Electronic Supplementary Information

Highly efficient synthesis of medium-sized lactones via oxidative lactonization: Concise total synthesis of isolaurepan

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General Methods. All reactions sensitive to moisture and/or air were carried out under an atmosphere of argon in dry, freshly distilled solvents under anhydrous conditions using oven-dried glasswares unless otherwise noted. Anhydrous dichloromethane (CH_2Cl_2) was purchased from Kanto Chemical Co. Inc. and used directly without further drying. Anhydrous tetrahydrofuran was purchased from Wako Pure Chemical Industries, Ltd. and further purified by a Glass Contour solvent purification system under an atmosphere of argon immediately prior to use. Hexamethylphosphoramide (HMPA) was distilled from calcium hydride under reduced pressure. All other chemicals were purchased at highest commercial grade and used directly. Analytical thin-layer chromatography was performed using E. Merck silica gel 60 F_{254} plates (0.25-mm thickness). Flash column chromatography was carried out using Kanto Chemical silica gel 60N (40-100 mesh, spherical, neutral) or Fuji Silysia silica gel BW-300 (200-400 mesh). Optical rotations were recorded on a JASCO P-1020 digital polarimeter. IR spectra were recorded on a JASCO FT/IR-4100 spectrometer. ^{1}H and ^{13}C NMR spectra were recorded on a Varian Unity INOVA-500 or INOVA-600 spectrometer, and chemical shift values are reported in ppm (δ) downfield from tetramethylsilane with reference to internal residual solvent [^{1}H NMR, $CHCl_3$ (7.24), C_6D_5H (7.15); ^{13}C NMR, $CDCl_3$ (77.0), C_6D_6 (128.0)]. Coupling constants (*J*) are reported in Hertz (Hz). The following abbreviations were used to designate the multiplicities: s = singlet; d = doublet; m = multiplet; br = broad. EI and FAB mass spectra were recorded on a JEOL JMS-700 spectrometer, and ESI-TOF mass spectra were measured on a Bruker microTOFfocus spectrometer.

Representative experimental procedures for the TEMPO/PhI(OAc)$_2$-mediated oxidative lactonization: To a solution of diol **1** (1.02 g, 4.05 mmol) in CH_2Cl_2 (40 mL) were added $PhI(OAc)_2$ (2.88 g, 8.94 mmol) and TEMPO (60.8 mg, 0.389 mmol). After being stirred at room temperature for 12 h, the reaction mixture was quenched with 1:1 saturated aqueous $NaHCO_3$/saturated aqueous Na_2SO_3 and extracted with EtOAc. The organic layer was washed with brine, dried (Na_2SO_4), filtered, and concentrated under reduced pressure. Purification of the residue by flash chromatography (silica gel, 30% EtOAc/hexanes) gave lactone **2** (0.930 g, 93%) as a colorless solid.

Spectroscopic data for all newly synthesized compounds 4, 6, 8, 10, 12, 14a,b, 18, 20, 27, and isolaurepan (23).

Compound 4: colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.71 (ddd, *J* = 7.8, 7.8, 1.2

Hz, 4H), 7.43—7.35 (m, 6H), 4.36 (ddd, *J* = 4.8, 3.5, 3.5 Hz, 1H), 4.28 (ddd, *J* = 10.2, 5.4, 1.8 Hz, 1H), 4.12 (dd, *J* = 10.8, 4.8 Hz, 1H), 4.00 (dd, *J* = 11.4, 1.8 Hz, 1H), 3.92 (dd, *J* = 11.4, 5.4 Hz, 1H), 3.00 (ddd, *J* = 13.2, 13.2, 1.5 Hz, 1H), 2.44 (ddd, *J* = 14.4, 6.0, 3.0 Hz, 1H), 2.32 (m, 1H), 2.08 (m, 1H), 1.33 (s, 3H), 1.32 (s, 3H), 1.04 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 174.4, 135.74 (2C), 135.68 (2C), 133.3, 133.1, 129.7, 129.6, 127.7 (2C), 127.6 (2C), 107.9, 76.7, 73.8, 73.7, 63.3, 28.3, 27.9, 26.7 (3C), 25.9, 23.9, 19.3; $[\alpha]_D^{26}$ +41.3 (*c* 1.00, $CHCl_3$); IR (film) 3071, 2932, 2857, 1744, 1112, 1064, 704 cm^{-1}; HRMS (ESI) calcd for $C_{26}H_{34}O_5SiNa^+$ [$(M+Na)^+$] 477.2068, found 477.2076.

Compound **6** was isolated as a 55:45 mixture of diastereomers, which were separable by flash chromatography on silica gel.

Compound 6 (less polar): colorless solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36—7.26 (m, 5H), 4.83 (ddd, *J* = 10.0, 5.0, 5.0 Hz, 1H), 4.58 (d, *J* = 14.0 Hz, 1H), 4.56 (d, *J* = 14.0 Hz, 1H), 4.32 (m, 1H), 4.25 (ddd, *J* = 11.5, 4.0, 4.0 Hz, 1H), 3.63 (dd, *J* = 10.0, 6.0 Hz, 1H), 3.51 (dd, *J* = 10.0, 5.0 Hz, 1H), 3.05 (dd, *J* = 12.0, 12.0 Hz, 1H), 2.62 (dd, *J* = 12.5, 4.0 Hz, 1H), 2.36 (dd, 16.5, 3.0 Hz, 1H), 2.00 (ddd, *J* = 16.5, 10.0, 3.5 Hz, 1H), 1.47 (s, 3H), 1.34 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 169.8, 137.6, 128.5 (2C), 127.90, 127.87 (2C), 107.9, 73.7, 73.4, 72.7, 72.0, 71.7, 37.4, 31.1, 28.6, 26.0; $[\alpha]_D^{26}$ –12.5 (*c* 0.32, $CHCl_3$); IR (film) 2923, 1736, 1280, 1221, 1038 cm^{-1}; HRMS (ESI) calcd for $C_{17}H_{22}O_5Na^+$ [$(M+Na)^+$] 329.1359, found 329.1367. **Compound 6 (more polar)**: colorless solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36—7.26 (m, 5H), 4.56 (d, *J* = 14.0 Hz, 1H), 4.54 (d, *J* = 14.0 Hz, 1H), 4.39—4.31 (m, 2H), 4.24 (ddd, *J* = 9.5, 6.0, 6.0 Hz, 1H), 3.63 (dd, *J* = 10.0, 5.5 Hz, 1H), 3.48 (dd, *J* = 10.0, 5.5 Hz, 1H), 3.04, (dd, *J* = 14.5, 6.0 Hz, 1H), 2.97 (dd, *J* = 15.0, 3.0 Hz, 1H), 2.20 (ddd, *J* = 15.0, 6.0, 1.5 Hz, 1H), 1.95 (ddd, *J* = 15.0, 10.0, 10.0 Hz, 1H), 1.46 (s, 3H), 1.32 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 169.8, 137.5, 128.5 (2C), 127.9, 127.8 (2C), 108.9, 74.1, 73.8, 73.7, 71.7, 71.6, 34.6, 33.8, 28.2, 25.8; $[\alpha]_D^{26}$ –26.9 (*c* 1.00, $CHCl_3$); IR (film) 2985, 2933, 1735, 1199, 1050 cm^{-1}; HRMS (ESI) calcd for $C_{17}H_{22}O_5Na^+$ [$(M+Na)^+$] 329.1359, found 329.1366.

Compound 8: colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36—7.26 (m, 5H), 5.69 (ddd, *J* = 11.0, 8.0, 3.0 Hz, 1H), 5.53 (ddd, *J* = 11.0, 8.5, 2.0 Hz, 1H), 4.85 (dddd, *J* = 11.0, 5.5, 5.5, 3.0 Hz, 1H), 4.59 (d, *J* = 11.5 Hz, 1H), 4.55 (d, *J* = 11.5 Hz, 1H), 3.67 (dd, *J* = 10.0, 5.5 Hz, 1H), 3.63 (ddd, *J* = 3.0, 3.0, 3.0 Hz, 1H), 3.53 (dd, *J* = 10.0, 5.0 Hz, 1H), 3.03 (dd, *J* = 16.5, 9.0 Hz, 1H), 2.52—2.38 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 172.1, 137.6, 128.54, 128.45 (2C), 127.9, 127.8 (2C), 118.7, 75.6, 73.6, 71.7, 33.8, 32.4; $[\alpha]_D^{25}$ +18.1 (*c* 1.00, $CHCl_3$); IR (film) 3030, 2867, 1736, 1271, 1120 cm^{-1};

HRMS (ESI) calcd for $C_{14}H_{16}O_3Na^+$ [$(M+Na)^+$] 255.0992, found 255.0998.

Compound 10: colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36—7.25 (m, 5H), 4.57 (d, *J* = 12.5 Hz, 1H), 4.53 (d, *J* = 12.5 Hz, 1H), 4.37 (m, 1H), 3.63 (dd, *J* = 10.0, 5.5 Hz, 1H), 3.46 (dd, *J* = 10.0, 5.5 Hz, 1H), 2.66 (dd, *J* = 14.0, 7.0 Hz, 1H), 2.56 (m, 1H), 2.05 (m, 1H), 1.99—1.88 (m, 2H), 1.62—1.88 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 172.1, 137.6, 128.54, 128.45 (2C), 127.9, 127.8 (2C), 118.7, 75.6, 73.6, 71.7, 33.8, 32.4; $[\alpha]_D^{25}$ +6.0 (*c* 1.00, $CHCl_3$); IR (film) 2933, 2861, 1732, 1455, 1104 cm^{-1}; HRMS (ESI) calcd for $C_{14}H_{18}O_3Na^+$ [$(M+Na)^+$] 257.1148, found 257.1141.

Compound 12: colorless oil; ^{1}H NMR (500 MHz, C_6D_6) δ 7.36—7.25 (m, 5H), 4.44 (d, *J* = 7.0 Hz, 1H), 4.37 (d, *J* = 7.0 Hz, 1H), 4.29 (d, *J* = 12.0 Hz, 1H), 4.26 (d, *J* = 12.0 Hz, 1H), 4.20 (ddd, *J* = 11.0, 6.0, 6.0 Hz, 1H), 3.38 (dd, *J* = 10.0, 5.5 Hz, 1H), 3.35 (m, 1H), 3.30 (m, 1H), 3.18 (dd, *J* = 9.5, 6.5 Hz, 1H), 3.14 (s, 3H), 1.90 (m, 1H), 1.61—1.51 (m, 2H), 1.29 (m, 1H), 0.80 (d, *J* = 8.0 Hz, 3H):^{13}C NMR (125 MHz, C_6D_6) δ 172.4, 138.6, 128.6 (2C), 128.0 (2C), 127.8, 95.2, 77.0, 74.8, 73.5, 72.9, 55.4, 48.0, 27.6, 26.2, 12.7; $[\alpha]_D^{25}$ +23.7 (*c* 1.00, $CHCl_3$); IR (film) 2934, 1723, 1258, 1097, 1031 cm^{-1}; HRMS (ESI) calcd for $C_{17}H_{24}O_5Na^+$ [$(M+Na)^+$] 331.1516, found 331.1526.

Compound 14a: colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 4.35 (d, *J* = 2.4 Hz, 1H), 4.19—4.13 (m, 2H), 3.92 (m, 1H), 3.02 (ddd, *J* = 14.4, 14.4, 2.4 Hz, 1H), 2.44 (ddd, *J* = 14.4, 6.6, 2.4 Hz, 1H), 2.30 (m, 1H), 2.02 (m, 1H), 1.43 (s, 3H), 1.33 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 174.4, 108.4, 73.6, 73.5, 64.9, 28.1, 27.5, 25.8, 23.1; $[\alpha]_D^{27}$ –63.9 (*c* 1.00, $CHCl_3$); IR (film) 2986, 2937, 1746, 1160, 1053 cm^{-1}; HRMS (EI) calcd for $C_9H_{14}O_4^+$ [M^+] 186.0892, found 186.0891.

Compound 14b: colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 4.98 (d, *J* = 7.8 Hz, 1H), 4.41 (ddd, *J* = 12.0, 7.8, 3.6 Hz, 1H), 4.31 (dd, *J* = 13.2, 7.8 Hz, 1H), 4.09 (ddd, *J* = 12.6, 12.6, 4.8 Hz, 1H), 2.20 (m, 1H), 2.10 (m, 1H), 1.69 (m, 1H), 1.59 (m, 1H), 1.54 (s, 3H), 1.36 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.2, 110.3, 76.8, 73.7, 64.6, 26.54, 26.50, 24.4, 22.2; $[\alpha]_D^{27}$ –9.6 (*c* 1.00, $CHCl_3$); IR (film) 2988, 2941, 1752, 1382, 1160, 1092 cm^{-1}; HRMS (EI) calcd for $C_9H_{14}O_4^+$ [M^+] 186.0892, found 186.0890.

Compound 18: colorless solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.48—7.43 (m, 2H), 7.39—7.32 (m, 3H), 5.51 (s, 1H), 4.03 (d, *J* = 11.5 Hz, 1H), 3.82 (dd, *J* = 12.0, 4.0 Hz, 1H), 3.79 (d, *J* = 11.0 Hz, 1H), 2.83 (dd, *J* = 15.0, 6.0 Hz, 1H), 2.59 (ddd, *J* = 15.0, 15.0,

2.5 Hz, 1H), 2.08 (m, 2H), 1.93 (m, 2H), 1.67 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 173.2, 137.0, 129.1, 128.3 (2C), 126.0 (2C), 101.7, 81.6, 75.7, 74.6, 36.7, 30.6, 20.0, 17.3; $[\alpha]_D^{25}$ +37.8 (*c* 1.00, $CHCl_3$); IR (film) 2940, 2868, 1717, 1203, 1096, 1015 cm^{-1}; HRMS (ESI) calcd for $C_{15}H_{18}O_4Na^+$ $[(M+Na)^+]$ 285.1097, found 285.1099.

Compound 20: colorless solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46 (m, 2H), 7.38—7.32 (m, 3H), 5.88—5.75 (m, 2H), 5.49 (s, 1H), 4.64 (ddd, *J* = 10.0, 10.0, 5.5 Hz, 1H), 4.38 (dd, *J* = 10.0, 5.5 Hz, 1H), 3.85 (m, 1H), 3.83 (dd, *J* = 10.0, 10.0 Hz, 1H), 3.44 (dd, *J* = 15.5, 5.5 Hz, 1H), 3.20 (dd, *J* = 15.5, 5.5 Hz, 1H), 2.66—2.54 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.9, 136.9, 129.2, 128.3 (2C), 126.54, 126.47, 126.1 (2C), 101.4, 79.4, 71.5, 68.4, 35.7, 30.2; $[\alpha]_D^{19}$ +128.3 (*c* 0.50, $CHCl_3$); IR (film) 2983, 2955, 2924, 2872, 1733, 1455, 1389, 1279, 1135, 1099 cm^{-1}; HRMS (FAB) calcd for $C_{15}H_{17}O_4$ $[(M+H)^+]$ 261.1126, found 261.1129.

Compound 22: colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.75 (dd, *J* = 1.6, 1.6 Hz, 1H), 7.48—7.42 (m, 2H), 7.37—7.30 (m, 3H), 5.46 (s, 1H), 4.26 (dd, *J* = 9.0, 3.5 Hz, 1H), 3.63—3.50 (m, 3H), 2.45 (ddd, *J* = 7.0, 7.0, 1.5 Hz, 2H), 1.96—1.56 (m, 6H), 1.46 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 202.8, 137.8, 128.9, 128.2 (2C), 126.0 (2C), 100.9, 81.6, 71.2, 65.8, 43.8, 31.4, 24.6, 21.9; $[\alpha]_D^{27}$ –43.7 (*c* 1.00, $CHCl_3$); IR (film) 3444, 1636, 1456, 1397, 1071, 1027 cm^{-1}; HRMS (ESI) calcd for $C_{15}H_{20}O_4Na^+$ $[(M+Na)^+]$ 287.1254, found 287.1243.

Compound 27: colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.19 (m, 1H), 2.64 (dd, *J* = 13.5, 6.0 Hz, 1H), 2.58 (dd, *J* = 13.5, 13.5 Hz, 1H), 1.94—1.84 (m, 3H), 1.72—1.39 (m, 6H), 1.35—1.20 (m, 7H), 0.85 (dd, *J* = 6.5, 6.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 175.8, 80.6, 36.4, 34.9, 34.5, 31.7, 29.0, 28.3, 25.3, 23.0, 22.5, 14.0; IR (film) 2929, 2859, 1729, 1591, 1457, 1281, 1254, 1175, 1013 cm^{-1}; HRMS (EI) calcd for $C_{12}H_{22}O_2^+$ $[M^+]$ 198.1620, found 198.1620.

Isolaurepan (23): colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.40—3.32 (m, 2H), 1.73—1.59 (m, 4H), 1.56—1.40 (m, 8H), 1.37—1.20 (m, 10H), 0.89 (dd, *J* = 7.0, 7.0 Hz, 3H), 0.86 (dd, *J* = 7.0, 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 80.3, 80.0, 39.6, 37.4, 36.92, 36.87, 31.9, 29.3, 26.3, 25.33, 25.29, 22.6, 19.5, 14.11, 14.10; IR (film) 2956, 2928, 2857, 1457, 1377, 1141, 1101, 1004 cm^{-1}; HRMS (EI) calcd for $C_{15}H_{30}O^+$ $[M^+]$ 226.2301, found 226.2297.

HF26108/CDCl3

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Sample directory: vnmr1_08Jul2002-151843
File: PROTON

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 20.0 C / 293.1 K
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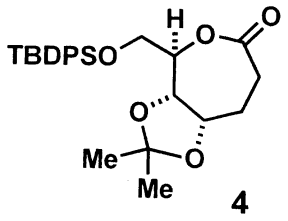

4

C13 STD parameter

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User: 1-14-87
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64 repetitions
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DECOUPLE H1, 599.9152462 MHz
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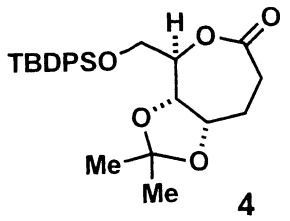

4

STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 20.0 C / 293.1 K
INOVA-500 "varian"

Relax. delay 1.000 sec
Pulse 45.0 degrees
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OBSERVE H1, 499.8593228 MHz
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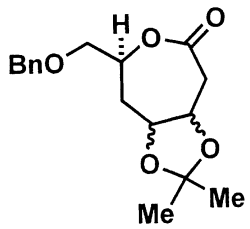
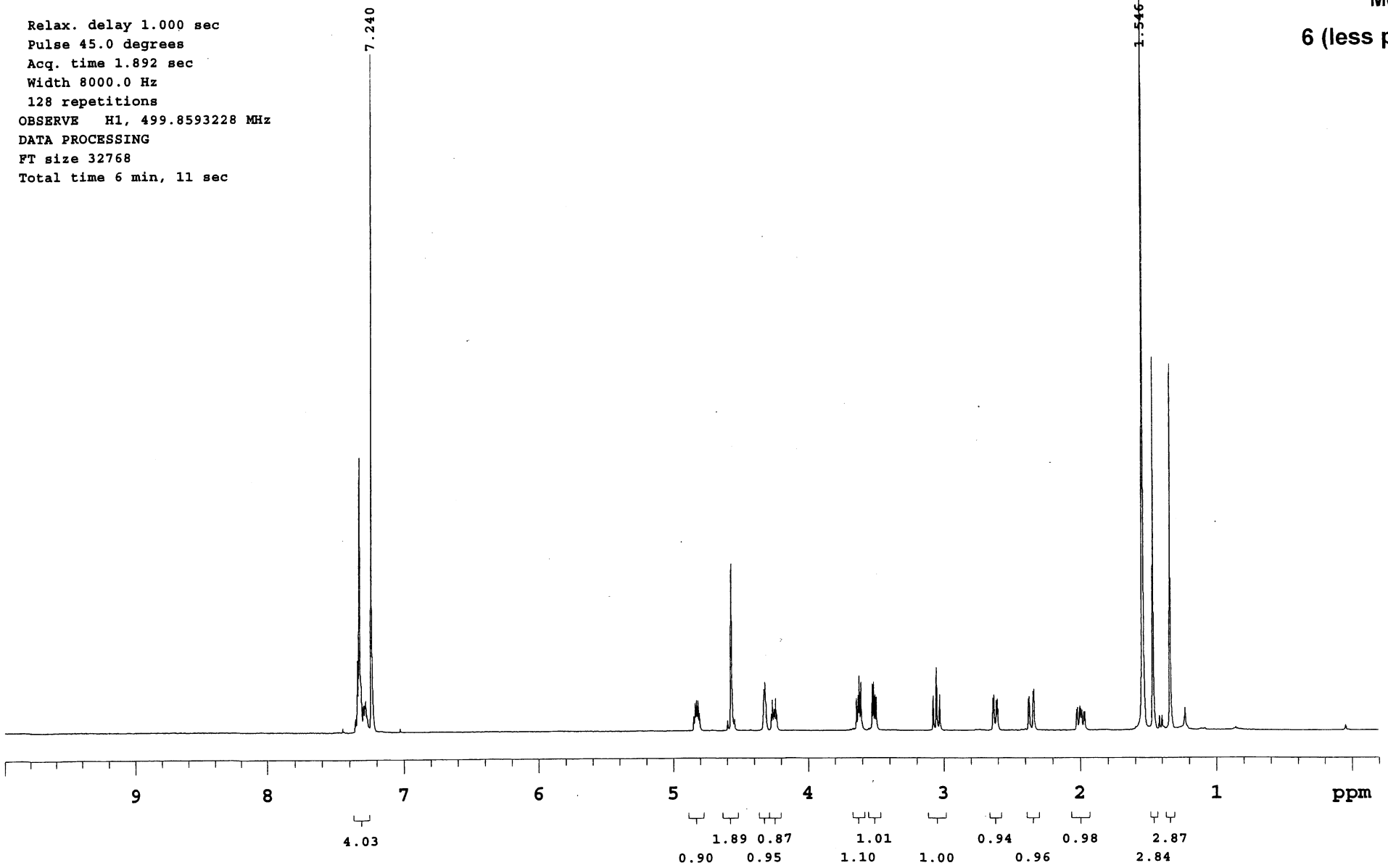


6 (less polar)



STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul

Solvent: CDCl3
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User: 1-14-87
INOVA-500 "varian"

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432 repetitions
OBSERVE C13, 125.6897254 MHz
DECOUPLE H1, 499.8618041 MHz
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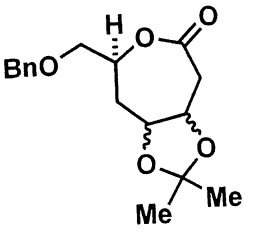

6 (less polar)

STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 20.0 C / 293.1 K
INOVA-500 "varian"

Relax. delay 1.000 sec
Pulse 45.0 degrees
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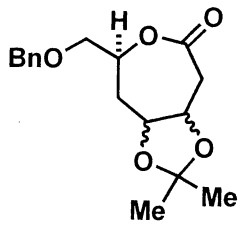
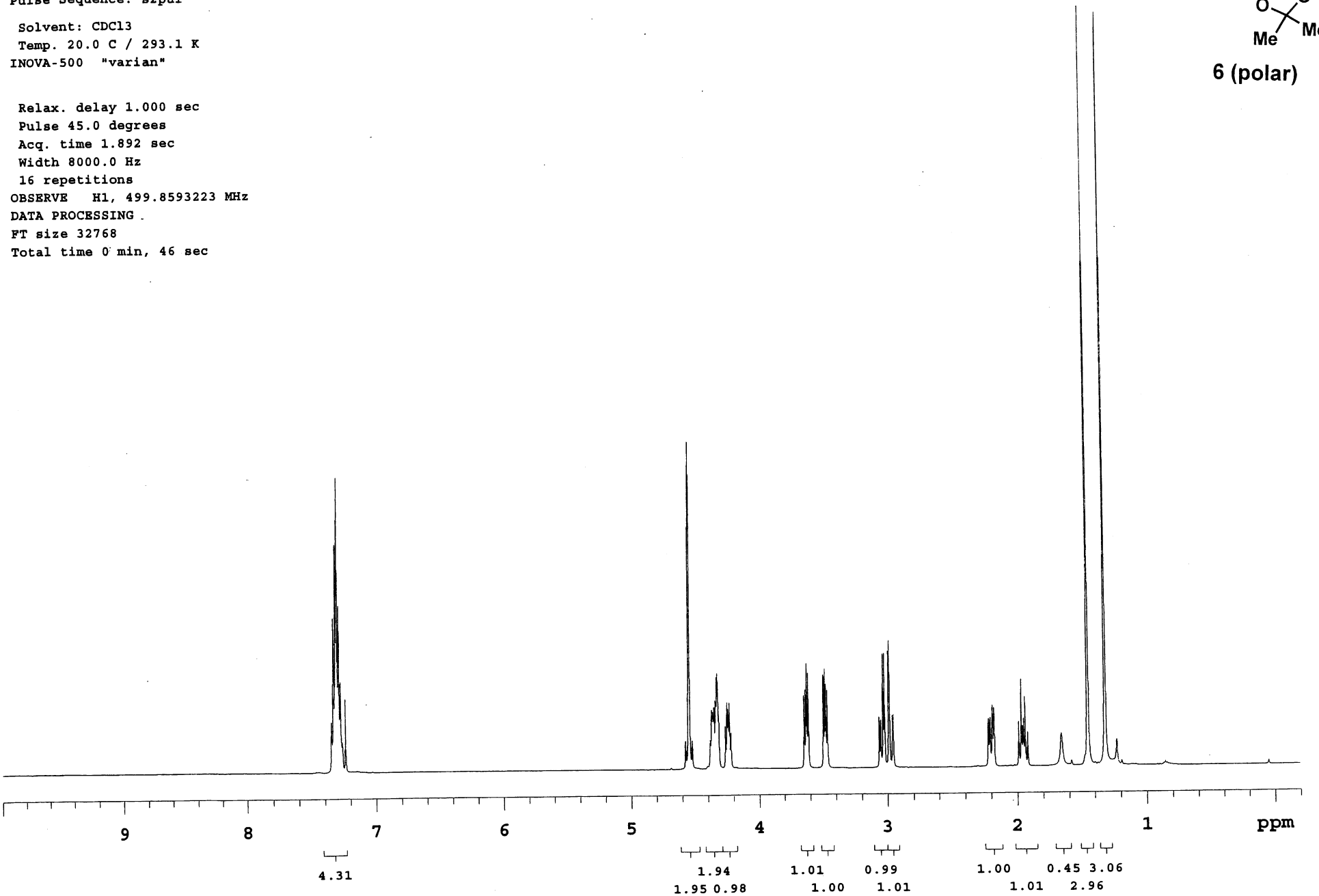


6 (polar)



STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 20.0 C / 293.1 K
User: 1-14-87
INOVA-500 "varian"

Relax. delay 0.700 sec
Pulse 45.0 degrees
Acq. time 1.298 sec
Width 37735.8 Hz
108 repetitions
OBSERVE C13, 125.6897300 MHz
DECOUPLE H1, 499.8618041 MHz
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continuously on
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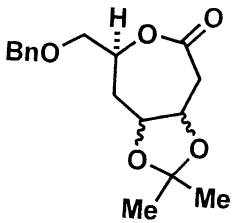
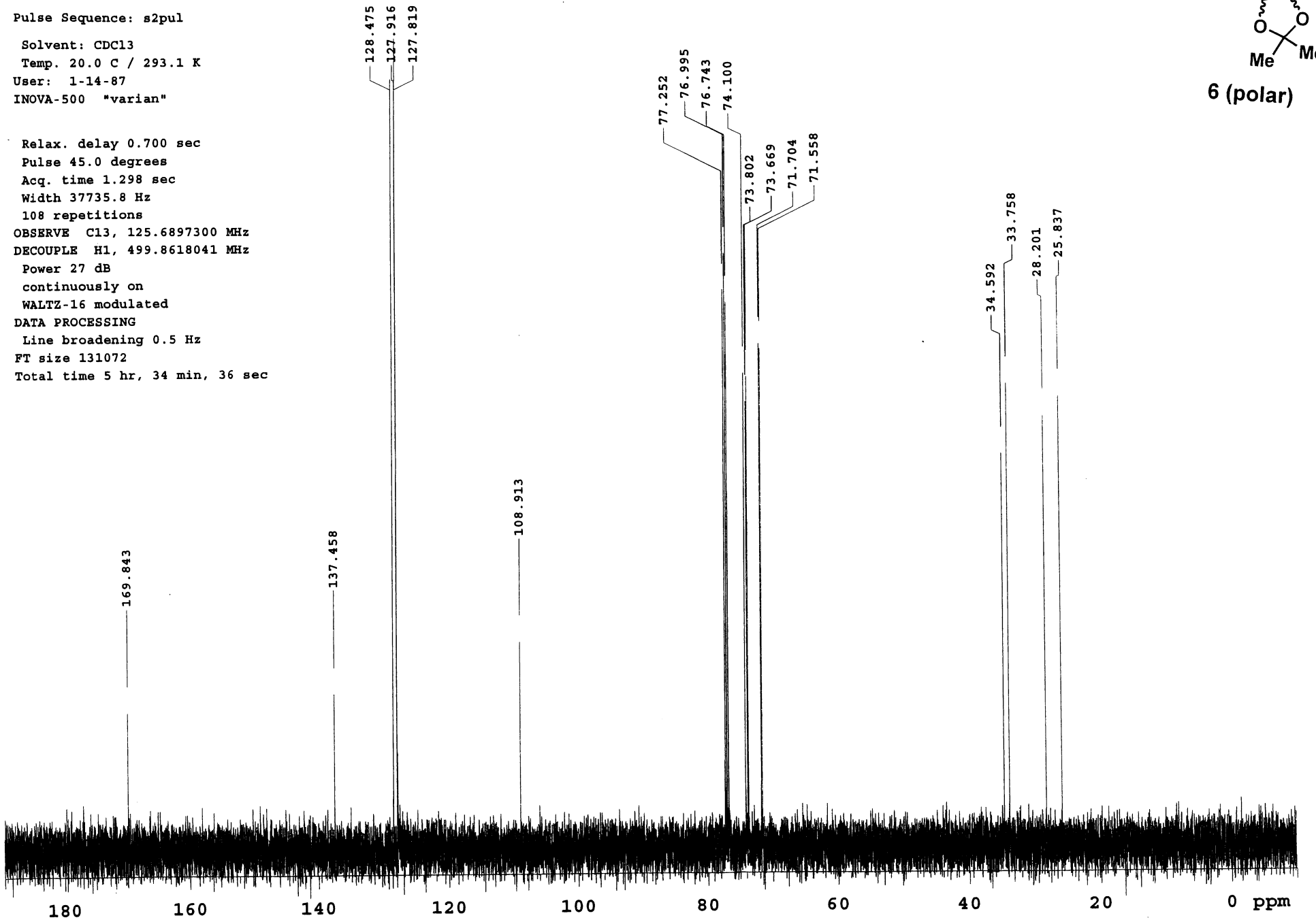


6 (polar)



STANDARD PROTON PARAMETERS

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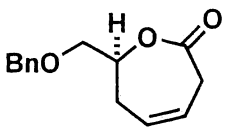
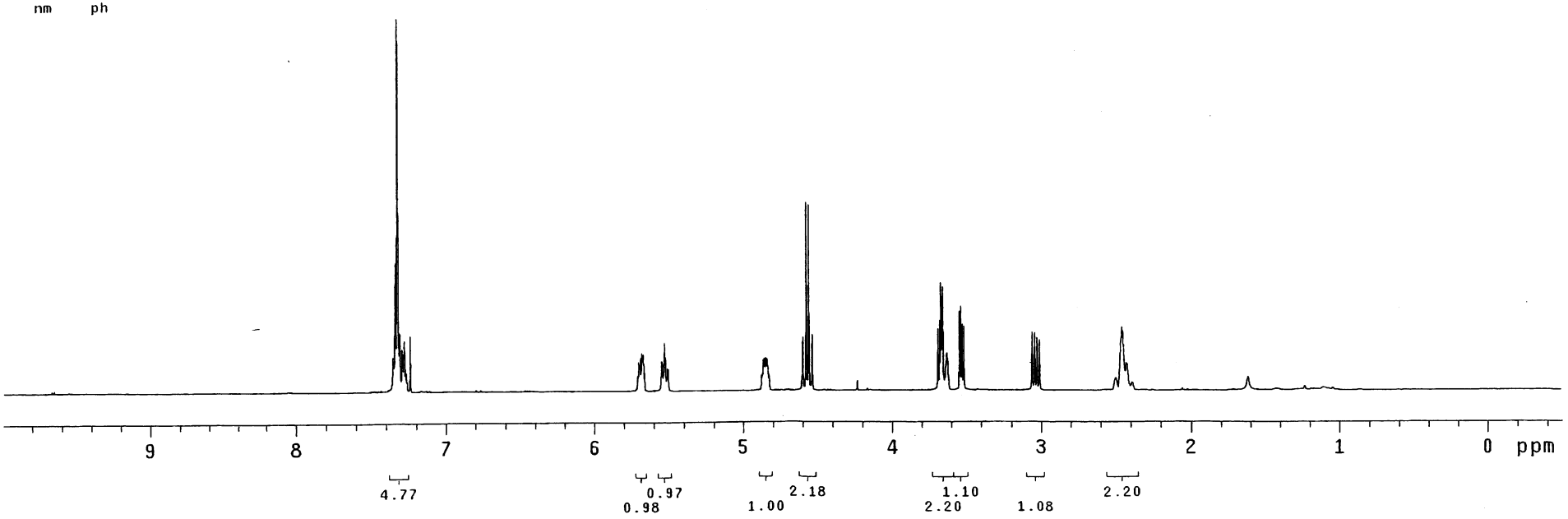
tn	H1	dmm	c
at	1.892	dmf	200
np	30272	dseq	
sw	8000.0	dres	1.0
fb	not used	homo	n
bs	4	temp	20.0
tpwr	51	PROCESSING	
pw	3.4	wtfile	
d1	1.000	proc	lp
tof	-140.0	fn	not used
nt	64	math	f
ct	16		
alock	n	werr	
gain	not used	wexp	
FLAGS		wbs	
il	n	wnt	wft
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-250.4		
wp	5248.4		
vs	59		
sc	0		
wc	250		
hzmm	20.99		
is	4095.97		
rfl	5277.2		
rfp	3619.0		
th	7		
ins	1.000		
nm	ph		



8

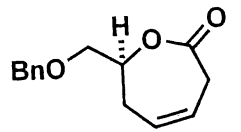


STANDARD CARBON PARAMETERS

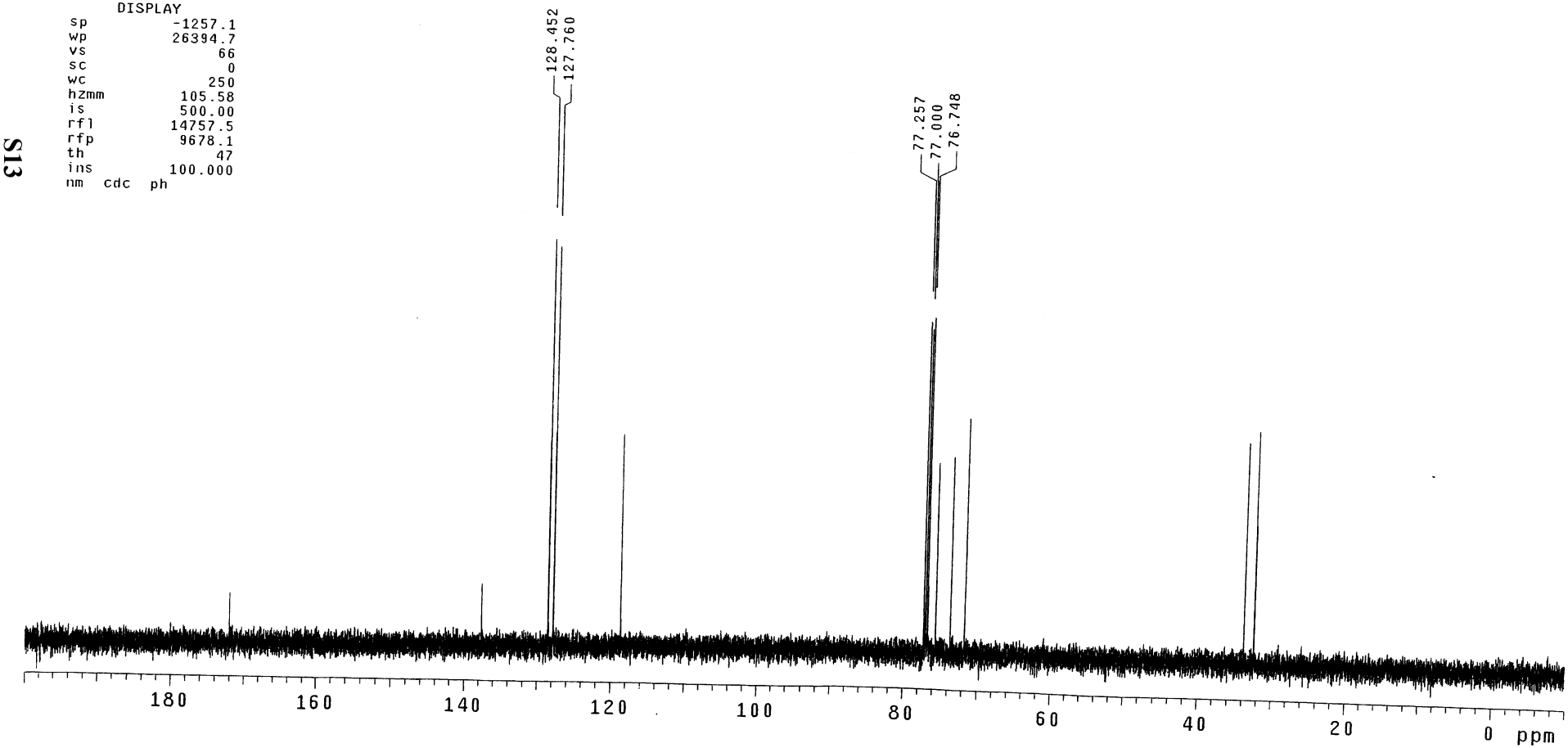
exp3 s2pul

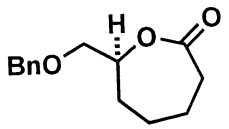
SAMPLE		DEC. & VT	
date	Jun 5 2008	dfrq	499.862
solvent	CDCl3	dn	H1
file	exp	dpwr	27
ACQUISITION		dof	0
sfrq	125.704	dm	yyy
tn	C13	dmm	w
at	1.298	dmf	12579
np	97994	dseq	
sw	37735.8	dres	1.0
fb	not used	homo	n
bs	16	temp	20.0
tpwr	57	PROCESSING	
pw	6.1	lb	0.50
d1	0.700	wtfile	
tof	1883.8	proc	ft
nt	5120	fn	131072
ct	112	math	f
alock	n		
gain	56	werr	
FLAGS		wexp	wft
il	n	wbs	wft
in	n	wnt	
dp	y		
hs	nn		
DISPLAY			
sp	-1257.1		
wp	26394.7		
vs	66		
sc	0		
wc	250		
hzmm	105.58		
is	500.00		
rfl	14757.5		
rfp	9678.1		
th	47		
ins	100.000		

nm cdc ph



8





10

STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

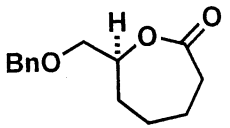
Solvent: CDCl3
Temp. 20.0 C / 293.1 K
INOVA-500 "varian"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.8593223 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 32 sec

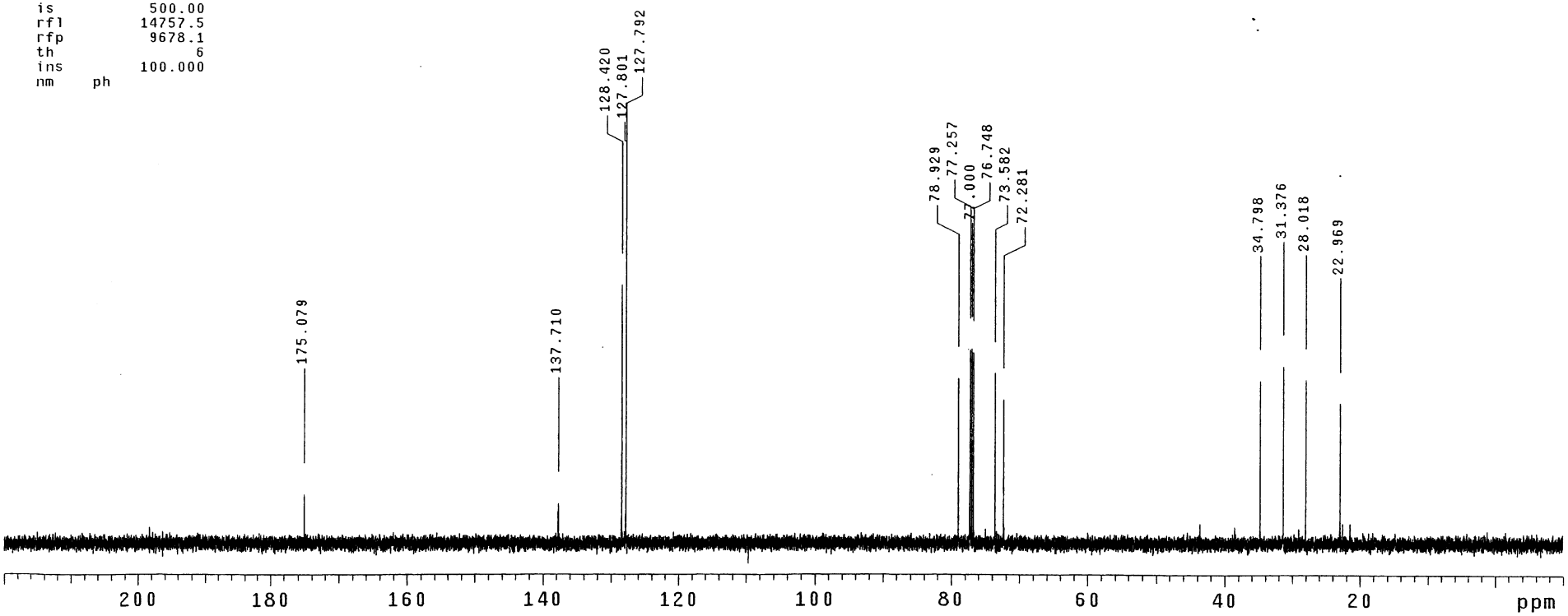
STANDARD CARBON PARAMETERS

exp3 s2pul

SAMPLE		DEC. & VT	
date	Jun 7 2008	dfrq	499.862
solvent	CDCl3	dn	H1
file	exp	dpwr	27
ACQUISITION		dof	0
sfrq	125.704	dm	yyy
tn	C13	dmm	w
at	1.298	dmf	12579
np	97994	dseq	
sw	37735.8	dres	1.0
fb	not used	homo	n
bs	16	temp	20.0
tpwr	57	PROCESSING	
pw	6.1	lb	0.50
d1	0.700	wtfile	
tof	1883.8	proc	ft
nt	5120	fn	131072
ct	160	math	f
alock	n		
gain	56	werr	
FLAGS		wexp	wft
il	n	wbs	wft
in	n	wnt	
dp	y		
hs	nn		
DISPLAY			
sp	-1257.1		
wp	28908.1		
vs	47		
sc	0		
wc	250		
hzmm	115.63		
is	500.00		
rfl	14757.5		
rfp	9678.1		
th	6		
ins	100.000		
nm	ph		



10

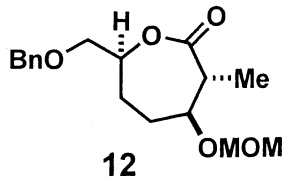


STANDARD PROTON PARAMETERS

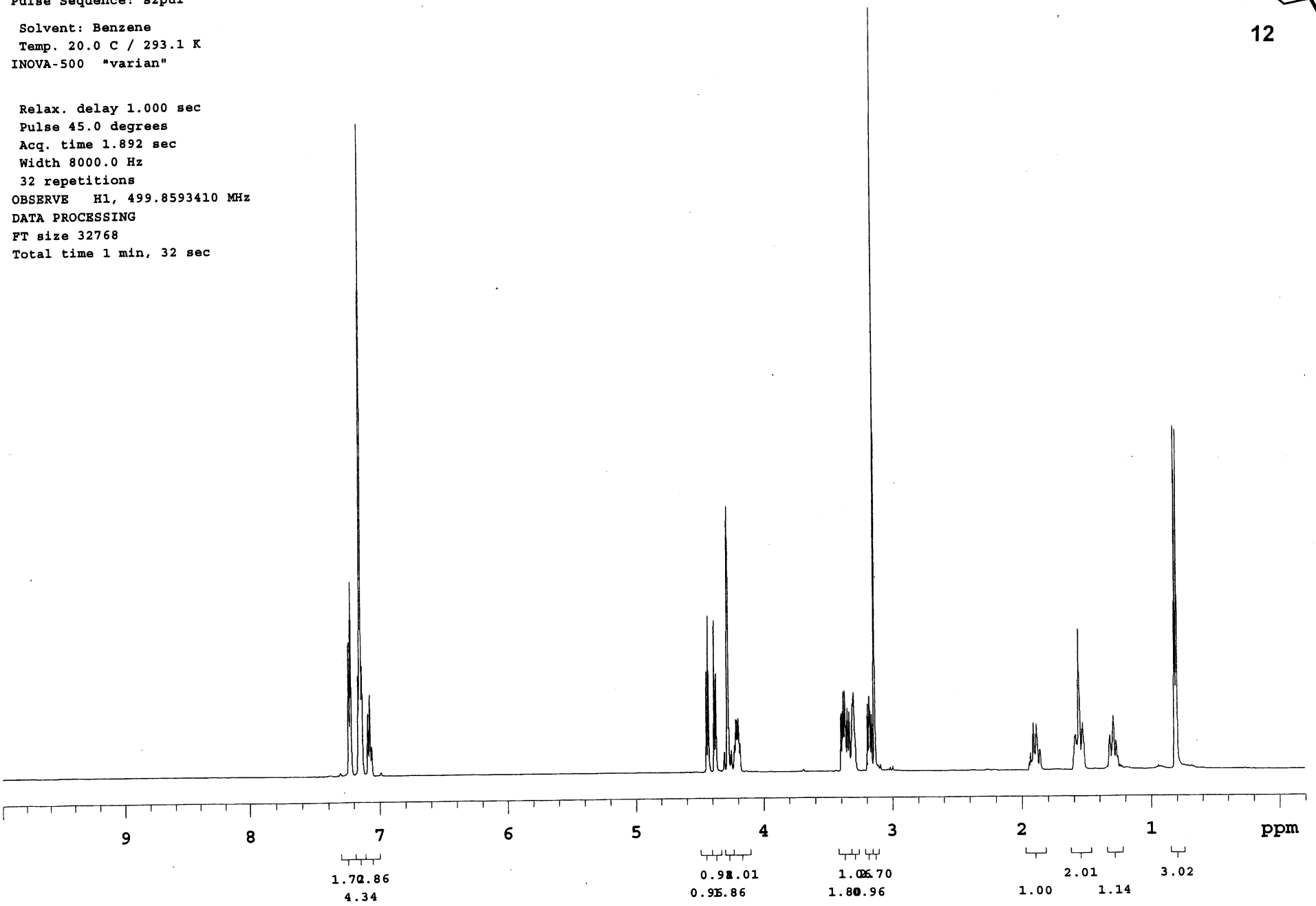
Pulse Sequence: s2pul

Solvent: Benzene
Temp. 20.0 C / 293.1 K
INOVA-500 "varian"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.8593410 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 32 sec



12

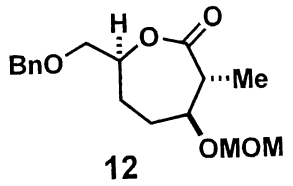


STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul

Solvent: Benzene
Temp. 20.0 C / 293.1 K
User: 1-14-87
INOVA-500 "varian"

Relax. delay 0.700 sec
Pulse 45.0 degrees
Acq. time 1.298 sec
Width 37735.8 Hz
560 repetitions
OBSERVE C13, 125.6896933 MHz
DECOUPLE H1, 499.8618491 MHz
Power 27 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 2 hr, 13 min, 50 sec



12

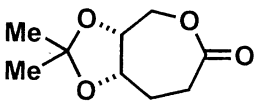
HF26109b/CDCl3

Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory: vnmr1_08Jul2002-151843
File: PROTON

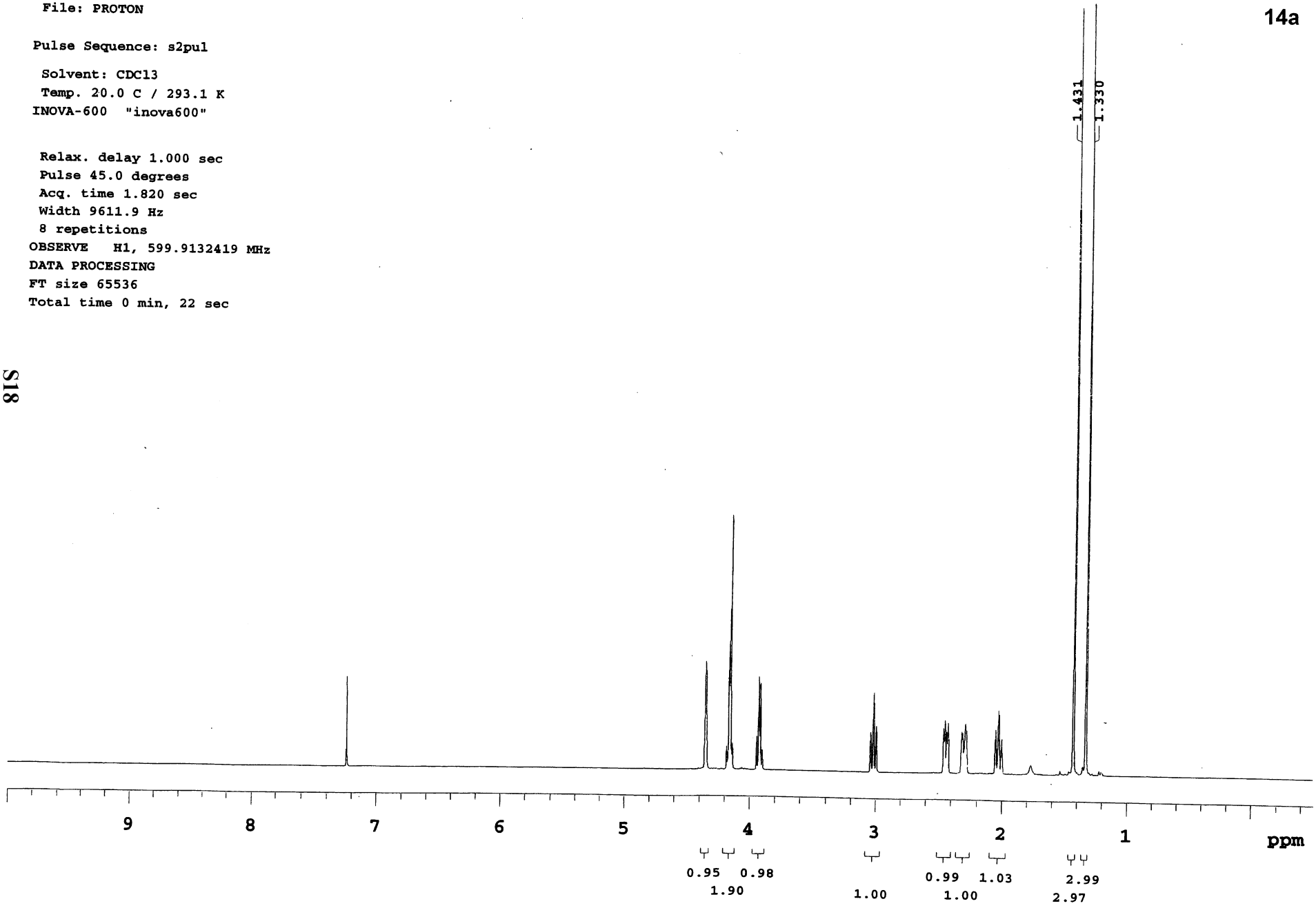
Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 20.0 C / 293.1 K
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.820 sec
Width 9611.9 Hz
8 repetitions
OBSERVE H1, 599.9132419 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 22 sec



14a

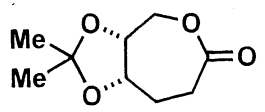


C13 STD parameter

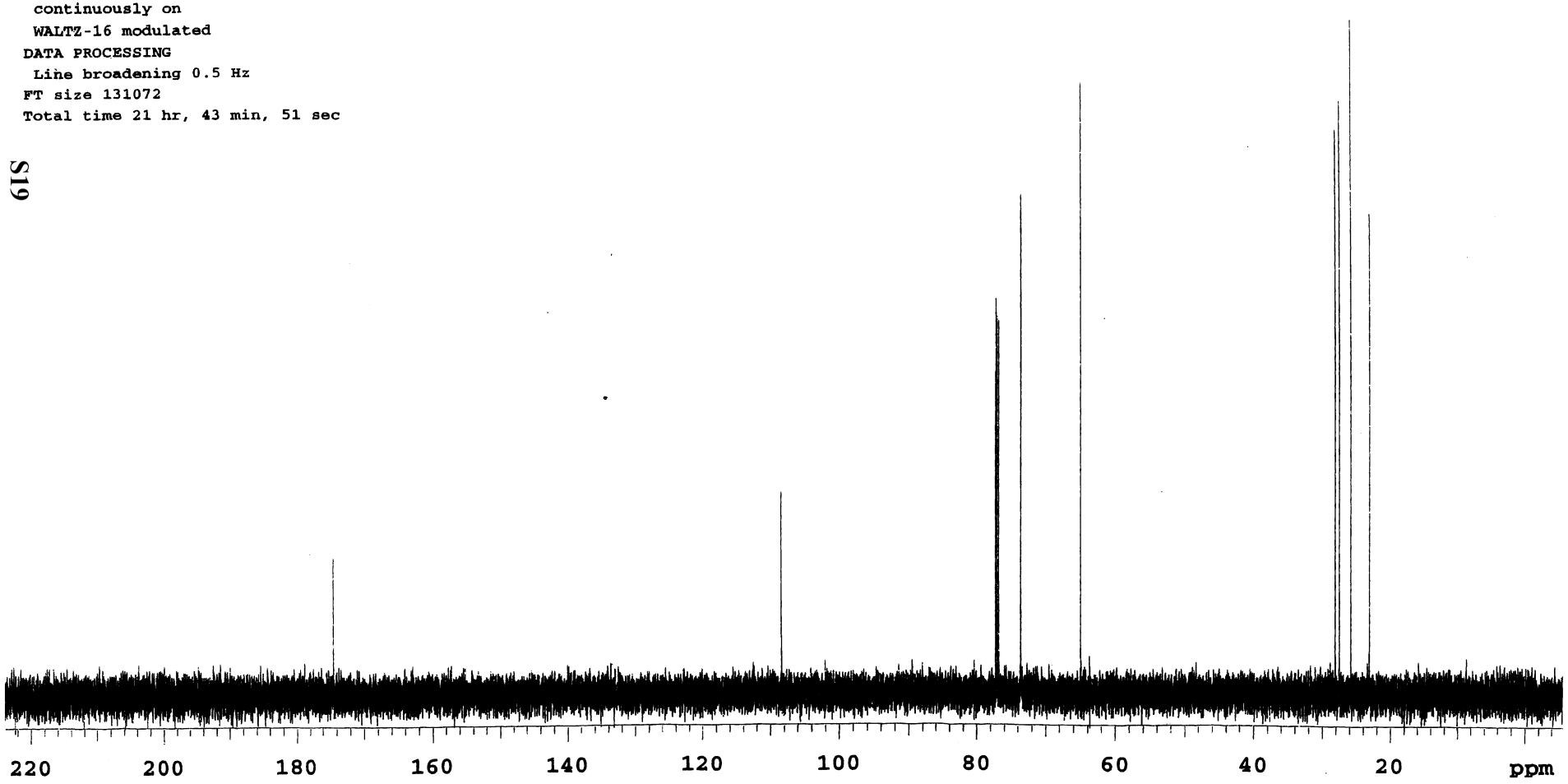
Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 20.0 C / 293.1 K
User: 1-14-87
INOVA-600 "inova600"

Relax. delay 0.689 sec
Pulse 45.0 degrees
Acq. time 0.867 sec
Width 34632.0 Hz
64 repetitions
OBSERVE C13, 150.8483119 MHz
DECOUPLE H1, 599.9152462 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 21 hr, 43 min, 51 sec



14a



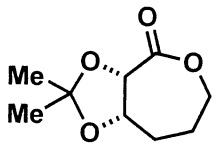
HF26109/CDCl3

Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory: vnmr1_08Jul2002-151843
File: PROTON

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 20.0 C / 293.1 K
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.820 sec
Width 9611.9 Hz
8 repetitions
OBSERVE H1, 599.9132422 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 22 sec



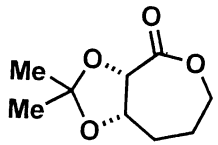
14b

C13 STD parameter

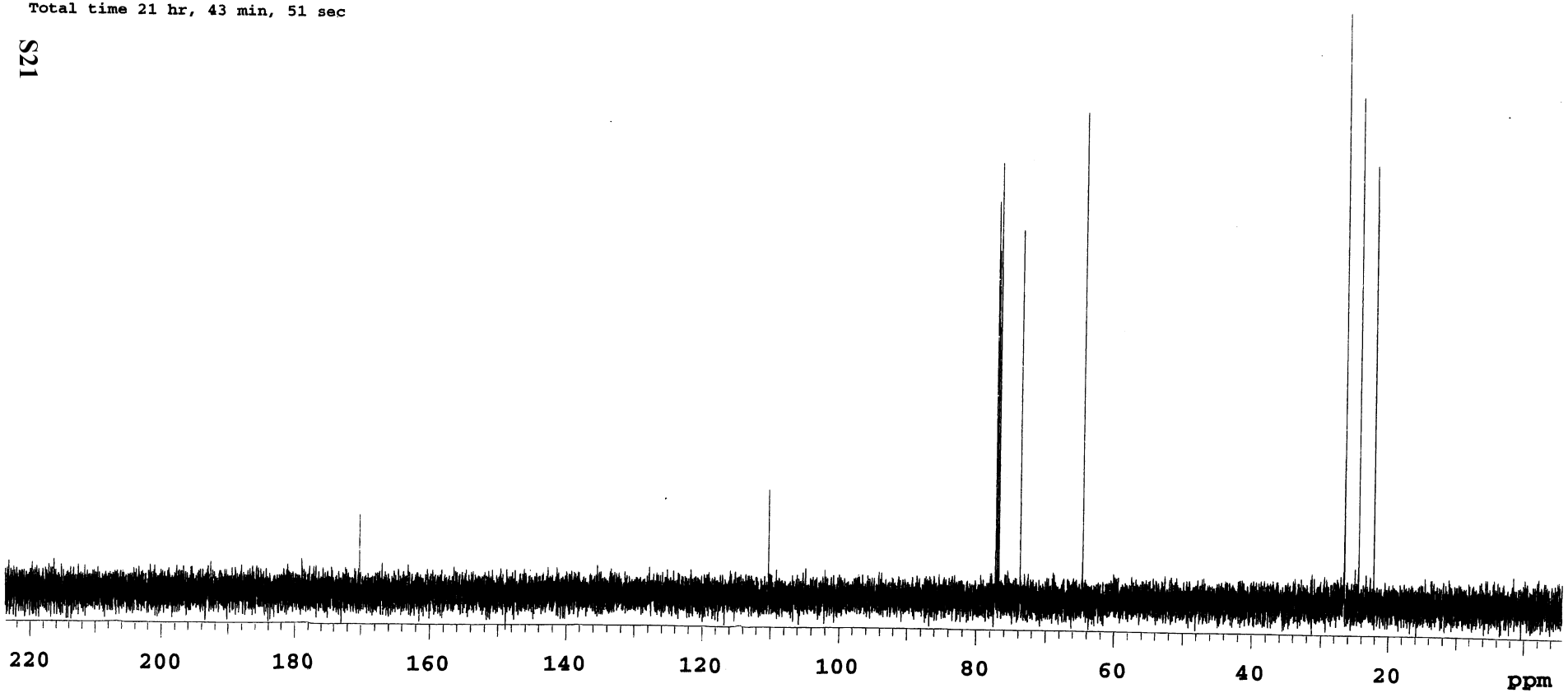
Pulse Sequence: s2pul

Solvent: CDC13
Temp. 20.0 C / 293.1 K
User: 1-14-87
INOVA-600 "inova600"

Relax. delay 0.689 sec
Pulse 45.0 degrees
Acq. time 0.867 sec
Width 34632.0 Hz
96 repetitions
OBSERVE C13, 150.8483098 MHz
DECOUPLE H1, 599.9152462 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 21 hr, 43 min, 51 sec



14b

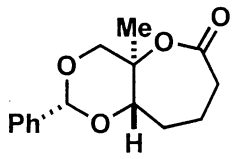


STANDARD PROTON PARAMETERS

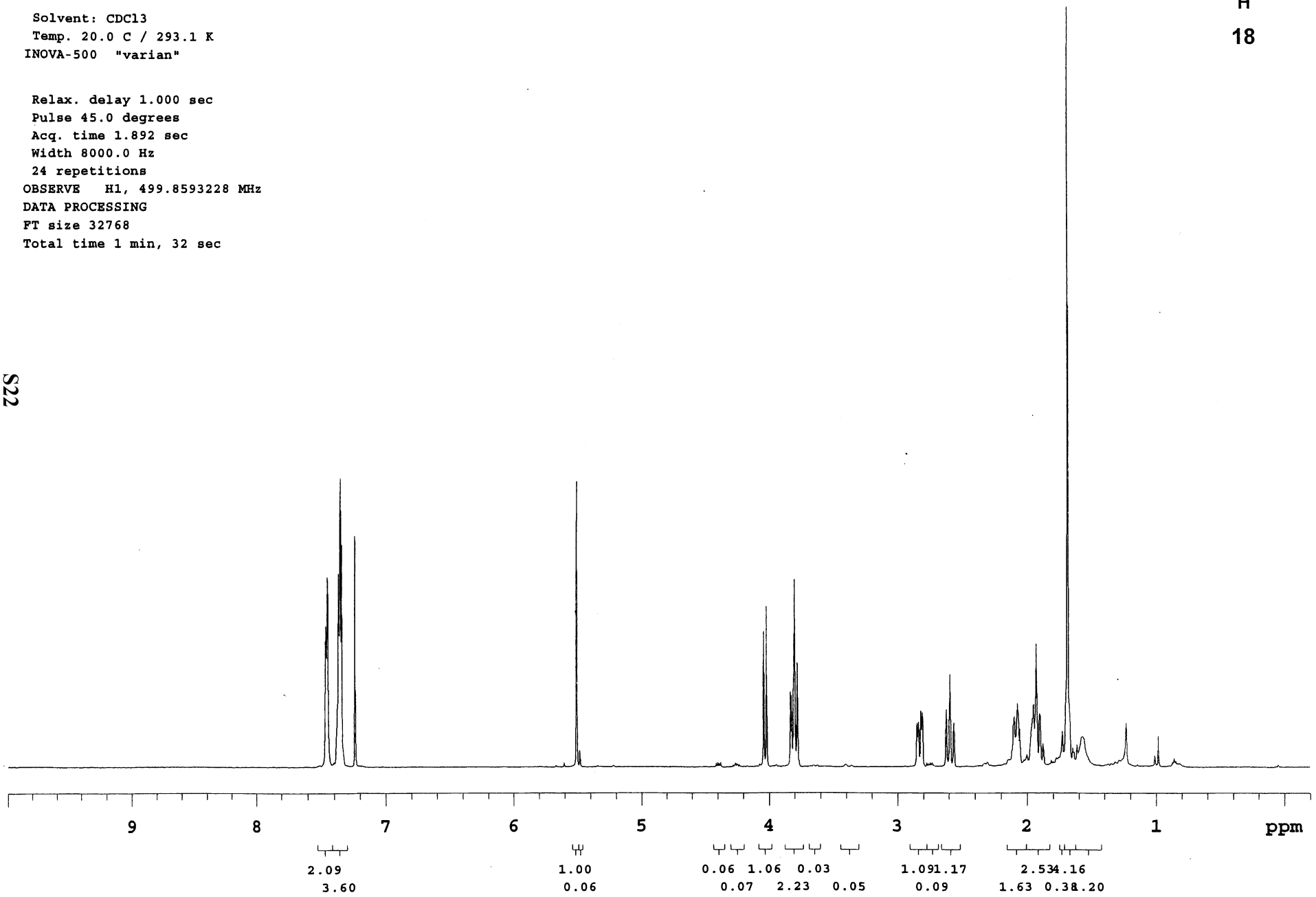
Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 20.0 C / 293.1 K
INOVA-500 "varian"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
24 repetitions
OBSERVE H1, 499.8593228 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 32 sec



18

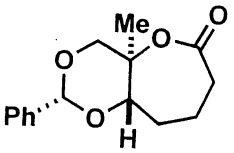


STANDARD CARBON PARAMETERS

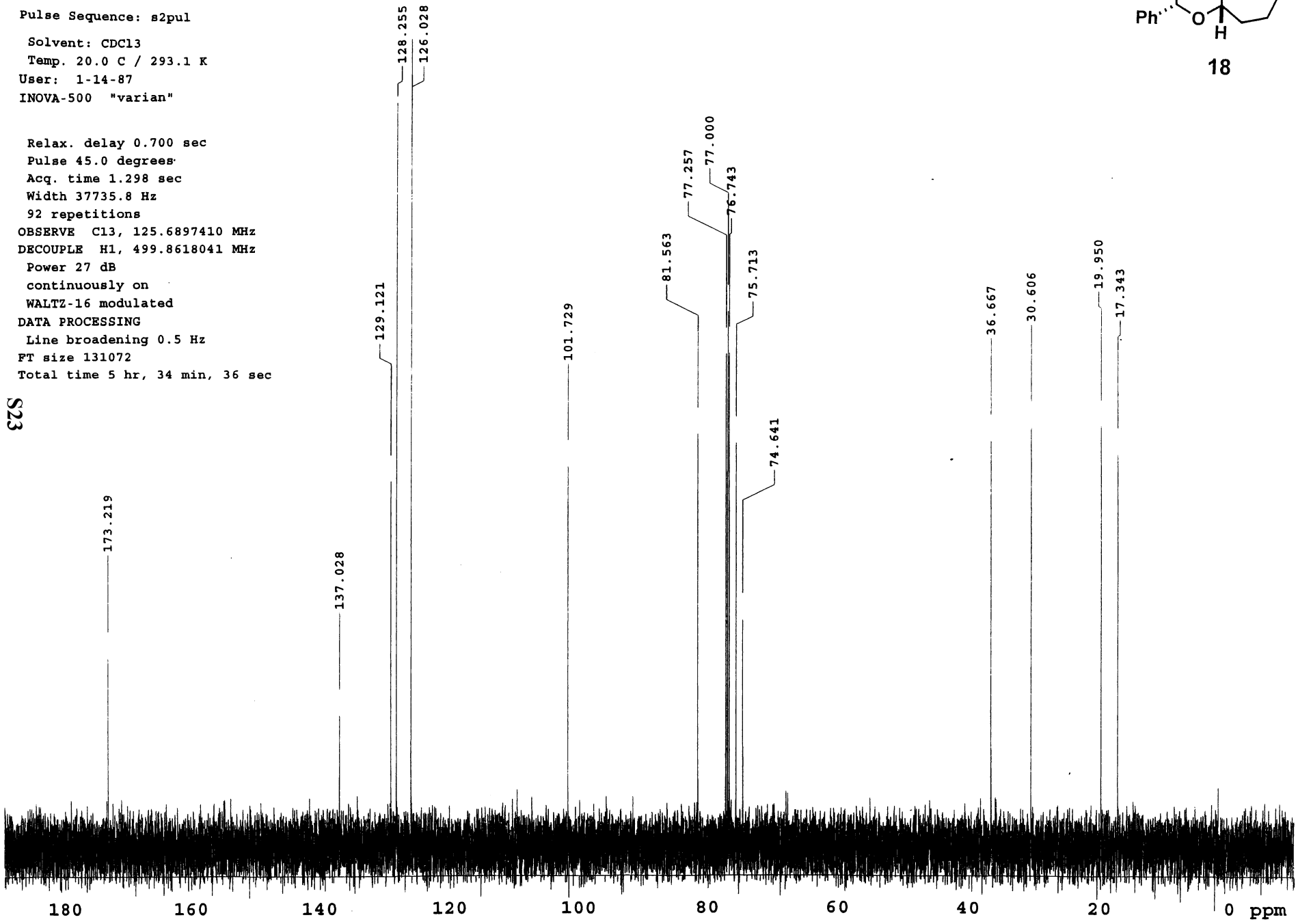
Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 20.0 C / 293.1 K
User: 1-14-87
INOVA-500 "varian"

Relax. delay 0.700 sec
Pulse 45.0 degrees
Acq. time 1.298 sec
Width 37735.8 Hz
92 repetitions
OBSERVE C13, 125.6897410 MHz
DECOUPLE H1, 499.8618041 MHz
Power 27 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 5 hr, 34 min, 36 sec



18

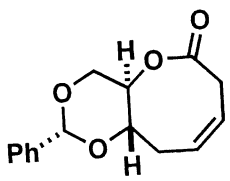


SugaI-39/CDCl3

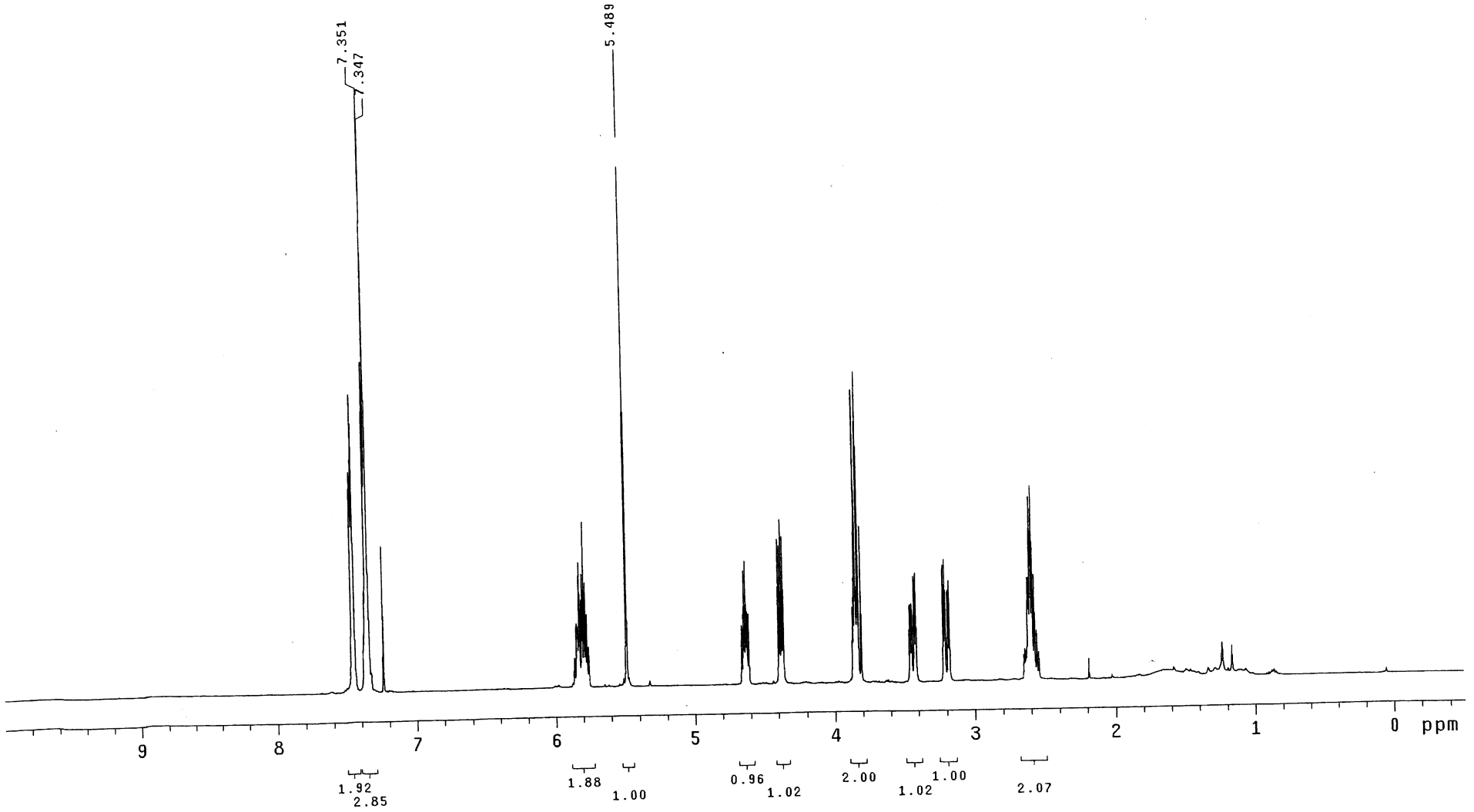
Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 20.0 C / 293.1 K
INOVA-500 "varian"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.8593223 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 23 sec



20

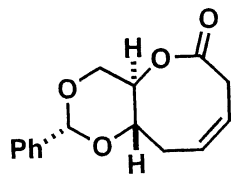


STANDARD CARBON PARAMETERS

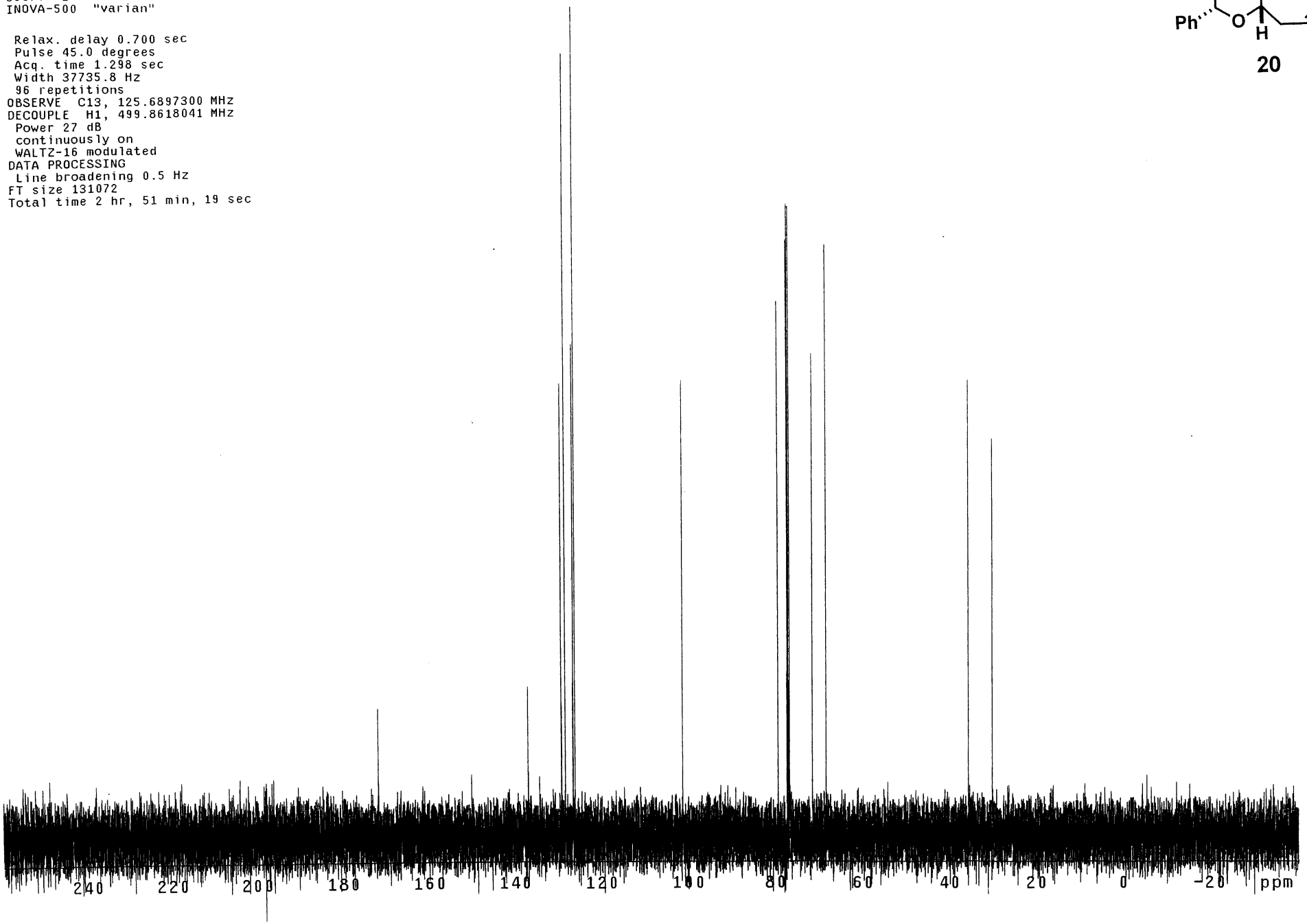
Pulse Sequence: s2pul

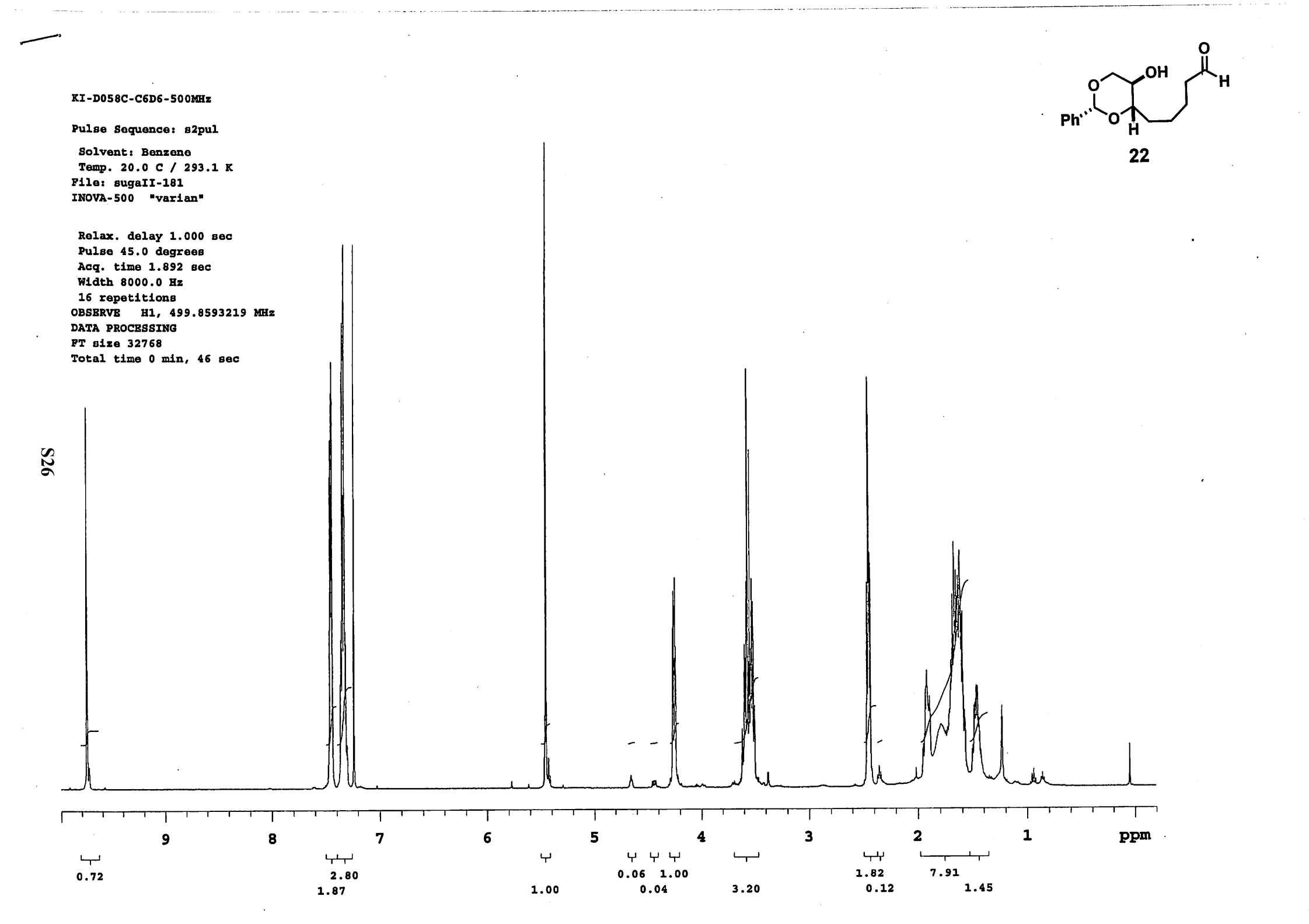
Solvent: CDCl3
Temp. 20.0 C / 293.1 K
User: 1-14-87
INOVA-500 "varian"

Relax. delay 0.700 sec
Pulse 45.0 degrees
Acq. time 1.298 sec
Width 37735.8 Hz
96 repetitions
OBSERVE C13, 125.6897300 MHz
DECOUPLE H1, 499.8618041 MHz
Power 27 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 2 hr, 51 min, 19 sec



20



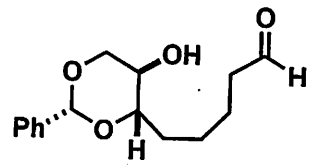

KI-D058C-C6D6-500MHz
Pulse Sequence: s2pul
Solvent: Benzene
Temp. 20.0 C / 293.1 K
File: sugaII-181
INOVA-500 "varian"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.8593219 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 46 sec
OH
O
H
O
Ph
O
H
22
9
8
7
6
5
4
3
2
1
ppm
0.72
2.80
1.87
1.00
0.06
1.00
0.04
3.20
1.82
0.12
7.91
1.45

C13 STD parameter

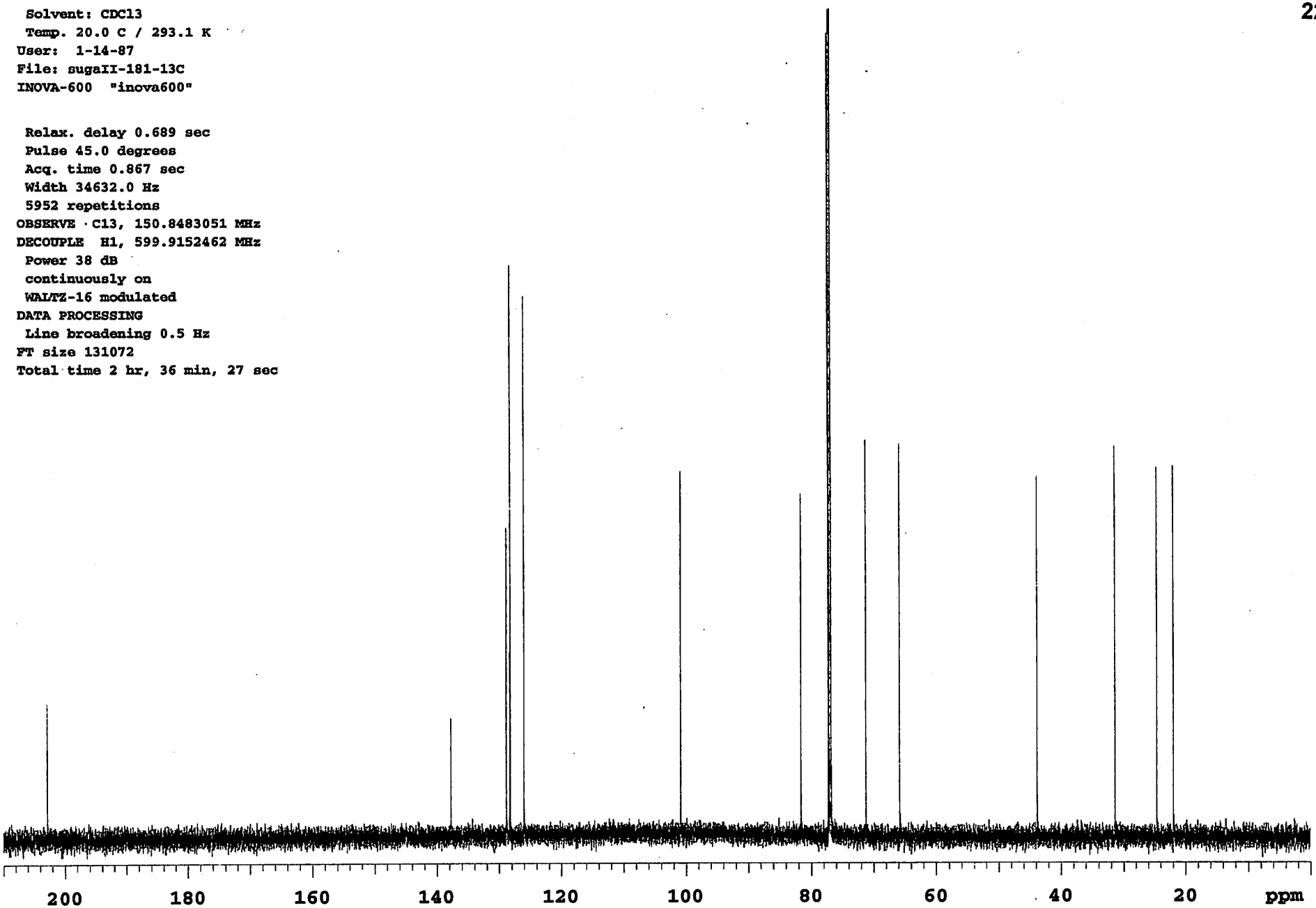
Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 20.0 C / 293.1 K
User: 1-14-87
File: sugaII-181-13C
INOVA-600 "inova600"

Relax. delay 0.689 sec
Pulse 45.0 degrees
Acq. time 0.867 sec
Width 34632.0 Hz
5952 repetitions
OBSERVE C13, 150.8483051 MHz
DECOUPLE H1, 599.9152462 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 2 hr, 36 min, 27 sec



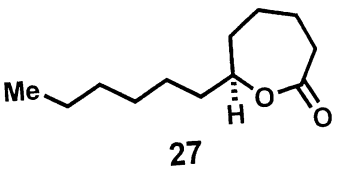
22



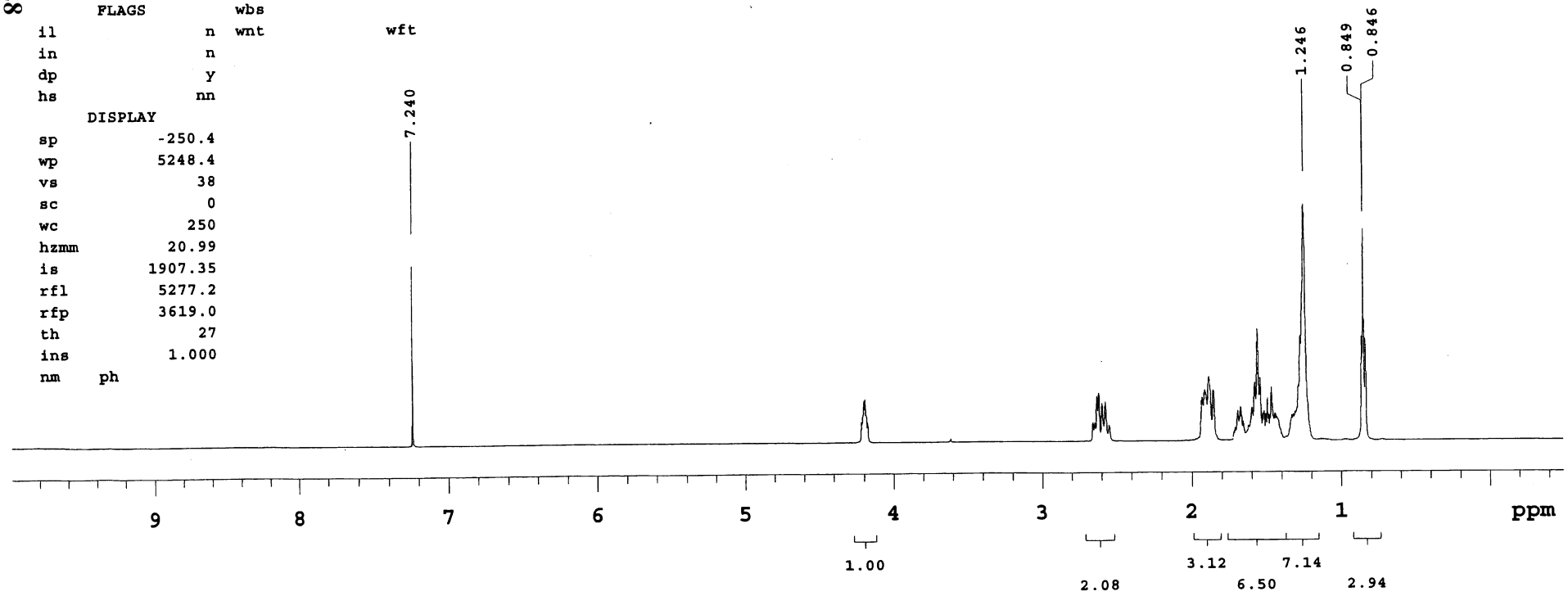
STANDARD PROTON PARAMETERS

exp5 s2pul

SAMPLE		DEC. & VT	
date	Feb 14 2009	dfrq	499.862
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.862	dm	nnn
tn	H1	dmm	c
at	1.892	dmf	200
np	30272	dseq	
sw	8000.0	dres	1.0
fb	not used	homo	n
bs	4	temp	20.0
tpwr	51	PROCESSING	
pw	3.4	wtfile	
d1	1.000	proc	lp
tof	-140.0	fn	not used
nt	16	math	f
ct	16		
alock	n	werr	
gain	not used	wexp	
FLAGS		wbs	
il	n	wnt	wft
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-250.4		
wp	5248.4		
vs	38		
sc	0		
wc	250		
hzmm	20.99		
is	1907.35		
rfl	5277.2		
rfp	3619.0		
th	27		
ins	1.000		
nm	ph		



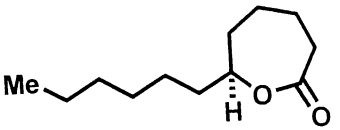
27



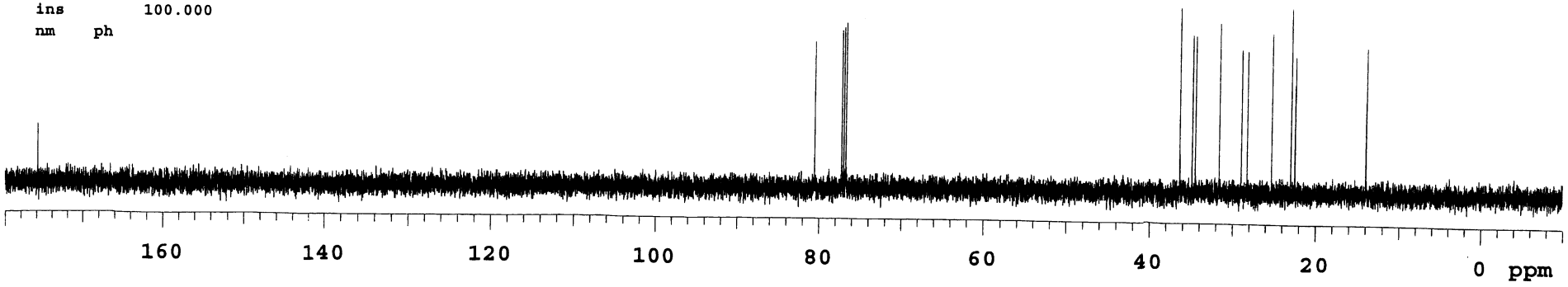
STANDARD CARBON PARAMETERS

exp4 s2pul

SAMPLE		DEC. & VT	
date	Feb 14 2009	dfrq	499.862
solvent	CDCl3	dn	H1
file	exp	dpwr	27
ACQUISITION		dof	0
sfrq	125.704	dm	YYY
tn	C13	dmm	w
at	1.298	dmf	12579
np	97994	dseq	
sw	37735.8	dres	1.0
fb	not used	homo	n
bs	16	temp	20.0
tpwr	57	PROCESSING	
pw	6.1	lb	0.50
d1	0.700	wtfile	
tof	1883.8	proc	ft
nt	5120	fn	131072
ct	64	math	f
alock	n		
gain	56	werr	
FLAGS		wexp	wft
il	n	wbs	wft
in	n	wnt	
dp	y		
hs	nn		
DISPLAY			
sp	-1257.1		
wp	23880.7		
vs	30		
sc	0		
wc	250		
hzmm	95.52		
is	500.00		
rfl	14755.2		
rfp	9678.1		
th	34		
ins	100.000		
nm	ph		



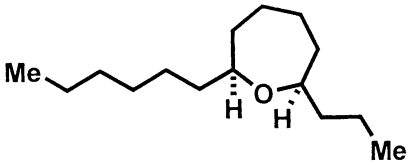
27



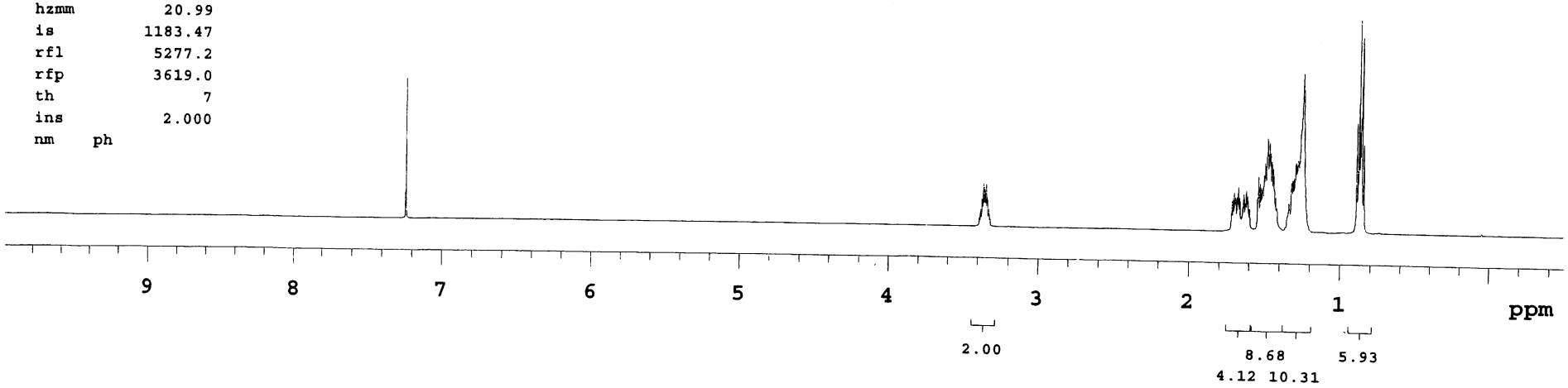
STANDARD PROTON PARAMETERS

exp2 s2pul

SAMPLE		DEC. & VT	
date	Feb 14 2009	dfrq	499.862
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.862	dm	nnn
tn	H1	dmm	c
at	1.892	dmf	200
np	30272	dseq	
sw	8000.0	dres	1.0
fb	not used	homo	n
bs	4	temp	20.0
tpwr	51	PROCESSING	
pw	3.4	wtfile	
d1	1.000	proc	lp
tof	-140.0	fn	not used
nt	64	math	f
ct	16		
alock	n	werr	
gain	not used	wexp	
FLAGS		wbs	
il	n	wnt	wft
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-250.4		
wp	5248.4		
vs	34		
sc	0		
wc	250		
hzmm	20.99		
is	1183.47		
rfl	5277.2		
rfp	3619.0		
th	7		
ins	2.000		
nm	ph		



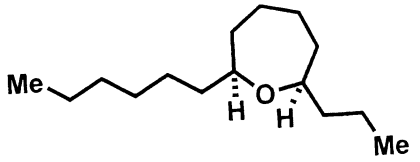
23: (±)-isolaurepan



STANDARD CARBON PARAMETERS

exp3 s2pul

SAMPLE		DEC. & VT	
date	Feb 14 2009	dfrq	499.862
solvent	CDCl3	dn	H1
file	exp	dpwr	27
ACQUISITION		dof	0
sfrq	125.704	dm	YYY
tn	C13	dmm	w
at	1.298	dmf	12579
np	97994	dseq	
sw	37735.8	dres	1.0
fb	not used	homo	n
bs	16	temp	20.0
tpwr	57	PROCESSING	
pw	6.1	lb	0.50
d1	0.700	wtfile	
tof	1883.8	proc	ft
nt	5120	fn	131072
ct	128	math	f
alock	n		
gain	56	werr	
FLAGS		wexp	wft
il	n	wbs	wft
in	n	wnt	
dp	y		
hs	nn		
DISPLAY			
sp	-1257.1		
wp	23880.7		
vs	60		
sc	0		
wc	250		
hzmm	95.52		
is	500.00		
rfl	14752.3		
rfp	9678.1		
th	58		
ins	100.000		
nm	ph		



23: (±)-isolaurepan