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Contractor Report ARAED-CR-91008

ANALYSIS OF NITROMETHANE

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June 1991

U.S. ARMY ARMAMENT RESEARCH, DEVELOPMENT AND ENGINEERING CENTER

Armament Engineering Directorate
Picatinny Arsenal, New Jersey

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& CHEMICAL COMMAND
ARMAMENT RDE CENTER

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13. ABSTRACT *(Maximum 200 words)*

Samples were taken at random from 60 barrels of nitromethane which were stored at Yuma Proving Grounds and sent to Southwest Research Institute for analyses. The nitromethane content was determined for each along with contaminate analyses for nitroethane, 2-nitropropane, water, and metals. The results indicated the following:

Component	Percent
Nitromethane	98 or greater
Nitroethane	1.1 or less
2-Nitropropane	0.2 or less
Water	0.11 or less
Metals	Trace (less than 10 ppb)

14. SUBJECT TERMS
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1. INTRODUCTION

Originally, the Government had purchased and stored 60 barrels of nitromethane at the Yuma Proving Grounds for use in the TEXS system. This system required that the materials be housed in the all-purpose storage area. DOD has classified nitromethane as a Class 4, mass detonating material and not suitable for storage in the all-purpose area. This restriction resulted in the rejection of nitromethane as a candidate for the TEXS system.

These barrels have been exposed under varying degrees of environmental conditions and changes. Contamination and degradation to the nitromethane may have occurred during the storage period. An analysis is required to determine whether the material has deviated dramatically from the specifications furnished by the supplier. This analysis will determine whether the stored nitromethane is suitable for use in another application.

Samples of nitromethane were taken at random from the 60 barrels of stored nitromethane. These samples were taken from the top, middle, and bottom to ascertain the uniformity of nitromethane composition. Replicate samples of each were analyzed in the following manner for these components and contaminates that may be present:

Component	Analytical Method
Nitromethane	FTIR
Nitroethane	GC/Mass Spectroscopy
2-Nitropropane	GC/Mass Spectroscopy
Water	Karl Fischer
Metals	AA Graphite Furnace

FTIR - Fourier transform infrared
GC - gas chromatograpy
AA - atomic absorption

2. PROCEDURES AND RESULTS

Analysis of nitromethane was performed by FTIR. SwRI received a 99%+ sample of nitromethane that was certified by Angus Chemical. An FTIR analysis was performed and the results compared to the Chem Sources 98%+ sample used during the program. The results confirmed the purity level of the Chem Sources sample. The results are given in Table 1 and raw data are found in Appendix A.

TABLE 1. FTIR ANALYSIS FOR PERCENT NITROMETHANE

Sample	% Nitromethane
Y1-B	99.6
Y1-M	98.3
Y1-T	100.0
Y1-MM	99.3
A1-B	99.3
A1-M	99.9
A1-MM	99.0
N1-B	100.2
N1-M	99.5
N1-T	99.5
Standard	98%

B	Bottom
M	Middle
T	Top
MM	Middle mixed

The FTIR spectra of the neat nitromethane samples were compared to that of the Mallinckrodt sample and found to be virtually identical. Computer subtraction of the Mallinckrodt sample from the other samples failed to show any gross contaminant. It was concluded that any impurities present were only in low concentration.

Weighed quantities of each of the samples were dissolved in carbon tetrachloride to make 25 mL total volume. A solution prepared similarly from Chem Service nitromethane (98.0%) was utilized as a standard. This standard was used due to the higher degree of purity of the material. The nitromethane concentration of the samples was determined by comparing absorption intensities at 657 cm^{-1} in a sealed liquid cell. The 657 cm^{-1} absorption is specific for nitromethane and is unaffected by nitroethane and 2-nitropropane. The precision was estimated to be ca. ±1 nitromethane.

An overlay, offset comparison spectra of Y1-M and the Chem Service 98% standard is given in Appendix A. The two spectra are identical except for a few insignificant peaks in the 800-830 range. The spectra for all the samples are included in Appendix B.

Analysis of volatile compounds other than nitromethane was performed using GC/MS. The results for nitroethane and 2-nitropropane are given in Table 2 and raw data are found in Appendix C.

TABLE 2. GC/MS ANALYSIS FOR VOLATILE CONTAMINANTS

Sample	% Composition	
	Nitroethane	2-Nitropropane
Y1-B	0.9	~0.1
Y1-M	0.9	~0.1
Y1-T	0.8	~0.1
Y1-MM	0.9	~0.1
A1-B	~0.1	~0.1
A1-M	~0.1	~0.1
A1-MM	~0.4	0.2
N1-T	~0.1	~0.1
N1-M	~0.1	~0.1
N1-B	1.1	~0.1

See legend Table 1.

Analysis for semivolatile compounds was performed using GC/MS. Two samples were selected for analysis. The results are given in Table 3.

TABLE 3. GC/MS ANALYSIS FOR SEMIVOLATILE CONTAMINANTS IN SELECTED NITROMETHANE SAMPLES

Component	Sample Y1-M ppm	Sample N1-M ppm
Paraldehyde	70.0	38.0
2-Pentanone-4-methyl	42.0	0.0
Total other unknowns	13.4	2.9
% Total Semivolatiles	0.01%	0.004%

See legend Table 1.

No single semivolatile contaminant was found to have a concentration >0.007%.

Karl Fischer analyses were performed to assay the water content of each of the samples. The results can be compared to standard 95% nitromethane sample from Mallinckrodt. The results are given in Table 4.

TABLE 4. KARL FISCHER ANALYSIS OF NITROMETHANE SAMPLES

Sample	ppm H_2O	%
Standard	1,370	0.14
Y1-T	629	0.06
Y1-MM	697	0.07
Y1-M	1077	0.11
Y1-B	693	0.07
N1-T	705	0.07
N1-M	506	0.05
N1-B	432	0.04
A1-M	549	0.05
A1-MM	589	0.06
A1-B	590	0.06

See legend Table 1.

The average percent water of the samples was 0.06.

Metals contamination analysis was performed on a Perkin-Elmer 5000 graphite furnace atomic absorption spectrophotometer. Calibration standards were made up in methanol and samples were diluted 1:1 in methanol before analysis. A methanol blank was run with each sample set. No solvent contamination was observed.

The results are presented in Table 5.

TABLE 5. METALS ANALYSIS OF NITROMETHANE SAMPLES

	ppb		
Sample	Cu	Fe	Ni
Standard	<1.0	<2.0	<5.0
Y1-T	8.2	<4.0	<10.0
Y1-MM	9.0	<4.0	<10.0
Y1-M	9.0	<4.0	<10.0

TABLE 5. METALS ANALYSIS OF NITROMETHANE SAMPLES			
	ppb		
Sample	Cu	Fe	Ni
Y1-B	6.6	<4.0	<10.0
N1-T	6.6	<4.0	<10.0
N1-M	6.6	<4.0	<10.0
N1-B	5.8	<4.0	<10.0
A1-MM	9.8	<4.0	<10.0
A1-M	8.0	<4.0	<10.0
A1-B	9.8	<4.0	<10.0

See legend Table 1.

A compendium of all the analytical results are given in Table 6.

TABLE 6. COMPUTATION OF VARIOUS ANALYSIS ON NITROMETHANE SAMPLES						
	% Composition					
Sample	Nitromethane	H_2O	Nitroethane	2-Nitropropane	Semivolatiles	Metal Contaminants (ppb)
Y1-B	99.6	0.07	0.9	~0.1		<10
Y1-M	98.3	0.11	0.9	~0.1	0.01	<10
Y1-T	100.0	0.06	0.8	~0.1		<10
Y1-MM	99.3	0.07	0.9	~0.1		<10
A1-B	99.3	0.06	~0.1	~0.1		<10
A1-M	99.9	0.05	~0.1	~0.1		<10
A1-MM	99.0	0.06	0.4	0.2		<10
N1-B	100.2	0.04	1.1	~0.1		<10
N1-M	99.5	0.05	~0.1	~0.1	<0.01	<10
N1-T	99.5	0.07	~0.1	~0.1		<10

See legend Table 1.

3. DISCUSSION

A high degree of purity of nitromethane was found for all the samples. The FTIR analysis showed that all the samples had a concentration of 98% or greater nitromethane. This finding is based on the assumption that the Chem Service nitromethane standard used for comparison is of 98% purity. Other peaks in the FTIR spectra were of insufficient prominence to quantify, so alternate analytical techniques were utilized to identify the other compounds present.

Analysis of other volatile and semivolatiles, especially nitroethane and 2-nitropropane, was accomplished utilizing GC/MS. It was ascertained that all the samples contained 1.1% or less of nitroethane and 0.2% or less of 2-nitropropane. All other peaks in the volatile analysis were significantly less prominent indicating much lower concentrations of any other volatile contaminants.

The semivolatile analysis yielded only trace quantities of contamination of the nitromethane. Only two samples were selected for analysis due to the similarity of the samples preceding analysis and the small percentage of unaccounted for components. Quantification of contaminants was accomplished by subtracting the effects of the nitromethane. It is possible that a higher percentage of paraldehyde is present due to the elution interference of the solvent, but the results of the prior analysis make this unlikely.

Karl Fisher water analysis yielded results of 0.11% water or less. This is consistent with the results of the metals analysis, which showed only trace amounts of metals contamination. For nitromethane to be corrosive to a steel container, the H_2O concentration must exceed 0.2%. The H_2O concentration was below this limit; hence, there was no or little interaction with the containers.

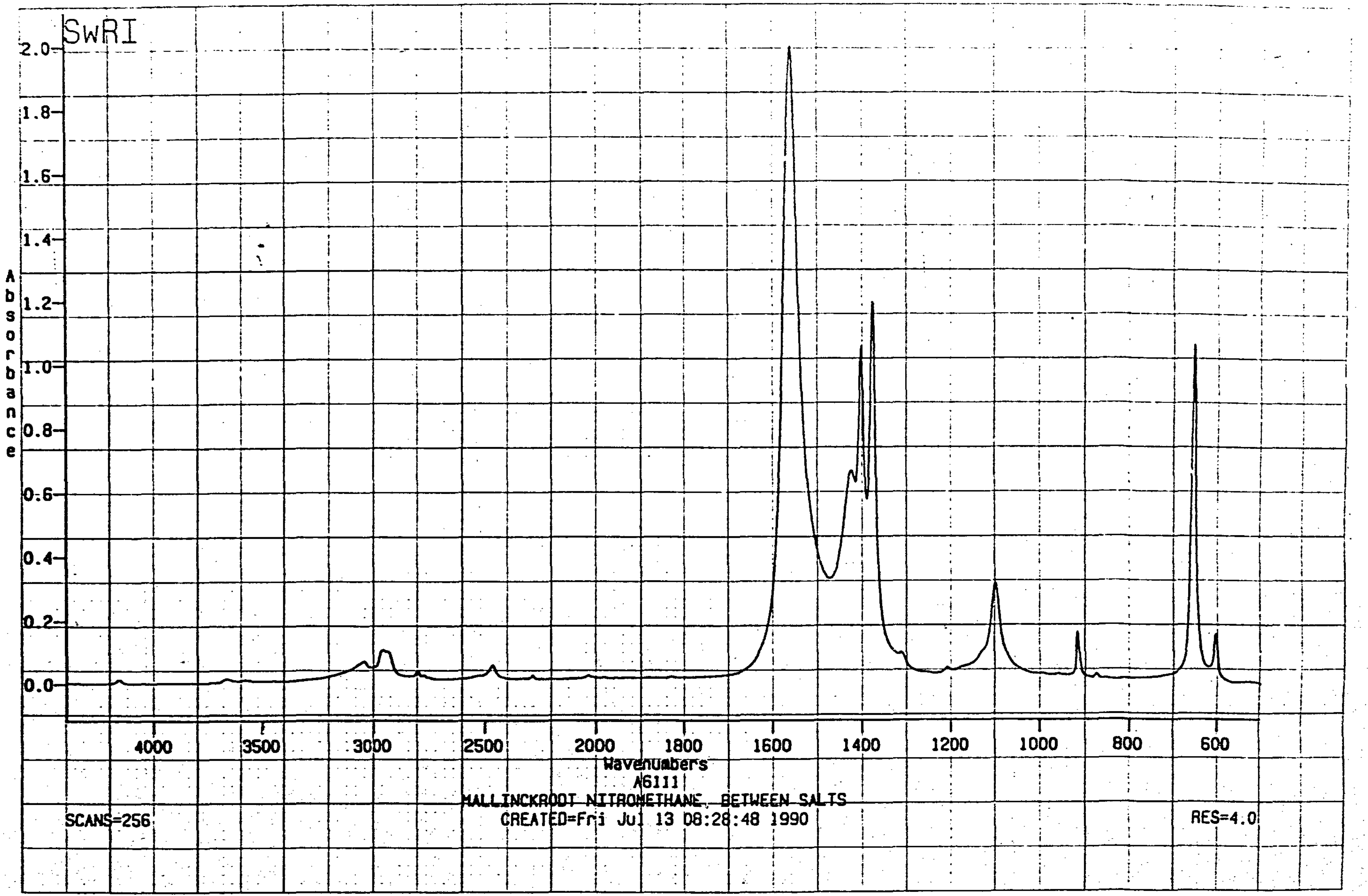
Sampling procedures did not accompany the samples. The method used to sample the barrels could have an effect on the analytical findings. It will be important in the future to ensure that a standard method of sampling is used, preferably a technique that yields a representative sample from each of the layers within a single drum of nitromethane.

It will also be necessary to establish a chain-of-custody record to ensure proper handling of the samples from the field to the analyst. Such a record could help to facilitate understanding any gross contamination that appears, that are not accounted for in future analysis.

Two possible explanations exist for the discrepancy between the original reported purity of the nitromethane, and the purity found in this program. First, during telephone conversations with Angus Chemical, the original shipment of nitromethane was certified at 96%+. This designation commonly results in product which is percentage points higher. Second, and most probable, if a point sampling procedure was utilized, as opposed to a cross-sectional sampling procedure, then a higher degree of homogeneity would result. Point sampling techniques miss any differences in product due to stratification.

APPENDIX A

FTIR COMPARISON SPECTRA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4000
3500
3000
2500
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1400
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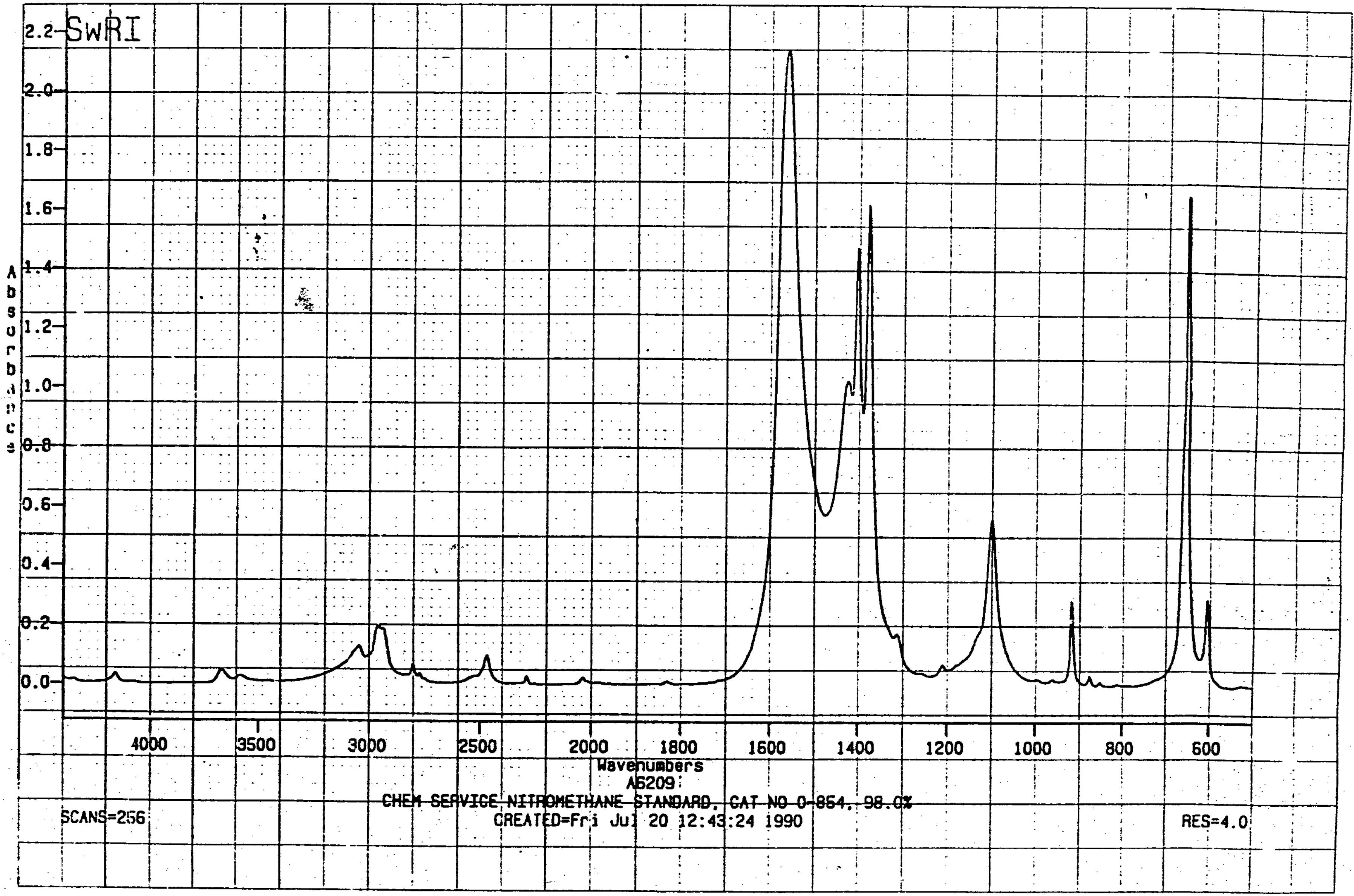
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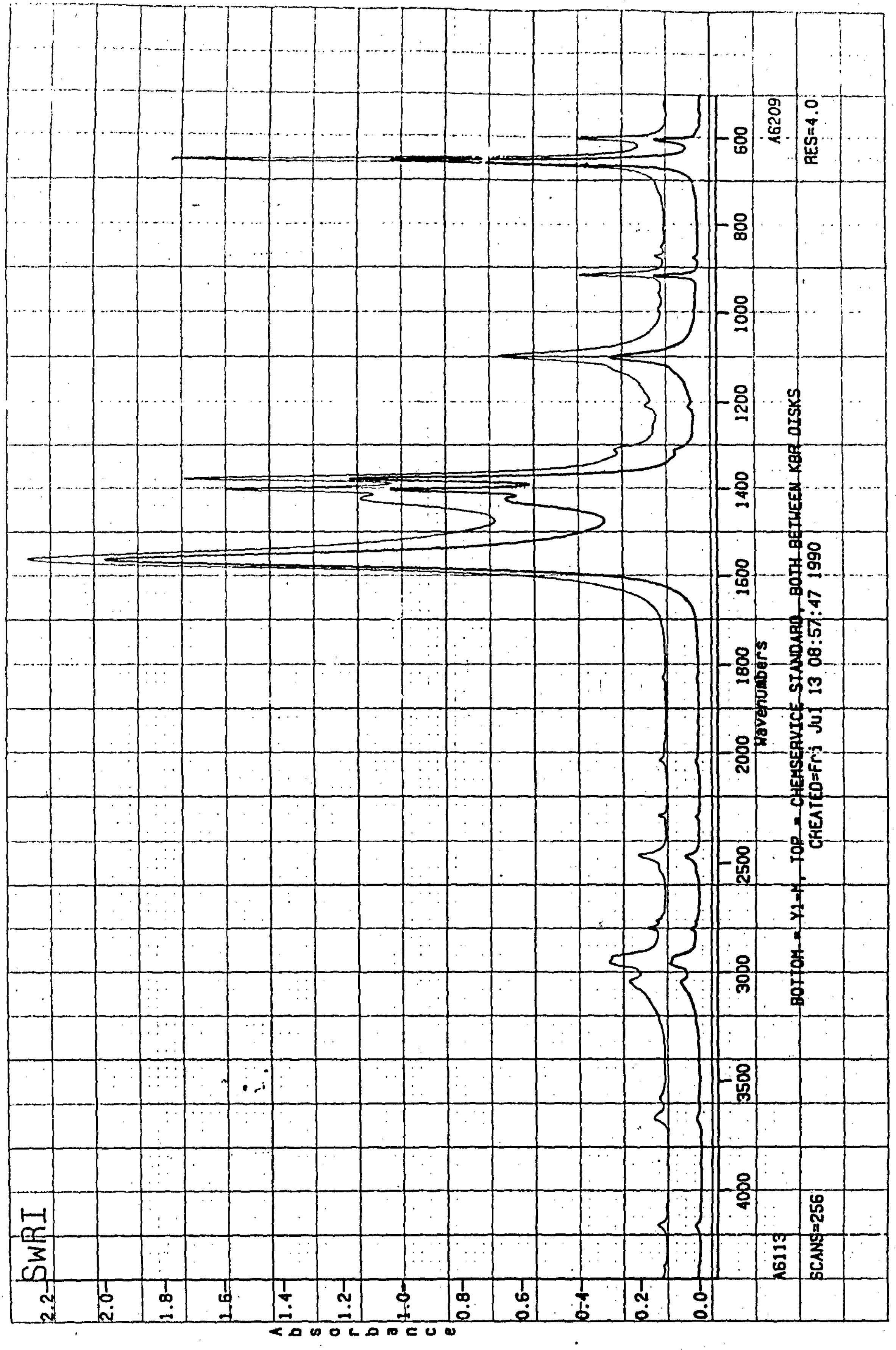
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A6113
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A6209
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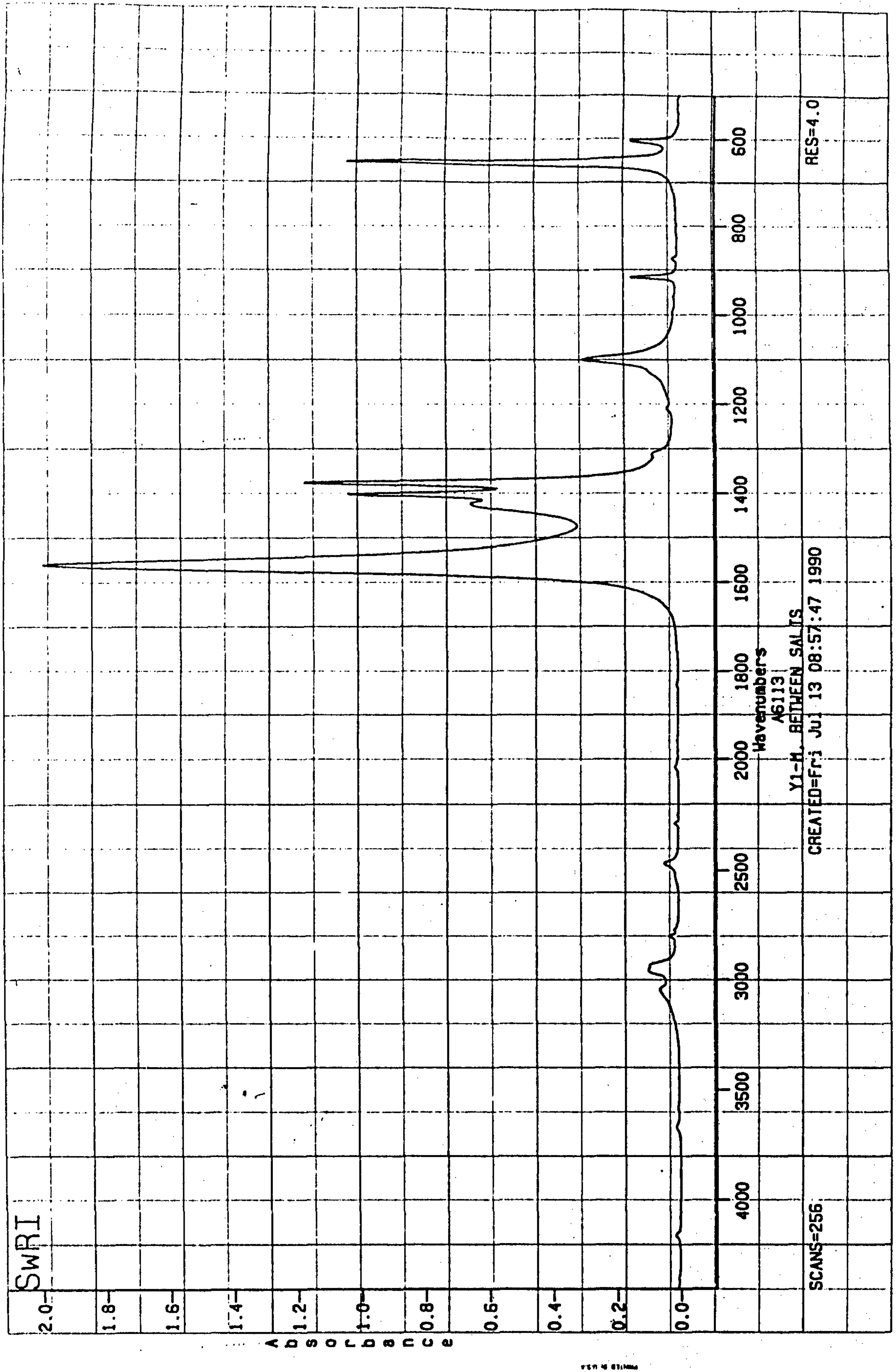
APPENDIX B

FTIR SPECTRA OF SAMPLES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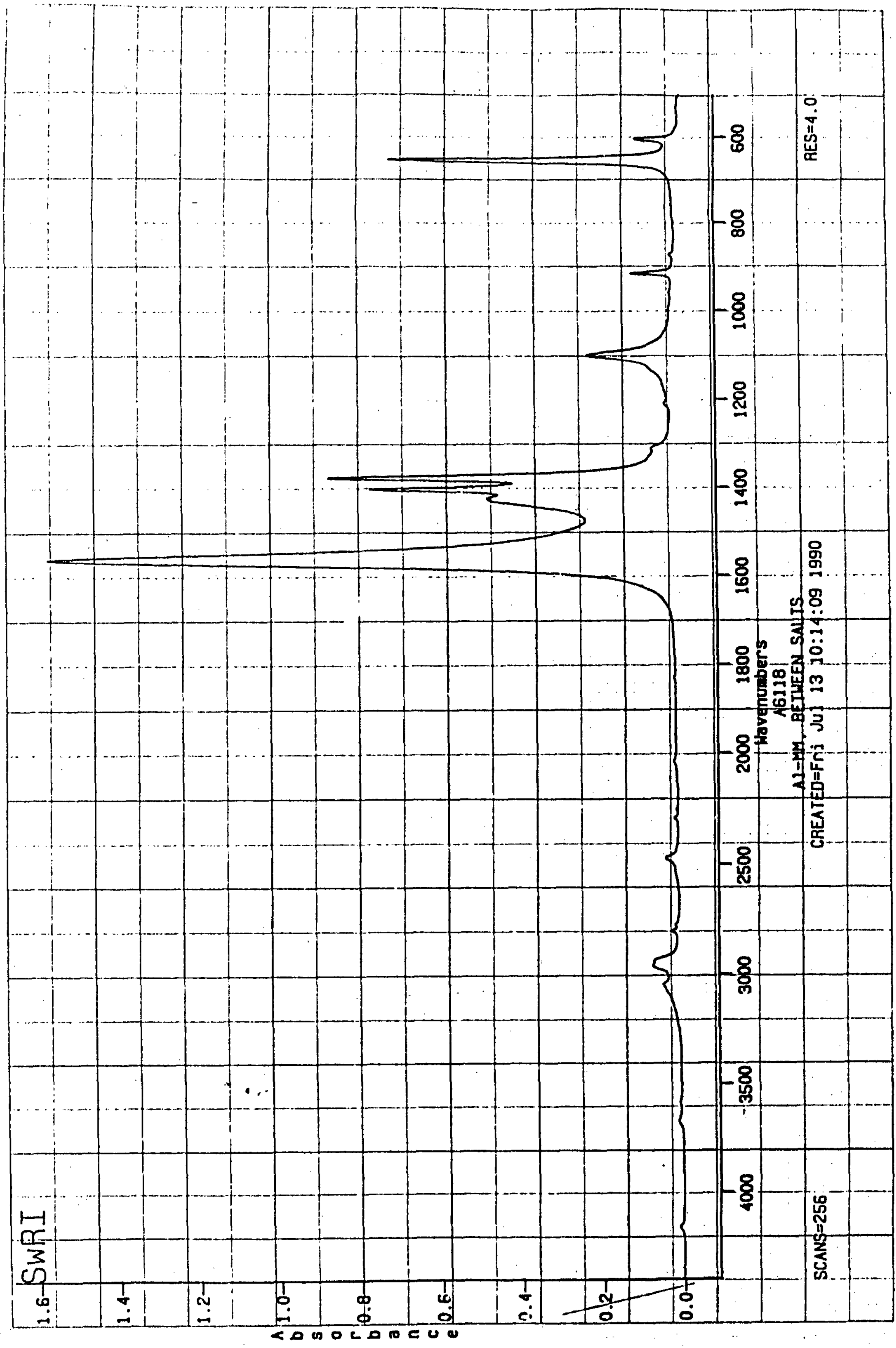
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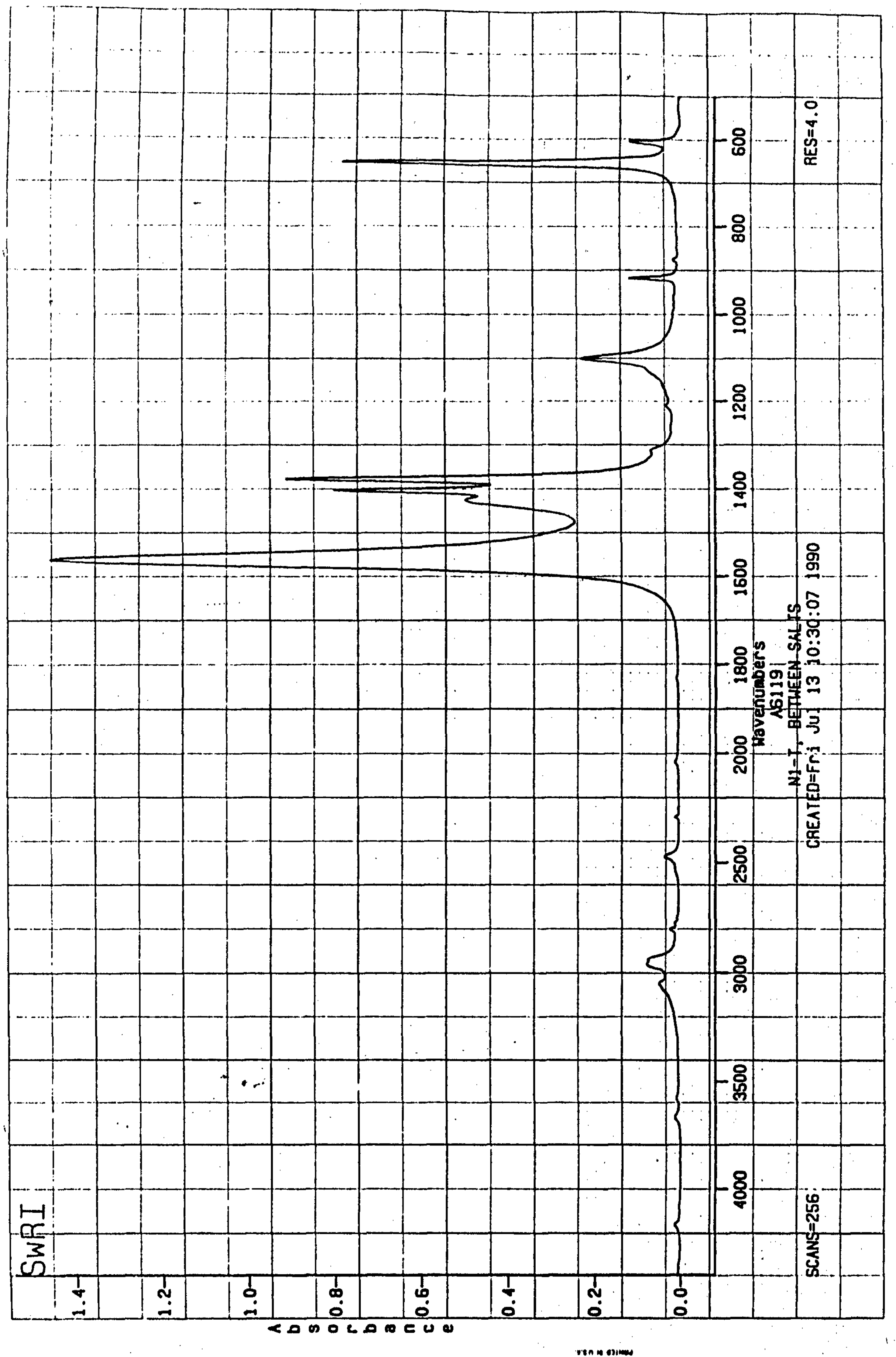
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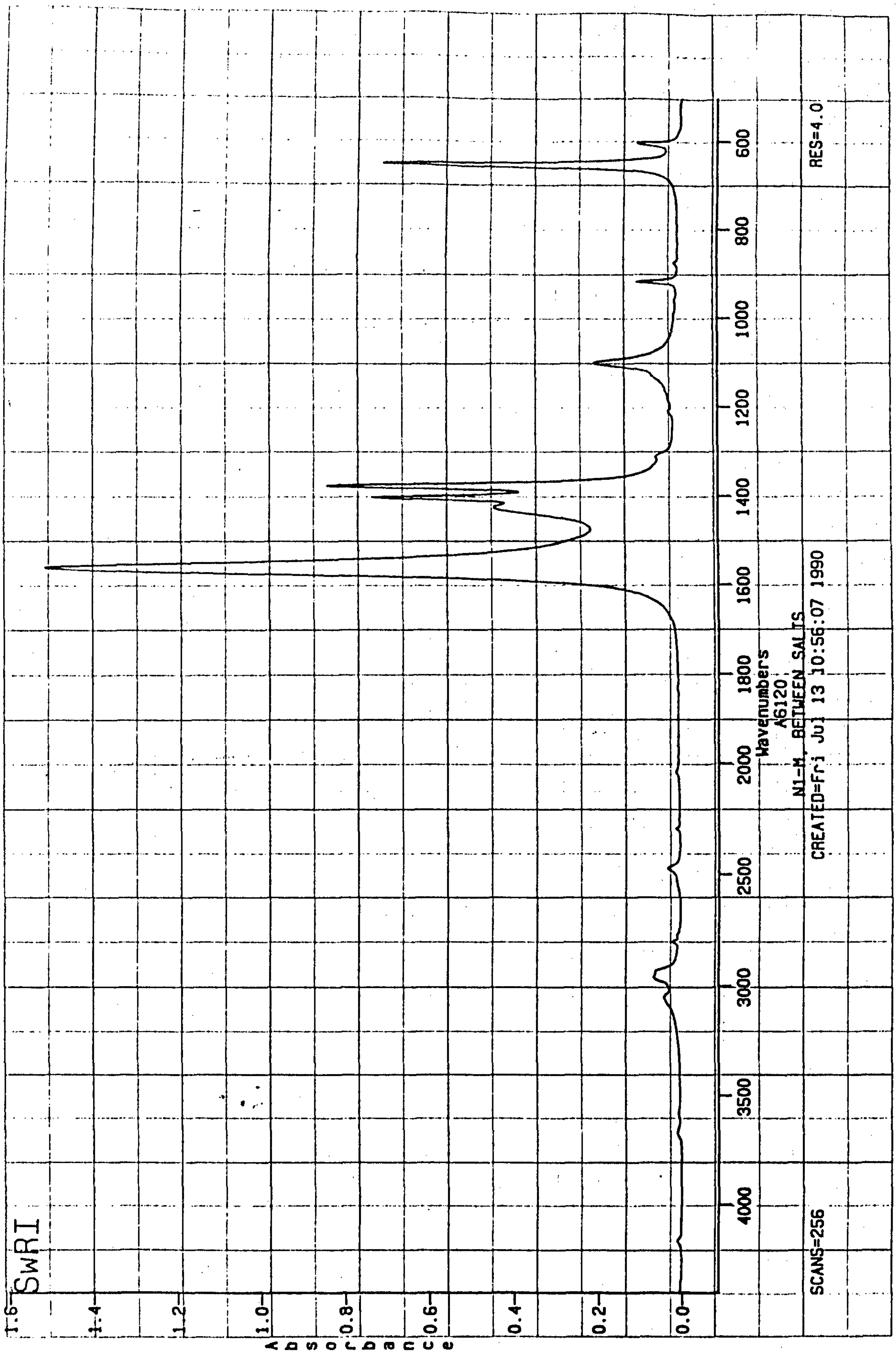
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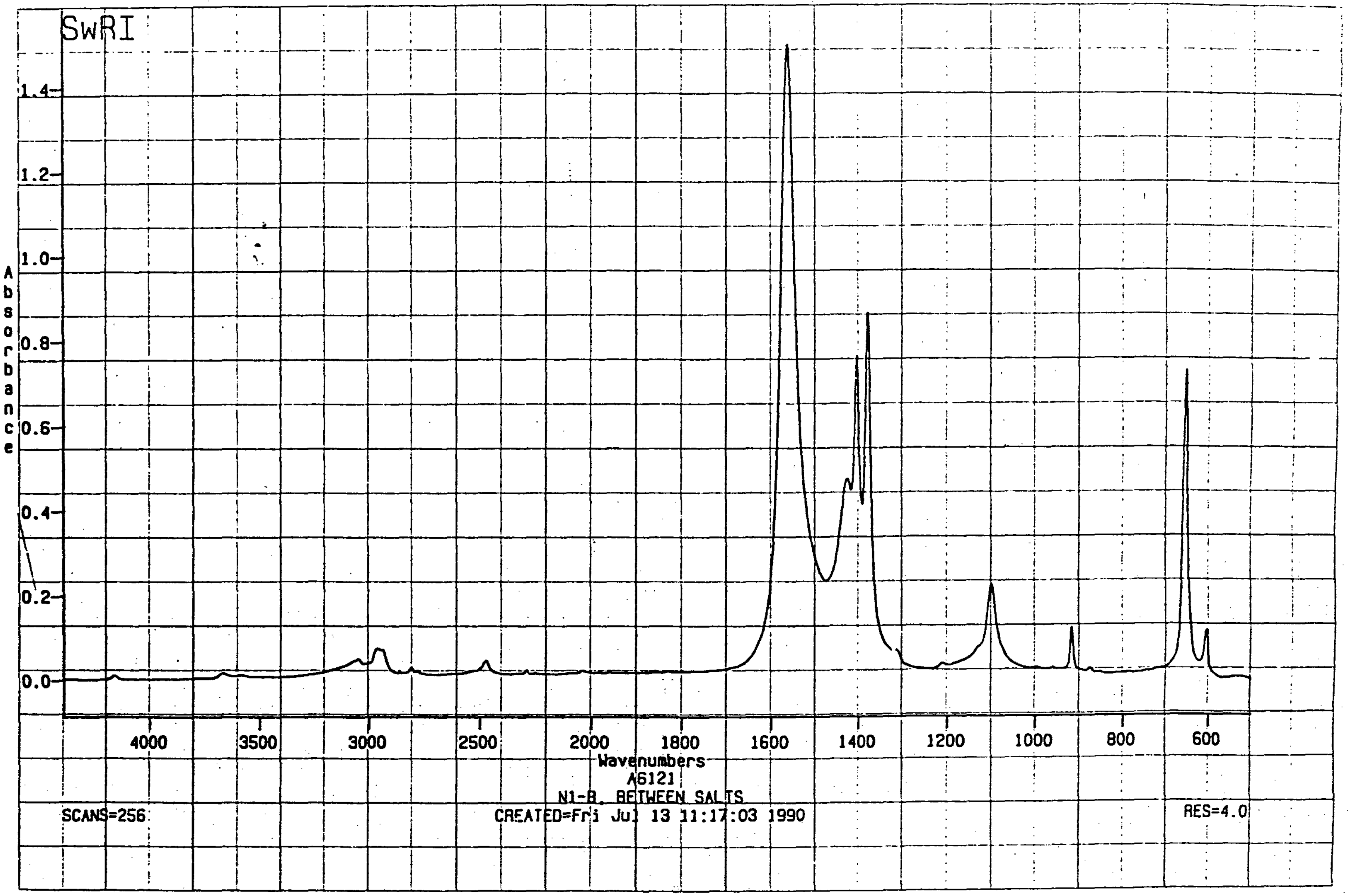
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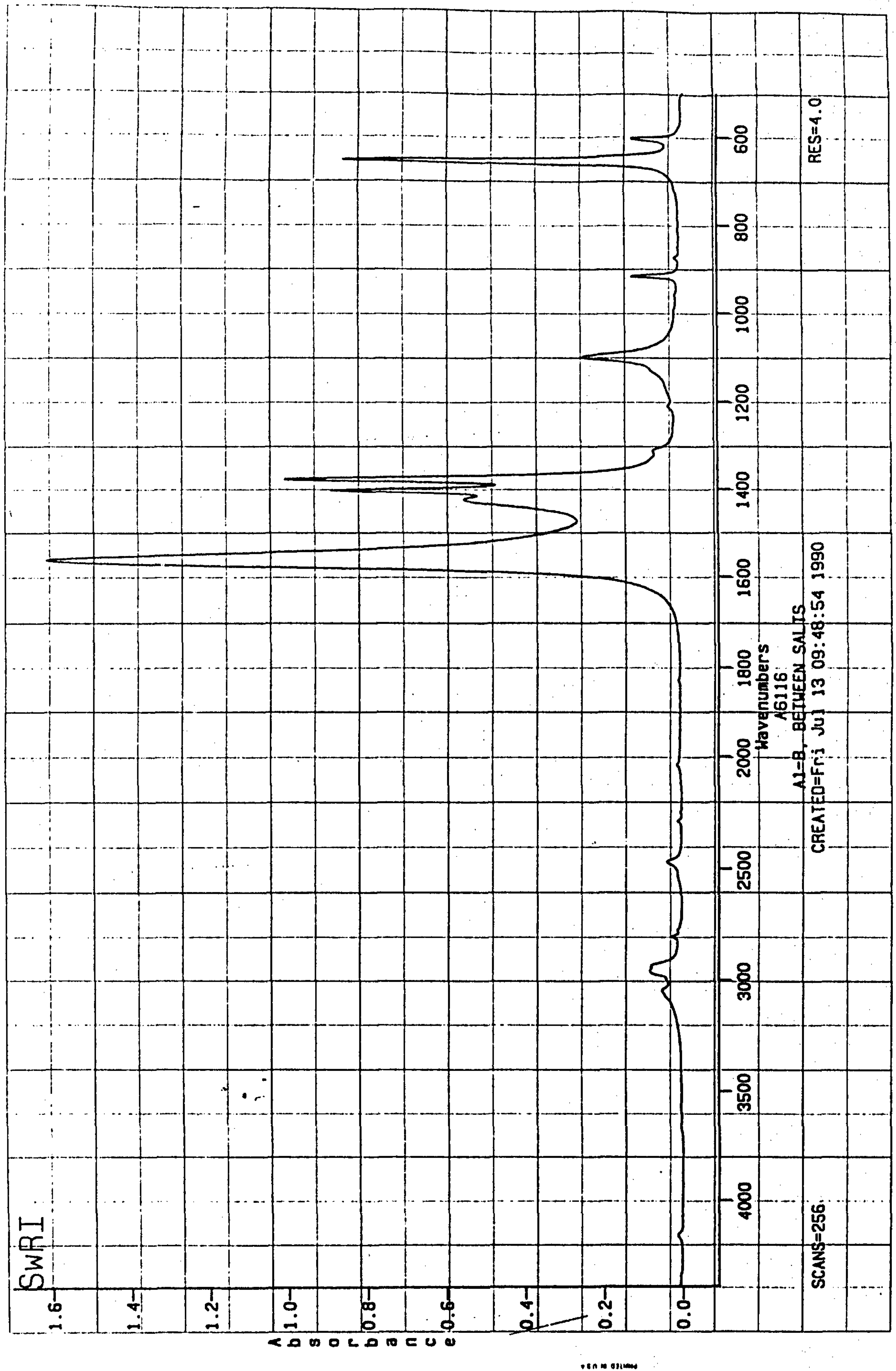
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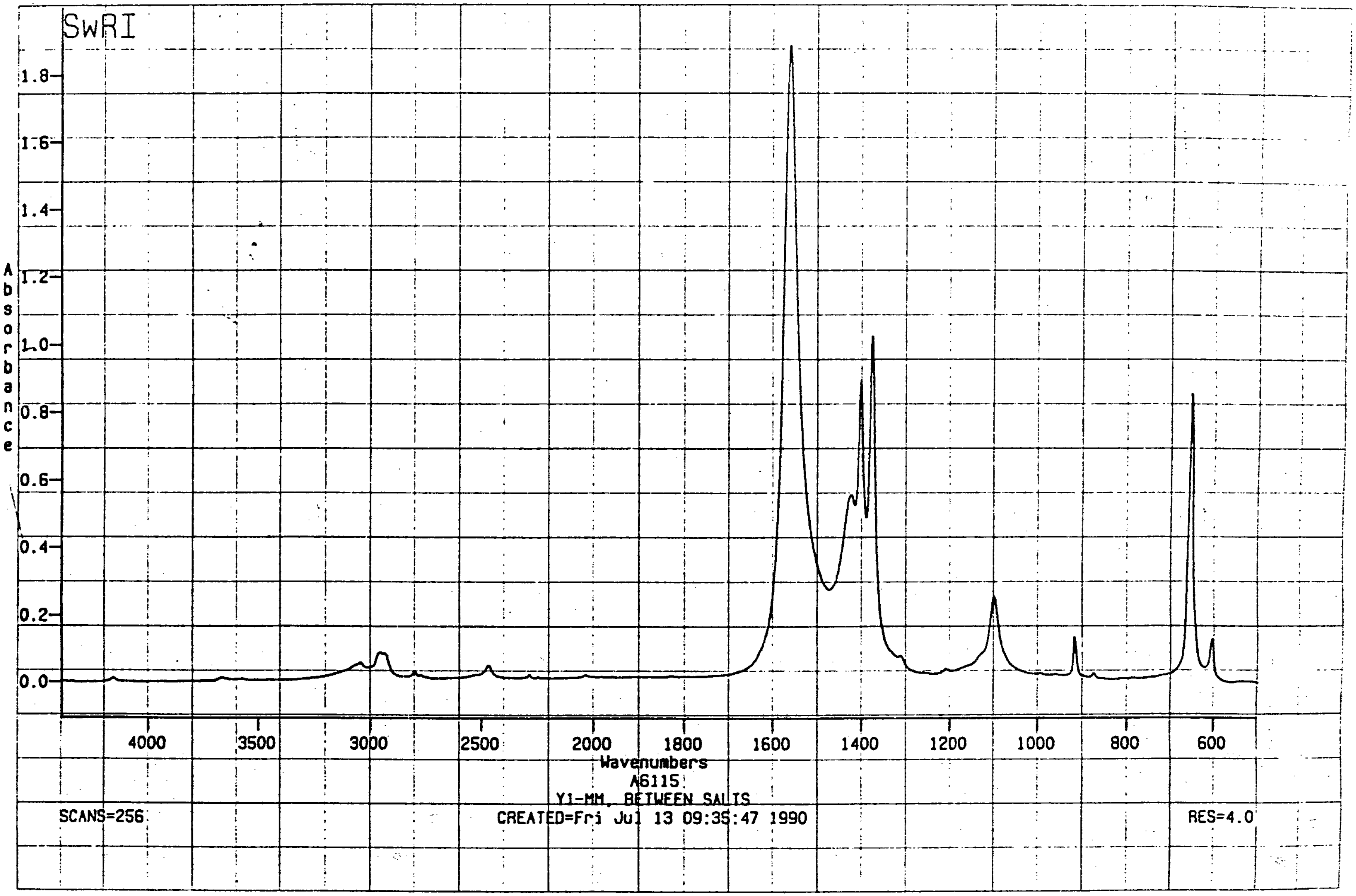SwRI
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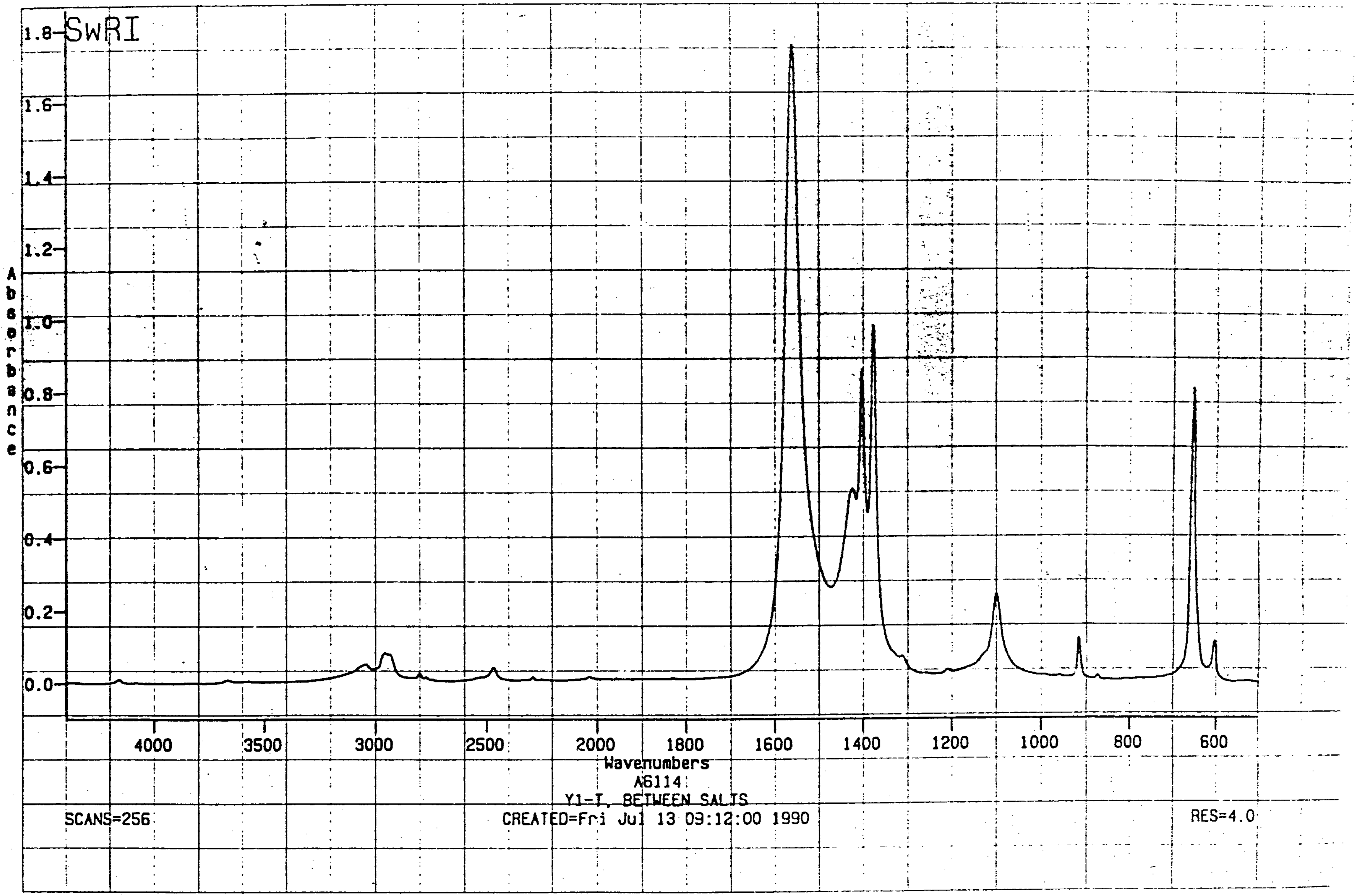
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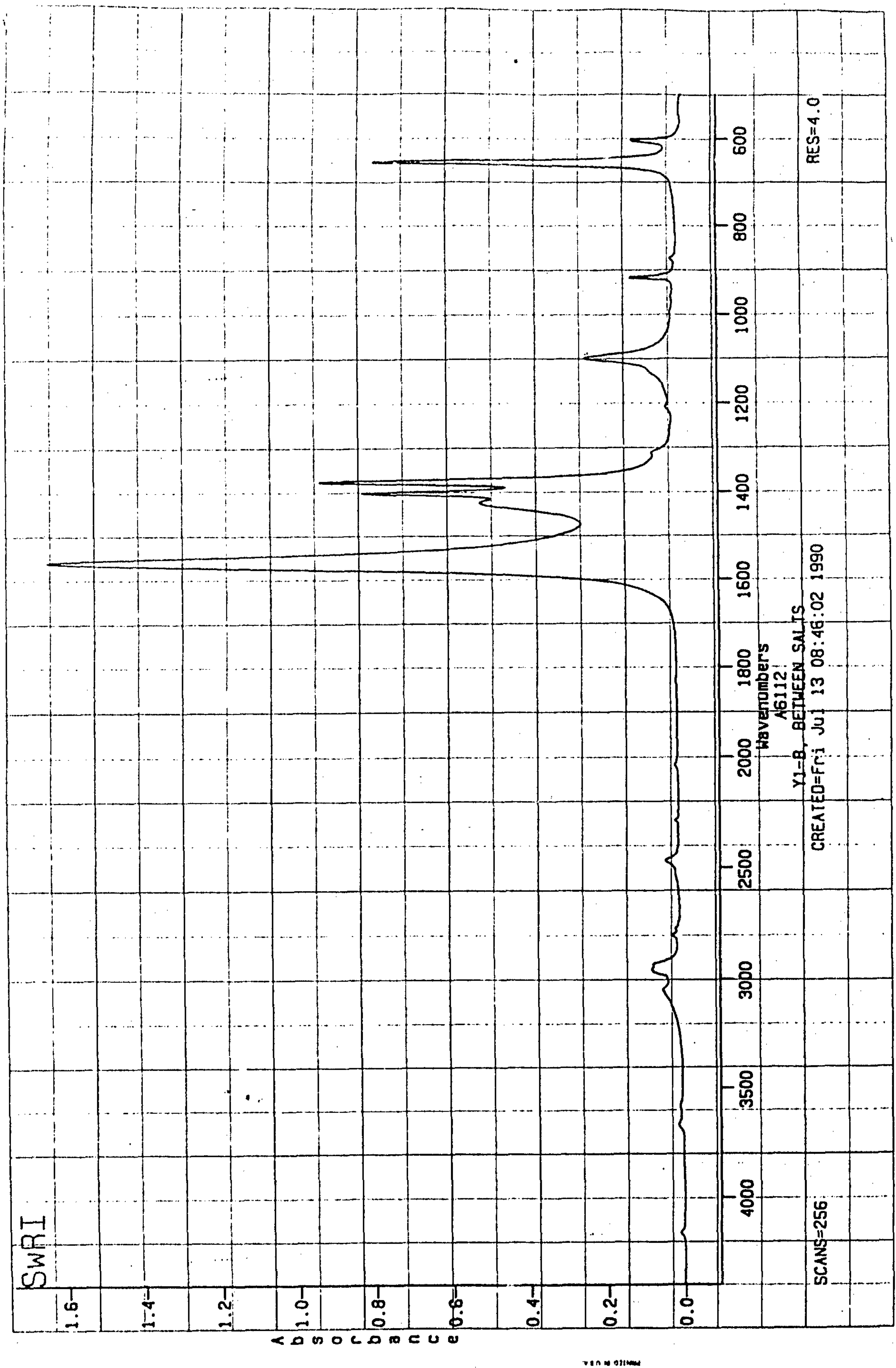
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Wavenumbers
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APPENDIX C

VOLATILE ANALYSES

```
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SAMPLE: NITROMETHANE/2-NITROPROPANE 5000NG STD:1UL PURGE
CONDS.: 30DEG 4MIN/4DEG/MIN/FINAL 180 DEG
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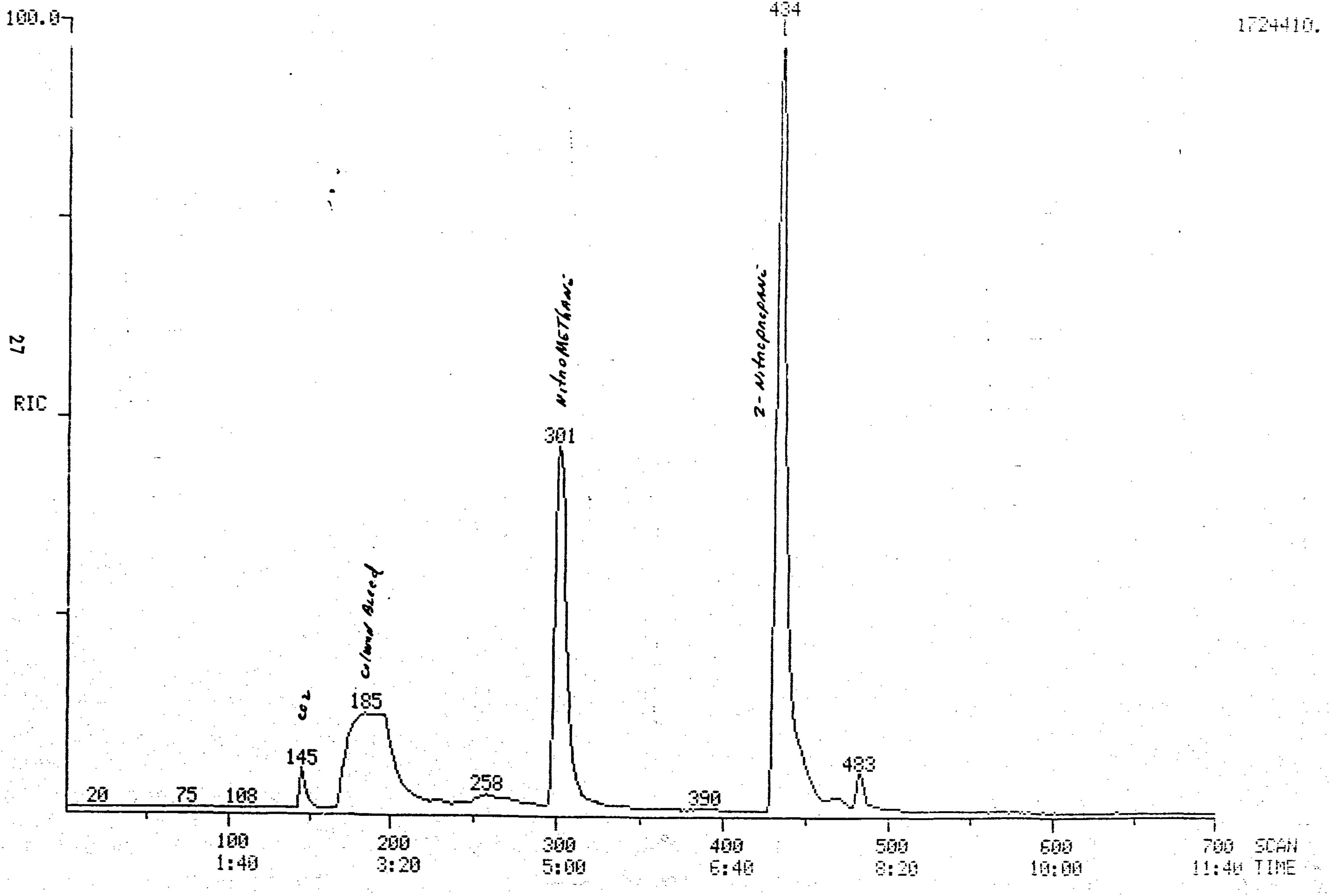

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CONDS.: 30DEG 4MIN/4DEG/MIN/FINAL 180 DEG
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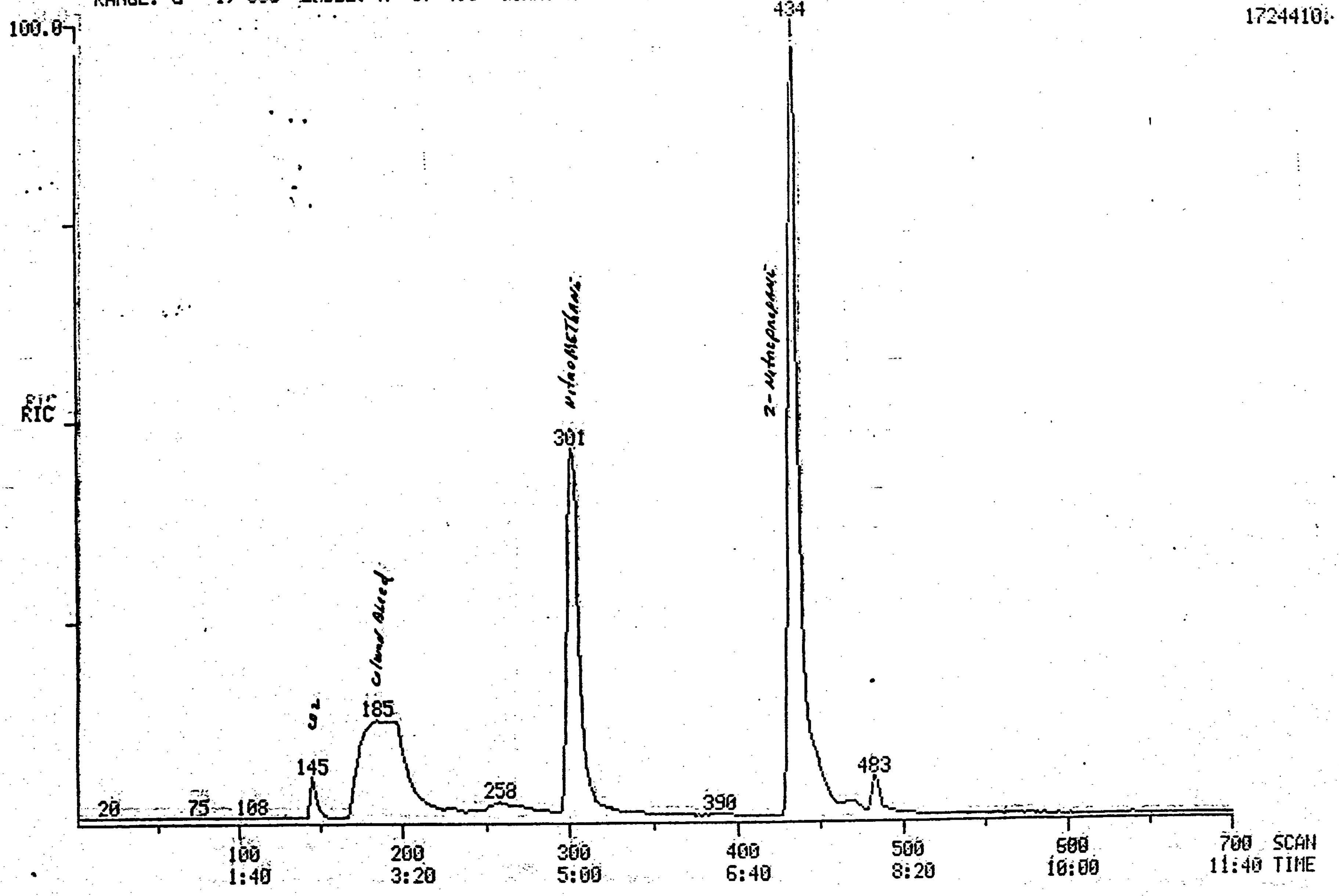

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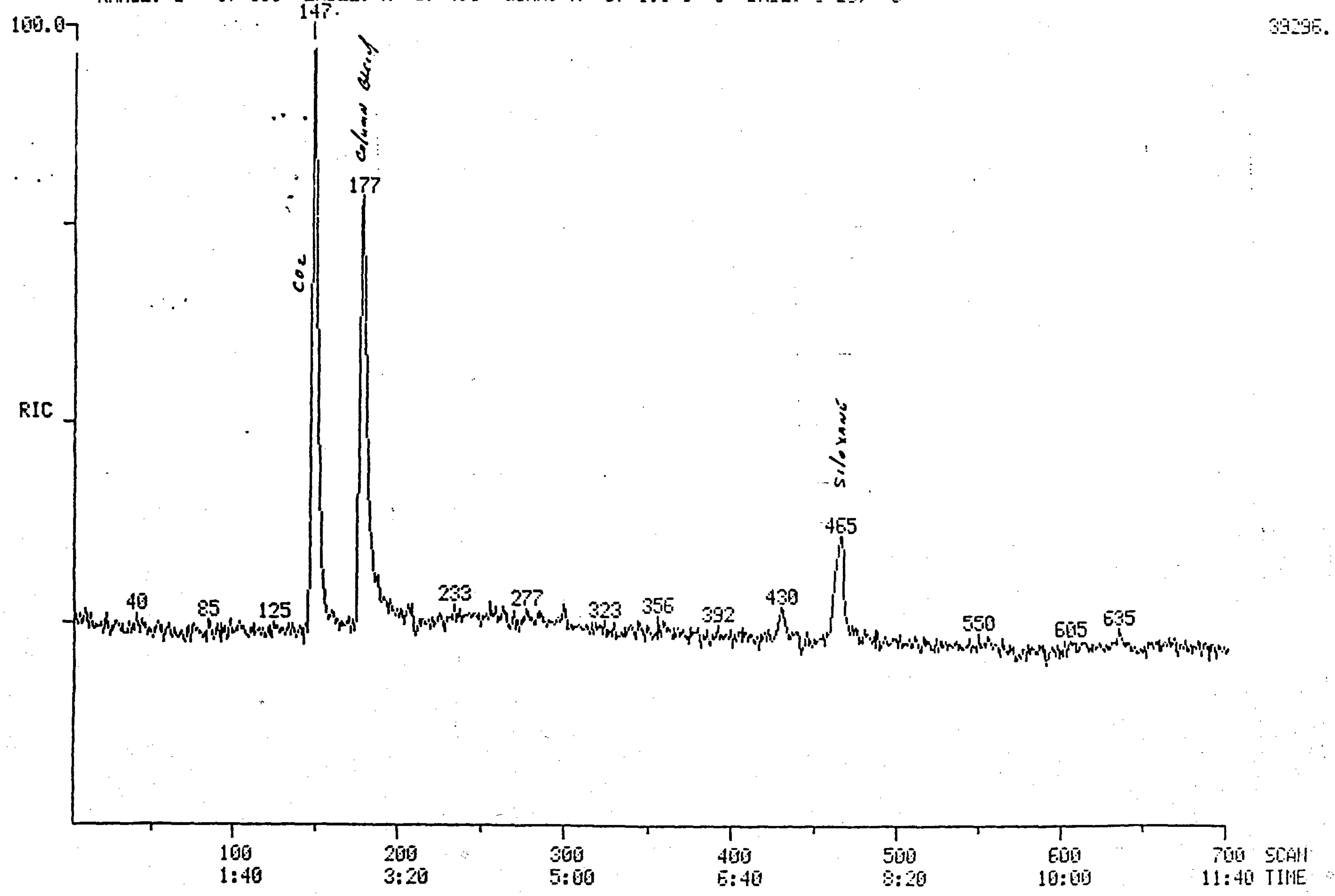

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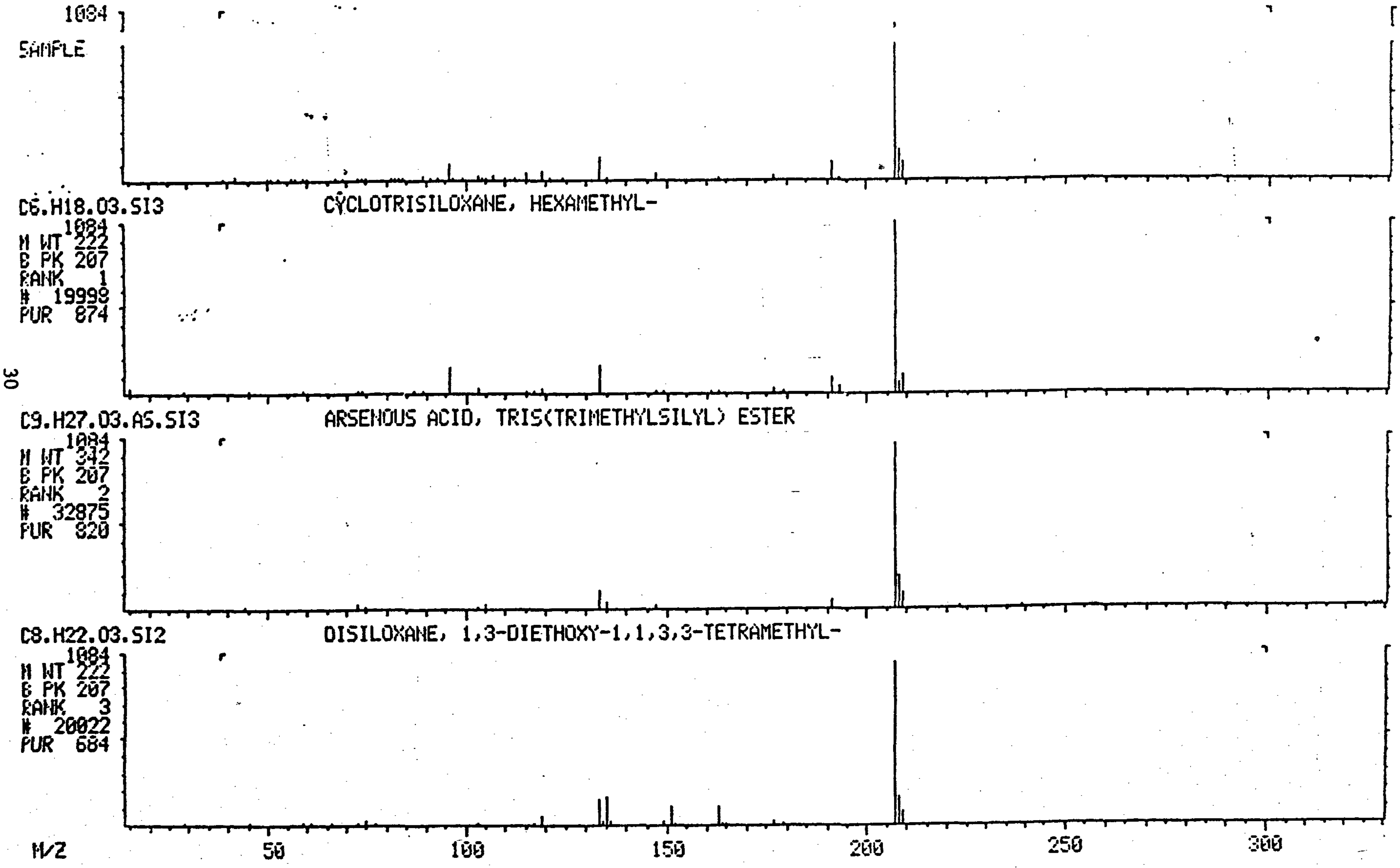

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1084
SAMPLE

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   1084
M WT 222
B PK 207
RANK   1
#  19999
PUR  874

C9.H27.O3.AS.SI3       ARSENOUS ACID, TRIS(TRIMETHYLSILYL) ESTER
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M WT 342
B PK 207
RANK   2
#  32875
PUR  820

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 # 462 TO # 466 SUMMED - # 470 TO # 475 - # 432 TO # 443 X1.00
```
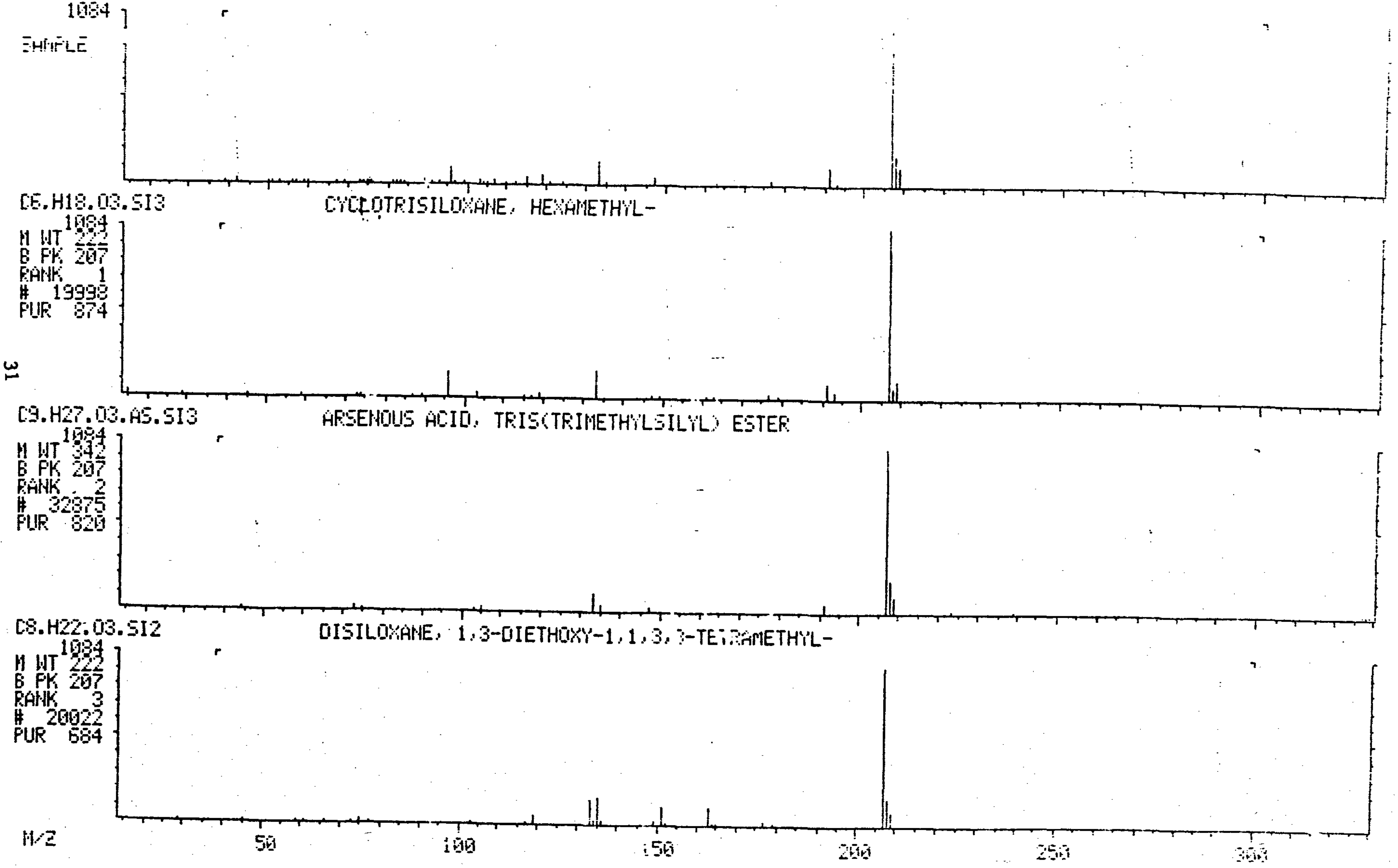
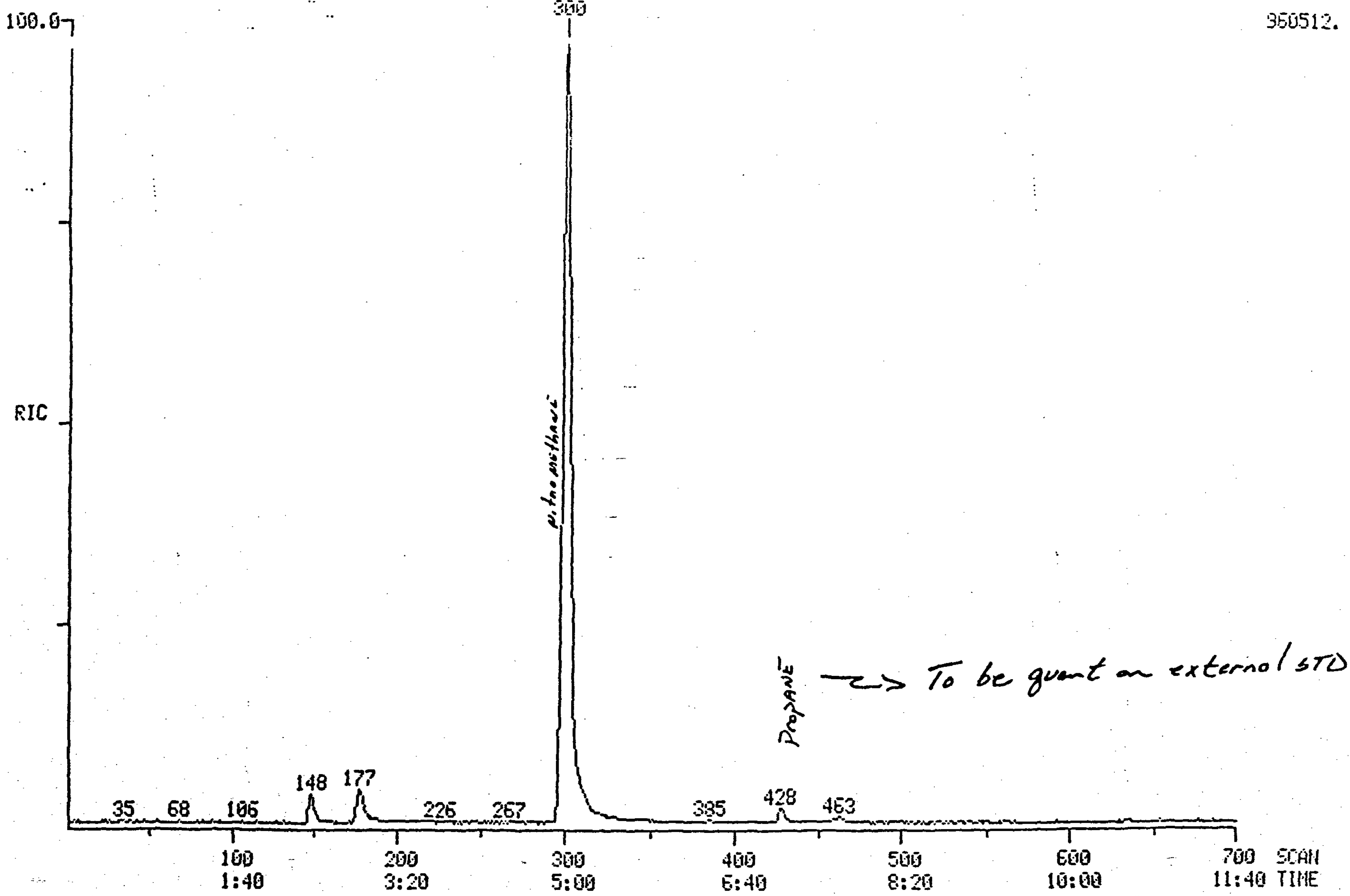



```
SAMPLE

C6.H18.O3.SI3        CYCLOTRISILOXANE, HEXAMETHYL-
M WT  222
B PK  207
RANK    1
#   19998
PUR   874

C9.H27.O3.AS.SI3     ARSENOUS ACID, TRIS(TRIMETHYLSILYL) ESTER
M WT  342
B PK  207
RANK    2
#   32875
PUR   820

C8.H22.O3.SI2        DISILOXANE, 1,3-DIETHOXY-1,1,3,3-TETRAMETHYL-
M WT  222
B PK  207
RANK    3
#   20022
PUR   684
```

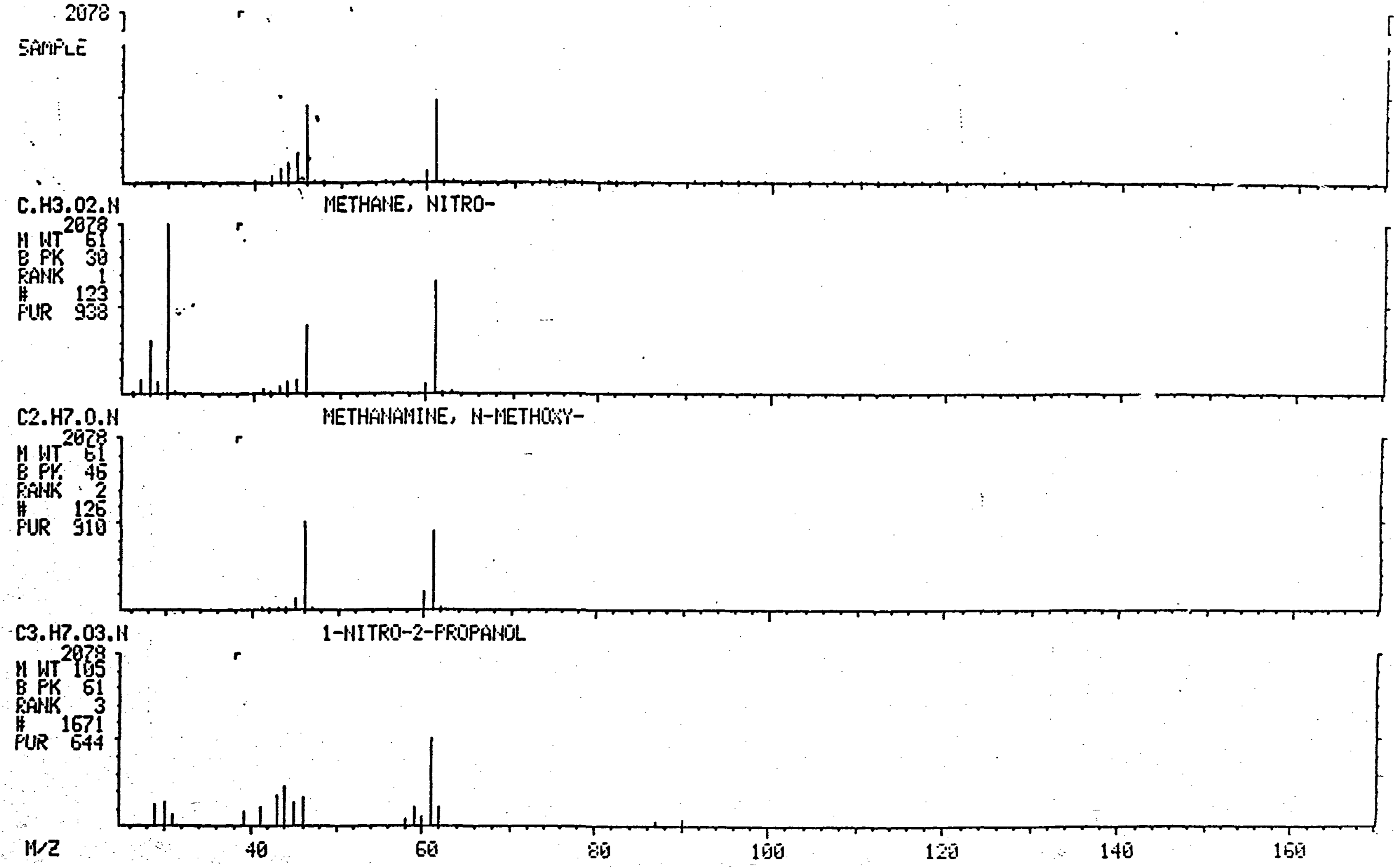
```
RIC                                             DATA: C0621001 #427      SCANS    1 TO  700
06/21/90 22:21:00                               CALI: C06150F1 #3
SAMPLE: B.H.:A1-B (SW):1UL(50UL SAMPLE/10ML MEOH)
CONDS.: 30DEG 4MIN/4DEG/MIN/FINAL 180 DEG
RANGE: G   1, 485  LABEL: N  0, 4.0  QUAN: A  0, 1.0 J  0  BASE: U 20,  3
```



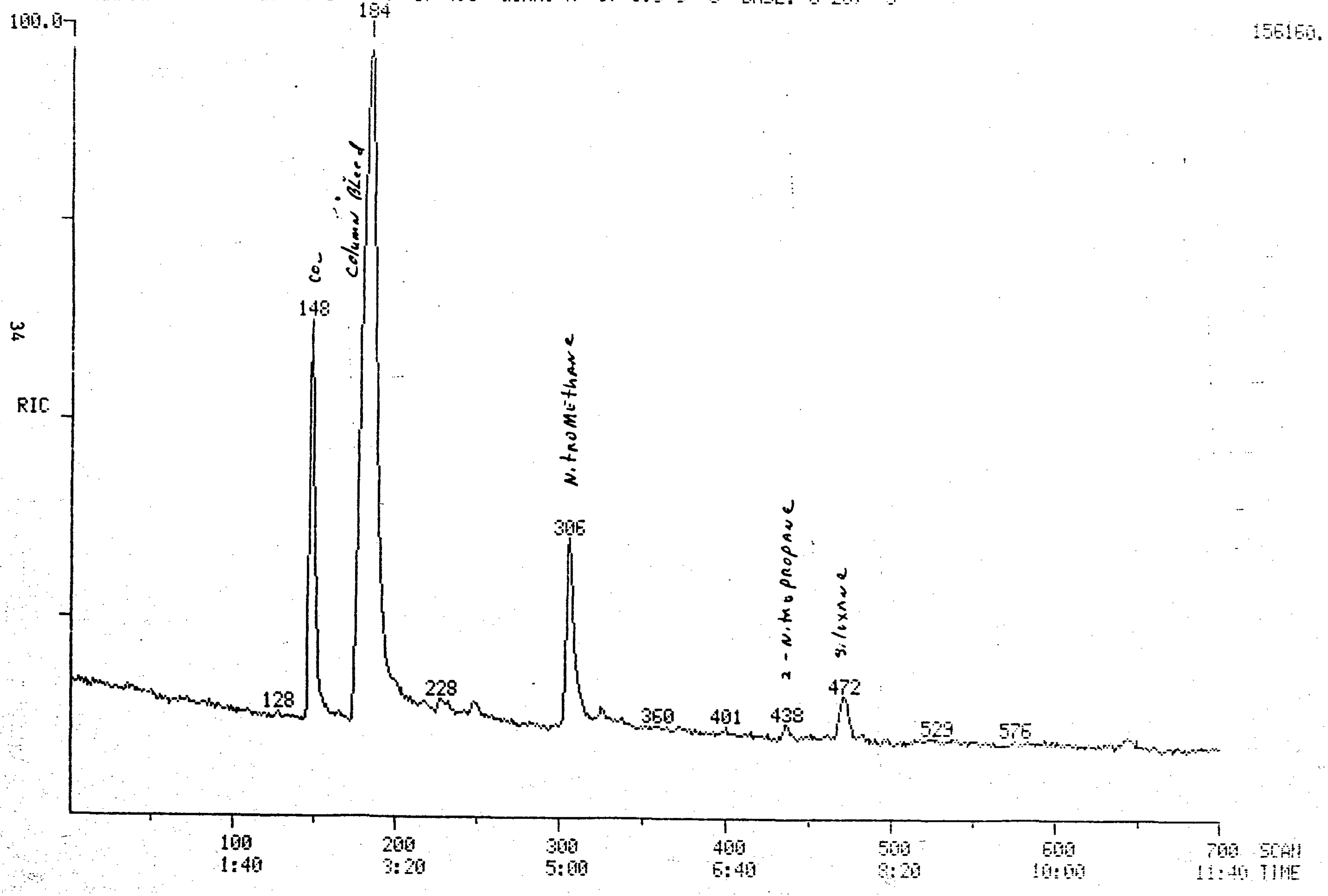
LIBRARY SEARCH
06/21/90 22:21:00 + 5:01
SAMPLE: B.H.:A1-B (SW):1UL(50UL SAMPLE/10ML MEOH)
CONDS.: 30DEG 4MIN/4DEG/MIN/FINAL 180 DEG
300 TO # 302 SUMMED - # 309 TO # 315 - # 280 TO # 290 X1.00

DATA: C0621001 # 301
CALI: C0615GF1 # 3

BASE M/Z: 61
RIC: 2203640.



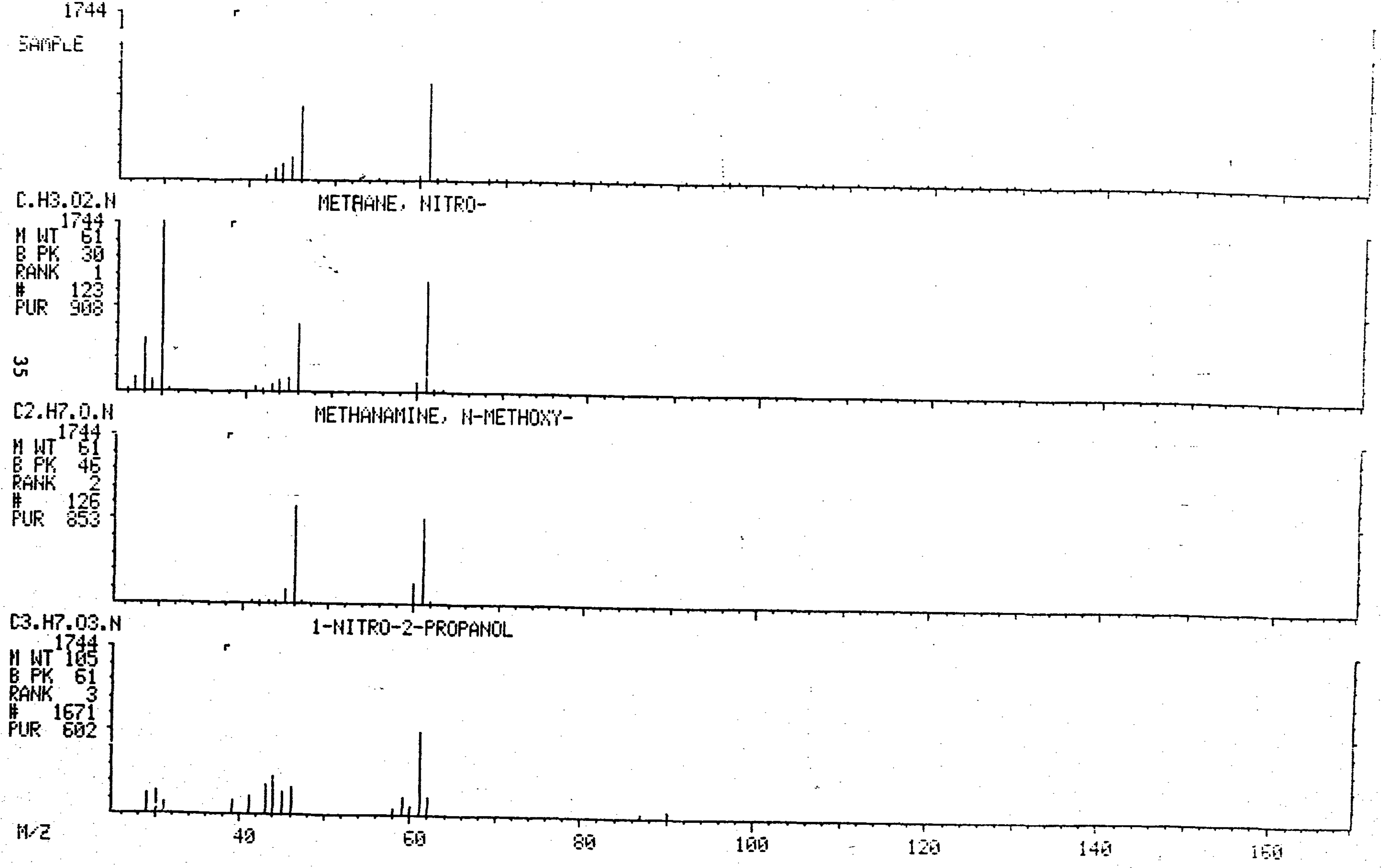
```
RIC                                      DATA: C0621002 #434      SCANS   1 TO 700
06/22/90  0:22:00                        CALI: C06150F1 #3
SAMPLE: B.H. :1UL (100UL/2ML MEOH)50UL/10ML MEOH
CONDS.: 30DEG 4MIN/4DEG/MIN/FINAL 180 DEG
RANGE: G   1, 629  LABEL: N  0, 4.0  QUAN: A  0, 1.0 J  0  BASE: U 20,  3
```



LIBRARY SEARCH
06/22/90 0:22:00 + 5:06
SAMPLE: B.H. :1UL (100UL/2ML MEOH)50UL/10ML MEOH
CONDS.: 30DEG 4MIN/4DEG/MIN/FINAL 180 DEG
 # 304 TO # 308 SUMMED - # 275 TO # 293 - # 333 TO # 363 X1.00

DATA: C0621002 # 306
CALI: C06150F1 # 3

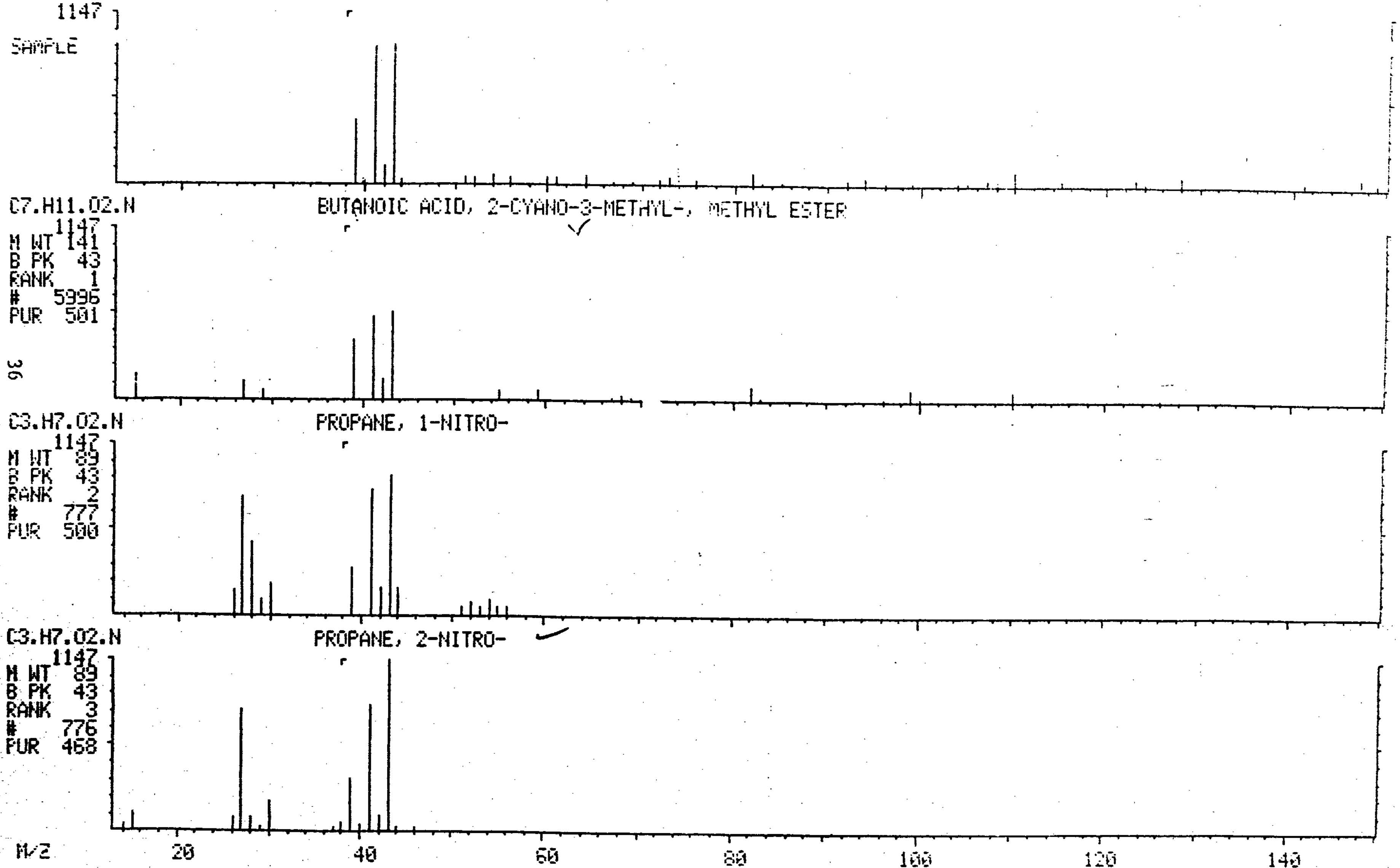
BASE M/Z: 61
RIC: 148583.



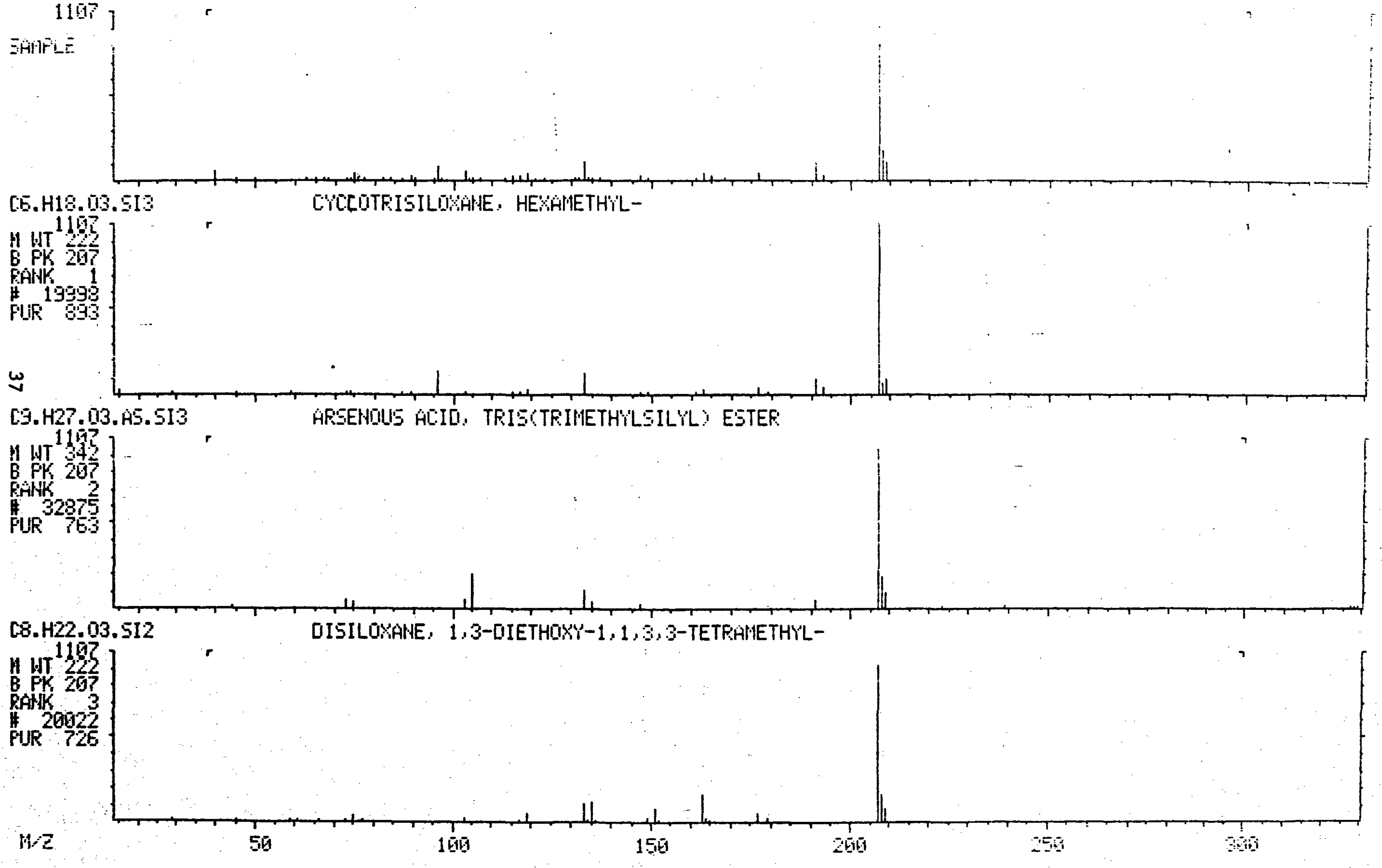
```
LIBRARY SEARCH
06/22/90  0:22:00 + 7:16
SAMPLE: B.H. :1UL (100UL/2ML MEOH)50UL/10ML MEOH
CONDS.: 30DEG 4MIN/4DEG/MIN/FINAL 180 DEG
 # 434 TO # 439 SUMMED - # 448 TO # 456 - # 414 TO # 424 X1.00
```

```
DATA: C0621002 # 435
CALI: C0615OF1 #   3
```

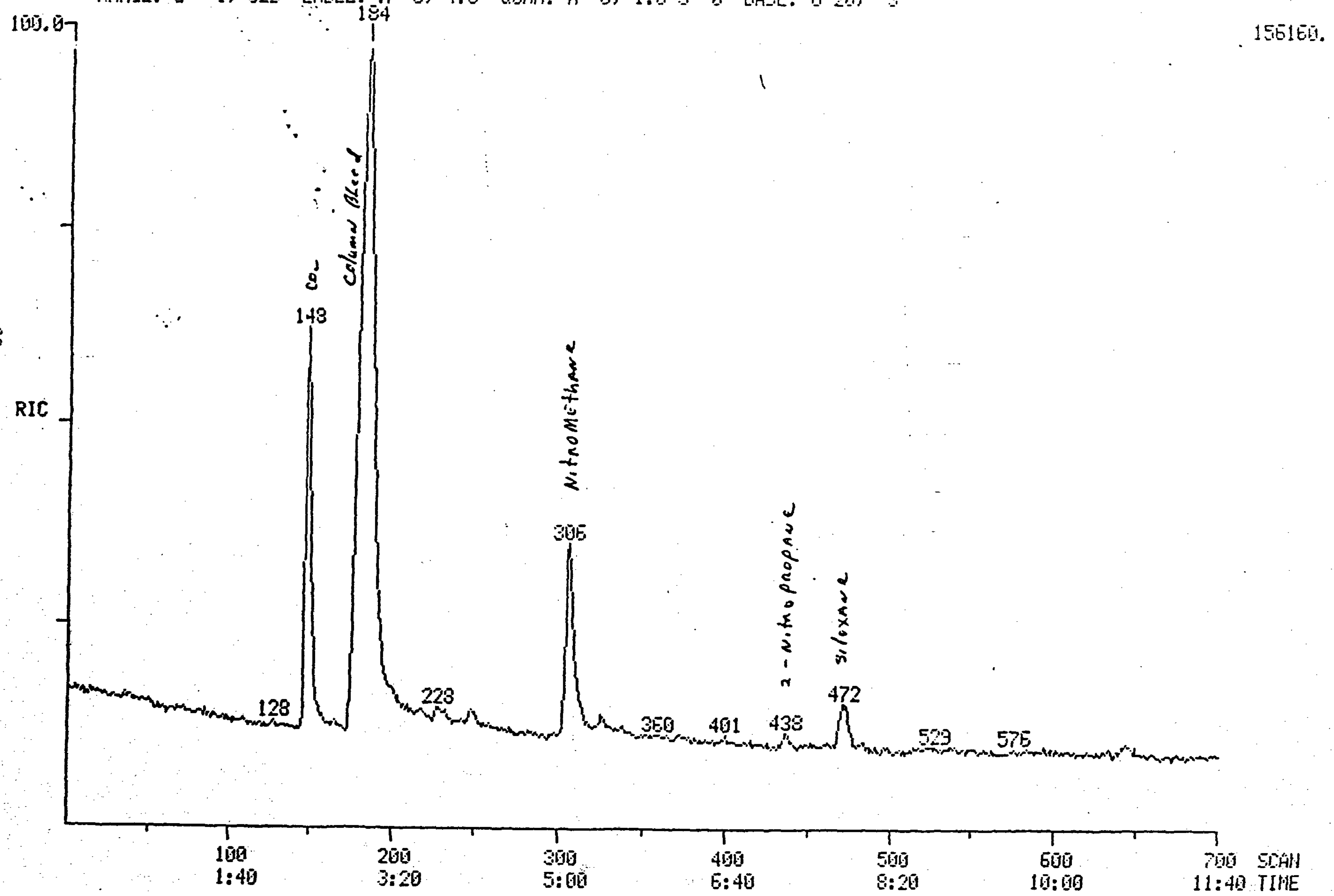
```
BASE M/Z:   41
RIC:     11551.
```



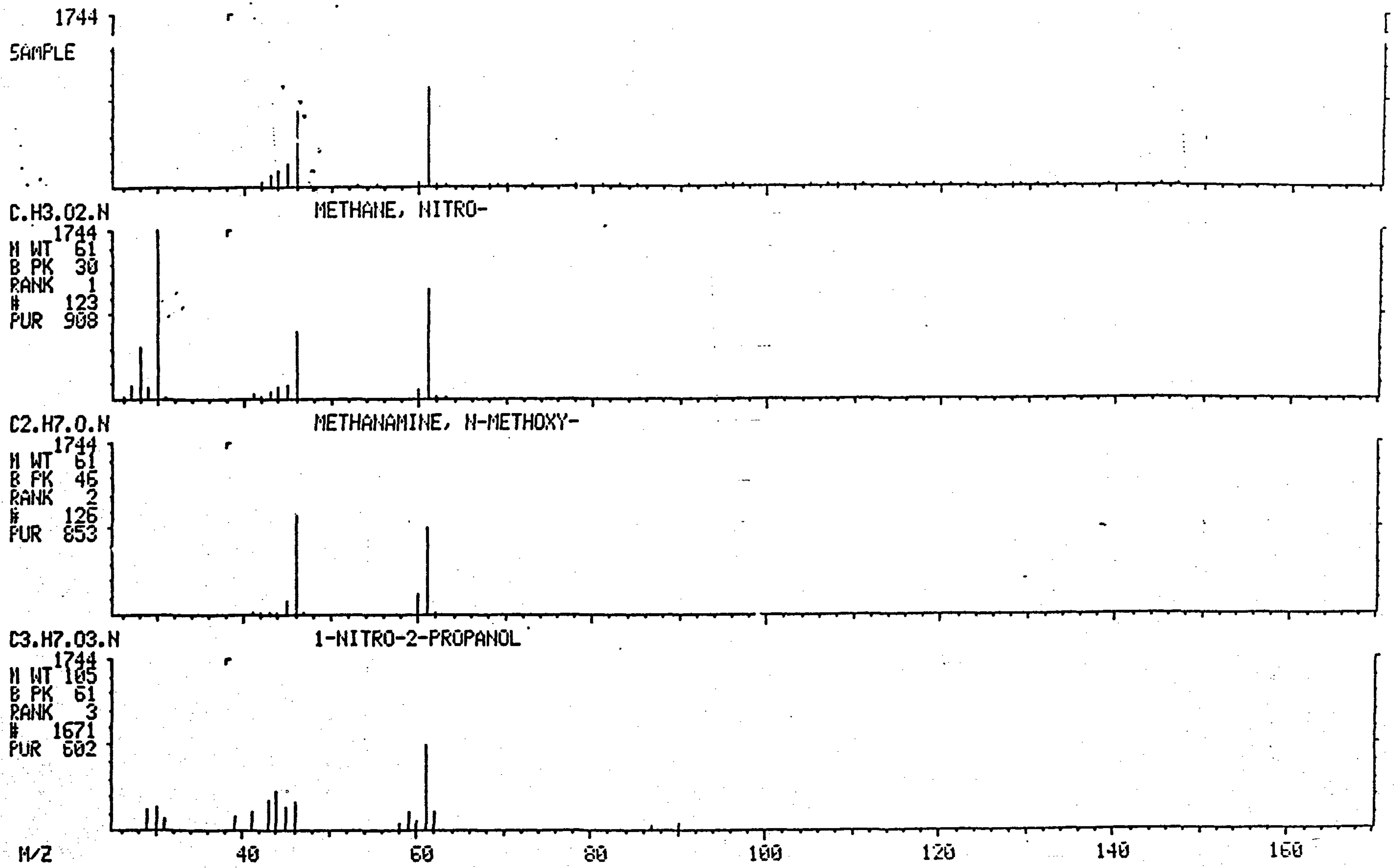
```
LIBRARY SEARCH                                              DATA: C0621002 # 473        BASE M/Z: 207
06/22/90  0:22:00 +  7:50                                   CALI: C06150F1 #   3        RIC:     41471.
SAMPLE: B.H. :1UL (100UL/2ML MEOH)50UL/10ML MEOH
CONDS.: 30DEG 4MIN/4DEG/MIN/FINAL 180 DEG
 # 468 TO # 473 SUMMED - # 485 TO # 499 - # 434 TO # 448 X1.00
```



```
RIC                                    DATA: C0621002 #434     SCANS    1 TO  700
06/22/90  0:22:00                      CALI: C06150F1 #3
SAMPLE: B.H. :1UL (100UL/2ML MEOH)50UL/10ML MEOH
CONDS.: 30DEG 4MIN/4DEG/MIN/FINAL 180 DEG
RANGE: G   1, 629  LABEL: N  0, 4.0  QUAN: A  0, 1.0 J  0  BASE: U 20,  3
```



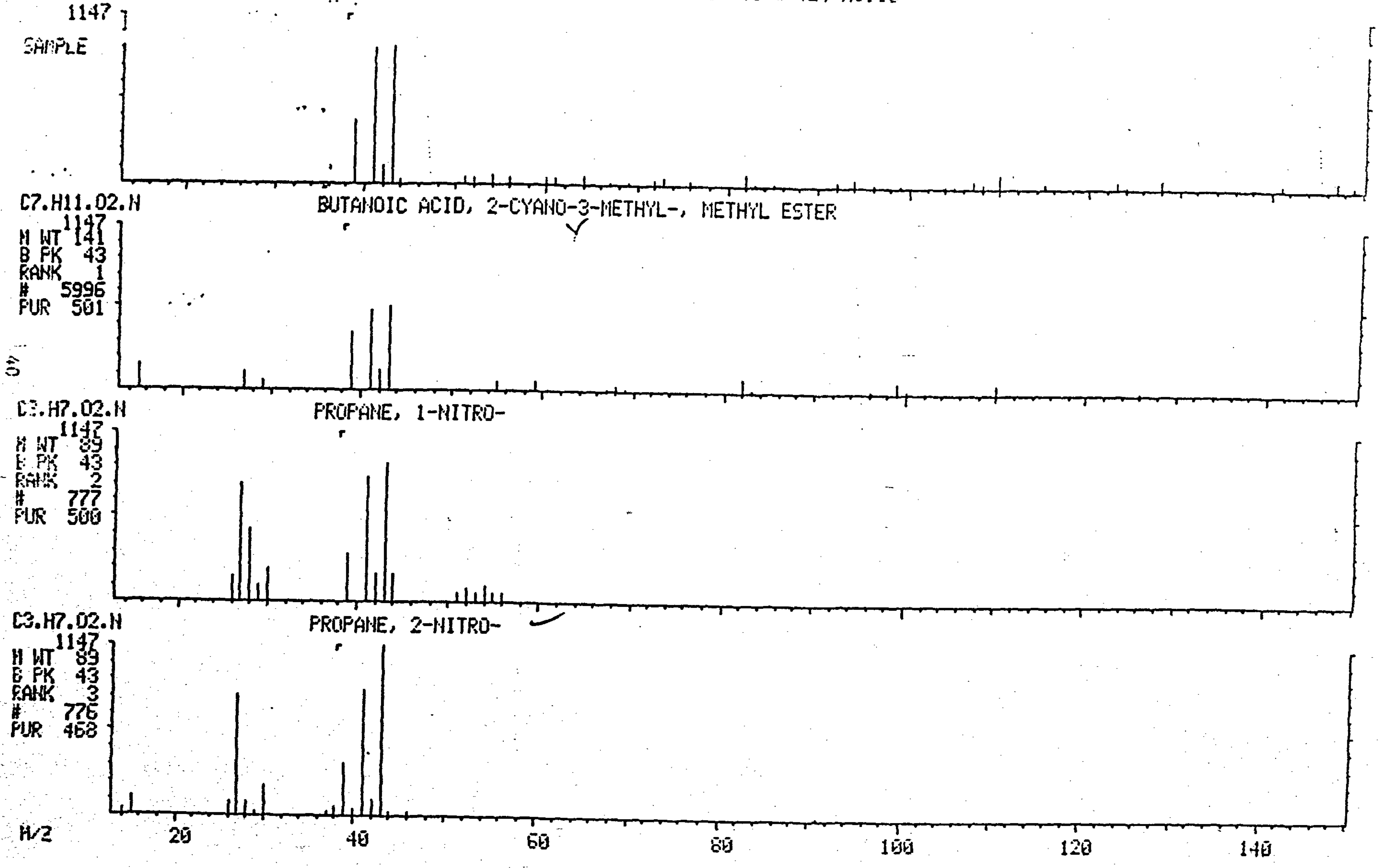
```
LIBRARY SEARCH                                           DATA: C0621002 # 306       BASE M/Z:  61
06/22/90  0:22:00 +  5:06                                CALI: C06150F1 #   3       RIC:    149503.
SAMPLE: B.H. :1UL (100UL/2ML MEOH)50UL/10ML MEOH
CONDS.: 30DEG 4MIN/4DEG/MIN/FINAL 180 DEG
 # 304 TO # 308 SUMMED - # 275 TO # 293 - # 333 TO # 363 X1.00
```

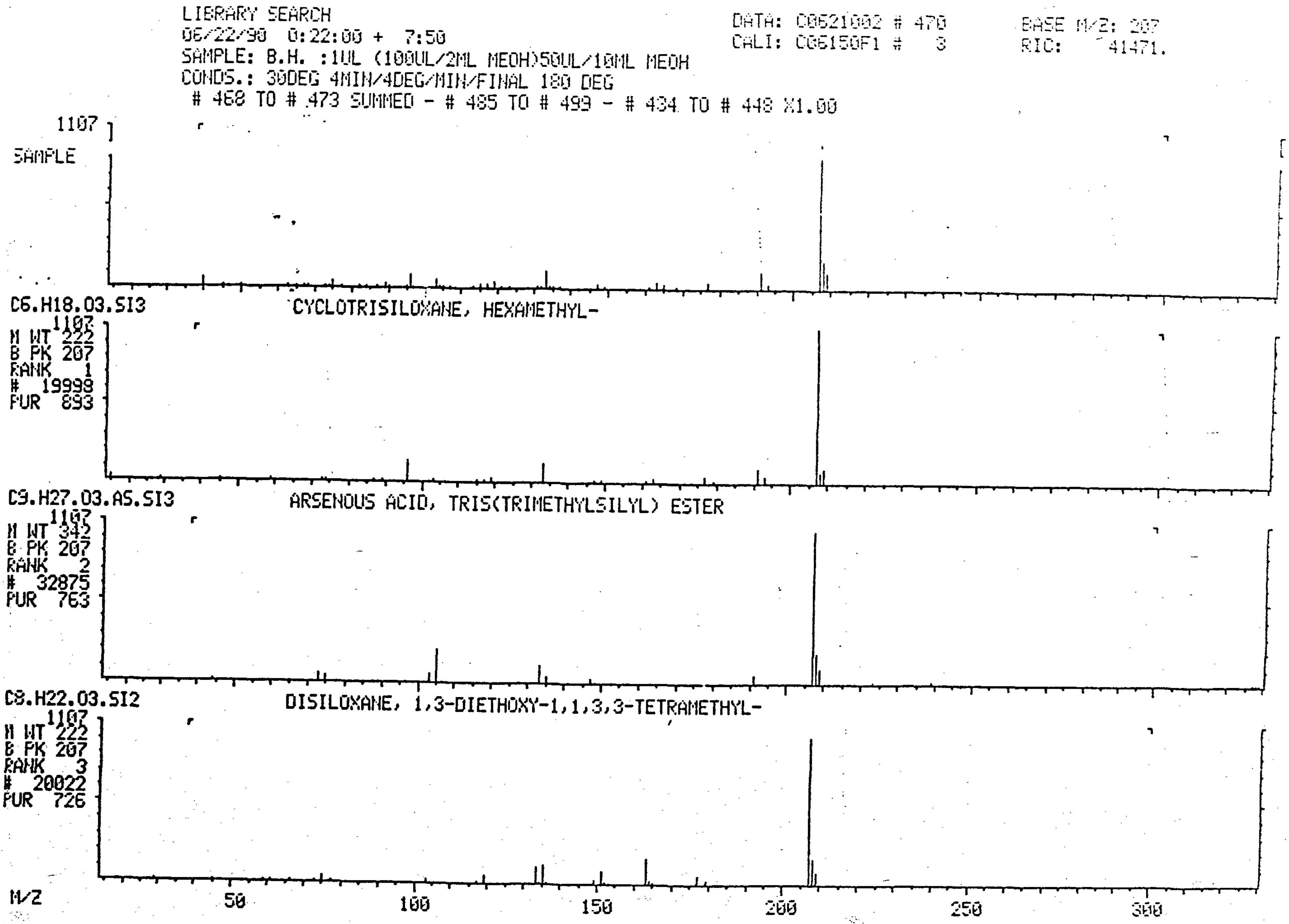


LIBRARY SEARCH
06/22/90 0:22:00 + 7:16
SAMPLE: B.H. :1UL (100UL/2ML MEOH)50UL/10ML MEOH
CONDS.: 30DEG 4MIN/4DEG/MIN/FINAL 180 DEG
434 TO # 439 SUMMED - # 448 TO # 456 - # 414 TO # 424 X1.00

DATA: C0621002 # 436
CALI: C06150F1 # 3

BASE M/Z: 43
RIC: 11951.





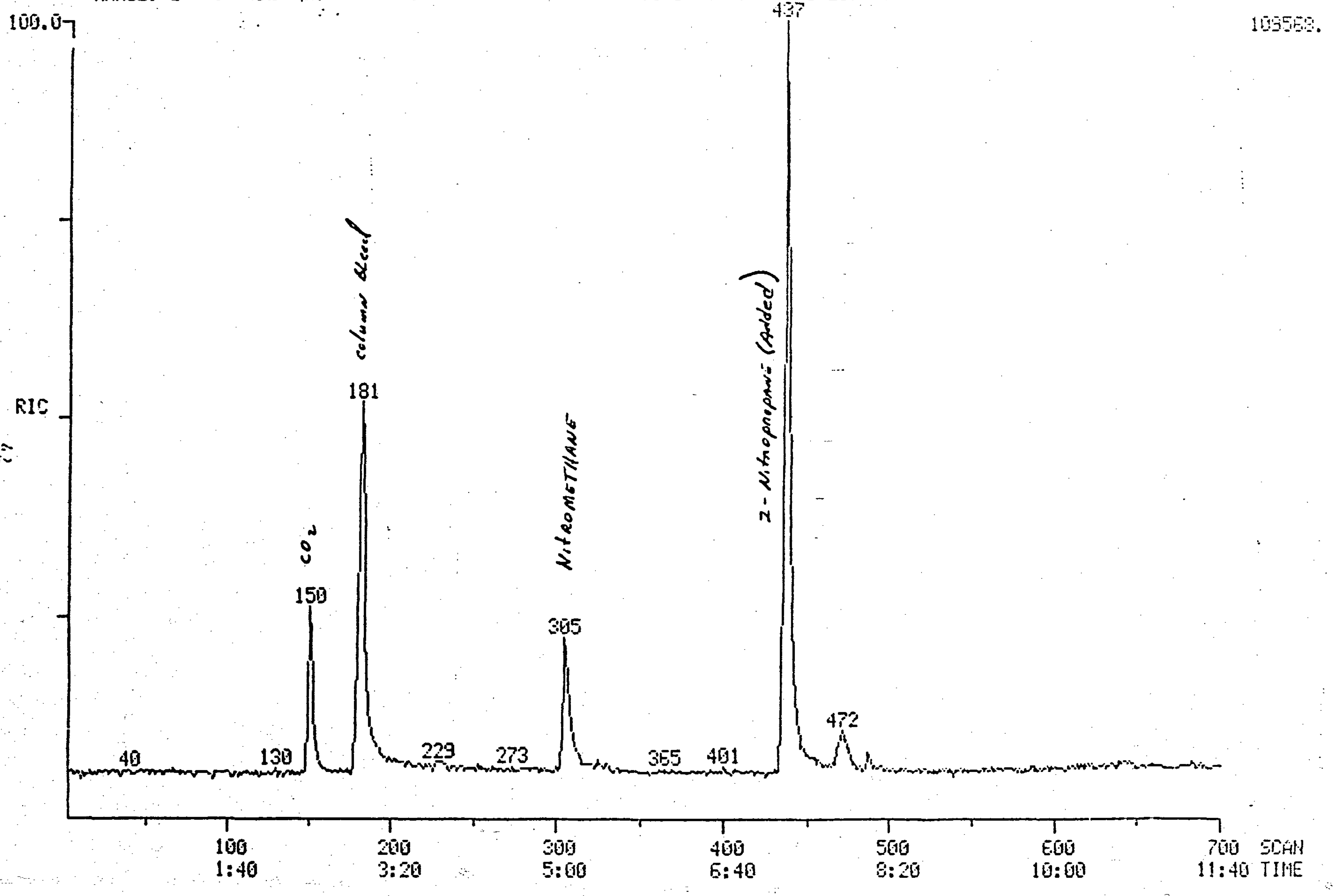
```
LIBRARY SEARCH                                          DATA: C0621002 # 470      BASE M/Z: 207
06/22/90  0:22:00 +  7:50                               CALI: C06150F1 #   3      RIC:    41471.
SAMPLE: B.H. :1UL (100UL/2ML MEOH)50UL/10ML MEOH
CONDS.: 30DEG 4MIN/4DEG/MIN/FINAL 180 DEG
 # 468 TO # 473 SUMMED - # 485 TO # 499 - # 434 TO # 448 X1.00
```

```
C6.H18.O3.SI3          CYCLOTRISILOXANE, HEXAMETHYL-
M WT 222
B PK 207
RANK   1
#  19998
PUR  893
```

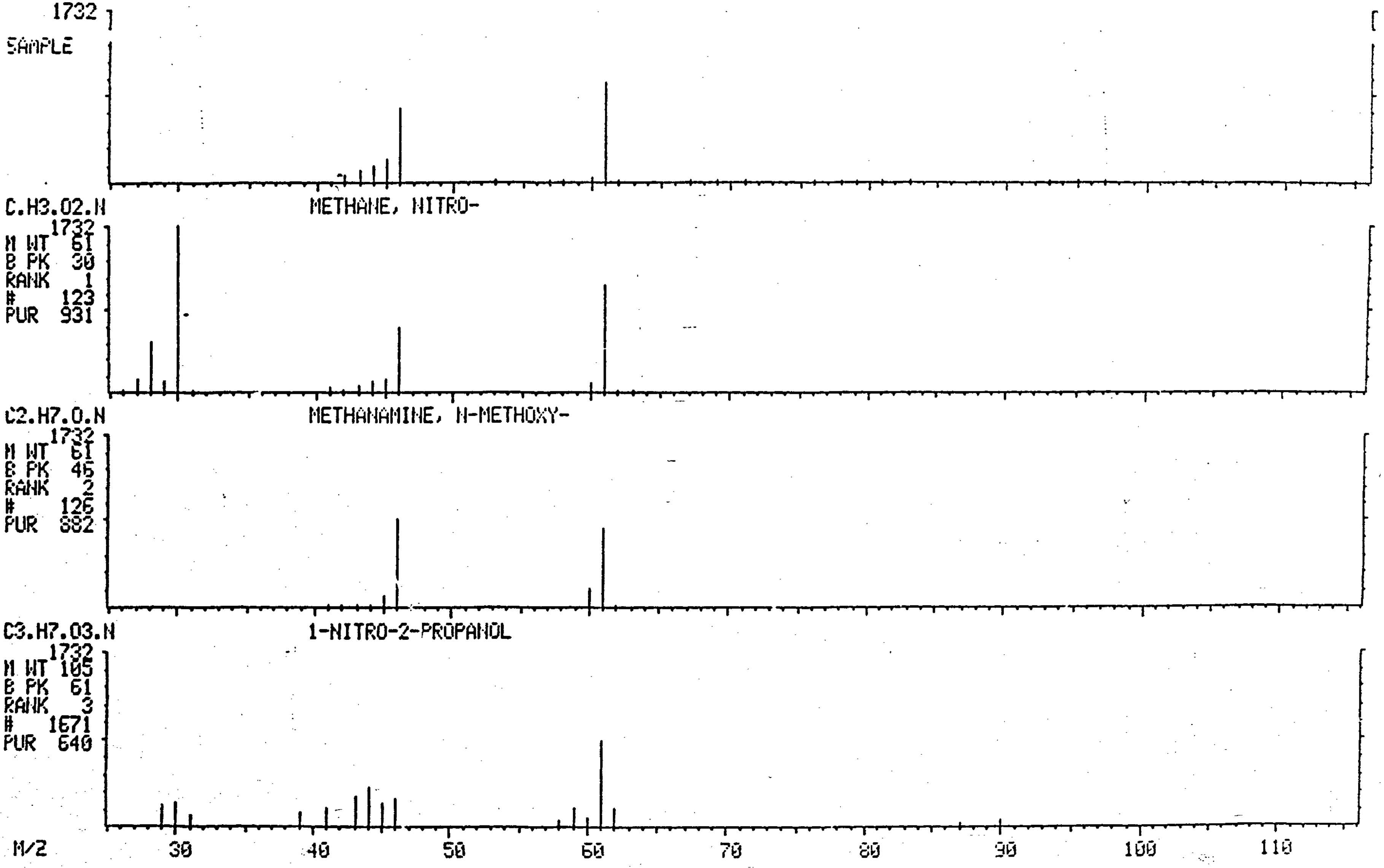
```
C9.H27.O3.AS.SI3       ARSENOUS ACID, TRIS(TRIMETHYLSILYL) ESTER
M WT 342
B PK 207
RANK   2
#  32875
PUR  763
```

```
C8.H22.O3.SI2          DISILOXANE, 1,3-DIETHOXY-1,1,3,3-TETRAMETHYL-
M WT 222
B PK 207
RANK   3
#  20022
PUR  726
```

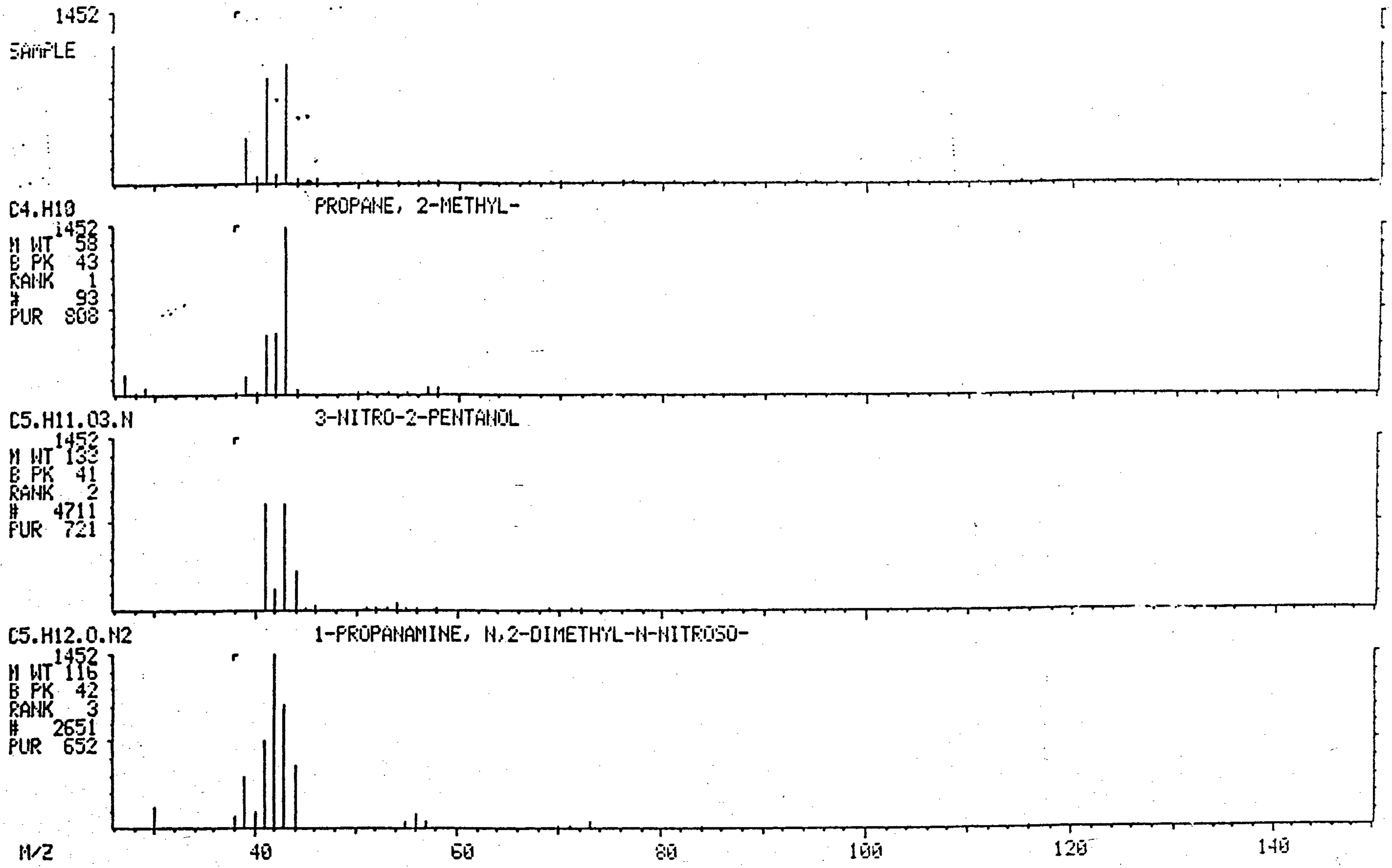
```
RIC                                       DATA: C0621052 #1        SCANS    1 TO  700
06/21/90 20:45:00                         CALI: C06150F1 #3
SAMPLE: 50NG NITROMETHANE/2-NITROPROPANE STD: 1UL PURGE
CONDS.: 30DEG 4MIN/4DEG/MIN/FINAL 180 DEG
RANGE: G   1, 492  LABEL: N  0, 4.0  QUAN: A  0, 1.0 J  0  BASE: U 20,  3
```



```
LIBRARY SEARCH                                      DATA: C0621052 # 306      BASE M/Z:  61
06/21/90 20:45:00 +  5:06                           CALI: C06150F1 #   3      RIC:     68095.
SAMPLE: 50NG NITROMETHANE/2-NITROPRPPANE STD: 1UL PURGE
CONDS.: 30DEG 4MIN/4DEG/MIN/FINAL 180 DEG
 # 304 TO # 308 SUMMED - # 315 TO # 321 - # 283 TO # 294 X1.00
```



```
LIBRARY SEARCH                                         DATA: C0621052 # 438      BASE M/Z:  43
06/21/90 20:45:00 +  7:18                              CALI: C06150F1 #   3      RIC:    222453.
SAMPLE: 50NG NITROMETHANE/2-NITROPROPANE STD: 1UL PURGE
CONDS.: 30DEG 4MIN/4DEG/MIN/FINAL 180 DEG
 # 437 TO #.439 SUMMED - # 442 TO # 451 - # 426 TO # 435 X1.00
```



```
LIBRARY SEARCH                                      DATA: C0621052 # 437        BASE M/Z:  43
06/21/90 20:45:00 +  7:17                           CALI: C06150F1 #   3        RIC:    337407.
SAMPLE: 50NG NITROMETHANE/2-NITROPRPPANE STD: 1UL PURGE
CONDS.: 30DEG 4MIN/4DEG/MIN/FINAL 180 DEG
 # 435 TO # 439 SUMMED - # 443 TO # 449 - # 404 TO # 428 X1.00
```