SYNTEX RESEARCH
DIVISION OF SYNTEX (U.S.A.) INC.
3401 HILLVIEW AVENUE, P.O. BOX 10850
PALO ALTO, CALIFORNIA 94303

Site: Syntex-Verona
ID #: MOD007245215 4
Break: 17.8
Other: Spring River Report 11-29-89

ANALYTICAL AND ENVIRONMENTAL RESEARCH

0751

40033365
SUPERFUND RECORDS

AER: 10554
November 27, 1989

RECEIVED
NOV 29 1989
SUPERFUND BRANCH

Mr. Robert Morby
Chief, Waste Management Branch
Air and Waste Management Division
USEPA
Region VII
726 Minnesota Avenue
Kansas City, Kansas 66101

Dear Mr. Morby:

Enclosed is the annual report of analysis of fish samples taken from the SpringRiver in 1989. This report is being provided in compliance with the Verona Plant Fish and Sediment Plan, as approved by the U.S. Environmental Protection agency on March 24, 1984 Docket No. 83-H-008, and with the terms of a one year extention of sampling and analysis. Also enclosed is a letter dated July 17, 1989 from Gary Pendergrass that describes the terms of the extention, and a letter dated July 21, 1989 from Glenn Curtis concurring in the description.

The origins of the fish samples are documented in a letter dated September 28, 1989, from Ms. Cynthia S. Morris of the Missouri Department of Conservation to you. A previous letter to you on November 11, 1986 from Dr. Throop stated our intention to analyze the fish samples at Syntex Research. The report by Dr. Kelvin Chan, et al., of the analysis of fish collected in August 1989 is attached (AER: wp0103). Table I of this report is the summary of analytical results. The required deliverables are included in Dr. Chan's report. As in previous years we have calculated the equivalent of a whole fish analysis for fish in Group B at each site using the laboratory analyses for fillets and remainders, and the weights of fillets and whole fish supplied by Ms. Morris. The calculated equivalent levels of TCDD in whole fish are summarized in the attached Table II.

A report on the statistical analysis of data for TCDD in fish is being prepared, also according to Paragraph 42 of the Order. I expect to be sending the results of the statistical analyses to you soon.

Please do not hesitate to telephone me if you have any questions concerning these results or if I can provide you with any additional information.

Yours sincerely,

David Robertson

David Robertson, Ph.D.
Department Head
Environmental Analysis and
Process Control

cc: Kent Stormer (w/attach)
Lewis Throop

TABLE II

Calculated 2,3,7,8-TCDD Levels in Whole Fish
Based on Analyses of Fillets and Remainders from Group B

Site	Reference Number	Weight of Fillets (1)	TCDD Fillets ppt (2)	Weight of Whole Fish (3)	TCDD in Remainder ppt (2)	Calc. TCDD in Whole Fish ppt
1	MDC-4	81g	3.3	598g	19.7 (4)	17.5
2	MDC-9	96g	4.1	641g	20.7	18.2
3	MDC-13	82g	1.5	512g	9.8	8.5
4	MDC-17	127g	1.3	799g	9.9	8.5

$$\text{Calculated TCDD in Whole Fish} = \frac{[\text{wt. of fillets X TCDD in fillets}] + [(\text{wt. of whole-fillets})\text{X TCDD in remainder}]}{\text{WT. OF WHOLE FISH}}$$

(1) Data provided by Ms. Morris, September 28, 1989
(2) Data provide by Dr. Chan, November 9, 1989.
(3) The total weight of whole fish was obtained by adding the individual weights provide by Ms. Morris.
(4) Average of two analyses

Calculations by David Robertson

2155y/lw

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION VII
726 MINNESOTA AVENUE
KANSAS CITY, KANSAS 66101

JUL 21 1989

Mr. Gary J. Pendergrass
Manager
Environmental Projects Department
Syntex Agribusiness, Inc.
Post Office Box 1246
Springfield, Missouri 65801

Dear Mr. Pendergrass:

Re: Spring River Fish Sampling

This letter is being written to recognize and concur with the actions described in your letter dated July 17, 1989, regarding the subject issue. These activities will include the collection and analysis of fish at sample locations No. 1 through 4 and preparation of a report and statistical analysis of the data. The described actions appear consistent as stated with our May 24, 1989, conference call.

In addition, as was discussed during and subsequent to the stated conference call, it is recommended that Syntex assess the uses of the Spring River in the vicinity and downstream of the sample stations. These efforts should be conducted to determine the frequency and type (i.e., gigging) of fishing conducted near and downstream of the Verona facility. This information will be important in determining the potential risks posed by the TCDD levels detected.

The Environmental Protection Agency (EPA) will look forward to receiving your report and analysis of the data.

Sincerely yours,

Glenn M. Curtis

Glenn M. Curtis
Remedial Section
Superfund Branch
Waste Management Division

Enclosure

cc: John Young, MDNR, (w/enclosure)
Ron Crunkilton, MDOC (w/enclosure)
Gail Carlson, MDOH (w/enclosure)

SYNTEX agribusiness inc NUTRITION AND CHEMICAL DIVISION ENVIRONMENTAL PROJECTS DEPARTMENT

July 17, 1989

RECEIVED
JUL 18 1989
REMD SECTION

Glenn Curtis
United States Environmental Protection Agency
Region VII
726 Minnesota Avenue
Kansas City, Kansas 66101

Re: Spring River Fish and Sediment Plan

Dear Glenn:

This letter follows up on our conference call on May 24, 1989 concerning the sampling and analysis of fish in the Spring River. Specifically, this letter outlines the agreement of Syntex Agribusiness, Inc. ("Syntex") to collect and analyze data for an additional year at selected sampling points on the Spring River.

As we discussed, Syntex agreed in a September 9, 1983 Consent Agreement and Order ("Consent Agreement") with the U.S. Environmental Protection Agency ("EPA") to prepare a plan for sampling, analyzing and reporting levels of 2,3,7,8-Tetrachlorodibenzo-p-dioxin ("2,3,7,8-TCDD") in the fish and sediment in the Spring River downstream from the Syntex Verona, Missouri, facility. Syntex subsequently prepared, and EPA approved, a Verona Fish and Sediment Plan, as submitted to EPA on October 12, 1983 and revised on January 31, 1984 and March 9, 1984 ("Plan"). The Consent Agreement and Plan require Syntex, among other things, to sample and analyze Spring River fish and sediment for a five year period, with provisions for yearly extensions of the program under certain specified conditions. The Consent Agreement and Order also require Syntex to prepare a final report ("Report"), including a statistical analysis of the collected data, at the end of the five year sampling program.

Syntex submitted the final report and statistical package to EPA on January 30, 1989. The data and conclusions in the report were the subject of our conference call.

Upon careful analysis of the data collected from the Spring River over the five year period, Syntex concluded in the Report that additional sampling and analysis would not be productive. As explained more fully in the Report, the levels of 2,3,7,8-TCDD detected were very low (actually <u>below</u> the detection limits anticipated by Paragraph III of the Plan); even using detection levels below those anticipated by the Plan, there were a significant number of samples with no detected 2,3,7,8-TCDD; and the detected levels of 2,3,7,8-TCDD in fish downstream from the Verona facility were well below those considered by EPA, CDC, and the FDA as presenting a concern to human health or the environment. These and other points discussed in the Syntex final Report emphasize the extremely low levels of 2,3,7,8-TCDD detected by the testing.

MAILING ADDRESS: P.O. BOX 1246, SPRINGFIELD, MO 65801 • (417) 866-4050 ALTERNATE NO. (417) 866-7291
SHIPPING ADDRESS: 2460 WEST BENNETT, SPRINGFIELD, MO 65807

Syntex remains convinced that additional sampling is not necessary. However, to accommodate EPA's wishes for additional data, Syntex will conduct additional sampling and analysis as outlined below.

Using the same sampling techniques that are set forth in the Consent Agreement and Plan, fish will be collected during August, 1989 from the Spring River at Location 1 (0.3 miles downstream from the Verona facility); Location 2 (3.0 miles downstream from the facility); Location 3 (6.0 miles downstream from the facility); and at Location 4 (9.0 miles downstream at road H near Hoberg). As we discussed during our conference call, sampling is not needed at Location 5 (12.0 miles downstream near intersection with road V). All of the data from Location 5 showed either extremely low levels of 2,3,7,8-TCDD or no detected 2,3,7,8-TCDD. EPA did not request additional testing of the sediment, and we concur that the data does not support such a request.

The collection of samples will take place during the period August 1 to August 31. Syntex will analyze the collected fish samples for 2,3,7,8-TCDD in accordance with the procedures described in Paragraph III of the Plan, and will subject the resulting data to the statistical analysis described in Paragraph V of the Plan. The appropriateness of additional sampling and analysis in future years will be governed by the provisions of paragraph 42 of the Consent Agreement.

Syntex will prepare a report to EPA concerning the sampling and analytical results developed during the sixth year of the study. The report will provide a statistical analysis of the data, and will include relevant graphs and statistical computations. The report will be provided within 30 days of receipt and acceptance by Syntex of the analysis of the collected fish samples.

We believe this plan conforms with the requests made by EPA personnel during the May 24 conference call. Accordingly, and in light of the imminence of the dates of the proposed sampling, Syntex will proceed as outlined in this letter unless it hears from you to the contrary by July 24, 1989. If you have any comments or questions, please call me at (417) 866-4050.

Very truly yours,

Gary J Pendergrass

Gary J. Pendergrass, P.E.
Manager, Environmental Projects

GJP:rlr/0454P

MEMORANDUM

November 9, 1989

AER: wp0103

TO: D. Robertson (w/ attachments)

FROM: K. Chan KD for KC
B. Berridge
K. Deloria KD

CC: K. Straub
L. Throop
L. Tokes

SUBJECT: Determination of 2,3,7,8-Tetrachlorodibenzo-p-Dioxin (2,3,7,8-TCDD) in Spring River Fish Collected in August, 1989.

As an extension of a five year study of Verona fish, the levels of 2,3,7,8-TCDD in Catostomus commersoni (white suckers) collected from Verona's Spring River were determined using Syntex Method AR# 10,349 ("Determination of 2,3,7,8-TCDD in Fish by Capillary Gas Chromatography High Resolution Mass Spectrometry Using The Selected Ion Monitoring Technique (C-GC/HRMS-SIM)"). A summary of the results is shown in Table 1.

The fish were collected at similar locations as in previous years(1984 - 1988) by the Missouri Department of Conservation (MDC) on August 22, 1989. The exact locations are described in the sampling records (Attachment 1). Subsequently, MDC prepared samples containing homogenate of fish fillets, remainders, and whole fish. Portions of each of these samples were packaged in plastic bags and were released for analysis. Syntex (c/o Dr. D. Robertson) received these samples from Ms. Cynthia S. Morris of MDC on September 26, 1989; the samples were frozen and in good condition upon arrival. At Syntex, these samples were stored frozen until just before the preparation for C-GC/HRMS-SIM analysis.

The samples were prepared for analysis by B. Berridge. 1.874 ng of ^{14}C labelled 2,3,7,8-TCDD was added to approximately 50 g of sample. The samples were saponified, extracted, and purified by column chromatography. Finally, the samples were reconstituted in 50 ul of toluene and submitted for C-GC/HRMS-SIM analysis.

These analyses were carried out by K. Chan and K. Deloria using a Finnigan-MAT 8230 mass spectrometer directly coupled with a Varian 3700 gas chromatograph. Data were obtained using Finnigan SS300 version 6.01C software. Experimental conditions are shown with the raw data in the attachments. Areas of the chromatographic peaks were obtained and reported using SS300 program "PAREA" and "PLIST". As in previous years, these data were then entered into a "TCDD Report Program" (written by B. Brunck, last revision February 11, 1988) which was executed on an IBM PC to perform linear regression analysis on the calibration curves, to calculate the amount of 2,3,7,8-TCDD in the fish samples, and to generate reports as shown in the attachments.

As quality control, a standard addition experiment was carried out. 0.0336 ng of native 2,3,7,8-TCDD was added to 45.2 g of fillet of group B fish collected at site 4 (sample I.D. MDC-15s). Analysis of this spiked sample showed a concentration of 2.1 ppt 2,3,7,8-TCDD, which is +5% from the expected value of 2.0 ppt.

In conclusion, the concentration of 2,3,7,8-TCDD in the fish samples collected from Spring River this year is in the same general order of magnitude as last year.

Attachments:
1. Sampling Records.
2. Documentation of TCDD Standards.
3. Raw data and "TCDD Reports".

TABLE 1

Concentration (in parts per trillion, ppt) of 2,3,7,8-TCDD in *Catostomus commersoni* Collected From the Verona Spring River in 1989.

Sample I.D.	Site-Group	Type	Results (ppt)
MDC-1	1-A	Fillet	4.7
MDC-2	1-B	Fillet	3.3
MDC-3	1-B	Remainder	19.3/20.0[1]
MDC-5	1-C	Whole fish	20.1
MDC-6	2-A	Fillet	3.5
MDC-7	2-B	Fillet	4.1
MDC-8	2-B	Remainder	20.7
MDC-10	3-A	Fillet	1.3
MDC-11	3-B	Fillet	1.5
MDC-12	3-B	Remainder	9.8
MDC-14	4-A	Fillet	1.6
MDC-15	4-B	Fillet	1.3
MDC-15s	4-B	Fillet	2.1[2]
MDC-16	4-B	Remainder	9.9

1. Duplicate sample preparation and analysis.
2. 0.0336 ng of native TCDD was added to 45.2 g of MDC-15 (equivalent to 0.7 ppt spike).

A-1

September 28, 1989

Mr. Robert Morby
Region VII
U.S. Environmental Protection Agency
726 Minnesota Avenue
Kansas City, Kansas 55101

Dear Mr. Morby:

On August 22, 1989 white suckers (Catostomus commersoni) were collected from four locations on the upper Spring River for TCDD analysis. This is in compliance with the sixth year of a continuing requirement outlined in the revised Verona Fish and Sediment Sampling Plan. The fish were collected by electroshocking. A representative from Syntex was present during sampling. The four sites correspond to those identified in the "Verona Plant, Fish and Sediment Plan" with the exception of Site 4 which was moved .5 miles upstream. Site 5 was dropped in 1989. These sites were identical to those sampled in August of 1984, 1985, 1986, 1987, and 1988. The sampling locations are identified in Attachment A. The size and weight of each fish and the identifying number is listed in Attachment B. The recommended minimum numbers of fish were met at all locations.

The fish were taken to our facility at Columbia, Missouri, thawed and prepared accordingly. The fish at sites 2, 3, and 4 were weighed and measured and sequentially placed into two equal size groups designated as Groups A and B. The right skinless fillets of the fish in Groups A and B were removed and placed in separate polyethylene bags. These two groups are to be analyzed separately. The remainder of Group B fish (the entire fish minus the right fillet) was placed in a third bag for analysis. A fourth whole body estimate will be calculated. The fish at Site 1 were prepared in a similar manner except they were sorted into three equal size groups. Group A and B were prepared in a manner identical to sites 2-4 and the fish in Group C were simply left whole and refrozen. Thus a total of 13 composites were prepared which will generate 17 measurements (four calculated).

The frozen fish samples were delivered to the Environmental Trace Substances Research Laboratory in Columbia, thoroughly homogenized, a maximum of 100 grams were removed, refrozen, and delivered to Dr. David Robertson, Syntex Research, Palo Alto, California by Federal Express on September 26. Excess homogenized tissue will be held at our laboratory until analyses are complete.

Sincerely,

Cynthia S. Morris
Fisheries Environmental Specialist

CSM:aa

bcc: Mr. Michaleson

Attachment B

Site 1 - Group A - Fillets Only

Total Length (mm)	Weight (kg)	MDC Number
213	.094	MDC-1
293	.249	
241	.140	
245	.129	

Site 1 - Group B - Fillets and Remainder

Total Length (mm)	Weight (kg)	MDC Number
226	.111	MDC-2 (fillets)
264	.205	and
229	.119	MDC-3 (remainder)
252	.163	and MDC-4* (to be calculated)

Site 1 - Group C - Whole Fish

Total Length (mm)	Weight (kg)	MDC Number
215	.101	MDC-5
240	.153	
261	.180	
248	.142	

Site 2 - Group A - Fillets Only

Total Length (mm)	Weight (kg)	MDC Number
287	.237	MDC-6
240	.132	
275	.214	
282	.238	

Site 2 - Group B - Fillets and Remainder

Total Length (mm)	Weight (kg)	MDC Number
289	.260	MDC-7 (fillets)
245	.136	and
243	.162	MDC-8 (remainder)
207	.083	and MDC-9* (to be calculated)

* Total weight of fillets for Group 1B fish is .081 kg for calculation purposes.

* Total weight of fillets for Group 2B fish is .096 kg for calculation purposes.

Site 3 - Group A - Fillets Only

Total Length (mm)	Weight (kg)	MDC Number
192	.076	MDC-10
247	.146	
317	.376	
243	.147	

Site 3 - Group B - Fillets and Remainder

Total Length (mm)	Weight (kg)	MDC Number
209	.098	MDC-11 (fillets)
202	.085	and
239	.159	MDC-12 (remainder)
250	.170	and MDC-13* (to be calculated)

Site 4 - Group A - Fillets Only

Total Length (mm)	Weight (kg)	MDC Number
234	.138	MDC-14
208	.090	
217	.110	
235	.129	

Site 4 - Group B - Fillets and Remainder

Total Length (mm)	Weight (kg)	MDC Number
200	.077	MDC-15 (fillets)
235	.117	and
237	.130	MDC-16 (remainder)
347	.475	and MDC-17* (to be calculated)

* Total weight of fillets for Group 3B fish is .082 kg for calculation purposes.

* Total weight of fillets for Group 4B fish is .127 kg for calculation purposes.

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Preparation of Standards for Fish Samples 9/29/89

These standards were prepared using the stock solutions prepared and verified, as shown on pages 128 - 138.

Label	Vol Native TCDD (10179-	Vol. ^{13}C-TCDD (10179-128-2)	Verified amounts: native/^{13}C-TCDD	Final Volume
2.0	0	100 µl	0 / 1.874 ng	50 µl
2.1	50 µl (-128-C)	100 µl	~~0.03316~~ 0.01658/1.874 ng	50 µl
2.2	100 µl (-128-C)	100 µl	~~0.06632~~ 0.03316/1.874 ng	50 µl
2.3	200 µl (-128-C)	100 µl	~~1.874~~ ~~0.1658~~ 0.06632/1.874 ng	50 µl
2.4	50 µl (-128-B)	100 µl	~~0.3316~~ 0.1658/1.874 ng	50 µl
2.5	100 µl (-128-B)	100 µl	~~0.6632~~ 0.3316/1.874 ng	50 µl
2.6	200 µl (-128-B)	100 µl	~~1.658~~ 0.6632/1.874 ng	50 µl
2.7	50 µl (-128-A)	100 µl	~~3.31~~ 1.658/1.874 ng	50 µl
2.8	100 µl (-128-A)	100 µl	3.316/1.874 ng	50 µl
2.9	200 µl (-128-A)	100 µl	6.632/1.874 ng	50 µl

R. Pierridge 9/29/89

Read and Understood by _____ Date _____

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National Bureau of Standards Certificate of Analysis for Standard Reference Material 1614

A-6

9/28/89

rec'd 7/21/85

Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 1614

Dioxin (2,3,7,8-TCDD in Isooctane)

Standard Reference Material (SRM) 1614 consists of separate solutions of unlabeled and labeled 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD) in 2,2,4-trimethylpentane (isooctane). Three ampoules contain approximately 1.2 mL each of an isooctane solution of unlabeled 2,3,7,8-TCDD, and three ampoules contain approximately 1.2 mL each of an isooctane solution of ^{13}C-labeled 2,3,7,8-TCDD. This SRM is intended primarily for use in the evaluation of analytical methods used in the determination of 2,3,7,8-TCDD. It can also be used to fortify samples with known amounts of 2,3,7,8-TCDD. The ^{13}C-labeled 2,3,7,8-TCDD can be used as an internal standard in methods based on gas chromatography/mass spectrometry (GC/MS).

Certified Concentrations of 2,3,7,8-TCDD

The certified concentrations and estimated uncertainties of the unlabeled and ^{13}C-labeled 2,3,7,8-TCDD solutions are given in Table 1. The concentration values are certified in ng/g units, but are also reported in ng/mL units for user convenience. The ^{13}C-labeled solution is certified for the total concentration of all isotopic forms of 2,3,7,8-TCDD. The isotopic purity of the ^{13}C-labeled material was determined to be 98.2 ± 0.1 atom percent ^{13}C by mass spectrometry. The fully ^{13}C-labeled compound, 2,3,7,8-TCDD-$^{13}C_{12}$, accounts for 80.7 ± 0.5 percent of the 2,3,7,8-TCDD molecules in the sample.

The certified values are the weighted averages of gravimetric values, based on the concentration calculated from the mass of 2,3,7,8-TCDD added to a known mass of isooctane and on the analytical results obtained using capillary gas chromatography with electron capture detection (GC/ECD). The uncertainties are two standard deviations of the certified values. These uncertainties include the gravimetric and GC measurement variability and any observed material heterogeneity.

NOTICE AND WARNING TO USERS

Handling

The toxicity and/or carcinogenicity of 2,3,7,8-TCDD has not been precisely defined; however, this material should be treated as a potential health hazard. Ampoules should be opened and the contents used only by persons trained in proper handling techniques. Techniques used in handling radioactive and infectious materials are applicable to 2,3,7,8-TCDD. Users in the United States should contact their regional offices of the U.S. Environmental Protection Agency for information regarding proper disposal of these materials; in other countries, they should contact the appropriate organization responsible for public health or environmental control.

Trimethylpentane (isooctane), used as a diluent in this SRM, is stable when stored in closed containers at room temperature. It will not undergo hazardous polymerization. However, it is highly flammable and should be kept away from oxidizing agents.

Gaithersburg, MD 20899
July 8, 1985

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

(over)

Preparation and Analysis

Samples of the unlabeled 2,3,7,8-TCDD and ^{13}C-2,3,7,8-TCDD used in the preparation of SRM 1614 were donated by R. Mitchum, National Center for Toxicological Research, Jefferson AK. The unlabeled 2,3,7,8-TCDD was originally obtained from ECO Control, Inc., Cambridge, MA; and the ^{13}C-labeled 2,3,7,8-TCDD from Midwest Research Institute, Kansas City, MO. The purities of the labeled and unlabeled 2,3,7,8-TCDD used in the preparation of this SRM were determined at NBS using mass spectrometry, nuclear magnetic resonance spectrometry, and GC with flame ionization detection. The purities were found to be greater than 95%.

Solutions of the unlabeled 2,3,7,8-TCDD and the ^{13}C-labeled 2,3,7,8-TCDD were prepared at NBS by weighing and mixing the appropriate compound and isooctane. Each solution was dispensed into 2-mL amber ampoules which were then flame sealed. Aliquots from randomly selected ampoules were analyzed with a gas chromatograph equipped for split injection and a 30m x 0.25mm i.d. wall-coated open-tubular column with a 0.25 μm film of a non-polar, immobilized phase. A constant current electron capture detector (^{63}Ni) was used for these analyses. Quantitative results were obtained through the use of 2,2',4,4',5,5'-hexachlorobiphenyl as an internal standard. Calibration solutions consisting of weighed amounts of the analyte and the internal standard compound in isooctane were analyzed chromatographically to determine response factors.

A trichlorodibenzo-p-dioxin impurity present in both solutions was quantified using GC/MS with electron impact ionization, selected ion monitoring, and the method of standard additions. Standard additions of unlabeled 2,3,7-trichlorodibenzo-p-dioxin were made to the unlabeled and ^{13}C-labeled 2,3,7,8-TCDD solutions, and although the retention time of the trichlorodibenzo-p-dioxin impurity was coincident with that of the 2,3,7-isomer, this was not sufficient to positively identify which isomer was present. Concentrations of the trichlorodibenzo-p-dioxin in the SRM solutions are provided, for information only, in Table 2.

Expiration of Certification

This certification is valid within the specified limit of uncertainty for one year from the date of purchase. In the event that the certification should become invalid before then, purchasers will be notified by NBS.

Storage

Sealed ampoules, as received, should be stored in the dark at temperatures between 10 and 30 °C. It is recommended that these materials be stored in a secure area in a double-sealed container.

Use

Samples of the SRM should be withdrawn from ampoules (at 23 ± 8 °C) immediately after opening and used without delay for the certified values listed in Table 1 to be valid within the stated uncertainties. Certified values are not applicable to material in ampoules stored after opening, even if they are resealed.

Preparation and analytical determinations were performed in the NBS Organic Analytical Research Division by S.N. Chesler, B. Coxon, L.R. Hilpert, R.M. Parris, R.E. Rebbert, M.J. Welch, and E. White. V.

Consultation on the statistical design of the experimental work and evaluation of the data was provided by R.C. Paule of the NBS National Measurement Laboratory.

The coordination of the technical measurements leading to the certification of SRM 1614 was under the direction of L.R. Hilpert, R.M. Parris, and W.E. May.

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Office of Standard Reference Materials by T.E. Gills.

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Table 1

Certified Concentrations of 2,3,7,8-TCDD[a] in SRM 1614

Compound	Concentration[b] ng/g	Concentration[b] ng/mL[c], 23 °C
2,3,7,8-TCDD	98.3 ± 3.3	67.8 ± 2.3
2,3,7,8-TCDD-$^{13}C^{d}$	95.6 ± 1.5	65.9 ± 1.0 X 80.7% = 53.2

[a]CAS Registry Numbers: 2,3,7,8-TCDD-$^{12}C_{12}$: 1746-01-6; 2,3,7,8-TCDD-$^{13}C_{12}$: 76523-40-5, Chemical Abstracts, Tenth Collective Index, Index Guide, American Chemical Society, Columbus, Ohio, 1982.

[b]The uncertainties given represent two standard deviations of the certified values. These uncertainties include the gravimetric and GC/ECD 2,3,7,8-TCDD measurement variability, the trichlorodibenzo-p-dioxin measurement variability, and, for the unlabeled 2,3,7,8-TCDD, the observed sample heterogeneity.

[c]The concentration and uncertainty expressed in mass/volume units are applicable for use of this material at 23.0 °C. Since the density of 2,2,4-trimethylpentane changes with temperature, the concentration will change at temperatures other than 23.0 °C. The concentration will change by less than 1 percent of the value listed if the SRM is used at temperatures in the 15 to 31 °C range.

[d]The concentrations given represent the total concentrations for all isotopic forms of 2,3,7,8-TCDD in the solution. The fully ^{13}C-labeled 2,3,7,8-TCDD accounts for 80.7 ± 0.5 percent of the 2,3,7,8-TCDD molecules in the sample. This value is provided for information only. X

Table 2

Concentrations of Trichlorodibenzo-p-dioxin in SRM 1614

Solution	Compound	Concentration[a] ng/g	Concentration[a] ng/mL, 23 °C
Unlabeled	trichlorodibenzo-p-dioxin-$^{12}C_{12}$	(1.5)	(1.0)
Labeled (^{13}C)	trichlorodibenzo-p-dioxin-$^{13}C_{12}$	(3.9)	(2.7)

[a]Values not certified; provided for information only.

B. Berridge 9/28/89

Read and Understood by _____ Date _____

A-8

~~Preparation of Stock Solutions for Fish Standards and Spiking Solutions~~ 9/28/89

see p. 128

~~^{13}C-TCDD~~
~~Opened DDR-10179 8b and diluted to mark.~~
~~conc. 1,000 ng/ml = 100 ng/100 µl~~

10179-

Preparation of Verification Mixtures

Opened new vials of NBS Reference Material 1614
see p. 129, 130 this notebook

VERIFICATION MIXTURE 1a " " 1b	100 µl (6.78 ng) native TCDD Cert. Std 100 µl ^{13}C-TCDD (10179-128-1)
VERIFICATION MIXTURE 2a " " 2b	200 µl native TCDD (10179-128-A) 100 µl (5.32 ng) ^{13}C-TCDD Cert. Std.
VERIFICATION MIXTURE 3a " " 3b	200 µl native TCDD (10179-128-A) 100 µl ^{13}C-TCDD (10179-128-1)

Calculation of Verified Amounts

VERIFICATION MIXTURE 1a $\frac{\text{native}}{^{13}\text{C-TCDD}} = \frac{82435}{123271} = 0.6687$ } 0.6716

1b $\frac{71076}{105381} = 0.6745$

2a $\frac{65704}{56782} = 1.1571$ } 1.1574

2b $\frac{55937}{48321} = 1.1576$

3a $\frac{57426}{86751} = 0.6620$ } 0.6570

3b $\frac{37408}{57367} = 0.6521$

VER. MIX 3 ÷ VER. MIX 1 = 0.6570 ÷ 0.6716 = 0.9783

$\frac{200\,\mu l \text{ native TCDD (-128-A)}}{\cancel{100\,\mu l\ ^{13}\text{C-TCDD (-128-1)}}} \times \frac{\cancel{100\,\mu l\ ^{13}\text{C-TCDD (-128-1)}}}{100\,\mu l\ (6.78\,ng) \text{ native Cert Std}} = \cancel{0.9786}\ 0.9783$

200 µl native TCDD (-128-A) = ~~4.5534~~ 6.6326

$\boxed{\text{native TCDD (-128-A)} = \cancel{2.2767/100\,\mu l}\ 3.316\,ng/100\,\mu l}$ Verified Amt.

VER. MIX 2 ÷ VER. MIX 3 = 1.1574 ÷ 0.6570 = 1.7616

$\frac{\cancel{200\,\mu l \text{ native TCDD (-128-A)}}}{100\,\mu l\ (5.32\,ng)\ ^{13}\text{C-TCDD Cert. Std.}} \times \frac{100\,\mu l\ ^{13}\text{C-TCDD (-128-1)}}{\cancel{200\,\mu l \text{ native TCDD (-128-A)}}} = 1.7616$

$\boxed{100\,\mu l\ ^{13}\text{C-TCDD (-128-1)} = 9.372\,ng/100\,\mu l}$ Verified Amt.

Raw data appears on pages 133 through 138.

[illegible] 9/25/89

A-10

VERIFICATION MIXTURE 1a

*** Area Percent ***

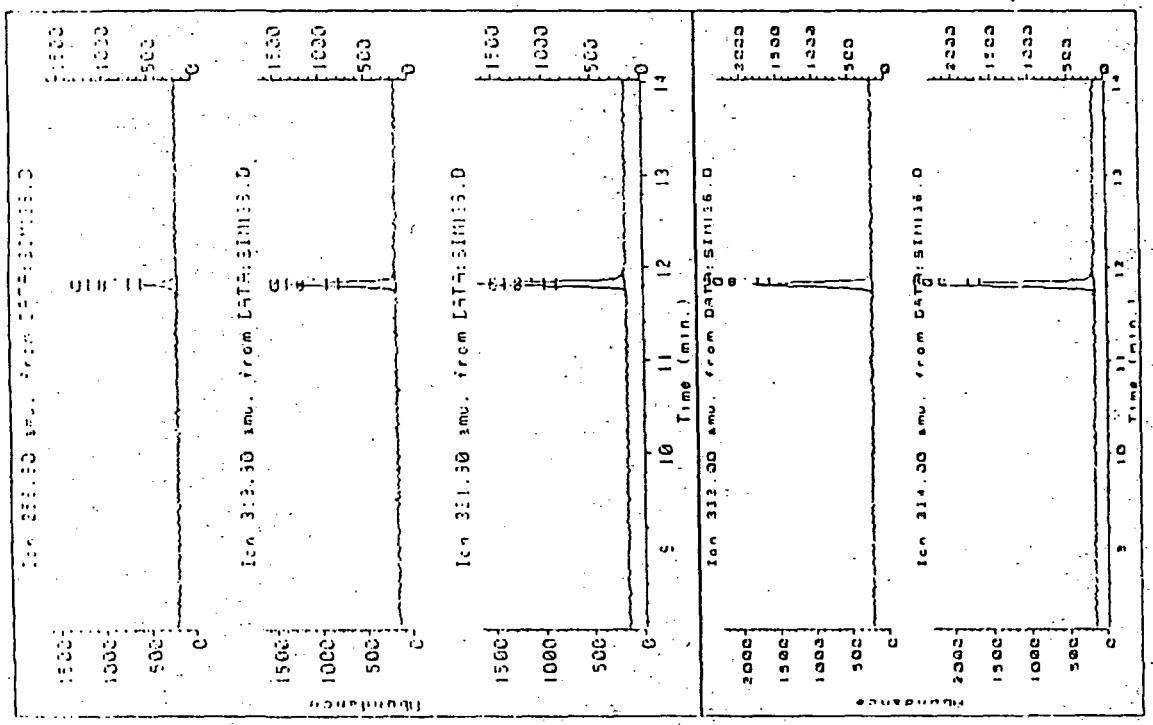
Report by Retention Time

Operator: B. BERRIDGE 28 Sep 89 9:15 am
Method File Name : SIMTCDD.M
Sample Info : VERIFICATION MIXTURE 1a 9/27/89
Misc Info: SIM136
Integration File Name : RESULTS.I
Bottle Number : 0

Ret Time	Signal Descr		Type	Area	Height	% Pk	% Sq	% LPk	% LSq
11.809	Mass	334.00 amu	BV	68200	2062	31.21	100.00	100.00	100.00
11.806	Mass	332.00 amu	BB	55071	1646	25.20	100.00	80.75	100.00
11.813	Mass	321.90 amu	BV	45370	1420	20.76	100.00	65.53	100.00
11.810	Mass	319.90 amu	PV	37065	1120	16.96	100.00	54.35	100.00
11.810	Mass	256.90 amu	VV	12838	372	5.87	100.00	18.82	100.00

$$\frac{\Sigma\ m/z\ 334 + m/z\ 332}{\Sigma\ m/z\ 322 + m/z\ 320}$$

$$\frac{82435}{123271} = 0.6687$$



Read and Understood by

VERIFICATION MIXTURE 1b

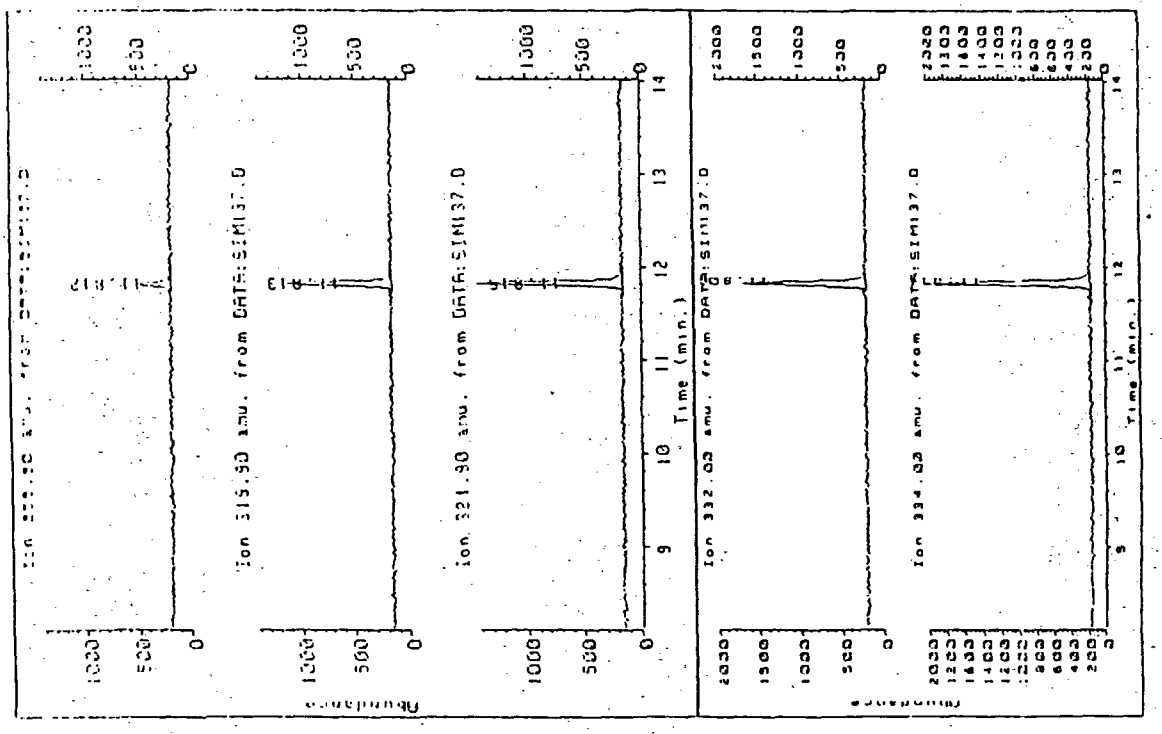
*** Area Percent ***

Report by Retention Time

Operator: B. BERRIDGE 28 Sep 89 9:53 am
Method File Name : SIMTCDD.M
Sample Info : VERIFICATION MIXTURE 1b 9/27/89
Misc Info: SIM137
Integration File Name : RESULTS.I
Bottle Number : 0

Ret Time	Signal Descr	Type	Area	Height	% Pk	% Sg	% LPk	% LSg
11.811	Mass 334.00 amu	VV	57916	1775	31.11	100.00	100.00	100.00
11.808	Mass 332.00 amu	VV	47465	1442	25.50	100.00	81.56	100.00
11.815	Mass 321.90 amu	PV	39648	1220	21.30	100.00	68.46	100.00
11.813	Mass 319.90 amu	VV	31428	946	16.88	100.00	54.27	100.00
11.812	Mass 256.90 amu	VV	9677	294	5.20	100.00	16.71	100.00

$$\frac{71076}{105391} = 0.6745$$



A-12

VERIFICATION MIXTURE 2a

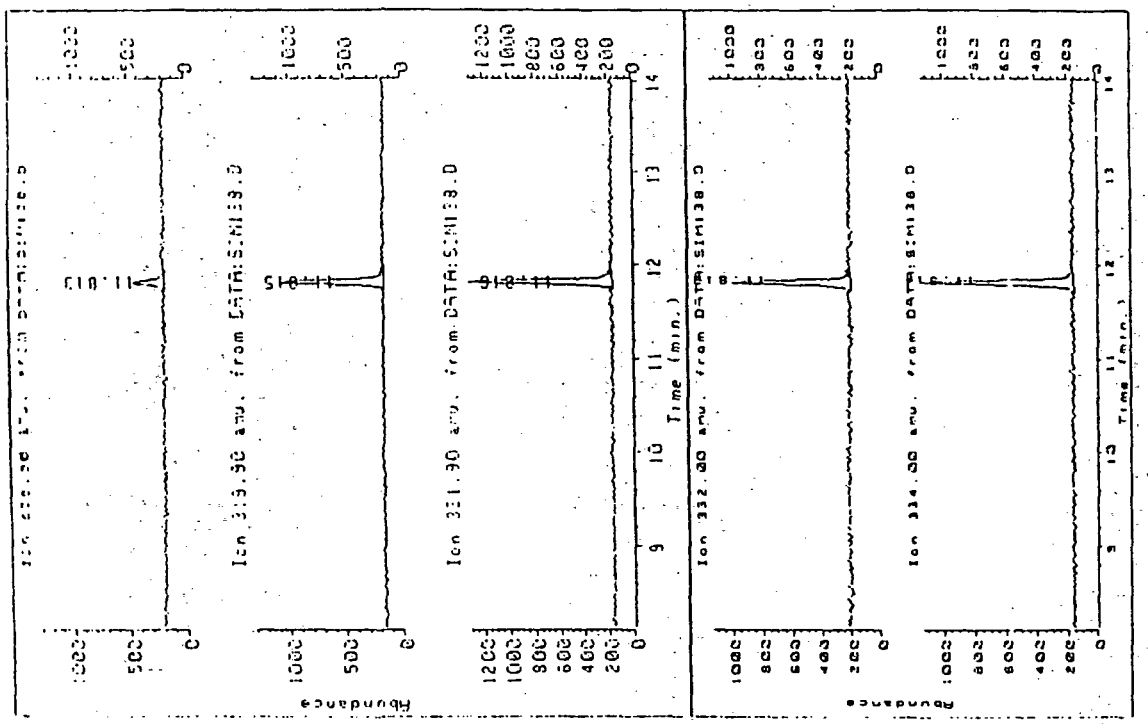
*** Area Percent ***

Report by Retention Time

Operator: B. BERRIDGE 28 Sep 89 10:30 am
Method File Name : SIMTCCO.M
Sample Info : VERIFICATION MIXTURE 2a 9/27/89
Misc Info: SIM138
Integration File Name : RESULTS.I
Bottle Number : 0

Ret Time	Signal Descr	Type	Area	Height	% Pk	% Sq	% LPk	% LSq
11.814	Mass 334.00 amu	VV	31038	842	23.45	100.00	65.94	100.00
11.813	Mass 332.00 amu	BV	25744	775	19.45	100.00	71.28	100.00
11.816	Mass 321.90 amu	PV	36117	1126	27.28	100.00	100.00	100.00
11.815	Mass 319.90 amu	VV	29587	904	22.35	100.00	81.92	100.00
11.813	Mass 256.90 amu	VV	9890	273	7.47	100.00	27.38	100.00

$$\frac{65704}{56782} = 1.1571$$



VERIFICATION MIXTURE 2b

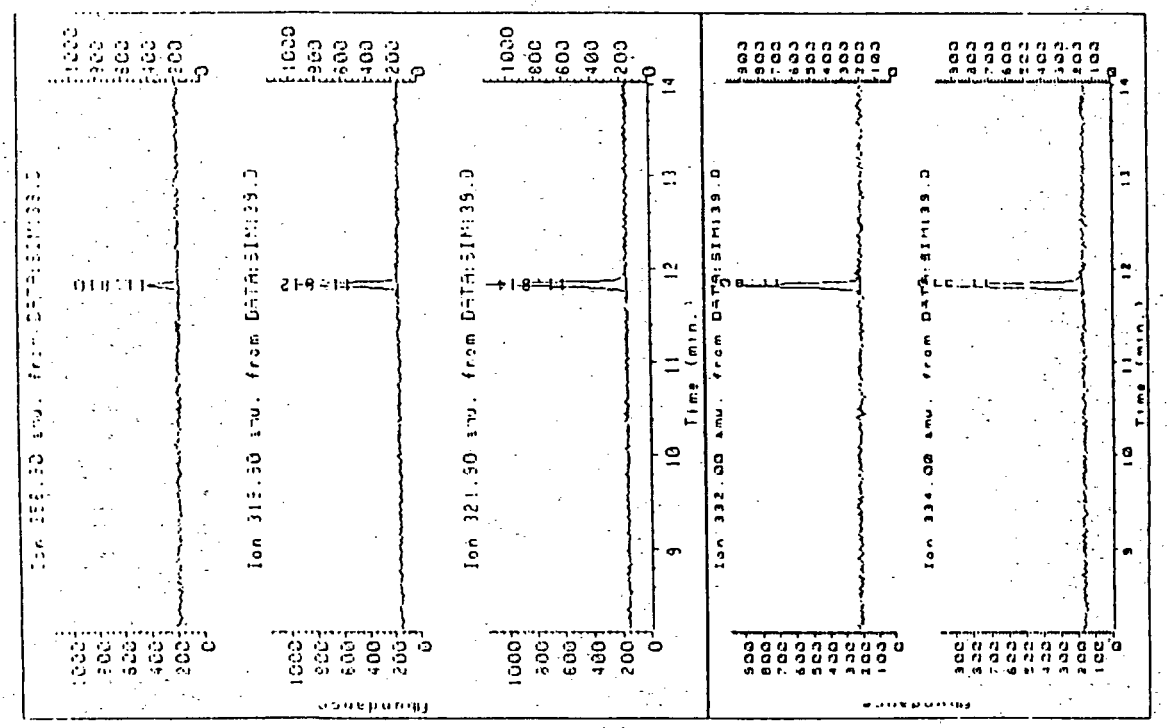
*** Area Percent ***

Report by Retention Time

Operator: B. BERRIDGE 28 Sep 89 11:07 am
Method File Name : SIMTCCO.M
Sample Info : VERIFICATION MIXTURE 2b 9/27/89
Misc Info: SIM139
Integration File Name : RESULTS.I
Bottle Number : 0

Ret Time	Signal Descr	Type	Area	Height	% Pk	% Sg	% LPk	% LSg
11.810	Mass 334.00 amu	PV	25224	825	23.31	100.00	82.14	100.00
11.807	Mass 332.00 amu	VV	22087	691	19.64	100.00	69.21	100.00
11.814	Mass 321.90 amu	BB	31926	955	28.38	100.00	100.00	100.00
11.812	Mass 319.90 amu	VV	24011	737	21.34	100.00	75.21	100.00
11.810	Mass 256.90 amu	PV	8243	245	7.33	100.00	25.82	100.00

$$\frac{55937}{48321} = 1.1576$$



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VERIFICATION MIXTURE 3a

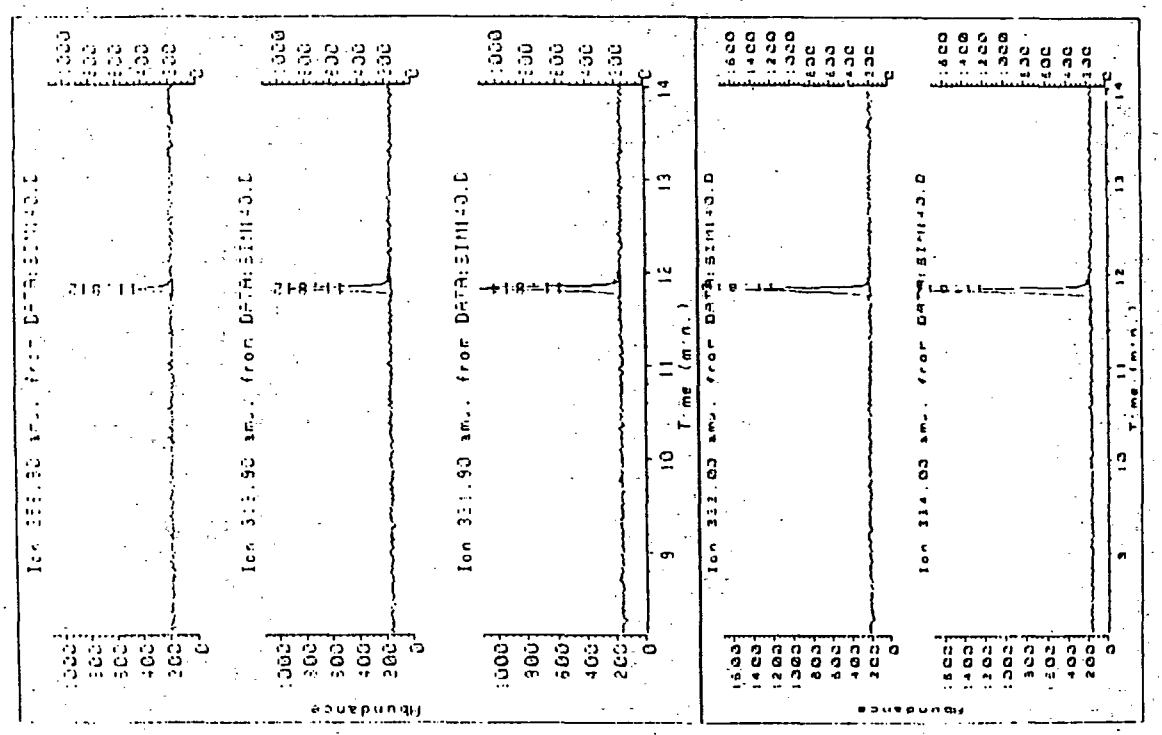
*** Area Percent ***

Report by Retention Time

Operator: B. EERRIDGE 28 Sep 89 11:38 am
Method File Name : SIMTCCO.M
Sample Info : VERIFICATION MIXTURE 3a 9/27/89
Misc Info: SIM140
Integration File Name : RESULTS.I
Bottle Number : 0

Ret Time	Signal Descr		Type	Area	Height	% Pk	% Sg	% LPk	% LSg
11.811	Mass	334.00 amu	PV	47776	1480	31.24	100.00	100.00	100.00
11.810	Mass	332.00 amu	VV	38975	1212	25.48	100.00	81.58	100.00
11.814	Mass	321.90 amu	VB	31670	919	20.71	100.00	66.29	100.00
11.812	Mass	319.90 amu	PV	25756	770	16.84	100.00	53.91	100.00
11.812	Mass	256.90 amu	BV	8764	267	5.73	100.00	18.34	100.00

$$\frac{57426}{86751} = 0.6620$$



VERIFICATION MIXTURE 3b

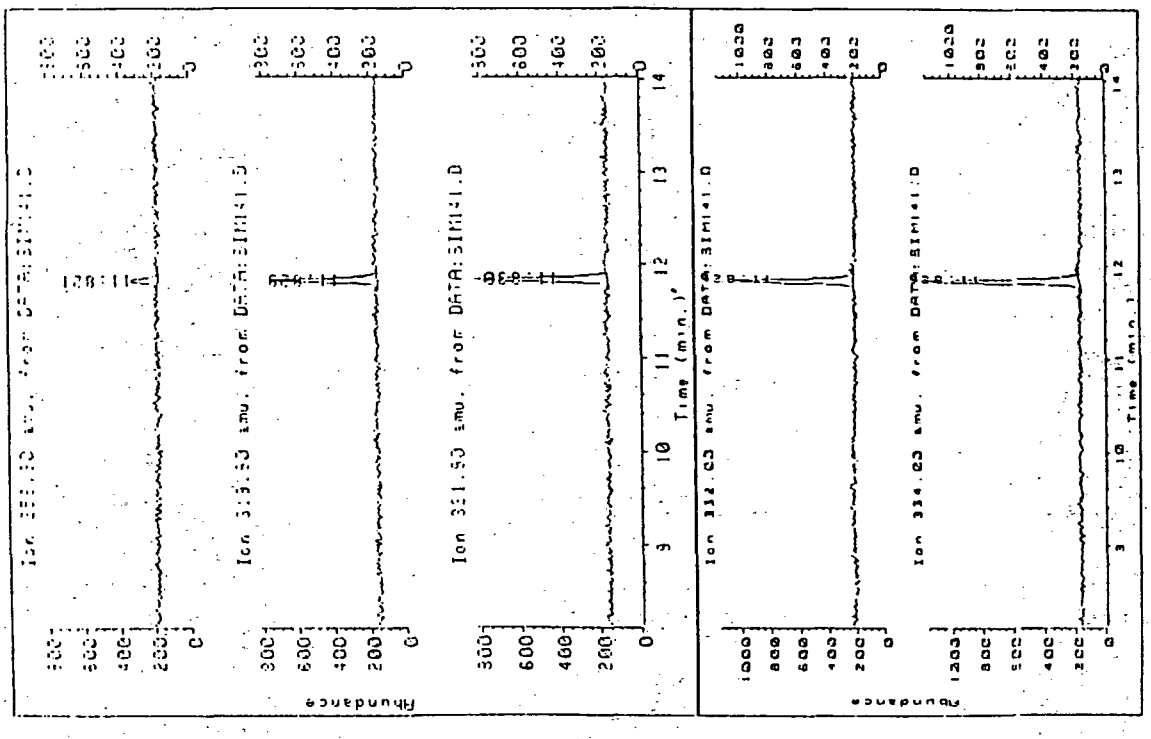
*** Area Percent ***

Report by Retention Time

Operator: B. BERRIDGE 28 Sep 89 12:53 pm
Method File Name : SIMFCDO.M
Sample Info : VERIFICATION MIXTURE 3b 9/27/89
Misc Info: SIM141
Integration File Name : RESULTS.I
Bottle Number : 0

Ret Time	Signal Descr	Type	Area	Height	% Pk	% Sg	% LPk	% LSg
11.828	Mass 334.00 amu	PV	31463	957	31.42	100.00	100.00	100.00
11.828	Mass 332.00 amu	BV	25904	799	25.86	100.00	82.33	100.00
11.830	Mass 321.90 amu	BV	20342	641	20.31	100.00	64.65	100.00
11.828	Mass 319.90 amu	VV	17066	564	17.04	100.00	54.24	100.00
11.821	Mass 256.90 amu	BV	5378	152	5.37	100.00	17.09	100.00

$$\frac{37408}{57367} = 0.6521$$



Preparation of Standards for Fish Samples 9/29/89

These standards were prepared using the stock solutions prepared and verified, as shown on pages 128 – 138.

Label	Vol. Native TCDD (10179-	Vol. ^{13}C-TCDD (10179-128-2)	Verified amounts: native/^{13}C-TCDD	Final Volume
2.0	0	100 µl	0 / 1.874 ng	50 µl
2.1	50 µl (-128-C)	100 µl	~~0.03316~~ 0.01658/1.874 ng	50 µl
2.2	100 µl (-128-C)	100 µl	~~0.06632~~ 0.0336/1.874 ng	50 µl
2.3	200 µl (-128-C)	100 µl	~~1.874~~ ~~0.1658~~ 0.06632/1.874 ng	50 µl
2.4	50 µl (-128-B)	100 µl	~~0.3316~~ 0.1658/1.874 ng	50 µl
2.5	100 µl (-128-B)	100 µl	~~0.6632~~ 0.3316/1.874 ng	50 µl
2.6	200 µl (-128-B)	100 µl	~~1.658~~ 0.6632/1.874 ng	50 µl
2.7	50 µl (-128-A)	100 µl	~~3.31~~ 1.658/1.874 ng	50 µl
2.8	100 µl (-128-A)	100 µl	3.316/1.874 ng	50 µl
2.9	200 µl (-128-A)	100 µl	6.632/1.874 ng	50 µl

B. Berridge 9/29/89

Analysis Name: DM00:[300,303]TCDD7.MIS;34 Window: 1

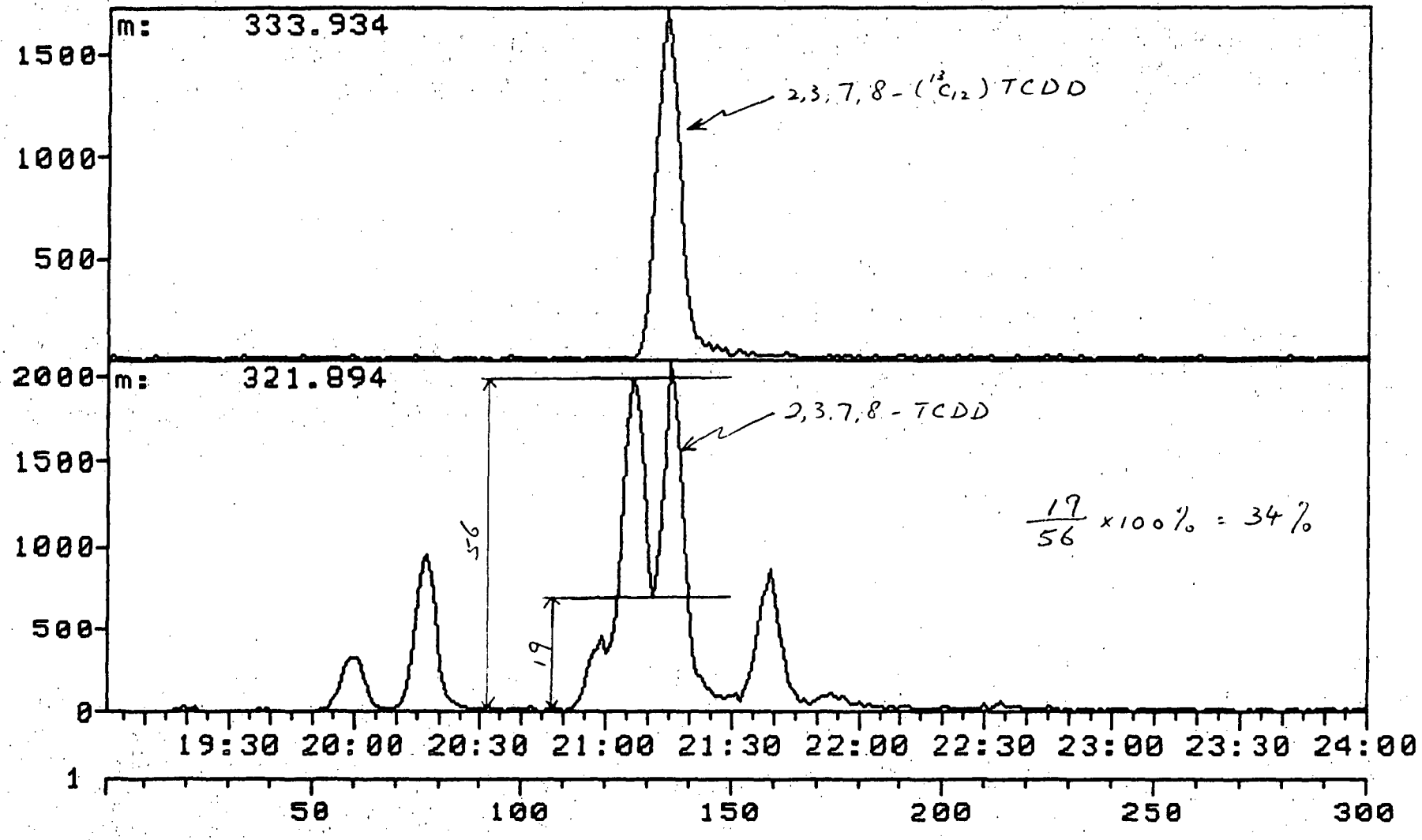
Operator: KC/KD Spc: 5

Sample ID: 7 ISO MIX Date: 11-OCT-89 11:42:34

Commission: R=7000 10-11-89

50M HP-5 160(2) 25/M 270(20) SEM 1.6KV 1UL

100HZ FILTER 12PTS/GC PEAK



A-17

A-18

Date 10/12/89

MAT 8230 C-GC-MS CONDITIONS

Column HP-5 50m x 0.2mm 0.33μm film

Injector 250°C splitless 0.7min delay 30psi He

GC Oven Program 160°C (2min) $\xrightarrow{25°C/min}$ 270°C (20min)

Separator 280°C (688)

Line of Sight 290°C (678)

Ion Source EI/CI 220°C 1mA 70eV

Ua/Ub 825

Y1 129

Y2 062

X1 574

X2 440

L1 431

L2 532

S1 1

Z1 536

Z2 548

P 544

Filter 100Hz

Multiplier 1.4KV (150)

MSCHAR 200:10:40:25:6:131000 (315.4 magnet rest mass)

HR Slits 494 525 Resolution 7100

LR Slits 578 800 Resolution 1000

HR : Ion 331 Resp.(V) 0.5 LR : Ion 331 Resp.(V) 5

LR/HR Resp. 10

txt01:hrgcms.txt

SSX: MASSMENU TCDD7.MMN;2
Finnigan MAT Oct 12 89 12:55:00 Page: 4

Beginning MASS MENU creation

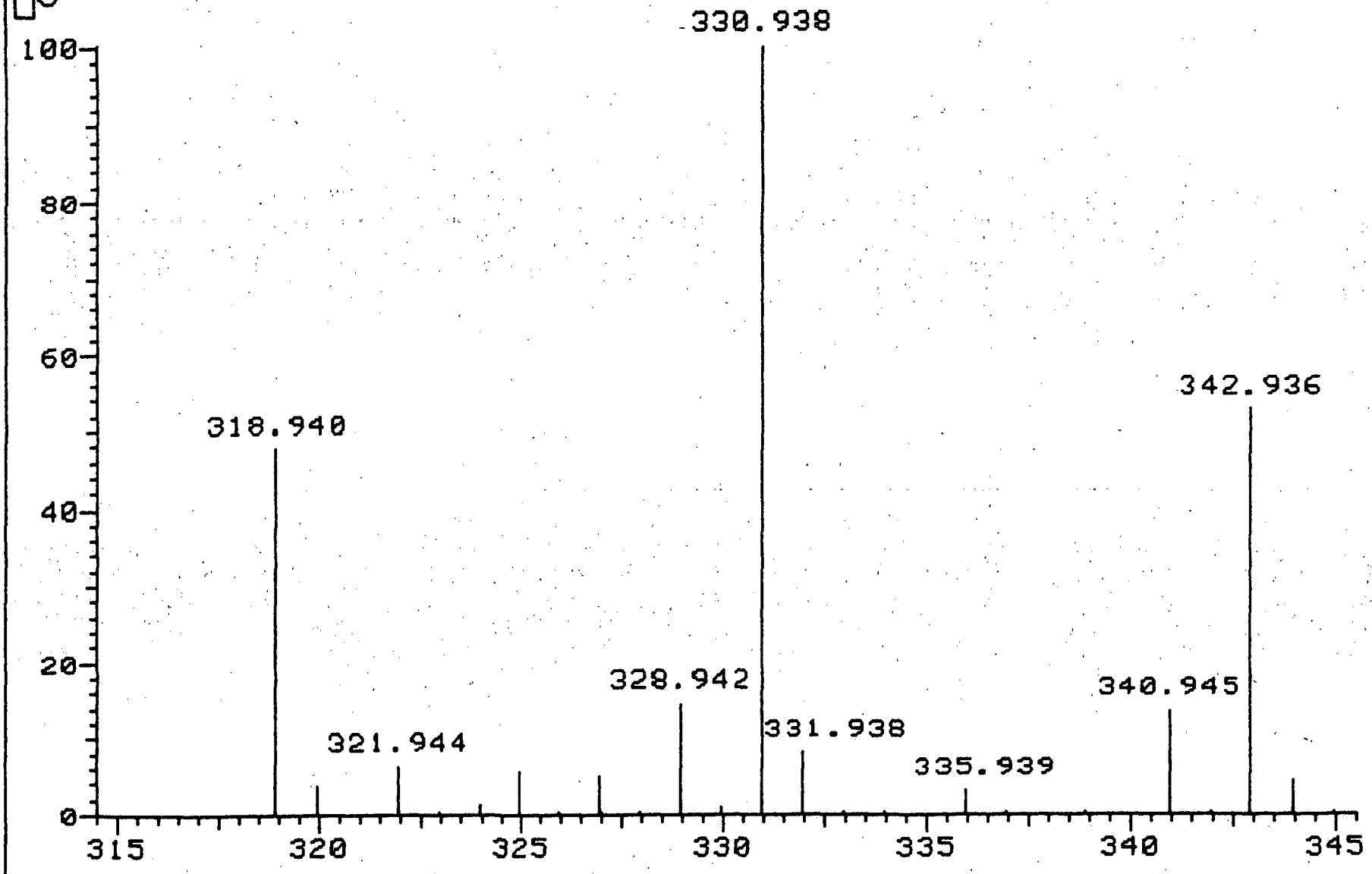
Magnet settle time 200 msec
Magnet jump time 1000 usec
Magnet focus time 40 msec
EDAC jump time 25 msec
EDAC focus time 6 msec
EDAC capabilities: rated = 131000, calibrated = 661890
used = 661890

Window number 1

from 2164 to 3030

MASS	MDAC	EDAC	DELDAC	NSTEP	NSUM	CYCTIME
47671	47671	120633	100012	24	0	2734
47771	0	173751	0	0	20	2734
50171	0	163754	0	0	20	2734
51277	0	120633	0	0	1	2734
51374	0	115154	0	0	4	2734
51574	0	105621	0	0	4	2734

***** MASS MENU Processing complete *****



Analysis Name: PFK.DAT;5 Spec# 3 Norm: B /Scale: 4468
Date: OCT 12 89 09:30:37 Nmparam: 0.5:0.5 Tolerance: 500:MMU

SSX: EDAC CALIBRATION RESULTS
Finnigan MAT Oct 12 89 09:27:10 Page: 1

MASS CORRESPONDENCE:

SAMPLE PEAK NUMBER	SAMPLE MASS	REFERENCE MASS
1	318.9325	318.9793
22	343.2122	342.9793

EDAC CONTROL OF MASS RANGE = 1.1099

X-ACT VALUE = 661890

**** ECAL PROCESSING COMPLETE ****

SSX: SLIST of DM00:[300,303]PFK.DAT;2
Finnigan MAT Oct 12 89 09:23:32 Page: 1

Spectrum Number: 2
Number of Peaks: 265

Norm. Factors: 1. 1127.20

PEAK#	MASS	A	B
21	50.000	1264.	1.12
22	50.016	2000.	1.77
23	51.000	3360.	2.98
24	51.016	2544.	2.26
29	55.094	3040.	2.70
30	56.141	2656.	2.36
32	57.125	13520.	11.99
36	63.094	2512.	2.23
39	65.125	3392.	3.01
43	69.016	112720.	100.00
44	69.109	2048.	1.82
45	70.031	1264.	1.12
46	70.094	1744.	1.55
47	71.125	8880.	7.88
50	73.078	1232.	1.09
66	85.031	4736.	4.20
68	88.984	1456.	1.29
70	91.031	20448.	18.14
71	92.047	13648.	12.11
72	92.984	4112.	3.65
75	96.016	2128.	1.89
81	99.141	1584.	1.41
82	100.031	5440.	4.83
89	113.063	1680.	1.49
94	119.000	28448.	25.24
99	131.031	30416.	26.98
100	132.031	1328.	1.18
101	133.063	2592.	2.30
105	143.047	3712.	3.29
109	150.016	1520.	1.35
110	151.031	1872.	1.66
111	155.016	2512.	2.23
114	162.000	2864.	2.54
119	169.016	23552.	20.89
126	181.016	22032.	19.55
128	186.016	1520.	1.35
129	191.016	1632.	1.45
130	193.000	4896.	4.34
136	205.000	3648.	3.24
137	207.063	11248.	9.98
139	208.063	2288.	2.03
140	209.047	1728.	1.53
142	212.016	1344.	1.19
144	217.000	1872.	1.66
145	218.984	11248.	9.98
149	231.031	11952.	10.60
152	236.000	2032.	1.80
154	243.000	7088.	6.29

SSX: SLIST of DM00:[300,303]PFK.DAT;2
Finnigan MAT Oct 12 89 09:23:32 Page: 2

Spectrum Number 2 continued

PEAK#	MASS	A	B
160	254.969	3472.	3.08
165	267.047	2960.	2.63
166	269.031	5488.	4.87
170	281.000	9264.	8.22
171	281.078	1856.	1.65
179	293.000	5296.	4.70
183	305.031	1920.	1.70
188	317.016	1632.	1.45
189	318.984	3376.	3.00
193	331.000	6176.	5.48
197	343.000	3296.	2.92
202	355.031	1584.	1.41
213	381.000	3520.	3.12
218	392.953	2192.	1.94
220	405.000	1264.	1.12
225	431.016	2800.	2.48
228	443.000	1952.	1.73
230	455.000	1184.	1.05
235	481.016	2432.	2.16
239	493.016	1440.	1.28
247	531.000	1200.	1.06
250	543.000	1200.	1.06
252	555.047	1136.	1.01
254	567.016	1200.	1.06
256	580.969	1248.	1.11

***** SLIST processing complete. *****

TCDD Report Program HRTCDD.COM
2,3,7,8-TCDD by C-GC/HRMS-SIM
Revised - February 11, 1988

AER Log No.	Sample I.D.	Date Sampled	Date Extrcted	Sample Wt.(g)	Result TCDD(ppt)	320/322	332/334	Notes
				50.0	ND(0.11)	--	0.78	1
	MDC-6	8-22-89	10-9-89	51.4	3.5	0.69	0.83	
	MDC-10	8-22-89	10-5-89	50.9	1.3	0.78	0.78	
	MDC-14	8-22-89	10-9-89	50.2	1.6	0.78	0.83	

1. Detection limit was calculated from the noise level in STD1 and 50g sample

ppt - parts per trillion
ND - None Detected (detection limit)

File : 00125 Created : October 12, 1989 17:15
Printed : October 25, 1989 16:47

Raw Data for Sample Entries

AER Log No.	13C12-TCDD Spike(ng)	Data Type	ion 257	ion 319.897	ion 321.894	ion 331.937	ion 333.934	(320+322) / (332+334)
	1.874	Height		0.3-N	0.3-N	294	377	0.00224
	1.874	Area		43.82	63.31	658.5	789.0	0.07401
	1.874	Area		13.85	17.86	539.1	687.5	0.02585
	1.874	Area		27.14	34.89	872.1	1054.0	0.03220

N - Noise Level
I - Interfering Peak Level

Raw Data for Standard Entries

TCDD (ng) Native	13C12	Data Type	ion 257	ion 319.897	ion 321.894	ion 331.937	ion 333.934	(320+322) / (332+334)
0.01658	1.874	Area		11.14	14.48	1868	2474	0.00590
		Height		2	2	294	377	0.00596
0.03316	1.874	Area		77.52	96.90	5443	6940	0.01409
		Height		12	13	778	1012	0.01397
0.06632	1.874	Area		141.5	177.4	4914	6168	0.02878
		Height		24	27	747	985	0.02945
0.1658	1.874	Area		94.37	119.7	1432	1835	0.06552
		Height		15	18	217	286	0.06561
0.6632	1.874	Area		291.3	339.0	978.6	1316	0.27469
		Height		47	56	153	204	0.28852
1.658	1.874	Area		1070	1370	1537	2013	0.68732
		Height		171	213	244	303	0.70201

File : 00125

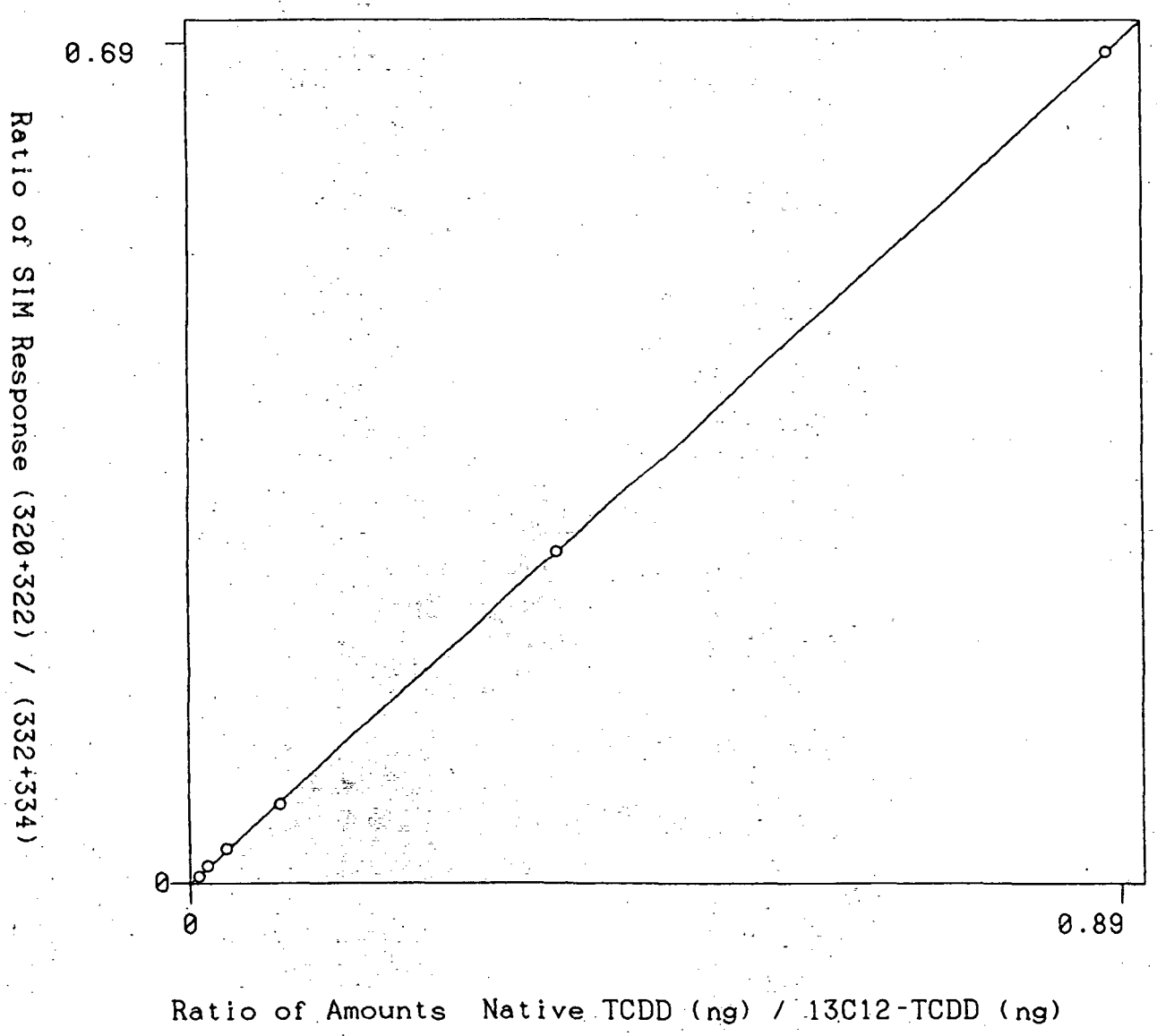
Created : October 12, 1989 17:15
Printed : October 25, 1989 16:47

X = Ratio of Amounts Native TCDD (ng) / 13C12-TCDD (ng)
Y = Ratio of SIM Areas (320 + 322) / (332 + 334)

X	Y	Y (Reg.)	% Rel. Diff (Y)
0.00885	0.00590	0.00629	-6.34
0.01769	0.01409	0.01316	6.76
0.03539	0.02878	0.02692	6.66
0.08847	0.06552	0.06819	-3.98
0.35390	0.27469	0.27453	0.06
0.88474	0.68732	0.68721	0.02

Y = -0.00059 + 0.77741 (X)

Correlation Coefficient : 0.9999839
Standard Error of Estimate : 0.0017011



File : 00125 Created : October 12, 1989 17:15
Printed : October 25, 1989 16:47

Analysis Name: DM00:[300,303]STD1.MIS;20 Window: 1

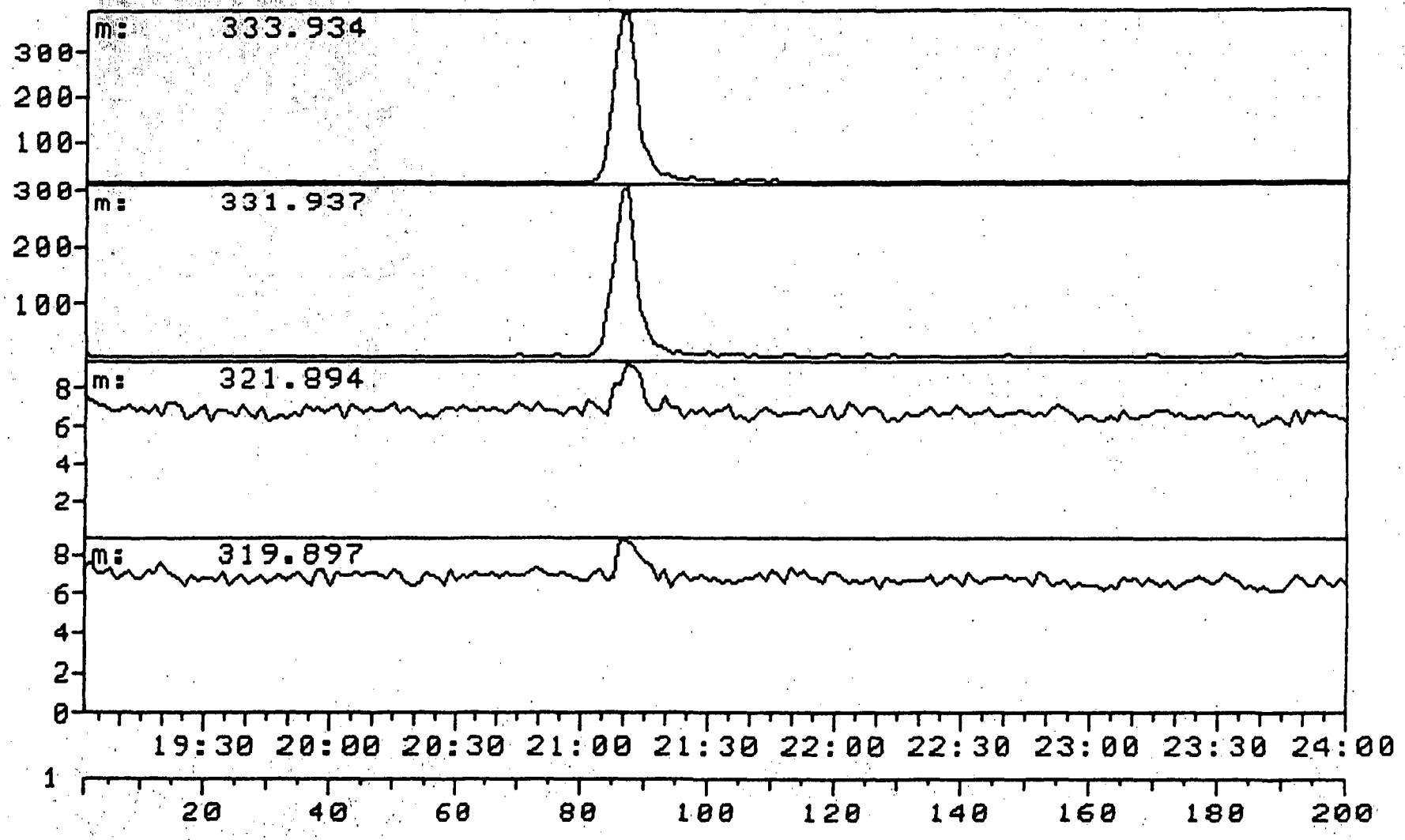
Operator: KC/KD Spc: 5

Sample ID: STD 2.1 Date: 12-OCT-89 12:55:39

Commission: R=7000 10-12-89

50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5UL

100HZ FILTER 8 CYCLE/GC PEAK



A-27

SSX: PLIST of DM00:[300,303]STD1.PEA;13
Finnigan MAT Oct 12 89 15:58:53 Page: 1

Calibration curve not well-defined for
Area , Height - use PPLOT to examine data
Sample Identification: STD 2.1
Filename: DM00:[300,303]STD1.MIS;20
Creation Date & Time: 12-OCT-89 12:55:39
Integrated Area: 4.368E+03 Integrated Height: 675
Maximum Area: 2.474E+03 Maximum Height: 377
Comment: 50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5U
L

Time Centroid	AREA Abs	AREA Base	AREA Sum	HEIGHT Abs	HEIGHT Base	HEIGHT Sum
Trace:	319.8965					
21:10	1.114E+01	0.45	0.26	2	0.53	0.30
Trace:	321.8937					
21:11	1.448E+01	0.59	0.33	2	0.53	0.30
Trace:	331.9368					
21:10	1.868E+03	75.51	42.77	294	77.98	43.56
Trace:	333.9339					
21:10	2.474E+03	100.00	56.64	377	100.00	55.85

**** PLIST Processing complete ****

Analysis Name: DM00:[300,303]STD2.MIS;15 Window: 1

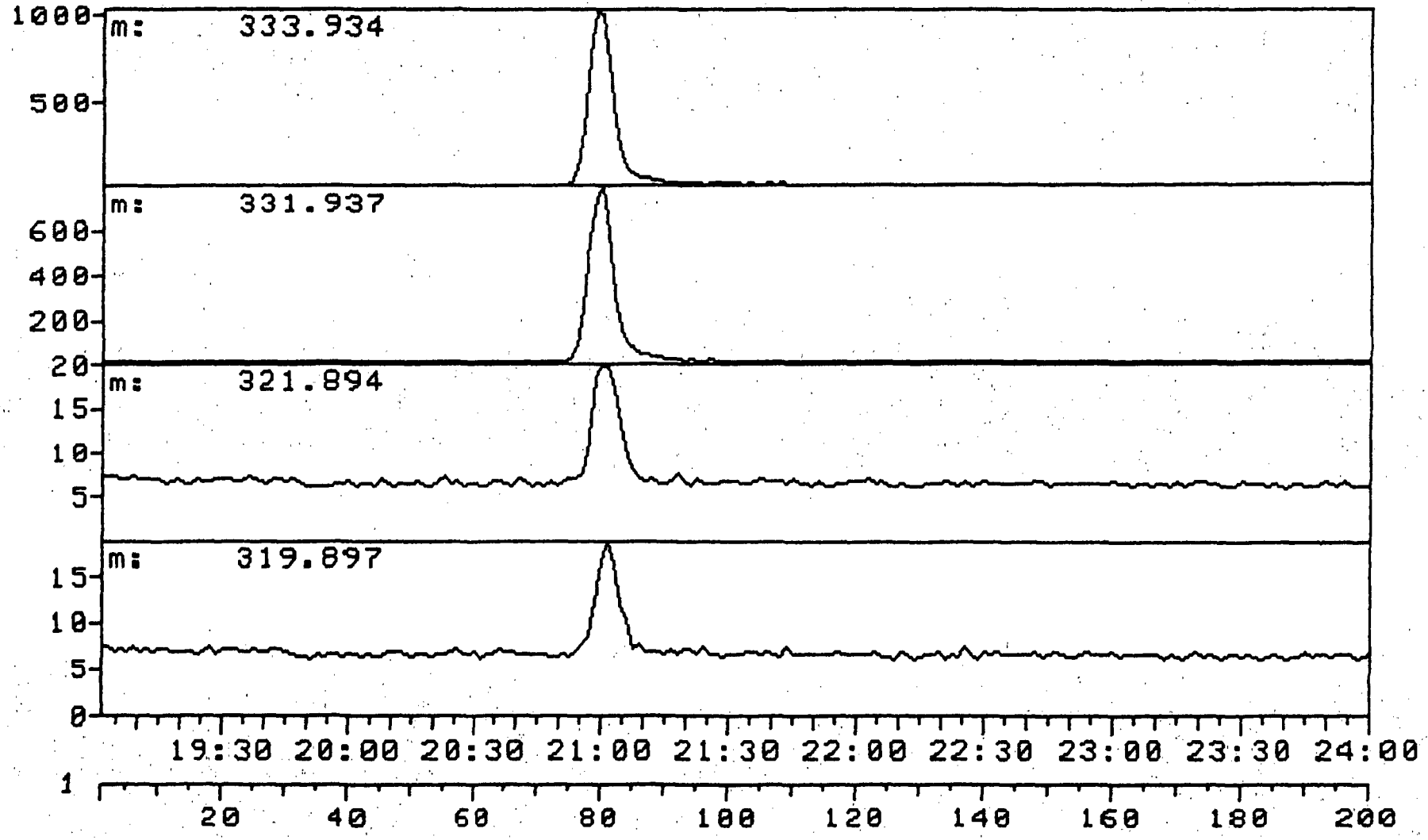
Operator: KC/KD Spc: 5

Sample ID: STD 2.2 Date: 12-OCT-89 13:29:09

Commission: R=7000 10-12-89

50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5UL

100HZ FILTER 8 CYCLE/GC PEAK



A-29

Calibration curve not well-defined for
Area , Height - use PPLOT to examine data
Sample Identification: STD 2.2
Filename: DM00:[300,303]STD2.MIS;15
Creation Date & Time: 12-OCT-89 13:29:09
Integrated Area: 1.256E+04 Integrated Height: 1815
Maximum Area: 6.940E+03 Maximum Height: 1012
Comment: 50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5U
L

Time Centroid	AREA Abs	AREA Base	AREA Sum	HEIGHT Abs	HEIGHT Base	HEIGHT Sum
Trace:	319.8965					
21:01	7.752E+01	1.12	0.62	12	1.19	0.66
Trace:	321.8937					
21:00	9.690E+01	1.40	0.77	13	1.28	0.72
Trace:	331.9368					
21:00	5.443E+03	78.44	43.35	778	76.88	42.87
Trace:	333.9339					
21:00	6.940E+03	100.00	55.26	1012	100.00	55.76

**** PLIST Processing complete ****

Analysis Name: DM00:[300,303]STD3.MIS;14 Window: 1

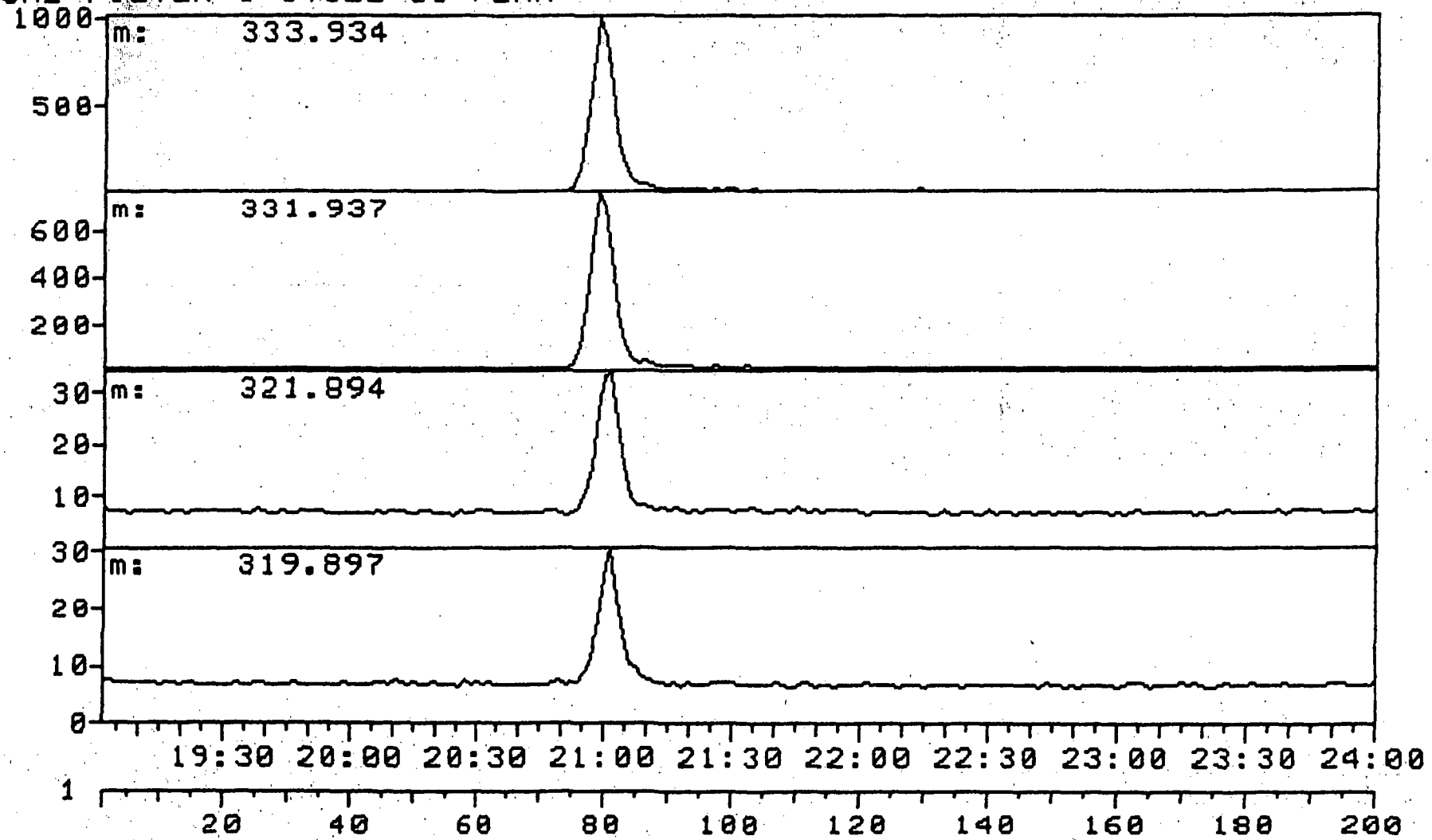
Operator: KC/KD Spc: 5

Sample ID: STD 2.3 Date: 12-OCT-89 13:57:45

Commission: R=7000 10-12-89

50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5UL

100HZ FILTER 8 CYCLE/GC PEAK



A-31

SSX: PLIST of DM00:[300,303]STD3.PEA;13
Finnigan MAT Oct 12 89 14:31:05 Page: 1

Calibration curve not well-defined for
Area , Height - use PPLOT to examine data
Sample Identification: STD 2.3
Filename: DM00:[300,303]STD3.MIS;14
Creation Date & Time: 12-OCT-89 13:57:45
Integrated Area: 1.140E+04 Integrated Height: 1783
Maximum Area: 6.168E+03 Maximum Height: 985
Comment: 50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5U
L

Time Centroid	AREA Abs	AREA Base	AREA Sum	HEIGHT Abs	HEIGHT Base	HEIGHT Sum
Trace: 319.8965						
21:01	1.415E+02	2.29	1.24	24	2.44	1.35
Trace: 321.8937						
21:01	1.774E+02	2.88	1.56	27	2.74	1.51
Trace: 331.9368						
20:59	4.914E+03	79.67	43.10	747	75.84	41.90
Trace: 333.9339						
20:59	6.168E+03	100.00	54.10	985	100.00	55.24

**** PLIST Processing complete ****

Analysis Name: DM00:[300,303]STD4.MIS;11 Window: 1

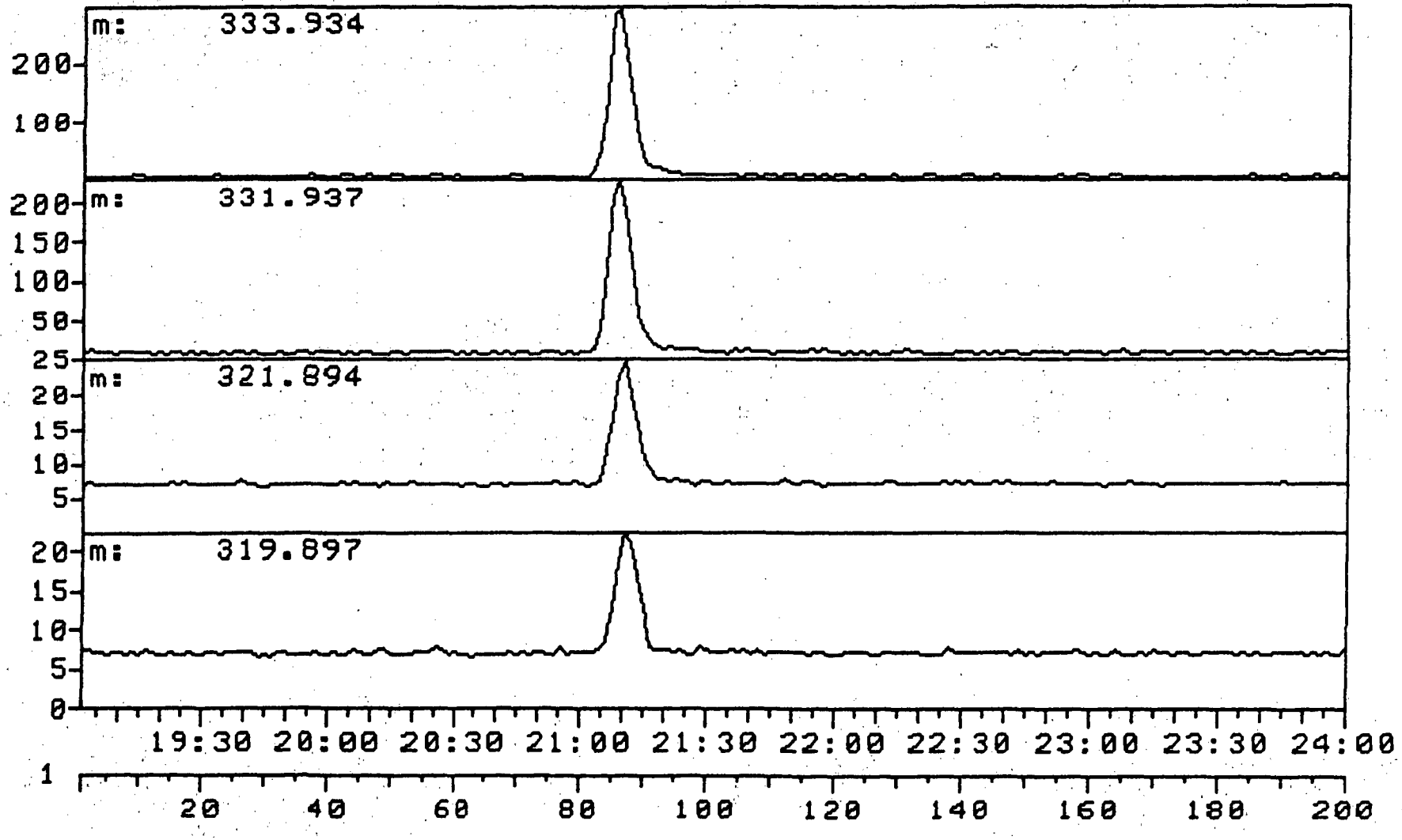
Operator: KC/KD Spc: 5

Sample ID: STD 2.4 Date: 12-OCT-89 14:27:46

Commission: R=7000 10-12-89

50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5UL

100HZ FILTER 8 CYCLE/GC PEAK



A-33

SSX: PLIST of DM00:[300,303]STD4.PEA;12
Finnigan MAT Oct 12 89 15:00:47 Page: 1

Calibration curve not well-defined for
Area , Height - use PPLOT to examine data
Sample Identification: STD 2.4
Filename: DM00:[300,303]STD4.MIS;11
Creation Date & Time: 12-OCT-89 14:27:46
Integrated Area: 3.481E+03 Integrated Height: 536
Maximum Area: 1.835E+03 Maximum Height: 286
Comment: 50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5U
L

Time Centroid	AREA Abs	AREA Base	AREA Sum	HEIGHT Abs	HEIGHT Base	HEIGHT Sum
Trace:	319.8965					
21:11	9.437E+01	5.14	2.71	15	5.24	2.80
Trace:	321.8937					
21:11	1.197E+02	6.52	3.44	18	6.29	3.36
Trace:	331.9368					
21:09	1.432E+03	78.02	41.13	217	75.87	40.49
Trace:	333.9339					
21:09	1.835E+03	100.00	52.72	286	100.00	53.36

**** PLIST Processing complete ****

Analysis Name: DM00:[300,303]STD6.MIS;7 Window: 1

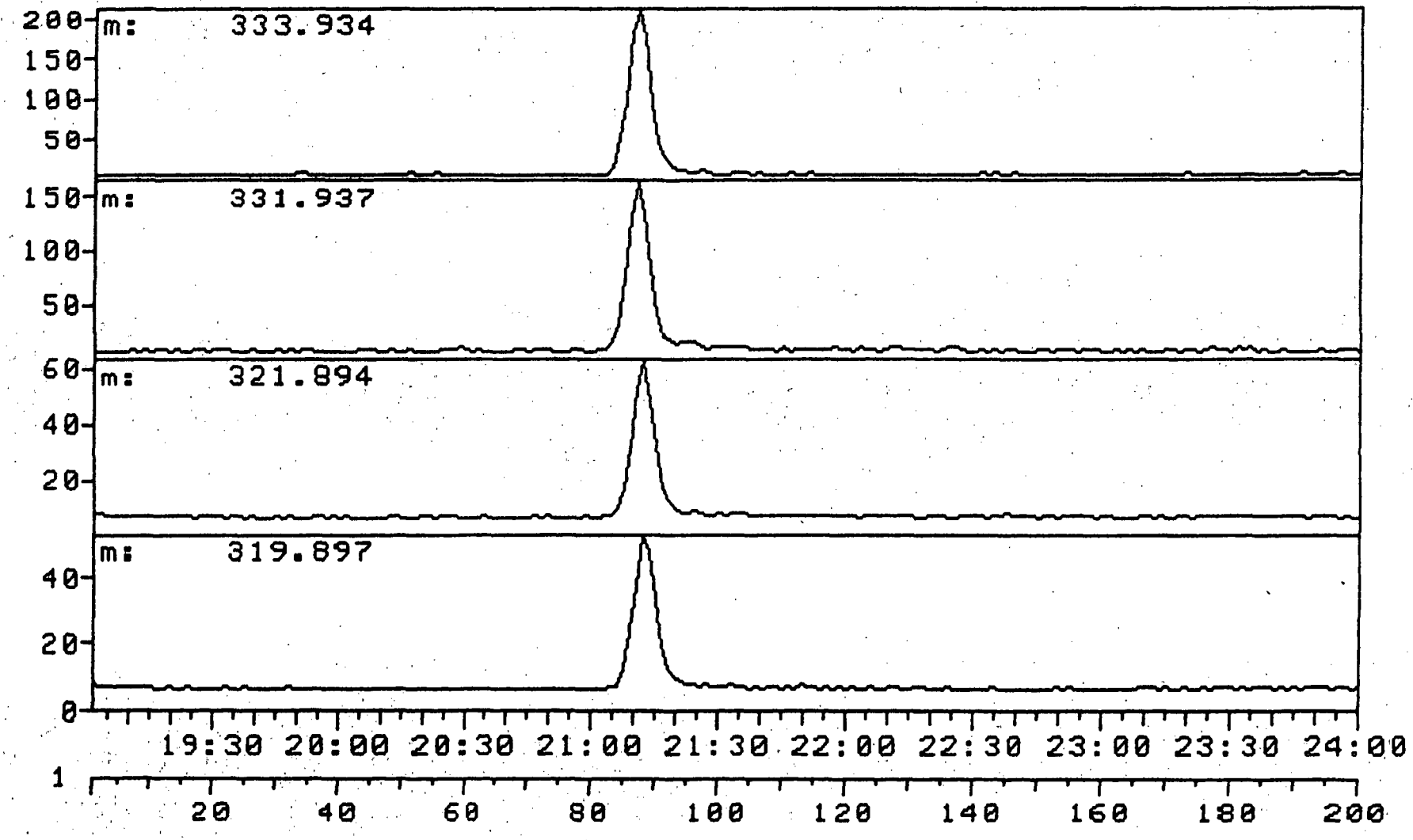
Operator: KC/KD Spc: 5

Sample ID: STD 2.6 Date: 12-OCT-89 15:00:33

Commission: R=7000 10-12-89

50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5UL

100HZ FILTER 8 CYCLE/GC PEAK



A-35

SSX: PLIST of DM00:[300,303]STD6.PEA;6
Finnigan MAT Oct 12 89 15:31:35 Page: 1

Calibration curve not well-defined for
Area , Height - use PPLOT to examine data
Sample Identification: STD 2.6
Filename: DM00:[300,303]STD6.MIS;7
Creation Date & Time: 12-OCT-89 15:00:33
Integrated Area: 2.925E+03 Integrated Height: 460
Maximum Area: 1.316E+03 Maximum Height: 204
Comment: 50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5U
L

Time	AREA			HEIGHT		
Centroid	Abs	Base	Sum	Abs	Base	Sum
Trace:	319.8965					
21:13	2.913E+02	22.13	9.96	47	23.04	10.22
Trace:	321.8937					
21:12	3.390E+02	25.75	11.59	56	27.45	12.17
Trace:	331.9368					
21:11	9.786E+02	74.34	33.45	153	75.00	33.26
Trace:	333.9339					
21:11	1.316E+03	100.00	45.00	204	100.00	44.35

**** PLIST Processing complete ****

Analysis Name: DM00:[300,303]STD7.MIS;5 Window: 1

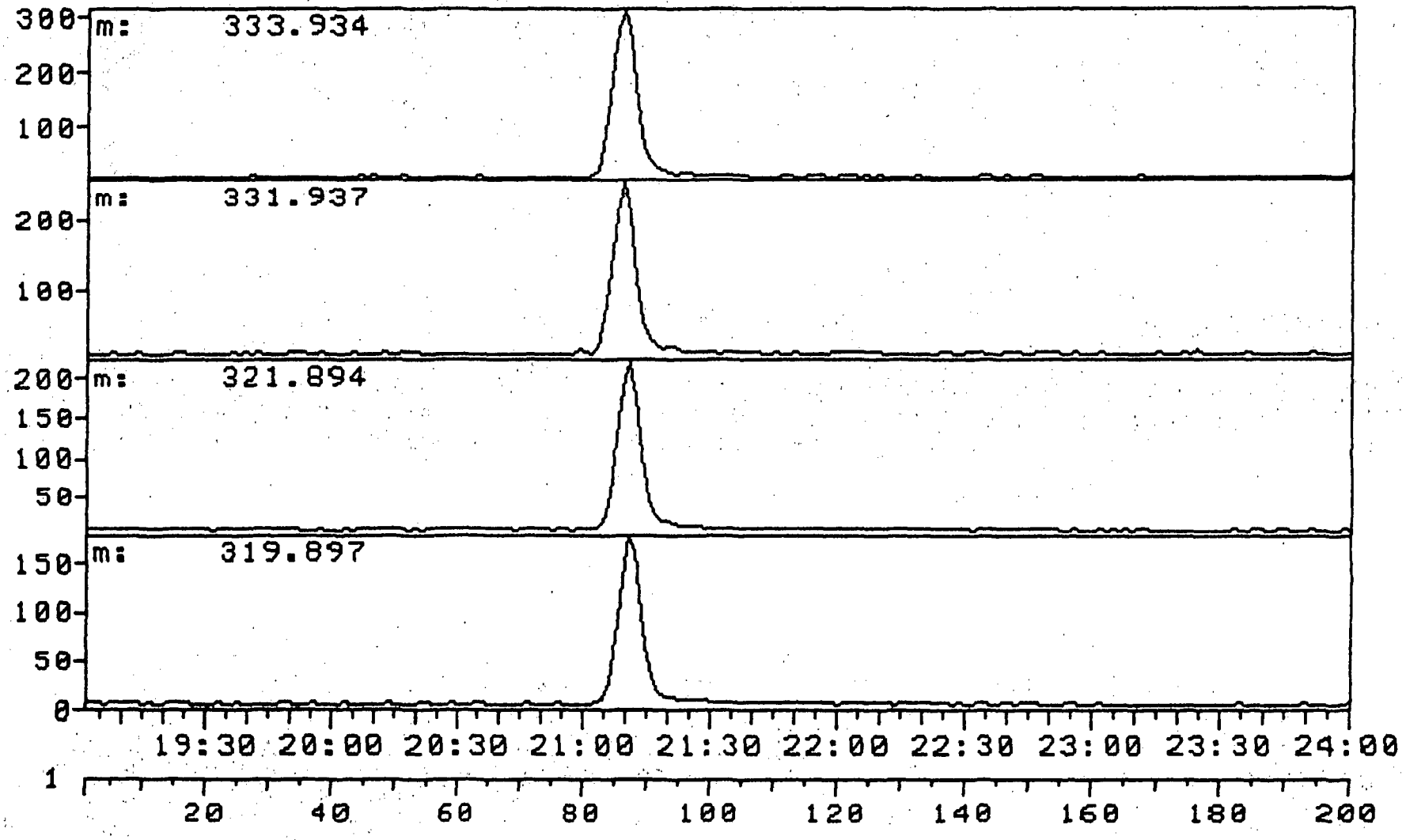
Operator: KC/KD Spc: 5

Sample ID: STD 2.7 Date: 12-OCT-89 15:28:43

Commission: R=7000 10-12-89

50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5UL

100HZ FILTER 8 CYCLE/GC PEAK



A-37

Calibration curve not well-defined for
Area , Height - use PPLOT to examine data
Sample Identification: STD 2.7
Filename: DM00:[300,303]STD7.MIS;5
Creation Date & Time: 12-OCT-89 15:28:43
Integrated Area: 5.990E+03 Integrated Height: 931
Maximum Area: 2.013E+03 Maximum Height: 303
Comment: 50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5U
L

Time	AREA			HEIGHT		
Centroid	Abs	Base	Sum	Abs	Base	Sum
Trace: 319.8965						
21:11	1.070E+03	53.15	17.87	171	56.44	18.37
Trace: 321.8937						
21:11	1.370E+03	68.04	22.87	213	70.30	22.88
Trace: 331.9368						
21:09	1.537E+03	76.33	25.66	244	80.53	26.21
Trace: 333.9339						
21:09	2.013E+03	100.00	33.61	303	100.00	32.55

**** PLIST Processing complete ****

Analysis Name: DM00:[300,303]MDC6.MIS;3 Window: 1

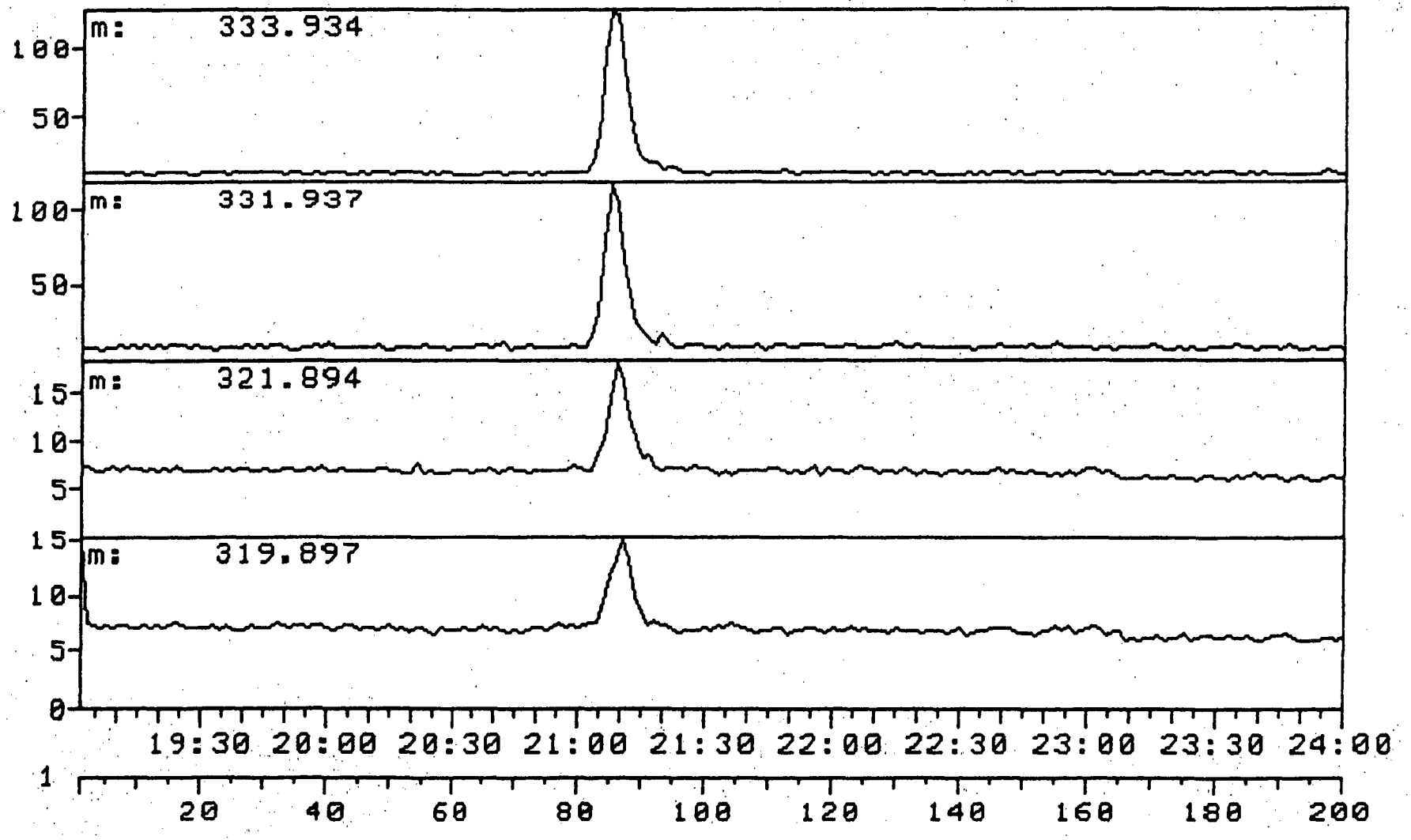
Operator: KC/KD Spc: 5

Sample ID: FISH MDC6 Date: 12-OCT-89 17:01:20

Commission: R=7000 10-12-89

50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5UL

100HZ FILTER 8 CYCLE/GC PEAK



A-40

Calibration curve not well-defined for
Area , Height - use PPLOT to examine data
Sample Identification: FISH MDC6
Filename: DM00:[300,303]MDC6.MIS;3
Creation Date & Time: 12-OCT-89 17:01:20
Integrated Area: 1.555E+03 Integrated Height: 247
Maximum Area: 7.890E+02 Maximum Height: 119
Comment: 50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5U
L

Time Centroid	AREA Abs	AREA Base	AREA Sum	HEIGHT Abs	HEIGHT Base	HEIGHT Sum
Trace: 319.8965						
21:09	4.382E+01	5.55	2.82	8	6.72	3.24
Trace: 321.8937						
21:09	6.331E+01	8.02	4.07	11	9.24	4.45
Trace: 331.9368						
21:09	6.585E+02	83.46	42.36	109	91.60	44.13
Trace: 333.9339						
21:09	7.890E+02	100.00	50.75	119	100.00	48.18

**** PLIST Processing complete ****

Analysis Name: DM00:[300,303]MDC10.MIS;5 Window: 1

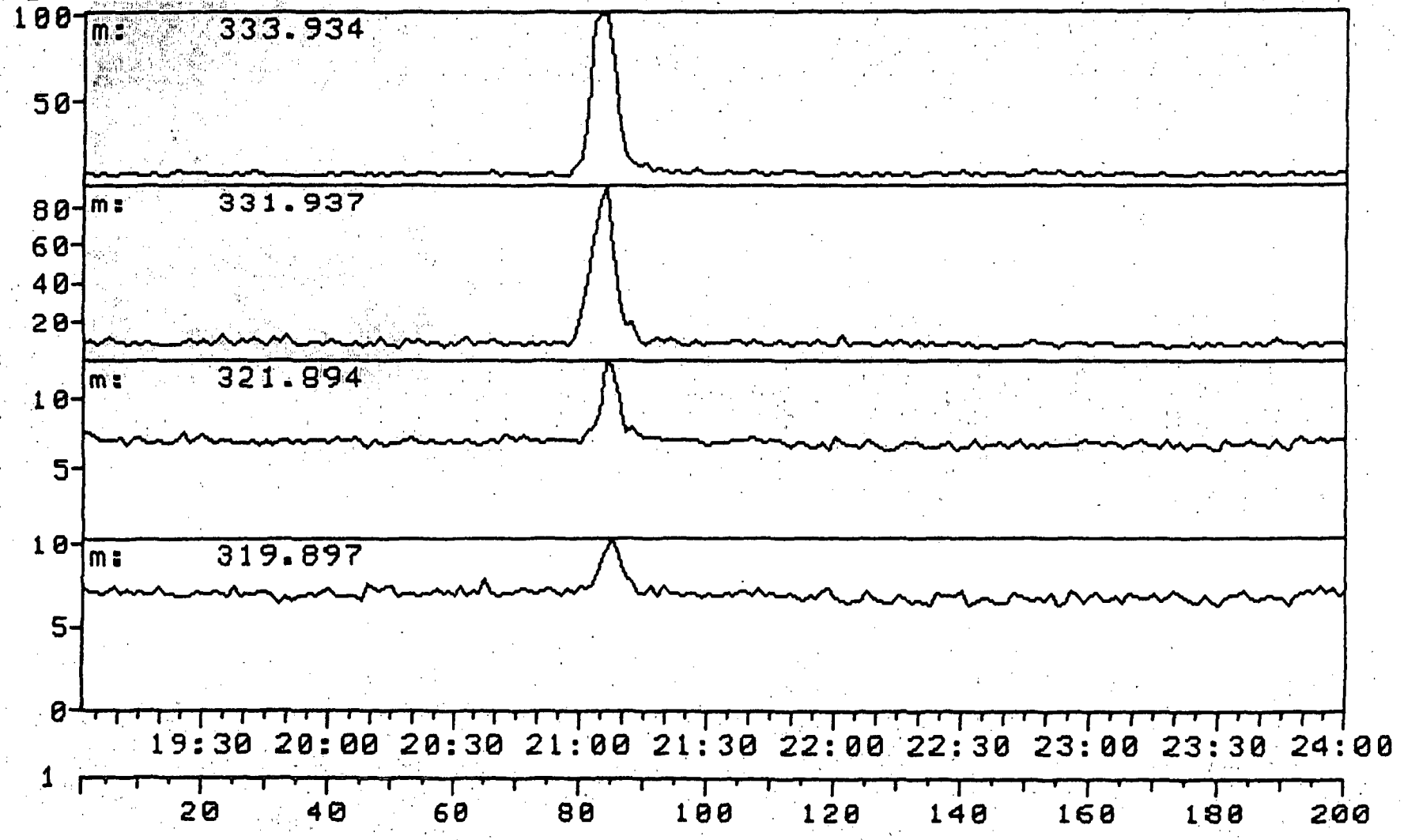
Operator: KC/KD Spc: 5

Sample ID: FISH MDC10 Date: 12-OCT-89 18:02:04

Commission: R=7000 10-12-89

50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5UL

100HZ FILTER 8 CYCLE/GC PEAK



A-41

A-42

```
        SS    PP          LL                ;;;;          11
        SS    PP          LL                ;;;;          11
        SS    PP          LL                  ;;          11
        SS    PP          LL                  ;;          11
SSSSSSSS      PP          LLLLLLLLLL        ;;          111111
SSSSSSSS      PP          LLLLLLLLLL        ;;          111111
```

```
01234567890123456789    ** RSX-11M V4.0 **    23-OCT-89    17:20:37    DMO:[
01234567890123456789    ** RSX-11M V4.0 **    23-OCT-89    17:20:37    DMO:[
01234567890123456789    ** RSX-11M V4.0 **    23-OCT-89    17:20:37    DMO:[
```

SSX: PLIST of DM00:[300,303]MDC10.PEA;5
Finnigan MAT Oct 23 89 17:20:36 Page: 1

Calibration curve not well-defined for
Area , Height - use PPLOT to examine data
Sample Identification: FISH MDC10
Filename: DM00:[300,303]MDC10.MIS;5
Creation Date & Time: 12-OCT-89 18:02:04
Integrated Area: 1.258E+03 Integrated Height: 184
Maximum Area: 6.875E+02 Maximum Height: 94
Comment: 50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5U
L

Time Centroid	AREA Abs	AREA Base	AREA Sum	HEIGHT Abs	HEIGHT Base	HEIGHT Sum
Trace:	319.8965					
21:06	1.385E+01	2.02	1.10	3	3.19	1.63
Trace:	321.8937					
21:06	1.786E+01	2.60	1.42	5	5.32	2.72
Trace:	331.9368					
21:05	5.391E+02	78.41	42.84	82	87.23	44.57
Trace:	333.9339					
21:05	6.875E+02	100.00	54.64	94	100.00	51.09

**** PLIST Processing complete ****

Analysis Name: DM00:[300,303]MDC14.MIS;5 Window: 1

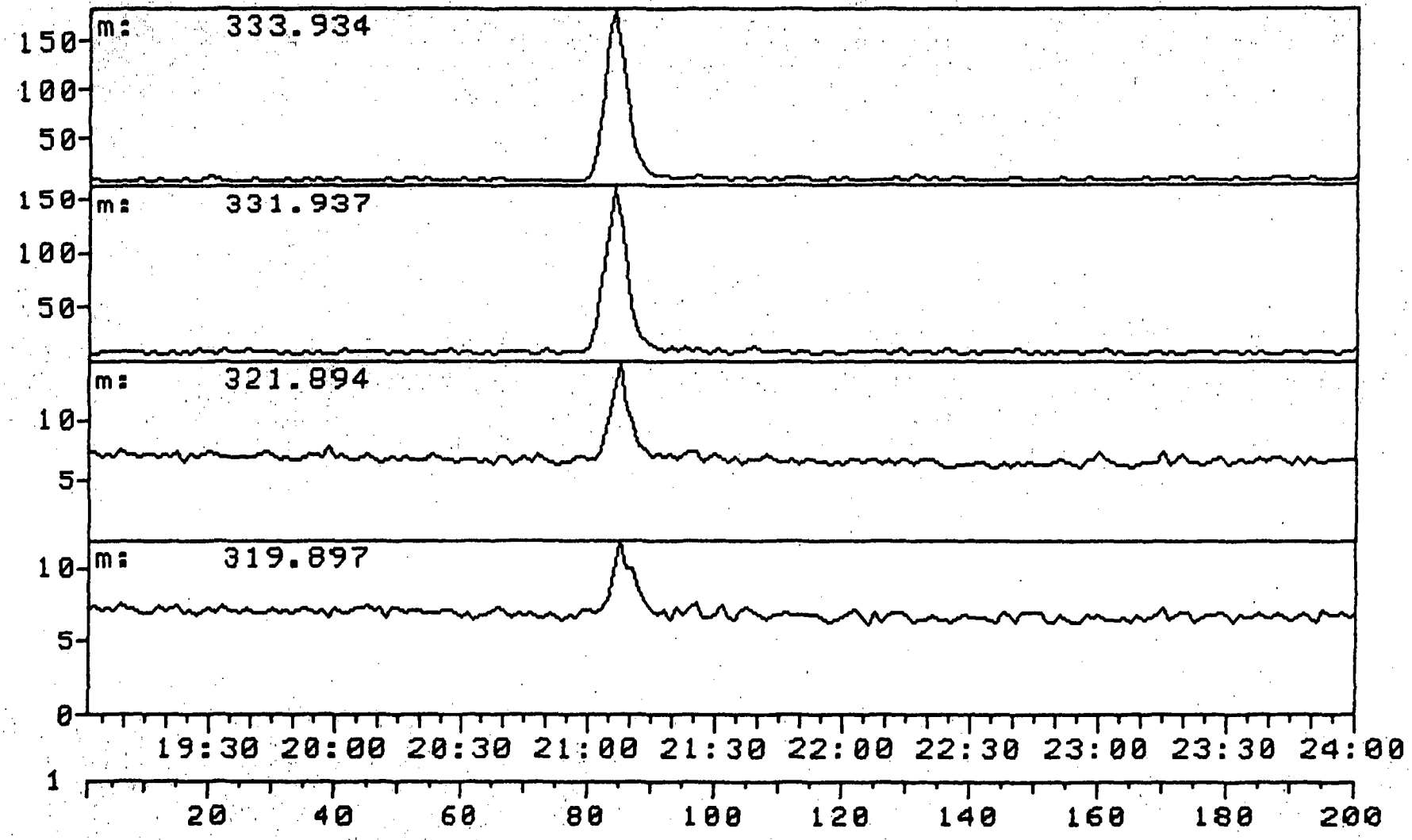
Operator: KC/KD Spc: 5

Sample ID: FISH MDC14 Date: 12-OCT-89 17:32:17

Commission: R=7000 10-12-89

50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5UL

100HZ FILTER 8 CYCLE/GC PEAK



SSX: PLIST of DM00:[300,303]MDC14.PEA;4
Finnigan MAT Oct 23 89 17:16:31 Page: 1

Calibration curve not well-defined for
Area , Height - use PPLOT to examine data
Sample Identification: FISH MDC14
Filename: DM00:[300,303]MDC14.MIS;5
Creation Date & Time: 12-OCT-89 17:32:17
Integrated Area: 1.988E+03 Integrated Height: 334
Maximum Area: 1.054E+03 Maximum Height: 173
Comment: 50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5U
L

Time Centroid	AREA Abs	AREA Base	AREA Sum	HEIGHT Abs	HEIGHT Base	HEIGHT Sum
Trace:	319.8965					
21:09	2.714E+01	2.58	1.37	5	2.89	1.50
Trace:	321.8937					
21:08	3.489E+01	3.31	1.76	8	4.62	2.40
Trace:	331.9368					
21:06	8.721E+02	82.78	43.88	148	85.55	44.31
Trace:	333.9339					
21:06	1.054E+03	100.00	53.00	173	100.00	51.80

**** PLIST Processing complete ****

Date 10/13/89

MAT 8230 C-GC-MS CONDITIONS

Column HP-5 50m x 0.2mm 0.33μm film

Injector 250°C splitless 0.7 min delay 30psi He

GC Oven Program 160°C (2min) $\xrightarrow{25°C/min}$ 270°C (20min)

Separator 280°C (688)

Line of Sight 290°C (678)

Ion Source EI/CI 220°C 1mA 70eV

Ua/Ub 830

Y1 211

Y2 86

X1 583

X2 445

L1 429

L2 540

S1 1

Z1 539

Z2 548

P 542

Filter 100Hz

Multiplier 1.4 KV (150)

MSCHAR 200:10:40:25:6:131000 (315.4 magnet rest mass)

HR Slits 599 566 Resolution 7100

LR Slits 578 800 Resolution 1000

HR : Ion 331 Resp.(V) 0.5 LR : Ion 331 Resp.(V) 4.5

LR/HR Resp. 10

txt01:hrgcms.txt

SSX: MASSMENU TCDD7.MMN;2
Finnigan MAT Oct 13 89 10:00:02 Page: 4

Beginning MASS MENU creation

Magnet settle time 200 msec
Magnet jump time 1000 usec
Magnet focus time 40 msec
EDAC jump time 25 msec
EDAC focus time 6 msec
EDAC capabilities: rated = 131000, calibrated = 661895
used = 661895

Window number 1

from 12 to 3030

MASS	MDAC	EDAC	DELDAC	NSTEP	NSUM	CYCTIME
47671	47671	120633	100012	24	0	2734
47771	0	173751	0	0	20	2734
50171	0	163753	0	0	20	2734
51277	0	120633	0	0	1	2734
51374	0	115154	0	0	4	2734
51574	0	105621	0	0	4	2734

***** MASS MENU Processing complete *****

SSX: SLIST of DM00:[300,303]PFK.DAT;2
Finnigan MAT Oct 13 89 11:26:10 Page: 1

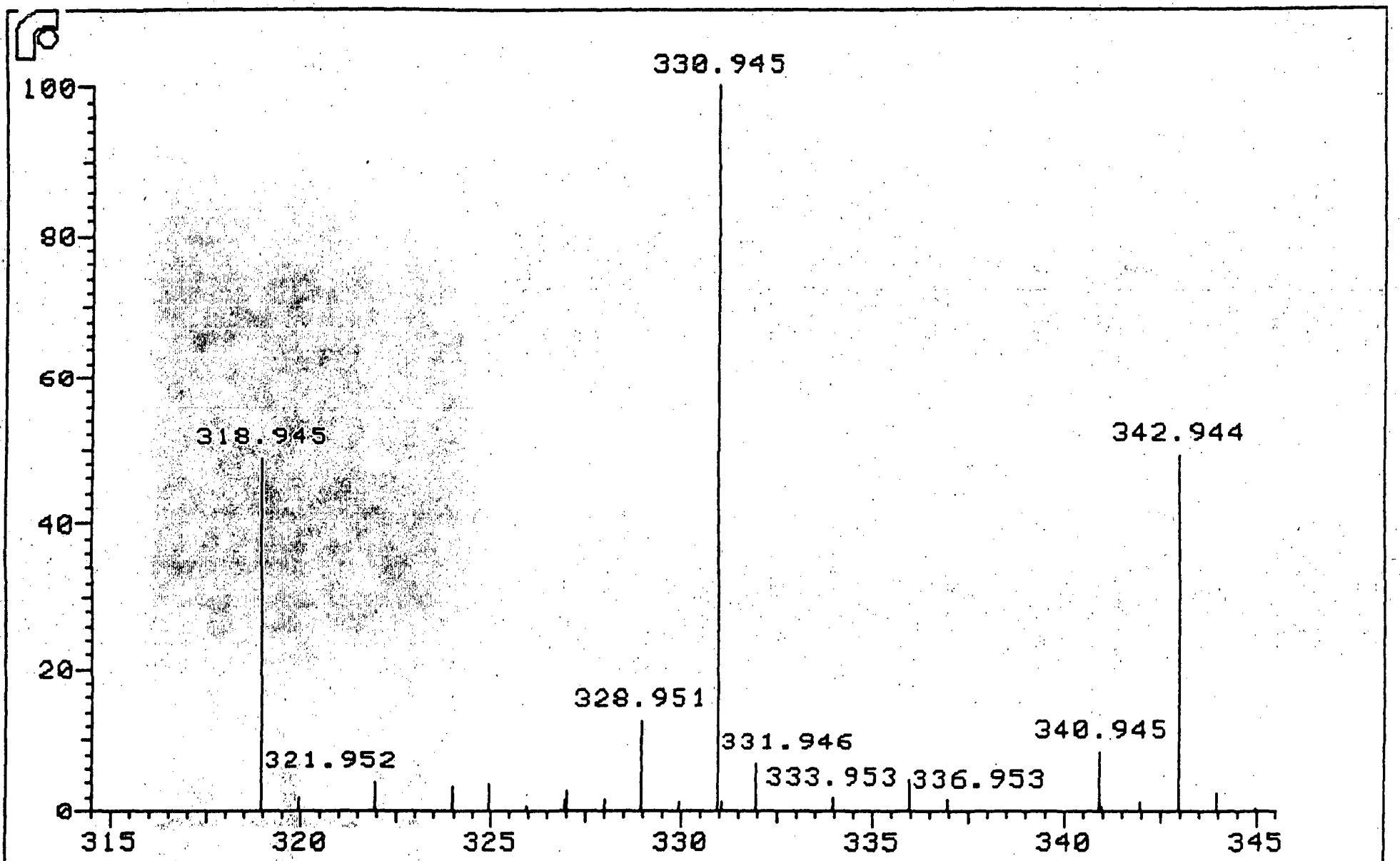
A-47

Spectrum Number: 3
Number of Peaks: 322

Norm. Factors: 1. 2256.32

PEAK#	MASS	A	B
10	50.984	6384.	2.83
15	69.016	225632.	100.00
19	73.078	4800.	2.13
34	92.984	4400.	1.95
41	100.031	11152.	4.94
49	113.031	3328.	1.47
52	118.984	51952.	23.03
56	131.016	49680.	22.02
64	143.016	4208.	1.86
69	150.000	2304.	1.02
70	151.016	4720.	2.09
71	154.984	2608.	1.16
75	161.984	5792.	2.57
76	163.016	2320.	1.03
81	169.000	39936.	17.70
88	181.000	37376.	16.57
93	192.969	7664.	3.40
104	204.984	5488.	2.43
107	207.047	8016.	3.55
116	218.969	20656.	9.15
124	231.000	20368.	9.03
132	242.984	9760.	4.33
140	253.000	3824.	1.69
142	254.953	4352.	1.93
149	267.016	2592.	1.15
151	269.016	9536.	4.23
159	280.969	14080.	6.24
160	281.047	3600.	1.60
168	293.000	8688.	3.85
174	305.000	3072.	1.36
181	318.953	4560.	2.02
191	330.969	10256.	4.55
199	342.984	5632.	2.50
207	355.000	2912.	1.29
221	380.969	7456.	3.30
228	392.953	4320.	1.91
234	404.938	3184.	1.41
254	430.969	5600.	2.48
258	442.953	4464.	1.98
262	454.969	2736.	1.21
272	480.984	3360.	1.49
275	492.969	2672.	1.18
294	542.922	2608.	1.16

***** SLIST processing complete. *****



Analysis Name: PFK.DAT;6 Spec# 2 Norm: B /Scale: 12416
Date: OCT 13 89 10:27:13 Nmparam: 0.5:0.5 Tolerance: 500:MMU

A-48

A-49

MASS CORRESPONDENCE:

SAMPLE PEAK NUMBER	SAMPLE MASS	REFERENCE MASS
1	318.9526	318.9793
24	343.2317	342.9793

EDAC CONTROL OF MASS RANGE = 1.1099

X-ACT VALUE = 661835

**** ECAL PROCESSING COMPLETE ****

TCDD Report Program HRTCDD.COM
2,3,7,8-TCDD by C-GC/HRMS-SIM
Revised - February 11, 1988

AER Log No.	Sample I.D.	Date Sampled	Date Extrcted	Sample Wt.(g)	Result TCDD(ppt)	320/322	332/334	Notes
	CONTROL		10-5-89	50.0	ND(1.2)	--	0.85	1
	MDC-16	8-22-89	10-10-89	53.6	9.9	0.76	0.75	
	MDC-15S	8-22-89	10-12-89	45.2	2.1	0.73	0.71	2
	MDC-15	8-22-89	10-9-89	52.5	1.3	0.72	0.86	
	MDC-12	8-22-89	10-11-89	50.4	9.8	0.81	0.76	
	MDC-11	8-22-89	10-9-89	51.2	1.5	0.86	0.77	
	MDC-8	8-22-89	10-11-89	50.1	20.7	0.78	0.81	
	MDC-7	8-22-89	10-9-89	51.3	4.1	0.83	0.82	
	MDC-5	8-22-89	10-11-89	52.4	20.1	0.79	0.83	
	MDC-3	8-22-89	10-12-89	46.4	20.0	0.77	0.74	3
	MDC-3	8-22-89	10-11-89	49.9	19.3	0.71	0.79	3
	MDC-2	8-22-89	10-9-89	50.0	3.3	0.75	0.72	
	MDC-1	8-22-89	10-10-89	51.3	4.7	0.71	0.70	

1. None detected. Detection limit calculated from 2.5 times noise level.
2. 0.0336ng native TCDD spiked to 45.2g MDC-15 (equivalent to 0.7 ppt spike).
3. Duplicate sample preparation and analysis.

ppt - parts per trillion
ND - None Detected (detection limit)

File : 00126 Created : October 12, 1989 17:15
Printed : October 25, 1989 18:19

Raw Data for Sample Entries

A-51

AER Log No.	13C12-TCDD Spike(ng)	Data Type	ion 257	ion 319.897	ion 321.894	ion 331.937	ion 333.934	(320+322) / (332+334)
	1.874	Height		6.0-N	3.0-N	412	485	0.02508
	1.874	Area		45.50	59.49	204.9	271.7	0.22029
	1.874	Area		29.44	40.59	744.7	1049.0	0.03904
	1.874	Area		9.37	13.09	380.8	444.0	0.02723
	1.874	Area		90.76	111.4	428.2	561.5	0.20426
	1.874	Area		4.454	5.166	135.2	176.2	0.03089
	1.874	Area		79.48	102.3	189.5	232.8	0.43045
	1.874	Area		47.55	57.51	544.1	665.0	0.08689
	1.874	Area		161.4	205.3	381.1	460.1	0.43592
	1.874	Area		152.7	199.4	388.0	526.0	0.38523
	1.874	Area		151.3	214.4	403.9	510.3	0.40002
	1.874	Area		6.182	8.190	87.82	121.8	0.06856
	1.874	Area		35.01	49.06	348.4	494.9	0.09969

N - Noise Level
I - Interfering Peak Level

Raw Data for Standard Entries

TCDD (ng) Native	13C12	Data Type	ion 257	ion 319.897	ion 321.894	ion 331.937	ion 333.934	(320+322) / (332+334)
0.01658	1.874	Area		11.14	14.48	1868	2474	0.00590
		Height		2	2	294	377	0.00596
0.03316	1.874	Area		77.52	96.90	5443	6940	0.01409
		Height		12	13	778	1012	0.01397
0.06632	1.874	Area		141.5	177.4	4914	6168	0.02878
		Height		24	27	747	985	0.02945
0.06632	1.874	Area		51.75	92.90	2389	2785	0.02796
		Height		10	14	339	407	0.03217
0.1658	1.874	Area		94.37	119.7	1432	1835	0.06552
		Height		15	18	217	286	0.06561
0.6632	1.874	Area		291.3	339.0	978.6	1316	0.27469
		Height		47	56	153	204	0.28852
1.658	1.874	Area		1070	1370	1537	2013	0.68732
		Height		171	213	244	303	0.70201

File : 00126

Created : October 12, 1989 17:15
Printed : October 25, 1989 18:19

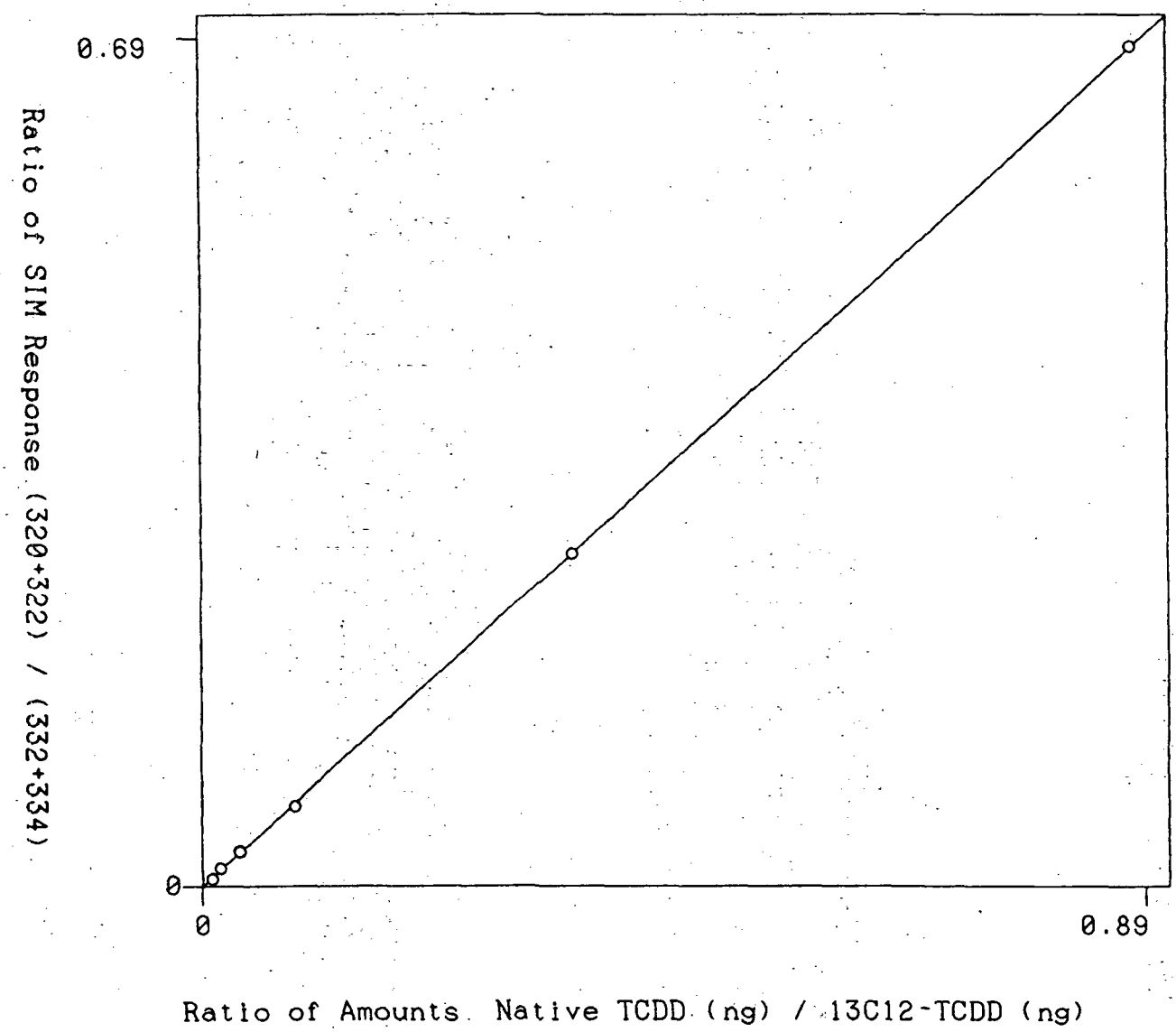
X = Ratio of Amounts Native TCDD (ng) / 13C12-TCDD (ng)
Y = Ratio of SIM Areas (320 + 322) / (332 + 334)

A-52

X	Y	Y (Reg.)	% Rel. Diff (Y)
0.00885	0.00590	0.00649	-9.49
0.01769	0.01409	0.01336	5.26
0.03539	0.02878	0.02712	5.94
0.03539	0.02796	0.02712	3.06
0.08847	0.06552	0.06837	-4.25
0.35390	0.27469	0.27464	0.02
0.88474	0.68732	0.68717	0.02

Y = -0.00039 + 0.77713 (X)

Correlation Coefficient : 0.9999836
Standard Error of Estimate : 0.0015778



File : 00126 Created : October 12, 1989 17:15
Printed : October 25, 1989 18:19

Analysis Name: DM00:[300,303]TCDD7.MIS;35 Window: 1

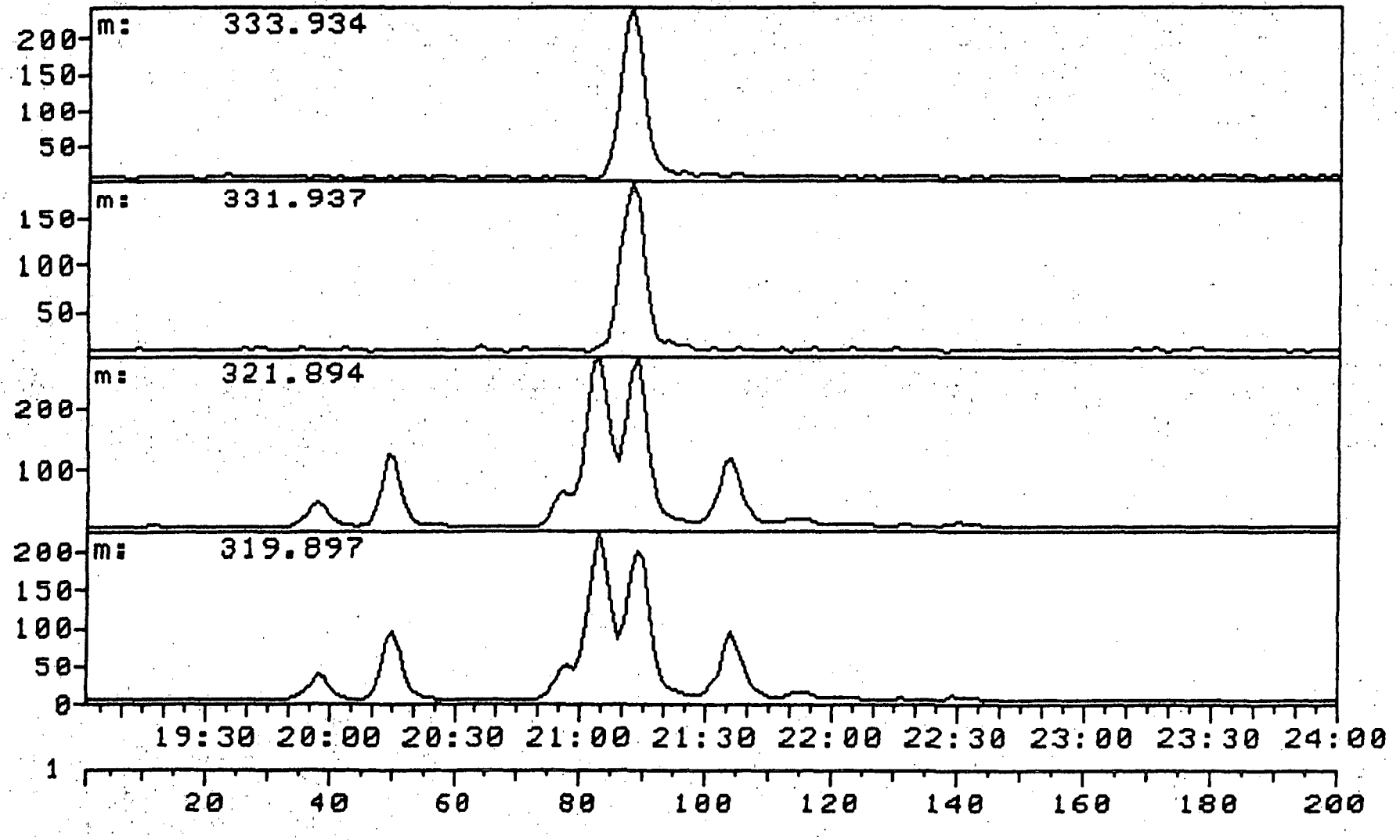
Operator: KC/KD Spc: 5

Sample ID: 7 ISO MIX Date: 13-OCT-89 11:28:29

Commission: R=7000 10-13-89

50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 1UL

100HZ FILTER 8 CYCLE/GC PEAK



Analysis Name: DM00:[300,303]STD3.MIS;15 Window: 1

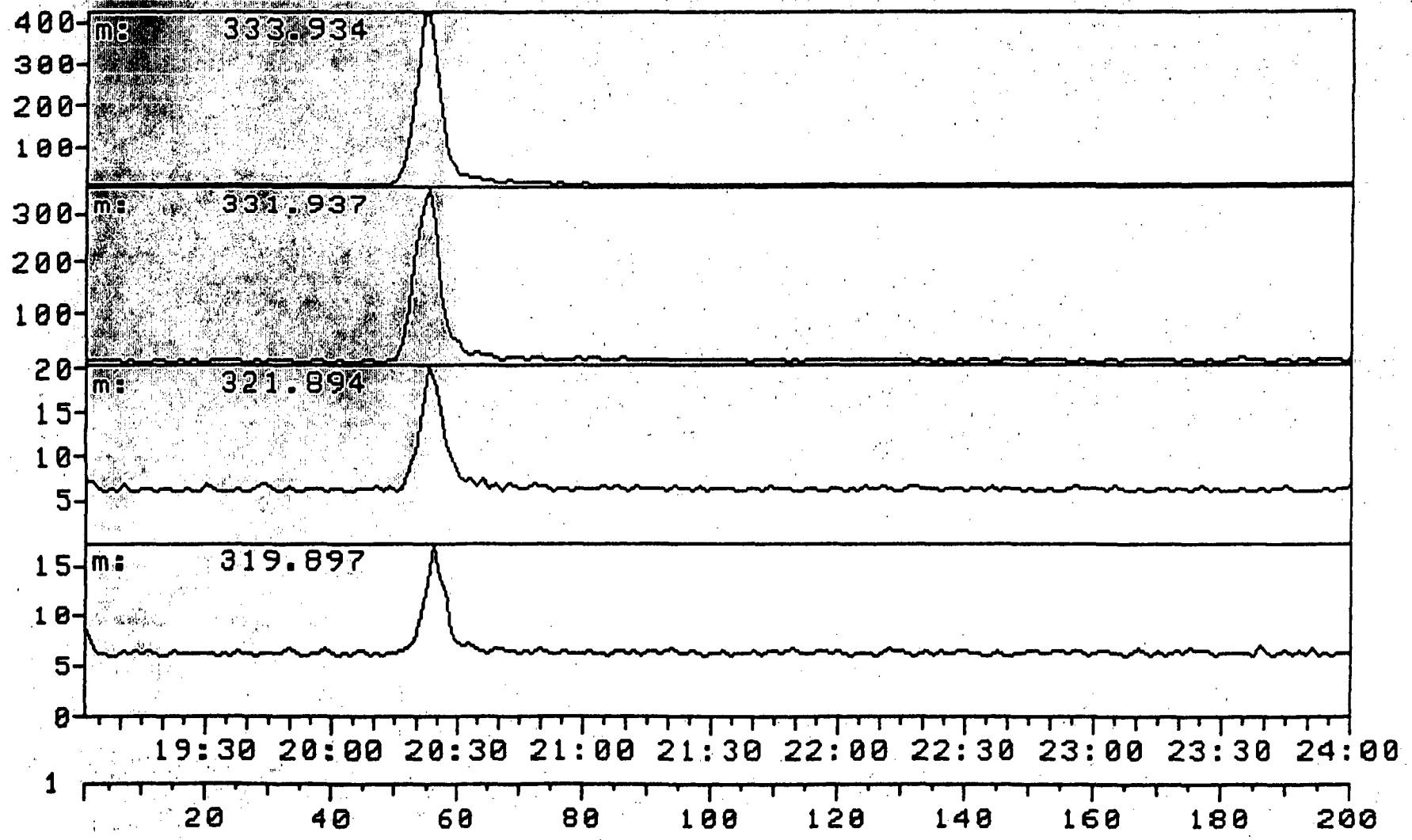
Operator: KC/KD Spc: 5

Sample ID: STD 2.3 Date: 13-OCT-89 13:46:01

Commission: R=7000 10-13-89

50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5UL

100HZ FILTER 8 CYCLE/GC PEAK



A-54

SSX: PLIST of DM00:[300,303]STD3.PEA;15
Finnigan MAT Oct 13 89 14:29:45 Page: 1

Calibration curve not well-defined for
Area , Height - use PPLOT to examine data
Sample Identification: STD 2.3
Filename: DM00:[300,303]STD3.MIS;15
Creation Date & Time: 13-OCT-89 13:46:01
Integrated Area: 5.319E+03 Integrated Height: 769
Maximum Area: 2.785E+03 Maximum Height: 406
Comment: 50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5U
L

Time	AREA			HEIGHT		
Centroid	Abs	Base	Sum	Abs	Base	Sum
Trace:	319.8965					
20:24	5.175E+01	1.86	0.97	10	2.46	1.30
Trace:	321.8937					
20:24	9.290E+01	3.34	1.75	14	3.45	1.82
Trace:	331.9368					
20:23	2.389E+03	85.79	44.92	339	83.50	44.08
Trace:	333.9339					
20:22	2.785E+03	100.00	52.36	406	100.00	52.80

**** PLIST Processing complete ****

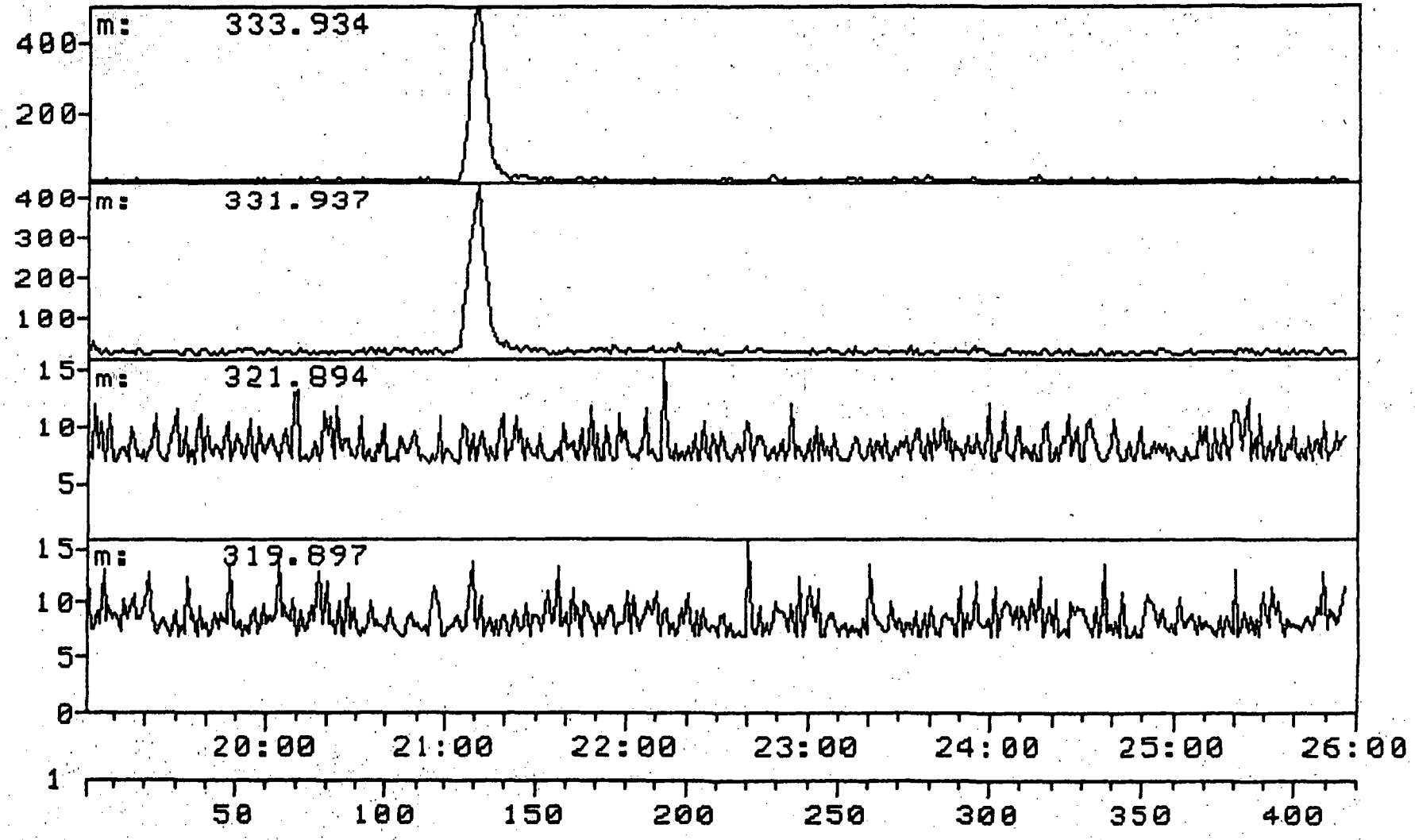
Analysis Name: DM00:[300,303]CONTROL1.MIS;1 Window: 1

Operator: KC/KD Spc: 5

Sample ID: CONTROL1 Date: 10-OCT-89 18:07:01

Commission: R=7000 10-10-89

50M HP-5 160(2) 25/M 270(20) SEM 1.6KV 5UL



A-52

A-57

SSX: PLIST of DM00:[300,303]CONTROL1.PEA;2
Finnigan MAT Oct 25 89 18:02:27 Page: 1

Calibration curve not well-defined for
Area , Height - use PPLOT to examine data
Sample Identification: CONTROL1
Filename: DM00:[300,303]CONTROL1.MIS;1
Creation Date & Time: 10-OCT-89 18:07:01
Integrated Area: 5.861E+03 Integrated Height: 906
Maximum Area: 3.223E+03 Maximum Height: 485
Comment: 50M HP-5 160(2) 25/M 270(20) SEM 1.6KV 5U
L

Time	AREA			HEIGHT		
Centroid	Abs	Base	Sum	Abs	Base	Sum
Trace:	319.8965					
21:05	2.363E+01	0.73	0.40	6	1.24	0.66
Trace:	321.8937					
21:24	1.511E+01	0.47	0.26	3	0.62	0.33
Trace:	331.9368					
21:11	2.599E+03	80.63	44.34	412	84.95	45.47
Trace:	333.9339					
21:12	3.223E+03	100.00	55.00	485	100.00	53.53

**** PLIST Processing complete ****

Analysis Name: DM00:[300,303]MDC16.MIS;1 Window: 1

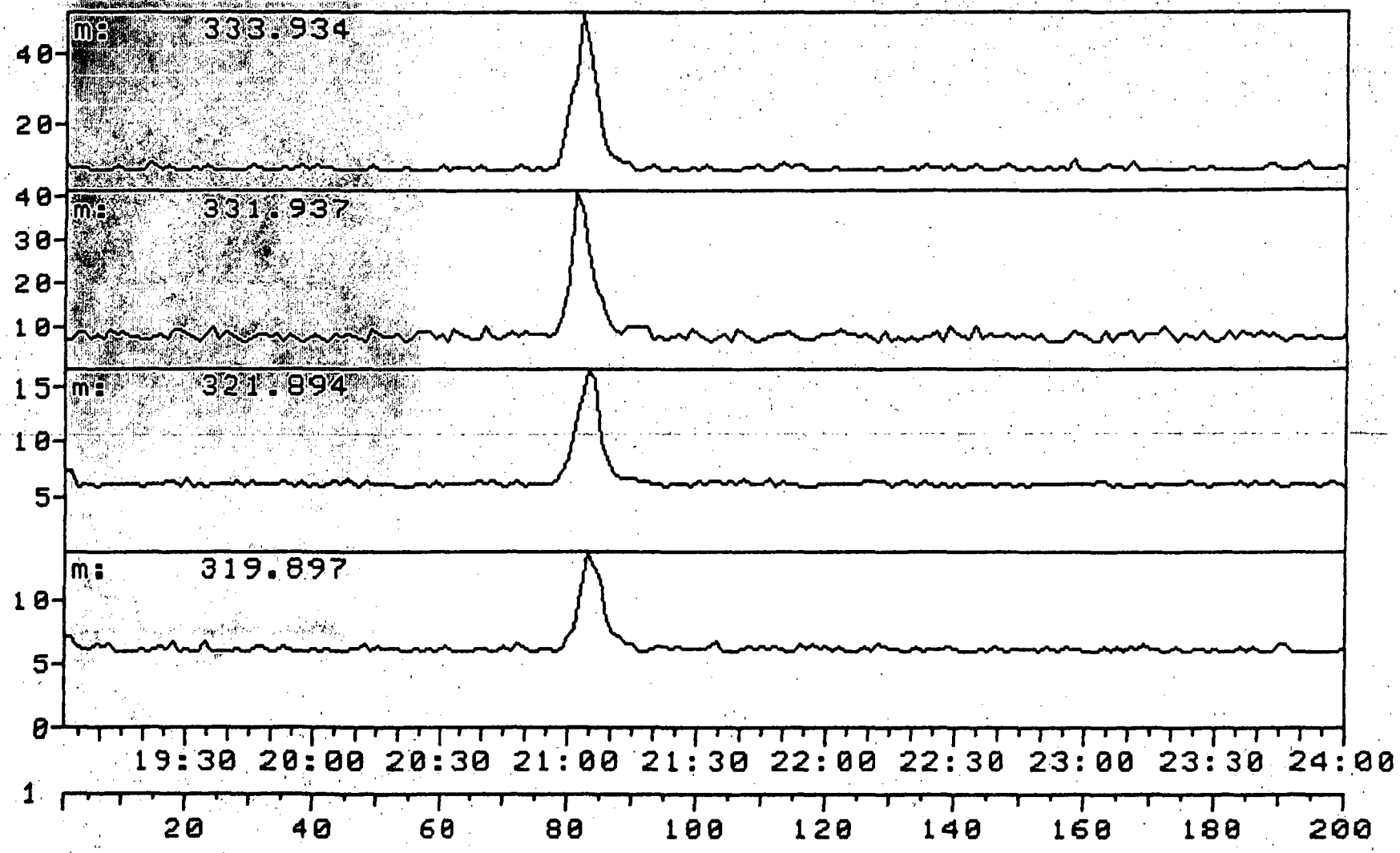
Operator: KC/KD Spc: 5

Sample ID: FISH MDC16 Date: 13-OCT-89 16:03:24

Commission: R=7000 10-13-89

50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5UL

100HZ FILTER 8 CYCLE/GC PEAK



A-59

Calibration curve not well-defined for
Area , Height - use PPLOT to examine data
Sample Identification: FISH MDC16
Filename: DM00:[300,303]MDC16.MIS;1
Creation Date & Time: 13-OCT-89 16:03:24
Integrated Area: 5.817E+02 Integrated Height: 96
Maximum Area: 2.717E+02 Maximum Height: 45
Comment: 50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5U
L

Time	AREA			HEIGHT		
Centroid	Abs	Base	Sum	Abs	Base	Sum
Trace:	319.8965					
21:05	4.550E+01	16.75	7.82	8	17.78	8.33
Trace:	321.8937					
21:04	5.949E+01	21.89	10.23	10	22.22	10.42
Trace:	331.9368					
21:04	2.049E+02	75.42	35.23	33	73.33	34.38
Trace:	333.9339					
21:03	2.717E+02	100.00	46.72	45	100.00	46.88

**** PLIST Processing complete ****

Analysis Name: DM00:[300,303]MDC15S.MIS;1 Window: 1

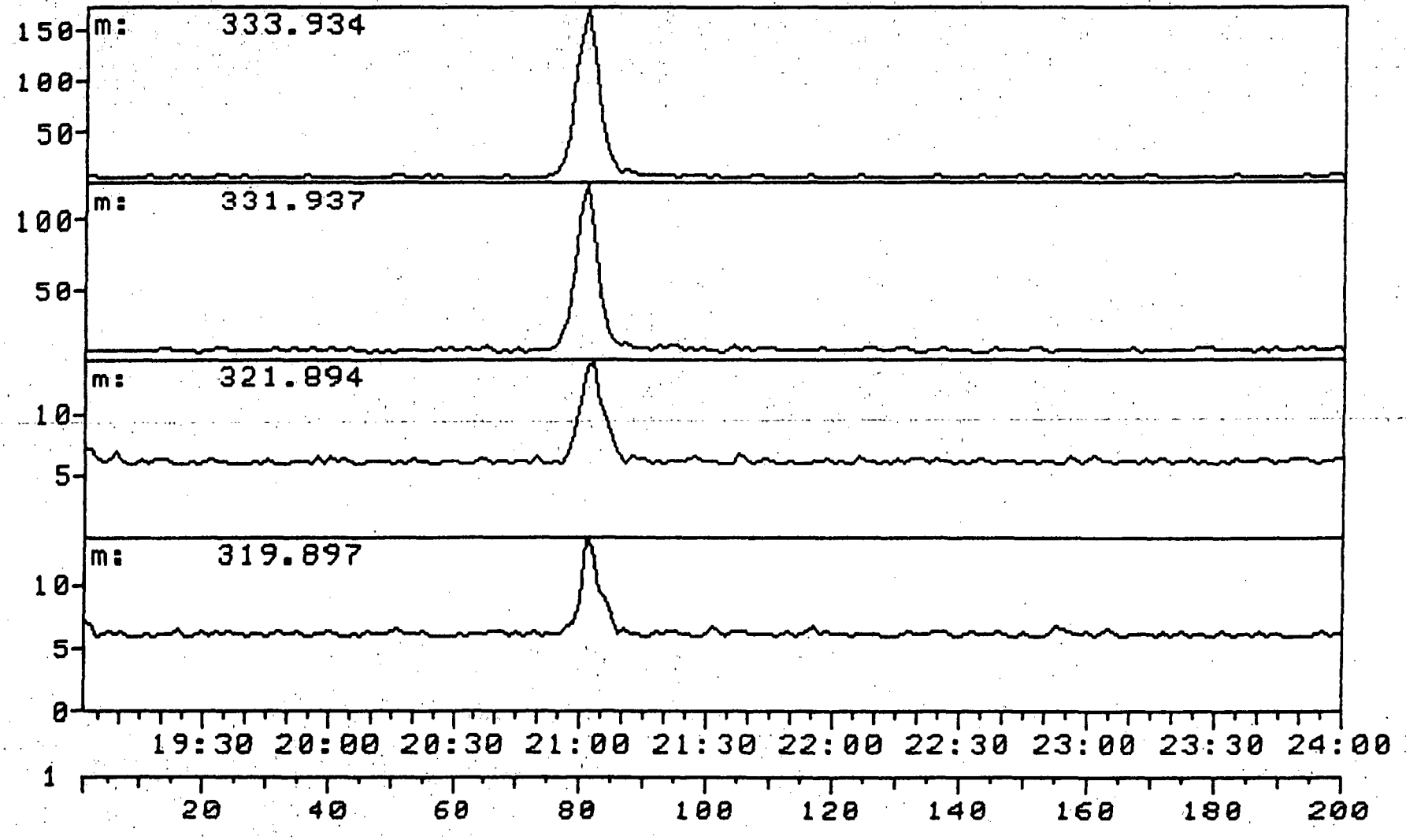
Operator: KC/KD Spc: 5

Sample ID: FISH MDC15 Date: 13-OCT-89 19:39:42

Commission: R=7000 10-13-89

50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5UL SPIKED W/ 0.0336NG TCDD

100HZ FILTER 8 CYCLE/GC PEAK



A-61

SSX: PLIST of DM00:[300,303]MDC15S.PEA;5
Finnigan MAT Oct 25 89 12:58:00 Page: 1

Calibration curve not well-defined for
Area , Height - use PPLOT to examine data
Sample Identification: FISH MDC15
Filename: DM00:[300,303]MDC15S.MIS;1
Creation Date & Time: 13-OCT-89 19:39:42
Integrated Area: 1.864E+03 Integrated Height: 296
Maximum Area: 1.049E+03 Maximum Height: 164
Comment: 50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5U
L SPIKED W/ 0.0336NG TCDD

Time Centroid	AREA Abs	AREA Base	AREA Sum	HEIGHT Abs	HEIGHT Base	HEIGHT Sum
Trace: 319.8965						
21:02	2.944E+01	2.81	1.58	7	4.27	2.36
Trace: 321.8937						
21:02	4.059E+01	3.87	2.18	8	4.88	2.70
Trace: 331.9368						
21:01	7.447E+02	70.96	39.95	117	71.34	39.53
Trace: 333.9339						
21:01	1.049E+03	100.00	56.30	164	100.00	55.41

**** PLIST Processing complete ****

Analysis Name: DM00:[300,303]MDC15.MIS;2 Window: 1

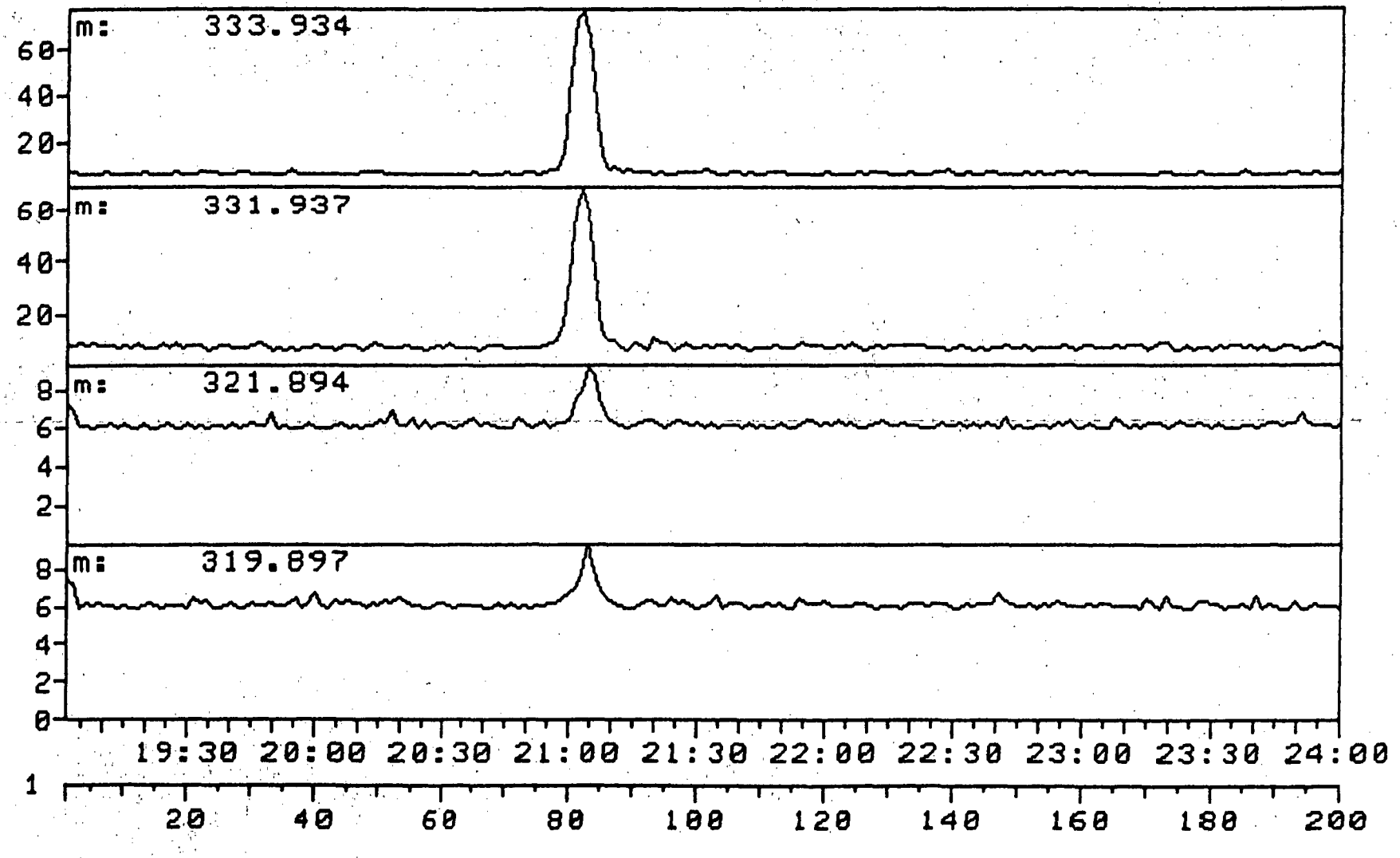
Operator: KC/KD Spc: 5

Sample ID: FISH MDC15 Date: 13-OCT-89 20:09:28

Commission: R=7000 10-13-89

50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5UL

100HZ FILTER 8 CYCLE/GC PEAK



A-62

SSX: PLIST of DM00:[300,303]MDC15.PEA;7
Finnigan MAT Oct 25 89 12:52:20 Page: 1

Calibration curve not well-defined for
Area , Height - use PPLOT to examine data
Sample Identification: FISH MDC15
Filename: DM00:[300,303]MDC15.MIS;2
Creation Date & Time: 13-OCT-89 20:09:28
Integrated Area: 8.473E+02 Integrated Height: 138
Maximum Area: 4.440E+02 Maximum Height: 71
Comment: 50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5U
L

Time Centroid	AREA Abs	AREA Base	AREA Sum	HEIGHT Abs	HEIGHT Base	HEIGHT Sum
Trace:	319.8965					
21:03	9.370E+00	2.11	1.11	3	4.23	2.17
Trace:	321.8937					
21:03	1.309E+01	2.95	1.54	3	4.23	2.17
Trace:	331.9368					
21:03	3.808E+02	85.77	44.95	61	85.92	44.20
Trace:	333.9339					
21:03	4.440E+02	100.00	52.40	71	100.00	51.45

**** PLIST Processing complete ****

Analysis Name: DM00:[300,303]MDC12.MIS;1 Window: 1

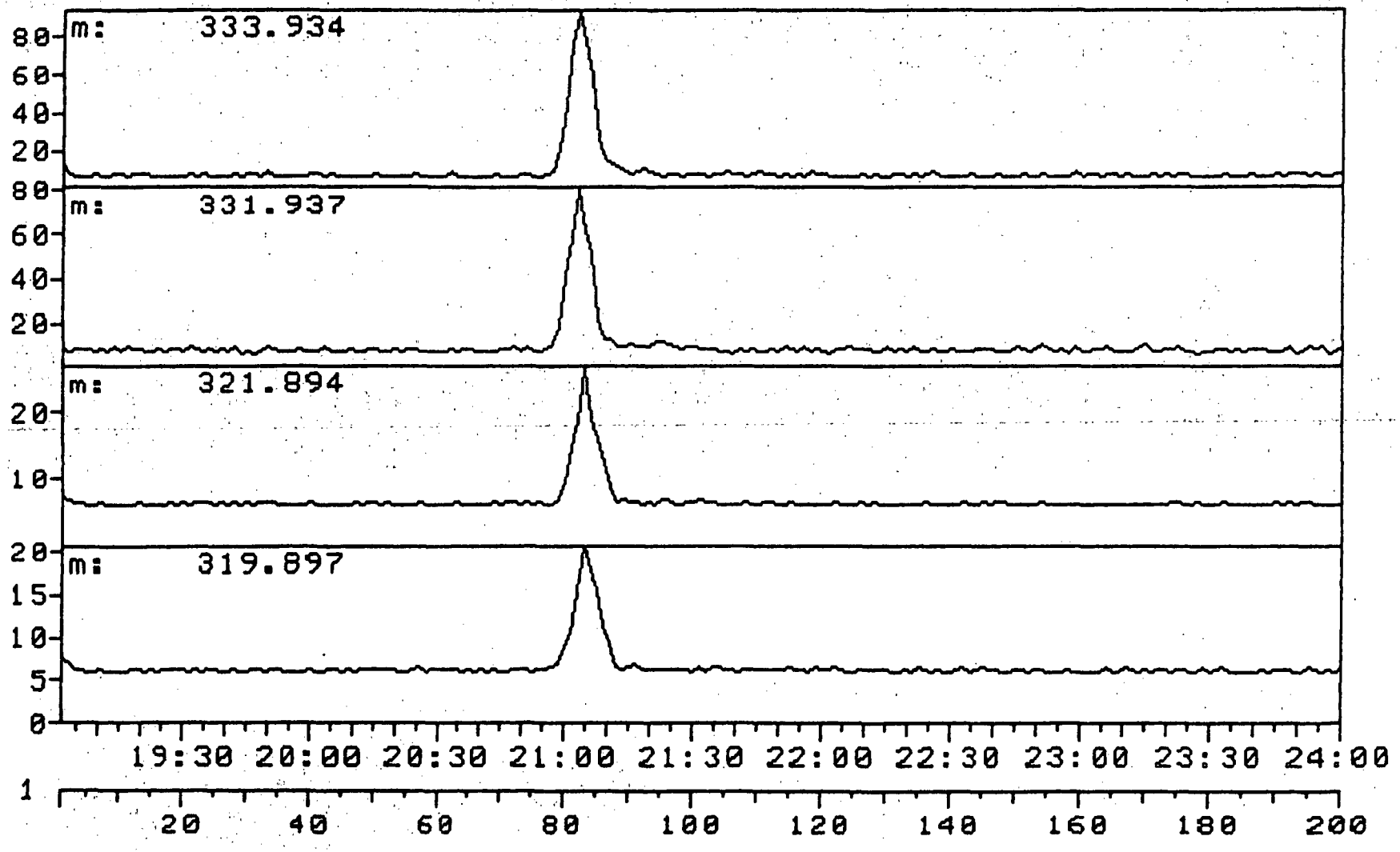
Operator: KC/KD Spc: 5

Sample ID: FISH MDC12 Date: 13-OCT-89 16:33:42

Commission: R=7000 10-13-89

50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5UL

100HZ FILTER 8 CYCLE/GC PEAK



A-64

SSX: PLIST of DM00:[300,303]MDC12.PEA;2
Finnigan MAT Oct 23 89 18:18:26 Page: 1

Calibration curve not well-defined for
Area , Height - use PPLOT to examine data
Sample Identification: FISH MDC12
Filename: DM00:[300,303]MDC12.MIS;1
Creation Date & Time: 13-OCT-89 16:33:42
Integrated Area: 1.192E+03 Integrated Height: 192
Maximum Area: 5.615E+02 Maximum Height: 86
Comment: 50M HP-5 160(2) 25/M 270(20) SEM 1 4KV 5U
L

Time Centroid	AREA Abs	AREA Base	AREA Sum	HEIGHT Abs	HEIGHT Base	HEIGHT Sum
Trace: 319.8965						
21:05	9.076E+01	16.16	7.62	14	16.28	7.29
Trace: 321.8937						
21:05	1.114E+02	19.84	9.35	20	23.26	10.42
Trace: 331.9368						
21:03	4.282E+02	76.26	35.93	72	83.72	37.50
Trace: 333.9339						
21:04	5.615E+02	100.00	47.11	86	100.00	44.79

**** PLIST Processing complete ****

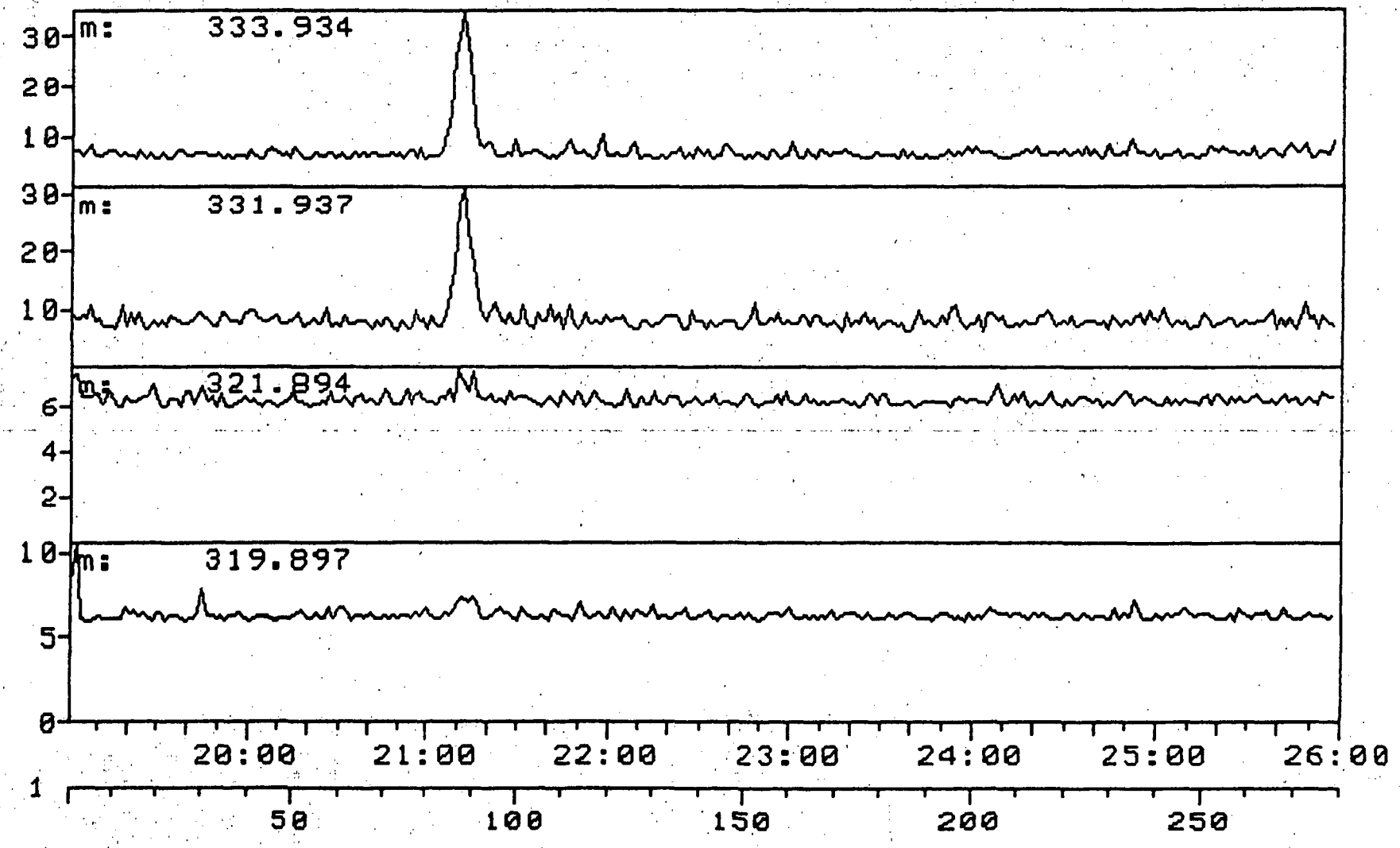
Analysis Name: DM00:[300,303]MDC11.MIS;1 Window: 1

Operator: KC/KD Spc: 5

Sample ID: MDC11 Date: 13-OCT-89 14:30:31

Commission: R=7000 10-13-89

50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5UL



A-66

A-67

SSX: PLIST of DM00:[300,303]MDC11.PEA;5
Finnigan MAT Oct 25 89 13:03:49 Page: 1

Calibration curve not well-defined for
Area , Height - use PPLOT to examine data
Sample Identification: MDC11
Filename: DM00:[300,303]MDC11.MIS;1
Creation Date & Time: 13-OCT-89 14:30:31
Integrated Area: 3.210E+02 Integrated Height: 54
Maximum Area: 1.762E+02 Maximum Height: 29
Comment: 50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5U
L

Time Centroid	AREA Abs	AREA Base	AREA Sum	HEIGHT Abs	HEIGHT Base	HEIGHT Sum
Trace: 319.8965						
21:07	4.454E+00	2.53	1.39	1	3.45	1.85
Trace: 321.8937						
21:07	5.166E+00	2.93	1.61	1	3.45	1.85
Trace: 331.9368						
21:13	1.352E+02	76.76	42.12	23	79.31	42.59
Trace: 333.9339						
21:11	1.762E+02	100.00	54.88	29	100.00	53.70

**** PLIST Processing complete ****

Analysis Name: DM00:[300,303]MDC8.MIS;1 Window: 1

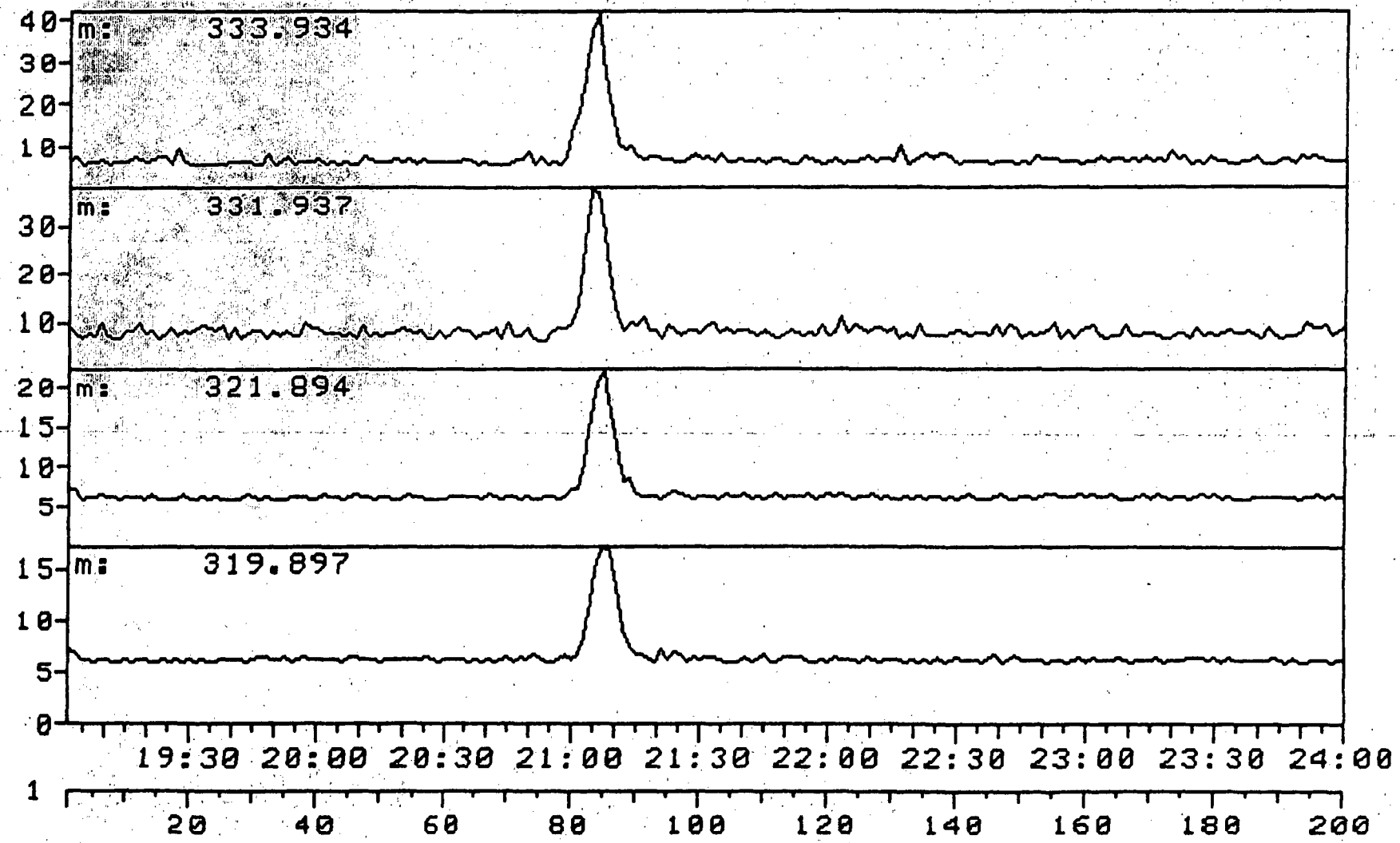
Operator: KC/KD Spc: 5

Sample ID: FISH MDC8 Date: 13-OCT-89 17:07:08

Commission: R=7000 10-13-89

50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5UL

100HZ FILTER 8 CYCLE/GC PEAK



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A-69

Calibration curve not well-defined for
Area , Height - use PPLOT to examine data
Sample Identification: FISH MDC8
Filename: DM00:[300,303]MDC8.MIS;1
Creation Date & Time: 13-OCT-89 17:07:08
Integrated Area: 6.041E+02 Integrated Height: 92
Maximum Area: 2.328E+02 Maximum Height: 35
Comment: 50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5U
L

Time Centroid	AREA Abs	AREA Base	AREA Sum	HEIGHT Abs	HEIGHT Base	HEIGHT Sum
Trace:	319.8965					
21:07	7.948E+01	34.14	13.16	11	31.43	11.96
Trace:	321.8937					
21:07	1.023E+02	43.93	16.93	16	45.71	17.39
Trace:	331.9368					
21:06	1.895E+02	81.40	31.37	30	85.71	32.61
Trace:	333.9339					
21:06	2.328E+02	100.00	38.54	35	100.00	38.04

**** PLIST Processing complete ****

Analysis Name: DM00:[300,303]MDC7.MIS;1 Window: 1

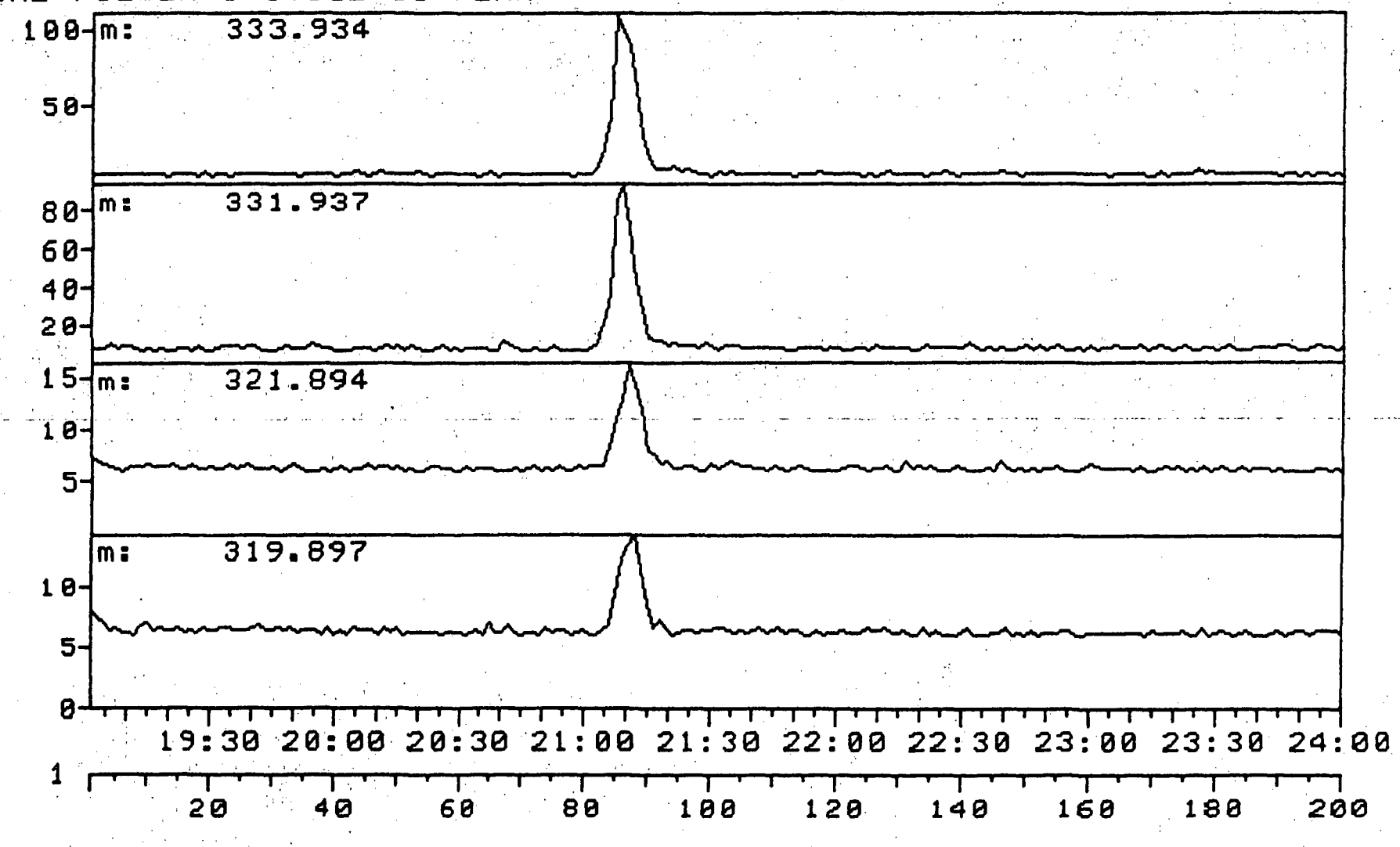
Operator: KC/KD Spc: 5

Sample ID: MDC7 Date: 13-OCT-89 14:59:20

Commission: R=7000 10-13-89

50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5UL

100HZ FILTER 8 CYCLE/GC PEAK



A-70

SSX: PLIST of DMOO:[300,303]MDC7.PEA;3
Finnigan MAT Oct 23 89 17:33:24 Page: 1

Calibration curve not well-defined for
Area , Height - use PPLOT to examine data
Sample Identification: MDC7
Filename: DMOO:[300,303]MDC7.MIS;1
Creation Date & Time: 13-OCT-89 14:59:20
Integrated Area: 1.314E+03 Integrated Height: 206
Maximum Area: 6.650E+02 Maximum Height: 103
Comment: 50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5U
L

Time	AREA			HEIGHT		
Centroid	Abs	Base	Sum	Abs	Base	Sum
Trace: 319.8965						
21:11	4.755E+01	7.15	3.62	8	7.77	3.88
Trace: 321.8937						
21:11	5.751E+01	8.65	4.38	10	9.71	4.85
Trace: 331.9368						
21:10	5.441E+02	81.81	41.40	85	82.52	41.26
Trace: 333.9339						
21:10	6.650E+02	100.00	50.60	103	100.00	50.00

**** PLIST Processing complete ****

Analysis Name: DM00:[300,303]MDC5.MIS;1 Window: 1

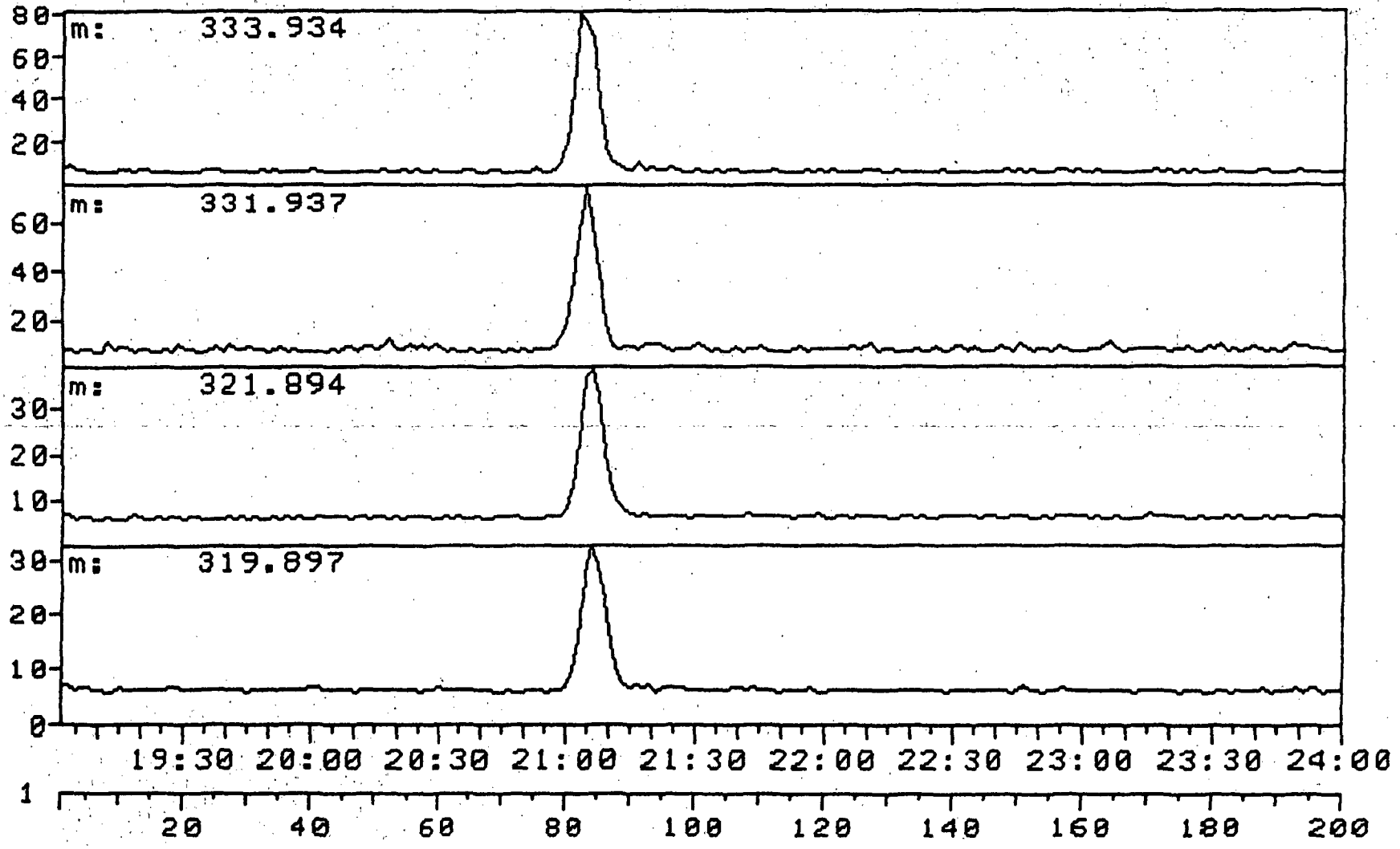
Operator: KC/KD Spc: 5

Sample ID: FISH MDC5 Date: 13-OCT-89 19:07:55

Commission: R=7000 10-13-89

50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5UL

100HZ FILTER 8 CYCLE/GC PEAK



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A-73

Calibration curve not well-defined for
Area , Height - use PPLOT to examine data
Sample Identification: FISH MDC5
Filename: DM00:[300,303]MDC5.MIS;1
Creation Date & Time: 13-OCT-89 19:07:55
Integrated Area: 1.208E+03 Integrated Height: 199
Maximum Area: 4.601E+02 Maximum Height: 74
Comment: 50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5U
L

Time	AREA			HEIGHT		
Centroid	Abs	Base	Sum	Abs	Base	Sum
Trace: 319.8965						
21:07	1.614E+02	35.09	13.36	26	35.14	13.07
Trace: 321.8937						
21:06	2.053E+02	44.62	16.99	32	43.24	16.08
Trace: 331.9368						
21:04	3.811E+02	82.84	31.55	67	90.54	33.67
Trace: 333.9339						
21:04	4.601E+02	100.00	38.09	74	100.00	37.19

**** PLIST Processing complete ****

Analysis Name: DM00:[300,303]MDC3B.MIS;1 Window: 1

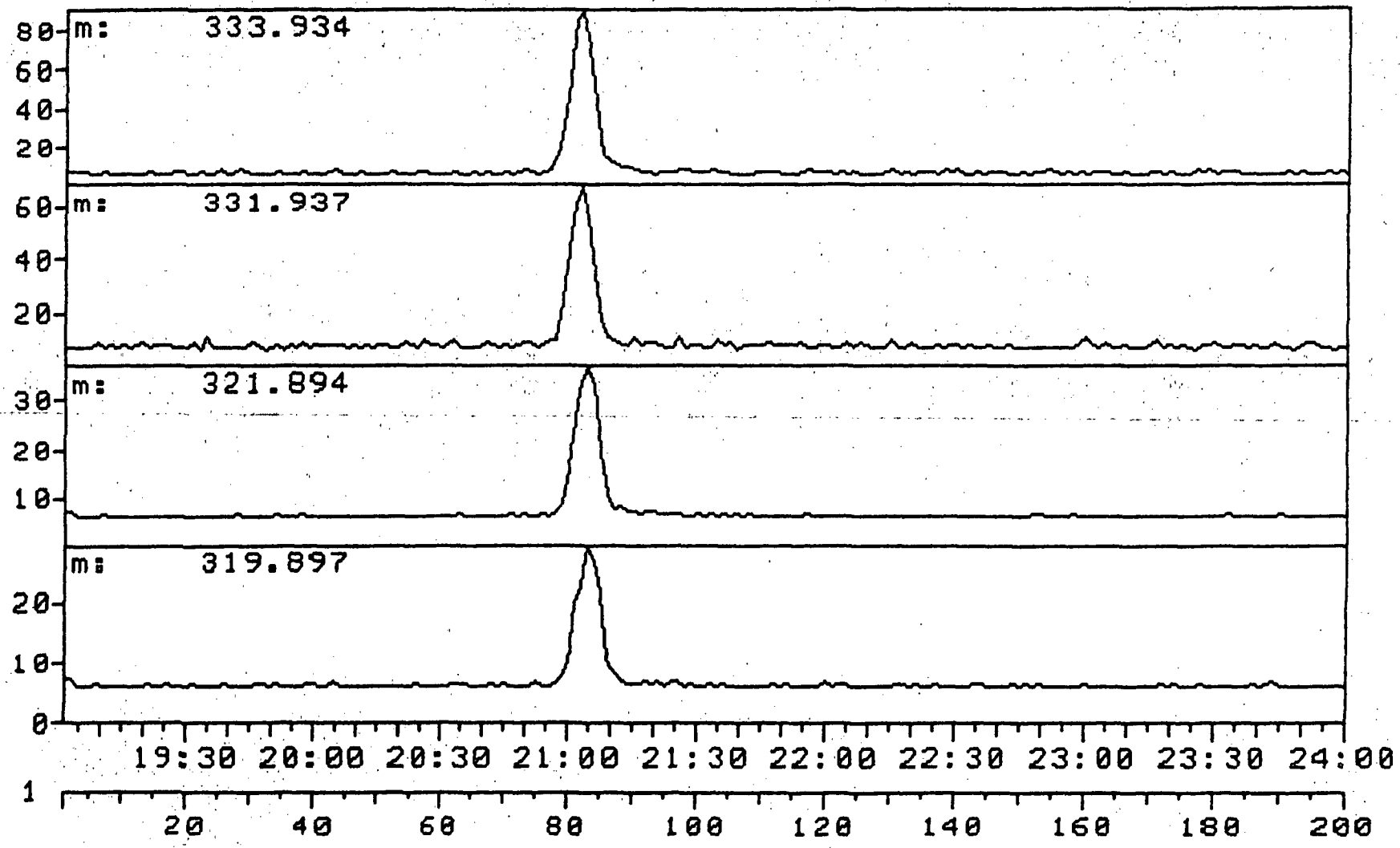
Operator: KC/KD Spc: 5

Sample ID: FISH MDC3 Date: 13-OCT-89 18:37:34

Commission: R=7000 10-13-89

50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5UL

100HZ FILTER 8 CYCLE/GC PEAK DUPLICATE ANALYSIS



A-44

A-75

Calibration curve not well-defined for
Area , Height - use PPLOT to examine data
Sample Identification: FISH MDC3
Filename: DM00:[300,303]MDC3B.MIS;1
Creation Date & Time: 13-OCT-89 18:37:34
Integrated Area: 1.266E+03 Integrated Height: 195
Maximum Area: 5.260E+02 Maximum Height: 82
Comment: 50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5U
L

Time Centroid	AREA Abs	AREA Base	AREA Sum	HEIGHT Abs	HEIGHT Base	HEIGHT Sum
Trace: 21:05	319.8965 1.527E+02	29.03	12.06	23	28.05	11.79
Trace: 21:04	321.8937 1.994E+02	37.90	15.75	30	36.59	15.38
Trace: 21:03	331.9368 3.880E+02	73.77	30.65	60	73.17	30.77
Trace: 21:03	333.9339 5.260E+02	100.00	41.54	82	100.00	42.05

**** PLIST Processing complete ****

Analysis Name: DM00:[300,303]MDC3A.MIS;1 Window: 1

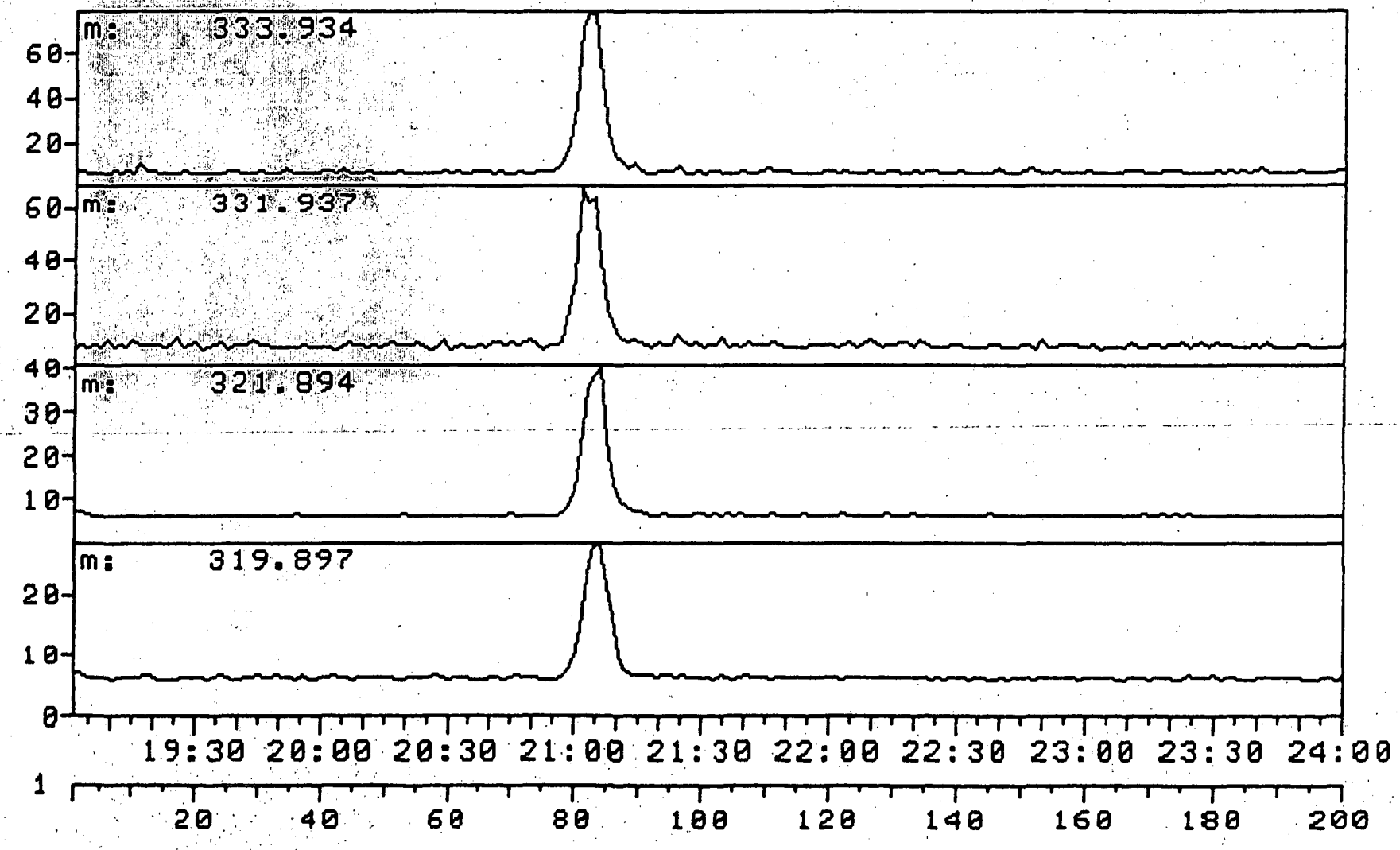
Operator: KC/KD Spc: 5

Sample ID: FISH MDC3 Date: 13-OCT-89 18:07:37

Commission: R=7000 10-13-89

50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5UL

100HZ FILTER 8 CYCLE/GC PEAK



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A-77

Calibration curve not well-defined for
Area , Height - use PPLOT to examine data
Sample Identification: FISH MDC3
Filename: DM00:[300,303]MDC3A.MIS;1
Creation Date & Time: 13-OCT-89 18:07:37
Integrated Area: 1.280E+03 Integrated Height: 186
Maximum Area: 5.103E+02 Maximum Height: 71
Comment: 50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5U
L

Time Centroid	AREA Abs	AREA Base	AREA Sum	HEIGHT Abs	HEIGHT Base	HEIGHT Sum
Trace: 319.8965						
21:05	1.513E+02	29.65	11.82	22	30.99	11.83
Trace: 321.8937						
21:05	2.144E+02	42.01	16.75	33	46.48	17.74
Trace: 331.9368						
21:03	4.039E+02	79.14	31.56	60	84.51	32.26
Trace: 333.9339						
21:03	5.103E+02	100.00	39.87	71	100.00	38.17

**** PLIST Processing complete ****

Analysis Name: DM00:[300,303]MDC2.MIS;2 Window: 1

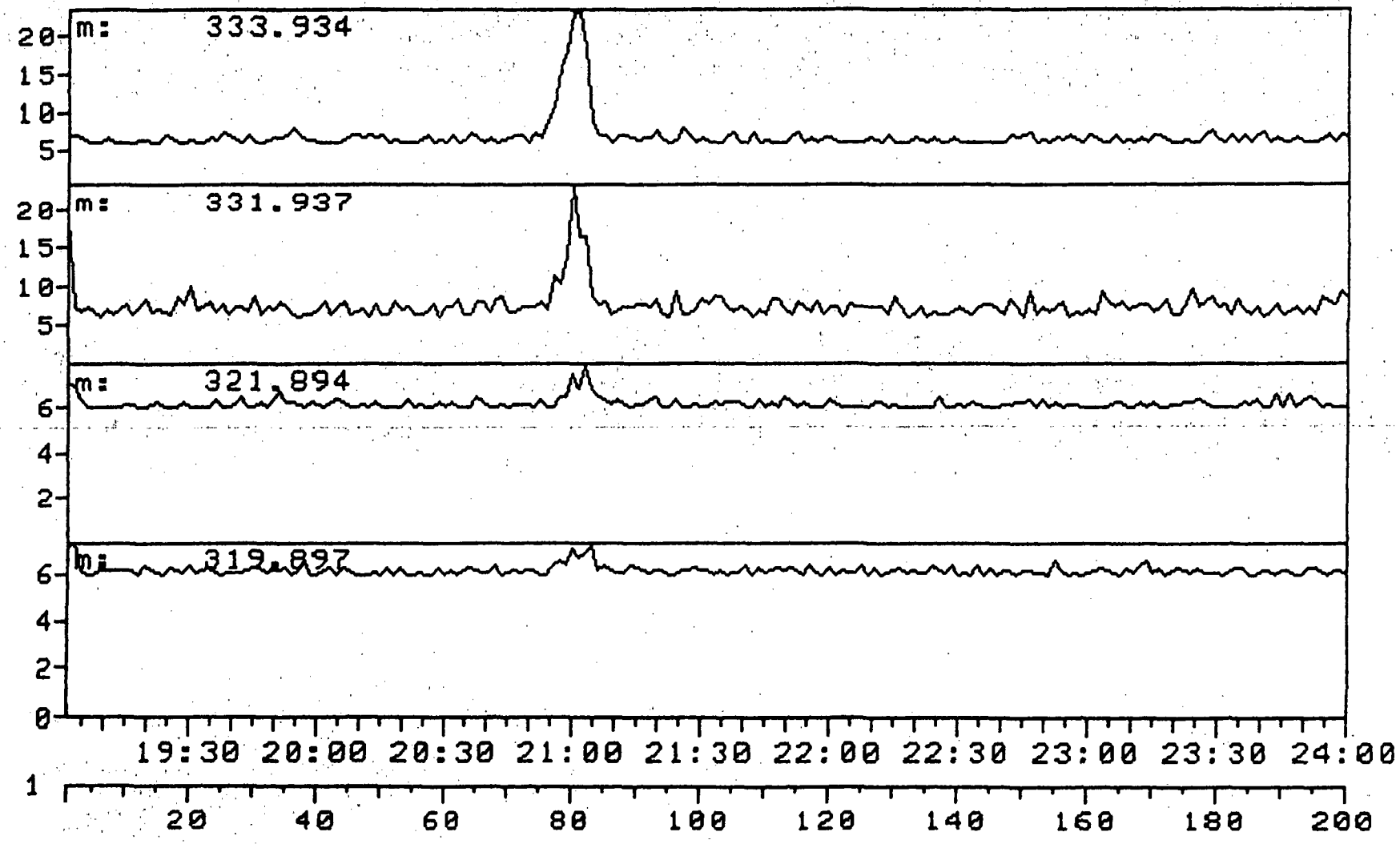
Operator: KC/KD Spc: 5

Sample ID: FISH MDC2 Date: 13-OCT-89 20:40:15

Commission: R=7000 10-13-89

50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5UL

100HZ FILTER 8 CYCLE/GC PEAK



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A-79

SSX: PLIST of DM00:[300,303]MDC2.PEA;10
Finnigan MAT Oct 25 89 12:45:47 Page: 1

Calibration curve not well-defined for
Area , Height - use PPLOT to examine data
Sample Identification: FISH MDC2
Filename: DM00:[300,303]MDC2.MIS;2
Creation Date & Time: 13-OCT-89 20:40:15
Integrated Area: 2.239E+02 Integrated Height: 36
Maximum Area: 1.218E+02 Maximum Height: 17
Comment: 50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5U
L

Time Centroid	AREA Abs	AREA Base	AREA Sum	HEIGHT Abs	HEIGHT Base	HEIGHT Sum
Trace: 319.8965						
20:58	6.182E+00	5.08	2.76	1	5.88	2.78
Trace: 321.8937						
21:02	8.190E+00	6.73	3.66	2	11.76	5.56
Trace: 331.9368						
21:00	8.782E+01	72.13	39.21	16	94.12	44.44
Trace: 333.9339						
21:00	1.218E+02	100.00	54.37	17	100.00	47.22

**** PLIST Processing complete ****

Analysis Name: DM00:[300,303]MDC1.MIS;1 Window: 1

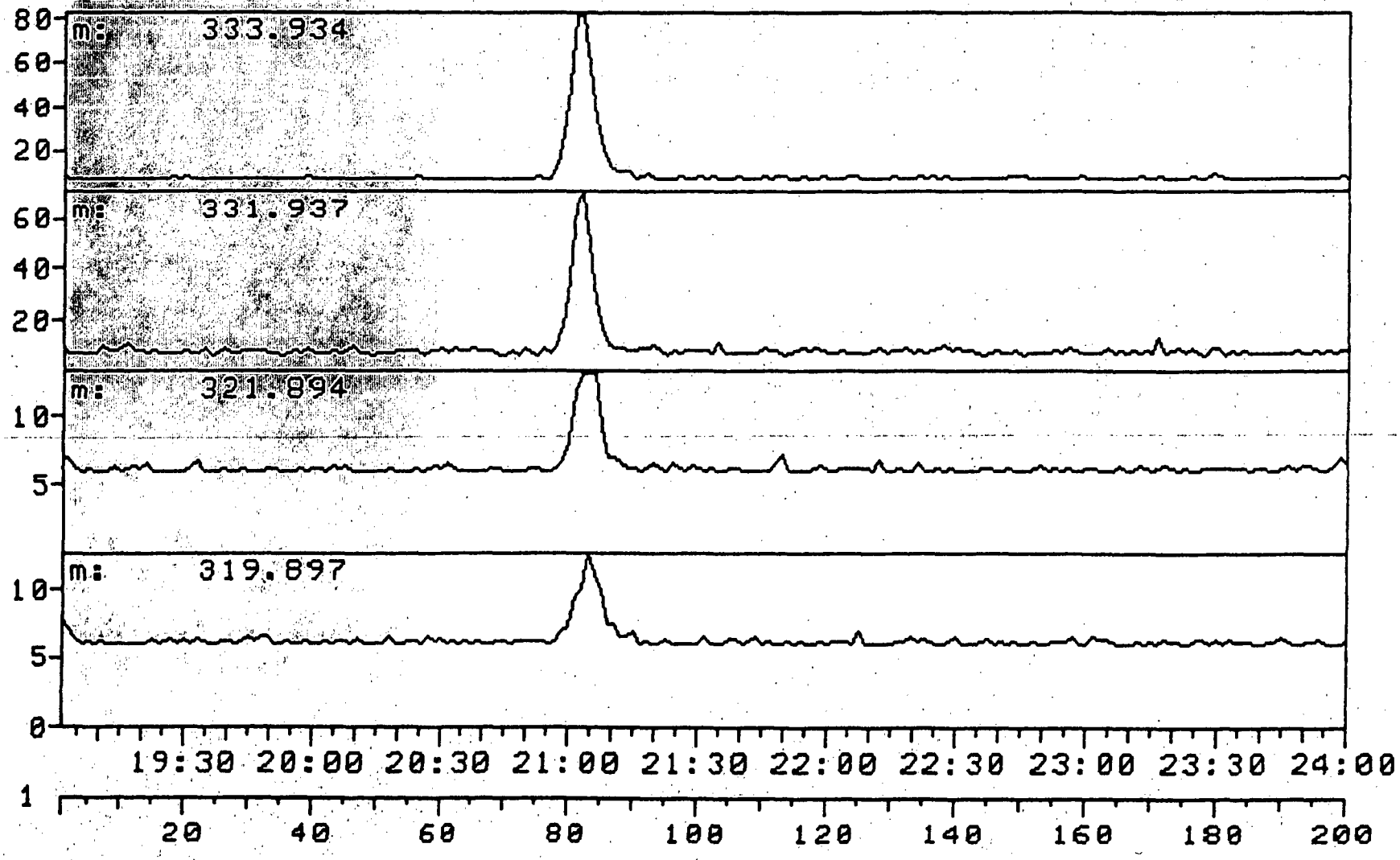
Operator: KC/KD Spc: 5

Sample ID: FISH MDC1 Date: 13-OCT-89 17:36:24

Commission: R=7000 10-13-89

50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5UL

100HZ FILTER 8 CYCLE/GC PEAK



A-80

SSX: PLIST of DM00:[300,303]MDC1.PEA;3
Finnigan MAT Oct 23 89 17:51:41 Page: 1

Calibration curve not well-defined for
Area , Height - use PPLOT to examine data
Sample Identification: FISH MDC1
Filename: DM00:[300,303]MDC1.MIS;1
Creation Date & Time: 13-OCT-89 17:36:24
Integrated Area: 9.274E+02 Integrated Height: 151
Maximum Area: 4.949E+02 Maximum Height: 76
Comment: 50M HP-5 160(2) 25/M 270(20) SEM 1.4KV 5U
L

Time Centroid	AREA Abs	AREA Base	AREA Sum	HEIGHT Abs	HEIGHT Base	HEIGHT Sum
Trace: 319.8965						
21:04	3.503E+01	7.08	3.78	6	7.89	3.97
Trace: 321.8937						
21:04	4.906E+01	9.91	5.29	7	9.21	4.64
Trace: 331.9368						
21:03	3.484E+02	70.39	37.57	62	81.58	41.06
Trace: 333.9339						
21:03	4.949E+02	100.00	53.37	76	100.00	50.33

**** PLIST Processing complete ****

D-45 DEC 0 1989

REQUEST FOR DUPLICATING SERVICE

Name Glenn Curtis Division/Branch WSTM/SPFD Phone 726

Current Date 12/18/89 Date Required 12/21/89

Description of Work Letter & Data From Symtex

No. Originals 50 Est Paper Size (letter, legal) ____ 1 Staple ____

No. Copies 5 Collate ✓ Reduce ____ 2 Staples ____ (up to 50 pages)

Duplex ____ Glue Bind ____ (up to 100 pages)

(2 sided)

Additional Comments/Instructions ____

Superfund material: Yes ✓ No ____ Ia. RCRA Material: Yes ____ No ____

12/19/89 Gerd

Gale A. Wright

Supervisor's Signature

7-EPA-1750.3 (Rev. 6/87)