

# Photorelease of Tyrosine from Alpha- Carboxy-6-nitroveratryl ( $\alpha$ CNV) Derivatives

## Supplementary Information

*Alexander G. Russell,<sup>†</sup> Matthew J. Sadler,<sup>†</sup> Helen J. Laidlaw,<sup>†</sup> Agustín Gutiérrez-  
Loriente,<sup>†</sup> Christopher W. Wharton,<sup>‡</sup> David Carteau,<sup>§</sup> Dario M. Bassani<sup>\*,§</sup> and John  
S. Snaith<sup>\*,†</sup>*

School of Chemistry, University of Birmingham, Edgbaston, Birmingham, B15 2TT,  
U.K., School of Biosciences, University of Birmingham, Edgbaston, Birmingham,  
B15 2TT, U.K. and ISM CNRS UMR 5255, Université Bordeaux 1, 33405 Talence,  
France.

j.s.snaith@bham.ac.uk

d.bassani@ism.u-bordeaux1.fr

<sup>†</sup> School of Chemistry, University of Birmingham

<sup>‡</sup> School of Biosciences, University of Birmingham

<sup>§</sup> University Bordeaux 1

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**Diethyl 2,2'-((4-(1-diazo-2-(2-ethoxy-2-oxoethoxy)-2-oxoethyl)-5-nitro-1,2-phenylene)bis(oxy))diacetate:**

<sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) S24

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) S25

**2-(4,5-Bis(ethoxycarbonylmethoxy)-2-nitrophenyl)-2-bromoacetic acid ethoxycarbonylmethyl ester (9):**

<sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) S26

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) S27

**Diethyl 2,2'-((4-(1-(4-(*tert*-butyl)phenoxy)-2-(2-ethoxy-2-oxoethoxy)-2-oxoethyl)-5-nitro-1,2-phenylene)bis(oxy))diacetate (10):**

<sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) S28

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) S29

**Diethyl 2,2'-((4-(1-(4-((*S*)-2-((*tert*-butoxycarbonyl)amino)-3-methoxy-3-oxopropyl)phenoxy)-2-(2-ethoxy-2-oxoethoxy)-2-oxoethyl)-5-nitro-1,2-phenylene)bis(oxy))diacetate (11):**

<sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) S30

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) S31

**2,2'-((4-((4-(*tert*-Butyl)phenoxy)(carboxy)methyl)-5-nitro-1,2-phenylene)bis(oxy))diacetic acid (12):**

<sup>1</sup>H NMR (300 MHz, d<sub>6</sub>-acetone) S32

<sup>13</sup>C NMR (75 MHz, d<sub>6</sub>-acetone) S33

**(1*S*)-2-(4-((4,5-Bis(carboxymethoxy)-2-nitrophenyl)(carboxy)methoxy)phenyl)-1-carboxyethanaminium 2,2,2-trifluoroacetate (13):**

<sup>1</sup>H NMR (300 MHz, D<sub>2</sub>O) S34

<sup>13</sup>C NMR (75 MHz, D<sub>2</sub>O) S35

***tert*-Butyl 2-(4,5-dimethoxy-2-nitrophenyl)acetate:**

<sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) S36

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) S37

***tert*-Butyl 2-diazo-2-(4,5-dimethoxy-2-nitrophenyl)acetate**

<sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) S38

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) S39

***tert*-Butyl 2-bromo-2-(4,5-dimethoxy-2-nitrophenyl)acetate (14):**

<sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) S40

<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) S41

**(2*S*)-*tert*-Butyl 3-(4-(2-(allyloxy)-1-(4,5-dimethoxy-2-nitrophenyl)-2-oxoethoxy)phenyl)-2-((*tert*-butoxycarbonyl)amino)propanoate (17):**

<sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) S42

<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) S43

**(2*S*)-Methyl 3-(4-(2-(*tert*-butoxy)-1-(4,5-dimethoxy-2-nitrophenyl)-2-oxoethoxy)phenyl)-2-(2,2,2-trifluoroacetamido)propanoate (18):**

<sup>1</sup>H NMR (300 MHz, d<sub>6</sub>-acetone) S44

<sup>13</sup>C NMR (75 MHz, d<sub>6</sub>-acetone) S45

**(1*S*)-2-(4-(2-(Allyloxy)-1-(4,5-dimethoxy-2-nitrophenyl)-2-oxoethoxy)phenyl)-1-carboxyethanaminium 2,2,2-trifluoroacetate**

<sup>1</sup>H NMR (300 MHz, d<sub>6</sub>-acetone) S46

**(2*S*)-2-(((9*H*-Fluoren-9-yl)methoxy)carbonyl)amino)-3-(4-(2-(allyloxy)-1-(4,5-dimethoxy-2-nitrophenyl)-2-oxoethoxy)phenyl)propanoic acid (19)**

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) S47

<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) S48

**(2*S*)-2-(((9*H*-Fluoren-9-yl)methoxy)carbonyl)amino)-3-(4-(2-(*tert*-butoxy)-1-(4,5-dimethoxy-2-nitrophenyl)-2-oxoethoxy)phenyl)propanoic acid (20)**

<sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) S49

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) S50

**Mosher's amide of +/- tyrosine**

<sup>19</sup>F NMR (282 MHz, d<sub>6</sub>-acetone) S51

**Mosher's amide of tyrosine released by photolysis of Mosher's amide of 7**

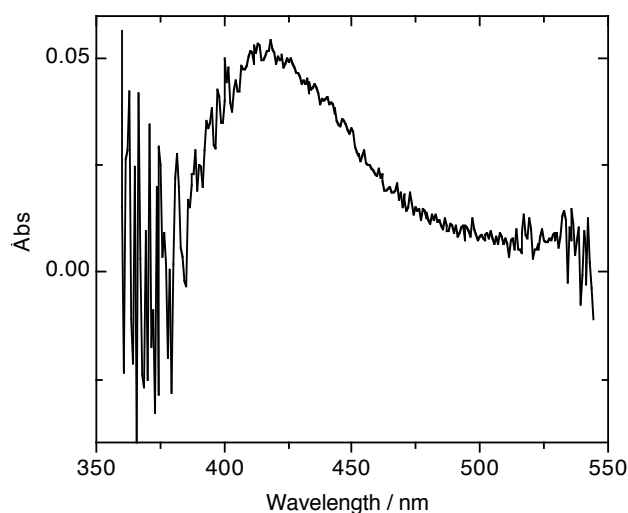
<sup>19</sup>F NMR (282 MHz, d<sub>6</sub>-acetone) S52

## Kinetic measurements

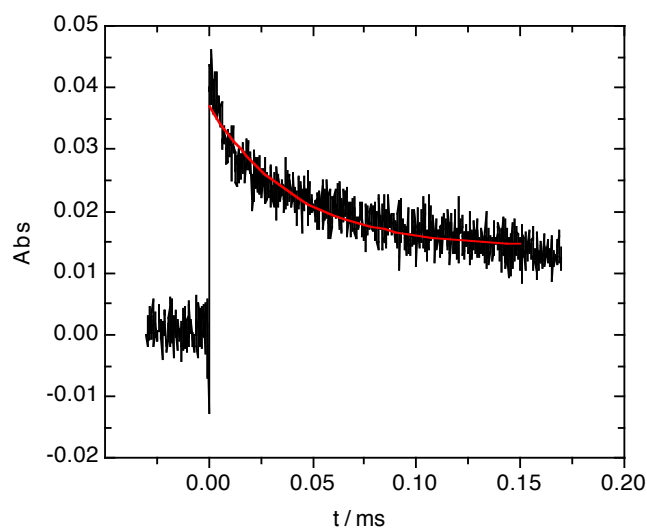
The measurements were performed in aerated H<sub>2</sub>O or EtOH/H<sub>2</sub>O (4:1) solutions, as indicated, using a pump – probe setup equipped with a frequency - tripled Nd-YAG laser (BMI, 355 nm, 6 ns pulses) as an excitation source. An intensified CCD (Andor Technologies Instaspec V) or a photomultiplier (Hamamatsu R 446 UR connected to a transient digitizer) was used for detection for the spectral and temporal measurements, respectively.

## Kinetic Measurements for Compound 7

Measurements made in water.



Transient absorption after 10  $\mu$ s

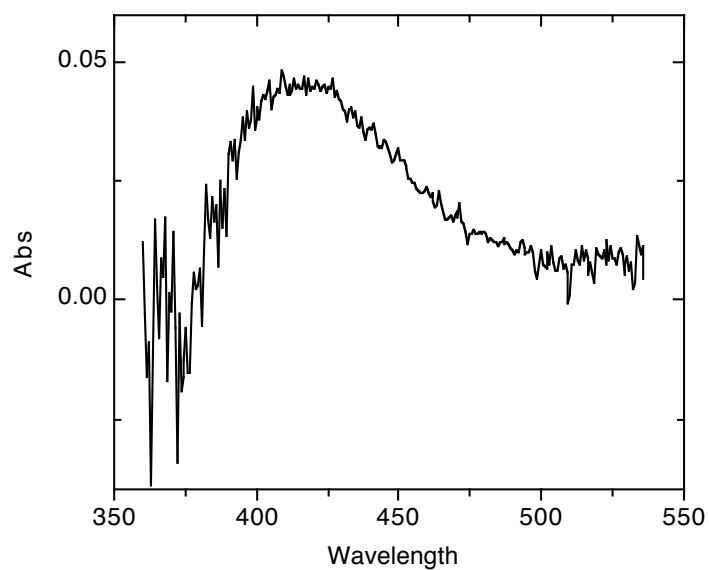


Decay monitored at 420 nm ( $k = 2.4 \times 10^4 \text{ s}^{-1}$ )

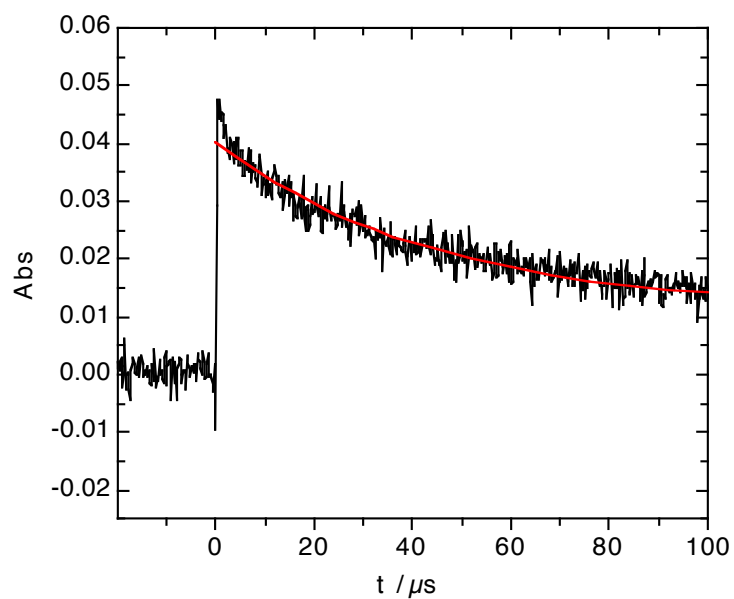
Chi squared = 5.2555e-3  
Parameters: Standard deviations:  
A = 2.9284e-2  $\Delta A$  = 1.4412e+4  
x0 = -9.9136e-6  $\Delta x0$  = 20.1604  
t0 = 4.0963e-5  $\Delta t0$  = 1.5494e-6  
const = 1.4108e-2  $\Delta const$  = 2.0486e-4

### Kinetic Measurements for Compound 13

Measurements made in water.



Transient absorption after 10  $\mu\text{s}$

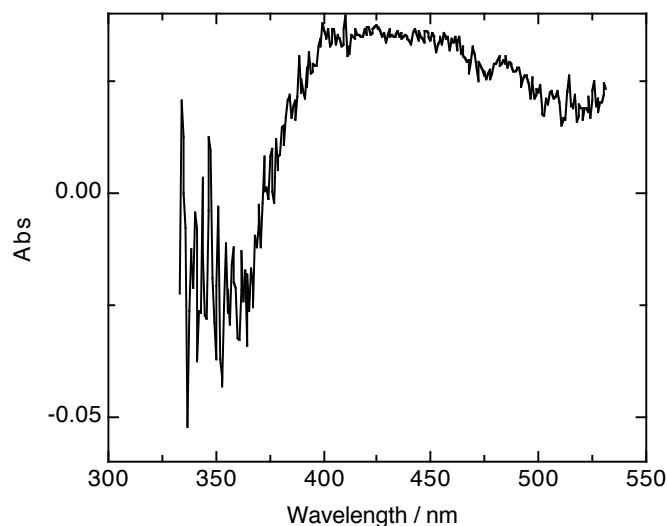


Decay monitored at 420 nm ( $k = 2.3 \times 10^4 \text{ s}^{-1}$ )

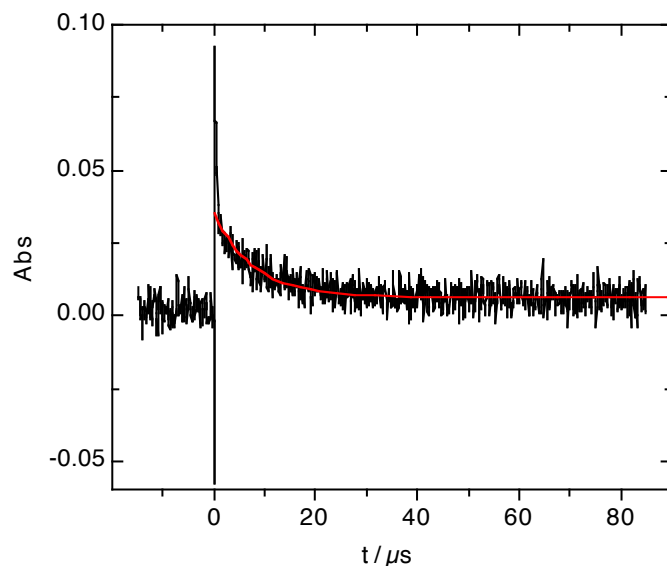
Chi squared = 3.7828e-3  
Parameters: Standard deviations:  
A = 3.3573e-2  $\Delta A$  = 4436.0902  
x0 = -6.2146e-6  $\Delta x0$  = 5.7719  
t0 = 4.3683e-5  $\Delta t0$  = 1.1500e-6  
const = 1.1171e-2  $\Delta const$  = 1.8916e-4

### Kinetic Measurements for Compound 5

Measurements made in ethanol/water (4:1)



Transient absorption after 1  $\mu s$



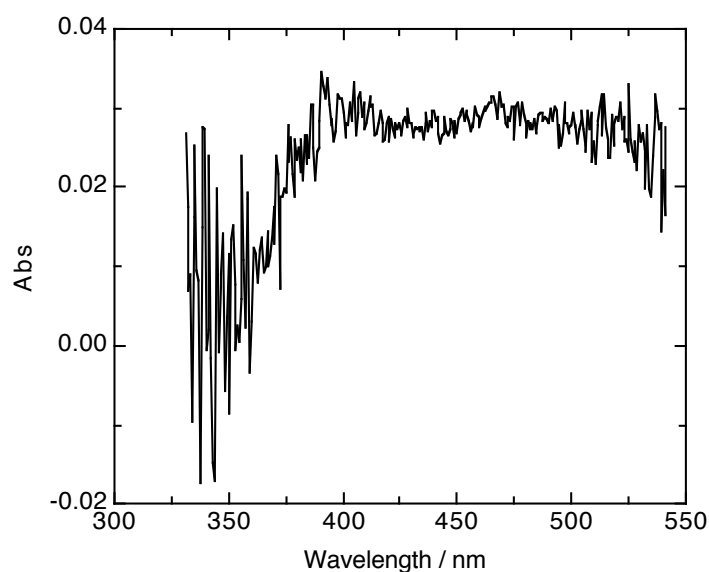
Decay monitored at 420 nm ( $k = 1.3 \times 10^5 \text{ s}^{-1}$ )

Chi squared = 1.3074e-2  
Parameters: Standard deviations:  
A = 3.7668e-2  $\Delta A$  = 2.9417e+4  
x0 = -2.0372e-6  $\Delta x0$  = 5.9850  
t0 = 7.6637e-6  $\Delta t0$  = 4.0326e-7  
const = 6.4542e-3  $\Delta const$  = 1.6965e-4

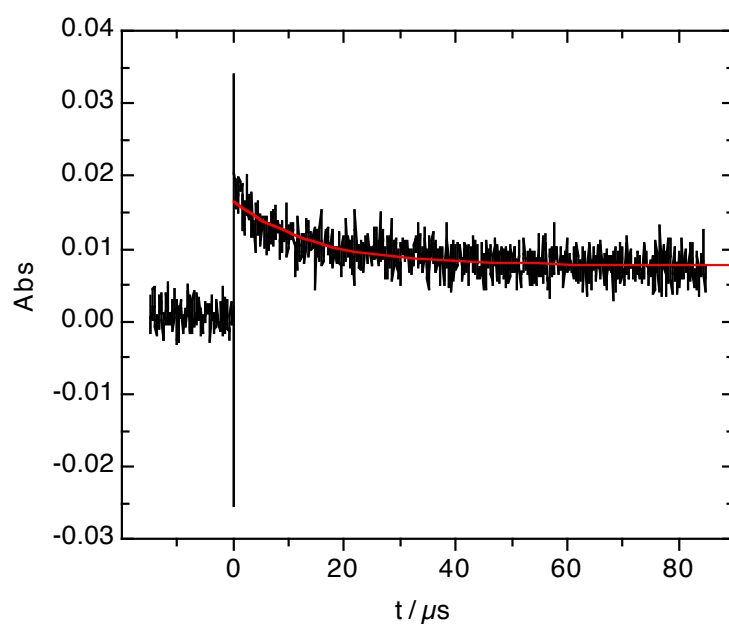


## Kinetic Measurements for Compound 11

Measurements made in ethanol/water (4:1)



Transient absorption after 1  $\mu$ s



Decay monitored at 420 nm ( $k = 7.0 \times 10^4 \text{ s}^{-1}$ )

|             |   |            |                       |             |
|-------------|---|------------|-----------------------|-------------|
| Chi squared | = | 3.1589e-3  |                       |             |
| Parameters: |   |            | Standard deviations:  |             |
| A           | = | 2.3738e-2  | $\Delta A$            | = 3.7685e+4 |
| x0          | = | -1.3965e-5 | $\Delta x0$           | = 22.5644   |
| t0          | = | 1.4214e-5  | $\Delta t0$           | = 1.0238e-6 |
| const       | = | 7.7570e-3  | $\Delta \text{const}$ | = 1.1187e-4 |

## Quantum yield determination

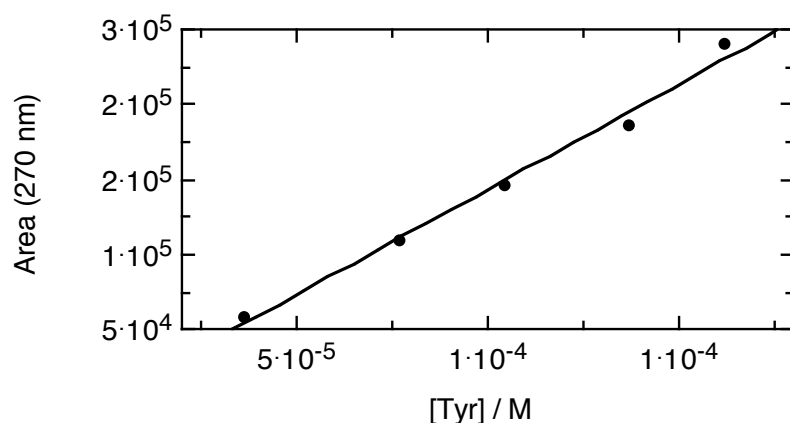
Quantum yields were determined using an optical bench consisting of a 0101 Schoeffel lamp housing equipped with a xenon-mercury lamp (150 W) and a 77250 Oriel monochromator. Irradiations were carried out in aerated water at 20 °C at 365 nm (band-width of 10 nm) and followed by HPLC analysis.

The photon flux was determined using potassium ferrioxalate actinometry (Hatchard – Parker actinometer):<sup>1</sup> A 3.0 mL solution of ferrioxalate (0.006 M) in H<sub>2</sub>SO<sub>4</sub> (0.05M) was irradiated and the moles of photoproduct were determined from the change in optical density at 510 nm and the extinction coefficient (11100 M<sup>-1</sup>cm<sup>-1</sup>) 1 h after addition of 0.1 % of 1,10-phenanthroline to the photolyzed solution. The known quantum yield of photoproduct (1.26)<sup>2</sup> was used to calculate the photon flux (I = 5.48x10<sup>-7</sup> E/min).

### 1. HPLC Analysis of Tyr.

Conditions: 70:30 acetonitrile:water (isocratic), 1 mL/min, detector at 270 nm.

Calibration curve:

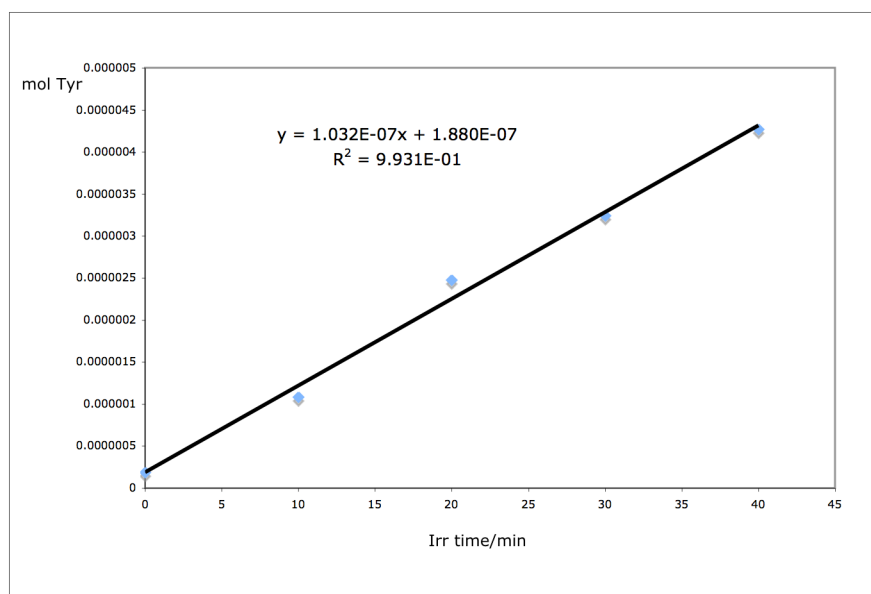


|       |             |        |             |
|-------|-------------|--------|-------------|
| const | = 3175.5518 | Δconst | = 9674.1127 |
| a1    | = 1.4036e+9 | Δa1    | = 8.6066e+7 |

Correlation coefficient: = 0.9944  
Significance of correlation: = 18.3480 %

## 2. Quantum yield determination for compound 7

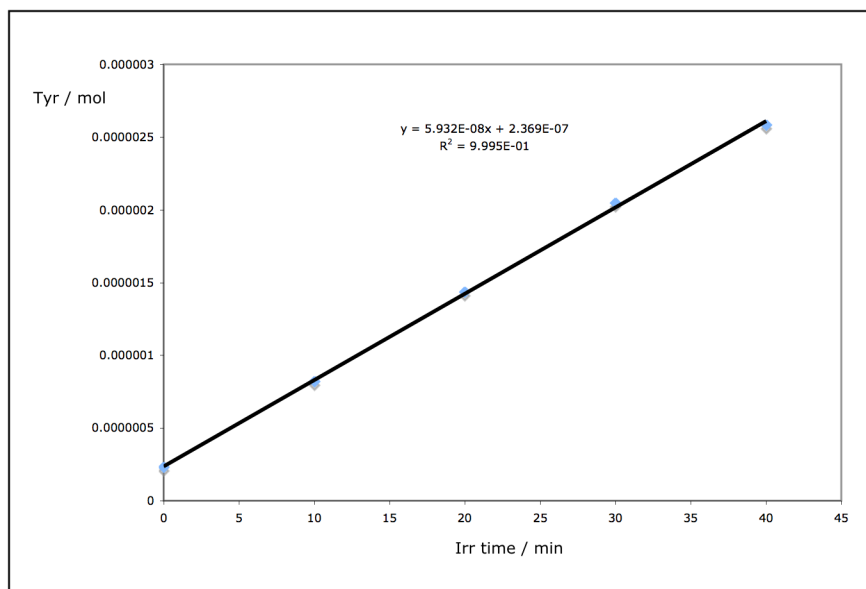
Solution in water at 1.389 mM, irradiated at 365 and analyzed at 10 min intervals.



Quantum yield = 0.19

## 3. Quantum yield determination for compound 13

Solution in water at 1.095 mM, irradiated at 365 and analyzed at 10 min intervals.



Quantum yield = 0.11

<sup>1</sup> Hatchard, C. G. ; Parker, C. A. *Proc. R. Soc.* **1956**, A235, 518-536.

<sup>2</sup> Scaiano, J. C. *Handbook of Organic Photochemistry*, Vol. I, CRC Press, Florida, **1989**.

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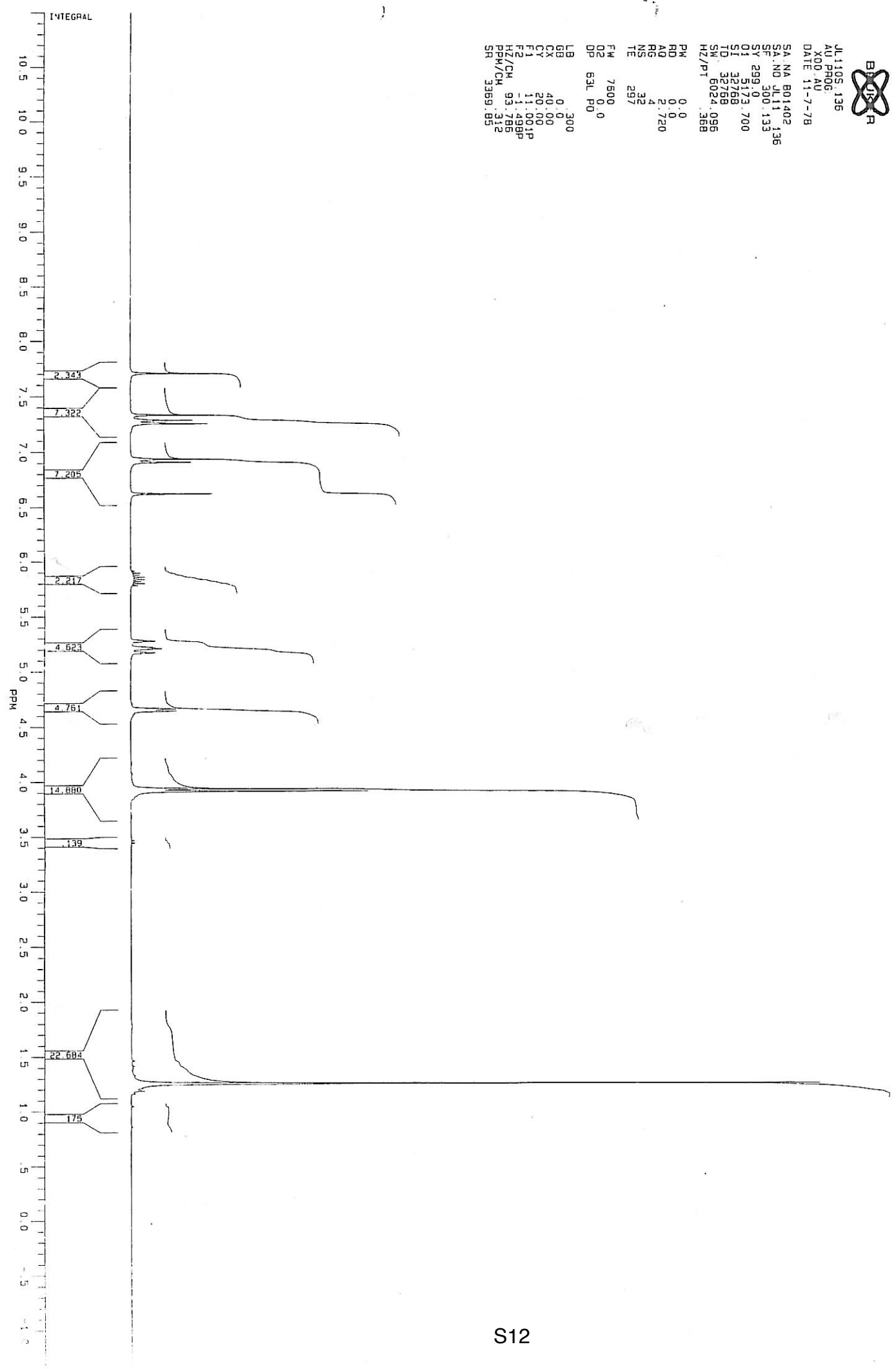
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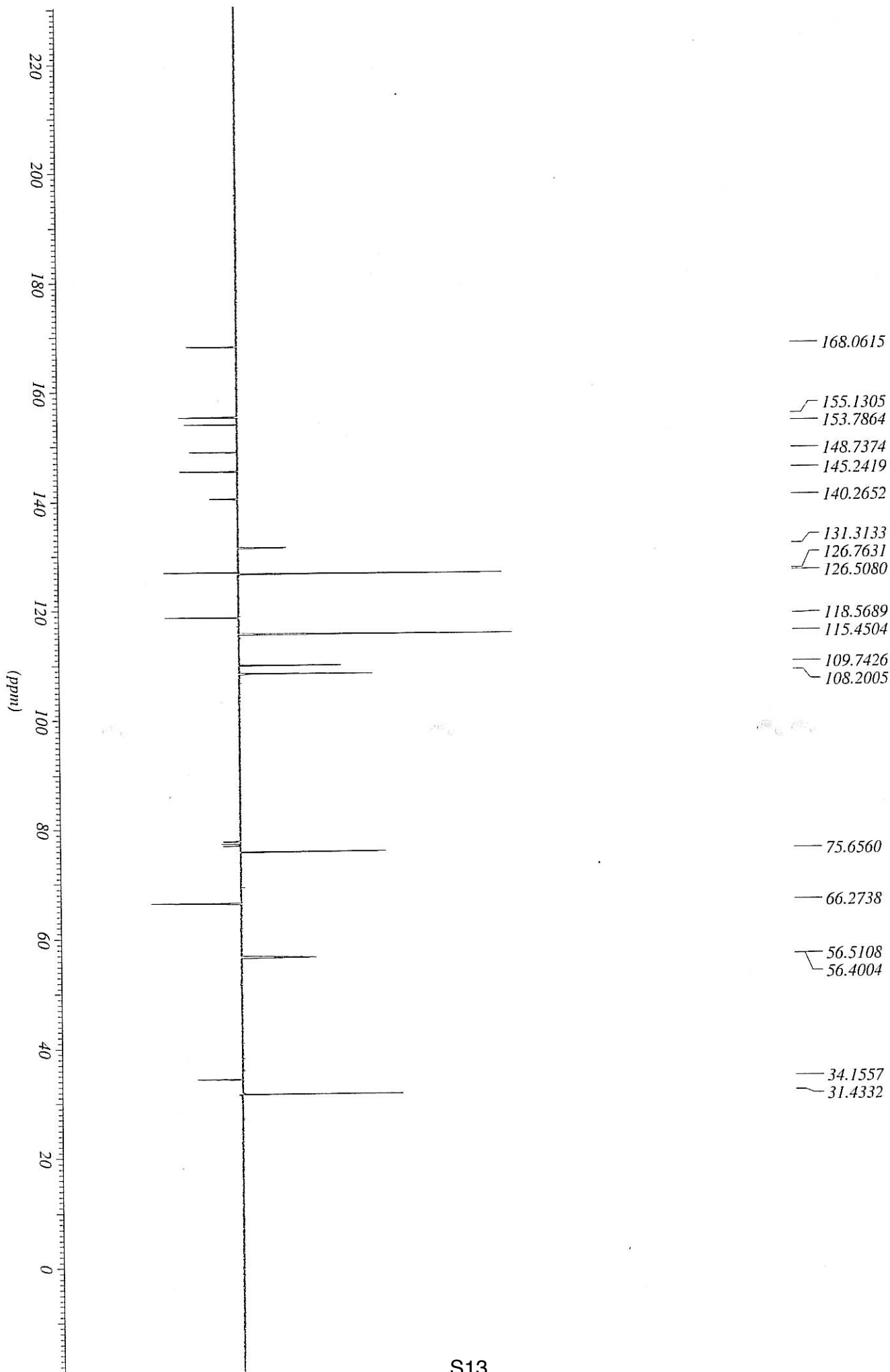
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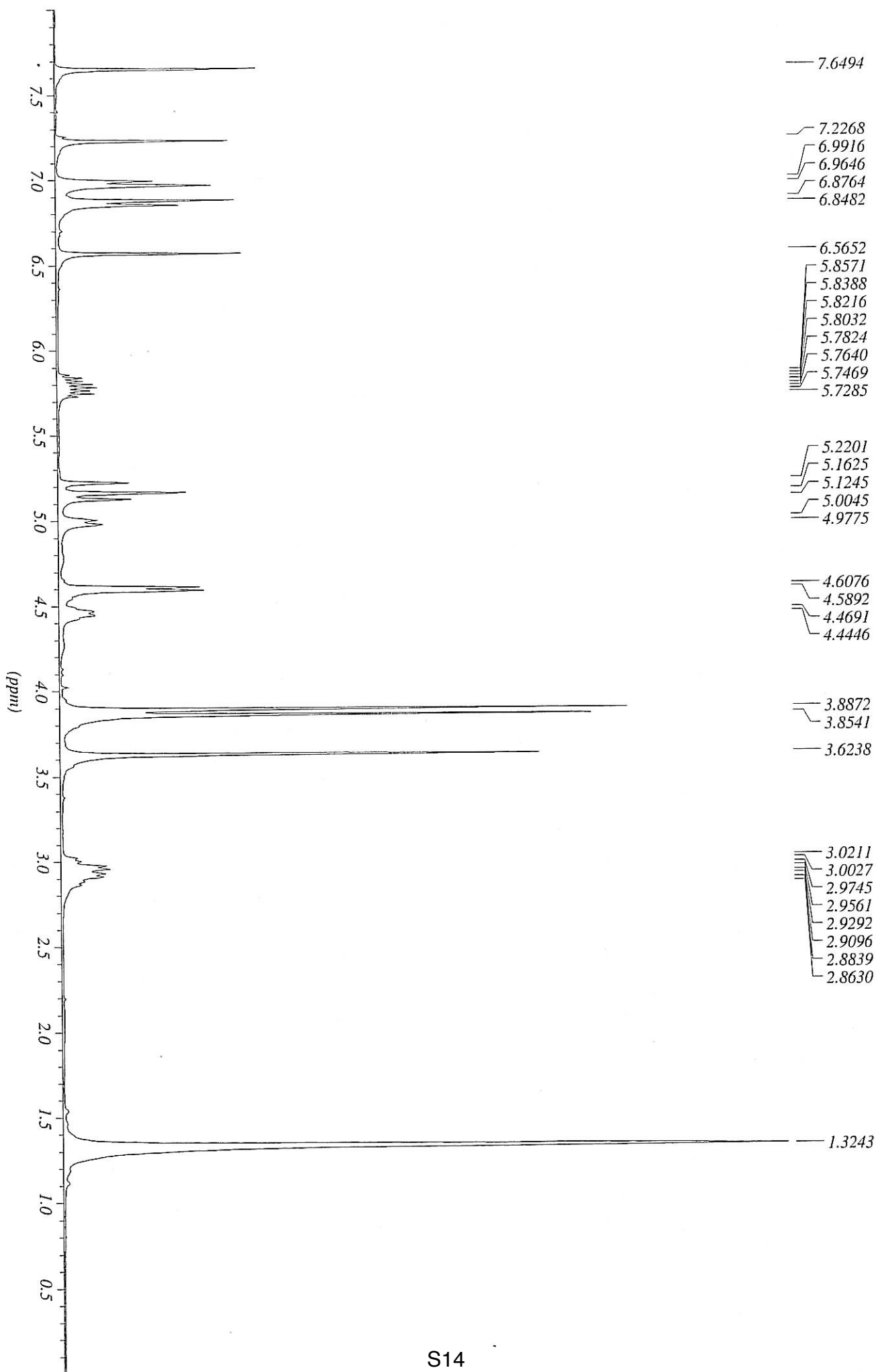
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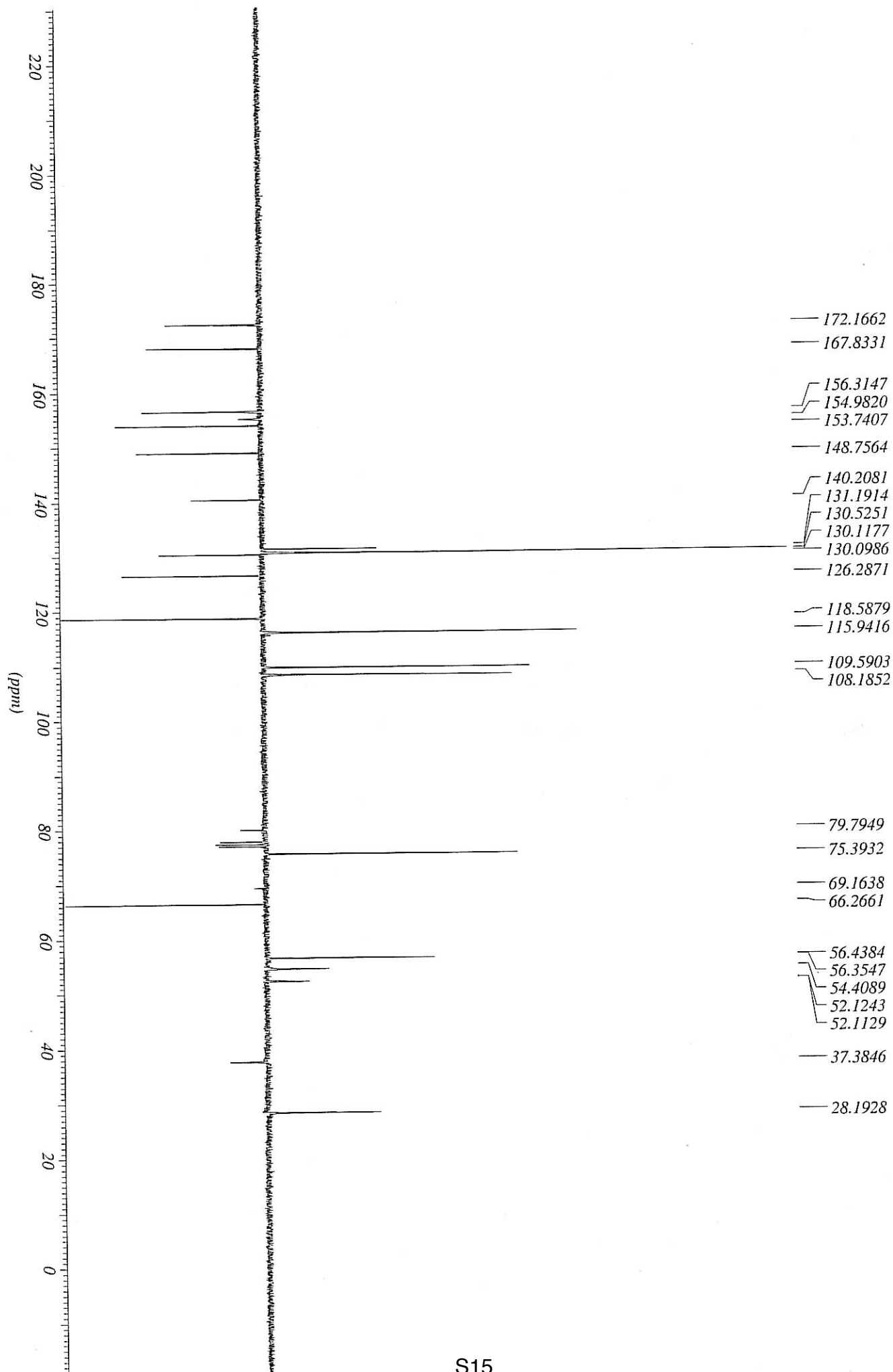
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SR 3369.85









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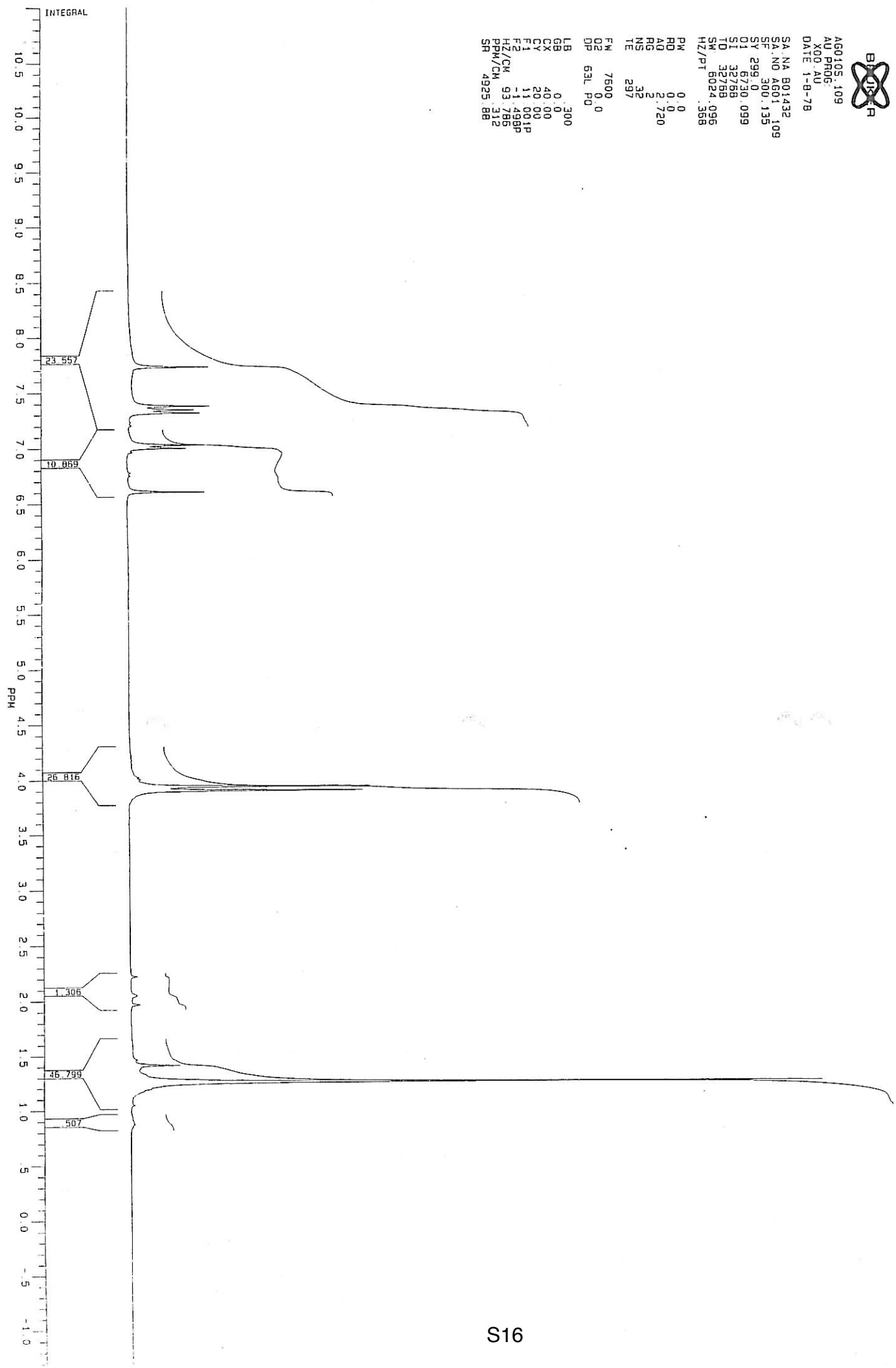
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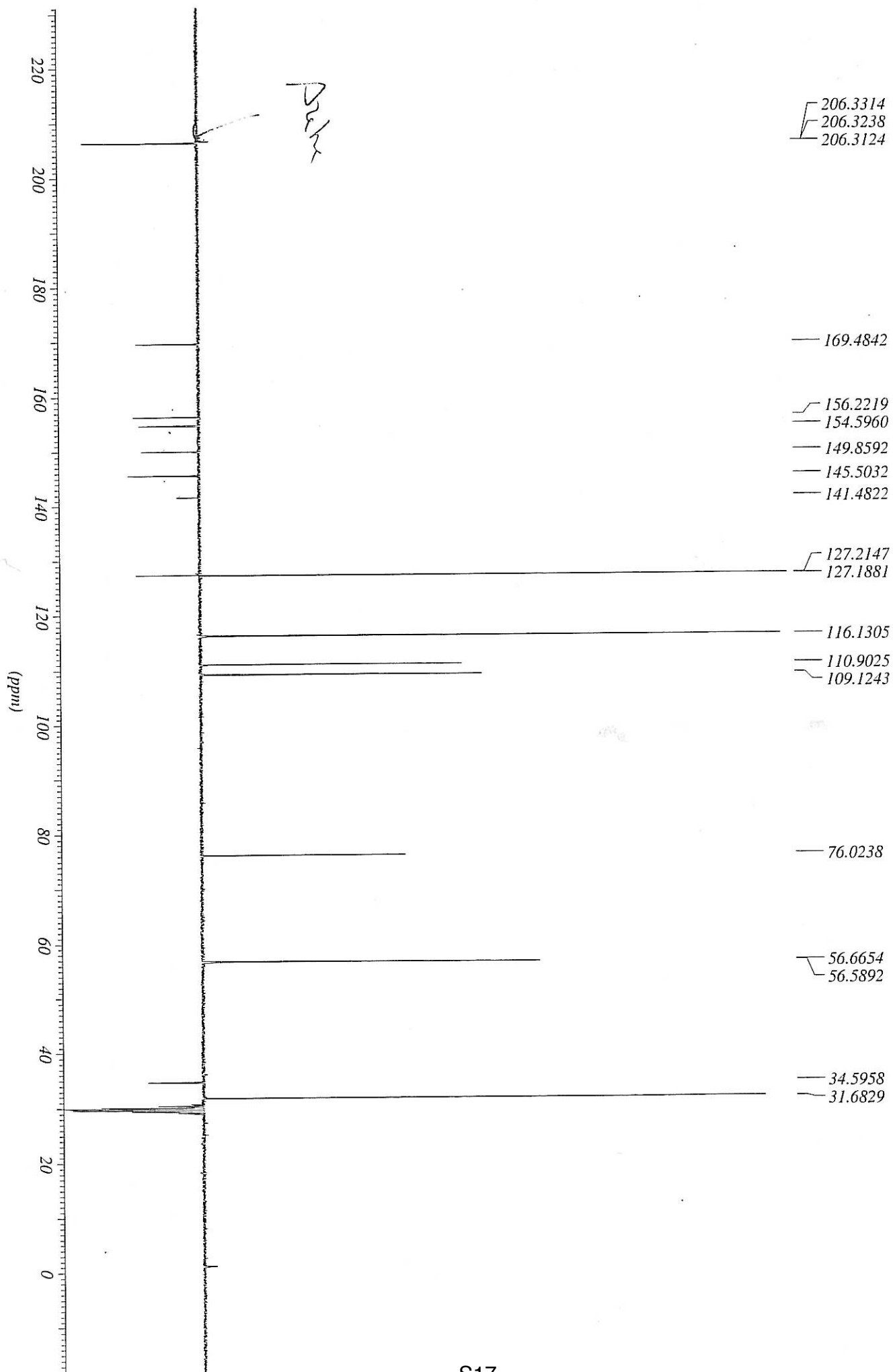
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D...-...-...

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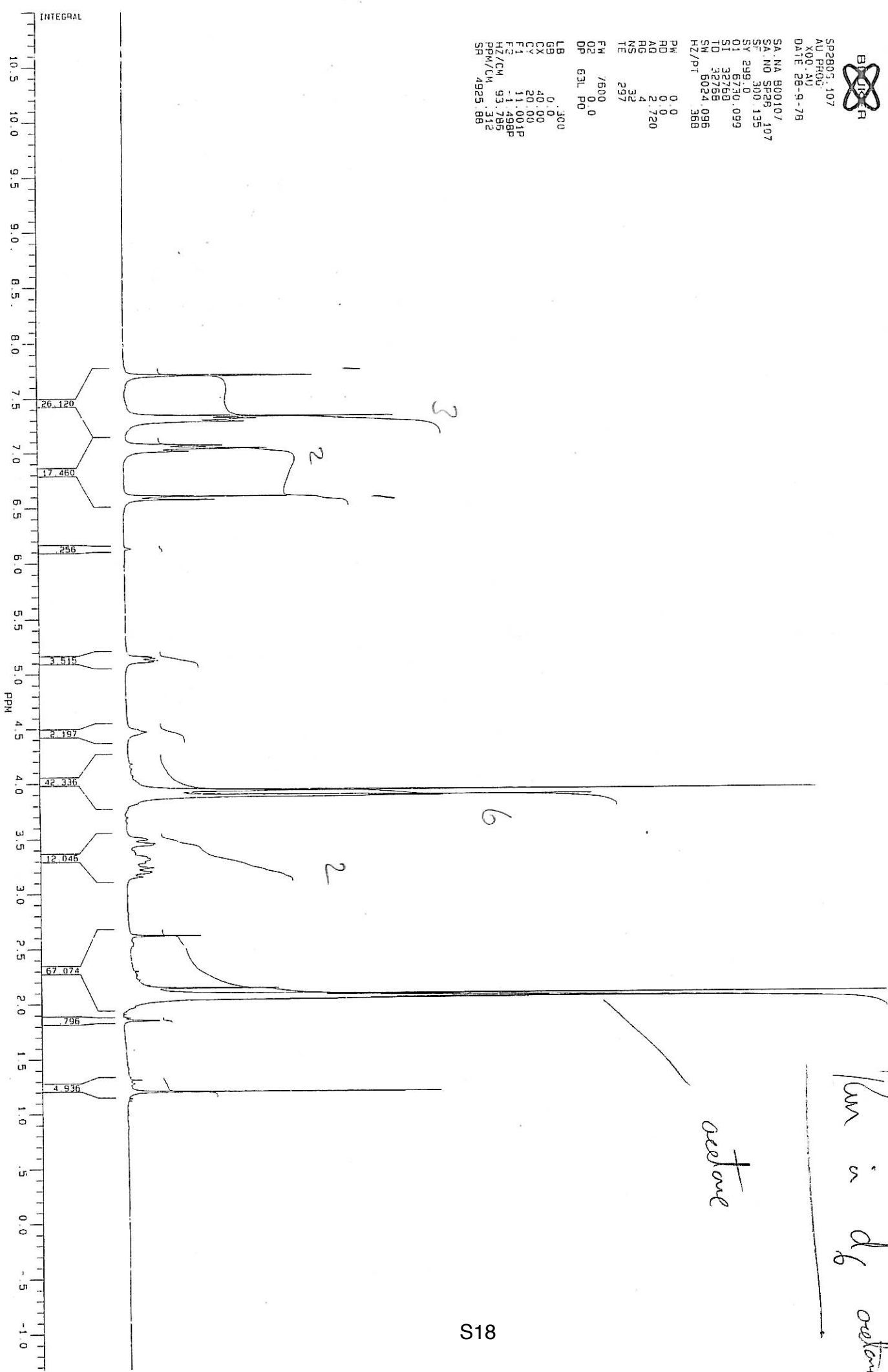
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SR 4925.88



Run in d<sub>6</sub> acetone

acetone

Alex Fossati, 06-acetone, +270, AV300  
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 Barcode label 1415

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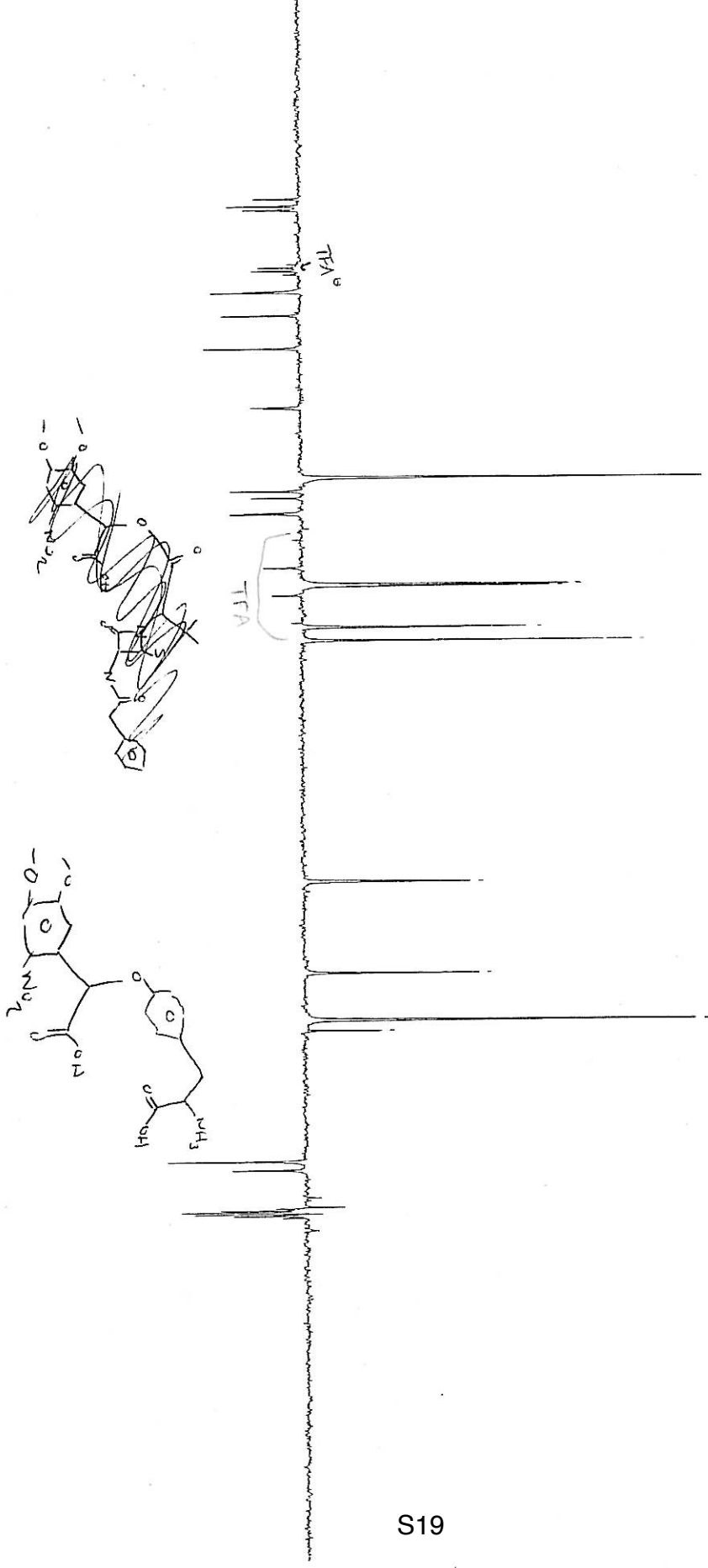
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74 + 512

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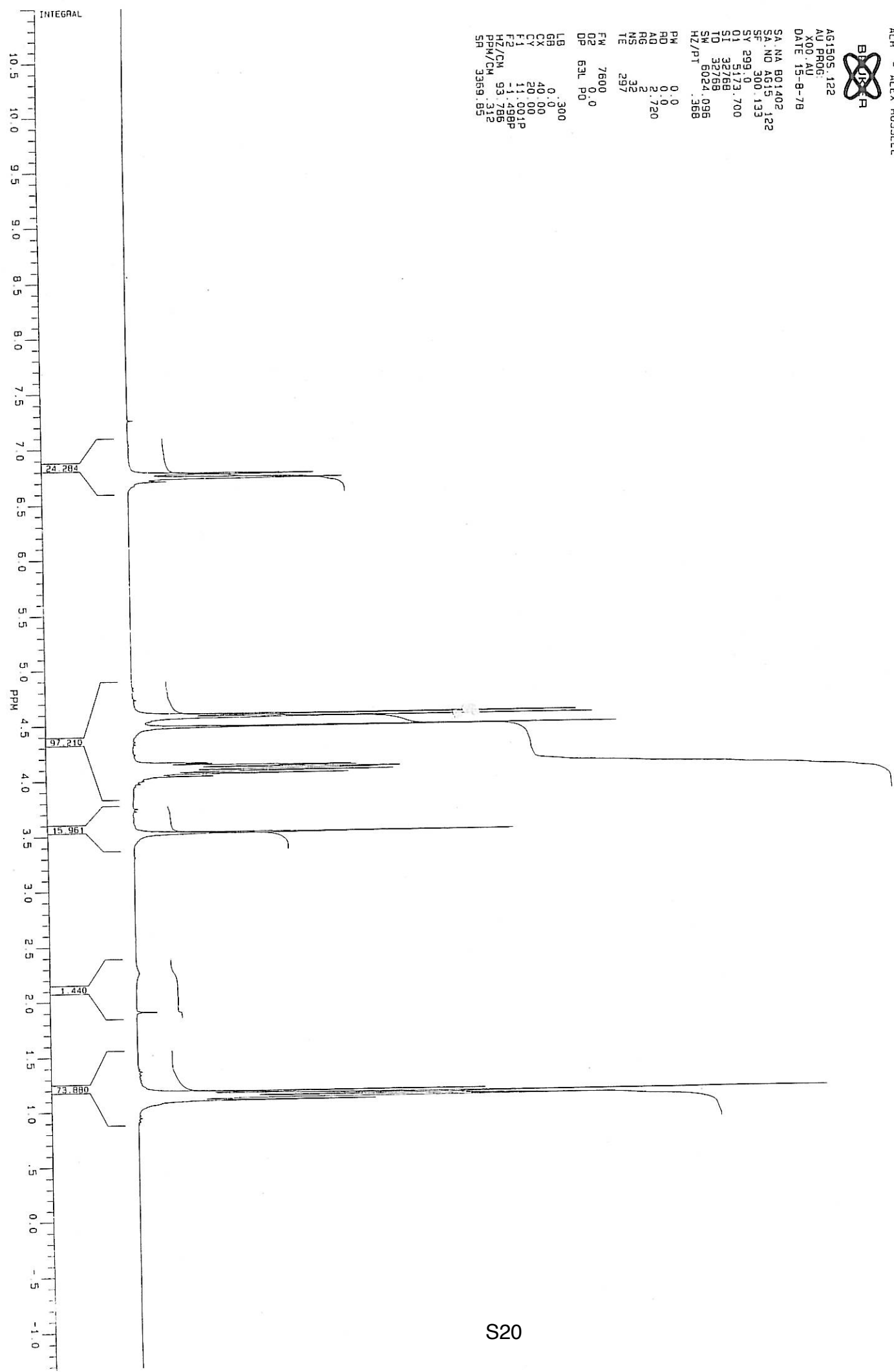
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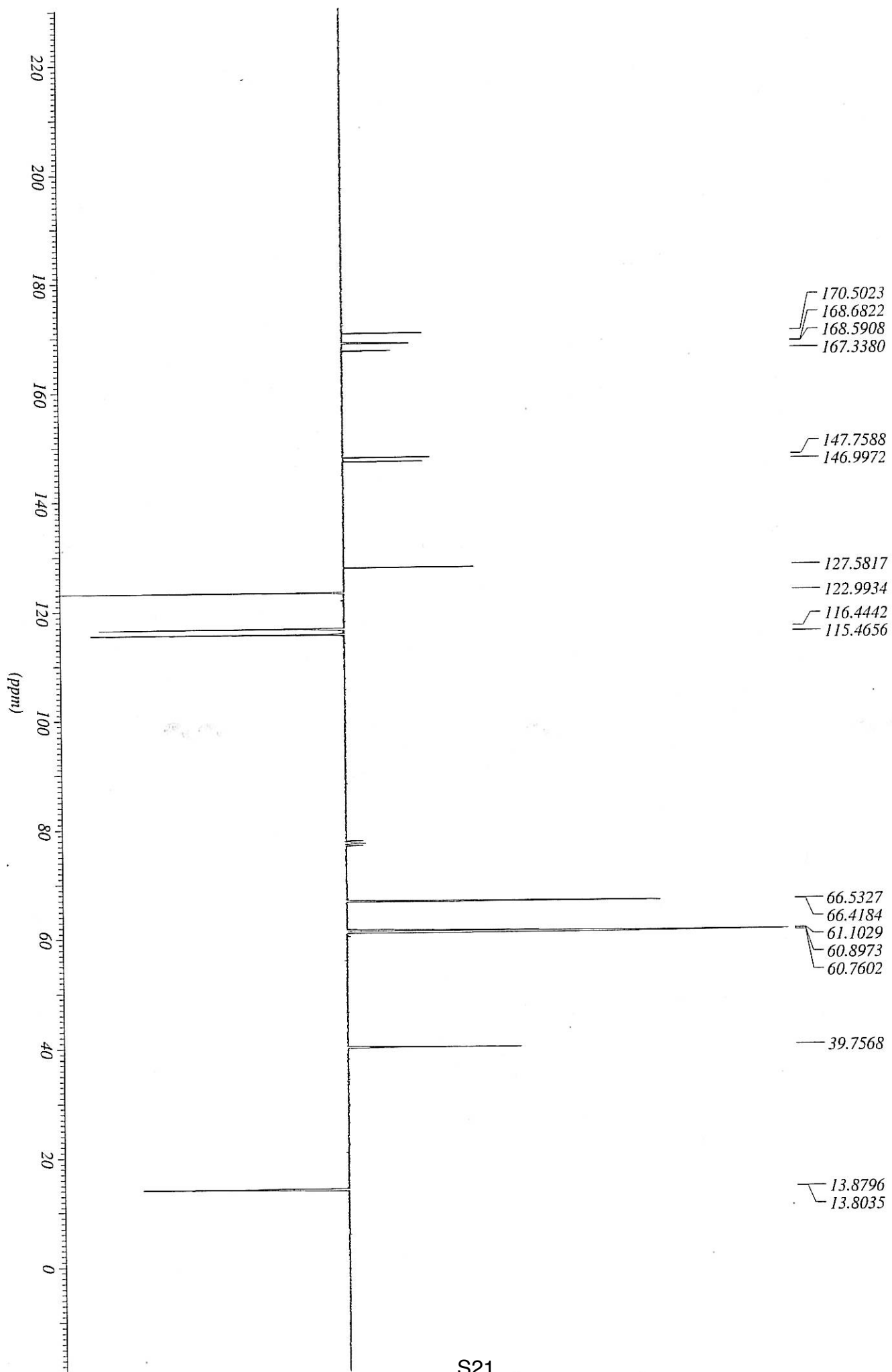
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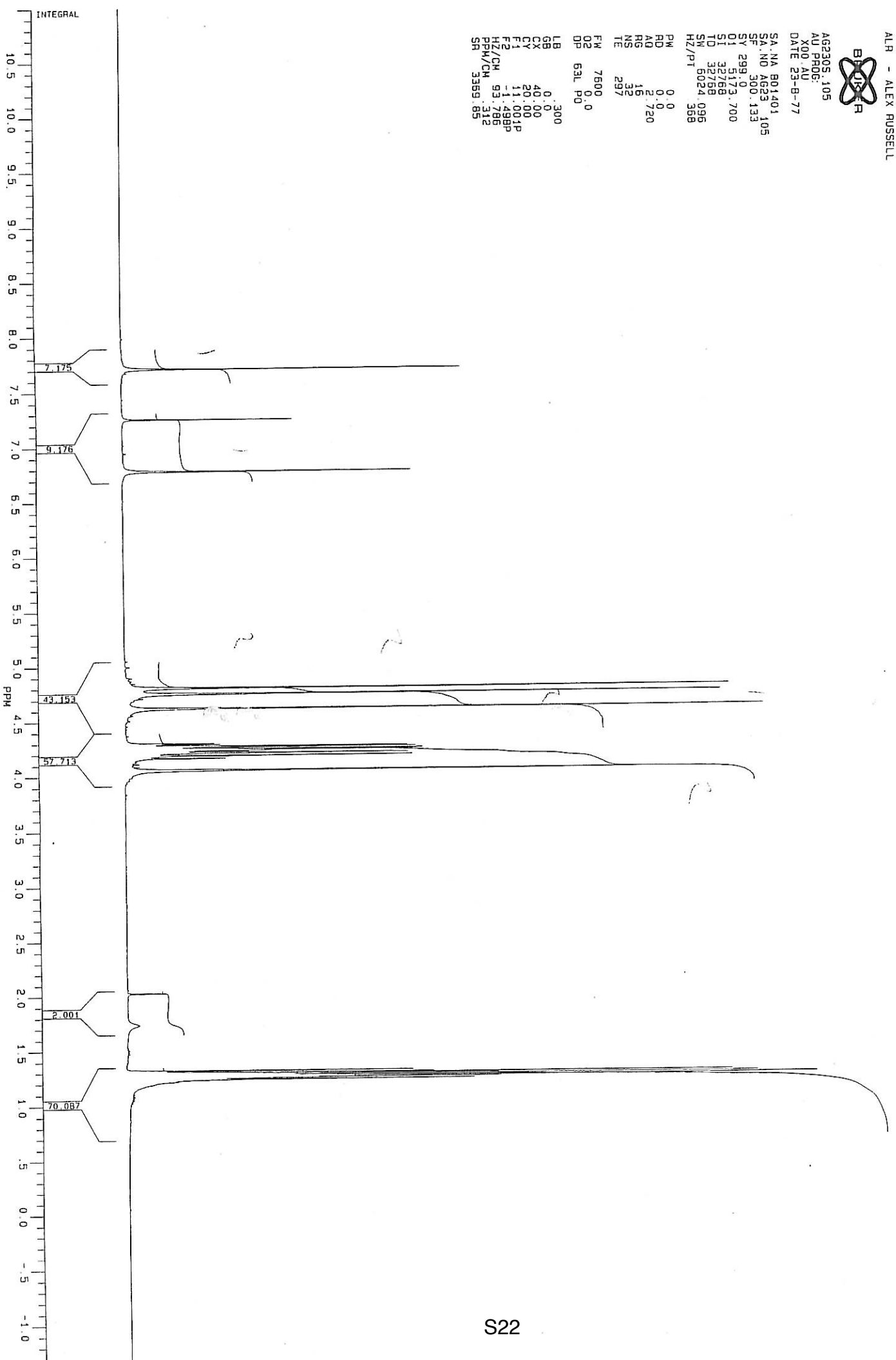
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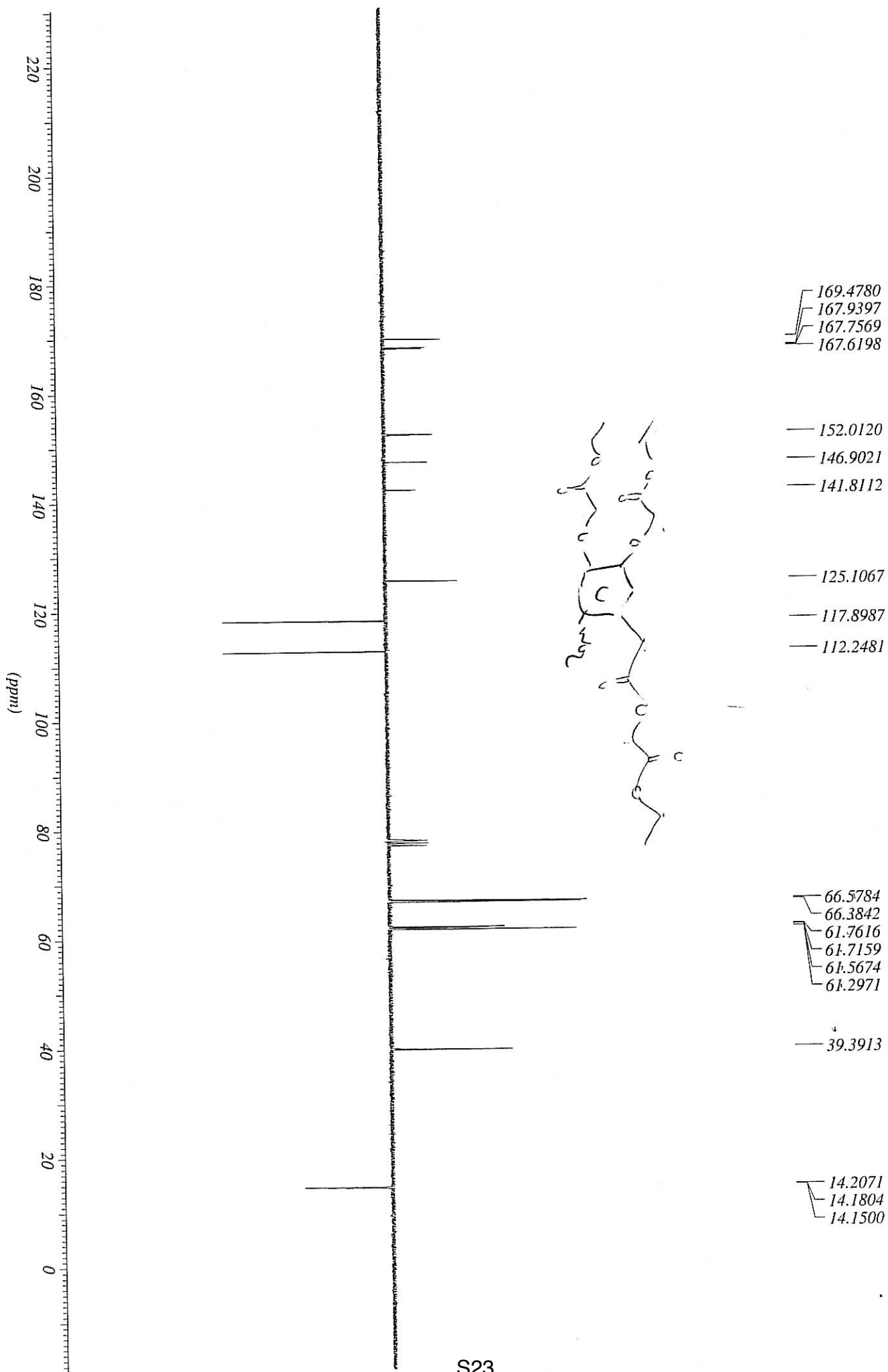
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SR 3359.85





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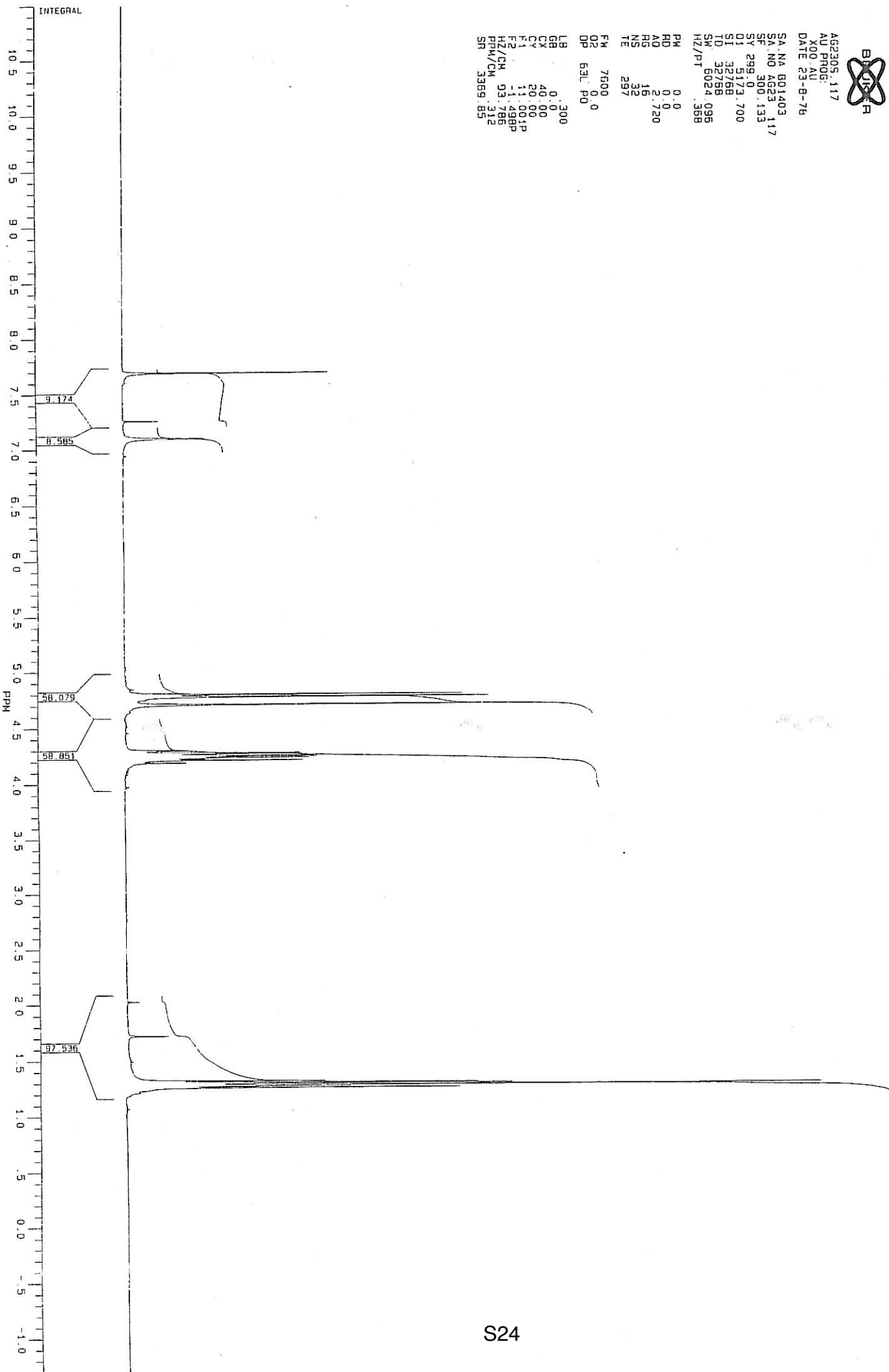
AG2305\_117  
AU PROG:  
X00 AU  
DATE 23-8-78

SA. NA. B01403  
SA. NO. AG23 117  
SF. 300. 133  
SY. 299. 0  
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Q1. 32168  
T1. 32168  
S1. 32168  
SM. 6024. 095  
HZ/PT. 388

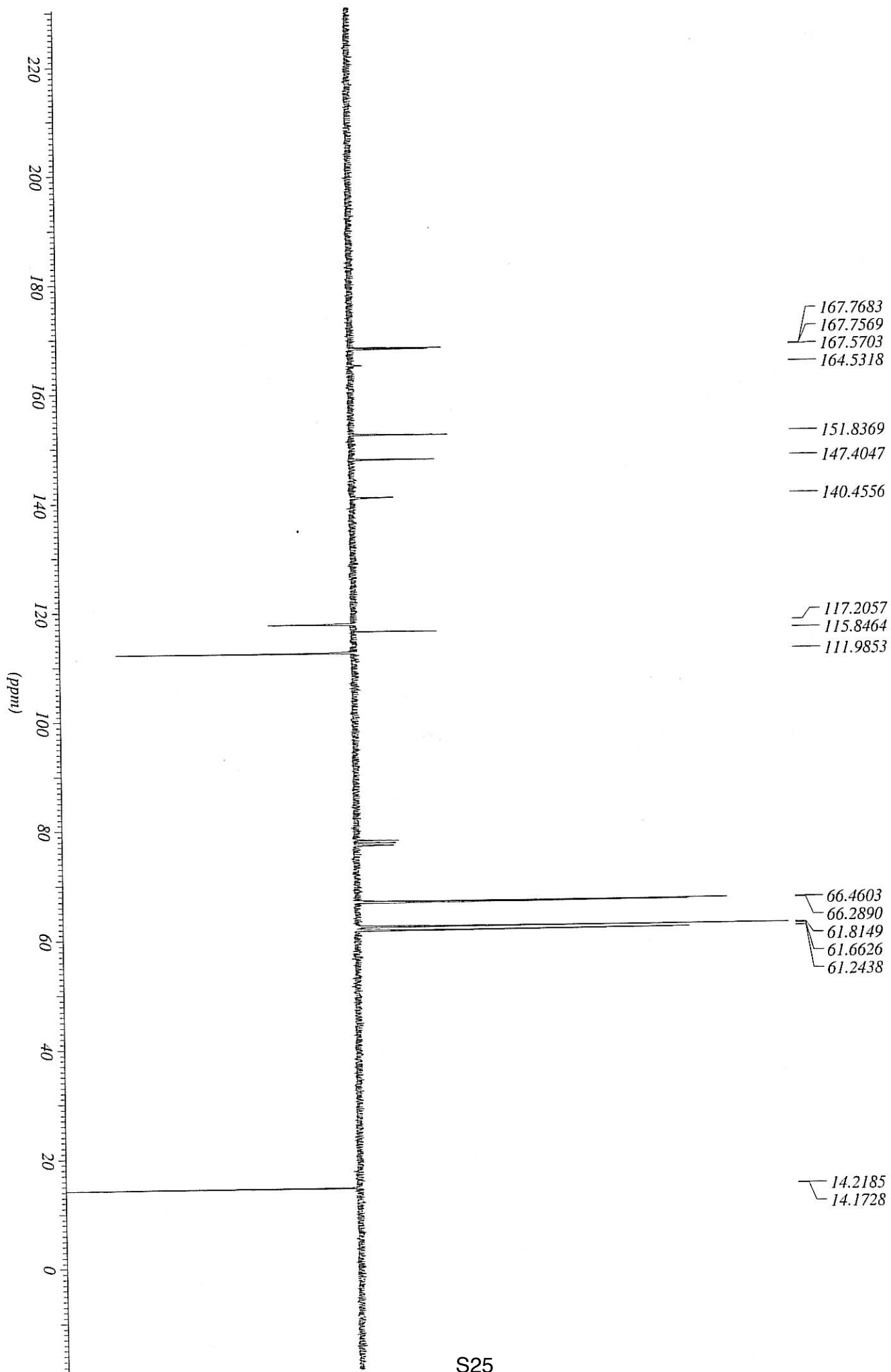
PH. 0. 0  
RD. 0. 0  
AD. 2. 720  
NS. 15  
NS. 32  
NS. 297

FM. 7500  
Q2. 0. 0  
DP. 63L P0

LB. 0. 300  
CB. 0. 0  
C2. 40. 00  
C1. 20. 00  
F1. 11. 00  
F2. 11. 00  
R2/CM. 93. 785  
R1/CM. 93. 312  
SM. 3389. 85







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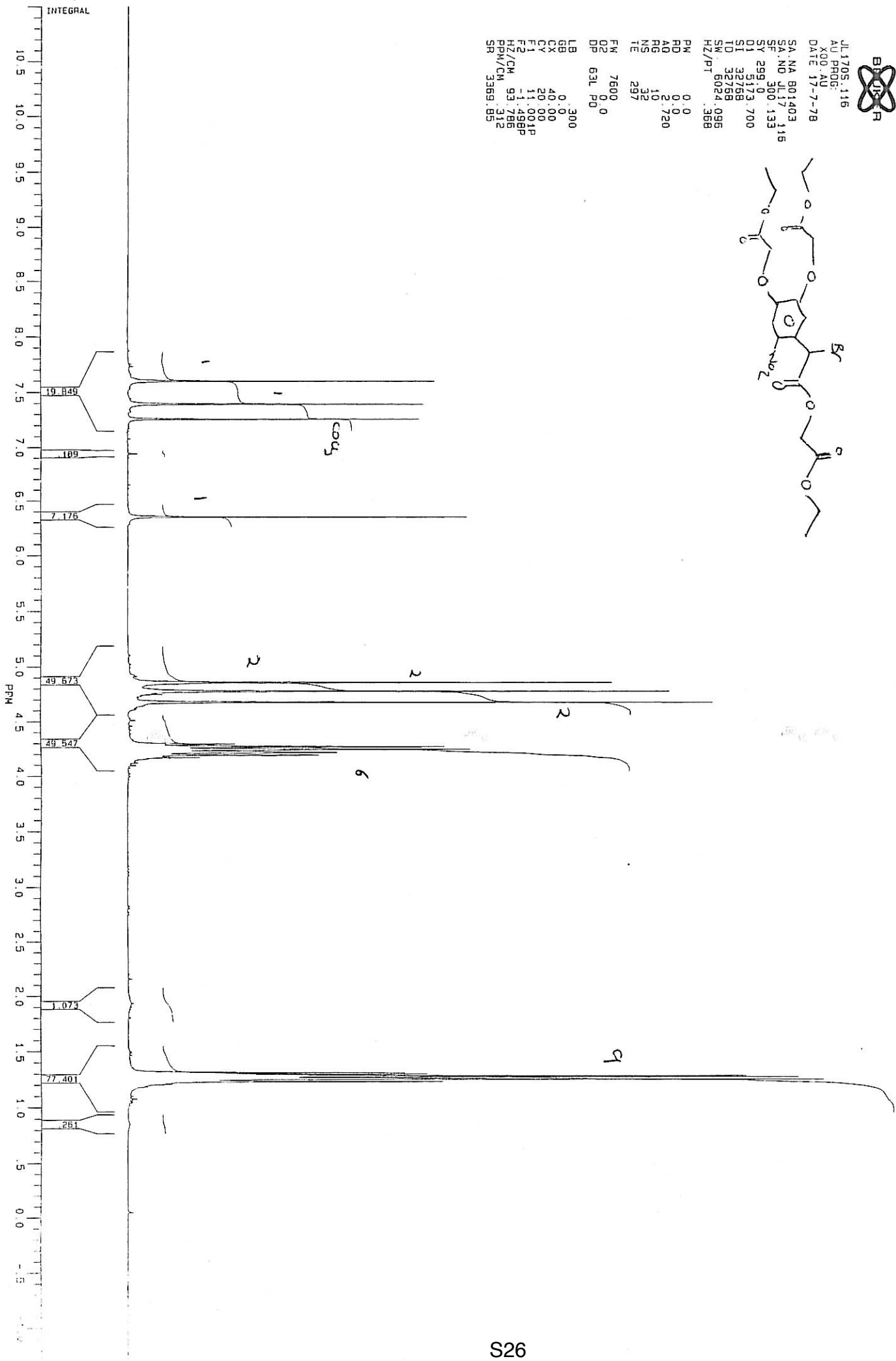
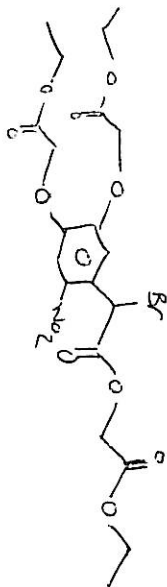
JL1705.116  
 AU PROG: X00.AU  
 DATE 17-7-78

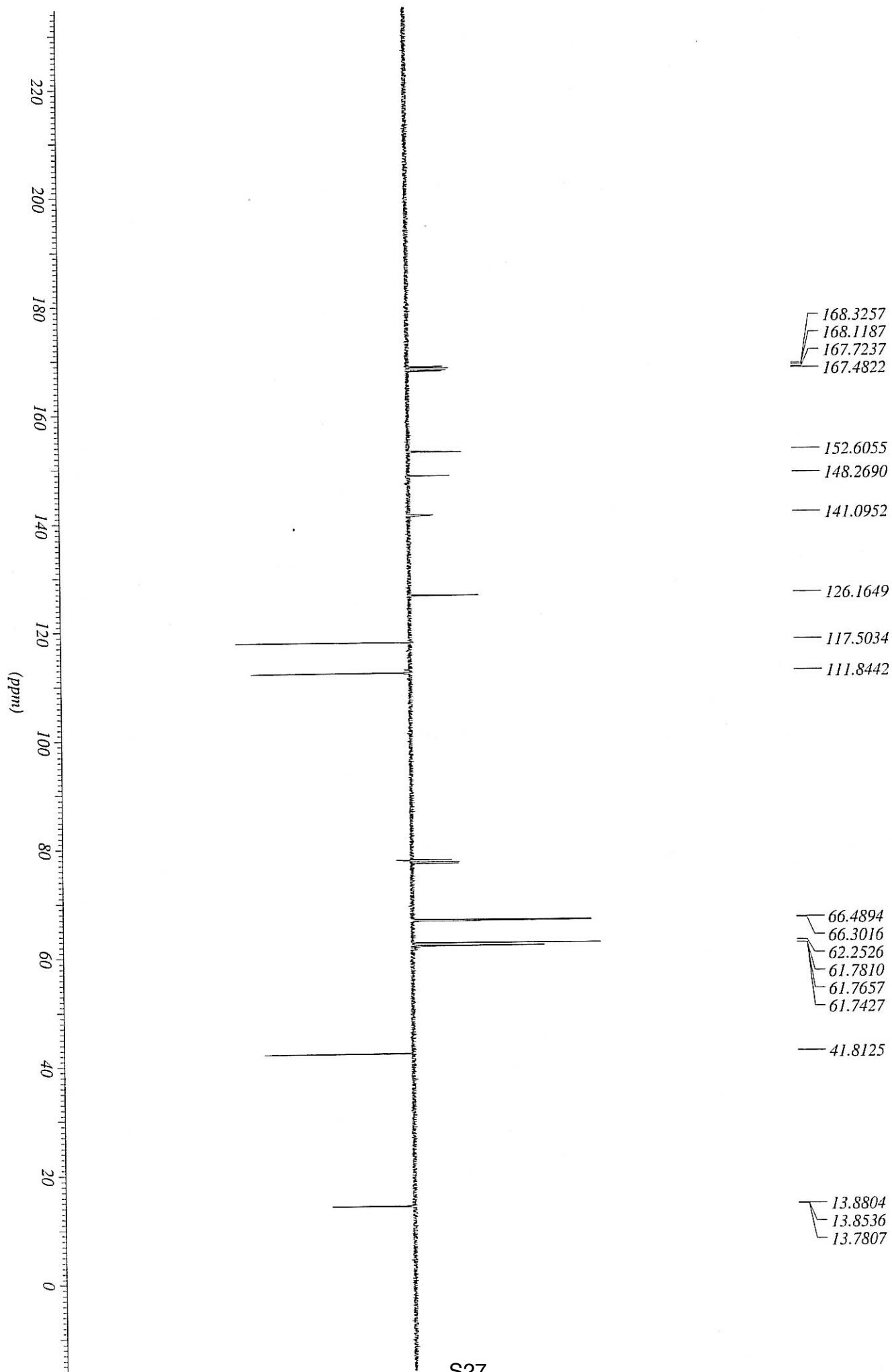
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 SA.NO JL17 116  
 SF 300.133  
 SY 299.0  
 O1 5173.700  
 S1 32758  
 TD 32758  
 SW 6024.095  
 HZ/PT .368

PW 0.0  
 RD 0.0  
 AG 2.720  
 RG 10  
 NS 32  
 TE 297

FW 7600  
 D2 0.0  
 DP 63L PD

LB 300  
 GB 0  
 CX 40.00  
 CY 20.00  
 F1 11.001P  
 F2 -1.498P  
 HZ/CM 93.785  
 PPM/CM 312  
 SR 3369.85





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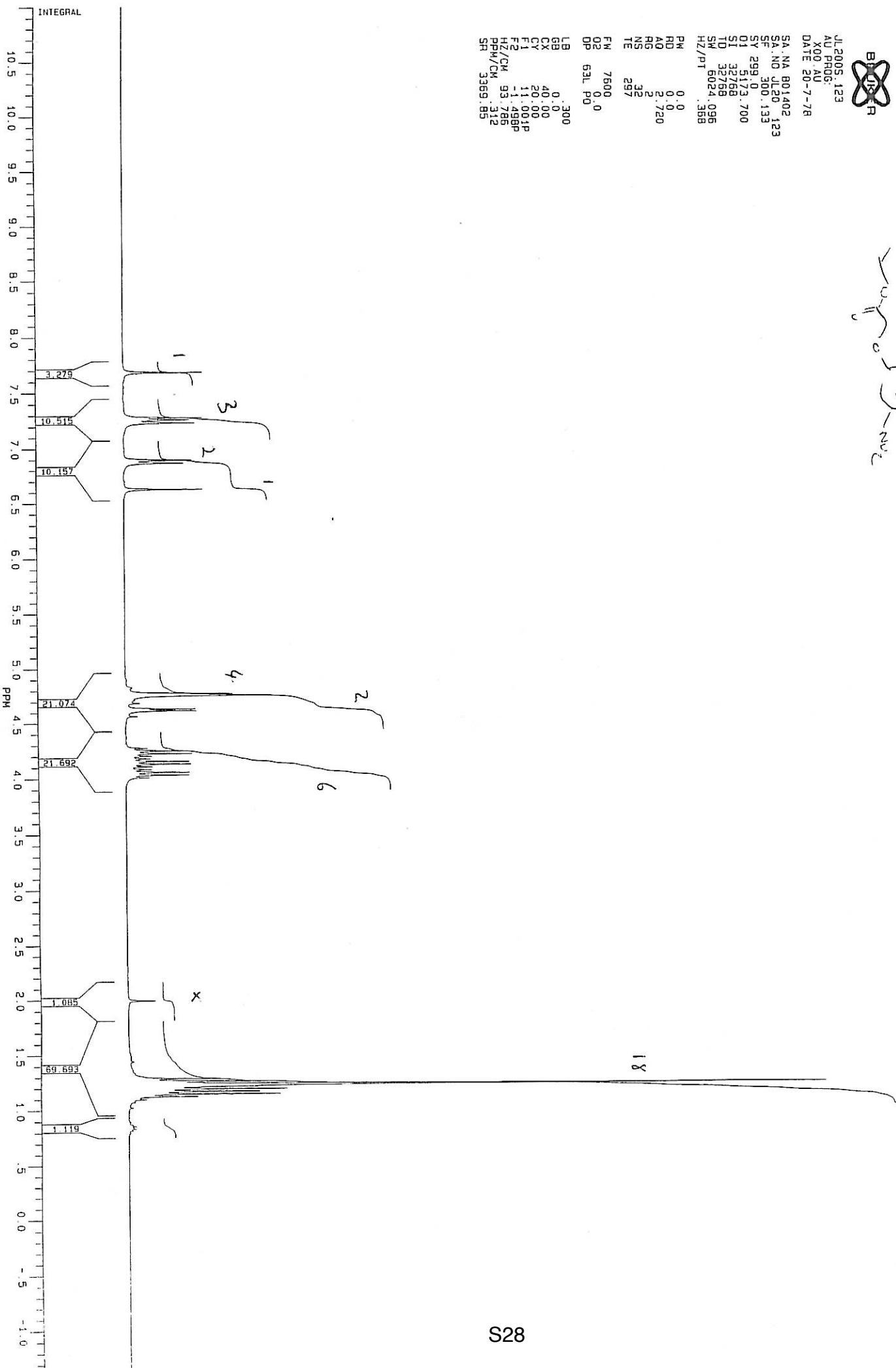
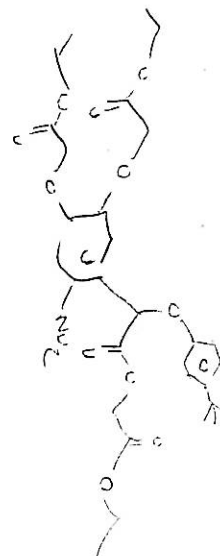


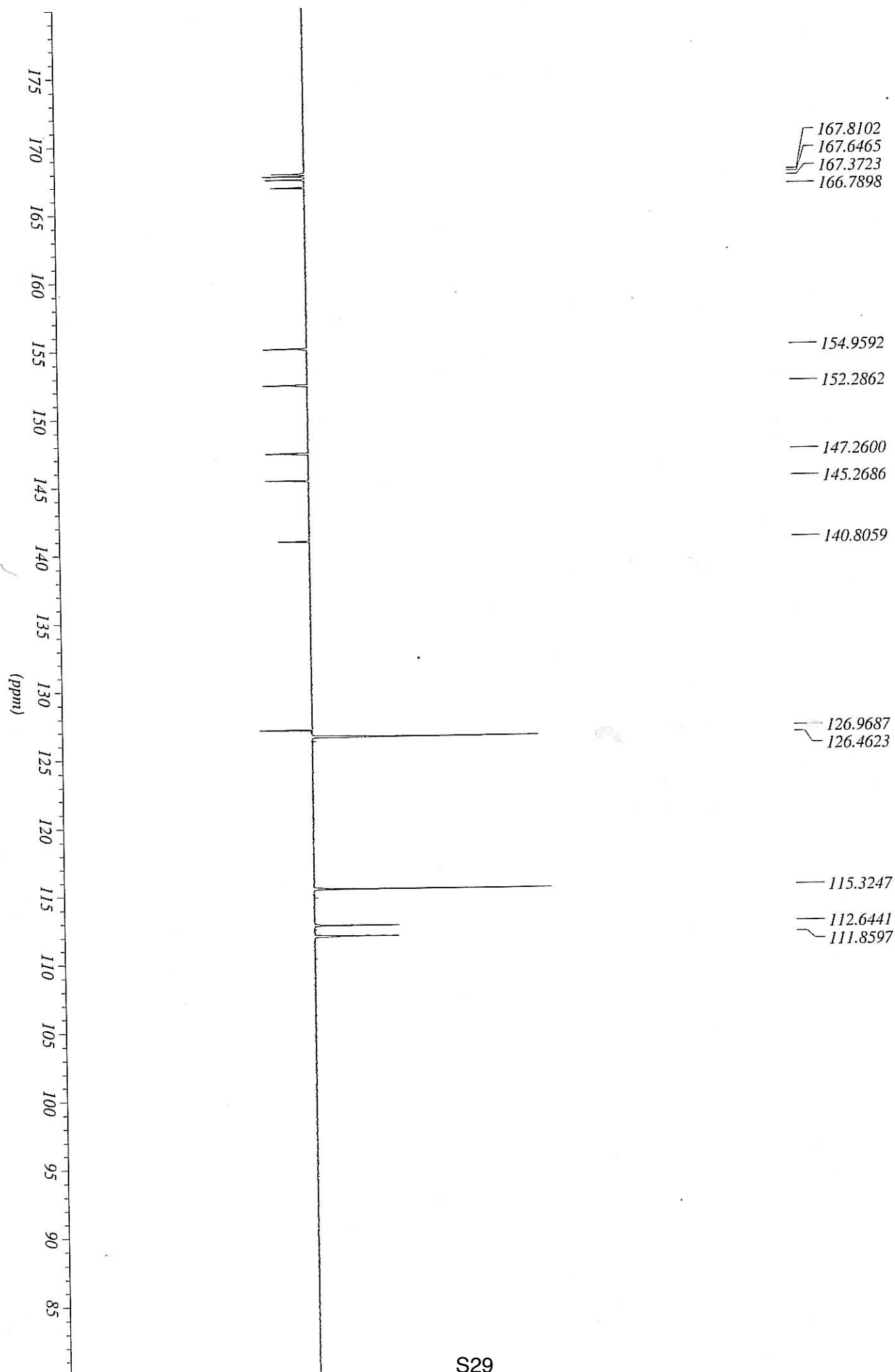
JL2005.123  
 AU PROJ:  
 X00 AU  
 DATE 20-7-78

SA:NA B01402  
 SA:NO JL20.123  
 SF 300.133  
 SY 299.0  
 O1 3173.700  
 S1 32768  
 TD 32768  
 SW 6024.096  
 HZ/PT .368

PW 0.0  
 RD 0.0  
 AQ 2.720  
 RG 2  
 NS 32  
 TE 297  
 FW 7600  
 O2 0.0  
 DP 63L PQ

LB 0.300  
 GB 0.0  
 CX 40.00  
 CY 20.00  
 F1 11.001P  
 F2 -1.488P  
 HZ/CM 93.785  
 PPM/CM .312  
 SR 3369.85





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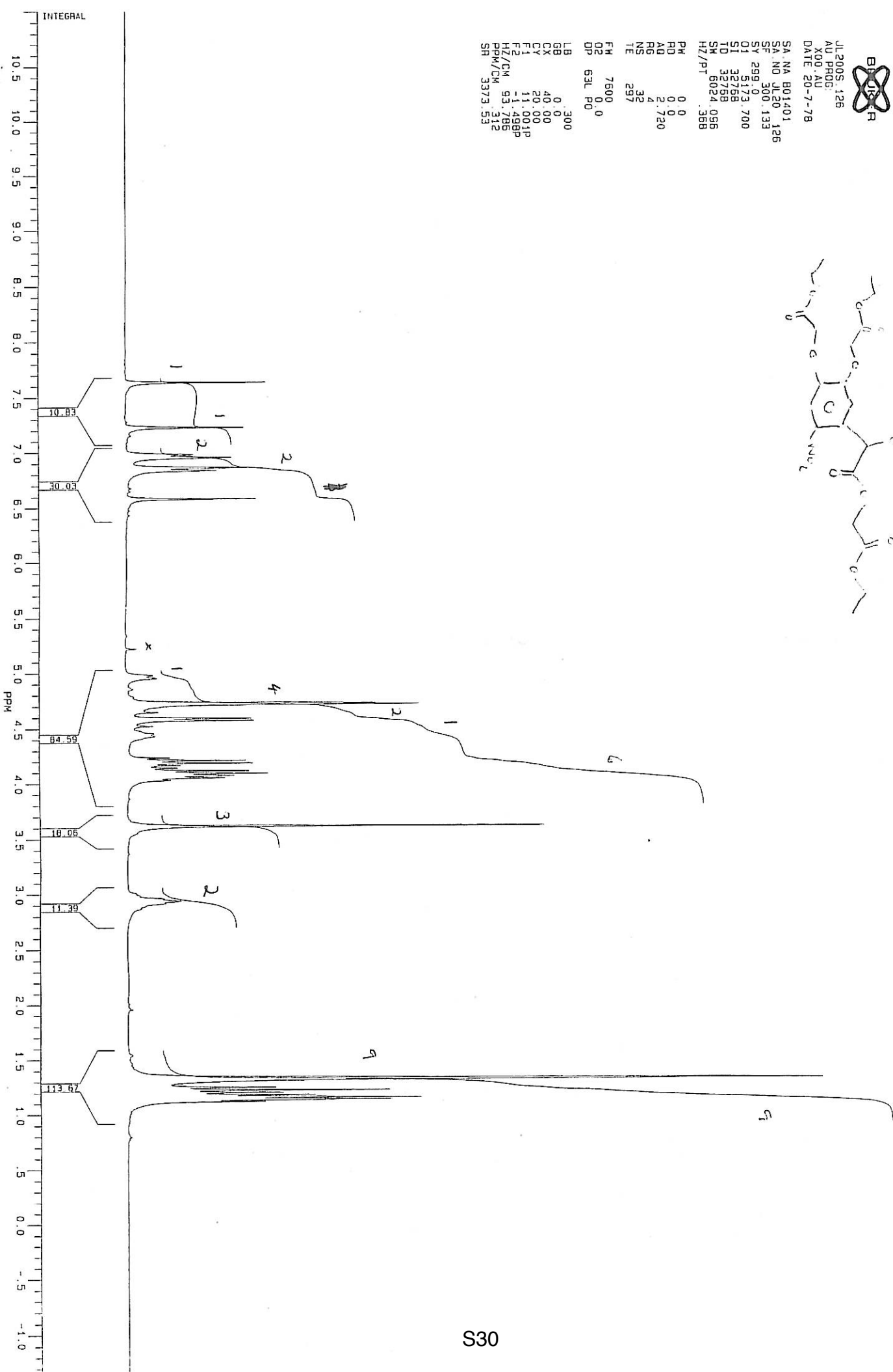
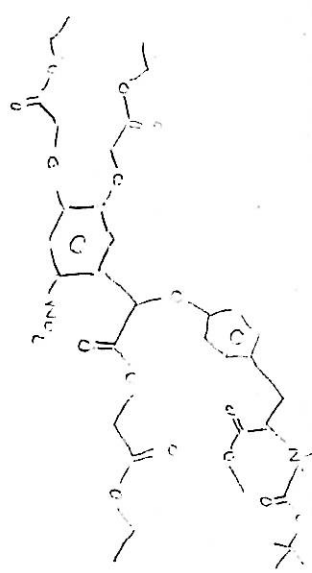
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 AU PROG:  
 X00.AU  
 DATE 20-7-78

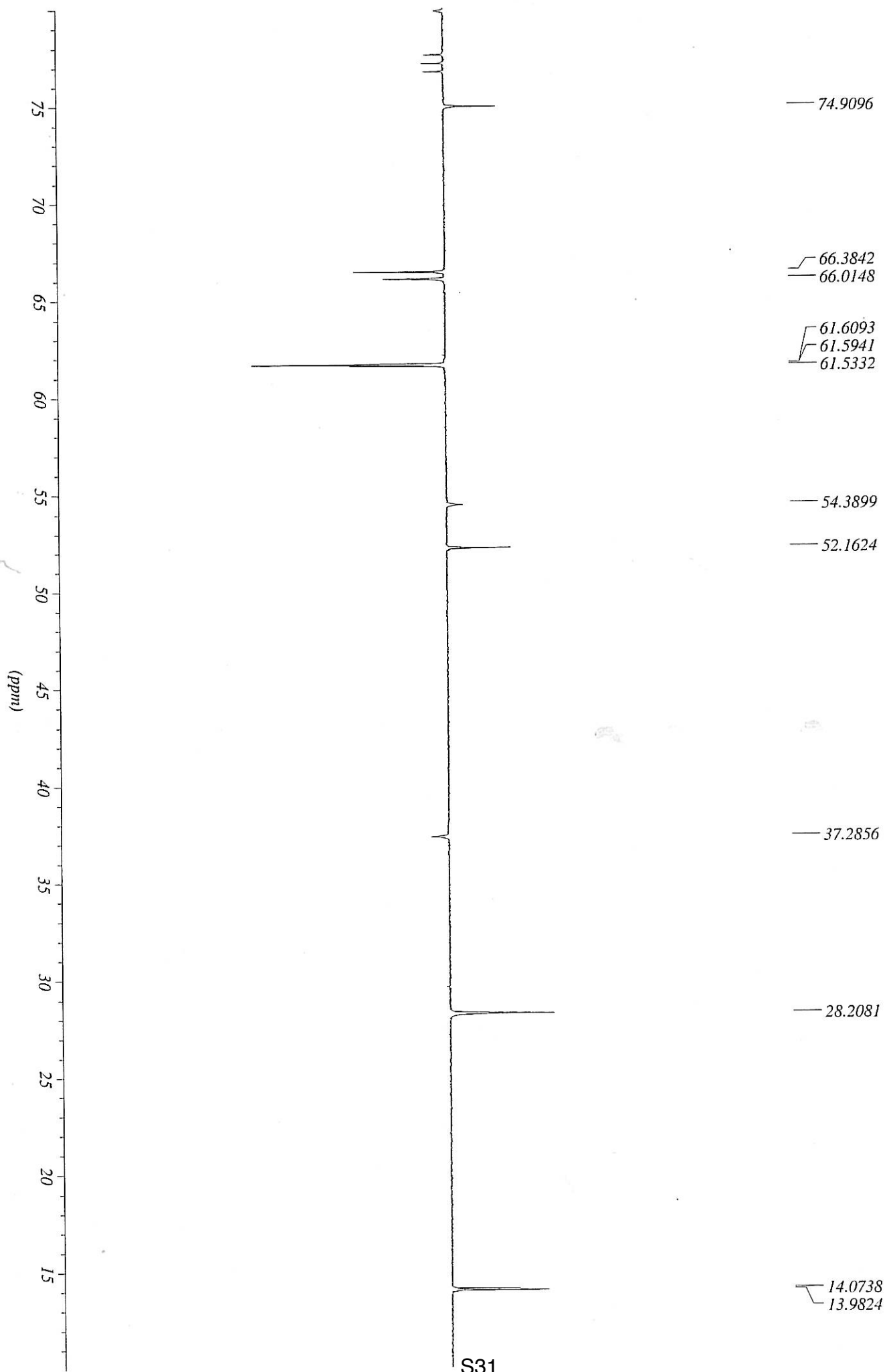
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 SA NO JL20.126  
 SF 300.133  
 SY 299.0  
 O1 5173.700  
 S1 32768  
 TD 32768  
 SM 6024.096  
 HZ/PT .368

PM 0 0  
 RD 0 0  
 AD 2.720  
 RG 4  
 NS 32  
 TE 297

FH 7600  
 O2 0.0  
 DP 63L P0

LB .300  
 GB 0 0  
 CX 40 00  
 CY 20 00  
 F1 11.001P  
 F2 -1.498P  
 HZ/CM 93.785  
 PPM/CM 312  
 SR 3373.53





ALR - ALEX RUSSELL



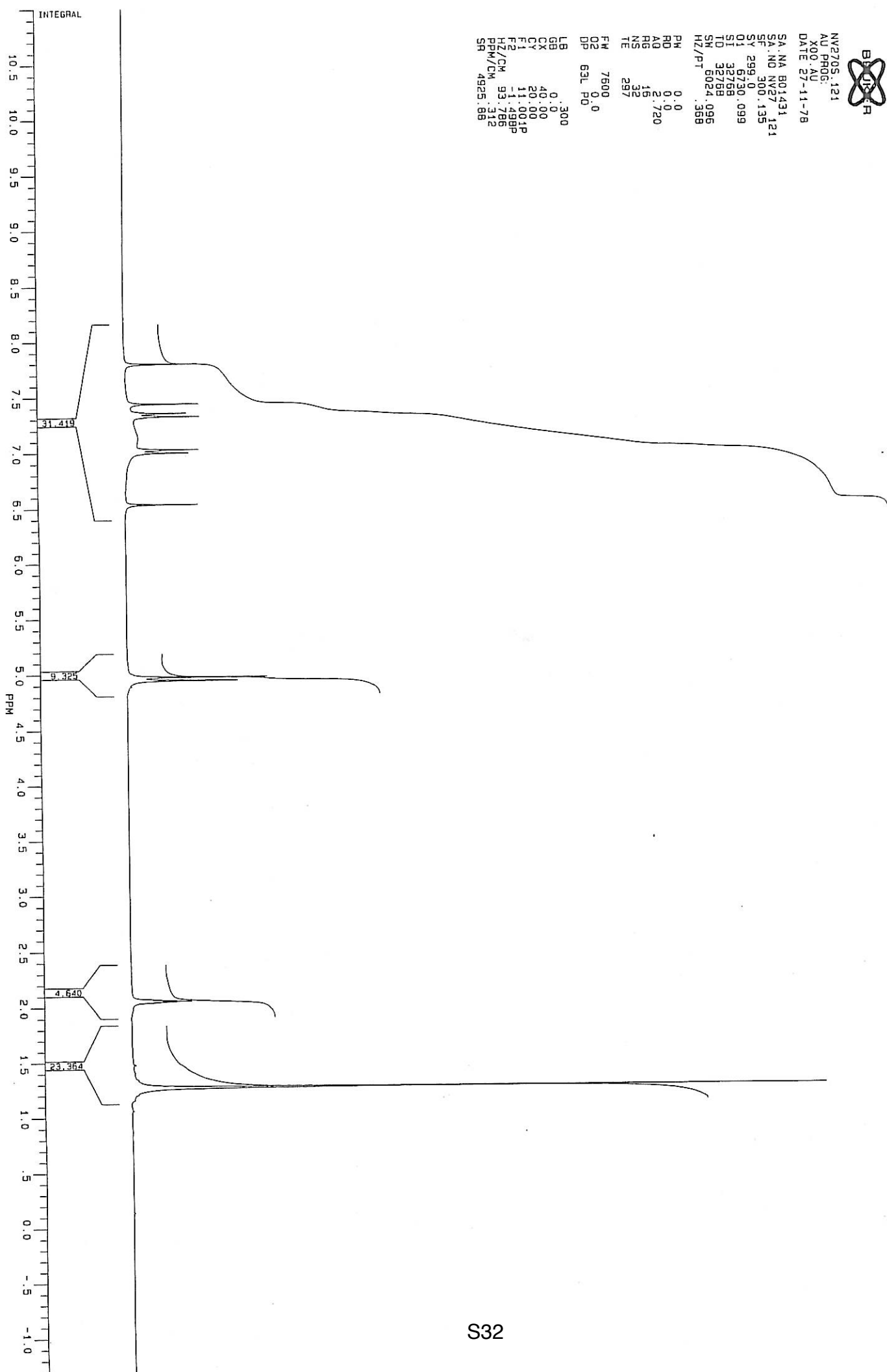
NV2705.121  
AU PROG:  
X00.AU  
DATE 27-11-78

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SA .ND NV27 .121  
SE 300.135  
SY 299.10  
Q1 6730.099  
S1 32768  
T0 32768  
SN 6024.096  
HZ/PT .368

PM 0.0  
RD 0.0  
AQ 2.720  
RG 16  
NS 32  
TE 297

FW 7600  
Q2 0.0  
DP 63L PD

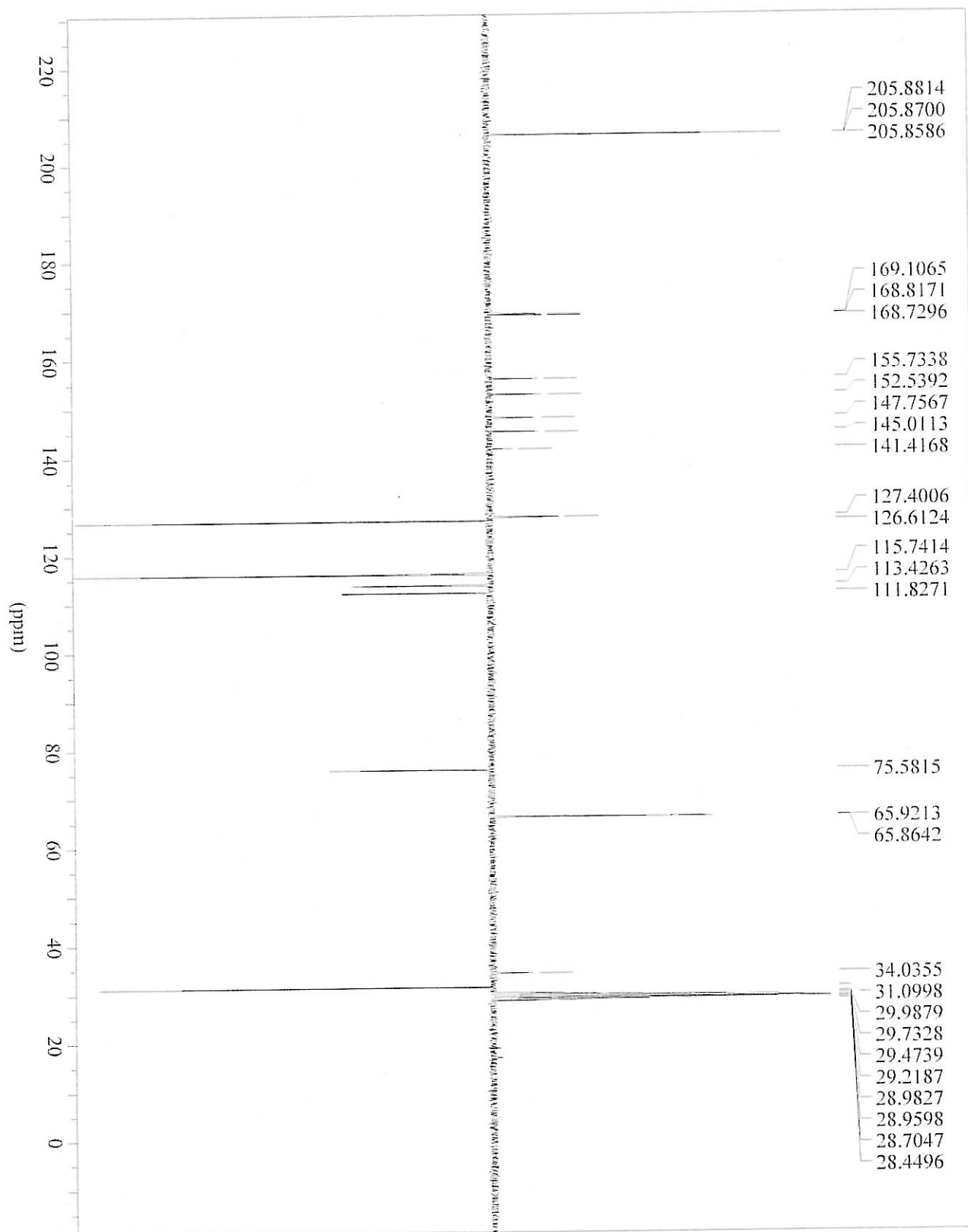
LB .300  
GB 0.0  
CX 40.00  
CY 20.00  
F1 11.001P  
F2 -1.498P  
HZ/CM 93.786  
PPM/CM .312  
SR 4925.88



Trans-Procl Report



Alex Russell, d6-acetone, +27C, AV300  
13C-PENDANT, 1H-decoupled  
Barcode label 1415



\*\*\* Current Data Parameters \*\*\*  
NAME :  
EXPNO : 10  
PROCNO : 1

ALR - ALEX RUSSELL



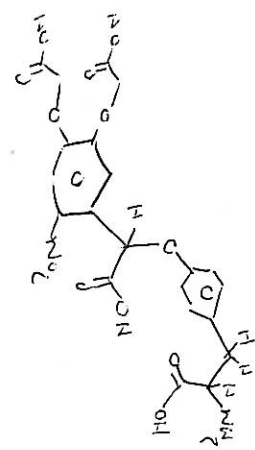
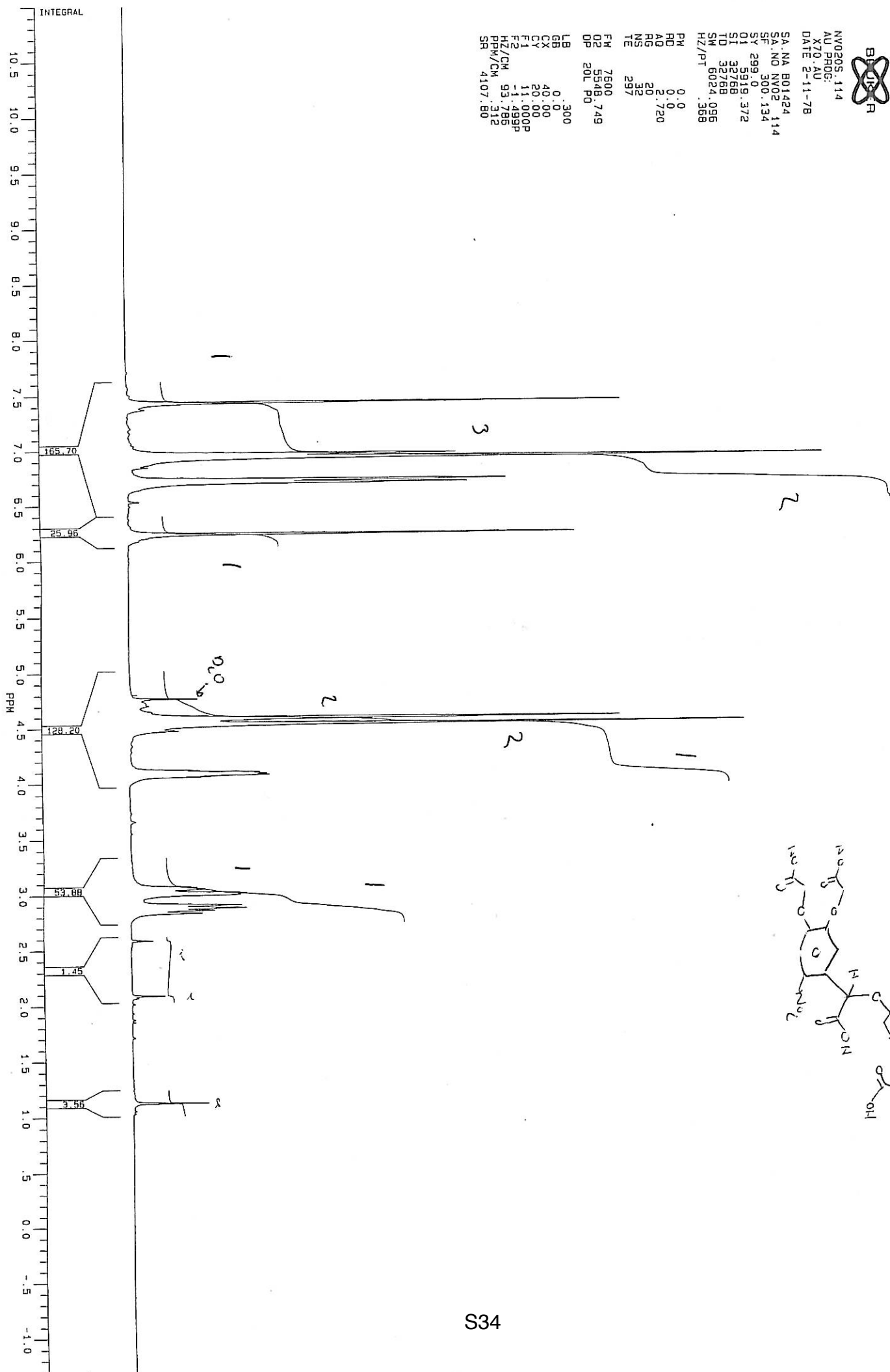
NV0205.114  
 AU PROD:  
 X70.AU  
 DATE 2-11-78

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 SA. NO NV02.114  
 SF. 300.134  
 SY 299.0  
 O1 5919.372  
 O1 32768  
 TD 32768  
 SM 6024.095  
 HZ/PT .368

PM 0.0  
 RD 0.0  
 AD 2.720  
 RG 20  
 NS 32  
 TE 297

FW 7500  
 O2 5548.749  
 DP 20L P0

LB 300  
 GB 0.0  
 CY 40.00  
 F1 20.00  
 F1 11.000P  
 F2/CM -1.499P  
 HZ/CM 93.785  
 PM/CM 312  
 SR 4107.80



Current Data Parameters  
 NAME 11-02 5.FAC  
 EXPNO 10  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20061102  
 Time 15.52  
 INSTRUM AV300  
 PROBRD 5 cm GNP 1H/1  
 PULPROG pendant  
 ID 66560  
 SOLVENT D2O  
 NS 500  
 DS 4  
 SWH 10832.393 Hz  
 FLORES 0.282939 Hz  
 AQ 1.7672160 sec  
 RG 18390.4  
 DM 26.550 usec  
 DE 6.00 usec  
 TE 300.0 K  
 CHST2 145.0000000  
 CHST3 1.0000000  
 CHST4 5.0000000  
 D1 1.5000000 sec  
 D2 0.0017244 sec  
 D3 0.0043034 sec  
 D12 0.0000200 sec  
 D13 0.00000400 sec

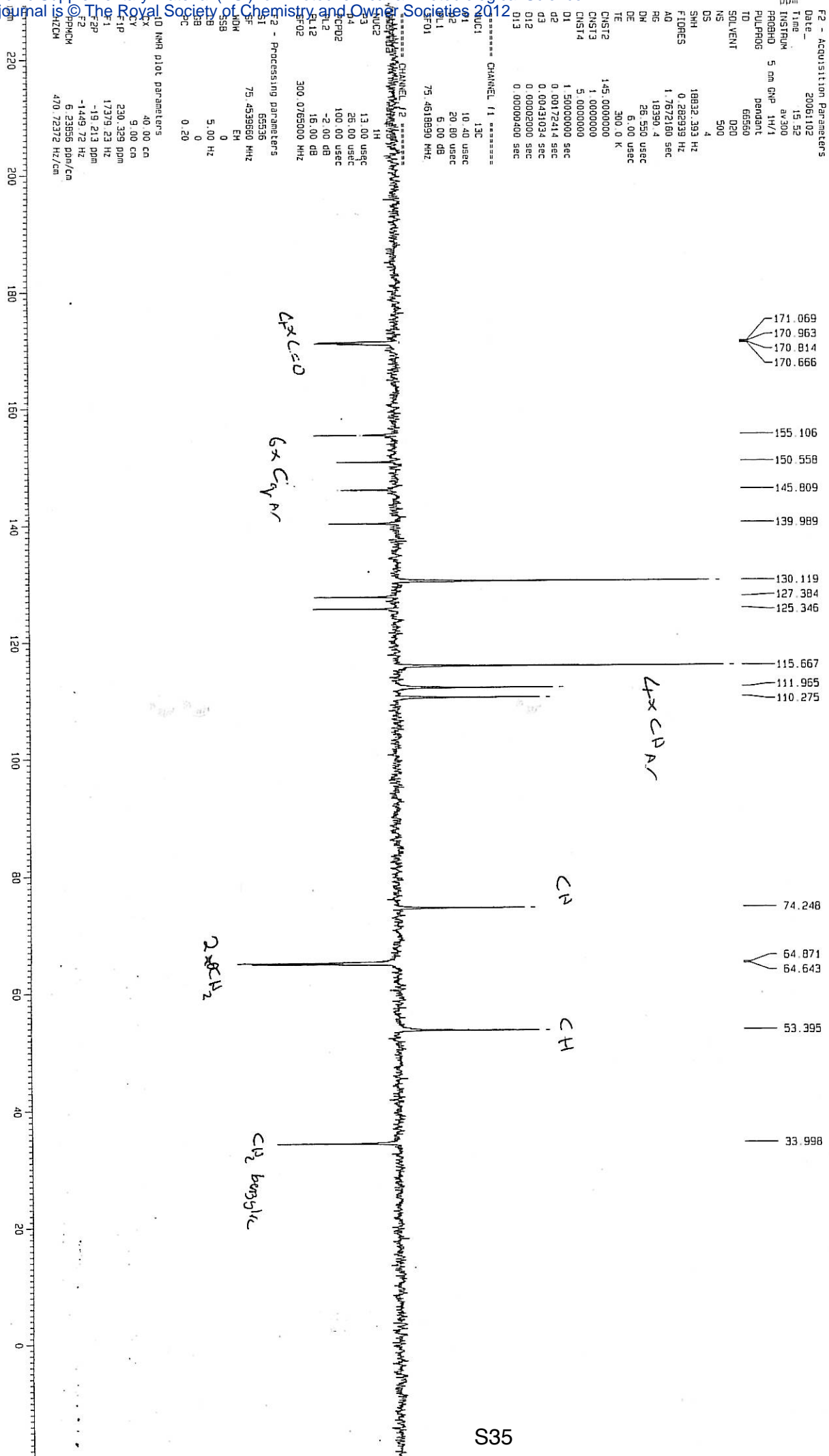
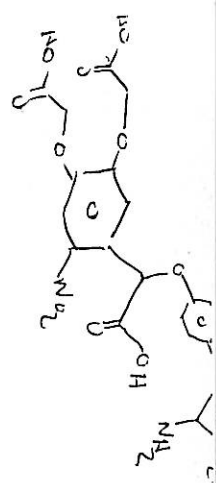
\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NU1C1 13C  
 P1 10.40 usec  
 P2 20.80 usec  
 PL1 5.00 dB  
 PL2 75.4818890 MHz

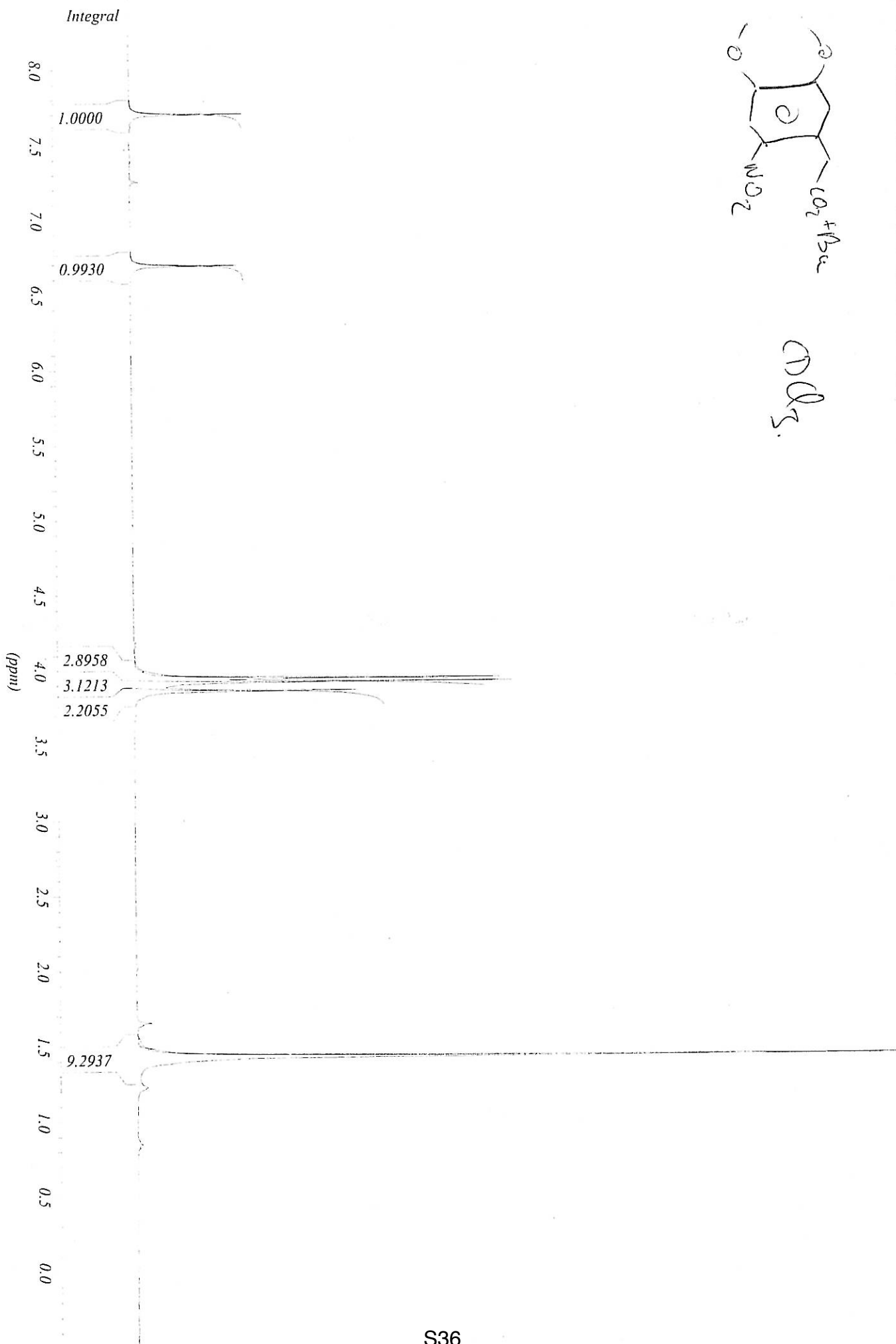
\*\*\*\*\* CHANNEL f2 \*\*\*\*\*  
 NU2C2 1H  
 P3 13.00 usec  
 P4 26.00 usec  
 PCPD2 100.00 usec  
 PL2 -2.00 dB  
 PL12 16.00 dB  
 PFC02 300.0765000 MHz

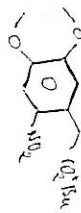
2 - Processing parameters  
 SI 65536  
 SF 75.4539660 MHz  
 AQ 75.4539660 MHz  
 EQ 0  
 SSB 0  
 GB 5.00 Hz  
 HB 0  
 TC 0.20

10 NMR plot parameters  
 SI 40.00 cm  
 SF 9.00 cm  
 F1P 230.329 ppm  
 F2P 17379.23 Hz  
 F2 -19.213 ppm  
 F2 -1449.72 Hz  
 SFO1CM 6.23655 ppm/cm  
 SFO2CM 470.72372 Hz/cm

Alex Russell, 020, +27C, AV300  
 13C-PENDANT, 1H-decoupled  
 Barcode label 1425





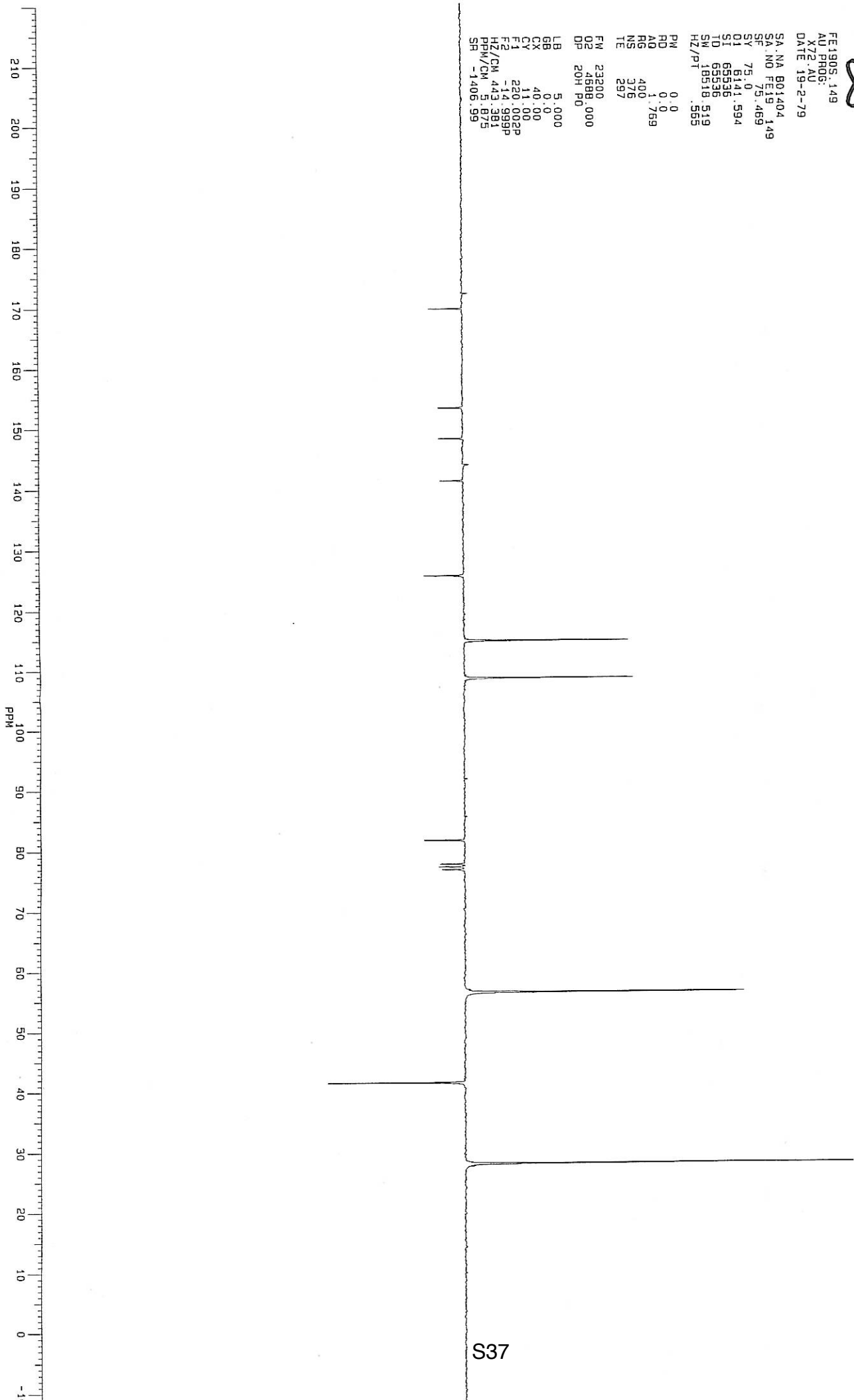


ALR - ALEX RUSSELL



FE1905.149  
AU PROG:  
X72.AU  
DATE 19-2-79

SA NA B01404  
SA NO FE19 149  
SF 75.469  
SY 75.0  
O1 6141.594  
SI 65536  
TD 65536  
SW 18518.519  
HZ/PT .555  
PW 0.0  
RD 0.0  
AQ 1.769  
RG 400  
NS 376  
TE 297  
FW 23800  
O2 4688.000  
DP 20H PD  
LB 5.000  
GB 0.0  
CX 40.00  
CY 11.00  
F1 220.002P  
F2 -14.999P  
HZ/CM 443.381  
PPM/CM 5.875  
SR -1406.99

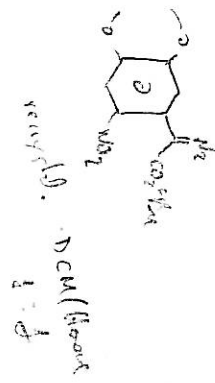


S37

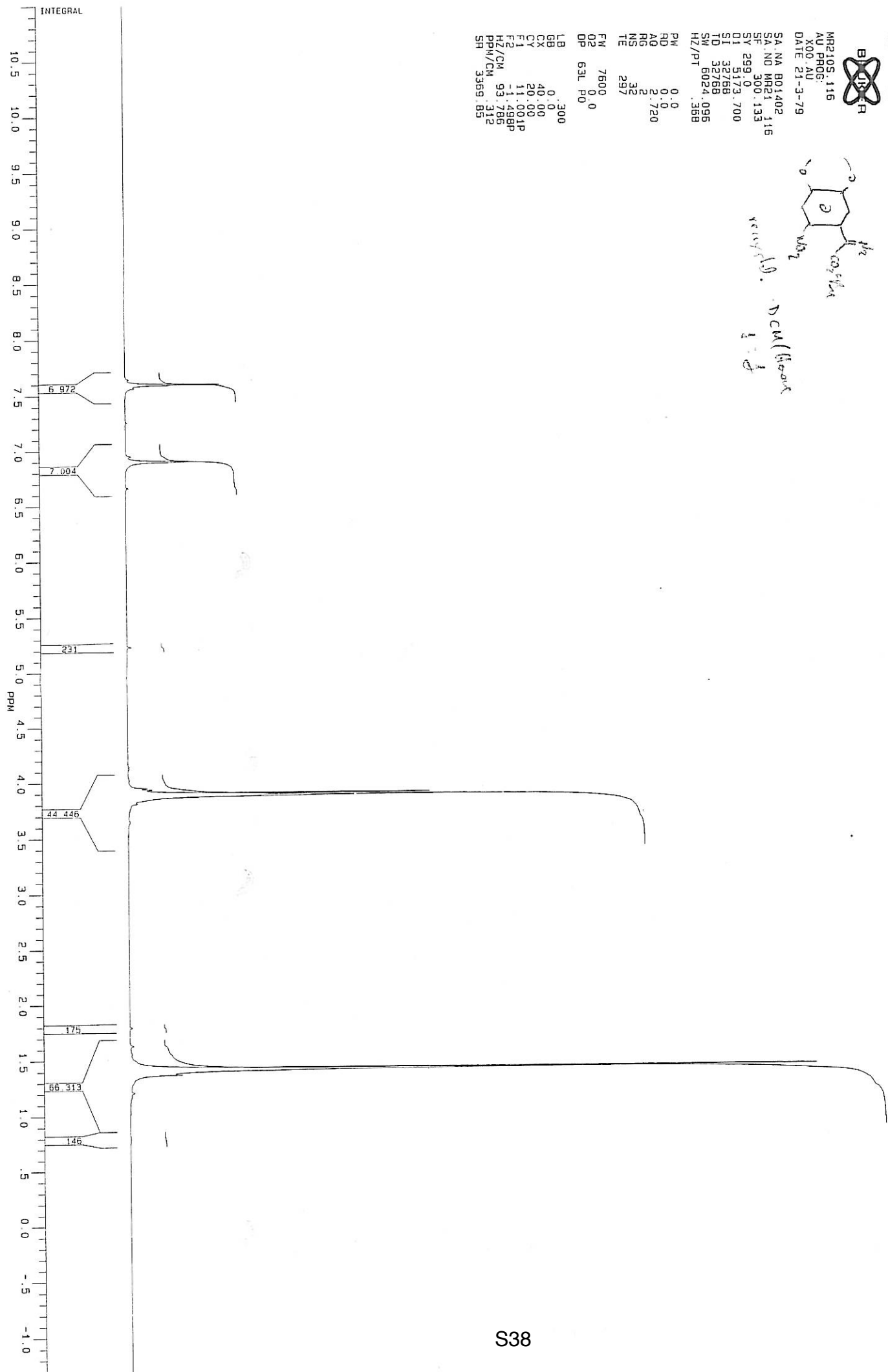
ALR - ALEX RUSSELL



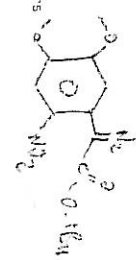
MR2105.116  
AU PROG:  
X00 AU  
DATE 21-3-79



SA NA B01402  
SA ND MR21.116  
SF 300.133  
SY 299.0  
O1 5173.700  
SI 32768  
TD 32768  
SW 6024.095  
HZ/PT .368  
PM 0.0  
RD 0.0  
AG 2.720  
RG 2  
NS 32  
TE 297  
FW 7600  
O2 0.0  
DP 63L P0  
LB .300  
GB 0.0  
CX 40.00  
CY 20.00  
F1 11.001P  
F2 -1.498P  
HZ/CM 93.786  
PPM/CM 312  
SR 3369 85



Alex Russell, CDCl<sub>3</sub>, +27°C, AV300  
13C-PENDANT, 1H-decoupled  
Barcode label 1404



Current Data Parameters  
NAME 03-21-4-FAC  
EXPRNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20070321  
Time 14.25

ED INSTRUM av300  
PROBHD 5 mm QNP 1H/1  
PULPROG pendant  
TD 65560  
SOLVENT CDCl<sub>3</sub>  
NS 500

D5 4  
SWM 18832.393 HZ  
FIDRES 0.282939 HZ  
AQ 1.7672180 sec  
RG 18390.4  
DQ 26.550 usec  
DE 6.00 usec  
TE 300.0 K

CHST2 145.0000000  
CHST3 1.0000000  
CHST4 5.0000000  
D1 1.50000000 sec  
D2 0.00172414 sec  
D3 0.00431034 sec  
D12 0.00002000 sec  
D13 0.00000400 sec

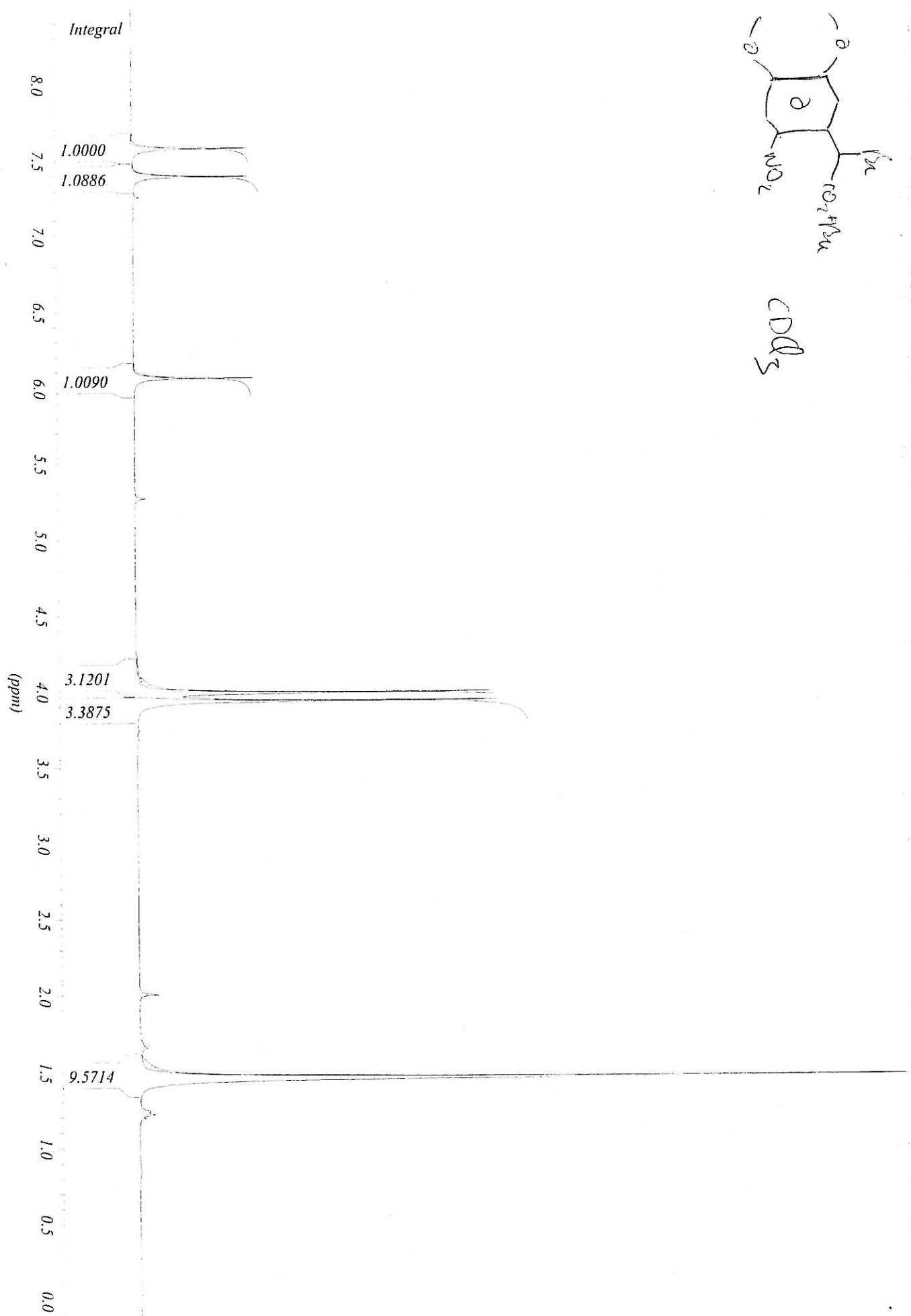
NUC1 13C  
P1 10.40 usec  
P2 20.80 usec  
PL1 6.00 dB  
SF01 75.4618990 MHz

\*\*\*\*\* CHANNEL 11 \*\*\*\*\*  
CPDPRG2 H1  
NUC2 1H  
P3 12.00 usec  
P4 24.00 usec  
PCPD2 100.00 usec  
PL2 -5.00 dB  
PL12 14.50 dB  
SF02 300.0765000 MHz

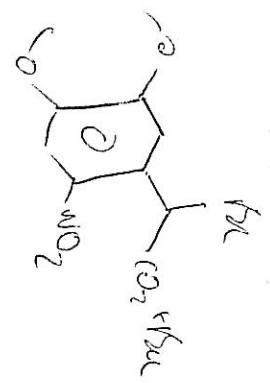
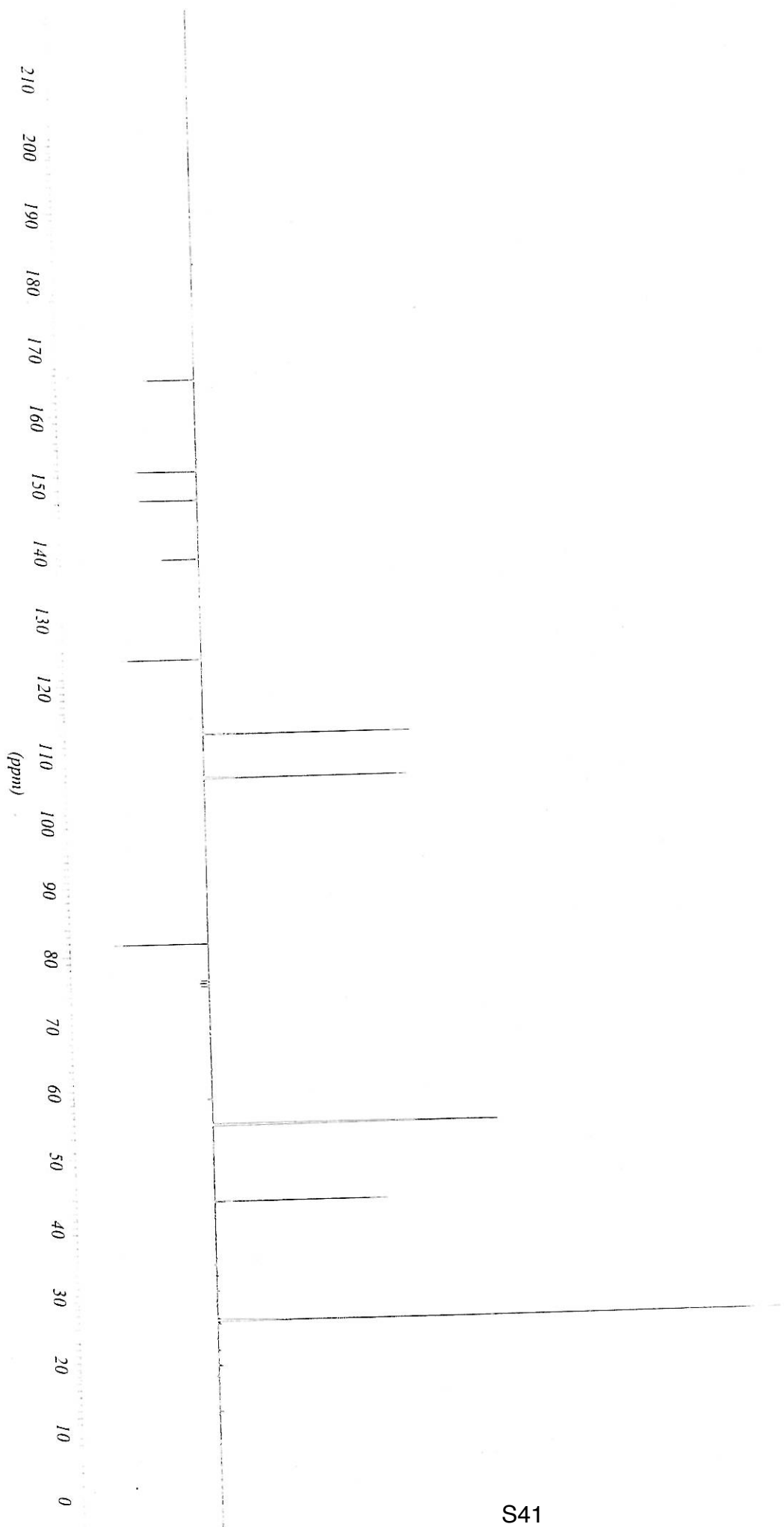
F2 - Processing parameters  
S1 51 65536  
SF 75.4538346 MHz  
WDW EM  
SSB 0  
LB 5.00 HZ  
GB 0  
PC 0.20

10. NMR plot parameters  
CX 40.00 cm  
CY 9.00 cm  
ZP 230.329 mm  
F1 17379.20 HZ  
F2P -19.213 mm  
F2 -1449.72 HZ  
FREQM 6.23556 ppm/cm  
HZCM 470.72893 Hz/cm







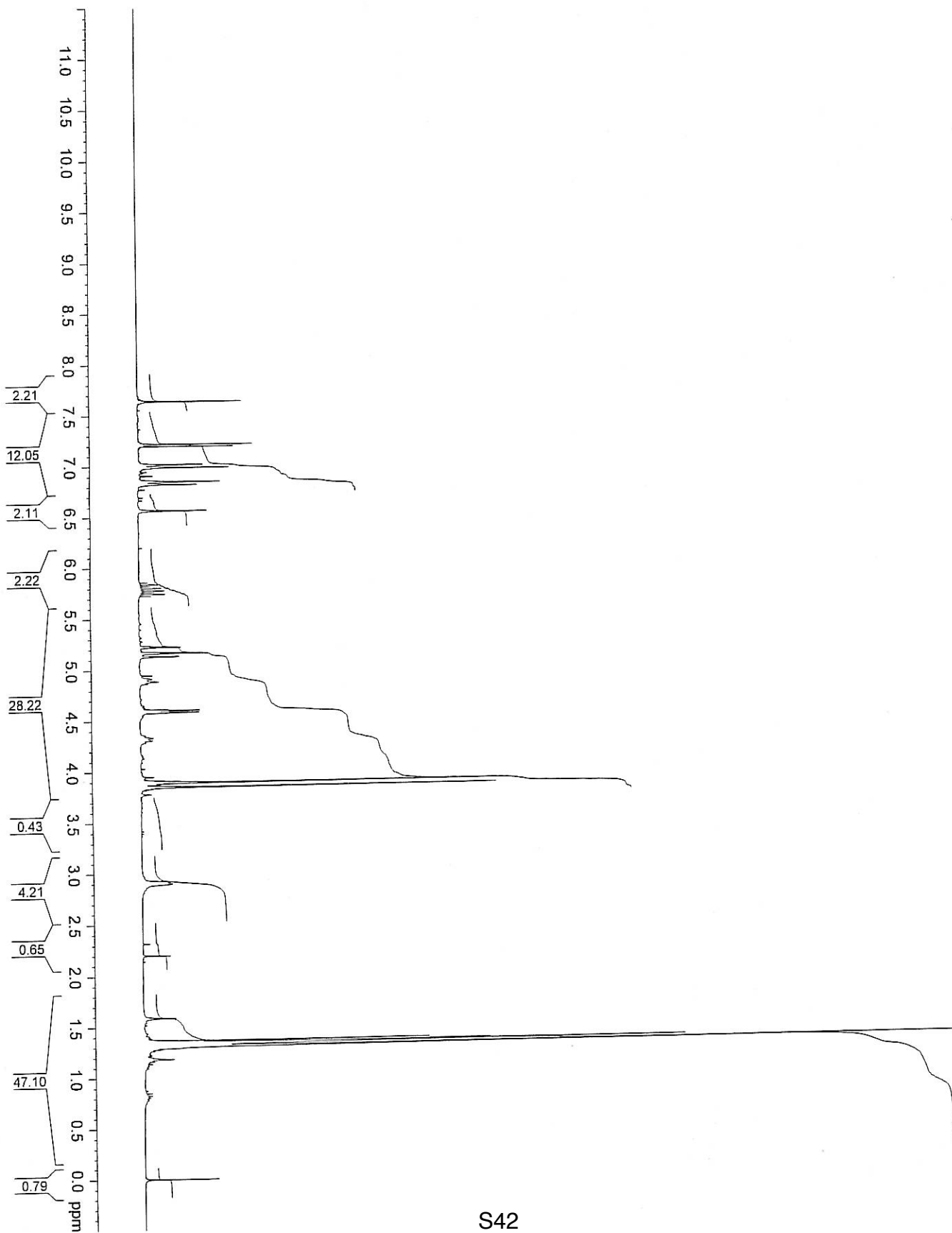


CDL3

John Snath Tyr second NaOH wash

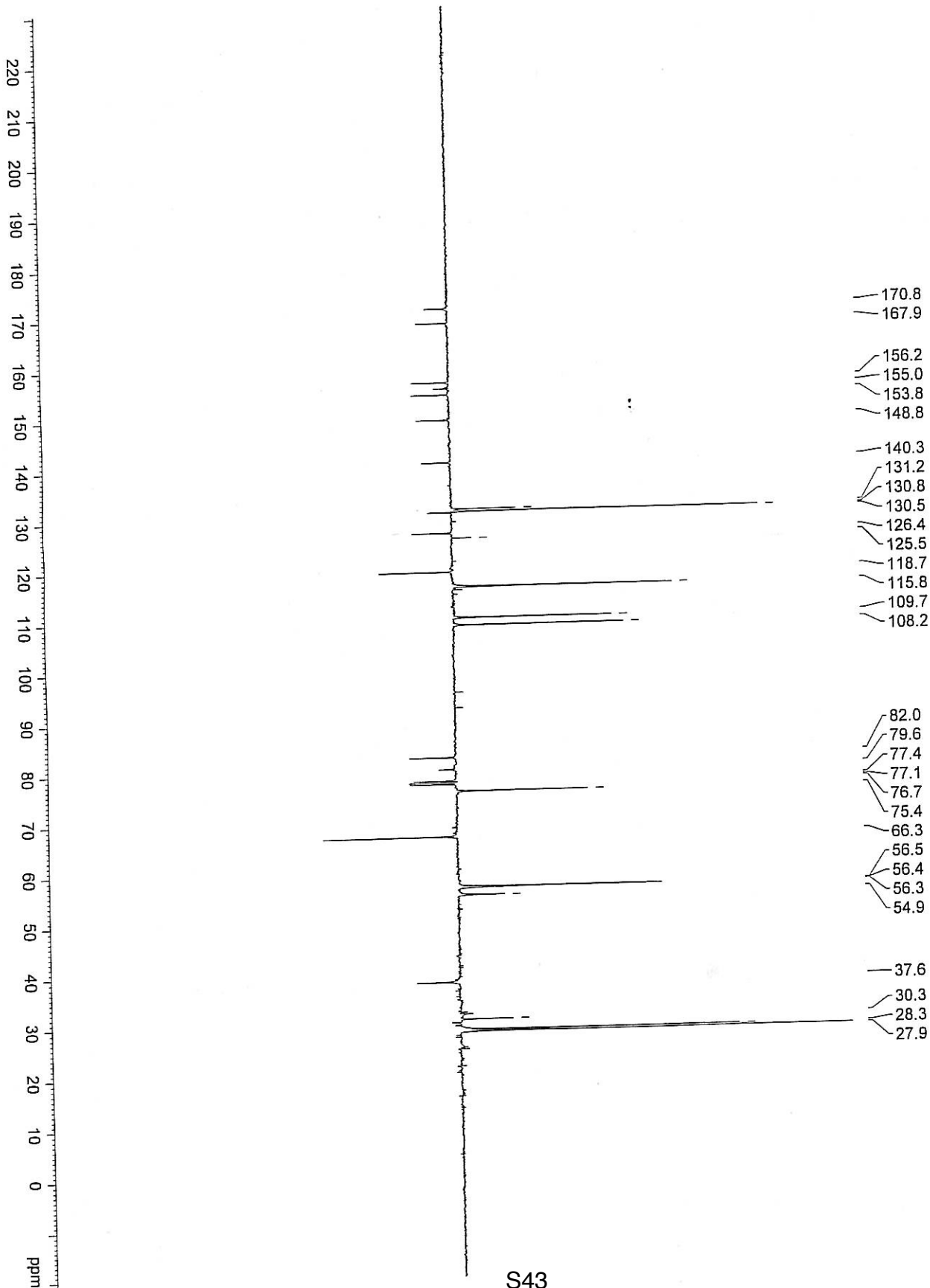
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NAME 08-04-Snath-1
EXPNO 10
PROCNO 1
Date_ 20090804
Time 9.50
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 128
DW 83.200 usec
DE 13.19 usec
TE 293.8 K
D1 1.00000000 sec
TD0 1

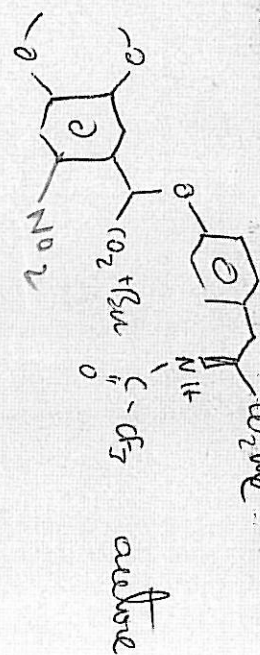
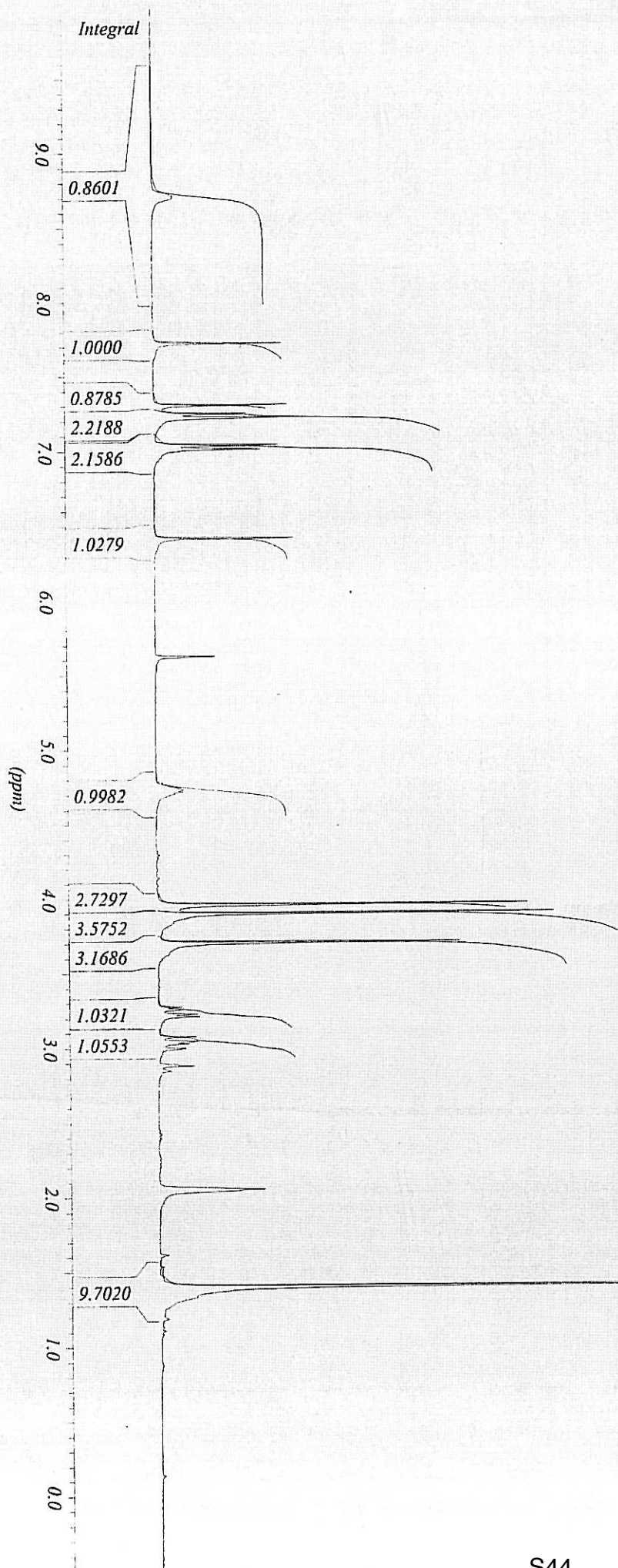
===== CHANNEL f1 =====
NUC1 1H
P1 12.80 usec
PL1 1.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300251 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
```



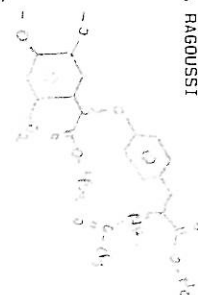
John Snaitth Tyr

```
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EXPNO 10
PROCNO 2009pp05
Date_ 15/4/
Time 15:44
INSTRUM spect
PROBHD 5 mm PABUL 13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 302.0 K
CNST2 145.0000000
CNST3 1.0000000
CNST4 5.0000000
D1 1.50000000 sec
D2 0.00172414 sec
D3 0.00431034 sec
D12 0.00002000 sec
D13 0.00000400 sec
TD0 1
===== CHANNEL f1 =====
NUC1 13C
P1 8.80 usec
PL1 -17.80 dB
SFO1 58.63890457 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P3 9.70 usec
PCPD2 19.40 usec
PL2 -4.00 dB
PL12 15.35 dB
PL2W 24.29185867 W
SFO2 400.1316005 MHz
SI 65536
SF 100.6172690 MHz
WDW EM
SSB 0
LB 4.00 Hz
GB 0
PC 1.00
```

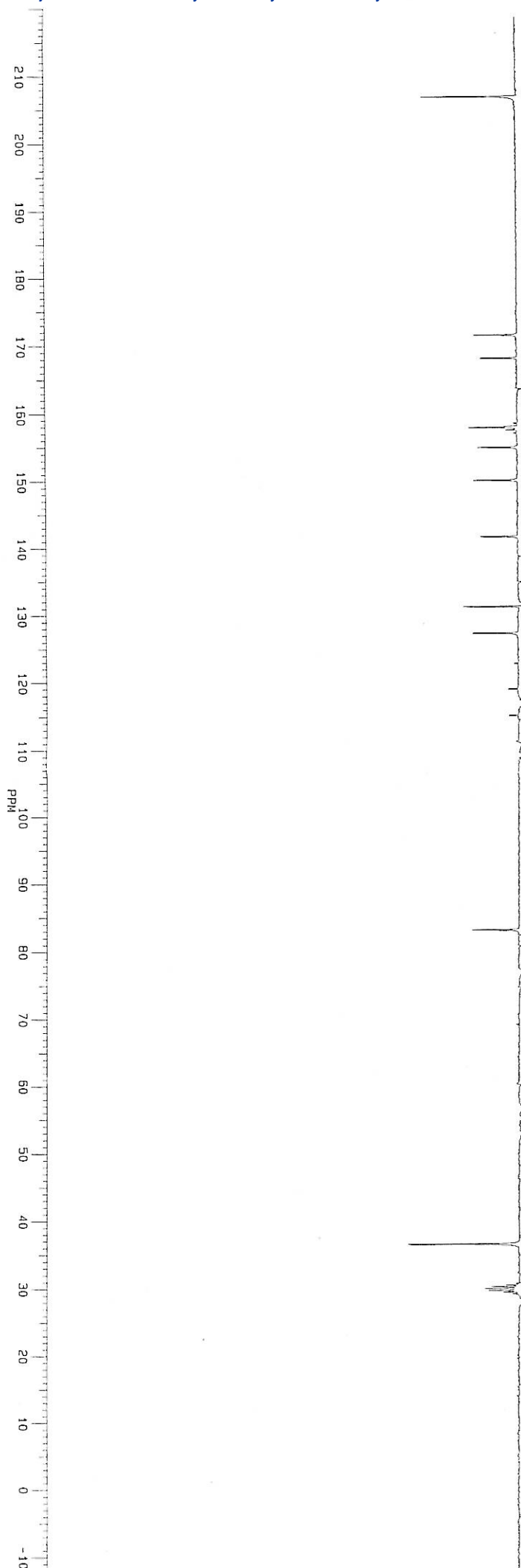




MCS - MARILENA RAGOUSSTI



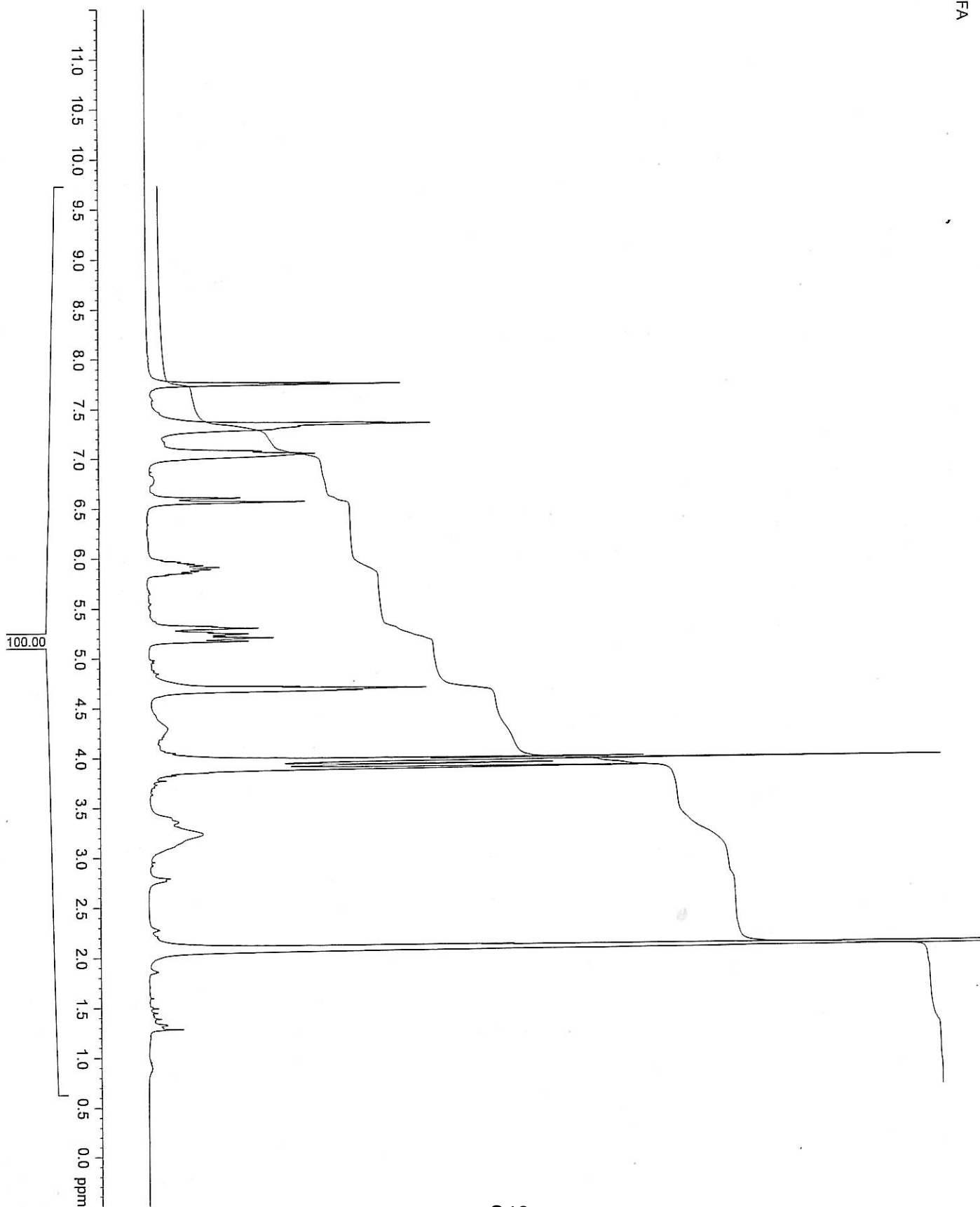
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AU PROG: X72 AU  
DATE 26-3-79  
SA:NA B00111.136  
SA:NO MR26.136  
SF 75.0  
SY 75.0  
O1 6141.594  
S1 65536  
TD 65536  
SW 18518.519  
HZ/PT 565  
PW 0.0  
RD 0.0  
AO 1.769  
RG 200  
NS 376  
TE 297  
FW 23200  
O2 6250.000  
DP 20H PO  
LB 5.000  
GB 0.0  
CX 40.00  
CY 11.00  
F1 220.001P  
F2 -14.999P  
HZ/CM 443.391  
PPM/CM 5.875  
SR -1072.42



John Snaith DMNPA Tyr after TFA  
after vac line

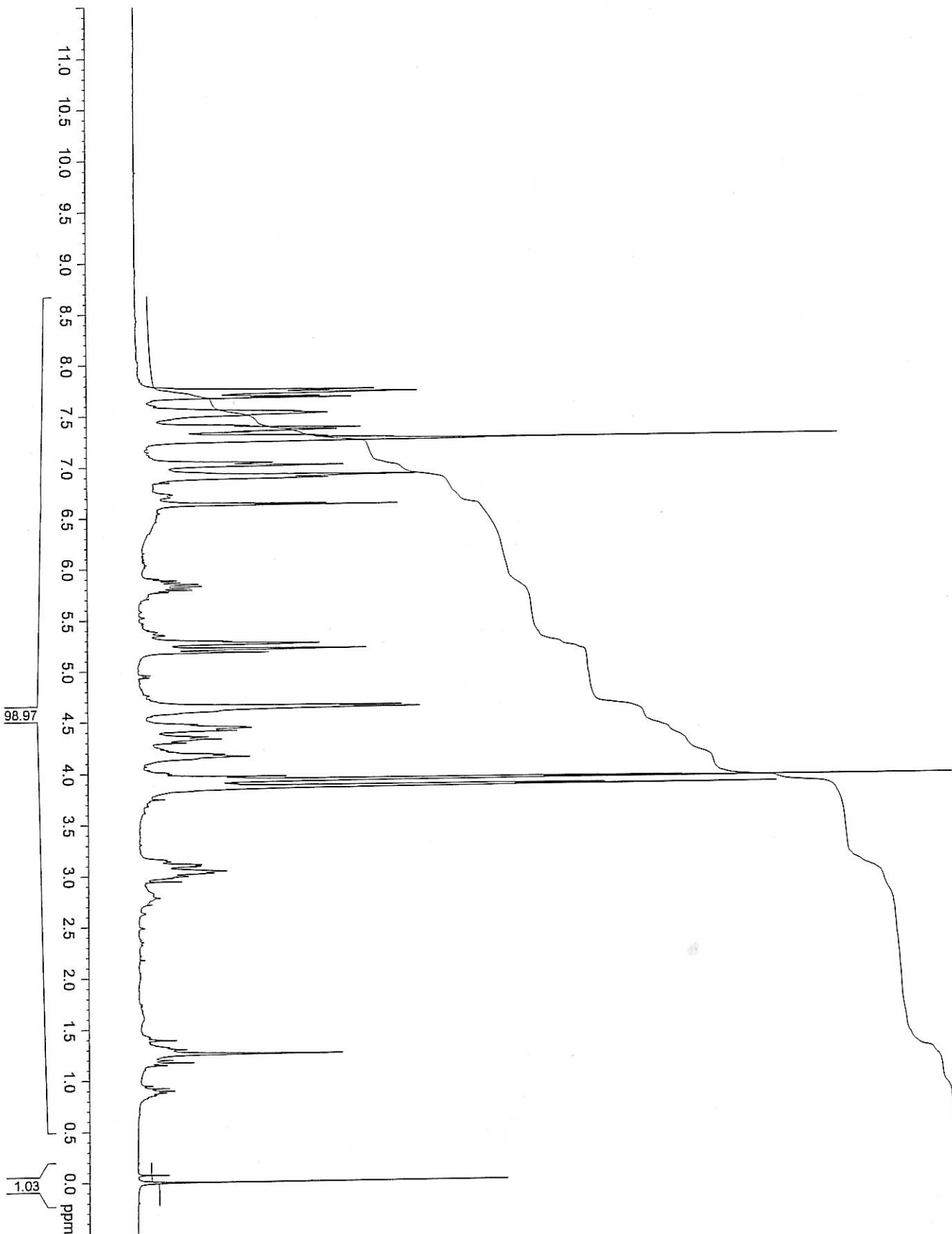
NAME 08-19-Snaith-4  
EXPNO 10  
PROCNO 1  
Date\_ 20090819  
Time 10.01  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 32768  
SOLVENT Acetone  
NS 32  
DS 2  
SWH 6009.615 Hz  
FIDRES 0.183399 Hz  
AQ 2.7263477 sec  
RG 80.5  
DW 83.200 usec  
DE 13.19 usec  
TE 293.7 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 12.80 usec  
PL1 1.00 dB  
PL1W 9.57725906 W  
SF01 300.1318534 MHz  
SI 32768  
SF 300.1300000 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



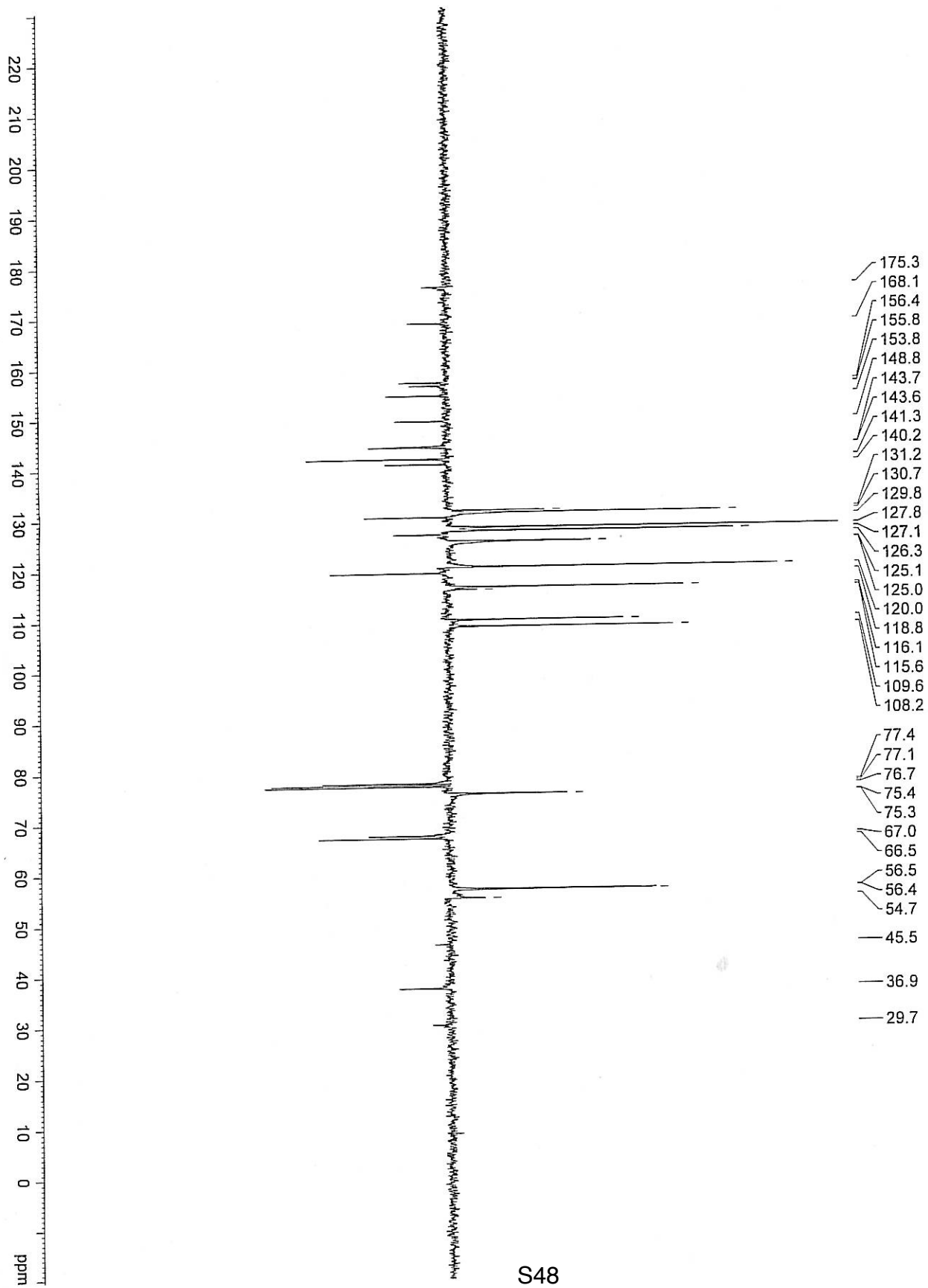
NAME 06-24-Snath-36  
EXPNO 10  
PROCNO 1  
Date\_ 20110624  
Time 14.46  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 32768  
SOLVENT CDCl3  
NS 32  
DS 2  
SWH 6009.615 Hz  
FIDRES 0.183399 Hz  
AQ 2.7263477 sec  
RG 161  
DW 83.200 usec  
DE 13.19 usec  
TE 293.0 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 <sup>1</sup>H  
P1 12.80 usec  
PL1 1.00 dB  
SFO1 300.1318534 MHz  
SI 32768  
SF 300.1300065 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



```

NAME 11-19-Snath-15
EXPNO 10
PROCNO 1
Date_ 20101119
Time 21.39
INSTRUM spect
PROBHD 5 mm PABUL 13C
PULPROG pendulans
TD 65536
SOLVENT CDC13
NS 512
DS 4
SWH 25252.325 Hz
FIDRES 0.386323 Hz
AQ 1.2976529 sec
RG 2050
DE 19.800 usec
TE 297.1 K
CNST2 145.0000000
CNST3 5.0000000
CNST4 5.0000000
D1 1.5800000 sec
D2 0.00172414 sec
D3 0.000431034 sec
TD0 0.00000400 sec
===== CHANNEL f1 =====
NUC1 13C
P1 8.80 usec
P2 17.60 usec
PL1 -3.00 dB
SFO1 100.6233333 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P3 9.70 usec
P4 19.40 usec
PCPD2 4.00 dB
PL2 15.35 dB
PL2W 24.28169591 W
PL2V 0.28213742 W
PL2W 40843.16093 MHz
SFO2 40843.16093 MHz
SF 100.6127690 MHz
VWDW 0
SSB 0
LB 4.00 Hz
GB 0
PC 1.00
  
```





212-3  
221

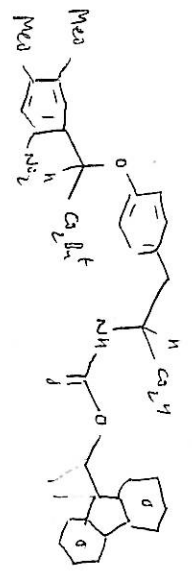
MSD - MATT SADLER



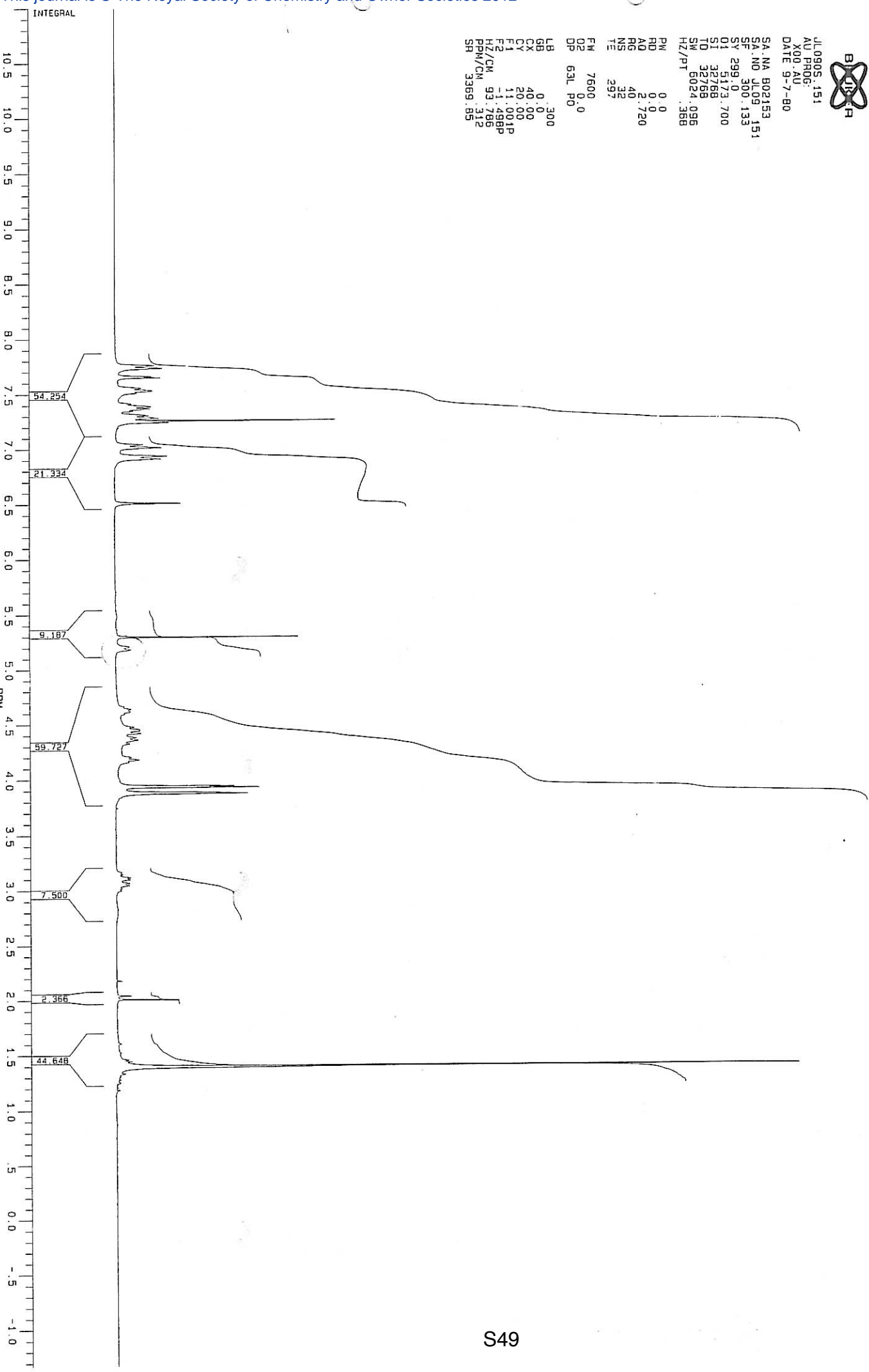
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DATE 9-7-80

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NS 32  
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FM 7600  
DZ 0.0  
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LB 0.300  
GB 0.0  
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CY 20.00  
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SR 3369.85



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MSD - MATT SADLER

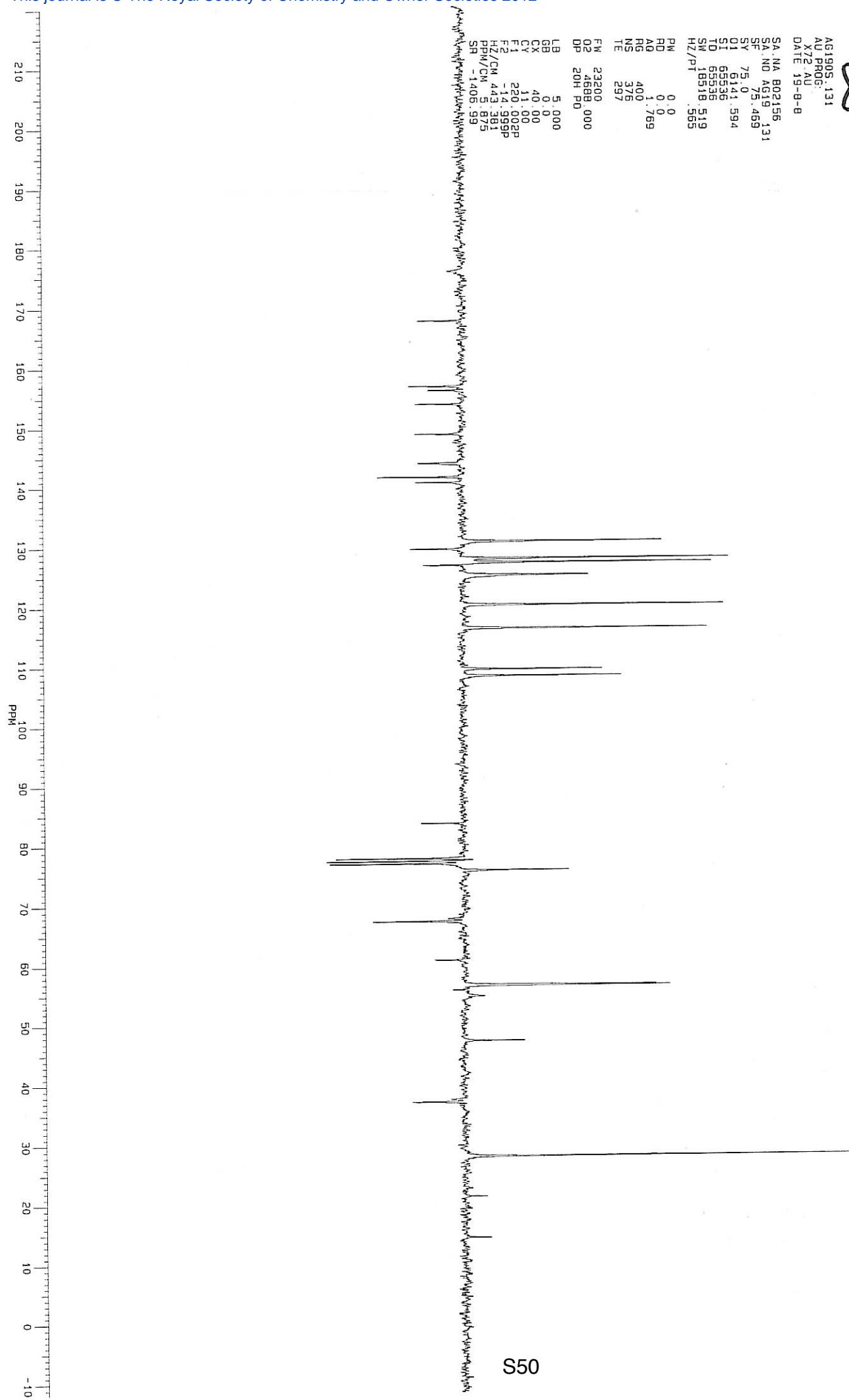
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DATE 19-8-8

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PW 0.0  
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NS 375  
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DP 20H PD

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CY 11.00  
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F2 -14.999P  
HZ/CM 443.381  
PPM/CM 5.875  
SR -1406.99



S50

