

Supplementary information for:

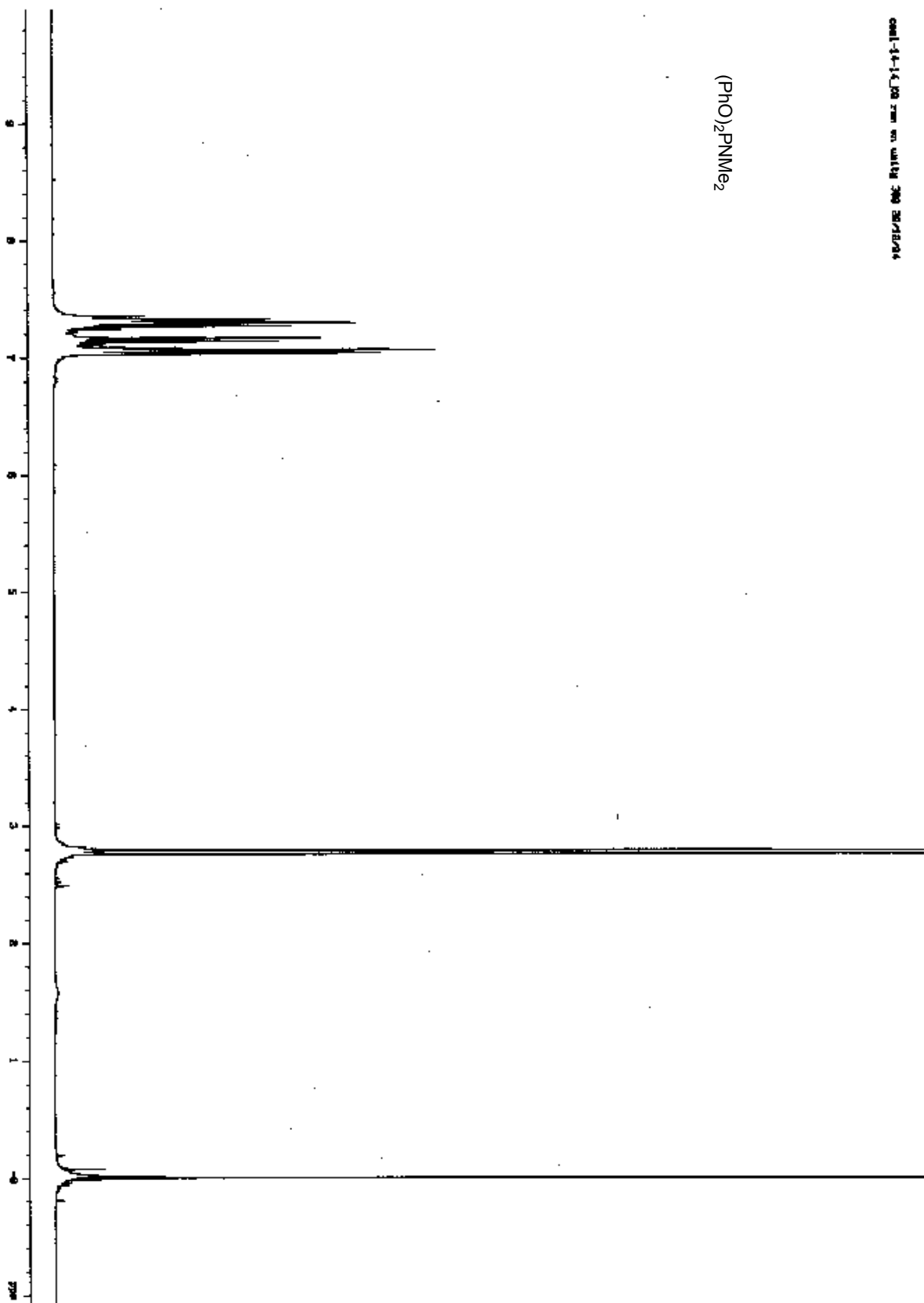
Galactose-derived phosphonate analogues of inositol-1-phosphate and phosphatidylinositol

Zoran Dinev,^{a,b} Carlie T. Gannon,^{a,b} Caroline Egan,^{a,b} Jacinta A. Watt,^{a,b} Malcolm J. McConville^{b,c} and Spencer J. Williams^{*a,b}

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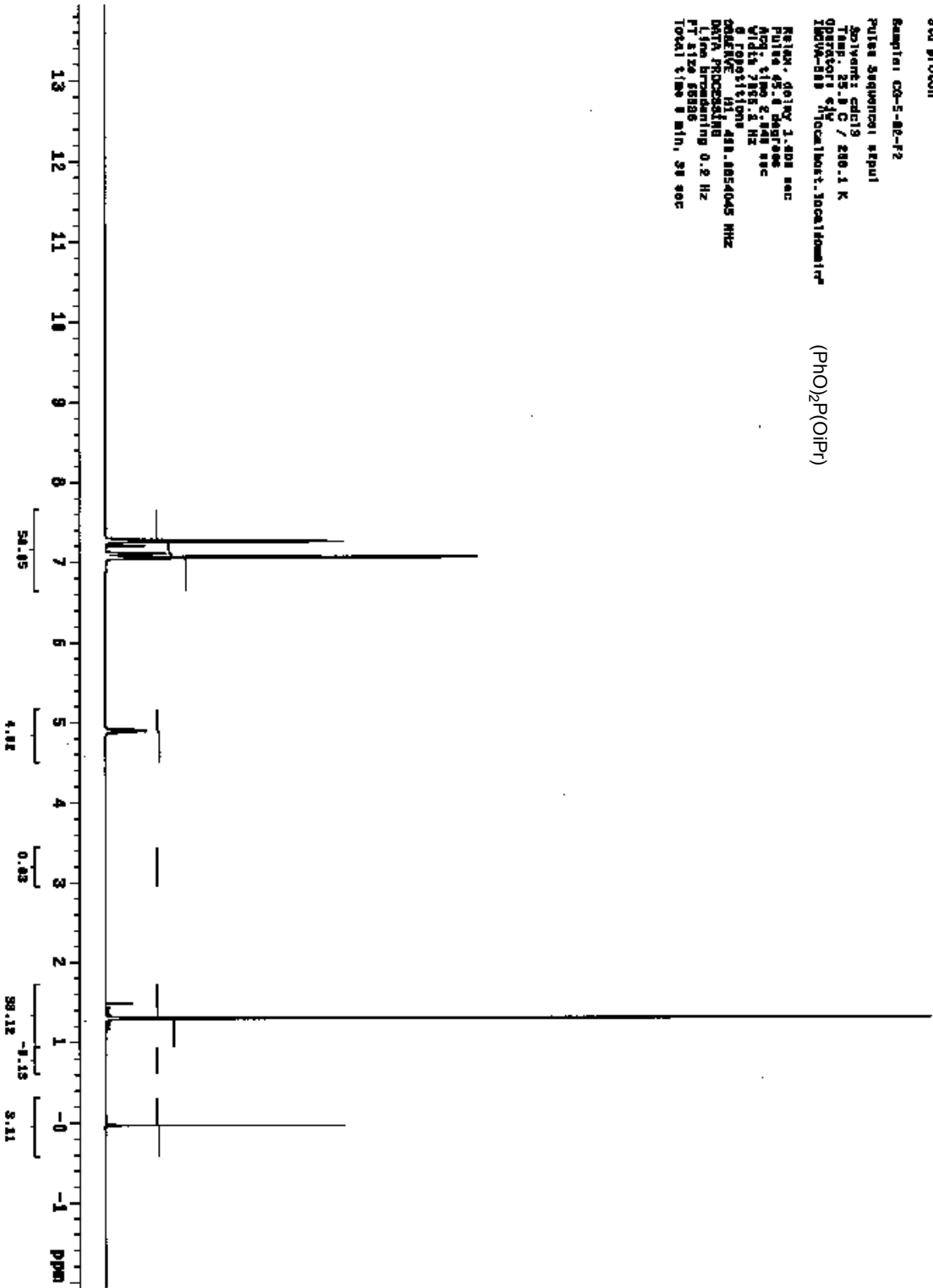
- S2: ¹H NMR for (PhO)₂PNMe₂
- S3: ¹H NMR for (PhO)₂P(OiPr)
- S4: ¹³C NMR for (PhO)₂P(OiPr)
- S5: ¹H NMR for **3**
- S6: ¹³C NMR for **3**
- S7: ¹H NMR for **7**
- S8: ¹³C NMR for **7**
- S9: ³¹P NMR for **7**
- S10: ¹H NMR for **8**
- S11: ¹³C NMR for **8**
- S12: ³¹P NMR for **8**
- S13: ¹H NMR for **11**
- S14: ¹³C NMR for **11**
- S15: ³¹P NMR for **11**
- S16: ¹H NMR for **12**
- S17: ¹³C NMR for **12**
- S18: ³¹P NMR for **12**
- S19: ¹H NMR for **13**
- S20: ¹³C NMR for **13**
- S21: ¹H NMR for **1**
- S22: ³¹P NMR for **1**
- S23: ³¹P NMR for **2**
- S24: HRMS data for **2**
- S25: HPTLC analysis of octyl α -D-mannoside control experiment.

cont-14-14.DJ2 run on date 200 08/18/04

 $(\text{PhO})_2\text{PNiMe}_2$ 

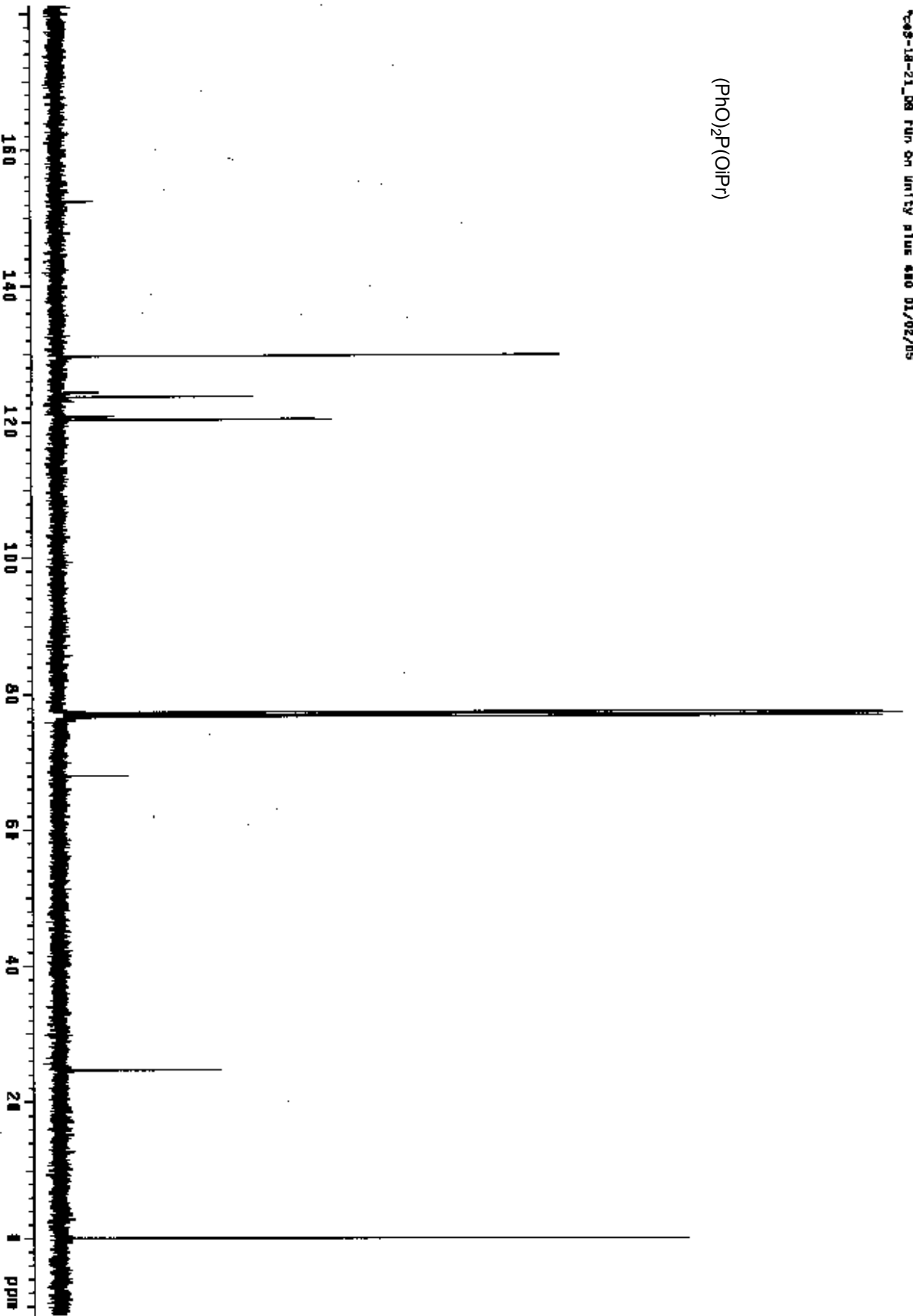
Std proton
 Sampiran CO-5-82-F2
 Pulsa Sequence: sfpul
 Solvent: cdcl3
 Temp: 45.0 C / 280.1 K
 Q: 10000 Hz
 P: 10000 Hz
 I: 10000 Hz
 Local: 10000 Hz
 Reten: 40100.100 sec
 Pulse: 95.0 degree
 Acq: 1.00 sec
 SFO: 100.625 MHz
 Q: 10000 Hz
 Q: 10000 Hz
 Q: 10000 Hz
 Data: 10000 Hz
 Data: 10000 Hz
 FT size: 65536
 Total time: 8 min, 38 sec

(PhO)₂P(O)Pr

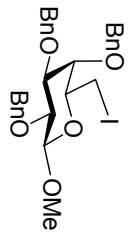


cas-18-21_08 run on unity plus 880 01/02/05

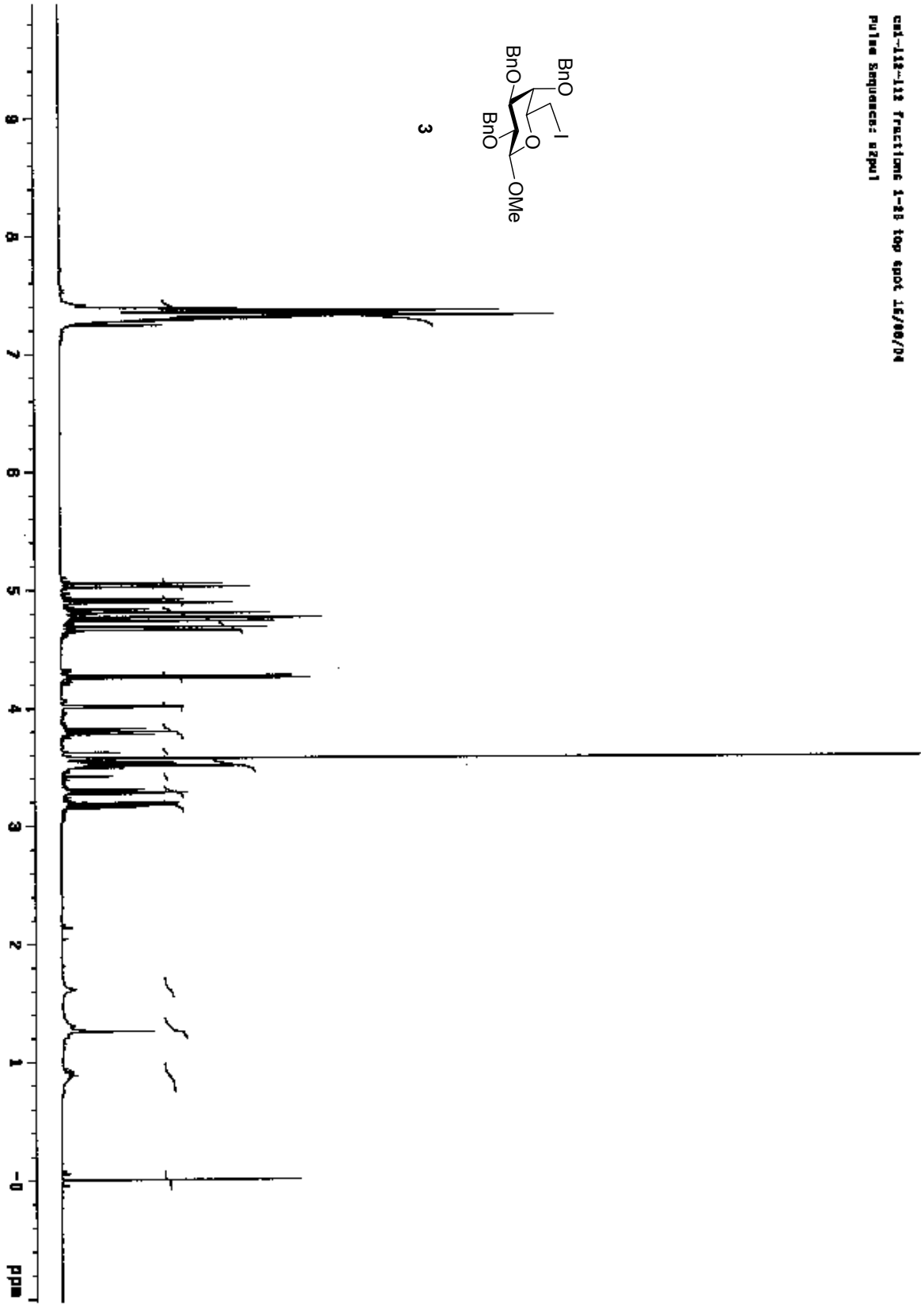
(PhO)₂P(O)Pr₁



cat-118-118 Fraction 1-28 top spot 16/09/04
Pulse Sequence: zgpg1

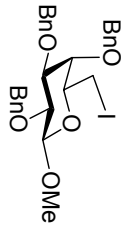


3

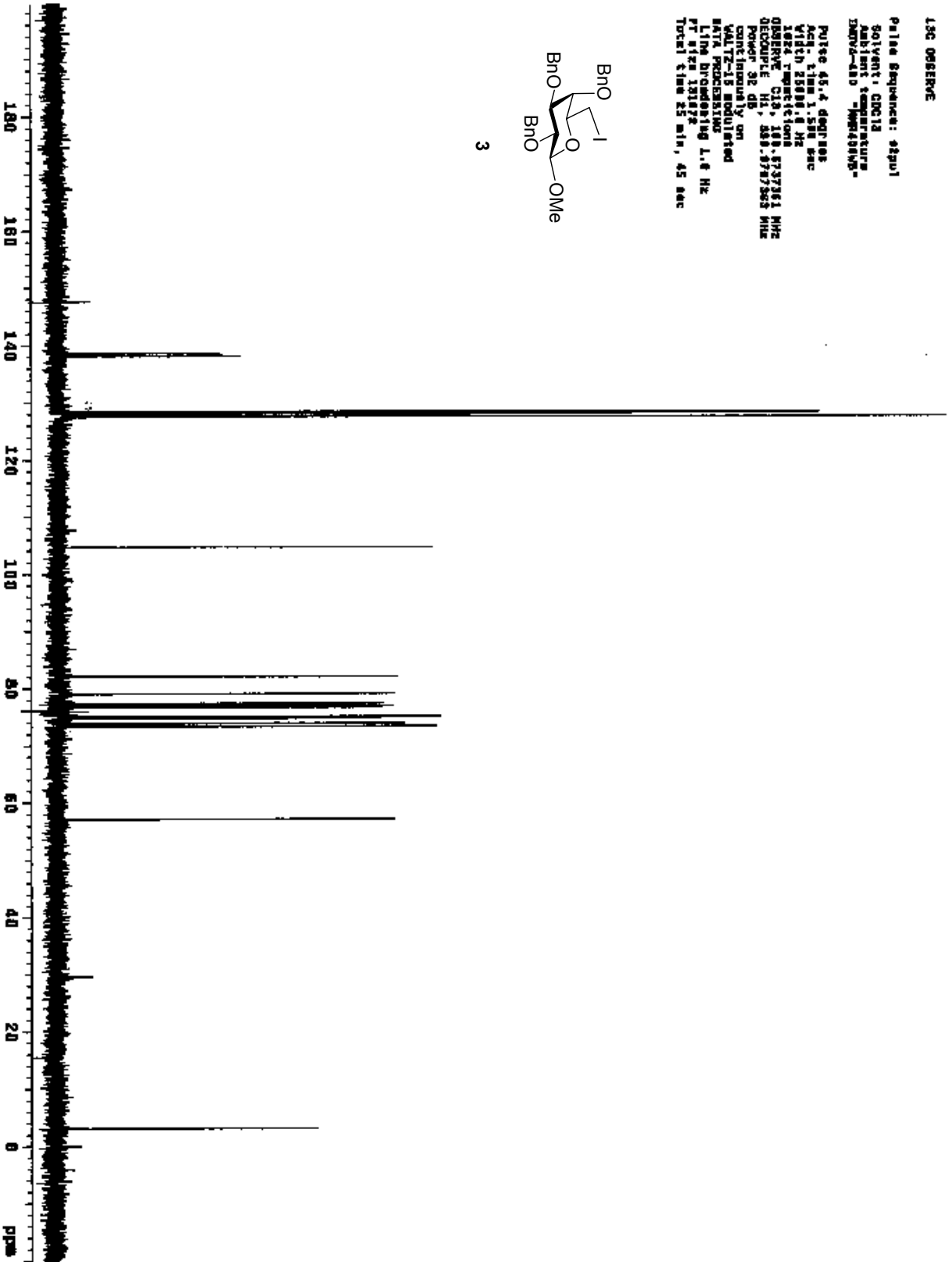


13C NMR SPECTRUM

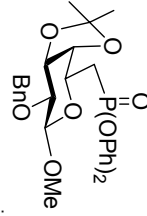
Pulse Sequence: zgpg30
Solvent: CDCl3
Ambient temperature
SAMPLING: 400 MHz
Pulse 45.4 degrees
Acq. Time 1.580 sec
Width 25000.0 Hz
1st 2nd 3rd 4th 5th 6th 7th 8th 9th 10th
OBSERVE C19, 100.873961 MHz
DECUPLE H1, 500.873961 MHz
Power 32 dB
canceled on
WALTZ-16 MODULATED
DATA PROCESSING
LINE BROADENING 1.0 Hz
PT 1.000000
PT 1.000000
Total time 15 min, 45 sec



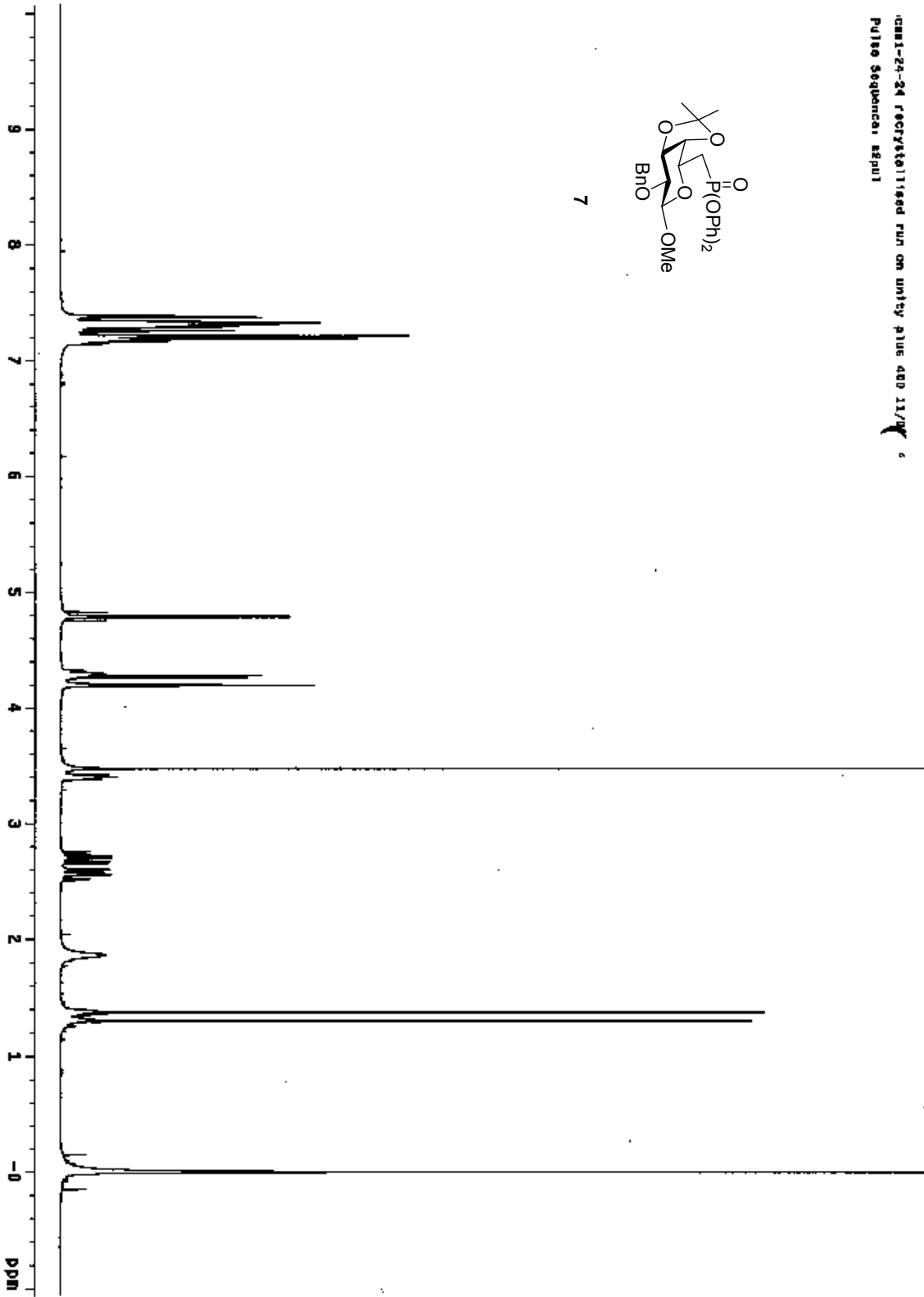
3



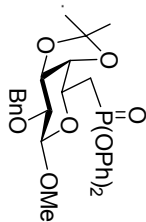
NAME: 24-24 (acrylate) used run on unit by plus AND 11/11/11
Pulse Sequence: zgpg30



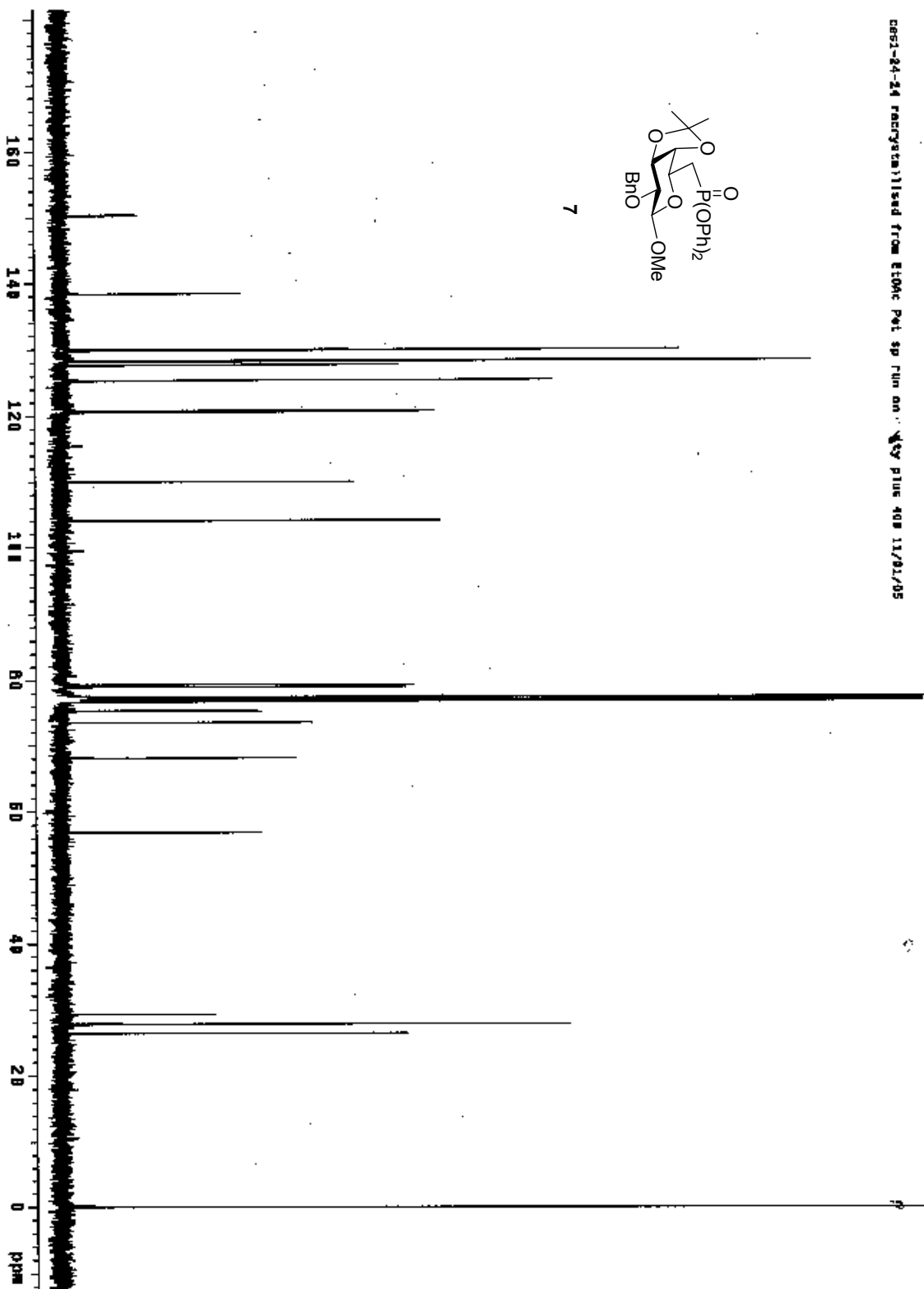
7



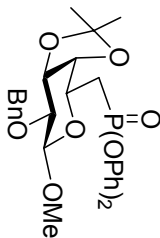
0661-84-84 recrystallised from EtOAc Pet sp run on 400 plus 400 11/01/05



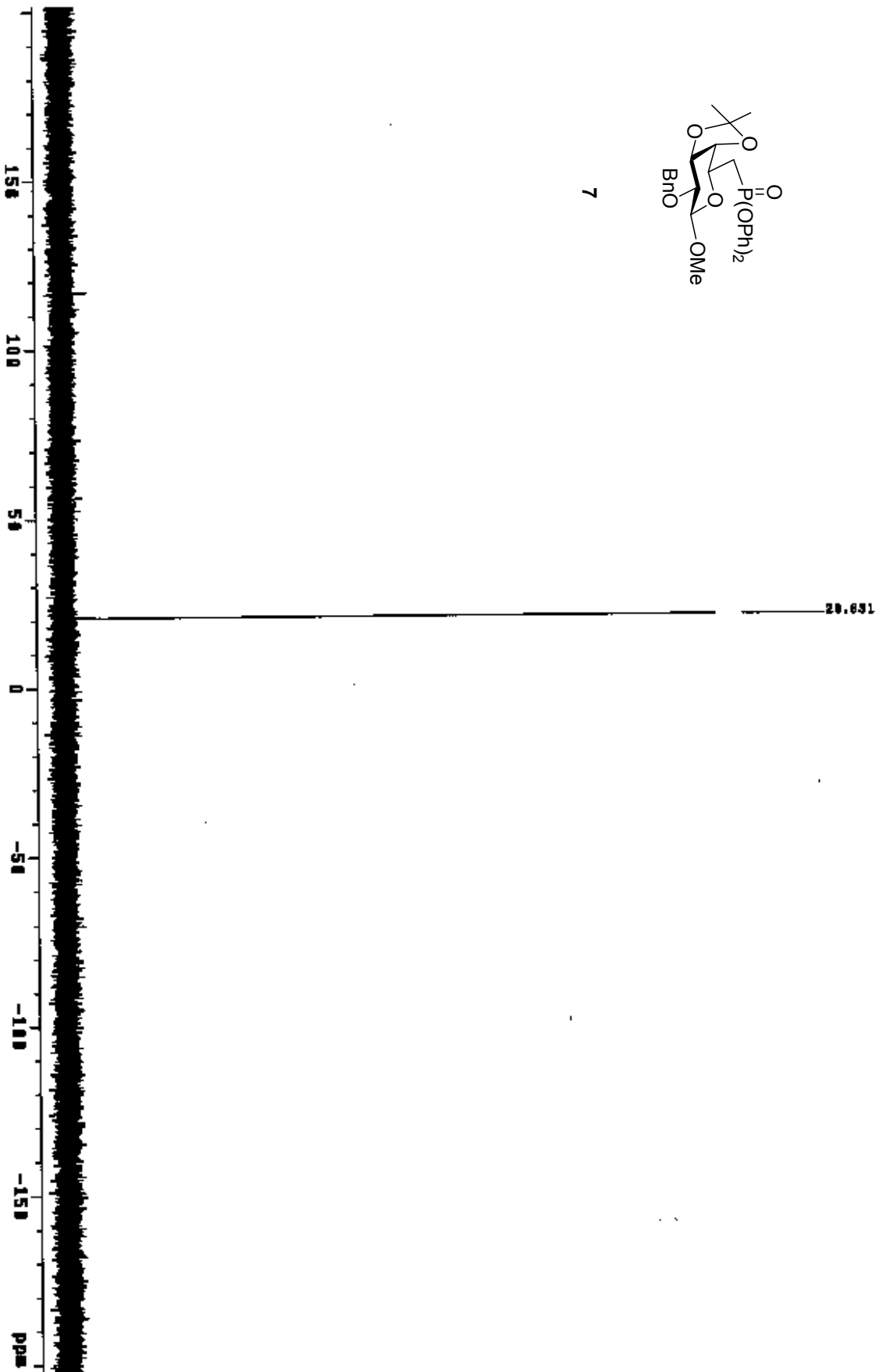
7



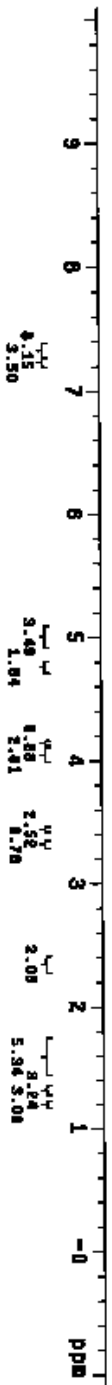
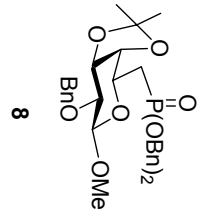
31P
 CAS-24-28_P021-39P4 run on INOVA 400 04/04/05
 Pulse Sequence: zgpg31



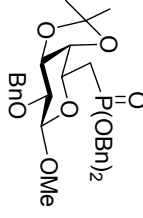
7



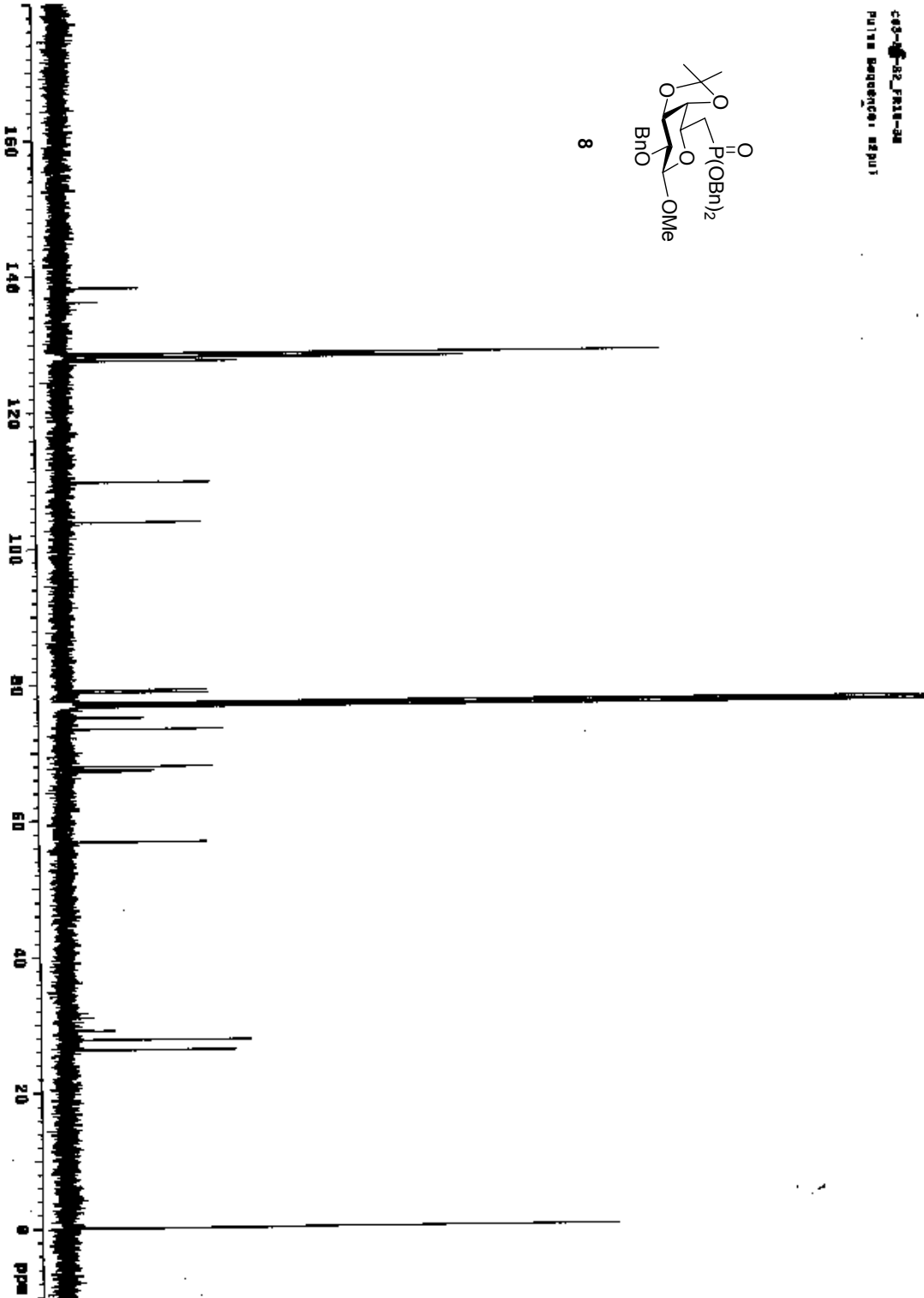
3-3-ALLPATICES combined batches of ribose/ribosephosphate
Pulse Bankancai ezgüi



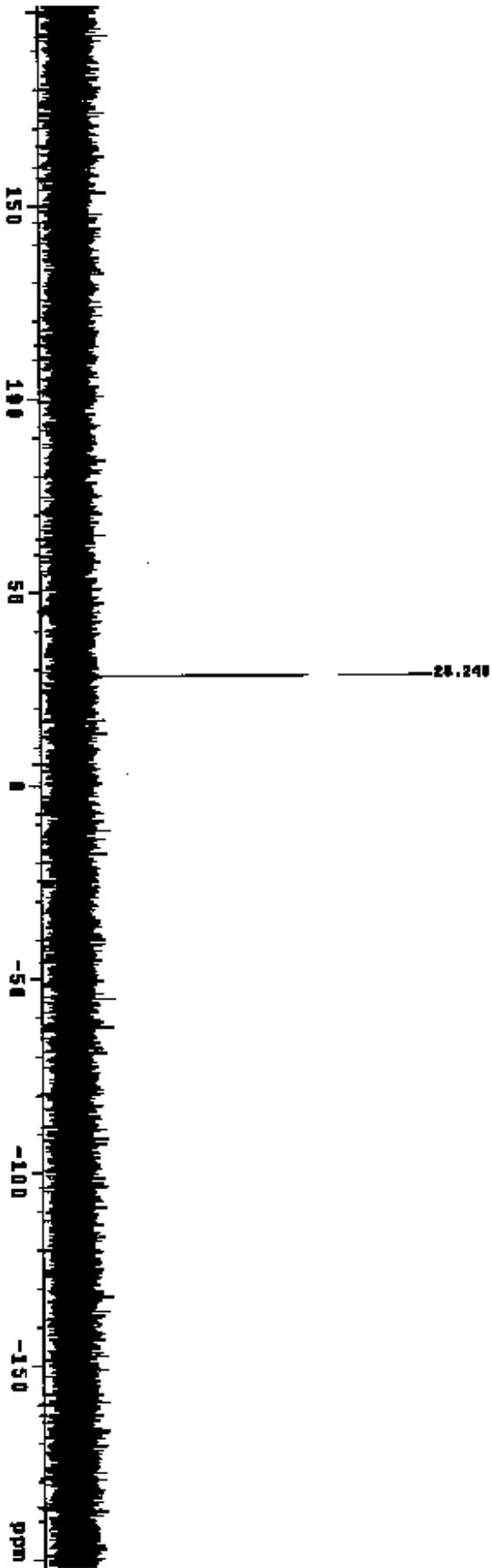
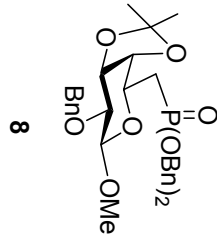
03-08-2011
Pulsed Sequence: zgpg30



8



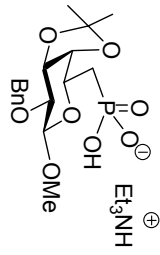
045-30-441.BATCHE66 31P run on 1mprova 400 04/04/15
Pulse sequence: vzpa1



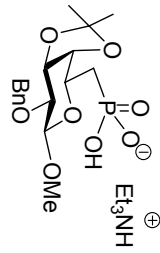
10-24-80

Pulse Sequence: zgpg30
 Solvent: d2o
 Ambient Temperature
 Director: njw
 INOVA-400 13C/1Houbt. Local Admin/1/4

Ratio: d1w/1.000 sec
 Pulse: 48.0 degrees
 Acq: 1.000 2.000 sec
 Width: 6300.0 Hz
 4 repeats/1.000 sec
 OBSERVE: H1, 393.758178 MHz
 SMTA PROCESSING
 1.000 80000
 1.000 80000
 Total Time: 9 min., 38 sec

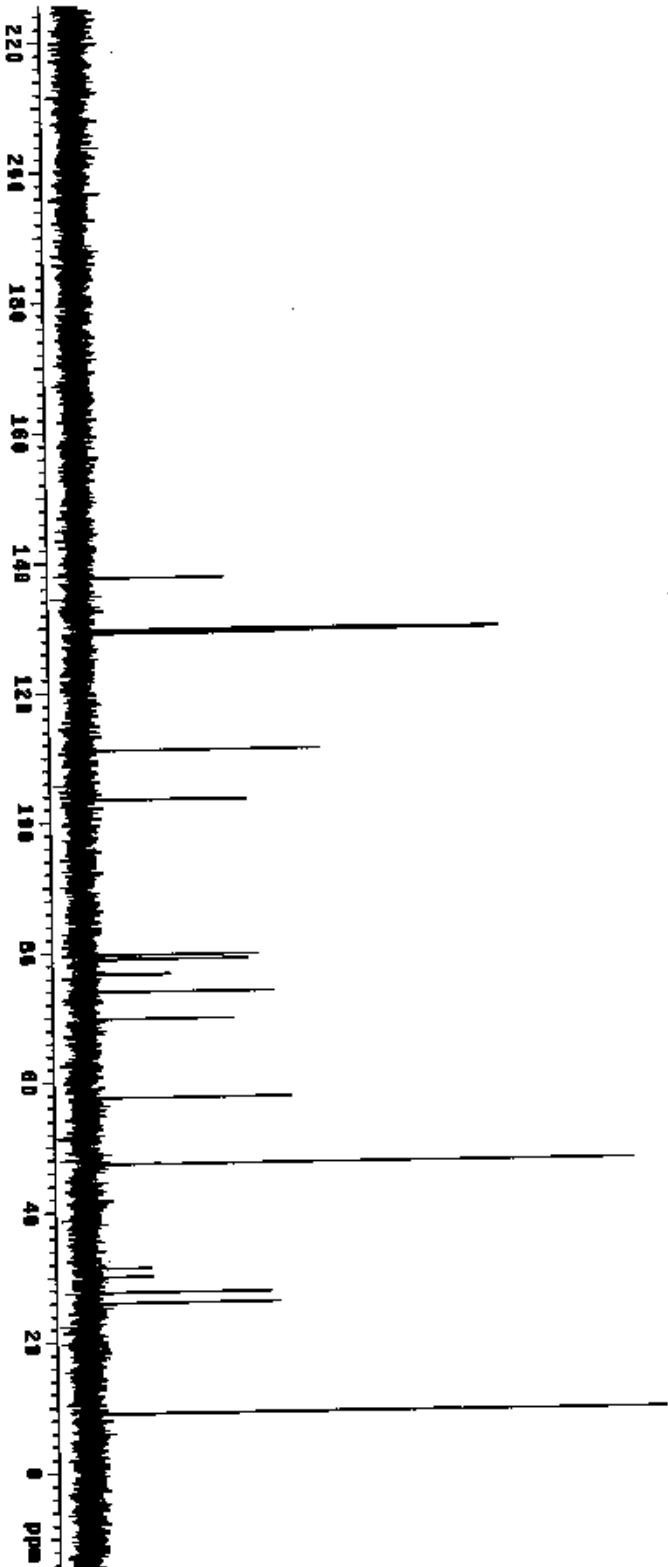


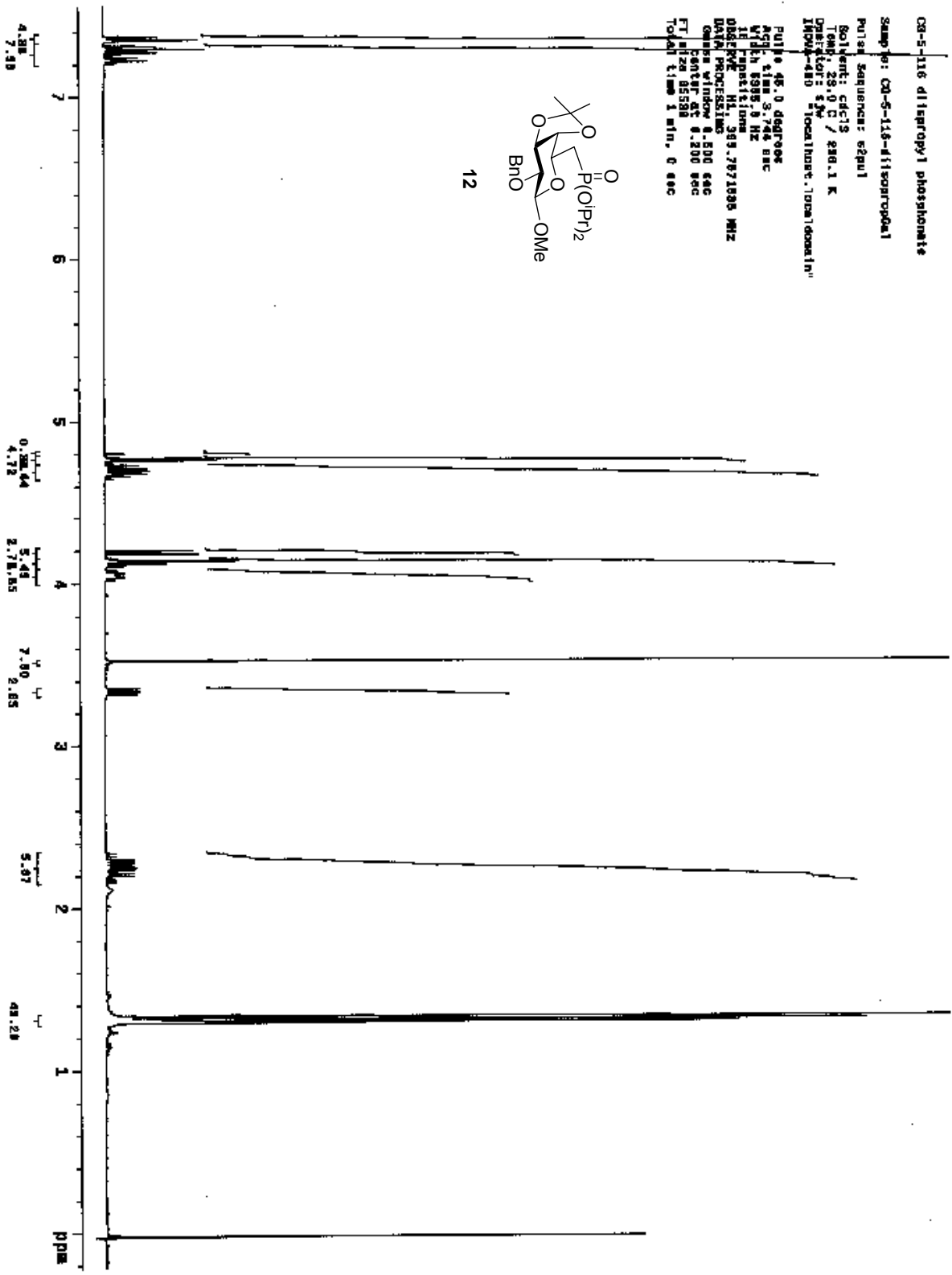
³¹P NMR
 Sample: ~~1000000~~ [4-~~2~~]-~~6~~
 Pulse sequence: zgpg30
 Solvent: d₂O
 Temp: 20.0 C / 293.1 K
 Operator: vlv
 INOVA-400 10cmhnut.localidmain1
 Relax. delay: 1.200 sec
 Pulse: zgpg30
 Acq. time: 1.288 sec
 Width: 2415.8 Hz
 178 repetitions
 OBSERVE: 31P 100.625000 MHz
 DECOUPLE: 13C 101.250000 MHz
 CHANNEL: 31P
 Copied from:
 Contained by:
 DATE_ PROCESSED:
 File browser: 1.0 Hz
 FT size: 80836
 Total time in hr.: 42 min, 00 sec



11

INDEX	FREQUENCY	PPM	HEIGHT
1	12891.280	127.684	29.5
2	12087.334	118.408	67.5
3	12080.863	118.329	65.3
4	12074.284	118.473	61.7
5	11189.038	111.113	39.8
6	10369.630	103.383	26.0
7	8669.182	74.818	26.2
8	7331.749	73.386	26.5
9	7718.717	76.653	14.8
10	7618.256	75.569	13.4
11	7420.698	73.805	20.7
12	7400.810	73.292	24.3
13	5789.317	57.695	39.3
14	5358.112	53.238	-4.7
15	4794.688	47.648	68.2
16	3149.327	31.261	10.4
17	2814.328	28.050	10.4
18	2772.182	27.578	20.9
19	2607.810	25.844	31.2
20	888.401	8.928	89.0

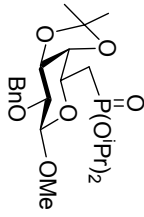




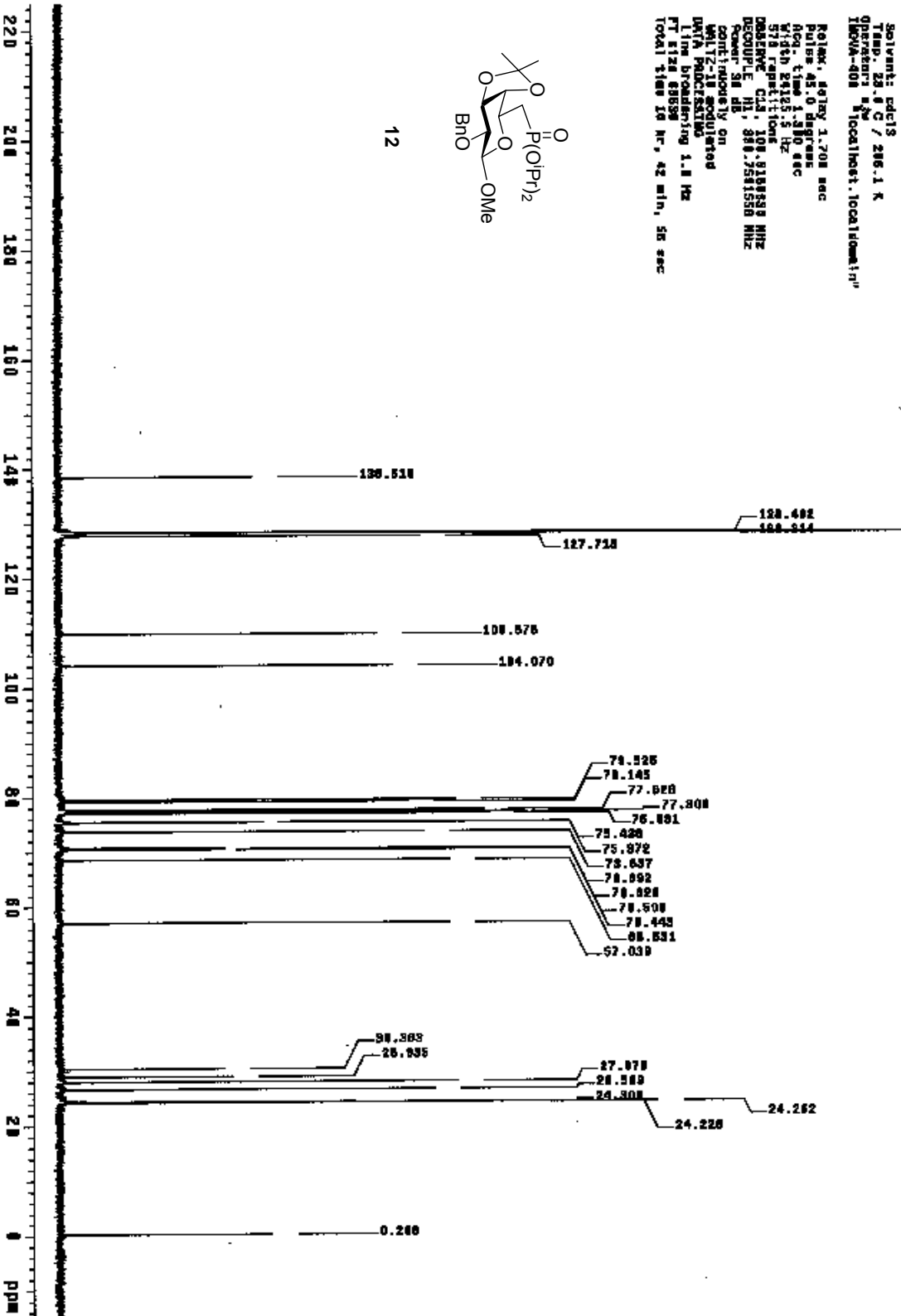
Std proton

Sample: G3-5-115-111501r000a1
 Pulse Sequence: sgpu1
 Solvent: cdcl3
 Temp: 25.0 C / 286.1 K
 Operator: m...
 INOVA-400 Localhost: localhost:317

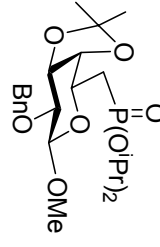
Relax: delay 1.700 sec
 Pulse: 45.0 degree
 Acq. time: 1.380 sec
 Width: 2312.5 Hz
 S/F: 100.628159 MHz
 RESERVE: C13: 100.628159 MHz
 DECOUPLE: H1: 500.136159 MHz
 Continuously on
 WALTZ-16 scheduled
 DATA PROCESSING
 Line broadening: 1.0 Hz
 FT: 128 65536
 Total time: 16 hr, 42 min, 56 sec



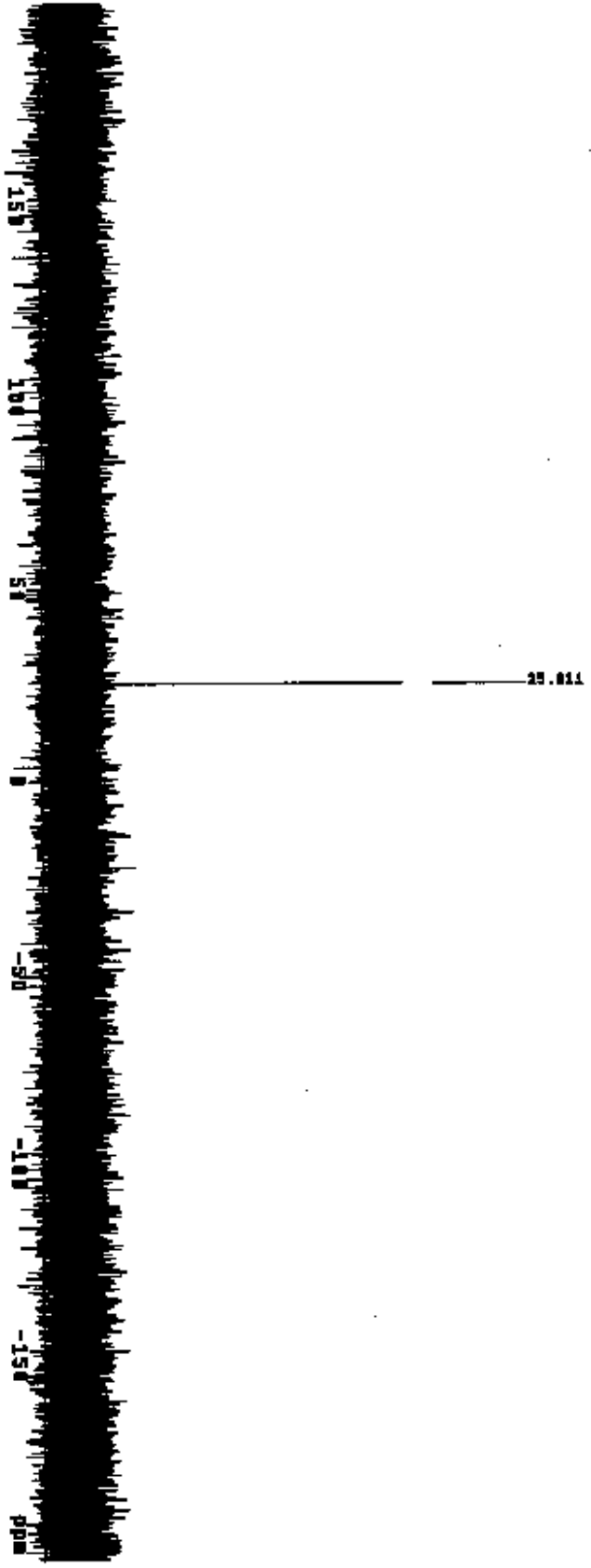
12



05-16-10_JRM_18-17_28-28 run on 100ms 400 05/04/05
Pulse sequence: zgpg1

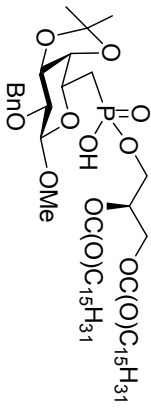


12

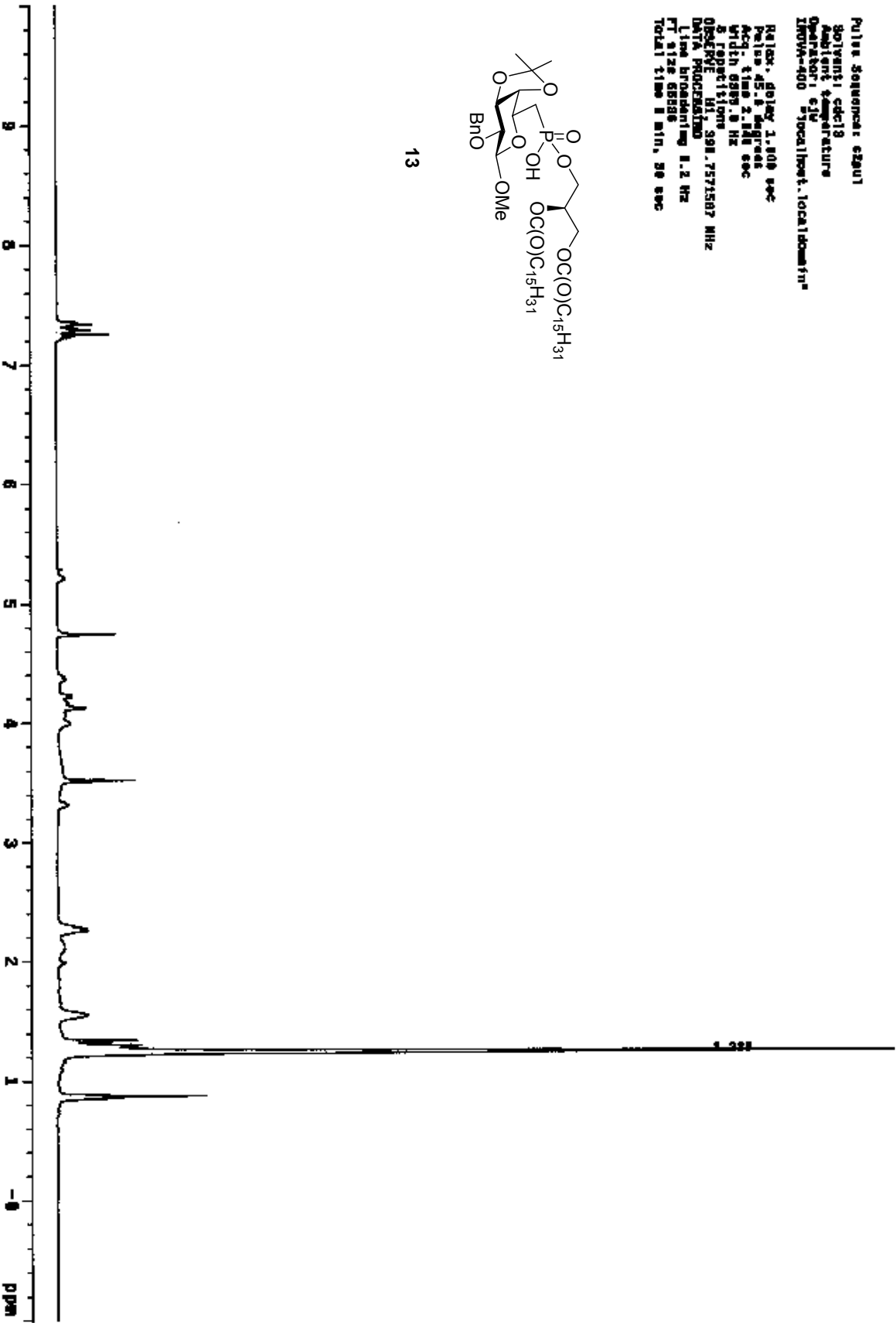


10-29-64 F14-26
 51d proton

Pulse Sequence: ezau1
 Solvent: cdcl3
 Acquire Temperature
 Operator: sju
 INOVA-400 500MHzpt. localdominr
 Relax: delay 1.800 sec
 Pulse: 45.8 degrees
 Acq. time 2.888 sec
 Width 6989.0 Hz
 S. Repetitions
 OBSERVE H1, 398.7571587 MHz
 DATA PROCESSING
 Line broadening 1.2 Hz
 FT size 65536
 Total time 8 min, 58 sec



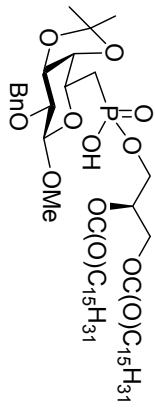
13



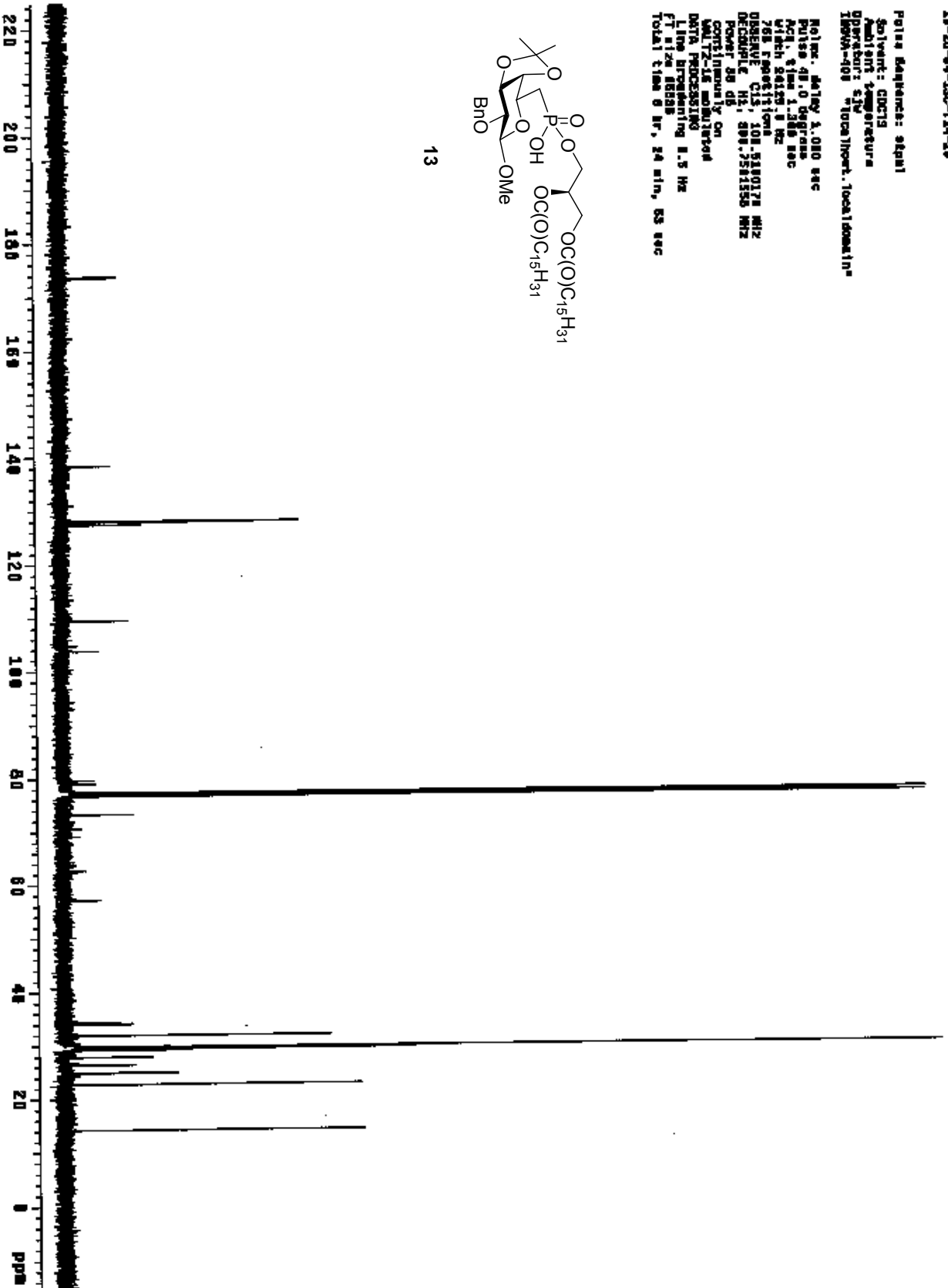
13-20-04-130-124-28

Polymer Name: 369M1
 Solvent: CDCl3
 Ambient Temperature
 Operator: sly
 ID: 130-124-28
 Total Time: 100.1500 min

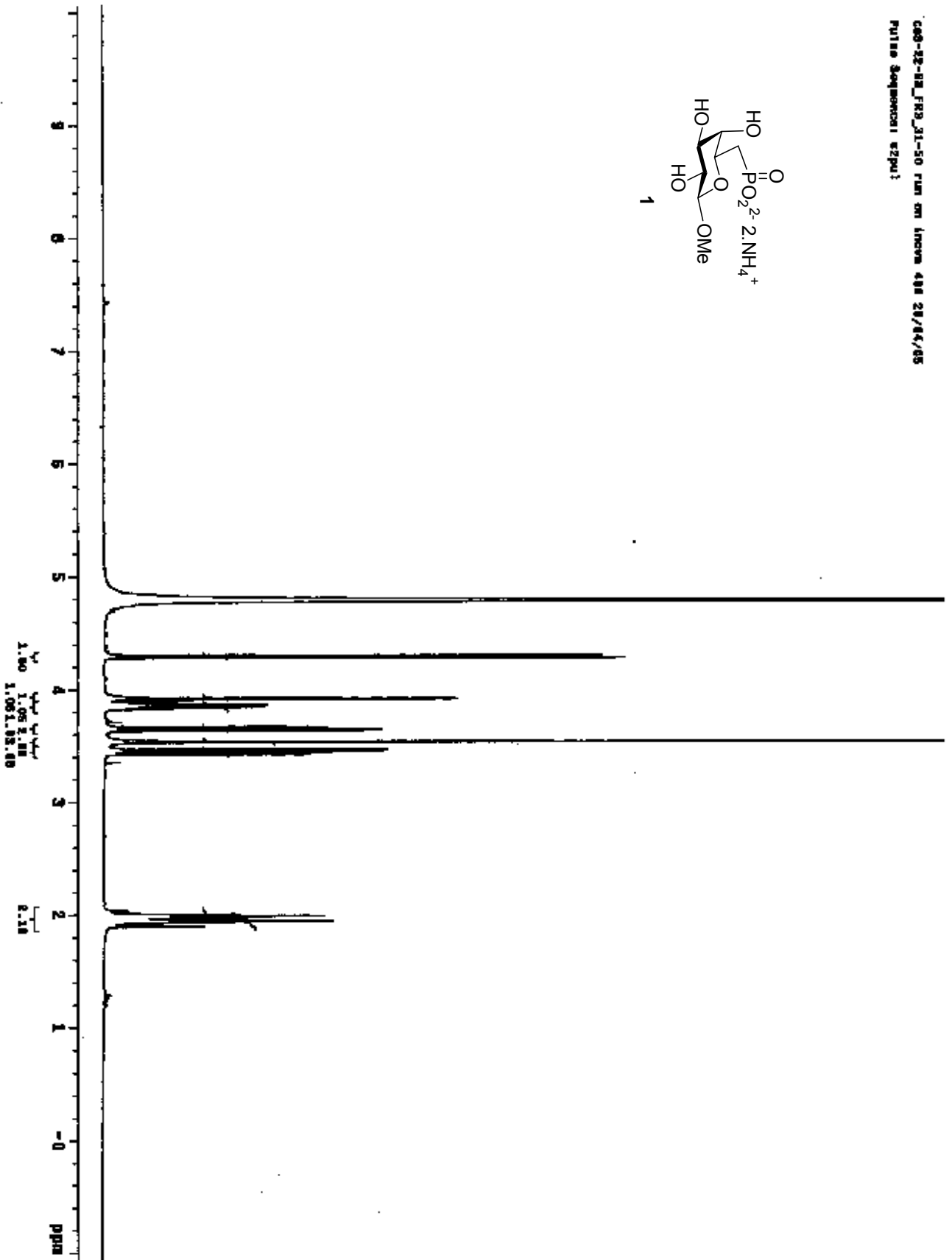
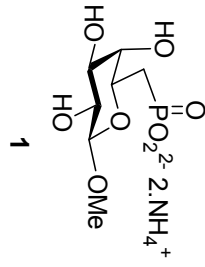
Pulse: 4.100 sec
 Pulse Pr: 0.000000 sec
 Acq Time: 1.300000 sec
 F1: 101.325000 MHz
 F2: 101.325000 MHz
 DECADE: C13, 100, 53.80178 MHz
 DECADE: H1, 400, 253.15500 MHz
 Power: 20 dB
 Continuum: On
 SALT: 16 mol/L
 DATA PROCESSING
 Line Broadening: 0.5 Hz
 FT: 1638555
 Total Time: 6 hr, 24 min, 55 sec



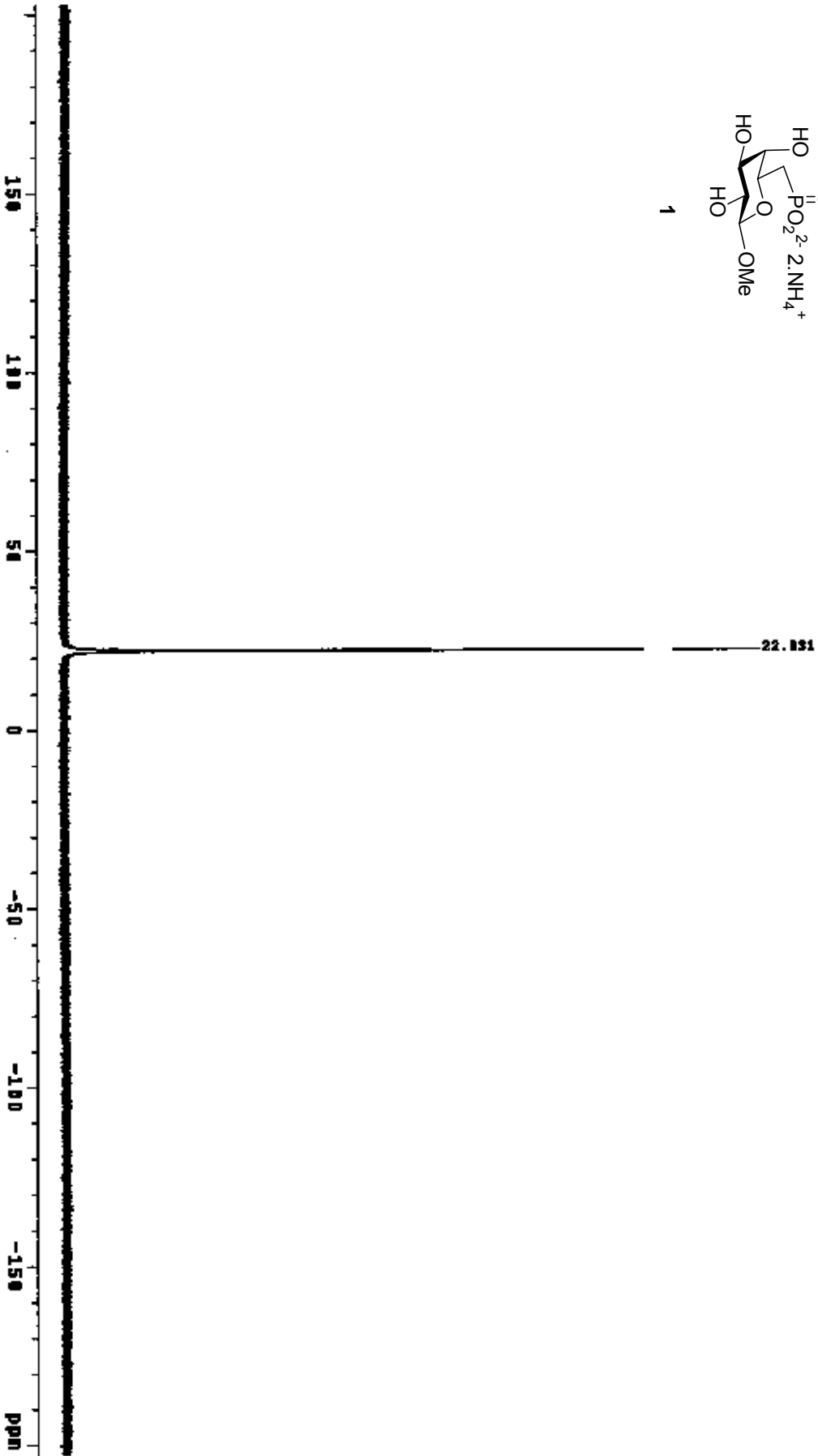
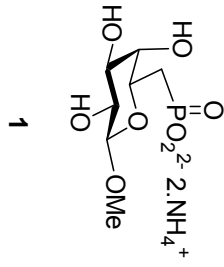
13



C80-12-18_FR3_31-50 run on linux 401 28/04/85
Pulse Sequence: vzpu1



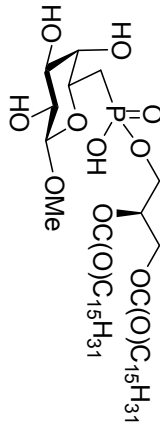
040-23-00_F02_31-01 run on Inova 400 25/04/05
Pulse Sequence: zgpg30



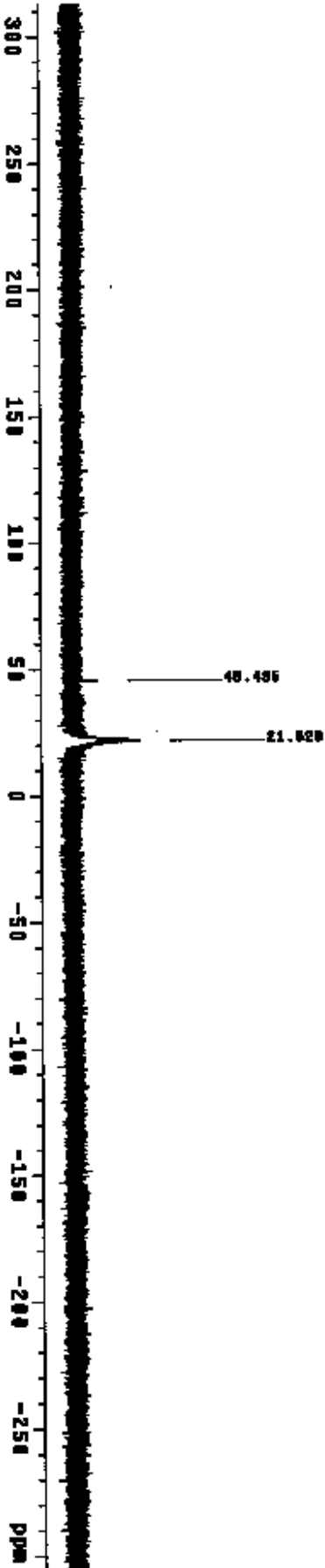
18-28-84

Sample: 18-28-84
 Pulse Sequence: zgpg30
 300K
 Ambient Temperature
 Operator: sju
 INOVA-413 "10001M05C.K01210001"

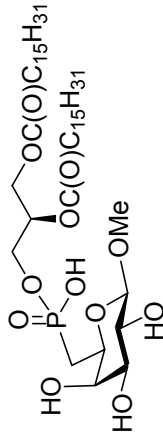
Relax. delay: 1.508 sec
 Pulse P. B deq: 90
 Acq. time: 8.810 sec
 Width: 180.0 Hz
 256 repetitions
 CHANNEL: P31, 181.4247484 MHz
 DECUPLE: H1, 598.7601832 MHz
 Power: 28 dB
 Continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening: 1.0 Hz
 FT size: 282364
 Total time: 1 hr, 16 min, 57 sec



2



SJW-2006-03-16-05 (CG-01) full MS (M+Na)⁺



2

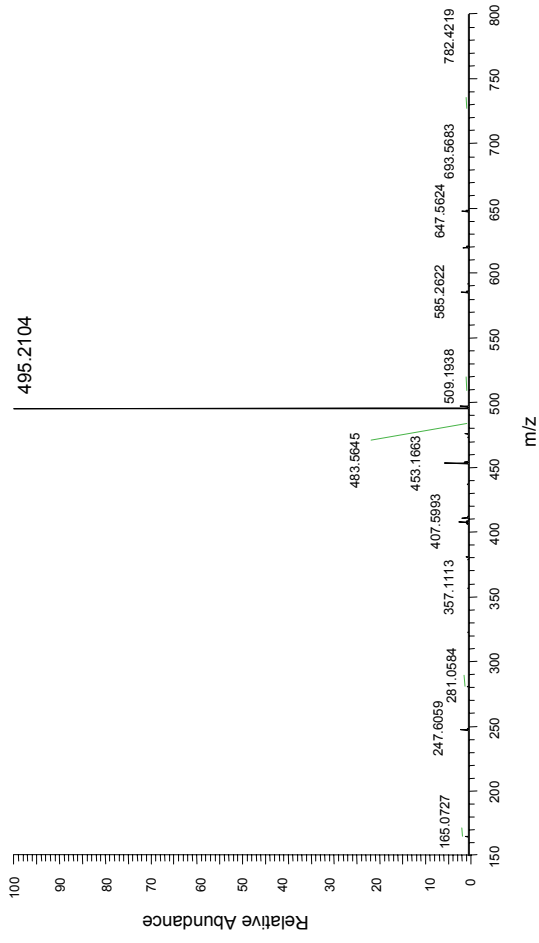
C:\Xcalibur\...SJW-2006-03-16-05_CG-01

3/16/2006 3:25:18 PM

Carlie Gammon (CG-01) 0.01mg/mL

SJW-2006-03-16-05_CG-01 # 1:30 RT: 0.00-0.71 AV:30 NL: 1.54E7

T: FTMS + p ESI Full ms [190.00-800.00]



C:\Xcalibur\...SJW-2006-03-16-06_CG-01

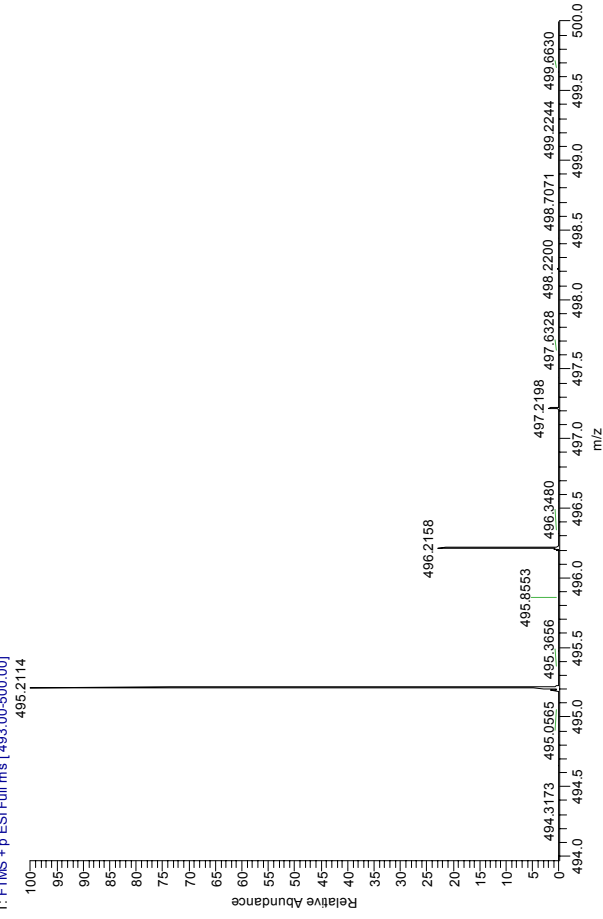
3/16/2006 3:26:27 PM

Carlie Gammon (CG-01) 0.01mg/mL

FTMS [M+Na]⁺

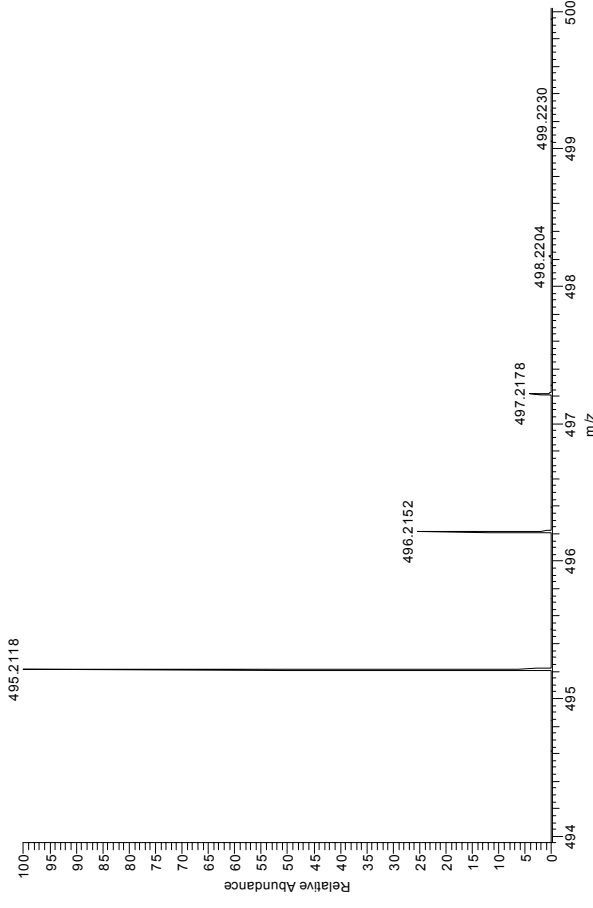
SJW-2006-03-16-06_CG-01 #1:30 RT: 0.00-0.57 AV:30 NL: 1.20E7

T: FTMS + p ESI Full ms [493.00-500.00]

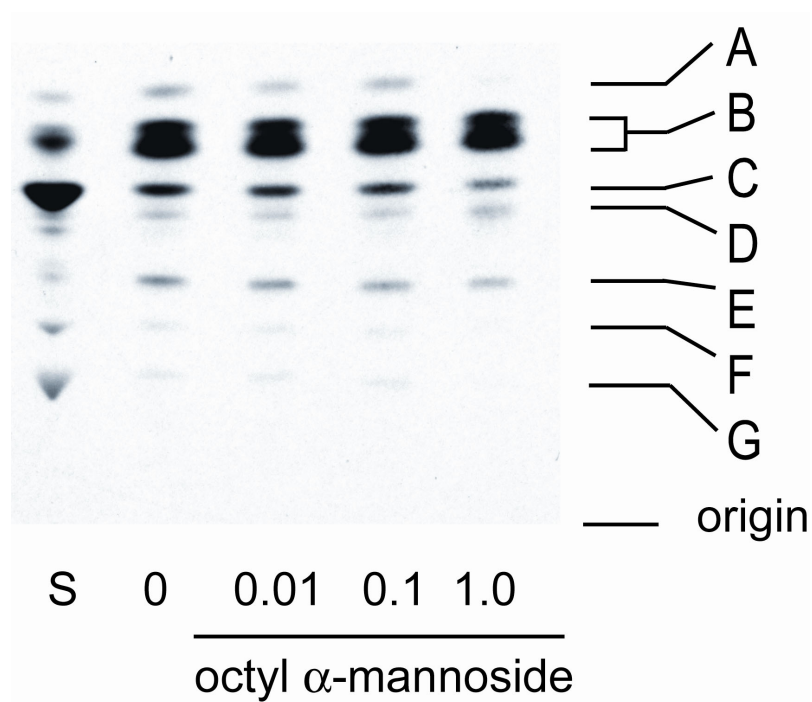


Experimental peaks

C23H37O8PNa: C23 H37 O8 P 1 Na 1 p(gss, s(p:8)) Chrg 1R: 84000 Res.Pwr. @FWHM



Theoretical peaks



Detergent control experiment: HPTLC analysis of octyl α -D-mannoside. *M. smegmatis* cell lysates were incubated with GDP- ^{3}H mannose in the absence (0) or presence of different concentrations of octyl α -D-mannoside (concentrations in mM). S = authentic PIM standards derived from *in vivo* ^{3}H mannose labelling; A = Ac₂PIM1; B = PPM, Ac₂PIM2, AcPIM1; C = AcPIM2; D = PIM1; E = PIM2; F = AcPIM5, AcPIM5'; G = AcPIM6. The abbreviations used are: Ac_xPIM_y, PIM species with *x* (1 or 2) fatty acyl chains, linked to either the core α -1,2-linked Man or the *myo*-inositol head group, and *y* Man residues; PPM, polyprenol (C₃₅/C₅₀) phosphomannose.