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Analysis
(Type of Report)

REPORT NO.: MSL-5268
ESC-UAG-85-72

JOB/PROJECT NO.: 538

DATE: Dec. 18, 1985

TITLE: ANALYSIS OF DELAWARE RIVER PLANT WATER SAMPLES FOR PCBs AND OTHER SEMI VOLATILE COMPOUNDS

AUTHORS: B.M. Hughes, D. McKenzie and C. Trang

ABSTRACT: Four water samples were analyzed for PCBs and other semi-volatile compounds. Two samples contained measurable quantities of PCBs and other semi-volatile compounds.

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APPROVAL DATE: 12-18-85

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R-1088(A)

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REPORT NO.: MSL-528
ESC-UAG-85-72

JOB/PROJECT NO.: 538

DATE: Dec. 18, 1985

TITLE: ANALYSIS OF DELAWARE RIVER PLANT WATER SAMPLES FOR PCBs AND OTHER SEMI VOLATILE COMPOUNDS

AUTHORS: B.M. Hughes, D. McKenzie and C. Trang

ABSTRACT: Four water samples were analyzed for PCBs and other semi-volatile compounds. Two samples contained measurable quantities of PCBs and other semi-volatile compounds.

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APPROVAL DATE: 12-18-85

MONS 023754

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REPT. NO.: MSL-5268
ESC-UAG-85-72
COPY NO.:
AUTHORS: B.M. Hughes, D. McKenzie and C. Trang
TITLE: ANALYSIS OF DELAWARE RIVER PLANT WATER SAMPLES FOR PCBs AND OTHER SEMI VOLATILE COMPOUNDS

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R 1C88:C) (REV 1:80)

MONS 023755

Time Stamp - <851220.1036>

ANALYSIS REPORT

DATE: December 18, 1985

LOG NUMBER: U-85-12-04-01 PROJECT NO: 760.18-538

TO: Ed Jamro/Delaware River

FROM:

Environmental Sciences Center
Monsanto Company
800 N. Lindbergh Blvd.
St. Louis, Mo. 63167

Phone No: (314) 694-1464

Title: Analysis of Delaware River Plant Water Samples for PCBs and Other Semi-Volatile Compounds

ABSTRACT

Four water samples were analyzed for PCBs and other semi-volatile compounds. Two samples contained measurable quantities of PCBs and other semi-volatile compounds.

INTRODUCTION

Four water samples were analyzed for PCBs and other semi-volatile compounds using liquid/liquid extraction followed by capillary GC/EC and GC/MS extract analysis. Wide-scan extraction techniques were used in order to obtain data on PCB concentrations along with other major organic compounds which also may be present in these samples. The following sections give details of these analyses.

SUMMARY

PCB Analyses.

Tables 1 and 2 summarize analysis results for the PCB determinations. Note that two samples contained measurable quantities of Aroclor 1248.

Semi-Volatile Compound Analyses.

Table 3 summarizes the results of analysis for semi-volatile, extractable organic compounds in these samples. Total ion GC/MS chromatograms obtained from the analysis of the standard used for instrument calibration, and the three sample extracts shown in Table 3, are shown in Figures 1 - 4 in a later section of this report. All concentrations reported in Table 3 were calculated assuming 100% extraction efficiency for all compounds and assuming a response factor equal to the average response factors of 1,3-dichlorobenzene, 1,2-dichlorobenzene, naphthalene, hexachlorobutadiene, acenaphthene, fluorene, diethylphthalate, and hexachlorobenzene.

In addition to the total ion chromatograms, Figures 2 and 3 also include computer outputs from the automated identification of all components listed in Table 3, along with calculations of concentrations of these compounds. These quantitative data should only be used for order of magnitude estimates, since both the extraction efficiencies and response factors of these compounds are unknown at this time. Extraction efficiencies can be estimated from the surrogate recoveries listed in Tables 1 - 3 and the response factors can be estimated from the standard shown analyzed in Figure 1.

QC RESULTS

Limited quantities of sample prevented full QA/QC analyses for these samples. Table 2 summarizes the results of low and high native spikes of Burdick and Jackson Distilled in Glass water. Although sufficient sample was not available for native spiking studies of the submitted samples, recoveries of surrogate compounds were measured for all samples. The recovery results for these surrogate compounds are included in Tables 1 - 3.

DETAILS

Standard Sampling Method No(s): N.A.

Standard Extraction Method No(s): WSEX05

Standard Analysis Method No(s): ECAN02, MSAN02

Final Report File No(s): A53818

Table File No(s): B53818, C53818, D53818, E53818

Also included in this report are:

1) Brief descriptions of the above referenced extraction and analysis methods.

2) Raw data documentation, showing results of capillary GC/MS analyses and library searches.

3) Analytical Request Information generated for this project.

4) A copy of the ESC/UAG Good Laboratory Practices Manual.

ANALYST(S): B. Mason Hughes, David McKenzie, and Chi Trang

ANALYST APPROVAL: B. Mason Hughes

TABLE 1. Summary of PCB Concentrations in Water Samples.

ESC Project No: 760.18 - 53818 Date: December 12, 1985

Sample I.D.	Surrogate Recoveries(%Recovery)		PCB Concentration (ug/l))				
	CL5-benzene	2,4,6-CL3-biphenyl	A1232	A1242	A1248	A1254	Total
Monsanto 23S-A	113	100	ND(a)	ND(a)	ND(a)	ND(a)	ND(a)
Monsanto 3S	105	74	"	"	"	"	"
Monsanto 21S	35	34	"	"	2.5	"	2.5
Monsanto 4S	- (b)	- (b)	"	"	890.0	"	890.0
Method Blank	96	97	"	"	ND(a)	"	ND(a)

Footnotes:

a. ND= None Detected (<1.0 ug/l)
b. Recoveries could not be measured due to extract dilution.

TABLE FILE NO: B53818:MH

TABLE 2. Summary of Aroclor 1248 Spiking Concentrations and Recoveries in Quality Control Experiments for Water Samples.

ESC Project No: 760.18 - 53818 Date: December 12, 1985

Sample I.D.	Surrogate Recoveries (%Recovery)		Aroclor 1254	
	CL5-benzene	2,4,6-CL3-Biphenyl	Amount Added (ug/l)	Recovery (%)
B&J Water + L. Spike	100	70	.62	100
B&J Water + H. Spike	92	69	6.70	95

TABLE FILE NO: C53818:MH

MONS 023759

Table 3. Results of Wide-Scan Capillary GC/MS Analysis of Water Samples using Standard Analysis Method MSAN02.

Time Stamp - <851220.1034>

Project No: 760.18-538 Date: December 16, 1985

Analyte	RT (min)	LOQ(a) (ug/l)	Sample Identification: 23S-A Concentration (ug/l)	21S Concentration (ug/l)	Method Blank Concentration (ug/l)
Chloromethylbenzene isomer	4.41	10(E,S)	23 \	921	NQ(b)
" " "	4.56	"	5 /		"
Benzaldehyde	4.70	"	32	NQ	"
Chlorophenol-d4 (surrogate)	5.40	-	-	-	-
Dichlorobenzene-d4 (surrogate)	5.93	-	-	-	-
Unknown	6.61	10(E,S)	885	NQ	NQ(b)
Dichloromethylbenzene isomer	8.50,8.60	"	NQ	10	"
Unknown	8.84	"	3	NQ	"
Unknown	9.44	"	62	"	"
Tetrahydronephthelene	9.45	"	NQ	12	"
Unknown	10.27	"	"	2	"
N-(1,1-dimethylethyl)benzene-methenamine	10.35	"	1,885	NQ	"
Benzylisomeyl ether, or butoxymethyl-, propoxymethyl-benzene	11.41	"	14	196	"
Unknown Compounds	11.8 - 13.5	"	63	NQ	"
Chloromethylphenol isomer	12.15	"	NQ	2	"
Unknown	14.14	"	5	NQ	"
Unknown	15.37	"	1	"	"
Unknown	15.93	"	3	2	1
Anthracene-d10 (internal standard)	19.77	-	-	-	-
Unknown phthalate	22.22	10(E,S)	NQ	1	NQ
Unknown	22.88	"	"	2	"
Pyrene-d10 (surrogate)	23.83	-	-	-	-
Methoxydimethylbenzene butanal isomer	26.63	10(E,S)	NQ	10	NQ
Unknown	27.14	"	"	2	"
Unknown	28.15	"	"	3	"
Unknown	28.65	"	"	1	"
Unknown	29.13	"	"	8	"
Total (ug/l) =			2,981	1,172	1

Surrogate Spiking Compound	Amount Added(ug/l)	Recovery (%)		
Phenol-d5	10	11	40	< 5
1,2-CL2-benzene-d4	10	95	120	70

(continued)

MONS 023760

Table 3. (cont'd)

Documentation			
ESC Extraction Method	WSEX05	WSEX05	WSEX05
Date of Extraction	12/9/85	12/9/85	12/9/85
Sample Amount Extracted (L)	0.87	0.97	1.0
Extract Final Volume (ml)	1.0	1.0	1.0
Extract Dilution	none	none	none
ESC Extract or Sample Number	WS-1414	WS-1416	WS-1418
Date of CGC/MS Analysis	12/11/85	12/11/85	12/11/85
MS FRN	>L0778	>L0779	>L0780

Footnotes:

(a) LOQ - Limit of Quantitation in ug/l. Signal-to-noise ratio is greater than 10 when this amount is analyzed using the above referenced Analytical and Extraction Methods. The following letters indicate how this value was determined:
E - estimated from standard analyzed under the same conditions.
N - measured from native spiking of sample matrix.
S - estimated from surrogate spiking of sample matrix.
V - measured in validation experiments of similar sample matrix.

(b) NQ - Not Quantitateble. The analyte concentration is below the LOQ value.

Table stored in file: D53818

Analyst(s): B. Mason Hughes, David McKenzie, and Chi Trang

Analyst Approval: B. Mason Hughes Chi Trang

The following pages describe Standard Extraction Methods and Standard Analysis Methods used for this project.

MONS 023762

ESC Standard Extraction Method (SEM) TITLE: Wide scan extraction method for water.

ESC Standard Extraction Method No: WSEX05

MATRIX:	TYPICAL SAMPLE SIZE (Liters)	TYPICAL EXTRACT VOLUME (ml)
water	0.01 - 4	1 - 40

TYPICAL ANALYTES: Aroclors, PCBs, priority pollutants

ESC Standard Analysis Method Nos: ECANxx and MSANxx

INTERFERENCES: Various; See individual interferences for individual analytes.

TYPICAL SURROGATES	TYPICAL RECOVERIES (%)
phenol-d5	20-80
CL-phenol-d4	20-80
CL2-benzene-d4	50-120
naphthalene-d8	50-120
pyrene-d10	50-120
chrysene-d12	50-120
pentachlorobenzene	50-120
2,4,6-trichloro-biphenyl	50-120

APPARATUS AND MATERIALS:
Burdick and Jackson methylene chloride
Separatory funnels
Sodium sulfate for extract drying
Sodium hydroxide and sulfuric acid for pH adjustments
Nitrogen for extract volume reduction
Water bath and K-D apparatus for extract volume reduction

LIMITATIONS: Sufficient sample amount to provide GC-MS LOD.

SAFETY PRECAUTIONS: See Safe Laboratory Practices.

DATES OF METHOD CREATION AND UPDATES: 5/16/84, 10/5/84, 12/7/84

FLOW SHEET WSF805
FOR ESC EXTRACTION METHOD WSEX05

SAMPLE ID: ---------- MRC EXTRACT #----------

---------- LOG NO: ----------

EXTRACTION DATE and TIME: ----------/----------

RECORD NATIVE pH ---------- WAS pH ADJSTED YES/NO IF YES TO WHAT pH----------

PLACE SAMPLE INTO AN APPROPRIATELY SIZED SEPERATORY FUNNEL (1/2 HEAD SPACE SUGGESTED FOR ADEQUATE MIXING) [ ]

SAMPLE VOLUME ----------

SURROGATE SPIKE

IF pH ADJUSTED ( ) ADJUST WITH,50% NaOH OR H_2SO_4

ADD METHYLENE CHLORIDE IN THE APPROPRIATE AMOUNT (1 L SAMPLE 30 mL SOLVENT, 100 mL SAMPLE 15 mL SOLVENT) [ ]

RECORD AMOUNT METHYLENE CHLORIDE ADDED FOR EACH EXTRACTION

1)----------

2)----------

3)----------

SHAKE SAMPLE ONE TIME AND VENT THROUGH STOPCOCK REPEAT DOUBLING THE NUMBER OF SHAKES BETWEEN VENTING SAMPLES UNTIL THE NUMBER OF SHAKES EXCEED 80 [ ]

OBSERVATIONS

ALLOW PHASES TO SEPARATE [ ]

REPEAT EXTRACTION PROCEDURE TWO MORE TIMES [ ] [ ]

IF ACID OR BASE WASHED RECORD TYPE AND AMOUNT. [ ]

ACID/BASE WASH COMENTS

DRY EXTRACT WITH 4 INCH SODIUM SULFATE COLUMN (COLUMN PRERINSED WITH 30 mL METHYLENE CHLORIDE) [ ]

[ ] EXTRACT DRYING NOT REQUIRED

RINSE SODIUM SULFATE COLUMN WITH 2 X 30 mL [ ]

REDUCE EXTRACT VOLUME TO 1 mL BY K-D TECHNIQUE AND OR NITROGEN SOLVENT VOLUME REDUCTION [ ]

[ ] TRANSFER EXTRACT INTO VIAL FOR ANALYSIS

EXTRACT NO: ---------- DATE/TIME COMPLETED----------/----------

ANALYST: ---------- ESC JOB NO: ----------

MONS 023764

ESC Standard Analysis Method (SAM) — ESC Standard Analysis Method No:
TITLE: Aroclor Formulations in Solvents — ECAM02

ANALYTICAL INSTRUMENT AND MODE OF OPERATION: HP-5880 Capillary GC/EC interfaced to an HP-3357-LAS Laboratory Data System

---

TYPICAL ANALYTE	TYPICAL INSTRUMENT LEVEL OF DETECTION (LOD) (ug/ml)	TYPICAL PRECISION @ 10X LOD (%)	TYPICAL ACCURACY @ 10X LOD (%)
Aroclor formulations 1232, 1242, 1248, 1254, 1260, and 1262	0.1	30	30

INTERFERENCES AND LIMITATIONS: Any electron capturing species in the retention time region of trichlorobiphenyl through octachlorobiphenyl isomers are potential interferences. Exact retention times of most likely PCB isomers can be obtained from the analysis of Aroclor 1248, Aroclor 1254 or Aroclor 1260 formulations.

CHROMATOGRAPHIC COLUMN: 30-meter fused silica J&W DB-5 (0.25 uM) with wide bore (0.32mm).

COLUMN TEMPERATURE PROGRAM: 130(1)/4/300 — INJECTION TYPE: Splitless

---

CALIBRATION AND STANDARDIZATION: Use internal standard quantitation technique and calibrate instrument response factors from authentic Aroclor formulation standards. Use a minimum of 10 PCB isomers for the determination of formulation identity and quantity.

CALCULATIONS: Formulation concentration =
(isomer RRF)(isomer area/internal standard area)

where: RRF is the relative response factor for the appropriate PCB isomer in a given formulation. Formulation concentration is obtained from the average of at least 10 such calculations.

SAFETY PRECAUTIONS: See PCB handling protocols in laboratory safety manual.

DATE OF METHOD CREATION: 10/25/83, 2/3/84 Time Stamp - <851218.0906>

ESC Standard Analysis Method (SAM) **ESC Standard Analysis Method No:**
TITLE: Organic Compounds in Solvents **MSAM02**

ANALYTICAL INSTRUMENT AND MODE OF OPERATION: HP-5985/5987 Capillary GC/MS-Scanning with RTE-VI operating system.

Typical Run Time:	3 - 40 min.
Typical Multiplier Voltage:	1800 - 2400 eV
Typical Threshold:	100 counts
Typical Mass Range:	10 - 500 amu
Typical Scan Rate:	2/second

TYPICAL ANALYTE	TYPICAL INSTRUMENT LEVEL OF DETECTION (LOD) (ug/ml)	TYPICAL PRECISION @ 10X LOD (%)	TYPICAL ACCURACY @ 10X LOD (%)
Organic Compounds eluting between toluene and n-C34-ane	1	30	30

INTERFERENCES AND LIMITATIONS: Analyte must be chromatographically resolved from other organic compounds.

CHROMATOGRAPHIC COLUMN: 30-meter fused silica J&W DB-5 (0.25 uM) with wide bore (0.32 mm)

COLUMN TEMPERATURE PROGRAM: 50(4)/8/300 **INJECTION TYPE:** Splitless

---

CALIBRATION AND STANDARDIZATION:

Quantitative: Use either internal or external standard quantitation technique and calibrate instrument response factors either from the internal standard response or from other standard compounds analyzed under identical conditions.

Qualitative: Mass spectrum of unknown compounds are compared to standard mass spectra in the NBA/EPA Mass Spectral Library which contains over 30,000 compounds. Positive identification is made only after a trained mass spectroscopist has visually compared the unknown compound mass spectrum with those identified as most probable compounds based upon their Match Factor.

CALCULATIONS: Analyte concentration =

[internal standard concentration][(analyte area)/(internal standard area)]

- or -

[standard compound concentration][(analyte area)/(standard compound area)]

where: Areas are the total ion areas of the analyte, the internal standard or standard compound.

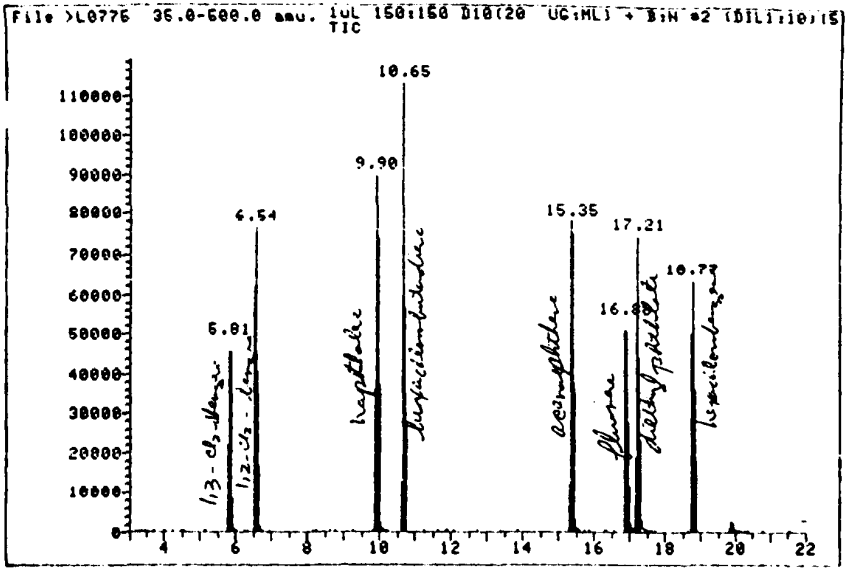
note: This calculation is made when unknown compounds are present or when no authentic standards are available.

SAFETY PRECAUTIONS: See PCB handling protocols in laboratory safety manual.

DATE OF METHOD CREATION: 10/31/83 Time Stamp - <851217.0836>

The following pages show calculations or raw data documentation of raported results.

Figure 1. Total ion chromatogram obtained from the analysis of a B/N standard containing 1,3-CL2-benzene, 1,2-CL2-benzene, naphthalene, hexachlorobutadiene, acenaphthene, fluorene, diethylphthalate, and hexachlorobenzene, each at approximately 30 ug/ml.



S

reg	type	# pts	scan#	range: amu\r.t.	base	file	ion range
X	GC	10583	1	3.17- 50.02	112721.0	>L0775	35.00- 500.00

>L0775 1uL 150:150 D10(20 UG:ML) + B:N #2 (DIL1:10)(50) 12:11:85

35.01 500.0 TIC

Peak #	R.T. min.	first scan	max scan	last scan	peak height	(corr. ion)	raw area	corr. area	corr. % max.	% of total
1	5.811	584	598	613	45238	49.1	130847	130847	59.26	9.339
2	6.537	748	762	781	76194	49.0	194462	194462	88.07	13.879
3	9.897	1507	1521	1533	89487	50.0	187509	187509	84.92	13.383
4	10.645	1678	1690	1706	112721	48.7	220800	220800	100.00	15.759
5	15.348	2741	2753	2772	77928	49.0	208133	208133	94.26	14.854
6	16.884	3089	3100	3123	50771	50.7	139666	139666	63.25	9.968
7	17.212	3162	3174	3198	74403	51.4	178552	178552	80.87	12.743
8	18.770	3515	3526	3542	63303	50.7	141179	141179	63.94	10.076

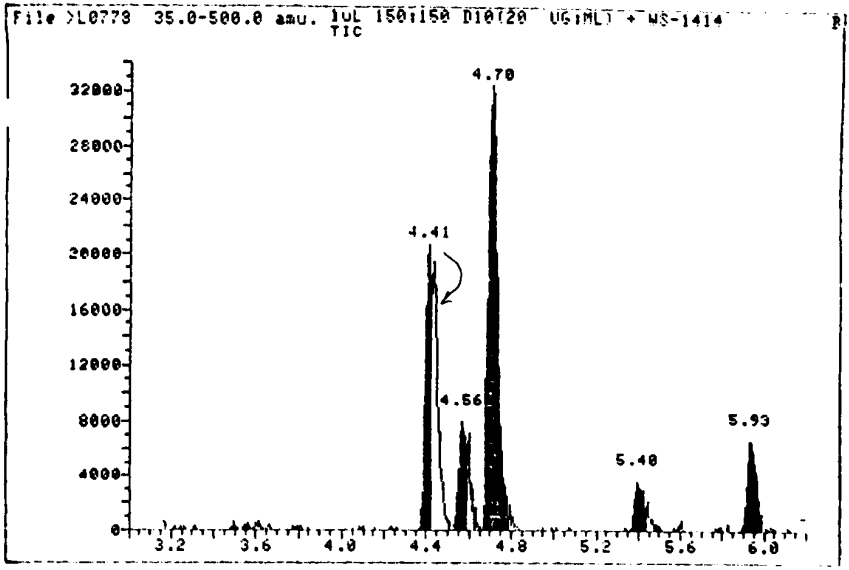
399.1 µg/ml 1,401,148

RF = AREA / CONC = 3,511 cts/µg/ml

MONS 023769

Figure 2. Total ion chromatograms, mass spectra, and library search outputs from the analysis of the WS-1414 extract of 238-A.

MONS 023770



RT= 4.41 Chloromethyl benzene isomer

~ 70,000/(3511)0.87 = 23

Constant = (3511)(0.87)

RT= 4.56 Chloromethyl benzene isomer

16,026/constant = 5.25

RT= 4.70 Benzaldehyde

98,647/constant =

RT= 5.40 Cl phenol-dy - Surrogate

RT= 5.93 Cl_2-benzene-dy - Surrogate

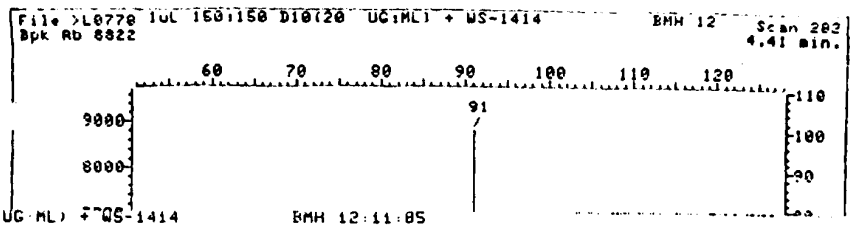
)L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

35.01 500.0 TIC

Peak #	R.T. min.	first scan	max scan	last scan	peak height	raw area	corr. area	corr. % max.	% of total
1	4.408	271	282	285	20730	34419	34419	34.89	19.504
2	4.563	308	317	323	7933	16026	16026	16.25	9.081
3	4.705	340	349	367	32382	98647	98647	100.00	55.900
4	5.396	496	505	514	3580	9589	9589	9.72	5.434
5	5.931	616	626	640	6688	17789	17789	18.03	10.080

Sum of corrected areas: 176470.

MONS 023772



282

File: >L0778 Scan #: 282 Retn. time: 4.41

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
50.95	394.00	73.80	284.00	90.05	731.00	124.95	1167.00	126.95	675.00
63.05	906.00	74.95	310.00	91.05	8822.00	125.95	3595.00	127.95	998.00
64.95	891.00	88.95	1957.00						

Complete spectrum not available; see following page.

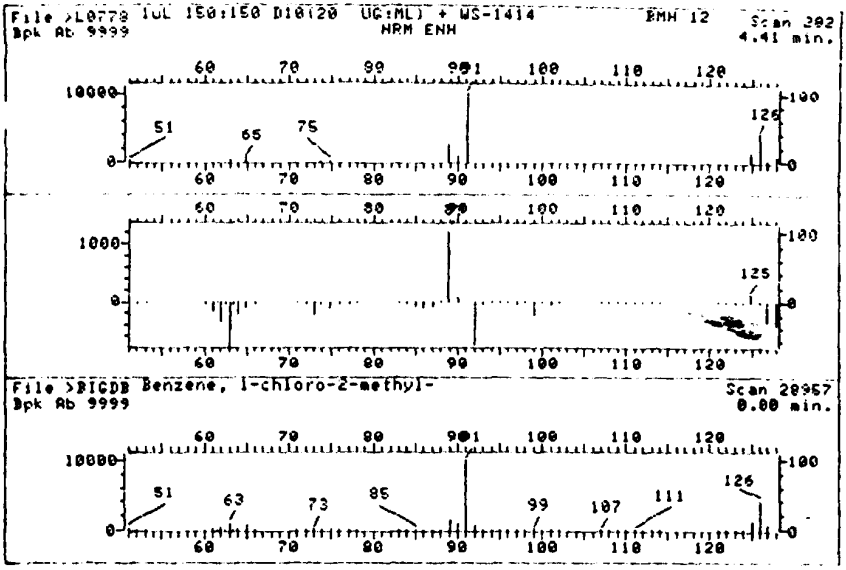
S

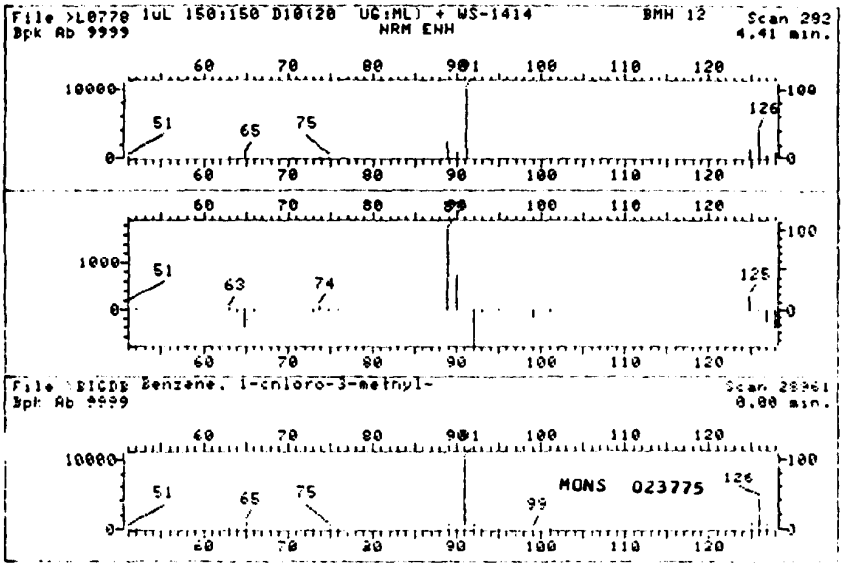
1.	Benzene, 1-chloro-2-methyl-	126 C7H7Cl
2.	Benzene, 1-chloro-3-methyl-	126 C7H7Cl
3.	Benzene, 1-chloro-4-methyl-	126 C7H7Cl
4.	Benzene, 1-chloro-4-methyl-	126 C7H7Cl
5.	Benzene, 1-chloro-2-methyl-	126 C7H7Cl
6.	Benzene, 1-chloro-3-methyl-	126 C7H7Cl

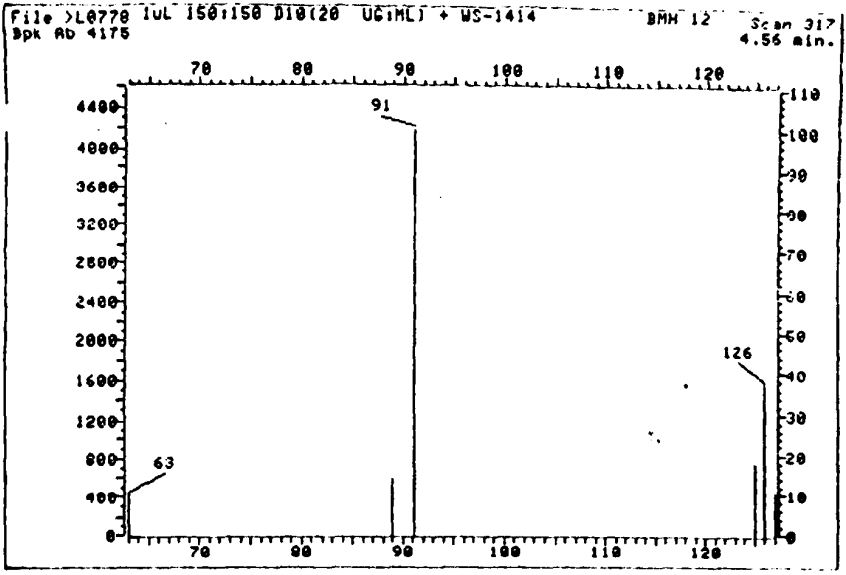
Sample file:)L0778 Spectrum #: 282
Search speed: 2 Tilting option: N No. of ion ranges searched: 61

	Prob.	CAS #	CON #	ROOT	K	DK	#FLG	TILT	%	CON	C_I	R_IV
1.	79*	95498	28957	"BIGDB	55	42	2	0	85	7	48	32
2.	67*	108418	28961	"BIGDB	42	44	2	0	86	15	34	22
3.	67*	106434	28963	"BIGDB	48	43	2	0	75	11	34	28
4.	67*	106434	28962	"BIGDB	43	42	2	0	100	14	34	23
5.	60*	95498	28959	"BIGDB	38	48	2	0	100	14	30	19
6.	58*	108418	28960	"BIGDB	43	49	2	0	72	16	25	23

MONS 023774


File >L0778 1uL 150:150 D10(20 UG:ML) + WS-1414
Bpk Ab 9999
NRM ENH
BMH 12
Scan 292
4.41 min.
File >BIGDB Benzene, 1-chloro-2-methyl-
Bpk Ab 9999
Scan 28957
0.00 min.


File >L0778 1uL 150:150 D10(20 UG:ML) + WS-1414
Bpk Ab 9999
NRM ENH
BMH 12
Scan 292
4.41 min.
File >BIGDB Benzene, 1-chloro-3-methyl-
Bpk Ab 9999
Scan 28961
0.00 min.
MONS 023775



>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

317

File: >L0778 Scan #: 317 Retn. time: 4.56

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
62.95	416.00	91.05	4175.00	124.95	750.00	125.95	1559.00	126.95	449.00
88.95	584.00								

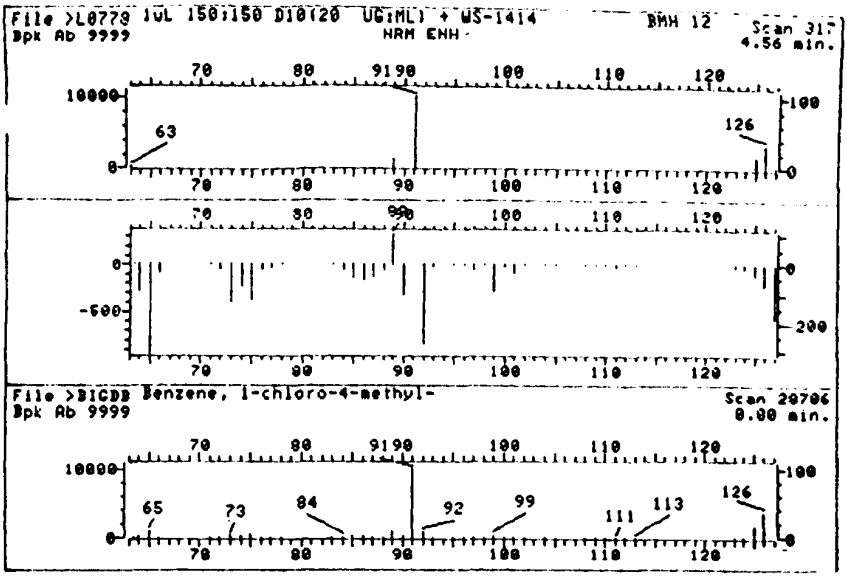
S

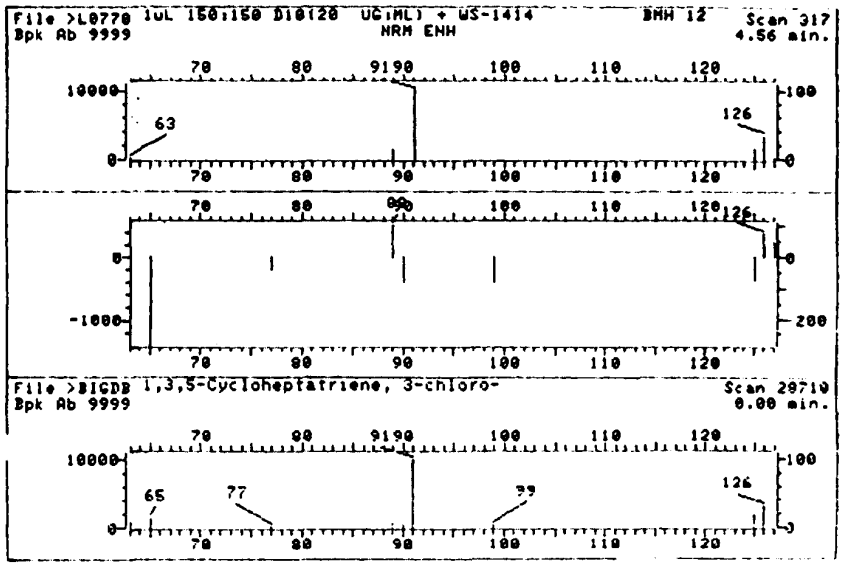
1.	Benzene, 1-chloro-4-methyl-	126 C7H7Cl
2.	1,3,5-Cycloheptatriene, 3-chloro-	126 C7H7Cl
3.	1,3,5-Cycloheptatriene, 2-chloro-	126 C7H7Cl
4.	Benzene, 1-chloro-4-methyl-	126 C7H7Cl

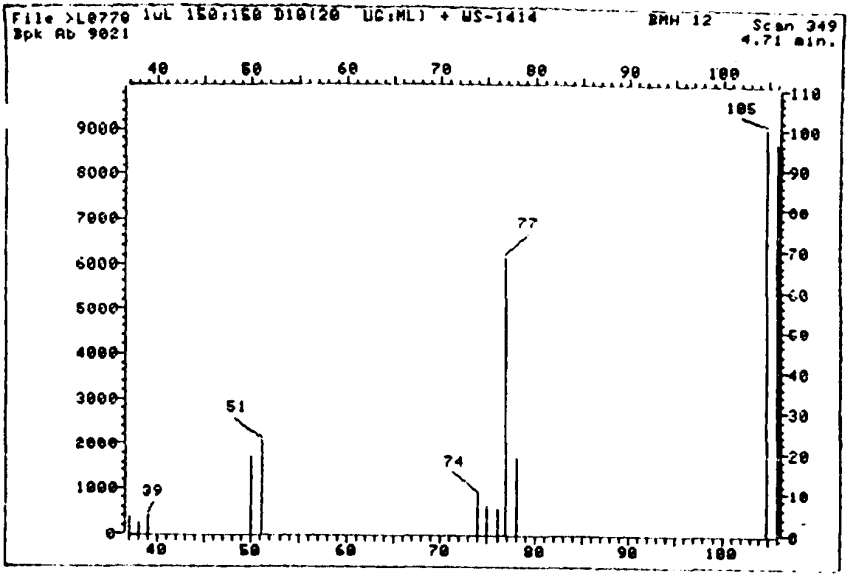
Sample file: >L0778 Spectrum #: 317
Search speed: 2 Tilting option: N No. of ion ranges searched: 69

	Prob.	CAS #	CON #	ROOT	K	DK	#FLG	TILT	%	CON	C_I	R_IV
1.	→70*	106434	28706	"BIGDB	36	64	3	0	100	6	42	13
2.	60*	55619051	28710	"BIGDB	26	40	3	0	100	13	30	13
3.	60*	34896792	28709	"BIGDB	25	40	3	0	100	13	30	13
4.	60*	106434	28962	"BIGDB	26	59	3	0	100	11	30	13

MONS 023777


File >L0779 1UL 150:150 D10(20 UG(ML) + WS-1414
Bpk Ab 9999
NRM ENH
BMH 12
Scan 317
4.56 min.
10000
63
126
-500
File >BIGDB Benzene, 1-chloro-4-methyl-
Bpk Ab 9999
Scan 29706
0.00 min.
65
73
84
92
99
111
113
126


File >L0779 1UL 150:150 D10(20 UG(ML) + WS-1414
Bpk Ab 9999
NRM ENH
BMH 12
Scan 317
4.56 min.
10000
63
126
-1000
File >BIGDB 1,3,5-Cycloheptatriene, 3-chloro-
Bpk Ab 9999
Scan 29710
0.00 min.
65
77
99
126



>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

349

File: >L0778 Scan #: 349 Retn. time: 4.71

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
36.95	378.00	49.95	1713.00	74.95	612.00	76.95	6139.00	104.95	9021.00
37.95	256.00	51.05	2055.00	76.05	552.00	78.05	1696.00	105.95	8696.00
38.95	406.00	73.95	858.00						

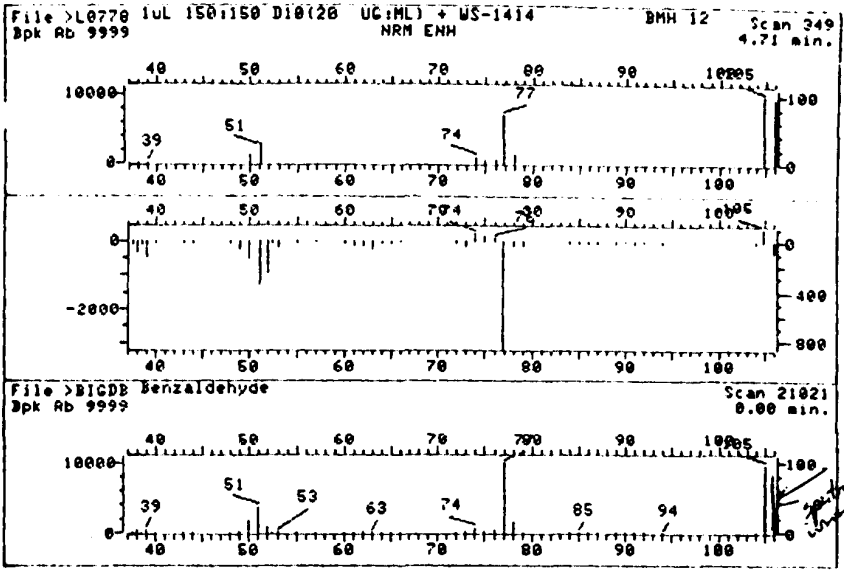
S

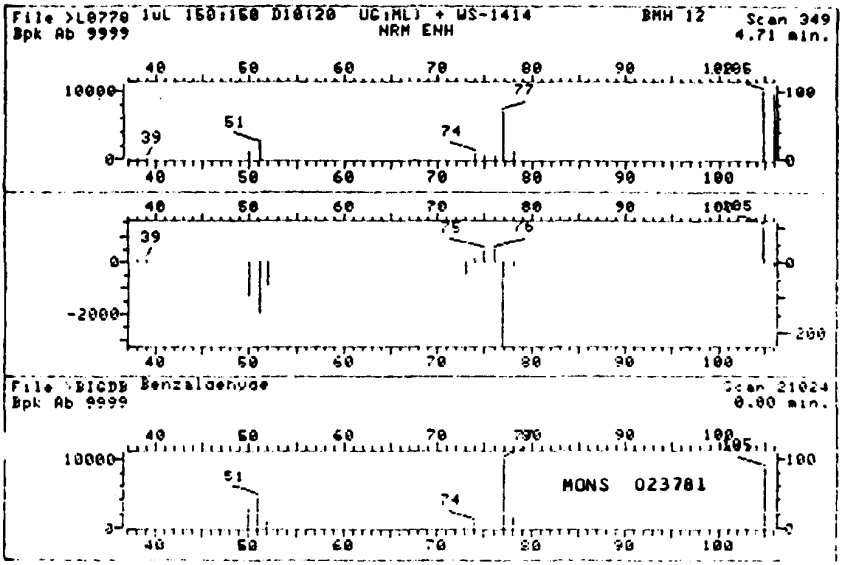
1. Benzaldehyde 106 C7H6O

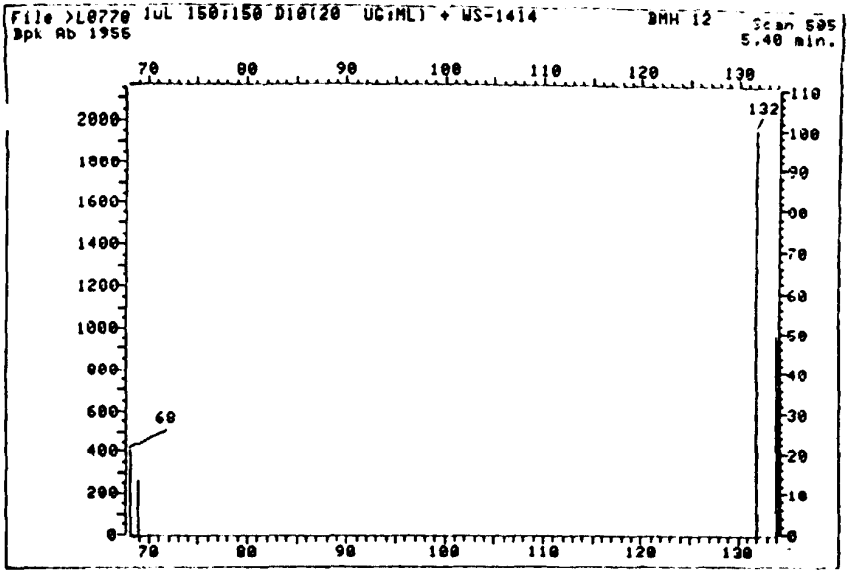
Sample file: >L0778 Spectrum #: 349
Search speed: 2 Tilting option: N No. of ion ranges searched: 47

	Prob.	CAS #	CON #	ROOT	K	DK	#FLG	TILT	%	CON	C_I	R_IV
1.	=> 52*	100527	21021	"BIGDB	23	75	3	0	68	18	20	12

MONS 023780


File >L0770 1uL 150:150 D10(20 UC:ML) + WS-1414 BMH 12 Scan 349
Bpk Ab 9999 NRM ENH 4.71 min.
39
51
74
77
File >BIGDB Benzaldehyde Scan 21021
Bpk Ab 9999 0.00 min.
39
51
53
63
74
85
94


File >L0770 1uL 150:150 D10(20 UC:ML) + WS-1414 BMH 12 Scan 349
Bpk Ab 9999 NRM ENH 4.71 min.
39
51
74
77
File >BIGDB Benzaldehyde Scan 21024
Bpk Ab 9999 0.00 min.
51
74
MONS 023781

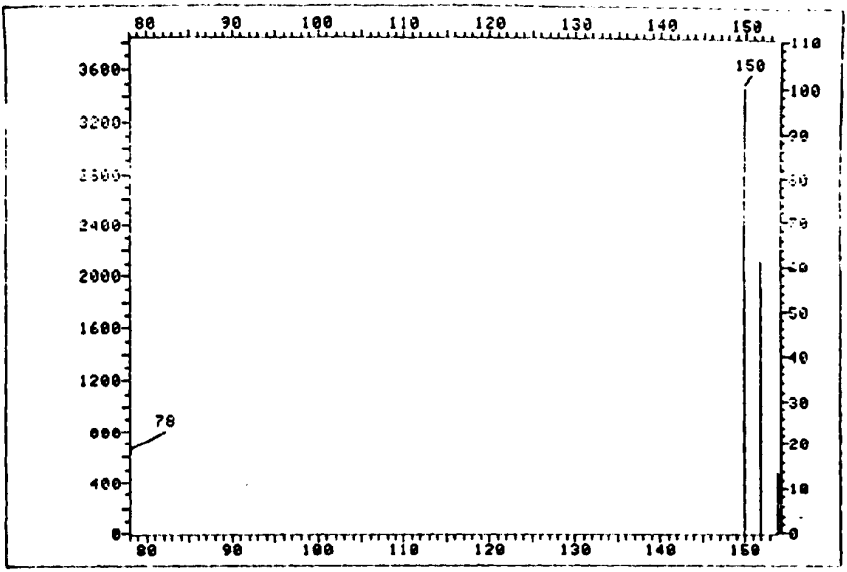


>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

505

File: >L0778 Scan #: 505 Retn. time: 5.40

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
68.85	400.00	68.95	264.00	131.95	1955.00	133.95	961.00		

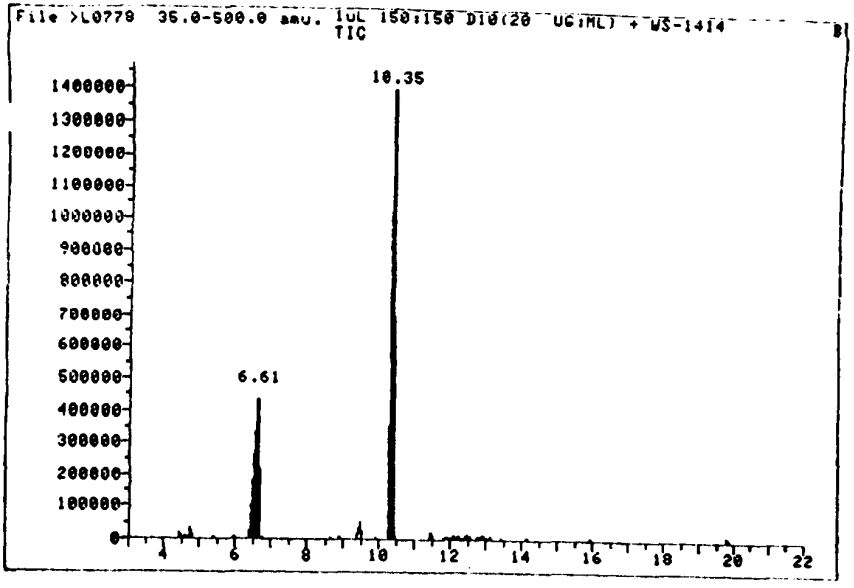


>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

626

File: >L0778 Scan #: 626 Retn. time: 5.93

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
77.95	621.00	150.00	3474.00	152.00	2116.00	153.90	477.00		



Peak at 6.61 minutes - unknown - $\frac{3,014,792}{3,511}$ µg/ml = 859 µg/ml

1 ml = 0.87 liters 885 µg/Liter

Peak at 10.35 minutes - N-(1,1-dimethylethyl) benzenemethane amine

$\frac{5,625,921}{3511}$ = 1,602 µg/ml

= 1,885 µg/Liter

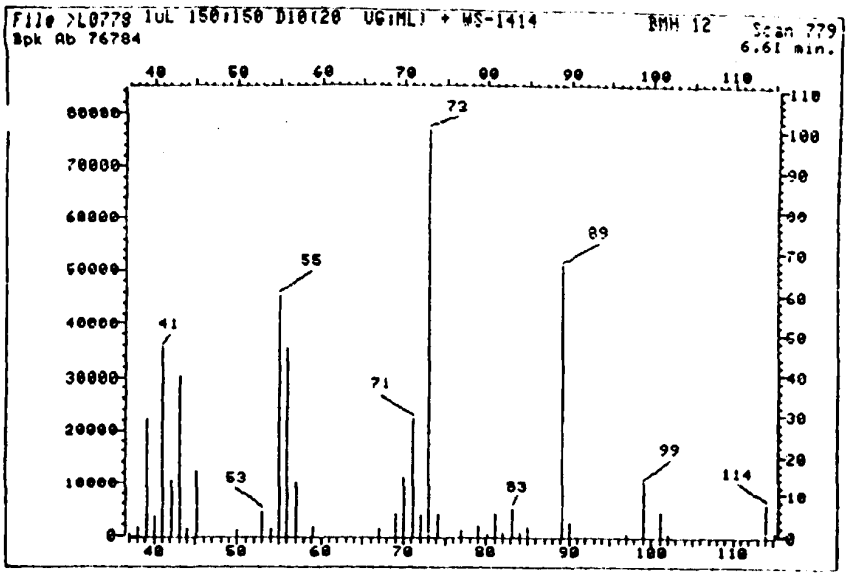
MONS 023784

>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

35.01 500.0 TIC

Peak #	R.T. min.	first scan	max scan	last scan	peak height	raw area	corr. area	corr. % max.	% of total
1	6.610	721	779	786	431046	3014792	3014792	53.59	34.891
2	10.352	1593	1624	1648	1398019	5625921	5625921	100.00	65.109

Sum of corrected areas: 8640714.



>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

779

File: >L0778 Scan #: 779 Retn. time: 6.61

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
36.95	365.0	45.05	12428.0	67.05	1313.0	79.05	1912.0	90.05	2400.0
38.05	1435.0	50.05	906.0	69.05	4060.0	80.20	350.0	96.95	330.0
39.05	22296.0	53.05	4535.0	70.05	11283.0	81.05	4208.0	99.05	10205.0
40.05	3637.0	54.05	1247.0	71.05	22424.0	81.95	560.0	101.05	4499.0
41.05	35648.0	55.05	45544.0	72.05	3722.0	83.05	5105.0	102.05	287.0
42.05	10625.0	56.05	35384.0	73.05	76784.0	84.05	510.0	114.05	5714.0
43.05	30288.0	57.05	10099.0	74.05	4037.0	85.05	1364.0	115.05	622.0
44.05	1198.0	59.05	1534.0	76.95	900.0	89.05	51256.0		

MONS 023786

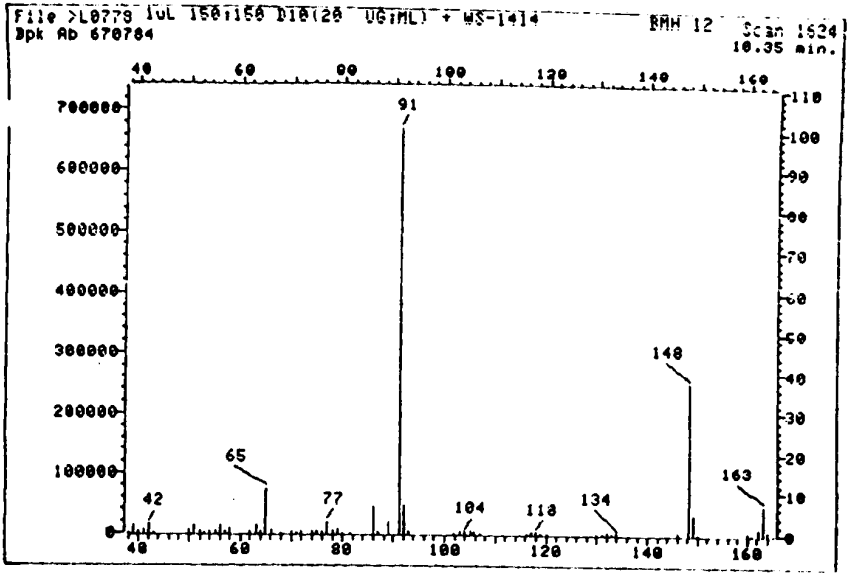
s

1. 1-Propanamine, N-propyl- 101 C6H15N

Sample file: >L0778 Spectrum #: 779
Search speed: 2 Tilting option: N No. of ion ranges searched: 47

	Prob.	CAS #	CON #	ROOT	K	DK	#FLG	TILT	%	CON	C_I	R_IV
MC→ 1.	28*	142847	8571	"BIGDB	49	43	2	0	72	53	8	30

MONS 023787



>L0778 1uL 150:150 D10(20 UG/ML) + WS-1414 BMH 12:11:85

1624

File: >L0778 Scan #: 1624 Retn. time: 10.35

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
37.9S	1298.	57.0S	2155.	75.9S	2434.	103.0S	2266.	133.0S	670.
39.0S	12266.	58.0S	7765.	76.9S	18800.	104.0S	4793.	133.9S	2781.
40.0S	1896.	61.9S	2893.	77.9S	5016.	105.0S	4196.	146.1S	3242.
41.0S	6504.	63.0S	13256.	79.0S	8262.	105.9S	2503.	148.1S	251072.
42.0S	15077.	64.0S	3848.	79.9S	553.	106.9S	540.	149.1S	32192.
43.0S	1355.	64.9S	72072.	81.5S	602.	116.2U	555.	150.1S	1292.
50.0S	5339.	66.0S	4363.	86.0S	43272.	116.9S	3245.	160.1S	1686.
51.0S	13870.	68.0S	481.	87.0S	2810.	118.0S	3500.	161.1S	814.
52.0S	3472.	70.0S	3124.	88.9S	18496.	119.0S	639.	162.1S	10023.
52.9S	1246.	70.9S	645.	91.0S	670784.	120.0S	684.	163.1S	47616.
54.0S	2547.	72.0S	2662.	92.0S	45072.	129.9S	1117.	164.1S	4690.
55.0S	1626.	73.9S	2649.	92.9S	1776.	131.0S	523.	165.1S	418.
56.0S	12368.	75.0S	1760.	101.9S	619.	132.0S	1869.		

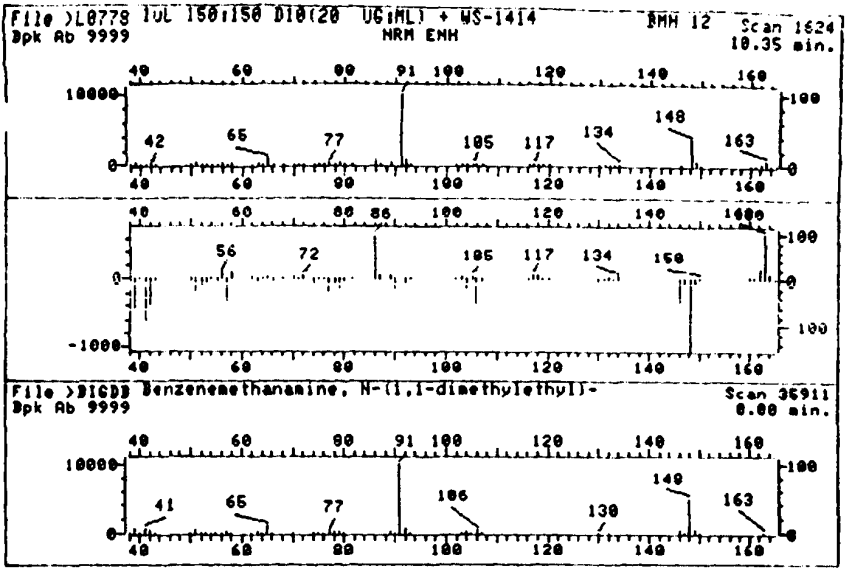
6

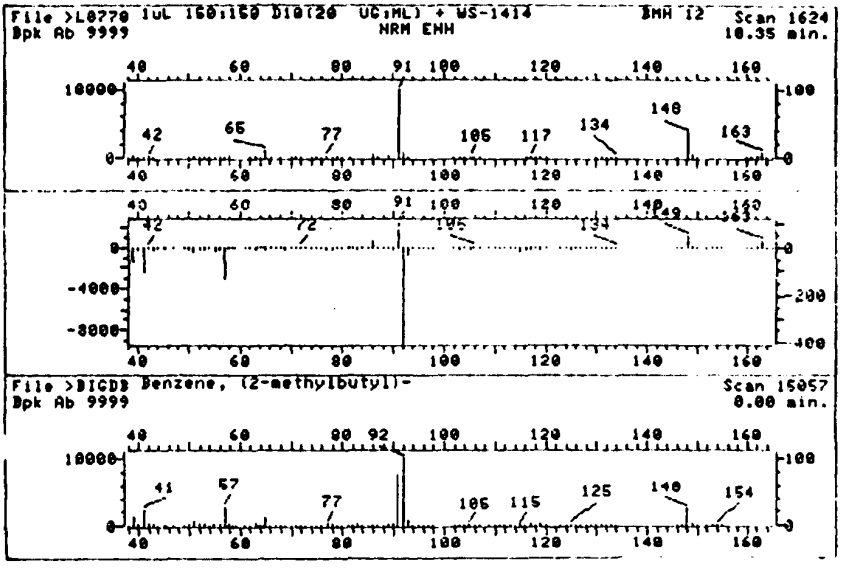
1. Benzenemethanamine, N-(1,1-dimethylethyl)- 163 C11H17N
2. Benzene, (2-methylbutyl)- 148 C11H16

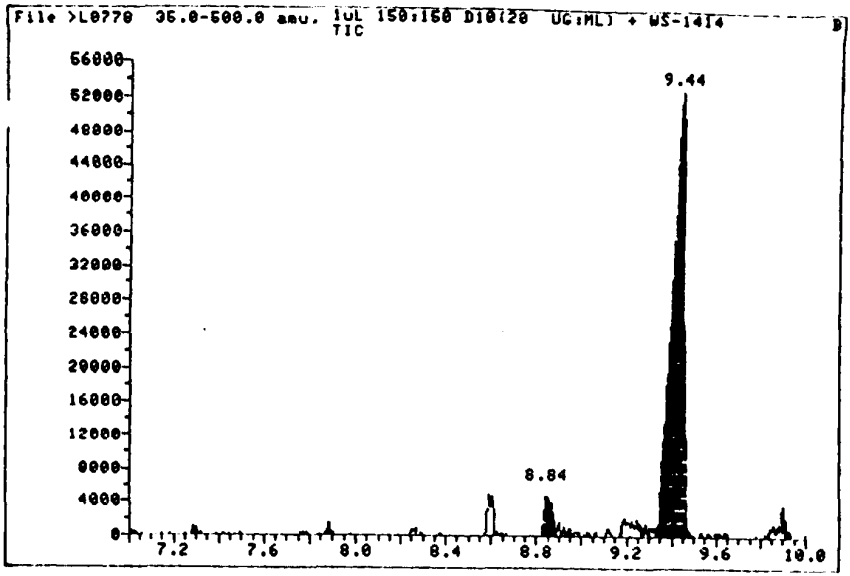
Sample file: >L0778 Spectrum #: 1624
Search speed: 2 Tilting option: N No. of ion ranges searched: 48

	Prob.	CAS #	CON #	ROOT	K	DK	#FLG	TILT	%	CON	C_I	R_IV
YES→1.	70*	3378721	35911	*BIGDB	38	46	2	0	78	9	42	19
NO→2.	60*	3968852	15057	*BIGDB	24	65	3	0	129	14	30	12

MONS 023789


File >L0778 1uL 150:150 D10(20 UG/ML) + WS-1414
Bpk Ab 9999
NRM ENH
EMH 12
Scan 1624
10.35 min.
File >DIGDB Benzenemethanamine, N-(1,1-dimethylethyl)-
Bpk Ab 9999
Scan 36911
0.00 min.


File >L0778 1uL 150:150 D10(20 UG/ML) + WS-1414
Bpk Ab 9999
NRM ENH
EMH 12
Scan 1624
10.35 min.
File >DIGDB Benzene, (2-methylbutyl)-
Bpk Ab 9999
Scan 15057
0.00 min.



RT = 8.84 unknown

10,528 / count = 3.45

RT = 9.44 unknown

190,785 / count = 62.5

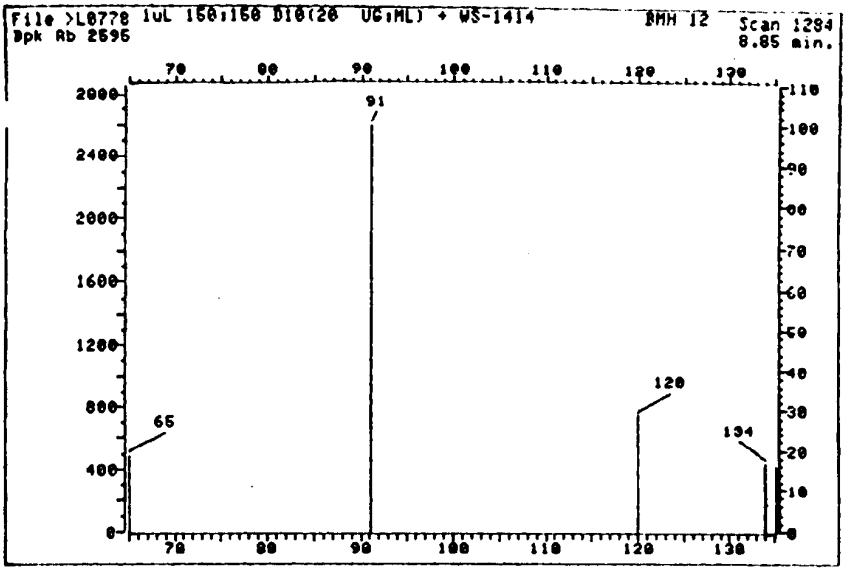
>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

35.01 500.0 TIC

Peak #	R.T. min.	first scan	max scan	last scan	peak height	raw area	corr. area	corr. % max.	% of total
1	8.845	1278	1284	1293	4689	10528	10528	5.52	5.230
2	9.444	1392	1419	1431	53192	190785	190785	100.00	94.770

Sum of corrected areas: 201313.

MONS 023792

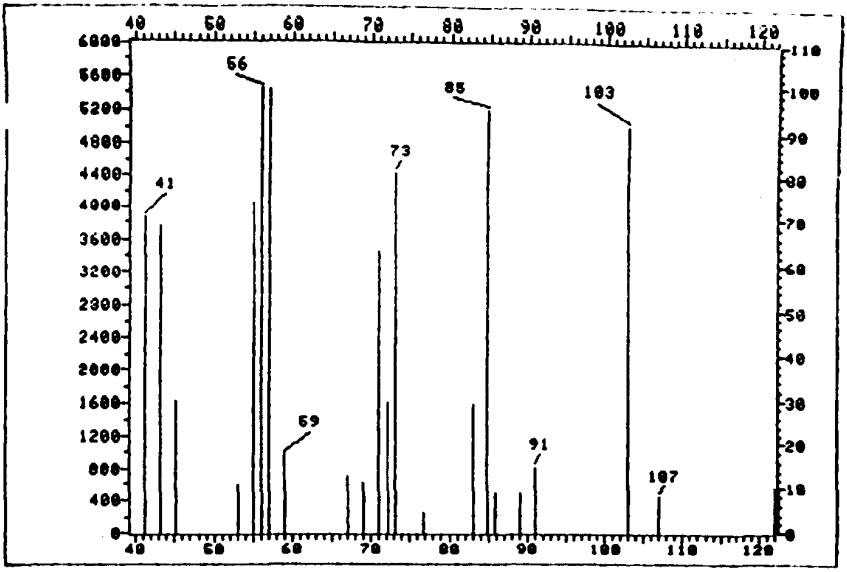


>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

1284

File: >L0778 Scan #: 1284 Retn. time: 8.85

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
64.95	490.00	91.05	2595.00	120.05	749.00	133.95	438.00	135.05	417.00

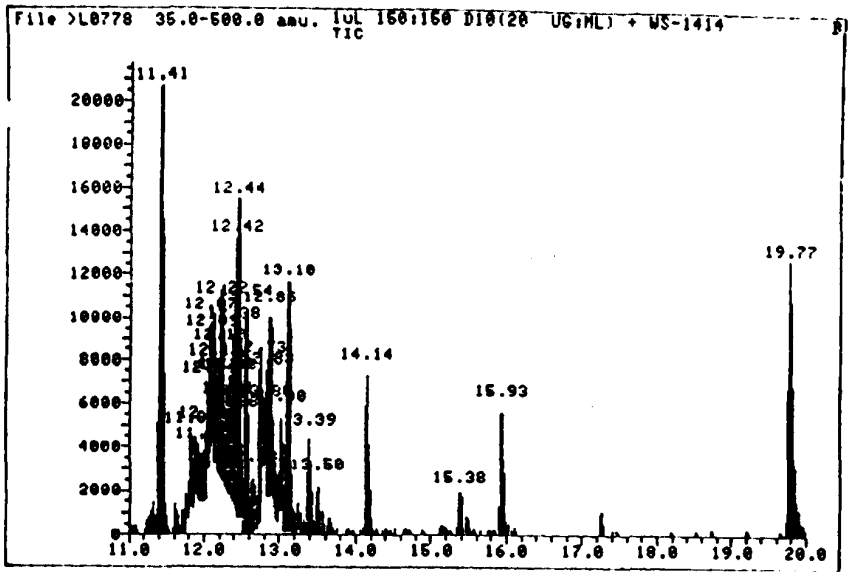


>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

1419

File: >L0778 Scan #: 1419 Retn. time: 9.44

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
39.05	1986.00	55.05	4046.00	68.95	604.00	83.05	1582.00	98.95	823.00
41.05	3876.00	56.05	5450.00	71.05	3438.00	84.95	5168.00	103.05	4981.00
43.05	3736.00	57.05	5431.00	72.05	1606.00	85.95	475.00	106.95	444.00
45.05	1633.00	59.05	959.00	73.05	4407.00	88.95	476.00	121.95	541.00
53.05	586.00	67.05	692.00	76.80	252.00				



RT = 11.41 unknown 41,302/control = 13.52

RT = 11.8 - 13.58 unknown complex
total area 191,025/control = 62.35

RT = 14.14 unknown
13,892/control = 4.58

RT = 15.37 unknown
3,088/control = 1.01

RT = 15.93 unknown 8,132/control = 2.68

RT = 19.77 acetaminophen-d_{10} internal std.
32,914

MONS 023795

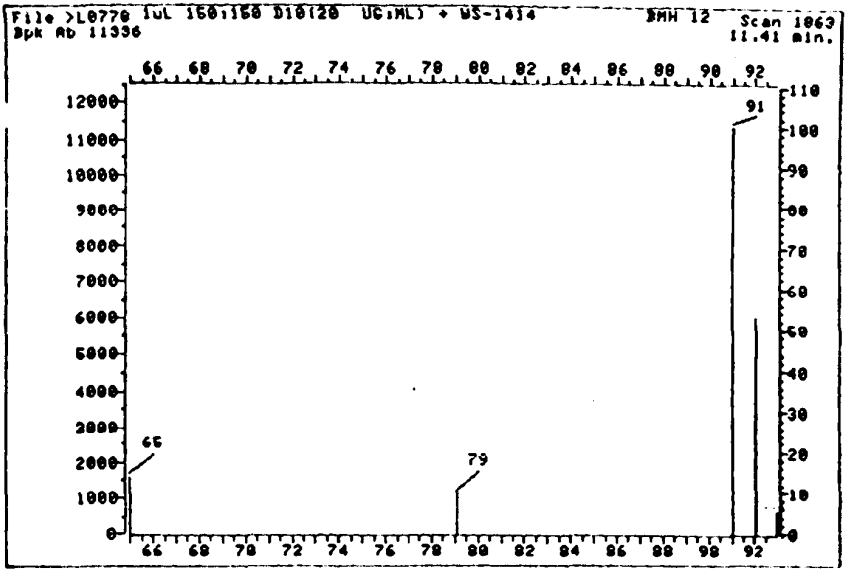
)L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

35.01 500.0 TIC

Peak #	R.T. min.	first scan	max scan	last scan	peak height	raw area	corr. area	corr. % max.	% of total
1	11.414	1854	1863	1877	20670	41302	41302	100.00	14.220
2	11.817	1951	1954	1956	3014	3824	2138	5.18	.736
3	11.968	1984	1988	1994	2110	6701	2658	6.44	.915
4	12.016	1994	1999	2001	2893	6678	3665	8.87	1.262
5	12.039	2001	2004	2007	3628	7828	3067	7.43	1.056
6	12.070	2007	2011	2014	5522	13493	5995	14.51	2.064
7	12.092	2014	2016	2017	3359	6548	2233	5.41	.769
8	12.136	2024	2026	2032	4091	12582	5617	13.60	1.934
9	12.185	2032	2037	2039	5208	10626	5192	12.57	1.788
10	12.220	2040	2045	2047	8238	15359	10382	25.14	3.574
11	12.247	2048	2051	2052	4704	6238	3607	8.73	1.242
12	12.282	2057	2059	2063	4590	6853	3399	8.23	1.170
13	12.309	2063	2065	2066	3675	4111	2494	6.04	.859
14	12.327	2066	2069	2071	4895	7566	5035	12.19	1.733
15	12.358	2074	2076	2077	3340	4135	2788	6.75	.960
16	12.380	2077	2081	2083	7569	11236	8763	21.22	3.017
17	12.420	2083	2090	2093	12360	28559	25092	60.75	8.639
18	12.438	2093	2094	2103	14588	17548	15307	37.06	5.270
19	12.544	2111	2118	2130	10164	15639	15639	37.86	5.384
20	12.633	2134	2138	2141	2490	2896	2896	7.01	.997
21	12.726	2155	2159	2162	6948	10167	9041	21.89	3.113
22	12.802	2174	2176	2178	1995	5907	2102	5.09	.724
23	12.833	2182	2183	2185	5266	4185	2794	6.76	.962
24	12.855	2185	2188	2189	8150	8924	7070	17.12	2.434
25	13.005	2218	2222	2225	4133	6211	4035	9.77	1.389
26	13.103	2238	2244	2255	11399	27815	26685	64.61	9.187
27	13.387	2300	2308	2318	4224	9812	9812	23.76	3.378
28	13.502	2326	2334	2338	2174	3571	3571	8.65	1.229
29	14.139	2470	2478	2491	7285	13892	13892	33.63	4.783
30	15.375	2751	2757	2764	1558	3088	3088	7.48	1.063
31	15.928	2874	2882	2889	5594	8182	8182	19.81	2.817
32	19.774	3741	3751	3767	12800	32914	32914	79.69	11.332

Sum of corrected areas: 290455.

MONS 023796



>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

1863

File: >L0778 Scan #: 1863 Retn. time: 11.41

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
64.950	1575.0	79.050	1113.0	91.050	11336.0	92.050	6031.0	92.950	615.0

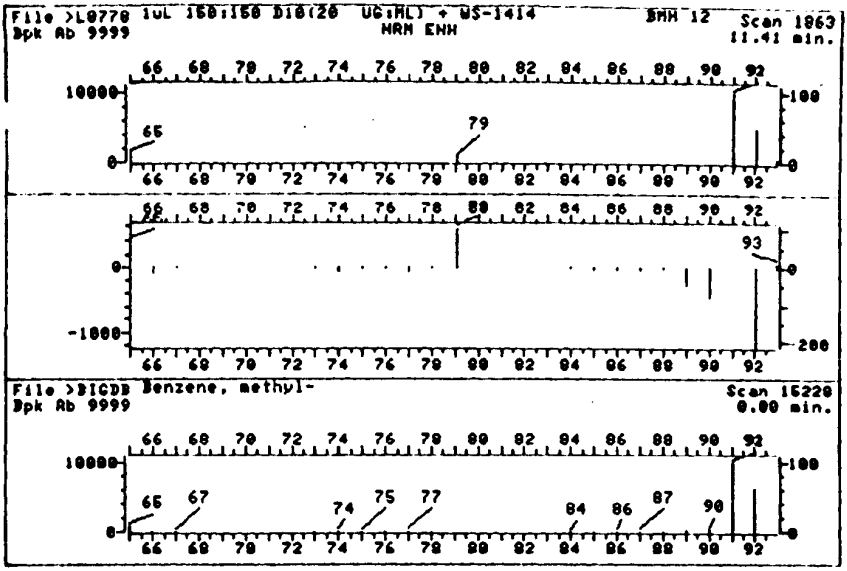
1.	Benzene, methyl-	92 C7H8
2.	Benzene, methyl-	92 C7H8
3.	Benzene, methyl-	92 C7H8
4.	Benzene, methyl-	92 C7H8
5.	Benzene, methyl-	92 C7H8
6.	Benzene, methyl-	92 C7H8
7.	1,3,5-Cycloheptatriene	92 C7H8
8.	1,3,5-Cycloheptatriene	92 C7H8
9.	TRICYCLO(4.1.0.0**2,7)HEPT-3-ENE	92 C7H8

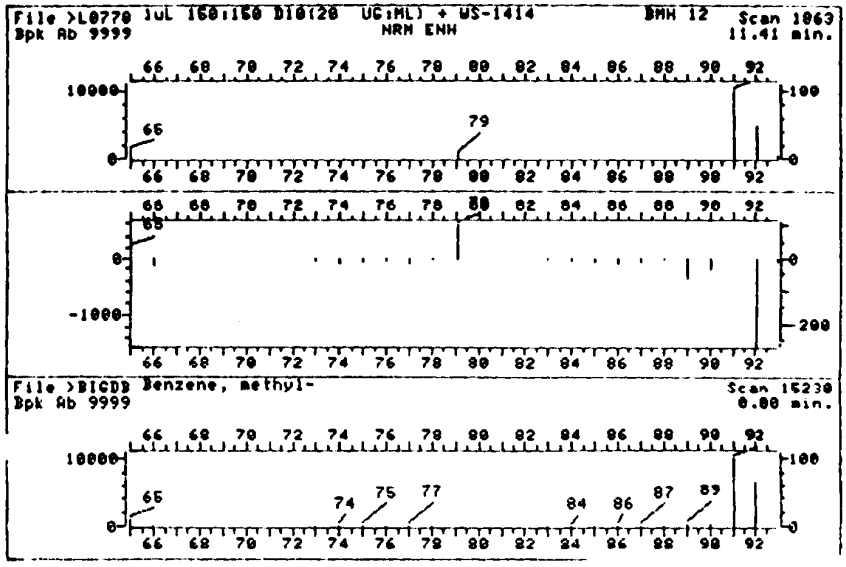
Sample file:)L0778 Spectrum #: 1863
Search speed: 2 Tilting option: N No. of ion ranges searched: 70

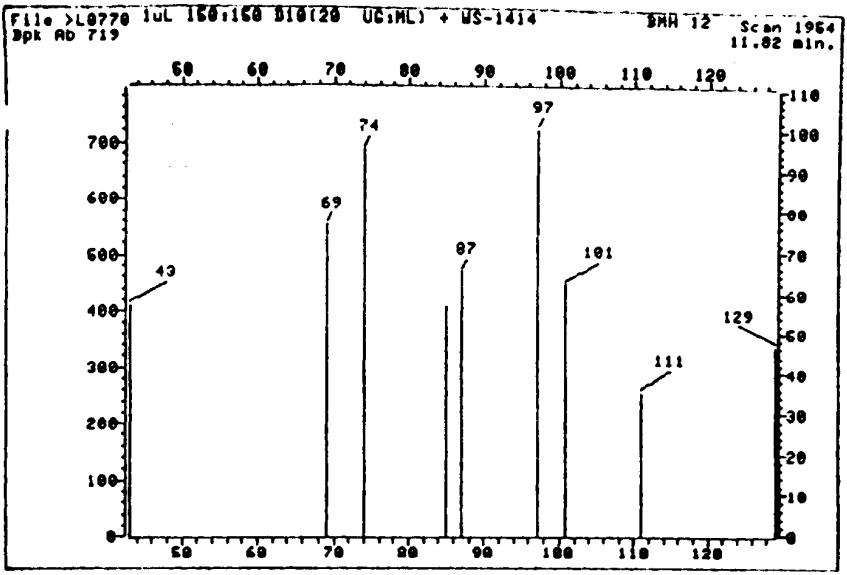
	Prob.	CAS #	CON #	ROOT	K	DK	#FLG	TILT	%	CON	C_I	R_IV
1.	60%	108883	15228	"BIGDB	25	53	2	0	85	14	30	14
2.	60%	108883	15230	"BIGDB	26	58	2	0	81	14	30	14
3.	60%	108883	15010	"BIGDB	27	63	2	0	73	14	30	14
4.	60%	108883	15229	"BIGDB	28	67	3	0	67	14	30	13
5.	60%	108883	15231	"BIGDB	28	68	2	0	71	14	30	14
6.	60%	108883	15234	"BIGDB	26	20	2	0	89	14	30	14
7.	52%	544252	15237	"BIGDB	22	30	1	0	96	17	20	14
8.	52%	544252	15235	"BIGDB	22	63	2	0	81	17	20	13
9.	52%	0	15013	"BIGDB	22	0	0	0	100	17	20	16

No!! Wrong RT for toluene

MONS 023798


File >L0770 1uL 150:150 DI0(20 UG:ML) + WS-1414
Bpk Ab 9999
NRM ENH
BMH 12
Scan 1063
11.41 min.
File >BIGDB Benzene, methyl-
Bpk Ab 9999
Scan 15220
0.00 min.


File >L0770 1uL 150:150 DI0(20 UG:ML) + WS-1414
Bpk Ab 9999
NRM ENH
BMH 12
Scan 1063
11.41 min.
File >BIGDB Benzene, methyl-
Bpk Ab 9999
Scan 15230
0.00 min.

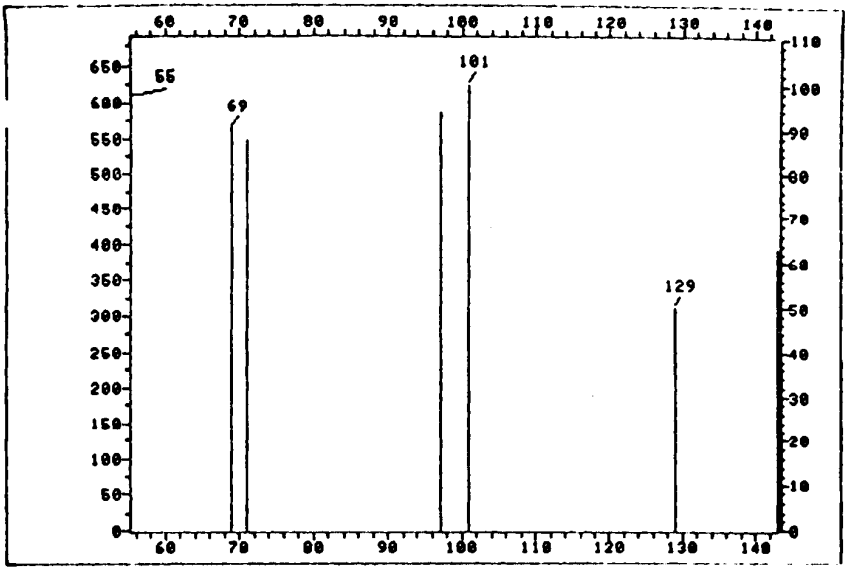


>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

1954

File: >L0778 Scan #: 1954 Retn. time: 11.82

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
42.95	409.00	73.95	687.00	86.95	471.00	100.95	446.00	129.05	335.00
69.05	552.00	85.05	409.00	97.05	719.00	111.05	253.00		

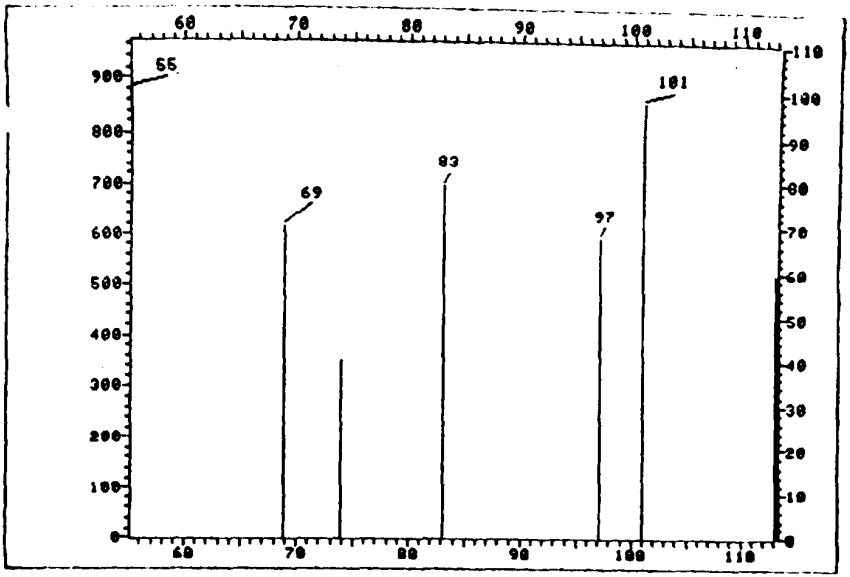


>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

1988

File: >L0778 Scan #: 1988 Retn. time: 11.97

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
55.05	604.00	70.95	547.00	100.95	624.00	128.95	312.00	143.00	394.00
68.95	564.00	97.05	585.00						



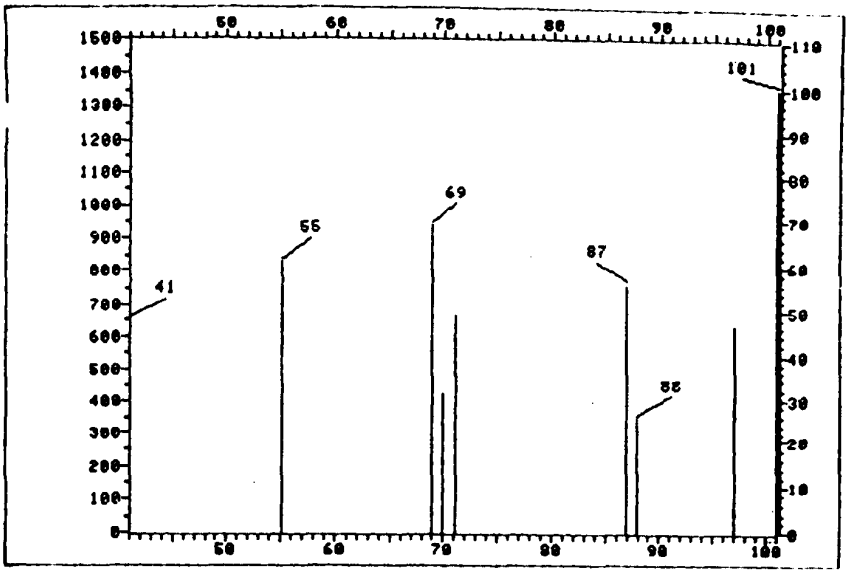
>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

1999

File: >L0778 Scan #: 1999 Retn. time: 12.02

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
55.05	877.00	73.95	351.00	97.05	594.00	100.95	855.00	113.05	523.00
68.95	615.00	83.05	696.00						

MONS 023802

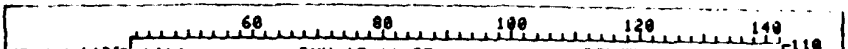


>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

2004

File: >L0778 Scan #: 2004 Retn. time: 12.04

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
40.95	649.00	68.95	939.00	71.05	665.00	87.95	348.00	100.95	1360.00
55.05	829.00	69.95	427.00	86.95	761.00	97.05	633.00		



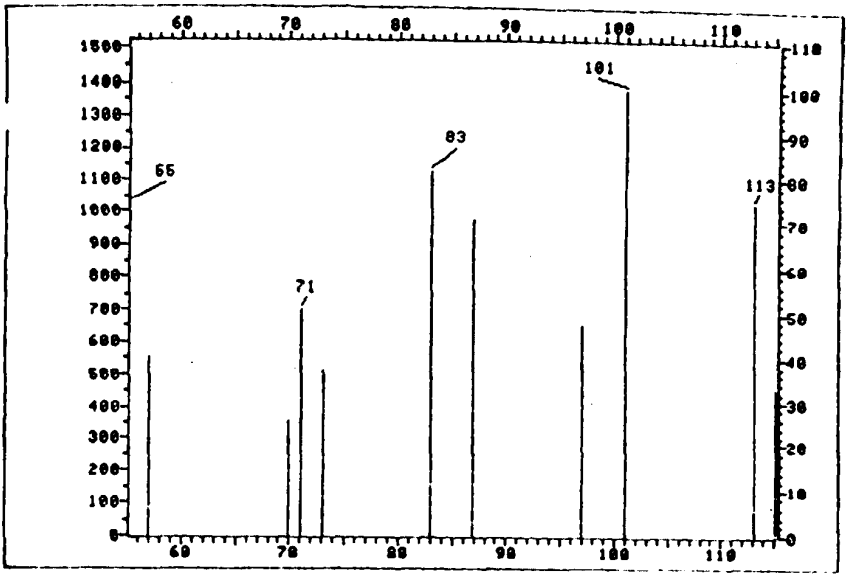
UG:ML) + WS-1414 BMH 12:11:85

2011

File:)L0778 Scan #: 2011 Retn. time: 12.07

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
40.95	807.00	71.05	730.00	87.05	1221.00	101.05	1386.00	115.05	596.00
55.05	877.00	73.80	435.00	97.05	677.00	113.05	674.00	142.00	303.00
68.95	789.00	83.05	1134.00						

MONS 023804



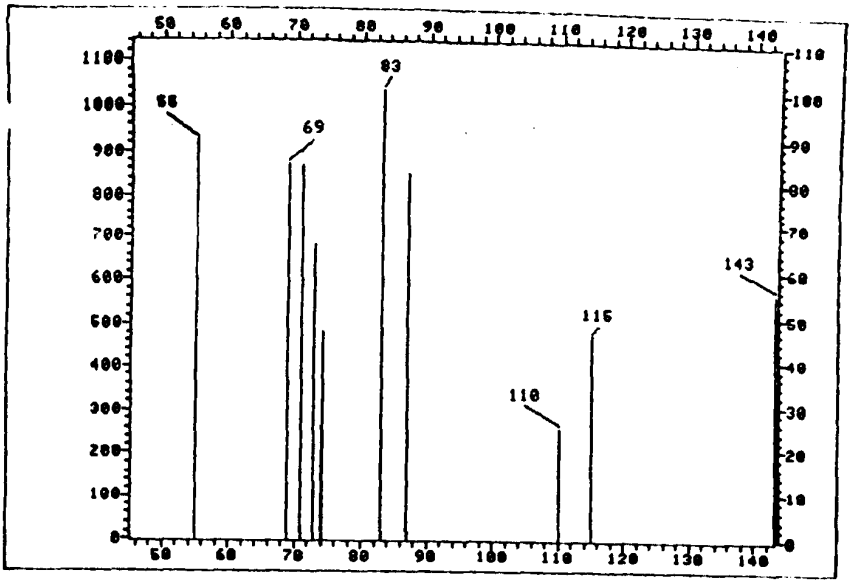
>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

2016

File: >L0778 Scan #: 2016 Retn. time: 12.09

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
55.05	1021.00	71.05	702.00	83.05	1127.00	97.05	653.00	113.05	1031.00
57.05	552.00	73.05	508.00	86.95	979.00	100.95	1381.00	115.05	454.00
69.95	357.00								

MONS 023805

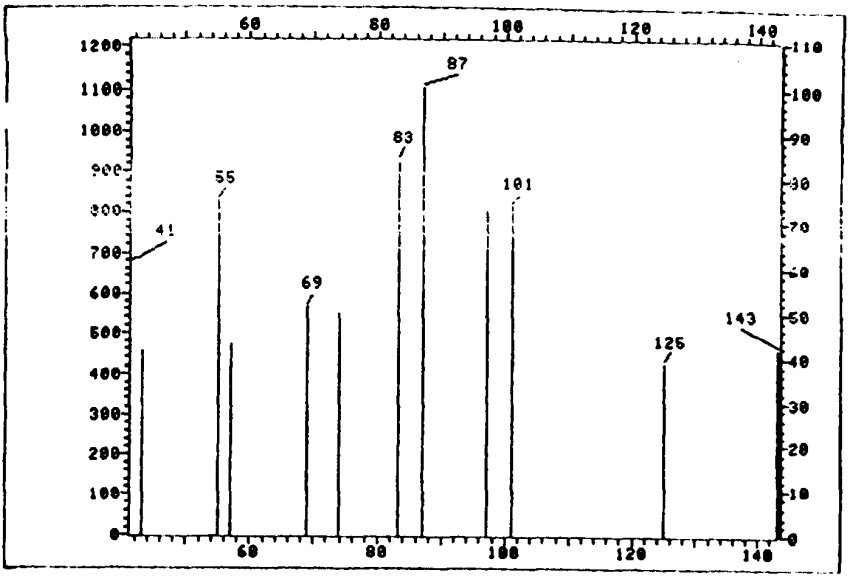


>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

2026

File: >L0778 Scan #: 2026 Retn. time: 12.14

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
44.95	340.00	71.05	866.00	74.05	482.00	86.95	850.00	115.05	475.00
55.05	927.00	72.95	682.00	83.05	1037.00	110.05	260.00	143.15	573.00
68.95	872.00								

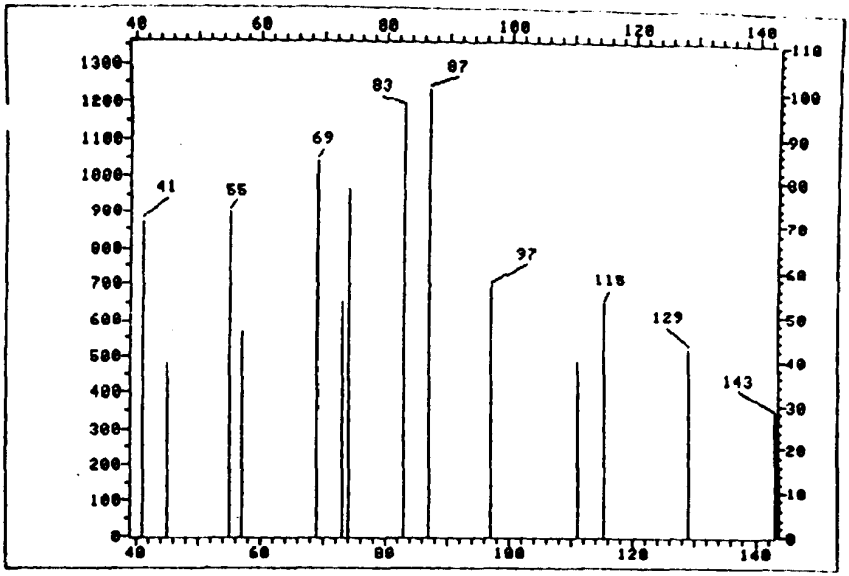


>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

2037

File: >L0778 Scan #: 2037 Retn. time: 12.18

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
40.95	667.00	57.05	473.00	83.05	930.00	97.05	803.00	125.05	427.00
43.05	452.00	68.95	568.00	87.05	1106.00	101.05	816.00	143.00	462.00
55.05	830.00	73.95	547.00						

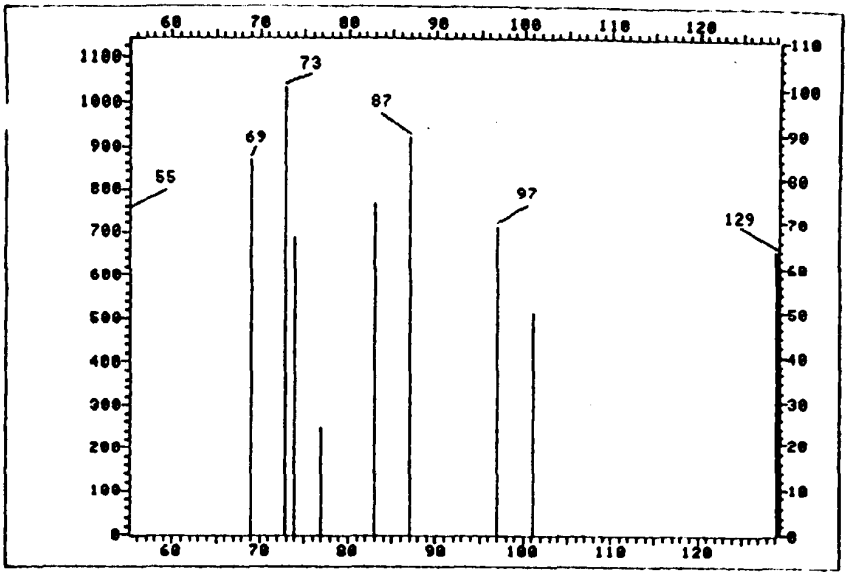


>L0778 ivL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

2045

File: >L0778 Scan #: 2045 Retn. time: 12.22

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
38.95	277.00	55.05	890.00	72.95	655.00	87.05	1231.00	115.05	653.00
40.95	873.00	57.05	570.00	73.95	961.00	97.05	696.00	128.95	523.00
44.95	477.00	68.95	1038.00	83.05	1181.00	111.05	487.00	143.00	345.00

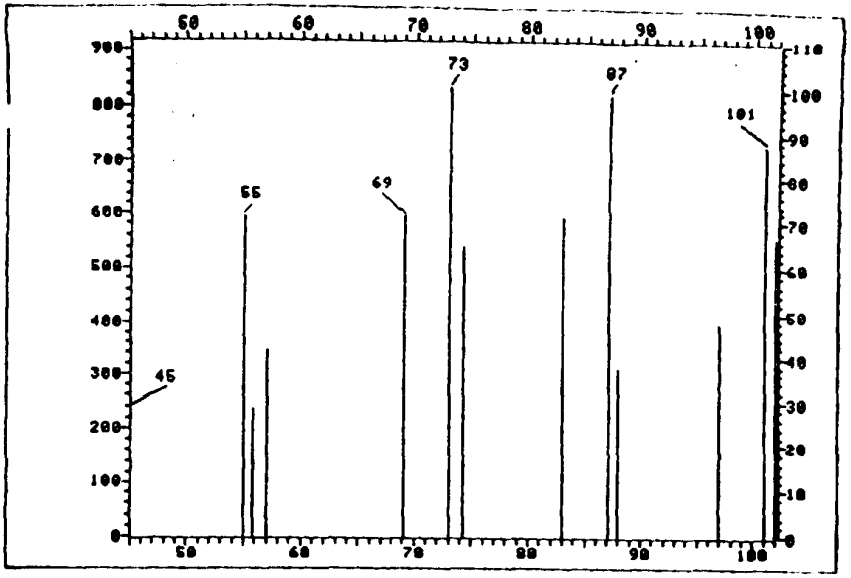


>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

2051

File: >L0778 Scan #: 2051 Retn. time: 12.25

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
55.05	746.00	72.95	1032.00	76.95	246.00	87.05	918.00	101.05	513.00
68.95	867.00	73.95	688.00	83.05	767.00	96.95	710.00	129.05	659.00

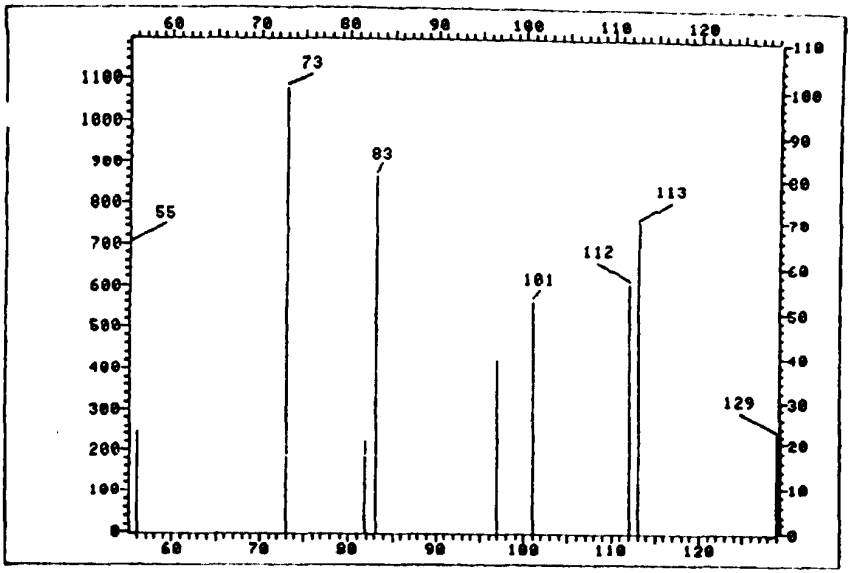


>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

2059

File: >L0778 Scan #: 2059 Retn. time: 12.28

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
44.95	232.00	57.05	345.00	74.20	541.00	87.95	313.00	101.05	729.00
55.05	595.00	69.05	595.00	83.05	593.00	96.95	395.00	101.95	559.00
55.80	234.00	73.05	833.00	87.05	822.00				

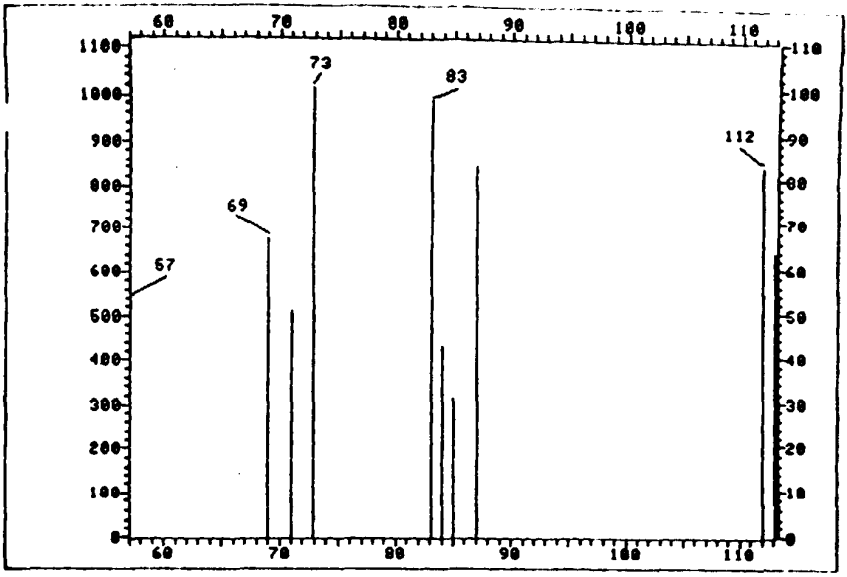


>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

2065

File: >L0778 Scan #: 2065 Retn. time: 12.31

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
55.05	693.00	73.05	1079.00	83.05	866.00	101.05	562.00	113.05	755.00
55.95	244.00	81.95	223.00	96.95	418.00	112.05	605.00	128.95	241.00

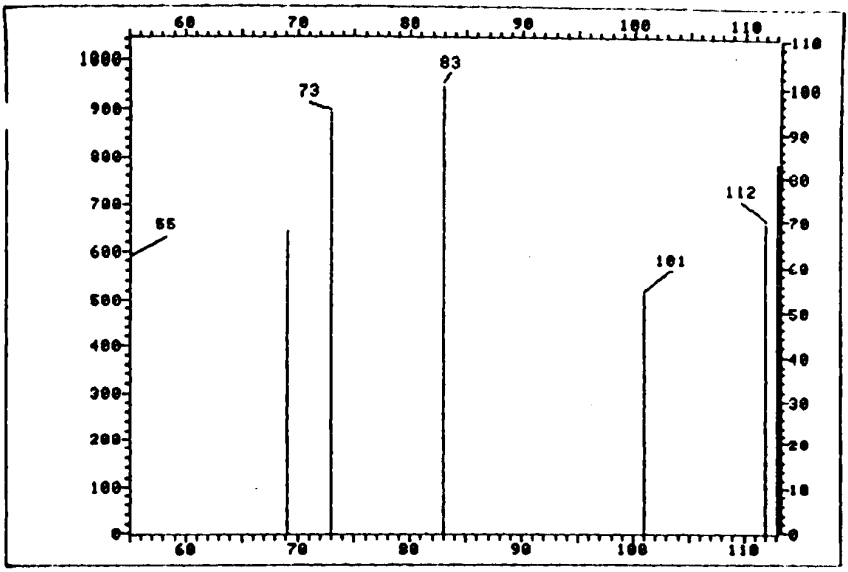


>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

2069

File: >L0778 Scan #: 2069 Retn. time: 12.33

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
56.95	537.00	71.05	511.00	83.05	984.00	84.95	312.00	112.05	838.00
69.05	676.00	72.95	1014.00	84.05	428.00	87.05	837.00	113.05	646.00

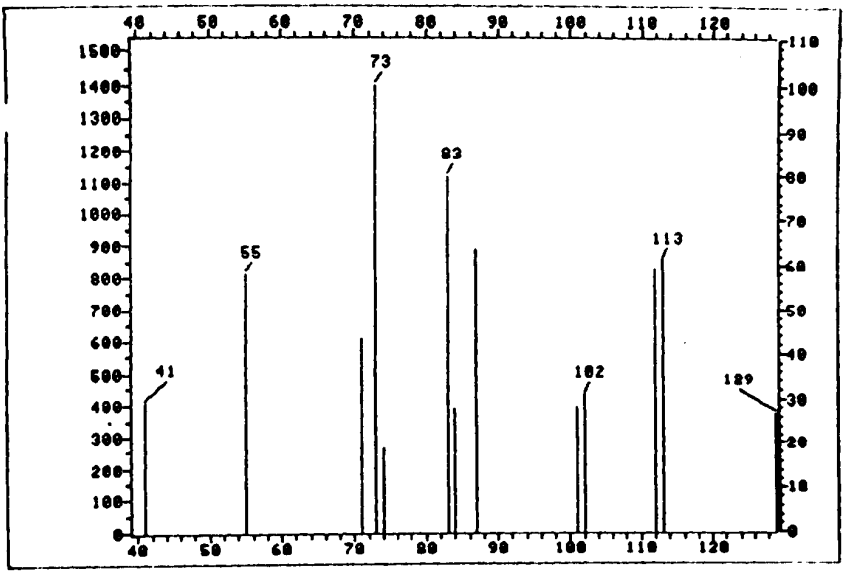


>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

2076

File: >L0778 Scan #: 2076 Retn. time: 12.36

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
54.95	583.00	72.95	889.00	100.95	512.00	112.05	658.00	113.05	705.00
69.05	640.00	83.05	946.00						



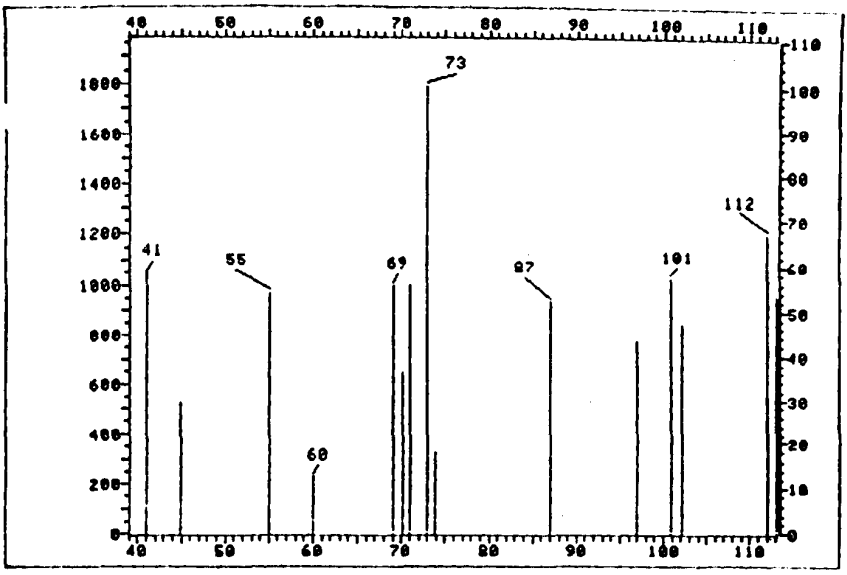
>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

2081

File: >L0778 Scan #: 2081 Retn. time: 12.38

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
38.95	346.00	71.05	606.00	83.05	1115.00	101.05	391.00	113.05	855.00
40.95	405.00	72.95	1396.00	84.05	389.00	102.05	431.00	128.95	373.00
55.05	810.00	73.95	261.00	86.95	888.00	112.05	822.00		

MONS 023814



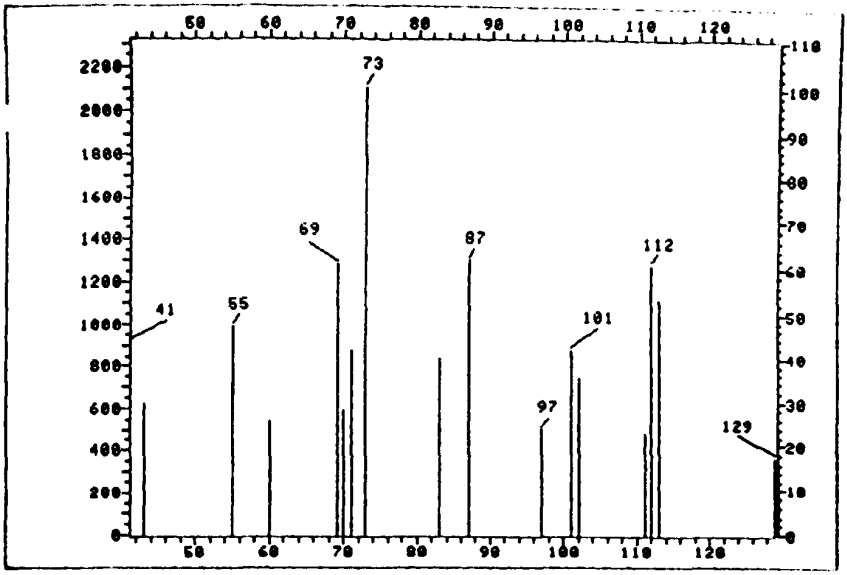
>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

2090

File: >L0778 Scan #: 2090 Retn. time: 12.42

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
38.95	356.00	59.95	227.00	71.05	997.00	87.05	936.00	102.05	842.00
41.05	1046.00	69.05	998.00	72.95	1787.00	97.05	777.00	112.85	1197.00
44.95	527.00	70.05	643.00	73.95	325.00	100.95	1024.00	113.05	952.00
55.05	968.00								

MONS 023815



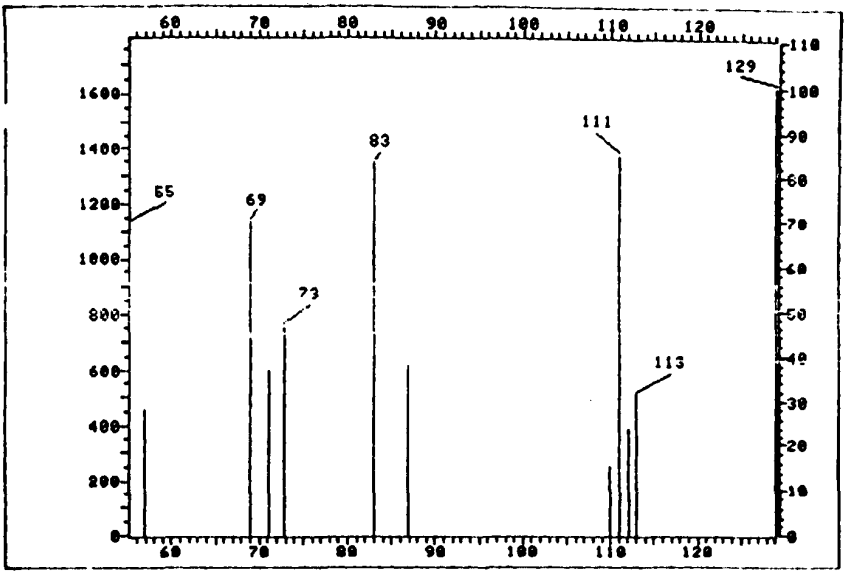
>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMM 12:11:85

2094

File: >L0778 Scan #: 2094 Retn. time: 12.44

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
40.95	911.00	69.05	1282.00	83.05	837.00	100.95	878.00	112.05	1279.00
43.05	624.00	70.05	593.00	87.05	1304.00	101.95	742.00	113.05	1115.00
55.05	995.00	71.05	874.00	97.05	502.00	111.05	482.00	128.95	367.00
59.95	541.00	72.95	2104.00						

MONS 023816

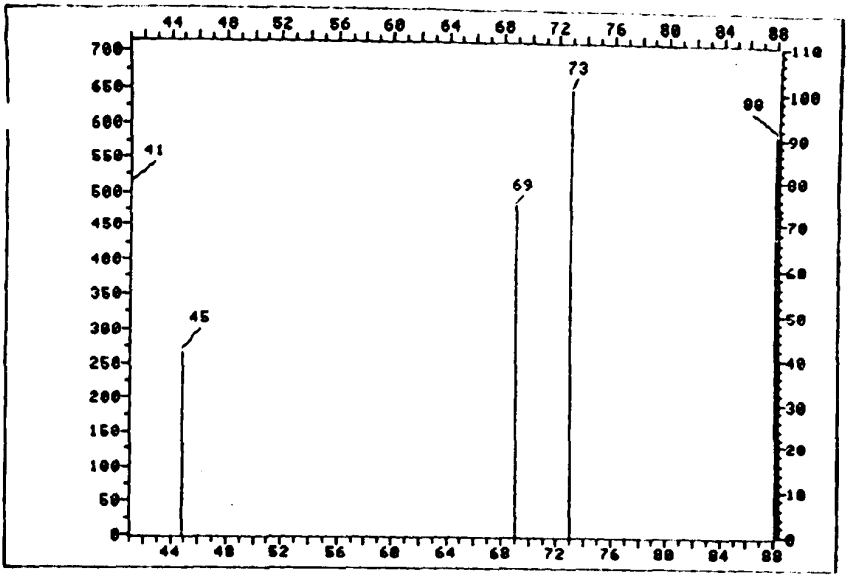


>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

2118

File: >L0778 Scan #: 2118 Retn. time: 12.54

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
55.05	1122.00	71.05	595.00	86.95	617.00	111.05	1377.00	113.05	505.00
56.95	455.00	72.80	751.00	110.05	248.00	112.05	390.00	129.05	1624.00
69.05	1134.00	83.05	1346.00						

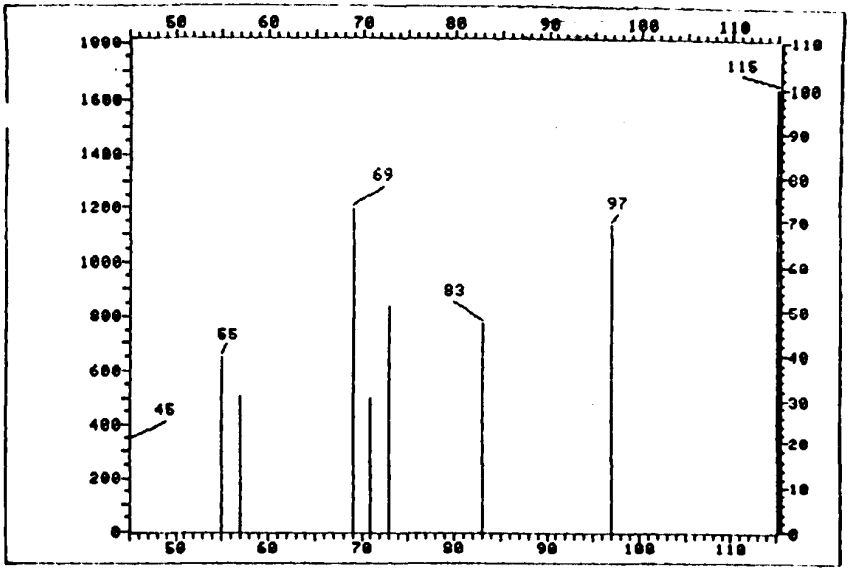


>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

2138

File: >L0778 Scan #: 2138 Retn. time: 12.63

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
40.950	509.00	44.000	266.00	69.050	480.00	72.950	648.00	88.050	587.00

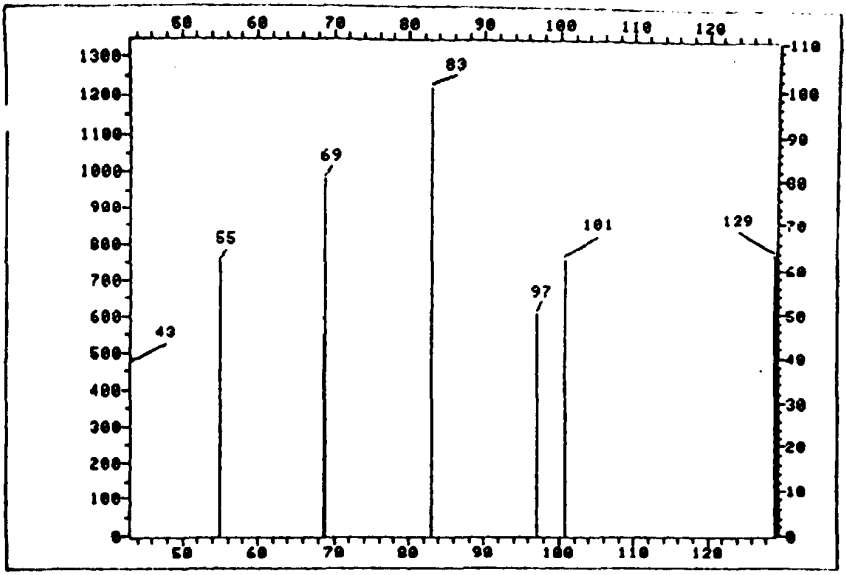


>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

2159

File: >L0778 Scan #: 2159 Retn. time: 12.73

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
44.95	328.00	56.95	502.00	70.95	496.00	83.05	771.00	115.05	1646.00
54.95	647.00	69.05	1195.00	72.95	832.00	97.05	1136.00		



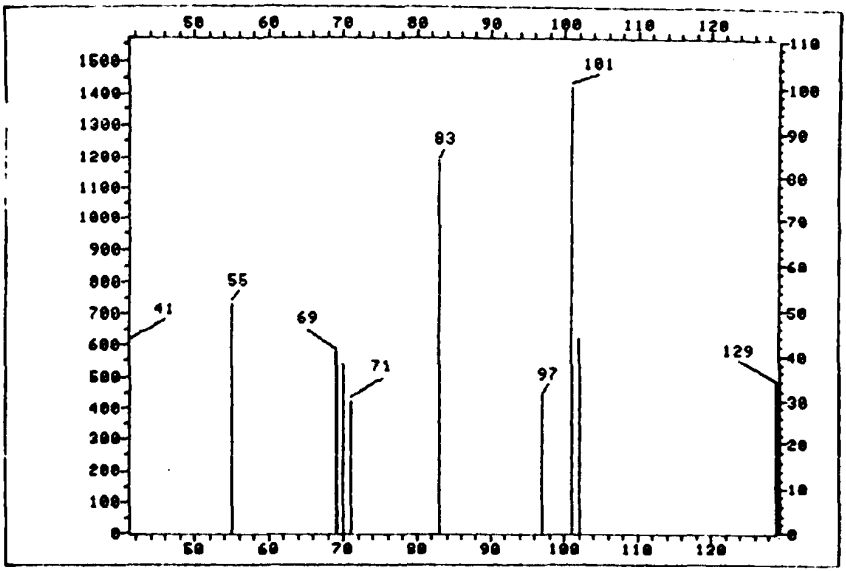
>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

2176

File: >L0778 Scan #: 2176 Retn. time: 12.80

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
42.95	464.00	68.95	977.00	97.05	611.00	100.95	759.00	129.05	773.00
55.05	753.00	83.05	1220.00						

MONS 023820



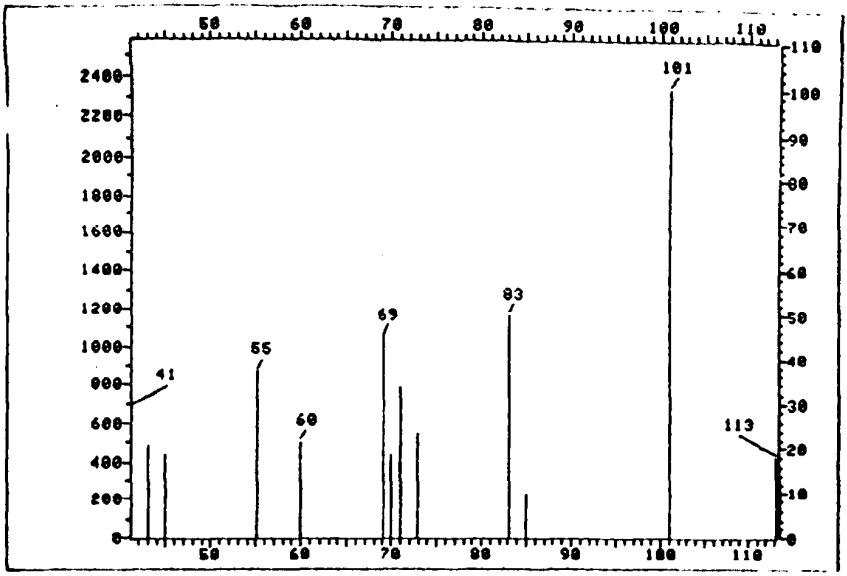
>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

2183

File: >L0778 Scan #: 2183 Retn. time: 12.83

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
40.95	606.00	69.85	575.00	71.05	421.00	97.05	437.00	101.95	621.00
55.05	731.00	70.85	536.00	83.05	1183.00	100.95	1421.00	128.95	478.00

MONS 023821



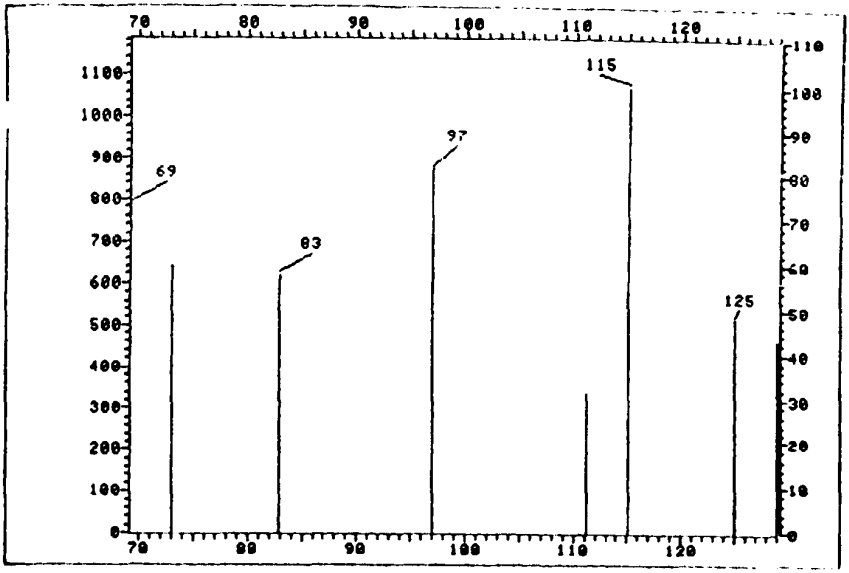
>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

2188

File: >L0778 Scan #: 2188 Retn. time: 12.05

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
41.05	668.00	55.05	867.00	69.95	430.00	83.05	1161.00	101.05	2335.00
42.95	475.00	59.95	498.00	71.05	781.00	84.95	230.00	113.05	420.00
44.95	434.00	69.05	1050.00	72.95	544.00				

MONS 023822



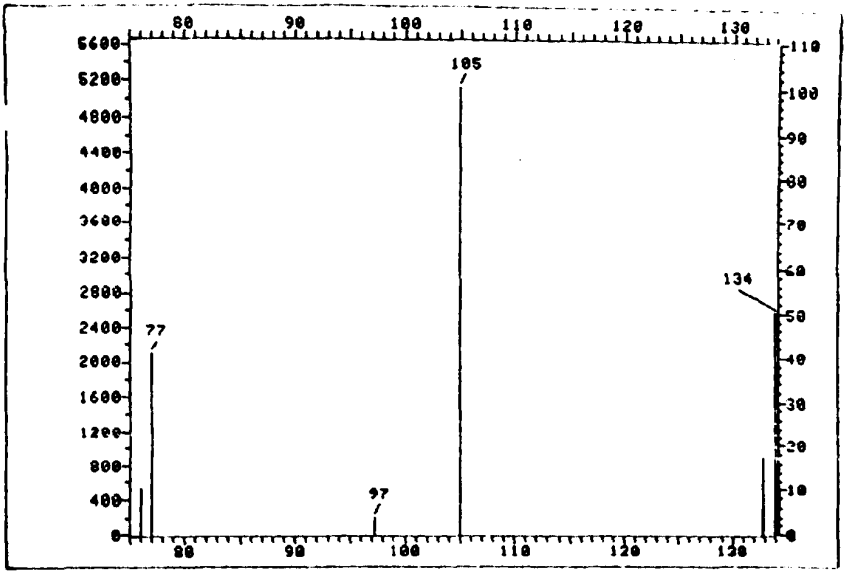
>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

2222

File: >L0778 Scan #: 2222 Retn. time: 13.01

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
69.05	785.00	82.95	615.00	111.20	339.00	124.95	516.00	128.95	461.00
72.95	639.00	97.05	874.00	115.05	1073.00				

MONS 023823



>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

2244

File: >L0778 Scan #: 2244 Retn. time: 13.10

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
74.95	215.00	76.95	2100.00	104.95	5124.00	132.95	889.00	133.95	2576.00
75.95	541.00	97.20	204.00						

MONS 023824

S

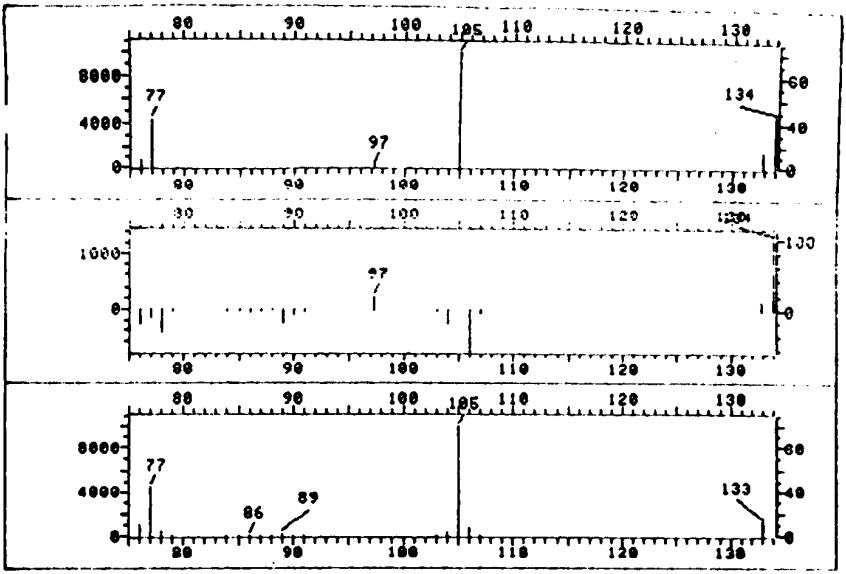
1. 1(3H)-Isobenzofuranone 134 C8H6O2
2. 1(3H)-Isobenzofuranone 134 C8H6O2

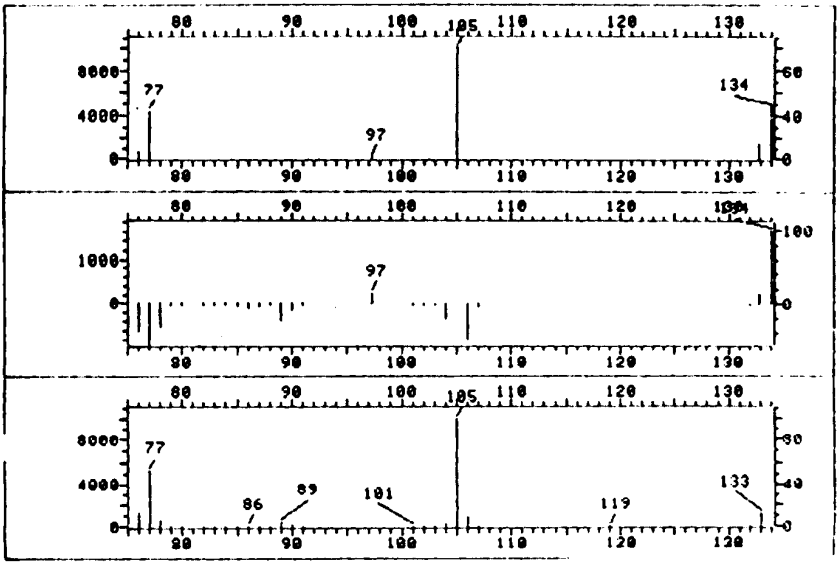
Sample file: >L0778 Spectrum #: 2244
Search speed: 2 Tilting option: N No. of ion ranges searched: 82

	Prob.	CAS #	CON #	ROOT	K	DK	#FLG	TILT	%	CON	C_I	R_IV
1.	52*	87412	31496	"BIGDB	27	68	2	0	92	16	20	14
2. →	52*	87412	31225	"BIGDB	29	71	2	0	78	18	20	14

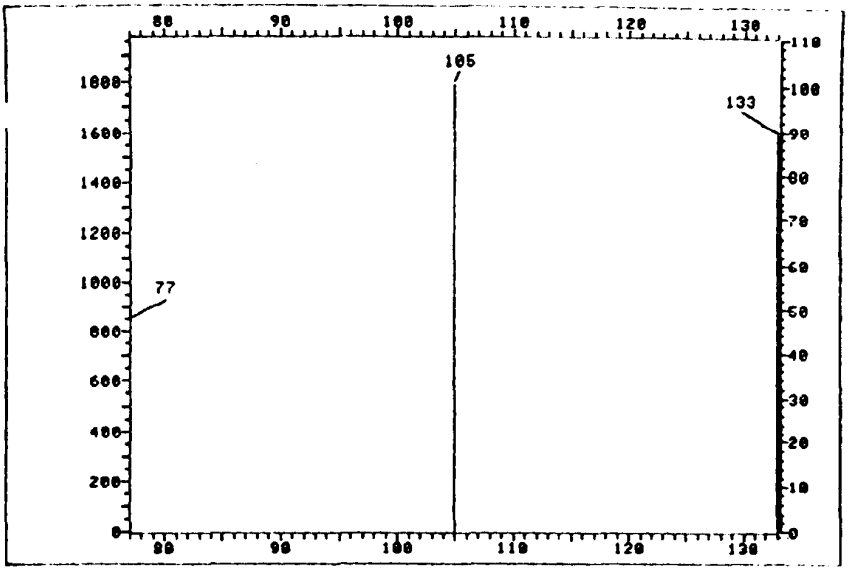
yes

MONS 023825


80
90
100
105
110
120
130
8000
4000
0
77
97
134
60
40
0
1000
100
86
89
133


80
90
100
105
110
120
130
8000
4000
0
77
97
134
60
40
1000
100
86
89
101
119
133

MONS 023826

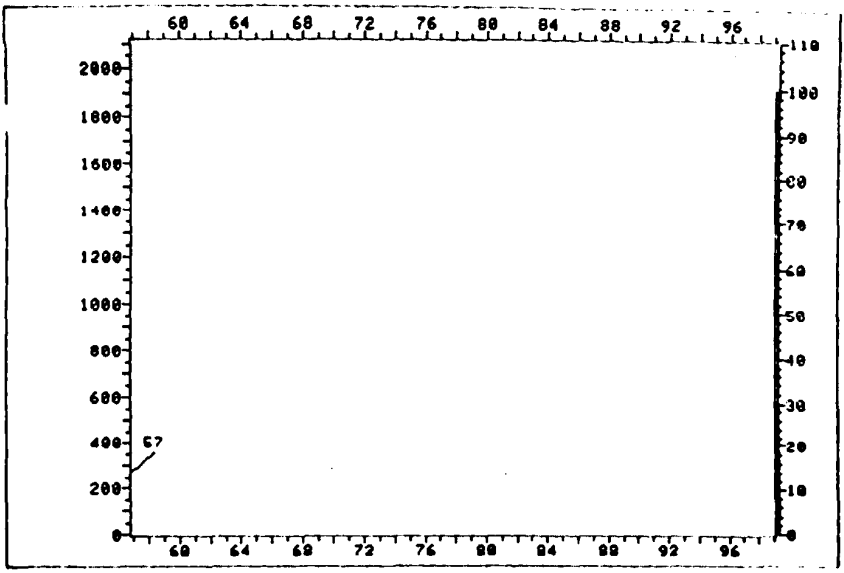


>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

2308

File: >L0778 Scan #: 2308 Retn. time: 13.39

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
76.95	840.00	104.95	1789.00	132.95	1595.00				

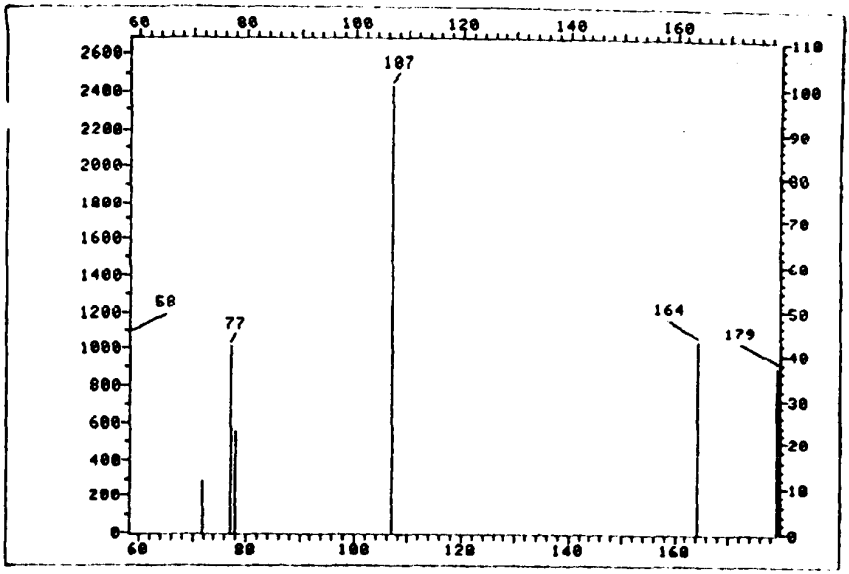


>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

2334

File: >L0778 Scan #: 2334 Retn. time: 13.50

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
56.700	252.00	99.050	1922.00						

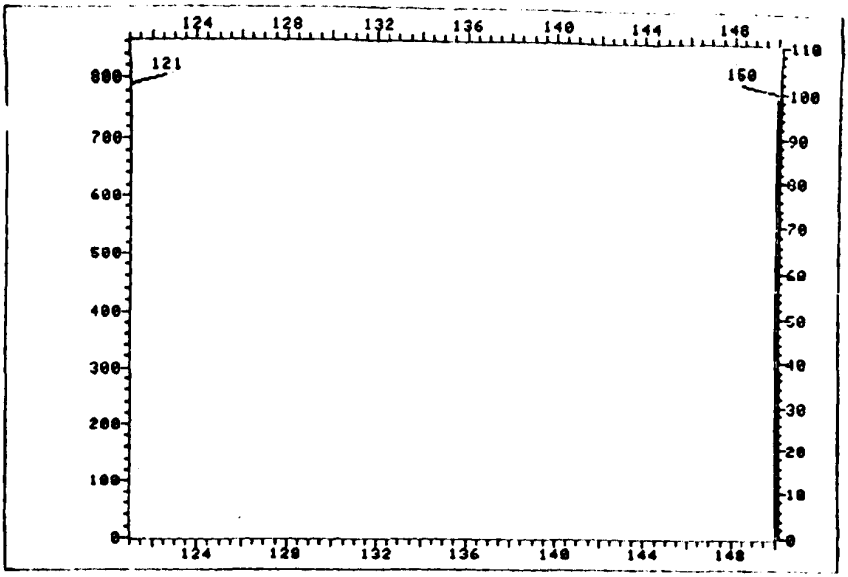


>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

2478

File: >L0778 Scan #: 2478 Retn. time: 14.14

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
58.05	1065.00	76.95	1014.00	107.05	2431.00	164.15	1041.00	179.15	905.00
71.95	276.00	77.95	553.00						

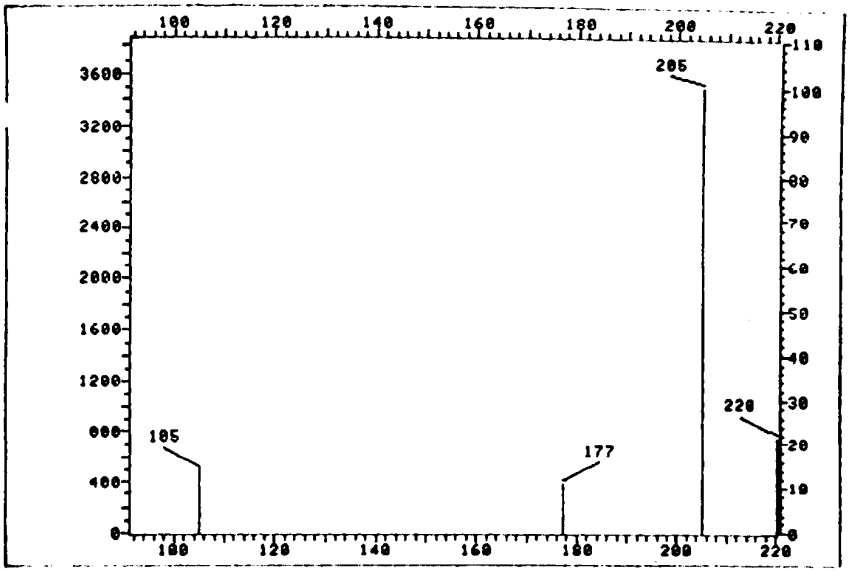


>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

2757

File: >L0778 Scan #: 2757 Retn. time: 15.37

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
120.95	783.00	150.00	775.00						

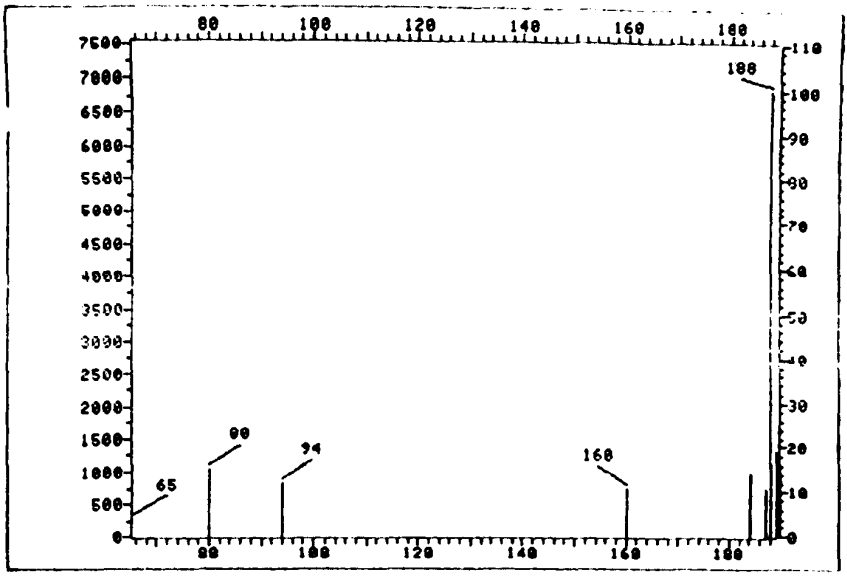


>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

2882

File: >L0778 Scan #: 2882 Retn. time: 15.93

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
90.95	473.00	104.95	494.00	177.15	379.00	205.15	3503.00	220.15	745.00



>L0778 1uL 150:150 D10(20 UG:ML) + WS-1414 BMH 12:11:85

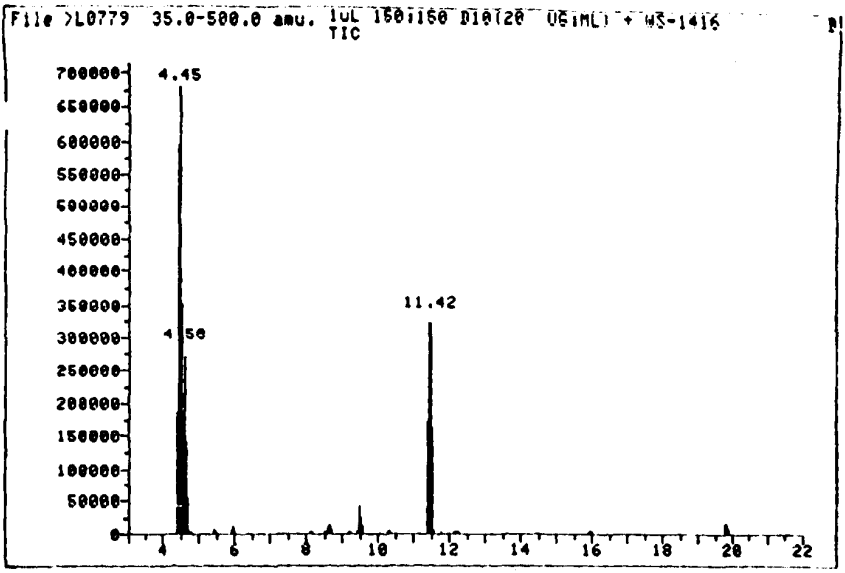
3751

File: >L0778 Scan #: 3751 Retn. time: 19.77

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
64.95	283.00	94.05	837.00	184.15	985.00	188.15	6847.00	189.15	1317.00
79.95	1056.00	160.15	741.00	187.15	734.00				

MONS 023832

Figure 3. Total ion chromatograms, mass spectra, and library search outputs obtained from the analysis of the WS-1416 extract of 21S.



Peak at (4.45 + 4.58) mins – Chloromethylbenzene isomer

$\frac{3,135,392}{3511}$ = 893 µg/ml

= 921 µg/Liter

Peak at 11.42 $\frac{667038}{3571}$ = 190 µg/ml – isopropylbenzene, or butylbenzene, or propylmethylbenzene

= 196 µg/Liter

>L0779 1uL 150:150 D10(20 UG:ML) + WS-1416 BMH 12:11:85

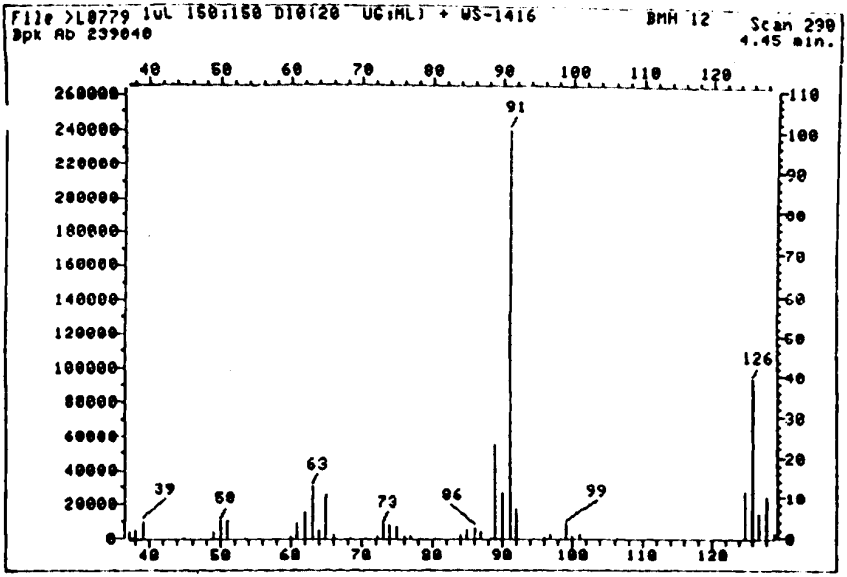
35.01 500.0 TIC

Peak #	R.T. min.	first scan	max scan	last scan	peak height	raw area	corr. area	corr. % max.	% of total
1	4.448	267	290	308	680217	2404933	2404933	100.00	63.247
2	4.581	308	320	340	270079	746371	730459	30.37	19.210
3	11.422	1851	1865	1887	322243	667038	667038	27.74	17.542

Sum of corrected areas: 3802430.

3,135,392

MONS 023835



>L0779 1uL 150:150 D10(20 UG:ML) + WS-1416 BMH 12:11:85

290

File: >L0779 Scan #: 290 Retn. time: 4.45

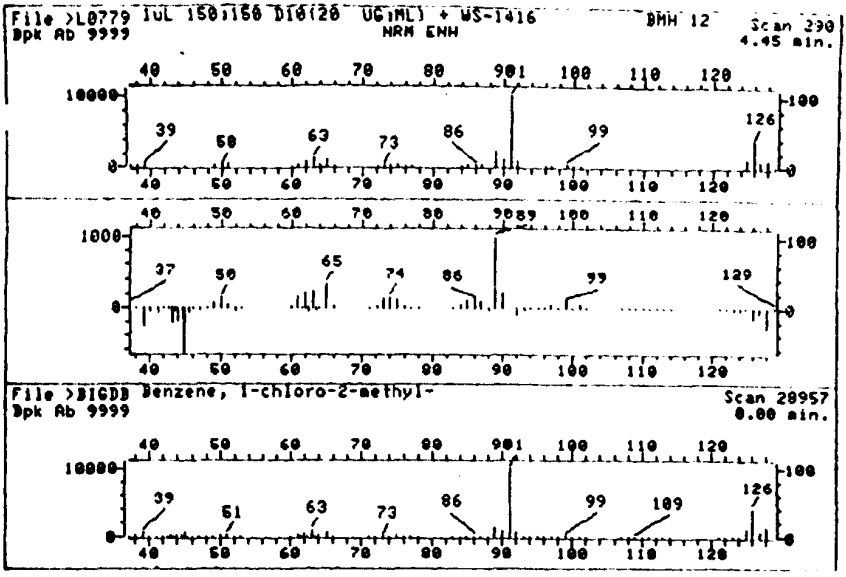
m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
36.95	3410.	60.95	8138.	73.95	6975.	88.95	55120.	99.95	632.
37.95	4282.	62.05	14328.	74.95	6933.	90.05	26480.	100.95	2192.
39.05	9317.	63.05	30272.	76.05	1203.	91.05	239040.	124.95	27336.
44.80	295.	64.05	4900.	76.95	678.	92.05	16528.	125.95	93760.
48.95	3387.	64.95	25776.	83.95	2213.	95.95	376.	126.95	13831.
50.05	9878.	66.05	1538.	84.95	4721.	96.95	1976.	127.95	23880.
51.05	9749.	72.05	1205.	85.95	5748.	98.95	8273.	128.95	2585.
60.05	1466.	72.95	7901.	86.95	3706.				

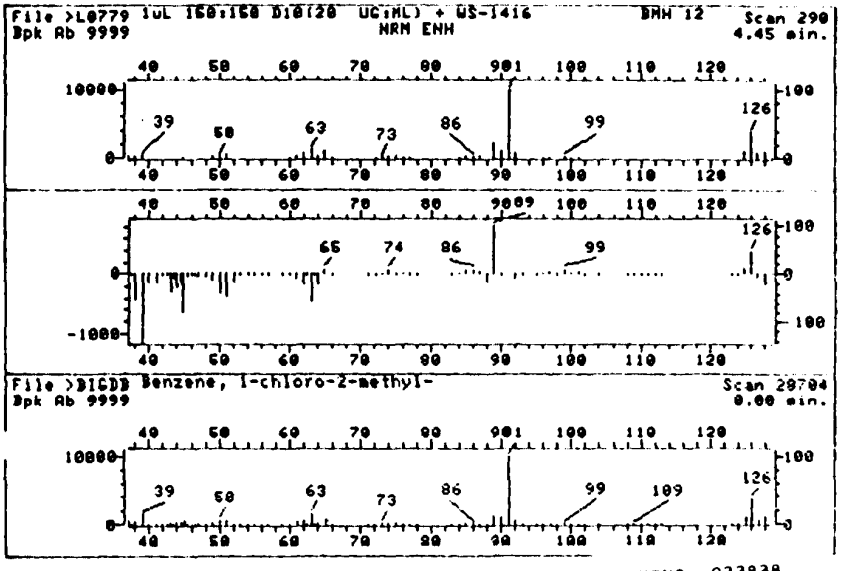
S

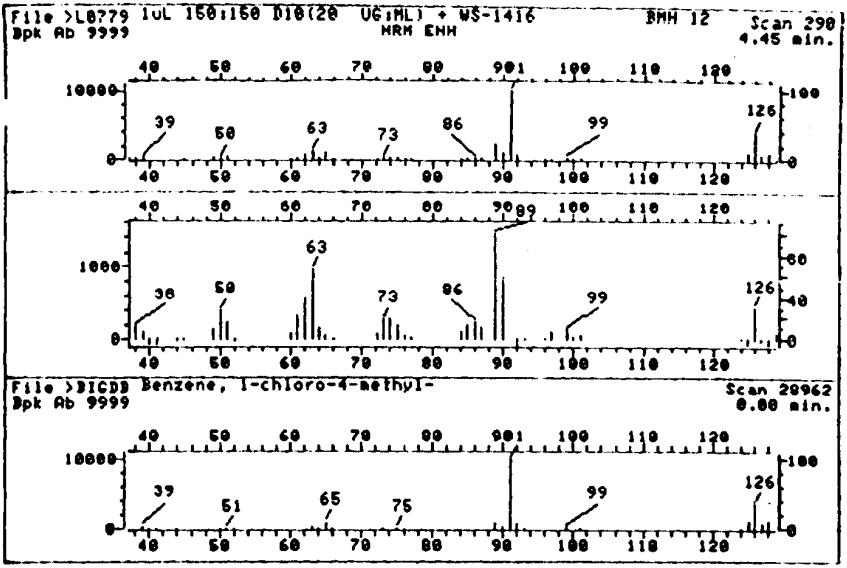
1.	Benzene, 1-chloro-2-methyl-	126	C7H7Cl
2.	Benzene, 1-chloro-2-methyl-	126	C7H7Cl
3.	Benzene, 1-chloro-4-methyl-	126	C7H7Cl
4.	Benzene, 1-chloro-3-methyl-	126	C7H7Cl
5.	Benzene, 1-chloro-4-methyl-	126	C7H7Cl
6.	Benzene, 1-chloro-2-methyl-	126	C7H7Cl
7.	Benzene, 1-chloro-3-methyl-	126	C7H7Cl
8.	Benzene, 1-chloro-4-methyl-	126	C7H7Cl
9.	Benzene, 1-chloro-3-methyl-	126	C7H7Cl
10.	Benzene, (chloromethyl)-	126	C7H7Cl

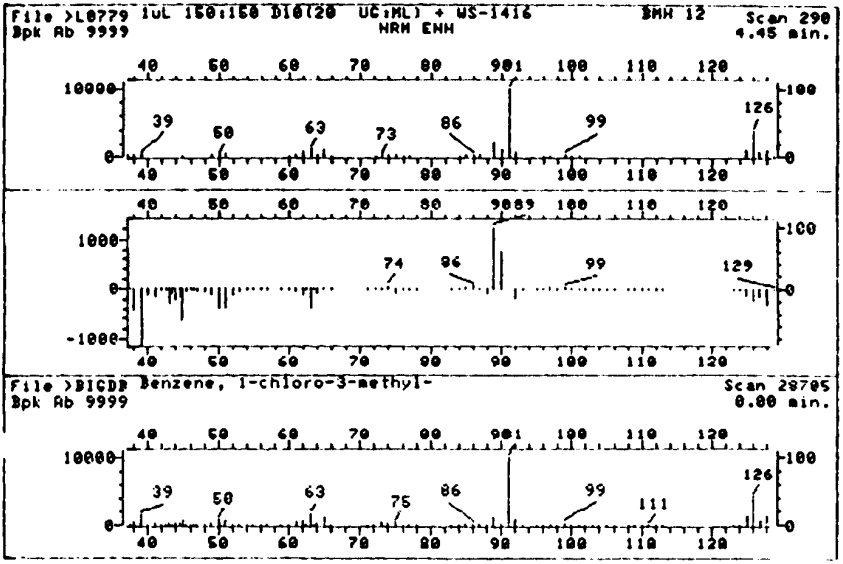
Sample file:)L0779 Spectrum #: 290
Search speed: 2 Tilting option: N No. of ion ranges searched: 47

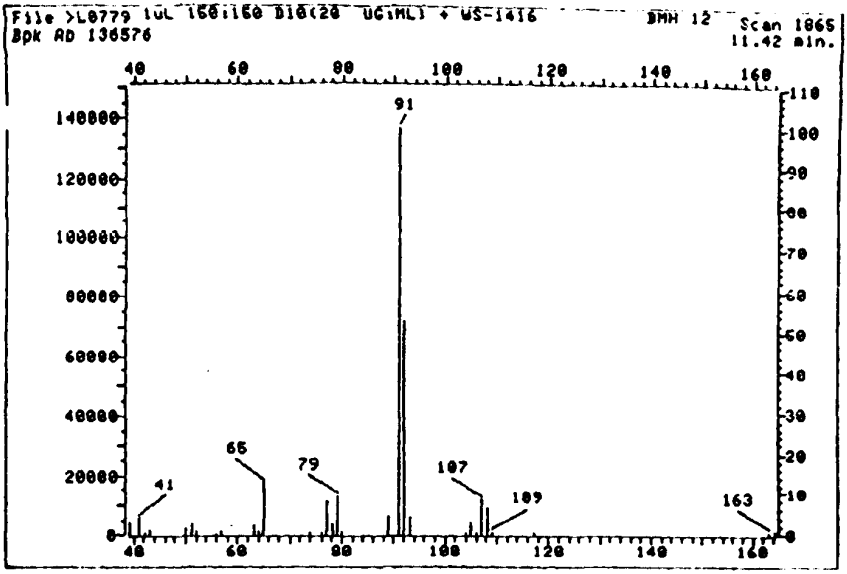
	Prob.	CAS #	CON #	ROOT	K	DK	#FLG	TILT	%	CON	C_I	R_IV
1.	95%	95498	28957	*BIGDB	81	16	0	0	75	7	68	94
2.	84%	95498	28704	*BIGDB	73	29	0	0	56	42	43	92
3.	83%	106434	28962	*BIGDB	62	23	0	0	91	14	51	78
4.	76%	108418	28705	*BIGDB	74	27	2	0	74	14	40	56
5.	76%	106434	28706	*BIGDB	72	28	2	0	71	15	40	54
6.	74%	95498	28958	*BIGDB	59	29	0	0	46	30	37	72
7.	74%	108418	28961	*BIGDB	52	34	1	0	76	15	39	44
8.	72%	106434	28963	*BIGDB	60	31	0	0	65	35	32	73
9.	69%	108418	28960	*BIGDB	55	37	0	0	64	35	26	66
10.	59%	100447	28966	*BIGDB	46	43	0	0	81	38	21	54


File >L0779 1uL 150:150 D10(20 UG:ML) + WS-1416 BMH 12
Bpk Ab 9999
NRM ENH
Scan 290
4.45 min.
39
50
63
73
86
99
126
37
50
65
74
86
99
129
File >BIGDB Benzene, 1-chloro-2-methyl-
Bpk Ab 9999
Scan 28957
0.00 min.
39
51
63
73
86
99
109
126


File >L0779 1uL 150:150 D10(20 UG:ML) + WS-1416 BMH 12
Bpk Ab 9999
NRM ENH
Scan 290
4.45 min.
39
50
63
73
86
99
126
65
74
86
99
126
File >BIGDB Benzene, 1-chloro-2-methyl-
Bpk Ab 9999
Scan 29704
0.00 min.
39
50
63
73
86
99
109
126


File >L0779 1uL 150:150 D10(20 UG:ML) + WS-1416 BMH 12 Scan 290
Bpk Ab 9999 MRM ENH 4.45 min.
39 50 63 73 86 99 126
File >BICDB Benzene, 1-chloro-4-methyl- Scan 29962
Bpk Ab 9999 0.00 min.
39 51 65 75 99 126


File >L0779 1uL 150:150 D10(20 UG:ML) + WS-1416 BMH 12 Scan 290
Bpk Ab 9999 MRM ENH 4.45 min.
39 50 63 73 86 99 126
74 96 99 129
File >BICDB Benzene, 1-chloro-3-methyl- Scan 28705
Bpk Ab 9999 0.00 min.
39 50 63 75 86 99 111 126



>L0779 1uL 150:150 D10(20 UG:ML) + WS-1416 BMH 12:11:85

1865

File: >L0779 Scan #: 1865 Retn. time: 11.42

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
39.0S	4842.	56.0S	254.	75.9S	767.	91.0S	136576.	107.0S	11445.
41.0S	5610.	57.0S	1017.	76.9S	11186.	92.0S	71800.	108.0S	8677.
42.0S	643.	63.0S	3024.	78.0S	3527.	93.0S	6008.	108.9S	818.
43.0S	1762.	64.0S	1169.	79.0S	12581.	104.0S	791.	117.0S	703.
50.0S	2014.	64.9S	17728.	80.0S	1161.	104.9S	4328.	163.1S	603.
51.0S	3667.	73.9S	821.	88.9S	6157.	106.0S	671.	164.1S	1364.
52.0S	1301.								

MONS 023840

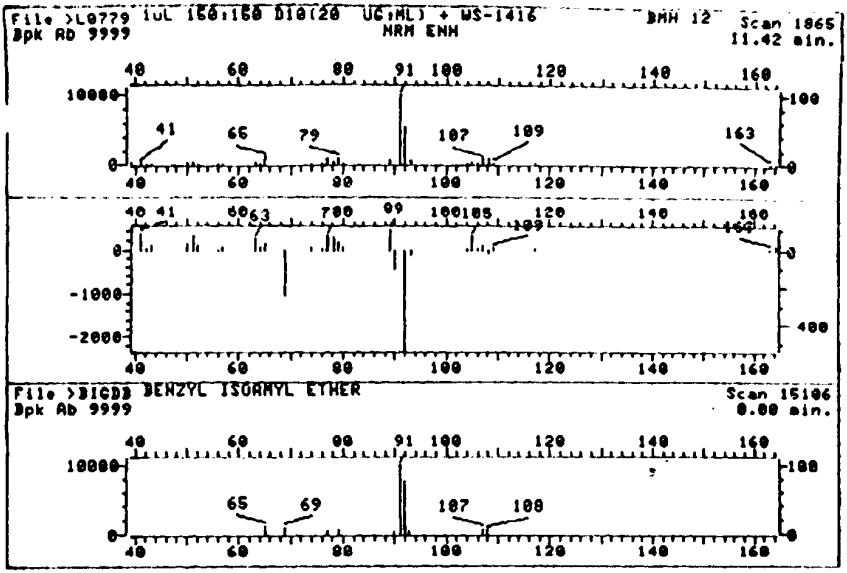
S

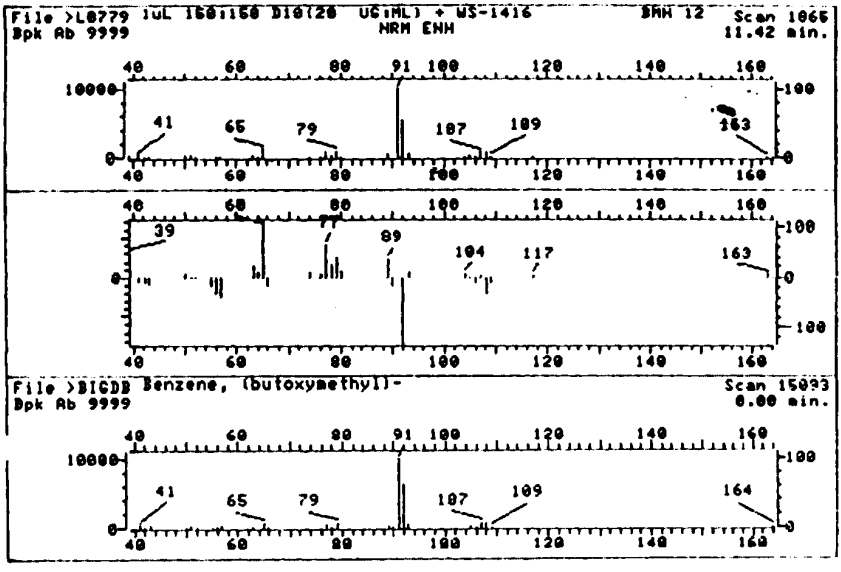
1.	BENZYL ISOAMYL ETHER	178 C12H18O
2.	Benzene, (butoxymethyl)-	164 C11H16O
3.	Benzene, (propoxymethyl)-	150 C10H14O
4.	Benzene, (butoxymethyl)-	164 C11H16O
5.	Benzene, methyl-	92 C7H8
6.	Benzene, methyl-	92 C7H8
7.	Benzene, methyl-	92 C7H8
8.	Benzene, methyl-	92 C7H8
9.	Benzene, methyl-	92 C7H8
10.	1,3,5-Cycloheptatriene	92 C7H8

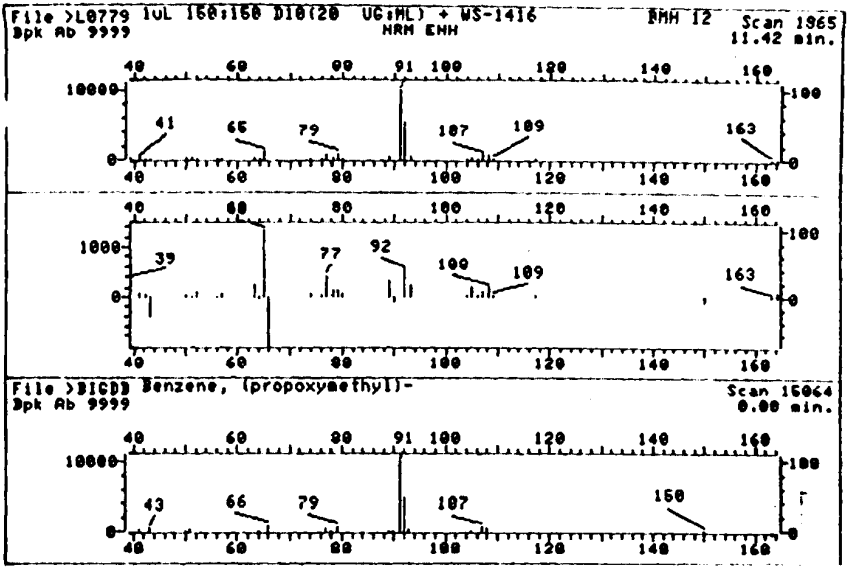
Sample file: >L0779 Spectrum #: 1865
Search speed: 2 Tilting option: N No. of ion ranges searched: 49

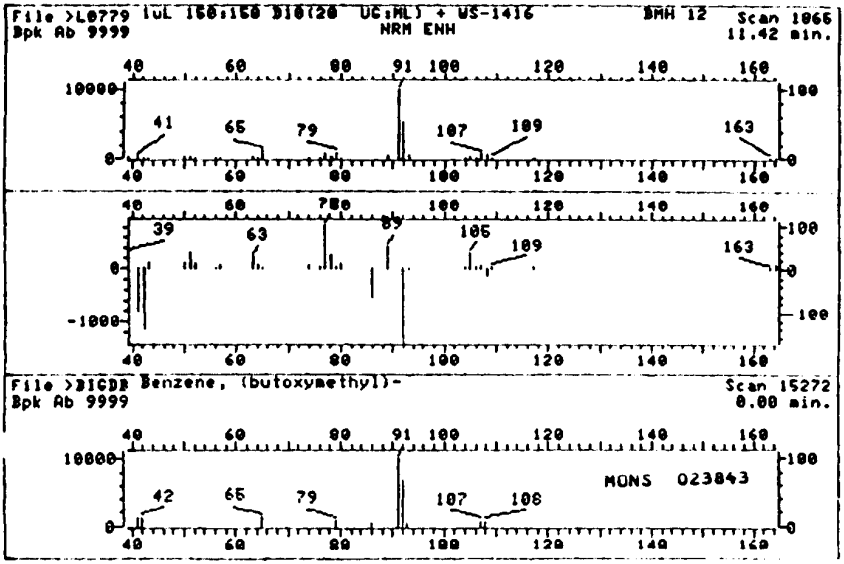
	Prob.	CAS #	CON #	ROOT	K	DK	#FLG	TILT	%	CON	C_I	R_IV
1.	78	0	15106	*BIGDB	44	19	2	0	68	4	55	14
2.	76*	588670	15093	*BIGDB	46	35	1	0	79	7	45	29
3.	70	937611	15064	*BIGDB	37	44	2	0	100	10	42	12
4.	70	588670	15272	*BIGDB	45	17	2	0	77	7	42	15
5.	52*	108883	15228	*BIGDB	30	48	2	0	84	16	20	15
6.	52*	108883	15230	*BIGDB	32	52	2	0	80	16	20	16
7.	52*	108883	15010	*BIGDB	33	57	2	0	72	16	20	16
8.	52*	108883	15229	*BIGDB	34	61	3	0	66	16	20	13
9.	52*	108883	15231	*BIGDB	34	62	2	0	71	16	20	14
10.	52*	544252	15235	*BIGDB	28	57	2	0	75	19	20	14

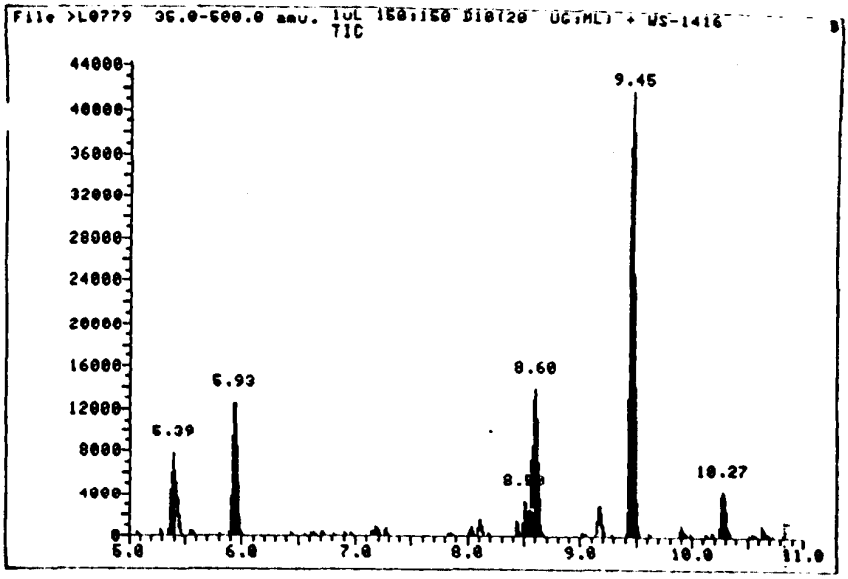
MONS 023841


File >L0779 1uL 150:150 D10(20 UC:ML) + WS-1416
Bpk Ab 9999
NRM ENH
BHH 12
Scan 1865
11.42 min.
File >BIGDB BENZYL ISOAMYL ETHER
Bpk Ab 9999
Scan 15106
0.00 min.


File >L0779 1uL 150:150 D10(20 UC:ML) + WS-1416
Bpk Ab 9999
NRM ENH
BHH 12
Scan 1865
11.42 min.
File >BIGDB Benzene, (butoxymethyl)-
Bpk Ab 9999
Scan 15093
0.00 min.


File >L0779 1uL 150:150 D10(20 UG:ML) + WS-1416 BMH 12 Scan 1965
Bpk Ab 9999 NRM ENH 11.42 min.
41 65 79 107 109 163
39 77 92 100 109 163
File >BIGDB Benzene, (propoxymethyl)- Scan 16064
Bpk Ab 9999 0.00 min.
43 66 79 107 150


File >L0779 1uL 150:150 D10(20 UG:ML) + WS-1416 BMH 12 Scan 1966
Bpk Ab 9999 NRM ENH 11.42 min.
41 65 79 107 109 163
39 63 89 105 109 163
File >BIGDB Benzene, (butoxymethyl)- Scan 15272
Bpk Ab 9999 0.00 min.
MONS 023843
42 65 79 107 108



Peak at 5.39 - Cl. phenol-d_4 surrogate

" " 5.93 - Cl_2 benzene-d_4 surrogate

Peak at (8.50+8.60) - dichlorocresyl benzene isomer

$$\frac{35,393}{3511} = 10.1\ \mu g/ml$$

$$= 10\ \mu g/Liter$$

Peak at 9.45 min - tetrahydronaphthalene

$$\frac{41532}{3511} = 11.8\ \mu g/ml$$

$$= 12\ \mu g/Liter$$

Peak at 10.27 min - unknown

$$\frac{8041}{3511} = 2.3\ \mu g/ml$$

$$= 2.4\ \mu g/liter$$

MONS 023844

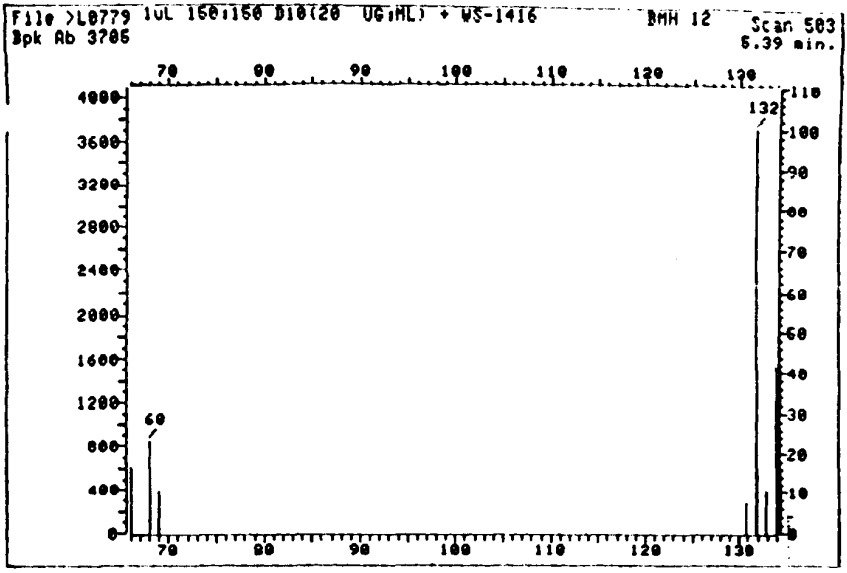
)L0779 1uL 150:150 D10(20 UG:ML) + WS-1416 BMH 12:11:85

35.0: 500.0 TIC

Peak #	R.T. min.	first scan	max scan	last scan	peak height	raw area	corr. area	corr. % max.	% of total
1	5.391	493	503	506	7722	14003	14003	15.12	7.307
2	5.931	614	625	641	12442	28421	28421	30.69	14.831
3	8.504	1198	1206	1214	3294	6972	6972	7.53	3.638
4	8.597	1214	1227	1238	13851	41552	41552	44.87	21.684
5	9.447	1409	1419	1431	42030	92600	92600	100.00	48.323
6	10.266	1596	1604	1617	4231	8081	8081	8.73	4.217

Sum of corrected areas: 191629.

MONS 023845



>L0779 1uL 150:150 D10(20 UG:ML) + WS-1416 BMH 12:11:85

503

File: >L0779 Scan #: 503 Retn. time: 5.39

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
65.95	608.00	68.95	367.00	131.95	3705.00	132.95	384.00	133.95	1538.00
68.05	851.00	130.00	269.00						

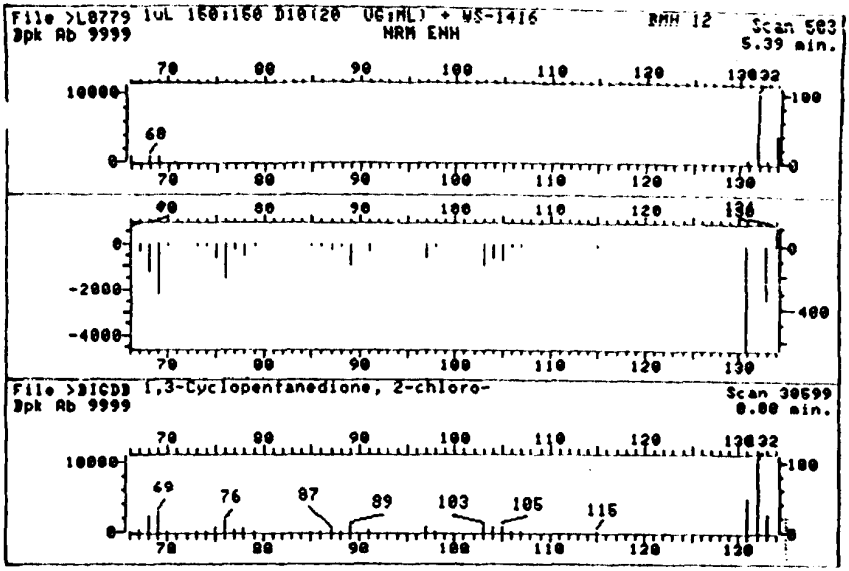
S

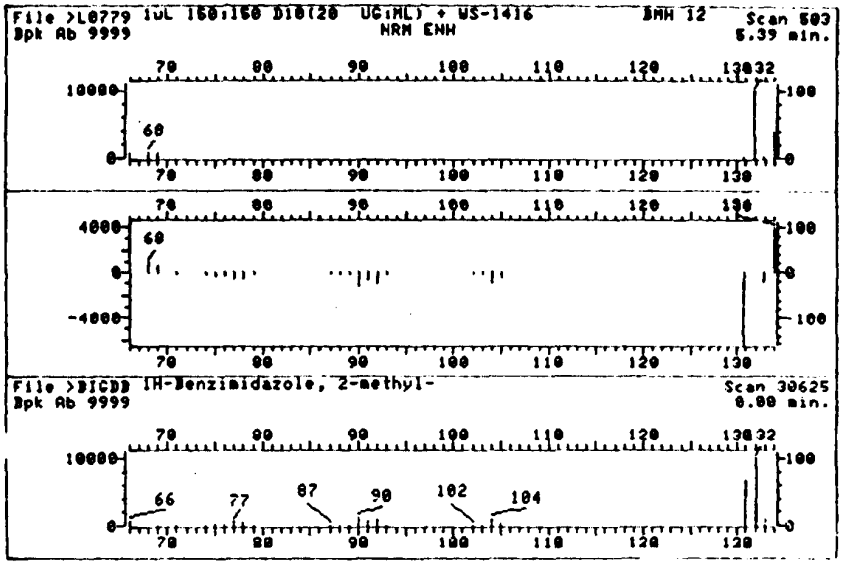
1. 1,3-Cyclopentanedione, 2-chloro- 132 C5H5ClO2

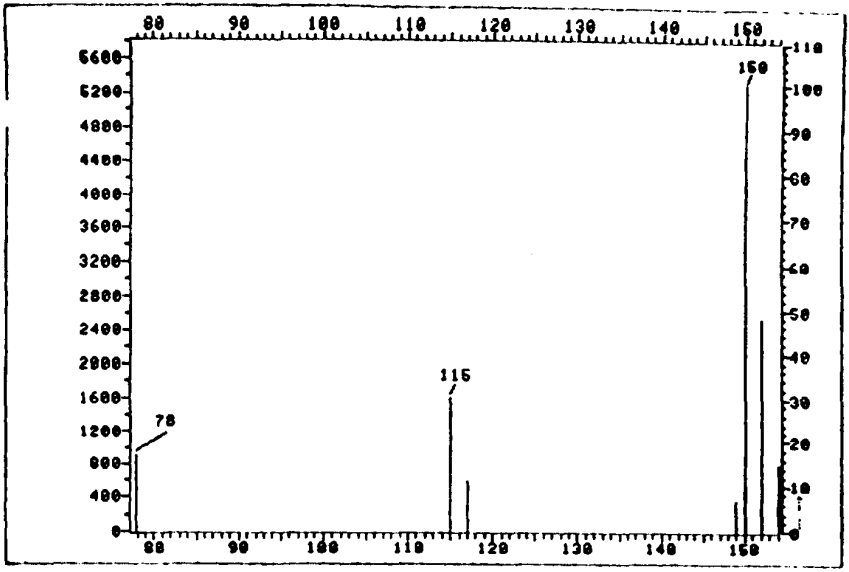
Sample file:)L0779 Spectrum #: 503
Search speed: 2 Tilting option: N No. of ion ranges searched: 73

	Prob.	CAS #	CON #	ROOT	K	DK	#FLG	TILT	%	CON	C_I	R_IV
1.	60*	14203191	30599	*BIGDB	25	86	3	0	100	15	30	13

MONS 023847


File >L0779 1uL 150:150 D10(20 UG:ML) + WS-1416
Bpk Ab 9999
NRM ENH
BMH 12
Scan 503
5.39 min.
File >DICDB 1,3-Cyclopentanedione, 2-chloro-
Bpk Ab 9999
Scan 30699
0.00 min.


File >L0779 1uL 150:150 D10(20 UG:ML) + WS-1416
Bpk Ab 9999
NRM ENH
BMH 12
Scan 503
5.39 min.
File >DICDB 1H-Benzimidazole, 2-methyl-
Bpk Ab 9999
Scan 30625
0.00 min.



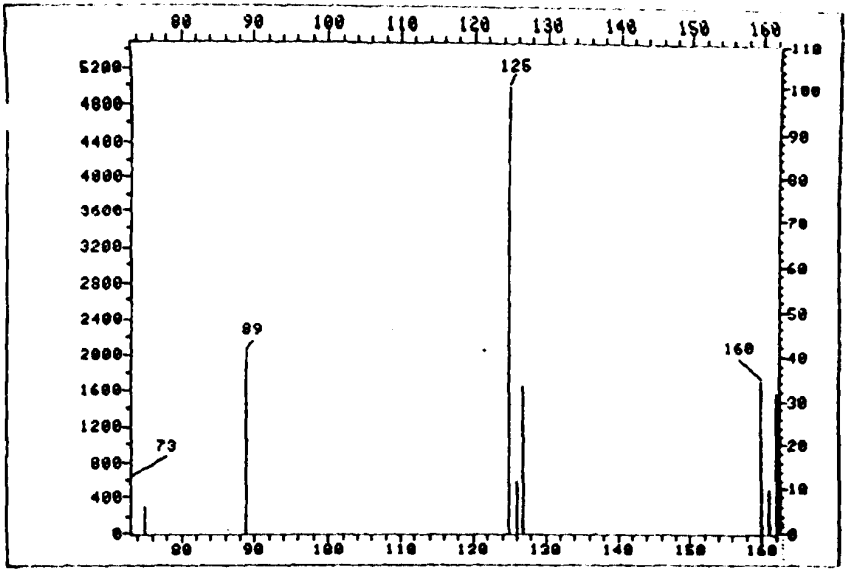
>L0779 1uL 150:150 D10(20 UG:ML) + WS-1416 BMH 12:11:85

625

File: >L0779 Scan #: 625 Retn. time: 5.93

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
77.05	361.00	114.95	1604.00	148.90	357.00	152.00	2555.00	154.00	794.00
77.95	904.00	116.95	587.00	150.00	5280.00				

MONS 023849



>L0779 1uL 150:150 D10(20 UG:ML) + WS-1416 BMH 12:11:85

1227

File: >L0779 Scan #: 1227 Retn. time: 8.60

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
72.95	605.00	88.95	2016.00	125.95	567.00	159.90	1701.00	162.00	1575.00
74.95	296.00	124.95	4973.00	126.95	1641.00	161.00	477.00		

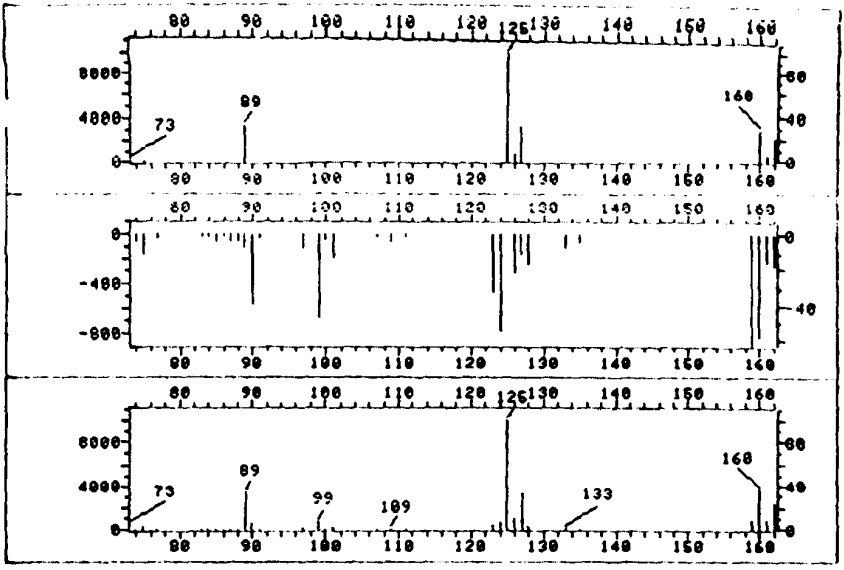
S

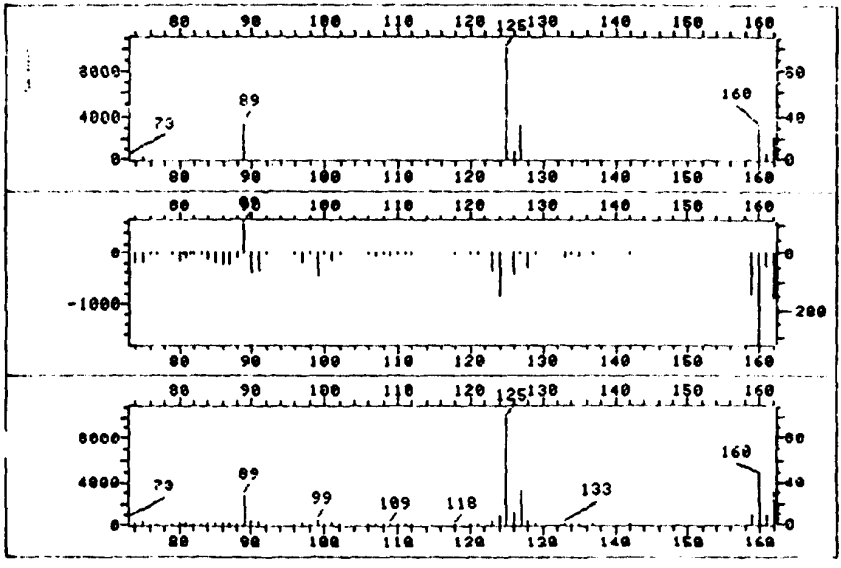
1.	Benzene, 2,4-dichloro-1-methyl-	160 C7H6Cl2
2.	Benzene, 1,3-dichloro-2-methyl-	160 C7H6Cl2
3.	2,3-DICHLOROTOLUENE	160 C7H6Cl2

Sample file: >L0779 Spectrum #: 1227
Search speed: 2 Tilting option: N No. of ion ranges searched: 82

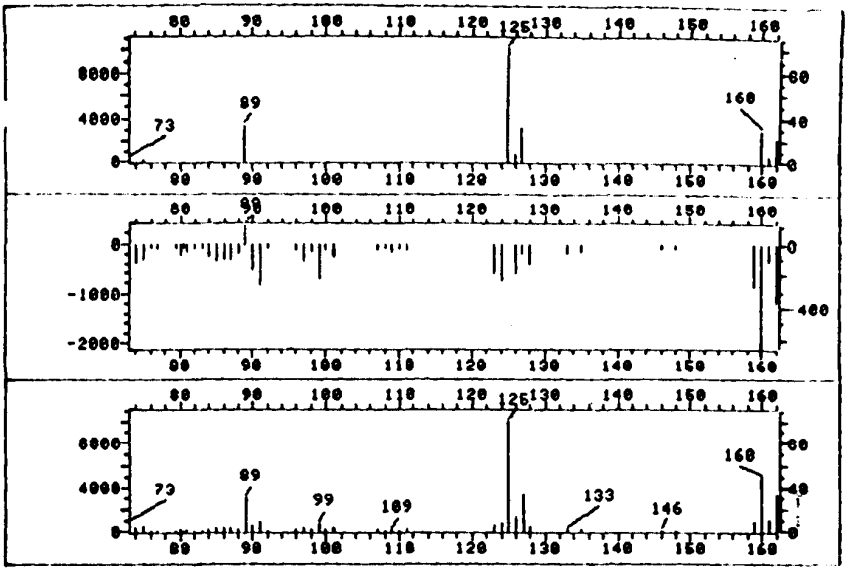
	Prob.	CAS #	CON #	ROOT	K	DK	#FLG	TILT	X	CON	C_I	R_IV
1.	86%	95738	28643	*BIGDB	58	50	2	0	92	5	60	35
2.	67%	118694	28396	*BIGDB	49	62	2	0	73	14	34	22
3.	58%	0	28405	*BIGDB	51	62	2	0	69	17	25	25

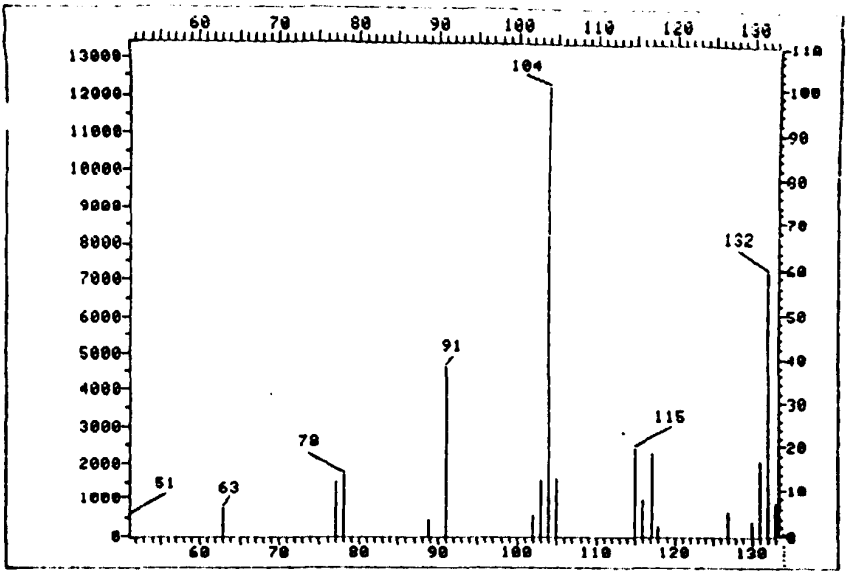
MONS 023851


80 90 100 110 120 125 130 140 150 160
8000
4000
0
73
89
160
80
40
0
-400
-800
99
109
133


80 90 100 110 120 125 130 140 150 160
8000
4000
0
73
89
160
50
40
-1000
200
99
109
118
133

MONS 023852


73
89
99
109
125
133
146
160
MONS 023853



>L0779 1uL 150:150 D10(20 UG:ML) + WS-1416 BMH 12:11:85

1419

File: >L0779 Scan #: 1419 Retn. time: 9.45

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
51.05	483.0	88.95	447.0	104.05	12169.0	117.05	2254.0	131.05	2025.0
62.95	703.0	91.05	4584.0	105.05	1565.0	117.95	256.0	132.05	7211.0
77.05	1469.0	102.05	554.0	115.05	2358.0	126.95	650.0	132.95	850.0
78.05	1630.0	103.05	1513.0	116.05	937.0	130.05	372.0		

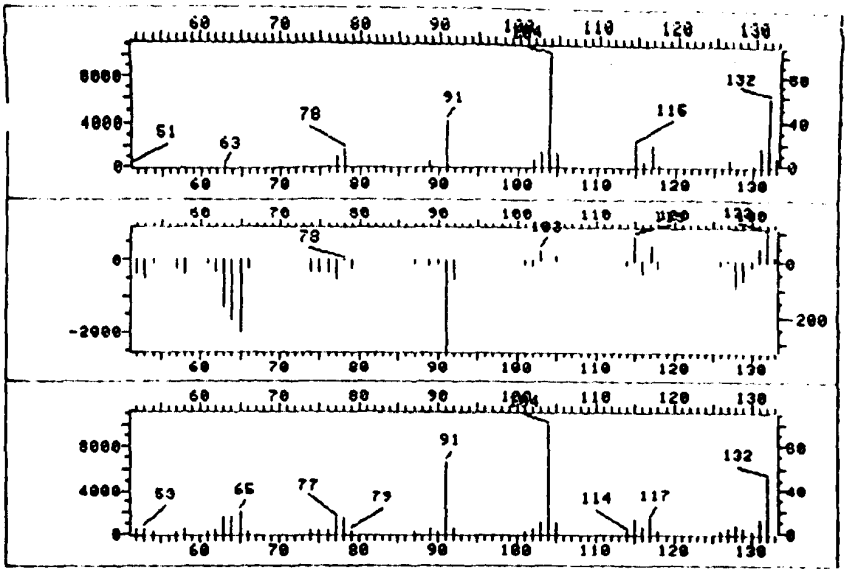
S

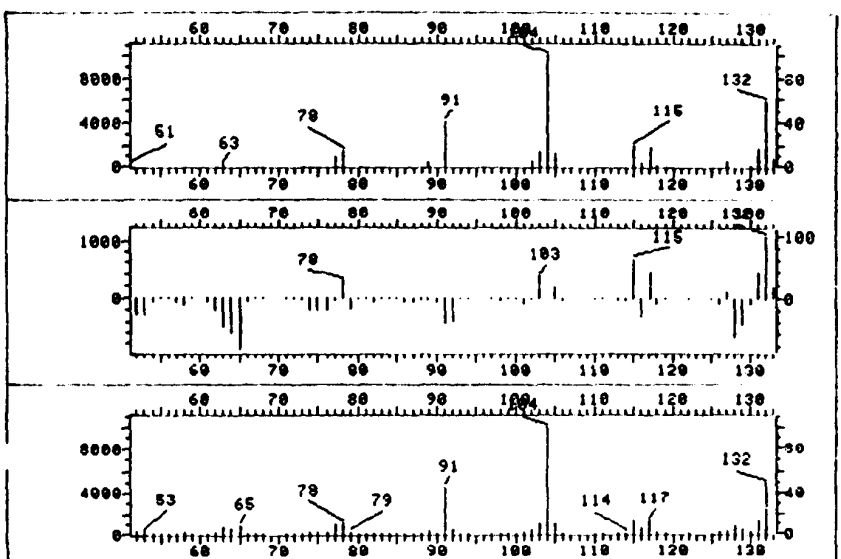
1.	Naphthalene, 1,2,3,4-tetrahydro-	132 C10H12
2.	Naphthalene, 1,2,3,4-tetrahydro-	132 C10H12
3.	1,3,5,7-Cyclooctatetraene-1-carboxaldehyde	132 C9H8O
4.	CYCLOPROPANE, BENZYL-	132 C10H12

Sample file:)L0779 Spectrum #: 1419
Search speed: 2 Tilting option: N No. of ion ranges searched: 61

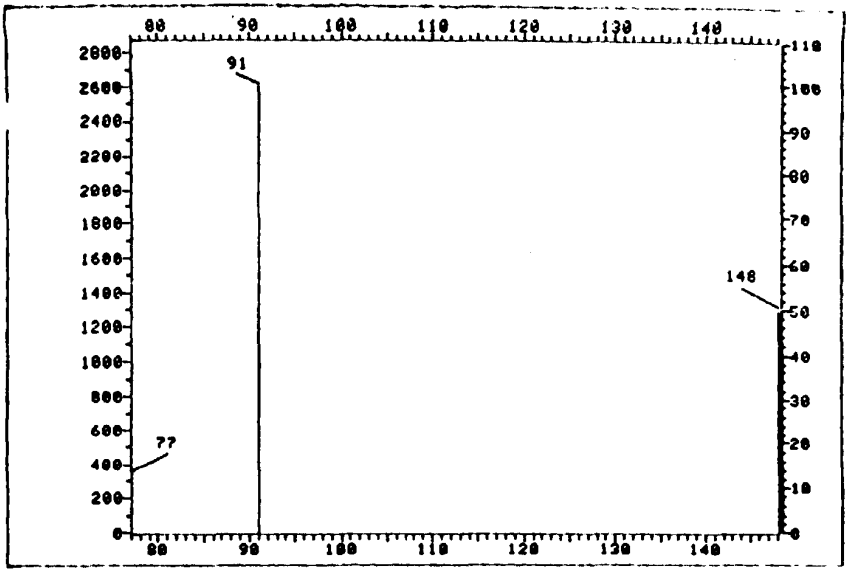
	Prob.	CAS #	CON #	ROOT	K	DK	#FLG	TILT	%	CON	C_I	R_IV
1.	86*	119642	20606	*BIGDB	68	40	3	0	100	2	60	35
2.	81*	119642	20282	*BIGDB	69	38	2	0	85	7	53	49
3.	52*	30844123	19978	*BIGDB	27	82	3	0	155	20	20	13
4.	52*	0	20284	*BIGDB	44	43	3	0	191	16	20	18

MONS 023855


8000
4000
0
51
63
78
91
116
132
-2000
200
53
65
77
79
114
117


8000
4000
0
51
63
78
91
103
115
132
1000
100
53
65
79
114
117

MONS 023856



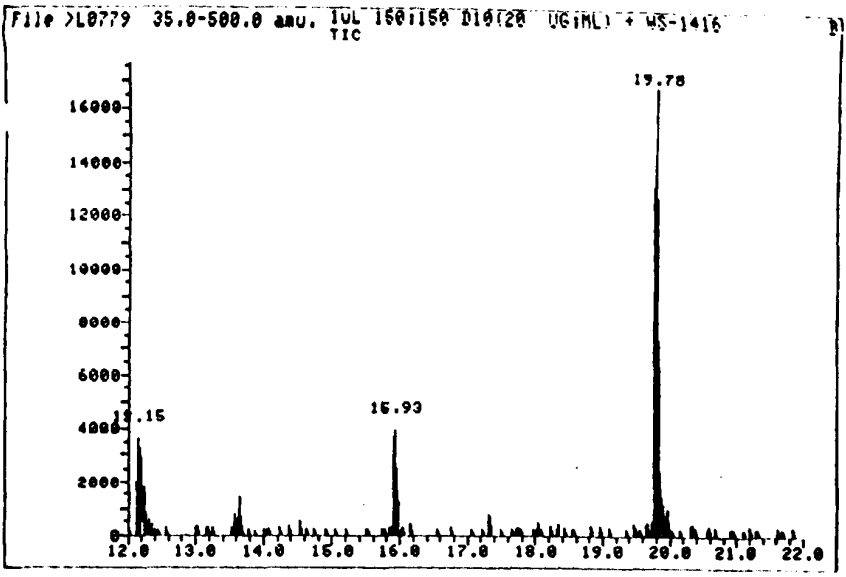
>L0779 1uL 150:150 D10(20 UG:ML) + WS-1416 BMH 12:11:85

1604

File: >L0779 Scan #: 1604 Retn. time: 10.27

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
76.95	328.00	91.05	2606.00	148.15	1297.00				

MONS 023857



Peak at 12.15 into Chloroacetyl phenol isomer

$\frac{6234}{3571} = 1.8 \mu g/ml$

$= 1.8 \mu g/L$

Peak at 15.93 - unknown

$2.30 \mu g/ml$

$= 2.4 \mu g/liter$

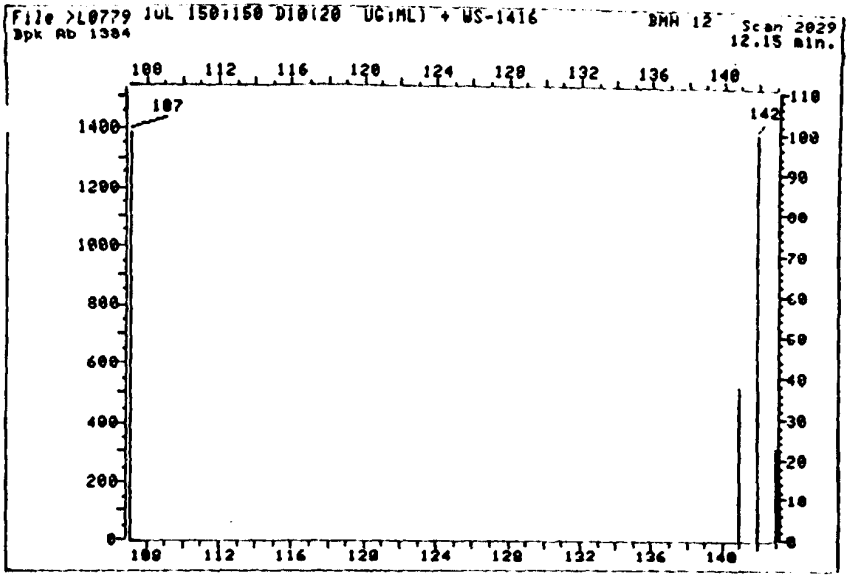
Peak at 19.78 - anthracene-d10 internal standard

>L0779 1uL 150:150 D10(20 UG:ML) + WS-1416 BMH 12:11:85

35.01 500.0 TIC

Peak #	R.T. min.	first scan	max scan	last scan	peak height	raw area	corr. area	corr. % max.	% of total
1	12.149	2021	2029	2033	3595	6234	6234	13.92	10.550
2	15.929	2873	2883	2891	3965	8075	8075	18.03	13.665
3	19.780	3742	3753	3770	16724	44782	44782	100.00	75.785

Sum of corrected areas: 59091.



>L0779 1uL 150:150 D10(20 UG:ML) + WS-1416 BMH 12:11:85

2029

File: >L0779 Scan #: 2029 Retn. time: 12.15

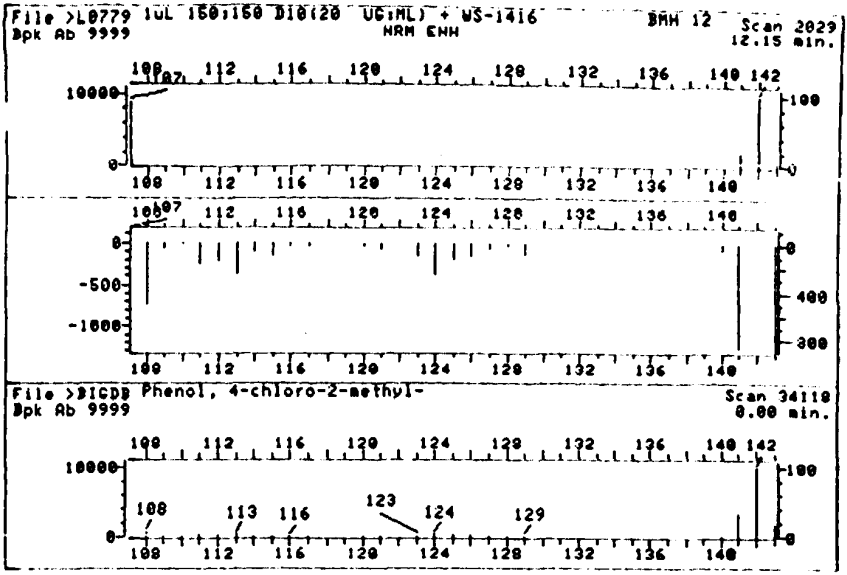
m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
107.05	1384.00	141.00	521.00	142.00	1378.00	143.00	312.00		

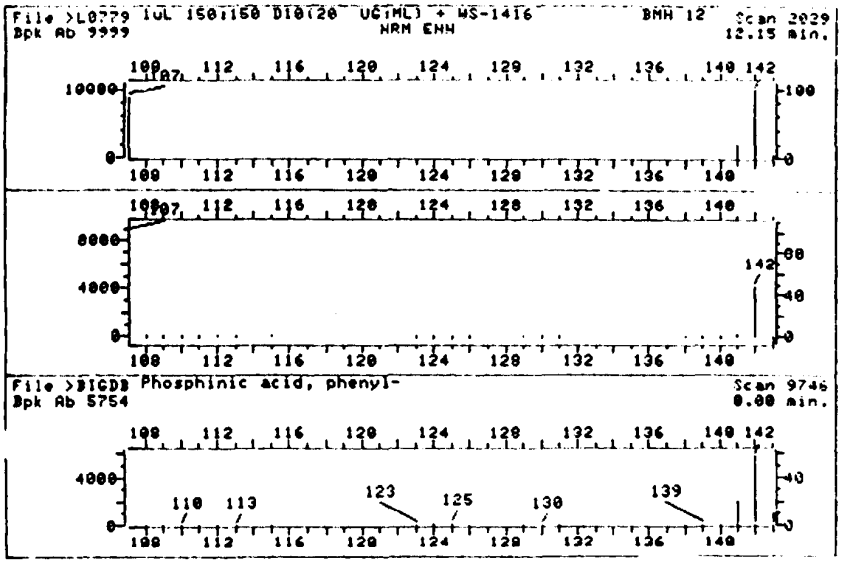
5

1. Phenol, 4-chloro-2-methyl- 142 C7H7ClO

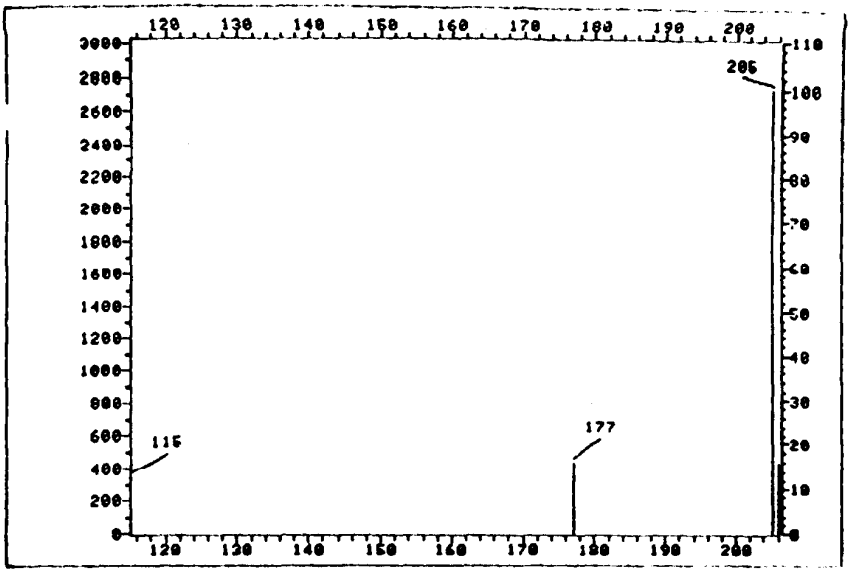
Sample file: >L0779 Spectrum #: 2029
Search speed: 2 Tilting option: N No. of ion ranges searched: 111

	Prob.	CAS #	CON #	ROOT	K	DK	#FLG	TILT	%	CON	C_I	R_IV
1.	70*	1570645	34118	*BIGDB	36	73	3	0	99	6	42	13


File >L0779 1uL 150:150 D10(20 UG:ML) + WS-1416
Bpk Ab 9999
NRM ENH
BMH 12
Scan 2029
12.15 min.
File >BIGDB Phenol, 4-chloro-2-methyl-
Bpk Ab 9999
Scan 34118
0.00 min.


File >L0779 1uL 150:150 D10(20 UG:ML) + WS-1416
Bpk Ab 9999
NRM ENH
BMH 12
Scan 2029
12.15 min.
File >BIGDB Phosphinic acid, phenyl-
Bpk Ab 5754
Scan 9746
0.00 min.

MONS 023862

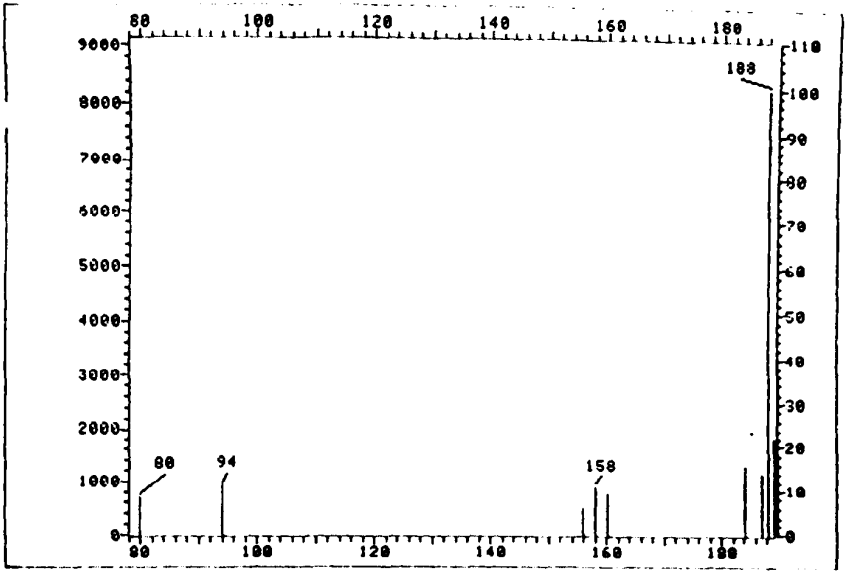


>L0779 1uL 150:150 D10(20 UG:ML) + WS-1416 BMH 12:11:85

2883

File: >L0779 Scan #: 2883 Retn. time: 15.93

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
114.95	350.00	177.00	441.00	205.15	2745.00	206.00	429.00		

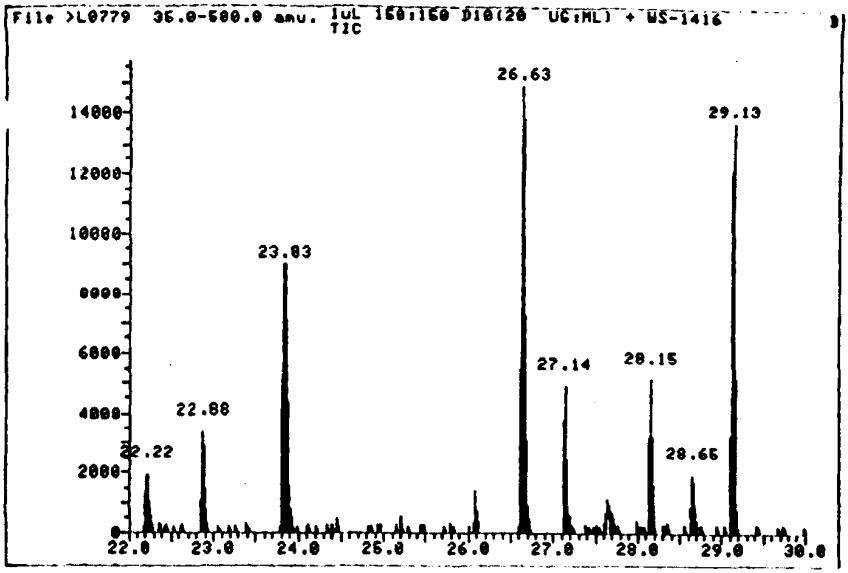


>L0779 1uL 150:150 D10(20 UG:ML) + WS-1416 BMH 12:11:85

3753

File: >L0779 Scan #: 3753 Retn. time: 19.78

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
77.95	370.00	94.05	942.00	158.15	907.00	184.15	1294.00	188.15	8290.00
80.05	708.00	156.00	494.00	160.15	773.00	187.15	1140.00	189.15	1806.00



RT = 22.22 phthalate
4213/contact = 1.78

RT = 22.88 unknown
5030/contact = 1.65

RT = 23.83 pyrene-d10 surrogate

RT = 26.63 29529/contact = 9.67 methoxy, dimethyl benzenebutanol isomer

RT = 27.14 unknown
6486/contact = 2.12

RT = 28.15 unknown
8385/contact = 2.75

MONS 023865

RT = 28.65 unknown
2765/contact = 0.905

RT = 29.13 unknown
23,357/contact = 7.65

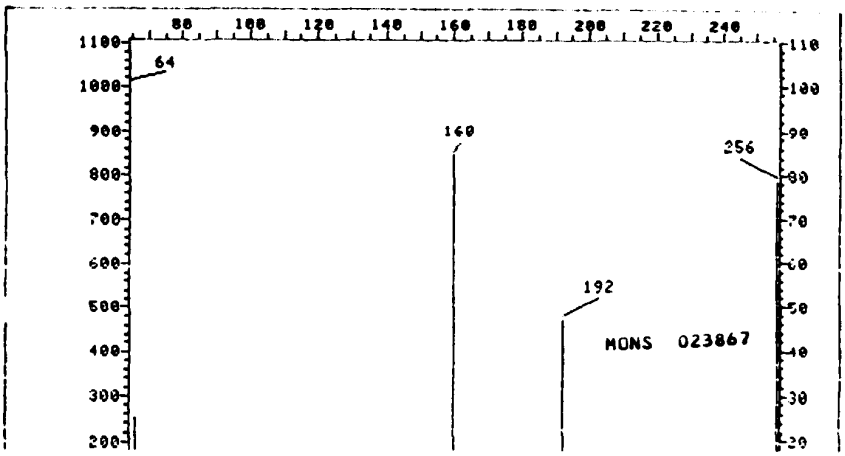
>L0779 1uL 150:150 D10(20 UG:ML) + WS-1416 BMH 12:11:85

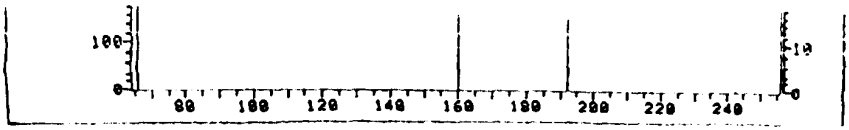
35.01 500.0 TIC

Peak #	R.T. min.	first scan	max scan	last scan	peak height	raw area	corr. area	corr. % max.	% of total
1	22.220	4293	4304	4309	1905	4213	4213	14.27	4.181
2	22.875	4443	4452	4461	3367	5030	5030	17.03	4.991
3	23.832	4657	4668	4684	8988	21005	21005	71.13	20.844
4	26.634	5289	5301	5312	14910	29529	29529	100.00	29.302
5	27.138	5408	5415	5419	4906	6486	6486	21.96	6.436
6	28.148	5636	5643	5652	5150	8389	8389	28.41	8.325
7	28.648	5748	5756	5759	1901	2765	2765	9.36	2.744
8	29.131	5856	5865	5875	13738	23357	23357	79.10	23.178

Sum of corrected areas: 100774.

MONS 023866


88
100
120
140
160
180
200
220
240
1100
1000
900
800
700
600
500
400
300
200
110
100
90
80
70
60
50
40
30
20
64
160
256
192
MONS 023867

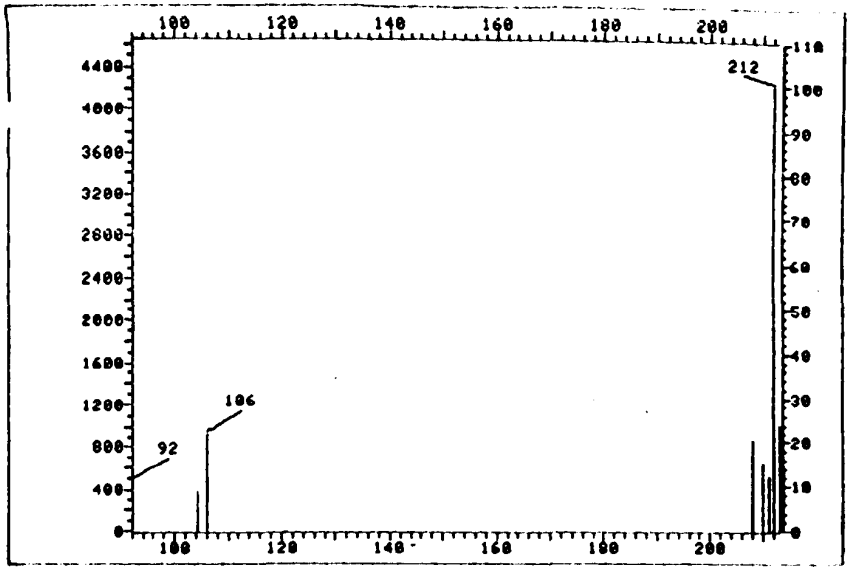


>L0779 10L 150:150 D10(20 UG:ML) + WS-1416 BMH 12:11:85

4452

File: >L0779 Scan #: 4452 Retn. time: 22.88

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
63.95	1004.00	65.95	250.00	159.90	849.00	191.90	472.00	255.75	792.00



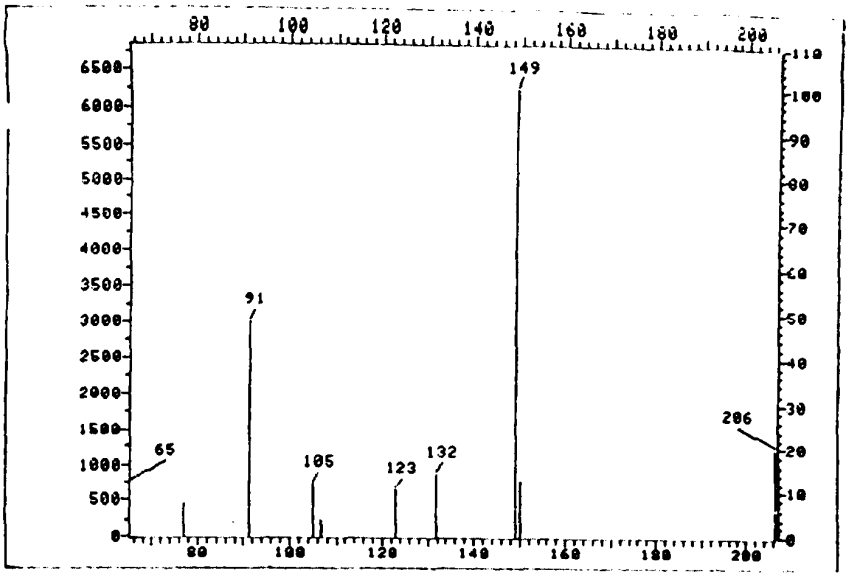
>L0779 1uL 150:150 D10(20 UG:ML) + WS-1416 BMH 12:11:85

4668

File: >L0779 Scan #: 4668 Retn. time: 23.83

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
91.95	465.00	106.85	920.00	210.15	638.00	212.15	4214.00	213.15	1086.00
104.30	363.00	208.15	863.00	211.15	519.00				

MONS 023869



>L0779 1uL 150:150 D10(20 UG:ML) + WS-1416 BMH 12:11:85

5301

File: >L0779 Scan #: 5301 Retn. time: 26.63

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
64.95	737.00	91.05	3004.00	106.80	227.00	131.95	873.00	150.00	776.00
76.95	435.00	104.95	732.00	123.05	646.00	149.00	6235.00	206.15	1245.00

MONS 023870

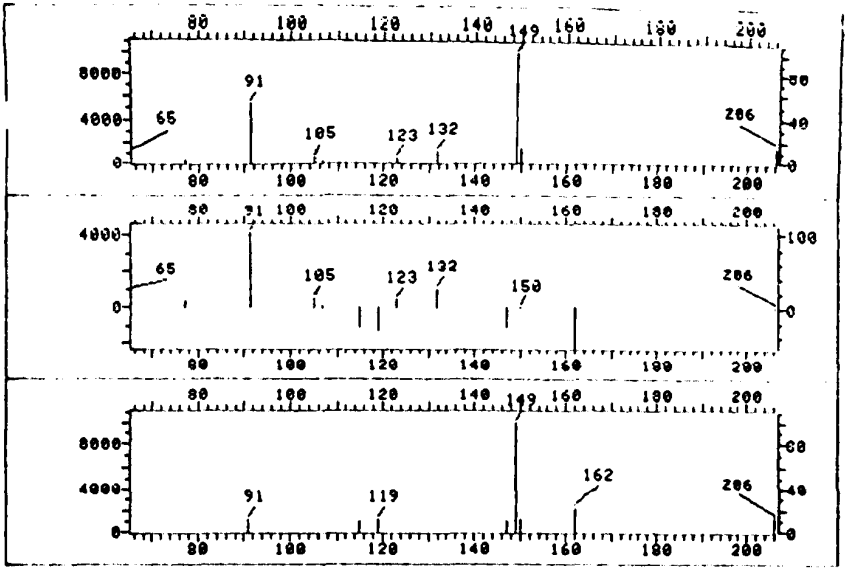
S

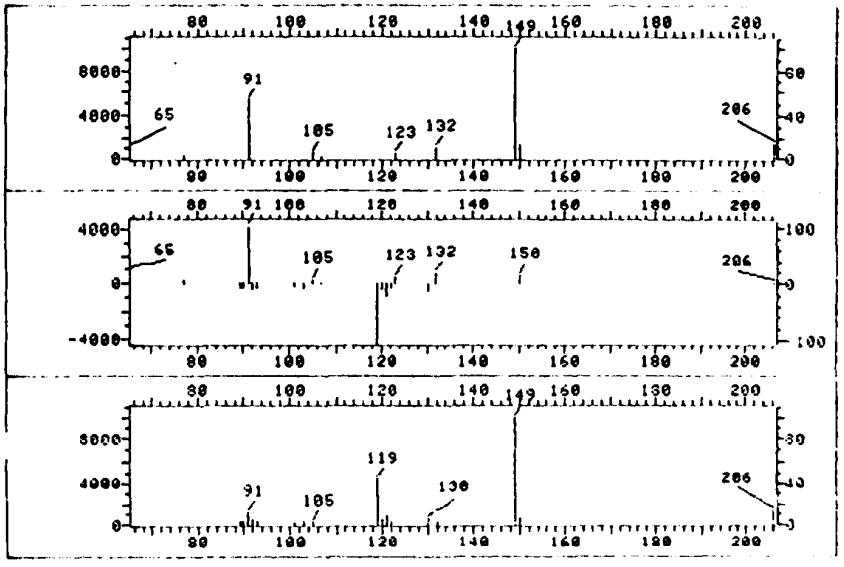
1. Benzenebutanal, 2-methoxy-.gamma.,4-dimethyl- 206 C13H18O2

Sample file:)L0779 Spectrum #: 5301
Search speed: 2 Tilting option: N No. of ion ranges searched: 75

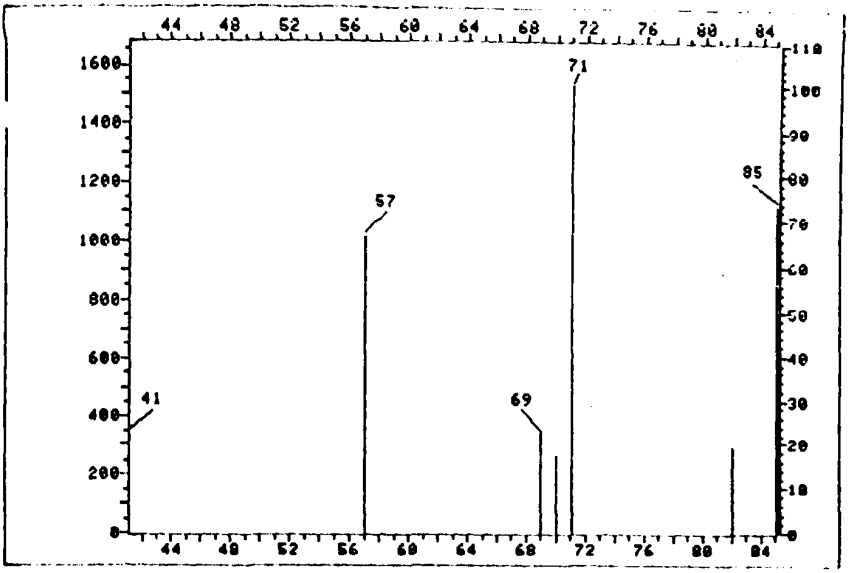
	Prob.	CAS #	CON #	ROOT	K	DK	#FLG	TILT	%	CON	C_I	R_IV
1. →	27*	63646902	36250	"BIGDB	25	39	3	0	100	40	10	13

MONS 023871


80 100 120 140 149 160 180 200
8000
4000
0
91
65
105
123
132
206
80 91 100 120 140 160 180 200
4000
0
65
105
123
132
150
206
100
80 100 120 140 149 160 180 200
8000
4000
0
91
119
162
206
60
40


80 100 120 140 149 160 180 200
8000
4000
0
91
65
105
123
132
206
60
40
80 91 100 120 140 160 180 200
4000
0
-4000
65
105
123
132
150
206
100
80 100 120 140 149 160 180 200
8000
4000
0
119
91
105
130
206
80
40

MONS 023872



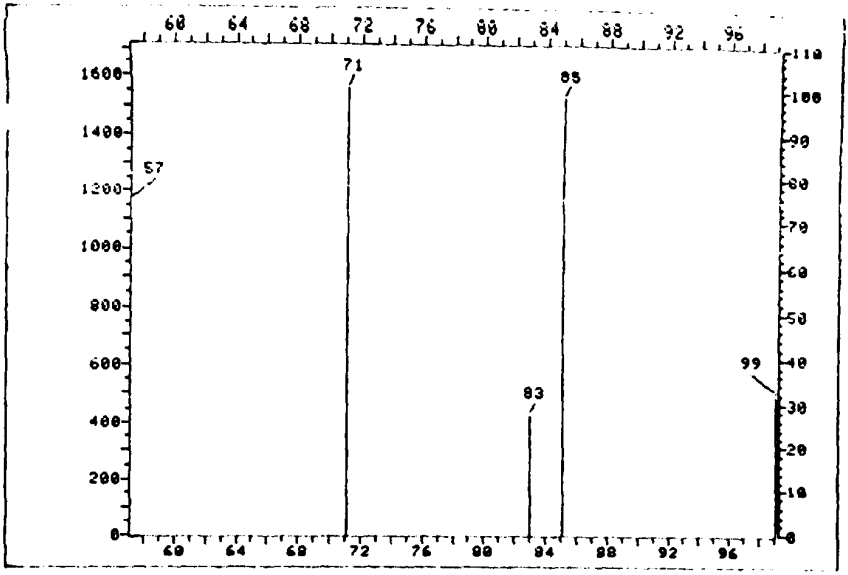
>L0779 1uL 150:150 D10(20 UG:ML) + WS-1416 BMH 12:11:85

5415

File: >L0779 Scan #: 5415 Retn. time: 27.14

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
41.050	342.00	68.950	343.00	71.050	1525.00	82.050	290.00	85.050	1123.00
57.050	1018.00	70.050	265.00						

MONS 023873

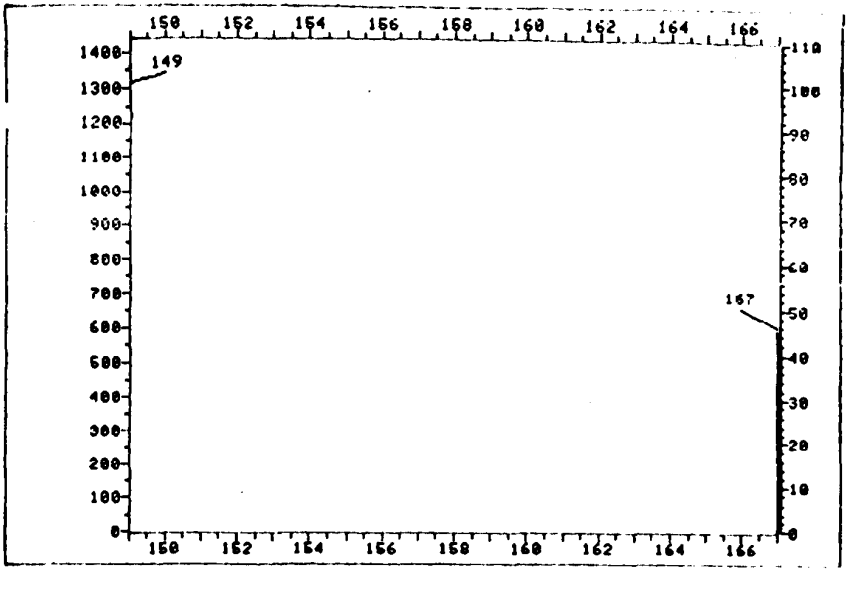


)L0779 1uL 150:150 D10(20 UG:ML) + WS-1416 BMH 12:11:85

5643

File:)L0779 Scan #: 5643 Retn. time: 28.15

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
57.050	1159.00	71.050	1554.00	82.950	422.00	85.050	1522.00	99.050	493.00

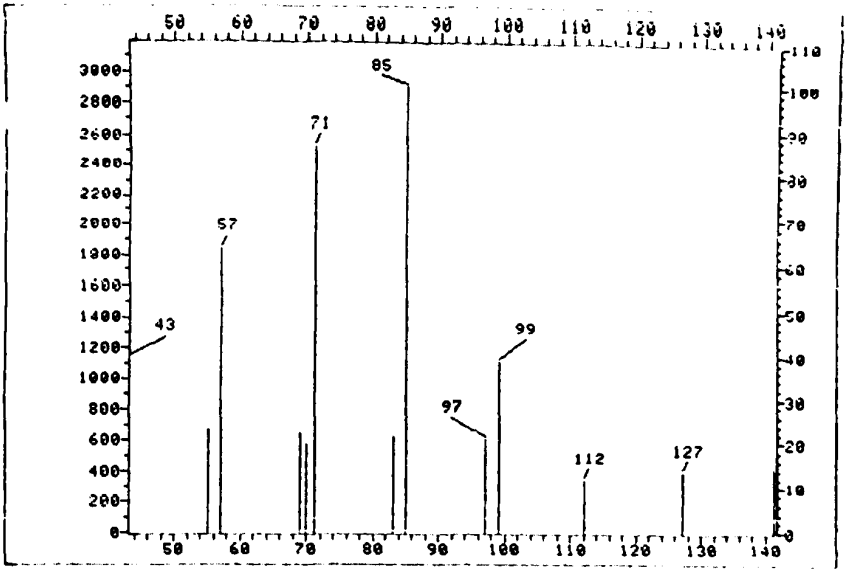


>L0779 1uL 150:150 D10(20 UG:ML) + WS-1416 BMH 12:11:85

5756

File: >L0779 Scan #: 5756 Retn. time: 28.65

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
149.00	1308.00	167.00	593.00						



>L0779 10L 150:150 D10(20 UG:ML) + WS-1416 BMH 12:11 85

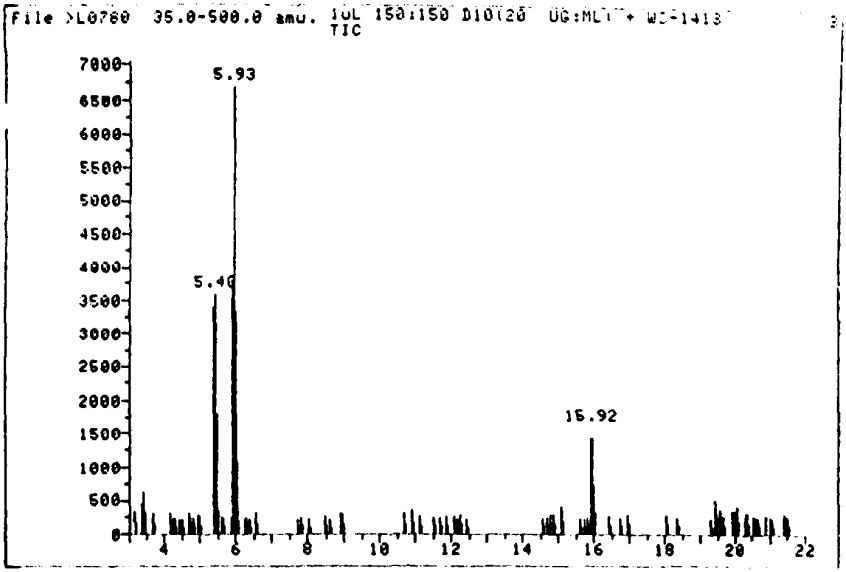
5865

File: >L0779 Scan #: 5865 Retn. time: 29.13

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
43.05	1128.00	69.05	643.00	83.05	620.00	99.05	1111.00	127.05	394.00
55.05	671.00	70.05	568.00	85.05	2889.00	112.05	344.00	141.15	419.00
57.05	1842.00	71.05	2510.00	96.95	599.00				

MONS 023876

Figure 4. Total ion chromatogram obtained from the analysis of the WS-1418 extract of the Method Blank.



S

reg	type	# pts	scan#	range: amu\r.t.	base	file	ion range
X	GC	10583	1	3.17- 50.03	6691.0	>L0780	35.00- 500.00
Adjusting detection and area thresholds:					.4	669.1	
Adjusting detection and area thresholds:					.8	1338.2	

>L0780 1uL 150:150 D10(20 UG:ML) + WS-1418 BMH 12:11:85

35.01 500.0 TIC

Peak #	R.T. min.	first scan	max scan	last scan	peak height	raw area	corr. area	corr. % max.	% of total
1	5.398	495	505	506	3604	5779	5779	39.31	7.642
2	5.929	615	625	638	6691	14701	14701	100.00	19.339
3	15.921	2876	2882	2887	1444	2320	2320	15.78	3.054
4	33.890	6937	6941	6945	551	885	885	6.02	1.164
5	34.125	6992	6994	6998	644	813	813	5.53	1.069
6	34.480	7069	7074	7076	623	888	888	6.04	1.168
7	34.701	7121	7124	7127	865	951	951	6.47	1.251

15.92 unknown

$\frac{2320}{3511} = 0.67$

MONS 023878

>L0777 5-6 → >L0778

pinit d5	5510	611	(11%)
Ci-pinit-d4	9705	9246	(95%)
"	2982	2469	
Ci-bergi-d4	18544	9790	(53%)
	—	anthra-d10	23,463
	—	pyrene-d10	7915

>L0779

2199	(40%)
11667	(120%)
4395	
14059	(76%)
30,694	
12597	

>L0780

—	
6770	(70%)
2253	
7832	(42%)
—	(~~[illegible]~~)
—	

MONS 023879

The following pages describe Analytical Request Information generated for this project.

Time Stamp - <851218.0919>

MONSANTO COMPANY/ENVIRONMENTAL SCIENCES CENTER

ANALYSIS REQUEST

DATE: 12/04/85 LOG NO: U-85-12-04-01

REQUESTER: JAMERO, DELAWARE RIVER PROJECT NO: 760.18 - 538

REPORT RESULTS TO: FILES/MEES, HUGHES, MCKENZIE, SHERMAN, TRANG

ANALYSIS REQUESTS SENT TO: FILES, SHERMAN, HUGHES, MCKENZIE, TRANG

SAMPLE PICKUP BY: MCKENZIE

SAMPLE LOCATION: U-414 REFRIGERATOR

SAMPLE DESCRIPTION FOR 4 SAMPLES

SAMPLE IDENTIFICATION	NUMBER OF BOTTLES	ESC NUMBER(S)	a
SAMPLE 3S (RUN TOTAL PCBS ON WHOLE SAMPLE)	1 X 1 LITER	WS-1415	(910 mL)
SAMPLE 4S (RUN TOTAL PCBS-WATCH OUT HIGH TOC AND PCBS)	1 X 1/2 LITER	WS-1417	(345 mL)
SAMPLE 23S-A (RUN BASE-NEUTRAL SCAN)	1 X 1 LITER	WS-1414	(870 mL)
SAMPLE 21-S (RUN TOTAL PCBS AND BASE-NEUTRAL SCAN)	1 X 1 LITER	WS-1416	(970 mL)
BLANK B&J WATER	1 LITER	WS-1418	(1000 mL)
BLANK B&J WATER (DUPLICATE)	1 LITER	WS-1410	(1000 mL)
BLANK (HOUSE DEIONIZED)	1 LITER	WS-1411	(1000 mL)
LOW SPIKED B&J WATER	1 LITER	WS-1412	(1000 mL)
HIGH SPIKED B&J WATER	1 LITER	WS-1413	(1000 mL)

a - VOLUME LISTED IN PARENTHESIS IS VOLUME OF SAMPLE EXTRACTED)

RECORDED BY: MCKENZIE

REQUIRED ANALYSES: PCB AND BASE NEUTRAL ON SELECT SAMPLES (INDICATED ON BOTTLES)

ANALYTICAL TECHNIQUES:

TECHNIQUE (EN)	DATE COMPLETED
x CCGC/MS (18)	12/18/85
x GC/EC (18)	12/12/85

QUALITATIVE: X SEMIQUANTITATIVE: QUANTITATIVE: X

REQUIRED QA/QC: SEE GOOD LABORATORY PRACTICES MANUAL

COMPLETION DATE REQUESTED:

COMMENTS: Results phoned in to Jamro on 12/13/85.

SAMPLE DISPOSITION: HOLD FOR 60 DAYS

SAFETY PRECAUTIONS: SEE SAFE LABORATORY PRACTICES MANUAL

ANALYSIS REQUEST FILE NAME: AA538

The following is a copy of the ESC/UAG Good Laboratory Practices Manual.

Copy Number: _____

GOOD LABORATORY PRACTICES MANUAL

A Guide to Quality Control/Quality Assurance

Ultratrace Analysis Group

March 1984



Joseph J. Brooks
Group Leader, Ultratrace Analysis



H. A. Woltermann
Manager, Environmental Sciences Center

Issued to: _____

Date: _____

Environmental Sciences Center
Dayton Laboratory
Dayton, Ohio 45407

JR3/JOB A

MONS 023884

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FIGURES

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I. Objectives

The main objectives of the Ultratrace Analysis Group quality assurance/quality control program are: 1) to assure that our laboratory generates high quality results; and 2) to maintain the necessary records that document laboratory performance.

II. Scope

The scope of this manual includes areas of GLP not specifically addressed in the ESC section manual and expands upon certain areas where QA/QC practices differ from those described in the section manual. It is intended to be used along with the section manual to provide a complete picture of the GLP within the Ultratrace Analysis Group.

III. Analysis

The generation of analytical results within the Ultratrace Analysis Group routinely involves two processes: sample preparation and instrumental analysis. Sample preparation involves one or more of a variety of techniques including extraction, dilution, acid/base washes, column chromatography, concentration and numerous other techniques to render the sample in a suitable condition for instrumental analysis. Instrumental analysis takes the suitably prepared sample and introduces it into an analytical instrument to measure the desired parameter(s). Both of these processes can be described by clearly defined and separable methods called sample preparation methods (SPMs) and instrumental analysis methods (IAMs). SPMs and IAMs may be combined in numerous ways (depending upon the nature of the samples and the needs of the customer) to produce a successful analytical protocol. For example, several different SPMs may be used to produce samples that are suitable for analysis by a single IAM. The measures incorporated into each of these experimental processes to ensure that an assessment of the quality of the results can be made constitute the QA/QC program for a project. The following subsections describe the elements of the analysis process:

A. Methods

Methods are the written documentation that describe the analytical process. They contain sufficient detail such that a competent scientist or technician can readily perform the required analyses. The usual practice is to have methods that describe the sample preparation process (SPMs) and methods that describe the instrumental analysis process (IAMs). Each method developed within the Ultratrace Analysis Groups is assigned a method number as described in the ESC Section Manual.

Methods are obtained from various sources including in-house development, modification/adaptation of literature methods, or specific protocol methods dictated by government agencies. Methods may be formally validated by a series of experiments that describe a classical validation protocol or may be less vigorously proven owing to factors such as frequency or use, customer needs and

MONS 023886

cost/timing restrictions. In any case a QA/QC program is applied to the analysis process to provide a measure of the quality of the results. Unless a QA/QC program is prescribed by a required protocol (e.g., EPA Dioxin in Soil protocol), certain minimum QA/QC measures (depending upon the analysis type) are incorporated into all Ultratrace Analysis projects. The following subsection describes the Ultratrace QA/QC program:

B. QA/QC Program

An effective QA/QC program incorporates experiments that provide information relative to each of the following questions:

(1) Are there any impurities, background or contamination that will influence the results?

(2) How reproducible are the results?

(3) How well can the analyte be recovered from the sample matrix?

(4) What is the limit of detection for the analysis?

(5) What is the dynamic range of the analysis?

(6) What is the accuracy of the analysis?

All of these questions are addressed by properly selecting/applying the use of blanks, replicates, spikes and standards in a QA/QC program. The discussion of the use of these QA/QC techniques as they apply to Ultratrace Analysis Group activities is best done by considering the sample preparation and instrumental analysis processes separately.

1. Sample Preparation QA/QC

a. Method blanks - Representative glassware, solvents and reagents are used with sample matrices which are similar to those for which results are being reported, yet do not contain the analytes of interest. Method blanks are analyzed at a frequency equal to 10% of the analyzed samples for sample sets which are multiples of 10. At least one method blank is analyzed for sample sets containing between 1 and 9 samples.

b. Replicates - If sufficient sample amounts have been submitted for analysis, replicate frequency is the same as the method blank frequency. If at all possible, replicates should be obtained from the same sample bottle in order to minimize sample inhomogeneity problems due to samples being taken at slightly different times.

c. Spikes - If sufficient sample is available for each sample type, the sample is split and spiked at two levels with

MONS 023887

the native (unlabeled) analyte(s). The levels are typically 2x (low) and 20x (high) the anticipated detection limit and serve as a technique for estimating the limit of detection for each matrix. These low and high spikes are conducted at a frequency equal to the Method Blank frequency. Obviously spiking with the native analyte cannot be done for widescan analysis since this presupposes knowledge of the identity of the analyte(s) in the sample.

Other spiking techniques are used to evaluate the extraction/recovery efficiencies for the analyte(s) of interest. Ideally extraction/recovery efficiencies are determined for each sample, since, in principle, each sample may represent a different matrix. This can be done very easily when the detection technique is mass spectrometry if an isotopically labeled version of the analyte(s) is available. Documentation of extraction/recovery efficiencies is not required for each sample if the sample matrix for a set of samples is constant. However, for a sample set for which the similarity of samples is not known, and for which adequate sample is available, each sample must be split and spiked with a known amount of the analyte(s) of interest to determine extraction efficiency. If adequate sample size is not available or the identity of the analyte(s) is not known before the fact (i.e., widescan analysis), surrogate spiking compounds may be used to assess extraction efficiencies in each sample without actually spiking each sample with the analyte(s) of interest. The choice of the surrogate spiking compound is tailored for each analyte. This surrogate must be chosen so that it is either chemically identical to the analyte of interest (for example using isotopically labeled analogues of the analytes), or is closely similar in terms of liquid/liquid partition properties, chemical reaction properties, column elution properties or other matrix isolation properties which are used to isolate the analyte(s) from the sample matrix. Another property of the surrogate spiking compound is that it not be present in the sample being analyzed.

2. Instrumental Analysis QA/QC

The final step in obtaining analytical results involves the use of an appropriate instrumental technique to produce the analytical data. Several QA/QC practices must be incorporated in this process to ensure validity of the data.

a. Instrumentation

Careful documentation of instrument usage, calibration, and maintenance is essential for the generation of high quality results. Each major instrument within the Ultratrace Analysis Group will be the assigned responsibility of a professional employee designated as the Instrument Steward. The Instrument Steward is responsible for making sure that logbooks are available and properly completed by instrument users. It is the

responsibility of each individual using the equipment to record all the pertinent data required in the logbook. An example of a page from a GC/MS logbook is given in Figure 1 and that of a GC/EC logbook is given in Figure 2.

Instruments will be calibrated according to the instrument manufacturers' specifications or according to the method being used with the instrument. A log of instrument calibration will be maintained with the instrument.

The Instrument Steward will be responsible for ensuring that preventative and corrective maintenance programs are carried out on all instruments under his/her responsibility. If a specific problem is noted by an instrument user, it should be noted in the logbook and the Instrument Steward notified so that appropriate corrective action can be taken.

b. Blanks

A solvent blank will be analyzed at a frequency equal to the method blank. The method blank can serve as the solvent blank provided it is clean in the area of the analyte(s) of interest. This practice eliminates the possibility of background instrumental contamination.

c. Quantitation Internal Standard

A quantitation internal standard will be added to each sample following sample preparation but prior to instrumental analysis. This compound will serve for determining relative response factors and for correcting for variations in sample injection volumes and evaporation of solvents.

d. Standards

Standards are solutions of the analyte(s) of interest used to determine the response of the detector over the range of analyte concentrations found in the samples. For Quantitative analyses, sufficient standards and replication of standards will be run to demonstrate linearity and precision of the detector response over the range of analyte concentrations. Should sample concentrations fall outside the demonstrated linear range, either additional standards are prepared to expand the range, or appropriate dilution/concentration of the sample is done to place it in the proper range.

C. Degrees of Quantitation

The information provided for any particular analysis is tailored to the needs of the customer. In actual practice these needs vary across the broad continuum from the grossly qualitative to the precisely quantitative. It is informative to examine what is involved in providing the varying degrees of quantitation required by our customers.

HP 5985A

MRC-DA
Nº 15604

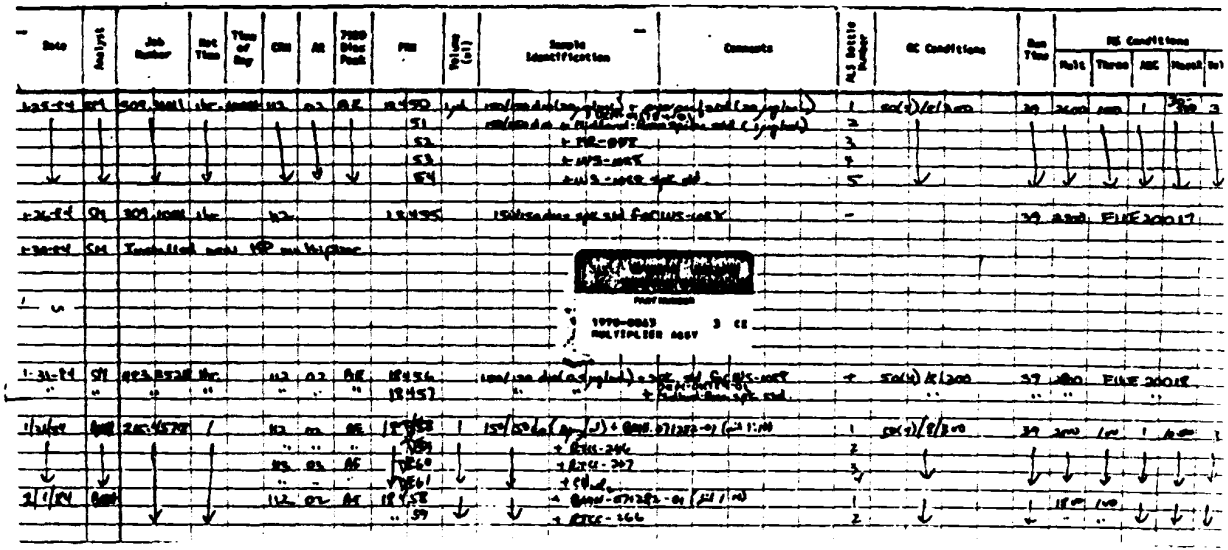

FIGURE 1. Example of GC/MS Logbook page.

MONS 023890

HP 5880 GC/EC

MONSANTO RESEARCH CORPORATION
DAYTON LABORATORY

SUBJECT _____ DATE _____ 19__ READ AND UNDERSTOOD BY ME

_____ SIGNATURE _____ DATE _____ 19__

JOB NO. _____

MRC-DA
Nº 156363

		Name	Bit #	Device No.	Ramp No.
		MIPCA 234	90	AP 190	AR 190
		MIPCA 235	91	91	191
		MIPCA 236	92	92	192
		MIPCA 237	93	93	193
		MIPCA 238	94	94	194
2/22/84	CK	ANISV	01	01	01
		COMPPCA 229, 244	02	02	02
		MIPCA 232	03	03	03
		MIPCA 233	04	04	04
		MIPCA 234	05	05	05
		MIPCA 235	06	06	06
		MIPCA 236	07	07	07
		ANISV (1.0)	08	08	08
		MIPCA 239	09	09	09
		MIPCA 231	10	10	10
		MIPCA 226	11	11	11
		MIPCA 227	12	12	12
		MIPCA 228	13	13	13
		ANISV (1.0)	14	14	14
2/22/84	CK	ANISV (1.0)	15	15	15
		COMPPCA 240, 241	16	16	16
		MIPCA 230	17	17	17
		MIPCA 239	18	18	18
		MIPCA 240	19	19	19
		MIPCA 241	20	20	20

05	A1234(1.0)	05.	BP105	BR105	100.00	1.0000	1.0000	
06	BM1221-220	06.	BP106	BR106	100.00	1.0000	1.0000	
07	M1221	07.	BP107	BR107	100.00	1.0000	1.0000	
08	M1222	08.	BP108	BR108	100.00	1.0000	1.0000	
09	M1223	09.	BP109	BR109	100.00	1.0000	1.0000	
90	M1224	90.	BP190	BR190	100.00	1.0000	1.0000	
91	M1225	91.	BP191	BR191	100.00	1.0000	1.0000	
92	M1226	92.	BP192	BR192	100.00	1.0000	1.0000	
93	M1227	93.	BP193	BR193	100.00	1.0000	1.0000	
94	M1228	94.	BP194	BR194	100.00	1.0000	1.0000	

1	A1234(1.0)	1.	BP101	BR101	100.00	1.0000	1.0000	
2	BM1229-244	2.	BP102	BR102	100.00	1.0000	1.0000	
3	M1229	3.	BP103	BR103	100.00	1.0000	1.0000	
4	M1230	4.	BP104	BR104	100.00	1.0000	1.0000	
5	M1231	5.	BP105	BR105	100.00	1.0000	1.0000	
6	M1232	6.	BP106	BR106	100.00	1.0000	1.0000	
7	M1233	7.	BP107	BR107	100.00	1.0000	1.0000	
8	A1234(1.0)	8.	BP108	BR108	100.00	1.0000	1.0000	
9	M1234	9.	BP109	BR109	100.00	1.0000	1.0000	
10	M1235	10.	BP110	BR110	100.00	1.0000	1.0000	
11	M1236	11.	BP111	BR111	100.00	1.0000	1.0000	
12	M1237	12.	BP112	BR112	100.00	1.0000	1.0000	
13	M1238	13.	BP113	BR113	100.00	1.0000	1.0000	
14	A1234(1.0)	14.	BP114	BR114	100.00	1.0000	1.0000	
15	A1234(1.0)	15.	BP115	BR115	100.00	1.0000	1.0000	
16	BM1229-244	16.	BP116	BR116	100.00	1.0000	1.0000	
17	M1238	17.	BP117	BR117	100.00	1.0000	1.0000	
18	M1239	18.	BP118	BR118	100.00	1.0000	1.0000	
19	M1240	19.	BP119	BR119	100.00	1.0000	1.0000	
20	M1241	20.	BP120	BR120	100.00	1.0000	1.0000	
21	M1242	21.	BP121	BR121	100.00	1.0000	1.0000	
22	M1243	22.	BP122	BR122	100.00	1.0000	1.0000	
23	M1244	23.	BP123	BR123	100.00	1.0000	1.0000	
24	A1234(1.0)	24.	BP124	BR124	100.00	1.0000	1.0000	

FIGURE 2. Example of GC/EC Logbook page.

MONS 023891

1. Qualitative Analysis

Qualitative analysis implies no quantitation at all but rather seeks only the identity of the compound(s) present. GC/MS is particularly applicable to this type of analysis problem. Two types of identifications can be made:

a. Tentative

A tentative identification is made on the basis of the analyst's judgement of a match between the mass spectrum from the sample and a mass spectrum from a reference library. This is often done for the large number of compounds found in a widescan organic analysis.

b. Confirmed

A confirmed identification requires that the chromatographic retention time and the mass spectrum of the unknown compound match those of a known standard of the tentatively identified compound. This is often done in widescan analyses when standards can be obtained for tentatively identified compounds that are of concern.

The normal QA/QC requirements for qualitative analysis are (1) analysis of a blank, (2) analysis of duplicate samples and (3) use of a calibration compound (usually Decafluorotriphenylphosphine, DFTPP) to assure that the mass spectrometer will produce mass spectra that can be compared with library spectra.

2. Semi-Quantitative Analysis

Semi-Quantitative Analysis extends the information provided by a qualitative analysis to include some estimate of the amount of analyte(s) present. The method of semi-quantitation involves the comparison of detector response of the analyte(s) with that of an internal standard. The estimated concentration is obtained by assuming a unit response factor between the analyte(s) and the internal standard(s). An indication of recovery from the sample matrix is obtained from the recovery of the surrogate internal standard(s).

3. Quantitative Analysis

Quantitative Analysis provides for the accurate assessment of the amount of analyte(s) present in a sample. To be able to do quantitative analysis one must know the identity of the analyte so that standards can be prepared for spiking and for the generation of calibration curves. The method of quantitation is based on a three-point calibration curve using standards of the analyte(s) of interest over the range of concentrations in the samples. A one-point calibration method can be used (as with the laboratory data

system) provided that linearity has been demonstrated over the concentration range of the samples using standards of the analyte(s) at three concentrations in the range. This calibration curve is then used to provide concentrations of the analyte(s) in the samples via an external standards method or more preferably via an internal standards method. For the internal standards method, the quantitation internal standard is added to each of the standards (just as with the samples) prior to instrumental analysis and relative response factors are determined.

For quantitative analysis the mass spectrometer is operated as a gas chromatographic detector. The MS is therefore tuned and operated in a manner specified in the approved method rather than using a calibration compound as required for qualitative analyses.

D. Quality Criteria and Remedial Action

The inclusion of QA/QC measures into an analysis program has little meaning unless the results are interpreted and appropriate action is taken when necessary. The ability to do this assumes some set of criteria that will trigger a response or remedial action. The following subsections describe the criteria that are used in the Ultratrace Analysis Group to evaluate when quality control data indicate a need for corrective measures and what remedial action will be taken.

1. Quality Criteria

The overriding quality criterion above all others is: Do the results meet the needs of the customer. This determination will be made jointly between the customer and the analyst. The analyst must exercise caution in not allowing the customer to overstate, misinterpret or unintentionally misrepresent the data. In addition to this prime quality criterion, the following conditions must be met:

a. Blanks - In the event that a blank produces a positive value, it may not be used as a valid blank if it exceeds 10% of any reported value for a sample.

b. Recoveries - Recoveries for all surrogate and native spikes must lie in the range of 50%<Recovery<150%.

c. Precision - Precision will be calculated based on the recoveries of surrogate internal standards. Average recoveries will be calculated with ± standard deviation for each sample set. Replicate analyses and low and high native spike recoveries may also be used to provide additional information/insight into the precision of the analysis.

MONS 023893

d. Limit of Detection - The limit of detection (LOD) will normally be stated as ½ the value of the low level native spike if it is recovered and quantifiable. Should the low level native spike not be recovered, the LOD will be stated as ½ the value of the high level native spike if it is recovered and quantifiable. Should neither spike be recovered, a decision must be made regarding the advissbility of reprocessing the sample. Should all samples contain such large quantities of the analyte(s) that the levels of the low and high spikes are insignificant (≤10%) by comparison, the value of the LOD becomes less important and is simply stated as the LOD for the instrumental analysis process determined by the analysis of standards and the ability to recover the surrogate spikes.

e. Accuracy - The accuracy of the analyses is best determined by analyzing some sample which contains the analyte at some predetermined level (e.g., an NBS standardized sample). If this type of standard is not available then the low level and high level spikes can be used as a measure of the accuracy of the analyses. Results must fall within a range of 75% to 120% of the excepted value for the analyses to be considered valid.

If the above quality criteria are not met the analyst will immediately assess the need for and extent of remedial action that may be necessary.

2. Remedial Action

The first course of action will be for the analyst and/or group leader to contact the customer and discuss the results pointing out the limitations of the data indicated by the quality control results. At this point the overriding quality criterion of customer needs will be addressed first. It could be that the customer's needs are met without meeting one or more of the other criteria. In such a case no remedial action is necessary. It could also be that the customer has such a demand for tightness in the results that his needs are not met even though the other quality criteria are. This would trigger further analyses with tighter quality control designed around the results obtained (e.g., replications of positives to better establish precision or spikes of non-detected samples to better establish the limit of detection).

In consultation with the customer additional experiments will be designed including the appropriate quality control to ultimately provide the results of sufficient quality to meet the customer's needs.

IV. Data

This section deals with the subject of data and how they are obtained, recorded and reduced so as to be useable in a report and yet readily available for referral and inspection.

The samples will be labeled with a log number at sample login according to the procedure described in the ESC Section GLP Manual. When delivered to the responsible technical person, the samples may then receive additional labeling as deemed necessary. This additional labeling will be documented in a laboratory logbook and/or notebook. These logbooks and notebooks will be used by the analysts to record all pertinent work and results obtained from the samples. The laboratory notebooks will be filled out in accordance with Monsanto Uniform Notebook Procedures.

All raw data generated for a given project, which cannot be conveniently placed in the laboratory notebook (e.g., chromatograms, computer output, etc.), will be filed under some prescribed order (i.e., log number) in a central file under the supervison of the Project Leader and/or QA Specialist.

Most of the data generated by the Ultratrace Analysis Group are computer data stored on magnetic discs. In order to archive these data the specific file name and disc number associated with the analysis will be written down in the laboratory notebook and/or instrument logbook. When the disc is completely full of data an entire unedited disc image or copy of the files on the disc will be transferred to magnetic tape. A logbook will be kept for recording the disc number, name and prefix associated with it, along with the associated tape and file number to which the disc was transferred. An example of such a logbook is given in Figure 3. When the data are transferred a verification program is used to check all transferred records for correctness. When tapes are filled (they typically hold seven to ten disc images) they should be stored in a fireproof vault.

All calculations of the results of the data will be specified in the method used. These calculations will be entered in the analysts laboratory notebook unless they are computer generated. In this case, a copy of the computer output will be filed with the raw data. The Project Leader will be responsible for checking the correctness of the data.

V. Sample Disposal

Samples will be disposed of in accordance with the instructions on the Analysis Request form. The only exception to this would result when the analysis reveals a problem which requires sample disposal in a manner other than that originally specified. If the Project Leader notes from the results of the analysis that a potentially toxic product was present in the samples then the samples will be disposed of in a manner deemed safe by the Safety Department. The date of sample disposal and the manner should be noted on the request form stored with the raw data completing the chain of custody of the sample.

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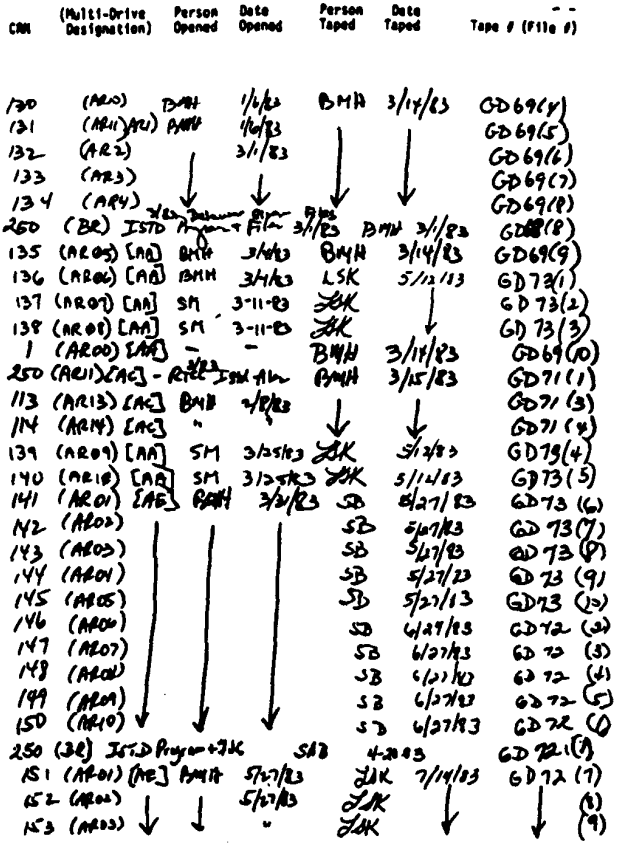

FIGURE 3. Example of DISC/Tape Logbook page.

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