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

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Supporting Information

Table of Contents: ^{1}H 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

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%).

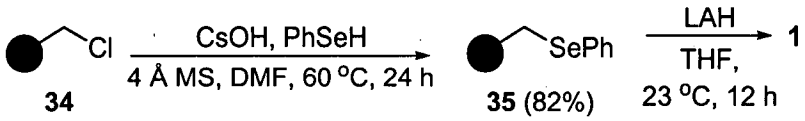
General Procedure for the Alkynylation of 1-Hexyne (32) with Phenylselenyl Bromide Using CsOH.

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



34 **35** (82%)

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:

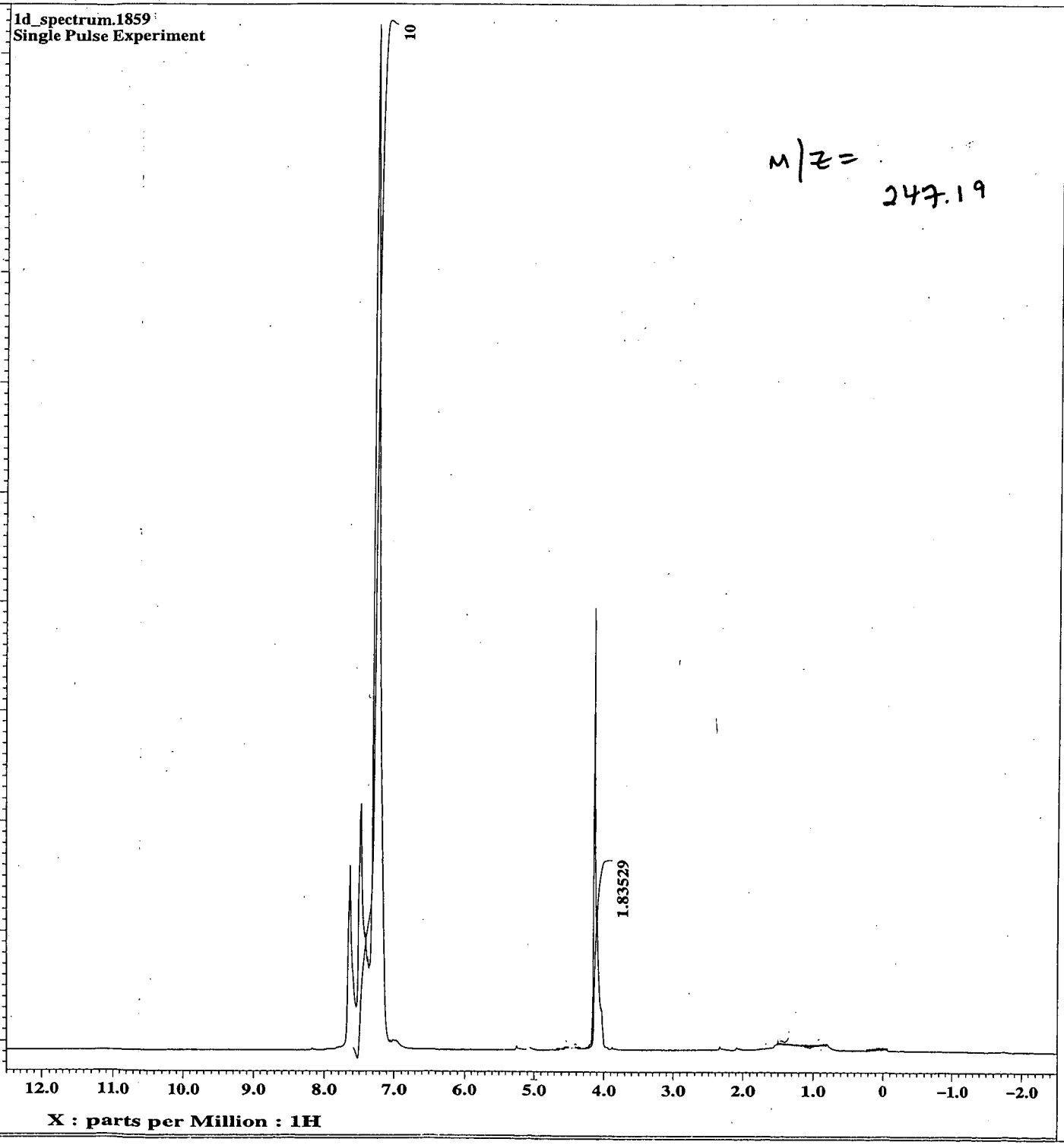
1.2 g of selenide resin **35** – 1.0 g of Merrifield'ds resin = 0.20 g mass loaded
0.20 g = (157.08) (mol of selenol added) – 35.45 g/mol (mol Cl^-)
Assuming that mol of selenol loaded = mol of Cl^- displaced.
0.20 = 157.08 (x) – 35.45 g/mol) (x)
0.20 mmol = 121.63 (x)
1.644 mmol = x
1.644 mmol of selenol loaded/1.0 g of resin = 1.644 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 mmol loaded / 2 mmol) x 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 (^{1}H 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.



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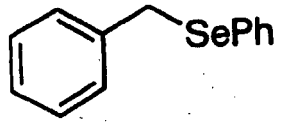

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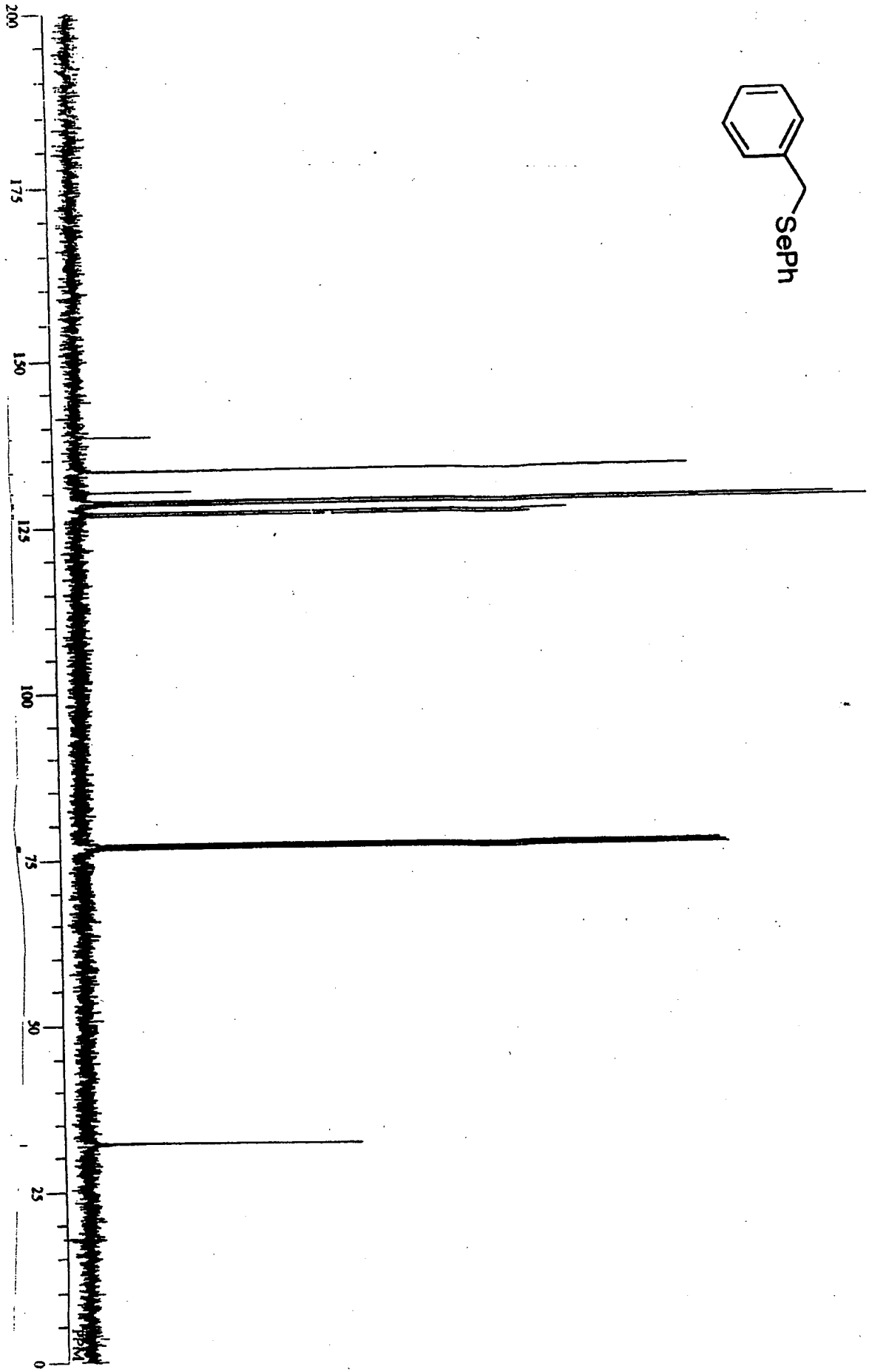
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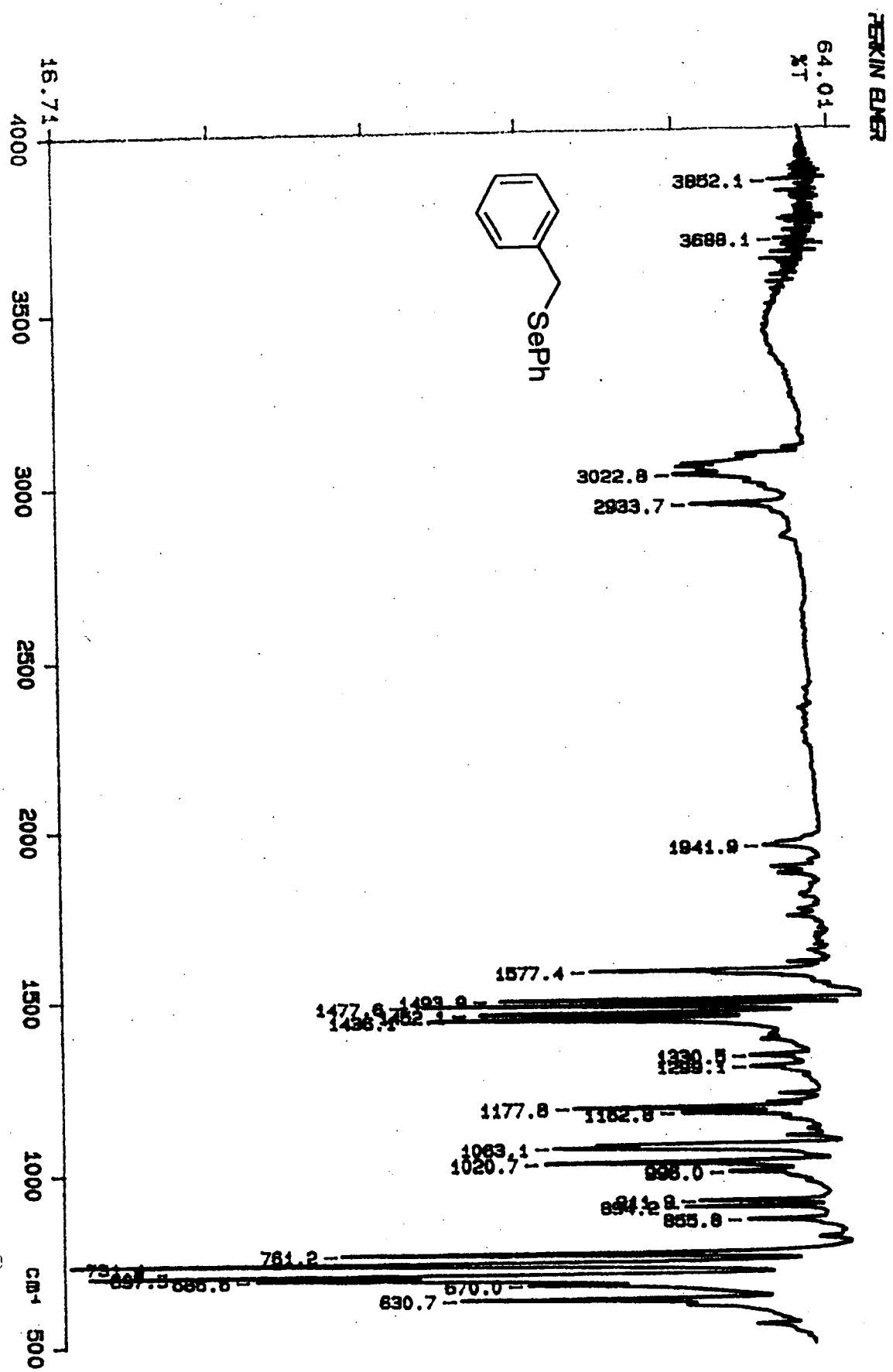

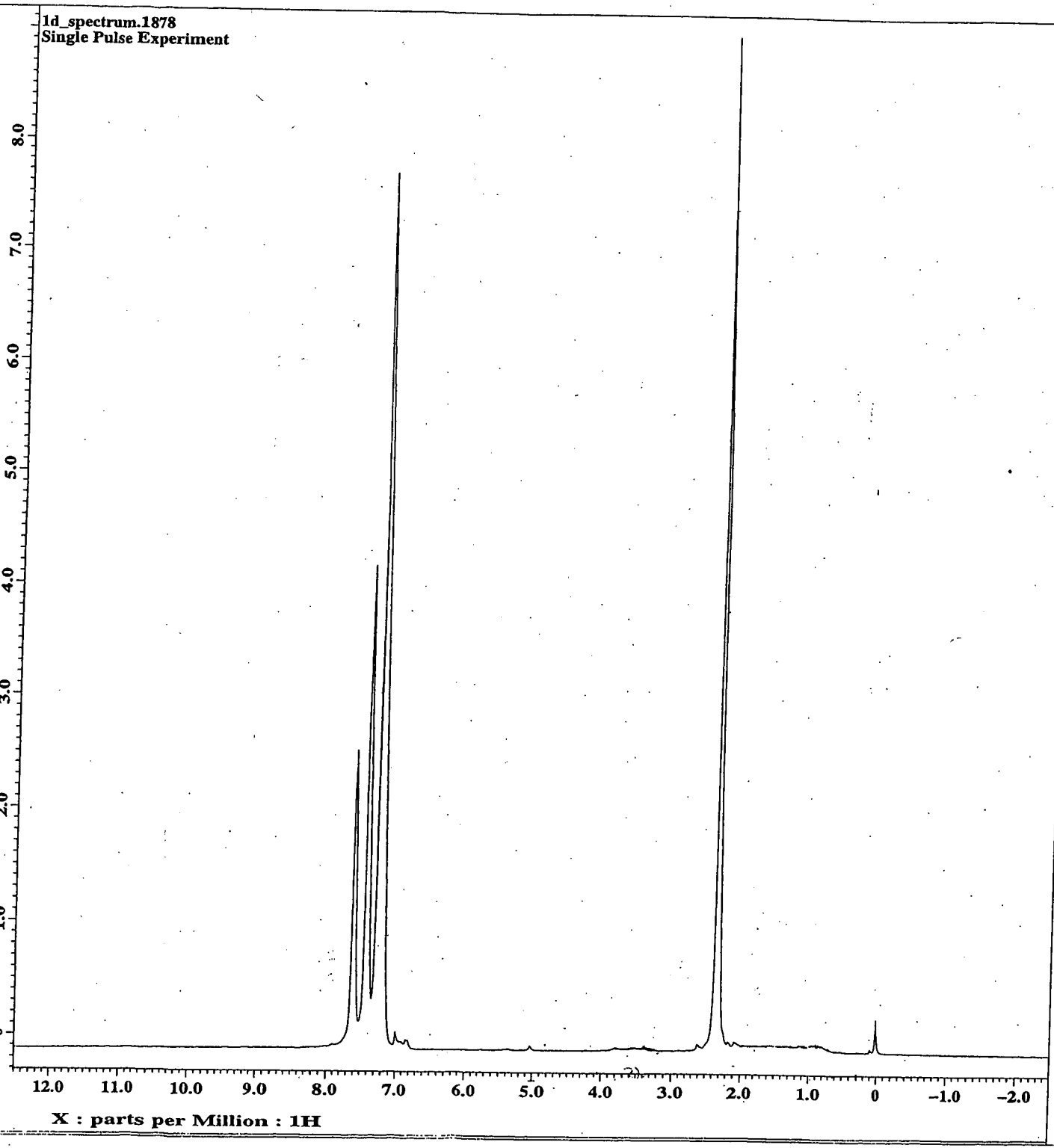

JEOL

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Pinene

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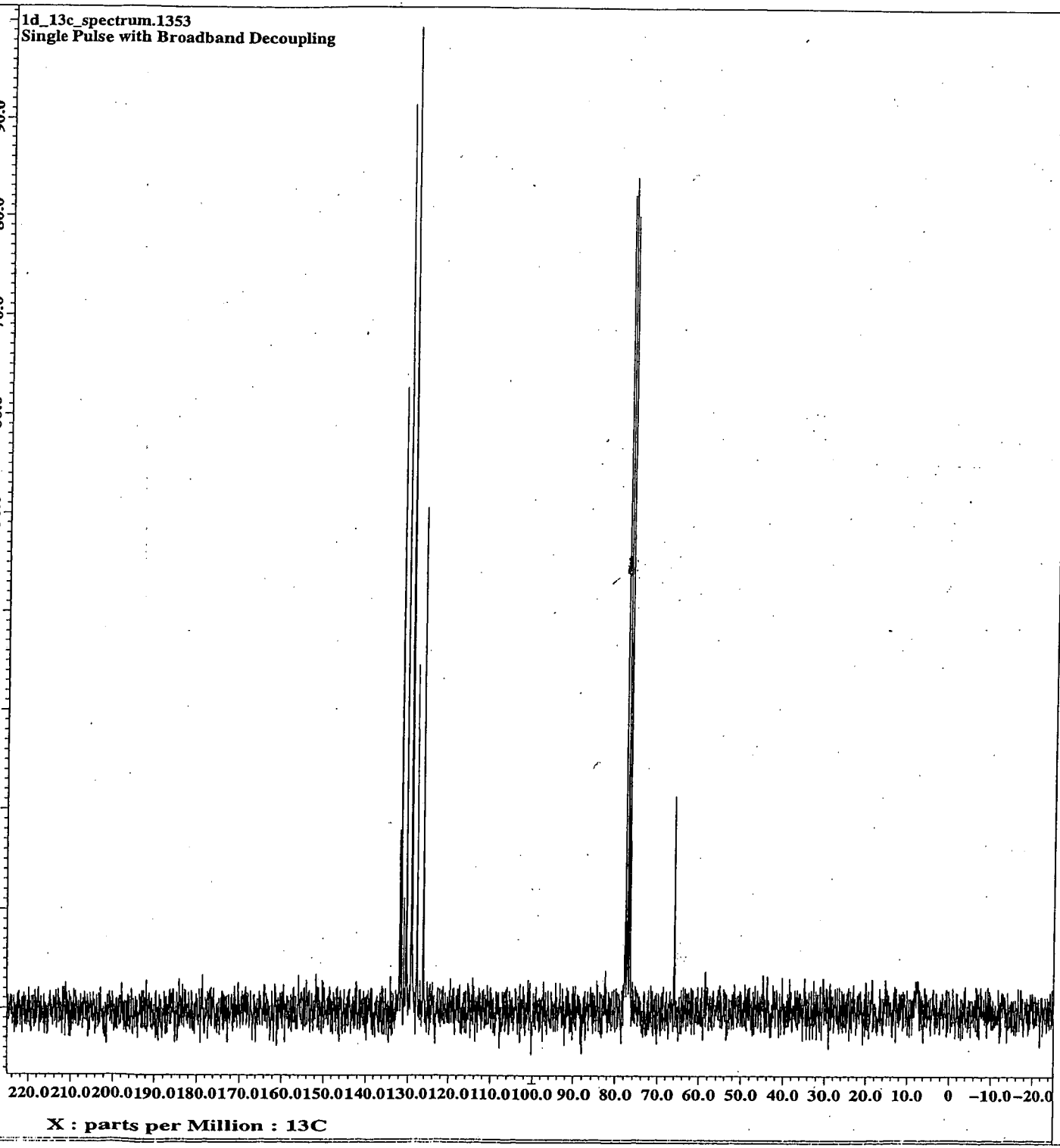

JEOL

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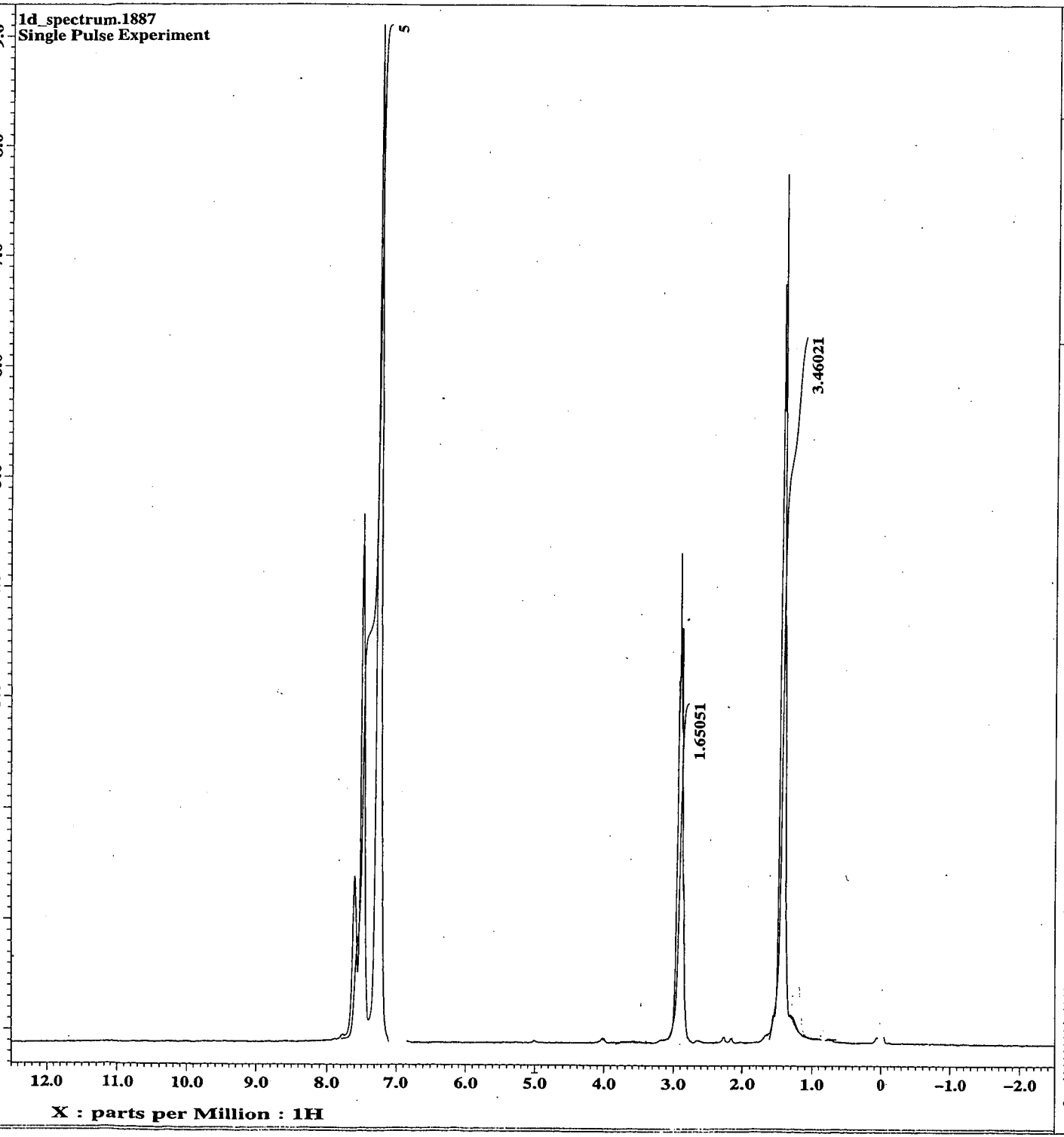
59



JEOL

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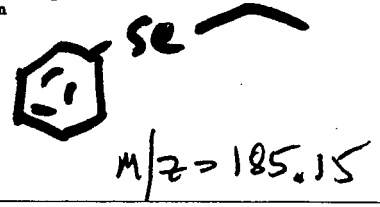

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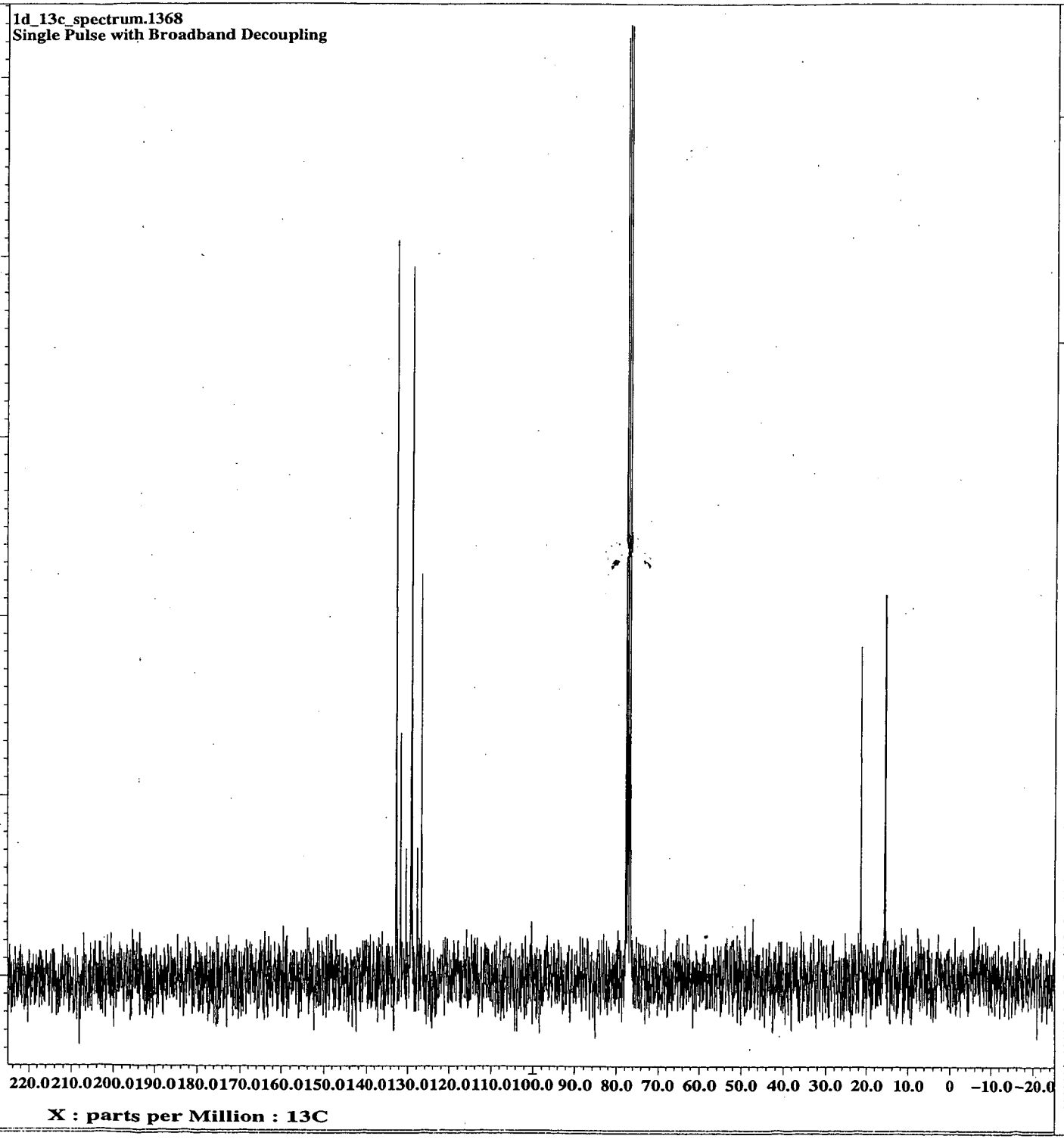

JEOL

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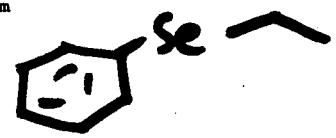

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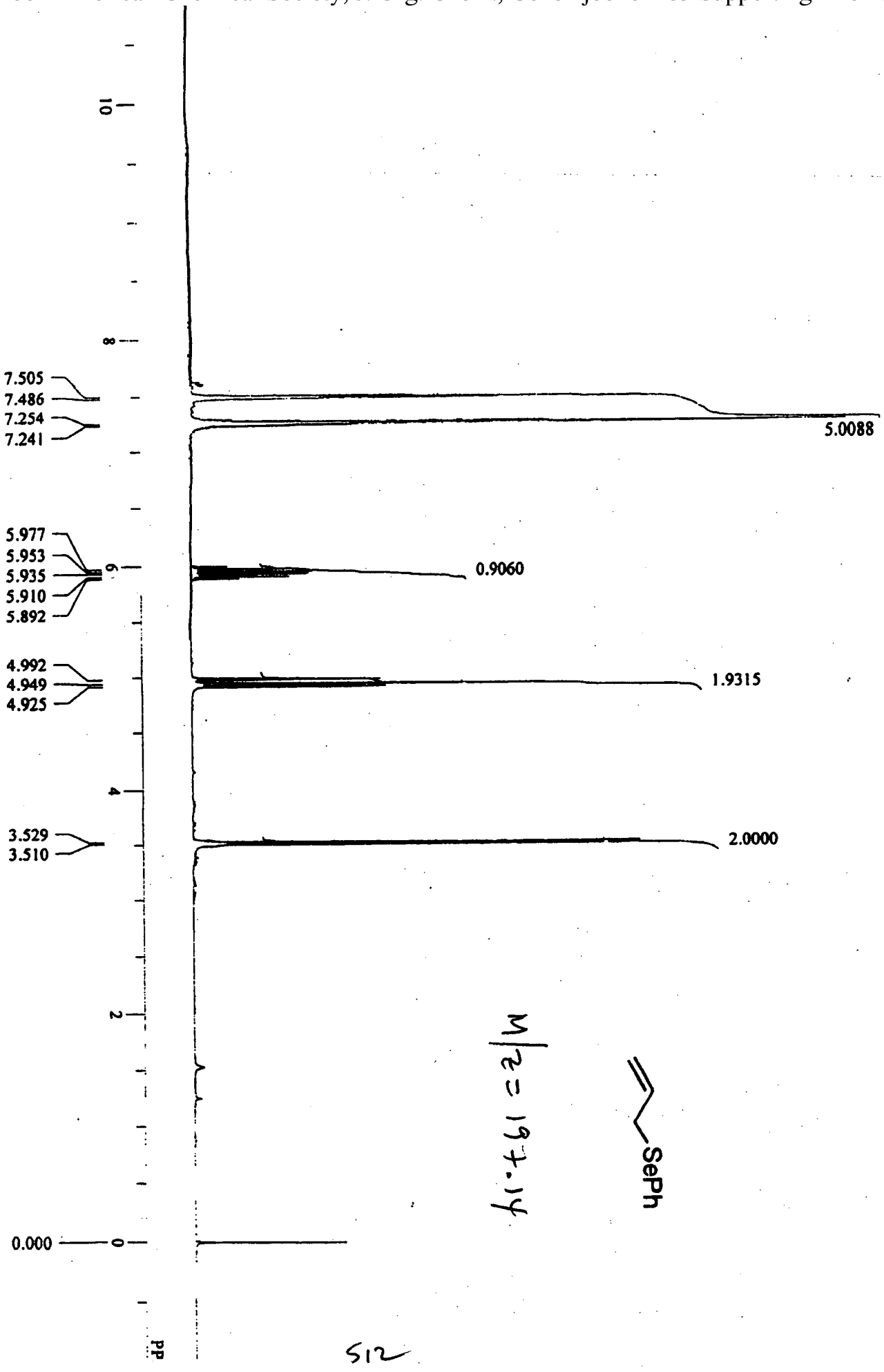

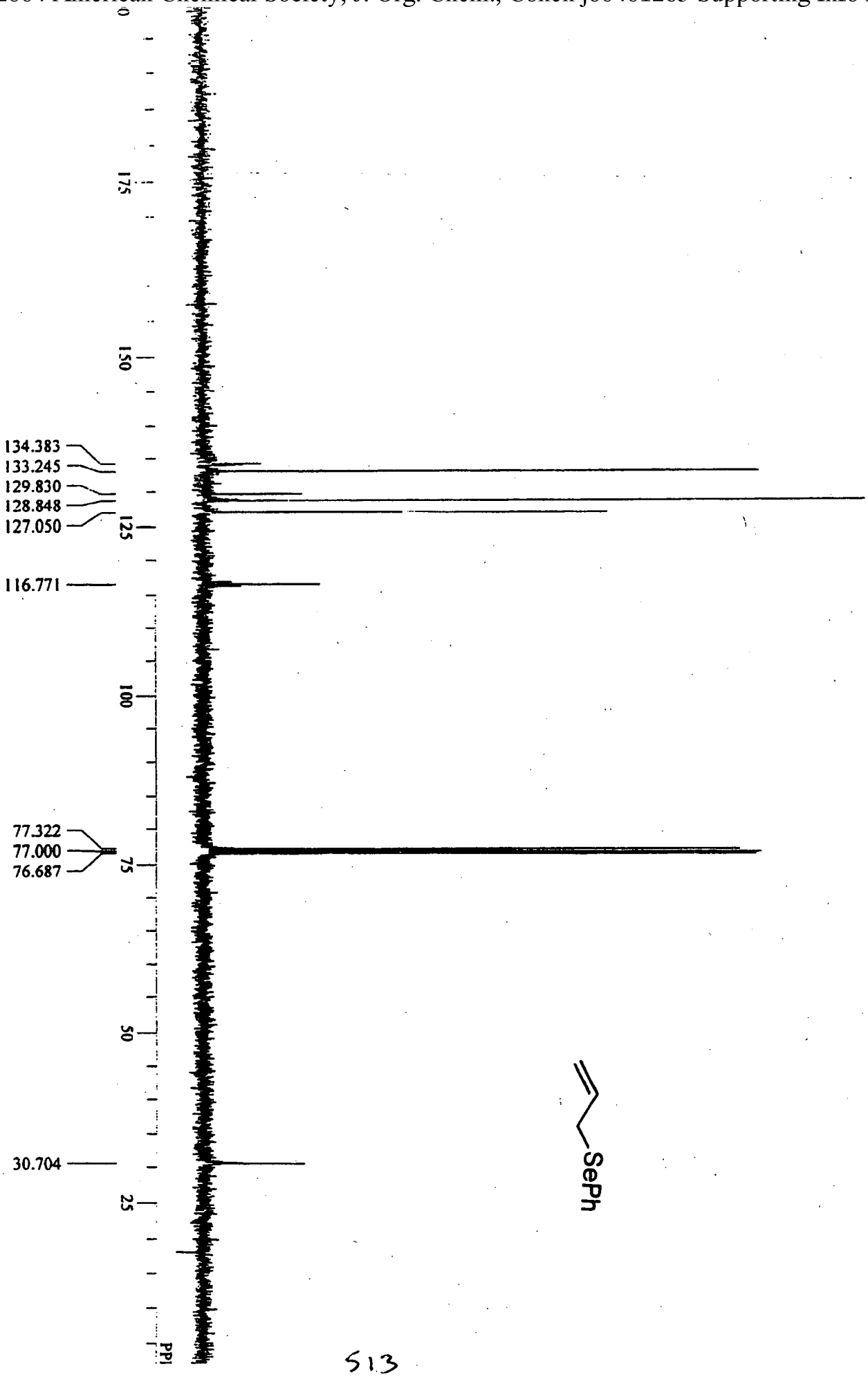

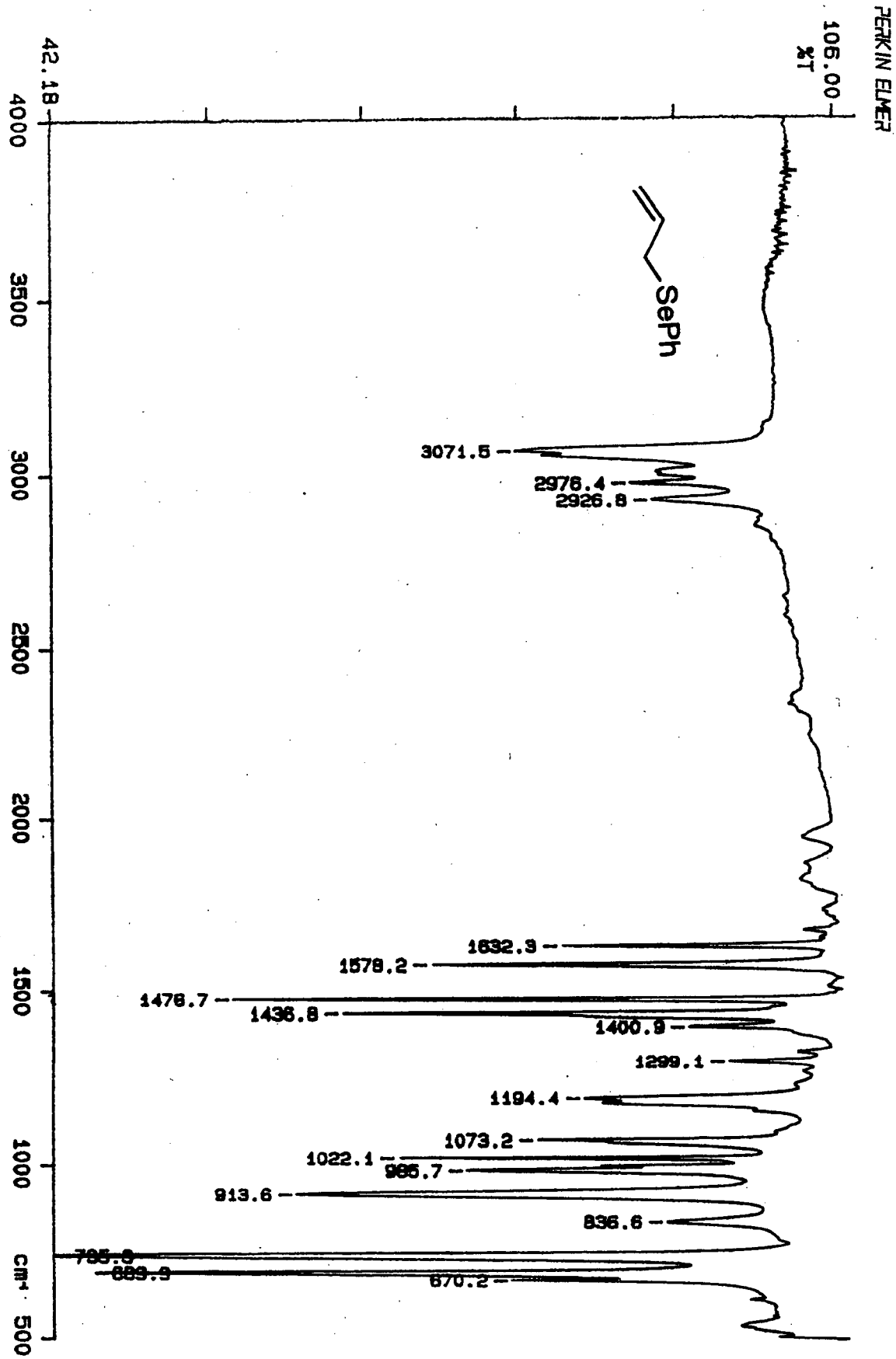
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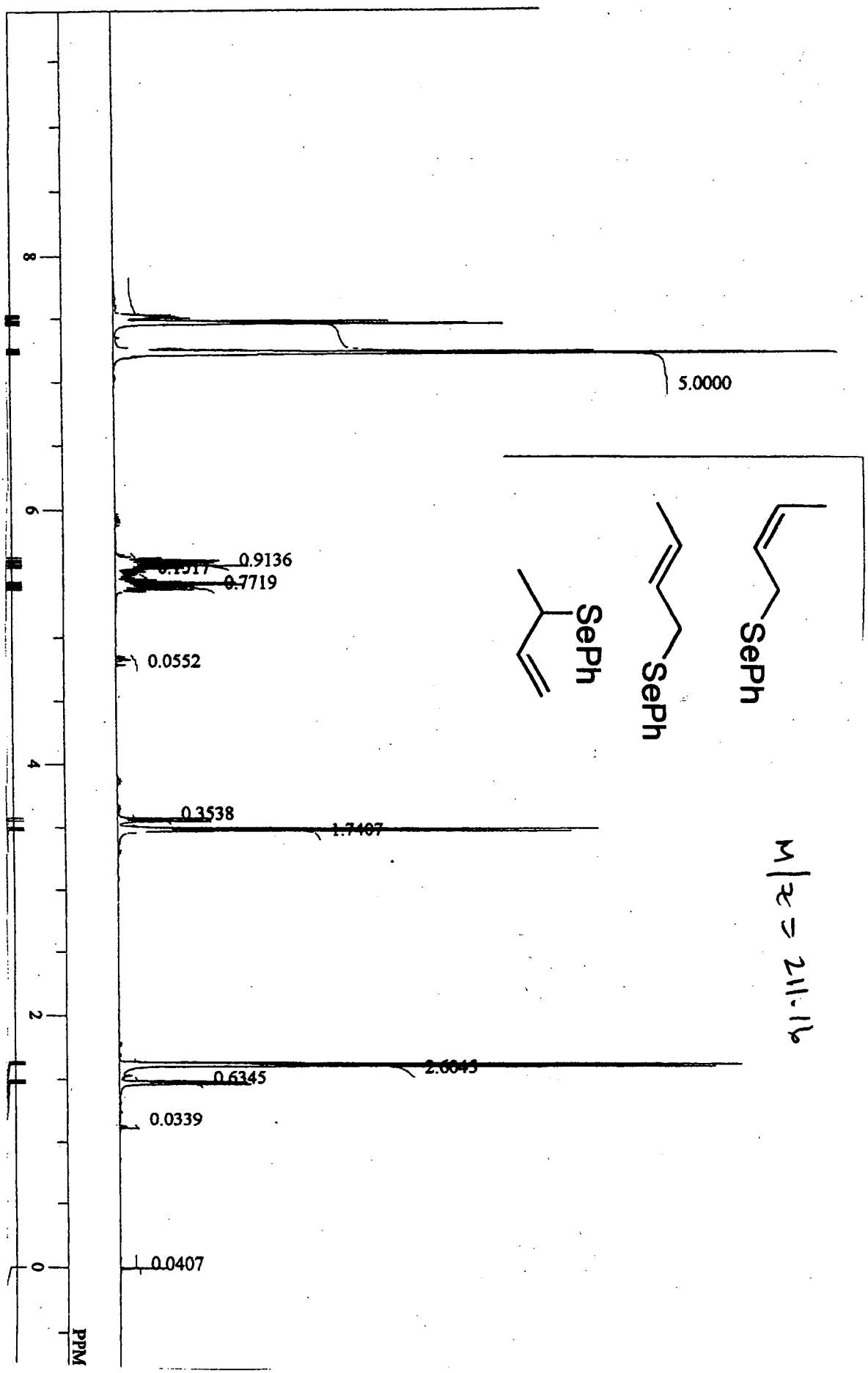
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SePh
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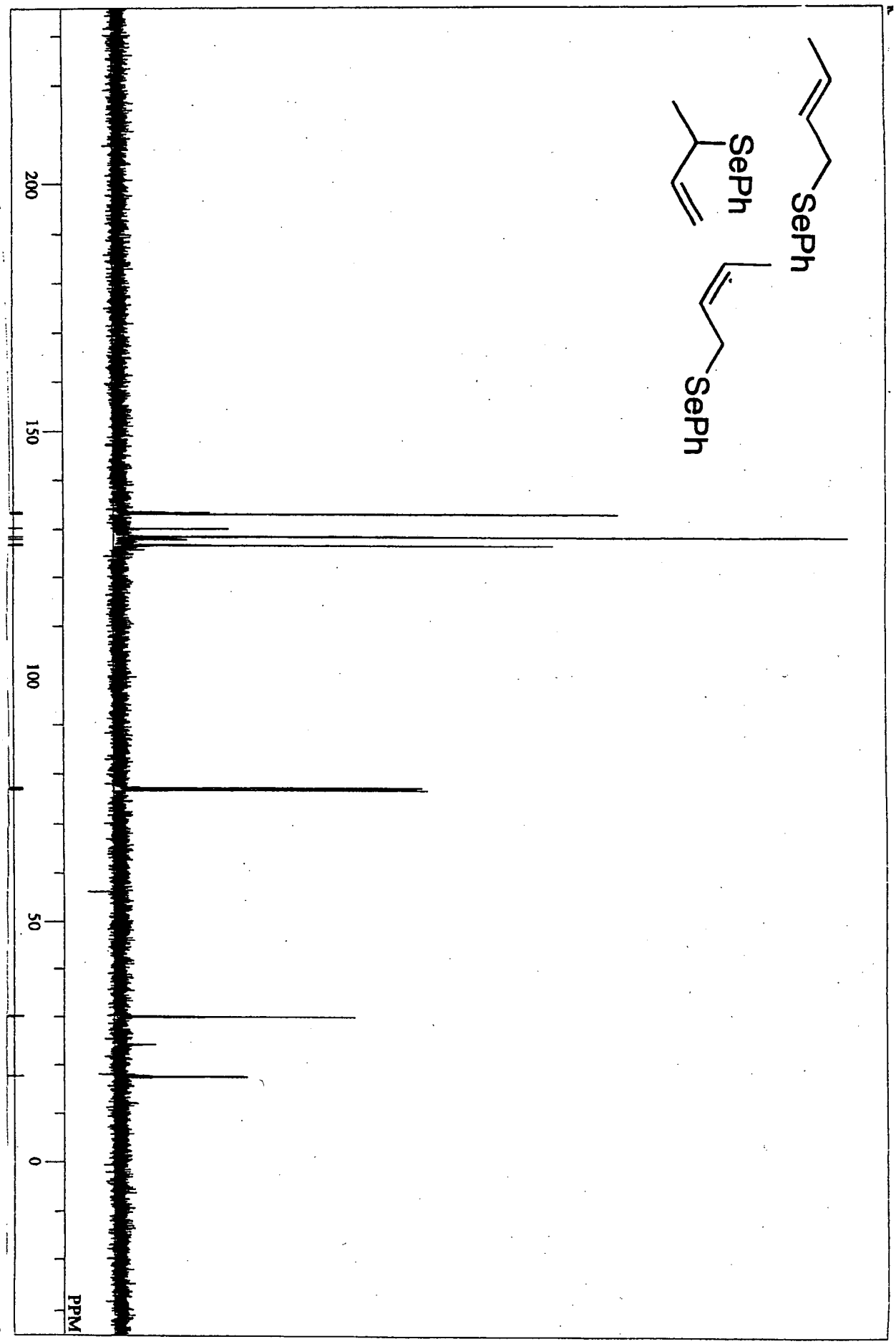
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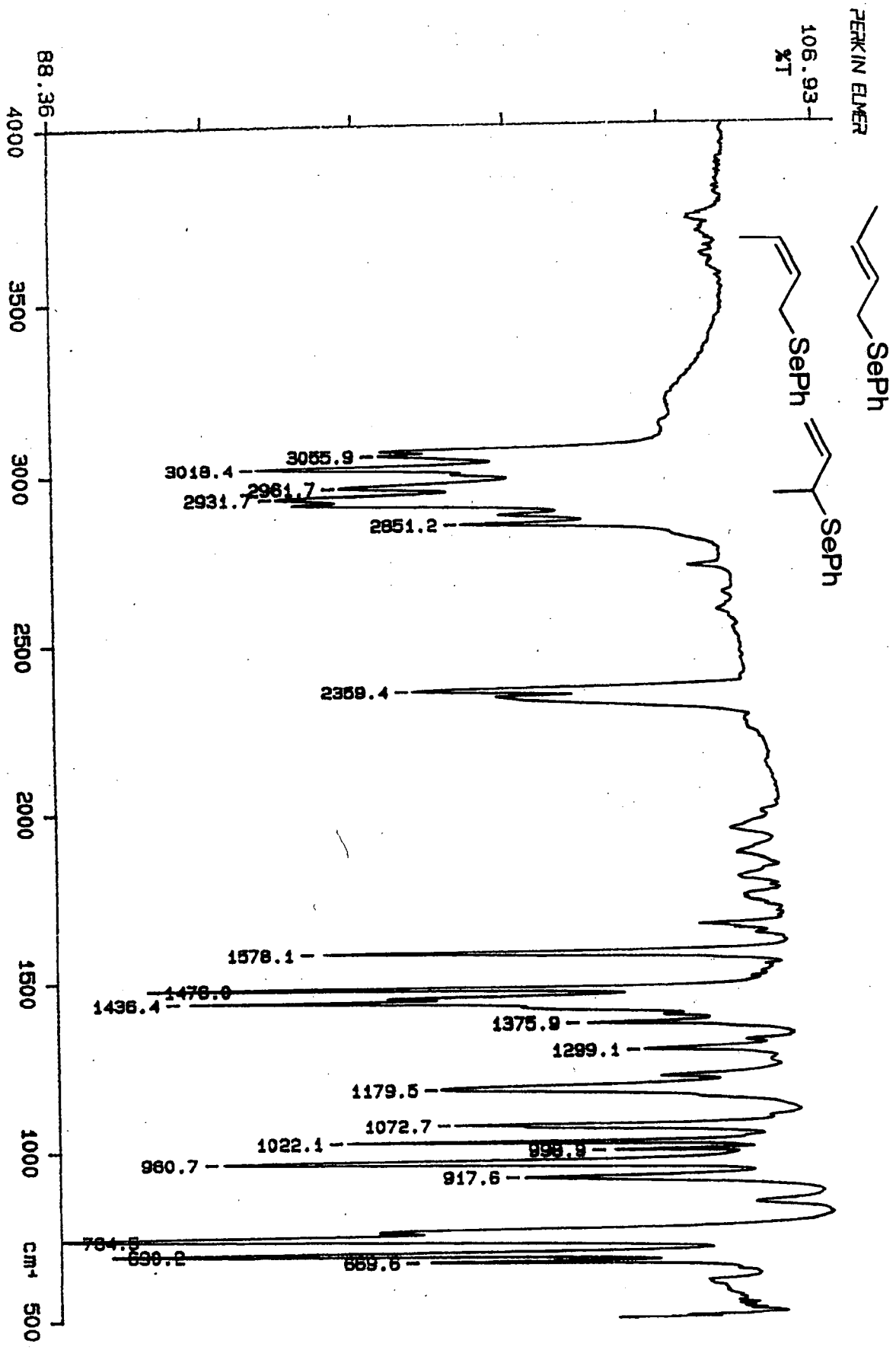

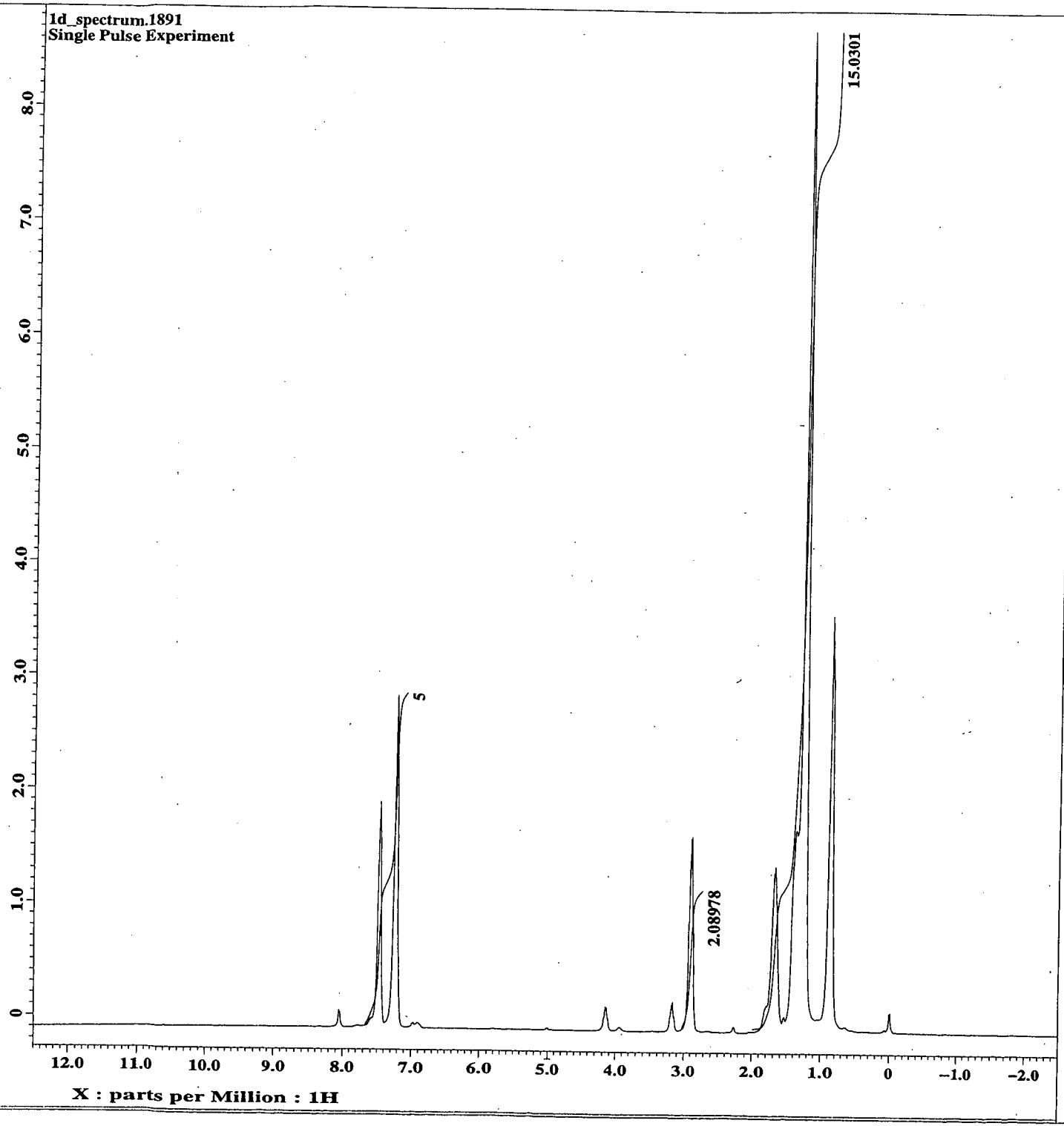

JEOL

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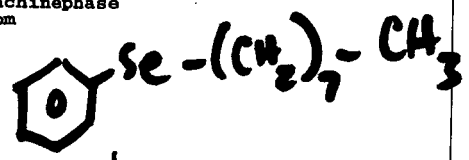
ppm



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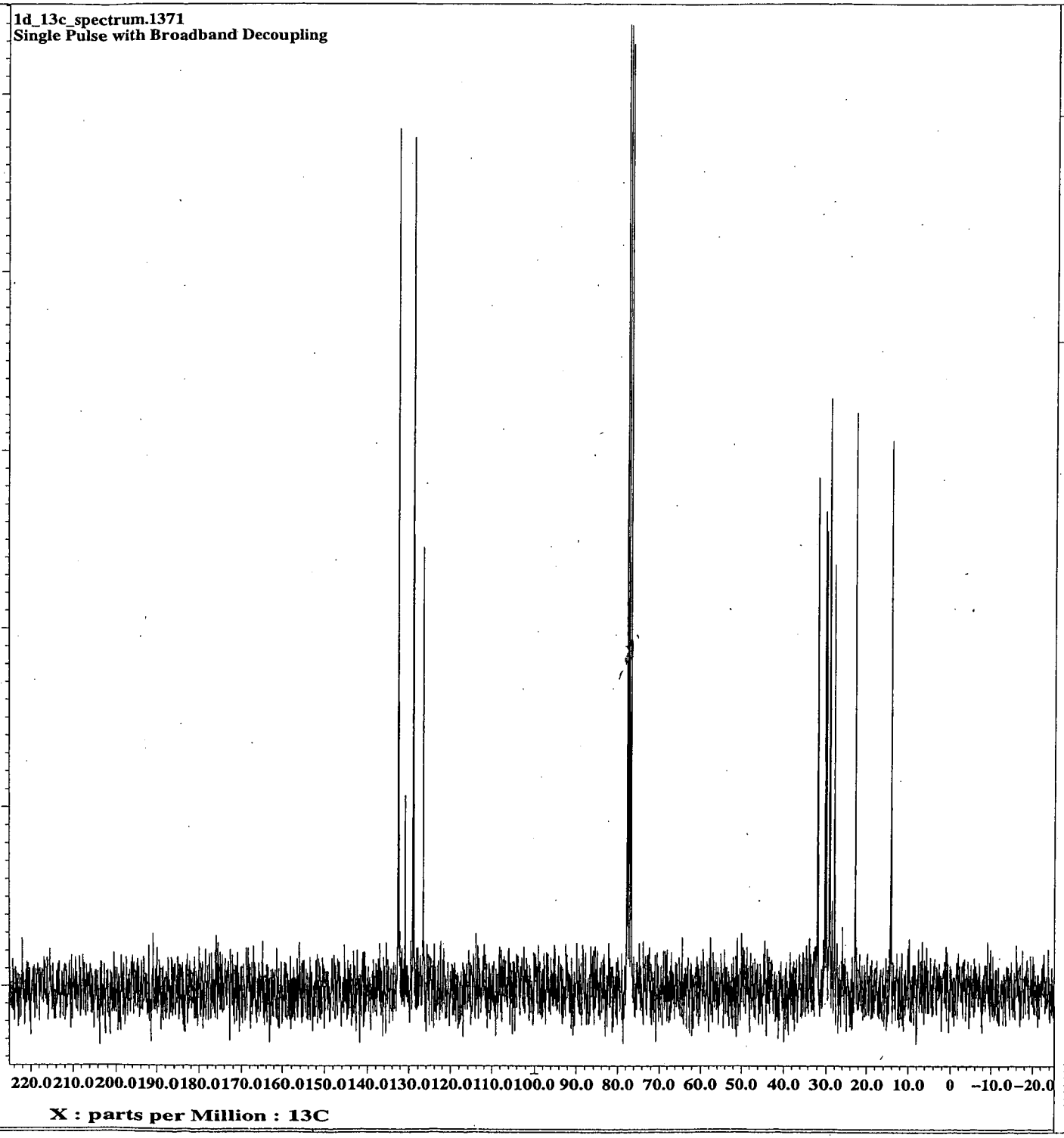
X_acq_duration	= 4.0419328[s]
Digital_filter	= FALSE
Filter_factor	= 1
Delay_of_start	= 1[s]
Actual_start_time	= 22-DEC-2003 02:08:
Acq_delay	= 0.244[ms]
X90	= 11[us]
Irr90	= 11[us]
Tri90	= 10[us]
Qua90	= 10[us]
X90_hi	= 18[us]
Irr90_hi	= 18[us]
Tri90_hi	= 10[us]
Qua90_hi	= 10[us]
X90_lo	= 55[us]
Irr90_lo	= 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]
Deut_grad_shim_attn	= 10[dB]
Adc_card	= 16/1MHz/20
Field_strength	= 6.345446[T]
Filter_mode	= BUTTERWORTH
Filter_width	= 2.02675314[kHz]
Recvr_gain	= 13
Irr_code	= 4
Obs_pwidth	= 1[us]

815



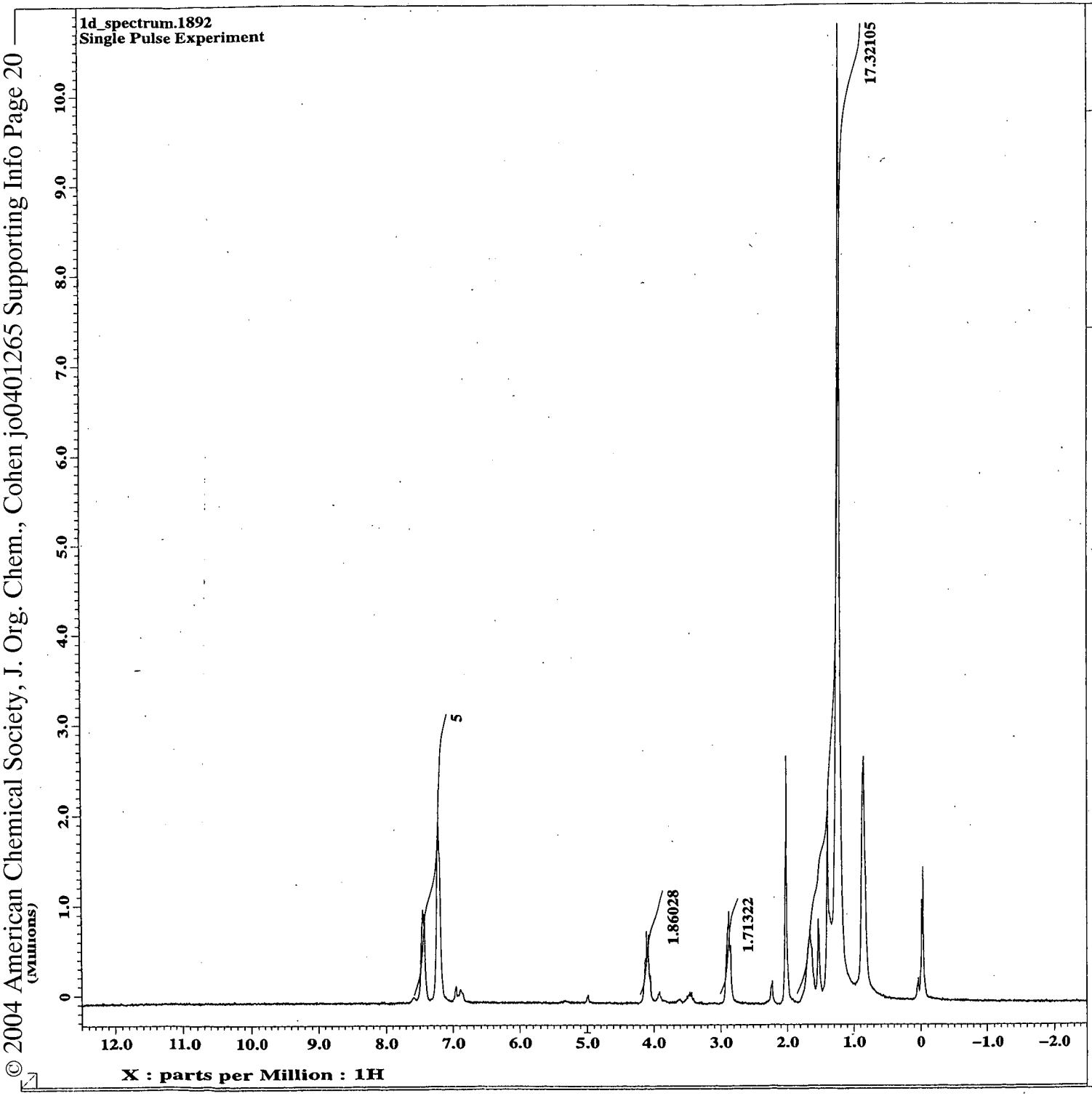
JEOL

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

Ph-se

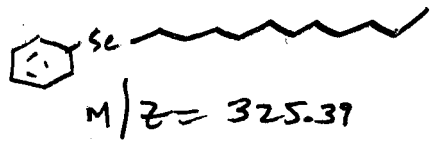
```
---- ACQUISITION PARAMETERS ----
File Name            = 1d_13c_spectrum.13
Author               =
Sample ID            = S#43262
Content              = Single Pulse with
Creation Date        = 22-DEC-2003 02:19:

Revision Date        = 22-DEC-2003 01:20:
Spec Site            = WKU GSX-270

Spec Type            = DELTA_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_prescans           = 4
X_domain             = 13C
X_offset             = 100[ppm]
X_freq               = 67.93330993[MHz]
X_sweep              = 17.00680272[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    = 22-DEC-2003 02:12:
Acq_delay            = 57.4[us]
X90                  = 8.5[us]
Irr90                = 11[us]
Tri90                = 10[us]
Qua90                = 10[us]
X90_hi               = 8.9[us]
Irr90_hi             = 18[us]
Tri90_hi             = 10[us]
Qua90_hi             = 10[us]
X90_lo               = 39[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]
Deut_grad_shim_attn  = 10[dB]
Adc_card             = 16/1MHz/20
Field_strength       = 6.345446[T]
Filter_mode          = BUTTERWORTH
Filter_width         = 8.50340136[kHz]
```

S19



JEOL

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

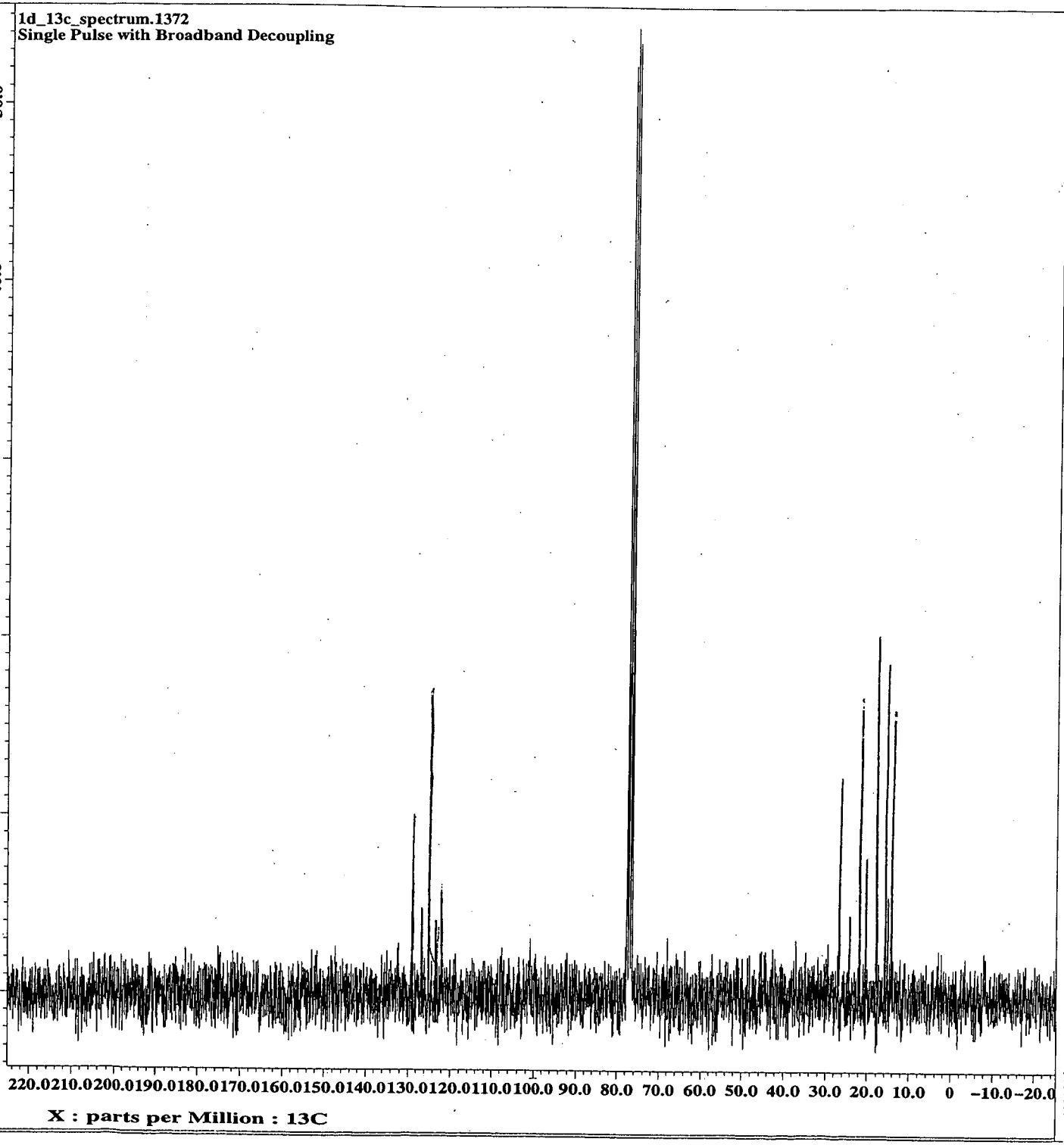


M/Z = 325.39

---- 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	= 270.16608844[MHz]
X_sweep	= 4.05350628[kHz]
X_resolution	= 0.24740639[Hz]
X_acq_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]
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]
Deut_grad_shim_attn	= 10[dB]
Adc_card	= 16/1MHz/20
Field_strength	= 6.345446[T]
Filter_mode	= BUTTERWORTH
Filter_width	= 2.02675314[kHz]
Recvr_gain	= 20
Irr_code	= 4
Obs_pwidth	= 1[us]

S20



JEOL

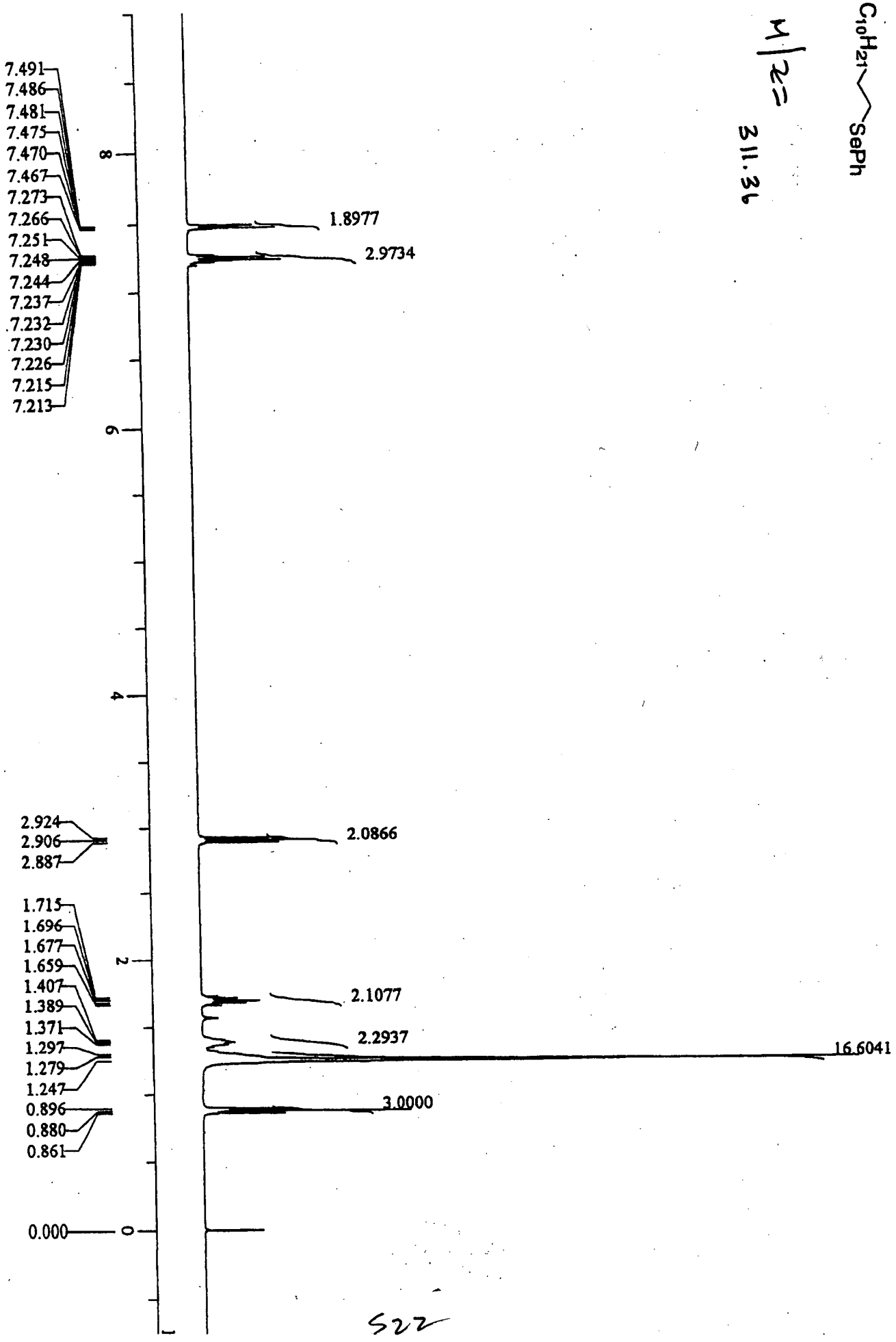
---- PROCESSING PARAMETERS ----
dc_balance
sexp : 2[Hz]
fft : 1 : TRUE
machinephase
ppm

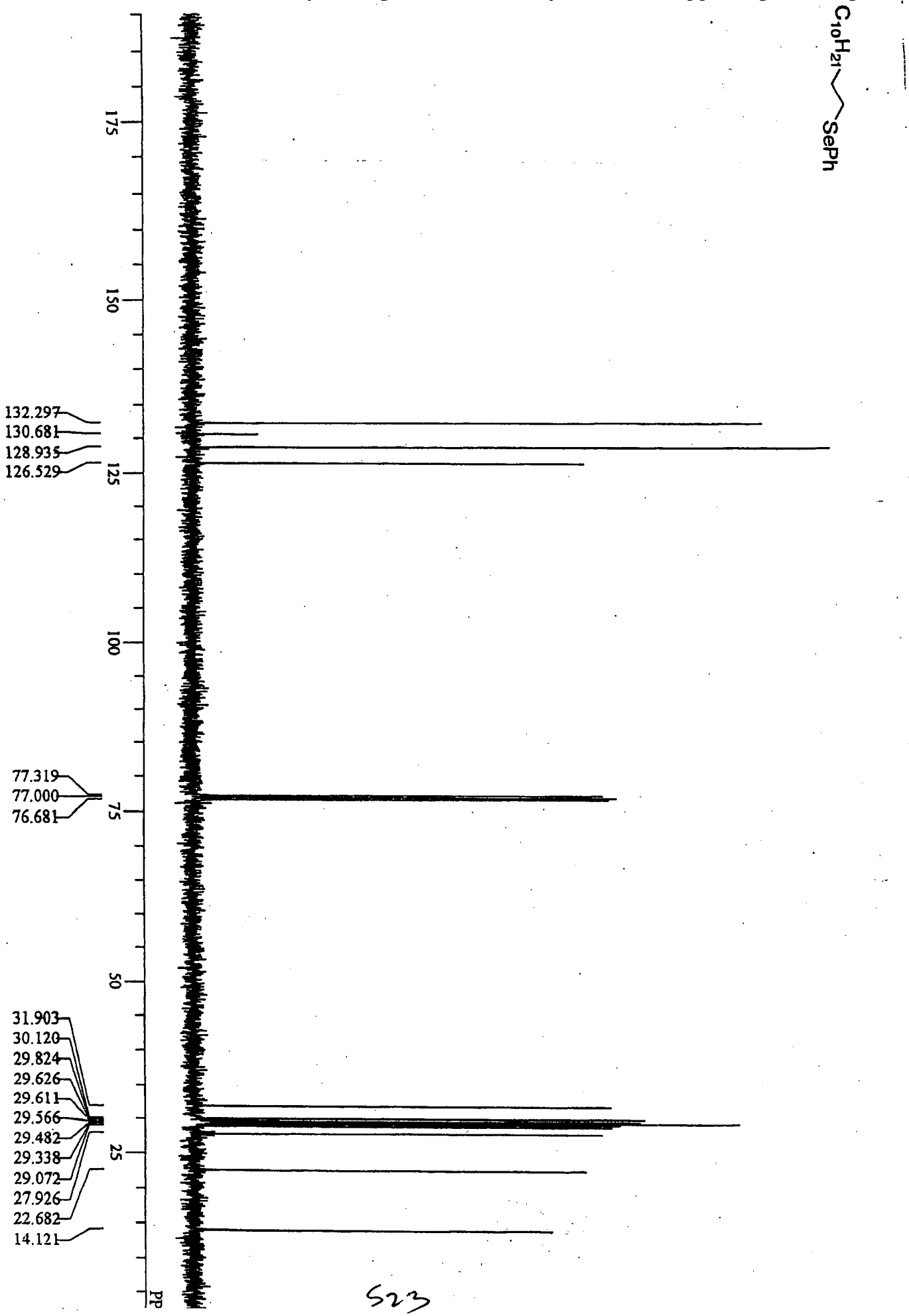
$Ph\text{-}Se\text{-}(CH_2)_{10}\text{-}CH_3$

---- ACQUISITION PARAMETERS ----

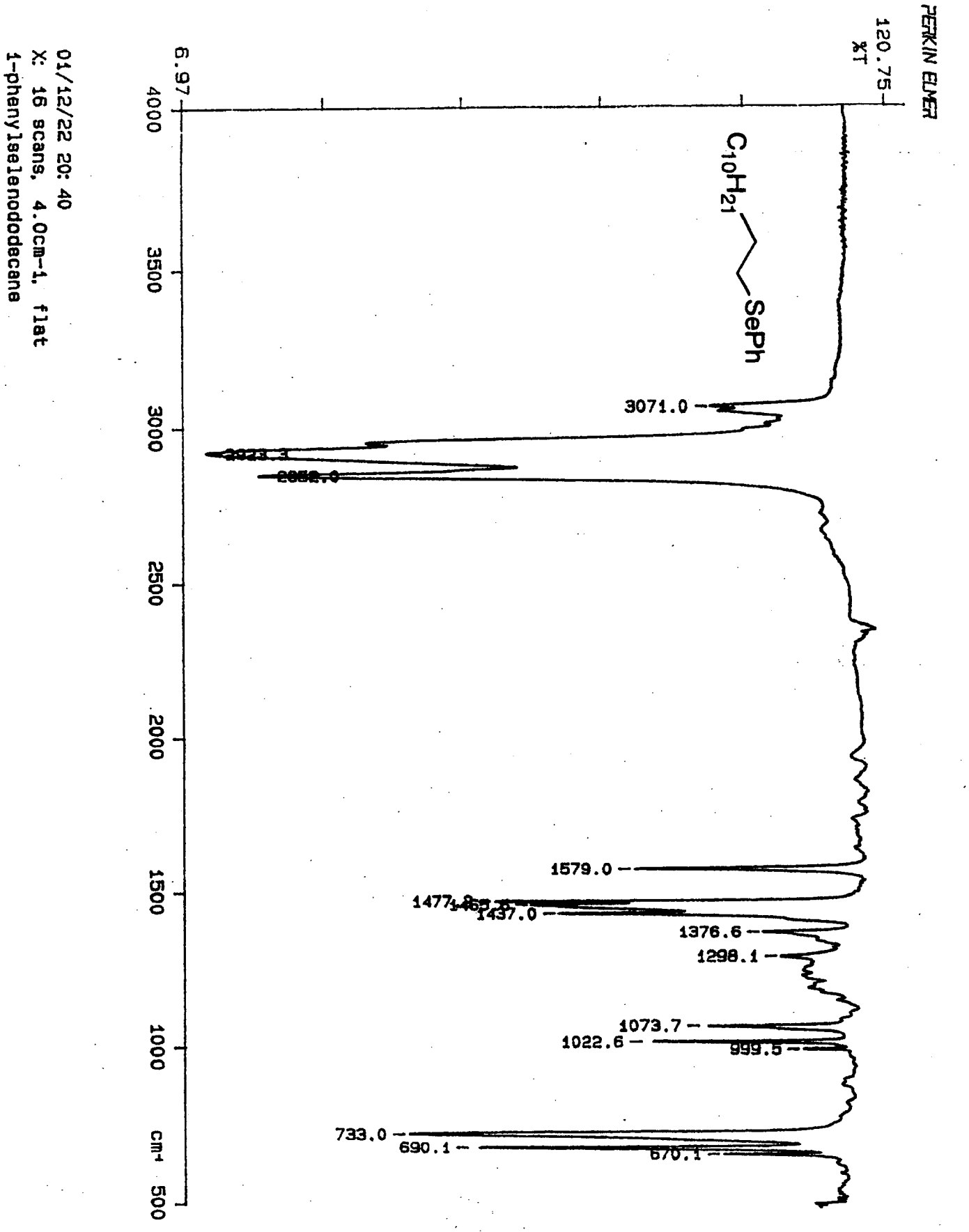
File Name	= 1d_13c_spectrum.13
Author	=
Sample ID	= S#66089
Content	= Single Pulse with
Creation Date	= 22-DEC-2003 02:58:
Revision Date	= 22-DEC-2003 01:59:
Spec Site	= WKU GSX-270
Spec Type	= DELTA_NMR
Data Format	= 1D COMPLEX
Dimensions	= X
Dim Title	= 13C
Dim Size	= 32768
Dim Units	= [ppm]
Scans	= 80
Mod_return	= 1
X_points	= 32768
X_prescans	= 4
X_domain	= 13C
X_offset	= 100[ppm]
X_freq	= 67.93330993[MHz]
X_sweep	= 17.00680272[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	= 22-DEC-2003 02:51:
Acq_delay	= 57.4[us]
X90	= 8.5[us]
Irr90	= 11[us]
Tri90	= 10[us]
Qua90	= 10[us]
X90_hi	= 8.9[us]
Irr90_hi	= 18[us]
Tri90_hi	= 10[us]
Qua90_hi	= 10[us]
X90_lo	= 39[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]
Deut_grad_shim_attn	= 10[dB]
Adc_card	= 16/1MHz/20
Field_strength	= 6.345446[T]
Filter_mode	= BUTTERWORTH
Filter_width	= 8.50340136[kHz]

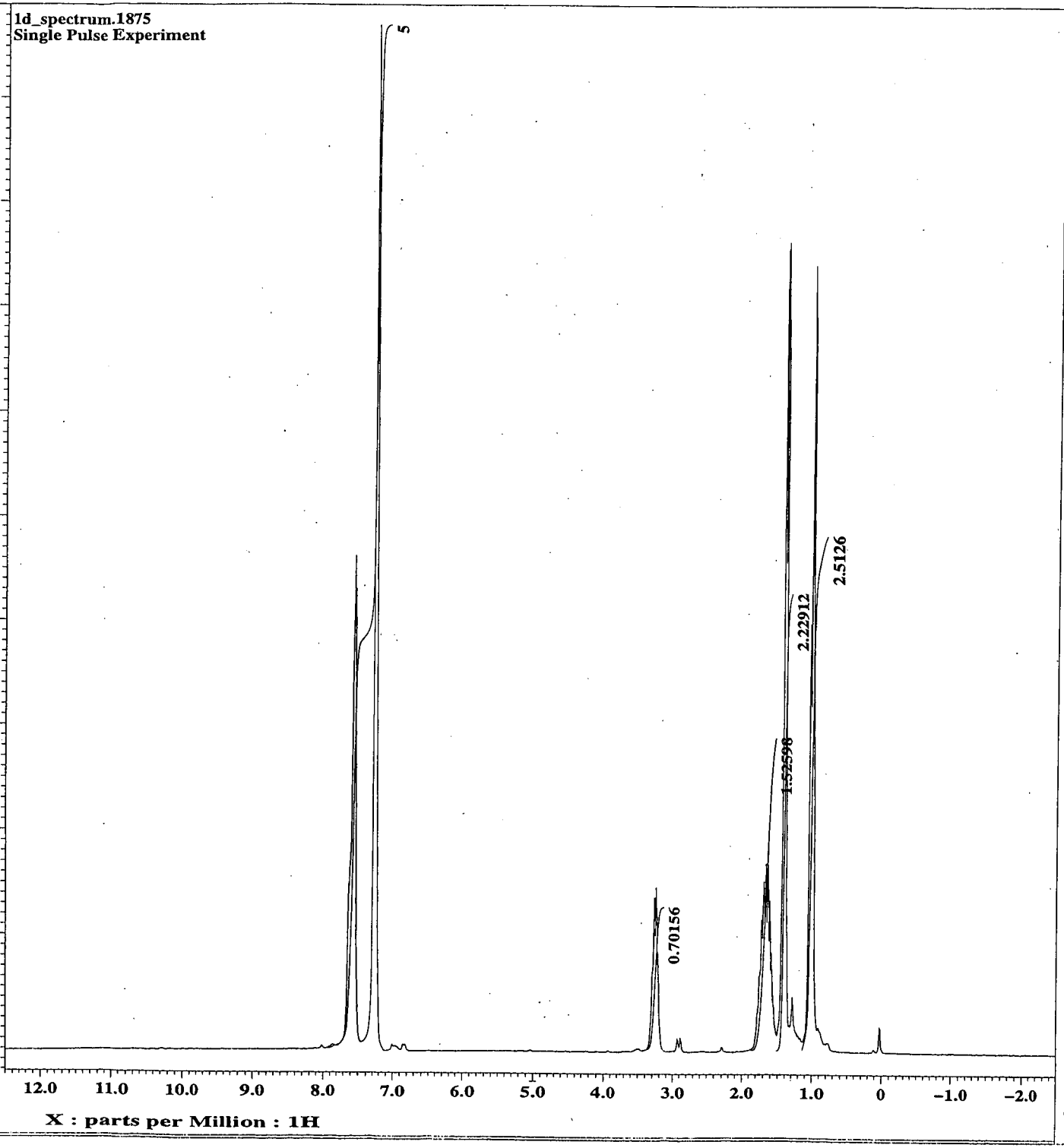
S21





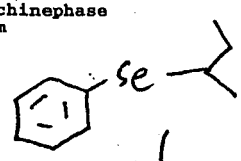
S23





JEOL

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

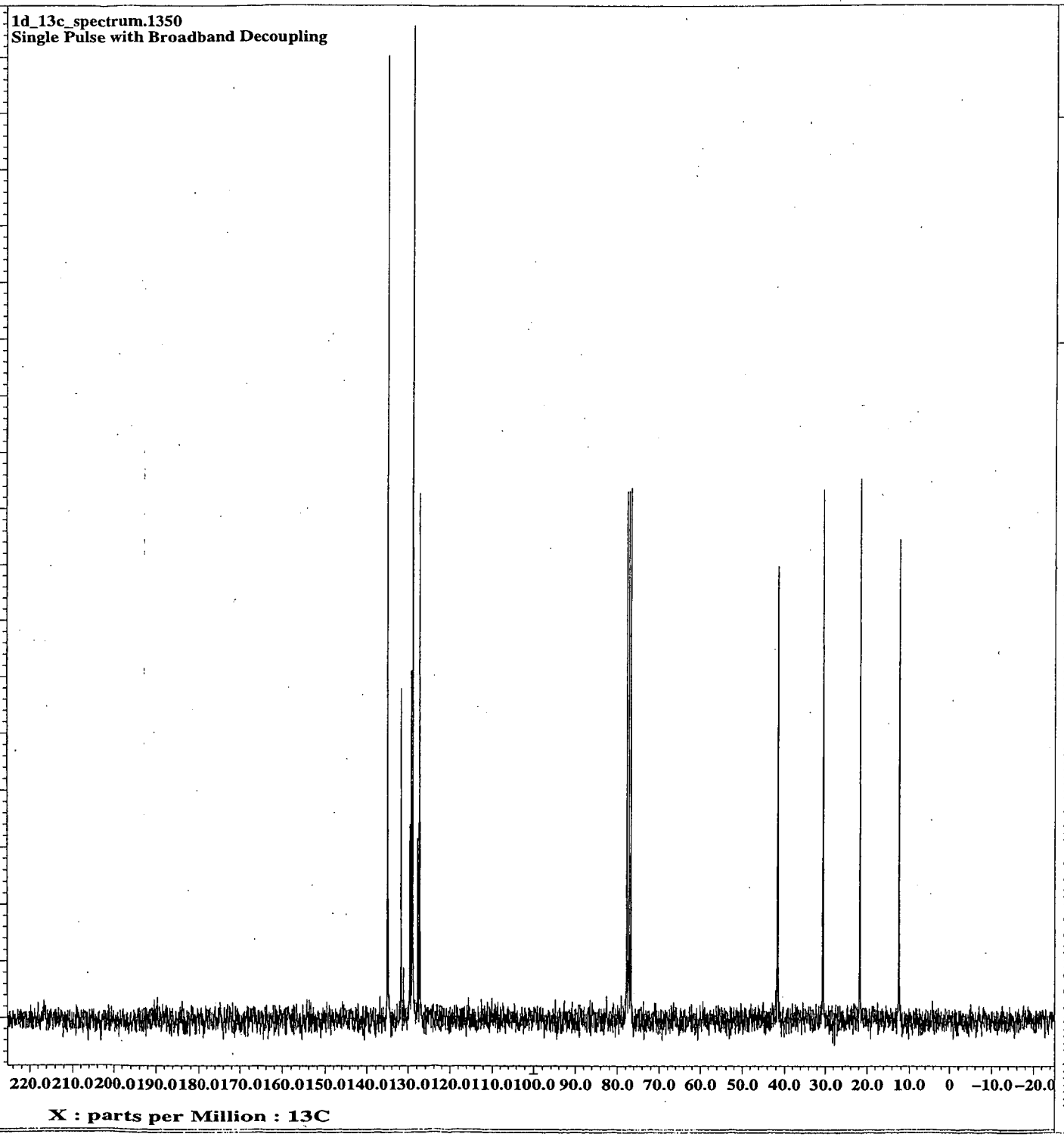


M/z = 229.22

---- ACQUISITION PARAMETERS ----

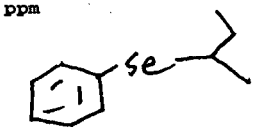
File Name	= 1d_spectrum.1875
Author	=
Sample ID	= S#245470
Content	= Single Pulse Exper
Creation Date	= 12-DEC-2003 07:52:
Revision Date	= 12-DEC-2003 07:10:
Spec Site	= WKU GSX-270
Spec Type	= DELTA_NMR
Data Format	= 1D COMPLEX
Dimensions	= X
Dim Title	= 1H
Dim Size	= 16384
Dim Units	= [ppm]
Scans	= 8
Mod_return	= 1
X_points	= 16384
X_prescans	= 0
X_domain	= 1H
X_offset	= 5[ppm]
X_freq	= 270.16608844[MHz]
X_sweep	= 4.05350628[kHz]
X_resolution	= 0.24740639[Hz]
X_acq_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]
X90	= 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]
Deut_grad_shim_attn	= 10[dB]
Adc_card	= 16/1MHz/20
Field_strength	= 6.345446[T]
Filter_mode	= BUTTERWORTH
Filter_width	= 2.02675314[kHz]
Recvr_gain	= 13
Irr_code	= 4
Obs_pwidth	= 1[us]

S25



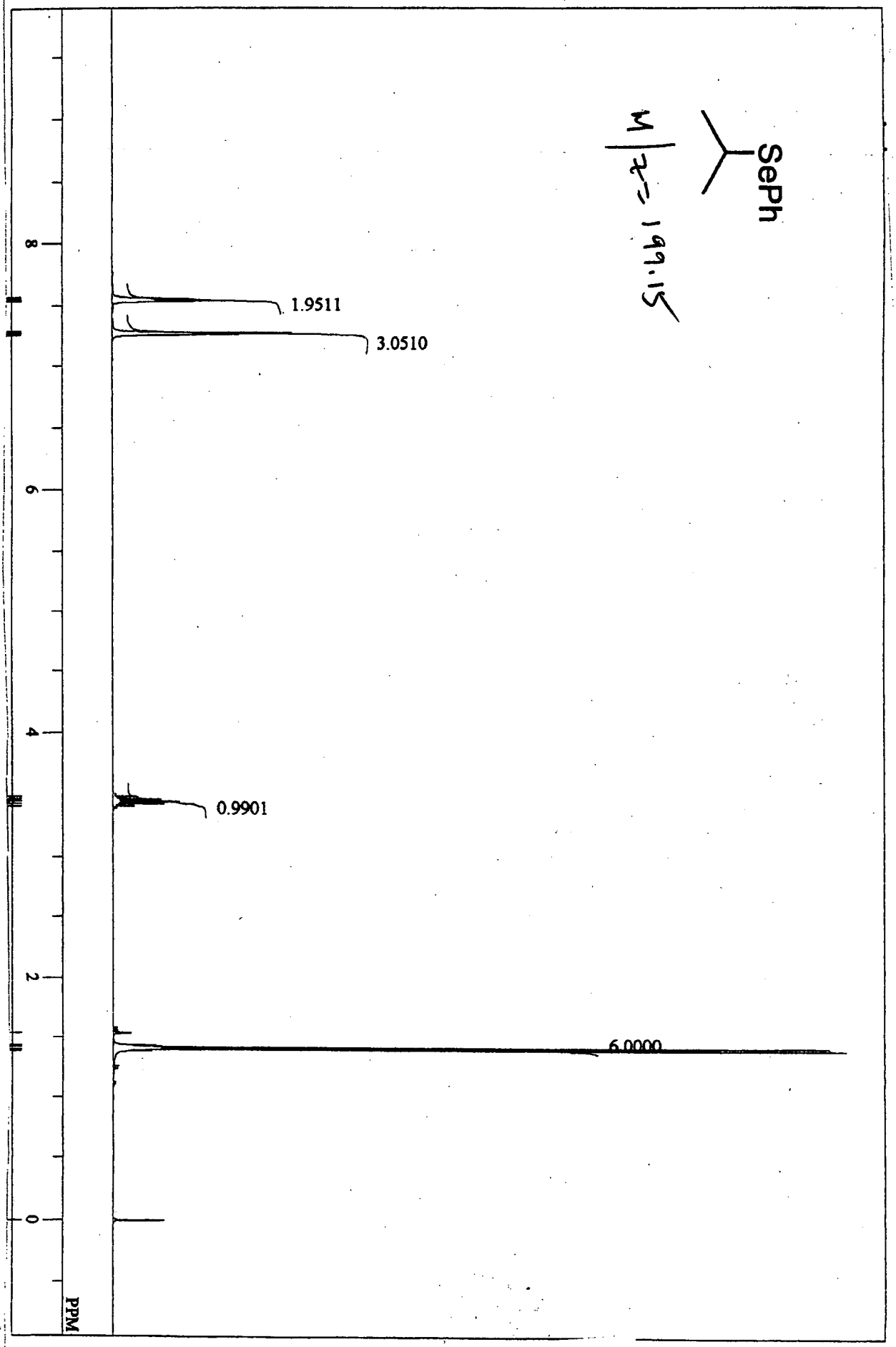
JEOL

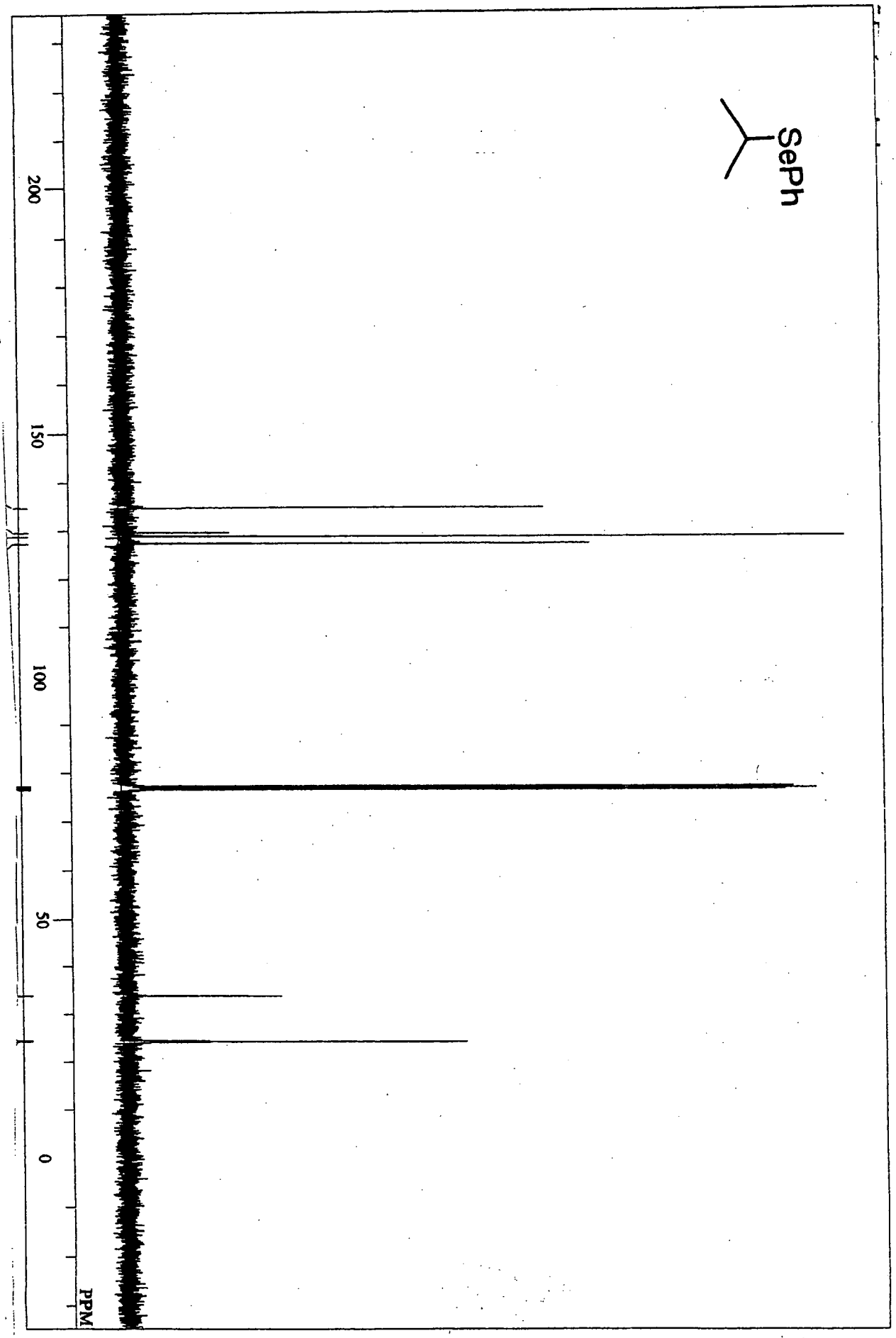
```
---- PROCESSING PARAMETERS ----
dc_balance
sexp : 2[Hz]
fft : 1 : TRUE
machinephase
ppm
```

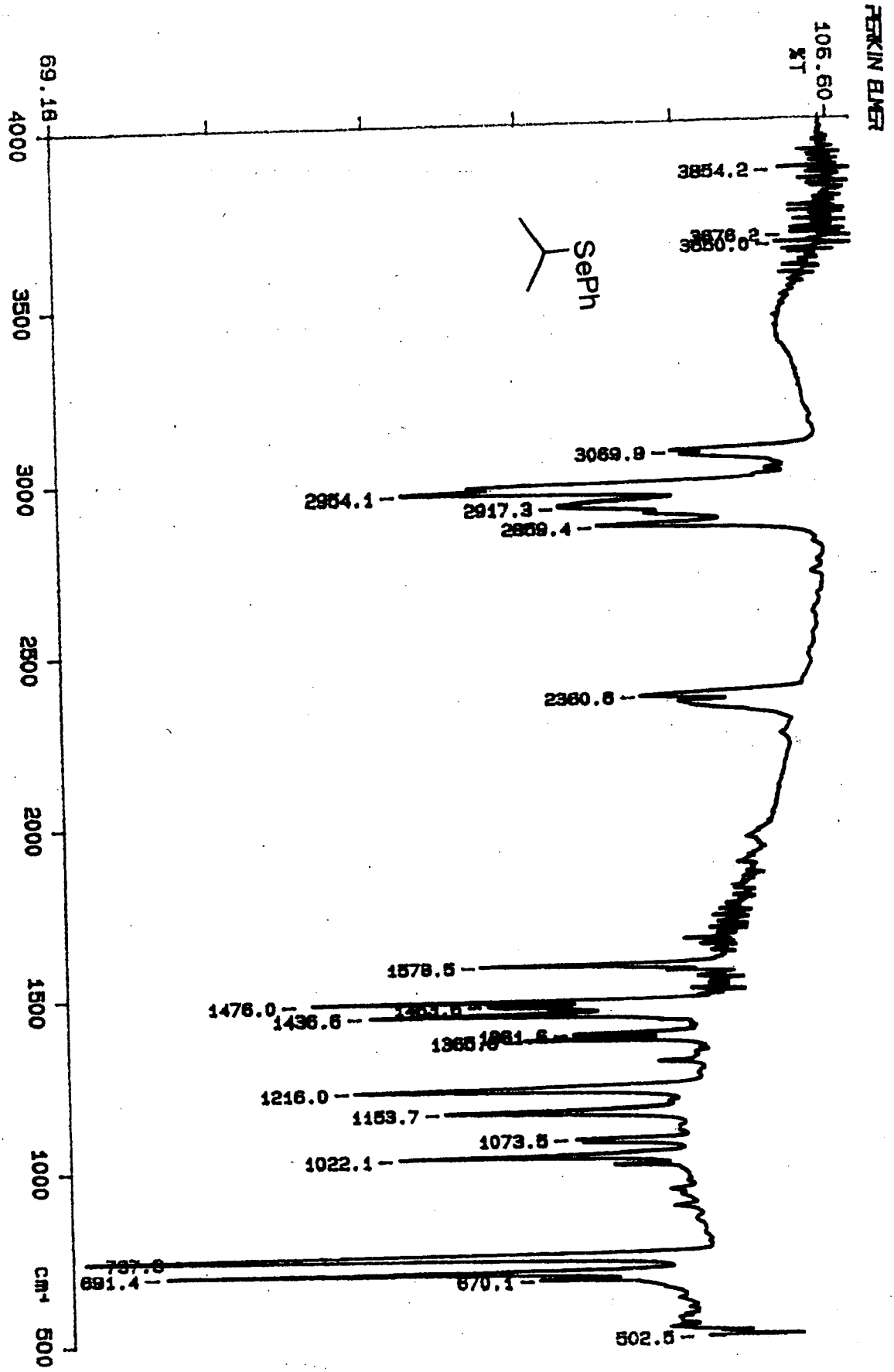


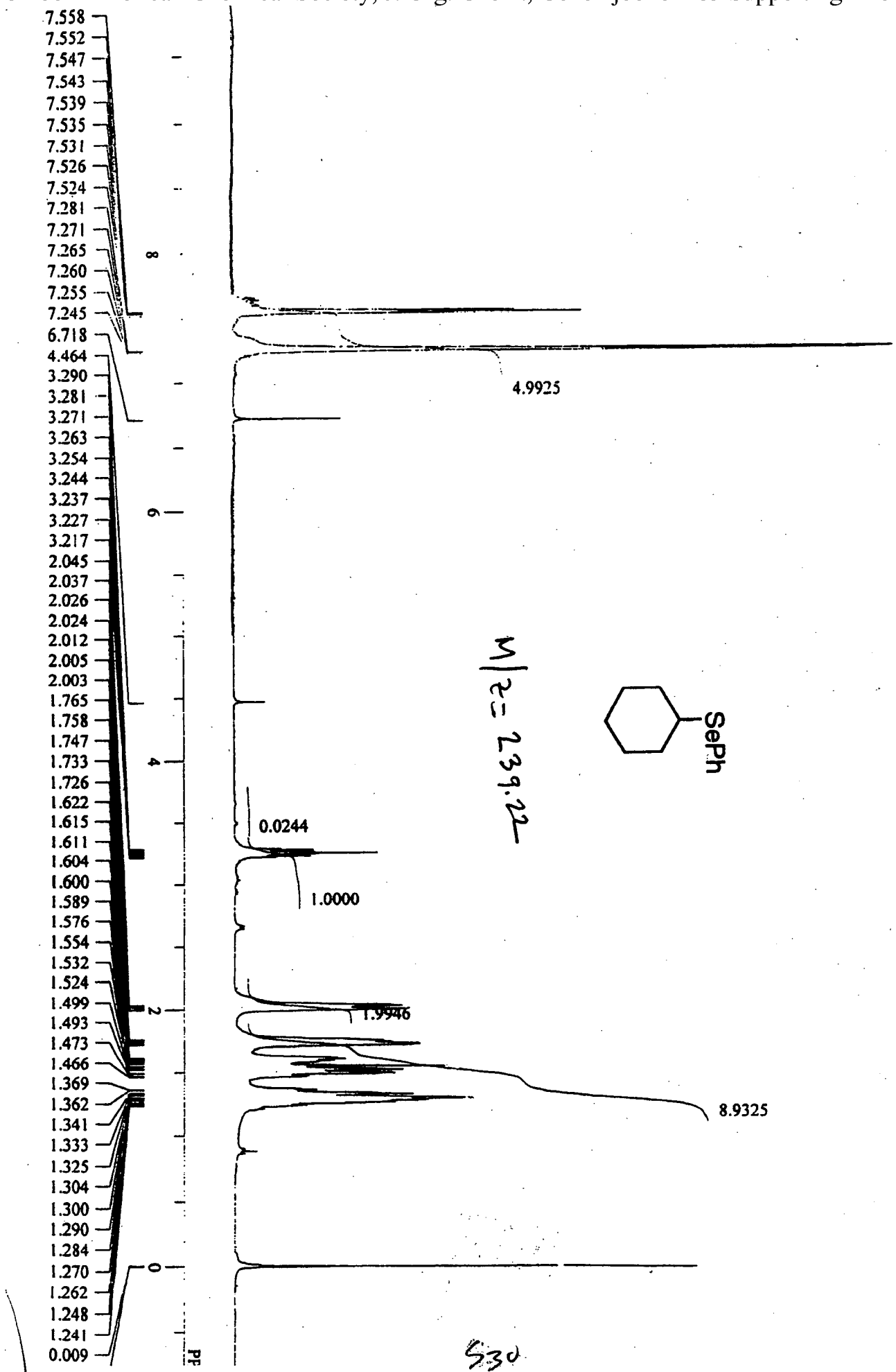

```
---- ACQUISITION PARAMETERS ----
File Name            = 1d_13c_spectrum.13
Author               =
Sample ID            = S#250483
Content              = Single Pulse with
Creation Date        = 12-DEC-2003 08:10:

Revision Date        = 12-DEC-2003 07:08:
Spec Site            = WKU 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              = 17.00680272[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    = 12-DEC-2003 08:00:
Acq_delay            = 57.4[us]
X90                  = 8.5[us]
Irr90                = 11[us]
Tri90                = 10[us]
Qua90                = 10[us]
X90_hi               = 8.9[us]
Irr90_hi             = 18[us]
Tri90_hi             = 10[us]
Qua90_hi             = 10[us]
X90_lo               = 39[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]
Deut_grad_shim_attn  = 10[dB]
Adc_card             = 16/1MHz/20
Field_strength       = 6.345446[T]
Filter_mode          = BUTTERWORTH
Filter_width         = 8.50340136[kHz]
```

S26

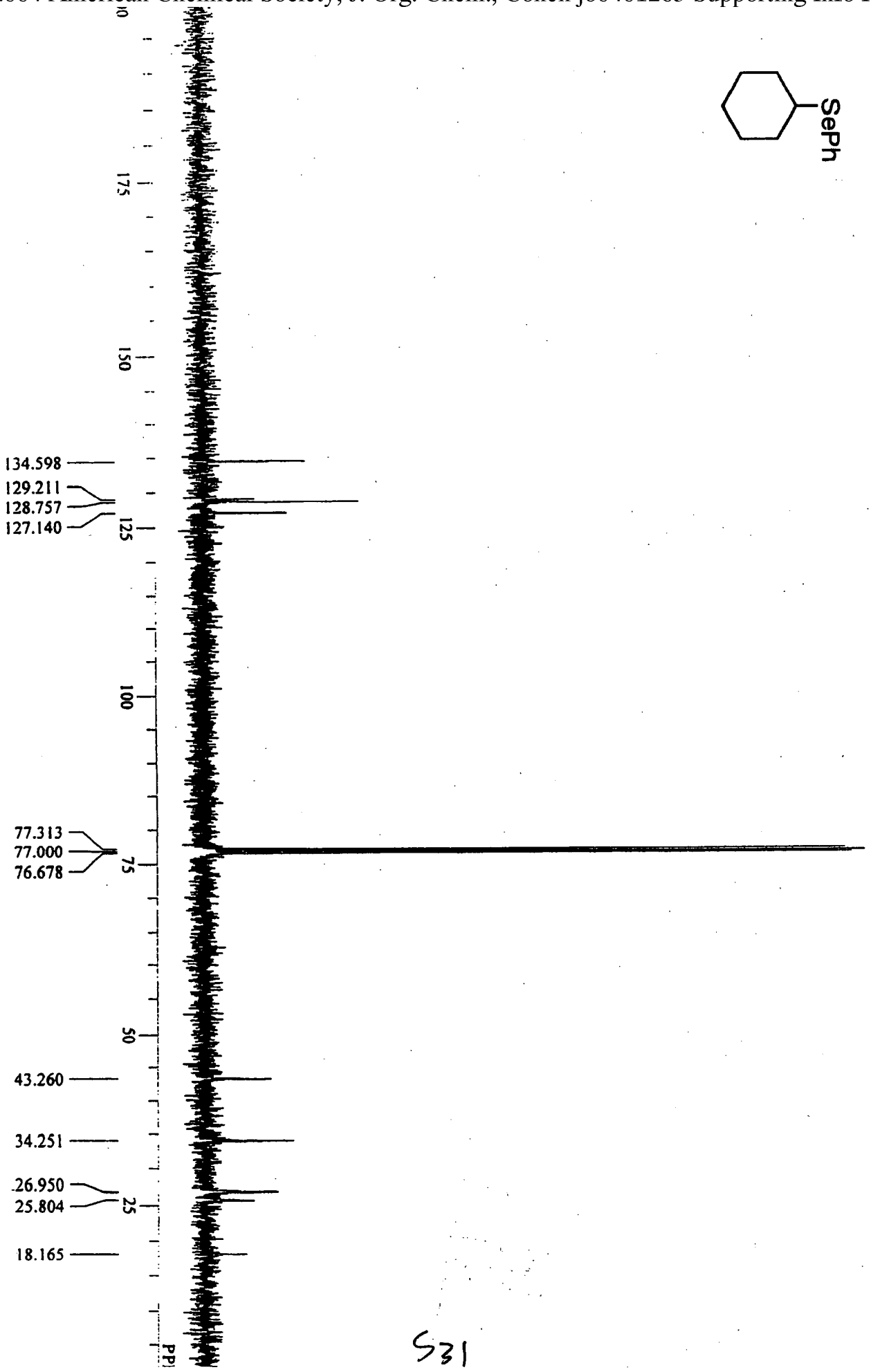


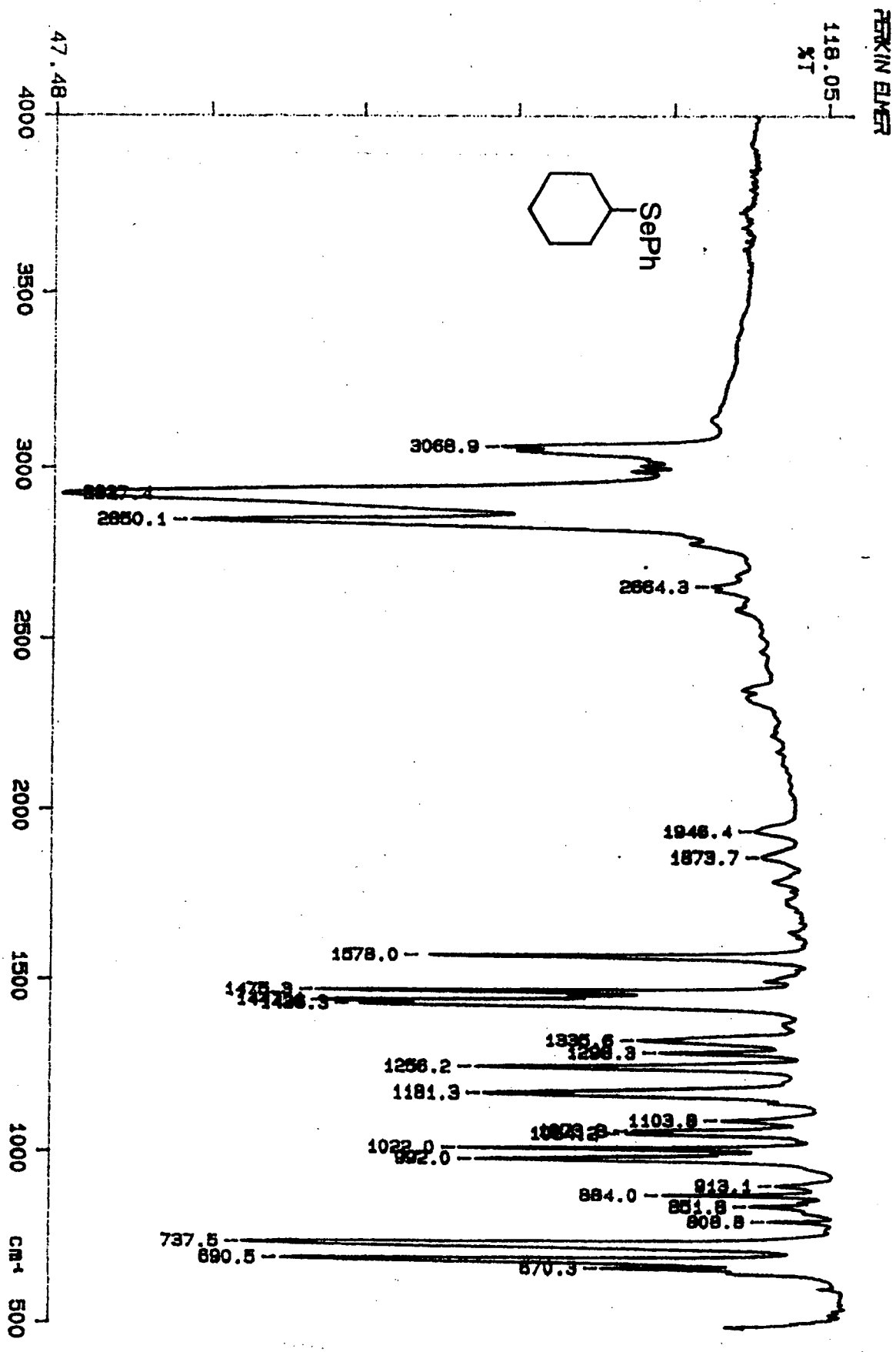


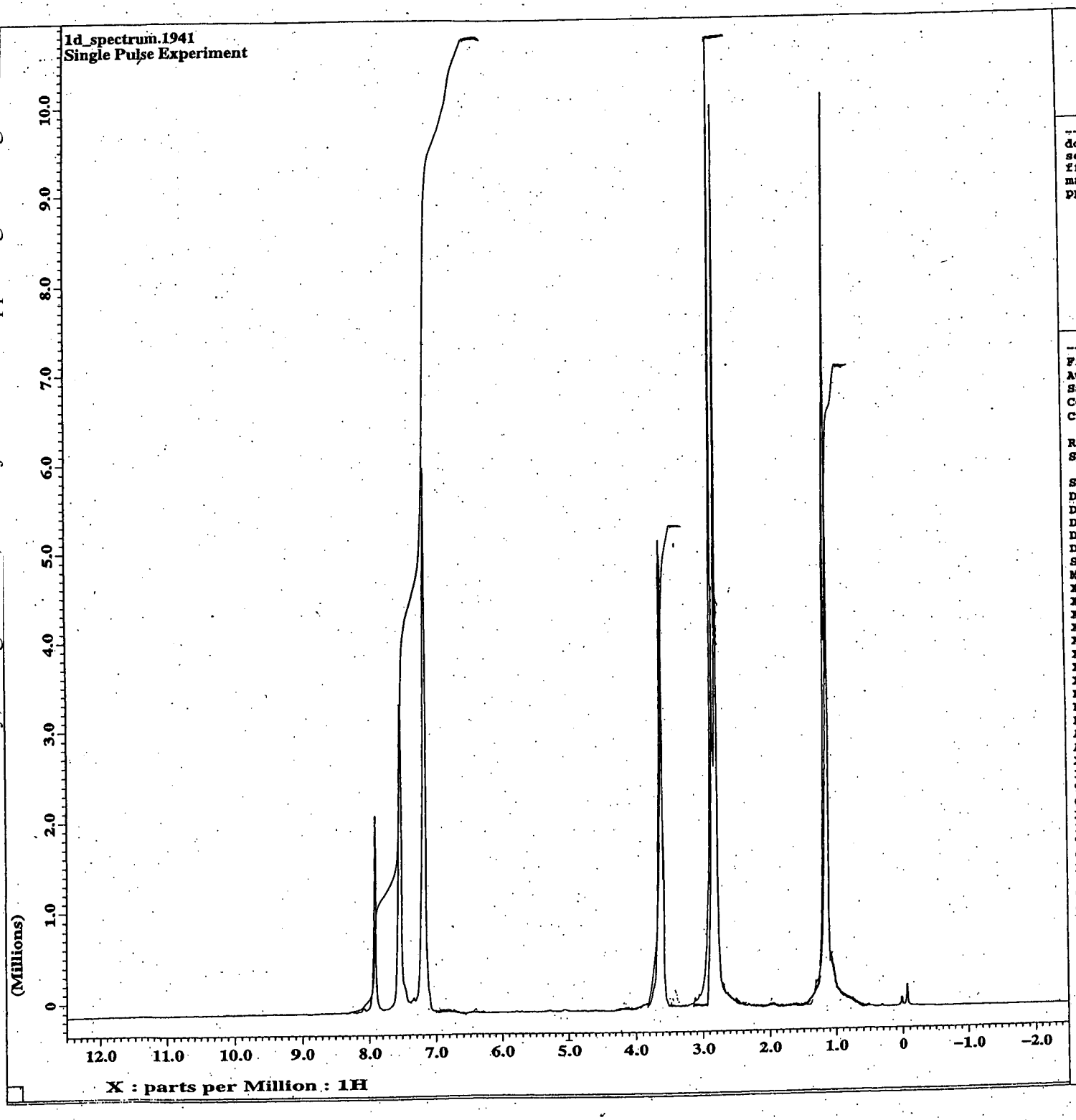




S30

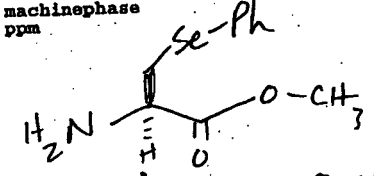






JEOL

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

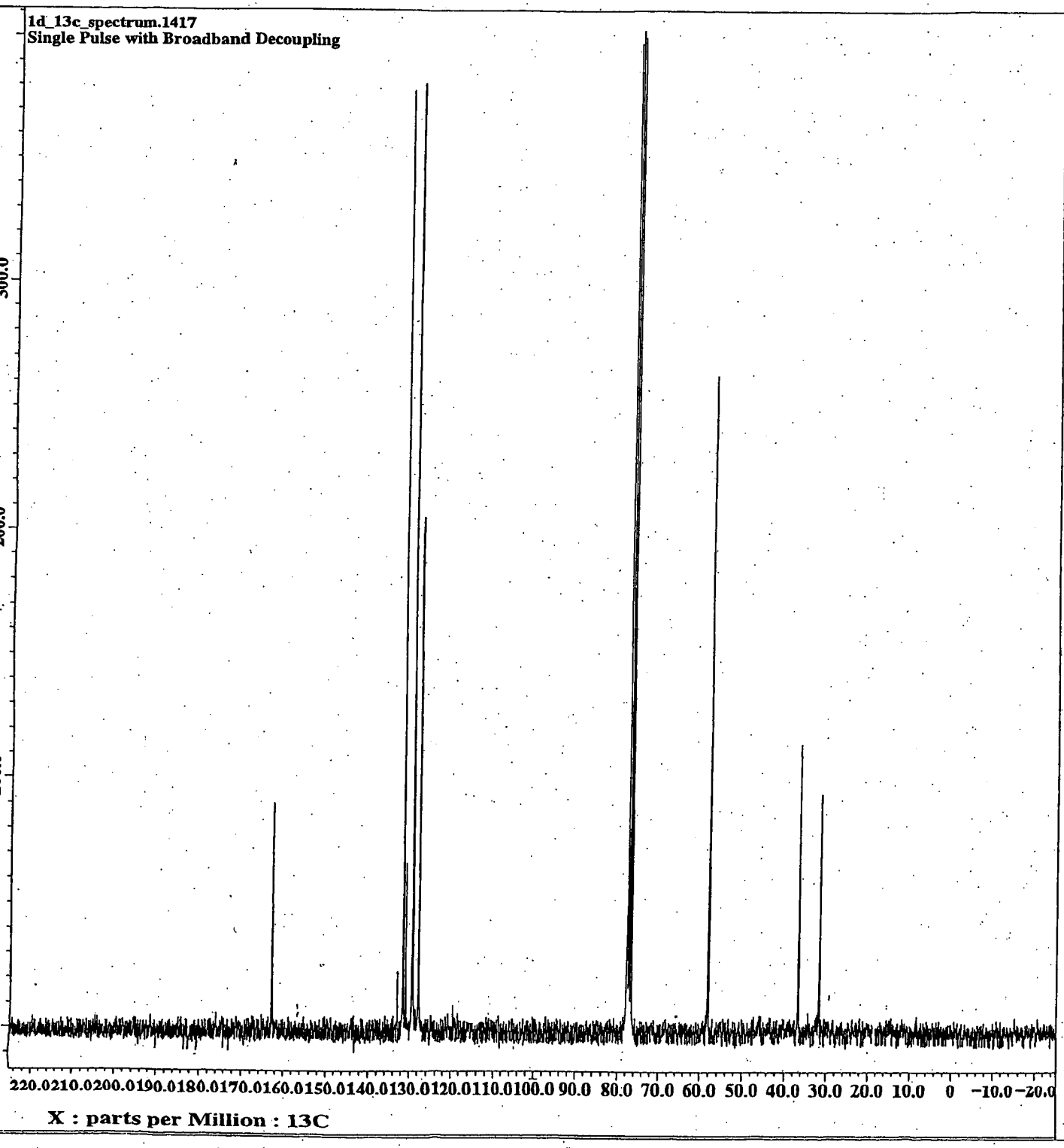


M/Z = 258.18

---- ACQUISITION PARAMETERS ----

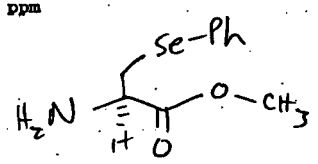
File_Name	= 1d_spectrum.1941
Author	=
Sample_ID	= S#120673
Content	= Single Pulse Exper
Creation_Date	= 19-JAN-2004 04:12:
Revision_Date	= 19-JAN-2004 03:
Spec_Site	= WKU GSX-270
Spec_Type	= DELTA_NMR
Data_Format	= 1D COMPLEX
Dimensions	= X
Dim_Title	= 1H
Dim_Size	= 16384
Dim_Units	= [ppm]
Scans	= 8
Mod_return	= 1
X_points	= 16384
X_prescans	= 0
X_domain	= 1H
X_offset	= 5[ppm]
X_freq	= 270.16608844[MHz]
X_sweep	= 4.05350628[kHz]
X_resolution	= 0.24740639[Hz]
X_acq_duration	= 4.0419328[s]
Digital_filter	= FALSE
Filter_factor	= 1
Delay_of_start	= 1[s]
Actual_start_time	= 19-JAN-2004 04:11:
Acq_delay	= 0.244[ms]
X90	= 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]
Deut_grad_shim_attn	= 10[dB]
Adc_card	= 16/1MHz/20
Field_strength	= 6.345446[T]
Filter_mode	= BUTTERWORTH
Filter_width	= 2.02675314[kHz]
Recvr_gain	= 14
Irr_code	= 4
Obs_pwidth	= 1[us]

S33



JEOL

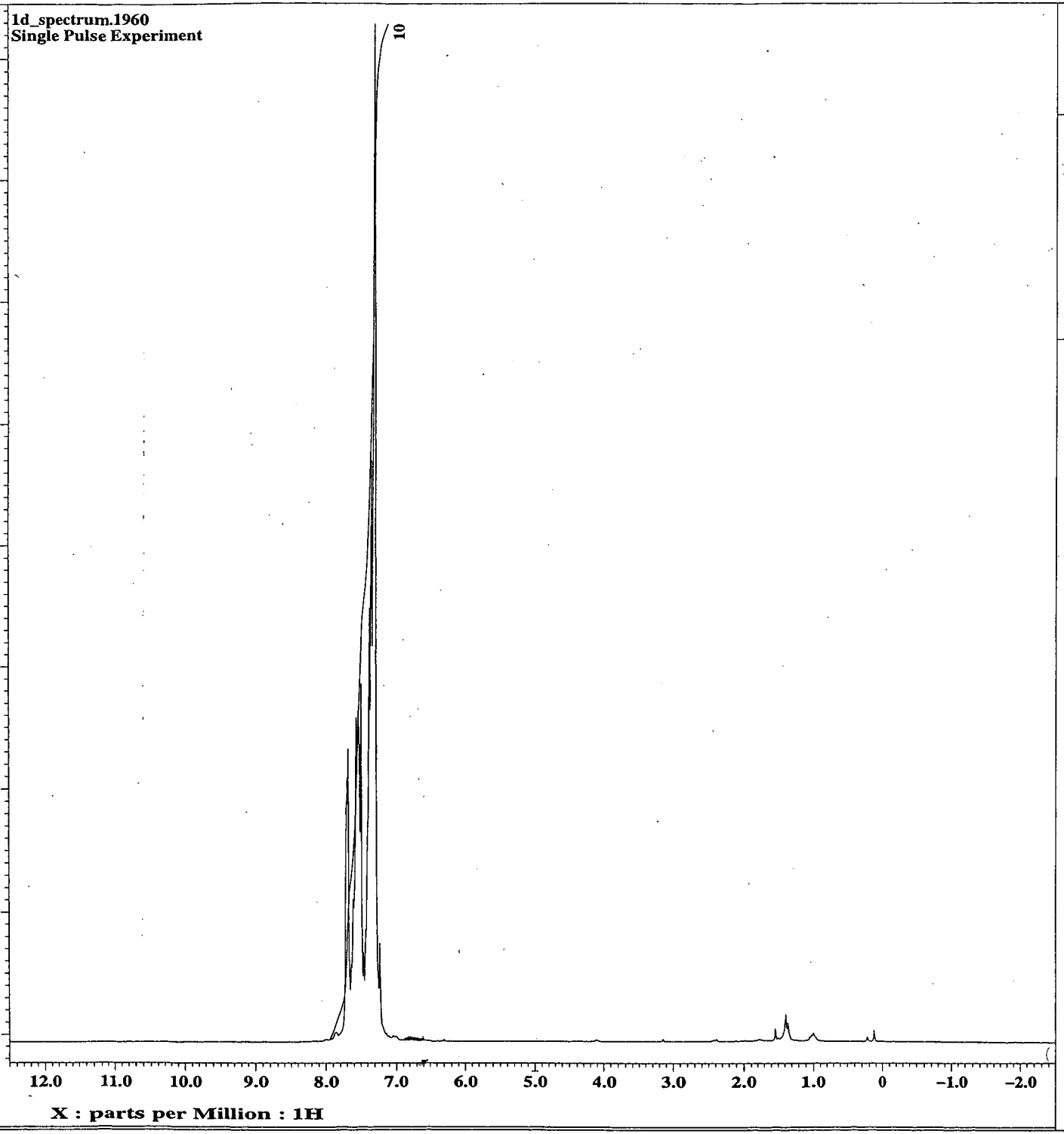
---- PROCESSING PARAMETERS ----
dc_balance
sexp : 2[Hz]
fft : 1 : TRUE
machinephase
ppm



---- ACQUISITION PARAMETERS ----

File Name	= 1d_13c_spectrum.14
Author	=
Sample ID	= S#133633
Content	= Single Pulse with
Creation Date	= 19-JAN-2004 05:15:
Revision Date	= 19-JAN-2004 04:26
Spec Site	= WKU GSX-270
Spec Type	= DELTA_NMR
Data Format	= 1D COMPLEX
Dimensions	= X
Dim Title	= 13C
Dim Size	= 32768
Dim Units	= [ppm]
Scans	= 512
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[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	= 19-JAN-2004 04:33:
Acq_delay	= 57.4[us]
X90	= 8.5[us]
Irr90	= 11[us]
Tri90	= 10[us]
Qua90	= 10[us]
X90_hi	= 8.9[us]
Irr90_hi	= 18[us]
Tri90_hi	= 10[us]
Qua90_hi	= 10[us]
X90_lo	= 39[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]
Deut_grad_shim_atten	= 10[dB]
Adc_card	= 16/18
Field_strength	= 6.345446[T]
Filter_mode	= BUTTERWORTH
Filter_width	= 8.50340136[kHz]

S34



JEOL

Cat. aww CSOH

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

Ph − C ≡ C − Se − Ph

```
---- ACQUISITION PARAMETERS ----
File Name              = 1d_spectrum.1960
Author                 =
Sample ID              = S#132119
Content                = Single Pulse Exper
Creation Date          = 21-JAN-2004 04:31:

Revision Date          = 21-JAN-2004 03:45:
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                 = 270.16608844[MHz]
X_sweep                = 4.05350628[kHz]
X_resolution           = 0.24740639[Hz]
X_acq_duration         = 4.0419328[s]
Digital_filter         = FALSE
Filter_factor          = 1
Delay_of_start         = 1[s]
Actual_start_time      = 21-JAN-2004 04:30:
Acq_delay              = 0.244[ms]
X90                    = 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]
Deut_grad_shim_attn    = 10[dB]
Adc_card               = 16/1MHz/20
Field_strength         = 6.345446[T]
Filter_mode            = BUTTERWORTH
Filter_width           = 2.02675314[kHz]
Recvr_gain             = 11
Irr_code               = 4
Obs_pwidth             = 1[us]
```

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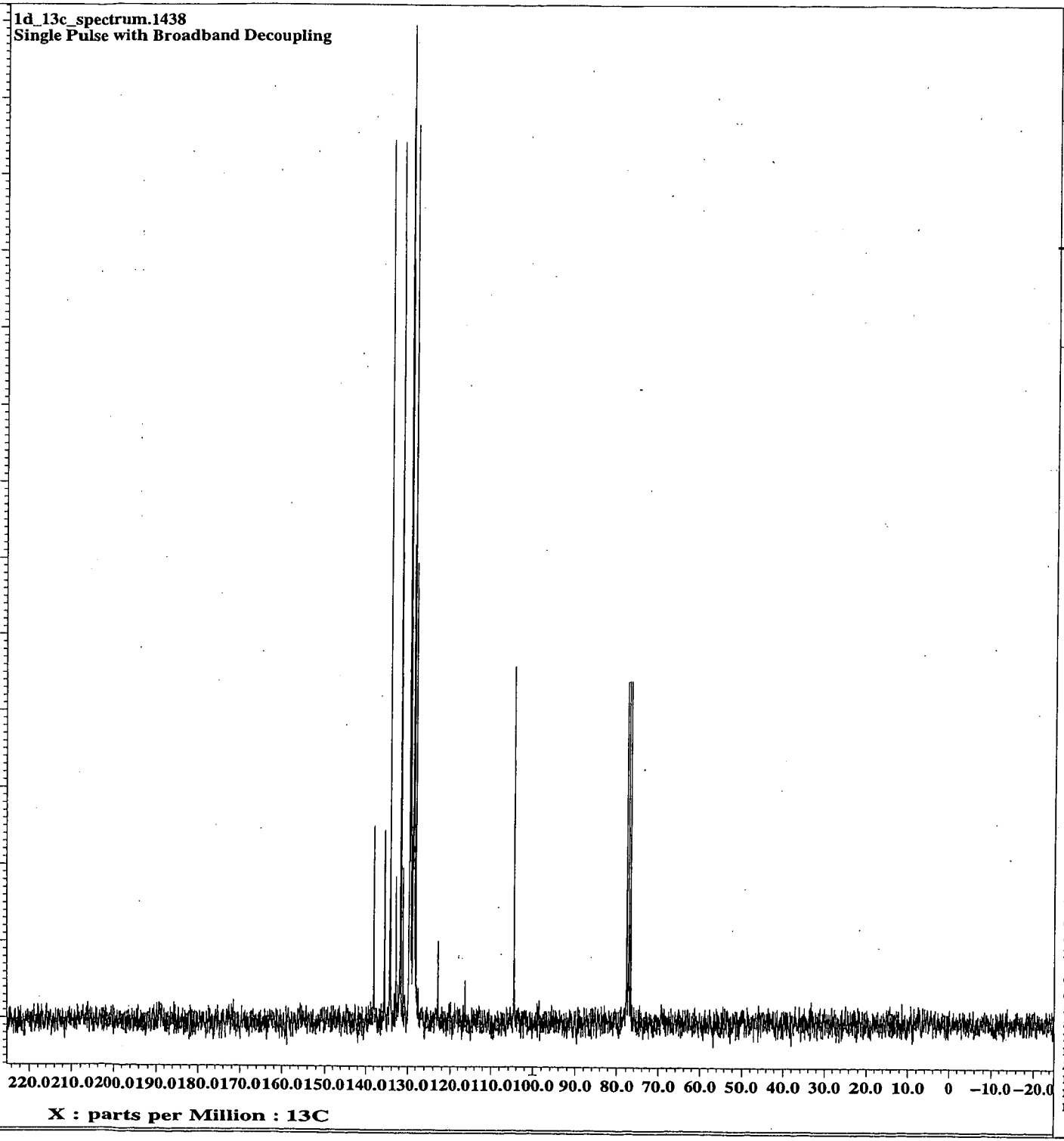


JEOL

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

Ph-C≡C-Se-Ph

```
---- ACQUISITION PARAMETERS ----
File Name            = 1d_13c_spectrum.14
Author               =
Sample ID            = S#134194
Content              = Single Pulse with
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            = 13C
Dim Size             = 32768
Dim Units            = [ppm]
Scans                = 75
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[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]
Irr90                = 11[us]
Tri90                = 10[us]
Qua90                = 10[us]
X90_hi               = 8.9[us]
Irr90_hi             = 18[us]
Tri90_hi             = 10[us]
Qua90_hi             = 10[us]
X90_lo               = 39[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]
Deut_grad_shim_attn  = 10[dB]
Adc_card             = 16/1MHz/20
Field_strength       = 6.345446[T]
Filter_mode          = BUTTERWORTH
Filter_width         = 8.50340136[kHz]
```

S36

Acquired : 22 Jan 2004 10:13 using AcqMethod PHIL

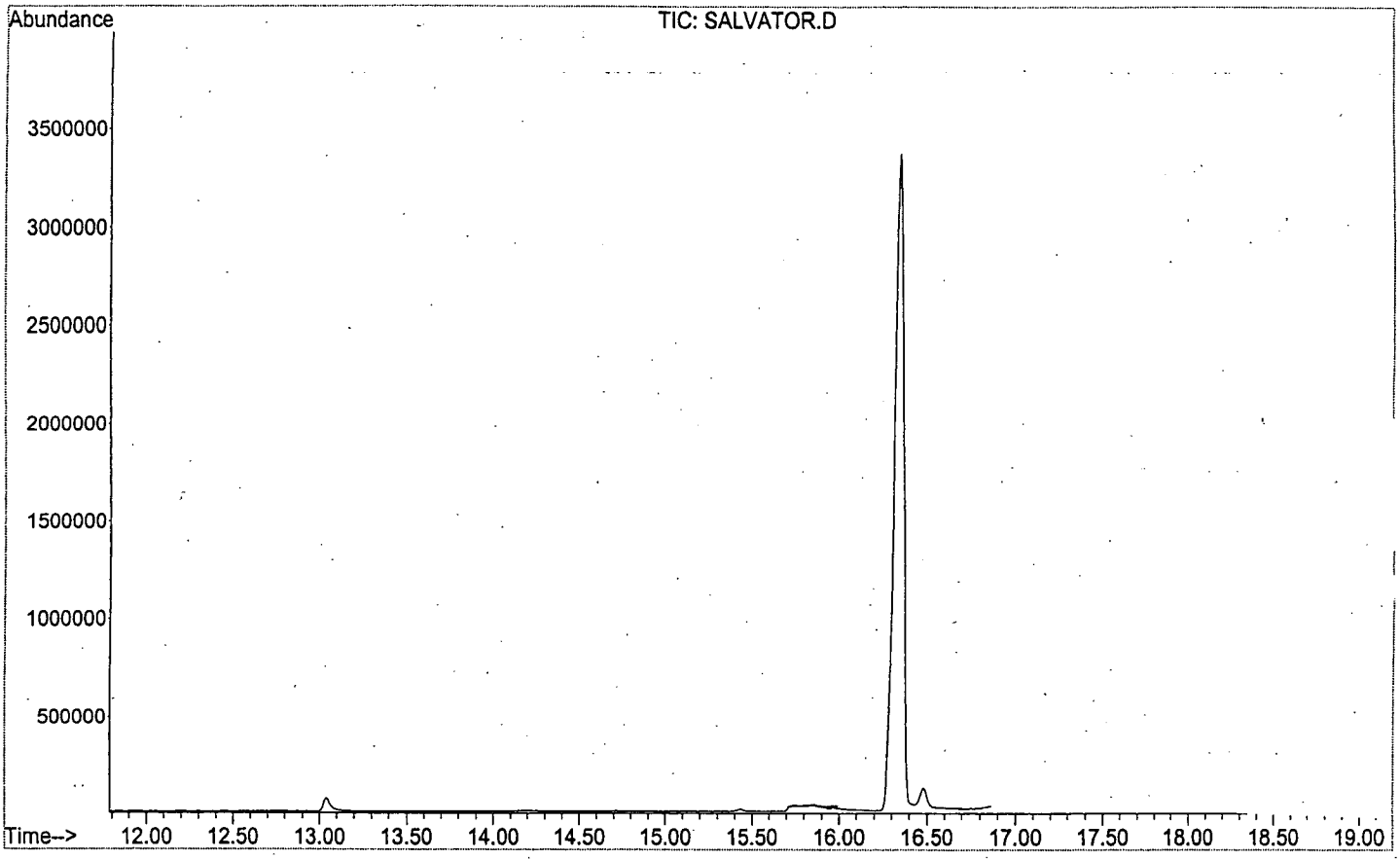
Instrument : 5973 MSD

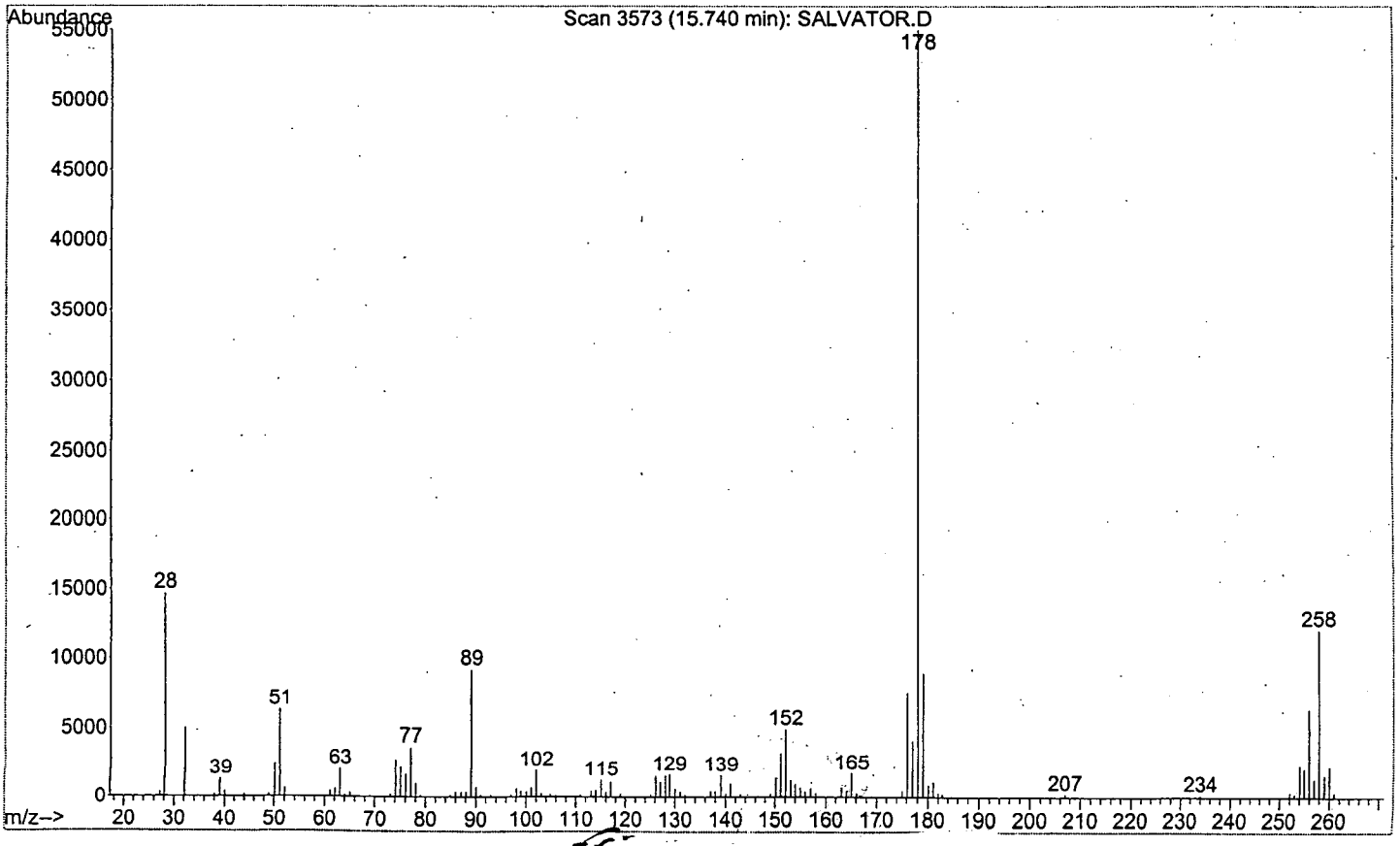
Sample Name: Diphenyl alkyne selenium type thing

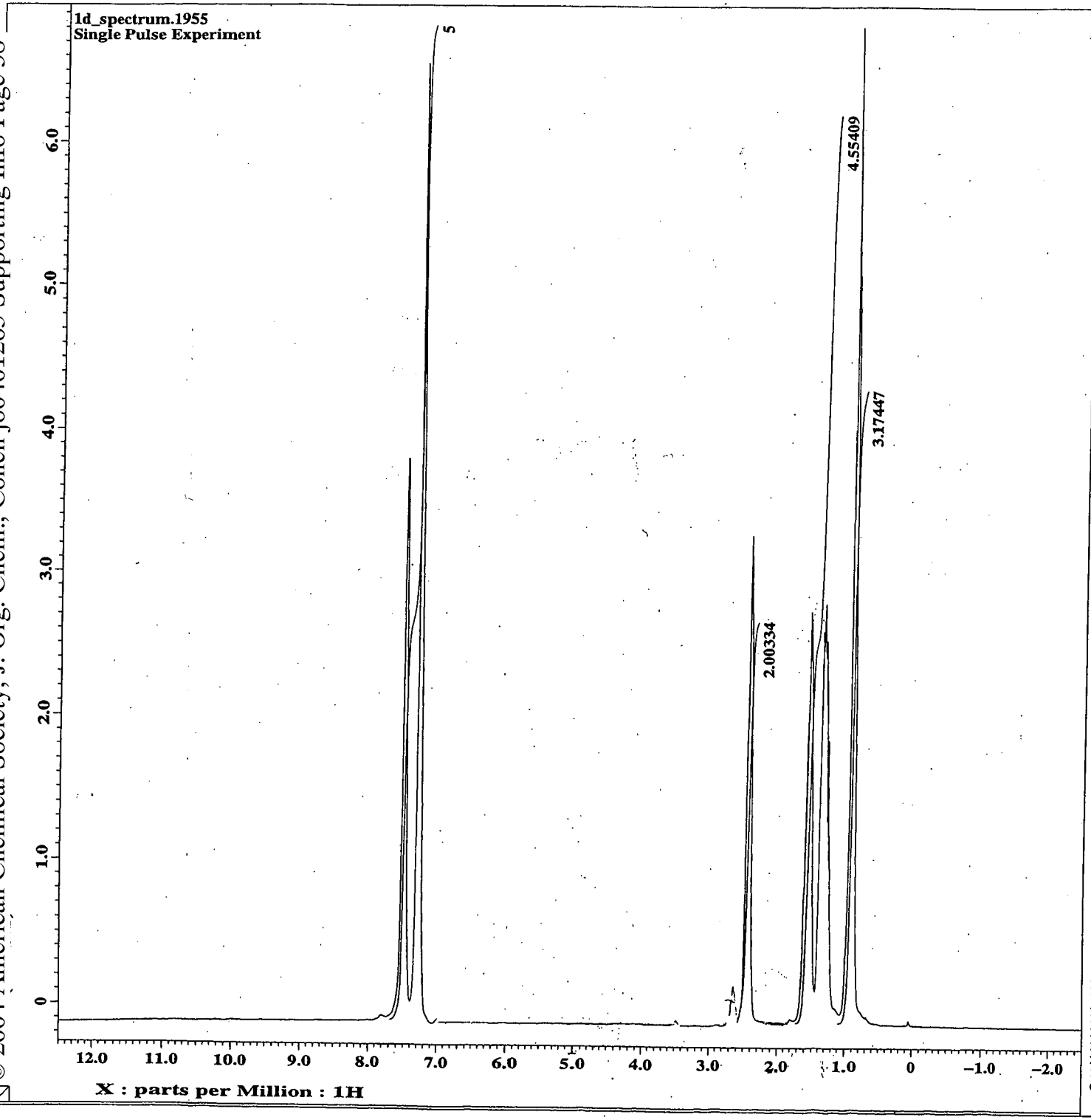
Misc Info :

Vial Number: 1

Ph C≡C–Se–Ph







JEOL

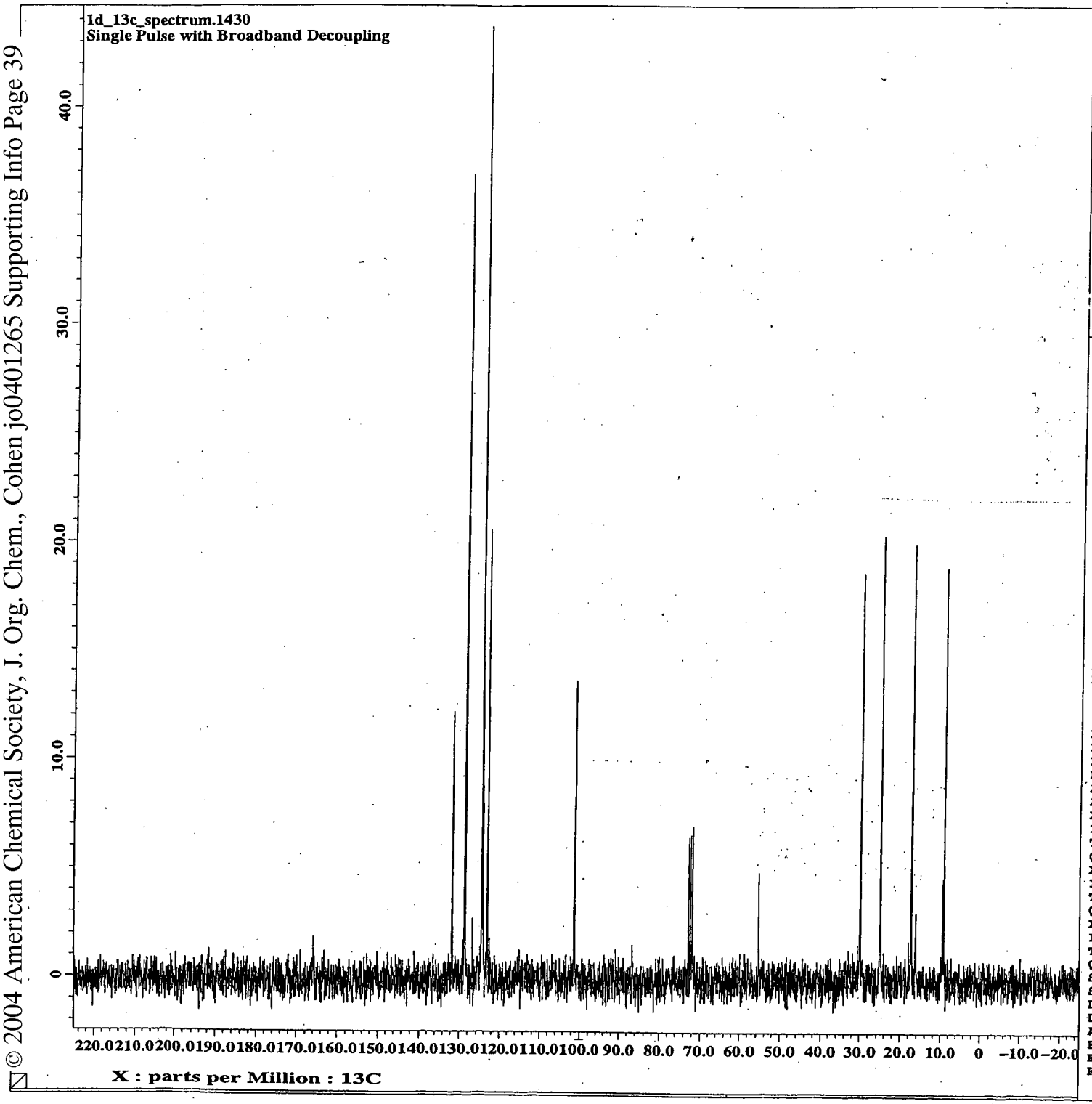
---- PROCESSING PARAMETERS ----
dc_balance
sexp : 0.2[Hz]
fft : 1 : TRUE
machinephase
ppm

M/z=237.20

---- ACQUISITION PARAMETERS ----

File Name	= 1d_spectrum.1955
Author	=
Sample ID	= S#55273
Content	= Single Pulse Exper
Creation Date	= 21-JAN-2004 02:23:
Revision Date	= 21-JAN-2004 01:37:
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	= 270.16608844[MHz]
X_sweep	= 4.05350628[kHz]
X_resolution	= 0.24740639[Hz]
X_acq_duration	= 4.0419328[s]
Digital_filter	= FALSE
Filter_factor	= 1
Delay_of_start	= 1[s]
Actual_start_time	= 21-JAN-2004 02:22:
Acq_delay	= 0.244[ms]
X90	= 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]
Deut_grad_shim_attn	= 10[dB]
Adc_card	= 16/1MHz/20
Field_strength	= 6.345446[T]
Filter_mode	= BUTTERWORTH
Filter_width	= 2.02675314[kHz]
Recvr_gain	= 10
Irr_code	= 4
Obs_pwidth	= 1[us]

S28



JEOL

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

seph

```
---- ACQUISITION PARAMETERS ----
File Name              = 1d_13c_spectrum.14
Author                 =
Sample ID              = S#528018
Content                = Single Pulse with
Creation Date          = 20-JAN-2004 15:32:

Revision Date          = 20-JAN-2004 14:43:
Spec Site              = WKU GSX-270

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[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      = 20-JAN-2004 15:30:
Acq_delay              = 57.4[us]
X90                    = 8.5[us]
Irr90                  = 11[us]
Tri90                  = 10[us]
Qua90                  = 10[us]
X90_hi                 = 8.9[us]
Irr90_hi               = 18[us]
Tri90_hi               = 10[us]
Qua90_hi               = 10[us]
X90_lo                 = 39[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]
Deut_grad_shim_attn    = 10[dB]
Adc_card               = 16/1MHz/20
Field_strength         = 6.345446[T]
Filter_mode            = BUTTERWORTH
Filter_width           = 8.50340136[kHz]
```

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