51895

Site: NBH
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REPORT

RESULTS OF ANALYSES OF NEW BEDFORD HARBOR SEDIMENT EXTRACTS RECEIVED FROM U.S. COAST GUARD R&D CENTER AND OF NEW BEDFORD HARBOR SEDIMENTS RECEIVED FROM NUS CORPORATION FOR POLYCHLORINATED DIBENZO-P-DIOXINS (PCDD), POLYCHLORINATED DIBENZOFURANS (PCDF) AND POLYCHLORINATED BIPHENYLS (PCB)

Prepared by:

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DAYTON, OHIO 45435

Submitted to:

CHARLES BERING
U.S. ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF REGIONAL COUNSEL
JFK FEDERAL BUILDING
ROOM 2203
BOSTON, MASSACHUSETTS 02203

November 21, 1986

SDMS DocID 51895

I. INTRODUCTION

In support of the continuing investigation by the U.S. Environmental Protection Agency of the contamination of New Bedford Harbor in Massachusetts with polychlorinated biphenyls (PCB) and related contaminants, Dr. Thomas O. Tiernan of Wright State University, who is a consultant to the U.S. EPA in connection with litigation relating to this environmental pollution episode, arranged to have relevant samples of sediments from New Bedford Harbor analyzed for PCB, and also for polychlorinated dibenzo-p-dioxins (PCDD) and polychlorinated dibenzofurans (PCDF). Although numerous samples of sediment, water, lobster and fish collected from New Bedford Harbor and its environs have been analyzed for PCB by various laboratories over the past several years, apparently none of these samples have been analyzed for PCDD and PCDF. The latter compounds are now known to have been present in some PCB fluids which were used in electrical devices, or in degradation products from these fluids. Moreover, certain PCDD and PCDF isomers are apparently significantly more toxic (and therefore more hazardous) than PCB. It was therefore of interest to determine if PCDD and PCDF are present in New Bedford Harbor sediments as a consequence of the PCB pollution. Another important objective of the analyses reported herein was to obtain more definitive Gas Chromatograhy-Mass Spectrometry (GC-MS) data on PCB in New Bedford Harbor samples which had been previously analyzed only by GC, or by other techniques such as High Performance Liquid Chromatography (HPLC) and Thin-Layer Chromatography (TLC). Most previous analyses of PCB accomplished on New

Bedford Harbor samples have utilized only GC as the quantitating instrumentation. Only analyses accomplished within the past 1-2 years, such as those currently in progress by Battelle New England Marine Research Laboratory, have utilized GC-MS instrumentation. It is important to obtain some PCB data using GC-MS in such cases, because the latter technique can yield more reliable information on the concentrations of specific chlorinated congener groups of PCB (that is, the mono-, di-, tri-, tetra- and so on chlorinated groups) and even, in some instances, on the concentrations of specific PCB isomers. The latter information is more useful in terms of assessing toxic potential, bioaccumulation, and related factors bearing on the pollution episode than are the GC data which attempt to identify contaminants solely as Aroclors, which are mixtures of large numbers of various PCB isomers, and are the form in which the PCB were originally introduced into the harbor. The latter capability (GC determination of Aroclors) is made more difficult by the fact that all of the components of the originally introduced Aroclor mixture of PCB may no longer be present in the environmental samples analyzed several years following the pollution. This is due to the fact that selective volatilization, degradation or other processes occurring in the environment can alter the observed concentration of some PCB isomers to a greater extent than others, that is, these processes can distort the original Aroclor pattern of PCB components.

Consistent with the objectives cited above, two different sets of samples were shipped to Dr. Tiernan for analyses. One of

these was a set of extracts of New Bedford Harbor sediments which had previously been collected and analyzed by the U.S. Coast Guard (USCG) R&D Center in Groton, CT. Arrangements to obtain these samples were made through Dr. J.R. Jadamec of the USCG R&D Center. The USCG laboratory had extracted some of these samples using hexane-acetone as the solvent, whereas others had been extracted with methanol. These extracts had previously been analyzed by the USCG laboratory using a combination of instrumental techniques, including GC/ECD (Electron Capture Detector), HPLC and TLC. Portions of the very same extracts which the USCG laboratory had analyzed were provided to Dr. Tiernan for analyses by his laboratory. Copies of the shipping and sample identification documentation for these samples are shown in Attachment A to this report. Of the extracts shipped by Dr. Jadamec, it was possible to analyze only two samples in the present study, and it was decided that only the hexane-acetone extracts would be examined. Accordingly, two of the extracts for which the USCG laboratory had found the highest concentrations of PCB, namely USCG Sample Numbers 9 (B,C) and 27, were selected for GC-MS analysis to determine the content of PCB, and in addition, the content of PCDD and PCDF.

The second set of samples provided to Dr. Tiernan by U.S. EPA were actual New Bedford Harbor area sediments which had been collected by coring. These samples were collected by an EPA contractor, NUS Corporation, Superfund Division of Bedford, MA, and sections of the cores were provided to Dr. Tiernan as separate samples. Copies of the shipping and chain-of-custody docu-

mentation for these samples are shown in Attachment B to this report. As seen from the Sample Receipt Memorandum shown in Attachment B, six sample containers, corresponding to three actual sediment samples in duplicate, were received from NUS Corp. Aliquots of each of these three samples, which are from three different core depth sections, were removed from the sample containers and analyzed.

II. ANALYTICAL PROCEDURES UTILIZED

The analytical procedures utilized by Dr. Tiernan's laboratory in determining PCB, PCDD, and PCDF in both the sediment extracts provided by USCG and in the sediments provided by NUS Corp. are described in detail in the Analytical Protocol which is shown as Attachment C to this report. Additional details of the procedures applied in the present analyses are provided in the Intralaboratory Sample Tracking Forms which are shown in Attachments D and E to this report. Shown therein are the quantities of the samples which were analyzed, the amounts of isotopically-labelled internal standards which were added to each sample, the identities of the analysts, the dates of analyses, and references to the actual laboratory notebooks in which details of the analyses are recorded. It should be noted that in the case of the sediments, the labelled internal standards were added to these prior to extraction, and therefore serve as true internal standards for the entire extraction, cleanup and GC-MS measurement sequence. In the case of the sediment extracts received from USCG, of course, the labelled standards could only be added to the extracts as received, and therefore the resulting

data are not corrected for possible losses of the analytes which might be incurred in the extraction process.

III. RESULTS OF THE PRESENT ANALYSES

The results of the analyses for PCDD and PCDF are shown in Attachment F to this report. Table 1 shows the measured concentrations of total tetra-, penta-, hexa-, hepta-, and octachlorinated dibenzo-p-dioxins (CDD) and dibenzofurans (CDF), as well as for 2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD) and 2,3,7,8-tetrachlorodibenzofuran (TCDF) in the two USCG extracts which were analyzed here. Corresponding data for the three NUS sediment samples are shown in Table 2. Tables 3 and 4 present more detailed supporting data for the 2,3,7,8-TCDD analyses, while Tables 5 and 6 give similar data for the 2,3,7,8-TCDF analyses. Tables 7 and 8 show recovery data for the internal standards added to the samples. These recoveries are generally within acceptable bounds, indicating that overall, the analytical method is efficacious. Tables 9 and 10 show calibration data (response factors for standards) and the results of analyzing a performance evaluation mixture to demonstrate adequate GC resolution of the 2,3,7,8-TCDD isomer. Tables 11 through 22 show additional supporting data for the results summarized in Tables 1 and 2. It should be noted that data reported in this section for the sediment samples are on a wet sample weight basis. It should also be noted that the data for the sediment extracts are reported in terms of quantities of analytes per total sample received (rather than in the more customary concentration units.) This is due to the fact that we do not know what quantities of

the sediment samples were extracted to obtain the extracts provided to us by USCG. Also, presumably, we did not receive the entire extract, since USCG had already analyzed portions of these.

The results of the analyses for PCB are shown in Attachment G to this report. Data for total tri-, tetra-, penta-, hexa-, and hepta-CB are given in Tables 23 and 24 for the USCG extracts and the NUS Corp. sediments, respectively. Supporting data and calibration data for the results summarized in Tables 23 and 24 are presented in Tables 25-34. Again, data reported in this section for the sediments received from NUS are on a wet sample weight basis. The moisture contents of the samples analyzed are summarized in Table 35, so the data can be corrected to dry sample weight basis if desired. Here also, the data for the USCG extracts are reported as quantities of analytes in the total extracts, as received, whereas the results reported for the NUS Corp. sediments are expressed in concentration units.

Copies of the actual mass chromatograms obtained in the GC-MS analyses reported herein are shown in Attachments H and I to this report.

The results of the PCDD/PCDF analyses, shown in Tables 1 and 2 (Attachment F), indicate that only higher chlorinated CDD (hexa-, hepta- and octa-) are present in the extracts and in the sediment samples, but that readily detectable quantities of the tetra-, penta-, hexa-, hepta-, and octa-CDF are present in both sample sets. In addition, 2,3,7,8-TCDF is apparently present in these samples, although additional analyses using another capil-

lary GC column would be required to definitively confirm that this isomer is indeed 2,3,7,8-TCDF. These results probably indicate that the PCDF detected originate from the PCB sources, while the higher chlorinated dioxins are associated with other sources. The latter tend to be found in many environmental samples, but are not usually associated with PCB sources.

The results of the PCB analyses summarized in Tables 23 and 24 (Attachment G) indicate that quantities of the tri-, tetra-, penta-, hexa- and hept-CBs are present in the samples. In the sediments, the order of concentrations is tri-CB > tetra-CB > penta-CB > hexa-CB > hepta-CB, and the summed total of all PCBs detected is 232 ppm, 243 ppm, and 124 ppm for NUS/EPA Sample Nos. 13855, 13858, and 13867, respectively. The quantities of total PCBs in the USCG sediment extracts are quite high, being 21,502 micrograms and 16,074 micrograms in USCG Sample Nos. 27 and 9 (B,C), respectively. Presumably these are consistent with the very high concentrations reported by USCG for these same extracts, but as already noted, we cannot directly relate our results to the USCG data since we do not know what quantities of the sediments were extracted by USCG. The order of concentrations of the PCB congener groups in the extracts is slightly different than that found in the sediments, and somewhat different even in the two extract samples analyzed here.

IV. DISCUSSION

In general, the results obtained from the present analyses fully support the previous observations of PCBs in New Bedford

Harbor sediment samples, and tend to confirm the validity of the U.S. Coast Guard R&D Center analyses. In addition, for the first time, as far as we are aware, PCDD and PCDF have been detected in such samples.

ATTACHMENT A

SAMPLE RECEIPT DOCUMENTATION FOR SAMPLE EXTRACTS
RECEIVED FROM U.S. COAST GUARD R&D CENTER,
GROTON, CT

WRIGHT STATE

Wright State University
Campus Communication

Date: Dec. 16, 1985

To: Memorandum for File

From: Dr. T.O. Tiernan

Subject: Sample Extracts

Received From: J.R. Jadamec
U.S. Coast Guard R&D Center
Avery Point
Groton, CT 06340-6096

Date Shipped: Dec. 11, 1985
Date Received: Dec. 16, 1985
Shipped By: U.S. Mail, Registered
Registered Mail I.D. No.: R 227-161-671

WSU Sample No.	U.S. Coast Guard Identification	Core Depth (Section Extracted)	Matrix Provided	Comments	Approximate Sample Quantity	Sample Condition
USC 6-1	27	5 1/2 - 6 1/2 inches	Hexane-Acetone Extract	Bright Yellow Solution	10 mL	Good
USC 6-2	9(B,C)	5 1/2 - 6 1/2 inches	Hexane-Acetone Extract	Bright Yellow Solution	5 mL	Good
USC 6-3	10(A,B)	8 1/2 - 9 1/2 inches	Hexane-Acetone Extract	Bright Yellow Solution	5 mL	Good
USC 6-4	31	9 - 10 inches	Hexane-Acetone Extract	Bright Yellow Solution	10 mL	Good
USC 6-5	2	5 1/2 - 6 1/2 inches	Hexane-Acetone Extract	Bright Yellow Solution	10 mL	Good
USC 6-6	28	0 - 1 inch	Methanol Extract	Yellow Solution	2 mL	Good
USC 6-7	28	5 1/2 - 6 1/2 inches	Methanol Extract	Yellow Residue, Nearly Dry	0.5 mL	Good
USC 6-8	28	12 - 13 inches	Methanol Extract	Yellow Residue, Dry	Dry	Good

The samples are solvent extracts of sediment cores which were shipped in vials (screw capped and sealed with tape on the outside) in a cardboard box lined with plastic foam pieces, by Registered U.S. Mail, addressed to Dr. Tiernan. Dr. Tiernan opened the box, after removing the outer paper wrapping, inspected the samples and prepared the sample receipt memorandum. Information regarding the samples was provided on the label

affixed to each vial, as well as in an attached letter from J. Richard Jadamec (copy attached). Note that the letter from Mr. Jadamec, of the U.S. Coast Guard R&D Center, indicates that the second set of three samples are identified as methanol extracts of USCG Sample No. 27, corresponding to three different core sections. However, the actual sample vials received for this set showed sample number 28 on the labels attached to the vials. No formal chain-of-custody accompanied these samples, although chain-of-custody was maintained. A copy of the front portion of the paper wrapper which covered the cardboard shipping box which contained the sample extracts is also attached. After opening the container, the samples were placed in the refrigerator in 067 Brehm Laboratory until some of these were analyzed.

U.S. Department of Transportation
United States Coast Guard

USCG R&D Center
Avery Point
Groton, CT 06340-6096

764154.1
Ser: 2154B (JRJ)
December 10, 1985

Dr. Thomas O. Tiernan
Brehm Laboratory
Wright State University
Dayton, Ohio 45435

Dear Tom:

I have enclosed a draft copy of a paper presented at a local spill workshop describing our methods used in the analysis of Acushnet River Sediments. We are going to restructure this paper, include additional data, and in the near future submit it for publication.

As you requested at the 18 November 1985 meeting I am forwarding the following samples under separate cover:

Sample No.	Core Depth	Concentration (ppm)
2	5 1/2 - 6 1/2 inches	30,700
9 (B,C)	5 1/2 - 6 1/2 inches	30,700
10 (A,B)	8 1/2 - 9 1/2 inches	19,650
27	5 1/2 - 6 1/2 inches	66,500
31	9 - 10 inches	2

These above samples are hexane/acetone extracts. The samples were prepared by sonification. As evidenced, they have undergone some evaporative losses. Additionally, I have enclosed a complete set of the methanol extracts of sample number 27, i.e., 0-1 inch, 5 1/2 - 6 1/2 inches, and 12-13 inches. These concentrations are 1,980; 66,500; and 27 ppm, respectively. All concentrations are reported relative to Aroclor 1254.

If you have any questions feel free to contact me by telephone (203) 441-2723.

Sincerely,

J. Richard Jadamec

J. RICHARD JADAMEC

Encl: (1) Draft, "Studies of PCB's in the Acushnet River Estuary"

Copy: Ms. Patti Saris
Room 1017
John W. McCormick Building
Boston, MA 02116

16 DEC 1985
RECEIVED

DEPARTMENT OF TRANSPORTATION
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Dayton, Ohio 45435

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ATTACHMENT B

SAMPLE RECEIPT DOCUMENTATION FOR SEDIMENT SAMPLES
RECEIVED FROM NUS CORP., SUPERFUND DIVISION,
BEDFORD, MA.

BREHM LABORATORY, WRIGHT STATE UNIVERSITY, DAYTON, OHIO 45435

SAMPLE LOG-IN CHECK LIST

1. Samples Received by: Garrett Van Ness Date: 11/1/85

   At Location: Shipping + Receiving

2. Sample Received from: NUS CORP. MARK MENGEL Date Shipped 10/31/85
   19 CROSBY DR, 1ST FLOOR
   BEDFORD, MA 01730

3. Project Number: N/A

4. Chain of Custody Seals Intact: N/A

5. Chain of Custody Records Signed by: MARK J. MENGEL

6. WSU Laboratory Sample Designation: NBC5

7. Sample Logged in Notebook: YES

8. Sample Log-In Sheet Completed: (EPA Samples) YES

9. Comments on Sample Shipment:

   a) Samples Received and Number of Each Sample Type: 6 SEDIMENT CORE SAMPLES

   b) Shipping Container Type: COLEMAN COOLER, SEALED WITH FIBER TAPE

   c) Condition of Shipping Container: GOOD

   d) Packing Material (Type): SHREDDED CELLULITE; INDIVIDUAL METAL CANISTER PER SAMPLE, WITH ABOVE CELLULITE AND ZIPLOCK PLASTIC BAG.

   e) Is Amount of Packing Material Sufficient? YES

   f) Type of Sample Vessel and Its Condition: 4 OZ GLASS JARS; GOOD

   g) Type of Seal on Sample Vessels and Condition of Seal: PLASTIC SCREW-TYPE CAPLIDS; GOOD

   h) Type(s) of Sample Identification: SHIPPING LABEL, AIRBILL, CHAIN OF CUSTODY RECORD, SAMPLE VESSEL LABELS

   i) Condition of Samples: GOOD

   j) Miscellaneous Comments: NBC5-1, NBC5-3, AND NBC5-5 HAVE WHITE SAMPLE VESSEL LIDS
   NBC5-2, NBC5-4, AND NBC5-6 HAVE BLACK SAMPLE VESSEL LIDS

10. Approved by Sample Custodian: [signature] Date: 11/18/85

SAMPLE LOG-IN SHEET

ASSISTANT SAMPLE CUSTODIAN SIGNATURE: [signature] DATE: 11/15/85

SAMPLE CUSTODIAN SIGNATURE: [signature] DATE: 11/18/85

CIRCLE THE APPROPRIATE RESPONSE

1. Custody Seal — present/absent; intact/not intact
2. Chain-of-Custody — present/absent
3. Sample Tags — present/absent
   Sample Tag Numbers — listed/not listed on chain-of-custody

DATE RECEIVED 11/1/85

TIME RECEIVED 0920

EPISODE NUMBER

AIRBILL NUMBER 777352951

CHAIN OF CUSTODY RECORD NUMBER	WSU NUMBER	EPA SAMPLE NUMBER	BATCH NUMBERS	CORRESPONDING ~~SAMPLE TAG NUMBERS~~ STATION LOCATION	CORRESPONDING STATION NUMBERS	DOES INFO ON ALL RECORDS AGREE?	REMARKS: CONDITION OF SAMPLE SHIPMENT, ETC.
02995	NBC5-1	13858		0"-6"	I-10	YES	THE SAMPLE VESSEL LIDS FOR
02995	NBC5-2	13858		0"-6"	I-10	YES	NBC5-1, NBC5-3, AND NBC5-
02995	NBC5-3	13855		3"-8"	K-9	YES	WERE WHITE.
02995	NBC5-4	13855		3"-8"	K-9	YES	THOSE FOR NBC5-2, NBC5-4, AN
02995	NBC5-5	13867		6"-12"	I-13	YES	NBC5-6 WERE BLACK.
02995	NBC5-6	13867		6"-12"	I-13	YES	

NUS CORPORATION
SUPERFUND DIVISION

CHAIN OF CUSTODY RECORD
REM/FIT PROJECT

PROJECT NO.:

WORK PLAN NO.:

SITE NAME: New Bedford Cores

SAMPLERS (SIGNATURE): Mark L. Mengel

STATION NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION	NO. OF CONTAINERS	4oz GLASS JAR						REMARKS
K-9	10/29	0927			3"-8"	2	2						Sediment cores from wetlands
I-10	10/29	1003			0"-6"	2	2						" " " "
I-13	10/29	1130			6"-12"	2	2						" " " "

RELINQUISHED BY (SIGNATURE):	DATE/TIME:	RECEIVED BY (SIGNATURE):	RELINQUISHED BY (SIGNATURE):	DATE/TIME:	RECEIVED BY (SIGNATURE):
Mark L. Mengel	10-31-85 1300	Airbill # 777352951			
RELINQUISHED BY (SIGNATURE):	DATE/TIME:	RECEIVED BY (SIGNATURE):	RELINQUISHED BY (SIGNATURE):	DATE/TIME:	RECEIVED BY (SIGNATURE):
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CITY: BEDFORD STATE: MA

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TO (Recipient's Name): Dr. Tiernan

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COMPANY: Beckman Research Lab. Wright St. Univ.

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CITY: STATE: ZIP:

RECEIVED BY: (Signature) X

DATE/TIME RECEIVED: 11/1/85 9:20

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NAME AND TITLE OF PERSON SIGNING CERTIFICATION	EMERGENCY TELEPHONE NO.	SIGNATURE OF SHIPPER

ATTACHMENT C

ANALYTICAL PROTOCOL APPLIED BY THE BREHM LABORATORY TO DETERMINE POLYCHLORINATED DIBENZO-P-DIOXINS (PCDD), POLYCHLORINATED DIBENZOFURANS (PCDF) AND POLYCHLORINATED BIPHENYLS (PCB) IN SEDIMENT SAMPLES AND IN U.S. COAST GUARD-PREPARED EXTRACTS OF SEDIMENT SAMPLES

BREHM LABORATORY, WRIGHT STATE UNIVERSITY,
DAYTON, OHIO 45435

ANALYTICAL PROTOCOL APPLIED FOR EXTRACTION AND QUANTITATION OF POLYCHLORINATED DIBENZO-P-DIOXINS (PCDD), POLYCHLORINATED DIBENZOFURANS (PCDF), AND POLYCHLORINATED BIPHENYLS (PCB) IN SEDIMENT SAMPLES AND IN EXTRACTS OF SEDIMENTS PREPARED BY ANOTHER LABORATORY

A. Drying Procedure, Calculation of Percent Moisture For Sediment Core Samples

1. Withdraw an aliquot of the sediment (approximately 10-20 grams) after stirring the sample to make it homogeneous, and accurately weigh the aliquot by placing it into a new tared 250 mL flint glass bottle. New glass bottles are pre-cleaned prior to use by rinsing with methanol, followed by rinsing with methylene chloride, and air drying. The bottles are fitted with teflon-lined screw caps. The balance used to weigh samples should be capable of weighing to the nearest hundredth of a gram. To the sample, add a known quantity (approximately 60 g) of purified anhydrous sodium sulfate and mix thoroughly with a spatula. Allow the sample to stand for a period of approximately 4 hours. After this period has elapsed, the sample/sodium sulfate mixture should flow smoothly within the bottle. If this is not the case and the sample is still insufficiently dry, add another 20 g of anhydrous purified sodium sulfate, mix thoroughly, and allow the sample mixture to stand for another 2 hours. Repeat until the sample mixture is dry and free flowing. The dried sample mixture is then transferred to a pre-cleaned Soxhlet apparatus, as discussed in Section B.1..

2. Place a second aliquot (approximately 5 grams) of the sediment sample into an aluminum boat, weigh the aluminum boat

and the sample after transferring the sediment to it, and then dry this aliquot for 24 hours by heating it at 105°C in an oven. Remove the aluminum boat containing the dried sample from the oven, place it in a desiccator and allow it to cool to room temperature. Remove the aluminum boat from the desiccator after cooling, obtain a final weight of the dried sample and the boat and calculate the percent moisture in the soil sample as received. Discard the dried sample aliquot after determining the percent moisture.

B. Isolation of PCDD and PCDF from the Sample Matrix

1. Prepare a glass Soxhlet extraction thimble (90 mm by 35 mm) for use by rinsing sequentially with methanol, acetone, and methylene chloride. Add silica to form a 3-6 mm layer on the surface of the glass frit at the bottom of the thimble and place a 10 mm layer of glass wool over the layer of silica.

2. Prepare a soxhlet extraction apparatus, consisting of a Soxhlet extraction tube, a 250 mL Erlenmeyer flask and a water-cooled condenser, for use by rinsing sequentially with methanol, acetone, and methylene chloride, and allowing it to air-dry. Place 175 mL of a solution consisting of 50% benzene and 50% acetone (by volume) along with 6-10 pre-cleaned 2 mm glass beads, into the Erlenmeyer flask. Place the Soxhlet thimble (prepared as described in Step #1) into the Soxhlet extraction tube, assemble the Soxhlet extraction apparatus, heat the contents of the Erlenmeyer flask to reflux temperature and continue the Soxhlet extraction procedure for a period of 3 hours.

3. Remove the heat source from the Soxhlet apparatus, allow

the apparatus to cool, and then decant the benzene/acetone solution into a clean, 250 mL flint glass bottle and seal the bottle with a Teflon-lined screw cap. This solution is retained in case additional analyses are required to check the cleanliness of the Soxhlet apparatus, as a QA/QC measure.

4. Place a fresh 175 mL aliquot of 50:50 (volume/volume) benzene/acetone into the precleaned Erlenmeyer flask of the Soxhlet extraction apparatus and re-connect the Soxhlet extraction tube to the Erlenmeyer flask. Transfer the sediment/sodium sulfate mixture contained in the screw-capped bottle (see Step A.1.) to the Soxhlet extraction thimble by inverting the bottle and allowing the sample to flow into the Soxhlet extraction thimble. Replace the screw cap on the bottle and re-weigh the bottle. Subtract the weight of the empty bottle from the combined weight of the bottlee and sample mixture to obtain the weight of sediment/sodium sulfate actually transferred to the Soxhlet extraction thimble and subsequently analyzed. Calculate the sample weight utilizing the known ratio of sediment to sodium sulfate.

5. Using a microsyringe, add, typically, 1-2 ng of an internal standard solution containing $^{13}C_{12}$-2,3,7,8-TCDD, $^{37}Cl_4$-2,3,7,8-TCDD, $^{37}Cl_4$-2,3,7,8-TCDF, $^{13}C_{12}$-)CDD, and d_6-3,3',4,4'-tetrachlorobiphenyl to the Soxhlet extraction thimble. Place the condenser on the Soxhlet extraction tube and heat the solvent reservoir so that the extraction solvent refluxes. Soxhlet extract the sample using refluxing benzene/acetone for a period of 16 hours, then discontinue heating the apparatus.

6. Remove the Soxhlet extractor from the Erlenmeyer flask reservoir and replace it with a 3-ball Snyder column. Resume heating the reservoir and concentrate the benzene/acetone extract to a volume of about 15 mL. Rinse the Snyder column twice with small quantities of 50% benzene/50% acetone solution, then continue heating and concentrating the solution until a final volume of 10 mL is attained.

7. Using a 10 mL disposable pipette, transfer the concentrated solution obtained in Step B.6. to a pre-rinsed, 125 mL flint glass bottle fitted with a Teflon-lined screw cap, and rinse the Erlenmeyer flask four times using 10 mL aliquots of hexane (transferring each rinse solution to the bottle) to effect a quantitative transfer of the concentrate from the Erlenmeyer flask to the bottle.

8. Add 30 mL of aqueous potassium hydroxide (20% w/v) to the organic (hexane) extract in the bottle, seal the bottle and agitate it for a period of 15 minutes. Remove and discard the aqueous phase, retaining the organic layer.

9. Add 30 mL of twice-distilled water to the residual organic extract in the bottle, seal the bottle, and agitate it for a period of 1 minute. Again, remove and discard the aqueous layer, retaining the organic layer.

10. Add 30 mL of concentrated sulfuric acid to the residual organic extract in the bottle, seal the bottle, and agitate it for a period of 15 minutes. Remove the acid layer and discard, retaining the organic layer. Repeat this step until the acid layer is visually colorless or until no further color change is

discernible.

11. Repeat step B.9.

12. Add 5 g of anhydrous sodium sulfate to the organic extract in the bottle, seal the bottle, and allow it to stand for at least 20 minutes.

13. Quantitatively transfer the organic extract from the bottle to a 45 mL glass culture tube, along with three 5 mL hexane rinses of the bottle and the sodium sulfate drying agent.

14. Concentrate the extract in the culture tube to a volume of approximately 5 mL by passing a stream of pre-purified nitrogen over the extract while maintaining the glass tube at a temperature of 55°C by immersing it in a water bath.

15. Fabricate a glass Macro-column (20 mm OD x 230 mm long) tapered to 6 mm OD on one end. Pack the column successively with 1.0 g silica, 2.0 g silica containing 28% (w/w) 1 M NaOH, 1.0 g silica, 4.0 g silica containing 30% (w/w) sulfuric acid, and 2.0 g silica.

16. Quantitatively transfer the concentrated extract obtained in Step B.14. to the silica column, along with two hexane rinses of the tube (1 mL each) and elute the column with 90 mL of hexane. Collect the entire eluate in a culture tube and then concentrate it (by passing a stream of pre-purified nitrogen over the extract) to a volume of 1-2 mL.

17. Construct a disposable liquid chromatography column as described in the following. Cut off a 10 mL Pyrex disposable pipette at the 4 mL mark and retain the lower portion of the pipette. Pack the small end with a plug of silanized glass wool.

Next add 3 grams of Woelm basic alumina previously activated overnight at 600°C in a muffle furnace and placed in a desiccator for 30 minutes just prior to use.

18. Using a disposable pipette, transfer the concentrated extract from Step B.16. to the liquid chromatography column prepared in Step B.17. Rinse the test tube which contained the concentrate successively with two 1 mL portions of hexane, each time transferring the rinsate to the alumina column.

19. Elute the alumina column with 15 mL of hexane and discard the eluent.

20. Elute the alumina column with 10 mL of hexane containing 8% of methylene chloride (v/v). Collect the alumina eluent in a glass culture tube. Retain this fraction for PCB analysis.

21. Elute the alumina column with 15 mL of hexane containing 50% methylene chloride (v/v), collecting the column eluent in a glass culture tube.

22. Concentrate the eluents from Step B.20. and B.21. to a volume of about 0.5 mL by passing streams of pre-purified nitrogen over each solution while heating the culture tubes in a 55°C water bath.

23. To the residues obtained from Steps B.20. and B.21., add 1 ml of methylene chloride, gently swirl each culture tube to dissolve the residue and, using a pipette, transfer the solutions from each tube into separate 3 mL reaction vials. Using a second 1 mL portion of methylene chloride, rinse each tube which contained the residue and transfer this rinsate into the appropriate 3 mL reaction vial. By flowing a stream of pre-purified nitrogen

over the solution in each reaction vial, concentrate these solutions to incipient dryness.

24. To the extract obtained from Step B.21., add 10 uL of the external standard solution containing 0.05 ng $^{13}C_{12}$-1,2,3,4-TCDD per uL of dodecane and proceed to quantitate the PCDD and PCDF in the sample extracts using the GC-MS procedure described below in Section D. Both extracts from Steps B.20. and B.21. are combined for the PCB analyses, using GC-MS procedures described below in Section E. Sample extracts which are not immediately analyzed are stored (shielded from light) at room temperature (20° C ± 3° C).

25. Soil extracts which, following concentration to near dryness, appear (on the basis of visual inspection) to contain extraneous compounds, such as oily or colored residues, which are likely to interfere with the quantitation of PCDD and PCDF, are subjected to an additional chromatographic clean-up procedure prior to addition of the external standard solution, as described in Step B.24. This additional clean-up step is described below.

Prepare a gravity-flow liquid chromatography column by cutting off a 0.5 inch section from the constricted tip of a 9-inch disposable Pasteur pipette. Insert a filter paper disk at the top of the tube, and position the disk 2.5 cm below the constriction. Add a sufficient quantity of PX-21 Carbon/Celite 545 (prepared as described in Section D.8. of this Protocol) to the tube to form a 2 cm length of the Carbon/Celite. Insert a glass wool plug on top of the Carbon/Celite. Pre-elute the column sequentially with 2 mL of a 50% benzene/50% ethyl acetate

solution (v/v), 1 mL of a 50% methylene chloride/50% cyclohexane solution (v/v), and 2 mL of hexane, and discard these eluents. Transfer the residual extract (in 1 mL of hexane) resulting from the alumina column cleanup (Step B.21.) onto the top of the Carbon/Celite column, along with 1 mL of a hexane rinse of the original sample vessel. Elute the column with 2 mL of 50% methylene chloride/50% cyclohexane solution and 2 mL of 50% benzene/50% ethyl acetate and discard these eluents. Invert the column and elute it in the reverse direction with 4 mL of toluene, retaining this eluent. Concentrate the eluent using a stream of pre-purified nitrogen and repeat Steps B.17. - B.24.

C. Preparation Procedure for Sediment Extracts Obtained by Another Laboratory

For samples which have been extracted by another laboratory, the extracts being provided to Wright State for analyses, the following procedures are utilized.

1. Measure the total weight of the liquid extract provided, then remove an aliquot of known weight and transfer it to a 125 mL flint glass bottle filled with a teflon-lined lid.

2. Dilute the aliquot of the extract to be analyzed by adding 40 mL of hexane to the 125 mL glass bottle containing the extract.

3. Add isotopically-labelled internal standards to the bottle containing the sample, as described in Step B.5. in the foregoing section of this protocol.

4. Proceed with Step B.8. and the following procedures, as described in the foregoing section.

D. Reagents and Chemicals

Reagents and chemicals used in implementing the procedures described herein and the sources of these are described in the following:

1. Potassium hydroxide (anhydrous), granular sodium sulfate and sulfuric acid (all Reagent Grade): J.T. Baker Chemical Co., Glen Ellyn, IL, or Fisher Scientific Co., Cincinnati, OH. The granular sodium sulfate is purified prior to use by placing a beaker containing the sodium sulfate in a 400°C oven for four hours, then removing the beaker and allowing it to cool in a desiccator. Store the purified sodium sulfate in a bottle equipped with a Teflon-lined screw cap.

2. Hexane, Methylene Chloride, Benzene, Ethyl Acetate, Methanol, Toluene, Cyclohexane, Isooctane: "Distilled in Glass" Burdick and Jackson, Muskegon, MI.

3. Tridecane, Reagent Grade: Sigma Chemical Co., St. Louis, MO.

4. Basic Alumina, Activity Grade 1: ICN Pharmaceuticals, Cleveland, OH. Immediately prior to use, the alumina is activated by heating for at least 16 hours at 600°C in a muffle furnace and then allowed to cool in a desiccator for 30 minutes prior to use. Store conditioned alumina in a desiccator.

5. Silica (Bio-Sil A 100/200 mesh): Bio-Rad, Rockville Centre, NY. The Bio-Sil A is conditioned prior to use by placing it in a 30 mm x 30 cm long glass tube (the silica gel is held in place by glass wool plugs) which is placed in a tube furnace. The glass tube is connected to a prepurified nitrogen cylinder,

through a series of four traps (stainless steel tubes, 1.0 cm O.D. x 10 cm long)*. Trap Number 1 contains a mixture composed of Chromosorb W/AW (60/80 mesh coated with 5% Apiezon L), graphite (100 mesh, 1-M-USP), and activated carbon (50 to 200 mesh), in a 7:1.5:1.5 ratio. Chromosorb W/AW and Apiezon L were obtained from Supelco, Inc., Bellefonte, Pennsylvania; graphite was obtained from Ultracarbon Corporation, Bay City, Michigan; activated carbon was obtained from Fisher Scientific Co., Cincinnati, Ohio. Trap Number 2 contains Molecular Sieve 13X (60/80 mesh, obtained from Supelco, Inc., Bellefonte, Pennsylvania). Trap Number 3 contains silica gel impregnated with 30% (w/w) sulfuric acid (prepared as described in D.6. below). Trap Number 4 contains Carbosieve S (80/100 mesh, obtained from Supelco, Inc., Bellefonte, Pennsylvania). The first step in conditioning the Bio-Sil A entails heating the glass tube containing the 200 g aliquot of silica for 30 minutes at 180°C while purging with nitrogen (flow rate 50-100 mL/minute), subsequently the tube is removed from the furnace and allowed to cool to room temperature. Methanol (175 mL) is then passed through the tube, followed by 175 mL methylene chloride. The tube containing the silica is then returned to the furnace, the nitrogen purge is again established (50-100 mL/minute flow), the tube is heated at 50°C for 10 minutes, then the temperature is gradually increased to 180°C over a period of 25 minutes and maintained at 180°C for 90 minutes. Heating is then discontinued

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*. See T.J. Nestrick and L.L. Lamparski, Anal. Chem 53, 122 (1981) for additional details and rationale regarding use of these traps.

but the nitrogen purge is maintained until the tube cools to room temperature. Finally, the silica is transferred to a clean, dry, glass bottle and capped with a Teflon-lined screw cap for storage.

6. Silica Gel Impregnated with Sulfuric Acid (30% w/w): Concentrated sulfuric acid (4.4 g) is combined with 10.0 g silica gel (conditioned as described above) in a screw capped bottle and agitated to mix thoroughly. Aggregates are dispersed with a stirring rod until a uniform mixture is obtained. The H_2SO_4 - silica gel is stored in a screw-capped bottle (Teflon-lined cap).

7. Silica Gel Impregnated with Sodium Hydroxide: 1N Sodium Hydroxide (30 g) is combined with 100 g Bio-Sil A (conditioned as described above) in a screw capped bottle and agitated to mix thoroughly. Aggregates are dispersed with a stirring rod until a uniform mixture is obtained. The NaOH-silica gel is stored in a screw-capped bottle (Teflon-lined cap).

8. Carbon/Celite: Combine Amoco PX-21 carbon (Amoco, Naperville, IL) (10.7 g) with Celite 545 (Fisher Scientific Co.) (125 g) in a 250 mL glass bottle fitted with a Teflon-lined cap. Agitate the mixture to combine thoroughly. Store in the screw-capped bottle.

9. Nitrogen (Prepurified) and Hydrogen (Ultra High Purity): Airco, Inc. Montvale, NJ.

D. Calibration Standards

1. PCDD and PCDF

Stock standard solutions of various PCDD and PCDF isomers and mixtures thereof are prepared in a glovebox, using weighed quantities of the authentic isomers. These stock solutions are

contained in appropriate amber bottles and are stored tightly stoppered in a refrigerator. Aliquots of the stock standards are removed for direct use or for subsequent serial dilutions to prepare working standards. These standards must be checked regularly (by comparing instrument response factors for them over a period of time) to ensure that solvent evaporation or other losses have not occurred which would alter the standard concentration. The several standards solutions required are listed below.

a. Standard Mixture A: Prepare a stock solution containing the following isotopically-labelled PCDD and PCDF in isooctane at the indicated concentrations: 0.25 ng/uL $^{13}C_{12}$-2,3,7,8-TCCD, 0.10 ng/uL $^{37}Cl_4$-2,3,7,8-TCDD, 0.10 ng/uL $^{37}Cl_4$-2,3,7,8-TCDF, and 0.25 ng $^{13}C_{12}$-OCDD. Portions of this isomer mixture are added to all samples prior to analyses and serve as internal standards for use in quantitation. Recovery of these standards is also used to guage the overall efficacy of the analytical procedures. (Other isotopically-labelled PCDD/PCDF internal standards may also be used if these are available).

b. Standard B: Prepare a stock solution containing 0.05 ng of $^{13}C_{12}$-1,2,3,4-TCDD/uL dodecane. This standard is coinjected with aliquots of the final sample extract to reliably estimate the recovery of the TCDD internal standard.

c. Standard Mixture C: Prepare a stock solution containing 5 ng/uL of isooctane of each of the following PCDD and PCDF: 2,3,7,8-TCDD; 2,3,7,8-TCDF; 1,2,3,7,8-PeCDD; 2,3,4,7,8-PeCDF; 1,2,3,4,7,8-HxCDD; 1,2,3,6,7,8-HxCDF; 1,2,3,4,6,7,8-HpCDD;

1,2,3,4,6,7,8-HpCDF, OCDD and OCDF. This isomer mixture is used to determine GC-MS response factors for representative isomers of each of the penta-, hexa-, hepta-, and octachlorinated groups of CDDs and CDFs.

d. Standard Mixture D: Prepare a stock solution containing 50 pg/uL of isooctane of each of the following TCDD isomers: 1,3,6,8-TCDD; 1,2,3,7-TCDD; 1,2,3,9-TCDD; 2,3,7,8-TCDD; and 1,2,8,9-TCDD. Two of the isomers in this mixture are used to define the gas chromatographic retention time window for TCDDs (1,3,6,8-TCDD is the first eluting TCDD isomer and 1,2,8,9-TCDD is the last eluting TCDD isomer on the DB-5 GC column). The remaining isomers serve to demonstrate that the 2,3,7,8-TCDD isomer is resolved from the other nearest eluting TCDD isomers, and that the column therefore yields quantitative data for the 2,3,7,8-TCDD isomer alone.

2. <u>PCB</u>

Stock standard solutions of various PCB isomers and mixtures thereof are prepared in a blovebox, using weighed quantities of the authentic isomers. These stock solutions are contained in appropriate amber bottles and are stored tightly stoppered in a refrigerator. Aliquots of the stock standards are removed for direct use or for subsequent serial dilutions to prepare working standards. These standards must be checked regularly (by comparing instrument response factors for them over a period of time) to ensure that solvent evaporation or other losses have not occurred which would alter the standard concentration. The several standards solutions utilized are listed below.

PCB Calibration Standards Employed: These were obtained from Ultra Scientific, Hope, Rhode Island (native PCBs), and from KOR Isotopes, Cambridge, Mass. (labelled PCB). The PCB standards utilize an isomer from each PCB class to determine response factors and elution time windows.

a. Low Level PCB Standard: Prepare a stock solution containing the following native PCBs at 1 ng/uL isooctane: 2-chlorobiphenyl, 3,3'-dichlorobiphenyl, 2,3,5-trichlorobiphenyl, 2,2',3,3'-tetrachlorobiphenyl, 2,3,3',4,4'-pentachlorobiphenyl, 2,2',4,4',5,5'-hexachlorobiphenyl, 2,2',3,4,4',5,5'-heptachlorobiphenyl, 2,2,3,3',4,4',5,5'-octachlorobiphenyl, 2,2',3,3',4,4',-5,6,6' nonachlorobiphenyl, and decachlorobiphenyl and also internal standard at 2 ng/mL: d_6-3,3',4,4'-tetrachlorobiphenyl.

b. Medium Level PCB Standard: Prepare a stock solution containing the native PCBs listed above at 5 ng/uL and the internal standard at 2 ng/uL isooctane.

c. High Level PCB Standard: Prepare a stock solution containing the native PCBs listed above at 25 ng/uL and the internal standard at 2 ng/uL isooctane.

d. Internal Standard PCB MIxture: Prepare a stock solution containing 1 ng of d_6-3,3',4,4'-tetrachorobiphenyl/uL isooctane. This isomer is added to all samples prior to analyses and serves as an internal standard for use in quantitation. Recovery of this standard is also used to gauge the overall efficacy of the analytical procedure.

E. Analysis of Sample Extracts for PCDD and PCDF Using Gas Chromatography-Mass Spectrometry (GC-MS).

Sample extracts prepared by the procedures described in

the foregoing are analyzed by GC-MS utilizing the following instrumental parameters. Typically, 1 to 5 uL portions of the extract are injected into the GC. Sample extracts are initially analyzed using the DB-5 capillary GC column to obtain data on the concentrations of total tetra- through octa-CDDs and CDFs, and on 2,3,7,8-TCDD. If tetra-CDFs are detected in this analysis, then another aliquot of the sample may be analyzed in a separate run, using the DB-225 column,if more definitive data on the concentration of 2,3,7,8-TCDF is desired.

1. Gas Chromatograph: Carlo Erba 4200 or Varian 3740

a. Injector: Configured for capillary column, splitless/split injection (split flow on 60 seconds following injection), injector temperature, 250° C.

b. Carrier gas: Hydrogen, 30 lb head pressure.

c. Capillary Column: For total tetra- through octa-CDDs/CDFs and 2,3,7,8-TCDD, 60 M x 0.25 mm I.D. fused silica DB-5; temperature, programmed, see Table 1 for temperature program. Capillary Column 2 (Optional): For 2,3,7,8-TCDF only, 30 M x 0.25 mm I.D. fused silica DB-225, temperature programmed (180° for 1 min., then increase from 180° to 240° @ 5° C/min, hold at 240° for 1 min)

d. Interface Temperature: 250° C

2. Mass Spectrometer: Kratos MS-30 or Kratos MS-25

a. Ionization Mode: Electron impact (70 eV)

b. Static Resolution: 1:600 (10% valley) or 1:10,000 depending upon requirements. Usually the sample extracts are initially analyzed using low resolution MS, then if PCDD/PCDF are

detected, it is desirable to analyze a second portion of the sample extract using high resolution MS.

c. Source Temperature: 250° C

d. Accelerating Voltage: 2KV or 4KV, depending upon instrument

e. Ions Monitored: Computer controlled Selected Ion Monitoring, See Table 1 for list of ion masses monitored and time intervals during which ions characteristic of each class of PCDD and PCDF are monitored.

3. Calibration Procedures:

a. Calibrating the MS Mass Scale: Perfluorokerosene, decafluorotriphenyl phosphine, or any other accepted mass marker compound must be introduced into the MS, in order to calibrate the mass scale through at least m/z 500. The procedures specified by the manufacturer for the particular MS instrument used are to be employed for this purpose. The mass calibration should be rechecked at least at 8 hr. operating intervals.

b. Table 1 shows the GC temperature program typically used to resolve each chlorinated class of CDDs and CDFs from the other chlorinated classes, and indicates the corresponding time intervals during which ions indicative of each chlorinated class are monitored by the MS. This temperature program and ion monitoring time cycle must be established by each analyst for the particular instrumentation used by injecting aliquots of Standard Mixtures C and D. It may be necessary to adjust the temperature program and ion monitoring cycles slightly based on the observations from analysis of these mixtures.

c. Checking GC Column Resolution for 2,3,7,8-TCDD and 2,3,7,8-TCDF: Utilize the column resolution TCDD and TCDF isomer mixtures (Standard Mixture D), containing 50 pg/ uL, respectively of the appropriate TCDD and TCDF isomers to verify that 2,3,7,8-TCDD and 2,3,7,8-TCDF are separated from the other TCDD and TCDF isomers, respectively. A 20% valley or less must be obtained between the mass chromatographic peak observed for 2,3,7,8-TCDD and adjacent peaks arising from other TCDD isomers and similar separation of 2,3,7,8-TCDF from other neighboring TCDFs is required. Analyze the column performance standard using the instrumental parameters specified above and in Table 1. The column performance evaluation must be performed each time a new column is installed in the gas chromatograph, and at least once during each 8 hour operating period. Providing that the same column is employed for a period of time, its performance can also be gauged by noting the peak width (at 1/2 peak height) for 2,3,7,8-TCDD or for 2,3,7,8-TCDF. If this peak width is observed to broaden by 20% or more as compared to the usual width for satisfactory operation, then the column resolution is suspect and must be checked. If the column resolution is found to be insufficient to resolve 2,3,7,8-TCDD and 2,3,7,8-TCDF from their neighboring TCDD and TCDF isomers, respectively, (as measured on the two different columns used for resolving these two isomers), then a new DB-5 column must be installed.

d. Calibration of the GC-MS-DS system to accomplish quantitative analysis of 2,3,7,8-TCDD and 2,3,7,8-TCDF, and semi-quantitative analysis of the total tetra- through octa-CDDs and

CDFs contained in the sample extract is accomplished by analyzing a series of at least three working calibration standards. Each of these standards is prepared to contain the same concentration of each of the stable-isotopically labelled internal standards used here (Standard Mixture A) but a different concentration of native CDDs/CDFs (Standard Mixture C). Typically, mixtures will be prepared so that the ratio of native PCDD and PCDF to isotopically-labelled PCDD/PCDF will be on the order of 0.1, 0.5 and 1.0 in the three working calibration mixtures. The actual concentrations of both native and isotopically-labelled PCDD and PCDF in the working calibration standards will be selected by the analyst on the basis of the concentrations to be measured in the actual sample extracts. At the time when aliquots of each of the standards are injected (and also when injecting aliquots of actual sample extracts) an aliquot of a standard containing typically 0.1 ng of $^{13}C_{12}$-1,2,3,4-TCDD may be drawn into the micro syringe containing the calibration solution described above (or the sample extract) and is co-injected along with the sample extract in order to obtain data permitting calculation of the percent recovery of the $^{13}C_{12}$-2,3,7,8-TCDD in internal standard. Equations for calculating relative response factors from the calibration data derived from the calibration standard analyses, and for calculating the recovery of the $^{13}C_{12}$-2,3,7,8-TCDD and the other isotopically-labelled PCDD and PCDF, and the concentration of native PCDD/PCDF in the sample (from the extract analysis) are summarized below. In these calculations, as can be seen, 2,3,7,8-TCDD is employed as the illustrative model.

However, the calculations for each of the other native dioxins and furans in the sample analyzed are accomplished in an analogous manner. It should be noted that in view of the fact that stable isotopically-labelled internal standards corresponding to each tetra- through octachlorinated class were not used here (owing to limited availability) the following approach is adopted: For quantitation of tetrachlorinated dibenzofurans $^{13}C_{12}$-2,3,7,8-TCDF is used as the internal standard. For quantitation of tetrachlorodibenzo-p-dioxins, $^{13}C_{12}$-2,3,7,8-TCDD is used as the internal standard. For quantitation of PeCDD, HxCDD, PeCDF, and HxCDF, the stable-isotopically labelled TCDD and TCDF internal standards are used. For quantitation of HpCDD, OCDD, and HpCDF, OCDF, the isotopically-labelled OCDD is used. Inherent in this approach is the assumption that the response factors for each of the isomers of each chlorinated class are the same, and in the case of the penta-, hexa-, and hepta-CDDs and CDFs, the assumption is made that the responses for these three classes are equivalent to those for the tetra-isomers and the octa-isomers, respectively. These limitations, coupled with the fact that the method described here cannot be demonstrated to resolve all PCDD and PCDF of a given chlorinated class into uniquely separated components, dictate that the data reported for the concentrations of total tetra-, penta-, hexa-, and hepta-CDDs and CDFs are semi-quantitative.

4. Equations for Calculating Response Factors, Concentrations of PCDD/PCDF, and Recoveries of Internal Standards.

a. Equation 1: Response Factor (RRF) for native, 2,3,7,8-TCDD using $^{13}C_{12}$-2,3,7,8-TCDD as an internal standard.

$$RRF_d = (A_s C_{is} / A_{is} C_s)$$

where: A_s = SIM response for 2,3,7,8-TCDD ion at m/z 320 + 322

A_{is} = SIM response for $^{13}C_{12}$-2,3,7,8-TCDD internal standard ion at m/z 332

C_{is} = Concentration of the internal standard (pg/uL)

C_s = Concentration of the 2,3,7,8-TCDD (pg/uL)

b. Equation 2: Response Factor (RRF) for $^{37}C_4$-2,3,7,8-TCDD, the co-injected external standard.

$$RRF = (A_{is} C_{es} / A_{es} C_{is})$$

where: A_{is} = SIM response for $^{37}Cl_{12}$-2,3,7,8-TCDD internal standard ion at m/z 322

A_{es} = SIM response for $^{37}C_4$-2,3,7,8-TCDD external standard at m/z 328 - 0.009 (SIM response for native 2,3,7,8-TCDD at m/z 322)

C_{is} = Concentration of the internal standard (pg/uL)

C_{es} = Concentration of the 2,3,7,8-TCDD (pg/uL.)

c. Equation 3: Calculation of concentration of native 2,3,7,8-TCDD using $^{13}C_{12}$-2,3,7,8-TCDD as internal standard

Concentration, p g./g. = $(A_s)(I_s)/(A_{is})(RRF_d)(W)$

where: A_s = SIM response for 2,3,7,8-TCDD ion at m/z 320 = 322

A_{is} = SIM response for the $^{13}C_{12}$-2,3,7,8-TCDD internal standard ion at m/z 332

I_s = Amount of internal standard added to each sample (pg.)

W = Weight of soil or waste in grams

RRF_d = Relative response factor from Equation 1

d. Equation 4: Calculation of % recovery of $^{13}C_{12}$-2,3,7,8-TCDD internal standard

% Recovery = $100(A_{is})(E_s)/(A_{es})(I_i)(RRF_f)$

A_{is} = SIM response for $^{13}C_{12}$-2,3,7,8-TCDD internal standard ion at m/z 332

A_{es} = SIM response for $^{37}Cl_4$-2,3,7,8-TCDD external standard ion at m/z 328 - 0.009 (SIM Response for native 2,3,7,8-TCDD at m/z 322)

E_s = Amount of $^{37}Cl_4$-2,3,7,8-TCDD external standard co-injected with sample extract

I_i = Theoretical amount of $^{13}C_{12}$-2,3,7,8-TCDD internal standard in injection

RRF_f = Relative response factor from Equation 2

5. Criteria for Qualitative Identification of PCDD/PCDF Are Specified Below.

a. Mass Spectral responses must be observed at both the molecular and fragment ion masses corresponding to the ions indicative of each chlorinated class of CDDs/CDFs identified (see Table 1) and intensities of these ions must maximize essentially

simultaneously (within ± 1 second). In addition, the chromatographic retention times observed for each CDD/CDF signal must be correct relative to the appropriate stable-isotopically labelled internal standard.

b. The ratio of the intensity of the molecular ion $[M]^+$ signals to that of the $[M+2]^+$ signal must be within ±20% of the theoretically expected ratio (for example, 0.77 in the case of TCDD; therefore, the acceptable range for this ratio is 0.62 to 0.92).

c. The intensities of the ion signals are considered to be detectable if each exceeds the baseline noise by a factor of at least 2.5:1. The ion intensities are considered to be quantitatively measurable if each ion intensity exceeds the baseline noise by a factor of at least 5:1*.

d. For reliable detection and quantitation of PCDF it is also necessary to monitor signals arising from chlorinated diphenyl ethers which, if present could give rise to fragment ions yielding ion masses identical to those monitored as indicators of the CDFs. Accordingly, in Table 1, appropriate chlorinated diphenyl ether masses are specified which must be monitored simultaneously with the CDF ion-masses. Only when the response for the diphenyl ether ion mass is not detected at the

---

* In practice, the analyst can estimate the baseline noise by measuring the extention of the baseline immediately prior to each of the two mass chromatographic peaks attributed to a given CDD or CDF. Spurious signals may arise either from electronic noise or from other organic compounds. Since it may be desirable to evaluate the judgement of the analyst in this respect, copies of original mass chromatograms must be included in the report of analytical results.

same time as the PCDF ion mass can the signal obtained for an apparent PCDF be considered unique.

e. Semi-Quantitative measurement of the concentration of the congeners in a chlorinated class using the methods described herein is based on the assumption that all of the congeners are identical to the calibration standards employed in terms of their respective chemical and separation properties and in terms of their respective gas chromatographic and mass spectrometric responses. Using these assumptions, for example, the $^{13}C_{12}$-2,3,7,8-TCDD internal standard is utilized as the internal calibration standard for all of the 22 TCDD isomers or congeners. Furthermore, the concentration of the TCDDs present in a sample extract is determined by calculating, on the basis of the standard procedure outlined above, the concentration of each TCDD isomer peak (or peaks for co-eluting isomers) and these individual concentrations are subsequently summed to obtain the concentration of "total" TCDD.

f. Frequently, during the analysis of actual sample extracts, extraneous compounds which are present in the extract (those organic compounds not completely removed during the clean-up phase of the analysis) can cause changes in the liquid and gas chromatographic elution characteristics of the PCDD/PCDF (typically retention times for the PCDD/PCDF are prolonged). Such extraneous organic compounds, when introduced into the mass spectrometer source may also result in a decrease in the sensitivity of the MS because of suppression of ionization, and other affects such as charge transfer phenomena. The shifts in

chromatographic retention times are usually general shifts, that is, the relative retention times for the PCDD/PCDF are not changed, although the entire elution time scale is prolonged. The analyst's intervention in the GC-MS operating sequence can correct for the lengthened GC retention times which are sometimes observed due to the presence of extraneous organics in the sample extract. For example, using the program outlined in Table 1, if the retention time observed for 2,3,7,8-TCDD (which normally is 19.5 minutes) is lengthened by 30 seconds or more, appropriate adjustments in the programming sequence outlined in Table 1 can be made, that is, each selected ion-monitoring program is delayed by a length of time proportionate to the lengthening of the retention time for the 2,3,7,8-TCDD isomer. In the case of ionization suppression, this phenomenon is inherently counteracted by the internal standard approach. However, if loss of sensitivity due to ionization suppression is severe, additional clean-up of the sample extract may be required in order to achieve the desired detection limits

F. Quality Assurance/Quality Control

Quality assurance and quality control are ensured by any or all of the following provisions, depending upon the requirements of the analysis.

1. Each sample analyzed is spiked with stable isotopically labelled internal standards, prior to extraction and analysis. Recoveries obtained for each of these standards should typically be in the range from 60-90%. Since these compounds are used as true internal standards however, lower recoveries do not

necessarily invalidate the analytical results for native PCDD/PCDF, but may result in higher detection limits than are desired.

2. Processing and analysis of at least one method blank sample is generally accomplished for each set of samples (a set being defined as 20 samples or less).

3. It is desirable to analyze at least one sample spiked with representative native PCDD/PCDF for each set of 20 or fewer samples. The results of this analysis provide an indication of the efficacy of the entire analytical procedure. The results of this analysis will be considered acceptable if the detected concentration of each of the native PCDD/PCDF added to the sample is within ±50% of the known concentration. (The appropriate native isomers to be added here are those indicated in Standard Mixture F.)

4. At least one of the samples analyzed out of each set (of 20 samples or less) is usually analyzed in duplicate and the results of the duplicate analysis are included in the report of data.

G. Analysis of Sample Extracts for PCB Using Gas Chromatography-Mass Spectrometry (GC-MS)

Sample extracts prepared by the procedures described in Sections A, B, and C are analyzed for PCB by GC-MS utilizing the following instrumental parameters. Ten to twenty percent of the total sample extract is injected into the GC. Sample extracts are analyzed using the DB-5 capillary GC column to obtain data on the concentrations of total mono- through deca-CB.

1. Gas Chromatograph: Carlo Erba 4200 or Varian 3740

a. Injector: Configured for capillary column, splitless/split injection (split flow on 60 seconds following injection): injector temperature, 300° C.

b. Carrier gas: Hydrogen, 30 lb. head pressure.

c. Capillary Column: For total mono-through deca-chlorinated biphenyls, 60 M x 0.25 mm ID fused silica DB-5; temperature, programmed, see Table 2 for temperature program.

d. Interface Temperature: 250° C

2. Mass spectrometer: Kratos MS-30 or Kratos MS-25

a. Ionization Mode: Electron impact (70 eV)

b. Static Resolution: 1:600 (10% valley) or 1:10,000 depending upon requirements.

c. Source Temperature: 250° C

d. Accelerating Voltage: 2KV or 4KV, depending upon instrument.

e. Ions Monitored: Computer controlled Selected ion Monitoring, See Table 2 for list of ion masses monitored.

3. Calibration Procedures:

a. Calibrating the MS Mass Scale: Perfluorokerosene is introduced into the MS, in order to calibrate the mass scale through m/z 500. The mass calibration is rechecked at least at 8 hr. operating intervals.

b. Table 2 shows the GC temperature program typically used to resolve each chlorinated class of PCBs from the other chlorinated classes, and indicates the corresponding time intervals during which ions are monitored by the MS. This

temperature program and ion monitoring time cycle is established by injecting aliquots of Standard Mixtures of PCBs. It is sometimes necessary to adjust the temperature program and ion monitoring cycles slightly based on the observations from analysis of these mixtures.

c. Calibration Standards for mono-deca CB consist of the solutions described in section D.2.

4. Quality Assurance/Quality Control for PCB Analyses

Quality assurance and quality control are ensured by following provisions.

a. Each sample analyzed by GC-MS is spiked with stable isotopically labelled internal standards, prior to extraction and analysis. Recoveries obtained for each of these standards should typically be in the range from 60-90%. since these compounds are used as true internal standards, however, lower recoveries do not necessarily invalidate the analytical results for native PCBs, but may result in higher detection limits than are desired.

b. Processing and analysis of at least one method blank sample is accomplished for each set of samples (a set being defined as 12 samples or less).

c. It is desirable to analyze at least one sample spiked with representative native PCBs for each set of 20 or fewer samples. The result of this analysis provides an indication of the efficacy of the entire analytical procedure. the results of this analysis will be considered acceptable if the detected concentration of the native PCBs are within ±50% of the known spike concentration.

TABLE 1. Sequence of Operations in GC-MS-DS Quantitation of PCDD/CPDF in Extracts of Environmental Samples

Elapsed Time (min)	Event	GC Column Temperature (°C)	Temperature Program Rate (°C/min)	Ions Monitored by Mass Spectrometer (m/z)	Identity of Fragment Ion	Compounds Monitored	Approximate Theoretical Ratio of [M]+:[M+2]+
0.00	Injection, splitless	190					
1.00	Turn on split valve	190					
1.00	Begin temp. program to 220 C	190	5				
6.00	Open column flow to mass spectrometer	215	5				
7.00	Column temperature hold	220					
14.00	Start Tetra Program; sweep = 350 ppm; time/mass = 0.08 sec.			240.938	[M-COCl]+	TCDF	
				258.930	[M-COCl]+	TCDD	
				303.902	[M]+	TCDF	0.77
				305.899	[M+2]+	TCDF	
				311.890	[M]+	$^{37}Cl_4$-TCDF	
				319.897	[M]+	TCDD	0.77
				321.894	[M+2]+	TCDD	
				327.885	[M]+	$^{37}Cl_4$-TCDD	
				331.937	[M]+	$^{13}C_{12}$-TCDD	
				373.840	[M]+	HxDPE(a)	
22.00	Stop Tetra Program	220					
22.50	Start Penta Program; sweep = 350 ppm; time/mass = 0.12 sec.			274.899	[M-COCl]+	PeCDF	
				290.894	[M-COCl]+	PeCDD	
				337.863	[M]+	PeCDF	1.54
23.00	Begin temp. program to 235	220	5	339.860	[M+2]+	PeCDF	
26.00	Column Temperature hold	235		353.858	[M]+	PeCDD	1.54
				365.898	[M]+	$^{13}C_{12}$-PeCDD	
				355.855	[M+2]+	PeCDD	
				407.801	[M]+	HpDPE (a)	
32.00	Stop Penta Program						

01.Pro

TABLE 1. Sequence of Operations in GC-MS-DS Quantitation of PCDD/PCDF in Extracts of Environmental Samples - Continued

Elapsed Time (min)	Event	GC Column Temperature (°C)	Temperature Program Rate (°C/min)	Ions Monitored by Mass Spectrometer (m/z)	Identity of Fragment Ion	Compounds Monitored	Approximate Theoretical Ratio of [M]+:[M+2]+
32.50	Start Hexa Program; sweep = 350 ppm; time/mass = 0.20 sec.	235		310.857	[M-COCl]+	HxCDF	
				326.852	[M-COCl]+	HxCDD	
				373.821	[M]+	HxCDF	1.23
				375.821	[M+2]+	HxCDF	
				389.816	[M]+	HxCDD	1.23
				411.856	[M]+	$^{13}C_{12}$-HxCDD	
				391.813	[M+2]+	HxCDD	
				443.759	[M]+	ODPE (a)	
33.00	Begin temp. program to 250 C	235	5				
36.00	Column temperature hold	250					
42.50	Stop Hexa Program						
43.00	Start Hepta Program; sweep = 350 ppm; time/mass = 0.30 sec.	250		344.818	[M-COCl]+	HpCDF	
				360.813	[M-COCl]+	HpCDD	
				407.782	[M]+	HpCDF	1.03
				409.779	[M+2]+	HpCDF	
				423.777	[M]+	HpCDD	1.03
				425.774	[M+2]+	HpCDD	
				435.817	[M]+	$^{13}C_{12}$-HpCDD	
				477.720	[M+]	NDPE (a)	
53.00	Stop Hepta Program	250					
53.50	Start Octa Program; sweep = 350 ppm; time/mass = 0.30 sec.	250		378.768	[M-COCl]+	OCDF	
				394.774	[M-COCl]+	OCDD	
				441.732	[M]+	OCDF	0.88
54.00	Begin temp. program to 270	250	5	443.740	[M+2]+	OCDF	
58.00	Column temperature hold	270					
				457.738	[M]+	OCDD	0.88
				459.735	[M+2]+	OCDD	

TABLE 1. Sequence of Operations in GC-MS-DS Quantitation of PCDD/PCDF in Extracts of Environmental Samples - Continued

Elapsed Time (min)	Event	GC Column Temperature (°C)	Temperature Program Rate (°C/min)	Ions Monitored by Mass Spectrometer (m/z)	Identity of Fragment Ion	Compounds Monitored	Approximate Theoretical Ratio of [M]+:[M+2]+
				471.776	[M+2]+	$^{13}C_{12}$-OCDD	
				511.681	[M]+	DDPE (a)	
65.00	Stop Octa Program						
65.00	Begin temp. program to 300	270	5				
71.00	Column temperature hold	300					
75.00	Cool Column to 190						

(a) HxDFE, HpDPE, ODPE, NDPE, DDPE are abbreviations which designative (respective) hexachloro-, heptachloro-, octachloro-, nonachloro-, and decachlorodiphenyl ethers.

TABLE 2

Sequence of Operations in GC-MS-DS Quantitation of PCBs in Sample Extracts

Elapsed Time (min)	Event	GC Column Temperature (°C)	Temperature Program/Rate (°C/min)	Ions Monitored by Mass Spectrometer (m/z)	PCB Monitored
0.00	Injection, splitless	190			
1.50	Split valve on	190			
7.00	Open column flow to mass spectrometer; begin temp. program to 270°C	190			
	Start MID program: sweep width = 100; time on ion mass = 0.1 sec	190	5	188.039	Mono-
	Time on d_6-tetra-CBP ion masses = 0.1 sec			190.036	Mono-
				222.000	Di-
				223.997	Di-
				255.961	Tri-
				257.958	Tri-
				289.922	Tetra-
				291.919	Tetra-
				325.880	Penta-
				327.877	Penta-
				359.841	Hexa-
				361.839	Hexa-
				393.802	Hepta-
				395.800	Hepta-
				427.763	Octa-
				429.761	Octa-
				463.722	Nona-
				465.719	Nona-
				497.683	Deca-
				499.680	Deca-
				295.922	d_6-Tetra
				297.919	d_6-Tetra
23.00	Hold column temp	270			
35.00	Stop MID program	270			
40.00	Cool column to initial temp.				

ATTACHMENT D

INTRALABORATORY SAMPLE TRACKING FORMS FOR BREHM LABORATORY ANALYSES OF U.S. COAST GUARD EXTRACTS OF SEDIMENT SAMPLES AND OF SEDIMENT SAMPLES RECEIVED FROM NUS CORP. FOR PCDD AND PCDF

SAMPLE TRACKING FORM

Project: EPA/US. Coast Guard

Lab Prep Notebook No. 126
Pages 10-11
Lab Analysis Notebook No. 39
Pages 48

WSU Sample #	Customer I.D.	ASAM Line #	Sample Type	TOTAL Sample Weight	INTERNAL STANDARD SPIKE LEVELS		Date Prepared	Date An
USC 6-1	27	2	HEXANE ACETONE EXTRACT	20g	2,3,7,8-TCDD		4/28/86	4-29-86
USC 6-2	9(B,C)	3	HEXANE/ACETONE EXTRACT	20g	1,2,3,7,8-PCDD		4/28/86	4-29-86
USC 6-OB	—	6	—	—	$^{13}C_{12}$ 1,2,3,4,7,8-HxCDD	1.0 ng	4/28/86	4-29-86
					1,2,3,4,6,7,8-HpCDD			
					OCDD			
		+10 -3						
					$^{37}Cl_4$ 2,3,7,8-TCDD			
					2,3,7,8-TCDF	0.4 ng		

Sample Prep Accomplished By: Joe McDaniel, Walter Hanes 4/28/86

Analysis Accomplished By: C. Hanes

Spiking Standard: Ful 129038-2A

SAMPLE TRACKING FORM

Project: EPA/US. Coast Guard

Lab Prep Notebook No. 126
Pages 10-11
Lab Analysis Notebook No.
Pages

WSU Sample #	Customer I.D.	Sample Type	TOTAL Sample Weight	INTERNAL STANDARD SPIKE LEVELS		Date Prepared	Date Ana
USC 6-1	27	HEXANE ACETONE EXTRACT	20%			4/28/86	
USC 6-2	9(B,C)	HEXANE/ACETONE EXTRACT	20%			4/28/86	
USC 6-OB	—	—	—	$^{13}C_{12}$	2,3,7,8-TCDD; 1,2,3,7,8-PCDD; 1,2,3,4,7,8-HxCDD; 1,2,3,4,6,7,8-HpCDD; OCDD — 1.0 ng	4/28/86	
				$^{37}Cl_4$	2,3,7,8-TCDD; 2,3,7,8-TCDF — 0.4 ng		

Sample Prep Accomplished By: Joe McDaniel, Marlee Haines 4/28/86

Spiking Standard: Eul 129038-1A

Analysis Accomplished By:

SUMMARY OF CDD/CDF ANALYTICAL REQUIREMENTS

Date requirements established: 4/25/86

Project name: EPA/Coast Guard P.O. number : _____ WSU sample number(s): 45C6-12

Sample type : Hexane/Acetone Extract Hazard level : _____ Total number of sample types and forms/project: 1

Samples to be prepped for analysis by: GC/MS: __ 015 MS-25 __ New MS-25 __ 015 MS-30 __ 067 MS-30 GC : __ FI __ EC

Required analyses: X 2378 TCDD(specific) __ 2378 TCDF(specific) __ 2378 TCDD/TCDF (not 2378 TCDF specific) __ TCDD isomers __ TCDF isomers __ TCDD/F-HxCDD/F X TCDD/F-OCDD/F

Detect. limit : Region1 2378-TCDD Target 20pg Required ____ Region2 _____ Target ____ Required ____

Sample Prep Size : 0.8-2g (20% of sample)

Internal Standard Name	Internal Standard Number	2378 37CL4 TCDD	2378 6C12 TCDD	2378 13C12 TCDD	2378 37CL4 TCDF	2378 13C12 TCDF	12378 13C12 PCDD	12378 13C12 PCDF	123478 13C12 HxCDD	123478 13C12 HxCDF	1234678 13C12 HpCDD	1234678 13C12 HpCDF	13C12 OCDD	13C12 OCDF
		Concentration of Internal Standard Component (ng/microliter)												
TCDD only	102094-1	-	-	0.025	-	-	-	-	-	-	-	-	-	-
TCDD/TCDF	EPA 1833-1	0.100	-	0.250	0.100	-	-	-	-	-	-	-	-	-
Ext. Std.	102092-11	-	0.050	-	-	-	-	-	-	-	-	-	-	-
T-O CDD/F	102110-1	0.100	-	0.250	0.100	0.100	-	-	-	-	-	-	0.250	-
New T-O		0.050	-	0.125	0.050		0.125		0.125		0.125	-	0.125	-
NIEHS	102096-5	-	-	-	-	0.050	-	0.050	-	0.050	-	-	-	-
Total ng Int Std added		0.4		1	0.4		1		1		1		1	

Internal Std # D9038-1A Microliters Internal Std/sample 8 Microliters External Std/sample ____

Native Std Name	Native Std No.	TCDD	TCDF	PCDD	23467 PCDF	12478 PCDF	23478 PCDF	HxCDD	HxCDF	HpCDD	HpCDF	OCDD	OCDF
		Concentration of Native Standard Component (ng/microliter)											
TCDD only	102094-2	0.005	-	-	-	-	-	-	-	-	-	-	-
TCDD/TCDF	EPA 1835-4	0.020	0.020	-	-	-	-	-	-	-	-	-	-
T-O CDD/F	EPA 1840-3	0.050	0.050	0.050	(0.050)	-	-	0.050	0.050	0.050	0.050	0.050	0.050
NIEHS	102096-4	-	0.026	-	0.026	0.026	0.026	-	0.026	-	-	-	-
Total ng Native added													

Native spike std # — Microliters Native Std/sample —

QA/QC : Lab blanks 1 per 2 samples Matrix blanks — per ____ samples Native spikes — per ____ samples Duplicates — per ____ samples

List sample numbers and requirements (if specified by contract) for QA/QC: _____

Other(detect. limit verification,ect): _____

Special Prep Requirements: __ Digestion with _____ Other digestion conditions _____ X Bottle extraction Soxhlet __ Other(specify) Florisil Alumina Clean-up

GC/MS specifics: Column: X DB5(60M) (no 2378 specific TCDF) __ DB225(30M) 2378 TCDF specific only Other: _____

Stds required daily: X Pemix __ Lowest std curve member __ Detection limit verification

Data requirements: X Original chromatograms __ Data must match previous format (describe in detail) _____

SAMPLE TRACKING FORM

Project: EPA / Bedford Mass

Lab Prep Notebook No. 126
Pages 11
Lab Analysis Notebook No. 39
Pages 48

WSU Sample #	Customer I.D.	Sample Type	Sample Weight	INTERNAL STANDARD SPIKE LEVELS		Date Prepared	Date Ana
NBCS-3	138555	Sediment Core 3"-8"	15.63	2,3,7,8-TCDD		4/29/86	* 4-27-86
NBCS-0-B	-	Lab Blank	-	12,3,7,8-PeCDD		4/29/86	4-27-86
			$^{13}C_{12}$	1,2,3,4,7,8-HxCDD	1.0 ng		
				1,2,3,4,6,7,8-HpCDD			* NERION 5.5
				OCDD			
			$^{37}Cl_4$	2,3,7,8-TCDD	0.4 ng		
				2,3,7,8-TCDF			

Sample Prep Accomplished By: J. McDaniel, L. Anderson, D. Martin

Analysis Accomplished By: MS-25 B J. Jewell

Spiking Standard: I.S. 102038-1A/8 ng

SAMPLE TRACKING FORM

Project: EPA / Bedford Mass

Lab Prep Notebook No. 126
Pages 11
Lab Analysis Notebook No.
Pages

WSU Sample #	Customer I.D.	Sample Type	Sample Weight	INTERNAL STANDARD SPIKE LEVELS		Date Prepared	Date Ana
NBCS-3	13855	Sediment Core 3"-8"	15.63	2,3,7,8-TCDD		4/29/86	
NBCS-0-B	-	Lab Blank	-	12,3,7,8-PeCDD		4/29/86	
			$^{13}C_{12}$	1,2,3,4,7,8-HxCDD	1.0 ng		
				1,2,3,4,6,7,8-HpCDD			
				OCDD			
			$^{37}Cl_4$	2,3,7,8-TCDD	0.4 ng		
				2,3,7,8-TCDF			

Sample Prep Accomplished By: J. McDaniel, L. Anderson, D. Nevin

Spiking Standard: E.S. 102038-1A (8 pt)

Analysis Accomplished By:

SUMMARY OF CDD/CDF ANALYTICAL REQUIREMENTS

Date requirements established: _____

Project name: EPA/Bedford Mass P.O. number : _____ WSU sample number(s): NBC5-3

Sample type : Sediment Hazard level : _____ Total number of sample types and forms/project: _____

Samples to be prepped for analysis by: GC/MS: __ 015 MS-25 __ 015 MS-30 __ New MS-25 __ 067 MS-30 GC : __ FI __ EC

Required analyses: / 2378 TCDD(specific) __ 2378 TCDF(specific) __ 2378 TCDD/TCDF (not 2378 TCDF specific) __ TCDD isomers __ TCDF isomers __ TCDD/F-HxCDD/F X TCDD/F-OCDD/F

Detect. limit : Region1 25pg-100 Target ____ Required 1pg Region2 _____ Target ____ Required ____

Sample Prep Size : 20g

Concentration of Internal Standard Component (ng/microliter)

Internal Standard Name	Internal Standard Number	2378 37CL4 TCDD	2378 6C12 TCDD	2378 13C12 TCDD	2378 37CL4 TCDF	2378 13C12 TCDF	12378 13C12 PCDD	12378 13C12 PCDF	123478 13C12 HxCDD	123478 13C12 HxCDF	1234678 13C12 HpCDD	1234678 13C12 HpCDF	13C12 OCDD	13C12 OCDF
TCDD only	102094-1	-	-	0.025	-	-	-	-	-	-	-	-	-	-
TCDD/TCDF	EPA 1833-1	0.100	-	0.250	0.100	-	-	-	-	-	-	-	-	-
Ext. Std.	102092-11	-	0.050	-	-	-	-	-	-	-	-	-	-	-
T-O CDD/F	102110-1	0.100	-	0.250	0.100	0.100	-	-	-	-	-	-	0.250	-
New T-O	129038-1A	0.050	-	0.125	0.050		0.125		0.125		0.125	-	0.125	-
NIEHS	102096-5	-	-	-	-	0.050	-	0.050	-	0.050	-	-	-	-
Total ng Int Std added		0.4		/	0.4		/		/		/		/	

Internal Std # 129038-1A Microliters Internal Std/sample 8 Microliters External Std/sample ____

Concentration of Native Standard Component (ng/microliter)

Native Std Name	Native Std No.	TCDD	TCDF	PCDD	23467 PCDF	12478 PCDF	23478 PCDF	HxCDD	HxCDF	HpCDD	HpCDF	OCDD	OCDF
TCDD only	102094-2	0.005	-	-	-	-	-	-	-	-	-	-	-
TCDD/TCDF	EPA 1835-4	0.020	0.020	-	-	-	-	-	-	-	-	-	-
T-O CDD/F	EPA 1840-3	0.050	0.050	0.050	(0.050)	-	-	0.050	0.050	0.050	0.050	0.050	0.050
NIEHS	102096-4	-	0.026	-	0.026	0.026	0.026	-	0.026	-	-	-	-
Total ng Native added													

Native spike std # — Microliters Native Std/sample —

QA/QC : Lab blanks / per / samples
Matrix blanks — per ____ samples
Native spikes — per ____ samples
Duplicates — per ____ samples

List sample numbers and requirements (if specified by contract) for QA/QC: _____

Other(detect. limit verification,ect): _____

Special Prep Requirements: __ Digestion with _____ Other digestion conditions _____
__ Bottle extraction X Soxhlet Benzene/Acetone
__ Other(specify) _____

GC/MS specifics: Column: X DB5(60M) (no 2378 specific TCDF) __ DB225(30M) 2378 TCDF specific only Other: _____

Stds required daily: X Pemix __ Lowest std curve member __ Detection limit verification

Data requirements: X Original chromatograms
__ Data must match previous format _____
(describe in detail) _____

SAMPLE TRACKING FORM

Project: EPA/New Bedford

CDD, CDF
50%

Lab Prep Notebook No. 126
Pages 12, 14, 16, 17
Lab Analysis Notebook No. 106
Pages 163

WSU Sample #	Customer I.D.	Sample Type	Sample Weight	INTERNAL STANDARD SPIKE LEVELS		Date Prepared	Date Ana
NBC5-1	13858	Sediment Core	11.73	2,3,7,8-TCDD		5/19/86	5-19
NBC5-5	13867	Sediment Core	14.47	1,2,3,7,8-PCDD		5/19/86	5-19
NBC5-OB-B	-	Blank	-	$^{13}C_{12}$ 1,2,3,4,7,8-H_6CDD	1ng	5/19/86	5-20
				1,2,3,4,6,7,8-H_7CDD			
				OCDD			
				$^{37}Cl_4$ 2,3,7,8-TCDD	0.4ng		
				2,3,7,8-TCDF			

Sample Prep Accomplished By: J. Young, D. Navin, J. McDaniel, MS Hames 5/19/86

Analysis Accomplished By: MS-25-B L. Durell

Spiking Standard: 129038-1A / 8µL

SAMPLE TRACKING FORM

Project: EPA/New Bedford

CDD, CDF
50%

Lab Prep Notebook No. 126
Pages 12, 14, 16, 17
Lab Analysis Notebook No.
Pages

WSU Sample #	Customer I.D.	Sample Type	Sample Weight	INTERNAL STANDARD SPIKE LEVELS		Date Prepared	Date Ana
NBC5-1	13858	Sediment Core	11.73	2378-TCDD	1ng	5/19/86	
NBC5-5	13867	Sediment Core	14.47	12378-PCDD		5/19/86	
NBC5-OB-B	-	Blank	-	$^{13}C_{12}$ 123478-HxCDD		5/19/86	
				123678-HpCDD			
				OCDD			
				$^{37}Cl_4$ 2378-TCDD	0.4ng		
				2378-TCDF			

Sample Prep Accomplished By: J. Young, D. Navin, J. McDaniel, MS Homes 5/19/86

Spiking Standard: 129038-1A (8µL)

Analysis Accomplished By:

SUMMARY OF CDD/CDF ANALYTICAL REQUIREMENTS

Date requirements established: _____

ject name: EPA Gulfport Mass P.O. number : _____ ASU sample number(s): NBC5-1,5

ple type : Sediment Hazard level : _____ Total number of sample types and forms/project: _____

ples to be prepped for analysis by: GC/MS: __ 015 MS-25 __ 015 MS-30 __ New MS-25 __ 867 MS-30 GC : __ F1 __ SC

uired analyses: X 2378 TCDD(specific) __ 2378 TCDF(specific) __ 2378 TCDD/TCDF (not 2378 TCDF specific) __ TCDD isomers __ TCDF isomers __ TCDD/F-HxCDD/F X TCDD/F-OCDD/F

ect. limit : Region1 2378-TCDF Target: ____ Required 1 pt Region2 _____ Target ____ Required ____

ple Prep Size : 2 g

Internal Standard Name	Internal Standard Number	2378 37Cl4 TCDD	2378 6C12 TCDD	2378 13C12 TCDD	2378 37Cl4 TCDF	2378 13C12 TCDF	12378 13C12 PCDD	12378 13C12 PCDF	123478 13C12 HxCDD	123478 13C12 HxCDF	1234678 13C12 HpCDD	1234678 13C12 HpCDF	13C12 OCDD	13C12 OCDF
		Concentration of Internal Standard Component (ng/microliter)												
DD only	102094-1	-	-	0.025	-	-	-	-	-	-	-	-	-	-
DD/TCDF	EPA 1833-1	0.100	-	0.250	0.100	-	-	-	-	-	-	-	-	-
t. Std.	102092-11	-	0.050	-	-	-	-	-	-	-	-	-	-	-
D CDD/F	102110-1	0.100	-	0.250	0.100	0.100	-	-	-	-	-	-	0.250	-
w T-O	102038-1A	0.050	-	0.125	0.050		0.125		0.125		0.125	-	0.125	-
NIEHS	102095-5	-	-	-	-	0.050	-	0.050	-	0.050	-	-	-	-
tal ng Int Std added		0.4		1	0.4		1		1		1		1	

nternal Std # 129038-1A Microliters Internal Std/sample 5 µl Microliters External Std/sample ____

Native Std Name	Native Std No.	TCDD	TCDF	PCDD	23467 PCDF	12478 PCDF	23478 PCDF	HxCDD	HxCDF	HpCDD	HpCDF	OCDD	OCDF
		Concentration of Native Standard Component (ng/microliter)											
DD only	102094-2	0.025	-	-	-	-	-	-	-	-	-	-	-
DD/TCDF	EPA 1835-4	0.020	0.020	-	-	-	-	-	-	-	-	-	-
-O CDD/F	EPA 1840-3	0.050	0.050	0.050	(0.050)	-	-	0.050	0.050	0.050	0.050	0.050	0.050
NIEHS	102096-4	-	0.026	-	0.026	0.026	0.026	-	0.026	-	-	-	-
tal ng Native added													

ative spike std # ____ Microliters Native Std/sample ____

A/QC : Lab blanks 1 per 2 samples Matrix blanks 0 per ____ samples Native spikes 0 per ____ samples Duplicates 0 per ____ samples

List sample numbers and requirements (if specified by contract) for QA/QC: _____

Other(detect. limit verification,ect): _____

pecial Prep Requirements: __ Digestion with _____ Other digestion conditions _____ __ Bottle extraction X Soxhlet Benzene/Acetone __ Other(specify) _____

C/MS specifics: Column: X DB5(60M) (no 2378 specific TCDF) __ DB225(32M) 2378 TCDF specific only Other: _____

Stds required daily: X Pemix __ Lowest std curve member __ Detection limit verification

Data requirements: X Original chromatograms __ Data must match previous format (describe in detail) _____

ATTACHMENT E

INTRALABORATORY SAMPLE TRACKING FORMS FOR BREHM LABORATORY ANALYSES OF U.S. COAST GUARD EXTRACTS OF SEDIMENT SAMPLES AND OF SEDIMENT SAMPLES RECEIVED FROM NUS CORP. FOR PCB

SAMPLE TRACKING FORM

Project: EPA/Coast Guard PCB

87 Fraction

Total

Lab Prep Notebook No. 126
Pages 10-11
Lab Analysis Notebook No. 106
Pages 160

INTERNAL STANDARD SPIKE LEVELS decachlorobiphenyl

WSU Sample #	Customer I.D.	Sample Type	Sample Weight		Date Prepared	Date Ana
USC 6-1	37	Hexane Acetone Rinse	20 g	960 ng	5/7/86	5-14-86
USC 6-2	(BC)	Hexane Acetone Rinse	20 g	960 ng	5/7/86	5-14-86
USC 6-0-B	-	Lab Blank	-	960 ng	5/7/86	5-14-86

Sample Prep Accomplished By: John McDaniel, Mark Hare

Analysis Accomplished By: MS-25-B P. Barnett

Spiking Standard: decachlorobiphenyl (6)

SAMPLE TRACKING FORM

Lab Prep Notebook No. 126, 114
Pages 11, 33
Lab Analysis Notebook No. 106
Pages 16, 1

Project: EPA/Bedford, Mass

PCB
8% Fraction

INTERNAL STANDARD SPIKE LEVELS

WSU Sample #	Customer I.D.	Sample Type	Sample Weight	decachlorobiphenyl		Date Prepared	Date Ana
NBCS-3	13855	Sediment Core 3-8"	15.63g	960ng		4/29/86	5/14/86
NBCS-0-C	-	Lab Blank	-	960ng		4/29/86	5/14/86

Sample Prep Accomplished By: T. McDaniel, L. Anderson, D. Navin

Analysis Accomplished By: J. Garrett

Spiking Standard: decachlorobiphenyl

SAMPLE TRACKING FORM

Project: EPA/New Bedford PCBs 8+507

Lab Prep Notebook No. 126
Pages 12, 14, 16, 17
Lab Analysis Notebook No. 106
Pages 165

WSU Sample #	Customer I.D.	Sample Type	Sample Weight	INTERNAL STANDARD SPIKE LEVELS decachlorobiphenyl		Date Prepared	Date Ana
NBC5-1	13858	Sediment Core	11.73	960 ng		5/19/86	5-21
NBC5-5	13867	Sediment Core	14.47	960 ng		5/19/86	5-21
NBC5-OB-B	-	Blank	-	960 ng		5/19/86	5-21

Sample Prep Accomplished By: J. Young, D. Davin, J. McDaniel, M. Hamed 5/19/86

Analysis Accomplished By: R. Hamed

Spiking Standard: decachlorobiphenyl

ATTACHMENT F

TABLES OF DATA

RESULTS OF BREHM LABORATORY ANALYSES OF U.S. COAST GUARD SAMPLE EXTRACTS AND OF SEDIMENTS RECEIVED FROM NUS CORP. FOR PCDD AND PCDF

TABLE 1

Brehm Laboratory, Wright State University, Dayton, Ohio 45435

Results of HRGC-LRMS analyses of samples for TetraChlorinated through OctaChlorinated DIBENZO-p-DIOXINS (CDDs), DIBENZOFURANS (CDFs), 2,3,7,8-TETRA-CDD & 2,3,7,8-TETRA-CDF

Concentrations Found (nanograms per total sample) a.

USCG/EPA Sample I.D. NO	Brehm Lab NO.	2378 Tetra TCDD	%Rec of 13C12 2378 TCDD	%Rec of Surro-gate	Total Tetra CDDs	Total Penta CDDs	Total Hexa CDDs	Total Hepta CDDs	Total Octa CDD	%Rec of 13C12 OCDD	2378 Tetra TCDF	%Rec of 37C14 2378 TCDF	Total Tetra CDFs	Total Penta CDFs	Total Hexa CDFs	Total Hepta CDFs	Total Octa CDF
27	USC 6-1	ND 0.0661	129.6	98.8	ND 0.0322	ND 0.0825	ND 0.150	0.472	0.768	124.0	11.7	135.4	41.7	26.6	11.8	1.02 10 ml	0.929
9 (B,C)	USC 6-2	ND 0.0977	e.	164.9	ND 0.0656	ND 0.143	ND 0.561	0.592	0.968	154.6	5.91	191.2	23.3	33.9	25.9	2.06 5 ml	1.13
Lab Blank	USC6-0B	ND 0.0311	56.5	94.1	ND 0.0480	ND 0.130	ND 0.558	ND 0.192	ND 0.177	40.7	ND 0.0330	55.9	ND 0.0371	ND 0.0770	ND 0.171	ND 0.547	ND 0.349

a. The designation ND indicates "None Detected" in excess of the minimum detectable concentration which is listed directly below the ND designation.

b. Unless otherwise indicated, the concentrations listed for 2,3,7,8 TCDF could include contributions from co-eluting TCDF isomers.

e. The internal standard region failed to yield a value because of chemical interference. The native regions that were to be calculated by the failed internal were calculated by using the nearest non-interfered internal standard.

Extracts

TABLE 2

Brehm Laboratory, Wright State University, Dayton, Ohio 45435

Results of HRGC-LRMS analyses of samples for TetraChlorinated through OctaChlorinated DIBENZO-p-DIOXINS (CDDs), DIBENZOFURANS (CDFs), 2,3,7,8-TETRA-CDD & 2,3,7,8-TETRA-CDF

Concentrations Found (nanograms per gram of sample or parts-per-billion) a.

2 – 4 ppb

NUS/EPA Sample I.D. NO	Brehm Lab NO.	2378 Tetra TCDD	%Rec of 13C12 2378 TCDD	%Rec of Surro-gate	Total Tetra CDDs	Total Penta CDDs	Total Hexa CDDs	Total Hepta CDDs	Total Octa CDD	%Rec of 13C12 OCDD	2378 Tetra TCDF	%Rec of 37C14 2378 TCDF	Total Tetra CDFs	Total Penta CDFs	Total Hexa CDFs	Total Hepta CDFs	Total Octa CDF
13855	NBC5-3	ND 0.0039	55.6	73.4	ND 0.0052	ND 0.0056	0.203	0.675	1.82	88.8	0.474	74.7	4.07	1.54	1.03	0.462	0.428
Lab Blank	NBC5-0-B	ND 0.0007	54.6	90.4	ND 0.0008	ND 0.0020	ND 0.0223	ND 0.0062	0.0265	39.2	ND 0.0003	50.2	ND 0.0004	ND 0.0020	ND 0.0045	ND 0.0243	ND 0.0056
13858	NBC5-1	ND 0.0039	44.5	106.2	ND 0.0031	ND 0.0074	0.352	0.673	2.80	190.3	0.730	73.8	3.63	2.43	1.91	0.628	0.679
13867	NBC5-5	ND 0.0043	54.3	105.9	ND 0.0030	ND 0.0061	0.171	0.272	0.921	60.2	0.406	59.2	2.50	1.64	2.24	0.343	0.301
Lab Blank	NBC5-0B-B	ND 0.0026	32.1	92.4	ND 0.0023	ND 0.0076	ND 0.0102	0.0234	0.0782	26.7	ND 0.0025	27.4	ND 0.0020	ND 0.0038	ND 0.0067	ND 0.0064	ND 0.0203

a. The designation ND indicates "None Detected" in excess of the minimum detectable concentration which is listed directly below the ND designation.

b. Unless otherwise indicated, the concentrations listed for 2,3,7,8 TCDF could include contributions from co-eluting TCDF isomers.

1 - I10 core 0-6"

3 I9 core 3-8"

5 I13 core 6-12

TABLE 3

Brehm Laboratory, Wright State University, Dayton, Ohio 45435

Results of HRGC-LRMS Analyses of Extracts for 2,3,7,8-TETRACHLORODIBENZO-p-DIOXIN

USCG/EPA Sample Number	Aliquot of Total	ng TCDD Meas.	ng TCDD DL	Instr. ID	Date	Time	Ion Int. Ratios 320/322	Ion Int. Ratios 332/334	Sur. Acc.%	Ion Intensities a.b. 320	322	257	328	332	334
27	0.2000	ND	0.0651	MS25B	042986	11:44	0.81		98.5	-1.1E+04	-7.6E+03	-4.5E+04	1.37E+06	1.40E+06	1.72E+06
9 (B,C)	0.2000	ND	0.0977	MS25B	042986	13:28	0.73		164.9	-8.9E+03	-1.3E+04	-1.7E+04	1.42E+06	-3.3E+05	-4.5E+05
Lab Blank	0.2000	ND	0.0311	MS25B	042986	15:10	0.84		94.1	7.02E+03	5.25E+03	2.33E+04	8.60E+05	9.43E+05	1.18E+06

a. The notation used here to designate ion intensities is exponential notation (for example, 7.80E +05) indicates the following: The number preceding E should be multiplied by a factor of 10 raised to the power of the number following E. So, the designation 7.80E+05 indicates 780,000.

b. A negative sign preceding the peak intensity value cited indicates that no response was observed at that m/z which exceeds the noise level by a factor of 2.5 or greater. The number following the negative sign is the observed noise level at that m/z.

TABLE 4

Brehm Laboratory, Wright State University, Dayton, Ohio 45435

Results of HRGC-LRMS Analyses of Extracts for 2,3,7,8-TETRACHLORODIBENZO-p-DIOXIN

NUS/EPA Sample Number	Aliquot Wt. (g)	PPB TCDD Meas.	PPB TCDD DL	Instr. ID	Date	Time	Ion Int. Ratios 320/322	Ion Int. Ratios 332/334	Sur. Acc.%	Ion Intensities a,b. 320	322	257	328	332	334
13855	15.63	ND	0.0039	MS25B	052586	16:07	0.69		79.4	-1.6E+04	-1.2E+04	-2.5E+05	3.79E+05	4.40E+05	5.35E+05
Lab Blank	15.00	ND	0.0007	MS25B	042986	15:51	0.60		90.4	-2.9E+03	-4.6E+03	-1.4E+04	8.16E+05	9.07E+05	1.13E+06
13858	11.73	ND	0.0039	MS25B	051986	14:52	0.85		106.2	-8.5E+03	-1.7E+04	2.25E+05	6.20E+05	6.07E+05	7.29E+05
13867	14.47	ND	0.0043	MS25B	051986	15:31	0.77		105.9	-1.7E+05	-1.5E+04	-2.1E+05	5.19E+05	5.72E+05	7.47E+05
Lab Blank	13.00	ND	0.0025	MS25B	052086	12:19	0.84		92.4	-2.8E+03	-2.8E+03	-5.5E+03	1.78E+05	1.99E+05	2.37E+05

a. The notation used here to designate ion intensities is exponential notation (for example, 7.80E +05) indicates the following: The number preceding E should be multiplied by a factor of 10 raised to the power of the number following E. So, the designation 7.80E+05 indicates 780,000.

b. A negative sign preceding the peak intensity value cited indicates that no response was observed at that m/z which exceeds the noise level by a factor of 2.5 or greater. The number following the negative sign is the observed noise level at that m/z.

TABLE 5

Brehm Laboratory, Wright State University, Dayton, Ohio 45435

Results of HRGC-LRMS Analyses of Extracts for 2,3,7,8-TETRACHLORODIBENZOFURAN

USCG/EPA Sample Number	Aliquot of Total	ng TCDF Meas.	ng TCDF DL	Instr. ID	Date	Time	Ratios 304/306	Ion Intensities a.b. 304	306	241	312
27	0.2000	11.7		MS25B	042986	11:44	0.81	4.58E+06	5.65E+06	1.58E+06	1.48E+06
9 (B,C)	0.2000	5.91		MS25B	042986	13:28	0.82	2.55E+06	3.13E+06	8.17E+05	1.63E+06
Lab Blank	0.2000	ND	0.0330	MS25B	042986	15:10		-5.1E+03	-3.9E+03	-2.2E+04	8.95E+05

a. The notation used here to designate ion intensities is exponential notation (for example, 7.80E +05) indicates the following: The number preceding E should be multiplied by a factor of 10 raised to the power of the number following E. So, the designation 7.80E+05 indicates 780,000.

b. A negative sign preceding the peak intensity value cited indicates that no resoonse was observed at that m/z which exceeds the noise level by a factor of 2.5 or greater. The number following the negative sign is the observed noise level at that m/z.

c. Unless otherwise indicated, the concentrations listed for 2,3,7,8 TCDF could include contributions from co-eluting TCDF isomers.

TABLE 6

Brehm Laboratory, Wright State University, Dayton, Ohio 45435

Results of HRGC-LRMS Analyses of Extracts for 2,3,7,8-TETRACHLORODIBENZOFURAN

NUS/EPA

Sample Number	Aliquot Wt. (g)	PPB TCDF Meas.	PPB TCDF DL	Instr. ID	Date	Time	Ratios 304/306	Ion Intensities a,b. 304	306	241	312
13355	15.53	0.474		MS25B	050586	16:07	0.76	6.00E+06	7.90E+06	1.17E+06	6.34E+05
Lab Blank	15.00	ND	0.0003	MS25B	242986	16:51		-3.9E+03	-2.5E+03	9.13E+05	6.23E+05
13356	11.73	0.730		MS25B	051586	14:53	0.73	1.25E+07	1.33E+07	2.11E+06	9.53E+05
13867	14.47	0.406		MS25B	051986	16:31	0.80	4.85E+06	6.06E+06	1.43E+06	6.31E+05
Lab Blank	13.00	ND	0.0025	MS25B	052086	12:19		-7.7E+03	-3.5E+03	-3.0E+04	1.63E+05

a. The notation used here to designate ion intensities is exponential notation (for example, 7.80E +05) indicates the following: The number preceding E should be multiplied by a factor of 10 raised to the power of the number following E. So, the designation 7.80E+05 indicates 780,000.

b. A negative sign preceding the peak intensity value cited indicates that no response was observed at that m/z which exceeds the noise level by a factor of 2.5 or greater. The number following the negative sign is the observed noise level at that m/z.

c. Unless otherwise indicated, the concentrations listed for 2,3,7,8 TCDF could include contributions from co-eluting TCDF isomers.

TABLE 7

Brehm Laboratory, Wright State University, Dayton, Ohio 45435

GA/GC Result Summary for USCG/EPA

Internal Standard Results for 3 Samples (Average Recovery +- Std Dev)

37Cl4 TCDF	37Cl4 TCDD	13C12 TCDD	1234 TCDD	13C12 OCDD
128.8+/-55.7	119.3+/-32.3	48.7+/-69.5	23.3+/-143.9	106.4+/-46.1

TABLE 8

Brehm Laboratory, Wright State University, Dayton, Ohio 45435

QA/QC Result Summary for USCG/EPA

Internal Standard Results for 5 Samples (Average Recovery +- Std Dev)

37Cl4 TCDF	37Cl4 TCDD	13C12 TCDD	1234 TCDD	13C12 OCDD
57.0+/-17.5	94.9+/-10.2	48.2+/- 5.0	256.6+/-135.2	82.9+/-58.5

TABLE 9

BREHM LABORATORY, WRIGHT STATE UNIVERSITY, DAYTON, OHIO 45435

SUMMARY OF 2,3,7,8-TCDD CALIBRATION DATA GC-MS MS25B

DATE	INSTR ID	SOL ID	MEASURED RF NATIVE	MEASURED RF SURRAGATE	MEAN RF USED NATIVE	MEAN RF USED SURRAGATE	% VALLEY(PE MIX)
04/29/86	MS25B	PEMIX #2					15
04/29/86	MS25B	1840 - 1	0.89	1.16	0.82	1.11	
05/05/86	MS25B	PEMIX #2					20
05/05/86	MS25B	1840 - 1	0.81	1.06	0.82	1.11	
05/19/86	MS25B	PEMIX #2					24
05/19/86	MS25B	1840 - 1	0.88	1.09	0.82	1.11	
05/20/86	MS25B	PEMIX #2					21
05/20/86	MS25B	1840 - 1	0.83	1.06	0.82	1.11	

TABLE 10

BREHM LABORATORY, WRIGHT STATE UNIVERSITY, DAYTON, OHIO 45435

SUMMARY OF TETRA TO OCTA CDD AND CDF CALIBRATION RF AND MEAN RF

ARD	DATE	37C14 TCDF	37C14 TCDD	13C12 TCDD	13C12 OCDD	TCDF	TCDD	PCDF	PCDD	HXCDF	HXCDD	HPCDF	HPCDD	OCDF	OCDD
ATION	CURVE	2.47	1.11	2.25	3762506.07	1.18	0.82	0.76	0.51	0.57	0.33	2.67	1.24	0.75	0.86
- 1	04/29/86	2.17	1.16	2.13	3370372.90	1.19	0.89	0.84	0.47	0.71	0.31	2.59	1.08	0.72	0.89
- 1	05/05/86	2.36	1.06	2.13	2824702.99	1.18	0.81	0.73	0.47	0.55	0.32	3.03	1.35	0.76	0.86
- 1	05/19/86	1.77	1.09	1.67	2804630.53	1.15	0.88	0.70	0.45	0.57	0.27	4.18	1.82	0.37	1.00
- 1	05/20/86	1.81	1.06	1.87	2852274.97	1.15	0.83	0.71	0.41	0.63	0.32	3.65	1.60	0.34	0.94

TABLE 11

Sample Summary for TCDF - OCDD 2378 TCDF,TCDD
Customer ID 27 WSU ID USC 6-1
Date: 04/29/86 Time: 11:44
Run No. CT04#80 File # 2
Sample Size 0.2000 total sample Injection Amount 0.200 of Sample
Instrument ID MS25B

Spike Levels 37Cl4 TCDF 37Cl4 TCDD 13C12 TCDD 1234 TCDD 13C12 OCDD
0.4 NG 0.4 NG 1.0 NG 0.1 NG 1.0 NG

Internal Standard Summary

M/Z	MAX TIC	% TIC	Ratio	Region	Recovery
312	27251	55	0.00	37Cl4 TCDF	139.4
328	13821	99	0.00	37Cl4 TCDD	98.8
332	14124	99	0.81	13C12 TCDD	129.5
334	17422	99			
332	14124	17	0.81	1234 TCDD	124.5
334	17422	17			
470	3524	97	0.69*	13C12 OCDD	124.0
472	5041	98			

2378 Isomers Summary ng Levels

M/Z	MAX TIC	% TIC	RATIO	REGION	VALUE	MDQ
241	21669	73				
304	45782	100				
306	56539	100	0.81	TCDF	11.727	0.000
257	1503	-30				
320	539	-20				
322	255	-30	0.00*	TCDD	0.000	0.066

TCDF - OCDD 2378 TCDF,TCDD ng Levels

	TCDF	TCDD	PCDF	PCDD	HXCDF	HXCDD	HPCDF	HPCDD	OCDF	OCDD
	41.7	ND	26.6	ND	11.8	ND	1.02	0.472	0.929	0.788
EMDQ		0.0322		0.0826		0.150				
CMDQ		0.0322		0.0826		0.150				
INT										

Isomer Summary Report

M/Z	MAX TIC	% TIC	RATIO	REGION	PK1	PK2	PK3	PK4	PK5	PK6	PK7	PK8	PK9	P10	P11	P12	P13	P14	P15	P16	P17	P18	P19	P20	SUMP
241	29916	45																							
304	45965	99																							
306	57050	99	0.81	TCDF	2	10	3	38	20	22	44	19	10	35	7	99	18	14	12						353
257	132601	-1																							
320	1483	29																							
322	533	42	1.92*	TCDD	-7																				0
275	26620	31																							
338	18413	99																							
340	28707	99	0.64	PCDF	4	99	29	22	13	14	3	36	55	40											315
291	6545	-8																							
354	1323	-3	0.00*	PCDD	-3																				0
356	1272	-10																							
311	9040	-15																							
374	9401	99																							
376	7391	99	1.27	HXCDF	3	75	4	27	99	41	5	13	8	22											297
327	466902	-1																							
390	909	-10																							
392	368	-13	0.00*	HXCDD	-15																				0
345	6670	4																							
408	2641	50																							
410	1304	98	1.03	HPCDF	98	73																			171
361	7357	-3																							
424	273	93																							
426	299	95	0.89*	HPCDD	78	95																			173
379	782	25																							
442	813	71																							
444	671	87	0.99	OCDF	87																				87
395	901	15																							
458	553	91																							
460	686	95	0.77	OCDD	95																				95

TABLE 12

Sample Summary for Change Native-Internal Std. RF
Customer ID 9 (B,C) WSU ID USC 6-2
Date: 04/29/86 Time: 13:28
Run No. CT04#81 File # 3
Sample Size 0.2000 total sample Injection Amount 0.200 of Sample
Instrument ID MS25B

Spike Levels 37C14 TCDF 37C14 TCDD 13C12 TCDD 1234 TCDD 13C12 OCDD
0.4 NG 0.4 NG 1.0 NG 0.1 NG 1.0 NG

Internal Standard Summary

M/Z	MAX TIC	% TIC	Ratio	Region	Recovery
312	26241	63	0.00	37C14 TCDF	191.2
323	14445	93	0.00	37C14 TCDD	164.9
332	327751	-1	0.73	13C12 TCDD	-40.1
334	448448	-1			
332	327751	-1	0.73	1234 TCDD	-180.2
334	448448	-1			
470	16251	28	0.78	13C12 OCDD	154.6
472	17779	33			

2378 Isomers Summary ng Levels

M/Z	MAX TIC	% TIC	RATIO	REGION	VALUE	MDQ
241	28157	29				
304	26043	98				
306	31907	98	0.82	TCDF	5.909	0.000
257	4285	-4				
320	2225	-4				
322	1496	-10	0.00*	TCDD	0.000	0.098

Change Native-Internal Std. RF ng Levels

	TCDF	TCDD	PCDF	PCDD	HXCDF	HXCDD	HPCDF	HPCDD	OCDF	OCDD
	23.3	ND	33.9	ND	25.9	ND	2.06	0.592	1.13	0.968
EMDQ		0.0656		0.143		0.561				
CMDQ		0.0656		0.143		0.561				
INT										

Isomer Summary Report

M/Z	MAX TIC	% TIC	RATIO	REGION	PK1	PK2	PK3	PK4	PK5	PK6	PK7	PK8	PK9	P10	P11	P12	P13	P14	P15	P16	P17	P18	P19	P20	SUMP
241	28116	30																							
304	26341	98																							
306	31843	99	0.82	TCDF	72	20	57	30	18	11	37	99	35	8											387
257	265037	-1																							
320	3106	-3																							
322	1941	-4	0.00*	TCDD	-4																				0
275	13924	66																							
338	27351	98																							
340	42468	99	0.64	PCDF	4	99	34	13	15	11	3	41	47	32											299
291	2665	98																							
354	2363	98																							
356	2312	70	1.43*	PCDD	-5																				0
311	9825	43																							
374	21978	99																							
376	17653	99	1.24	HXCDF	69	4	25	99	46	7	15	11	27												303
327	430138	-1																							
390	5184	-4	0.00*	HXCDD	-5																				0
392	2301	-15																							
345	5588	10																							
408	12912	26																							
410	3891	75	1.15	HPCDF	75	62																			137
361	6981	3																							
424	928	49																							
426	467	94	1.04	HPCDD	67	94																			161
379	644	42																							
442	1530	59																							
444	931	93	1.04*	OCDF	93																				93
395	590	55																							
458	986	90																							
460	918	96	1.01*	OCDD	96																				96

TABLE 13

Sample Summary for TCDF - OCDD 2378 TCDF,TCDD
Customer ID Lab Blanx WSU ID USC6-0B
Date: 04/29/86 Time: 15:10
Run No. CTD4#0082 File # 6
Sample Size 0.2000 total sample Injection Amount 0.300 of Sample
Instrument ID MS25B

Spike Levels 37C14 TCDF 37C14 TCDD 13C12 TCDD 1234 TCDD 13C12 OCDD
0.4 NG 0.4 NG 1.0 NG 0.0 NG 1.0 NG

Internal Standard Summary

M/Z	MAX TIC	% TIC	Ratio	Region	Recovery
312	14669	61	0.00	37C14 TCDF	55.9
328	8626	99	0.00	37C14 TCDD	94.1
332	9525	99	0.84	13C12 TCDD	56.5
334	11286	99			
332	9525	13	0.84	1234 TCDD	125.6
334	11296	13			
470	1776	95	0.69*	13C12 OCDD	40.7
472	2507	97			

2378 Isomers Summary ng Levels

M/Z	MAX TIC	% TIC	RATIO	REGION	VALUE	MDQ
241	22383	-1				
304	5102	-1				
306	493	-8	0.00*	TCDF	0.000	0.033
257	2915	8				
320	702	10				
322	164	32	1.34*	TCDD	0.000	0.031

TCDF - OCDD 2378 TCDF,TCDD ng Levels

	TCDF	TCDD	PCDF	PCDD	HXCDF	HXCDD	HPCDF	HPCDD	OCDF	OCDD
	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
EMDQ	0.0371	0.0480	0.0770	0.130	0.171	0.558	0.547	0.192	0.349	0.177
CMDQ	0.152	0.577	0.120	0.146	0.239	0.558	1.64	1.46	0.559	0.248
INT										

Isomer Summary Report

M/Z	MAX TIC	% TIC	RATIO	REGION	PK1	PK2	PK3	PK4	PK5	PK6	PK7	PK8	PK9	P10	P11	P12	P13	P14	P15	P16	P17	P18	P19	P20	SUMP
241	22457	98																							
304	5107	10																							
306	493	92	1.13*	TCDF	-9	-92																			0
257	3675	-10																							
320	2094	-2																							
322	1223	-3	0.00*	TCDD	-3	-90																			0
275	475	-20																							
338	250	-20																							
340	353	-18	0.00*	PCDF	-18	-70																			0
291	382	-30																							
354	165	-30	0.00*	PCDD	-70	-25																			0
356	2101	-30																							
311	8618	-20																							
374	482	-20	0.00*	HXCDF	-70	-20																			0
376	747	-30																							
327	329167	-2																							
390	1281	-13	0.00*	HXCDD	-13	-30																			0
392	2461	-13																							
345	802	-30																							
408	12635	-15																							
410	2962	-15	0.00*	HPCDF	-60	-8																			0
361	3550	-20																							
424	374	-15																							
426	773	-5	0.00*	HPCDD	-95	-5																			0
379	319	-30																							
442	281	-30																							
444	229	-20	0.00*	OCDF	-80	-20																			0
395	217	-30																							
458	165	-30																							
460	136	-20	0.00*	OCDD	-70	-20																			0

TABLE 14

Sample Summary for TCDF - OCDD 2378 TCDF,TCDD
Customer ID 13855 WSU ID NBC5-3
Date: 05/05/86 Time: 16:07
Run No. CT04#0097 File # 7
Sample Size 15.63 gram Injection Amount 0.100 of Sample
Instrument ID #5253

Spike Levels 37Cl4 TCDF 37Cl4 TCDD 13C12 TCDD 1234 TCDD 13C12 OCDD
0.4 NG 0.4 NG 1.0 NG 0.1 NG 1.0 NG

Internal Standard Summary

M/Z	MAX TIC	% TIC	Ratio	Region	Recovery
312	38115	5	0.00	37Cl4 TCDF	74.7
326	3824	99	0.00	37Cl4 TCDD	79.4
332	55030	6	0.55	13C12 TCDD	55.5
334	105770	6			
332	55030	10	0.74	1234 TCDD	185.1
334	105770	7			
470	1313	80	0.69*	13C12 OCDD	88.0
472	1735	85			

2378 Isomers Summary PPB Levels

M/Z	MAX TIC	% TIC	RATIO	REGION	VALUE	MDQ
241	97746	12				
304	60003	100				
306	78971	100	0.76	TCDF	0.474	0.000
257	51981	-5				
320	1603	-10				
322	612	-20	0.00*	TCDD	0.000	0.004

TCDF - OCDD 2378 TCDF,TCDD PPB Levels

	TCDF	TCDD	PCDF	PCDD	HXCDF	HXCDD	HPCDF	HPCDD	OCDF	OCDD
	4.07	ND	1.64	ND	1.03	0.203	0.482	0.675	0.420	1.82
EMDQ	0.0172	0.0093	0.0164	0.0098	0.0129	0.0148	0.0205	0.0268	0.0522	0.0901
CMDQ	0.0172	0.0693	0.0328	0.0315	0.0181	0.100	0.0205	0.0268	0.104	0.0901
INT										

Isomer Summary Report

M/Z	MAX TIC	% TIC	RATIO	REGION	PK1	PK2	PK3	PK4	PK5	PK6	PK7	PK8	PK9	P10	P11	P12	P13	P14	P15	P16	P17	P18	P19	P20	SUMP
241	166436	20																							
304	88826	100																							
306	113929	100	0.78	TCDF	6	28	9	69	72	100	10	38	29	21	31	20	70	52	29	4	-2	-1			588
257	54925	-5																							
320	4464	93	0.00*	TCDD	-93	-5																			0
322	1576	-30																							
275	68962	20																							
338	45913	99																							
340	76114	100	0.60	PCDF	5	100	22	6	17	8	13	8	18	18	18	19	-5	-1							252
291	23911	-10																							
354	2510	90																							
356	2087	80	1.35*	PCDD	-80	-10																			0
311	75025	15																							
374	20946	98																							
376	16400	98	1.28	HXCDF	87	5	7	32	50	99	48	6	15	11	30	-7	-2								390
327	723308	1																							
390	6039	60																							
392	2919	98	1.27	HXCDD	50	24	55	-85	-5																169
345	31184	13																							
408	14361	97																							
410	13969	98	1.02	HPCDF	98	6	55	18	-5	-3															177
361	50301	7																							
424	9290	97																							
426	8493	97	1.09	HPCDD	86	97	-5	-3																	183
379	12250	-40																							
442	6451	94																							
444	6447	95	0.99	OCDF	95	-25	-5																		95
395	26763	-30																							
458	29913	99																							
460	32662	99	0.92	OCDD	99	-2	-2																		99

TABLE 15

Sample Summary for TCDF - OCDD 2378 TCDF,TCDD
Customer ID Lab Blank WSU ID NBC5-0-B
Date: 04/23/86 Time: 16:51
Run No. CT04#0083 File # 8
Sample Size 15.00 gram Injection Amount 0.300 of Sample
Instrument ID MS25B

Spike Levels 37C14 TCDF 37C14 TCDD 13C12 TCDD 1234 TCDD 13C12 OCDD
0.4 NG 0.4 NG 1.0 NG 0.0 NG 1.0 NG

Internal Standard Summary

M/Z	MAX TIC	% TIC	Ratio	Region	Recovery
312	16804	49	0.00	37C14 TCDF	50.2
328	8293	99	0.00	37C14 TCDD	90.4
332	9159	99	0.30	13C12 TCDD	54.5
334	11455	99			
332	9159	14	0.86	1234 TCDD	129.7
334	11455	13			
470	1700	97	0.71*	13C12 OCDD	39.2
472	2363	98			

2378 Isomers Summary PPB Levels

M/Z	MAX TIC	% TIC	RATIO	REGION	VALUE	MDQ
241	20748	44				
304	4956	-2				
306	507	-5	0.00*	TCDF	0.000	0.000
257	2749	-5				
320	719	-4				
322	183	-25	0.00*	TCDD	0.000	0.001

TCDF - OCDD 2378 TCDF,TCDD PPB Levels

	TCDF	TCDD	PCDF	PCDD	HXCDF	HXCDD	HPCDF	HPCDD	OCDF	OCDD
	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.0255
EMDQ	0.0004	0.0008	0.0020	0.0020	0.0045	0.0223	0.0243	0.0062	0.0056	0.0035
CMDQ	0.0023	0.0010	0.0020	0.0020	0.0045	0.0223	0.0243	0.0066	0.0068	0.0035
INT										

Isomer Summary Report

M/Z	MAX TIC	% TIC	RATIO	REGION	PK1	PK2	PK3	PK4	PK5	PK6	PK7	PK8	PK9	P10	P11	P12	P13	P14	P15	P16	P17	P18	P19	P20	SUMP
241	20748	99																							
304	4956	10																							
306	507	93	1.05*	TCDF	-93	-7																			0
257	2749	-5																							
320	719	-5																							
322	183	-25	0.00*	TCDD	-75	-25																			0
275	663	-30																							
338	251	-40																							
340	292	-40	0.00*	PCDF	-60	-40																			0
291	304	-40																							
354	157	-30	0.00*	PCDD	-70	-30																			0
356	1729	-30																							
311	8145	-10																							
374	546	-40																							
376	714	-40	0.00*	HXCDF	-20	-40																			0
327	323368	-1																							
390	1236	-40	0.00*	HXCDD	-50	-40																			0
392	2084	-40																							
345	815	-30																							
408	10649	-30																							
410	2531	-30	0.00*	HPCDF	-40	-30																			0
361	3235	-20																							
424	206	60																							
426	302	80	0.51*	HPCDD	-80	-30																			0
379	338	-30																							
442	232	-25																							
444	213	-25	0.00*	OCDF	-75	-25																			0
395	189	-40																							
458	670	95																							
460	779	96	0.85	OCDD	96	-5	-5																		96

TABLE 16

Sample Summary for TCDF - OCDD 2378 TCDF,TCDD
Customer ID 13858 WSU ID NBC5-1
Date: 05/19/86 Time: 14:52
Run No. CT04#186 File # 14
Sample Size 11.73 gram Injection Amount 0.100 of Sample
Instrument ID MS25B

Spike Levels 37Cl4 TCDF 37Cl4 TCDD 13C12 TCDD 1234 TCDD 13C12 OCDD
0.4 NG 0.4 NG 1.0 NG 0.1 NG 1.0 NG

Internal Standard Summary

M/Z	MAX TIC	% TIC	Ratio	Region	Recovery
312	22572	48	0.00	37Cl4 TCDF	73.8
328	6323	98	0.00	37Cl4 TCDD	106.2
332	9067	67	0.66	13C12 TCDD	44.5
334	10892	63			
332	9067	99	0.23	1234 TCDD	493.2
334	10892	99			
470	2579	90	0.77*	13C12 OCDD	198.3
472	3121	97			

2378 Isomers Summary PPB Levels

M/Z	MAX TIC	% TIC	RATIO	REGION	VALUE	MDQ
241	60203	35				
304	104872	100				
306	137997	100	0.76	TCDF	0.730	0.000
257	75072	3				
320	340	-25				
322	674	-25	0.00*	TCDD	0.000	0.004

TCDF - OCDD 2378 TCDF,TCDD PPB Levels

	TCDF	TCDD	PCDF	PCDD	HXCDF	HXCDD	HPCDF	HPCDD	OCDF	OCDD
	3.63	ND	2.43	ND	1.91	0.352	0.628	0.673	0.679	2.80
EMDQ		0.0031		0.0074						
CMDQ		0.0031		0.0074						
INT										

Isomer Summary Report

M/Z	MAX TIC	% TIC	RATIO	REGION	PK1	PK2	PK3	PK4	PK5	PK6	PK7	PK8	PK9	P10	P11	P12	P13	P14	P15	P16	P17	P18	P19	P20	SUMP
241	135310	22																							
304	106121	100																							
306	139514	100	0.76	TCDF	2	3	5	22	20	42	7	72	37	53	17	100	67	35	10						492
257	79206	-2																							
320	684	-10	0.00*	TCDD	-10																				0
322	1190	-12																							
275	96992	27																							
338	77466	100																							
340	125898	100	0.62	PCDF	5	100	23	21	10	16	6	23	22	28											254
291	33454	-5																							
354	3221	-3																							
356	2413	-6	0.00*	PCDD	-6																				0
311	46802	33																							
374	51206	99																							
376	41382	99	1.24	HXCDF	31	67	10	28	25	99	47	15	9												331
327	810404	-1																							
390	7280	71																							
392	4144	98	1.27	HXCDD	15	46	14	16	98																189
345	33510	18																							
408	25225	100																							
410	23774	100	1.06	HPCDF	100	13	73	28																	214
361	43103	7																							
424	13719	99																							
426	13087	99	1.05	HPCDD	99	96																			195
379	13106	43																							
442	15480	99																							
444	16647	99	0.93	OCDF	99																				99
395	61779	33																							
458	72582	100																							
460	80966	100	0.90	OCDD	100																				100

TABLE 17

Sample Summary for TCDF - OCDD 2378 TCDF,TCDD
Customer ID 13867 WSU ID NBC5-5
Date: 05/19/86 Time: 16:31
Run No. CT04#107 File # 15
Sample Size 14.47 gram Injection Amount 0.100 of Sample
Instrument ID MS25B

Spike Levels 37Cl4 TCDF 37Cl4 TCDD 13C12 TCDD 1234 TCDD 13C12 OCDD
0.4 NG 0.4 NG 1.0 NG 0.1 NG 1.0 NG

Internal Standard Summary

M/Z	MAX TIC	% TIC	Ratio	Region	Recovery
312	14582	47	0.00	37Cl4 TCDF	59.2
328	6320	98	0.00	37Cl4 TCDD	105.9
332	52025	11	0.77	13C12 TCDD	54.3
334	63315	8			
332	156275	5	0.93*	1234 TCDD	404.5
334	250045	3			
470	4431	75	0.92	13C12 OCDD	60.2
472	4443	80			

2378 Isomers Summary PPB Levels

M/Z	MAX TIC	% TIC	RATIO	REGION	VALUE	MDQ
241	95475	15				
304	49129	99				
306	61431	99	0.80	TCDF	0.406	0.000
257	5553	-35				
320	4900	-35				
322	439	-35	0.00*	TCDD	0.000	0.004

TCDF - OCDD 2378 TCDF,TCDD PPB Levels

	TCDF	TCDD	PCDF	PCDD	HXCDF	HXCDD	HPCDF	HPCDD	OCDF	OCDD
	2.50	ND	1.64	ND	2.24	0.171	0.343	0.272	0.301	0.921
EMDQ		0.0030		0.0061						
CMDQ		0.0030		0.0061						
INT										

Isomer Summary Report

M/Z	MAX TIC	% TIC	RATIO	REGION	PK1	PK2	PK3	PK4	PK5	PK6	PK7	PK8	PK9	P10	P11	P12	P13	P14	P15	P16	P17	P18	P19	P20	SUMP
241	131828	44																							
304	116640	66																							
306	101062	99	0.77	TCDF	17	10	42	99	7	22	27	21	15	61	29	27									377
257	22070	-12																							
320	877	-7																							
322	1505	-5	0.00*	TCDD	-7																				0
275	106627	16																							
338	47785	98																							
340	78178	99	0.61	PCDF	4	99	33	5	14	10	21	16	23												225
291	55159	-3																							
354	1175	90																							
356	3676	30	0.96*	PCDD	-4																				0
311	120045	40																							
374	44736	98																							
376	36209	98	1.24	HXCDF	35	82	28	20	98	55	12	10	20												360
327	801684	-1																							
390	3482	87																							
392	2898	87	1.20	HXCDD	19	62	87																		
345	54034	12																							
408	26669	97																							
410	25017	97	1.07	HPCDF	97	11	42	25																	
361	64265	2																							
424	9169	96																							
426	8747	96	1.05	HPCDD	96	90																			
379	18380	30																							
442	11639	92																							
444	12413	93	0.93	OCDF	93																				0
395	27150	40																							
458	39014	97																							
460	43258	97	0.90	OCDD	97																				95

TABLE 18

Sample Summary for TCDF - OCDD 2378 TCDF,TCDD
Customer ID Lab Blank WSU ID NBC5-08-B
Date: 05/20/86 Time: 12:19
Run No. CT04#109 File # 16
Sample Size 13.00 gram Injection Amount 0.100 of Sample
Instrument ID MS25B

Spike Levels 37C14 TCDF 37C14 TCDD 13C12 TCDD 1234 TCDD 13C12 OCDD
0.4 NG 0.4 NG 1.0 NG 0.1 NG 1.0 NG

Internal Standard Summary

M/Z	MAX TIC	% TIC	Ratio	Region	Recovery
312	1667	96	0.00	37C14 TCDF	27.4
326	1920	98	0.00	37C14 TCDD	92.4
332	4144	48	0.84	13C12 TCDD	32.1
334	4929	48			
332	4144	100	0.84	1234 TCDD	281.6
334	4929	100			
470	361	92	0.77*	13C12 OCDD	26.7
472	453	95			

2378 Isomers Summary PPB Levels

M/Z	MAX TIC	% TIC	RATIO	REGION	VALUE	MDQ
241	494	-60				
304	129	-60				
306	59	-60	0.00*	TCDF	0.000	0.002
257	78	-70				
320	47	-60				
322	46	-60	0.00*	TCDD	0.000	0.003

TCDF - OCDD 2378 TCDF,TCDD PPB Levels

	TCDF	TCDD	PCDF	PCDD	HXCDF	HXCDD	HPCDF	HPCDD	OCDF	OCDD
	ND	ND	ND	ND	ND	ND	ND	0.0234	ND	0.0782
EMDQ	0.0020	0.0023	0.0038	0.0076	0.0067	0.0102	0.0064		0.0203	
CMDQ	0.0020	0.0023	0.0038	0.0076	0.0067	0.0102	0.0064		0.0203	
INT										

Isomer Summary Report

M/Z	MAX TIC	% TIC	RATIO	REGION	PK1	PK2	PK3	PK4	PK5	PK6	PK7	PK8	PK9	P10	P11	P12	P13	P14	P15	P16	P17	P18	P19	P20	SUMP
241	500	-40																							
304	119	-40																							
306	63	-45	0.00*	TCDF	-45																				0
257	171	-35																							
320	55	-35																							
322	69	-35	0.00*	TCDD	-35																				0
275	269	-35																							
338	84	-35																							
340	109	-35	0.00*	PCDF	-35																				0
291	180	-40																							
354	94	-35	0.00*	PCDD	-35																				0
356	1075	-8																							
311	28565	-20																							
374	406	-10																							
376	362	-10	0.00*	HXCDF	-10																				0
327	859568	-1																							
390	578	-15																							
392	379	-8	0.00*	HXCDD	-9																				0
345	698	-40																							
408	420	-40																							
410	83	-40	0.00*	HPCDF	-40																				0
361	136	80																							
424	94	60																							
426	111	80	0.85*	HPCDD	60	80																			140
379	232	-40																							
442	98	-40																							
444	80	-40	0.00*	OCDF	-40																				0
395	149	70																							
458	302	95																							
460	413	95	0.73*	OCDD	95																				95

TABLE 19

ARD SUMMARY FOR TETRA THROUGH OCTA CHLORINATED DIOXINS AND FURANS
ARD ID 1840 - 1
24/29/86 TIME: 10:10
D. CTO4#0079 FILE # 41 STD CURVE # 2
TION AMOUNT 1.000 UL OF STANDARD
UMENT ID MS25B
NAL STANDARD LEVELS NG PER UL
TCDF 37C14 TCDD 13C12 TCDD 13C12 1234 13C12 OCDD
NG 0.4 NG 1.0 NG 0.1 NG 1.0 NG
E LEVELS NG PER UL
0.400
NTERNAL STANDARD SUMMARY

MAX TIC	% TIC	RATIO	REGION	MEASURED RF	MEAN RF
33075	99	0.00	37C14 TCDF	2.17*	2.47
43293	99	0.00	37C14 TCDD	1.15	1.11
40552	99	0.79	13C12 TCDD		
51354	100				
40968	7	0.80	13C12 1234	4308360.00	7131246.67
51354	7				
14548	99	0.75*	13C12 OCDD	3370372.90	3762506.07
19502	99				

E SUMMARY REPORT FOR STANDARD

MAX TIC	% TIC	RATIO	REGION	MEASURED RF	MEAN RF
4835	98				
19598	99				
25370	99	0.77	TCDF	1.19	1.18
6496	99				
14449	99				
18763	99	0.77	TCDD	0.89	0.82
3106	97				
12262	99				
19624	99	0.62	PCDF	0.84*	0.76
3088	98				
6758	99				
10904	98	0.63	PCDD	0.47	0.51
19749	17				
15129	99				
11827	99	1.28	HXCDF	0.71*	0.57
871628	1				
6390	99				
4994	99	1.28	HXCDD	0.31	0.33
3720	97				
17965	99				
17349	99	1.04	HPCDF	2.59	2.67
2291	96				
7518	99				
7224	99	1.04	HPCDD	1.08*	1.24
1216	95				
4841	99				
4989	99	0.97	OCDF	0.72	0.75
1374	97				
5698	99				
6477	99	0.88	OCDD	0.89	0.88

TABLE 20

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ARD SUMMARY FOR TETRA THROUGH OCTA CHLORINATED DIOXINS AND FURANS
ARD ID     1840 - 1
 05/05/86    TIME: 09:51
D.     CT04#0095    FILE # 50  STD CURVE # 2
TION AMOUNT 1.000 UL OF STANDARD
UMENT ID  MS25B
NAL STANDARD LEVELS NG PER UL
 TCDF 37C14 TCDD 13C12 TCDD 13C12 1234 13C12 OCDD
NG     0.4 NG     1.0 NG     0.1 NG     1.0 NG
E LEVELS NG PER UL
  0.400
NTERNAL STANDARD SUMMARY
 MAX TIC % TIC RATIO  REGION      MEASURED RF    MEAN RF
   44542    99  0.00  37C14 TCDF        2.35       2.47
   42557    99  0.00  37C14 TCDD        1.06       1.11
   45095    99  0.82  13C12 TCDD
   54498   100
   45095     7  0.83  13C12 1234       4647673.33 7131246.67
   54498     7
   11718    99  0.70* 13C12 OCDD       2824702.98w3762606.07
   16819    99
E SUMMARY REPORT FOR STANDARD
 MAX TIC % TIC  RATIO  REGION     MEASURED RF    MEAN RF
    5753    97
   22816    99
   29526    99   0.77  TCDF          1.18          1.18
    7638    98
   14383    99
   18181    99   0.79  TCDD          0.81          0.82
    3643    96
   12429    99
   20068    99   0.62  PCDF          0.73          0.76
    3549    98
    7232    99
   11711    98   0.62  PCDD          0.47          0.51
   23434    12
   13509    99
   11058    99   1.22  HXCDF         0.55          0.57
  956314     1
    7145    97
    5751    97   1.24  HXCDD         0.32          0.33
    3713    95
   18563    95
   16987    98   1.06  HPCDF         3.03*         2.67
    2517    95
    8015    99
    7478    99   1.07  HPCDD         1.36          1.24
    1034    92
    4343    98
    4299   100   0.99  OCDF          0.76          0.75
    1191    97
    4675    99
    5163    99   0.91  OCDD          0.86          0.88
```

TABLE 21

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ARD SUMMARY FOR TETRA THROUGH OCTA CHLORINATED DIOXINS AND FURANS
ARD ID     1840 - 1
 05/19/86    TIME: 11:30
O.     CTO4#0105    FILE # 56  STD CURVE # 2
TION AMOUNT 1.000 UL OF STANDARD
UMENT ID  MS25B
NAL STANDARD LEVELS NG PER UL
 TCDF 37C14 TCDD 13C12 TCDD 13C12 1234 13C12 OCDD
NG     0.4 NG     1.0 NG     0.1 NG     1.0 NG
E LEVELS NG PER UL
  0.400
NTERNAL STANDARD SUMMARY
 MAX TIC % TIC RATIO  REGION      MEASURED RF    MEAN RF
   57219   100  0.00  37C14 TCDF        1.77*       2.47
   58585   100  0.00  37C14 TCDD        1.05        1.11
   53468   100  0.80  13C12 TCDD
   74080   100
   53468     9  0.80  13C12 1234       2670960.00 7131246.67
   74080     9
   12213    99  0.76* 13C12 OCDD       2804630.583762506.07
   16122    99
E SUMMARY REPORT FOR STANDARD
 MAX TIC % TIC  RATIO  REGION     MEASURED RF    MEAN RF
    7158    99
   28837    99
   38056    99   0.76  TCDF          1.16          1.18
    9350    99
   21150    99
   26204    99   0.81  TCDD          0.88          0.82
    3738    97
   15306    99
   24721    99   0.62  PCDF          0.70          0.76
    4030    99
    9512    99
   15122    98   0.64  PCDD          0.45*         0.51
   25981    12
   18100    99
   14541    99   1.24  HXCDF         0.57          0.57
  810197     1
    8202    99
    6489    99   1.26  HXCDD         0.27*         0.33
    4570    95
   24490    99
   22844    99   1.07  HPCDF         4.18*         2.67
    3018    98
   10618    99
   10044    99   1.06  HPCDD         1.82*         1.24
    1384    92
    5479    99
    5540    99   0.99  OCDF          0.97*         0.75
    1526    95
    5296    99
    5998    99   0.88  OCDD          1.00*         0.88
```

TABLE 22

ARD SUMMARY FOR TETRA THROUGH OCTA CHLORINATED DIOXINS AND FURANS
ARD ID 1840 - 1
05/20/86 TIME: 10:43
D. CTO4#0108 FILE # 58 STD CURVE # 2
TION AMOUNT 1.000 UL OF STANDARD
UMENT ID MS253
NAL STANDARD LEVELS NG PER UL
TCDF 37C14 TCDD 13C12 TCDD 13C12 1234 13C12 OCDD
NG 0.4 NG 1.0 NG 0.1 NG 1.0 NG
E LEVELS NG PER UL
0.400
NTERNAL STANDARD SUMMARY

MAX TIC	% TIC	RATIO	REGION	MEASURED RF	MEAN RF
46989	100	0.00	37C14 TCDF	1.81*	2.47
51247	100	0.00	37C14 TCDD	1.06	1.11
53735	100	0.80	13C12 TCDD		
67088	100				
53735	8	0.80	13C12 1234	2147964.44	7131246.67
67066	8				
12491	99	0.77*	13C12 OCDD	2852274.97	3762606.07
16325	99				

E SUMMARY REPORT FOR STANDARD

MAX TIC	% TIC	RATIO	REGION	MEASURED RF	MEAN RF
5894	99				
23658	99				
30699	99	0.77	TCDF	1.15	1.18
7529	99				
17977	99				
22662	99	0.79	TCDD	0.83	0.82
3197	97				
12911	99				
20763	99	0.62	PCDF	0.71	0.76
3412	98				
7607	99				
12769	98	0.60	PCDD	0.41*	0.51
26168	12				
16472	99				
13282	99	1.24	HXCDF	0.63	0.57
824513	1				
8730	98				
6819	98	1.28	HXCDD	0.32	0.33
3994	95				
21757	99				
20341	99	1.07	HPCDF	3.65*	2.67
2497	98				
9530	99				
8903	99	1.07	HPCDD	1.60*	1.24
1327	95				
5375	99				
5439	99	0.99	OCDF	0.94*	0.75
1525	98				
5125	99				
5713	99	0.90	OCDD	0.94	0.88

ATTACHMENT G

TABLES OF DATA

RESULTS OF BREHM LABORATORY ANALYSES OF U.S. COAST GUARD SAMPLE EXTRACTS AND OF SEDIMENTS SUBMITTED BY NUS CORP. FOR PCB

TABLE 23

Brenm Laboratory, Wright State University, Dayton, Ohio 45435

Results for PCB s
Concentrations Found (micrograms per Total Sample) a.

S/EPA D	Brenm Lab NO.	% Rec of 6C13 Tetra PCB	Total Tri PCBs	Total Tetra PCBs	Total Penta PCBs	Total Hexa PCBs	Total Hepta PCBs	Summed Total PCBS
	USC6-1	80.4	5015	12986	2999	628	52.5	21582
C)	USC6-2	127.1	4019	5090	4553	2218	194	16074
lank	USC6-0-B	80.5	0.0005	0.0016	ND 0.0001	ND 0.0001	ND 0.0001	0.0021

The designation ND indicates "None Detected" in excess of the minimum detectable concentration which is listed directly below the ND designation.

TABLE 24

Brehm Laboratory, Wright State University, Dayton, Ohio 45435

Results for PCB s
Concentrations Found (micrograms per gram of sample or parts-per-million) a.

NUS/EPA Sample I.D. NO	Brehm Lab NO.	% Rec of 6C13 Tetra PCB	Total Tri PCBs	Total Tetra PCBs	Total Penta PCBs	Total Hexa PCBs	Total Hepta PCBs	Summed Total PCBS
13855	NBC5-3	124.6	120	83.5	23.3	5.53	0.424	232
Lab Blank	NBC5-0-3	90.3	0.0001	0.0001	0.0002	0.0002	0.0000	0.0006
13856	NBC5-1	56.9	114	98.6	27.6	2.96	0.386	243
13867	NBC5-5	37.1	28.1	42.0	34.5	11.9	1.04	124
Lab Blank	NBC5-0B-3	69.4	0.0002	0.0002	0.0001	0.0001	0.0000	0.0006

a. The designation ND indicates "None Detected" in excess of the minimum detectable concentration which is listed directly below the ND designation.

TABLE 25

Sample Summary for PCBs
Sample ID 27 WSU ID USC6-1
Date: 05/14/86 Time: 16:18
Run NO. PCBM#7 FILE # 1
Sample Weight 0.20 G Injection Amount 0.000 of Sample
Instrument ID MS25B

Spike Levels D6 TBD
125000.0 NG

Internal Standard Summary

m/z	Max TIC	% TIC	Ratio	Region		Recovery
256	297528	100	0.80	D6	TBD	80.4
258	369644	100				

Total PCB Results PPM Levels

Tri	Tetra	Penta	Hexa	Hepta
5015	12506	2839	628	52.5

EMDQ
CMDQ

Isomer Summary Report for Sample

m/z	Max TIC	% TIC	Ratio	Region	PK1	PK2	PK3	PK4	PK5	PK6	PK7	PK8	PK9	P10	P11	P12	P13	P14	P15	P16	P17	P18	P19	P20	SUMP
256	297528	100	0.80	Tri	100	93	26	95	69	39	98	100	100	93	2	2	3	3	3	70					896
258	369644	100																							
290	455221	100	0.90	Tetra	21	6	24	12	76	92	42	90	50	38	52	19	4	4	72	95	100	52	12		870
292	506698	100																							
326	251210	100	1.63	Penta	3	67	24	3	32	42	3	3	25	7	4	100	2	3	4	65	2	2	9		401
328	154462	100																							
360	36561	100	1.36	Hexa	37	7	15	7	100	12	2	2	13	12	7	7	3	86	14	3	13	10	2		352
362	26917	100																							
394	1743	98	1.19	Hepta	60	12	24	4	4	93	60	31	3	3	52	6	98								450
396	1470	98																							

TABLE 26

Sample Summary for PCBs
Sample ID 9 (B.C) WSU ID USC6-2
Date: 05/14/86 Time: 16:13
Run NO. PCBM#8 FILE # 2
Sample Weight 0.20 G Injection Amount 0.000 of Sample
Instrument ID MS25B

Spike Levels D6 TBD
125000.0 NG

Internal Standard Summary

m/z	Max TIC	% TIC	Ratio	Region		Recovery
296	584562	100	0.92	D6	TBD	127.1
298	550480	100				

Total PCB Results PPM Levels

Tri	Tetra	Penta	Hexa	Hepta
4019	5050	4553	2218	194

EMDQ
CMDQ

Isomer Summary Report for Sample

m/z	Max TIC	% TIC	Ratio	Region	PK1	PK2	PK3	PK4	PK5	PK6	PK7	PK8	PK9	P10	P11	P12	P13	P14	P15	P16	P17	P18	P19	P20	SUMP
256	580480	100	1.02	Tri	97	58	10	68	2	32	92	100	13	93	56	6	2	2	3	24					658
258	570535	100																							
290	424797	100	0.92	Tetra	10	4	12	4	100	66	18	70	14	21	43	7	2	2	35	91	58	28	4		583
292	461389	100																							
325	485666	100	1.30	Penta	2	60	13	12	81	35	2	4	22	33	12	100	5	2	3	65	20				471
328	372182	100																							
360	164178	100	1.17	Hexa	22	13	11	3	70	7	2	10	65	33	14	6	6	100	15	5	17	10			409
362	140683	100																							
394	9016	100	1.05	Hepta	32	20	12	3	57	33	5	73	35	27	2	10	100	7	3	62					481
396	8558	100																							

TABLE 27

Sample Summary for PCBs
Sample ID Lab Blank WSU ID USC6-0-B
Date: 05/14/86 Time: 12:28
Run NO. PCBM#4 FILE # 4
Sample Weight 0.20 G Injection Amount 0.200 of Sample
Instrument ID MS25B

Spike Levels D6 TBD
3.0 NG

Internal Standard Summary

m/z	Max TIC	% TIC	Ratio	Region	Recovery
296	108858	100	0.83	D6	80.5
298	131639	100		TBD	

Total PCB Results PPM Levels

Tri	Tetra	Penta	Hexa	Hepta
0.2005	0.2016	ND	ND	ND
EMDQ		0.0001	0.0001	0.0001
CMDQ		0.0001	0.0001	0.0001

Isomer Summary Report for Sample

m/z	Max TIC	% TIC	Ratio	Region	PK1	PK2	PK3	PK4	PK5	PK6	PK7	PK8	PK9	P10	P11	P12	P13	P14	P15	P16	P17	P18	P19	P20	SUMP
256	5489	40	1.20*	Tri	40	20	30																		90
258	9131	20																							
290	1916	90	0.83	Tetra	95	50	30	61	32	10	13	70	25	15											401
292	2176	95																							
325	1254	-10	0.00*	Penta	-10																				0
328	1280	-10																							
360	297	-10	0.00*	Hexa	-4																				0
362	1636	-2																							
394	305	-15	0.00*	Hepta	-15																				0
396	310	-15																							

TABLE 28

Sample Summary for PCDs
Sample ID 13833 WSU ID NBCS-3
Date: 05/14/86 Time: 17:00
Run NO. PCDMS9 FILE # 3
Sample Weight 15.53 G Injection Amount 0.000 of Sample
Instrument ID MS25B

Spike Levels D6 TBD
500000.0 NG
Internal Standard Summary

m/z	Max TIC	% TIC	Ratio	Region		Recovery
298	495739	100	0.92	D6	TBD	124.5
298	539096	100				

Total PCB Results PPM Levels

Tri	Tetra	Penta	Hexa	Hepta
120	83.5	22.3	5.93	0.424

EMDQ
CMDQ
Isomer Summary Report for Sample

m/z	Max TIC	% TIC	Ratio	Region	PK1	PK2	PK3	PK4	PK5	PK6	PK7	PK8	PK9	P10	P11	P12	P13	P14	P15	P16	P17	P18	P19	P20	SUMP
256	459865	100	1.05	Tri	3	57	34	5	36	48	35	100	92	27	38	7									482
258	436882	100																							
290	115538	100	0.78	Tetra	7	7	17	6	62	100	40	60	27	25	50	13	8	4	45	62	55	30	5		623
292	147524	100																							
326	66981	99	1.54	Penta	3	45	24	5	25	51	5	4	22	3	99	3	51	3							343
328	43407	99																							
360	13108	100	1.31	Hexa	28	7	12	7	100	11	3	17	4	2	2	45	13	8	11	6					276
362	10033	100																							
394	381	95	0.98	Hepta	50	52	77	50	25	88	22	95													459
396	390	95																							

TABLE 29

Sample Summary for PCBs
Sample ID Lab Blank WSU ID NBC5-0-9
Date: 05/14/86 Time: 13:18
Run NO. PCBM#5 FILE # 5
Sample Weight 15.00 G Injection Amount 0.100 of Sample
Instrument ID MS25B

Spike Levels D5 TBD
3.0 NG

Internal Standard Summary

m/z	Max TIC	% TIC	Ratio	Region		Recovery
296	42000	100	0.30	D6	TBD	90.3
299	52000	100				

Total PCB Results PPM Levels

Tri	Tetra	Penta	Hexa	Hepta
0.0001	0.0001	0.0002	0.0002	0.0000

EMDQ
CMDQ

Isomer Summary Report for Sample

m/z	Max TIC	% TIC	Ratio	Region	PK1	PK2	PK3	PK4	PK5	PK6	PK7	PK8	PK9	P10	P11	P12	P13	P14	P15	P16	P17	P18	P19	P20	SUMP
256	4682	90	0.80*	Tri	35	25	30	60	100	30	15	50	25												370
258	5881	90																							
290	2887	97	0.77	Tetra	85	30	25	52	25	15	98	45	25												403
292	3767	97																							
326	7533	98	1.63	Penta	42	6	10	82	25	19	42	12	98	6	78	60									480
328	4614	98																							
360	3023	98	1.40	Hexa	15	12	70	6	13	96	18	40	10	7	98	12	6	24	13						442
362	2164	98																							
394	212	90	1.00	Hepta	35	72	45	65	25	90	50														382
396	213	90																							

TABLE 30

Sample Summary for PCBs
Sample ID 13858 WSU ID NBC5-1
Date: 05/14/86 Time: 15:31
Run NO. PCBM#17 FILE # 6
Sample Weight 11.73 G Injection Amount 0.000 of Sample
Instrument ID MS25B

Spike Levels D6 TBD
125000.0 NG

Internal Standard Summary

m/z	Max TIC	% TIC	Ratio	Region		Recovery
296	200631	100	0.74	D6	TBD	56.9
298	271548	100				

Total PCB Results PPM Levels

Tri	Tetra	Penta	Hexa	Hepta
114	98.5	27.6	2.96	0.386

EMDQ
CMDQ

Isomer Summary Report for Sample

m/z	Max TIC	% TIC	Ratio	Region	PK1	PK2	PK3	PK4	PK5	PK6	PK7	PK8	PK9	P10	P11	P12	P13	P14	P15	P16	P17	P18	P19	P20	SUMP
256	638110	100	1.00	Tri	15	12	4	63	10	38	40	100	41	87	3	27									440
258	638149	100																							
290	254430	100	0.72	Tetra	3	2	15	12	2	4	7	44	41	25	100	16	8	12	76	55	2	10			435
292	355454	100																							
326	220273	100	1.60	Penta	12	15	30	8	100	13	2														180
328	137445	100																							
360	8984	99	1.24	Hexa	3	7	40	37	3	24	99	13	23	17	3										269
362	7265	99																							
394	1419	98	1.10	Hepta	13	35	17	98																	163
396	1290	98																							

TABLE 31

Sample Summary for PCBs
Sample ID 13867 WSU ID NBCS-5
Date: 05/21/86 Time: 15:26
Run NO. PCBM#18 FILE # 7
Sample Weight 14.47 G Injection Amount 0.000 of Sample
Instrument ID MS25B

Spike Levels D6 TED
125000.0 NG

Internal Standard Summary

m/z	Max TIC	% TIC	Ratio	Region		Recovery
255	127453	100	0.75	D6	TED	37.1
258	170831	100				

Total PCB Results PPM Levels

Tri	Tetra	Penta	Hexa	Hepta
25.9	48.0	34.5	11.9	1.04

EMDQ
CMDQ

Isomer Summary Report for Sample

m/z	Max TIC	% TIC	Ratio	Region	PK1	PK2	PK3	PK4	PK5	PK6	PK7	PK8	PK9	P10	P11	P12	P13	P14	P15	P16	P17	P18	P19	P20	SUMP
255	155490	100	1.02	Tri	65	33	4	32	26	18	71	100	37	23	2	2	7								420
258	152872	100																							
290	88929	100	0.80	Tetra	6	2	4	80	100	39	32	23	38	43	3	3	2	23	58	81	18	3	2		570
292	111305	100																							
326	107776	100	1.58	Penta	43	35	13	86	95	8	3	24	3	100	2	6	82	2							502
328	68333	100																							
360	33253	100	1.29	Hexa	16	15	8	100	7	18	100	3	2	2	47	6	8	4							336
362	25819	100																							
394	2090	99	1.05	Hepta	38	7	28	15	88	35	22	13	10	7	99	11	2	38							413
396	1985	99																							

TABLE 32

Sample Summary for PCBs
Sample ID Lab Blank WSU ID NBCS-08-B
Date: 05/21/86 Time: 14:37
Run NO. PCB#16 FILE # 8
Sample Weight 13.00 G Injection Amount 0.100 of Sample
Instrument ID MS25B

Spike Levels D6 TBD
25.0 NG

Internal Standard Summary

m/z	Max TIC	% TIC	Ratio	Region	Recovery
255	241625	100	0.77	D6	69.4
256	315630	100		TBD	

Total PCB Results PPM Levels

Tri	Tetra	Penta	Hexa	Hepta
0.0002	0.0002	0.0001	0.0001	0.0000

EMDQ
CMDQ

Isomer Summary Report for Sample

m/z	Max TIC	% TIC	Ratio	Region	PK1	PK2	PK3	PK4	PK5	PK6	PK7	PK8	PK9	P10	P11	P12	P13	P14	P15	P16	P17	P18	P19	P20	SUMP
256	7435	98	1.04	Tri	98	53	61	23	15	61	92	52	28	12	7										502
258	7133	98																							
290	3018	98	0.80	Tetra	5	11	9	98	53	55	60	18	34	8	21	52	34	22							480
292	3751	98																							
326	4321	98	1.54	Penta	46	20	12	64	40	25	22	98	63	16											406
328	2631	98																							
360	1162	98	1.21	Hexa	22	14	16	82	11	13	78	35	13	98	15	10									407
362	963	98																							
394	143	90	0.87	Hepta	57	35	15	67	33	52	23	15	90	52											439
396	165	90																							

TABLE 33

ARD SUMMARY FOR TETRA THROUGH OCTA CHLORINATED DIOXINS AND FURANS
ARD ID 7699-2
05/14/86 TIME: 11:12
0. PCBM#3 FILE # 5 STD CURVE # 2
TION AMOUNT 0.500 UL OF STANDARD
UMENT ID MS-25
NAL STANDARD LEVELS NG PER UL
TCB
NG
E LEVELS NG PER UL
5.000
NTERNAL STANDARD SUMMARY

MAX TIC	% TIC	RATIO	REGION	MEASURED RF	MEAN RF
146065	100	0.79	6213 TCB	332111100.00	664222000.00
185045	100				

E SUMMARY REPORT FOR STANDARD

MAX TIC	% TIC	RATIO	REGION	MEASURED RF	MEAN RF
650000	100				
450000	100	1.44	Mono	1.32	1.32
625000	100				
400000	100	1.56	Di	1.23	1.23
483057	100				
443893	100	1.09	Tri	1.12	1.12
215659	100				
288673	100	0.75	Tetra	0.61	0.61
257254	100				
179206	100	1.44*	Penta	0.53	0.53
149975	100				
126549	100	1.19	Hexa	0.33	0.33
109718	100				
104467	100	1.05	Hepta	0.26	0.26
90466	100				
104176	100	0.87	Octa	0.23	0.23
128509	100				
84647	100	1.52*	Nano	0.26	0.26
75391	100				
64453	100	1.17	Deca	0.17	0.17

TABLE 34

ARD SUMMARY FOR TETRA THROUGH OCTA CHLORINATED DIOXINS AND FURANS
ARD ID 7699-2
05/21/86 TIME: 13:19
O. PC3M#15 FILE # 6 STD CURVE # 3
TION AMOUNT 0.500 UL OF STANDARD
UMENT ID MS-25
NAL STANDARD LEVELS NG PER UL
TC3
NG
E LEVELS NG PER UL
5.000
NTERNAL STANDARD SUMMARY

MAX TIC	% TIC	RATIO	REGION	MEASURED RF	MEAN RF
147449	100	0.85	6C13 TC3	32128100.00	32128100.00
173832	100				

E SUMMARY REPORT FOR STANDARD

MAX TIC	% TIC	RATIO	REGION	MEASURED RF	MEAN RF
300000	100				
600000	100	0.50*	Mono	1.12	1.12
400000	100				
500000	100	0.80	Di	1.12	1.12
540782	100				
530089	100	1.02	Tri	1.33	1.33
235373	100				
317811	100	0.74	Tetra	0.69	0.69
364219	100				
232491	100	1.57*	Penta	0.74	0.74
220765	100				
165762	100	1.33	Hexa	0.48	0.48
191956	100				
184574	100	1.04	Hepta	0.47	0.47
90000	100				
100000	100	0.90	Octa	0.24	0.24
128000	100				
84000	100	1.52*	Nano	0.26	0.26
155000	100				
136493	100	1.14	Deca	0.36	0.36

TABLE 35

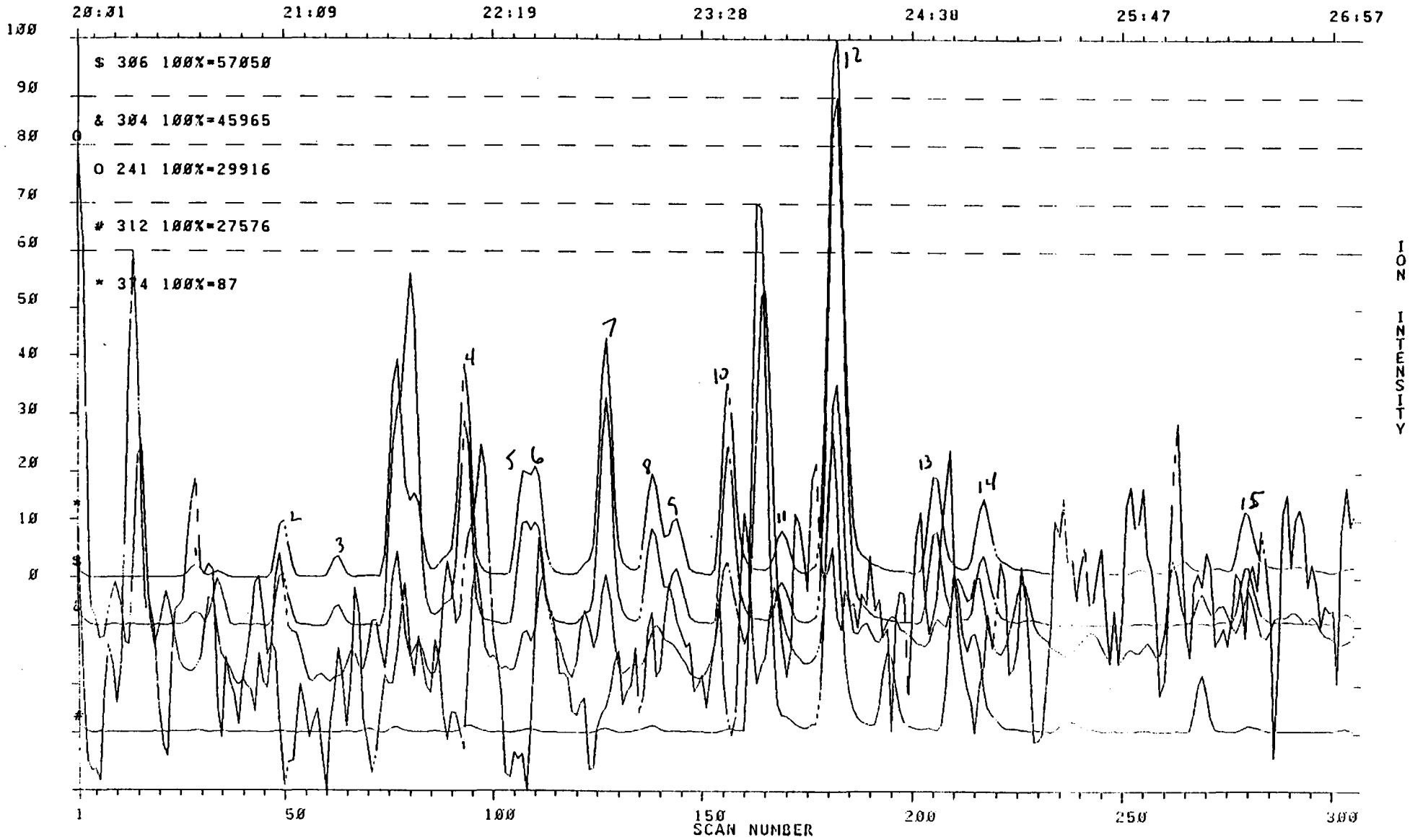
MOISTURE CONTENT OF NEW BEDFORD HARBOR AREA SEDIMENT SAMPLES RECEIVED FROM NUS CORP. AND ANALYZED FOR PCB, PCDD, AND PCDF

NUS/EPA	WSU	% Moisture
13855	NBC 5-3	59%
13858	NBC 5-1	66%
13867	NBC 5-5	55%

ATTACHMENT H

MASS CHROMATOGRAMS OBTAINED BY THE BREHM LABORATORY IN GC-MS ANALYSES OF U.S. COAST GUARD SAMPLE EXTRACTS AND OF SEDIMENTS RECEIVED FROM NUS CORP. FOR PCDD AND PCDF

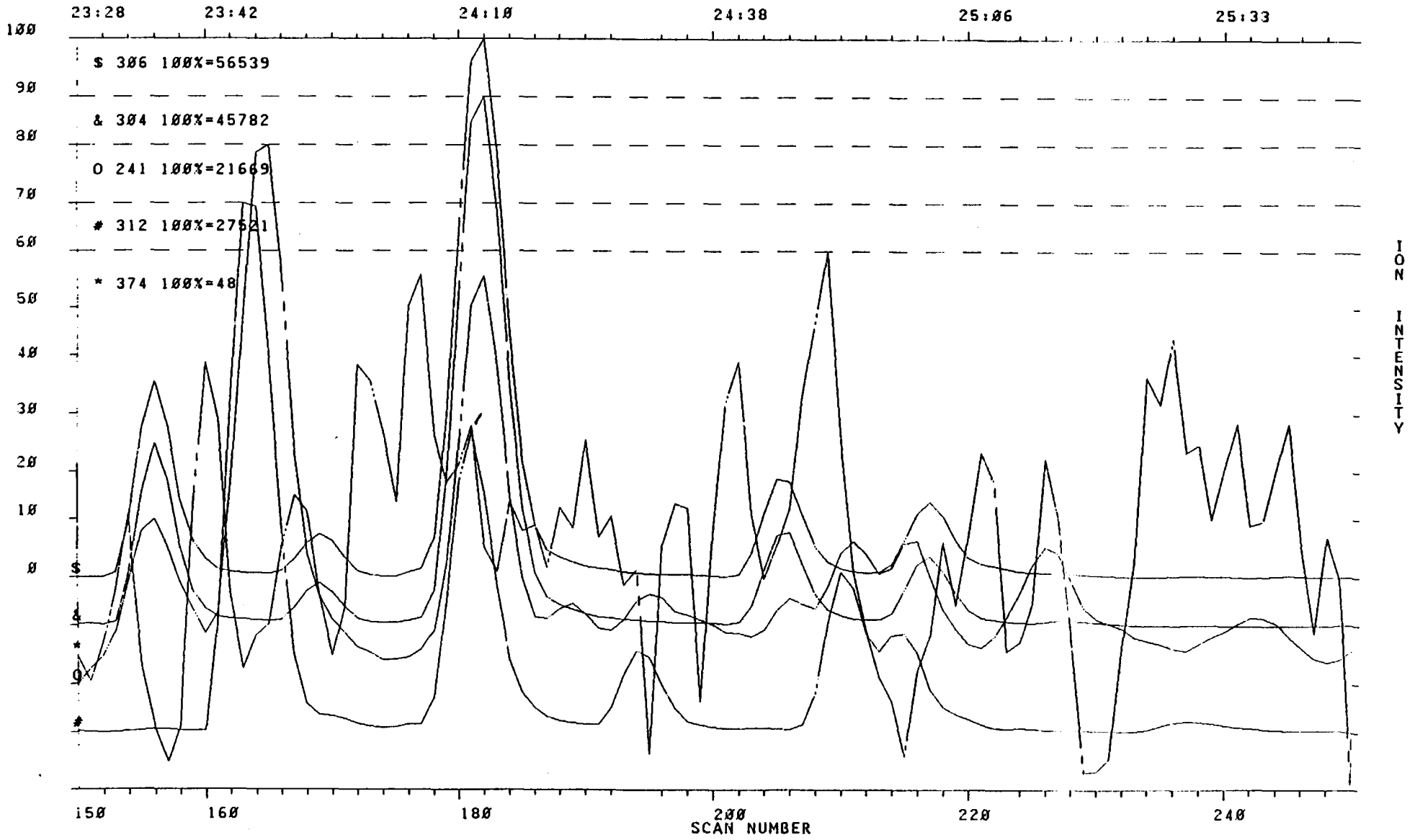
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.

FIGURE: 1

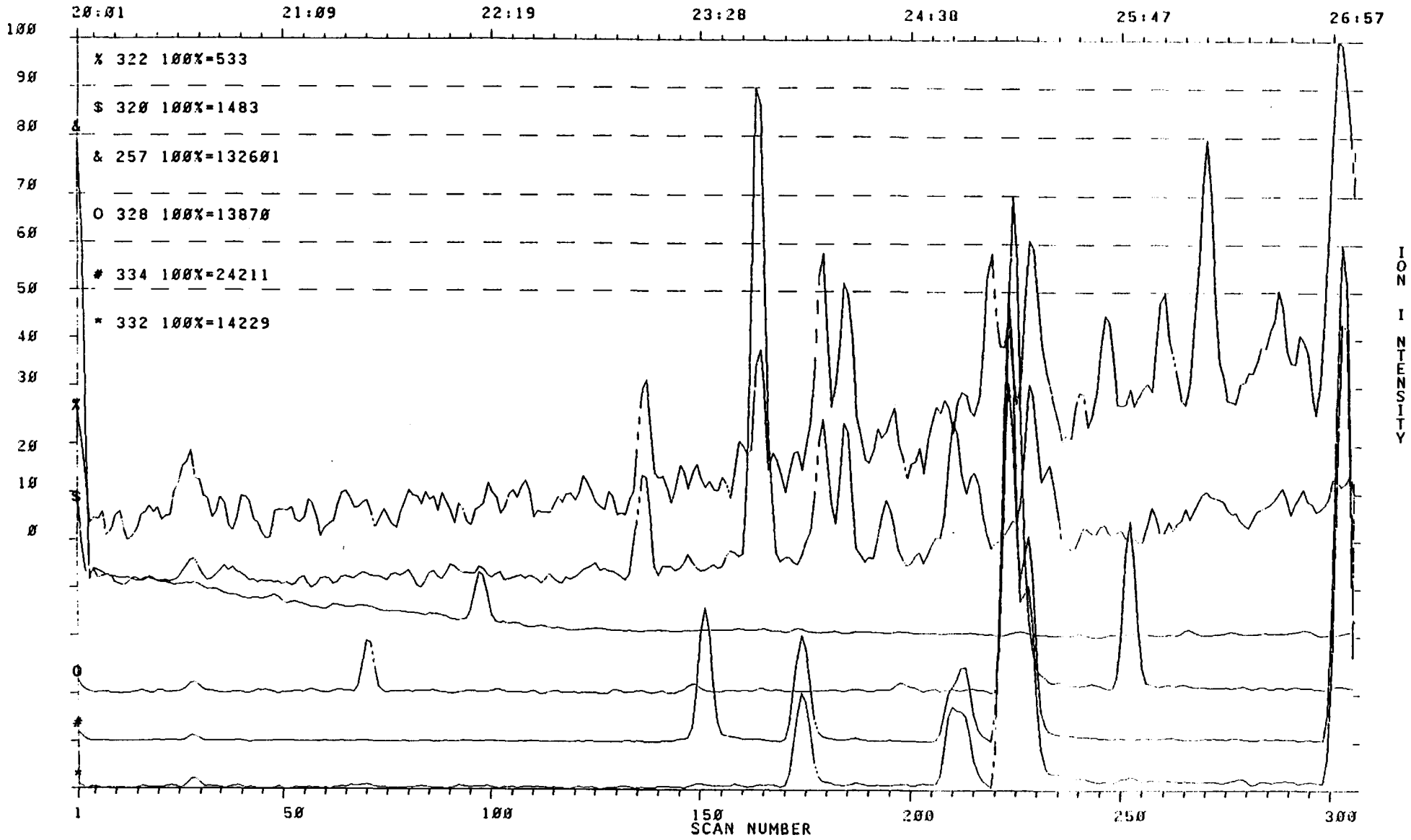
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.

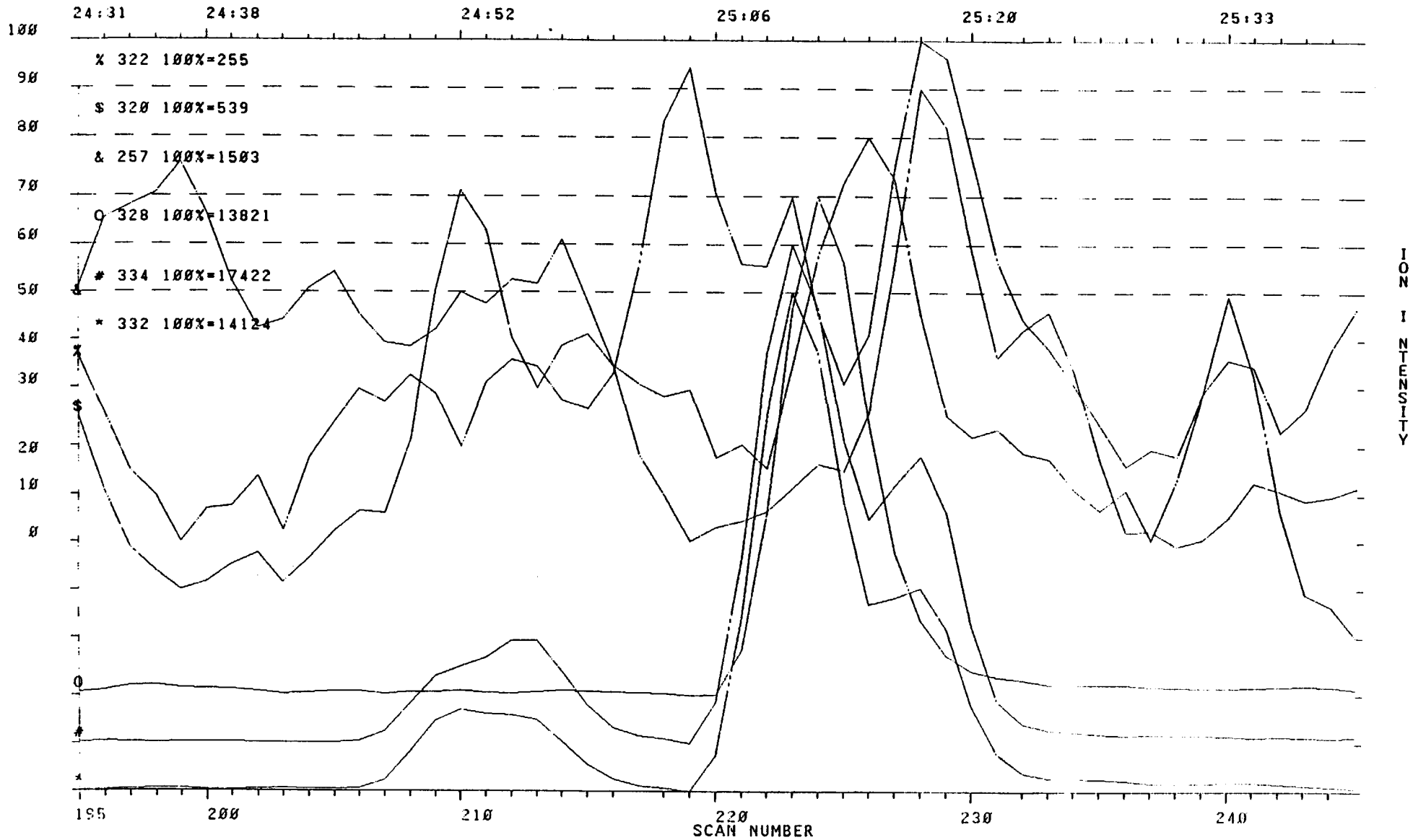
FIGURE: 2

KRATOS MS25, DS55 SOFTWARE, RUN: CTO40080, WSU NAME: USC6-1
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZO-P-DIOXINS



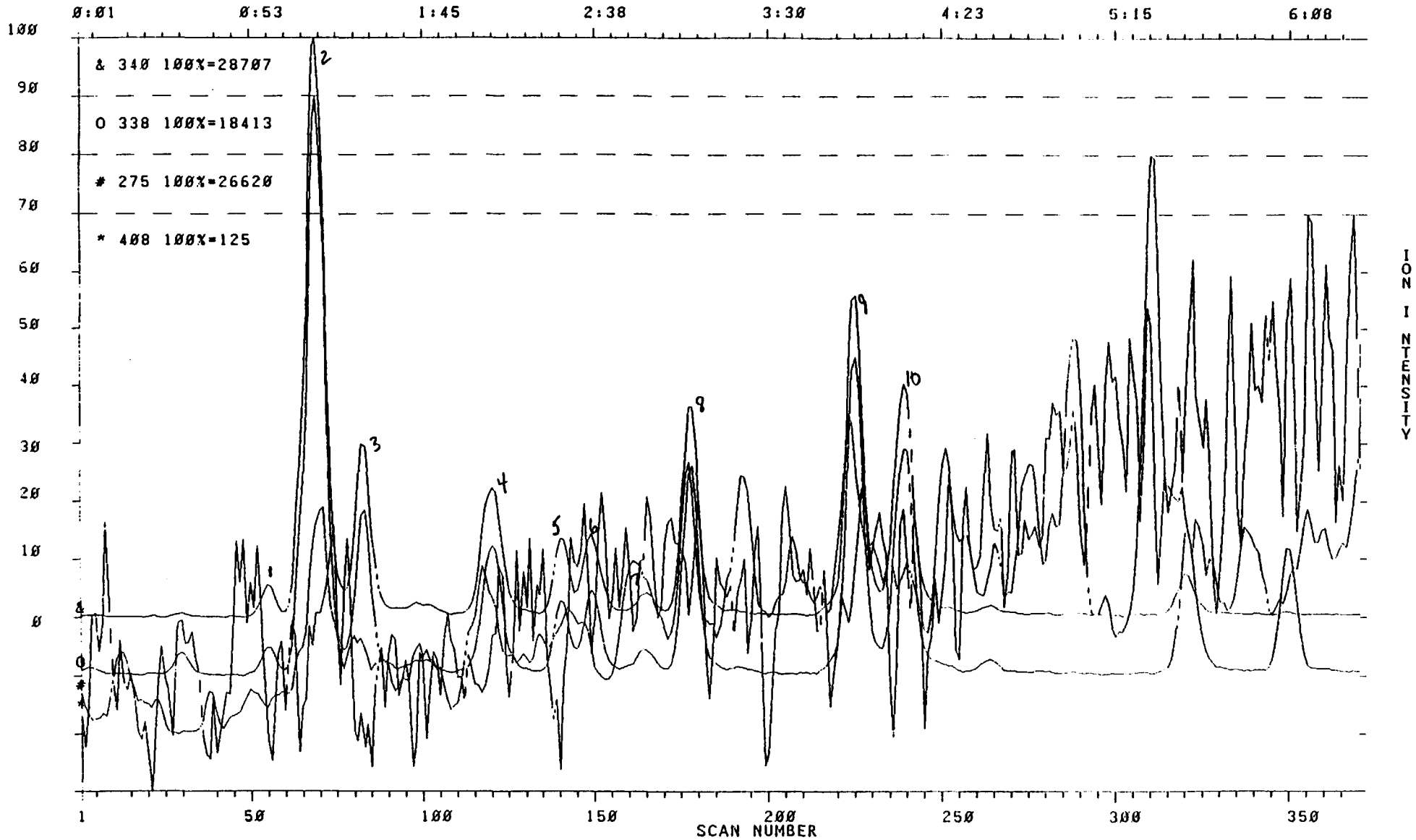
HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.
FIGURE: 3

KRATOS MS25, DS55 SOFTWARE, RUN: CTO40080, WSU NAME: USC6-1
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.
FIGURE: 4

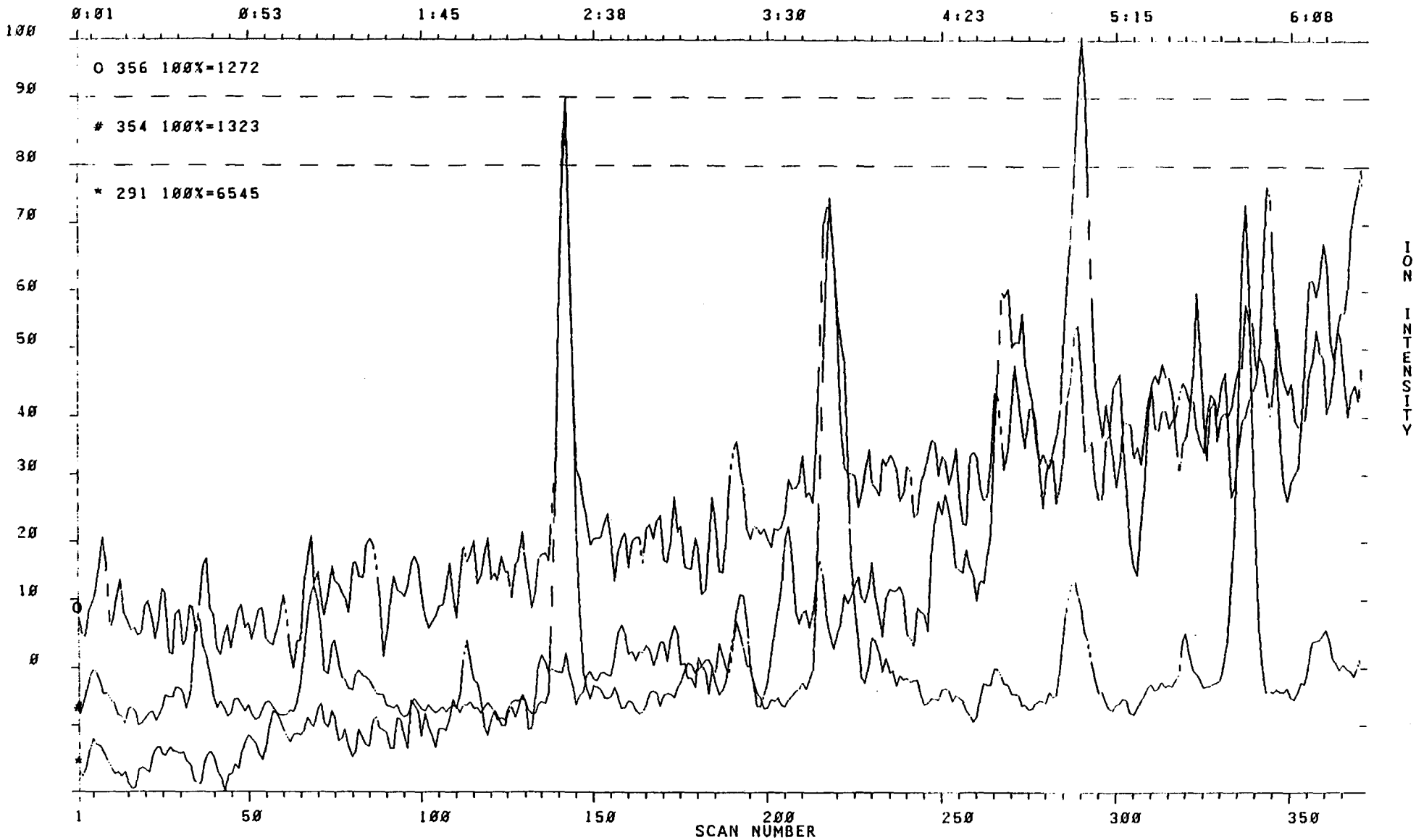
KRATOS MS25, DS55 SOFTWARE, RUN: CTO5008Ø, WSU NAME: USC6-1
SELECTED-ION MASS CHROMATOGRAMS FOR PENTACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.

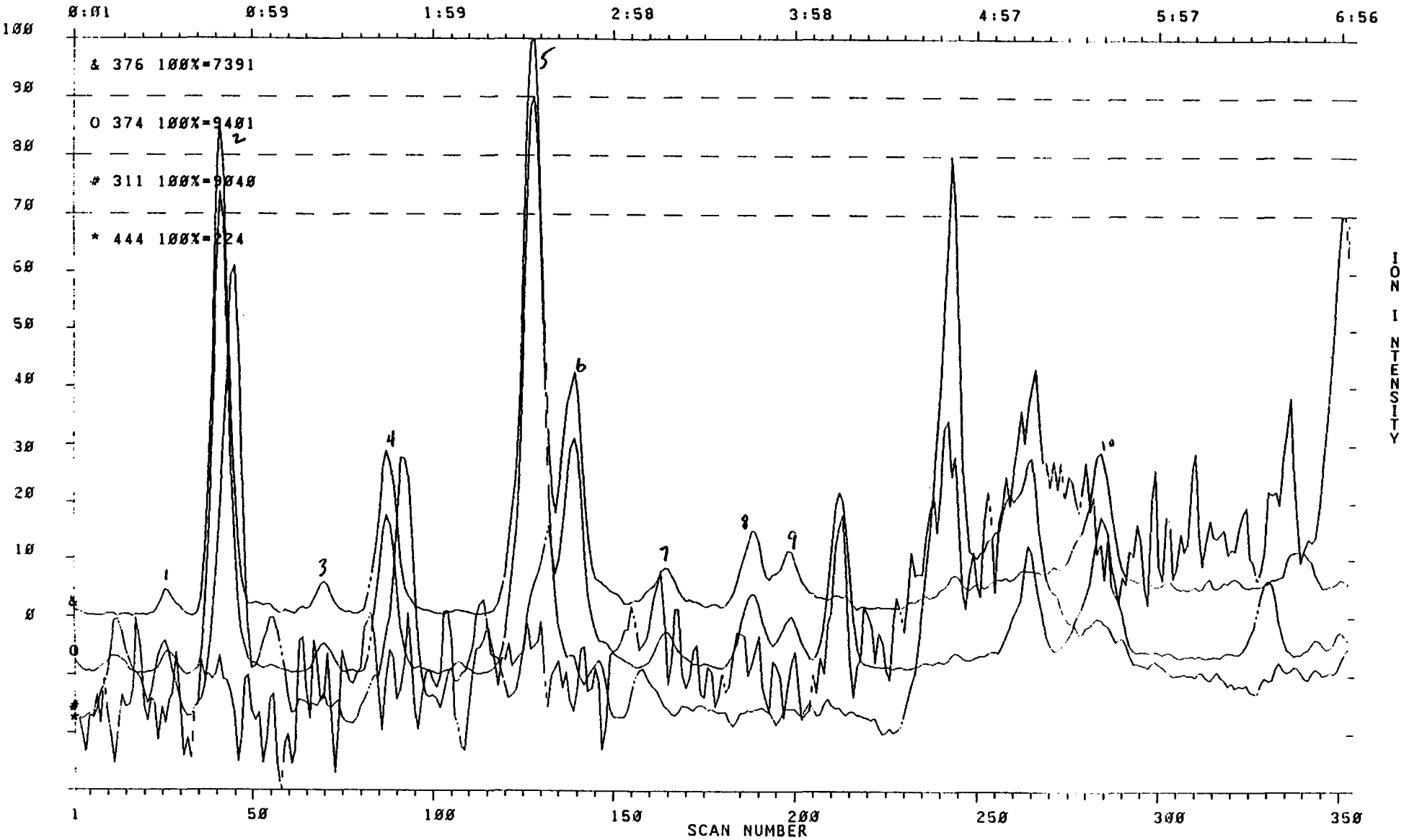
FIGURE: 5

KRATOS MS25, DS55 SOFTWARE, RUN: CTO5ØØ8Ø, WSU NAME: USC6-1
SELECTED-ION MASS CHROMATOGRAMS FOR PENTACHLORODIBENZO-P-DIOXINS



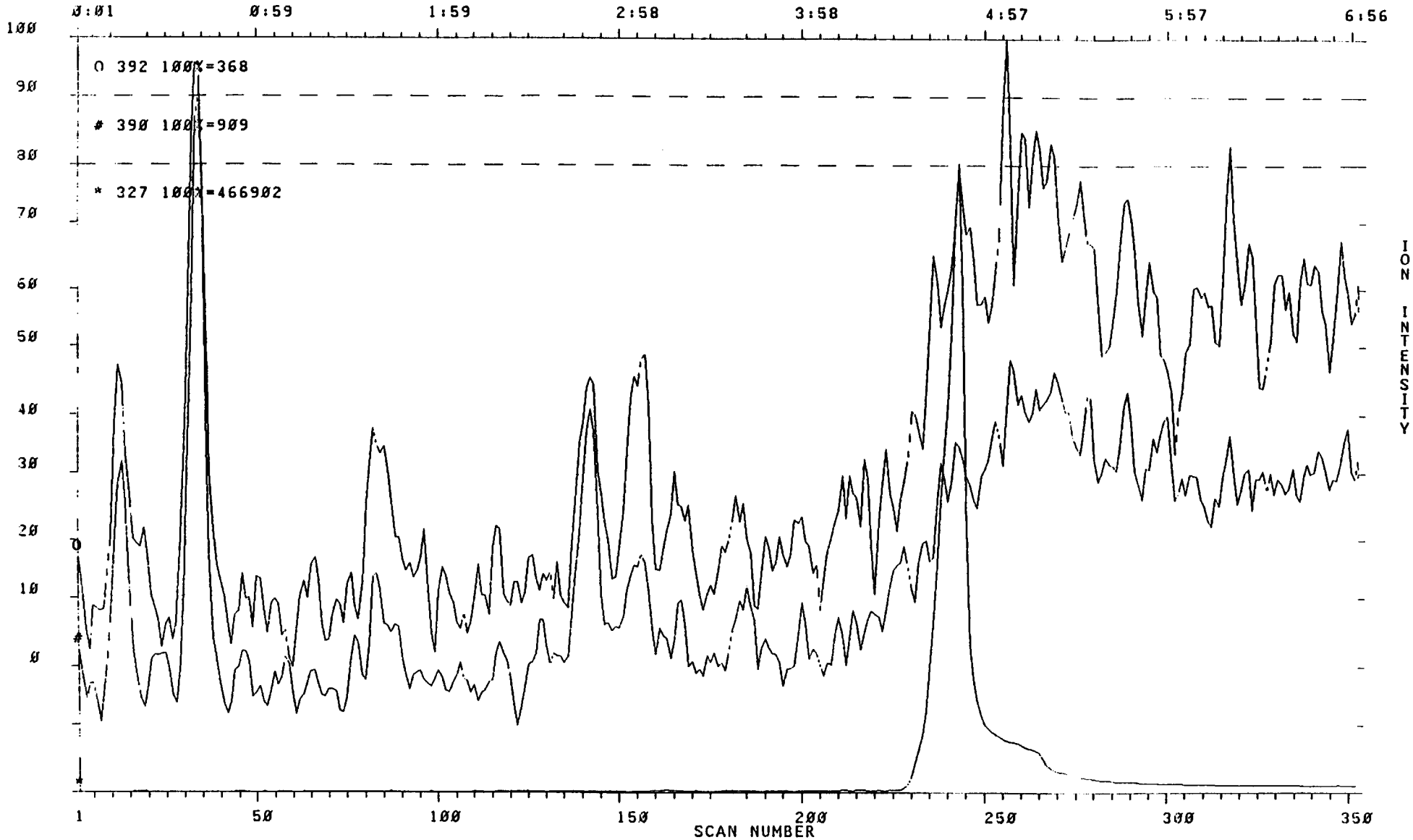
HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.
FIGURE: 6

KRATOS MS25, DS55 SOFTWARE, RUN: CTO6ØØ8Ø, WSU NAME: USC6-1
SELECTED-ION MASS CHROMATOGRAMS FOR HEXACHLORODIBENZOFURANS

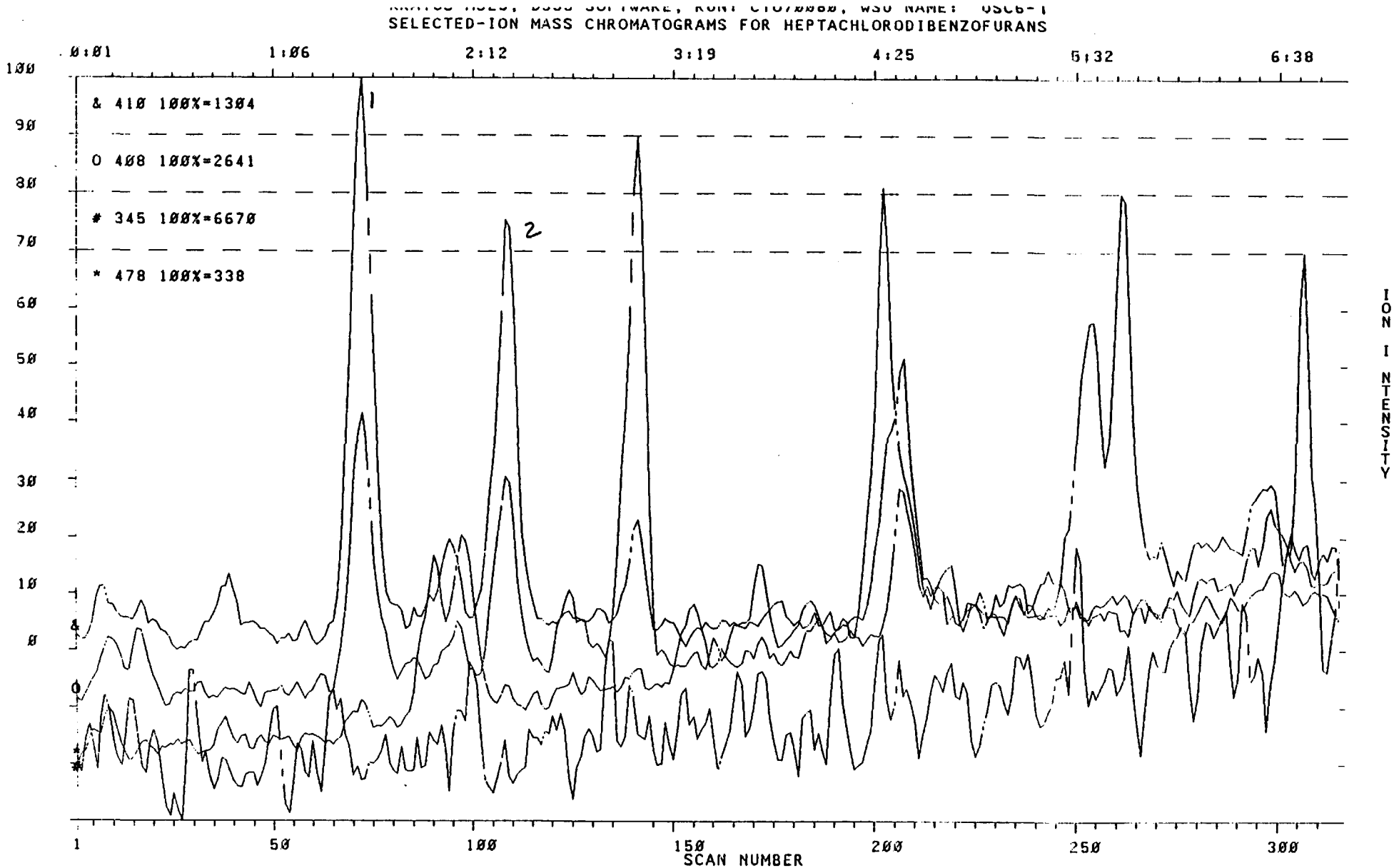


HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.
FIGURE: 7

KRATOS MS25, DS55 SOFTWARE, RUN: CTO60080, WSU NAME: USC6-\
SELECTED-ION MASS CHROMATOGRAMS FOR HEXACHLORODIBENZO-P-DIOXINS



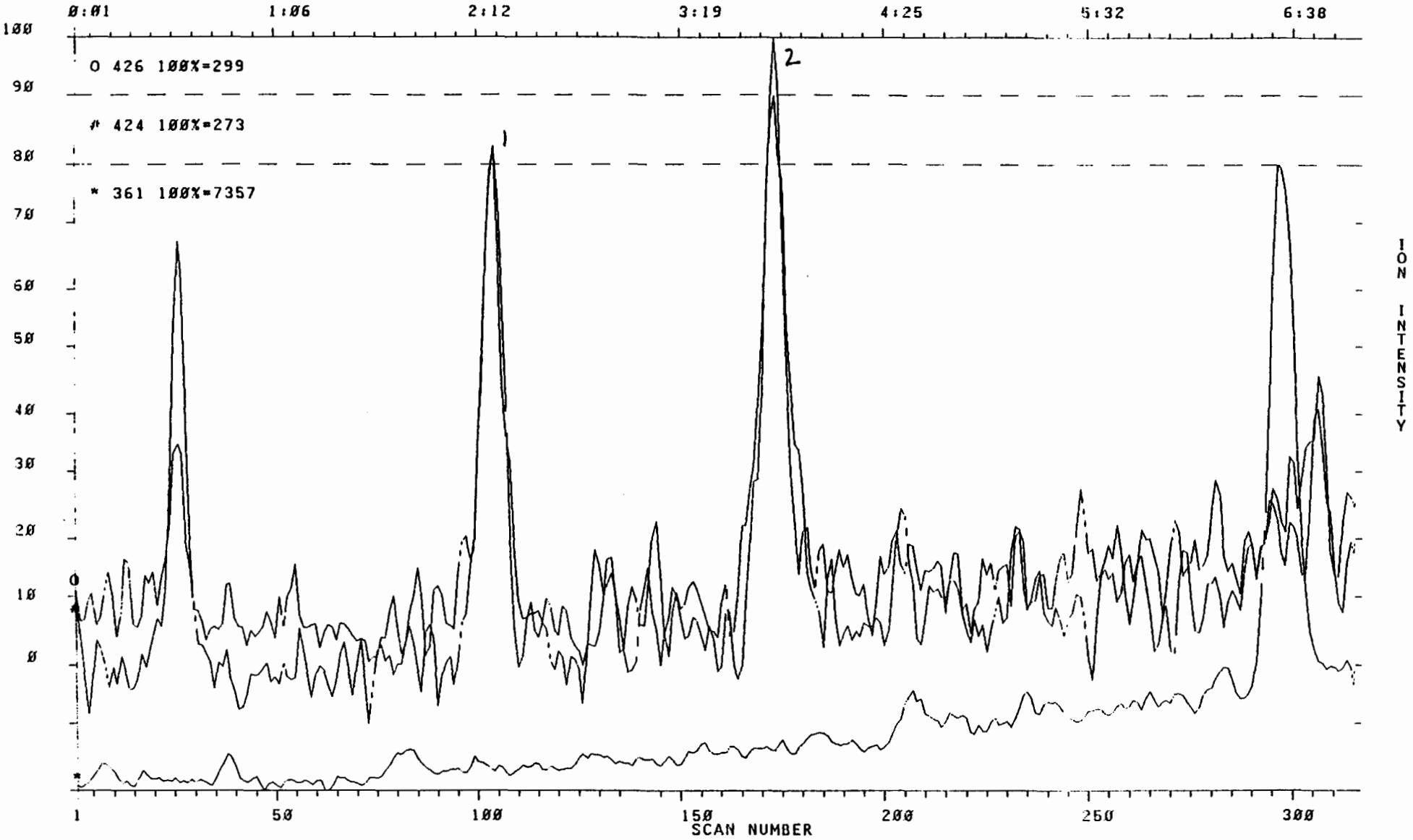
HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.
FIGURE: 8



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.

FIGURE: 9

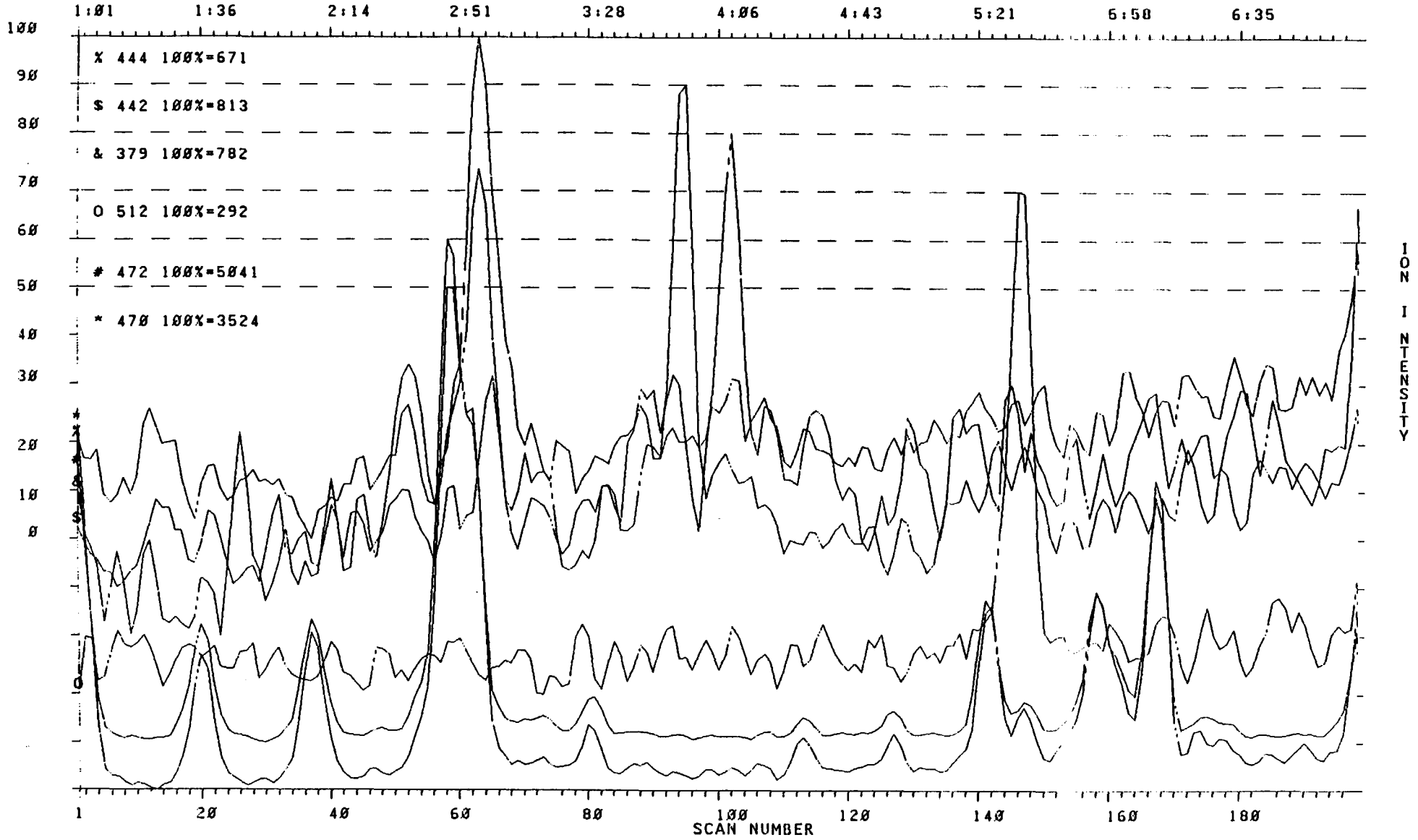
SELECTED-ION MASS CHROMATOGRAMS FOR HEPTACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.

FIGURE:10

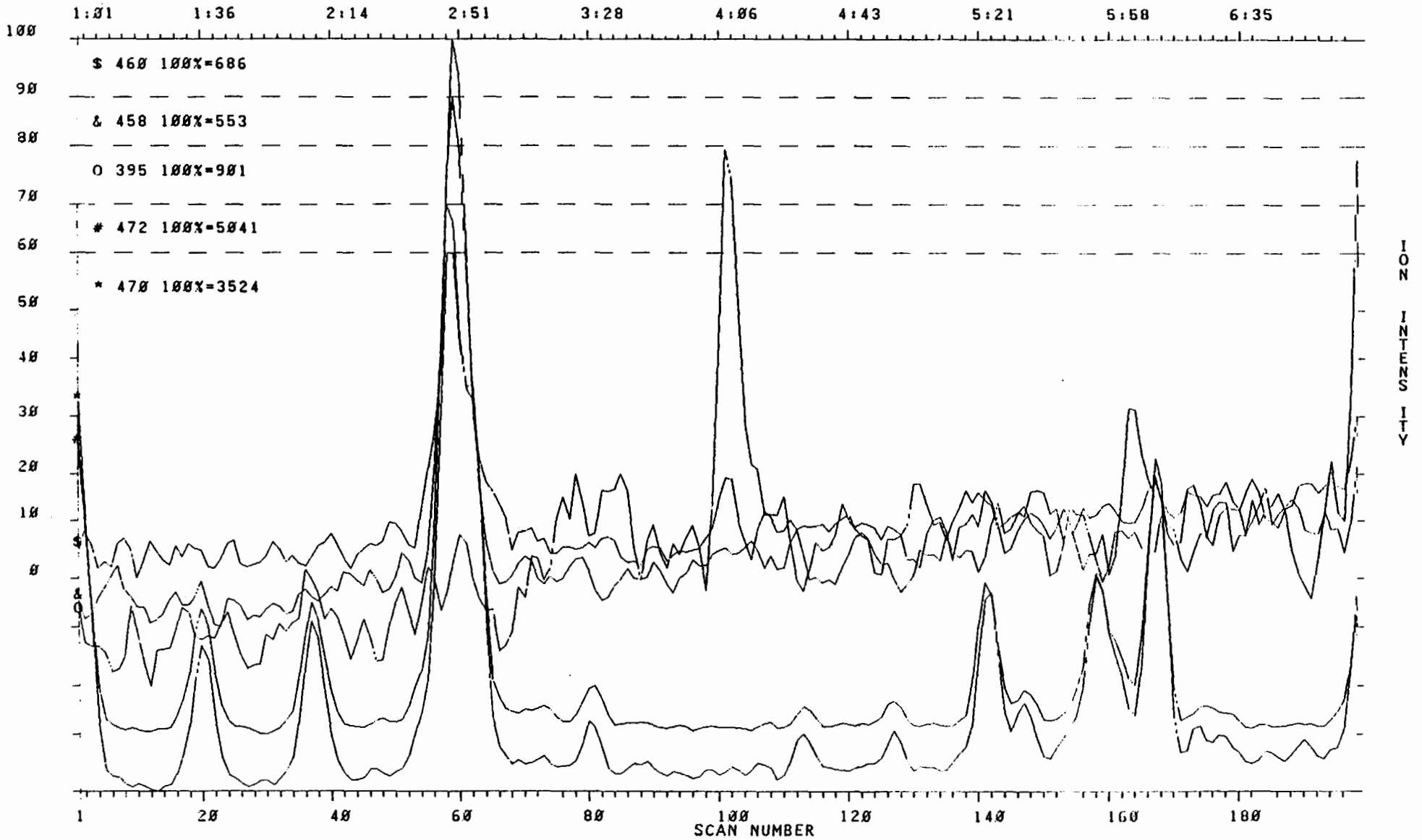
KRATOS MS25, DS55 SOFTWARE, RUN: CTO8ØØ8Ø, WSU NAME: USC6-1
SELECTED-ION MASS CHROMATOGRAMS FOR OCTACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.

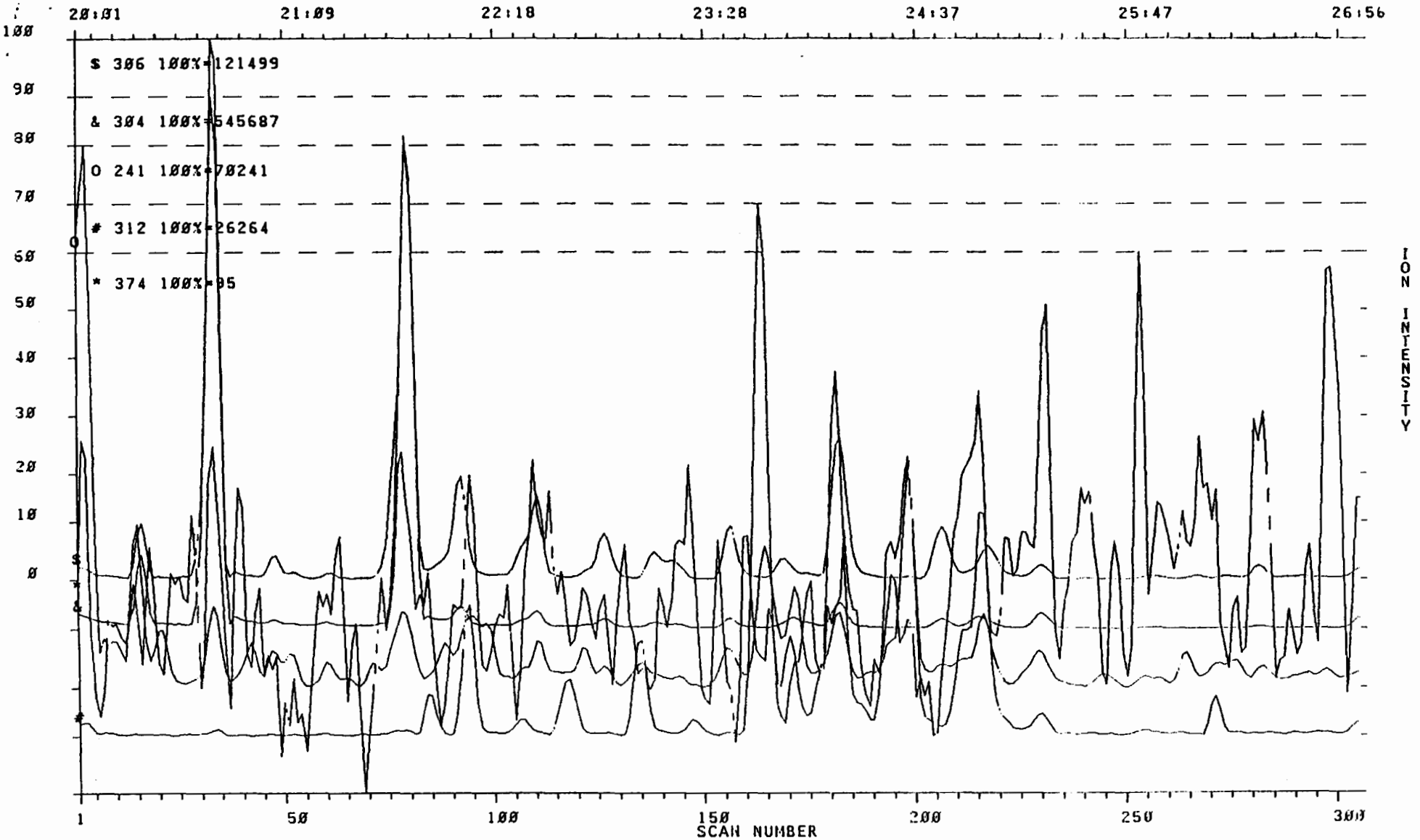
FIGURE: 11

KRATOS MS25, DS55 SOFTWARE, RUN: CTO8ØØ8Ø, WSU NAME: USC6-\
SELECTED-ION MASS CHROMATOGRAMS FOR OCTACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.
FIGURE: 12

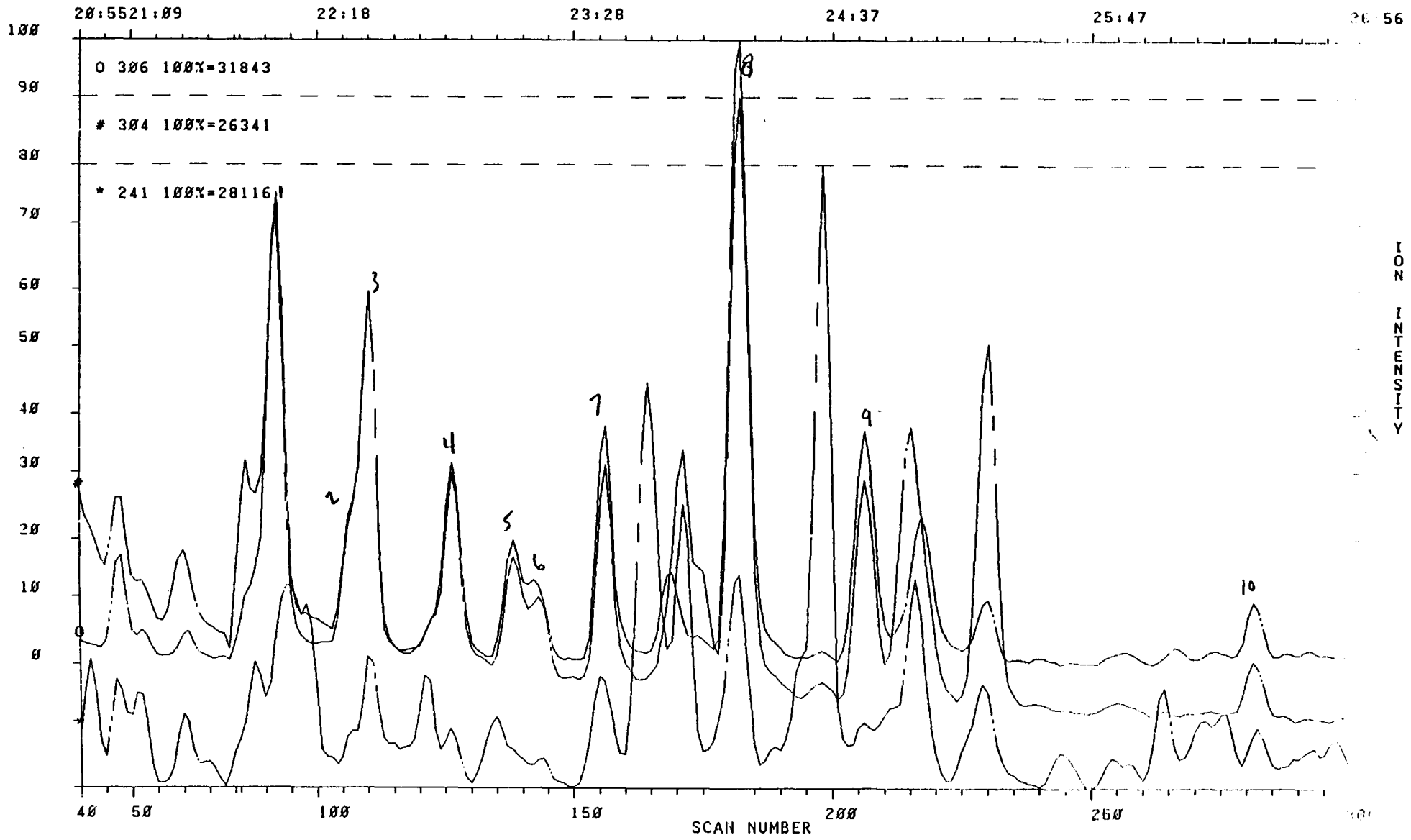
KRATOS MS25, DS55 SOFTWARE, RUN: CTO40081, WSU NAME: USC6-2
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 9 (B,C)

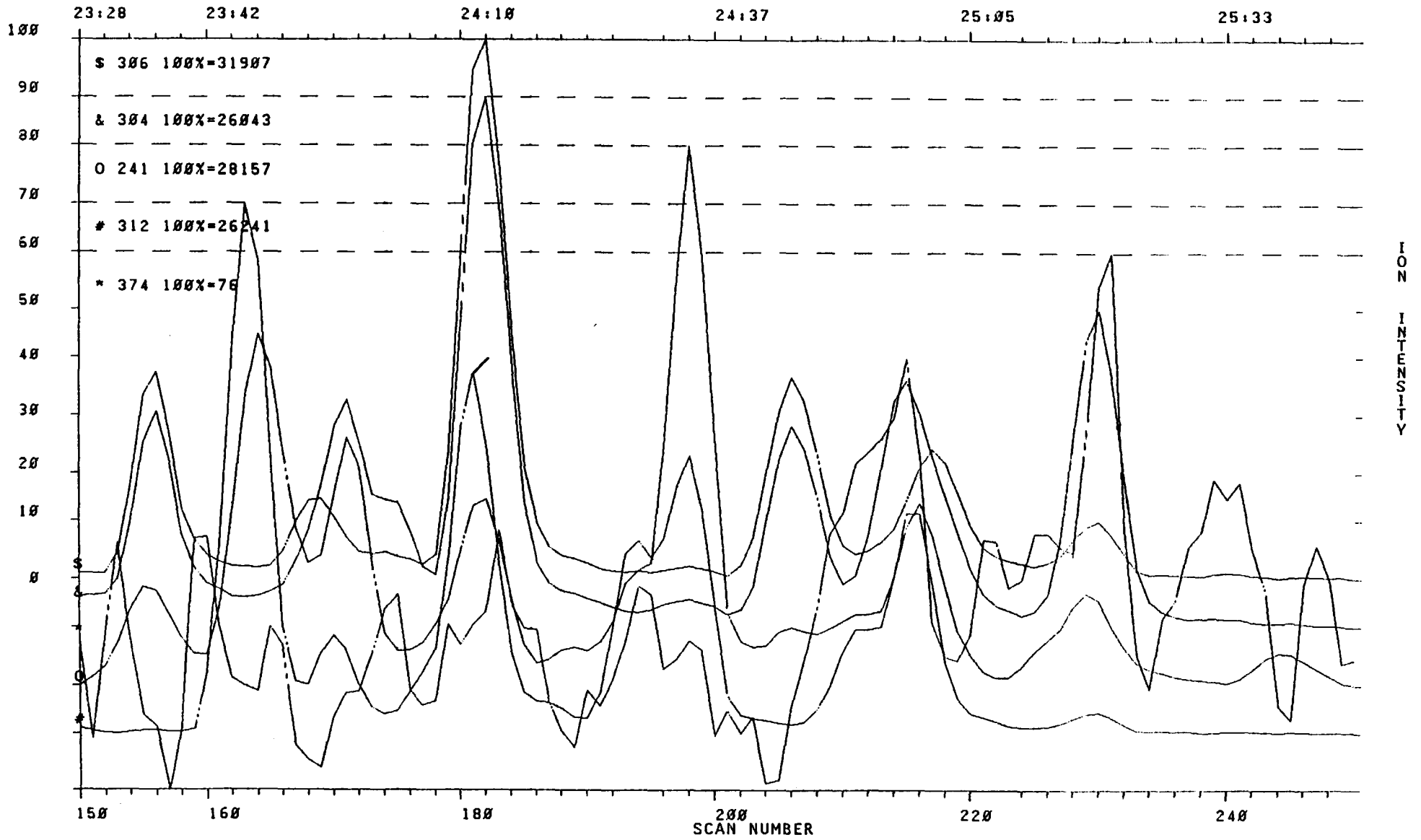
FIGURE: 13

KRATOS MS25, DS55 SOFTWARE, RUN: CTO4ØØ81, WSU NAME: USC6-2
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZOFURANS



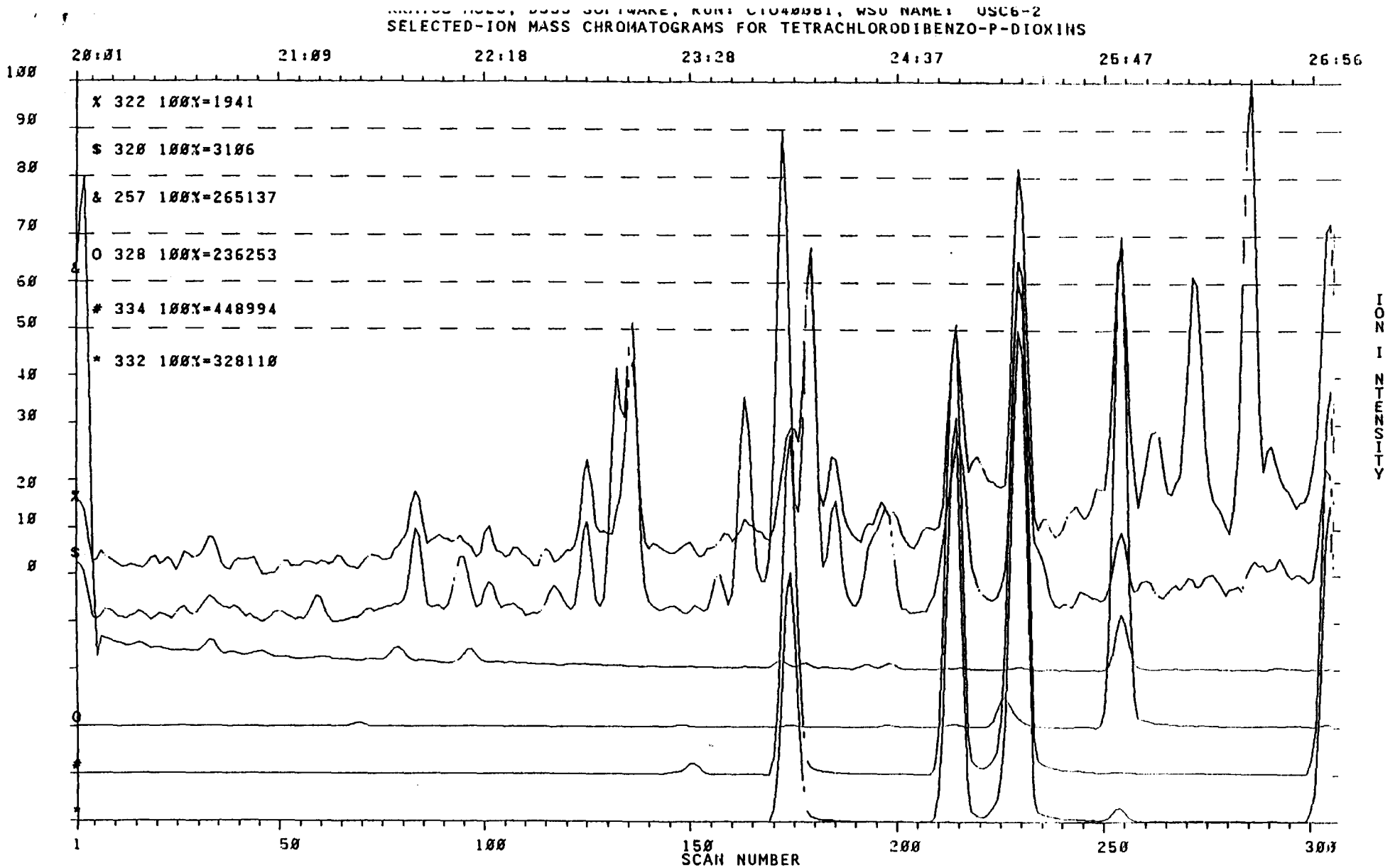
HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.9 (B,C)
FIGURE: 14

KRATOS MS25, DS58 SOFTWARE, RUN: CT04000001, WSU NAME: USC6-2
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 9 (B,C)

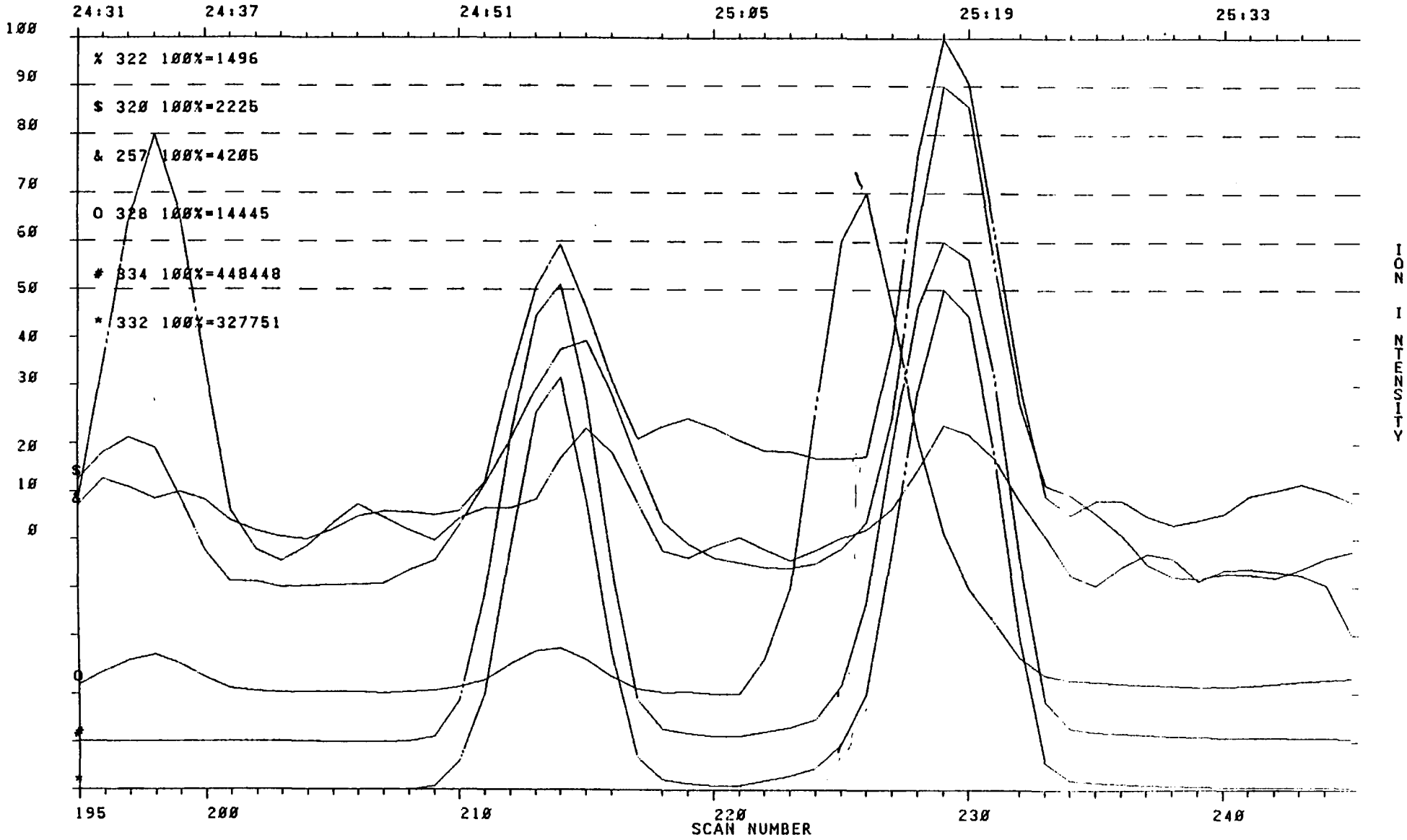
FIGURE: 15



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 9 (B,C)

FIGURE: 16

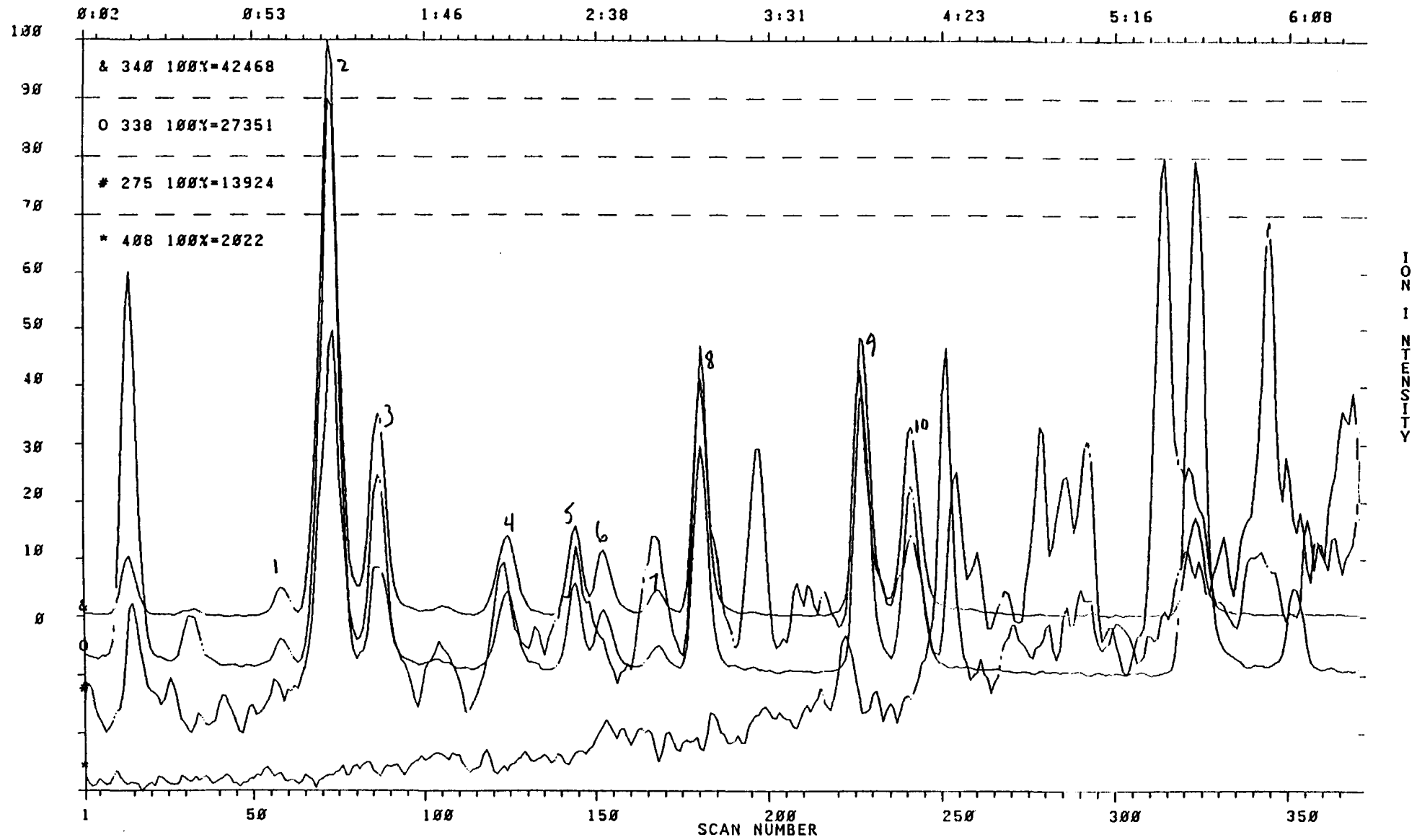
KRATOS MS25, DS55 SOFTWARE, RUN: CTO40081, WSU NAME: USC6-2
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 9 (B,C)

FIGURE: 17

KRATOS MS25, DS55 SOFTWARE, RUN: CTO50081, WSU NAME: USC6-2
SELECTED-ION MASS CHROMATOGRAMS FOR PENTACHLORODIBENZOFURANS

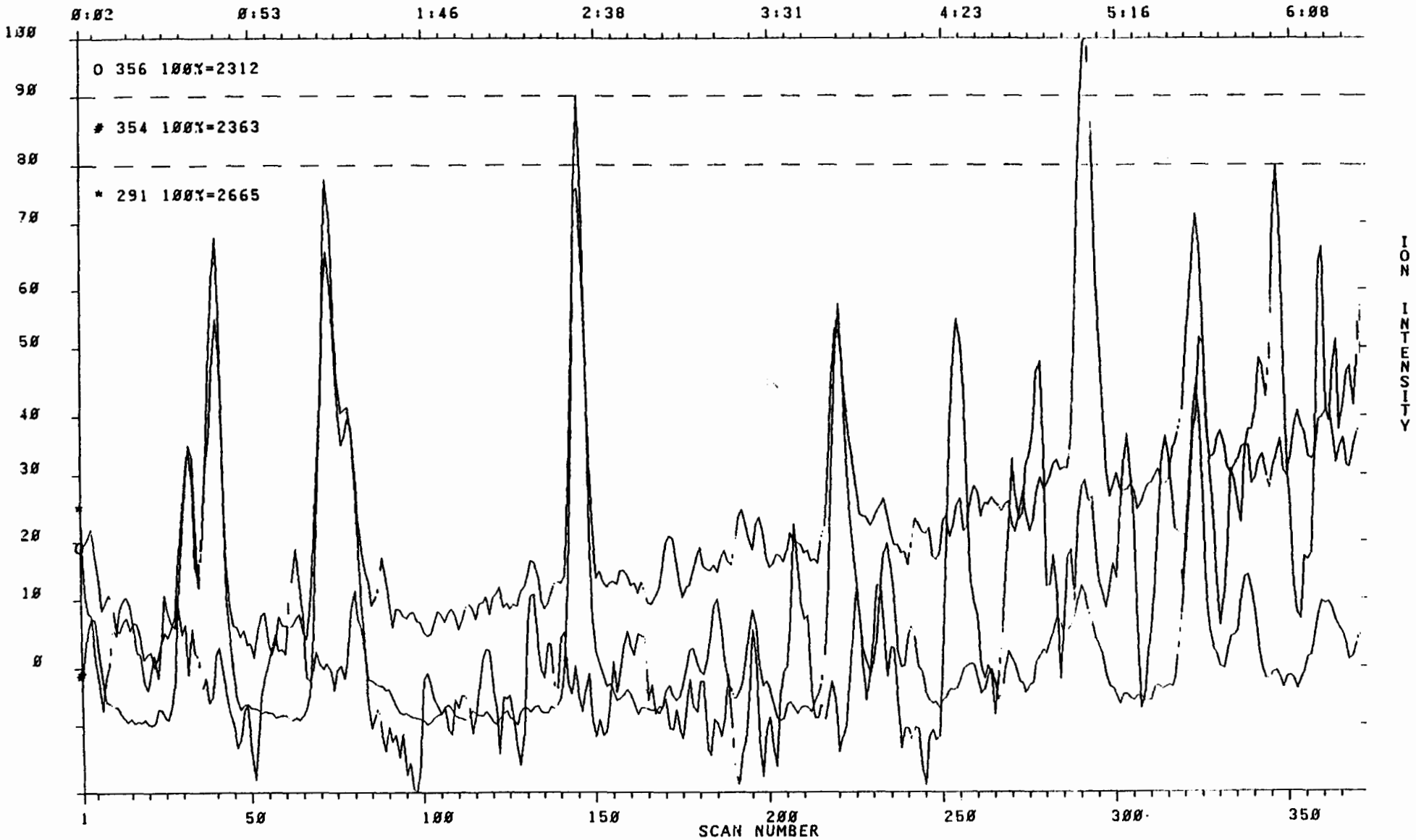


HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 9 (B,C)

FIGURE: 18

KRATOS MS25, DSS SOFTWARE, RUN: CT050081, WSO NAME: OSC6-2

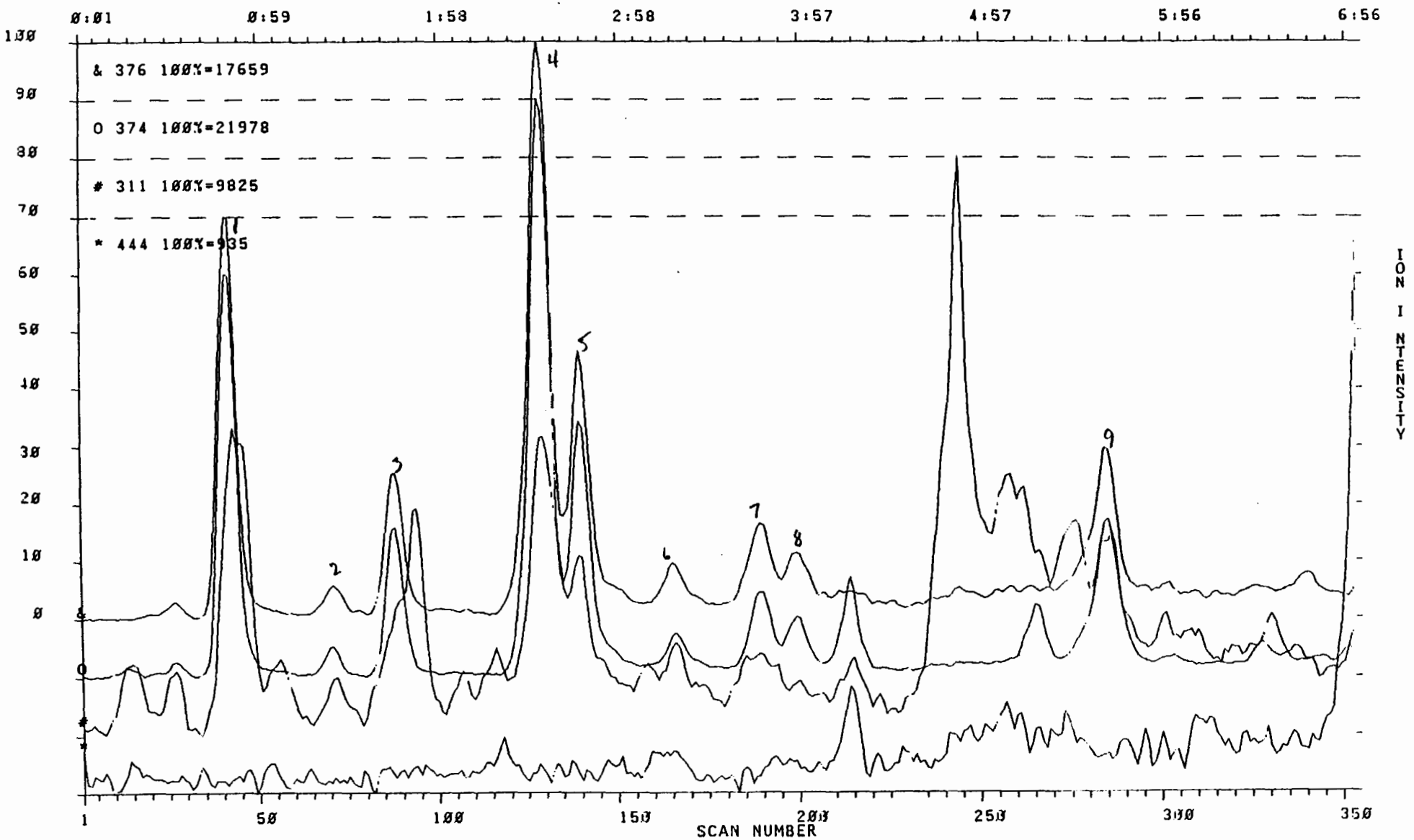
SELECTED-ION MASS CHROMATOGRAMS FOR PENTACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 9 (B,C)

FIGURE: 19

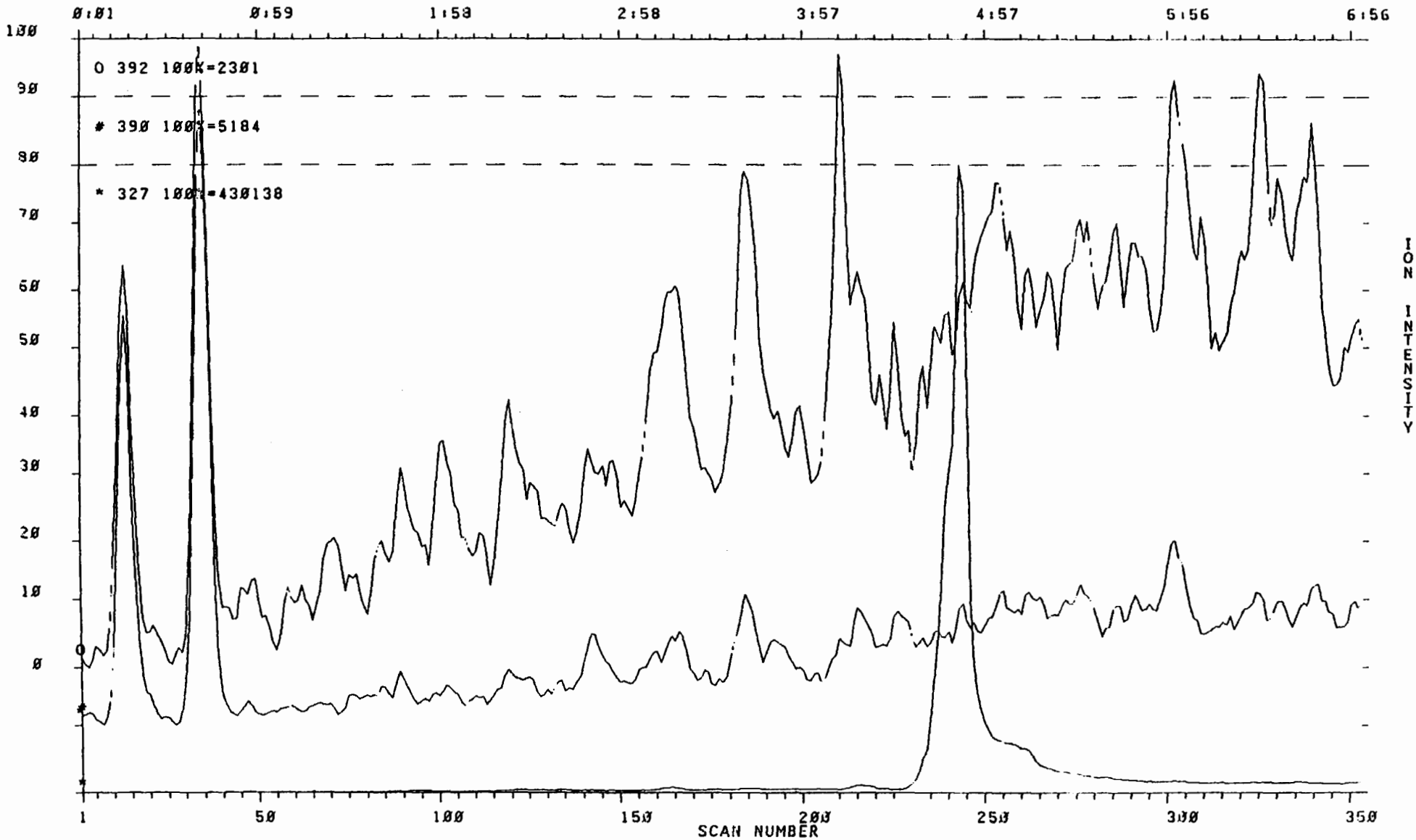
KRATOS MS25, DS55 SOFTWARE, RUN: C106Ø081, WSU NAME: USC6-2
SELECTED-ION MASS CHROMATOGRAMS FOR HEXACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 9 (B,C)

FIGURE: 20

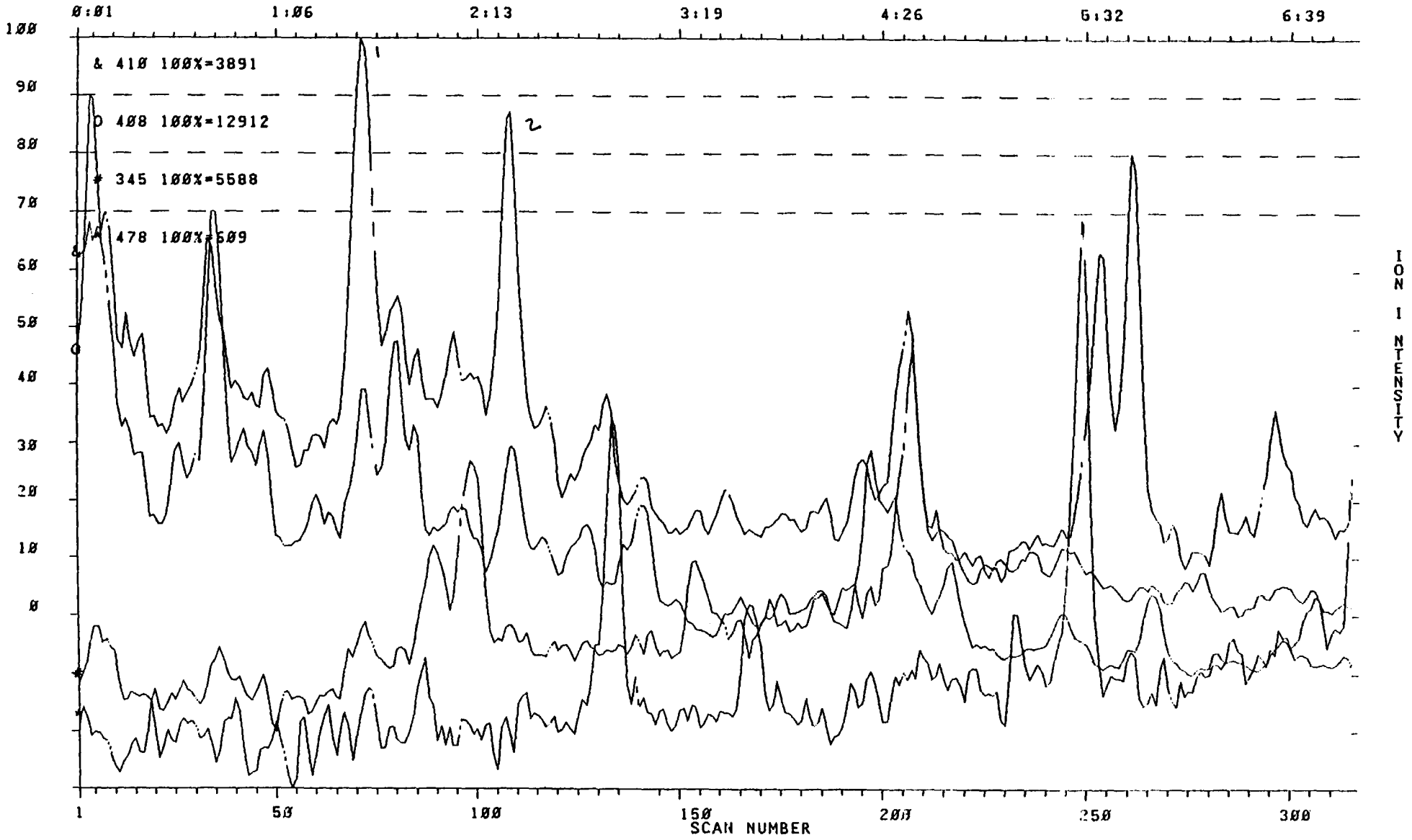
KRATOS MS25, DS55 SOFTWARE, RUN: CTO60081, WSU NAME: USC6-2
SELECTED-ION MASS CHROMATOGRAMS FOR HEXACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 9 (B,C)

FIGURE: 21

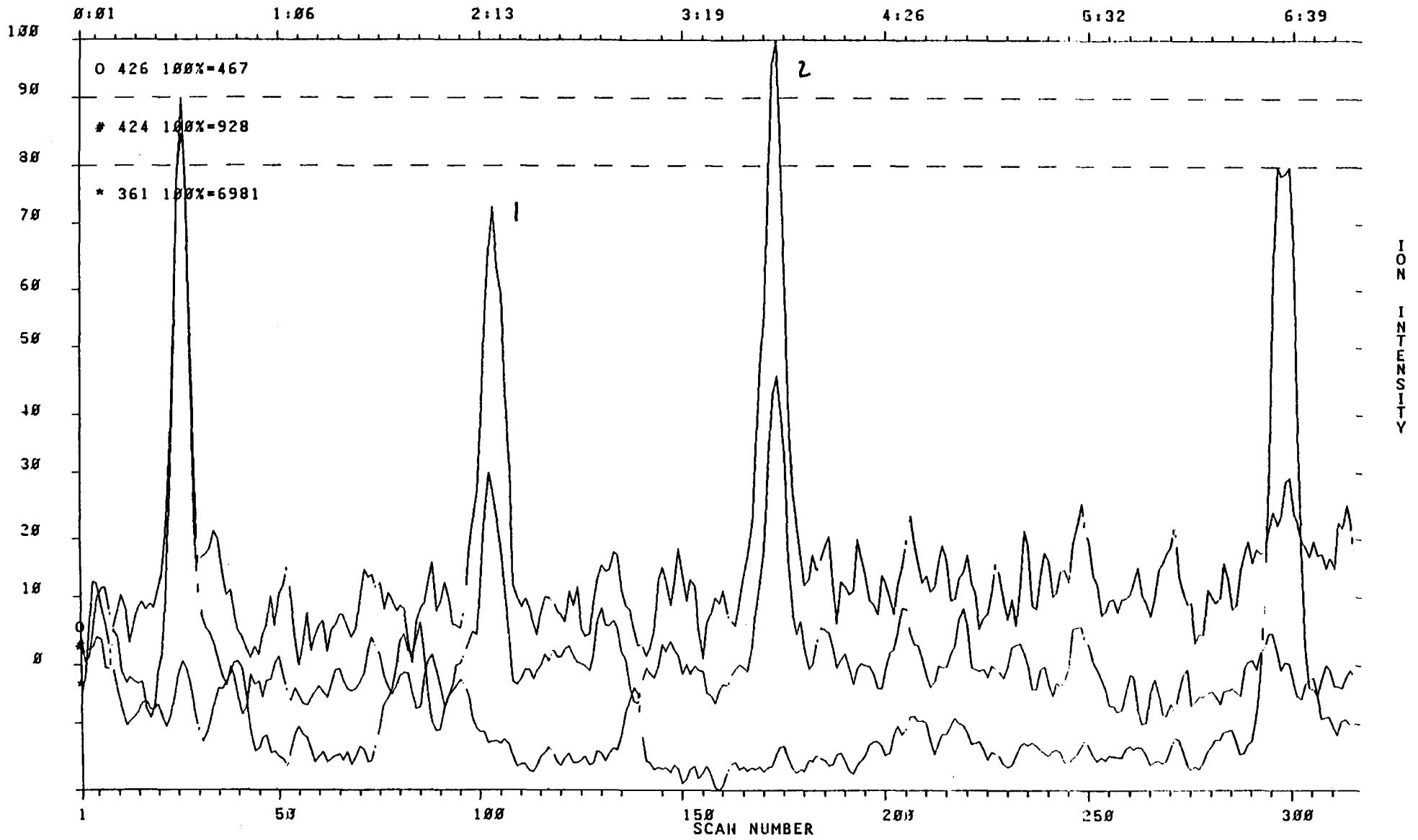
KRATOS MS25, DS55 SOFTWARE, RUN: CTO70081, WSU NAME: USC6-2
SELECTED-ION MASS CHROMATOGRAMS FOR HEPTACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 9 (B,C)

FIGURE: 22

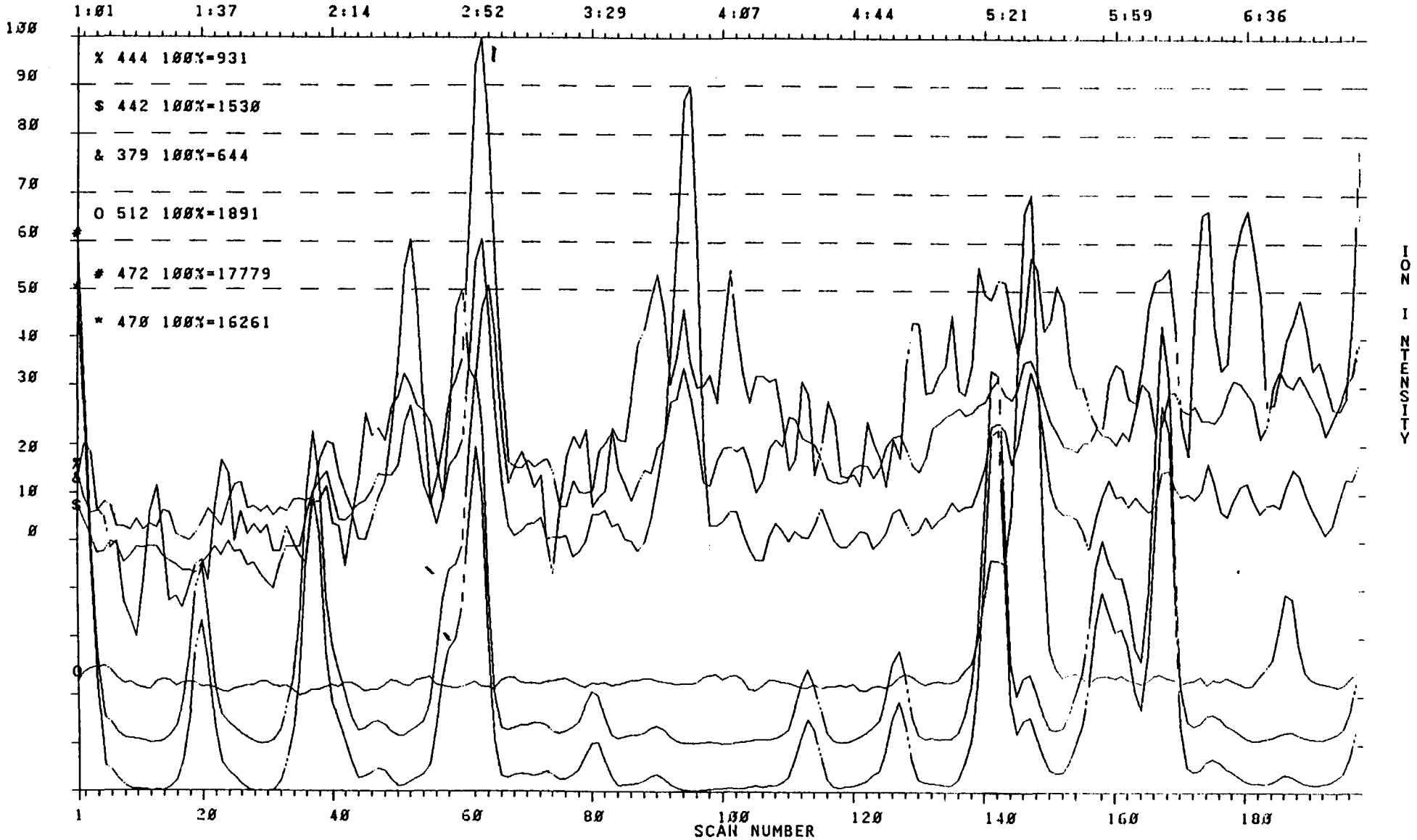
KRATOS MS25, DS55 SOFTWARE, RUN: CT070081, WSU NAME: USC6-2
SELECTED-ION MASS CHROMATOGRAMS FOR HEPTACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 9 (B,C)

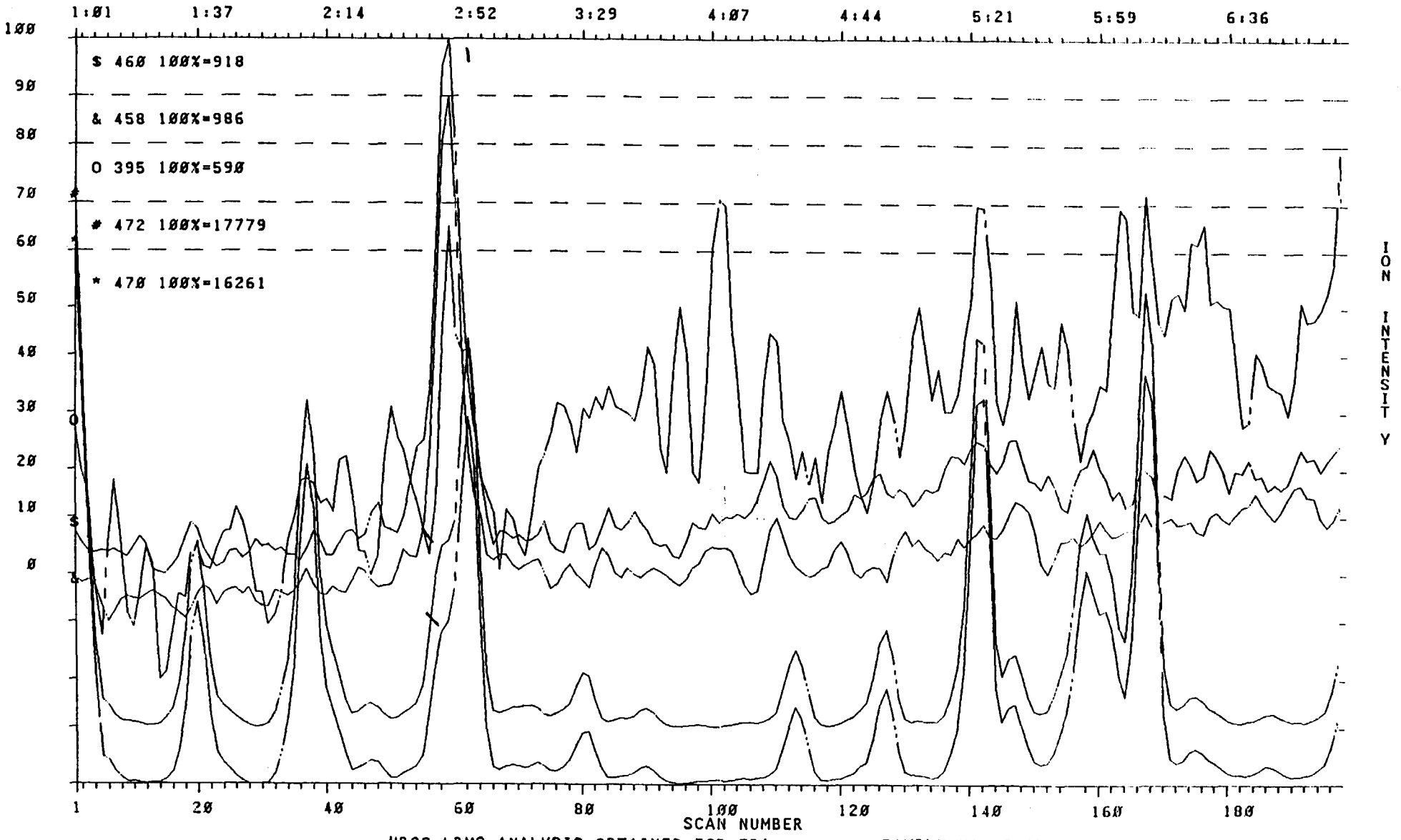
FIGURE: 23

KRATOS MS25, DS55 SOFTWARE, RUN: CTO80081, WSU NAME: USC6-2
SELECTED-ION MASS CHROMATOGRAMS FOR OCTACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 9 (B,C)
FIGURE: 24

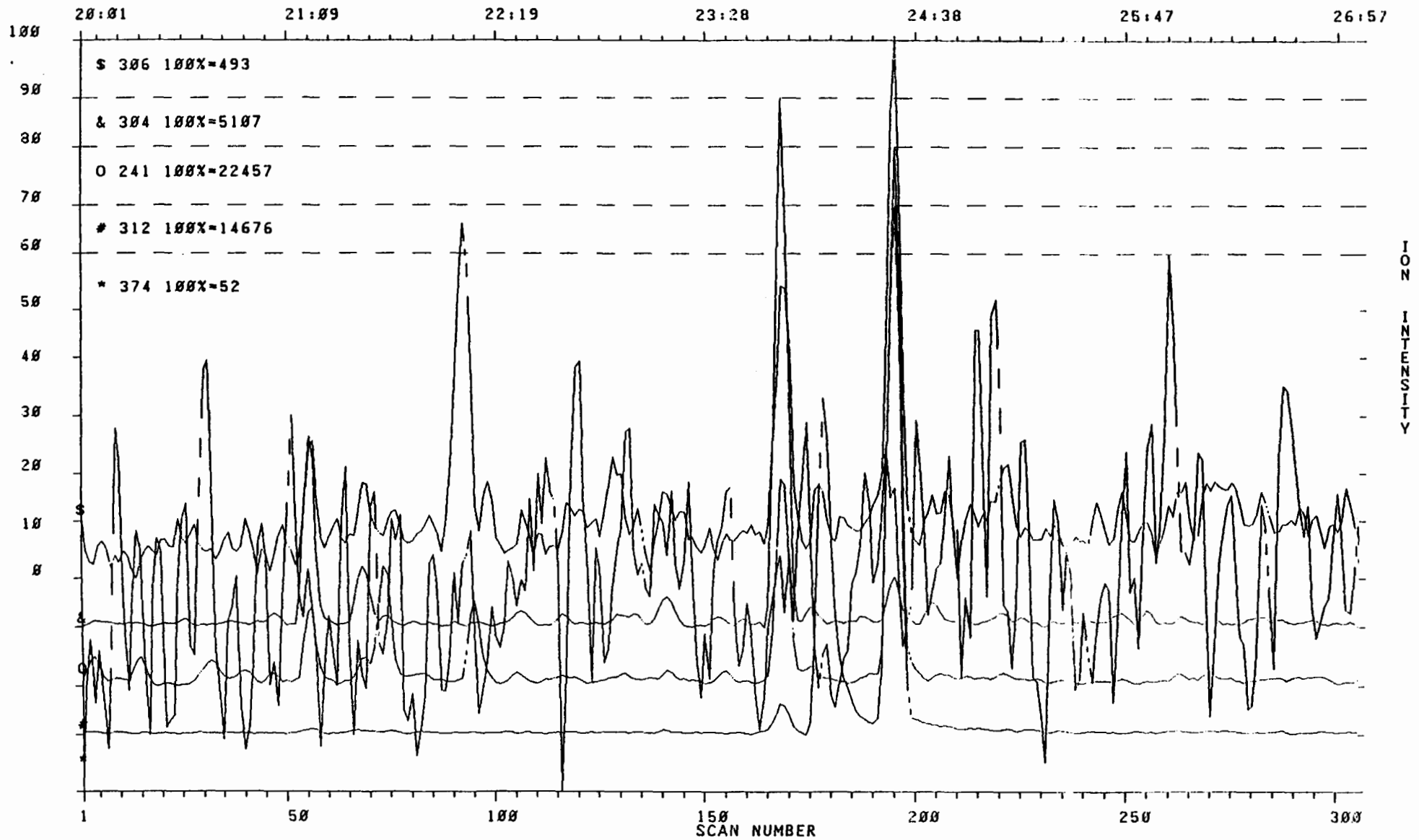
KRATOS MS25, DS55 SOFTWARE, RUN: CTO8ØØ81, WSU NAME: USC6-2
SELECTED-ION MASS CHROMATOGRAMS FOR OCTACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 9 (B,C)

FIGURE:25

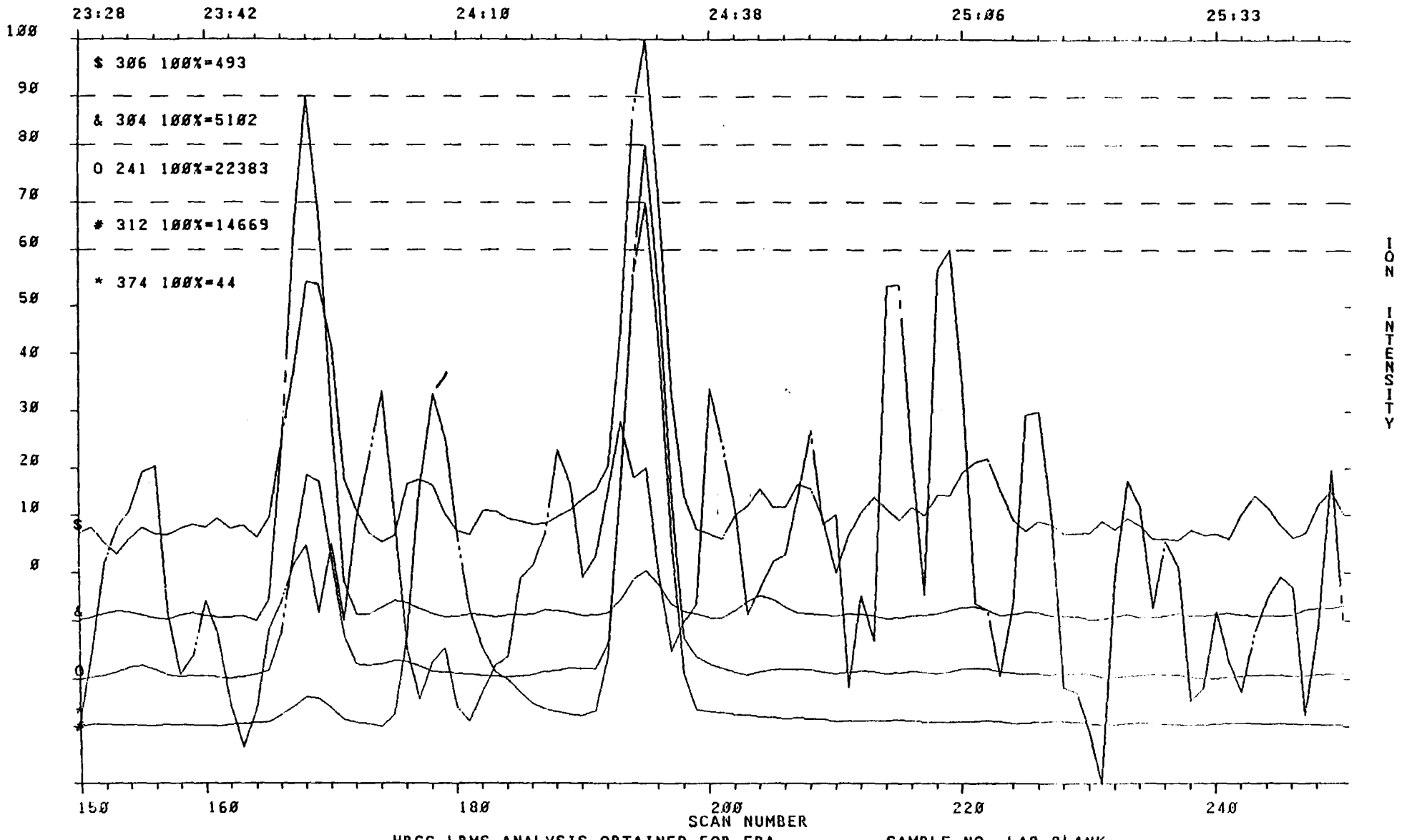
KRATOS MS25, DS55 SOFTWARE, RUN: CTO4ØØ82, WSU NAME: USC6-ØB
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK
FIGURE: 26

KRATOS MS25, DS55 SOFTWARE, RUN: CTO40082, WSU NAME: USC6-0B

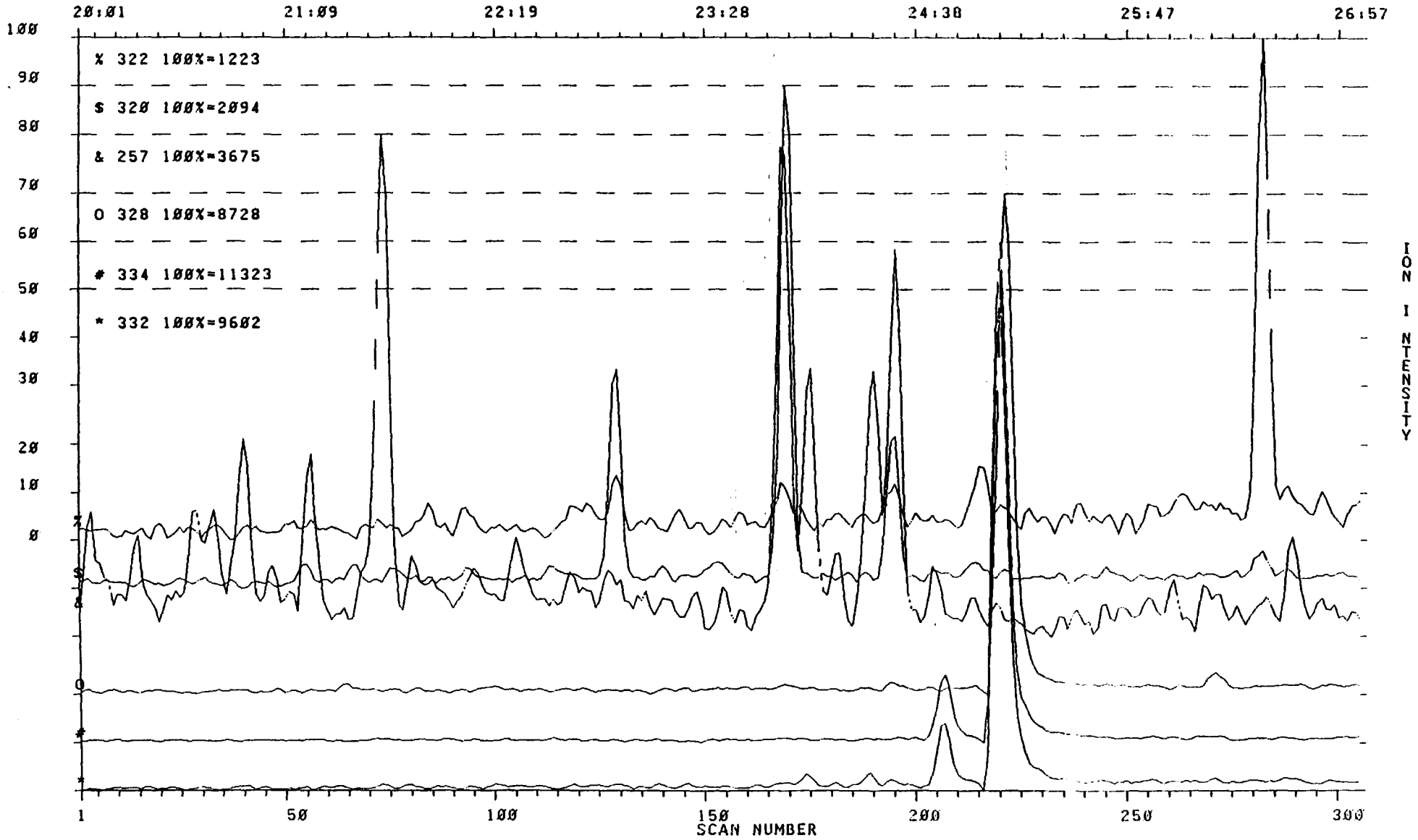
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

FIGURE: 27

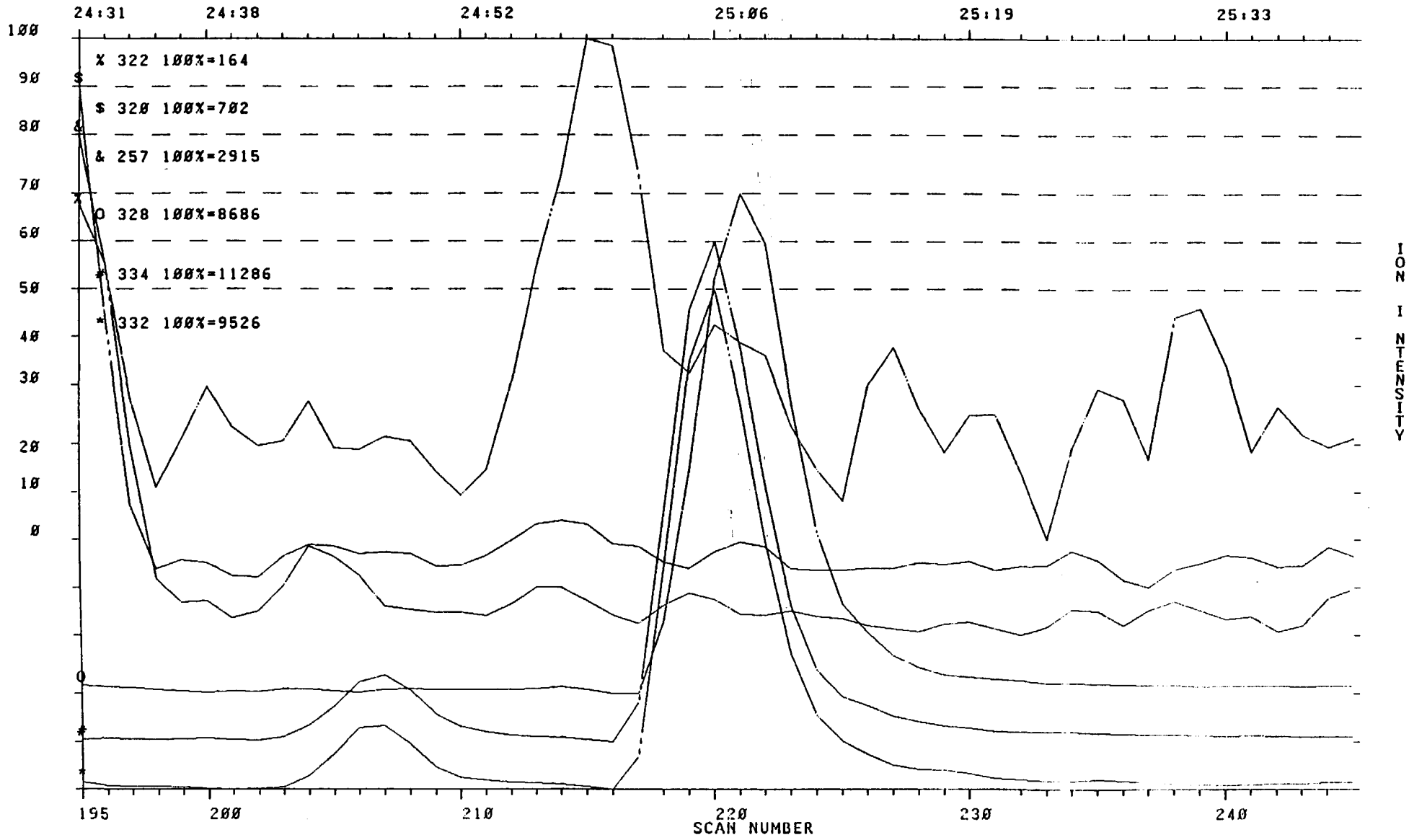
KRATOS MS25, DS55 SOFTWARE, RUN: CTO40082, WSU NAME: USC6-0B
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

FIGURE:28

KRATOS MS25, DS55 SOFTWARE, RUN: CTO4ØØ82, WSU NAME: USC6-ØB
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZO-P-DIOXINS

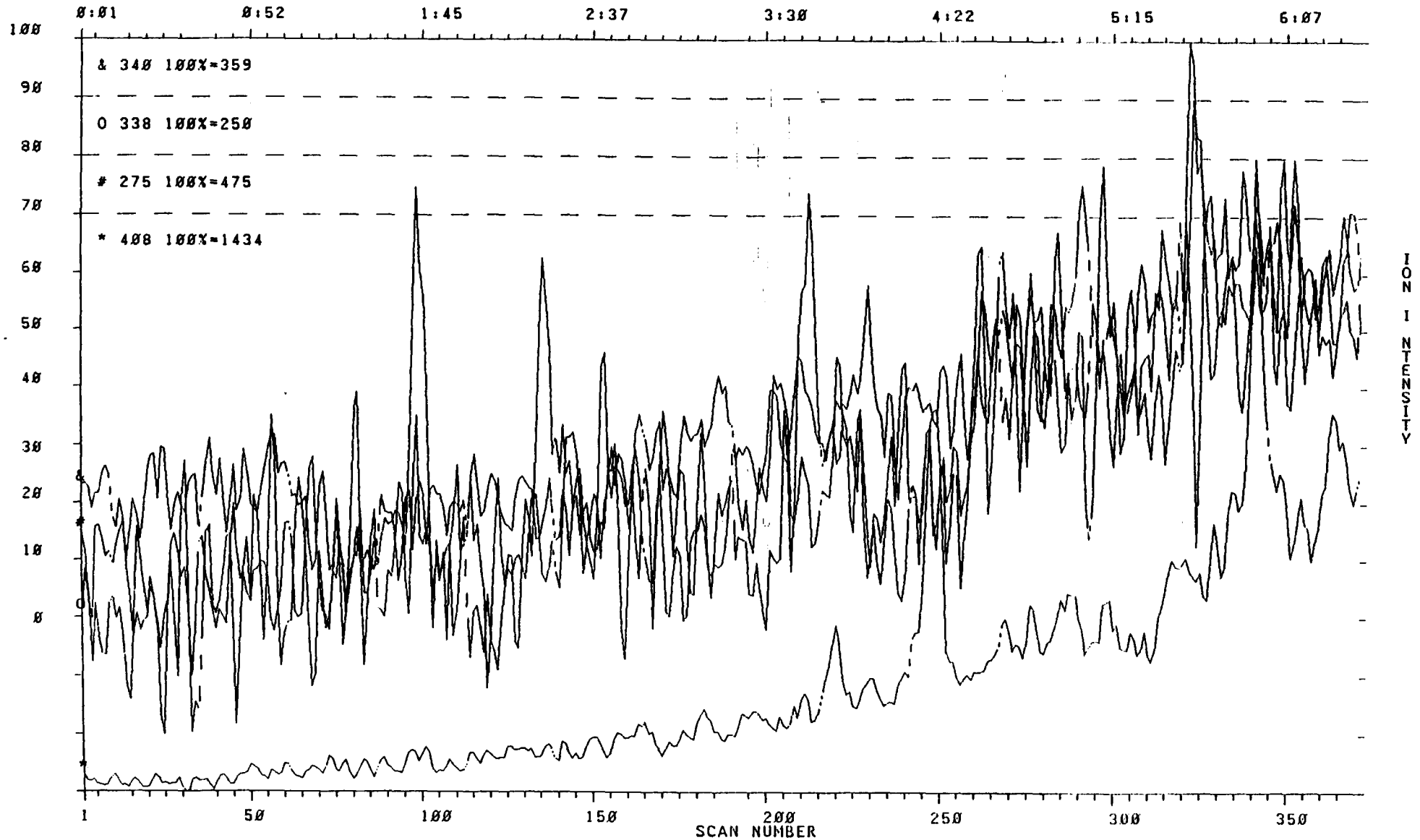


HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

FIGURE:29

KRATOS MS25, DS55 SOFTWARE, RUN: C105ØØ82, WSU NAME: USC6-ØB

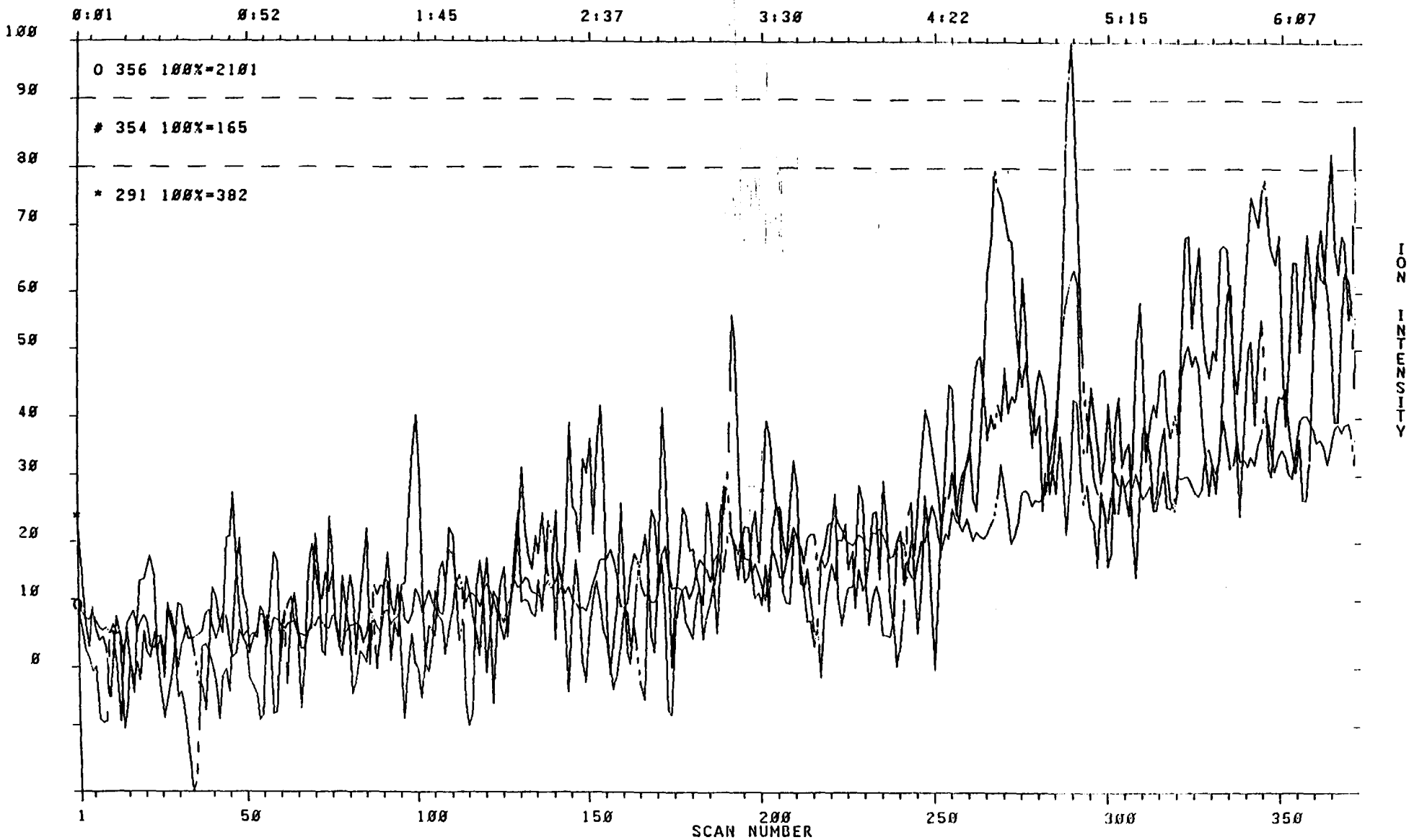
SELECTED-ION MASS CHROMATOGRAMS FOR PENTACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

FIGURE: 30

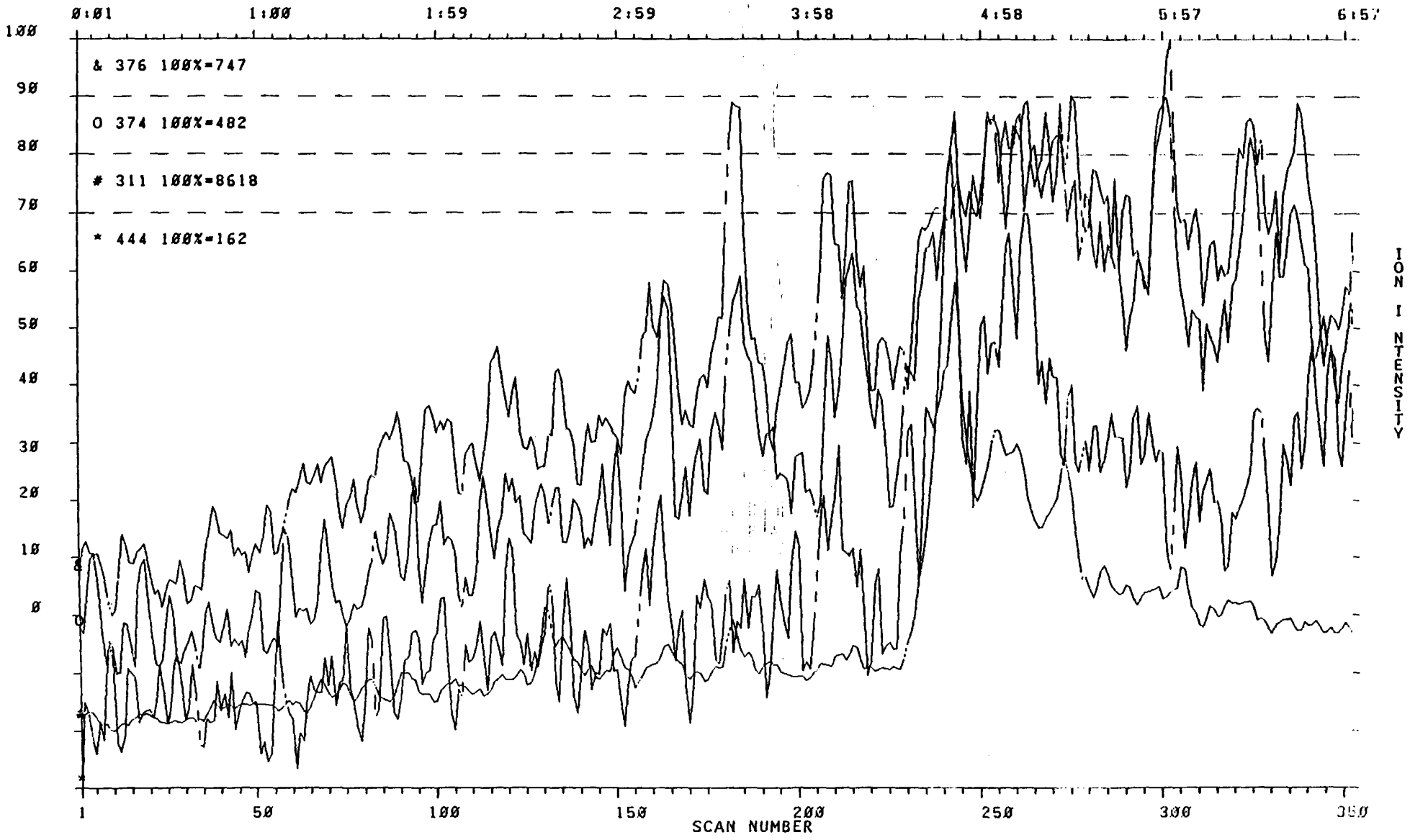
KRATOS MS25, DS55 SOFTWARE, RUN: CTO5ØØ82, WSU NAME: USC6-ØB
SELECTED-ION MASS CHROMATOGRAMS FOR PENTACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

FIGURE: 31

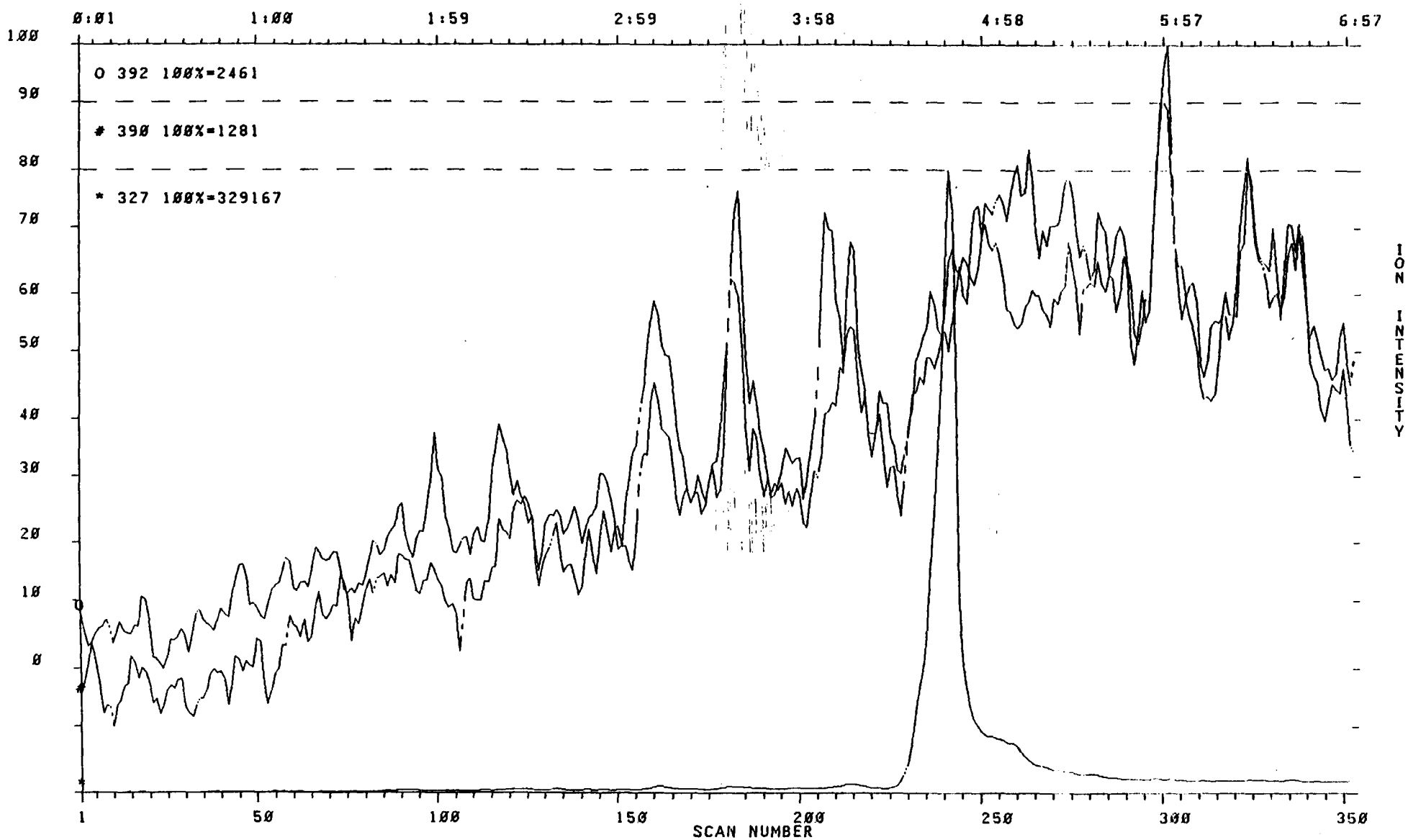
KRATOS MS25, DS55 SOFTWARE, RUN: CTO60082, WSU NAME: USC6-0C
SELECTED-ION MASS CHROMATOGRAMS FOR HEXACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

FIGURE: 32

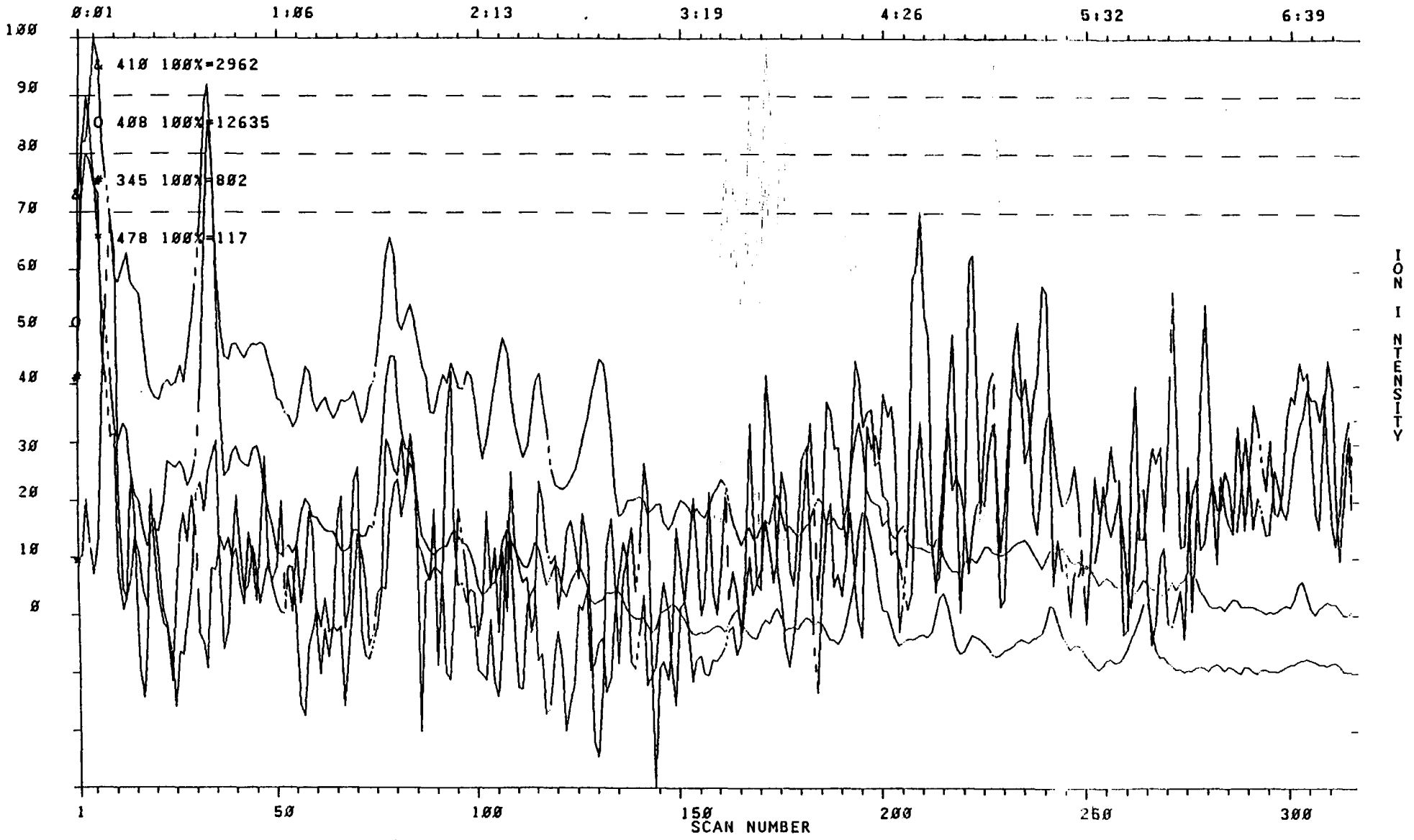
KRATOS MS25, DS55 SOFTWARE, RUN: CT06ØØ82, WSU NAME: USC6-ØB
SELECTED-ION MASS CHROMATOGRAMS FOR HEXACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

FIGURE: 33

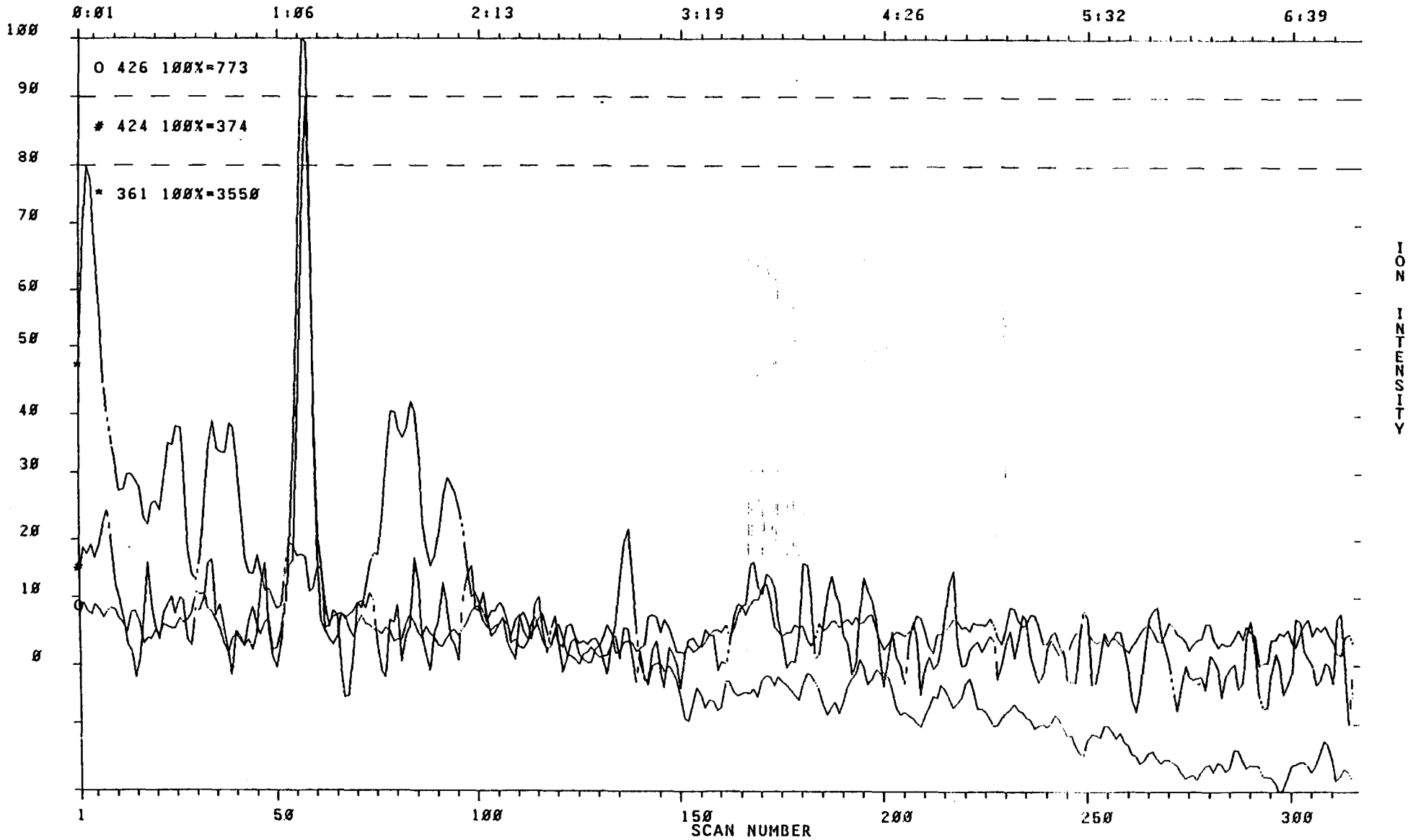
KRATOS MS25, DS55 SOFTWARE, RUN: CTO7ØØØ2, WSU NAME: USC6-ØB
SELECTED-ION MASS CHROMATOGRAMS FOR HEPTACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

FIGURE: 34

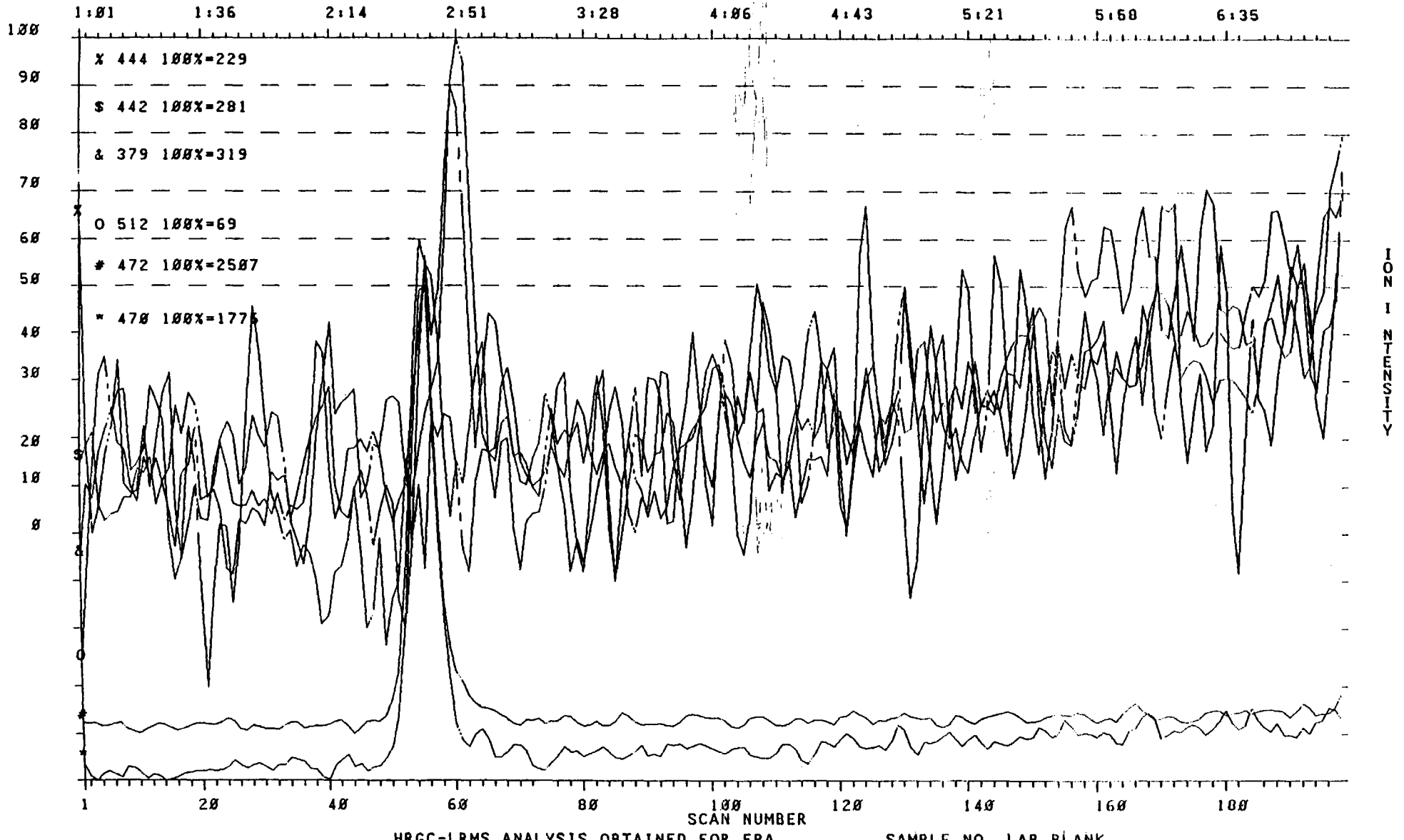
KRATOS MS25, DS55 SOFTWARE, RUN: CTO7ØØ82, WSU NAME: USC6-ØB
SELECTED-ION MASS CHROMATOGRAMS FOR HEPTACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

FIGURE: 35

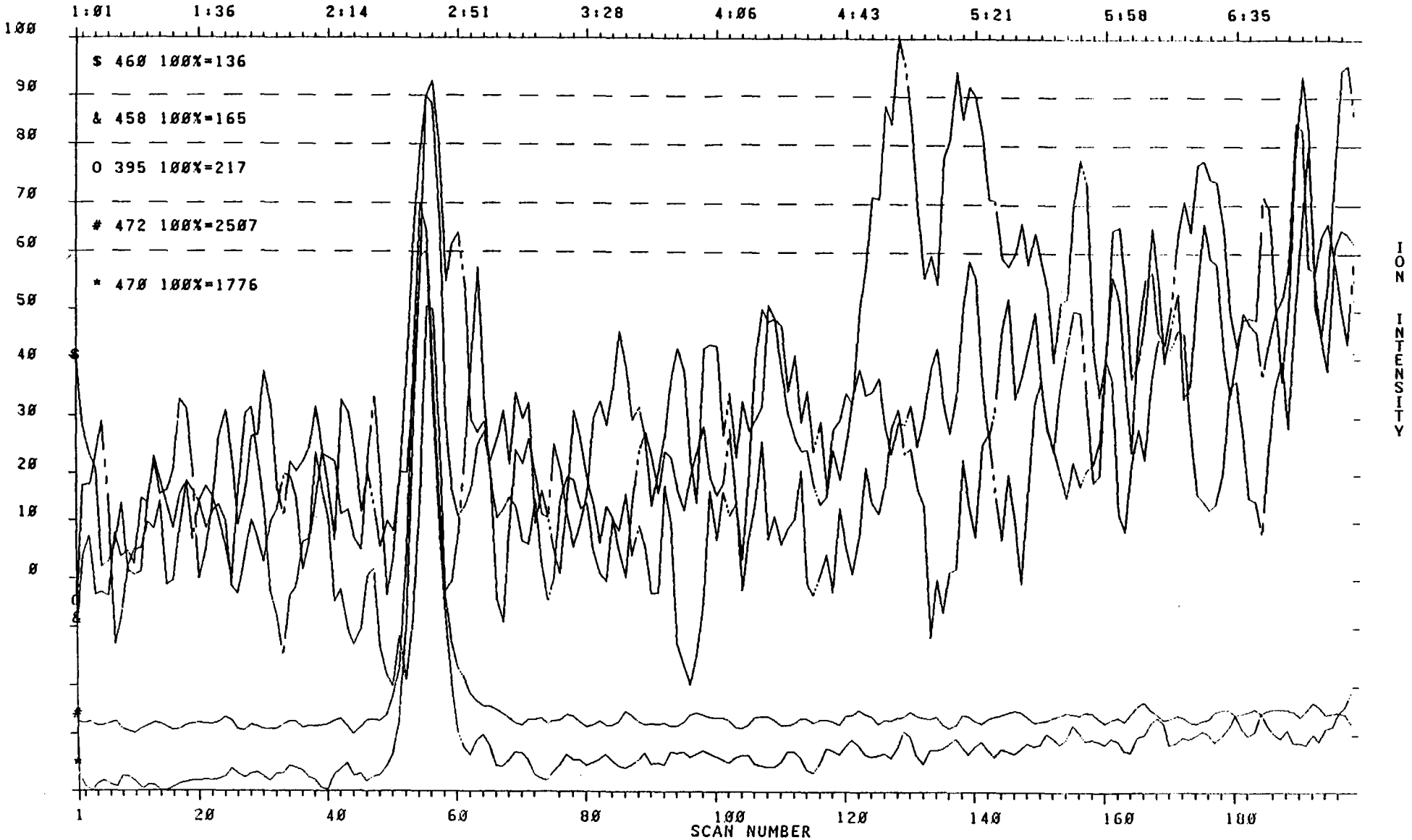
KRATOS MS25, DS55 SOFTWARE, RUN: CTO8ØØB2, WSU NAME: USC6-ØB
SELECTED-ION MASS CHROMATOGRAMS FOR OCTACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

FIGURE: 36

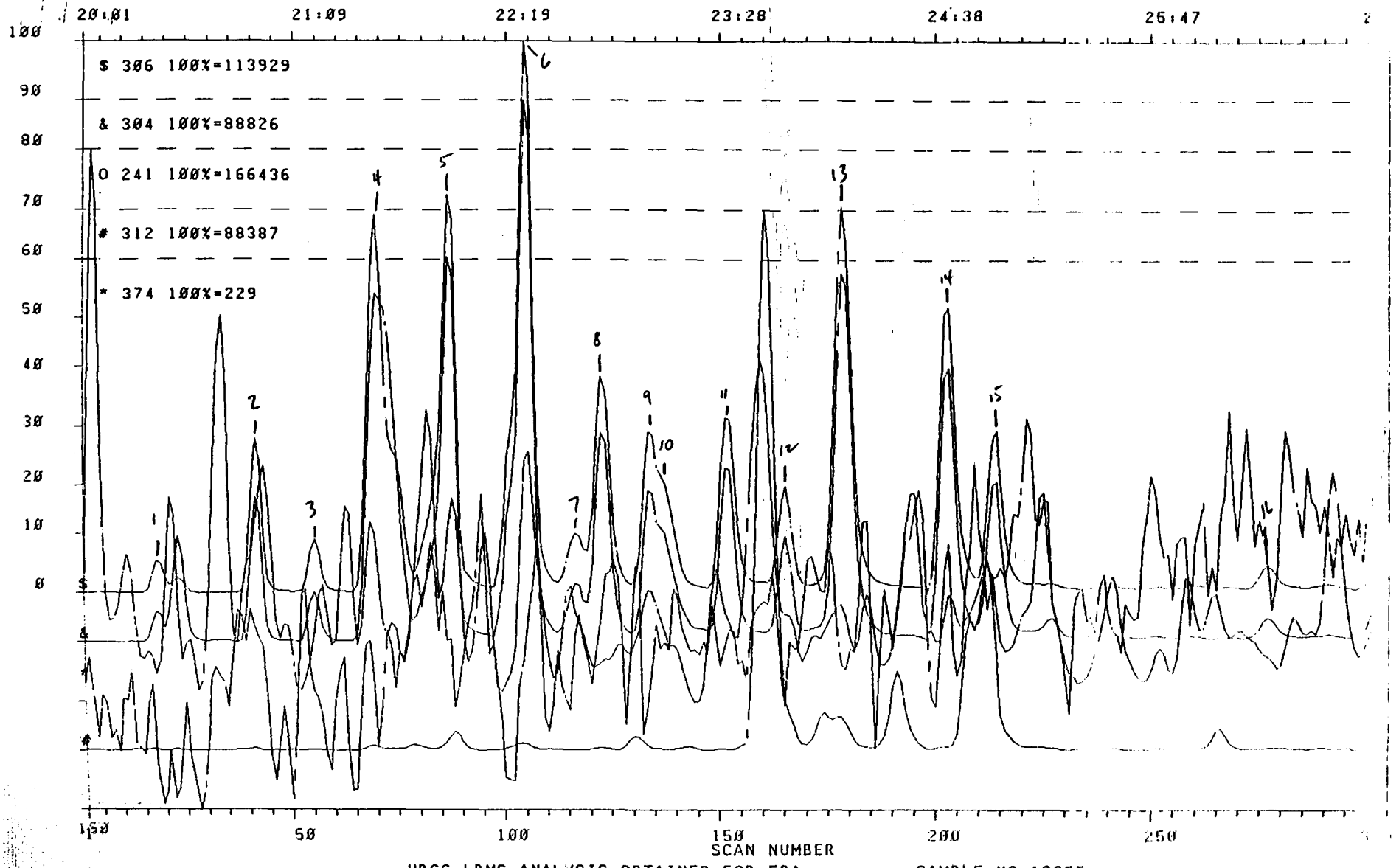
KRATOS MS25, DS55 SOFTWARE, RUN: CT080082, WSU NAME: USC6-0B
SELECTED-ION MASS CHROMATOGRAMS FOR OCTACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

FIGURE: 37

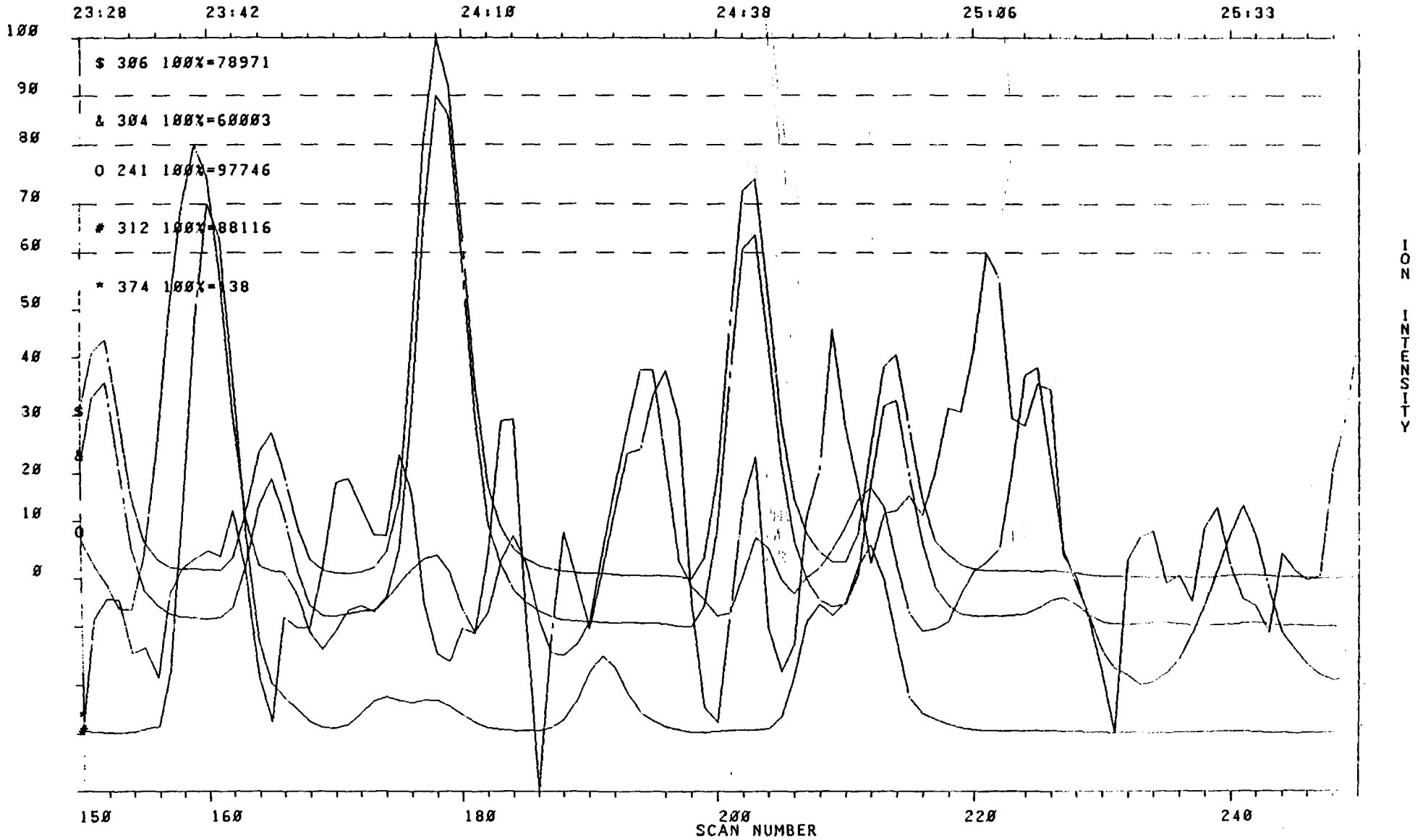
KRATOS MS25, DS55 SOFTWARE, RUN: CTO40097, WSU NAME: NBC5-3
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.13855

FIGURE: 38

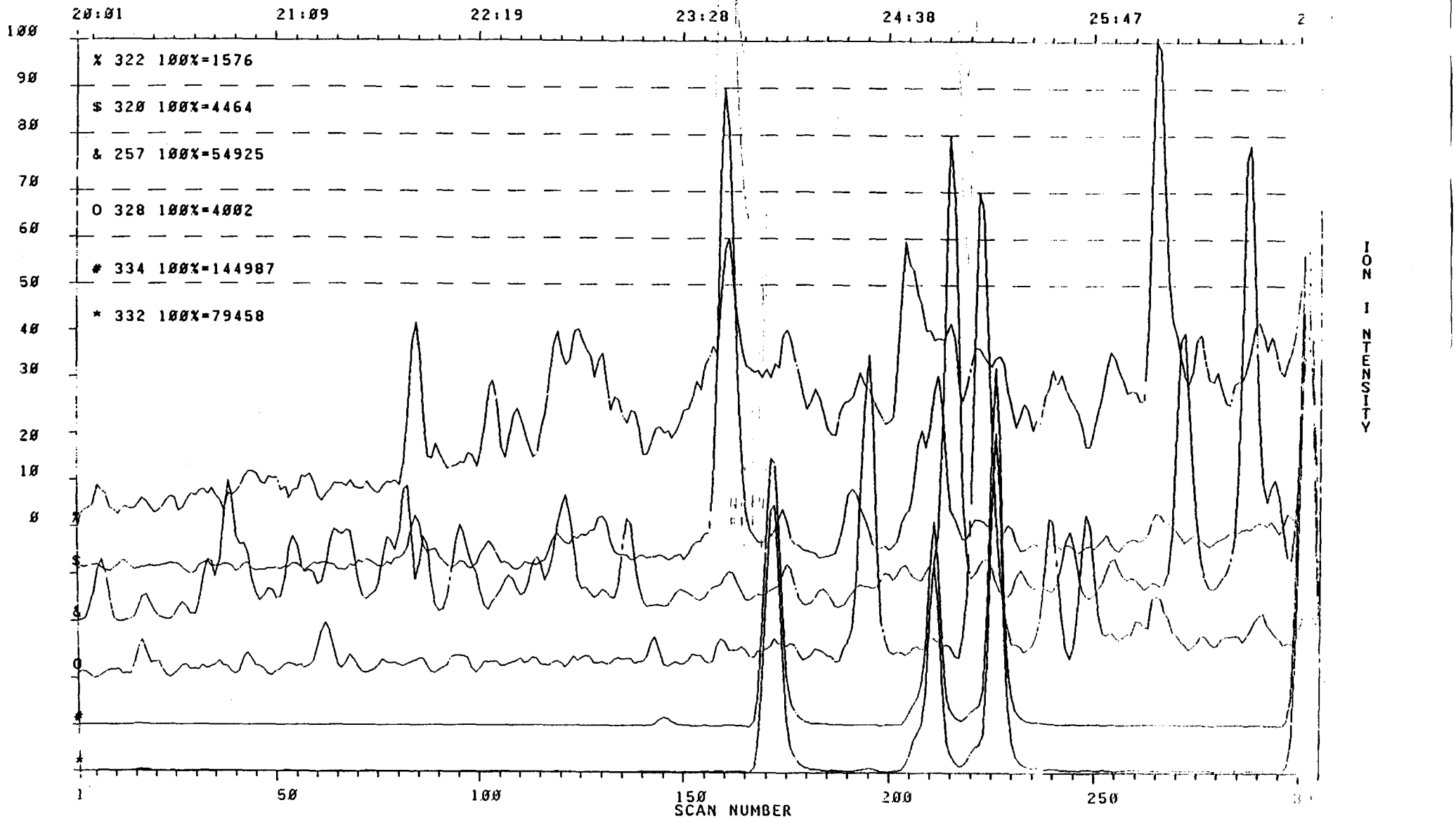
KRATOS MS25, DS55 SOFTWARE, RUN: CTO40097, WSU NAME: NBC5-3
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZOFURANS



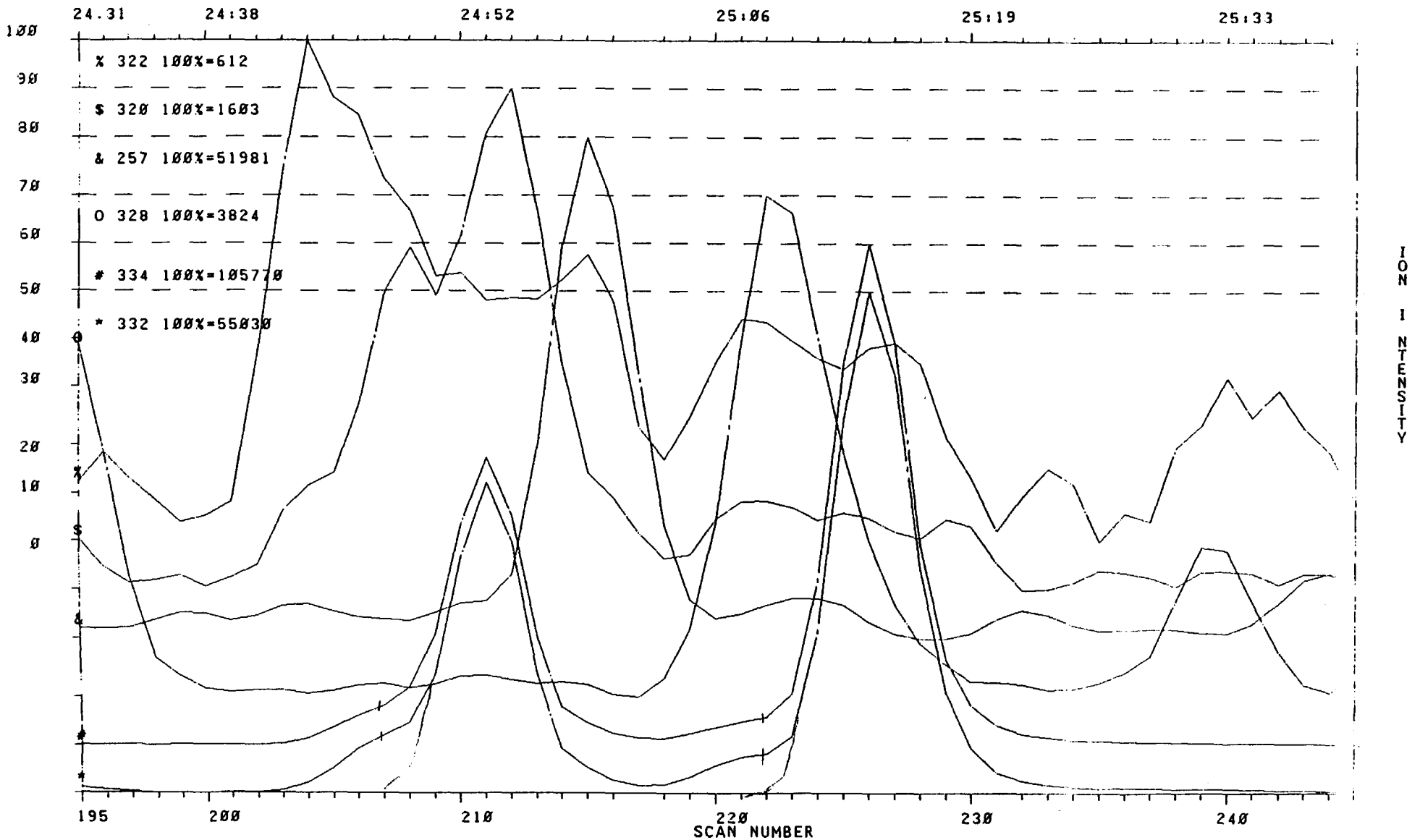
HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.13855

FIGURE: 39

KRATOS MS25, DS55 SOFTWARE, RUN: CTO40097, WSU NAME: NBC5-3
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZO-P-DIOXINS



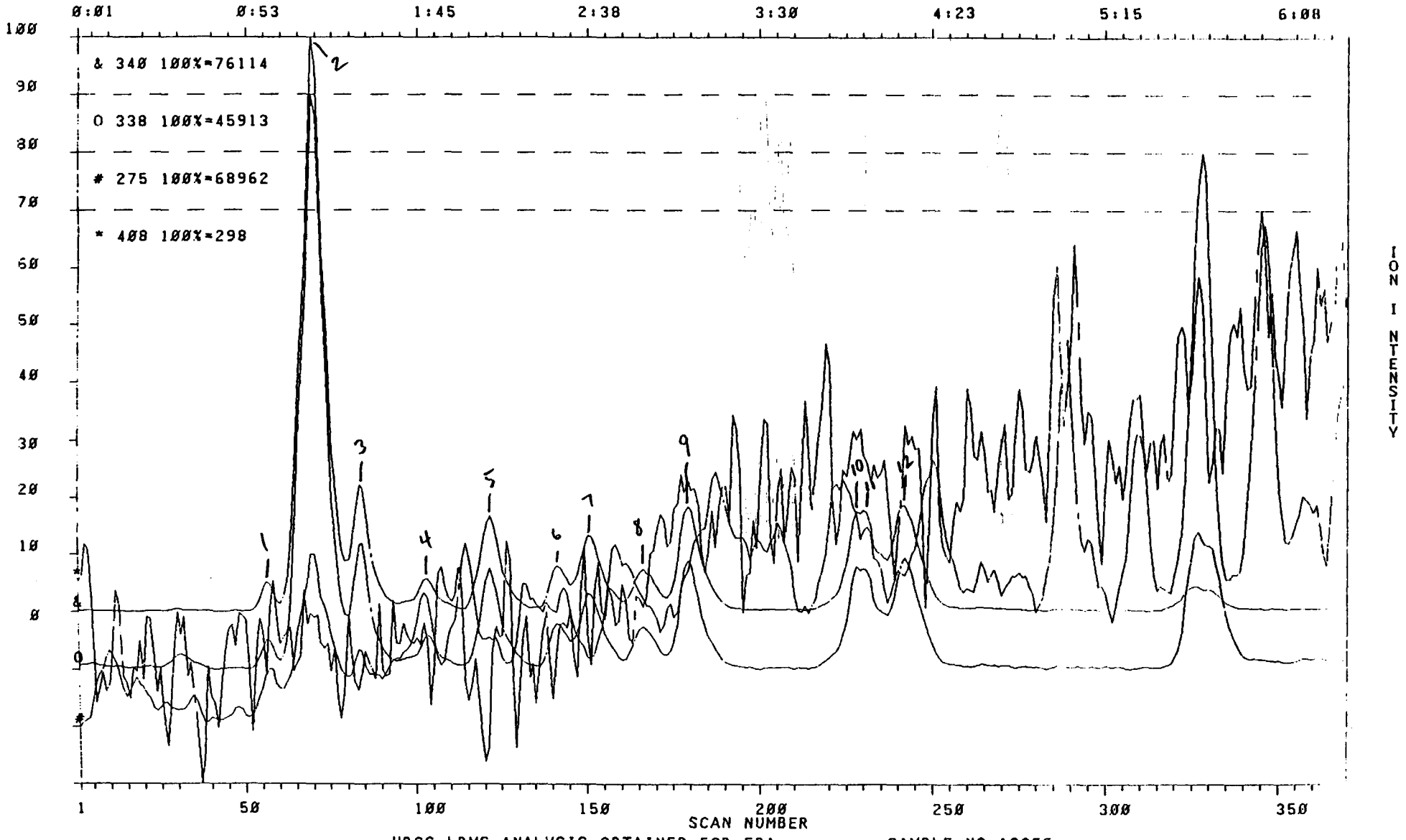
HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.13855
FIGURE: 40



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.13855

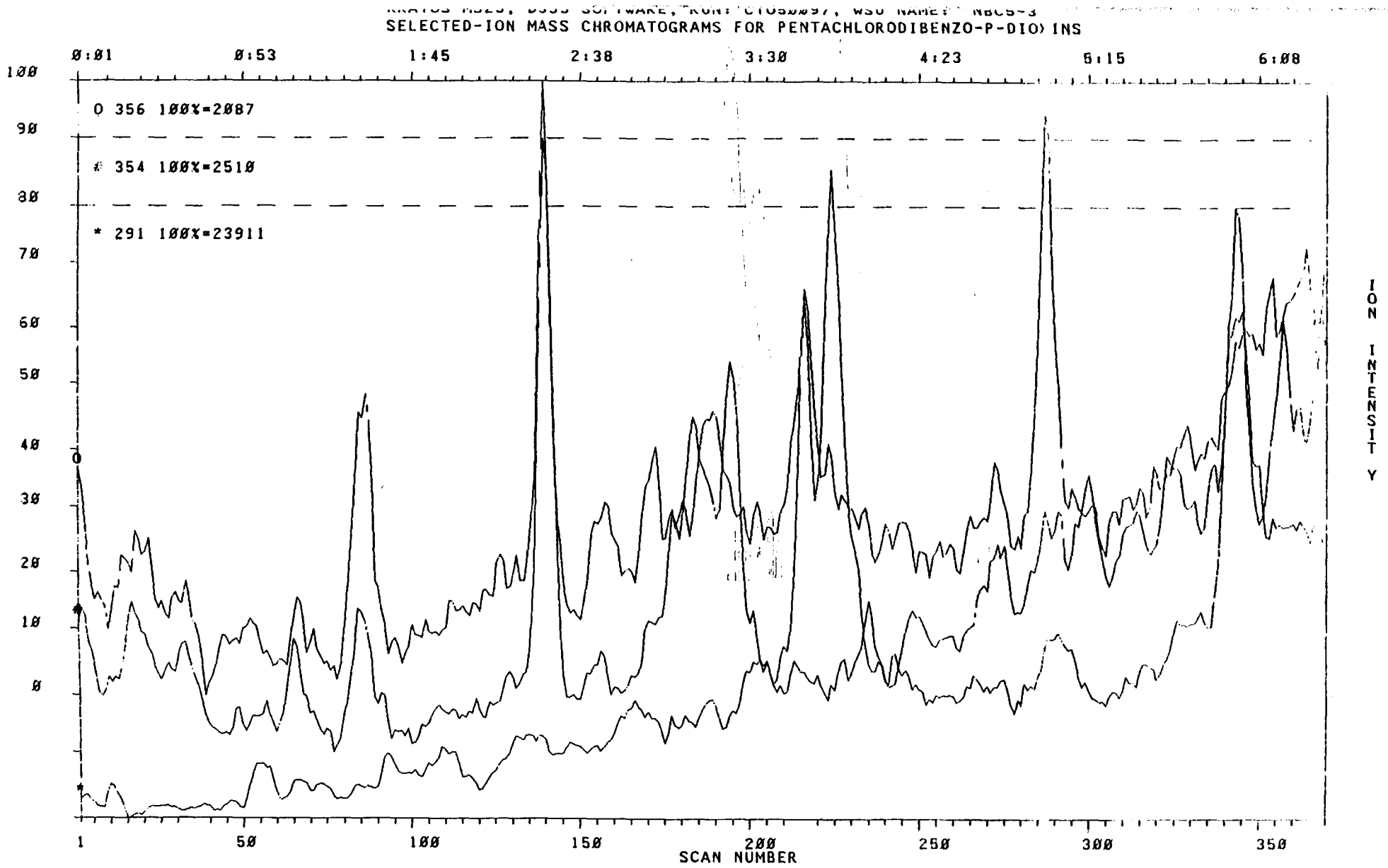
FIGURE: 41

KRATOS MS25, DS55 SOFTWARE, RUN: CTO50097, WSU NAME: NBC5-3
SELECTED-ION MASS CHROMATOGRAMS FOR PENTACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.13855

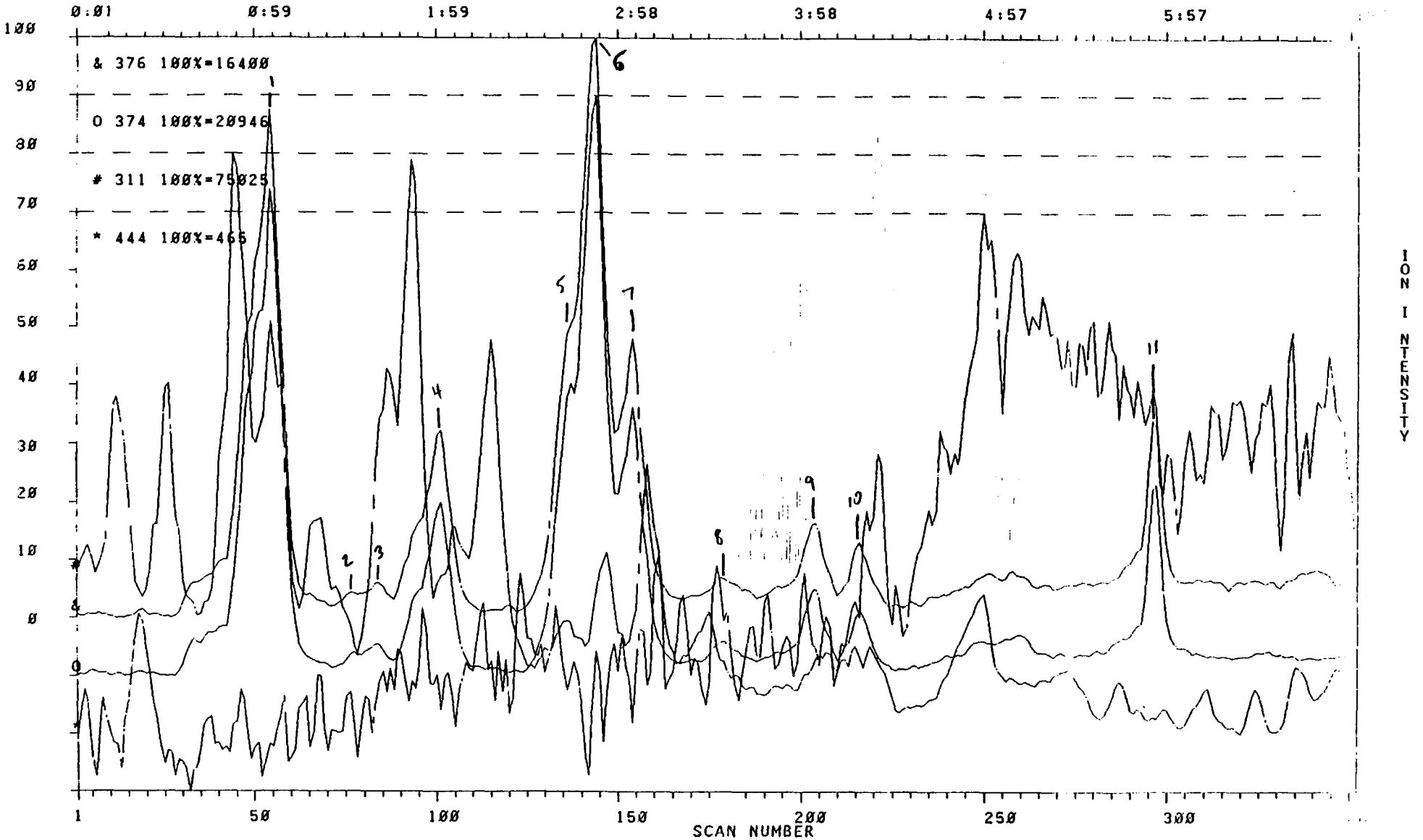
FIGURE: 42



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.13855

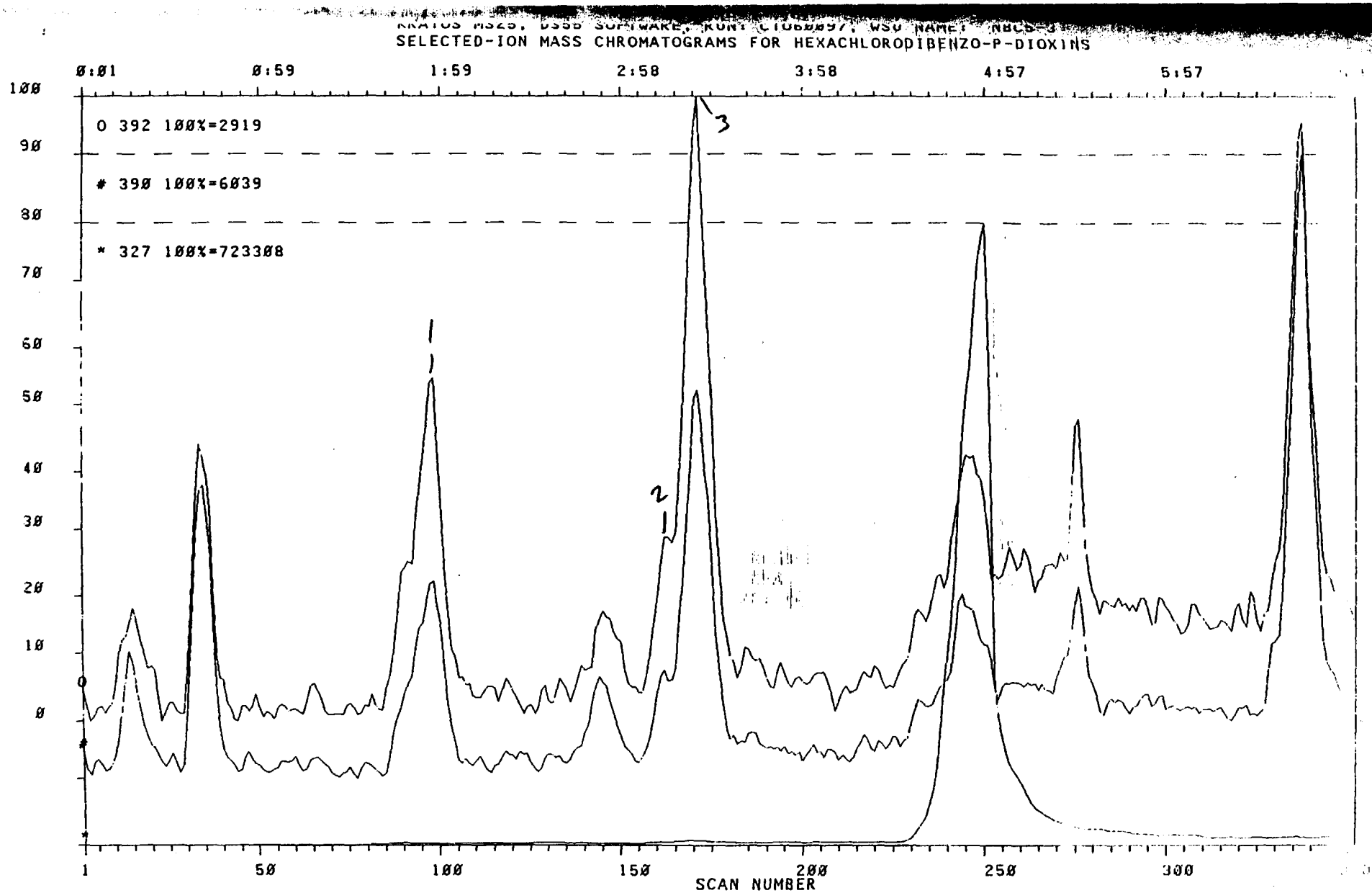
FIGURE: 43

KRATOS MS25, DS55 SOFTWARE, RUN: CTO60097, WSU NAME: NBC5-3
SELECTED-ION MASS CHROMATOGRAMS FOR HEXACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.13855

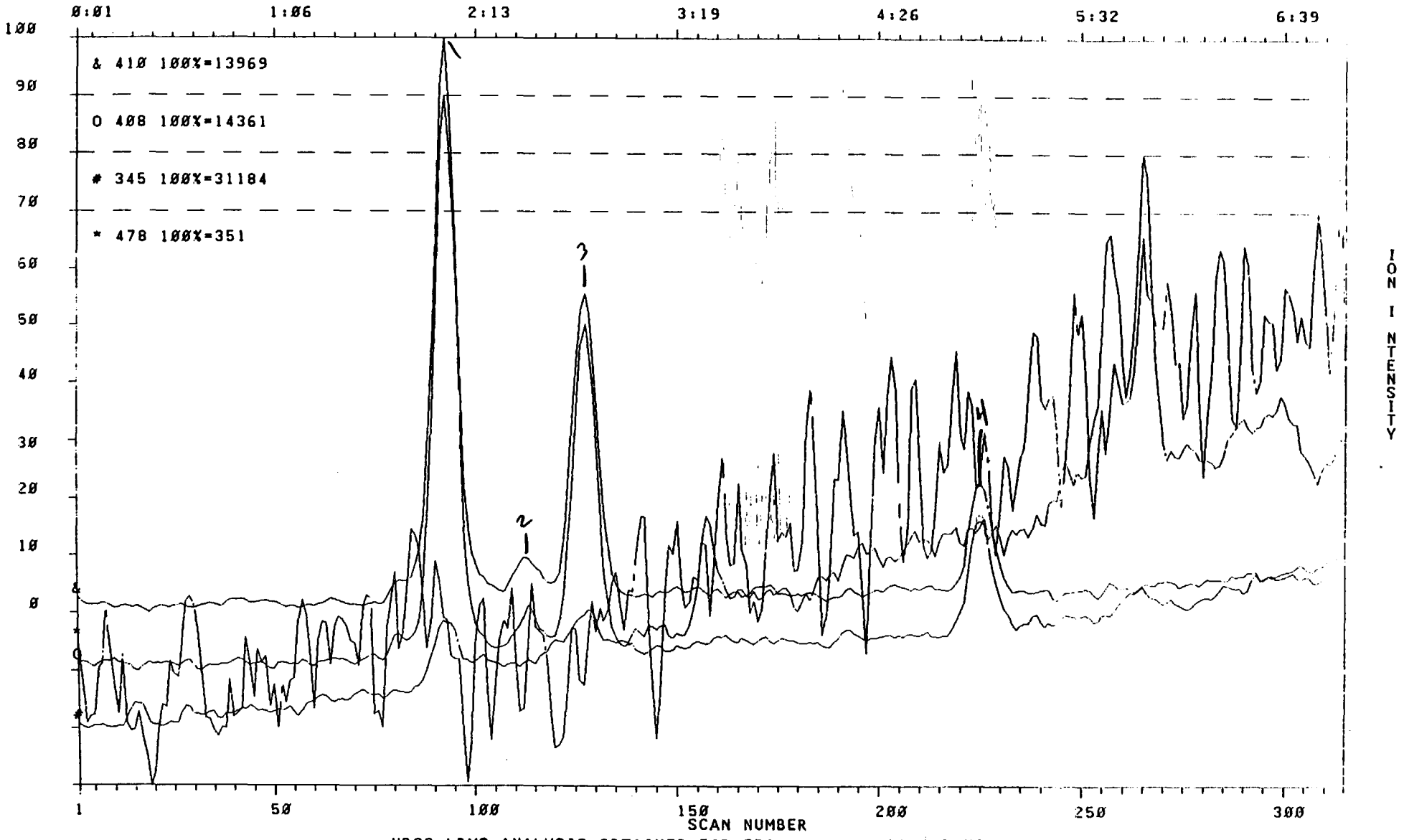
FIGURE: 44



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.13855

FIGURE: 45

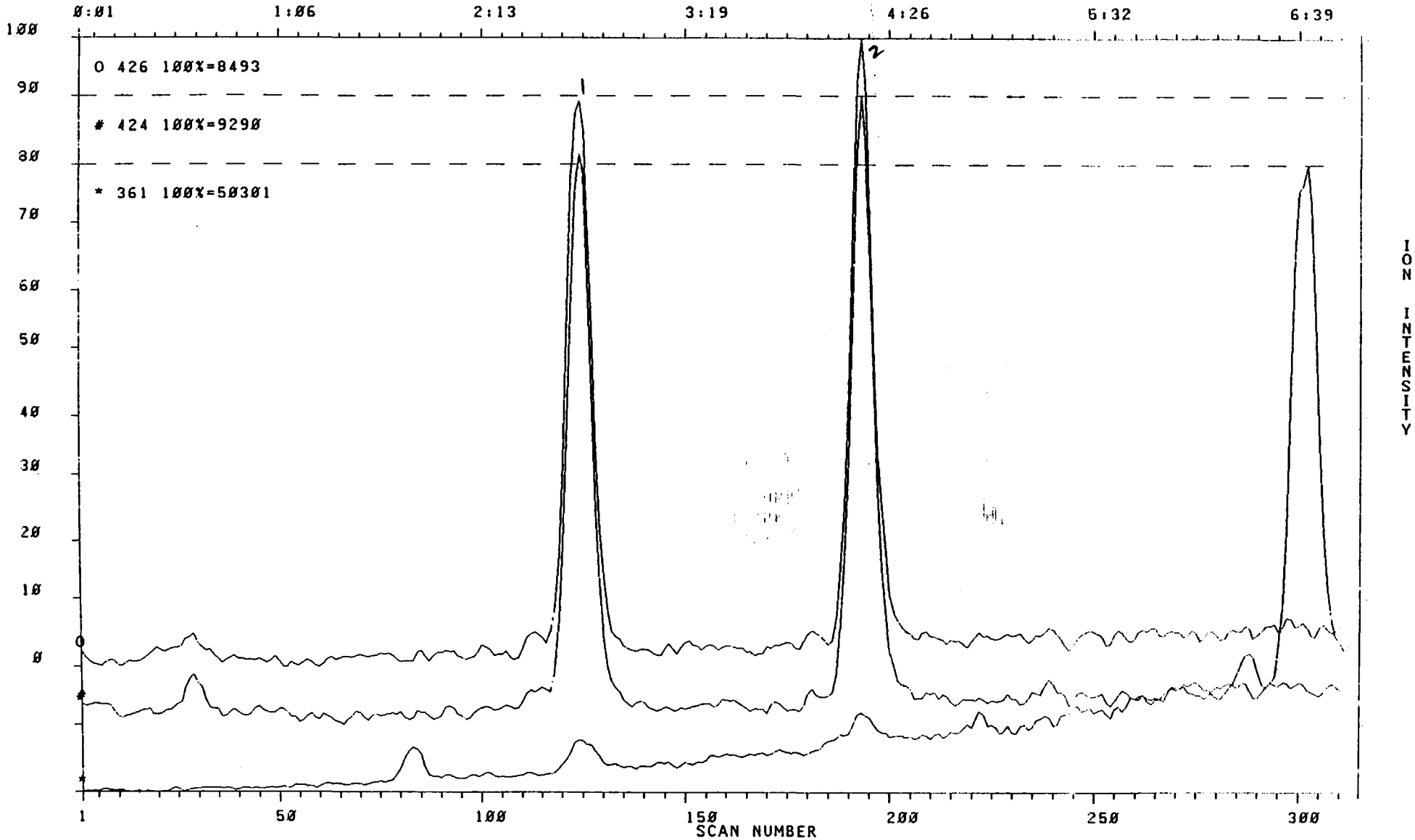
KRATOS MS25, DS55 SOFTWARE, RUN: CTO70097, WSU NAME: NBC5-3
SELECTED-ION MASS CHROMATOGRAMS FOR HEPTACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.13855

FIGURE: 46

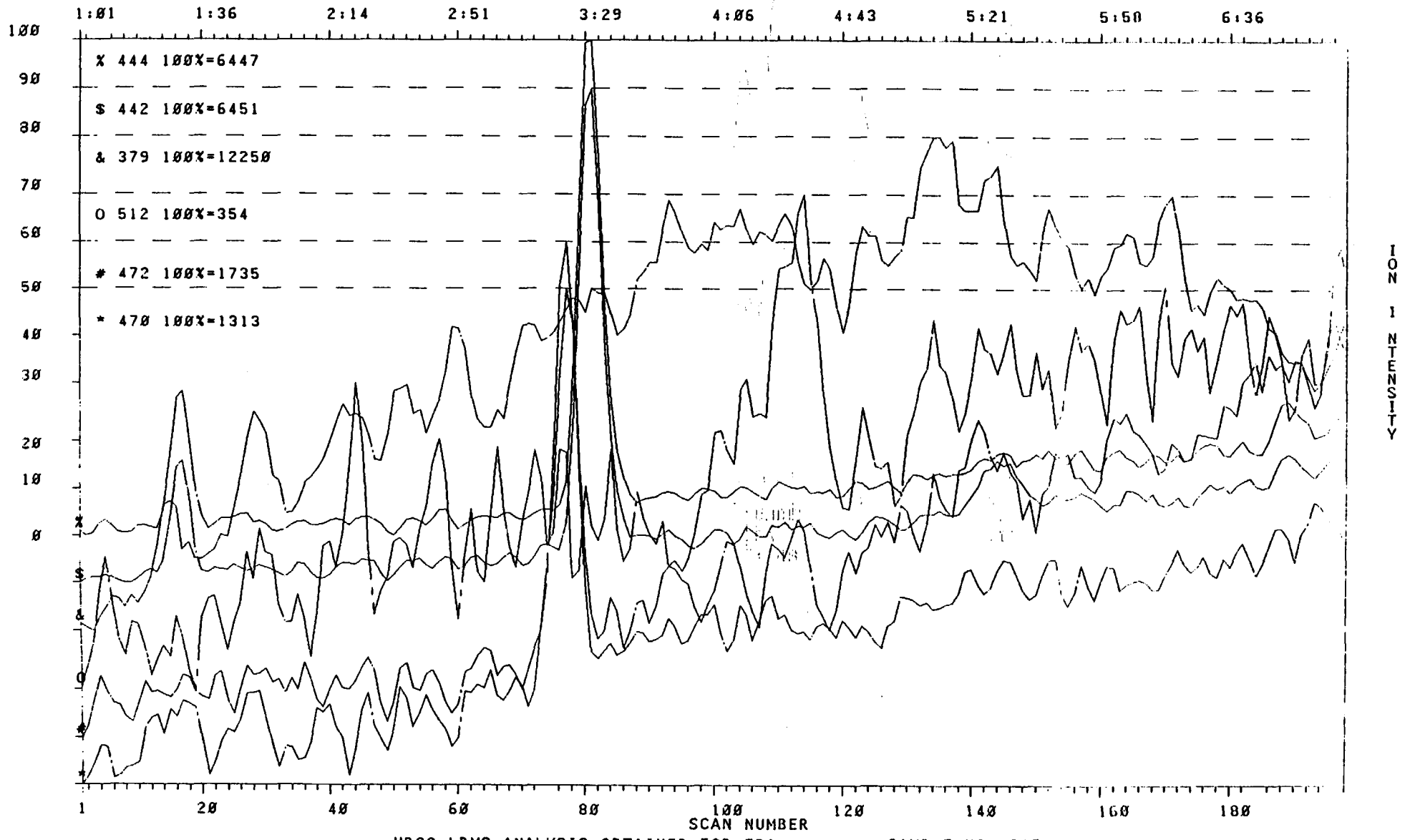
KRATOS MS25, DS55 SOFTWARE, RUN: CTO70097, WSU NAME: NBC5-3
SELECTED-ION MASS CHROMATOGRAMS FOR HEPTACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.13855

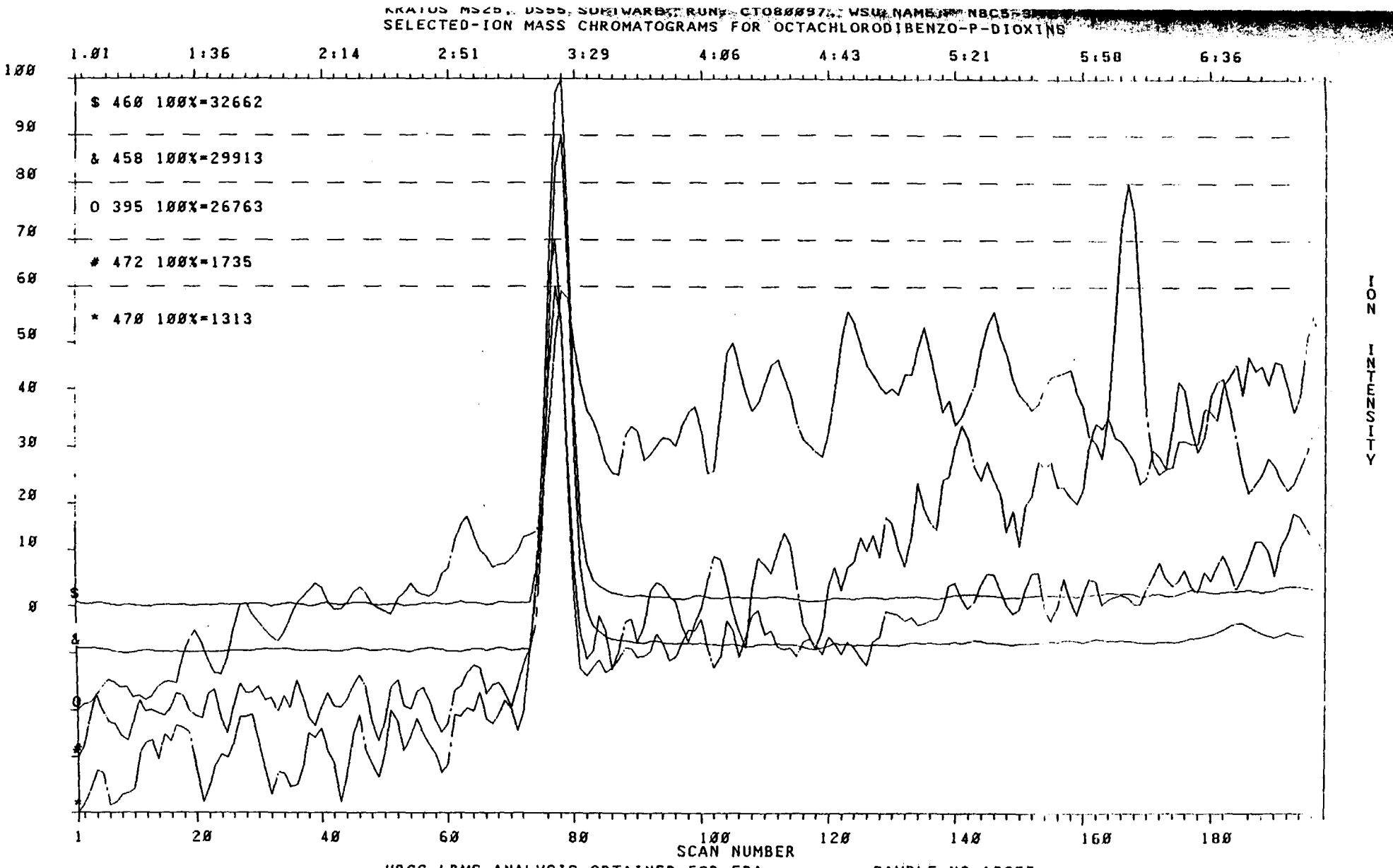
FIGURE: 47

KRATOS MS25, DS55 SOFTWARE, RUN: CTO80097, WSU NAME: NBC5-3
SELECTED-ION MASS CHROMATOGRAMS FOR OCTACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.13855

FIGURE: 48

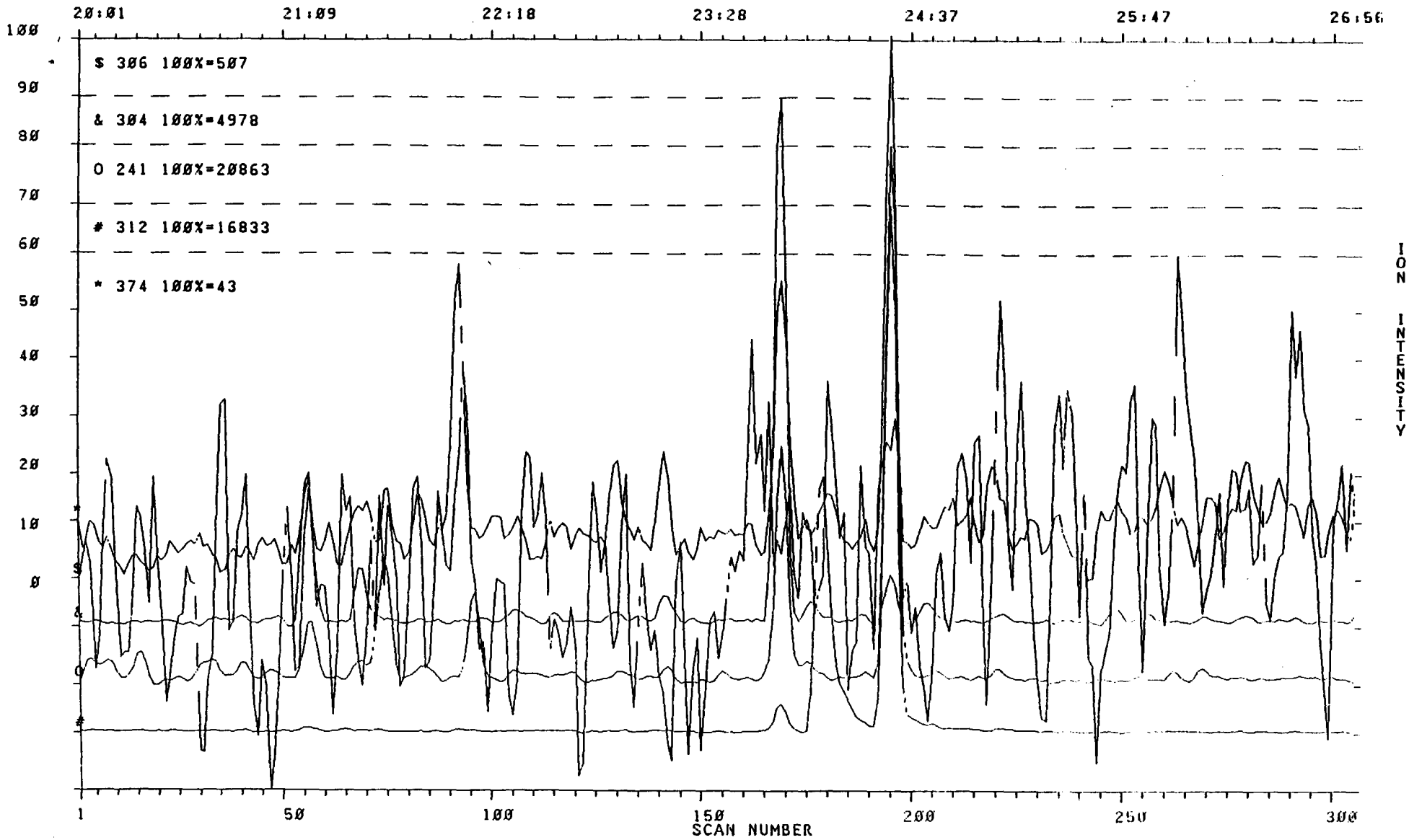


HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.13855

FIGURE: 49

KRATOS MS25, DS55 SOFTWARE, RUN: CTO40083, WSU NAME: NBC6-0-B

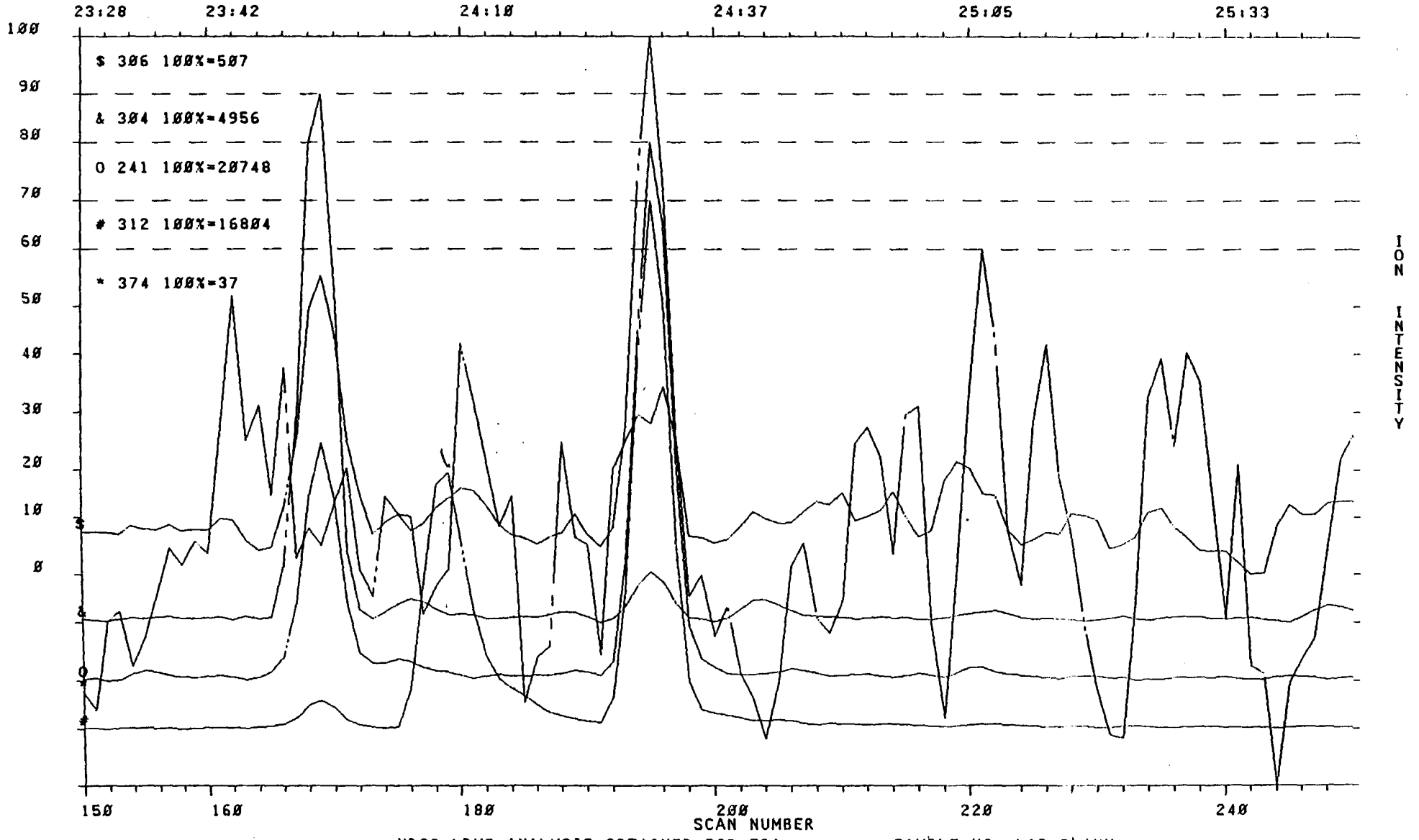
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

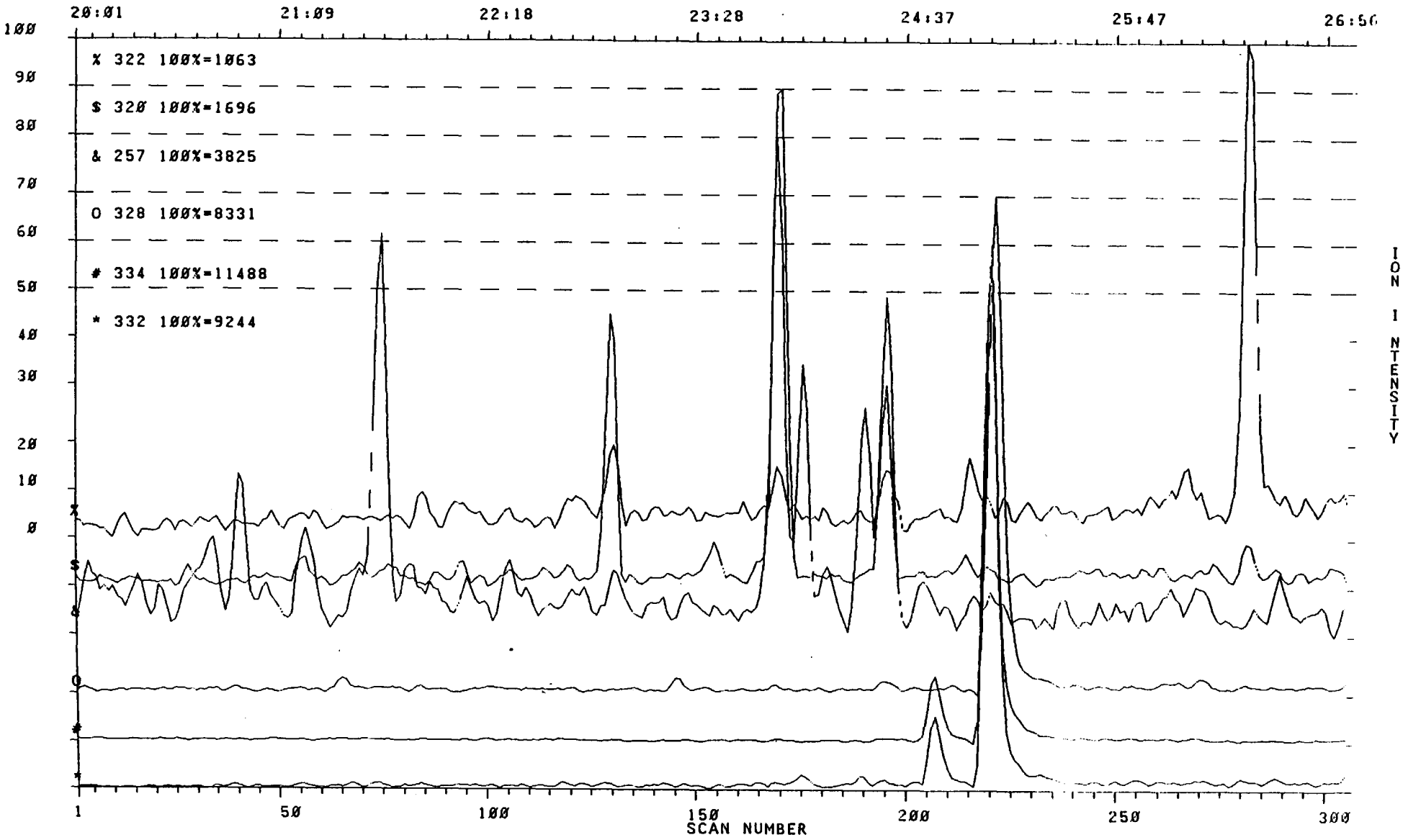
FIGURE: 50

DATE: 04/29/86 TIME: 16:51
KRATOS MS25, DS55 SOFTWARE, RUN: CTO40083, WSU NAME: NBC5-0-8
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK
FIGURE: 51

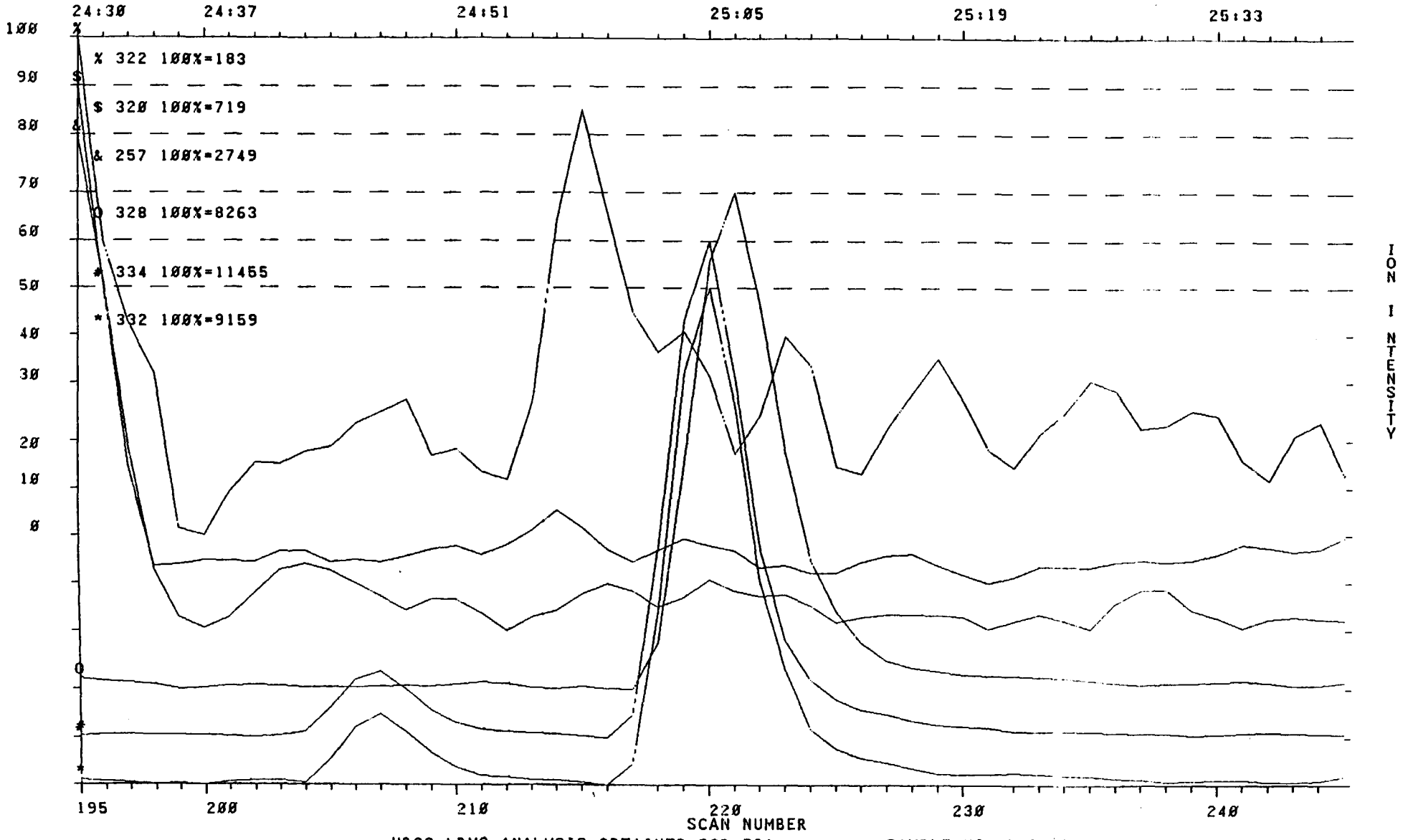
KRATOS MS25, DS55 SOFTWARE, RUN: CTO40083, WSU NAME: NBC5-0-B
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

FIGURE: 52

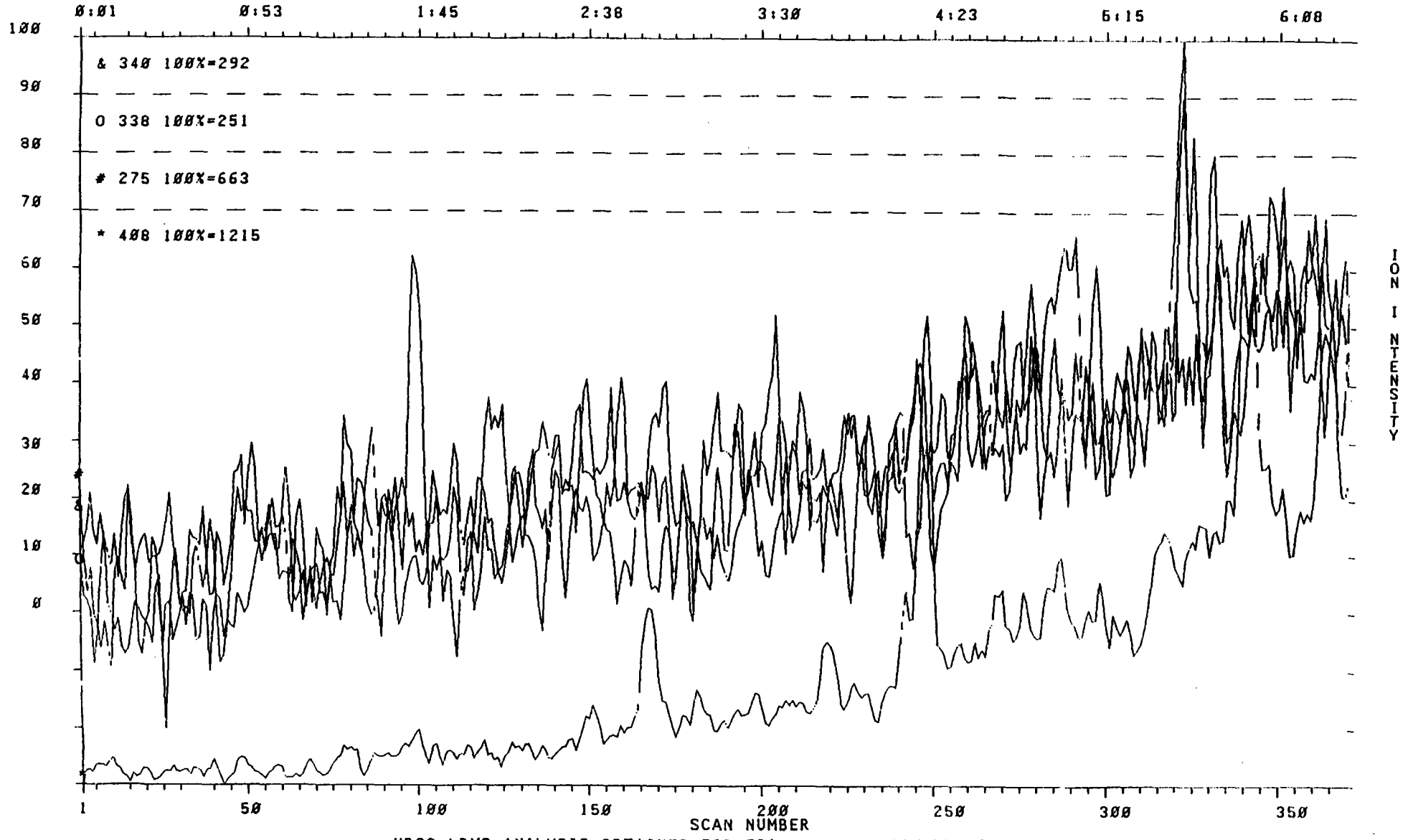
DATE: 04/29/86 TIME: 16:51
KRATOS MS25, DS55 SOFTWARE, RUN: CT04003, WSU NAME: NBC5-0-B
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK
FIGURE:53

KRATOS MS50, DS55 SOFTWARE, RUN: CT05D083, WSO NAME: NBC5-0-B

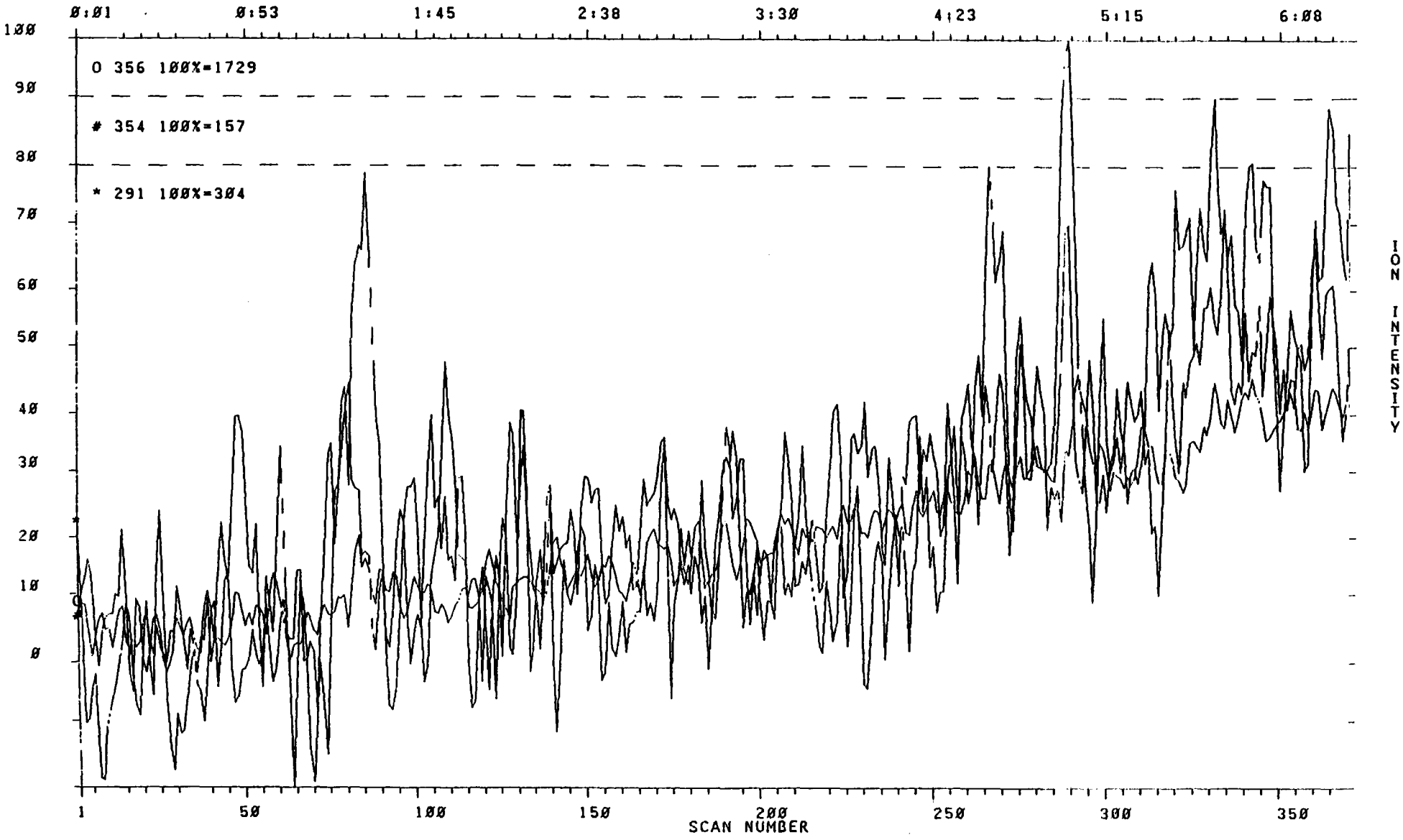
SELECTED-ION MASS CHROMATOGRAMS FOR PENTACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

FIGURE: 54

KRATOS MS25, DS55 SOFTWARE, RUN: CTO5ØØ83, WSU NAME: NBC5-Ø-B
SELECTED-ION MASS CHROMATOGRAMS FOR PENTACHLORODIBENZO-P-DIOXINS

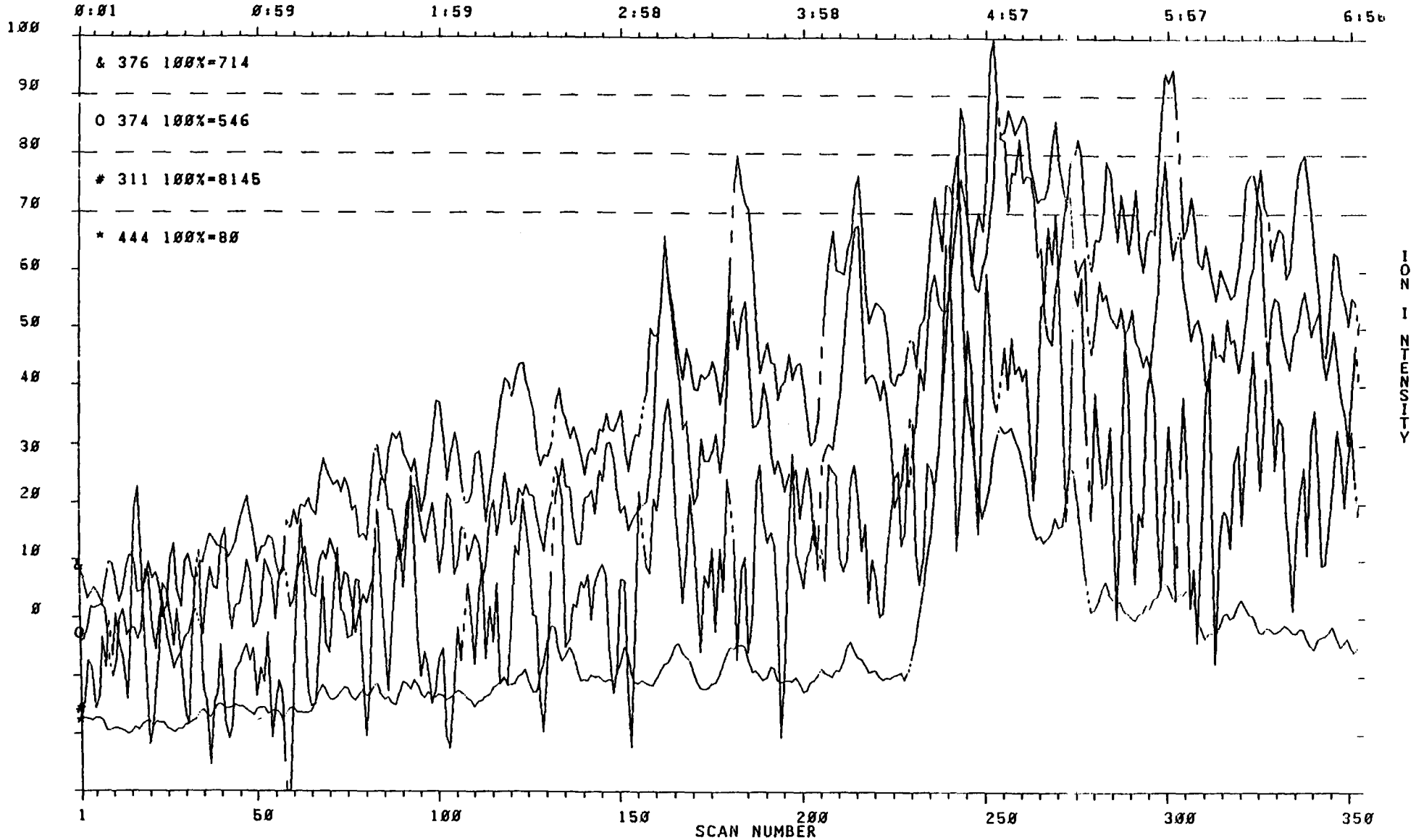


HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

FIGURE: 55

KRATOS MS25, DS55 SOFTWARE, RUN: C106ØØ83, WSU NAME: [illegible]

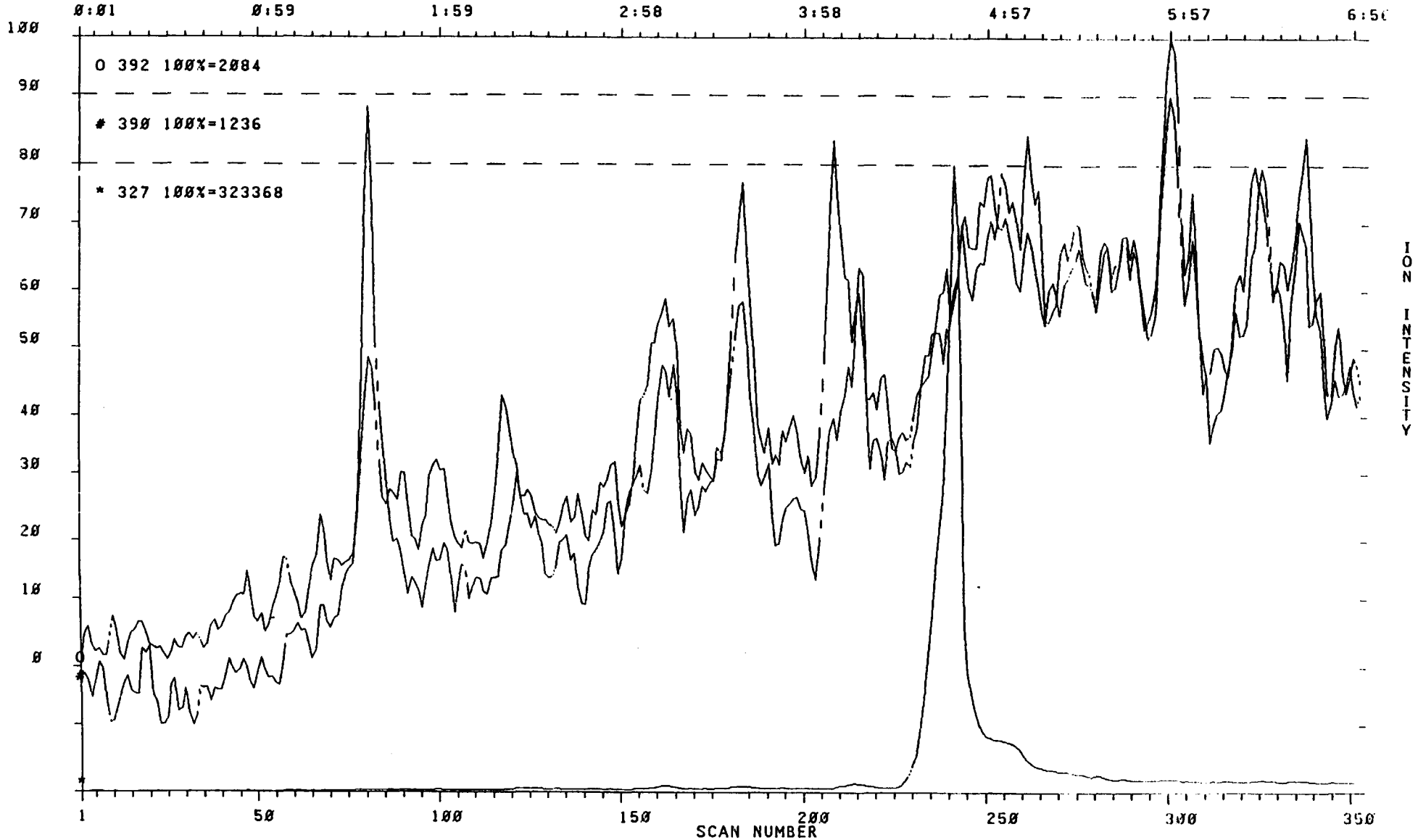
SELECTED-ION MASS CHROMATOGRAMS FOR HEXACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

FIGURE: 56

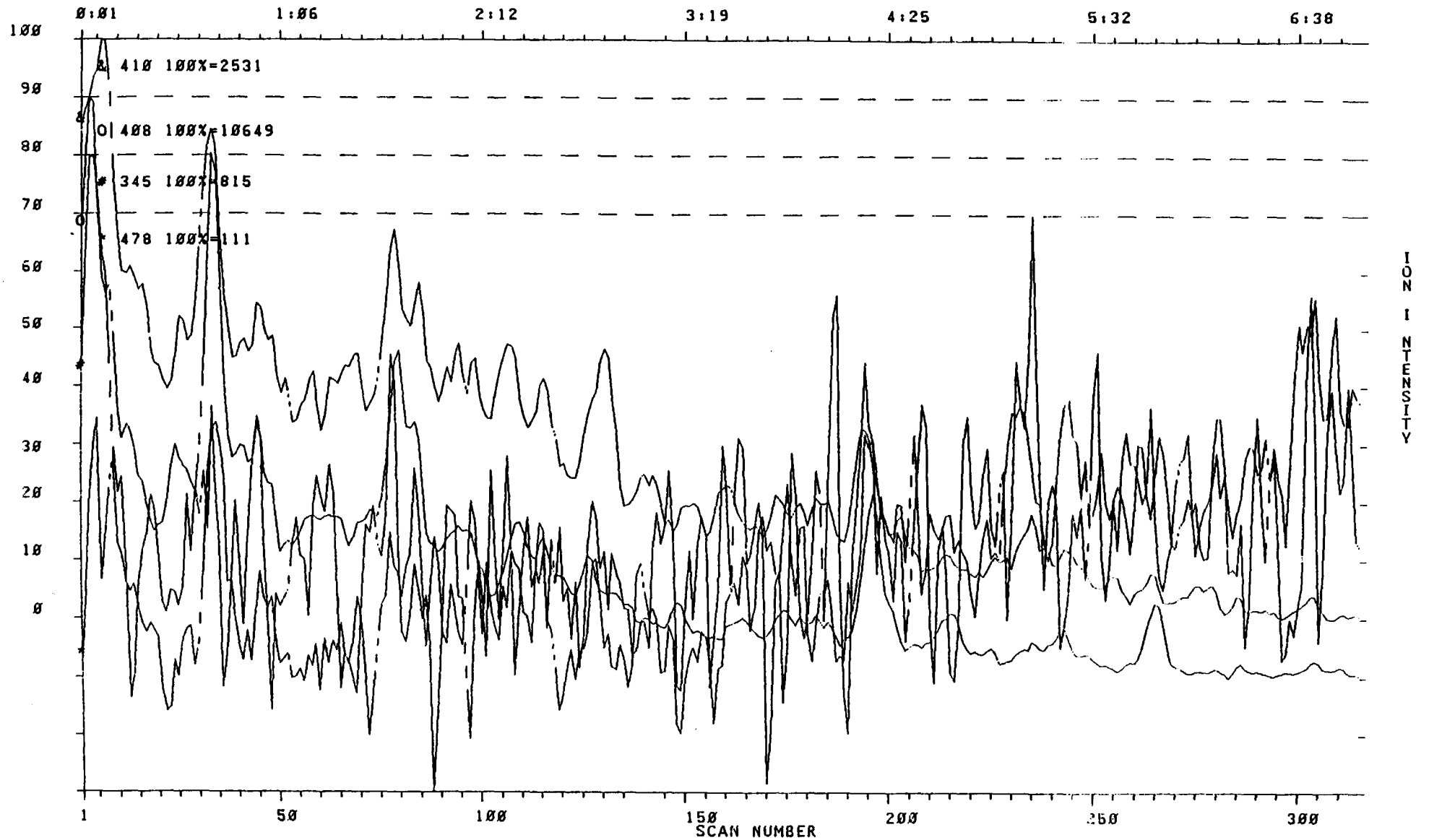
KRATOS MS25, DS55 SOFTWARE, RUN: CTO6ØØB3, WSU NAME: NBC5-Ø-B
SELECTED-ION MASS CHROMATOGRAMS FOR HEXACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

FIGURE: 57

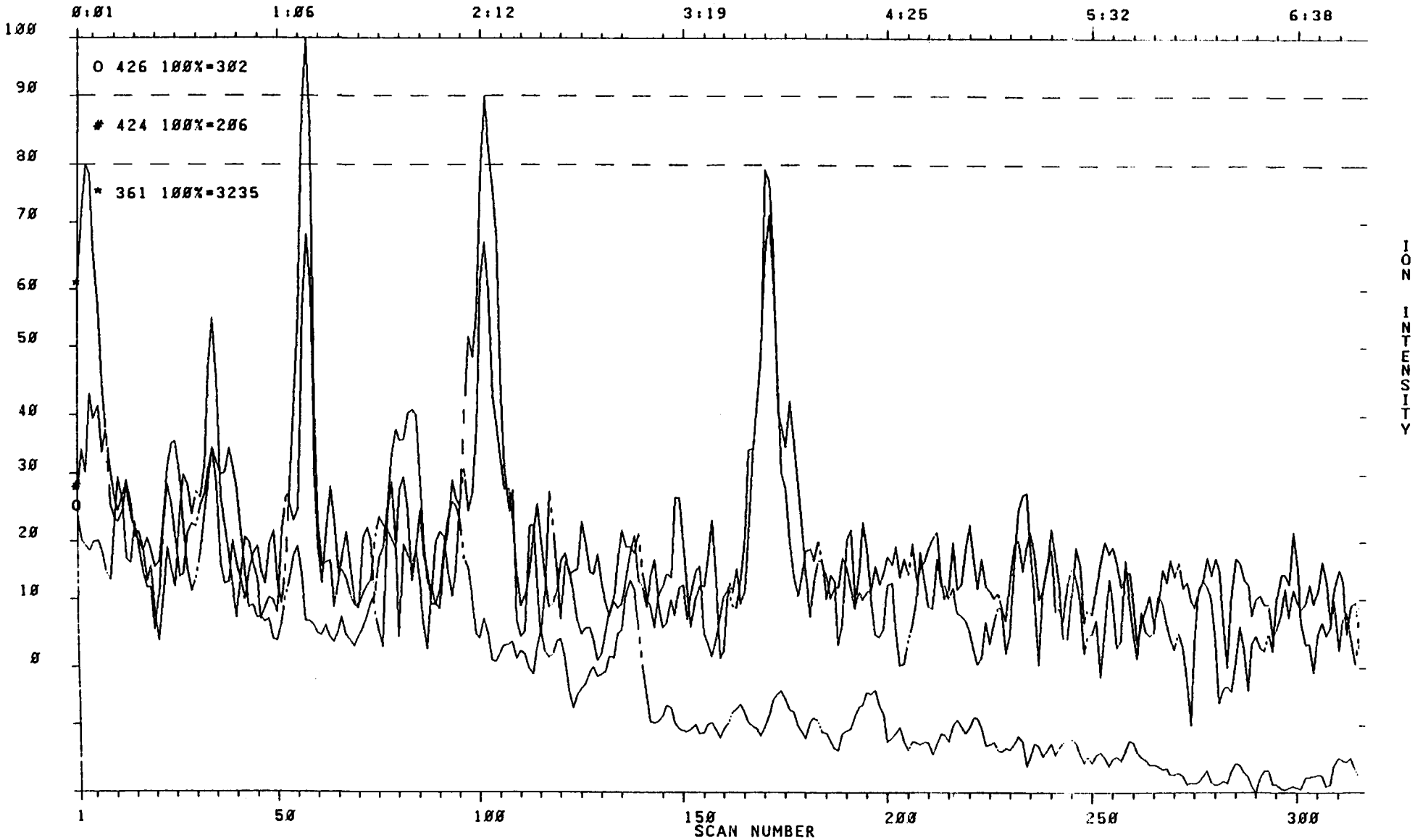
KRATOS MS25, DS55 SOFTWARE, RUN: CTO70083, WSU NAME: NBC5-0-B
SELECTED-ION MASS CHROMATOGRAMS FOR HEPTACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

FIGURE: 58

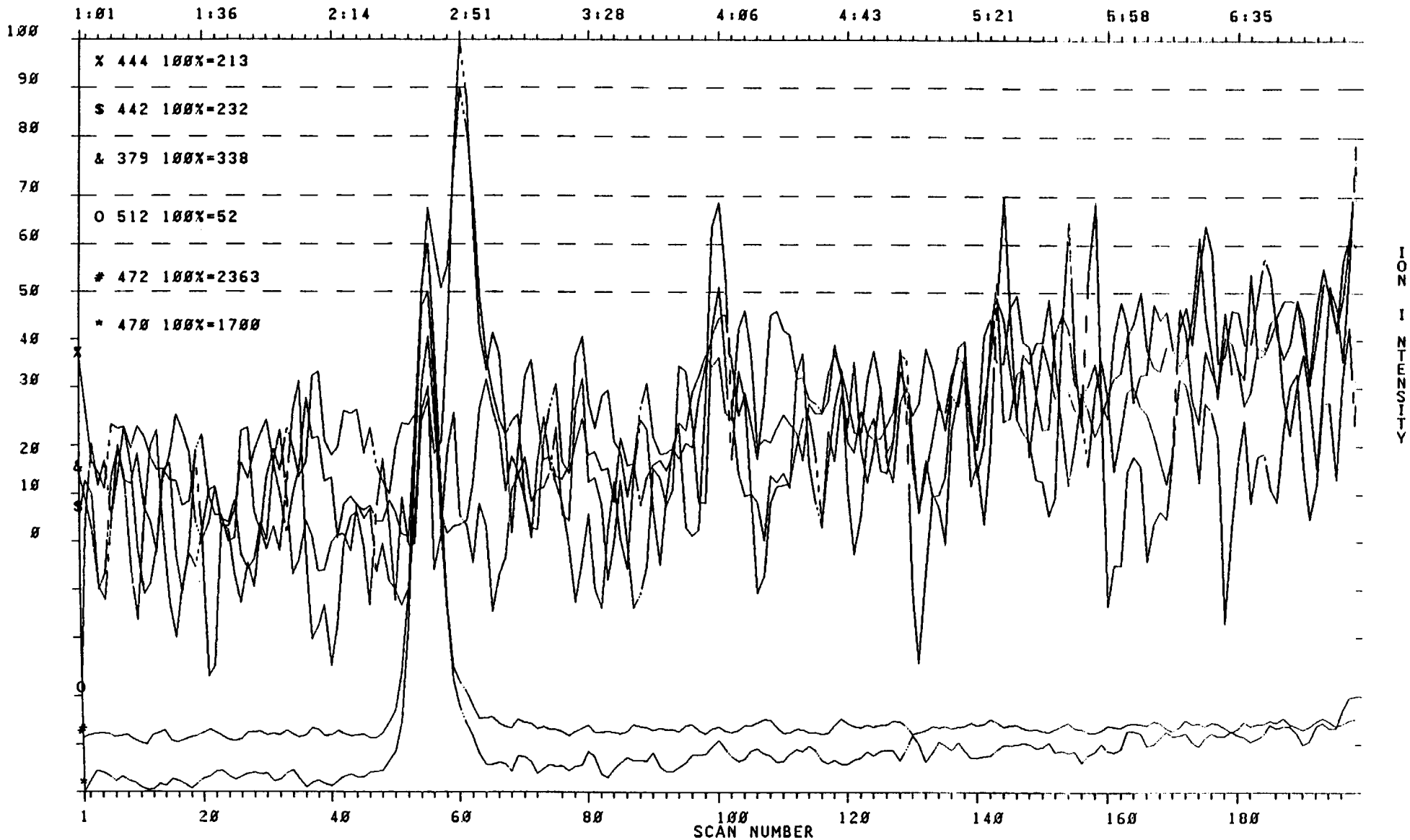
KRATOS MS25, DS55 SOFTWARE, RUN: CTO7ØØ83, WSU NAME: NBC5-Ø-B
SELECTED-ION MASS CHROMATOGRAMS FOR HEPTACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

FIGURE: 59

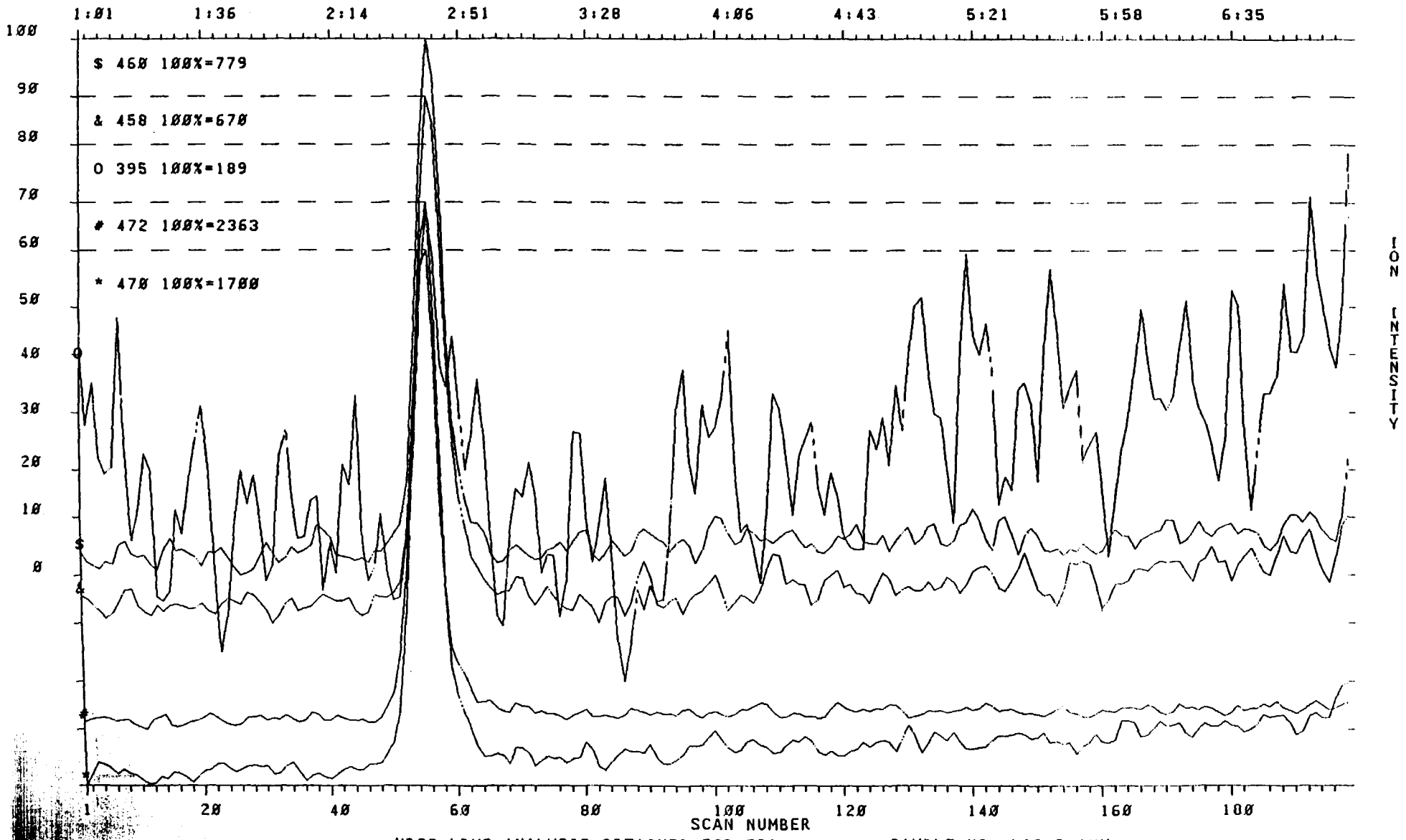
KRATOS MS25, DS55 SOFTWARE, RUN: CTO80083, WSU NAME: NBC5-0-B
SELECTED-ION MASS CHROMATOGRAMS FOR OCTACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

FIGURE: 60

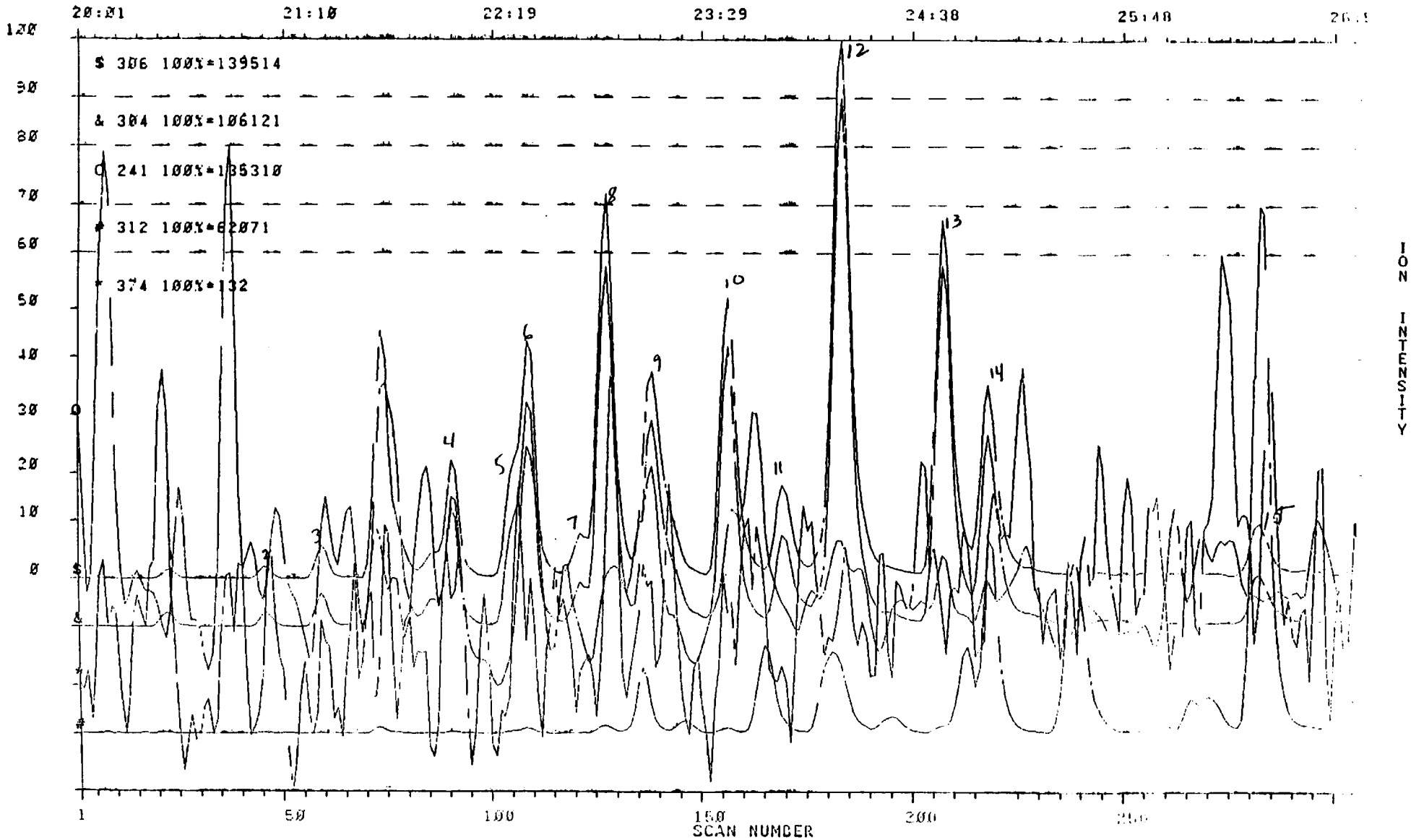
KRATOS MS25, DS55 SOFTWARE, RUN: CTO8ØØ83, WSU NAME: NBC5-Ø-B
SELECTED-ION MASS CHROMATOGRAMS FOR OCTACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

FIGURE: 61

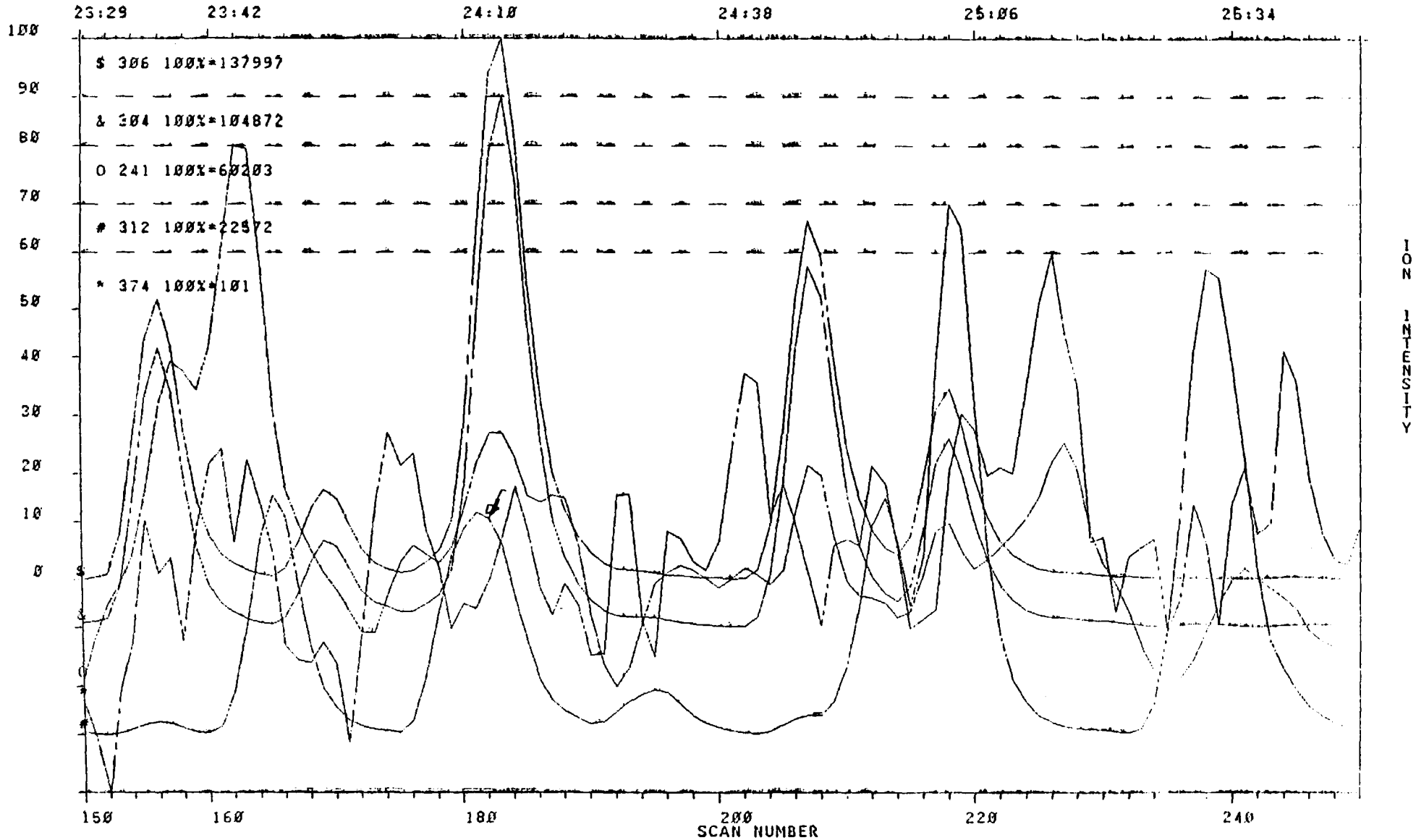
KRATOS MS25, DS55 SOFTWARE, RUN: CTO40106, WSU NAME: NBC5-1
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 13858

FIGURE 62

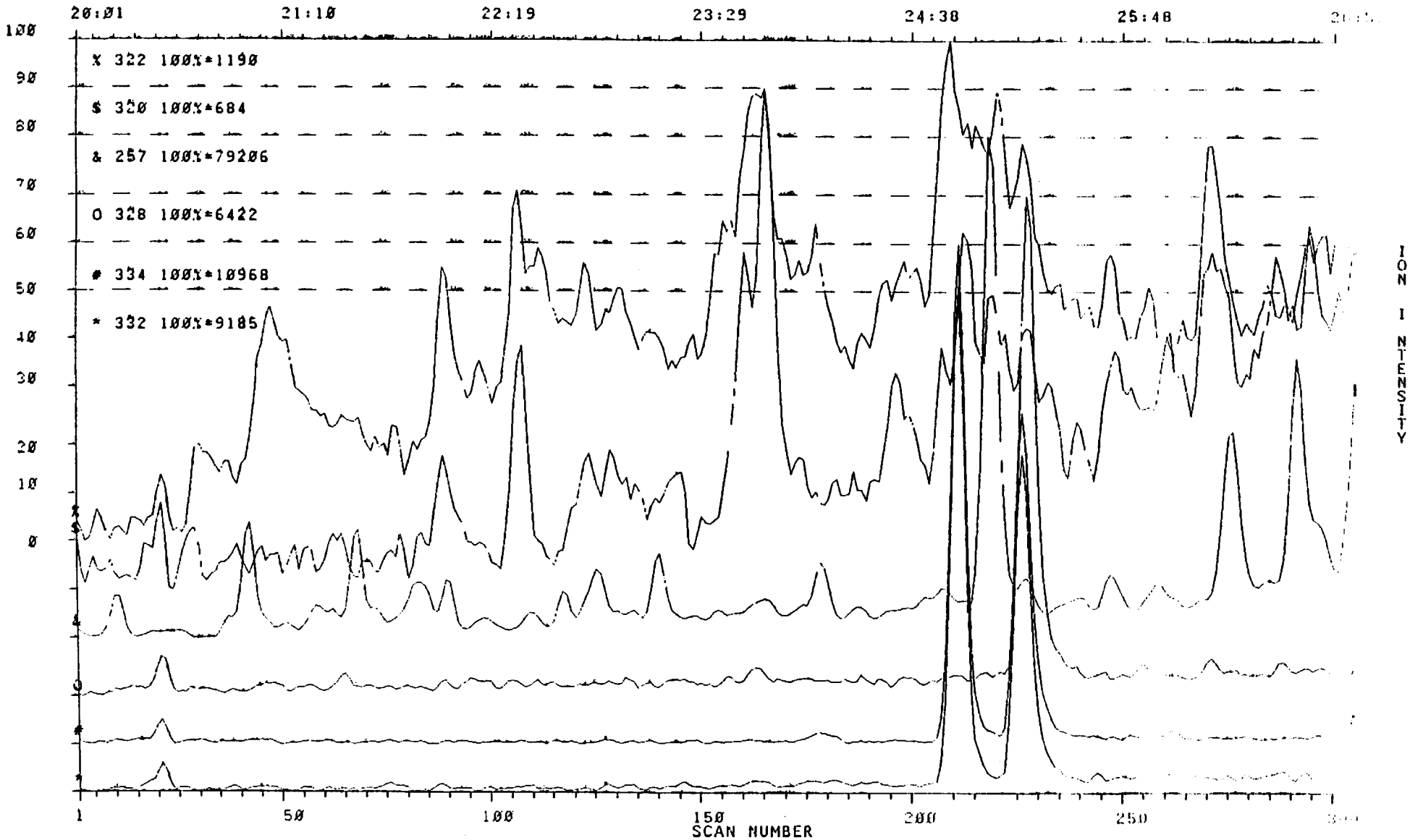
KRATOS MS25, DS55 SOFTWARE, RUN: CTO4Ø1Ø6, WSU NAME: NBC6-1
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 13850

FIGURE: 63

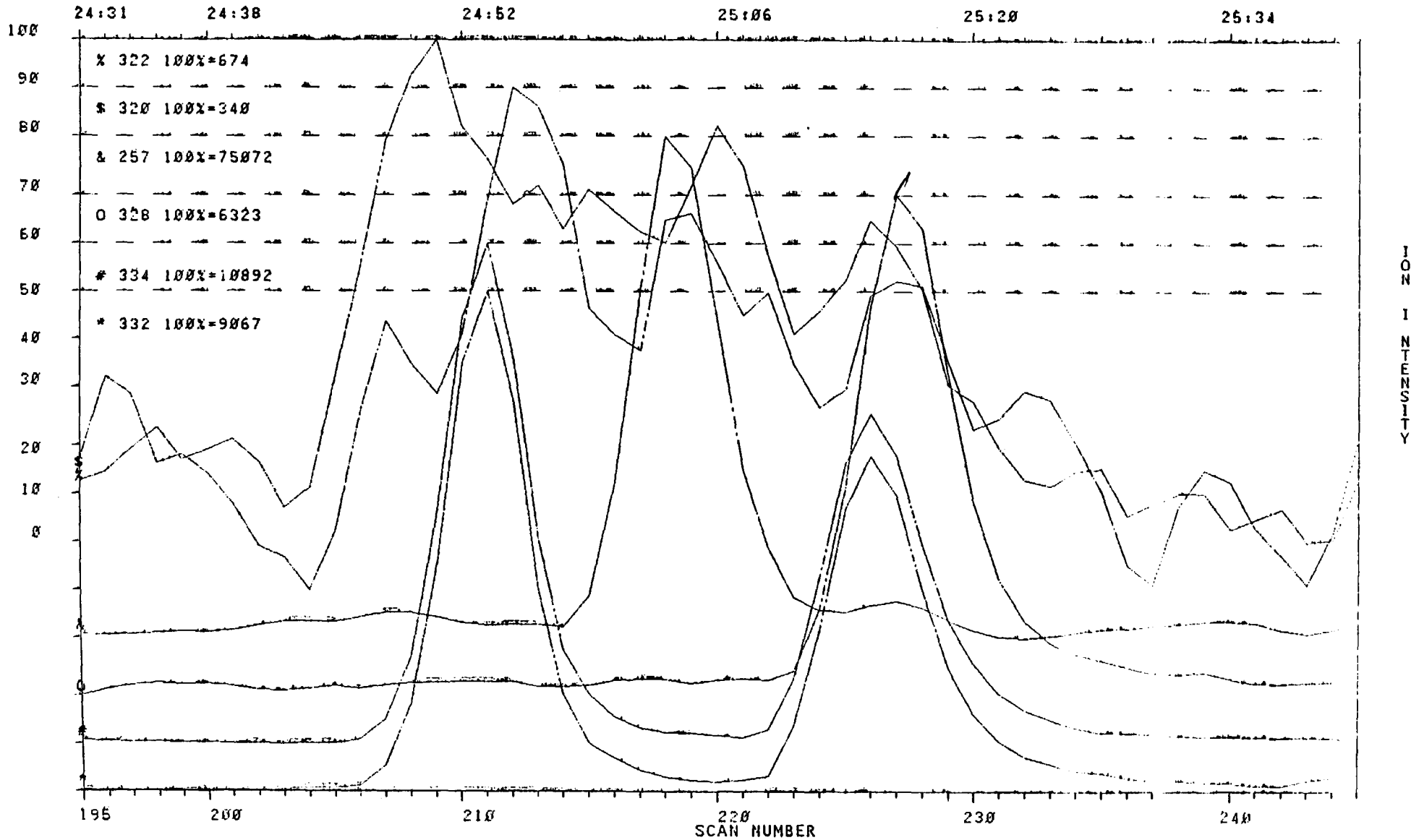
KRATOS MS25, DS55 SOFTWARE, RUN: CTO40106, WSU NAME: NBC5-1
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 13858

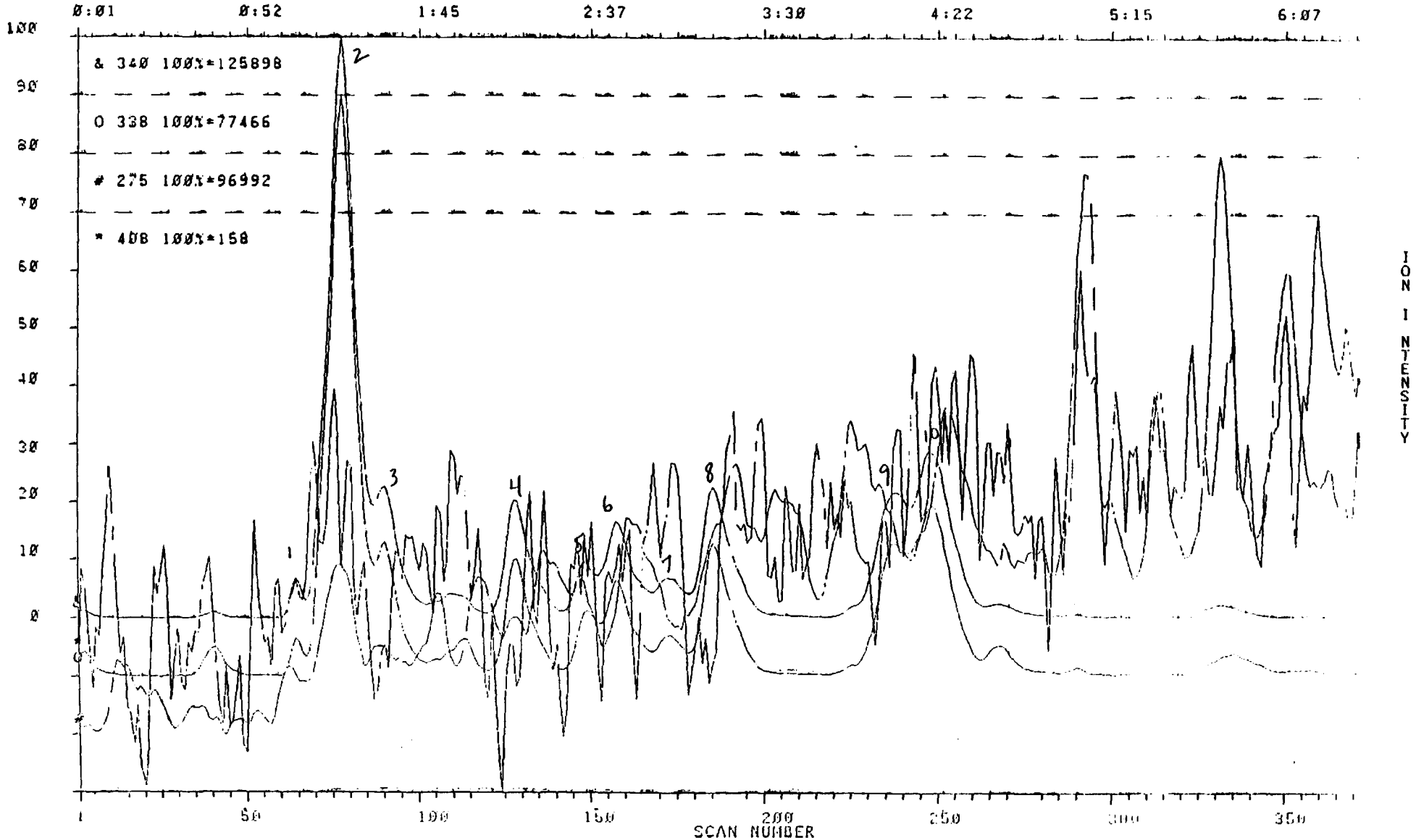
FIGURE: 64

KRATOS MS25, DS55 SOFTWARE, RUN: CTO4Ø1Ø6, WSU NAME: NBC5-1
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 13858
FIGURE: 65

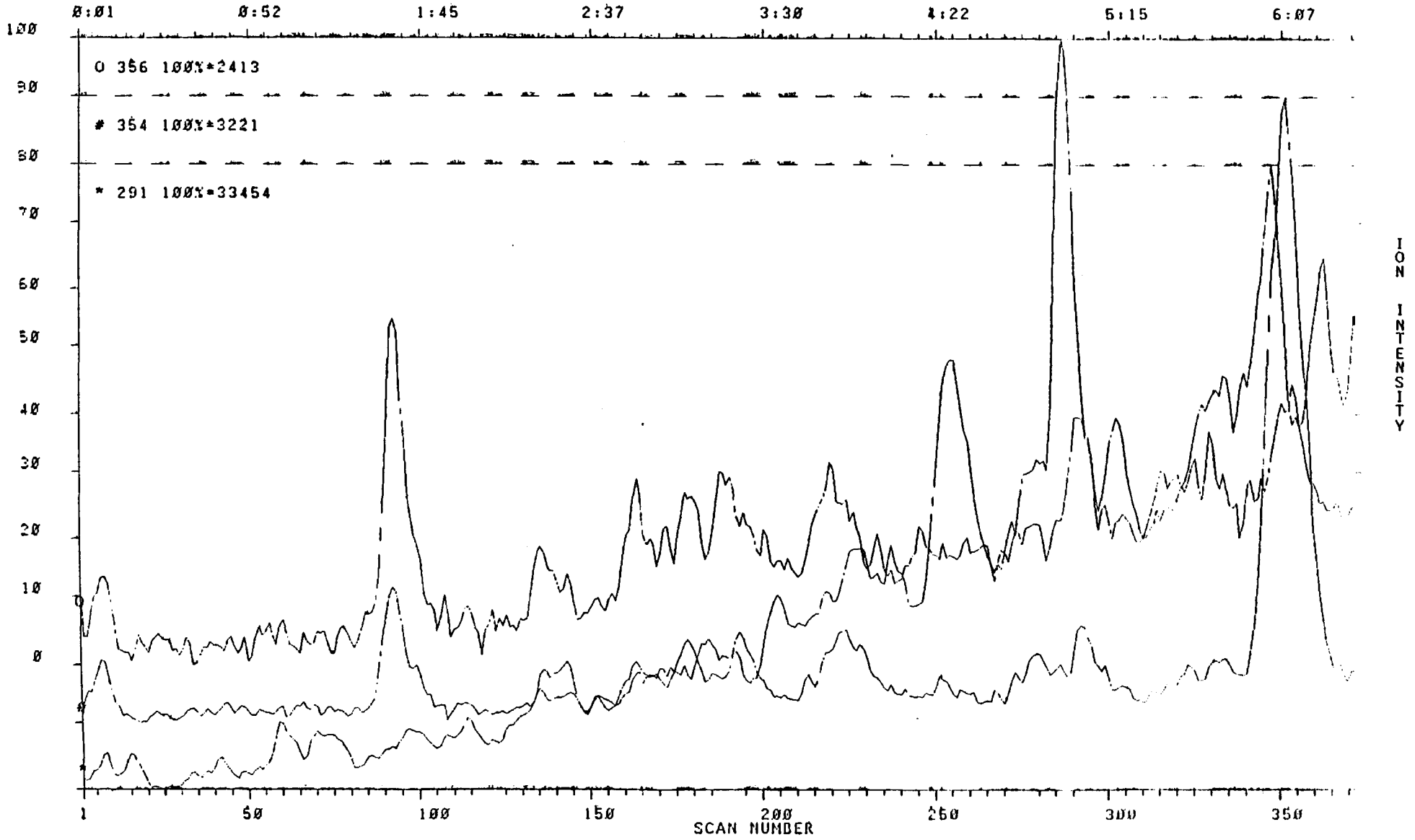
KRATOS MS25, DS55 SOFTWARE, RUN: CT050106, WSU NAME: NBC5-1
SELECTED-ION MASS CHROMATOGRAMS FOR PENTACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 13858

FIGURE 66

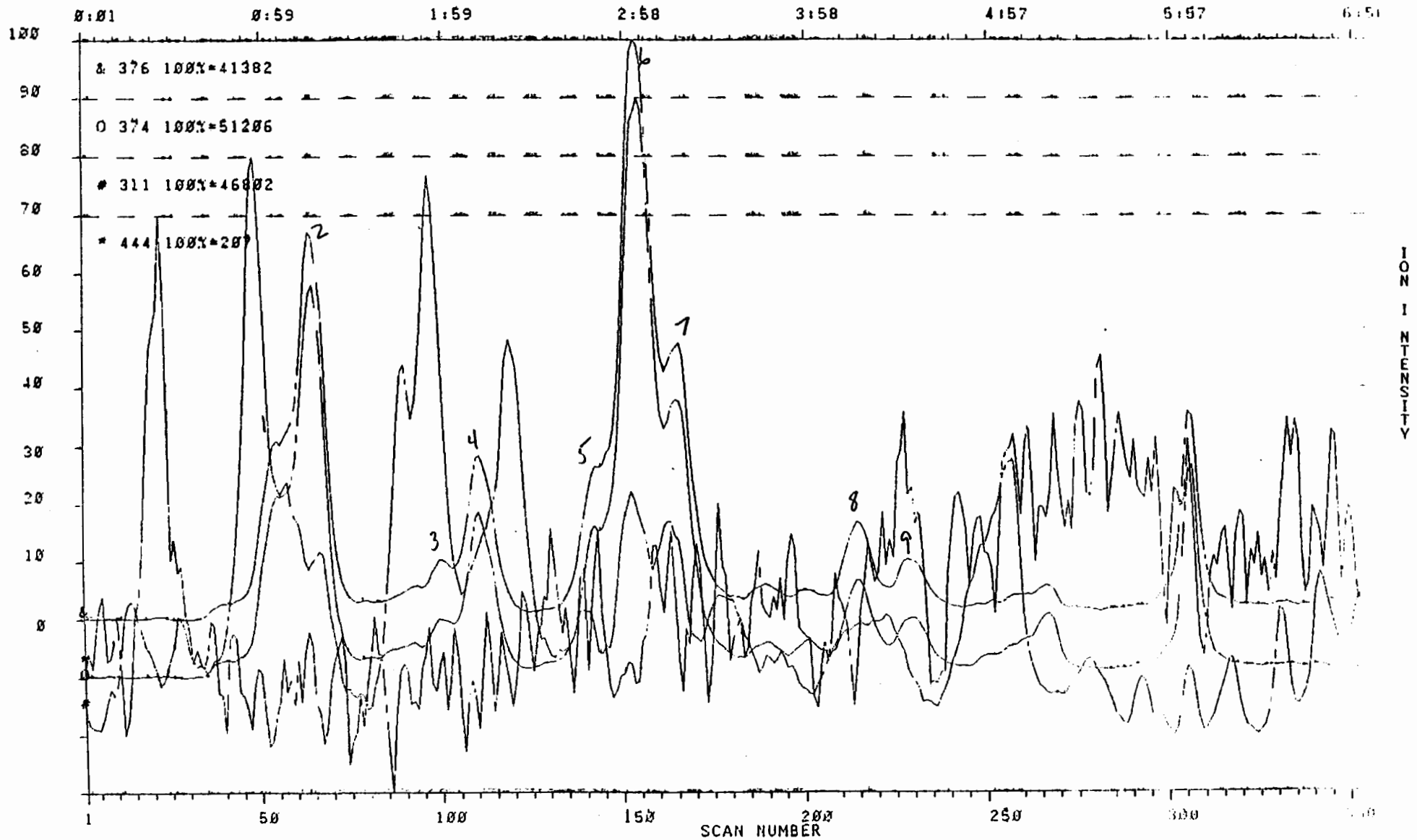
KRATOS MS2F, DS55 SOFTWARE, RUN: CTO5Ø1Ø6, WSU NAME: NBC5-1
SELECTED-ION MASS CHROMATOGRAMS FOR PENTACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 1385R

FIGURE: 67

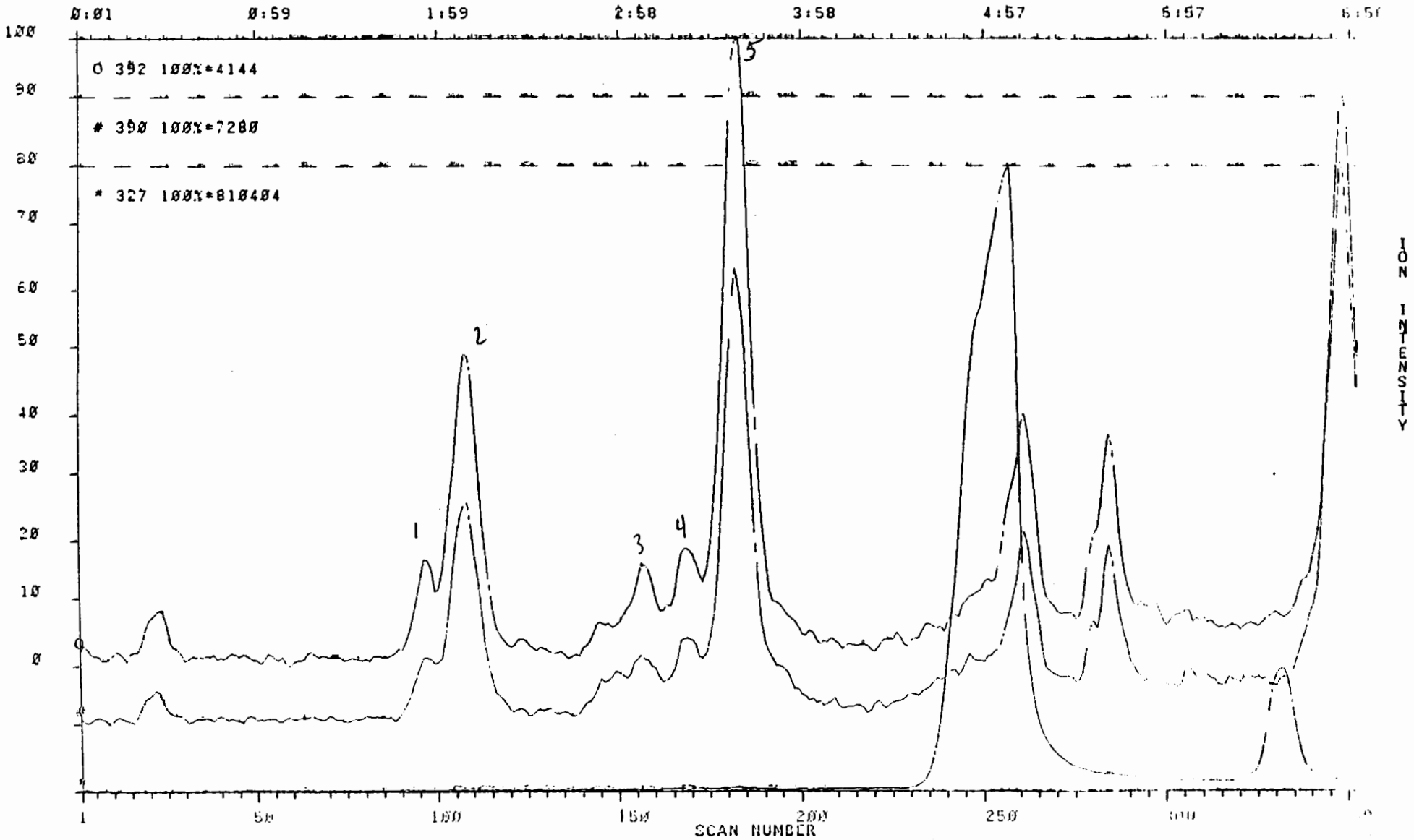
KRATOS MS25, DS55 SOFTWARE, RUN: CTO6Ø1Ø6, WSU NAME: NBC5-1
SELECTED-ION MASS CHROMATOGRAMS FOR HEXACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 13858

FIGURE: 68

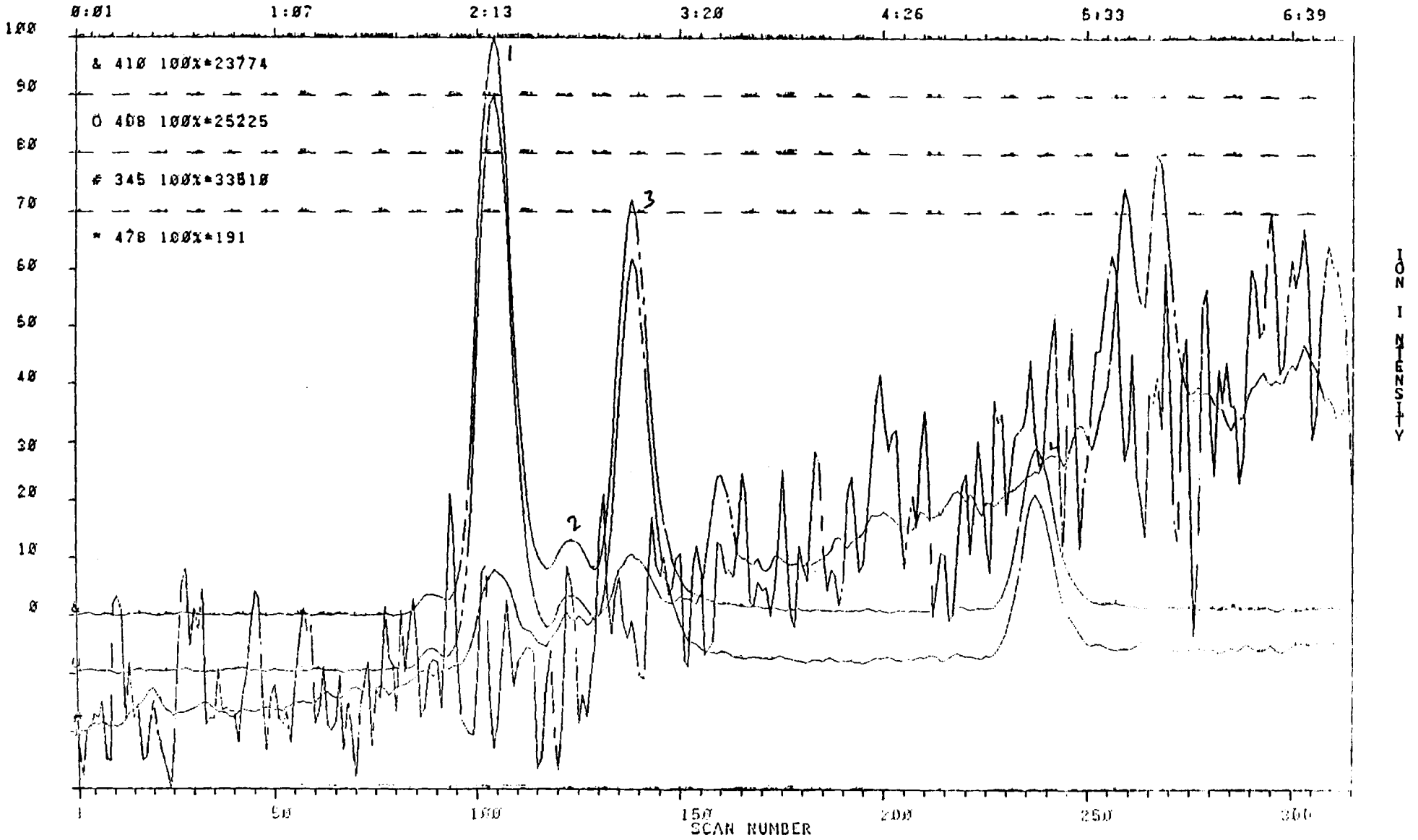
KRATOS MS25, DS55 SOFTWARE, RUN: CT060106, WSU NAME: NBC5-1
SELECTED-ION MASS CHROMATOGRAMS FOR HEXACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 13868

FIGURE: 69

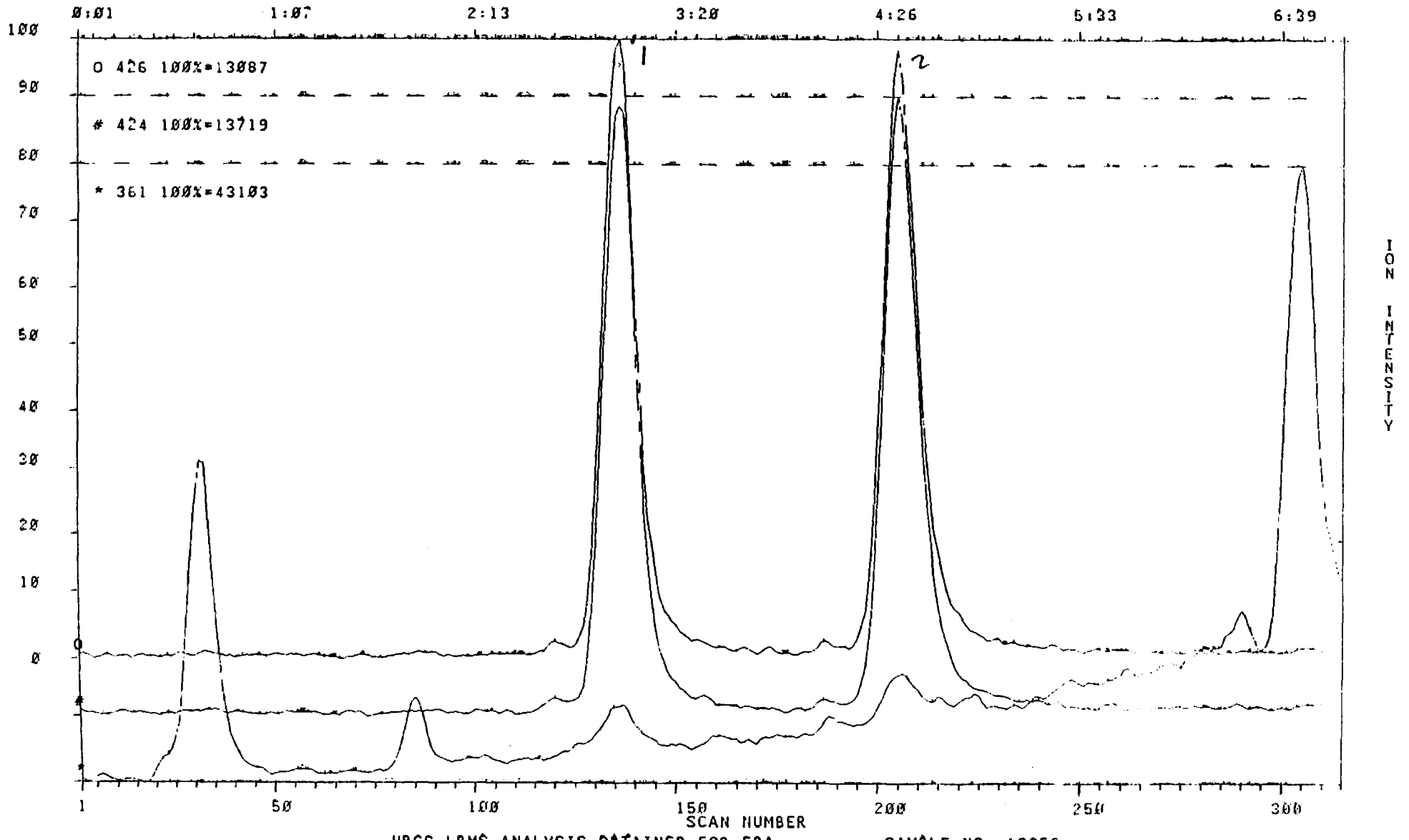
KRATOS MS25, DS55 SOFTWARE, RUN: CTO7Ø1Ø6, WSU NAME: NBC5-1
SELECTED-ION MASS CHROMATOGRAMS FOR HEPTACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 13858

FIGURE: 70

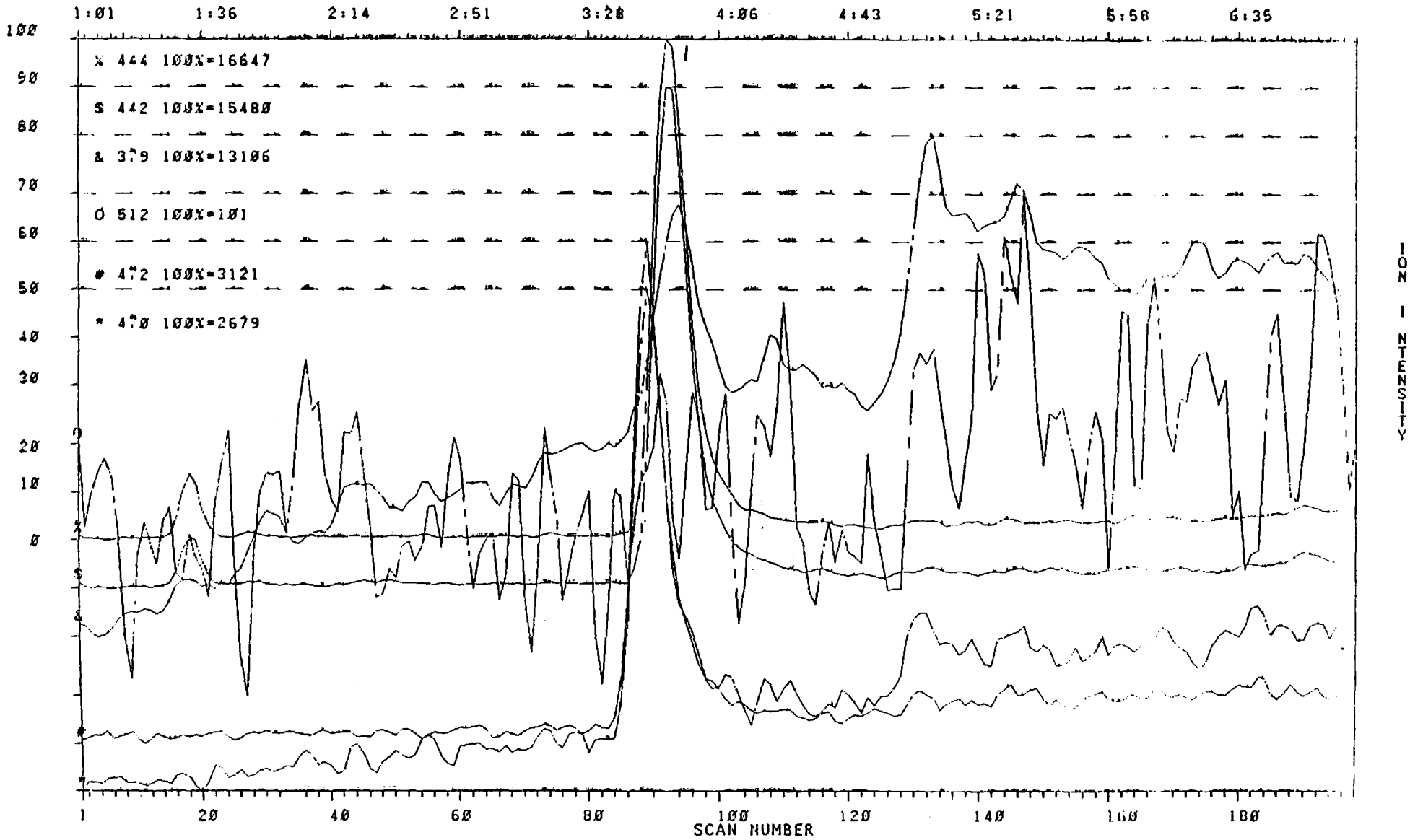
KRATOS MS25, DS55 SOFTWARE, RUN: CT07Ø1Ø6, WSU NAME: NBC5-1
SELECTED-ION MASS CHROMATOGRAMS FOR HEPTACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 13858

FIGURE: 71

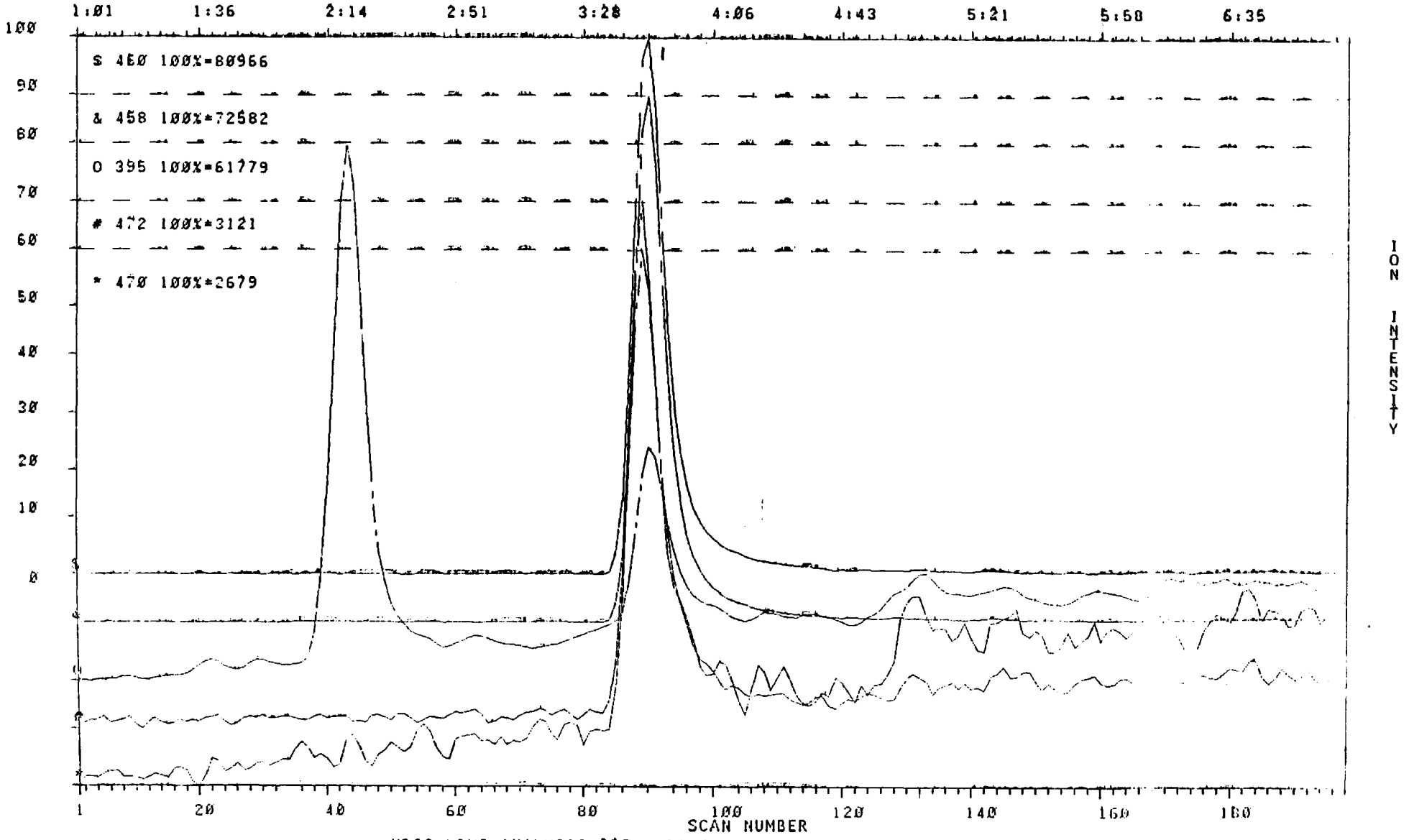
KRATOS MS25, DS55 SOFTWARE, RUN: CTO8Ø1Ø6, WSU NAME: NBC5-1
SELECTED-ION MASS CHROMATOGRAMS FOR OCTACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 13858

FIGURE: 72

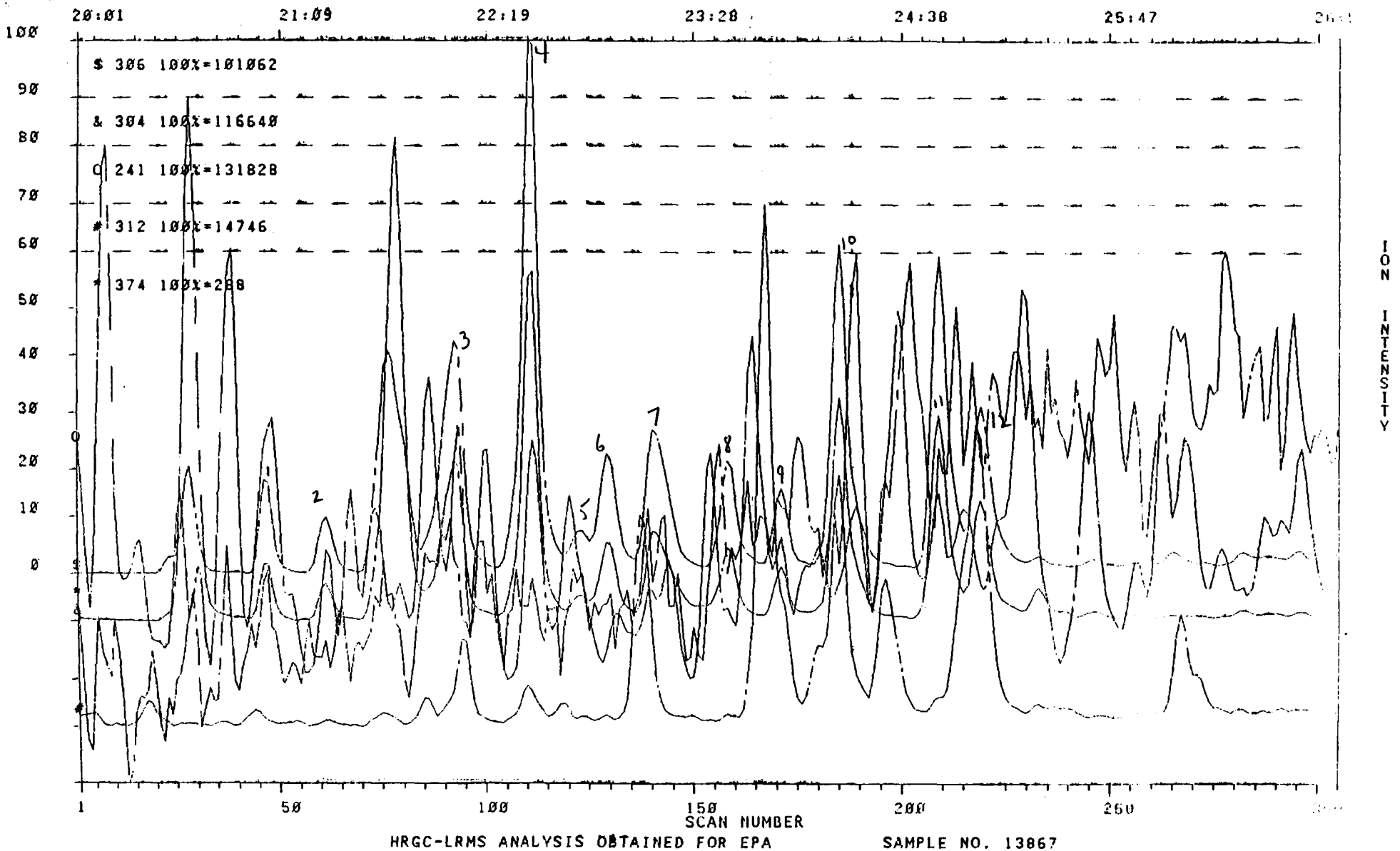
KRATOS MS25, DS55 SOFTWARE, RUN: CT080106, WSU NAME: NBC5-1
SELECTED-ION MASS CHROMATOGRAMS FOR OCTACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 13856

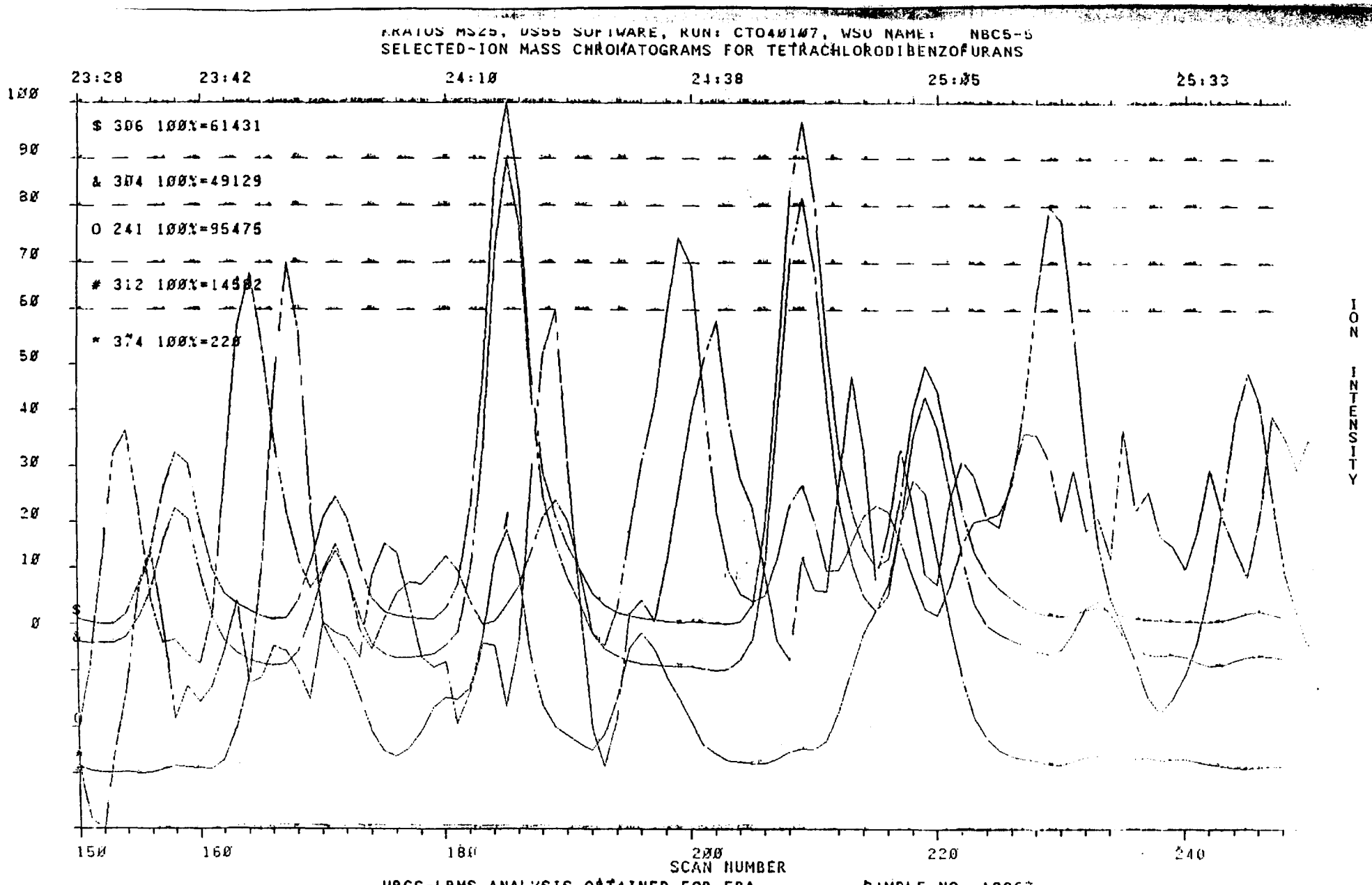
FIGURE: 73

KRATOS MS25, DS55 SOFTWARE, RUN: CTO4Ø1Ø7, WSU NAME: NBC5-6
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 13867

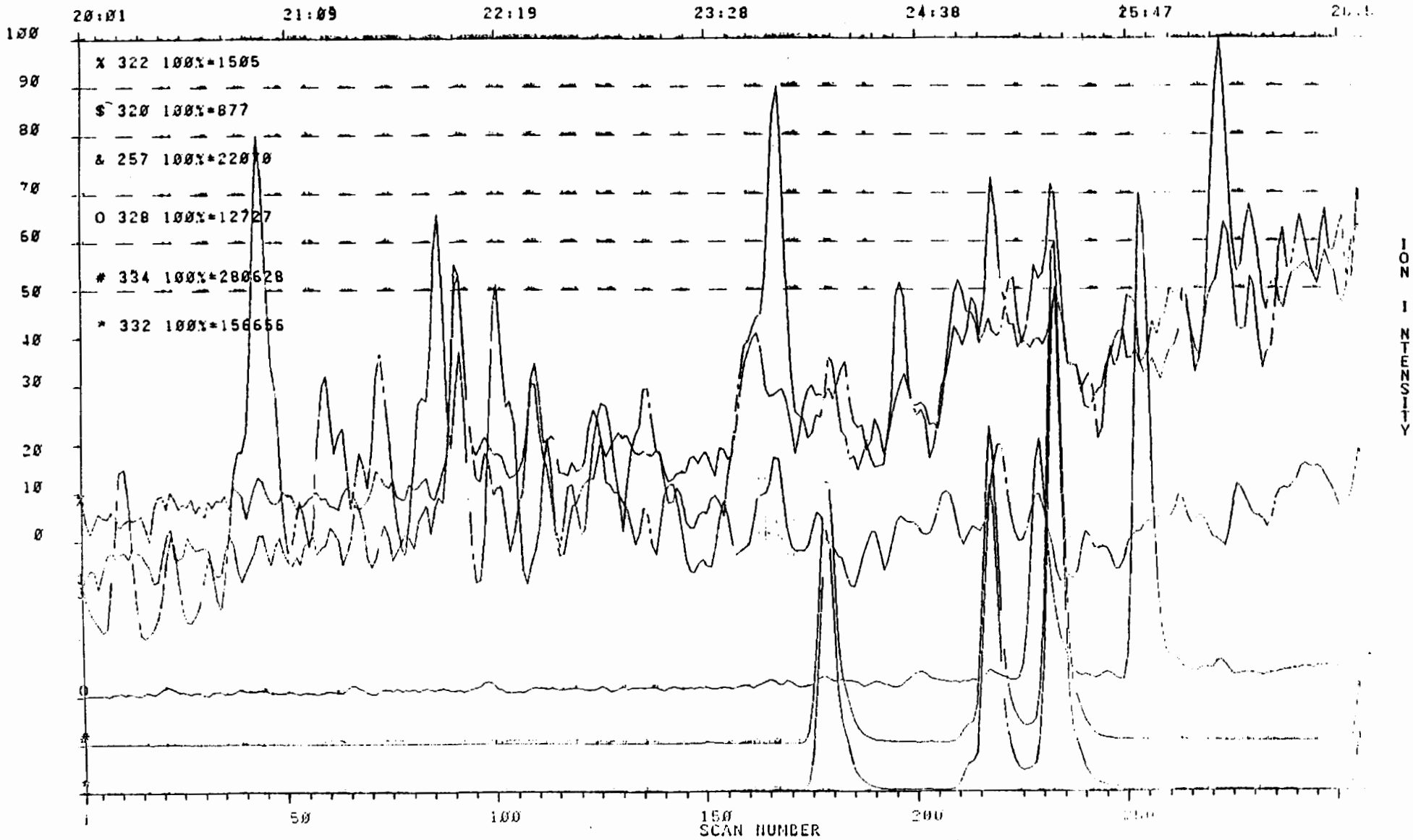
FIGURE: 74



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 13867

FIGURE: 75

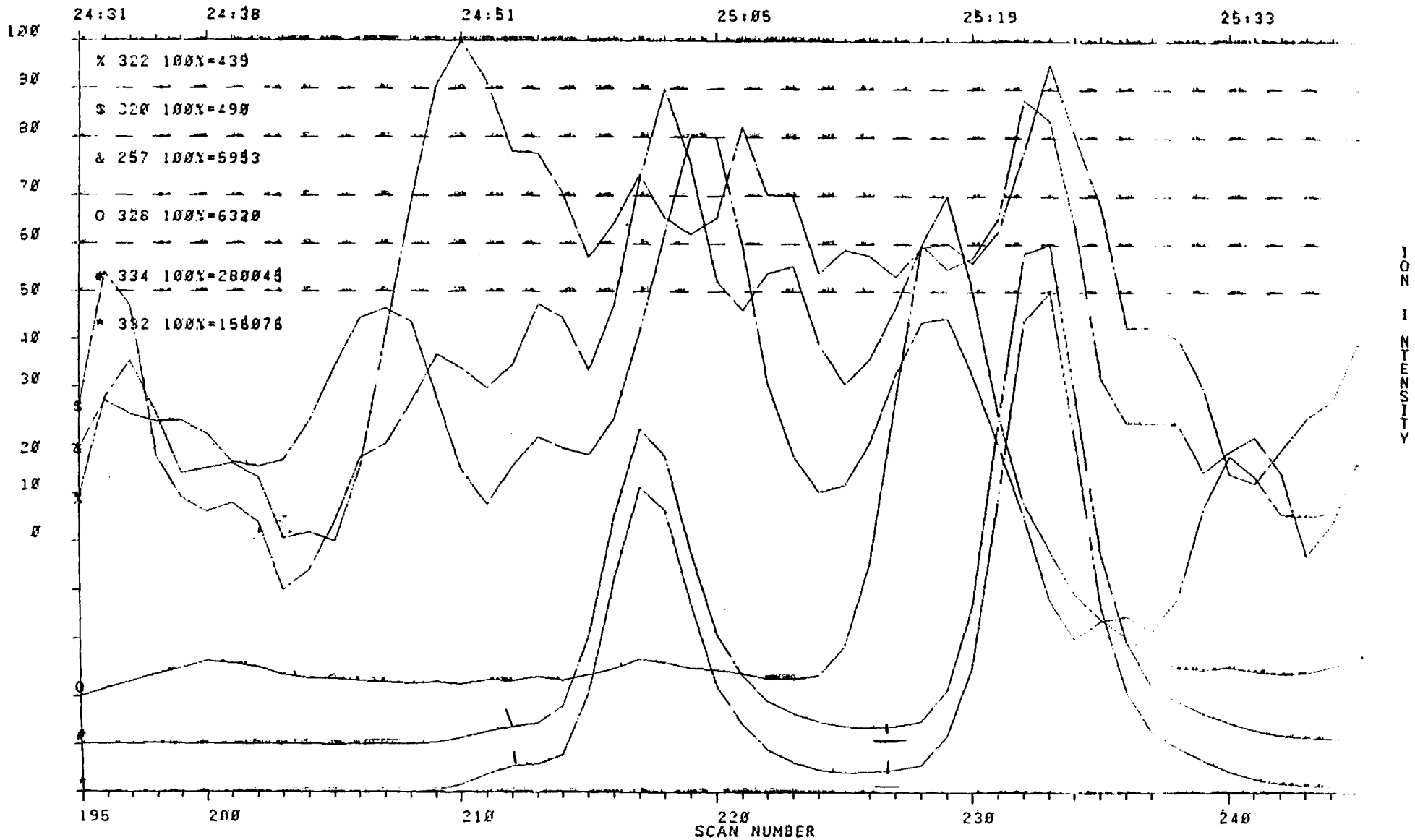
KRATOS MS25, DS55 SOFTWARE, RUN: CTO40107, WSU NAME: NBC5-5
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA . SAMPLE NO. 13867

FIGURE: 76

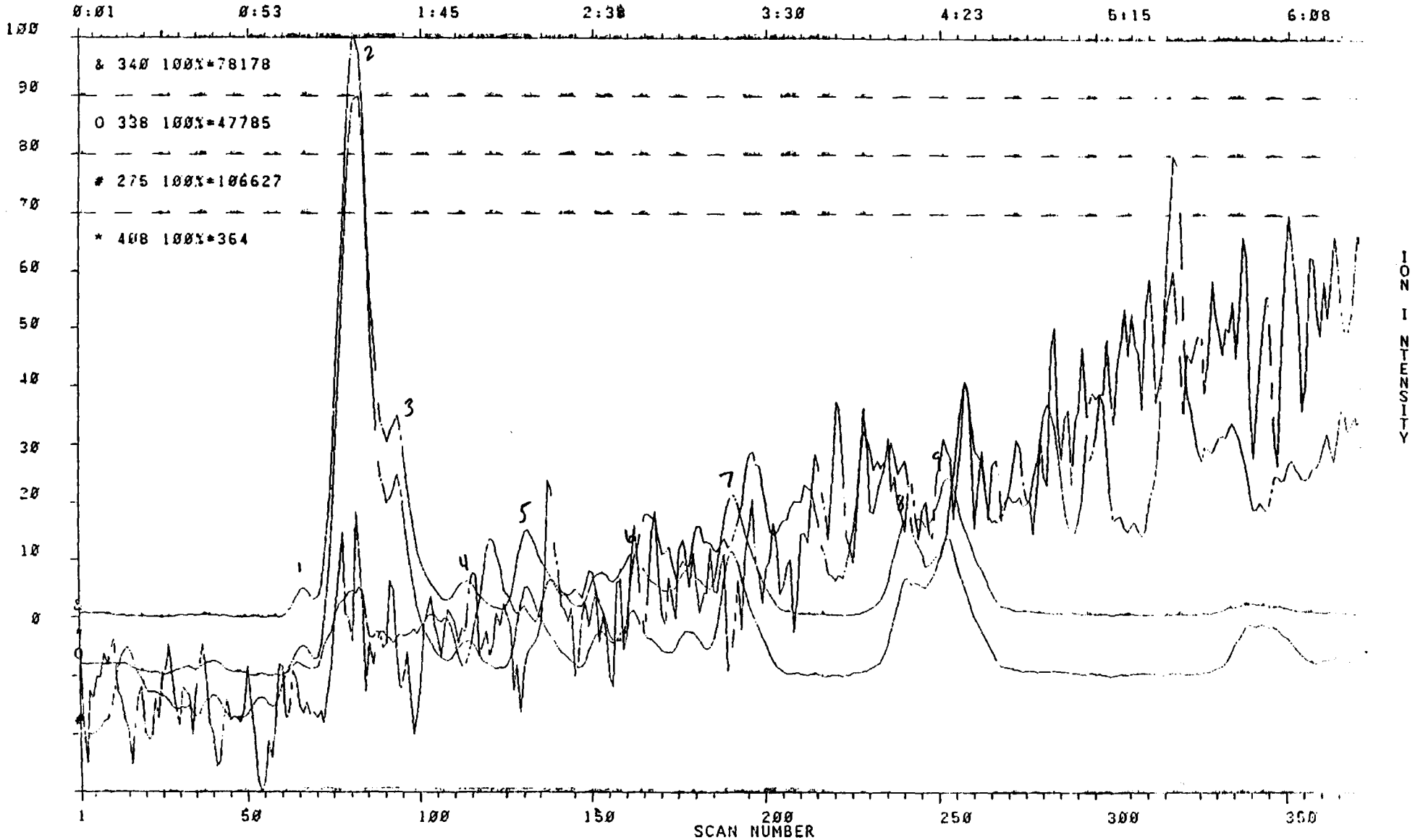
KRATOS MS25, DS55 SOFTWARE, RUN: CTO4Ø1Ø7, WSU NAME: NBC6-5
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 13867

FIGURE: 77

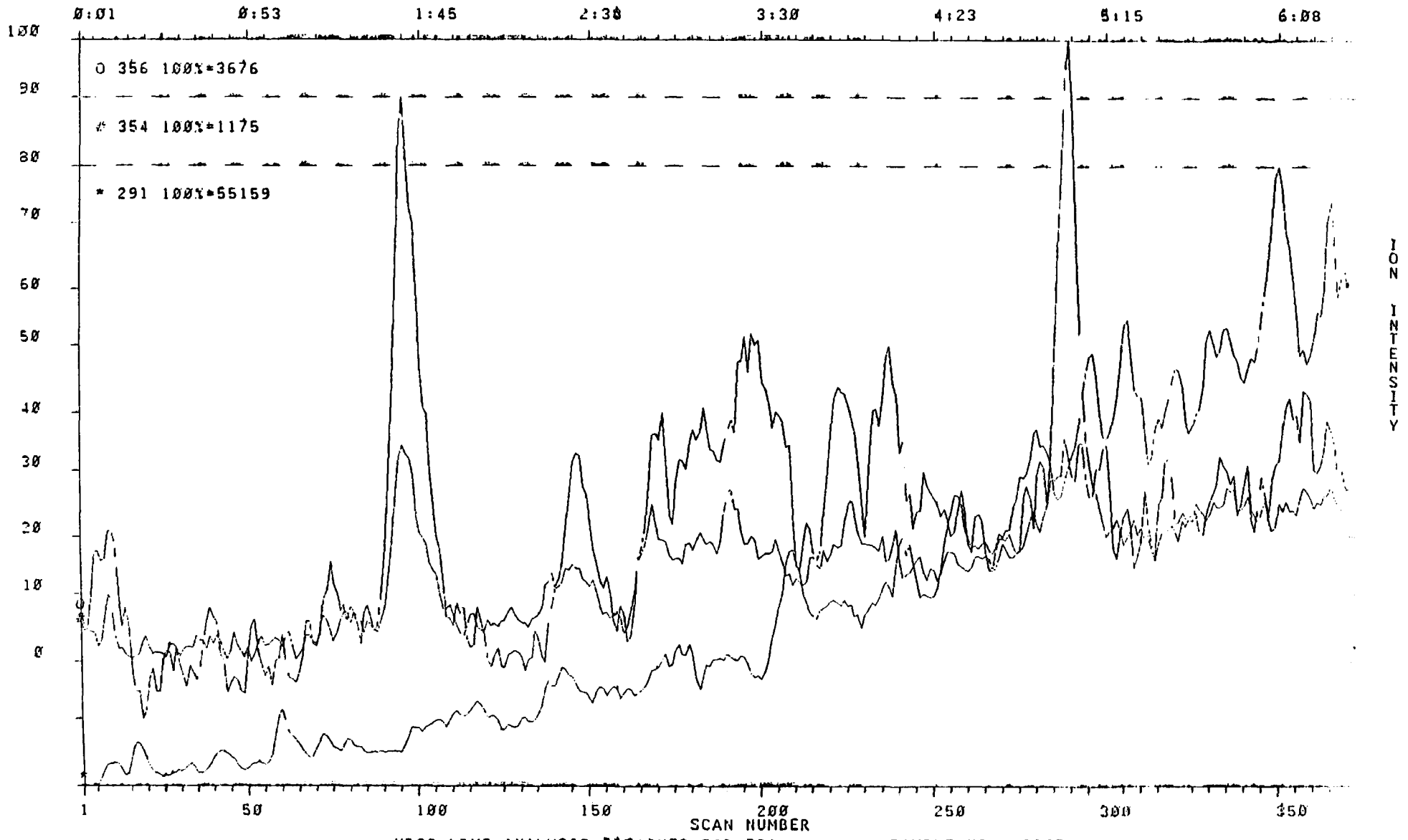
KRATOS MS25, DS55 SOFTWARE, RUN: CTO50107, WSU NAME: NBC5-5
SELECTED-ION MASS CHROMATOGRAMS FOR PENTACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 13867

FIGURE: 78

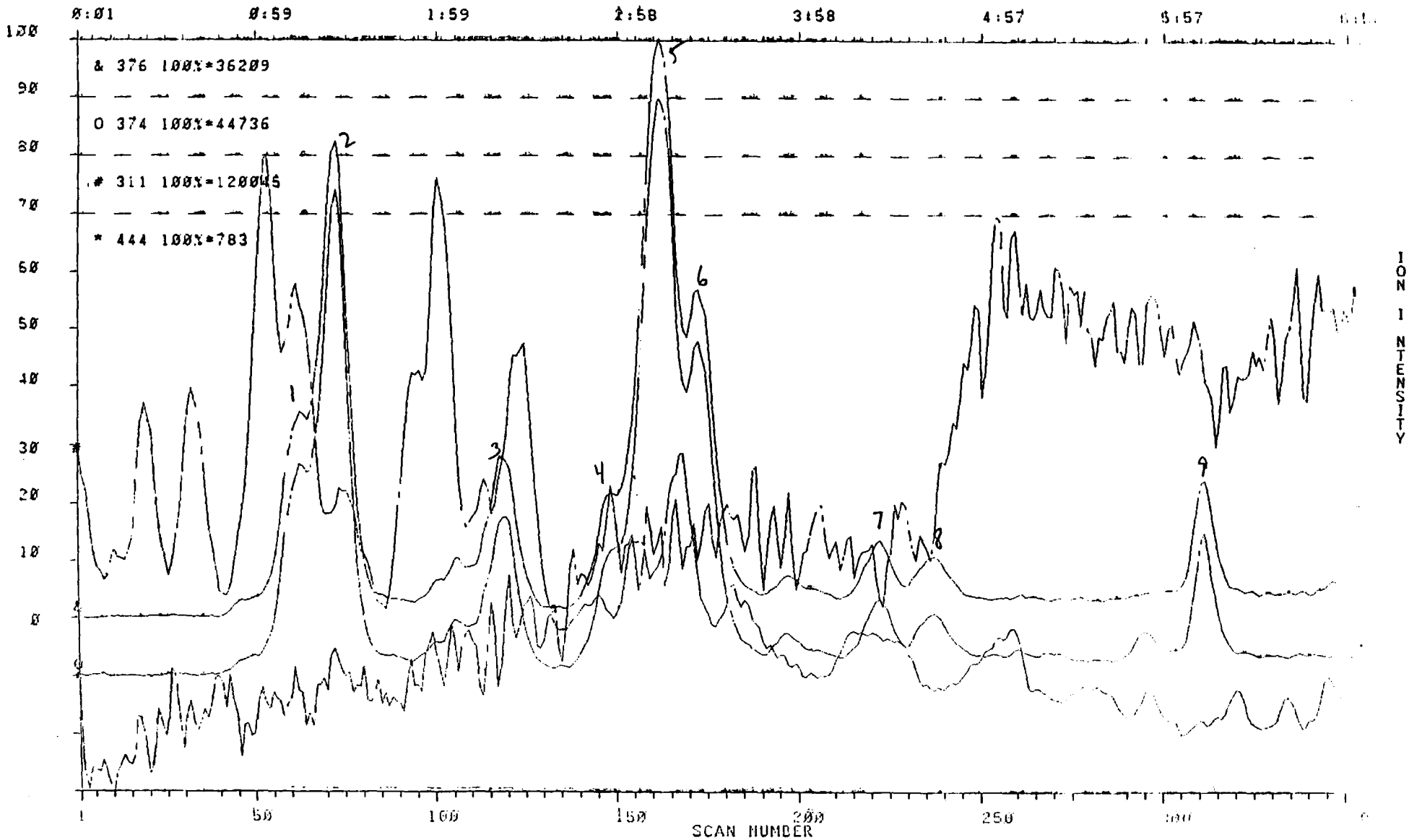
KRATOS MS25, DS55 SOFTWARE, RUN: CTO5Ø1Ø7, WSU NAME: NBC5-5
SELECTED-ION MASS CHROMATOGRAMS FOR PENTACHLORODIBENZO-P-DIOXINS



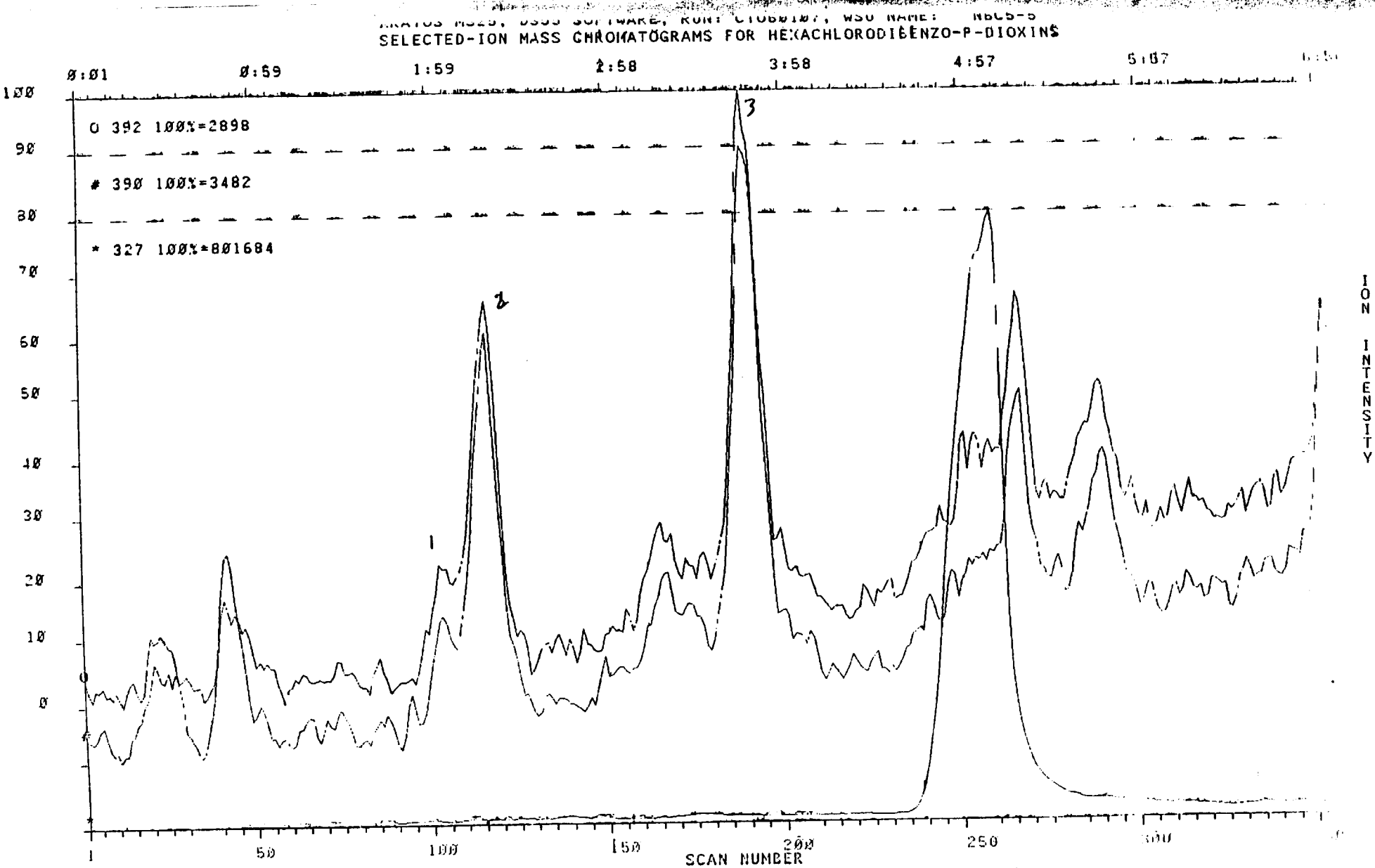
HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 13867

FIGURE:79

KRATOS MS25, DS55 SOFTWARE, RUN: CTO601Ø7, WSU NAME: NBC5-5
SELECTED-ION MASS CHROMATOGRAMS FOR HEXACHLORODIBENZOFURANS



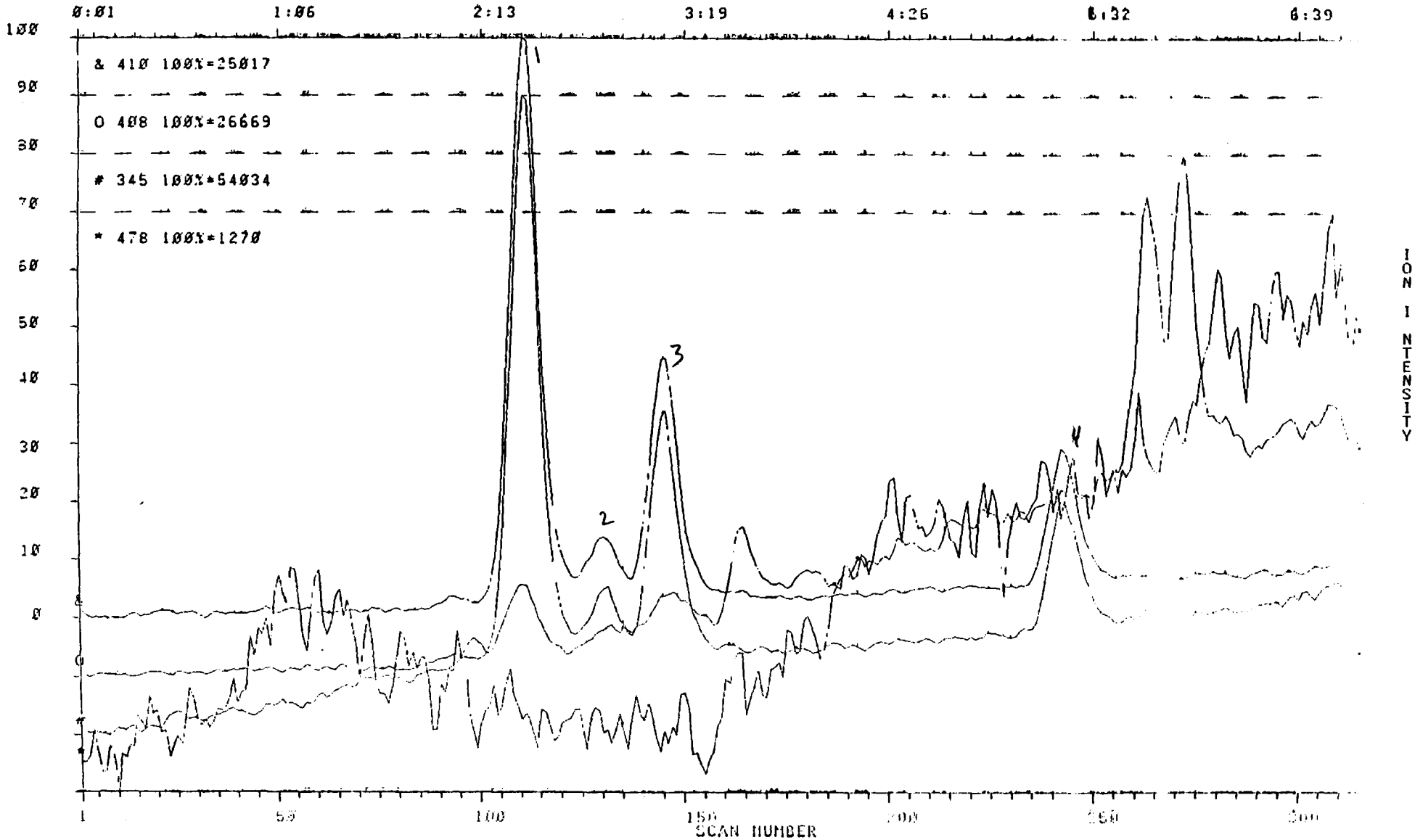
HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 13867
FIGURE: 80



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 13867

FIGURE: 81

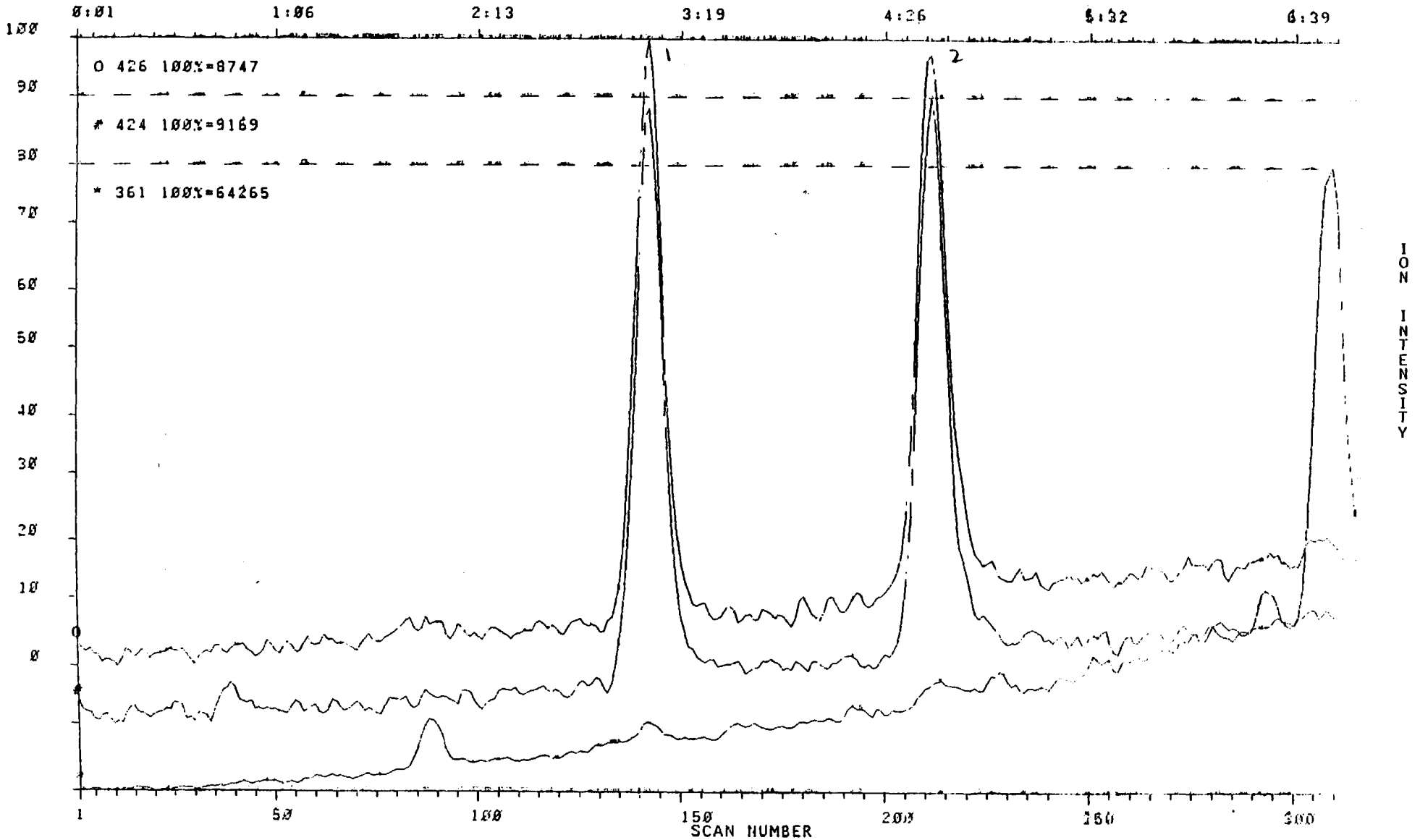
KRATOS MS25, DS55 SOFTWARE, RUN: CTO701Ø7, WSU NAME: NBC5-6
SELECTED-ION MASS CHROMATOGRAMS FOR HEPTACHLORODIBENZOFURANS



HPGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 13867
FIGURE: 82

KRATOS MS25, DS55 SOFTWARE, RUN: CTO70107, WSU NAME: NBC5-5

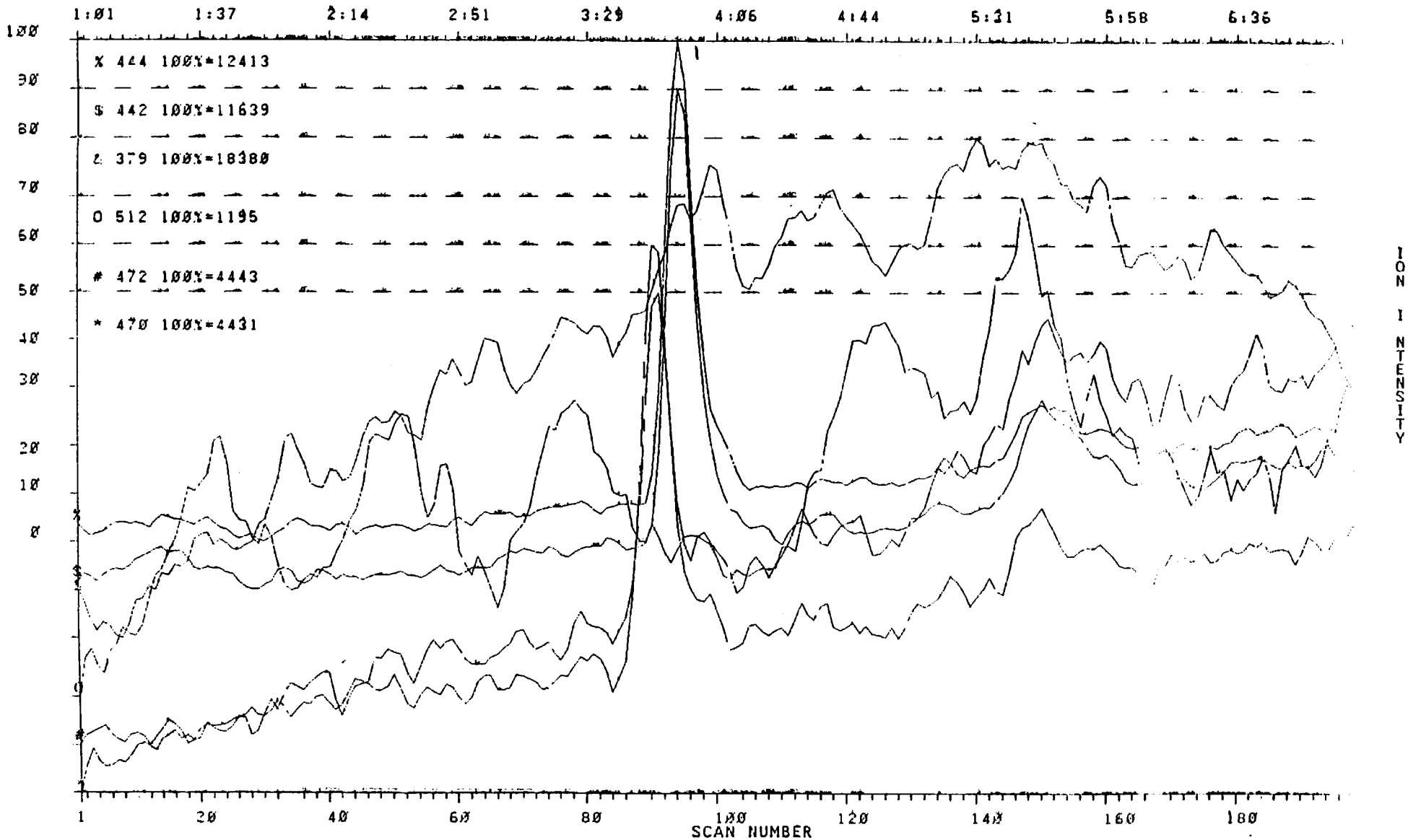
SELECTED-ION MASS CHROMATOGRAMS FOR HEPTACHLORODIBENZO-P-DIOXINS



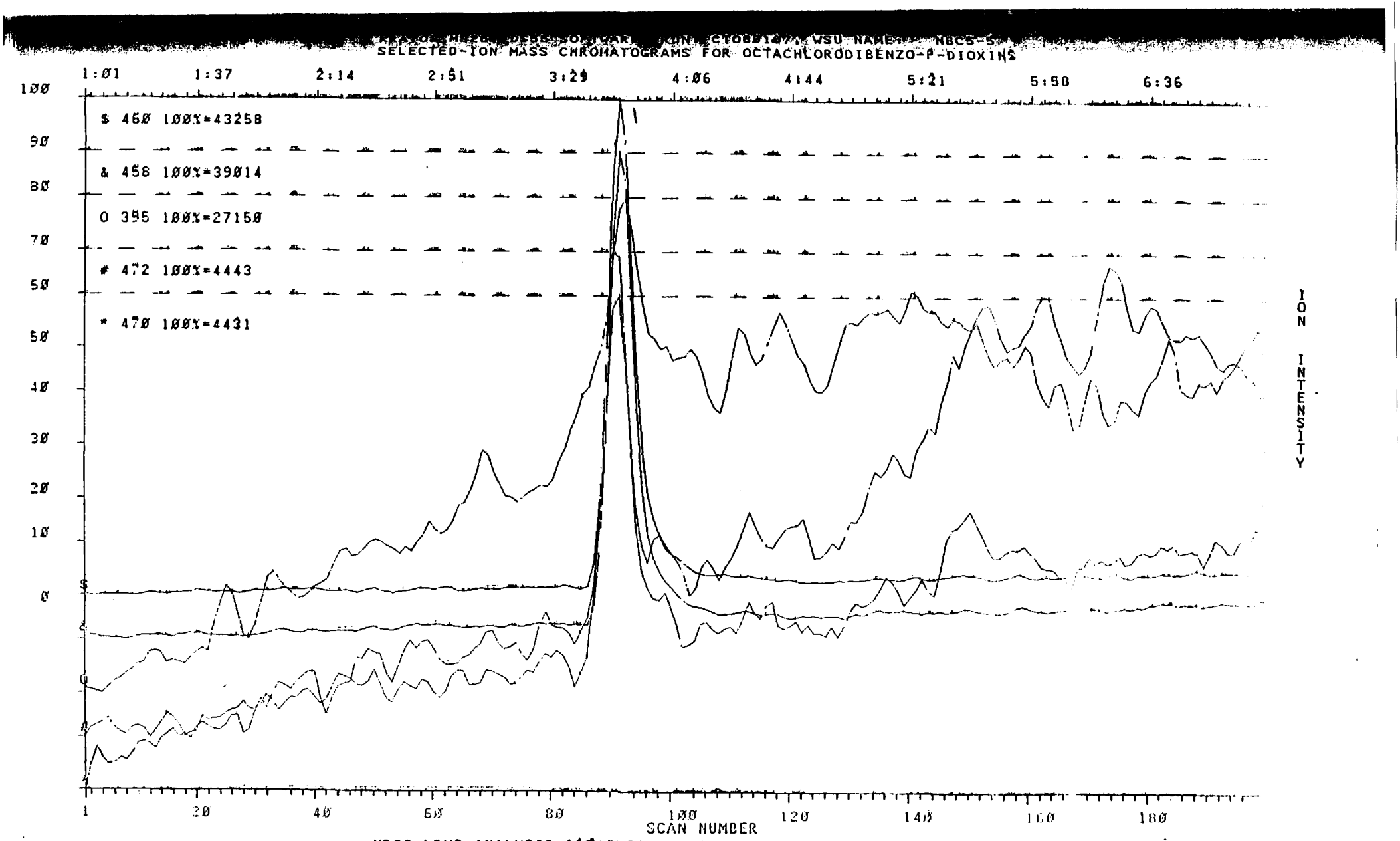
HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 13867

FIGURE: 83

KRATOS MS25, DS55 SOFTWARE, RUN: CTO80107, WSU NAME: NBC5-5
SELECTED-ION MASS CHROMATOGRAMS FOR OCTACHLORODIBENZOFURANS



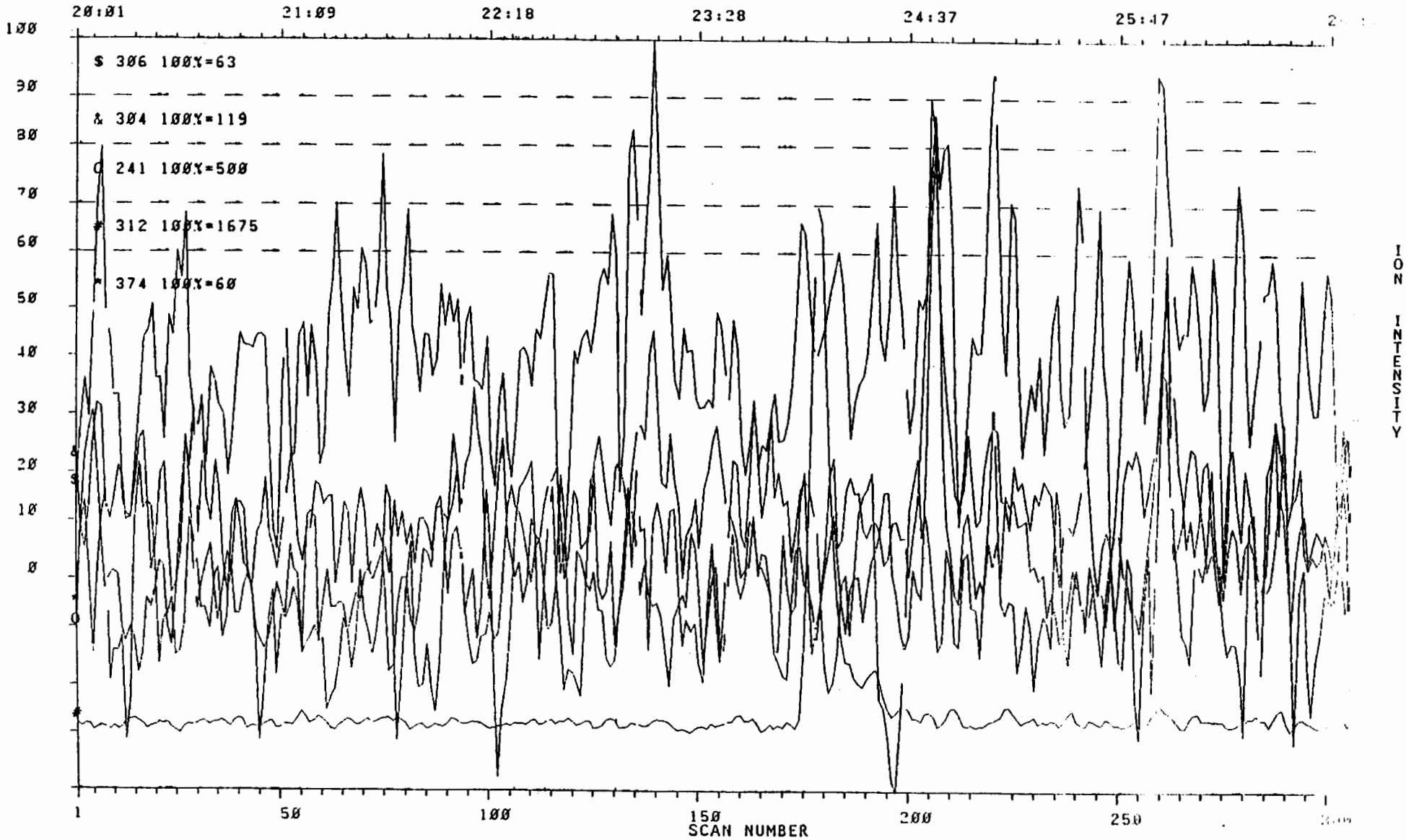
HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 13867
FIGURE: 84



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 13867

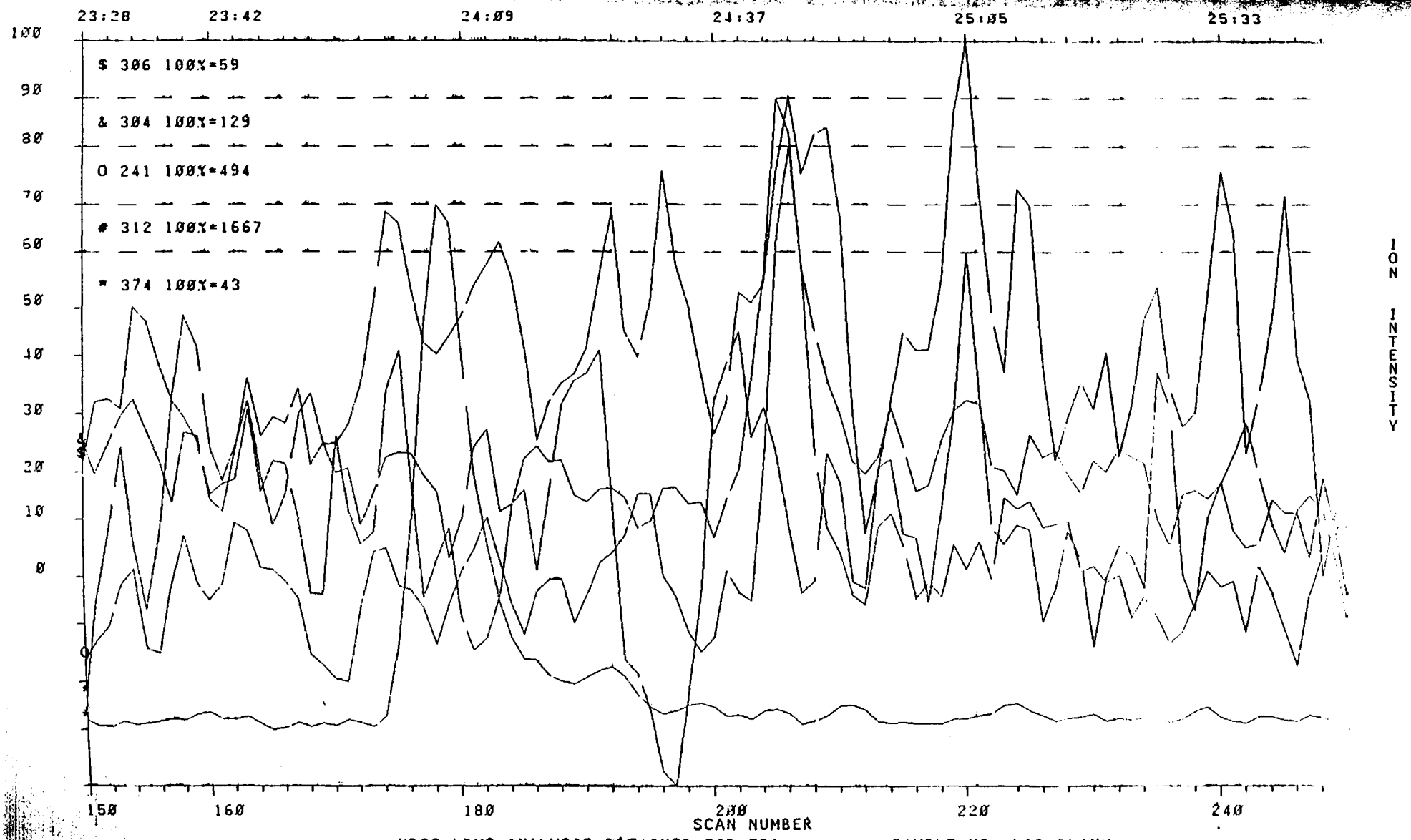
FIGURE: 85

KRATOS MS25, DS55 SOFTWARE, RUN: CTO4Ø1Ø9, WSU NAME: NBC5-ØB-B
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

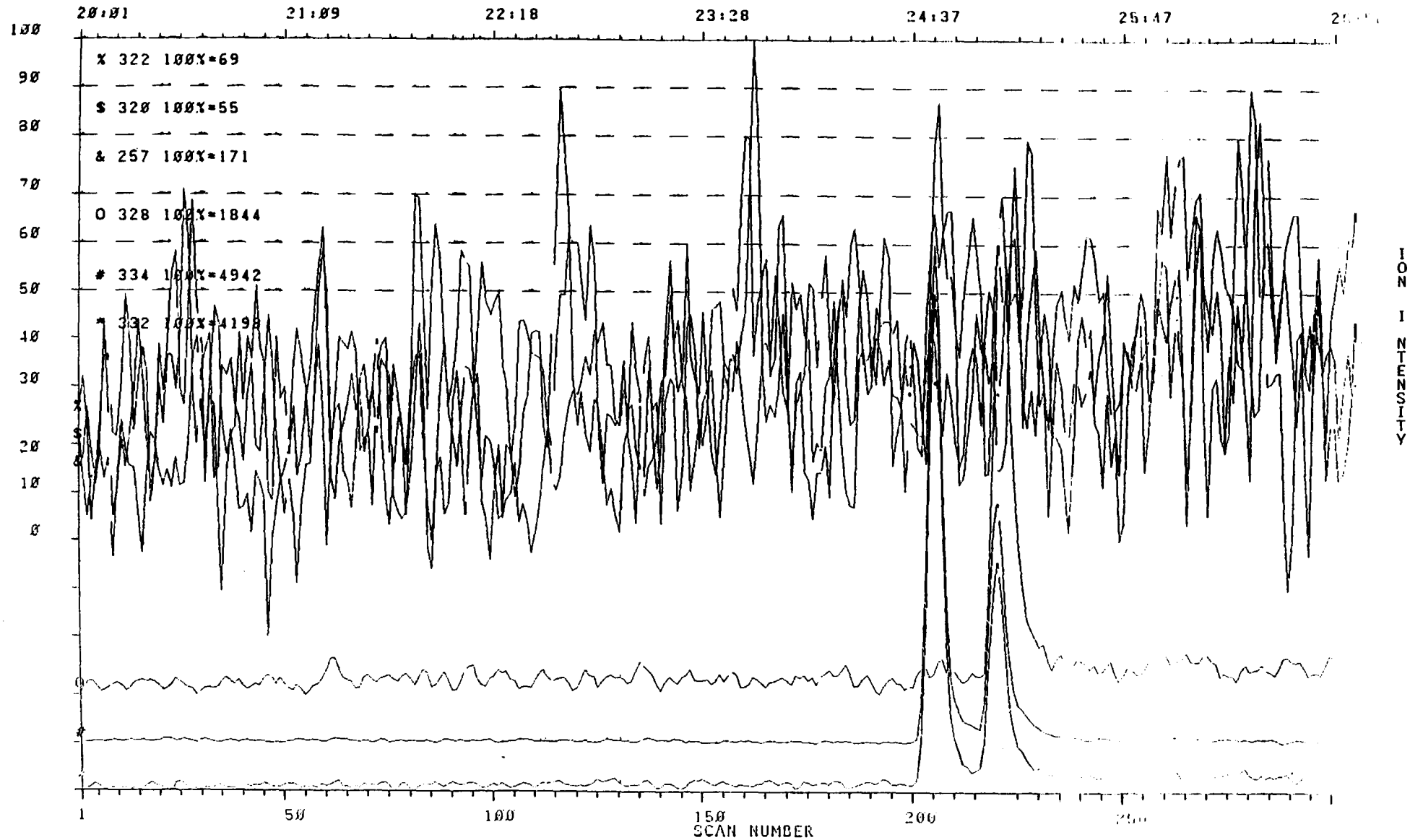
FIGURE: 86



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

FIGURE: 87

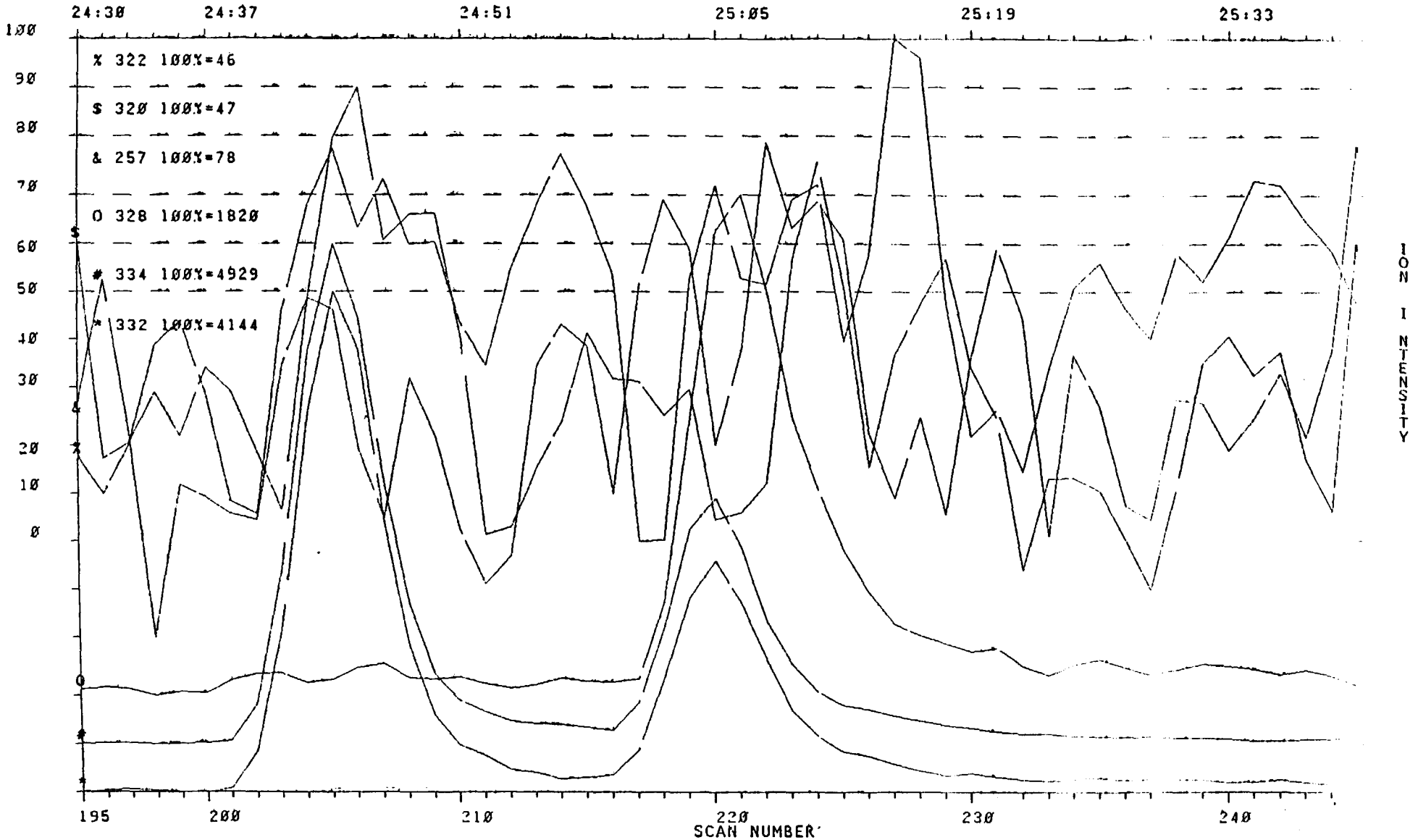
KRATOS MS25, DS55 SOFTWARE, RUN: CTO40109, WSU NAME: NBC5-0B-B
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

FIGURE: 88

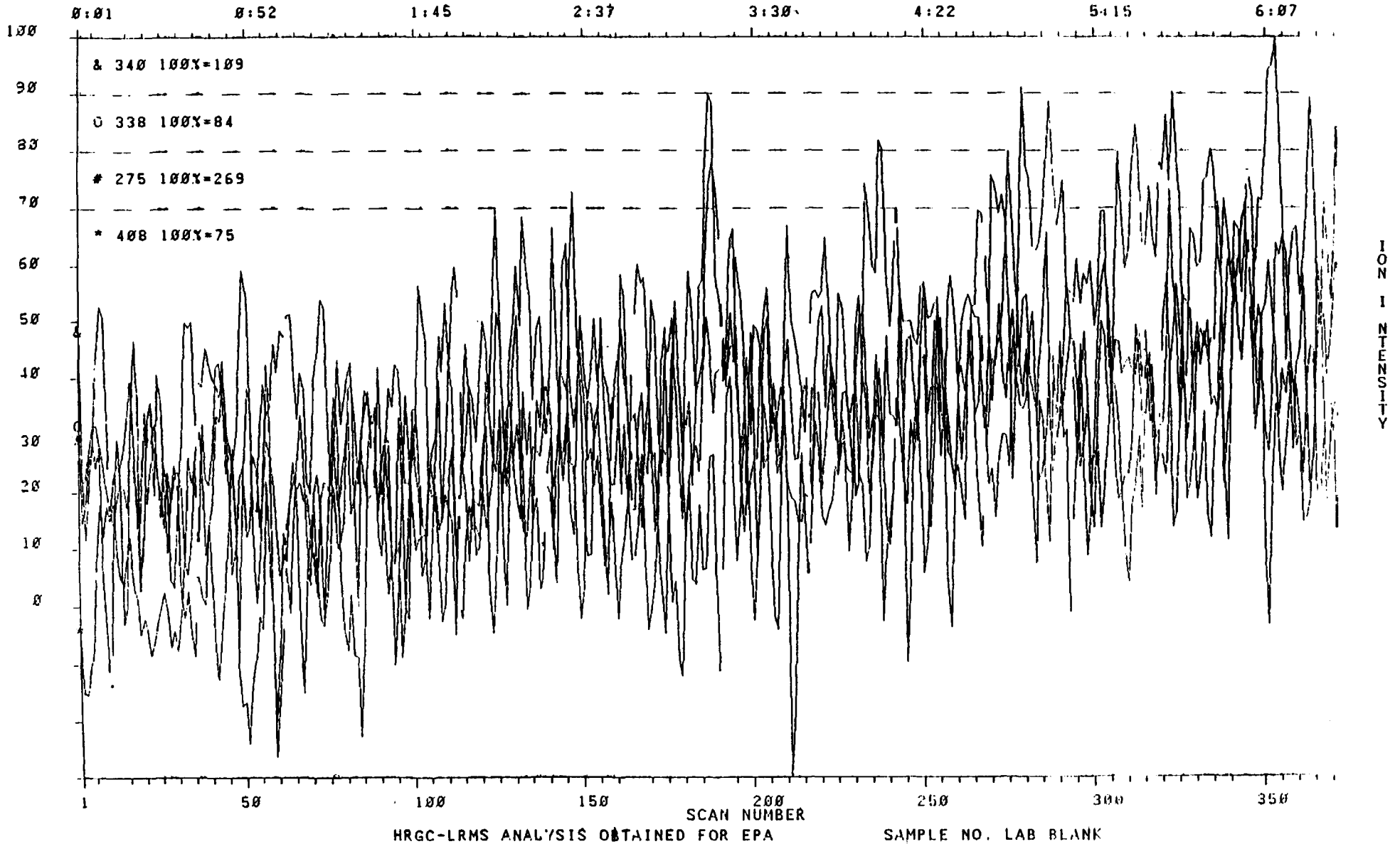
KRATOS MS25, DS55 SOFTWARE, RUN: CTO4Ø1Ø9, WSU NAME: NBC5-ØB-B
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

FIGURE: 89

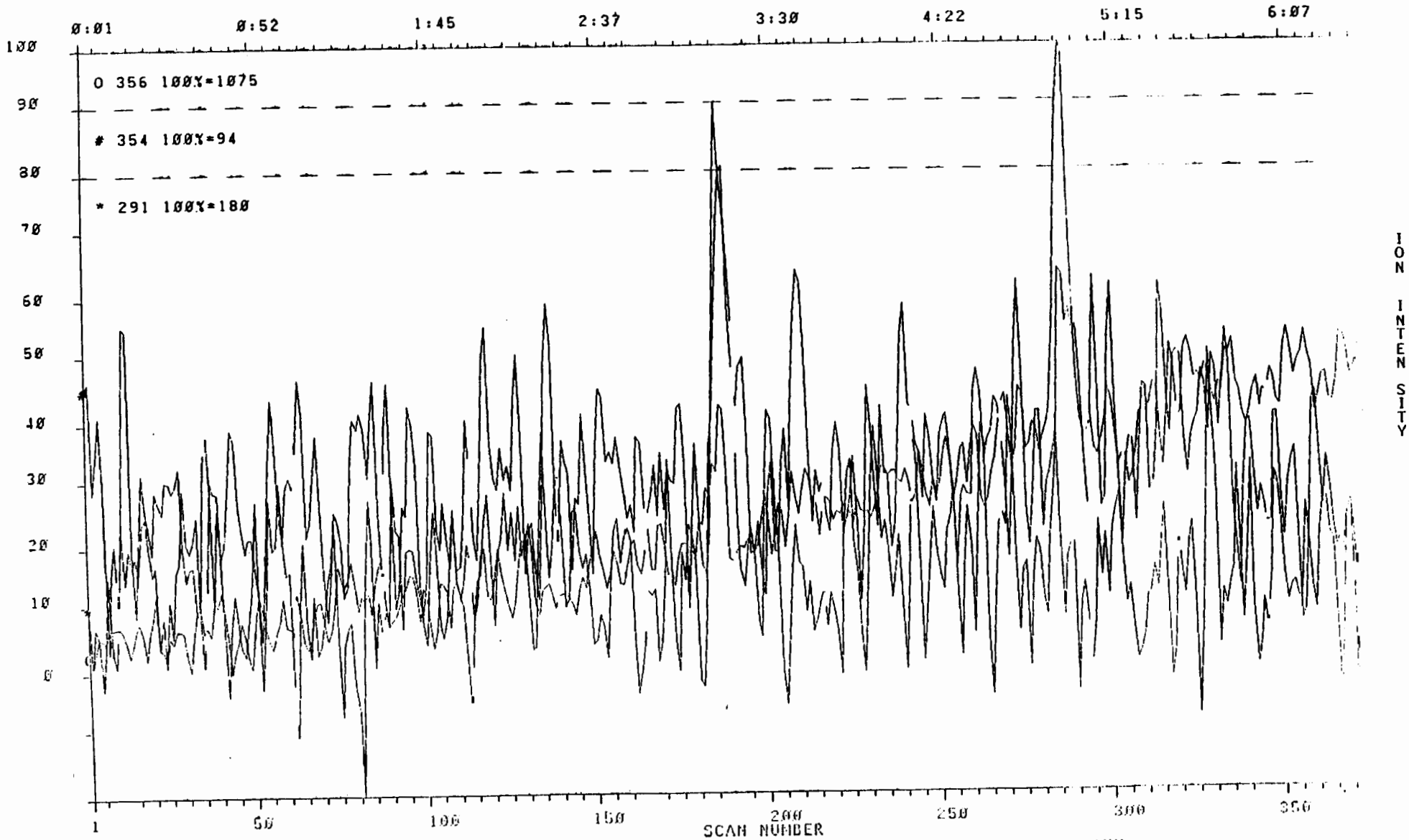
KRATOS MS25, DS55 SOFTWARE, RUN: CTO50109, WSU NAME: NBC5-0B-B
SELECTED-ION MASS CHROMATOGRAMS FOR PENTACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

FIGURE: 90

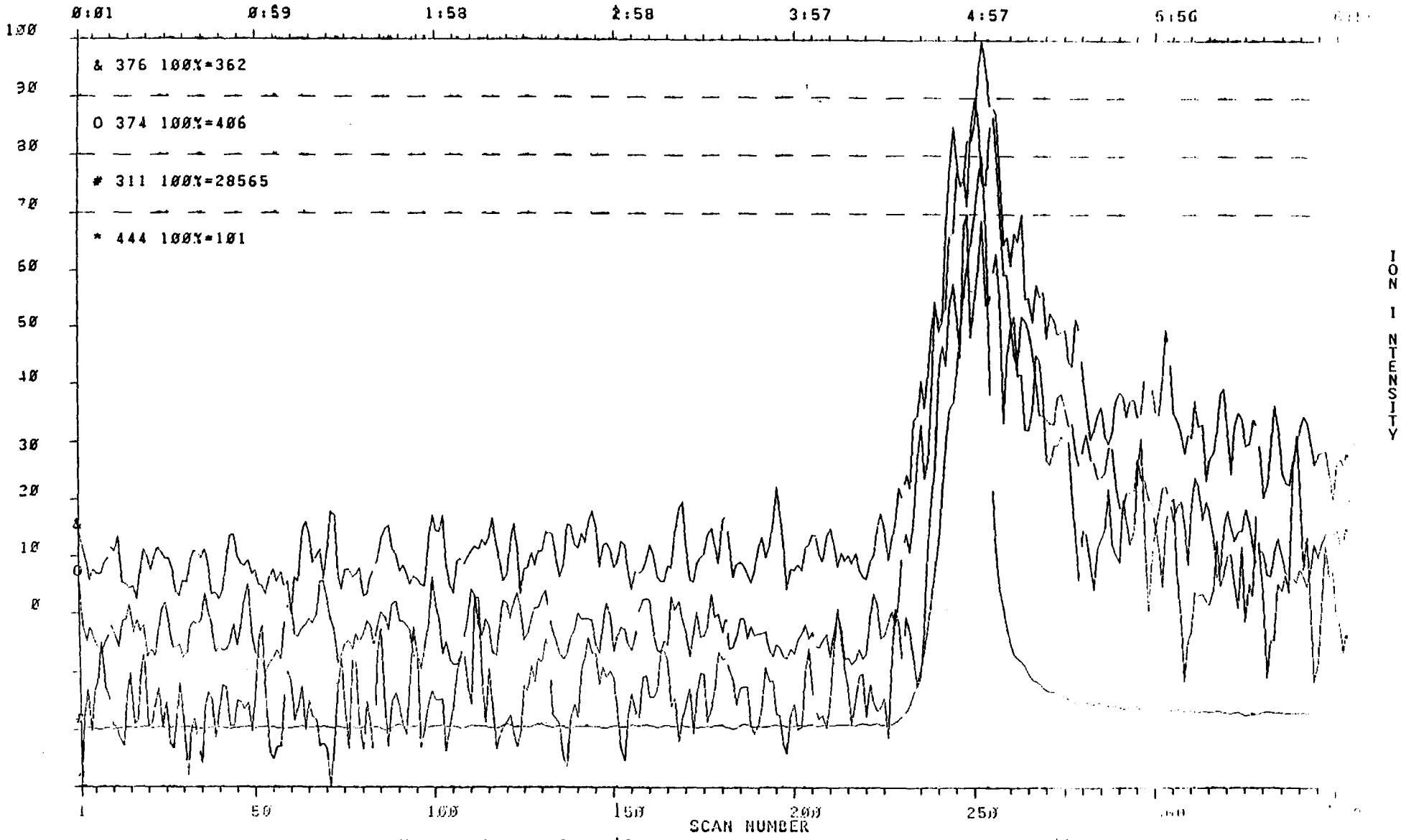
KRATOS MS25, DS55 SOFTWARE, RUN: CTO5Ø1Ø9, WSU NAME: NBC5-ØØ-B
SELECTED-ION MASS CHROMATOGRAMS FOR PENTACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

FIGURE: 91

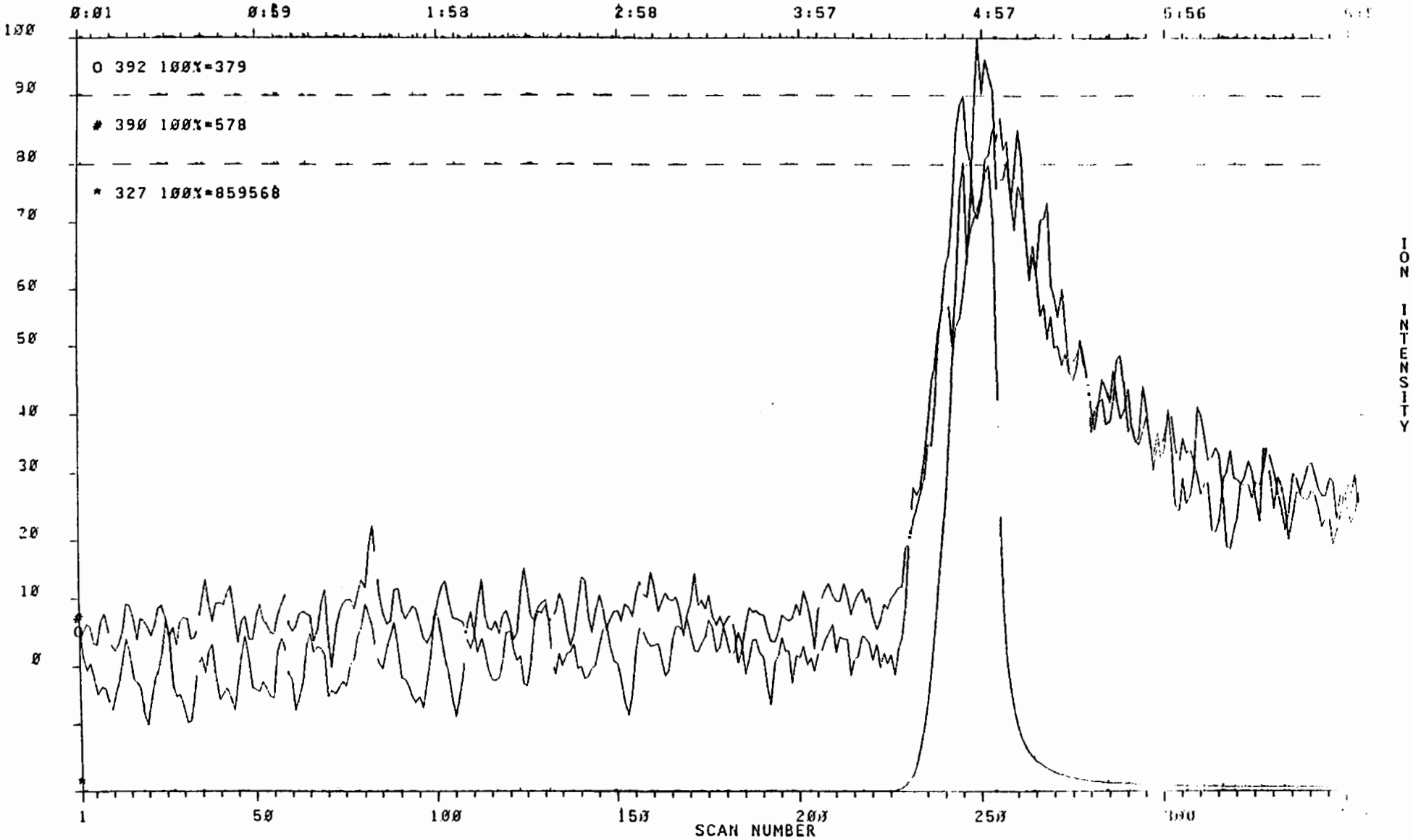
KRATOS MS25, DS55 SOFTWARE, RUN: CTO6Ø1Ø9, WSU NAME: NBC5-ØB-B
SELECTED-ION MASS CHROMATOGRAMS FOR HEXACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

FIGURE: 92

KRATOS MS25, DS55 SOFTWARE, RUN: CTO6Ø1Ø9, WSU NAME: NBC5-ØB-B
SELECTED-ION MASS CHROMATOGRAMS FOR HEXACHLORODIBENZO-P-DIOXINS

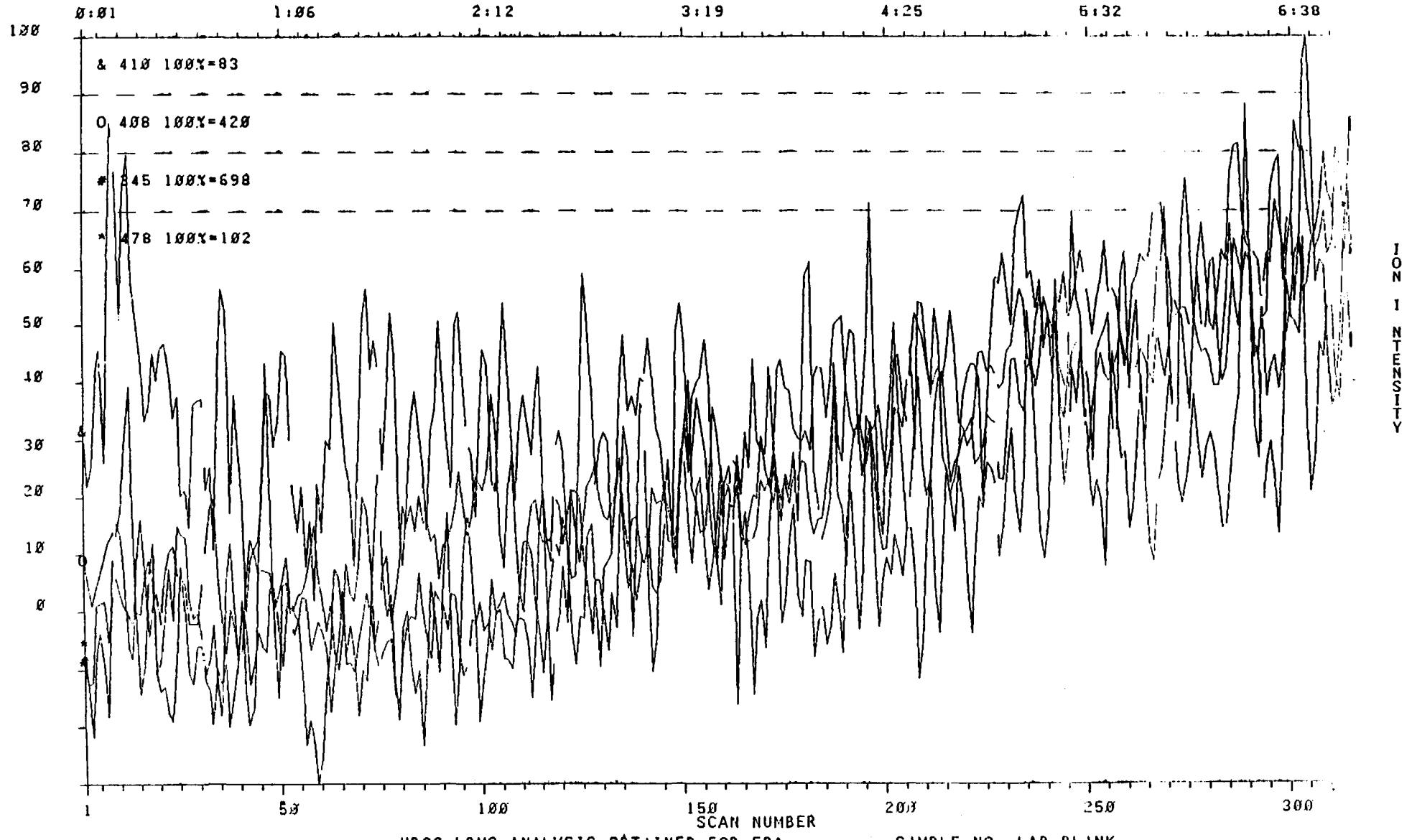


HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

FIGURE: 93

KRATOS MS25, DS55 SOFTWARE, RUN: CTO70109, WSU NAME: NBC5-BB-0

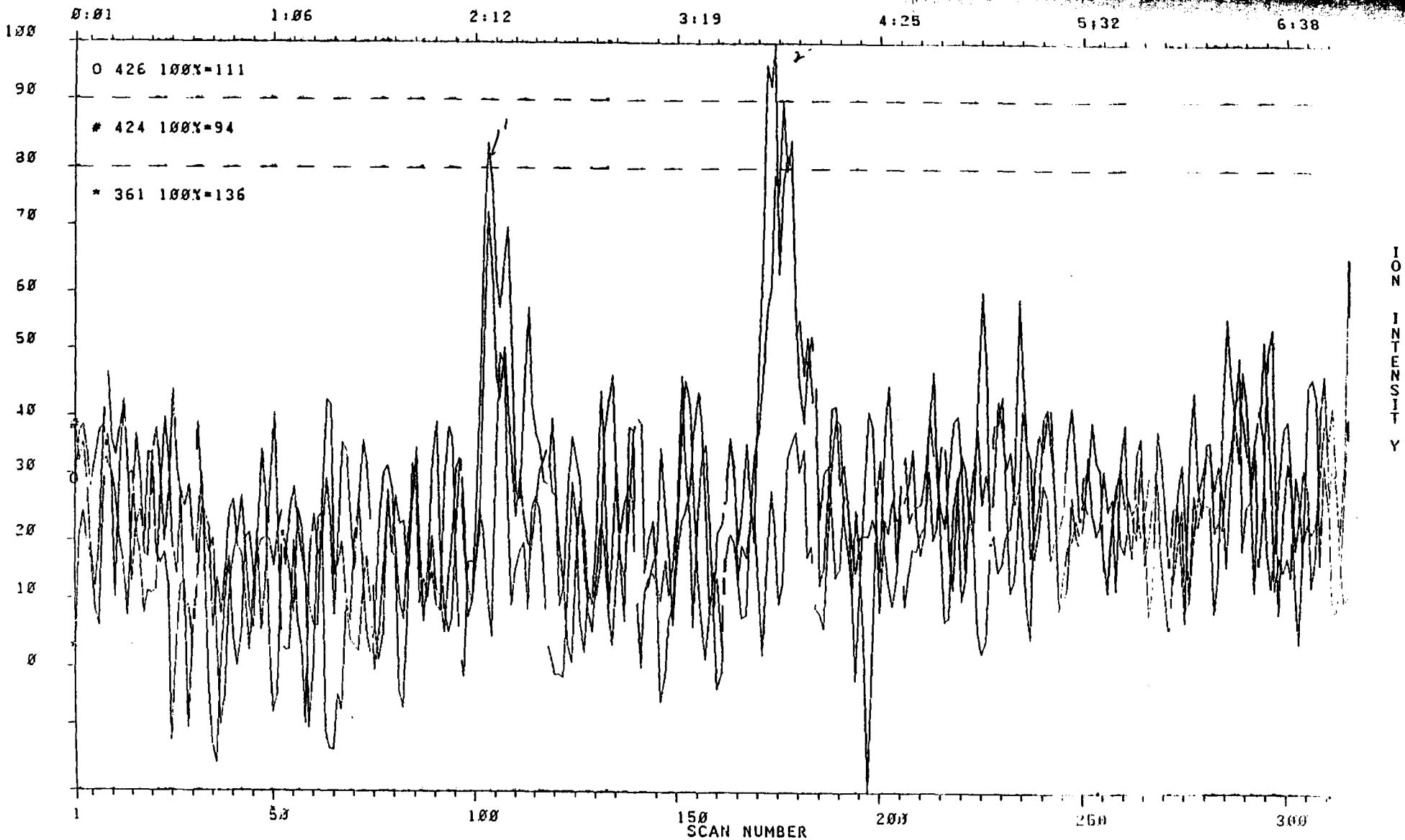
SELECTED-ION MASS CHROMATOGRAMS FOR HEPTACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

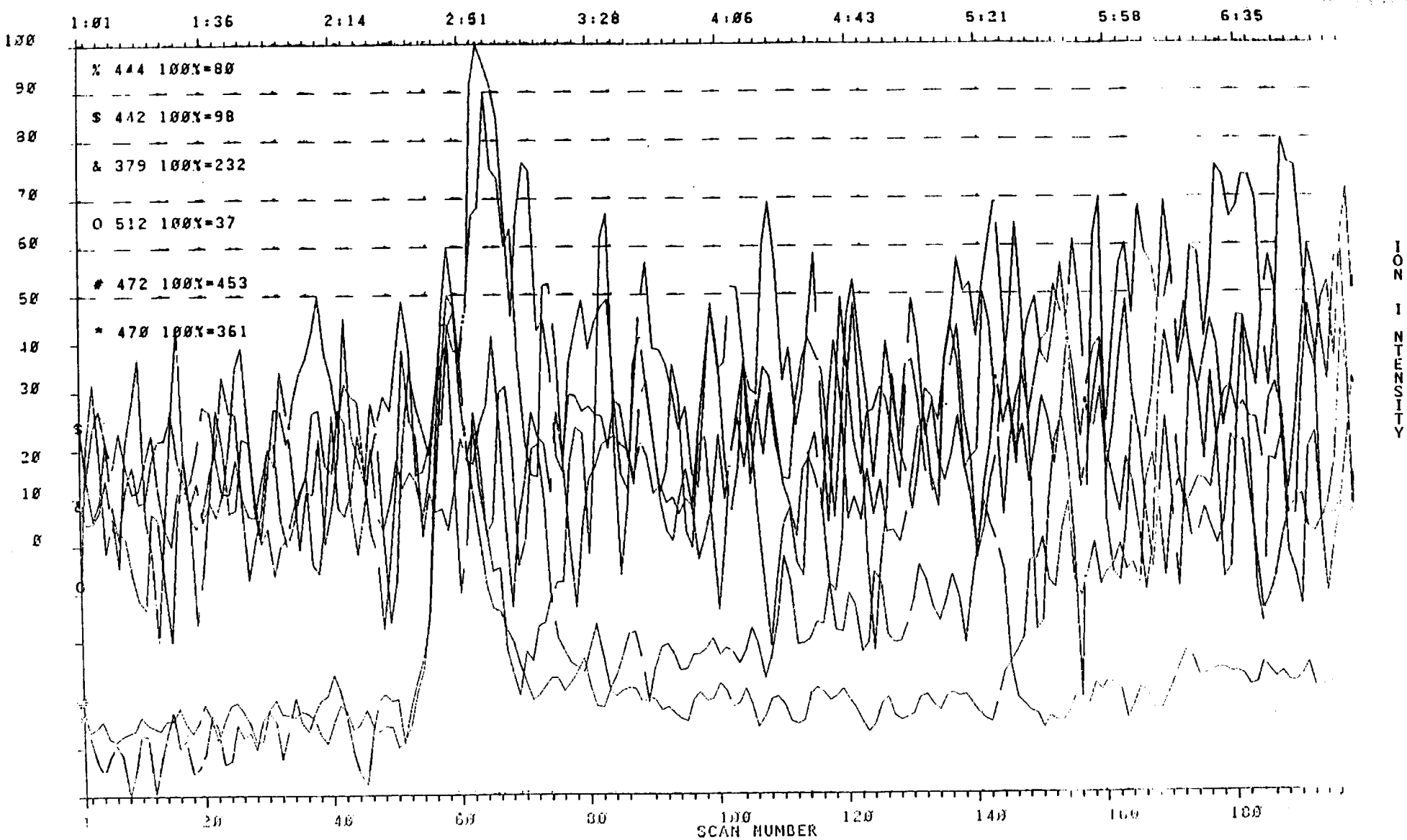
FIGURE: 94

SELECTED-ION MASS CHROMATOGRAMS FOR HEPTACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

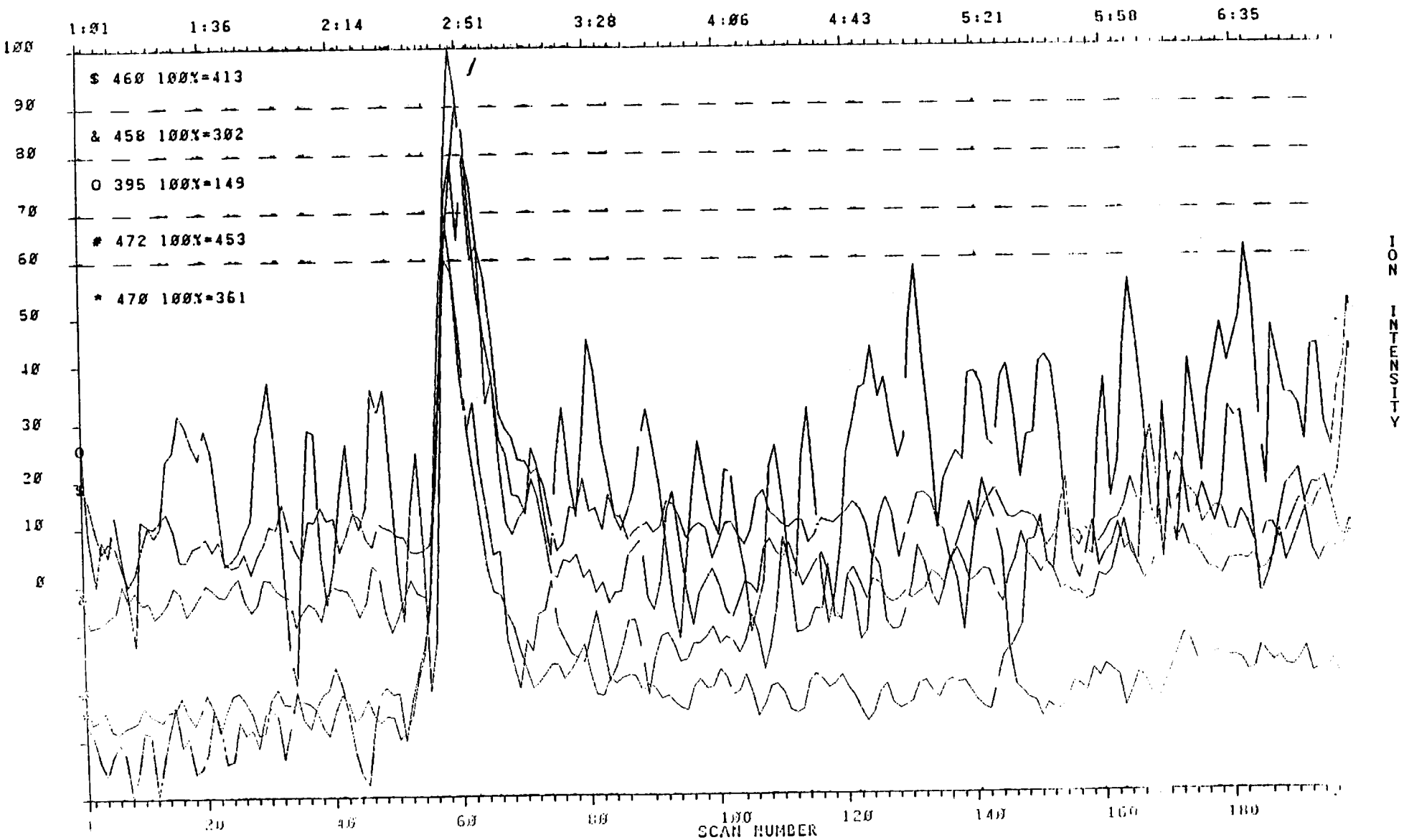
FIGURE: 95



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

FIGURE: 96

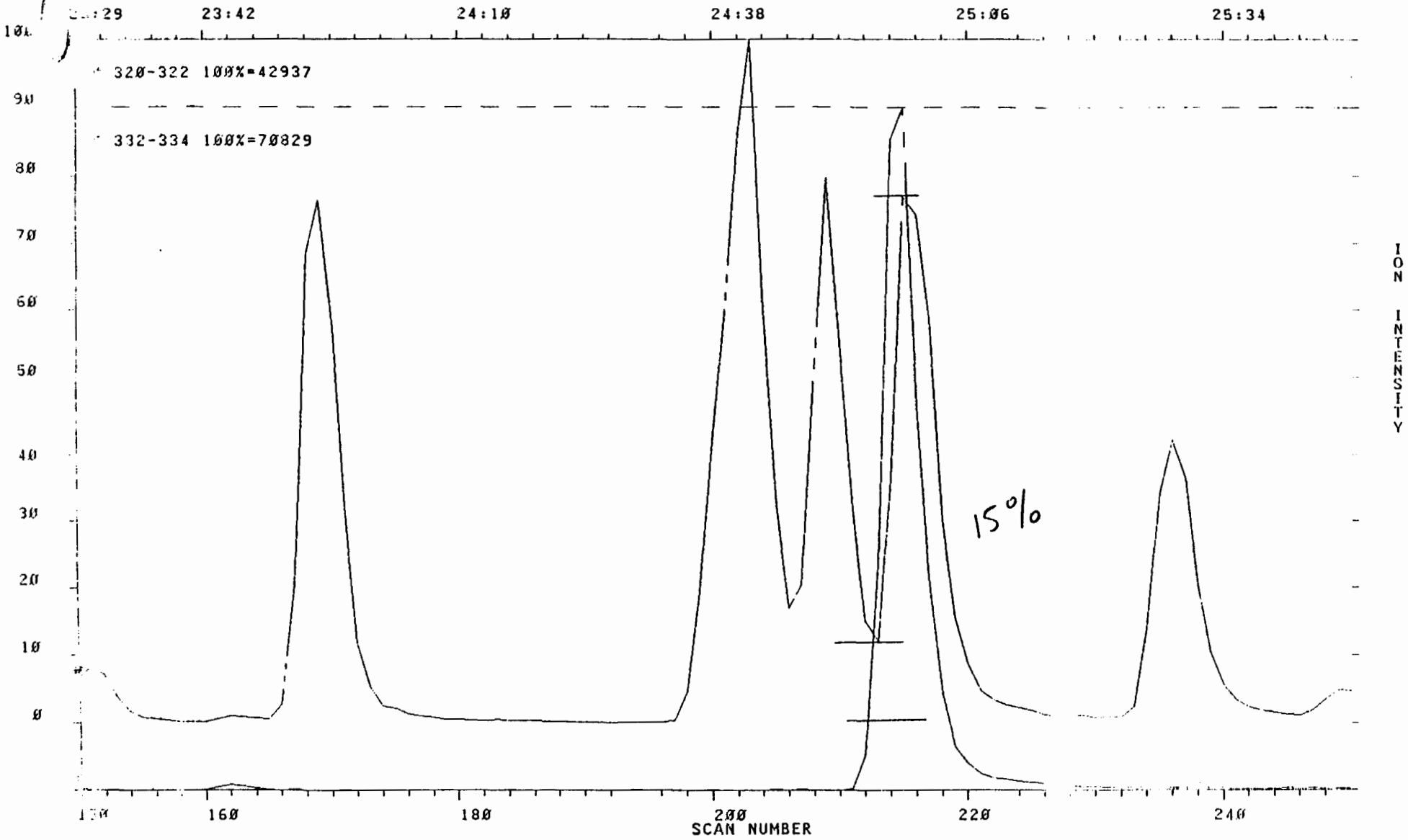
KRATOS MS25, DS55 SOFTWARE, RUN: CTO80109, WSU NAME: NBC5-00-B
SELECTED-ION MASS CHROMATOGRAMS FOR OCTACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

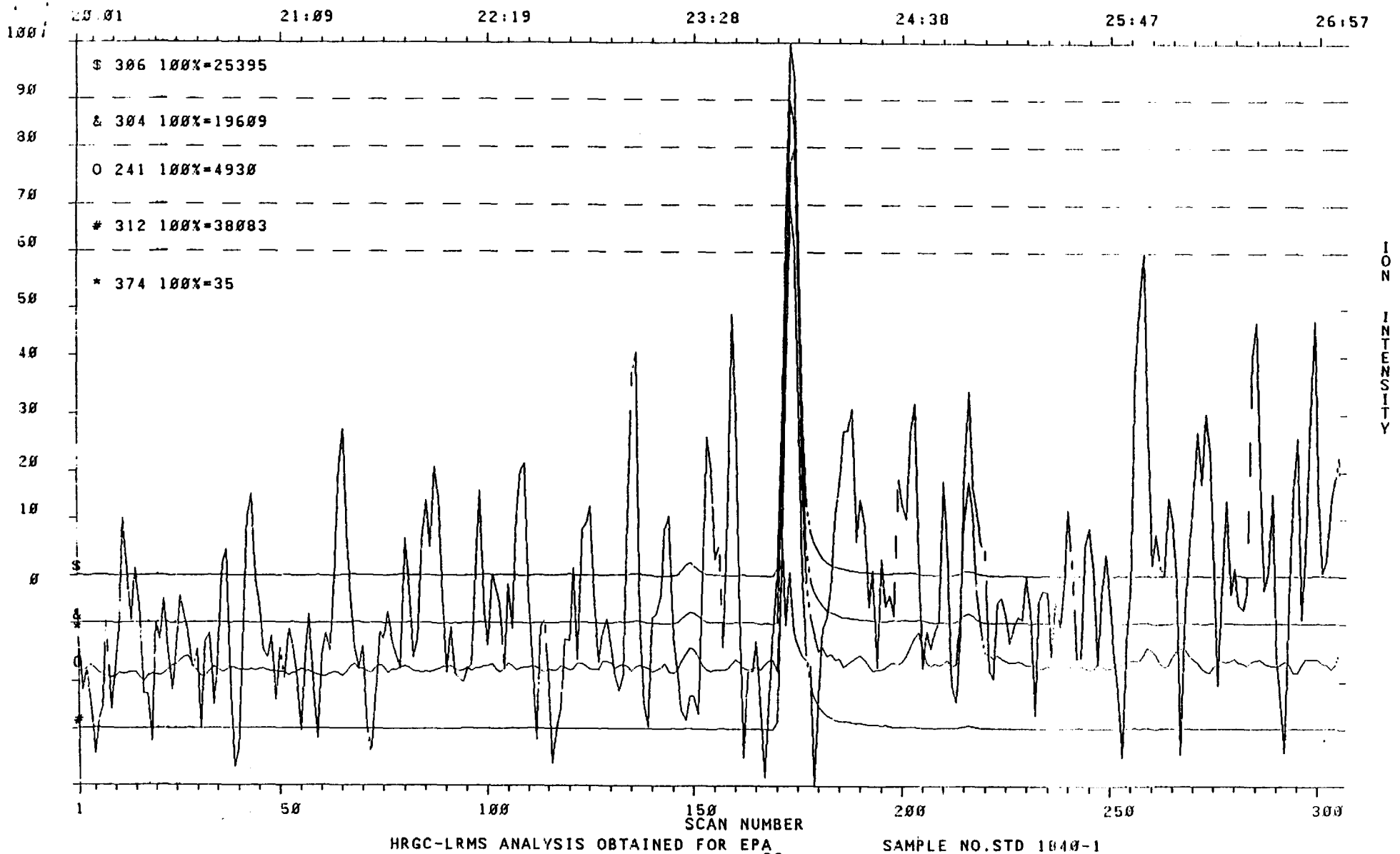
FIGURE: 97

KRATOS MS25, DS55 SOFTWARE, RUN: FGH40101, WSU NAME: PEMIX#2
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZO-P-DIO INS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.PEMIX 2
FIGURE: 98

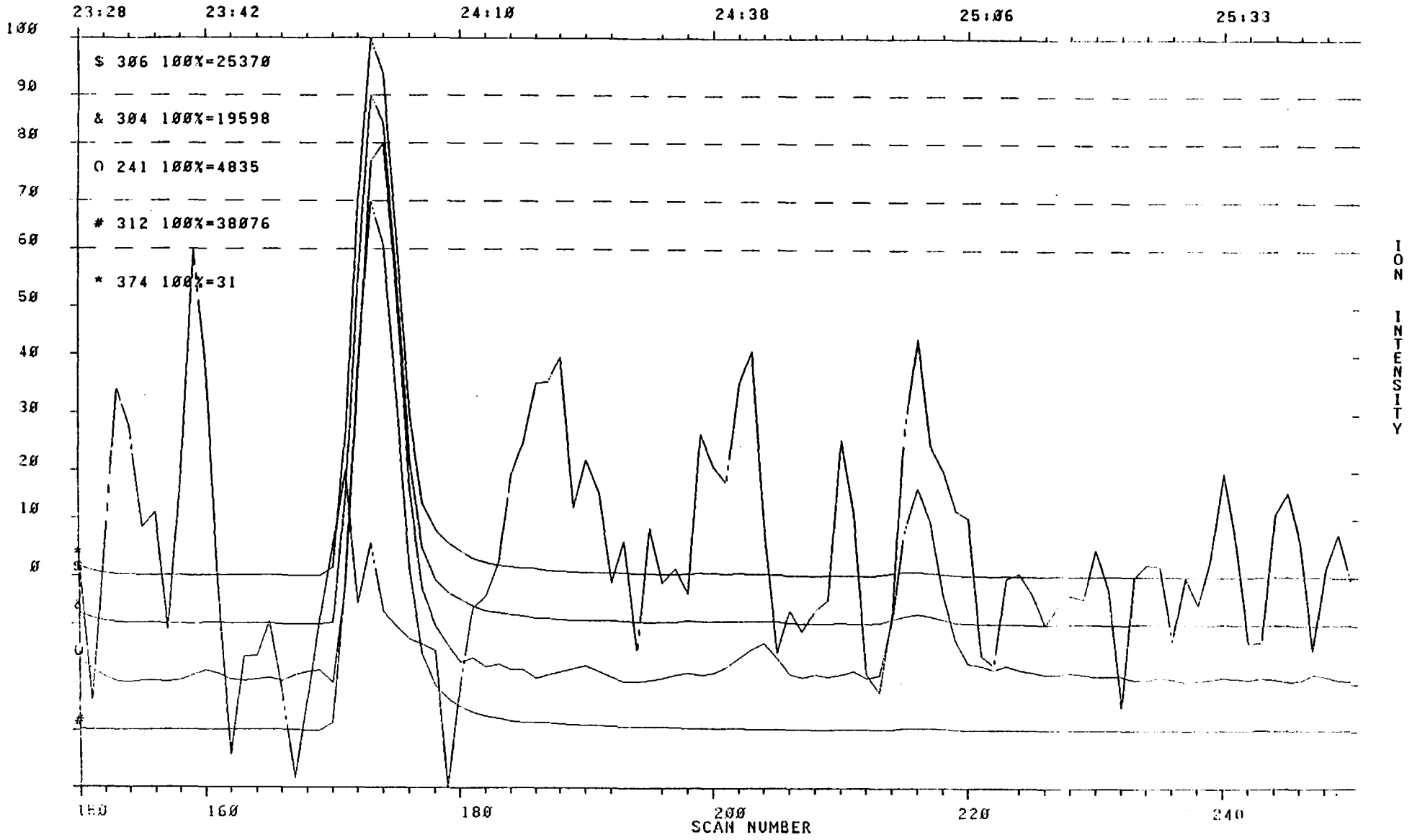
KRATOS MS25, DS55 SOFTWARE, RUN: CTO4ØØ79, WSU NAME: STD 1840-1
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.STD 1840-1

FIGURE: 99

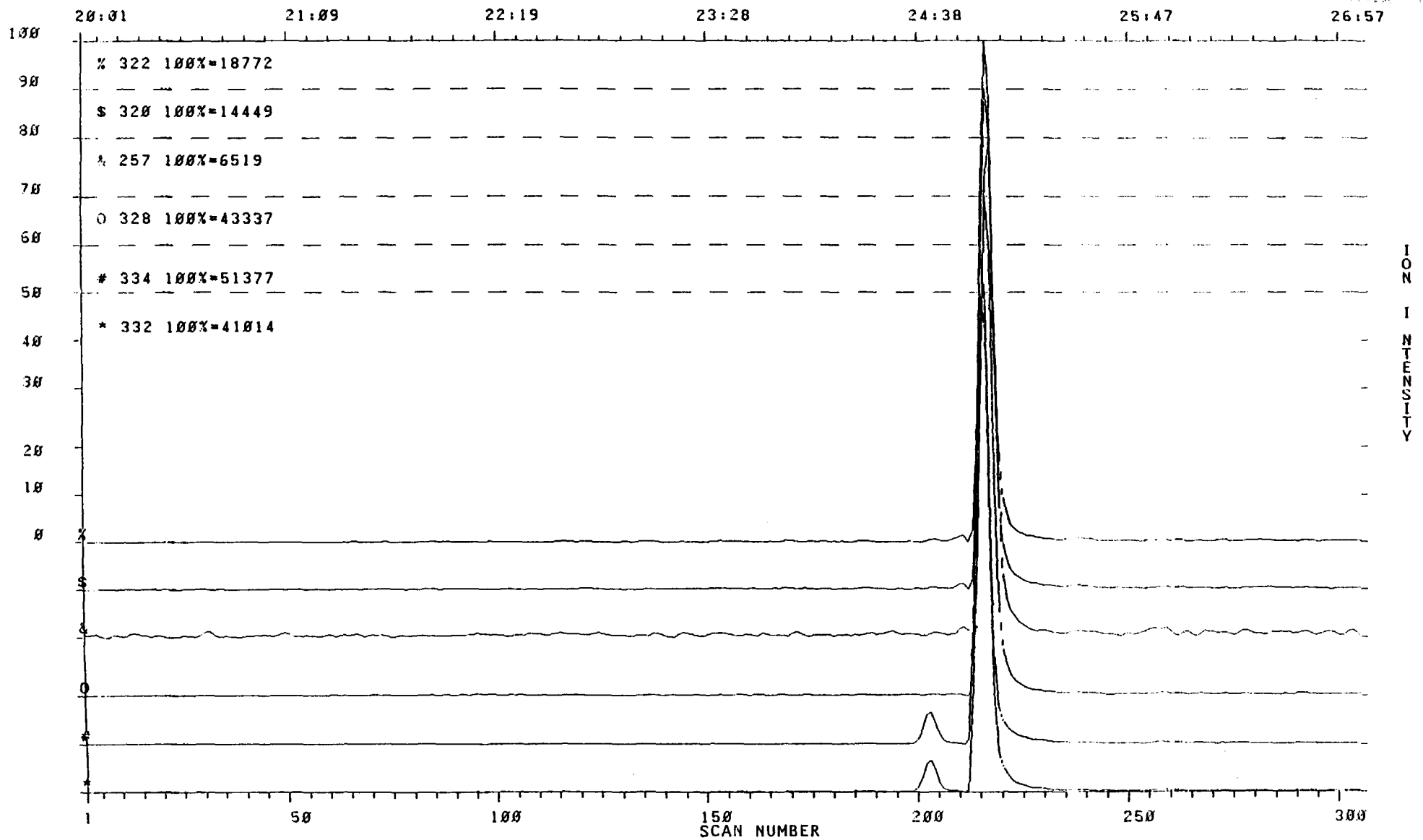
KRATOS MS25, DS55 SOFTWARE, RUN: CTO4ØØ79, WSU NAME: STD 184Ø-1
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.STD 184Ø-1

FIGURE: 100

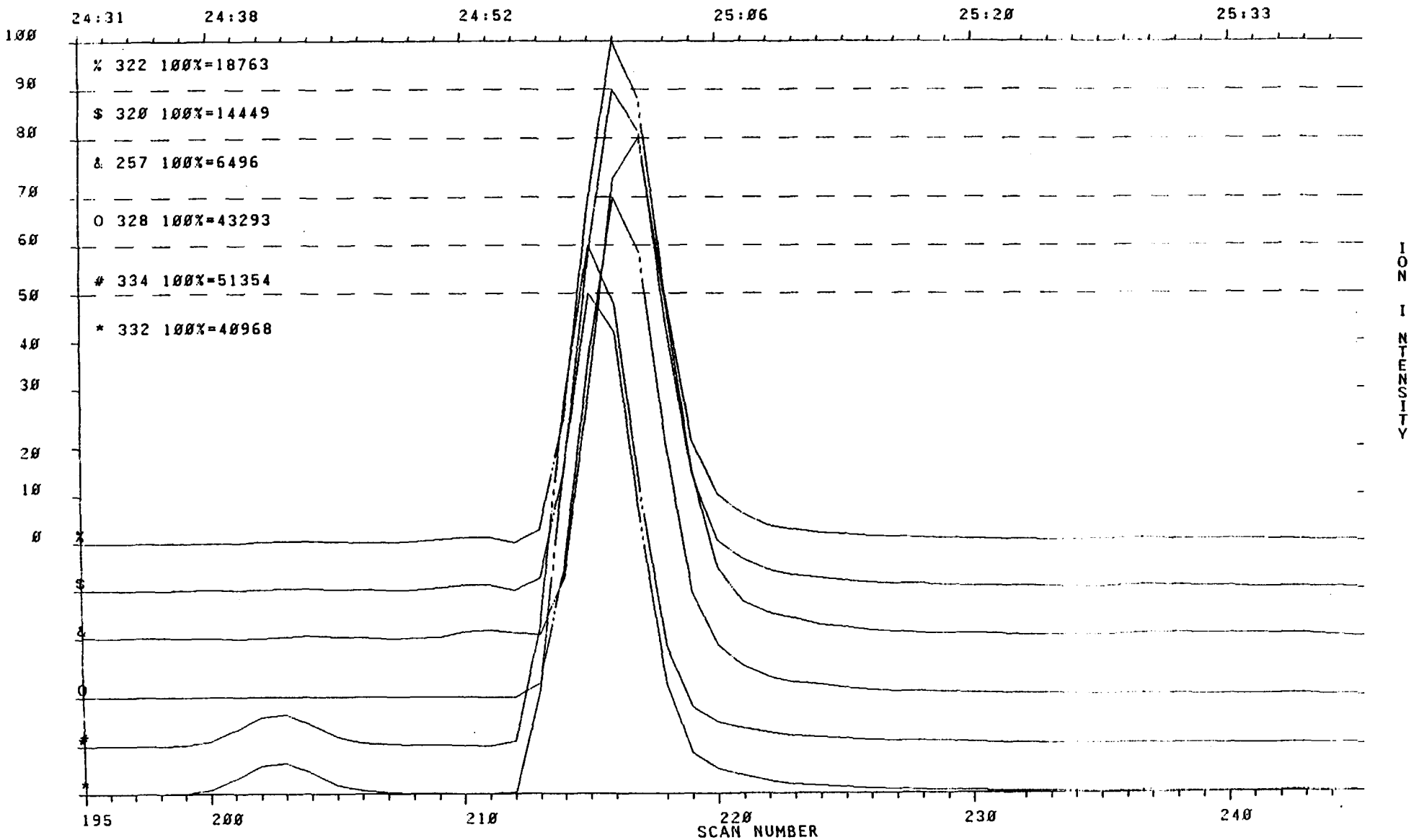
KRATOS MS25, DS55 SOFTWARE, RUN: CTO40079, WSU NAME: STD 1840-1
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.STD 1840-1

FIGURE: 101

KRATOS MS25, DS55 SOFTWARE, RUN: CTO4ØØ79, WSU NAME: STD 1840-1
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZO-P-DIOXINS

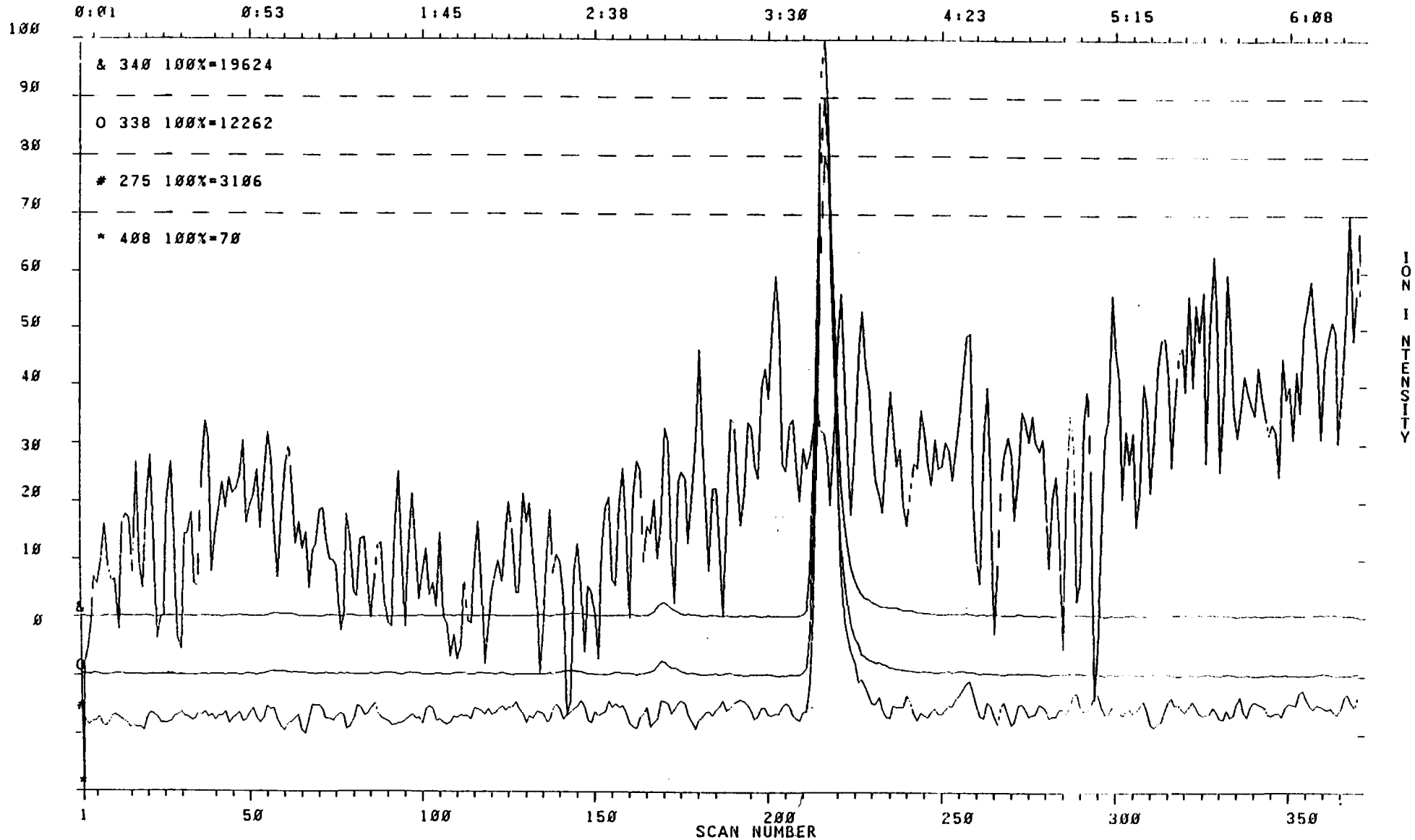


HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.STD 1840-1

FIGURE: 102

KRATOS MS25, DS55 SOFTWARE, RUN: CTO50079, WSU NAME: STD 18:0-1

SELECTED-ION MASS CHROMATOGRAMS FOR PENTACHLORODIBENZOFURANS

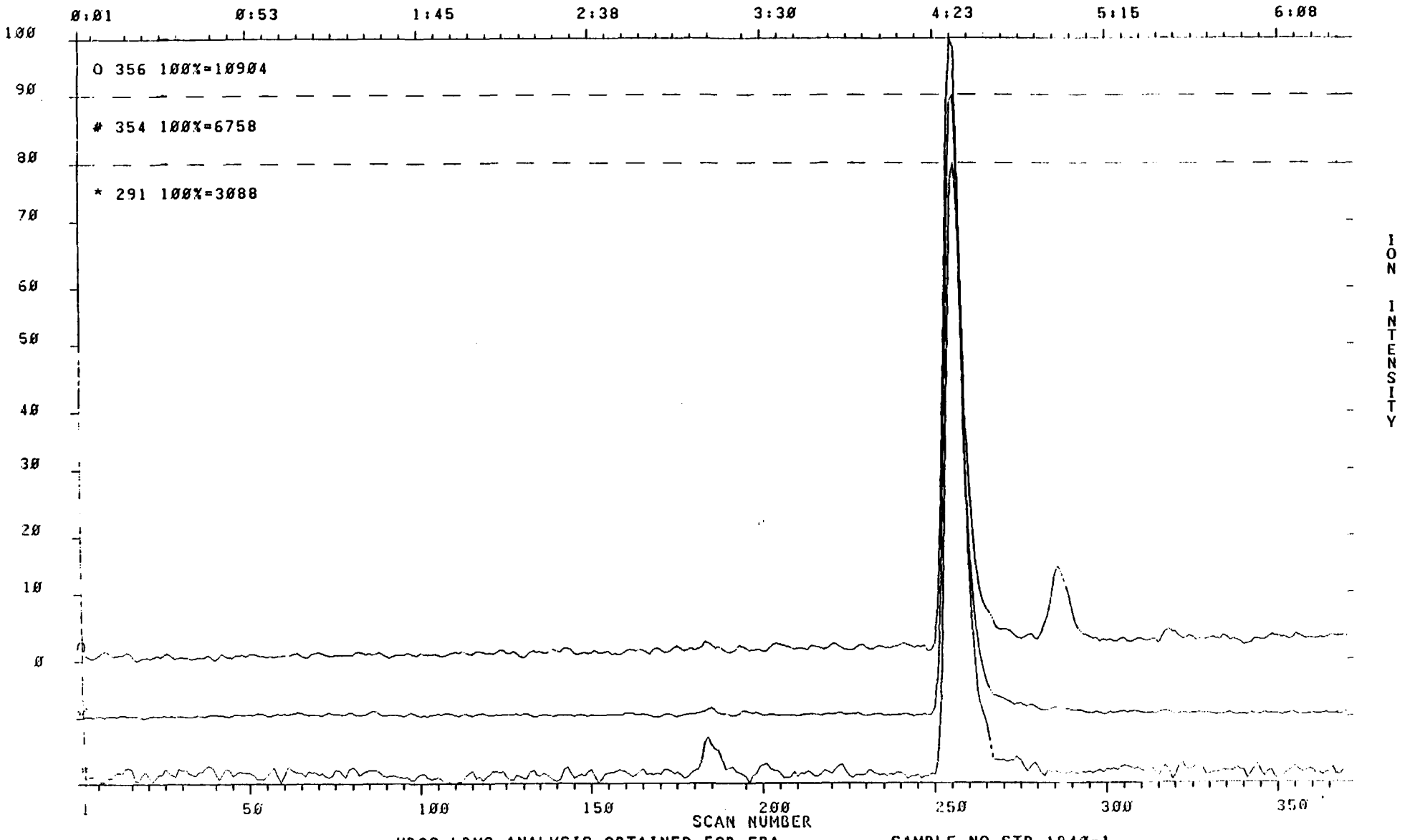


HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.STD 1040-1

FIGURE: 103

KRATOS MS25, DS55 SOFTWARE, RUN: CT050079, WSO NAME: STD 1840-1

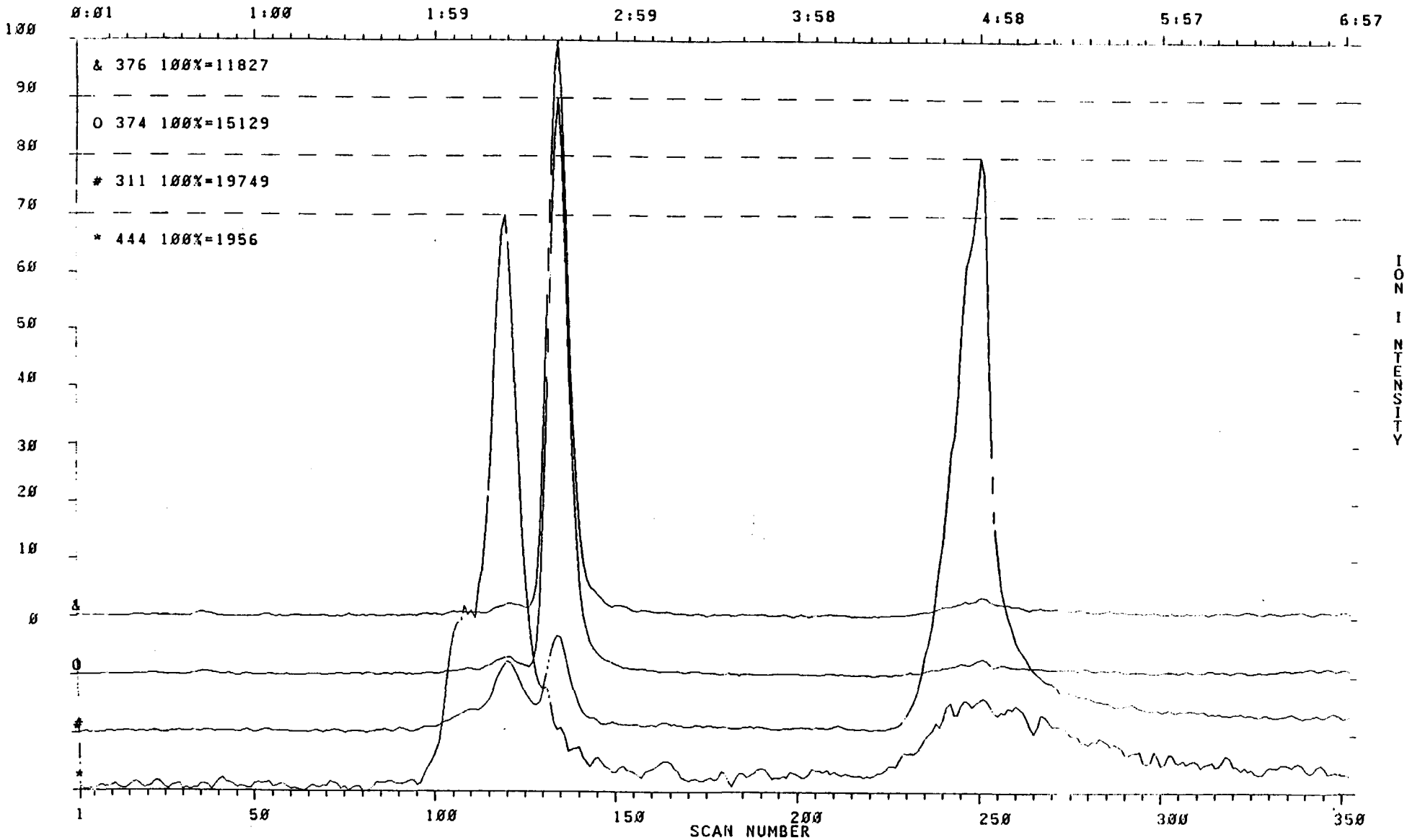
SELECTED-ION MASS CHROMATOGRAMS FOR PENTACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.STD 1840-1

FIGURE: 104

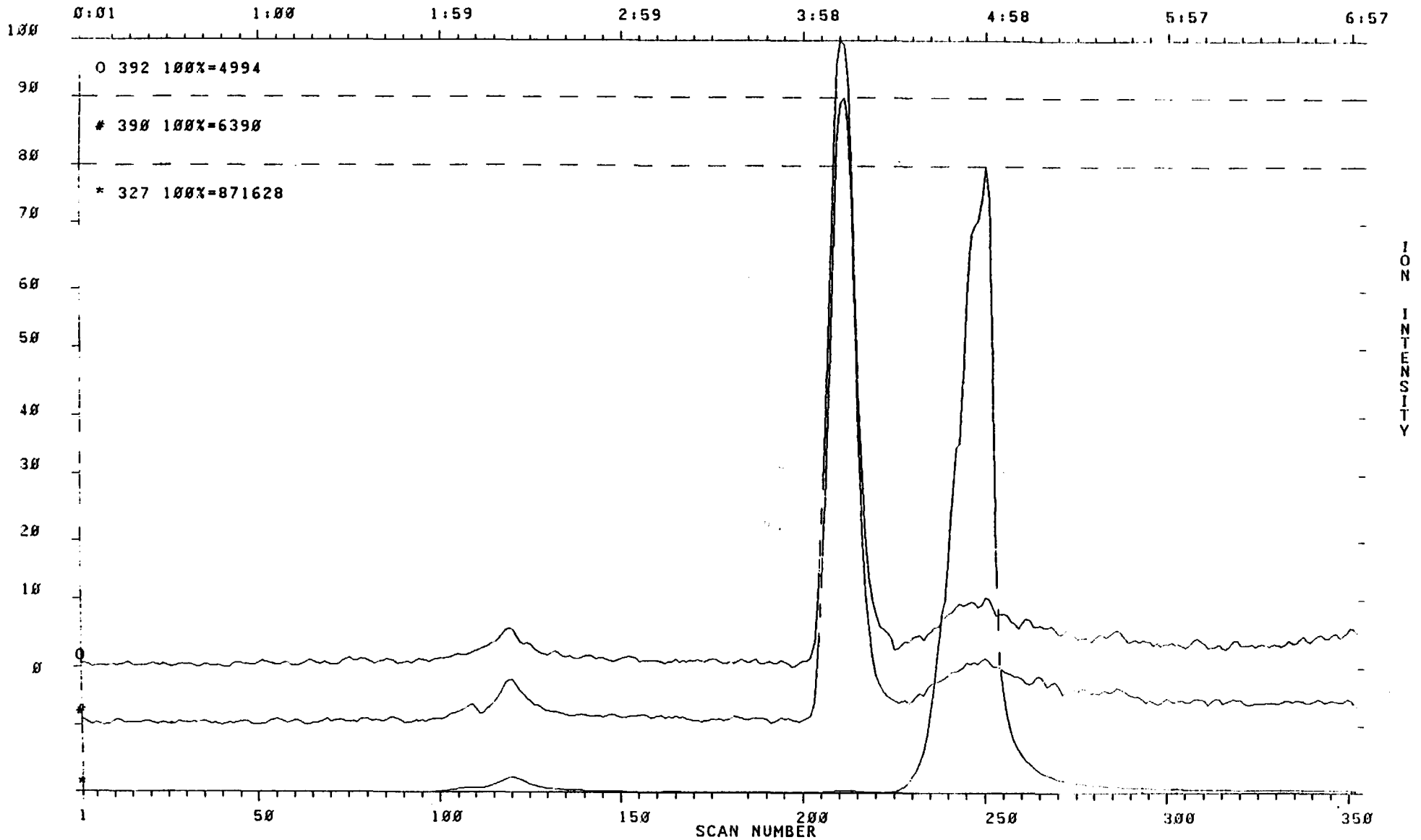
KRATOS MS25, DS55 SOFTWARE, RUN: CTO6ØØ79, WSU NAME: STD 184Ø-1
SELECTED-ION MASS CHROMATOGRAMS FOR HEXACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.STD 184Ø-1

FIGURE: 105

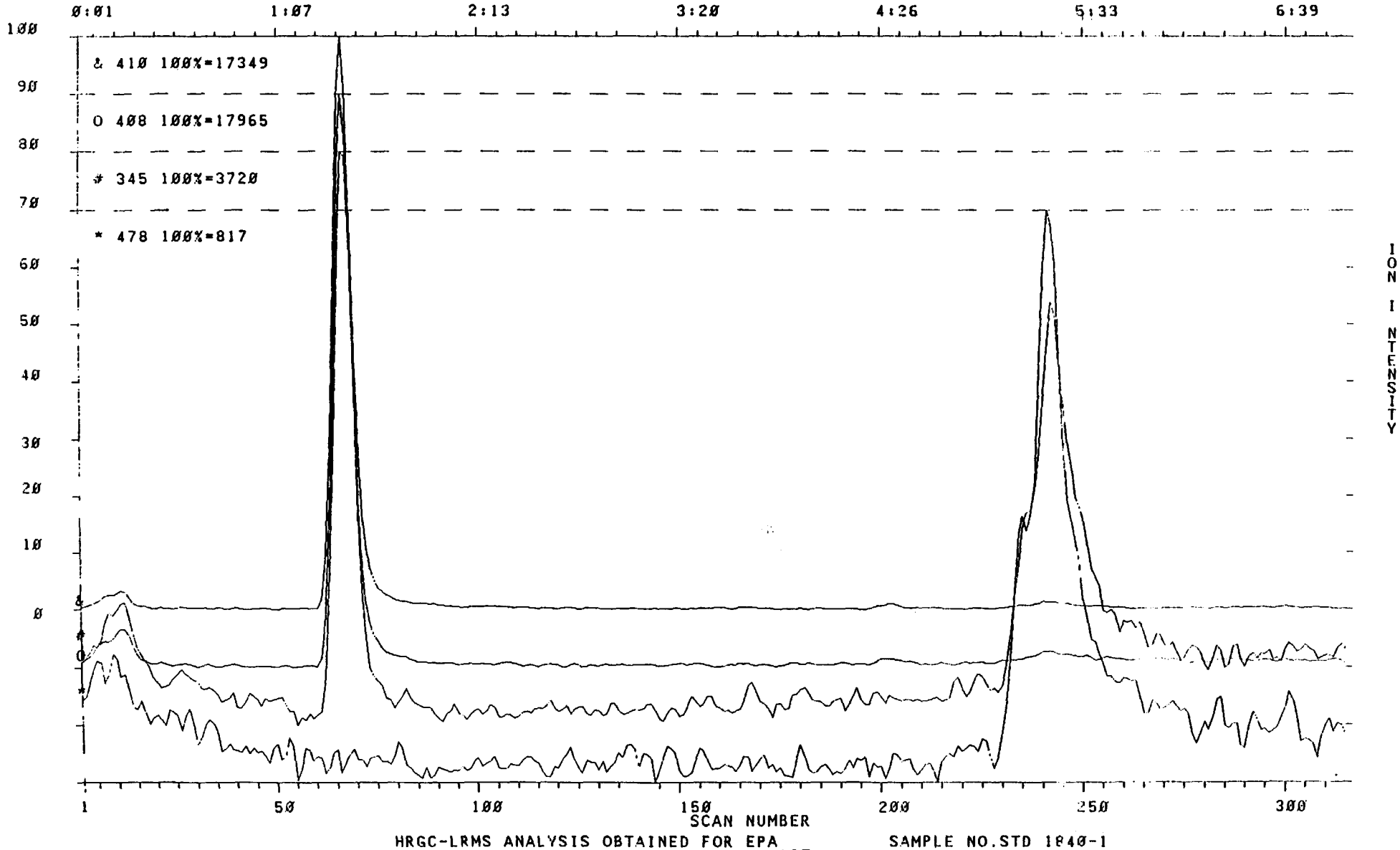
KRATOS MS25, DS55 SOFTWARE, RUN: CTO6ØØ79, WSU NAME: STD 184Ø-1
SELECTED-ION MASS CHROMATOGRAMS FOR HEXACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.STD 184Ø-1

FIGURE: 106

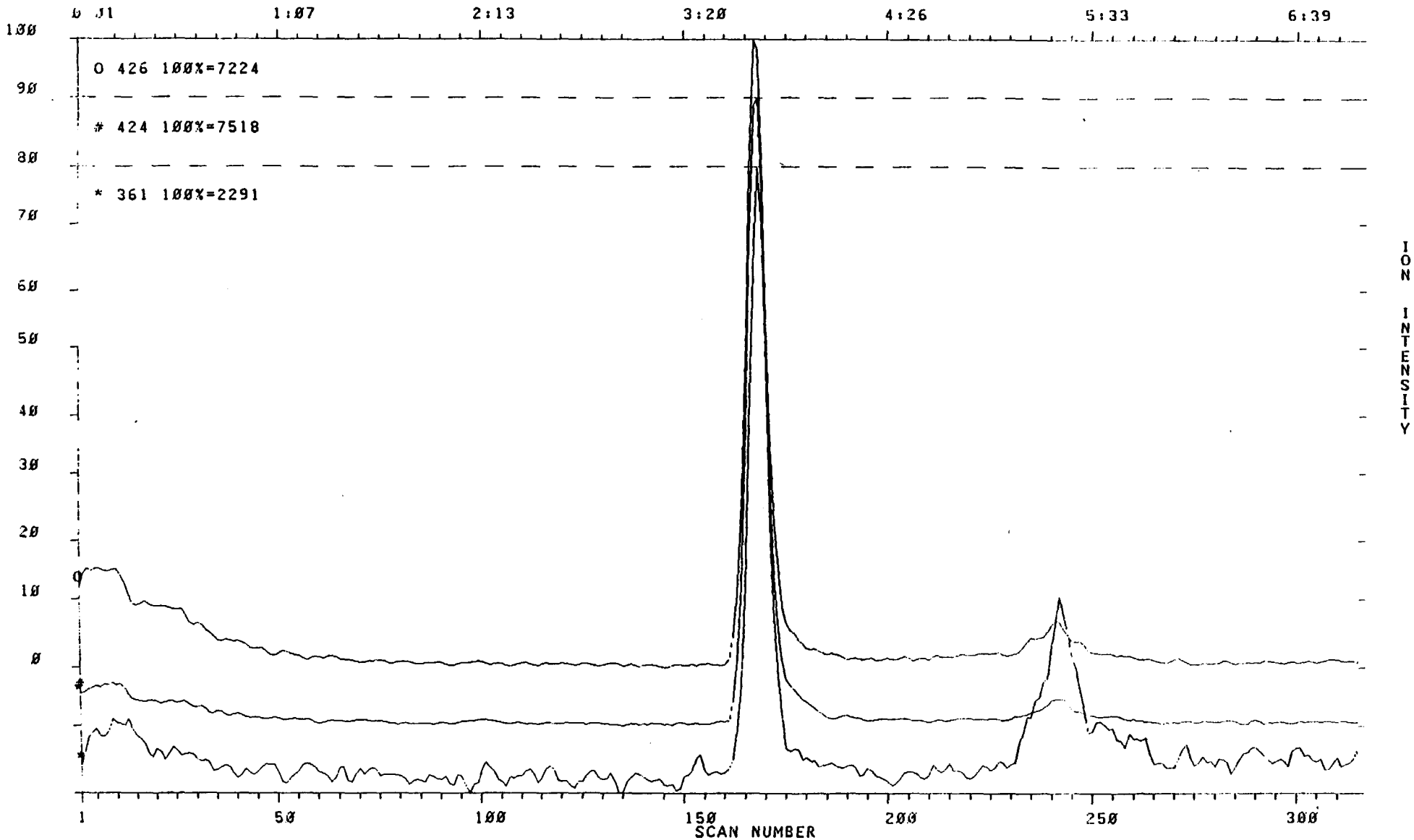
KRATOS MS25, DS55 SOFTWARE, RUN: CTO70079, WSU NAME: STD 1840-1
SELECTED-ION MASS CHROMATOGRAMS FOR HEPTACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.STD 1840-1

FIGURE: 107

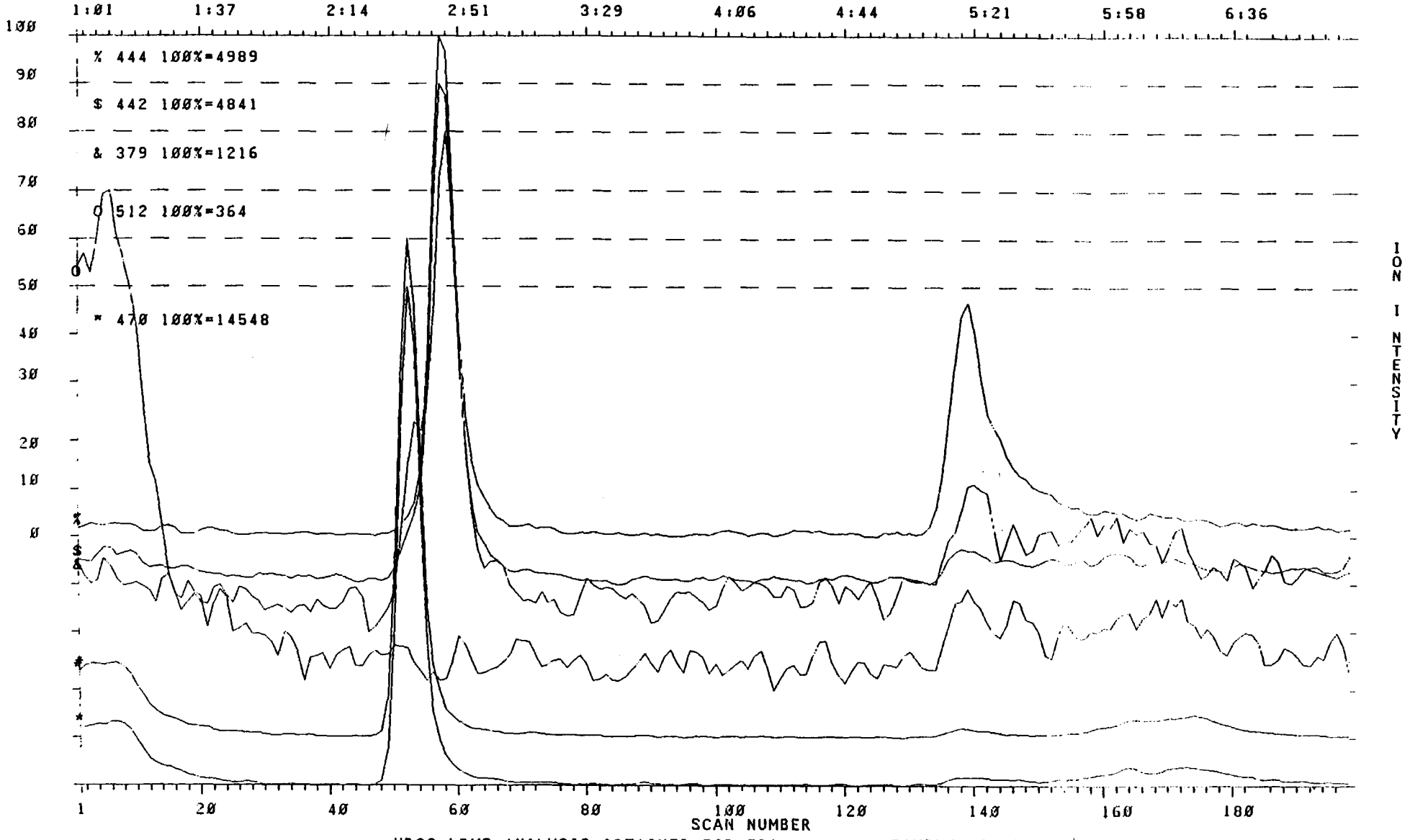
SELECTED-ION MASS CHROMATOGRAMS FOR HEPTACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.STD 1840-1

FIGURE: 108

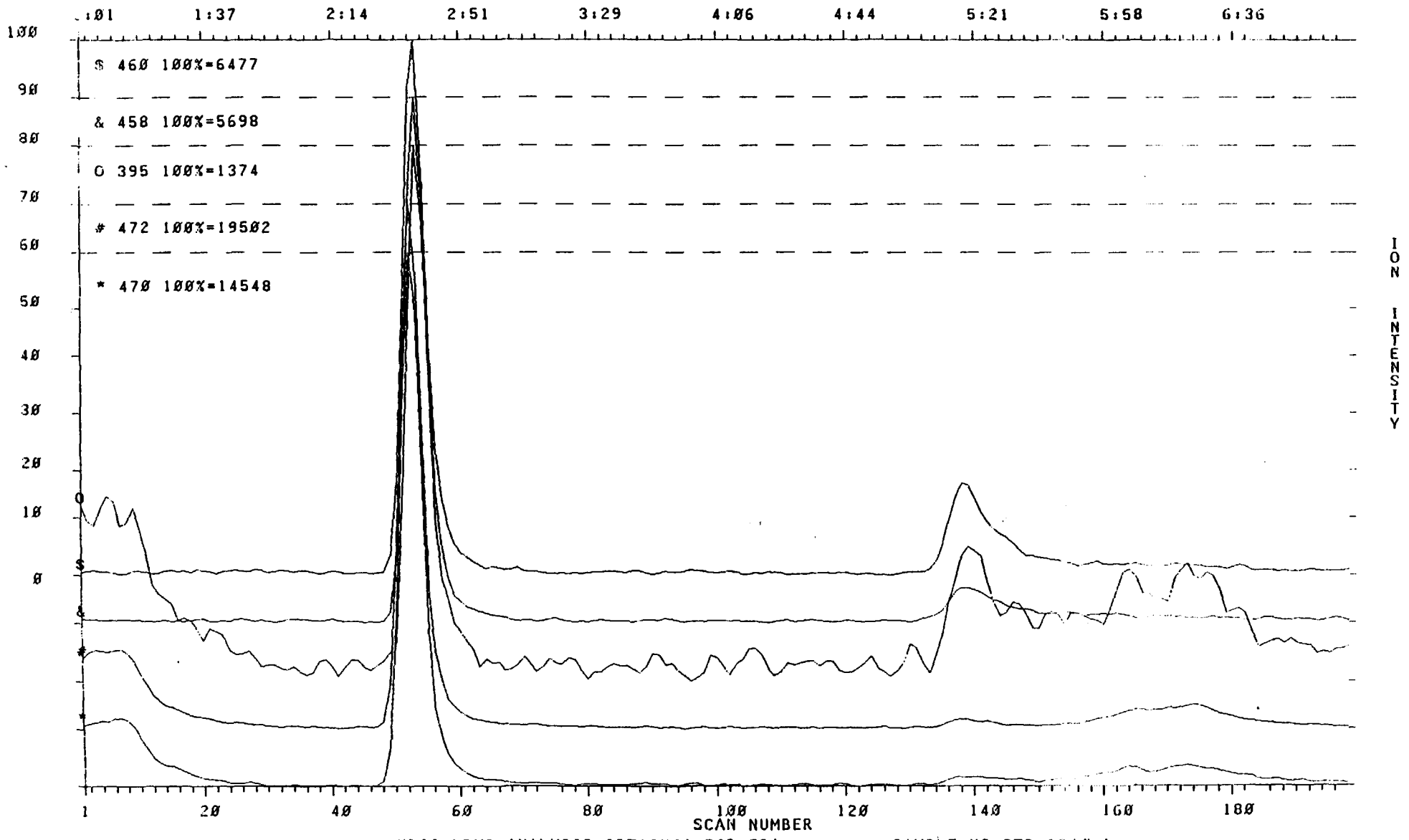
KRATOS MS25, DS55 SOFTWARE, RUN: CTO8ØØ79, WSU NAME: STD 1840-1
SELECTED-ION MASS CHROMATOGRAMS FOR OCTACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.STD 1840-1

FIGURE: 109

KRATOS MS25, DS55 SOFTWARE, RUN: CT080079, WSU NAME: STD 1840-1
SELECTED-ION MASS CHROMATOGRAMS FOR OCTACHLORODIBENZO-P-DIOXINS

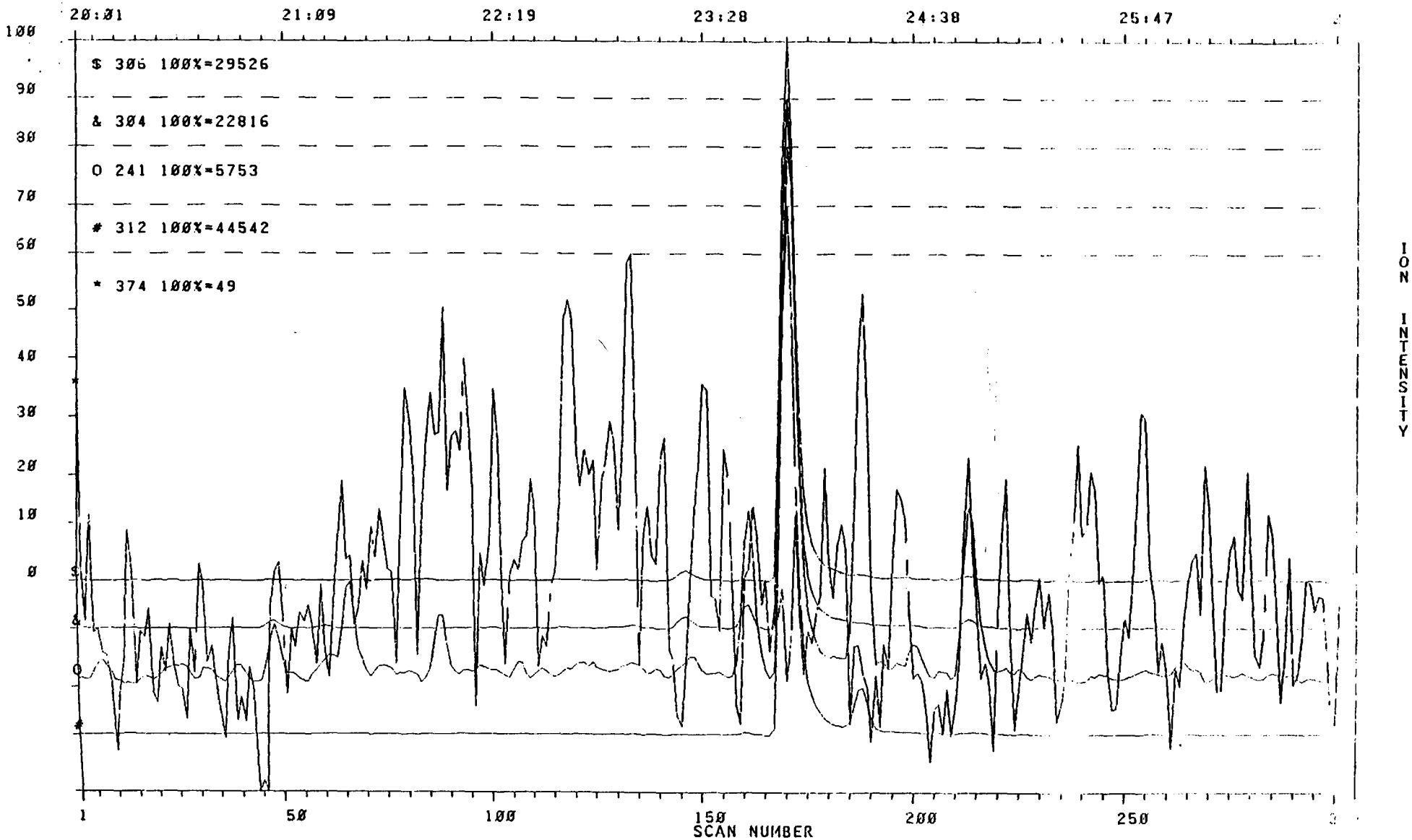


HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.STD 1840-1
FIGURE: 110

DATE: 05/05/86 TIME: 09:51

KRATOS MS25, DS55 SOFTWARE, RUN: CTO40095, WSU NAME: STD 18 0-1

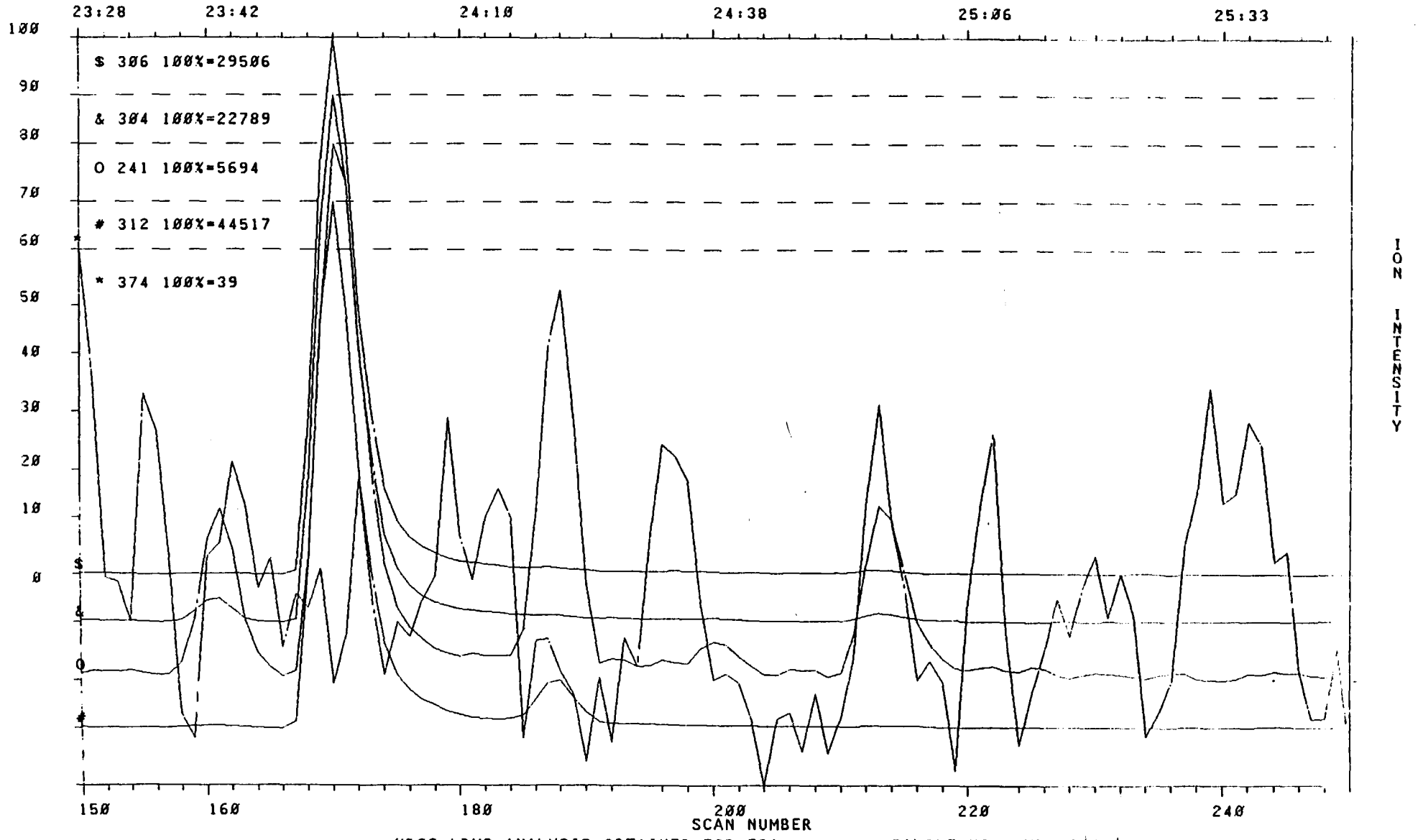
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. STD 1840-1

FIGURE: 111

KRATOS MS25, DS55 SOFTWARE, RUN: CTO40095, WSU NAME: STD 1840-1
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZOFURANS



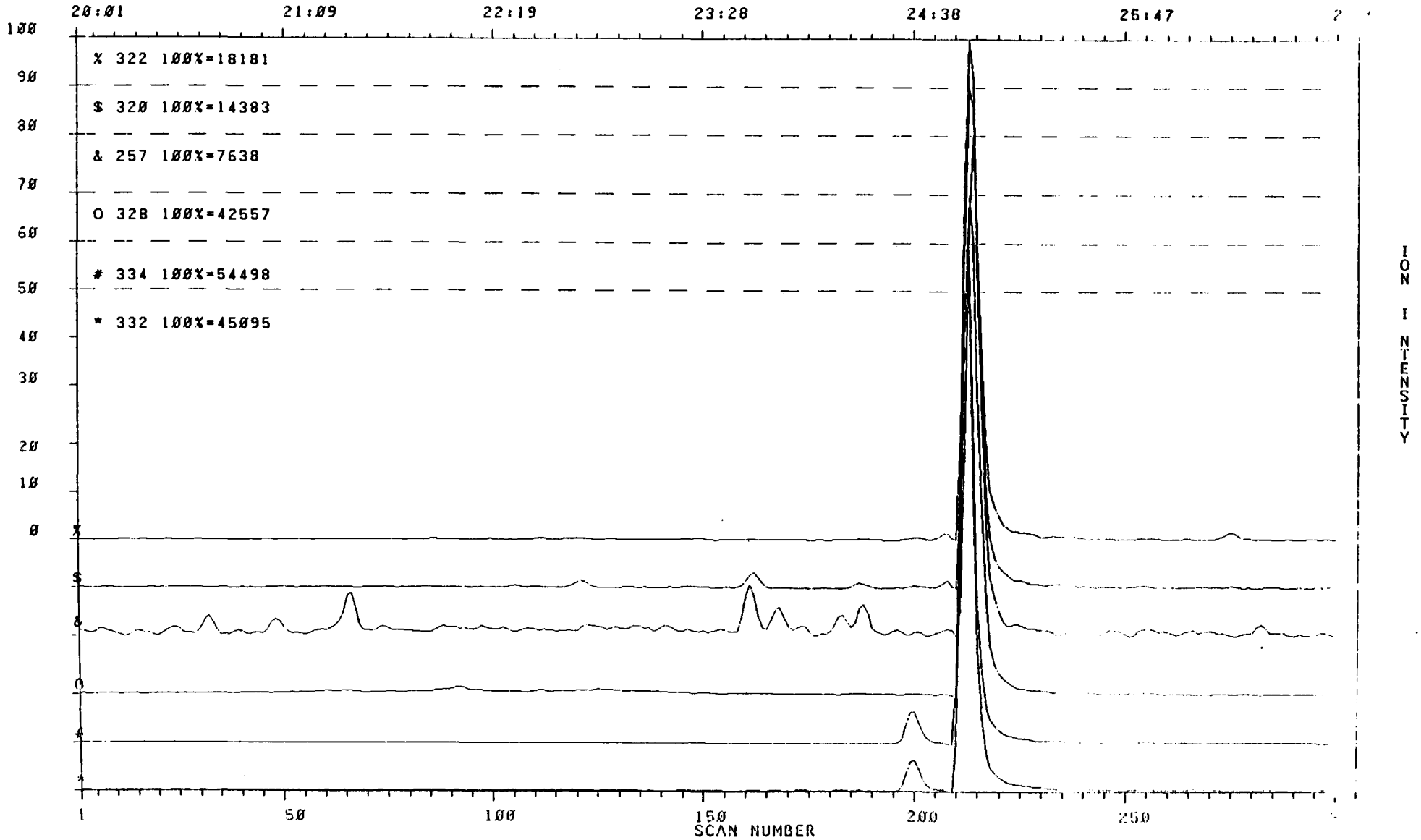
HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. STD 1840-1

FIGURE: 11

DATE: 05/05/86 TIME: 09:51

KRATOS MS25, DS55 SOFTWARE, RUN: CTO40095, WSU NAME: STD 1040-1

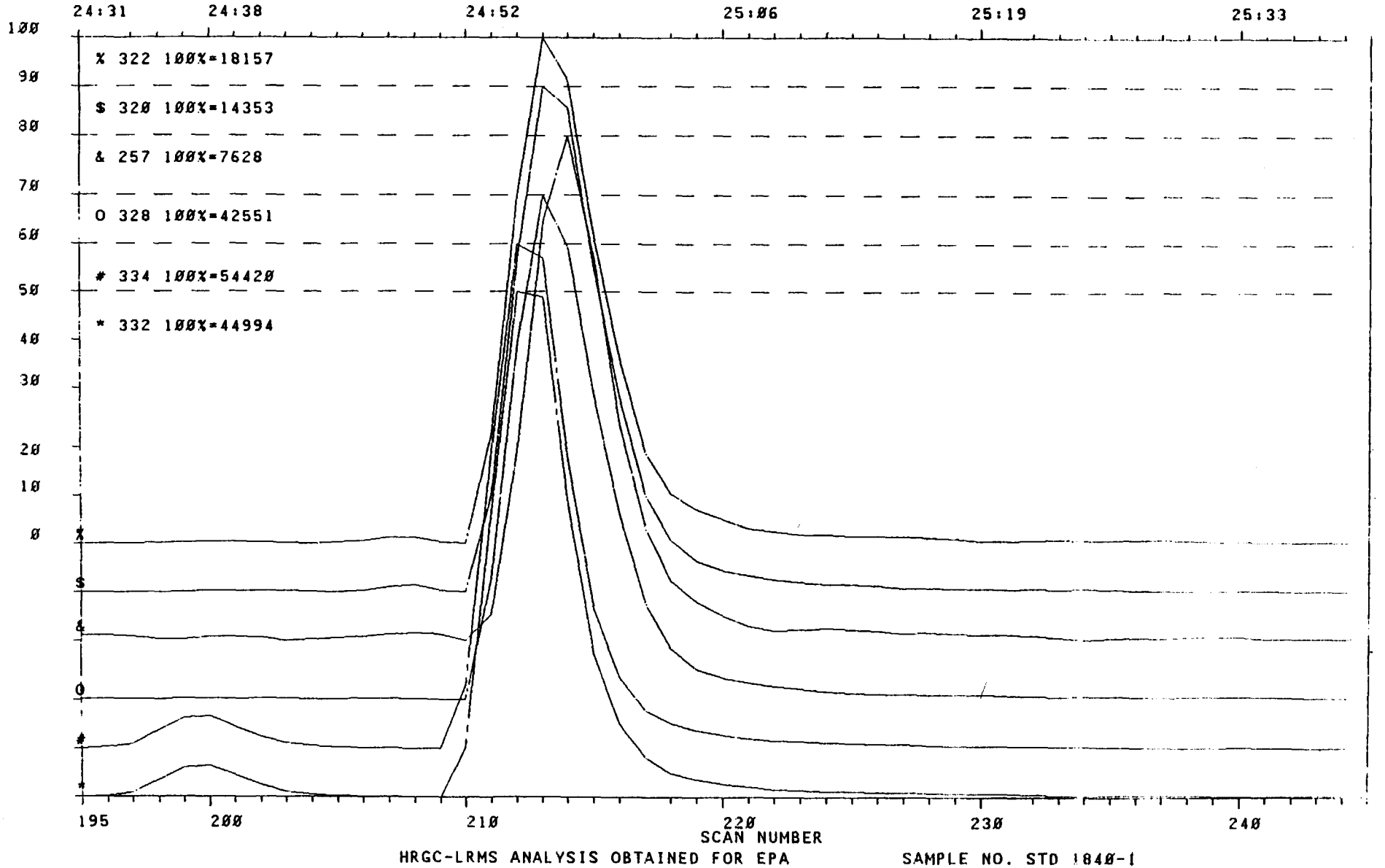
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. STD 1040-1

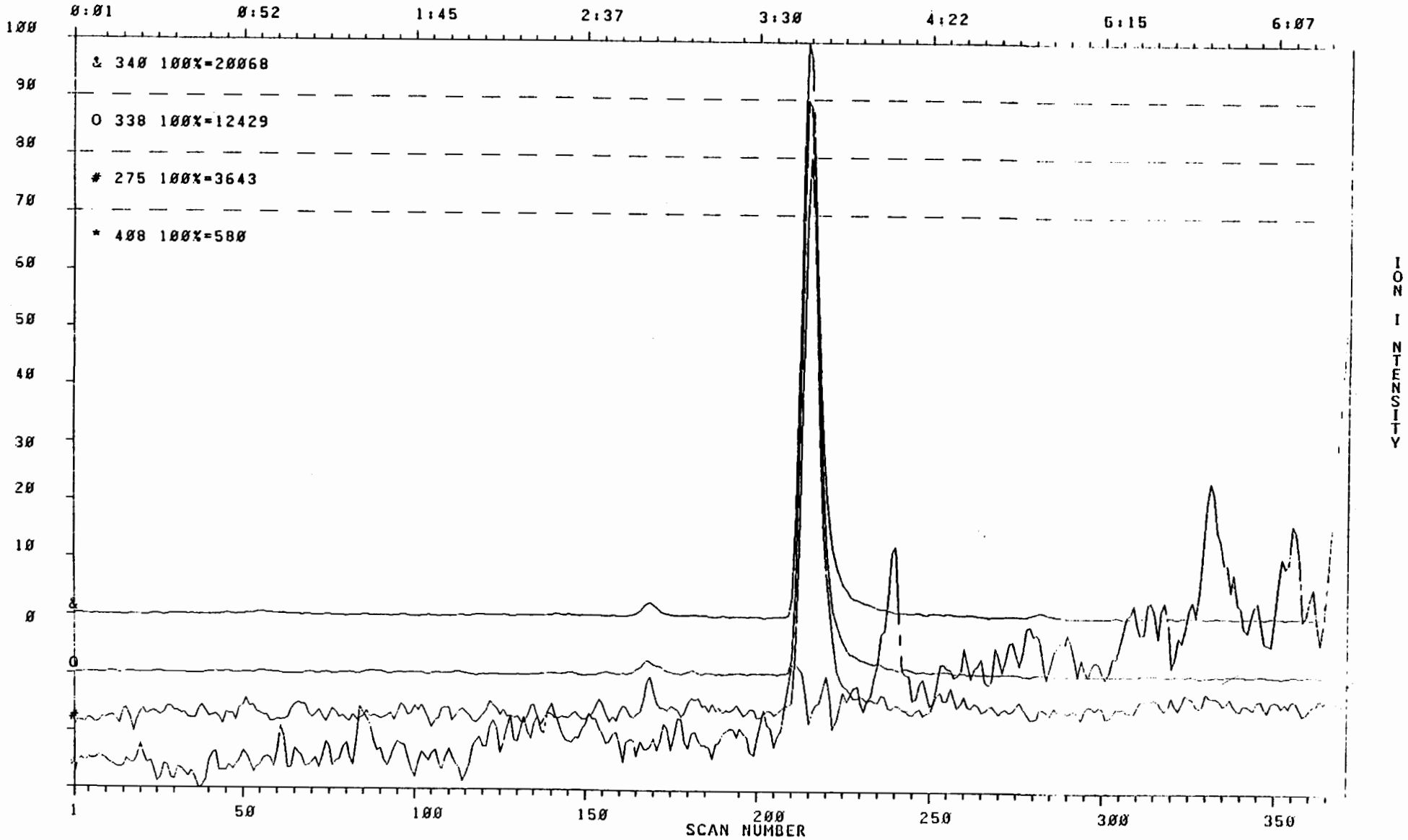
FIGURE: 113

KRATOS MS25, DS55 SOFTWARE, RUN: CTO4ØØ95, WSU NAME: STD 1840-1
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZO-P-DIOXINS



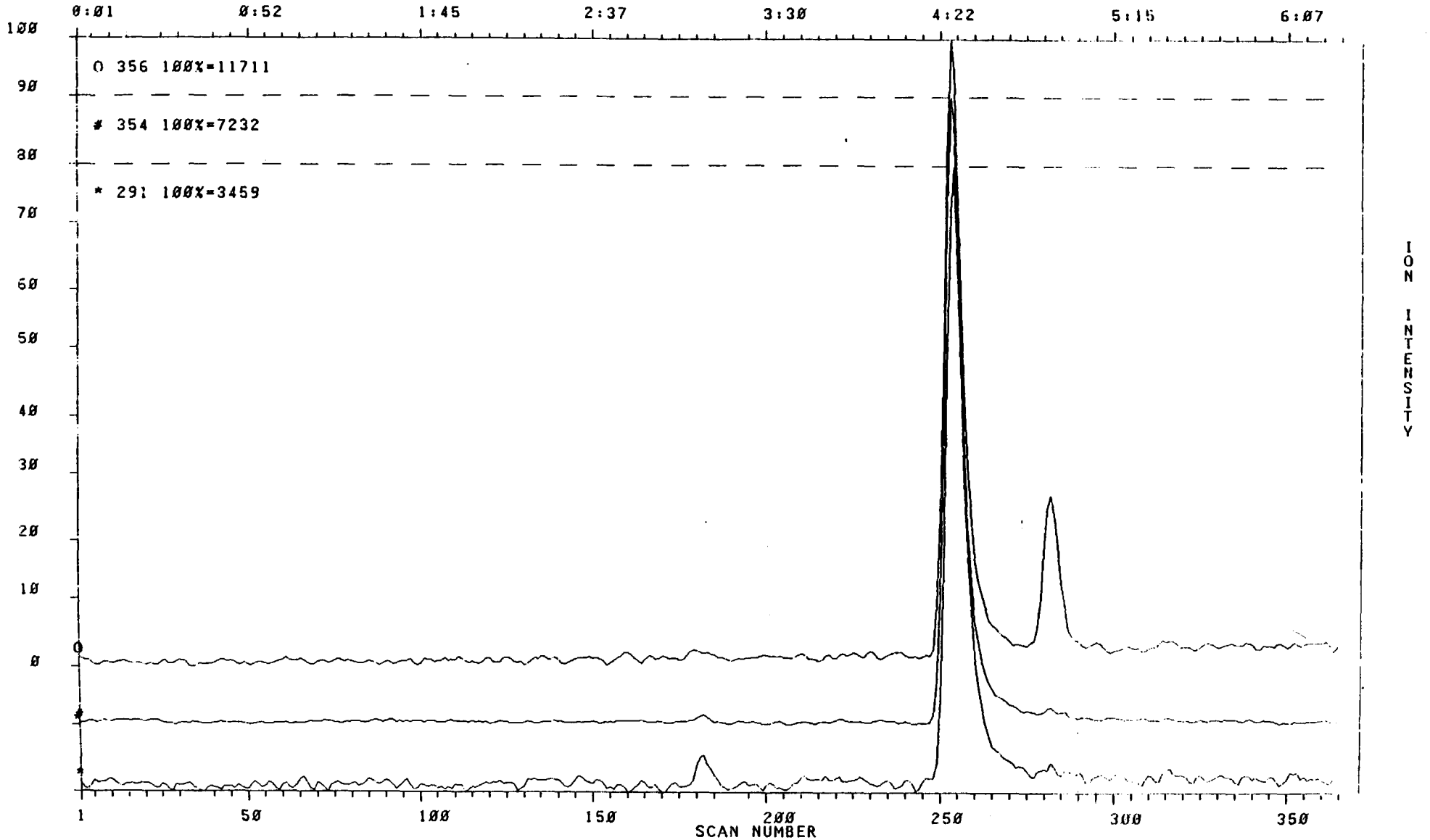
HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. STD 1840-1
FIGURE:114

DATE: 05/05/86 TIME: 09:58
KRATOS MS25, DS55 SOFTWARE, RUN: CTO50095, WSU NAME: STD 1840-1
SELECTED-ION MASS CHROMATOGRAMS FOR PENTACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. STD 1840-1
FIGURE: 115

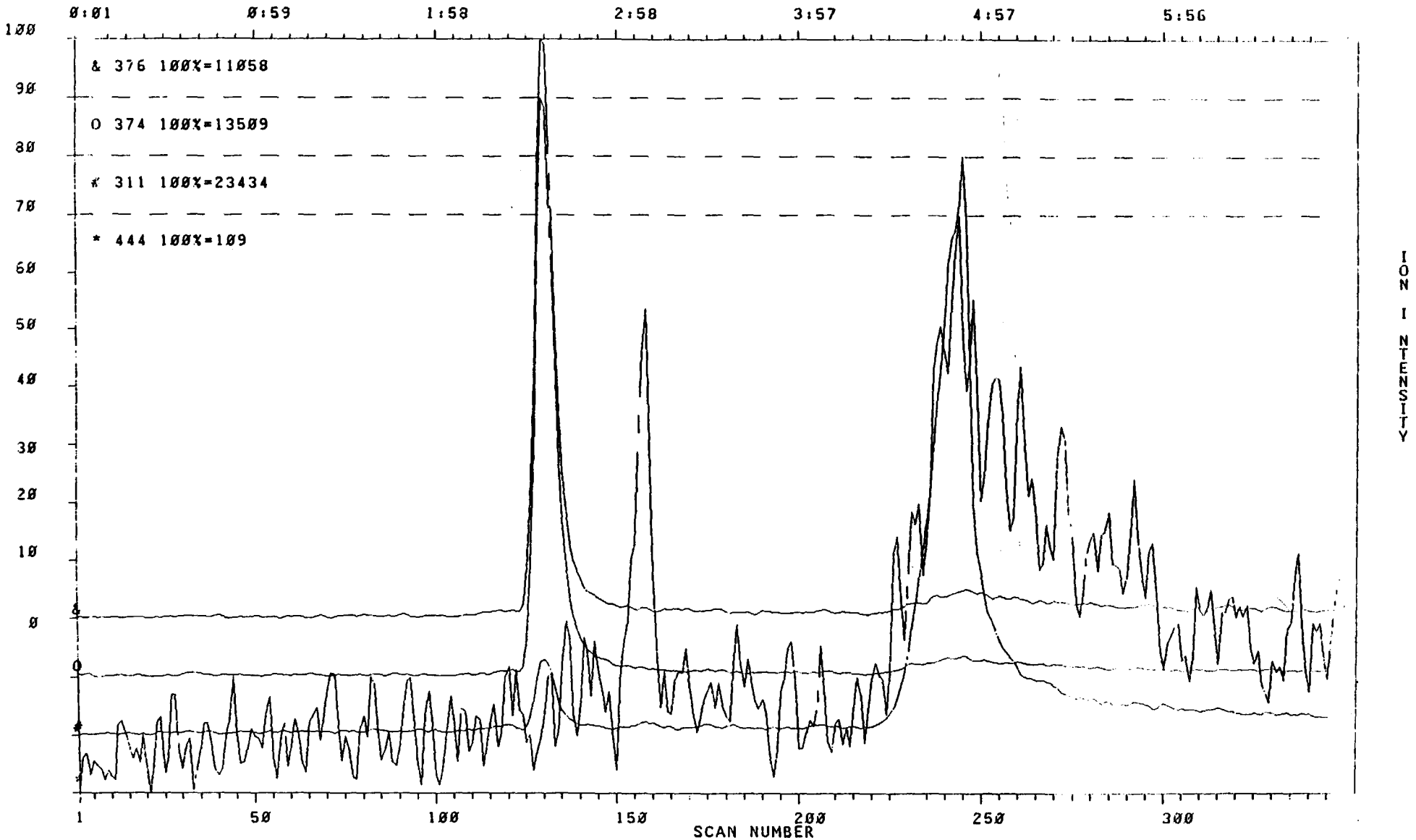
KRATOS MS25, DS55 SOFTWARE, RUN: CTO50095, WSU NAME: STD 1840-1
SELECTED-ION MASS CHROMATOGRAMS FOR PENTACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. STD 1840-1

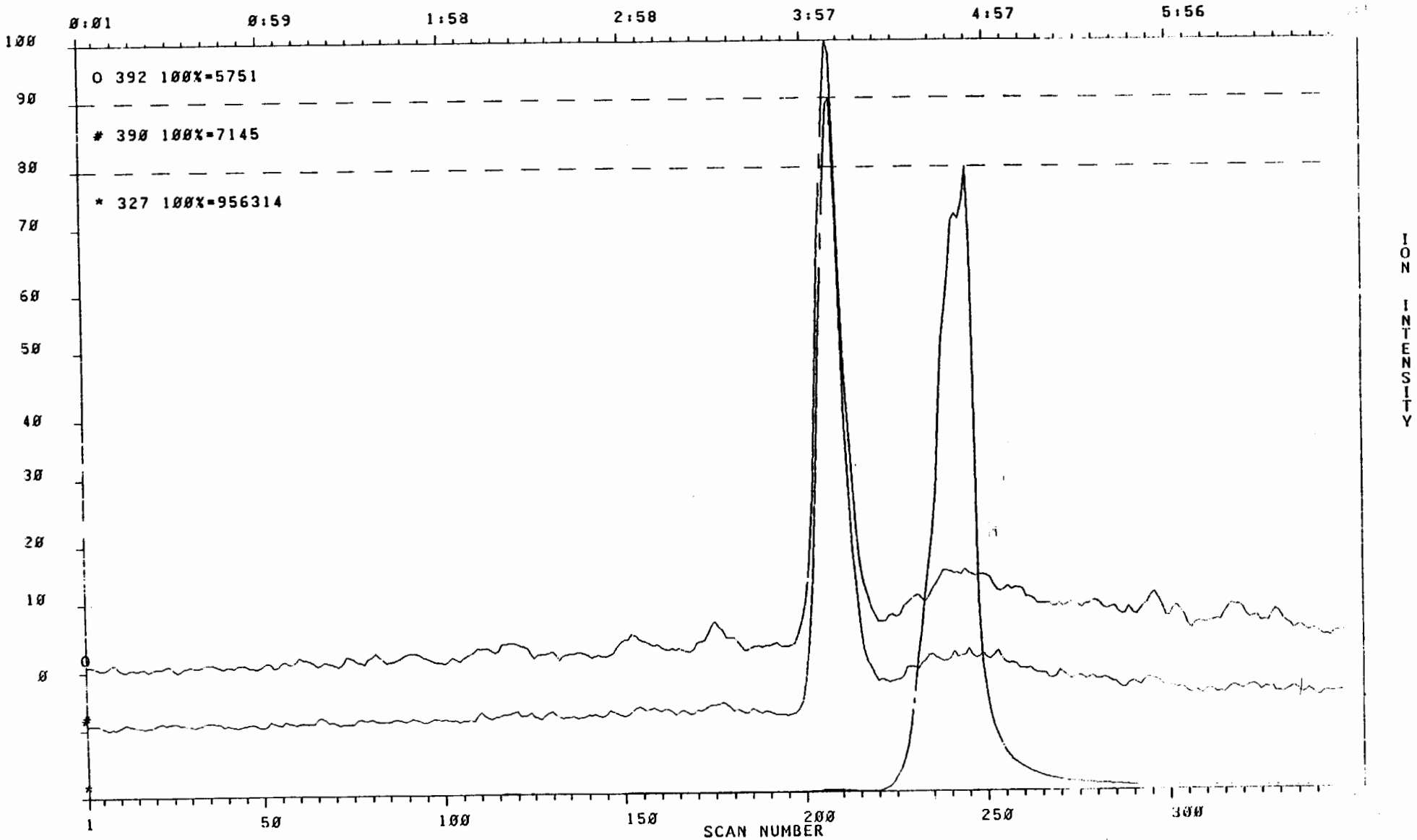
FIGURE: 116

KRATOS MS25, DS55 SOFTWARE, RUN: CTO60095, WSU NAME: STD 1840-1
SELECTED-ION MASS CHROMATOGRAMS FOR HEXACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. STD 1840-1
FIGURE: 117

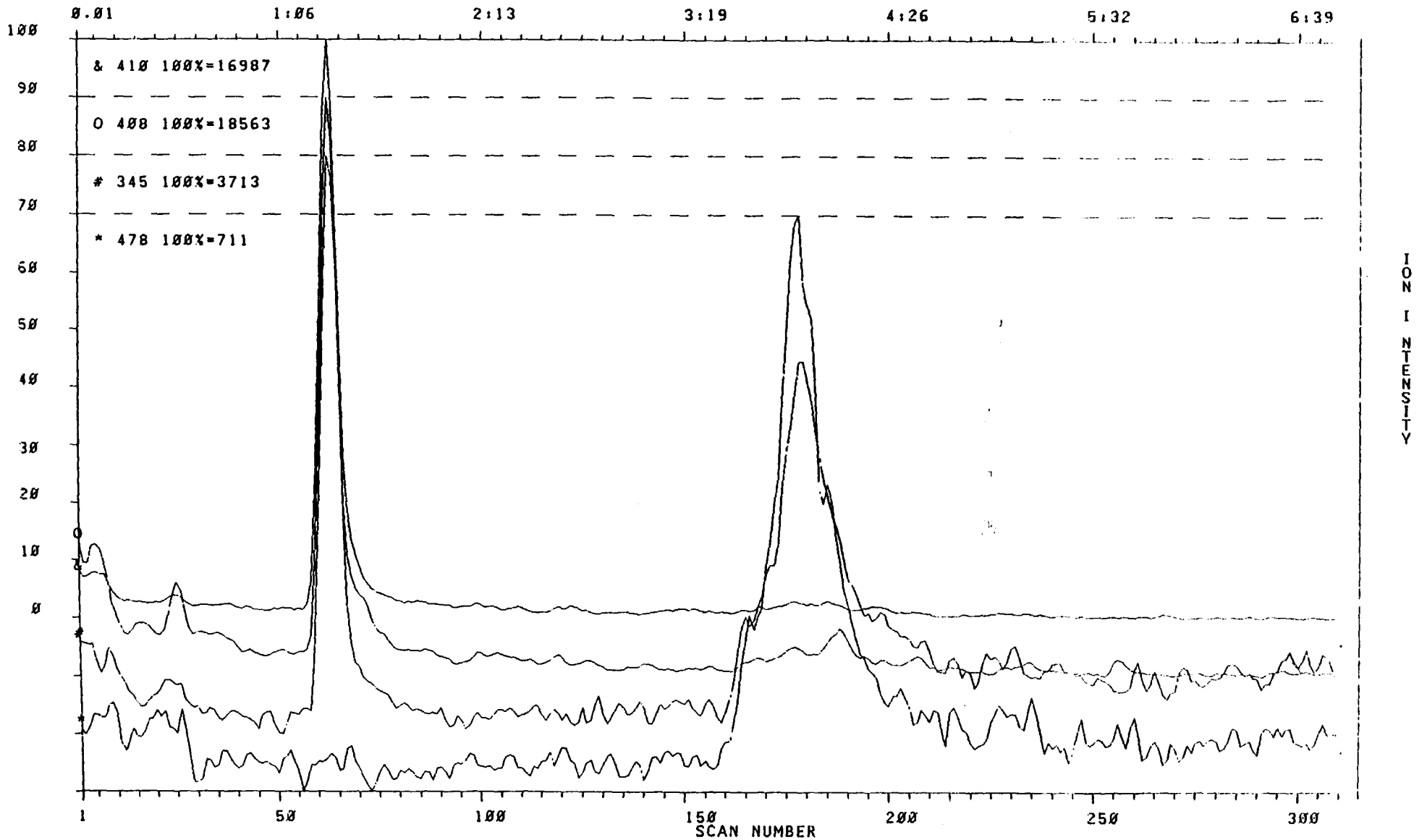
KRATOS MS25, DS55 SOFTWARE, RUN: CTO60095, WSU NAME: STD 1840-1
SELECTED-ION MASS CHROMATOGRAMS FOR HEXACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. STD 1840-1

FIGURE: 118

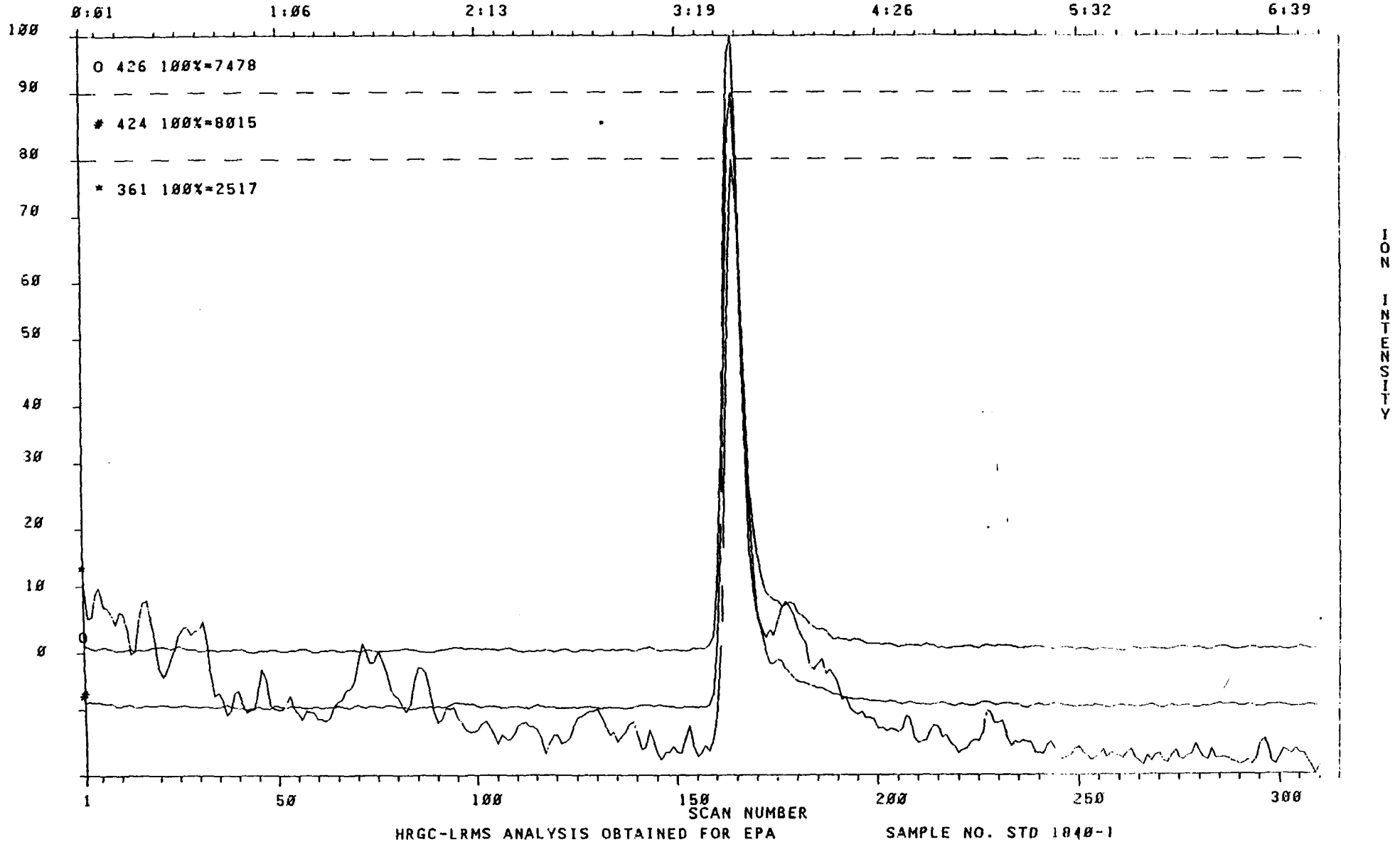
KRATOS MS25, DS55 SOFTWARE, RUN: CTO7ØØ95, WSU NAME: STD 184Ø-1
SELECTED-ION MASS CHROMATOGRAMS FOR HEPTACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. STD 184Ø-1

FIGURE: 119

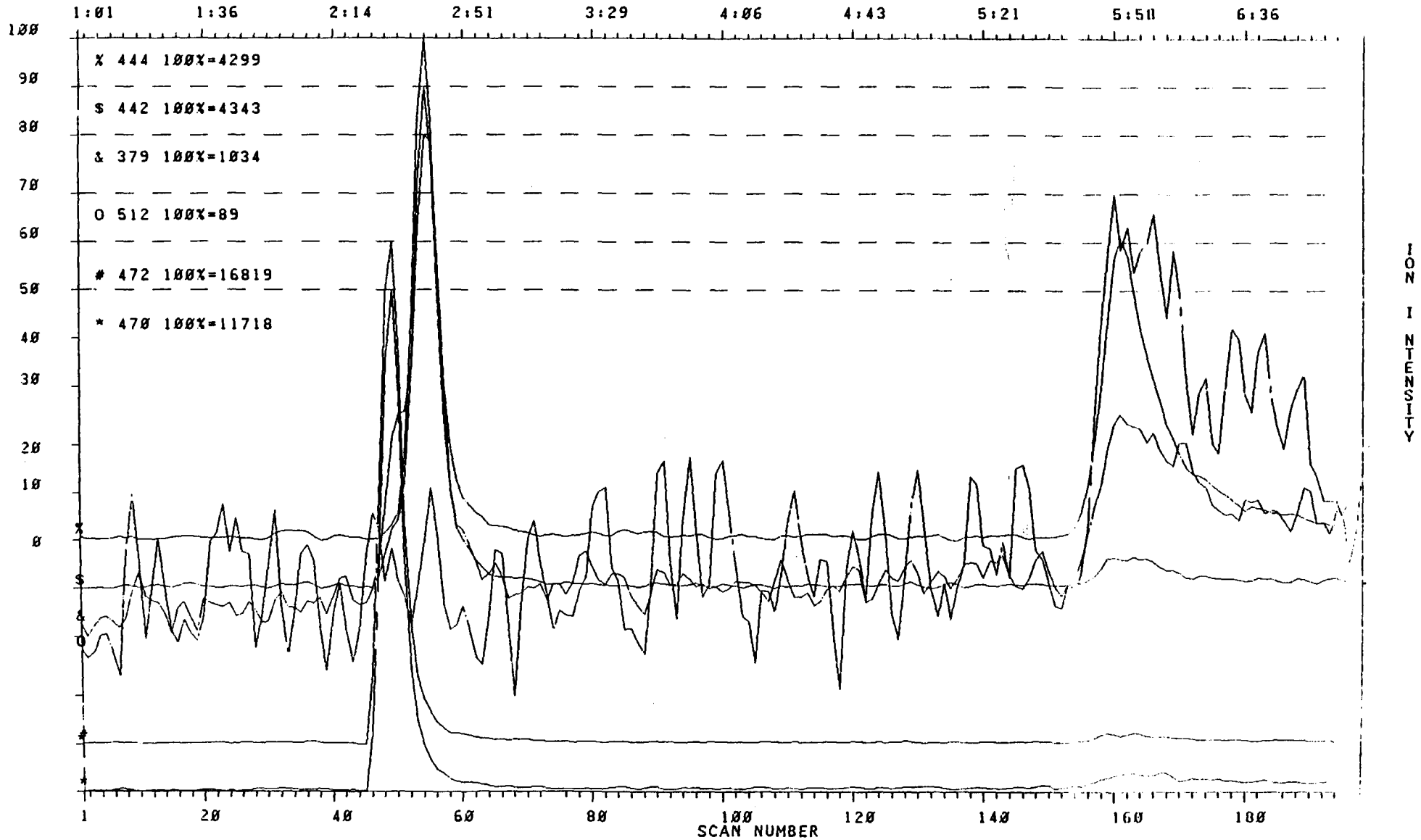
KRATOS MS25, DS55 SOFTWARE, RUN: CTO70095, WSU NAME: STD 1840-1
SELECTED-ION MASS CHROMATOGRAMS FOR HEPTACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. STD 1840-1

FIGURE: 120

KRATOS MS25, DS55 SOFTWARE, RUN: CTO80095, WSU NAME: STD 1840-1
SELECTED-ION MASS CHROMATOGRAMS FOR OCTACHLORODIBENZOFURANS

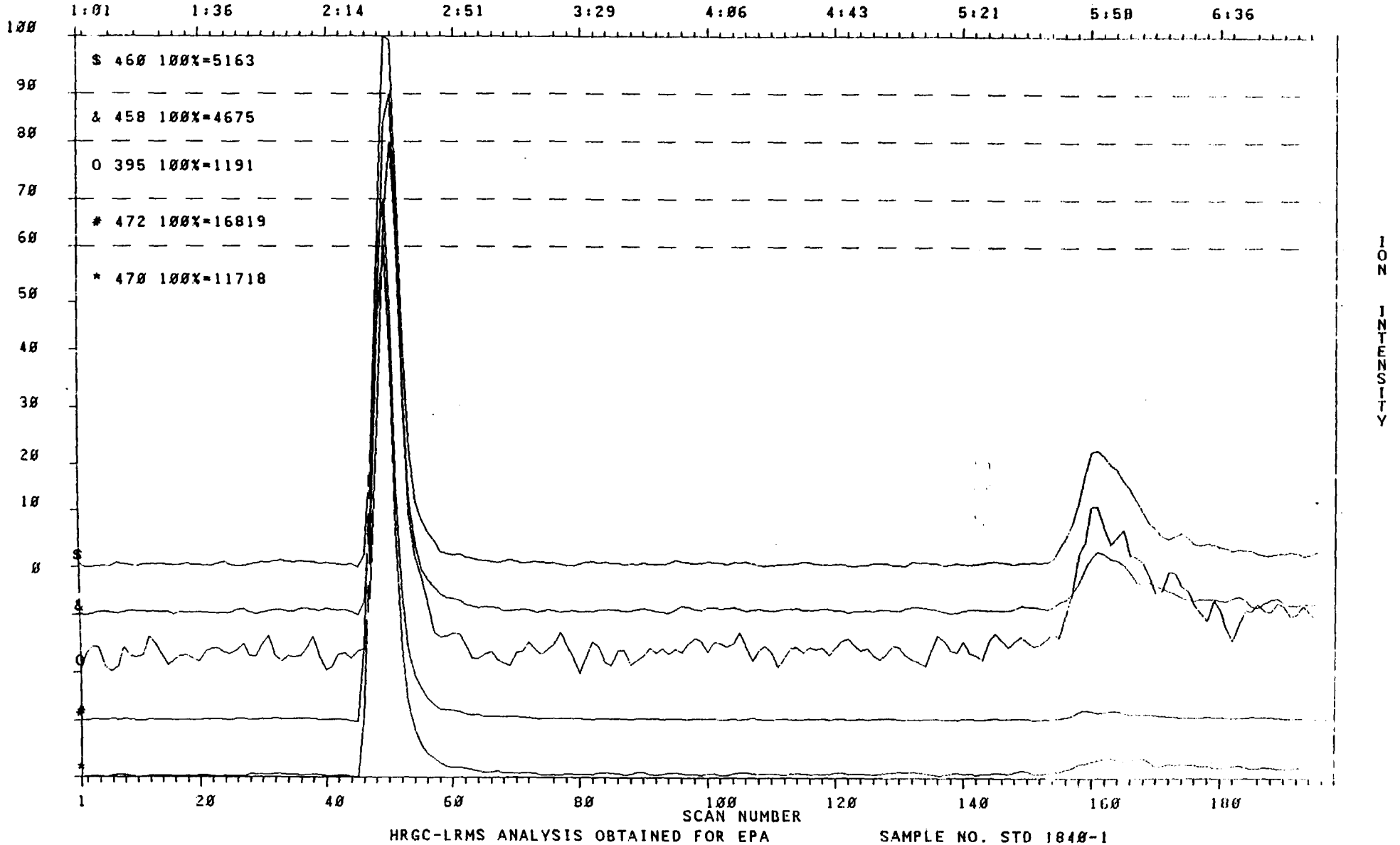


HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. STD 1840-1
FIGURE:121

DATE: 05/05/86 TIME: 10:21

KRATOS MS25, DS55 SOFTWARE, RUN: CTO80095, WSU NAME: STD 1840-1

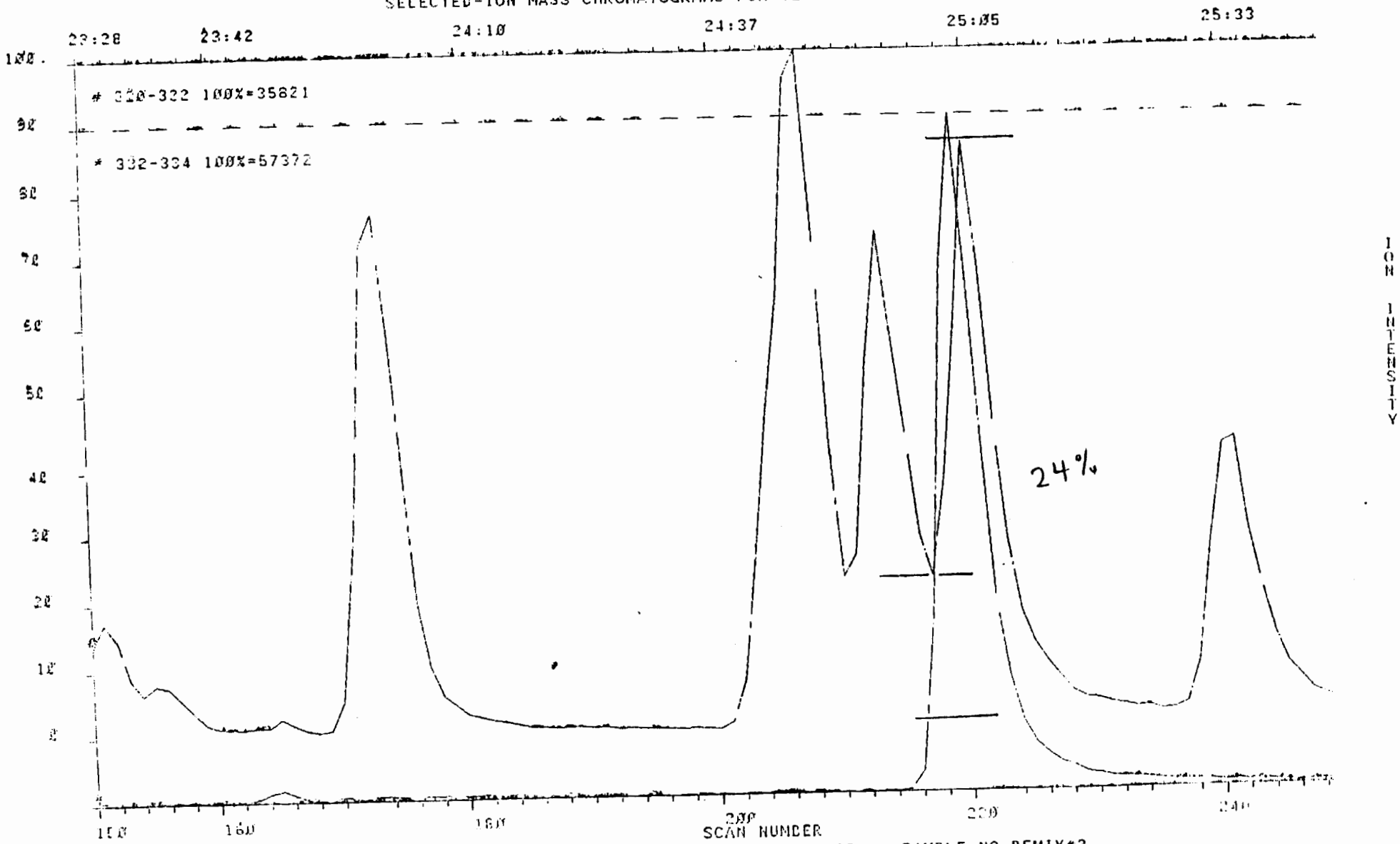
SELECTED-ION MASS CHROMATOGRAMS FOR OCTACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. STD 1840-1

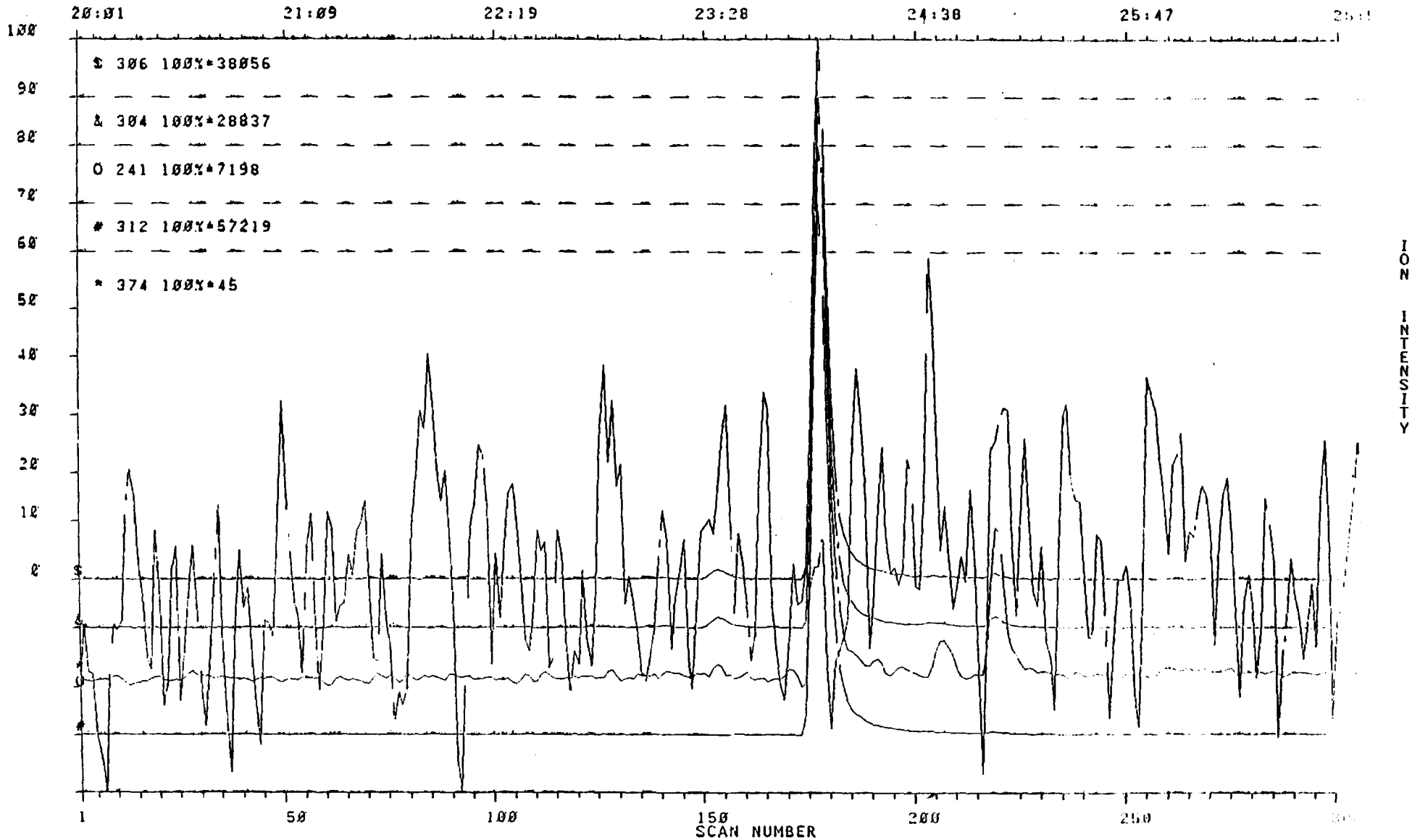
FIGURE: 122

KRATOS MS25, DS55 SOFTWARE, RUN: FGH40125, WSU NAME: PEMIX#2
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZO-P-DIOXINS



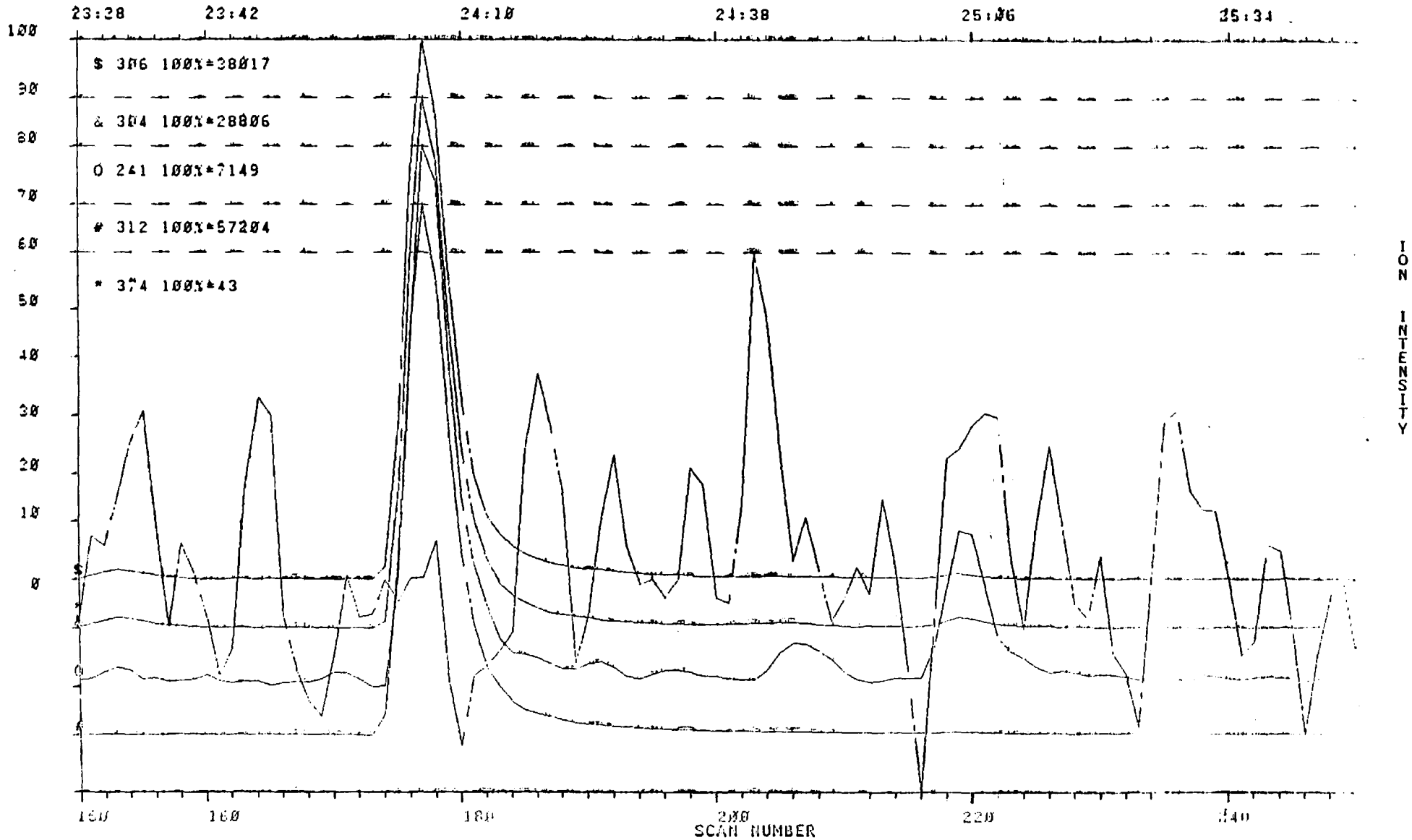
HRGC-LRMS ANALYSIS OBTAINED FOR BREHM LAB SAMPLE NO.PEMIX#2
FIGURE: 123

KRATOS MS25, DS55 SOFTWARE, RUN: CTO40105, WSU NAME: STD 1010-1
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZOFURANS



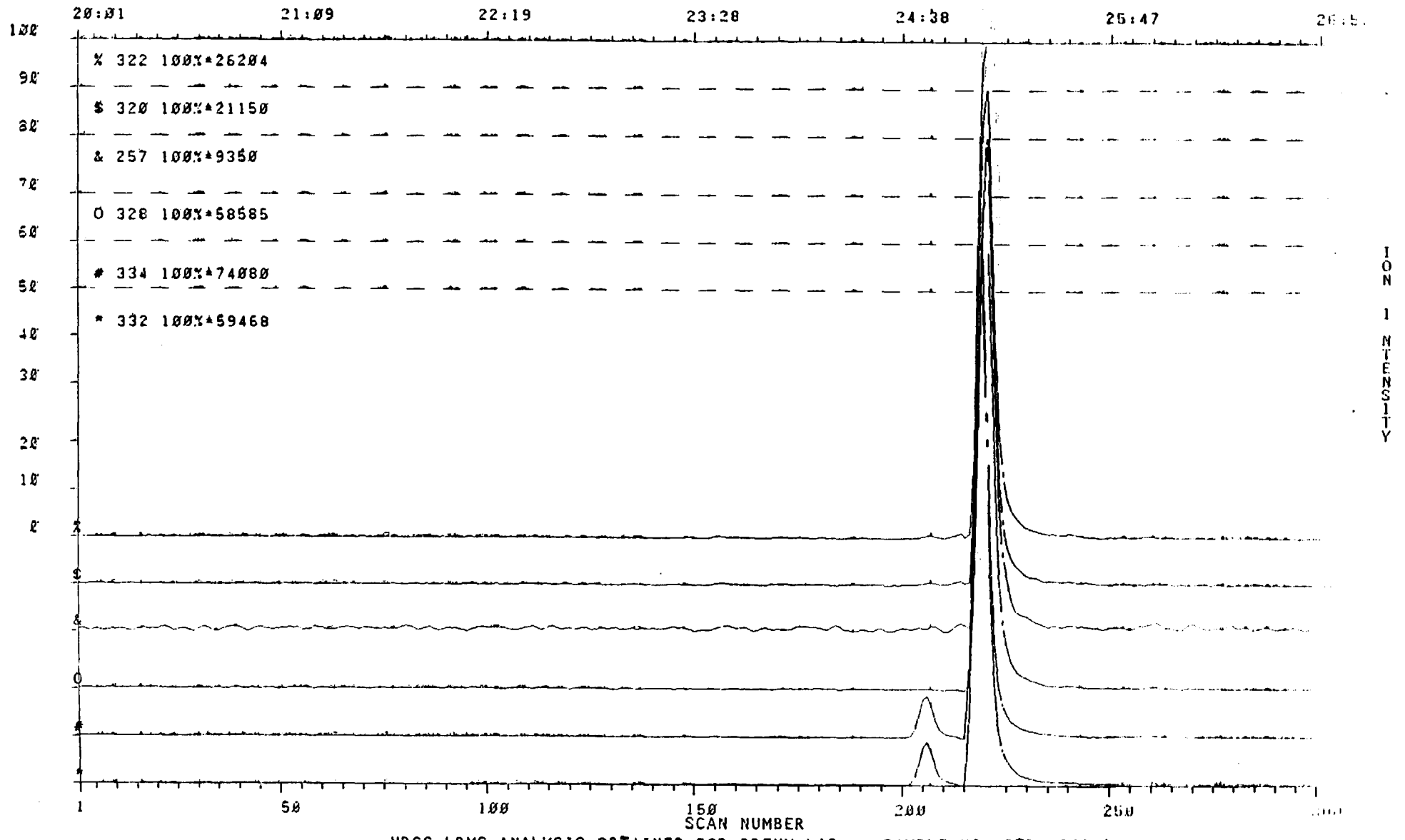
HRGC-LRMS ANALYSIS OBTAINED FOR BREHM LAB SAMPLE NO. STD 1010-1
FIGURE: 124

KRATOS MS25, DS56 SOFTWARE, RUN: CTO4Ø1Ø5, WSU NAME: STD 1840-1
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZOFURANS

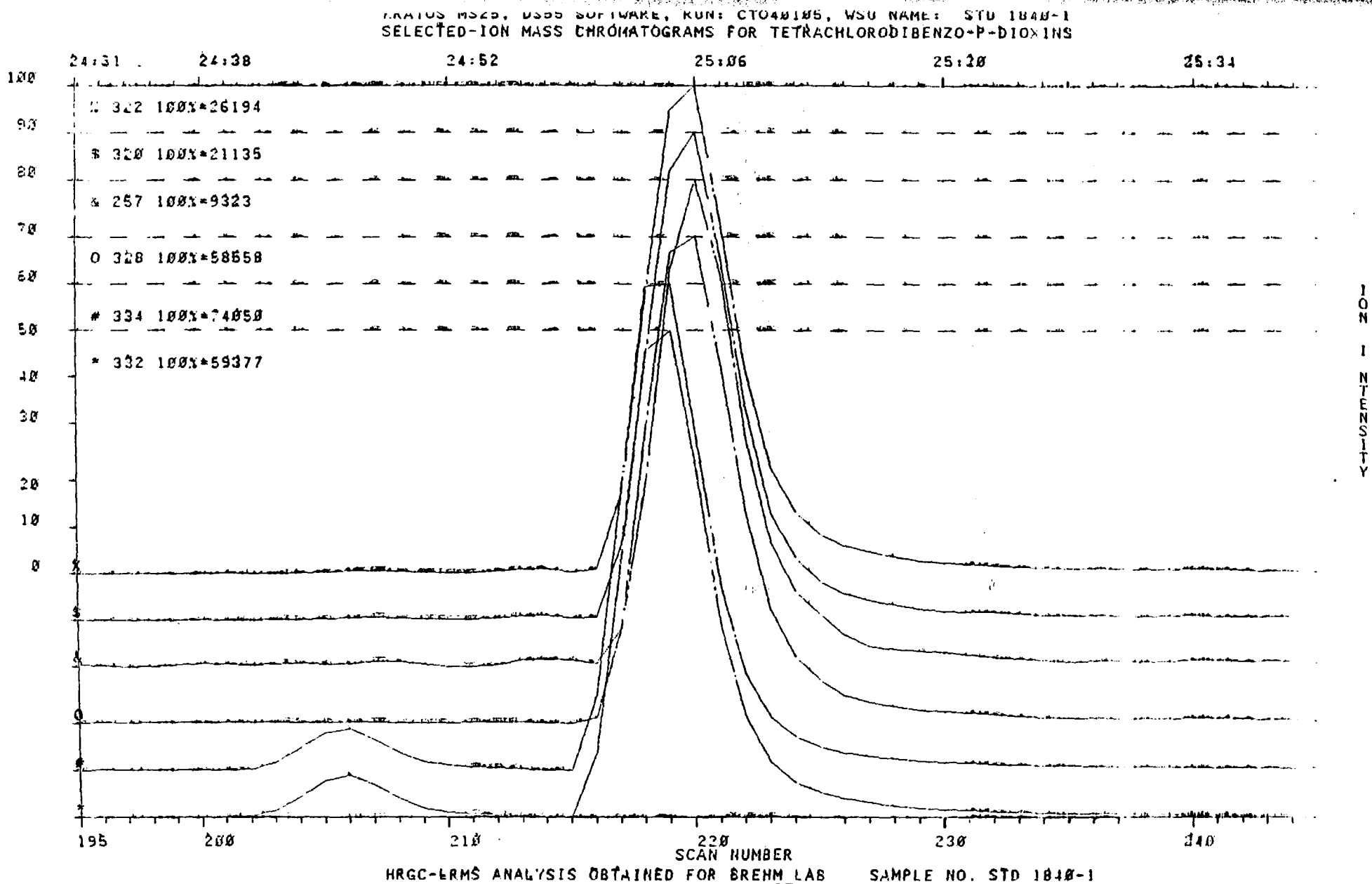


HRGC-ERMS ANALYSIS OBTAINED FOR BREHM LAB SAMPLE NO. STD 1840-1
FIGURE: 125

KRATOS MS25, DS55 SOFTWARE, RUN: CTO4Ø1Ø5, WSU NAME: STD 1840-1
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZO-P-DIOXINS



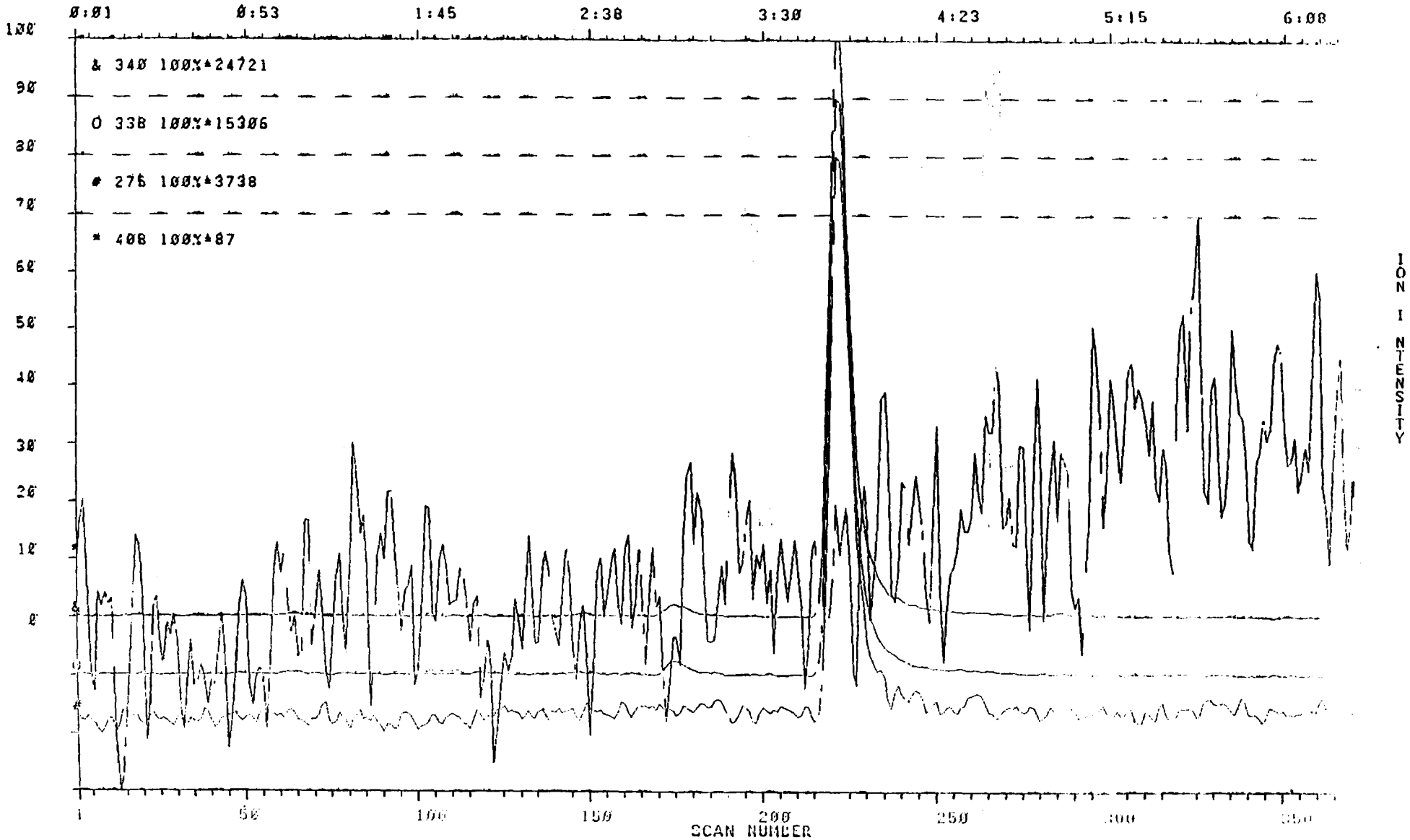
HRGC-LRMS ANALYSIS OBTAINED FOR BREHM LAB SAMPLE NO. STD 1840-1
FIGURE: 126



HRGC-LRMS ANALYSIS OBTAINED FOR BREHM LAB SAMPLE NO. STD 1040-1

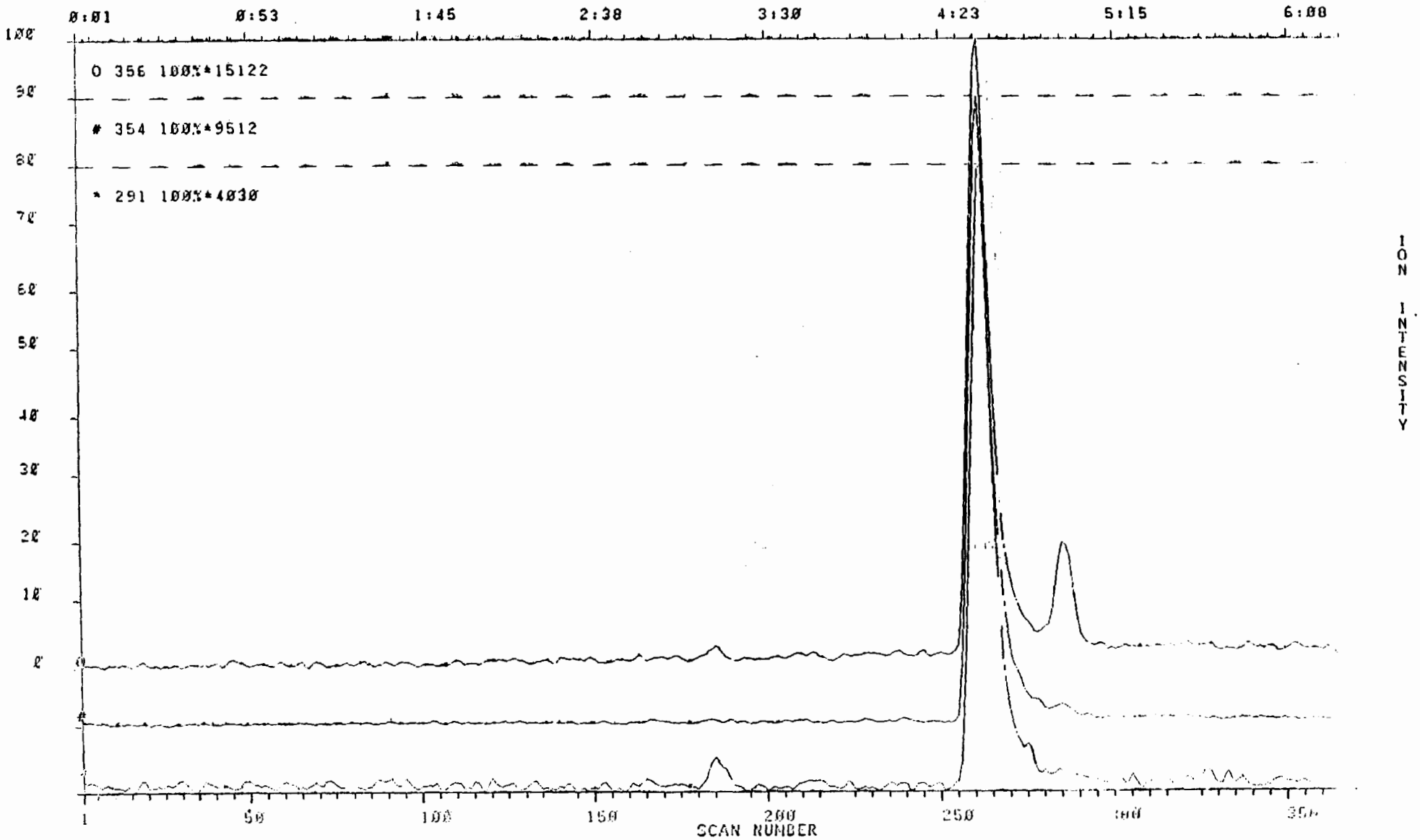
FIGURE: 127

KRATOS MS25, DS55 SOFTWARE, RUN: CT050105, WSU NAME: STD 1840-1
SELECTED-ION MASS CHROMATOGRAMS FOR PENTACHLORODIBENZOFURANS



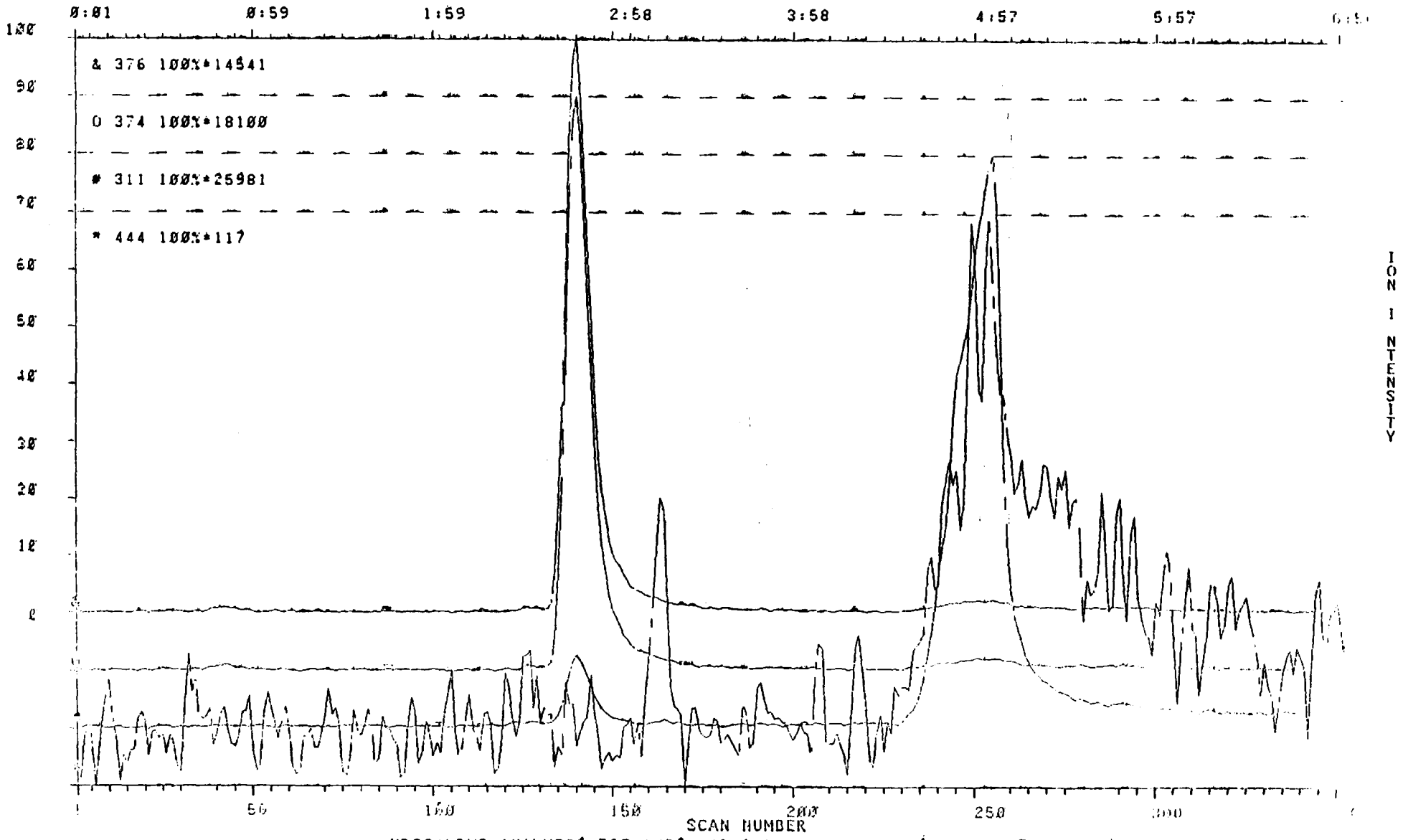
HRGC-LRMS ANALYSIS OBTAINED FOR BREHM LAB SAMPLE NO. STD 1840-1
FIGURE: 128

KRATOS MS25, DS55 SOFTWARE, RUN: CTO50105, WSU NAME: STD 1840-1
SELECTED-ION MASS CHROMATOGRAMS FOR PENTACHLORODIBENZO-P-DIOXINS



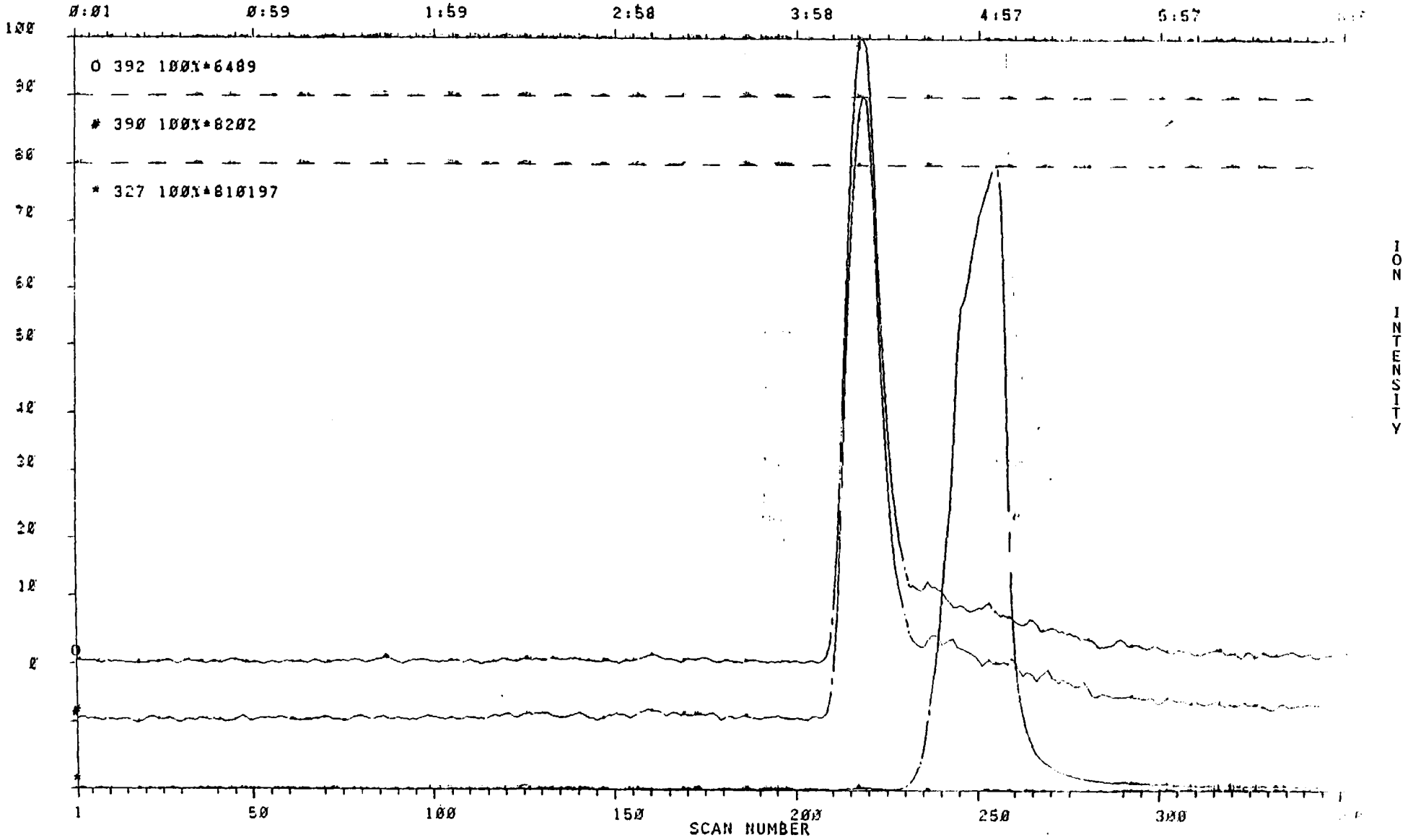
HRGC-LRMS ANALYSIS OBTAINED FOR BREHM LAB SAMPLE NO. STD 1810-1
FIGURE: 129

KRATOS MS25, DS55 SOFTWARE, RUN: CTO60105, WSU NAME: STD 1840-1
SELECTED-ION MASS CHROMATOGRAMS FOR HEXACHLORODIBENZOFURANS



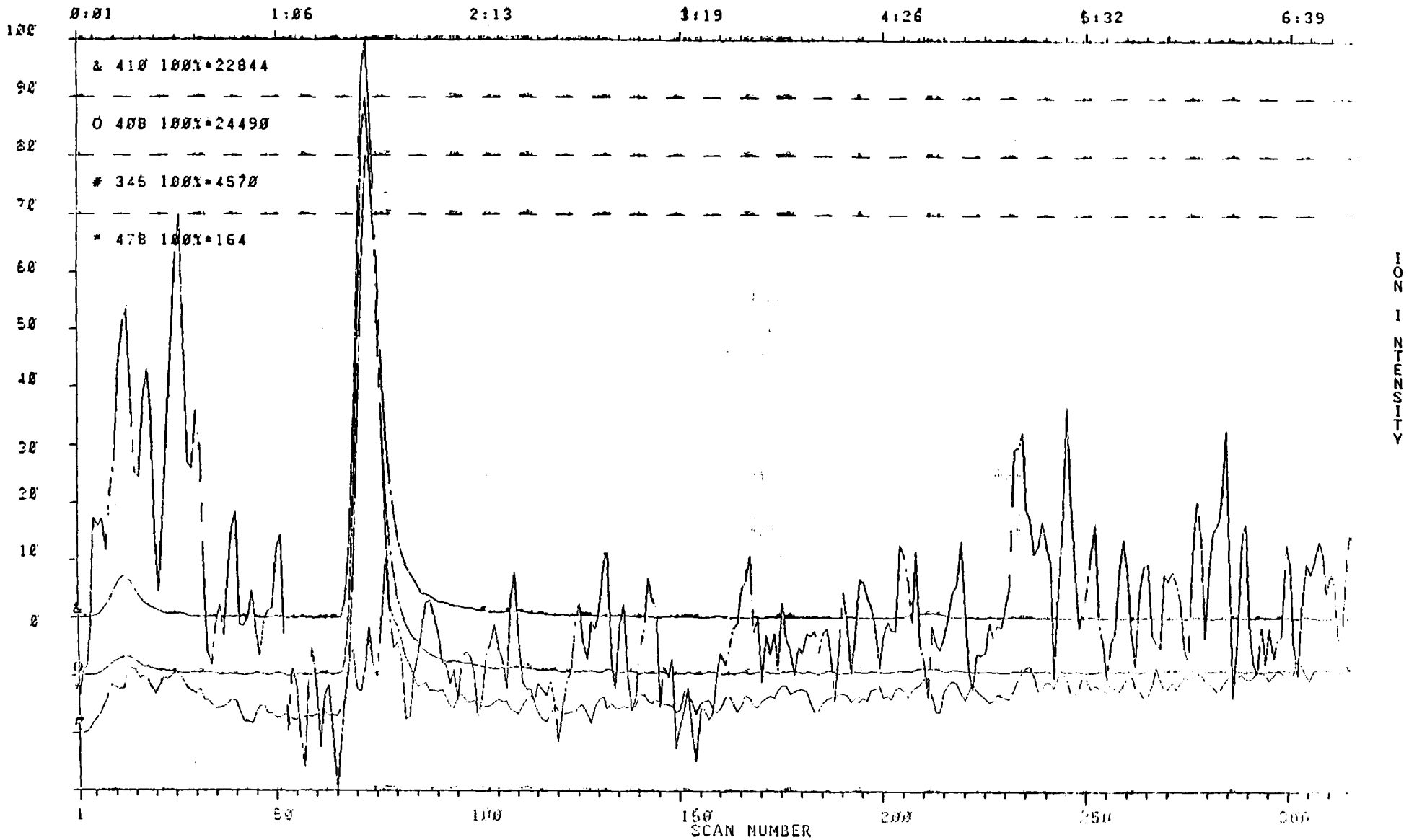
HRGC*LRMS ANALYSIS OBTAINED FOR BREHM LAB SAMPLE NO. STD 1840-1
FIGURE: 130

KRATOS MS25, DS55 SOFTWARE, RUN: CTO60105, WSU NAME: STD 1840-1
SELECTED-ION MASS CHROMATOGRAMS FOR HEXACHLORODIBENZO-P-DIOXINS



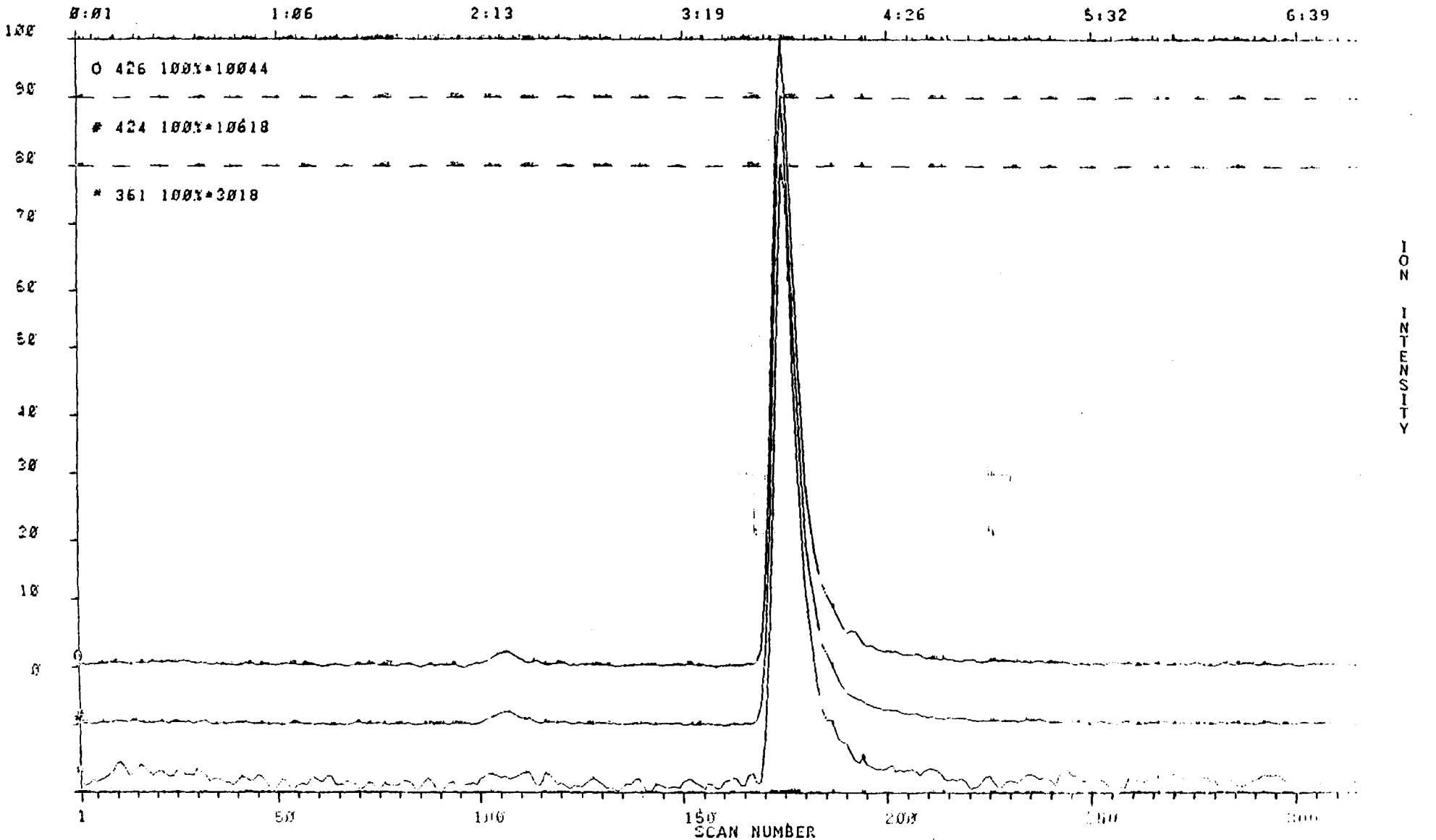
HRGC-LRMS ANALYSIS OBTAINED FOR BREHM LAB SAMPLE NO. STD 1840-1
FIGURE: 131

KRATOS MS25, DS55 SOFTWARE, RUN: CTO70105, WSU NAME: STD 1840-1
SELECTED-ION MASS CHROMATOGRAMS FOR HEPTACHLORODIBENZOFURANS



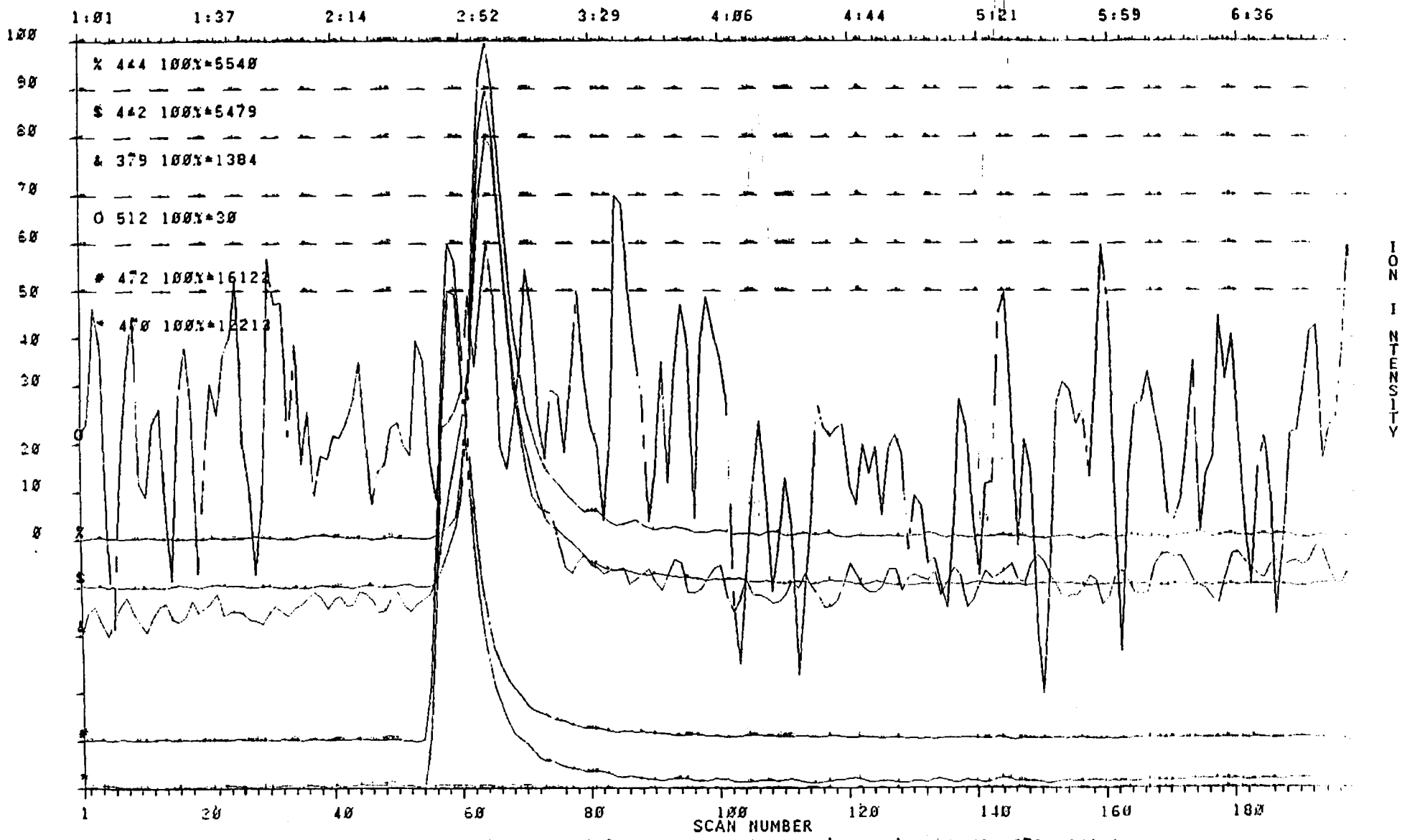
HRGC-LRMS ANALYSIS OBTAINED FOR BREHM LAB SAMPLE NO. STD 1840-1
FIGURE: 132

KRATOS MS25, DS55 SOFTWARE, RUN: CT07Ø1Ø5, WSU NAME: STD 1840-1
SELECTED-ION MASS CHROMATOGRAMS FOR HEPTACHLORODIBENZO-P-DIOXINS



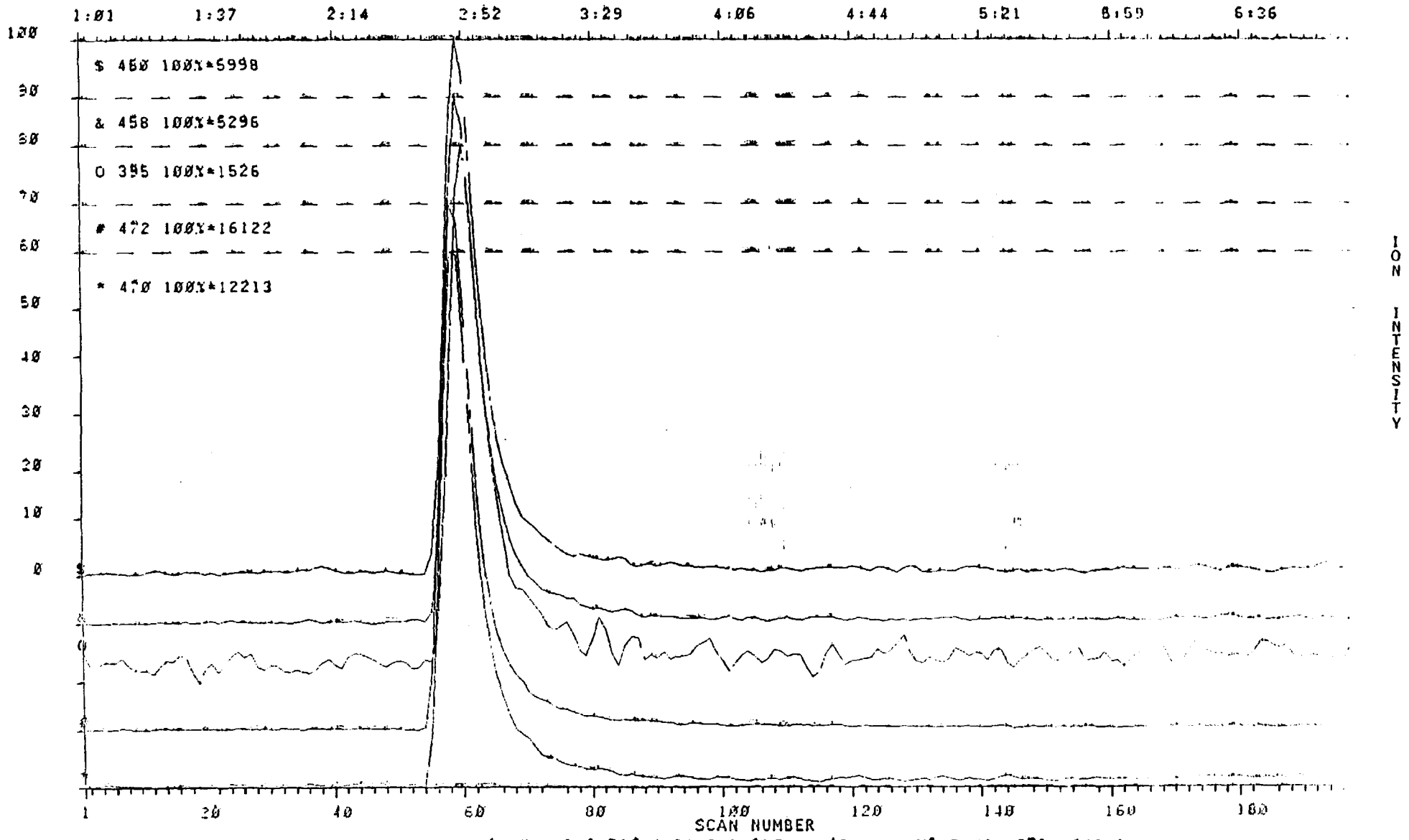
HRGC-LRMS ANALYSIS OBTAINED FOR BREHM LAB SAMPLE NO. STD 1818-1
FIGURE: 133

KRATOS MS25, DS56 SOFTWARE, RUN: CTO80105, WSU NAME: STD 1840-1
SELECTED-ION MASS CHROMATOGRAMS FOR OCTACHLORODIBENZOFURANS



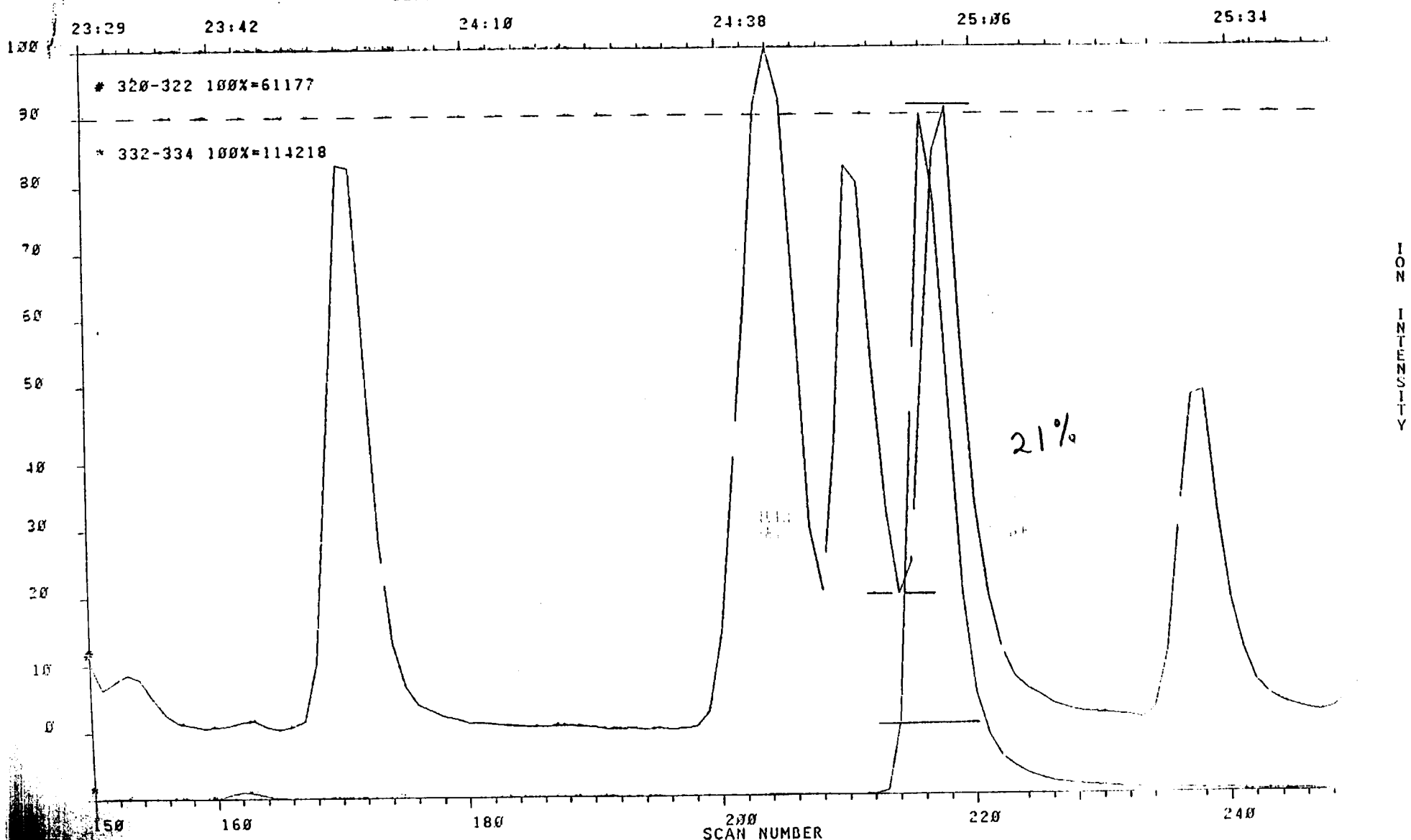
HRGC-LRMS ANALYSIS OBTAINED FOR BREHM LAB SAMPLE NO. STD 1840-1
FIGURE: 134

KRATOS MS25, DS55 SOFTWARE, RUN: CTOB0105, WSU NAME: STD 1840-1
SELECTED-ION MASS CHROMATOGRAMS FOR OCTACHLORODIBENZO-P-DIOXINS



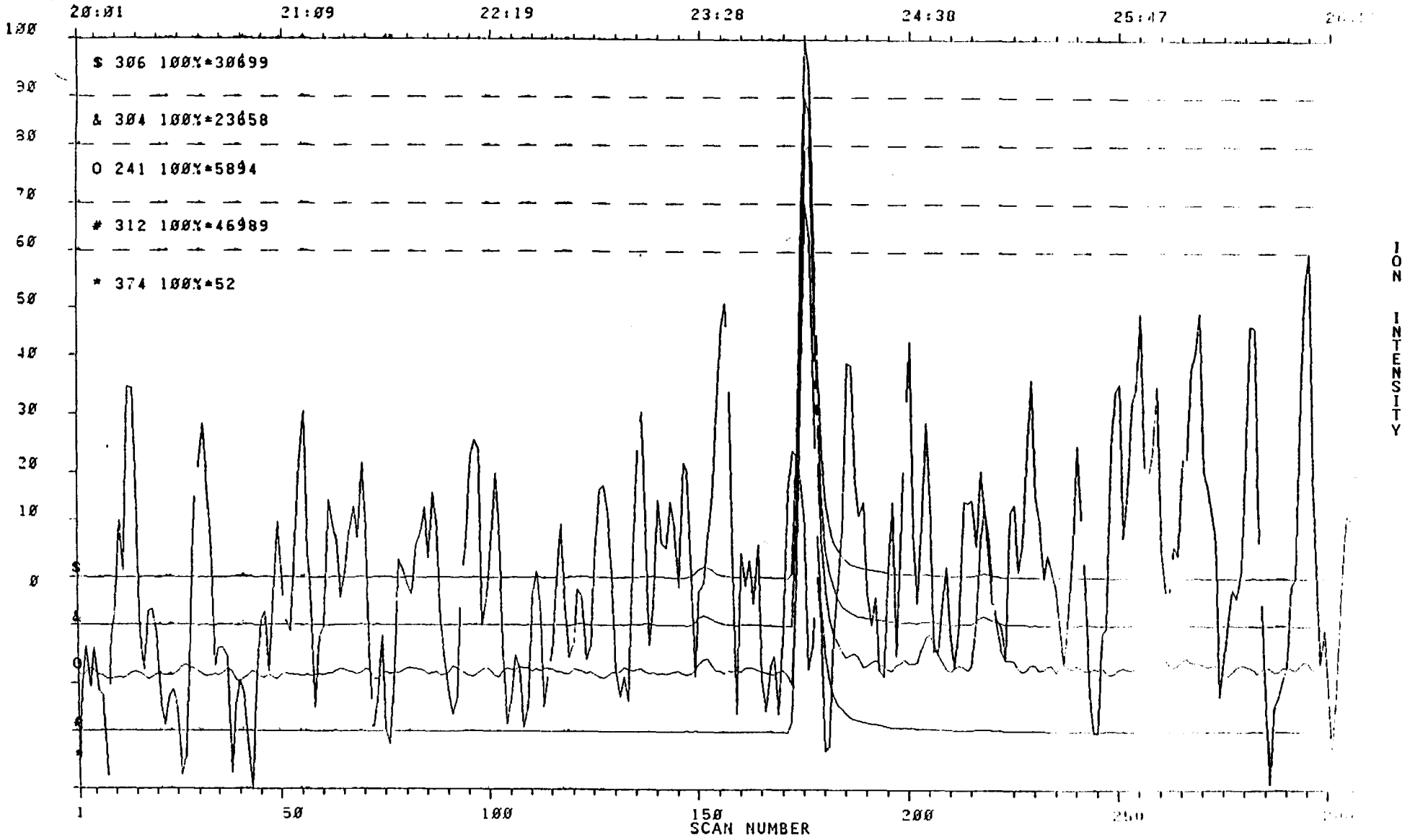
HRGC-LRMS ANALYSIS OBTAINED FOR BREHM LAB SAMPLE NO. STD 1840-1
FIGURE: 135

KRATOS MS25, DS55 SOFTWARE, RUN: FGH40126, WSU NAME: PEMIX#2
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZO-P-DIOXINS

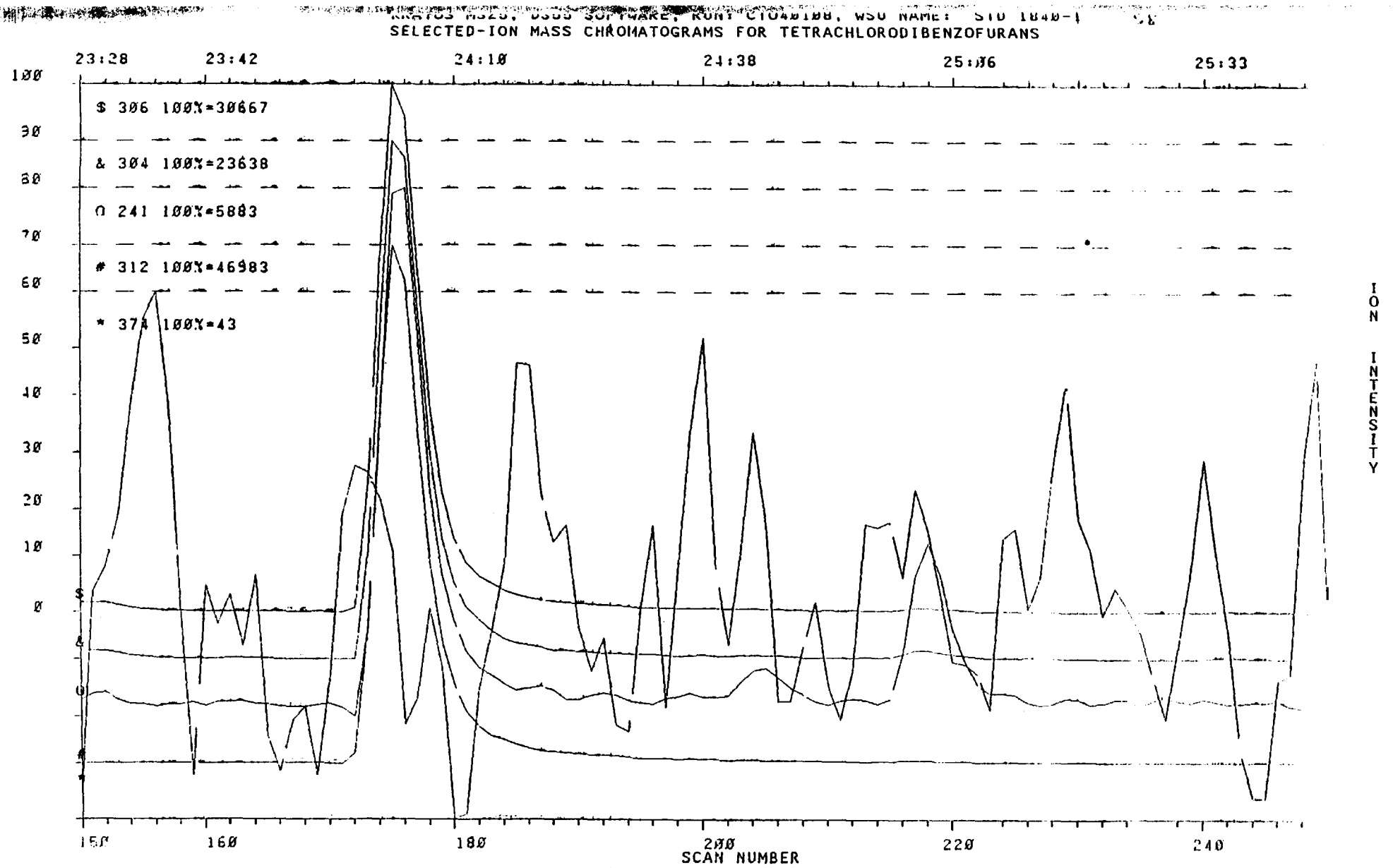


HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.PEMIX#2
FIGURE: 136

KRATOS MS25, DS55 SOFTWARE, RUN: CTO401Ø8, WSU NAME: STD 1Ø4Ø-1
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZOFURANS



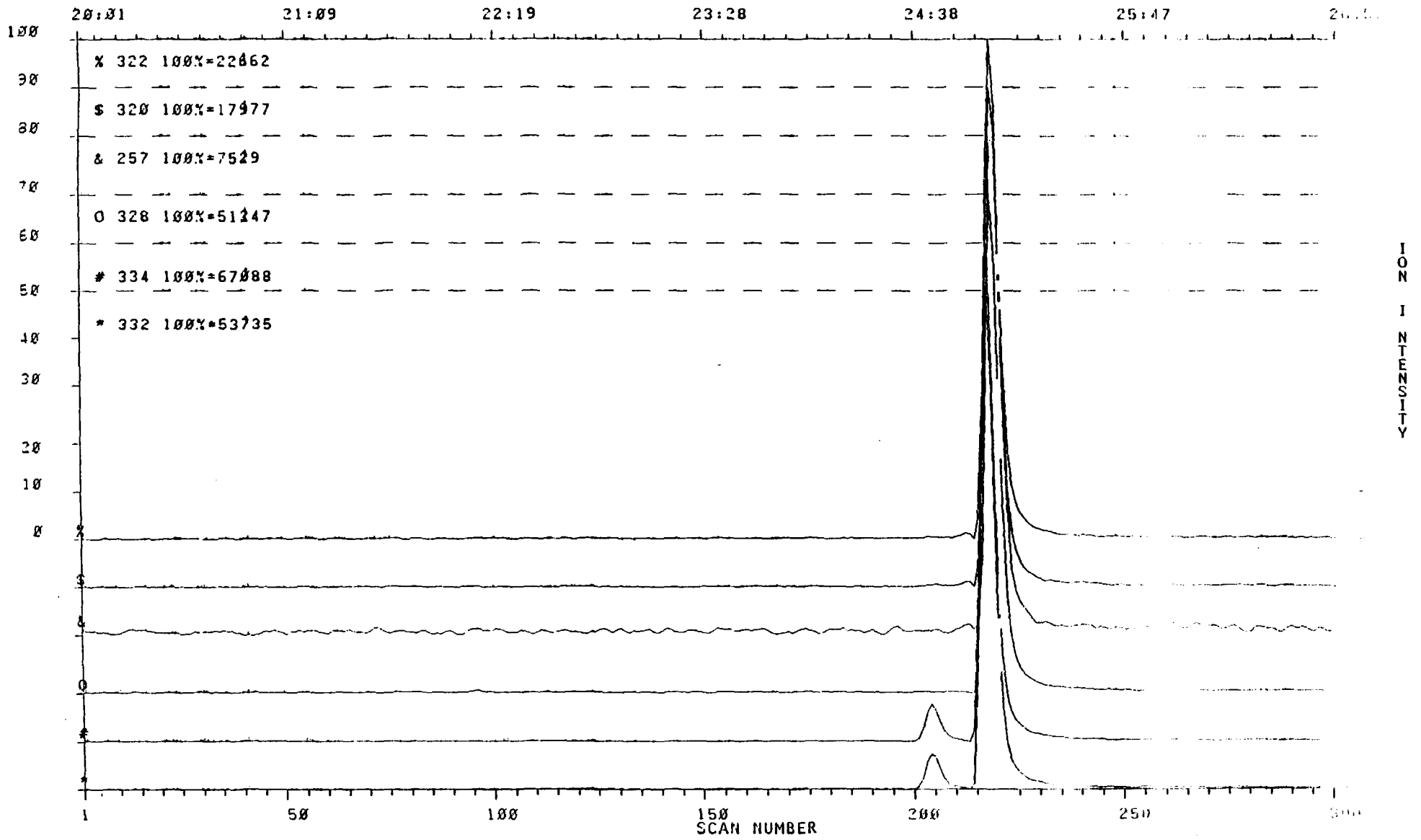
HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.STD 1Ø4Ø-1
FIGURE: 137



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.STD 1840-1

FIGURE: 138

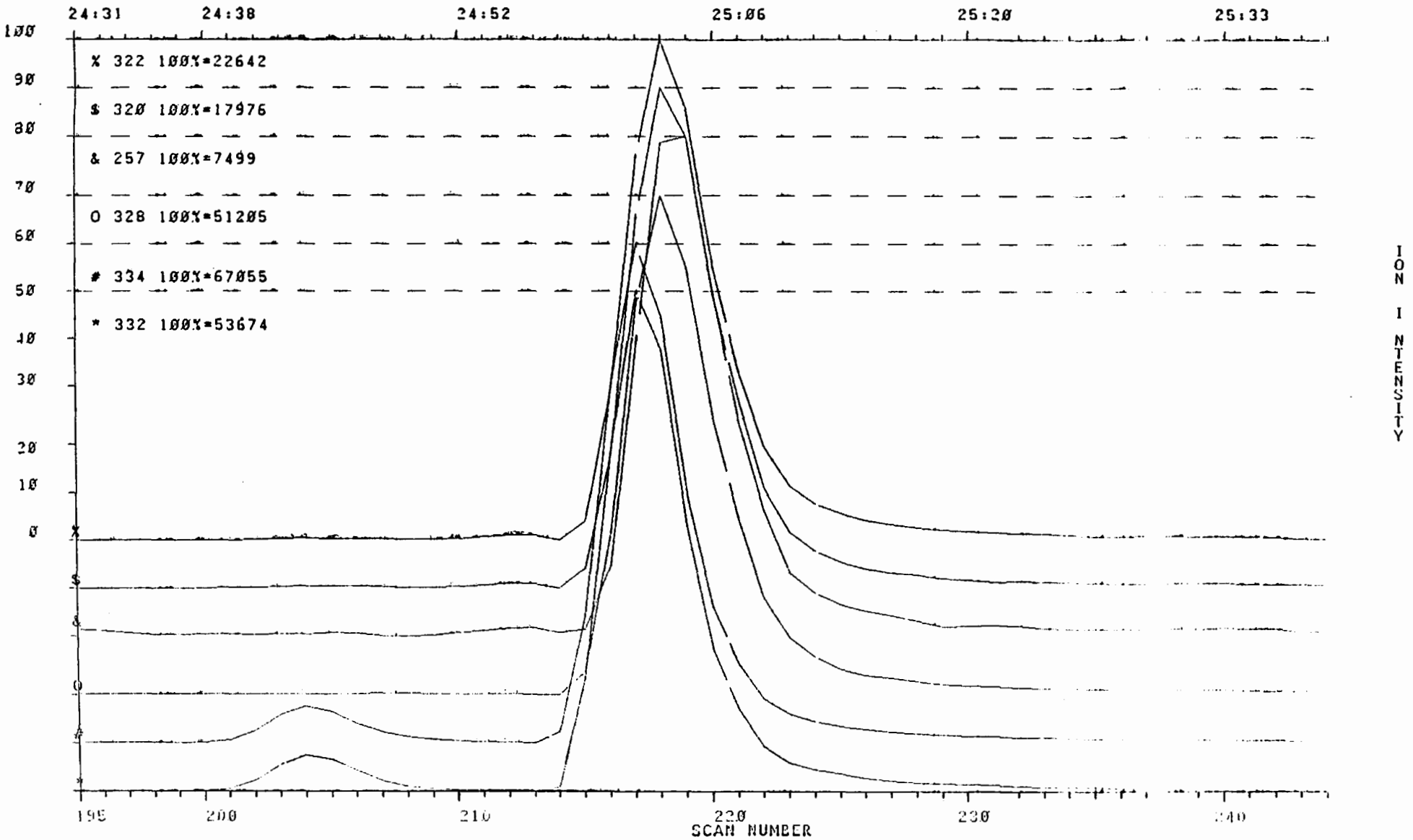
KRATOS MS25, DS55 SOFTWARE, RUN: CTO40108, WSU NAME: STD 1840-1
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.STD 1840-1

FIGURE: 139

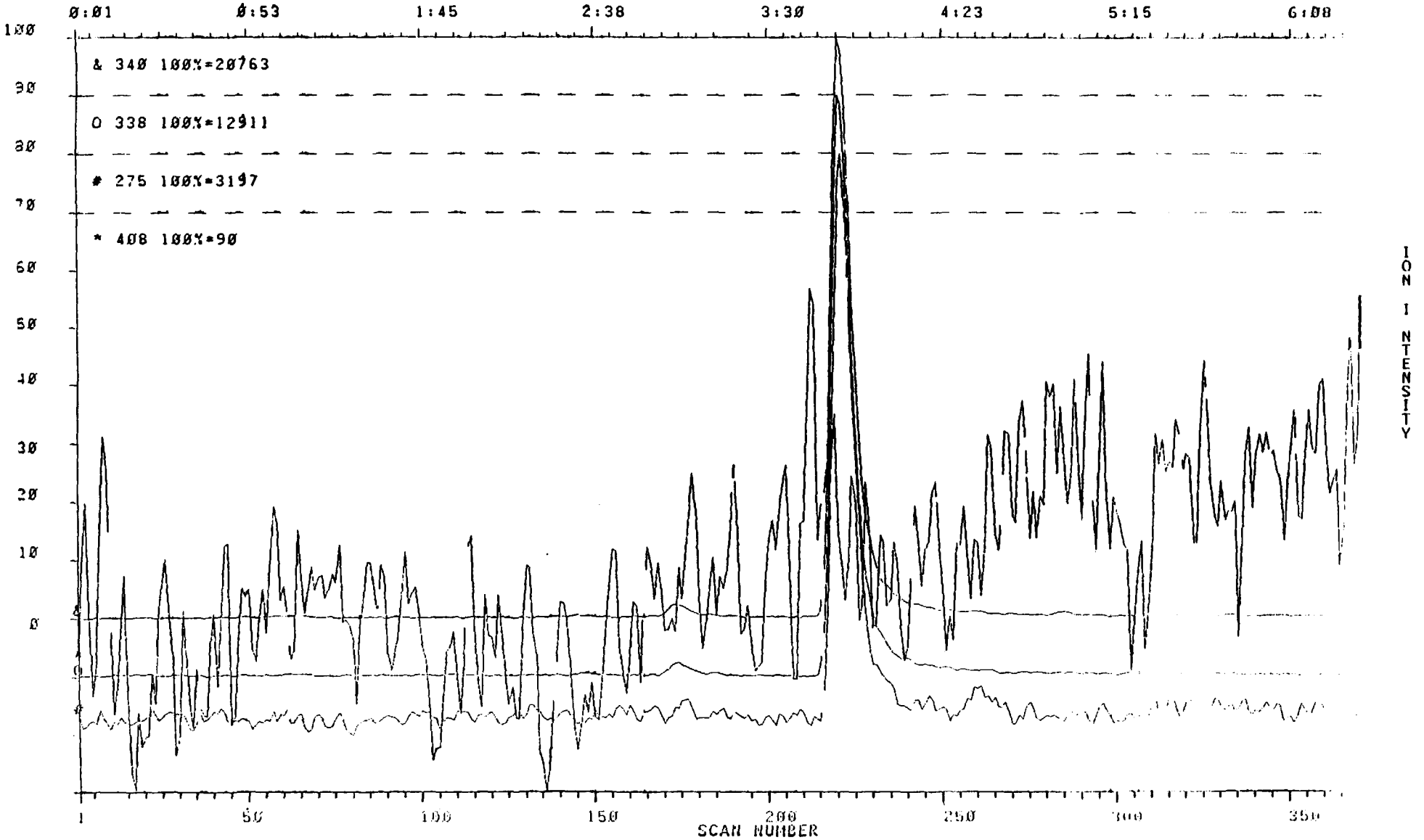
KRATOS MS25, DS55 SOFTWARE, RUN: CTO40108, WSU NAME: STD 1840-1
SELECTED-ION MASS CHROMATOGRAMS FOR TETRACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.STD 1840-1

FIGURE: 140

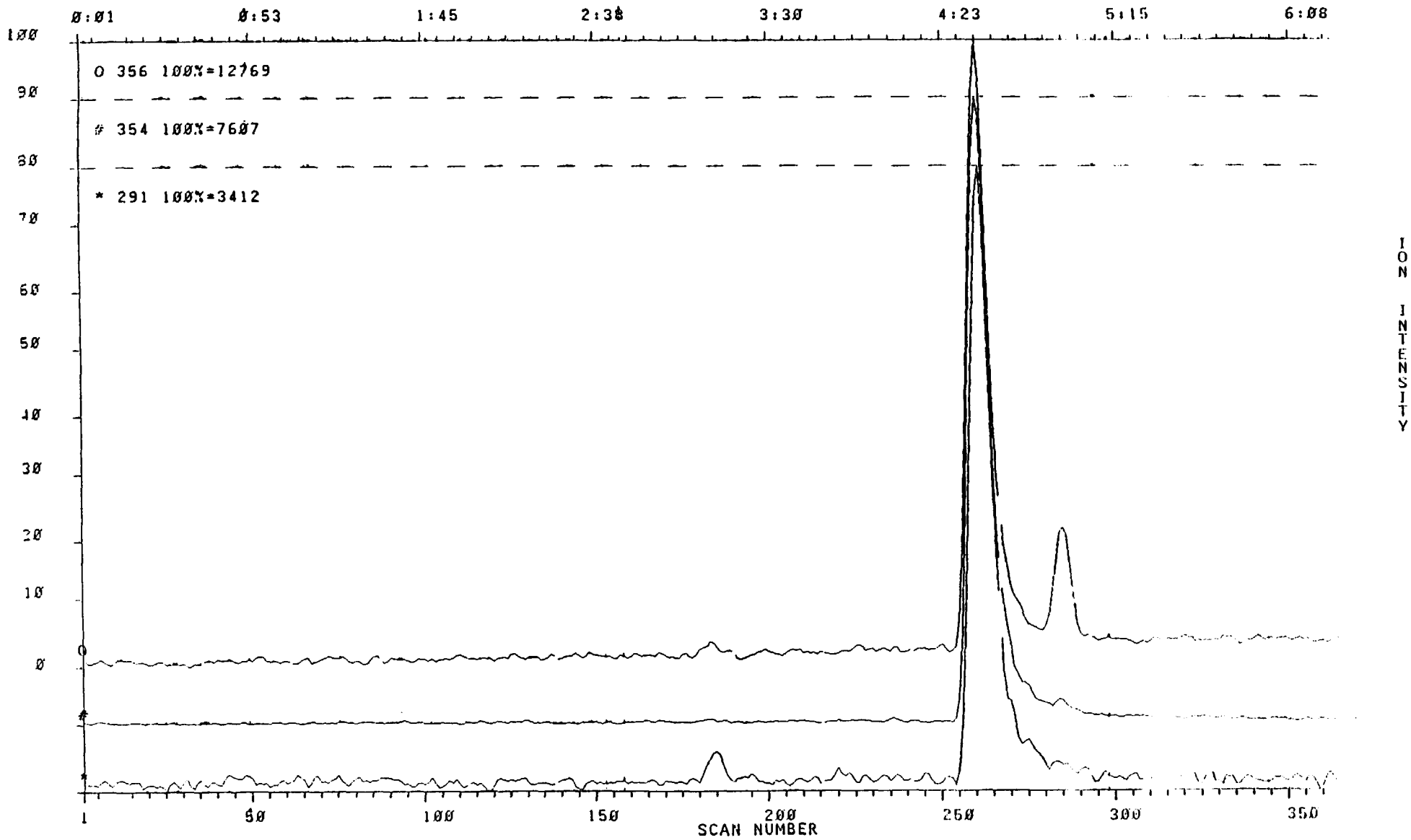
KRATOS MS25, DS55 SOFTWARE, RUN: CTO50108, WSU NAME: STD 1040-1
SELECTED-ION MASS CHROMATOGRAMS FOR PENTACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.STD 1040-1

FIGURE: 141

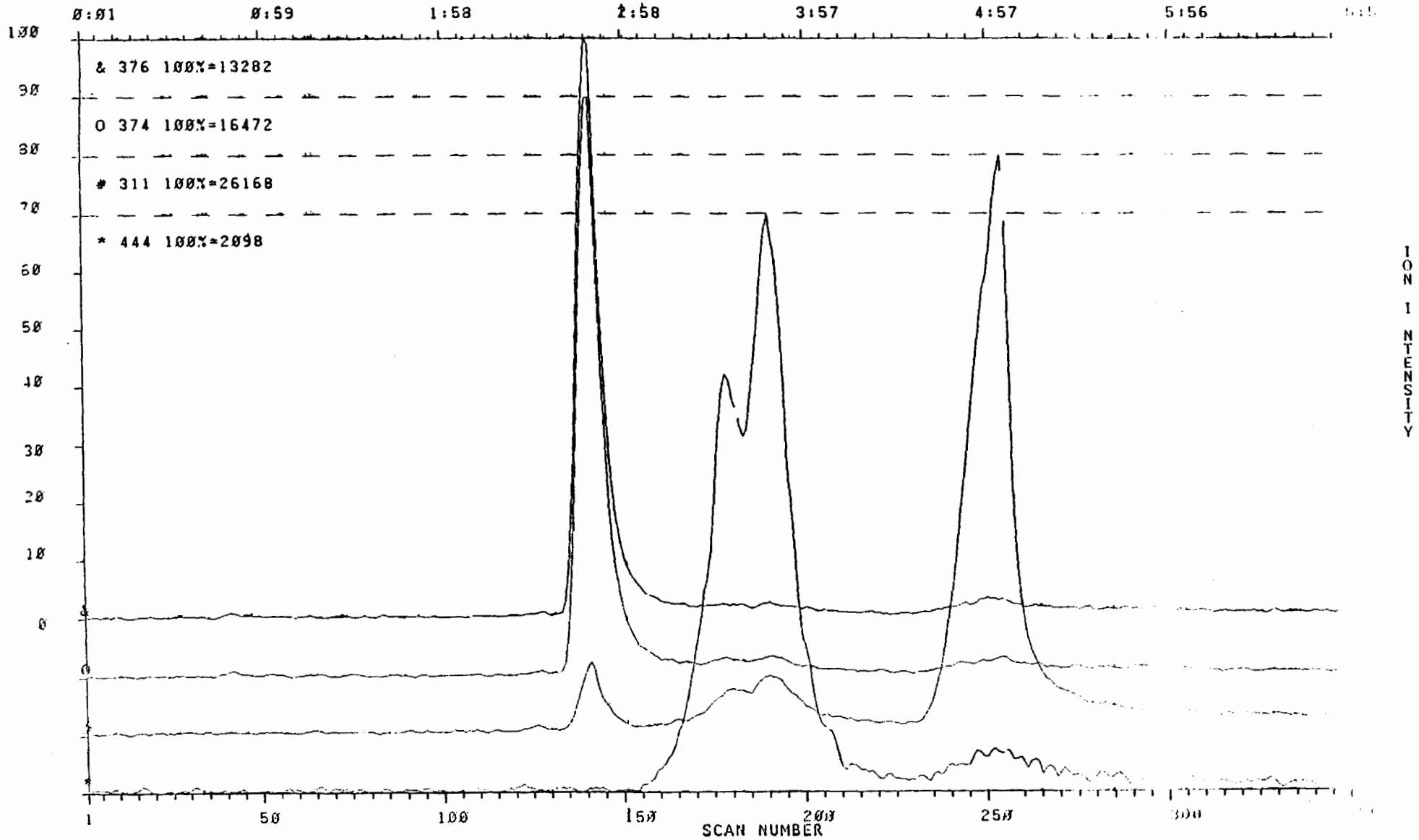
KRATOS MS25, DS55 SOFTWARE, RUN: CTO5Ø1ØØ, WSU NAME: STD 184Ø-1
SELECTED-ION MASS CHROMATOGRAMS FOR PENTACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.STD 184Ø-1

FIGURE: 142

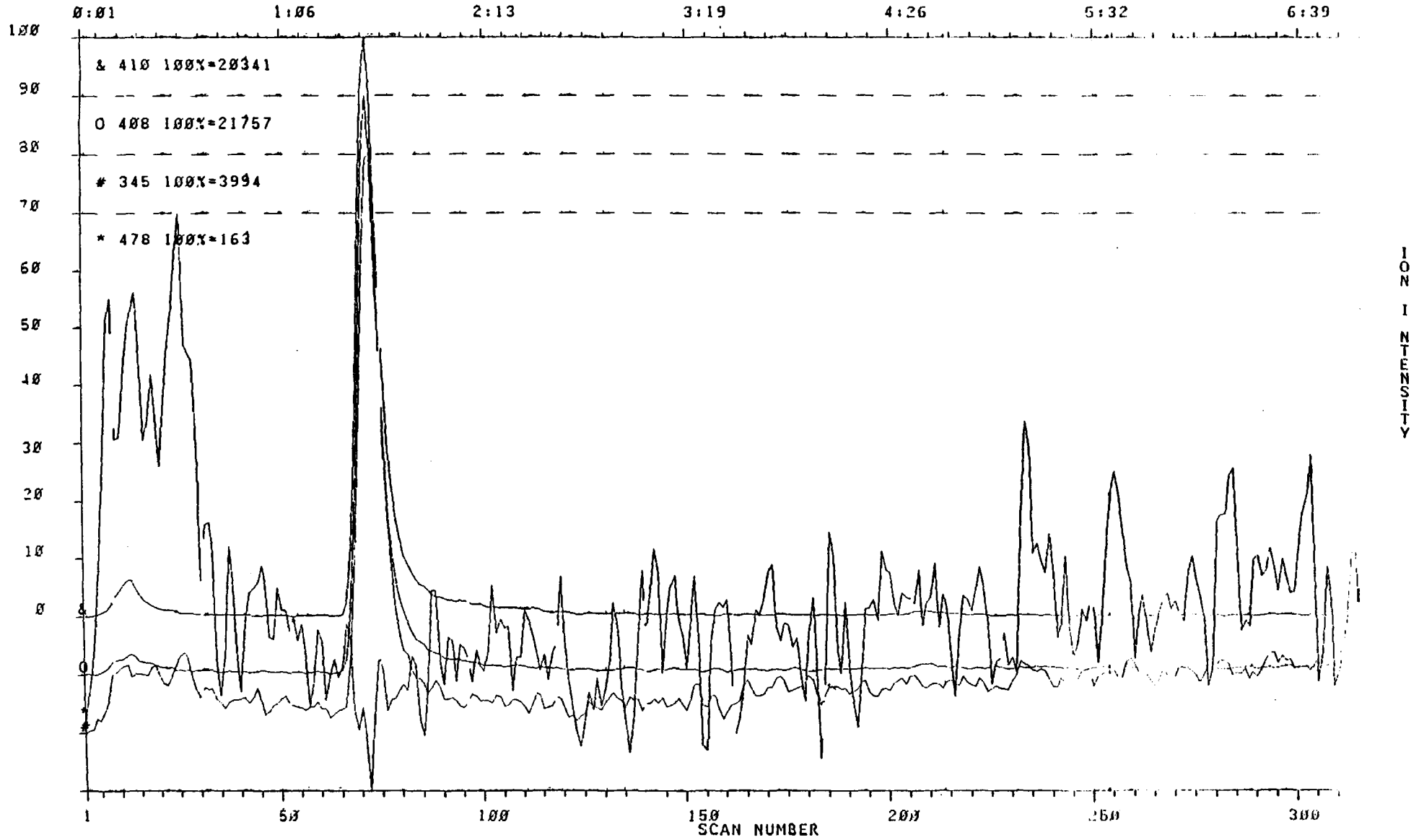
KRATOS MS25, DS55 SOFTWARE, RUN: CT060108, WSU NAME: STD 1840-1
SELECTED-ION MASS CHROMATOGRAMS FOR HEXACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.STD 1840-1

FIGURE: 143

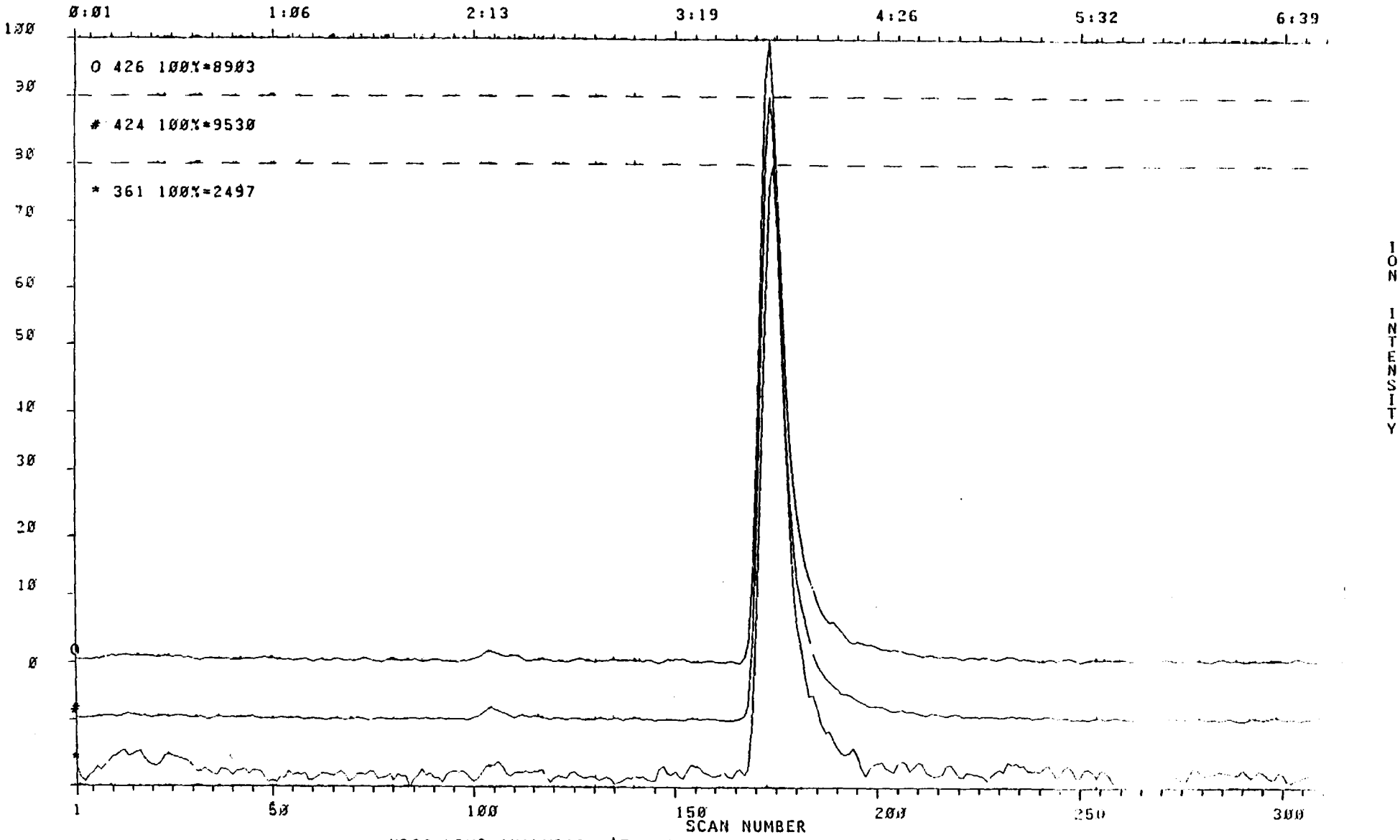
KRATOS MS25, DS55 SOFTWARE, RUN: CTO70108, WSU NAME: STD 1840-1
SELECTED-ION MASS CHROMATOGRAMS FOR HEPTACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.STD 1840-1

FIGURE: 144

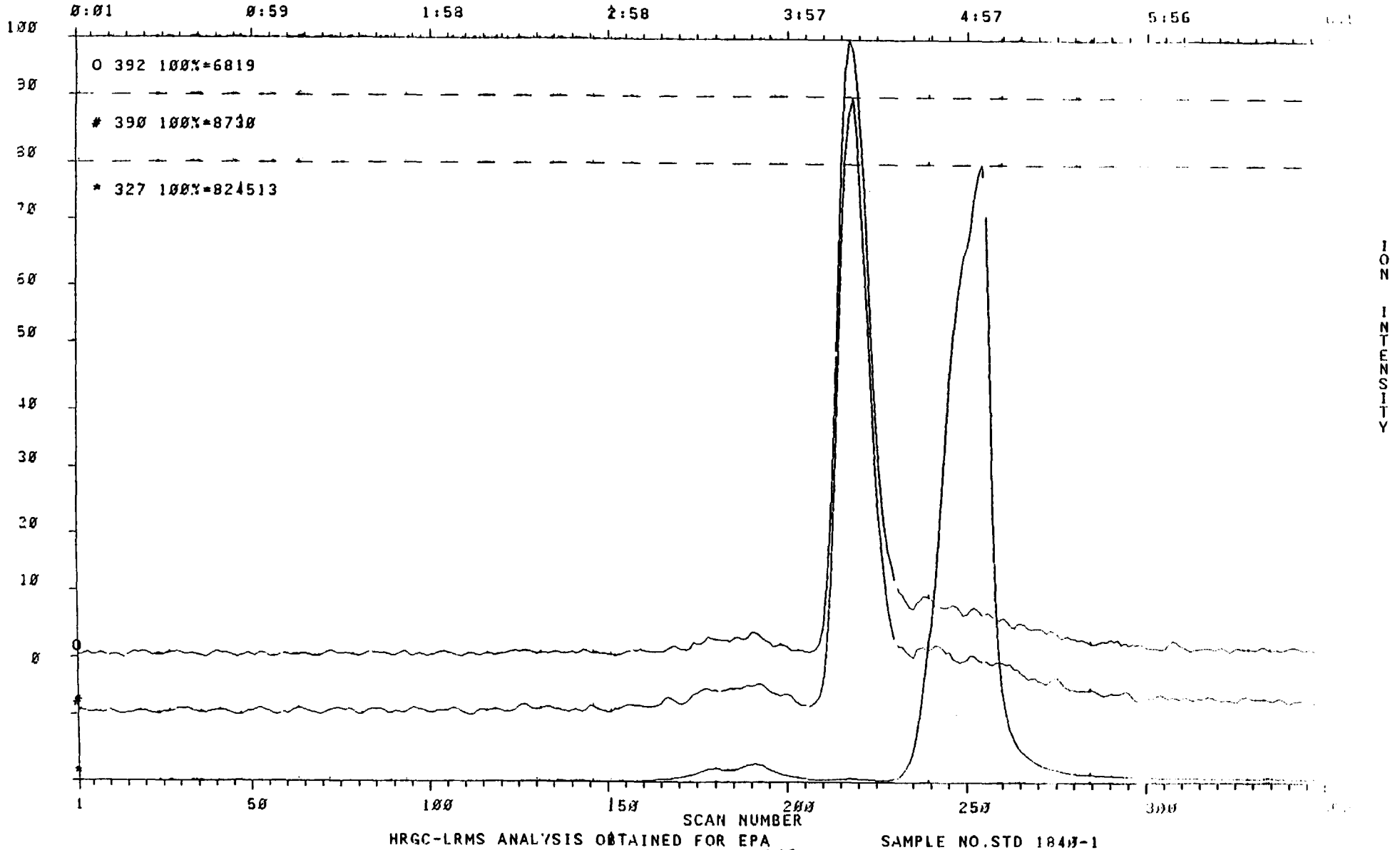
KRATOS MS25, DS55 SOFTWARE, RUN: CTO70108, WSU NAME: STD 1040-1
SELECTED-ION MASS CHROMATOGRAMS FOR HEPTACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.STD 1040-1

FIGURE: 145

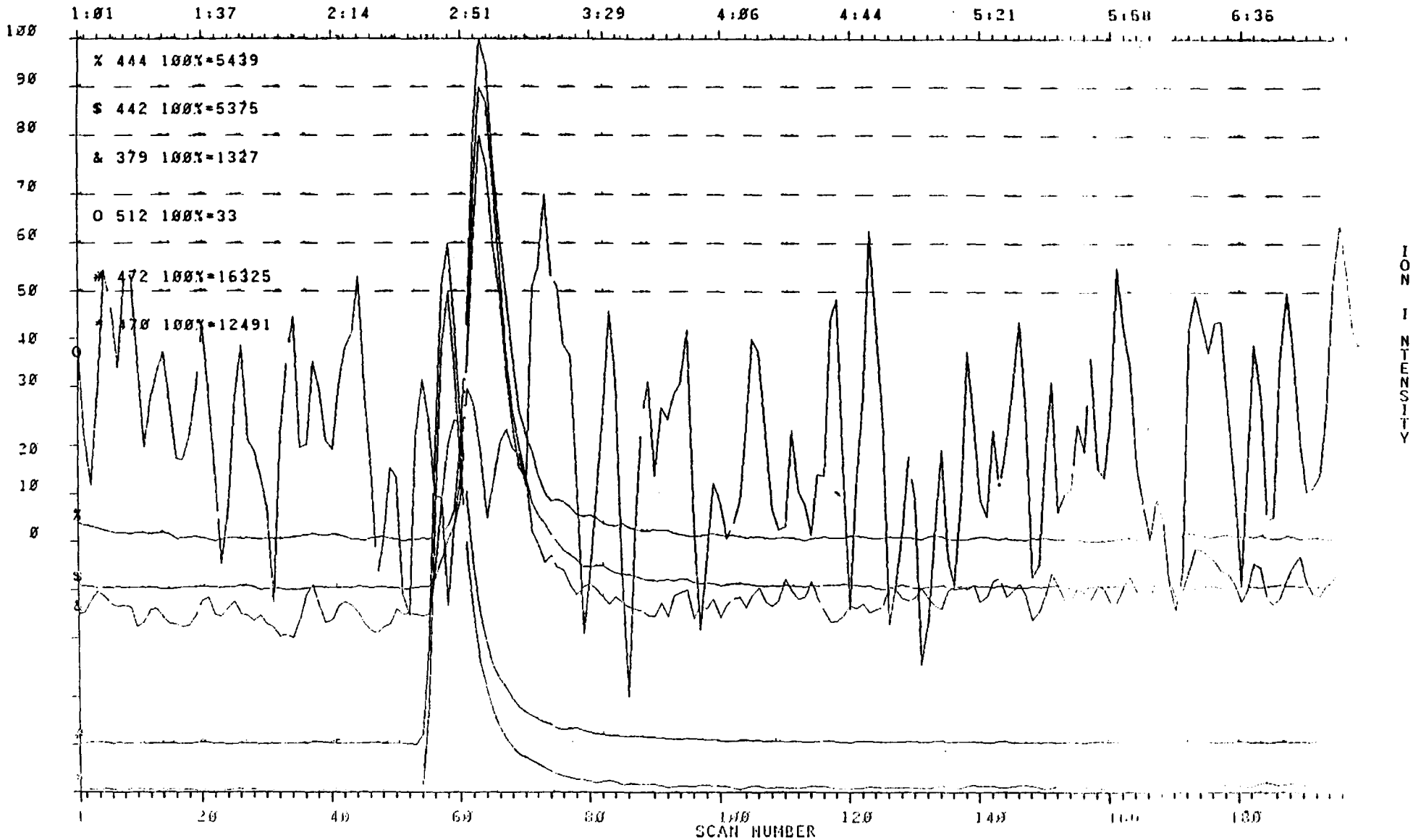
KRATOS MS25, DS55 SOFTWARE, RUN: CTO60108, WSU NAME: STD 1840-1
SELECTED-ION MASS CHROMATOGRAMS FOR HEXACHLORODIBENZO-P-DIOXINS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.STD 1840-1

FIGURE: 146

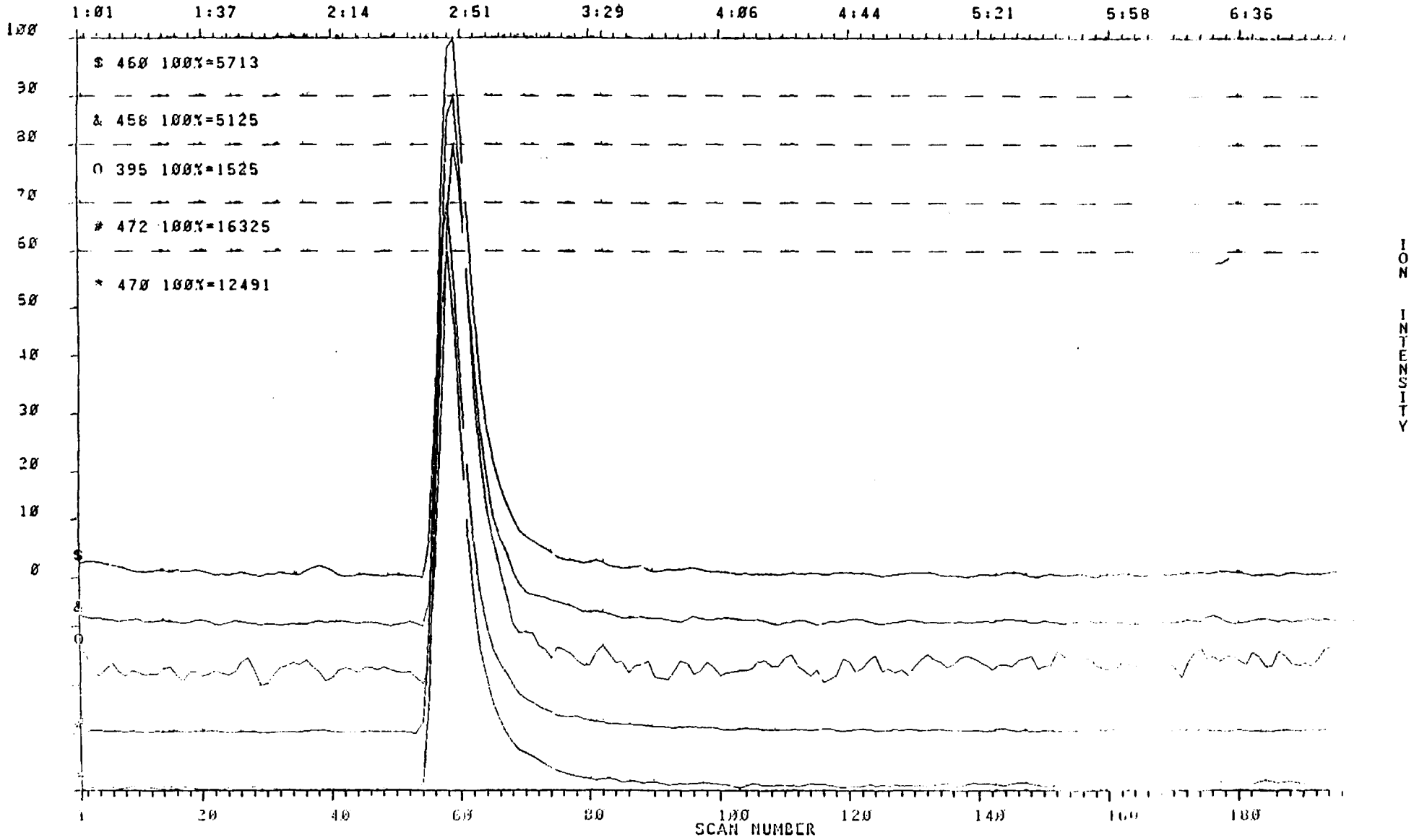
KRATOS MS25, DS55 SOFTWARE, RUN: CTO8Ø1Ø8, WSU NAME: STD 1840-1
SELECTED-ION MASS CHROMATOGRAMS FOR OCTACHLORODIBENZOFURANS



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.STD 1840-1

FIGURE: 147

KRATOS MS25, DS55 SOFTWARE, RUN: CTO80108, WSU NAME: STD 1840-1
SELECTED-ION MASS CHROMATOGRAMS FOR OCTACHLORODIBENZO-P-DIOXINS



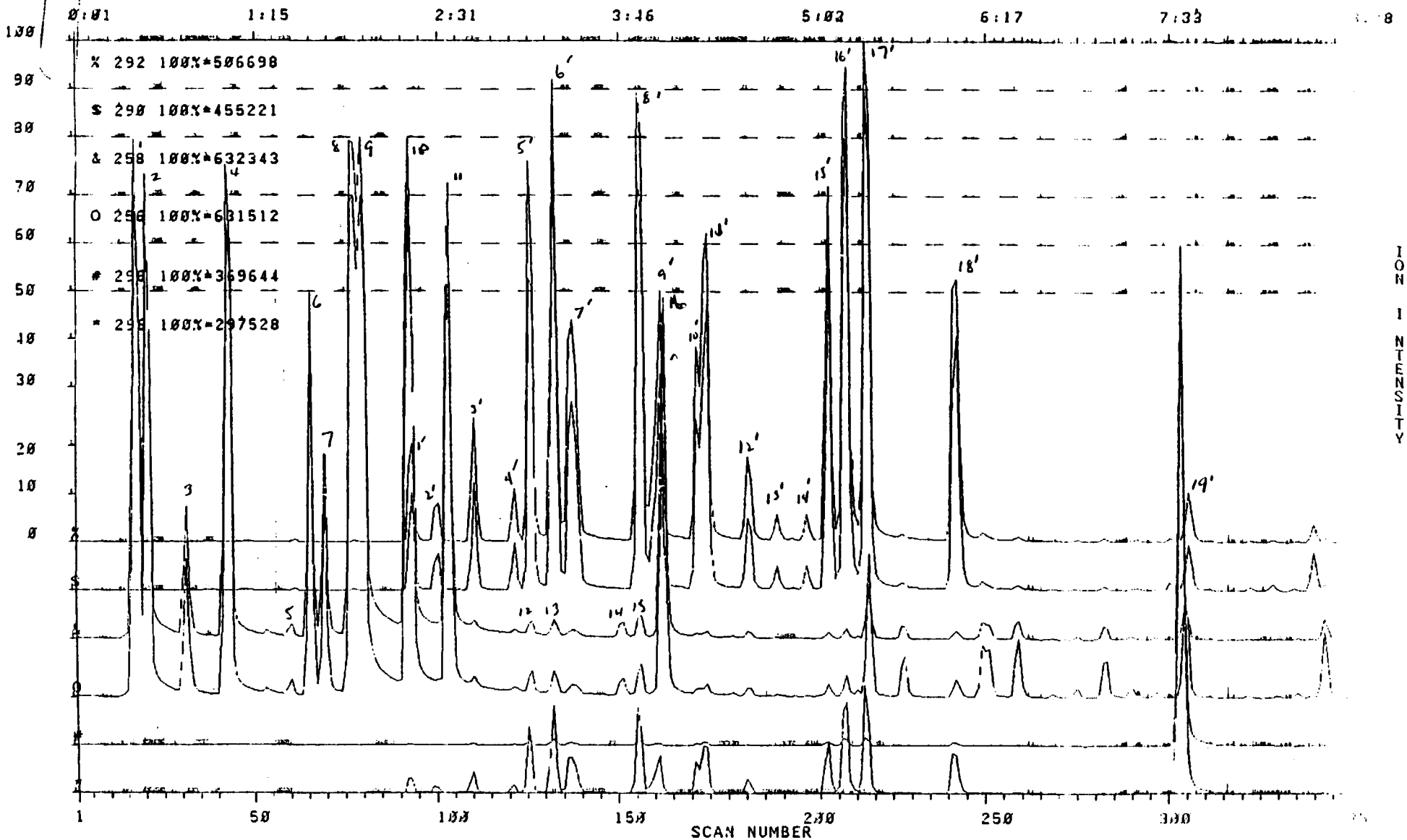
HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.STD 1840-1

FIGURE: 148

ATTACHMENT I

MASS CHROMATOGRAMS OBTAINED BY THE BREHM LABORATORY
IN GC-MS ANALYSES OF U.S. COAST GUARD SAMPLE EXTRACTS
AND OF SEDIMENTS RECEIVED FROM NUS CORP. FOR PCB

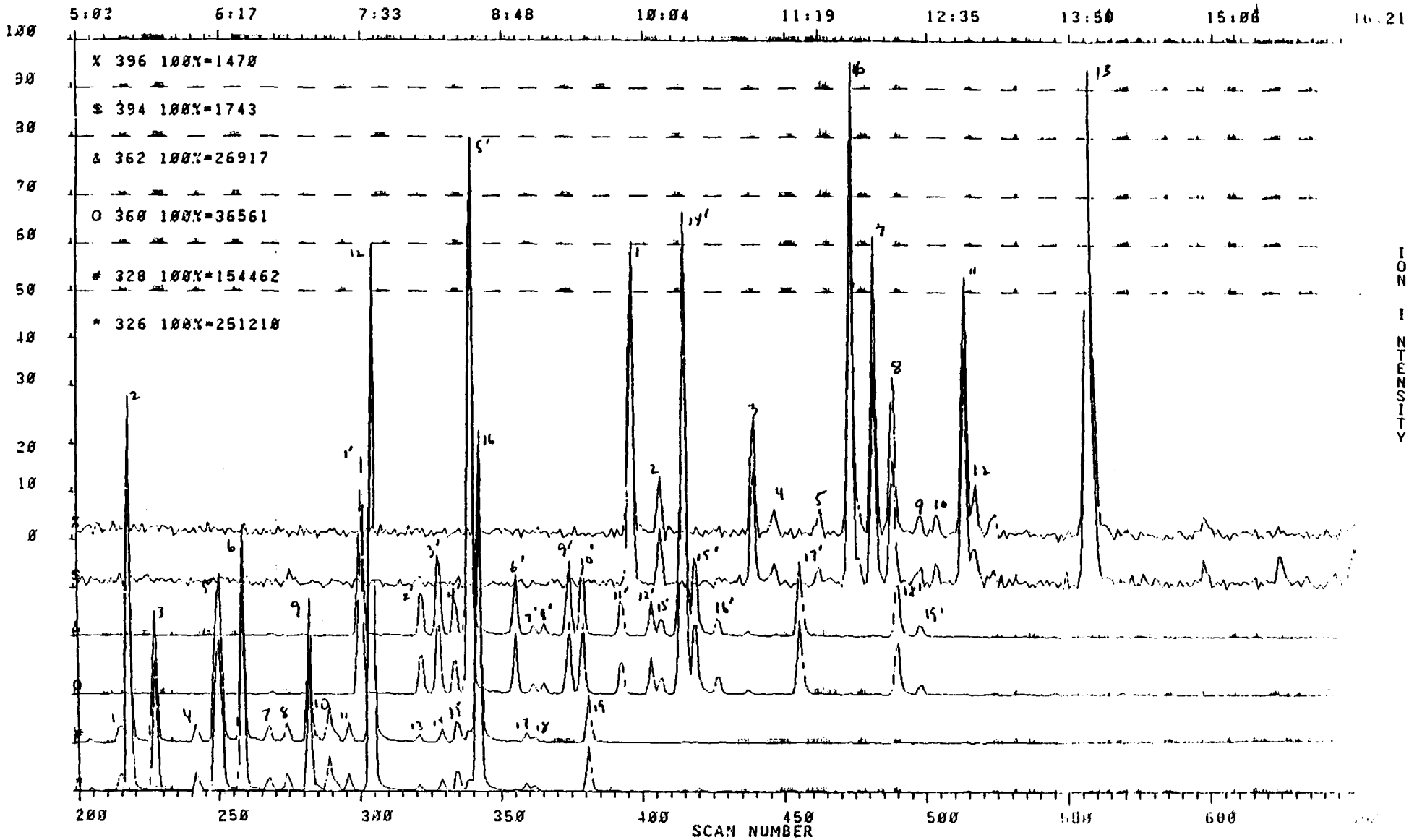
KRATOS MS25, DS55 SOFTWARE, RUN: PCBM0007, WSU NAME: USC6-1



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 27

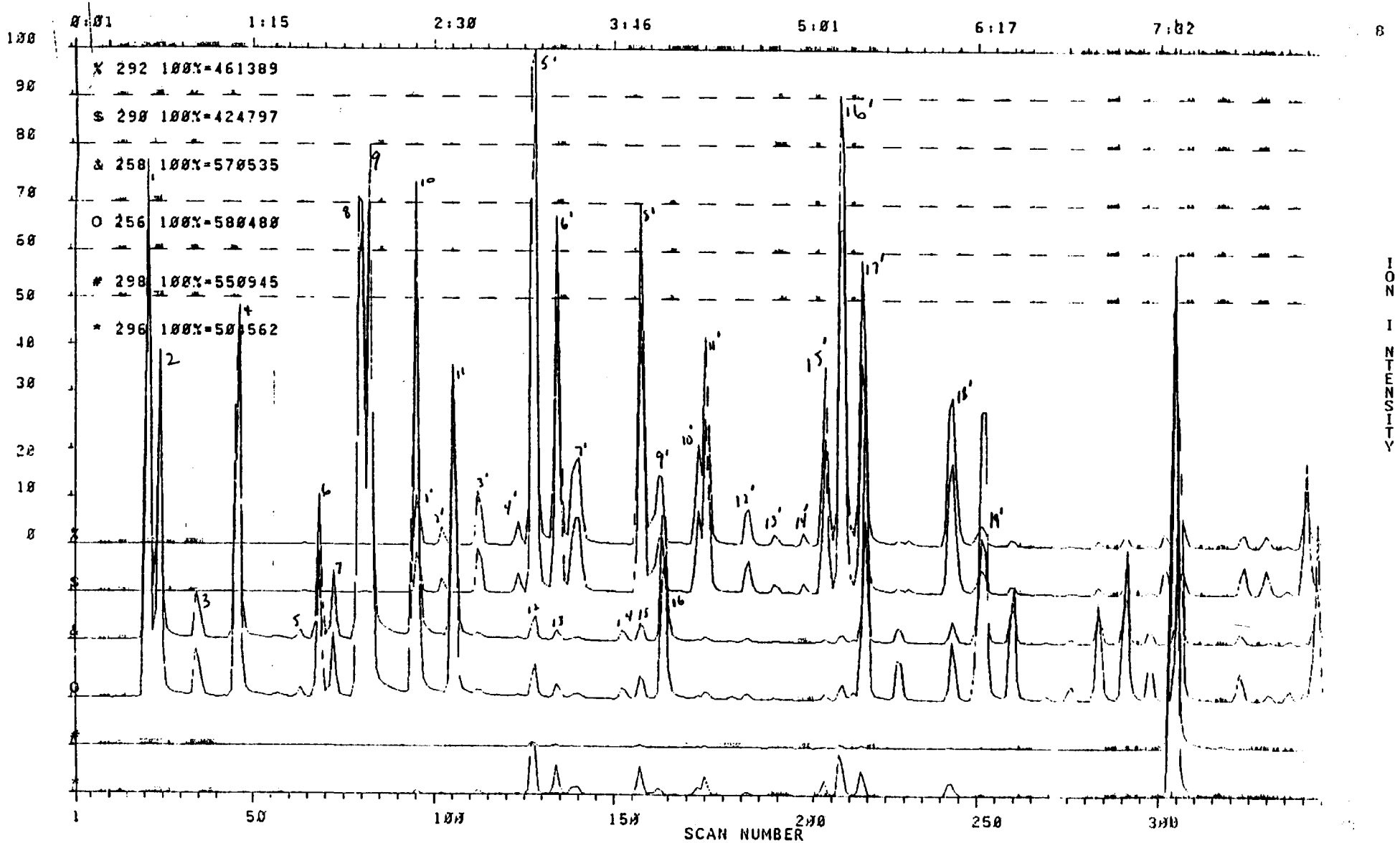
FIGURE: 149

KRATOS MS25, DS55 SOFTWARE, RUN: PCBM0007, WSU NAME: USC6-1



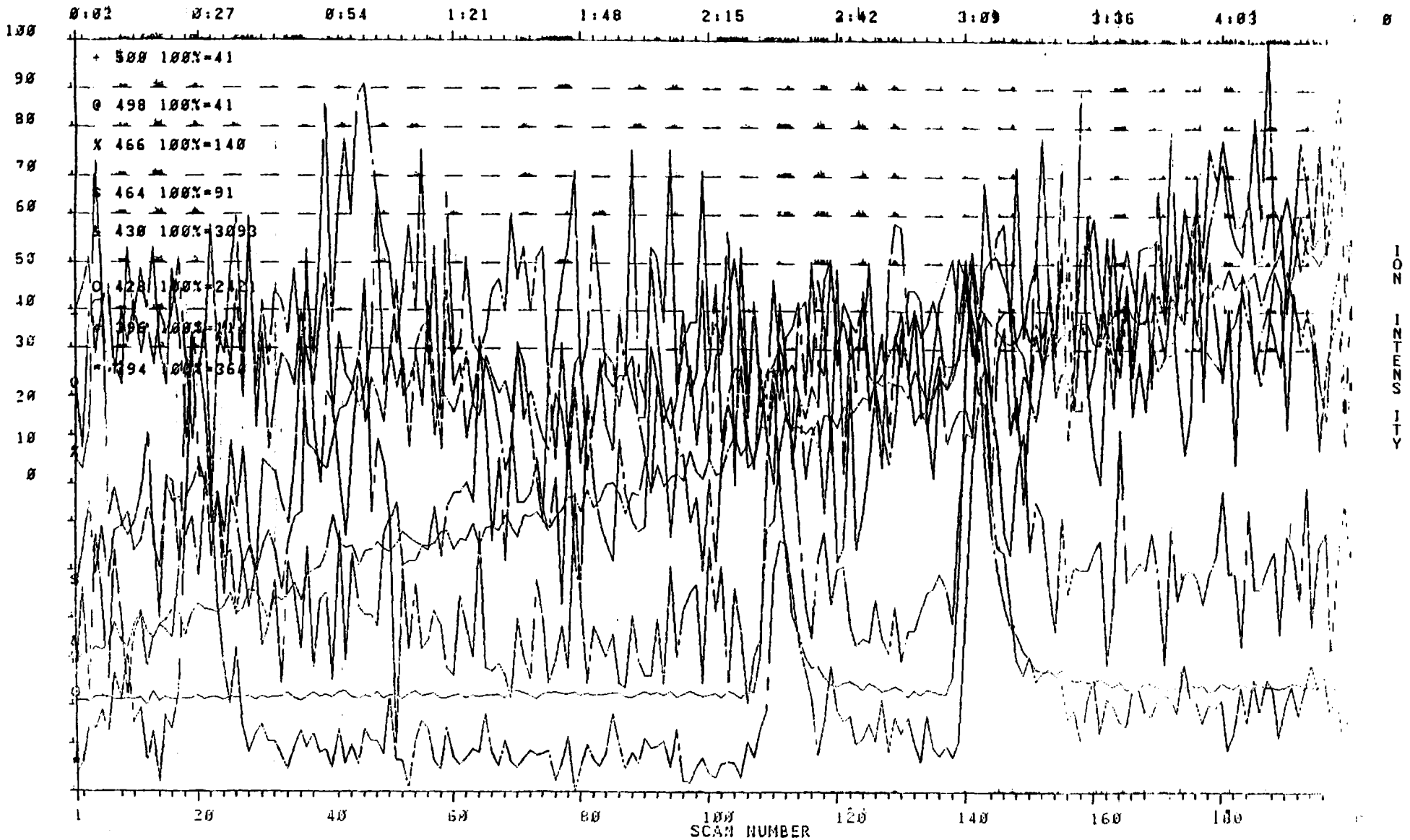
HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 27

FIGURE: 150



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 9 (B,C)

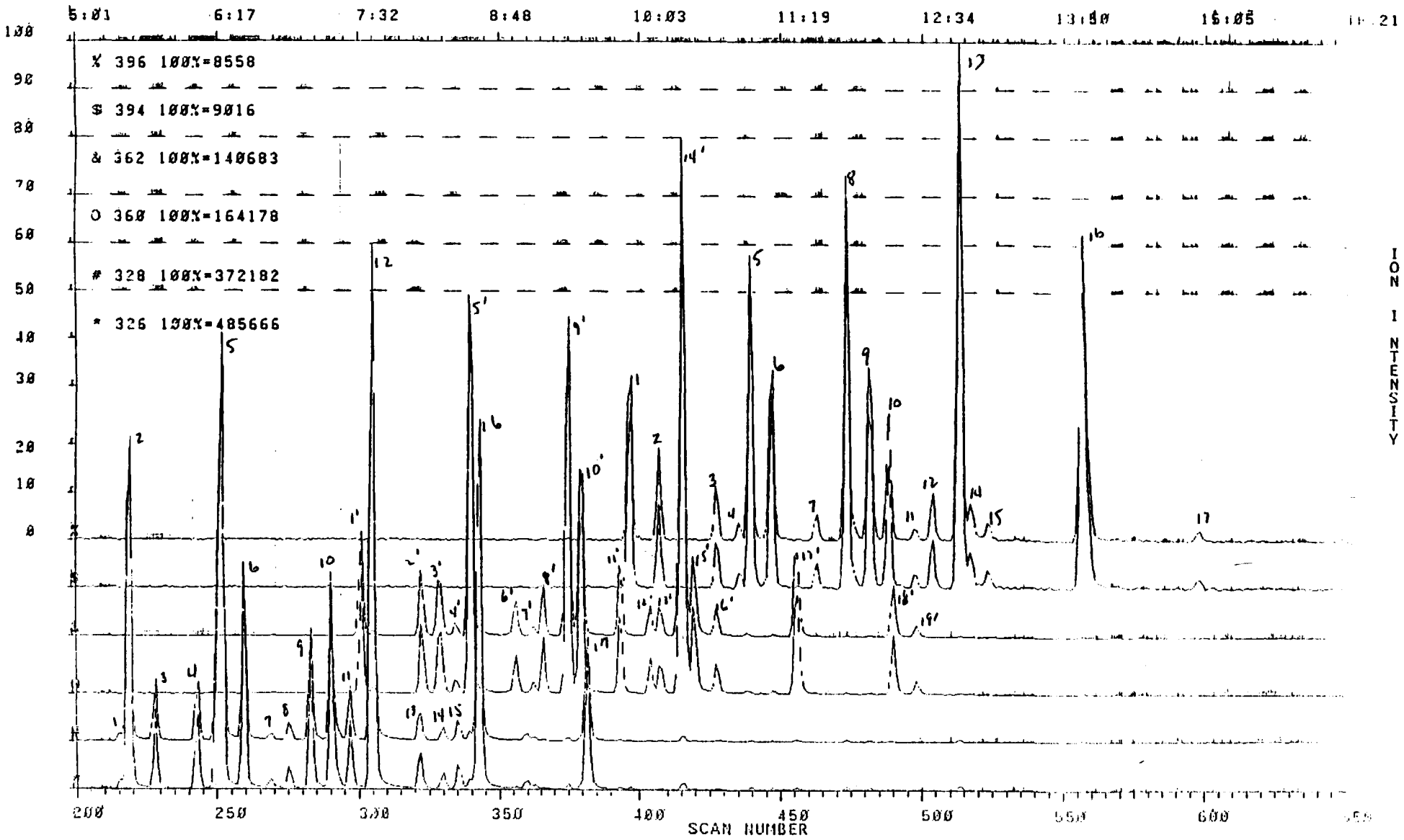
FIGURE: 152



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 27

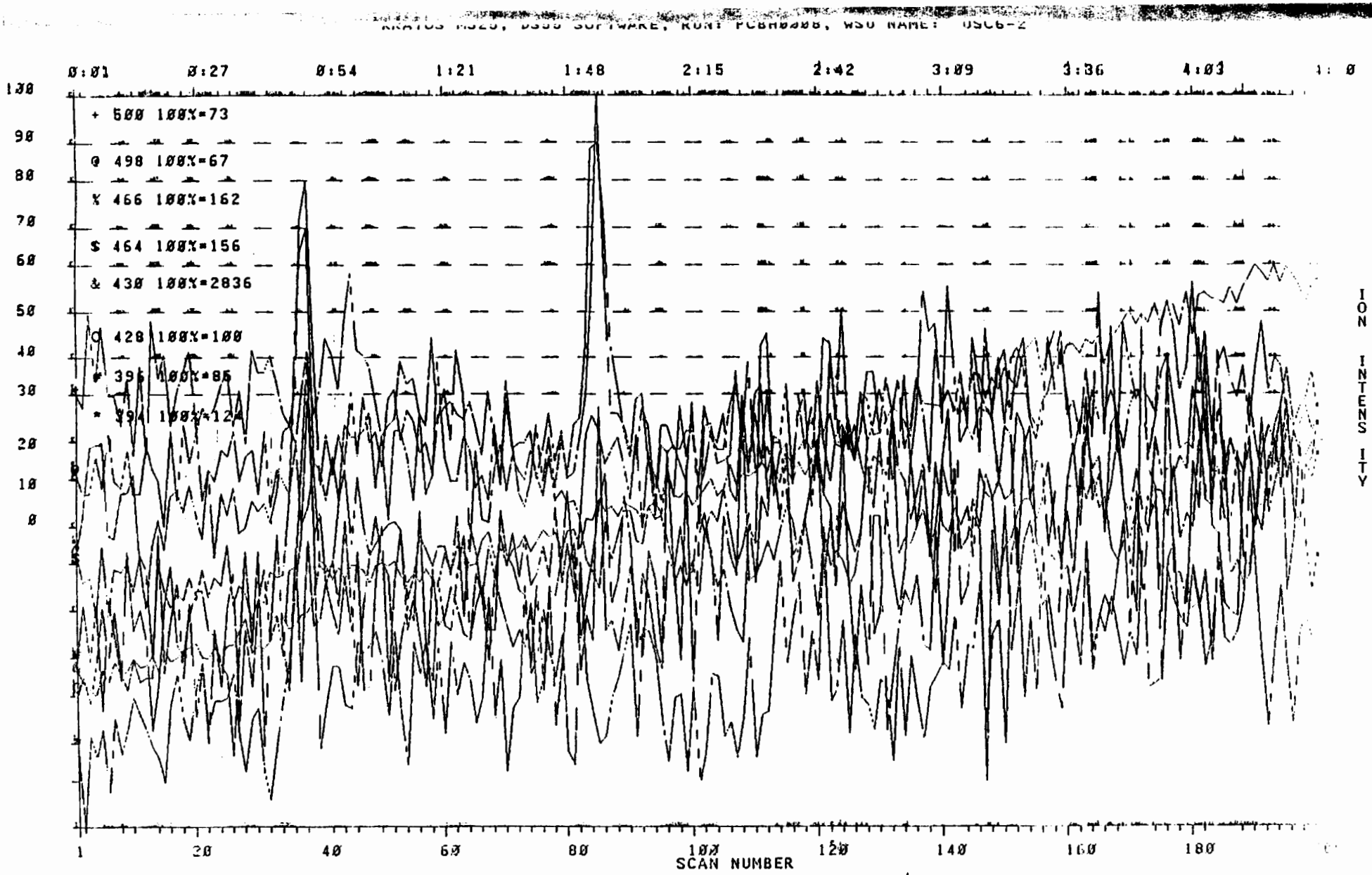
FIGURE: 151

KRATOS MS25, DS55 SOFTWARE, RUN: PCBM0006, WSU NAME: USC6-2



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. $ (B,C)

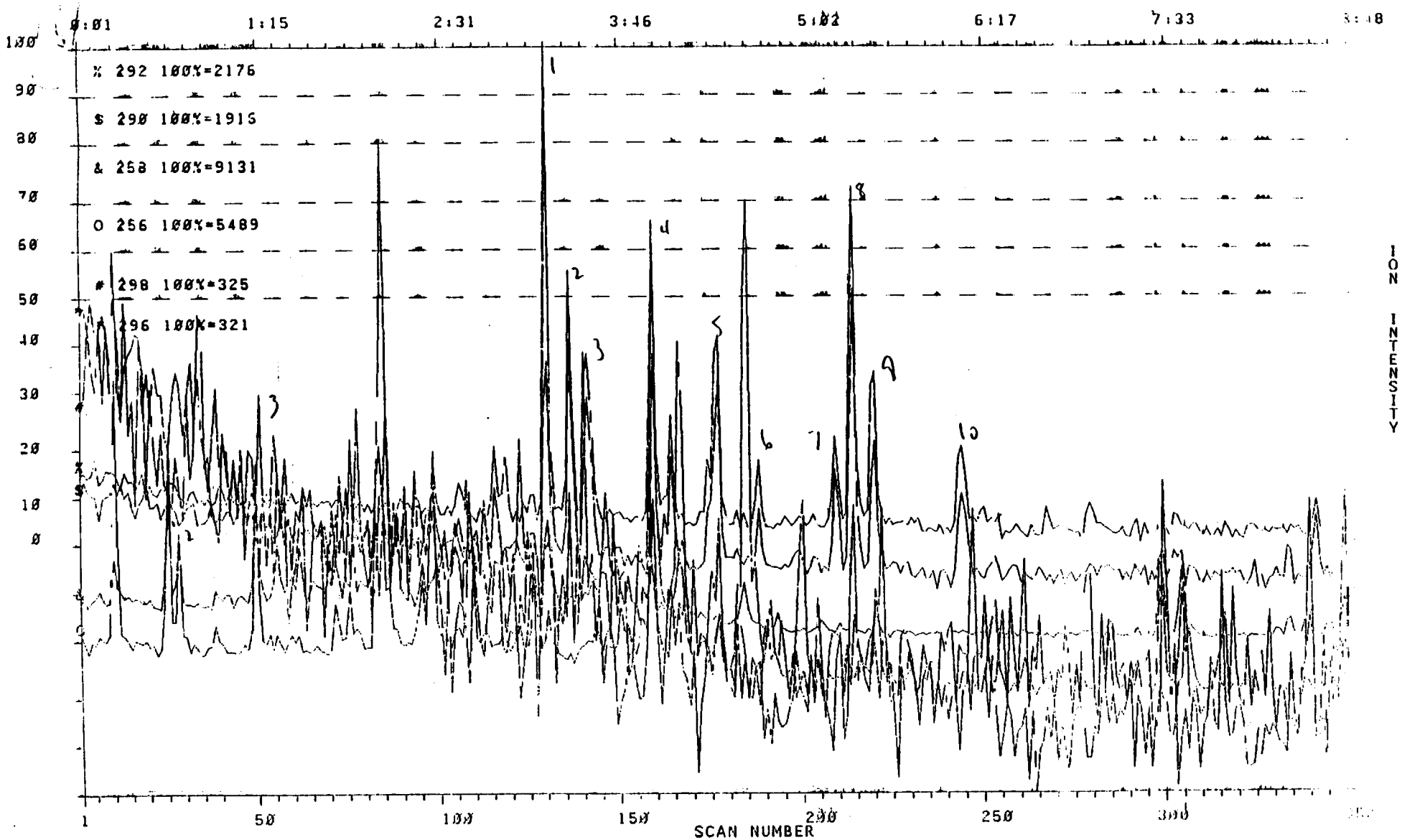
FIGURE: 153



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 9 (B.C)

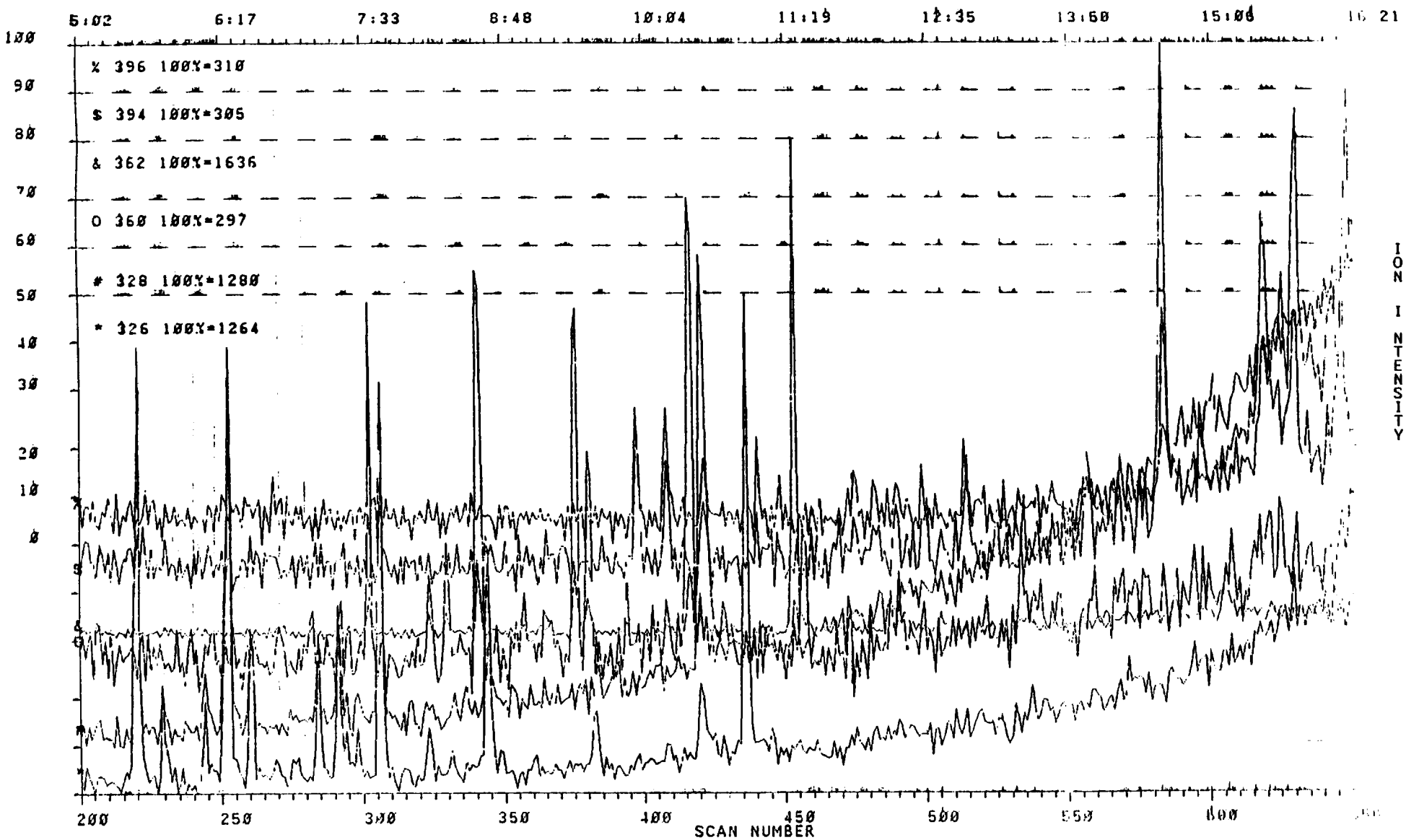
FIGURE: 154

KRATOS MS25, DS55 SOFTWARE, RUN: PCBM0004, WSU NAME: LAB BLANK



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.USC6-0-B

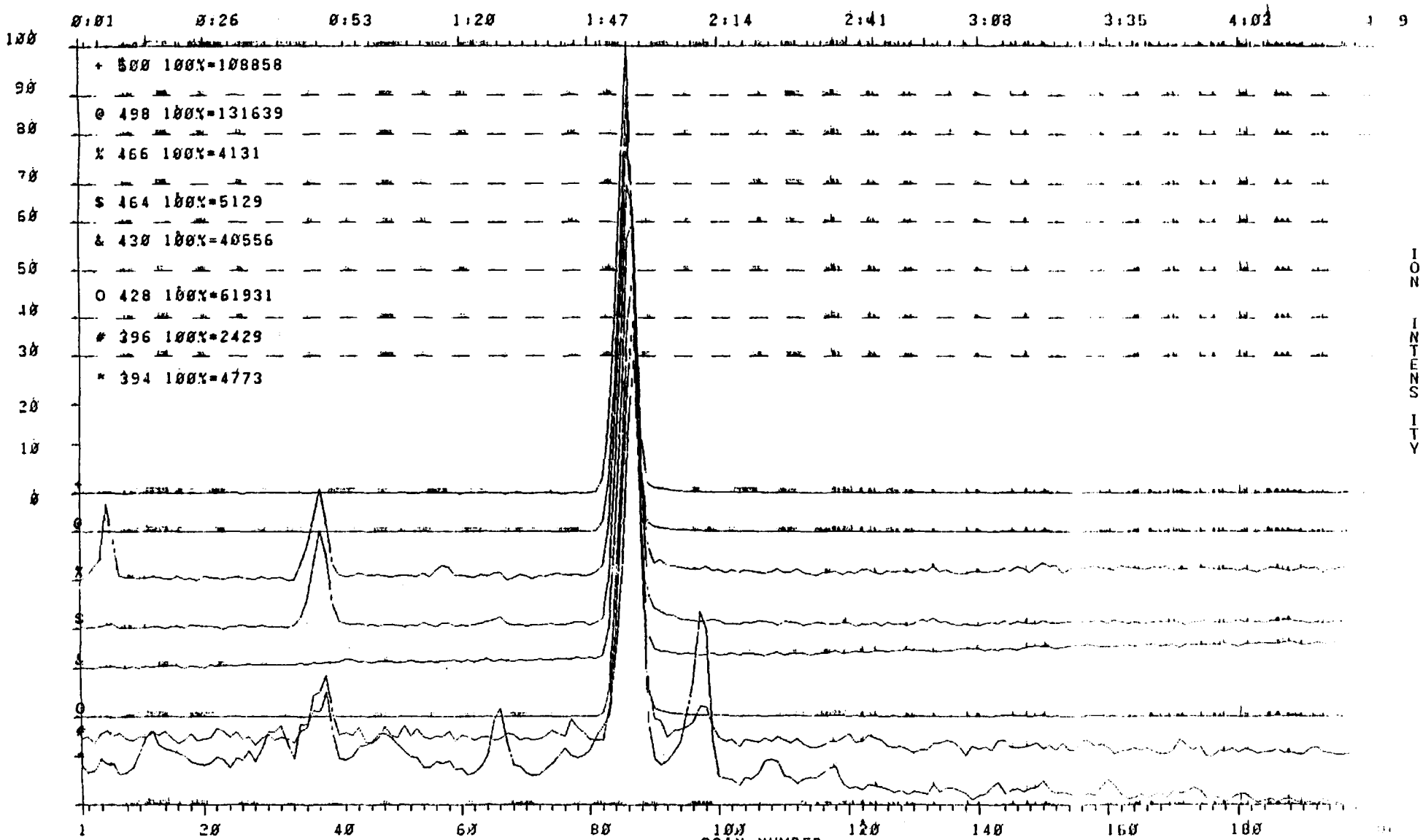
FIGURE: 155



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.USG6-D-B

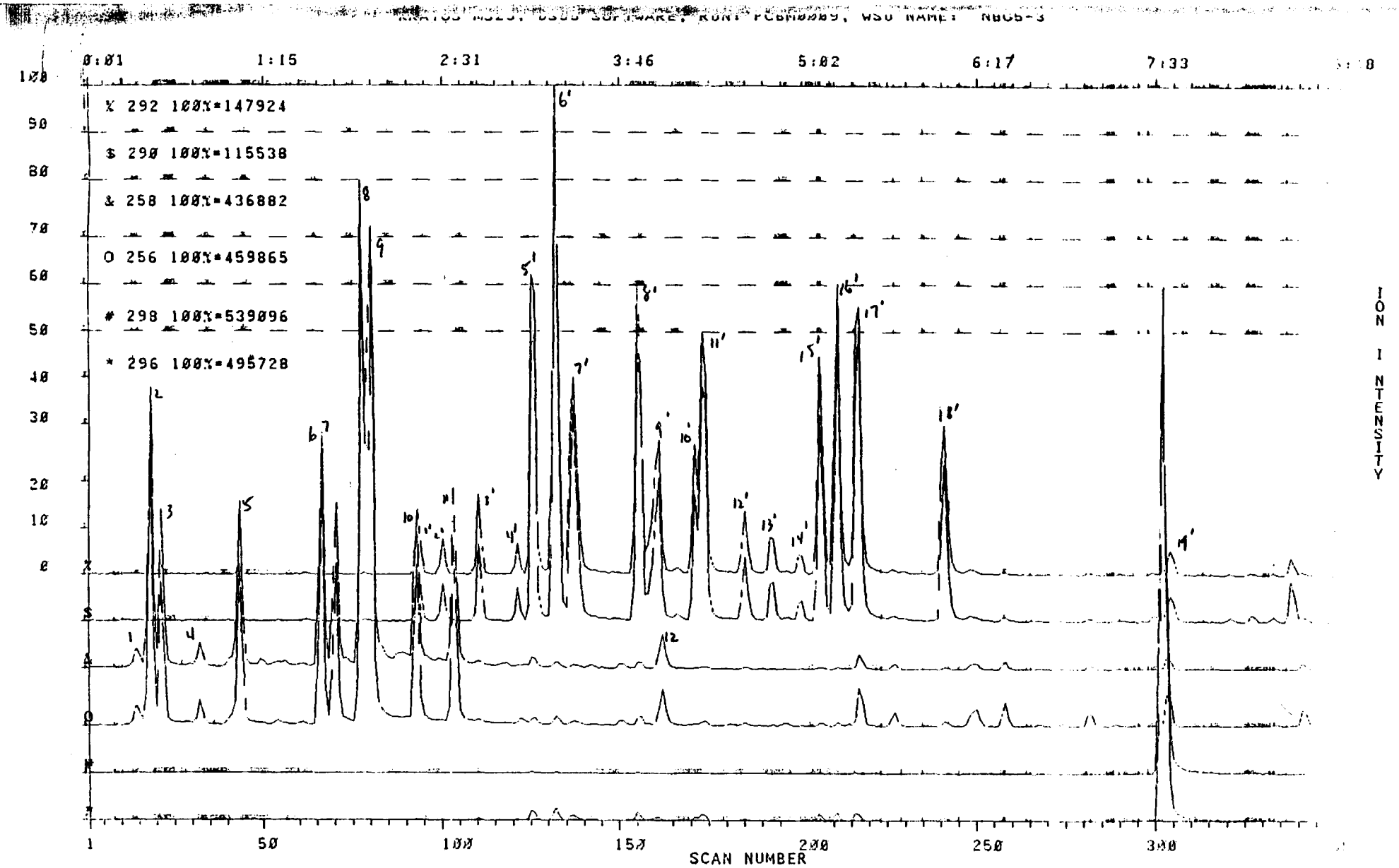
FIGURE: 156

KRATOS MS25, DS55 SOFTWARE, RUN: PCBH0004, WSU NAME: LAB BLANK



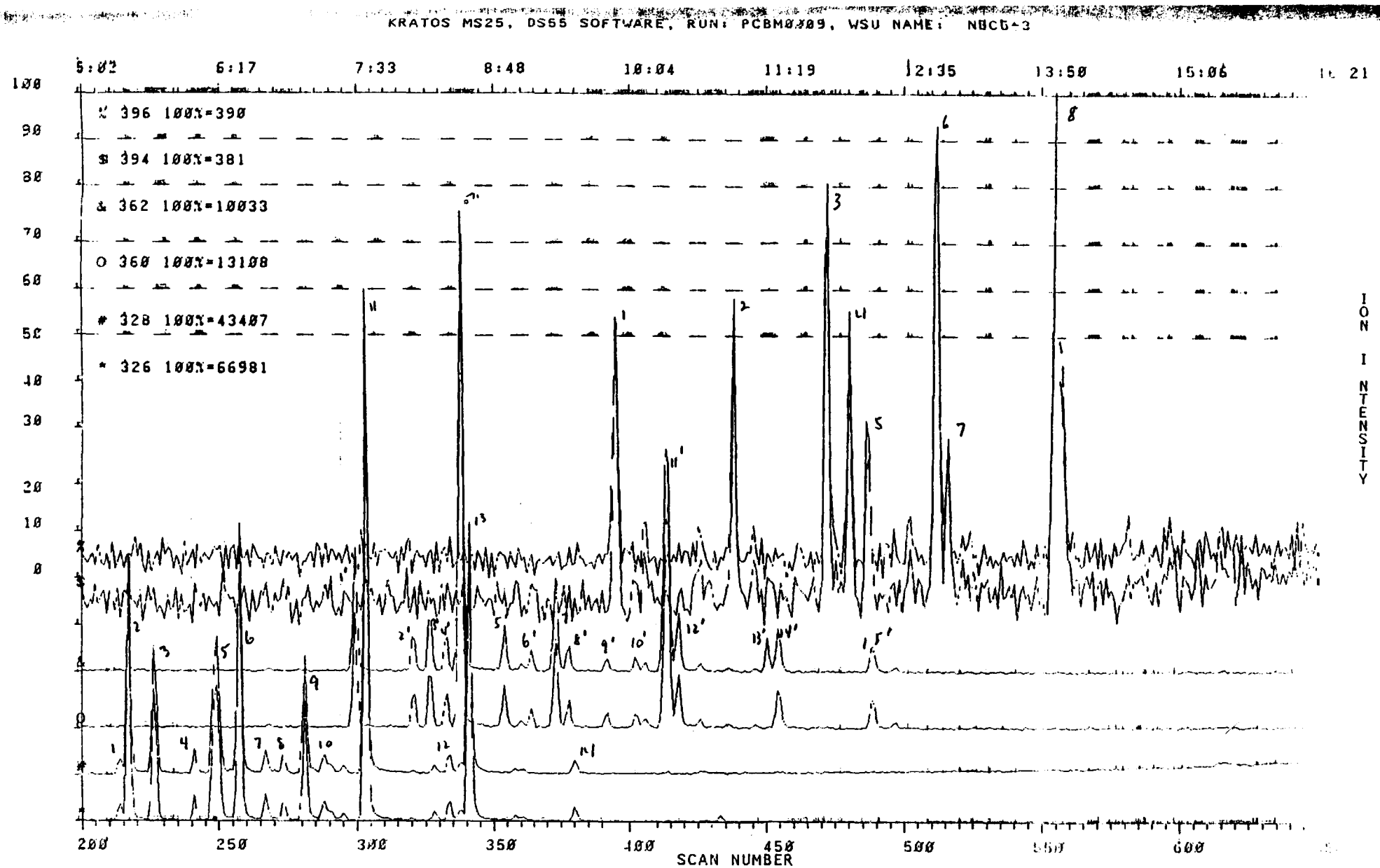
HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.USC6-[illegible]-B

FIGURE: 157



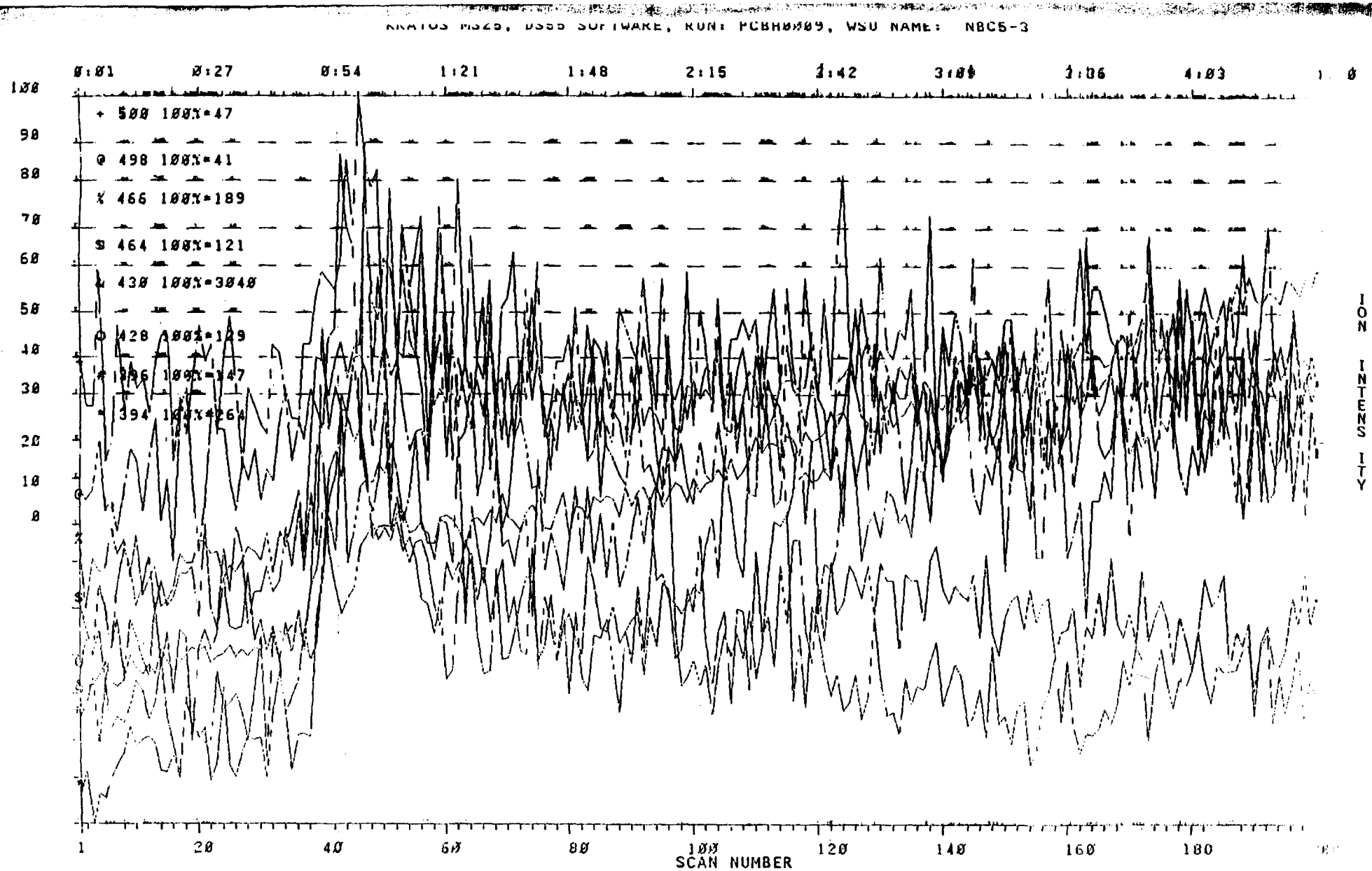
HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 13855

FIGURE: 158



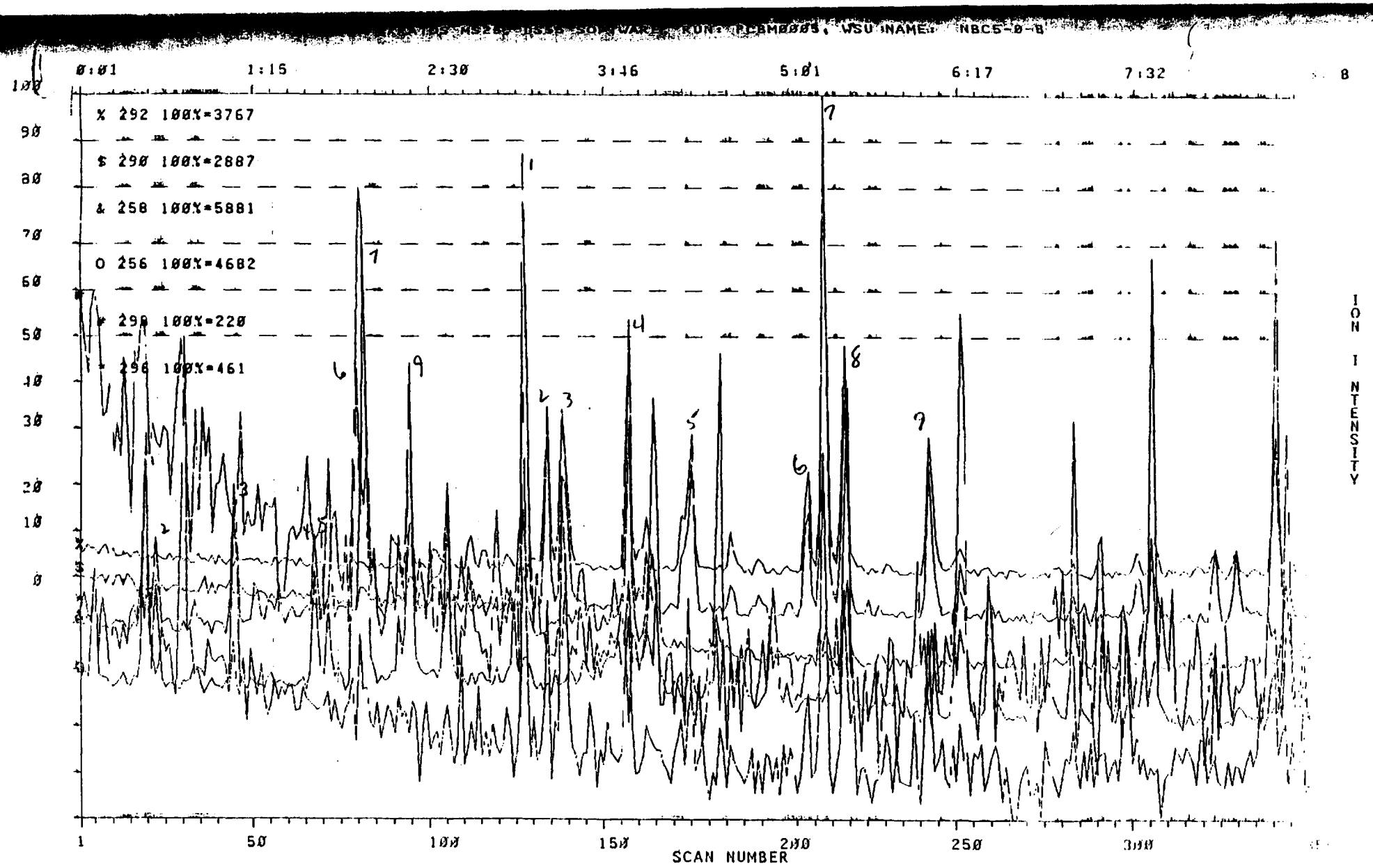
HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 13855

FIGURE: 159



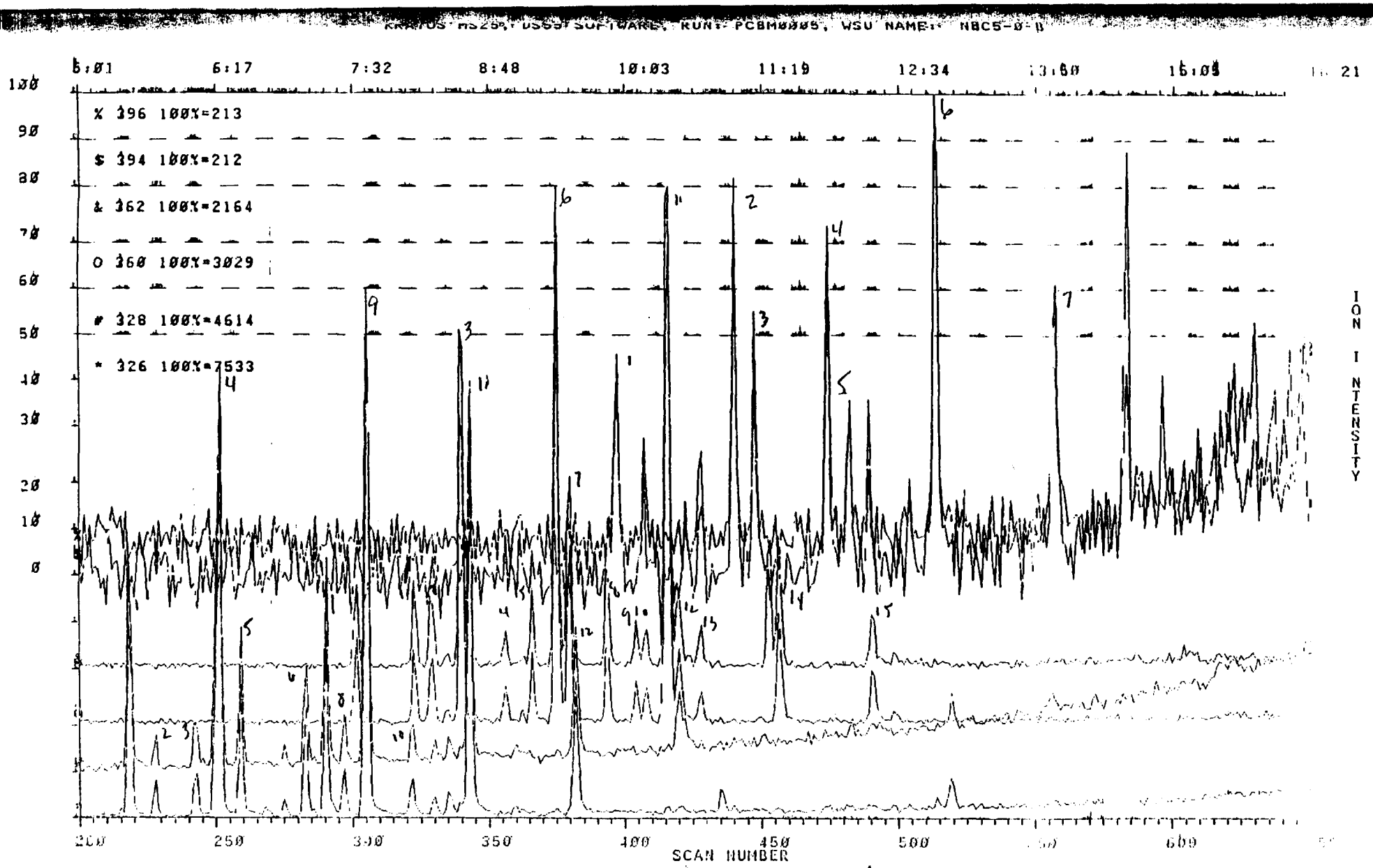
HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 13855

FIGURE: 160



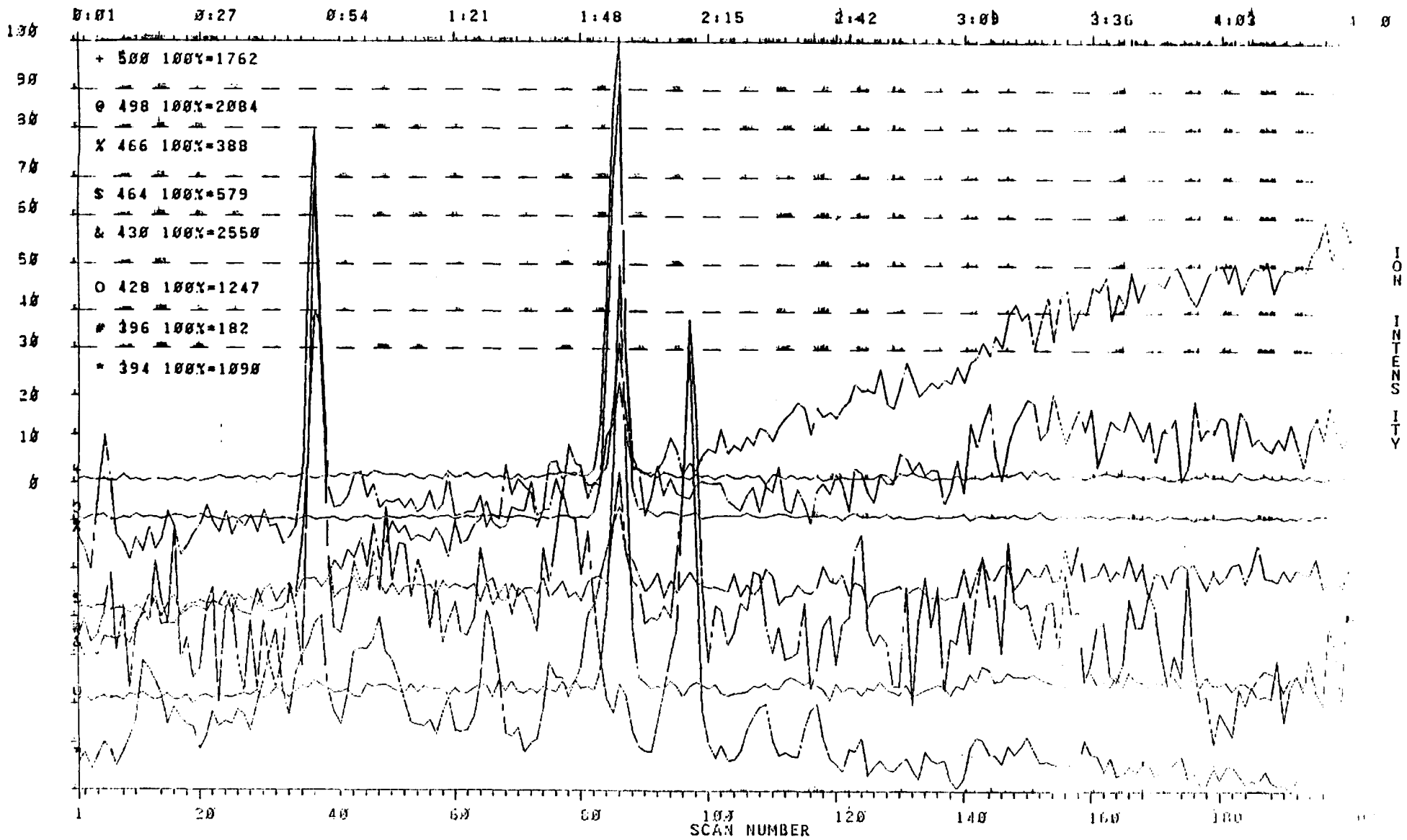
HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

FIGURE: 161



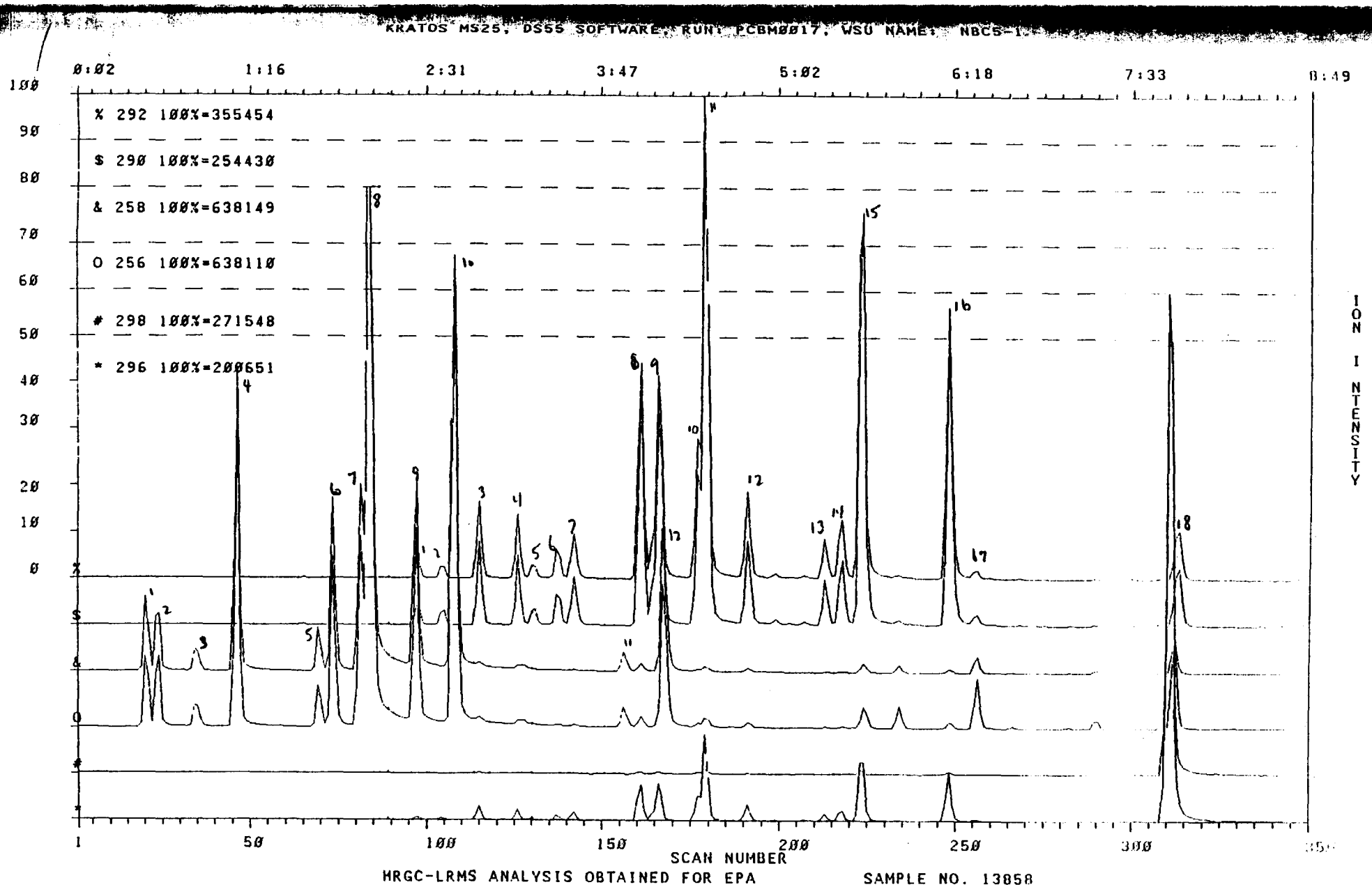
HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

FIGURE: 162



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. LAB BLANK

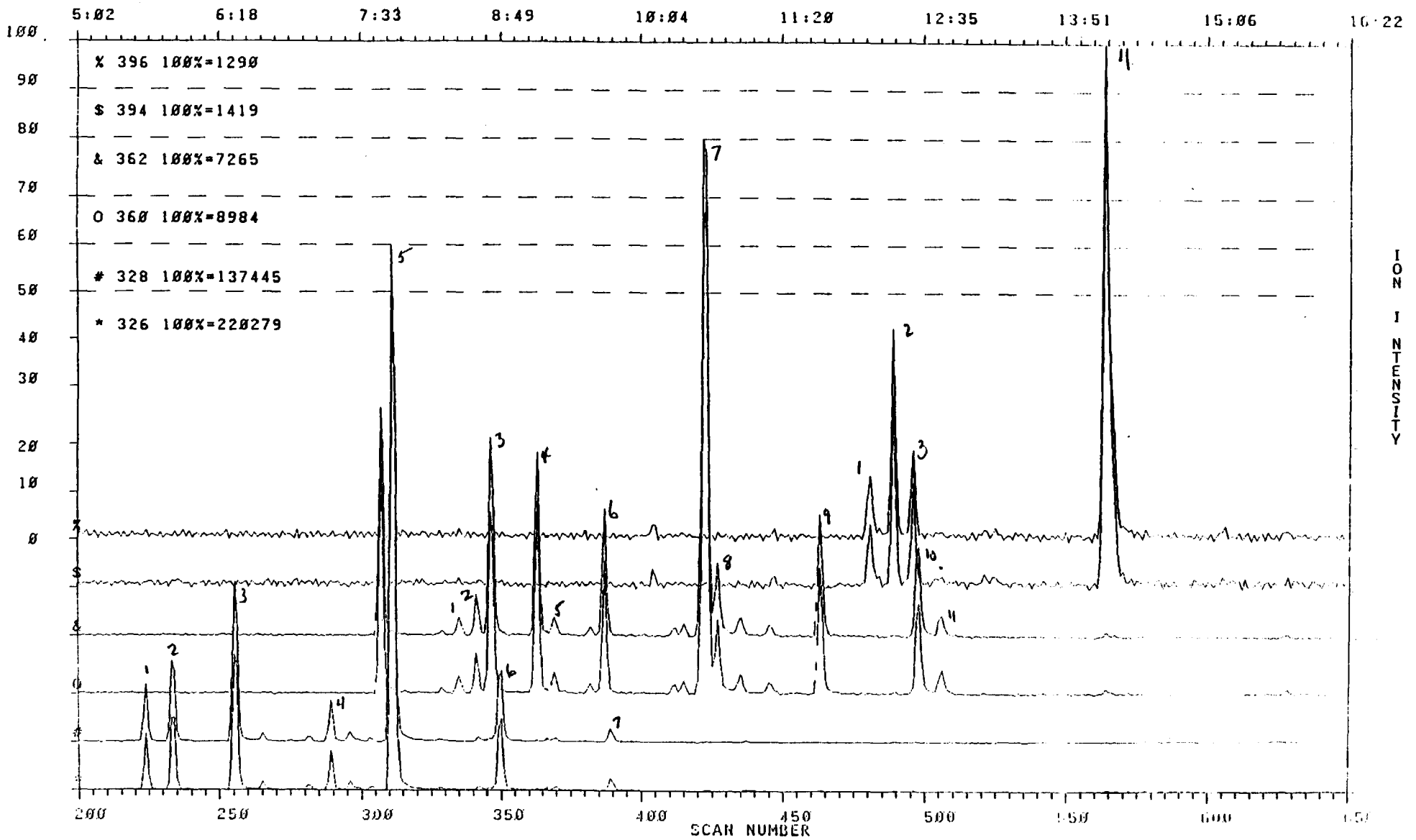
FIGURE: 163



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 13858

FIGURE: 164

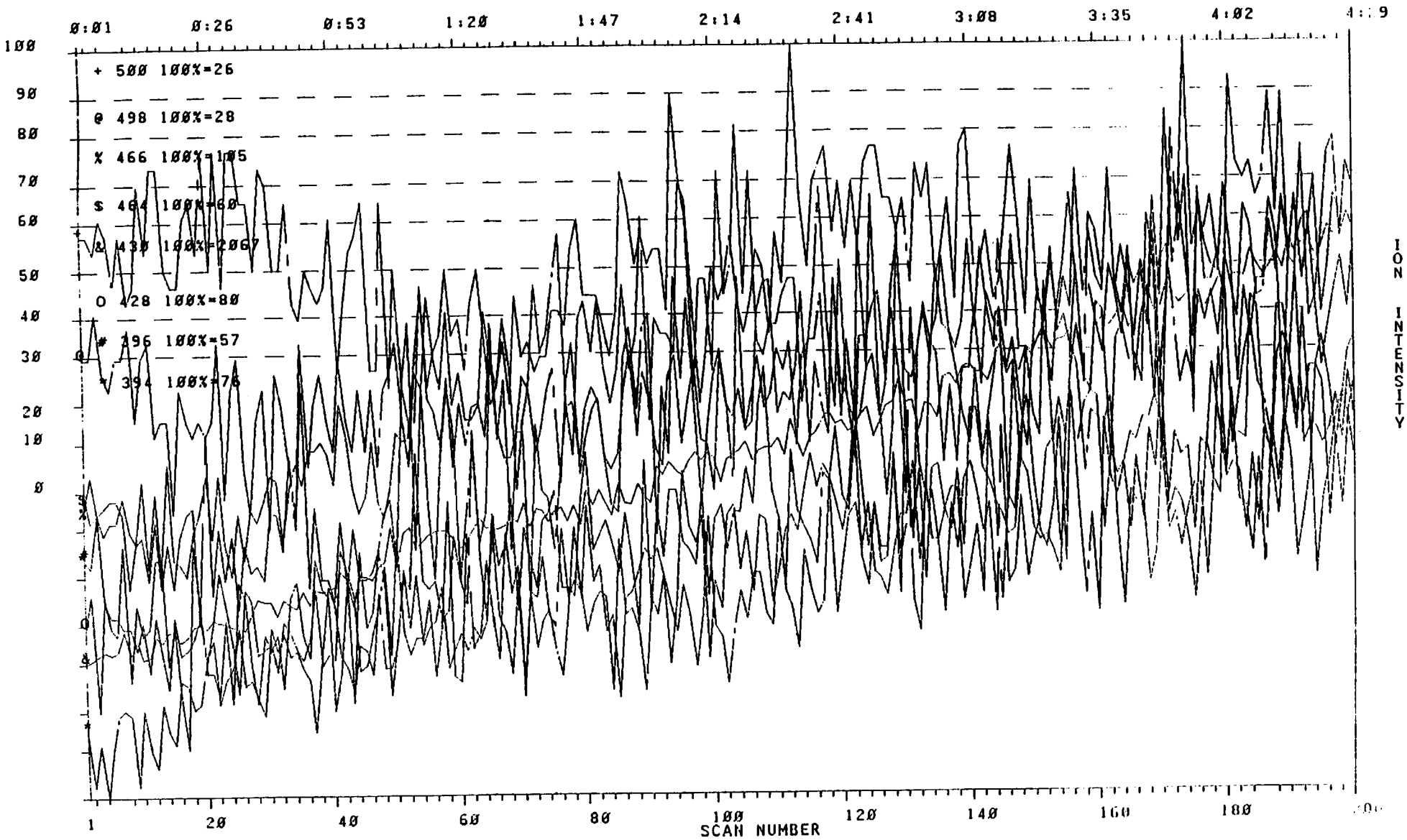
KRATOS MS25, DS55 SOFTWARE, RUN: PCBM0017, WSU NAME: NBC5-1



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 13858

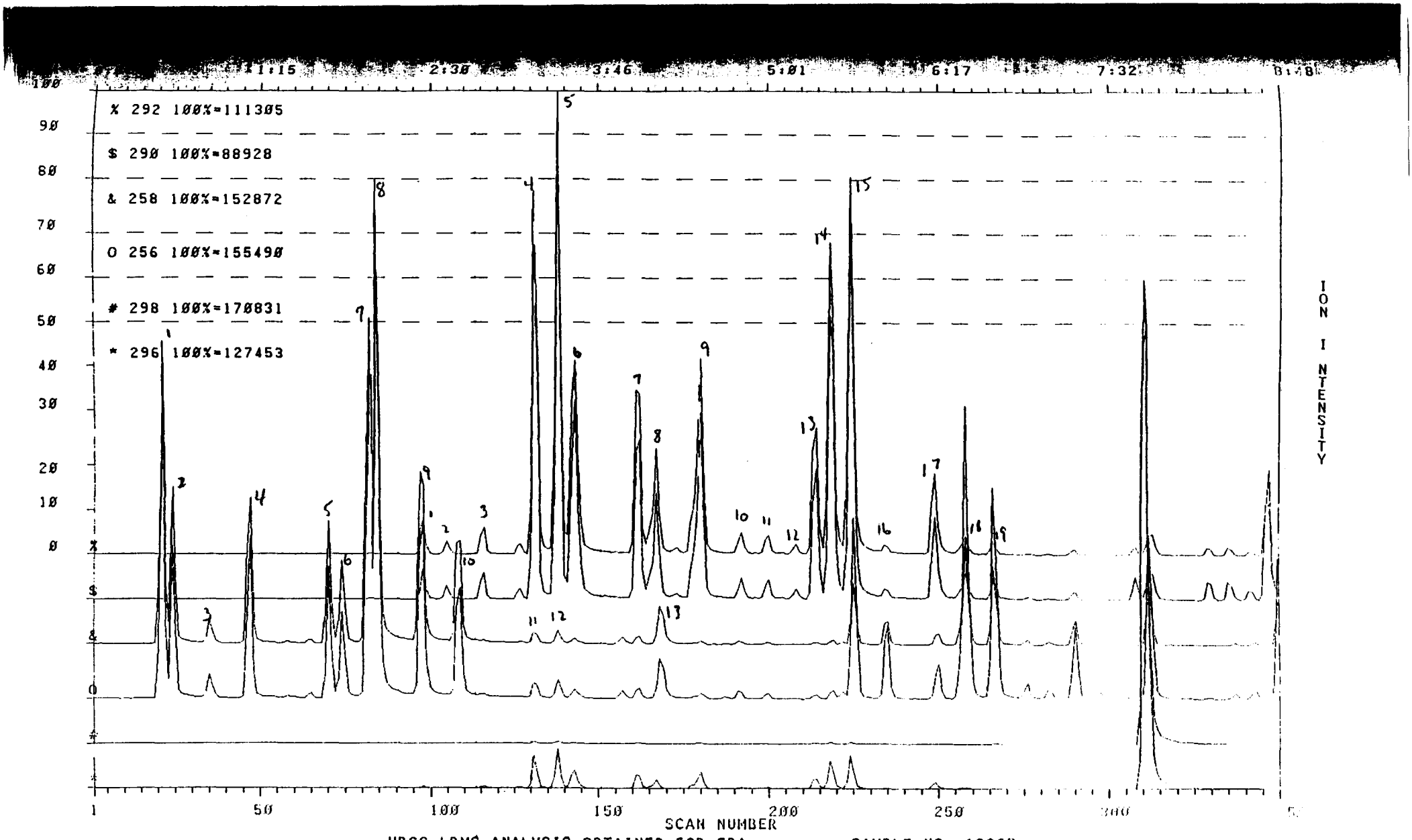
FIGURE: 165

KRATOS MS25, DS55 SOFTWARE, RUN: PCBH0017, WSU NAME: NBC5-1



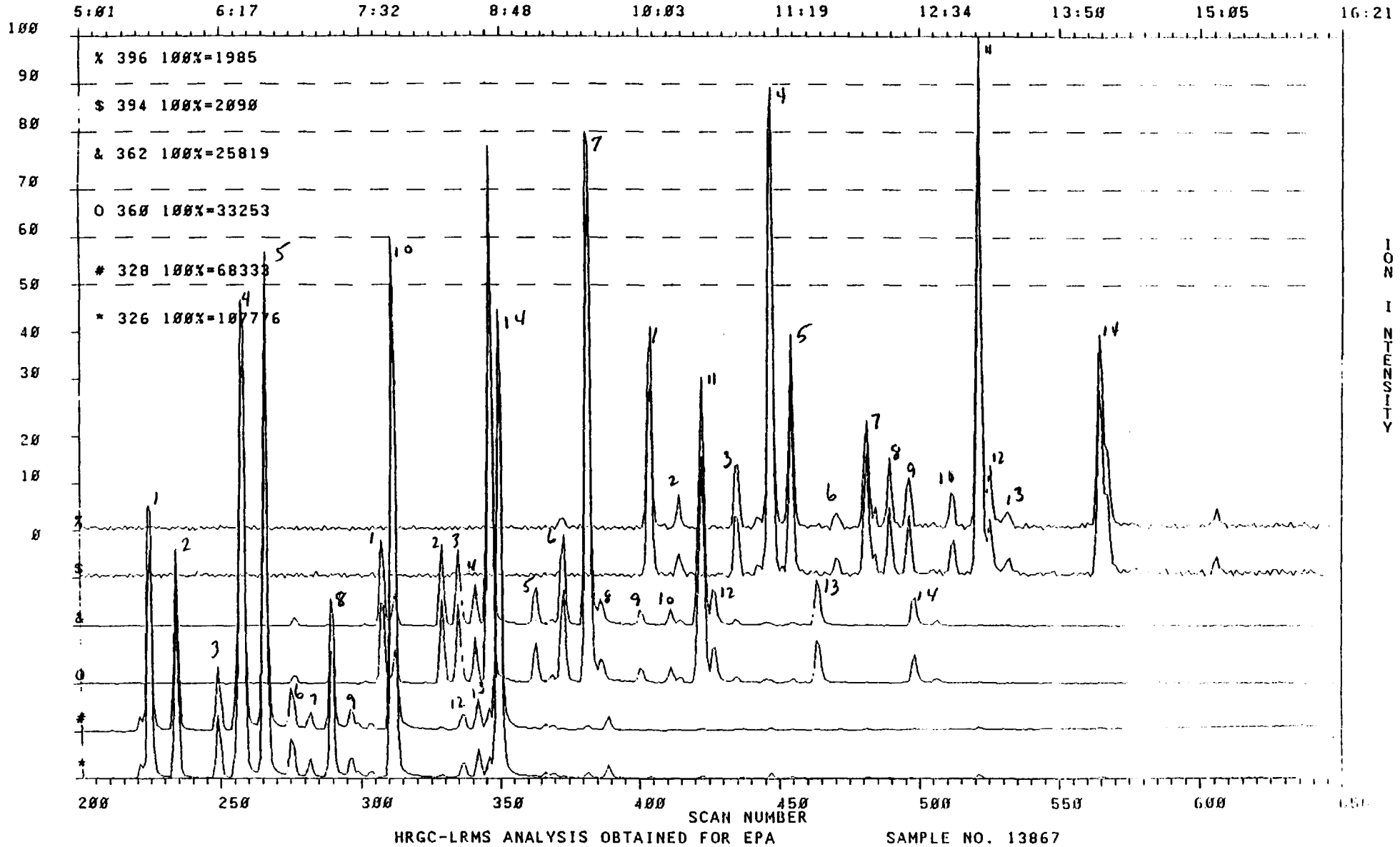
HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 13858

FIGURE: 166



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 13867

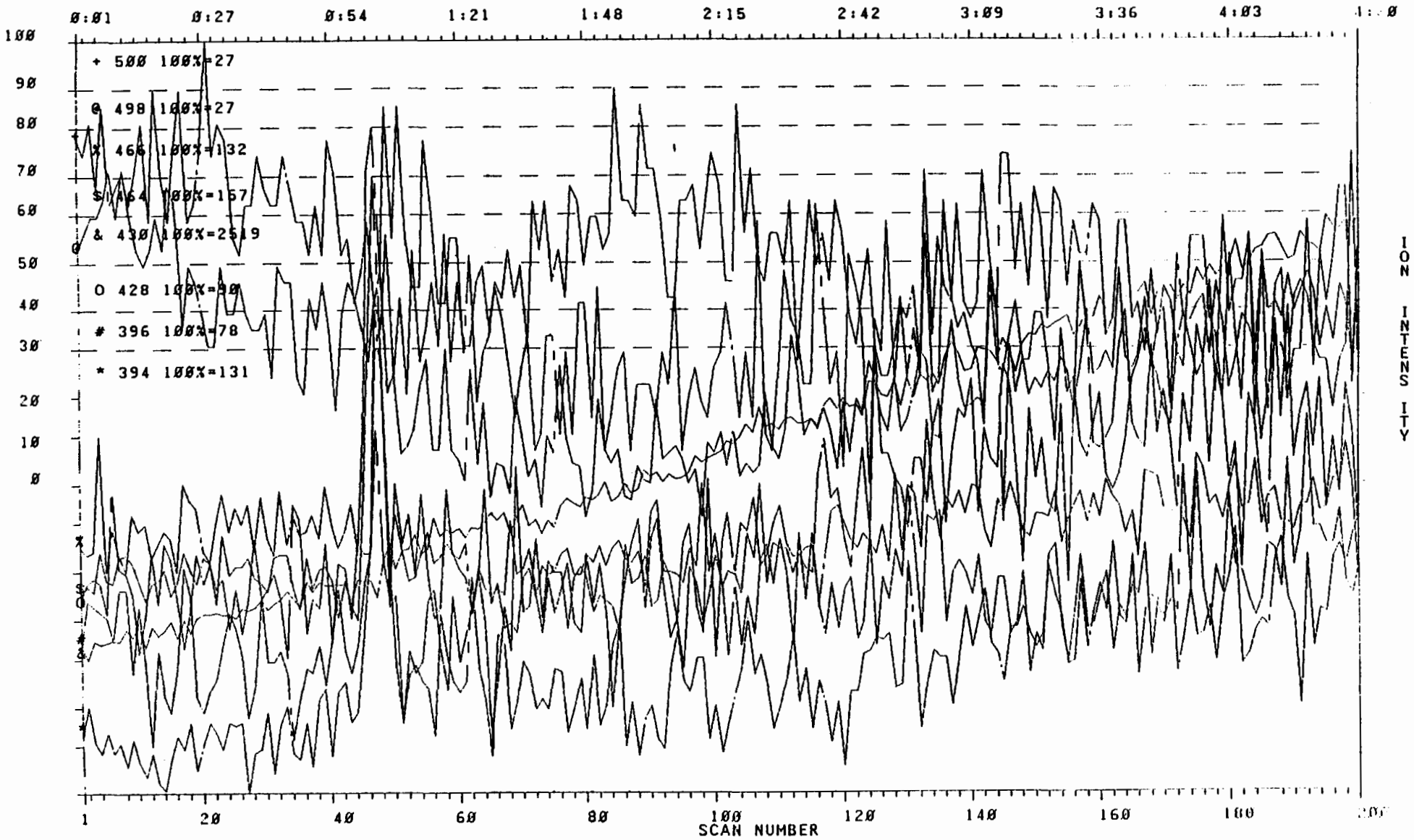
FIGURE: 167



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 13867

FIGURE: 168

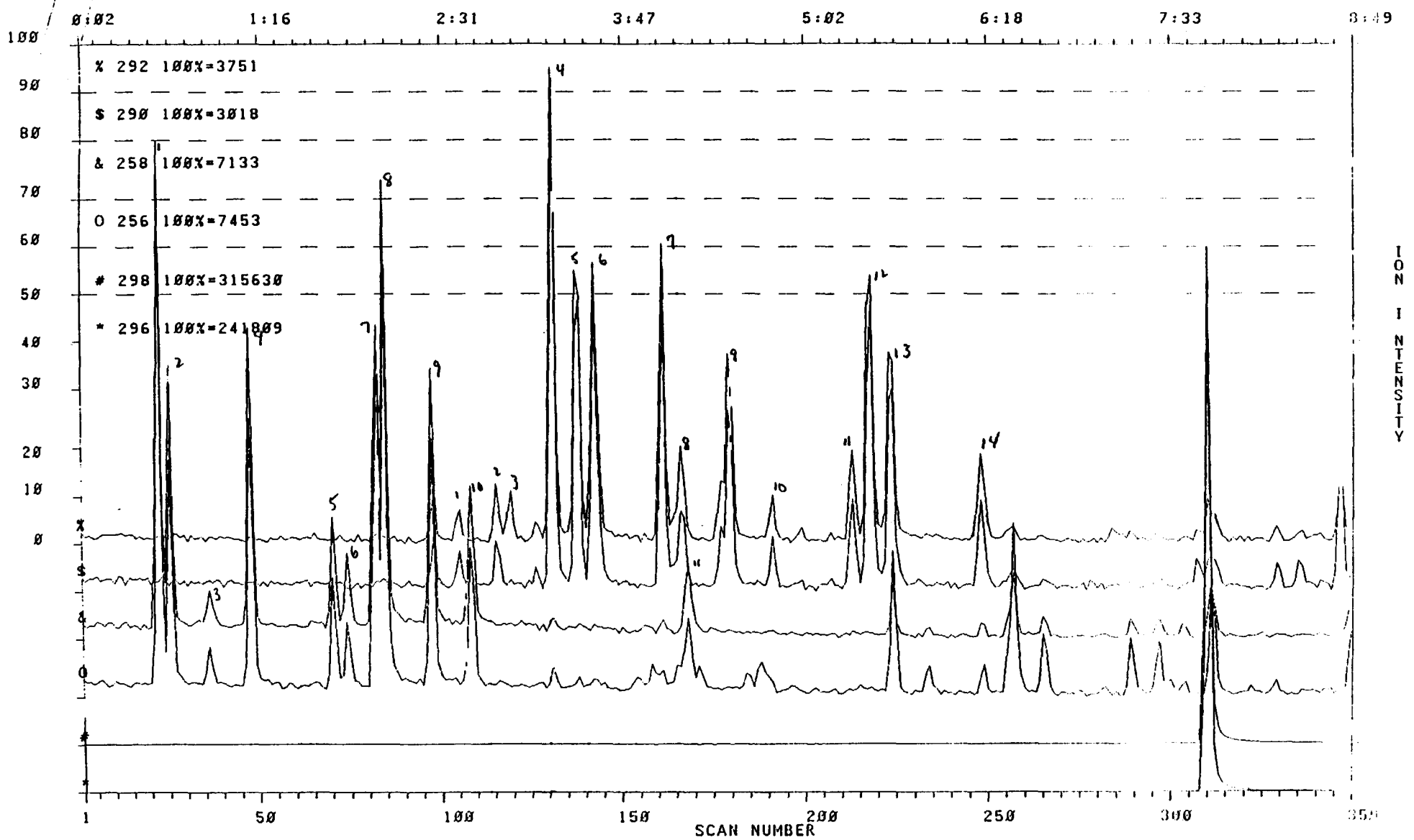
KRATOS MS25, DS55 SOFTWARE, RUN: PCBH0018, WSU NAME: NBC5-5



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. 13867

FIGURE: 169

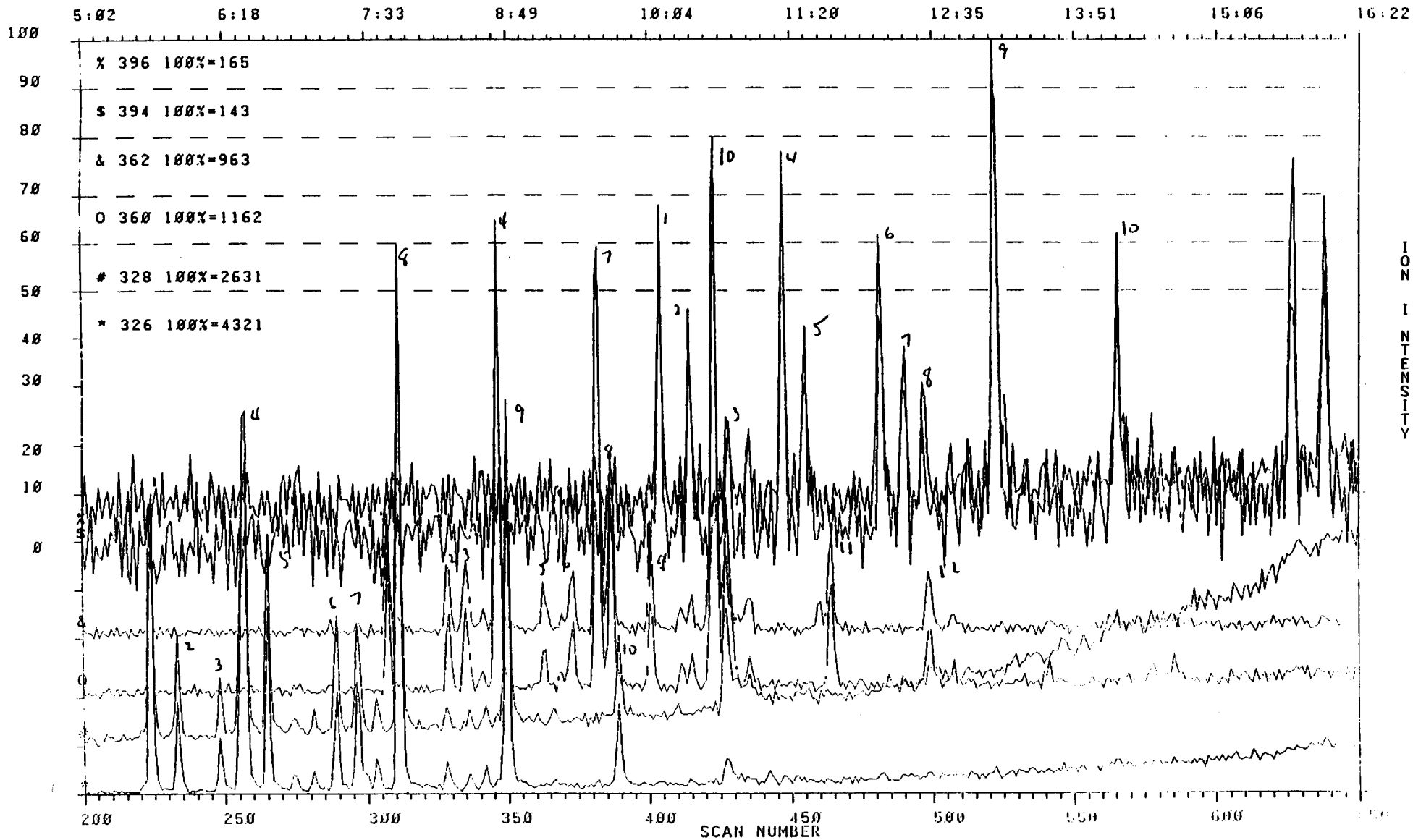
KRATOS MS25, DS55 SOFTWARE, RUN: PCBM0016, WSU NAME: NBC5-0B-B



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.LAB BLANK

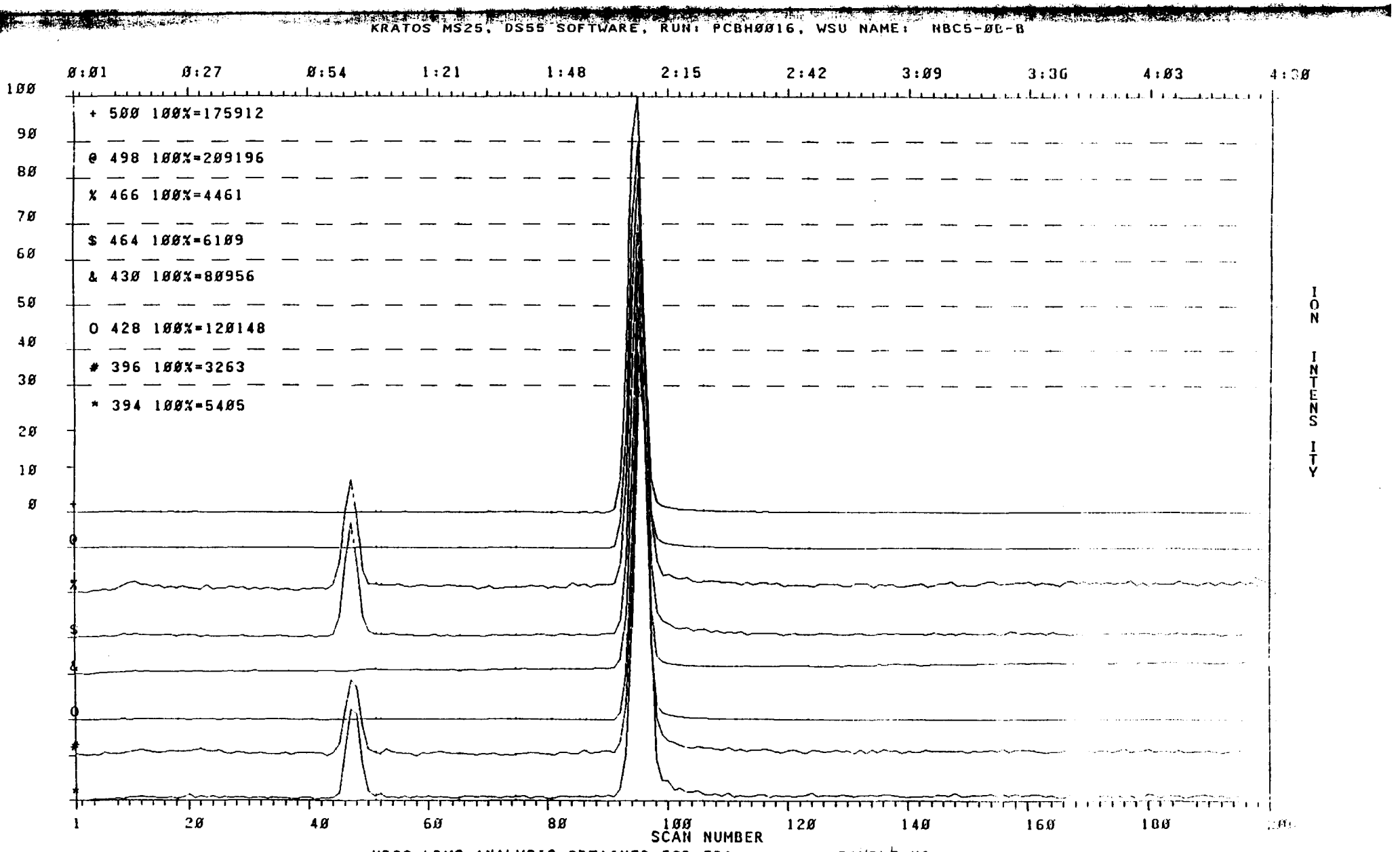
FIGURE: 170

KRATOS MS25, DS55 SOFTWARE, RUN: PCBM00016, WSU NAME: NBC5-00-B



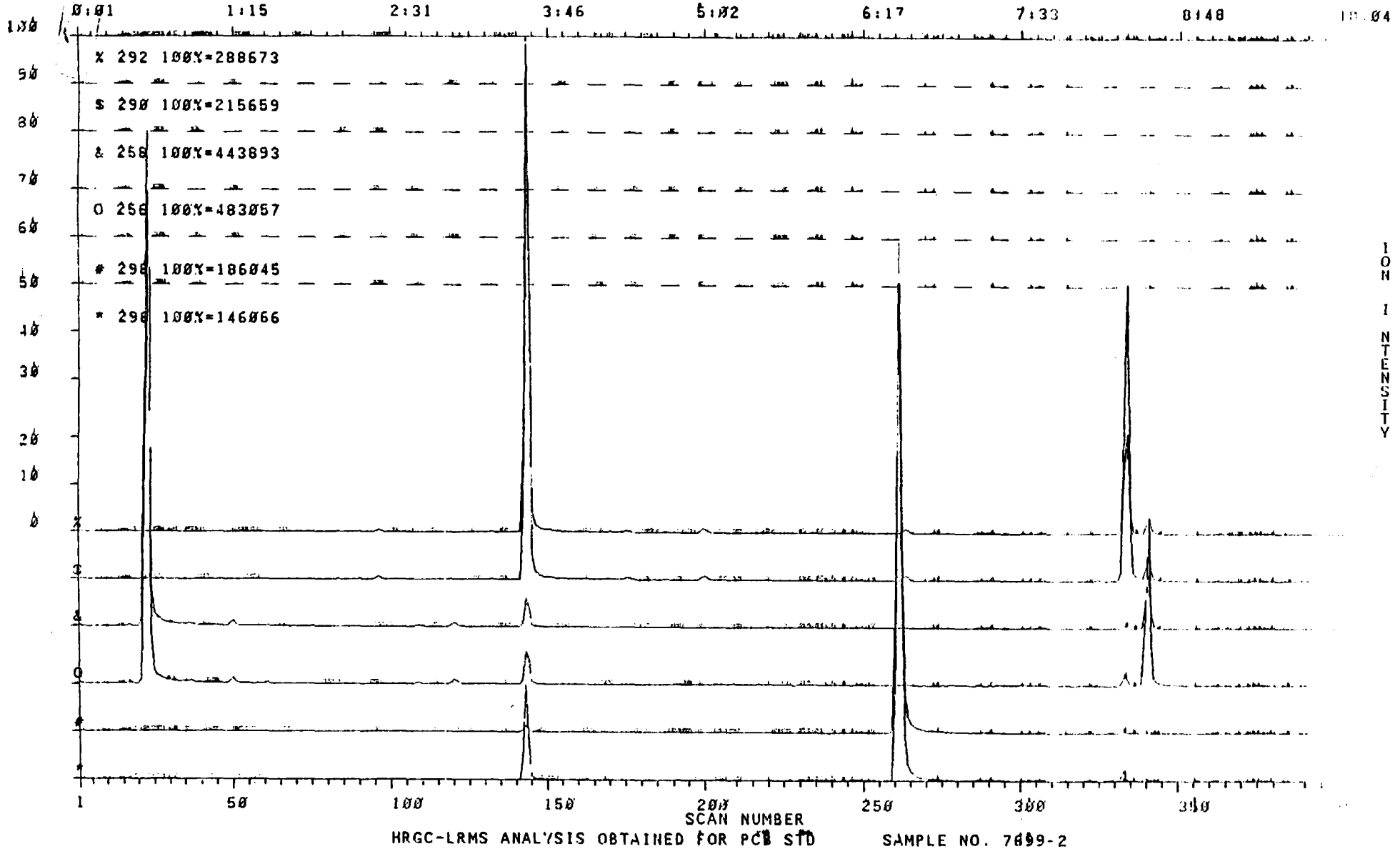
HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.LAB BLANK

FIGURE: 171



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO.

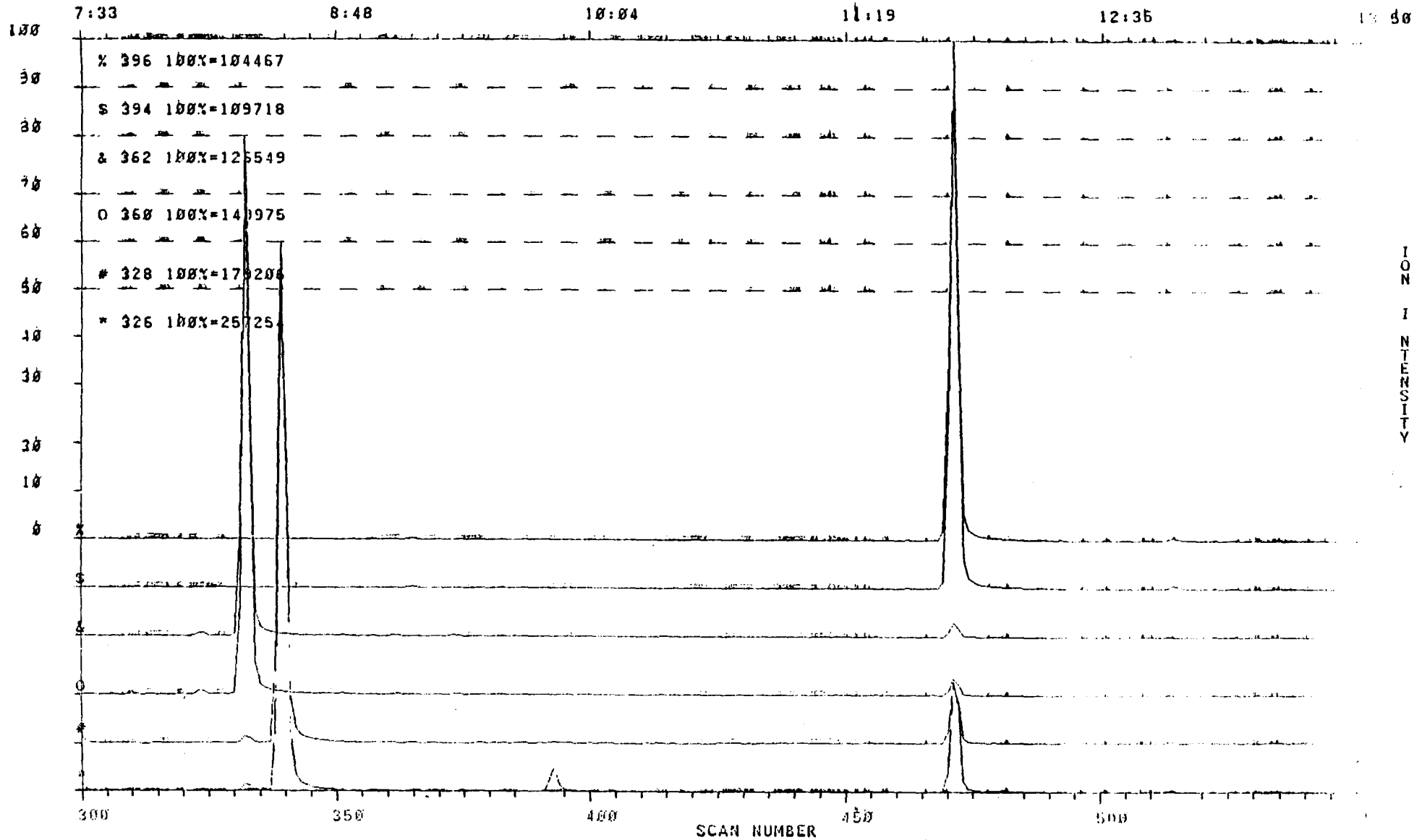
FIGURE: 172



HRGC-LRMS ANALYSIS OBTAINED FOR PCB STD SAMPLE NO. 7699-2

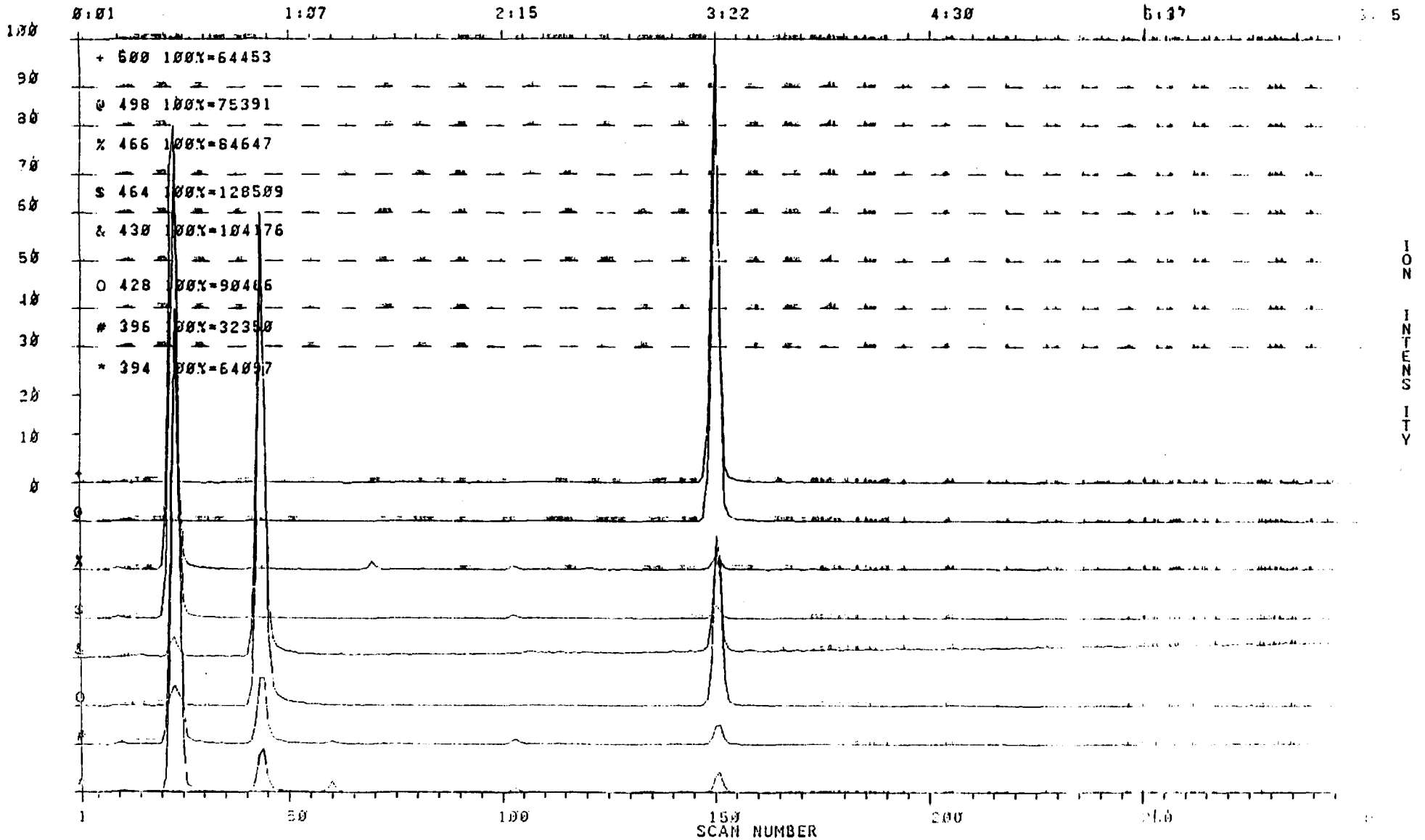
FIGURE: 173

KRATOS MS25, DS55 SOFTWARE, RUN: PCBM0003, WSU NAME: PCB STD



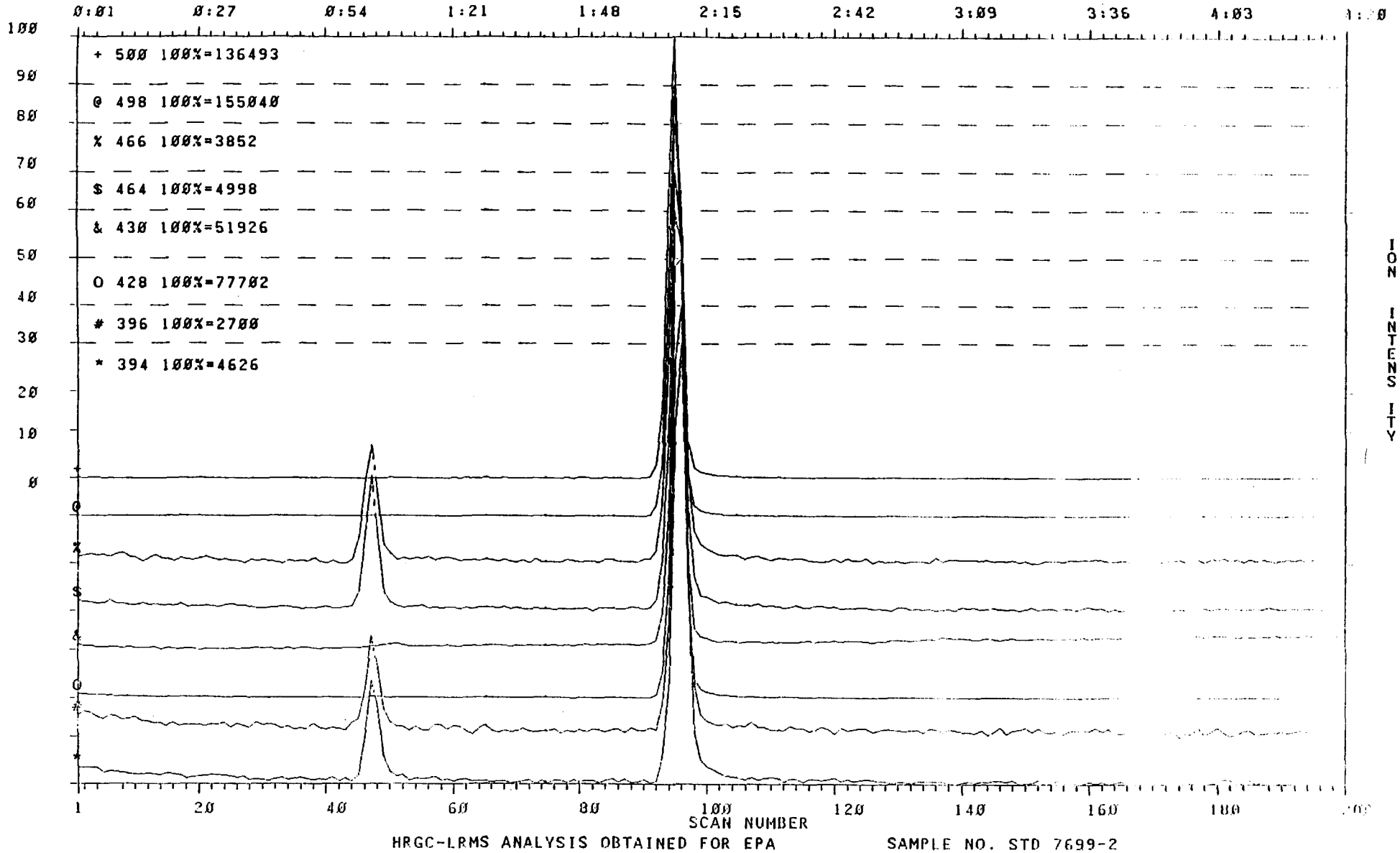
HRGC-LRMS ANALYSIS OBTAINED FOR PCB STD SAMPLE NO. 7699-2

FIGURE: 174



HRGC-LRMS ANALYSIS OBTAINED FOR PCB STD SAMPLE NO. 7699-2

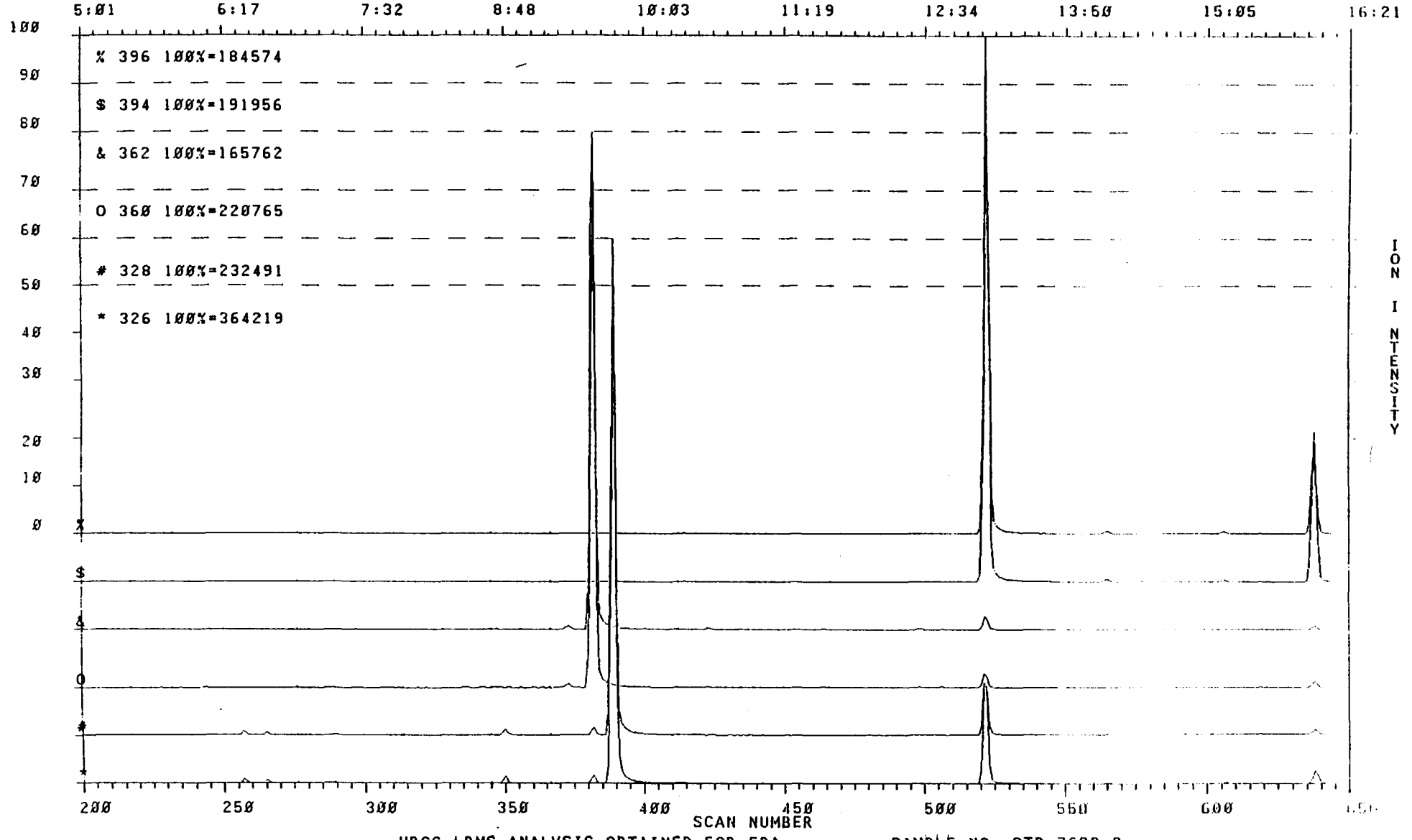
FIGURE: 175



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. STD 7699-2

FIGURE: 178

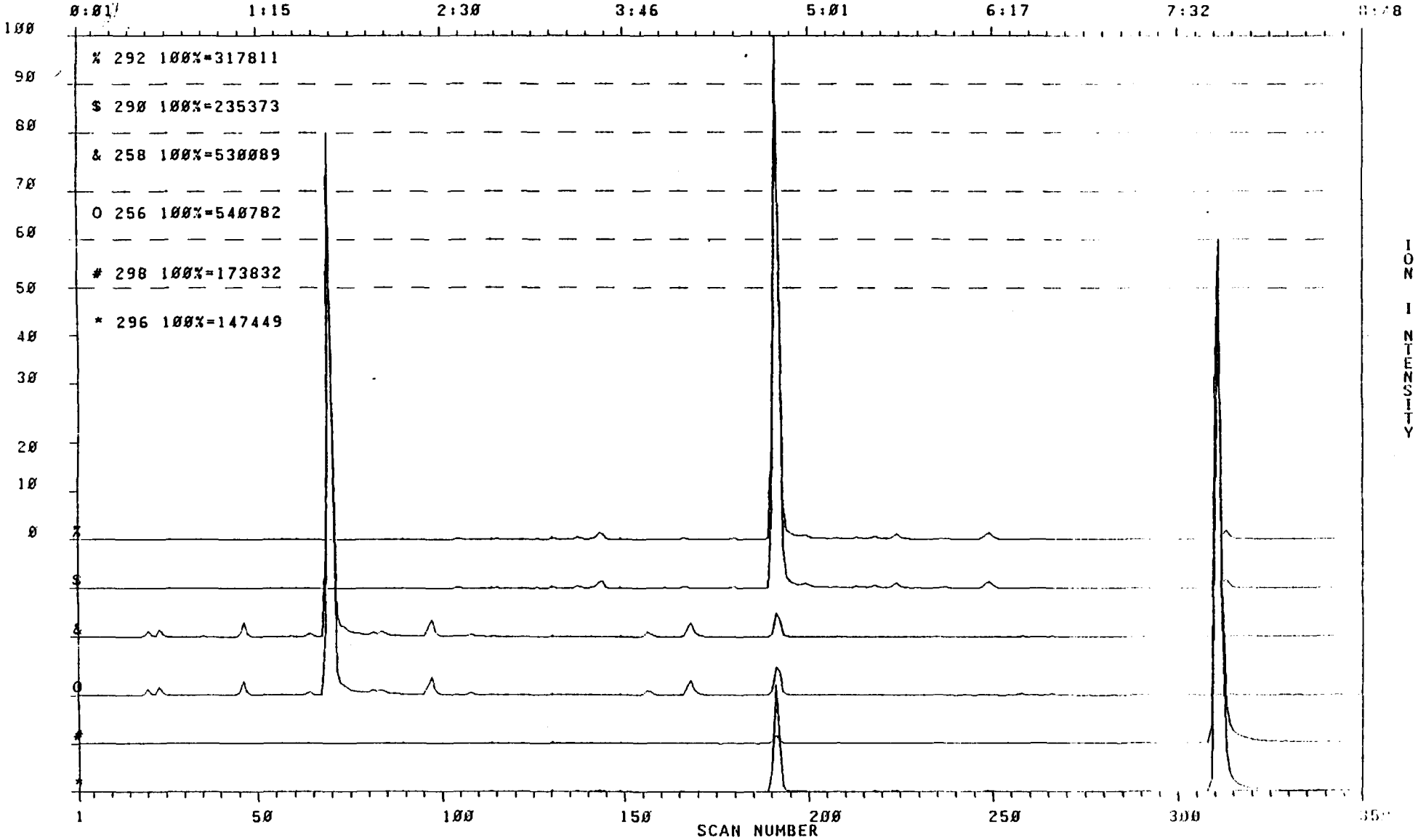
KRATOS MS25, DS55 SOFTWARE, RUN: PCBM0015, WSU NAME: PCB STD



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. STD 7699-2

FIGURE: 177

KRATOS MS25, DS55 SOFTWARE, RUN: PCBMØØ15, WSU NAME: PCB STD



HRGC-LRMS ANALYSIS OBTAINED FOR EPA SAMPLE NO. STD 7699-2

FIGURE: 176