

FILE NITRO
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MONSANTO COMPANY / DAYTON LABORATORY / DAYTON, OHIO 45407

ESC/DAYTON

(Department/Section)

ANALYTICAL

(Type of Report)

REPORT NO.: MDA-565

JOB/PROJECT NO.: 484.4420

DATE: 8 January 1985

PERIOD COVERED:

TITLE: Analyses of Wipes for 2,3,7,8-TCDD

AUTHORS: G. L. Thomas

ABSTRACT: Wipe samples from the Nitro plant were analyzed for possible concentrations of 2,3,7,8-TCDD. The results of these analyses are shown in this report.

TECHNICAL APPROVAL:

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APPROVAL DATE:

1-8-85

RESTRICTED DISTRIBUTION: (see over)

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C11059

AUTHORS: G. L. Thomas
TITLE: ANALYSES OF WIPES FOR 2,3,7,8-TCDD

REPORT NO.: MDA-565

COPY NO.: 4

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ABSTRACT ONLY

H. A. Woltermann

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ACKNOWLEDGEMENT

Contributions of the following Dayton Laboratory personnel to this project are gratefully acknowledged:

Analyses: G. L. Thomas

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C11061

ANALYSIS REPORT FOR 2,3,7,8-TCDD IN NITRO SAMPLES

INTRODUCTION

On 18 Dec 84 eight wipe samples were received for the analysis of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD). For internal purposes, the samples were assigned MRC Log #1-84-12-18-01. The samples were analyzed according to the September 1983 EPA Region VII method "Determination of 2,3,7,8-TCDD in Soil and Sediment". For quality control purposes MRC Method #07-QAQC-R3 was followed. Briefly this method requires that for every set of samples of the same matrix type a replicate sample, a low (9.6 ng in this case) spiked sample, a high (96 ng in this case) spiked sample, and a method blank must be analyzed.

PREPARATION AND ANALYSIS (WIPES)

The wipes were spiked with 24.0 ng of carbon-13 labeled 2,3,7,8-TCDD and 9.6 ng of chlorine-37 labeled 2,3,7,8-TCDD. Then ten grams of washed anhydrous sodium sulfate was added and the samples were stirred, allowed to stand and stirred again. Samples were mixed again immediately prior to addition of extraction solvents. The extraction process involved the addition of 15 mL of methanol (all solvents are Burdick and Jackson "distilled in glass" grade), stirring and the subsequent addition of 150 mL of hexane. The samples were then shaken for one hour on a wrist action shaker and the extraction solvent removed. The solvent was then successively extracted with 50 mL of water, concentrated sulfuric acid (until clear), water, 20% potassium hydroxide and water. The hexane was then dried with 10 grams of anhydrous sodium sulfate. Each sample was then placed on the head of a mixed phase column consisting of (top to bottom) 2 g of silica, 4 g of silica plus 40% sulfuric acid, 1 g of silica, 2 g of silica plus 36% potassium hydroxide and 1 g of silica. The column was eluted directly onto a column containing 2.5 g of Woelm basic alumina. The alumina column was first eluted with 10 mL of 2% methylene chloride/hexane (v/v) and this fraction discarded. An additional elution was made with 15 mL of a 50% methylene chloride/hexane (v/v) solution to elute the TCDD off the column.

All samples had 50 uL of dodecane keeper added to them and then were concentrated under a gentle stream of nitrogen and submitted for GC/MS analysis. The GC/MS analyses were conducted on a Hewlett/Packard 5987 GC/MS instrument. The analysis was conducted using a 60 m SP2330 capillary column. The mass spectrometer was operated in the selected ion monitoring mode analyzing for ions characteristic of both the native (unlabeled) 2,3,7,8-TCDD (m/z 257,320,322), chlorine-37 labeled 2,3,7,8-TCDD (m/z 328), and carbon-13 labeled 2,3,7,8-TCDD (m/z 332,334).

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STANDARDS

Standard solutions were prepared from standards that had been cross validated with a standard provided as a check sample by the EPA. Concentration of the internal standard conformed to the levels specified by the method.

RESULTS

The results of the analyses are given in tables 1-3. The tables also give all pertinent QA/QC information.

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TCDD DATA REPORT
Monsanto Company, Dayton Laboratory

GC COLUMN: SP2330
RF NATIVE: .378
RF SURROGATE: 1.121

SAMPLE NUMBER	EXTR MTHD	CLEAN	NG TCDD	D.L.	ANALYTICAL DATE	TIME	SURR %		320	322	257	323+	332	334	M
							320	332							
1	J	BC	1.15	—	12/19	1041	.90	.80	108.	4210.	5238.	3404.	117652.	105329.	132009.
2	J	BC	.94	—	12/19	1101	.82	.81	103.	3981.	4881.	3180.	130100.	121734.	149508.
3	J	BC	ND	.27	12/19	1122	****	.81	108.	0.	0.	0.	87404.	72763.	90262. X
4	J	BC	ND	.11	12/19	1142	****	.80	108.	0.	0.	0.	202749.	177694.	222731. X
5	J	BC	ND	.18	12/19	1202	****	.80	104.	0.	0.	0.	115823.	104544.	129927. X
6	J	BC	1.24	—	12/19	1242	.83	.80	107.	7473.	8978.	4848.	192439.	170592.	213347.
7	J	BC	1.28	—	12/19	1302	.80	.81	108.	5934.	7449.	3984.	151415.	134805.	164051.
8	J	BC	.89	—	12/19	1322	.83	.81	107.	4525.	5437.	3918.	161143.	143664.	177982.
MS	J	BC	ND	.14	12/19	1342	****	.81	108.	0.	0.	0.	152741.	135676.	167083. X
HIGH SPIKE	J	BC	106.76	—	12/19	1442	.80	.79	109.	396327.	492503.	165039.	122085.	106076.	134320.

+ CORRECTED FOR CONTRIBUTION BY NATIVE TCDD (SUBTRACT .009 OF M/E 322)

NOTE CODES:

X: ISOTOPE RATIO OUT OF ACCEPTABLE RANGE
Y: % ACCURACY OUT OF ACCEPTABLE RANGE

MB=METHOD BLANK
P=PARTIAL SCAN
R=SEPARATE WEIGHING AND WORKUP
D=DUPLICATE (INTERLAB)
FB=FIELD BLANK

L=NATIVE TCDD SPIKES
ND=NOT DETECTED
DL=DETECTION LIMIT
J=JAR

FILENAME =TCDD033

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TABLE 2. CALIBRATION SUMMARY

ug/Kg NATIVE TCDD	RRF (NATIVE)	DATE	TIME	RRF (SURROGATE)
0.5010	0.9868	12/19	1001	1.123
5.010	0.8551	12/19	1021	1.224
50.10	0.8558	12/19	1222	2.174
1.2515	0.9019	12/19	1422	1.017
12.515	0.8881	12/19	1505	1.895
125.15	0.8551	11/27	1525	1.798

GTT2X

06007
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TABLE 3. QUALITY CONTROL SUMMARY

ITEM	# DATA POINTS	MEAN $\pm$ S.D.
SURROGATE ACCURACY	10	107.2 $\pm$ 1.7
SPIKE RECOVERY	1	*

* Not technically correct to report recoveries for internal standard calculated results.

GTT3X

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DOCUMENTATION INVENTORY FORM

Monsanto Research Corporation
Dayton Laboratory

I. Sample Information

Sample Identification: NITRO

Number of Samples: 8

Analyte: 2,3,7,8-TCDD

Matrix: W, TES

Method: Determination of 2,3,7,8-TCDD in Soil and Sediment

II. Documentation Control

	<u>Copies Attached</u>	<u>Initials</u>
(a) Sample Receipt Form	<u>✓</u>	<u>Bmw</u>
(b) Internal Sample Tracking Form	<u>—</u>	<u>—</u>
(c) Sample Tags	<u>—</u>	<u>—</u>
(d) Airbill (Number <u>—</u>)	<u>—</u>	<u>—</u>
(e) Chain of Custody Form	<u>✓</u>	<u>Bmw</u>
(f) Analysis Request (Log Number <u>1-84-12-13-49</u>)	<u>✓</u>	<u>Bmw</u>
(g) Extraction/Preparation notebook pages (NBP) <u>2831630</u>	<u>✓</u>	<u>Bmw</u>
(h) Analysis NBP <u>2273276</u>	<u>✓</u>	<u>Bmw</u>
Instrument Logbook pages <u>156907</u>	<u>✓</u>	<u>Bmw</u>
Computer Data Files hard copy output	<u>✓</u>	<u>Bmw</u>
Chromatograms	<u>✓</u>	<u>Bmw</u>
(i) Data Reduction NBP <u>2273276</u>	<u>✓</u>	<u>Bmw</u>
(j) Final Report	<u>✓</u>	<u>Bmw</u>
(k) Additional Documentation	<u>✓</u>	<u>Bmw</u>

(note immediately upon acquisition)

Standards Preparation
NBP 2273264, 2273265

File Complete

Barbara M. Williams
(signature)

1-4-85
(date)

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Sample Receipt Form

Monsanto Research Corporation
Dayton Laboratory

Customer Identification: NHRO
MRC Log Number: 1-84-12-1801
Airbill Number: —
Date: 18 DEC 84

Sample Receipt Information:

				Initials
(a) Shipping Containers	<u>intact</u>	damaged (describe below)	No. <u>1</u>	<u>GT</u>
(b) Custody Seals	present	<u>absent</u> (describe sealing below)	intact <u>—</u>	<u>GT</u>
(c) Chain of Custody Forms	<u>present</u>	absent	No. <u>2</u>	<u>GT</u>
(d) Sample Tags	<u>present</u>	absent	No. <u>8</u>	<u>GT</u>
(e) Sample Containers	<u>intact</u>	damaged (describe below)	No. <u>8</u>	<u>GT</u>
(f) Agreement of Sample Documentation	<u>yes</u>	no (describe discrepancies)		<u>GT</u>

Additional Description (if sample numbers not listed on chain of custody form, list here and contact SMO).

b) SEALED WITH PACKING TAPE.

Gary Thomas
(Signature)

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C11068

CHAIN OF CUSTODY RECORD

MRC JOB NUMBER _____

CUSTOMER REPRESENTATIVE Keith Miller

CONTRACT NUMBER _____

MRC CONTRACT MANAGER _____

PLANT NAME OR CODE Monsanto Nitro

MRC TEAM LEADER _____

(Specific information on samples is available on attached "Sample Identification Log")

Signatures		Date	Time	Remarks
Team Leader Verification of Collected Samples:				
Relinquished by: <u>K. Miller</u>	Received by:			
Relinquished by:	Received by: <u>Roy Noble</u>	<u>12 Aug 84</u>	<u>820</u>	
Logged-in-by:				
Received for Analysis by:				
Method of Shipment:		Number of Containers:		

Special Instructions (Sample preservation, security, delivery to, etc.)

Deliver to Mr. Roy Noble
Monsanto Research Corp
Dayton Laboratory
1515 Nicholas Rd
Box 8
Dayton Ohio 45407

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011069

SAMPLE IDENTIFICATION LOG

MRC Job No. _____

Customer Representative Keith Miller

Contract No. _____

MRC Contract Manager _____

Plant Name or Code Nitro

Team Leader _____

[illegible]

00012

C11070

ANALYSIS REQUEST		DATE 18 DEC 84	LOG NO. 1-84-12-R-01	REPORT RESULTS TO FILES/KULIK SECRETARY
MONSANTO RESEARCH CORPORATION DAYTON LABORATORY		REQUESTER** NITRO/K. MILLER	CHARGE NO.** 454.44229	
SAMPLE DESCRIPTION	NUMBER OF BOTTLES: 8	NUMBER OF SAMPLES ** 8		
NO. 1 LAB BLANK (NITRO) NO. 2 LAB BLANK (NITRO) NO. 3 LAB BLANK (DAYTON) NO. 4 LAB BLANK (DAYTON) NO. 5 79 BLANK (DAYTON) NO. 6 FUEL VALVE ROUSE (DAYTON) NO. 7 FUEL VALVE ROUSE (NITRO) NO. 8 79 BLANK (NITRO)				ANALYSIS REQUEST FILES SECRETARY SECRETARY
REQUIRED ANALYSIS**				SAMPLES FOR: NO. 1-5
2,3,7,8-TCDD RECORDED BY: John Thomas				SAMPLE LOCATION CSL

KNOWN HEALTH HAZARD PRESENTED BY SAMPLE: **

REFERENCE TO PREVIOUS WORK (REQUEST #, JOURNAL, ETC.) **

COMPLETION DATE _____

TIME ALLOTTED FOR ANALYSES

ANALYST

HOURS

INST. TIME

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ANALYTICAL TECHNIQUE **

- | | |
|---|--|
| ☐ GAS CHROMATOGRAPHY | ☐ NMR |
| ☐ ULTRAVIOLET | ☐ HPLC |
| ☐ VISIBLE | ☐ XRD |
| ☐ INFRARED | ☐ ICP |
| ☐ EMISSION | ☐ ATOMIC ABSORPTION |
| ☐ EDAX XRF | ☐ MASS SPEC |
| ☐ MICROSCOPY | ☒ GC/MS |
| ☐ OTHER _____ | ☐ AUTO ANALYZER |
| QUAL. _____ SEMIQUANT. _____ QUANT. _____ | |

REQUIRED QA/QC

**SAMPLE DISPOSAL:
AFTER REPORT COMPLETE

☐ DISCARD

☐ RETURN

HOLD FOR ☐ 30 DAYS

☐ 60 DAYS

OTHER

** THIS INFORMATION MUST BE SUPPLIED BY ANALYSIS REQUEST ORIGINATOR

C11071

Nº 2831620

Nitro Wipes

NO.

484.44229

PREPARED BY (SIGNATURE)

PREPARED BY (SIGNATURE)
Benet R. Curtis

DATE _____

19 Dec 84

Date 18 Dec 84

Nitro Wipes

Log No.

Test No.

Log No. 454.44229
Job No. 454.44229

Data Sheet

weighed by

Spitak by

B. R. Curtis

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06014

READ AND UNDERSTOOD BY

PAT

C11072

MONSANTO COMPANY

Nº 2273276

SUBJECT

Nitro Wipes

JOB NO.

484.44229

PREPARED BY (SIGNATURE)

Ray Thomas

DATE

4 JAN 8

TICD DATA REPORT
MONSANTO COMPANY, DAYTON LABORATORYGC COLUMN SP2330
RF NITROGEN .009
RF CARBONATE 1.121

SAMPLE NUMBER	EXTR MTHD	CLEAN	NO TICD	D.L.	ANALYTICAL DATE	TIME	330		337	334		337	331+		332	334	
							322	334		322	334		322	334		322	334
1	J	BC	1.15	--	12/14	1041	.30	.30	103.	4210.	5238.	3606.	119452.	105329.	133008.		
2	J	BC	.94	--	12/14	1101	.32	.31	103.	3981.	4981.	3100.	130100.	121204.	149902.		
3	J	BC	NO	.27	12/14	1122	****	.31	103.	0.	0.	0.	92406.	72743.	70242.	X	
4	J	BC	NO	.11	12/14	1142	****	.30	108.	0.	0.	0.	202749.	177478.	220731.	X	
5	J	BC	NO	.18	12/14	1202	****	.30	106.	0.	0.	0.	115323.	104544.	129927.	X	
6	J	BC	1.24	--	12/14	1242	.33	.30	107.	7473.	3979.	4942.	172479.	170392.	213347.		
7	J	BC	1.23	--	12/14	1302	.33	.31	108.	5934.	7449.	3984.	151412.	136925.	144651.		
8	J	BC	.39	--	12/14	1322	.33	.31	107.	4525.	5417.	3913.	161143.	143464.	177382.		
9	J	BC	NO	.14	12/14	1342	****	.31	109.	0.	0.	0.	152741.	135626.	167083.	X	
HIGH SPIKE	J	BC	106.76	--	12/14	1442	.30	.79	109.	376327.	473503.	115009.	122052.	104076.	134720.		

* CORRECTED FOR CONTRIBUTION BY NATIVE TICD (EXTRACT .009 OF N/E 322)

NOTE CODES:

X: ISOTOPE RATIO OUT OF ACCEPTABLE RANGE

Y: Z ACCURACY OUT OF ACCEPTABLE RANGE

NOMINATING BLANK

PARTIAL SCAN

REPEATABLE WEIGHING AND WORKUP

DUPLICATE/INTERLAB

FRONTLOAD BLANK

L: NATIVE TICD SPIKES

NOMINALLY DETECTED

DETECTION LIMIT

J: LAB

FILENAME #100003

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C11073

READ AND UNDERSTOOD BY

DATE

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MONSANTO RESEARCH CORPORATION

DAYTON LABORATORY

SUBJECT _____ DATE _____

SIGNATURE _____

READ AND UNDERSTOOD BY ME _____ DATE _____

MRC-DA
No 15691

Date	Analyst	Job Number	Met Time	Time of Day	CRH	AP	7920 Disc Pack	FMN	g (in)	Sample Identification	Comments	ALS Bottle Number	GC Conditions	Run Time	MS Conditions				
															Multi	Thres	ADC	MassA/D	
180-PM	FW				04			7F1632	2	LT 3244				18	3000				
								3	1	UT 3245									
								4	1	UT 3246									
								5	1	7E mix									
								6	1	100% 14/2									
								7	1	100% 14/2									
								8	1	UT-3207									
								9	1										
								10	1										
								11	1										
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								98	1										
								99	1										
								100	1										

00016

180-PM

ANALYST

Job Number

Met Time

Time of Day

CRH

AP

7920 Disc Pack

FMN

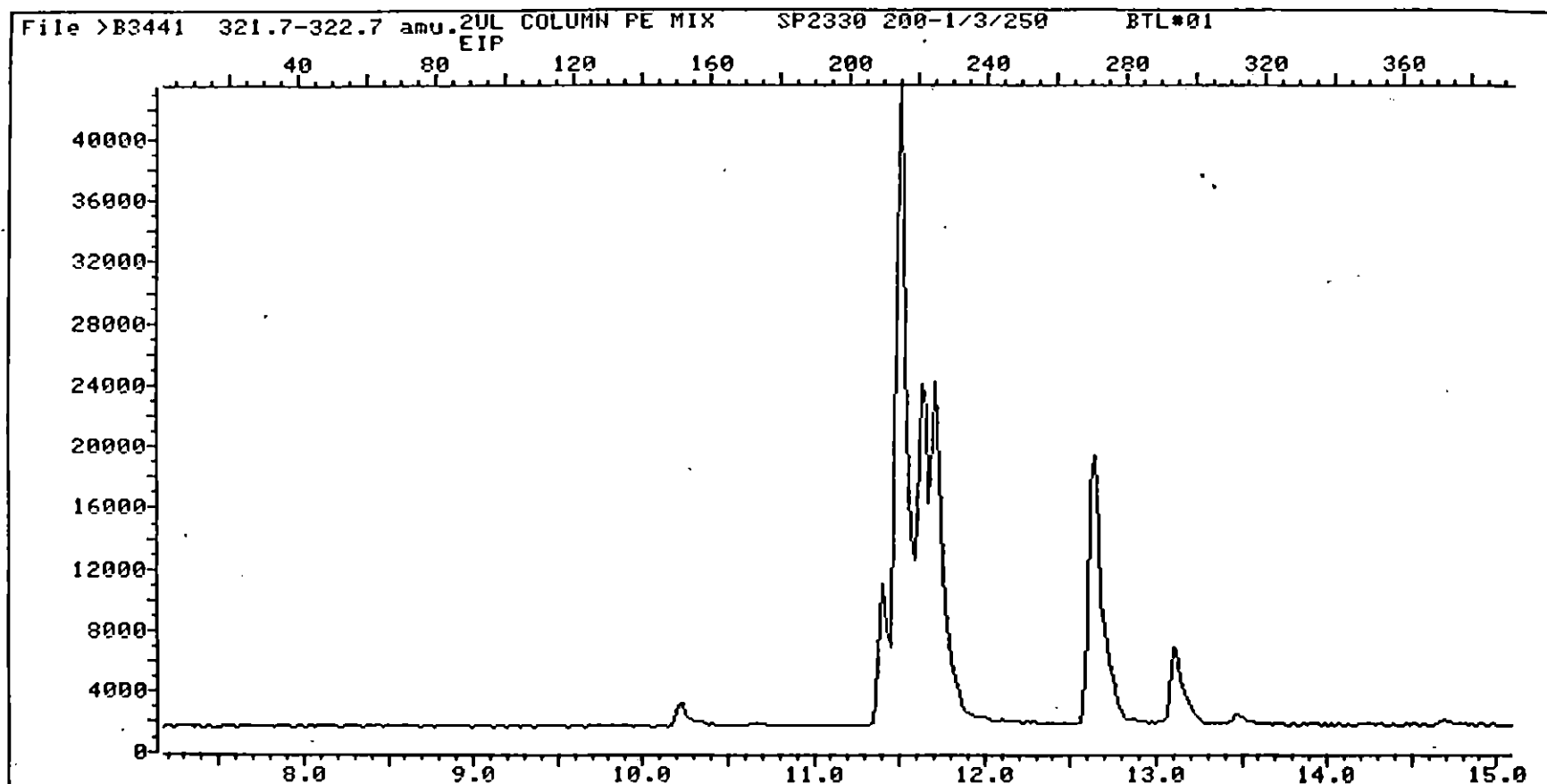
g (in)

Sample Identification

Comments

ALS Bottle Number

GC Conditions

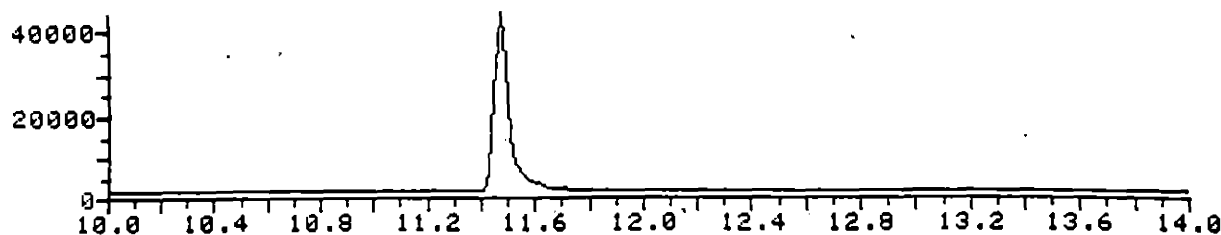


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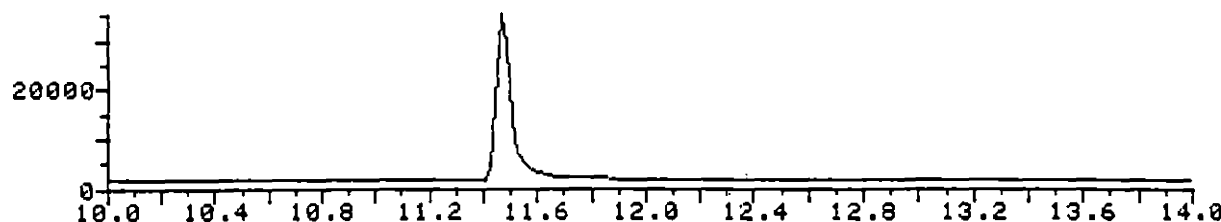
66017

C11075

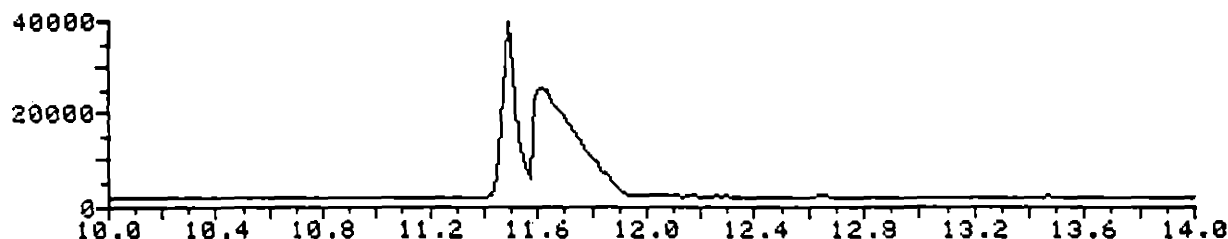
File >B3442 333.6-334.6 amu.2UL 0.1002/.48/.20 SP2330 200-1/3/250 BTL



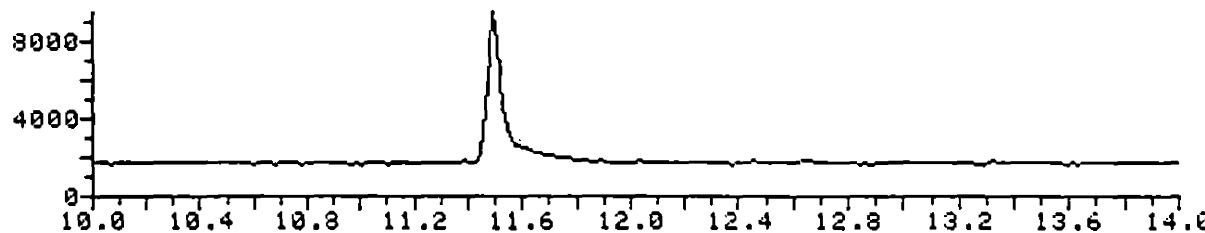
File >B3442 331.6-332.6 amu.2UL 0.1002/.48/.20 SP2330 200-1/3/250 BTL



File >B3442 327.6-328.6 amu.2UL 0.1002/.48/.20 SP2330 200-1/3/250 BTL



File >B3442 321.6-322.6 amu.2UL 0.1002/.48/.20 SP2330 200-1/3/250 BTL



File >B3442 319.6-320.6 amu.2UL 0.1002/.48/.20 SP2330 200-1/3/250 BTL



File >B3442 256.7-257.7 amu.2UL 0.1002/.48/.20 SP2330 200-1/3/250 BTL



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C11076

MS data file header from : >B3442

Sample: 2UL 0.1002/.48/.20 Operator: GARY MS 12/19/84-10r0r...
Misc : SP2330 200-1/3/250 BTL#03BTL# 3
Sys. #: 1 MS model: 87 SW/HW rev.: CA ALS #: 0
Method file: SP2330 Tuning file: APEI01 No. of extra records: 2
Source temp.: 250 Analyzer temp.: 0 Transfer line temp.: 0

Chromatographic temperatures : 0. 0. 0. 0. 0.
Chromatographic times, min. : 0.0 0.0 0.0 0.0 0.0
Chromatographic rate, deg/min: 0.0 0.0 0.0 0.0 0.0

M/Z = 333.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.469	212	215	226	14	160949	133551	100.00
2	11.469	212	215	226	14	160949	130401	97.64

M/Z = 331.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.469	211	215	230	19	146639	112933	100.00
2	11.469	211	215	230	19	146639	109522	96.98

332/334 CALCULATION

R-Global(0) = .8456

M/Z = 327.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.490	212	216	220	8	131339	115579	100.00
2	11.490	212	216	220	8	131339	99627	86.20

M/Z = 321.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.490	213	216	227	14	54304	29216	100.00
2	11.490	213	216	227	14	54304	26605	91.06

CONFIDENTIAL 00019

SUBJECT TO PROTECTIVE ORDER.

C11077

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> 03442

M/Z = 319.9

First peak is baseline drop corrected area.
 Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.490	212	216	224	12	43110	21558	100.00
2	11.490	212	216	224	12	43110	19806	91.87

320/322 CALCULATION

R-Global(1) = .7379

M/Z = 257.0

First peak is baseline drop corrected area.
 Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.510	213	217	226	13	40802	11393	100.00
2	11.510	213	217	226	13	40802	10980	96.46

(320 + 322)/(332 + 334) CALCULATION

R-Global(3) = .2060

(328)/(332 + 334) CALCULATION

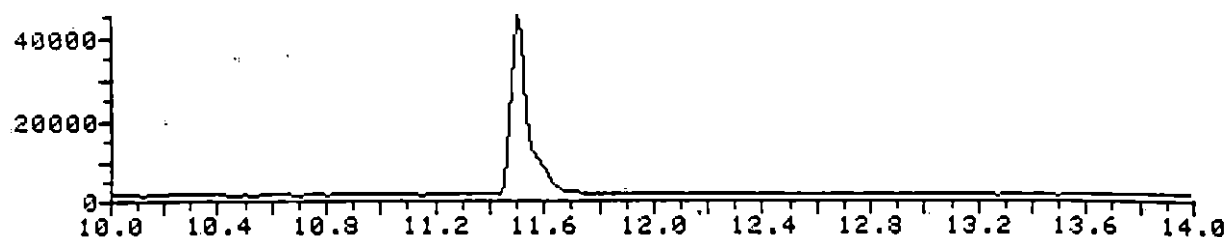
R-Global(3) = .4678

06020

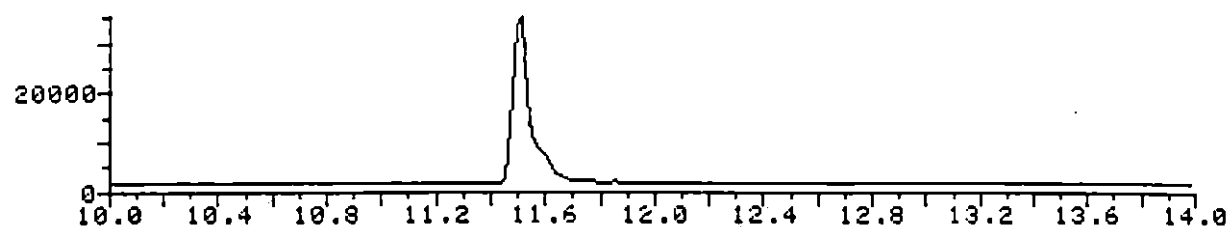
CONFIDENTIAL
 SUBJECT TO PROTECTIVE ORDER

C11078

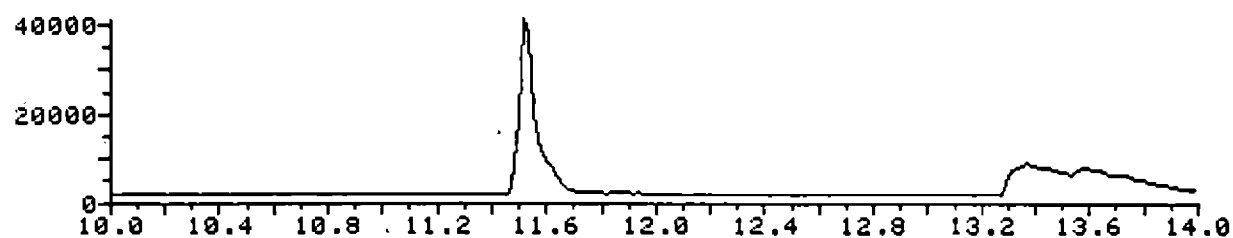
File >B3443 333.6-334.6 amu.2UL 1.002/.48/.20 SP2330 200-1/3/250 BTL...



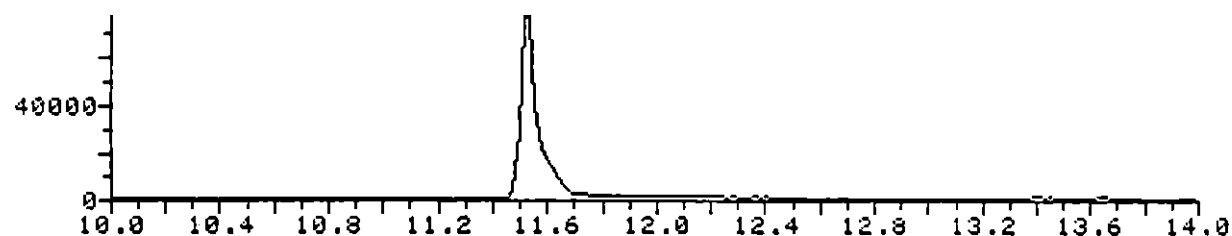
File >B3443 331.6-332.6 amu.2UL 1.002/.48/.20 SP2330 200-1/3/250 BTL...



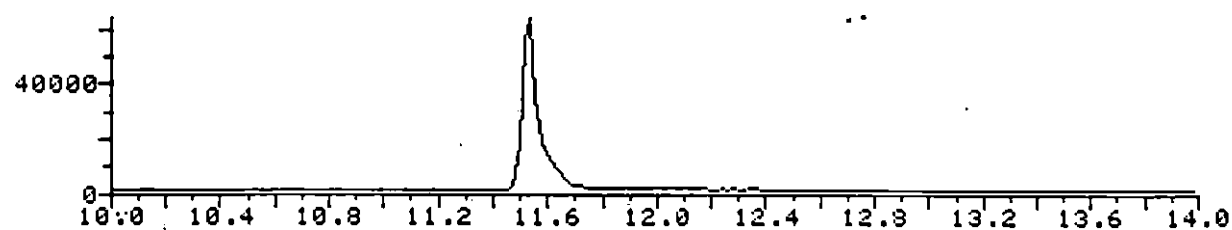
File >B3443 327.6-328.6 amu.2UL 1.002/.48/.20 SP2330 200-1/3/250 BTL...



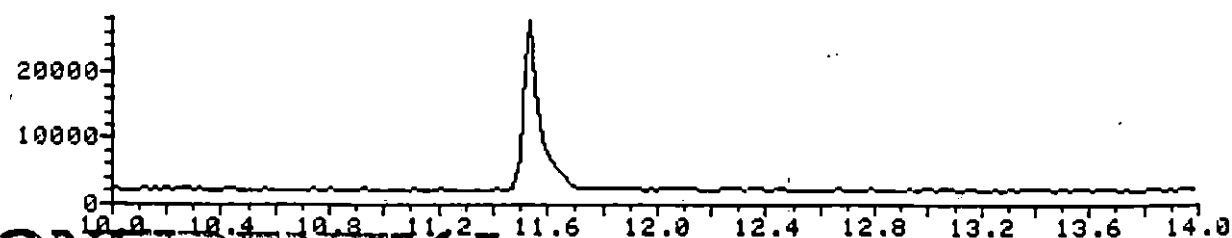
File >B3443 321.6-322.6 amu.2UL 1.002/.48/.20 SP2330 200-1/3/250 BTL...



File >B3443 319.6-320.6 amu.2UL 1.002/.48/.20 SP2330 200-1/3/250 BTL...



File >B3443 256.7-257.7 amu.2UL 1.002/.48/.20 SP2330 200-1/3/250 BTL...



CONFIDENTIAL

00021

C11079

SUBJECT TO PROTECTIVE ORDER

MS data file header from : >B3443

Sample: 2UL 1.002/.48/.20 Operator: GARY MS 12/19/84 10:21
Misc : SP2330 200-1/3/250 BTL#05BTL# 5
Sys. #: 1 MS-model: 87 SW/HW rev.: CA ALS # : 0
Method file: SP2330 Tuning file: APEI01 No. of extra records: 2
Source temp.: 250 Analyzer temp.: 0 Transfer line temp.: 0

Chromatographic temperatures : 0. 0. 0. 0. 0.
Chromatographic times, min. : 0.0 0.0 0.0 0.0 0.0
Chromatographic rate, deg/min: 0.0 0.0 0.0 0.0 0.0

M/Z = 333.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.495	208	213	225	17	190450	160598	100.00
2	11.495	208	213	225	17	190450	156654	97.54

M/Z = 331.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.515	210	214	225	15	154735	127510	100.00
2	11.515	210	214	225	15	154735	124562	97.69

332/334 CALCULATION

R-Global(0) = .7940

M/Z = 327.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.515	210	214	228	18	184240	149554	100.00
2	11.515	210	214	228	18	184240	146764	98.13

M/Z = 321.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.515	211	214	227	16	320991	290655	100.00
2	11.515	211	214	227	16	320991	284407	97.85

S

>B3443

M/Z = 319.9

First peak is baseline drop corrected area.
 Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.536	211	215	222	11	244228	223625	100.00
2	11.536	211	215	222	11	244228	210793	94.26

320/322 CALCULATION

R-Global(1) = .7694

M/Z = 257.0

First peak is baseline drop corrected area.
 Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.536	211	215	225	14	123296	92762	100.00
2	11.536	211	215	225	14	123296	89990	97.01

(320 + 322)/(332 + 334) CALCULATION

R-Global(3) = 1.7850

(328)/(332 + 334) CALCULATION

R-Global(3) = .5100

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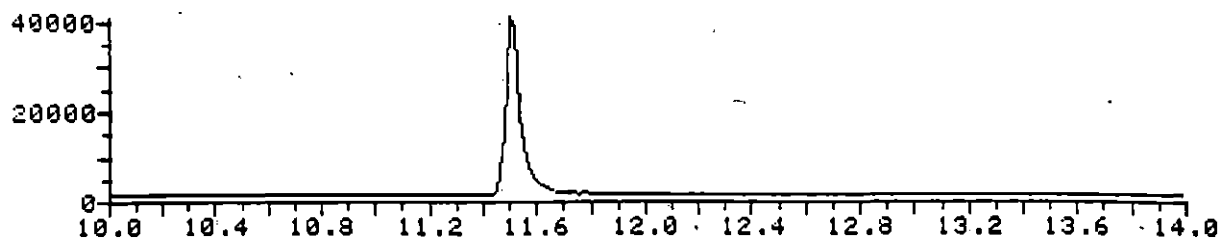
00023

C11081

File >B3444 333.6-334.6 amu.2UL UT-3267

SP2330 200-1/3/250

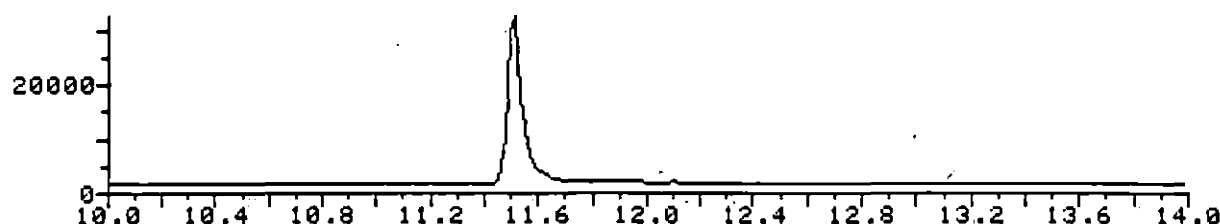
BTL



File >B3444 331.6-332.6 amu.2UL UT-3267

SP2330 200-1/3/250

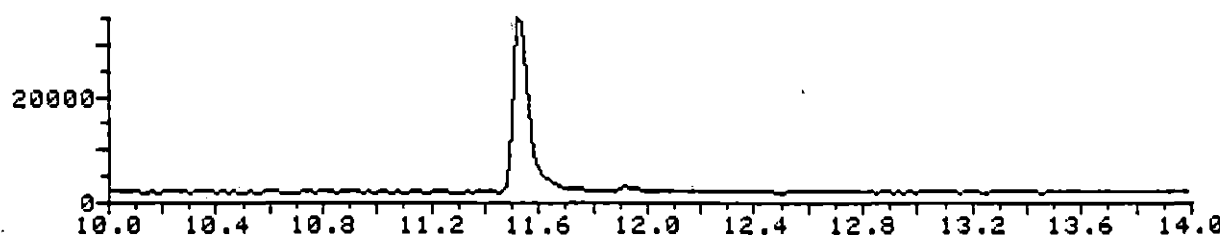
BTL



File >B3444 327.6-328.6 amu.2UL UT-3267

SP2330 200-1/3/250

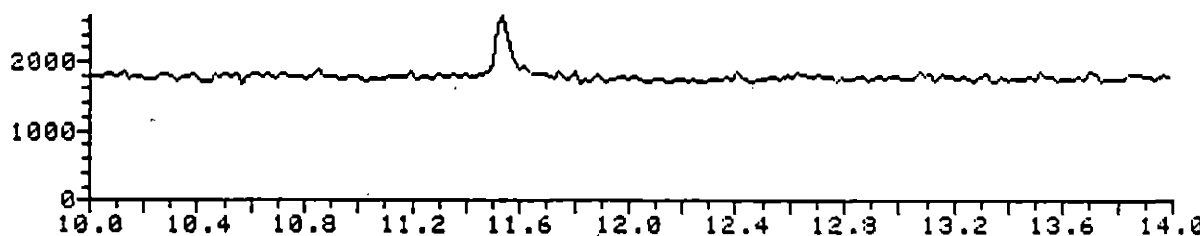
BTL



File >B3444 321.6-322.6 amu.2UL UT-3267

SP2330 200-1/3/250

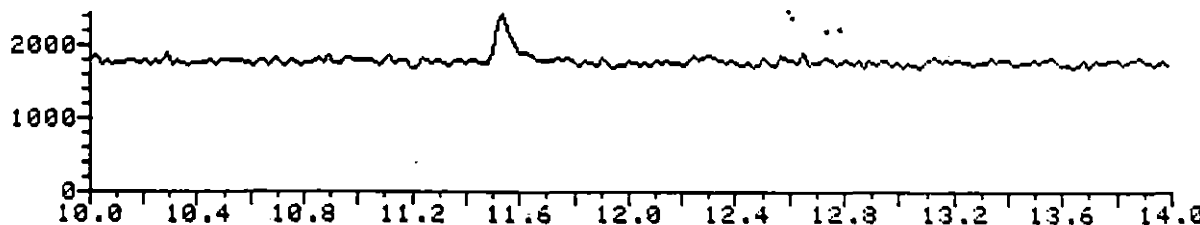
BTL



File >B3444 319.6-320.6 amu.2UL UT-3267

SP2330 200-1/3/250

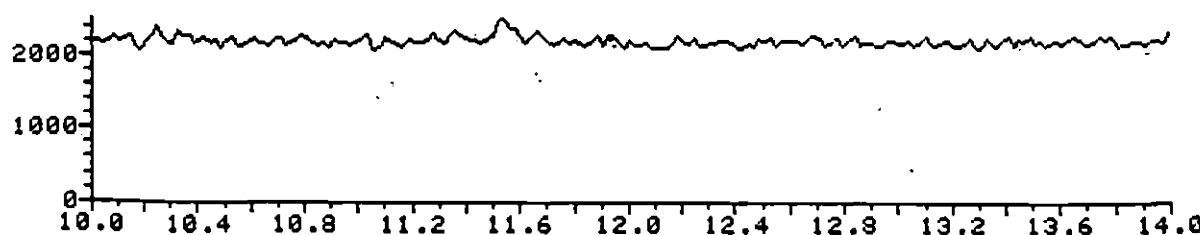
BTL



File >B3444 256.7-257.7 amu.2UL UT-3267

SP2330 200-1/3/250

BTL



00024

C11082

MS data file header from : >B3444

Sample: 2UL UT-3267 Operator: GARY MS 12/19/84 10:41 ..
Misc : SP2330 200-1/3/250 BTL#07BTL#7
Sys. #: 1 MS model: 87. SW/HW rev.: CA ALS # : 0
Method file: SP2330 Tuning file: APEI01 No. of extra records: 2
Source temp.: 250 Analyzer temp.: 0 Transfer line temp.: 0 ..

Chromatographic temperatures : 0. 0. 0. 0. 0.
Chromatographic times, min. : 0.0 0.0 0.0 0.0 0.0
Chromatographic rate, deg/min: 0.0 0.0 0.0 0.0 0.0

M/Z = 333.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.497	213	216	228	15	159473	132008	100.00
2	11.497	213	216	228	15	159473	129188	97.86

M/Z = 331.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.518	213	217	228	15	132464	105329	100.00
2	11.518	213	217	228	15	132464	103199	97.98

332/334 CALCULATION

R-Global(0) = .7979

M/Z = 327.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.518	214	217	229	15	148244	119699	100.00
2	11.518	214	217	229	15	148244	116189	97.07

M/Z = 321.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.538	212	218	227	15	31518	5238	100.00
2	11.538	212	218	227	15	31518	4953	94.56

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SUBJECT TO PROTECTIVE ORDER

00075

C11083

S

>B3444

M/Z = 319.9

First peak is baseline drop corrected area.
 Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.538	215	218	224	9	20140	4210	100.00
2	11.538	215	218	224	9	20140	4007	95.18

320/322 CALCULATION

R-Global(1) = .8037

M/Z = 257.0

First peak is baseline drop corrected area.
 Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.538	214	218	222	8	20846	3606	100.00
2	11.538	214	218	222	8	20846	3506	97.23

(320 + 322)/(332 + 334) CALCULATION

R-Global(3) = .0398

(328)/(332 + 334) CALCULATION

R-Global(3) = .5041

00026

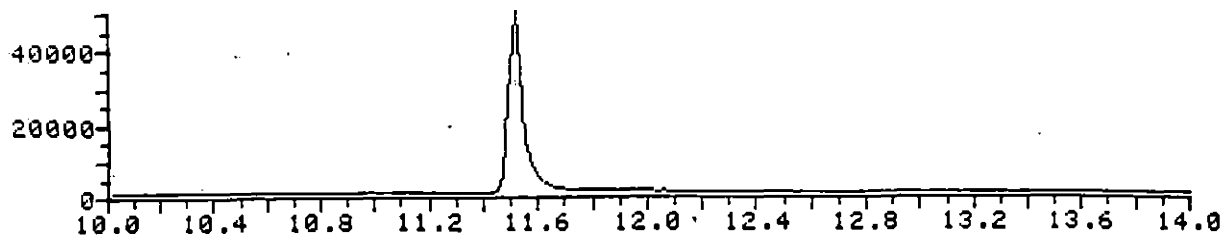
SUBJECT TO PROTECTIVE ORDER

C11084

File >B3445 333.6-334.6 amu.2UL UT-3268

SP2330 200-1/3/250

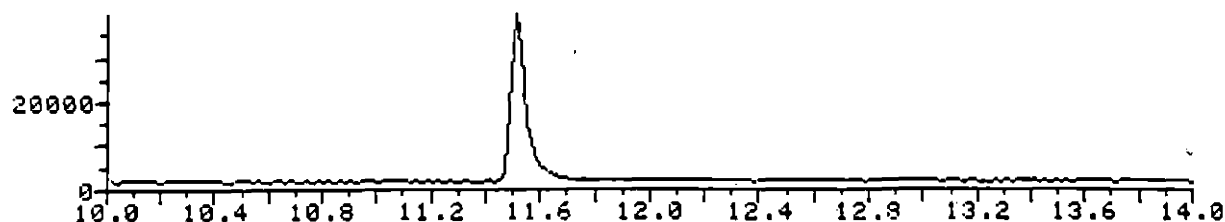
BTL



File >B3445 331.6-332.6 amu.2UL UT-3268

SP2330 200-1/3/250

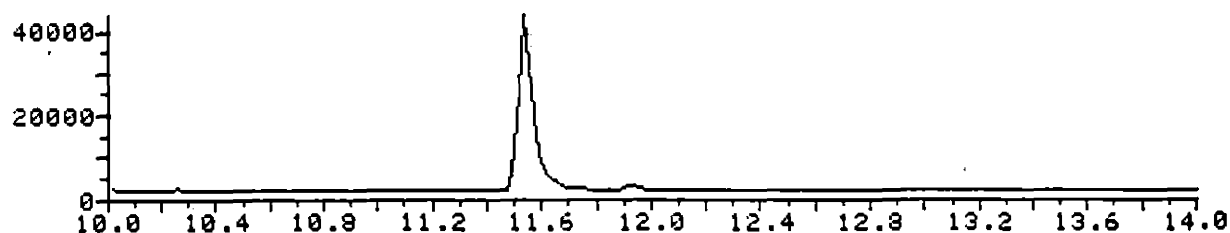
BTL



File >B3445 327.6-328.6 amu.2UL UT-3268

SP2330 200-1/3/250

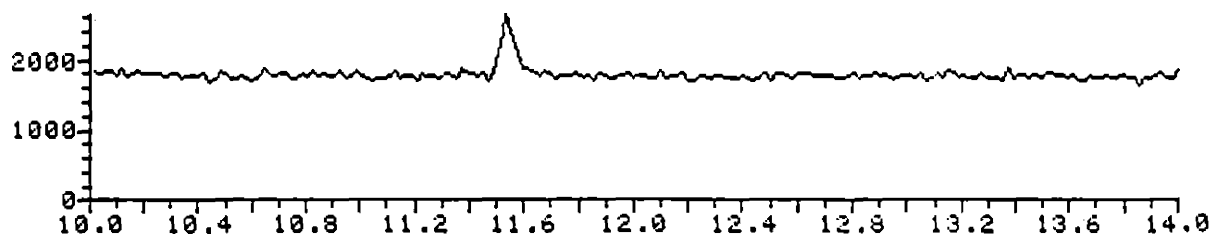
BTL



File >B3445 321.6-322.6 amu.2UL UT-3268

SP2330 200-1/3/250

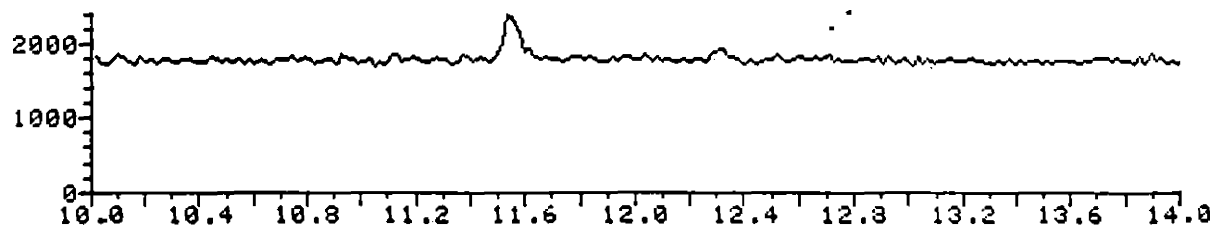
BTL



File >B3445 319.6-320.6 amu.2UL UT-3268

SP2330 200-1/3/250

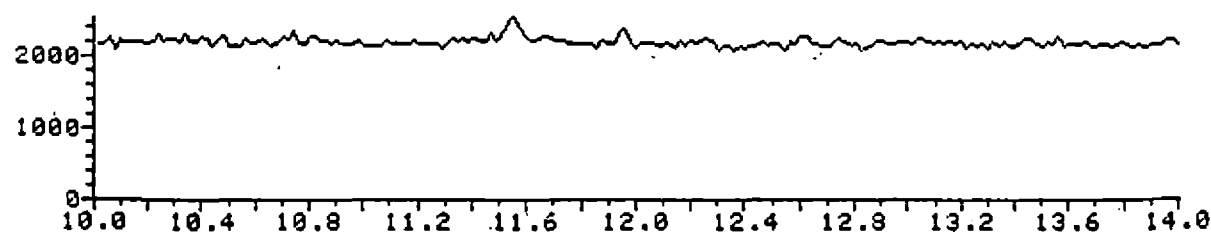
BTL



File >B3445 256.7-257.7 amu.2UL UT-3268

SP2330 200-1/3/250

BTL



CONFIDENTIAL 66027

SUBJECT TO PROTECTIVE ORDER.

C11085

MS data file header from : >B3445

Sample: 2UL UT-3268 Operator: GARY MS 12/19/84 11:01 ..
Misc : SP2330 200-1/3/250 BTL#09BTL# 9
Sys. #: 1 MS model: 87 SW/HW rev.: CR ALS #: 0
Method file: SP2330 Tuning file: APEI01 No. of extra records: 2
Source temp.: 250 Analyzer temp.: 0 Transfer line temp.: 0 ..

Chromatographic temperatures : 0. 0. 0. 0. 0.
Chromatographic times, min. : 0.0 0.0 0.0 0.0 0.0
Chromatographic rate, deg/min: 0.0 0.0 0.0 0.0 0.0

M/Z = 333.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.515	210	214	225	15	174963	149508	100.00
2	11.515	210	214	225	15	174963	144265	96.49

M/Z = 331.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.515	210	214	226	16	148514	121234	100.00
2	11.515	210	214	226	16	148514	117402	96.94

332/334 CALCULATION

R-Global(0) = .8109

M/Z = 327.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.535	212	215	226	14	160846	130144	100.00
2	11.535	212	215	226	14	160846	129772	98.95

M/Z = 321.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.535	212	215	221	9	20406	4881	100.00
2	11.535	212	215	221	9	20406	4570	93.63

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SUBJECT TO PROTECTIVE ORDER

00028

C11086

7B3445

S

M/Z = 319.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.535	212	215	220	8	18141	3981	100.00
2	11.535	212	215	220	8	18141	3793	95.28

320/322 CALCULATION

R-Global(1) = .8156

M/Z = 257.0

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.555	214	216	219	5	14230	3180	100.00
2	11.555	214	216	219	5	14230	2845	89.47

(320 + 322)/(332 + 334) CALCULATION

R-Global(3) = .0327

(328)/(332 + 334) CALCULATION

R-Global(3) = .4805

00029

CONFIDENTIAL

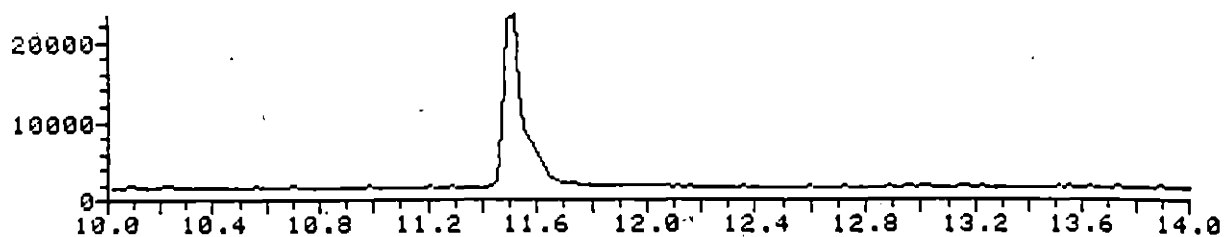
SUBJECT TO PROTECTIVE ORDER.

C11087

File >B3446 333.6-334.6 amu.2UL UT-3269

SP2330 200-1/3/250

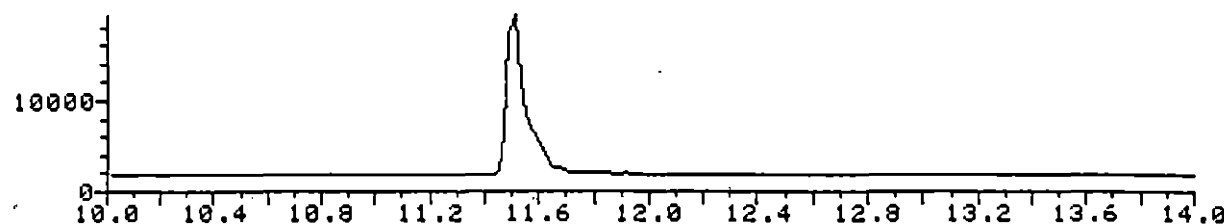
BTL



File >B3446 331.6-332.6 amu.2UL UT-3269

SP2330 200-1/3/250

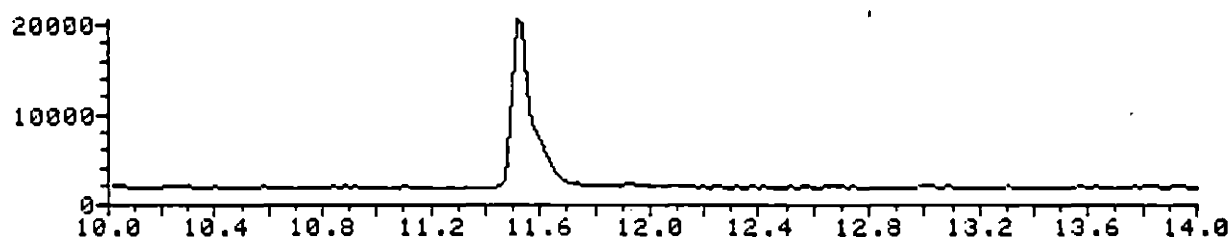
BTL



File >B3446 327.6-328.6 amu.2UL UT-3269

SP2330 200-1/3/250

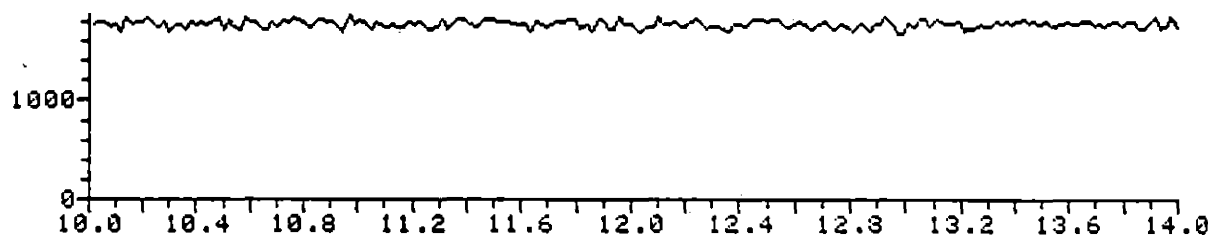
BTL



File >B3446 321.6-322.6 amu.2UL UT-3269

SP2330 200-1/3/250

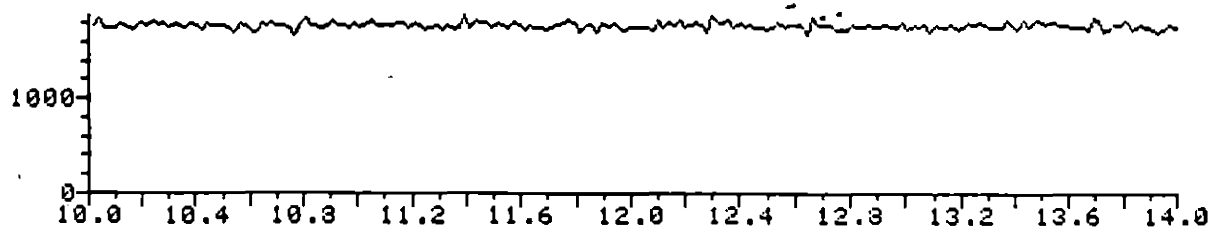
BTL



File >B3446 319.6-320.6 amu.2UL UT-3269

SP2330 200-1/3/250

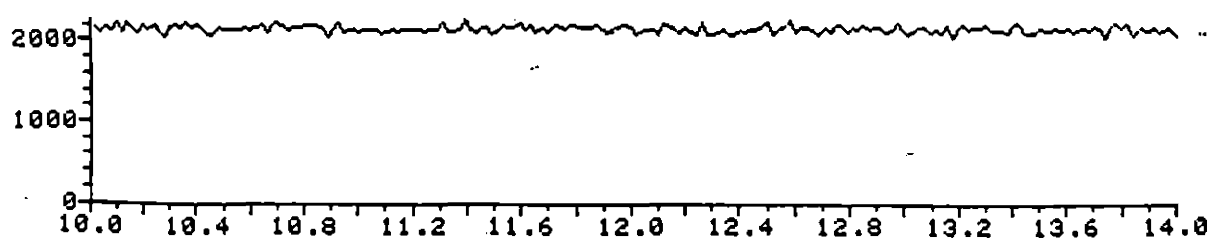
BTL



File >B3446 256.7-257.7 amu.2UL UT-3269

SP2330 200-1/3/250

BTL



06030

C11088

MS data file header from : >B3446

Sample: 2UL UT-3269 Operator: GARY MS 12/19/84 11:22 ..
Misc : SP2330 200-1/3/250 BTL#11BTL#11
Sys. #: 1 MS model: 87 SW/HW rev.: CA ALS #: 0
Method file: SP2330 Tuning file: APEI01 No. of extra records: 2
Source temp.: 248 Analyzer temp.: 0 Transfer line temp.: 0 ..

Chromatographic temperatures : 0. 0. 0. 0. 0.
Chromatographic times, min. : 0.0 0.0 0.0 0.0 0.0
Chromatographic rate, deg/min: 0.0 0.0 0.0 0.0 0.0

M/Z = 333.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.513	211	215	226	15	117442	90262	100.00
2	11.513	211	215	226	15	117442	87764	97.23

M/Z = 331.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.513	211	215	225	14	96885	72763	100.00
2	11.513	211	215	225	14	96885	70012	96.22

332/334 CALCULATION

R-Global(0) = .8061

M/Z = 327.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.513	211	215	227	16	112326	82406	100.00
2	11.513	211	215	227	16	112326	79942	97.01

M/Z = 321.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	0.000	228	0	227	-1	0	1803	100.00
2	0.000	228	0	227	-1	0	1803	100.00

CONFIDENTIAL 00031
SUBJECT TO PROTECTIVE ORDER.

C11089

S

7B 3446

M/Z = 319.9

First peak is baseline drop corrected area.
 Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	0.000	228	0	227	-1	0	1809	100.00
2	0.000	228	0	227	-1	0	1809	100.00

320/322 CALCULATION

R-Global(1) = 1.0033

M/Z = 257.0

First peak is baseline drop corrected area.
 Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	0.000	228	0	227	-1	0	2153	100.00
2	0.000	228	0	227	-1	0	2153	100.00

(320 + 322)/(332 + 334) CALCULATION

R-Global(3) = .0222

(328)/(332 + 334) CALCULATION

R-Global(3) = .5054

06032

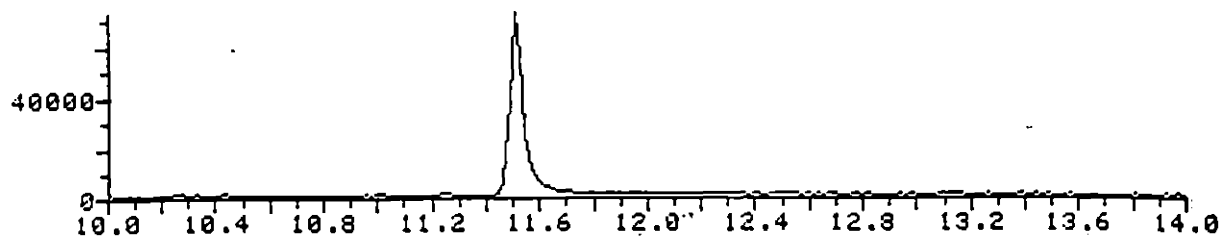
SUBJECT TO REVIEW

C11090

File >B3447 333.6-334.6 amu.2UL UT-3270

SP2330 200-1/3/250

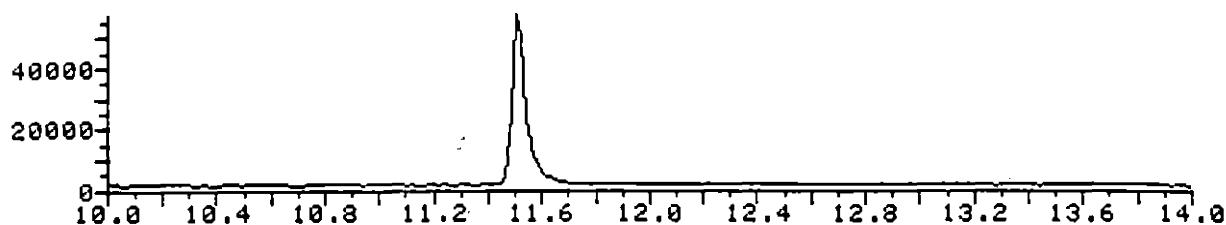
BTL



File >B3447 331.6-332.6 amu.2UL UT-3270

SP2330 200-1/3/250

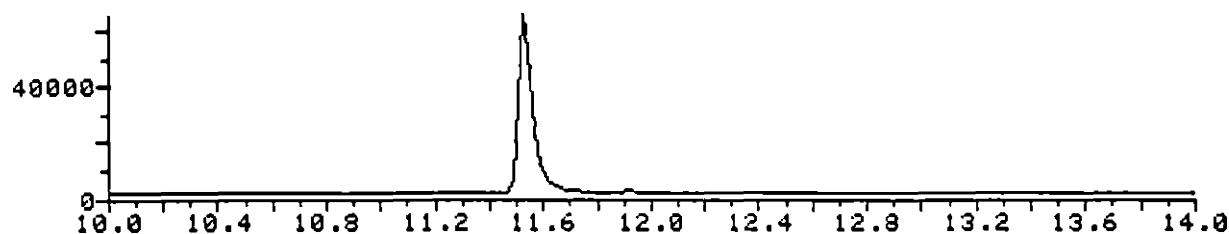
BTL



File >B3447 327.6-328.6 amu.2UL UT-3270

SP2330 200-1/3/250

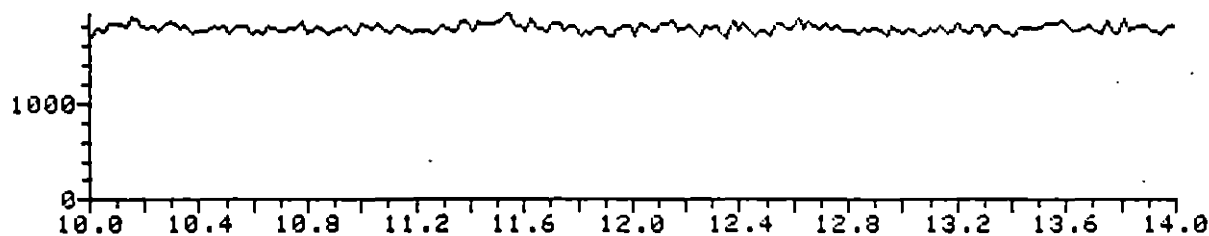
BTL



File >B3447 321.6-322.6 amu.2UL UT-3270

SP2330 200-1/3/250

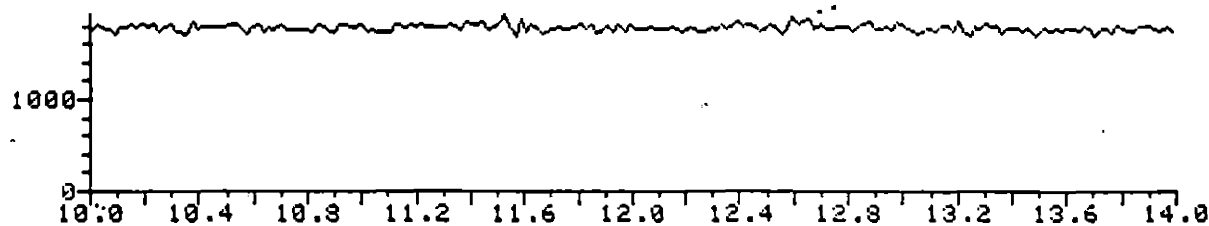
BTL



File >B3447 319.6-320.6 amu.2UL UT-3270

SP2330 200-1/3/250

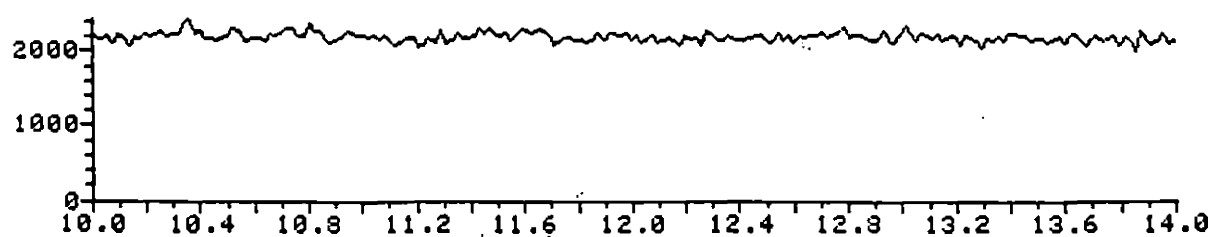
BTL



File >B3447 256.7-257.7 amu.2UL UT-3270

SP2330 200-1/3/250

BTL



CONFIDENTIAL 00033

SUBJECT TO PROTECTIVE ORDER.

C11091

MS data file header from : >B3447

Sample: 2UL UT-3270 Operator: GARY MS 12/19/84 11:42
Misc : SP2330 200-1/3/250 BTL#13BTL#13
Sys. #: 1 MS model: 87 SW/HW rev.: CA ALS #: 0
Method file: SP2330 Tuning file: APEI01 No. of extra records: 2
Source temp.: 250 Analyzer temp.: 0 Transfer line temp.: 0

Chromatographic temperatures : 0. 0. 0. 0. 0.
Chromatographic times, min. : 0.0 0.0 0.0 0.0 0.0
Chromatographic rate, deg/min: 0.0 0.0 0.0 0.0 0.0

M/Z = 333.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.506	210	214	224	14	247707	222731	100.00
2	11.506	210	214	224	14	247707	215430	96.72

M/Z = 331.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.506	211	214	225	14	204168	177694	100.00
2	11.506	211	214	225	14	204168	173949	97.89

332/334 CALCULATION

R-Global(0) = .7978

M/Z = 327.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.526	211	215	225	14	230609	202749	100.00
2	11.526	211	215	225	14	230609	197590	97.46

M/Z = 321.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	0.000	226	0	225	-1	0	1785	100.00
2	0.000	226	0	225	-1	0	1785	100.00

00034

C11092

S

M/Z = 319.9

703447

First peak is baseline drop corrected area.
 Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	0.000	226	0	225	-1	0	1772	100.00
2	0.000	226	0	225	-1	0	1772	100.00

320/322 CALCULATION

R-Global(1) = .9924

M/Z = 257.0

First peak is baseline drop corrected area.
 Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	0.000	226	0	225	-1	0	2151	100.00
2	0.000	226	0	225	-1	0	2151	100.00

(320 + 322)/(332 + 334) CALCULATION

R-Global(3) = .0089

(328)/(332 + 334) CALCULATION

R-Global(3) = .5063

CONFIDENTIAL 00035

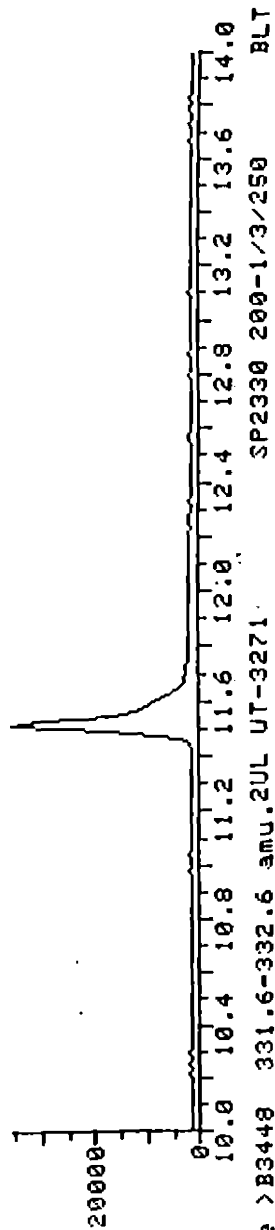
SUBJECT TO PROTECTIVE ORDER

C11093

File >B3448 333.6-334.6 amu.2UL UT-3271

SP2330 200-1/3/250

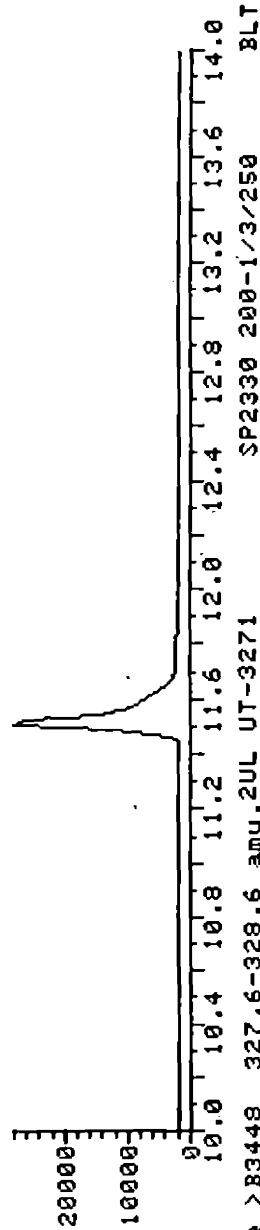
BLT



File >B3448 331.6-332.6 amu.2UL UT-3271

SP2330 200-1/3/250

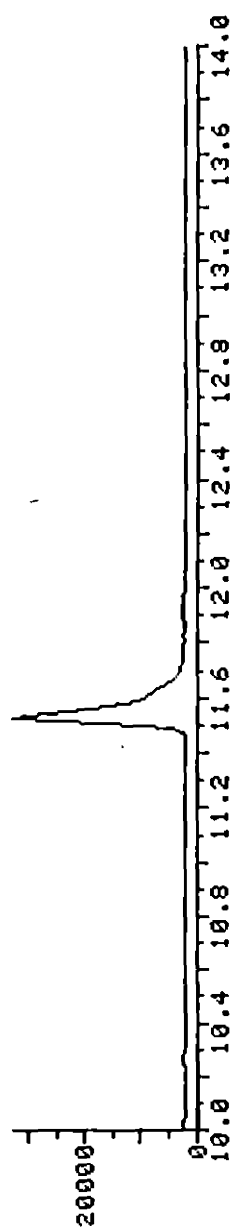
BLT



File >B3448 327.6-328.6 amu.2UL UT-3271

SP2330 200-1/3/250

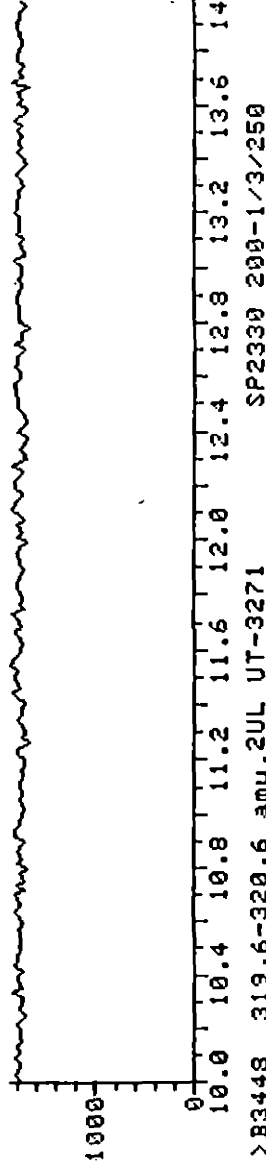
BLT



File >B3448 321.6-322.6 amu.2UL UT-3271

SP2330 200-1/3/250

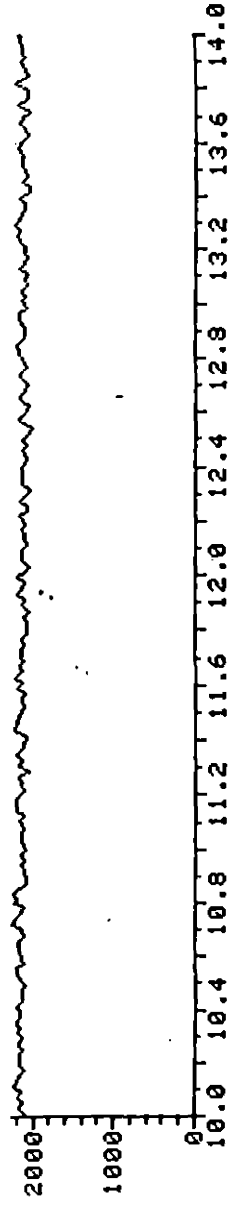
BLT



File >B3448 256.7-257.7 amu.2UL UT-3271

SP2330 200-1/3/250

BLT



00036

C11094

MS data file header from : >B3448

Sample: 2UL UT-3271 Operator: GARY MS 12/19/84 12:02
Misc : SP2330 200-1/3/250 BLT#15BTL#15
Sys. #: 1 MS model: 87 SW/HW rev.: CA ALS #: 0
Method file: SP2330 Tuning file: APEI01 No. of extra records: 2
Source temp.: 250 Analyzer temp.: 0 Transfer line temp.: 0

Chromatographic temperatures : 0. 0. 0. 0. 0.
Chromatographic times, min. : 0.0 0.0 0.0 0.0 0.0
Chromatographic rate, deg/min: 0.0 0.0 0.0 0.0 0.0

M/Z = 333.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.506	210	214	225	15	155877	129927	100.00
2	11.506	210	214	225	15	155877	125599	96.67

M/Z = 331.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.506	211	214	226	15	131604	104544	100.00
2	11.506	211	214	226	15	131604	101559	97.14

332/334 CALCULATION

R-Global(0) = .8046

M/Z = 327.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.526	212	215	226	14	145349	115823	100.00
2	11.526	212	215	226	14	145349	113282	97.81

M/Z = 321.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	0.000	227	0	226	-1	0	1736	100.00
2	0.000	227	0	226	-1	0	1736	100.00

CONFIDENTIAL
SUBJECT TO PROTECTIVE ORDER

00037

C11095

S

M/Z = 319.9

7B3448

First peak is baseline drop corrected area.
 Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	0.000	227	0	226	-1	0	1772	100.00
2	0.000	227	0	226	-1	0	1772	100.00

320/322 CALCULATION

R-Global(1) = 1.0207

M/Z = 257.0

First peak is baseline drop corrected area.
 Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	0.000	227	0	226	-1	0	2131	100.00
2	0.000	227	0	226	-1	0	2131	100.00

(320 + 322)/(332 + 334) CALCULATION

R-Global(3) = .0150

(328)/(332 + 334) CALCULATION

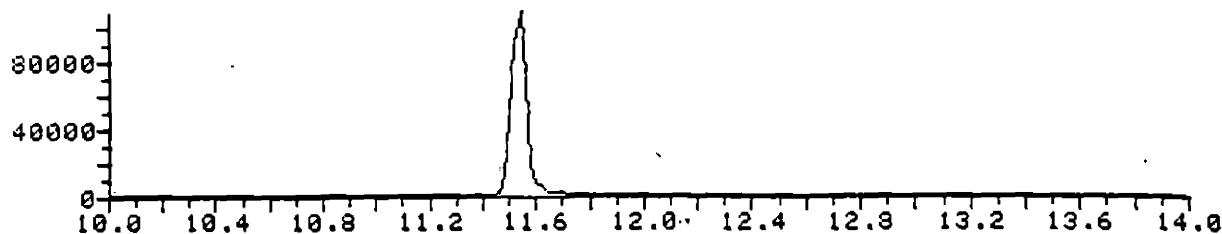
R-Global(3) = .4939

00038

C11096

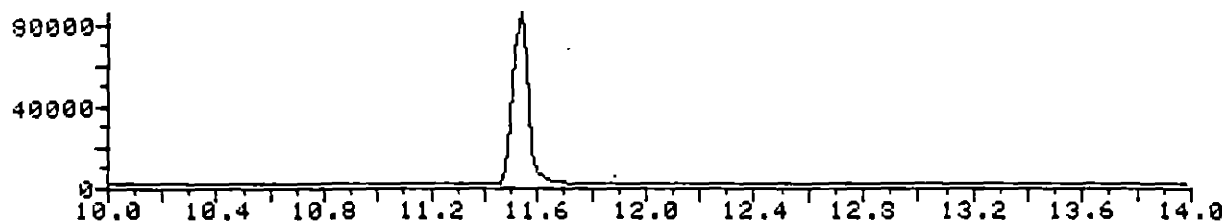
File >B3449 333.6-334.6 amu.2UL 10.02/.48/.20 SP2330 200-1/3/250

BTL...



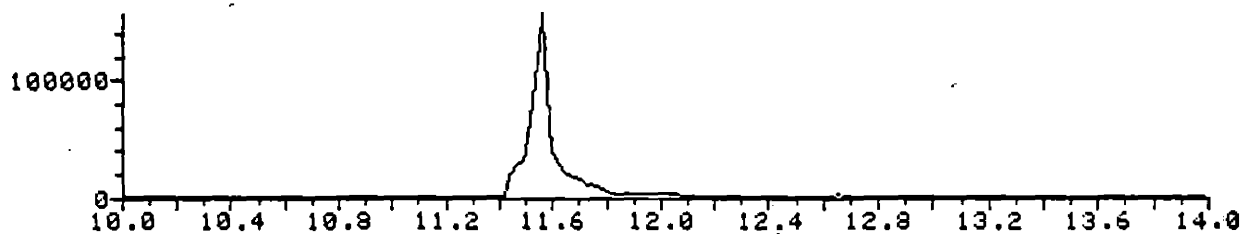
File >B3449 331.6-332.6 amu.2UL 10.02/.48/.20 SP2330 200-1/3/250

BTL...



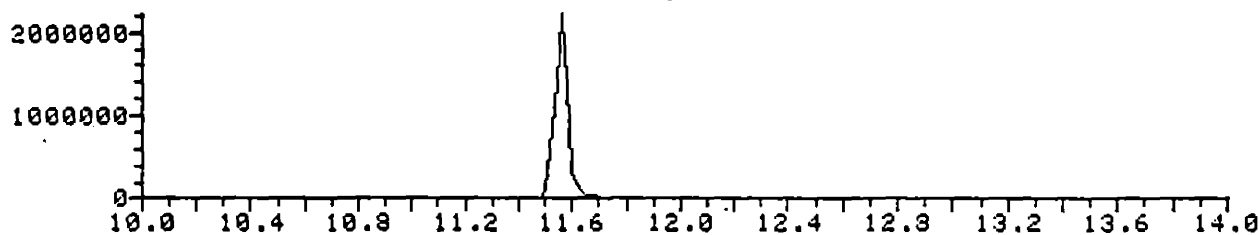
File >B3449 327.6-328.6 amu.2UL 10.02/.48/.20 SP2330 200-1/3/250

BTL



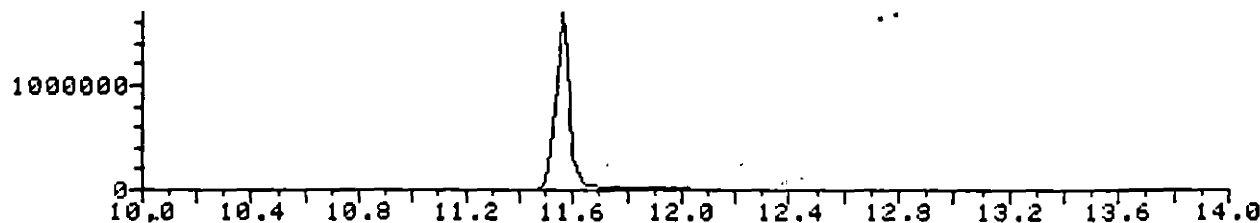
File >B3449 321.6-322.6 amu.2UL 10.02/.48/.20 SP2330 200-1/3/250

BTL...



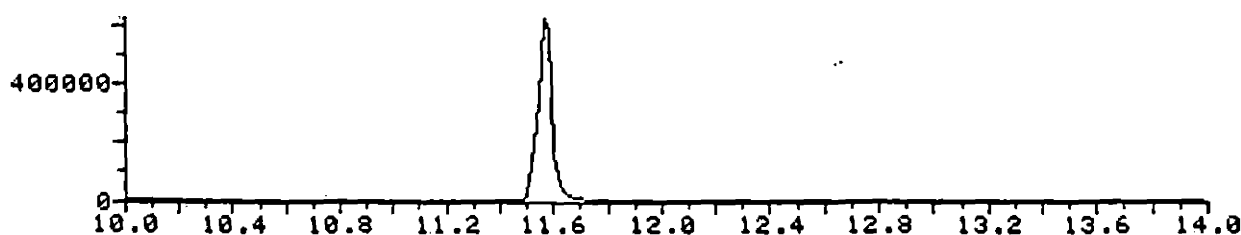
File >B3449 319.6-320.6 amu.2UL 10.02/.48/.20 SP2330 200-1/3/250

BTL...



File >B3449 256.7-257.7 amu.2UL 10.02/.48/.20 SP2330 200-1/3/250

BTL...



CONFIDENTIAL

00039

C11097

SUBJECT TO PROTECTIVE ORDER

MS data file header from : >B3449

Sample: 2UL 10.02/.48/.20 Operator: GARY MS 12/19/84 12:22
Misc : SP2330 200-1/3/250 BTL#17BTL#17
Sys. #: 1 MS model: 87 SW/HW rev.: CA ALS #: 0
Method file: SP2330 Tuning file: APEI01 No. of extra records: 2
Source temp.: 250 Analyzer temp.: 0 Transfer line temp.: 0

Chromatographic temperatures : 0. 0. 0. 0. 0.
Chromatographic times, min. : 0.0 0.0 0.0 0.0 0.0
Chromatographic rate, deg/min: 0.0 0.0 0.0 0.0 0.0

M/Z = 333.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.540	213	219	226	13	375586	352524	100.00
2	11.540	213	219	226	13	375586	344119	97.62

M/Z = 331.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.540	213	219	228	15	311230	285295	100.00
2	11.540	213	219	228	15	311230	279340	97.91

332/334 CALCULATION

R-Global(0) = .8093

M/Z = 327.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.560	213	220	225	12	659448	635088	100.00
2	11.560	213	220	225	12	659448	530627	83.55

M/Z = 321.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.560	215	220	227	12	6397695	6369783	100.00
2	11.560	215	220	227	12	6397695	6229515	97.80

00040

C11098

S

7B 3449

M/Z = 319.9

First peak is baseline drop corrected area.
 Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.560	216	220	228	12	5116912	5025148	100.00
2	11.560	216	220	228	12	5116912	4966198	98.83

320/322 CALCULATION

R-Global(1) = .7889

M/Z = 257.0

First peak is baseline drop corrected area.
 Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.560	216	220	230	14	2017666	1969254	100.00
2	11.560	216	220	230	14	2017666	1946945	98.87

(320 + 322)/(332 + 334) CALCULATION

R-Global(3) = 17.8655

(328)/(332 + 334) CALCULATION

R-Global(3) = .9058

CONFIDENTIAL
 SUBJECT TO PROTECTIVE ORDER.

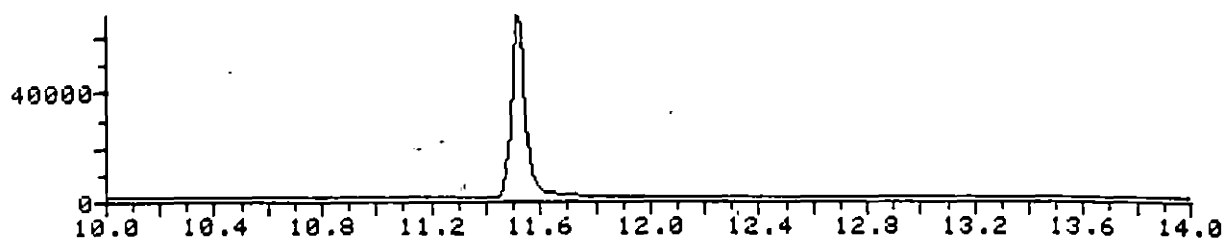
00041

C11099

File >B3450 333.6-334.6 amu.2UL UT-3272

SP2330 200-1/3/250

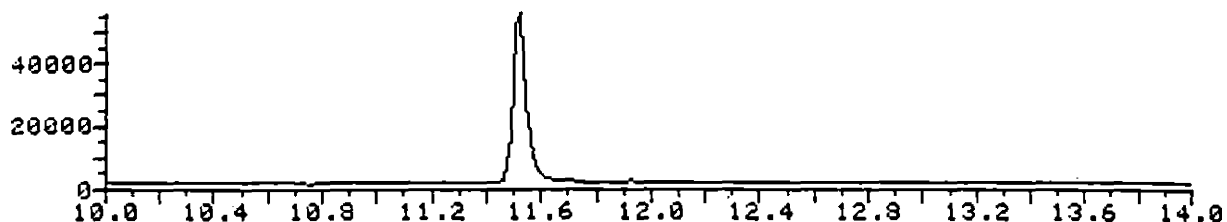
BTL



File >B3450 331.6-332.6 amu.2UL UT-3272

SP2330 200-1/3/250

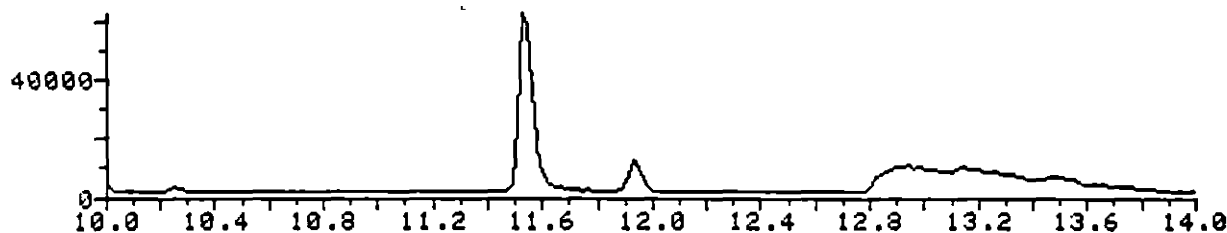
BTL



File >B3450 327.6-328.6 amu.2UL UT-3272

SP2330 200-1/3/250

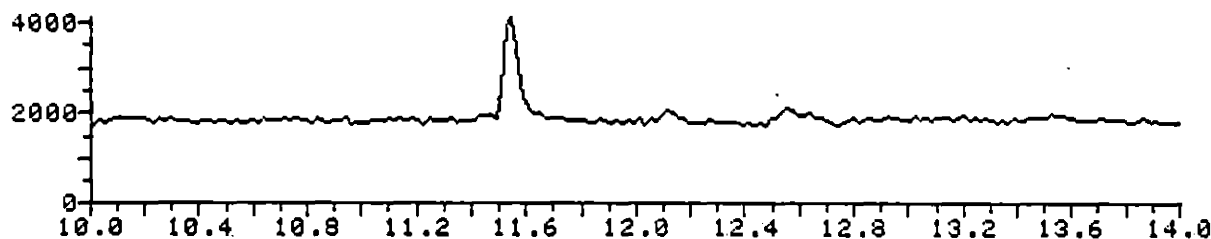
BTL



File >B3450 321.6-322.6 amu.2UL UT-3272

SP2330 200-1/3/250

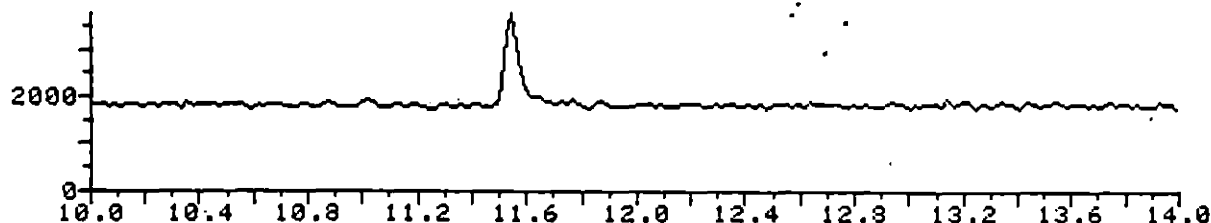
BTL



File >B3450 319.6-320.6 amu.2UL UT-3272

SP2330 200-1/3/250

BTL



File >B3450 256.7-257.7 amu.2UL UT-3272

SP2330 200-1/3/250

BTL



LOG042

SUBJECT

C11100

MS data file header from : >B3450

Sample: 2UL UT-3272 Operator: GARY MS 12/19/84 12:42 ..
Misc : SP2330 200-1/3/250 BTL#19BTL#19
Sys. #: 1 MS model: 87 SW/HW rev.: CA ALS #: 0
Method file: SP2330 Tuning file: APEI01 No. of extra records: 2
Source temp.: 250 Analyzer temp.: 0 Transfer line temp.: 0 ..

Chromatographic temperatures : 0. 0. 0. 0. 0.
Chromatographic times, min. : 0.0 0.0 0.0 0.0 0.0
Chromatographic rate, deg/min: 0.0 0.0 0.0 0.0 0.0

M/Z = 333.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.506	210	214	225	15	238902	213347	100.00
2	11.506	210	214	225	15	238902	206537	96.91

M/Z = 331.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.527	210	215	226	16	199520	170592	100.00
2	11.527	210	215	226	16	199520	166048	97.34

332/334 CALCULATION

R-Global(0) = .7996

M/Z = 327.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.527	212	215	225	13	220262	192520	100.00
2	11.527	212	215	225	13	220262	188425	97.87

M/Z = 321.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.547	213	216	222	9	25907	8978	100.00
2	11.547	213	216	222	9	25907	8789	97.89

CONFIDENTIAL

00043

SUBJECT TO PROTECTIVE ORDER.

C11101

S

703450

M/Z = 319.9

First peak is baseline drop corrected area.
 Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.547	213	216	223	10	26183	7473	100.00
2	11.547	213	216	223	10	26183	7303	97.73

320/322 CALCULATION

R-Global(1) = .8324

M/Z = 257.0

First peak is baseline drop corrected area.
 Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.547	214	216	221	7	20443	4868	100.00
2	11.547	214	216	221	7	20443	4388	90.14

(320 + 322)/(332 + 334) CALCULATION

R-Global(3) = .0428

(328)/(332 + 334) CALCULATION

R-Global(3) = .5012

00044

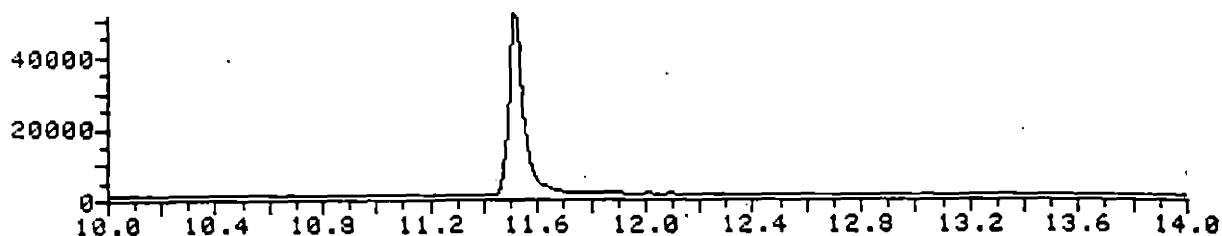
C11102

SUB

File >B3451 333.6-334.6 amu.2UL UT-3273

SP2330 200-1/3/250

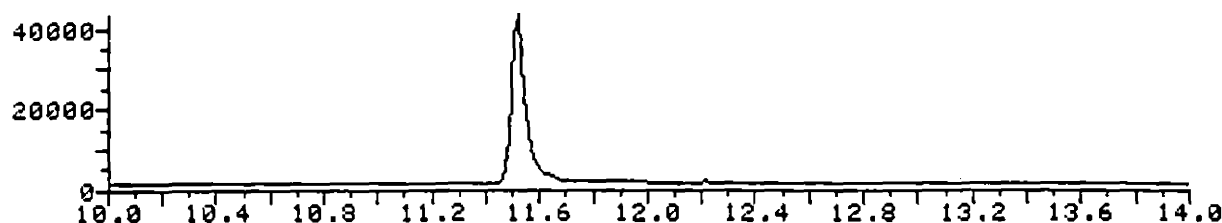
BTL



File >B3451 331.6-332.6 amu.2UL UT-3273

SP2330 200-1/3/250

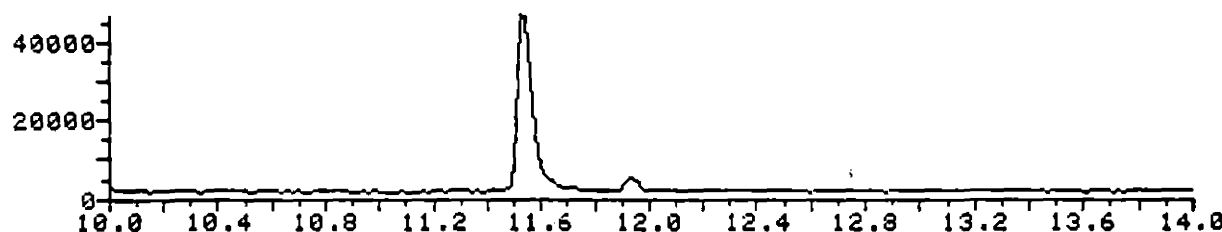
BTL



File >B3451 327.6-328.6 amu.2UL UT-3273

SP2330 200-1/3/250

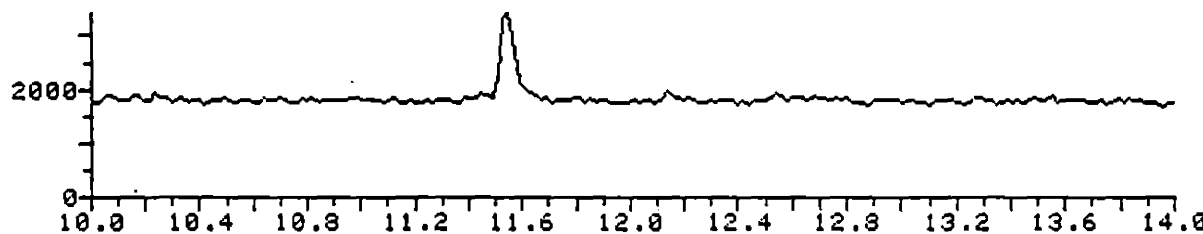
BTL



File >B3451 321.6-322.6 amu.2UL UT-3273

SP2330 200-1/3/250

BTL



File >B3451 319.6-320.6 amu.2UL UT-3273

SP2330 200-1/3/250

BTL



File >B3451 256.7-257.7 amu.2UL UT-3273

SP2330 200-1/3/250

BTL



CONFIDENTIAL

SUBJECT TO PROTECTIVE ORDER

00045

C11103

MS data file header from : >B3451

Sample: 2UL UT-3273 Operator: GARY MS 12/19/84 13:02 ..
Misc : SP2330 200-1/3/250 BTL#21BTL#21
Sys. #: 1 MS model: 87 SW/HW rev.: CA ALS # : 0
Method file: SP2330 Tuning file: APEI01 No. of extra records: 2
Source temp.: 250 Analyzer temp.: 0 Transfer line temp.: 0 ..

Chromatographic temperatures : 0. 0. 0. 0. 0.
Chromatographic times, min. : 0.0 0.0 0.0 0.0 0.0
Chromatographic rate, deg/min: 0.0 0.0 0.0 0.0 0.0

M/Z = 333.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.508	214	217	228	14	191195	166051	100.00
2	11.508	214	217	228	14	191195	161459	97.23

M/Z = 331.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.528	214	218	228	14	159669	134805	100.00
2	11.528	214	218	228	14	159669	130794	97.02

332/334 CALCULATION

R-Global(0) = .8118

M/Z = 327.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.528	215	218	228	13	178652	151482	100.00
2	11.528	215	218	228	13	178652	147855	97.61

M/Z = 321.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.548	216	219	227	11	26941	7449	100.00
2	11.548	216	219	227	11	26941	7080	95.05

CONFIDENTIAL
SUBJECT TO PROTECTIVE ORDER

00046

C11104

S

783451

M/Z = 319.9

First peak is baseline drop corrected area.
 Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.548	216	219	225	9	22125	5934	100.00
2	11.548	216	219	225	9	22125	5745	96.81

320/322 CALCULATION

R-Global(1) = .7966

M/Z = 257.0

First peak is baseline drop corrected area.
 Second peak is tangent skimmed corrected area.

Peak #	R.T. min.	first scan	max scan	last scan	peak width	raw area	corr. area	corr. %
1	11.548	216	219	223	7	19363	3984	100.00
2	11.548	216	219	223	7	19363	3781	94.90

(320 + 322)/(332 + 334) CALCULATION

R-Global(3) = .0445

(328)/(332 + 334) CALCULATION

R-Global(3) = .5033

SUBJECT TO PROTECTIVE ORDER

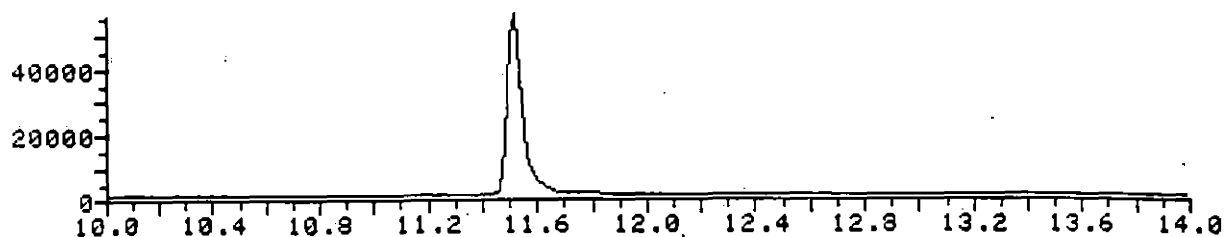
00007

C11105

File >B3452 333.6-334.6 amu.2UL UT-3274

SP2330 200-1/3/250

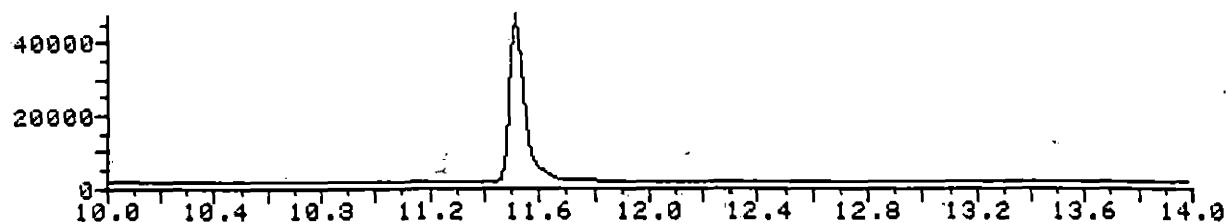
BTL



File >B3452 331.6-332.6 amu.2UL UT-3274

SP2330 200-1/3/250

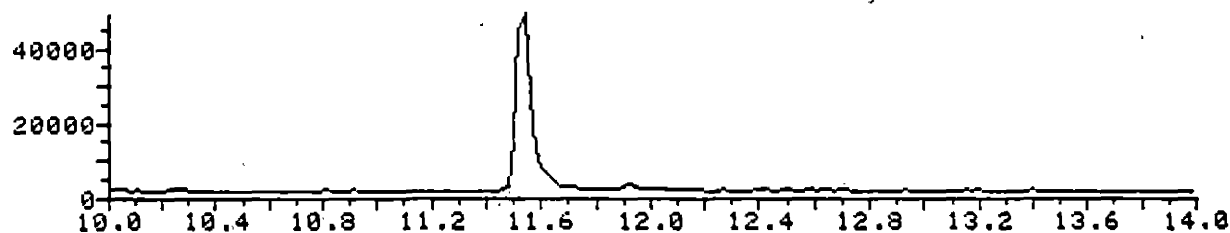
BTL



File >B3452 327.6-328.6 amu.2UL UT-3274

SP2330 200-1/3/250

BTL



File >B3452 321.6-322.6 amu.2UL UT-3274

SP2330 200-1/3/250

BTL



File >B3452 319.6-320.6 amu.2UL UT-3274

SP2330 200-1/3/250

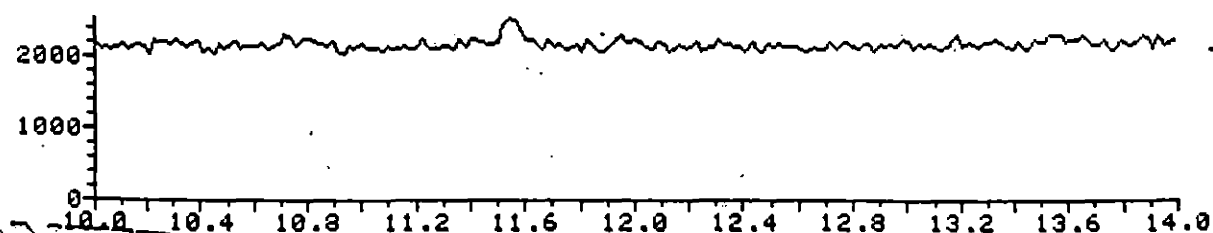
BTL



File >B3452 256.7-257.7 amu.2UL UT-3274

SP2330 200-1/3/250

BTL



CONFIDENTIAL 66748
SUBJECT TO PROTECTIVE ORDER.

C11106

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1. 2. 3. 4. 5. 6. 7. 8. 9. 10. 11. 12. 13. 14. 15. 16. 17. 18. 19. 20. 21. 22. 23. 24. 25. 26. 27. 28. 29. 30. 31. 32. 33. 34. 35. 36. 37. 38. 39. 40. 41. 42. 43. 44. 45. 46. 47. 48. 49. 50. 51. 52. 53. 54. 55. 56. 57. 58. 59. 60. 61. 62. 63. 64. 65. 66. 67. 68. 69. 70. 71. 72. 73. 74. 75. 76. 77. 78. 79. 80. 81. 82. 83. 84. 85. 86. 87. 88. 89. 90. 91. 92. 93. 94. 95. 96. 97. 98. 99. 100. 101. 102. 103. 104. 105. 106. 107. 108. 109. 110. 111. 112. 113. 114. 115. 116. 117. 118. 119. 120. 121. 122. 123. 124. 125. 126. 127. 128. 129. 130. 131. 132. 133. 134. 135. 136. 137. 138. 139. 140. 141. 142. 143. 144. 145. 146. 147. 148. 149. 150. 151. 152. 153. 154. 155. 156. 157. 158. 159. 160. 161. 162. 163. 164. 165. 166. 167. 168. 169. 170. 171. 172. 173. 174. 175. 176. 177. 178. 179. 180. 181. 182. 183. 184. 185. 186. 187. 188. 189. 190. 191. 192. 193. 194. 195. 196. 197. 198. 199. 200. 201. 202. 203. 204. 205. 206. 207. 208. 209. 210. 211. 212. 213. 214. 215. 216. 217. 218. 219. 220. 221. 222. 223. 224. 225. 226. 227. 228. 229. 230. 231. 232. 233. 234. 235. 236. 237. 238. 239. 240. 241. 242. 243. 244. 245. 246. 247. 248. 249. 250. 251. 252. 253. 254. 255. 256. 257. 258. 259. 260. 261. 262. 263. 264. 265. 266. 267. 268. 269. 270. 271. 272. 273. 274. 275. 276. 277. 278. 279. 280. 281. 282. 283. 284. 285. 286. 287. 288. 289. 290. 291. 292. 293. 294. 295. 296. 297. 298. 299. 300. 301. 302. 303. 304. 305. 306. 307. 308. 309. 310. 311. 312. 313. 314. 315. 316. 317. 318. 319. 320. 321. 322. 323. 324. 325. 326. 327. 328. 329. 330. 331. 332. 333. 334. 335. 336. 337. 338. 339. 340. 341. 342. 343. 344. 345. 346. 347. 348. 349. 350. 351. 352. 353. 354. 355. 356. 357. 358. 359. 360. 361. 362. 363. 364. 365. 366. 367. 368. 369. 370. 371. 372. 373. 374. 375. 376. 377. 378. 379. 380. 381. 382. 383. 384. 385. 386. 387. 388. 389. 390. 391. 392. 393. 394. 395. 396. 397. 398. 399. 400. 401. 402. 403. 404. 405. 406. 407. 408. 409. 410. 411. 412. 413. 414. 415. 416. 417. 418. 419. 420. 421. 422. 423. 424. 425. 426. 427. 428. 429. 430. 431. 432. 433. 434. 435. 436. 437. 438. 439. 440. 441. 442. 443. 444. 445. 446. 447. 448. 449. 450. 451. 452. 453. 454. 455. 456. 457. 458. 459. 460. 461. 462. 463. 464. 465. 466. 467. 468. 469. 470. 471. 472. 473. 474. 475. 476. 477. 478. 479. 480. 481. 482. 483. 484. 485. 486. 487. 488. 489. 490. 491. 492. 493. 494. 495. 496. 497. 498. 499. 500. 501. 502. 503. 504. 505. 506. 507. 508. 509. 510. 511. 512. 513. 514. 515. 516. 517. 518. 519. 520. 521. 522. 523. 524. 525. 526. 527. 528. 529. 530. 531. 532. 533. 534. 535. 536. 537. 538. 539. 540. 541. 542. 543. 544. 545. 546. 547. 548. 549. 550. 551. 552. 553. 554. 555. 556. 557. 558. 559. 560. 561. 562. 563. 564. 565. 566. 567. 568. 569. 570. 571. 572. 573. 574. 575. 576. 577. 578. 579. 580. 581. 582. 583. 584. 585. 586. 587. 588. 589. 590. 591. 592. 593. 594. 595. 596. 597. 598. 599. 600. 601. 602. 603. 604. 605. 606. 607. 608. 609. 610. 611. 612. 613. 614. 615. 616. 617. 618. 619. 620. 621. 622. 623. 624. 625. 626. 627. 628. 629. 630. 631. 632. 633. 634. 635. 636. 637. 638. 639. 640. 641. 642. 643. 644. 645. 646. 647. 648. 649. 650. 651. 652. 653. 654. 655. 656. 657. 658. 659. 660. 661. 662. 663. 664. 665. 666. 667. 668. 669. 670. 671. 672. 673. 674. 675. 676. 677. 678. 679. 680. 681. 682. 683. 684. 685. 686. 687. 688. 689. 690. 691. 692. 693. 694. 695. 696. 697. 698. 699. 700. 701. 702. 703. 704. 705. 706. 707. 708. 709. 710. 711. 712. 713. 714. 715. 716. 717. 718. 719. 720. 721. 722. 723. 724. 725. 726. 727. 728. 729. 730. 731. 732. 733. 734. 735. 736. 737. 738. 739. 740. 741. 742. 743. 744. 745. 746. 747. 748. 749. 750. 751. 752. 753. 754. 755. 756. 757. 758. 759. 760. 761. 762. 763. 764. 765. 766. 767. 768. 769. 770. 771. 772. 773. 774. 775. 776. 777. 778. 779. 780. 781. 782. 783. 784. 785. 786. 787. 788. 789. 790. 791. 792. 793. 794. 795. 796. 797. 798. 799. 800. 801. 802. 803. 804. 805. 806. 807. 808. 809. 810. 811. 812. 813. 814. 815. 816. 817. 818. 819. 820. 821. 822. 823. 824. 825. 826. 827. 828. 829. 830. 831. 832. 833. 834. 835. 836. 837. 838. 839. 840. 84

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[illegible]

C11107

S

783452

M/Z = 319.9

First peak is baseline drop corrected area.
 Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 11.540 | 212 | 215 | 221 | 9 | 20428 | 4525 | 100.00 |
| 2 | 11.540 | 212 | 215 | 221 | 9 | 20428 | 4426 | 97.81 |

320/322 CALCULATION

R-Global(1) = .8323

M/Z = 257.0

First peak is baseline drop corrected area.
 Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 11.540 | 212 | 215 | 221 | 9 | 23016 | 3918 | 100.00 |
| 2 | 11.540 | 212 | 215 | 221 | 9 | 23016 | 3648 | 93.11 |

(320 + 322)/(332 + 334) CALCULATION

R-Global(3) = .8310

(328)/(332 + 334) CALCULATION

R-Global(3) = .5010

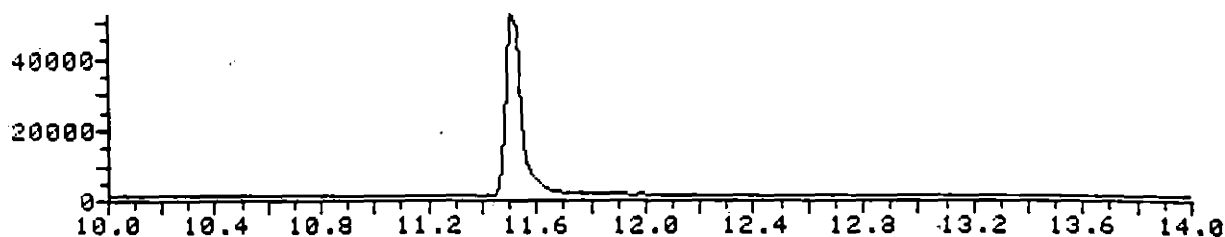
CONFIDENTIAL 20150
 SUBJECT TO PROTECTIVE ORDER

C11108

File >B3453 333.6-334.6 amu.2UL UT-3275

SP2330 200-1/3/250

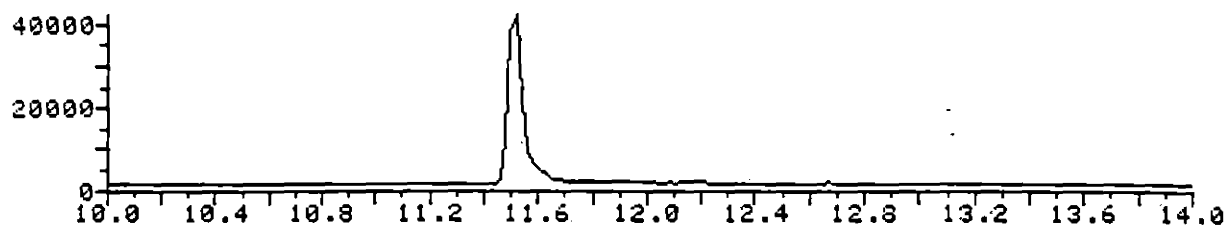
BTL



File >B3453 331.6-332.6 amu.2UL UT-3275

SP2330 200-1/3/250

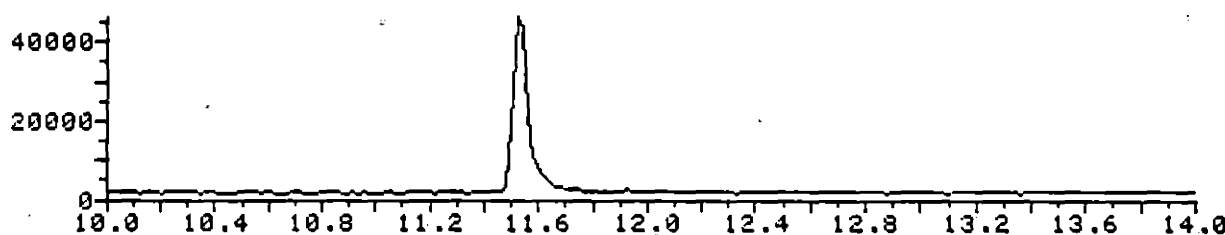
BTL



File >B3453 327.6-328.6 amu.2UL UT-3275

SP2330 200-1/3/250

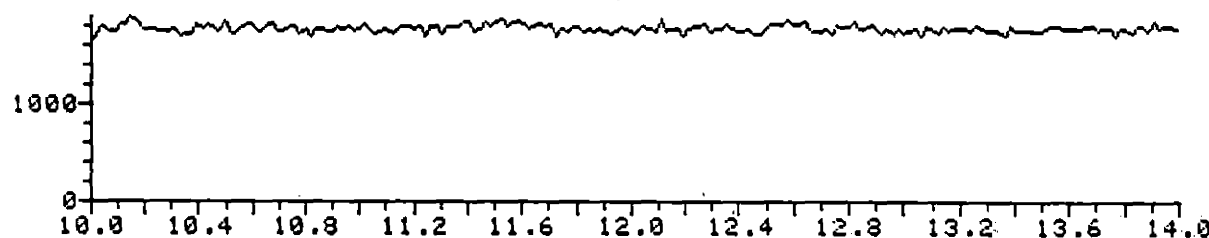
BTL



File >B3453 321.6-322.6 amu.2UL UT-3275

SP2330 200-1/3/250

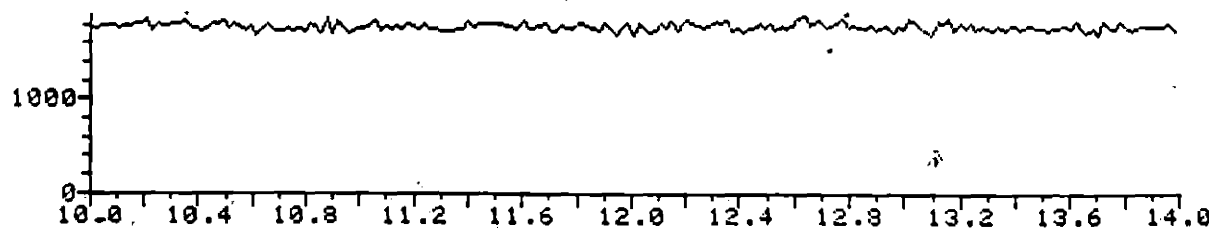
BTL



File >B3453 319.6-320.6 amu.2UL UT-3275

SP2330 200-1/3/250

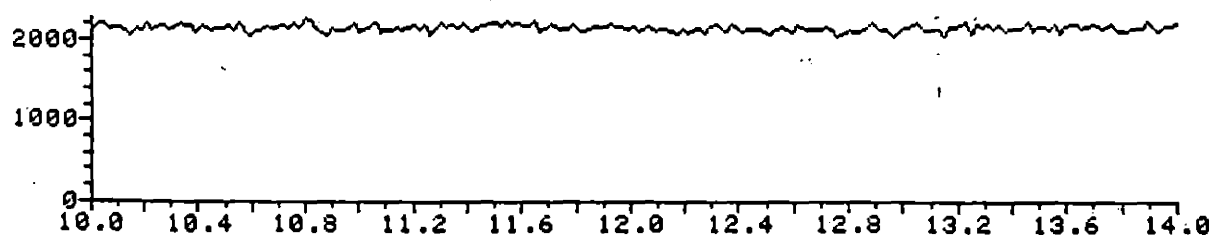
BTL



File >B3453 256.7-257.7 amu.2UL UT-3275

SP2330 200-1/3/250

BTL



30051

C11109

MS data file header from : >B3453

Sample: 2UL UT-3275 Operator: GARY MS 12/19/84 13:42
Misc : SP2330 200-1/3/250 BTL#25BTL#25
Sys. #: 1 MS model: 87 SW/HW rev.: CA ALS #: 0
Method file: SP2330 Tuning file: APEI01 No. of extra records: 2
Source temp.: 250 Analyzer temp.: 0 Transfer line temp.: 0

Chromatographic temperatures : 0. 0. 0. 0. 0.
Chromatographic times, min. : 0.0 0.0 0.0 0.0 0.0
Chromatographic rate, deg/min: 0.0 0.0 0.0 0.0 0.0

M/Z = 333.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 11.502 | 213 | 216 | 228 | 15 | 194788 | 167083 | 100.00 |
| 2 | 11.502 | 213 | 216 | 228 | 15 | 194788 | 163130 | 97.63 |

M/Z = 331.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 11.523 | 213 | 217 | 230 | 17 | 165359 | 135626 | 100.00 |
| 2 | 11.523 | 213 | 217 | 230 | 17 | 165359 | 132438 | 97.65 |

332/334 CALCULATION

R-Global(0) = .8117

M/Z = 327.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 11.523 | 213 | 217 | 230 | 17 | 185976 | 152741 | 100.00 |
| 2 | 11.523 | 213 | 217 | 230 | 17 | 185976 | 149001 | 97.55 |

M/Z = 321.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 0.000 | 231 | 0 | 230 | -1 | 0 | 1802 | 100.00 |
| 2 | 0.000 | 231 | 0 | 230 | -1 | 0 | 1802 | 100.00 |

CONFIDENTIAL 00052

SUBJECT TO PROTECTIVE ORDER

C111110

S

783453

M/Z = 319.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

| Peak
| R.T.
min. | first
scan | max
scan | last
scan | peak
width | raw
area | corr.
area | corr.
% |
|-----------|--------------|---------------|-------------|--------------|---------------|-------------|---------------|------------|
| 1 | 0.000 | 231 | 0 | 230 | -1 | 0 | 1794 | 100.00 |
| 2 | 0.000 | 231 | 0 | 230 | -1 | 0 | 1794 | 100.00 |

320/322 CALCULATION

R-Global(1) = .9956

M/Z = 257.0

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

| Peak
| R.T.
min. | first
scan | max
scan | last
scan | peak
width | raw
area | corr.
area | corr.
% |
|-----------|--------------|---------------|-------------|--------------|---------------|-------------|---------------|------------|
| 1 | 0.000 | 231 | 0 | 230 | -1 | 0 | 2175 | 100.00 |
| 2 | 0.000 | 231 | 0 | 230 | -1 | 0 | 2175 | 100.00 |

(320 + 322)/(332 + 334) CALCULATION

R-Global(3) = .0119

(328)/(332 + 334) CALCULATION

R-Global(3) = .5045

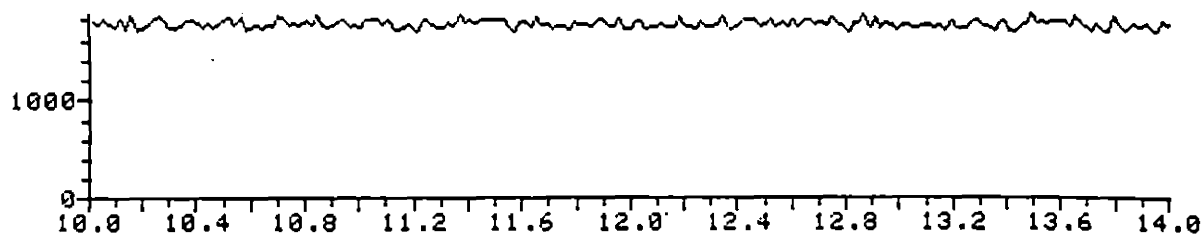
00053

C11111

File >B3454 333.6-334.6 amu.2UL UT-3276

SP2330 200-1/3/250

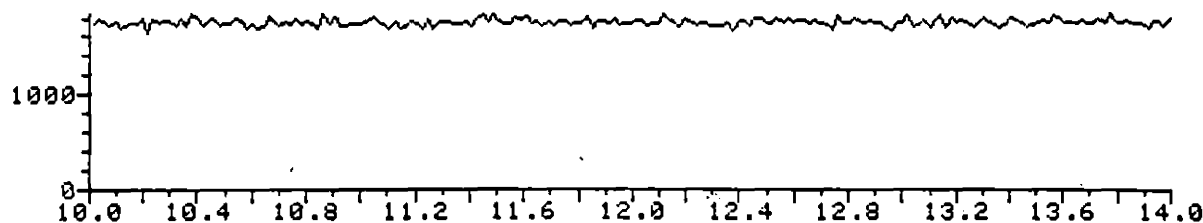
BTL



File >B3454 331.6-332.6 amu.2UL UT-3276

SP2330 200-1/3/250

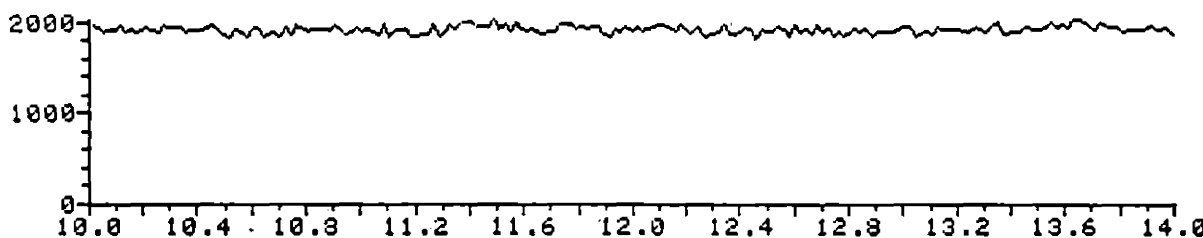
BTL



File >B3454 327.6-328.6 amu.2UL UT-3276

SP2330 200-1/3/250

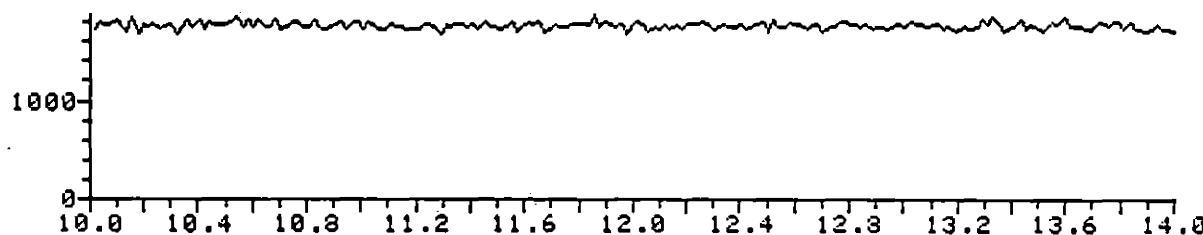
BTL



File >B3454 321.6-322.6 amu.2UL UT-3276

SP2330 200-1/3/250

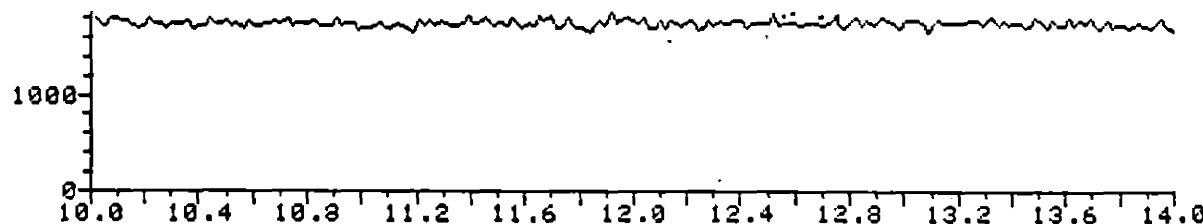
BTL



File >B3454 319.6-320.6 amu.2UL UT-3276

SP2330 200-1/3/250

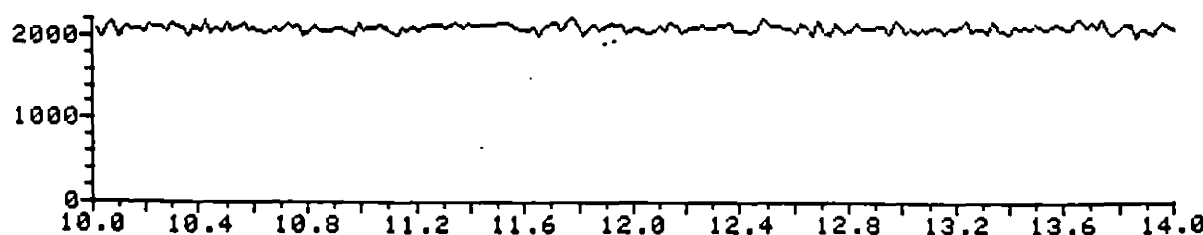
BTL



File >B3454 256.7-257.7 amu.2UL UT-3276

SP2330 200-1/3/250

BTL



CONFIDENTIAL

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C11112

MS data file header from : >B3454

Sample: 2UL UT-3276 Operator: GARY MS 12/19/84 14:02
Misc : SP2330 200-1/3/250 BTL#27BTL#27
Sys. #: 1 MS model: 87 SW/HW rev.: CA ALS #: 0
Method file: SP2330 Tuning file: APEI01 No. of extra records: 2
Source temp.: 250 Analyzer temp.: 0 Transfer line temp.: 0

Chromatographic temperatures : 0. 0. 0. 0. 0.
Chromatographic times, min. : 0.0 0.0 0.0 0.0 0.0
Chromatographic rate, deg/min: 0.0 0.0 0.0 0.0 0.0

M/Z = 333.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 0.000 | 231 | 0 | 230 | -1 | 0 | 1751 | 100.00 |
| 2 | 0.000 | 231 | 0 | 230 | -1 | 0 | 1751 | 100.00 |

M/Z = 331.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 0.000 | 231 | 0 | 230 | -1 | 0 | 1736 | 100.00 |
| 2 | 0.000 | 231 | 0 | 230 | -1 | 0 | 1736 | 100.00 |

332/334 CALCULATION

R-Global(0) = .9914

M/Z = 327.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 0.000 | 231 | 0 | 230 | -1 | 0 | 1940 | 100.00 |
| 2 | 0.000 | 231 | 0 | 230 | -1 | 0 | 1940 | 100.00 |

M/Z = 321.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 0.000 | 231 | 0 | 230 | -1 | 0 | 1781 | 100.00 |
| 2 | 0.000 | 231 | 0 | 230 | -1 | 0 | 1781 | 100.00 |

CC

20075

SUBMIT

C11113

703454

S

M/Z = 319.9

First peak is baseline drop corrected area.

Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 0.000 | 231 | 0 | 230 | -1 | 0 | 1717 | 100.00 |
| 2 | 0.000 | 231 | 0 | 230 | -1 | 0 | 1717 | 100.00 |

320/322 CALCULATION

R-Global(1) = .9641

M/Z = 257.0

First peak is baseline drop corrected area.

Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 0.000 | 231 | 0 | 230 | -1 | 0 | 2055 | 100.00 |
| 2 | 0.000 | 231 | 0 | 230 | -1 | 0 | 2055 | 100.00 |

<320 + 322>/<332 + 334> CALCULATION

R-Global(3) = 1.0029

<328'>/<332 + 334> CALCULATION

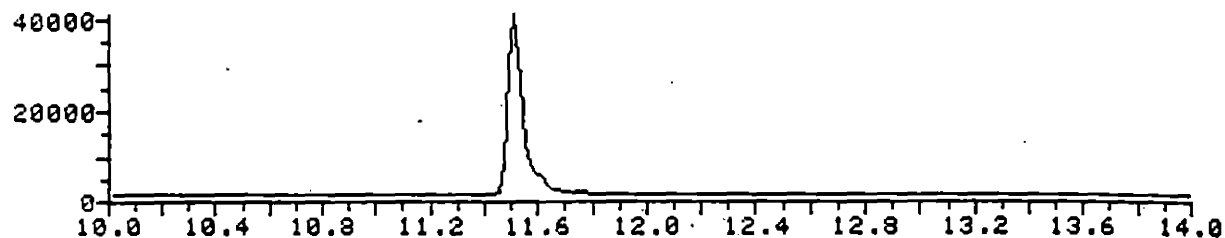
R-Global(3) = .5516

CONFIDENTIAL 30056

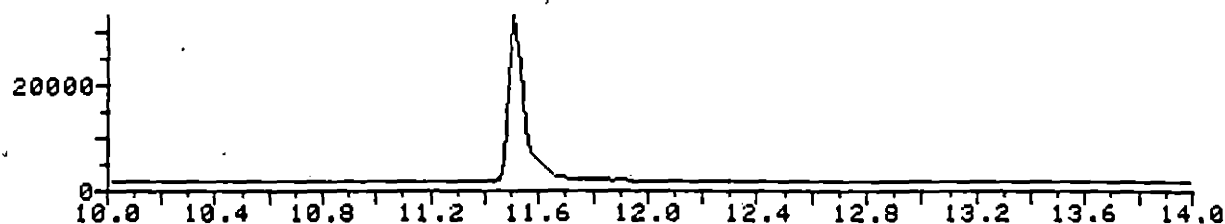
SUBJECT TO PROTECTIVE ORDER

C11114

File >B3455 333.6-334.6 amu.2UL .2503/.48/.20 SP2330 200-1/3/250 BTL..



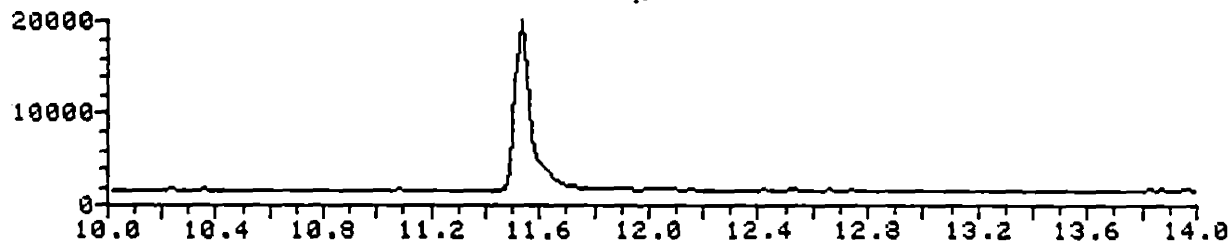
File >B3455 331.6-332.6 amu.2UL .2503/.48/.20 SP2330 200-1/3/250 BTL..



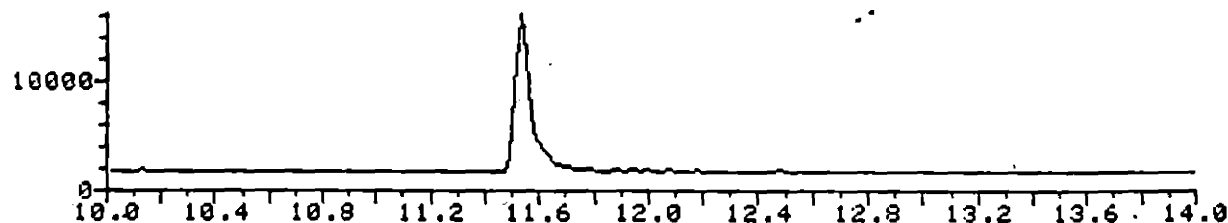
File >B3455 327.6-328.6 amu.2UL .2503/.48/.20 SP2330 200-1/3/250 BTL..



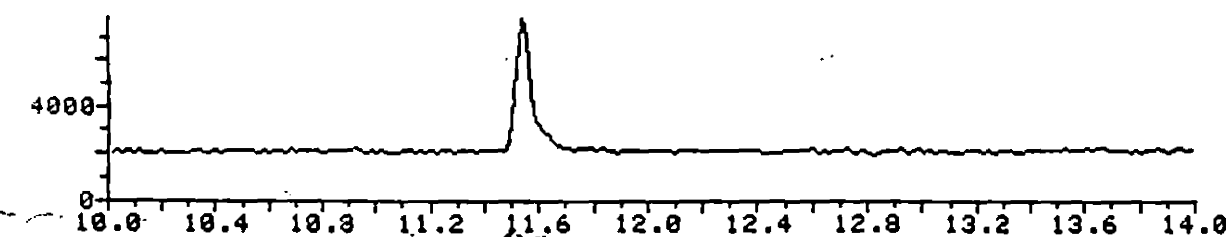
File >B3455 321.6-322.6 amu.2UL .2503/.48/.20 SP2330 200-1/3/250 BTL..



File >B3455 319.6-320.6 amu.2UL .2503/.48/.20 SP2330 200-1/3/250 BTL..



File >B3455 256.7-257.7 amu.2UL .2503/.48/.20 SP2330 200-1/3/250 BTL



C11115

MS data file header from : >B3455

Sample: 2UL .2503/.48/.20 Operator: GARY MS 12/19/84 14:22
Misc : SP2330 200-1/3/250 BTL#29BTL#29
Sys. #: 1 MS model: 87 SW/HW rev.: CR ALS # : 0
Method file: SP2330 Tuning file: APEI01 No. of extra records: 2
Source temp.: 250 Analyzer temp.: 0 Transfer line temp.: 0

Chromatographic temperatures : 0. 0. 0. 0. 0.
Chromatographic times, min. : 0.0 0.0 0.0 0.0 0.0
Chromatographic rate, deg/min: 0.0 0.0 0.0 0.0 0.0

M/Z = 333.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 11.511 | 211 | 215 | 225 | 14 | 156520 | 131978 | 100.00 |
| 2 | 11.511 | 211 | 215 | 225 | 14 | 156520 | 127701 | 96.76 |

M/Z = 331.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 11.511 | 210 | 215 | 226 | 16 | 135374 | 107950 | 100.00 |
| 2 | 11.511 | 210 | 215 | 226 | 16 | 135374 | 103582 | 95.95 |

332/334 CALCULATION

R-Global(0) = .8179

M/Z = 327.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 11.531 | 212 | 216 | 218 | 6 | 113807 | 102269 | 100.00 |
| 2 | 11.531 | 212 | 216 | 218 | 6 | 113807 | 70481 | 68.92 |

M/Z = 321.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 11.531 | 212 | 216 | 225 | 13 | 86205 | 63169 | 100.00 |
| 2 | 11.531 | 212 | 216 | 225 | 13 | 86205 | 60731 | 96.14 |

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C11116

703455

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

| Peak
| R.T.
min. | first
scan | max
scan | last
scan | peak
width | raw
area | corr.
area | corr.
% |
|-----------|--------------|---------------|-------------|--------------|---------------|-------------|---------------|------------|
| 1 | 11.531 | 213 | 216 | 226 | 13 | 72984 | 49662 | 100.00 |
| 2 | 11.531 | 213 | 216 | 226 | 13 | 72984 | 48089 | 96.83 |

```
R-Global( 1) = .7862
```

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

| Peak
| R.T.
min. | first
scan | max
scan | last
scan | peak
width | raw
area | corr.
area | corr.
% |
|-----------|--------------|---------------|-------------|--------------|---------------|-------------|---------------|------------|
| 1 | 11.531 | 213 | 216 | 226 | 13 | 49277 | 22328 | 100.00 |
| 2 | 11.531 | 213 | 216 | 226 | 13 | 49277 | 21704 | 97.21 |

R-Global(3) = .4703

```
R-Global( 3) = .4239
```

00059

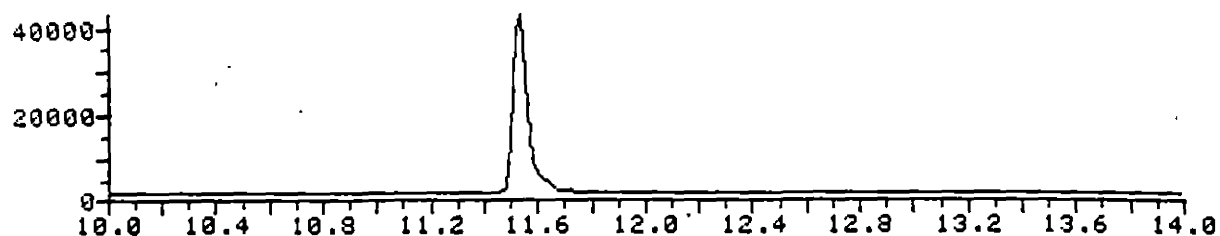
C11117

1. LIST OF PROTECTIVE ORDER /

File >B3456 333.6-334.6 amu.2UL UT-3277

SP2330 200-1/3/250

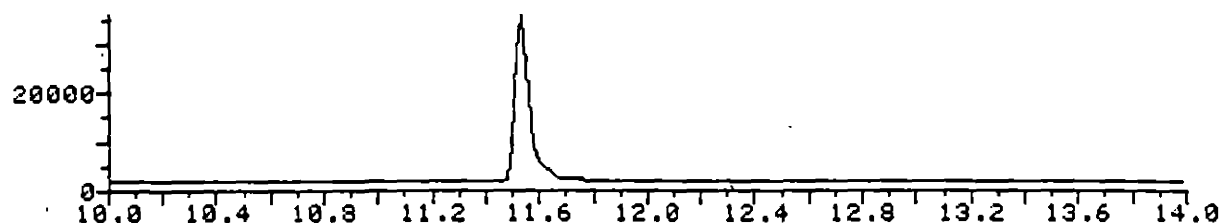
BTL



File >B3456 331.6-332.6 amu.2UL UT-3277

SP2330 200-1/3/250

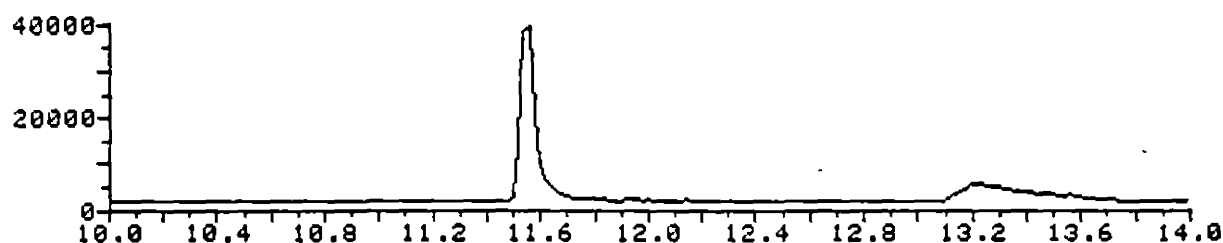
BTL



File >B3456 327.6-328.6 amu.2UL UT-3277

SP2330 200-1/3/250

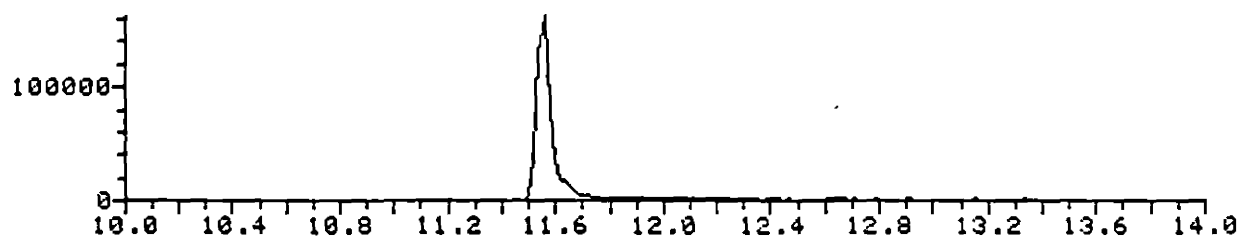
BTL



File >B3456 321.6-322.6 amu.2UL UT-3277

SP2330 200-1/3/250

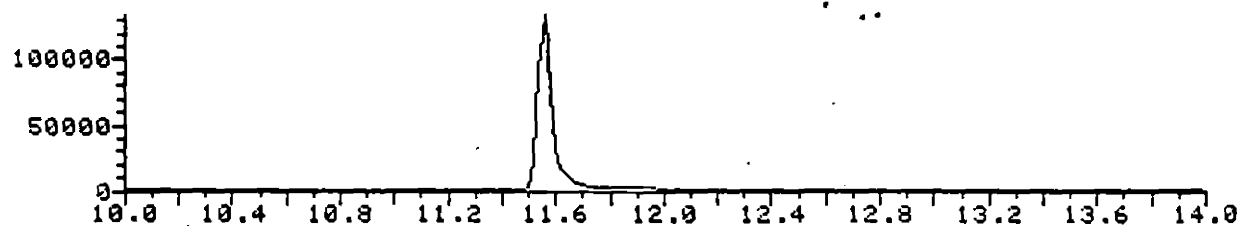
BTL



File >B3456 319.6-320.6 amu.2UL UT-3277

SP2330 200-1/3/250

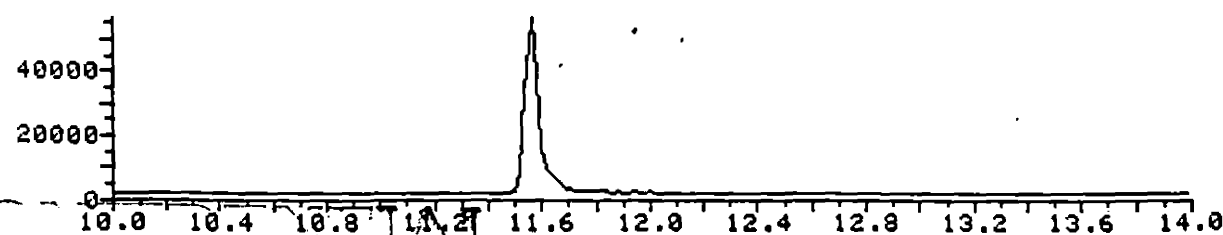
BTL



File >B3456 256.7-257.7 amu.2UL UT-3277

SP2330 200-1/3/250

BTL



SUBJECT TO PROTECTIVE ORDER 00060

C11118

MS data file header from : >B3456

Sample: 2UL NT-3277 Operator: GARY MS 12/19/84 14:42...
Misc : SP2330 200-1/3/250 BTL#31BTL#31
Sys. #: 1 MS model: 87 SW/HW rev.: CA ALS # : 0
Method file: SP2330 Tuning file: APEI01 No. of extra records: 2
Source temp.: 250 Analyzer temp.: 0 Transfer line temp.: 0 ...

Chromatographic temperatures : 0. 0. 0. 0. 0.
Chromatographic times, min. : 0.0 0.0 0.0 0.0 0.0
Chromatographic rate, deg/min: 0.0 0.0 0.0 0.0 0.0

M/Z = 333.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 11.537 | 212 | 216 | 226 | 14 | 159982 | 134320 | 100.00 |
| 2 | 11.537 | 212 | 216 | 226 | 14 | 159982 | 132101 | 98.35 |

M/Z = 331.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 11.537 | 212 | 216 | 225 | 13 | 130399 | 106076 | 100.00 |
| 2 | 11.537 | 212 | 216 | 225 | 13 | 130399 | 103918 | 97.97 |

332/334 CALCULATION

R-Global(0) = .7897

M/Z = 327.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 11.558 | 213 | 217 | 226 | 13 | 152228 | 126527 | 100.00 |
| 2 | 11.558 | 213 | 217 | 226 | 13 | 152228 | 123868 | 97.90 |

M/Z = 321.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 11.558 | 213 | 217 | 227 | 14 | 519977 | 493503 | 100.00 |
| 2 | 11.558 | 213 | 217 | 227 | 14 | 519977 | 483157 | 97.90 |

ORIGINAL 00051
SUBJECT TO PROTECTIVE ORDER

C111119

S

78 3456

M/Z = 319.9

First peak is baseline drop corrected area.
 Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 11.558 | 213 | 217 | 226 | 13 | 419649 | 396327 | 100.00 |
| 2 | 11.558 | 213 | 217 | 226 | 13 | 419649 | 385732 | 97.33 |

320/322 CALCULATION

R-Global(1) = .8031

M/Z = 257.0

First peak is baseline drop corrected area.
 Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 11.558 | 213 | 217 | 226 | 13 | 193015 | 165039 | 100.00 |
| 2 | 11.558 | 213 | 217 | 226 | 13 | 193015 | 160671 | 97.35 |

(320 + 322)/(332 + 334) CALCULATION

R-Global(3) = 3.7015

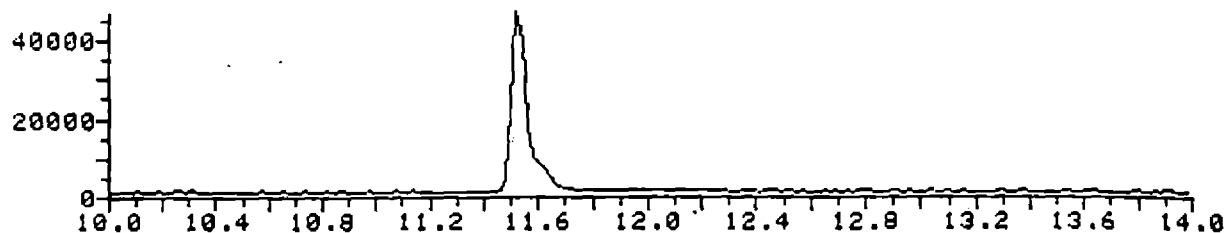
(328')/(332 + 334) CALCULATION

R-Global(3) = .5079

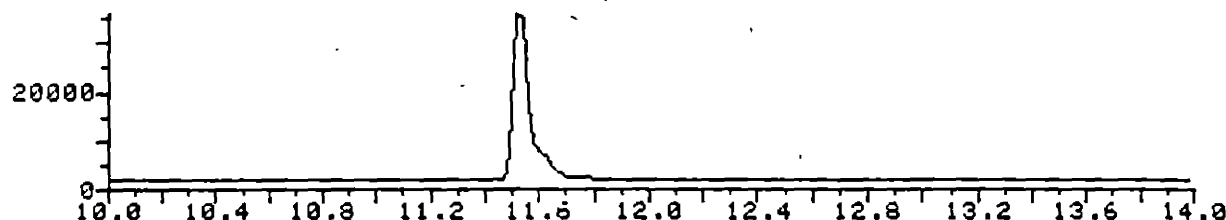
ALL INFORMATION CONTAINED
 HEREIN IS UNCLASSIFIED
 DATE 11-11-2011 BY 60322
 SUBJECT TO PROTECTIVE ORDER.

C11120

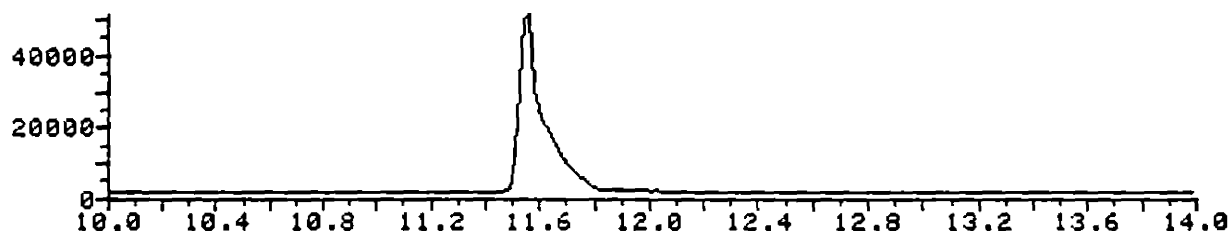
File >B3457 333.6-334.6 amu.2UL 2.503/.48/.20 SP2330 200-1/3/250 BTL...



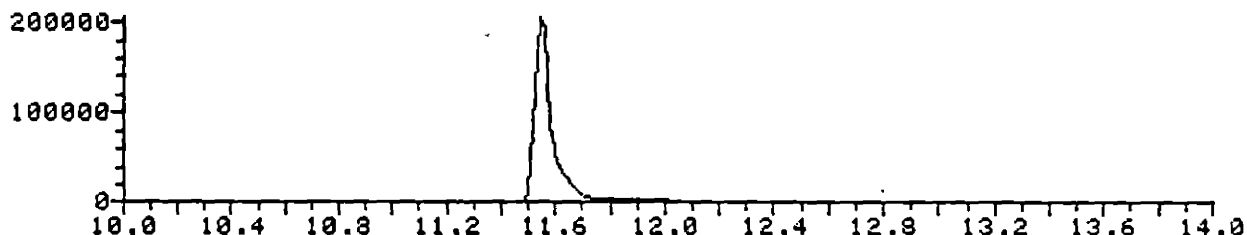
File >B3457 331.6-332.6 amu.2UL 2.503/.48/.20 SP2330 200-1/3/250 BTL...



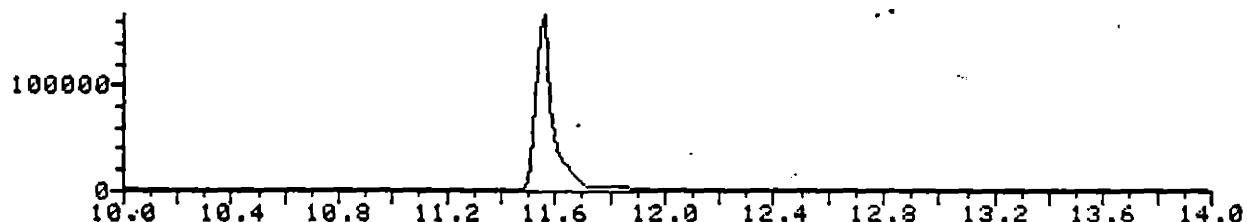
File >B3457 327.6-328.6 amu.2UL 2.503/.48/.20 SP2330 200-1/3/250 BTL...



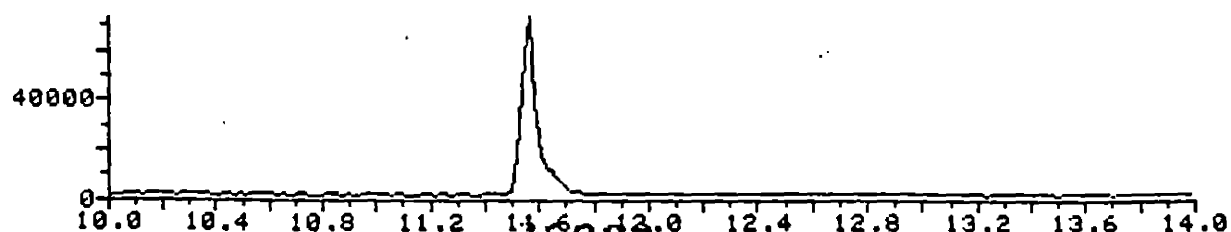
File >B3457 321.6-322.6 amu.2UL 2.503/.48/.20 SP2330 200-1/3/250 BTL...



File >B3457 319.6-320.6 amu.2UL 2.503/.48/.20 SP2330 200-1/3/250 BTL...



File >B3457 256.7-257.7 amu.2UL 2.503/.48/.20 SP2330 200-1/3/250 BTL...



CONFIDENTIAL
SUBJECT TO PROTECTIVE ORDER

C11121

MS data file header from : >B3457

Sample: 2UL 2.503/.48/.20 Operator: GARY MS 12/19/84 15:05--
Misc : SP2330 200-1/3/250 BTL#41BTL#41
Sys. #: 1 MS model: 87 SW/HW rev.: CA ALS #: 0
Method file: SP2330 Tuning file: APEI01 No. of extra records: 2
Source temp.: 250 Analyzer temp.: 0 Transfer line temp.: 0

Chromatographic temperatures : 0. 0. 0. 0. 0.
Chromatographic times, min. : 0.0 0.0 0.0 0.0 0.0
Chromatographic rate, deg/min: 0.0 0.0 0.0 0.0 0.0

M/Z = 333.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 11.521 | 207 | 210 | 220 | 13 | 178007 | 152722 | 100.00 |
| 2 | 11.521 | 207 | 210 | 220 | 13 | 178007 | 149576 | 97.94 |

M/Z = 331.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 11.521 | 207 | 210 | 221 | 14 | 151288 | 126382 | 100.00 |
| 2 | 11.521 | 207 | 210 | 221 | 14 | 151288 | 123554 | 97.76 |

332/334 CALCULATION

R-Global(0) = .8275

M/Z = 327.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 11.562 | 208 | 212 | 218 | 10 | 247964 | 226824 | 100.00 |
| 2 | 11.562 | 208 | 212 | 218 | 10 | 247964 | 179939 | 79.33 |

M/Z = 321.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 11.542 | 208 | 211 | 222 | 14 | 745665 | 714193 | 100.00 |
| 2 | 11.542 | 208 | 211 | 222 | 14 | 745665 | 700333 | 98.06 |

CONFIDENTIAL
SUBJECT TO PROTECTIVE ORDER

10041

C11122

S

783457

M/Z = 319.9

First peak is baseline drop corrected area.
 Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 11.562 | 208 | 212 | 222 | 14 | 605571 | 578425 | 100.00 |
| 2 | 11.562 | 208 | 212 | 222 | 14 | 605571 | 565776 | 97.81 |

320/322 CALCULATION

R-Global(1) = .8099

M/Z = 257.0

First peak is baseline drop corrected area.
 Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 11.562 | 208 | 212 | 222 | 14 | 268977 | 239297 | 100.00 |
| 2 | 11.562 | 208 | 212 | 222 | 14 | 268977 | 234334 | 97.93 |

(320 + 322)/(332 + 334) CALCULATION

R-Global(3) = 4.6313

(328)/(332 + 334) CALCULATION

R-Global(3) = .7897

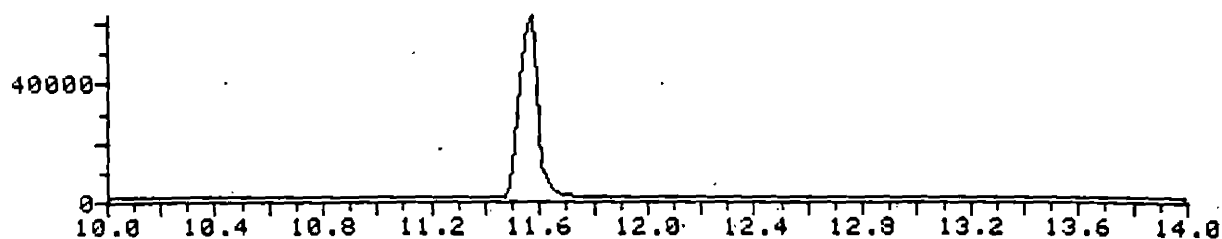
CONFIDENTIAL

SUBJECT TO PROTECTIVE ORDER

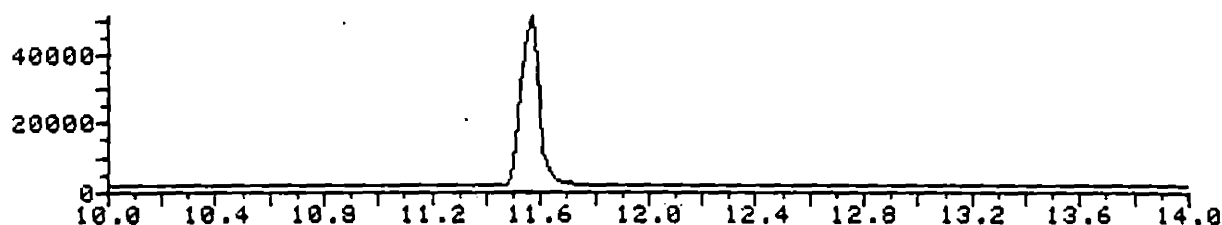
0.065

C11123

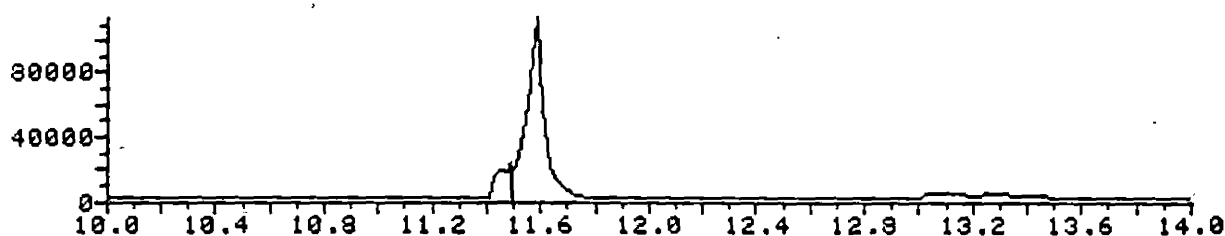
File >B3458 333.6-334.6 amu.2UL 25.03/.48/.20 SP2330 200-1/3/250 BTL



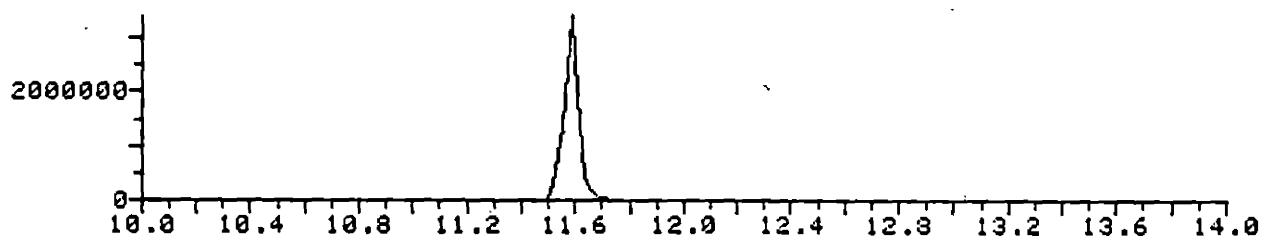
File >B3458 331.6-332.6 amu.2UL 25.03/.48/.20 SP2330 200-1/3/250 BTL



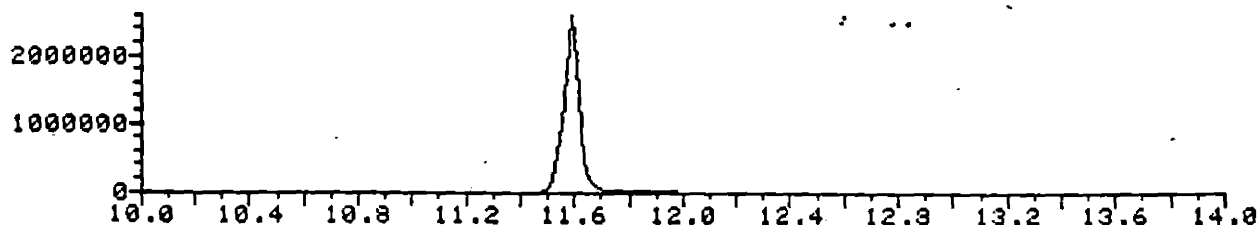
File >B3458 327.6-328.6 amu.2UL 25.03/.48/.20 SP2330 200-1/3/250 BTL



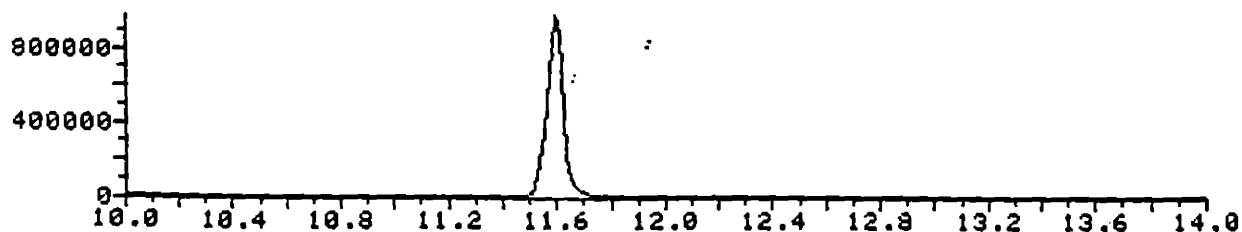
File >B3458 321.6-322.6 amu.2UL 25.03/.48/.20 SP2330 200-1/3/250 BTL



File >B3458 319.6-320.6 amu.2UL 25.03/.48/.20 SP2330 200-1/3/250 BTL



File >B3458 256.7-257.7 amu.2UL 25.03/.48/.20 SP2330 200-1/3/250 BTL



CONFIDENTIAL

046

C11124

SUBJECT TO PROTECTIVE ORDER

MS data file header from : >B3458

Sample: 2UL 25.03/.48/.20 Operator: GARY MS 12/19/84-15r25-
Misc : SP2330 200-1/3/250 BTL#43BTL#43
Sys. #: 1 MS model: 87 SW/HW rev.: CA ALS #: 0
Method file: SP2330 Tuning file: APEI01 No. of extra records: 2
Source temp.: 250 Analyzer temp.: 0 Transfer line temp.: 0

Chromatographic temperatures : 0. 0. 0. 0. 0.
Chromatographic times, min. : 0.0 0.0 0.0 0.0 0.0
Chromatographic rate, deg/min: 0.0 0.0 0.0 0.0 0.0

M/Z = 333.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 11.569 | 212 | 218 | 226 | 14 | 265886 | 240630 | 100.00 |
| 2 | 11.569 | 212 | 218 | 226 | 14 | 265886 | 236892 | 98.45 |

M/Z = 331.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 11.569 | 212 | 218 | 226 | 14 | 221396 | 193858 | 100.00 |
| 2 | 11.569 | 212 | 218 | 226 | 14 | 221396 | 192199 | 99.14 |

332/334 CALCULATION

R-Global(0) = .8056

M/Z = 327.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 11.590 | 214 | 219 | 227 | 13 | 468201 | 422324 | 100.00 |
| 2 | 11.590 | 214 | 219 | 227 | 13 | 468201 | 324206 | 76.77 |

M/Z = 321.9

First peak is baseline drop corrected area.
Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 11.590 | 213 | 219 | 226 | 13 | 10803684 | 10758432 | 100.00 |
| 2 | 11.590 | 213 | 219 | 226 | 13 | 10803684 | 10548668 | 98.05 |

CONFIDENTIAL
SUBJECT TO PROTECTIVE ORDER

C11125

783458

S

M/Z = 319.9

First peak is baseline drop corrected area.

Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 11.590 | 213 | 219 | 228 | 15 | 8654242 | 8615902 | 100.00 |
| 2 | 11.590 | 213 | 219 | 228 | 15 | 8654242 | 8487532 | 98.51 |

320/322 CALCULATION

R-Global(1) = .8009

M/Z = 257.0

First peak is baseline drop corrected area.

Second peak is tangent skimmed corrected area.

| Peak # | R.T. min. | first scan | max scan | last scan | peak width | raw area | corr. area | corr. % |
|--------|-----------|------------|----------|-----------|------------|----------|------------|---------|
| 1 | 11.590 | 213 | 219 | 228 | 15 | 3489204 | 3455499 | 100.00 |
| 2 | 11.590 | 213 | 219 | 228 | 15 | 3489204 | 3488336 | 98.48 |

(320 + 322)/(332 + 334) CALCULATION

R-Global(3) = 44.5912

(328)/(332 + 334) CALCULATION

R-Global(3) = .7492

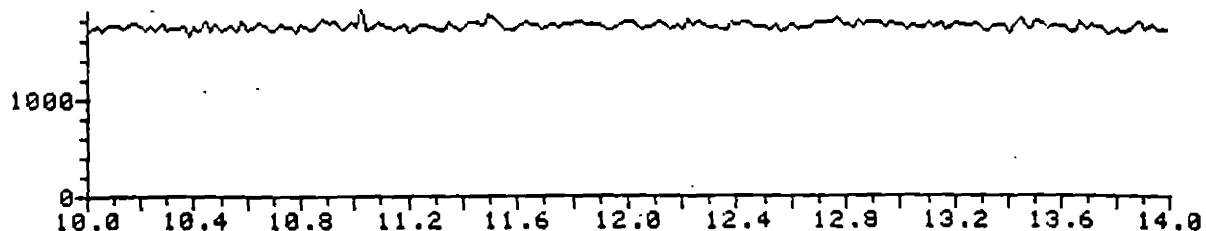
J0008

SUBJECT TO

C11126

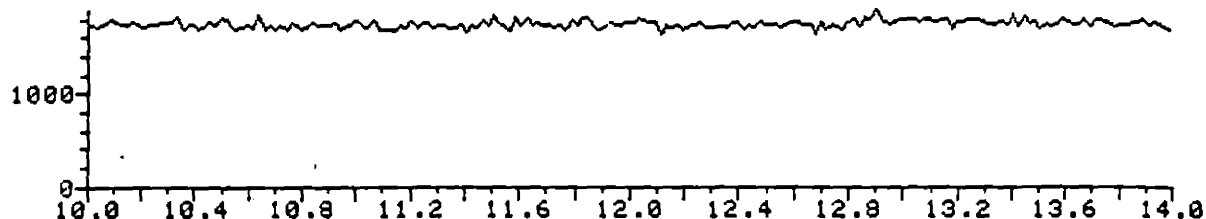
File >B3459 333.6-334.6 amu.DODECANE

SP2330 200-1/3/250 RUN.....BT...



File >B3459 331.6-332.6 amu.DODECANE

SP2330 200-1/3/250 RUN.....BT...



File >B3459 327.6-328.6 amu.DODECANE

SP2330 200-1/3/250 RUN.....BT...



File >B3459 321.6-322.6 amu.DODECANE

SP2330 200-1/3/250 RUN.....BT...



File >B3459 319.6-320.6 amu.DODECANE

SP2330 200-1/3/250 RUN.....BT...



File >B3459 256.7-257.7 amu.DODECANE

SP2330 200-1/3/250 RUN.....BT...



CONFIDENTIAL
SUBJECT TO PROTECTIVE ORDER

C11127

SUBJECT

Standards Prep

JOB NO.

309.10011

PREPARED BY (SIGNATURE)

Gary Thomas

DATE

12 OCT 84

Native TCDD stock: Using Cahn microbalance in HML.
 NBS weight. True Value = 1.0000 ⁵⁷mg OBSERVED = 1.0006 mg
 Weight of TCDD = 500.7 μ g. Diluted to 10 ml w/ o-xylene
 Final conc = 50.07 μ g/ml

³⁷Cl-TCDD stock: KOK ISOTOPES 100 μ g in 0.25 ml anisole
 LOT NO. J-II-70. Diluted to 25 ml in dodecane. Final conc =
 4 μ g/ml.

¹³C-TCDD stock solution (prepared 4/10/84) 12 μ g/ml

Intermediates stock solution: 2 ml of 12 μ g/ml ¹³C-TCDD and 2.5 ml
 of 4 μ g/ml ³⁷Cl-TCDD diluted to 25 ml w/ dodecane. Final conc =
 0.96 μ g/ml ¹³C/.40 ³⁷Cl (For 1:1 dilutions w/ ¹³C) ⁵⁷

Internal standard spiking solution: 2 ml of 12 μ g/ml ¹³C-TCDD and 2.5 ml
 of 4 μ g/ml ³⁷Cl-TCDD diluted to 100 ml w/ iso-octane. Final
 conc = 0.24 μ g/ml ¹³C/.10 ³⁷Cl

¹³C intermediate stock solutions: (all in dodecane)

1 ml of 50.07 μ g/ml diluted to 10 ml. Final conc = 5.007 μ g/ml

1 ml of 5.007 μ g/ml diluted to 10 ml. Final conc = 0.5007 μ g/ml

2 ml of 50.07 μ g/ml diluted to 5 ml. Final conc. = 20.03 μ g/ml

2 ml of 5.007 μ g/ml diluted to 5 ml. Final conc. = 2.003 μ g/ml

2 ml of 0.5007 μ g/ml diluted to 5 ml. Final conc. = 0.2003 μ g/ml

(cont. next page)

READ AND UNDERSTOOD BY

DATE

C11128