

A Novel and Highly Efficient Synthetic Route to Unsymmetrical Organoselenides Using Cesium Bases

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Supporting Information

Table of Contents: ^1H NMR, ^{13}C NMR spectra and analytical data (MS) are included for all the products, selected IR spectra and GC/MS for compound 31 are enclosed.

Experimental Section

Synthesis of compound 4	S2-S3
Synthesis of compound 33	S2-S3
Synthesis of compound 35	S2-S3
Sample yield calculation	S3-S4

<u>Compound #</u>	<u>Page #</u>
4	S5-S7
6	S8-S9
8	S10-S11
10	S12-S14
13	S15-S17
15	S18-S19
17	S20-S21
19	S22-S24
21	S25-S26
23	S27-S29
25	S30-S32
29	S33-S34
31	S38-S39
33	S37-S38
35	S40-S41
1	S42-S43

Experimental Section

General Procedure for the Synthesis of Benzyl Phenyl Selenide (4): To a solution containing 4 Å molecular sieves (250 mg), cesium hydroxide monohydrate (0.106 g, 0.631 mmol, 1 equiv) and anhydrous DMF (3 mL), benzeneselenol 1 was added (0.099 g, 0.631 mmol, 1 equiv) and the dark orange suspension was vigorously stirred under a dry nitrogen atmosphere for 1 hour at room temperature. Benzyl bromide 4 (0.119 g, 0.696 mmol, 1.1 equiv) was added in one portion, at which point, the solution turned to pale yellow and was allowed to stir for 14 h. Upon consumption of the starting material (1), the mixture was then filtered and rinsed with ether (~100 mL). The resulting organic layer was washed with water (3 x 30 mL), brine (30 mL), dried over anhydrous sodium sulfate and evaporated. The resulting yellow oil was purified by flash column chromatography over silica gel using hexane to afford the product 4 as clear yellow oil (147 mg, 94%).

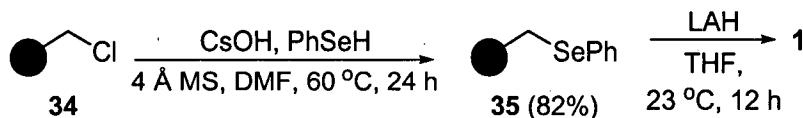
General Procedure for the Alkylation of 1-Hexyne (32) with Phenylselenyl Bromide Using CsOH.

To a stirred solution of cesium hydroxide monohydrate (100 mg, 0.595 mmol) and activated 4 Å molecular sieves (300 mg) in anhydrous DMF (13 mL) was added 1-hexyne (32) (200 mg, 2.44 mmol, 0.28 mL, 1 equiv) at ambient temperature, at which point the solution turned to pale yellow. After 1 hour, phenylselenyl bromide (630 mg, 2.67 mmol, 1.1 equiv) was added and the orange suspension was stirred for an additional 4.5 hours under a nitrogen atmosphere. The reaction mixture was then filtered to remove the molecular sieves and any other undissolved inorganic salts and washed several times with diethyl ether. The ether layer was transferred to a 125 mL separatory funnel and washed with water (3 x 30 mL), brine (30 mL), and the combined organic layers were dried over anhydrous sodium sulfate. After evaporation of the solvent, the residue was purified by flash column chromatography (hexane) to afford the desired product 33 as a colorless oil (530 mg, 92%).

Representative Experimental Procedure for the Solid-Phase Synthesis of Unsymmetrical Selenide 35.

Benzeneselenol 1 (942 mg, 6 mmol, 1 equiv) was dissolved in anhydrous *N,N*-dimethylformamide (20 mL). Cesium carbonate (1.95 g, 6 mmol, 3 equiv) and tetrabutylammonium iodide (2.21 g, 6 mmol, 3 equiv) were then added to the solution under nitrogen with vigorous stirring. The temperature of the reaction was then raised to 60 °C, after which point Merrifield's peptide resin (34) (1.00 g, 2 mmol, 1 equiv) was added, and the reaction was continually stirred at 60 °C for 24 h. The mixture was cooled to room temperature and diluted with water. The reaction was then subsequently filtered through a coarse fritted filter disc. The resin was washed successively with 30 mL aliquots of water, ethanol/water (1:1, v/v), water, tetrahydrofuran, methanol, and dichloromethane in that order and then dried in vacuo for 24 h to yield the polymer-bound phenyl selenide 35 as an off-white solid (1.2 g, 82%).

Sample Yield Calculation for Solid Phase Reaction



Preparation of Unsymmetrical Selenide bound resin 35: Benzeneselenol 1 (942 mg, 6 mmol, 1 equiv) was dissolved in anhydrous *N,N*-dimethylformamide (20 mL). Cesium carbonate (1.95 g, 6 mmol, 3 equiv) and tetrabutylammonium iodide (2.21 g, 6 mmol, 3 equiv) were then added to the solution under nitrogen with vigorous stirring. The temperature of the reaction was then raised to 60 °C, after which point Merrifield's peptide resin (34) (1 g, 2 mmol, 1 equiv) was added, and the reaction was continually stirred at 60 °C for 24 h. The mixture was cooled to room temperature, and diluted with water. The reaction was then subsequently filtered through a coarse fritted filter disc. The resin was washed successively with 30 mL aliquots of water, ethanol/water (1:1, v/v), water, tetrahydrofuran, methanol, and dichloromethane in that order and then dried in *vacuo* for 24 h to yield the polymer-bound phenyl selenide 35 as an off-white solid (1.20 g, 82%). IR (KBr pellet) 3071, 3075, 2951, 2846, 1943, 1874, 1711, 1611, 1605, 1495, 1462, 1420, 1253, 1172, 1036, 1000, 765, 700 cm^{-1}

The loading yield for the selenide resin 35 was determined by gravimetric analysis as follows. (See the Supporting Information: Hunt, J. A.; Roush, W. R. *J. Am. Chem. Soc.* 1996, 118, 9998.) The following assumptions are: (1) The change in the mass of the resin corresponds to the mass of the selenol loaded minus the mass of the Cl⁻ displaced, and (2) that the number of moles loaded equals the number of moles of Cl⁻ displaced.

Loading:

$$1.2 \text{ g of selenide resin 35} - 1.0 \text{ g of Merrifield's resin} = 0.20 \text{ g mass loaded}$$

$$0.20 \text{ g} = (157.08) \text{ (mol of selenol added)} - 35.45 \text{ g/mol (mol Cl⁻)}$$

Assuming that mol of selenol loaded = mol of Cl⁻ displaced.

$$0.20 = 157.08 (x) - 35.45 \text{ g/mol} (x)$$

$$0.20 \text{ mmol} = 121.63 (x)$$

$$1.644 \text{ mmol} = x$$

$$1.644 \text{ mmol of selenol loaded}/1.0 \text{ g of resin} = 1.644 \text{ mmol/g}$$

The yield of the reaction is calculated from the loading of the starting material (Merrifield's resin: 2 % cross linked) and the number of moles of selenol loaded; thus, the yield is 82% in this example.

Yield:

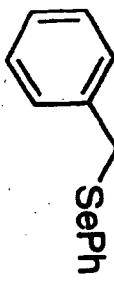
$$(1.644 \text{ mmol loaded} / 2 \text{ mmol}) \times 100 = 82 \%$$

The resulting resin-bound selenide **35** was converted back to the starting benzeselenol **1** by cleavage using lithium aluminum hydride in anhydrous THF (20 mL) after stirring at room temperature for 12 hours. The product was spectroscopically (¹H NMR) compared with the authentic sample **1** and the mass spectrum was also taken and compared to the starting material as well as the reported value. The observed value was *m/z* = 157.10 whereas the reported value is *m/z* = 157.07, respectively.

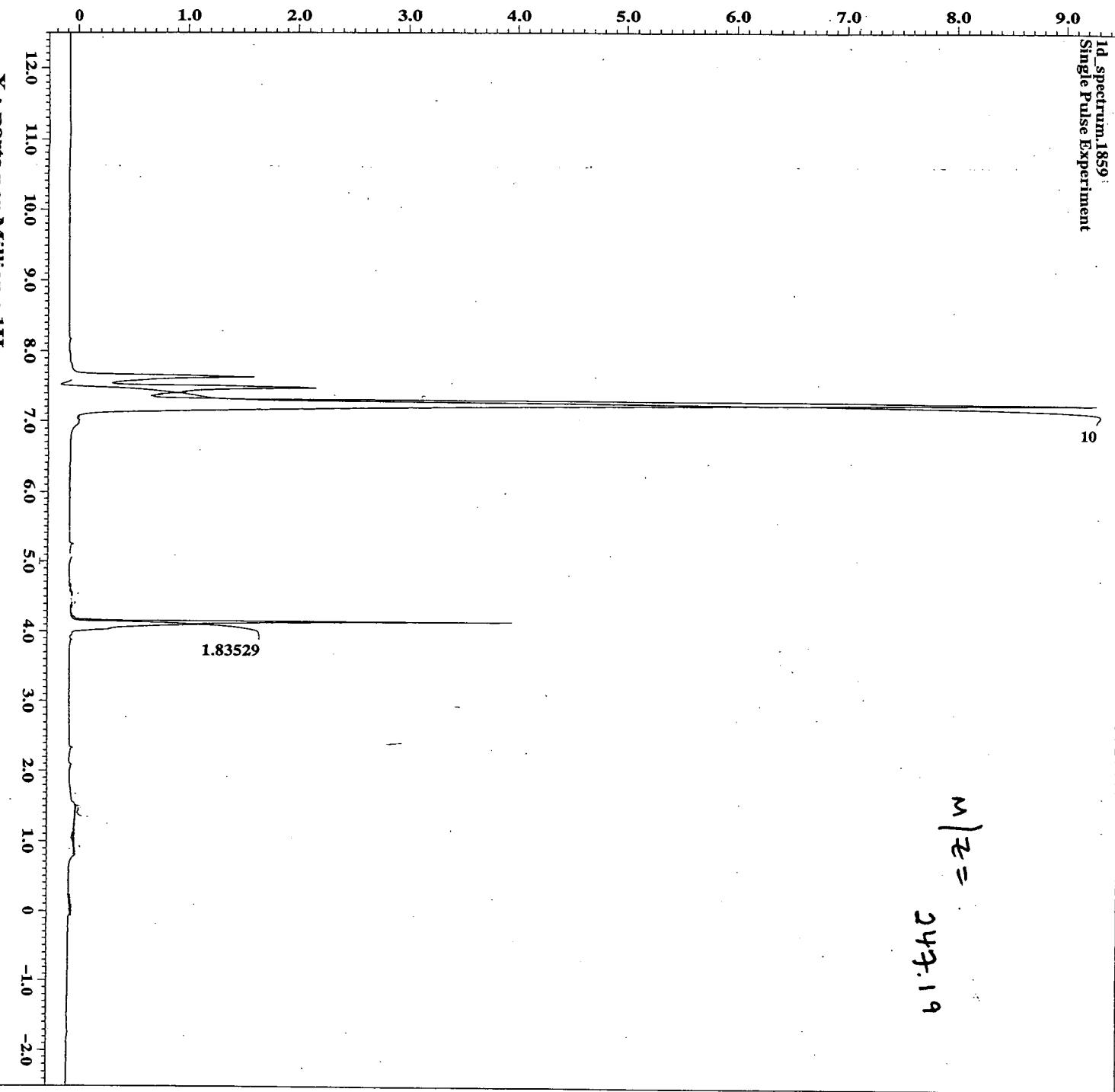
1d_spectrum.1859
Single Pulse Experiment

10

$\omega_0 =$
247.19



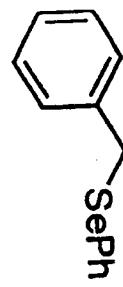
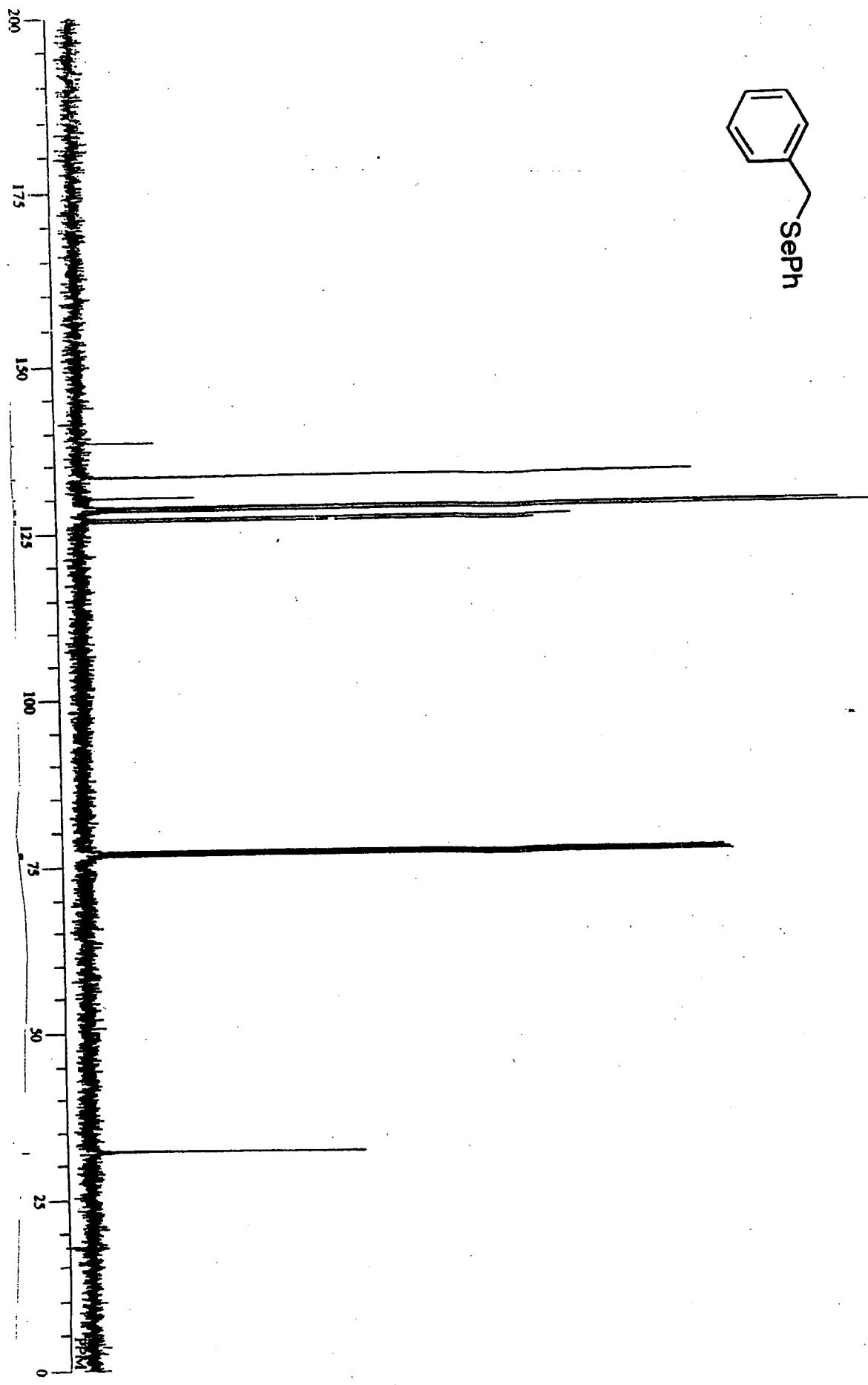
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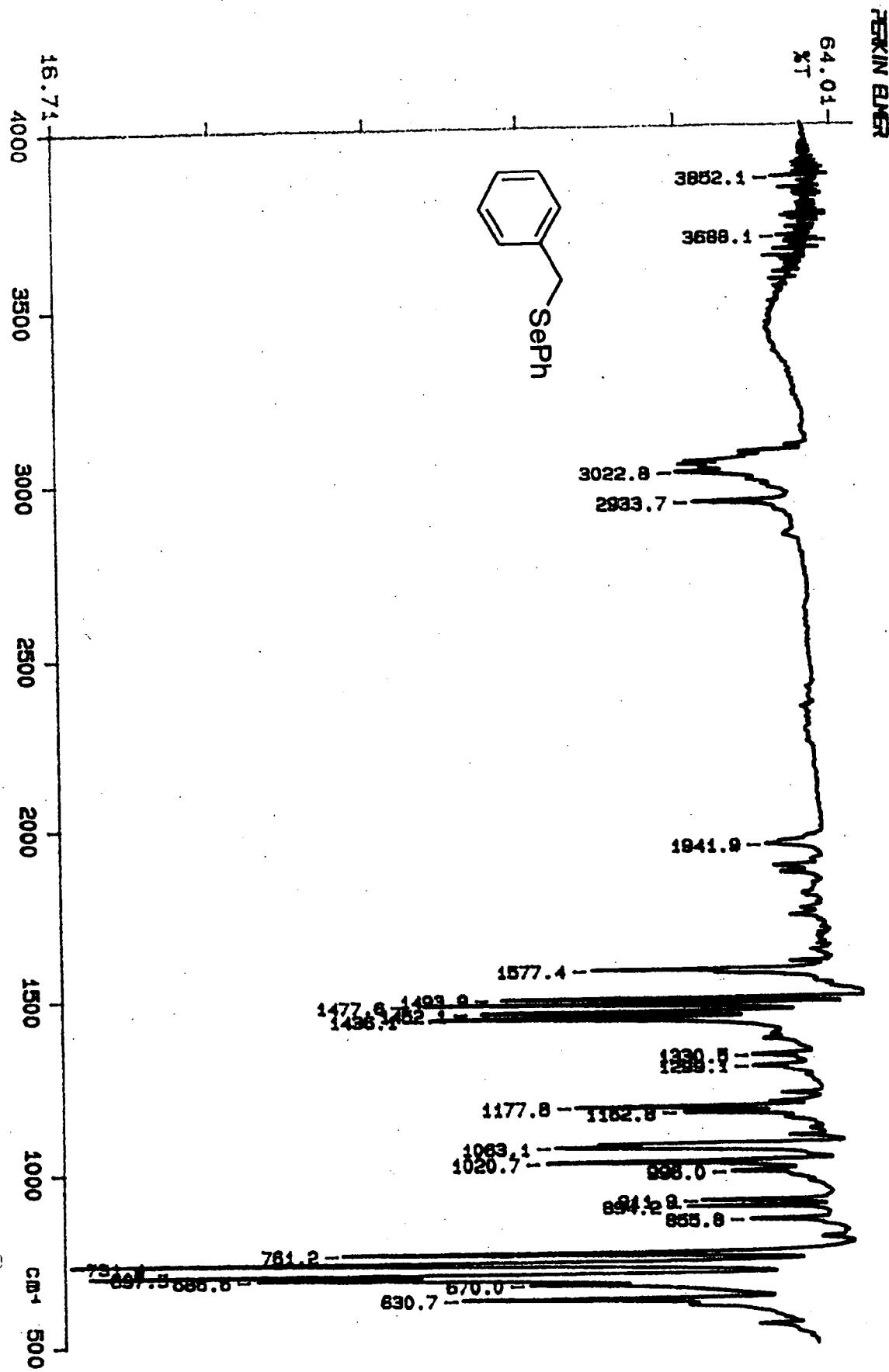


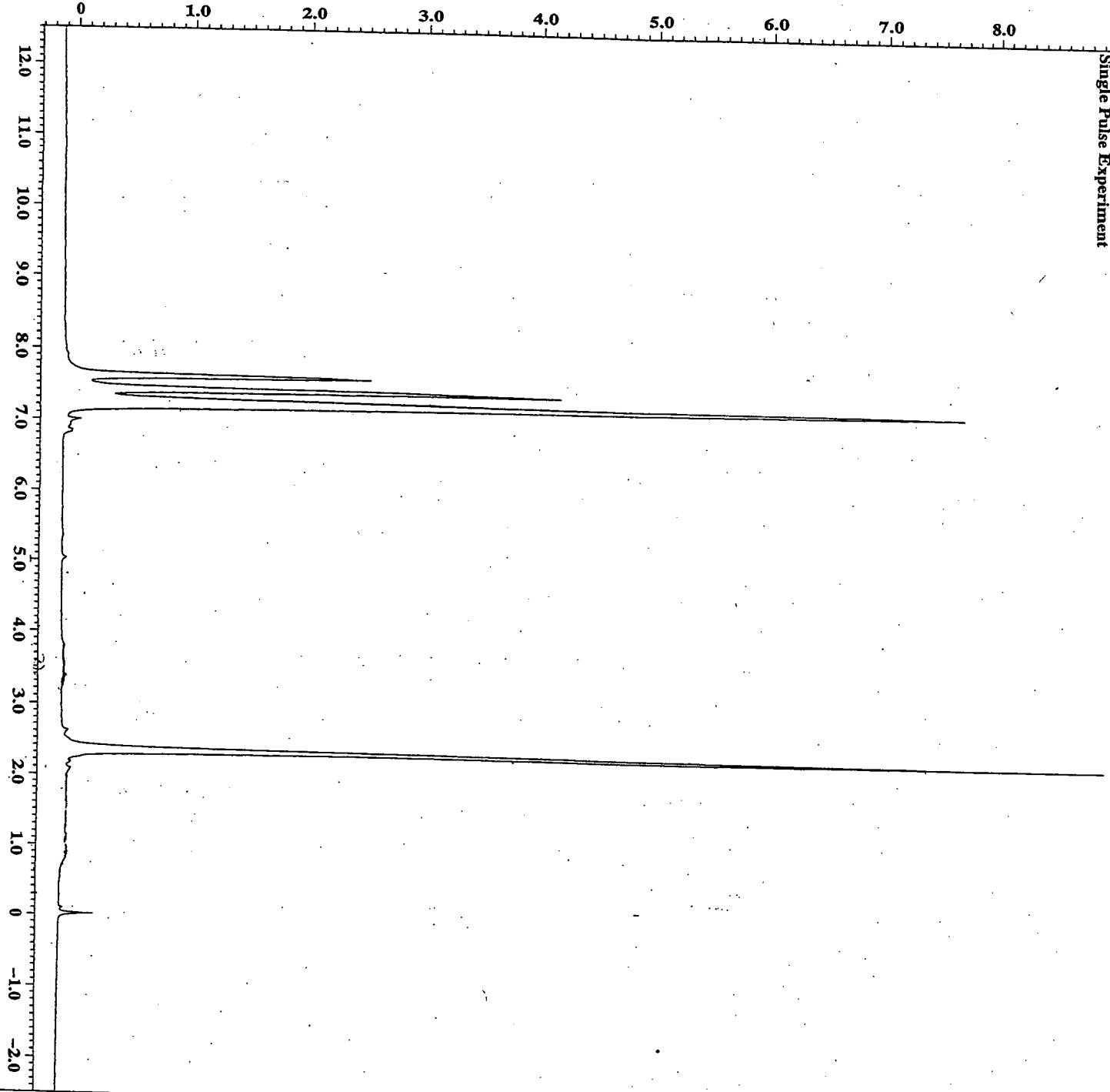
JEOL

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X_resolution	= 0.24770639[Hz]
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Filter_Factor	= 1
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Irr90	= 11[us]
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Qua90	= 10[us]
X90_h1	= 18[us]
Irr90_h1	= 18[us]
Trig0_h1	= 10[us]
Qua90_h1	= 10[us]
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Qua90_l0	= 10[us]
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Spin_Lock_Att	= 17.5[dB]
Deut_Grad_Shim_90	= 0.105[ms]
Deut_Grad_Shim_Att	= 10[dB]
Adc_card	= 16/1MHz/20
Field_Strength	= 6.343446[T]
Field_Mode	= BUTTERWORTH
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Recv_Gain	= 13
Irr_Code	= 4
Obs_PwWidth	= 1[us]

55







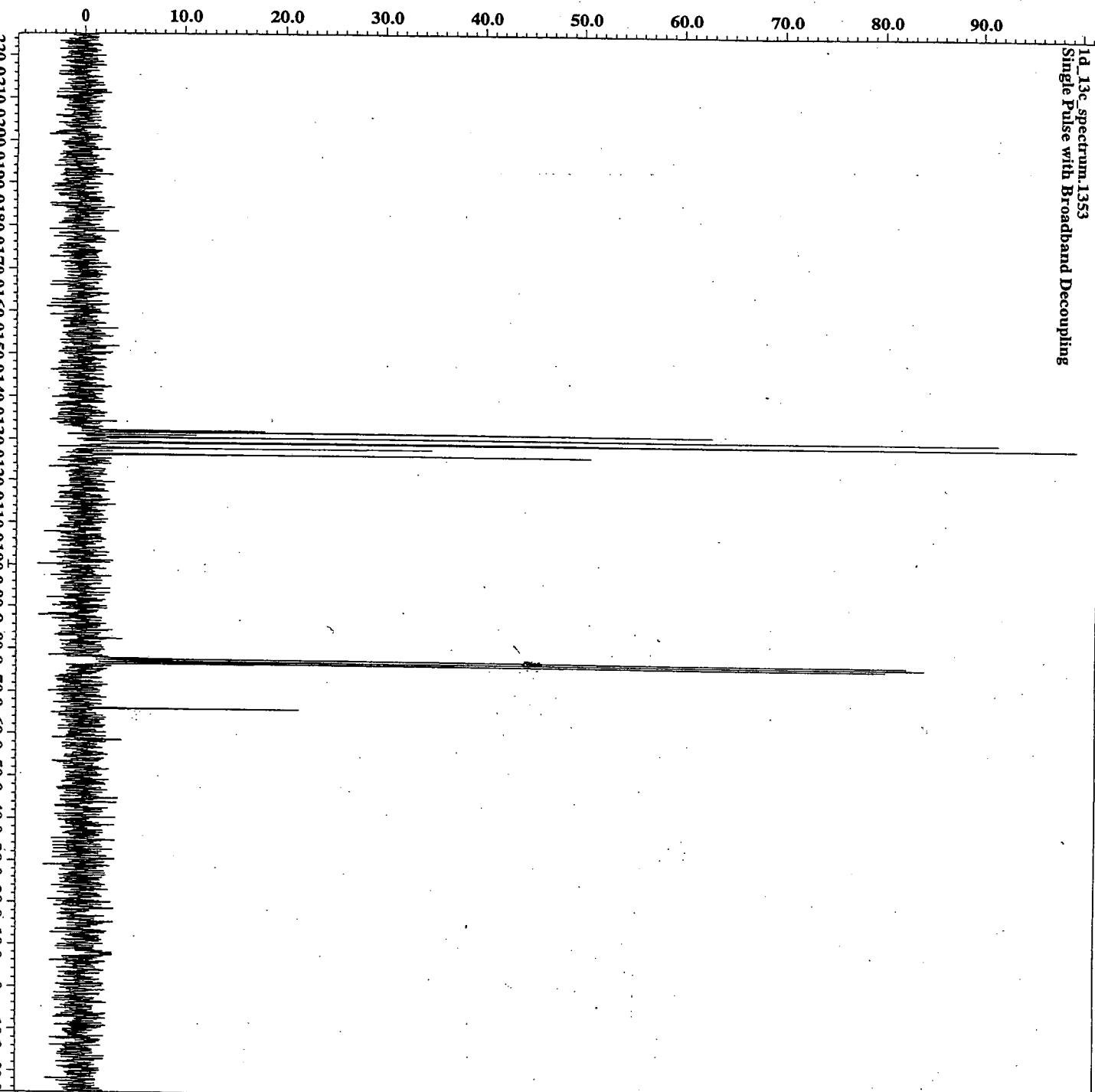
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X_prescans	0
X_domain	1H
X_Offset	51ppm
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X_resolution	0.24770639 [Hz]
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Digital_Filter	FALSE
Filter_Factor	1
Delay_of_start	1 [s]
Actual_start_time	15-DEC-2003 10:08:
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Tr190	10 [us]
Qu90	10 [us]
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rr90_hi	18 [us]
tr190_hi	10 [us]
Qu90_hi	10 [us]
x90_lo	55 [us]
rr90_lo	55 [us]
tr190_lo	10 [us]
Qu90_lo	10 [us]
spin_lock_90	0.1 [ms]
spin_lock_90	17.5 [dB]
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deut_grd_shim_attn	10 [dB]
adc_card	16.1 MHz/20
Field_strength	6.345446 [T]
Filter_mode	BUTTERWORTH
Filter_width	2.02675314 [kHz]
Reovr_Gain	14
Irr_code	4
Obs_Dwidth	1 [us]

JEOL

58

1d_13c_spectrum.1353
Single Pulse with Broadband Decoupling



X : parts per Million : 13C

JEOL

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machinephase
ppm

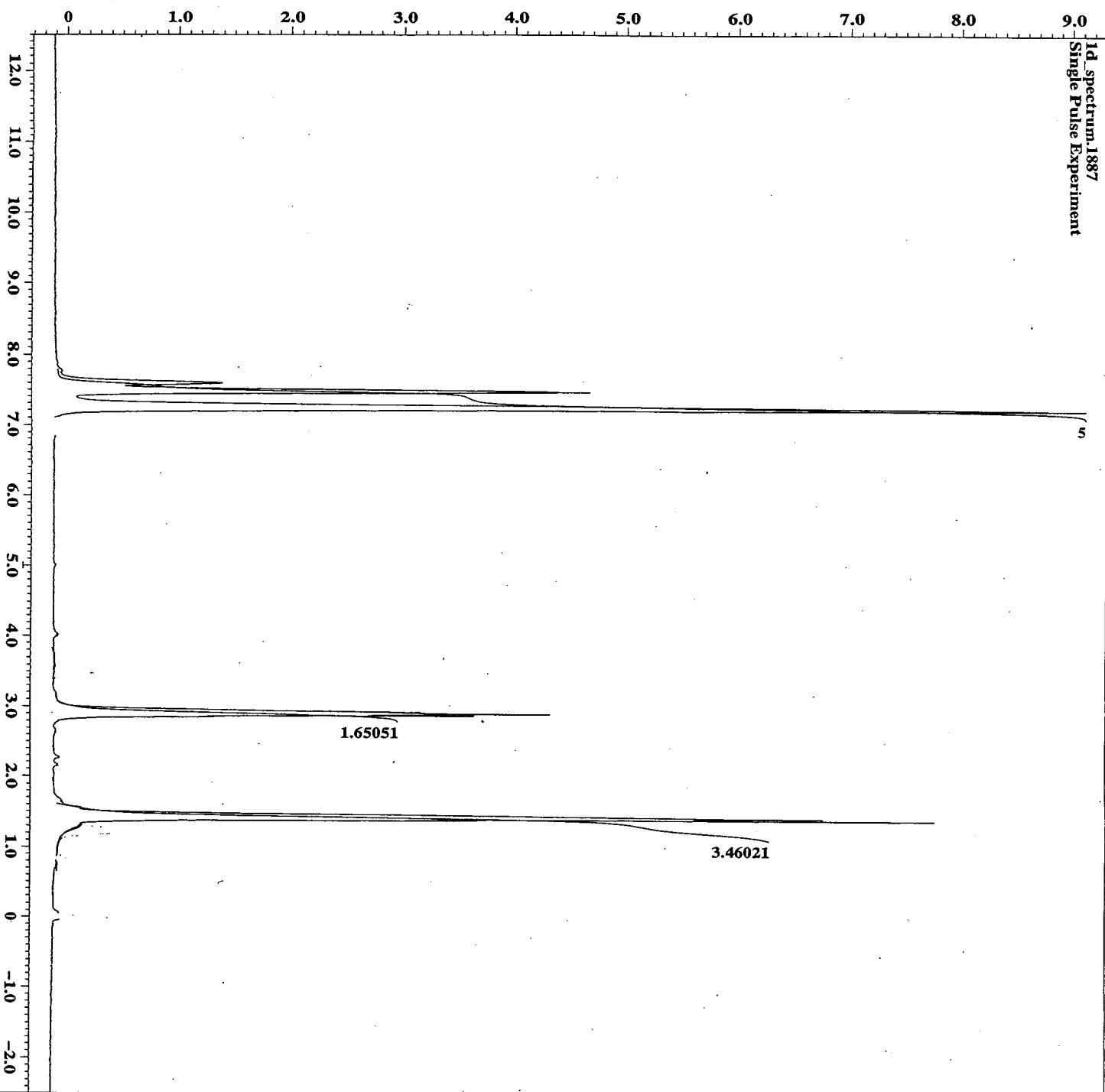


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Irr90_1o	= 55 [us]
Tr180_1o	= 10 [us]
Qu90_1o	= 10 [us]
Tr180_1o	= 18 [us]
Qu90_hi	= 10 [us]
Qu90_hi	= 10 [us]
X90_hi	= 10 [us]
Irr90_hi	= 39 [us]
Qu90_10	= 55 [us]
Qu90_10	= 10 [us]
Spin_lock_90	= 10 [ms]
Spin_lock_attn	= 17.5 [dB]
Deut_grad_shim_90	= 0.105 [ms]
Deut_grad_shim_attn	= 10 [dB]
Adc_card	= 16.1MHz/20
Field_strength	= 6.345446 [T]
Filter_width	= 8.50340136 [kHz]

SG

1d_spectrum-1887
Single Pulse Experiment



JEOL

M/Z = 185,15
se

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Author		scpd : 0.2 [Hz]	
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X-sweep	= 4.05330628 [MHz]		
X-resolution	= 0.24740639 [Hz]		
X-acq_duration	= 4.0419328 [s]		
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IRr90	= 11 [us]		
Qu90	= 10 [us]		
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IRr90_hi	= 18 [us]		
Tr90_hi	= 18 [us]		
Tr90_hi	= 10 [us]		
Qu90_hi	= 10 [us]		
X90_lo	= 55 [us]		
IRr90_lo	= 55 [us]		
Tr90_lo	= 10 [us]		
Qu90_lo	= 10 [us]		
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Spin_lock_attn	= 17.5 [dB]		
Deut_grad_shim_90	= 0.105 [ms]		
Deut_grad_shim_attn	= 10 [dB]		
Adc_card	= 16/1MHz/20		
Field_strength	= 6.34546 [T]		
Filter_mode	= BUTTERWORTH		
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Recvr_gain	= 17		
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Obs_pwidth	= 1 [us]		

1d_13c_spectrum.1368
Single Pulse with Broadband Decoupling

JEOL

dc_balance
sep : 2[Hz]
fft : 1 : TRUE
machinemphase
ppm



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Author
Sample ID
Content
Creation Date
Revision Date
Spec Site

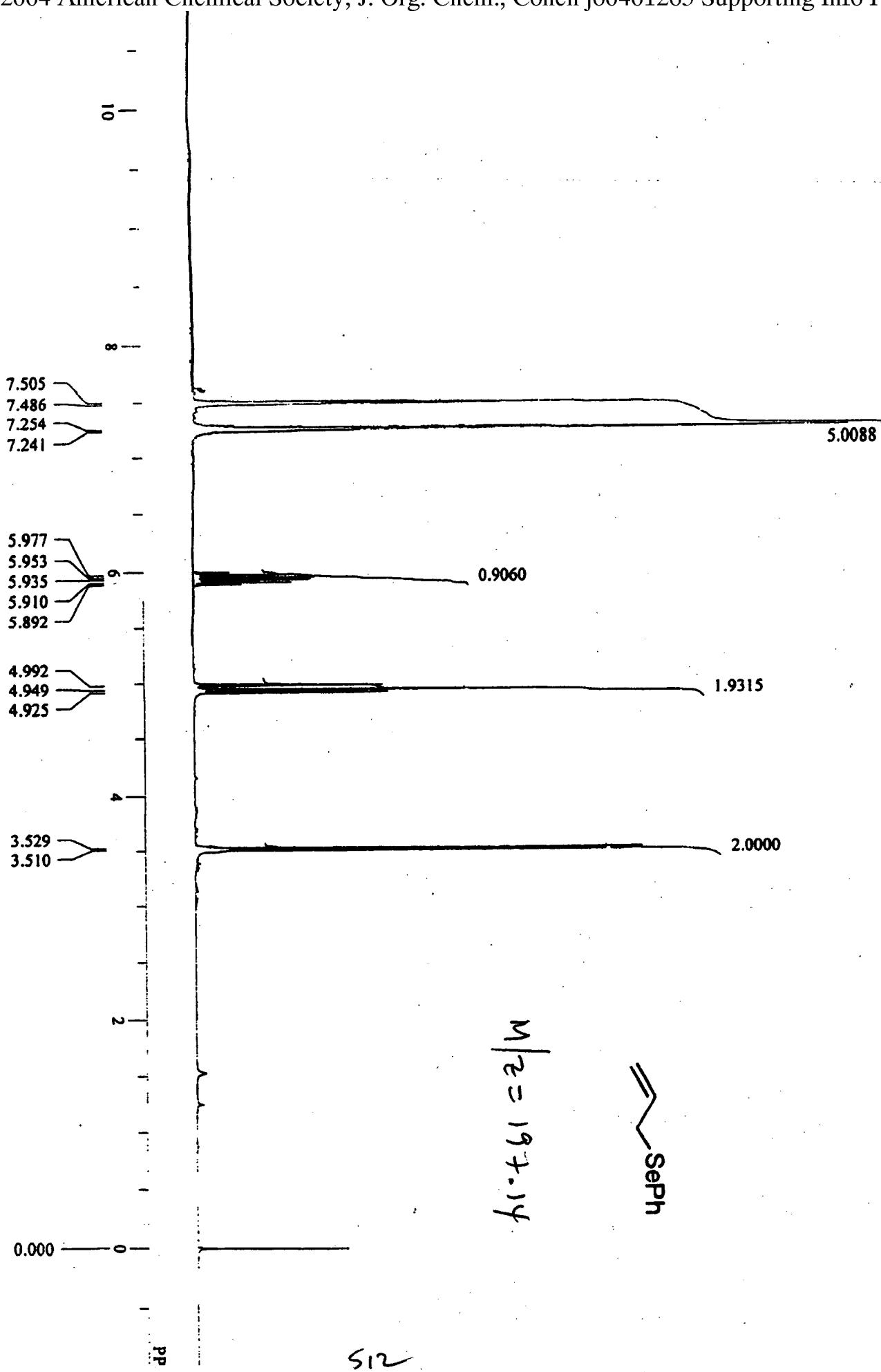
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= WU GSX-270

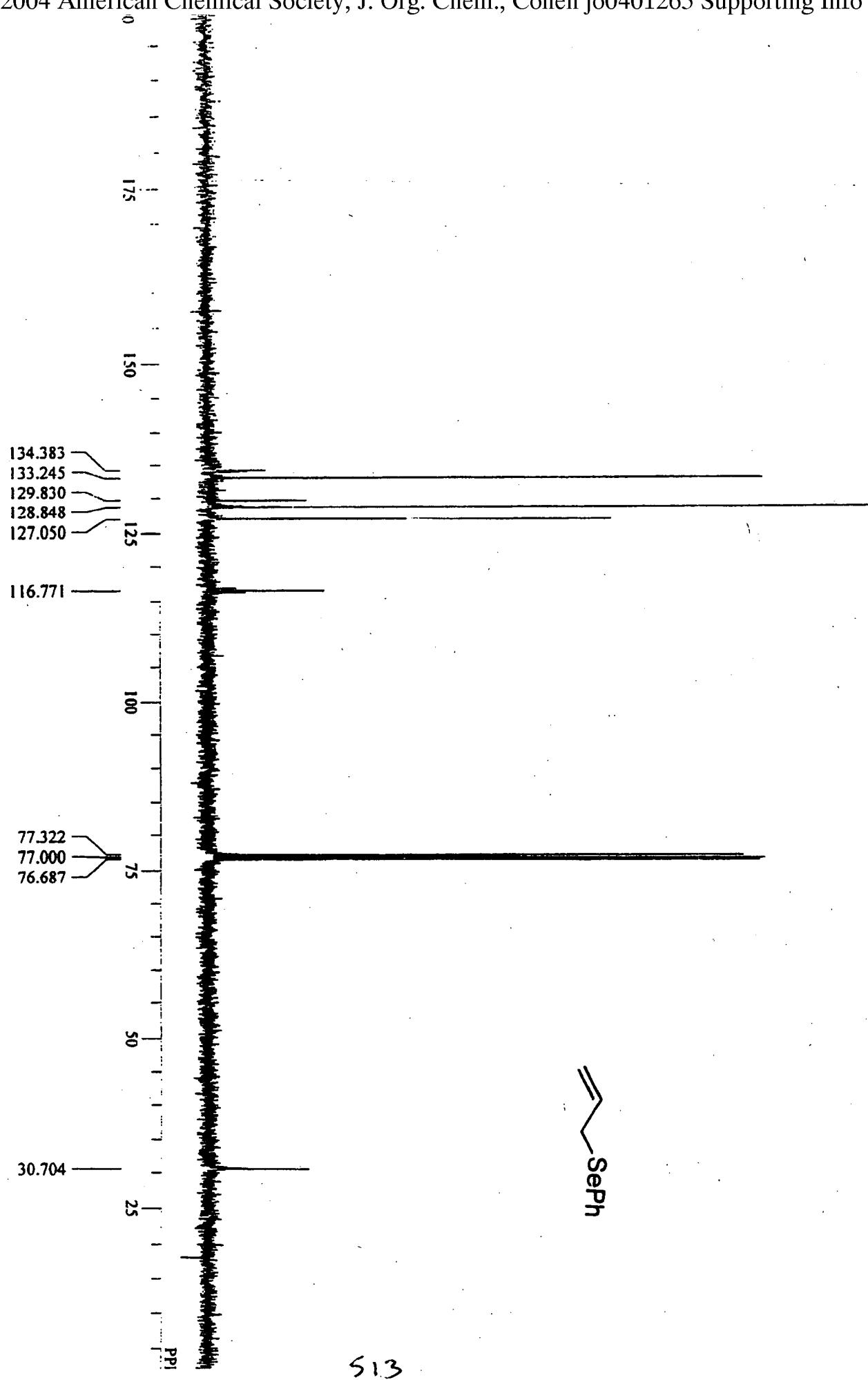
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IRR90_hi	= 18[us]
Qua90_hi	= 10[us]
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Qua90_lo	= 10[us]
IRR90_lo	= 18[us]
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Spin_lock_atten	= 17.5[dB]
Deut_grad_shim_90	= 0.105[ms]
Deut_grad_shim_atten	= 10[dB]
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X : parts per Million : 13C

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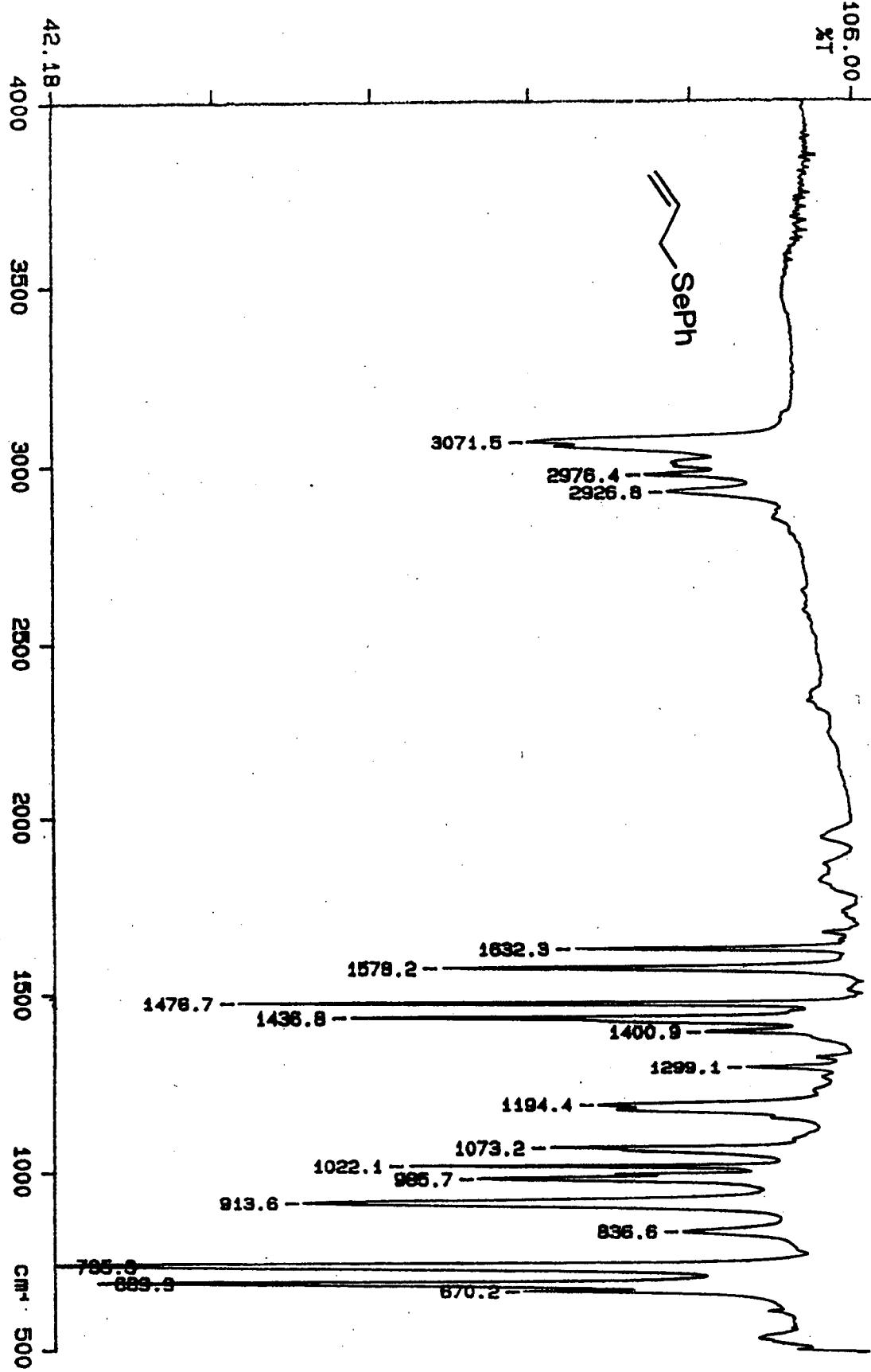
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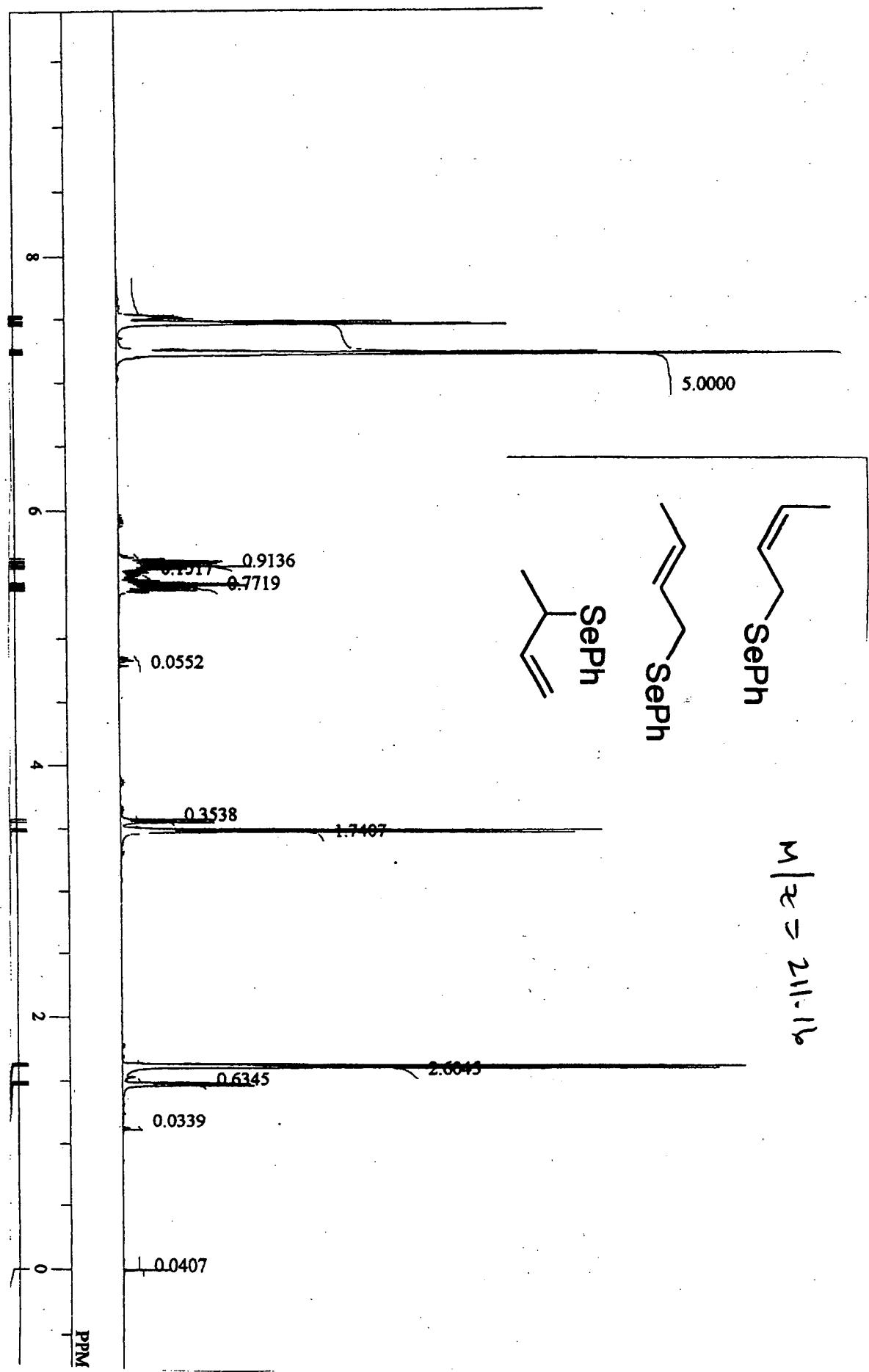




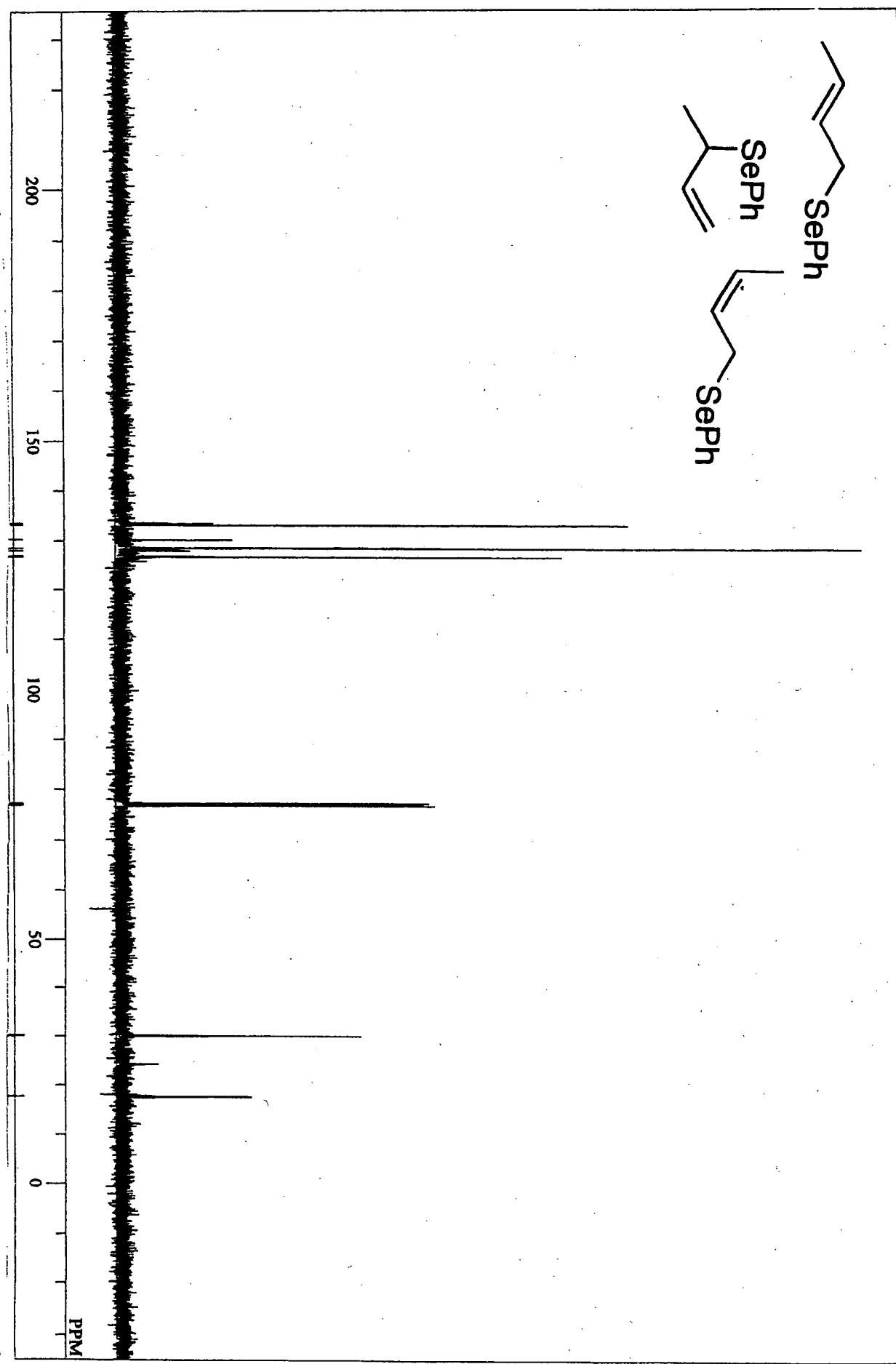
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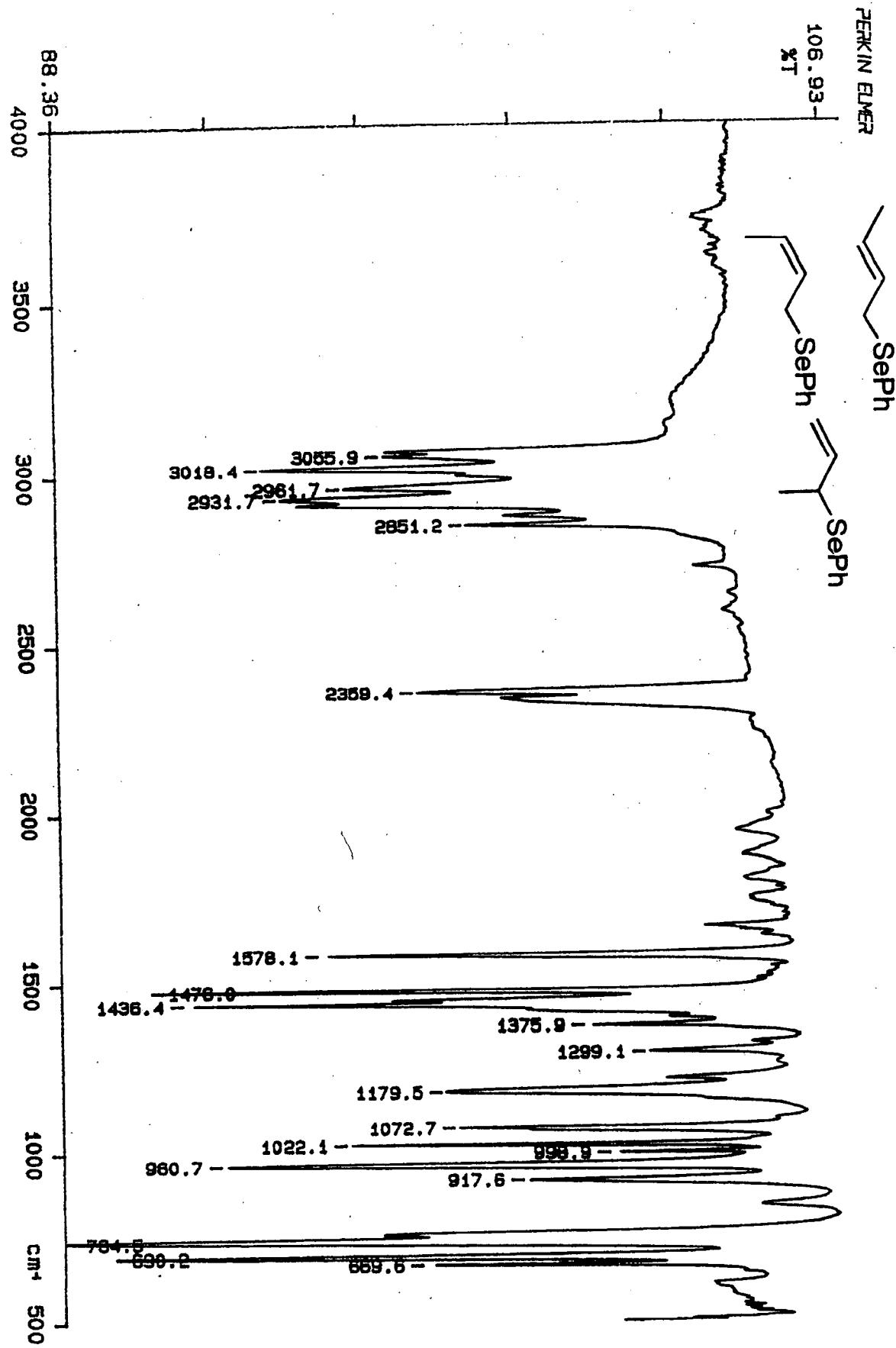
PERKIN ELMER
XT



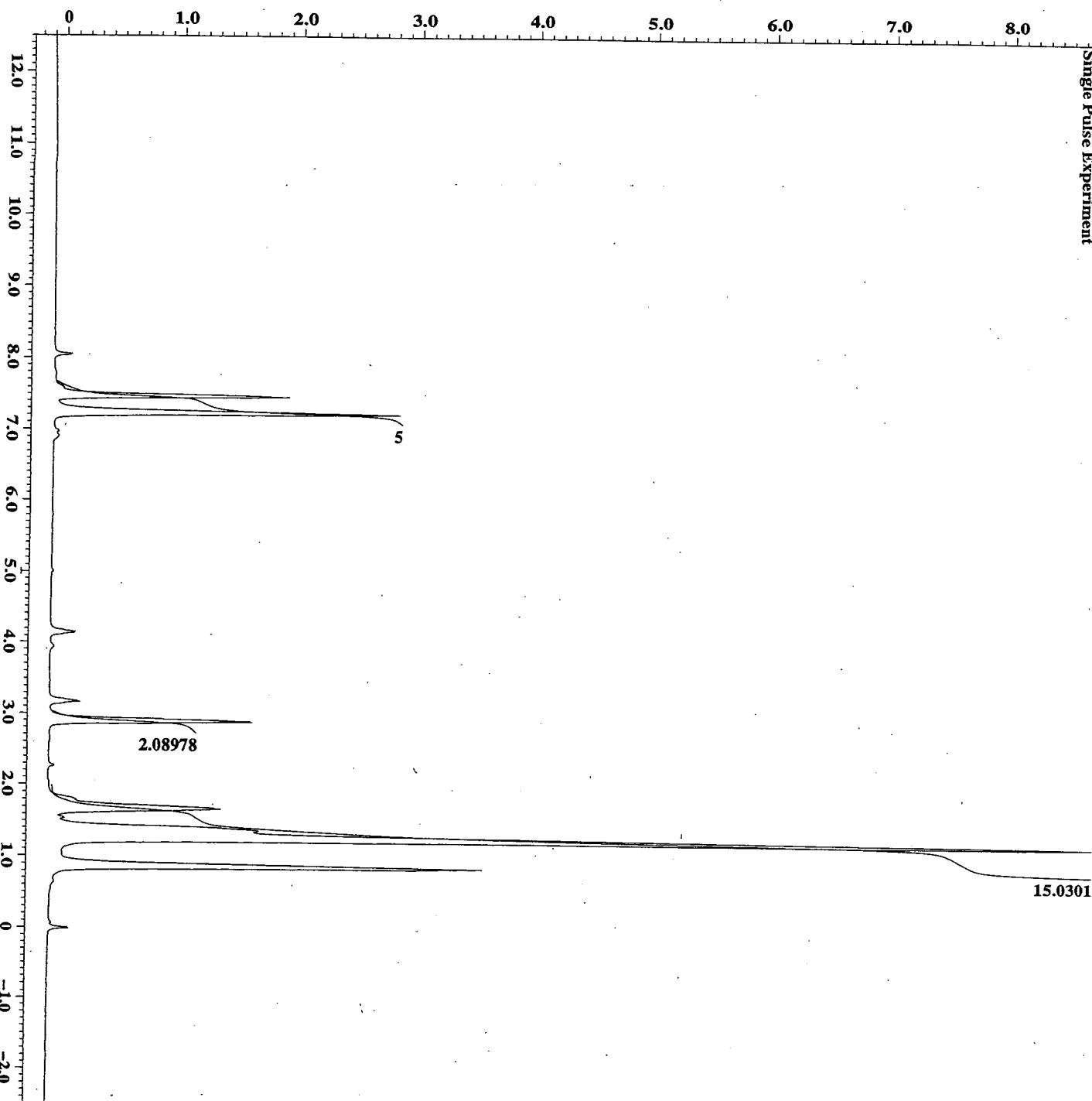


SIS





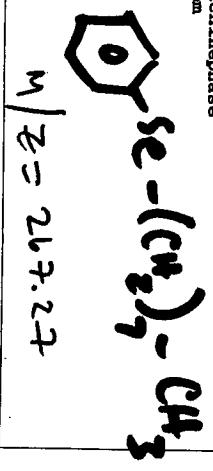
1d_spectrum.1891
Single Pulse Experiment



JEOL

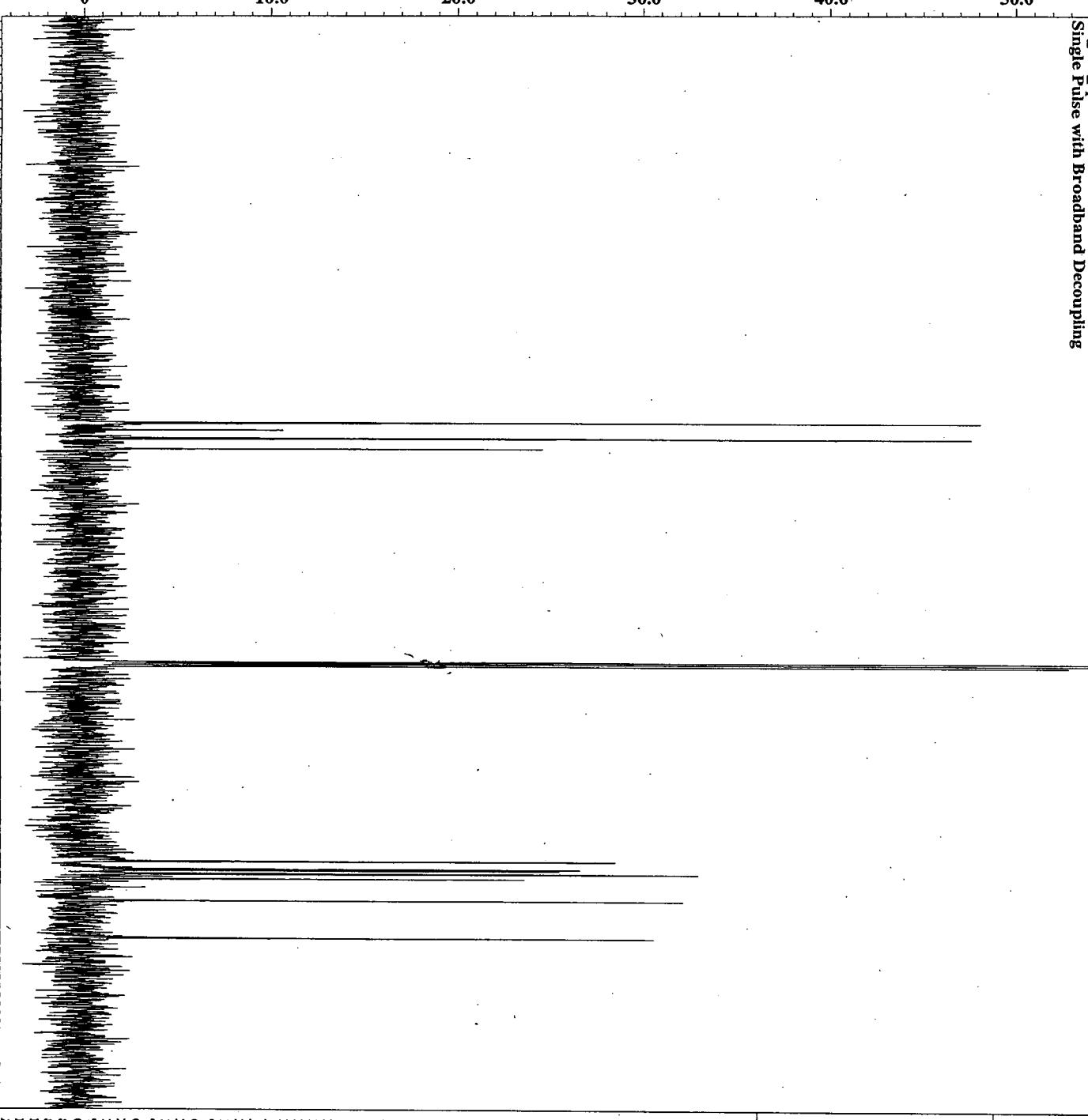
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Revision Date = 22-DEC-2003 01:1:
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fft : 1 : TRUE
machinephase



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Qua90_h1	= 10 [us]	
X90_J0	= 55 [us]	
Irr90_J0	= 55 [us]	
Tr190_J0	= 10 [us]	
Qua90_J0	= 10 [us]	
Spin_lock_90	= 0.1 [ms]	
Spin_lock_attn	= 17.5 [dB]	
Deut_grad_shim_90	= 0.105 [ms]	
Deut_grad_shim_attn	= 10 [dB]	
Adc_card	= 16 / MHz / 20	
Field_strength	= 6.35446 [T]	
Filter_mode	= BUTTERWORTH	
Filter_width	= 2.0567534 [kHz]	
Recv_gain	= 13	
Irr_code	= 4	
Obs_Dwidth	= 1 [us]	

1d_13c_spectrum.1371
Single Pulse with Broadband Decoupling



X : parts per Million : 13C

JEOL

----- PROCESSING PARAMETERS -----
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machinemphase
ppm

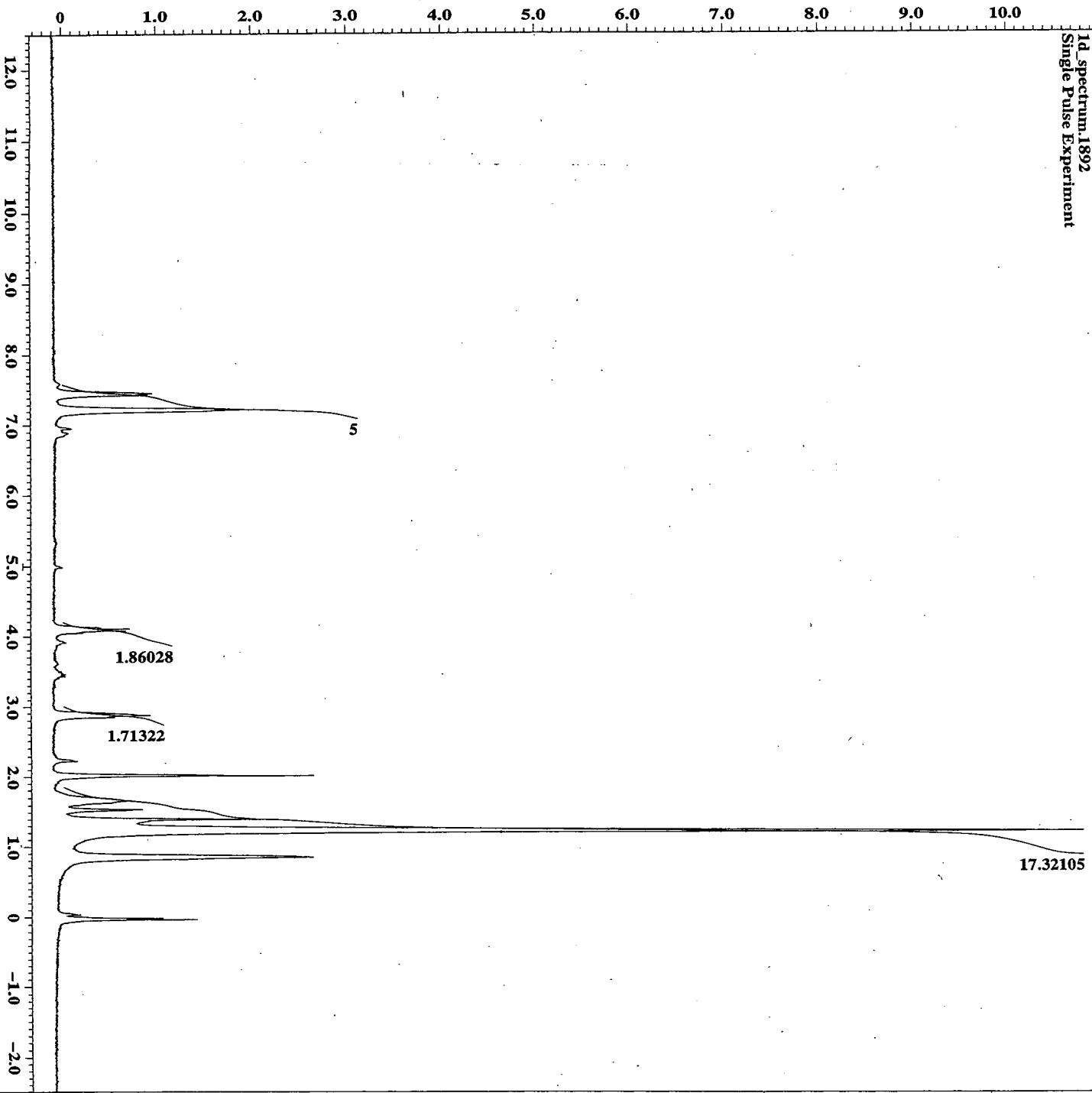
Phase

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X_Points	= 32768
X_Precans	= 4
X_Domain	= ¹³ C
X_Offset	= 10 [ppm]
X_Freq	= 67.93330593 [MHz]
X_Sweep	= 17.006680272 [KHz]
X_Resolution	= 0.51900643 [Hz]
IRR_Domain	= 1H
IRR_Offset	= 5 [ppm]
IRR_Freq	= 271.16608444 [MHz]
Digital_Filter	= 1.9267584 [s]
Filter_Factor	= FALSE
Delay_of_Start	= 1 [s]
Actual_Start_Time	= 22-DEC-2003 02:12:
Acq_Delay	= 57.4 [us]
X90	= 8.5 [us]
IRR90	= 11 [us]
TR190	= 10 [us]
Qua90	= 10 [us]
X90_10	= 8.9 [us]
IRR90_10	= 18 [us]
TR190_10	= 10 [us]
Qua90_10	= 10 [us]
IRR90_10	= 39 [us]
TR190_10	= 55 [us]
Qua90_10	= 10 [us]
Spin_Lock_90	= 10 [us]
Spin_Lock_Attn	= 0.1 [ms]
Deut_Grad_Shim_90	= 17.5 [dB]
Deut_Grad_Shim_Atttn	= 0.1 [ms]
Adc_Gard	= 10 [dB]
Field_Strength	= 16.1 MHz/20
Filter_Mode	= BURGERWORTH
Filter_Width	= 8.50340136 [kHz]

519

220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0

1d spectrum.1892
Single Pulse Experiment



JEOL

---- PROCESSING PARAMETERS ----
dc_balance : 0.2 [Hz]
sepD : 0.2 [Hz]
fft : 1 : TRUE
machinephase
ppm

m/z = 325.39

---- ACQUISITION PARAMETERS ----
File Name : 1d.spectrum.1892
Author :
Sample ID : S#65109
Content : Single Pulse Exper.
Creation Date : 22-DEC-2003 02:49:
Revision Date : 22-DEC-2003 01:53:
Spec Site : WKU GSX-270

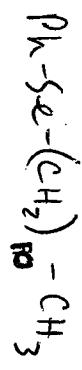
Spec Type : DELTA_NMR
Data Format : 1D COMPLEX
Dimensions : X
Dim Title : 1H
Dim Size : 16384
Dim Units : [ppm]
Scans : 8
Mod. return : 1
X_points : 16384
X_prescans : 0
X_domain : 1H
X_offset : 5 [ppm]
X_freq : 270.1660844 [MHz]
X_sweep : 4.03350628 [MHz]
X_resolution : 0.24740659 [Hz]
X_acc_duration : 4.0419328 [s]
Digital_filter : FALSE
Filter_factor : 1
delay_of_start : 1 [s]
Actual_start_time : 22-DEC-2003 02:48:
Acq_delay : 0.244 [ms]
x90 : 11 [us]
Irr90 : 11 [us]
Tri90 : 10 [us]
Qu90 : 10 [us]
x90_hi : 18 [us]
Irr90_hi : 18 [us]
Tri90_hi : 10 [us]
Qu90_hi : 10 [us]
x90_lo : 55 [us]
Irr90_lo : 10 [us]
Tri90_lo : 10 [us]
Qu90_lo : 10 [us]
Spin_lock_90 : 0.1 [ms]
Spin_lock_attn : 17.5 [dB]
Deut_grad_shim_90 : 0.105 [ms]
Deut.grad.shim.attn : 10 [dB]
16 / MHz / 20
adc_card : 6.315446 [T]
Field_strength : BUTTERWORTH
Filter_mode : 2.02675314 [kHz]
Filter_width : 20
Recv_gain : 4
Irr_code : 1 [us]
Obs_width : 1 [us]

520

1d_13c_spectrum.1372
Single Pulse with Broadband Decoupling

JEOL

----- PROCESSING PARAMETERS -----
dc_balance :
sep : 2 [Hz]
fft : 1 : TRUE
machinephase
ppm



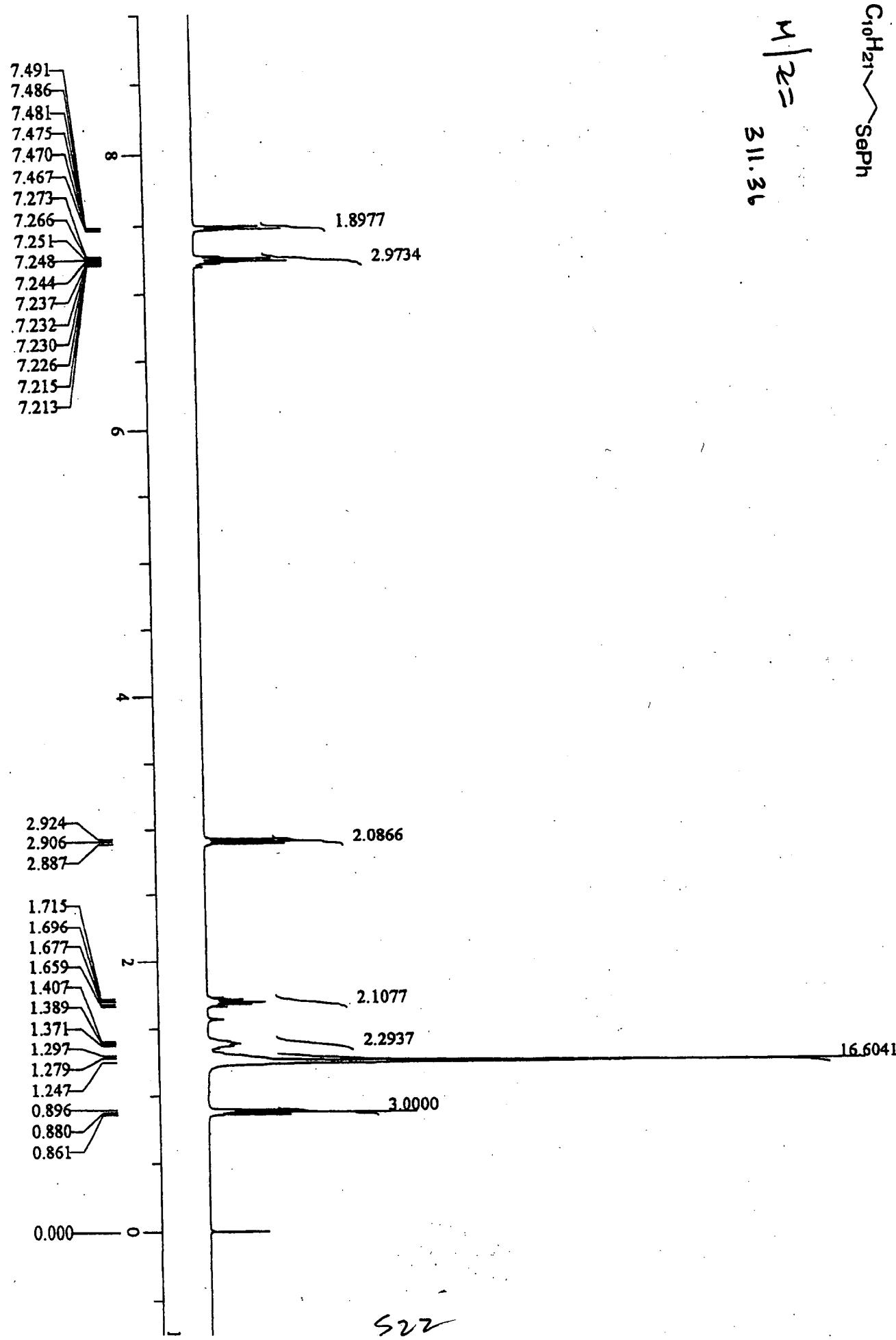
----- ACQUISITION PARAMETERS -----
File Name = 1d_13c_spectrum.13
Author =
Sample ID = S466089
Content = Single Pulse with
Creation Date = 22-DEC-2003 02:58:
Revision Date = 22-DEC-2003 01:59:
Spec Size = WET GSF-270

Spec Type = DEFLATE_NMR
Data Format = 1D COMPLEX
Dimensions = X
Dim Title = 13C
Dim Size = 32768
Dim Units = [PPM]
Scans = 80
Mod_return = 1
X_points = 32768
X_prsescans = 4
X_domain = 13C
X_offset = 10 [ppm]
X_freq = 67.93330993 [MHz]
X_sweep = 17.00680272 [kHz]
X_resolution = 0.51800643 [Hz]
Irr_domain = 1H
Irr_offset = 5 [ppm]
Irr_freq = 270.16608844 [MHz]
X_acq_duration = 1.9267584 [s]
Digital_filter = FALSE
Filter_factor = 1
Delay_of_start = 1 [s]
Actual_start_time = 22-DEC-2003 02:51:
Acq_delay = 57.4 [us]
x90 = 8.5 [us]
Irr90 = 11 [us]
Tri90 = 10 [us]
Qua90 = 10 [us]
x90_11 = 8.9 [us]
Irr90_11 = 18 [us]
Tri90_hi = 10 [us]
Qua90_hi = 10 [us]
x90_10 = 39 [us]
Irr90_10 = 55 [us]
Tri90_10 = 10 [us]
Qua90_10 = 10 [us]
Spin_lock_90 = 0.1 [ms]
Spin_lock_attn = 17.5 [dB]
Deut_grad_shim_90 = 0.1 [ms]
Deut_grad_shim_attn = 10 [dB]
Adc_card = 16.1MHz/20
Field_strength = 6.45446 [T]
Filter_mode = BUTTERWORTH
Filter_width = 8.50340136 [kHz]

S21

220.0210.0200.0190.0180.0170.0160.0150.0140.0130.0120.0110.0100.090.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0

X : Parts Per Million : 13C





132.297

130.681

128.935

126.529

77.319

77.000

76.681

31.903

30.120

29.824

29.626

29.611

29.566

29.482

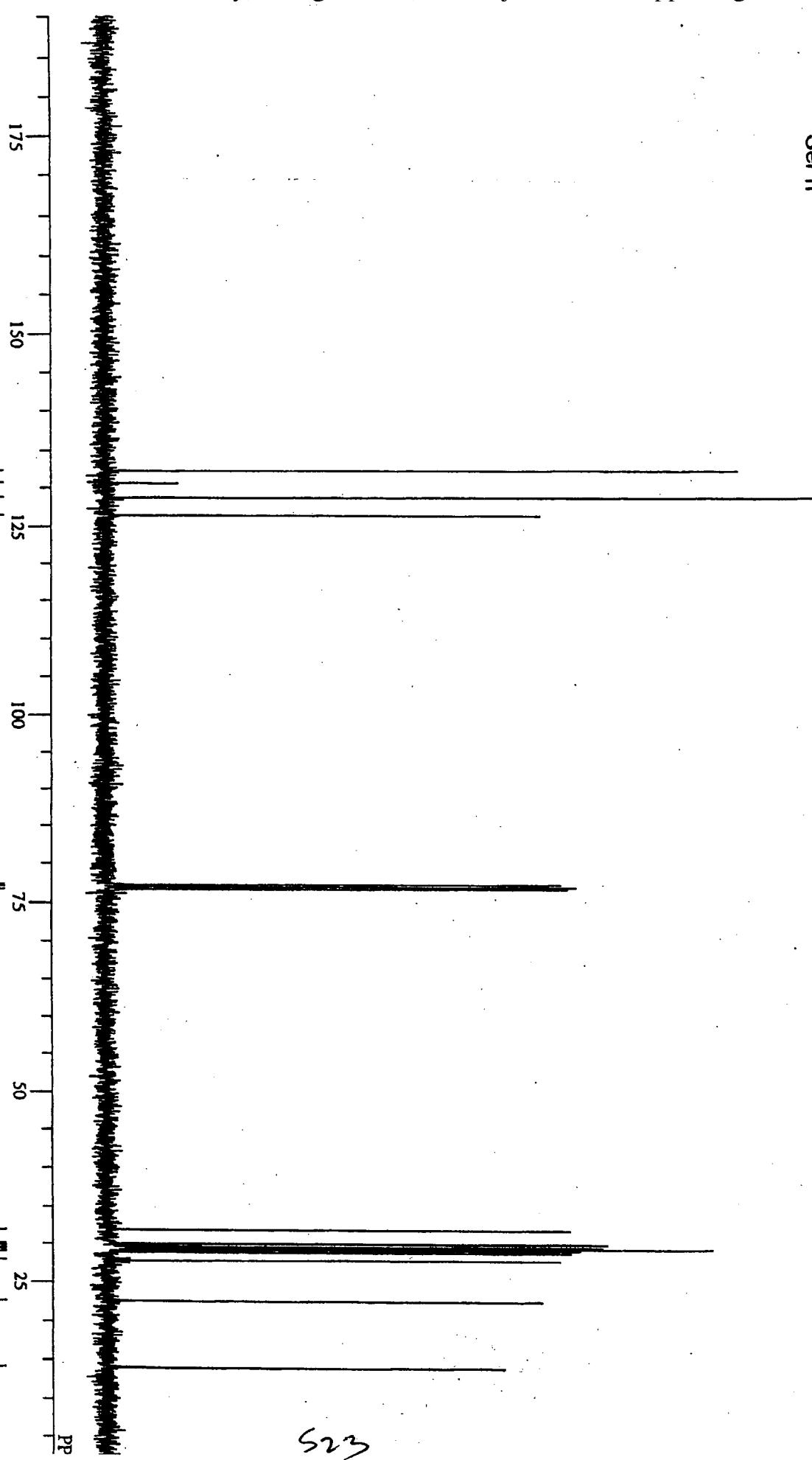
29.338

29.072

27.926

22.682

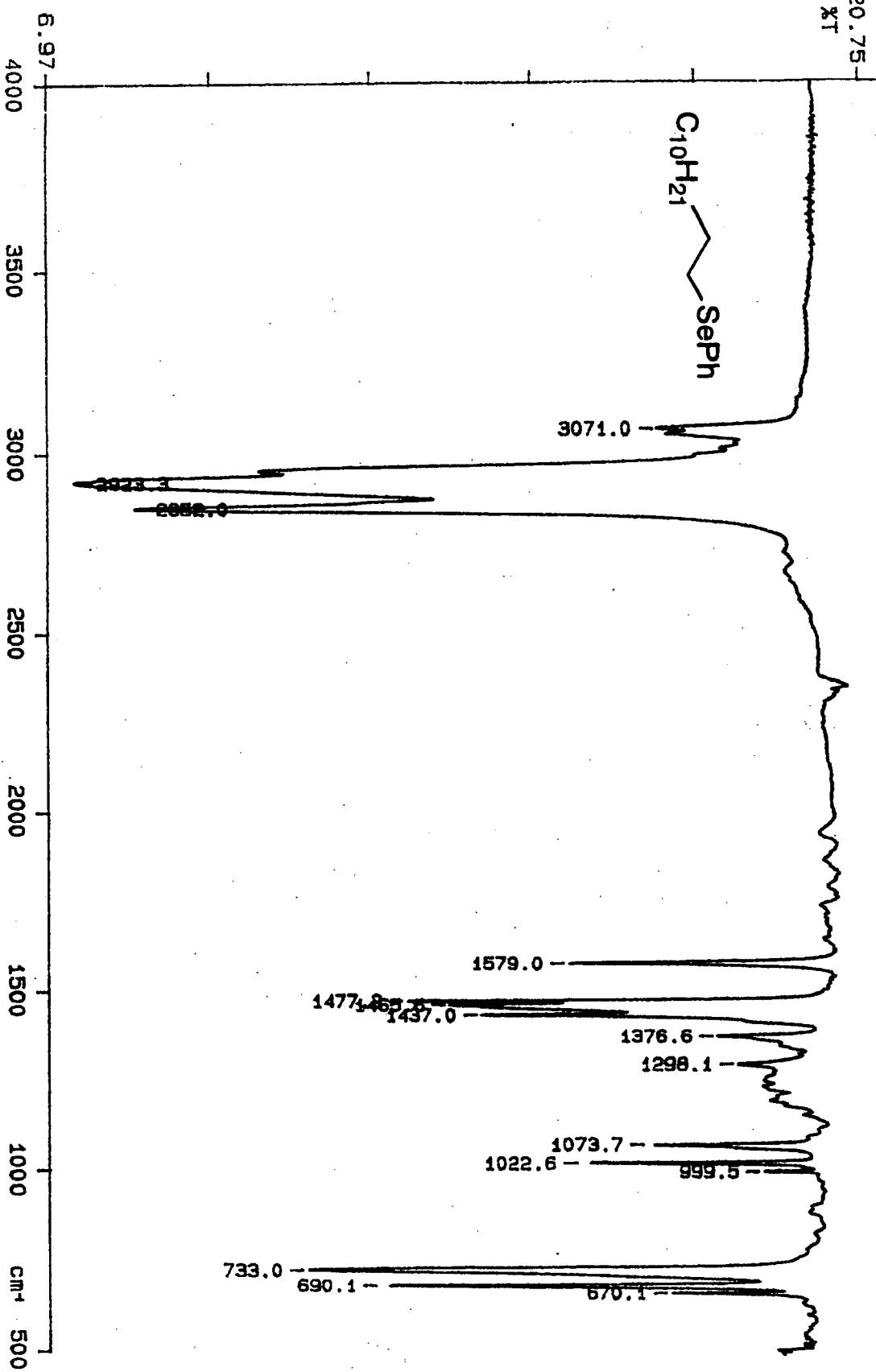
14.121



523

PERKIN ELMER

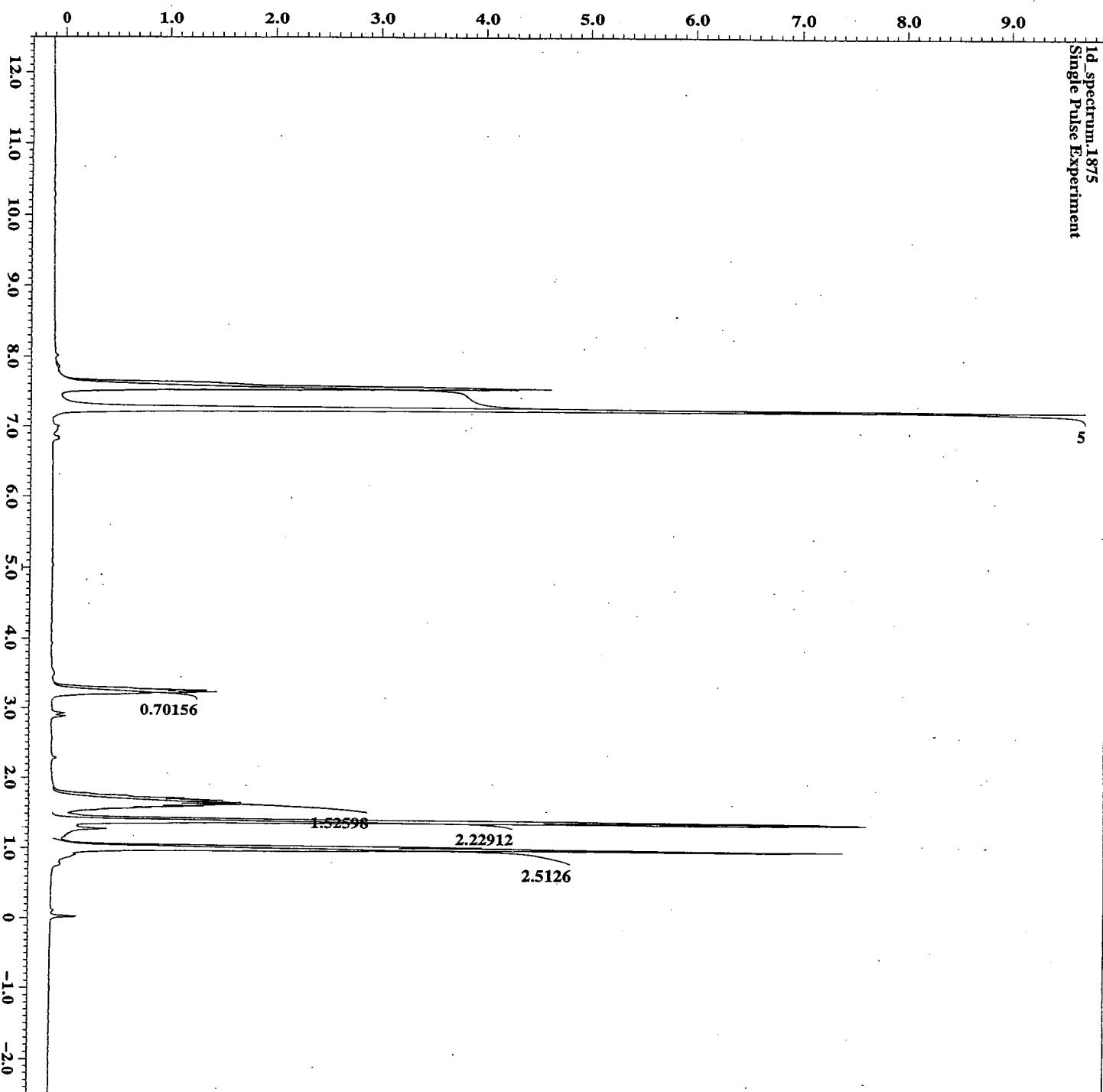
120.75
%T



01/12/22 20: 40
X: 16 scans, 4.0cm⁻¹, flat
1-phenylselenododecane

1d spectrum.1875
Single Pulse Experiment

5



JEOL

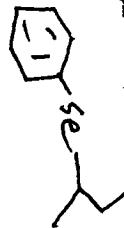
— PROCESSING PARAMETERS —
dc_balance = 0.1 [Hz]
sep = 0.2 [Hz]
fit = 1 : TRUE
machinephase
ppm

--- ACQUISITION PARAMETERS ---	
File Name	= 1d.spectrum.1875
Author	= S#445470
Sample ID	= Single Pulse Exper
Content	= 12-DEC-2003 07:52:
Creation Date	= 12-DEC-2003 07:10:
Revision Date	= WUR GSX-270
Spec Site	
Spec Type	= DELTA_NMR
Data Format	= 1D COMPLEX
Dimensions	= X
Dim Title	= ¹ H
Dim Units	= [ppm]
Scans	= 8
Mod_return	= 1
X_points	= 16384
X_prescans	= 0
X_domain	= ¹ H
X_offset	= 5 [ppm]
X_freq	= 270.04668844 [MHz]
X_sweep	= 4.0530628 [MHz]
X_resolution	= 0.24740639 [Hz]
X_act_duration	= 4.0419328 [s]
Digital_Filter	= FALSE
Filter_factor	= 1
delay_of_start	= 1 [s]
Actual_start_time	= 12-DEC-2003 07:51:
Acq_delay	= 0.244 [ms]
X0	= 11 [us]
IRR90	= 11 [us]
Tri90	= 10 [us]
Qua90	= 10 [us]
X90_hi	= 18 [us]
IRR90_hi	= 18 [us]
Tri90_hi	= 10 [us]
Qua90_hi	= 10 [us]
X90_lo	= 55 [us]
IRR90_lo	= 55 [us]
Tri90_lo	= 10 [us]
Qua90_lo	= 10 [us]
Spin_lock_90	= 0.1 [ms]
Spin_lock_attn	= 17.5 [dB]
Deut_grad_shim_90	= 0.105 [ms]
Adc_grad_shim_attn	= 10 [dB]
Adc_card	= 16 / 1MHz / 20
Field_strength	= 6.35446 [T]
Field_mode	= BUTTERWORTH
Filter_width	= 2.03675314 [kHz]
Recvr_gain	= 13
Irr_code	= 4
Obs_pwidth	= 1 [us]

1d_13c_spectrum.1350
Single Pulse with Broadband Decoupling

JEOL

— PROCESSING PARAMETERS —
dc_balance
scxp : 2[Hz]
fft : 1 : TRUE
machinephase
ppm



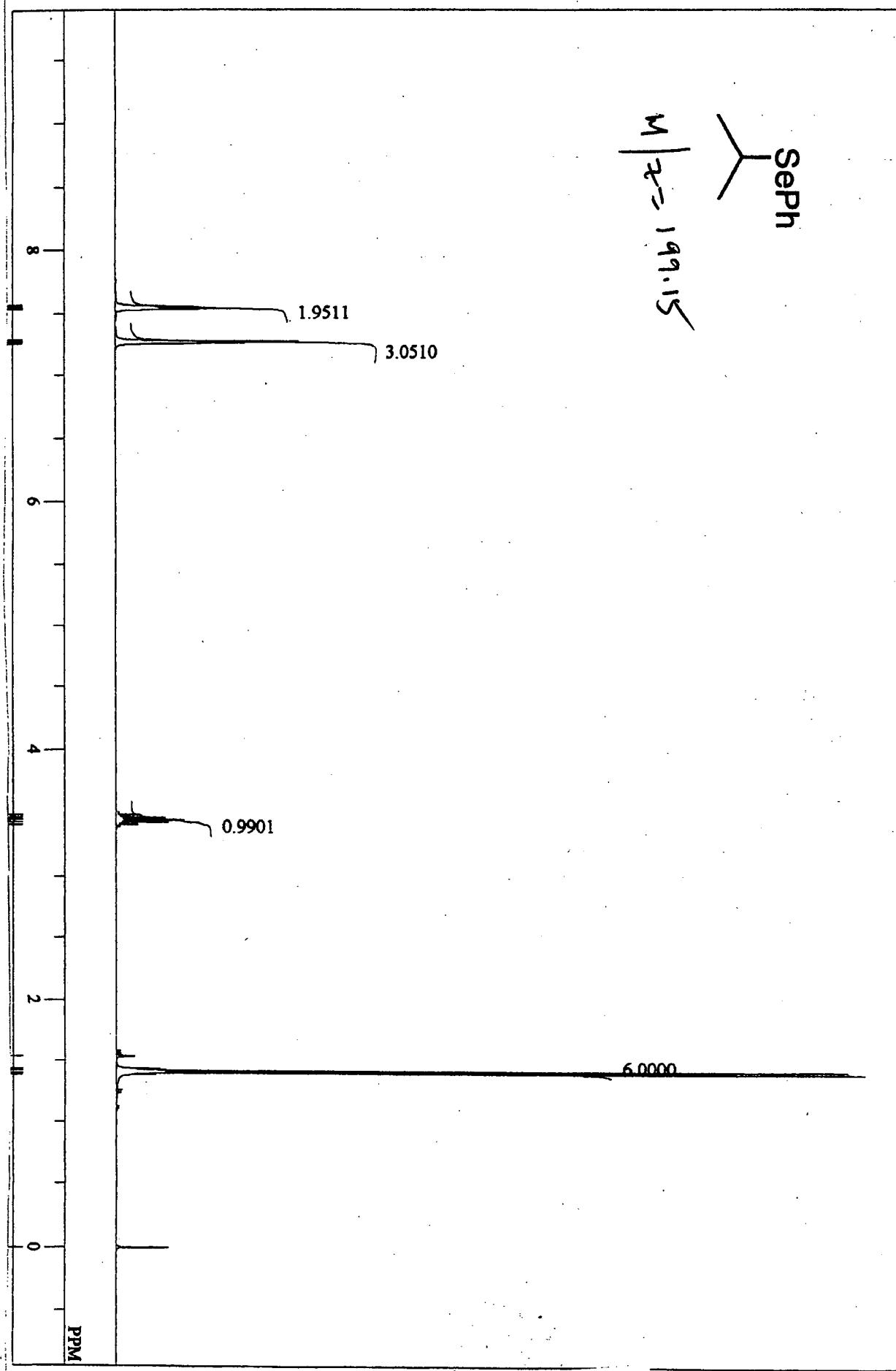
— ACQUISITION PARAMETERS —
File Name = 1d_13c_spectrum.13
Author =
Sample ID = S1250433
Content = Single Pulse with
Creation Date = 12-DEC-2003 08:10:
Revision Date = 12-DEC-2003 07:08:
Spec Site = WUR GSX-270

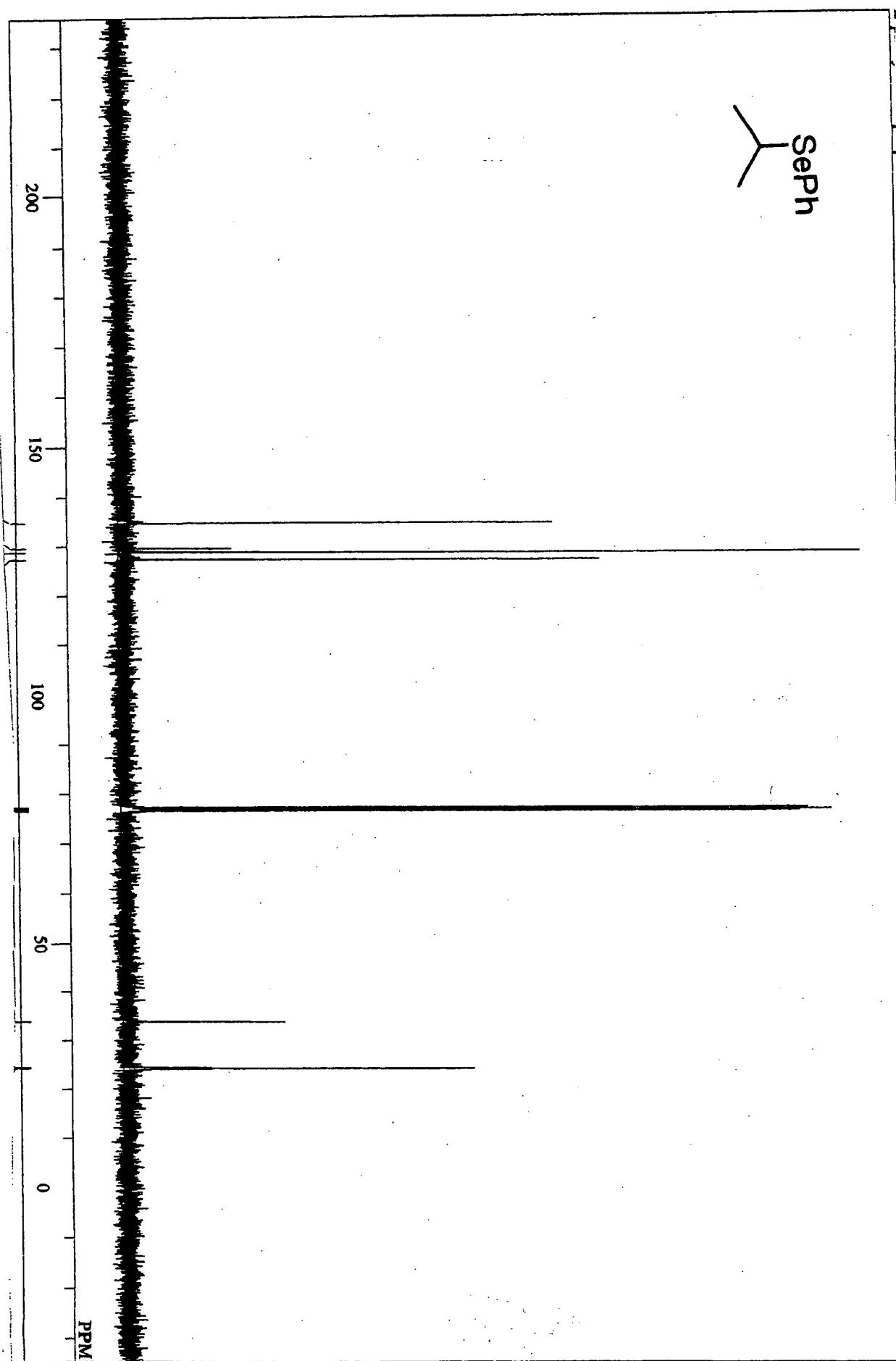
Spec Type	DELTA_NMR
Data Format	1D COMPLEX
Dimensions	X
Dim Title	-13C
Dim Size	32768
Dim Units	[ppm]
Scans	120
Mod_return	1
X_Points	32768
X_Prescans	4
X_Domain	-13C
X_Offset	100[ppm]
X_Freq	67.93330993 [MHz]
X_Sweep	17.00680271 [kHz]
X_Resolution	0.5190643[Hz]
Irr_Domain	1H
Irr_Offset	5[ppm]
Irr_Freq	270.16608844 [MHz]
Digital_Filter	1.9267584[s]
Filter_Factor	1
Delay_Start	1[s]
Actual_Start_Time	12-DEC-2003 08:00:
Acq_Delay	57.4[us]
X90	8.5[us]
Tr190	11[us]
Tr190	10[us]
Qu90	10[us]
X90_10	8.9[us]
Irr90_hi	18[us]
Tr190_hi	10[us]
Qu90_hi	10[us]
Deut_Grad_Shim_Lattn	39[us]
Adc_Card	55[us]
Filter_Strength	10[us]
Filter_Mode	0.1[ms]
Filter_Width	17.5[ms]
Spin_Lock_AltN	0.105[ms]
Deut_Grad_Shim_90	10[dB]
Deut_Grad_Shim_Lattn	16/1MHz/20
Adc_Card	6.345446[T]
Filter_Strength	8.50340136[kHz]

S26

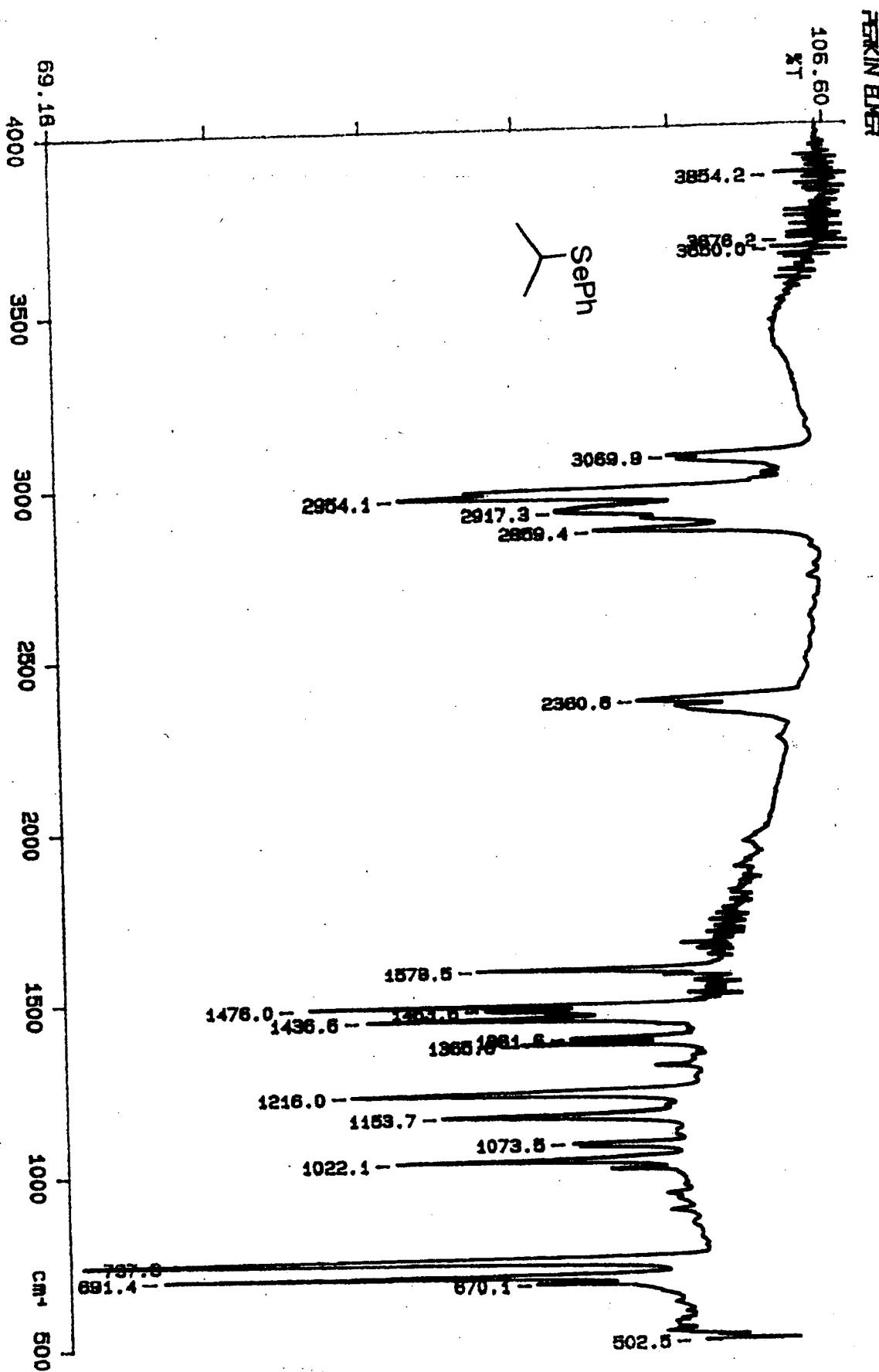
220.0210.0200.0190.0180.0170.0160.0150.0140.0130.0120.0110.0100.090.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0-20.0

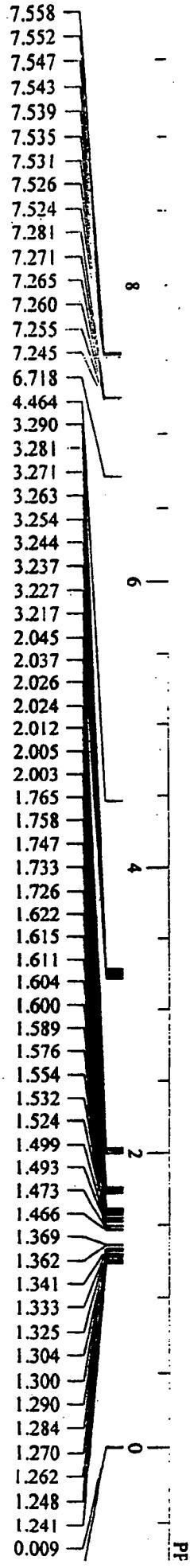
X : parts per Million : 13C

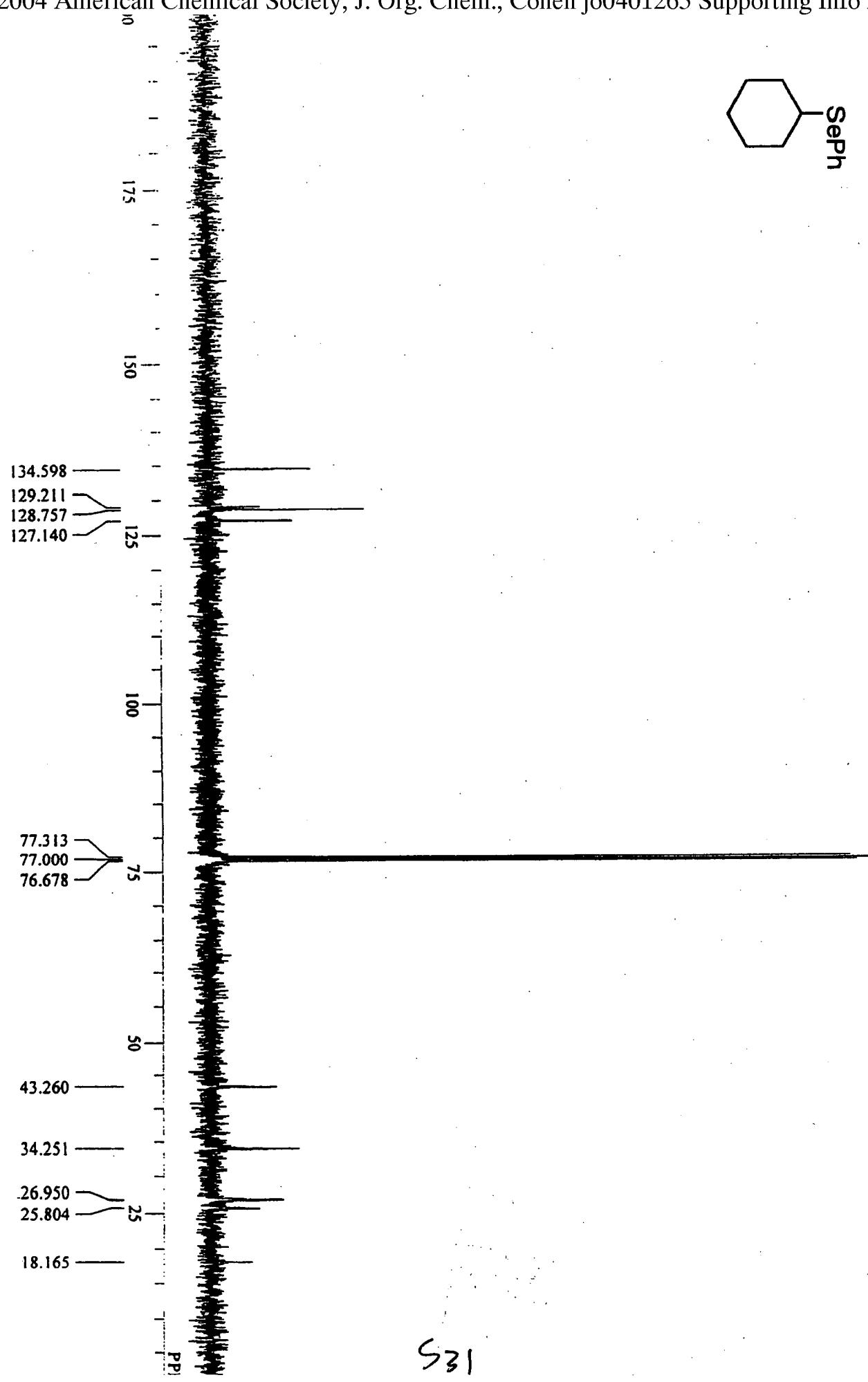
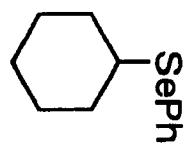




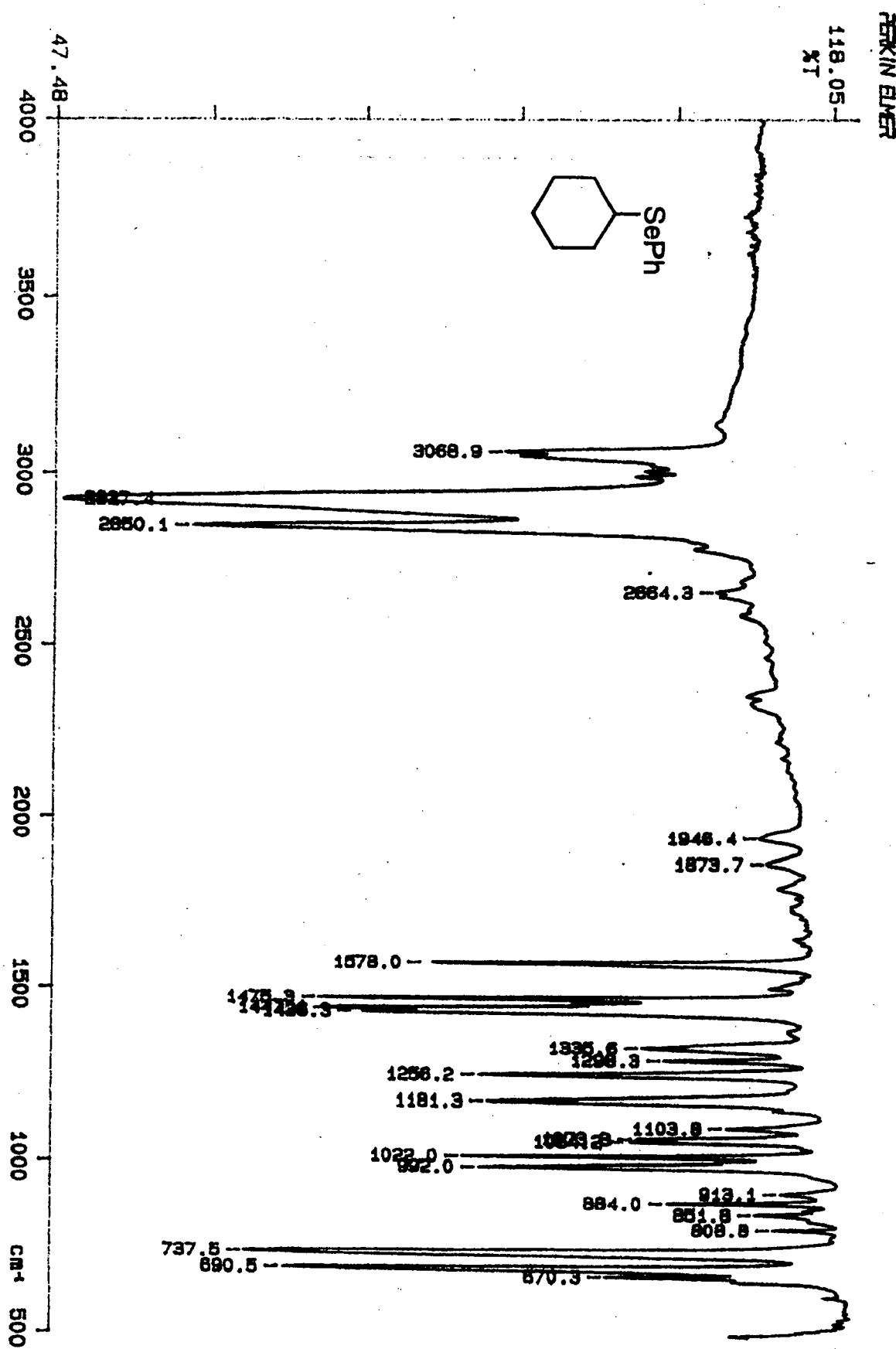
528.





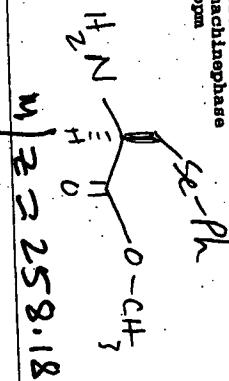
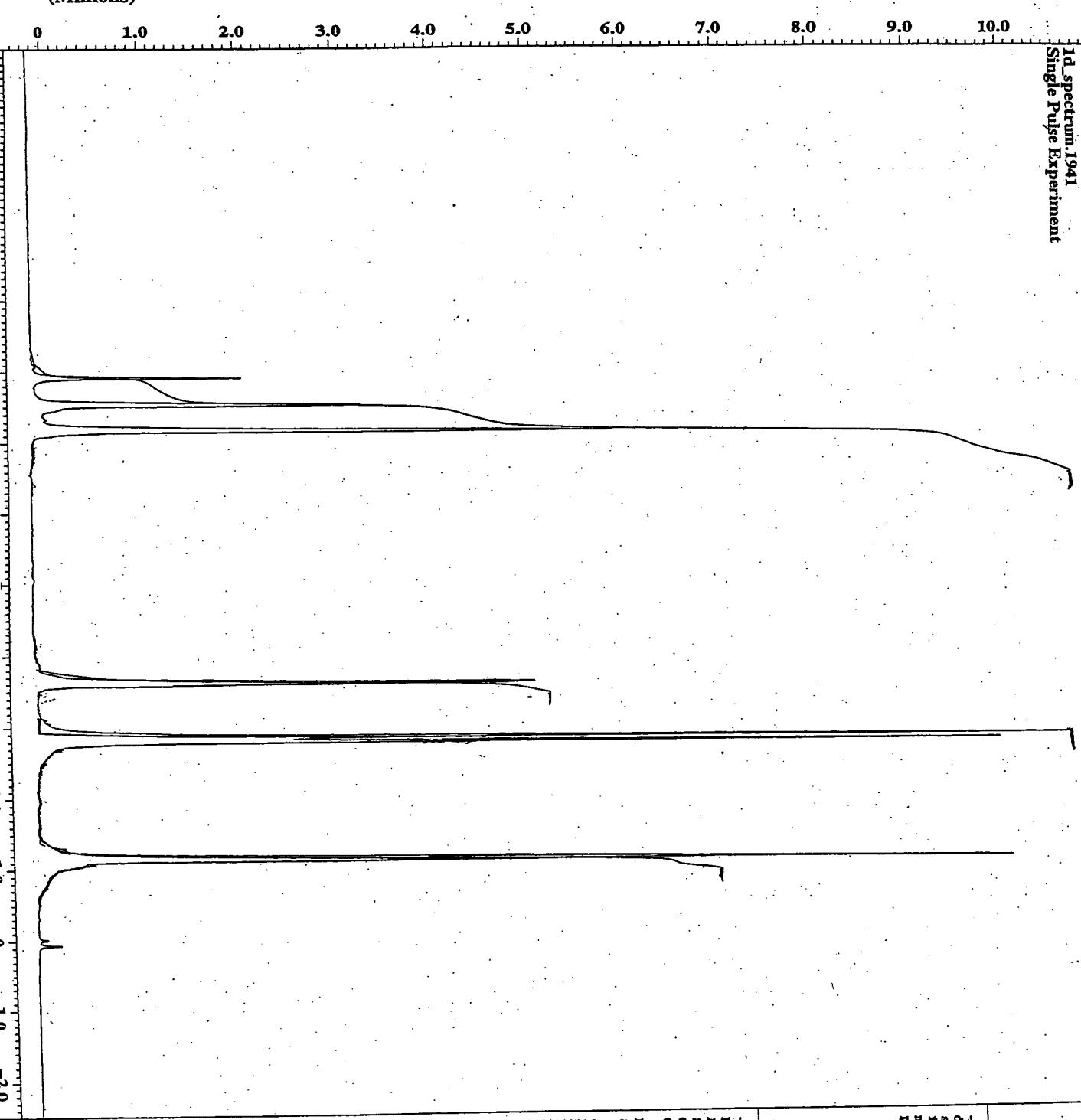


S31



1d_spectrum.1941
Single Pulse Experiment

JEOL



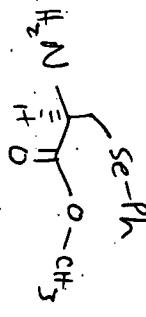
--- PROCESSING PARAMETERS ---	
File Name	1d_spectrum.1941
Author	S#110673
Sample ID	Single Pulse Exper
Content	19-JAN-2004 04:12:
Creation Date	19-JAN-2004 03:11
Revision Date	19-JAN-2004 03:11
Spec Site	WKU GSX-270
Spec Type	1D COMPLEX
Data Format	DELTA_NMR
Dimensions	1H
dim title	16384
dim size	16384
dim units	[ppm]
scans	8
Mod_Return	1
X_Points	16384
X_prescans	0
X_domain	1H
X_offset	51[ppm]
X_freq	270.1660884[MHz]
X_sweep	4.05350628[Hz]
X_resolution	0.2470639[Hz]
X_acq_duration	4.0419328[8]
Digital_Filter	FALSE
Filter_Factor	1
Filter_Or_Start	1[s]
Delay_of_Start_time	19-JAN-2004 04:11:
Actual_start_time	19-JAN-2004 04:11:
Acc_delay	0.244[ms]
X90	11[us]
IRF90	11[us]
TR190	10[us]
Q90	10[us]
Q90_10	55[us]
IRF90_10	10[us]
X90_hi	18[us]
IRF90_hi	18[us]
TR190_hi	10[us]
Qua90_hi	10[us]
Q90_10	55[us]
IRF90_10	10[us]
Qua90_lo	10[us]
Spin90_lo	0.1[ms]
Spin_lock_90	17.5[db]
Spin_lock_sttn	0.105[ms]
Deut_grad_shim_90'	10[db]
Deut_grad_shim_attn	16[MHz/20
Adic_card	6.345446[F]
Field_strength	BUTTERWORTH
Filter_mode	2.02675314[MHz]
Filter_width	14
Reovr_gain	1
IRF_code	1[us]
Obs_width	

533

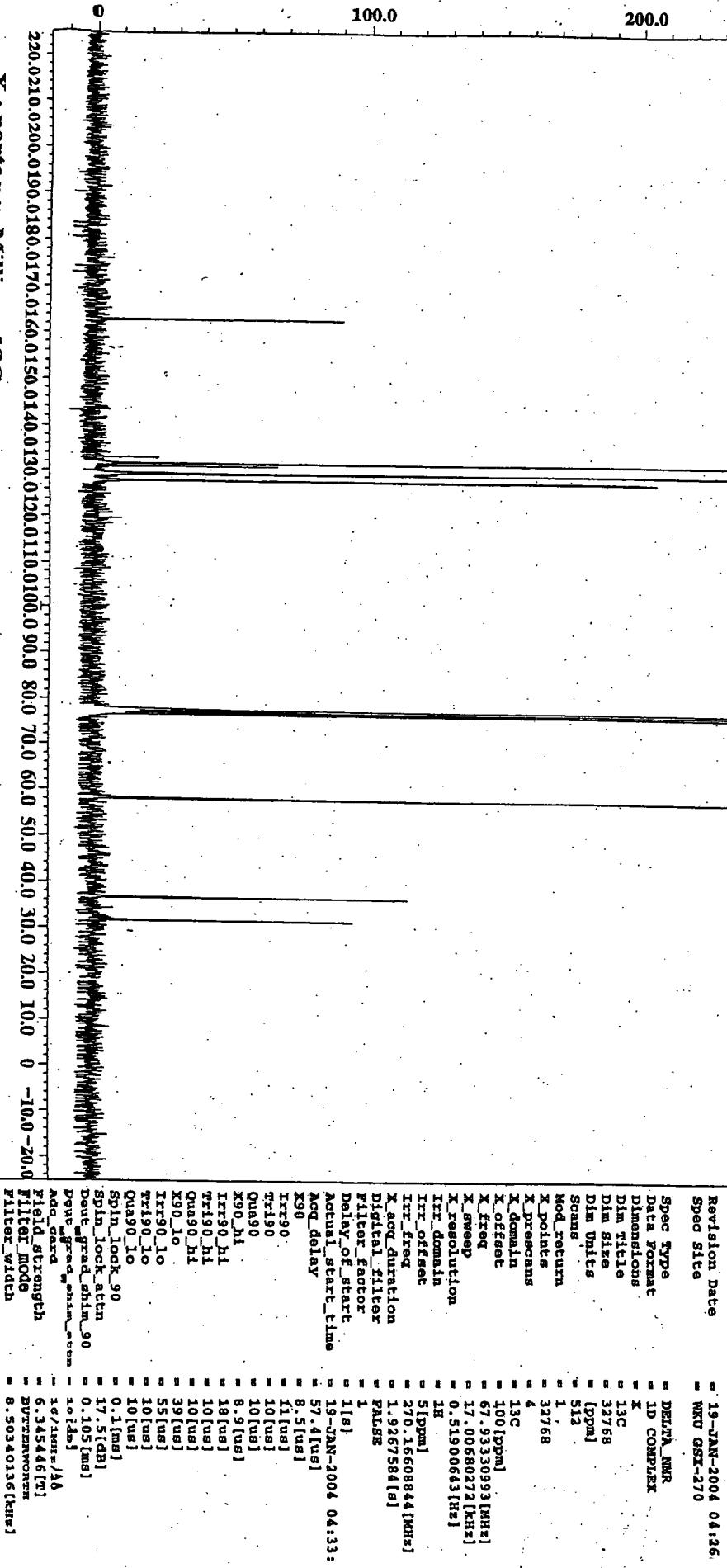
1d_13c_spectrum.1417
Single Pulse with Broadband Decoupling

JEOL

----- PROCESSING PARAMETERS -----
dc_balance : 0 [Hz]
sep0 : 2 [Hz]
fft : 1 : TRUE
machinephase : ppm



----- ACQUISITION PARAMETERS -----
File Name : 1d_13c_spectrum.14
Author :
Sample ID : #133633
Content : Single pulse with
Creation Date : 19-JAN-2004 05:15,
Revision Date : 19-JAN-2004 04:26
Spec Site : WKE GSX-270



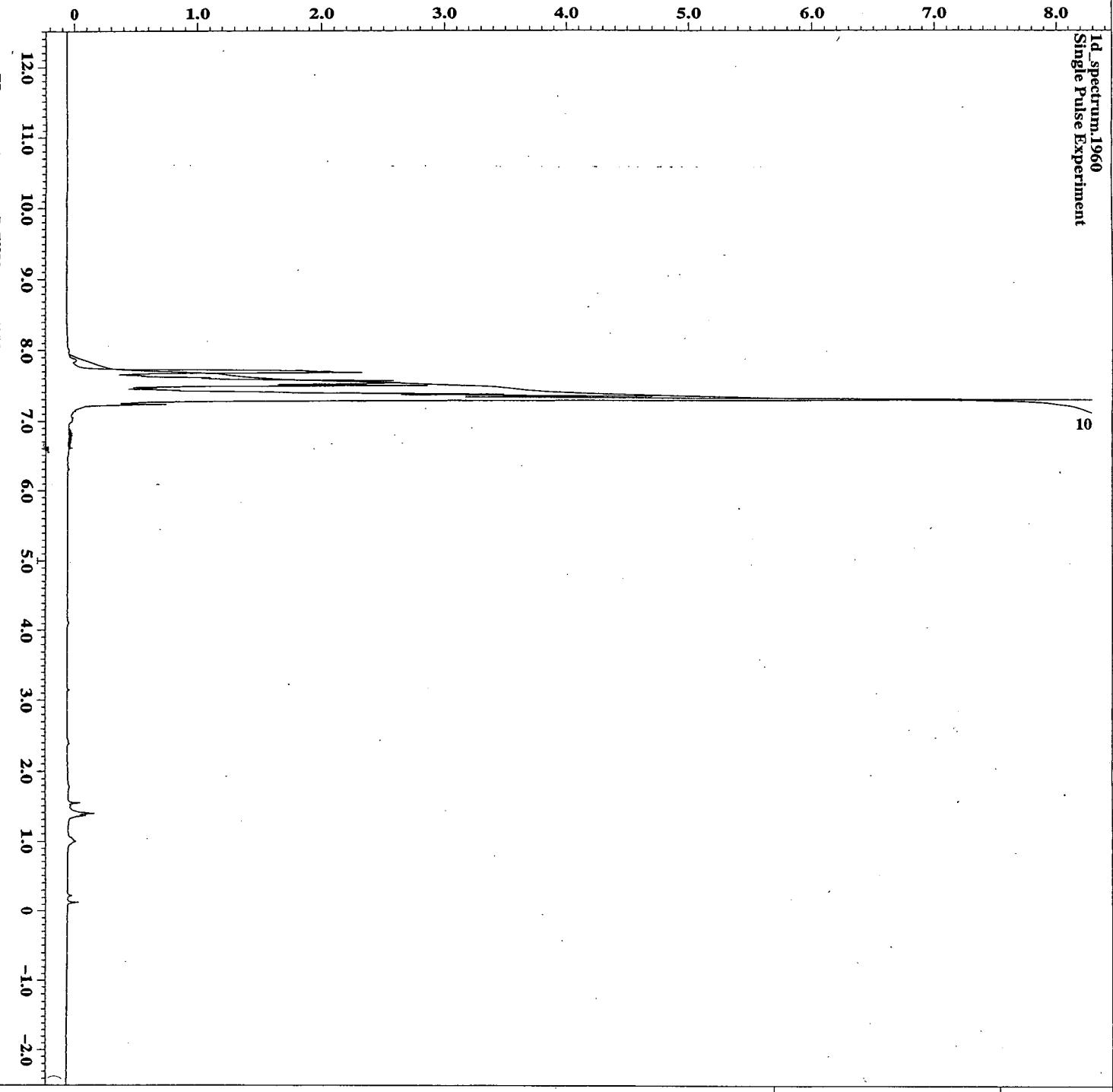
534

1d_spectrum.1960
Single Pulse Experiment

JEOL
(ext. card
C50H)

----- PROCESSING PARAMETERS -----
dc_balance = 1.0Hz
sep : 0.21Hz
fft : 1 : TRUE
machinephase
ppm

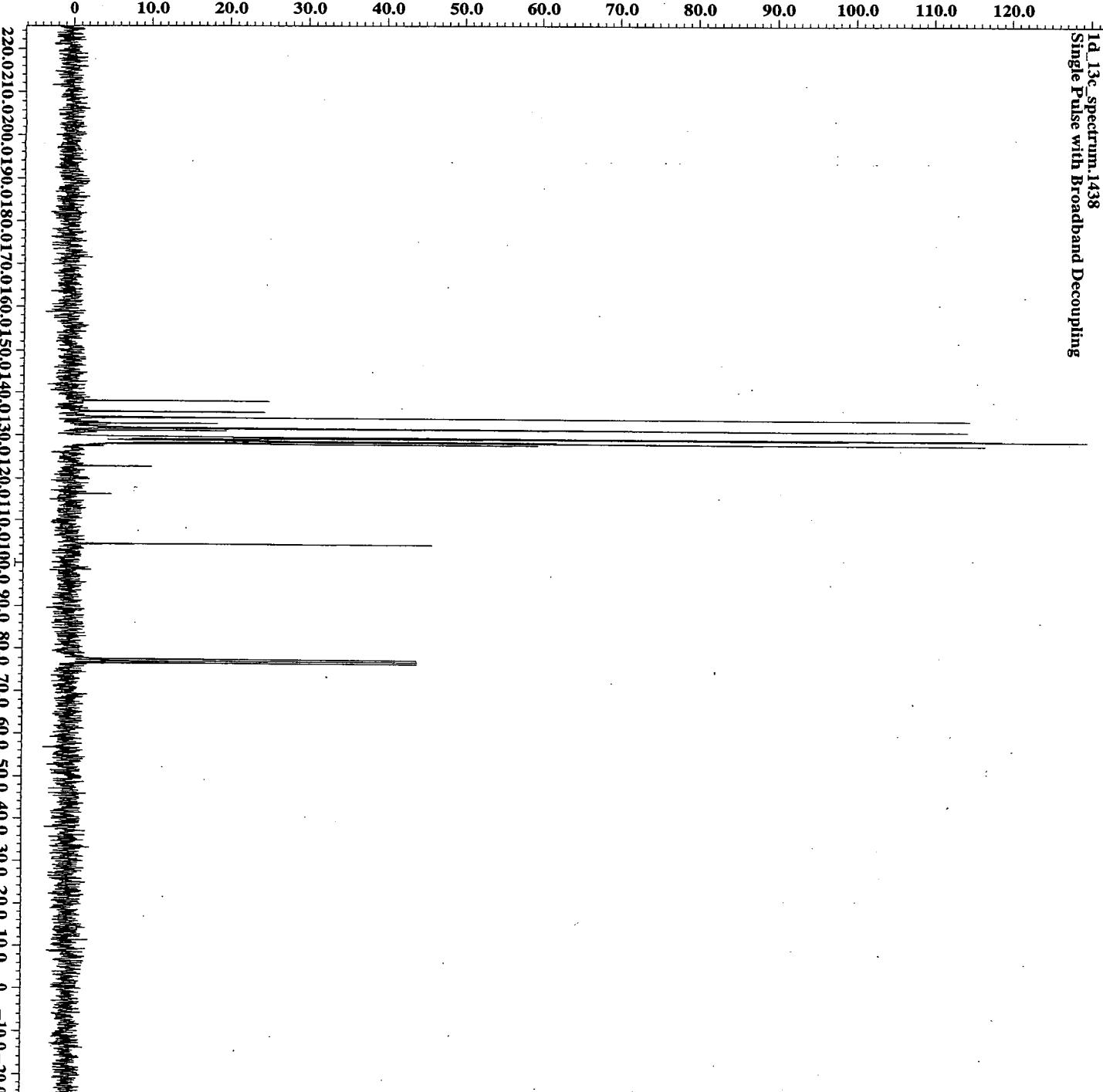
Ph - C = C - Se - Ph



---- ACQUISITION PARAMETERS ----	
File Name	= 1d_spectrum.1960
Author	= SH132119
Sample ID	= Single Pulse Exper
Content	= 21-JAN-2004 04:31:
Creation Date	
Revision Date	= 21-JAN-2004 03:45:
Spec Site	= WDU GSX-270
Spec Type	= DELTA_NMR
Data Format	= 1D COMPLEX
Dimensions	= X
Dim Title	= 1H
Dim Size	= 10384
Dim Units	= [ppm]
Scans	= 8
Mod. return	= 1
X_Points	= 16384
X_Prescans	= 0
X_Domain	= 1H
X_Offset	= 51(ppm)
X_Freq	= 270.16808846(MHZ)
X_Sweep	= 4.0535625(MHZ)
X_Resolution	= 0.24746639(HZ)
X_Acq_duraion	= 4.0419328(s)
Digital_Filter	= FALSE
Filter_Factor	= 1(s)
Delay_of_start	= 1(s)
Actual_start_time	= 21-JAN-2004 04:30:
Acq_delay	= 0.244(ms)
X90	= 11[us]
IRF90	= 11[us]
TRI90	= 10[us]
Q90	= 10[us]
X90_hi	= 18[us]
IRF90_hi	= 18[us]
TRI90_hi	= 10[us]
Q90_hi	= 10[us]
X0_10	= 55[us]
IRF90_10	= 55[us]
TRI90_10	= 10[us]
Q90_10	= 10[us]
Spin_lock_90	= 0.1(ms)
Spin_lock_attn	= 17.5(dB)
Dent_grad_shim_90	= 0.105(ms)
Dent_grad_shim_attn	= 10(gB)
Adv_card	= 16/1MHz/20
Field_strength	= 6.345446(T)
Filter_mode	= AUTOPERNORTH
Filter_width	= 2.02675314(Hz)
Revr_gain	= 11
IRF_code	= 4
Obs_width	= 1[us]

535

1d_13c_spectrum.1438
Single Pulse with Broadband Decoupling



X : parts per Million : 13C

JEOL

— PROCESSING PARAMETERS —
dc_balance
saxp : 2 [Hz]
fft : 1 : TRUE
machinephase
ppm

Triclo-Soph

--- ACQUISITION PARAMETERS ---	
File Name	= 1d_13c_spectrum.14
Author	= S#134194
Sample ID	= Single Pulse with
Content	= 13C
Creation Date	= 21-JAN-2004 04:40:
Revision Date	= 21-JAN-2004 03:52:
Spec Site	= WKU GSX-270
Spec Type	= DELTA_NMR
Data Format	= 1D COMPLEX
Dimensions	= X
Dim Title	=
Dim Size	= 32768
Dim Units	= [ppm]
Scans	= 75
Mod_Return	= 1
X_points	= 32768
X_precs	= 4
X_domain	= 13C
X_offset	= 100 [ppm]
X_freq	= 67.9330993 [MHz]
X_sweep	= 17.90680272 [kHz]
X_resolution	= 0.51900643 [Hz]
IRR_domain	= 1H
IRR_offset	= 5 [ppm]
IRR_freq	= 270.16608844 [MHz]
X_acq_duration	= 1.9267584 [s]
Digital_Filter	= FALSE
Filter_factor	= 1
Delay_of_start	= 1 [s]
Actual_start_time	= 21-JAN-2004 04:34:
Acq_delay	= 57.4 [us]
X90	= 8.5 [us]
Tr90	= 11 [us]
Tr190	= 10 [us]
Qu90	= 10 [us]
X90_10	= 8.9 [us]
Tr90_10	= 18 [us]
Tr190_hi	= 10 [us]
Qu90_hi	= 10 [us]
Dant_grad_shim_90	= 0.105 [ms]
Dant_grad_shim_attn	= 10 [dB]
Adv_card	= 16/1MHz/20
Field_strength	= 6.34546 [T]
Filter_mode	= BUTTERWORTH
Filter_width	= 8.50340136 [MHz]

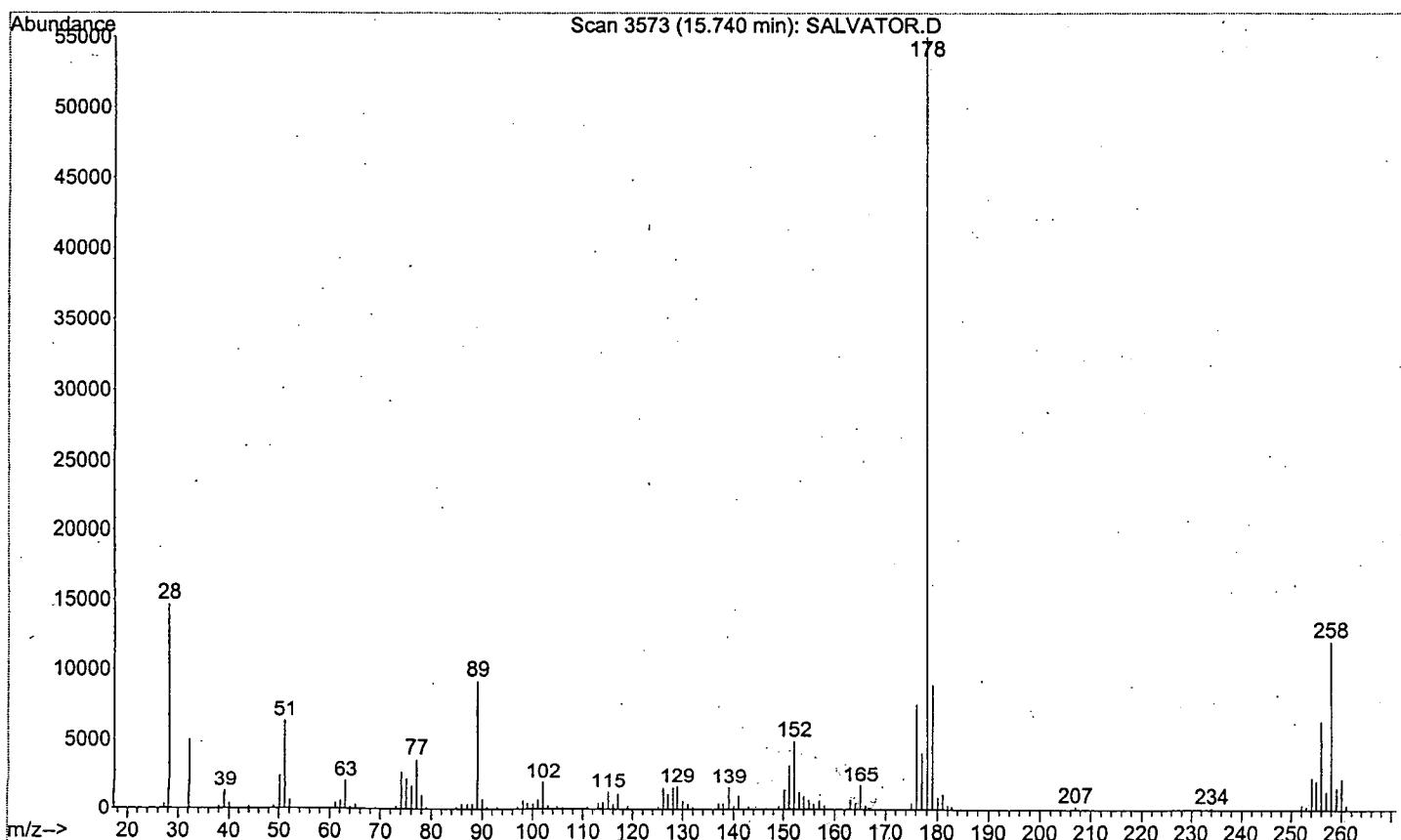
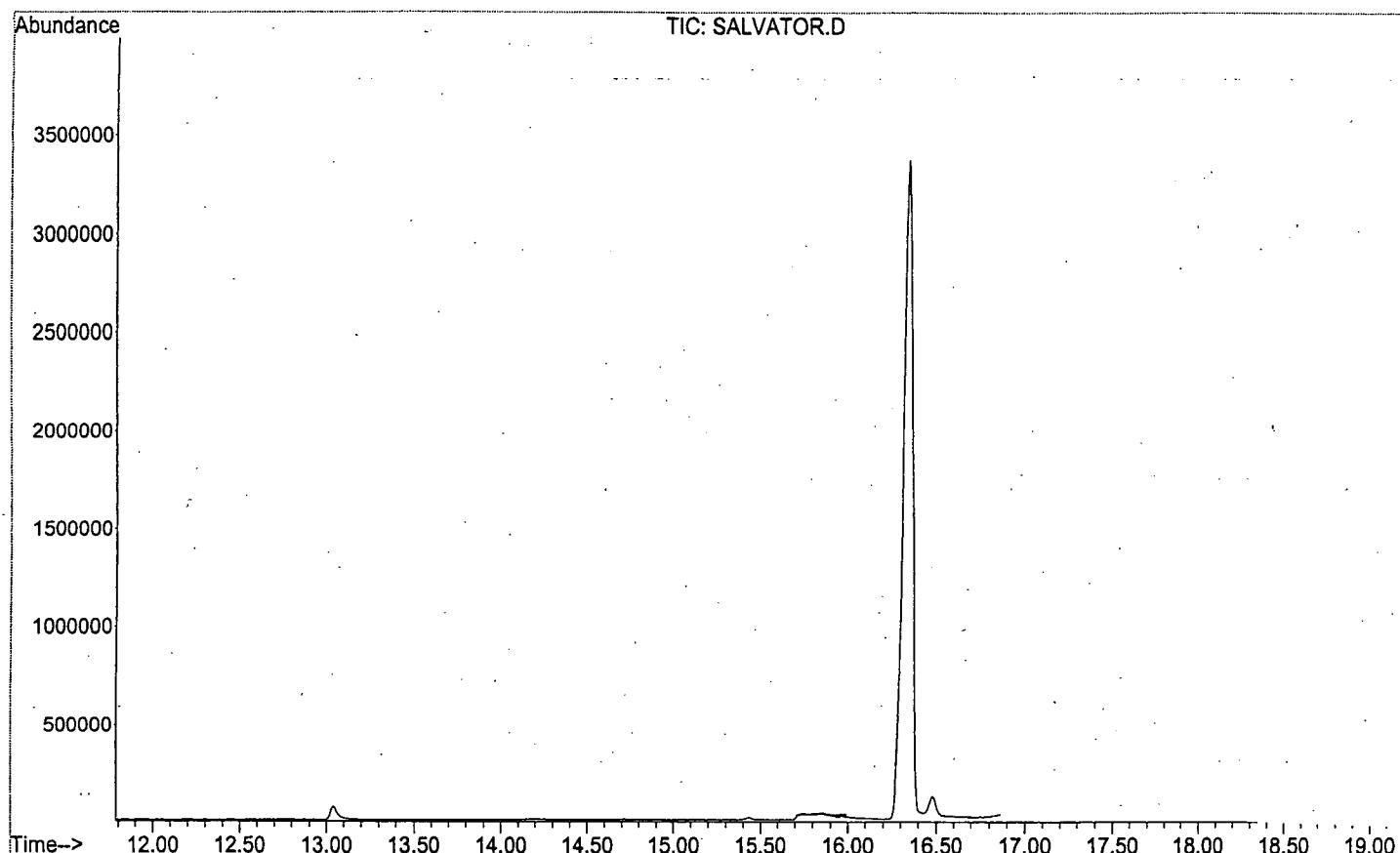
Acquired : 22 Jan 2004 10:13 using AcqMethod PHIL

Instrument : 5973 MSD

Sample Name: Diphenyl alkyne selenium type thing

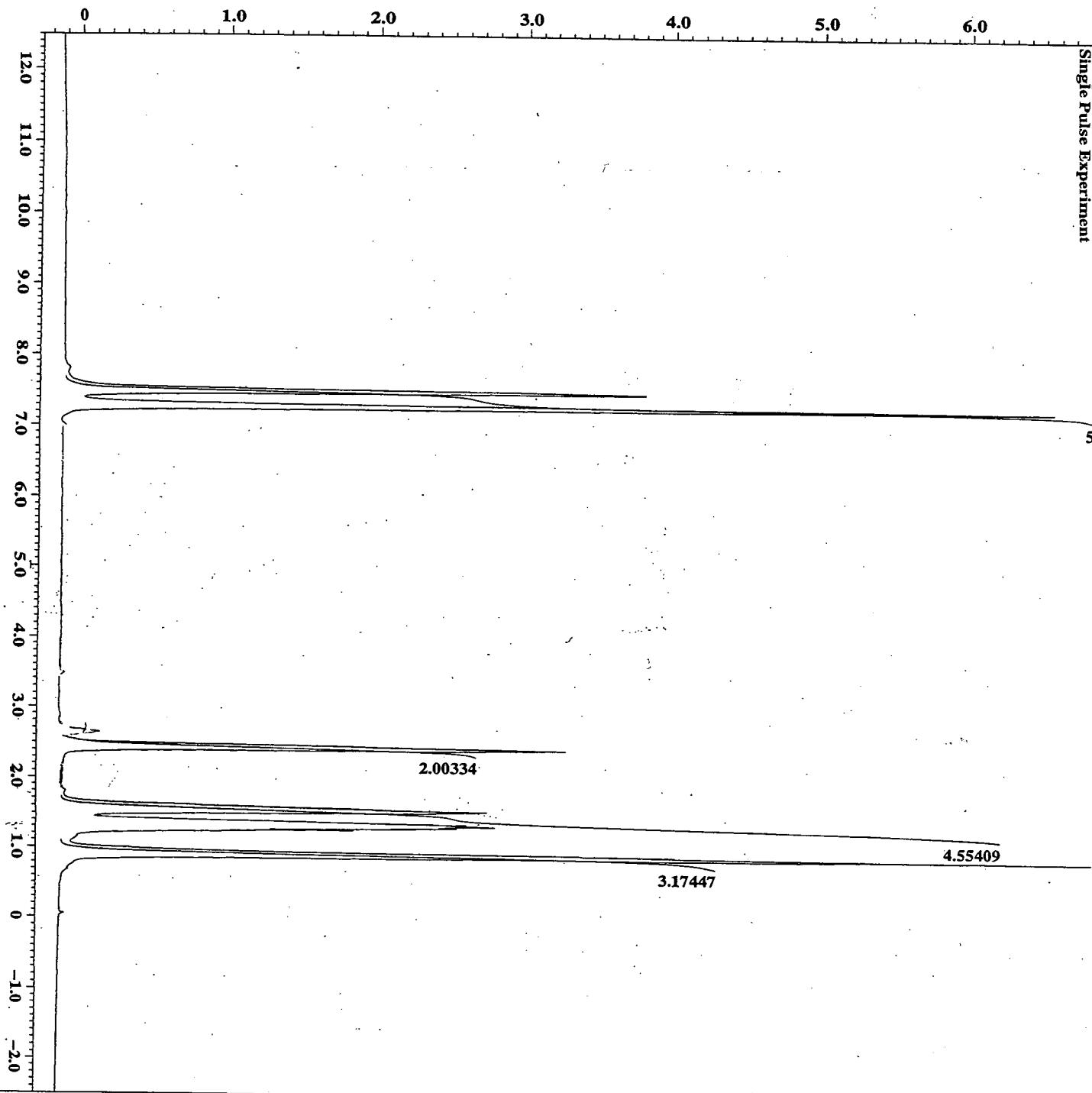
Misc Info :

Vial Number: 1

Ph-C≡C-Se-Ph

1d Spectrum.1955
Single Pulse Experiment

5



JEOL	
----- PROCESSING PARAMETERS -----	
dc_balance	0.2 [Hz]
seed	0.2 [Hz]
fft	1 : TRUE
machinephase	
ppm	
m/z	237.20
----- ACQUISITION PARAMETERS -----	
File Name	1d_Spectrum.1955
Author	
Sample ID	S155273
Content	Single Pulse Exper
Creation Date	21-JAN-2004 02:23:
Revision date	21-JAN-2004 01:37:
Spec Site	WCU GSX-270
Spec Type	DELT A_NMR
Data Format	1D COMPLEX
Dimensions	X
Dim Title	1H
Dim Size	16384
Dim Units	[ppm]
Scans	8
Mod return	1
X_Points	16384
X_Precs	0
X_domain	1H
X_Offset	5 [ppm]
X_Freq	270.16608844 [MHz]
X_Sweep	4.053350638 [kHz]
X_Resolution	0.24740659 [Hz]
X_Acq_duration	4.019321 [s]
Digital_Filter	FALSE
Filter_Factor	1 [s]
Delay_of_start	
Actual_start_time	21-JAN-2004 02:22:
Acq_delay	0.244 [ms]
X90	11.1 [us]
Ir90	11.1 [us]
Tr190	10 [us]
Qu90	10 [us]
X90_hi	18 [us]
Ir90_hi	18 [us]
Tr190_hi	10 [us]
Qu90_hi	10 [us]
Spin_lock	10 [us]
Spin_lock_90	10 [us]
Spin_lock_180	10 [us]
Spinsynchro	10 [us]
Spinsynchro_90	10 [us]
Spinsynchro_180	10 [us]
Spinsynchro_270	10 [us]
Spinsynchro_360	10 [us]
Spinsynchro_450	10 [us]
Spinsynchro_540	10 [us]
Spinsynchro_630	10 [us]
Spinsynchro_720	10 [us]
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Spinsynchro_990	10 [us]
Spinsynchro_1080	10 [us]
Spinsynchro_1170	10 [us]
Spinsynchro_1260	10 [us]
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Spinsynchro_1440	10 [us]
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Spinsynchro_24090	10 [us]
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Spinsynchro_33180	10 [us]
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Spinsynchro_33450	10 [us]
Spinsynchro_33540	10 [us]
Spinsynchro_33630	10 [us]
Spinsynchro_33720	10 [us]
Spinsynchro_33810	10 [us]
Spinsynchro_339	

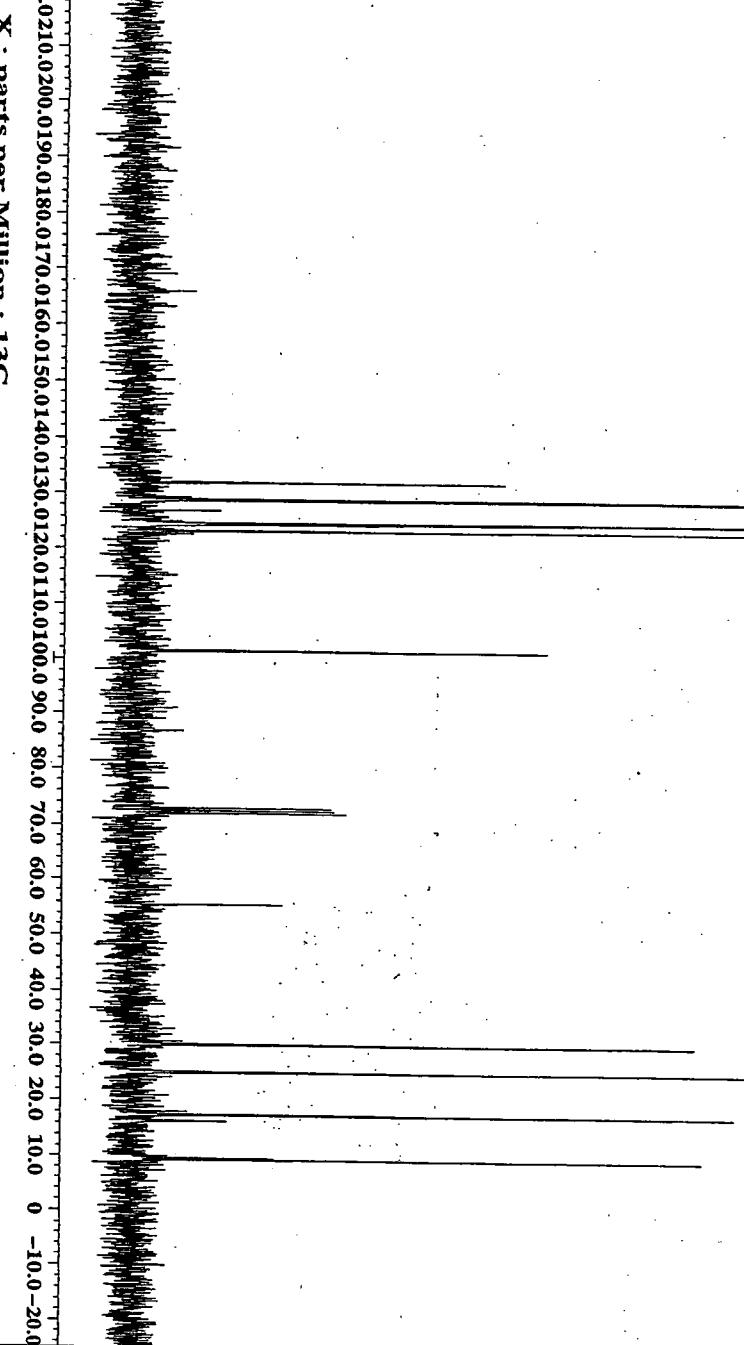
1d_13c_spectrum.1430
Single Pulse with Broadband Decoupling

JEOL

----- PROCESSING PARAMETERS -----
dc_balance
sep0 : 2[Hz]
fft : 1 : TRUE
machinephase
ppm

536

----- ACQUISITION PARAMETERS -----
File Name = 1d_13c_spectrum.14
Author = S#528018
Sample ID = Single Pulse with
Content = 20-JAN-2004 15:32:
Creation Date = 20-JAN-2004 14:43:
Revision Date = WKU GSX-270
Spec Site =
Spec Type = DELTA_NMR
Data Format = 1D COMPLEX
Dimensions = X
Dim Title = 13C
Dim Size = 32768
Dim Units = [ppm]
Scans = 18
Mod. return = 1
X_points = 32768
X_prescans = 4
X_domain = 13C
X_offset = 100[ppm]
X_freq = 67.93330993[MHz]
X_sweep = 17.00680272[MHz]
X_resolution = 0.51900643[Hz]
Irr_domain = 1H
Irr_offset = 5[ppm]
Irr_freq = 20.16608844[MHz]
X_acq_duration = 1.9267584[s]
Digital_filter = PULSE
Filter_factor = 1
Delay_of_start = 1[s]
Actual_start_time = 20-JAN-2004 15:30:
Acc_delay = 57.4[us]
X90 = 8.5[us]
IRx90 = 11[us]
TRx90 = 10[us]
Qu90 = 10[us]
X90_hi = 8.9[us]
IRx90_hi = 18[us]
TRx90_hi = 10[us]
Qu90_hi = 10[us]
X90_lo = 39[us]
IRx90_lo = 55[us]
TRx90_lo = 10[us]
Qu90_lo = 10[us]
Spin_lock_90 = 0.1[ms]
Spin_lock_attn = 17.5[db]
Daut_grad_shim_90 = 0.105[ms]
Daut_grad_sim_90 = 10[dB]
Field_card = 16/IM22/20
Field_strength = 6.345446[T]
Filter_mode = BUTTERWORTH
Filter_width = 8.50340136[MHz]



X : parts per Million : 13C

