b, 1H), 5.78-5.76 (m, 3H), 5.32 (d, J = 2.4 Hz, 1H), 5.17-5.14 (m, 1H), 4.83-4.79 (m, 1H), 4.44 (d, J = 3.9 Hz, 1H), 4.25-4.20 (m, 1H), 4.16-4.13 (m, 1H), 3.90-3.79 (m, 1H), 3.63-3.57 (m, 1H), 3.43-3.39 (m, 1H), 2.63-2.59 (m, 2H), 2.38-2.29 (m, 1H), 1.54 (s, 3H), 1.33 (s, 3H). MS (ESI) calculated for $C_{23}H_{28}N_8O_{10}$ 576.2, found $[M+H]^+$ 577.0.

³ Isono, K.; Azuma, T. *Chem. Pharm. Bull.* **1972**, 20, 193-196.

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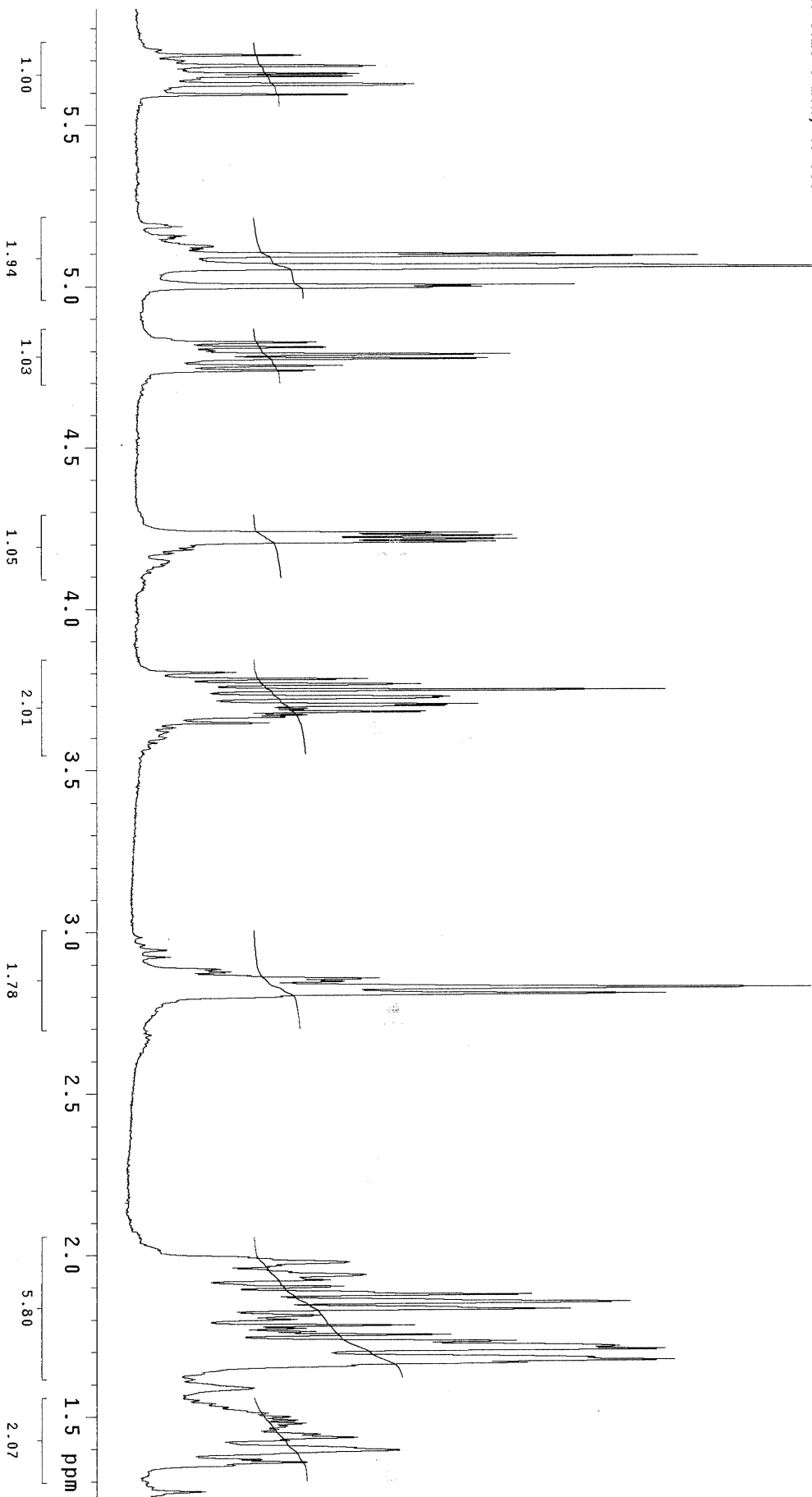
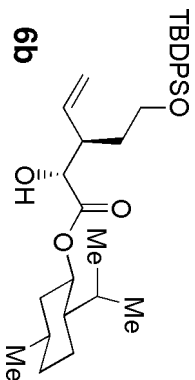
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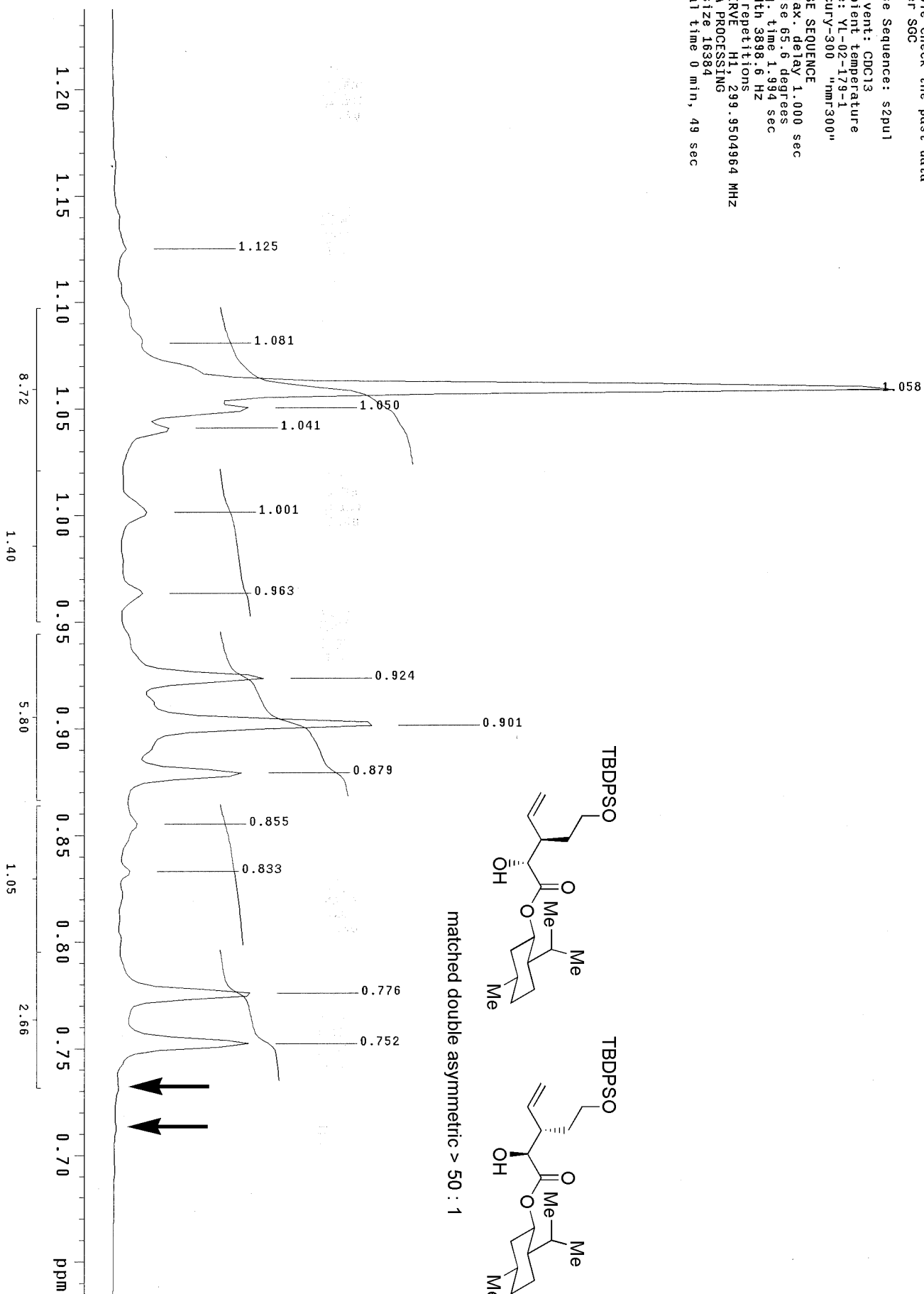
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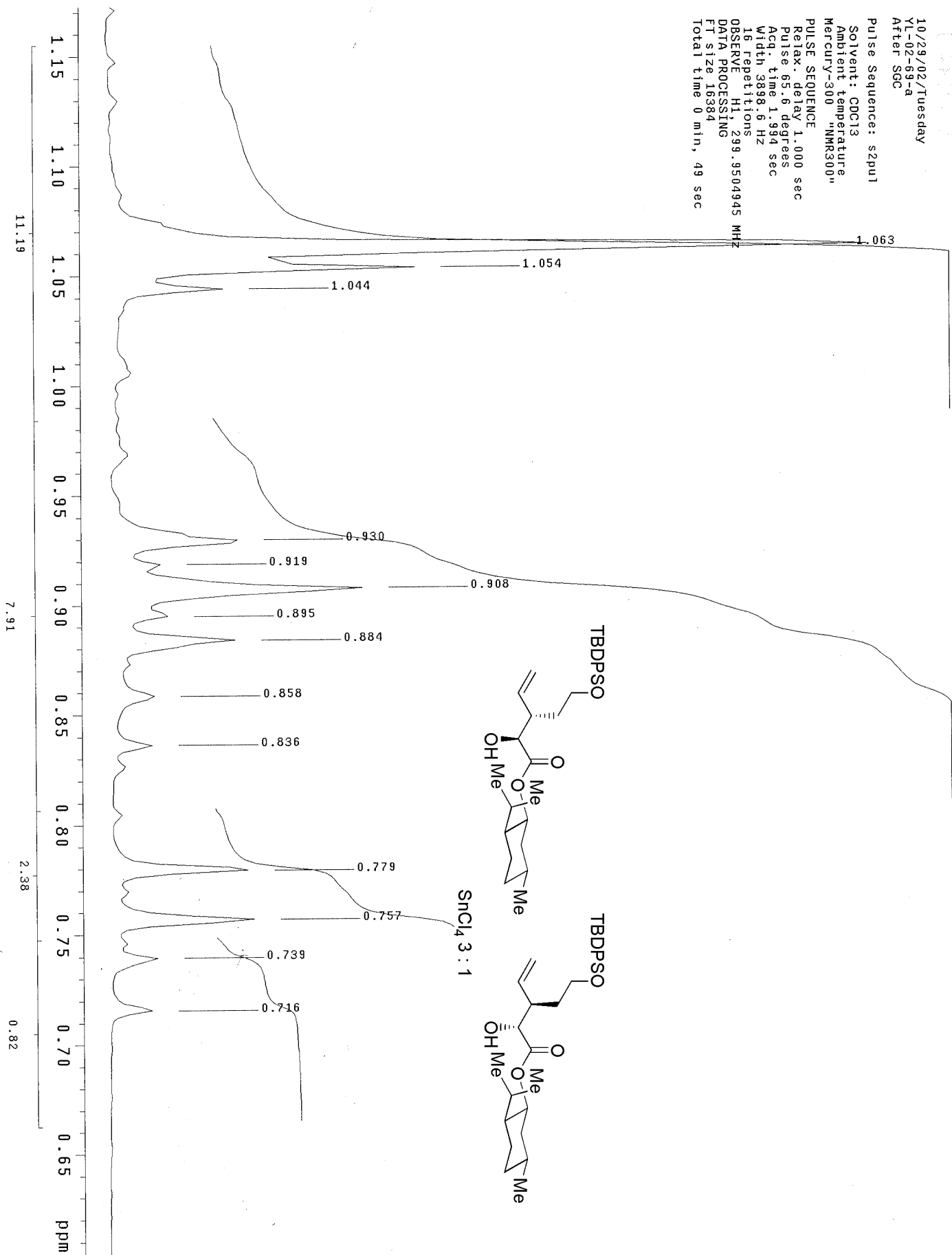
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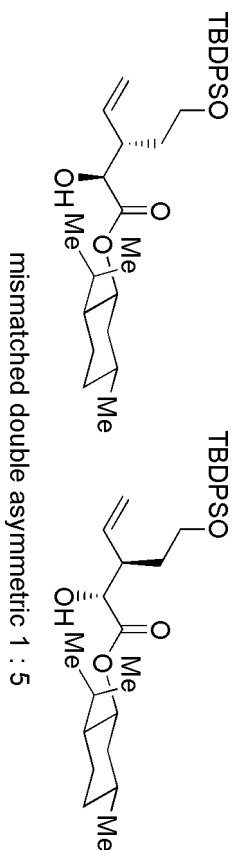
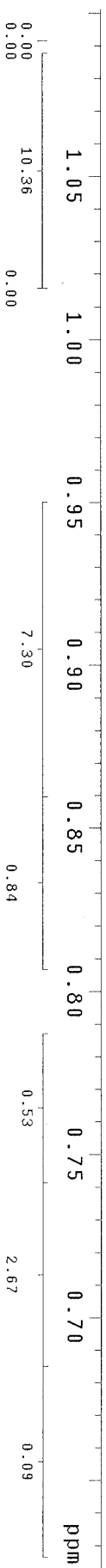
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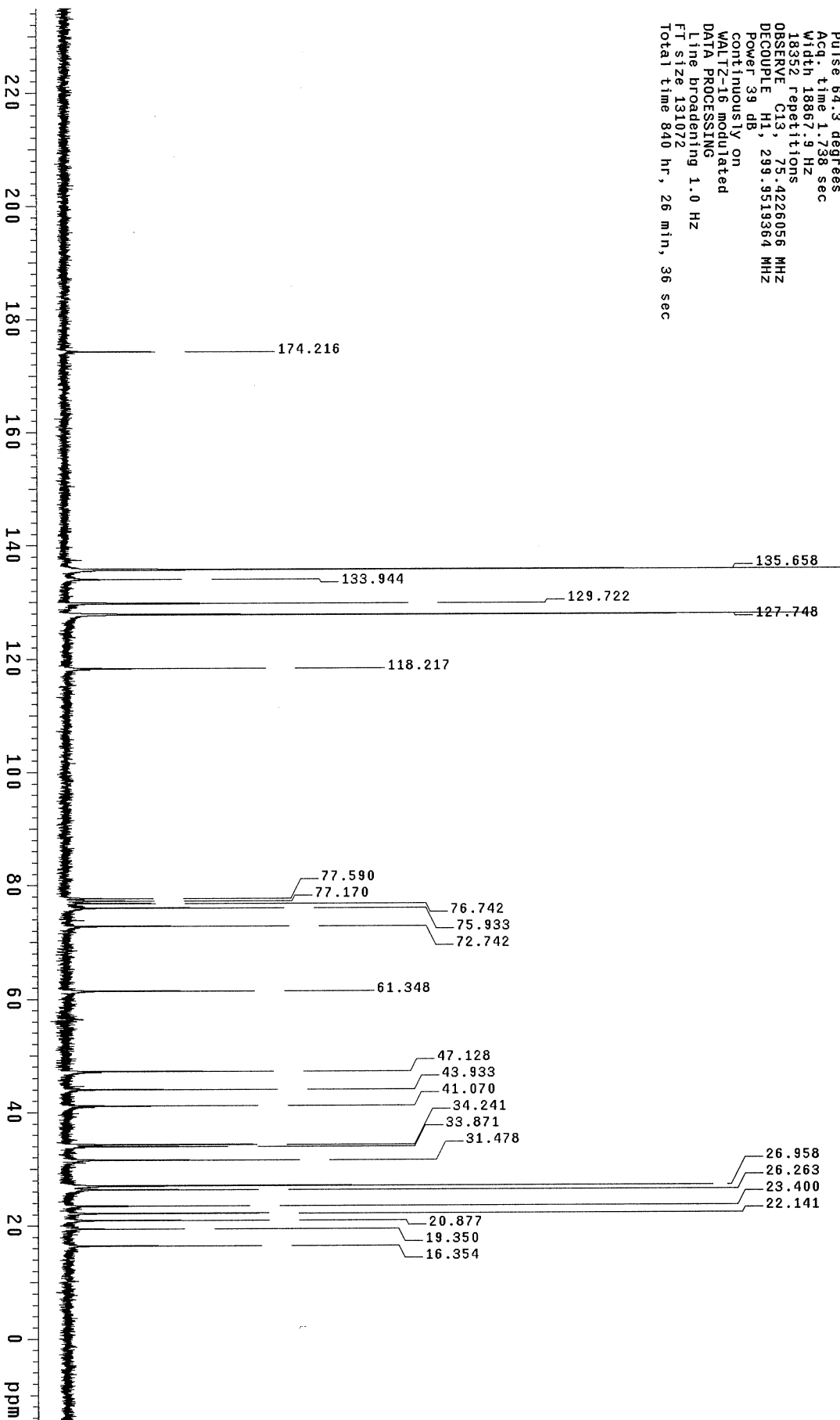
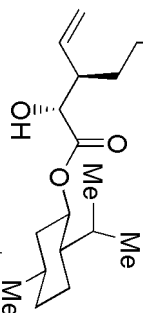
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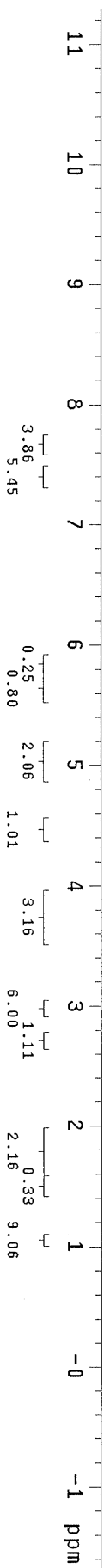
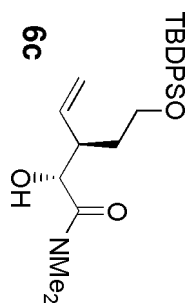
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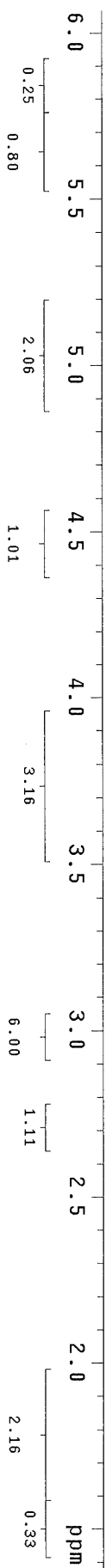
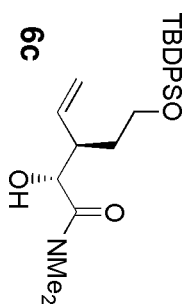
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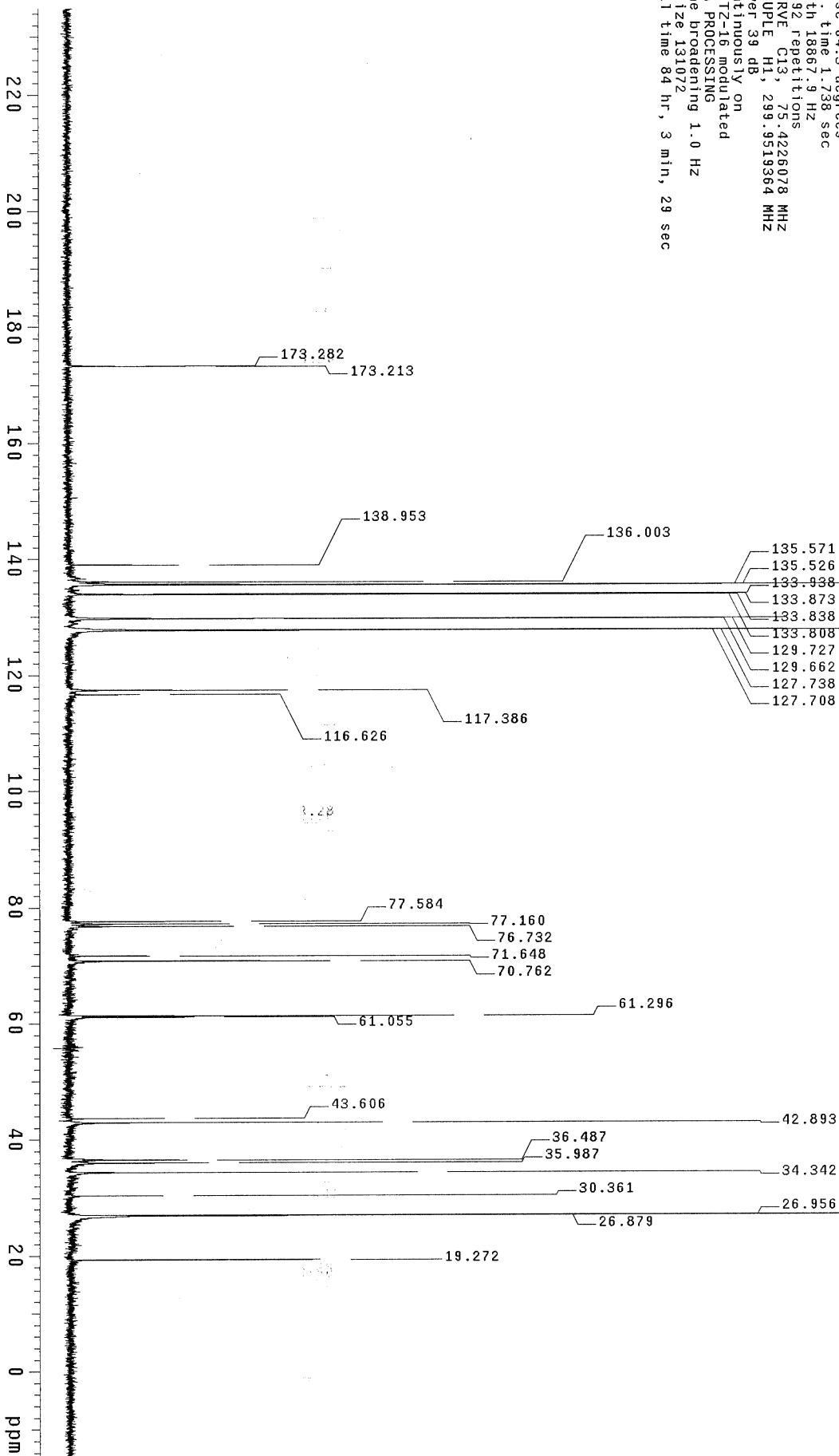
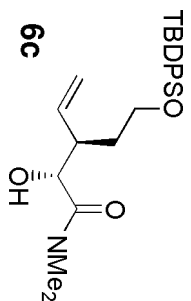
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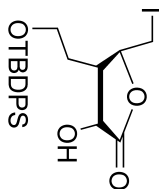
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OBSERVE C13, 75.4226078 MHz
DECOUPLE H1, 299.9519364 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 84 hr, 3 min, 29 sec

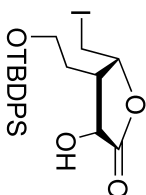


01/13/05/Thursday
YL-03-181-1
After SGC

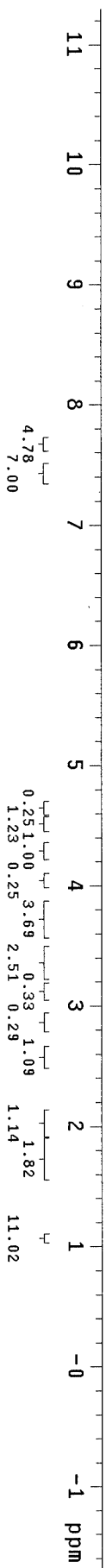
Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
Mercury-300 "nmr300"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 65.6 degrees
Acq. time 1.994 sec
Width 3898.6 Hz
16 repetitions
ORSERVE H1 299.9504974 MHz
DATA PROCESSING
FT size 16384
Total time 0 min, 49 sec



Major **5a**



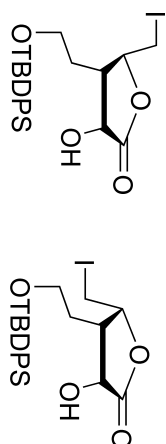
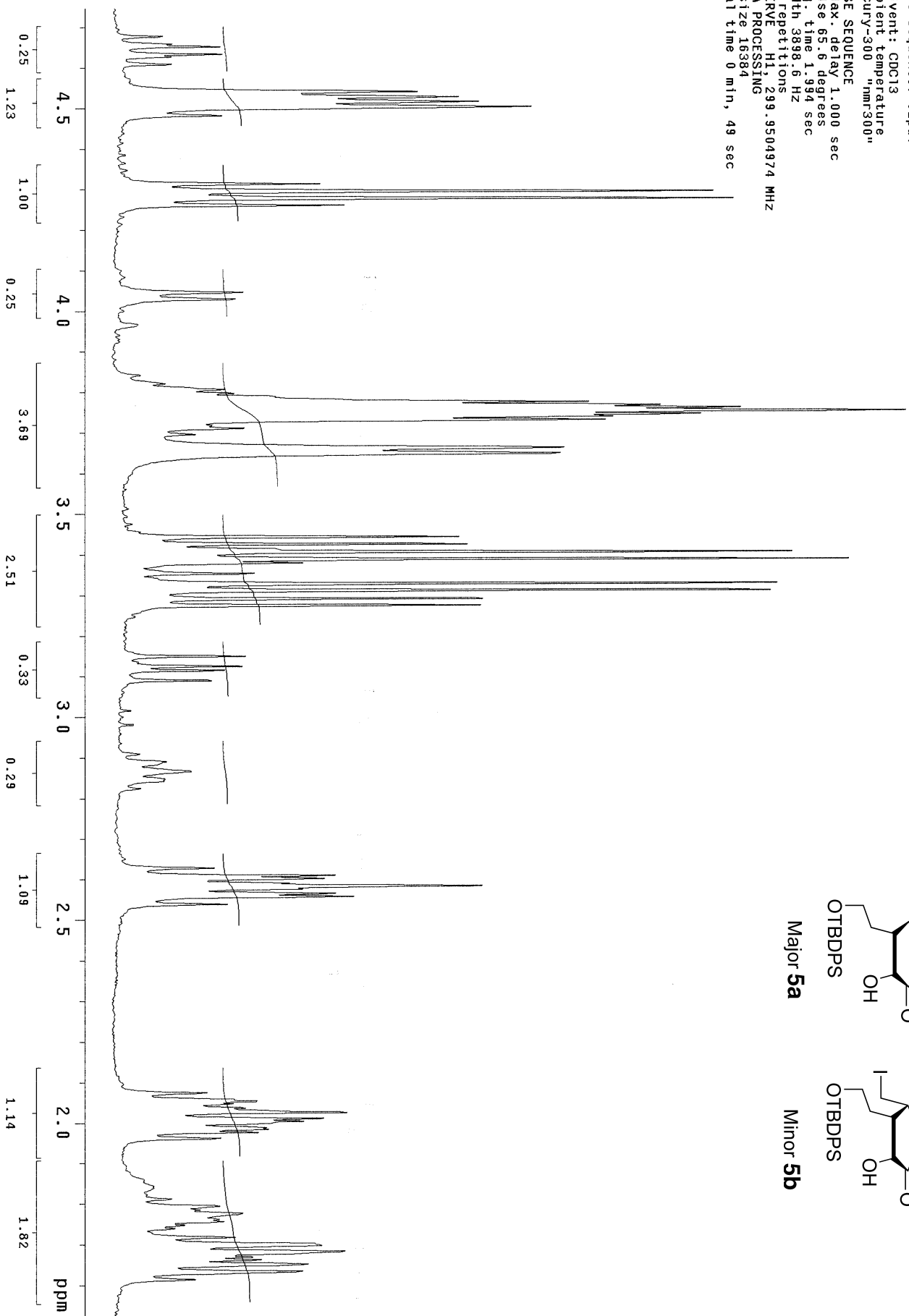
Minor **5b**



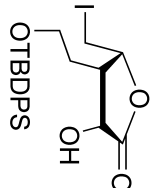
01/13/05/Thursday
YL-03-181-1
After SGC

Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
Mercury-300 "nmr300"

PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 65.6 degrees
Acq. time 1.994 sec
Width 3898.6 Hz
16 repetitions
OBSERVE H1, 299.9504974 MHz
DATA PROCESSING
FT size 16384
Total time 0 min, 49 sec



Major **5a**



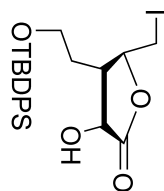
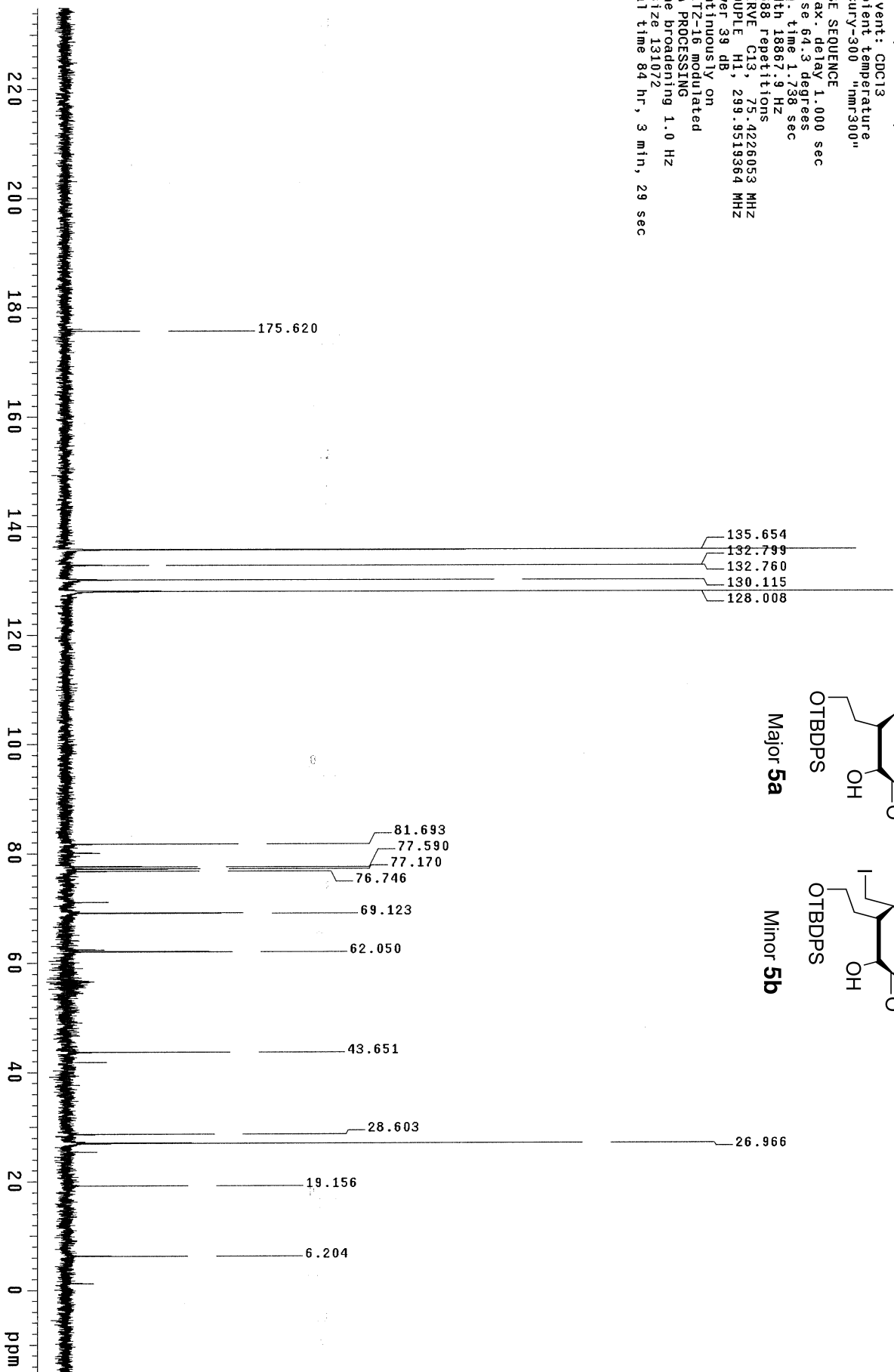
Minor **5b**

```

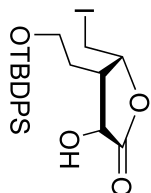
Pulse sequence: s2p1
Solvent: CDC13
Ambient temperature
Mercury-300 "nmr300"

PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse: 64.3 degrees
Acq. time 1.36 sec
Width 18867.9 Hz
18668 repetitions
OBSERVE C13, 75.4226053 MHz
DECOUPLE H1, 299.9519364 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 84 hr, 3 min, 29 sec

```



Major 5a

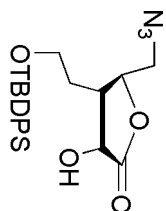


Minor 5b

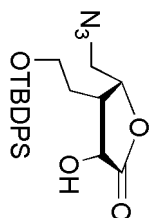
07/02/04/Friday
YL-02-239-1-1
After SGC

Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
Mercury-300 "nmr300"

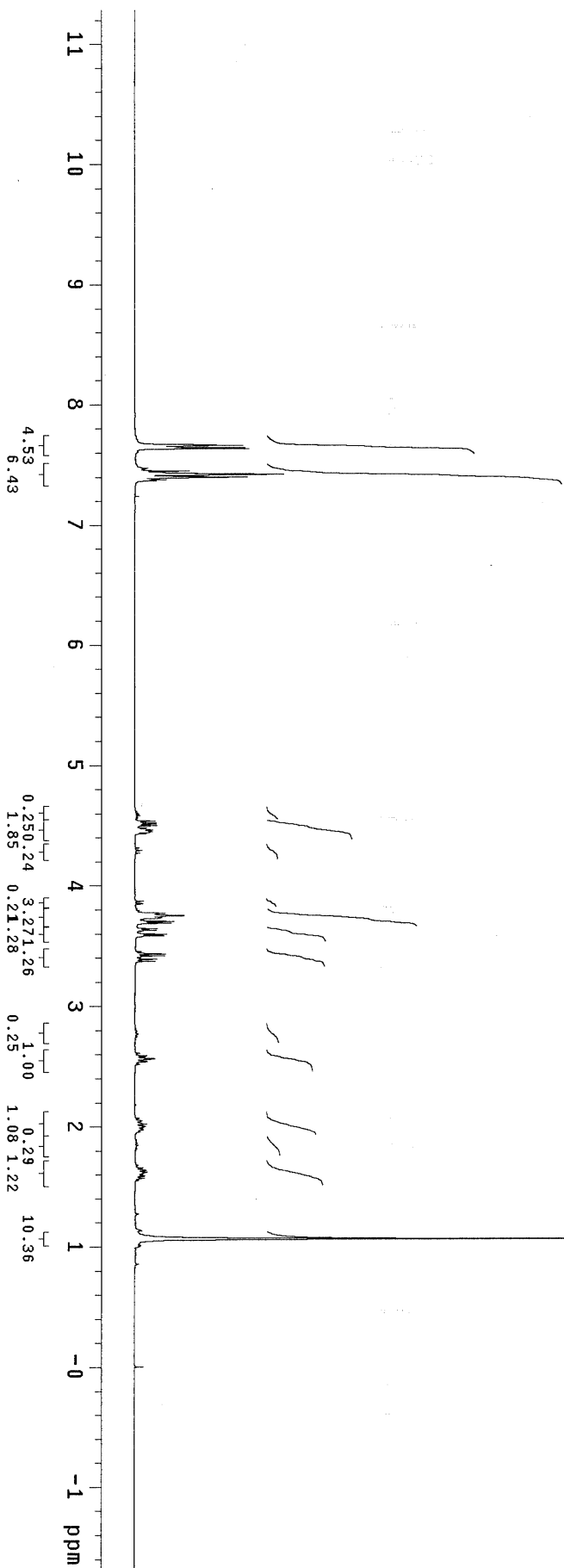
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 65.6 degrees
Acq. time 1.994 sec
Width 3898.6 Hz
16 repetitions
OBSERVE H1, 299.9505007 MHz
DATA PROCESSING
FT size 16384
Total time 0 min, 49 sec



Major **11a**



Minor **11b**



07/02/04/Friday
YL-02-239-1-1
After SGC

Pulse Sequence: s2pu1

Solvent: CDCl3
Ambient temperature
Mercury-300 "nmr-300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.994 sec

Width 3898.6 Hz

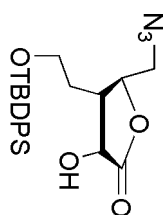
16 repetitions

OBSERVE H1 299.9505007 MHz

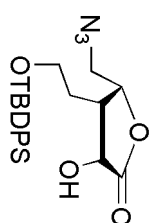
DATA PROCESSING

FT size 16384

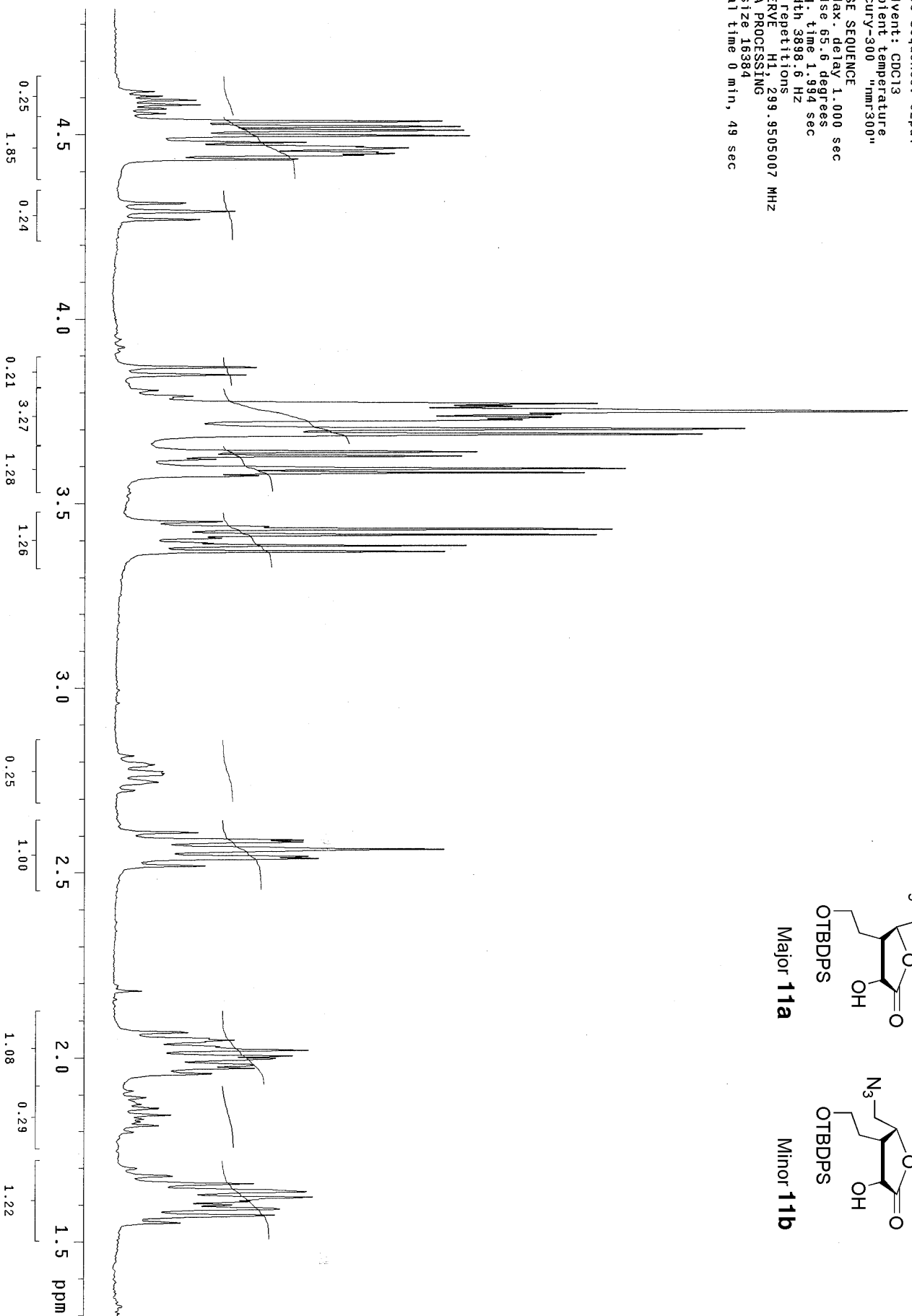
Total time 0 min, 49 sec



Major **11a**

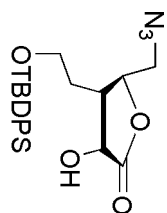
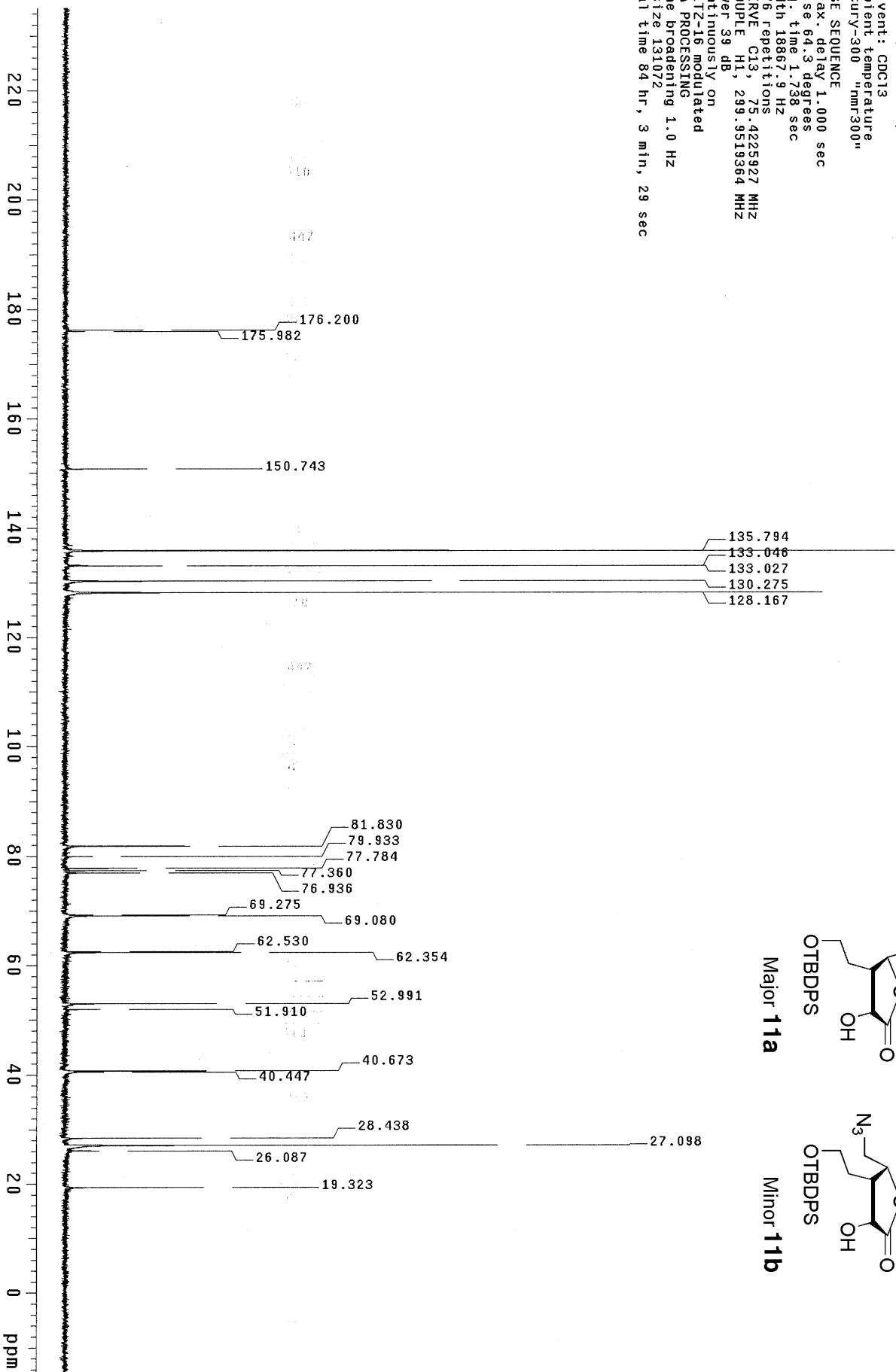


Minor **11b**

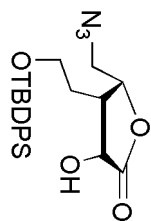


07/02/04/Friday
VI-02-239-1-1
After SGC

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300 "nmr300"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 64.3 degrees
Acq. time 1.738 sec
Width 1867.9 Hz
2576 repetitions
OBSERVE C13, 75.4225927 MHz
DECOUPLE H1, 299.9519364 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 84 hr, 3 min, 29 sec



Major 11a



Minor 11b

07/12/04/Monday
YL-03-87-2
After SGC

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: YL-03-87-2

Mercury-300 "nmr300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.994 sec

Width 3898.6 Hz

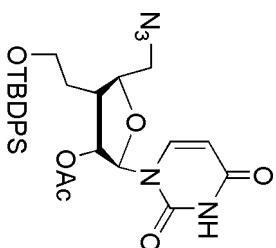
16 repetitions

OBSERVE H1 299.9504955 MHz

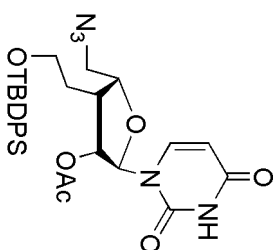
DATA PROCESSING

FT size 16384

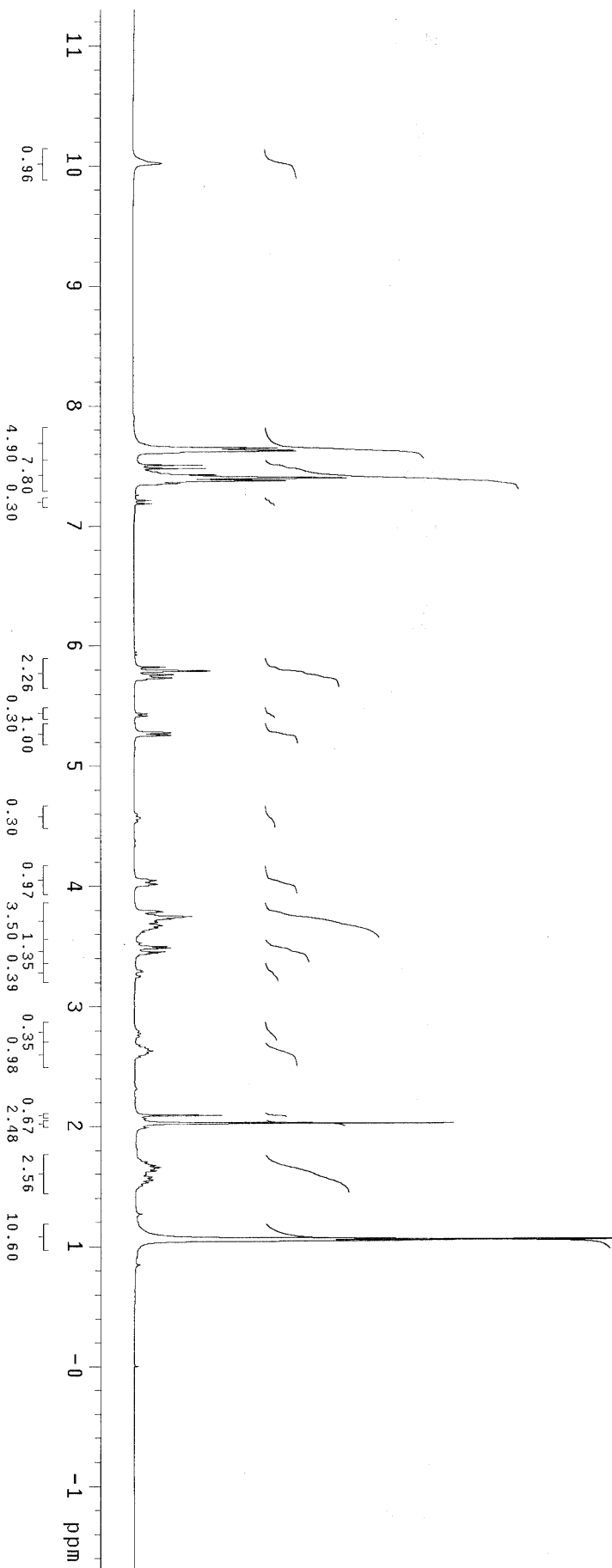
Total time 0 min, 49 sec



Major 12a



Minor



07/12/04/Monday
YL-03-87-2
After SGC

Pulse Sequence: szpu1

Solvent: CDCl3

Ambient temperature

File: YL-03-87-2

Mercury-300 "nmr300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.994 sec

Width 3898.6 Hz

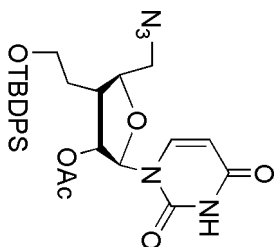
16 repetitions

OBSERVE H1, 299.9504955 MHz

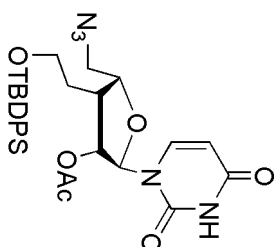
DATA PROCESSING

FT size 16384

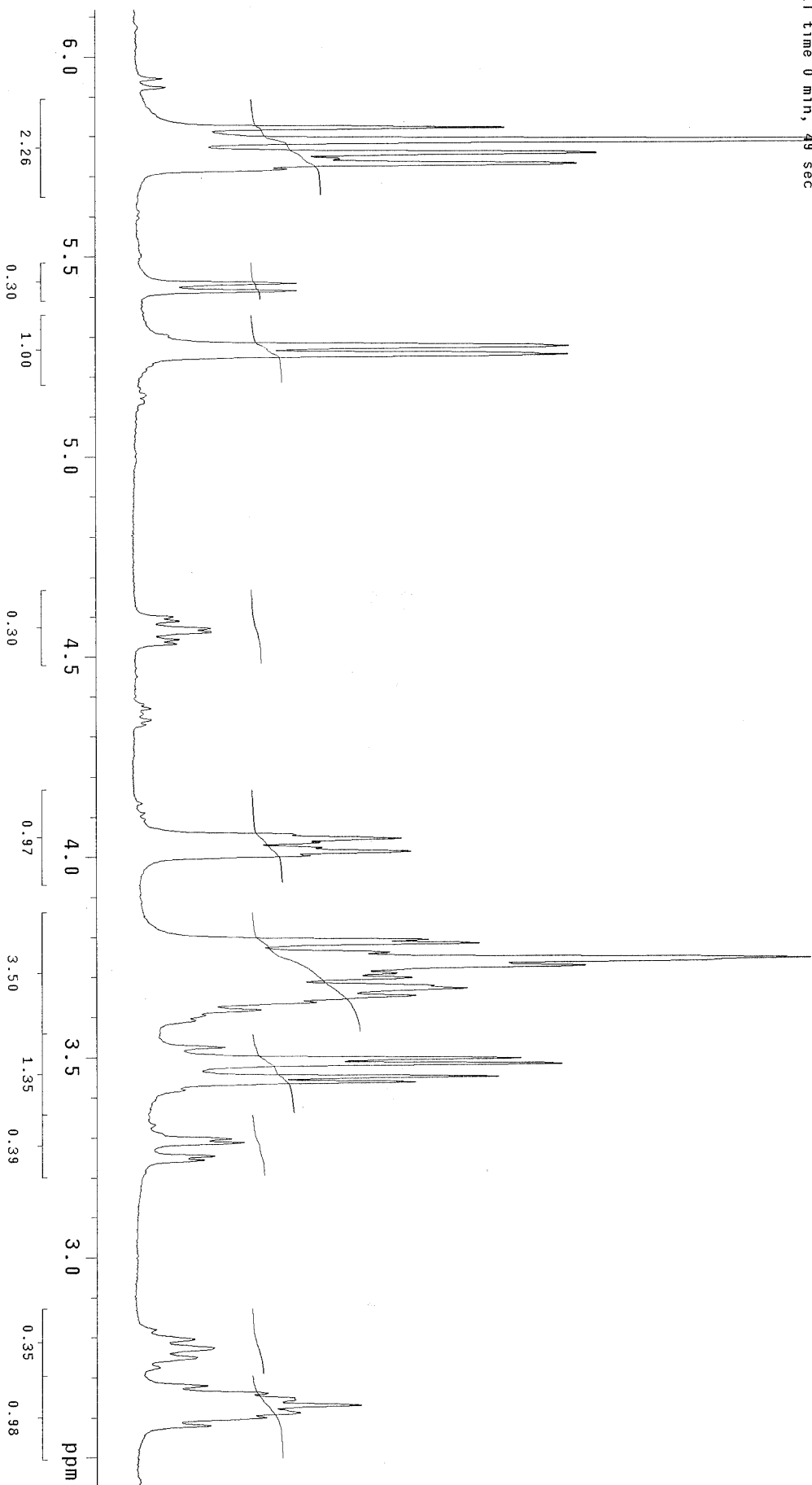
Total time 0 min, 49 sec



Major **12a**



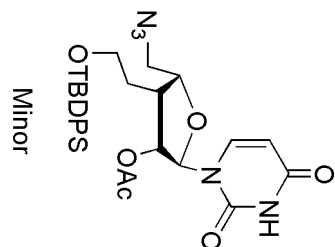
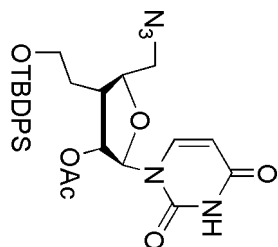
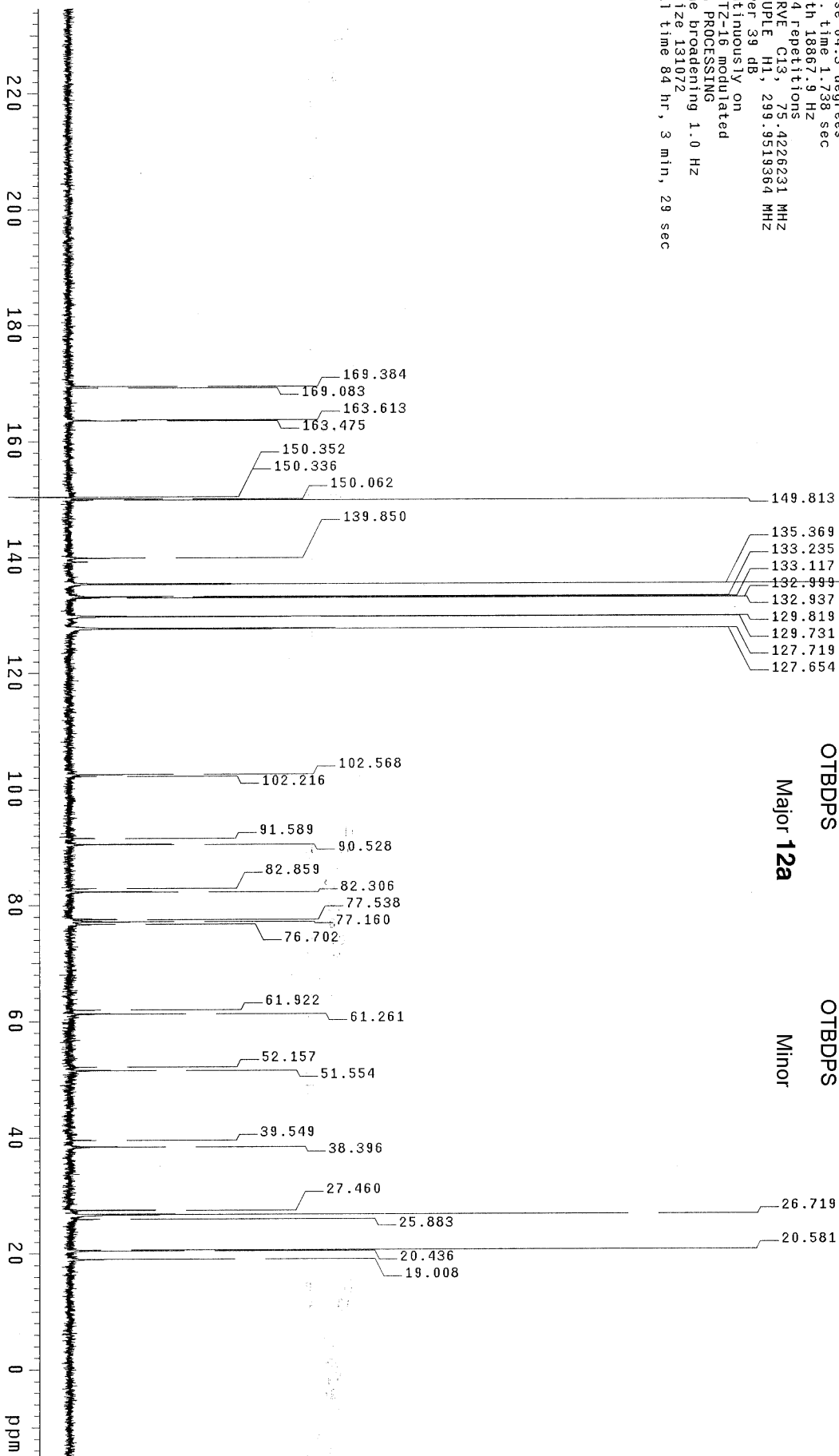
Minor



07/12/04/Monday
YL-03-87-2
After SGC

Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
File: YL-03-87-2-C
Mercury-300 "nmr-300"

PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 64.3 degrees
Acq. time 1.738 sec
Width 18667.9 Hz
1104 repetitions
OBSERVE G13, 75.4226231 MHz
DECOUPLE H1, 299.9519364 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 84 hr, 3 min, 29 sec



07/21/04/Wed.
YL-03-94-1
After SGC

Pulse Sequence: s2pu1

Solvent: CDC13
Ambient temperature
Mercury-300 "nmr300"

PULSE SEQUENCE

Relax. delay 1.000 sec
Pulse 65.6 degrees

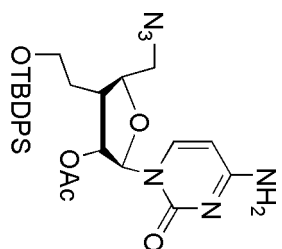
Acq. time 1.994 sec

Width 3898.6 Hz
16 repetitions

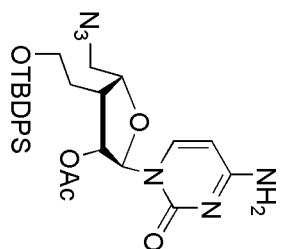
10 repetitions
OBSERVE H1, 299.9504950 MHz

FT size 16384

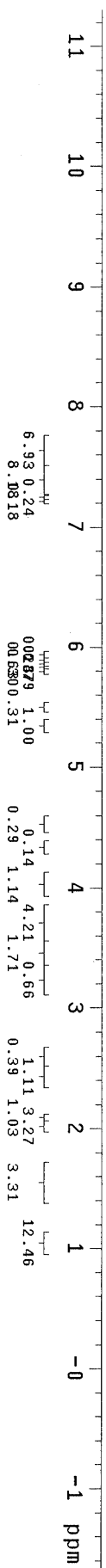
Total time 0 min, 49 sec



Major 12b'



Minor



07/21/04/Wed.
YL-03-94-1
After SGC

Pulse Sequence: s2pu1

Solvent: CDCl3
Ambient temperature
Mercury-300 "nmr300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.994 sec

Width 3898.6 Hz

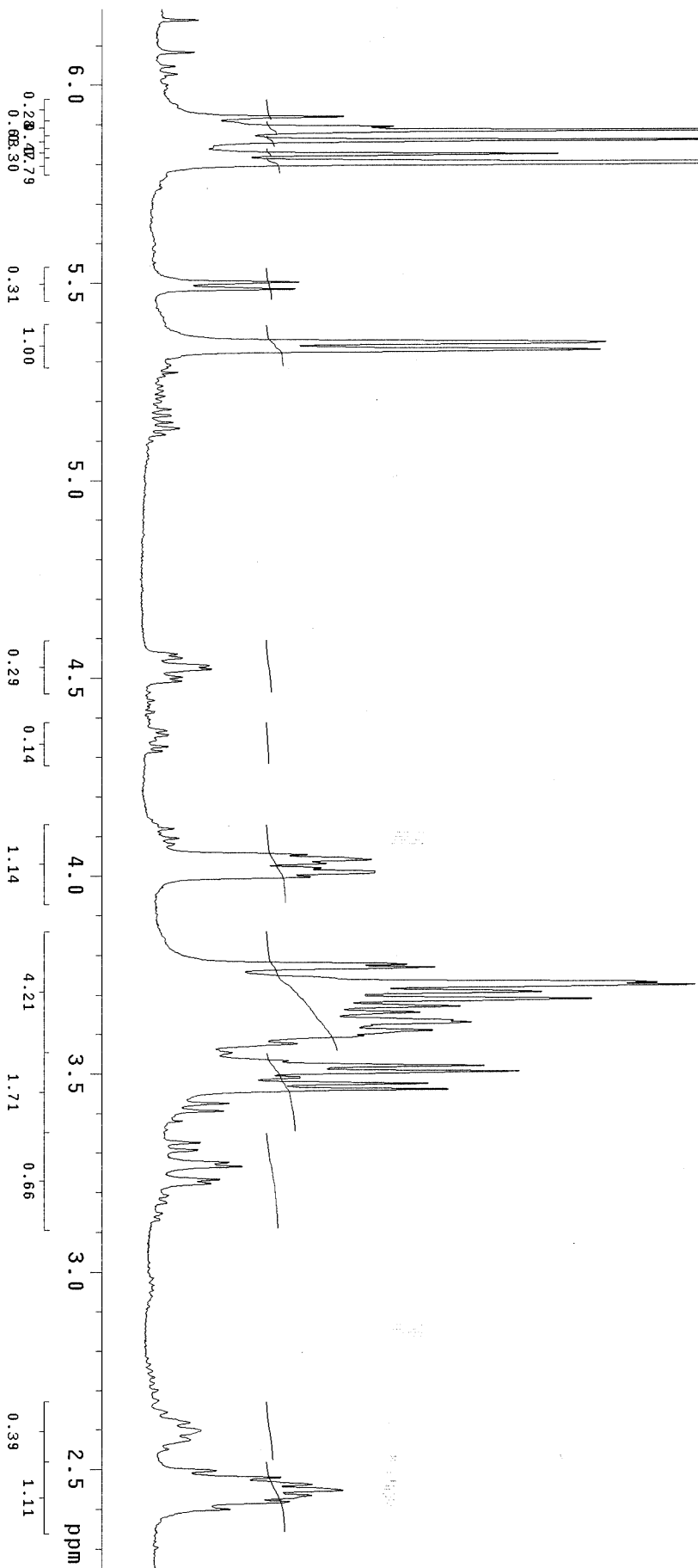
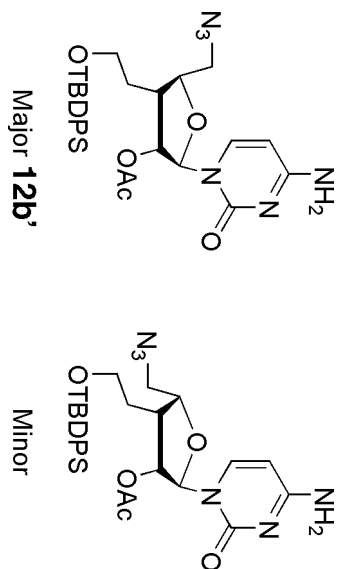
16 repetitions

ORSEVYE H1

DATA PROCESSING

FT size 16384

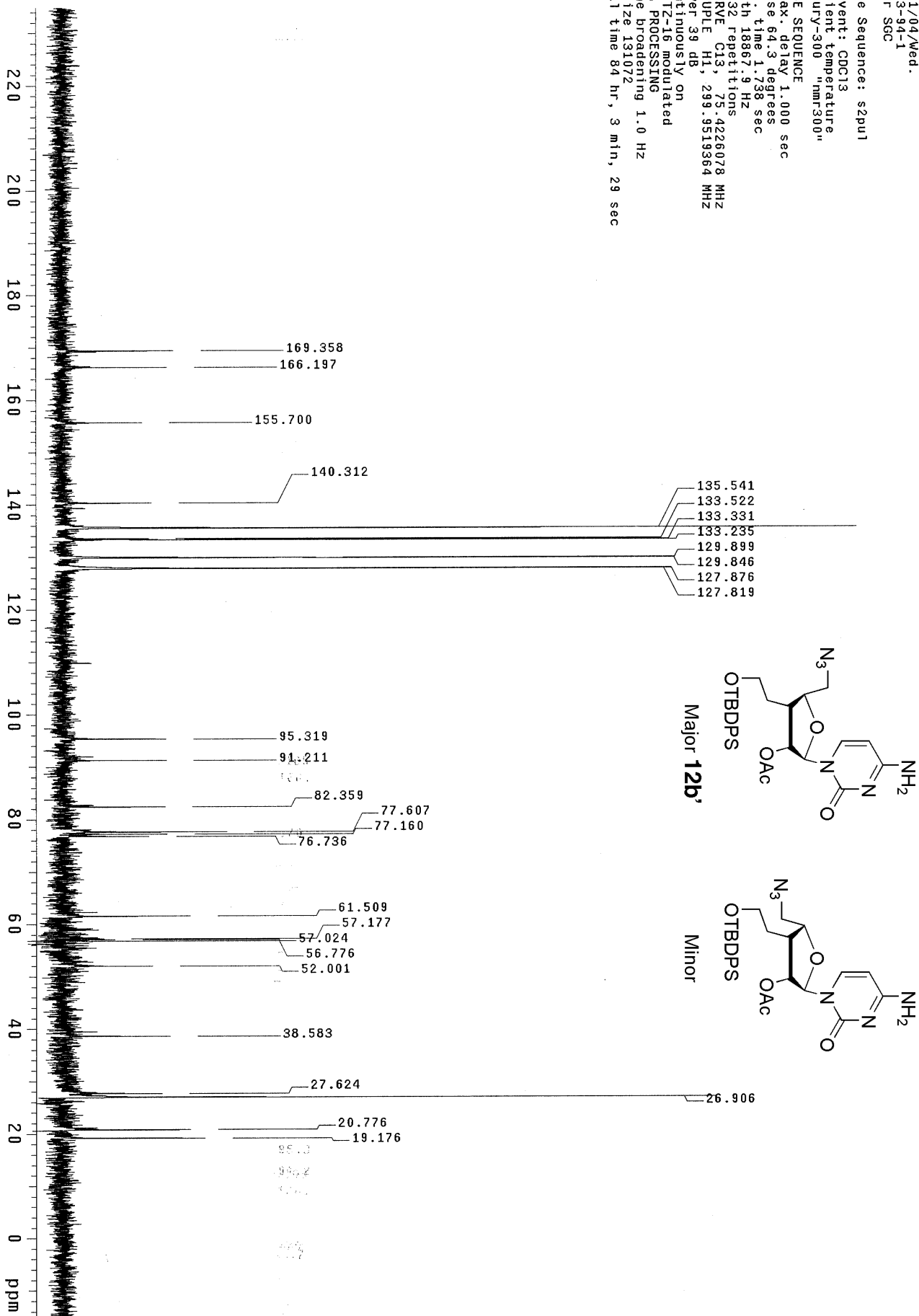
Total time 0 min, 49 sec



07/21/04/Wed.
YL-03-94-1
After SGC

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300 "nmr300"

PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 64.3 degrees
Acq. time 1.738 sec
Width 16867.9 Hz
19232 repetitions
OBSERVE C13, 75.4226078 MHz
DECOUPLE H1, 299.9519364 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 84 hr, 3 min, 29 sec



08/21/04 Saturday
YL-03-107-1
After SGC

Pulse Sequence: s2pu1

Solvent: CDCl3
Ambient temperature
Mercury-300 "nmr-300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.994 sec

Width 3898.6 Hz

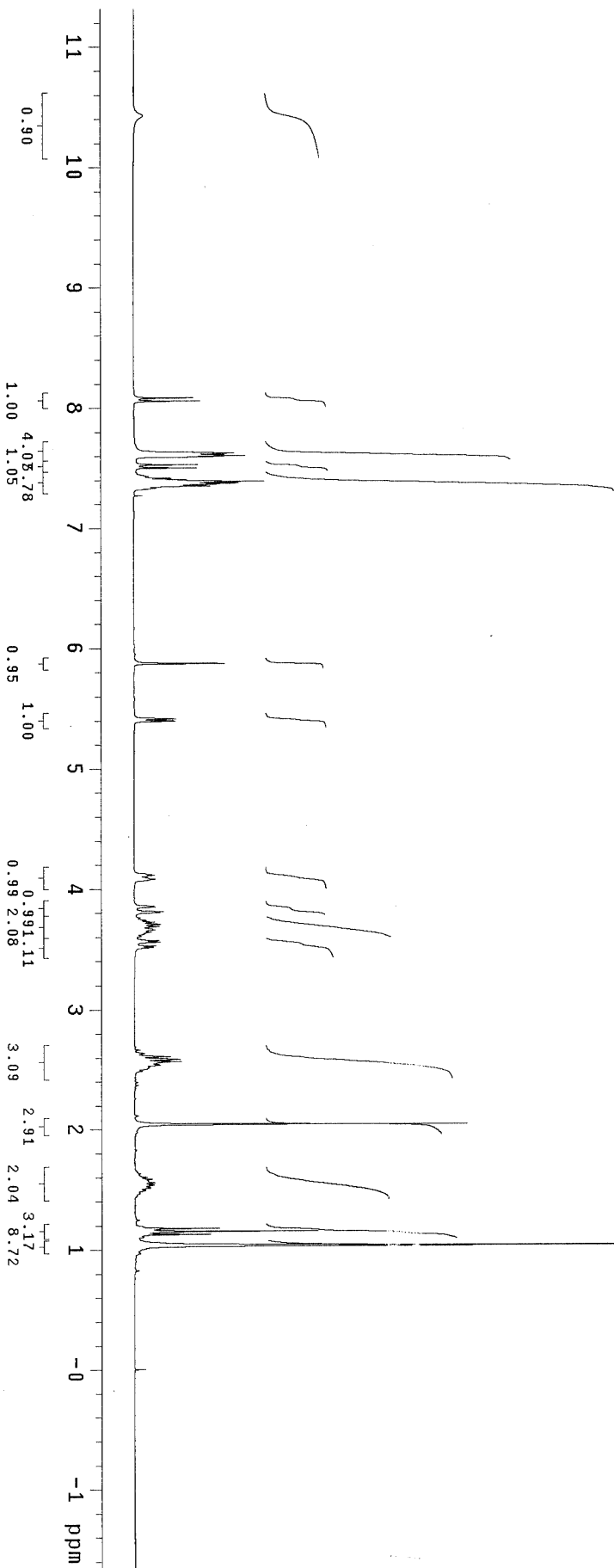
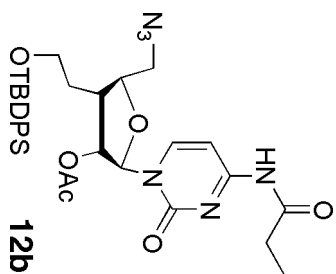
16 repetitions

ORSERVE H1 299.9504907 MHz

DATA PROCESSING

FT size 16384

Total time 0 min, 49 sec



08/21/04 Saturday
YL-03-107-1
After SGC

Pulse Sequence: s2pu1

Solvent: CDC13
Ambient temperature
Mercury-300 "nmr300"

PULSE SEQUENCE

Relax. delay 1.000 sec
Pulse 65.6 degrees

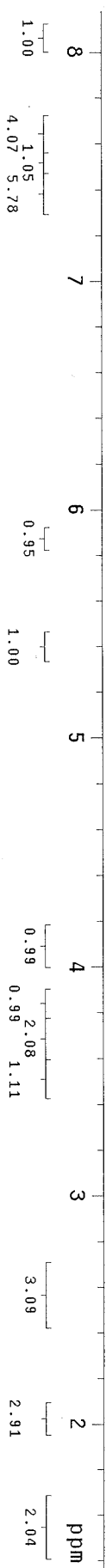
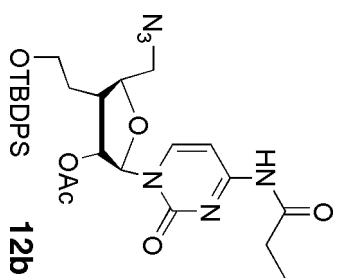
Acq. time 1.994 sec
Width 3898.6 Hz

16 repetitions

OBJECTIVE	UNIT	DATE	INITIALS
DATA PROCESSING			

FT size 16384

Total time 0 min, 49 sec



08/21/04/Saturday
YL-03-107-1
After SGC

Pulse Sequence: s2pu1

Solvent: CDCl3
Ambient temperature
Mercury-300 "nmr300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 64.3 degrees

Acq. time 1.738 sec

Width 18867.9 Hz

19984 repetitions

OBSERVE C13, 75.4226150 MHz

DECOUPLE H1, 299.9519364 MHz

Power 39 db

continuously on

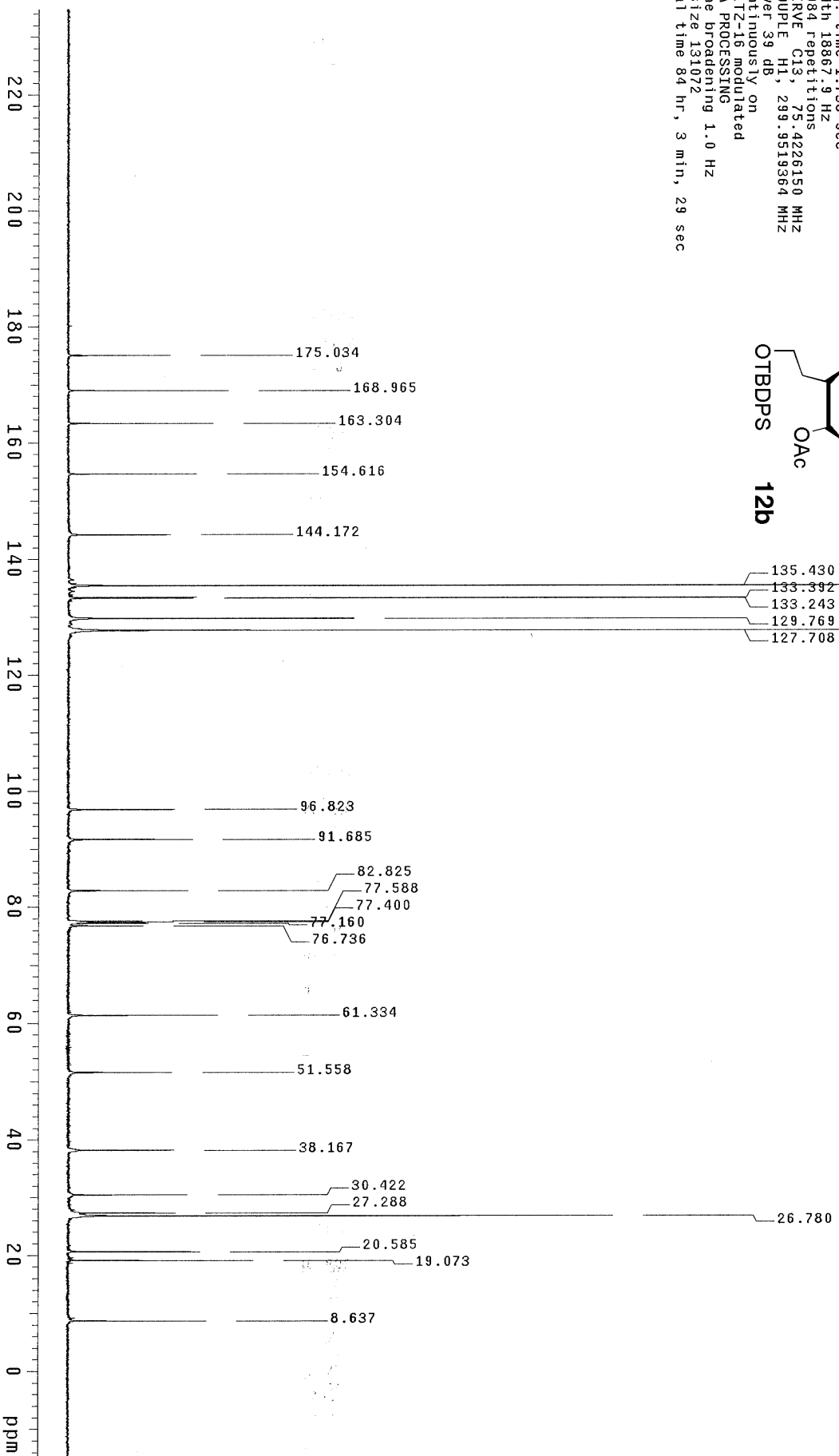
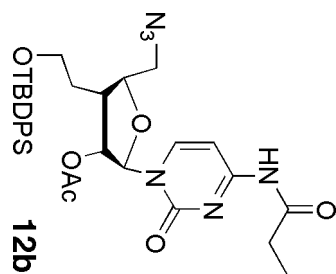
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

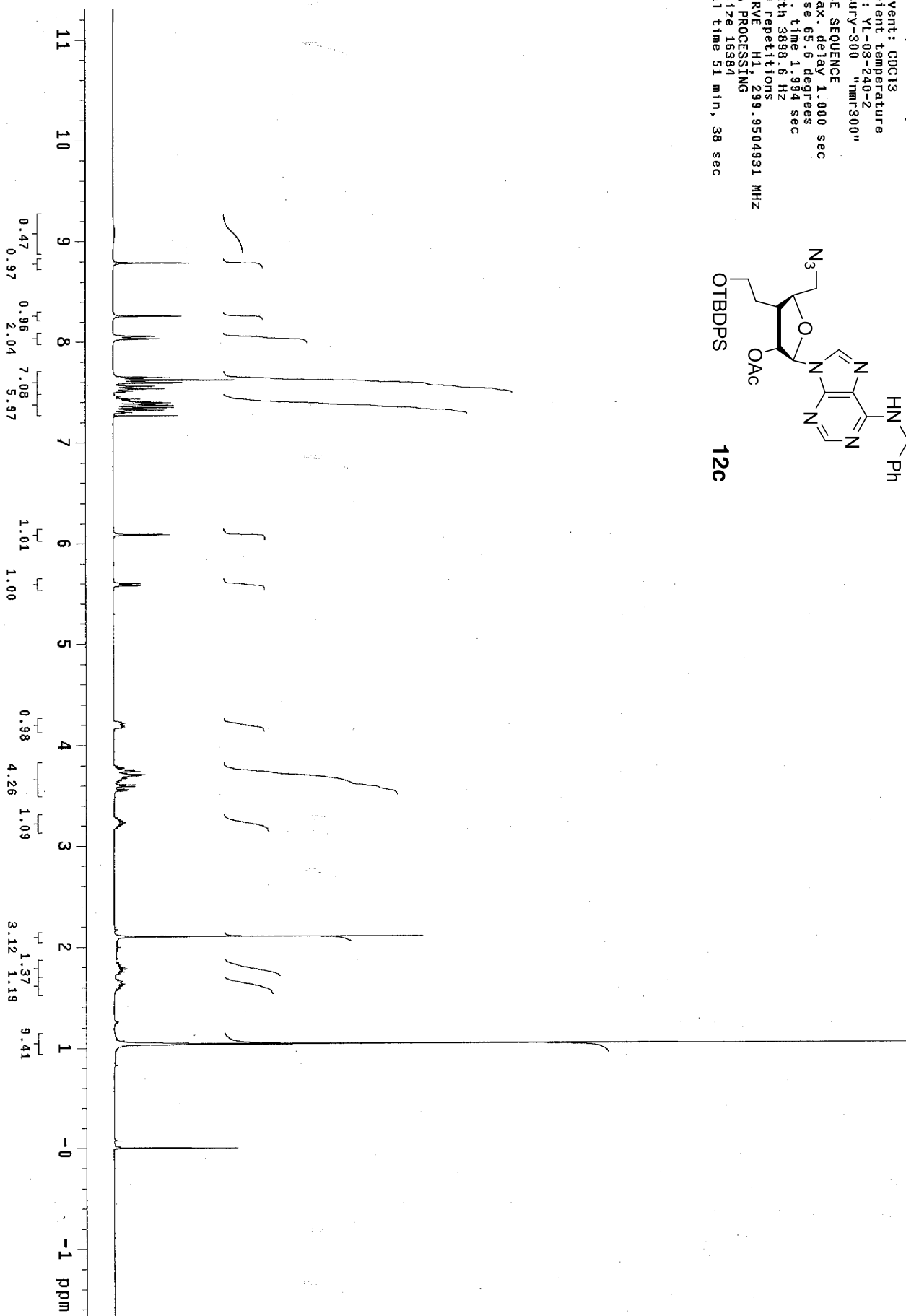
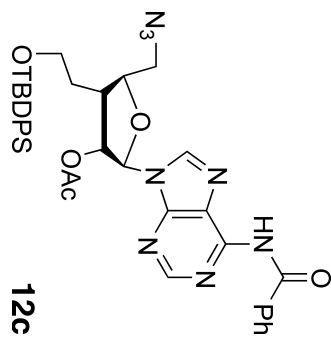
Total time 84 hr, 3 min, 29 sec



06/03/05/Friday
YL-03-240-2
After SGC

Pulse Sequence: szpu1
Solvent: CDCl3
Ambient temperature
File: YL-03-240-2
Mercury-300 1mm300"

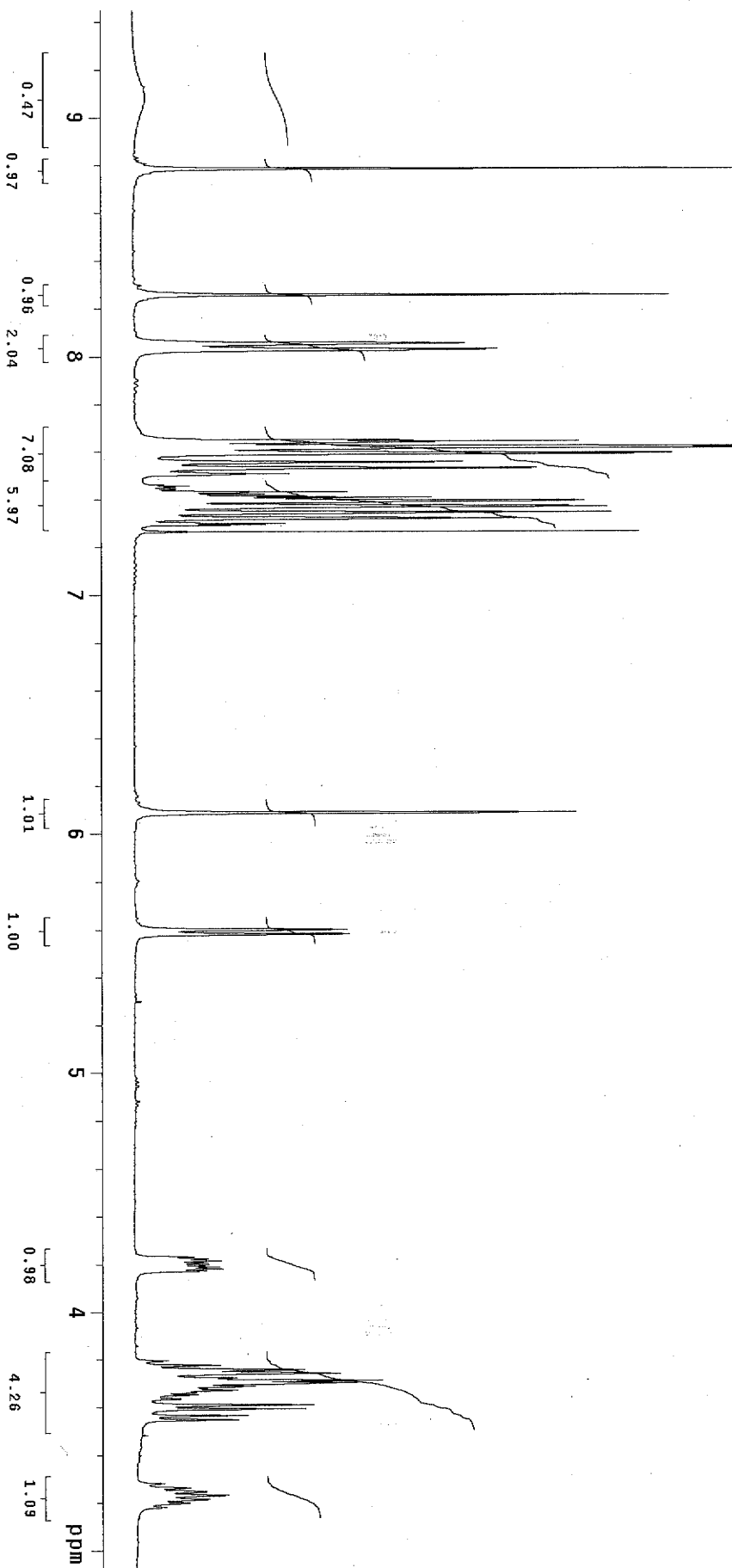
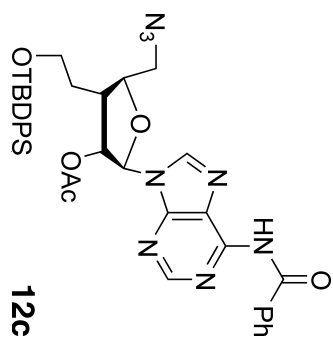
RELAX. SEQUENCE
Relax. delay 1.000 sec
Pulse 65.6 degrees
Acq. time 1.394 sec
Width 3888.6 Hz
160 repetitions
OBSERVE H1; 299.9504931 MHz
DATA PROCESSING
FI size 16384
Total time 51 min, 38 sec



06/03/05/Friday
YL-03-240-2
After SGC

Pulse Sequence: szpu1
Solvent: CDCl3
Ambient temperature
File: YL-03-240-2
Mercury-300 "nmr300"

PULSE SEQUENCE
Relax: delay 1.000 sec
Pulse: 65.0 degrees
Acq. time 1.994 sec
Width 3898.6 Hz
160 repetitions
OBSERVE H1; 299.9504931 MHz
DATA PROCESSING
FI size 16384
Total time 51 min, 38 sec



06/04/05/Sater.
YL-03-240

Pulse Sequence: szpu1

Solvent: CDCl3

Ambient temperature

File: YL-03-240

Mercury-300 "nmr300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 64.3 degrees

Acq. time 1.738 sec

Width 18867.9 Hz

20688 repetitions

OBSERVE C13, 75.4226015 MHz

DECOUPLE H1, 299.9519364 MHz

Power 39 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FI size 131072

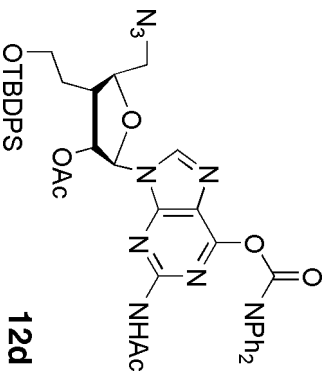
Total time 840 hr, 26 min, 36 sec

Handwritten notes: 0.15-0.16, 0.17-0.18, 0.19-0.20, 0.21-0.22, 0.23-0.24, 0.25-0.26, 0.27-0.28, 0.29-0.30, 0.31-0.32, 0.33-0.34, 0.35-0.36, 0.37-0.38, 0.39-0.40, 0.41-0.42, 0.43-0.44, 0.45-0.46, 0.47-0.48, 0.49-0.50, 0.51-0.52, 0.53-0.54, 0.55-0.56, 0.57-0.58, 0.59-0.60, 0.61-0.62, 0.63-0.64, 0.65-0.66, 0.67-0.68, 0.69-0.70, 0.71-0.72, 0.73-0.74, 0.75-0.76, 0.77-0.78, 0.79-0.80, 0.81-0.82, 0.83-0.84, 0.85-0.86, 0.87-0.88, 0.89-0.90, 0.91-0.92, 0.93-0.94, 0.95-0.96, 0.97-0.98, 0.99-1.00, 1.01-1.02, 1.03-1.04, 1.05-1.06, 1.07-1.08, 1.09-1.10, 1.11-1.12, 1.13-1.14, 1.15-1.16, 1.17-1.18, 1.19-1.20, 1.21-1.22, 1.23-1.24, 1.25-1.26, 1.27-1.28, 1.29-1.30, 1.31-1.32, 1.33-1.34, 1.35-1.36, 1.37-1.38, 1.39-1.40, 1.41-1.42, 1.43-1.44, 1.45-1.46, 1.47-1.48, 1.49-1.50, 1.51-1.52, 1.53-1.54, 1.55-1.56, 1.57-1.58, 1.59-1.60, 1.61-1.62, 1.63-1.64, 1.65-1.66, 1.67-1.68, 1.69-1.70, 1.71-1.72, 1.73-1.74, 1.75-1.76, 1.77-1.78, 1.79-1.80, 1.81-1.82, 1.83-1.84, 1.85-1.86, 1.87-1.88, 1.89-1.90, 1.91-1.92, 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08/06/04/Friday
YL-03-101-2
After SGC

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Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
Mercury-300 "nmr300"
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PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 65.6 degrees
Acq. time 1.994 sec
Width 3898.6 Hz
16 repetitions
OBSERVE H1, 299.9504955 MHz
DATA PROCESSING
FT size 16384
Total time 0 min, 49 sec
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08/06/04/Friday
YL-03-101-2
After SGC

Pulse Sequence: s2pu1

Solvent: CDCl₃
Ambient temperature
Mercury-300 "nmr300"

PULSE SEQUENCE

Relax. delay 1.000 sec
Pulse 65.6 degrees

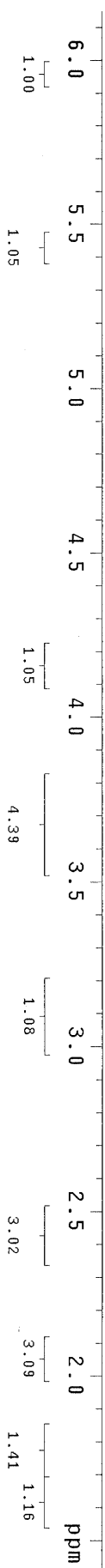
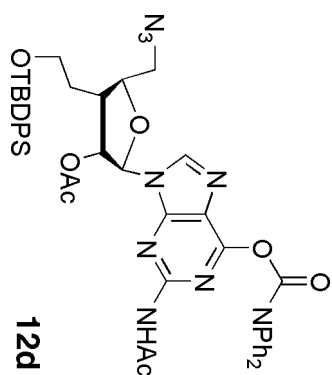
Acq. time 1.994 sec

Width 3898.6 Hz
16 repetitions

TO OBSERVE H1, 299.9504955 MHZ

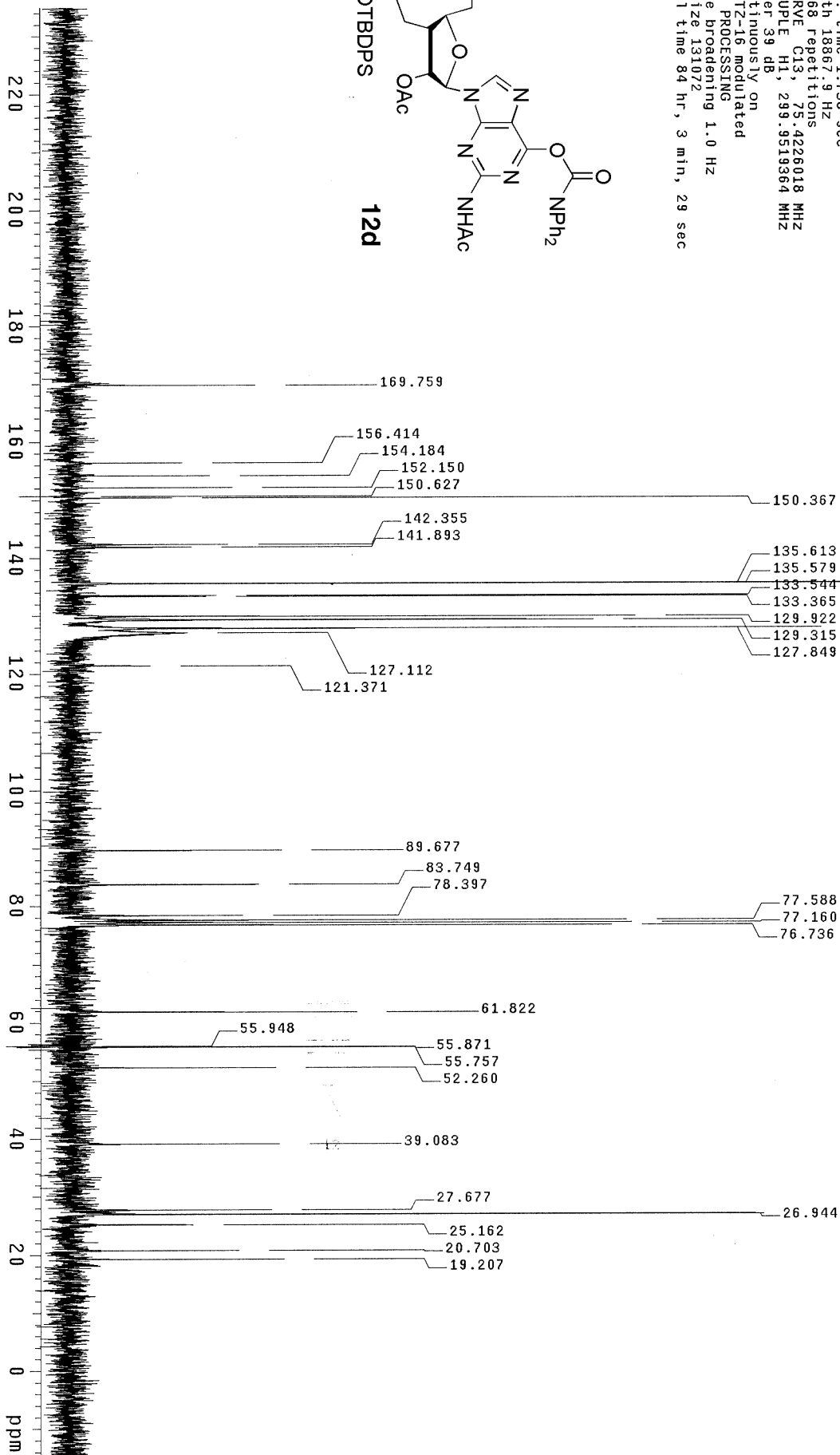
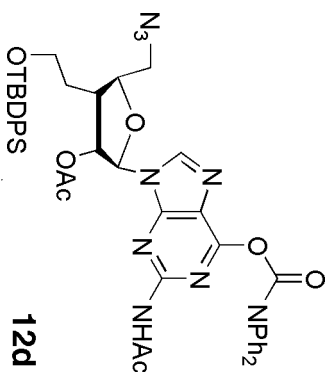
DATA PROCESSING
ET size 16384

Total time 0 min, 49 sec

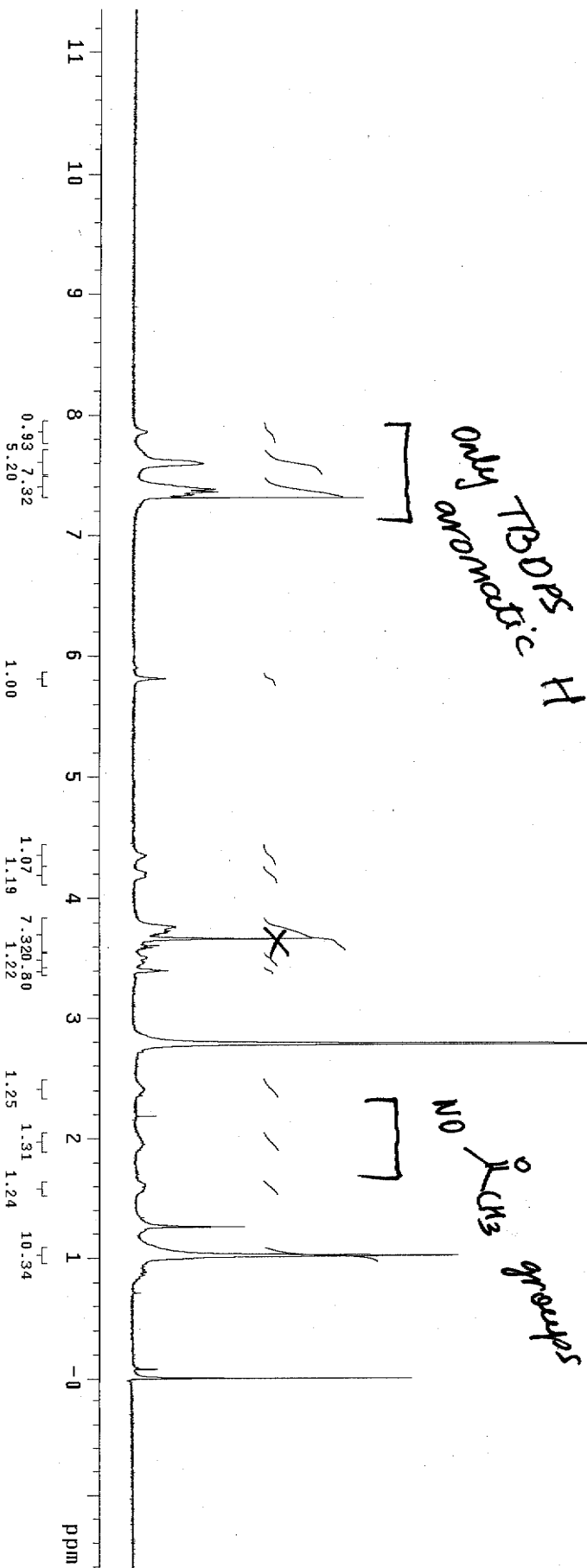
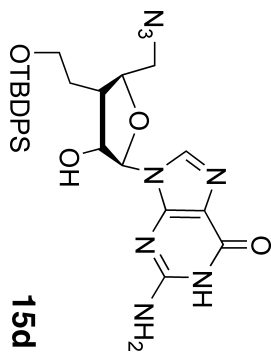


08/10/04/Tuesday
VI-03-101-2
After SGC

Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
Mercury-300 "nmr300"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 64.3 degrees
Acq. time 1.738 sec
Width 18867.9 Hz
18368 repetitions
OBSERVE C13, 75.4226018 MHz
DECOUPLE H1, 299.9519364 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 84 hr, 3 min, 29 sec



05/10/04/Monday
 YL-03-52-23
 CDCl3 + drops of CD3OD
 Pulse Sequence: szpu1
 Solvent: CDCl3
 Ambient temperature
 File: YL-03-52-23-CDCl3CD3OD
 Mercury-300 "nmr300"
 PULSE SEQUENCE
 Relax: delay 1.000 sec
 Pulse: 65.6 degrees
 Acq. time 1.994 sec
 Width 3898.6 Hz
 16 repetitions
 OBSERVE H1, 299.9504788 MHz
 DATA PROCESSING
 FT size 16384
 Total time 0 min, 49 sec



04/13/05/Wed.
YL-03-222ef-1
After HPLC

Pulse Sequence: s2pu1

Solvent: CDCl3

Ambient temperature

File: YL-03-222ef-1

Mercury-300 "nmr300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.994 sec

Width 3898.6 Hz

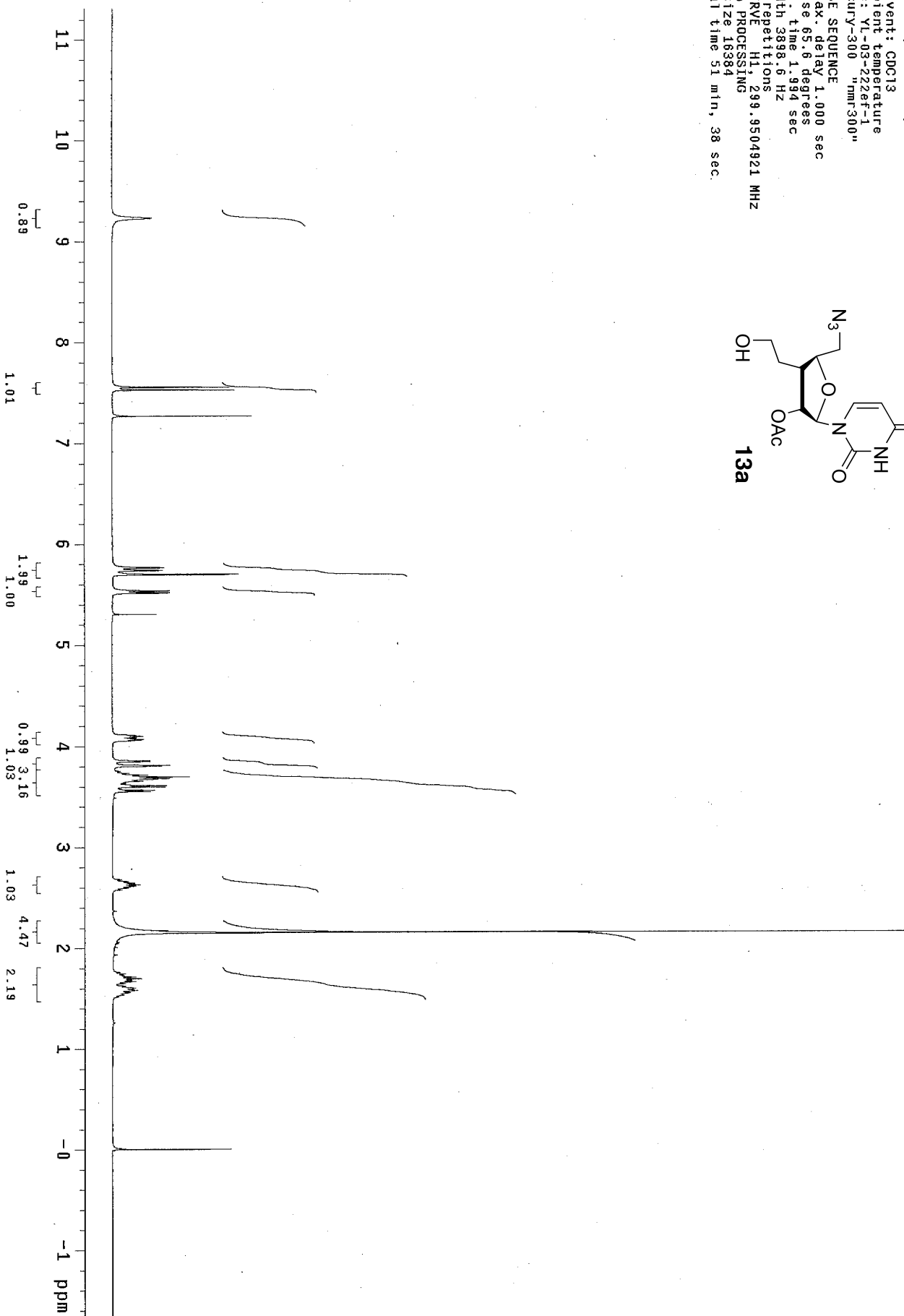
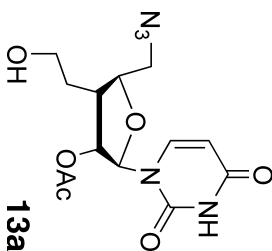
48 repetitions

OBSERVE H1, 299.9504921 MHz

DATA PROCESSING

FT size 16384

Total time 51 min, 38 sec.



04/13/05/Wed.
YL-03-222ef-1
After HPLC

Pulse Sequence: szpu1

Solvent: CDCl3
Ambient temperature
File: YL-03-222ef-1
Mercury-300 "nmr300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.994 sec

Width 3898.6 Hz

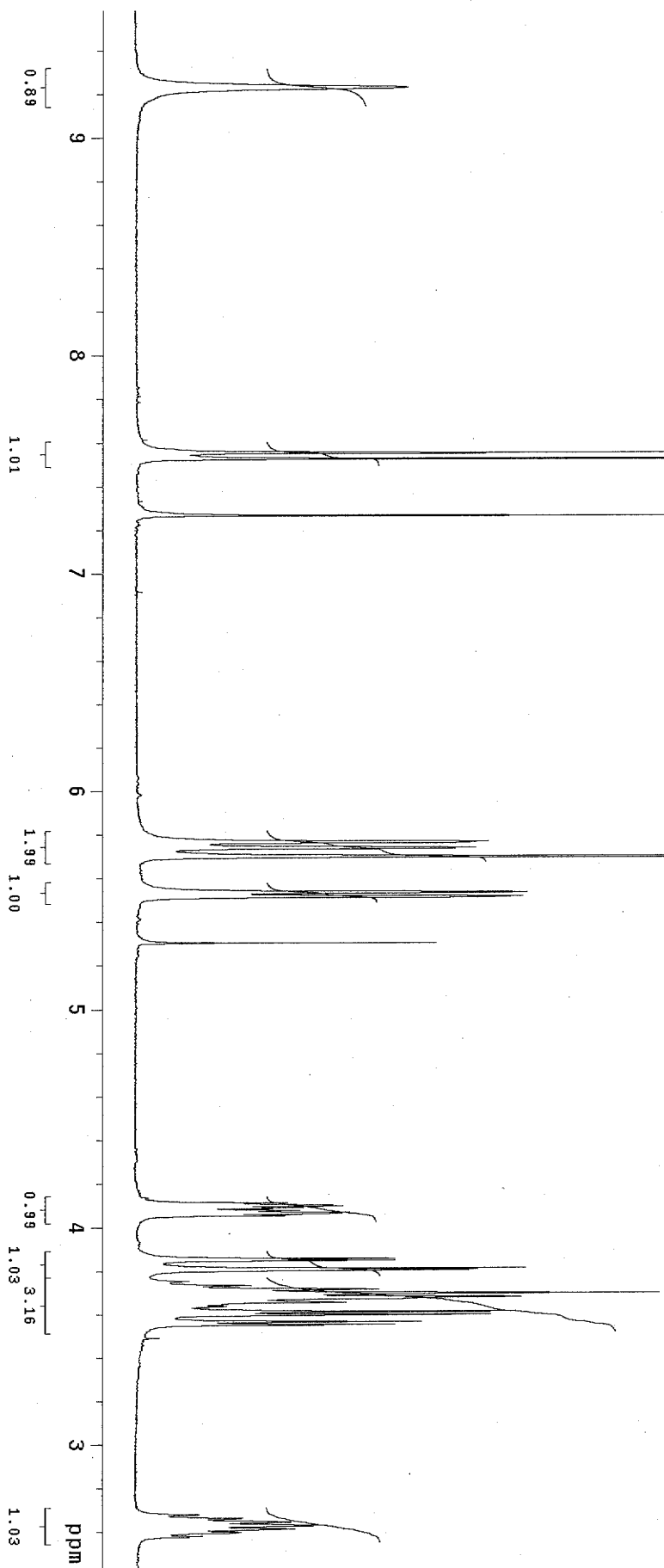
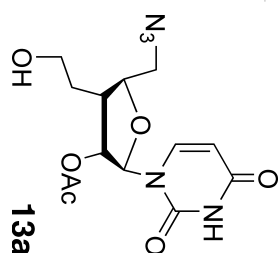
48 repetitions

OBSERVE H1, 299.9504921 MHz

DATA PROCESSING

FT size 16384

Total time 51 min, 38 sec



01/06/05/Thurs.
YL-03-175-trans
After SGC

Pulse Sequence: szpu1

Solvent: CDCl3
Ambient temperature
File: YL-03-175-trans-C
Mercury-300 "nmr300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 64.3 degrees

Acq. time 1.738 sec

Width 18667.9 Hz

19856 repetitions

OBSERVE C13, 75.4226033 MHz

DECOUPLE H1, 299.9519364 MHz

Power 39 dB

continuously on

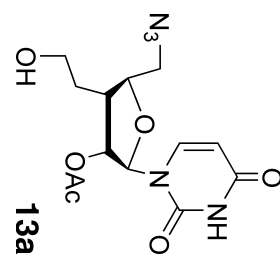
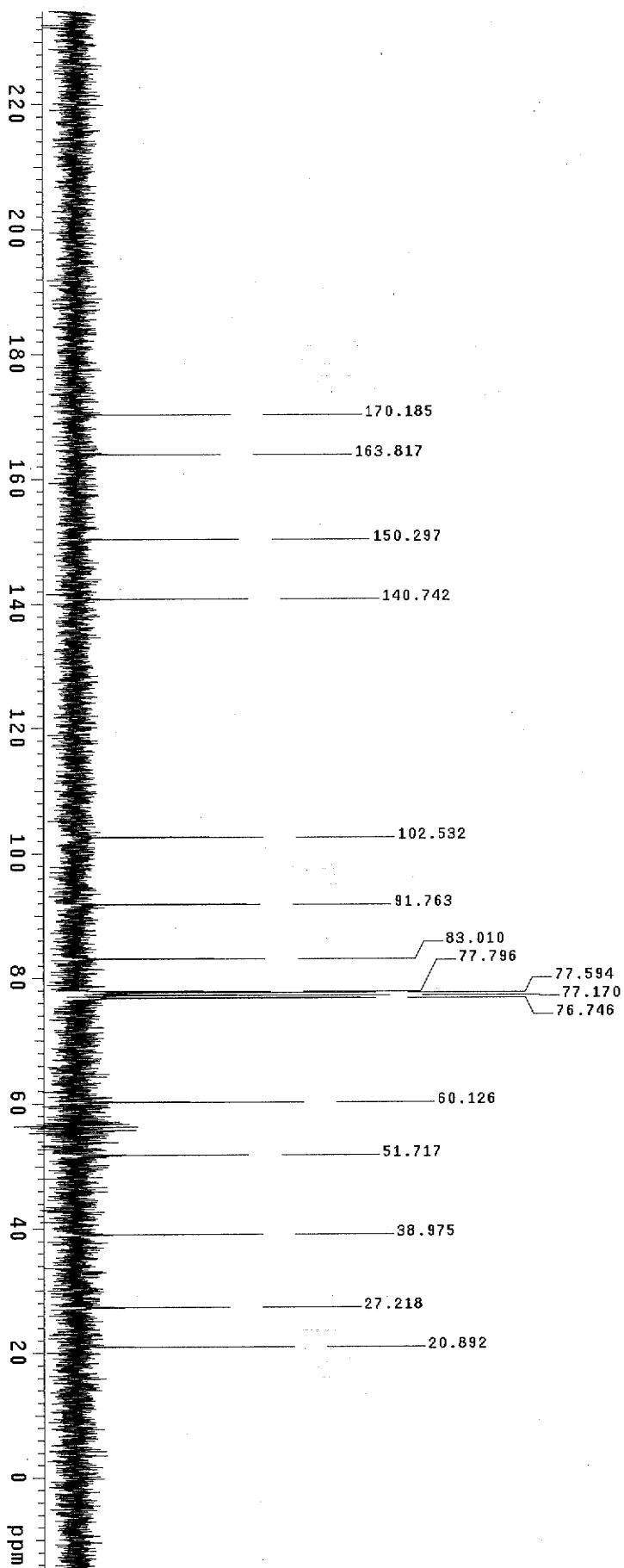
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

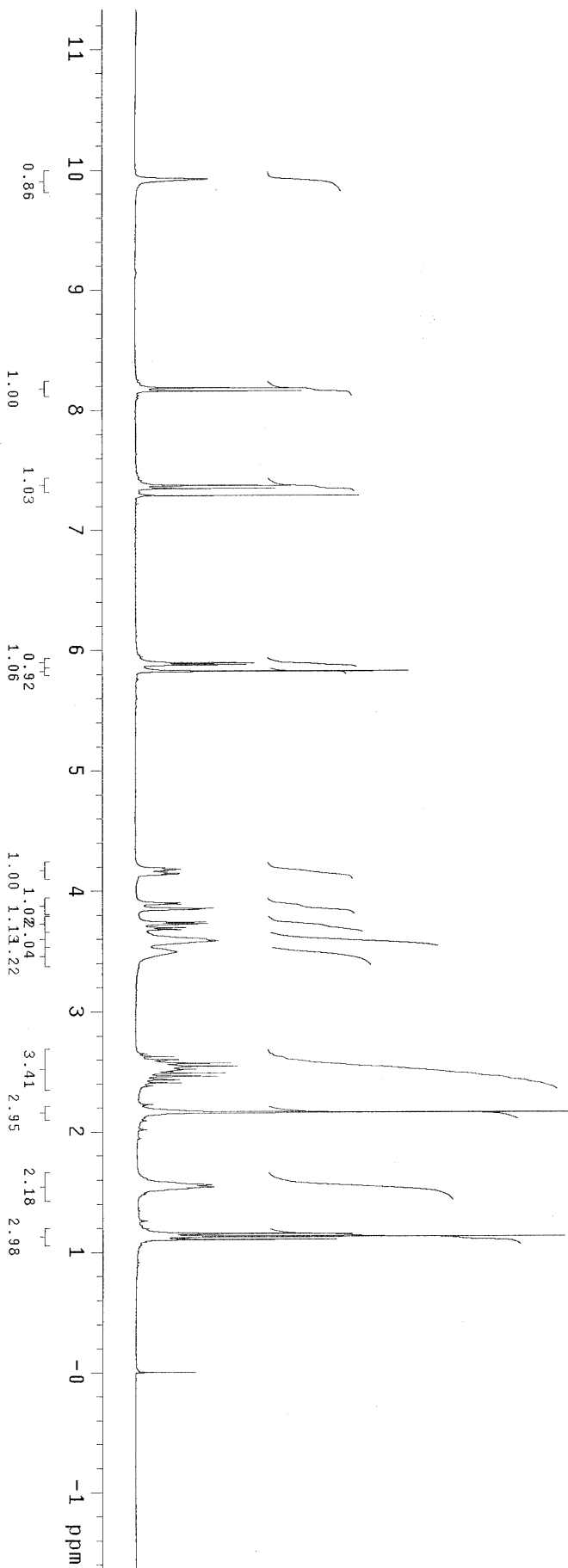
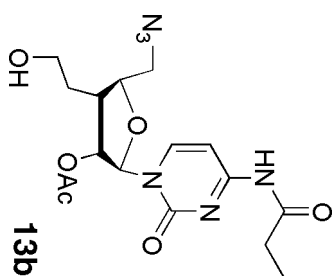
Total time 84 hr, 3 min, 29 sec



10/11/04/Monday
VL-03-116-1
After SGC

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300 "mm300"

PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 65.6 degrees
Acq. time 1.934 sec
Width 3898.6 Hz
16 repetitions
OBSERVE H1, 299.9504859 MHz
DATA PROCESSING
FT size 16384
Total time 0 min, 49 sec



10/11/04/Monday
YL-03-116-1
After SGC

Pulse Sequence: s2pu1

Solvent: CDCl3
Ambient temperature
Mercury-300 "nmr300"

PULSE SEQUENCE

Relax. delay 1.000 sec
Pulse 65.6 degrees

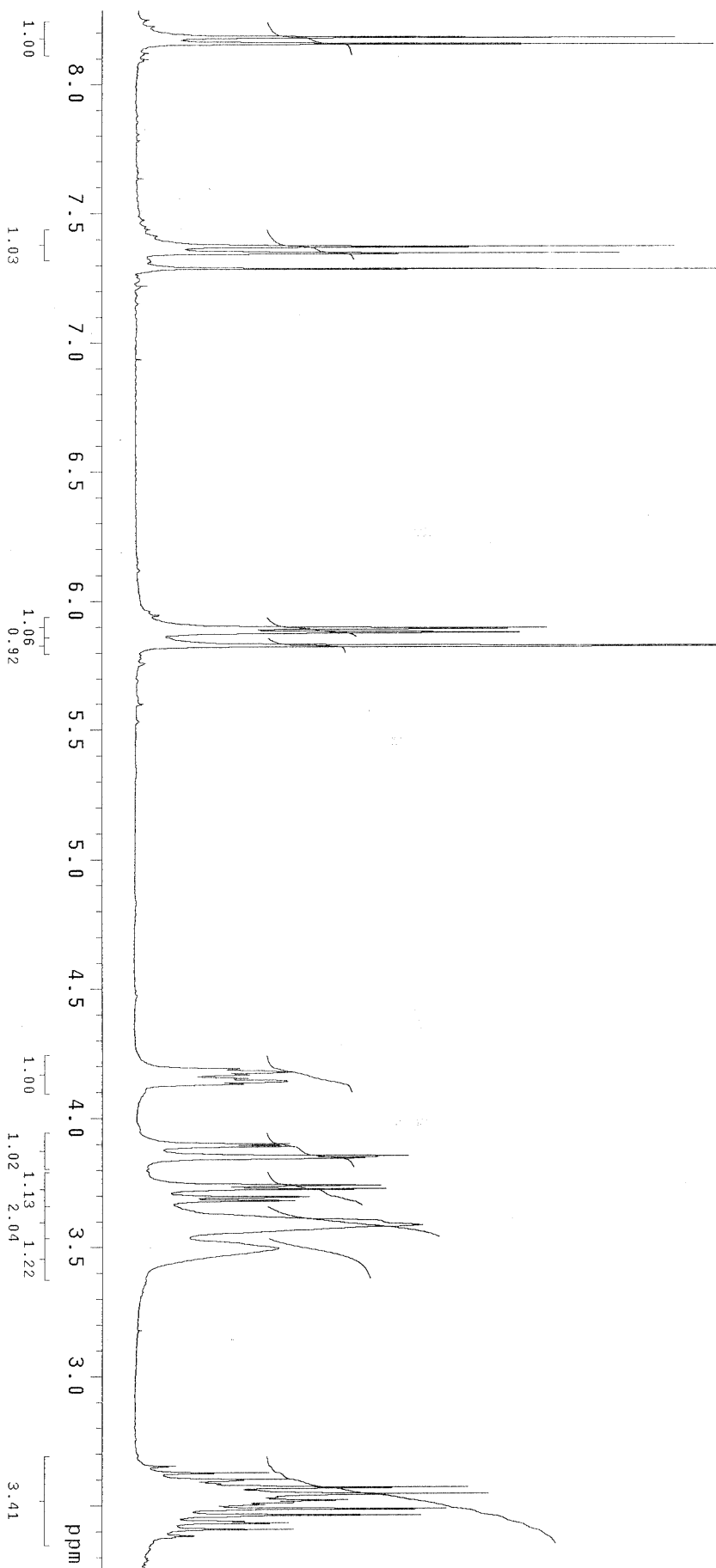
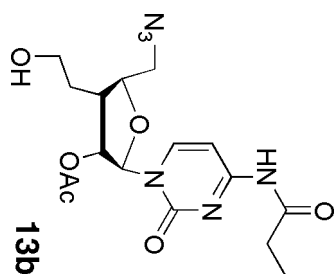
Acq. time 1.994 sec
Width 3898.6 Hz

16 repetitions

OBSERVE H1, 299.9504859 MHz
DATA PROCESSING

FT size 16384

Total time 0 min, 49 sec



10/12/04/Tuesday
VL-03-116-1
After SGC

Pulse Sequence: s2pu1

Solvent: CDCl₃
Ambient temperature
Mercury-300 "nmr-300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 64.3 degrees

Acq. time 1.738 sec

Width 18867.9 Hz

18944 repetitions

OBSERVE C13, 75.4226033 MHz

DECOUPLE H1, 299.9519364 MHz

Power 39 dB

continuously on

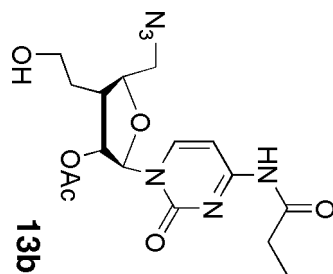
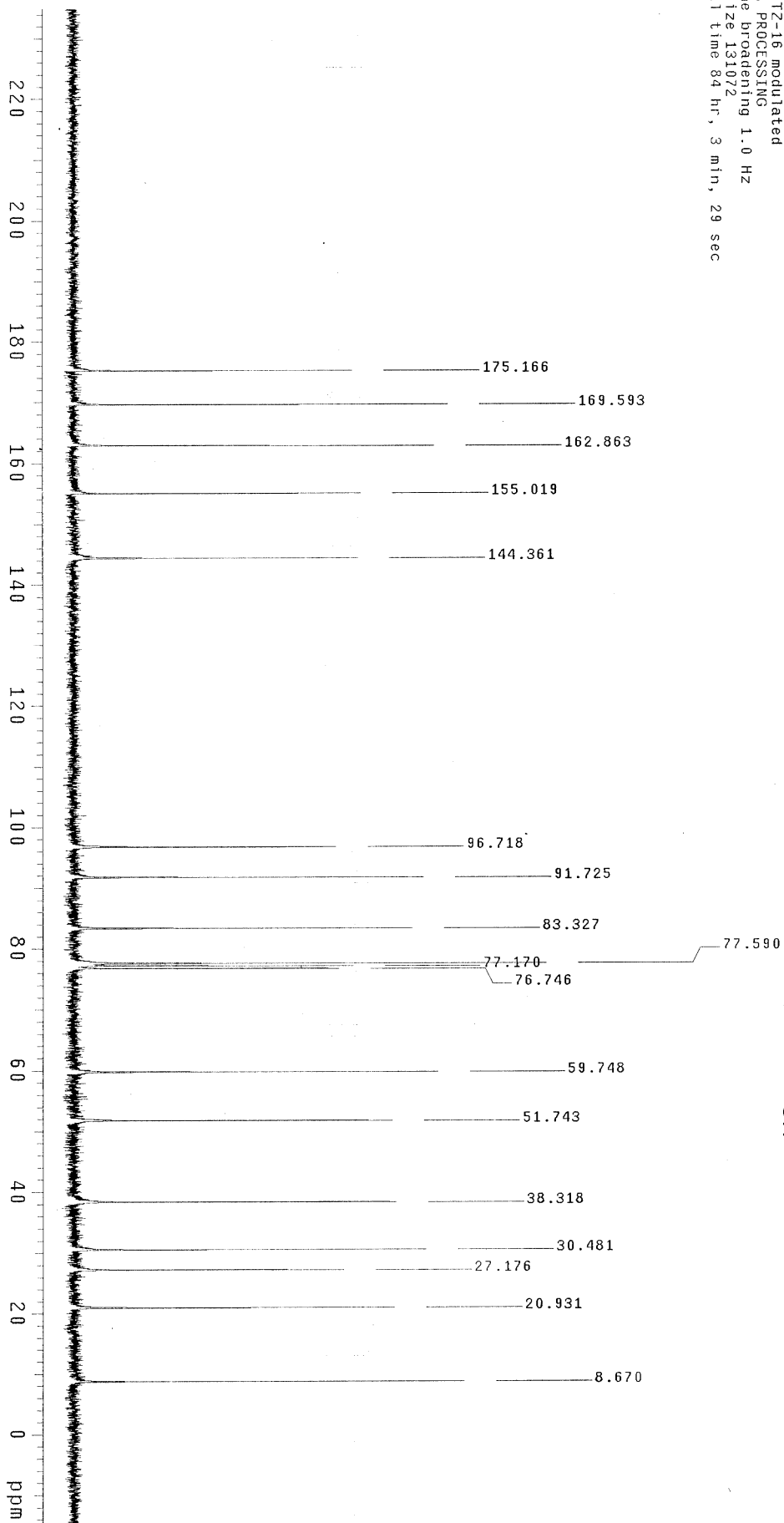
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

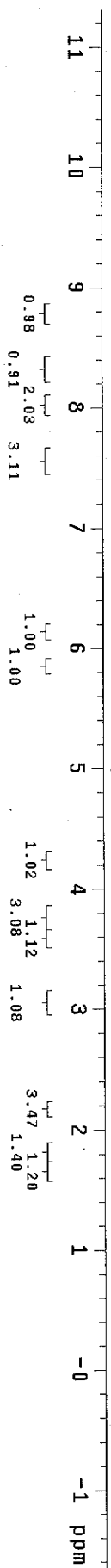
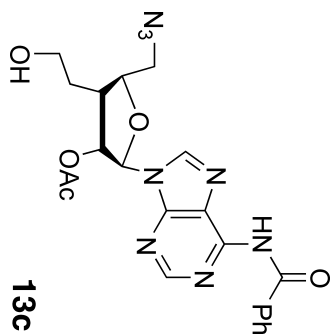
Total time 84 hr, 3 min, 29 sec



06/09/05/Thurs.
YL-03-242-new-3
After SGC

Pulse Sequence: szpul
Solvent: CDCl3
Ambient temperature
File: YL-03-242-3
Mercury-300 "nm"300"

PULSE SEQUENCE
Relax: delay 1.000 sec
Pulse: 65.6 degrees
Acq. time 1.994 sec
Width 3898.6 Hz
224 repetitions
OBSERVE H1, 299.9504926 MHz
DATA PROCESSING
FI size 16384
Total time 51 min, 38 sec



06/09/05/Thurs.
YL-03-242-new-3
After SGC

Pulse Sequence: szpu1

Solvent: CDCl3

Ambient temperature

File: YL-03-242-3

Mercury-300 "nmr300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.994 sec

Width 3898.6 Hz

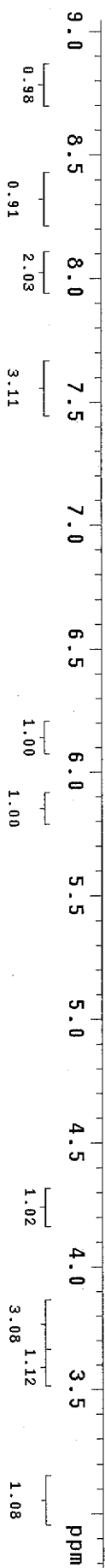
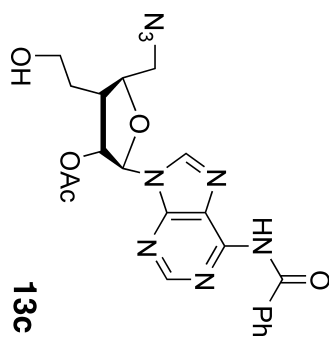
224 repetitions

OBSERVE H1, 299.9504926 MHz

DATA PROCESSING

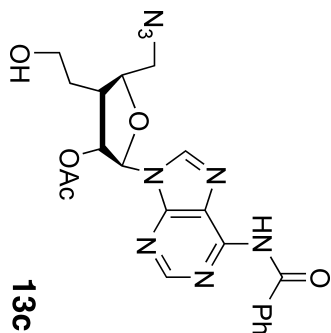
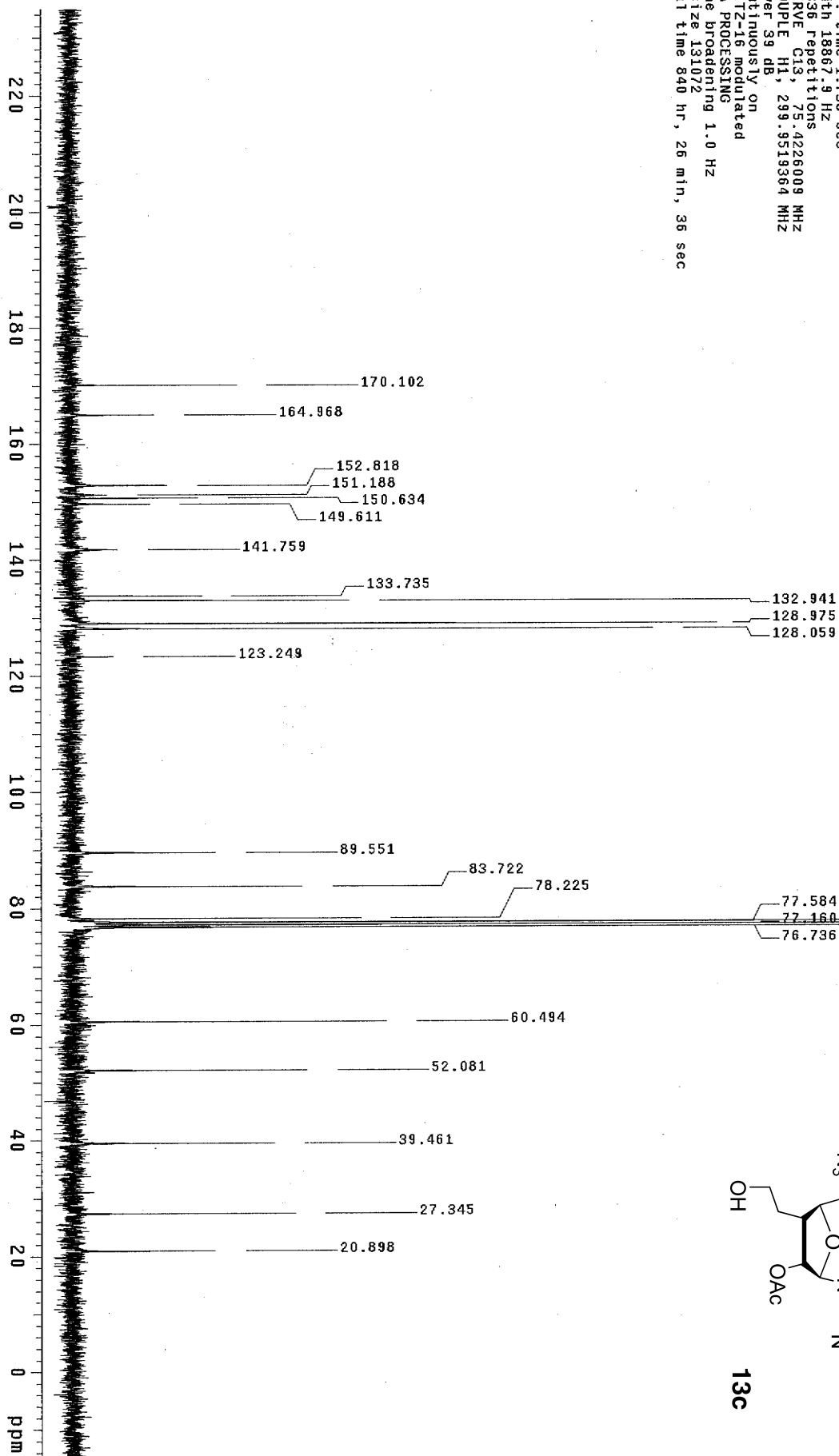
FT size 16384

Total time 51 min, 38 sec



06/09/05/Thurs.
YL-03-241-3
new

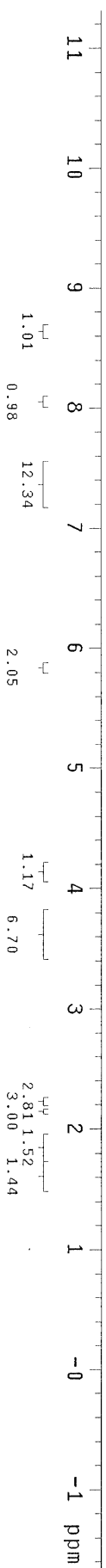
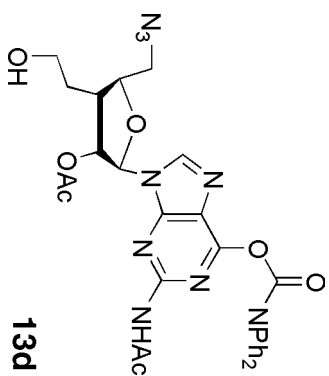
Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
Mercury-300 "nmr-300"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 64.3 degrees
Acq. time 1.738 sec
Width 18867.9 Hz
18336 repetitions
OBSERVE C13, 75.4226009 MHz
DECOUPLE H1, 299.9519364 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 840 hr, 26 min, 36 sec



10/06/04/Wed.
YL-03-117-1
After SGC

Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
Mercury-300 "nmr300"

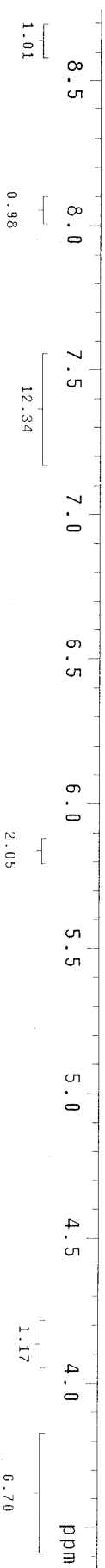
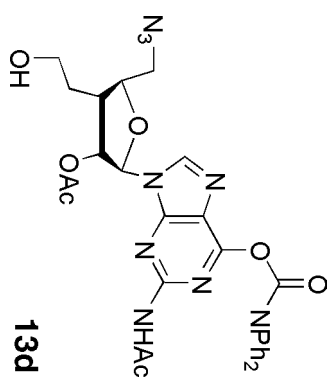
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 65.6 degrees
Acq. time 1.994 sec
Width 3898.6 Hz
16 repetitions
OBSERVE H1, 299.9504926 MHz
DATA PROCESSING
FT size 16384
Total time 0 min, 49 sec



10/06/04/Wed.
YL-03-117-1
After SGC

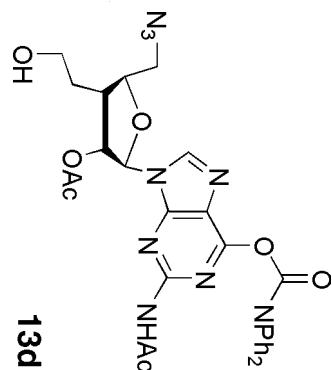
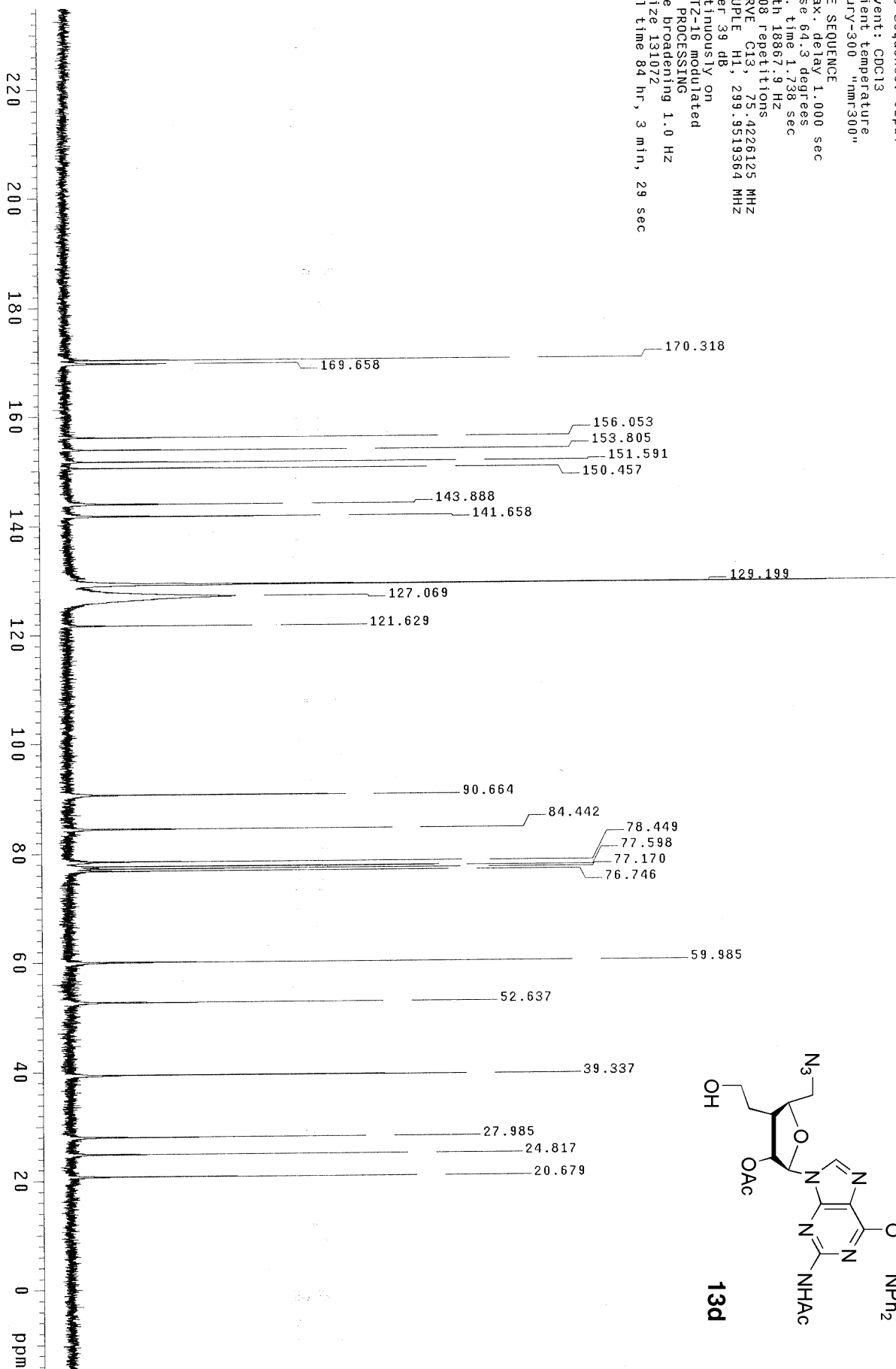
Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
Mercury-300 "mm-300"

RELAX. SEQUENCE
Relax. delay 1.000 sec
Pulse 65.6 degrees
Acq. time 1.994 sec
Width 3898.6 Hz
16 repetitions
OBSERVE H1, 299.9504926 MHz
DATA PROCESSING
FT size 16384
Total time 0 min, 49 sec



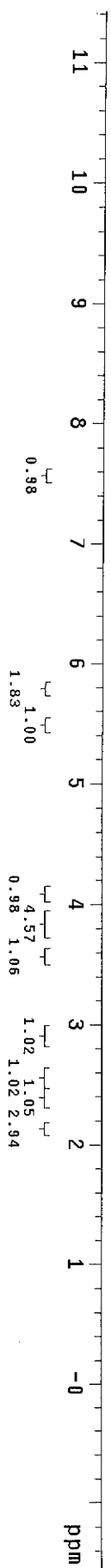
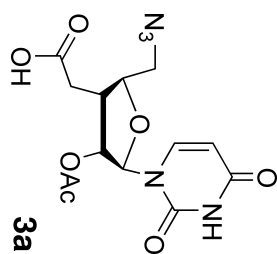
10/07/04/Thursday
YL-03-117-1
After SGC

Pulse Sequence: s2pul
Solvent: CDCl₃
Ambient temperature
Mercury-300 "hmr300"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 64.3 degrees
Acq. time 1.738 sec
Width 18867.9 Hz
15008 repetitions
OBSERVE C13, 75.4226125 MHz
DECOUPLE H1, 299.9519364 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 84 hr, 3 min, 29 sec



05/06/05/Friday
YL-03-224
(6)

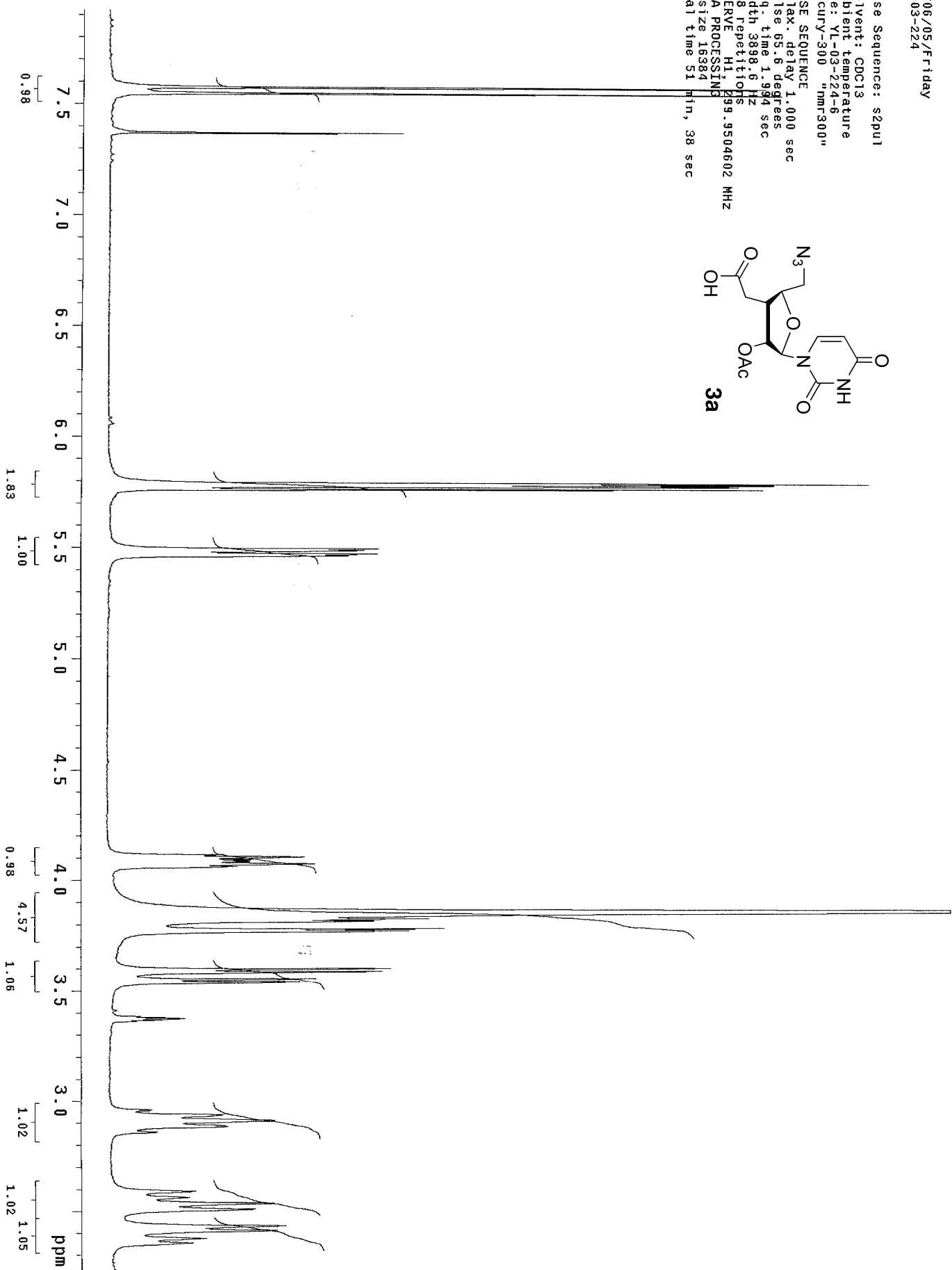
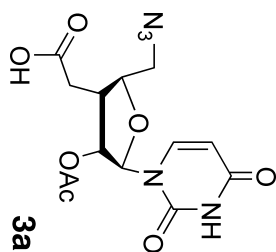
Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
File: YL-03-224-6
Mercury-300 "nmr300"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 65.6 degrees
Acq. time 1.994 sec
Width 3898.6 Hz
128 Repetitions
OBSERVE H1 239.9504602 MHz
DATA PROCESSING
F1 size 16384
Total time 51 min, 38 sec



05/06/05/Friday
YL-03-224
(b)

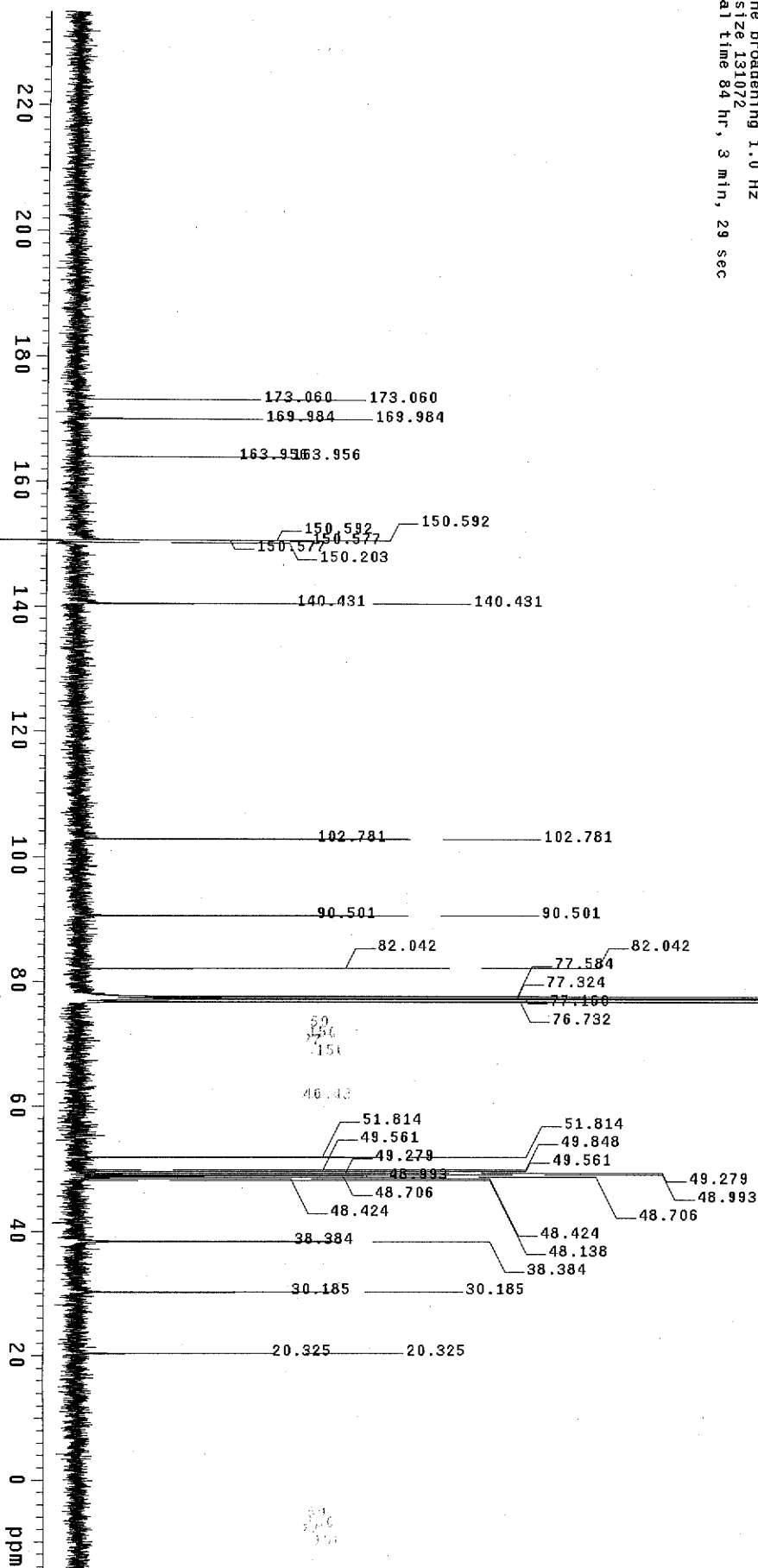
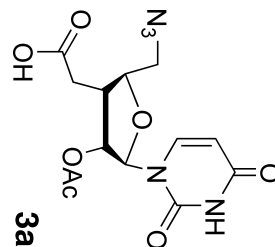
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: YL-03-224-6
Mercury-300 "nm-300"

PULSE SEQUENCE
Relax.: delay 1.000 sec
Pulse: 65.6 degrees
Acq. time 1.984 sec
Width 3898.6 Hz
128 repetitions
OBSERVE H1; 299.9504602 MHz
DATA PROCESSING
FT size 16384
Total time 51 min, 38 sec



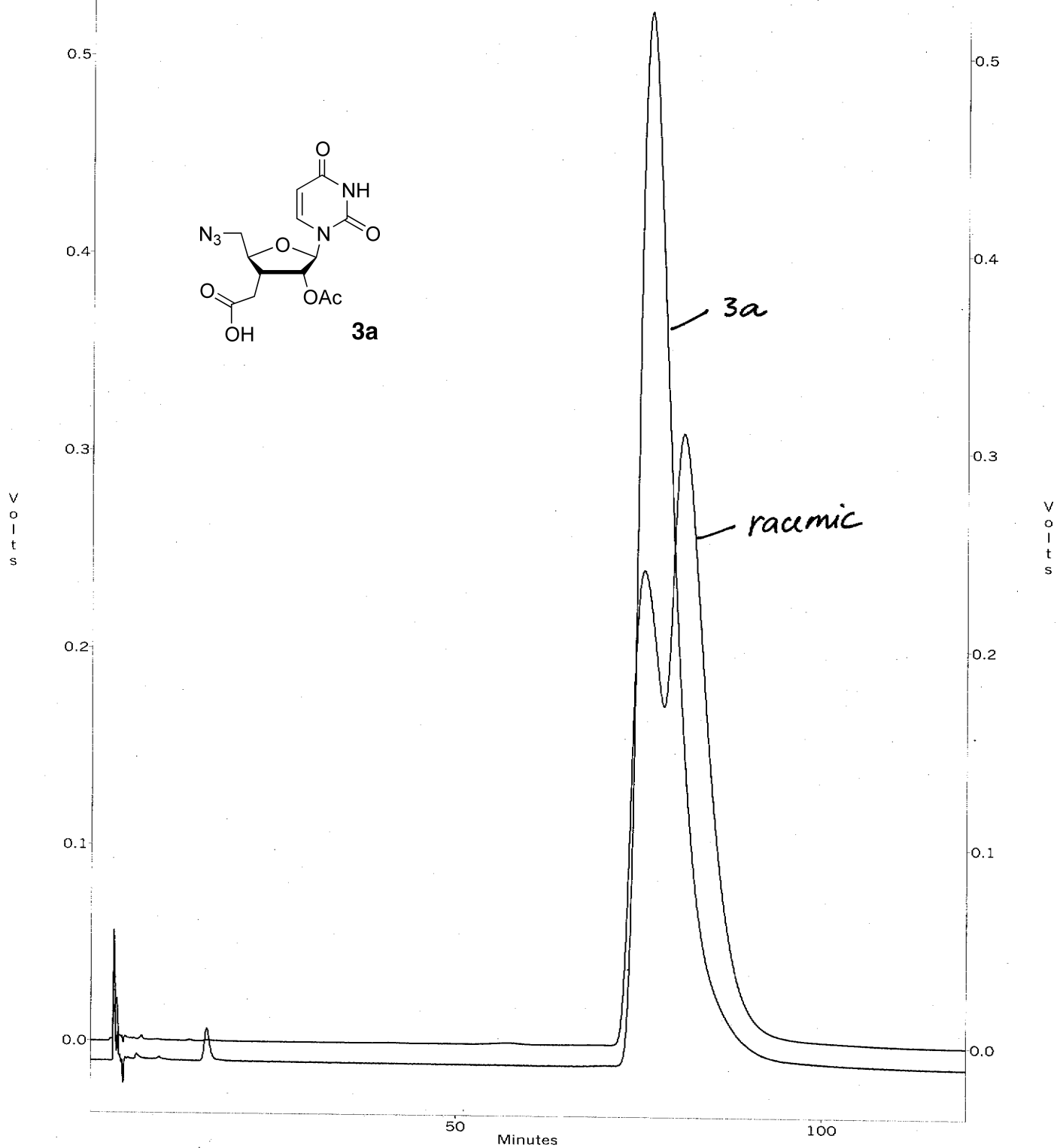
05/06/05/Friday
YL-03-224
(5)

Pulse Sequence: szpu1
Solvent: CDCl3
Ambient temperature
File: YL-03-224-C
Mercury-300 "nmr-300"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 64.3 degrees
Acq. time 1.738 sec
Width 18867.9 Hz
16016 repetitions
OBSERVE C13, 75.4226049 MHz
DECOUPLE H1, 299.9519364 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 84 hr, 3 min, 29 sec



Overlaid Traces

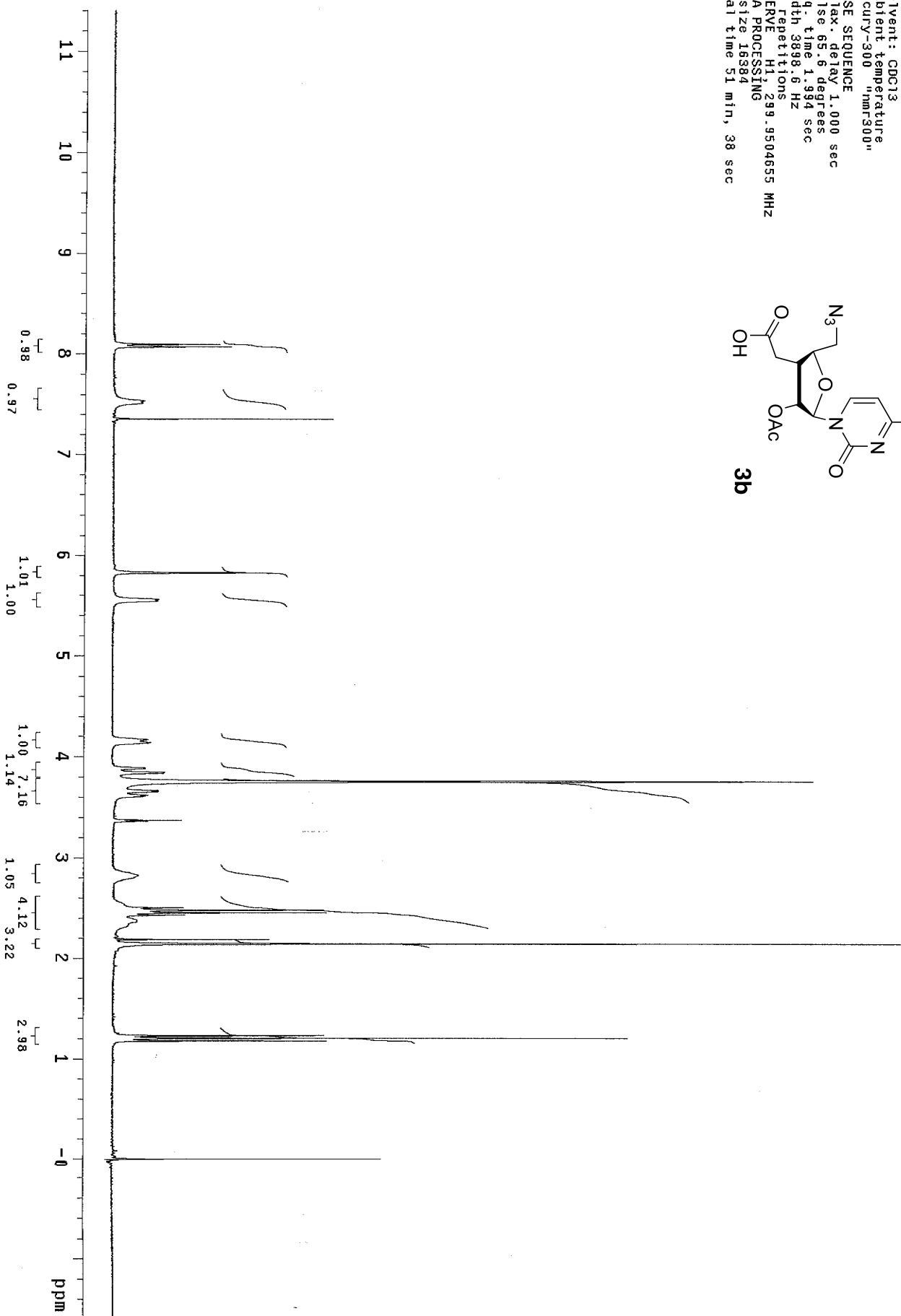
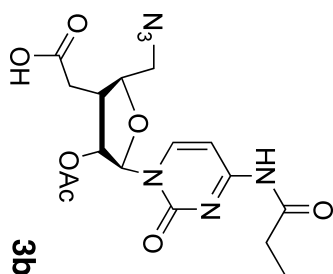
YI_3_224.001, Chan A
YI_3_13.200, Chan A



05/25/05/Friday
YL-03-235
7 again

Pulse Sequence: szpu1
Solvent: CDCl3
Ambient temperature
Mercury-300 "nmr300"

PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 65.6 degrees
Acq. time 1.994 sec
Width 3898.6 Hz
64 repetitions
OBSERVE H1; 299.9504655 MHz
DATA PROCESSING
FT size 16384
Total time 51 min, 38 sec



05/25/05/Friday
YL-03-235
7 again

Pulse Sequence: s2pu1

Solvent: CDC13
Ambient temperature
Mercury-300 "nmr-300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.994 sec

Width 3898.6 Hz

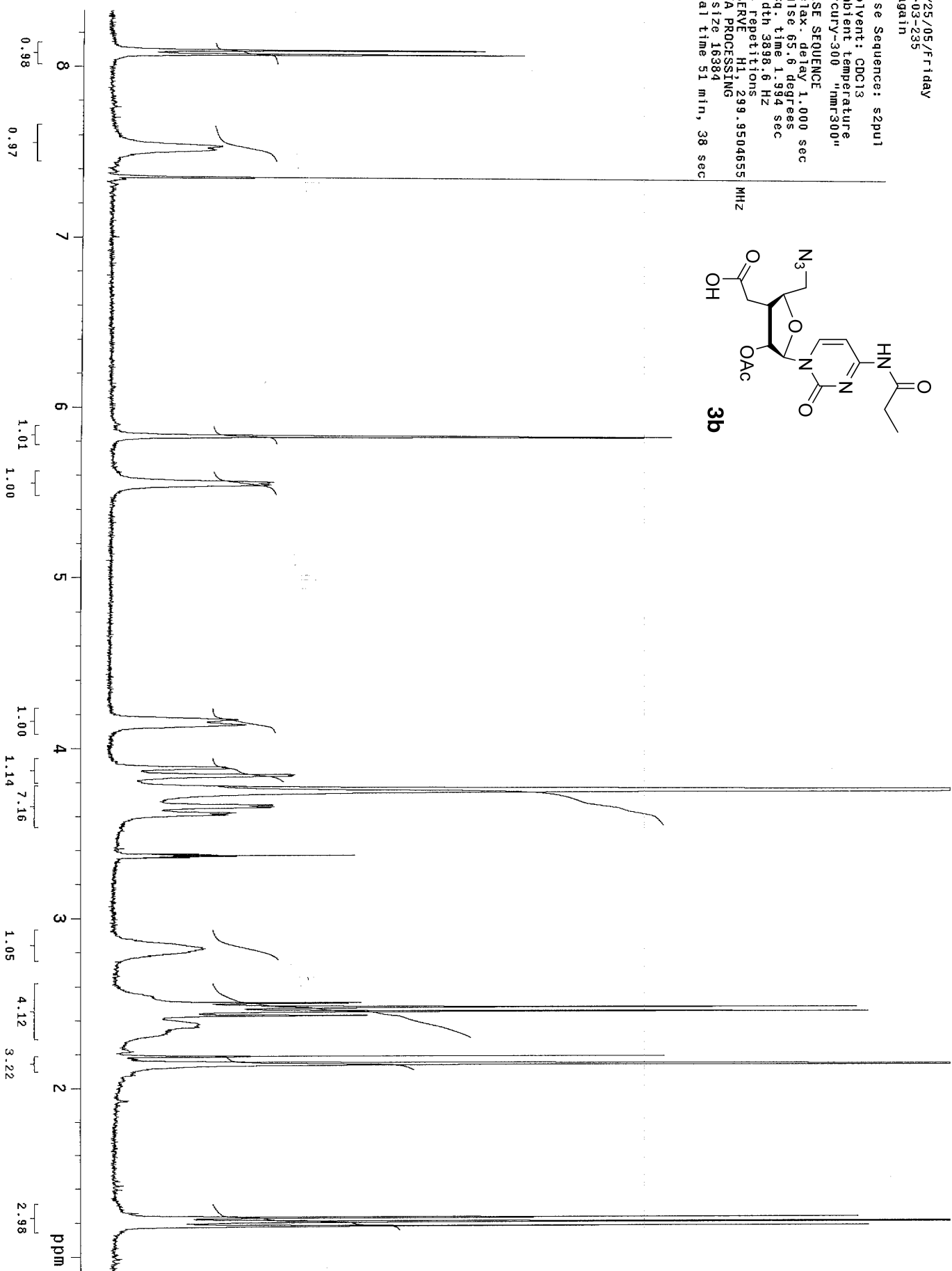
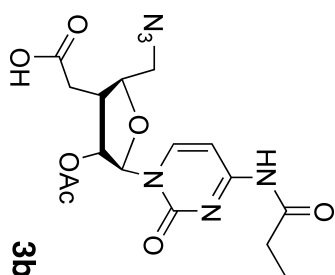
64 repetitions

OBSERVE H1 299.9504655 MHz

DATA PROCESSING

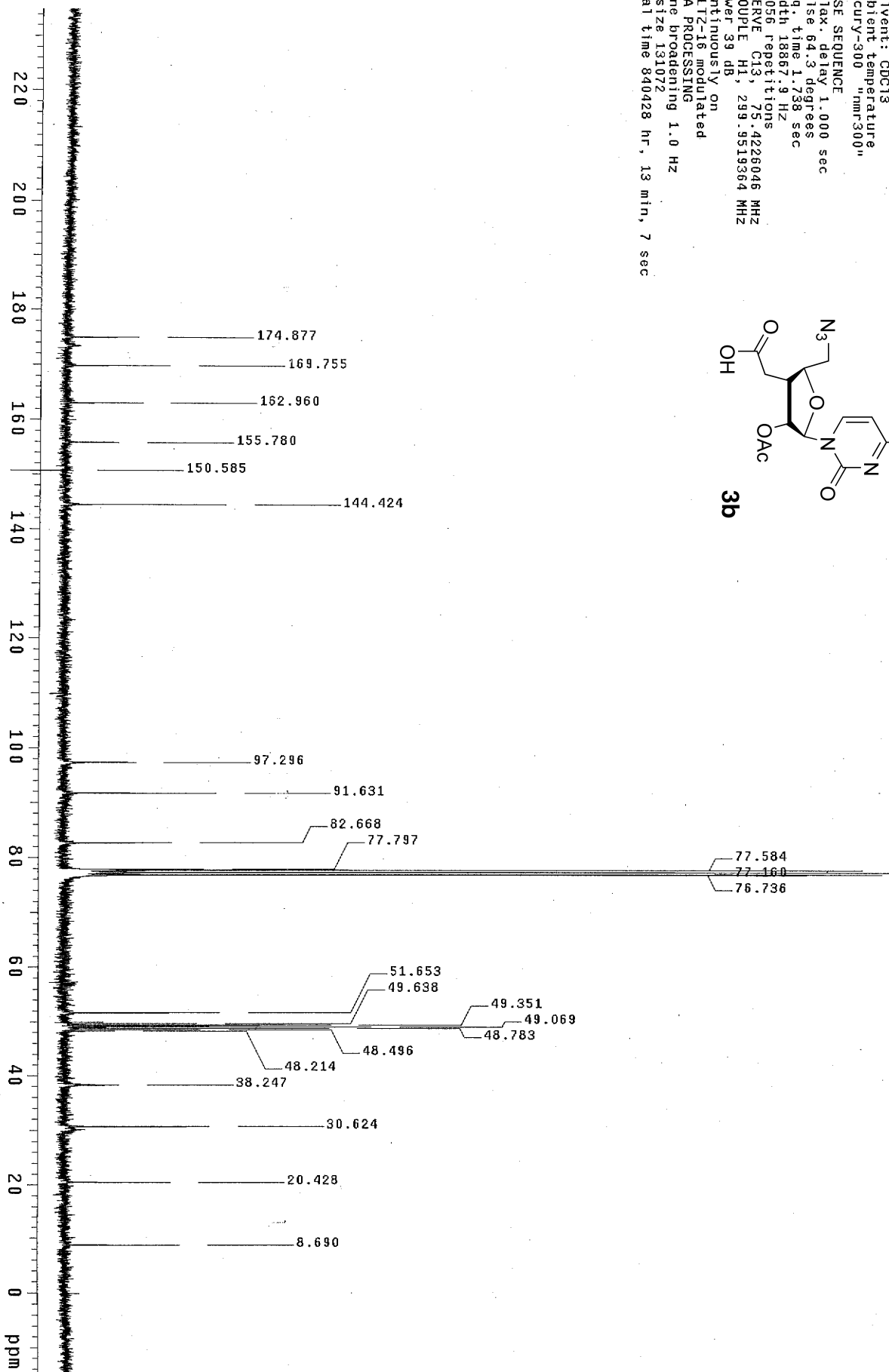
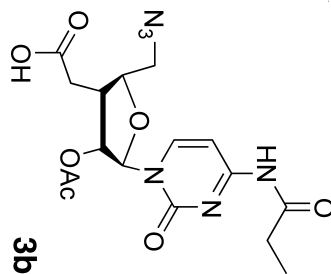
FT size 16384

Total time 51 min, 38 sec

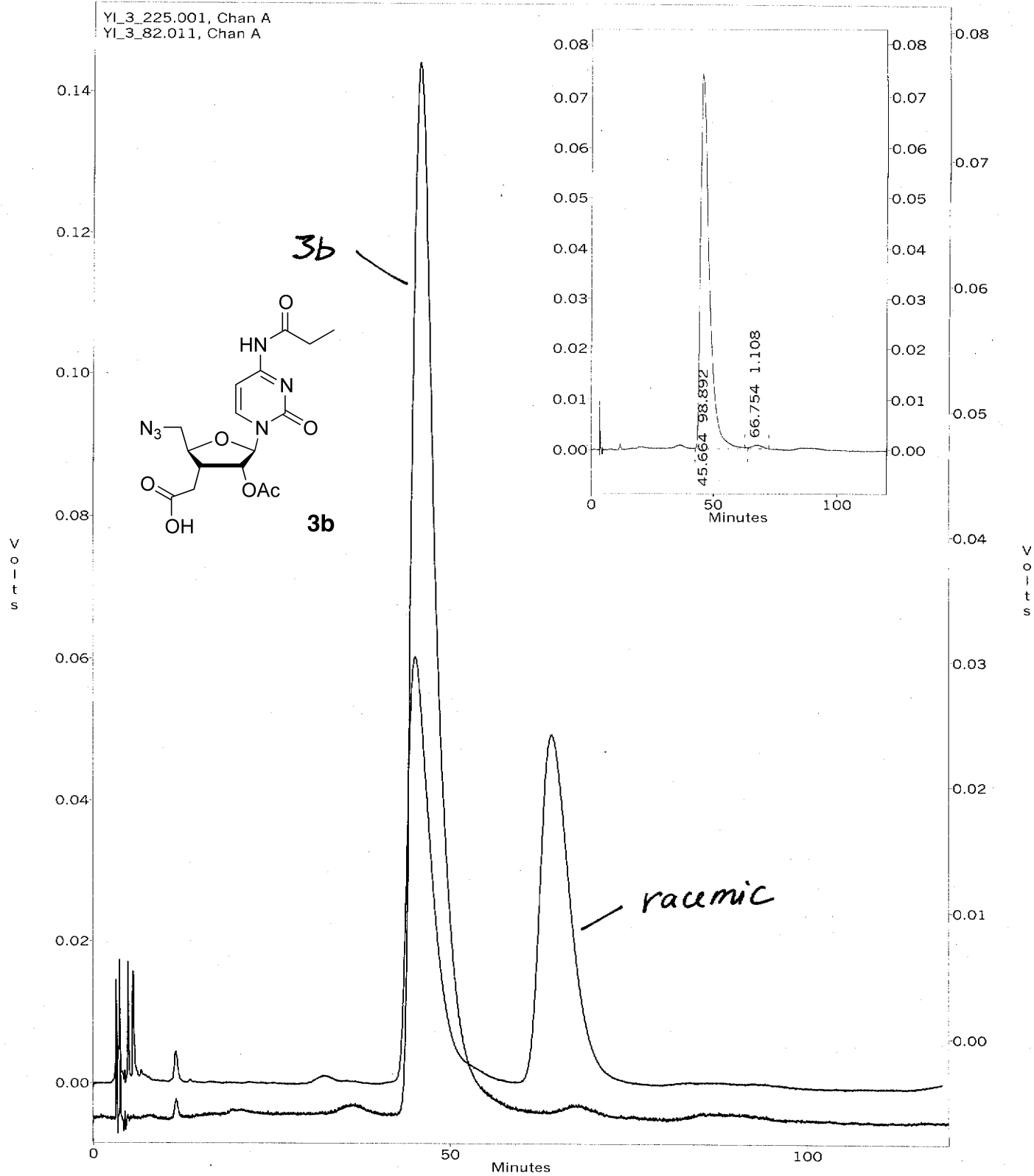


07/01/05/Fri:day
YL-03-225-new-6
C

Pulse Sequence: s2pul1
Solvent: CDCl3
Ambient temperature
Mercury-300 "nmr300"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 64.3 degrees
Acq. time 1.738 sec
Width 18867.9 Hz
81056 repetitions
OBSERVE C13, 75.4226046 MHz
DECOUPLE H1, 299.9519364 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 840428 hr, 13 min, 7 sec



Overlaid Traces



07/13/05/Wed.
YL-03-263
2 again

Pulse Sequence: szpu1

Solvent: CDCl3

Ambient temperature

Mercury-300 "nmr300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.994 sec

Width 3898.6 Hz

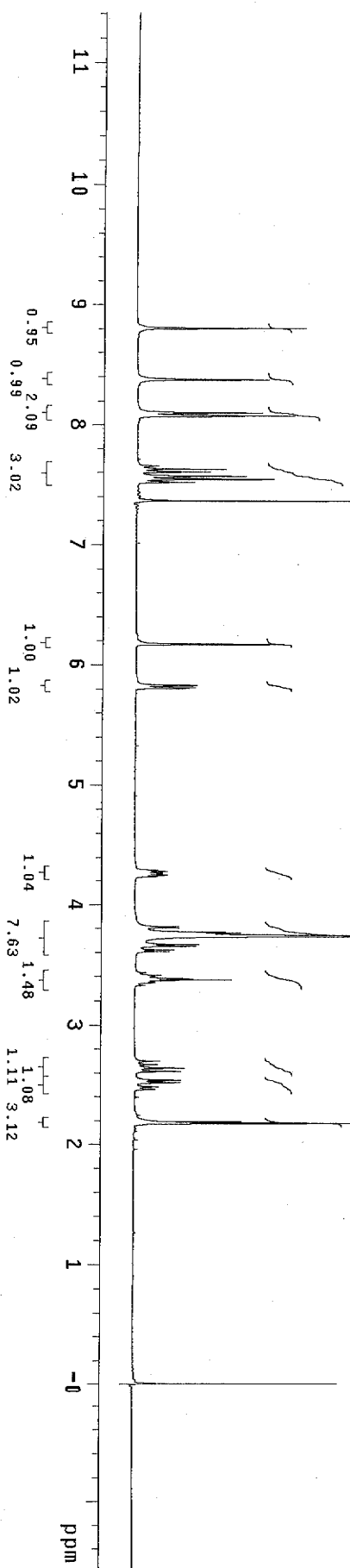
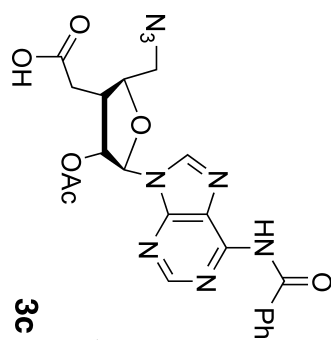
160 Repetitions

OBSERVE H1 299.9504631 MHz

DATA PROCESSING

FT size 16384

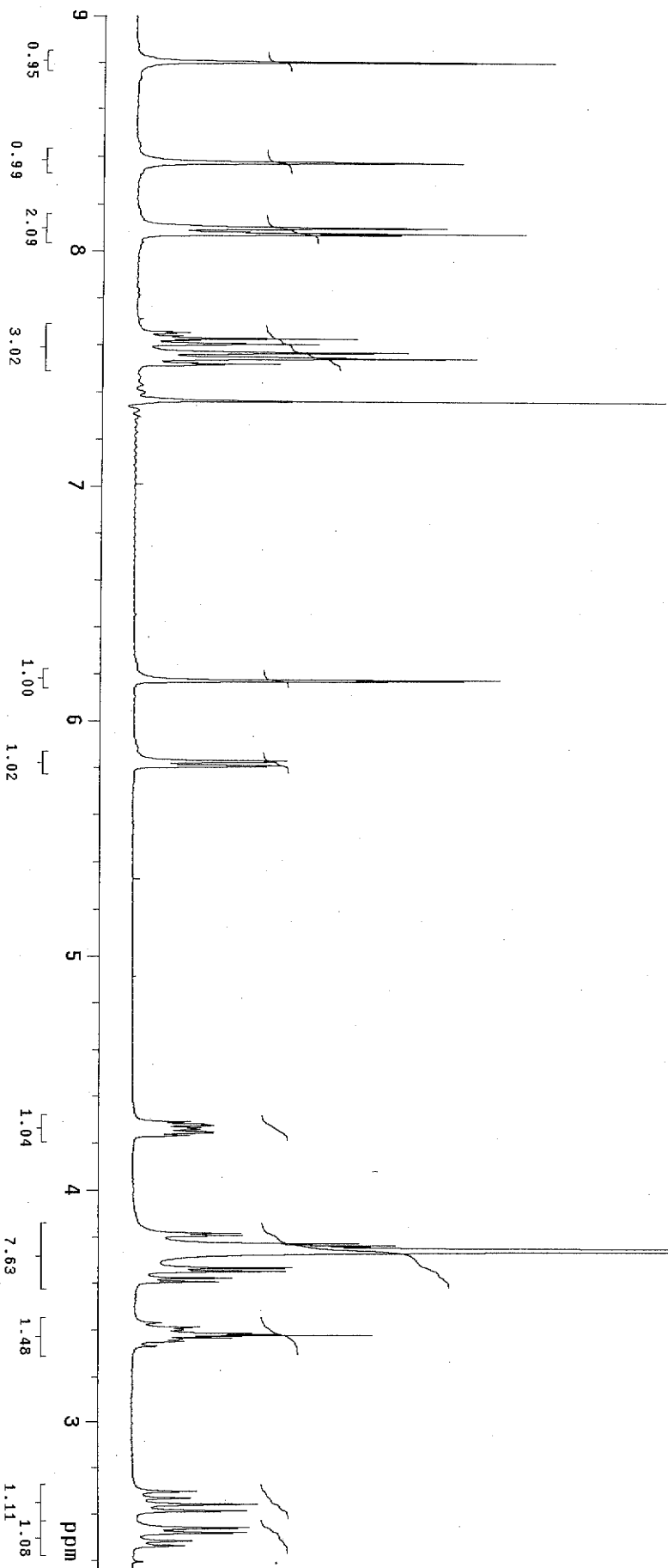
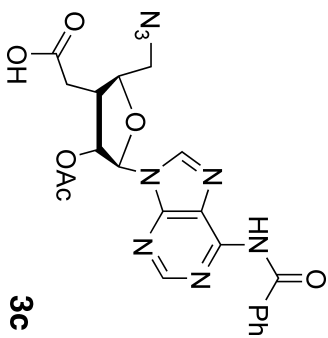
Total time 51 min, 38 sec



07/13/05/Wed.
YL-03-263
2 again

Pulse Sequence: szpu1
Solvent: CDC13
Ambient temperature
Mercury-300 "nmr300"

PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 65.6 degrees
Acq. time 1.994 sec
Width 3888.6 Hz
176 repetitions
OBSERVE H1, 299.9504631 MHz
DATA PROCESSING
FT size 16384
Total time 51 min, 38 sec



07/16/05/Sater.
YL-03-263

Pulse Sequence: szpu1

Solvent: CDCl₃

Ambient temperature

Mercury-300 "nmr-300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 64.3 degrees

Acq. time 1.738 sec

Width 18667.9 Hz

50650 repetitions

OBSERVE C13, 75.4226003 MHz

DECOUPLE H1, 299.9519364 MHz

Power 39 dB

continuously on

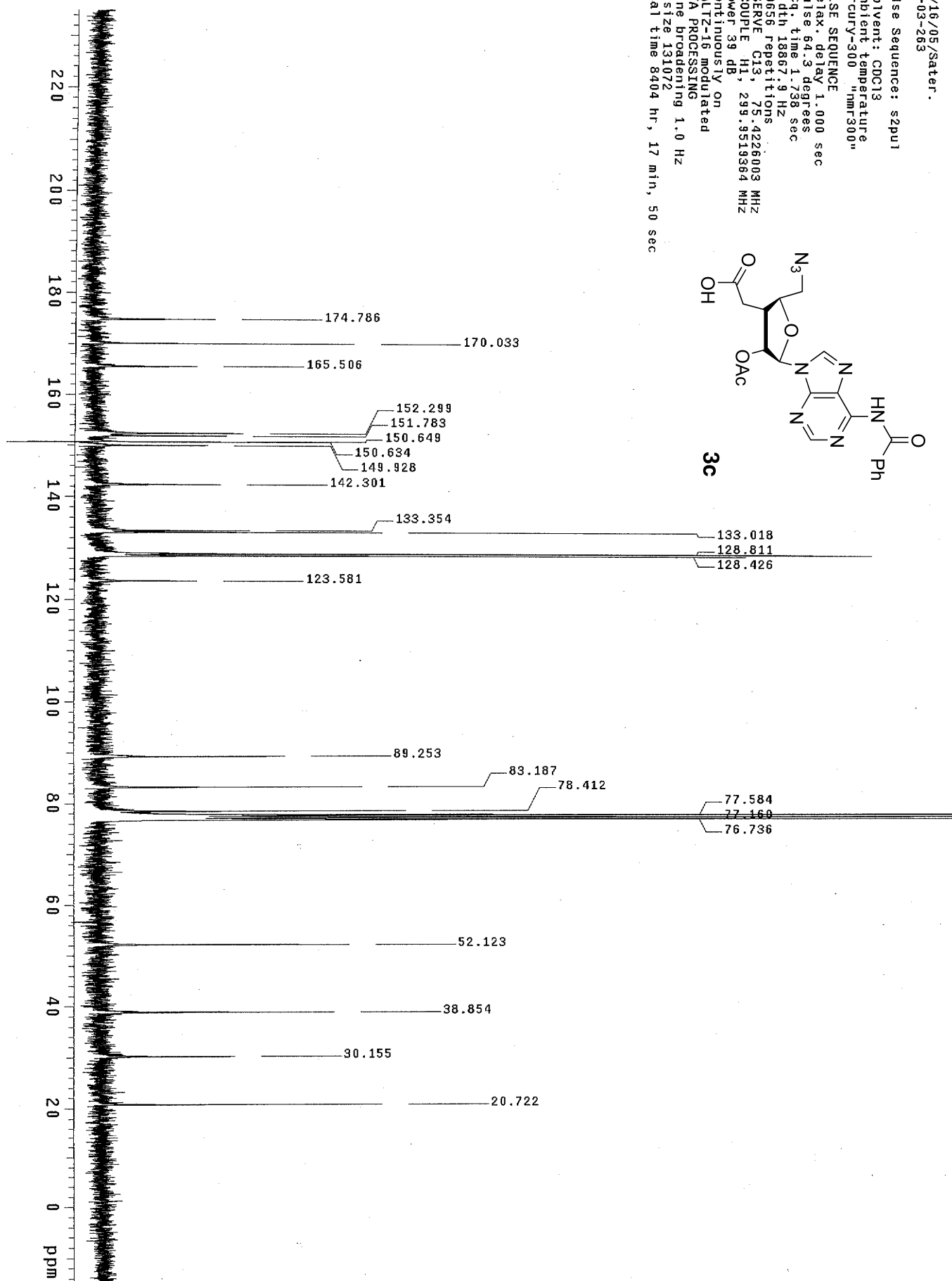
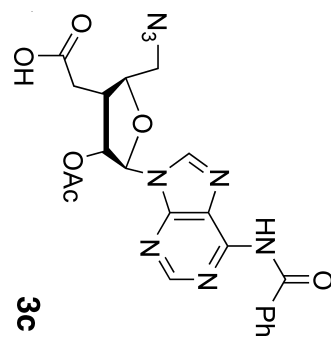
WALTZ-16 modulated

DATA PROCESSING

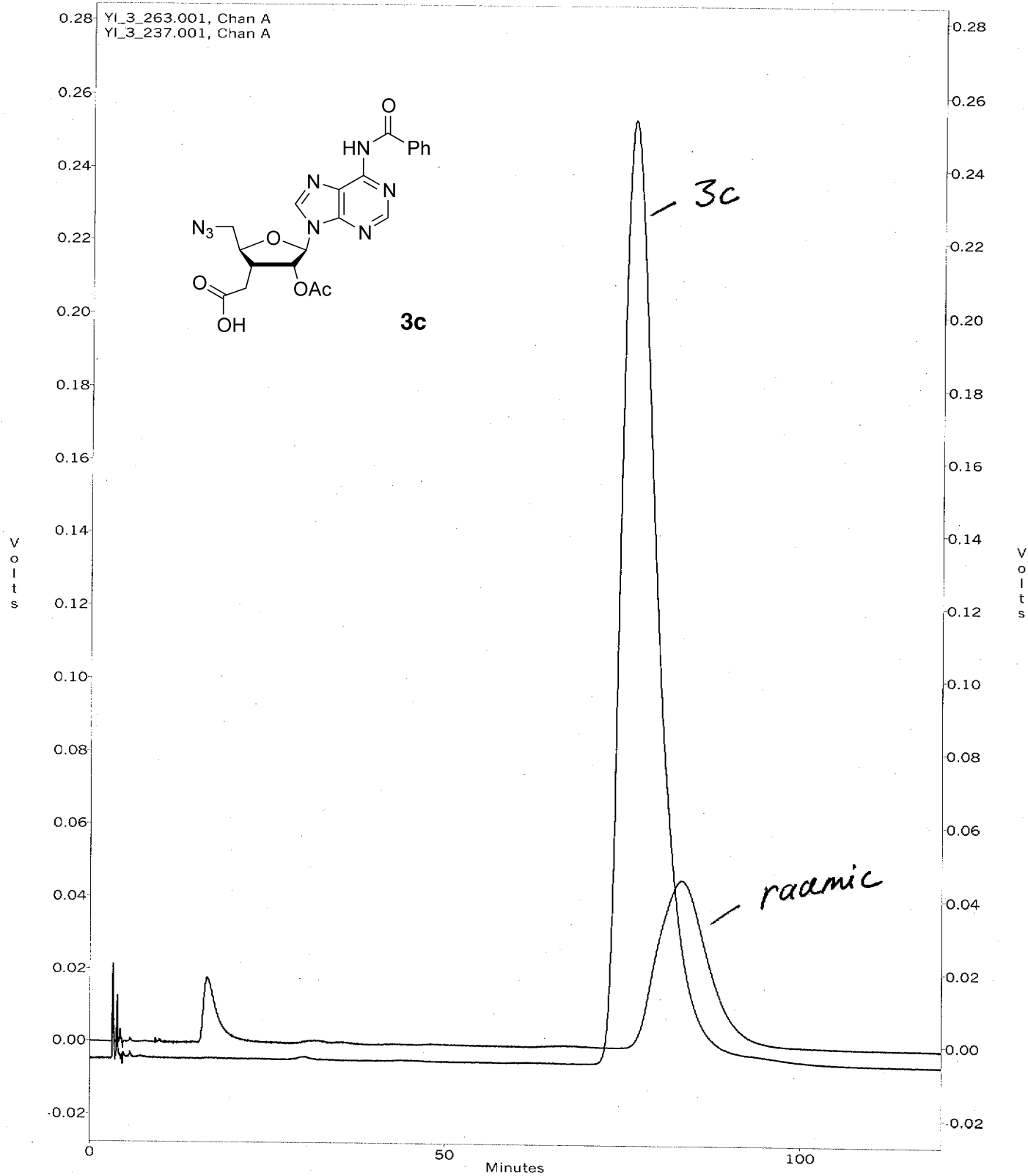
Line broadening 1.0 Hz

FT size 131072

Total time 8404 hr, 17 min, 50 sec



Overlaid Traces



05/23/05/Monday
YL-03-226
6 again

Pulse Sequence: s2pu1

Solvent: CDCl3

Ambient temperature

File: YL-03-226-6-again

Mercury-300 "nmr300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.994 sec

Width 3898.6 Hz

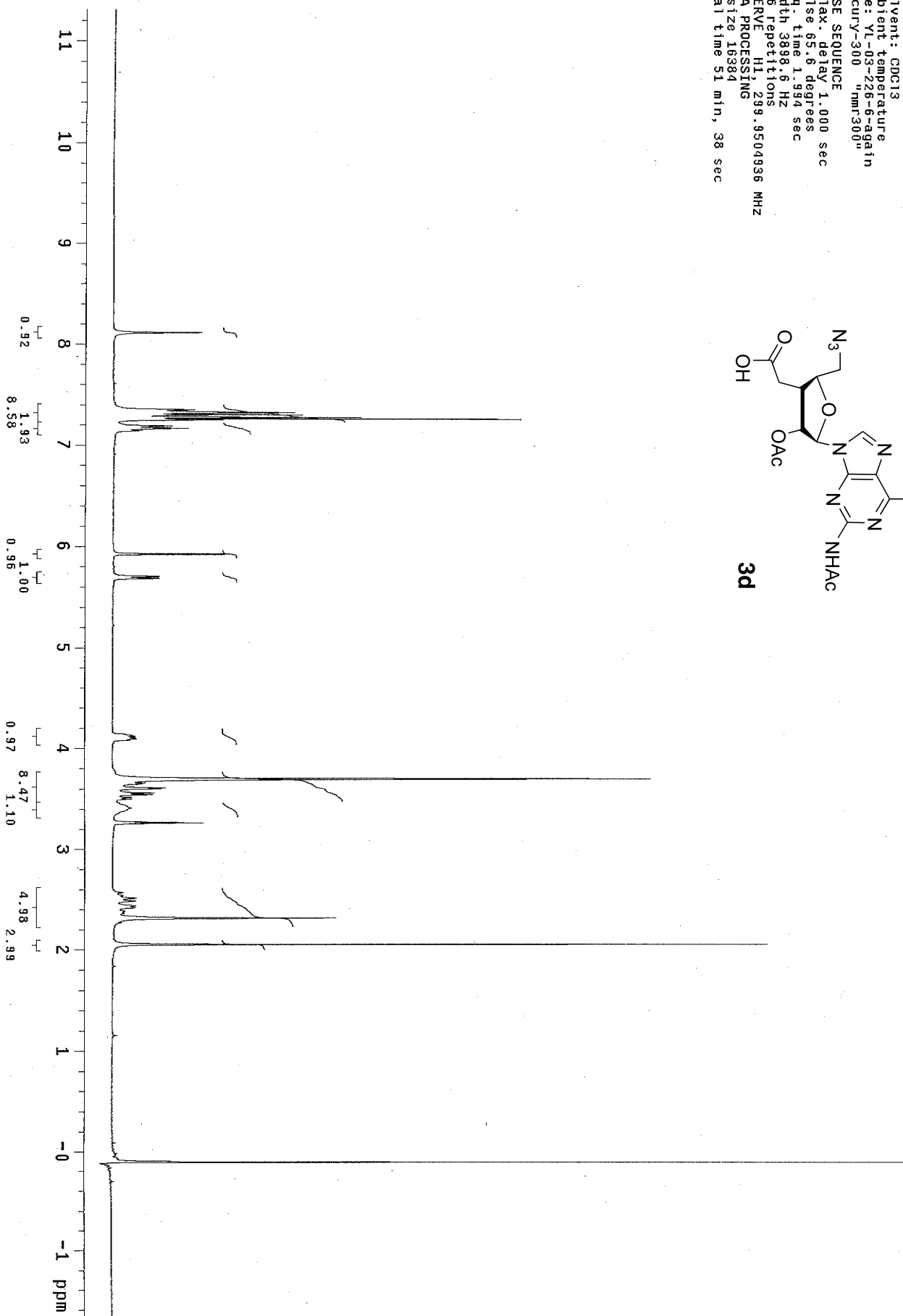
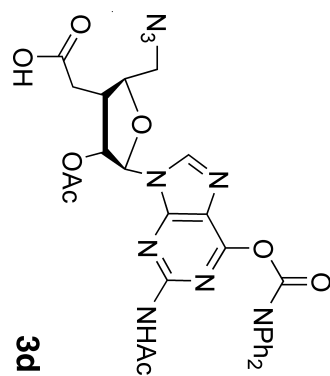
176 repetitions

OBSERVE H1, 299.8504936 MHz

DATA PROCESSING

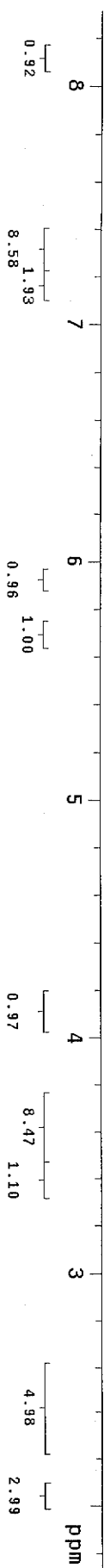
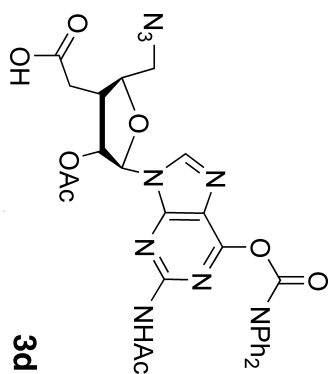
File size 16384

Total time 31 min, 38 sec



05/23/05/Monday
YL-03-226
6 again

Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
File: YL-03-226-6-again
Mercury-300 "nmr-300"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 65.6 degrees
Acq. time 1.994 sec
Width 3898.6 Hz
176 repetitions
OBSERVE H1 299.9504936 MHz
DATA PROCESSING
F1 size 16384
Total time 51 min, 38 sec



05/27/05/Friday
YL-03-226

Pulse Sequence: szpul

Solvent: CDCl₃

Ambient temperature

File: YL-03-226-C-C

Mercury-300

"nmr300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 64.3 degrees

Acq. time 1.738 sec

Width 18867.9 Hz

38848 repetitions

OBSERVE C13, 75.4226015 MHz

DECOUPLE H1, 299.9519364 MHz

Power 39 dB

continuously on

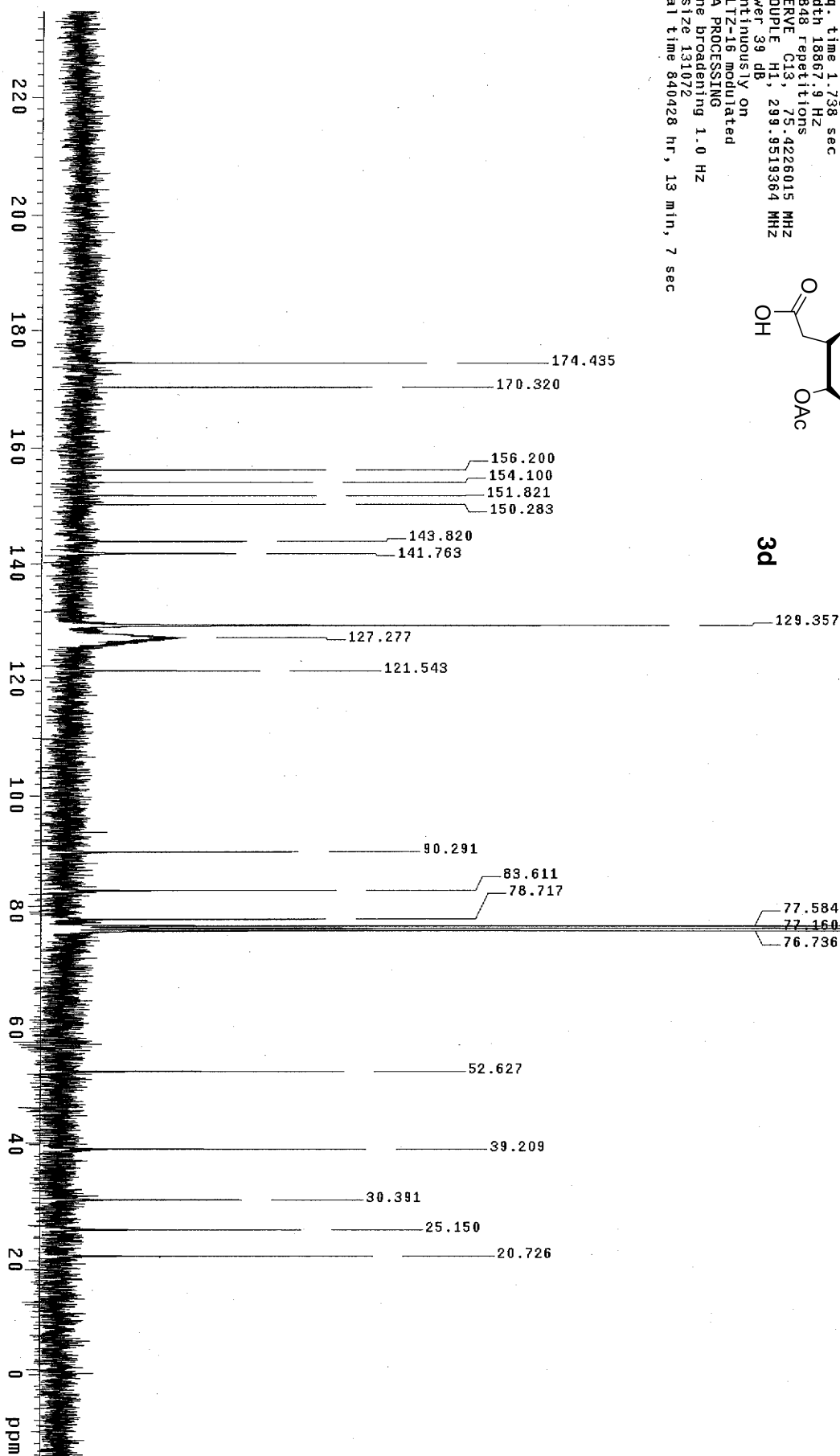
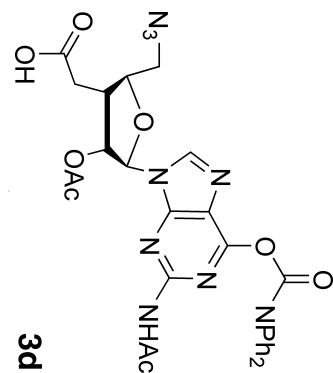
WALTZ-16 modulated

DATA PROCESSING

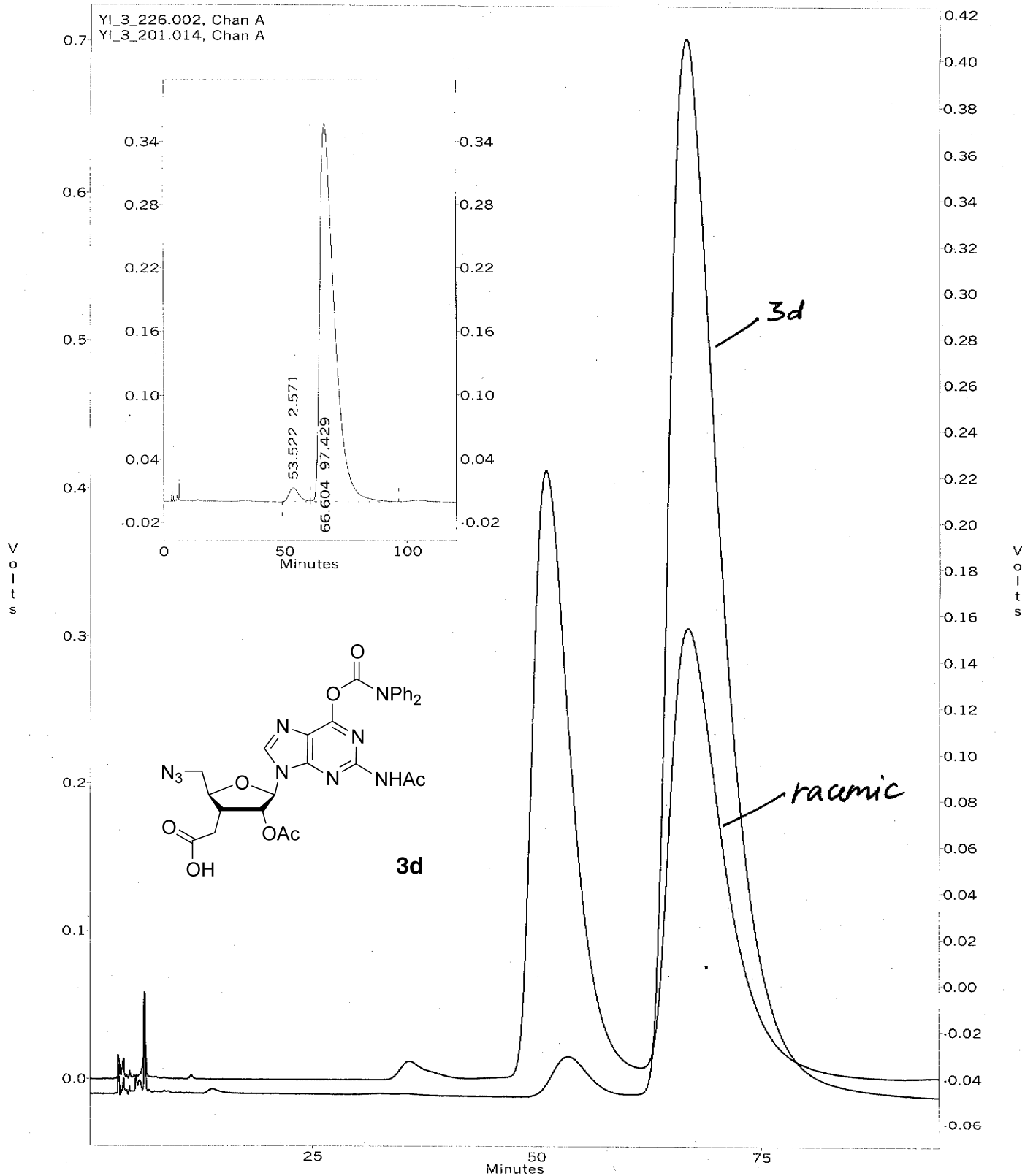
Line broadening 1.0 Hz

FT size 131072

Total time 840428 hr, 13 min, 7 sec



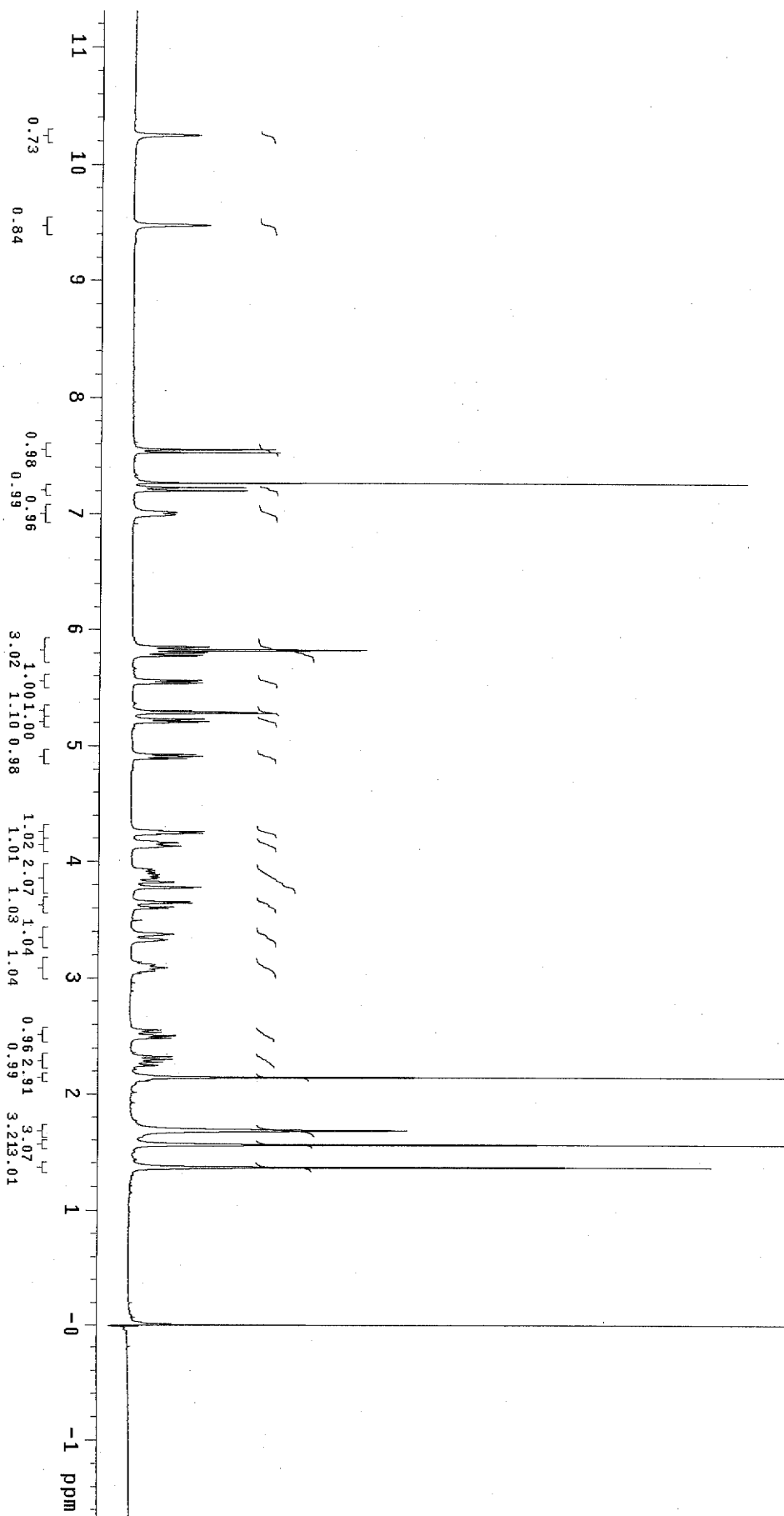
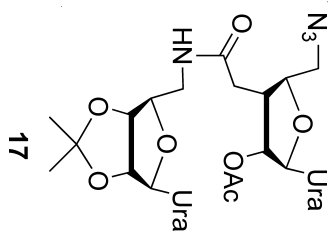
Overlaid Traces



07/18/05/Monday
YL-03-265
3

Pulse Sequence: szpu1
Solvent: CDCl3
Ambient temperature
File: YL-03-265
Mercury-300 "nmr300"

PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 65.6 degrees
Acq. time 1.934 sec
Width 3898.6 Hz
192 repetitions
OBSERVE H1, 299.9504931 MHz
DATA PROCESSING
FI size 16384
Total time 51 min, 38 sec



07/18/05/Monday
YL-03-265
3

Pulse Sequence: s2put

Solvent: CDCl₃
Ambient temperature

File: YL-03-265
Mercury-300 "nmr300"

PULSE SEQUENCE
Relax delay

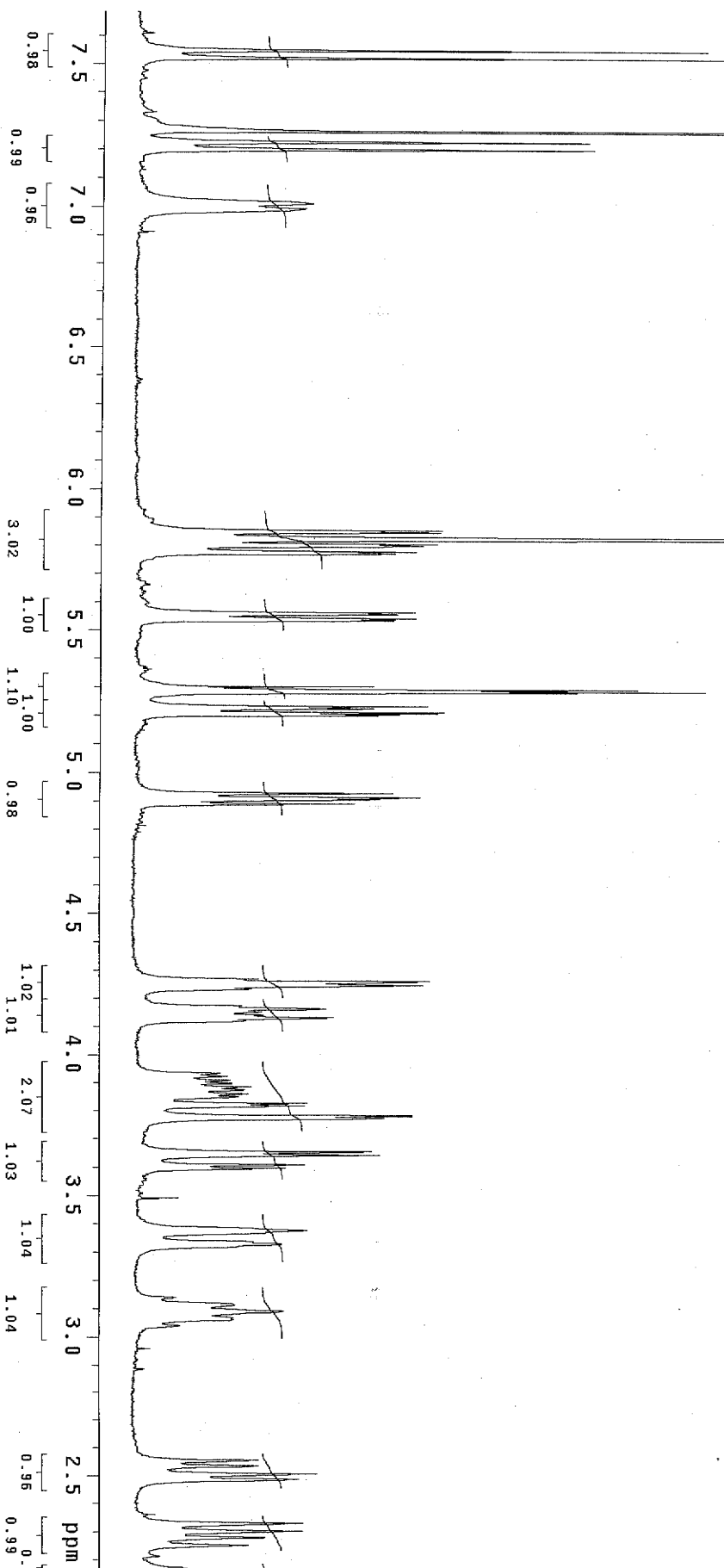
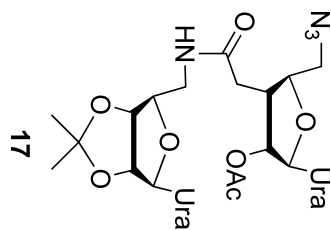
Pulse 65.6 degrees

Acq. time 1.994	sec
Width 3898.6	HZ

192 repetitions
OBSERVE H1, 29

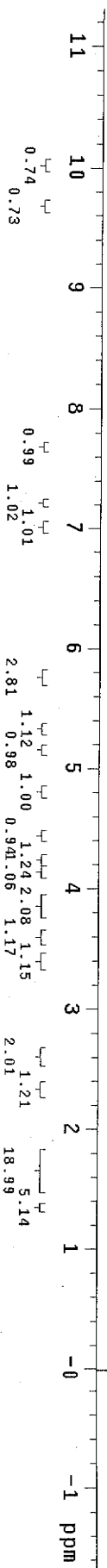
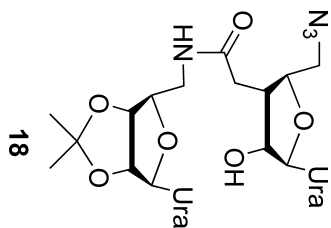
DATA PROCESSING
ET size 16384

Total time 51 min, 38 sec



07/20/05/Wed.
YL-03-266

Pulse Sequence: szpul
Solvent: CDCl3
Ambient temperature
Mercury-300 "hmr300"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 65.6 degrees
Acq. time 1.994 sec
Width 3838.6 Hz
256 repetitions
OBSERVE H1, 299.950436 MHz
DATA PROCESSING
Ft size 16384
Total time 51 min, 38 sec



07/20/05/Wed.
YL-03-266

Pulse Sequence: szpu1
Solvent: CDCl3
Ambient temperature
Mercury-300 "nmr300"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 65.6 degrees
Acq. time 1.984 sec
Width 3898.6 Hz
256 repetitions
OBSERVE H1, 299.9504836 MHz
DATA PROCESSING
FT size 16384
Total time 51 min, 38 sec

