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# Final Report

# Manual and Continuous Emissions Testing, Kiln No. 3 Wet Scrubber Stack Huron Lime Huron, Ohio

# FINAL REPORT

# MANUAL AND CONTINUOUS EMISSIONS TESTING LIME KILN NO. 3 SCRUBBER STACK

## HURON LIME COMPANY
## HURON, OHIO

EPA Contract No. 68-D98-004
Work Assignment No. 3-03

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# 1.0 INTRODUCTION

The U.S. Environmental Protection Agency (EPA) Office of Air Quality Planning and Standards (OAQPS) Emission Standards Division (ESD) is investigating the lime manufacturing industry to identify and quantify hazardous air pollutants (HAPs) emitted from lime kilns. ESD requested that EPA OAQPS Emissions, Monitoring and Analysis Division (EMAD) conduct the required testing. EMAD issued a work assignment to Pacific Environmental Services, Inc. (PES) to conduct a screening test to collect air emissions data as specified in the ESD test request. Initial planning, pre-test site survey, and preparation activities were conducted under EPA Contract No. 68-D7-0002, Work Assignment No. 0/005. Remaining portions of the preparation and field mobilization were conducted under EPA Contract No. 68-D7-0002, Work Assignment No. 1/007, and EPA Contract No. 68-D98-004, Work Assignment 1-09. The draft final report was completed under EPA Contract No. 68-D98-004, Work Assignment Nos. 1-09 and 2-04. Generation of the Final Report, incorporating EPA's comments on the Draft Final Report, was completed under EPA Contract No. 68-D98-004, Work Assignment 3-03

The primary objective was to characterize the controlled emissions of selected HAPs from a rotary kiln located at Huron Lime Company's Huron, Ohio facility. The screening tests were conducted to quantify emission rates of total hydrocarbons (THC), and polychlorinated dibenzo-*p*-dioxins and polychlorinated dibenzofurans (PCDDs/PCDFs) at the Kiln No. 3 stack. Although hydrogen chloride (HCl) was also a target compound, testing was not conducted for HCl due to an instrument malfunction. The basic test methods that were employed were EPA Test Methods 1 (sample point location), 2 (effluent gas velocity), 3A (oxygen and carbon dioxide content), 4 (moisture content), Method 23 (PCDDs/PCDFs content) with proposed revisions, and 25A (THC content). Testing at the facility was conducted on August 31, 1998. One 3-hour test run was conducted at the scrubber stack to determine PCDDs/PCDFs emissions. Concurrent with the Method 23 testing, sampling was conducted at the stack breeching to determine concentrations of oxygen ($O_2$), carbon dioxide ($CO_2$), and THC. Table 1.1 presents the Emissions Test Log, which summarizes the sample run designators, test dates and times, target pollutants, and run durations for each of the sampling methods.

PES used three subcontractors for this effort: Air Pollution Characterization and Control Ltd. (APCC), Paradigm Analytical Laboratories, Inc. (PAL), and Atlantic Technical Services, Inc. (ATS). APCC provided field testing support for measurement of $O_2$, $CO_2$, and THC concentrations using Continuous Emission Monitors (CEMs); PAL prepared the XAD®-2 sorbent resin traps and performed the analysis of the Method 23 sample fractions to determine catch weights of PCDDs/PCDFs congeners; and ATS provided field testing support and field data reduction.

The PES test crew consisted of Michael D. Maret (who served as the Field Team Leader), Troy Abernathy, Gary Gay, and Paul Siegel. APCC was represented by Aaron Christie and Peter Day, and ATS was represented by Emil Stewart. Also present during the testing was Michael L. Toney, the EPA Work Assignment Manager, Joseph P. Wood from EPA ESD, and Cybele M. Brockmann of Research Triangle Institute, an ESD contractor. Huron Lime Company was represented by Mr. A. J. (Tony) Paris.

Figure 1.1 shows the project organization and major lines of communication. Section 2.0 presents the results of the testing; Section 3.0 has a brief process description, Section 4.0 gives descriptions of the sampling locations; Section 5.0 gives descriptions of the sampling and analysis procedures; and Section 6.0 gives the Quality Assurance/Quality Control procedures that were employed during the testing program, and the results of calibrations and analytical QA data. Copies of all field data generated during the testing, the subcontracting laboratory analytical report, computer calculations and example calculations, calibration data and compressed gas certifications of analysis, project participants and reprints of the EPA Test Methods are presented in the appendices to this document. Appendix F has process and operational data supplied by RTI.

## TABLE 1.1

## EMISSIONS TEST LOG
## HURON LIME COMPANY - HURON, OHIO

| Run No. | Date | Pollutant | Run Time (24-hr Clock) | Sampling Duration, (minutes) |
|---|---|---|---|---|
| Kiln No. 3 Scrubber Stack | | | | |
| M23-O-3 | 08/31/98 | PCDDs/PCDFs | 1750-2128 | 180 |
| M3A-O-3 | 08/31/98 | $CO_2$ / $O_2$ | 1749-2134 | 225 |
| M25A-O-3 | 08/31/98 | THC | 1749-2134 | 225 |

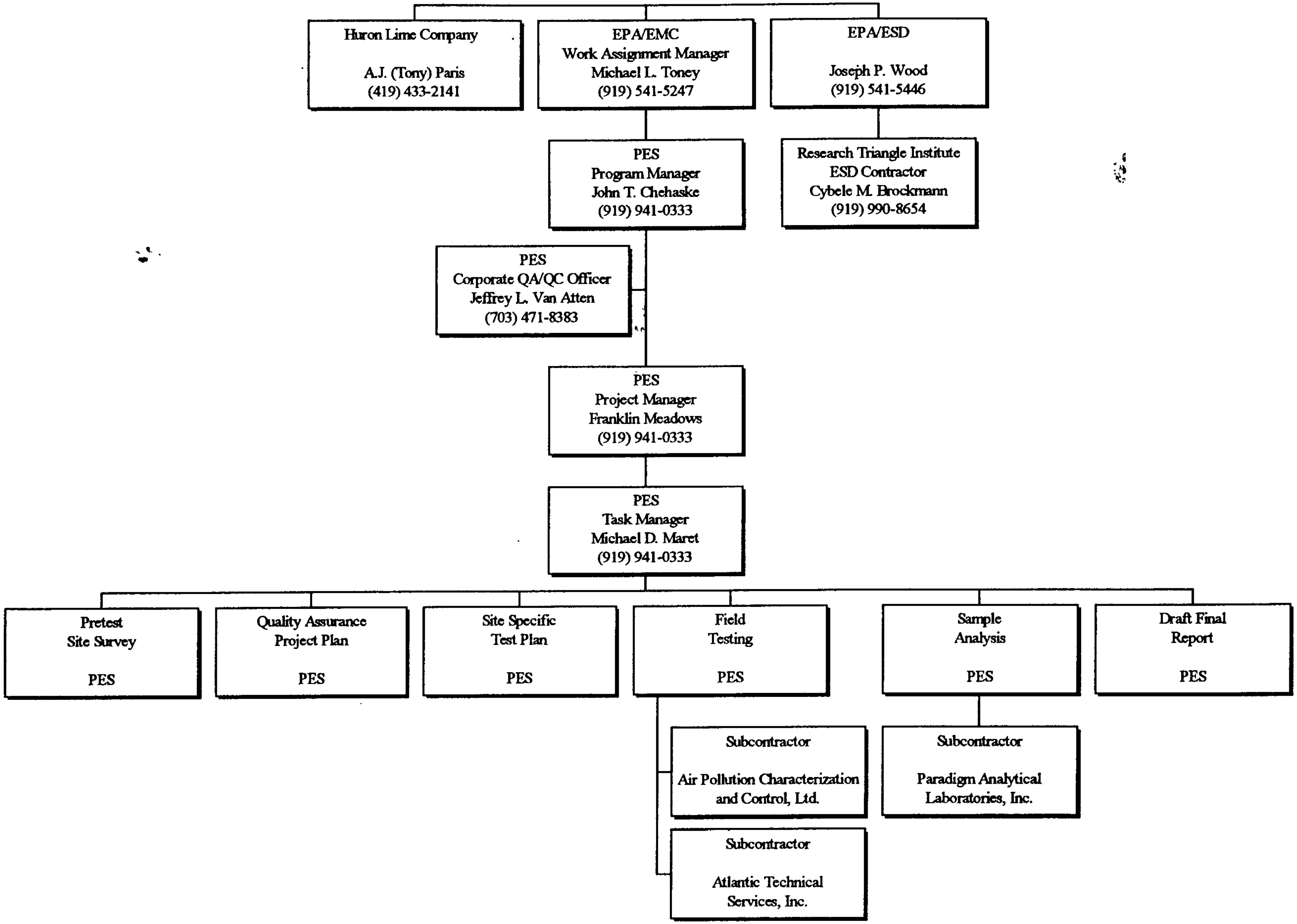


**Figure 1.1 Project Organization - US EPA Ohio Lime Kiln Screening, Huron Lime Company - Huron, Ohio**

## 2.0 SUMMARY OF RESULTS

This section summarizes the results of the testing that was conducted on Rotary Kiln No. 3 at Huron Lime Company's facility in Huron, Ohio. Due to the design of the kiln and scrubber, a location did not exist for the inlet. Therefore, testing was conducted only at the scrubber stack. The following pages present summaries of exhaust gas parameters, pollutant concentrations, and pollutant mass emission rates.

### 2.1 PCDDs/PCDFs MEASUREMENTS

Table 2.1 presents the Method 23 sampling parameters and the parameters of the scrubber stack exhaust gases. Although two sampling ports were available for the collection of an isokinetic sample, PES only conducted sampling through one of the test ports. This modification to the testing was made because PES was unsure of the capability of the existing rail system to support the Method 23 sampling train. Therefore, with the approval of the WAM, PES conducted tests by traversing two times through a single port. One Method 23 sampling run was performed at the scrubber stack location. The isokinetic sampling ratio for run M23-O-3 was 102.1 percent (%), which is within the EPA sampling ratio criterion of 100 ± 10%. For purposes of the calculation of the volumetric flow rates, $O_2$ and $CO_2$ data were determined from the Method 3A CEM data, and moisture content was determined by calculating the mass of condensate collected in the impinger train during the run.

In-stack concentrations and associated mass emission rates of the PCDDs/PCDFs congeners are presented in Table 2.2 for the sampling run. From time to time during the Method 23 analyses, a peak elutes at the position expected for a particular congener, but the peak fails validation based on the theoretical split of chlorine isotopes. That is to say the number of $Cl^{35}$ isotopes and the number of $Cl^{37}$ isotopes attached to the PCDDs/PCDFs congeners should agree with the $Cl^{35}/Cl^{37}$ ratio occurring in nature. For each congener, this ratio must agree within 15%. If the mass ratio of chlorine isotopes does not agree with the natural chlorine isotope ratio, the peak is then flagged as an Estimated Maximum Possible Concentration, or "EMPC".

The values presented as "Total PCDDs" are the sum of the "12346789 OCDD" polychlorinated dibenzo-p-dioxin and all of the dioxins labeled "Total"; "Total PCDFs" is the sum of the "12346789 OCDF" polychlorinated dibenzofuran and all of the furans labeled "Total". "Total PCDDs + Total PCDFs" values are the sum of the "Total PCDDs" and the "Total PCDFs" values. Values that have been qualified as EMPC have been included in the sums.

# TABLE 2.1

## PCDDs/PCDFs SAMPLING AND EXHAUST GAS PARAMETERS
## KILN NO. 3 SCRUBBER STACK
## HURON LIME COMPANY - HURON, OHIO

| **Run No.** | **M23-O-3** |
|---|---|
| Date | [illegible] |
| Clock Time | 1750-2128 |
| Total Sampling Time, minutes | 180 |
| Average Sampling Rate, dscfm [a] | 0.583 |
| Sample Volume: | |
| dscf [b] | 104.912 |
| dscm [c] | 2.971 |
| Average Exhaust Gas Temperature, ° F | 156 |
| $O_2$ Concentration, % by Volume | 6.5 |
| $CO_2$ Concentration, % by Volume | 20.9 |
| Moisture, % by Volume | |
| As Measured | 35.5 |
| Saturation, At Gas Temperature | 29.7 |
| Exhaust Gas Volumetric Flow Rate: | |
| acfm [d] | 49,500 |
| dscfm [a] | 29,500 |
| dscmm [e] | 837 |
| Isokinetic Sampling Ratio, % | 102.1 |

[a] Dry standard cubic feet per minute at 68° F (20° C) and 1 atm.

[b] Dry standard cubic feet at 68° F (20° C) and 1 atm.

[c] Dry standard cubic meters at 68° F (20° C) and 1 atm.

[d] Actual cubic feet per minute at exhaust gas conditions.

[e] Dry standard cubic meters per minute at 68° F (20° C) and 1 atm.

# TABLE 2.2

# PCDDs/PCDFs CONCENTRATIONS AND EMISSION RATES
## KILN NO. 3 SCRUBBER STACK
## HURON LIME COMPANY - HURON, OHIO

| CONGENER | CONCENTRATION [a] (ng/dscm) | EMISSION RATE [b] (µg/hr) |
|---|---|---|
| **DIOXINS:** | | |
| 2378 TCDD | 0.00343 | 0.172 |
| Total TCDD | 0.0508 | 2.55 |
| 12378 PeCDD | 0.00128 | 0.0642 |
| Total PeCDD | 0.0151 | 0.757 |
| 123478 HxCDD | 0.000707 | 0.0355 |
| 123678 HxCDD | 0.00101 | 0.0507 |
| 123789 HxCDD | 0.00104 | 0.0524 |
| Total HxCDD | 0.00498 | 0.250 |
| 1234678 HpCDD | {0.00252} | {0.127} |
| Total HpCDD | (0.000572) | (0.0287) |
| 12346789 OCDD | {0.0107} | {0.539} |
| **Total PCDDs** | **(0.0822)** | **(4.13)** |
| **FURANS:** | | |
| 2378 TCDF | 0.104 | 5.22 |
| Total TCDF | 1.91 | 96.0 |
| 12378 PeCDF | 0.0340 | 1.71 |
| 23478 PeCDF | 0.0203 | 1.02 |
| Total PeCDF | 0.316 | 15.9 |
| 123478 HxCDF | 0.00697 | 0.350 |
| 123678 HxCDF | 0.00401 | 0.201 |
| 234678 HxCDF | 0.00229 | 0.115 |
| 123789 HxCDF | (0.000539) | (0.0270) |
| Total HxCDF | 0.0264 | 1.32 |
| 1234678 HpCDF | 0.00353 | 0.177 |
| 1234789 HpCDF | (0.00114) | (0.0574) |
| Total HpCDF | 0.00350 | 0.176 |
| 12346789 OCDF | (0.00189) | (0.0946) |
| **Total PCDFs** | **(2.260)** | **(113.4)** |
| **Total PCDDs + PCDFs** | **(2.342)** | **(117.6)** |

[a] Nanograms per dry standard cubic meter at 20°C and 1 atm.

[b] Micrograms per hour.

( ) Not Detected. Value shown is the detection limit and is included in totals.

{ } Estimated Maximum Possible Concentration. EMPC values are included in totals.

Concentrations and emission rates based on or including EMPC values are denoted by braces ( {} ). Concentrations and emission rates based on values that have been qualified as being below the detection limit (Not Detected), or ND, are denoted by parentheses ( ( ) ).

Table 2.3 gives two PCDDs/PCDFs concentration-based measurements for the outlet sampling location. In the second column of the table, the in-stack concentrations of the 2378-PCDDs/PCDFs congeners as well as the homologues (i.e., PCDDs and PCDFs groups that have the same degree of chlorination) are shown adjusted to 7% oxygen. The fourth column of the table has the 2378 tetra-chloro dibenzodioxin (TCDD) toxic equivalent values for those congeners chlorinated at the 2, 3, 7, and 8 positions. This column represents the in-stack concentrations of the 2378 congeners after being adjusted for toxicity relative to 2378-TCDDs. PCDDs/PCDFs congeners that are not chlorinated at the 2, 3, 7, and 8 positions have a relative toxicity of zero, therefore, the total homologues (e.g., Total TCDD) are not presented in the Toxic Equivalency columns.

## 2.2 CEM MEASUREMENTS

Gas samples were extracted from the stack breeching at the outlet of the induced draft (ID) fan, conditioned, and transported to the CEMs using heat-traced Teflon® sample lines for the real-time determination of $O_2$, $CO_2$, and THC concentrations. Table 2.4 presents the THC concentrations and mass emission rates. $O_2$ and $CO_2$ concentrations have been corrected for observed calibration and bias errors using Equation 6C-1, as required in Method 3A. THC concentrations are presented uncorrected, as required in Method 25A; the uncorrected $O_2$ and $CO_2$ concentrations are given in Appendix A.2. Refer to Appendix D for example equations.

The THC sampling system drift for the upscale calibration gas was -25.7%. This means that the reported average result of 0.8 ppm is biased low. If the assumption is made that the drift was linear for the run, the actual THC concentration would be approximately 1.6 ppm.

# TABLE 2.3

## PCDDs/PCDFs CONCENTRATIONS AND 2378-TCDD TOXIC EQUIVALENT CONCENTRATIONS ADJUSTED TO 7 PERCENT OXYGEN
## KILN NO. 3 SCRUBBER STACK
## HURON LIME COMPANY - HURON, OHIO

| CONGENER | CONCENTRATION [a] (ng/dscm adjusted to 7 percent $O_2$) | 2378-TCDD Toxicity Equivalence Factor | 2378 TOXIC EQUIVALENCIES (ng/dscm adjusted to 7 percent $O_2$) |
|---|---|---|---|
| | M23-O-3 | | M23-O-3 |
| **DIOXINS:** | | | |
| 2378 TCDD | 0 00331 | 1.000 | 0.00331 |
| Total TCDD | 0.0491 | | |
| 12378 PeCDD | 0.00123 | 0.500 | 0.000617 |
| Total PeCDD | 0.0146 | | |
| 123478 HxCDD | 0.000682 | 0.100 | 0.0000682 |
| 123678 HxCDD | 0.000975 | 0.100 | 0.0000975 |
| 123789 HxCDD | 0.00101 | 0.100 | 0.000101 |
| Total HxCDD | 0.00481 | | |
| 1234678 HpCDD | {0.00244} | 0.010 | {0.0000244} |
| Total HpCDD | (0.000552) | | |
| 12346789 OCDD | {0.0104} | 0.001 | {0.0000104} |
| **Total PCDDs** | **(0.0793)** | **Total PCDDs TEQ** | **{0.00423}** |
| **FURANS:** | | | |
| 2378 TCDF | 0.100 | 0.100 | 0.0100 |
| Total TCDF | 1.85 | | |
| 12378 PeCDF | 0.0328 | 0.050 | 0.00164 |
| 23478 PeCDF | 0.0196 | 0.500 | 0.00978 |
| Total PeCDF | 0.305 | | |
| 123478 HxCDF | 0.00673 | 0.100 | 0.000673 |
| 123678 HxCDF | 0.00387 | 0.100 | 0.000387 |
| 234678 HxCDF | 0.00221 | 0.100 | 0.000221 |
| 123789 HxCDF | (0.000520) | 0.100 | (0.0000520) |
| Total HxCDF | 0.0255 | | |
| 1234678 HpCDF | 0.00341 | 0.010 | 0.0000341 |
| 1234789 HpCDF | (0.00110) | 0.010 | (0.0000110) |
| Total HpCDF | 0.00338 | | |
| 12346789 OCDF | (0.00182) | 0.001 | (0.00000182) |
| **Total PCDFs** | **(2.182)** | **Total PCDFs TEQ** | **(0.0228)** |
| **Total PCDDs + PCDFs** | **(2.261)** | **Total TEQ** | **(0.0271)** |

[a] Nanograms per dry standard cubic meter at 20°C and 1 atm and corrected to 7 percent oxygen.

( ) Not Detected. Value shown is the detection limit and is included in totals.

{ } Estimated Maximum Possible Concentration. EMPC values are included in totals.

## TABLE 2.4

## THC CONCENTRATIONS AND EMISSION RATES
## KILN NO. 3 SCRUBBER STACK BREECHING
## HURON LIME COMPANY - HURON, OHIO

| **Run No.** | **O-3** |
|---|---|
| Date | |
| Clock Time | 1749-2134 |
| Total Sampling Time, minutes | 225 |
| $O_2$ Concentration, % by Volume | 6.5 |
| $CO_2$ Concentration, % by Volume | 20.9 |
| Moisture, % by Volume | 29.7 |
| Volumetric Flow Rate, dscfi | 29,500 |
| | |
| THC (as propane): | |
| Formula Weight, lb/lb-mole | 44.11 |
| Concentration, ppmvw [b] | 0.8 |
| Concentration, ppmvd [c] | 1.14 |
| Concentration, ppmvd @ $7\%O_2$ [d] | 1.10 |
| Emission Rate, lb/hr [e] | [illegible] |

[a] Dry standard cubic feet per minute at 68° F (20° C) and 1 atm.

[b] Parts per million by volume wet.

[c] Parts per million by volume dry.

[d] Parts per million by volume dry basis corrected to 7% oxygen.

[e] Pounds per hour.

## 3.0 PROCESS DESCRIPTION

Kiln No. 3 is an inclined rotating kiln built in 1971. High calcium limestone quarried from Alpena, Michigan, enters the back of the kiln at the highest point of incline and tumbles toward the front of the kiln via gravity and the rotating motion of the kiln. Combustion air and fuel enter at the front of the kiln; the primary fuel is coal, with natural gas used during start-up of the kiln. The lime exits from the front of the kiln

Exhaust from Kiln No. 3 passes through a venturi scrubber, cyclonic mist eliminator, fan, and exhaust stack. The exhaust stack uses dampers to regulate air flow through the system. Water is sprayed in the exhaust as it enters the scrubber. Water from the mist eliminator drains to a sump; river water and clarified water from the settling ponds are also added to the sump. Refer to Appendix F for a description of how and when this occurs. A portion of the water from the settling ponds is also pumped to the venturi.

During the testing, an ESD contractor, Research Triangle Institute, monitored and recorded process operational data; the tabulated data are in Appendix F.

# 4.0 SAMPLING LOCATIONS

Source sampling was performed to determine the controlled emissions of PCDDs/PCDFs, and THC from Kiln No. 3 located at Huron Lime Company's Huron, Ohio facility. Testing was conducted at the scrubber stack and the fan-to-stack breeching ductwork. Figure 4.1 is a simplified process air flow schematic showing the sampling locations.

The scrubber stack was 70.5 inches inside diameter (ID) and exhausted emissions to the atmosphere. As shown in Figure 4.2, the two sampling ports were positioned approximately 240 inches (3.4 diameters) downstream from the breeching to the stack and approximately 840 inches (11.9 diameters) upstream from the stack opening to the atmosphere. As specified by Method 1, the isokinetic testing required a 24 point traverse matrix consisting of 12 traverse points on each of the two perpendicular traverse axes. Although two sampling ports were available for the collection of an isokinetic sample, PES only conducted sampling through one of the test ports. This modification to the testing was made because PES was unsure of the capability of the existing rail system to support the Method 23 sampling train. Therefore, with the approval of the WAM, PES conducted tests by traversing two times through a single port. The ports used for the CEMs testing were located in the breeching immediately downstream from the fan and prior to the stack.

A check for the presence of non-parallel or cyclonic flow, as outlined in Section 2.4 of EPA Method 1, was performed prior to testing. The results of the cyclonic flow test indicated an average yaw angle ($\alpha$) of 6.7°. Because the average yaw angle was less than 20°, which is the maximum allowed by Method 1, the location was considered suitable for isokinetic sampling and required no adjustment to the alignment of the nozzle direction.

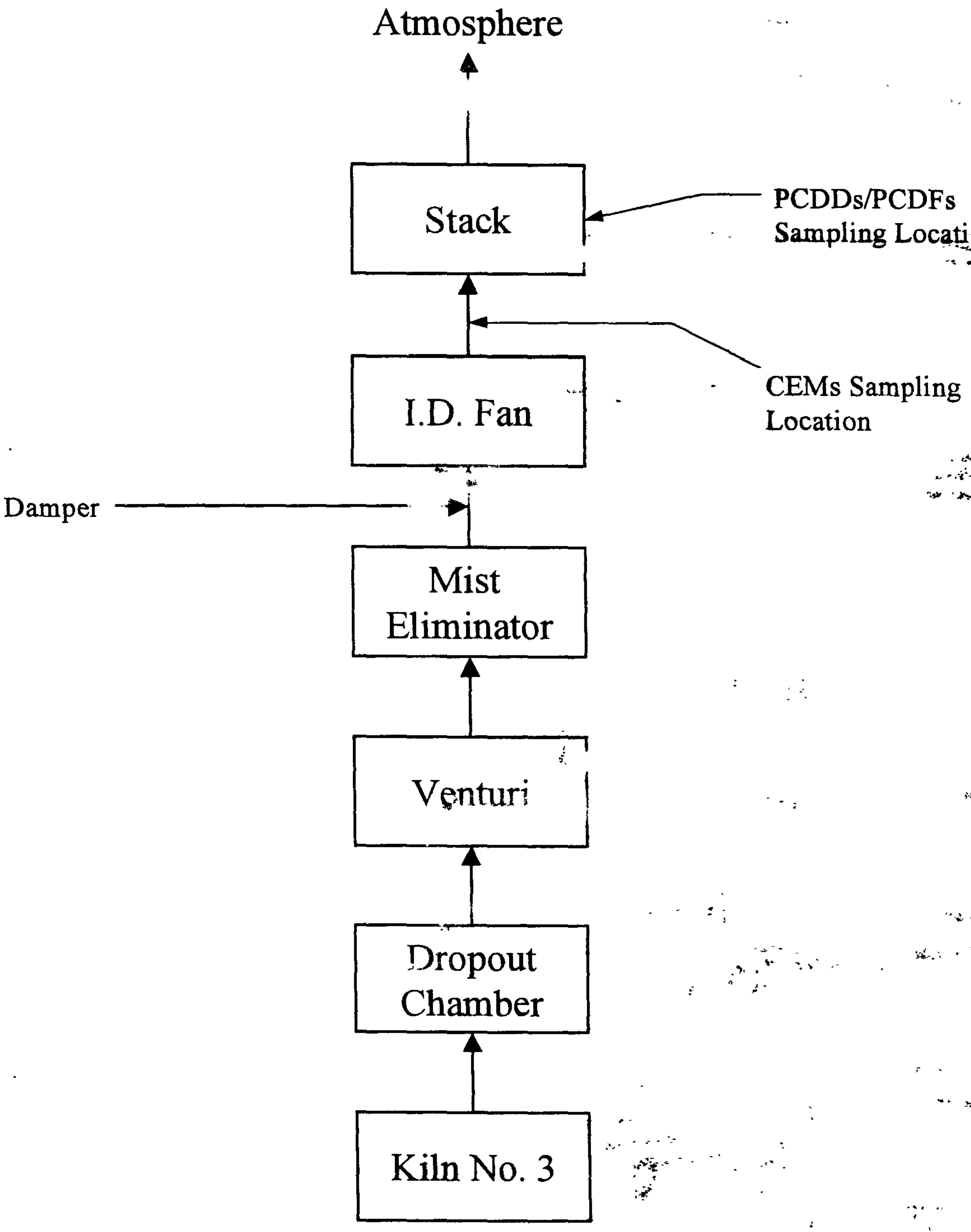


**Figure 4.1 Kiln No. 3 Process Air Flow Schematic, Huron Lime Company - Huron, Ohio**

| Traverse Point Number | Distance From Inside Wall (in.) |
|---|---|
| 1 | 1.48 |
| 2 | 4.72 |
| 3 | 8.32 |
| 4 | 12.5 |
| 5 | 17.6 |
| 6 | 25.1 |
| 7 | 45.4 |
| 8 | 52.9 |
| 9 | 58.0 |
| 10 | 62.2 |
| 11 | 65.8 |
| 12 | 69.0 |

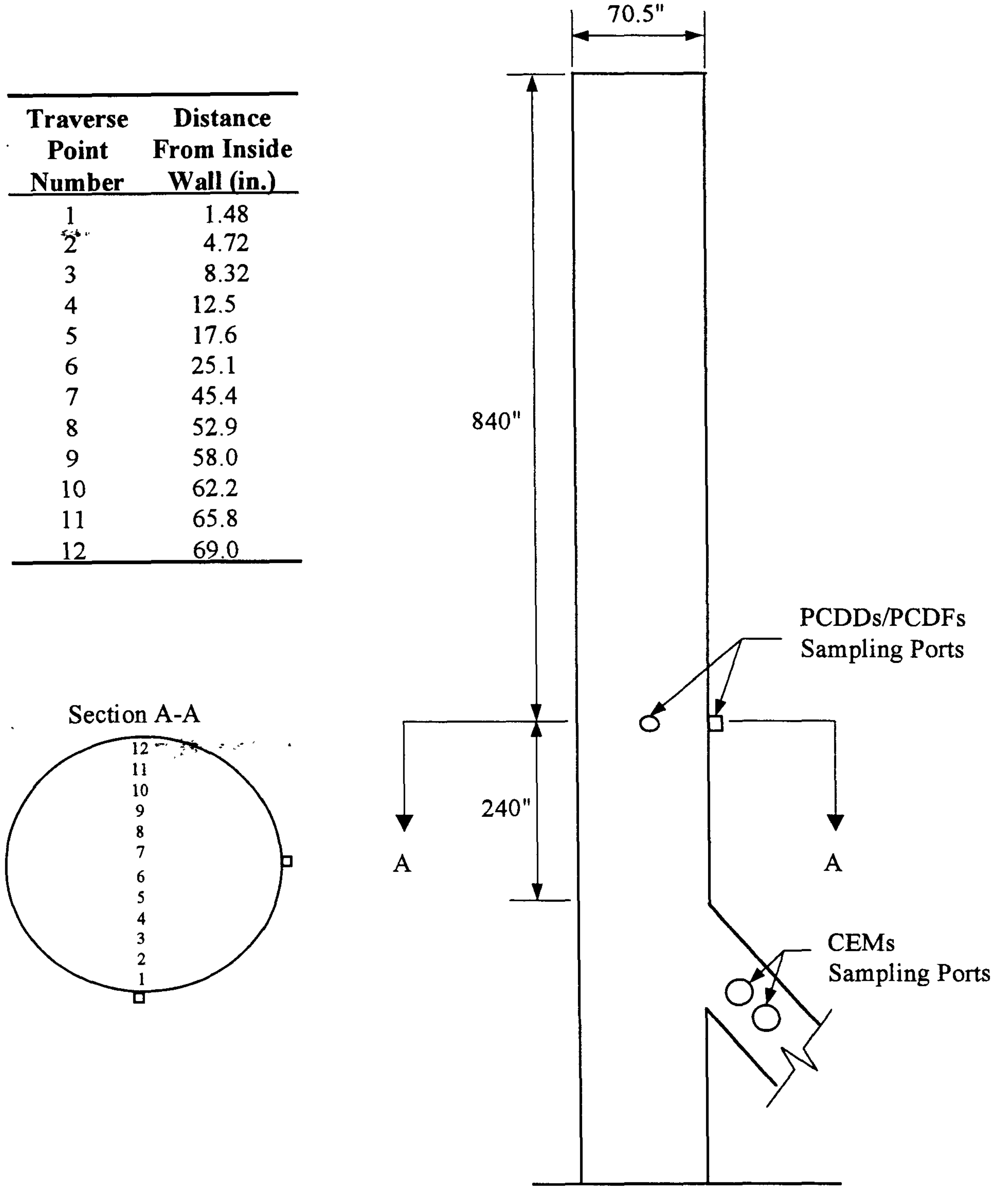


**Figure 4.2 Kiln No. 3 Scrubber Outlet Sample Port and Sample Point Locations, Huron Lime Company - Huron, Ohio**

## 5.0 SAMPLING AND ANALYSIS PROCEDURES

Source sampling was performed at the scrubber stack to determine the concentrations and mass emission rates of PCDDs/PCDFs and THC. One test run was performed at the stack location, with the PCDDs/PCDFs run having a net sampling time of 180 minutes and the THC run having a net sampling time of 225 minutes. The sampling and analytical methods that were used are summarized in Table 5.1. In Table 5.2, the parameters measured, the sampling methods, the number of tests performed, and the duration of each test are summarized. Brief descriptions of the sampling and analysis procedures used are presented below. Copies of all the methods that were used are presented in Appendix G.

### 5.1 LOCATION OF MEASUREMENT SITES AND SAMPLE/VELOCITY TRAVERSE POINTS

EPA Method 1, "Sample and Velocity Traverses for Stationary Sources," was used to establish velocity and sample traverse point locations. The process ductwork, and the locations of measurement sites and traverse points, are discussed in Section 4.0 of this document.

### 5.2 DETERMINATION OF EXHAUST GAS VOLUMETRIC FLOW RATE

EPA Method 2, "Determination of Stack Gas Velocity and Volumetric Flow Rate (Type S Pitot Tube)," was used in conjunction with EPA Method 23 to determine exhaust gas velocity. A Type S Pitot tube, constructed according to Method 2 criteria and having an assigned coefficient of 0.84, was connected to an inclined-vertical manometer. The pitot tube was inserted into the duct and the velocity pressure ($\Delta p$) was recorded at each traverse point. The effluent gas temperature was also recorded at each traverse point using a Type K thermocouple. The average exhaust gas velocity was calculated from the average square roots of the velocity pressure, average exhaust gas temperature, exhaust gas molecular weight, and absolute stack pressure. The volumetric flow rate is the product of velocity and the stack cross-sectional area of the duct at the sampling location.

### 5.3 DETERMINATION OF OXYGEN AND CARBON DIOXIDE

EPA Method 3A, "Determination of Oxygen and Carbon Dioxide Concentrations in Emissions from Stationary Sources," was used to determine the $O_2$ and $CO_2$ concentrations at the scrubber outlet test location.

## TABLE 5.1

## SUMMARY OF SAMPLING AND ANALYSIS METHODS, HURON LIME COMPANY - HURON, OHIO

| Sampling Method | Parameter or Target | Measurement Principle |
|---|---|---|
| EPA Method 1 | Traverse Point Location | [illegible]ement |
| EPA Method 2 | Velocity and Flow Rate | Differential Pressure, Thermocouple, and Angular Measurement |
| EPA Method 3A | $CO_2$ and $O_2$ Content | Micro-Fuel Cell, FINOR |
| EPA Method 4 | Moisture Content | Gravimetric |
| EPA Method 23 (Proposed Revisions) | PCDDs/PCDFs | Gas Chromatography / Mass Spectrometry (GC/MS) |
| EPA Method 25A | THC | Flame Ionization Detector |

## TABLE 5.2

## SUMMARY OF SAMPLING LOCATIONS, TEST PARAMETERS, SAMPLING METHODS, AND NUMBER AND DURATION OF TESTS, HURON LIME COMPANY - HURON, OHIO

| Sampling Location | Test Parameter | Sampling Methods | Number of Tests | Duration, (minutes) |
|---|---|---|---|---|
| Kiln No. 3 Scrubber Stack | Exhaust Gas Flow Rate | EPA Method 2 | 1 | 180 |
| | $CO_2$ & $O_2$ Content | EPA Method 3A | 1 | 225 |
| | Moisture Content | EPA Method 4 | 1 | 180 |
| | PCDDs/PCDFs | EPA Method 23 (Proposed Revisions) | 1 | 180 |
| | THC | EPA Method 25A | 1 | 225 |

Continuous emission monitoring (CEM) was performed at the scrubber outlet in the breeching immediately downstream from the fan. All CEM data was recorded using a Tracor/Westronics 3000 automatic digital data logger. The CEMs were housed in the APCC Environmental Monitoring Laboratory positioned at the base of the stack. Stack gas was drawn from the stack through a heated Teflon® sample line which was maintained at a temperature of approximately 375°F. A portion of the extracted sample was conditioned to remove moisture and directed to the $O_2$ and $CO_2$ analyzers to determine diluent concentrations on a dry basis. The remaining portion of the stack gas sample was directed to the THC analyzer. Figure 5.1 shows a schematic of the sampling system.

A Teledyne Analytical Instruments Model 326 $O_2$ analyzer was utilized to measure the percentage concentration of $O_2$ in the gas stream. The analyzer utilizes a unique micro-fuel cell to measure the concentration of $O_2$. The output signal is linear over the specified ranges of analysis.

A Westinghouse/Maihak FINOR $CO_2$ analyzer was used to monitor $CO_2$ concentrations. The measurement principle for $CO_2$ is IR absorption. Radiation absorbed by $CO_2$ in the sample cell produces a capacitance change in the detector which is proportional to the $CO_2$ concentration.

## 5.4 DETERMINATION OF EXHAUST GAS MOISTURE CONTENT

EPA Method 4, "Determination of Moisture Content in Stack Gases," was used to determine the exhaust gas moisture content. EPA Method 4 was performed in conjunction with the EPA Method 23 test run. Integrated, multi-point, isokinetic sampling was performed. Condensed moisture was determined by recording pre-test and post-test weights of the impingers, XAD® sorbent module, and silica gel.

## 5.5 DETERMINATION OF PCDDs/PCDFs

EPA Method 23, "Determination of Polychlorinated Dibenzo-P-Dioxins and Polychlorinated Dibenzofurans from Stationary Sources," was used to collect dioxins and furans at the test location. The proposed rules amending Method 23 as published in the Federal Register, Volume 60, No. 104, May 31, 1995, were employed. These proposed rules correct existing errors in the method, eliminate the methylene chloride rinse, and clarify the quality assurance requirements of the method. A multi-point integrated sample was extracted isokinetically from the 24 traverse points at the scrubber stack as shown in Section 4.0. Each traverse point was sampled twice for 7.5 minutes each time. The total run time was of 180 minutes.

The EPA Method 23 sample was pulled through a borosilicate glass nozzle, a heated glass-lined probe, a precleaned and heated glass fiber filter, a water-cooled condenser coil, and a sorbent trap containing approximately 40 g of XAD®-2 sorbent resin. The EPA Method 23 sampling train is shown in Figure 5.2.

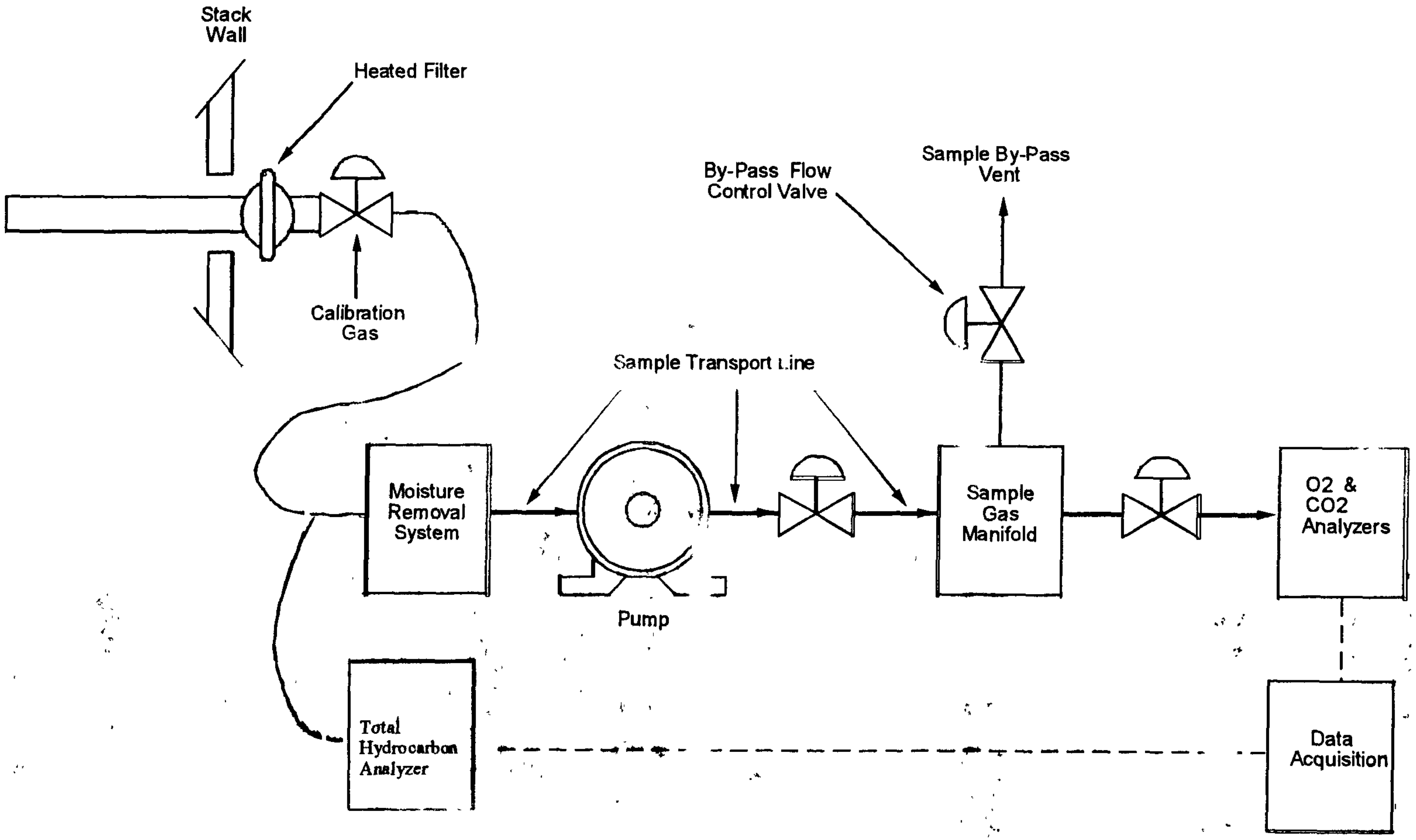


Figure 5.1 Sampling Train Schematic for EPA Methods 3A and 25A.

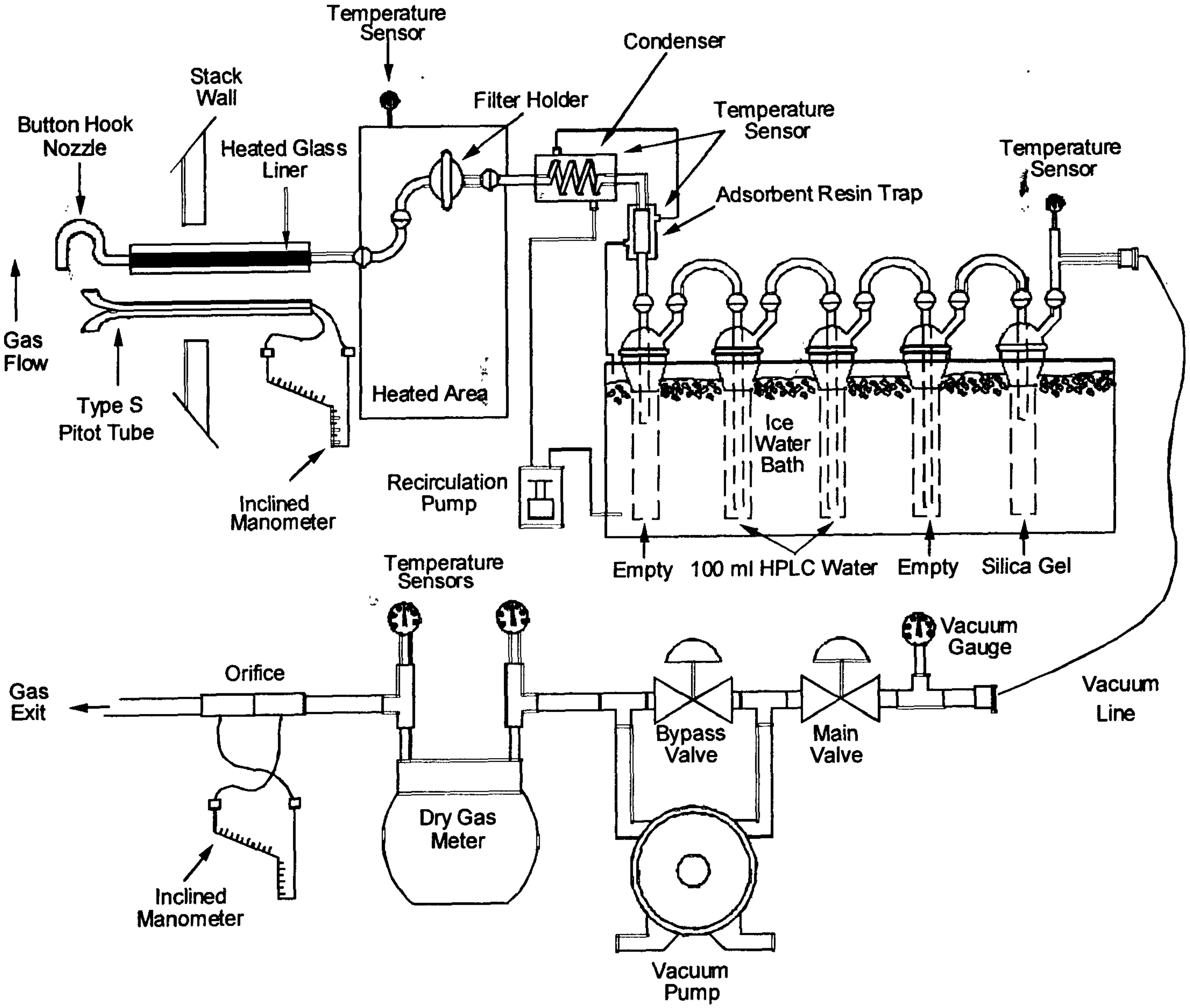


**Figure 5.2 Sampling Train Schematic for EPA Method 23.**

The collected samples were extracted and analyzed according to EPA Method 23 and the above mentioned proposed rules amendment. The sample components (filter, XAD®, and rinses) were Soxhlet extracted and combined. The sample was then split with half being archived and the other half analyzed. Analysis was performed on a high resolution Gas Chromatograph with a high resolution Mass Spectrometer (GC/MS) detector.

## 5.6 DETERMINATION OF TOTAL HYDROCARBONS

EPA Method 25A, "Determination of Total Gaseous Organic Concentration using a Flame Ionization Analyzer," was used to determine the THC concentrations at the test location. A VIG Industries THC Analyzer (or equivalent), which utilizes a flame ionization detector (FID) to measure THCs, was calibrated with propane-in-air standards. Approximately 5.0 liters per minute (lpm) of sample gas is drawn from the source through a heated Teflon® sample line. The sample gas is drawn through a heated filter and valves by a heated pump. The sample gas was introduced into the FID chamber and hydrocarbons in the sample were ionized by a hydrogen flame. The flame was positioned between two charged plates, and the associated electric field induced the migration of the ions towards the charged plates. The ion migration resulted in the generation of a current, which is directly proportional to the amount of THCs present in the sample.

## 5.7 CEMs DATA ACQUISITION AND HANDLING

Analyzer responses were recorded by a Tracor/Westronics 3000 digital data logger which recorded the $O_2$, $CO_2$, and THC concentrations using its integral color printer. Trends were monitored using the strip chart mode with averages printed digitally at 20-minute intervals and at the conclusion of the test period. Analyzer responses were recorded by the data logger at 5 second intervals.

# 6.0 QUALITY ASSURANCE/QUALITY CONTROL (QA/QC) PROCEDURES AND RESULTS

For any environmental measurement, a degree of uncertainty exists in the data generated due to the inherent limitations of the measurement system employed. The goals of a QA/QC program are to ensure, to the highest degree possible, the accuracy of the data collected. This section summarizes the QA/QC procedures that were employed by PES in the performance of this test program. The procedures contained in the reference test methods and in the "Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III, Stationary Source Specific Methods," EPA/600/R-94/038c, served as the basis for performance for all testing and related work activities in this project.

## 6.1 CALIBRATION AND PREPARATION OF APPARATUS

The preparation and calibration of source sampling equipment is essential in maintaining data quality. Brief descriptions of the calibration procedures used by PES are presented below. The results of equipment and sensor calibrations may be found in Appendix E. Detailed procedures as presented in the EPA test methods are presented in Appendix G.

### 6.1.1 Barometers

PES used aneroid barometers which were calibrated against the barometric pressure reported by a nearby National Weather Service station.

### 6.1.2 Temperature Sensors

Bimetallic dial thermometers and Type K thermocouples were calibrated using the procedure described in Calibration Procedure 2e of EPA/600/R-94/038c. Each temperature sensor was calibrated over the expected range of use against an ASTM 3C or 3F thermometer. Table 6.1 summarizes the type of calibrations performed, the acceptable levels of variance, and the results. Digital thermocouple displays were calibrated using a thermocouple simulator having a range of 0-2400°F.

### 6.1.3 Pitot Tubes

PES used Type S pitot tubes constructed according to EPA Method 2 specifications. Each pitot tube was inspected for conformance to the geometric specifications by the application of Calibration Procedure 2 of EPA/600/R-94/038c. Pitot tubes that meet these requirements are

## TABLE 6.1

## SUMMARY OF TEMPERATURE SENSOR CALIBRATION DATA

| Temp. Sensor I.D. | Usage | Temperature, °R | | Temperature Difference | Tolerances |
|---|---|---|---|---|---|
| | | Reference | Sensor | % | |
| 7C | Stack Gas | 534 | 534 | 0.0% | <±1.5% |
| | | 500 | 501 | 0.20% | <±1.5% |
| | | 666 | 665 | -0.15% | <±1.5% |
| | | 800 | 801 | 0.12% | <±1.5% |
| MB-10 | Meter Box Inlet | 493 | 494 | 0.20% | <±1.0% |
| | | 536 | 536 | 0.0% | <±1.0% |
| | | 666 | 665 | -0.15% | <±1.0% |
| | Outlet | 492 | 494 | 0.40% | <±1.0% |
| | | 536 | 537 | 0.19% | <±1.0% |
| | | 666 | 66[illegible] | [illegible] | <±1.0% |

assigned a pitot coefficient, $C_p$, of 0.84. The dimensional criteria and results for each pitot tube used are presented in Table 6.2.

### 6.1.4 Differential Pressure Gauges

PES used Dwyer inclined/vertical manometers to measure differential pressures. The differential pressure measurements included velocity pressure, static pressure, and meter orifice pressure. Manometers were selected with sufficient sensitivity to accurately measure pressures over the entire range of expected values. Manometers are primary standards and require no calibration.

### 6.1.5 EPA Method 23 Dry Gas Meters and Orifices

The EPA Method 23 dry gas meter and orifice was calibrated in accordance with Sections 5.3.1 and 5.3.2 of EPA Method 5. This procedure involves direct comparison of the metered volume passed through the dry gas meter to a reference dry test meter. The reference dry test meter is calibrated annually using a wet test meter. Before its initial use in the field and annually thereafter, the metering system is calibrated over the entire range of operation as specified in EPA Method 5.

Acceptable tolerances for the individual dry gas meter correction factor ($\gamma$) and orifice calibration factor ($\Delta H_@$) during initial or annual calibrations are ± 0.02 and ± 0.20 from the average, respectively. The orifice coefficient for meter MB-10 was out of tolerance for the four inches of water orifice setting; however, the orifice coefficient was within tolerance as operated during the tests. After field use, a calibration check of the metering system was performed at a single intermediate setting based on the previous field test. The post-test calibration check of the dry gas meter correction factor must agree within 5% of the correction factor generated during the initial or annual calibration. The results for the gas meter and orifice used in this test program is summarized in Table 6.3.

## 6.2 REAGENTS AND GLASSWARE PREPARATION

Sample reagents consisted of pesticide (or better) grade acetone and toluene for glassware preparation and sample recoveries, and pesticide (or better) grade hexane for glassware preparation. Sample filters and the XAD®-2 sorbent resin traps were prepared by PAL according to the procedures outlined in Method 23. Water used in the impinger trains was HPLC-grade reagent water.

After preparation of the XAD®-2 sorbent resin traps by PAL, each trap was spiked with a mixture of PCDDs/PCDFs surrogates, and capped with glass balls and sockets until used in the field.

Prior to the field testing portion of the program, all sampling train components and sample recovery apparatus were prepared according to the following procedure.

## TABLE 6.2

## SUMMARY OF PITOT TUBE DIMENSIONAL DATA

| Measurement | Criteria | Results |
|---|---|---|
| | | Pitot Tube Identification |
| | | 7C |
| $\alpha_1$ | < 10° | 1 |
| $\alpha_2$ | < 10° | 3 |
| $\beta_1$ | < 5° | 1 |
| $\beta_2$ | [illegible] | 1 |
| $\gamma$ | - | 1 |
| $\theta$ | - | 1.3 |
| A | - | 0.966 |
| z | ≤ 0.125" | 0.017 |
| w | ≤ 0.0313" | 0.022 |
| $D_t$ | 0.1875 ≤ $D_t$ ≤ 0.375" | 0.375 |
| $(A/2)/D_t$ | 1.05 ≤ $(A/2)/D_t$ ≤ 1 50 | 1.29 |
| | Acceptable | Yes |
| | Assigned Coefficient | 0.84 |

## TABLE 6.3

## SUMMARY OF DRY GAS METER AND ORIFICE CALIBRATION DATA

| Meter No. | Dry Gas Meter Correction Factor, $\gamma$ | | | | Orifice Coefficient, $\Delta H_@$ | | |
|---|---|---|---|---|---|---|---|
| | Pre-test | Post-test | % Diff. | EPA Criteria | Average | Range | EPA Criteria |
| MB-10 | 1.021 | 1.013 | -0.79 | ± 5% | 1.92 | 1.73 - 2.44 | 1.72 - 2.12 |

1. Wash in hot soapy water (Alconox®).
2. Rinse three times with tap water.
3. Rinse three times with distilled/deionized water.
4. Rinse with pesticide-grade acetone.
5. Rinse with pesticide-grade toluene.
6. Rinse with pesticide-grade hexane.
7. Allow to air dry.
8. Cap all openings with hexane-rinsed aluminum foil.

## 6.3 ON-SITE SAMPLING

The on-site QA/QC activities included:

### 6.3.1 Measurement Sites

Prior to sampling, the stack was checked dimensionally to determine measurement site locations, location of velocity and sample test ports, inside stack/duct dimensions, and sample traverse point locations. Inside stack/duct dimensions were checked through both traverse axes to confirm uniformity of the stack/duct inside diameter. The inside stack/duct dimensions, wall thickness, and sample port depths were measured to the nearest 1/16 inch.

### 6.3.2 Velocity Measurements

All velocity measurement apparatus were assembled, leveled, zeroed, and leak-checked prior to use and at the end of each determination. The static pressure was determined at a single point near the center of the stack or duct cross-section.

### 6.3.3 Moisture

The EPA Method 23 train was used to determine stack gas moisture. During sampling, the exit gas of the last impinger was maintained below 68°F to ensure adequate condensation of the exhaust gas water vapor. The total moisture was determined on-site gravimetrically using an electronic platform balance with 0.1 gram sensitivity. The amount of moisture collected by the XAD® trap was also measured.

### 6.3.4 EPA Method 23

The field sampling QA/QC for EPA Method 23 began in the sample recovery area. The sample train was set up and leak-checked to verify sample train integrity before transport to the sampling site. At the sampling site, the sample train was leak checked a second time. Leaks found in excess of 0.02 cubic feet per minute (cfm) were corrected prior to beginning the test run. Leak checks were also conducted before and after any sample train component changes and upon completion of the test run. Sampling was conducted within the isokinetic sampling criteria of 100 ± 10%. Table 6.4 summarizes the EPA Method 23 field sampling QA/QC measurements and EPA's acceptability criteria.

In addition to the stack sample, one field blank sample was collected. An EPA Method 23 sampling train was assembled, transported to the outlet sampling location, and leak-checked three times. The sample train was then recovered using the same procedures employed during the recovery of the sample trains used during actual sample runs. The collected fractions were transferred to labeled, pre-cleaned sample bottles, transported to the subcontract laboratory, and analyzed in the same manner as the collected samples.

PES also collected samples of the reagents that were used during the testing program as blanks. Samples were collected of the acetone and toluene, [illegible] and XAD®-2 sorbent module were also collected. These reagent blank samples were transported to the subcontract laboratory and analyzed for PCDDs/PCDFs using the same procedures as during the analysis of the collected samples.

### 6.3.5 Continuous Emission Monitors

CEMs were used to quantify the in-[illegible] concentrations of $O_2$, $CO_2$, and THC, using EPA Methods 3A and 25A, respectively. QA/QC checks performed included direct calibrations, bias checks, and drift checks. Table 6.5 summarizes the compressed gas standards that were used during the test program.

#### 6.3.5.1 EPA Method 3A

Prior to the start of each day of testing, the $O_2$ and $CO_2$ analyzers were calibrated with a zero gas standard and two upscale standards corresponding to approximately [illegible] and 85% of the instrument measurement ranges. The calibration error of the analyzers on direct calibration was less than or equal to 2% of span. The sampling line bias was then checked with the zero gas and one upscale gas for each analyzer. The sampling line bias was less than or equal to 5% of the response of the analyzer to the calibration standard when injected directly into the analyzer. At the conclusion of the sampling run, the sampling system was again checked by introducing the zero and upscale standard into the system at the probe end. The sampling system drift was less than 3% of the instrument span for both the zero and upscale calibration gases. The true concentration of the gases measured was then calculated from the average instrument response and the results of the calibration responses using Equation 6C-1 as found in Method 6C, which is the procedure specified in Method 3A. The gases used for calibrations were certified by the manufacturer and prepared according to the procedures in "EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards (September 1993)."

#### 6.3.5.2 EPA Method 25A

Prior to the start of each day of testing, the THC sampling system was calibrated with a zero gas standard and three upscale propane-in-air standards corresponding to approximately 25, 50, and 85% of the instrument measurement range. The calibration errors of the THC system were less than 5% of the instrument operating range. At the conclusion of the sampling run, the sampling system was again checked by introducing the zero and one upscale standard into

## TABLE 6.4

## SUMMARY OF EPA METHOD 23 FIELD SAMPLING QA/QC DATA

| **Run No.** | **M23-O-2** |
|---|---|
| Site | Kiln No. 3<br>Scrubber Stack |
| Date | 08/31/98 |
| Leak Rate, acfm | |
| Pretest | 0.005 @ 15" Hg |
| Posttest | 0.01 @ 16" Hg |
| EPA Criteria | 0.02 |
| Percent Isokinetic | |
| Actual | 102.1 |
| EPA Criteria | 90-110% |

## TABLE 6.5

## SUMMARY OF CALIBRATION GAS CYLINDERS

| **Cylinder Number** | **Contents** | **Expiration Date** |
|---|---|---|
| AAL-13302 | 30.0 ppm $C_3H_8$ in air | 5/01/01 |
| ALM-029561 | 50.14 ppm $C_3H_8$ in air | 8/13/01 |
| ALM-044152 | 85.37 ppm $C_3H_8$ in air | 8/13/01 |
| CC86779 | 10.97 % $CO_2$ in $N_2/O_2/CO_2$ | 3/02/01 |
| CC86779 | 11.10 % $O_2$ in $N_2/O_2/CO_2$ | 3/02/01 |
| CC86922 | 19.01 % $CO_2$ in $N_2/O_2/CO_2$ | 3/02/01 |
| CC86922 | 19.17 % $O_2$ in $N_2/O_2/CO_2$ | 3/02/01 |

the system at the probe. The sampling system drift was -25.7% of the instrument span for the upscale calibration gas, which was 50.8 ppm propane-in-air. The THC results are reported as the average of the instrument responses over the period of the sampling run. The gases used for calibration were certified by the manufacturer and prepared according to the procedures in "EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards (September 1993)."

## 6.4 LABORATORY ANALYSES

### 6.4.1 Analysis of Blank Samples

The EPA Method 23 blank samples were analyzed following the procedures of EPA Method 23. Field blanks (FB), reagent blanks (RB), and laboratory blanks were used to evaluate the effectiveness of the sample train clean-up procedures and to check for contamination of the reagent materials. In addition, the subcontract laboratory conducted the Laboratory Method Blank (LMB) to evaluate the presence of contamination of the samples during analysis. The results of these blank analyses and the actual run sample catches are presented in Table 6.6.

### 6.4.2 Standards Recovery Efficiencies

Prior to shipment of the XAD®-2 sorbent modules by PAL, each module was spiked with a mixture of surrogate (sampling) standards. Upon analysis, the recoveries of the surrogate standards provide a measure of the capture and holding efficiency of the XAD®-2 sorbent traps for the sampled PCDDs/PCDFs. A low recovery efficiency may indicate the loss of PCDDs/PCDFs congeners from the XAD®-2 sorbent module after its recovery from the sampling train. The HxCDF surrogate standard recovery efficiency for sample M23-FB-3 was 147% which is above the 130% criteria. Similar recoveries were noted in the LMB sample. The PAL lab report remarks state that "...these observations originate from a variation in the response factors and should not affect the reported amounts of HxCDFs in the sample."

Upon receipt of the XAD®-2 sorbent modules by the laboratory after sampling, the XAD®-2 sorbent resin modules were spiked with a mixture of internal (extraction) standards. The purpose of these standards was to evaluate the efficiency of the extraction of the PCDDs/PCDFs congeners from the sample fractions. The results of these recoveries are presented in Table 6.7.

## TABLE 6.6

## SUMMARY OF EPA METHOD 23 BLANKS & SAMPLE CATCHES

| | Catch, ng Per Sample | | | |
|---|---|---|---|---|
| | LMB | M23-RB [a] | M23-FB-3 | M23-O-3 |
| PAL Lab Report Page Numbers in Appendix B. | 020 | 190 [a] | 079 | 004 / 052 |
| Analyte | | | | |
| 2378-TCDD | (0.0012) | (0.0010) | (0.0012) | 0.0102 |
| 12378-PeCDD | 0.0018 | (0.0005) | (0.0007) | 0.0038 |
| 123478-HxCDD | {0.0018} | (0.0008) | (0.0013) | 0.0021 |
| 123678-HxCDD | 0.0019 | (0.0007) | {0.0013} | 0.0030 |
| 123789-HxCDD | {0.0018} | 0.0012 | (0.0010) | 0.0031 |
| 1234678-HpCDD | (0.0017) | 0.0027 | {0.0034} | {0.0075} |
| OCDD | (0.0096) | (0.0055) | {0.0092} | {0.0319} |
| 2378-TCDF [b] | (0.0016) | (0.0015) | (0.0015) | 0.309 |
| 12378-PeCDF | 0.0023 | (0.0005) | (0.0008) | 0.101 |
| 23478-PeCDF | {0.0020} | (0.0005) | (0.0007) | 0.0602 |
| 123478-HxCDF | {0.0016} | (0.0010) | 0.0022 | 0.0207 |
| 123678-HxCDF | {0.0013} | (0.0008) | {0.0015) | 0.0119 |
| 234678-HxCDF | {0.0013} | (0.0009) | (0.0011) | 0.0068 |
| 123789-HxCDF | {0.0018} | (0.0011) | (0.0013) | (0.0016) |
| 1234678-HpCDF | {0.0030} | 0.0038 | (0.0022) | 0.0105 |
| 1234789-HpCDF | (0.0028) | (0.0011) | (0.0028) | (0.0034) |
| OCDF | (0.0041) | (0.0031) | (0.0047) | (0.0056) |
| Total TCDDs | (0.0012) | (0.0010) | (0.0012) | 0.151 |
| Total PeCDDs | 0.0016 | (0.0005) | (0.0007) | 0.0448 |
| Total HxCDDs | 0.0020 | 0.0012 | 0.0016 | 0.0148 |
| Total HpCDDs | (0.0017) | 0.0028 | (0.0014) | (0.0017) |
| Total TCDFs | (0.0016) | 0.0016 | (0.0015) | 5.68 |
| Total PeCDFs | 0.0024 | (0.0005) | (0.0007) | 0.940 |
| Total HxCDFs | (0.0006) | (0.0008) | 0.0024 | 0.0784 |
| Total HpCDFs | (0.0022) | 0.0036 | (0.0022) | 0.0104 |
| **Total PCDD/Fs [c]** | **0.0060** | **0.0092** | **0.0040** | **6.9194** |

[a] Sample RB-1 collected at a different lime kiln facility tested during the same mobilization. The pages are inserted at the end of Appendix B; the page numbers are out of sequence.

[b] Result obtained from the DB-225 analysis.

[c] Total PCDD/Fs represent the sum of all polychlorinated dibenzo-p-dioxins & dibenzofurans.

( ) Denotes a non-detect value using the detection limit.

{} Denotes an EMPC value.

## TABLE 6.7

## SUMMARY OF EPA METHOD 23 STANDARDS RECOVERY EFFICIENCIES

| | Percent Recovery | | | | QC Limits |
|---|---|---|---|---|---|
| | LMB | M23-O-3 | M23-FB-3 | M23-RB[a] | |
| FULL SCREEN ANALYSIS<br>PAL Lab Report Page Number | 021 | 053 | 080 | 191 | |
| Internal (Extraction) Standards | | | | | |
| $^{13}C_{12}$-2378-TCDD | 87.1 | 87.5 | 81.3 | 84.7 | 40-130% |
| $^{13}C_{12}$-12378-PeCDD | 107.2 | 105.9 | 101.7 | 100.1 | 40-130% |
| $^{13}C_{12}$-123678-HxCDD | 98.5 | 98.8 | 96.2 | 84.5 | 40-130% |
| $^{13}C_{12}$-1234678-HpCDD | 85.1 | 77.3 | 76.3 | 78.4 | 40-130% |
| $^{13}C_{12}$-OCDD | 67.0 | 64.2 | 61.6 | 52.4 | 40-130% |
| $^{13}C_{12}$-2378-TCDF | 74.6 | 83.9 | 74.0 | 77.9 | 40-130% |
| $^{13}C_{12}$-12378-PeCDF | 69.7 | 83.6 | 72.7 | 78.1 | 25-130% |
| $^{13}C_{12}$-123678-HxCDF | 85.8 | 78.6 | 60.6 | 61.6 | 25-130% |
| $^{13}C_{12}$-1234678-HpCDF | 54.9 | 63.5 | 54.3 | 55.9 | 25-130% |
| Surrogate (Sampling) Standards | | | | | |
| $^{37}Cl_4$-2378-TCDD | 107.3 | 10[illegible] | [illegible] | | 70-130% |
| $^{13}C_{12}$-23478-PeCDF | **146.5** | 103.8 | 106.4 | - | 70-130% |
| $^{13}C_{12}$-123478-HxCDD | 92.5 | 80.7 | 78.9 | - | 70-130% |
| $^{13}C_{12}$-123478-HxCDF | 85.9 | 118.3 | **147.1** | - | 70-130% |
| $^{13}C_{12}$-1234789-HpCDF | **169.0** | 85.4 | 78.9 | - | 70-130% |

[a] The "M23-RB" sample was collected at a different lime kiln facility tested during the same mobilization. The pages are inserted at the end of Appendix B, resulting in the page numbers being out of sequence.

Note: Recovery efficiencies in **bold** are outside the QC limits.

# APPENDIX A

# RAW FIELD DATA

# Appendix A.1

## Raw Field Data

## Kiln No. 3 Scrubber Stack

# TRAVERSE POINT LOCATION FOR CIRCULAR DUCTS

Plant: PORT HURON

Date: 8·29·98

Sampling Location: #3 SCRUBBER OUTLET

Inside of Far Wall to Outside of Nipple: 76.5

Inside of Near Wall to Outside of Nipple (Nipple Length): 6

Stack I.D.: 70.5

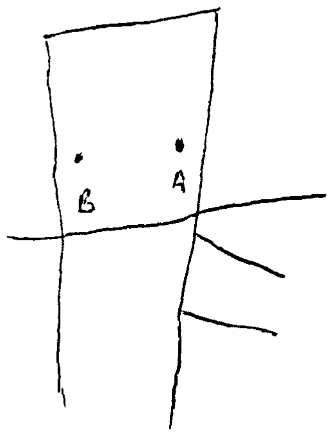
Distance Downstream from Flow Disturbance (Distance B):

240 inches / Stack I.D. = 3.4 dd

Distance Upstream from Flow Disturbance (Distance A):

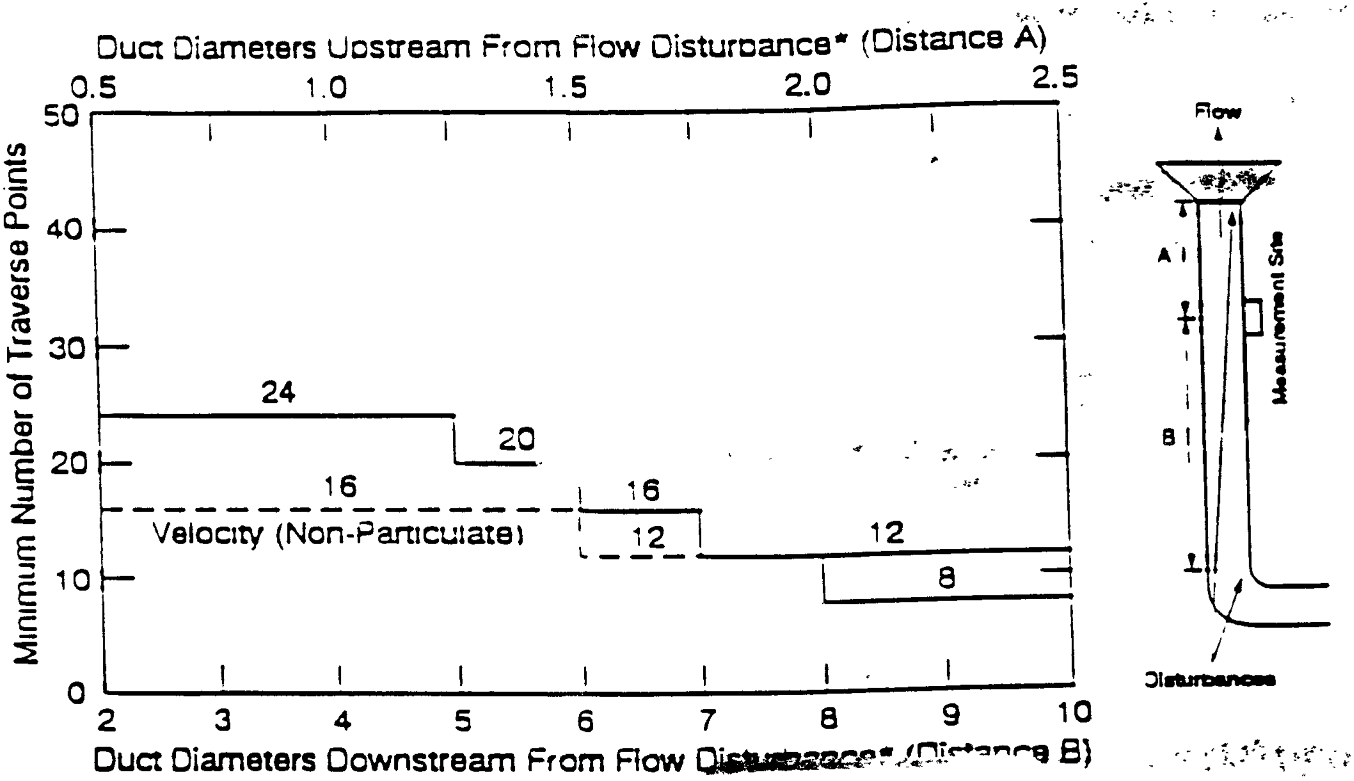
840 inches / Stack I.D. = 11.9 dd

Calculated By: GG



Schematic of Sampling Location

| Traverse Point Number | Fraction of Length | Length (inches) | Product of Columns 2 & 3 (To nearest 1/8") | Nipple Length (inches) | Traverse Point Location (Sum of Col. 4 & 5) |
|---|---|---|---|---|---|
| 1 | .021 | 70.5 | 1.48 | 6 | 7.4 |
| 2 | .067 | | 4.72 | | 10.7 |
| 3 | .118 | | 8.3 | | 14.3 |
| 4 | .177 | | 12.48 | | 18.4 |
| 5 | .250 | | 17.6 | | 23.6 |
| 6 | .356 | | 25.10 | | 31.0 |
| 7 | .644 | | 45.4 | | 51.4 |
| 8 | .750 | | 52.88 | | 58.8 |
| 9 | .823 | | 58 | | 64 |
| 10 | .882 | | 62.18 | | 68 |
| 11 | .933 | | 65.78 | | 71.7 |
| 12 | .979 | | 69.0 | | 75.0 |



* From Point of Any Type of Disturbance (Bend, Expansion, Contraction, etc)

## LOCATION OF TRAVERSE POINTS IN CIRCULAR DUCTS
(Fraction of Stack Diameter from Inside Wall to Traverse Point)

| Traverse Point Number on a Diameter | Number of Traverse Points on a Diameter | | | | |
|---|---|---|---|---|---|
| | 4 | 6 | 8 | 10 | 12 |
| 1 | 0.067 | 0.044 | 0.032 | 0.026 | 0.021 |
| 2 | 0.250 | 0.146 | 0.105 | 0.082 | 0.067 |
| 3 | 0.750 | 0.296 | 0.194 | 0.146 | 0.118 |
| 4 | 0.933 | 0.704 | 0.323 | 0.226 | 0.177 |
| 5 | | 0.854 | 0.677 | 0.342 | 0.250 |
| 6 | | 0.956 | 0.806 | 0.658 | 0.356 |
| 7 | | | 0.895 | 0.774 | 0.644 |
| 8 | | | 0.968 | 0.854 | 0.750 |
| 9 | | | | 0.918 | 0.823 |
| 10 | | | | 0.974 | 0.882 |
| 11 | | | | | 0.933 |
| 12 | | | | | 0.979 |

# GAS VELOCITY AND VOLUMETRIC FLOW RATE

Plant: Port Huron Date: ~~336~~ 829.9
Sampling Location: Clock Time: 33c
Run #: Operators: GC PS
Barometric Pressure, in. Hg: 29.70 Static Pressure, in. $H_2O$: -.23
Moisture, %: 25 Molecular wt., Dry: Pitot Tube, Cp: .84
Stack Dimension, in. Diameter or Side 1: 70.5 Side 2:
Wet Bulb, °F: Dry Bulb, °F:

| | Traverse Point Number | Velocity Head in. $H_2O$ | Stack Temp. °F | |
|---|---|---|---|---|
| | 1 | .19 | 154 | 0 |
| | 2 | .14 | 154 | 5cw |
| | 3 | .20 | 155 | 0 |
| | 4 | .18 | 155 | 3ccw |
| | 5 | .16 | 155 | 0 |
| | 6 | .19 | 155 | 3ccw |
| | 7 | .14 | 155 | 8ccw |
| | 8 | .16 | 155 | 10ccw |
| | 9 | .21 | 155 | 12ccw |
| | 10 | .20 | 153 | 16ccw |
| | 11 | .120 | 153 | 15ccw |
| | 12 | .16 | 150 | 10 |
| 13 | 1 | .14 | 150 | 21 cw |
| | 2 | .12 | 152 | 6cw |
| | 3 | .12 | 152 | 0 |
| | 4 | .10 | 153 | 0 |
| | 5 | .09 | 153 | 0 |
| | 6 | .11 | 154 | 0 |
| | 7 | .15 | 154 | 11 cw |
| | 8 | .19 | 154 | 14 cw |
| | 9 | .23 | 154 | 10 ccw |
| | 10 | .25 | 154 | 10 ccw |
| | 11 | .24 | 154 | 0 |
| | 12 | .18 | 137 | 7 cw |
| 21% | | | | |
| | | $\overline{\sqrt{\Delta P}}$ = .4073 | $\overline{Ts}$ = 152 | |

$Md = (0.44 \times \%CO_2) + (0.32 \times \%O_2) + (0.28 \times \%N_2)$

$Md = (0.44 \times \quad) + (0.32 \times \quad) + (0.28 \times \quad)$

$Md =$

$Ms = Md \times (1 - \frac{\% H_2O}{100}) + 18 (\frac{\% H_2O}{100})$

$Ms = (\quad) \times (1 - \frac{}{100}) + 18 (\frac{}{100})$

$Ms =$ ~~29.07~~ 26.2

$\overline{Ts} =$ °F = °R (°F + 460)

$Ps = Pb + \frac{S.P.}{13.6} = (\quad) + \frac{}{13.6}$

$Ps =$ 29.64 in. Hg

$\overline{\sqrt{\Delta P}} =$

$Vs = 85.49 \times Cp \times \overline{\sqrt{\Delta P}} \times \sqrt{\frac{Ts\ (°R)}{Ps \times Ms}}$

$Vs = 85.49 \times (\quad) \times (\quad) \times \sqrt{\quad}$

$Vs =$ ft/s

$As =$ $ft^2$

$Qs = Vs \times As \times 60$ s/m

$Qs = \quad \times \quad \times 60$

$Qs =$ acfm

$Qs_{std} = Qs \times 17.647 \times \frac{Ps}{Ts} \times (1 - \frac{\% H_2O}{100})$

$Qs_{std} = \quad \times 17.647 \times \frac{}{} \times (1 - \frac{}{100})$

6.7° $Qs_{std} =$ dscfm

.1687

13659

# FIELD DATA SHEET

K ≈ 5.45

**Plant:** Port Huron
**Sampling Location** #3 Kiln Outlet
**Run Number:** M2303 **Date:** 831-98
**Pretest Leak Rate:** .005 **cfm @** 15 **in. Hg.**
**Pretest Leak Check: Pitot:** ✓ **Orsat:** NA

**Sample Type:** M23 **Operator:** GGray/P Siegel
**Pbar:** 29.70 **Ps:** - .23
**CO2:** **O2:**
**Probe Length/Type:** 7' Glass **Pitot #:** 7C
**Stack Diameter:** 70.5 **As:** 27.10

**Nozzle ID:** .318 **Thermocouple #:** 7C
**Assumed Bws:** 30 **Filter #:**
**Meter Box #:** 10 **Y:** 1.021 **ΔH@:** 1.92
**Post-Test Leak Rate:** .001 **cfm @** 16 **in. Hg.**
**Post-Test Leak Check: Pitot:** ✓ **Orsat:** NA

| Traverse Point Number | Sampling Time (min) | Clock Time (24-hour clock) | Gas Meter Reading (Vm) ft³ | Velocity Head (Δp) in H2O | Orifice Pressure Differential (ΔH) in H2O | | Stack Temp. (Ts) | Temperature °F | | Impinger Temp. °F | Dry Gas Meter Temp. | | Pump Vacuum (in. Hg) | COND Temp |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | Desired | Actual | | Probe | Filter | | Inlet (Tm in°F) | Outlet (Tm out°F) | | |
| | 0 | 1750 | 945.031 | | | | | | | | | | | |
| 1 | 7.5 | 1757 | 950.5 | .33 | 1.69 | 1.7 | 151 | 253 | 255 | 61 | 76 | 75 | 6 | 52 |
| 2 | 15 | 1806 | 955.3 | .30 | 1.53 | 1.5 | 155 | 257 | 258 | 50 | 79 | 77 | 6 | 51 |
| 3 | 22.5 | 1813 | 960.3 | .31 | 1.59 | 1.6 | 156 | 251 | 260 | 56 | 84 | 79 | 7 | 52 |
| 4 | 30 | 1821 | 965.3 | .30 | 1.54 | 1.5 | 157 | 253 | 258 | 51 | 86 | 81 | 9 | 55 |
| 5 | 37.5 | 1828 | 969.7 | .28 | 1.4 | 1.4 | 157 | 262 | 257 | 53 | 86 | 84 | 10 | 54 |
| 6 | 45.0 | 1836 | 974.2 | .25 | 1.3 | 1.3 | 157 | 259 | 258 | 53 | 86 | 81 | 10 | 55 |
| 7 | 52.5 | 1843 | 978.6 | .23 | 1.2 | 1.2 | 157 | 253 | 256 | 55 | 86 | 84 | 10 | 56 |
| 8 | 60 | 1852 | 982.4 | .17 | .88 | .88 | 157 | 255 | 259 | 55 | 87 | 85 | 9 | 51 |
| 9 | 67.5 | 1859 | 986.3 | .18 | .93 | .93 | 157 | 256 | 257 | 55 | 89 | 86 | 10 | 55 |
| 10 | 75 | 1909 | 990.2 | .18 | .93 | .93 | 157 | 259 | 256 | 56 | 90 | 88 | 11 | 56 |
| 11 | 82.5 | 1915 | 994.0 | .16 | .83 | .83 | 157 | 257 | 255 | 52 | 91 | 84 | 11 | 53 |
| 12 | 90 | 1922 | 997.6 | .16 | .83 | .83 | 157 | 255 | 255 | 49 | 2 | 90 | 11 | 48 |
| | | 1924 | | | | | | | | | | | | |
| 1 | 97.5 | 1932 | [illegible]1.2 | .16 | .83 | 33 | 157 | 256 | 258 | 48 | 4 | 92 | 11 | 47 |
| 2 | 105 | 1939 | [illegible]4.9 | .16 | .83 | 3 | 157 | 255 | 257 | 48 | 5 | 93 | 12 | 50 |
| 3 | 112.5 | 2000 | 009.1 | .20 | 1.0 | 1 | 157 | 251 | 255 | 98 | 6 | 94 | 14 | 49 |
| 4 | 120 | 2007 | 013.1 | .18 | .94 | 4 | 157 | 250 | 255 | 46 | 6 | 94 | 14 | 47 |
| 5 | 127.5 | 2015 | 017.6 | .28 | .94 | 4 | 157 | 253 | 259 | 45 | 5 | 93 | 15 | 46 |
| 6 | 135 | 2043 | 021.6 | .24 | 1.26 | 1.3 | 157 | 255 | 257 | 44 | 6 | 94 | 6 | 53 |
| 7 | 1425 | 2050 | 026.1 | .25 | 1.31 | 1.3 | 157 | 257 | 258 | 45 | 97 | 96 | 6 | 52 |
| 8 | 150 | 2058 | 030.9 | .26 | 1.4 | 1.4 | 156 | 258 | 257 | 46 | 99 | 97 | 6 | 50 |
| 9 | 157.5 | 2106 | 036.5 | .35 | 1.86 | 1.9 | 157 | 255 | 260 | 48 | 102 | 99 | 10 | 51 |
| 10 | 165 | 2113 | 041.9 | .33 | 1.75 | 1.8 | 157 | 255 | 257 | 48 | 103 | 100 | 10 | 50 |
| 11 | 1725 | 2121 | 047.3 | .34 | 1.8 | 1.8 | 155 | 255 | 256 | 49 | 102 | 100 | 11 | 51 |
| 12 | 180 | 2128 | 052.979 | .33 | 1.76 | 1.8 | 154 | 256 | 259 | 50 | 102 | 101 | 12 | 52 |
| | | | | | | | | | | | | | | |

STOP 1943
1956 START
STOP 2020

Leak Ck
.017 @ 7"
.017 @ 8"
.002 @ 17"

# SAMPLE RECOVERY DATA

PLANT Huron Lime Company Run No. M23-O-3

DATE 8-31-98 Sample Box No. N-5 Job No. S509.000

SAMPLE LOCATION Kiln #3 Scrubber Outlet Filter No. M23-0-3-1 Glass

TRAIN PREPARER TA

SAMPLE RECOVERY PERSON TA

COMMENTS

**FRONT HALF**

Acetone Container No. M23-0-3-2 Liquid Level Marked ✓ Sealed ✓

Filter Container No. M23-0-3-1 Sealed ✓

Description of Filter Heavy Yellowish Grey tint

Samples Stored and Locked ✓

Front 1/2 Toluene

~~**BACK HALF/MOISTURE**~~

Container No. M23-0-3-3

Liquid Level Marked ✓ Sealed ✓

d to
se,
other
imp. due
high
$H_2O$.

| IMP. NO. | CONTENTS | INITIAL VOL (ml) | WEIGHT (grams) | | |
|---|---|---|---|---|---|
| | | | INITIAL | FINAL | NET |
| 1 | RAD Trap | 40g | 304.6 | 329.8 | 25.2 |
| 2 | Empty | 2nd Imp.<br>1st imp. | ~~462.2~~<br>463.0 | ~~942.9~~<br>1147.7 | ~~480.7~~<br>684.7 |
| 3 | HPLC $H_2O$ | 100 | 576.1 | 575.1 | - 1.0 |
| 4 | HPLC $H_2O$ | 100 | 718.3 | 716.9 | - 1.4 |
| 5 | Empty | — | 518.4 | 519.7 | 1.3 |
| 6 | Silica Gel | 200g | 888.7 | 924.2 | 35.5 |
| TOTAL | | | | | 1225.0 |

**Description of Impinger Catch:** Clear S.G. 85% Spent

# FIELD DATA SHEET

Plant: PORT HURON
Sampling Location #3 KILN OUTLET
Run Number: F0 Date: 8-31-94
Pretest Leak Rate: See Bw. cfm @ 15 in. Hg.
Pretest Leak Check: Pitot: ✓ Orsat: NA

Sample Type: m23 Operator: GGray/PSimcl
Pbar: 29.70 Ps: -.23
CO2: O2:
Probe Length/Type: 7' Glas Pitot #: 7C
Stack Diameter: 70.5 As: 27.10

Nozzle ID: .310 Thermocouple #: 7C
Assumed Bws: 30 Filter #: —
Meter Box #: 10 Y: 1.021 ΔH@: 1.92
Post-Test Leak Rate: — cfm @ — in. Hg.
Post-Test Leak Check: Pitot: — Orsat: NA

| Traverse Point Number | Sampling Time (min) | Clock Time (24-hour clock) | Gas Meter Reading (Vm) ft$^3$ | Velocity Head (Δp) in H2O | Orifice Pressure Differential (ΔH) in H2O | | Stack Temp (Ts) | Temperature °F | | Impinger Temp. °F | Dry Gas Meter Temp. | | Pump Vacuum (in. Hg) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | Desired | Actual | | Probe | Filter | | Inlet (Tm in°F) | Outlet (Tm out°F) | |
| | | | 052.926 | | | | | | | | | | |
| | .002 | 15" | 053.001 | — | — | - | — | 255 | 259 | 50 | — | — | 15 |
| | .002 | 15" | .053 110 | | | | | | | | | | |
| | .002 | 15" | 053.215 | | | | | | | | | | |
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49

ΔVm= $\sqrt{\Delta p}$= $\overline{\Delta H}$= $\overline{Ts}$= $\overline{Tm}$=

# SAMPLE RECOVERY DATA

PLANT Huron Lime Company Run No. M23-FB-3

DATE 8-31-98 Sample Box No. 104-1 Job No. S509.000

SAMPLE LOCATION Kiln #3 Scrubber Outlet Filter No. M23-FB-3-1 Glass

TRAIN PREPARER TA

SAMPLE RECOVERY PERSON TA

COMMENTS —

**FRONT HALF**

Acetone Container No. M23-FB-3-2 Liquid Level Marked ✓ Sealed ✓

Filter Container No. M23-FB-3-1 Sealed ✓

Description of Filter Clean

Samples Stored and Locked ✓

Front 1/2 Toluene

~~**BACK HALF/MOISTURE**~~

Container No. M23-FB-3-3 ✓

Liquid Level Marked ✓ Sealed ✓

| IMP. NO. | CONTENTS | INITIAL VOL (ml) | WEIGHT (grams) | | |
|---|---|---|---|---|---|
| | | | INITIAL | FINAL | NET |
| 1 | XAD Trap | 40g | 309.3 | 309.3 | 0 |
| 2 | Empty | — | 496.0 | 496.0 | 0 |
| 3 | HPLC $H_2O$ | 100 | 601.2 | 601.3 | 0.1 |
| 4 | HPLC $H_2O$ | 100 | 603.0 | 603.0 | 0 |
| 5 | Empty | — | 518.0 | 518.0 | 0 |
| 6 | Silica Gel | 200g | 904.2 | 904.4 | 0.2 |
| TOTAL | | | | | 0.3 |

**Description of Impinger Catch:** Clear

# Appendix A.2

# Raw Field Data

# CEMs Summary & Strip Charts

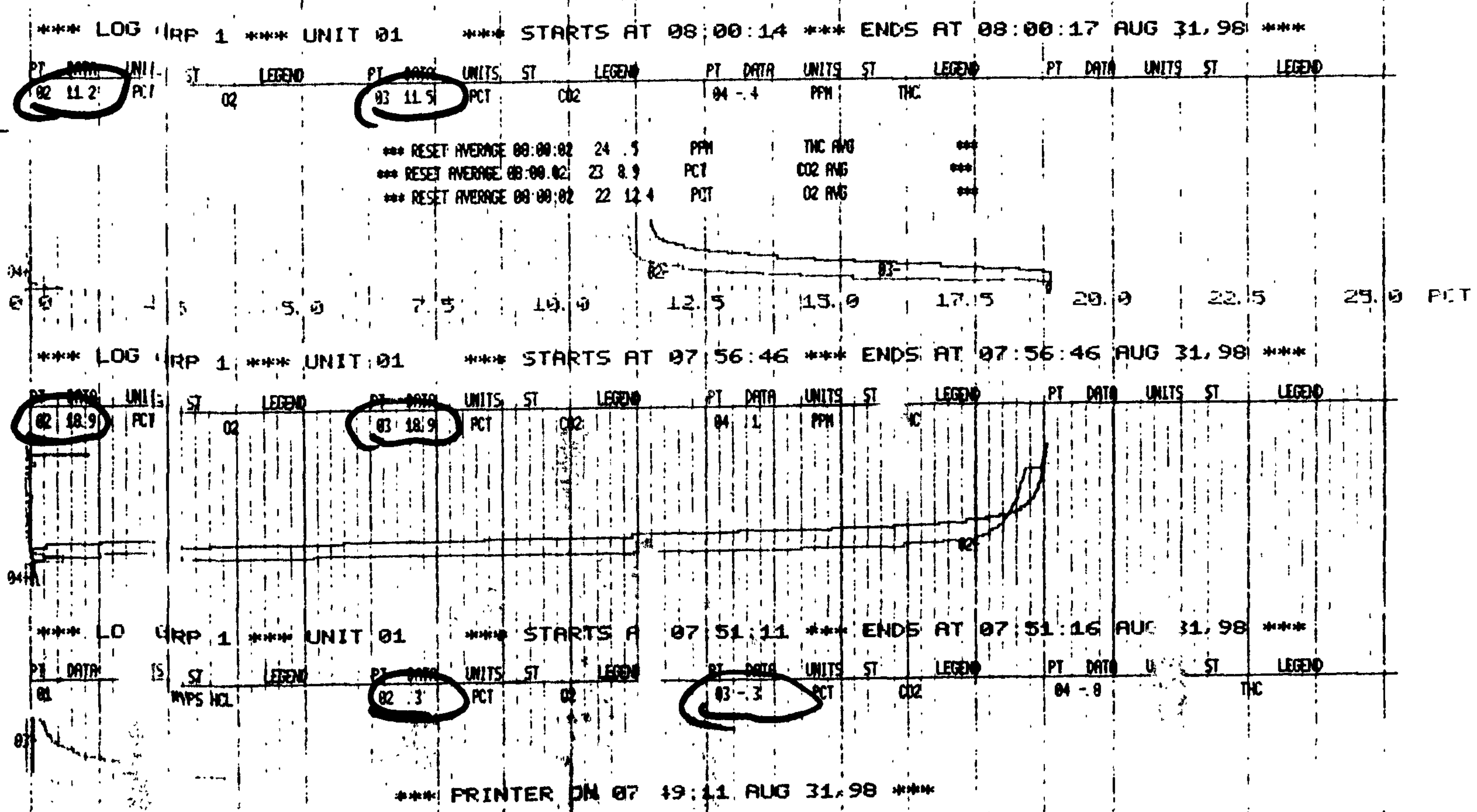
### HCl Emission Measurements from a Rotary Kiln
### Huron Lime Company
### Huron, Ohio

| Time | Date | Inlet/Outlet | THC<br>ppm | O2<br>% | CO2<br>% |
|---|---|---|---|---|---|
| | | | | | |
| 1749-1804 | 8/31/98 | Outlet | 1.1 | 6.6 | 21.4 |
| 1804-1819 | 8/31/98 | | 1.1 | 6.6 | 21.3 |
| 1819-1834 | 8/31/98 | | 1.1 | 6.6 | 21.3 |
| 1834-1849 | 8/31/98 | | 1.1 | 6.7 | 21.3 |
| 1849-1904 | 8/31/98 | | 1.0 | 6.7 | 21.1 |
| 1904-1919 | 8/31/98 | | 1.0 | 6.7 | 21.1 |
| 1919-1934 | 8/31/98 | | 1.0 | 6.6 | 21.2 |
| 1934-1949 | 8/31/98 | | 1.1 | 6.5 | 21.3 |
| 1949-2004 | 8/31/98 | | 0.9** | 6.5 | 21.3 |
| 2004-2019 | 8/31/98 | | 0.7 | 6.5 | 21.3 |
| 2019-2034 | 8/31/98 | | 0.5 | 6.6 | 21.4 |
| 2034-2049 | 8/31/98 | | 0.2 | 6.9 | 20.9 |
| 2049-2104 | 8/31/98 | | 0.5 | 7.0 | 20.5 |
| 2104-2119 | 8/31/98 | | 0.3 | 7.0 | 20.3 |
| 2119-2134 | 8/31/98 | | 0.5 | 7.0 | 20.1 |
| | | | **0.8** | **6.7** | **21.1** |

*No HCl data available, analyzer down

**It is believed that THC analyzer started to drift down at this point ending at half actual value.

$CO_2/O_2$
11.0/4.1



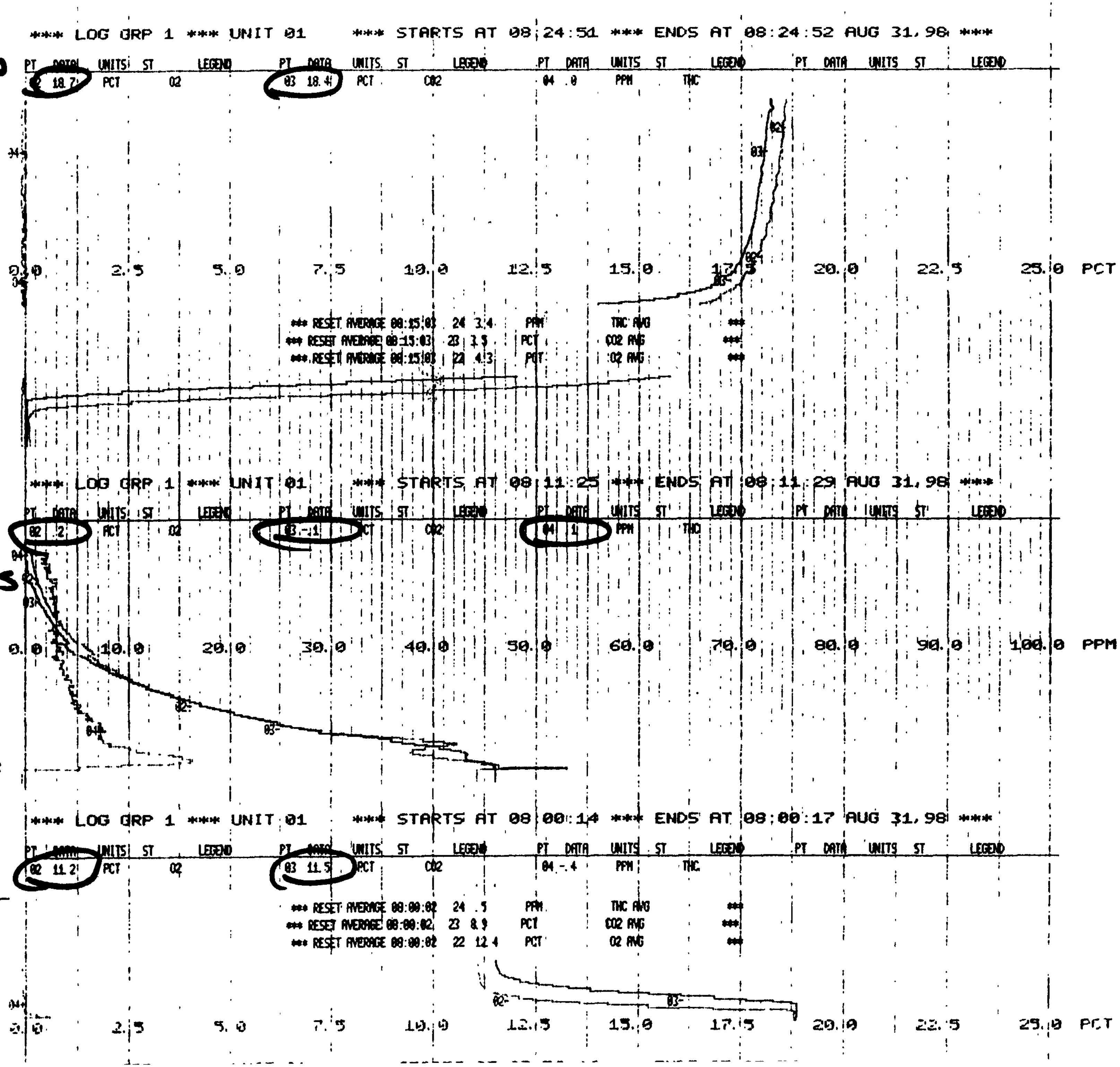
```
*** LOG GRP 1 *** UNIT 01    *** STARTS AT 08:00:14 *** ENDS AT 08:00:17 AUG 31,98 ***
PT DATA UNITS ST   LEGEND    PT DATA UNITS ST   LEGEND    PT DATA UNITS ST   LEGEND    PT DATA UNITS ST   LEGEND
02 11.2  PCT       O2        03 11.5  PCT       CO2       04 -.4  PPM        THC
AUG 31,98
        *** RESET AVERAGE 08:00:02  24  .5   PPM    THC AVG  ***
        *** RESET AVERAGE 08:00.02  23  8.9  PCT    CO2 AVG  ***
        *** RESET AVERAGE 08:00:02  22 12.4  PCT    O2 AVG   ***
0.0    2.5    5.0    7.5    10.0    12.5    15.0    17.5    20.0    22.5    25.0 PCT
*** LOG GRP 1 *** UNIT 01    *** STARTS AT 07:56:46 *** ENDS AT 07:56:46 AUG 31,98 ***
PT DATA UNITS ST   LEGEND    PT DATA UNITS ST   LEGEND    PT DATA UNITS ST   LEGEND    PT DATA UNITS ST   LEGEND
02 18.9  PCT       O2        03 18.9  PCT       CO2       04 1    PPM        THC
*** LO  GRP 1 *** UNIT 01    *** STARTS A  07:51:11 *** ENDS AT 07:51:16 AUC 31,98 ***
PT DATA UNITS ST   LEGEND    PT DATA UNITS ST   LEGEND    PT DATA UNITS ST   LEGEND    PT DATA UNITS ST   LEGEND
01              NVPS HCL     02 .3    PCT       O2        03 -.3  PCT        CO2       04 -.8              THC
        *** PRINTER ON 07 49:11 AUG 31,98 ***
```

$CO_2/O_2$
19.0/19.2

Zero
RACK

HURON LIME
8/31/98

19.0/19.2



```
*** LOG GRP 1 *** UNIT 01    *** STARTS AT 08:24:51 *** ENDS AT 08:24:52 AUG 31,98 ***
PT DATA UNITS ST   LEGEND    PT DATA UNITS ST   LEGEND    PT DATA UNITS ST   LEGEND    PT DATA UNITS ST   LEGEND
02 18.7 PCT        02        03 18.4 PCT        CO2       04 .0   PPM        THC

1A  0.0   2.5   5.0   7.5   10.0   12.5   15.0   17.5   20.0   22.5   25.0 PCT

*** RESET AVERAGE 08:15:03  24  3.4   PPM   THC AVG  ***
*** RESET AVERAGE 08:15:03  23  1.5   PCT   CO2 AVG  ***
*** RESET AVERAGE 08:15:03  22  4.3   PCT   02 AVG   ***
```

zero

STACK BIAS

```
*** LOG GRP 1 *** UNIT 01    *** STARTS AT 08:11:25 *** ENDS AT 08:11:29 AUG 31,98 ***
PT DATA UNITS ST   LEGEND    PT DATA UNITS ST   LEGEND    PT DATA UNITS ST   LEGEND    PT DATA UNITS ST   LEGEND
02 .2   PCT        02        03 -.1 PCT         CO2       04 .1   PPM        THC

1B  0.0   10.0   20.0   30.0   40.0   50.0   60.0   70.0   80.0   90.0   100.0 PPM

01
GRP N/A
 12.00 IN/HR
08:00:11
```

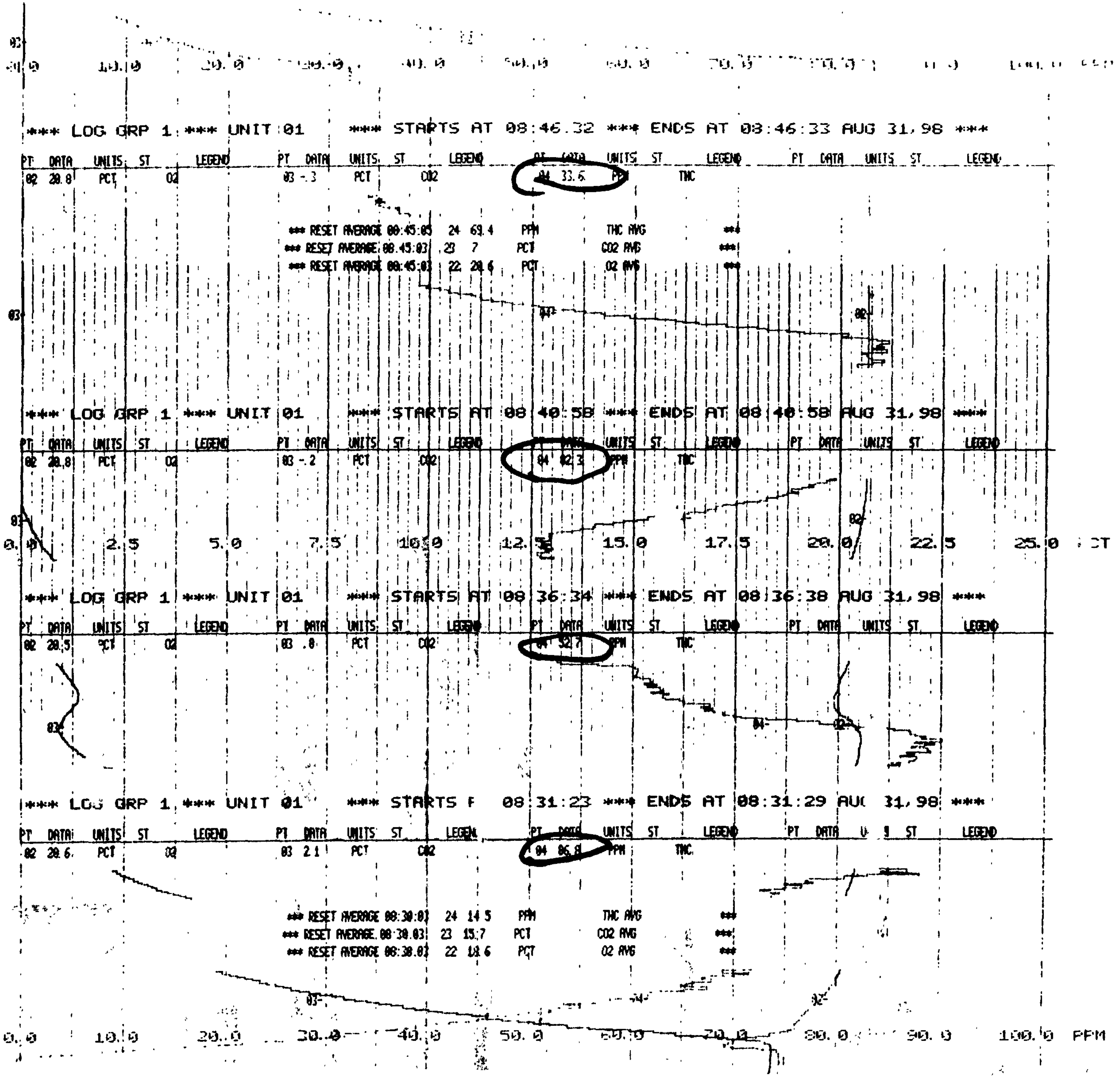
CO2/O2

11.0/4.1

```
*** LOG GRP 1 *** UNIT 01    *** STARTS AT 08:00:14 *** ENDS AT 08:00:17 AUG 31,98 ***
PT DATA UNITS ST   LEGEND    PT DATA UNITS ST   LEGEND    PT DATA UNITS ST   LEGEND    PT DATA UNITS ST   LEGEND
02 11.2 PCT        02        03 11.5 PCT        CO2       04 -.4  PPM        THC
AUG 31,98

*** RESET AVERAGE 08:00:02  24  .5    PPM   THC AVG  ***
*** RESET AVERAGE 08:00:02  23  8.9   PCT   CO2 AVG  ***
*** RESET AVERAGE 08:00:02  22  12.4  PCT   02 AVG   ***

1A  0.0   2.5   5.0   7.5   10.0   12.5   15.0   17.5   20.0   22.5   25.0 PCT
```



THC
30.0

```
*** LOG GRP 1 *** UNIT 01    **** STARTS AT 08:46.32 *** ENDS AT 08:46:33 AUG 31,98 ***
PT  DATA  UNITS  ST   LEGEND   PT  DATA  UNITS  ST   LEGEND   PT  DATA  UNITS  ST   LEGEND   PT  DATA  UNITS  ST   LEGEND
02  20.8  PCT         O2       03  -.3   PCT         CO2      04  33.6  PPM         THC

          *** RESET AVERAGE 08:45:05  24  69.4   PPM    THC AVG  ***
          *** RESET AVERAGE 08.45:03  23  7      PCT    CO2 AVG  ***
          *** RESET AVERAGE 08:45:03  22  20.6   PCT    O2 AVG   ***
```

THC
85.4

```
*** LOG GRP 1 *** UNIT 01    **** STARTS AT 08 40:58 *** ENDS AT 08:40:58 AUG 31,98 ***
PT  DATA  UNITS  ST   LEGEND   PT  DATA  UNITS  ST   LEGEND   PT  DATA  UNITS  ST   LEGEND   PT  DATA  UNITS  ST   LEGEND
02  20.8  PCT         O2       03  -.2   PCT         CO2      04  82.3  PPM         THC
```

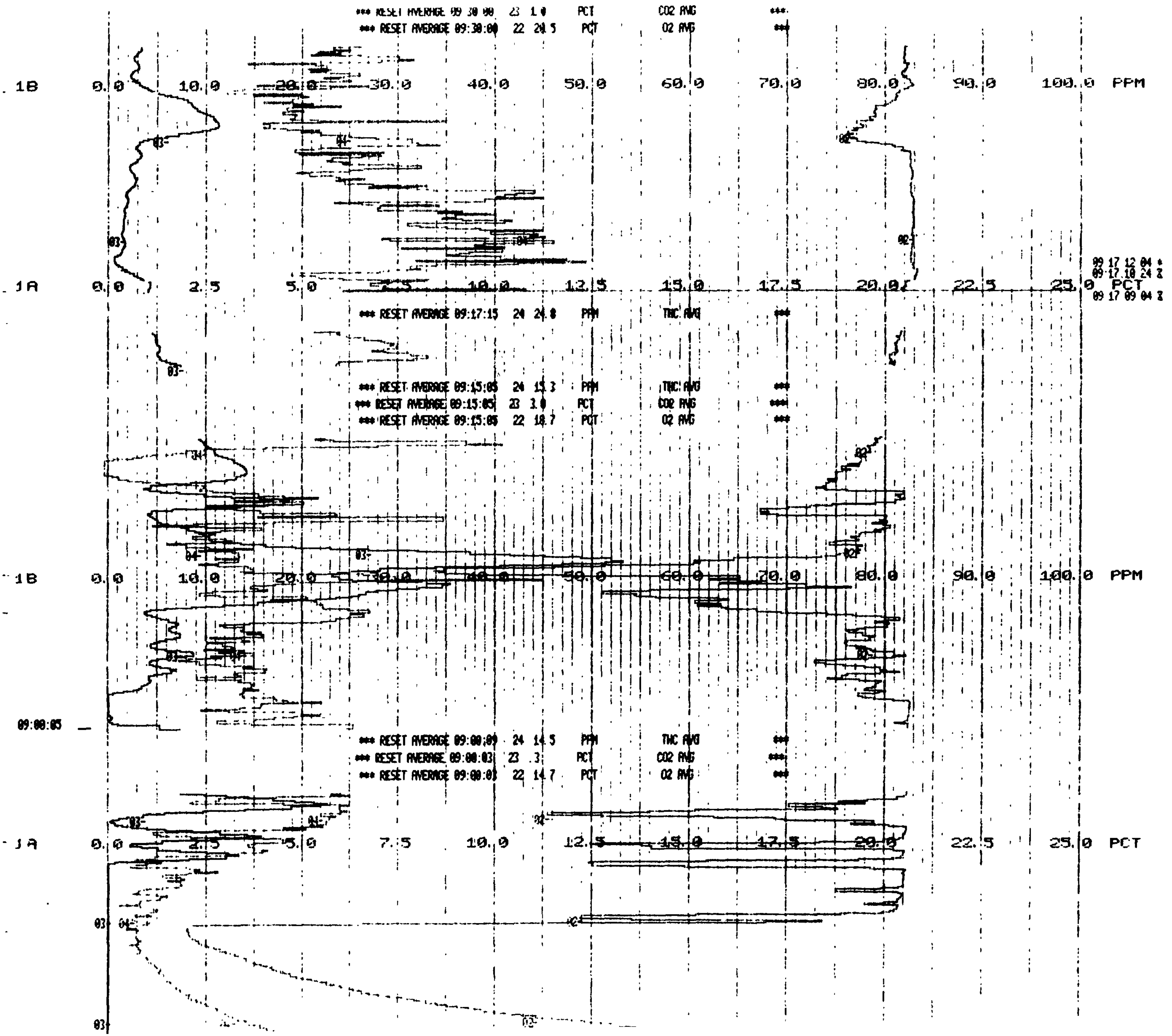
THC
50.4

```
*** LOG GRP 1 *** UNIT 01    **** STARTS AT 08 36:34 *** ENDS AT 08 36:38 AUG 31,98 ***
PT  DATA  UNITS  ST   LEGEND   PT  DATA  UNITS  ST   LEGEND   PT  DATA  UNITS  ST   LEGEND   PT  DATA  UNITS  ST   LEGEND
02  20.5  PCT         O2       03  .8    PCT         CO2      04  52.7  PPM         THC
```

TH~~C~~ NC
~~85.4~~

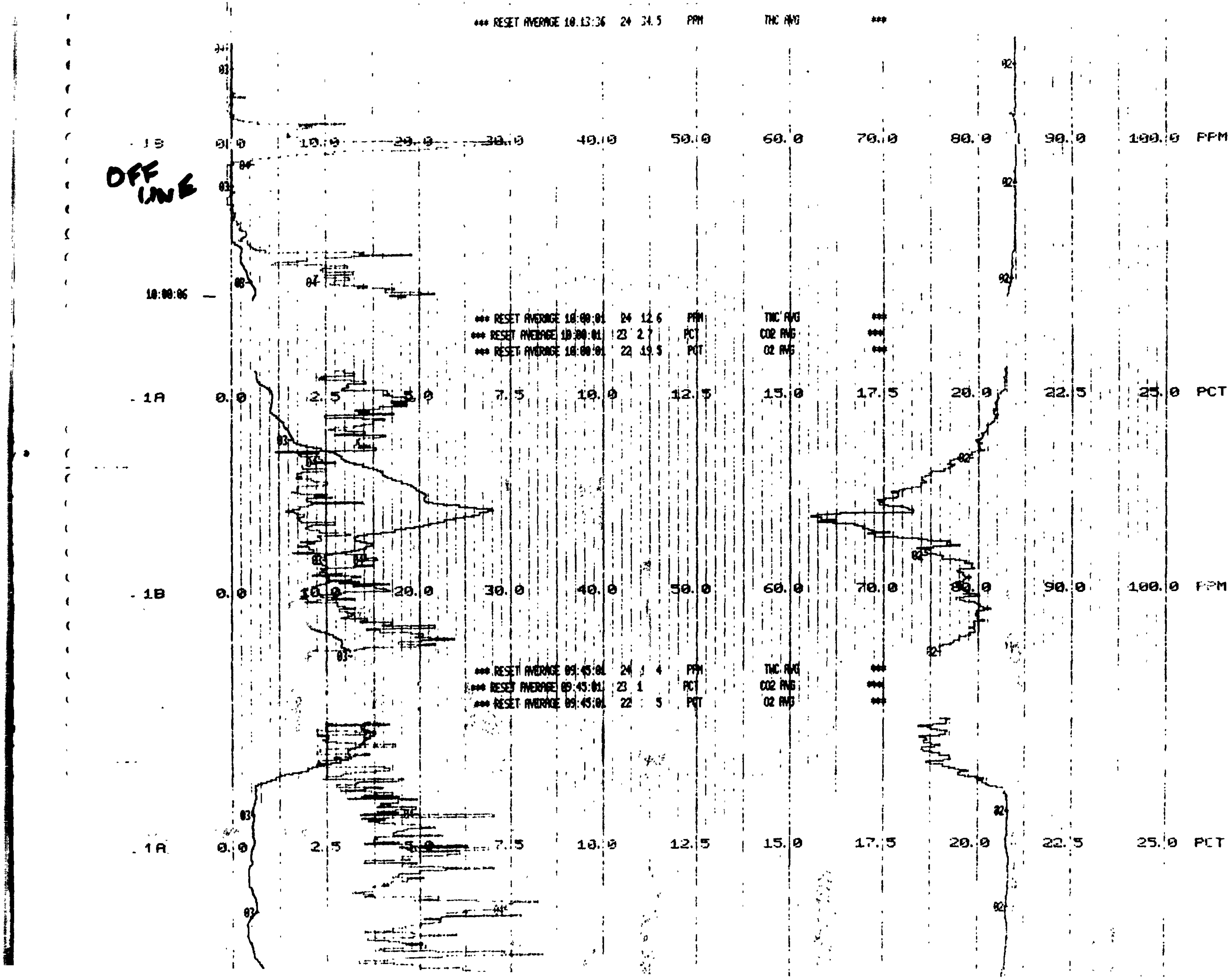
```
*** LOG GRP 1 *** UNIT 01    **** STARTS F  08 31:23 *** ENDS AT 08:31:29 AU( 31,98 ***
PT  DATA  UNITS  ST   LEGEND   PT  DATA  UNITS  ST   LEGEN    PT  DATA  UNITS  ST   LEGEND   PT  DATA  U  S  ST   LEGEND
02  20.6  PCT         O2       03  2.1   PCT         CO2      04  86.8  PPM         THC.

          *** RESET AVERAGE 08:30:03  24  14.5   PPM    THC AVG  ***
          *** RESET AVERAGE.08:30.03  23  15.7   PCT    CO2 AVG  ***
          *** RESET AVERAGE 08:30.03  22  18.6   PCT    O2 AVG   ***
```



```
*** RESET AVERAGE 09 30 00   23  1.0     PCT          CO2 AVG         ***
*** RESET AVERAGE 09:30:00   22  20.5    PCT          O2 AVG          ***
*** RESET AVERAGE 09:17:15   24  24.0    PPM          THC AVG         ***
*** RESET AVERAGE 09:15:05   24  15.3    PPM          THC AVG         ***
*** RESET AVERAGE 09:15:05   23  3.0     PCT          CO2 AVG         ***
*** RESET AVERAGE 09:15:05   22  18.7    PCT          O2 AVG          ***
*** RESET AVERAGE 09:00:09   24  14.5    PPM          THC AVG         ***
*** RESET AVERAGE 09:00:03   23  .3      PCT          CO2 AVG         ***
*** RESET AVERAGE 09:00:03   22  14.7    PCT          O2 AVG          ***
```

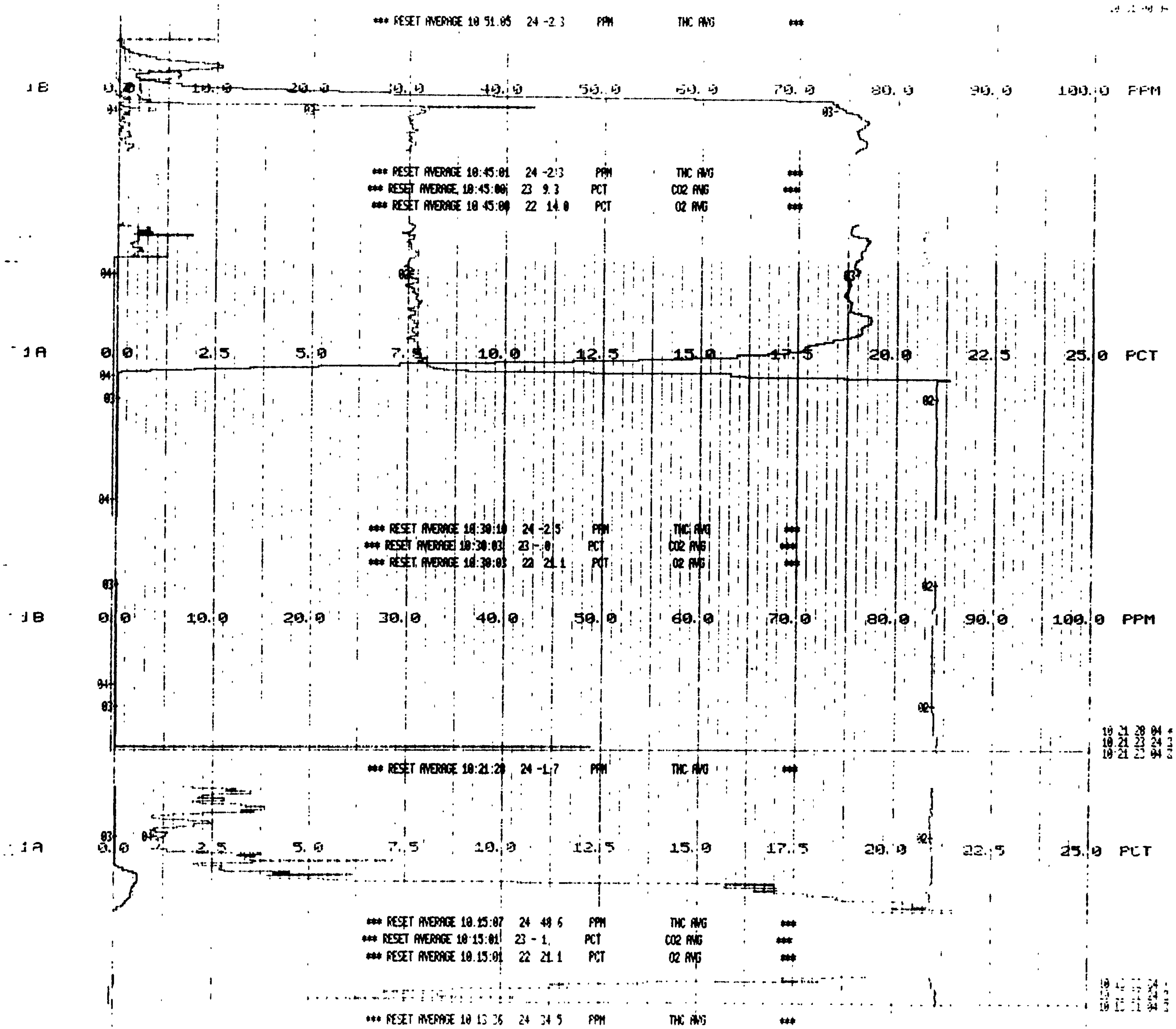
OFF LINE



```
*** RESET AVERAGE 10.13:36   24  34.5     PPM          THC AVG           ***
```

```
*** RESET AVERAGE 10:00:01   24  12.6     PPM          THC AVG           ***
*** RESET AVERAGE 10:00:01   23  2.7      PCT          CO2 AVG           ***
*** RESET AVERAGE 10:00:01   22  19.5     PCT          O2 AVG            ***
```

```
*** RESET AVERAGE 09:45:01   24   4       PPM          THC AVG           ***
*** RESET AVERAGE 09:45:01   23  1        PCT          CO2 AVG           ***
*** RESET AVERAGE 09:45:01   22   5       PCT          O2 AVG            ***
```



```
*** RESET AVERAGE 10 51.05   24 -2.3     PPM          THC AVG          ***

*** RESET AVERAGE 10:45:01   24 -2.3     PPM          THC AVG          ***
*** RESET AVERAGE 10:45:00   23  9.3     PCT          CO2 AVG          ***
*** RESET AVERAGE 10 45:00   22  14.0    PCT          O2 AVG           ***

*** RESET AVERAGE 10:30:10   24 -2.5     PPM          THC AVG          ***
*** RESET AVERAGE 10:30:03   23 -.0      PCT          CO2 AVG          ***
*** RESET AVERAGE 10:30:03   22  21.1    PCT          O2 AVG           ***

*** RESET AVERAGE 10:21:20   24 -1.7     PPM          THC AVG          ***

*** RESET AVERAGE 10.15:07   24  48 6    PPM          THC AVG          ***
*** RESET AVERAGE 10 15:01   23 - 1.     PCT          CO2 AVG          ***
*** RESET AVERAGE 10.15:01   22  21.1    PCT          O2 AVG           ***

*** RESET AVERAGE 10 13 36   24  34 5    PPM          THC AVG          ***
```

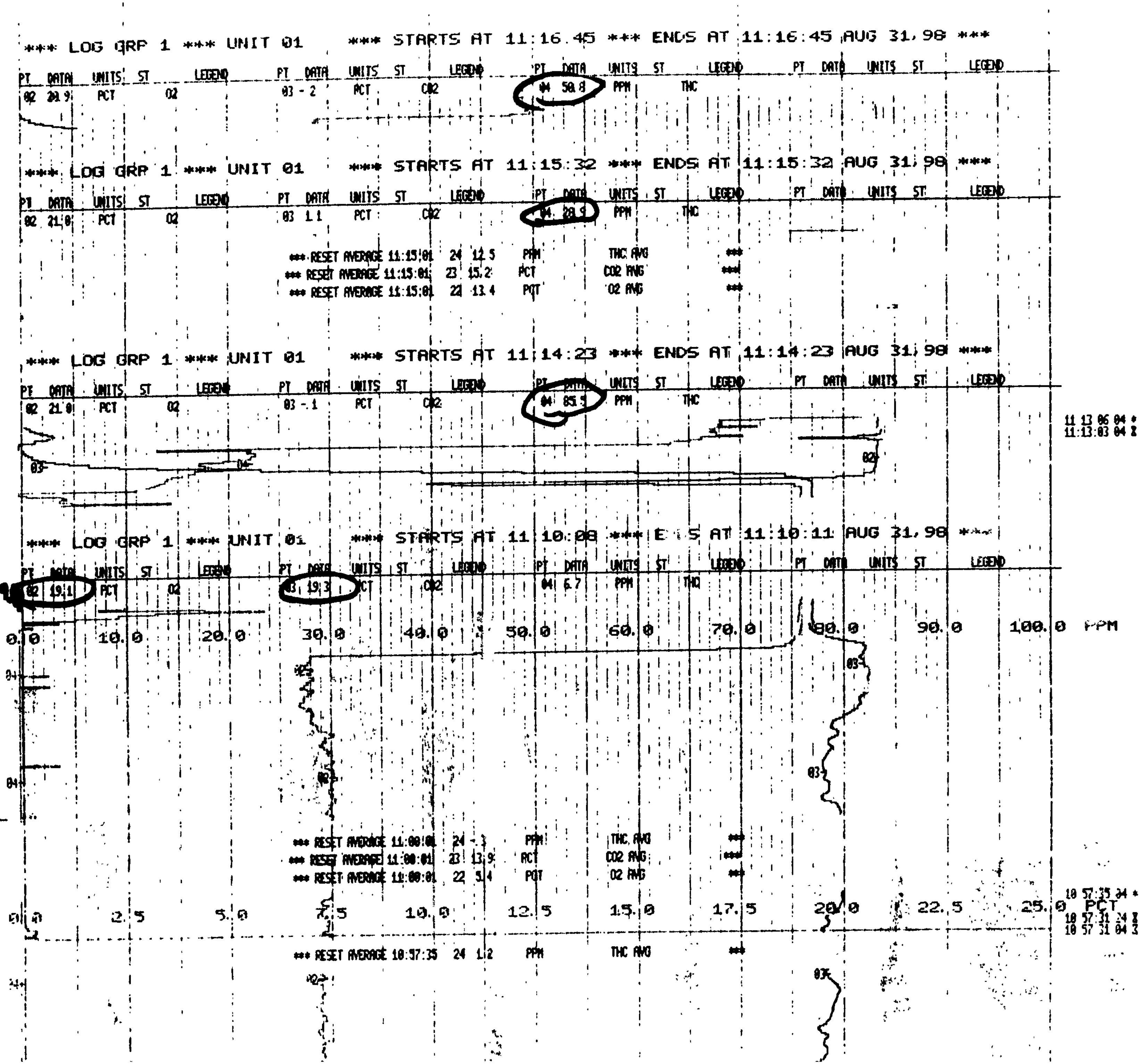
THC 50.1

THC 30.0

THC 85.4

CO2/O2 New 190/19 Bias

*Sample Pressure Change



```
*** LOG GRP 1 *** UNIT 01    *** STARTS AT 11:16.45 *** ENDS AT 11:16:45 AUG 31, 98 ***
PT DATA UNITS ST  LEGEND   PT DATA UNITS ST  LEGEND   PT DATA UNITS ST  LEGEND   PT DATA UNITS ST  LEGEND
02 20.9  PCT      O2       03 - 2  PCT      CO2      04 50.8  PPM      THC

*** LOG GRP 1 *** UNIT 01    *** STARTS AT 11:15:32 *** ENDS AT 11:15:32 AUG 31, 98 ***
PT DATA UNITS ST  LEGEND   PT DATA UNITS ST  LEGEND   PT DATA UNITS ST  LEGEND   PT DATA UNITS ST  LEGEND
02 21.0  PCT      O2       03 1.1  PCT      CO2      04 28.9  PPM      THC

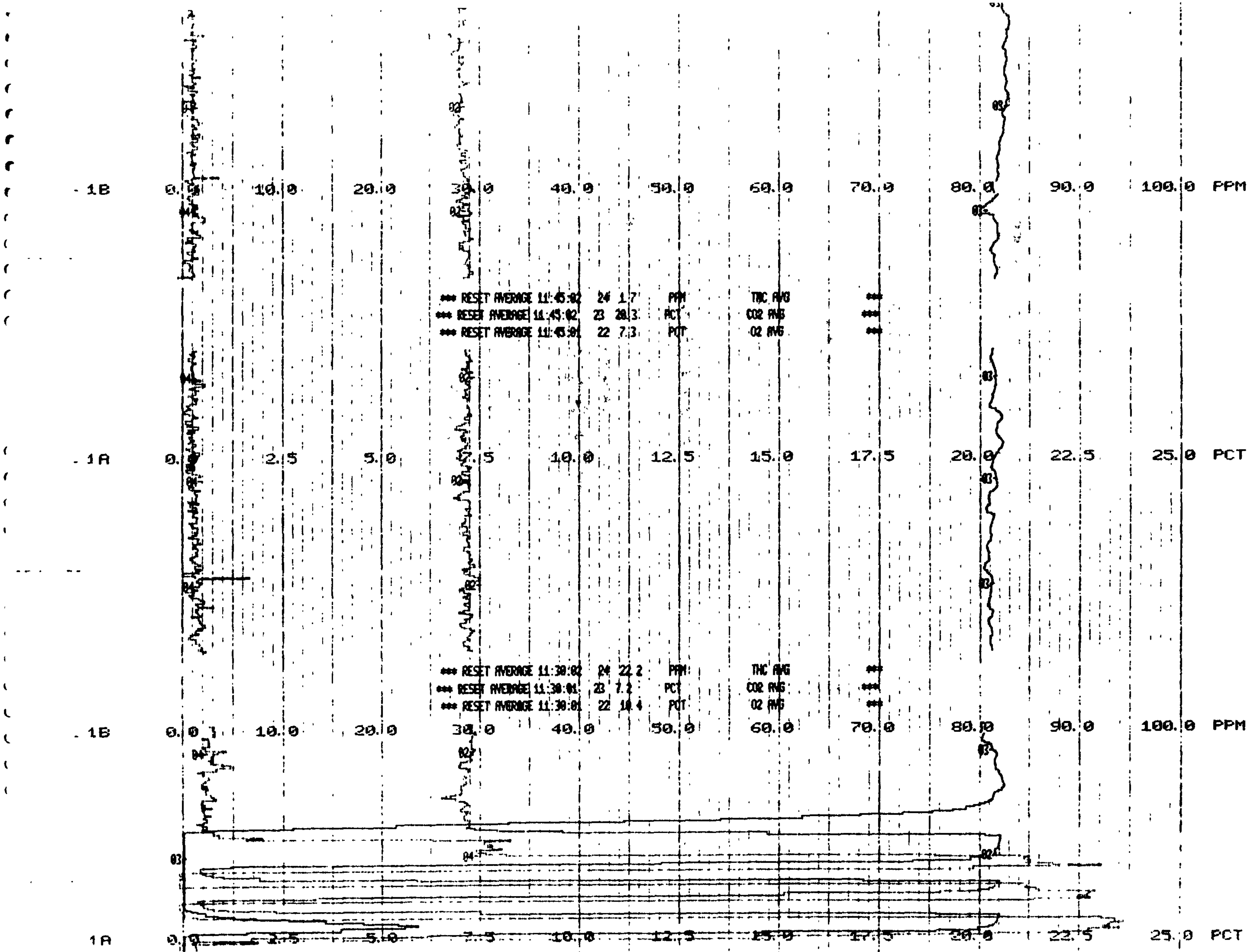
        *** RESET AVERAGE 11:15:01   24  12.5   PPM    THC AVG   ***
        *** RESET AVERAGE 11:15:01   23  15.2   PCT    CO2 AVG   ***
        *** RESET AVERAGE 11:15:01   22  13.4   PCT    O2 AVG    ***

*** LOG GRP 1 *** UNIT 01    *** STARTS AT 11:14:23 *** ENDS AT 11:14:23 AUG 31, 98 ***
PT DATA UNITS ST  LEGEND   PT DATA UNITS ST  LEGEND   PT DATA UNITS ST  LEGEND   PT DATA UNITS ST  LEGEND
02 21.0  PCT      O2       03 -.1  PCT      CO2      04 85.5  PPM      THC

*** LOG GRP 1 *** UNIT 01    *** STARTS AT 11 10:08 *** E  S AT 11:10:11 AUG 31, 98 ***
PT DATA UNITS ST  LEGEND   PT DATA UNITS ST  LEGEND   PT DATA UNITS ST  LEGEND   PT DATA UNITS ST  LEGEND
02 19.1  PCT      O2       03 19.3 PCT      CO2      04 6.7   PPM      THC

        *** RESET AVERAGE 11:00:01   24 -.3   PPM    THC AVG   ***
        *** RESET AVERAGE 11:00:01   23 13.9   PCT    CO2 AVG   ***
        *** RESET AVERAGE 11:00:01   22  5.4   PCT    O2 AVG    ***

        *** RESET AVERAGE 10:57:35   24  1.2   PPM    THC AVG   ***
```

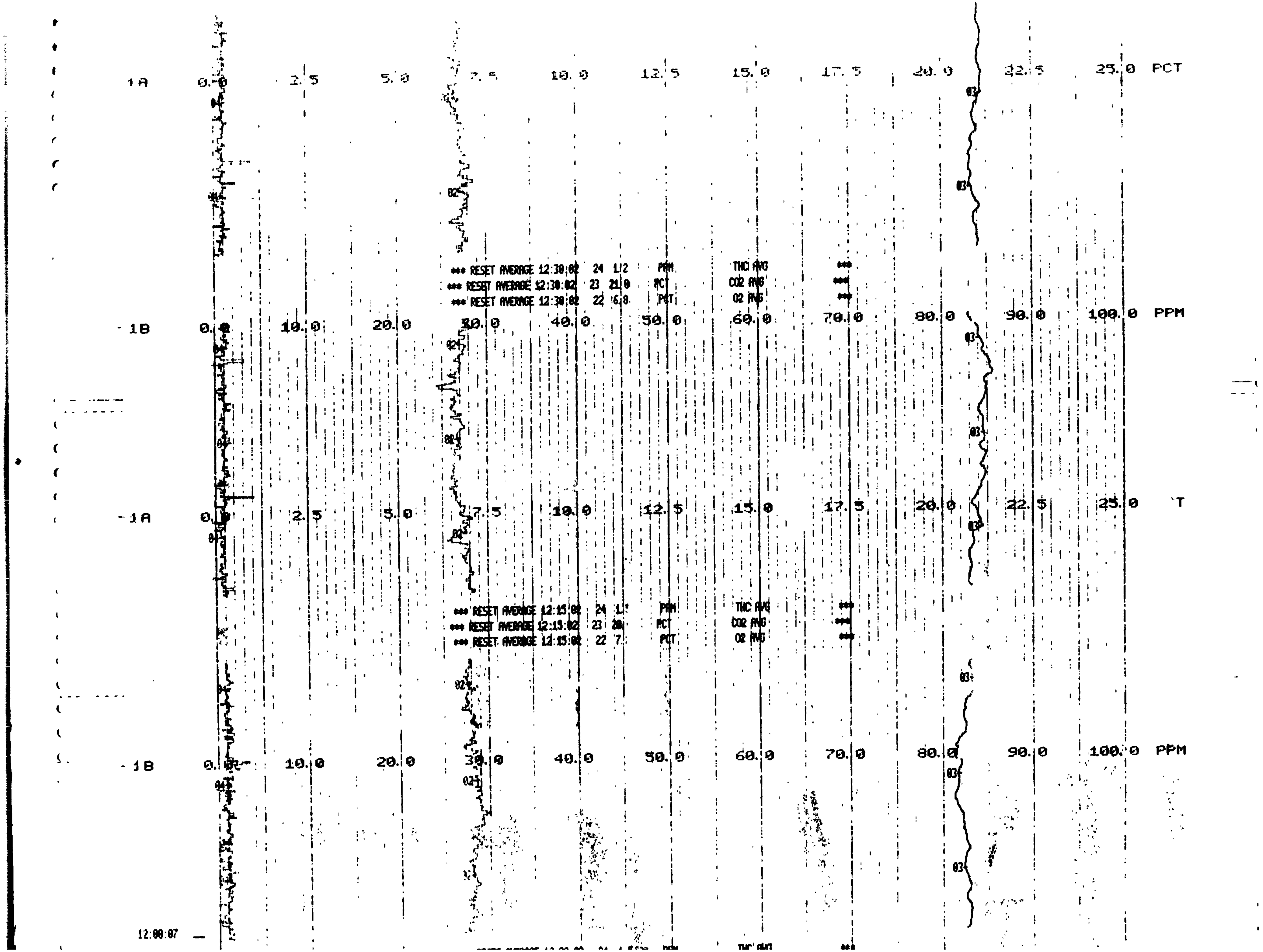


```
*** RESET AVERAGE 11:45:02   24  1.7    PPM        THC AVG          ***
*** RESET AVERAGE 11:45:02   23 20.3    PCT        CO2 AVG          ***
*** RESET AVERAGE 11:45:01   22  7.3    PCT        O2 AVG           ***

*** RESET AVERAGE 11:30:02   24 22.2    PPM        THC AVG          ***
*** RESET AVERAGE 11:30:01   23  7.2    PCT        CO2 AVG          ***
*** RESET AVERAGE 11:30:01   22 10.4    PCT        O2 AVG           ***
```

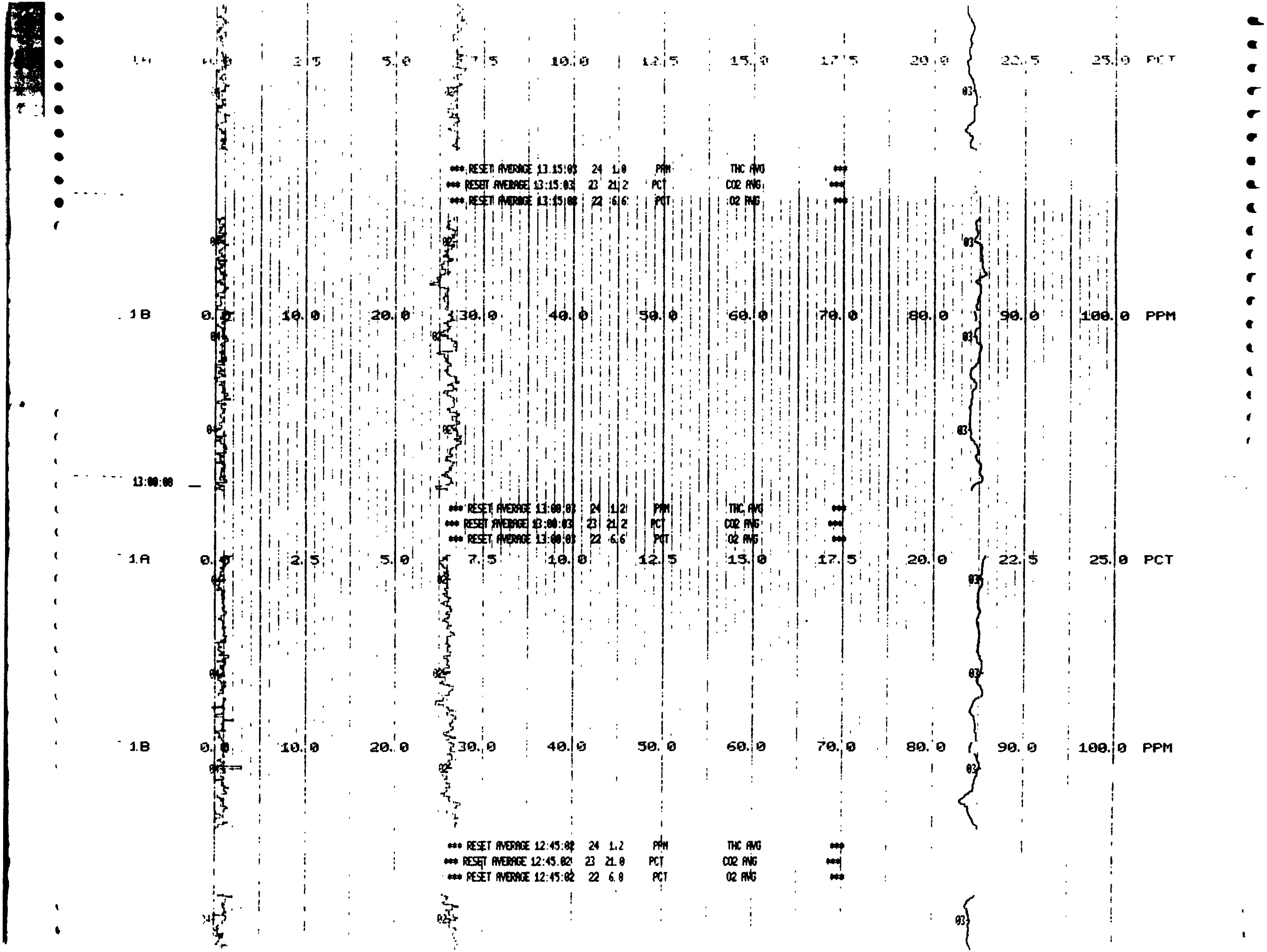
```
*** LOG GRP 1 *** UNIT 01    *** STARTS AT 11:18:48 *** ENDS AT 11:18:49 AUG 31,98 ***
PT DATA  UNITS ST    LEGEND    PT DATA  UNITS ST    LEGEND    PT DATA  UNITS ST    LEGEND    PT DATA  UNITS ST    LEGEND
02  .4   PCT       O2          03 - 2   PCT       CO2         04 -.2   PPM       THC
```

Zero



```
*** RESET AVERAGE 12:30:02   24  1.2     PPM          THC AVG       ***
*** RESET AVERAGE 12:30:02   23  21.0    PCT          CO2 AVG       ***
*** RESET AVERAGE 12:30:02   22  6.8     PCT          O2 AVG        ***
```

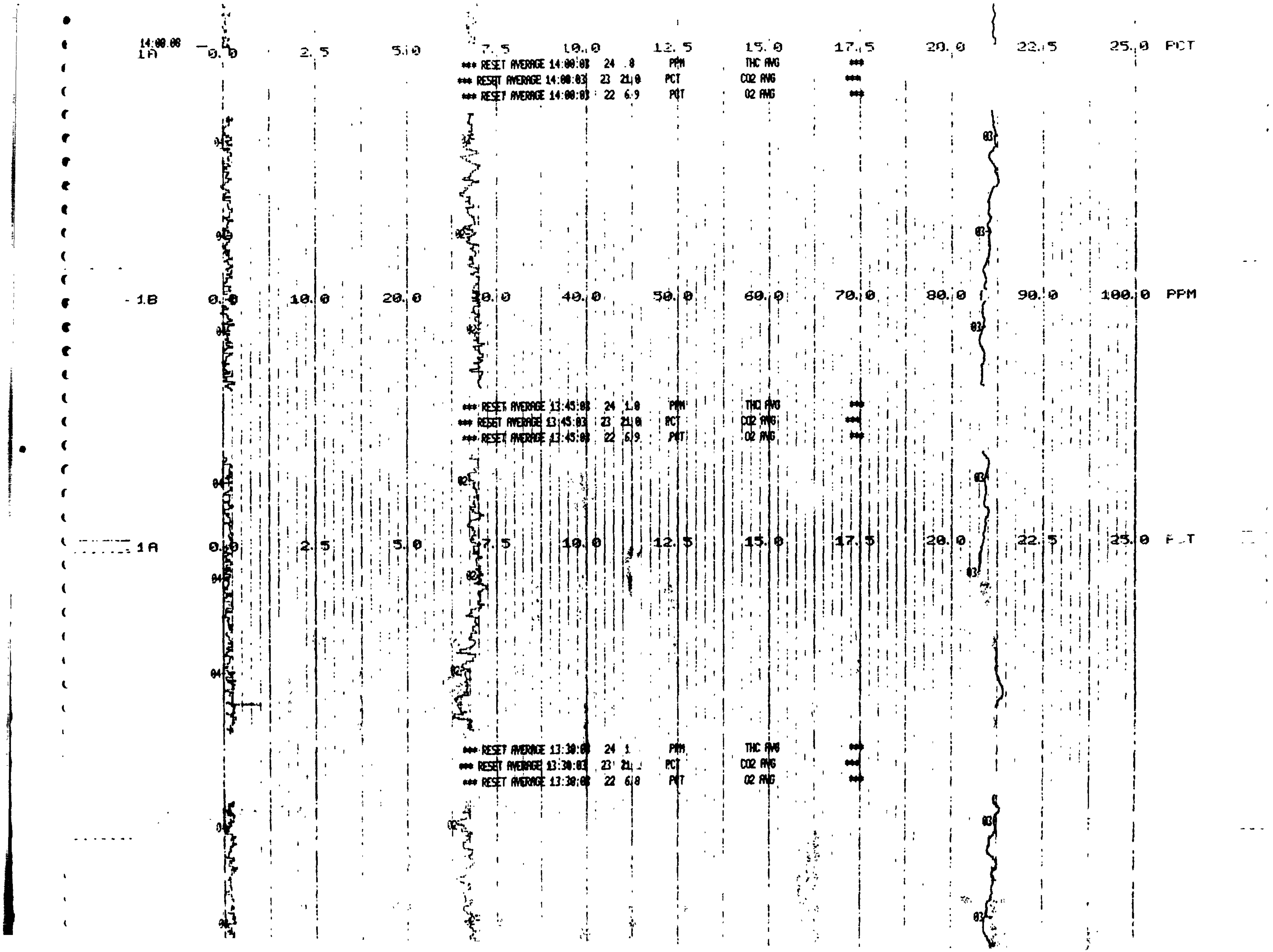
```
*** RESET AVERAGE 12:15:02   24  1.     PPM          THC AVG       ***
*** RESET AVERAGE 12:15:02   23  20     PCT          CO2 AVG       ***
*** RESET AVERAGE 12:15:02   22  7.     PCT          O2 AVG        ***
```



```
*** RESET AVERAGE 13.15:03   24  1.0     PPM          THC AVG          ***
*** RESET AVERAGE 13:15:03   23  21.2    PCT          CO2 AVG          ***
*** RESET AVERAGE 13:15:03   22  6.6     PCT          O2 AVG           ***
```

```
*** RESET AVERAGE 13:00:03   24  1.2     PPM          THC AVG          ***
*** RESET AVERAGE 13:00:03   23  21.2    PCT          CO2 AVG          ***
*** RESET AVERAGE 13:00:03   22  6.6     PCT          O2 AVG           ***
```

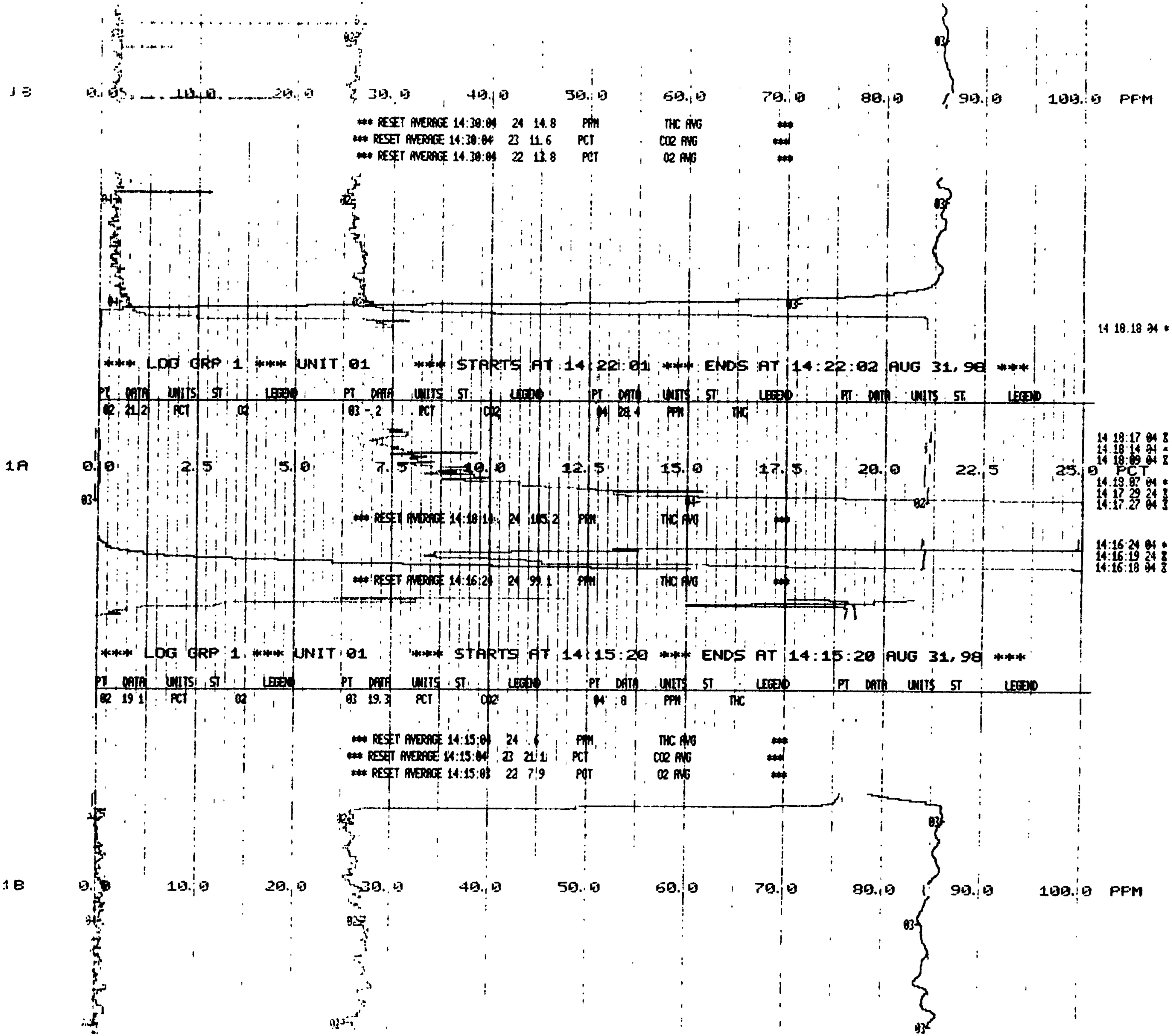
```
*** RESET AVERAGE 12:45:02   24  1.2     PPM          THC AVG          ***
*** RESET AVERAGE 12:45.02   23  21.0    PCT          CO2 AVG          ***
*** RESET AVERAGE 12:45:02   22  6.0     PCT          O2 AVG           ***
```



```
*** RESET AVERAGE 14:00:03   24  .8    PPM      THC AVG     ***
*** RESET AVERAGE 14:00:03   23  21.0  PCT      CO2 AVG     ***
*** RESET AVERAGE 14:00:03   22  6.9   PCT      O2 AVG      ***

*** RESET AVERAGE 13:45:03   24  1.0   PPM      THC AVG     ***
*** RESET AVERAGE 13:45:03   23  21.0  PCT      CO2 AVG     ***
*** RESET AVERAGE 13:45:03   22  6.9   PCT      O2 AVG      ***

*** RESET AVERAGE 13:30:03   24  1     PPM      THC AVG     ***
*** RESET AVERAGE 13:30:03   23  21.   PCT      CO2 AVG     ***
*** RESET AVERAGE 13:30:03   22  6.8   PCT      O2 AVG      ***
```



```
*** RESET AVERAGE 14:30:04   24  14.8     PPM          THC AVG        ***
*** RESET AVERAGE 14:30:04   23  11.6     PCT          CO2 AVG        ***
*** RESET AVERAGE 14.30:04   22  13.8     PCT          02 AVG         ***

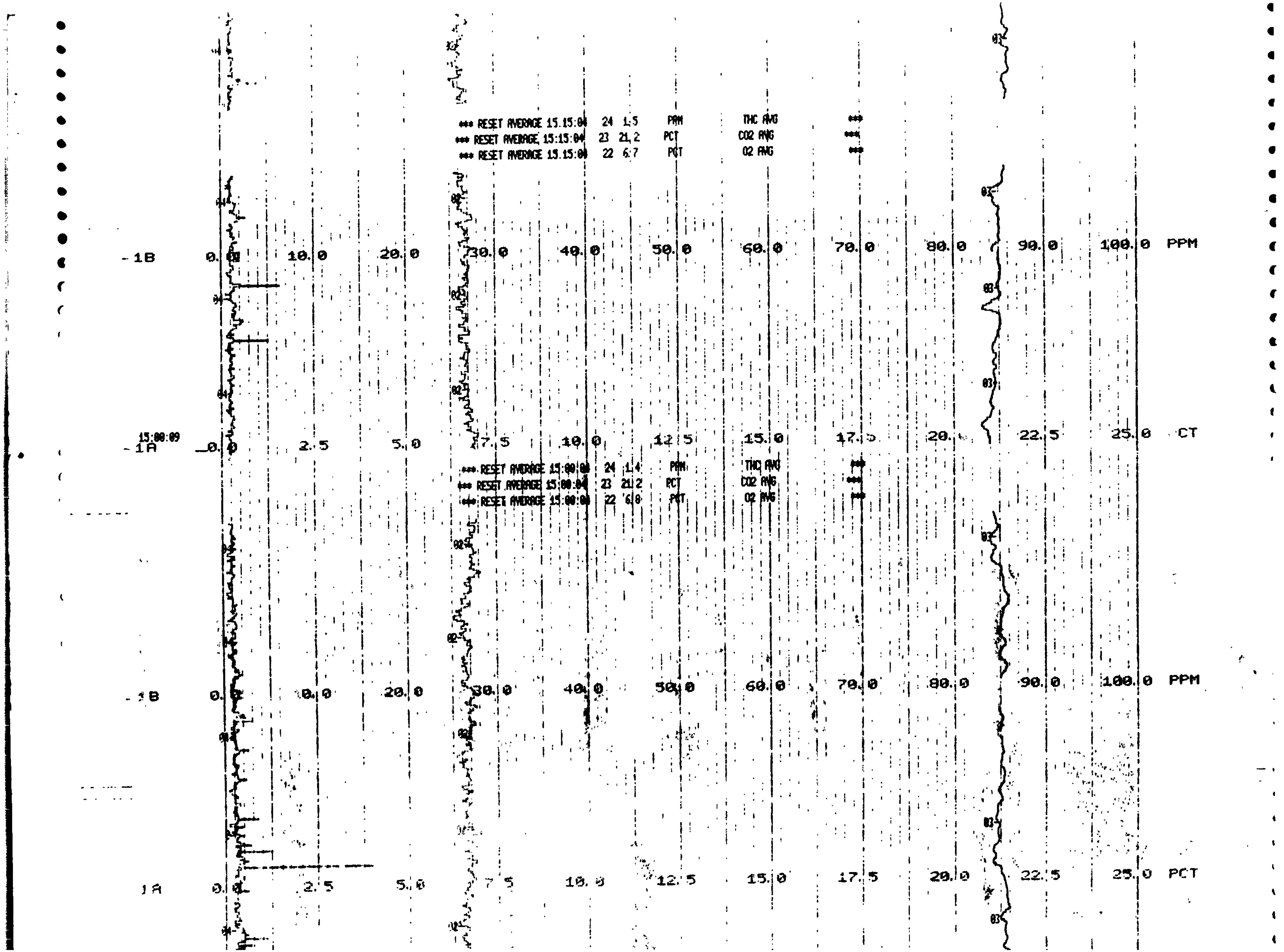
*** LOG GRP 1 *** UNIT 01    *** STARTS AT 14:22:01 *** ENDS AT 14:22:02 AUG 31,98 ***
PT  DATA  UNITS  ST   LEGEND    PT  DATA  UNITS  ST   LEGEND    PT  DATA  UNITS  ST   LEGEND    PT  DATA  UNITS  ST   LEGEND
02  21.2  PCT         02        03  -.2   PCT         CO2       04  28.4  PPM         THC

*** RESET AVERAGE 14:18:14   24  105.2    PPM          THC AVG        ***

*** RESET AVERAGE 14:16:23   24  99.1     PPM          THC AVG        ***

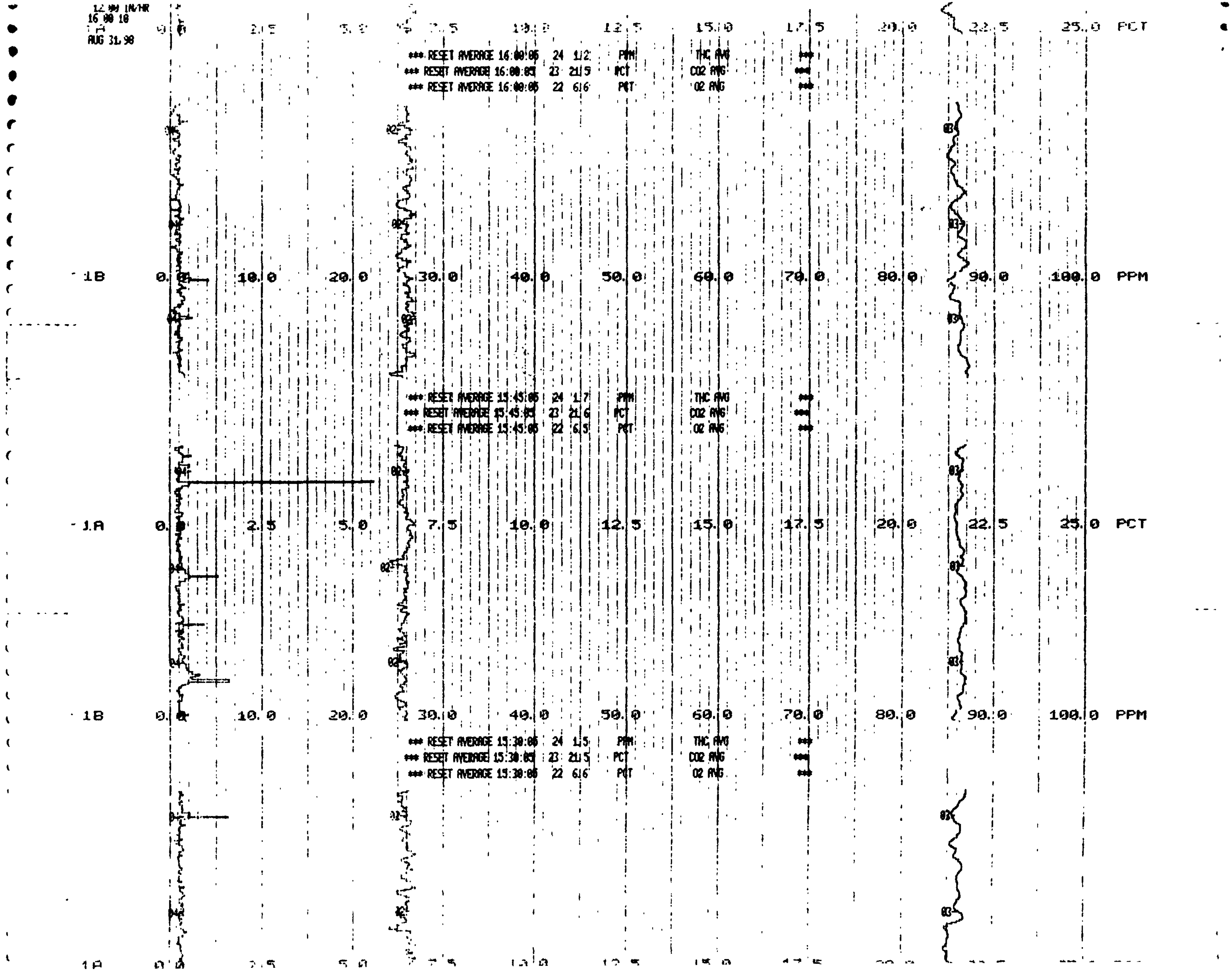
*** LOG GRP 1 *** UNIT 01    *** STARTS AT 14:15:20 *** ENDS AT 14:15:20 AUG 31,98 ***
PT  DATA  UNITS  ST   LEGEND    PT  DATA  UNITS  ST   LEGEND    PT  DATA  UNITS  ST   LEGEND    PT  DATA  UNITS  ST   LEGEND
02  19 1  PCT         02        03  19.3  PCT         CO2       04  .8    PPM         THC

*** RESET AVERAGE 14:15:04   24  .6       PPM          THC AVG        ***
*** RESET AVERAGE 14:15:04   23  21.1     PCT          CO2 AVG        ***
*** RESET AVERAGE 14:15:03   22  7.9      POT          02 AVG         ***
```



```
*** RESET AVERAGE 15.15:04   24  1.5    PPM         THC AVG        ***
*** RESET AVERAGE 15:15:04   23  21.2   PCT         CO2 AVG        ***
*** RESET AVERAGE 15.15:04   22  6.7    PCT         02 AVG         ***
```

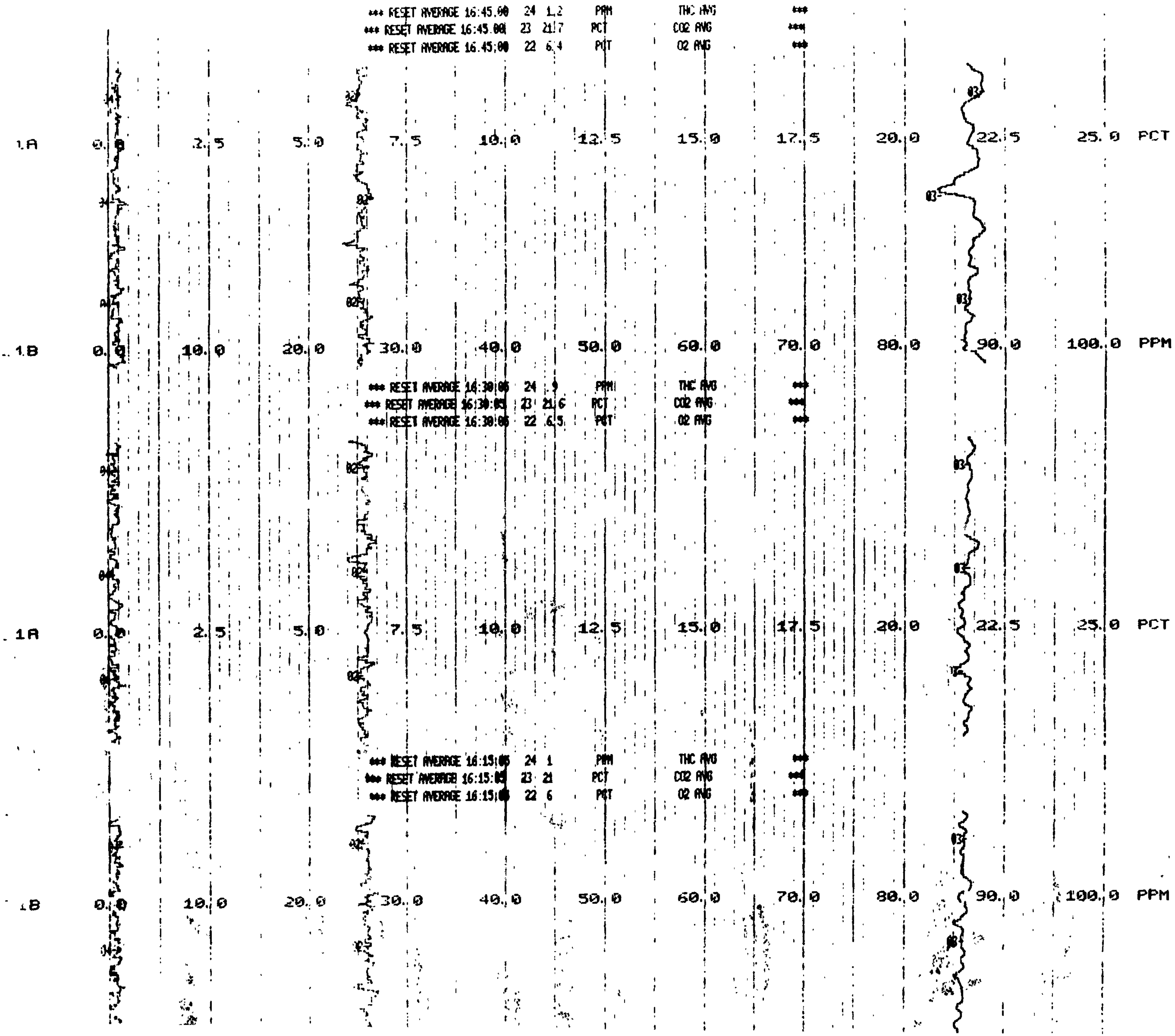
```
*** RESET AVERAGE 15:00:04   24  1.4    PPM         THC AVG        ***
*** RESET AVERAGE 15:00:04   23  21.2   PCT         CO2 AVG        ***
*** RESET AVERAGE 15:00:04   22  6.8    PCT         02 AVG         ***
```

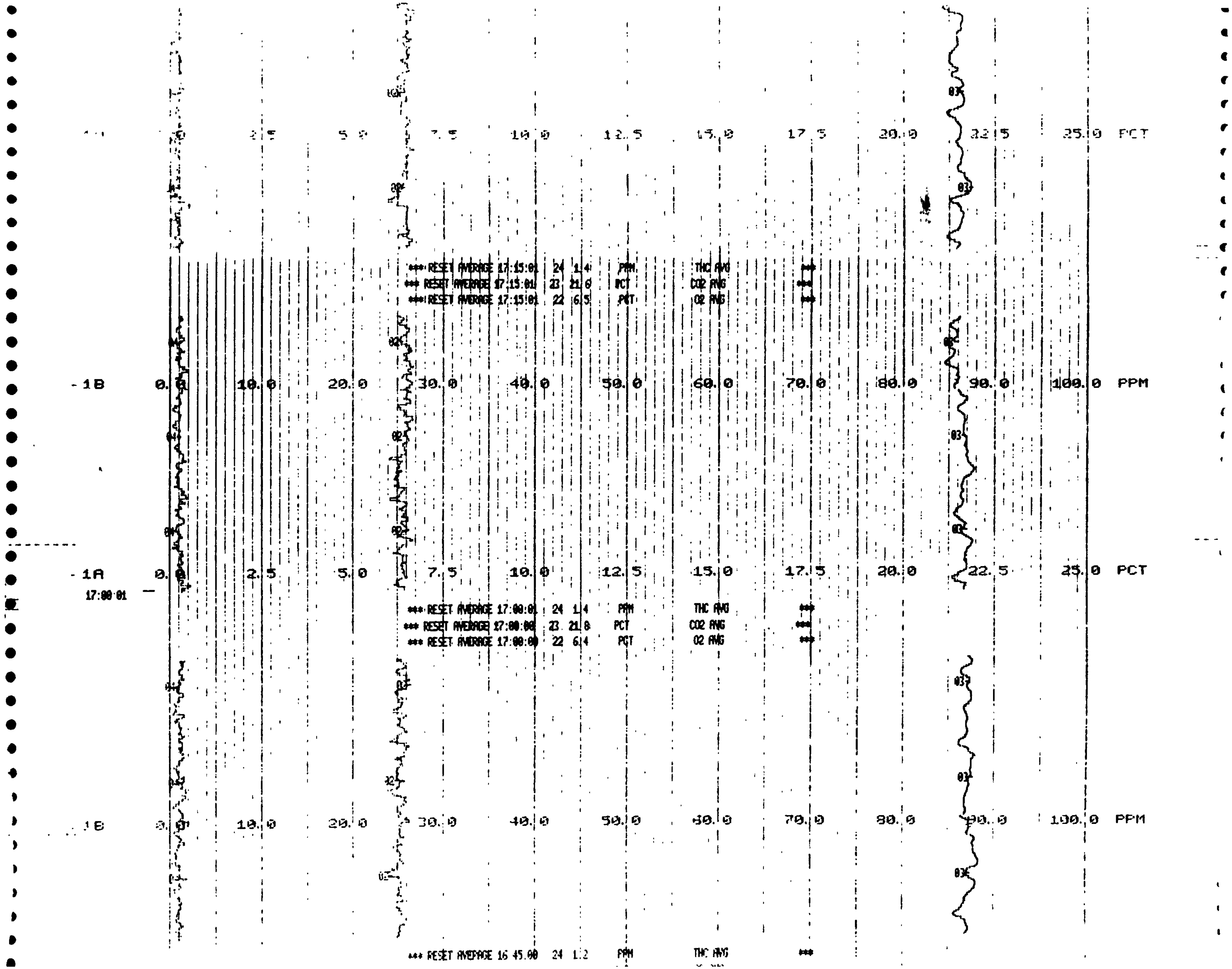


```
*** RESET AVERAGE 16:00:05  24  1.2   PPM   THC AVG  ***
*** RESET AVERAGE 16:00:05  23  21.5  PCT   CO2 AVG  ***
*** RESET AVERAGE 16:00:05  22  6.6   PCT   O2 AVG   ***
```

```
*** RESET AVERAGE 15:45:05  24  1.7   PPM   THC AVG  ***
*** RESET AVERAGE 15:45:05  23  21.6  PCT   CO2 AVG  ***
*** RESET AVERAGE 15:45:05  22  6.5   PCT   O2 AVG   ***
```

```
*** RESET AVERAGE 15:30:05  24  1.5   PPM   THC AVG  ***
*** RESET AVERAGE 15:30:05  23  21.5  PCT   CO2 AVG  ***
*** RESET AVERAGE 15:30:05  22  6.6   PCT   O2 AVG   ***
```



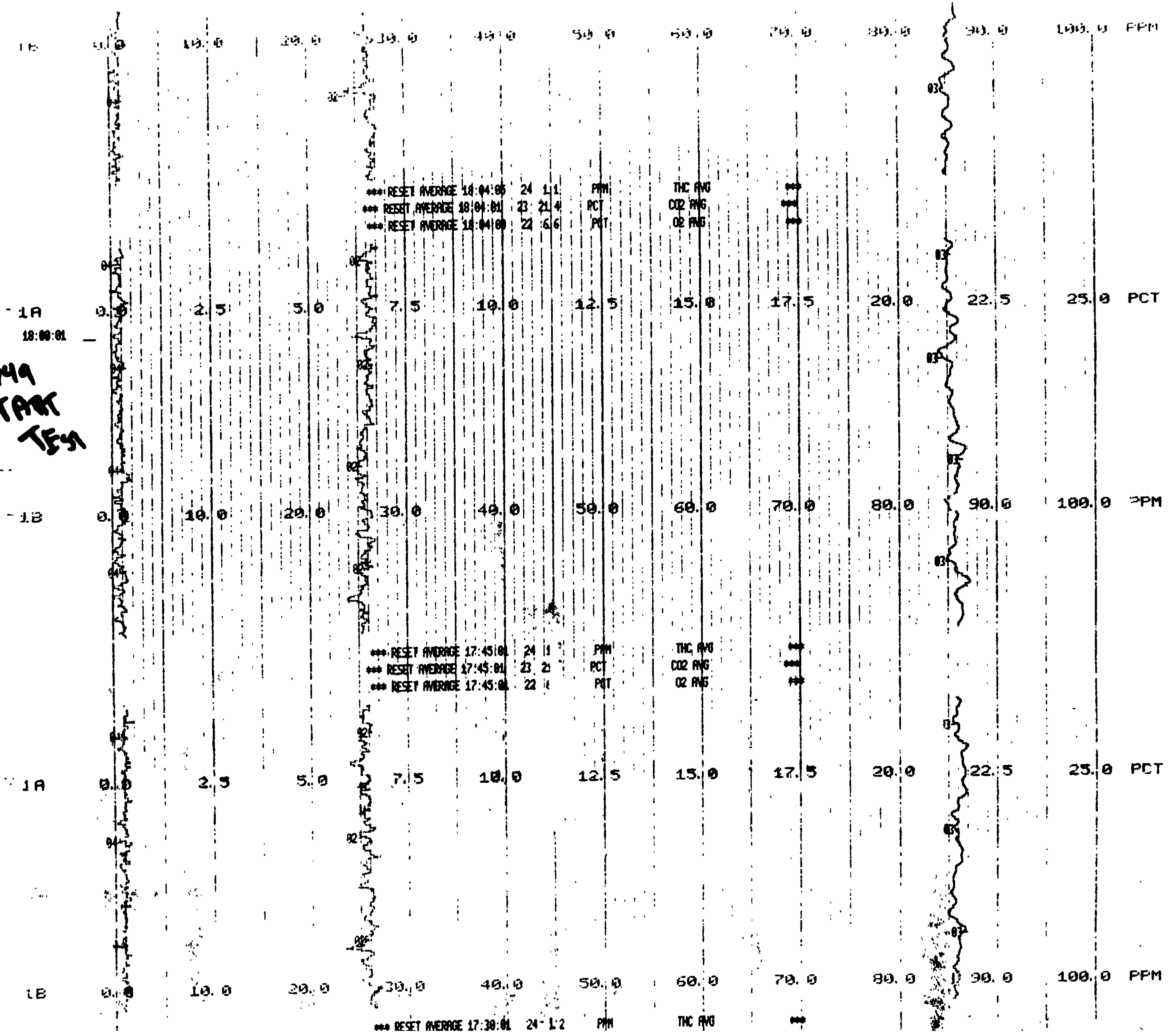


```
*** RESET AVERAGE 17:15:01   24   1.4     PPM          THC AVG        ***
*** RESET AVERAGE 17:15:01   23  21.6     PCT          CO2 AVG        ***
*** RESET AVERAGE 17:15:01   22   6.5     PCT          O2 AVG         ***
```

```
*** RESET AVERAGE 17:00:01   24   1.4     PPM          THC AVG        ***
*** RESET AVERAGE 17:00:00   23  21.8     PCT          CO2 AVG        ***
*** RESET AVERAGE 17:00:00   22   6.4     PCT          O2 AVG         ***
```

```
*** RESET AVERAGE 16 45.00   24   1.2     PPM          THC AVG        ***
```

1749 START TEST

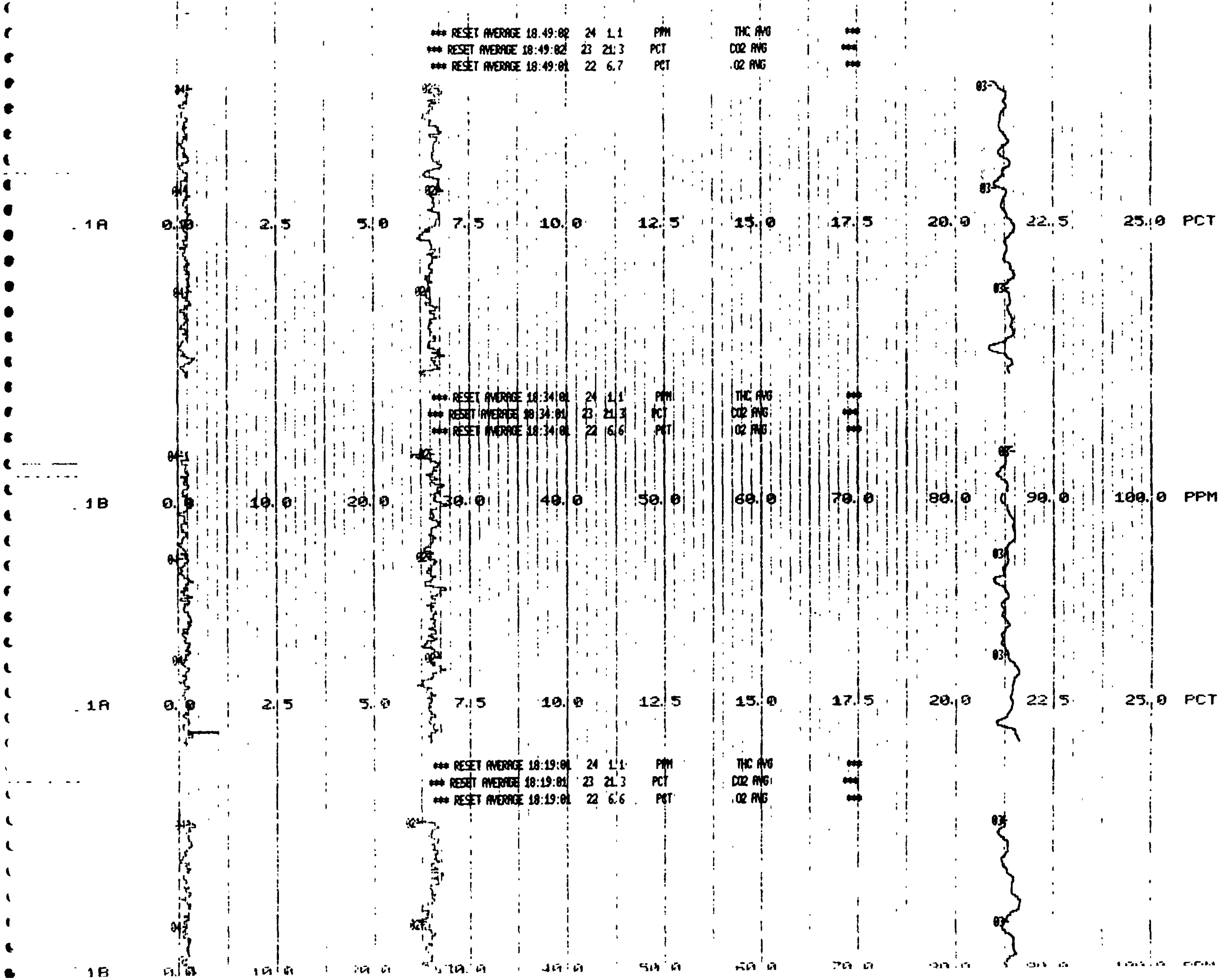


```
*** RESET AVERAGE 18:04:05   24  1.1      PPM        THC AVG        ***
*** RESET AVERAGE 18:04:01   23  21.4     PCT        CO2 AVG        ***
*** RESET AVERAGE 18:04:00   22  6.6      PCT        O2 AVG         ***
```

```
*** RESET AVERAGE 17:45:01   24  1        PPM        THC AVG        ***
*** RESET AVERAGE 17:45:01   23  2        PCT        CO2 AVG        ***
*** RESET AVERAGE 17:45:01   22  6        PCT        O2 AVG         ***
```

```
*** RESET AVERAGE 17:30:01   24  1.2      PPM        THC AVG        ***
```

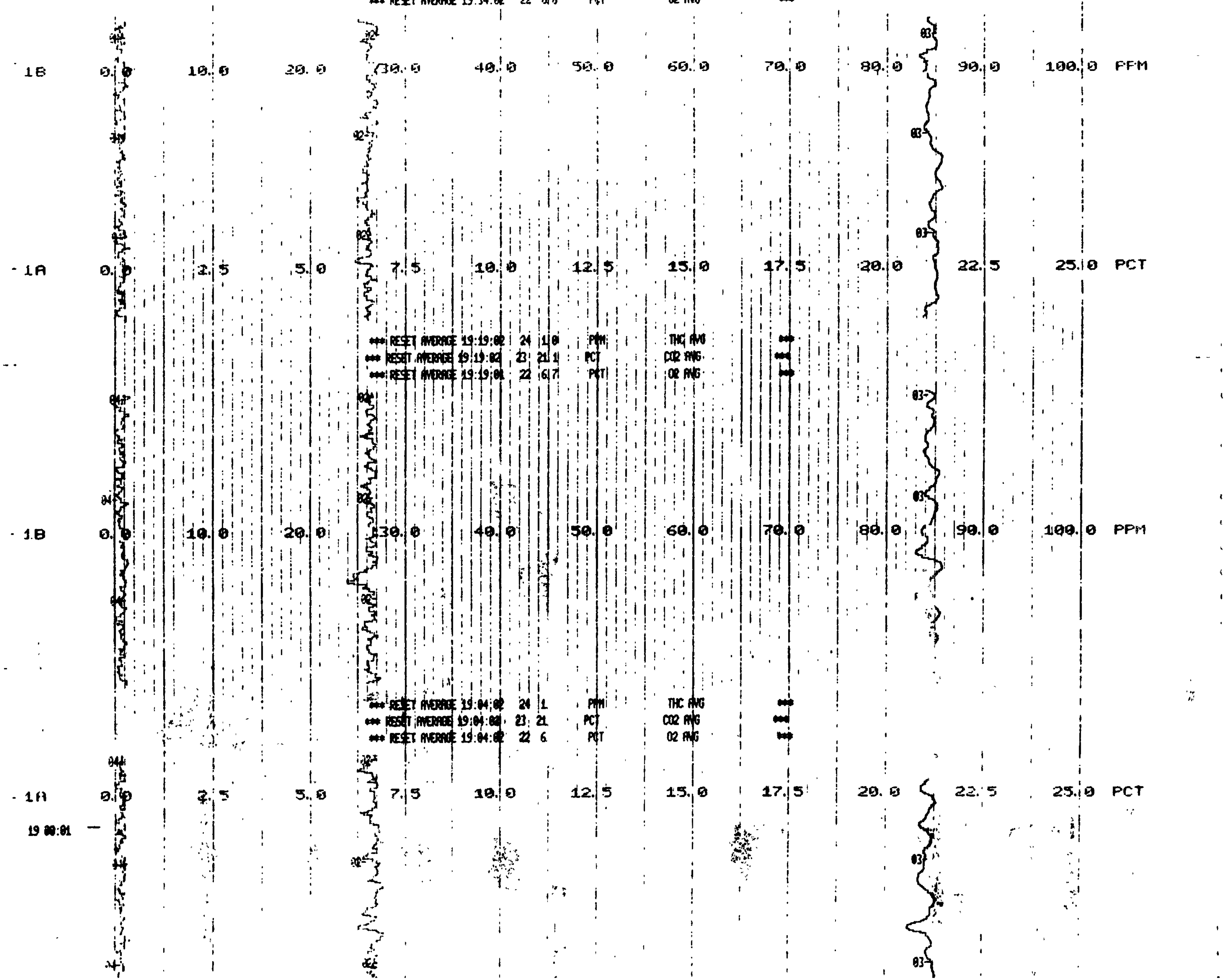
*** NO ALARMS PRESENT *** UNIT 01 *** STARTS AT 18:49:07 *** ENDS AT 18:49:07 AUG 31,98 ***



```
*** RESET AVERAGE 18:49:02   24  1.1    PPM    THC AVG   ***
*** RESET AVERAGE 18:49:02   23  21.3   PCT    CO2 AVG   ***
*** RESET AVERAGE 18:49:01   22  6.7    PCT    O2 AVG    ***

*** RESET AVERAGE 18:34:01   24  1.1    PPM    THC AVG   ***
*** RESET AVERAGE 18:34:01   23  21.3   PCT    CO2 AVG   ***
*** RESET AVERAGE 18:34:01   22  6.6    PCT    O2 AVG    ***

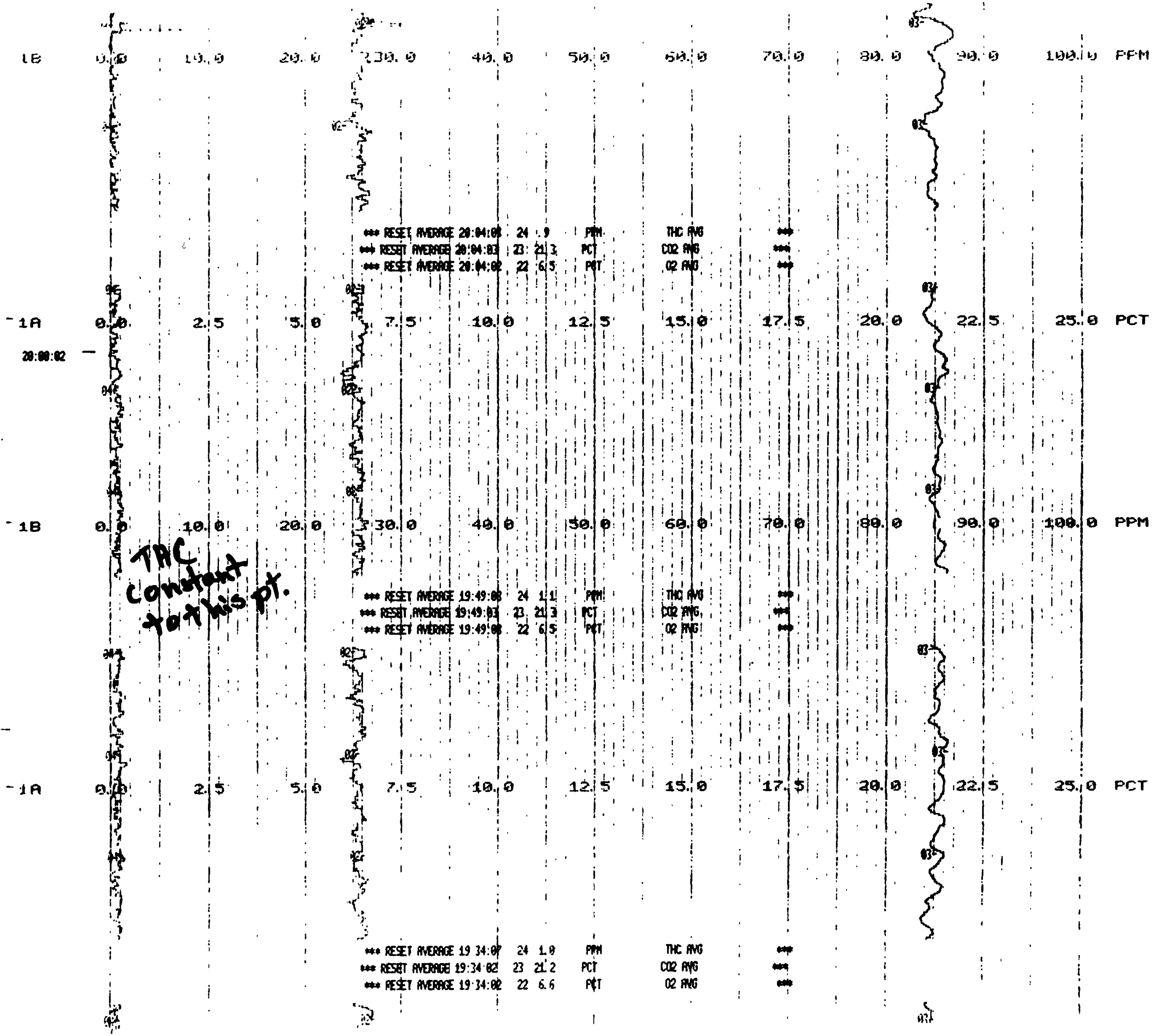
*** RESET AVERAGE 18:19:01   24  1.1    PPM    THC AVG   ***
*** RESET AVERAGE 18:19:01   23  21.3   PCT    CO2 AVG   ***
*** RESET AVERAGE 18:19:01   22  6.6    PCT    O2 AVG    ***
```



```
*** RESET AVERAGE 19:34 02   23  21.2     PCT          CO2 AVG          ***
 *** RESET AVERAGE 19.34:02   22  6.6      PCT          02 AVG           ***

 *** RESET AVERAGE 19:19:02   24  1.0      PPM          THC AVG          ***
*** RESET AVERAGE 19:19:02   23  21.1     PCT          CO2 AVG          ***
 *** RESET AVERAGE 19:19:01   22  6.7      PCT          02 AVG           ***

 *** RESET AVERAGE 19:04:02   24  1        PPM          THC AVG          ***
*** RESET AVERAGE 19:04:02   23  21       PCT          CO2 AVG          ***
 *** RESET AVERAGE 19:04:02   22  6        PCT          02 AVG           ***
```

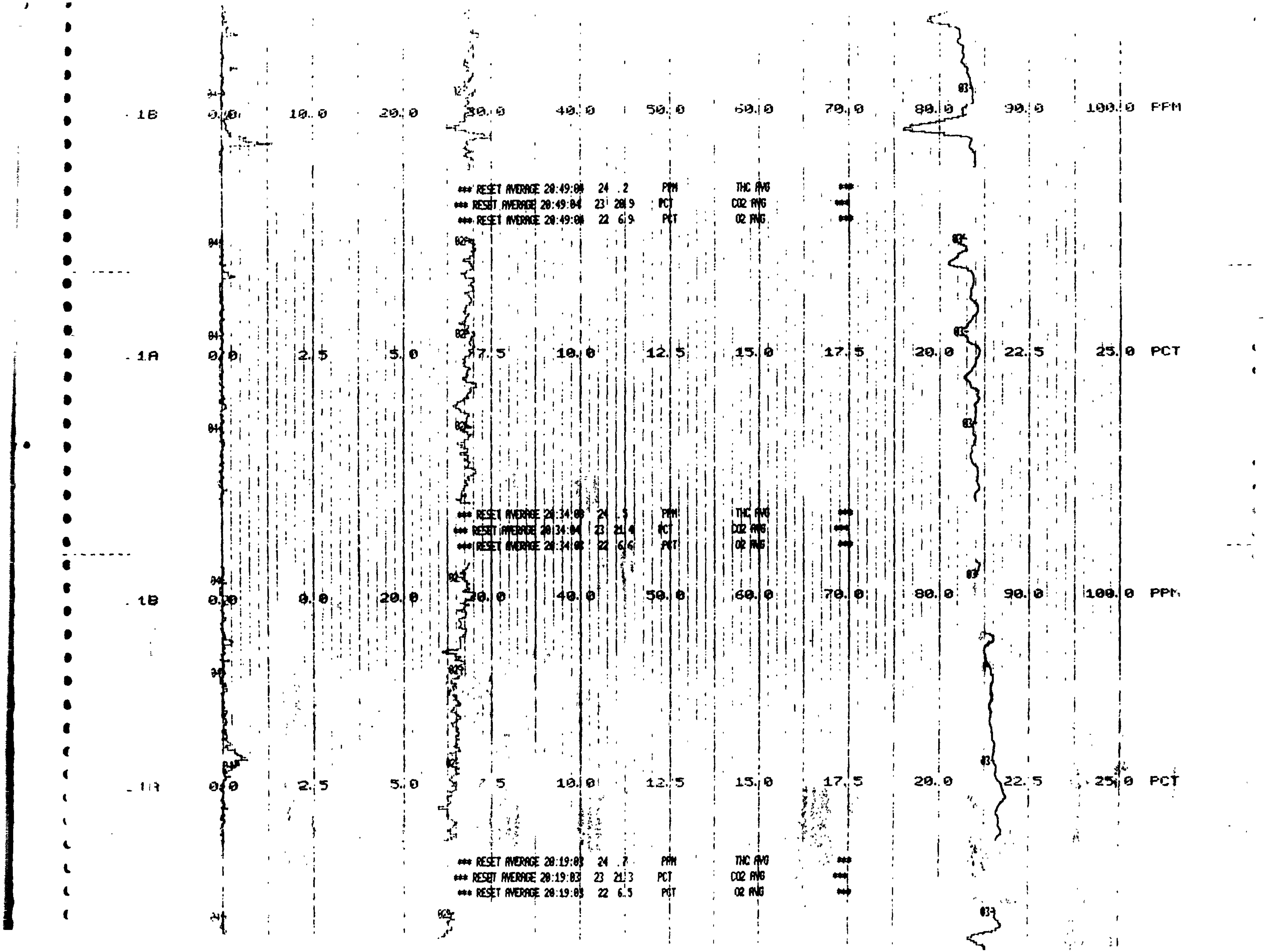


```
*** RESET AVERAGE 20:04:08   24   .9    PPM        THC AVG        ***
*** RESET AVERAGE 20:04:03   23  21.3   PCT        CO2 AVG        ***
*** RESET AVERAGE 20:04:02   22   6.5   PCT        02 AVG         ***
```

THC constant to this pt.

```
*** RESET AVERAGE 19:49:08   24  1.1    PPM        THC AVG        ***
*** RESET AVERAGE 19:49:03   23  21.3   PCT        CO2 AVG        ***
*** RESET AVERAGE 19:49:08   22   6.5   PCT        02 AVG         ***
```

```
*** RESET AVERAGE 19 34:07   24  1.0    PPM        THC AVG        ***
*** RESET AVERAGE 19:34-02   23  21.2   PCT        CO2 AVG        ***
*** RESET AVERAGE 19·34:02   22  6.6    PCT        02 AVG         ***
```



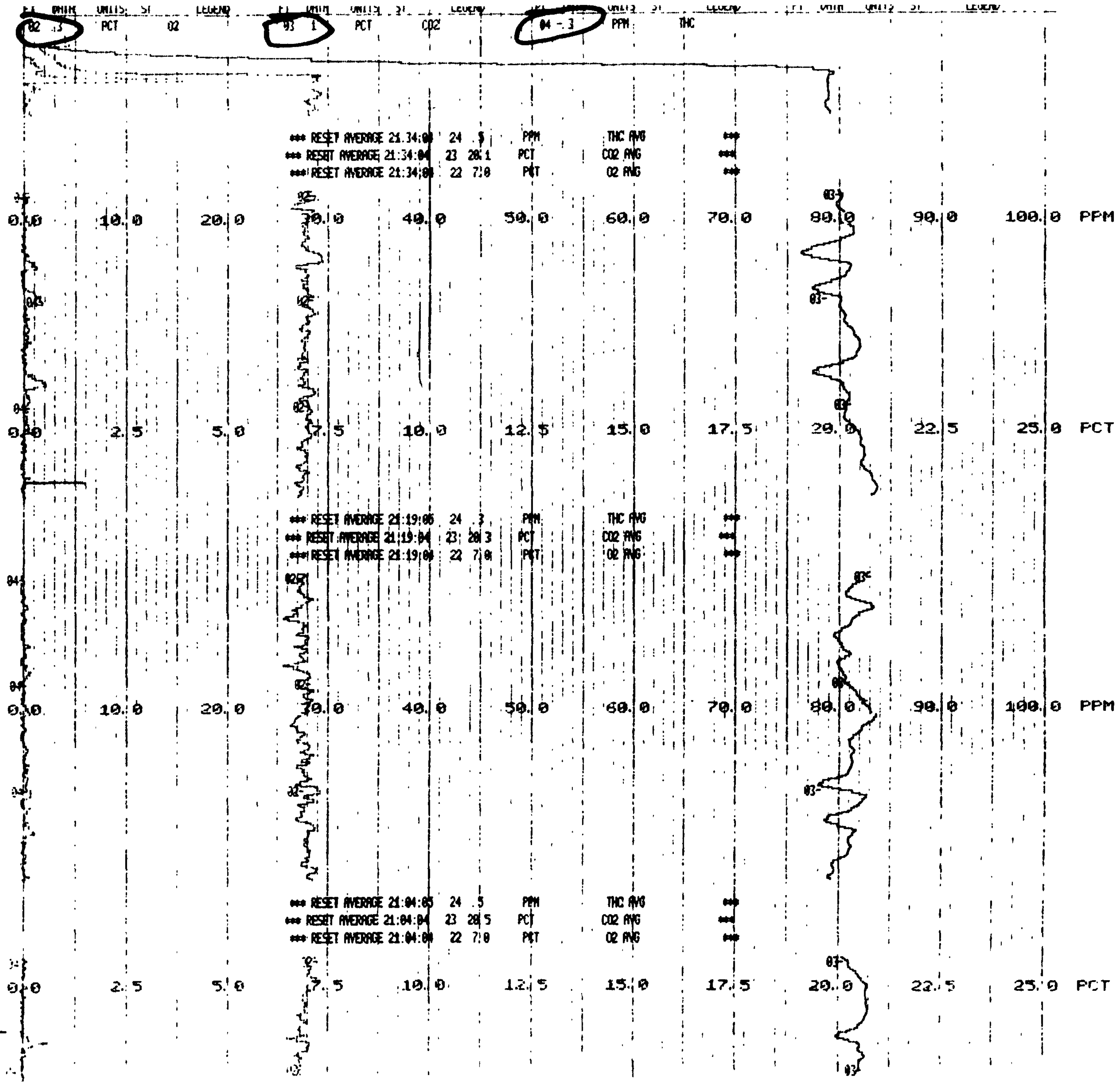
```
*** RESET AVERAGE 20:49:04   24   .2    PPM    THC AVG    ***
*** RESET AVERAGE 20:49:04   23   20.9  PCT    CO2 AVG    ***
*** RESET AVERAGE 20:49:04   22   6.9   PCT    O2 AVG     ***
```

```
*** RESET AVERAGE 20:34:03   24   .5    PPM    THC AVG    ***
*** RESET AVERAGE 20:34:04   23   21.4  PCT    CO2 AVG    ***
*** RESET AVERAGE 20:34:03   22   6.6   PCT    O2 AVG     ***
```

```
*** RESET AVERAGE 20:19:03   24   .7    PPM    THC AVG    ***
*** RESET AVERAGE 20:19:03   23   21.3  PCT    CO2 AVG    ***
*** RESET AVERAGE 20:19:03   22   6.5   PCT    O2 AVG     ***
```

Zero
Bias

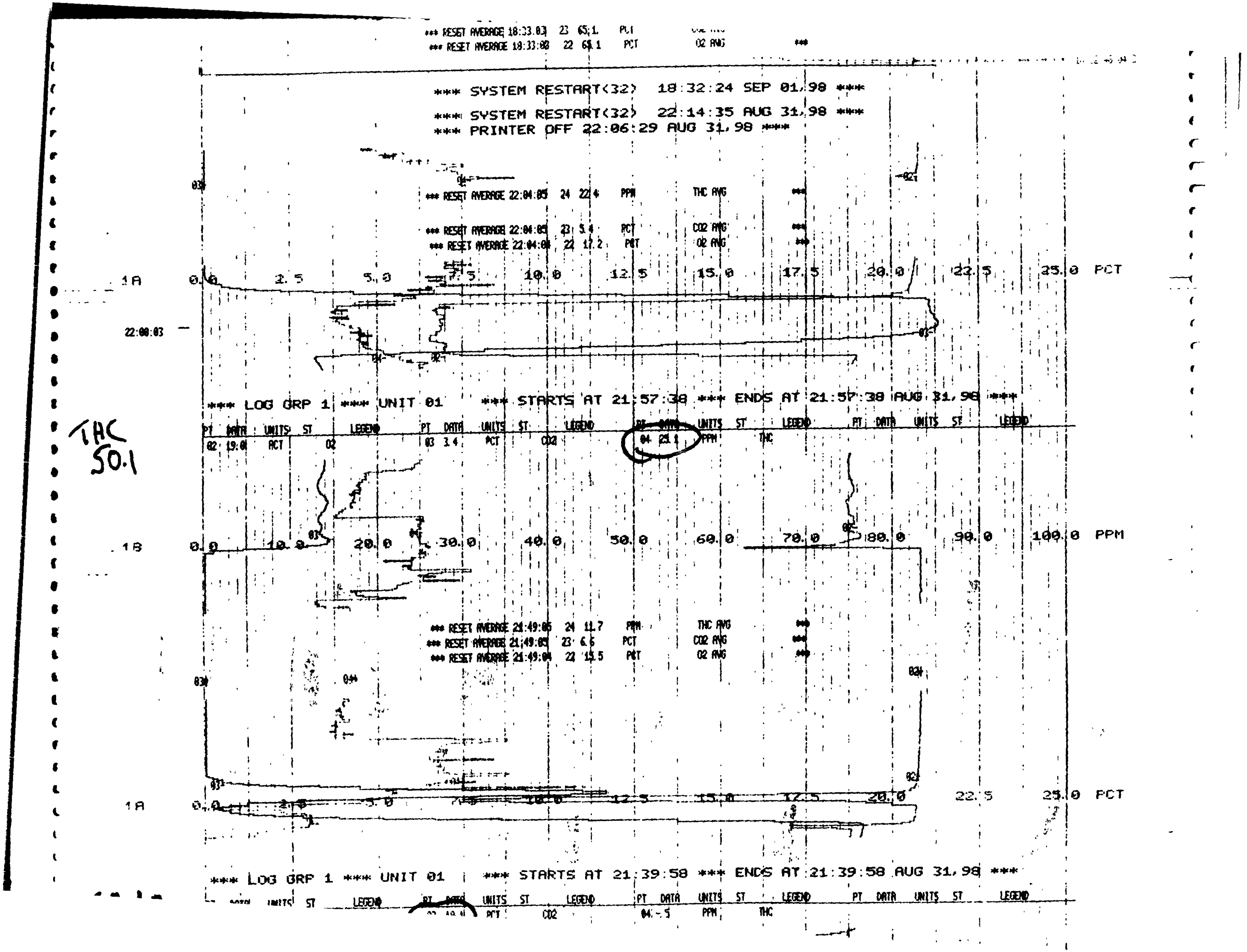
END
TEST



```
*** RESET AVERAGE 21:34:08  24  .5   PPM   THC AVG  ***
*** RESET AVERAGE 21:34:04  23  20.1 PCT   CO2 AVG  ***
*** RESET AVERAGE 21:34:00  22  7.0  PCT   02 AVG   ***

*** RESET AVERAGE 21:19:06  24  .3   PPM   THC AVG  ***
*** RESET AVERAGE 21:19:04  23  20.3 PCT   CO2 AVG  ***
*** RESET AVERAGE 21:19:00  22  7.0  PCT   02 AVG   ***

*** RESET AVERAGE 21:04:05  24  .5   PPM   THC AVG  ***
*** RESET AVERAGE 21:04:04  23  20.5 PCT   CO2 AVG  ***
*** RESET AVERAGE 21:04:00  22  7.0  PCT   02 AVG   ***
```



```
*** RESET AVERAGE 18:33:03  23  65.1   PCT
*** RESET AVERAGE 18:33:08  22  65.1   PCT        O2 AVG       ***

**** SYSTEM RESTART(32)  18:32:24 SEP 01, 98 ****

**** SYSTEM RESTART(32)  22:14:35 AUG 31, 98 ****
**** PRINTER OFF 22:06:29 AUG 31, 98 ****

*** RESET AVERAGE 22:04:05  24  22.4   PPM        THC AVG      ***

*** RESET AVERAGE 22:04:05  23  5.4    PCT        CO2 AVG      ***
*** RESET AVERAGE 22:04:04  22  17.2   PCT        O2 AVG       ***

 1A    0.0   2.5   5.0   7.5   10.0   12.5   15.0   17.5   20.0   22.5   25.0  PCT

22:00:03

*** LOG GRP 1 *** UNIT 01    *** STARTS AT 21:57:38 *** ENDS AT 21:57:38 AUG 31, 98 ***
PT DATA  UNITS ST  LEGEND   PT DATA UNITS ST  LEGEND   PT DATA UNITS ST  LEGEND   PT DATA UNITS ST  LEGEND
02 19.0  PCT       O2       03 3.4  PCT       CO2      04 21.1 PPM       THC

 1B    0.0   10.0   20.0   30.0   40.0   50.0   60.0   70.0   80.0   90.0   100.0  PPM

*** RESET AVERAGE 21:49:05  24  11.7   PPM        THC AVG      ***
*** RESET AVERAGE 21:49:05  23  6.6    PCT        CO2 AVG      ***
*** RESET AVERAGE 21:49:04  22  15.5   PCT        O2 AVG       ***

 1A    0.0   2.5   5.0   7.5   10.0   12.5   15.0   17.5   20.0   22.5   25.0  PCT

*** LOG GRP 1 *** UNIT 01    *** STARTS AT 21:39:58 *** ENDS AT 21:39:58 AUG 31, 98 ***
PT DATA  UNITS ST  LEGEND   PT DATA UNITS ST  LEGEND   PT DATA UNITS ST  LEGEND   PT DATA UNITS ST  LEGEND
                            PCT       CO2               04 -.5  PPM       THC
```

THC
50.1

# APPENDIX B

# METHOD 23 LABORATORY ANALYTICAL DATA

**PARADIGM ANALYTICAL LABORATORIES, INC.**
2627 Northchase Parkway S.E.
Wilmington, North Carolina 28405
(910) 350-1903
Fax (910) 350-1557

30 SEP 98

Michael Maret
Pacific Environmental Services, Inc.
5001 S. Miami Blvd
Research Triangle Park, NC 27709-2077

| | |
|---|---|
| Contract: | 68D70002 |
| Sub-Contract: | R012-002 |
| Work Assignment: | 1-007 |

Subject: Polychlorinated Dibenzo-*p*-Dioxins & Dibenzofurans Measurements (PAL Project No. L-1114)

Dear Mike;

Enclosed are the final results for the flue gas samples under your Project S509.000 Ohio Lime Kiln. As you requested, we divided up the set of 15 samples into three separate projects (L-1113, L-1114, and L-1115; see Table 1 for a summary). This report covers the first set under PAL L-1114. The analytical procedures conformed or exceeded the ones described in Method 23 using isotope-dilution high-resolution gas chromatography combined with high-resolution mass spectrometry. The **Level II** reporting format is described on the next page. A general summary of the analytical results is presented in Table 2. Figures 1and 2 show the TEQs and total homologues corresponding to Tables 2 data.

| | |
|---|---|
| **No. of Samples Received:** | **2** |
| **No. of Samples Analyzed:** | **2** |
| **No. of Lab. Method Blanks:** | **1** |
| **Your Project Number:** | **S509.000 Ohio Lime Kiln** |
| **PAL Project No.:** | **L-1114** |

**Remarks:**

- **Data meet QA/QC requirements.**
- **No analytical difficulties to be reported.**
- **The HxCDF sampling standard recovery in sample M23-FB-3 is above the 130 percent level, i.e., 147 percent). Similar observations were made in the LMB. We believe these observations originate from a variation in the response factors and should not affect the reported amounts of HxCDFs in the sample.**

We wanted to thank you for the opportunity to serve you. Please, feel free to contact us if you have questions or should you need additional technical support.

Sincerely,

Yves Tondeur, Ph.D.

## Level II Report

Section 1: **Cover Letter**, contains a brief description of the project, the client and PAL Project Numbers, the number and type of samples, the methodology used to process the samples, QC remarks where any analytical difficulties are discussed and the impact on the quality of the data presented, a summary table with the analyte concentrations, detection limits, the client sample identification numbers, units to report the concentrations, and a graphical representation of the TEQs and totals.

Section 2: **Project Synopsis**, contains the Sample Tracking & Management Forms, Communications Form, any correspondence, chain-of-custody and the last page is always a copy of the sample injection log(s). This section is designed to help the laboratory and the data reviewer with an overall view of the entire analytical procedure, the initials and dates of who did what when on which sample. Spiking solution IDs are recorded along with the batch numbers of the supplies and reagents used.

Section 3: **Analytical Results**, contain the sample results topsheets (one set of two per sample), the raw data (i.e., the selected ion current profiles, the areas, heights, ion abundance ratios, signal-to-noise ratios, and retention times of the GC peaks).

Section 4: **System Performance**, contains the documentation on the GC/MS system performance. In particular, the mass resolution checks, GC column performance checks, initial and continuing calibration summary tables and, when applicable, associated raw data for both column types.

Table 1: Project No. S509.000; Project Name: US EPA Lime Kiln Screening, OhioLime;
Sample and Project Identifications.

| PES Sample ID | PAL Sample ID | PAL Project No. |
|---|---|---|
| M23-I-1 | 1113-1 | L-1113 |
| M23-O-1 | 1113-3 | L-1113 |
| M23-FB-1 | 1113-5 | L-1113 |
| M23-RB | 1113-7 | L-1113 |
| M23-I-1-FH | 1113-8 | L-1113 |
| M23-I-2 | 1113-2 | L-1113 |
| M23-O-2 | 1113-4 | L-1113 |
| M23-FB-2 | 1113-6 | L-1113 |
| M23-I-2-FH | 1113-9 | L-1113 |
| M23-O-3 | 1114-1 | L-1114 |
| M23-FB-3 | 1114-2 | L-1114 |
| M23-I-4 | 1115-1 | L-1115 |
| M23-O-4 | 1115-2 | L-1115 |
| M23-FB-4 | 1115-3 | L-1115 |
| M23-I-4-FH | 1115-4 | L-1115 |

Table 2: Analyte Concentrations in "ng" per Sampling Train

| **Analyte** | **S509.000 LMB** | **S509.000 M23-O-3** | **S509.000 [illegible]** |
|---|---|---|---|
| 2,3,7,8-TCDD | (0.001) | 0.010 | (0.001) |
| 1,2,3,7,8-PeCDD | 0.002 | 0.004 | (0.001) |
| 1,2,3,4,7,8-HxCDD | [0.0018] | 0.002 | (0.001) |
| 1,2,3,6,7,8-HxCDD | 0.002 | 0.003 | [0.00128] |
| 1,2,3,7,8,9-HxCDD | [0.0018] | 0.003 | (0.001) |
| 1,2,3,4,6,7,8-HpCDD | [0.00208] | [0.00748] | [illegible] |
| OCDD | (0.010) | [0.03192] | [0.00924] |
| 2,3,7,8-TCDF[a] | (0.002) | 0.309 | (0.002) |
| 1,2,3,7,8-PeCDF | 0.002 | 0.101 | (0.001) |
| 2,3,4,7,8-PeCDF | [0.00204] | 0.060 | (0.001) |
| 1,2,3,4,7,8-HxCDF | [0.00156] | 0.021 | 0.002 |
| 1,2,3,6,7,8-HxCDF | [0.00132] | 0.012 | [0.00148] |
| 2,3,4,6,7,8-HxCDF | [0.00128] | 0.007 | (0.001) |
| 1,2,3,7,8,9-HxCDF | [0.00184] | [illegible] | (0.001) |
| 1,2,3,4,6,7,8-HpCDF | [0.00296] | 0.011 | (0.002) |
| 1,2,3,4,7,8,9-HpCDF | (0.003) | (0.003) | (0.003) |
| OCDF | (0.004) | (0.006) | (0.005) |
| Total TCDDs | (0.001) | 0.151 | (0.001) |
| Total PeCDDs | 0.002 | 0.045 | [0.0008] |
| Total HxCDDs | 0.002 | 0.015 | 0.002 |
| Total HpCDDs | [0.002] | [0.016] | [illegible] |
| Total TCDFs | (0.002) | 5.684 | (0.002) |
| Total PeCDFs | 0.002 | 0.940 | (0.001) |
| Total HxCDFs | [0.006] | 0.078 | 0.002 |
| Total HpCDFs | [0.0052] | 0.010 | (0.002) |
| **Total PCDD/Fs[b]** | **0.006** | **6.924** | **0.004** |
| TEQ (ND=0)[c] | 0.001 | 0.083 | 0.000 |
| TEQ (ND=1/2)[d] | 0.002 | .083 | 0.002 |
| TEQ EMPC(ND=0)[e] | 0.003 | 0.083 | 0.001 |
| TEQ EMPC (ND=1/2) | 0.004 | 0.083 | 0.002 |

a) Result obtained from the DB-225 analysis.
b) Total PCDD/Fs represent the sum of all polychlorinated dibenzo-p-dioxins & dibenzofurans
c) TEQ computed using ITEF and setting non detected analytes with a "Zero" concentration.
d) TEQ computed using ITEF and setting non detected analytes with a concentration half the calculated detection limit.
e) TEQ computed using ITEF and setting the concentration of EMP[illegible]MPC value.

NOTE:

( ) = ND using DL value.
[ ] = EMPC value.

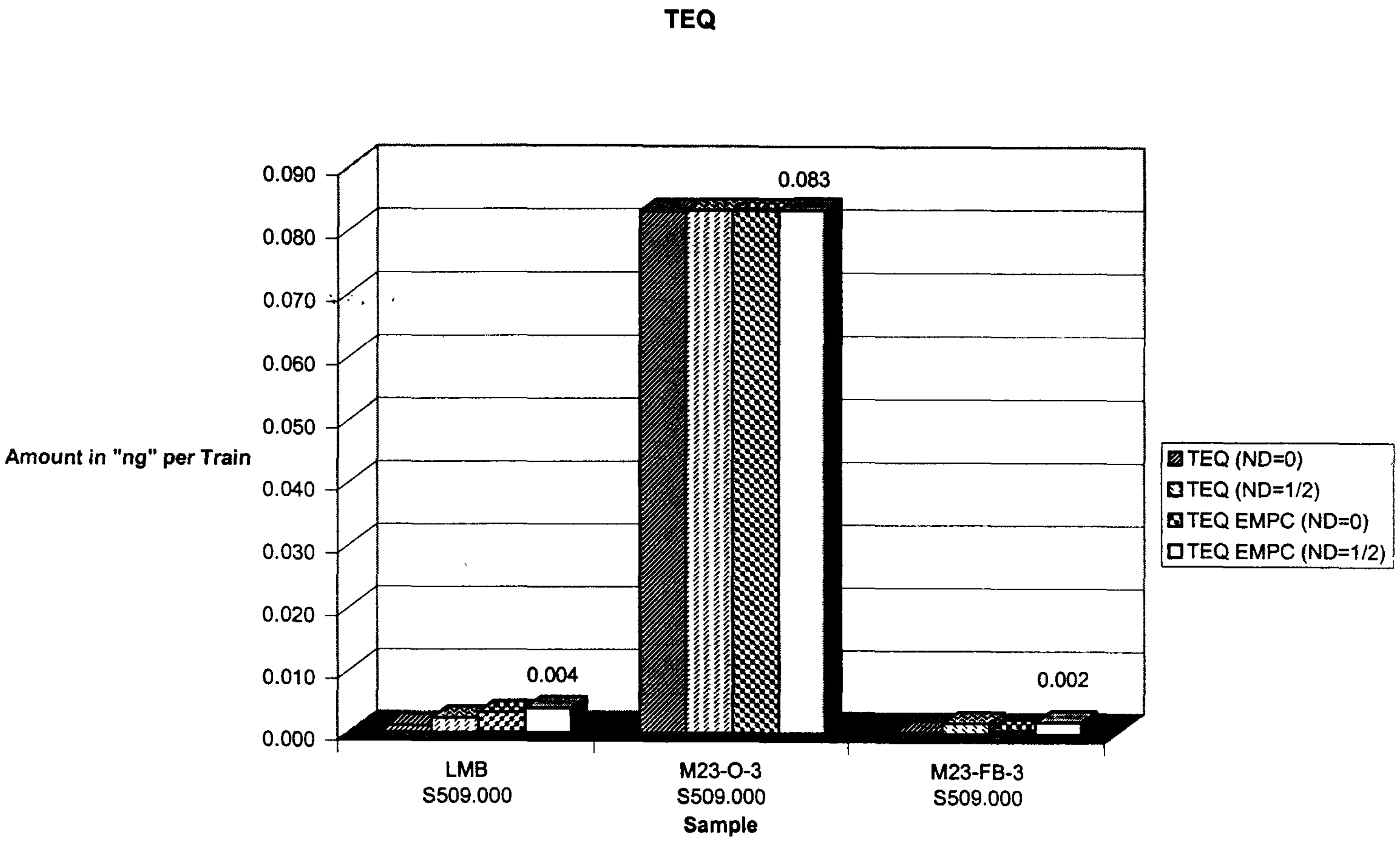


Figure 1: Graphical representation of the TEQs based on the data presented in Table 2

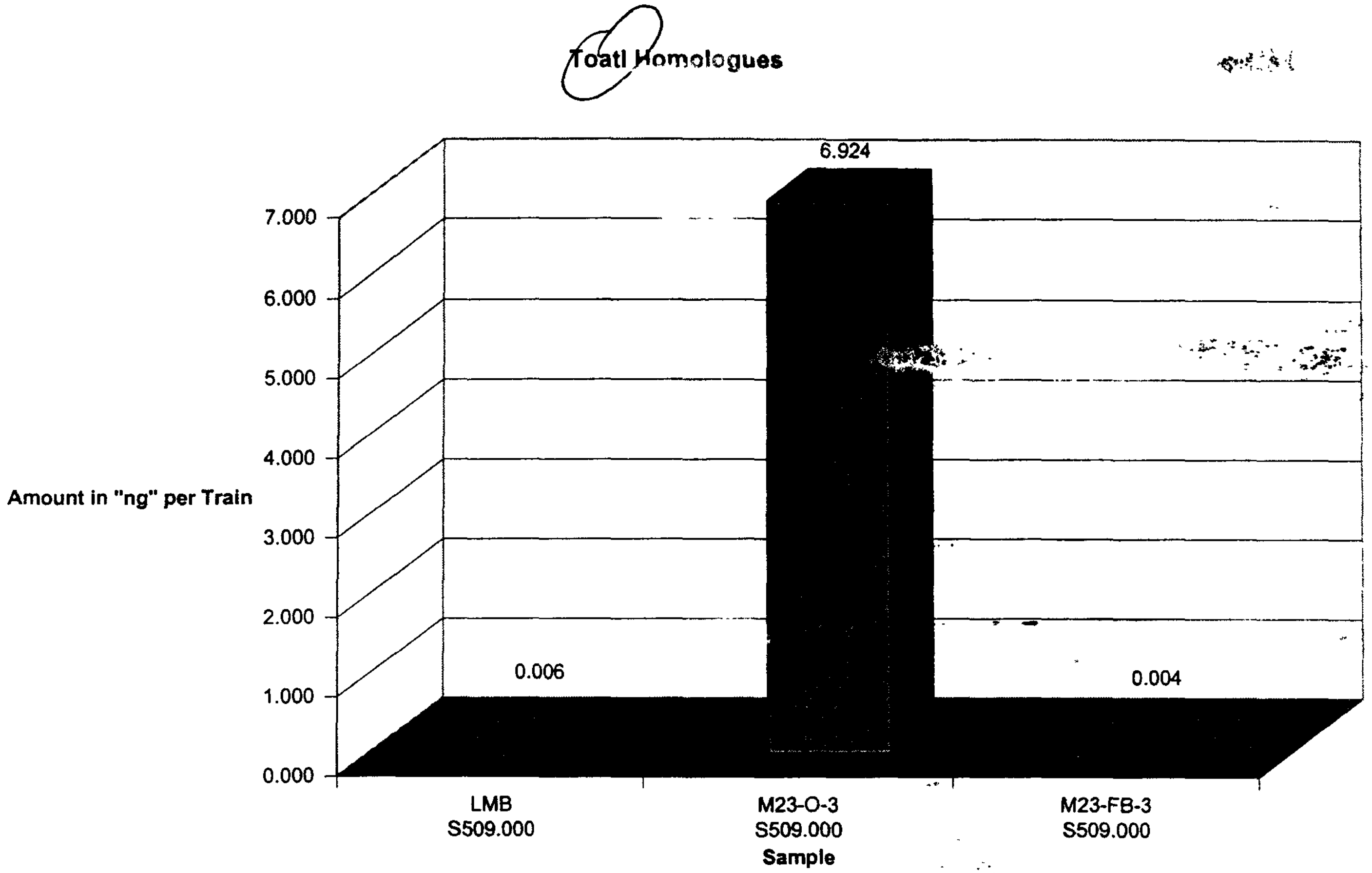


Figure 2: Graphical representation of the totals (tetra- through octachlorinated congeners) based on the data presented in Table 2.

**PAL Project No.: L-1114**

# Paradigm Analytical Labs

## Section 2

## Project Overview

## &

## Sample Tracking & Communication Forms

# Project Overview for the Analysis of Polychlorinated Dibenzo-*p*-Dioxins & Dibenzofurans

No. of Field Samples: 2
No. of Billable Samples: 2

**PAL Project No.: L-1114**

Date Received: 08 SEP 98
Due Date: 30 SEP 98
Client Project ID: S509.000

*Method 23* *Method 23* *Method 23* *Method 23* *Method 23*

**Probe Rinse**

**Concentration** *SOP SP-N-02*

**Filter**

**XAD**
*Sampling Modules Prep. Project No.:* L-1102

S02 wm 9/16/98

**Add** *M23-ES-091598-~~S01~~-162*
**Vol.: 40 µ L ; Conc.: 0.1 ng/ µ l** *SOP SP-S-01*

**Spike Profile**

| | | |
|---|---|---|
| ES: | 23 | 4 ng (1-2) |
| SS: | 23 | 4 ng (1-1) |
| JS: | 23 | 2 ng (1) |

Tridecane batch No.: 25-MAR-98
Thimbles batch No.: 26-FEB-98
Toluene batch No.: 976049
Pre-Soxhlet: 100 hrs
Others:

**Soxhlet 16 H Toluene** *SOP SP-E-01*

**Concentration & Solvent Exchange** *SOP SP-N-01*

**Split Extract** *SOP SP-D-01*

Hexane batch No. 973742
$CH_2Cl_2$ batch No.: 980179
Silica batch No.: 31-JUL-98
Alumina batch No.: N/A
J-F batch No.: SP1407
$SO_4$ batch No.: 12-MAR-98

**Archive 50%** *SOP SP-D-01*

**Fractionation** *SOP SP-U-03*

**Concentration** *SOP SP-N-01*

2 wm 9/21/98

**Add** *M23-JS-091498-S0~~1~~-160*
**Vol.: 20 µ L ; Conc.: 0.1 ng/ µ L** *SOP SP-S-01*

**HRGC - HRMS** *SOP SP-A-01*

Special Instructions:

# Project Overview for the Analysis of Polychlorinated Dibenzo-*p*-Dioxins & Dibenzofurans

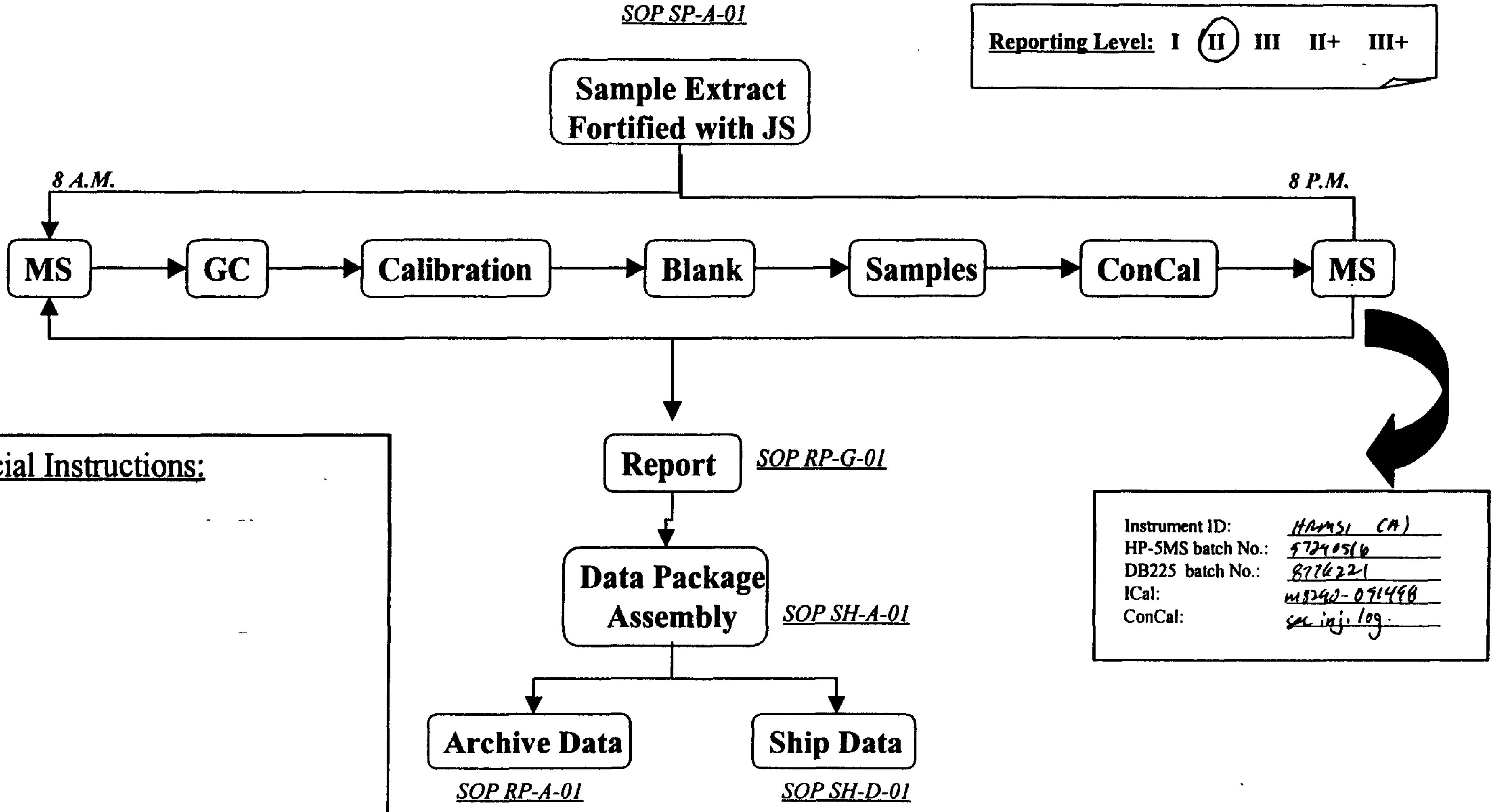
**No. of Field Samples:** 2
**No. of Billable Samples:** 2

**PAL Project No.:** L-1114

**Date Received:** 08 SEP 98
**Due Date:** 30 SEP 98
**Client Project ID:** S509.000

***Method 23*** ***Method 23*** ***Method 23*** ***Method 23*** ***Method 23***

**Reporting Level:** I [✓ II] III II+ III+



Instrument ID: HRMS1 (A)
HP-5MS batch No.: 57240516
DB225 batch No.: 8776221
ICal: m8290-091498
ConCal: see inj. log.

Special Instructions:

# Sample Tracking for the Analysis of Polychlorinated Dibenzo-*p*-Dioxins & Dibenzofurans

**No. of Field Samples:** 2
**Page** 1 **of** 1

**PAL Project No.: L-1114**

**Date Received:** 08 SEP 98
**Due Date:** 30 SEP 98
**Client Project ID:** S509.000

*Method 23* *Method 23* *Method 23* *Method 23* *Method 23*

| Lab Sample ID | Client Sample ID | Observations (use attached communication exchanges form if needed) | ES | SDS | Conc. S.Ex. | Split Arch. | PCU | Conc. | JS | TCDF | Misc. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| L-1114-0 | Lab Method Blank | see ~~1113-0~~ G.T. LAB 09169Y-5723 30SEP98 | N/A | N/A | N/A | N/A | N/A | N/A | N/A | N/A | |
| L-1114-1 | M23-O-3 | ~200 mL DC; ~250 uL Tol. | 9/14/98 | 9/16/98 | 9/21 | 9/21 | 9/21 | 9/21 | 9/21 | Needs CC ✓ 7/29 | |
| L-1114-2 | M23-FB-3 | ~100 mL DC/AC; ~100 mL Tol. | ↓ | ↓ | ↓ | ↓ | ↓ | ↓ | ↓ | N/A | |

30Sep98

# Communication Exchanges Form for the Analysis of PCDD/PCDFs

**No. of Field Samples:** _2_
**Page** _1_ **of** _1_

**PAL Project No.: L-1114**

**Date Received:** 08 SEP 98
**Due Date:** 30 SEP 98
**Client Project ID:** S509.000

| *Method 23* | *Method 23* | *Method 23* | *Method 23* | *Method 23* |
|---|---|---|---|---|

20Sep98

## Paradigm Analytical Labs

### Login Report (ln01)

*Aug. 06, 1998*
*10:30 AM*

**Login Number:** L1114
**Account:** 1027 Pacific Environmental Services, Ir
**Project:** S509.000 US EPA Lime Kiln Screening- OH **Page:** 1 of 1

| Laboratory Sample Number | Client Sample Number | Collect Date | Receive Date | PR | Due Date | Comments |
|---|---|---|---|---|---|---|
| L1114-1 | M23-O-3 | 31-AUG-98 | 08-SEP-98 | | 29-SEP-98 | |
| StackAir | P 23-TO | Hold: | | | | |
| StackAir | C 8290-TO-FT | Hold: 07-SEP-98 | 4 oz. Glass | 1 | Bottles | |
| StackAir | C 8290-TO-SL | Hold: 07-SEP-98 | 4 oz. Glass | 1 | Bottles | |
| L1114-2 | M23-FB-3 | 31-AUG-98 | 08-SEP-98 | | 29-SEP-98 | |
| StackAir | P 23-TO | Hold: | | | | |
| StackAir | C 8290-TO-FT | Hold: 07-SEP-98 | 4 oz. Glass | 1 | Bottles | |
| StackAir | C 8290-TO-SL | Hold: 07-SEP-98 | 4 oz. Glass | 1 | Bottles | |
| L1114-3 | M23-RB | 02-SEP-98 | 08-SEP-98 | | 29-SEP-98 | Report only -<br>Copy only |
| StackAir | P 23-TO | Hold: | | | | |
| StackAir | C 8290-TO-FT | Hold: 09-SEP-98 | 4 oz. Glass | 1 | Bottles | |
| StackAir | C 8290-TO-SL | Hold: 09-SEP-98 | 4 oz. Glass | 1 | Bottles | |

**Signature :** 
**Date :** 09-SEP-98

# Paradigm
## Sample Receipt Checklist

**Client:** 1027

**Client Project ID:** S509.000 **Lab Project:** L1114

| No | Check | Description | Notes |
|---|---|---|---|
| 1 | YES / [✓ NO]<br>[✓ YES] / NO | Shipped<br>Hand Delivered | |
| 2 | [✓ YES] / NO<br>YES / NO | COC Present on Receipt<br>Additional Transmittal Form | |
| 3 | YES / [✓ NO] | COC Tape on Shipping Container | |
| 4 | [✓ YES] / NO | Samples Intact | |
| 5 | °C | Temperature | |
| 6 | [✓ YES] / NO | Sufficient Sample Submitted | |
| 7 | [✓ YES] / NO<br>YES / NO | Samples Preserved Correctly<br>No Preservative Noted<br>N/A (none recommeded) | |
| 8 | [✓ YES] / NO | Received within Holding time<br>N/A | |
| 9 | YES / [✓ NO] | Discrepancies between COC & Label<br>N/A (no COC Received) | |

Note : Use this form to record, comment and report any damages, observations (be specific) of signficance or potentially important for the resolution of downstream problems.

**Additional Comments:**

**Inspected & Logged in by:** ayd-

**Date:** 09-SEP-98

**Time:**

L-1113 = WI...

ACIFIC ENVIRONMENTAL SERVICES, INC.

Central Park West
5001 South Miami Boulevard, P.O. Box 12077
Research Triangle Park, North Carolina 27709-2077
(919) 941-0333 FAX: (919) 941-0234

## Chain of Custody Record

Num S509.000

Project Name US EPA Lime Kiln Screening - Ohio Lime

ers: Abernathy, Gay, Maret, D.D Holzschuh, Siegal, Stewart

Analysis Requested

| ate | Time | Field Sample ID | Sample Description | PCDDs | PCDFs | | | | | | Remarks |
|---|---|---|---|---|---|---|---|---|---|---|---|
| /28/98 | | M23-O-2-4 | Container No. 4 - XAD Sorbent Resin | • | • | | | | | | Report No. 1 |
| /31/98 | | M23-O-3-1 | Container No. 1 - Filter | • | • | | | | | | Report No. 2 |
| /31/98 | | M23-O-3-2 | Container No. 2 - Train Acetone Rinse | • | • | | | | | | Report No. 2 |
| /31/98 | | M23-O-3-3 | Container No. 3 - Train Toluene Rinse | • | • | | | | | | Report No. 2 |
| /31/98 | | M23-O-3-4 | Container No. 4 - XAD Sorbent Resin | • | • | | | | | | Report No. 2 |
| 9/2/98 | | M23-O-4-1 | Container No. 1 - Filter | • | • | | | | | | Report No. 3 |
| 9/2/98 | | M23-O-4-2 | Container No. 2 - Train Acetone Rinse | • | • | | | | | | Report No. 3 |
| 9/2/98 | | M23-O-4-3 | Container No. 3 - Train Toluene Rinse | • | • | | | | | | Report No. 3 |
| 9/2/98 | | M23-O-4-4 | Container No. 4 - XAD Sorbent Resin | • | • | | | | | | Report No. 3 |
| 3/27/98 | | M23-FB-1-1 | Container No. 1 - Filter | • | • | | | | | | FIELD BLANK 1 - Report No. 1 |
| 3/27/98 | | M23-FB-1-2 | Container No. 2 - Train Acetone Rinse | • | • | | | | | | FIELD BLANK 1 - Report No. 1 |
| 3/27/98 | | M23-FB-1-3 | Container No. 3 - Train Toluene Rinse | • | • | | | | | | FIELD BLANK 1 - Report No. 1 |
| 8/27/98 | | M23-FB-1-4 | Container No. 4 - XAD Sorbent Resin | • | • | | | | | | FIELD BLANK 1 - Report No. 1 |
| 8/28/98 | | M23-FB-2-1 | Container No. 1 - Filter | • | • | | | | | | FIELD BLANK 2 - Report No. 1 |
| 8/28/98 | | M23-FB-2-2 | Container No. 2 - Train Acetone Rinse | • | • | | | | | | FIELD BLANK 2 - Report No. 1 |
| 8/28/98 | | M23-FB-2-3 | Container No. 3 - Train Toluene Rinse | • | • | | | | | | FIELD BLANK 2 - Report No. 1 |
| 8/28/98 | | M23-FB-2-4 | Container No. 4 - XAD Sorbent Resin | • | • | | | | | | FIELD BLANK 2 - Report No. 1 |
| 8/31/98 | | M23-FB-3-1 | Container No. 1 - Filter | • | • | | | | | | FIELD BLANK 3 - Report No. 2 |
| 8/31/98 | | M23-FB-3-2 | Container No. 2 - Train Acetone Rinse | • | • | | | | | | FIELD BLANK 3 - Report No. 2 |

L-111

**PACIFIC ENVIRONMENTAL SERVICES, INC.**

Central Park West
5001 South Miami Boulevard, P.O. Box 12077
Research Triangle Park, North Carolina 27709-2077
(919) 941-0333 FAX: (919) 941-0234

# Chain of Custody Record

ject Num: S509.000

Project Name: US EPA Lime Kiln Screening - Ohio Lime

mplers: Abernathy, Gay, Maret, D.D Holzschuh, Siegal, Stewart

| Date | Time | Field Sample ID | Sample Description | PC DDs | PC DFs | | | | | | Remarks |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Analysis Requested | | | | | | | |
| 8/31/98 | | M23-FB-3-3 | Container No. 3 - Train Toluene Rinse | • | • | | | | | | FIELD BLANK 3 - Report No. 2 |
| 8/31/98 | | M23-FB-3-4 | Container No. 4 - XAD Sorbent Resin | • | • | | | | | | FIELD BLANK 3 - Report No. 2 |
| 9/2/98 | | M23-FB-4-1 | Container No. 1 - Filter | • | • | | | | | | FIELD BLANK 4 - Report No. 3 |
| 9/2/98 | | M23-FB-4-2 | Container No. 2 - Train Acetone Rinse | • | • | | | | | | FIELD BLANK 4 - Report No. 3 |
| 9/2/98 | | M23-FB-4-3 | Container No. 3 - Train Toluene Rinse | • | • | | | | | | FIELD BLANK 4 - Report No. 3 |
| 9/2/98 | | M23-FB-4-4 | Container No. 4 - XAD Sorbent Resin | • | • | | | | | | FIELD BLANK 4 - Report No. 3 |
| 9/2/98 | | M23-RB-1 | Container No. 1 - Filter | • | • | | | | | | REAGENT BLANK - All reports |
| 9/2/98 | | M23-RB-2 | Container No. 2 - Train Acetone Rinse | • | • | | | | | | REAGENT BLANK - All reports |
| 9/2/98 | | M23-RB-3 | Container No. 3 - Train Toluene Rinse | • | • | | | | | | REAGENT BLANK - All reports |
| 9/2/98 | | M23-RB-4 | Container No. 4 - XAD Sorbent Resin | • | • | | | | | | REAGENT BLANK - All reports |

| | | | | | |
|---|---|---|---|---|---|
| linquished by: (Signature) | Date/Time | Received by: (Signature) | Relinquished by: (Signature) | Date/Time | Received by: (Signature) |
| linquished by: (Signature) MDMARET | Date/Time 9/9/98 1740 | Received for lab by: (Signature) YVES TONDEUR | REMARKS | | |

L-11B = 1m
Copy for L1114

15 Trains back.
(one labeled void, 3 un-opened).

ACIFIC ENVIRONMENTAL SERVICES, INC.

Central Park West
5001 South Miami Boulevard, P.O. Box 12077
Research Triangle Park, North Carolina 27709-2077
(919) 941-0333 FAX: (919) 941-0234

# Chain of Custody Record

| Num<br>S509.000 | | Project Name<br>US EPA Lime Kiln Screening - Ohio Lime | | Analysis Requested | | | | | | | Remarks |
|---|---|---|---|---|---|---|---|---|---|---|---|
| ers:<br>Abernathy, Gay, Maret, D.D Holzschuh, Siegal, Stewart | | | | | | | | | | | |
| ate | Time | Field Sample ID | Sample Description | PCDDs | PCDFs | Estimated amount of extracts | | | | | |
| /27/98 | | M23-I-1-1 | Container No. 1 - Filter | • | • | 25g | | | | | Report No. 1 |
| /27/98 | | M23-I-1-2 | Container No. 2 - Train Acetone Rinse | • | • | | | | | | Report No. 1 |
| /27/98 | | M23-I-1-3 | Container No. 3 - Train Toluene Rinse | • | • | | | | | | Report No. 1 |
| /27/98 | | M23-I-1-4 | Container No. 4 - XAD Sorbent Resin | • | • | | | | | | Report No. 1 |
| /28/98 | | M23-I-2-1 | Container No. 1 - Filter | • | • | 80g | | | | | Report No. 1 |
| /28/98 | | M23-I-2-2 | Container No. 2 - Train Acetone Rinse | • | • | | | | | | Report No. 1 |
| /28/98 | | M23-I-2-3 | Container No. 3 - Train Toluene Rinse | • | • | | | | | | Report No. 1 |
| /28/98 | | M23-I-2-4 | Container No. 4 - XAD Sorbent Resin | • | • | | | | | | Report No. 1 |
| 9/2/98 | | M23-I-4-1 | Container No. 1 - Filter | • | • | 15g | | | | | Report No. 3 |
| 9/2/98 | | M23-I-4-2 | Container No. 2 - Train Acetone Rinse | • | • | | | | | | Report No. 3 |
| 9/2/98 | | M23-I-4-3 | Container No. 3 - Train Toluene Rinse | • | • | | | | | | Report No. 3 |
| 9/2/98 | | M23-I-4-4 | Container No. 4 - XAD Sorbent Resin | • | • | | | | | | Report No. 3 |
| 8/27/98 | | M23-O-1-1 | Container No. 1 - Filter | • | • | | | | | | Report No. 1 |
| 8/27/98 | | M23-O-1-2 | Container No. 2 - Train Acetone Rinse | • | • | | | | | | Report No. 1 |
| 8/27/98 | | M23-O-1-3 | Container No. 3 - Train Toluene Rinse | • | • | | | | | | Report No. 1 |
| 8/27/98 | | M23-O-1-4 | Container No. 4 - XAD Sorbent Resin | • | • | | | | | | Report No. 1 |
| 8/28/98 | | M23-O-2-1 | Container No. 1 - Filter | • | • | | | | | | Report No. 1 |
| 8/28/98 | | M23-O-2-2 | Container No. 2 - Train Acetone Rinse | • | • | | | | | | Report No. 1 |
| 8/28/98 | | M23-O-2-3 | Container No. 3 - Train Toluene Rinse | • | • | | | | | | Report No. 1 |

```
OPUSquan    29-SEP-1998           Page 1

            Paradigm Sample Log                                                             Page 1 of 1

            Data File       S         Sample ID               Acq. Date     Time

            a26sep98m       1      DB-5 Retchk ✓              26-SEP-98  16:46:46
            a26sep98m       2               SB ✓              26-SEP-98  17:32:59
            a26sep98m       3          1613-CS1               26-SEP-98  18:19:05
            a26sep98m       4          1613-CS2               26-SEP-98  19:07:33
            a26sep98m       5          1613-CS3               26-SEP-98  19:57:04
            a26sep98m       6          1613-CS4               26-SEP-98  20:45:27
            a26sep98m       7          1613-CS5               26-SEP-98  21:31:33
            a26sep98m       8                SB               26-SEP-98  22:17:35
            a26sep98m       9      1698m23 x1/1               26-SEP-98  23:08:22
            a26sep98m      10      0998m23 x1/1               26-SEP-98  23:54:27
            a26sep98m      11      1698m23 x1/1               27-SEP-98  00:40:41
            a26sep98m      12        1104-0 x1/1              27-SEP-98  01:31:40
            a26sep98m      13 -      1114-1 x1/1              27-SEP-98  02:17:51
            a26sep98m      14 -      1114-2 x1/1              27-SEP-98  03:05:54
            a26sep98m      15        1104-1 x1/2              27-SEP-98  03:53:14
            a26sep98m      16        1104-2 x1/2              27-SEP-98  04:39:51
            a26sep98m      17        1104-3 x1/2              27-SEP-98  05:32:01
            a26sep98m      18        1104-4 x1/2              27-SEP-98  06:26:03
            a26sep98m      19        1104-5 x1/2              27-SEP-98  07:15:16
            a26sep98m      20        1104-6 x1/2              27-SEP-98  08:01:16
            a26sep98m      21            BE CS3 ✓             27-SEP-98  08:49:13
```

```
OPUSquan   30-SEP-1998            Page 1
           Paradigm Sample Log                                                                 Page 1 of 1

           Data File        S           Sample ID                  Acq. Date      Time

           a29sep98m        1        B-225 Retchk ✓                29-SEP-98  16:13:59
           a29sep98n        1                 CS3 ✓                29-SEP-98  17:07:50
           a29sep98n        2                  SB                  29-SEP-98  17:49:53
           a29sep98n        3              1120-2                  29-SEP-98  18:31:58
           a29sep98n        4              1115-1                  29-SEP-98  19:16:01
           a29sep98n        5              1115-2                  29-SEP-98  19:58:07
           a29sep98n        6              1115-4                  29-SEP-98  20:40:14
           a29sep98n        7              1114-1                  29-SEP-98  21:24:44
           a29sep98n        8              1113-1                  29-SEP-98  22:06:58
           a29sep98n        9              1113-2                  29-SEP-98  22:49:04
           a29sep98n       10              1113-3                  29-SEP-98  23:31:14
           a29sep98n       11              1113-4                  30-SEP-98  00:15:21
           a29sep98n       12              1113-8                  30-SEP-98  01:01:50
           a29sep98n       13              1113-9                  30-SEP-98  01:43:55
           a29sep98n       14              1104-6                  30-SEP-98  02:26:01
           a29sep98n       15                 CS3 ✓                30-SEP-98  03:08:06
```

# Paradigm Analytical Labs

## Section 3

## Analytical Results

### Documentation for the Analysis
### of
### Polychlorinated Dibenzo-*p*-Dioxins & Dibenzofurans

Method 23
**LMB**
PES

## Analytical Data Summary Sheet

| Analyte | Concentration (ng) | DL (ng) | EMPC (ng) | RT (min.) | Ratio | Qualifier |
|---|---|---|---|---|---|---|
| 2,3,7,8--TCDD | ND | 0.0012 | | | | |
| 1,2,3,7,8-PeCDD | 0.0018 | 0.0007 | | 33:12 | 1.64 | |
| 1,2,3,4,7,8-HxCDD | EMPC | 0.0013 | 0.0018 | 35:15 | 1.01 | |
| 1,2,3,6,7,8-HxCDD | 0.0019 | 0.0010 | | 35:24 | 1.06 | |
| 1,2,3,7,8,9-HxCDD | EMPC | 0.0011 | 0.0018 | 35:36 | 0.97 | |
| 1,2,3,4,6,7,8-HpCDD | ND | 0.0017 | | 37:48 | 0.84 | |
| OCDD | ND | 0.0096 | | 40:44 | 0.26 | |
| 2,3,7,8--TCDF | ND | 0.0016 | | 28:27 | 0.67 | |
| 1,2,3,7,8-PeCDF | 0.0023 | 0.0008 | | 32:35 | 1.34 | |
| 2,3,4,7,8-PeCDF | EMPC | 0.0007 | 0.0020 | 33:01 | 1.86 | |
| 1,2,3,4,7,8-HxCDF | EMPC | 0.0008 | 0.0016 | 34:48 | 0.84 | |
| 1,2,3,6,7,8-HxCDF | EMPC | 0.0006 | 0.0013 | 34:52 | 0.97 | |
| 2,3,4,6,7,8-HxCDF | EMPC | 0.0007 | 0.0013 | 35:14 | 2.28 | |
| 1,2,3,7,8,9-HxCDF | EMPC | 0.0008 | 0.0018 | 35:45 | 1.64 | |
| 1,2,3,4,6,7,8-HpCDF | EMPC | 0.0022 | 0.0030 | 36:59 | 1.78 | |
| 1,2,3,4,7,8,9-HpCDF | ND | 0.0028 | | 38:11 | 0.74 | |
| OCDF | ND | 0.0041 | | | | |
| Total TCDDs | ND | 0.0012 | | | | |
| Total PeCDDs | 0.0016 | 0.0007 | 0.0024 | | | |
| Total HxCDDs | 0.0020 | 0.0010 | 0.0060 | | | |
| Total HpCDDs | ND | 0.0017 | 0.0020 | | | |
| Total TCDFs | ND | 0.0016 | | | | |
| Total PeCDFs | 0.0024 | 0.0007 | 0.0044 | | | |
| Total HxCDFs | ND | 0.0006 | 0.0060 | | | |
| Total HpCDFs | ND | 0.0022 | 0.0052 | | | |
| TEQ (ND=0) | 0.0012 | | 0.0032 | | | ITEF |
| TEQ (ND=1/2) | 0.0023 | | 0.0039 | | | ITEF |

### Client Information

| | |
|---|---|
| Project Name: | S509.000 |
| Sample ID: | LMB |

### Laboratory Information

| | |
|---|---|
| Project ID: | L1115 |
| Sample ID: | lmb091698m23 |
| Collection Date: | NA |
| Receipt Date: | NA |
| Extraction Date: | 16-Sep-98 |
| Analysis Date: | 27-Sep-98 |

### Sample Information

| | | |
|---|---|---|
| Matrix: | Air | |
| Weight / Volume: | 1 | |
| Moisture / Lipids: | 0.0 | % |

| | |
|---|---|
| Filename: | a26sep98m-11 |
| Retchk: | a26sep98m-1 |
| Begin ConCal: | a26sep98m-12 |
| End ConCal: | a26sep98m-21 |
| Initial_Cal: | a26sep98m-21 |

0

*Method 23*

**LMB**

PES

## Analytical Data Summary Sheet

| Labeled Standard | Expected Amount (ng) | Measured Amount (ng) | Percent Recovery (%) | RT (min.) | Ratio | Qualifier |
|---|---|---|---|---|---|---|
| **Extraction Standards** | | | | | | |
| $^{13}C_{12}$-2,3,7,8-TCDD | 4 | 3.48 | 87.1 | 29:25 | 0.8 | |
| $^{13}C_{12}$-1,2,3,7,8-PeCDD | 4 | 4.29 | 107.2 | 33:13 | 1.59 | |
| $^{13}C_{12}$-1,2,3,6,7,8-HxCDD | 4 | 3.94 | 98.5 | 35:22 | 1.28 | |
| $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD | 4 | 3.33 | 83.1 | 37:48 | 1.05 | |
| $^{13}C_{12}$-OCDD | 8 | 5.36 | 67.0 | 40:44 | 0.92 | |
| $^{13}C_{12}$-2,3,7,8-TCDF | 4 | 2.98 | 74.6 | 28:23 | 0.8 | |
| $^{13}C_{12}$-1,2,3,7,8-PeCDF | 4 | 2.79 | 69.7 | 32:34 | 1.6 | |
| $^{13}C_{12}$-1,2,3,6,7,8-HxCDF | 4 | 3.43 | 85.8 | 34:47 | 0.52 | |
| $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF | 4 | 2.20 | 54.9 | 36:59 | 0.44 | |
| **Sampling Standards** | | | | | | |
| $^{37}Cl_4$-2,3,7,8-TCDD | 4 | 4.29 | 107.3 | | | |
| $^{13}C_{12}$-2,3,4,7,8-PeCDF | 4 | 5.86 | 146.5 | 33:01 | 1.58 | |
| $^{13}C_{12}$-1,2,3,4,7,8-HxCDD | 4 | 3.70 | 92.5 | 35:19 | 1.25 | |
| $^{13}C_{12}$-1,2,3,4,7,8-HxCDF | 4 | 3.44 | 85.9 | 34:52 | 0.53 | |
| $^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF | 4 | 6.76 | 169.0 | 38:11 | 0.42 | |
| **Injection Standards** | | | | | | |
| $^{13}C_{12}$-1,2,3,4-TCDD | | | | 29:08 | 0.8 | |
| $^{13}C_{12}$-1,2,3,7,8,9-HxCDD | | | | 35:36 | 1.21 | |

### Client Information

Project Name: S509.000
Sample ID: LMB

### Laboratory Information

Project ID: L1115
Sample ID: lmb091698m23

Collection Date: NA
Receipt Date: NA
Extraction Date: 16-Sep-98
Analysis Date: 27-Sep-98

### Sample Information

Matrix: Air
Weight / Volume: 1
Moisture / Lipids: 0.0 %

Filename: a26sep98m-11
Retchk: a26sep98m-1
Begin ConCal: a26sep98m-12
End ConCal: a26sep98m-21
Initial Cal: a26sep98m-21

Reviewed by: Y.T.

Date Reviewed: 30 Sep 98

```
OPUSquan    30-SEP-1998              Page 1

         Filename ; a26sep98m
           Sample ; 11
         Acquired ; 27-SEP-98   00:40:41
        Processed ; 28-SEP-98   12:04:24
        Sample ID ; lmb091698m23 x1/1
        Cal Table ; m8290-092698m
    Results Table ; M8290-092698M-BE -
         Comments ;
Typ  ;                     Name;      Resp;     Ion 1;     Ion 2;   RA;?;       RT;      Conc;       DL;    S/N1;?;      S/N2;? ; mod?
Unk  ;           2,3,7,8-TCDD;         *;         *;         *;      *;n;NotFnd;          *;   0.0304;        *;n;        *;n ;   no
Unk  ;        1,2,3,7,8-PeCDD; 1.31e+05; 8.16e+04; 4.99e+04; 1.64;y;  33:13;      0.044;   0.0171;         6;y;        13;y ;   no
Unk  ;      1,2,3,4,7,8-HxCDD; 9.56e+04; 4.80e+04; 4.76e+04; 1.01;n;  35:19;      0.045;   0.0332;         6;y;         5;y ;   no
Unk  ;      1,2,3,6,7,8-HxCDD; 1.35e+05; 6.94e+04; 6.54e+04; 1.06;y;  35:24;      0.048;   0.0248;         8;y;         6;y ;   no
Unk  ;      1,2,3,7,8,9-HxCDD; 1.20e+05; 5.90e+04; 6.06e+04; 0.97;n;  35:36;      0.045;   0.0264;         9;y;         5;y ;   no
Unk  ;    1,2,3,4,6,7,8-HpCDD; 8.38e+04; 3.84e+04; 4.55e+04; 0.84;n;  37:48;      0.052;   0.0426;         4;y;         6;y ;   no
Unk  ;                   OCDD; 8.61e+04; 1.80e+04; 6.80e+04; 0.26;n;  40:44;      0.098;   0.2391;         1;n;        10;y ;   no

Unk  ;           2,3,7,8-TCDF; 4.35e+04; 1.74e+04; 2.60e+04; 0.67;y;  28:27;      0.012;   0.0387;         2;n;         1;n ;   no
Unk  ;        1,2,3,7,8-PeCDF; 1.60e+05; 9.14e+04; 6.82e+04; 1.34;y;  32:35;      0.058;   0.0188;        21;y;         5;y ;   no
Unk  ;        2,3,4,7,8-PeCDF; 1.58e+05; 1.03e+05; 5.52e+04; 1.86;n;  33:01;      0.051;   0.0169;        26;y;         5;y ;   no
Unk  ;      1,2,3,4,7,8-HxCDF; 1.12e+05; 5.12e+04; 6.10e+04; 0.84;n;  34:48;      0.039;   0.0198;         6;y;         7;y ;   no
Unk  ;      1,2,3,6,7,8-HxCDF; 1.32e+05; 6.50e+04; 6.67e+04; 0.97;n;  34:52;      0.033;   0.0143;         5;y;         6;y ;   no
Unk  ;      2,3,4,6,7,8-HxCDF; 1.04e+05; 7.20e+04; 3.15e+04; 2.28;n;  35:14;      0.032;   0.0174;         5;y;         4;y ;   no
Unk  ;      1,2,3,7,8,9-HxCDF; 1.25e+05; 7.77e+04; 4.73e+04  1.64;n;  35:45;      0.046;   0.0209;         5;y;         4;y ;   no
Unk  ;    1,2,3,4,6,7,8-HpCDF; 1.23e+05; 7.85e+04; 4.42e+04  1.78;n;  36:59;      0.074;   0.0550;         6;y;         5;y ;   no
Unk  ;    1,2,3,4,7,8,9-HpCDF; 8.35e+04; 3.54e+04  4.81e+04  0.74;n;  38:11;      0.064;   0.0698;         3;y;         4;y ;   no
Unk  ;                   OCDF;         *;         *:         *      *;n;NotFnd;          *;    .1014;        *;n;        *;n ;   no

ES/RT;       13C-2,3,7,8-TCDD; 2.65e+08; 1.18e+08; 1.47e+08; 0.80;y;  29:25;     87.069;   0.0985;      1411;y;      4061;y ;   no
ES   ;    13C-1,2,3,7,8-PeCDD; 2.22e+08; 1.36e+08; 8.59e+07; 1.59;y;  33:13;    107.152;   0.0804;      4769;y;     13050;y ;   no
ES   ;  13C-1,2,3,6,7,8-HxCDD; 2.90e+08; 1.63e+08; 1.27e+08; 1.28;y;  35:22;     98.483;   0.0659;      6456;y;      4314;y ;   no
ES   ;13C-1,2,3,4,6,7,8 HpCDD; 1.63e+08; 8.37e+07; 7.96e+07; 1.05;y;  37:48;     83.137;   0.1752;       932;y;       261;y ;   no
ES   ;               13C-OCDD; 1.63e+08; 7.85e+07; 8.49e+07; 0.92;y;  40:44;    133.962;   0.0643;      4479;y;        65;y ;   no

ES/RT;       13C-2,3,7,8-TCDF; 3.40e+08; 1.51e+08; 1.89e+08; 0.80;    28:23;     74.547;   0.0595;      3693;y;        14;y ;   no
ES   ;    13C-1,2,3,7,8-PeCDF; 2.87e+08; 1.77e+08; 1.11e+08; 1.60;    32:34;     69.717;   0.0139;     36158;y;        15;y ;   no
ES   ;  13C-1,2,3,6,7,8-HxCDF; 2.29e+08; 7.87e+07; 1.50e+08; 0.52;    34:47;     85.765;   1.1364;       408;y;        84;y ;   no
ES   ;13C-1,2,3,4,6,7,8-HpCDF; 1.20e+08; 3.64e+07; 8.36e+07; 0.44;    36:59;     54.905;   0.2712;       317;y;        08;y ;   no

JS   ;       13C-1,2,3,4-TCDD; 2.95e+08; 1.31e+08; 1.64e+08; 0.80;    29:08;     60.498;        -;      1872;y;        68;y ;   no
JS   ;  13C-1,2,3,7,8,9-HxCDD; 2.64e+08; 1.44e+08; 1.20e+08; 1.21;    35:36;     62.791;        -;      5089;y;        75;y ;   no

CS   ;      37Cl-2,3,7,8-TCDD; 2.94e+08; 2.94e+08;         -;      -;-;NotFnd;     93.403;   0.0538;      4361;y;        -; -;   no
CS   ;     13C-2,3,4,7,8-PeCDF; 4.40e+08; 2.70e+08; 1.70e+08; 1.58;y;  33:01;    102.151;   0.0133;     57554;y;     30852;y ;   no
CS   ;  13C-1,2,3,4,7,8-HxCDD; 1.59e+08; 8.87e+07; 7.08e+07; 1.25;y;  35:19;     91.131;   0.1109;      5155;y;      3356;y ;   no
CS   ;  13C-1,2,3,4,7,8-HxCDF; 2.85e+08; 9.83e+07; 1.86e+08; 0.53;y;  34:52;     73.679;   0.7845;       325;y;       301;y ;   no
CS   ;13C-1,2,3,4,7,8,9-HpCDF; 1.56e+08; 4.62e+07; 1.09e+08; 0.42;y;  38:11;     92.759;   0.3535;       369;y;      1361;y ;   no

SS   ;      37Cl-2,3,7,8-TCDD; 2.94e+08; 2.94e+08;         -;      -;-;NotFnd;    107.274;   0.0741;      4361;y;        -; -;   no
SS   ;     13C-2,3,4,7,8-PeCDF; 4.40e+08; 2.70e+08; 1.70e+08; 1.58;y;  33:01;    146.522;   0.0106;     57554;y;     30852;y ;   no
SS   ;  13C-1,2,3,4,7,8-HxCDD; 1.59e+08; 8.87e+07; 7.08e+07; 1.25;y;  35:19;     92.535;   0.0953;      5155;y;      3356;y ;   no
SS   ;  13C-1,2,3,4,7,8-HxCDF; 2.85e+08; 9.83e+07; 1.86e+08; 0.53;y;  34:52;     85.908;   0.5297;       325;y;       301;y ;   no
SS   ;13C-1,2,3,4,7,8,9-HpCDF; 1.56e+08; 4.62e+07; 1.09e+08; 0.42;y;  38:11;    168.945;   0.5999;       369;y;      1361;y ;   no
```

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                                                                 Page 1 of 8

 Ent: 39 Name: Total Tetra-Furans  F:1  Mass: 303.902 305.899 Mod? no   #Hom:4

 Run: 4  File: a26sep98m    S:11 Acq:27-SEP-98 00:40:41 Proc:28-SEP-98 12:04:24
 Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
 Version: V3.6 31-JUL-1998 10:51:59  Sample text: lmb091698m23 x1/1

   Amount: 0.02    of which 0.01    named and 0.01    unnamed
     Conc: 0.02    of which 0.01    named and 0.01    unnamed
   Tox #1: -          Tox #2: -          Tox #3: -

Name                 #     RT Respnse    RA     Conc    Area  Height     S/N  Mod?

                     1  27:00 8.5e+03  0.44 n    0.00
                              8.5e+03                 2.6e+03 1.6e+03 4.8e-01 n  n
                                                      5.9e+03 3.5e+03 5.7e-01 n  n
2,3,7,8-TCDF         2  28:27 4.3e+04  0.67 y    0.01
                              4.3e+04                 1.7e+04 6.0e+03 1.8e+00 n  n
                                                      2.6e+04 8.6e+03 1.4e+00 n  n
                     3  28:37 1.2e+04  0.59 n    0.00
                              1.2e+04                 4.3e+03 2.0e+03 6.0e-01 n  n
                                                      7.4e+03 3.2e+03 5.2e-01 n  n
                     4  28:54 1.5e+04  1.22 n    0.00
                              1.5e+04                 8.4e+03 3.7e+03 1.1e+00 n  n
                                                      6.9e+03 5.5e+03 9.0e-01 n  n


                                                                 Page 2 of 8

 Ent: 40 Name: Total Tetra-Dioxins F:1  Mass: 319.897 321.894 Mod? no   #Hom:4

 Run: 4  File: a26sep98m    S:11 Acq:27-SEP-98 00:40:41 Proc:28-SEP-98 12:04:24
 Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
 Version: V3.6 31-JUL-1998 10:51:59  Sample text: lmb091698m23 x1/1

   Amount: 0.11    of which *       named and 0.11    unnamed
     Conc: 0.11    of which *       named and 0.11    unnamed
   Tox #1: -          Tox #2: -          Tox #3: -

Name                 #     RT Respnse    RA     Conc    Area  Height     S/N  Mod?

                     1  28:07 1.1e+04  1.27 n    0.00
                              1.1e+04                 6.3e+03 4.8e+03 1.3e+00 n  n
                                                      4.9e+03 1.9e+03 9.4e-01 n  n
                     2  28:23 7.5e+04  3.16 n    0.02
                              7.5e+04                 5.7e+04 1.2e+04 3.3e+00 y  n
                                                      1.8e+04 4.7e+03 2.3e+00 n  n
                     3  29:25 2.4e+05  0.08 n    0.08
                              2.4e+05                 1.8e+04 8.1e+03 2.2e+00 n  n
                                                      2.2e+05 4.1e+04 2.1e+01 y  n
                     4  29:38 1.6e+04  0.37 n    0.01
                              1.6e+04                 4.2e+03 2.6e+03 7.0e-01 n  n
                                                      1.1e+04 3.5e+03 1.8e+00 n  n


                                                                 Page 3 of 8

 Ent: 41 Name: Total Penta-Furans  F:2  Mass: 339.860 341.857 Mod? no   #Hom:5

 Run: 4  File: a26sep98m    S:11 Acq:27-SEP-98 00:40:41 Proc:28-SEP-98 12:04:24
 Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
 Version: V3.6 31-JUL-1998 10:51:59  Sample text: lmb091698m23 x1/1

   Amount: 0.12    of which 0.11    named and 0.01    unnamed
     Conc: 0.12    of which 0.11    named and 0.01    unnamed
   Tox #1: -          Tox #2: -          Tox #3: -

Name                 #     RT Respnse    RA     Conc    Area  Height     S/N  Mod?

1,2,3,7,8-PeCDF      1  32:35 1.6e+05  1.34 y    0.06
```

```
                                1.6e+05                 9.1e+04 4.0e+04 2.1e+01 y  n
                                                        6.8e+04 2.7e+04 5.5e+00 y  n
                           2  32:40 1.6e+04  0.61 n    0.01
                                1.6e+04                 5.9e+03 3.1e+03 1.6e+00 n  n
                                                        9.7e+03 3.6e+03 7.2e-01 n  n
2,3,4,7,8-PeCDF            3  33:01 1.6e+05  1.86 n    0.05
                                1.6e+05                 1.0e+05 5.0e+04 2.6e+01 y  n
                                                        5.5e+04 2.3e+04 4.6e+00 y  n
                           4  33:29 8.1e+03  0.63 n    0.00
                                8.1e+03                 3.1e+03 1.6e+03 8.4e-01 n  n
                                                        5.0e+03 2.0 [illegible]
                           5  33:33 1.1e+04  1.30 n    0.00
                                1.1e+04                 6.3e+03 2.6e+03 1.3e+00 n  n
                                                        4.9e+03 3.0e+03 6.0e-01 n  n


                                                                         Page 4 of 8

 Ent: 42 Name: Total Penta-Dioxins F:2  Mass: 355.855 357.852 Mod? no   #Hom:4

 Run: 4  File: a26sep98m    S:11 Acq:27-SEP-98 00:40:41 Proc:28-SEP-98 12:04:24
 Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
 Version: V3.6 31-JUL-1998 10:51:59  Sample tex    [illegible]98m23 x1/1

   Amount: 0.08     of which 0.04    named and 0.04    unnamed
     Conc: 0.08     of which 0.04    named and 0.04    unnamed
   Tox #1: -            Tox #2: -         Tox #3: -

Name                       #     RT Respnse     RA     Conc     Area  Height     S/N  Mod?

                           1  32:34 3.1e+04  2.55 n    0.01
                                3.1e+04                 2.3e+04 9.5e+03 2.1e+00 n  n
                                                        8.9e+03 3.8e+03 2.7e+00 n  n
                           2  33:01 7.0e+04  3.11 n    0.02
                                7.0e+04                 5.3e+04 [illegible]
                                                        1.7e+04 5.2e+03 3.7e+00 y  n
                           3  33:05 1.2e+04  1.82 n    0.00
                                1.2e+04                 .9e+03 3.2e+03 1.1e+00 n  n
                                                        4.3e+03 1.3e+03 9.6e-01 n  n
1,2,3,7,8-PeCDD            4  33:13 1.3e+05  1.64 y    0.04
                                1.3e+05                 8.2e+04 2.9e+04 6.5e+00 y  n
                                                        5.0e+04 1.8e+04 1.3e+01 y  n
```

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                                                                  Page 5 of 8

 Ent: 43 Name: Total Hexa-Furans    F:3  Mass: 373.821 375.818 Mod? no   #Hom:17

 Run: 4  File: a26sep98m    S:11 Acq:27-SEP-98 00:40:41 Proc:28-SEP-98 12:04:24
 Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
 Version: V3.6 31-JUL-1998 10:51:59  Sample text: 1mb091698m23 x1/1

   Amount: 0.21    of which 0.15    named and 0.06    unnamed
     Conc: 0.21    of which 0.15    named and 0.06    unnamed
   Tox #1: -           Tox #2: -        Tox #3: -

Name                #     RT Respnse     RA     Conc    Area  Height     S/N  Mod?

1,2,3,4,7,8-HxCDF   1  34:48 1.1e+05  0.84 n    0.04
                                 1.1e+05                      5.1e+04 2.7e+04 5.9e+00 y  n
                                                              6.1e+04 2.3e+04 6.6e+00 y  n
1,2,3,6,7,8-HxCDF   2  34:52 1.3e+05  0.97 n    0.03
                         1.3e+05                      6.5e+04 2.3e+04 5.1e+00 y  n
                                                      6.7e+04 2.2e+04 6.2e+00 y  n
                    3  35:00 1.1e+04  0.63 n    0.00
                         1.1e+04                      4.2e+03 2.2e+03 4.8e-01 n  n
                                                      6.6e+03 2.8e+03 7.8e-01 n  n
                    4  35:03 1.3e+04  1.65 n    0.00
                         1.3e+04                      8.3e+03 5.7e+03 1.3e+00 n  n
                                                      5.1e+03 3.8e+03 1.1e+00 n  n
                    5  35:07 1.1e+04  0.92 n    0.00
                         1.1e+04                      5.5e+03 3.2e+03 7.1e-01 n  n
                                                      5.9e+03 3.1e+03 8.6e-01 n  n
2,3,4,6,7,8-HxCDF   6  35:14 1.0e+05  2.28 n    0.03
                         1.0e+05                      7.2e+04 2.1e+04 4.7e+00 y  n
                                                      3.2e+04 1.4e+04 3.8e+00 y  n
                    7  35:23 2.0e+04  4.59 n    0.01
                         2.0e+04                      1.7e+04 1.2e+04 2.6e+00 n  n
                                                      3.6e+03 1.3e+03 3.6e-01 n  n
                    8  35:25 1.9e+04  4.15 n    0.01
                         1.9e+04                      1.5e+04 5.9e+03 1.3e+00 n  n
                                                      3.6e+03 1.3e+03 3.6e-01 n  n
                    9  35:33 1.1e+04  1.91 n    0.00
                         1.1e+04                      7.0e+03 3.8e+03 8.3e-01 n  n
                                                      3.7e+03 1.5e+03 4.2e-01 n  n
                    10 35:35 2.1e+04  3.35 n    0.01
                         2.1e+04                      1.6e+04 7.7e+03 1.7e+00 n  n
                                                      4.8e+03 1.5e+03 4.3e-01 n  n
1,2,3,7,8,9-HxCDF   11 35:45 1.3e+05  1.64 n    0.05
                         1.3e+05                      7.8e+04 2.4e+04 5.4e+00 y  n
                                                      4.7e+04 1.4e+04 4.1e+00 y  n
                    12 35:53 2.0e+04  1.35 y    0.01
                         2.0e+04                      1.2e+04 4.2e+03 9.2e-01 n  n
                                                      8.7e+03 6.8e+03 1.9e+00 n  n
                    13 36:00 1.5e+04  0.55 n    0.00
                         1.5e+04                      5.3e+03 3.8e+03 8.4e-01 n  n
                                                      9.6e+03 2.9e+03 8.3e-01 n  n
                    14 36:04 1.3e+04  2.63 n    0.00
                         1.3e+04                      9.2e+03 6.2e+03 1.4e+00 n  n
                                                      3.5e+03 2.2e+03 6.3e-01 n  n
                    15 36:06 9.2e+03  1.69 n    0.00
                         9.2e+03                      5.8e+03 2.9e+03 6.5e-01 n  n
                                                      3.4e+03 1.7e+03 4.7e-01 n  n
                    16 36:10 9.2e+03  1.24 y    0.00
                         9.2e+03                      5.1e+03 2.1e+03 4.6e-01 n  n
                                                      4.1e+03 2.1e+03 6.0e-01 n  n
                    17 36:16 1.2e+04  0.64 n    0.00
                         1.2e+04                      4.8e+03 2.7e+03 6.0e-01 n  n
                                                      7.6e+03 2.8e+03 7.9e-01 n  n
```

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 Ent: 44 Name: Total Hexa-Dioxins  F:3  Mass: 389.816 391.813 Mod? no   #Hom:16

 Run: 4  File: a26sep98m    S:11 Acq:27-SEP-98 00:40:41 Proc:28-SEP-98 12:04:24
 Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
 Version: V3.6 31-JUL-1998 10:51:59  Sample text: lmb091698m23 x1/1

   Amount: 0.26    of which 0.14    named and 0.12    unnamed
     Conc: 0.26    of which 0.14    named and 0.12    unnamed
   Tox #1: -            Tox #2: -          Tox #3: -

Name                 #    RT Respnse    RA    Conc    Area  Height    S/N  Mod?

                     1  34:47 6.8e+04  3.96 n    0.03
                                6.8e+04                5.5e+04 2.2e+04 7.8e+00 y  n
                                                       1.4e+04 7.8e+03 1.9e+00 n  n
                     2  34:52 7.6e+04  3.03 n    0.03
                                7.6e+04                5.7e+04 1.9e+04 6.8e+00 y  n
                                                       1.9e+04 7.1e+03 1.8e+00 n  n
                     3  34:56 3.6e+04  1.35 y    0.01
                                3.6e+04                2.0e+04 5.9e+03 2.1e+00 n  n
                                                       1.5e+04 6.6e+03 1.6e+00 n  n
                     4  35:08 1.0e+04  1.05 n    0.00
                                1.0e+04                5.3e+03 2.4e+03 8.7e-01 n  n
                                                       5.1e+03 2.6e+03 6.6e-01 n  n
                     5  35:11 8.1e+03  0.51 n    0.00
                                8.1e+03                2.7e+03 1.2e+03 4.2e-01 n  n
                                                       5.4e+03 2.4e+03 5.9e-01 n  n
                     6  35:14 1.3e+04  1.04 n    0.01
                                1.3e+04                6.4e+03 2.4e+03 8.5e-01 n  n
                                                       6.2e+03 2.5e+03 6.3e-01 n  n
1,2,3,4,7,8-HxCDD    7  35:19 9.6e+04  1.01 n    0.05
                                9.6e+04                4.8e+04 1.6e+04 5.8e+00 y  n
                                                       4.8e+04 [illegible]
1,2,3,6,7,8-HxCDD    8  35:24 1.3e+05  1.06 y    0.05
                                1.3e+05                6.9e+04 2.2e+04 7.8e+00 y  n
                                                       6.5e+04 2.4e+04 6.0e+00 y  n
1,2,3,7,8,9-HxCDD    9  35:36 1.2e+05  0.97 n    0.05
                                1.2e+05                5.9e+04 2.6e+04 9.3e+00 y  n
                                                       6.1e+04 2.0e+04 5.1e+00 y  n
                     10 35:42 2.3e+04  0.41 n    0.01
                                2.3e+04                6.7e+03 4.1e+03 1.5e+00 n  n
                                                       1.6e+04 5.3e+03 1.3e+00 n  n
                     11 35:49 1.8e+04  0.63 n    0.01
                                1.8e+04                7.0e+03 3.6e+03 1.3e+00 n  n
                                                       1.1e+04 5.3e+03 1.3e+00 n  n
                     12 35:56 1.6e+04  0.59 n    0.01
                                1.6e+04                5.8e+03 2.9e+03 1.0e+00 n  n
                                                       9.9e+03 3.8e+03 9.4e-01 n  n
                     13 36:01 9.0e+03  1.41 y    0.00
                                9.0e+03                5.2e+03 2.4e+03 8.5e-01 n  n
                                                       3.7e+03 3.0e+03 7.4e-01 n  n
                     14 36:05 7.8e+03  0.40 n    0.00
                                7.8e+03                2.2e+03 1.4e+03 4.9e-01 n  n
                                                       5.6e+03 2.2e+03 5.5e-01 n  n
                     15 36:13 9.2e+03  0.50 n    0.00
                                9.2e+03                3.1e+03 [illegible]
                                                       6.1e+03 2.7e+03 6.7e-01 n  n
                     16 36:21 8.4e+03  1.38 y    0.00
                                8.4e+03                4.9e+03 1.4e+03 4.9e-01 n  n
                                                       3.5e+03 2.3e+03 5.8e-01 n  n


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 Ent: 45 Name: Total Hepta-Furans  F:4  Mass: 407.782 409.779 Mod? no   #Hom:8

 Run: 4  File: a26sep98m    S:11 Acq:27-SEP-98 00:40:41 Proc:28-SEP-98 12:04:24
 Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
 Version: V3.6 31-JUL-1998 10:51:59  Sample text: lmb091698m23 x1/1
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   Amount: 0.24    of which 0.14    named and 0.10    unnamed
     Conc: 0.24    of which 0.14    named and 0.10    unnamed
   Tox #1: -            Tox #2: -         Tox #3: -

Name                    #     RT Respnse    RA      Conc    Area  Height     S/N  Mod?

1,2,3,4,6,7,8-HpCDF1  36:59 1.2e+05  1.78 n    0.07
                            1.2e+05                   7.9e+04 2.5e+04 5.6e+00 y  n
                                                      4.4e+04 1.8e+04 5.0e+00 y  n
                   2  37:13 2.6e+04  1.02 y    0.02
                            2.6e+04                   1.3e+04 4.3e+03 9.5e-01 n  n
                                                      1.3e+04 3.2e+03 9.0e-01 n  n
                   3  37:20 1.7e+04  1.50 n    0.01
                            1.7e+04                   1.0e+04 4.7e+03 1.0e+00 n  n
                                                      6.8e+03 2.3e+03 6.5e-01 n  n
                   4  37:26 1.4e+04  1.76 n    0.01
                            1.4e+04                   8.7e+03 3.6e+03 8.0e-01 n  n
                                                      5.0e+03 2.5e+03 6.9e-01 n  n
                   5  37:43 1.7e+04  1.62 n    0.01
                            1.7e+04                   1.1e+04 3.7e+03 8.2e-01 n  n
                                                      6.5e+03 2.3e+03 6.5e-01 n  n
                   6  37:55 1.1e+04  1.16 y    0.01
                            1.1e+04                   6.1e+03 2.6e+03 5.8e-01 n  n
                                                      5.2e+03 2.0e+03 5.7e-01 n  n
1,2,3,4,7,8,9-HpCDF7  38:11 8.4e+04  0.74 n    0.06
                            8.4e+04                   3.5e+04 1.5e+04 3.4e+00 y  n
                                                      4.8e+04 1.5e+04 4.1e+00 y  n
                   8  38:13 7.0e+04  0.45 n    0.05
                            7.0e+04                   2.1e+04 8.5e+03 1.9e+00 n  n
                                                      4.8e+04 1.5e+04 4.1e+00 y  n
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 Ent: 46 Name: Total Hepta-Dioxins F:4  Mass: 423.777 425.774 Mod? no   #Hom:3

 Run: 4  File: a26sep98m    S:11 Acq:27-SEP-98 0[illegible]:40:41 Proc:28-SEP-98 12:04:24
 Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
 Version: V3.6 31-JUL-1998 10:51:59  Sample text: lmb091698m23 x1/1

   Amount: 0.09    of which 0.05    named and 0.03    unnamed
     Conc: 0.09    of which 0.05    named and 0.03    unnamed
   Tox #1: -           Tox #2: -         Tox #3: -

Name                 #     RT Respnse     RA    Conc     Area  Height     S/N  Mod?

1,2,3,4,6,7,8-HpCDD1  37:48 8.4e+04  0.84 n    0.05
                            8.4e+04                  3.8e+04 1.3e+04 4.2e+00 y  n
                                                     4.5e+04 1.4e+04 6.4e+00 y  n
                     2  38:11 4.2e+04  4.07 n    0.03
                            4.2e+04                  3.3e+04 8.8e+03 2.9e+00 n  n
                                                     8.2e+03 3.1e+03 1.5e+00 n  n
                     3  38:24 1.3e+04  1.31 n    0.01
                            1.3e+04                  7.6e+03 4.0e+03 1.3e+00 n  n
                                                     [illegible] 2.5e+03 1.2e+00 n  n
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 Ent: 39 Name: Total Tetra-Furans  F:1  Mass: 303.902 305.899 Mod? no   #Hom:4

 Run: 4  File: a26sep98m    S:11 Acq:27-SEP-98 00:40:41 Proc:28-SEP-98 12:04:24
 Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
 Version: V3.6 31-JUL-1998 10:51:59  Sample text: lmb091698m23 x1/1

   Amount: 0.02    of which 0.01    named and 0.01    unnamed
     Conc: 0.02    of which 0.01    named and 0.01    unnamed
   Tox #1: -            Tox #2: -         Tox #3: -

Name                  #     RT Respnse    RA     Conc     Area  Height     S/N  Mod?

                      1  27:00 8.5e+03  0.44 n    0.00
                               8.5e+03                2.6e+03 1.6e+03 4.8e-01 n  n
                                                      5.9e+03 3.5e+03 5.7e-01 n  n
2,3,7,8-TCDF          2  28:27 4.3e+04  0.67 y    0.01
                               4.3e+04                1.7e+04 6.0e+03 1.8e+00 n  n
                                                      2.6e+04 8.6e+03 1.4e+00 n  n
                      3  28:37 1.2e+04  0.59 n    0.00
                               1.2e+04                4.3e+03 2.0e+03 6.0e-01 n  n
                                                      7.4e+03 3.2e+03 5.2e-01 n  n
                      4  28:54 1.5e+04  1.22 n    0.00
                               1.5e+04                8.4e+03 3.7e+03 1.1e+00 n  n
                                                      6.9e+03 5.5e+03 9.0e-01 n  n


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 Ent: 40 Name: Total Tetra-Dioxins F:1  Mass: 319.897 321.894 Mod? no   #Hom:4

 Run: 4  File: a26sep98m    S:11 Acq:27-SEP-98 00:40:41 Proc:28-SEP-98 12:04:24
 Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
 Version: V3.6 31-JUL-1998 10:51:59  Sample text: lmb091698m23 x1/1

   Amount: 0.11    of which *       named and 0.11    unnamed
     Conc: 0.11    of which *       named and 0.11    unnamed
   Tox #1: -            Tox #2: -         Tox #3: -

Name                  #     RT Respnse    RA     Conc     Area  Height     S/N  Mod?

                      1  28:07 1.1e+04  1.27 n    0.00
                               1.1e+04                6.3e+03 4.8e+03 1.3e+00 n  n
                                                      4.9e+03 1.9e+03 9.4e-01 n  n
                      2  28:23 7.5e+04  3.16 n    0.02
                               7.5e+04                5.7e+04 1.2e+04 3.3e+00 y  n
                                                      1.8e+04 4.7e+03 2.3e+00 n  n
                      3  29:25 2.4e+05  0.08 n    0.08
                               2.4e+05                1.8e+04 8.1e+03 2.2e+00 n  n
                                                      2.2e+05 4.1e+04 2.1e+01 y  n
                      4  29:38 1.6e+04  0.37 n    0.01
                               1.6e+04                4.2e+03 2.6e+03 7.0e-01 n  n
                                                      1.1e+04 3.5e+03 1.8e+00 n  n


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 Ent: 41 Name: Total Penta-Furans  F:2  Mass: 339.860 341.857 Mod? no   #Hom:5

 Run: 4  File: a26sep98m    S:11 Acq:27-SEP-98 00:40:41 Proc:28-SEP-98 12:04:24
 Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
 Version: V3.6 31-JUL-1998 10:51:59  Sample text: lmb091698m23 x1/1

   Amount: 0.12    of which 0.11    named and 0.01    unnamed
     Conc: 0.12    of which 0.11    named and 0.01    unnamed
   Tox #1: -            Tox #2: -         Tox #3: -

Name                  #     RT Respnse    RA     Conc     Area  Height     S/N  Mod?

1,2,3,7,8-PeCDF       1  32:35 1.6e+05  1.34 y    0.06
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                                  1.6e+05                     9.1e+04 4.0e+04 2.1e+01 y  n
                                                              6.8e+04 2.7e+04 5.5e+00 y  n
                         2  32:40 1.6e+04  0.61 n     0.01
                                  1.6e+04                     5.9e+03 3.1e+03 1.6e+00 n  n
                                                              9.7e+03 3.6e+03 7.2e-01 n  n
2,3,4,7,8-PeCDF          3  33:01 1.6e+05  1.86 n     0.05
                                  1.6e+05                     1.0e+05 5.0e+04 2.6e+01 y  n
                                                              5.5e+04 2.3e+04 4.6e+00 y  n
                         4  33:29 8.1e+03  0.63 n     0.00
                                  8.1e+03                     3.1e+03 1.6e+03 8.4e-01 n  n
                                                              5.0e+03 2.0e[illegible]
                         5  33:33 1.1e+04  1.30 n     0.00
                                  1.1e+04                     6.3e+03 2.6e+03 1.3e+00 n  n
                                                              4.9e+03 3.0e+03 6.0e-01 n  n


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  Ent: 42 Name: Total Penta-Dioxins F:2  Mass: 355.855 357.852 Mod? no    #Hom:4

  Run: 4  File: a26sep98m     S:11 Acq:27-SEP-98 00:40:41 Proc:28-SEP-98 12:04:24
  Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
  Version: V3.6 31-JUL-1998 10:51:59  Sample text [illegible]1698m23 x1/1

    Amount: 0.08     of which 0.04     named and 0.04     unnamed
      Conc: 0.08     of which 0.04     named and 0.04     unnamed
    Tox #1: -            Tox #2: -            Tox #3: -

Name                     #     RT Respnse      RA     Conc     Area  Height     S/N  Mod?

                         1  32:34 3.1e+04  2.55 n     0.01
                                  3.1e+04                     2.3e+04 9.5e+03 2.1e+00 n  n
                                                              8.9e+03 3.8e+03 2.7e+00 n  n
                         2  33:01 7.0e+04  3.11 n     0.02
                                  7.0e+04                     5.3e+04 [illegible]
                                                              1.7e+04 5.2e+03 3.7e+00 y  n
                         3  33:05 1.2e+04  1.82 n     0.0[illegible]
                                  1.2e+04                     [illegible].9e+03 5.2e+03 1.1e+00 n  n
                                                              4.3e+03 1.3e+03 9.6e-01 n  n
1,2,3,7,8-PeCDD          4  33:13 1.3e+05  1.64 y     0.04
                                  1.3e+05                     8.2e+04 2.9e+04 6.5e+00 y  n
                                                              5.0e+04 1.8e+04 1.3e+01 y  n
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 Ent: 43 Name: Total Hexa-Furans    F:3  Mass: 373.821 375.818 Mod? no    #Hom:17

 Run: 4  File: a26sep98m     S:11 Acq:27-SEP-98 00:40:41 Proc:28-SEP-98 12:04:24
 Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
 Version: V3.6 31-JUL-1998 10:51:59  Sample text: 1mb091698m23 x1/1

   Amount: 0.21     of which 0.15     named and 0.06     unnamed
     Conc: 0.21     of which 0.15     named and 0.06     unnamed
   Tox #1: -              Tox #2: -           Tox #3: -

Name                #     RT Respnse     RA     Conc     Area  Height      S/N  Mod?

1,2,3,4,7,8-HxCDF   1  34:48 1.1e+05  0.84 n    0.04
                             1.1e+05                  5.1e+04 2.7e+04 5.9e+00 y  n
                                                      6.1e+04 2.3e+04 6.6e+00 y  n
1,2,3,6,7,8-HxCDF   2  34:52 1.3e+05  0.97 n    0.03
                             1.3e+05                  6.5e+04 2.3e+04 5.1e+00 y  n
                                                      6.7e+04 2.2e+04 6.2e+00 y  n
                    3  35:00 1.1e+04  0.63 n    0.00
                             1.1e+04                  4.2e+03 2.2e+03 4.8e-01 n  n
                                                      6.6e+03 2.8e+03 7.8e-01 n  n
                    4  35:03 1.3e+04  1.65 n    0.00
                             1.3e+04                  8.3e+03 5.7e+03 1.3e+00 n  n
                                                      5.1e+03 3.8e+03 1.1e+00 n  n
                    5  35:07 1.1e+04  0.92 n    0.00
                             1.1e+04                  5.5e+03 3.2e+03 7.1e-01 n  n
                                                      5.9e+03 3.1e+03 8.6e-01 n  n
2,3,4,6,7,8-HxCDF   6  35:14 1.0e+05  2.28 n    0.03
                             1.0e+05                  7.2e+04 2.1e+04 4.7e+00 y  n
                                                      3.2e+04 1.4e+04 3.8e+00 y  n
                    7  35:23 2.0e+04  4.59 n    0.01
                             2.0e+04                  1.7e+04 1.2e+04 2.6e+00 n  n
                                                      3.6e+03 1.3e+03 3.6e-01 n  n
                    8  35:25 1.9e+04  4.15 n    0.01
                             1.9e+04                  1.5e+04 5.9e+03 1.3e+00 n  n
                                                      3.6e+03 1.3e+03 3.6e-01 n  n
                    9  35:33 1.1e+04  1.91 n    0.00
                             1.1e+04                  7.0e+03 3.8e+03 8.3e-01 n  n
                                                      3.7e+03 1.5e+03 4.2e-01 n  n
                    10 35:35 2.1e+04  3.35 n    0.01
                             2.1e+04                  1.6e+04 7.7e+03 1.7e+00 n  n
                                                      4.8e+03 1.5e+03 4.3e-01 n  n
1,2,3,7,8,9-HxCDF   11 35:45 1.3e+05  1.64 n    0.05
                             1.3e+05                  7.8e+04 2.4e+04 5.4e+00 y  n
                                                      4.7e+04 1.4e+04 4.1e+00 y  n
                    12 35:53 2.0e+04  1.35 y    0.01
                             2.0e+04                  1.2e+04 4.2e+03 9.2e-01 n  n
                                                      8.7e+03 6.8e+03 1.9e+00 n  n
                    13 36:00 1.5e+04  0.55 n    0.00
                             1.5e+04                  5.3e+03 3.8e+03 8.4e-01 n  n
                                                      9.6e+03 2.9e+03 8.3e-01 n  n
                    14 36:04 1.3e+04  2.63 n    0.00
                             1.3e+04                  9.2e+03 6.2e+03 1.4e+00 n  n
                                                      3.5e+03 2.2e+03 6.3e-01 n  n
                    15 36:06 9.2e+03  1.69 n    0.00
                             9.2e+03                  5.8e+03 2.9e+03 6.5e-01 n  n
                                                      3.4e+03 1.7e+03 4.7e-01 n  n
                    16 36:10 9.2e+03  1.24 y    0.00
                             9.2e+03                  5.1e+03 2.1e+03 4.6e-01 n  n
                                                      4.1e+03 2.1e+03 6.0e-01 n  n
                    17 36:16 1.2e+04  0.64 n    0.00
                             1.2e+04                  4.8e+03 2.7e+03 6.0e-01 n  n
                                                      7.6e+03 2.8e+03 7.9e-01 n  n
```

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 Ent: 44 Name: Total Hexa-Dioxins  F:3  Mass: 389.816 391.813 Mod? no   #Hom:16

 Run: 4  File: a26sep98m    S:11 Acq:27-SEP-98 00:40:41 Proc:28-SEP-98 12:04:24
 Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
 Version: V3.6 31-JUL-1998 10:51:59  Sample text: 1mb091698m23 x1/1

   Amount: 0.26    of which 0.14    named and 0.12    unnamed
     Conc: 0.26    of which 0.14    named and 0.12    unnamed
   Tox #1: -            Tox #2: -         Tox #3: -

Name                 #     RT Respnse     RA      Conc     Area  Height      S/N  Mod?

                     1  34:47 6.8e+04  3.96 n    0.03
                                6.8e+04                  5.5e+04 2.2e+04 7.8e+00 y  n
                                                         1.4e+04 7.8e+03 1.9e+00 n  n
                     2  34:52 7.6e+04  3.03 n    0.03
                                7.6e+04                  5.7e+04 1.9e+04 6.8e+00 y  n
                                                         1.9e+04 7.1e+03 1.8e+00 n  n
                     3  34:56 3.6e+04  1.35 y    0.01
                                3.6e+04                  2.0e+04 5.9e+03 2.1e+00 n  n
                                                         1.5e+04 6.6e+03 1.6e+00 n  n
                     4  35:08 1.0e+04  1.05 n
                                1.0e+04                  5.3e+03 2.4e+03 8.7e-01 n  n
                                                         5.1e+03 2.6e+03 6.6e-01 n  n
                     5  35:11 8.1e+03  0.51 n    0.00
                                8.1e+03                  2.7e+03 1.2e+03 4.2e-01 n  n
                                                         5.4e+03 2.4e+03 5.9e-01 n  n
                     6  35:14 1.3e+04  1.04 n    0.01
                                1.3e+04                  6.4e+03 2.4e+03 8.5e-01 n  n
                                                         6.2e+03 2.5e+03 6.3e-01 n  n
1,2,3,4,7,8-HxCDD    7  35:19 9.6e+04  1.01 n    0.05
                                9.6e+04                  4.8e+04 1.6e+04 5.8e+00 y  n
                                                         4.8e+04 [illegible]
1,2,3,6,7,8-HxCDD    8  35:24 1.3e+05  1.06 y    0.05
                                1.3e+05                  6.9e+04 2.2e+04 7.8e+00 y  n
                                                         6.5e+04 2.4e+04 6.0e+00 y  n
1,2,3,7,8,9-HxCDD    9  35:36 1.2e+05  0.97 n    [illegible]
                                1.2e+05                  5.9e+04 2.6e+04 9.3e+00 y  n
                                                         6.1e+04 2.0e+04 5.1e+00 y  n
                    10  35:42 2.3e+04  0.41 n    0.01
                                2.3e+04                  6.7e+03 4.1e+03 1.5e+00 n  n
                                                         1.6e+04 5.3e+03 1.3e+00 n  n
                    11  35:49 1.8e+04  0.63 n    0.01
                                1.8e+04                  7.0e+03 3.6e+03 1.3e+00 n  n
                                                         1.1e+04 5.3e+03 1.3e+00 n  n
                    12  35:56 1.6e+04  0.59 n    0.01
                                1.6e+04                  5.8e+03 2.9e+03 1.0e+00 n  n
                                                         9.9e+03 3.8e+03 9.4e-01 n  n
                    13  36:01 9.0e+03  1.41 y    0.00
                                9.0e+03                  5.2e+03 2.4e+03 8.5e-01 n  n
                                                         3.7e+03 3.0e+03 7.4e-01 n  n
                    14  36:05 7.8e+03  0.40 n    0.00
                                7.8e+03                  2.2e+03 1.4e+03 4.9e-01 n  n
                                                         5.6e+03 2.2e+03 5.5e-01 n  n
                    15  36:13 9.2e+03  0.50 n    0.00
                                9.2e+03                  3.1e+03 1.0e[illegible]
                                                         6.1e+03 2.7e+03 [illegible].7e-01 n  n
                    16  36:21 8.4e+03  1.38 y    0.00
                                8.4e+03                  4.9e+03 1.4e+03 4.9e-01 n  n
                                                         3.5e+03 2.3e+03 5.8e-01 n  n


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 Ent: 45 Name: Total Hepta-Furans  F:4  Mass: 407.782 409.779 Mod? no   #Hom:8

 Run: 4  File: a26sep98m    S:11 Acq:27-SEP-98 00:40:41 Proc:28-SEP-98 12:04:24
 Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
 Version: V3.6 31-JUL-1998 10:51:59  Sample text: 1mb091698m23 x1/1
```

```
   Amount: 0.24     of which 0.14     named and 0.10     unnamed
     Conc: 0.24     of which 0.14     named and 0.10     unnamed
   Tox #1: -             Tox #2: -            Tox #3: -

Name                  #      RT Respnse      RA     Conc     Area  Height     S/N  Mod?

1,2,3,4,6,7,8-HpCDF1  36:59 1.2e+05  1.78 n    0.07
                            1.2e+05                 7.9e+04 2.5e+04 5.6e+00 y  n
                                                    4.4e+04 1.8e+04 5.0e+00 y  n
                   2  37:13 2.6e+04  1.02 y    0.02
                            2.6e+04                 1.3e+04 4.3e+03 9.5e-01 n  n
                                                    1.3e+04 3.2e+03 9.0e-01 n  n
                   3  37:20 1.7e+04  1.50 n    0.01
                            1.7e+04                 1.0e+04 4.7e+03 1.0e+00 n  n
                                                    6.8e+03 2.3e+03 6.5e-01 n  n
                   4  37:26 1.4e+04  1.76 n    0.01
                            1.4e+04                 8.7e+03 3.6e+03 8.0e-01 n  n
                                                    5.0e+03 2.5e+03 6.9e-01 n  n
                   5  37:43 1.7e+04  1.62 n    0.01
                            1.7e+04                 1.1e+04 3.7e+03 8.2e-01 n  n
                                                    6.5e+03 2.3e+03 6.5e-01 n  n
                   6  37:55 1.1e+04  1.16 y    0.01
                            1.1e+04                 6.1e+03 2.6e+03 5.8e-01 n  n
                                                    5.2e+03 2.0e+03 5.7e-01 n  n
1,2,3,4,7,8,9-HpCDF7  38:11 8.4e+04  0.74 n    0.06
                            8.4e+04                 3.5e+04 1.5e+04 3.4e+00 y  n
                                                    4.8e+04 1.5e+04 4.1e+00 y  n
                   8  38:13 7.0e+04  0.45 n    0.05
                            7.0e+04                 2.1e+04 8.5e+03 1.9e+00 n  n
                                                    4.8e+04 1.5e+04 4.1e+00 y  n
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                                                                    Page 8 of 8

  Ent: 46 Name: Total Hepta-Dioxins F:4  Mass: 423.777 425.774 Mod? no    #Hom:3

  Run: 4  File: a26sep98m    S:11 Acq:27-SEP-98 00:40:41 Proc:28-SEP-98 12:04:24
  Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
  Version: V3.6 31-JUL-1998 10:51:59  Sample text: 1mb091698m23 x1/1

    Amount: 0.09    of which 0.05    named and 0.03    unnamed
      Conc: 0.09    of which 0.05    named and 0.03    unnamed
    Tox #1: -           Tox #2: -         Tox #3: -

 Name                 #     RT Respnse     RA     Conc     Area  Height     S/N  Mod?

 1,2,3,4,6,7,8-HpCDD1  37:48 8.4e+04  0.84 n    0.05
                             8.4e+04                  3.8e+04 1.3e+04 4.2e+00 y  n
                                                      4.5e+04 1.4e+04 6.4e+00 y  n
                      2  38:11 4.2e+04  4.07 n    0.03
                             4.2e+04                  3.3e+04 8.8e+03 2.9e+00 n  n
                                                      8.2e+03 3.1e+03 1.5e+00 n  n
                      3  38:24 1.3e+04  1.31 n    0.01
                             1.3e+04                  7.6e+03 4.0e+03 1.3e+00 n  n
                                                [illegible] 2.5e+03 1.2e+00 n  n
```

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$$\%Rec\ HpCDF^{SS} = \frac{1.56\ 10^8 \times 4\ ng \times 100}{4\ ng \times 1.20\ 10^8 \times 0.6169} = 210.7\%.$$

```
          Filename ; a26sep98m
            Sample ; 11
          Acquired ; 27-SEP-98  00:40:41
         Processed ; 28-SEP-98  12:04:24
         Sample ID ; lmb091698m23 x1/1
         Cal Table ; m8290-092698m
     Results Table ; M8290-092698M
          Comments ;
Typ   ;                  Name;     Resp;    Ion 1;    Ion 2;   RA;?;      RT;      Conc;       DL;    S/N1;?;    S/N2;? ; mod?
Unk   ;          2,3,7,8-TCDD;        *;        *;        *;    *;n;NotFnd;         *;   0.0316;       *;n;       *;n ;  no
Unk   ;       1,2,3,7,8-PeCDD; 1.31e+05; 8.16e+04; 4.99e+04; 1.64;y;  33:13;     0.042;   0.0167;       6;y;      13;y ;  no
Unk   ;     1,2,3,4,7,8-HxCDD; 9.56e+04; 4.80e+04; 4.76e+04; 1.01;n;  35:19;     0.053;   0.0386;       6;y;       5;y ;  no
Unk   ;     1,2,3,6,7,8-HxCDD; 1.35e+05; 6.94e+04; 6.54e+04; 1.06;y;  35:24;     0.046;   0.0237;       8;y;       6;y ;  no
Unk   ;     1,2,3,7,8,9-HxCDD; 1.20e+05; 5.90e+04; 6.06e+04; 0.97;n;  35:36;     0.050;   0.0294;       9;y;       5;y ;  no
Unk   ;   1,2,3,4,6,7,8-HpCDD; 8.38e+04; 3.84e+04; 4.55e+04; 0.84;n;  37:48;     0.053;   0.0433;       4;y;       6;y ;  no
Unk   ;                  OCDD; 8.61e+04; 1.80e+04; 6.80e+04; 0.26;n;  40:44;     0.096;   0.2360;       1;n;      10;y ;  no

Unk   ;          2,3,7,8-TCDF; 4.35e+04; 1.74e+04; 2.60e+04; 0.67;y;  28:27;     0.012;   0.0389;       2;n;       1;n ;  no
Unk   ;       1,2,3,7,8-PeCDF; 1.60e+05; 9.14e+04; 6.82e+04; 1.34;y;  32:35;     0.057;   0.0187;      21;y;       5;y ;  no
Unk   ;       2,3,4,7,8-PeCDF; 1.58e+05; 1.03e+05; 5.52e+04; 1.86;n;  33:01;     0.050;   0.0164;      26;y;       5;y ;  no
Unk   ;     1,2,3,4,7,8-HxCDF; 1.12e+05; 5.12e+04; 6.10e+04; 0.84;n;  34:48;     0.041;   0.0206;       6;y;       7;y ;  no
Unk   ;     1,2,3,6,7,8-HxCDF; 1.32e+05; 6.50e+04; 6.67e+04; 0.97;n;  34:52;     0.027;   0.0117;       5;y;       6;y ;  no
Unk   ;     2,3,4,6,7,8-HxCDF; 1.04e+05; 7.20e+04; 3.15e+04; 2.28;n;  35:14;     0.028;   0.0153;       5;y;       4;y ;  no
Unk   ;     1,2,3,7,8,9-HxCDF; 1.25e+05; 7.77e+04; 4.73e+04; 1.64;n;  35:45;     0.049;   0.0222;       5;y;       4;y ;  no
Unk   ;   1,2,3,4,6,7,8-HpCDF; 1.23e+05; 7.85e+04; 4.42e+04; 1.78;n;  36:59;     0.076;   0.0567;       6;y;       5;y ;  no
Unk   ;   1,2,3,4,7,8,9-HpCDF; 8.35e+04; 3.54e+04; 4.81e+04; 0.74;n;  38:11;     0.082;   0.0893;       3;y;       4;y ;  no
Unk   ;                  OCDF;        *;        *;        *;    *;n;NotFnd;         *;   0.0883;       *;n;       *;n ;  no

ES/RT;      13C-2,3,7,8-TCDD; 2.65e+08; 1.18e+08; 1.47e+08; 0.80;y;  29:25;    90.049;   0.1019;    1411;y;    4061;y ;  no
ES    ;   13C-1,2,3,7,8-PeCDD; 2.22e+08; 1.36e+08; 8.59e+07; 1.59;y;  33:13;   128.189;   0.0961;    4769;y;   13050;y ;  no
ES    ; 13C-1,2,3,6,7,8-HxCDD; 2.90e+08; 1.63e+08; 1.27e+08; 1.28;y;  35:22;    92.355;   0.0618;    6456;y;    4314;y ;  no
ES    ;13C-1,2,3,4,6,7,8-HpCDD; 1.63e+08; 8.37e+07; 7.96e+07; 1.05;y;  37:48;   118.010;   0.2486;     932;y;    2261;y ;  no
ES    ;             13C-OCDD; 1.63e+08; 7.85e+07; 8.49e+07; 0.92;y;  40:44;   273.553;   0.1313;    4479;y;    4865;y ;  no

ES/RT;      13C-2,3,7,8-TCDF; 3.40e+08; 1.51e+08; 1.89e+08; 0.80;y;  28:23;    78.797;   0.0629;    3693;y;    3114;y ;  no
ES    ;   13C-1,2,3,7,8-PeCDF; 2.87e+08; 1.77e+08; 1.11e+08; 1.60;y;  32:34;    78.511;   0.0157;   36158;y;   19315;y ;  no
ES    ; 13C-1,2,3,6,7,8-HxCDF; 2.29e+08; 7.87e+07; 1.50e+08; 0.52;y;  34:47;    84.112;   1.1145;     408;y;     384;y ;  no
ES    ;13C-1,2,3,4,6,7,8-HpCDF; 1.20e+08; 3.64e+07; 8.36e+07; 0.44;y;  36:59;    60.490;   0.2987;     317;y;    1208;y ;  no

JS    ;      13C-1,2,3,4-TCDD; 2.95e+08; 1.31e+08; 1.64e+08; 0.80;y;  29:08;   129.122;        -;    1872;y;    5468;y ;  no
JS    ; 13C-1,2,3,7,8,9-HxCDD; 2.64e+08; 1.44e+08; 1.20e+08; 1.21;y;  35:36;   191.632;        -;    5089;y;    3275;y ;  no

CS    ;     37Cl-2,3,7,8-TCDD; 2.94e+08; 2.94e+08;        -;    -;-;NotFnd;   103.251;   0.0594;    4361;y;       -; -;  no
CS    ;   13C-2,3,4,7,8-PeCDF; 4.40e+08; 2.70e+08; 1.70e+08; 1.58;y;  33:01;   110.247;   0.0144;   57554;y;   30852;y ;  no
CS    ; 13C-1,2,3,4,7,8-HxCDD; 1.59e+08; 8.87e+07; 7.08e+07; 1.25;y;  35:19;    92.648;   0.1127;    5155;y;    3356;y ;  no
CS    ; 13C-1,2,3,4,7,8-HxCDF; 2.85e+08; 9.83e+07; 1.86e+08; 0.53;y;  34:52;    58.468;   0.6226;     325;y;     301;y ;  no
CS    ;13C-1,2,3,4,7,8,9-HpCDF; 1.56e+08; 4.62e+07; 1.09e+08; 0.42;y;  38:11;   127.336;   0.4853;     369;y;    1361;y ;  no

SS    ;     37Cl-2,3,7,8-TCDD; 2.94e+08; 2.94e+08;        -;    -;-;NotFnd;   114.681;   0.0793;    4361;y;       -; -;  no
SS    ;   13C-2,3,4,7,8-PeCDF; 4.40e+08; 2.70e+08; 1.70e+08; 1.58;y;  33:01;   140.314;   0.0102;   57554;y;   30852;y ;  no
SS    ; 13C-1,2,3,4,7,8-HxCDD; 1.59e+08; 8.87e+07; 7.08e+07; 1.25;y;  35:19;   100.651;   0.1037;    5155;y;    3356;y ;  no
SS    ; 13C-1,2,3,4,7,8-HxCDF; 2.85e+08; 9.83e+07; 1.86e+08; 0.53;y;  34:52;    69.108;   0.4261;     325;y;     301;y ;  no
SS    ;13C-1,2,3,4,7,8,9-HpCDF; 1.56e+08; 4.62e+07; 1.09e+08; 0.42;y;  38:11;   210.068;   0.7460;     369;y;    1361;y ;  no
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 Ent: 39 Name: Total Tetra-Furans  F:1  Mass: 303.902 305.899 Mod? no    #Hom:4

 Run: 9  File: a26sep98m     S:11 Acq:27-SEP-98 00:40:41 Proc:28-SEP-98 12:04:24
 Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
 Version: V3.6 31-JUL-1998 10:51:59  Sample text: lmb091698m23 x1/1

   Amount: 0.02    of which 0.01    named and 0.01    unnamed
     Conc: 0.02    of which 0.01    named and 0.01    unnamed
   Tox #1: -          Tox #2: -         Tox #3: -

Name                   #    RT Respnse     RA     Conc     Area  Height     S/N  Mod?

                       1  27:00 8.5e+03  0.44 n    0.00
                                8.5e+03                2.6e+03 1.6e+03 4.8e-01 n  n
                                                       5.9e+03 3.5e+03 5.7e-01 n  n
2,3,7,8-TCDF           2  28:27 4.3e+04  0.67 y    0.01
                                4.3e+04                1.7e+04 6.0e+03 1.8e+00 n  n
                                                       2.6e+04 8.6e+03 1.4e+00 n  n
                       3  28:37 1.2e+04  0.59 n    0.00
                                1.2e+04                4.3e+03 2.0e+03 6.0e-01 n  n
                                                       [illegible].4e+03 3.2e+03 5.2e-01 n  n
                       4  28:54 1.5e+04  1.22 n    0.00
                                1.5e+04                8.4e+03 3.7e+03 1.1e+00 n  n
                                                       6.9e+03 5.5e+03 9.0e-01 n  n


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 Ent: 40 Name: Total Tetra-Dioxins F:1  Mass: 319.897 321.894 Mod? no    #Hom:4

 Run: 9  File: a26sep98m     S:11 Acq:27-SEP-98 00:40:41 Proc:28-SEP-98 12:04:24
 Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
 Version: V3.6 31-JUL-1998 10:51:59  Sample text: lmb091698m23 x1/1

   Amount: 0.12    of which *       named and 0.12    unnamed
     Conc: 0.12    of which *       named and 0.12    unnamed
   Tox #1: -          Tox #2: -         Tox #3: -

Name                   #    RT Respnse     RA     Conc     Area  Height     S/N  Mod?

                       1  28:07 1.1e+04  1.27 n    0.00
                                1.1e+04                6.3e+03 4.8e+03 1.3e+00 n  n
                                                       4.9e+03 1.9e+03 9.4e-01 n  n
                       2  28:23 7.5e+04  3.16 n    0.03
                                7.5e+04                5.7e+04 1.2e+04 3.3e+00 y  n
                                                       1.8e+04 4.7e+03 2.3e+00 n  n
                       3  29:25 2.4e+05  0.08 n    0.08
                                2.4e+05                1.8e+04 8.1e+03 2.2e+00 n  n
                                                       2.2e+05 4.1e+04 2.1e+01 y  n
                       4  29:38 1.6e+04  0.37 n    0.01
                                1.6e+04                4.2e+03 2.6e+03 7.0e-01 n  n
                                                       1.1e+04 3.5e+03 1.8e+00 n  n


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 Ent: 41 Name: Total Penta-Furans  F:2  Mass: 339.860 341.857 Mod? no    #Hom:5

 Run: 9  File: a26sep98m     S:11 Acq:27-SEP-98 00:40:41 Proc:28-SEP-98 12:04:24
 Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
 Version: V3.6 31-JUL-1998 10:51:59  Sample text: lmb091698m23 x1/1

   Amount: 0.12    of which 0.11    named and 0.01    unnamed
     Conc: 0.12    of which 0.11    named and 0.01    unnamed
   Tox #1: -          Tox #2: -         Tox #3: -

Name                   #    RT Respnse     RA     Conc     Area  Height     S/N  Mod?

1,2,3,7,8-PeCDF        1  32:35 1.6e+05  1.34 y    0.06
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```
                               1.6e+05                    9.1e+04 4.0e+04 2.1e+01 y  n
                                                          6.8e+04 2.7e+04 5.5e+00 y  n
                       2  32:40 1.6e+04  0.61 n     0.01
                               1.6e+04                    5.9e+03 3.1e+03 1.6e+00 n  n
                                                          9.7e+03 3.6e+03 7.2e-01 n  n
2,3,4,7,8-PeCDF        3  33:01 1.6e+05  1.86 n     0.05
                               1.6e+05                    1.0e+05 5.0e+04 2.6e+01 y  n
                                                          5.5e+04 2.3e+04 4.6e+00 y  n
                       4  33:29 8.1e+03  0.63 n     0.00
                               8.1e+03                    3.1e+03 1.6e+03 8.4e-01 n  n
                                                          5.0e+03 2.0e+03 4.0e-01 n  n
                       5  33:33 1.1e+04  1.30 n     0.00
                               1.1e+04                    6.3e+03 2.6e+03 1.3e+00 n  n
                                                          4.9e+03 3.0e+03 6.0e-01 n  n


                                                                         Page 4 of 8

 Ent: 42 Name: Total Penta-Dioxins F:2  Mass: 355.855 357.852 Mod? no   #Hom:4

 Run: 9  File: a26sep98m    S:11 Acq:27-SEP-98 00:40:41 Proc:28-SEP-98 12:04:24
 Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
 Version: V3.6 31-JUL-1998 10:51:59  Sample text: lmb091698m23 x1/1

   Amount: 0.08    of which 0.04    named and 0.04    unnamed
     Conc: 0.08    of which 0.04    named and 0.04    unnamed
   Tox #1: -            Tox #2: -        Tox #3: -

Name                   #     RT Respnse    RA    Conc    Area  Height     S/N  Mod?

                       1  32:34 3.1e+04  2.55 n     0.01
                               3.1e+04                    2.3e+04 9.5e+03 2.1e+00 n  n
                                                          8.9e+03 3.8e+03 2.7e+00 n  n
                       2  33:01 7.0e+04  3.11 n     0.02
                               7.0e+04                    5.3e+04 2.3e+04 5.1e+00 y  n
                                                          1.7e+04 5.2e+03 3.7e+00 y  n
                       3  33:05 1.2e+04  1.82 n     0.00
                               1.2e+04                    7.9e+03 5.2e+03 1.1e+00 n  n
                                                          4.3e+03 1.3e+03 9.6e-01 n  n
1,2,3,7,8-PeCDD        4  33:13 1.3e+05  1.64 y     0.04
                               1.3e+05                    8.2e+04 2.9e+04 6.5e+00 y  n
                                                          5.0e+04 1.8e+04 1.3e+01 y  n
```

```
 Ent: 43 Name: Total Hexa-Furans    F:3  Mass: 373.821 375.818 Mod? no    #Hom:17

 Run: 9  File: a26sep98m     S:11 Acq:27-SEP-98 00:40:41 Proc:28-SEP-98 12:04:24
 Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
 Version: V3.6 31-JUL-1998 10:51:59  Sample text: 1mb091698m23 x1/1

   Amount: 0.20    of which 0.15    named and 0.05    unnamed
     Conc: 0.20    of which 0.15    named and 0.05    unnamed
   Tox #1: -             Tox #2: -          Tox #3: -

Name                #     RT Respnse    RA     Conc    Area  Height     S/N  Mod?

1,2,3,4,7,8-HxCDF   1  34:48 1.1e+05  0.84 n   0.04
                                 1.1e+05                5.1e+04 2.7e+04 5.9e+00 y  n
                                                        6.1e+04 2.3e+04 6.6e+00 y  n
1,2,3,6,7,8-HxCDF   2  34:52 1.3e+05  0.97 n   0.03
                                 1.3e+05                6.5e+04 2.3e+04 5.1e+00 y  n
                                                        6.7e+04 2.2e+04 6.2e+00 y  n
                    3  35:00 1.1e+04  0.63 n   0.00
                                 1.1e+04                4.2e+03 2.2e+03 4.8e-01 n  n
                                                        [illegible] 2.8e+03 7.8e-01 n  n
                    4  35:03 1.3e+04  1.65 n   0.00
                                 1.3e+04                8.3e+03 5.7e+03 1.3e+00 n  n
                                                        5.1e+03 3.8e+03 1.1e+00 n  n
                    5  35:07 1.1e+04  0.92 n   0.00
                                 1.1e+04                5.5e+03 3.2e+03 7.1e-01 n  n
                                                        5.9e+03 3.1e+03 8.6e-01 n  n
2,3,4,6,7,8-HxCDF   6  35:14 1.0e+05  2.28 n   0.03
                                 1.0e+05                7.2e+04 2.1e+04 4.7e+00 y  n
                                                        3.2e+04 1.4e+04 3.8e+00 y  n
                    7  35:23 2.0e+04  4.59 n   0.01
                                 2.0e+04                1.7e+04 1.2e+04 2.6e+00 n  n
                                                        3.6e+03 [illegible]
                    8  35:25 1.9e+04  4.15 n   0.01
                                 1.9e+04                1.5e+04 5.9e+03 1.3e+00 n  n
                                                        3.6e+03 1.3e+03 3.6e-01 n  n
                    9  35:33 1.1e+04  1.91 n   0.00
                                 1.1e+04                7.0e+03 3.8e+03 8.3e-01 n  n
                                                        3.7e+03 1.5e+03 4.2e-01 n  n
                   10  35:35 2.1e+04  3.35 n   0.01
                                 2.1e+04                1.6e+04 7.7e+03 1.7e+00 n  n
                                                        4.8e+03 1.5e+03 4.3e-01 n  n
 1,2,3,7,8,9-HxCDF 11  35:45 1.3e+05  1.64 n   0.05
                                 1.3e+05                7.8e+04 2.4e+04 5.4e+00 y  n
                                                        4.7e+04 1.4e+04 4.1e+00 y  n
                   12  35:53 2.0e+04  1.35 y   0.01
                                 2.0e+04                1.2e+04 4.2e+03 9.2e-01 n  n
                                                        8.7e+03 6.8e+03 1.9e+00 n  n
                   13  36:00 1.5e+04  0.55 n   0.00
                                 1.5e+04                5.3e+03 3.8e+03 8.4e-01 n  n
                                                        9.6e+03 2.9e+03 8.3e-01 n  n
                   14  36:04 1.3e+04  2.63 n   0.00
                                 1.3e+04                9.2e+03 6.2e+03 1.4e+00 n  n
                                                        3.5e+03 2.2e+03 6.3e-01 n  n
                   15  36:06 9.2e+03  1.69 n   0 00
                                 9.2e+03                5.8e+03 2.9e [illegible]
                                                        3.4e+03 1.7e+03 4.7e-01 n  n
                   16  36:10 9.2e+03  1.24 y   0 00
                                 9.2e+03                5.1e+03 2.1e+03 4.6e-01 n  n
                                                        4.1e+03 2.1e+03 6.0e-01 n  n
                   17  36:16 1.2e+04  0.64 n   0.00
                                 1.2e+04                4.8e+03 2.7e+03 6.0e-01 n  n
                                                        7.6e+03 2.8e+03 7.9e-01 n  n
```

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                                                                    Page 6 of 8

  Ent: 44 Name: Total Hexa-Dioxins  F:3  Mass: 389.816 391.813 Mod? no   #Hom:16

  Run: 9  File: a26sep98m    S:11 Acq:27-SEP-98 00:40:41 Proc:28-SEP-98 12:04:24
  Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
  Version: V3.6 31-JUL-1998 10:51:59  Sample text: lmb091698m23 x1/1

     Amount: 0.28    of which 0.15    named and 0.13    unnamed
       Conc: 0.28    of which 0.15    named and 0.13    unnamed
     Tox #1: -             Tox #2: -        Tox #3: -

Name                 #    RT Respnse    RA    Conc    Area  Height    S/N  Mod?

                     1  34:47 6.8e+04  3.96 n   0.03
                              6.8e+04                5.5e+04 2.2e+04 7.8e+00 y  n
                                                     1.4e+04 7.8e+03 1.9e+00 n  n
                     2  34:52 7.6e+04  3.03 n   0.03
                              7.6e+04                5.7e+04 1.9e+04 6.8e+00 y  n
                                                     1.9e+04 7.1e+03 1.8e+00 n  n
                     3  34:56 3.6e+04  1.35 y   0.01
                              3.6e+04                2.0e+04 5.9e+03 2.1e+00 n  n
                                                     1.5e+04 6.6e+03 1.6e+00 n  n
                     4  35:08 1.0e+04  1.05 n   0.00
                              1.0e+04                5.3e+03 2.4e+03 8.7e-01 n  n
                                                     5.1e+03 2.6e+03 6.6e-01 n  n
                     5  35:11 8.1e+03  0.51 n   0.00
                              8.1e+03                2.7e+03 1.2e+03 4.2e-01 n  n
                                                     5.4e+03 2.4e+03 5.9e-01 n  n
                     6  35:14 1.3e+04  1.04 n   0.01
                              1.3e+04                6.4e+03 2.4e+03 8.5e-01 n  n
                                                     6.2e+03 2.5e+03 6.3e-01 n  n
1,2,3,4,7,8-HxCDD    7  35:19 9.6e+04  1.01 n   0.05
                              9.6e+04                4.8e+04 1.6e+04 5.8e+00 y  n
                                                     4.8e+04 2.0e+04 4.9e+00 y  n
1,2,3,6,7,8-HxCDD    8  35:24 1.3e+05  1.06 y   0.05
                              1.3e+05                6.9e+04 2.2e+04 7.8e+00 y  n
                                                     6.5e+04 2.4e+04 6.0e+00 y  n
1,2,3,7,8,9-HxCDD    9  35:36 1.2e+05  0.97 n   0.05
                              1.2e+05                5.9e+04 2.6e+04 9.3e+00 y  n
                                                     6.1e+04 2.0e+04 5.1e+00 y  n
                     10 35:42 2.3e+04  0.41 n   0.01
                              2.3e+04                6.7e+03 4.1e+03 1.5e+00 n  n
                                                     1.6e+04 5.3e+03 1.3e+00 n  n
                     11 35:49 1.8e+04  0.63 n   0.01
                              1.8e+04                7.0e+03 3.6e+03 1.3e+00 n  n
                                                     1.1e+04 5.3e+03 1.3e+00 n  n
                     12 35:56 1.6e+04  0.59 n   0.01
                              1.6e+04                5.8e+03 2.9e+03 1.0e+00 n  n
                                                     9.9e+03 3.8e+03 9.4e-01 n  n
                     13 36:01 9.0e+03  1.41 y   0.00
                              9.0e+03                5.2e+03 2.4e+03 8.5e-01 n  n
                                                     3.7e+03 3.0e+03 7.4e-01 n  n
                     14 36:05 7.8e+03  0.40 n   0.00
                              7.8e+03                2.2e+03 1.4e+03 4.9e-01 n  n
                                                     5.6e+03 2.2e+03 5.5e-01 n  n
                     15 36:13 9.2e+03  0.50 n   0.00
                              9.2e+03                3.1e+03 1.8e+03 6.6e-01 n  n
                                                     6.1e+03 2.7e+03 6.7e-01 n  n
                     16 36:21 8.4e+03  1.38 y   0.00
                              8.4e+03                4.9e+03 1.4e+03 4.9e-01 n  n
                                                     3.5e+03 2.3e+03 5.8e-01 n  n


                                                                    Page 7 of 8

  Ent: 45 Name: Total Hepta-Furans  F:4  Mass: 407.782 409.779 Mod? no   #Hom:8

  Run: 9  File: a26sep98m    S:11 Acq:27-SEP-98 00:40:41 Proc:28-SEP-98 12:04:24
  Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
  Version: V3.6 31-JUL-1998 10:51:59  Sample text: lmb091698m23 x1/1
```

```
OPUSquan    28-SEP-1998                Page 5

   Amount: 0.28    of which 0.16    named and 0.12    unnamed
     Conc: 0.28    of which 0.16    named and 0.12    unnamed
   Tox #1: -           Tox #2: -        Tox #3: -

Name                    #     RT Respnse     RA     Conc     Area  Height      S/N  Mod?

1,2,3,4,6,7,8-HpCDF1  36:59 1.2e+05  1.78 n    0.08
                            1.2e+05                  7.9e+04 2.5e+04 5.6e+00 y  n
                                                     4.4e+04 1.8e+04 5.0e+00 y  n
                        2  37:13 2.6e+04  1.02 y    0.02
                            2.6e+04                  1.3e+04 4.3e+03 [illegible] n  n
                                                     1.3e+04 3.2e+03 9.0e-01 n  n
                        3  37:20 1.7e+04  1.50 n    0.01
                            1.7e+04                  1.0e+04 4.7e+03 1.0e+00 n  n
                                                     6.8e+03 2.3e+03 6.5e-01 n  n
                        4  37:26 1.4e+04  1.76 n    0.01
                            1.4e+04                  8.7e+03 3.6e+03 8.0e-01 n  n
                                                     5.0e+03 2.5e+03 6.9e-01 n  n
                        5  37:43 1.7e+04  1.62 n    0.01
                            1.7e+04                  1.1e+04 3.7e+03 8.2e-01 n  n
                                                     6.5e+03 2.3e+03 6.5e-01 n  n
                        6  37:55 1.1e+04  1.16 y    0.01
                            1.1e+04                  6.1e+03 2.6e+03 5.8e-01 n  n
                                                     5.2e+03 2.0e+03 5.7e-01 n  n
1,2,3,4,7,8,9-HpCDF7  38:11 8.4e+04  0.74 n    0.08
                            8.4e+04                  3.5e+04 1.5e+04 3.4e+00 y  n
                                                     4.8e+04 1.5e+04 4.1e+00 y  n
                        8  38:13 7.0e+04  0.45 n    0.05
                            7.0e+04                  2.1e+04 8.5e+03 1.9e+00 n  n
                                                     4.8e+04 1.5e+04 4.1e+00 y  n
```

```
                                                                Page 8 of 8

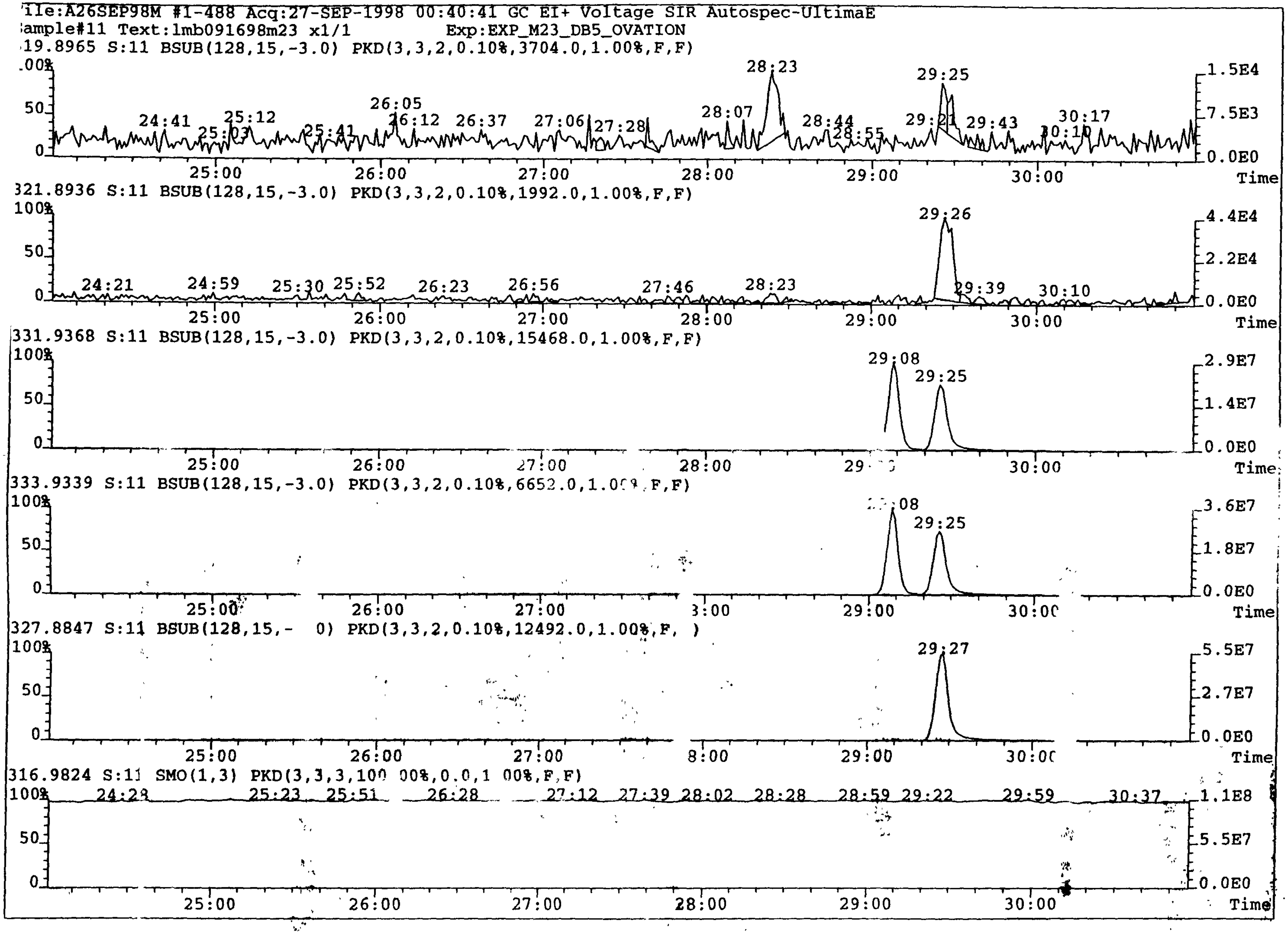
 Ent: 46 Name: Total Hepta-Dioxins F:4  Mass: 423.777 425.774 Mod? no    #Hom:3

 Run: 9  File: a26sep98m    S:11 Acq:27-SEP-98 00:40:41 Proc:28-SEP-98 12:04:24
 Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
 Version: V3.6 31-JUL-1998 10:51:59  Sample text: lmb091698m23 x1/1

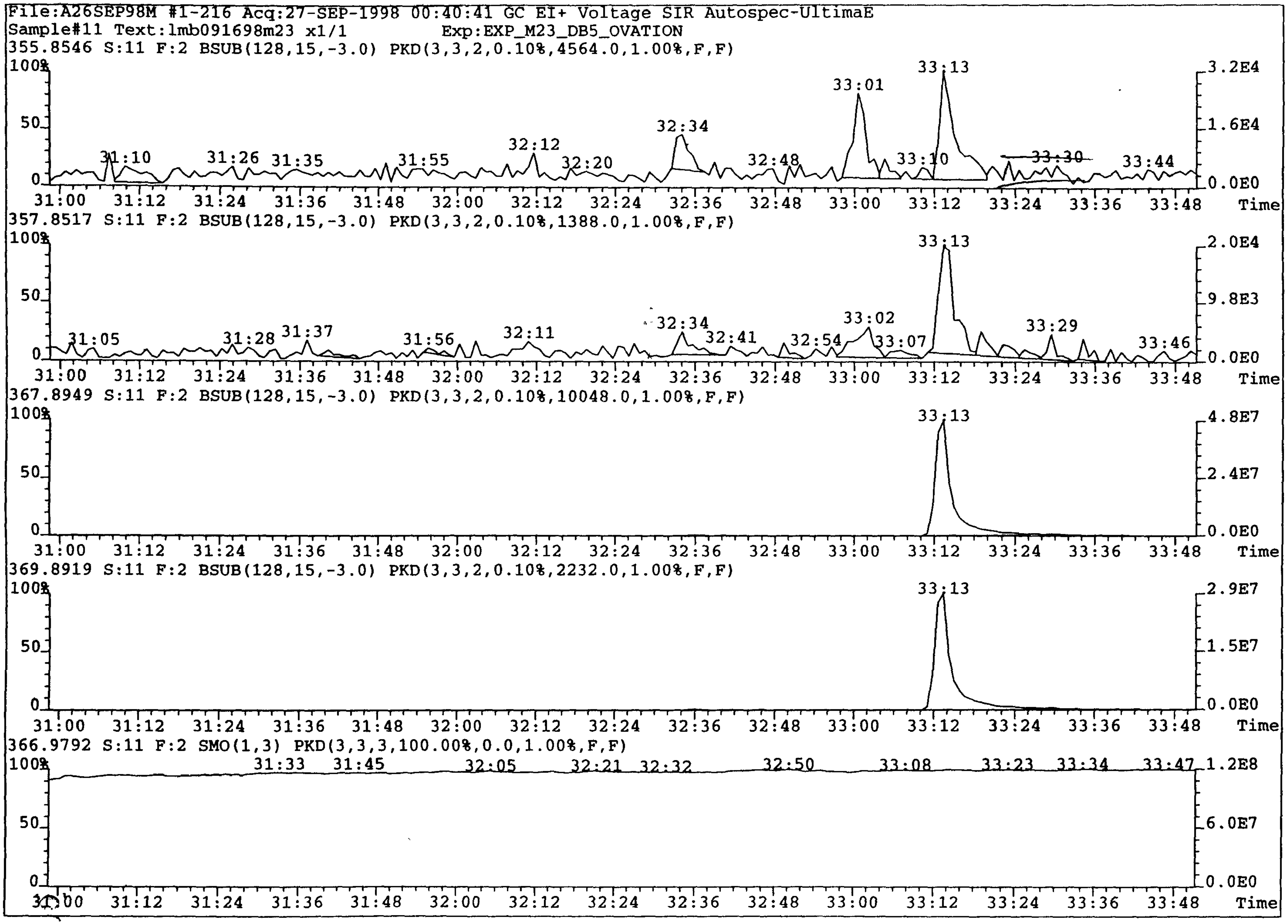
   Amount: 0.09     of which 0.05     named and 0.03     unnamed
     Conc: 0.09     of which 0.05     named and 0.03     unnamed
   Tox #1: -             Tox #2: -         Tox #3: -

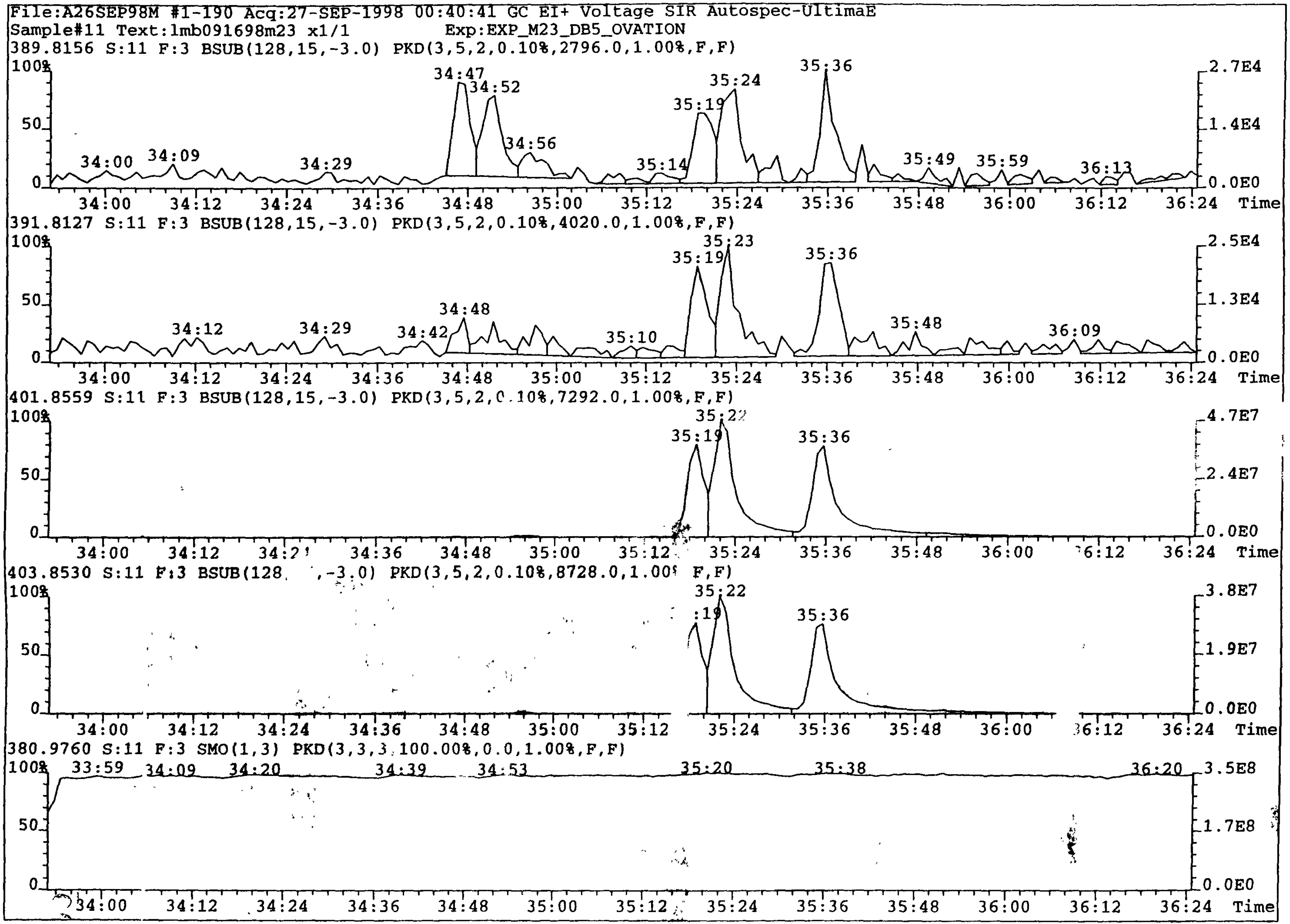
Name                   #     RT Respnse     RA    Conc     Area  Height     S/N  Mod?

1,2,3,4,6,7,8-HpCDD1  37:48 8.4e+04  0.84 n    0.05
                            8.4e+04                    3.8e+04 1.3e+04 4.2e+00 y  n
                                                       4.5e+04 1.4e+04 6.4e+00 y  n
                   2  38:11 4.2e+04  4.07 n    0.03
                            4.2e+04                    3.3e+04 8.8e+03 2.9e+00 n  n
                                                       8.2e+03 3.1e+03 1.5e+00 n  n
                   3  38:24 1.3e+04  1.31 n    0.01
                            1.3e+04                    7.6e+03 4.0e+03 1.3e+00 n  n
                                                       5.8e+03 2.5e+03 1.2e+00 n  n
```

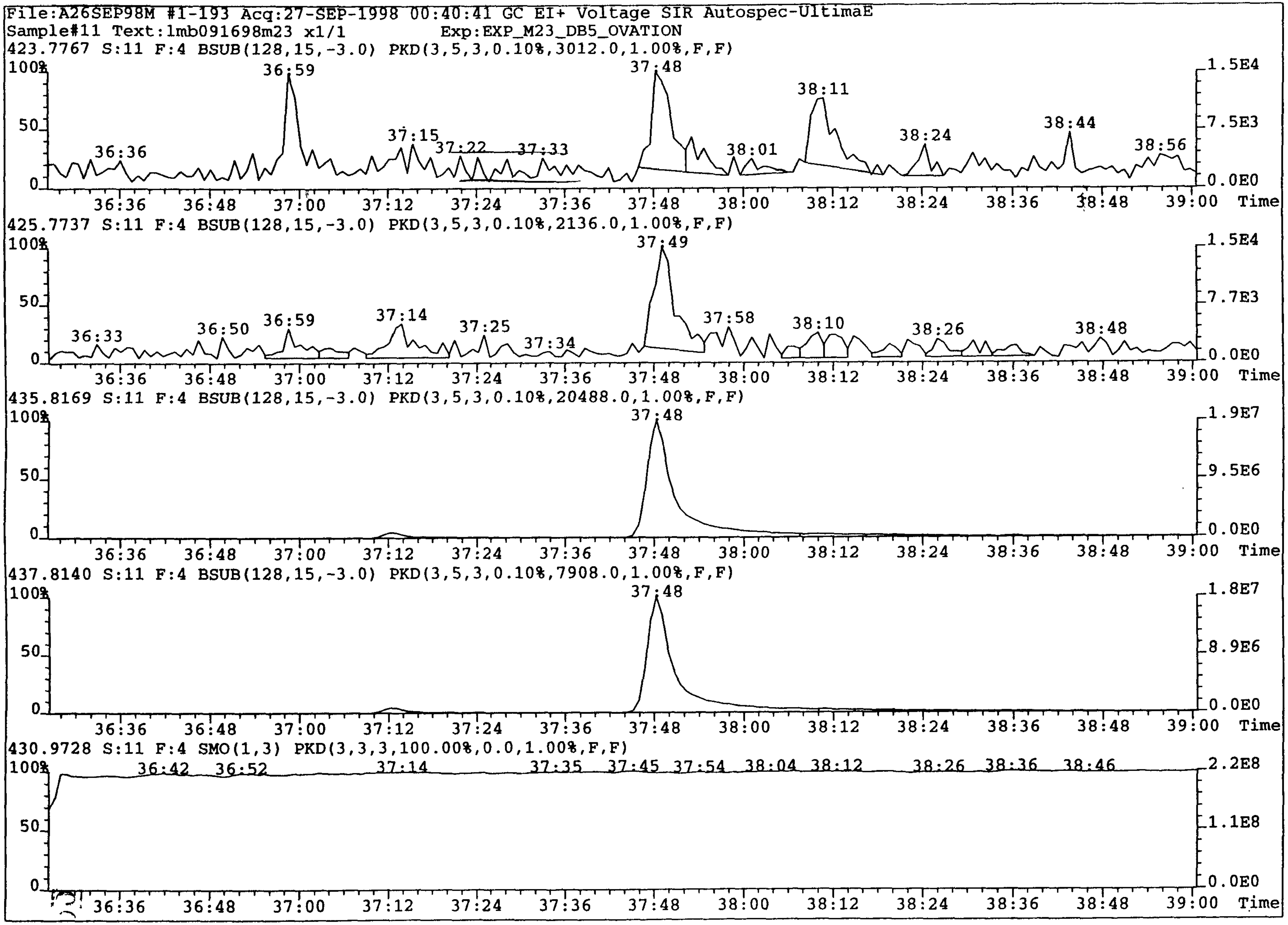


```
ile:A26SEP98M #1-488 Acq:27-SEP-1998 00:40:41 GC EI+ Voltage SIR Autospec-UltimaE
ample#11 Text:1mb091698m23 x1/1        Exp:EXP_M23_DB5_OVATION
19.8965 S:11 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,3704.0,1.00%,F,F)
321.8936 S:11 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,1992.0,1.00%,F,F)
331.9368 S:11 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,15468.0,1.00%,F,F)
333.9339 S:11 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,6652.0,1.00%,F,F)
327.8847 S:11 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,12492.0,1.00%,F,F)
316.9824 S:11 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)
```


File:A26SEP98M #1-216 Acq:27-SEP-1998 00:40:41 GC EI+ Voltage SIR Autospec-UltimaE
Sample#11 Text:lmb091698m23 x1/1 Exp:EXP_M23_DB5_OVATION
355.8546 S:11 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,4564.0,1.00%,F,F)
100%
50
0
31:10
31:26
31:35
31:55
32:12
32:20
32:34
32:48
33:01
33:10
33:13
~~33:30~~
33:44
3.2E4
1.6E4
0.0E0
31:00 31:12 31:24 31:36 31:48 32:00 32:12 32:24 32:36 32:48 33:00 33:12 33:24 33:36 33:48
Time
357.8517 S:11 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,1388.0,1.00%,F,F)
100%
50
0
31:05
31:28
31:37
31:56
32:11
32:34
32:41
32:54
33:02
33:07
33:13
33:29
33:46
2.0E4
9.8E3
0.0E0
31:00 31:12 31:24 31:36 31:48 32:00 32:12 32:24 32:36 32:48 33:00 33:12 33:24 33:36 33:48
Time
367.8949 S:11 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,10048.0,1.00%,F,F)
100%
50
0
33:13
4.8E7
2.4E7
0.0E0
31:00 31:12 31:24 31:36 31:48 32:00 32:12 32:24 32:36 32:48 33:00 33:12 33:24 33:36 33:48
Time
369.8919 S:11 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,2232.0,1.00%,F,F)
100%
50
0
33:13
2.9E7
1.5E7
0.0E0
31:00 31:12 31:24 31:36 31:48 32:00 32:12 32:24 32:36 32:48 33:00 33:12 33:24 33:36 33:48
Time
366.9792 S:11 F:2 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)
100%
50
0
31:33
31:45
32:05
32:21
32:32
32:50
33:08
33:23
33:34
33:47
1.2E8
6.0E7
0.0E0
31:00 31:12 31:24 31:36 31:48 32:00 32:12 32:24 32:36 32:48 33:00 33:12 33:24 33:36 33:48
Time

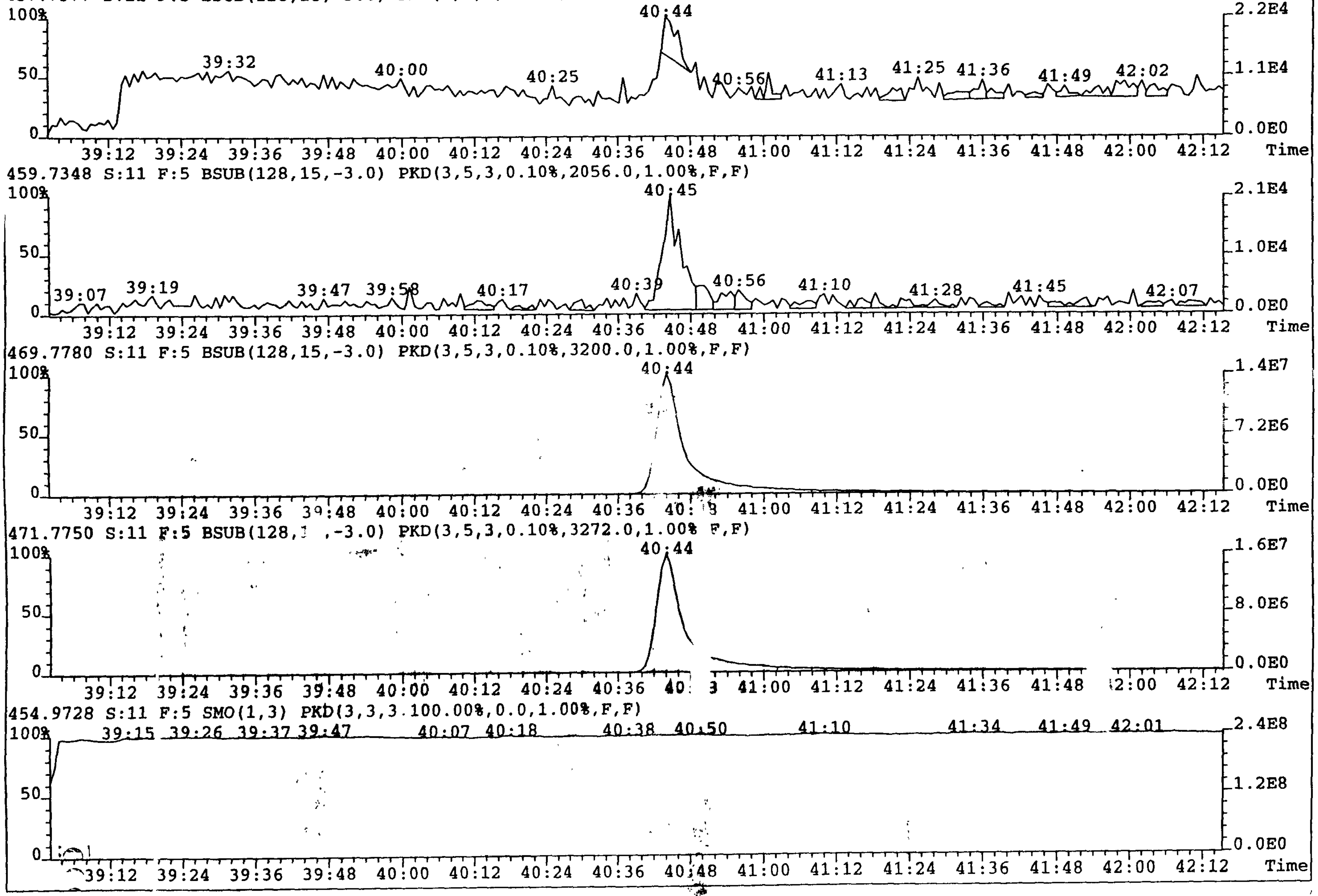


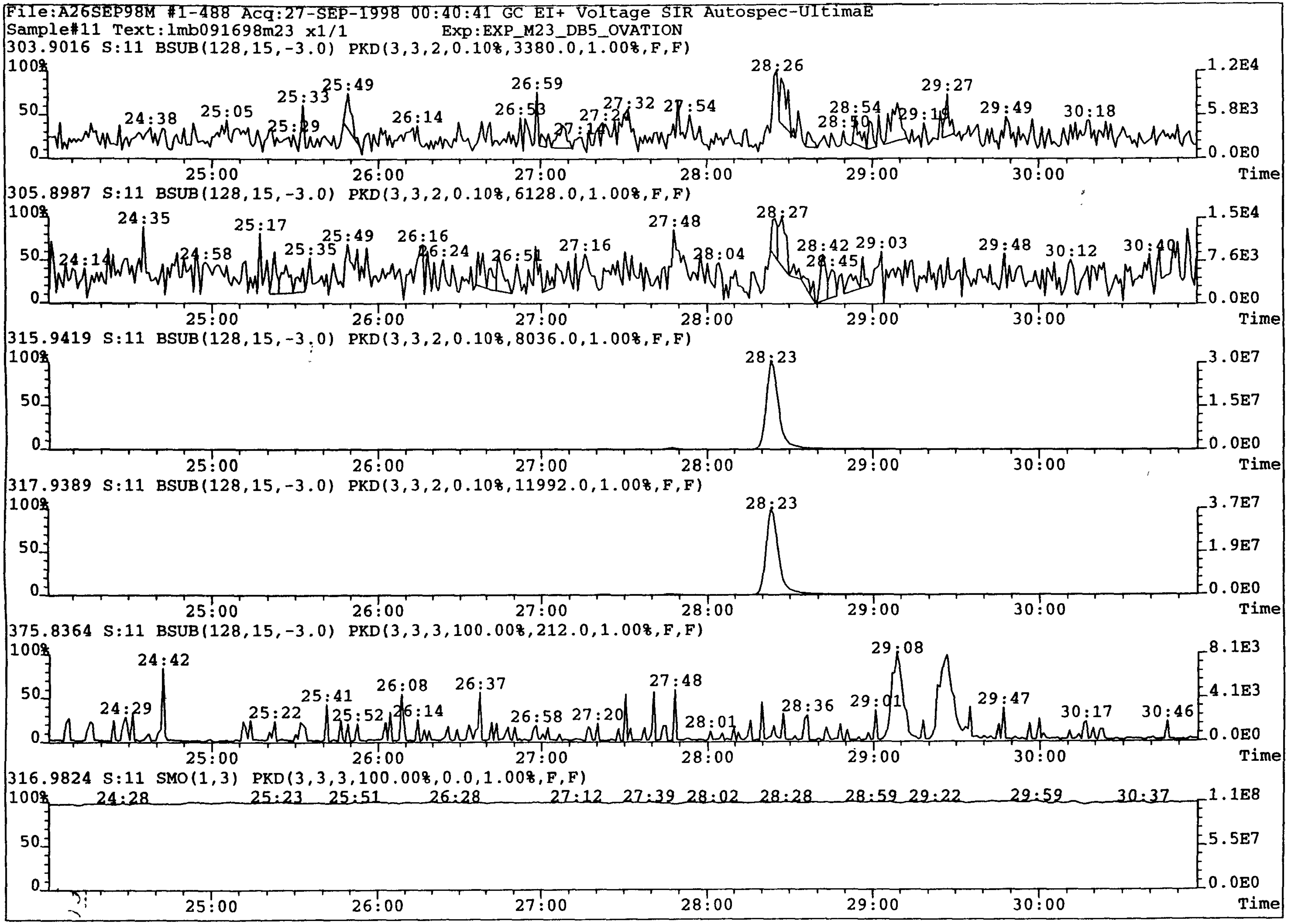
```
File:A26SEP98M #1-190 Acq:27-SEP-1998 00:40:41 GC EI+ Voltage SIR Autospec-UltimaE
Sample#11 Text:1mb091698m23 x1/1        Exp:EXP_M23_DB5_OVATION
389.8156 S:11 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,2796.0,1.00%,F,F)
391.8127 S:11 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,4020.0,1.00%,F,F)
401.8559 S:11 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,7292.0,1.00%,F,F)
403.8530 S:11 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,8728.0,1.00%,F,F)
380.9760 S:11 F:3 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)
```



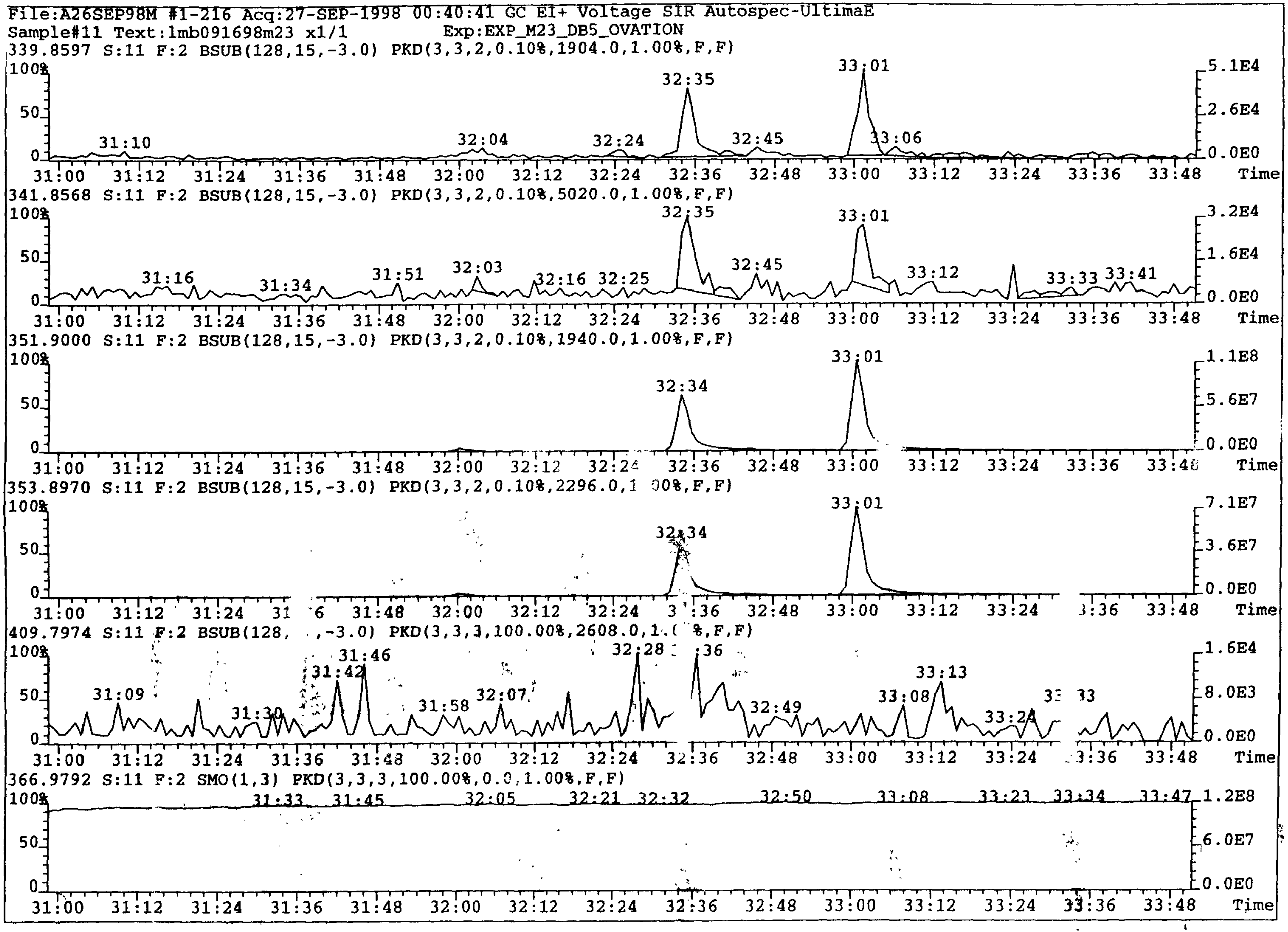
```
File:A26SEP98M #1-193 Acq:27-SEP-1998 00:40:41 GC EI+ Voltage SIR Autospec-UltimaE
Sample#11 Text:lmb091698m23 x1/1          Exp:EXP_M23_DB5_OVATION
423.7767 S:11 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,3012.0,1.00%,F,F)
425.7737 S:11 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,2136.0,1.00%,F,F)
435.8169 S:11 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,20488.0,1.00%,F,F)
437.8140 S:11 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,7908.0,1.00%,F,F)
430.9728 S:11 F:4 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)
```

```
ile:A26SEP98M #1-276 Acq:27-SEP-1998 00:40:41 GC EI+ Voltage SIR Autospec-UltimaE
ample#11 Text:lmb091698m23 x1/1          Exp:EXP_M23_DB5_OVATION
57.7377 S:11 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,10932.0,1.00%,F,F)
```





```
File:A26SEP98M #1-488 Acq:27-SEP-1998 00:40:41 GC EI+ Voltage SIR Autospec-UltimaE
Sample#11 Text:lmb091698m23 x1/1        Exp:EXP_M23_DB5_OVATION
303.9016 S:11 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,3380.0,1.00%,F,F)
305.8987 S:11 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,6128.0,1.00%,F,F)
315.9419 S:11 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,8036.0,1.00%,F,F)
317.9389 S:11 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,11992.0,1.00%,F,F)
375.8364 S:11 BSUB(128,15,-3.0) PKD(3,3,3,100.00%,212.0,1.00%,F,F)
316.9824 S:11 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)
```



File:A26SEP98M #1-216 Acq:27-SEP-1998 00:40:41 GC EI+ Voltage SIR Autospec-UltimaE
Sample#11 Text:lmb091698m23 x1/1 Exp:EXP_M23_DB5_OVATION

339.8597 S:11 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,1904.0,1.00%,F,F)

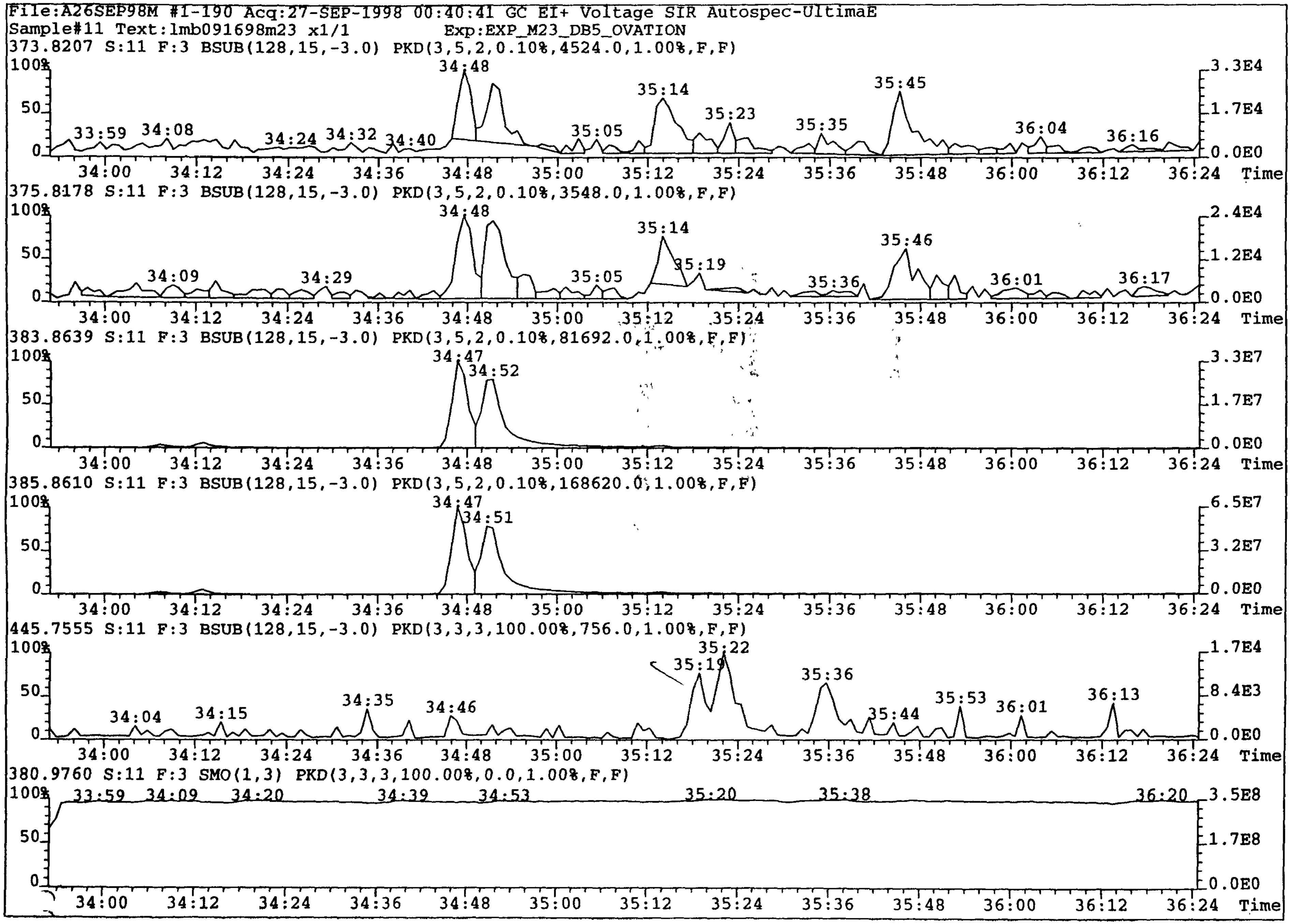
341.8568 S:11 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,5020.0,1.00%,F,F)

351.9000 S:11 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,1940.0,1.00%,F,F)

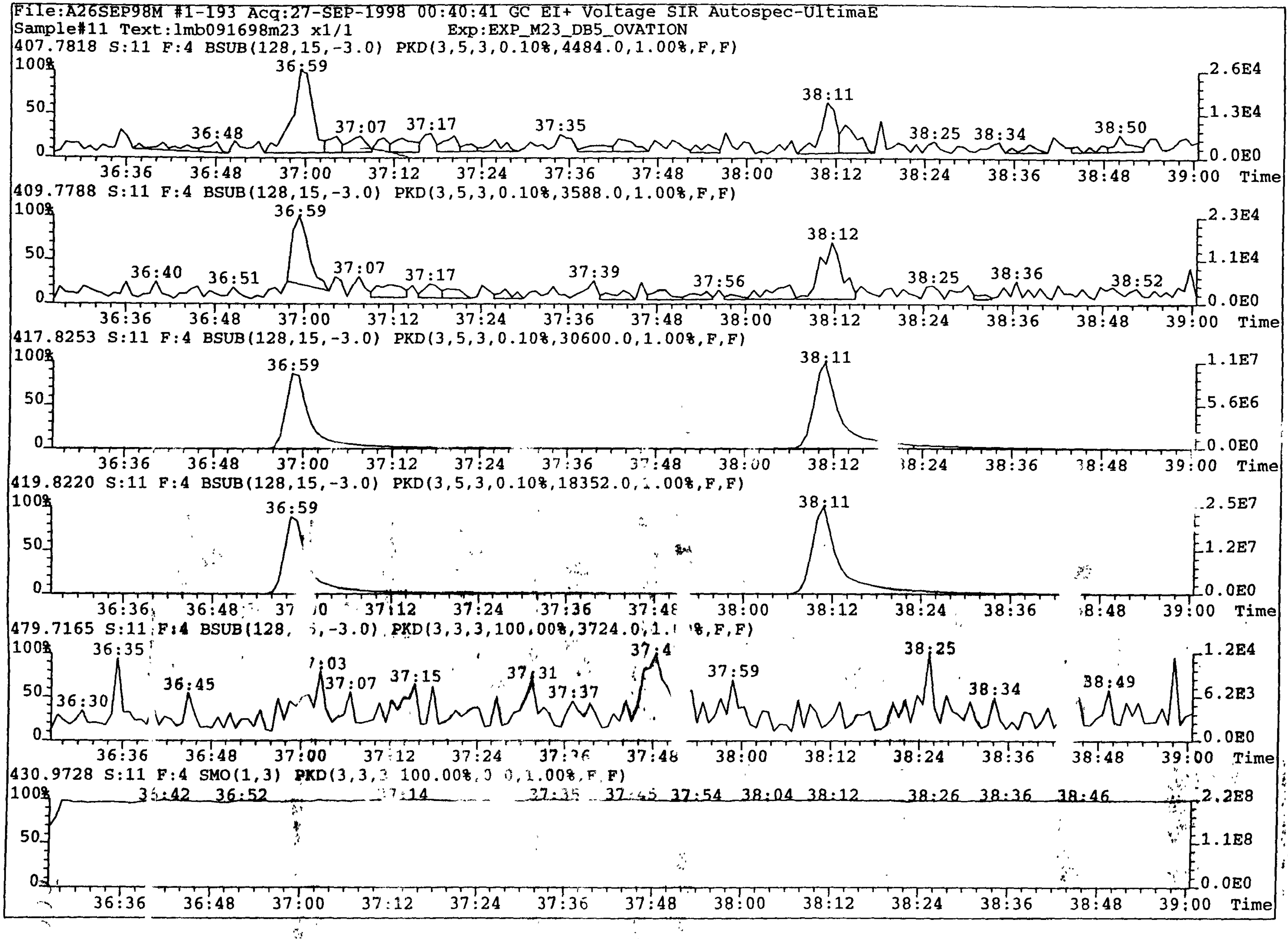
353.8970 S:11 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,2296.0,1 00%,F,F)

409.7974 S:11 F:2 BSUB(128, ,-3.0) PKD(3,3,3,100.00%,2608.0,1.( %,F,F)

366.9792 S:11 F:2 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)



```
File:A26SEP98M #1-190 Acq:27-SEP-1998 00:40:41 GC EI+ Voltage SIR Autospec-UltimaE
Sample#11 Text:lmb091698m23 x1/1          Exp:EXP_M23_DB5_OVATION
373.8207 S:11 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,4524.0,1.00%,F,F)
375.8178 S:11 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,3548.0,1.00%,F,F)
383.8639 S:11 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,81692.0,1.00%,F,F)
385.8610 S:11 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,168620.0,1.00%,F,F)
445.7555 S:11 F:3 BSUB(128,15,-3.0) PKD(3,3,3,100.00%,756.0,1.00%,F,F)
380.9760 S:11 F:3 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)
```



File:A26SEP98M #1-193 Acq:27-SEP-1998 00:40:41 GC EI+ Voltage SIR Autospec-UltimaE
Sample#11 Text:1mb091698m23 x1/1 Exp:EXP_M23_DB5_OVATION

407.7818 S:11 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,4484.0,1.00%,F,F)

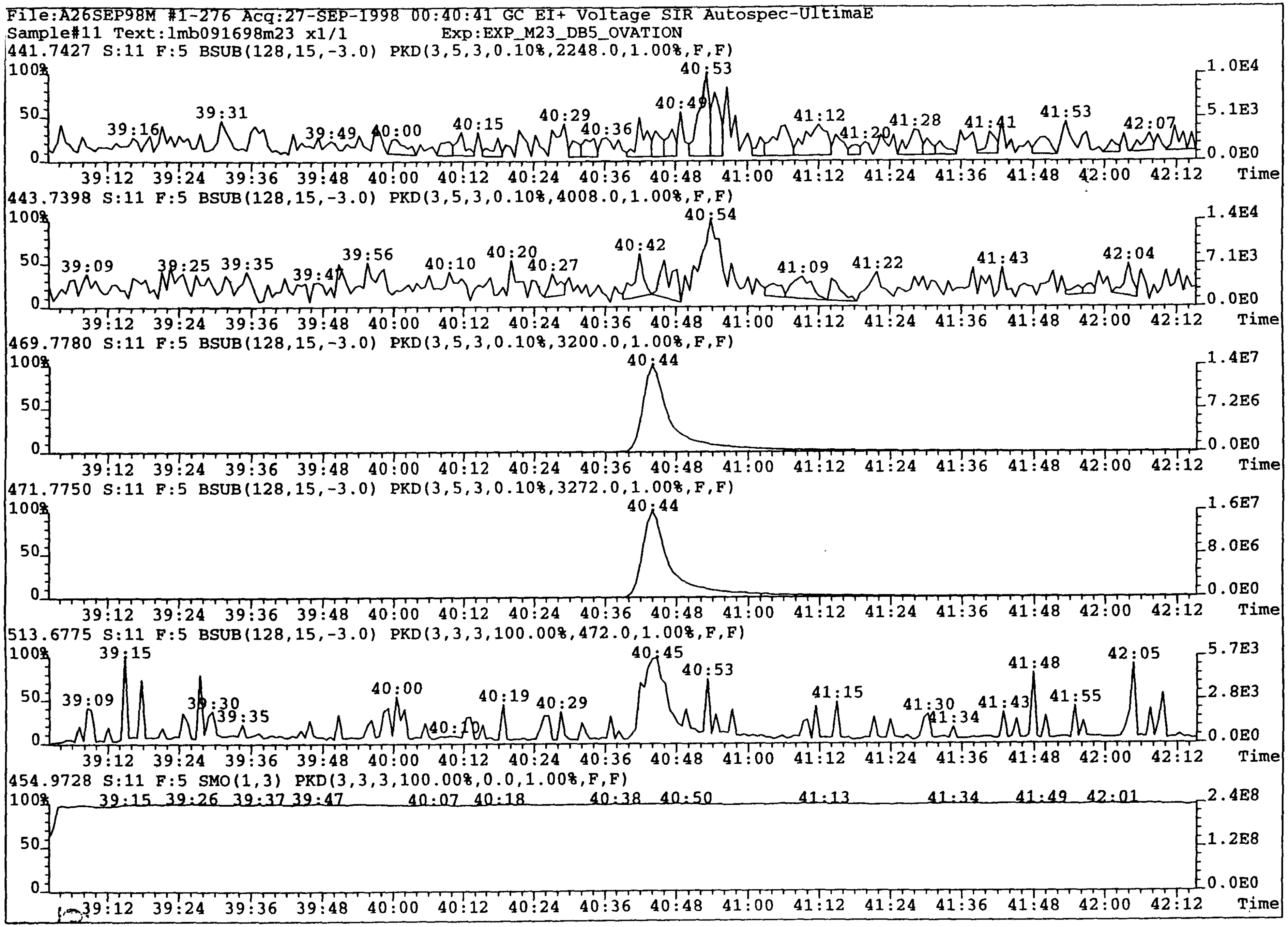
409.7788 S:11 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,3588.0,1.00%,F,F)

417.8253 S:11 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,30600.0,1.00%,F,F)

419.8220 S:11 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,18352.0,1.00%,F,F)

479.7165 S:11 F:4 BSUB(128,15,-3.0) PKD(3,3,3,100.00%,3724.0,1.00%,F,F)

430.9728 S:11 F:4 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)



```
File:A26SEP98M #1-276 Acq:27-SEP-1998 00:40:41 GC EI+ Voltage SIR Autospec-UltimaE
Sample#11 Text:lmb091698m23 x1/1          Exp:EXP_M23_DB5_OVATION
441.7427 S:11 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,2248.0,1.00%,F,F)
443.7398 S:11 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,4008.0,1.00%,F,F)
469.7780 S:11 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,3200.0,1.00%,F,F)
471.7750 S:11 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,3272.0,1.00%,F,F)
513.6775 S:11 F:5 BSUB(128,15,-3.0) PKD(3,3,3,100.00%,472.0,1.00%,F,F)
454.9728 S:11 F:5 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)
```

*Method 23*

## M23-O-3

PES

### Analytical Data Summary Sheet

| Analyte | Concentration (ng) | DL (ng) | EMPC (ng) | RT (min.) | Ratio | Qualifier |
|---|---|---|---|---|---|---|
| 2,3,7,8-TCDD | 0.0102 | 0.0015 |  | 29:27 | 0.82 |  |
| 1,2,3,7,8-PeCDD | 0.0038 | 0.0008 |  | 33:14 | 1.52 |  |
| 1,2,3,4,7,8-HxCDD | 0.0021 | 0.0020 |  | 35:19 | 1.26 |  |
| 1,2,3,6,7,8-HxCDD | 0.0030 | 0.0015 |  | 35:23 | 1.25 |  |
| 1,2,3,7,8,9-HxCDD | 0.0031 | 0.0016 |  | 35:36 | 1.37 |  |
| 1,2,3,4,6,7,8-HpCDD | EMPC | 0.0017 | 0.0075 | 37:49 | 0.87 |  |
| OCDD | EMPC | 0.0062 | 0.0319 | 40:44 | 1.14 |  |
| 2,3,7,8-TCDF | 0.309 | 0.0034 |  | 28:26 | 0.79 |  |
| 1,2,3,7,8-PeCDF | 0.101 | 0.0016 |  | 32:35 | 1.55 |  |
| 2,3,4,7,8-PeCDF | 0.0602 | 0.0015 |  | 33:01 | 1.58 |  |
| 1,2,3,4,7,8-HxCDF | 0.0207 | 0.0015 |  | 34:47 | 1.25 |  |
| 1,2,3,6,7,8-HxCDF | 0.0119 | 0.0011 |  | 34:52 | 1.17 |  |
| 2,3,4,6,7,8-HxCDF | 0.0068 | 0.0014 |  | 35:14 | 1.09 |  |
| 1,2,3,7,8,9-HxCDF | ND | 0.0016 |  | 35:45 | 2.55 |  |
| 1,2,3,4,6,7,8-HpCDF | 0.0105 | 0.0027 |  | 37:00 | 0.96 |  |
| 1,2,3,4,7,8,9-HpCDF | ND | 0.0034 |  | 38:11 | 0.73 |  |
| OCDF | ND | 0.0056 |  | 40:53 | 0.9 |  |
| Total TCDDs | 0.151 | 0.0015 |  |  |  |  |
| Total PeCDDs | 0.0448 | 0.0008 | 0.0600 |  |  |  |
| Total HxCDDs | 0.0148 | 0.0015 | 0.0488 |  |  |  |
| Total HpCDDs | ND | 0.0017 | 0.0160 |  |  |  |
| Total TCDFs | 5.68 | 0.0034 | 5.96 |  |  |  |
| Total PeCDFs | 0.940 | 0.0015 |  |  |  |  |
| Total HxCDFs | 0.0784 | 0.0011 | 0.0904 |  |  |  |
| Total HpCDFs | 0.0104 | 0.0027 |  |  |  |  |
| TEQ (ND=0) | 0.0830 |  | 0.0831 |  |  | ITEF |
| TEQ (ND=1/2) | 0.0831 |  | 0.0832 |  |  | ITEF |

**Client Information**

| | |
|---|---|
| Project Name: | S509.000 |
| Sample ID: | M23-O-3 |

**Laboratory Information**

| | |
|---|---|
| Project ID: | L1114 |
| Sample ID: | 1114-1 |
| Collection Date: | 31-Aug-98 |
| Receipt Date: | 08-Sep-98 |
| Extraction Date: | 16-Sep-98 |
| Analysis Date: | 27-Sep-98 |

**Sample Information**

| | |
|---|---|
| Matrix: | Air |
| Weight / Volume: | 1 |
| Moisture / Lipids: | 0.0 % |
| Filename: | a26sep98m-13 |
| Retchk: | a26sep98m-1 |
| Begin ConCal: | a26sep98m-5 |
| End ConCal: | a26sep98m-21 |
| Initial_Cal: | a26sep98m-21 |

*Method 23*

**M23-O-3**

PES

## Analytical Data Summary Sheet

| Labeled Standard | Expected Amount (ng) | Measured Amount (ng) | Percent Recovery (%) | RT (min.) | Ratio | Qualifier |
|---|---|---|---|---|---|---|
| **Extraction Standards** | | | | | | |
| $^{13}C_{12}$-2,3,7,8-TCDD | 4 | 3.50 | 87.5 | 29:26 | 0.8 | |
| $^{13}C_{12}$-1,2,3,7,8-PeCDD | 4 | 4.24 | 105.9 | 33:13 | 1.59 | |
| $^{13}C_{12}$-1,2,3,6,7,8-HxCDD | 4 | 3.95 | 98.8 | 35:22 | 1.27 | |
| $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD | 4 | 3.09 | 77.3 | 37:48 | 1.08 | |
| $^{13}C_{12}$-OCDD | 8 | 5.13 | 64.2 | 40:44 | 0.9 | |
| $^{13}C_{12}$-2,3,7,8-TCDF | 4 | 3.36 | 83.9 | 28:24 | 0.8 | |
| $^{13}C_{12}$-1,2,3,7,8-PeCDF | 4 | 3.34 | 83.6 | 32:34 | 1.59 | |
| $^{13}C_{12}$-1,2,3,6,7,8-HxCDF | 4 | 3.15 | 78.6 | 34:47 | 0.53 | |
| $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF | 4 | 2.54 | 63.5 | 36:59 | 0.44 | |
| **Sampling Standards** | | | | | | |
| $^{37}Cl_4$-2,3,7,8-TCDD | 4 | 4.06 | 101.5 | 29:27 | | |
| $^{13}C_{12}$-2,3,4,7,8-PeCDF | 4 | 4.15 | 103.8 | 33:01 | 1.59 | |
| $^{13}C_{12}$-1,2,3,4,7,8-HxCDD | 4 | 3.23 | 80.7 | 35:18 | 1.25 | |
| $^{13}C_{12}$-1,2,3,4,7,8-HxCDF | 4 | 4.73 | 118.3 | 34:51 | 0.53 | |
| $^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF | 4 | 3.42 | 85.4 | 38:10 | 0.43 | |
| **Injection Standards** | | | | | | |
| $^{13}C_{12}$-1,2,3,4-TCDD | | | | 29:09 | 0.81 | |
| $^{13}C_{12}$-1,2,3,7,8,9-HxCDD | | | | 35:35 | 1.28 | |

### Client Information

| | |
|---|---|
| Project Name: | S509.000 |
| Sample ID: | M23-O-3 |

### Laboratory Information

| | |
|---|---|
| Project ID: | L1114 |
| Sample ID: | 1114-1 |
| Collection Date: | 31-Aug-98 |
| Receipt Date: | 08-Sep-98 |
| Extraction Date: | 16-Sep-98 |
| Analysis Date: | 27-Sep-98 |

### Sample Information

| | | |
|---|---|---|
| Matrix: | Air | |
| Weight / Volume: | 1 | |
| Moisture / Lipids: | 0.0 | % |
| Filename: | a26sep98m-13 | |
| Retchk: | a26sep98m-1 | |
| Begin ConCal: | a26sep98m-5 | |
| End ConCal: | a26sep98m-21 | |
| Initial Cal: | a26sep98m-21 | |

Reviewed by: Y. T.

Date Reviewed: 30 Sep 98

CF = 7.733

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          Filename ; a26sep98m
            Sample ; 13
          Acquired ; 27-SEP-98  02:17:51
         Processed ; 28-SEP-98  12:05:57
         Sample ID ; 1114-1 x1/1
         Cal Table ; m8290-092698m
     Results Table ; M8290-092698M-BE
          Comments ;
Typ  ;                   Name;     Resp;    Ion 1;    Ion 2;  RA;?;   RT;      Conc;       DL;    S/N1;?;     S/N2;? ; mod?
Unk  ;           2,3,7,8-TCDD; 8.39e+05; 3.10e+05; 5.29e+05; 0.58;n; 29:27;    0.311;   0.0386;      21;y;       35;y ;  no
Unk  ;        1,2,3,7,8-PeCDD; 2.46e+05; 1.48e+05; 9.78e+04; 1.52;y; 33:14;    0.094;   0.0199;      14;y;       21;y ;  no
Unk  ;      1,2,3,4,7,8-HxCDD; 9.57e+04; 5.33e+04; 4.24e+04; 1.26;y; 35:19;    0.052;   0.0489;       6;y;        5;y ;  no
Unk  ;      1,2,3,6,7,8-HxCDD; 1.82e+05; 1.01e+05; 8.11e+04; 1.25;y; 35:23;    0.074;   0.0365;       8;y;        6;y ;  no
Unk  ;      1,2,3,7,8,9-HxCDD; 1.79e+05; 1.03e+05; 7.54e+04; 1.37;y; 35:36;    0.077;   0.0388;       8;y;        5;y ;  no
Unk  ;    1,2,3,4,6,7,8-HpCDD; 2.44e+05; 1.14e+05; 1.31e+05; 0.87;n; 37:49;    0.187;   0.0425;      14;y;       23;y ;  no
Unk  ;                   OCDD; 5.90e+05; 3.14e+05; 2.76e+05; 1.14;n; 40:44;    0.798;   0.1550;      11;y;       36;y ;  no

Unk  ;           2,3,7,8-TCDF; 7.02e+07; 3.09e+07; 3.93e+07; 0.79;y; 28:26;   18.990;   0.0846;     553;y;      487;y ;  no
Unk  ;        1,2,3,7,8-PeCDF; 7.40e+06; 4.50e+06; 2.90e+06; 1.55;y; 32:35;    2.531;   0.0405;     178;y;      137;y ;  no
Unk  ;        2,3,4,7,8-PeCDF; 4.88e+06; 2.99e+06; 1.89e+06; 1.58;y; 33:01;    1.504;   0.0365;     150;y;      108;y ;  no
Unk  ;      1,2,3,4,7,8-HxCDF; 1.19e+06; 6.59e+05; 5.27e+05; 1.25;y; 34:47;    0.517;   0.0383;      37;y;       27;y ;  no
Unk  ;      1,2,3,6,7,8-HxCDF; 9.44e+05; 5.09e+05; 4.35e+05; 1.17;y; 34:52;    0.297;   0.0277;      30;y;       23;y ;  no
Unk  ;      2,3,4,6,7,8-HxCDF; 4.44e+05; 2.32e+05; 2.12e+05; 1.09;y; 35:14;    0.170;     0337;      10;y;        8;y ;  no
Unk  ;      1,2,3,7,8,9-HxCDF; 9.32e+04; 6.69e+04; 2.62e+04  .55;n; 35:45;    0.043;     0404;       4;y;        2;n ;  no
Unk  ;    1,2,3,4,6,7,8-HpCDF; 4.41e+05; 2.16e+05; 2.25e+05  0.96;y; 37:00;    0.263;     0675;       9;y;       23;y ;  no
Unk  ;    1,2,3,4,7,8,9-HpCDF; 8.29e+04; 3.49e+04; 4.79e+04  0.73;n; 38:11;    0.063;     0856;       1;n;        4;y ;  no
Unk  ;                   OCDF; 9.16e+04; 4.35e+04; 4.81e+04  0.90;y; 40:53;    0.109;     1410;       4;y;        3;n ;  no

ES/RT;       13C-2,3,7,8-TCDD; 2.34e+08; 1.04e+08; 1.30e+08; 0.80;y; 29:26;   87.526;   0.1288;    1089;y;     3477;y ;  no
ES   ;    13C-1,2,3,7,8-PeCDD; 1.93e+08; 1.18e+08; 7.45e+07; 1.59;  ; 33:13;  105.891;   0.0507;   10664;y;    12441;y ;  no
ES   ;  13C-1,2,3,6,7,8-HxCDD; 2.55e+08; 1.42e+08; 1.12e+08; 1.27  ; 35:22;   98.777;   0.0674;    4116;y;      175;y ;  no
ES   ;13C-1,2,3,4,6,7  -HpCDD; 1.33e+08; 6.90e+07; 6.41e+07; 1.08  ; 37:48;   77.331;   0.1510;     922;y;       11;y ;  no
ES   ;                 -OCDD; 1.37e+08; 6.51e+07; 7.20e+07; 0.90  ; 40:44;  128.354;   0.0387;    3372;y;       57;y ;  no

ES/RT;       13C-2,3,  -TCDF; 3.37e+08; 1.50e+08; 1.87e+08; 0.80  ; 28:24;   83.919;   0.0592;    4003;y;       19;y ;  no
ES   ;    13C-1,2,3,   -PeCDF; 3.03e+08; 1.86e+08; 1.17e+08; 1.59  ; 32:34;   83.581;   0.0090;  495278;y;       22;y ;  no
ES   ;  13C-1,2,3,6,7  -HxCDF; 1.84e+08; 6.36e+07; 1.20e+08; 0.53  ; 34:47;   78.635;   0.6875;     507;y;       65;y ;  no
ES   ;13C-1,2,3,4,6,7  -HpCDF; 1.22e+08; 3.73e+07; 8.44e+07; 0.44  ; 36:59;   63.509;   0.1215;    1000;y;       59;y ;  no

JS   ;       13C-1,2,  4-TCDD; 2.59e+08; 1.16e+08; 1.44e+08; 0.81  ; 29:09;   53.153;        -;    1402;y;       58;y ;  no
JS   ;  13C-1,2,3,7,8,9-HxCDD; 2.31e+08; 1.30e+08; 1.01e+08; 1.28  ; 35:35;   55.023;        -;    3608;y;       96;y ;  no

CS   ;      37Cl-2,3,7,8-TCDD; 2.46e+08; 2.46e+08;        -;    -  ; 29:27;   88.790;   0.0549;    4469;y;      -; -;  no
CS   ;    13C-2,3,4,7,8-PeCDF; 3.29e+08; 2.02e+08; 1.27e+08; 1.59;y; 33:01;   86.754;   0.0086;  533114;y;    22712;y ;  no
CS   ;  13C-1,2,3,4,7,8-HxCDD; 1.22e+08; 6.80e+07; 5.42e+07; 1.25;y; 35:18;   79.667;   0.1133;    3165;y;     4030;y ;  no
CS   ;  13C-1,2,3,4,7,8-HxCDF; 3.15e+08; 1.09e+08; 2.06e+08; 0.53;y; 34:51;   92.998;   0.4747;     655;y;      738;y ;  no
CS   ;13C-1,2,3,4,7,8,9-HpCDF; 7.97e+07; 2.41e+07; 5.56e+07; 0.43;y; 38:10;   54.241;   0.1584;     520;y;     1081;y ;  no

SS   ;      37Cl-2,3,7,8-TCDD; 2.46e+08; 2.46e+08;        -;  -;-; 29:27;  101.443;   0.0728;    4469;y;      -; -;  no
SS   ;    13C-2,3,4,7,8-PeCDF; 3.29e+08; 2.02e+08; 1.27e+08; 1.59;y; 33:01;  103.796;   0.0055;  533114;y;    22712;y ;  no
SS   ;  13C-1,2,3,4,7,8-HxCDD; 1.22e+08; 6.80e+07; 5.42e+07; 1.25;y; 35:18;   80.653;   0.1116;    3165;y;     4030;y ;  no
SS   ;  13C-1,2,3,4,7,8-HxCDF; 3.15e+08; 1.09e+08; 2.06e+08; 0.53;y; 34:51;  118.265;   0.3800;     655;y;      738;y ;  no
SS   ;13C-1,2,3,4,7,8,9-HpCDF; 7.97e+07; 2.41e+07; 5.56e+07; 0.43;y; 38:10;   85.407;   0.2515;     520;y;     1081;y ;  no
```

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                                                              Page 1 of 8

 Ent: 39 Name: Total Tetra-Furans  F:1  Mass: 303.902 305.899 Mod? no   #Hom:23

 Run: 6  File: a26sep98m    S:13 Acq:27-SEP-98 02:17:51 Proc:28-SEP-98 12:05:57
 Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
 Version: V3.6 31-JUL-1998 10:51:59  Sample text: 1114-1 x1/1

   Amount: 149.02  of which 18.99   named and 130.03  unnamed
     Conc: 149.02  of which 18.99   named and 130.03  unnamed
   Tox #1: -            Tox #2: -          Tox #3: -

Name                  #     RT Respnse    RA     Conc     Area  Height     S/N  Mod?

                      1  24:35 3.2e+07  0.78 y    8.67
                                3.2e+07                 1.4e+07 3.4e+06 3.7e+02 y  n
                                                        1.8e+07 4.3e+06 3.4e+02 y  n
                      2  25:10 1.3e+07  0.78 y    3.47
                               1.3e+07                  5.6e+06 1.3e+06 1.5e+02 y  n
                                                        7.2e+06 1.6e+06 1.3e+02 y  n
                      3  25:19 1.8e+05  0.53 n    0.05
                               1.8e+05                  6.1e+04 2.5e+04 2.7e+00 n  n
                                                        1.2e+05 3.8e+04 3.0e+00 y  n
                      4  25:30 1.5e+07  0.78 y    4.04
                               1.5e+07                  6.6e+06 1.5e+06 1.7e+02 y  n
                                                        8.4e+06 1.9e+06 1.5e+02 y  n
                      5  25:48 1.0e+08  0.78 y   28.26
                               1.0e+08                  4.6e+07 8.6e+06 9.6e+02 y  n
                                                        5.9e+07 1.1e+07 8.8e+02 y  n
                      6  25:59 1.1e+07  0.79 y    3.02
                               1.1e+07                  4.9e+06 1.2e+06 1.4e+02 y  n
                                                        6.3e+06 1.5e+06 1.2e+02 y  n
                      7  26:07 2.2e+07  0.77 y    5.93
                               2.2e+07                  9.6e+06 2.1e+06 2.4e+02 y  n
                                                        1.2e+07 2.7e+06 2.1e+02 y  n
                      8  26:13 2.4e+07  1.61 n    6.48
                               2.4e+07                  1.5e+07 1.9e+06 2.1e+02 y  n
                                                        9.2e+06 2.4e+06 1.9e+02 y  n
                      9  26:37 1.6e+07  0.77 y    4.22
                               1.6e+07                  6.8e+06 1.9e+06 2.2e+02 y  n
                                                        8.8e+06 2.5e+06 2.0e+02 y  n
                      10 26:42 3.3e+07  0.79 y    8.96
                               3.3e+07                  1.5e+07 3.0e+06 3.4e+02 y  n
                                                        1.8e+07 3.8e+06 3.0e+02 y  n
                      11 26:58 1.8e+07  0.82 y    4.99
                               1.8e+07                  8.3e+06 1.8e+06 2.0e+02 y  n
                                                        1.0e+07 2.3e+06 1.8e+02 y  n
                      12 27:07 3.1e+07  0.76 y    8.41
                               3.1e+07                  1.3e+07 2.9e+06 3.3e+02 y  n
                                                        1.8e+07 3.8e+06 3.0e+02 y  n
                      13 27:24 4.0e+07  0.77 y   10.74
                               4.0e+07                  1.7e+07 3.5e+06 3.9e+02 y  n
                                                        2.3e+07 4.5e+06 3.5e+02 y  n
                      14 27:31 2.0e+07  0.78 y    5.37
                               2.0e+07                  8.7e+06 1.8e+06 2.0e+02 y  n
                                                        1.1e+07 2.3e+06 1.8e+02 y  n
                      15 27:49 4.1e+07  0.79 y   11.14
                               4.1e+07                  1.8e+07 3.7e+06 4.1e+02 y  n
                                                        2.3e+07 4.7e+06 3.7e+02 y  n
                      16 28:01 1.2e+06  0.55 n    0.34
                               1.2e+06                  4.4e+05 1.5e+05 1.6e+01 y  n
                                                        8.1e+05 2.0e+05 1.6e+01 y  n
                      17 28:09 1.6e+07  0.81 y    4.20
                               1.6e+07                  7.0e+06 1.5e+06 1.6e+02 y  n
                                                        8.6e+06 1.8e+06 1.4e+02 y  n
 2,3,7,8-TCDF         18 28:26 7.0e+07  0.79 y   18.99
                               7.0e+07                  3.1e+07 5.0e+06 5.5e+02 y  n
                                                        3.9e+07 6.2e+06 4.9e+02 y  n
                      19 29:02 2.6e+07  0.77 y    7.14
                               2.6e+07                  1.2e+07 2.4e+06 2.6e+02 y  n
                                                        1.5e+07 3.1e+06 2.4e+02 y  n
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                    20 29:19 1.3e+07  0.78 y    3.40
                             1.3e+07                5.5e+06 1.1e+06 1.2e+02 y  n
                                                    7.1e+06 1.4e+06 1.1e+02 y  n
                    21 29:33 2.6e+06  0.77 y    0.71
                             2.6e+06                1.1e+06 2.3e+05 2.6e+01 y  n
                                                    5[illegible] 2.7e+05 2.2e+01 y  n
                    22 30:36 1.9e+05  4.05 n    0.05
                             1.9e+05                1.5e+05 3.7e+04 4.2e+00 y  n
                                                    3.8e+04 1.3e+04 1.0e+00 n  n
                    23 30:47 1.7e+06  0.86 y    0.45
                             1.7e+06                [illegible]e+05 1.9e+05 2.1e+01 y  n
                                                    8.9e+05 2.4[illegible]
```

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                                                                   Page 2 of 8

 Ent: 40 Name: Total Tetra-Dioxins F:1  Mass: 319.897 321.894 Mod? no   #Hom:18

 Run: 6  File: a26sep98m    S:13 Acq:27-SEP-98 02:17:51 Proc:28-SEP-98 12:05:57
 Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
 Version: V3.6 31-JUL-1998 10:51:59  Sample text: 1114-1 x1/1

   Amount: 4.21    of which 0.31    named and 3.90    unnamed
     Conc: 4.21    of which 0.31    named and 3.90    unnamed
   Tox #1: -           Tox #2: -          Tox #3: -

Name                  #     RT Respnse    RA    Conc    Area  Height    S/N  Mod?

                      1  26:12 3.3e+06  0.77 y   1.22
                               3.3e+06                1.4e+06 3.2e+05 9.2e+01 y  n
                                                      1.9e+06 4.3e+05 1.5e+02 y  n
                      2  26:39 1.8e+06  0.78 y   0.67
                               1.8e+06                7.9e+05 1.7e+05 5.0e+01 y  n
                                                      1.0e+06 2.3e+05 7.8e+01 y  n
                      3  27:01 2.2e+05  0.61 n   0.08
                               2.2e+05                8.3e+04 2.3e+04 6.7e+00 y  n
                                                      1.4e+05 3.5e+04 1.2e+01 y  n
                      4  27:53 1.8e+06  0.76 y   0.67
                               1.8e+06                7.8e+05 1.5e+05 4.3e+01 y  n
                                                      1.0e+06 1.8e+05 6.2e+01 y  n
                      5  28:05 2.9e+05  0.72 y   0.11
                               2.9e+05                1.2e+05 2.2e+04 6.4e+00 y  n
                                                      1.7e+05 2.5e+04 8.6e+00 y  n
                      6  28:15 3.0e+05  0.76 y   0.11
                               3.0e+05                1.3e+05 3.0e+04 8.8e+00 y  n
                                                      1.7e+05 3.8e+04 1.3e+01 y  n
                      7  28:18 1.1e+05  0.25 n   0.04
                               1.1e+05                2.2e+04 1.6e+04 4.6e+00 y  n
                                                      8.6e+04 1.8e+04 6.2e+00 y  n
                      8  28:22 2.0e+05  1.32 n   0.07
                               2.0e+05                1.1e+05 2.3e+04 6.6e+00 y  n
                                                      8.6e+04 1.8e+04 6.2e+00 y  n
                      9  28:44 3.0e+05  1.12 n   0.11
                               3.0e+05                1.6e+05 3.0e+04 8.6e+00 y  n
                                                      1.4e+05 3.2e+04 1.1e+01 y  n
                     10  29:10 4.4e+05  0.86 y   0.16
                               4.4e+05                2.0e+05 4.3e+04 1.2e+01 y  n
                                                      2.4e+05 5.1e+04 1.7e+01 y  n
                     11  29:18 8.7e+05  0.84 y   0.32
                               8.7e+05                3.9e+05 8.2e+04 2.4e+01 y  n
                                                      4.7e+05 9.8e+04 3.3e+01 y  n
2,3,7,8-TCDD         12  29:27 8.4e+05  0.58 n   0.31
                               8.4e+05                3.1e+05 7.1e+04 2.1e+01 y  n
                                                      5.3e+05 1.0e+05 3.5e+01 y  n
                     13  29:39 1.7e+05  0.84 y   0.06
                               1.7e+05                7.5e+04 1.4e+04 4.2e+00 y  n
                                                      9.0e+04 1.9e+04 6.4e+00 y  n
                     14  29:57 5.4e+05  0.75 y   0.20
                               5.4e+05                2.3e+05 4.1e+04 1.2e+01 y  n
                                                      3.1e+05 6.6e+04 2.3e+01 y  n
                     15  30:04 2.4e+04  0.36 n   0.01
                               2.4e+04                6.5e+03 3.7e+03 1.1e+00 n  n
                                                      1.8e+04 1.0e+04 3.5e+00 y  n
                     16  30:10 1.6e+04  0.39 n   0.01
                               1.6e+04                4.4e+03 2.5e+03 7.2e-01 n  n
                                                      1.1e+04 4.6e+03 1.6e+00 n  n
                     17  30:18 1.0e+05  1.64 n   0.04
                               1.0e+05                6.4e+04 1.2e+04 3.6e+00 y  n
                                                      3.9e+04 1.1e+04 3.7e+00 y  n
                     18  30:51 6.4e+04  1.07 n   0.02
                               6.4e+04                3.3e+04 1.0e+04 2.9e+00 n  n
                                                      3.1e+04 9.6e+03 3.3e+00 y  n
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  Ent: 41 Name: Total Penta-Furans  F:2  Mass: 339.860 341.857 Mod? no   #Hom:18

  Run: 6  File: a26sep98m    S:13 Acq:27-SEP-98 02:17:51 Proc:28-SEP-98 12:05:57
  Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal. m8290-092»Results: M8290-09»
  Version: V3.6 31-JUL-1998 10:51:59  Sample text: 1114-1 x1/1

    Amount: 23.56   of which 4.04    named and 19.52   unnamed
      Conc: 23.56   of which 4.04    named and 19.52   unnamed
    Tox #1: -           Tox #2: -        Tox #3. -

Name                     #      RT Respnse     RA     Conc    Area  Height     S/N  Mod?

                         1   31:07 7.3e+06   1.57 y    2.37
                                   7.3e+06                  4.5e+06 1.3e+06 1.5e+02 y  n
                                                            2.8e+06 7.7e+05 1.0e+02 y  n
                         2   31:15 4.3e+04   3.07 n    0.01
                                   4.3e+04                  3.3e+04 1.2e+04 1.5e+00 n  n
                                                            1.1e+04 6.6e+03 8.8e-01 n  n
                         3   31:40 7.1e+04   1.71 y    0.02
                                   7.1e+04                  4.5e+04 1.6e+04 1.9e+00 n  n
                                                            2.6e+04 1.1e+04 1.5e+00 n  n
                         4   31:47 2.1e+04   1 90 n    0
                                   2.1e+04                  1.4e+04 3.3e+03 4.0e-01 n  n
                                                            7.3e+03 3.2e+03 4.2e-01 n  n
                         5   31:57 4.0e+06   1.53 y    1.30
                                   4.0e+06                  2.4e+06 1.0e+06 1.2e+02 y  n
                                                            1.6e+06 6.4e+05 8.5e+01 y  n
                         6   32:02 2.4e+07   1.57 y    7.88
                                   2.4e+07                  1.5e+07 5.4e+06 6.4e+02 y  n
                                                            9.4e+06 3.6e+06 4.8e+02 y  n
                         7   32:09 4.1e+06   1.45 y    1.33
                                   4.1e+06                  2.4e+06 6.6e+05 7.8e+01 y  n
                                                            1.7e+06 4.2e+05 5.7e+01
                         8   32:15 8.8e+05   1.47 y    0.28
                                   8.8e+05                  5.2e+05 2.2e+05 2.6e+01 y  n
                                                            3.5e+05 1.5e+05 2.0e+01 y  n
                         9   32:17 9.5e+05   1.58 y    0.3
                                   9.5e+05                  5.8e+05 2.8e+05 3.3e+01 y  n
                                                            3.7e+05 1.8e+05 2.4e+01 y  n
                         10  32:23 6.3e+06   1.52 y    2.03
                                   6.3e+06                  3.8e+06 1.3e+06 1.5e+02 y  n
                                                            2.5e+06 8.5e+05 1.1e+02 y  n
1,2,3,7,8-PeCDF          11  32:35 7.4e+06   1.55 y    2.53
                                   7.4e+06                  4.5e+06 1.5e+06 1.8e+02 y  n
                                                            2.9e+06 1.0e+06 1.4e+02 y  n
                         12  32:41 3.0e+06   1.56 y    0.98
                                   3.0e+06                  1.8e+06 7.5e+05 9.0e+01 y  n
                                                            1.2e+06 4.7e+05 6.3e+01 y  n
                         13  32:46 4.8e+06   1.57 y    1.56
                                   4.8e+06                  2.9e+06 1.2e+06 1.4e+02 y  n
                                                            1.9e+06 7.5e+05 1.0e+02 y  n
2,3,4,7,8-PeCDF          14  33:01 4.9e+06   1.58 y    1.50
                                   4.9e+06                  3.0e+06 1.3e+06 1.5e+02 y  n
                                                            1.9e+06 8.1e+05 1.1e+02 y  n
                         15  33:06 3.5e+06   1.43 y    1.13
                                   3.5e+06                  2.1e+06 8.1e+05 9.6e+01 y  n
                                                            1.4e+06 4.9e+
                         16  33:15 4.3e+05   1.58 y    0.14
                                   4.3e+05                  2.6e+05 9.0e+04 1.1e+01 y  n
                                                            1.7e+05 5.3e+04 7.1e+00 y  n
                         17  33:22 2.0e+04   0.90 n    0.01
                                   2.0e+04                  9.6e+03 5.0e+03 6.0e-01 n  n
                                                            1.1e+04 5.2e+03 7.0e-01 n  n
                         18  33:35 5.0e+05   1.52 y    0.16
                                   5.0e+05                  3.0e+05 1.3e+05 1.5e+01 y  n
                                                            2.0e+05 6.7e+04 9.0e+00 y  n
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                                                                    Page 4 of 8

 Ent: 42 Name: Total Penta-Dioxins F:2  Mass: 355.855 357.852 Mod? no   #Hom:14

 Run: 6  File: a26sep98m    S:13 Acq:27-SEP-98 02:17:51 Proc:28-SEP-98 12:05:57
 Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
 Version: V3.6 31-JUL-1998 10:51:59  Sample text: 1114-1 x1/1

   Amount: 1.54    of which 0.09    named and 1.44    unnamed
     Conc: 1.54    of which 0.09    named and 1.44    unnamed
   Tox #1: -            Tox #2: -         Tox #3: -

Name               #     RT Respnse     RA    Conc    Area  Height    S/N  Mod?

                   1  32:09 1.1e+06  1.67 y    0.43
                            1.1e+06                   7.0e+05 2.8e+05 6.2e+01 y  n
                                                      4.2e+05 1.7e+05 8.0e+01 y  n
                   2  32:18 3.0e+04  0.90 n    0.01
                            3.0e+04                   1.4e+04 5.4e+03 1.2e+00 n  n
                                                      1.6e+04 6.1e+03 2.9e+00 n  n
                   3  32:36 9.4e+05  1.77 y    0.36
                            9.4e+05                   6.0e+05 2.6e+05 5.6e+01 y  n
                                                      3.4e+05 1.5e+05 7.4e+01 y  n
                   4  32:42 2.0e+05  2.29 n    0.08
                            2.0e+05                   1.4e+05 5.0e+04 1.1e+01 y  n
                                                      6.0e+04 2.4e+04 1.2e+01 y  n
                   5  32:47 6.4e+05  1.63 y    0.24
                            6.4e+05                   4.0e+05 1.6e+05 3.5e+01 y  n
                                                      2.4e+05 1.1e+05 5.5e+01 y  n
                   6  32:53 6.5e+04  3.02 n    0.02
                            6.5e+04                   4.9e+04 2.0e+04 4.3e+00 y  n
                                                      1.6e+04 7.7e+03 3.7e+00 y  n
                   7  32:56 1.4e+05  2.16 n    0.06
                            1.4e+05                   9.9e+04 3.0e+04 6.5e+00 y  n
                                                      4.6e+04 1.5e+04 7.1e+00 y  n
                   8  33:03 3.5e+05  2.03 n    0.13
                            3.5e+05                   2.3e+05 8.7e+04 1.9e+01 y  n
                                                      1.2e+05 4.6e+04 2.2e+01 y  n
                   9  33:06 1.2e+05  1.97 n    0.05
                            1.2e+05                   8.0e+04 2.6e+04 5.5e+00 y  n
                                                      4.0e+04 1.6e+04 7.8e+00 y  n
1,2,3,7,8-PeCDD    10 33:14 2.5e+05  1.52 y    0.09
                            2.5e+05                   1.5e+05 6.6e+04 1.4e+01 y  n
                                                      9.8e+04 4.4e+04 2.1e+01 y  n
                   11 33:18 9.5e+04  2.05 n    0.04
                            9.5e+04                   6.4e+04 2.4e+04 5.3e+00 y  n
                                                      3.1e+04 1.2e+04 5.6e+00 y  n
                   12 33:31 3.9e+04  1.87 n    0.01
                            3.9e+04                   2.5e+04 1.3e+04 2.9e+00 n  n
                                                      1.4e+04 7.9e+03 3.8e+00 y  n
                   13 33:33 2.6e+04  0.95 n    0.01
                            2.6e+04                   1.3e+04 6.6e+03 1.4e+00 n  n
                                                      1.4e+04 7.9e+03 3.8e+00 y  n
                   14 33:39 1.1e+04  2.24 n    0.00
                            1.1e+04                   7.3e+03 3.9e+03 8.4e-01 n  n
                                                      3.3e+03 1.8e+03 8.5e-01 n  n
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  Ent: 43 Name: Total Hexa-Furans   F:3  Mass: 373.821 375.818 Mod? no   #Hom:24

  Run: 6  File: a26sep98m    S:13 Acq:27-SEP-98 02:17:51 Proc:28-SEP-98 12:05:57
  Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
  Version: V3.6 31-JUL-1998 10:51:59  Sample text: 1114-1 x1/1

     Amount: 2.47     of which 1.03     named and 1.45     unnamed
       Conc: 2.47     of which 1.03     named and 1.45     unnamed
     Tox #1: -            Tox #2: -          Tox #3: -

 Name                    #     RT Respnse     RA    Conc     Area  Height     S/N  Mod?

                         1  34:08 4.6e+05  1.30 y   0.18
                                  4.6e+05                 2.6e+05 1.1e+05 1.8e+01 y  n
                                                          2.0e+05 9.0e+04 1.3e+01 y  n
                         2  34:14 1.8e+06  1.24 y   0.70
                                  1.8e+06                 9.9e+05 3.6e+05 5.7e+01 y  n
                                                          8.0e+05 3.1e+05 4.6e+01 y  n
                         3  34:20 2.5e+05  1.03 n   0.10
                                  2.5e+05                 1.3e+05 4.8e+04 7.6e+00 y  n
                                                          1.2e+05 3.3e+04 5.0e+00 y  n
                         4  34:25 2.3e+05  1.20 y
                                  2.3e+05                 1.3e+05 4.6e+04 7.3e+00 y  n
                                                          1.1e+05 3.7e+04 5.5e+00 y  n
                         5  34:32 1.2e+05  1.29 y   0.05
                                  1.2e+05                 6.5e+04 2.6e+04 4.1e+00 y  n
                                                          5.1e+04 1.9e+04 2.8e+00 n  n
 1,2,3,4,7,8-HxCDF       6  34:47 1.2e+06  1.25 y   0.52
                                  1.2e+06                 6.6e+05 2.4e+05 3.7e+01 y  n
                                                          5.3e+05 1.8e+05 2.7e+01 y  n
 1,2,3,6,7,8-HxCDF       7  34:52 9.4e+05  1.17 y   0.30
                                  9.4e+05                 5.1e+05 1.9e+05 3.0e+01 y  n
                                                          4.3e+05 1.[illegible]
                         8  34:55 2.5e+05  1.52 n   0.10
                                  2.5e+05                 1.5e+05 4.4e+04 6.9e+00 y  n
                                                          1.0e+05 3.7e+04 5.5e+00 y  n
                         9  35:05 2.6e+05  1.05 n   0.10
                                  2.6e+05                 1.3e+05 3.6e+04 5.7e+00 y  n
                                                          1.3e+05 3.7e+04 5.4e+00 y  n
 2,3,4,6,7,8-HxCDF      10  35:14 4.4e+05  1.09 y   0.17
                                  4.4e+05                 2.3e+05 6.2e+04 9.8e+00 y  n
                                                          2.1e+05 5.2e+04 7.8e+00 y  n
                        11  35:18 6.8e+04  1.50 n   0.03
                                  6.8e+04                 4.1e+04 1.3e+04 2.1e+00 n  n
                                                          2.7e+04 9.3e+03 1.4e+00 n  n
                        12  35:22 3.1e+04  1.49 n   0.01
                                  3.1e+04                 1.9e+04 7.7e+03 1.2e+00 n  n
                                                          1.3e+04 4.7e+03 7.0e-01 n  n
                        13  35:26 1.4e+04  0.86 n   0.01
                                  1.4e+04                 6.6e+03 3.0e+03 4.8e-01 n  n
                                                          7.6e+03 4.3e+03 6.3e-01 n  n
                        14  35:33 2.1e+04  1.75 n   0.01
                                  2.1e+04                 1.3e+04 3.9e+03 6.2e-01 n  n
                                                          7.7e+03 5.3e+03 7.8e-01 n  n
                        15  35:37 2.2e+04  1.77 n   0.01
                                  2.2e+04                 1.4e+04 4.6e+03 7.3e-01 n  n
                                                          7.8e+03 4.7e[illegible]
 1,2,3,7,8,9-HxCDF      16  35:45 9.3e+04  2.55 n   0.04
                                  9.3e+04                 6.7e+04 2.6e+04 4.1e+00 y  n
                                                          2.6e+04 1.6e+04 2.4e+00 n  n
                        17  35:49 1.0e+05  1.60 n   0.04
                                  1.0e+05                 6.5e+04 1.6e+04 2.6e+00 n  n
                                                          4.0e+04 1.2e+04 1.8e+00 n  n
                        18  36:00 1.3e+04  1.31 y   0.01
                                  1.3e+04                 7.4e+03 3.9e+03 6.2e-01 n  n
                                                          5.7e+03 2.8e+03 4.2e-01 n  n
                        19  36:06 1.9e+04  0.80 n   0.01
                                  1.9e+04                 8.5e+03 3.5e+03 5.5e-01 n  n
                                                          1.1e+04 3.3e+03 4.9e-01 n  n
```

```
                    20 36:09 1.1e+04  1.66 n     0.00
                             1.1e+04                 6.6e+03 5.2e+03 8.1e-01 n  n
                                                     4.0e+03 2.7e+03 4.0e-01 n  n
                    21 36:11 2.2e+04  0.86 n     0.01
                             2.2e+04                 1.0e+04 4.5e+03 7.1e-01 n  n
                                                     1.2e+04 3.8e+03 5.6e-01 n  n
                    22 36:15 1.0e+04  11.94n     0.00
                             1.0e+04                 9.5e+03 4.3e+03 6.8e-01 n  n
                                                     8.0e+02 6.5e+02 9.6e-02 n  n
                    23 36:19 1.8e+04  1.48 n     0.01
                             1.8e+04                 1.1e+04 6.4e+03 1.0e+00 n  n
                                                     7.4e+03 2.4e+03 3.6e-01 n  n
                    24 36:22 1.3e+04  0.73 n     0.00
                             1.3e+04                 5.4e+03 3.3e+03 5.2e-01 n  n
                                                     7.4e+03 2.4e+03 3.6e-01 n  n
```

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                                                              Page 6 of 8

  Ent: 44 Name: Total Hexa-Dioxins  F:3  Mass: 389.816 391.813 Mod? no    #Hom:19

  Run: 6  File: a26sep98m    S:13 Acq:27-SEP-98 02:17:51 Proc:28-SEP-98 12:05:57
  Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
  Version: V3.6 31-JUL-1998 10:51:59  Sample text: 1114-1 x1/1

    Amount: 1.31     of which 0.20    named and 1.11     unnamed
      Conc: 1.31     of which 0.20    named and 1.11     unnamed
    Tox #1: -            Tox #2: -         Tox #3: -

Name                   #     RT Respnse     RA     Conc     Area  Height     S/N  Mod?

                       1  34:28 1.3e+05  1.89 n    0.06
                                1.3e+05                 8.2e+04 3.1e+04 7.5e+00 y  n
                                                        4.3e+04 2.6e+04 5.7e+00 y  n
                       2  34:33 5.5e+03  0.62 n    0.00
                                5.5e+03                 2.1e+03 1.3e+03 3.1e-01 n  n
                                                        3.4e+03 1.5e+03 3.3e-01 n  n
                       3  34:37 1.2e+04  0.33 n    0.01
                                1.2e+04                 2.9e+03 1.2e+03 2.8e-01 n  n
                                                        [illegible] 4.3e+03 9.4e-01 n  n
                       4  34:47 1.8e+06  1.44 n    [illegible]
                                1.8e+06                 1.0e+06 4.0e+05 9.6e+01 y  n
                                                        7.2e+05 2.9e+05 6.3e+01 y  n
                       5  34:57 3.7e+05  1.15 y    0.17
                                3.7e+05                 2.0e+05 5.8e+04 1.4e+01 y  n
                                                        1.7e+05 4.9e+04 1.1e+01 y  n
                       6  35:02 5.6e+04  1.37 y    0.03
                                5.6e+04                 3.2e+04 1.0e+04 2.4e+00 n  n
                                                        2.4e+04 1.1e+04 2.4e+00 n  n
                       7  35:07 1.7e+04  0.89 n    0.01
                                1.7e+04                 7.9e+03 4.2e+03 1.0e+00 n  n
                                                        8.9e+03 [illegible]
                       8  35:09 1.6e+04  0.73 n    0.01
                                1.6e+04                 6.6e+03 3.4e+03 8.1e-01 n  n
                                                        [illegible].9e+03 3.3e+03 7.2e-01 n  n
                       9  35:14 1.8e+04  0.87 n    [illegible]
                                1.8e+04                 8.3e+03 3.1e+03 7.5e-01 n  n
                                                        9.5e+03 4.5e+03 1.0e+00 n  n
1,2,3,4,7,8-HxCDD     10  35:19 9.6e+04  1.26 y    0.05
                                9.6e+04                 5.3e+04 2.3e+04 5.6e+00 y  n
                                                        4.2e+04 2.4e+04 5.2e+00 y  n
1,2,3,6,7,8-HxCDD     11  35:23 1.8e+05  1.25 y    [illegible]
                                1.8e+05                 1.0e+05 3.5e+04 8.4e+00 y  n
                                                        8.1e+04 2.7e+04 6.0e+00 y  n
                      12  35:30 1.8e+04  2.15 n    0.01
                                1.8e+04                 1.2e+04 3.8e+03 9.2e-01 n  n
                                                        5.6e+03 3.5e+03 7.7e-01 n  n
1,2,3,7,8,9-HxCDD     13  35:36 1.8e+05  1.37 y    0.08
                                1.8e+05                 1.0e+05 3.5e+04 8.4e+00 y  n
                                                        7.5e+04 2.4e+04 5.3e+00 y  n
                      14  36:02 1.6e+04  0.45 n    0.01
                                1.6e+04                 5.1e+03 3.5e+03 8.3e-01 n  n
                                                        1.1e+04 5.3e+03 1.2e+00 n  n
                      15  36:04 1.6e+04  0.39 n    0.01
                                1.6e+04                 4.4e+03 2.3e+03 5.4e-01 n  n
                                                        1.1e+04 5.3e+
                      16  36:09 1.2e+04  1.82 n    0.01
                                1.2e+04                 7.5e+03 4.0e+03 9.7e-01 n  n
                                                        4.1e+03 1.9e+03 4.2e-01 n  n
                      17  36:11 8.1e+03  0.96 n    0.00
                                8.1e+03                 4.0e+03 1.9e+03 4.7e-01 n  n
                                                        4.1e+03 1.9e+03 4.2e-01 n  n
                      18  36:14 8.4e+03  0.18 n    0.00
                                8.4e+03                 1.3e+03 1.0e+03 2.5e-01 n  n
                                                        7.1e+03 2.9e+03 6.4e-01 n  n
                      19  36:18 1.5e+04  1.11 y    0.01
                                1.5e+04                 7.8e+03 2.4e+03 5.8e-01 n  n
                                                        7.1e+03 2.9e+03 6.4e-01 n  n
```

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                                                          Page 7 of 8

Ent: 45 Name: Total Hepta-Furans  F:4  Mass: 407.782 409.779 Mod? no   #Hom:11

Run: 6  File: a26sep98m     S:13 Acq:27-SEP-98 02:17:51 Proc:28-SEP-98 12:05:57
Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
Version: V3.6 31-JUL-1998 10:51:59  Sample text: 1114-1 x1/1

  Amount: 0.52     of which 0.33     named and 0.20     unnamed
    Conc: 0.52     of which 0.33     named and 0.20     unnamed
  Tox #1: -               Tox #2: -          Tox #3: -

Name                   #     RT Respnse     RA     Conc     Area  Height     S/N  Mod?

1,2,3,4,6,7,8-HpCDF1  37:00 4.4e+05  0.96 y    0.26
                            4.4e+05                   2.2e+05 6.6e+04 8.8e+00 y  n
                                                      2.3e+05 6.9e+04 2.3e+01 y  n
                     2  37:12 9.3e+04  0.73 n    0.06
                            9.3e+04                   3.9e+04 9.3e+03 1.2e+00 n  n
                                                      5.4e+04 1.6e+04 5.4e+00 y  n
                     3  37:18 7.7e+04  0.89 y    0.05
                            7.7e+04                   3.6e+04 1.1e+04 1.4e+00 n  n
                                                      4.1e+04 1.2e+04 4.0e+00 y  n
                     4  37:26 1.6e+04  1.11 y    0.01
                            1.6e+04                   8.5e+03 4.0e+03 5.3e-01 n  n
                                                      7.7e+03 2.5e+03 8.4e-01 n  n
                     5  37:29 2.0e+04  1.62 n    0.01
                            2.0e+04                   1.2e+04 6.4e+03 8.5e-01 n  n
                                                      7.7e+03 2.5e+03 8.4e-01 n  n
                     6  37:41 2.7e+04  5.26 n    0.02
                            2.7e+04                   2.3e+04 6.6e+03 8.7e-01 n  n
                                                      4.3e+03 1.5e+03 5.2e-01 n  n
                     7  37:59 1.9e+04  2.10 n    0.01
                            1.9e+04                   1.3e+04 4.8e+03 6.4e-01 n  n
                                                      6.3e+03 3.0e+03 1.0e+00 n  n
1,2,3,4,7,8,9-HpCDF8  38:11 8.3e+04  0.73 n    0.06
                            8.3e+04                   3.5e+04 9.2e+03 1.2e+00 n  n
                                                      4.8e+04 1.1e+04 3.6e+00 y  n
                     9  38:33 1.2e+04  6.61 n    0.01
                            1.2e+04                   1.0e+04 4.1e+03 5.5e-01 n  n
                                                      1.6e+03 7.8e+02 2.6e-01 n  n
                    10  38:38 1.8e+04  2.82 n    0.01
                            1.8e+04                   1.4e+04 4.0e+03 5.4e-01 n  n
                                                      4.8e+03 2.1e+03 6.9e-01 n  n
                    11  38:47 1.6e+04  2.93 n    0.01
                            1.6e+04                   1.2e+04 3.7e+03 4.9e-01 n  n
                                                      4.1e+03 1.3e+03 4.2e-01 n  n


                                                          Page 8 of 8

Ent: 46 Name: Total Hepta-Dioxins F:4  Mass: 423.777 425.774 Mod? no   #Hom:13

Run: 6  File: a26sep98m     S:13 Acq:27-SEP-98 02:17:51 Proc:28-SEP-98 12:05:57
Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
Version: V3.6 31-JUL-1998 10:51:59  Sample text: 1114-1 x1/1

  Amount: 0.54     of which 0.19     named and 0.35     unnamed
    Conc: 0.54     of which 0.19     named and 0.35     unnamed
  Tox #1: -               Tox #2: -          Tox #3: -

Name                   #     RT Respnse     RA     Conc     Area  Height     S/N  Mod?

                     1  37:13 2.7e+05  1.24 n    0.21
                            2.7e+05                   1.5e+05 4.7e+04 1.9e+01 y  n
                                                      1.2e+05 3.5e+04 2.2e+01 y  n
                     2  37:26 1.5e+04  0.34 n    0.01
                            1.5e+04                   3.9e+03 2.5e+03 9.9e-01 n  n
                                                      1.1e+04 2.6e+03 1.7e+00 n  n
                     3  37:29 2.1e+04  0.82 n    0.02
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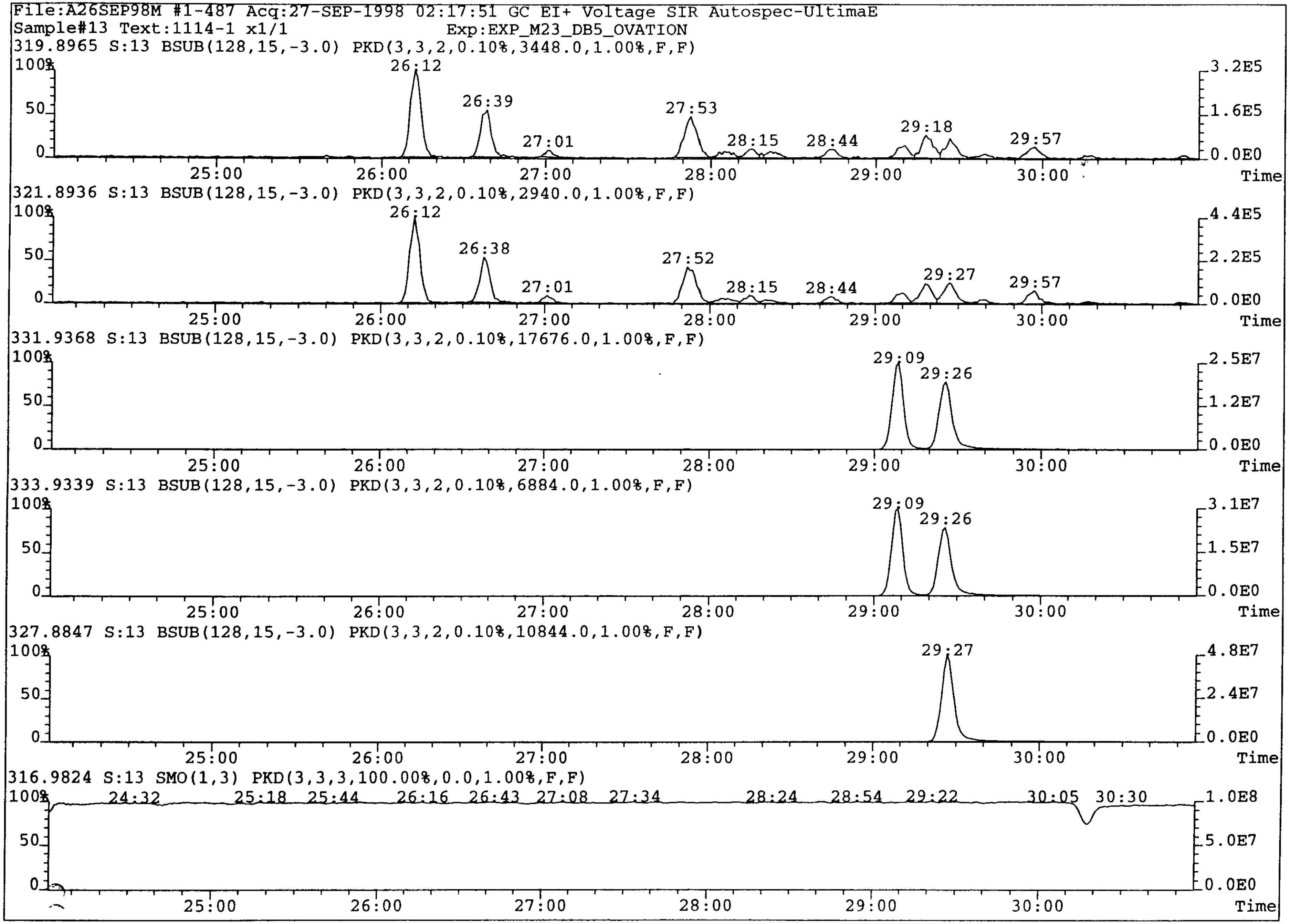
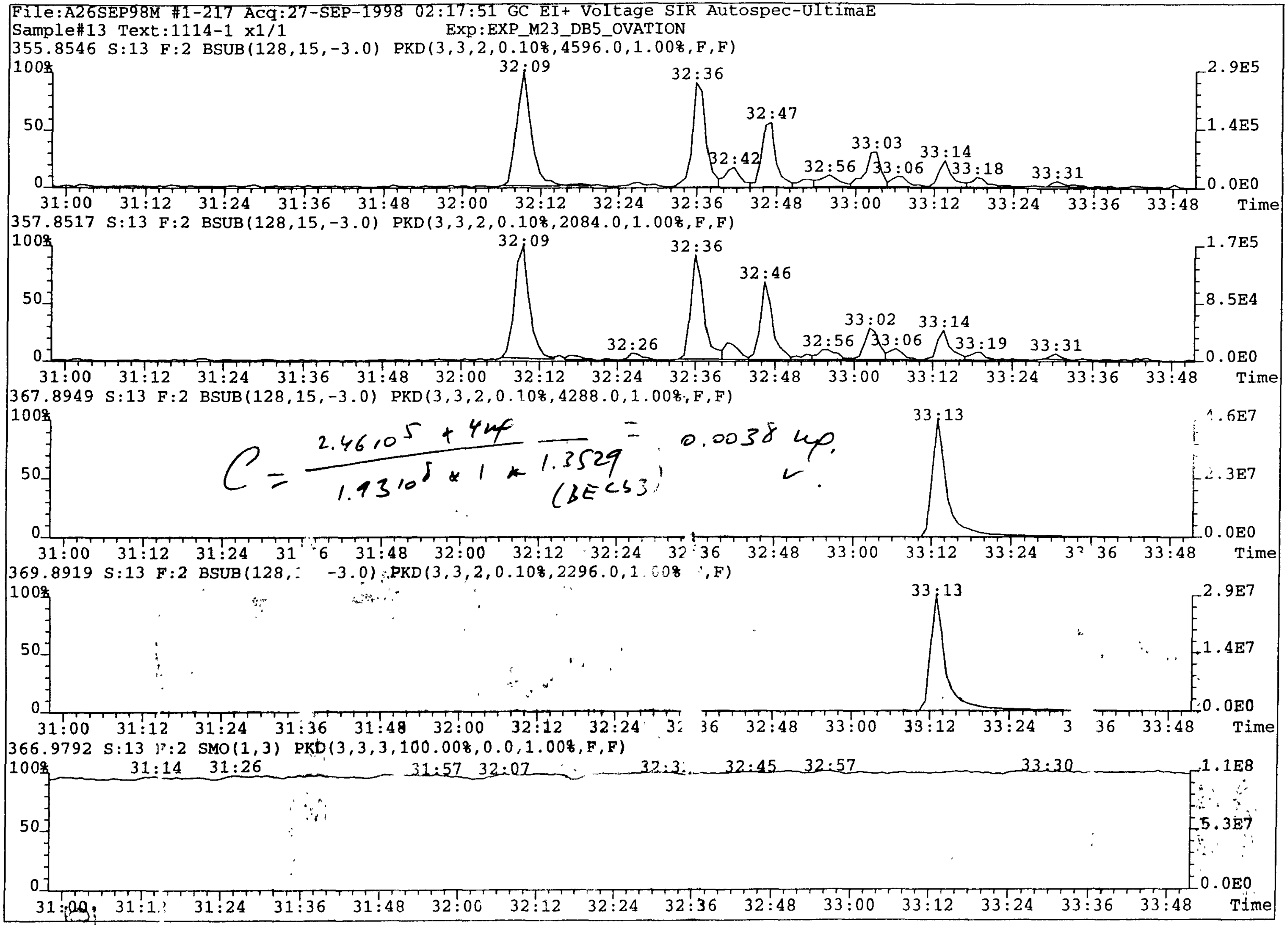
                              2.1e+04                  9.3e+03 2.9e+03 1.2e+00 n  n
                                                       1.1e+04 2.6e+03 1.7e+00 n  n
1,2,3,4,6,7,8-HpCDD4  37:49 2.4e+05  0.87 n    0.19
                              2.4e+05                  1.1e+05 3.4e+04 1.4e+01 y  n
                                                       1.3e+05 3.5e+04 2.3e+01 y  n
                      5   38:01 2.7e+04  1.13 y    0.02
                              2.7e+04                  1.4e+04 4.6e+03 1.9e+00 n  n
                                                       1.3e+04 5.4e+03 3.4e+00 y  n
                      6   38:09 2.4e+04  1.34 n    0.02
                              2.4e+04                  1.4e+04 6.3e+03 2.5e+00 n  n
                                                       1.0e+04 3.9e+03 2.4e+00 [illegible] n
                      7   38:12 2.2e+04  1.11 y    0.02
                              2.2e+04                  1.2e+04 5.9e+03 2.4e+00 n  n
                                                       1.0e+04 3.9e+03 2.4e+00 n  n
                      8   38:17 7.6e+03  2.52 n    0.01
                              7.6e+03                  5.5e+03 2.9e+03 1.2e+00 n  n
                                                       2.2e+03 1.2e+03 7.4e-01 n  n
                      9   38:21 1.1e+04  2.65 n    0.01
                              1.1e+04                  7.9e+03 3.7e+03 1.5e+00 n  n
                                                       3.0e+03 1.1e+03 7.1e-01 n  n
                      10  38:22 9.2e+03  2.08 n    0.01
                              9.2e+03                  [illegible] 2.6e+03 1.0e+00 n  n
                                                       [illegible] 1.1e+03 7.1e-01 n  n
                      11  38:37 1.7e+04  1.41 n    0.01
                              1.7e+04                  9.9e+03 3.2e+03 1.3e+00 n  n
                                                       7.0e+03 2.4e+03 1.5e+00 n  n
                      12  38:45 2.1e+04  1.17 y    0.02
                              2.1e+04                  1.1e+04 4.0e+03 1.6e+00 n  n
                                                       9.6e+03 3.2e+03 2.0e+00 n  n
                      13  38:51 1.5e+04  2.36 n    0.01
                              1.5e+04                  1.1e+04 5.0e+03 2.0e+00 n  n
                                                       4.5e+03 1.7e+03 1.1e+00 n  n
```


File:A26SEP98M #1-487 Acq:27-SEP-1998 02:17:51 GC EI+ Voltage SIR Autospec-UltimaE
Sample#13 Text:1114-1 x1/1 Exp:EXP_M23_DB5_OVATION
319.8965 S:13 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,3448.0,1.00%,F,F)
100%
50
0
26:12
26:39
27:01
27:53
28:15
28:44
29:18
29:57
3.2E5
1.6E5
0.0E0
25:00
26:00
27:00
28:00
29:00
30:00
Time
321.8936 S:13 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,2940.0,1.00%,F,F)
100%
50
0
26:12
26:38
27:01
27:52
28:15
28:44
29:27
29:57
4.4E5
2.2E5
0.0E0
25:00
26:00
27:00
28:00
29:00
30:00
Time
331.9368 S:13 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,17676.0,1.00%,F,F)
100%
50
0
29:09
29:26
2.5E7
1.2E7
0.0E0
25:00
26:00
27:00
28:00
29:00
30:00
Time
333.9339 S:13 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,6884.0,1.00%,F,F)
100%
50
0
29:09
29:26
3.1E7
1.5E7
0.0E0
25:00
26:00
27:00
28:00
29:00
30:00
Time
327.8847 S:13 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,10844.0,1.00%,F,F)
100%
50
0
29:27
4.8E7
2.4E7
0.0E0
25:00
26:00
27:00
28:00
29:00
30:00
Time
316.9824 S:13 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)
100%
50
0
24:32
25:18
25:44
26:16
26:43
27:08
27:34
28:24
28:54
29:22
30:05
30:30
1.0E8
5.0E7
0.0E0
25:00
26:00
27:00
28:00
29:00
30:00
Time

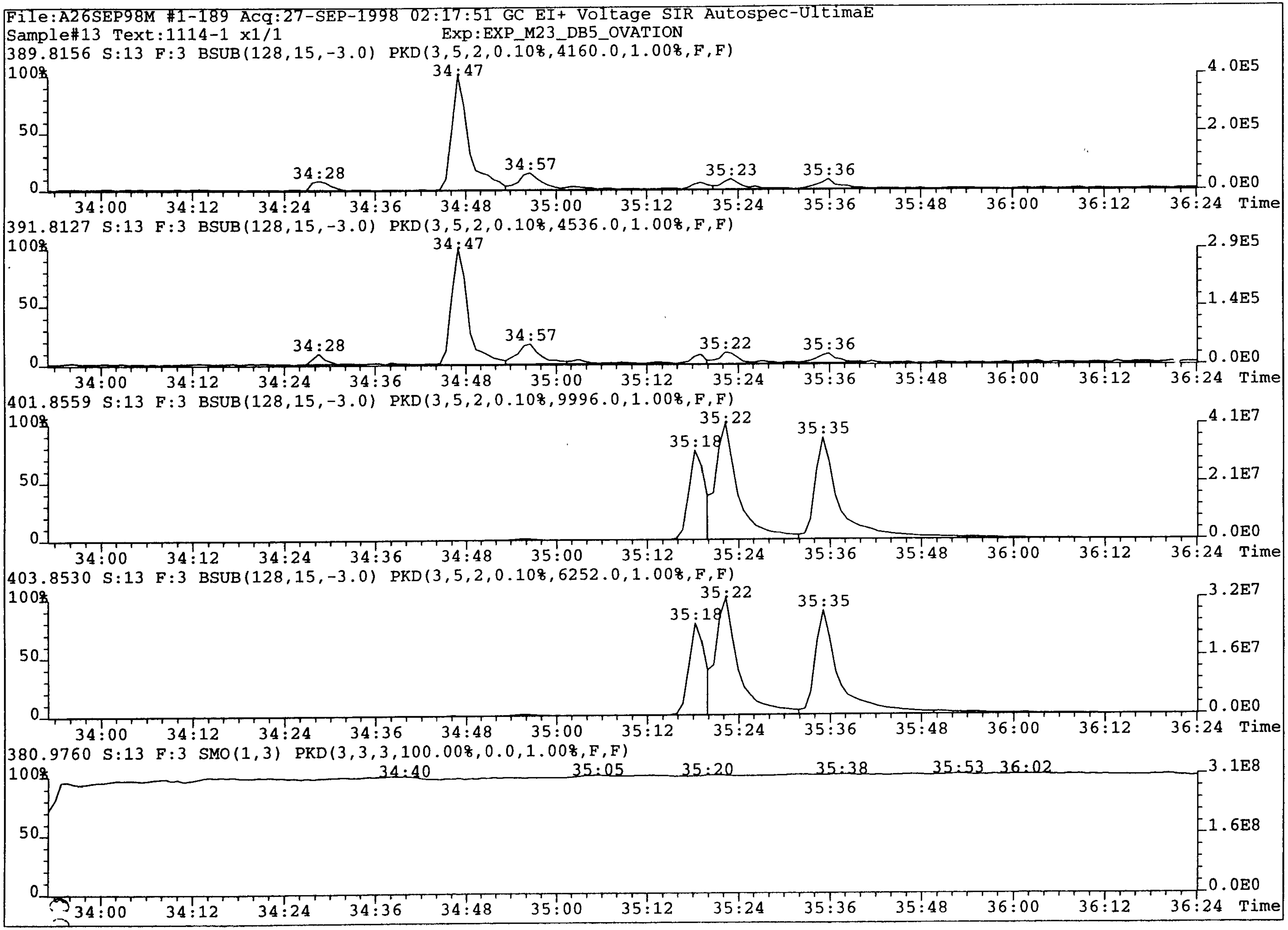


```
File:A26SEP98M #1-217 Acq:27-SEP-1998 02:17:51 GC EI+ Voltage SIR Autospec-UltimaE
Sample#13 Text:1114-1 x1/1                Exp:EXP_M23_DB5_OVATION
355.8546 S:13 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,4596.0,1.00%,F,F)
357.8517 S:13 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,2084.0,1.00%,F,F)
367.8949 S:13 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,4288.0,1.00%,F,F)
369.8919 S:13 F:2 BSUB(128,  -3.0) PKD(3,3,2,0.10%,2296.0,1.00%  ,F)
366.9792 S:13 F:2 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)
```

$$C = \frac{2.46\,10^5 * 4uf}{1.93\,10^8 * 1 * 1.3529} = 0.0038\ up.$$

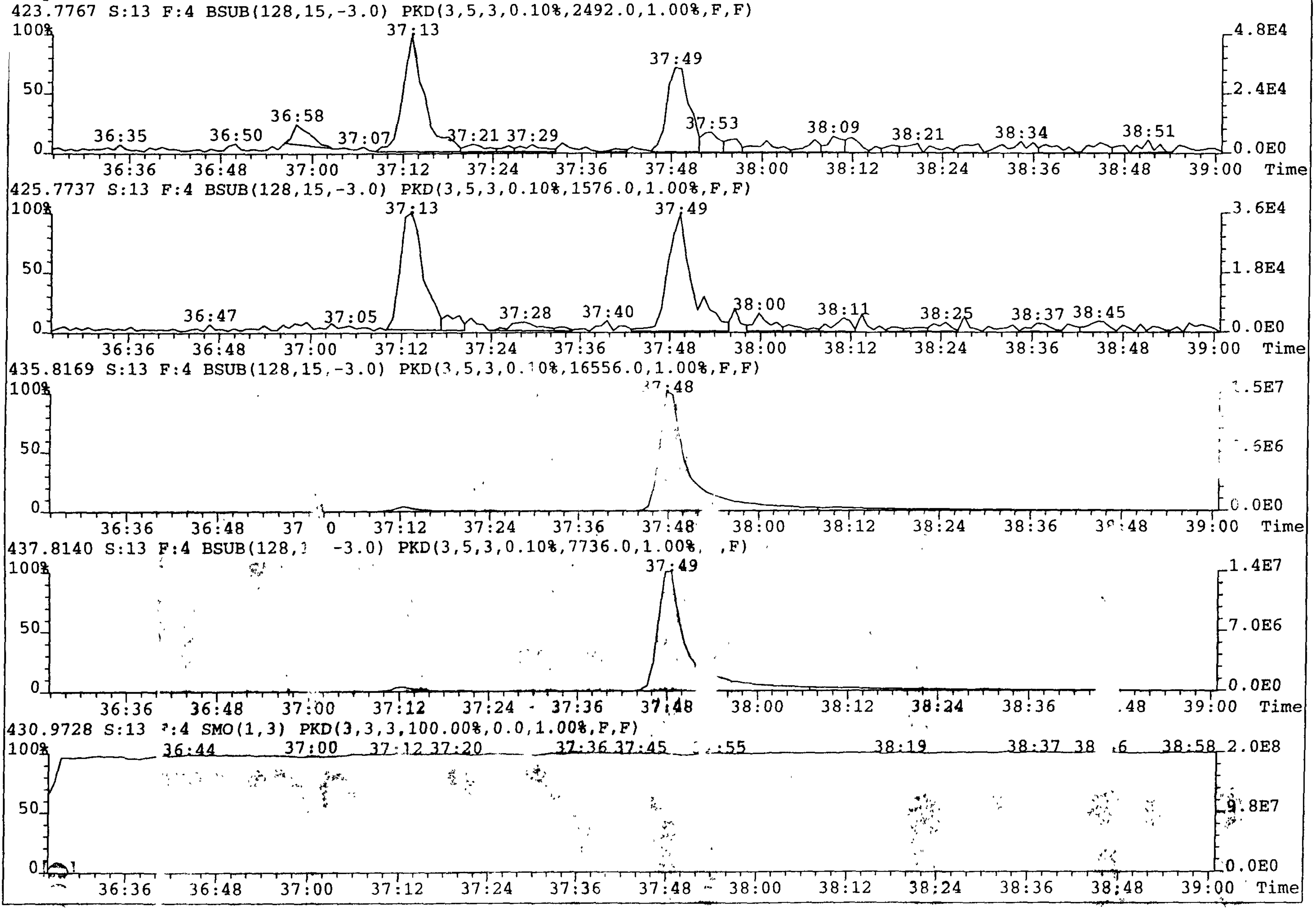
(BEC53)

✓



File:A26SEP98M #1-189 Acq:27-SEP-1998 02:17:51 GC EI+ Voltage SIR Autospec-UltimaE
Sample#13 Text:1114-1 x1/1 Exp:EXP_M23_DB5_OVATION

File:A26SEP98M #1-194 Acq:27-SEP-1998 02:17:51 GC EI+ Voltage SIR Autospec-UltimaE
Sample#13 Text:1114-1 x1/1 Exp:EXP_M23_DB5_OVATION


423.7767 S:13 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,2492.0,1.00%,F,F)

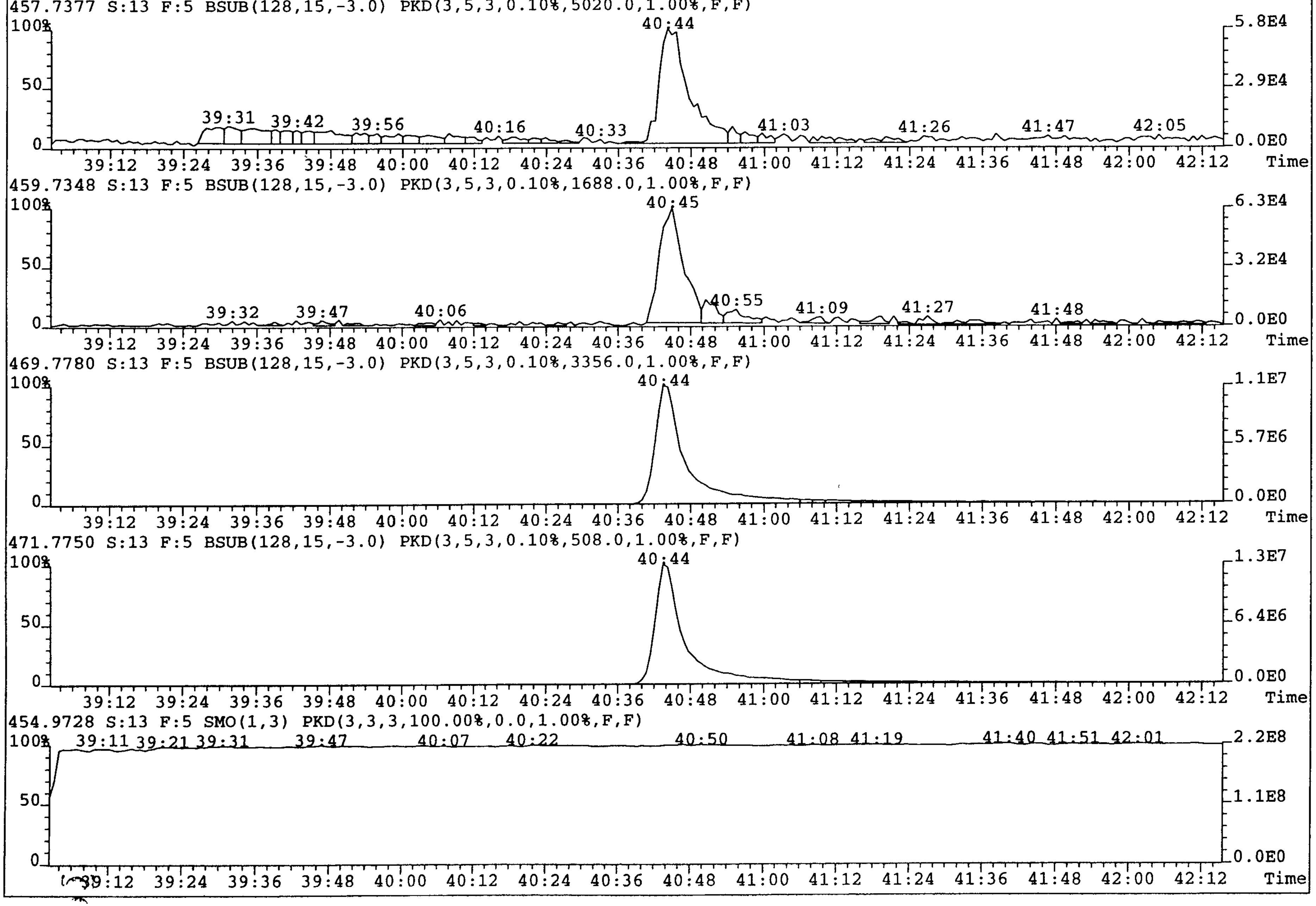
425.7737 S:13 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,1576.0,1.00%,F,F)

435.8169 S:13 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,16556.0,1.00%,F,F)

437.8140 S:13 F:4 BSUB(128,1 -3.0) PKD(3,5,3,0.10%,7736.0,1.00%, ,F)

430.9728 S:13 :4 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)

File:A26SEP98M #1-276 Acq:27-SEP-1998 02:17:51 GC EI+ Voltage SIR Autospec-UltimaE
Sample#13 Text:1114-1 x1/1 Exp:EXP_M23_DB5_OVATION



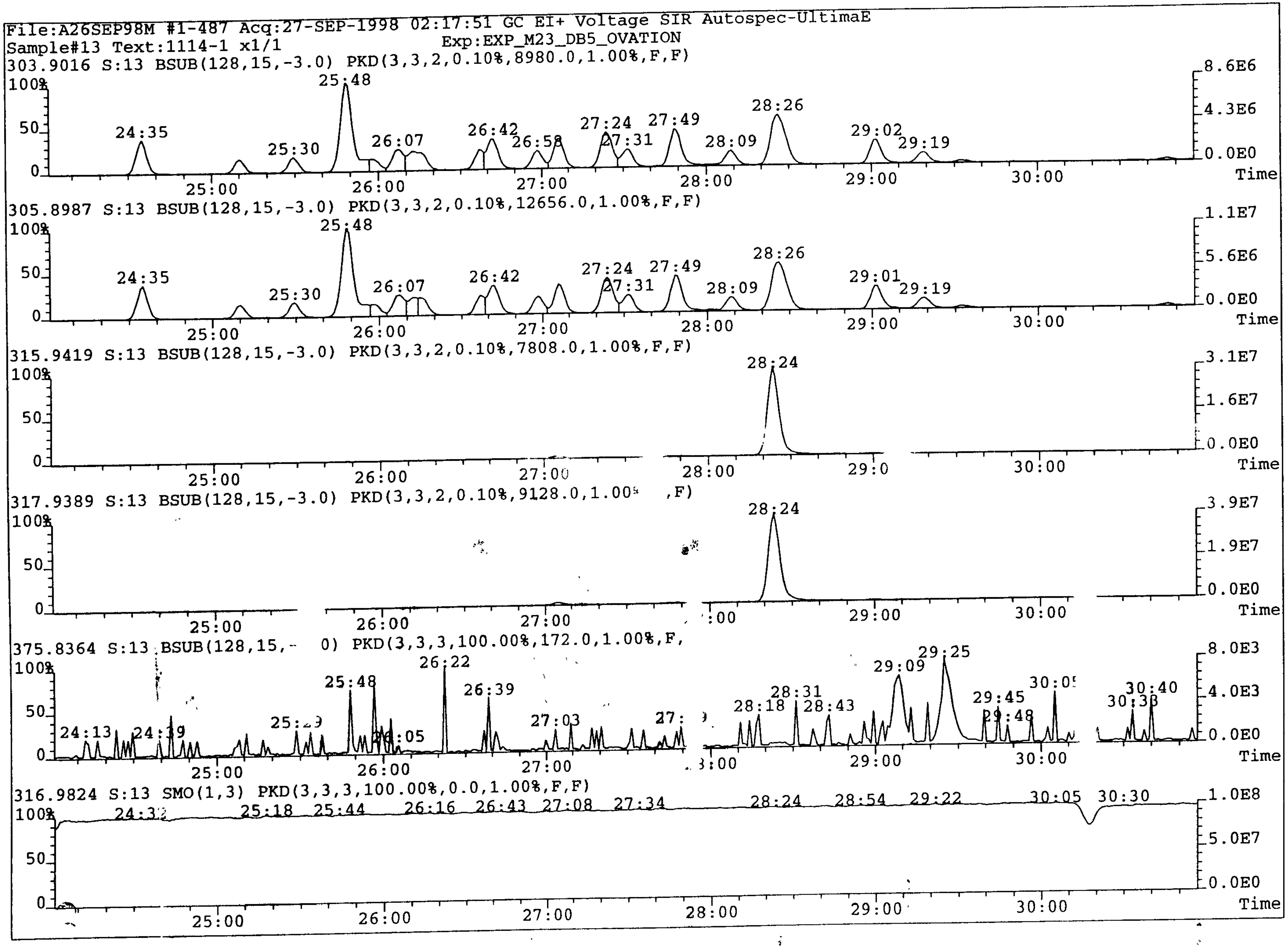
457.7377 S:13 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,5020.0,1.00%,F,F)

459.7348 S:13 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,1688.0,1.00%,F,F)

469.7780 S:13 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,3356.0,1.00%,F,F)

471.7750 S:13 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,508.0,1.00%,F,F)

454.9728 S:13 F:5 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)



File:A26SEP98M #1-487 Acq:27-SEP-1998 02:17:51 GC EI+ Voltage SIR Autospec-UltimaE

Sample#13 Text:1114-1 x1/1 Exp:EXP_M23_DB5_OVATION

303.9016 S:13 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,8980.0,1.00%,F,F)

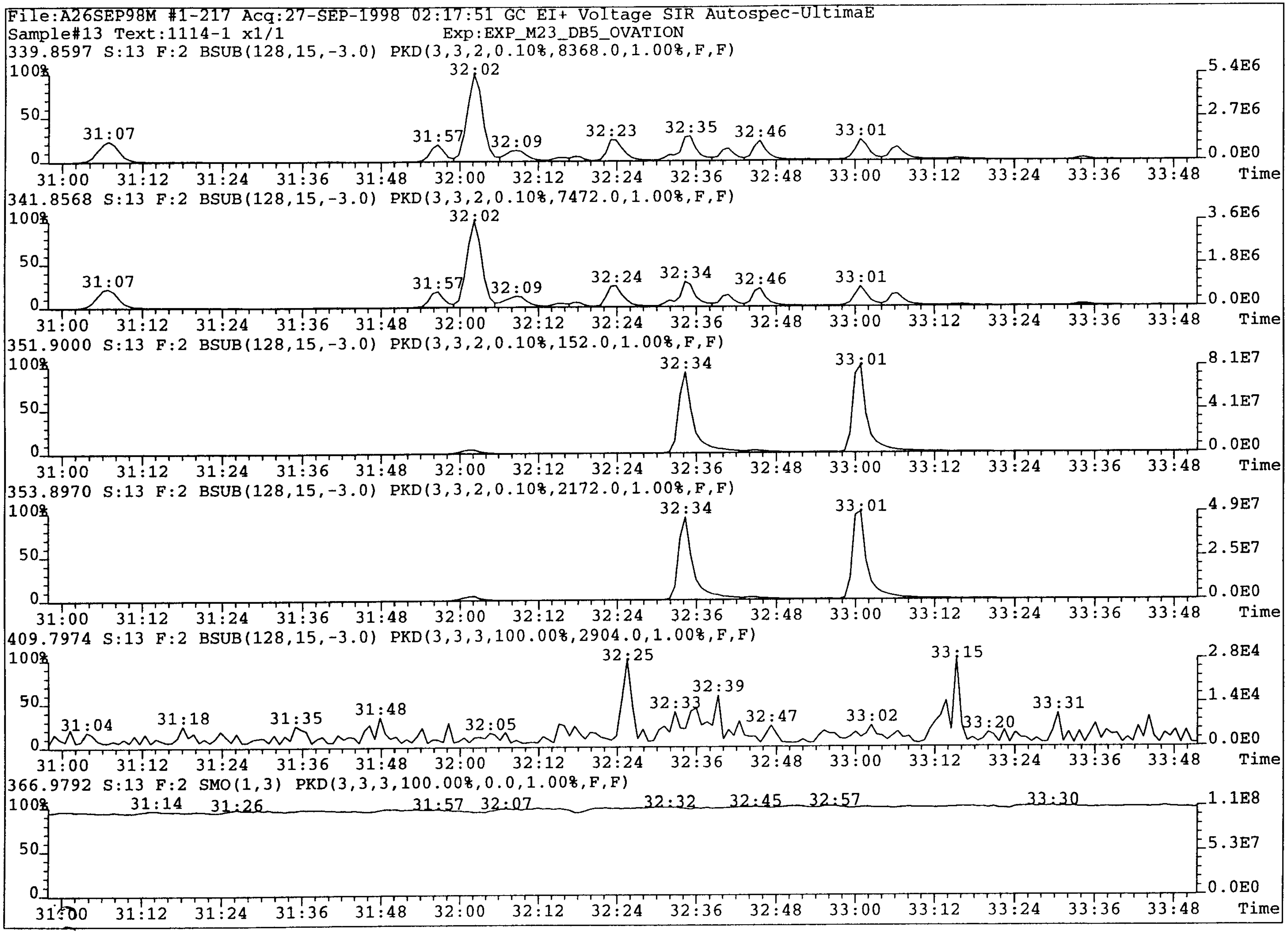
305.8987 S:13 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,12656.0,1.00%,F,F)

315.9419 S:13 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,7808.0,1.00%,F,F)

317.9389 S:13 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,9128.0,1.00% ,F)

375.8364 S:13 BSUB(128,15,- 0) PKD(3,3,3,100.00%,172.0,1.00%,F,

316.9824 S:13 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)



File:A26SEP98M #1-217 Acq:27-SEP-1998 02:17:51 GC EI+ Voltage SIR Autospec-UltimaE
Sample#13 Text:1114-1 x1/1 Exp:EXP_M23_DB5_OVATION

339.8597 S:13 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,8368.0,1.00%,F,F)

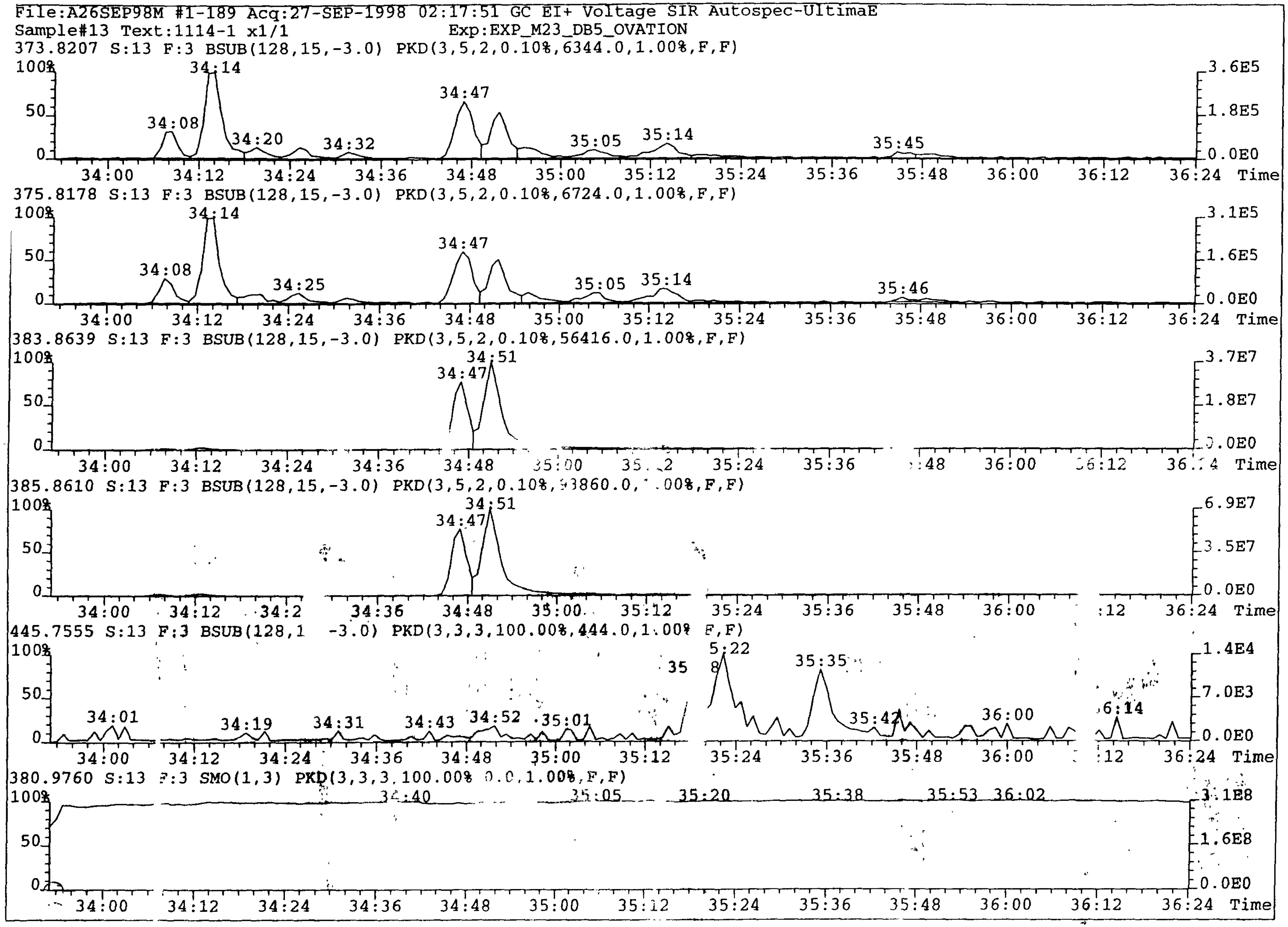
341.8568 S:13 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,7472.0,1.00%,F,F)

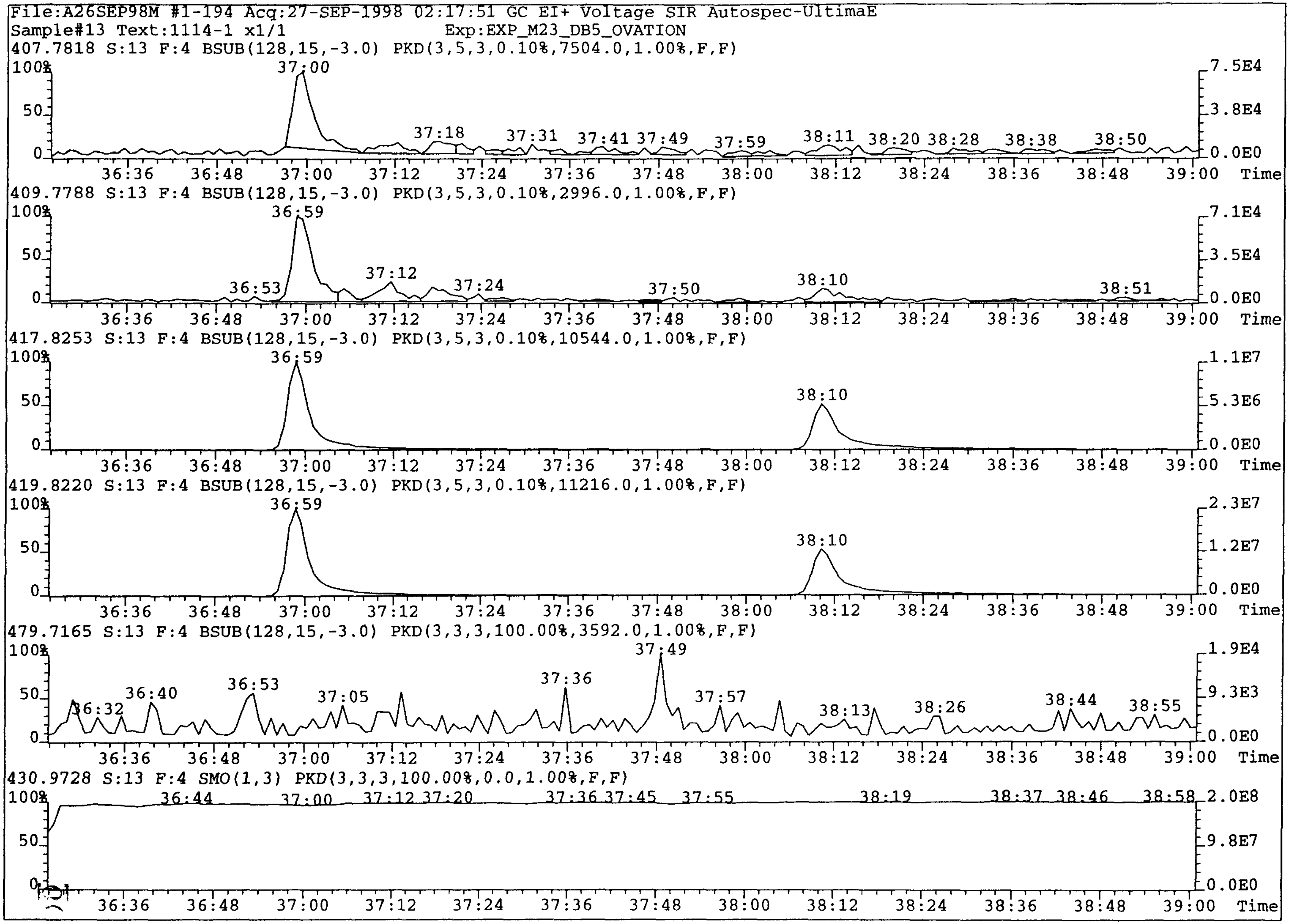
351.9000 S:13 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,152.0,1.00%,F,F)

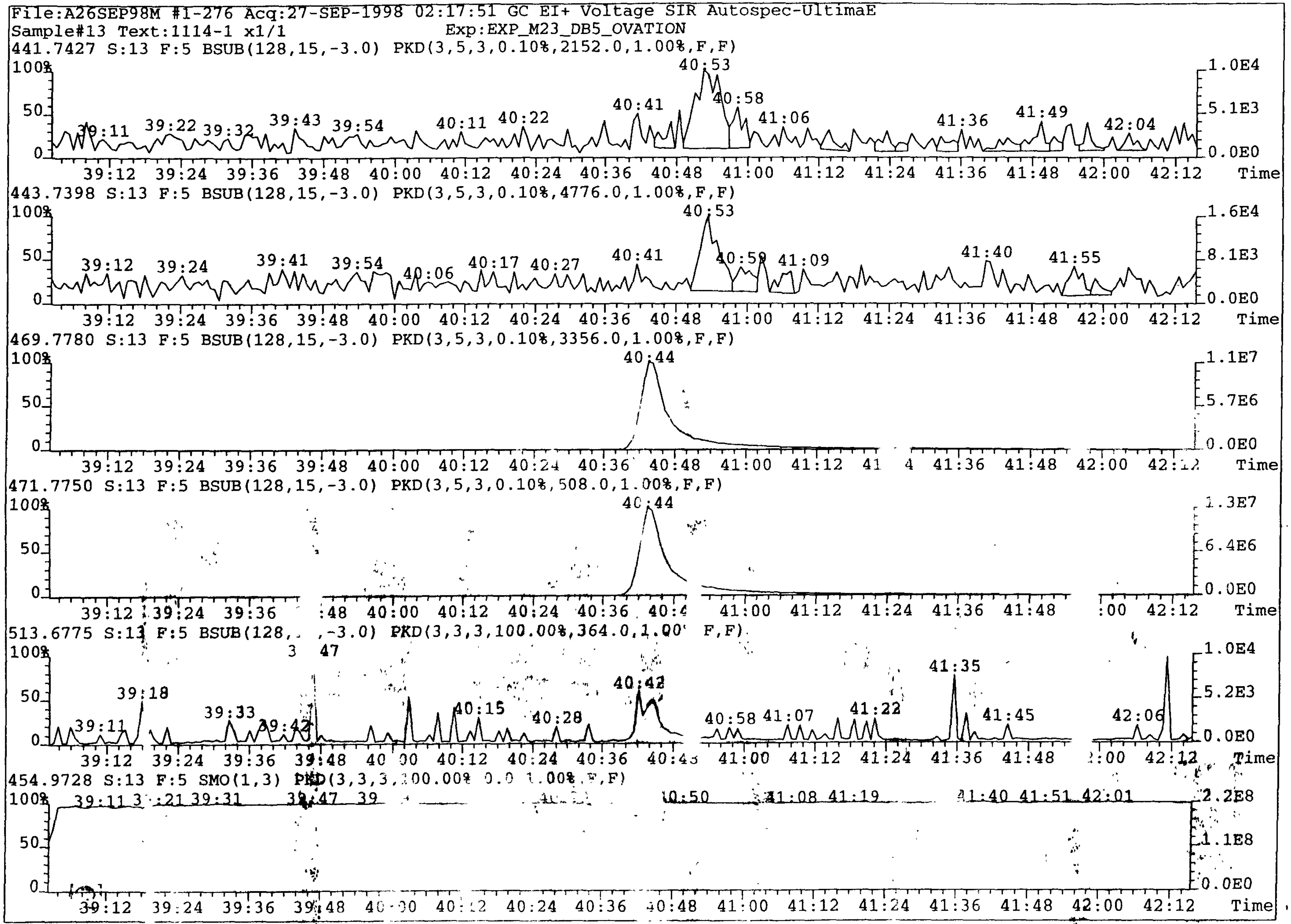
353.8970 S:13 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,2172.0,1.00%,F,F)

409.7974 S:13 F:2 BSUB(128,15,-3.0) PKD(3,3,3,100.00%,2904.0,1.00%,F,F)

366.9792 S:13 F:2 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)


File:A26SEP98M #1-189 Acq:27-SEP-1998 02:17:51 GC EI+ Voltage SIR Autospec-UltimaE
Sample#13 Text:1114-1 x1/1 Exp:EXP_M23_DB5_OVATION
373.8207 S:13 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,6344.0,1.00%,F,F)
100%
50
0
34:08
34:14
34:20
34:32
34:47
35:05
35:14
35:45
3.6E5
1.8E5
0.0E0
34:00 34:12 34:24 34:36 34:48 35:00 35:12 35:24 35:36 35:48 36:00 36:12 36:24 Time
375.8178 S:13 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,6724.0,1.00%,F,F)
34:08
34:14
34:25
34:47
35:05
35:14
35:46
3.1E5
1.6E5
0.0E0
383.8639 S:13 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,56416.0,1.00%,F,F)
34:47
34:51
3.7E7
1.8E7
0.0E0
385.8610 S:13 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,93860.0,1.00%,F,F)
34:47
34:51
6.9E7
3.5E7
0.0E0
445.7555 S:13 F:3 BSUB(128,1 -3.0) PKD(3,3,3,100.00%,444.0,1.00% F,F)
34:01
34:19
34:31
34:43
34:52
35:01
35
5:22
35:35
35:42
36:00
6:14
1.4E4
7.0E3
0.0E0
380.9760 S:13 F:3 SMO(1,3) PKD(3,3,3,100.00% 0.0,1.00%,F,F)
34:40
35:05
35:20
35:38
35:53
36:02
3.1E8
1.6E8
0.0E0


File:A26SEP98M #1-194 Acq:27-SEP-1998 02:17:51 GC EI+ Voltage SIR Autospec-UltimaE
Sample#13 Text:1114-1 x1/1 Exp:EXP_M23_DB5_OVATION
407.7818 S:13 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,7504.0,1.00%,F,F)
100%
50
0
37:00
37:18
37:31
37:41
37:49
37:59
38:11
38:20
38:28
38:38
38:50
7.5E4
3.8E4
0.0E0
36:36
36:48
37:00
37:12
37:24
37:36
37:48
38:00
38:12
38:24
38:36
38:48
39:00
Time
409.7788 S:13 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,2996.0,1.00%,F,F)
100%
50
0
36:59
36:53
37:12
37:24
37:50
38:10
38:51
7.1E4
3.5E4
0.0E0
36:36
36:48
37:00
37:12
37:24
37:36
37:48
38:00
38:12
38:24
38:36
38:48
39:00
Time
417.8253 S:13 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,10544.0,1.00%,F,F)
100%
50
0
36:59
38:10
1.1E7
5.3E6
0.0E0
36:36
36:48
37:00
37:12
37:24
37:36
37:48
38:00
38:12
38:24
38:36
38:48
39:00
Time
419.8220 S:13 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,11216.0,1.00%,F,F)
100%
50
0
36:59
38:10
2.3E7
1.2E7
0.0E0
36:36
36:48
37:00
37:12
37:24
37:36
37:48
38:00
38:12
38:24
38:36
38:48
39:00
Time
479.7165 S:13 F:4 BSUB(128,15,-3.0) PKD(3,3,3,100.00%,3592.0,1.00%,F,F)
100%
50
0
36:32
36:40
36:53
37:05
37:36
37:49
37:57
38:13
38:26
38:44
38:55
1.9E4
9.3E3
0.0E0
36:36
36:48
37:00
37:12
37:24
37:36
37:48
38:00
38:12
38:24
38:36
38:48
39:00
Time
430.9728 S:13 F:4 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)
100%
50
0
36:44
37:00
37:12
37:20
37:36
37:45
37:55
38:19
38:37
38:46
38:58
2.0E8
9.8E7
0.0E0
36:36
36:48
37:00
37:12
37:24
37:36
37:48
38:00
38:12
38:24
38:36
38:48
39:00
Time



File:A26SEP98M #1-276 Acq:27-SEP-1998 02:17:51 GC EI+ Voltage SIR Autospec-UltimaE
Sample#13 Text:1114-1 x1/1 Exp:EXP_M23_DB5_OVATION

441.7427 S:13 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,2152.0,1.00%,F,F)

443.7398 S:13 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,4776.0,1.00%,F,F)

469.7780 S:13 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,3356.0,1.00%,F,F)

471.7750 S:13 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,508.0,1.00%,F,F)

513.6775 S:13 F:5 BSUB(128, ,-3.0) PKD(3,3,3,100.00%,364.0,1.00 F,F)

454.9728 S:13 F:5 SMO(1,3) PKD(3,3,3,100.00% 0.0 1.00%,F,F)

```
OPUSquan    30-SEP-1998                Page 1

         Filename ; a29sep98n
           Sample ; 7
         Acquired ; 29-SEP-98  21:24:44
        Processed ; 30-SEP-98  08:57:40
        Sample ID ; 1114-1
        Cal Table ; 07feb-m23conf
    Results Table ; m8290cf-092998n
         Comments ;
Typ  ;                  #  Name;      Resp;     Ion 1;     Ion 2;  RA;?;     RT;       Conc;       DL;      S/N1;?;      S/N2;? ; mod?
Unk  ;              2,3,7,8-TCDF; 9.54e+06; 4.22e+06; 5.32e+06; 0.79;y; 27:52;      7.733;   0.1454;        213;y;        142;y ;  yes
ES/RT;          13C-2,3,7,8-TCDF; 1.30e+08; 5.73e+07; 7.25e+07; 0.79;y; 27:50;     31.809;        -;       1454;y;       1409;y ;   no
Total;              Tetra Furans; 1.78e+08; 4.78e+06; 6.06e+06; 0.79;y; 18:08;    144.386;   0.1454;        393;y;        270;y ;  yes
DPE  ;                 #  HxCDPE;        *;        *                    ;NotFnd;          *;        -;          *;n                ;   no
LMC  ;        QC CHK ION (Tetra);        *;        *                    ;NotFnd;          *;        -;     Div0;n                ;   no




                                     ;         -;     -;-; 27:52




                                     ;         -;     -;-; 27:52                                        ;        -; -;   yes
```

see m.t.

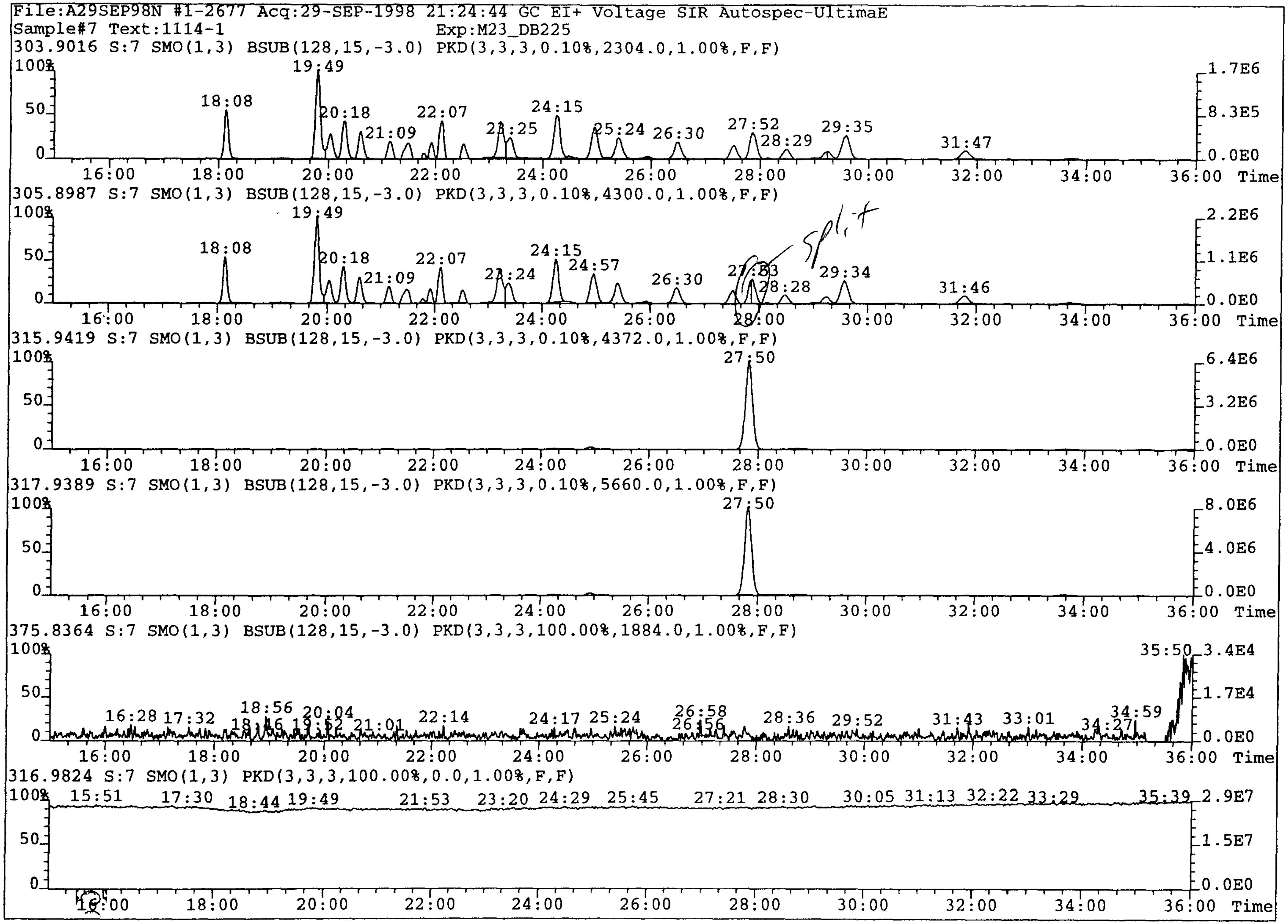
may be split.

```
        Filename ; a29sep98n
          Sample ; 7
        Acquired ; 29-SEP-98  21:24:44
       Processed ; 30-SEP-98  08:57:40
       Sample ID ; 1114-1
       Cal Table ; 07feb-m23conf
   Results Table ; m8290cf-092998n
        Comments ;
Typ  ;                  Name;      Resp;     Ion 1;     Ion 2;   RA;?;      RT;      Conc;        DL;     S/N1;?;      S/N2;? ; mod?
Unk  ;           2,3,7,8-TCDF; 7.26e+06; 4.22e+06; 3.04e+06; 1.39;n; 27:52;     5.884;    0.1454;      213;y;       142;y ;   no
ES/RT;       13C-2,3,7,8-TCDF; 1.30e+08; 5.73e+07; 7.25e+07; 0.79;y; 27:50;    31.809;        -;     1454;y;      1409;y ;   no
Total;           Tetra Furans; 1.76e+08; 4.78e+06; 6.06e+06; 0.79;y; 18:08;   142.507;    0.1454;      393;y;       270;y ;   no
DPE  ;                 HxCDPE;        *;        *                 ;NotFnd;         *;        -;        *;n                ;   no
LMC  ;      QC CHK ION (Tetra);        *;        *                 ;NotFnd;         *;        -;     Div0;n                ;   no




                                                  ;       -;     -  ; 27:52


                                                  ;       -;     -;-; 27:52                                 ;       -; -;   no
```



File:A29SEP98N #1-2677 Acq:29-SEP-1998 21:24:44 GC EI+ Voltage SIR Autospec-UltimaE
Sample#7 Text:1114-1 Exp:M23_DB225

303.9016 S:7 SMO(1,3) BSUB(128,15,-3.0) PKD(3,3,3,0.10%,2304.0,1.00%,F,F)

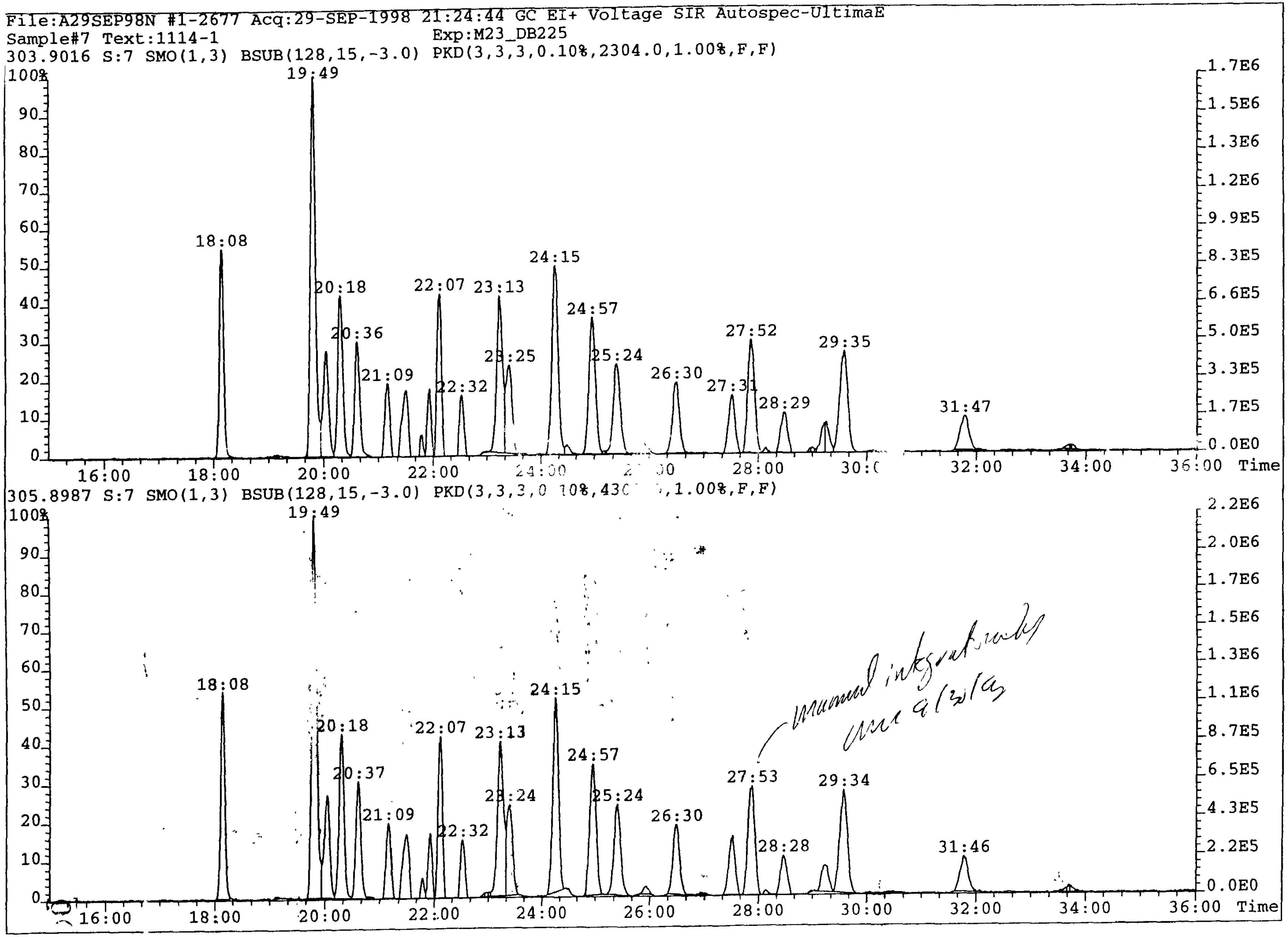
305.8987 S:7 SMO(1,3) BSUB(128,15,-3.0) PKD(3,3,3,0.10%,4300.0,1.00%,F,F)

315.9419 S:7 SMO(1,3) BSUB(128,15,-3.0) PKD(3,3,3,0.10%,4372.0,1.00%,F,F)

317.9389 S:7 SMO(1,3) BSUB(128,15,-3.0) PKD(3,3,3,0.10%,5660.0,1.00%,F,F)

375.8364 S:7 SMO(1,3) BSUB(128,15,-3.0) PKD(3,3,3,100.00%,1884.0,1.00%,F,F)

316.9824 S:7 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)



File:A29SEP98N #1-2677 Acq:29-SEP-1998 21:24:44 GC EI+ Voltage SIR Autospec-UltimaE
Sample#7 Text:1114-1 Exp:M23_DB225
303.9016 S:7 SMO(1,3) BSUB(128,15,-3.0) PKD(3,3,3,0.10%,2304.0,1.00%,F,F)

305.8987 S:7 SMO(1,3) BSUB(128,15,-3.0) PKD(3,3,3,0 10%,430[illegible],1.00%,F,F)

manual integrations
9/30/98

*Method 23*

**M23-FB-3**

PES

## Analytical Data Summary Sheet

| Analyte | Concentration (ng) | DL (ng) | EMPC (ng) | RT (min.) | Ratio | Qualifier |
|---|---|---|---|---|---|---|
| 2,3,7,8-TCDD | ND | 0.0012 | | 29:27 | 0.37 | |
| 1,2,3,7,8-PeCDD | ND | 0.0007 | | 33:14 | 1.09 | |
| 1,2,3,4,7,8-HxCDD | ND | 0.0013 | | 35:19 | 1.59 | |
| 1,2,3,6,7,8-HxCDD | EMPC | 0.0010 | 0.0013 | 35:23 | 0.94 | |
| 1,2,3,7,8,9-HxCDD | ND | 0.0010 | | 35:35 | 0.73 | |
| 1,2,3,4,6,7,8-HpCDD | EMPC | 0.0014 | 0.0034 | 37:50 | 1.33 | |
| OCDD | EMPC | 0.0087 | 0.0092 | 40:44 | 0.64 | |
| 2,3,7,8--TCDF | ND | 0.0015 | | 28:25 | 2.3 | |
| 1,2,3,7,8-PeCDF | ND | 0.0008 | | 32:34 | 2.12 | |
| 2,3,4,7,8-PeCDF | ND | 0.0007 | | 33:01 | 1.49 | |
| 1,2,3,4,7,8-HxCDF | 0.0022 | 0.0012 | | 34:47 | 1.13 | |
| 1,2,3,6,7,8-HxCDF | EMPC | 0.0009 | 0.0015 | 34:52 | 1.61 | |
| 2,3,4,6,7,8-HxCDF | ND | 0.0011 | | 35:14 | 1.24 | |
| 1,2,3,7,8,9-HxCDF | ND | 0.0013 | | 35:45 | 1.04 | |
| 1,2,3,4,6,7,8-HpCDF | ND | 0.0022 | | 37:01 | 0.61 | |
| 1,2,3,4,7,8,9-HpCDF | ND | 0.0028 | | 38:11 | 1.69 | |
| OCDF | ND | 0.0047 | | 40:54 | 0.55 | |
| Total TCDDs | ND | 0.0012 | | | | |
| Total PeCDDs | ND | 0.0007 | 0.0008 | | | |
| Total HxCDDs | 0.0016 | 0.0010 | | | | |
| Total HpCDDs | ND | 0.0014 | 0.0056 | | | |
| Total TCDFs | ND | 0.0015 | | | | |
| Total PeCDFs | ND | 0.0007 | | | | |
| Total HxCDFs | 0.0024 | 0.0009 | 0.0040 | | | |
| Total HpCDFs | ND | 0.0022 | | | | |
| TEQ (ND=0) | 0.0002 | | 0.0005 | | | ITEF |
| TEQ (ND=1/2) | 0.0017 | | 0.0019 | | | ITEF |

### Client Information

| | |
|---|---|
| Project Name: | S509.000 |
| Sample ID: | M23-FB-3 |

### Laboratory Information

| | |
|---|---|
| Project ID: | L1114 |
| Sample ID: | 1114-2 |
| Collection Date: | 31-Aug-98 |
| Receipt Date: | 08-Sep-98 |
| Extraction Date: | 16-Sep-98 |
| Analysis Date: | 27-Sep-98 |

### Sample Information

| | | |
|---|---|---|
| Matrix: | Air | |
| Weight / Volume: | 1 | |
| Moisture / Lipids: | 0.0 | % |

| | |
|---|---|
| Filename: | a26sep98m-14 |
| Retchk: | a26sep98m-1 |
| Begin ConCal: | a26sep98m-5 |
| End ConCal: | a26sep98m-21 |
| Initial_Cal: | a26sep98m-21 |

*Method 23*

**M23-FB-3**

PES

## Analytical Data Summary Sheet

| Labeled Standard | Expected Amount (ng) | Measured Amount (ng) | Percent Recovery (%) | RT (min.) | Ratio | Qualifier |
|---|---|---|---|---|---|---|
| **Extraction Standards** | | | | | | |
| $^{13}C_{12}$-2,3,7,8-TCDD | 4 | 3.25 | 81.3 | 29:25 | 0.8 | |
| $^{13}C_{12}$-1,2,3,7,8-PeCDD | 4 | 4.07 | 101.7 | 33:13 | 1.61 | |
| $^{13}C_{12}$-1,2,3,6,7,8-HxCDD | 4 | 3.85 | 96.2 | 35:22 | 1.27 | |
| $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD | 4 | 3.05 | 76.3 | 37:48 | 1.08 | |
| $^{13}C_{12}$-OCDD | 8 | 4.93 | 61.6 | 40:44 | 0.93 | |
| $^{13}C_{12}$-2,3,7,8-TCDF | 4 | 2.96 | 74.0 | 28:23 | 0.79 | |
| $^{13}C_{12}$-1,2,3,7,8-PeCDF | 4 | [illegible].91 | 72.7 | 32:34 | 1.58 | |
| $^{13}C_{12}$-1,2,3,6,7,8-HxCDF | 4 | 2.42 | 60.6 | 34:47 | 0.53 | |
| $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF | 4 | 2.17 | 54.3 | 36:59 | 0.46 | |
| **Sampling Standards** | | | | | | |
| $^{37}Cl_4$-2,3,7,8-TCDD | 4 | 3.98 | 99.5 | 29:27 | | |
| $^{13}C_{12}$-2,3,4,7,8-PeCDF | 4 | 4.26 | 106.4 | 33:00 | 1.58 | |
| $^{13}C_{12}$-1,2,3,4,7,8-HxCDD | 4 | 3.16 | [illegible] | [illegible] | 1.27 | |
| $^{13}C_{12}$-1,2,3,4,7,8-HxCDF | 4 | 5.88 | 147.1 | 34:51 | 0.53 | |
| $^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF | 4 | 3.16 | 78.9 | 38:10 | [illegible] | |
| **Injection Standards** | | | | | | |
| $^{13}C_{12}$-1,2,3,4-TCDD | | | | 29:08 | [illegible] | |
| $^{13}C_{12}$-1,2,3,7,8,9-HxCDD | | | | 35:35 | [illegible] | |

**Client Information**

| | |
|---|---|
| Project Name: | S509.000 |
| Sample ID: | M23-FB-3 |

**Laboratory Information**

| | |
|---|---|
| Project ID: | L1114 |
| Sample ID: | 1114-2 |
| Collection Date: | 31-Aug-98 |
| Receipt Date: | 08-Sep-98 |
| Extraction Date: | 16-Sep-98 |
| Analysis Date: | 27-Sep-98 |

**Sample Information**

| | | |
|---|---|---|
| Matrix: | Air | |
| Weight / Volume: | 1 | |
| Moisture / Lipids: | 0.0 | % |
| Filename: | a26sep98m-14 | |
| Retchk: | a26sep98m-1 | |
| Begin ConCal: | a26sep98m-5 | |
| End ConCal: | a26sep98m-21 | |
| Initial Cal: | a26sep98m-21 | |

Reviewed by: 1.T.

Date Reviewed: 30 Sep 98

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OPUSquan    30-SEP-1998                Page 1

          Filename ; a26sep98m
            Sample ; 14
          Acquired ; 27-SEP-98  03:05:54
         Processed ; 28-SEP-98  12:06:42
         Sample ID ; 1114-2 x1/1
         Cal Table ; m8290-092698m
     Results Table ; M8290-092698M-BE
          Comments ;
Typ  ;                    Name;     Resp;    Ion 1;    Ion 2;   RA;?;    RT;      Conc;       DL;    S/N1;?;     S/N2;? ; mod?
Unk  ;            2,3,7,8-TCDD; 2.35e+05; 2.23e+04; 2.12e+05; 0.11;n; 29:27;     0.085;   0.0306;       3;n;       16;y ;   no
Unk  ;         1,2,3,7,8-PeCDD; 4.44e+04; 2.31e+04; 2.13e+04; 1.09;n; 33:14;     0.016;   0.0166;       3;y;        6;y ;   no
Unk  ;       1,2,3,4,7,8-HxCDD; 3.48e+04; 2.13e+04; 1.34e+04; 1.59;n; 35:19;     0.018;   0.0322;       3;y;        3;n ;   no
Unk  ;       1,2,3,6,7,8-HxCDD; 8.34e+04; 4.03e+04; 4.31e+04; 0.94;n; 35:23;     0.032;   0.0240;       5;y;        3;y ;   no
Unk  ;       1,2,3,7,8,9-HxCDD; 6.22e+04; 2.63e+04; 3.59e+04; 0.73;n; 35:35;     0.025;   0.0256;       4;y;        3;y ;   no
Unk  ;     1,2,3,4,6,7,8-HpCDD; 1.17e+05; 6.68e+04; 5.03e+04; 1.33;n; 37:50;     0.084;   0.0360;       8;y;       10;y ;   no
Unk  ;                    OCDD; 1.77e+05; 6.92e+04; 1.08e+05; 0.64;n; 40:44;     0.231;   0.2179;       3;y;       14;y ;   no

Unk  ;            2,3,7,8-TCDF; 3.47e+04; 2.42e+04; 1.05e+04; 2.30;n; 28:25;     0.010;   0.0382;       3;y;        1;n ;   no
Unk  ;         1,2,3,7,8-PeCDF; 8.79e+04; 5.98e+04; 2.82e+04; 2.12;n; 32:34;     0.031;   0.0207;      14;y;        2;n ;   no
Unk  ;         2,3,4,7,8-PeCDF; 9.34e+04; 5.58e+04; 3.75e+04; 1.49;y; 33:01;     0.030;   0.0186;      16;y;        3;n ;   no
Unk  ;       1,2,3,4,7,8-HxCDF; 1.06e+05; 5.64e+04; 4.99e+04; 1.13;y; 34:47;     0.056;   0.0310;       4;y;        6;y ;   no
Unk  ;       1,2,3,6,7,8-HxCDF; 9.68e+04; 5.98e+04; 3.71e+04; 1.61;n; 34:52;     0.037;   0.0224;       5;y;        4;y ;   no
Unk  ;       2,3,4,6,7,8-HxCDF; 7.17e+04; 3.97e+04; 3.20e+04; 1.24;y; 35:14;     0.033;   0.0273;       2;n;        3;n ;   no
Unk  ;       1,2,3,7,8,9-HxCDF; 6.34e+04; 3.23e+04; 3.12e+04; 1.04;n; 35:45;     0.035;   0.0327;       2;n;        3;y ;   no
Unk  ;     1,2,3,4,6,7,8-HpCDF; 1.17e+05; 4.45e+04; 7.27e+04; 0.61;n; 37:01;     0.076;   0.0556;       3;n;        8;y ;   no
Unk  ;     1,2,3,4,7,8,9-HpCDF; 4.49e+04; 2.82e+04; 1.67e+04; 1.69;n; 38:11;     0.037;   0.0705;       1;n;        2;n ;   no
Unk  ;                    OCDF; 5.66e+04; 2.01e+04; 3.65e+04; 0.55;n; 40:54;     0.065;   0.1179;       4;y;        2;n ;   no

ES/RT;        13C-2,3,7,8-TCDD; 2.40e+08; 1.06e+08; 1.34e+08; 0.80;y; 29:25;    81.268;   0.0942;    1431;y;     4053;y ;   no
ES   ;     13C-1,2,3,7,8-PeCDD; 2.04e+08; 1.26e+08; 7.82e+07; 1.61;y; 33:13;   101.704;   0.0715;    6641;y;     9632;y ;   no
ES   ;   13C-1,2,3,6,7,8-HxCDD; 2.67e+08; 1.50e+08; 1.18e+08; 1.27;y; 35:22;    96.145;   0.0770;    3962;y;     3914;y ;   no
ES   ;13C-1,2,3,4,6,7,8-HpCDD; 1.42e+08; 7.36e+07; 6.80e+07; 1.08;y; 37:48;    76.315;   0.1585;     856;y;     1901;y ;   no
ES   ;               13C-OCDD; 1.42e+08; 6.85e+07; 7.35e+07; 0.93;y; 40:44;   123.245;   0.0409;    3085;y;    28588;y ;   no

ES/RT;        13C-2,3,7,8-TCDF; 3.28e+08; 1.45e+08; 1.83e+08; 0.79;y; 28:23;    74.008;   0.0428;    3805;y;     5908;y ;   no
ES   ;     13C-1,2,3,7,8-PeCDF; 2.90e+08; 1.78e+08; 1.12e+08; 1.58;y; 32:34;    72.647;   0.0150;   51326;y;    14046;y ;   no
ES   ;   13C-1,2,3,6,7,8-HxCDF; 1.53e+08; 5.27e+07; 1.00e+08; 0.53;y; 34:47;    60.591;   0.9026;     381;y;      289;y ;   no
ES   ;13C-1,2,3,4,6,7,8-HpCDF; 1.12e+08; 3.52e+07; 7.70e+07; 0.46;y; 36:59;    54.315;   0.1027;    1215;y;     2018;y ;   no

JS   ;        13C-1,2,3,4-TCDD; 2.86e+08; 1.28e+08; 1.58e+08; 0.81;y; 29:08;    58.661;        -;    2009;y;     5486;y ;   no
JS   ;   13C-1,2,3,7,8,9-HxCDD; 2.49e+08; 1.40e+08; 1.10e+08; 1.28;y; 35:35;    59.320;        -;    3525;y;     3454;y ;   no

CS   ;       37Cl-2,3,7,8-TCDD; 2.47e+08; 2.47e+08;        -;   -;-; 29:27;    80.816;   0.0414;    5193;y;       -; -;   no
CS   ;     13C-2,3,4,7,8-PeCDF; 3.23e+08; 1.98e+08; 1.25e+08; 1.58;y; 33:00;    77.307;   0.0143;   57315;y;    16406;y ;   no
CS   ;   13C-1,2,3,4,7,8-HxCDD; 1.25e+08; 7.00e+07; 5.53e+07; 1.27;y; 35:18;    75.842;   0.1296;    2899;y;     2851;y ;   no
CS   ;   13C-1,2,3,4,7,8-HxCDF; 3.25e+08; 1.12e+08; 2.13e+08; 0.53;y; 34:51;    89.113;   0.6231;     586;y;      446;y ;   no
CS   ;13C-1,2,3,4,7,8,9-HpCDF; 6.79e+07; 2.02e+07; 4.78e+07; 0.42;y; 38:10;    42.866;   0.1338;     561;y;      974;y ;   no

SS   ;       37Cl-2,3,7,8-TCDD; 2.47e+08; 2.47e+08;        -;   -;-; 29:27;    99.444;   0.0588;    5193;y;       -; -;   no
SS   ;     13C-2,3,4,7,8-PeCDF; 3.23e+08; 1.98e+08; 1.25e+08; 1.58;y; 33:00;   106.414;   0.0113;   57315;y;    16406;y ;   no
SS   ;   13C-1,2,3,4,7,8-HxCDD; 1.25e+08; 7.00e+07; 5.53e+07; 1.27;y; 35:18;    78.883;   0.1281;    2899;y;     2851;y ;   no
SS   ;   13C-1,2,3,4,7,8-HxCDF; 3.25e+08; 1.12e+08; 2.13e+08; 0.53;y; 34:51;   147.074;   0.6565;     586;y;      446;y ;   no
SS   ;13C-1,2,3,4,7,8,9-HpCDF; 6.79e+07; 2.02e+07; 4.78e+07; 0.42;y; 38:10;    78.921;   0.2343;     561;y;      974;y ;   no
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                                                                    Page 1 of 8

 Ent: 39 Name: Total Tetra-Furans  F:1  Mass: 303.902 305.899 Mod? no    #Hom:4

 Run: 7  File: a26sep98m    S:14 Acq:27-SEP-98 03:05:54 Proc:28-SEP-98 12:06:42
 Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
 Version: V3.6 31-JUL-1998 10:51:59  Sample text: 1114-2 x1/1

   Amount: 0.03     of which 0.01     named and 0.02     unnamed
     Conc: 0.03     of which 0.01     named and 0.02     unnamed
   Tox #1: -            Tox #2: -           Tox #3: -

Name                  #      RT Respnse      RA     Conc      Area  Height     S/N  Mod?

                      1   27:49 2.5e+04  1.31 n     0.01
                                2.5e+04                   1.4e+04 5.6e+03 2.0e+00 n  n
                                                          1.1e+04 7.6e+03 1.2e+00 n  n
                      2   27:52 1.5e+04  2.06 n     0.00
                                1.5e+04                   1.0e+04 3.6e+03 1.3e+00 n  n
                                                          4.9e+03 2.3e+03 3.6e-01 n  n
2,3,7,8-TCDF          3   28:25 3.5e+04  2.30 n     0.01
                                3.5e+04                   2.4e+04 9.4e+03 3.3e+00 y  n
                                                          1.1e+04 6.3e+03 9.9e-01 n  n
                      4   28:27 2.8e+04  1.63 n
                                2.8e+04                   1.7e+04 8.0e+03 2.8e+00 n  n
                                                          1.1e+04 6.3e+03 9.9e-01 n  n


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 Ent: 40 Name: Total Tetra-Dioxins F:1  Mass: 319.897 321.894 Mod? no    #Hom:6

 Run: 7  File: a26sep98m    S:14 Acq:27-SEP-98 03:05:54 Proc:28-SEP-98 12:06:42
 Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
 Version: V3.6 31-JUL-1998 10:51:59  Sample text: 1114-2 x1/1

   Amount: 0.13     of which 0.08     named and 0.04     unnamed
     Conc: 0.13     of which 0.08     named and 0.04     unnamed
   Tox #1: -            Tox #2: -           Tox #3:

Name                  #      RT Respnse      RA     Conc      Area  Height     S/N  Mod?

                      1   26:11 4.9e+04  0.31 n     0.02
                                4.9e+04                   1.2e+04 5.4e+03 1.8e+00 n  n
                                                          3.8e+04 9.0e+03 3.9e+00 y  n
2,3,7,8-TCDD          2   29:27 2.3e+05  0.11 n     0.08
                                2.3e+05                   2.2e+04 7.6e+03 2.5e+00 n  n
                                                          2.1e+05 3.8e+04 1.6e+01 y  n
                      3   29:36 1.8e+04  0.84 y     0.01
                                1.8e+04                   8.2e+03 3.4e+03 1.1e+00 n  n
                                                          9.8e+03 3.7e+03 1.6e+00 n  n
                      4   29:51 1.3e+04  1.58 n     0.00
                                1.3e+04                   8.0e+03 3.7e+03 1.2e+00 n  n
                                                          5.0e+03 2.4e+03 1.0e+00 n  n
                      5   30:44 1.8e+04  1.28 n     0.01
                                1.8e+04                   1.0e+04 7.6e+03 2.5e+00 n  n
                                                          8.0e+03 2.8e+03 1.2e+00 n  n
                      6   30:46 1.5e+04  0.87 y     0.01
                                1.5e+04                   7.0e+03 3.0e+[illegible]
                                                          8.0e+03 2.8e+03 1.2e+00 n  n


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 Ent: 41 Name: Total Penta-Furans  F:2  Mass: 339.860 341.857 Mod? no    #Hom:3

 Run: 7  File: a26sep98m    S:14 Acq:27-SEP-98 03:05:54 Proc:28-SEP-98 12:06:42
 Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal. m8290-092»Results: M8290-09»
 Version: V3.6 31-JUL-1998 10:51:59  Sample text: 1114-2 x1/1

   Amount: 0.07     of which 0.06     named and 0.01     unnamed
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     Conc: 0.07    of which 0.06    named and 0.01    unnamed
   Tox #1: -           Tox #2: -         Tox #3: -

Name                #     RT Respnse     RA    Conc     Area  Height     S/N  Mod?

1,2,3,7,8-PeCDF     1  32:34 8.8e+04  2.12 n    0.03
                             8.8e+04                6.0e+04 2.8e+04 1.4e+01 y  n
                                                    2.8e+04 1.2e+04 2.2e+00 n  n
2,3,4,7,8-PeCDF     2  33:01 9.3e+04  1.49 y    0.03
                             9.3e+04                5.6e+04 3.2e+04 1.6e+01 y  n
                                                    3.8e+04 1.5e+04 2.7e+00 n  n
                    3  33:07 2.2e+04  0.74 n    0.01
                             2.2e+04                9.3e+03 4.1e+03 2.1e+00 n  n
                                                    1.3e+04 5.6e+03 1.0e+00 n  n


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 Ent: 42 Name: Total Penta-Dioxins F:2  Mass: 355.855 357.852 Mod? no   #Hom:2

 Run: 7  File: a26sep98m    S:14 Acq:27-SEP-98 03:05:54 Proc:28-SEP-98 12:06:42
 Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
 Version: V3.6 31-JUL-1998 10:51:59  Sample text: 1114-2 x1/1

   Amount: 0.04    of which 0.02    named and 0.02    unnamed
     Conc: 0.04    of which 0.02    named and 0.02    unnamed
   Tox #1: -           Tox #2: -         Tox #3: -

Name                #     RT Respnse     RA    Conc     Area  Height     S/N  Mod?

                    1  32:10 5.7e+04  1.79 n    0.02
                             5.7e+04                3.7e+04 1.6e+04 3.8e+00 y  n
                                                    2.1e+04 5.2e+03 2.9e+00 n  n
1,2,3,7,8-PeCDD     2  33:14 4.4e+04  1.09 n    0.02
                             4.4e+04                2.3e+04 1.5e+04 3.5e+00 y  n
                                                    2.1e+04 1.0e+04 5.8e+00 y  n
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 Ent: 43 Name: Total Hexa-Furans   F:3  Mass: 373.821 375.818 Mod? no   #Hom:24

 Run: 7  File: a26sep98m    S:14 Acq:27-SEP-98 03:[illegible] Proc:28-SEP-98 12:06:42
 Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
 Version: V3.6 31-JUL-1998 10:51:59  Sample text: 1114-2 x1/1

   Amount: 0.34    of which 0.16    named and 0.18    unnamed
     Conc: 0.34    of which 0.16    named and 0.18    unnamed
   Tox #1: -            Tox #2: -        Tox #3: -

Name                #     RT Respnse    RA    Conc    Area  Height    S/N  Mod?

                    1  34:43 1.0e+04  0.76 n   0.00
                             1.0e+04                4.4e+03 3.0e+03 6.1e-01 n  n
                                                    5.7e+03 3.7e+03 1.1e+00 n  n
1,2,3,4,7,8-HxCDF   2  34:47 1.1e+05  1.13 y   0.06
                             1.1e+05                5.6e+04 1.8e+04 3.6e+00 y  n
                                                    5.0e+04 2.2e+04 6.4e+00 y  n
1,2,3,6,7,8-HxCDF   3  34:52 9.7e+04  1.61 n   0.04
                             9.7e+04                6.0e+04 2.4e+04 4.9e+00 y  n
                                                    3.[illegible]e+04 1.4e+04 4.1e+00 y  n
                    4  34:56 1.9e+04  0.96 n   0.
                             1.9e+04                9.4e+03 8.2e+03 1.6e+00 n  n
                                                    9.9e+03 5.1e+03 1.5e+00 n  n
                    5  34:58 2.1e+04  1.88 n   0.01
                             2.1e+04                1.3e+04 6.8e+03 1.4e+00 n  n
                                                    7.2e+03 3.8e+03 1.1e+00 n  n
                    6  35:02 2.5e+04  1.63 n   0.01
                             2.5e+04                1.6e+04 6.5e+03 1.3e+00 n  n
                                                    9.6e+03 4.8e+03 1.4e+00 n  n
2,3,4,6,7,8-HxCDF   7  35:14 7.2e+04  1.24 y   0.03
                             7.2e+04                4.0e+04 1.0e+04 2.0e+00 n  n
                                                    3.2e+04 9.[illegible]
                    8  35:19 3.4e+04  0.94 n   0.02
                             3.4e+04                1.7e+04 7.9e+03 1.6e+00 n  n
                                                    1.8e+04 5.9e+03 1.7e+00 n  n
                    9  35:24 3.8e+04  3.32 n   0.[illegible]
                             3.8e+04                2.9e+04 7.5e+03 1.5e+00 n  n
                                                    8.7e+03 3.7e+03 1.1e+00 n  n
                    10 35:28 1.5e+04  1.38 y   0.01
                             1.5e+04                8.8e+03 3.3e+03 6.7e-01 n  n
                                                    6.4e+03 2.8e+03 8.0e-01 n  n
                    11 35:31 1.1e+04  0.99 n   0.[illegible]
                             1.1e+04                5.4e+03 3.6e+03 7.1e-01 n  n
                                                    5.5e+03 2.6e+03 7.4e-01 n  n
                    12 35:38 2.4e+04  2.31 n   0.01
                             2.4e+04                1.7e+04 5.0e+03 1.0e+00 n  n
                                                    7.2e+03 2.4e+03 7.0e-01 n  n
                    13 35:39 1.9e+04  1.61 n   0.01
                             1.9e+04                1.2e+04 5.1e+03 1.0e+00 n  n
                                                    7.2e+03 2.4e+03 7.0e-01 n  n
 1,2,3,7,8,9-HxCDF  14 35:45 6.3e+04  1.04 n   0.04
                             6.3e+04                3.2e+04 9.9e+03 2.0e+00 n  n
                                                    3.1e+04 1.2e+04 3.4e+00 y  n
                    15 35:49 1.9e+04  1.12 y   0.01
                             1.9e+04                9.8e+03 5.4e+03 [illegible]
                                                    8.7e+03 4.2e+03 [illegible]
                    16 35:51 1.7e+04  0.92 n   0.01
                             1.7e+04                8.1e+03 3.9e+03 7.7e-01 n  n
                                                    8.7e+03 4.2e+03 1.2e+00 n  n
                    17 35:55 9.5e+03  1.47 n   0.00
                             9.5e+03                5.7e+03 3.5e+03 6.9e-01 n  n
                                                    3.8e+03 2.5e+03 7.1e-01 n  n
                    18 36:01 1.8e+04  0.65 n   0.01
                             1.8e+04                7.0e+03 4.1e+03 8.1e-01 n  n
                                                    1.1e+04 3.4e+03 9.8e-01 n  n
                    19 36:03 2.0e+04  0.85 n   0 01
                             2.0e+04                9.1e+03 5.7e+03 1.1e+00 n  n
                                                    1.1e+04 3.4e+03 9.8e-01 n  n
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                    20 36:06 8.9e+03  1.12 y    0.00
                             8.9e+03                4.7e+03 4.2e+03 8.4e-01 n  n
                                                    4.2e+03 2.8e+03 8.0e-01 n  n
                    21 36:08 1.3e+04  2.39 n    0.01
                             1.3e+04                9.2e+03 4.7e+03 9.4e-01 n  n
                                                    3.9e+03 2.1e+03 6.0e-01 n  n
                    22 36:15 1.7e+04  0.74 n    0.01
                             1.7e+04                7.3e+03 4.5e+03 9.0e-01 n  n
                                                    9.9e+03 3.2e+03 9.2e-01 n  n
                    23 36:17 2.6e+04  1.67 n    0.01
                             2.6e+04                1.6e+04 4.1e+03 8.1e-01 n  n
                                                    9.9e+03 3.2e+03 9.2e-01 n  n
                    24 36:23 1.1e+04  0.80 n    0.01
                             1.1e+04                5.0e+03 2.9e+03 5.7e-01 n  n
                                                    6.3e+03 3.8e+03 1.1e+00 n  n
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  Ent: 44 Name: Total Hexa-Dioxins  F:3  Mass: 389.816 391.813 Mod? no    #Hom:16

  Run: 7  File: a26sep98m    S:14 Acq:27-SEP-98 03:05:54 Proc:28-SEP-98 12:06:42
  Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
  Version: V3.6 31-JUL-1998 10:51:59  Sample text: 1114-2 x1/1

     Amount: 0.25    of which 0.08    named and 0.18    unnamed
       Conc: 0.25    of which 0.08    named and 0.18    unnamed
     Tox #1: -           Tox #2: -          Tox #3: -

Name                   #     RT Respnse    RA     Conc     Area  Height     S/N  Mod?

                       1  34:47 7.8e+04  3.07 n    0.03
                                7.8e+04                5.9e+04 2.2e+04 7.5e+00 y  n
                                                       1.9e+04 6.4e+03 2.1e+00 n  n
                       2  34:51 9.1e+04  3.42 n    0.04
                                9.1e+04                7.0e+04 2.4e+04 8.3e+00 y  n
                                                       2.0e+04 7.4e+03 2.4e+00 n  n
                       3  34:56 1.0e+05  1.36 y    0.04
                                1.0e+05                6.0e+04 2.4e+04 8.1e+00 y  n
                                                  [illegible] 1.5e+04 4.9e+00 y  n
                       4  35:02 2.4e+04  0.77 n    0.01
                                2.4e+04                1.1e+04 4.8e+03 1.6e+00 n  n
                                                       1.4e+04 6.8e+03 2.2e+00 n  n
                       5  35:13 1.7e+04  1.34 y    0.01
                                1.7e+04                9.6e+03 6.0e+03 2.0e+00 n  n
                                                       7.2e+03 5.1e+03 1.7e+00 n  n
1,2,3,4,7,8-HxCDD      6  35:19 3.5e+04  1.59 n    0.02
                                3.5e+04                2.1e+04 9.3e+03 3.2e+00 y  n
                                                       1.3e+04 8.1e+03 2.6e+00 n  n
1,2,3,6,7,8-HxCDD      7  35:23 8.3e+04  0.94 n    0.03
                                8.3e+04                4.0e+04 1.6e+04 5.5e+00 y  n
                                                       4.3e+04 9.[illegible]
                       8  35:29 1.1e+04  0.94 n    0.00
                                1.1e+04                5.4e+03 3.2e+03 1.1e+00 n  n
                                                       3.8e+03 4.1e+03 1.3e+00 n  n
1,2,3,7,8,9-HxCDD      9  35:35 6.2e+04  0.73 n    0.03
                                6.2e+04                2.6e+04 1.2e+04 4.0e+00 y  n
                                                       3.6e+04 9.4e+03 3.1e+00 y  n
                       10 35:43 1.1e+04  0.46 n    0.00
                                1.1e+04                3.5e+03 1.2e+03 4.1e-01 n  n
                                                       7.5e+03 2.9e+03 9.3e-01 n  n
                       11 35:51 1.3e+04  1.89 n    0.01
                                1.3e+04                8.8e+03 2.4e+03 8.0e-01 n  n
                                                       4.6e+03 2.8e+03 9.2e-01 n  n
                       12 35:56 1.2e+04  1.56 n    0.01
                                1.2e+04                7.3e+03 4.3e+03 1.5e+00 n  n
                                                       4.7e+03 2.3e+03 7.6e-01 n  n
                       13 36:01 1.1e+04  0.27 n    0.00
                                1.1e+04                2.3e+03 1.2e+03 4.1e-01 n  n
                                                       8.4e+03 4.7e+03 1.5e+00 n  n
                       14 36:10 1.7e+04  0.66 n    0.01
                                1.7e+04                6.7e+03 2.7e+03 9.1e-01 n  n
                                                       1.0e+04 3.7e+03 1.2e+00 n  n
                       15 36:13 1.4e+04  0.57 n    0.01
                                1.4e+04                4.9e+03 3.6e+03 [illegible]
                                                       8.6e+03 4.3e+03 [illegible]
                       16 36:21 1.0e+04  0.78 n    0.00
                                1.0e+04                4.5e+03 2.0e+03 6.7e-01 n  n
                                                       5.8e+03 3.0e+03 9.7e-01 n  n


                                                                Page 7 of 8

  Ent: 45 Name: Total Hepta-Furans  F:4  Mass: 407.782 409.779 Mod? no    #Hom:6

  Run: 7  File: a26sep98m    S:14 Acq:27-SEP-98 03:05:54 Proc:28-SEP-98 12:06:42
  Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
  Version: V3.6 31-JUL-1998 10:51:59  Sample text: 1114-2 x1/1
```

```
   Amount: 0.18    of which 0.11    named and 0.07    unnamed
     Conc: 0.18    of which 0.11    named and 0.07    unnamed
   Tox #1: -          Tox #2: -          Tox #3: -

Name                  #     RT Respnse     RA     Conc     Area  Height     S/N  Mod?

1,2,3,4,6,7,8-HpCDF1  37:01 1.2e+05  0.61 n    0.08
                            1.2e+05                  4.4e+04 1.6e+04 2.8e+00 n  n
                                                     7.3e+04 2.0e+04 7.6e+00 y  n
                   2  37:19 3.1e+04  1.24 n    0.02
                            3.1e+04                  1.7e+04 6.3e+03 1.1e+00 n  n
                                                     1.4e+04 3.6e+03 1.4e+00 n  n
                   3  37:45 1.1e+04  0.77 n    0.01
                            1.1e+04                  4.8e+03 1.9e+03 3.4e-01 n  n
                                                     6.1e+03 2.0e+03 7.6e-01 n  n
1,2,3,4,7,8,9-HpCDF4  38:11 4.5e+04  1.69 n    0.04
                            4.5e+04                  2.8e+04 7.3e+03 1.3e+00 n  n
                                                     1.7e+04 5.7e+03 2.2e+00 n  n
                   5  38:14 3.3e+04  0.98 y    0.02
                            3.3e+04                  1.6e+04 5.8e+03 1.0e+00 n  n
                                                     1.7e+04 5.7e+03 2.2e+00 n  n
                   6  38:40 1.8e+04  1.50 n    0.01
                            1.8e+04                  1.1e+04 3.3e+03 5.9e-01 n  n
                                                     7.2e+03 2.6e+03 9.7e-01 n  n
```

```
                                                                  Page 8 of 8

 Ent: 46 Name: Total Hepta-Dioxins F:4  Mass: 423.777 425.774 Mod? no   #Hom:5

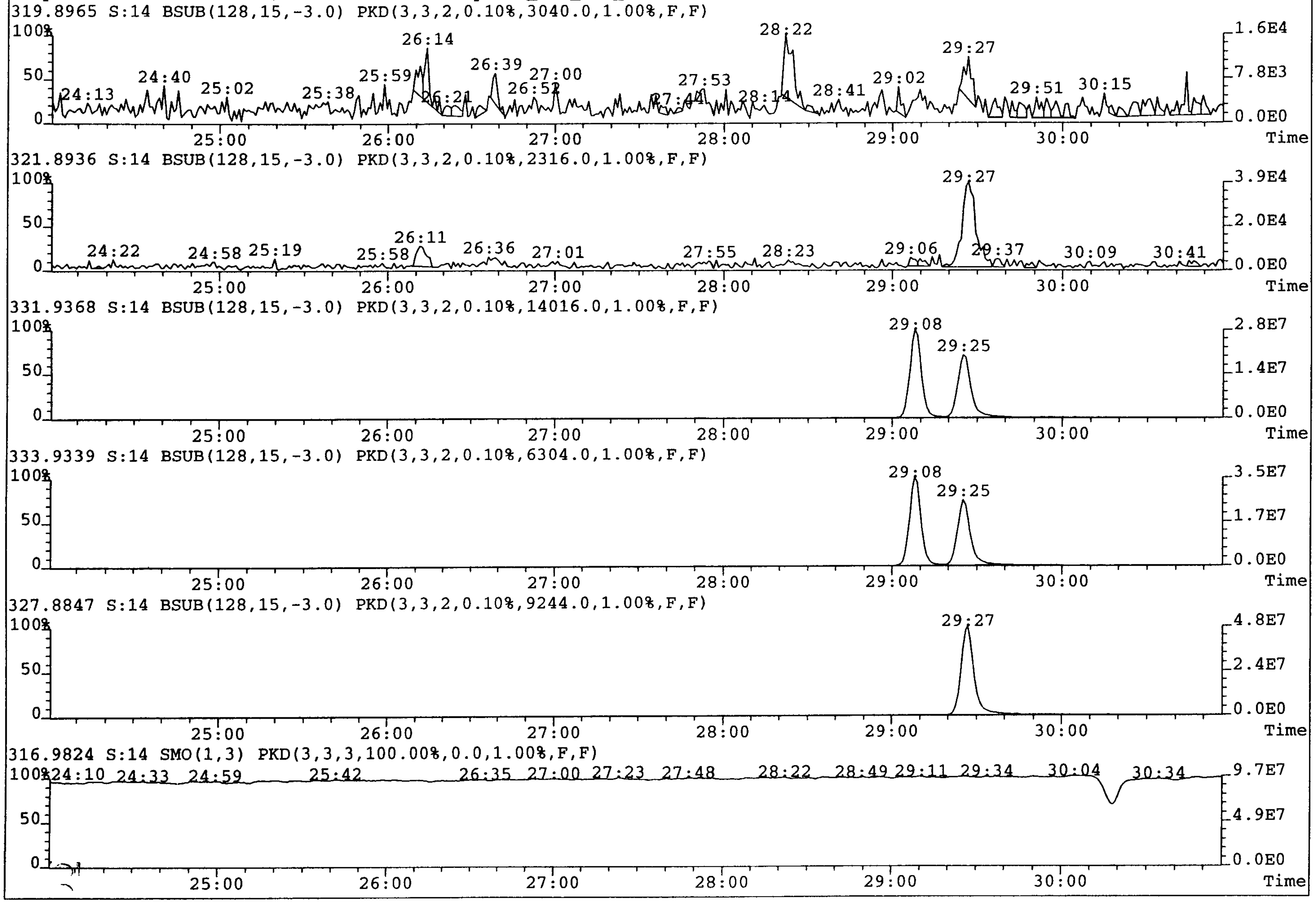
 Run: 7  File: a26sep98m    S:14 Acq:27-SEP-98 0[illegible]:05:[illegible]4 Proc:28-SEP-98 12:06:42
 Tables: Run: 26sep-crv  Analyte: m8290-092»  Cal: m8290-092»Results: M8290-09»
 Version: V3.6 31-JUL-1998 10:51:59  Sample text: 1114-2 x1/1

   Amount: 0.17    of which 0.08    named and 0.08    unnamed
     Conc: 0.17    of which 0.08    named and 0.08    unnamed
   Tox #1: -             Tox #2: -         Tox #3: -

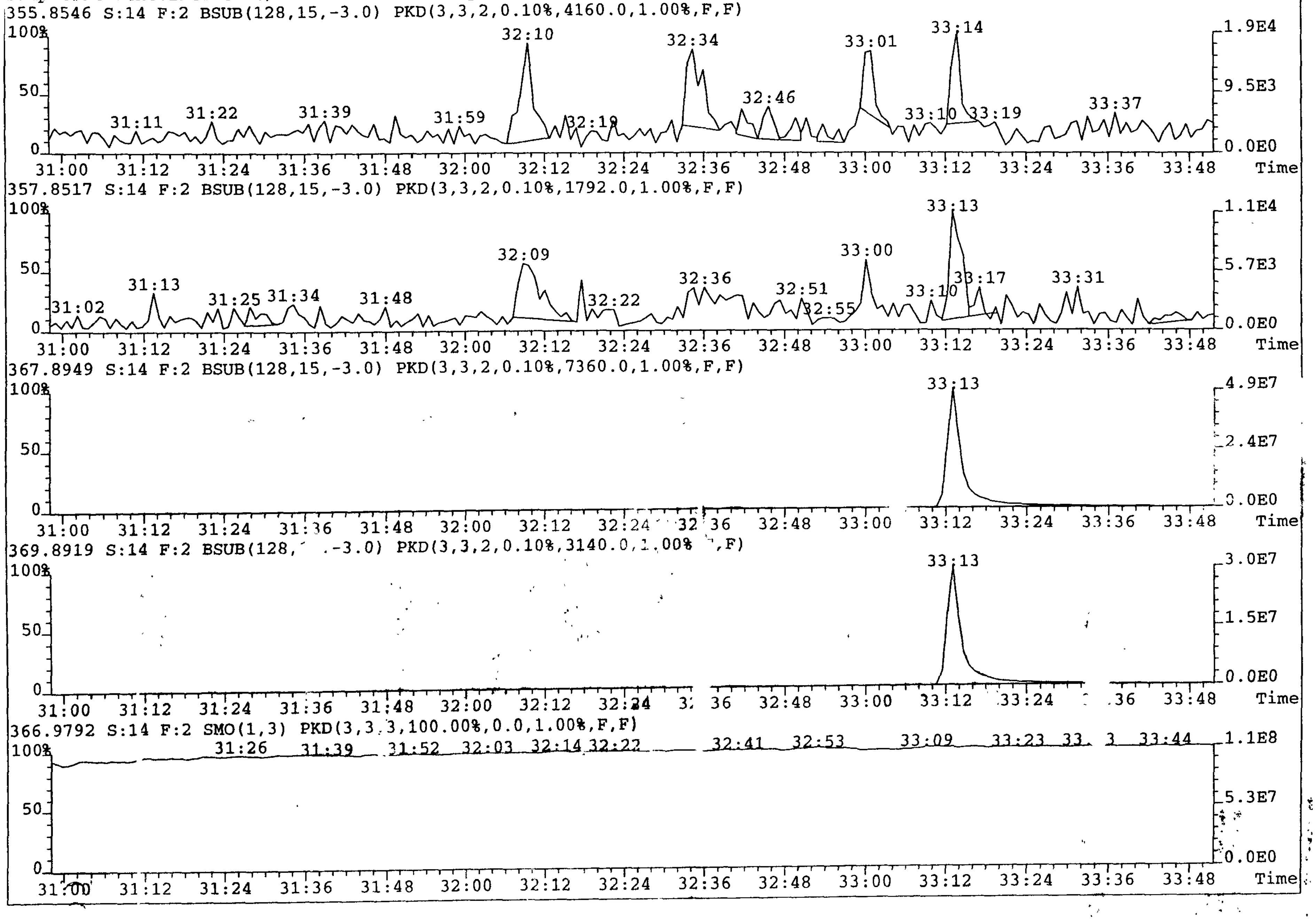
Name                   #     RT Respnse     RA     Conc     Area  Height     S/N  Mod?

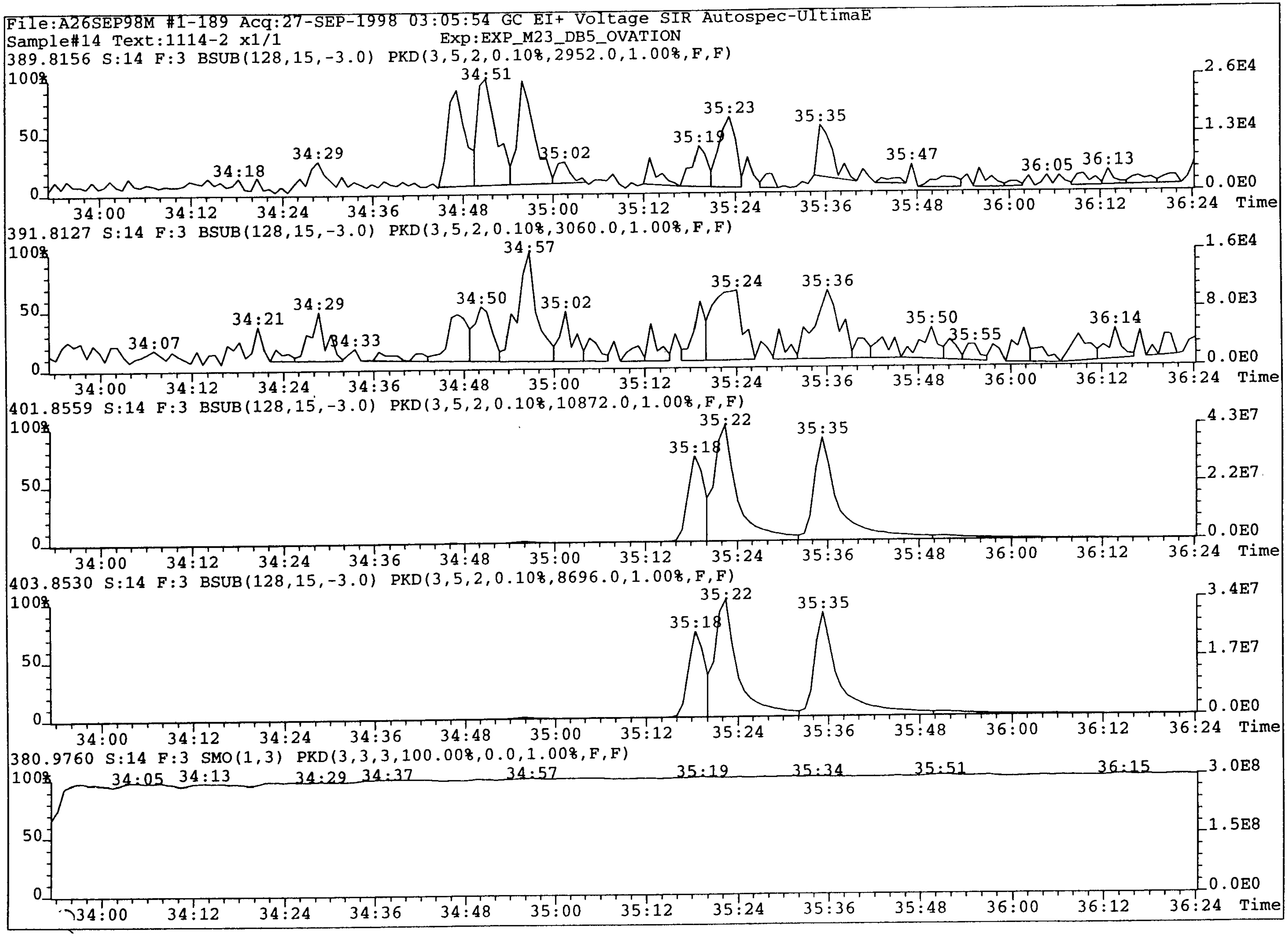
                       1  37:13 8.6e+04  1.71 n    0.06
                                8.6e+04                5.4e+04 1.6e+04 7.7e+00 y  n
                                                       3.2e+04 1.2e+04 7.6e+00 y  n
1,2,3,4,6,7,8-HpCDD2  37:50 1.2e+05  1.33 n    0.08
                                1.2e+05                6.7e+04 1.7e+04 8.2e+00 y  n
                                                       5.0e+04 1.7e+04 1.0e+01 y  n
                       3  38:20 6.0e+03  1.27 n    0.00
                                6.0e+03                3.[illegible]e+03 1.7e+03 7.9e-01 n  n
                                                       [illegible] 1.2e+03 7.3e-01 n  n
                       4  38:37 8.9e+03  0.99 y    0.01
                                8.9e+03                4.4e+03 1.7e+03 8.2e-01 n  n
                                                       4.5e+03 2.7e+03 1.7e+00 n  n
                       5  38:44 1.1e+04  0.69 n    0.01
                                1.1e+04                4.6e+03 1.7e+03 8.1e-01 n  n
                                                       6.7e+03 2.6e+03 1.6e+00 n  n
```

File:A26SEP98M #1-488 Acq:27-SEP-1998 03:05:54 GC EI+ Voltage SIR Autospec-UltimaE
Sample#14 Text:1114-2 x1/1 Exp:EXP_M23_DB5_OVATION



File:A26SEP98M #1-217 Acq:27-SEP-1998 03:05:54 GC EI+ Voltage SIR Autospec-UltimaE
Sample#14 Text:1114-2 x1/1 Exp:EXP_M23_DB5_OVATION





File:A26SEP98M #1-189 Acq:27-SEP-1998 03:05:54 GC EI+ Voltage SIR Autospec-UltimaE
Sample#14 Text:1114-2 x1/1 Exp:EXP_M23_DB5_OVATION

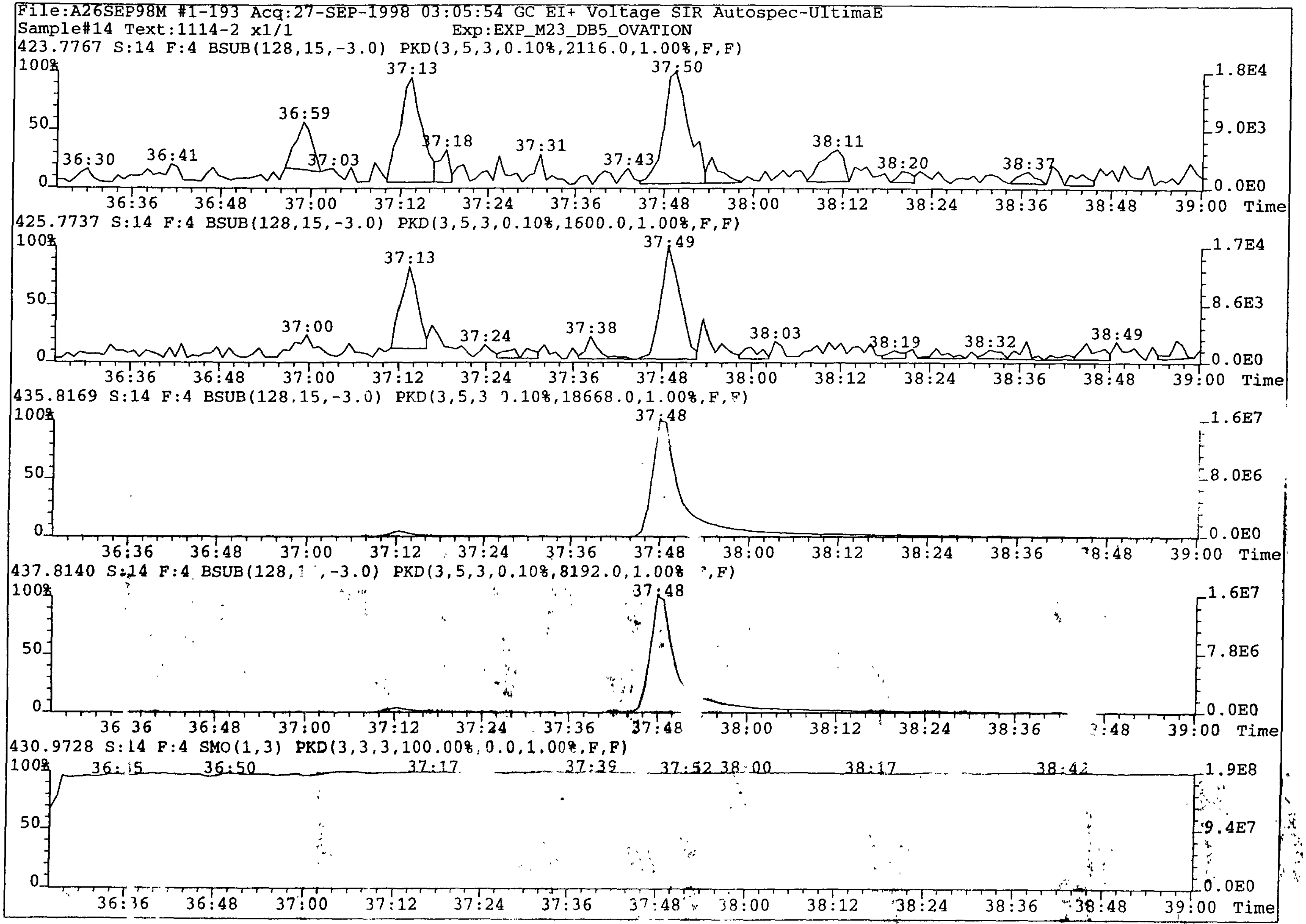
389.8156 S:14 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,2952.0,1.00%,F,F)

391.8127 S:14 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,3060.0,1.00%,F,F)

401.8559 S:14 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,10872.0,1.00%,F,F)

403.8530 S:14 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,8696.0,1.00%,F,F)

380.9760 S:14 F:3 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)



File:A26SEP98M #1-193 Acq:27-SEP-1998 03:05:54 GC EI+ Voltage SIR Autospec-UltimaE
Sample#14 Text:1114-2 x1/1 Exp:EXP_M23_DB5_OVATION

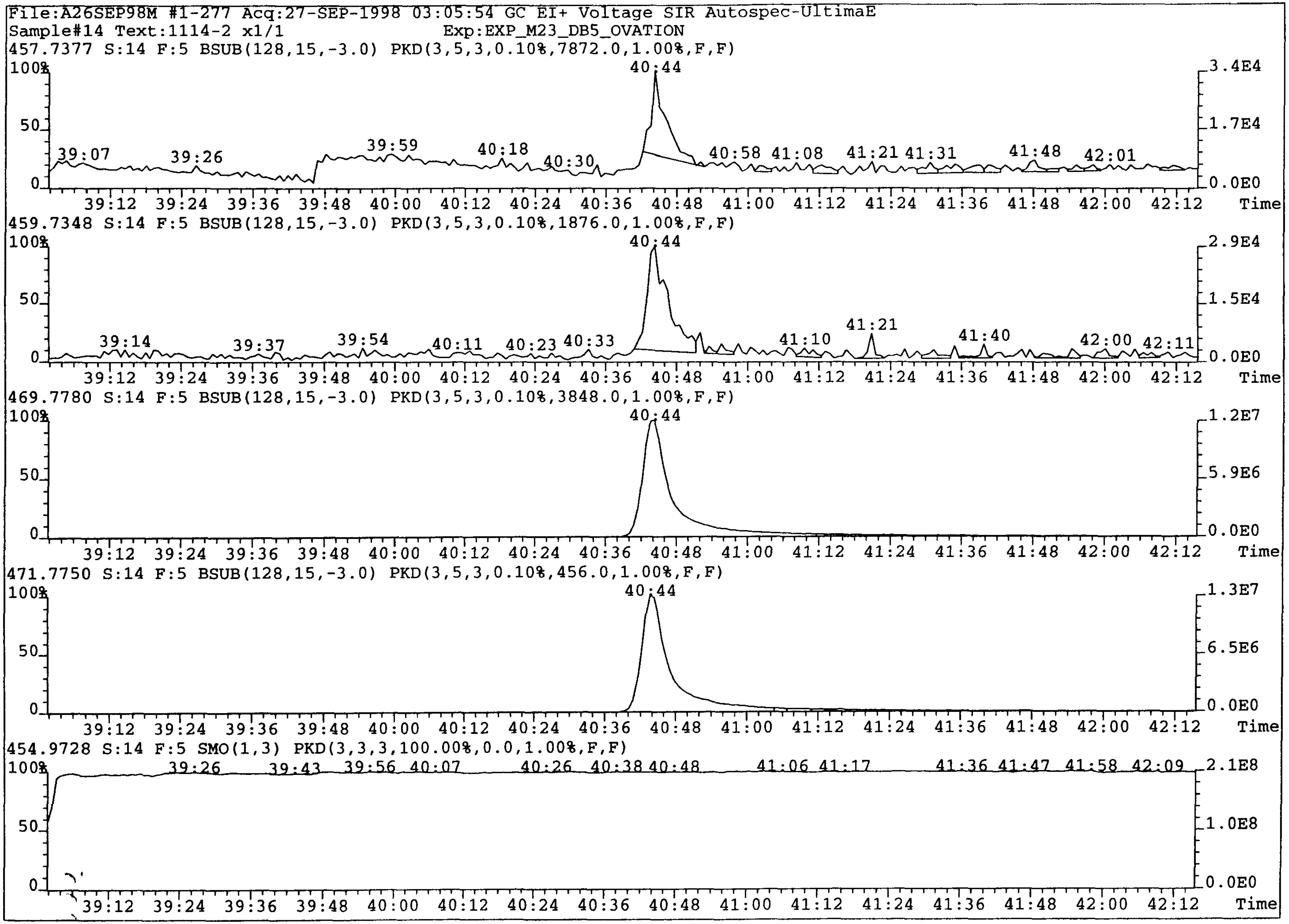
423.7767 S:14 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,2116.0,1.00%,F,F)

425.7737 S:14 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,1600.0,1.00%,F,F)

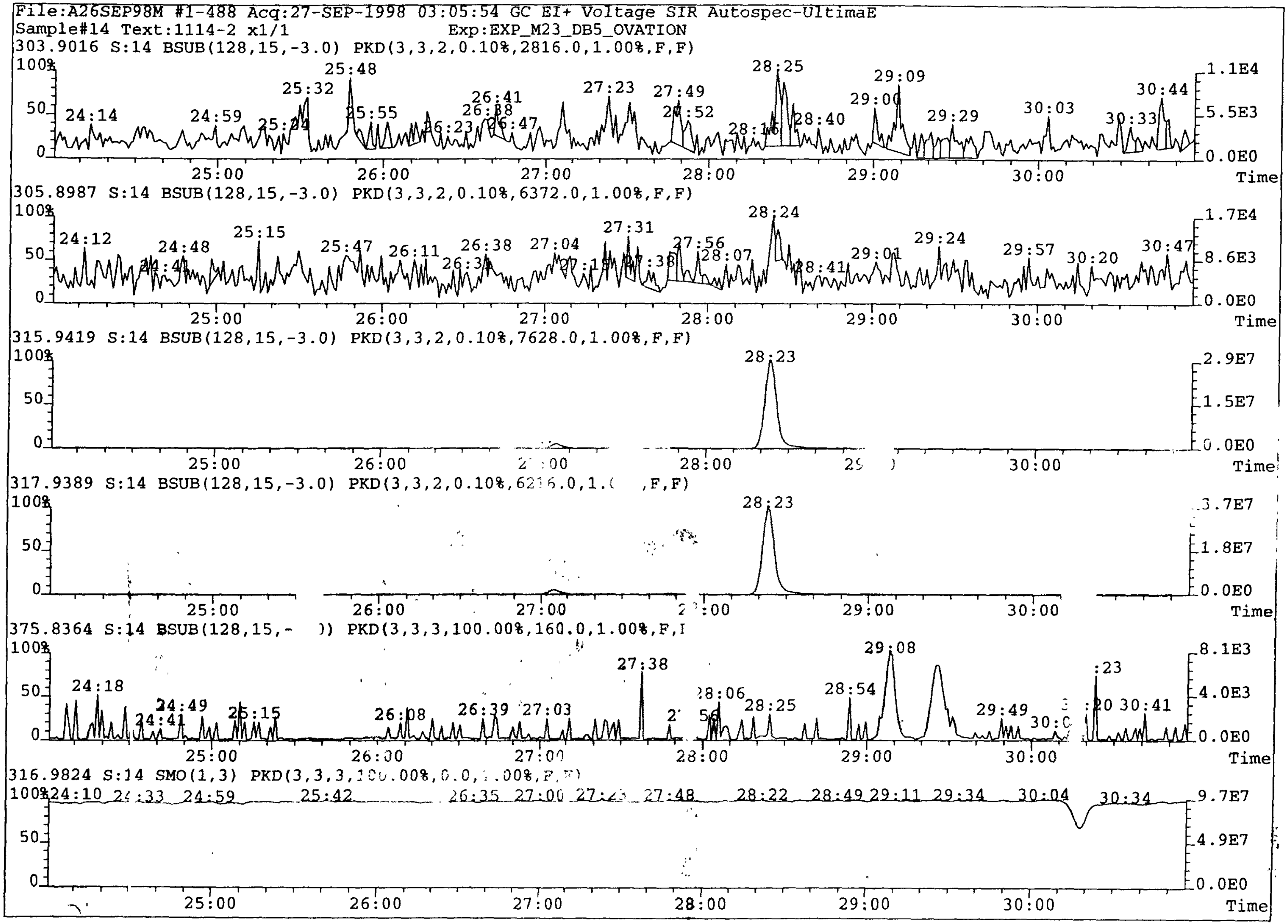
435.8169 S:14 F:4 BSUB(128,15,-3.0) PKD(3,5,3 0.10%,18668.0,1.00%,F,F)

437.8140 S:14 F:4 BSUB(128,1 ,-3.0) PKD(3,5,3,0.10%,8192.0,1.00% ,F)

430.9728 S:14 F:4 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)



```
File:A26SEP98M #1-277 Acq:27-SEP-1998 03:05:54 GC EI+ Voltage SIR Autospec-UltimaE
Sample#14 Text:1114-2 x1/1              Exp:EXP_M23_DB5_OVATION
457.7377 S:14 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,7872.0,1.00%,F,F)
459.7348 S:14 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,1876.0,1.00%,F,F)
469.7780 S:14 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,3848.0,1.00%,F,F)
471.7750 S:14 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,456.0,1.00%,F,F)
454.9728 S:14 F:5 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)
```



File:A26SEP98M #1-488 Acq:27-SEP-1998 03:05:54 GC EI+ Voltage SIR Autospec-UltimaE
Sample#14 Text:1114-2 x1/1 Exp:EXP_M23_DB5_OVATION

303.9016 S:14 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,2816.0,1.00%,F,F)

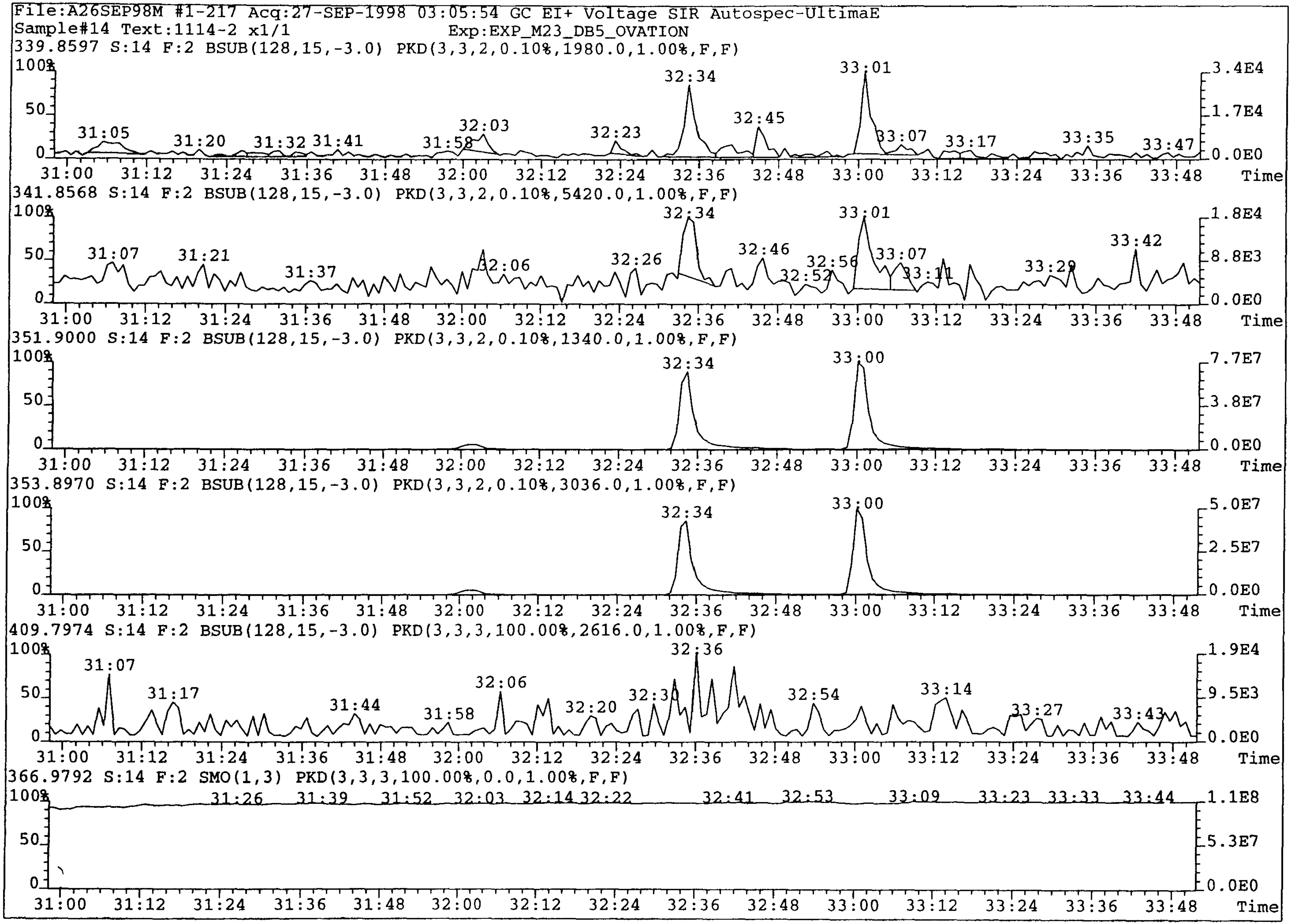
305.8987 S:14 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,6372.0,1.00%,F,F)

315.9419 S:14 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,7628.0,1.00%,F,F)

317.9389 S:14 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,6216.0,1.( ,F,F)

375.8364 S:14 BSUB(128,15,- )) PKD(3,3,3,100.00%,160.0,1.00%,F,I

316.9824 S:14 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)



File:A26SEP98M #1-217 Acq:27-SEP-1998 03:05:54 GC EI+ Voltage SIR Autospec-UltimaE
Sample#14 Text:1114-2 x1/1 Exp:EXP_M23_DB5_OVATION

339.8597 S:14 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,1980.0,1.00%,F,F)

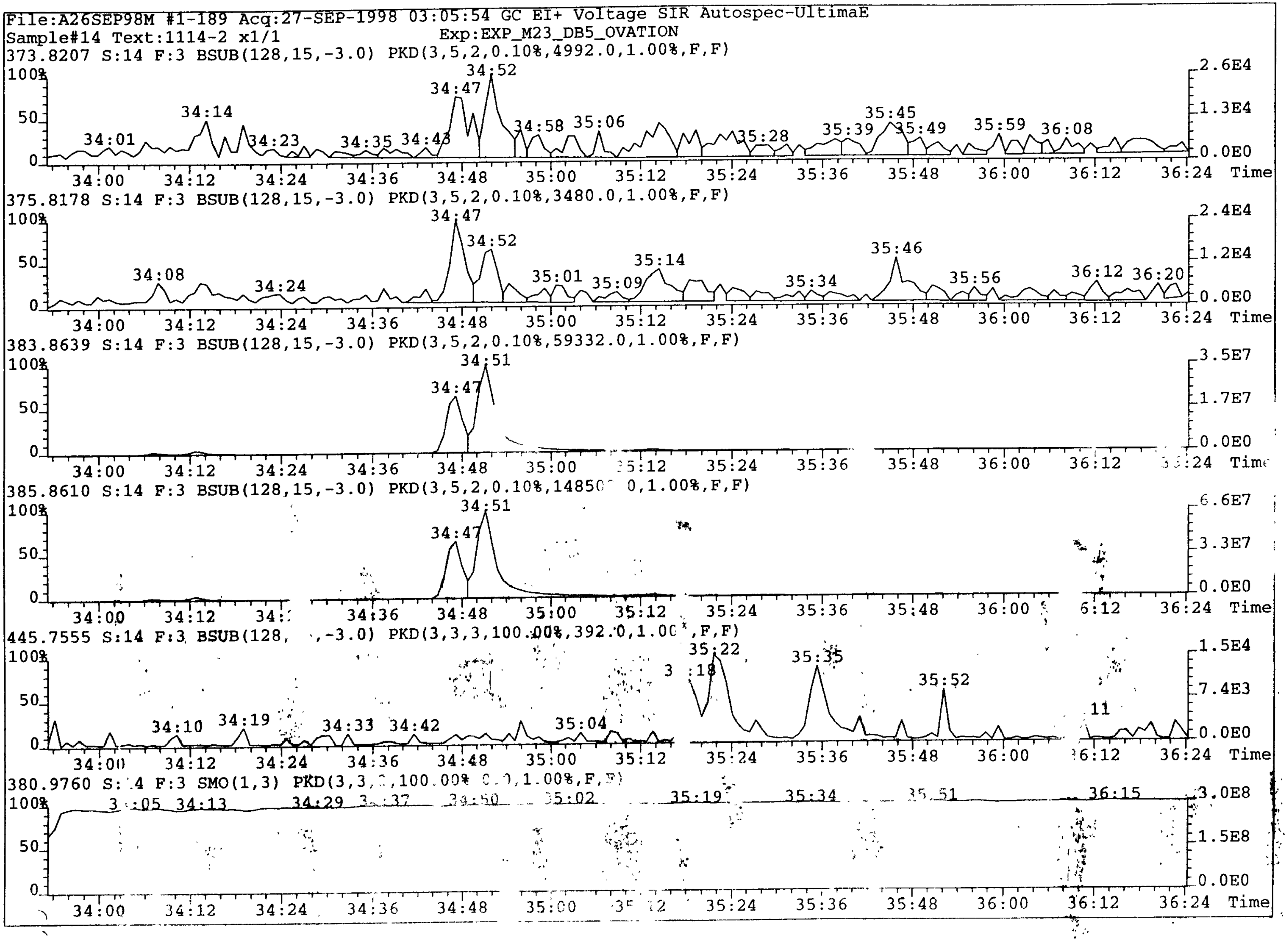
341.8568 S:14 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,5420.0,1.00%,F,F)

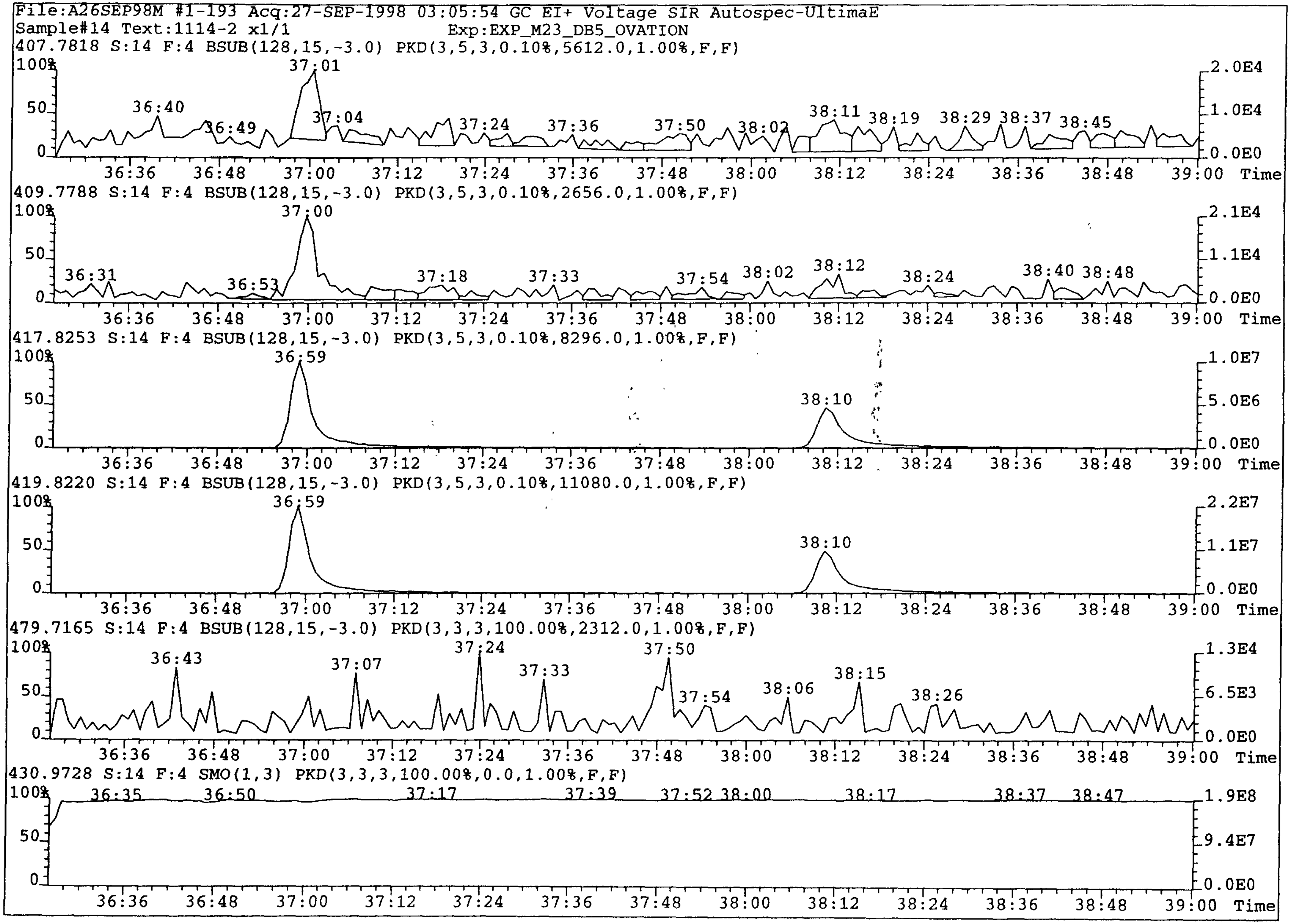
351.9000 S:14 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,1340.0,1.00%,F,F)

353.8970 S:14 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,3036.0,1.00%,F,F)

409.7974 S:14 F:2 BSUB(128,15,-3.0) PKD(3,3,3,100.00%,2616.0,1.00%,F,F)

366.9792 S:14 F:2 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)


File:A26SEP98M #1-189 Acq:27-SEP-1998 03:05:54 GC EI+ Voltage SIR Autospec-UltimaE
Sample#14 Text:1114-2 x1/1 Exp:EXP_M23_DB5_OVATION
373.8207 S:14 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,4992.0,1.00%,F,F)
34:01 34:14 34:23 34:35 34:43 34:47 34:52 34:58 35:06 35:28 35:39 35:45 35:49 35:59 36:08
2.6E4 1.3E4 0.0E0
375.8178 S:14 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,3480.0,1.00%,F,F)
34:08 34:24 34:47 34:52 35:01 35:09 35:14 35:34 35:46 35:56 36:12 36:20
2.4E4 1.2E4 0.0E0
383.8639 S:14 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,59332.0,1.00%,F,F)
34:47 34:51
3.5E7 1.7E7 0.0E0
385.8610 S:14 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,14850 0,1.00%,F,F)
34:47 34:51
6.6E7 3.3E7 0.0E0
445.7555 S:14 F:3 BSUB(128, ,-3.0) PKD(3,3,3,100.00%,392.0,1.00,F,F)
34:10 34:19 34:33 34:42 35:04 3 :18 35:22 35:35 35:52 11
1.5E4 7.4E3 0.0E0
380.9760 S:14 F:3 SMO(1,3) PKD(3,3,3,100.00% 0.0,1.00%,F,F)
3 :05 34:13 34:29 3 :37 34:50 35:02 35:19 35:34 35:51 36:15
3.0E8 1.5E8 0.0E0
100% 50 0
34:00 34:12 34:24 34:36 34:48 35:00 35:12 35:24 35:36 35:48 36:00 36:12 36:24 Time



File:A26SEP98M #1-193 Acq:27-SEP-1998 03:05:54 GC EI+ Voltage SIR Autospec-UltimaE
Sample#14 Text:1114-2 x1/1 Exp:EXP_M23_DB5_OVATION

407.7818 S:14 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,5612.0,1.00%,F,F)

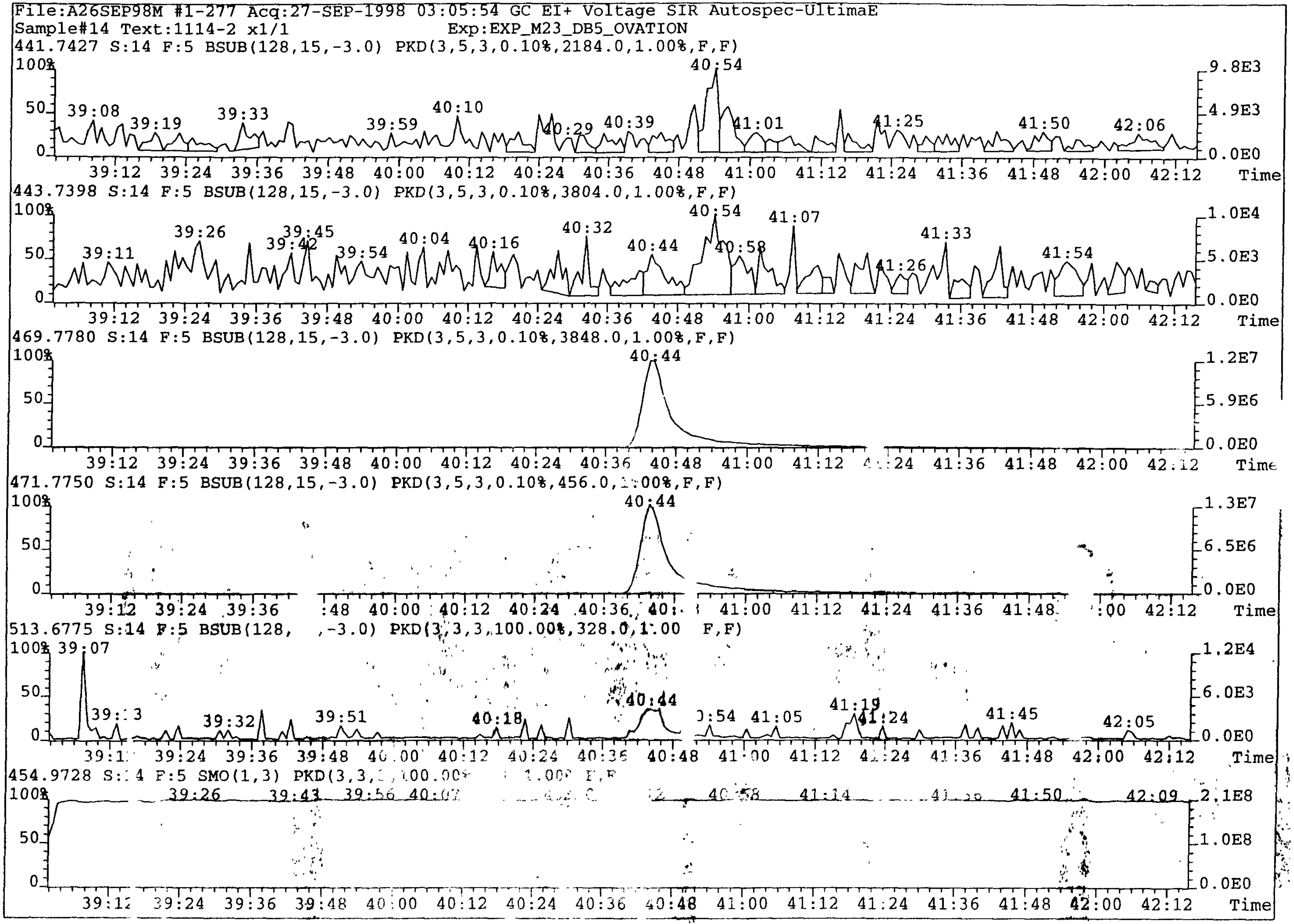
409.7788 S:14 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,2656.0,1.00%,F,F)

417.8253 S:14 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,8296.0,1.00%,F,F)

419.8220 S:14 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,11080.0,1.00%,F,F)

479.7165 S:14 F:4 BSUB(128,15,-3.0) PKD(3,3,3,100.00%,2312.0,1.00%,F,F)

430.9728 S:14 F:4 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)



File:A26SEP98M #1-277 Acq:27-SEP-1998 03:05:54 GC EI+ Voltage SIR Autospec-UltimaE
Sample#14 Text:1114-2 x1/1 Exp:EXP_M23_DB5_OVATION

# Section 4

## System Performance

Paradigm Analytical Labs

## Section 4-1

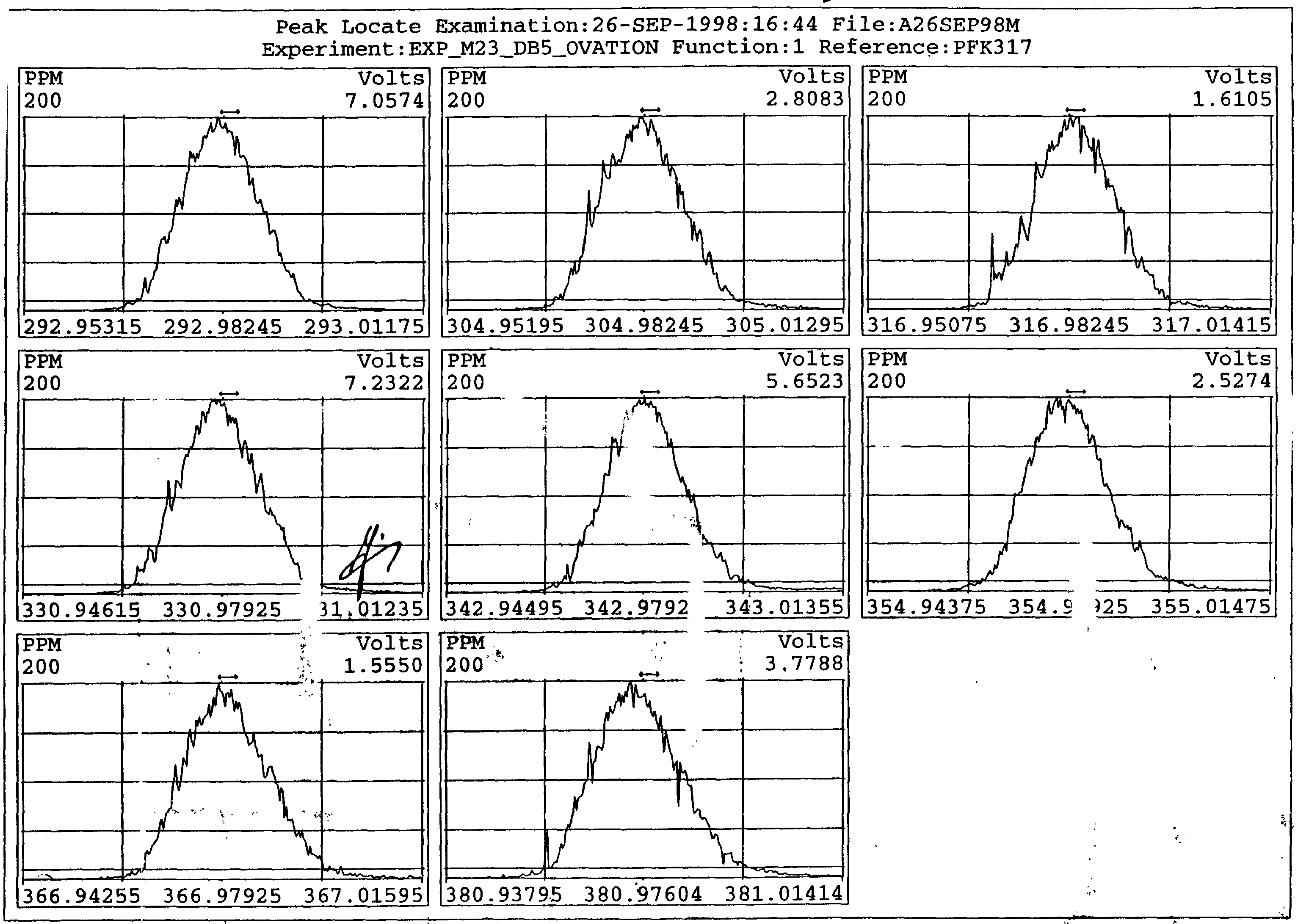
# Mass Spectrometer Performance Check

## Mass Resolution

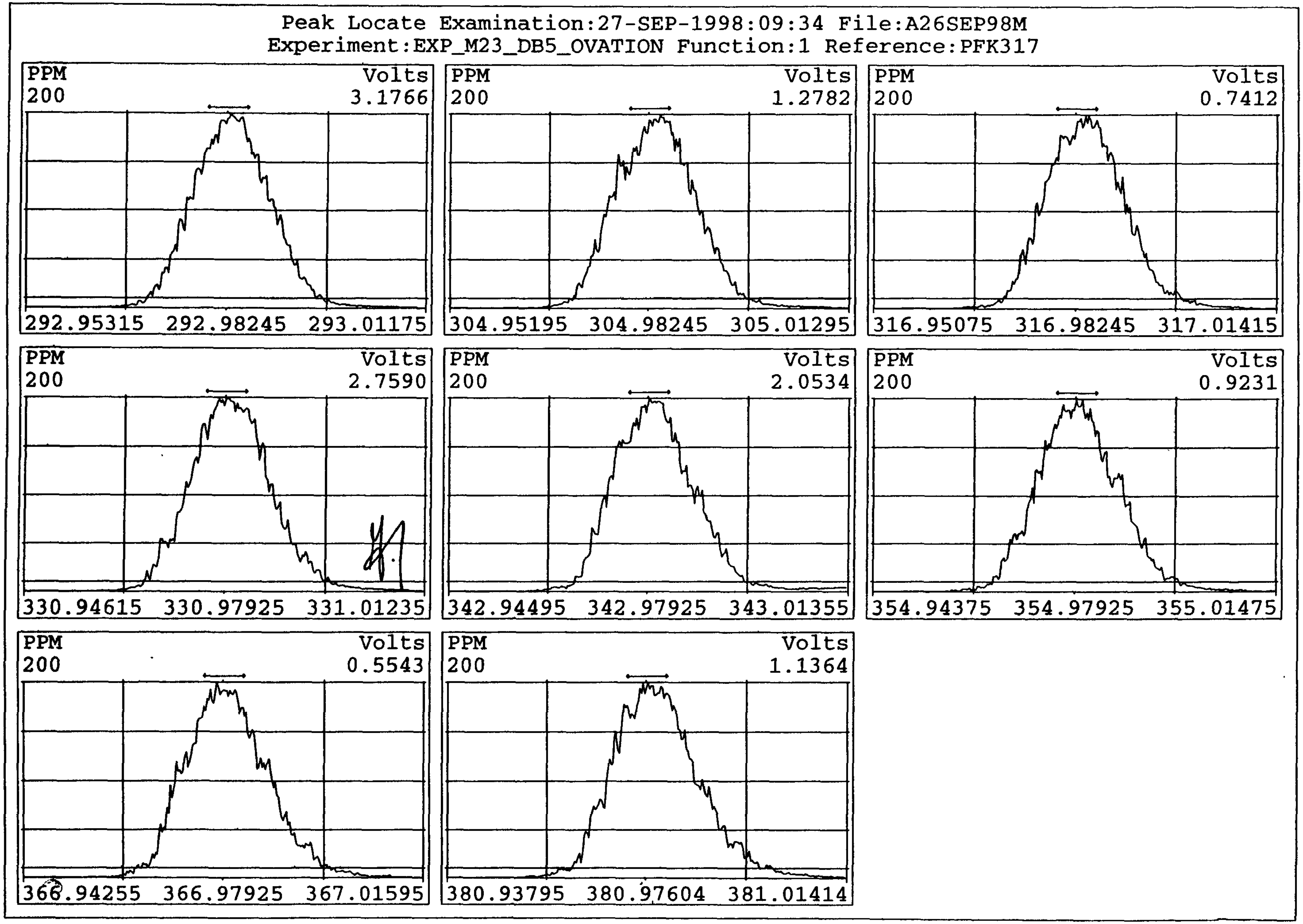
**Documentation for the Analysis**

**of**

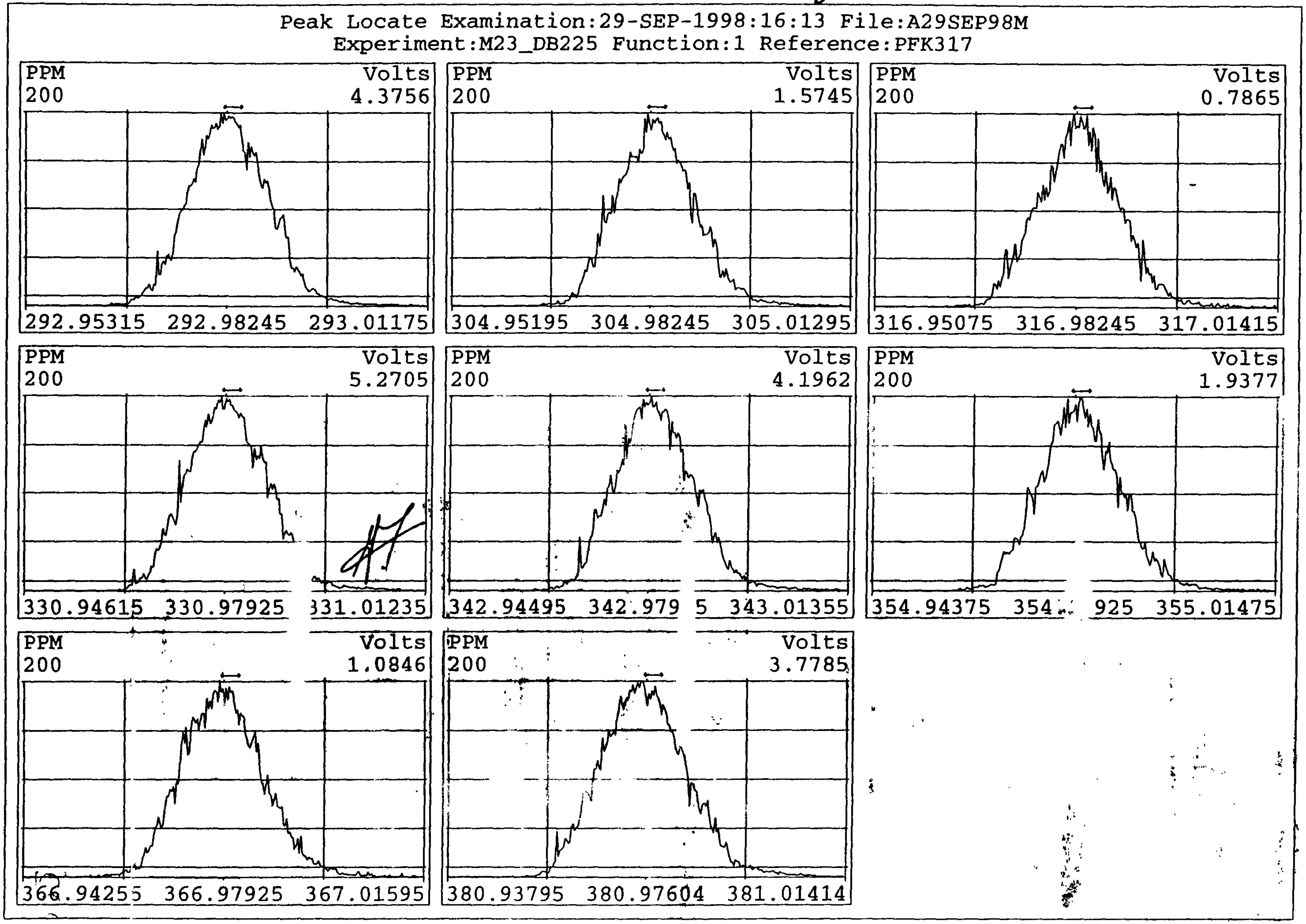
**Polychlorinated Dibenzo-*p*-Dioxins & Dibenzofurans**



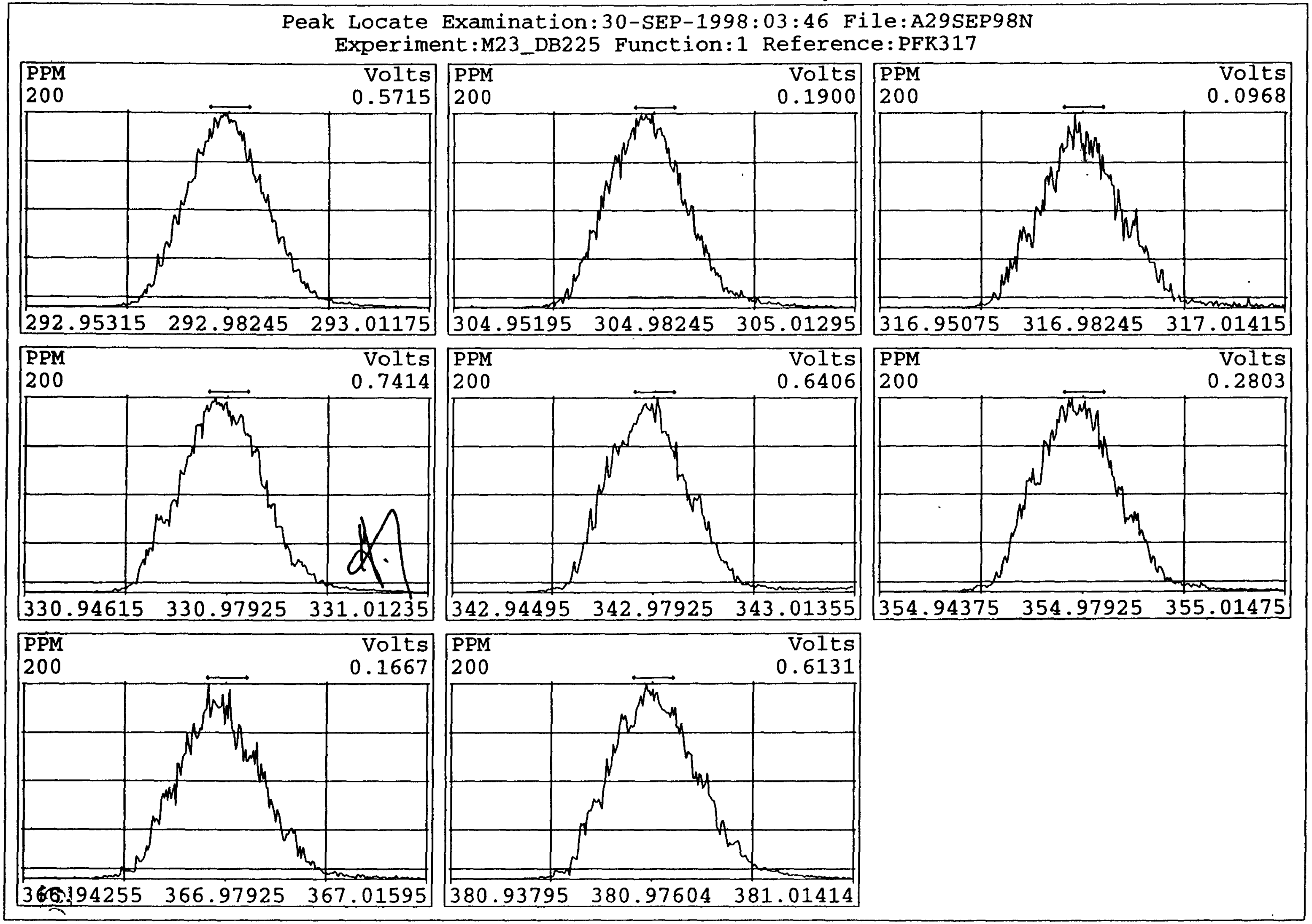
Peak Locate Examination:26-SEP-1998:16:44 File:A26SEP98M
Experiment:EXP_M23_DB5_OVATION Function:1 Reference:PFK317



Peak Locate Examination:27-SEP-1998:09:34 File:A26SEP98M
Experiment:EXP_M23_DB5_OVATION Function:1 Reference:PFK317



Peak Locate Examination:29-SEP-1998:16:13 File:A29SEP98M
Experiment:M23_DB225 Function:1 Reference:PFK317


Peak Locate Examination:30-SEP-1998:03:46 File:A29SEP98N
Experiment:M23_DB225 Function:1 Reference:PFK317

# Section 4

## System Performance

**Paradigm Analytical Labs**

# Section 4-2

# Gas Chromatography Performance Check

## Isomer Specificity & Retention Time Windows

### Documentation for the Analysis

### of

### Polychlorinated Dibenzo-*p*-Dioxins & Dibenzofurans

$B_{ws}$ = Water vapor in the gas stream (from Method 5 or Reference Method 4), proportion by volume.

$C_p$ = Pitot tube coefficient, dimensionless.

$K_p$ = Pitot tube constant,

$$34.97 \frac{m}{sec}\left[\frac{(g/g\text{-mole})\ (mmHg)}{(^{\circ}K)\ (mmH_2O)}\right]^{1/2}$$

for the metric system.

$$85.49 \frac{ft}{sec}\left[\frac{lb/lb\text{-mole})\ (in.Hg)}{(^{\circ}R)\ (in.H_2O)}\right]^{1/2}$$

for the English system.

$M_d$ = Molecular weight of stack gas, dry basis (see Section 3.6), g/g-mole (lb/lb-mole).

$M_s$ = Molecular weight of stack gas, wet basis, g/g-mole (lb/lb-mole).

$$= M_d(1-B_{ws}) + 18.0B_{ws}$$

Eq. 2-5

$P_{bar}$ = Barometric pressure at measurement site, mm Hg (in. Hg).

$P_g$ = Stack static pressure, mm Hg (in. Hg).

$P_s$ = Absolute stack pressure, mm Hg (in. Hg),

$$= P_{bar} + P_g$$

Eq. 2-6

$P_{std}$ = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

$Q_{sd}$ = Dry volumetric stack gas flow rate corrected to standard conditions, $dsm^3/hr$ (dscf/hr).

$t_s$ = Stack temperature, °C (°F).

carefully reexamined in top, side, and end views. If the pitot face openings are still aligned within the specifications illustrated in Figure 2-2 or 2-3, it can be assumed that the baseline coefficient of the pitot tube has not changed. If, however, the tube has been damaged to the extent that it no longer meets the specifications of the Figure 2-2 or 2-3, the damage shall either be repaired to restore proper alignment of the face openings, or the tube shall be discarded.

**4.1.6.2.2 Pitot Tube Assemblies.** After each field use, check the face opening alignment of the pitot tube, as in Section 4.1.6.2.1; also, remeasure the intercomponent spacings of the assembly. If the intercomponent spacings have not changed and the face opening alignment is acceptable, it can be assumed that the coefficient of the assembly has not changed. If the face opening alignment is no longer within the specifications of Figure 2-2 or 2-3, either repair the damage or replace the pitot tube (calibrating the new assembly, if necessary). If the intercomponent spacings have changed, restore the original spacings, or recalibrate the assembly.

**4.2 Standard Pitot Tube (if applicable).** If a standard pitot tube is used for the velocity traverse, the tube shall be constructed according to the criteria of Section 2.7 and shall be assigned a baseline coefficient value of 0.99. If the standard pitot tube is used as part of an assembly, the tube shall be in an interference-free arrangement (subject to the approval of the Administrator).

**4.3 Temperature Gauges.** After each field use, calibrate dial thermometers, liquid-filled bulb thermometers, thermocouple-potentiometer systems, and other gauges at a temperature within 10 percent of the average absolute stack temperature. For temperatures up to 405°C (761°F), use an ASTM mercury-in-glass reference thermometer, or equivalent, as a reference; alternatively, either a reference thermocouple and potentiometer (calibrated by NBS) or thermometric fixed points, e.g., ice bath and boiling water (corrected for barometric pressure) may be used. For temperatures above 405°C (761°F), use an NBS-calibrated reference thermocouple-potentiometer system or an alternative reference, subject to the approval of the Administrator.

If, during calibration, the absolute temperature measured with the gauge being calibrated and the reference gauge agree within 1.5 percent, the temperature data taken in the field shall be considered valid. Otherwise, the pollutant emission test shall either be considered invalid or adjustments (if appropriate) of the test results shall be made, subject to the approval of the Administrator.

**4.4 Barometer.** Calibrate the barometer used against a mercury barometer.

## 5. CALCULATIONS

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

### 5.1 Nomenclature.

A = Cross-sectional area of stack, $m^2$ ($ft^2$).

located at or near the center of the duct; however, insertion of a probe sheath into a small duct may cause significant cross-sectional area blockage and yield incorrect coefficient values (Citation 9 in the Bibliography). Therefore, to minimize the blockage effect, the calibration point may be a few inches off-center if necessary. The actual blockage effect will be negligible when the theoretical blockage, as determined by a projected-area model of the probe sheath, is 2 percent or less of the duct cross-sectional area for assemblies without external sheaths (Figure 2-10a), and 3 percent or less for assemblies with external sheaths (Figure 2-10b).

4.1.5.2 For those probe assemblies in which pitot tube-nozzle interference is a factor (i.e., those in which the pitot-nozzle separation distance fails to meet the specification illustrated in Figure 2-6A), the value of $C_{p(s)}$ depends upon the amount of free-space between the tube and nozzle, and therefore is a function of nozzle size. In these instances, separate calibrations shall be performed with each of the commonly used nozzle sizes in place. Note that the single-velocity calibration technique is acceptable for this purpose, even though the larger nozzle sizes (>0.635 cm or 1/4 in.) are not ordinarily used for isokinetic sampling at velocities around 915 m/min (3,000 ft/min), which is the calibration velocity; note also that it is not necessary to draw an isokinetic sample during calibration (see Citation 19 in the Bibliography).

4.1.5.3 For a probe assembly constructed such that its pitot tube is always used in the same orientation, only one side of the pitot tube need be calibrated (the side which will face the flow). The pitot tube must still meet the alignment specifications of Figure 2-2 or 2-3, however, and must have an average deviation ($\sigma$) value of 0.01 or less (see Section 4.1.4.4.)

**4.1.6 Field Use and Recalibration.**

**4.1.6.1 Field Use.**

4.1.6.1.1 When a Type S pitot tube (isolated or in an assembly) is used in the field, the appropriate coefficient value (whether assigned or obtained by calibration) shall be used to perform velocity calculations. For calibrated Type S pitot tubes, the A side coefficient shall be used when the A side of the tube faces the flow, and the B side coefficient shall be used when the B side faces the flow; alternatively, the arithmetic average of the A and B side coefficient values may be used, irrespective of which side faces the flow.

4.1.6.1.2 When a probe assembly is used to sample a small duct, 30.5 to 91.4 cm (12 to 36 in.) in diameter, the probe sheath sometimes blocks a significant part of the duct cross-section, causing a reduction in the effective value of $C_{p(s)}$. Consult Citation 9 in the Bibliography for details. Conventional pitot-sampling probe assemblies are not recommended for use in ducts having inside diameters smaller than 30.5 cm (12 in.) (see Citation 16 in the Bibliography).

**4.1.6.2 Recalibration.**

4.1.6.2.1 **Isolated Pitot Tubes.** After each field use, the pitot tube shall be mean B-side coefficient; calculate the difference between these two average values.

4.1.4.3 Calculate the deviation of each of the three A-side values of $C_{p(s)}$ from $\overline{C}_p$ (side A), and the deviation of each B-side values of $C_{p(s)}$ from $\overline{C}_p$ (side B). Use the following equation:

$$\text{Deviation} = C_{p_{(s)}} - \overline{C_p}(\text{A or B})$$

Eq. 2-3

4.1.4.4 Calculate σ, the average deviation from the mean, for both the A and B sides of the pitot tube. Use the following equation:

$$\sigma(\text{side A or B}) = \frac{\sum_{1}^{3} \left| C_{p(s)} - \overline{C_p}(\text{A or B}) \right|}{3}$$

Eq. 2-4

4.1.4.5 Use the Type S pitot tube only if the values of σ (side A) and σ (side B) are less than or equal to 0.01 and if the absolute value of the difference between $\overline{C}_p$ (A) and $\overline{C}_p$ (B) is 0.01 or less.

**4.1.5 Special Considerations.**

**4.1.5.1 Selection of Calibration Point.**

2.3

4.1.5.1.1 When an isolated Type S pitot tube is calibrated, select a calibration point at or near the center of the duct, and follow the procedures outlined in Sections 4.1.3 and 4.1.4 above. The Type S pitot coefficients so obtained, i.e., $\overline{C}_p$ (side A) and $\overline{C}_p$ (side B), will be valid, so long as either: (1) the isolated pitot tube is used; or (2) the pitot tube is used with other components (nozzle, thermocouple, sample probe) in an arrangement that is free from aerodynamic interference effects (see Figures 2-6 through 2-8).

4.

4.1.5.1.2 For Type S pitot tube-thermocouple combinations (without sample probe), select a calibration point at or near the center of the duct, and follow the procedures outlined in Sections 4.1.3 and 4.1.4 above. The coefficients so obtained will be valid so long as the pitot tube-thermocouple combination is used by itself or with other components in an interference-free arrangement (Figures 2-6 and 2-8).

4.1.5.1.3 For assemblies with sample probes, the calibration point should be

4.1.3.4 Read $\Delta p_{std}$, and record its value in a data table similar to the one shown in Figure 2-9. Remove the standard pitot tube from the duct, and disconnect it from the manometer. Seal the standard entry port.

4.1.3.5 Connect the Type S pitot tube to the manometer. Open the Type S entry port. Check the manometer level and zero. Insert and align the Type S pitot tube so that its A side impact opening is at the same point as was the standard pitot tube and is pointed directly into the flow. Make sure that the entry port surrounding the tube is properly sealed.

4.1.3.6 Read $\Delta p_s$, and enter its value in the data table. Remove the Type S pitot tube from the duct, and disconnect it from the manometer.

4.1.3.7 Repeat Steps 4.1.3.3 through 4.1.3.6 above until three pairs of Δp readings have been obtained.

4.1.3.8 Repeat Steps 4.1.3.3 through 4.1.3.7 above for the B side of the Type S pitot tube.

4.1.3.9 Perform calculations, as described in Section 4.1.4 below.

**4.1.4 Calculations.**

4.1.4.1 For each of the six pairs of Δp readings (i.e., three from side A and three from side B) obtained in Section 4.1.3 above, calculate the value of the Type S pitot tube coefficient as follows:

$$C_{p(s)} = C_{p(std)} \sqrt{\frac{\Delta p_{std}}{\Delta p_s}}$$

Eq. 2-2

Where:

$C_{p(s)}$ = Type S pitot tube coefficient.

$C_{p(std)}$ = Standard pitot tube coefficient; use 0.99 if the coefficient is unknown and the tube is designed according to the criteria of Sections 2.7.1 to 2.7.5 of this method.

$\Delta p_{std}$ = Velocity head measured by the standard pitot tube, cm (in.) $H_2O$.

$\Delta p_s$ = Velocity head measured by the Type S pitot tube, cm (in.) $H_2O$.

4.1.4.2 Calculate $\bar{C}_p$ (side A), the mean A-side coefficient, and $\bar{C}_p$ (side B), the

$D_e$ = Equivalent diameter.
L = Length.
W = Width.

To ensure the presence of stable, fully developed flow patterns at the calibration site, or "test section," the site must be located at least eight diameters downstream and two diameters upstream from the nearest disturbances.

**NOTE:** The eight- and two-diameter criteria are not absolute; other test section locations may be used (subject to approval of the Administrator), provided that the flow at the test site is stable and demonstrably parallel to the duct axis.

4.1.2.3 The flow system shall have the capacity to generate a test-section velocity around 915 m/min (3,000 ft/min). This velocity must be constant with time to guarantee steady flow during calibration. Note that Type S pitot tube coefficients obtained by single-velocity calibration at 915 m/min (3,000 ft/min) will generally be valid to ±3 percent for the measurement of velocities above 305 m/min (1,000 ft/min) and to ±5 to 6 percent for the measurement of velocities between 180 and 305 m/min (600 and 1,000 ft/min). If a more precise correlation between $C_p$ and velocity is desired, the flow system shall have the capacity to generate at least four distinct, time-invariant test-section velocities covering the velocity range from 180 to 1,525 m/min (600 to 5,000 ft/min), and calibration data shall be taken at regular velocity intervals over this range (see Citations 9 and 14 in the Bibliography for details).

4.1.2.4 Two entry ports, one each for the standard and Type S pitot tubes, shall be cut in the test section; the standard pitot entry port shall be located slightly downstream of the Type S port, so that the standard and Type S impact openings will lie in the same cross-sectional plane during calibration. To facilitate alignment of the pitot tubes during calibration, it is advisable that the test section be constructed of plexiglas or some other transparent material.

4.1.3 Calibration Procedure. Note that this procedure is a general one and must not be used without first referring to the special considerations presented in Section 4.1.5. Note also that this procedure applies only to single-velocity calibration. To obtain calibration data for the A and B sides of the Type S pitot tube, proceed as follows:

4.1.3.1 Make sure that the manometer is properly filled and that the oil is free from contamination and is of the proper density. Inspect and leak-check all pitot lines; repair or replace if necessary.

4.1.3.2 Level and zero the manometer. Turn on the fan, and allow the flow to stabilize. Seal the Type S entry port.

4.1.3.3 Ensure that the manometer is level and zeroed. Position the standard pitot tube at the calibration point (determined as outlined in Section 4.1.5.1), and align the tube so that its tip is pointed directly into the flow. Particular care should be taken in aligning the tube to avoid yaw and pitch angles. Make sure that the entry port surrounding the tube is properly sealed.

pitot tube is part of an assembly, calibration may still be required, despite knowledge of the baseline coefficient value (see Section 4.1.1).

If $D_t$, $P_A$, and $P_B$ are outside the specified limits, the pitot tube must be calibrated as outlined in Sections 4.1.2 through 4.1.5 below.

**4.1.1 Type S Pitot Tube Assemblies.** During sample and velocity traverses, the isolated Type S pitot tube is not always used; in many instances, the pitot tube is used in combination with other source-sampling components (thermocouple, sampling probe, nozzle) as part of an "assembly." The presence of other sampling components can sometimes affect the baseline value of the Type S pitot tube coefficient (Citation 9 in the Bibliography); therefore an assigned (or otherwise known) baseline coefficient value may or may not be valid for a given assembly. The baseline and assembly coefficient values will be identical only when the relative placement of the components in the assembly is such that aerodynamic interference effects are eliminated. Figures 2-6 through 2-8 illustrate interference-free component arrangements for Type S pitot tubes having external tubing diameters between 0.48 and 0.95 cm (3/16 and 3/8 in.). Type S pitot tube assemblies that fail to meet any or all of the specifications of Figures 2-6 through 2-8 shall be calibrated according to the procedure outlined in Sections 4.1.2 through 4.1.5 below, and prior to calibration, the values of the intercomponent spacings (pitot-nozzle, pitot-thermocouple, pitot-probe sheath) shall be measured and recorded.

**NOTE:** Do not use any Type S pitot tube assembly which is constructed such that the impact pressure opening plane of the pitot tube is below the entry plane of the nozzle (see Figure 2-6B).

**4.1.2 Calibration Setup.** If the Type S pitot tube is to be calibrated, one leg of the tube shall be permanently marked A, and the other, B. Calibration shall be done in a flow system having the following essential design features:

4.1.2.1 The flowing gas stream must be confined to a duct of definite cross-sectional area, either circular or rectangular. For circular cross sections, the minimum duct diameter shall be 30.5 cm (12 in.); for rectangular cross sections, the width (shorter side) shall be at least 25.4 cm (10 in.).

4.1.2.2 The cross-sectional area of the calibration duct must be constant over a distance of 10 or more duct diameters. For a rectangular cross section, use an equivalent diameter, calculated from the following equation, to determine the number of duct diameters:

$$D_e = \frac{2LW}{(L + W)}$$

Eq. 2-1

Where:

7.6 cm (3 in.) $H_2O$ velocity pressure registers on the manometer; then, close off the impact opening. The pressure shall remain stable for at least 15 seconds; (2) do the same for the static pressure side, except using suction to obtain the minimum of 7.6 cm (3 in.) $H_2O$. Other leak-check procedures, subject to the approval of the Administrator, may be used.

3.2 Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse. Record all necessary data as shown in the example data sheet (Figure 2-5).

3.3 Measure the velocity head and temperature at the traverse points specified by Method 1. Ensure that the proper differential pressure gauge is being used for the range of Δp values encountered (see Section 2.2). If it is necessary to change to a more sensitive gauge, do so, and remeasure the Δp and temperature readings at each traverse point. Conduct a post-test leak-check (mandatory), as described in Section 3.1 above, to validate the traverse run.

3.4 Measure the static pressure in the stack. One reading is usually adequate.

3.5 Determine the atmospheric pressure.

3.6 Determine the stack gas dry molecular weight. For combustion processes or processes that emit essentially $CO_2$, $O_2$, CO, and $N_2$, use Method 3. For processes emitting essentially air, an analysis need not be conducted; use a dry molecular weight of 29.0. For other processes, other methods, subject to the approval of the Administrator, must be used.

3.7 Obtain the moisture content from Reference Method 4 (or equivalent) or from Method 5.

3.8 Determine the cross-sectional area of the stack or duct at the sampling location. Whenever possible, physically measure the stack dimensions rather than using blueprints.

## 4. CALIBRATION

4.1 **Type S Pitot Tube.** Before its initial use, carefully examine the Type S pitot tube in top, side, and end views to verify that the face openings of the tube are aligned within the specifications illustrated in Figure 2-2 or 2-3. The pitot tube shall not be used if it fails to meet these alignment specifications.

After verifying the face opening alignment, measure and record the following dimensions of the pitot tube: (a) the external tubing diameter (dimension $D_t$, Figure 2-2b); and (b) the base-to-opening plane distances (dimensions $P_A$ and $P_B$, Figure 2-2b). If $D_t$ is between 0.48 and 0.95 cm (3/16 and 3/8 in.), and if $P_A$ and $P_B$ are equal and between 1.05 and 1.50 $D_t$, there are two possible options: (1) the pitot tube may be calibrated according to the procedure outlined in Sections 4.1.2 through 4.1.5 below, or (2) a baseline (isolated tube) coefficient value of 0.84 may be assigned to the pitot tube. Note, however, that if the

**2.5 Barometer.** A mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm (0.1 in.) Hg. See **NOTE** in Method 5, Section 2.1.9.

**2.6 Gas Density Determination Equipment.** Method 3 equipment, if needed (see Section 3.6), to determine the stack gas dry molecular weight, and Reference Method 4 or Method 5 equipment for moisture content determination; other methods may be used subject to approval of the Administrator.

**2.7 Calibration Pitot Tube.** When calibration of the Type S pitot tube is necessary (see Section 4), a standard pitot tube for a reference. The standard pitot tube shall, preferably, have a known coefficient, obtained either (1) directly from the National Bureau of Standards, Route 70 S, Quince Orchard Road, Gaithersburg, Maryland, or (2) by calibration against another standard pitot tube with an NBS-traceable coefficient. Alternatively, a standard pitot tube designed according to the criteria given in Sections 2.7.1 through 2.7.5 below and illustrated in Figure 2-4 (see also Citations 7, 8, and 17 in the Bibliography) may be used. Pitot tubes designed according to these specifications will have baseline coefficients of about 0.99 ± 0.01.

2.7.1 Hemispherical (shown in Figure 2-4) ellipsoidal, or conical tip.

2.7.2 A minimum of six diameters straight run (based upon D, the external diameter of the tube) between the tip and the static pressure holes.

2.7.3 A minimum of eight diameters straight run between the static pressure holes and the centerline of the external tube, following the 90-degree bend.

2.7.4 Static pressure holes of equal size (approximately 0.1 D), equally spaced in a piezometer ring configuration.

2.7.5 Ninety-degree bend, with curved or mitered junction.

**2.8 Differential Pressure Gauge for Type S Pitot Tube Calibration.** An inclined manometer or equivalent. If the single-velocity calibration technique is employed (see Section 4.1.2.3), the calibration differential pressure gauge shall be readable to the nearest 0.13 mm (0.005 in.) $H_2O$. For multivelocity calibrations, the gauge shall be readable to the nearest 0.13 mm (0.005 in.) $H_2O$ for $\Delta p$ values between 1.3 and 25 mm (0.05 and 1.0 in.) $H_2O$, and to the nearest 1.3 mm (0.05 in.) $H_2O$ for $\Delta p$ values above 25 mm (1.0 in.) $H_2O$. A special, more sensitive gauge will be required to read $\Delta p$ values below 1.3 mm (0.05 in.) $H_2O$ (see Citation 18 in the Bibliography).

## 3. PROCEDURE

3.1 Set up the apparatus as shown in Figure 2-1. Capillary tubing or surge tanks installed between the manometer and pitot tube may be used to dampen $\Delta p$ fluctuations. It is recommended, but not required, that a pretest leak-check be conducted as follows: (1) blow through the pitot impact opening until at least

$$T = \frac{\sum_{i=1}^{n} \sqrt{\Delta p_i + K}}{\sum_{i=1}^{n} \sqrt{\Delta p_i}}$$

Where:

$\Delta p_i$ = Individual velocity head reading at a traverse point, mm (in.) $H_2O$.

n = Total number of traverse points.

K = 0.13 mm $H_2O$ when metric units are used and 0.005 in. $H_2O$ when English units are used.

If T is greater than 1.05, the velocity head data are unacceptable and a more sensitive differential pressure gauge must be used.

**NOTE:** If differential pressure gauges other than inclined manometers are used (e.g., magnehelic gauges), their calibration must be checked after each test series. To check the calibration of a differential pressure gauge, compare Δp readings of the gauge with those of a gauge-oil manometer at a minimum of three points, approximately representing the range of Δp values in the stack. If, at each point, the values of Δp as read by the differential pressure gauge and gauge-oil manometer agree to within 5 percent, the differential pressure gauge shall be considered to be in proper calibration. Otherwise, the test series shall either be voided, or procedures to adjust the measured Δp values and final results shall be used, subject to the approval of the Administrator.

**2.3 Temperature Gauge.** A thermocouple, liquid-filled bulb thermometer, bimetallic thermometer, mercury-in-glass thermometer, or other gauge capable of measuring temperature to within 1.5 percent of the minimum absolute stack temperature. The temperature gauge shall be attached to the pitot tube such that the sensor tip does not touch any metal; the gauge shall be in an interference-free arrangement with respect to the pitot tube face openings (see Figure 2-1 and also Figure 2-7 in Section 4). Alternative positions may be used if the pitot tube-temperature gauge system is calibrated according to the procedure of Section 4. Provided that a difference of not more than 1 percent in the average velocity measurement is introduced, the temperature gauge need not be attached to the pitot tube; this alternative is subject to the approval of the Administrator.

**2.4 Pressure Probe and Gauge.** A piezometer tube and mercury- or water-filled U-tube manometer capable of measuring stack pressure to within 2.5 mm (0.1 in.) Hg. The static tap of a standard type pitot tube or one leg of a Type S pitot tube with the face opening planes positioned parallel to the gas flow may also be used as the pressure probe.

number shall be permanently marked or engraved on the body of the tube. A standard pitot tube may be used instead of a Type S, provided that it meets the specifications of Sections 2.7 and 4.2; note, however, that the static and impact pressure holes of standard pitot tubes are susceptible to plugging in particulate-laden gas streams. Therefore, whenever a standard pitot tube is used to perform a traverse, adequate proof must be furnished that the openings of the pitot tube have not plugged up during the traverse period; this can be done by taking a velocity head ($\Delta p$) reading at the final traverse point, cleaning out the impact and static holes of the standard pitot tube by "back-purging" with pressurized air, and then taking another $\Delta p$ reading. If the $\Delta p$ readings made before and after the air purge are the same (±5 percent), the traverse is acceptable. Otherwise, reject the run. Note that if $\Delta p$ at the final traverse point is unsuitably low, another point may be selected. If "back-purging" at regular intervals is part of the procedure, then comparative $\Delta p$ readings shall be taken, as above, for the last two back purges at which suitably high $\Delta p$ readings are observed.

**2.2 Differential Pressure Gauge.** An inclined manometer or equivalent device. Most sampling trains are equipped with a 10-in. (water column) inclined-vertical manometer, having 0.01-in. $H_2O$ divisions on the 0- to 1-in. inclined scale, and 0.1-in. $H_2O$ divisions on the 1- to 10-in. vertical scale. This type of manometer (or other gauge of equivalent sensitivity) is satisfactory for the measurement of $\Delta p$ values as low as 1.3 mm (0.05 in.) $H_2O$. However, a differential pressure gauge of greater sensitivity shall be used (subject to the approval of the Administrator), if any of the following is found to be true: (1) the arithmetic average of all $\Delta p$ readings at the traverse points in the stack is less than 1.3 mm (0.05 in.) $H_2O$; (2) for traverses of 12 or more points, more than 10 percent of the individual $\Delta p$ readings are below 1.3 mm (0.05 in.) $H_2O$; (3) for traverses of fewer than 12 points, more than one $\Delta p$ reading is below 1.3 mm (0.05 in.) $H_2O$. Citation 18 in the Bibliography describes commercially available instrumentation for the measurement of low-range gas velocities.

As an alternative to criteria (1) through (3) above, the following calculation may be performed to determine the necessity of using a more sensitive differential pressure gauge:

**EMISSION MEASUREMENT TECHNICAL INFORMATION CENTER**
**NSPS TEST METHOD**

# Method 2 - Determination of Stack Gas Velocity and Volumetric Flow Rate (Type S Pitot Tube)

## 1. PRINCIPLE AND APPLICABILITY

**1.1 Principle.** The average gas velocity in a stack is determined from the gas density and from measurement of the average velocity head with a Type S (Stausscheibe or reverse type) pitot tube.

**1.2 Applicability.** This method is applicable for measurement of the average velocity of a gas stream and for quantifying gas flow.

This procedure is not applicable at measurement sites that fail to meet the criteria of Method 1, Section 2.1. Also, the method cannot be used for direct measurement in cyclonic or swirling gas streams; Section 2.4 of Method 1 shows how to determine cyclonic or swirling flow conditions. When unacceptable conditions exist, alternative procedures, subject to the approval of the Administrator, U.S. Environmental Protection Agency, must be employed to make accurate flow rate determinations; examples of such alternative procedures are: (1) to install straightening vanes; (2) to calculate the total volumetric flow rate stoichiometrically, or (3) to move to another measurement site at which the flow is acceptable.

## 2. APPARATUS

Specifications for the apparatus are given below. Any other apparatus that has been demonstrated (subject to approval of the Administrator) to be capable of meeting the specifications will be considered acceptable.

**2.1 Type S Pitot Tube.** Pitot tube made of metal tubing (e.g., stainless steel) as shown in Figure 2-1. It is recommended that the external tubing diameter (dimension $D_t$, Figure 2-2b) be between 0.48 and 0.95 cm (3/16 and 3/8 inch). There shall be an equal distance from the base of each leg of the pitot tube to its face-opening plane (dimensions $P_A$ and $P_B$, Figure 2-2b); it is recommended that this distance be between 1.05 and 1.50 times the external tubing diameter. The face openings of the pitot tube shall, preferably, be aligned as shown in Figure 2-2; however, slight misalignments of the openings are permissible (see Figure 2-3).

The Type S pitot tube shall have a known coefficient, determined as outlined in Section 4. An identification number shall be assigned to the pitot tube; this

# Appendix G.2

## Sampling & Analysis Methods

## EPA Method 2

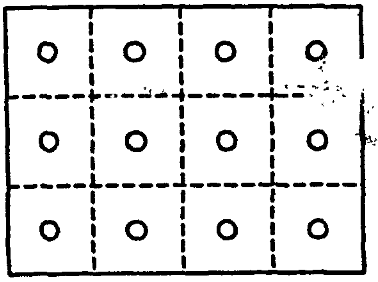


Figure 1-4. Example showing rectangular stack cross section divided into 12 equal areas, with a traverse point at centroid of each area.

| Traverse Point | Distance % of diameter |
|---|---|
| 1 | 4.4 |
| 2 | 14.7 |
| 3 | 29.5 |
| 4 | 70.5 |
| 5 | 85.3 |
| 6 | 95.6 |

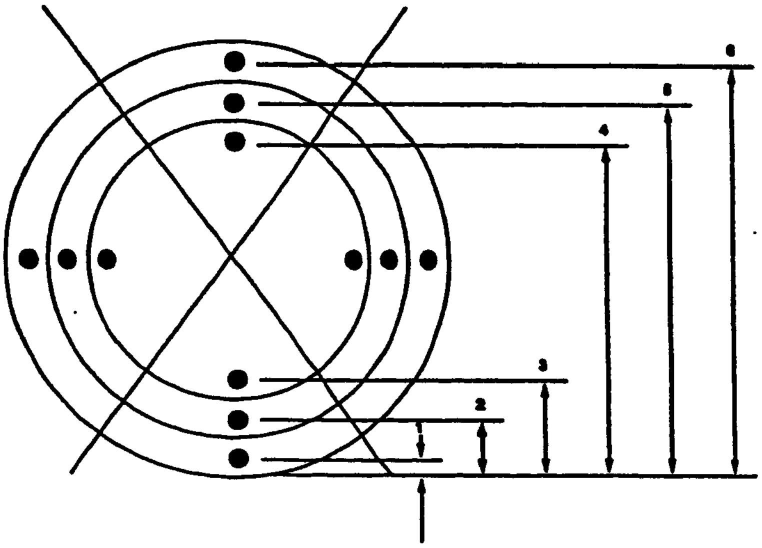


Figure 1-3. Example showing circular stack cross section divided into 12 equal areas, with location of traverse points indicated.

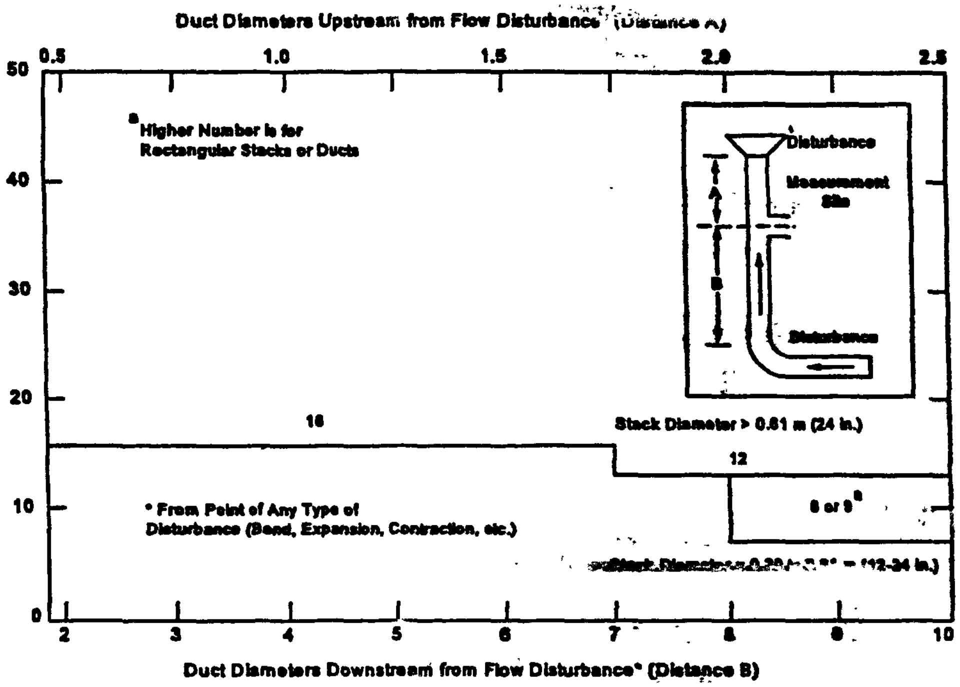


Figure 1-2. Minimum number of traverse points for velocity (nonparticulate) traverses.

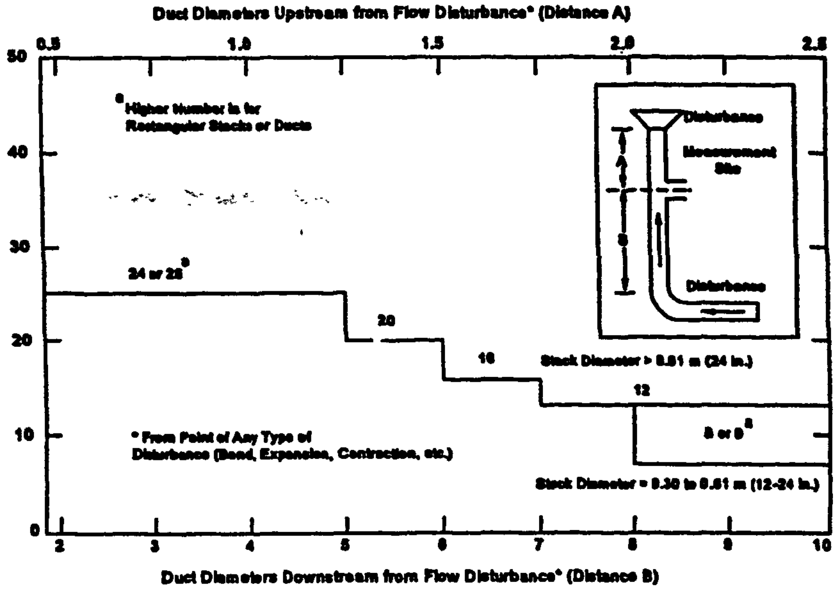


Figure 1-1. Minimum number of traverse points for particulate traverses.

| | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 15 . . . . | | | | | | | | 95.1 | 89.1 | 83.5 | 78.2 | 72.8 |
| 16 . . . . | | | | | | | | 98.4 | 92.5 | 87.1 | 82.0 | 77.0 |
| 17 . . . . | | | | | | | | | 95.6 | 90.3 | 85.4 | 80.6 |
| 18 . . . . | | | | | | | | | 98.6 | 93.3 | 88.4 | 83.9 |
| 19 . . . . | | | | | | | | | | 96.1 | 91.3 | 86.8 |
| 20 . . . . | | | | | | | | | | 98.7 | 94.0 | 89.5 |
| 21 . . . . | | | | | | | | | | | 96.5 | 92.1 |
| 22 . . . . | | | | | | | | | | | 98.9 | 94.5 |
| 23 . . . . | | | | | | | | | | | | 96.8 |
| 24 . . . . | | | | | | | | | | | | 98.9 |

### TABLE 1-2
### LOCATION OF TRAVERSE POINTS IN CIRCULAR STACKS
### (Percent of stack diameter from inside wall to traverse point)

| Traverse Point Number on a Diameter | Number of traverse points on a diameter | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 2 | 4 | 6 | 8 | 10 | 12 | 14 | 16 | 18 | 20 | 22 | 24 |
| 1 . . . . . . | 14.6 | 6.7 | 4.4 | 3.2 | 2.6 | 2.1 | 1.8 | 1.6 | 1.4 | 1.3 | 1.1 | 1.1 |
| 2 . . . . . . | 85.4 | 25.0 | 14.6 | 10.5 | 8.2 | 6.7 | 5.7 | 4.9 | 4.4 | 3.9 | 3.5 | 3.2 |
| 3 . . . . . . | | 75.0 | 29.6 | 19.4 | 14.6 | 11.8 | 9.9 | 8.5 | 7.5 | 6.7 | 6.0 | 5.5 |
| 4 . . . . . . | | 93.3 | 70.4 | 32.3 | 22.6 | 17.7 | 14.6 | 12.5 | 10.9 | 9.7 | 8.7 | 7.9 |
| 5 . . . . . . | | | 85.4 | 67.7 | 34.2 | 25.0 | 20.1 | 16.9 | 14.6 | 112.9 | 11.6 | 10.5 |
| 6 . . . . . . | | | 95.6 | 80.6 | 65.8 | 35.6 | 26.9 | 22.0 | 18.8 | 16.5 | 14.6 | 13.2 |
| 7 . . . . . . | | | | 89.5 | 77.4 | 64.4 | 36.6 | 28.3 | 23.6 | 20.4 | 18.0 | 16.1 |
| 8 . . . . . . | | | | 96.8 | 85.4 | 75.0 | 63.4 | 37.5 | 29.6 | 25.0 | 21.8 | 19.4 |
| 9 . . . . . . | | | | | 91.8 | 82.3 | 73.1 | 62.5 | 38.2 | 30.6 | 26.2 | 23.0 |
| 10 . . . . . | | | | | 97.4 | 88.2 | 79.9 | 71.7 | 61.8 | 38.8 | 31.5 | 27.2 |
| 11 . . . . . | | | | | | 93.3 | 85.4 | 78.0 | 70.4 | 61.2 | 39.3 | 32.3 |
| 12 . . . . . | | | | | | 97.9 | 90.1 | 83.1 | 76.4 | 69.4 | 60.7 | 39.8 |
| 13 . . . . . | | | | | | | 94.3 | 87.5 | 81.2 | 75.0 | 68.5 | 60.2 |
| 14 . . . . . | | | | | | | 98.2 | 91.5 | 85.4 | 79.6 | 73.8 | 67.7 |

Table 1-1. CROSS-SECTION LAYOUT FOR RECTANGULAR STACKS

| Number of traverse points | Matrix layout |
|---|---|
| 9 | 3x3 |
| 12 | 4x3 |
| 16 | 4x4 |
| 20 | 5x4 |
| 25 | 5x5 |
| 30 | 6x5 |
| 36 | 6x6 |
| 42 | 7x6 |
| 49 | 7x7 |

August 1983.

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13. Smith, W.S. and D.J. Grove. A Proposed Extension of EPA Method 1 Criteria. Pollution Engineering. XV (8):36-37.

2.5.6.2 To ensure that the gas flow is parallel to the central axis of the test section, follow the procedure in Section 2.4 for cyclonic flow determination to measure the gas flow angles at the centroid of the test section from two test ports located 90° apart. The gas flow angle measured in each port must be ±2° of 0°. Straightening vanes should be installed, if necessary, to meet this criterion.

2.5.6.3 **Pitch Angle Calibration.** Perform a calibration traverse according to the manufacturer's recommended protocol in 5° increments for angles from -60° to +60° at one velocity in each of the two ranges specified above. Average the pressure ratio values obtained for each angle in the two flow ranges, and plot a calibration curve with the average values of the pressure ratio (or other suitable measurement factor as recommended by the manufacturer) versus the pitch angle. Draw a smooth line through the data points. Plot also the data values for each traverse point. Determine the differences between the measured datavalues and the angle from the calibration curve at the same pressure ratio. The difference at each comparison must be within 2° for angles between 0° and 40° and within 3° for angles between 40° and 60°.

2.5.6.4 **Yaw Angle Calibration.** Mark the three-dimensional probe to allow the determination of the yaw position of the probe. This is usually a line extending the length of the probe and aligned with the impact opening. To determine the accuracy of measurements of the yaw angle, only the zero or null position need be calibrated as follows: Place the directional probe in the test section, and rotate the probe until the zero position is found. With a protractor or other angle measuring device, measure the angle indicated by the yaw angle indicator on the three-dimensional probe. This should be within 2° of 0°. Repeat this measurement for any other points along the length of the pitot where yaw angle measurements could be read in order to account for variations in the pitot markings used to indicate pitot head positions.

## BIBLIOGRAPHY

1. Determining Dust Concentration in a Gas Stream, ASME Performance Test Code No. 27. New York. 1957.

2. DeVorkin, Howard, et al. Air Pollution Source Testing Manual. Air Pollution Control District. Los Angeles, CA. November 1963.

3. Methods for Determining of Velocity, Volume, Dust and Mist Content of Gases. Western Precipitation Division of Joy

Where:

$R_i$ = resultant angle at traverse point i, degree.
$Y_i$ = yaw angle at traverse point i, degree.
$P_i$ = pitch angle at traverse point i, degree.

2.5.4.2 Calculate the average resultant for the measurements:

$$\overline{R} = \frac{\Sigma R_i}{n}$$

Eq. 1-3

Where:

$R_{avg}$ = average resultant angle, degree.
n = total number of traverse points.

2.5.4.3 Calculate the standard deviations:

$$S_d = \sqrt{\frac{\sum_{i=1}^{n} (R_i - \overline{R})^2}{(n-1)}}$$

Eq. 1-4

Where:

$S_d$ = standard deviation, degree.

2.5.5 The measurement location is acceptable if $R_{avg} \leq 20°$ and $S_d \leq 10°$.

2.5.6 **Calibration.** Use a flow system as described in Sections 4.1.2.1 and 4.1.2.2 of Method 2. In addition, the flow system shall have the capacity to generate two test-section velocities: one between 365 and 730 m/min (1200 and 2400 ft/min) and one between 730 and 1100 m/min (2400 and 3600 ft/min).

2.5.6.1 Cut two entry ports in the test section. The axes through the entry ports shall be perpendicular to each other and intersect in the centroid of the test section. The ports should be elongated slots parallel to the axis of the test section and of sufficient length to allow measurement of pitch angles while maintaining the pitot head position at the test-section centroid. To facilitate alignment of the directional probe during calibration, the test section should be constructed of plexiglass or some other transparent material. All calibration measurements should be made at the same point in the test section, preferably at the centroid of the test section.

the location and layout of the traverse points. If the measurement location is determined to be acceptable according to the criteria in this alternative procedure, use the same traverse point number and locations for sampling and velocity measurements.

**2.5.3 Measurement Procedure.**

**2.5.3.1** Prepare the directional probe and differential pressure gauges as recommended by the manufacturer. Capillary tubing or surge tanks may be used to dampen pressure fluctuations. It is recommended, but not required, that a pretest leak check be conducted. To perform a leak check, pressurize or use suction on the impact opening until a reading of at least 7.6 cm (3 in.) $H_2O$ registers on the differential pressure gauge, then plug the impact opening. The pressure of a leak-free system will remain stable for at least 15 seconds.

**2.5.3.2** Level and zero the manometers. Since the manometer level and zero may drift because of vibrations and temperature changes, periodically check the level and zero during the traverse.

**2.5.3.3** Position the probe at the appropriate locations in the gas stream, and rotate until zero deflection is indicated for the yaw angle pressure gauge. Determine and record the yaw angle. Record the pressure gauge readings for the pitch angle, and determine the pitch angle from the calibration curve. Repeat this procedure for each traverse point. Complete a "back-purge" of the pressure lines and the impact openings prior to measurements of each traverse point.

A post-test check as described in Section 2.5.3.1 is required. If the criteria for a leak-free system are not met, repair the equipment, and repeat the flow angle measurements.

**2.5.4** Calculate the resultant angle at each traverse point, the average resultant angle, and the standard deviation using the following equations. Complete the calculations retaining at least one extra significant figure beyond that of the acquired data. Round the values after the final calculations.

**2.5.4.1** Calculate the resultant angle at each traverse point:

$$R_i = \text{arc cosine}[(\text{cosine}Y_i)(\text{cosine}P_i)]$$

Eq. 1-2

described above.

**2.5 Alternative Measurement Site Selection Procedure.** This alternative applies to sources where measurement locations are less than 2 equivalent or duct diameters downstream or less than one-half duct diameter upstream from a flow disturbance. The alternative should be limited to ducts larger than 24 in. in diameter where blockage and wall effects are minimal. A directional flow-sensing probe is used to measure pitch and yaw angles of the gas flow at 40 or more traverse points; the resultant angle is calculated and compared with acceptable criteria for mean and standard deviation.

**NOTE:** Both the pitch and yaw angles are measured from a line passing through the traverse point and parallel to the stack axis. The pitch angle is the angle of the gas flow component in the plane that INCLUDES the traverse line and is parallel to the stack axis. The yaw angle is the angle of the gas flow component in the plane PERPENDICULAR to the traverse line at the traverse point and is measured from the line passing through the traverse point and parallel to the stack axis.

**2.5.1 Apparatus.**

**2.5.1.1 Directional Probe.** Any directional probe, such as United Sensor Type DA Three-Dimensional Directional Probe, capable of measuring both the pitch and yaw angles of gas flows is acceptable. (**NOTE:** Mention of trade name or specific products does not constitute endorsement by the U.S. Environmental Protection Agency.) Assign an identification number to the directional probe, and permanently mark or engrave the number on the body of the probe. The pressure holes of directional probes are susceptible to plugging when used in particulate-laden gas streams. Therefore, a system for cleaning the pressure holes by "back-purging" with pressurized air is required.

**2.5.1.2 Differential Pressure Gauges.** Inclined manometers, U-tube manometers, or other differential pressure gauges (e.g., magnehelic gauges) that meet the specifications described in Method 2, Section 2.2.

**NOTE:** If the differential pressure gauge produces both negative and positive readings, then both negative and positive pressure readings shall be calibrated at a minimum of three points as specified in Method 2, Section 2.2.

**2.5.2 Traverse Points.** Use a minimum of 40 traverse points for circular ducts and 42 points for rectangular ducts for the gas flow angle determinations. Follow Section 2.3 and Table 1-1 or 1-2 for

1, determine the grid configuration. Divide the stack cross-section into as many equal rectangular elemental areas as traverse points, and then locate a traverse point at the centroid of each equal area according to the example in Figure 1-4.
If the tester desires to use more than the minimum number of traverse points, expand the "minimum number of traverse points" matrix (see Table 1-1) by adding the extra traverse points along one or the other or both legs of the matrix; the final matrix need not be balanced. For example, if a 4 x 3 "minimum number of points" matrix were expanded to 36 points, the final matrix could be 9 x 4 or 12 x 3, and would not necessarily have to be 6 x 6. After constructing the final matrix, divide the stack cross-section into as many equal rectangular, elemental areas as traverse points, and locate a traverse point at the centroid of each equal area. The situation of traverse points being too close to the stack walls is not expected to arise with rectangular stacks. If this problem should ever arise, the Administrator must be contacted for resolution of the matter.

**2.4 Verification of Absence of Cyclonic Flow.** In most stationary sources, the direction of stack gas flow is essentially parallel to the stack walls. However, cyclonic flow may exist (1) after such devices as cyclones and inertial demisters following venturi scrubbers, or (2) in stacks having tangential inlets or other duct configurations which tend to induce swirling; in these instances, the presence or absence of cyclonic flow at the sampling location must be determined. The following techniques are acceptable for this determination. Level and zero the manometer. Connect a Type S pitot tube to the manometer. Position the Type S pitot tube at each traverse point, in succession, so that the planes of the face openings of the pitot tube are perpendicular to the stack cross-sectional plane; when the Type S pitot tube is in this position, it is at "0° reference." Note the differential pressure ($\Delta p$) reading at each traverse point. If a null (zero) pitot reading is obtained at 0° reference at a given traverse point, an acceptable flow condition exists at that point. If the pitot reading is not zero at 0° reference, rotate the pitot tube (up to ±90° yaw angle), until a null reading is obtained. Carefully determine and record the value of the rotation angle ($\alpha$) to the nearest degree. After the null technique
has been applied at each traverse point, calculate the average of the absolute values of $\alpha$; assign $\alpha$ values of 0° to those points for which no rotation was required, and include these in the overall average. If the average value of $\alpha$ is greater than 20°, the overall flow condition in the stack is unacceptable, and alternative methodology, subject to the approval of the Administrator, must be used to perform accurate sample and velocity traverses. The alternative procedure described in Section 2.5 may be used to determine the rotation angles in lieu of the procedure

**2.2.2 Velocity (Non-Particulate) Traverses.** When velocity or volumetric flow rate is to be determined (but not particulate matter), the same procedure as that used for particulate traverses (Section 2.2.1) is followed, except that Figure 1-2 may be used instead of Figure 1-1.

**2.3 Cross-Sectional Layout and Location of Traverse Points.**

**2.3.1 Circular Stacks.** Locate the traverse points on two perpendicular diameters according to Table 1-2 and the example shown in Figure 1-3. Any equation (for examples, see Citations 2 and 3 in the Bibliography) that gives the same values as those in Table 1-2 may be used in lieu of Table 1-2.

For particulate traverses, one of the diameters must be in a plane containing the greatest expected concentration variation, e.g., after bends, one diameter shall be in the plane of the bend. This requirement becomes less critical as the distance from the disturbance increases; therefore, other diameter locations may be used, subject to the approval of the Administrator.

In addition, for stacks having diameters greater than 0.61 m (24 in.), no traverse points shall be within 2.5 centimeters (1.00 in.) of the stack walls; and for stack diameters equal to or less than 0.61 m (24 in.), no traverse points shall be located within 1.3 cm (0.50 in.) of the stack walls. To meet these criteria, observe the procedures given below.

**2.3.1.1 Stacks With Diameters Greater Than 0.61 m (24 in.).** When any of the traverse points as located in Section 2.3.1 fall within 2.5 cm (1.00 in.) of the stack walls, relocate them away from the stack walls to: (1) a distance of 2.5 cm (1.00 in.); or (2) a distance equal to the nozzle inside diameter, whichever is larger. These relocated traverse points (on each end of a diameter) shall be the "adjusted" traverse points. Whenever two successive traverse points are combined to form a single adjusted traverse point, treat the adjusted point as two separate traverse points, both in the sampling (or velocity measurement) procedure, and in recording the data.

**2.3.1.2 Stacks With Diameters Equal To or Less Than 0.61 m (24 in.).** Follow the procedure in Section 2.3.1.1, noting only that any "adjusted" points should be relocated away from the stack walls to: (1) a distance of 1.3 cm (0.50 in.); or (2) a distance equal to the nozzle inside diameter, whichever is larger.

**2.3.2 Rectangular Stacks.** Determine the number of traverse points as explained in Sections 2.1 and 2.2 of this method. From Table 1-

$$D_e = \frac{2LW}{(L + W)}$$

Eq. 1-1

Where
L = Length and W = width.

An alternative procedure is available for determining the acceptability of a measurement location not meeting the criteria above. This procedure, determination of gas flow angles at the sampling points and comparing the results with acceptability criteria, is described in Section 2.5.

## 2.2 Determining the Number of Traverse Points.

**2.2.1 Particulate Traverses.** When the eight- and two-diameter criterion can be met, the minimum number of traverse points shall be: (1) twelve, for circular or rectangular stacks with diameters (or equivalent diameters) greater than 0.61 meter (24 in.); (2) eight, for circular stacks with diameters between 0.30 and 0.61 meter (12 and 24 in.); and (3) nine, for rectangular stacks with equivalent diameters between 0.30 and 0.61 meter (12 and 24 in.).

When the eight- and two-diameter criterion cannot be met, the minimum number of traverse points is determined from Figure 1-1. Before referring to the figure, however, determine the distances from the chosen measurement site to the nearest upstream and downstream disturbances, and divide each distance by the stack diameter or equivalent diameter, to determine the distance in terms of the number of duct diameters. Then, determine from Figure 1-1 the minimum number of traverse points that corresponds: (1) to the number of duct diameters upstream; and (2) to the number of diameters downstream. Select the higher of the two minimum numbers of traverse points, or a greater value, so that for circular stacks the number is a multiple of 4, and for rectangular stacks, the number is one of those shown in Table 1-1.

EMISSION MEASUREMENT TECHNICAL INFORMATION CENTER
NSPS TEST METHOD

# Method 1 - Sample and Velocity Traverses for Stationary Sources

## 1. PRINCIPLE AND APPLICABILITY

**1.1 Principle.** To aid in the representative measurement of pollutant emissions and/or total volumetric flow rate from a stationary source, a measurement site where the effluent stream is flowing in a known direction is selected, and the cross-section of the stack is divided into a number of equal areas. A traverse point is then located within each of these equal areas.

**1.2 Applicability.** This method is applicable to flowing gas streams in ducts, stacks, and flues. The method cannot be used when: (1) flow is cyclonic or swirling (see Section 2.4), (2) a stack is smaller than about 0.30 meter (12 in.) in diameter, or 0.071 $m^2$ (113 $in.^2$) in cross-sectional area, or (3) the measurement site is less than two stack or duct diameters downstream or less than a half diameter upstream from a flow disturbance.

The requirements of this method must be considered before construction of a new facility from which emissions will be measured; failure to do so may require subsequent alterations to the stack or deviation from the standard procedure. Cases involving variants are subject to approval by the Administrator, U.S. Environmental Protection Agency.

## 2. PROCEDURE

**2.1 Selection of Measurement Site.** Sampling or velocity measurement is performed at a site located at least eight stack or duct diameters downstream and two diameters upstream from any flow disturbance such as a bend, expansion, or contraction in the stack, or from a visible flame. If necessary, an alternative location may be selected, at a position at least two stack or duct diameters downstream and a half diameter upstream from any flow disturbance. For a rectangular cross section, an equivalent diameter ($D_e$) shall be calculated from the following equation, to determine the upstream and downstream distances:

# Appendix G.1

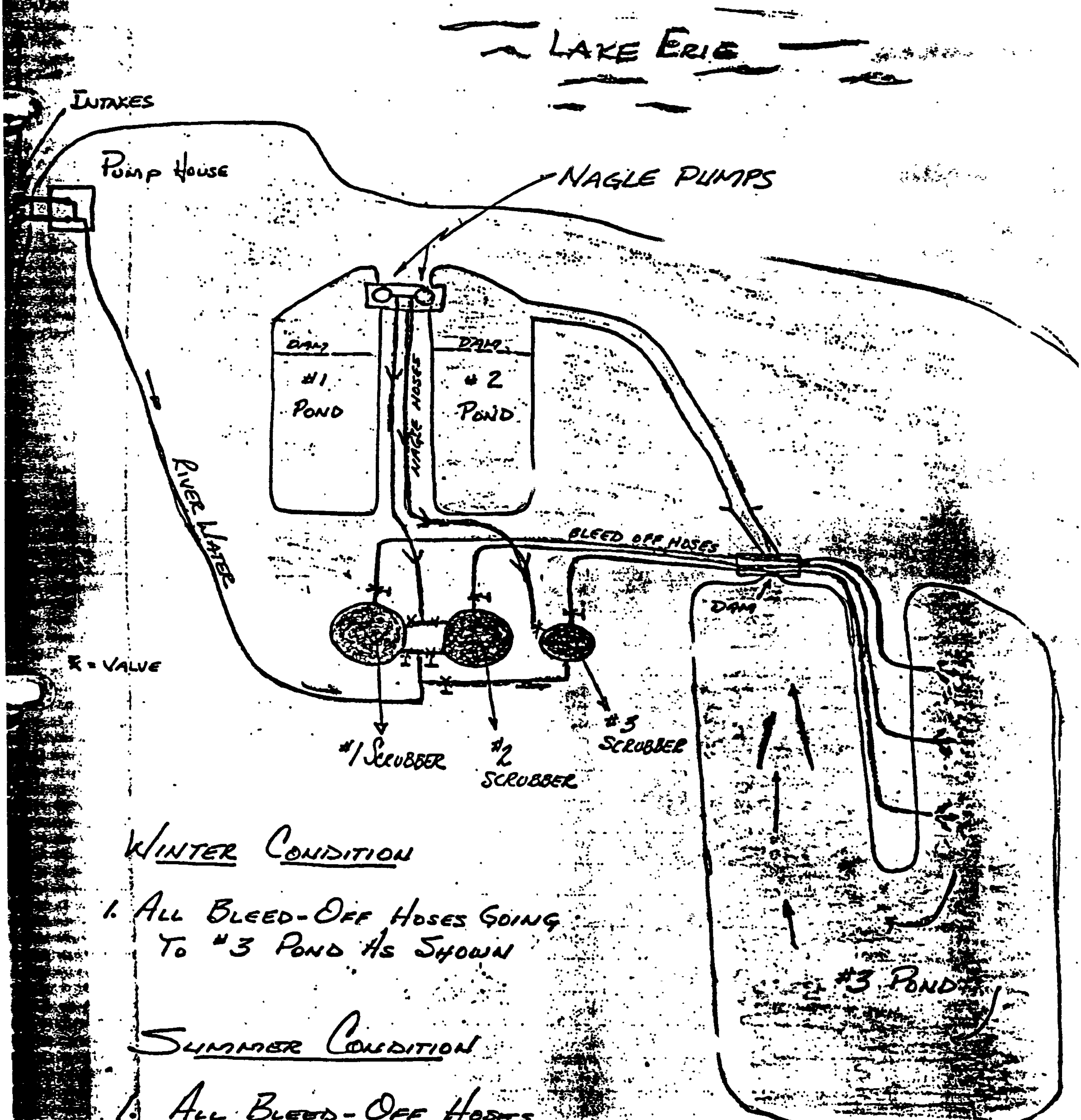
## Sampling & Analysis Methods

## EPA Method 1

# APPENDIX G

## SAMPLING & ANALYSIS METHODS

(EPA Methods 1, 2, 3A, 23 (Proposed Revisions), & 25A)

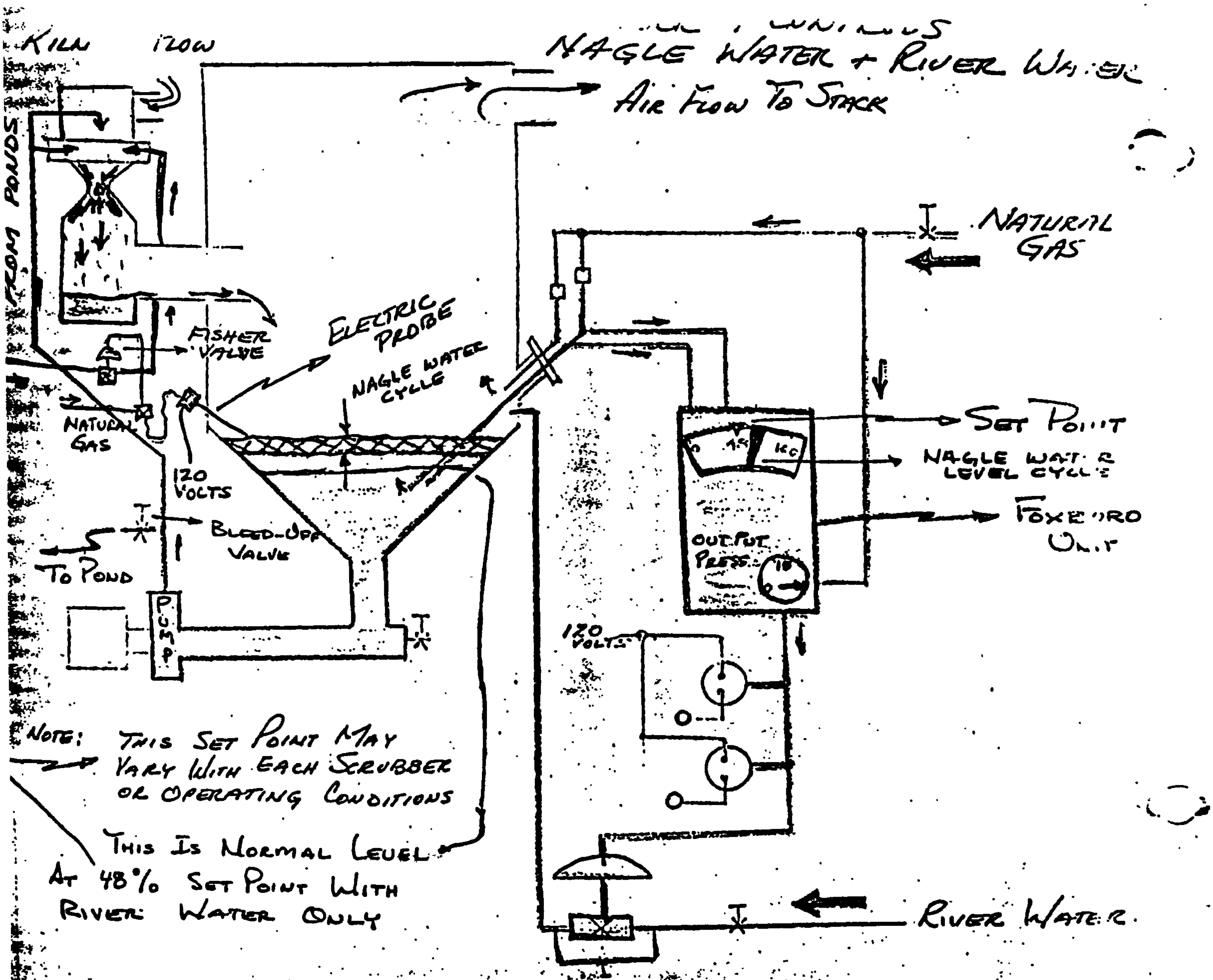


WINTER CONDITION

1. ALL BLEED-OFF HOSES GOING TO #3 POND AS SHOWN

SUMMER CONDITION

1. ALL BLEED-OFF HOSES WILL BE GOING TO EITHER #1 OR #2 POND. #3 POND WILL BE CLEANED

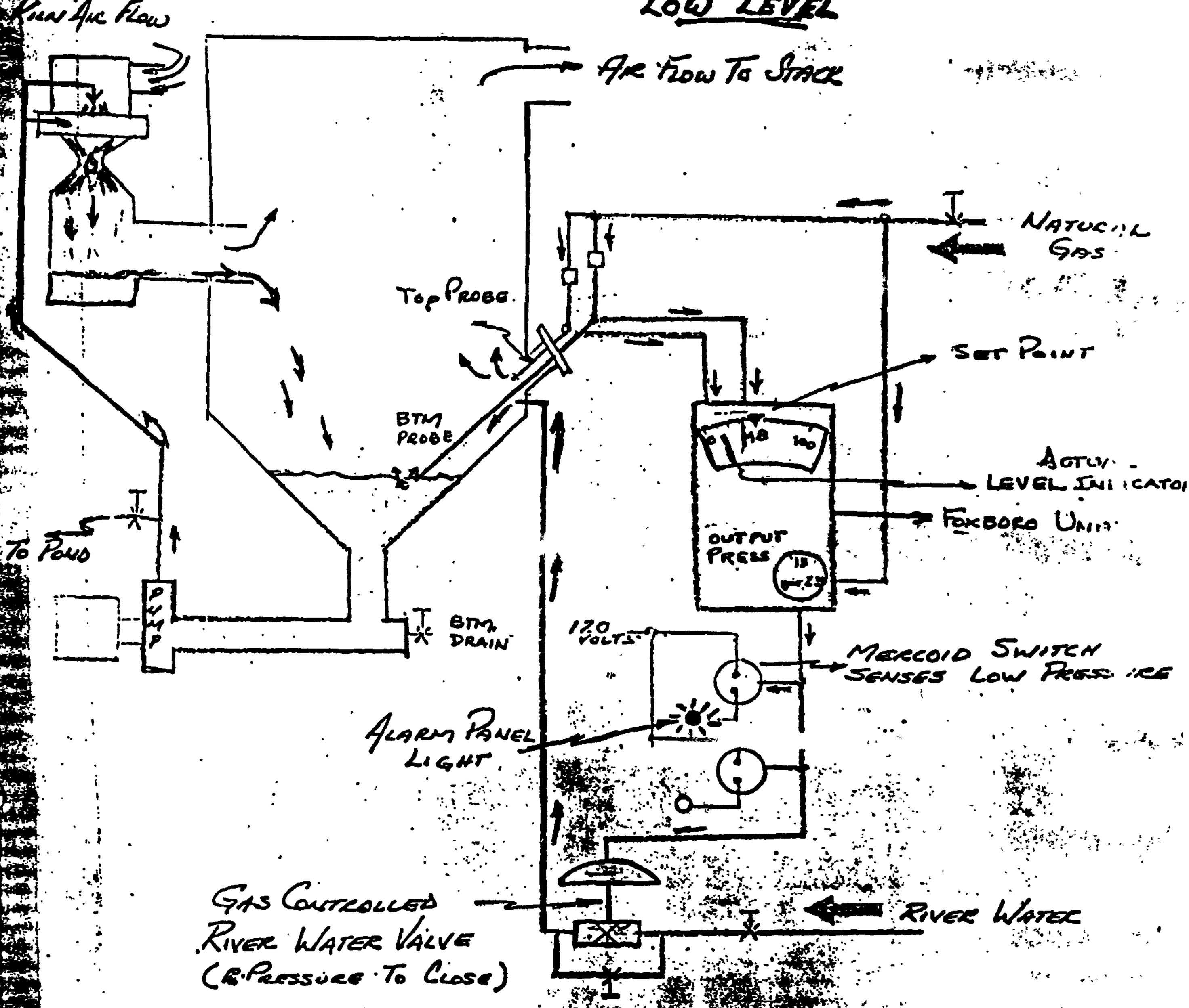


NOTE: THIS SET POINT MAY VARY WITH EACH SCRUBBER OR OPERATING CONDITIONS

THIS IS NORMAL LEVEL AT 48% SET POINT WITH RIVER WATER ONLY

THE FISHER VALVE CONTROLLING THE NAGLE WATER TO THE SCRUBBERS IS ELECTRICALLY CONTROLLED. WHEN THE SCRUBBER WATER LEVEL TOUCHES THE END OF THE NAGLE PROBE, THE NAGLE FISHER VALVE TURNS OFF. THE BLEED-OFF WATER ALLOWS THE SCRUBBER LEVEL TO DROP AWAY FROM THE NAGLE PROBE. AFTER A PRE-DETERMINED TIME, THE PROBE TURNS ON THE FISHER VALVE ALLOWING NAGLE WATER TO FLOW INTO THE SCRUBBER UNTIL THE WATER LEVEL REACHES THE PROBE TURNING OFF THE NAGLE VALVE, THUS STARTING THE CYCLE OVER. LOW LEVEL ALARM IS ACTUATED THE SAME W AS STATED FOR THE RIVER WATER. AFTER A PRE-DETERMINED TIME THE WATER IS STILL TOUCHING THE PROBE, A HIGH LEVEL ALARM WILL COME ON. 8.

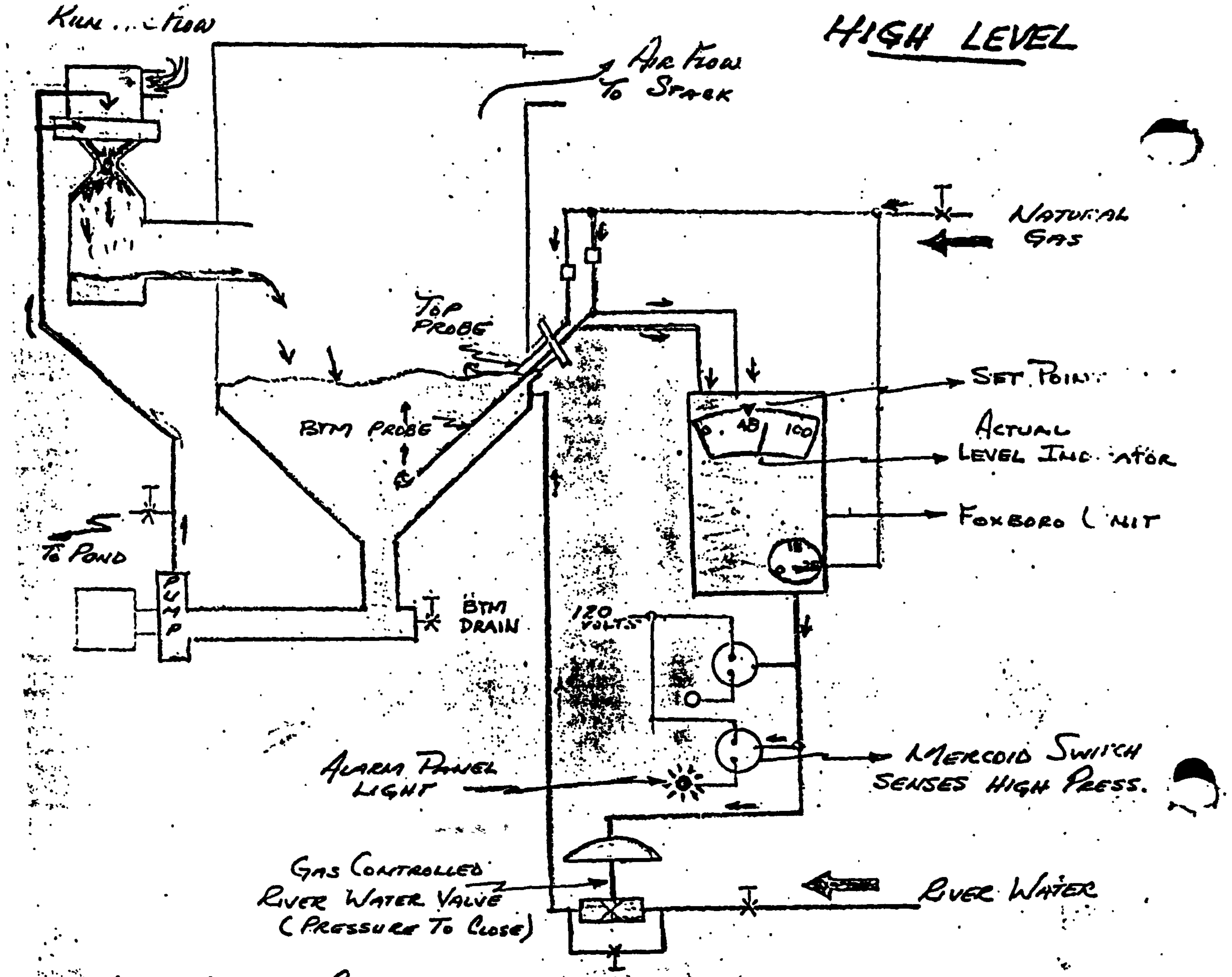
## LOW LEVEL



## LOW LEVEL CONDITION

DIFFERENCE IN GAS PRESSURE BETWEEN 2 PROBES IS SMALL AND INDICATED AS LOW % OR LOW LEVEL. THE FOXBORO UNIT SIGNALS A LOW OR ZERO OUT PUT PRESSURE WHICH IS SENSED BY THE MERCOID SWITCH WHICH TURNS ON THE LOW LEVEL LIGHT ON THE ALARM PANEL IN THE CONTROL ROOM. THE LOW OUTPUT PRESSURE ALSO CAUSES THE RIVER WATER CONTROL VALVE TO OPEN ALLOWING RIVER WATER TO ENTER THE SCRUBBER.

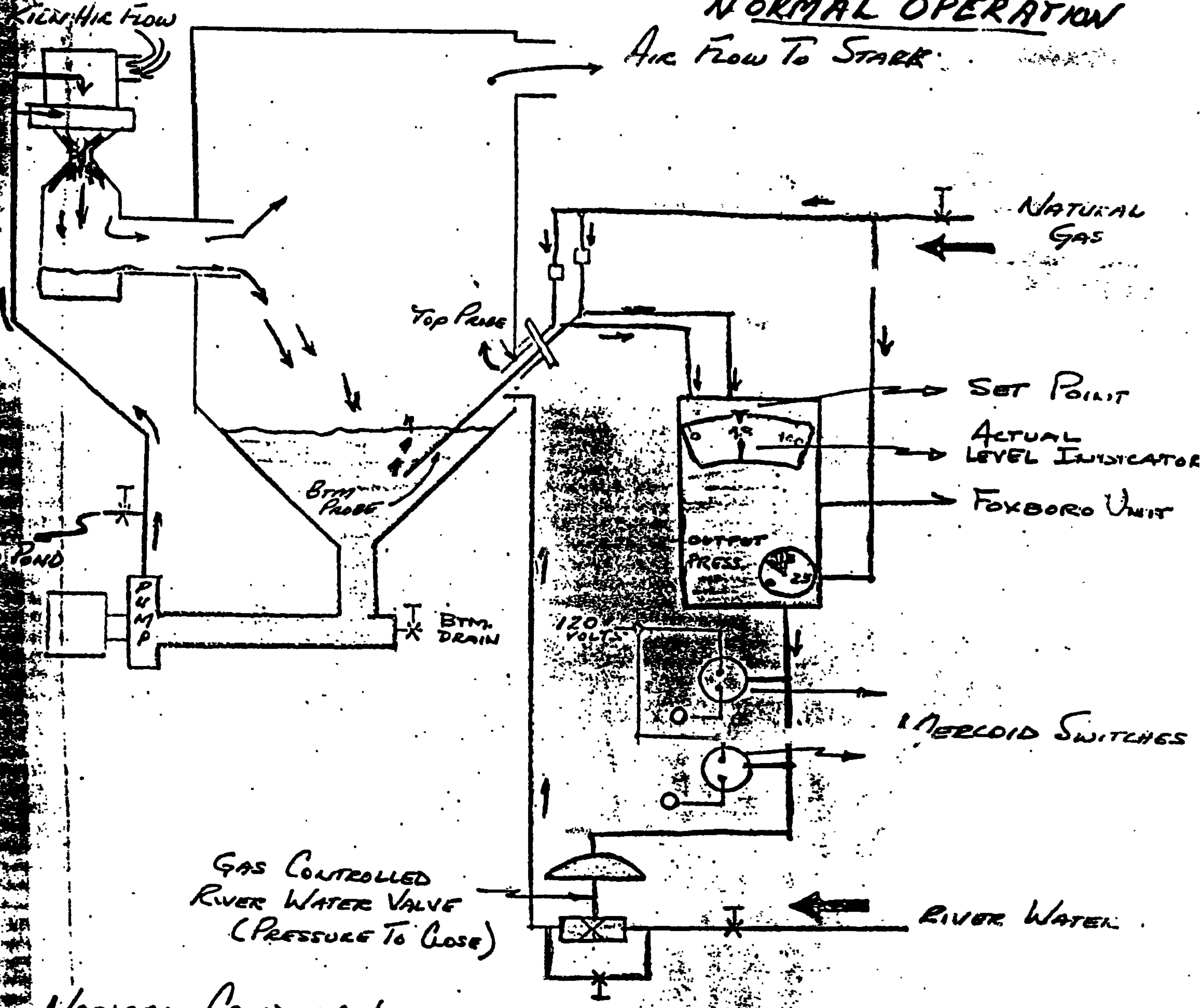
# HIGH LEVEL



## High Level Condition

Difference In Gas Pressure Between The Probes Is Large And Indicated As High % or High Level. The Foxboro Unit Signals A High Output Pressure, Which Is Sensed By The Mercoid Switch Which Turns On The High Level Light On The Alarm Panel In The Control Room. The High Output Pressure Keeps The River Water Control Valve Closed.

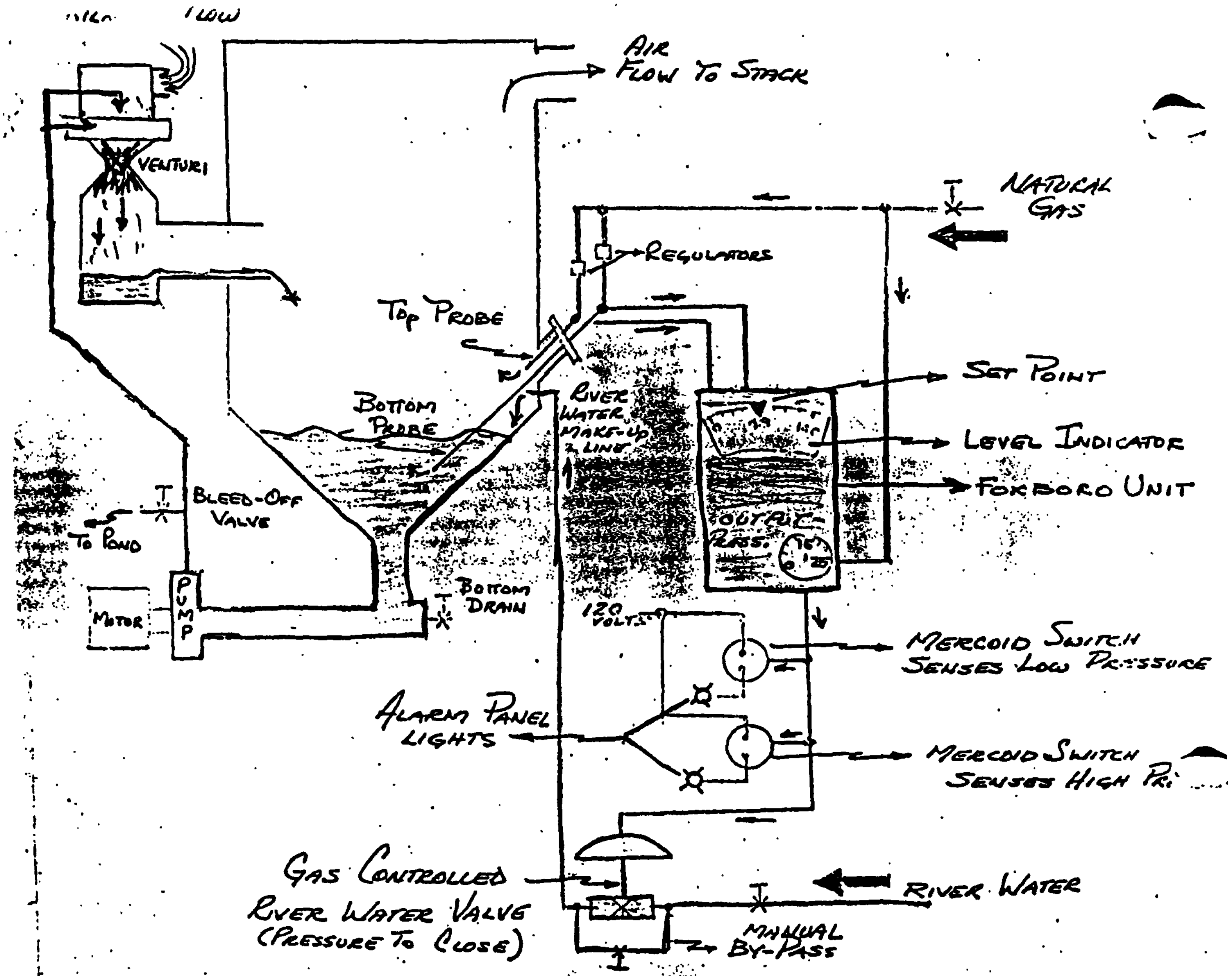
## Scrubber & Controls
## NORMAL OPERATION



### NORMAL CONDITION

For Normal Operation, The Difference In Gas Pressure Between The Probes Will Result In The % Water Level Indicator On The Foxboro Unit Will Be Near The Set Point Indication. The Foxboro Unit Will Regulate The Output Pressure To Control The River Valve To Maintain Proper Water Level In The Scrubber. There Will Be No Scrubber Alarms In The Control Room For Normal Operation.

# Attachment 1



NATURAL GAS IS SUPPLIED TO PROBES THROUGH REGULATORS. UNDER NORMAL OPERATION, GAS ESCAPES TO ATOMOSPHERE WITHOUT ANY RESISTANCE IN SHORT TOP PROBE FOR REFERENCE IN LEVEL INDICATOR. IN BOTTOM PROBE, GAS DEVELOPS PRESSURE IN LINE AS IT ATTEMPTS TO ESCAPE TO ATOMOSPHERE. THE DIFFERENCE IN GAS PRESSURE IN THE TWO PROBES IS MEASURED AND INDICATED AS % WATER LEVEL. FOXBORO UNIT THEN REGULATES OUTPUT GAS PRESSURE TO CONTROL RIVER VALVE.

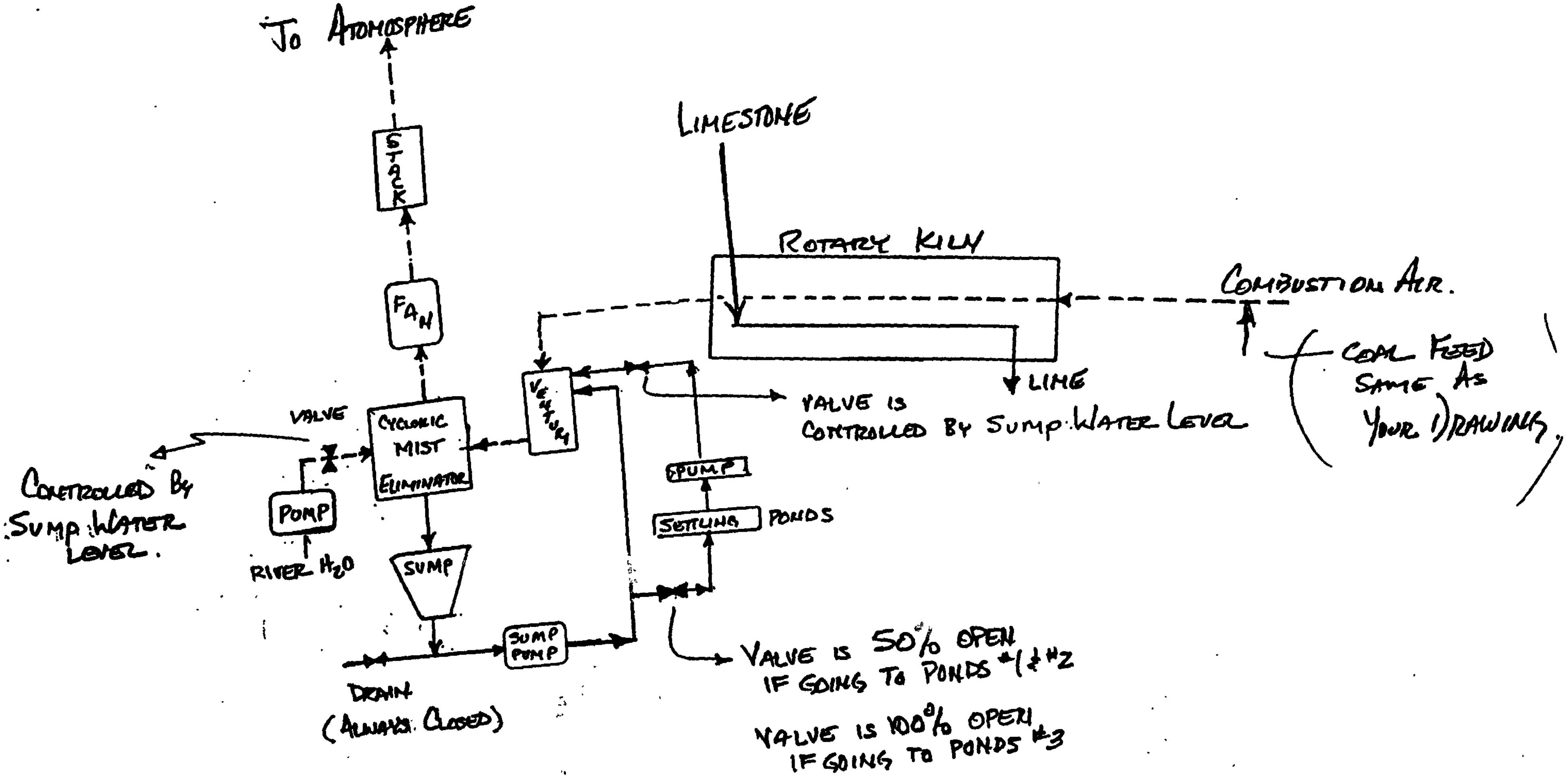

To Atmosphere
Stack
Fan
Limestone
Rotary Kiln
Combustion Air.
Coal Feed Same As Your Drawing
Lime
Valve
Controlled By Sump Water Level.
Cyclonic Mist Eliminator
Venturi
Valve is Controlled By Sump Water Level
Pump
Settling Ponds
Pump
River H2O
Sump
Sump Pump
Drain (Always Closed)
Valve is 50% open if going to ponds #1 & #2
Valve is 100% open if going to ponds #3

Figure 1

August 31

**Table 1. Process Parameters Recorded During Emissions Testing of Kiln 3 at Huron Lime ~~September 4~~, 1998**

| Time | Recorded limestone, tph | Fan, amperage | Scrubber pump, amperage | Feed end temp, °F | Kiln, rph | % $O_2$ | Coal feed indicator[1] |
|---|---|---|---|---|---|---|---|
| 3:00 PM | 28.0 | 82 | 34.5 | 1150 | 68.0 | 0.70 | 7.80 |
| 3:20 PM | 27.5 | 82 | 34.5 | 1150 | 69.0 | 0.70 | 7.80 |
| 3:35 PM | 28.5 | 83 | 34.5 | 1150 | 70.0 | 0.80 | 7.80 |
| 3:50 PM | 27.0 | 82 | 34.5 | 1150 | 70.0 | 0.60 | 7.80 |
| 4:05 PM | 29.0 | 84 | 34.5 | 1150 | 69.0 | 0.70 | 7.80 |
| 4:25 PM | 28.5 | 84 | 34.5 | 1150 | 69.5 | 0.70 | 7.80 |
| 4:45 PM | 27.5 | 82 | 34.5 | 1150 | 69.0 | 0.60 | 7.80 |
| 5:10 PM | 27.0 | 82 | 34.5 | 1150 | 62.5 | 0.75 | 7.80 |
| 5:50 PM | 26.5 | 82 | 34.5 | 1150 | 62.0 | 0.70 | 7.75 |
| 6:07 PM | 27.5 | 82 | 34.5 | 1150 | 62.0 | 0.70 | 7.80 |
| 6:29 PM | 27.5 | 82 | 34.5 | 1150 | 67.0 | 0.80 | 7.80 |
| 6:45 PM | 28.0 | 82 | 34.5 | 1150 | 66.5 | 0.70 | 7.80 |
| 7:00 PM | 28.0 | 82 | 34.5 | 1150 | 66.0 | 0.80 | 7.75 |
| 7:15 PM | 25.5 | 82 | 34.0 | 1150 | 66.5 | 0.90 | 7.80 |
| 7:30 PM | 26.5 | 82 | 34.5 | 1160 | 67.0 | 0.80 | 7.85 |
| 7:45 PM | 27.0 | 82 | 34.5 | 1160 | 66.5 | 0.70 | 7.80 |
| 8:00 PM | 26.0 | 82 | 34.5 | 1160 | 65.0 | 0.80 | 7.80 |
| 8:15 PM | 28.5 | 82 | 34.0 | 1165 | 60.5 | 0.70 | 7.85 |
| 8:30 PM | 27.0 | 82 | 34.0 | 1165 | 65.5 | 0.90 | 7.90 |
| 8:46 PM | 26.5 | 82 | 34.0 | 1165 | 65.5 | 1.00 | 7.85 |
| 9:01 PM | 27.5 | 82 | 34.0 | 1165 | 65.0 | 1.00 | 7.85 |
| 9:16 PM | 29.0 | 82 | 34.0 | 1165 | 65.5 | 0.90 | 7.95 |
| 9:30 PM | 28.5 | 82 | 34.0 | 1165 | 65.5 | 0.90 | 7.90 |
| 9:45 PM | 29.0 | 82 | 34.0 | 1165 | 65.5 | 0.90 | 7.90 |
| 10:00 PM | 27.5 | 82 | 34.0 | 1165 | 65.0 | 1.00 | 7.90 |
| 10:15 PM | 27.0 | 82 | 34.0 | 1165 | 65.0 | 0.90 | 7.90 |
| 10:30 PM | 25.5 | 82 | 34.5 | 1160 | 65.0 | 1.00 | 7.80 |

[1] The coal feed indicator parameter is a relative indicator of the feed rate of coal to the bowl mill, i.e., as the coal feed rate to the bowl mill increases, the value of the coal feed indicator increases and vice versa.

Acronyms and abbreviations: rph = rotations per hour; tph = tons per hour; ID = induced draft; °F = degrees Fahrenheit.

**Table 2. Statistical Analysis of Process Parameters Recorded During Emissions Testing of Kiln 3 at Huron Lime ~~September 4~~, 1998**

Aug 31

| | Recorded limestone, tph | Fan, amps | Scrubber pump, amps | Feed end temp, °F | Kiln, rph | % $O_2$ | Coal feed indicator |
|---|---|---|---|---|---|---|---|
| **# of recordings** | 27 | 27 | 27 | 27 | 27 | 27 | 27 |
| **Minimum rec'd value** | 25.5 | 82 | 34.0 | 1150 | 60.5 | 0.60 | 7.75 |
| **Maximum rec'd value** | 29.0 | 84 | 34.5 | 1165 | 70.0 | 1.00 | 7.95 |
| **Average of rec'd values** | 27.5 | 82 | 34.3 | 1156 | 66.1 | 0.80 | 7.83 |
| **% RSD of average of rec'd values** | 3.64 | 0.68 | 0.717 | 0.6092 | 3.74 | 15 | 0.647 |

Acronyms and abbreviations: rec'd = recorded; rph = rotations per hour; tph = tons per hour; ID = induced draft; °F = degrees Fahrenheit.; % RSD = percent relative standard deviation.

## References

1. Docket entry II-D-55 Letter and attachment, A. Paris, Huron Lime Company, to J. Wood, EPA:MICG, November 10, 1995, enclosing response to NLA/EPA voluntary questionnaire.

④

of the recorded values of fan amperage was 82, and the percent relative standard deviation of the recorded values was 0.68; the latter indicates little change in exhaust flow during testing.

- As shown in Figure 1, water from the settling ponds and sump is sprayed in the exhaust prior to the venturi throat; according to plant personnel, approximately 10 percent of the water is from the settling ponds and 90 percent is from the sump. The amperage of the sump pump was recorded during testing to monitor relative changes in the flow rate of sump water to the venturi. The average of the recorded values of pump amperage was 34.3 amps, and the percent relative standard deviation of the recorded values was 0.717; the latter indicates little change in the flow rate of sump water to the venturi.

- The morning of testing, plant personnel measured the pressure drop across the venturi throat; the measured pressure drop was 15 inches of water. During the pre-test site survey, the pressure drop across venturi throat was 11 inches of water.

- During testing, the averages of the recorded values of temperature and percent oxygen of the exhaust at the feed end of the kiln were 1156 °F and 0.80 percent, respectively; the percent relative standard deviations of the recorded values were 0.60 percent and 15 percent for kiln temperature and percent oxygen, respectively. During the pre-test site survey, the temperature and percent oxygen of the exhaust at the feed end of the kiln were 1050 °F during survey and 1.5 percent, respectively.

- The coal feed indicator parameter is a relative indicator of the feed rate of coal to the bowl mill, i.e., as the coal feed rate to the bowl mill increases, the value of the coal feed indicator increases and vice versa. As shown in Table 2, the percent relative standard deviation of the recorded values for this parameter were low, indicating little change in coal feed rate to the kiln.

- During testing, the average of the recorded values of kiln revolutions per hour (rph) was 66.1; during the pre-test site survey, kiln speed was noted as 72 rph.

## Process Information

Kiln 3 was built in 1971 (see Figure 1). The kiln is an inclined rotating kiln. High calcium limestone, which is quarried in Alpena, Michigan, enters through the back of the kiln (the highest point of incline), and tumbles toward the front end of the kiln via gravity and the rotating motion of the kiln. Combustion air and fuel enter at the front of the kiln. The primary fuel is coal; natural gas is used during start-up of the kiln. Lime exits the front of the kiln.

Exhaust from kiln 3 passes through a venturi scrubber, cyclonic mist eliminator, fan, and exhaust stack. The exhaust stack contains dampers, which are used to regulate air flow through the system. Water is sprayed in the exhaust as it enters the scrubber. Water from the mist eliminator drains to a sump; river water and clarified water from settling ponds are also added to the sump (see Attachment 1 for a description of how and when this occurs). Water is continuously pumped from the sump to the venturi. A portion of the water from the settling ponds is also pumped to the venturi.

## Process Monitoring

Table 1 lists the process parameters recorded during testing for the kiln and scrubber. Table 2 presents statistical analyses of the data in Table 1. The following points pertain to these tables.

- According to plant personnel, the instrument that reads tph of limestone is off by 5 units (on the plus side). The recorded values in Table 1 were not adjusted for this discrepancy.

- According to the plant's questionnaire, the design capacity of kiln 3 is 350 tons per day (tpd) of lime. During the pre-test site survey, plant personnel stated that kiln 3 was producing 300 tpd of lime. During testing, the average of the recorded values of tph of limestone was 27.5; subtracting five units from this average, multiplying it by 24 hours per day, and dividing it 2 tons of limestone per ton of lime gives 270 tpd of lime, which is approximately 77 percent of the design capacity.

- Fan amperage indicates a relative change in exhaust flow. During the pre-test site survey, fan amperage was recorded as 80 amps. During testing, the average

# APPENDIX F

## PROCESS DATA

Process data supplied by
Research Triangle Institute under a separate work assignment.

# Continuous Emissions Monitoring Data Sheet

## EPA Methods 3A, 25A, and 322

**Project Number** 98061

**Firm Name** PES **Testers** -

**Site Location** Huron Lime **Ambient Temp** 75

**Test Number** 1 **Time** 1749-2134

**Source** Outlet

**Date** 8/31/98

| Analyzer | Range | | Rack Cal. | Pre Test Sys. Cal. | Cal. Bias % of Span | Post Test Sys. Cal. | Cal. Bias % of Span | Drift % of Span |
|---|---|---|---|---|---|---|---|---|
| **Total Hydrocarbons** | 0-100ppm | zero | n/a | -0.2 | **n/a** | -0.3 | **n/a** | **0.1%** |
| | | upscale | n/a | 50.8 | **n/a** | 25.1 | **n/a** | **25.7%** |
| **Oxygen** | 0-25% | zero | 0.3 | 0.4 | **0.4%** | 0.3 | **0.0%** | **0.4%** |
| | | upscale | 18.9 | 19.1 | **0.8%** | 18.9 | **0.0%** | **0.8%** |
| **Carbon Dioxide** | 0-25% | zero | -0.3 | -0.2 | **0.5%** | 0.1 | **2.0%** | **-1.5%** |
| | | upscale | 18.9 | 19.3 | **2.0%** | 19.1 | **1.0%** | **1.0%** |
| | | | | | **±5%** | | **±5%** | **±3%** |

| Avg. Analyzer Response | Actual Gas Conc. |
|---|---|
| 0.8 | **n/a** |
| | |
| 6.7 | **6.5** |
| | |
| 21.1 | ~~**21.1**~~ |
| | |

20.9
ews

# TABLE C-3.1
## Huron Lime Calibration Table

## OUTLET

| Huron, Ohio | CALIBRATION ERROR TEST | | | |
|---|---|---|---|---|
| THC | Range 0 - 100ppm | | | |
| | ACTUAL CONC | RESPONSE | PREDICTED | % CAL ERR |
| ZERO GAS | 0.0 | 0.2 | - | - |
| LOW RANGE | 30.0 | 28.9 | 30.2 | -4.2% |
| MID RANGE | 50.1 | 50.8 | 50.2 | 1.1% |
| HIGH RANGE | 85.4 | 85.5 | 85.5 | 0.0% |
| O2 | Range 0 - 25% | | | |
| | ACTUAL CONC | RESPONSE | DIFFERENCE | % SPAN |
| ZERO GAS | 0.0 | 0.3 | 0.3 | 1.2% |
| MID RANGE | 11.1 | 11.2 | 0.1 | 0.4% |
| HIGH RANGE | 19.2 | 18.9 | -0.3 | -1.2% |
| CO2 | Range 0 - 25% | | | |
| | ACTUAL CONC | RESPONSE | DIFFERENCE | % SPAN |
| ZERO GAS | 0.0 | -0.3 | -0.3 | -1.2% |
| MID RANGE | 11.0 | 11.5 | 0.5 | 2.0% |
| HIGH RANGE | 19.0 | 18.9 | -0.1 | -0.4% |

Scott Specialty Gases

Shipped From: 1750 EAST CLUB BLVD
DURHAM NC 27704
Phone: 919-220-0803 Fax: 919-220-0808

# CERTIFICATE OF ANALYSIS

PACIFIC ENVIRONMENTAL SER

5001 SOUTH MIAMI
3RD FLOOR, SUITE #300
RESEARCH TRIANGLE PARK NC 27709-2077

PROJECT #: 12-30096-002
PO#: 104-99-0008
ITEM #: 1202RCOC AL
DATE 8/13/98

CYLINDER #: ALM044152
FILL PRESSURE: 1500PSIG

ANALYTICAL ACCURACY: +-1%
PRODUCT EXPIRATION: 8/13/2001

RECERTIFICATION

| COMPONENT | ANALYSIS |
|---|---|
| PROPANE | 85.37 PPM |
| AIR | BAL. |

ANALYTICAL METHOD: VARIAN 3400

ANALYST: B BECTON

APPROVED BY: G BARTNETT

**Scott Specialty Gases**

Shipped From:
1750 EAST CLUB BLVD
DURHAM NC 27704
Phone: 919-220-0803 Fax: 919-220-0808

## CERTIFICATE OF ANALYSIS

---

PACIFIC ENVIRONMENTAL SER

5001 SOUTH MIAMI
3RD FLOOR, SUITE #300
RESEARCH TRIANGLE PARK NC 27709-2077

PROJECT #: 12-30096-001
PO#: 104-99-0008
ITEM #: 1202RCOC AL
DATE: 8/13/98

---

CYLINDER #: ALM029561
FILL PRESSURE: 900PSIG

ANALYTICAL ACCURACY: +-1%
PRODUCT EXPIRATION: 8/13/2001

RECERTIFICATION

| COMPONENT | ANALYSIS |
|---|---|
| PROPANE | 50.14 PPM |
| AIR | BAL. |

ANALYTICAL METHOD: VARIAN 3400

ANALYST: B BECTON

APPROVED BY: G BARNETT

**Scott Specialty Gases**

1750 EAST CLUB BOULEVARD, DURHAM, NC 27704 (919) 220-0803 FAX: (919) 220-0808

# CERTIFICATE OF ANALYSIS: EPA PROTOCOL GAS

**Customer**
Pacific Environmental Services
Attn: Mr. Frank Meadows
P.O. Box 12077
Research Triangle Park, NC 27709

**Assay Laboratory**
Scott Specialty Gases, Inc.
1750 East Club Boulevard
Durham, NC 27704

**Purchase Order** 104-95-0121
**Scott Project #** 12-11271

## ANALYTICAL INFORMATION

Certified to exceed the minimum specifications of EPA Protocol Procedure #G1, issued September, 1993.

**Cylinder Number** AAL-13302 **Certification Date** 04-18-95 **Expiration Date** 04-18-98
**Cylinder Pressure** 2000 PSIG **Previous Certification** None

### ANALYZED CYLINDER

| Components | Certified Concentration | Analytical Uncertainty* |
|---|---|---|
| Propane | 29.93 PPM | +/- 1% NIST Directly Traceable |
| Air | | Balance |

Do not use when cylinder pressure is less than 150 PSIG.

*Analytical uncertainty is inclusive of usual known error sources which at least includes reference standard error & precision of the measurement processes.

### REFERENCE STANDARD

| Type | Expiration Date | Cylinder Number | Concentration |
|---|---|---|---|
| NTRM # 1668 | 06-96 | ALM-032005 | 95.5 PPM Balance in Air |

### INSTRUMENTATION

| Instrument/Model/Serial # | Last Date Calibrated | Analytical Principle |
|---|---|---|
| Varian /3400/16804 | 03-23-95 | Gas Chromatography |

## ANALYZER READINGS (Z=Zero Gas R=Reference Gas T=Test Gas r=Correlation Coefficient)

| Components | First Triad Analysis | Second Triad Analysis | Calibration Curve |
|---|---|---|---|
| Propane | Date: 04-18-95 Response Units: Area<br>STD=1397517 SPL=436968<br>SPL=437952 SPL=438070<br>STD=1396975 STD=1395705 | Date: Response Units:<br>STD= SPL=<br>SPL= SPL=<br>STD= STD= | Date: 03-23-95 |
| | Date: Response Units:<br>STD= SPL=<br>SPL= SPL=<br>STD= STD= | Date: Response Units:<br>STD= SPL=<br>SPL= SPL=<br>STD= STD= | Date: |
| | Date: Response Units:<br>STD= SPL=<br>SPL= SPL=<br>STD= STD= | Date: Response Units:<br>STD= SPL=<br>SPL= SPL=<br>STD= STD= | |

Analyst S. Vaughan

Scott Specialty Gases

Shipped From: 1750 EAST CLUB BLVD
DURHAM NC 27704
Phone: 919-220-0803 Fax: 919-220-0808

# CERTIFICATE OF ANALYSIS

PACIFIC ENVIRONMENTAL SER

5001 SOUTH MIAMI
3RD FLOOR, SUITE #300
RESEARCH TRIANGLE PA NC 27709-2077

PROJECT #: 12-28662-001
PO#: 104-98-0178
ITEM #: 12023411 CAL
DATE: 5/01/98

CYLINDER #: AAL13302
FILL PRESSURE: 1400 PSIG

ANALYTICAL ACCURACY: +-1%
PRODUCT EXPIRATION: 5/01/2001

BLEND TYPE : RECERTIFICATION OF CYLINDER

| **COMPONENT** | **REQUESTED GAS CONC MOLES** | | **ANALYSIS (MOLES)** | |
|---|---|---|---|---|
| PROPANE | 30. | PPM | 30.0 | PPM |
| AIR | | BALANCE | | BALANCE |

ANALYST: B. M. Becton
B.M. BECTON

Airgas

**Airgas Specialty G**

325 McCausland Court
Cheshire, CT 06410
Phone: (203) 250-6827
FAX. (203) 250-6842

# Certificate of Analysis: E.P.A. Protocol Gas Mixture

| | | | |
|---|---|---|---|
| Rec# | 4150 | | |
| Cylinder No : | CC86922 | Purchase Order # | 13980 |
| Cylinder Pressure: | 2000 | Expiration Date | 3/2/01 |
| Certification Date | 3/2/98 | Laboratory: | Cheshire, CT |

## Reference Standard Information:

| Type | Component | Cyl. Number | Concentration |
|---|---|---|---|
| GMIS | Carbon Dioxide | CC34977 | 14.08 % |
| GMIS | Oxygen | CC19914 | 20.98 % |

## Instrumentation:

| Instrument/Model/Serial No. | Analytical Principle |
|---|---|
| Rosemount/NGA2000/Rack#1 | NDIR |
| Servomex/244/701/488 | Parmagnetic |

Analytical Methodology does not require correction for analytical interferences.

## Certified Concentrations:

| Component | Concentration | Accuracy | Procedure |
|---|---|---|---|
| Carbon Dioxide | 19.01 % | +/- 2% | G1 |
| Oxygen | 19.17 % | +/- 2% | G1 |
| Nitrogen | Balance | | |

## Analytical Results:

**1st Component:** Carbon Dioxide

1st Analysis Date: 2/16/98

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| R | 3.259 | S | 3.726 | Z | 0.305 | Conc | 19.065 % |
| S | 3.782 | Z | 0.256 | R | 3.305 | Conc | 19.006 % |
| Z | 0.265 | R | 3.298 | S | 3.769 | Conc | 18.964 % |
| | | | | | | AVG: | 19.012 % |

**2nd Component:** Oxygen

1st Analysis Date: 3/2/98

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| R | 173.630 | S | 156.890 | Z | 1.480 | Conc | 19.175 % |
| S | 156.970 | Z | 1.420 | R | 173.810 | Conc | 19.165 % |
| Z | 1.890 | R | 173.630 | S | 157.030 | Conc | 19.158 % |
| | | | | | | AVG: | 19.166 % |

*Certification performed in accordance with "EPA Traceability Protocol (Jan. 1998)" using the assay procedures listed.*

Do not use cylinder below 150 psig.

Pete Holmes
Approved for Release

Airgas

**Airgas Specialty Gases**

325 McCausland Court
Cheshire, CT 06410
Phone: (203) 250-6827
FAX: (203) 250-6842

# Certificate of Analysis: E.P.A. Protocol Gas Mixture

|  |  |  |  |
|---|---|---|---|
| Rec# | 4149 |  |  |
| Cylinder No : | CC86779 | Purchase Order # | 139680 |
| Cylinder Pressure: | 2000 | Expiration Date: | 3/2/01 |
| Certification Date | 3/2/98 | Laboratory: | Cheshire, CT |

## Reference Standard Information:

| Type | Component | Cyl. Number | Concentration |
|---|---|---|---|
| GMIS | Carbon Dioxide | CC34977 | 14.08 % |
| GMIS | Oxygen | CC19914 | 20.98 % |

## Instrumentation:

| Instrument/Model/Serial No. | Analytical Principle |
|---|---|
| Rosemount/NGA2000/Rack#1 | NDIR |
| Servomex/244/701/488 | Parmagnetic |

Analytical Methodology does not require correction for analytical interferences.

## Certified Concentrations:

| Component | Concentration | Accuracy | Procedure |
|---|---|---|---|
| Carbon Dioxide | 10.97 % | +/- 2% | G1 |
| Oxygen | 11.10 % | +/- 2% | G1 |
| Nitrogen | Balance |  |  |

## Analytical Results:

**1st Component:** Carbon Dioxide

1st Analysis Date: 2/16/98

|  |  |  |  |  |  |  |  |
|---|---|---|---|---|---|---|---|
| R | 3.259 | S | 2.899 | Z | 0.305 | Conc | 10.931 % |
| S | 2.940 | Z | 0.256 | R | 3.305 | Conc | 10.981 % |
| Z | 0.265 | R | 3.298 | S | 2.939 | Conc | 11.012 % |
|  |  |  |  |  |  | AVG: | 10.975 % |

**2nd Component:** Oxygen

1st Analysis Date: 3/2/98

|  |  |  |  |  |  |  |  |
|---|---|---|---|---|---|---|---|
| R | 173.630 | S | 91.620 | Z | 1.460 | Conc | 11.118 % |
| S | 91.580 | Z | 1.420 | R | 173.810 | Conc | 11.103 % |
| Z | 1.890 | R | 173.630 | S | 91.690 | Conc | 11.084 % |
|  |  |  |  |  |  | AVG: | 11.102 % |

*Certification performed in accordance with "EPA Traceability Protocol (Jan. 1998)" using the assay procedures listed.*

Do not use cylinder below 150 psig.

Approved for Release

# NOZZLE CALIBRATION SHEET

DATE: 6-16-98 CALIBRATION BY: JWB

| Nozzle Identification Number | $D_1$, in. | $D_2$, in. | $D_3$, in. | $\Delta D$, in. | $D_{avg}$ |
|---|---|---|---|---|---|
| GLASS XXIV | 0.310 | 0 | 0.310 | 0 | 0.310 |

Where:

$D_{1,2,3}$ = nozzle diameter measured on a different diameter, in.
Tolerance = measure within 0.001 in.

$\Delta D$ = maximum difference in any two measurements, in.
Tolerance = 0.004 in.

$D_{avg}$ = average of $D_1$, $D_2$, $D_3$.

7C

# PACIFIC ENVIRONMENTAL SERVICES, INC.

4700 Duke Drive,
Suite 150
Mason, Ohio 45040
Phone: (513) 398-2556
Fax (513) 398-3342
www.pes.com

Pitot Tube Number: 7C

Date: 12/15/97

Effective Length: 85"

Calibrated By: S. Simon

Pitot Tube Openings Damaged? YES [✓ NO]

Pitot Tube Assembly Level? [✓ YES] NO

$\alpha_1$ = 1 °(< 10°)

$\alpha_2$ = 3 °(< 10°)

$\beta_1$ = 1 °(< 5°)

$\beta_2$ = 1 °(< 5°)

Y= 1

$\theta$ = 1.3

A = 0.966

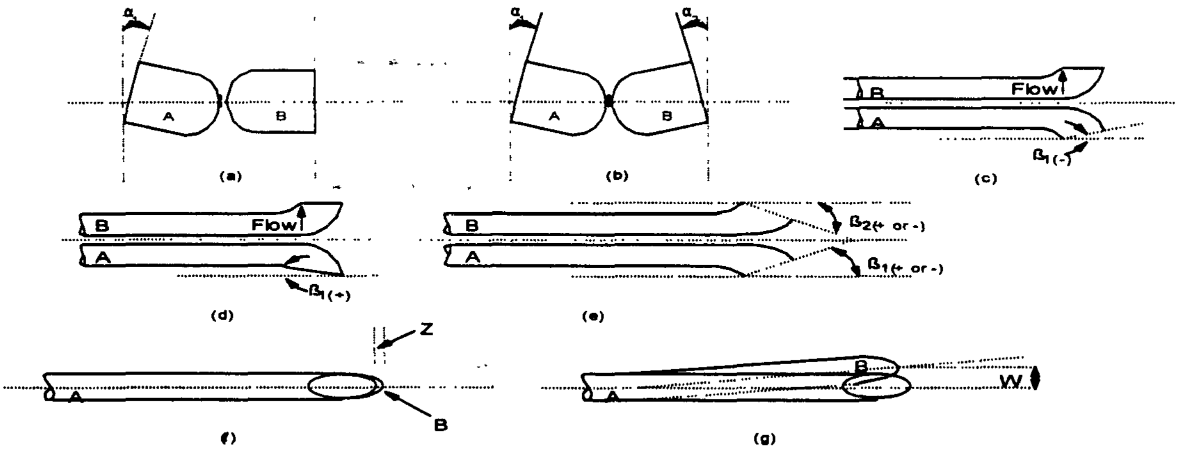
$z = A \sin \gamma$ = 0.01686 cm (in.) 0.32 cm ( < 1/8 in.)

$w = A \sin \theta$ = 0.02192 cm (in.) 0.08 cm ( < 1/32 in.)

$P_A$ = 0.483 cm (in.)

$P_\beta$ = 0.483 cm (in.)

$D_t$ = 0.375 cm (in.)



The types of face-opening misalignment shown above will not affect the baseline value of Cp(s) so long as $\alpha_1$ and $\alpha_2$ is less than or equal to 10°, $\beta_1$ and $\beta_2$ is less than or equal to 5°, z is less than or equal to 0.32 cm (1/8 in.), and w is less than or equal to 0.08 cm (1/32 in.) (reference 11.0 in Section 16.0).

**PACIFIC ENVIRONMENTAL SERVICES,INC.**

**4700 Duke Drive,**
**Suite 150**
**Mason, Ohio**
**Phone: (513) 398-2556**
**Fax: (513) 3983342**
**www.pes.com**

# TEMPERATURE SENSOR CALIBRATION DATA FOR STACK THERMOCOUPLES

**THERMOCOUPLE NUMBER:** 7C

**DATE:** 12/15/97

**BAROMETRIC PRES.(In.Hg):** 29.52

**REFERENCE:**
**Mercury-in-glass:** ASTM-3F

**Other:**

**AMBIENT TEMP. °F:** 74

**CALIBRATOR:** G. Gay

| Reference point number | Source[a] (Specify) | Reference Thermometer Temperature,°F | Thermocouple Potentiometer Temperature,°F | Temperature Difference,[b] % |
|---|---|---|---|---|
| 1 | Ambient Air | 74 | 74 | 0.00 |
| 2 | Cold Bath | 40 | 41 | 0.20 |
| 3 | Hot Bath | 206 | 205 | 0.15 |
| 4 | Hot Oil | 340 | 341 | 0.13 |

[a] Type of calibration used.

[b] $\frac{\text{(ref. temp.°F+460)-(test thermometer temp.°F+460)}}{\text{ref temp,°F+460}}$ X100 100≤1.5%

Comments:

# TEMPERATURE SENSOR CALIBRATION FORM

Temperature Sensor No. DGM-OUT MB-10 Sensor Type K-TC Length 12"
Ambient Temp. °F 76 Barometric Pressure, "Hg 29.61"
Reference Temp. Sensor:

| Date | Ref. Point No. | Temp. Source | Temp. °F | | Temp. Diff. % | Within Limits Y/N | Calibrated By |
|---|---|---|---|---|---|---|---|
| | | | Ref. Sensor | Test Sensor | | | |
| 3-20-98 | 1 | ICE H2O | 32 | 34 | .406 | Y | JWB |
| " | 2 | AMB. AIR | 76 | 77 | .186 | Y | JWB |
| " | 3 | BOIL H2O | 206 | 205 | .150 | Y | JWB |
| | 1 | | | | | | |
| | 2 | | | | | | |
| | 3 | | | | | | |
| | 1 | | | | | | |
| | 2 | | | | | | |
| | 3 | | | | | | |
| | 1 | | | | | | |
| | 2 | | | | | | |
| | 3 | | | | | | |
| | 1 | | | | | | |
| | 2 | | | | | | |
| | 3 | | | | | | |
| | 1 | | | | | | |
| | 2 | | | | | | |
| | 3 | | | | | | |

$$\% \text{ Temp. Diff} = \frac{(\text{Ref. Temp} + 460) - (\text{ Test Temp.} + 460)}{(\text{Ref. Temp.} + 460)} \times 100 \leq 1.5 \%$$

# TEMPERATURE SENSOR CALIBRATION FORM

DGM-IN

**Temperature Sensor No.** MB-10 **Sensor Type** K-TC **Length** 8'
**Ambient Temp. °F** 76 **Barometric Pressure, "Hg** 29.61
**Reference Temp. Sensor:** ____________

| Date | Ref. Point No. | Temp. Source | Temp. °F | | Temp. Diff. % | Within Limits Y/N | Calibrated By |
|---|---|---|---|---|---|---|---|
| | | | Ref. Sensor | Test Sensor | | | |
| 3-20-98 | 1 | ICE H2O | 33 | 34 | | | |
| " | 2 | AMB. AIR | 76 | 76 | 0 | | |
| " | 3 | BOIL H2O | 206 | 205 | | | |
| | 1 | | | | | | |
| | 2 | | | | | | |
| | 3 | | | | | | |
| | 1 | | | | | | |
| | 2 | | | | | | |
| | 3 | | | | | | |
| | 1 | | | | | | |
| | 2 | | | | | | |
| | 3 | | | | | | |
| | 1 | | | | | | |
| | 2 | | | | | | |
| | 3 | | | | | | |
| | 1 | | | | | | |
| | 2 | | | | | | |
| | 3 | | | | | | |

$$\%\ Temp.\ Diff = \frac{(Ref.\ Temp + 460) - (\ Test\ Temp. + 460)}{(Ref.\ Temp. + 460)} \times 100 \leq 1.5\ \%$$

# REFERENCE METER CALIBRATION
## ENGLISH REFERENCE METER UNITS

DGM Serial # 6841495
Date 8/28/96

Barometric Pressure 29.73
Meter Yw 1.00000
K ( deg R/inches Hg) 17.64

Filename: F:\DATAFILE\CALIBRAT\CAL_MENU.DSK\DGM_REF.
Revised: 06/08/95

<table>
<tr><th></th><th></th><th colspan="3">Dry Gas Meter (DGM)</th><th colspan="2">Temperature</th><th colspan="4">Wet Test Meter (WTM)</th><th></th><th></th><th></th></tr>
<tr><th>Time (min)</th><th>Pressure (in. H20)</th><th>Meter Readings Initial</th><th>Meter Readings Final</th><th>Volume (cubic feet)</th><th>Initial (deg F)</th><th>Final (deg F)</th><th>Meter Readings Initial</th><th>Meter Readings Final</th><th>Volume (cubic feet)</th><th>Temp (deg F)</th><th>DGM Coefficient Yds</th><th>Coefficient Variation Yds-(Avg.Yds)</th><th>Flow Rate (CFM)</th></tr>
<tr><td>6.00</td><td>-6.60</td><td>374.451</td><td>381.901</td><td>7.450</td><td>73.0</td><td>76.0</td><td>496.572</td><td>503.987</td><td>7.415</td><td>77.0</td><td>1.007</td><td>-0.004</td><td>1.207</td></tr>
<tr><td>24.00</td><td>-6.60</td><td>381.901</td><td>411.424</td><td>29.523</td><td>74.0</td><td>76.0</td><td>503.987</td><td>533.471</td><td>29.484</td><td>77.0</td><td>1.011</td><td>0.000</td><td>1.200</td></tr>
<tr><td>8.00</td><td>-6.60</td><td>411.424</td><td>421.233</td><td>9.809</td><td>76.0</td><td>76.0</td><td>533.471</td><td>543.279</td><td>9.808</td><td>77.0</td><td>1.015</td><td>0.004</td><td>1.197</td></tr>
<tr><td colspan="14">Max Yds - Min Yds =0.007489914 Must be no greater than 0.030</td></tr>
<tr><td colspan="14">Average Yds =1.011058546 Must be between 0.95 to 1.05</td></tr>
<tr><td>10.00</td><td>-4.00</td><td>421.233</td><td>430.675</td><td>9.442</td><td>76.0</td><td>77.0</td><td>543.279</td><td>552.761</td><td>9.482</td><td>77.0</td><td>1.013</td><td>0.009</td><td>0.926</td></tr>
<tr><td>35.00</td><td>-4.00</td><td>430.675</td><td>464.147</td><td>33.472</td><td>77.0</td><td>77.0</td><td>552.761</td><td>585.965</td><td>33.204</td><td>77.0</td><td>1.002</td><td>-0.003</td><td>0.926</td></tr>
<tr><td>16.50</td><td>-4.00</td><td>464.147</td><td>479.992</td><td>15.845</td><td>77.0</td><td>78.0</td><td>585.965</td><td>601.625</td><td>15.660</td><td>77.0</td><td>0.999</td><td>-0.006</td><td>0.927</td></tr>
<tr><td colspan="14">Max Yds - Min Yds =0.014197179 Must be no greater than 0.030</td></tr>
<tr><td colspan="14">Average Yds =1.004786738 Must be between 0.95 to 1.05</td></tr>
<tr><td>12.50</td><td>-2.80</td><td>479.992</td><td>489.698</td><td>9.706</td><td>78.0</td><td>78.0</td><td>601.625</td><td>611.270</td><td>9.645</td><td>77.0</td><td>1.003</td><td>0.002</td><td>0.754</td></tr>
<tr><td>14.00</td><td>-2.80</td><td>489.698</td><td>500.594</td><td>10.896</td><td>78.0</td><td>78.0</td><td>611.270</td><td>622.061</td><td>10.791</td><td>77.0</td><td>0.999</td><td>-0.002</td><td>0.753</td></tr>
<tr><td>58.50</td><td>-2.80</td><td>500.594</td><td>546.063</td><td>45.469</td><td>78.0</td><td>79.0</td><td>622.061</td><td>667.125</td><td>45.064</td><td>77.0</td><td>1.001</td><td>0.000</td><td>0.752</td></tr>
<tr><td colspan="14">Max Yds - Min Yds = 0.00338145 Must be no greater than 0.030</td></tr>
<tr><td colspan="14">Average Yds =1.000808891 Must be between 0.95 to 1.05</td></tr>
<tr><td>16.50</td><td>-1.60</td><td>574.496</td><td>583.672</td><td>9.176</td><td>79.0</td><td>79.0</td><td>695.390</td><td>704.530</td><td>9.140</td><td>77.0</td><td>1.004</td><td>0.000</td><td>0.541</td></tr>
<tr><td>42.00</td><td>-1.60</td><td>590.619</td><td>614.123</td><td>23.504</td><td>80.0</td><td>80.0</td><td>711.429</td><td>734.785</td><td>23.356</td><td>77.0</td><td>1.003</td><td>0.000</td><td>0.543</td></tr>
<tr><td>66.50</td><td>-1.60</td><td>614.123</td><td>651.520</td><td>37.397</td><td>80.0</td><td>81.0</td><td>734.785</td><td>771.901</td><td>37.116</td><td>77.0</td><td>1.003</td><td>0.000</td><td>0.545</td></tr>
<tr><td colspan="14">Max Yds - Min Yds =0.000835063 Must be no greater than 0.030</td></tr>
<tr><td colspan="14">Average Yds =1.003302205 Must be between 0.95 to 1.05</td></tr>
<tr><td>15.00</td><td>-1.00</td><td>651.520</td><td>657.572</td><td>6.052</td><td>81.0</td><td>82.0</td><td>771.901</td><td>777.994</td><td>6.093</td><td>78.0</td><td>1.016</td><td>0.011</td><td>0.396</td></tr>
<tr><td>13.50</td><td>-1.00</td><td>657.572</td><td>663.065</td><td>5.493</td><td>82.0</td><td>82.0</td><td>777.994</td><td>783.400</td><td>5.406</td><td>78.0</td><td>0.994</td><td>-0.010</td><td>0.390</td></tr>
<tr><td>35.00</td><td>-1.00</td><td>663.065</td><td>677.274</td><td>14.209</td><td>82.0</td><td>82.0</td><td>783.400</td><td>797.515</td><td>14.115</td><td>78.0</td><td>1.003</td><td>-0.001</td><td>0.393</td></tr>
<tr><td colspan="14">Max Yds - Min Yds =0.021724294 Must be no greater than 0.030</td></tr>
<tr><td colspan="14">Average Yds =1.004344616 Must be between 0.95 to 1.05</td></tr>
</table>

Overall Average Yds =1.004860199

I certify that the above Dry Gas Meter was calibrated in accordance with E.P.A. Method 5 , paragraph 7.1 ;CFR 40 Part 60, using the Precision Wet Test Meter # 11AE6, which in turn was calibrated using the American Bell Prover # 3785, certificate # F107, which is traceable to the National Bureau of Standards (N.I.S.T.).

Signature ______________ Date 9-24-96

**PACIFIC ENVIRONMENTAL SERVICES, INC.**

Central Park West
5001 South Miami Boulevard, P.O. Box 12077
Research Triangle Park, North Carolina 27709-2077
(919) 941-0333 FAX: (919) 941-0234

# Posttest Dry Gas Meter Calibration Form (English Units)

| | |
|---|---|
| Pretest Calibration Factor | 1.021 |
| System Vacuum Setting, (in Hg) | 11 |
| Reference Meter Correction Factor | 1.008 |

Date: 9/8/98 $P_{bar}$, in Hg 29.75 Calibrator: jwb Meter Box No. MB-10

ΔH = 1.41

| | | Dry Gas Meter | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Trial | Duration (min) | Initial ($ft^3$) | Final ($ft^3$) | Net ($ft^3$) | Initial, Inlet (°F) | Final, Inle (°F) | Avg. Inlet (°F) | Initial, Outlet (°F) | Final, Outlet (°F) | Avg. Outlet (°F) |
| 1 | 10 | 175.033 | 182.466 | 7.433 | 73 | 75 | 74 | 73 | 74 | 73.5 |
| 2 | 10 | 182.466 | 189.866 | 7.400 | 75 | 77 | 76 | 74 | 74 | 74 |
| 3 | 10 | 189.866 | 197.28 | 7.414 | 77 | 78 | 77.5 | 74 | 75 | 74.5 |

| | Reference Meter | | | | | | Meter Box Correction Factor | Reference Orifice Press |
|---|---|---|---|---|---|---|---|---|
| | Gas Volume | | | Meter Temperature | | | | |
| Trial | Initial ($ft^3$) | Final ($ft^3$) | Net ($ft^3$) | Initial (°F) | Final (°F) | Avg. (°F) | γ | $\Delta H_@$ (in. H2O) |
| 1 | 655.378 | 662.858 | 7.480 | 73 | 73 | 73 | 1.012 | 1.42 |
| 2 | 662.858 | 670.293 | 7.435 | 73 | 74 | 3.5 | 1.012 | 1.44 |
| 3 | 670.293 | 677.758 | 7.465 | 74 | 74 | 74 | 1.015 | 1.43 |

[redacted]

| ΔH = | 2.0 | Dry Gas Meter MB-10 | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Trial | Gas Volume | | | Meter Temperatures | | | | | |
| Trial | Duration (min) | Initial (ft$^3$) | Final (ft$^3$) | Net (ft$^3$) | Initial, Inlet (°F) | Final, Inlet (°F) | Avg. Inlet (°F) | Initial, Outlet (°F) | inal, Outle (°F) | Avg. Outlet (°F) |
| 1 | 10 | 55.868 | 63.519 | 7.651 | 84 | 86 | 85 | 81 | 81 | 81 |
| 2 | 10 | 63.519 | 71.182 | 7.663 | 86 | 86 | 86 | 81 | 81 | 81 |
| 3 | 10 | 71.182 | 78.845 | 7.663 | 86 | 87 | 86.5 | 81 | 81 | 81 |

| | Reference Meter | | | | | | Meter Box | Reference |
|---|---|---|---|---|---|---|---|---|
| | Gas Volume | | | Meter Temperature | | | Correction | Orifice Press |
| Trial | Initial (ft$^3$) | Final (ft$^3$) | Net (ft$^3$) | Initial (°F) | Final (°F) | Avg. (°F) | Factor $\gamma$ | $\Delta H_@$ (in. $H_2O$) |
| 1 | 662.729 | 670.472 | 7.743 | 78 | 78 | 78 | 1.021 | 1.87 |
| 2 | 670.472 | 678.244 | 7.772 | 78 | 78 | 78 | 1.025 | 1.86 |
| 3 | 678.244 | 686.010 | 7.766 | 78 | 78 | 78 | 1.024 | 1.86 |

| ΔH = | 4.0 | Dry Gas Meter MB-10 | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Trial | Gas Volume | | | Meter Temperatures | | | | | |
| Trial | Duration (min) | Initial (ft$^3$) | Final (ft$^3$) | Net (ft$^3$) | Initial, Inlet (°F) | Final, Inlet (°F) | Avg. Inlet (°F) | Initial, Outlet (°F) | inal, Outle (°F) | Avg. Outlet (°F) |
| 1 | 8 | 79.058 | 86.620 | 7.562 | 85 | 88 | 86.5 | 81 | 82 | 81.5 |
| 2 | 8 | 86.620 | 94.185 | 7.565 | 87 | 89 | 88 | 82 | 82 | 82 |
| 3 | 8 | 94.185 | 101.754 | 7.569 | 89 | 89 | 89 | 82 | 82 | 82 |

| | Reference Meter | | | | | | Meter Box | Reference |
|---|---|---|---|---|---|---|---|---|
| | Gas Volume | | | Meter Temperature | | | Correction | Orifice Press |
| Trial | Initial (ft$^3$) | Final (ft$^3$) | Net (ft$^3$) | Initial (°F) | Final (°F) | Avg. (°F) | Factor $\gamma$ | $\Delta H_@$ (in. $H_2O$) |
| 1 | 686.208 | 693.895 | 7.687 | 78 | 78 | 78 | 1.023 | 2.44 |
| 2 | 693.895 | 701.558 | 7.663 | 78 | 78 | 78 | 1.021 | 2.45 |
| 3 | 701.558 | 709.244 | 7.686 | 78 | 78 | 78 | 1.025 | 2.43 |

Calibration Results

| ΔH | $\gamma$ | $\Delta H_@$ |
|---|---|---|
| 0.50 | 1.020 | 1.73 |
| 0.75 | 1.020 | 1.79 |
| 1.0 | 1.020 | 1.78 |
| 2.0 | 1.023 | 1.86 |
| 4.0 | 1.023 | 2.44 |

| **Dry Gas Meter MB-10 on 09/01/97** | |
|---|---|
| Meter Box Calibration Factor | **1.021** |
| Meter Box Reference Orifice Pressure | **1.92** |

**PACIFIC ENVIRONMENTAL SERVICES, INC.**

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# Initial Dry Gas Meter Calibration Form (English Units)

Date: 9/1/97
Calibrator: Tom McDonald
Meter Box No.: MB-10
$P_{bar}$, in Hg 30.16
Reference Meter Correction Factor: 1.0049 (8/28/96)

ΔH = 0.5

| | Dry Gas Meter MB-10 | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Trial | Gas Volume | | | Meter Temperatures | | | | | |
| | Duration | Initial | Final | Net | Initial, Inlet | Final, Inlet | Avg. Inlet | Initial, Outlet | inal, Outle | Avg. Outlet |
| Trial | (min) | ($ft^3$) | ($ft^3$) | ($ft^3$) | (°F) | (°F) | (°F) | (°F) | (°F) | (°F) |
| 1 | 19 | 994.409 | 1001.982 | 7.573 | 74 | 78 | 76 | 73 | 75 | 74 |
| 2 | 19 | 1001.982 | 1009.513 | 7.531 | 77 | 80 | 78.5 | 75 | 77 | 76 |
| 3 | 19 | 1009.513 | 1017.050 | 7.537 | 80 | 81 | 80.5 | 77 | 78 | 77.5 |

| | Reference Meter | | | | | | Meter Box | Reference |
|---|---|---|---|---|---|---|---|---|
| | Gas Volume | | | Meter Temperature | | | Correction | Orifice Press |
| | Initial | Final | Net | Initial | Final | Avg. | Factor | $\Delta H_@$ |
| Trial | ($ft^3$) | ($ft^3$) | ($ft^3$) | (°F) | (°F) | (°F) | γ | (in. $H_2O$) |
| 1 | 600.523 | 608.185 | 7.662 | 72 | 74 | 73 | 1.019 | 1.71 |
| 2 | 608.185 | 615.801 | 7.616 | 74 | 76 | 75 | 1.019 | 1.74 |
| 3 | 615.801 | 623.430 | 7.629 | 76 | 77 | 76.5 | 1.021 | 1.74 |

ΔH = 0.75

| | Dry Gas Meter MB-10 | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Trial | Gas Volume | | | Meter Temperatures | | | | | |
| | Duration | Initial | Final | Net | Initial, Inlet | Final, Inlet | Avg. Inlet | Initial, Outlet | inal, Outle | Avg. Outlet |
| Trial | (min) | ($ft^3$) | ($ft^3$) | ($ft^3$) | (°F) | (°F) | (°F) | (°F) | (°F) | (°F) |
| 1 | 15 | 17.220 | 24.350 | 7.130 | 80 | 82 | 81 | 78 | 79 | 78.5 |
| 2 | 15 | 24.350 | 31.563 | 7.213 | 82 | 83 | 82.5 | 79 | 79 | 79 |
| 3 | 15 | 31.563 | 38.780 | 7.217 | 82 | 83 | 82.5 | 79 | 81 | 80 |

| | Reference Meter | | | | | | Meter Box | Reference |
|---|---|---|---|---|---|---|---|---|
| | Gas Volume | | | Meter Temperature | | | Correction | Orifice Press |
| | Initial | Final | Net | Initial | Final | Avg. | Factor | $\Delta H_@$ |
| Trial | ($ft^3$) | ($ft^3$) | ($ft^3$) | (°F) | (°F) | (°F) | γ | (in. $H_2O$) |
| 1 | 623.622 | 630.833 | 7.211 | 77 | 77 | 77 | 1.020 | 1.82 |
| 2 | 630.833 | 638.141 | 7.308 | 78 | 78 | 78 | 1.021 | 1.77 |
| 3 | 638.141 | 645.425 | 7.284 | 78 | 78.5 | 78.25 | 1.018 | 1.79 |

ΔH = 1.0

| | Dry Gas Meter MB-10 | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Trial | Gas Volume | | | Meter Temperatures | | | | | |
| | Duration | Initial | Final | Net | Initial, Inlet | Final, Inlet | Avg. Inlet | Initial, Outlet | inal, Outle | Avg. Outlet |
| Trial | (min) | ($ft^3$) | ($ft^3$) | ($ft^3$) | (°F) | (°F) | (°F) | (°F) | (°F) | (°F) |
| 1 | 10 | 38.946 | 44.490 | 5.544 | 81 | 83 | 82 | 80 | 80 | 80 |
| 2 | 10 | 44.490 | 50.050 | 5.560 | 83 | 84 | 83.5 | 80 | 80 | 80 |
| 3 | 10 | 50.050 | 55.585 | 5.535 | 84 | 84 | 84 | 80 | 80 | 80 |

| | Reference Meter | | | | | | Meter Box | Reference |
|---|---|---|---|---|---|---|---|---|
| | Gas Volume | | | Meter Temperature | | | Correction | Orifice Press |
| | Initial | Final | Net | Initial | Final | Avg. | Factor | $\Delta H_@$ |
| Trial | ($ft^3$) | ($ft^3$) | ($ft^3$) | (°F) | (°F) | (°F) | γ | (in. $H_2O$) |
| 1 | 645.614 | 651.22 | 5.606 | 78 | 78 | 78 | 1.019 | 1.79 |
| 2 | 651.220 | 656.829 | 5.609 | 78 | 78 | 78 | 1.018 | 1.78 |
| 3 | 656.829 | 662.435 | 5.606 | 78 | 78 | 78 | 1.023 | 1.78 |

# APPENDIX E

# QA/QC DATA

19. Method 3A System Bias Check, %. Values are for the oxygen, final upscale check.

$$\text{Sys Bias \%} = (100)\,(\text{Instr. Response}_{\text{CAL ERR}} - \text{Instr. Response}_{\text{SYS CAL}})/\text{Span}$$

$$\text{Sys Bias \%} = (100)\ (18.9 - 18.9)\ /\ 25$$

$$\text{Sys Bias \%} = 0.0\ \%$$

20. Method 3A Drift, %. Values are for the oxygen, upscale check.

$$\text{Drift \%} = (100)\,(\text{Instr. Response}_{\text{FINAL SYS CAL}} - \text{Instr. Response}_{\text{INIT SYS CAL}})/\text{Span}$$

$$\text{Drift \%} = (100)\ (18.9 - 19.1)\ /\ 25$$

$$\text{Drift \%} = -0.8\ \%$$

21. Method 3A Zero & Upscale Sampling System Check Adjustment. Values are for oxygen, %.

$$C_{gas} = (C_{avg} - C_O)\frac{C_{ma}}{C_m - C_O}$$

$$C_{gas} = (6.7 - 0.35)\frac{19.17}{19.0 - 0.35}$$

$$C_{gas} = 6.5\ \%$$

Where:
$C_{gas}$ = Adjusted gas concentration, ppm or %
$C_{avg}$ = Average unadjusted gas concentration from analyzer
$C_O$ = Average of zero gas initial & final system cal. bias check
$C_{ma}$ = Actual concentration of the upscale calibration gas
$C_m$ = Average of upscale initial & final system cal. bias check

16. CEM Pollutant (HCl) Concentration, $ppm_d$

$$ppm_d = ppm_w / (1 - B_{ws}/100)$$

$$ppm_d = 1.4 / (1 - 29.7/100)$$

$$ppm_d = 1.99 \text{ ppm}$$

17. CEM Pollutant (HCl) Emission Rate, lb/hr.

$$lb/hr = \frac{(60)\ (ppm_d)\ (Fwt)\ (Q_{s(std)})}{(10^6)\ (385.3)}$$

$$lb/hr = \frac{(60)\ (1.99)\ (44.11)\ (29{,}542)}{(10^6)\ (385.3)}$$

$$lb/hr = 0.405\ lb/hr$$

18. Method 3A Calibration Error, %. Values are for the oxygen, mid range.

$$\text{Cal Err \%} = (100)\ (\text{Instrument Response} - \text{Calibration Gas Concentration}) / \text{Span}$$

$$\text{Cal Err \%} = (100)\ (11.2 - 11.1) / 25$$

$$\text{Cal Err \%} = 0.4\ \%$$

13. Pollutant (2378 TCDD) concentration, ng/dscm.

$$\text{ng/dscm} = \frac{\text{ng}}{V_{m(std)m^3}}$$

$$\text{ng/dscm} = \frac{0.0102}{2.971}$$

$$\text{ng/dscm} = 0.00343 \quad \text{ng/dscm}$$

14. Pollutant (2378 TCDD) concentration, ng/dscm adjusted to 7 percent oxygen.

$$\text{ng/dscm@7\%O}_2 = (\text{ng/dscm}) \frac{(20.9 - 7)}{(20.9 - \%O_2)}$$

$$\text{ng/dscm@7\%O}_2 = (0.00343) \frac{13.9}{(20.9 - 6.5)}$$

$$\text{ng/dscm@7\%O}_2 = 0.00331 \quad \text{ng/dscm@7\%O}_2$$

15. Pollutant (2378 TCDD) emission rate, μg/hr.

$$\mu\text{g/hr} = \frac{(60)\ (\text{ng})\ (Q_{s(std)})}{(10^3)\ (V_{m(std)})}$$

$$\mu\text{g/hr} = \frac{(60)\ (0.0102)\ (29{,}542)}{(10^3)\ (104.912)}$$

$$\mu\text{g/hr} = 0.172 \quad \mu\text{g/hr}$$

10. Stack gas volumetric flow rate at stack conditions, acfm.

$$Q_a = (60)\ (A)\ (v_s)$$

$$Q_a = (60)\ (27.11)\ (30.43)$$

$$Q_a = 49{,}492 \text{ acfm}$$

11. Dry stack gas volumetric flow rate at standard conditions, dscfm.

$$Q_{s(std)} = 17.64\ Q_a \frac{P_s}{(t_s + 460)} \left(1 - B_{ws}/100\right)$$

$$Q_{s(std)} = (17.64)\ (49{,}492) \left(\frac{29.68}{156.4 + 460}\right) \left(1 - 29.7/100\right)$$

$$Q_{s(std)} = 29{,}542 \text{ dscfm}$$

12. Dry stack gas volumetric flow rate at standard conditions, dscmm.

$$Q_{s(cmm)} = Q_{s(std)}\ 0.028317$$

$$Q_{s(cmm)} = (29{,}542)\ (0.028317)$$

$$Q_{s(cmm)} = 837 \text{ dscmm}$$

7. Absolute stack gas pressure, in. Hg.

$$P_s = P_{bar} + \frac{P_{static}}{13.6}$$

$$P_s = 29.7 + \frac{-0.23}{13.6}$$

$$P_s = 29.68 \text{ inches Hg}$$

8. Stack velocity at stack conditions, fps.

$$v_s = 85.49\ C_p \left(\sqrt{\Delta p}\right)_{avg} \sqrt{\frac{t_s + 460}{M_s\ P_s}}$$

$$v_s = (85.49)(0.84)(0.4881) \sqrt{\frac{(156.4 + 460)}{(27.56)(29.68)}}$$

$$v_s = 30.43 \text{ fps}$$

9. Isokinetic Variation.

$$\%I = \frac{\left(V_{m(std)}\right)\left(t_s + 460\right)(17.32)}{\left(v_s\right)\left(D_n^2\right)(\theta)\left(P_s\right)\left(1 - B_{ws}/100\right)}$$

$$\%I = \frac{(104.912)\ (156.4 + 460)\ (17.32)}{(30.43)\ (0.310)^2\ (180)\ (29.68)\ (1 - 29.7/100)}$$

$$\%I = 102.1$$

4. Moisture content in stack gas, as measured.

$$B_{ws} = \frac{V_{w(std)}}{\left(V_{m(std)} + V_{w(std)}\right)}(100)$$

$$B_{ws} = \frac{57.661}{104.912 + 57.661}(100)$$

$$B_{wr} = 35.5$$

Moisture content in stack gas, at saturation. Used if lower than measured moisture.

$$B_{ws(sat)} = 10^{(6.691 - (3144/(ts + 390.86)))} \,/\, Ps * 100$$

$$B_{ws(sat)} = 10^{(6.691 - (3144/(156 + 390.86)))} \,/\, 29.68 * 100$$

$$B_{ws(sat)} = 29.5$$

5. Dry molecular weight of stack gas, lb/lb-mol.

$$M_d = 0.44(\%CO_2) + 0\,32(\%O_2) + 0.28(\%N_2 + \%CO)$$

$$M_d = 0.44(20.9) + 0.32(6.5) + 0.28(72.6 + 0)$$

$$M_d = 31.60 \text{ lb/lb}\cdot\text{mol}$$

6. Molecular weight of stack gas, lb/lb-mol.

$$M_s = M_d(1 - B_{ws}/100) + 18(B_{ws}/100)$$

$$M_s = 31.60(1 - 29.73/100) + 18(29.73/100)$$

$$M_s = 31.60(0.7027) + 18(0.2973)$$

$$M_s = 22.205 + 5.351$$

$$M_s = 27.56 \text{ lb/lb}\cdot\text{mol}$$

Example Calculations
Huron Lime Company - Huron, Ohio
US EPA Method 23-PCDDs/PCDFs
(Using Data from Run M23-O-3)

Note: Discrepancies may exist between the computer generated reported results, which use more significant figures, and the values manually calculated from the displayed values.

1. Volume of dry gas sampled corrected to standard conditions of 68°F, 29.92 in. Hg, $ft^3$.

$$V_{m(std)} = 17.64 V_m \gamma \left( \frac{P_{bar} + \frac{\Delta H}{13.6}}{460 + t_m} \right)$$

$$V_{m(std)} = (17.64)(107.709)(1.021) \left( \frac{29.7 + \frac{1.27}{13.6}}{460 + 90.9} \right)$$

$$V_{m(std)} = 104.912 \text{ dscf}$$

2. Volume of dry gas sampled corrected to standard conditions of 68°F, 29.92 in. Hg, $m^3$.

$$V_{m(std)m^3} = V_{m(std)}(0.028317)$$

$$V_{m(std)m^3} = (104.912)(0.028317)$$

$$V_{m(std)m^3} = 2.971 \text{ dscm}$$

3. Volume of water vapor at standard conditions, $ft^3$.

$$V_{w(std)} = 0.04707 V_{1c}$$

$$V_{w(std)} = (0.04707)(1225.0)$$

$$V_{w(std)} = 57.661 \text{ scf}$$

# Nomenclature

| | |
|---|---|
| $\gamma$ | Meter Box Correction Factor |
| $\Delta H$ | Avg Meter Orifice Pressure, in. $H_2O$ |
| $P_{bar}$ | Barometric Pressure, inches Hg |
| $V_m$ | Sample Volume, $ft^3$ |
| $t_m$ | Average Meter Temperature. °F |
| $P_{static}$ | Stack Static Pressure, inches $H_2O$ |
| $t_s$ | Average Stack Temperature, °F |
| $V_{lc}$ | Condensate Collected, ml |
| $CO_2$ | Carbon Dioxide content, % by volume |
| $O_2$ | Oxygen content, % by volume |
| $N_2$ | Nitrogen content, % by volume |
| $C_p$ | Pitot Tube Coefficient |
| $\Delta p^{1/2}$ | Average Square Root Dp, (in. $H_2O)^{1/2}$ |
| $\Theta$ | Sample Run Duration, minutes |
| $D_n$ | Nozzle Diameter, inches |
| $A_n$ | Nozzle Area, $ft^2$ |
| $V_{m(std)}$ | Standard Meter Volume, dscf |
| $V_{m(std)m3}$ | Standard Meter Volume, dscm |
| $P_s$ | Stack Pressure, inches Hg |
| $B_{ws}$ | Moisture, % by volume |
| $V_{w(std)}$ | Standard Water Vapor Volume, $ft^3$ |
| $1\text{-}B_{ws}$ | Dry Mole Fraction |
| $M_d$ | Molecular Weight, dry, lb/lb•mole |
| $M_s$ | Molecular Weight, wet, lb/lb•mole |
| $v_s$ | Stack Gas Velocity, ft/s |
| A | Stack Area, $ft^2$ |
| $Q_a$ | Stack Gas Volumetric flow, acfm |
| $Q_{s(std)}$ | Stack Gas Volumetric flow, dscfm |
| $Q_{s(cmm)}$ | Stack Gas Volumetric flow, dscmm |
| I | Isokinetic Sampling Ratio, % |
| ng/dscm | Concentration, ng/dscm |
| ng/dscm@7%$O_2$ | Concentration, ng/dscm adjusted to 7% oxygen |
| ug/hr | Emission Rate, ug/hr |
| $ppm_d$ | Concentration, parts per million, dry |
| $ppm_w$ | Concentration, parts per million, wet |
| lb/hr | Emission Rate, pounds per hour |

# APPENDIX D

# EXAMPLE EQUATIONS

# Summary of Stack Gas Parameters and Test Results
# Huron Lime Company - Huron, Ohio
## US EPA Test Method 23 - PCDDs / PCDFs
## Kiln No. 3 Scrubber Stack
Page 6 of 6

| | | |
|---|---|---|
| | **RUN NUMBER** | **M23-O-3** |
| | **RUN DATE** | **08/31/98** |
| | **RUN TIME** | **1750-2128** |
| | EMISSIONS DATA - Continued | |
| | Furans - Continued | |
| | Total HpCDF | |
| ng | Catch, ng | 0.0104 |
| ng/dscm | Concentration, ng/dscm, as measured | 0.00350 |
| µg/hr | Emission Rate, µg/hr | 0.176 |
| | OCDF | |
| ng | Catch, ng | (0.0056) |
| ng/dscm | Concentration, ng/dscm, as measured | (0.00189) |
| µg/hr | Emission Rate, µg/hr | (0.0946) |
| | Total PCDF | |
| ng | Catch, ng | (6.7144) |
| ng/dscm | Concentration, ng/dscm, as measured | (2.260) |
| µg/hr | Emission Rate, µg/hr | (113.4) |
| | Total PCDD + PCDF | |
| ng | Catch, ng | (6.9586) |
| ng/dscm | Concentration, ng/dscm, as measured | (2.342) |
| µg/hr | Emission Rate, µg/hr | (117.6) |

( ) Not Detected. Value shown is the detection limit and is included in totals.

{ } Estimated Maximum Possible Concentration. EMPC values are included in totals.

# Summary of Stack Gas Parameters and Test Results
## Huron Lime Company - Huron, Ohio
## US EPA Test Method 23 - PCDDs / PCDFs
## Kiln No. 3 Scrubber Stack
## Page 5 of 6

| | | |
|---|---|---|
| | RUN NUMBER | M23-O-3 |
| | RUN DATE | 08/31/98 |
| | RUN TIME | 1750-2128 |
| | EMISSIONS DATA - Continued | |
| | Furans - Continued | |
| | <u>123678 HxCDF</u> | |
| ng | Catch, ng | 0.0119 |
| ng/dscm | Concentration, ng/dscm, as measured | 0.00401 |
| µg/hr | Emission Rate, µg/hr | 0.201 |
| | <u>234678 HxCDF</u> | |
| ng | Catch, ng | 0.0068 |
| ng/dscm | Concentration, ng/dscm, as measured | 0.00229 |
| µg/hr | Emission Rate, µg/hr | 0.115 |
| | <u>123789 HxCDF</u> | |
| ng | Catch, ng | (0.0016) |
| ng/dscm | Concentration, ng/dscm, as measured | (0.000539) |
| µg/hr | Emission Rate, µg/hr | (0.0270) |
| | <u>Total HxCDF</u> | |
| ng | Catch, ng | 0.0784 |
| ng/dscm | Concentration, ng/dscm, as measured | 0.0264 |
| µg/hr | Emission Rate, µg/hr | 1.32 |
| | <u>1234678 HpCDF</u> | |
| ng | Catch, ng | 0.0105 |
| ng/dscm | Concentration, ng/dscm, as measured | 0.00353 |
| µg/hr | Emission Rate, µg/hr | 0.177 |
| | <u>1234789 HpCDF</u> | |
| ng | Catch, ng | (0.0034) |
| ng/dscm | Concentration, ng/dscm, as measured | (0.00114) |
| µg/hr | Emission Rate, µg/hr | (0.0574) |

( ) Not Detected. Value shown is the detection limit and is included in totals.

{ } Estimated Maximum Possible Concentration. EMPC values are included in totals.

## Summary of Stack Gas Parameters and Test Results
## Huron Lime Company - Huron, Ohio
## US EPA Test Method 23 - PCDDs / PCDFs
## Kiln No. 3 Scrubber Stack
## Page 4 of 6

| | | |
|---|---|---|
| | **RUN NUMBER** | **M23-O-3** |
| | **RUN DATE** | **08/31/98** |
| | **RUN TIME** | **1750-2128** |
| | EMISSIONS DATA - Continued | |
| | FURANS | |
| | 2378 TCDF | |
| ng | Catch, ng | 0.309 |
| ng/dscm | Concentration, ng/dscm, as measured | 0.104 |
| µg/hr | Emission Rate, µg/hr | 5.22 |
| | Total TCDF | |
| ng | Catch, ng | 5.68 |
| ng/dscm | Concentration, ng/dscm, as measured | 1.91 |
| µg/hr | Emission Rate, µg/hr | 96.0 |
| | 12378 PeCDF | |
| ng | Catch, ng | 0.101 |
| ng/dscm | Concentration, ng/dscm, as measured | 0.0340 |
| µg/hr | Emission Rate, µg/hr | 1.71 |
| | 23478 PeCDF | |
| ng | Catch, ng | 0.0602 |
| ng/dscm | Concentration, ng/dscm, as measured | 0.0203 |
| µg/hr | Emission Rate, µg/hr | 1.02 |
| | Total PeCDF | |
| ng | Catch, ng | 0.940 |
| ng/dscm | Concentration, ng/dscm, as measured | 0.316 |
| µg/hr | Emission Rate, µg/hr | 15.9 |
| | 123478 HxCDF | |
| ng | Catch, ng | 0.0207 |
| ng/dscm | Concentration, ng/dscm, as measured | 0.00697 |
| µg/hr | Emission Rate, µg/hr | 0.350 |

( ) Not Detected. Value shown is the detection limit and is included in totals.

{ } Estimated Maximum Possible Concentration. EMPC values are included in totals.

## Summary of Stack Gas Parameters and Test Results
## Huron Lime Company - Huron, Ohio
## US EPA Test Method 23 - PCDDs / PCDFs
## Kiln No. 3 Scrubber Stack
## Page 3 of 6

| | |
|---|---|
| RUN NUMBER | M23-O-3 |
| RUN DATE | 08/31/98 |
| RUN TIME | 1750-2128 |

EMISSIONS DATA -Continued

DIOXINS - Continued

| | | |
|---|---|---|
| | 123789 HxCDD | |
| ng | Catch, ng | 0.0031 |
| ng/dscm | Concentration, ng/dscm, as measured | 0.00104 |
| µg/hr | Emission Rate, µg | 0.0524 |
| | Total HxCDD | |
| ng | Catch, ng | 0.0148 |
| ng/dscm | Concentration, ng/dscm, as measured | 0.00498 |
| µg/hr | Emission Rate, µg/hr | 0.250 |
| | 1234678 HpCDD | |
| ng | Catch, ng | {0.0075} |
| ng/dscm | Concentration, ng/dscm, as measured | {0.00252} |
| µg/hr | Emission Rate, µg/hr | {0.127} |
| | Total HpCDD | |
| ng | Catch, ng | (0.0017) |
| ng/dscm | Concentration, ng/dscm, as measured | (0.000572) |
| µg/hr | Emission Rate, µg/hr | (0.0287) |
| | OCDD | |
| ng | Catch, ng | {0.0319} |
| ng/dscm | Concentration, ng/dscm, as measured | {0.0107} |
| µg/hr | Emission Rate, µg/hr | {0.539} |
| | Total PCDD | |
| ng | Catch, ng | (0.2442) |
| ng/dscm | Concentration, ng/dscm, as measured | (0.0822) |
| µg/hr | Emission Rate, µg/hr | (4.13) |

( ) Not Detected. Value shown is the detection limit and is included in totals.

{ } Estimated Maximum Possible Concentration. EMPC values are included in totals

## Summary of Stack Gas Parameters and Test Results
## Huron Lime Company - Huron, Ohio
## US EPA Test Method 23 - PCDDs / PCDFs
## Kiln No. 3 Scrubber Stack
## Page 2 of 6

| | | |
|---|---|---|
| | **RUN NUMBER** | **M23-O-3** |
| | **RUN DATE** | **08/31/98** |
| | **RUN TIME** | **1750-2128** |
| | EMISSIONS DATA | |
| | DIOXINS: | |
| | 2378 TCDD | |
| ng | Catch, ng | 0.0102 |
| ng/dscm | Concentration, ng/dscm, as measured | 0.00343 |
| µg/hr | Emission Rate, µg/hr | 0.172 |
| | Total TCDD | |
| ng | Catch, ng | 0.151 |
| ng/dscm | Concentration, ng/dscm, as measured | 0.0508 |
| µg/hr | Emission Rate, µg/hr | 2.55 |
| | 12378 PeCDD | |
| ng | Catch, ng | 0.0038 |
| ng/dscm | Concentration, ng/dscm, as measured | 0.00128 |
| µg/hr | Emission Rate, µg/hr | 0.0642 |
| | Total PeCDD | |
| ng | Catch, ng | 0.0448 |
| ng/dscm | Concentration, ng/dscm, as measured | 0.0151 |
| µg/hr | Emission Rate, µg/hr | 0.757 |
| | 123478 HxCDD | |
| ng | Catch, ng | 0.0021 |
| ng/dscm | Concentration, ng/dscm, as measured | 0.000707 |
| µg/hr | Emission Rate, µg/hr | 0.0355 |
| | 123678 HxCDD | |
| ng | Catch, ng | 0.0030 |
| ng/dscm | Concentration, ng/dscm, as measured | 0.00101 |
| µg/hr | Emission Rate, µg/hr | 0.0507 |

( ) Not Detected. Value shown is the detection limit and is included in totals.

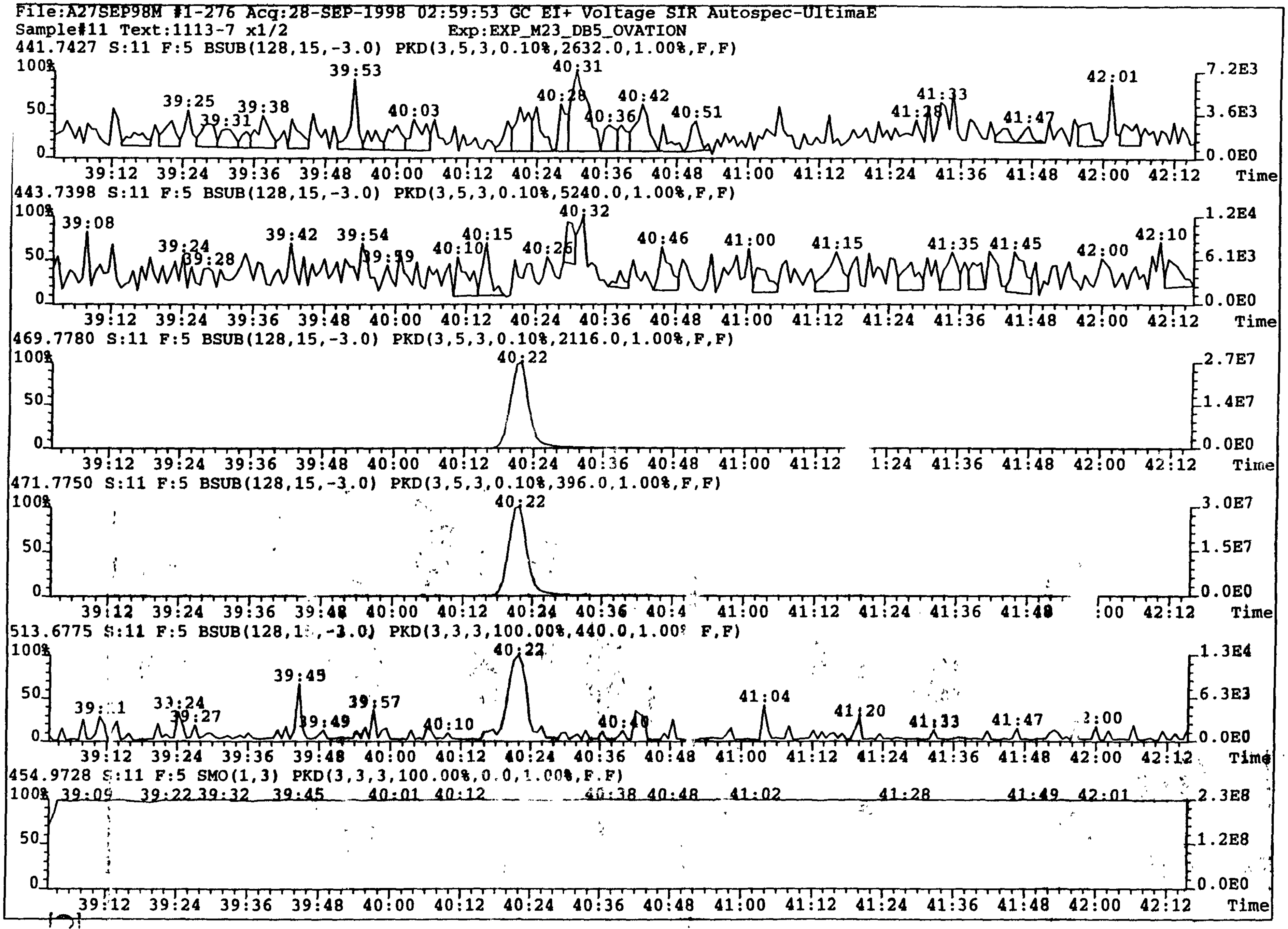
{ } Estimated Maximum Possible Concentration. EMPC values are included in totals.

# Summary of Stack Gas Parameters and Test Results
# Air Emissions Screening Test
# Huron Lime Company - Huron, Ohio
# US EPA Test Method 23 - PCDDs / PCDFs
# Kiln No. 3 Scrubber Stack
## Page 1 of 6

|  |  |  |
|---|---|---|
|  | **RUN NUMBER** | **M23-O-3** |
|  | **RUN DATE** | [illegible] |
|  | **RUN TIME** | **1750-2128** |
|  | MEASURED DATA |  |
| $\gamma$ | Meter Box Correction Factor | 1.021 |
| $\Delta H$ | Avg Meter Orifice Pressure, in. $H_2O$ | 1.27 |
| $P_{bar}$ | Barometric Pressure, inches Hg | 29.70 |
| $V_m$ | Sample Volume, $ft^3$ | 107.709 |
| $T_m$ | Average Meter Temperature, °F | 90.9 |
| $P_{static}$ | Stack Static Pressure, inches $H_2O$ | -0.23 |
| $T_s$ | Average Stack Temperature, °F | 156 |
| $V_{lc}$ | Condensate Collected, ml | 1225.0 |
| $CO_2$ | Carbon Dioxide content, % by volume | 20.9 |
| $O_2$ | Oxygen content, % by volume | 6.5 |
| $N_2$ | Nitrogen content, % by volume | [illegible] |
| $C_p$ | Pitot Tube Coefficient | 0.84 |
| $\Delta p^{1/2}$ | Average Square Root $\Delta p$, (in. $H_2O)^{1/2}$ | 0.4881 |
| $\Theta$ | Sample Run Duration, minutes | 180.0 |
| $D_n$ | Nozzle Diameter, inches | 0.310 |
|  | CALCULATED DATA |  |
| $A_n$ | Nozzle Area, $ft^2$ | 0.00052 |
| $V_{m(std)}$ | Standard Meter Volume, dscf | 104.912 |
| $V_{m(std)}$ | Standard Meter Volume, dscm | 2.971 |
| $P_s$ | Stack Pressure, inches Hg | 29.68 |
| $B_{ws}$ | Moisture, % by volume | 35.5 |
| $B_{ws(sat)}$ | Moisture (at saturation), % by volume | 29.7 (used) |
| $V_{wstd}$ | Standard Water Vapor Volume, $ft^3$ | 57.661 |
| $1\text{-}B_{ws}$ | Dry Mole Fraction | 0.703 |
| $M_d$ | Molecular Weight (d.b.), lb/lb•mole | 31.60 |
| $M_s$ | Molecular Weight (w.b.), lb/lb•mole | 27.56 |
| $V_s$ | Stack Gas Velocity, ft/s | 30.43 |
| $A$ | Stack Area, $ft^2$ | 27.11 |
| $Q_a$ | Stack Gas Volumetric flow, acfm | 49,492 |
| $Q_s$ | Stack Gas Volumetric flow, dscfm | 29,542 |
| $Q_{s(cmm)}$ | Stack Gas Volumetric flow, dscmm | 837 |
| $I$ | Isokinetic Sampling Ratio, % | 102.1 |

# APPENDIX C

# CALCULATIONS & COMPUTER SUMMARIES



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Sample#11 Text:1113-7 x1/2 Exp:EXP_M23_DB5_OVATION

441.7427 S:11 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,2632.0,1.00%,F,F)

443.7398 S:11 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,5240.0,1.00%,F,F)

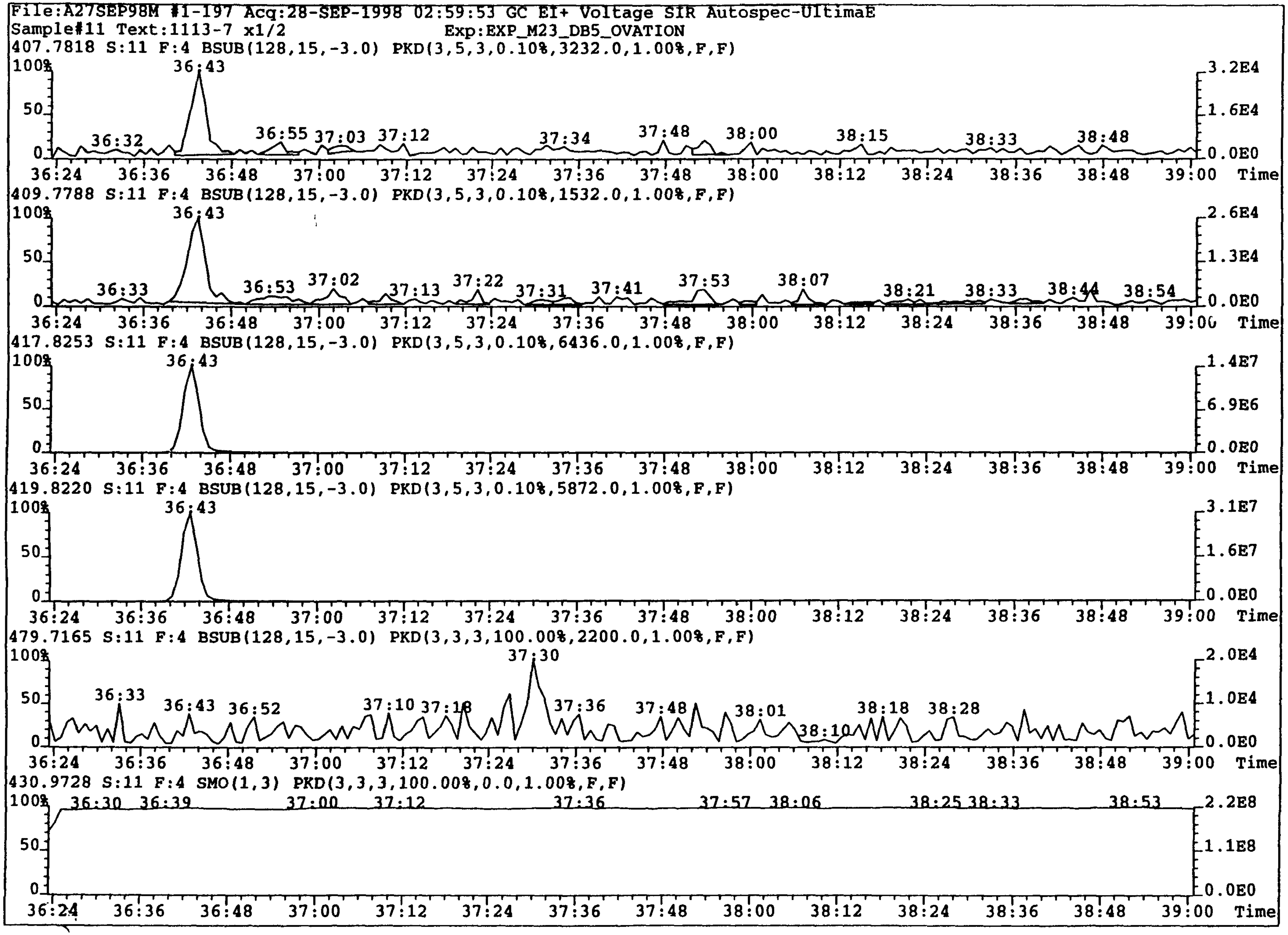
469.7780 S:11 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,2116.0,1.00%,F,F)

471.7750 S:11 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,396.0,1.00%,F,F)

513.6775 S:11 F:5 BSUB(128,15,-3.0) PKD(3,3,3,100.00%,440.0,1.00% F,F)

454.9728 S:11 F:5 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)

2



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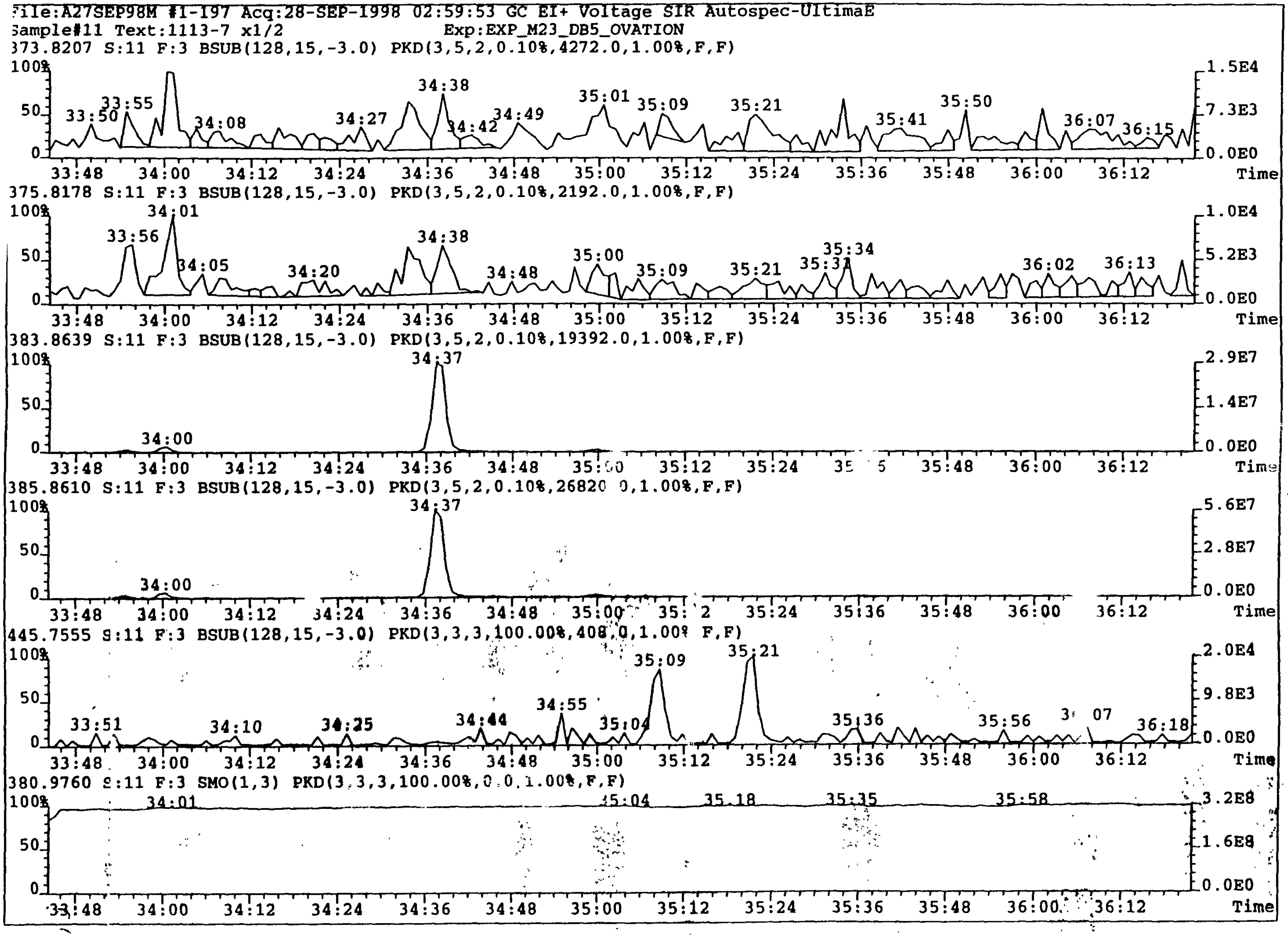
File:A27SEP98M #1-197 Acq:28-SEP-1998 02:59:53 GC EI+ Voltage SIR Autospec-UltimaE
Sample#11 Text:1113-7 x1/2           Exp:EXP_M23_DB5_OVATION
407.7818 S:11 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,3232.0,1.00%,F,F)
409.7788 S:11 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,1532.0,1.00%,F,F)
417.8253 S:11 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,6436.0,1.00%,F,F)
419.8220 S:11 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,5872.0,1.00%,F,F)
479.7165 S:11 F:4 BSUB(128,15,-3.0) PKD(3,3,3,100.00%,2200.0,1.00%,F,F)
430.9728 S:11 F:4 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)
```

209



File:A27SEP98M #1-197 Acq:28-SEP-1998 02:59:53 GC EI+ Voltage SIR Autospec-UltimaE
Sample#11 Text:1113-7 x1/2 Exp:EXP_M23_DB5_OVATION

373.8207 S:11 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,4272.0,1.00%,F,F)

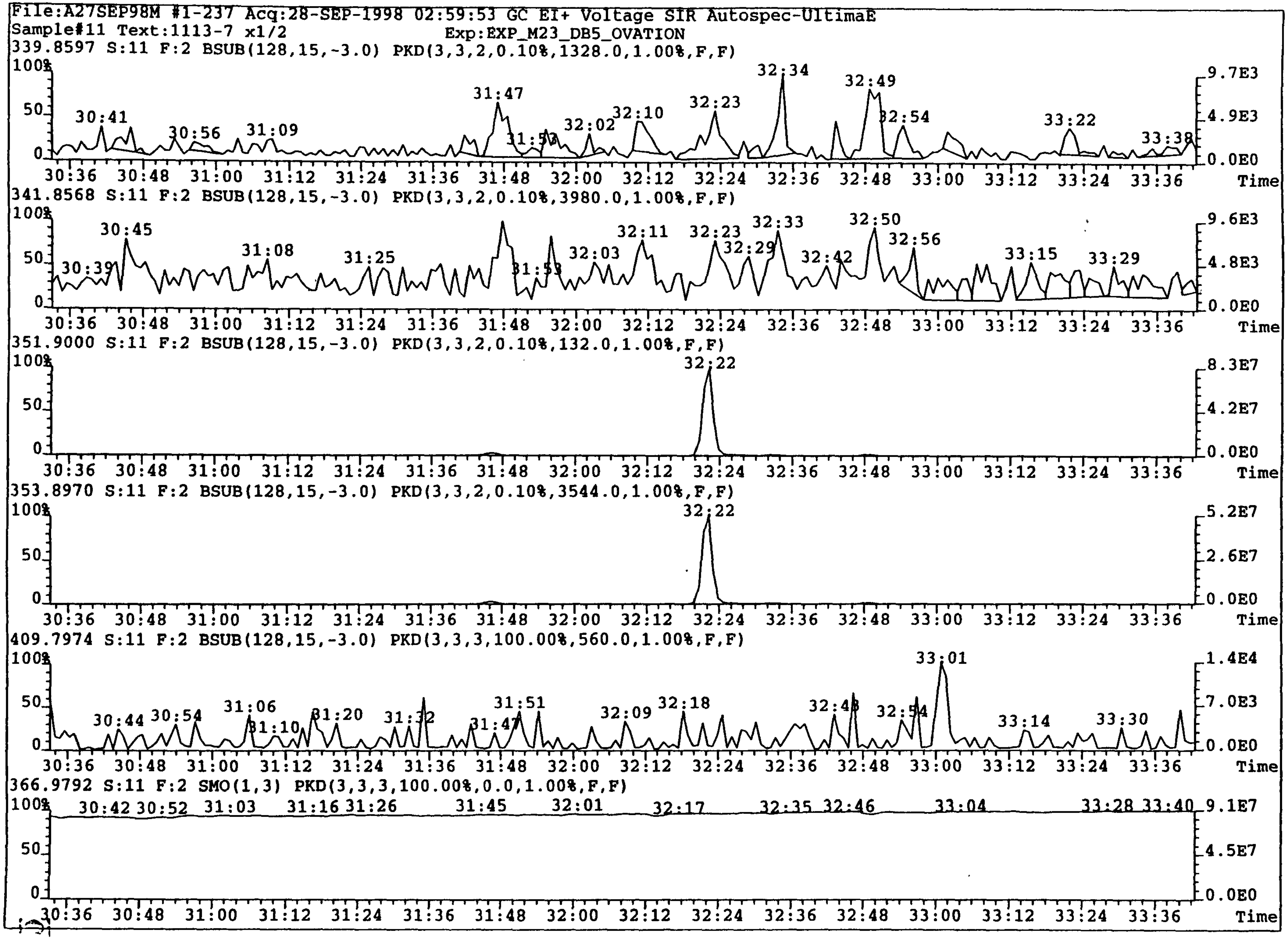
375.8178 S:11 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,2192.0,1.00%,F,F)

383.8639 S:11 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,19392.0,1.00%,F,F)

385.8610 S:11 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,26820.0,1.00%,F,F)

445.7555 S:11 F:3 BSUB(128,15,-3.0) PKD(3,3,3,100.00%,408.0,1.00% F,F)

380.9760 S:11 F:3 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)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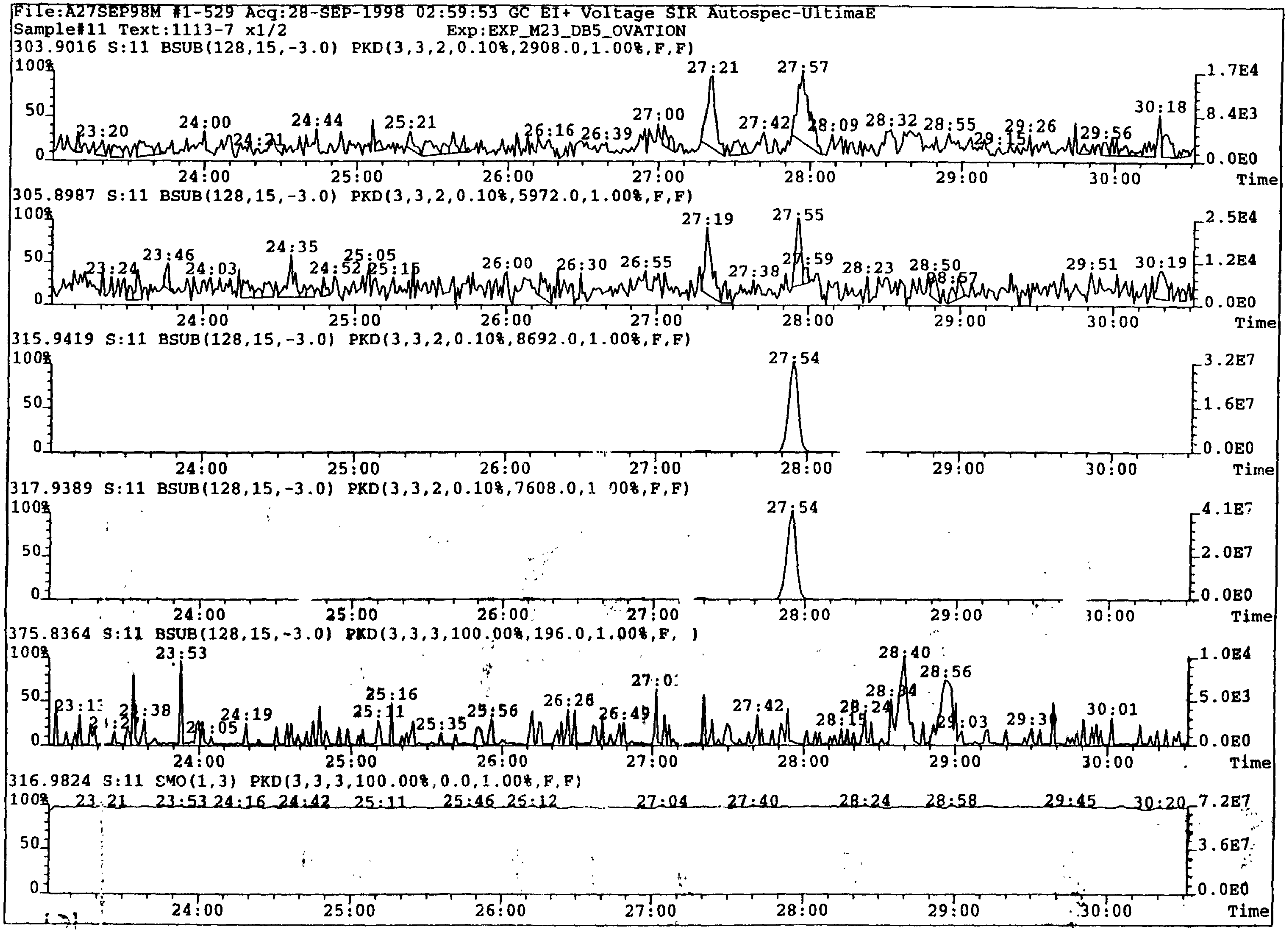
Sample#11 Text:1113-7 x1/2 Exp:EXP_M23_DB5_OVATION
339.8597 S:11 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,1328.0,1.00%,F,F)
100%
50
0
30:41
30:56
31:09
31:47
31:53
32:02
32:10
32:23
32:34
32:49
32:54
33:22
33:38
9.7E3
4.9E3
0.0E0
30:36 30:48 31:00 31:12 31:24 31:36 31:48 32:00 32:12 32:24 32:36 32:48 33:00 33:12 33:24 33:36
Time
341.8568 S:11 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,3980.0,1.00%,F,F)
100%
50
0
30:39
30:45
31:08
31:25
31:53
32:03
32:11
32:23
32:29
32:33
32:42
32:50
32:56
33:15
33:29
9.6E3
4.8E3
0.0E0
30:36 30:48 31:00 31:12 31:24 31:36 31:48 32:00 32:12 32:24 32:36 32:48 33:00 33:12 33:24 33:36
Time
351.9000 S:11 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,132.0,1.00%,F,F)
100%
50
0
32:22
8.3E7
4.2E7
0.0E0
30:36 30:48 31:00 31:12 31:24 31:36 31:48 32:00 32:12 32:24 32:36 32:48 33:00 33:12 33:24 33:36
Time
353.8970 S:11 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,3544.0,1.00%,F,F)
100%
50
0
32:22
5.2E7
2.6E7
0.0E0
30:36 30:48 31:00 31:12 31:24 31:36 31:48 32:00 32:12 32:24 32:36 32:48 33:00 33:12 33:24 33:36
Time
409.7974 S:11 F:2 BSUB(128,15,-3.0) PKD(3,3,3,100.00%,560.0,1.00%,F,F)
100%
50
0
30:44
30:54
31:06
31:10
31:20
31:32
31:47
31:51
32:09
32:18
32:43
32:54
33:01
33:14
33:30
1.4E4
7.0E3
0.0E0
30:36 30:48 31:00 31:12 31:24 31:36 31:48 32:00 32:12 32:24 32:36 32:48 33:00 33:12 33:24 33:36
Time
366.9792 S:11 F:2 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)
100%
50
0
30:42 30:52 31:03 31:16 31:26 31:45 32:01 32:17 32:35 32:46 33:04 33:28 33:40
9.1E7
4.5E7
0.0E0
30:36 30:48 31:00 31:12 31:24 31:36 31:48 32:00 32:12 32:24 32:36 32:48 33:00 33:12 33:24 33:36
Time



File:A27SEP98M #1-529 Acq:28-SEP-1998 02:59:53 GC EI+ Voltage SIR Autospec-UltimaE
Sample#11 Text:1113-7 x1/2 Exp:EXP_M23_DB5_OVATION

303.9016 S:11 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,2908.0,1.00%,F,F)

305.8987 S:11 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,5972.0,1.00%,F,F)

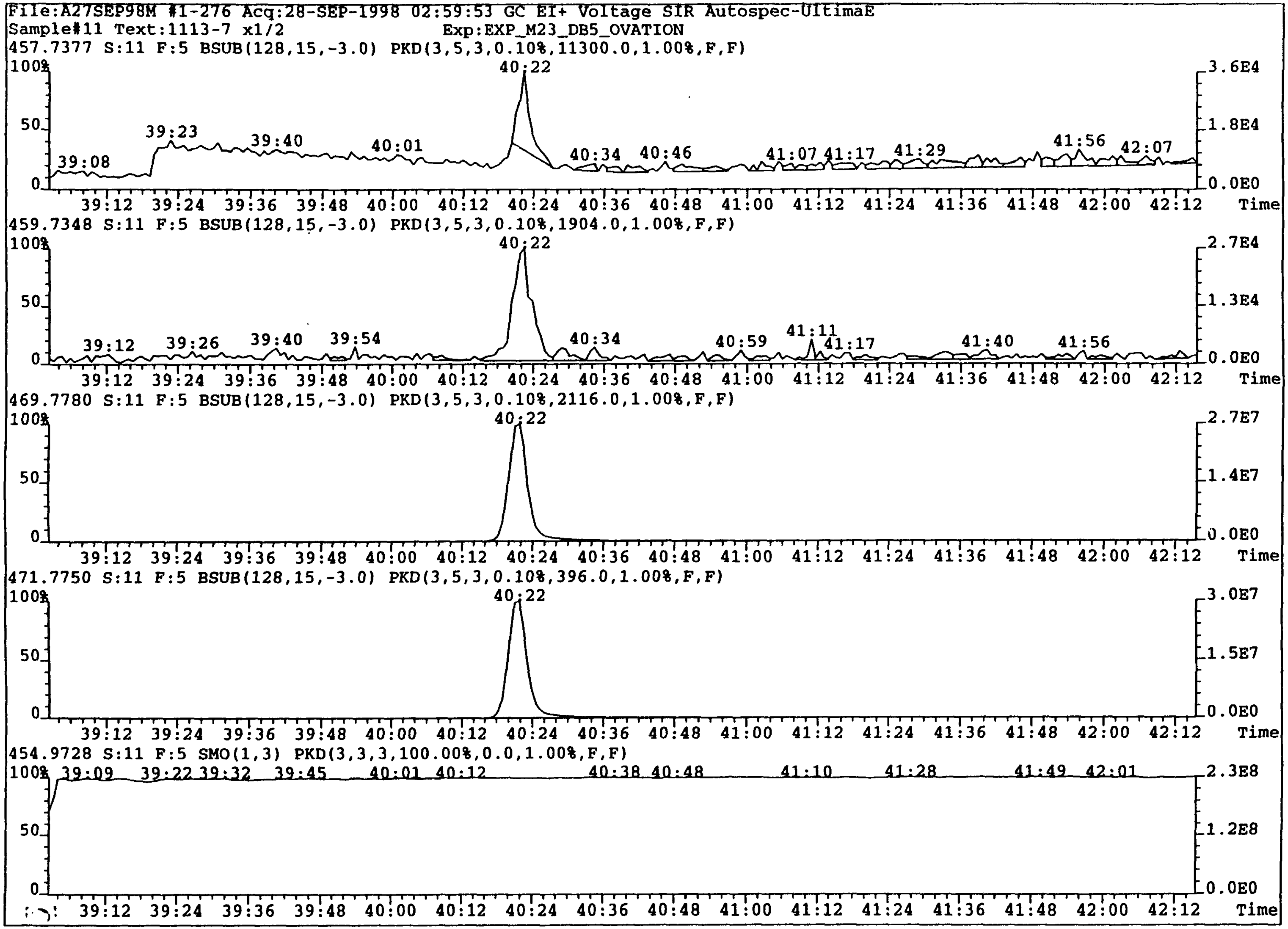
315.9419 S:11 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,8692.0,1.00%,F,F)

317.9389 S:11 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,7608.0,1.00%,F,F)

375.8364 S:11 BSUB(128,15,-3.0) PKD(3,3,3,100.00%,196.0,1.00%,F, )

316.9824 S:11 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)

2



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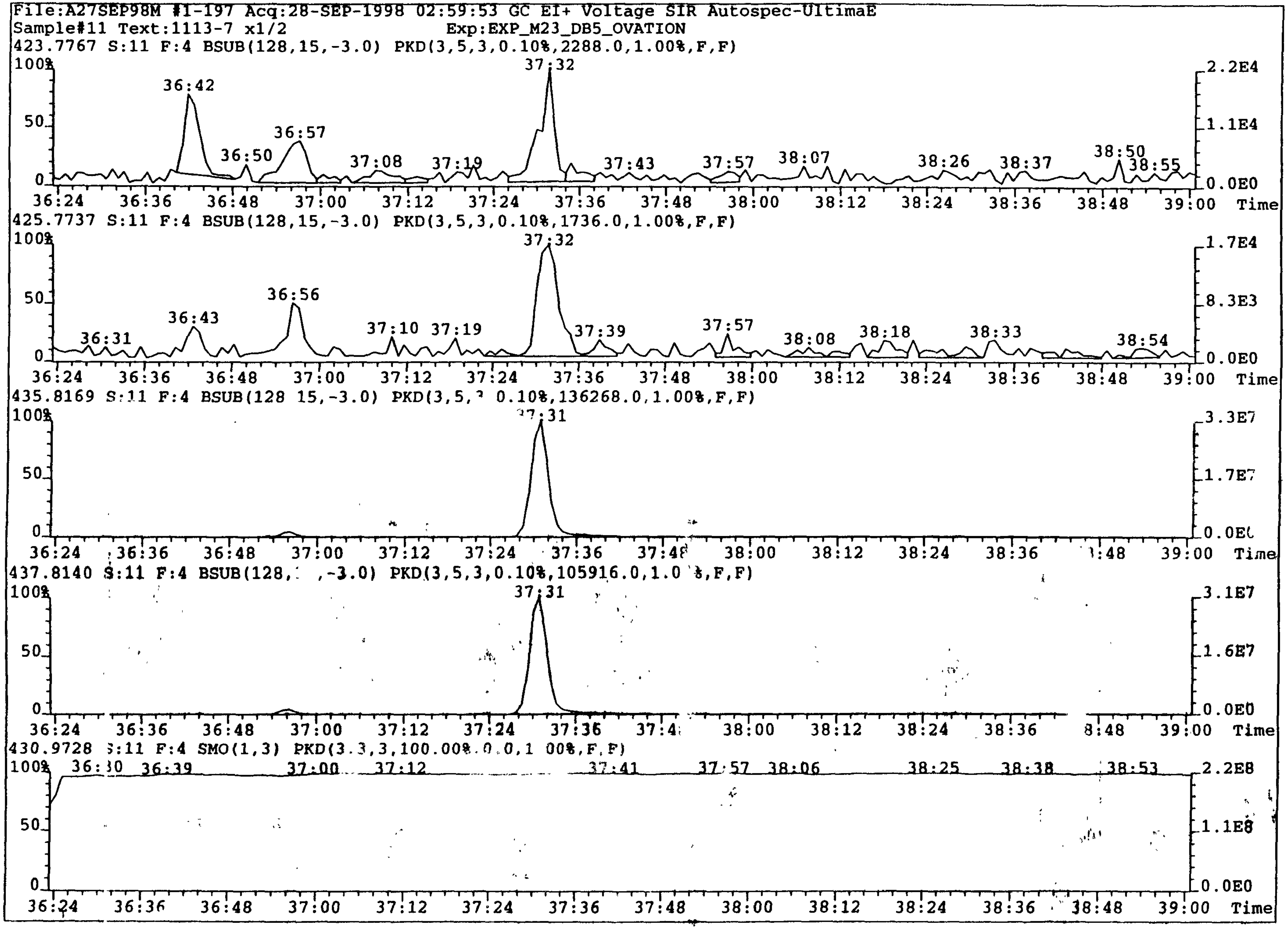
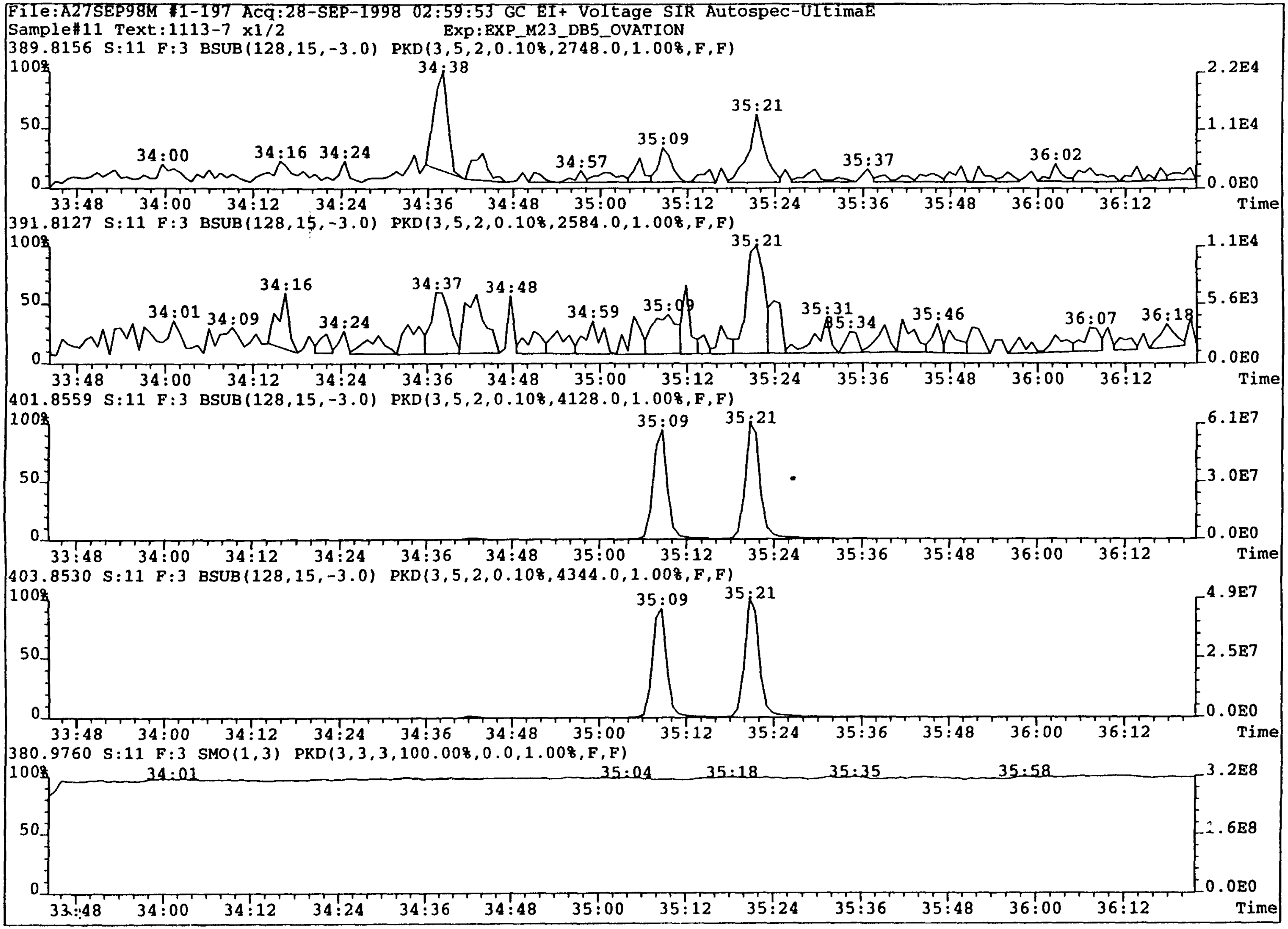
File:A27SEP98M #1-276 Acq:28-SEP-1998 02:59:53 GC EI+ Voltage SIR Autospec-UltimaE
Sample#11 Text:1113-7 x1/2              Exp:EXP_M23_DB5_OVATION
457.7377 S:11 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,11300.0,1.00%,F,F)
459.7348 S:11 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,1904.0,1.00%,F,F)
469.7780 S:11 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,2116.0,1.00%,F,F)
471.7750 S:11 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,396.0,1.00%,F,F)
454.9728 S:11 F:5 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)
```

205



```
File:A27SEP98M #1-197 Acq:28-SEP-1998 02:59:53 GC EI+ Voltage SIR Autospec-UltimaE
Sample#11 Text:1113-7 x1/2              Exp:EXP_M23_DB5_OVATION
423.7767 S:11 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,2288.0,1.00%,F,F)
425.7737 S:11 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,1736.0,1.00%,F,F)
435.8169 S:11 F:4 BSUB(128 15,-3.0) PKD(3,5,3 0.10%,136268.0,1.00%,F,F)
437.8140 S:11 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,105916.0,1.00%,F,F)
430.9728 S:11 F:4 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)
```

2



File:A27SEP98M #1-197 Acq:28-SEP-1998 02:59:53 GC EI+ Voltage SIR Autospec-UltimaE
Sample#11 Text:1113-7 x1/2 Exp:EXP_M23_DB5_OVATION

389.8156 S:11 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,2748.0,1.00%,F,F)

391.8127 S:11 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,2584.0,1.00%,F,F)

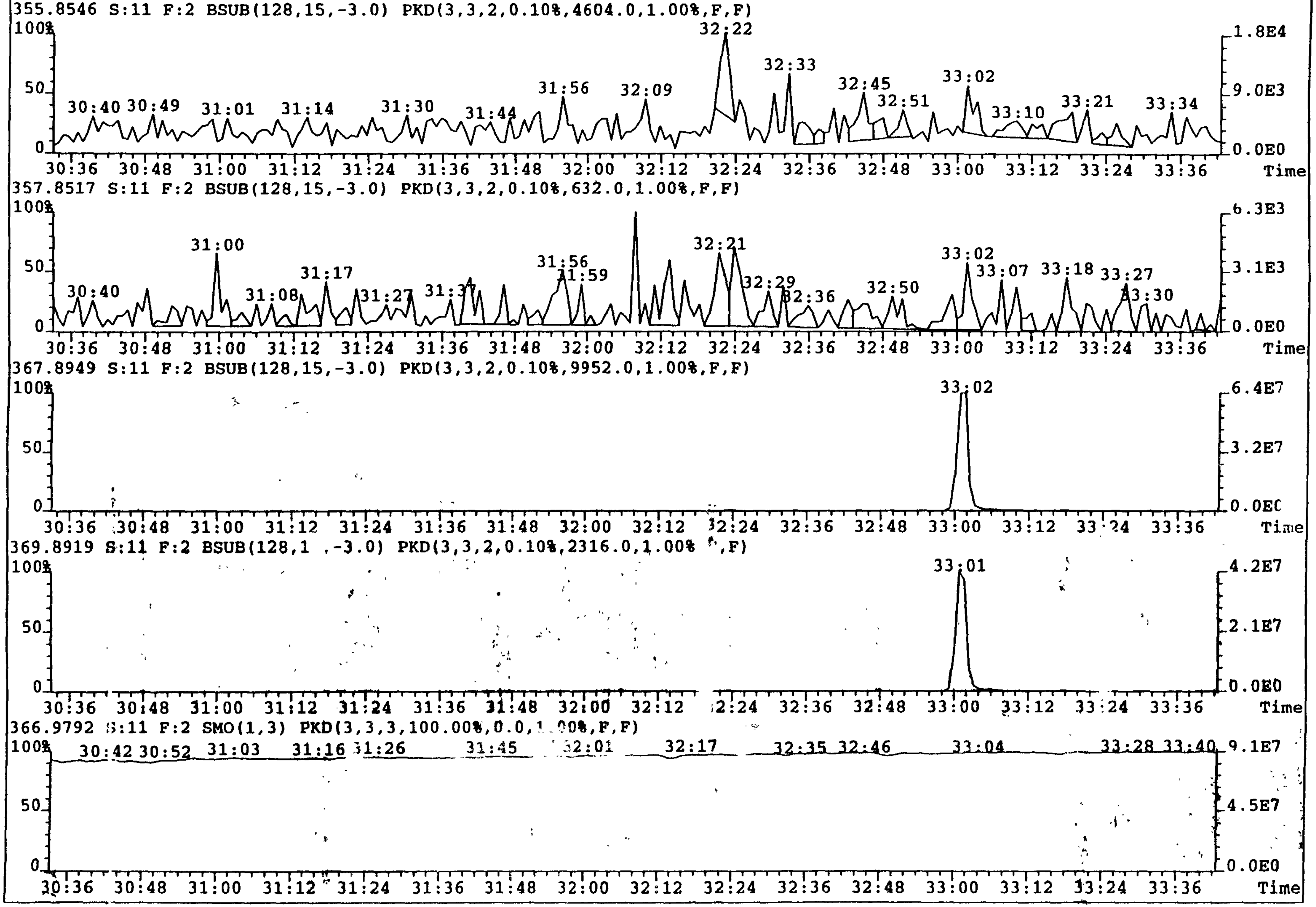
401.8559 S:11 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,4128.0,1.00%,F,F)

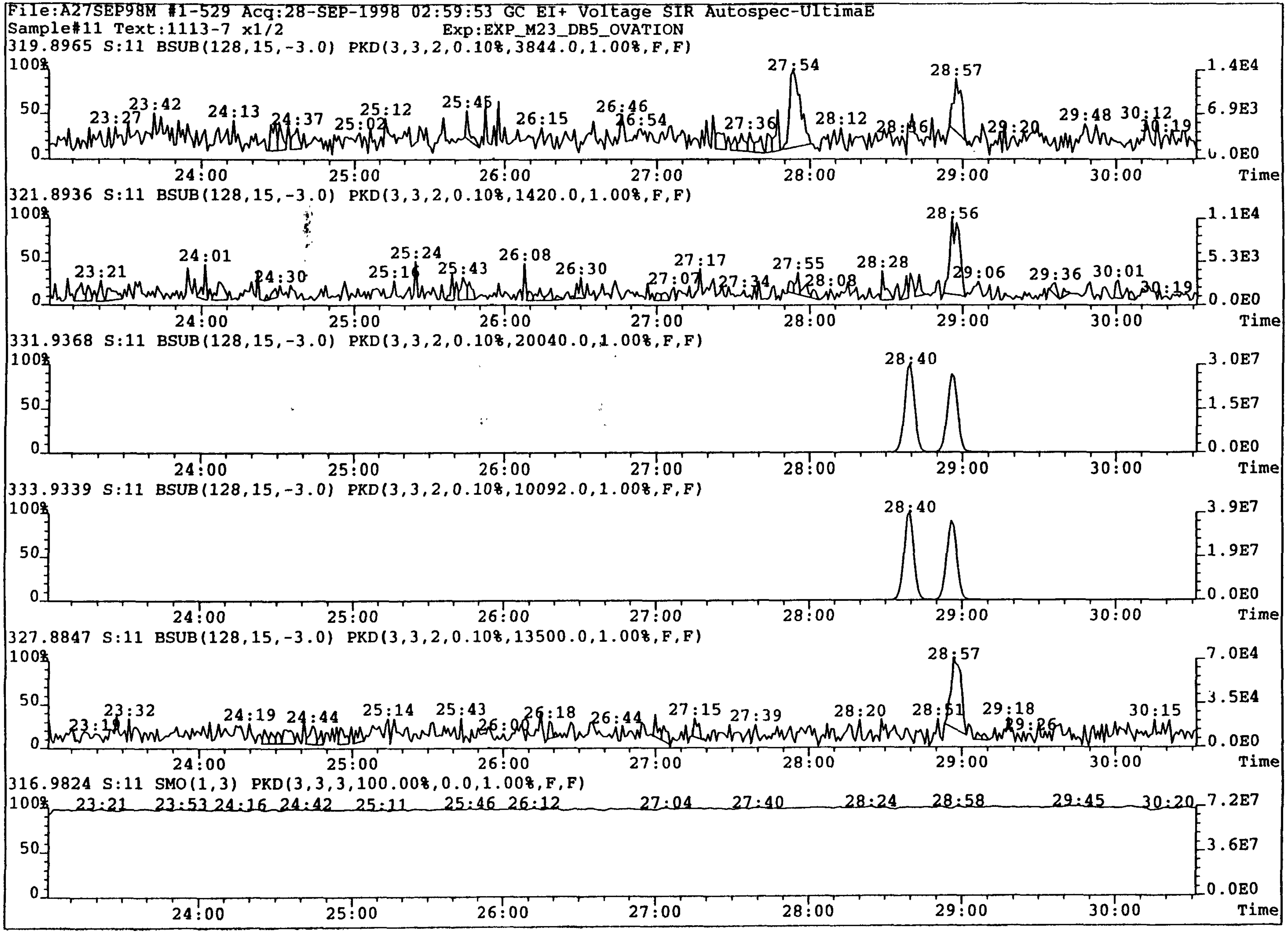
403.8530 S:11 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,4344.0,1.00%,F,F)

380.9760 S:11 F:3 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)

2

File:A27SEP98M #1-237 Acq:28-SEP-1998 02:59:53 GC EI+ Voltage SIR Autospec-UltimaE
Sample#11 Text:1113-7 x1/2 Exp:EXP_M23_DB5_OVATION





```
File:A27SEP98M #1-529 Acq:28-SEP-1998 02:59:53 GC EI+ Voltage SIR Autospec-UltimaE
Sample#11 Text:1113-7 x1/2              Exp:EXP_M23_DB5_OVATION
319.8965 S:11 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,3844.0,1.00%,F,F)
321.8936 S:11 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,1420.0,1.00%,F,F)
331.9368 S:11 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,20040.0,1.00%,F,F)
333.9339 S:11 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,10092.0,1.00%,F,F)
327.8847 S:11 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,13500.0,1.00%,F,F)
316.9824 S:11 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)
```

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                                                                   Page 8 of 8

  Ent: 46 Name: Total Hepta-Dioxins F:4  Mass: 423.777 425.774 Mod? no   #Hom:2

  Run: 17 File: a27sep98m    S:11 Acq:28-SEP-98 02:59:53 Proc:28-SEP-98 09:40:52
  Tables: Run: 14sep-crv  Analyte: m8290-092»  Cal: m8290-091»Results: M8290-09»
  Version: V3.5 17-APR-1997 11:14:34  Sample text  1113-7 x1/2

    Amount: 0.07    of which 0.07    named and 0.01    unnamed
      Conc: 0.07    of which 0.07    named and 0.01    unnamed
    Tox #1: -             Tox #2: -        Tox #3: -

 Name                   #     RT Respnse     RA    Conc     Area  Height    S/N  Mod?

 1,2,3,4,6,7,8-HpCDD1  37:32 1.1e+05  0.93 y    0.07
                             1.1e+05                5.2e+04 2.1e+04 9.3e+00 y  n
                                                    5.6e+04 1.6e+04 9.2e+00 y  n
                    2  37:57 1.1e+04  0.90 y    0.01
                             1.1e+04                5.3e+03 2.0e+03 8.7e-01 n  n
                                                    5.9e+03 3.4e+03 1.9e+00 n  n
```

```
                    20 36:17 8.5e+03  0.64 n    0.00
                             8.5e+03                 3.3e+03 2.4e+03 8.7e-01 n  n
                                                     5.2e+03 2.2e+03 8.6e-01 n  n


                                                                        Page 7 of 8

 Ent: 45 Name: Total Hepta-Furans  F:4  Mass: 407.782 409.779 Mod? no   #Hom:4

 Run: 17 File: a27sep98m    S:11 Acq:28-SEP-98 02:59:53 Proc:28-SEP-98 09:40:52
 Tables: Run: 14sep-crv  Analyte: m8290-092»  Cal: m8290-091»Results: M8290-09»
 Version: V3.5 17-APR-1997 11:14:34  Sample text: 1113-7 x1/2

   Amount: 0.14     of which 0.11     named and 0.03     unnamed
     Conc: 0.14     of which 0.11     named and 0.03     unnamed
   Tox #1: -            Tox #2: -          Tox #3: -

Name                   #    RT Respnse    RA     Conc     Area  Height     S/N  Mod?

1,2,3,4,6,7,8-HpCDF1  36:43 1.4e+05  1.06 y    0.09
                             1.4e+05                 7.4e+04 3.1e+04 9.6e+00 y  n
                                                     7.0e+04 2.5e+04 1.6e+01 y  n
                     2  36:55 2.2e+04  0.75 n    0.02
                             2.2e+04                 9.6e+03 4.5e+03 1.4e+00 n  n
                                                     1.3e+04 2.4e+03 1.6e+00 n  n
                     3  37:03 1.7e+04  0.59 n    0.01
                             1.7e+04                 6.4e+03 2.4e+03 7.5e-01 n  n
                                                     1.1e+04 4.5e+03 2.9e+00 n  n
1,2,3,4,7,8,9-HpCDF4  37:53 2.3e+04  0.85 n    0.02
                             2.3e+04                 1.0e+04 5.2e+03 1.6e+00 n  n
                                                     1.2e+04 4.3e+03 2.8e+00 n  n
```

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                                                       Page 6 of 8

 Ent: 44 Name: Total Hexa-Dioxins  F:3  Mass: 389.816 391.813 Mod? no   #Hom:20

 Run: 17 File: a27sep98m    S:11 Acq:28-SEP-98 02:59:53 Proc:28-SEP-98 09:40:52
 Tables: Run: 14sep-crv  Analyte: m8290-092»  Cal: m8290-091»Results: M8290-09»
 Version: V3.5 17-APR-1997 11:14:34  Sample text: 1113-7 x1/2

   Amount: 0.17    of which 0.04    named and 0.12    unnamed
     Conc: 0.17    of which 0.04    named and 0.12    unnamed
   Tox #1: -           Tox #2: -          Tox #3: -

Name                 #     RT Respnse     RA     Conc     Area  Height     S/N  Mod?

                     1  34:38 5.7e+04  2.38 n    0.03
                                5.7e+04                4.0e+04 1.8e+04 6.7e+00 y  n
                                                       1.7e+04 5.8e+03 2.2e+00 n  n
                     2  34:44 3.0e+04  0.71 n    0.02
                                3.0e+04                1.3e+04 4.9e+03 1.8e+00 n  n
                                                       1.8e+04 5.5e+03 2.1e+00 n  n
                     3  34:51 8.5e+03  0.57 n    0.00
                                8.5e+03                3.1e+03 1.9e+03 6.9e-01 n  n
                                                       5.4e+03 2.1e+03 8.1e-01 n  n
                     4  34:57 1.1e+04  0.40 n    0.01
                                1.1e+04                [illegible]e+03 2.2e+03 8.0e-01 n  n
                                                       8.1e+03 3.1e+03 1.2e+00 n  n
                     5  35:01 1.5e+04  0.86 n    0.01
                                1.5e+04                6.9e+03 1.8e+03 6.5e-01 n  n
                                                       8.1e+03 3.1e+03 1.2e+00 n  n
                     6  35:05 1.4e+04  0.97 n    0.01
                                1.4e+04                6.8e+03 4.5e+03 1.6e+00 n  n
                                                       7.1e+03 3.6e+03 1.4e+00 n  n
1,2,3,6,7,8-HxCDD    7  35:09 2.7e+04  0.95 n    0.01
                                2.7e+04                1.3e+04 6.3e+03 2.3e+00 n  n
                                                       1.4e+04 [illegible] [illegible] n
                     8  35:15 6.3e+03  2.05 n    0.00
                                6.3e+03                4.3e+03 2.4e+03 8.7e-01 n  n
                                                       2.1e+03 1.8e+03 6.8e-01 n  n
1,2,3,7,8,9-HxCDD    9  35:21 6.0e+04  1.14 y    0.03
                                6.0e+04                3.2e+04 1.3e+04 4.6e+00 y  n
                                                       2.8e+04 1.0e+04 4.0e+00 y  n
                    10  35:29 1.3e+04  0.68 n    0.01
                                1.3e+04                5.1e+03 2.4e+03 8.6e-01 n  n
                                                       7.5e+03 3.3e+03 1.3e+00 n  n
                    11  35:33 5.5e+03  0.43 n    0.00
                                5.5e+03                1.6e+03 7.6e+02 2.8e-01 n  n
                                                       3.9e+03 2.0e+03 7.8e-01 n  n
                    12  35:39 7.3e+03  0.41 n    0.00
                                7.3e+03                2.1e+03 1.2e+03 4.5e-01 n  n
                                                       5.1e+03 2.6e+03 1.0e+00 n  n
                    13  35:41 8.7e+03  0.34 n    0.00
                                8.7e+03                2.2e+03 1.1e+03 3.9e-01 n  n
                                                       6.5e+03 3.1e+03 1.2e+00 n  n
                    14  35:46 8.2e+03  1.12 y    0.00
                                8.2e+03                4.3e+03 1.5e+03 5.5e-01 n  n
                                                       3.9e+03 2.8e+03 1.1e+00 n  n
                    15  35:50 1.0e+04  0.99 n    0.01
                                1.0e+04                5.1e+03 3.0e+03 1.1e+00 n  n
                                                       5.2e+03 2.5e+[illegible] [illegible] n
                    16  35:52 9.4e+03  0.82 n    0.00
                                9.4e+03                4.2e+03 3.0e+03 1.1e+00 n  n
                                                       5.2e+03 2.5e+03 9.7e-01 n  n
                    17  35:56 5.9e+03  1.02 n    0.00
                                5.9e+03                3.0e+03 1.8e+03 6.6e-01 n  n
                                                       2.9e+03 1.5e+03 6.0e-01 n  n
                    18  36:02 9.5e+03  1.42 y    0.00
                                9.5e+03                5.6e+03 3.3e+03 1.2e+00 n  n
                                                       3.9e+03 1.6e+03 6.2e-01 n  n
                    19  36:07 1.4e+04  1.54 n    0.01
                                1.4e+04                8.7e+03 2.5e+03 9.0e-01 n  n
                                                       5.6e+03 2.2e+03 8.5e-01 n  n
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                     20 36:07 2.3e+04   3.42 n     0.01
                              2.3e+04                  1.8e+04 3.4e+03 7.9e-01 n  n
                                                       5.2e+03 2.3e+03 1.0e+00 n  n
                     21 36:15 7.1e+03   1.00 n     0.00
                              7.1e+03                  3.6e+03 1.8e+03 4.2e-01 n  n
                                                       3.6e+03 2.2e+03 1.0e+00 n  n
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                                                                Page 5 of 8

 Ent: 43 Name: Total Hexa-Furans    F:3  Mass: 373.821 375.818 Mod? no   #Hom:21

 Run: 17 File: a27sep98m    S:11 Acq:28-SEP-98 02:59:53 Proc:28-SEP-98 09:40:52
 Tables: Run: 14sep-crv  Analyte: m8290-092» Cal: m8290-091»Results: M8290-09»
 Version: V3.5 17-APR-1997 11:14:34  Sample text: 1113-7 x1/2

   Amount: 0.21    of which 0.05    named and 0.15    unnamed
     Conc: 0.21    of which 0.05    named and 0.15    unnamed
   Tox #1: -           Tox #2: -          Tox #3: -

Name                     #     RT Respnse     RA     Conc     Area   Height     S/N  Mod?

                         1  34:00 5.1e+04  1.51 n    0.03
                                  5.1e+04                  3.1e+04 1.3e+04 3.0e+00 n  n
                                                           2.0e+04 9.2e+03 4.2e+00 y  n
                         2  34:08 1.4e+04  1.36 y    0.01
                                  1.4e+04                  7.8e+03 2.8e+03 6.6e-01 n  n
                                                           5.8e+03 2.0e+03 9.3e-01 n  n
                         3  34:13 6.1e+03  3.58 n    0.00
                                  6.1e+03                  4.7e+03 2.4e+03 5.6e-01 n  n
                                                           1.3e+03 9.5e+02 4.3e-01 n  n
                         4  34:16 1.1e+04  3.90 n    0.01
                                  1.1e+04                  [illegible] 3.5e+03 8.3e-01 n  n
                                                           2.2e+03 1.3e+03 5.9e-01 n  n
                         5  34:20 1.3e+04  0.64 n    0.01
                                  1.3e+04                  4.9e+03 2.7e+03 6.3e-01 n  n
                                                           7.7e+03 1.9e+03 8.8e-01 n  n
                         6  34:22 1.2e+04  0.54 n    0.01
                                  1.2e+04                  4.2e+03 2.1e+03 5.0e-01 n  n
                                                           7.7e+03 1.9e+03 8.8e-01 n  n
                         7  34:27 1.2e+04  3.63 n    0.01
                                  1.2e+04                  9.3e+03 4.0e+03 9.3e-01 n  n
                                                           2.6e+03 1.5e+03 [illegible] n
1,2,3,4,7,8-HxCDF        8  34:33 4.1e+04  1.49 n    0.02
                                  4.1e+04                  2.5e+04 8.1e+03 1.9e+00 n  n
                                                           1.7e+04 5.6e+03 2.6e+00 n  n
1,2,3,6,7,8-HxCDF        9  34:38 3.1e+04  1.32 y    0.01
                                  3.1e+04                  1.7e+04 9.2e+03 2.2e+00 n  n
                                                           1.3e+04 5.6e+03 2.6e+00 n  n
                         10 35:09 1.5e+04  1.08 y    0.01
                                  1.5e+04                  7.8e+03 3.8e+03 9.0e-01 n  n
                                                           7.2e+03 2.3e+03 1.1e+00 n  n
                         11 35:19 1.6e+04  1.09 y    0.01
                                  1.6e+04                  8.2e+03 3.2e+03 7.4e-01 n  n
                                                           7.5e+03 2.5e+03 1.1e+00 n  n
                         12 35:21 3.1e+04  3.09 n    0.02
                                  3.1e+04                  2.3e+04 6.3e+03 1.5e+00 n  n
                                                           7.5e+03 2.5e+03 1.1e+00 n  n
                         13 35:27 7.5e+03  2.09 n    0.00
                                  7.5e+03                  5.1e+03 2.9e+03 6.7e-01 n  n
                                                           2.4e+03 1.7e+03 7.7e-01 n  n
1,2,3,7,8,9-HxCDF        14 35:33 2.7e+04  2.73 n    0.02
                                  2.7e+04                  2.0e+04 8.8e+03 2.1e+00 n  n
                                                           7.3e+03 4.8e+03 2.2e+00 n  n
                         15 35:41 2.2e+04  5.48 n    0.01
                                  2.2e+04                  1.9e+04 3.9e+03 9.0e-01 n  n
                                                           3.4e+03 2.2e+03 [illegible] n  n
                         16 35:48 9.3e+03  1.48 n    0.01
                                  9.3e+03                  5.5e+03 3.5e+03 8.3e-01 n  n
                                                           3.7e+03 2.2e+03 9.9e-01 n  n
                         17 35:53 1.4e+04  2.54 n    0.01
                                  1.4e+04                  1.0e+04 2.3e+03 5.4e-01 n  n
                                                           4.0e+03 2.8e+03 1.3e+00 n  n
                         18 35:58 8.1e+03  1.47 n    0.00
                                  8.1e+03                  4.8e+03 3.1e+03 7.2e-01 n  n
                                                           3.3e+03 2.0e+03 8.9e-01 n  n
                         19 36:01 1.4e+04  2.25 n    0.01
                                  1.4e+04                  9.6e+03 6.9e+03 1.6e+00 n  n
                                                           4.3e+03 2.8e+03 1.3e+00 n  n
```

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 Ent: 42 Name: Total Penta-Dioxins F:2  Mass: 355.855 357.852 Mod? no    #Hom:11

 Run: 17 File: a27sep98m    S:11 Acq:28-SEP-98 02:59:53 Proc:28-SEP-98 09:40:52
 Tables: Run: 14sep-crv  Analyte: m8290-092»  Cal: m8290-091»Results: M8290-09»
 Version: V3.5 17-APR-1997 11:14:34  Sample text: 1113-7 x1/2

   Amount: 0.06    of which 0.01    named and 0.05    unnamed
     Conc: 0.06    of which 0.01    named and 0.05    unnamed
   Tox #1: -          Tox #2: -          Tox #3: -

Name                #     RT Respnse     RA     Conc     Area  Height     S/N  Mod?

                    1  32:22 2.8e+04  2.67 n    0.01
                             2.8e+04                 2.1e+04 1.2e+04 2.6e+00 n  n
                                                     7.7e+03 3.9e+03 6.1e+00 y  n
                    2  32:35 1.1e+04  2.73 n    0.00
                             1.1e+04                 7.8e+03 3.3e+03 7.3e-01 n  n
                                                     2.8e+03 1.1e+03 1.8e+00 n  n
                    3  32:37 5.6e+03  0.98 n    0.00
                             5.6e+03                 2.8e+03 2.1e+03 4.7e-01 n  n
                                                     2.8e+03 1.1e+03 1.8e+00 n  n
                    4  32:45 1.9e+04  3.49 n    0.01
                             1.9e+04                 1.4e+04 7.2e+03 1.6e+00 n  n
                                                     4.2e+03 1.3e+03 2.1e+00 n  n
                    5  32:48 9.7e+03  1.38 y    0.00
                             9.7e+03                 5.6e+03 3.1e+03 6.8e-01 n  n
                                                     4.1e+03 1.7e+03 2.7e+00 n  n
                    6  32:51 1.0e+04  1.51 y    0.00
                             1.0e+04                 6.1e+03 4.1e+03 8.9e-01 n  n
                                                     4.1e+03 1.7e+03 2.7e+00 n  n
1,2,3,7,8-PeCDD     7  33:02 1.8e+04  2.23 n    0.01
                             1.8e+04                 1.2e+04 7.2e+03 1.6e+00 n  n
                                                     5.6e+03 3.6e+03 5.7e+00 y  n
                    8  33:10 9.6e+03  5.29 n    0.00
                             9.6e+03                 8.1e+03 2.5e+03 5.4e-01 n  n
                                                     1.5e+03 9.6e+02 1.5e+00 n  n
                    9  33:14 6.3e+03  3.13 n    0.00
                             6.3e+03                 4.8e+03 2.2e+03 4.8e-01 n  n
                                                     1.5e+03 9.6e+02 1.5e+00 n  n
                   10  33:18 1.7e+04  2.67 n    0.01
                             1.7e+04                 1.3e+04 4.5e+03 9.8e-01 n  n
                                                     4.7e+03 2.8e+03 4.5e+00 y  n
                   11  33:26 1.1e+04  1.21 n    0.00
                             1.1e+04                 5.8e+03 3.4e+03 7.4e-01 n  n
                                                     4.8e+03 2.5e+03 4.0e+00 y  n
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OPUSquan   28-SEP-1998                Page 2
     Conc: 0.03    of which *       named and 0.03     unnamed
   Tox #1: -            Tox #2: -         Tox #3: -

Name               #     RT Respnse     RA     Conc    Area  Height    S/N  Mod?

                   1  32:54 1.4e+04  1.13 n     0.01
                            1.4e+04                 7.6e+03 3.6e+03 2.7e+00 n  n
                                                    6.7e+03 5.0e+03 1.2e+00 n  n
                   2  33:02 1.4e+04  0.58 n     0.01
                            1.4e+04                 5.1e+03 2.0e+03 1.5e+00 n  n
                                                    8.8e+03 2.4e+03 6.1e-01 n  n
                   3  33:22 1.1e+04  0.99 n     0.00
                            1.1e+04                 5.6e+03 2.9e+03 2.2e+00 n  n
                                                    5.6e+03 3.0e+03 7.5e-01 n  n
                   4  33:24 5.5e+03  0.23 n     0.00
                            5.5e+03                 1.0e+03 4.8e+02 3.6e-01 n  n
                                                    4.5e+03 2.1e+03 5.2e-01 n  n
                   5  33:29 6.8e+03  0.22 n     0.00
                            6.8e+03                 1.2e+03 6.9e+02 5.2e-01 n  n
                                                    5.6e+03 3.3e+03 8.2e-01 n  n
                   6  33:35 1.1e+04  0.12 n     0.00
                            1.1e+04                 1.1e+03 8.5e+02 6.4e-01 n  n
                                                    9.5e+03 2.5e+03 6.2e-01 n  n
```

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                                                                Page 1 of 8

 Ent: 39 Name: Total Tetra-Furans  F:1  Mass: 303.902 305.899 Mod? no   #Hom:5

 Run: 17 File: a27sep98m    S:11 Acq:28-SEP-98 02:59:53 Proc:28-SEP-98 09:40:52
 Tables: Run: 14sep-crv  Analyte: m8290-092»  Cal: m8290-091»Results: M8290-09»
 Version: V3.5 17-APR-1997 11:14:34  Sample text: 1113-7 x1/2

   Amount: 0.09   of which 0.02   named and 0.06   unnamed
     Conc: 0.09   of which 0.02   named and 0.06   unnamed
   Tox #1: -           Tox #2: -         Tox #3: -

Name                #     RT Respnse    RA     Conc    Area  Height     S/N  Mod?

                    1  23:34 2.9e+04  0.89 y    0.01
                             2.9e+04                1.3e+04 3.2e+03 1.1e+00 n  n
                                                    1.5e+04 9.1e+03 1.5e+00 n  n
                    2  27:21 1.1e+05  0.81 y    0.04
                             1.1e+05                5.1e+04 1.4e+04 4.6e+00 y  n
                                                    6.3e+04 1.9e+04 3.2e+00 y  n
2,3,7,8-TCDF        3  27:57 7.8e+04  7.27 n    0.02
                             7.8e+04                6.8e+04 1.3e+04 4.5e+00 y  n
                                                    9.4e+03 4.5e+03 7.6e-01 n  n
                    4  30:20 4.7e+04  0.53 n    0.02
                             4.7e+04                1.6e+04 4.4e+03 1.5e+00 n  n
                                                    3.1e+04 8.3e+03 1.4e+00 n  n
                    5  30:27 1.1e+04  0.61 n    0.00
                             1.1e+04                4.2e+03 1.5e+03 5.0e-01 n  n
                                                    6.8e+03 3.7e+03 6.2e-01 n  n


                                                                Page 2 of 8

 Ent: 40 Name: Total Tetra-Dioxins F:1  Mass: 319.897 321.894 Mod? no   #Hom:5

 Run: 17 File: a27sep98m    S:11 Acq:28-SEP-98 02:59:53 Proc:28-SEP-98 09:40:52
 Tables: Run: 14sep-crv  Analyte: m8290-092»  Cal: m8290-091»Results: M8290-09»
 Version: V3.5 17-APR-1997 11:14:34  Sample text: 1113-7 x1/2

   Amount: 0.06   of which 0.02   named and 0.03   unnamed
     Conc: 0.06   of which 0.02   named and 0.03   unnamed
   Tox #1: -           Tox #2: -         Tox #3: -

Name                #     RT Respnse    RA     Conc    Area  Height     S/N  Mod?

                    1  24:29 1.2e+04  3.62 n    0.00
                             1.2e+04                9.8e+03 4.2e+03 1.1e+00 n  n
                                                    2.7e+03 1.1e+03 8.1e-01 n  n
                    2  24:30 1.0e+04  2.76 n    0.00
                             1.0e+04                7.4e+03 4.0e+03 1.0e+00 n  n
                                                    2.7e+03 1.1e+03 8.1e-01 n  n
                    3  25:45 1.2e+04  1.52 n    0.00
                             1.2e+04                7.0e+03 4.2e+03 1.1e+00 n  n
                                                    4.6e+03 2.2e+03 1.5e+00 n  n
                    4  27:54 6.1e+04  14.10n    0.02
                             6.1e+04                5.7e+04 1.2e+04 3.1e+00 y  n
                                                    4.0e+03 2.7e+03 1.9e+00 n  n
2,3,7,8-TCDD        5  28:57 6.8e+04  0.74 y    0.02
                             6.8e+04                2.9e+04 8.2e+03 2.1e+00 n  n
                                                    3.9e+04 9.4e+03 6.7e+00 y  n


                                                                Page 3 of 8

 Ent: 41 Name: Total Penta-Furans  F:2  Mass: 339.860 341.857 Mod? no   #Hom:6

 Run: 17 File: a27sep98m    S:11 Acq:28-SEP-98 02:59:53 Proc:28-SEP-98 09:40:52
 Tables: Run: 14sep-crv  Analyte: m8290-092»  Cal: m8290-091»Results: M8290-09»
 Version: V3.5 17-APR-1997 11:14:34  Sample text: 1113-7 x1/2

   Amount: 0.03   of which *      named and 0.03   unnamed
```

```
OPUSquan    28-SEP-1998              Page 1

         Filename ; a27sep98m
           Sample ; 11
         Acquired ; 28-SEP-98  02:59:53
        Processed ; 28-SEP-98  09:40:52
        Sample ID ; 1113-7 x1/2
        Cal Table ; m8290-091498
    Results Table ; M8290-092798M
         Comments ;
Typ  ;                 Name;      Resp;     Ion 1;     Ion 2;   RA;?;     RT;       Conc;     DL;     S/N1;?;      S/N2;? ; mod?
Unk  ;         2,3,7,8-TCDD; 6.81e+04; 2.89e+04; 3.91e+04; 0.74;y; 28:57;      0.024;  0.0251;        2;n;         7;y ;   no
Unk  ;      1,2,3,7,8-PeCDD; 1.80e+04; 1.24e+04; 5.56e+03; 2.23;n; 33:02;      0.007;  0.0131;        2;n;         6;y ;   no
Unk  ;    1,2,3,4,7,8-HxCDD; 2.72e+04; 1.32e+04; 1.39e+04; 0.95;n; 35:09;      0.015;  0.0190;        2;n;         1;n ;   no
Unk  ;    1,2,3,6,7,8-HxCDD; 2.72e+04; 1.32e+04; 1.39e+04; 0.95;n; 35:09;      0.013;  0.0171;        2;n;         1;n ;   no
Unk  ;    1,2,3,7,8,9-HxCDD; 6.01e+04; 3.20e+04; 2.81e+04; 1.14;y; 35:21;      0.030;  0.0176;        5;y;         4;y ;   no
Unk  ;  1,2,3,4,6,7,8-HpCDD; 1.07e+05; 5.16e+04; 5.56e+04; 0.93;y; 37:32;      0.067;  0.0207;        9;y;         9;y ;   no
Unk  ;                 OCDD; 1.51e+05; 5.43e+04; 9.70e+04; 0.56;n; 40:22;      0.146;  0.1379;        2;n;        14;y ;   no

Unk  ;         2,3,7,8-TCDF; 7.78e+04; 6.84e+04; 9.41e+03; 7.27;n; 27:57;      0.025;  0.0369;        5;y;         1;n ;   no
Unk  ;      1,2,3,7,8-PeCDF;        *;        *;        *;    *;n;NotFnd;          *;  0.0132;        *;n;         *;n ;   no
Unk  ;      2,3,4,7,8-PeCDF;        *;        *;        *;    *;n;NotFnd;          *;  0.0129;        *;n;         *;n ;   no
Unk  ;    1,2,3,4,7,8-HxCDF; 4.15e+04; 2.48e+04; 1.66e+04; 1.49;n; 34:33;      0.023;  0.0242;        2;n;         3;n ;   no
Unk  ;    1,2,3,6,7,8-HxCDF; 3.07e+04; 1.74e+04; 1.32e+04; 1.32;y; 34:38;      0.015;  0.0210;        2;n;         3;n ;   no
Unk  ;    2,3,4,6,7,8-HxCDF;        *;        *;        *;    *;n;NotFnd;          *;  0.0232;        *;n;         *;n ;   no
Unk  ;    1,2,3,7,8,9-HxCDF; 2.73e+04; 2.00e+04; 7.33 -03; 2.73;n; 35:33;      0.0     0.0264;        2;n;         2;n ;   no
Unk  ;  1,2,3,4,6,7,8-HpCDF; 1.45e+05; 7.44e+04; 7.02e+04; 1.06;y; 36:43;      0.094;  0.0233;       10;y;        16;y ;   no
Unk  ;  1,2,3,4,7,8,9-HpCDF; 2.25e+04; 1.04e+04; 1.22e+04; 0.85;n; 37:53;      0.017,  0.0270;        2;n;         3;n ;   no
Unk  ;                 OCDF; 3.06e+04; 2.13e+04; 9.25e+03; 2.30;n; 40:31;      0.027,  0.0764;        3;n;         1;n ;   no

ES/RT;     13C-2,3,7,8-TCDD; 2.75e+08; 1.20e+08; 1.55e+08; 0.77;y; 28:56;     84.666;  0.1238;     1343;y;      3443;y ;   no
ES   ;  13C-1,2,3,7,8-PeCDD; 2.16e+08; 1.32e+08; 8.44e+07; 1.57;y; 33:02;    100.074;  0.0757;     6410;y;     18004;y ;   no
ES   ; 13C-1,2,3,6,7,8-HxCDD; 2.25e+08; 1.25e+08; 9.99e+07; 1.25;y; 35:09;    84.488;  0.0219;    13908;y;     10363;y ;   no
ES   ;13C-1,2,3,4,6,7,8-HpCDD; 1.76e+08; 9.04e+07; 8.52e+07; 1.06;y; 37:31;    78.354;  0.7446;      242;y;      292;y ;   no
ES   ;              13C-OCDD; 2.06e+08; 9.73e+07; 1.09e+08; 0.89;y; 40:22;   104.788;  0.0088;    12834;y;      011;y ;   no

ES/RT;     13C-2,3,7,8-TCDF; 3.14e+08; 1.39e+08; 1.76e+08; 0.79;y; 27:54;     77.927;  0.0539;     3647;y;       326;y ;   no
ES   ;  13C-1,2,3,7,8-PeCDF; 2.62e+08; 1.60e+08; 1.02e+08; 1.56;y; 32:22;     78.125;  0.0146;   629056;y;       713;y ;   no
ES   ; 13C-1,2,3,6,7,8-HxCDF; 1.88e+08; 6.48e+07; 1.23e+08; 0.53;y; 34:37;    61.555;  0.1042;     1477;y;       082;y ;   no
ES   ;13C-1,2,3,4,6,7,8-HpCDF; 1.12e+08; 3.45e+07; 7.80e+07; 0.44;y; 36:43;    55.857;  0.0421;     2134;y;       347;y ;   no

JS   ;     13C-1,2,3,4-TCDD; 3.07e+08; 1.35e+08; 1.71e+08; 0.79 y; 28:40;     68.208;       -;     1518;y;       813;y ;   no
JS   ; 13C-1,2,3,7,8,9-HxCDD; 2.53e+08; 1.41e+08; 1.12e+08; 1.26 y; 35:21;    70.998;       -;    14769;y;      .331;y ;   no

CS   ;    37Cl-2,3,7,8-TCDD; 2.55e+05; 2.55e+05;        -;    -;-; 28:57;      0.080;  0.0567;        4;y;        -; -;   no
CS   ;  13C-2,3,4,7,8-PeCDF; 3.47e+06; 2.16e+06; 1.31e+06; 1.65;y; 32:49;      1.053;  0.0149;    10042;y;       232;y ;   no
CS   ; 13C-1,2,3,4,7,8-HxCDD; 2.25e+08; 1.25e+08; 9.99e+07; 1.25;y; 35:09;   112.010;  0.0291;    13908;y;     10363;y ;   no
CS   ; 13C-1,2,3,4,7,8-HxCDF; 1.25e+06; 4.40e+05; 8.08e+05; 0.54;y; 34:33;     0.494;  0.1259,       10;y;        13;y ;   no
CS   ;13C-1,2,3,4,7,8,9-HpCDF;        *;        *;        *;    *;n;NotFnd;         *;  0.0481;        *;n;         *;n ;   no

SS   ;    37Cl-2,3,7,8-TCDD; 2.55e+05; 2.55e+05;        -;    -;-; 28:57;      0.095;  0.0671;        4;y;        -; -;   no
SS   ;  13C-2,3,4,7,8-PeCDF; 3.47e+06; 2.16e+06; 1.31e+06; 1.65;y; 32:49;      1.348;  0.0083;    10042;y;       232;y ;   no
SS   ; 13C-1,2,3,4,7,8-HxCDD; 2.25e+08; 1.25e+08; 9.99e+07; 1.25;y; 35:09;   132.415;  0.0329;    13908;y;     10363;y ;   no
SS   ; 13C-1,2,3,4,7,8-HxCDF; 1.25e+06; 4.40e+05; 8.08e+05; 0.54;y; 34:33;     0.801;  0.1990;       10;y;        13;y ;   no
SS   ;13C-1,2,3,4,7,8,9-HpCDF;        *;        *;        *;    *;n;NotFnd;         *;  0.0937;        *;n;         *;n ;   no
```

*Method 23*

**M23-RB**

**PES**

## Analytical Data Summary Sheet

| Labeled Standard | Expected Amount (ng) | Measured Amount (ng) | Percent Recovery (%) | RT (min.) | Ratio | Qualifier |
|---|---|---|---|---|---|---|
| **Extraction Standards** | | | | | | |
| $^{13}C_{12}$-2,3,7,8-TCDD | 4 | 3.39 | 84.7 | 28:56 | 0.77 | |
| $^{13}C_{12}$-1,2,3,7,8-PeCDD | 4 | 4.00 | 100.1 | 33:02 | 1.57 | |
| $^{13}C_{12}$-1,2,3,6,7,8-HxCDD | 4 | 3.38 | 84.5 | 35:09 | 1.25 | |
| $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD | 4 | 3.13 | 78.4 | 37:31 | 1.06 | |
| $^{13}C_{12}$-OCDD | 8 | 4.19 | 52.4 | 40:22 | 0.89 | |
| $^{13}C_{12}$-2,3,7,8-TCDF | 4 | 3.12 | 77.9 | 27:54 | 0.79 | |
| $^{13}C_{12}$-1,2,3,7,8-PeCDF | 4 | 3.13 | 78.1 | 32:22 | 1.56 | |
| $^{13}C_{12}$-1,2,3,6,7,8-HxCDF | 4 | 2.46 | 61.6 | 34:37 | 0.53 | |
| $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF | 4 | 2.23 | 55.9 | 36:43 | 0.44 | |
| **Sampling Standards** | | | | | | |
| $^{37}Cl_4$-2,3,7,8-TCDD | | | | | | |
| $^{13}C_{12}$-2,3,4,7,8-PeCDF | | | | | | |
| $^{13}C_{12}$-1,2,3,4,7,8-HxCDD | | | | | | |
| $^{13}C_{12}$-1,2,3,4,7,8-HxCDF | | | | | | |
| $^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF | | | | | | |
| **Injection Standards** | | | | | | |
| $^{13}C_{12}$-1,2,3,4-TCDD | | | | 28:40 | 0.79 | |
| $^{13}C_{12}$-1,2,3,7,8,9-HxCDD | | | | 35:21 | 1.26 | |

**Client Information**

| | |
|---|---|
| Project Name: | S509.000 |
| Sample ID: | M23-RB |

**Laboratory Information**

| | |
|---|---|
| Project ID: | L1113 |
| Sample ID: | 1113-7 |
| Collection Date: | 02-Sep-98 |
| Receipt Date: | 08-Sep-98 |
| Extraction Date: | 15-Sep-98 |
| Analysis Date: | 28-Sep-98 |

**Sample Information**

| | | |
|---|---|---|
| Matrix: | Air | |
| Weight / Volume: | 1 | |
| Moisture / Lipids: | 0.0 | % |

| | |
|---|---|
| Filename: | a27sep98m-11 |
| Retchk: | a27sep98m-1 |
| Begin ConCal: | a27sep98m-2 |
| End ConCal: | a27sep98m-16 |
| Initial Cal: | m8290-091498 |

Reviewed by: G.S.

Date Reviewed: 30 Sep 98

*Method 23*
**M23-RB**
PES

## Analytical Data Summary Sheet

| Analyte | Concentration (ng) | DL (ng) | EMPC (ng) | RT (min.) | Ratio | Qualifier |
|---|---|---|---|---|---|---|
| 2,3,7,8-TCDD | ND | 0.0010 | | 28:57 | 0.74 | |
| 1,2,3,7,8-PeCDD | ND | 0.0005 | | 33:02 | 2.23 | |
| 1,2,3,4,7,8-HxCDD | ND | 0.0008 | | 35:09 | 0.95 | |
| 1,2,3,6,7,8-HxCDD | ND | 0.0007 | | 35:09 | 0.95 | |
| 1,2,3,7,8,9-HxCDD | 0.0012 | 0.0007 | | 35:21 | 1.14 | |
| 1,2,3,4,6,7,8-HpCDD | 0.0027 | 0.0008 | | 37:32 | 0.93 | |
| OCDD | ND | 0.0055 | | 40:22 | 0.56 | |
| 2,3,7,8--TCDF | ND | 0.0015 | | 27:57 | 7.27 | |
| 1,2,3,7,8-PeCDF | ND | 0.0005 | | | | |
| 2,3,4,7,8-PeCDF | ND | 0.0005 | | | | |
| 1,2,3,4,7,8-HxCDF | ND | 0.0010 | | 34:33 | 1.49 | |
| 1,2,3,6,7,8-HxCDF | ND | 0.0008 | | 34:38 | 1.32 | |
| 2,3,4,6,7,8-HxCDF | ND | 0.0009 | | | | |
| 1,2,3,7,8,9-HxCDF | ND | 0.0011 | | 35:33 | 2.73 | |
| 1,2,3,4,6,7,8-HpCDF | 0.0038 | 0.0009 | | 36:43 | 1.06 | |
| 1,2,3,4,7,8,9-HpCDF | ND | 0.0011 | | 37:53 | 0.85 | |
| OCDF | ND | 0.0031 | | 40:31 | 2.3 | |
| Total TCDDs | ND | 0.0010 | | | | |
| Total PeCDDs | ND | 0.0005 | | | | |
| Total HxCDDs | 0.0012 | 0.0007 | | | | |
| Total HpCDDs | 0.0028 | 0.0008 | | | | |
| Total TCDFs | 0.0016 | 0.0015 | | | | |
| Total PeCDFs | ND | 0.0005 | | | | |
| Total HxCDFs | ND | 0.0008 | 0.0012 | | | |
| Total HpCDFs | 0.0036 | 0.0009 | | | | |
| TEQ (ND=0) | 0.0002 | | 0.0002 | | | ITEF |
| TEQ (ND=1/2) | 0.0013 | | 0.0013 | | | ITEF |

**Client Information**

| | |
|---|---|
| Project Name: | S509.000 |
| Sample ID: | M23-RB |

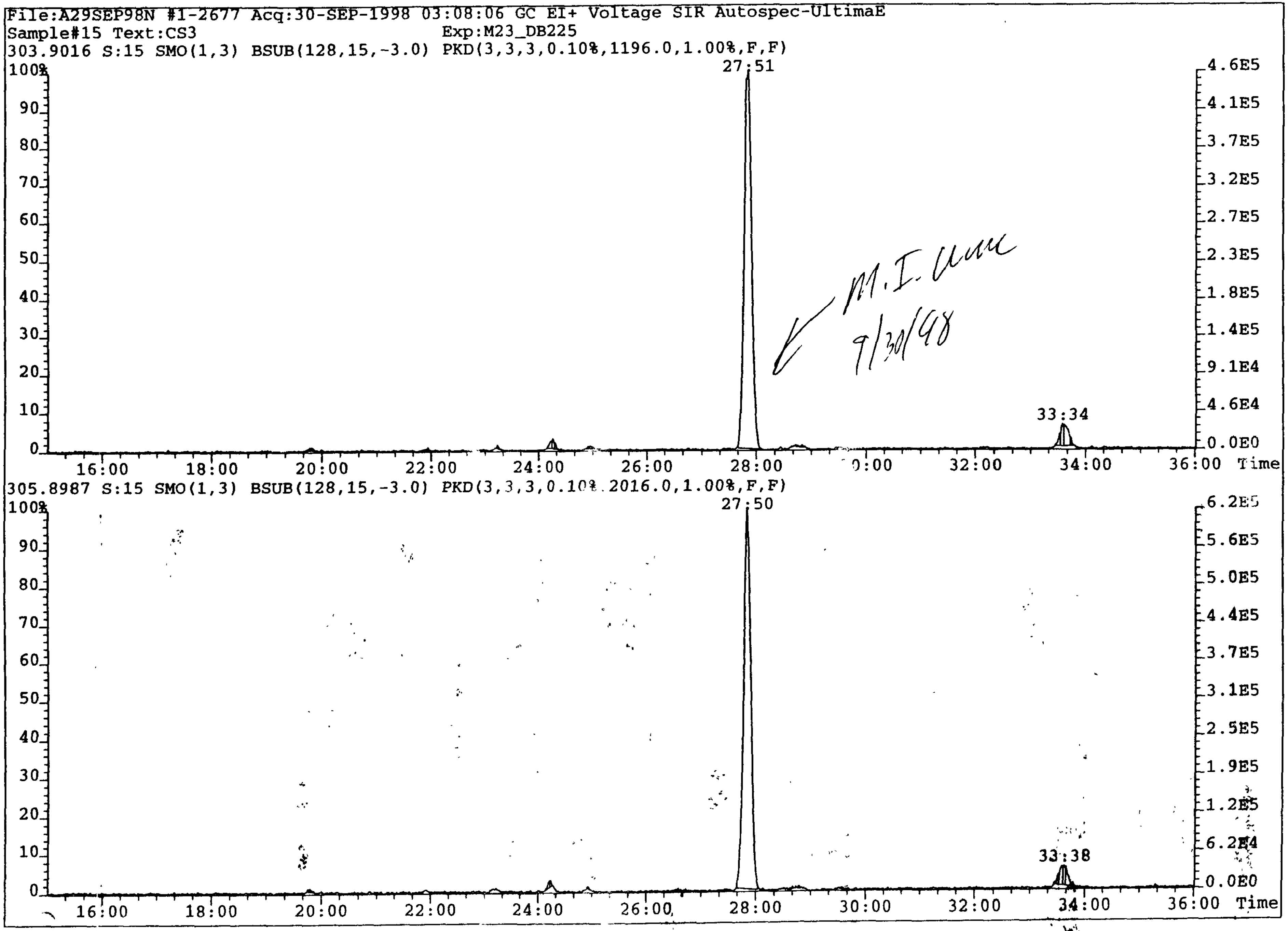
**Laboratory Information**

| | |
|---|---|
| Project ID: | L1113 |
| Sample ID: | 1113-7 |
| Collection Date: | 02-Sep-98 |
| Receipt Date: | 08-Sep-98 |
| Extraction Date: | 15-Sep-98 |
| Analysis Date: | 28-Sep-98 |

**Sample Information**

| | | |
|---|---|---|
| Matrix: | Air | |
| Weight / Volume: | 1 | |
| Moisture / Lipids: | 0.0 | % |
| Filename: | a27sep98m-11 | |
| Retchk: | a27sep98m-1 | |
| Begin ConCal: | a27sep98m-2 | |
| End ConCal: | a27sep98m-16 | |
| Initial_Cal: | m8290-091498 | |

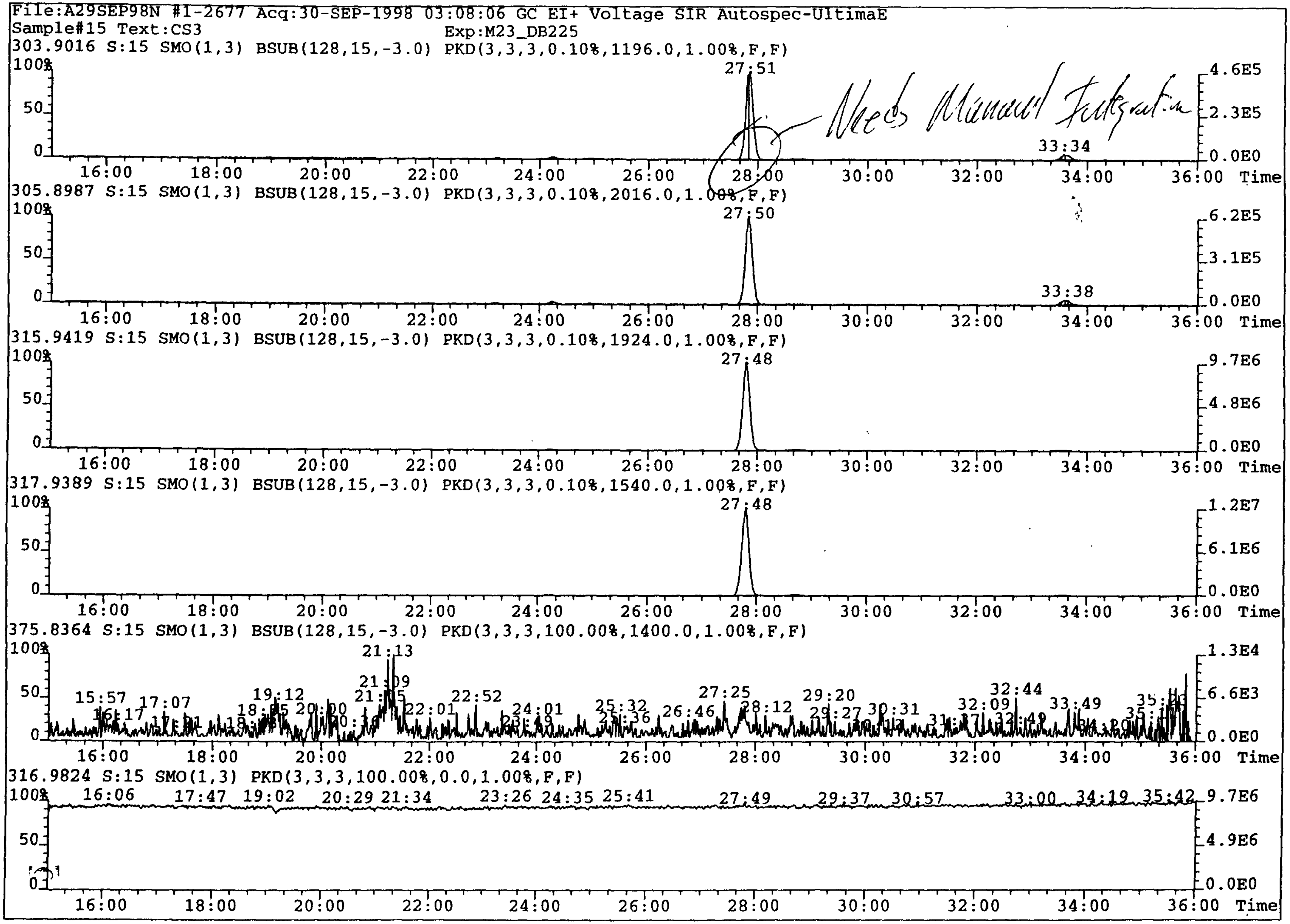
Reagent blank sample M23-RB analytical results are taken from PAL Project No. L-1113 (PAL pages 190-210). This project report details analytical results from another kiln tested during the same mobilization. One reagent blank sample was collected for all the facilities tested during the single mobilization.



```
File:A29SEP98N #1-2677 Acq:30-SEP-1998 03:08:06 GC EI+ Voltage SIR Autospec-UltimaE
Sample#15 Text:CS3                        Exp:M23_DB225
303.9016 S:15 SMO(1,3) BSUB(128,15,-3.0) PKD(3,3,3,0.10%,1196.0,1.00%,F,F)
```

M.I.
9/30/98

```
305.8987 S:15 SMO(1,3) BSUB(128,15,-3.0) PKD(3,3,3,0.10%,2016.0,1.00%,F,F)
```



```
File:A29SEP98N #1-2677 Acq:30-SEP-1998 03:08:06 GC EI+ Voltage SIR Autospec-UltimaE
Sample#15 Text:CS3                                 Exp:M23_DB225
303.9016 S:15 SMO(1,3) BSUB(128,15,-3.0) PKD(3,3,3,0.10%,1196.0,1.00%,F,F)
305.8987 S:15 SMO(1,3) BSUB(128,15,-3.0) PKD(3,3,3,0.10%,2016.0,1.00%,F,F)
315.9419 S:15 SMO(1,3) BSUB(128,15,-3.0) PKD(3,3,3,0.10%,1924.0,1.00%,F,F)
317.9389 S:15 SMO(1,3) BSUB(128,15,-3.0) PKD(3,3,3,0.10%,1540.0,1.00%,F,F)
375.8364 S:15 SMO(1,3) BSUB(128,15,-3.0) PKD(3,3,3,100.00%,1400.0,1.00%,F,F)
316.9824 S:15 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)
```

Needs Manual Integration

```
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                                                                                    Page 2 of 2

   Run #7   Filename a29sep98n        S: 15  I: 1  Acquired: 30-SEP-98 03:08:06 Processed: 30-SEP-98 08:25:30
   Run: a07feb98f  Analyte:            Cal: 07feb-m23» Results:                Quan : V3.5 17-APR-1997 11:14:34
   Sample text: CS3                         Comments:                          OPUS : V3.5X 17-APR-1997 11:31:23

        Typ                      Name          Resp       RA        RT      Conc    Dev'n               Mod?

        Unk               2,3,7,8-TCDF       7.8e+06    0.43  n   27:51     4.14    -17.2                n
        ES/RT          13C-2,3,7,8-TCDF      2.0e+08    0.79  y   27:48     48.8        -                n
        Total             Tetra Furans       1.6e+07    2.07  n   19:46     8.41    -17.2                n
        DPE                     HxCDPE             *              NotFnd       *        -                n
        LMC          QC CHK ION (Tetra)            *              NotFnd       *        -                n
```

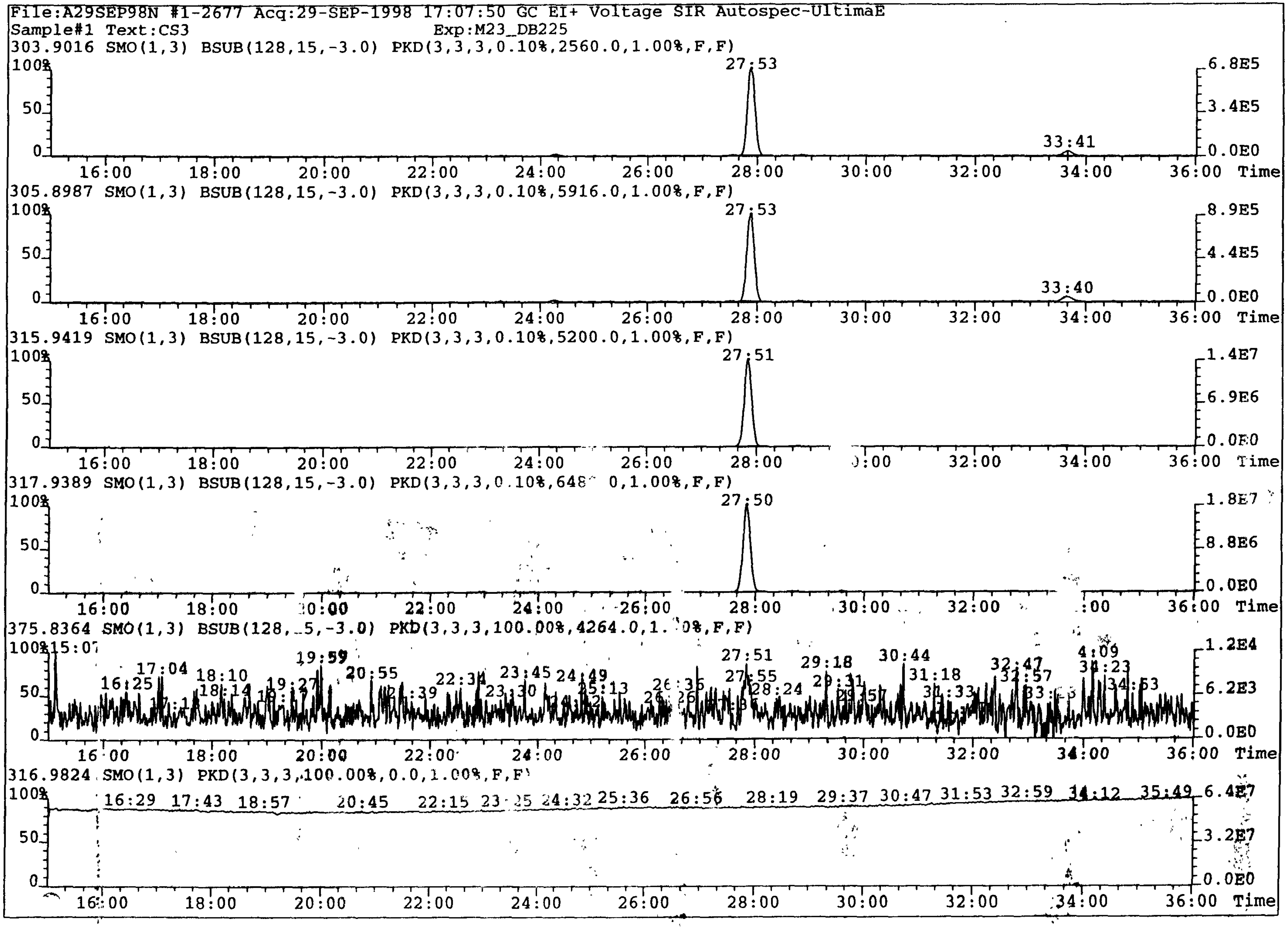
Split

```
OPUSquan    30-SEP-1998               Page 1
                                                                                          Page 2 of 3

    Run #7    Filename a29sep98n        S: 15   I: 1  Acquired: 30-SEP-98 03:08:06 Processed: 30-SEP-98 08:25:30
    Run: a07feb98f  Analyte:             Cal: 07feb-m23» Results:              Quan : V3.5 17-APR-1997 11:14:34
    Sample text: CS3                          Comments:                        OPUS : V3.5X 17-APR-1997 11:31:23

         Typ                        Name            Resp       RA       RT        Conc     Dev'n             Mod?

         Unk                2,3,7,8-TCDF         9.7e+06     0.78  y   27:51      5.15       2.9              y
         ES/RT           13C-2,3,7,8-TCDF         2.0e+08     0.79  y   27:48      48.8        -               n
         Total               Tetra Furans         1.0e+07     2.07  n   19:46      5.51       2.9              y
         DPE                       HxCDPE               *              NotFnd         *         -               n
         LMC          QC CHK ION (Tetra)               *              NotFnd         *         -               n
```



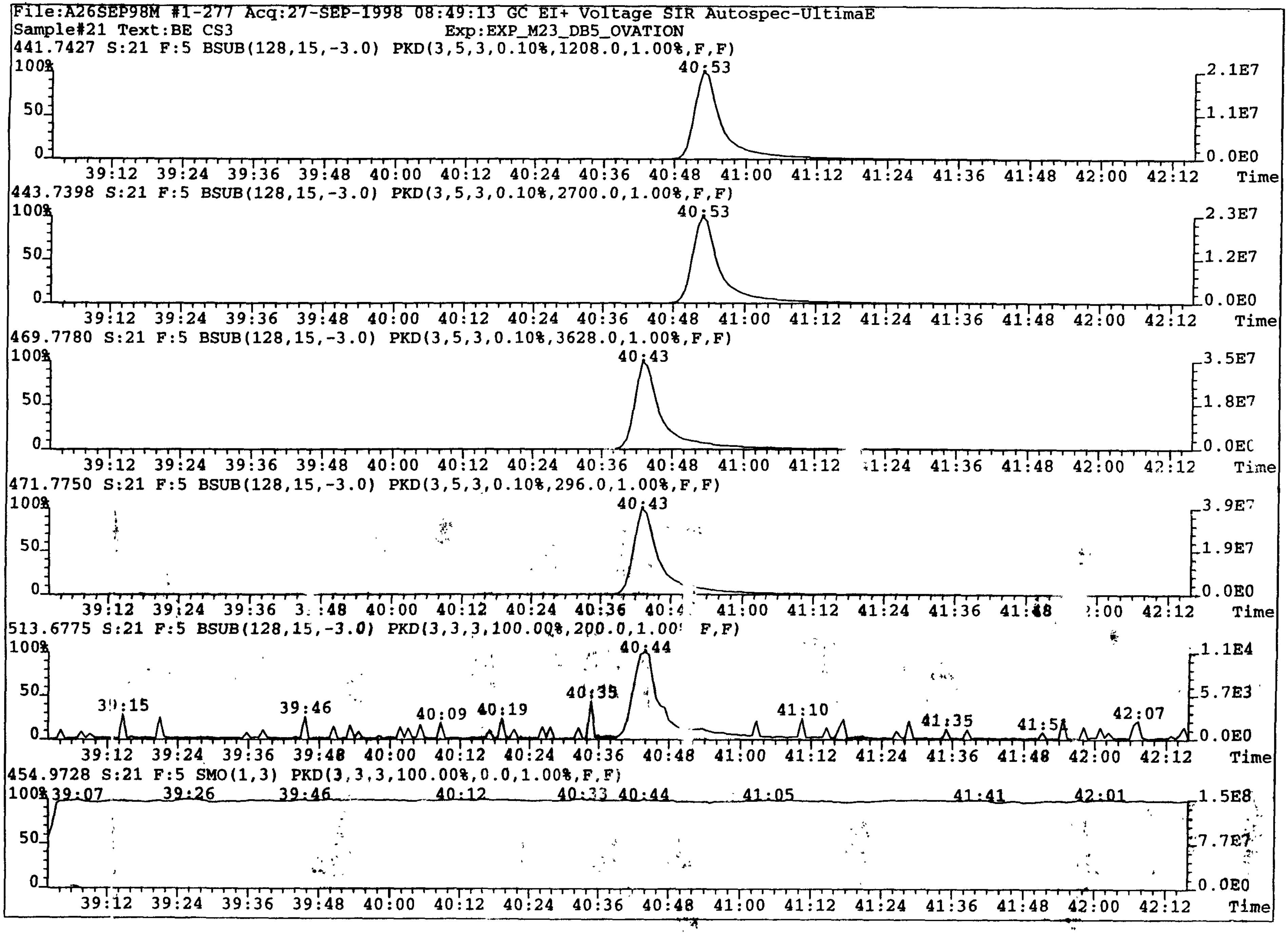
```
File:A29SEP98N #1-2677 Acq:29-SEP-1998 17:07:50 GC EI+ Voltage SIR Autospec-UltimaE
Sample#1 Text:CS3                        Exp:M23_DB225
303.9016 SMO(1,3) BSUB(128,15,-3.0) PKD(3,3,3,0.10%,2560.0,1.00%,F,F)
305.8987 SMO(1,3) BSUB(128,15,-3.0) PKD(3,3,3,0.10%,5916.0,1.00%,F,F)
315.9419 SMO(1,3) BSUB(128,15,-3.0) PKD(3,3,3,0.10%,5200.0,1.00%,F,F)
317.9389 SMO(1,3) BSUB(128,15,-3.0) PKD(3,3,3,0.10%,648  0,1.00%,F,F)
375.8364 SMO(1,3) BSUB(128,15,-3.0) PKD(3,3,3,100.00%,4264.0,1.00%,F,F)
316.9824 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)
```

```
                                                                                Page 1 of 2

  Run #6   Filename a29sep98n        S: 1    I: 1  Acquired: 29-SEP-98 17:07:50 Processed: 30-SEP-98 08:24:51
  Run: a07feb98f  Analyte:            Cal: 07feb-m23» Results:               Quan : V3.5 17-APR-1997 11:14:34
  Sample text: CS3                        Comments:                           OPUS : V3.5X 17-APR-1997 11:31:23

       Typ                        Name           Resp       RA       RT        Conc     Dev'n            Mod?

       Unk               2,3,7,8-TCDF         1.4e+07     0.78  y   27:53      5.26      5.2              n
       ES/RT          13C-2,3,7,8-TCDF         2.9e+08     0.79  y   27:51      70.6       -               n
       Total              Tetra Furans         1.5e+07     0.74  y   24:13      5.39      5.2              n
       DPE                      HxCDPE               *              NotFnd         *       -               n
       LMC          QC CHK ION (Tetra)               *              NotFnd         *       -               n
```



File:A26SEP98M #1-277 Acq:27-SEP-1998 08:49:13 GC EI+ Voltage SIR Autospec-UltimaE

Sample#21 Text:BE CS3 Exp:EXP_M23_DB5_OVATION

441.7427 S:21 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,1208.0,1.00%,F,F)

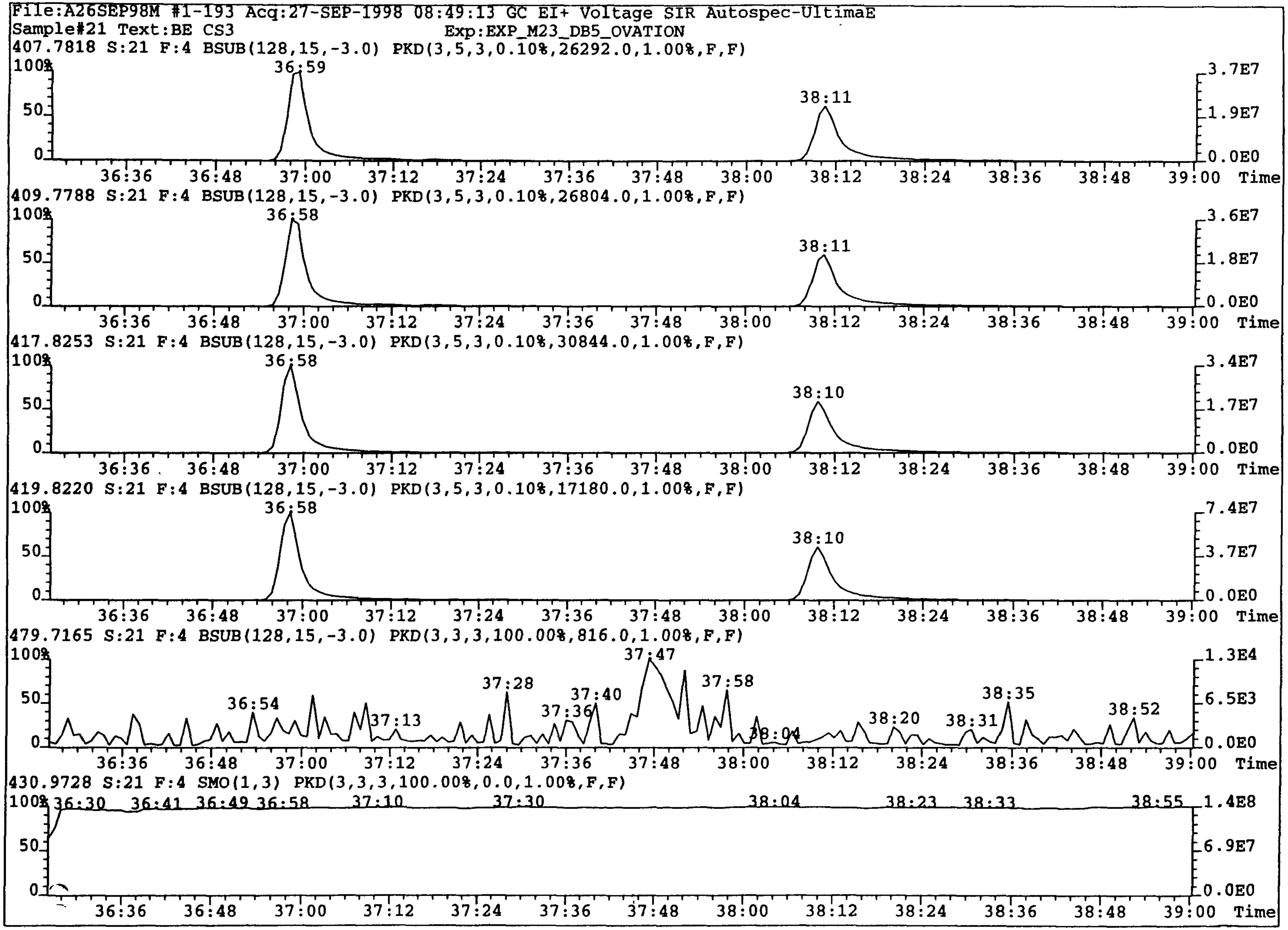
443.7398 S:21 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,2700.0,1.00%,F,F)

469.7780 S:21 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,3628.0,1.00%,F,F)

471.7750 S:21 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,296.0,1.00%,F,F)

513.6775 S:21 F:5 BSUB(128,15,-3.0) PKD(3,3,3,100.00%,200.0,1.00% F,F)

454.9728 S:21 F:5 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)



File:A26SEP98M #1-193 Acq:27-SEP-1998 08:49:13 GC EI+ Voltage SIR Autospec-UltimaE
Sample#21 Text:BE CS3 Exp:EXP_M23_DB5_OVATION

407.7818 S:21 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,26292.0,1.00%,F,F)

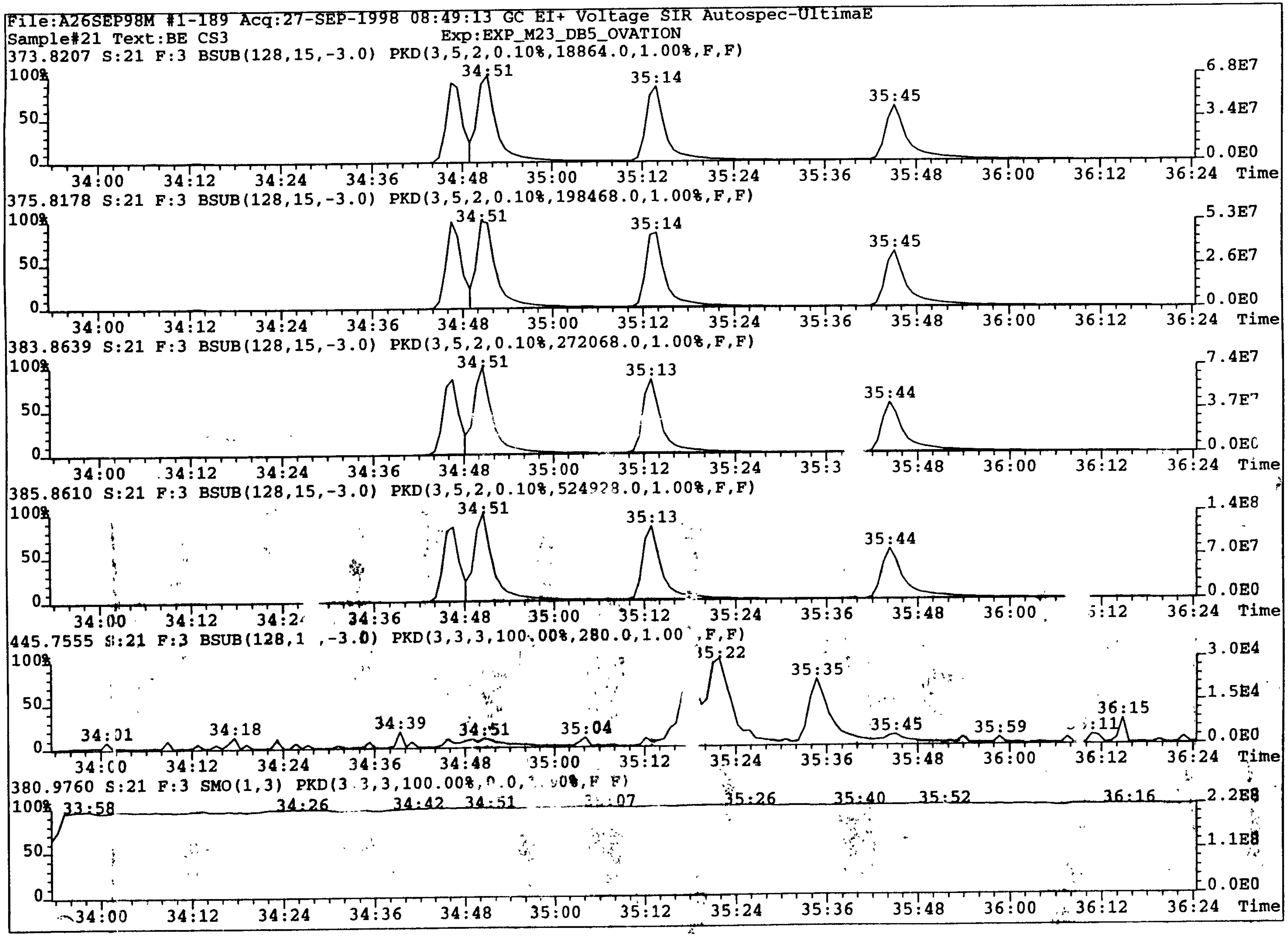
409.7788 S:21 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,26804.0,1.00%,F,F)

417.8253 S:21 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,30844.0,1.00%,F,F)

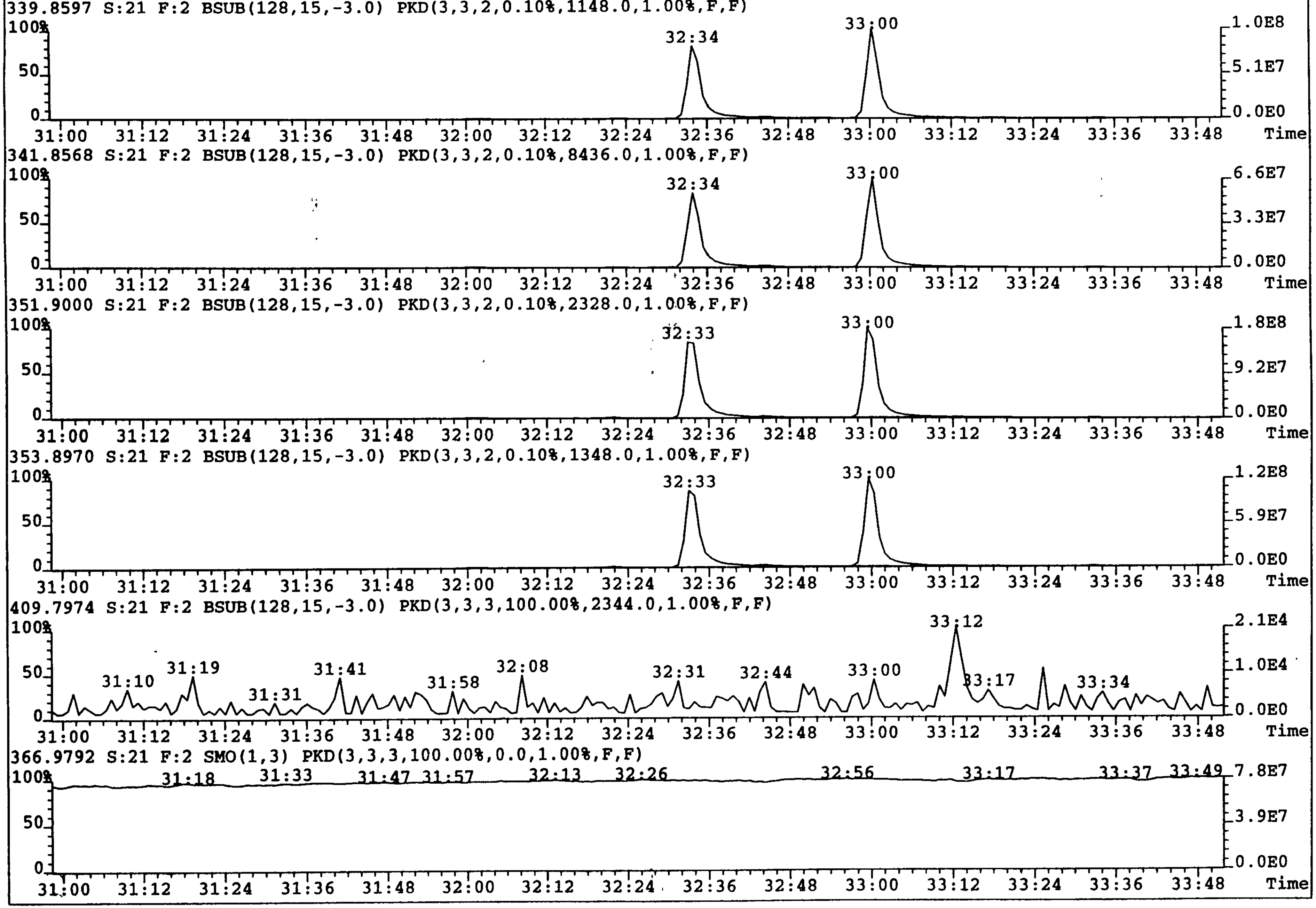
419.8220 S:21 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,17180.0,1.00%,F,F)

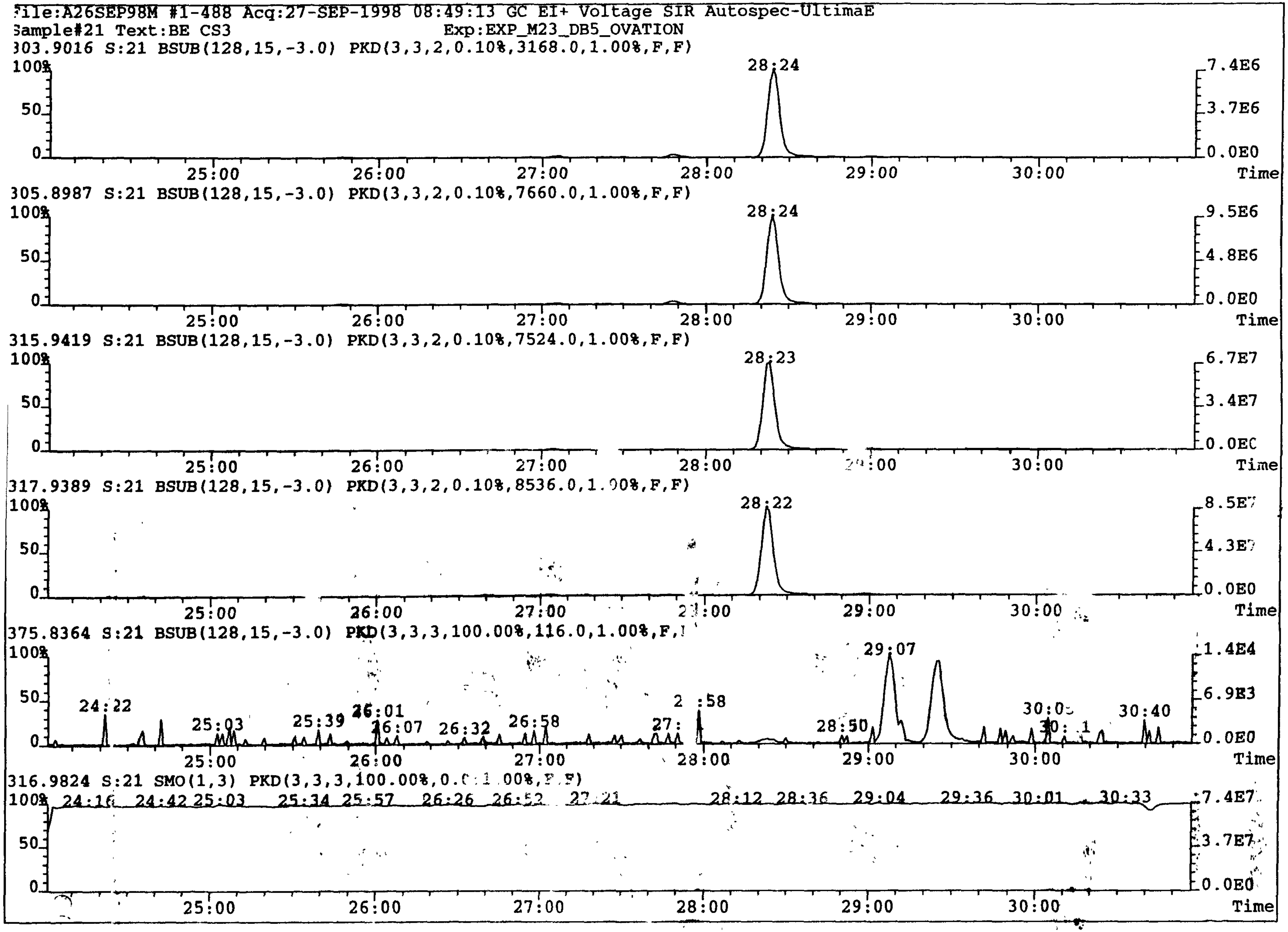
479.7165 S:21 F:4 BSUB(128,15,-3.0) PKD(3,3,3,100.00%,816.0,1.00%,F,F)

430.9728 S:21 F:4 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)


File:A26SEP98M #1-189 Acq:27-SEP-1998 08:49:13 GC EI+ Voltage SIR Autospec-UltimaE
Sample#21 Text:BE CS3
Exp:EXP_M23_DB5_OVATION
373.8207 S:21 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,18864.0,1.00%,F,F)
100%
50
0
34:51
35:14
35:45
6.8E7
3.4E7
0.0E0
34:00 34:12 34:24 34:36 34:48 35:00 35:12 35:24 35:36 35:48 36:00 36:12 36:24 Time
375.8178 S:21 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,198468.0,1.00%,F,F)
34:51
35:14
35:45
5.3E7
2.6E7
0.0E0
383.8639 S:21 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,272068.0,1.00%,F,F)
34:51
35:13
35:44
7.4E7
3.7E7
0.0E0
385.8610 S:21 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,524928.0,1.00%,F,F)
34:51
35:13
35:44
1.4E8
7.0E7
0.0E0
445.7555 S:21 F:3 BSUB(128,1 ,-3.0) PKD(3,3,3,100.00%,280.0,1.00 ,F,F)
34:01
34:18
34:39
34:51
35:04
35:22
35:35
35:45
35:59
36:11
36:15
3.0E4
1.5E4
0.0E0
380.9760 S:21 F:3 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)
33:58
34:26
34:42
34:51
35:07
35:26
35:40
35:52
36:16
2.2E8
1.1E8
0.0E0

File:A26SEP98M #1-217 Acq:27-SEP-1998 08:49:13 GC EI+ Voltage SIR Autospec-UltimaE
Sample#21 Text:BE CS3 Exp:EXP_M23_DB5_OVATION





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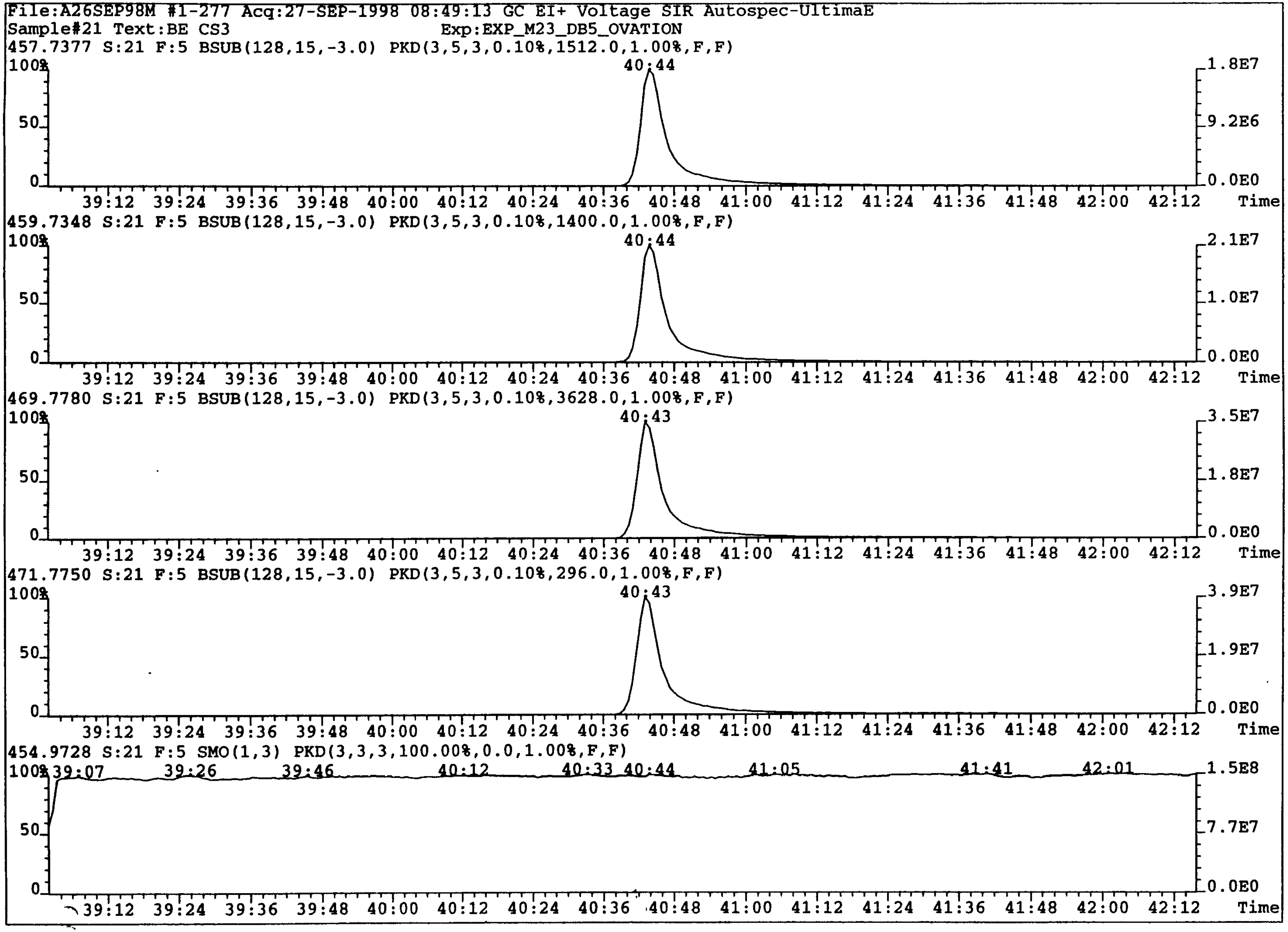
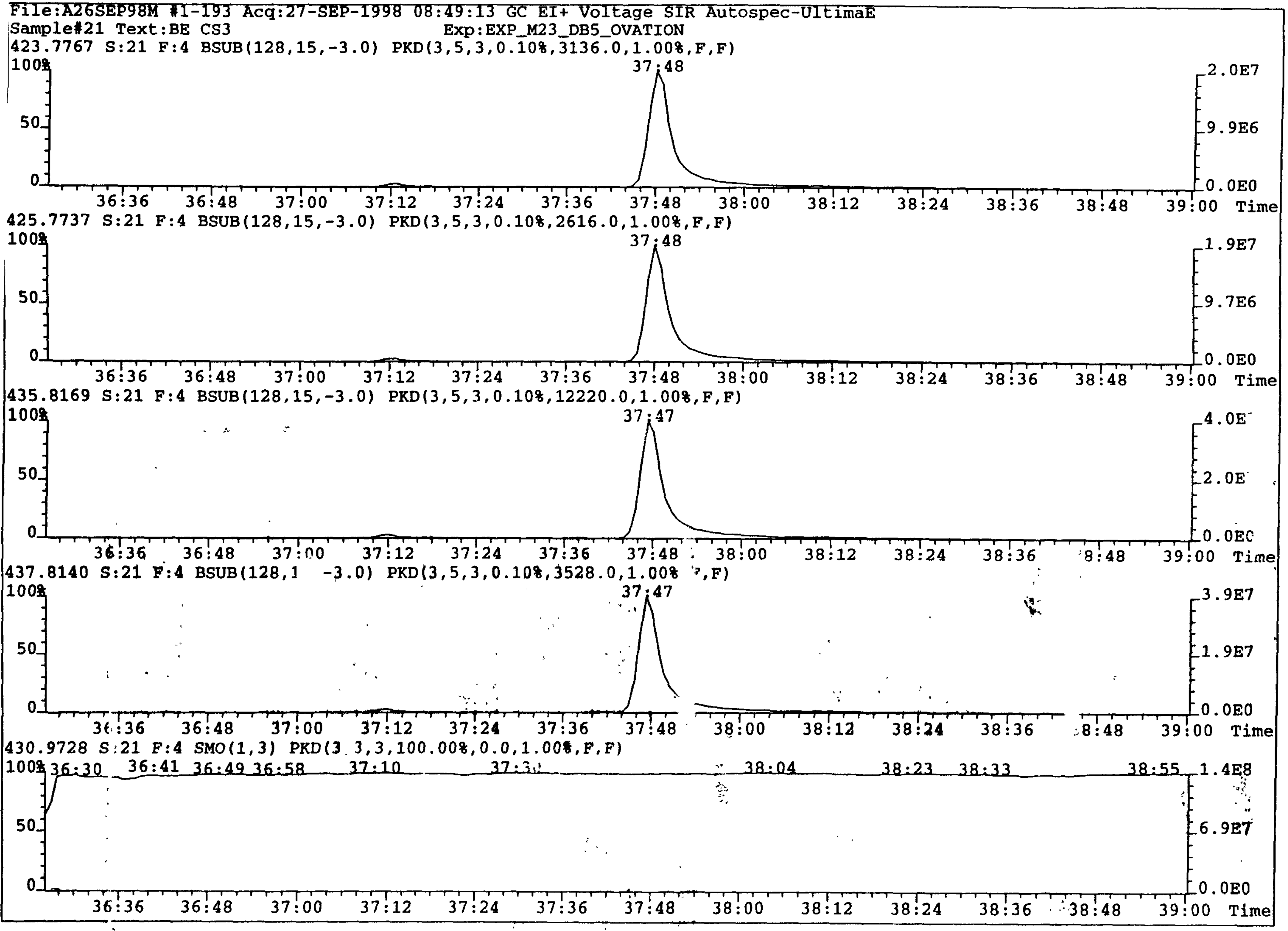
File:A26SEP98M #1-488 Acq:27-SEP-1998 08:49:13 GC EI+ Voltage SIR Autospec-UltimaE
Sample#21 Text:BE CS3                            Exp:EXP_M23_DB5_OVATION
303.9016 S:21 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,3168.0,1.00%,F,F)
305.8987 S:21 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,7660.0,1.00%,F,F)
315.9419 S:21 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,7524.0,1.00%,F,F)
317.9389 S:21 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,8536.0,1.00%,F,F)
375.8364 S:21 BSUB(128,15,-3.0) PKD(3,3,3,100.00%,116.0,1.00%,F,
316.9824 S:21 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)
```


File:A26SEP98M #1-277 Acq:27-SEP-1998 08:49:13 GC EI+ Voltage SIR Autospec-UltimaE
Sample#21 Text:BE CS3
Exp:EXP_M23_DB5_OVATION
457.7377 S:21 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,1512.0,1.00%,F,F)
40:44
100%
50
0
1.8E7
9.2E6
0.0E0
459.7348 S:21 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,1400.0,1.00%,F,F)
40:44
2.1E7
1.0E7
0.0E0
469.7780 S:21 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,3628.0,1.00%,F,F)
40:43
3.5E7
1.8E7
0.0E0
471.7750 S:21 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,296.0,1.00%,F,F)
40:43
3.9E7
1.9E7
0.0E0
454.9728 S:21 F:5 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)
39:07
39:26
39:46
40:12
40:33
40:44
41:05
41:41
42:01
1.5E8
7.7E7
0.0E0
39:12 39:24 39:36 39:48 40:00 40:12 40:24 40:36 40:48 41:00 41:12 41:24 41:36 41:48 42:00 42:12
Time



File:A26SEP98M #1-193 Acq:27-SEP-1998 08:49:13 GC EI+ Voltage SIR Autospec-UltimaE
Sample#21 Text:BE CS3 Exp:EXP_M23_DB5_OVATION

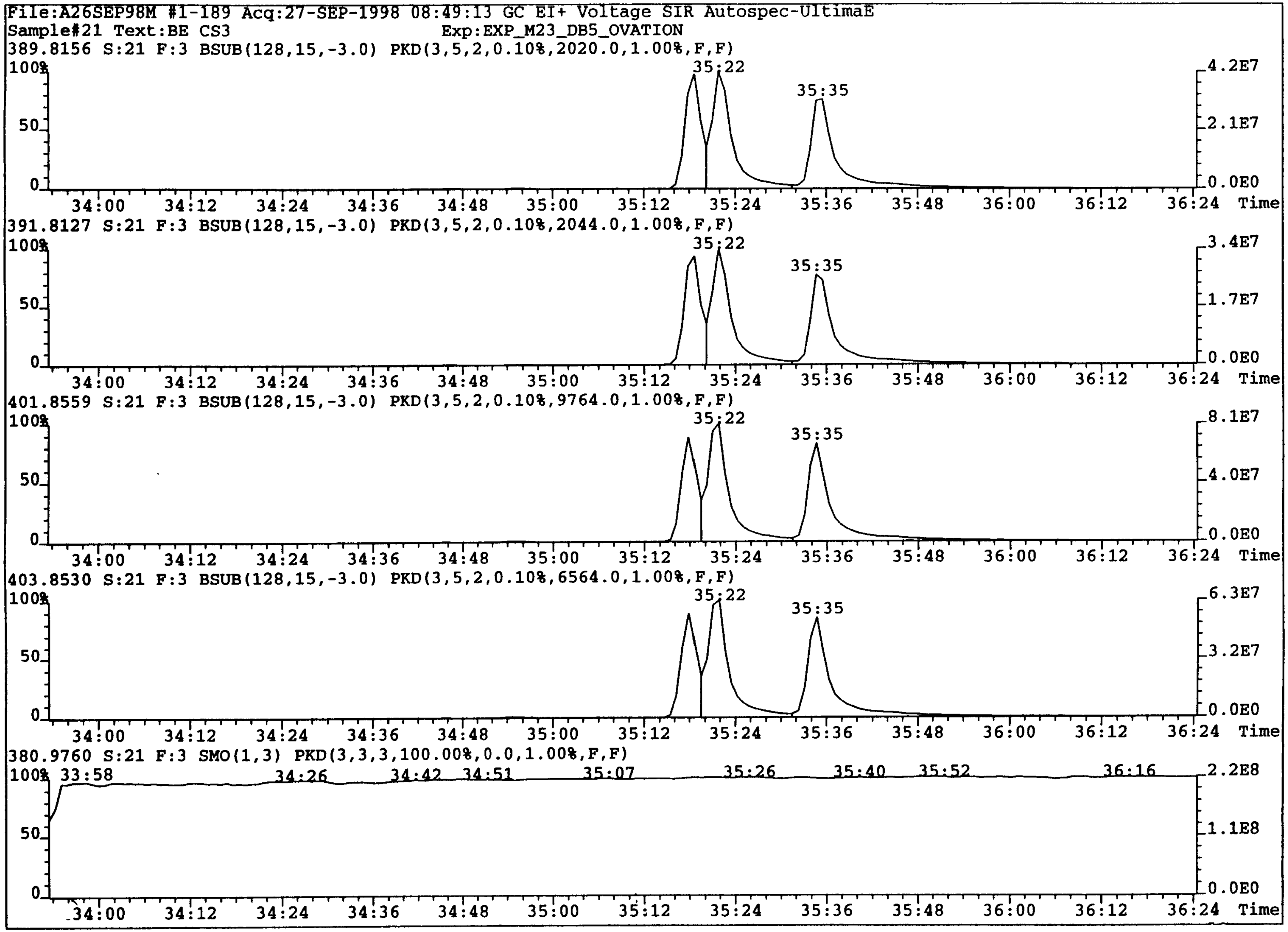
423.7767 S:21 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,3136.0,1.00%,F,F)

425.7737 S:21 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,2616.0,1.00%,F,F)

435.8169 S:21 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,12220.0,1.00%,F,F)

437.8140 S:21 F:4 BSUB(128,1 -3.0) PKD(3,5,3,0.10%,3528.0,1.00% ,F)

430.9728 S:21 F:4 SMO(1,3) PKD(3 3,3,100.00%,0.0,1.00%,F,F)



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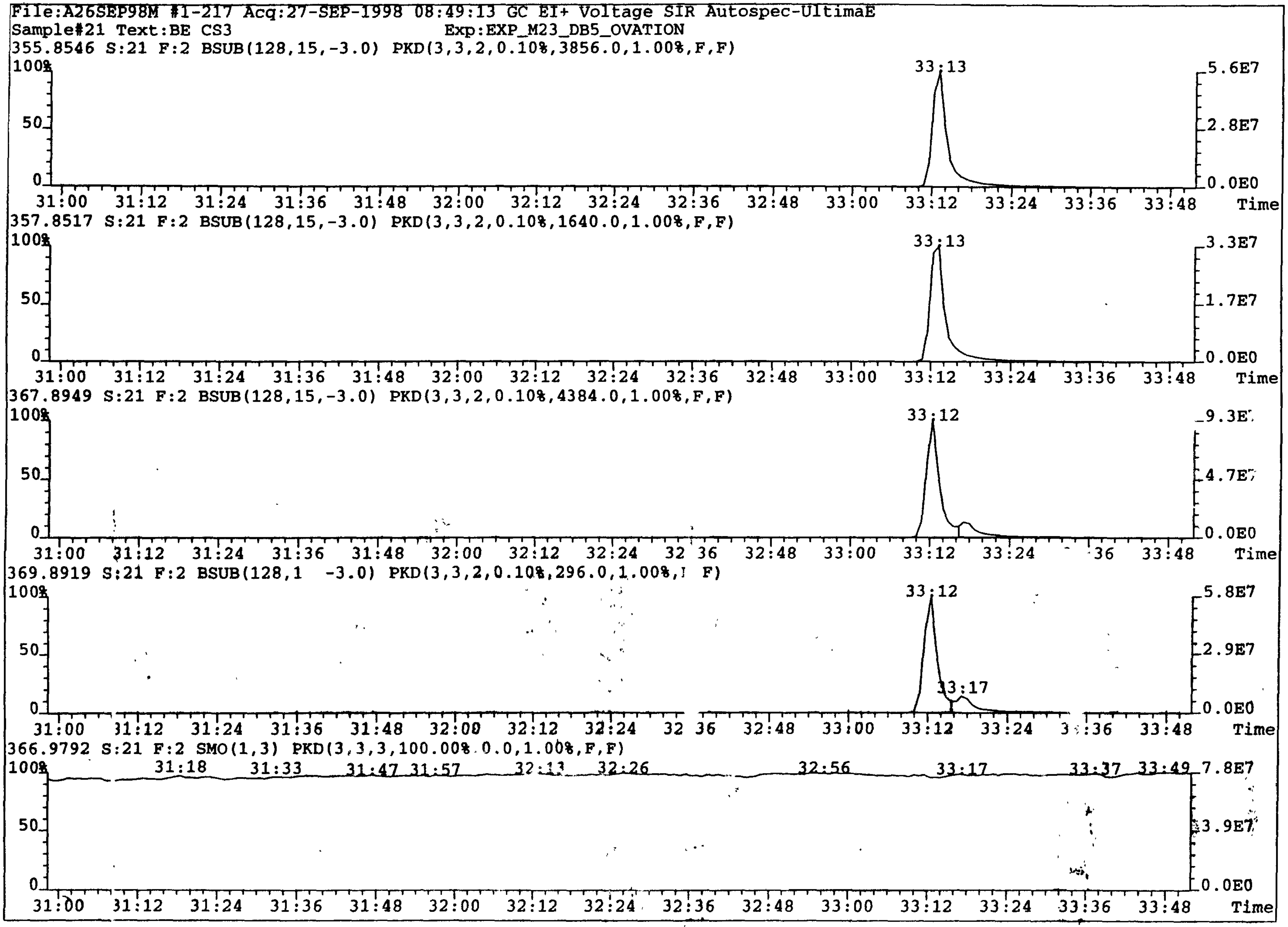
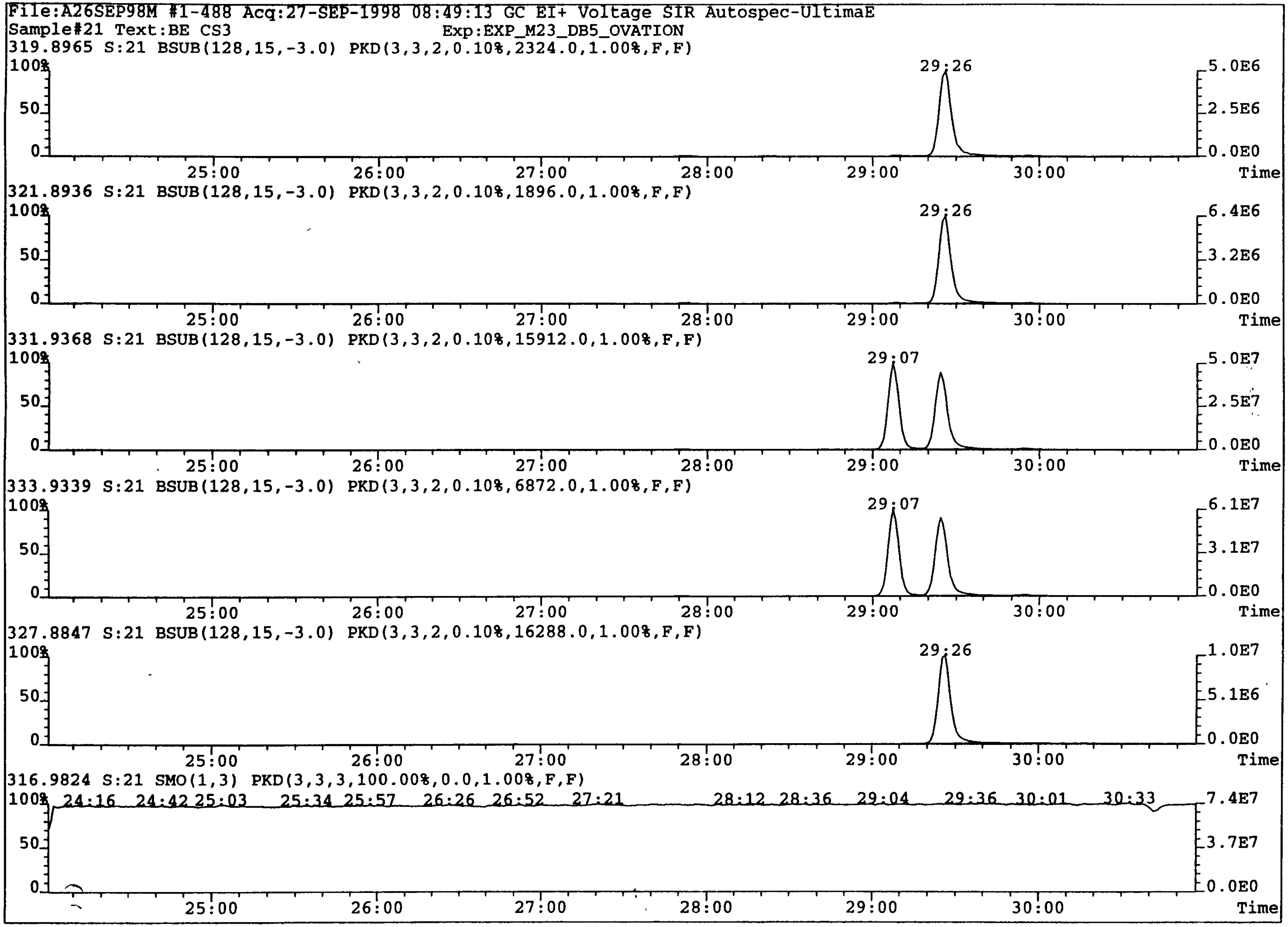
File:A26SEP98M #1-189 Acq:27-SEP-1998 08:49:13 GC EI+ Voltage SIR Autospec-UltimaE
Sample#21 Text:BE CS3                    Exp:EXP_M23_DB5_OVATION
389.8156 S:21 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,2020.0,1.00%,F,F)
391.8127 S:21 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,2044.0,1.00%,F,F)
401.8559 S:21 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,9764.0,1.00%,F,F)
403.8530 S:21 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,6564.0,1.00%,F,F)
380.9760 S:21 F:3 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)
```



```
File:A26SEP98M #1-217 Acq:27-SEP-1998 08:49:13 GC EI+ Voltage SIR Autospec-UltimaE
Sample#21 Text:BE CS3                    Exp:EXP_M23_DB5_OVATION
355.8546 S:21 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,3856.0,1.00%,F,F)
357.8517 S:21 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,1640.0,1.00%,F,F)
367.8949 S:21 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,4384.0,1.00%,F,F)
369.8919 S:21 F:2 BSUB(128,1  -3.0) PKD(3,3,2,0.10%,296.0,1.00%,I F)
366.9792 S:21 F:2 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)
```



File:A26SEP98M #1-488 Acq:27-SEP-1998 08:49:13 GC EI+ Voltage SIR Autospec-UltimaE
Sample#21 Text:BE CS3 Exp:EXP_M23_DB5_OVATION

319.8965 S:21 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,2324.0,1.00%,F,F)

321.8936 S:21 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,1896.0,1.00%,F,F)

331.9368 S:21 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,15912.0,1.00%,F,F)

333.9339 S:21 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,6872.0,1.00%,F,F)

327.8847 S:21 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,16288.0,1.00%,F,F)

316.9824 S:21 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)

```
        DPE                    OCDPE            *            NotFnd        *       -              n
        DPE                    NCDPE            *            NotFnd        *       -              n
        DPE                    DCDPE            *            NotFnd        *       -              n

        LMC         QC CHK ION (Tetra)          *            NotFnd        *       -              n
        LMC         QC CHK ION (Penta)          *            NotFnd        *       -              n
        LMC          QC CHK ION (Hexa)          *            NotFnd        *       -              n
        LMC         QC CHK ION (Hepta)          *            NotFnd        *       -              n
        LMC          QC CHK ION (Octa)          *            NotFnd        *       -              n
```

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OPUSquan    28-SEP-1998              Page 1

                                                                          Page 1 of 1

  Run #6   Filename a26sep98m        S: 21  I: 1  Acquired: 27-SEP-98 08:49:13 Processed: 28-SEP-98 11:52:19
  Run: 26sep-crv  Analyte: m8290-092» Cal: m8290-092» Results:             Quan : V3.6 31-JUL-1998 10:51:59
  Sample text: BE CS3                       Comments:                       OPUS : V3.6X 31-JUL-1998 11:15:12

     Typ                     Name           Resp      RA        RT      Conc    Dev'n                 Mod?

     Unk             2,3,7,8-TCDD        5.8e+07   0.80  y   29:26      10.4      3.8                 n
     Unk           1,2,3,7,8-PeCDD       2.3e+08   1.59  y   33:13      48.7     -2.7                 n
     Unk         1,2,3,4,7,8-HxCDD       1.7e+08   1.25  y   35:19      58.1     16.3                 n
     Unk         1,2,3,6,7,8-HxCDD       2.3e+08   1.29  y   35:22      47.9     -4.2                 n
     Unk         1,2,3,7,8,9-HxCDD       2.1e+08   1.26  y   35:35      55.7     11.5                 n
     Unk       1,2,3,4,6,7,8-HpCDD       1.5e+08   1.04  y   37:48      50.8      1.7                 n
     Unk                      OCDD       2.1e+08   0.90  y   40:44      98.7     -1.3                 n

     Unk             2,3,7,8-TCDF        8.3e+07   0.78  y   28:24      10.0      0.4                 n
     Unk           1,2,3,7,8-PeCDF       3.3e+08   1.55  y   32:34      49.8     -0.5                 n
     Unk           2,3,4,7,8-PeCDF       3.7e+08   1.56  y   33:00      48.6     -2.9                 n
     Unk         1,2,3,4,7,8-HxCDF       2.7e+08   1.24  y   34:47      52.0      3.9                 n
     Unk         1,2,3,6,7,8-HxCDF       3.7e+08   1.30  y   34:51      41.0    -18.0                 n
     Unk         2,3,4,6,7,8-HxCDF       3.0e+08   1.34  y   35:14      44.0    -11.9                 n
     Unk         1,2,3,7,8,9-HxCDF       2.5e+08   1.31  y   35:45      53.0      6.1                 n
     Unk       1,2,3,4,6,7,8-HpCDF       2.4e+08   1.03  y   36:59      51.5      3.1                 n
     Unk       1,2,3,4,7,8,9-HpCDF       1.9e+08   1.03  y   38:11      64.0     28.0 — ①            n
     Unk                      OCDF       2.4e+08   0.90  y   40:53      87.1    -12.9                 n

     ES/RT       13C-2,3,7,8-TCDD        5.0e+08   0.80  y   29:24       103      3.4                 n
     ES        13C-1,2,3,7,8-PeCDD       3.4e+08   1.63  y   33:12       120     19.6                 n
     ES      13C-1,2,3,6,7,8-HxCDD       4.7e+08   1.28  y   35:22      93.8     -6.2                 n
     ES    13C-1,2,3,4,6,7,8-HpCDD       3.1e+08   1.07  y   37:47       142     41.9  ???            n
     ES                   13C-OCDD       3.9e+08   0.89  y   40:43       408    104.2                 n

     ES/RT       13C-2,3,7,8-TCDF        7.5e+08   0.79  y   28:23       106      5.7                 n
     ES        13C-1,2,3,7,8-PeCDF       6.8e+08   1.58  y   32:33       113     12.6                 n
     ES      13C-1,2,3,6,7,8-HxCDF       4.2e+08   0.53  y   34:47      98.1     -1.9                 n
     ES    13C-1,2,3,4,6,7,8-HpCDF       3.5e+08   0.46  y   36:58       110     10.2                 n

     JS          13C-1,2,3,4-TCDD        4.9e+08   0.81  y   29:07       213        -                 n
     JS      13C-1,2,3,7,8,9-HxCDD       4.2e+08   1.26  y   35:35       305        -                 n

     CS          37Cl-2,3,7,8-TCDD       5.2e+07   1.59  y   29:26      11.1     10.5                 n
     CS        13C-2,3,4,7,8-PeCDF       7.1e+08   1.27  y   32:60       108      7.9                 n
     CS      13C-1,2,3,4,7,8-HxCDD       2.8e+08   0.53  y   35:18       102      1.7                 n
     CS      13C-1,2,3,4,7,8-HxCDF       6.2e+08   0.45  y   34:51      79.4    -20.6                 n
     CS    13C-1,2,3,4,7,8,9-HpCDF       2.7e+08             38:10       137     37.3                 n
                                                   1.59  y
     SS          37Cl-2,3,7,8-TCDD       5.2e+07   1.27  y   29:26      10.7      6.9                 n
     SS        13C-2,3,4,7,8-PeCDF       7.1e+08   0.53  y   32:60      95.8     -4.2                 n
     SS      13C-1,2,3,4,7,8-HxCDD       2.8e+08   0.45  y   35:18       109      8.8                 n
     SS      13C-1,2,3,4,7,8-HxCDF       6.2e+08             34:51      80.4    -19.6                 n
     SS    13C-1,2,3,4,7,8,9-HpCDF       2.7e+08             38:10       124     24.3                 n

     DPE                    HxCDPE             *           NotFnd          *        -                 n
     DPE                    HpCDPE             *           NotFnd          *        -                 n
```

Bad when compared to a bad I-cal. See note in margin dated 30Sep98

29Sep98

no explanation for this Δ. However, the same set of samples run (GC/MS) against this Ical and run (GC/MS) earlier using another Ical show clearly that the OCDD analyte is NOT affected by this low 13C-OCDD (relative)

... 30

=1.3527

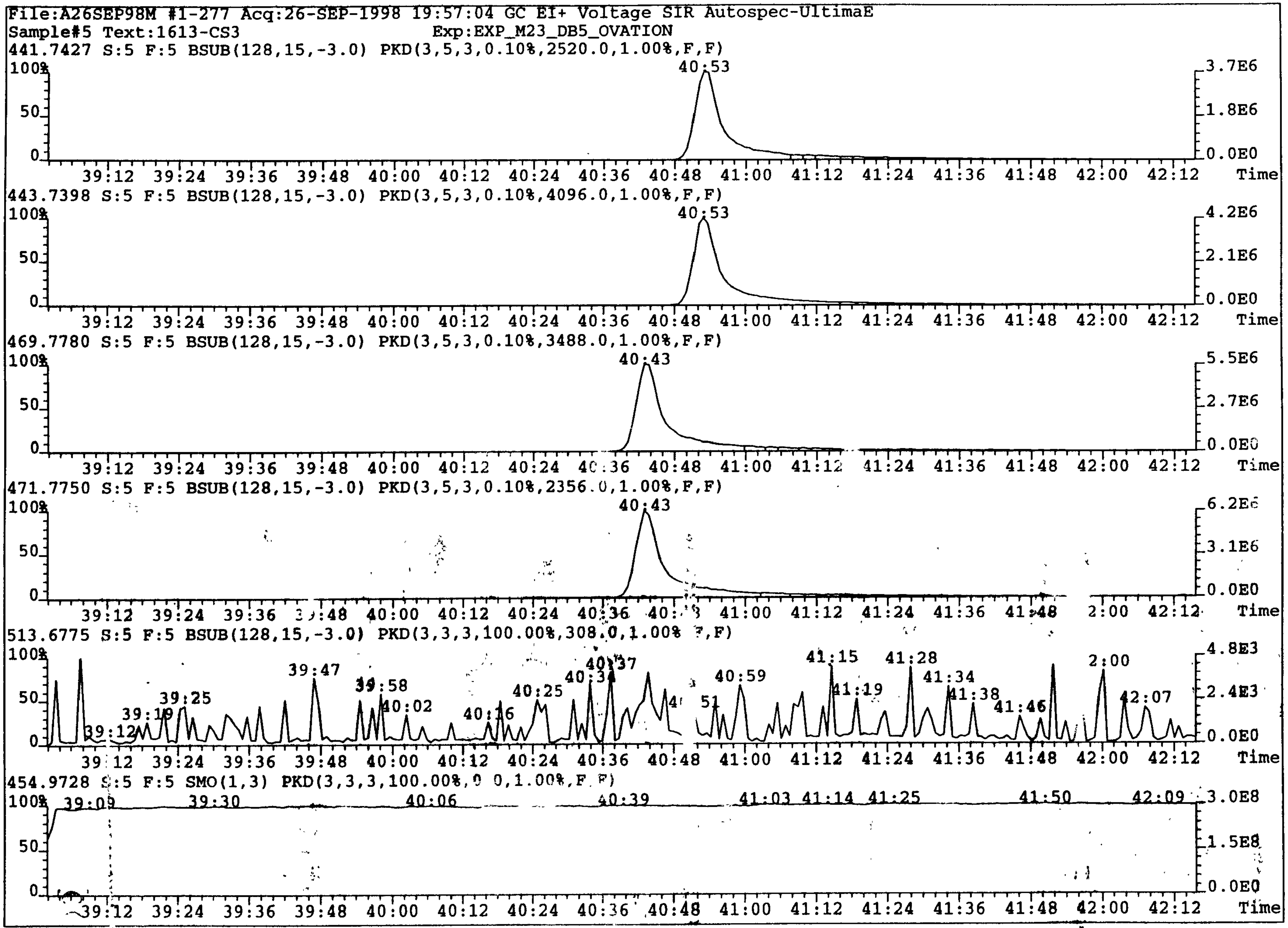
3.9 10^8 ~~a 100~~

~~200~~ & 4.2 10^8

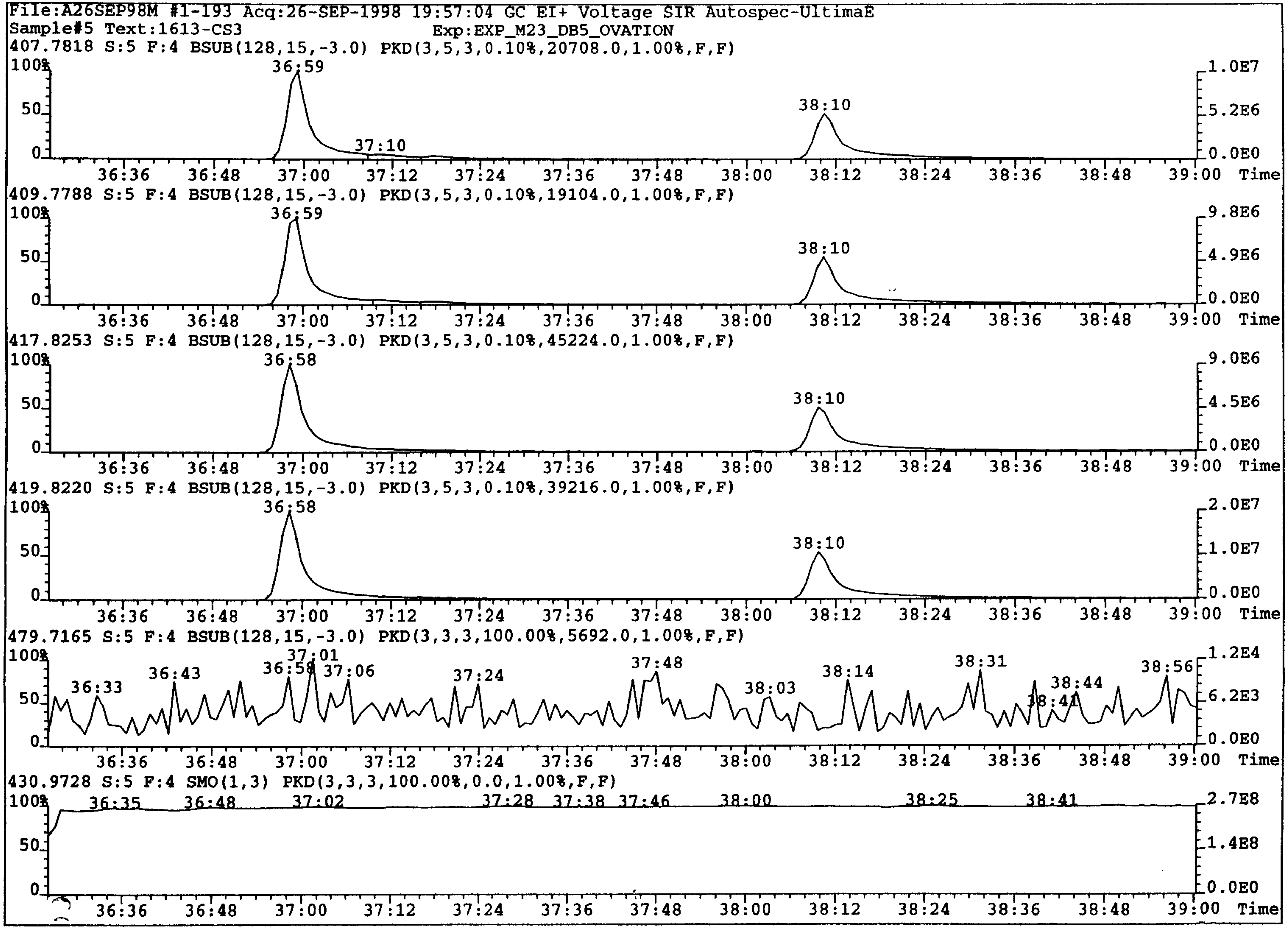
2

0.46

... was run, and showed lousy CtRs - The RRFs for OCDD & HpCDD (ES) were off. As the sequence of samples progressed, the RRFs became closer to normal. We decided to process the data using this BB CS3 instead of the Ical - Y.T. 30SEP98.



File:A26SEP98M #1-277 Acq:26-SEP-1998 19:57:04 GC EI+ Voltage SIR Autospec-UltimaE
Sample#5 Text:1613-CS3 Exp:EXP_M23_DB5_OVATION



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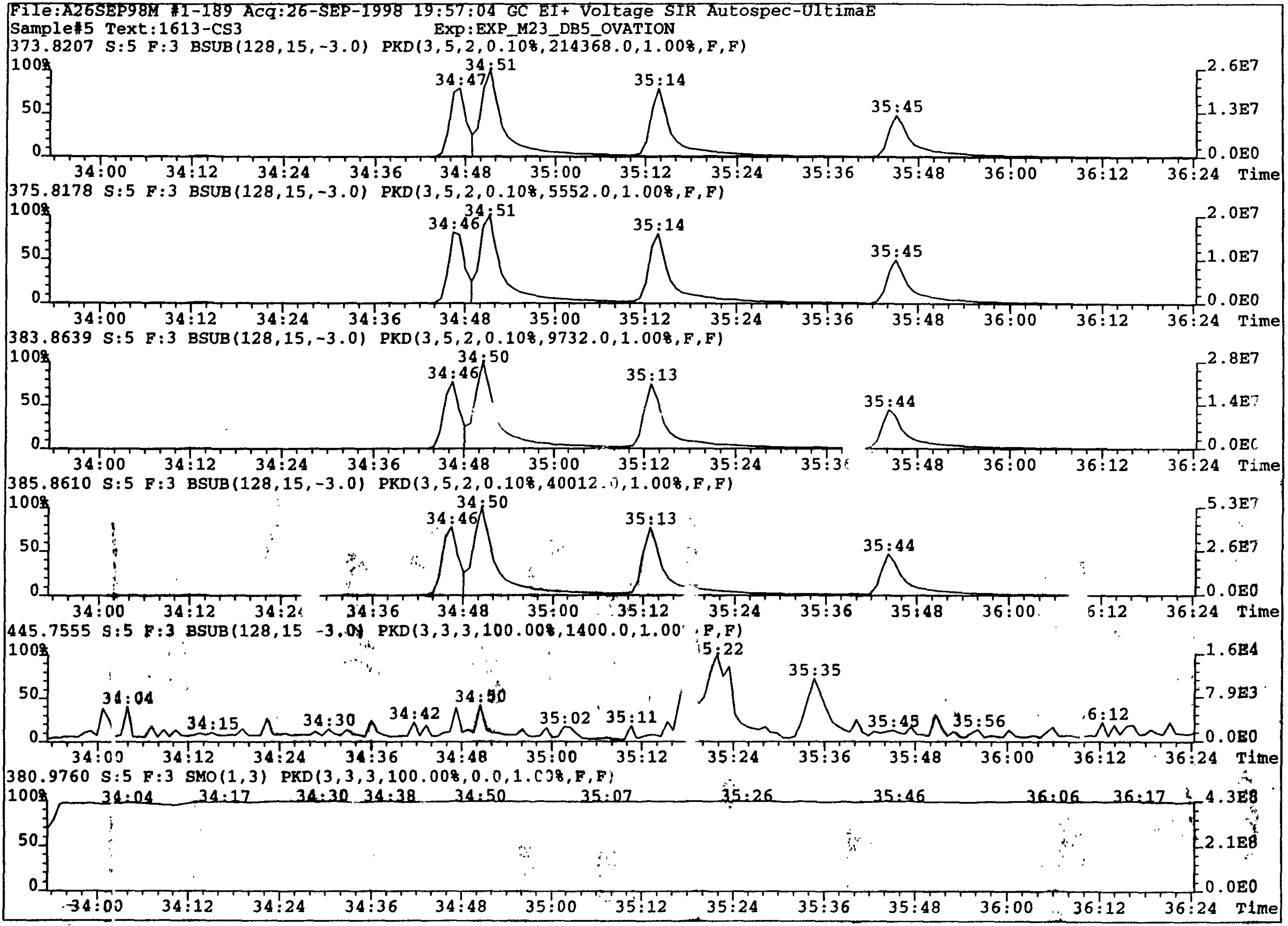
File:A26SEP98M #1-193 Acq:26-SEP-1998 19:57:04 GC EI+ Voltage SIR Autospec-UltimaE
Sample#5 Text:1613-CS3                    Exp:EXP_M23_DB5_OVATION
407.7818 S:5 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,20708.0,1.00%,F,F)
409.7788 S:5 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,19104.0,1.00%,F,F)
417.8253 S:5 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,45224.0,1.00%,F,F)
419.8220 S:5 F:4 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,39216.0,1.00%,F,F)
479.7165 S:5 F:4 BSUB(128,15,-3.0) PKD(3,3,3,100.00%,5692.0,1.00%,F,F)
430.9728 S:5 F:4 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)
```



File:A26SEP98M #1-189 Acq:26-SEP-1998 19:57:04 GC EI+ Voltage SIR Autospec-UltimaE
Sample#5 Text:1613-CS3 Exp:EXP_M23_DB5_OVATION

373.8207 S:5 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,214368.0,1.00%,F,F)

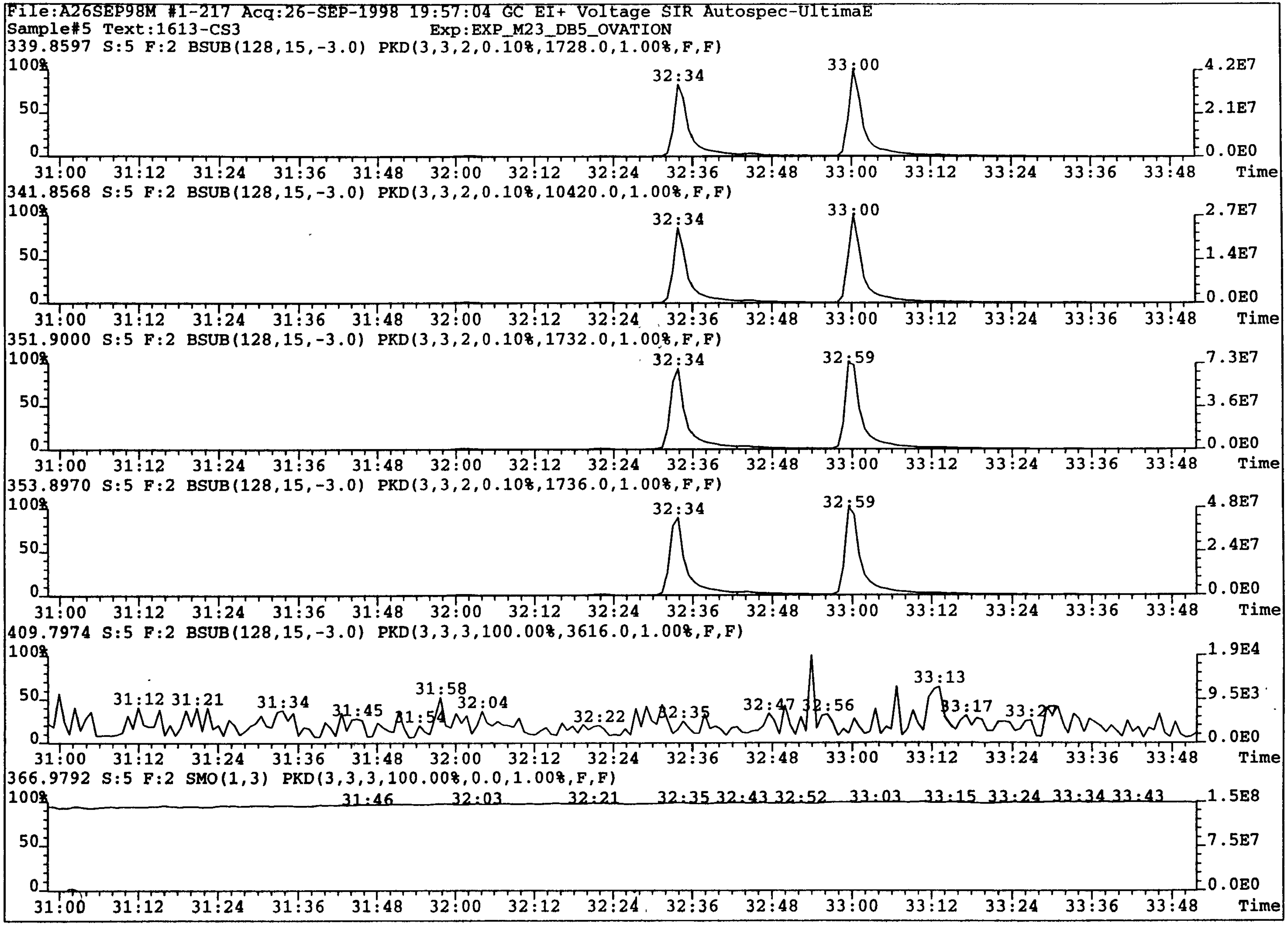
375.8178 S:5 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,5552.0,1.00%,F,F)

383.8639 S:5 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,9732.0,1.00%,F,F)

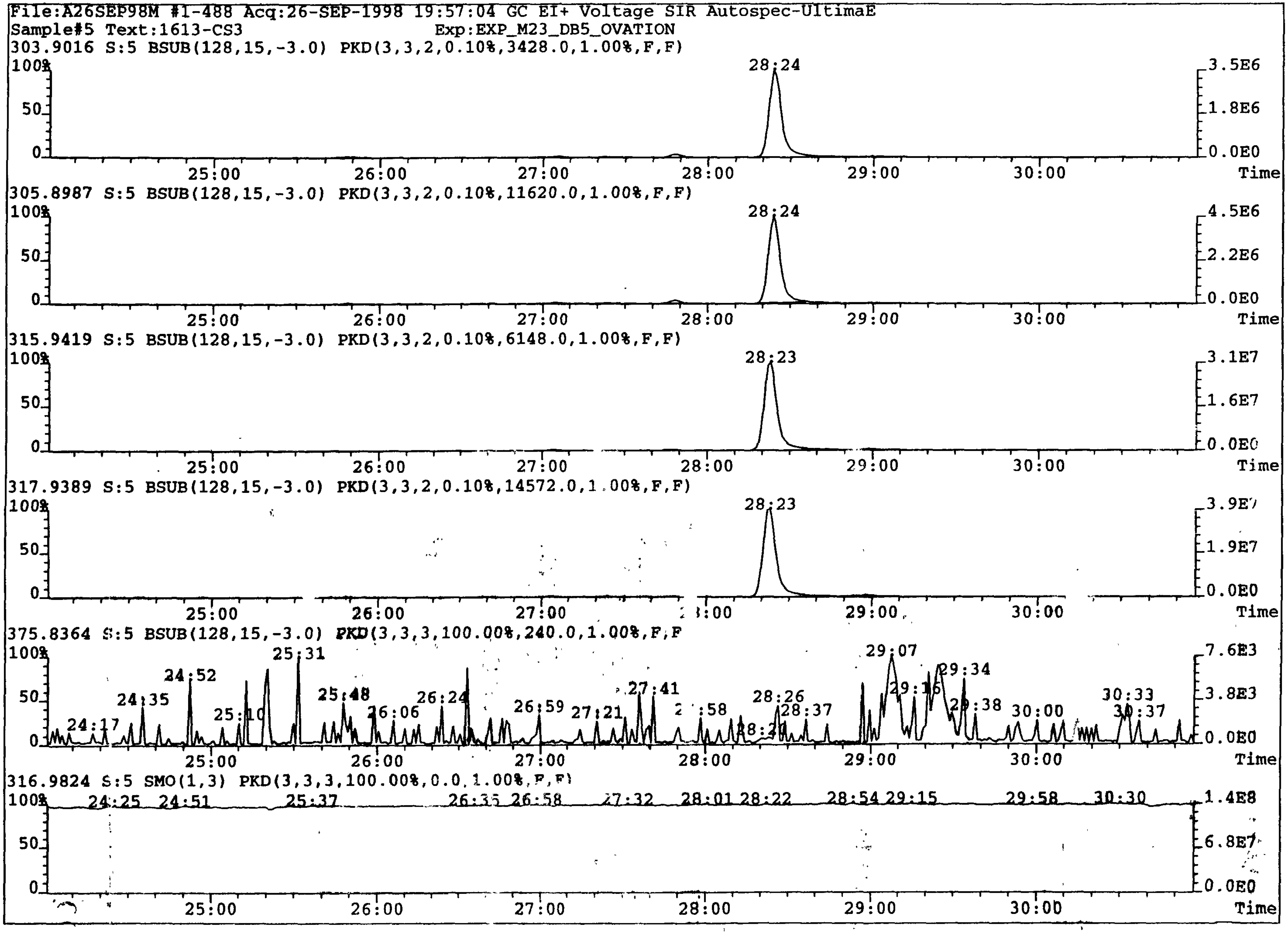
385.8610 S:5 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,40012.0,1.00%,F,F)

445.7555 S:5 F:3 BSUB(128,15,-3.0) PKD(3,3,3,100.00%,1400.0,1.00%,F,F)

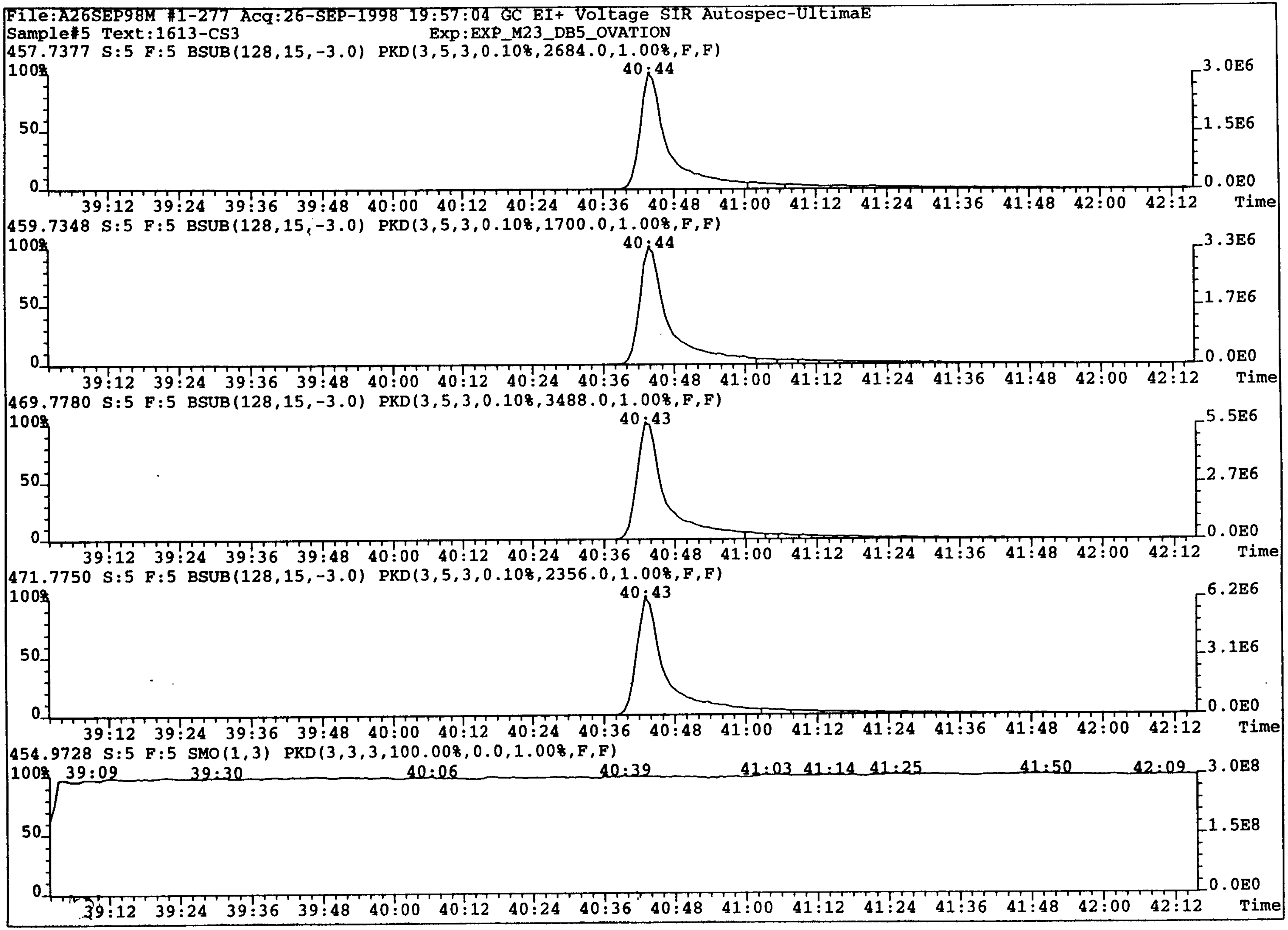
380.9760 S:5 F:3 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)



```
File:A26SEP98M #1-217 Acq:26-SEP-1998 19:57:04 GC EI+ Voltage SIR Autospec-UltimaE
Sample#5 Text:1613-CS3                    Exp:EXP_M23_DB5_OVATION
339.8597 S:5 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,1728.0,1.00%,F,F)
341.8568 S:5 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,10420.0,1.00%,F,F)
351.9000 S:5 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,1732.0,1.00%,F,F)
353.8970 S:5 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,1736.0,1.00%,F,F)
409.7974 S:5 F:2 BSUB(128,15,-3.0) PKD(3,3,3,100.00%,3616.0,1.00%,F,F)
366.9792 S:5 F:2 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)
```



```
File:A26SEP98M #1-488 Acq:26-SEP-1998 19:57:04 GC EI+ Voltage SIR Autospec-UltimaE
Sample#5 Text:1613-CS3                        Exp:EXP_M23_DB5_OVATION
303.9016 S:5 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,3428.0,1.00%,F,F)
305.8987 S:5 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,11620.0,1.00%,F,F)
315.9419 S:5 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,6148.0,1.00%,F,F)
317.9389 S:5 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,14572.0,1.00%,F,F)
375.8364 S:5 BSUB(128,15,-3.0) PKD(3,3,3,100.00%,240.0,1.00%,F,F
316.9824 S:5 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)
```



File:A26SEP98M #1-277 Acq:26-SEP-1998 19:57:04 GC EI+ Voltage SIR Autospec-UltimaE
Sample#5 Text:1613-CS3 Exp:EXP_M23_DB5_OVATION

457.7377 S:5 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,2684.0,1.00%,F,F)

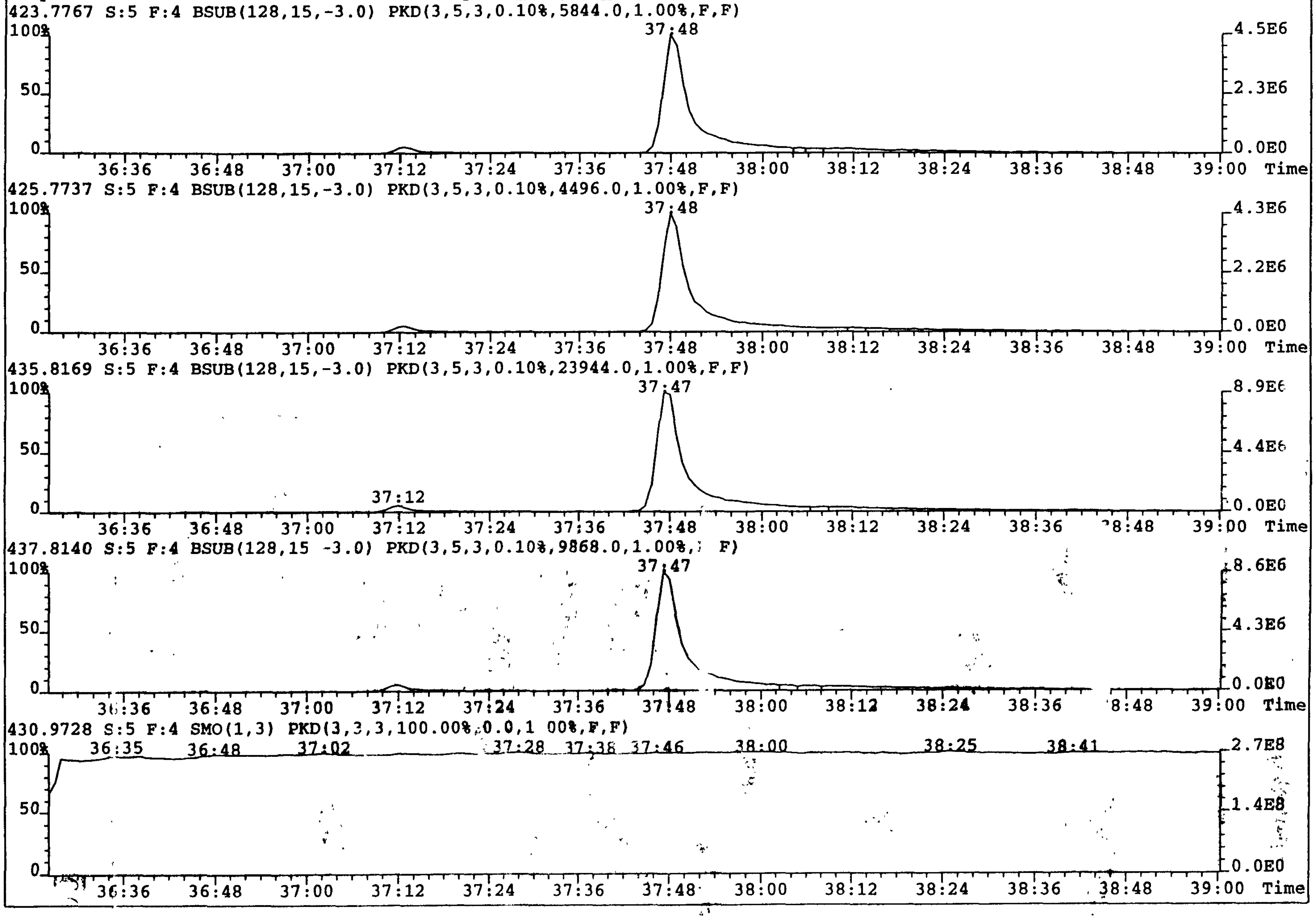
459.7348 S:5 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,1700.0,1.00%,F,F)

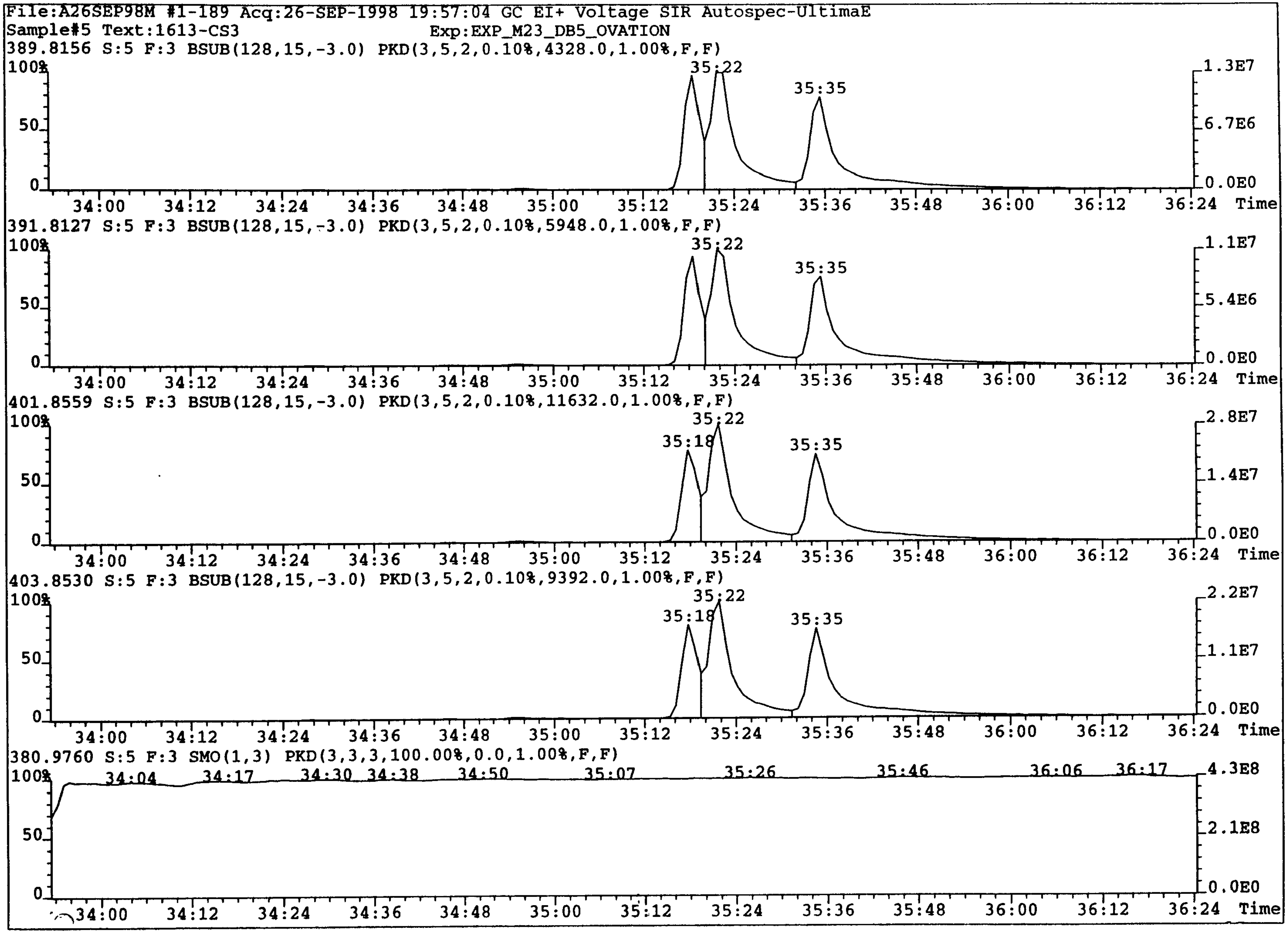
469.7780 S:5 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,3488.0,1.00%,F,F)

471.7750 S:5 F:5 BSUB(128,15,-3.0) PKD(3,5,3,0.10%,2356.0,1.00%,F,F)

454.9728 S:5 F:5 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)

File:A26SEP98M #1-193 Acq:26-SEP-1998 19:57:04 GC EI+ Voltage SIR Autospec-UltimaE
Sample#5 Text:1613-CS3 Exp:EXP_M23_DB5_OVATION





File:A26SEP98M #1-189 Acq:26-SEP-1998 19:57:04 GC EI+ Voltage SIR Autospec-UltimaE
Sample#5 Text:1613-CS3 Exp:EXP_M23_DB5_OVATION

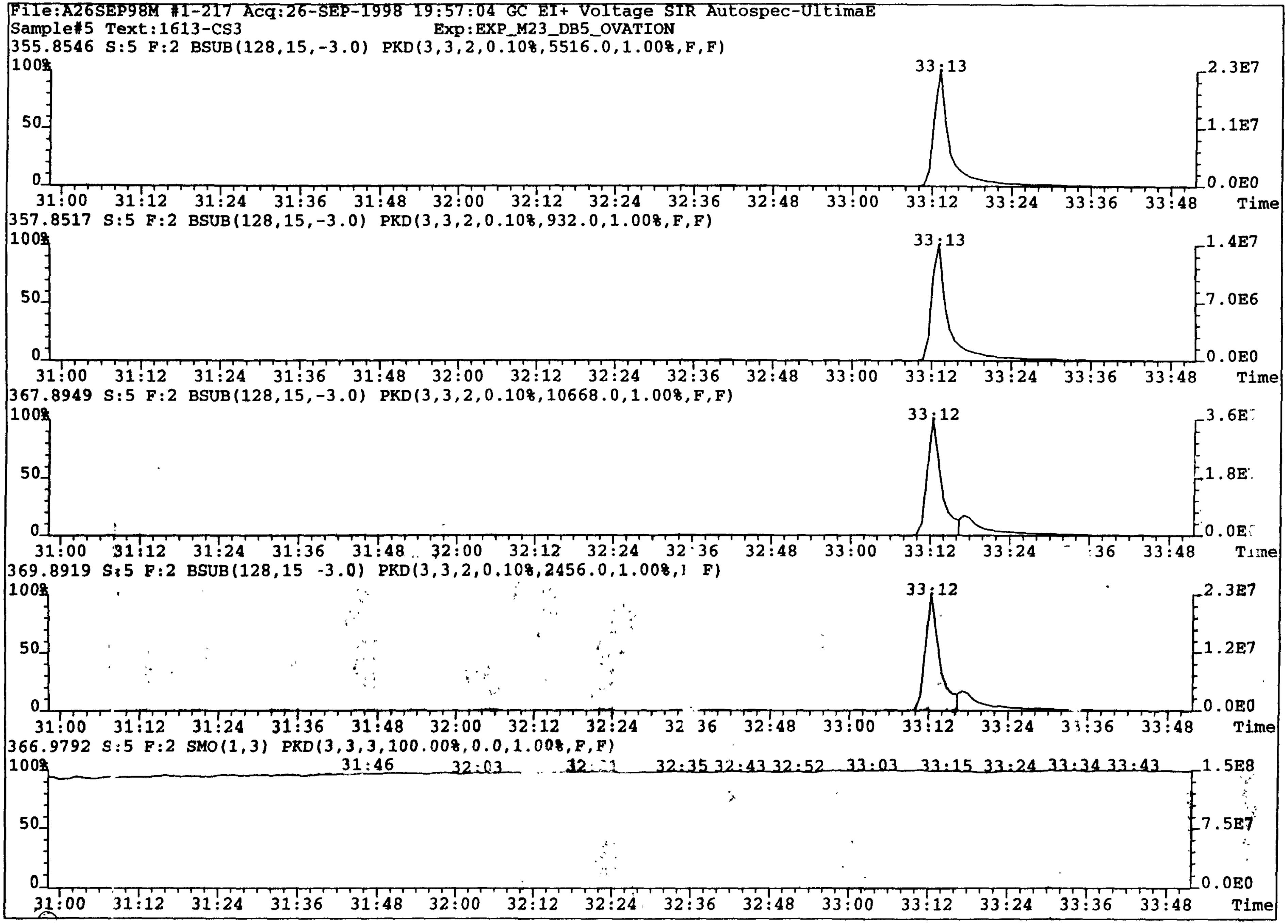
389.8156 S:5 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,4328.0,1.00%,F,F)

391.8127 S:5 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,5948.0,1.00%,F,F)

401.8559 S:5 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,11632.0,1.00%,F,F)

403.8530 S:5 F:3 BSUB(128,15,-3.0) PKD(3,5,2,0.10%,9392.0,1.00%,F,F)

380.9760 S:5 F:3 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)



File:A26SEP98M #1-217 Acq:26-SEP-1998 19:57:04 GC EI+ Voltage SIR Autospec-UltimaE
Sample#5 Text:1613-CS3 Exp:EXP_M23_DB5_OVATION

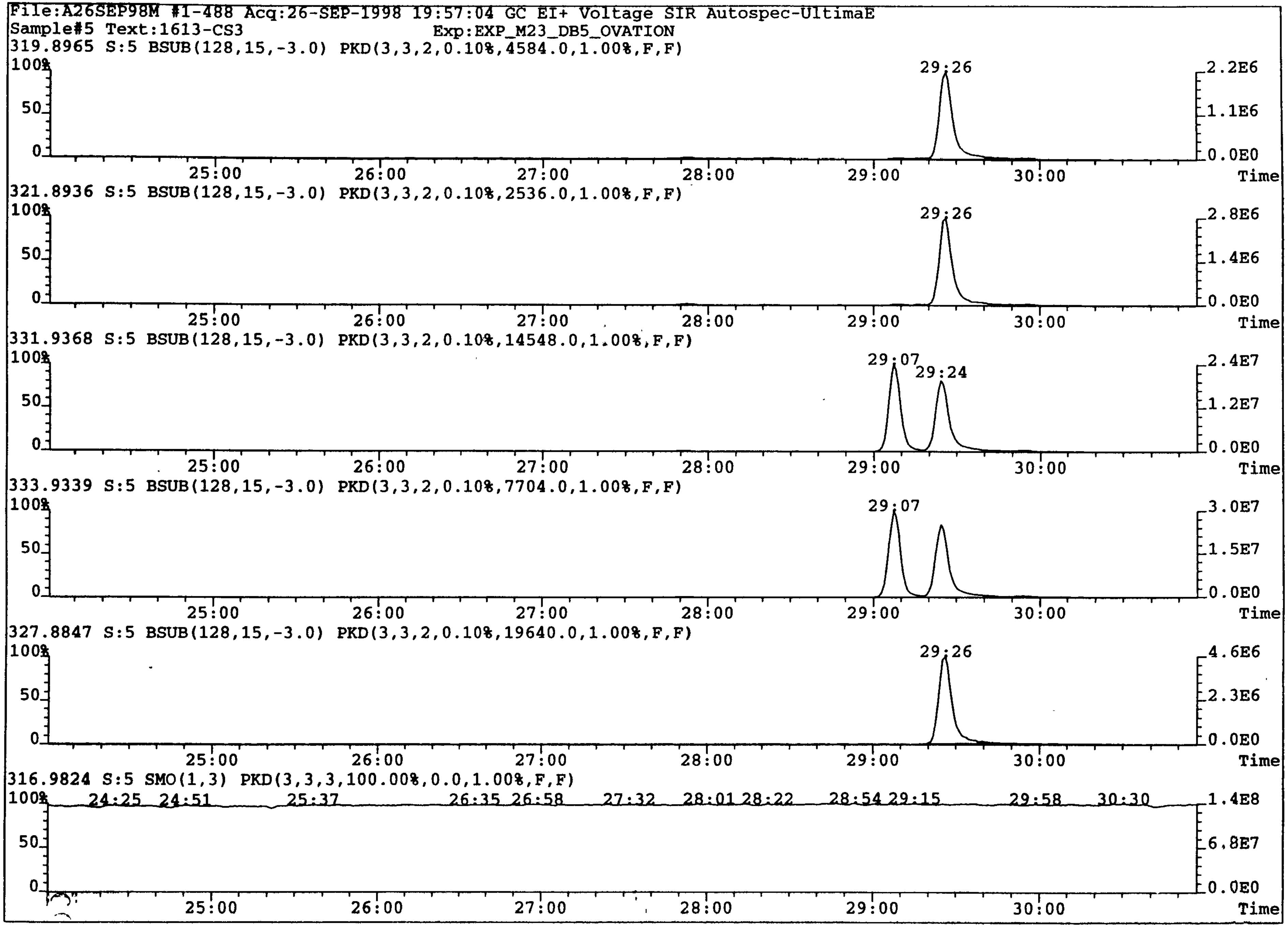
355.8546 S:5 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,5516.0,1.00%,F,F)

357.8517 S:5 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,932.0,1.00%,F,F)

367.8949 S:5 F:2 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,10668.0,1.00%,F,F)

369.8919 S:5 F:2 BSUB(128,15 -3.0) PKD(3,3,2,0.10%,2456.0,1.00%,F,F)

366.9792 S:5 F:2 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)


File:A26SEP98M #1-488 Acq:26-SEP-1998 19:57:04 GC EI+ Voltage SIR Autospec-UltimaE
Sample#5 Text:1613-CS3 Exp:EXP_M23_DB5_OVATION

319.8965 S:5 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,4584.0,1.00%,F,F)

321.8936 S:5 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,2536.0,1.00%,F,F)

331.9368 S:5 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,14548.0,1.00%,F,F)

333.9339 S:5 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,7704.0,1.00%,F,F)

327.8847 S:5 BSUB(128,15,-3.0) PKD(3,3,2,0.10%,19640.0,1.00%,F,F)

316.9824 S:5 SMO(1,3) PKD(3,3,3,100.00%,0.0,1.00%,F,F)

```
41    Tot        Total Penta-Furans    0.00   -          - n       -          -   1.04   y    n
42    Tot       Total Penta-Dioxins    0.00   -          - n       -          -   1.41   y    n
43    Tot         Total Hexa-Furans    0.00   -          - n       -          -   1.62   y    n
44    Tot        Total Hexa-Dioxins    0.00   -          - n       -          -   0.81   y    n
45    Tot        Total Hepta-Furans    0.00   -          - n       -          -   1.10   y    n
46    Tot       Total Hepta-Dioxins    0.00   -          - n       -          -   0.98   y    n

47    DPE                    HxCDPE    1.00   *                 NotF»       *       -   n    n
48    DPE                    HpCDPE    1.00   *                 NotF»       *       -   n    n
49    DPE                     OCDPE    1.00   *                 NotF»       *       -   n    n
50    DPE                     NCDPE    1.00   *                 NotF»       *       -   n    n
51    DPE                     DCDPE    1.00   *                 NotF»       *       -   n    n

52    LMC        QC CHK ION (Tetra)    1.00   *                 NotF»       *       -   n    n
53    LMC        QC CHK ION (Penta)    1.00   *                 NotF»       *       -   n    n
54    LMC         QC CHK ION (Hexa)    1.00   *                 NotF»       *       -   n    n
55    LMC        QC CHK ION (Hepta)    1.00   *                 NotF»       *       -   n    n
56    LMC         QC CHK ION (Octa)    1.00   *                 NotF»       *       -   n    n
```

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                                                                                          Page 3 of 5

   Run #3    Filename a26sep98m         S: 5    I: 1  Acquired: 26-SEP-98 19:57:04 Processed: 27-SEP-98 09:50:53
   Run: 26sep-crv  Analyte: m8290-092» Cal: m8290-092» Results:             Version: V3.6 31-JUL-1998 10:51:59
   Sample text: 1613-CS3                                     Comments:

     Typ                      Name    Amount   Resp          RA   ✓   RT           RF      RRF        Modified?

 1   Unk              2,3,7,8-TCDD     10.00   2.85e+07   0.78 y   29:26           -      1.10   y    n
 2   Unk            1,2,3,7,8-PeCDD    50.00   1.03e+08   1.59 y   33:13           -      1.41   y    n
 3   Unk          1,2,3,4,7,8-HxCDD    50.00   5.25e+07   1.25 y   35:19           -      0.56   y    n
 4   Unk          1,2,3,6,7,8-HxCDD    50.00   9.17e+07   1.28 y   35:22           -      0.99   y    n
 5   Unk          1,2,3,7,8,9-HxCDD    50.00   8.19e+07   1.30 y   35:35           -      0.88   y    n
 6   Unk        1,2,3,4,6,7,8-HpCDD    50.00   3.88e+07   0.98 y   37:48           -      0.98   y    n
 7   Unk                       OCDD   100.00   3.49e+07   0.88 y   40:44           -      1.05   y    n

 8   Unk              2,3,7,8-TCDF     10.00   4.10e+07   0.82 y   28:24           -      1.07   y    n
 9   Unk            1,2,3,7,8-PeCDF    50.00   1.50e+08   1.54 y   32:34           -      0.97   y    n
 10  Unk            2,3,4,7,8-PeCDF    50.00   1.72e+08   1.52 y   33:00           -      1.11   y    n
 11  Unk          1,2,3,4,7,8-HxCDF    50.00   8.89e+07   1.22 y   34:47           -      1.24   y    n
 12  Unk          1,2,3,6,7,8-HxCDF    50.00   1.66e+08   1.22 y   34:51           -      2.33   y    n
 13  Unk          2,3,4,6,7,8-HxCDF    50.00   1.23e+08   1.12 y   35:14           -      1.72   y    n
 14  Unk          1,2,3,7,8,9-HxCDF    50.00   8.60e+07   1.18 y   35:45           -      1.20   y    n
 15  Unk        1,2,3,4,6,7,8-HpCDF    50.00   7.37e+07   1.03 y   36:59           -      1.32   y    n
 16  Unk        1,2,3,4,7,8,9-HpCDF    50.00   4.92e+07   1.13 y   38:10           -      0.88   y    n
 17  Unk                       OCDF   100.00   4.42e+07   0.84 y   40:53           -      1.34   y    n

 18  ES/RT        13C-2,3,7,8-TCDD    100.00   2.58e+08   0.81 y   29:24           -      0.99   y    n
 19  ES        13C-1,2,3,7,8-PeCDD    100.00   1.45e+08   1.58 y   33:12           -      0.56   y    n
 20  ES      13C-1,2,3,6,7,8-HxCDD    100.00   1.86e+08   1.29 y   35:22           -      1.14   y    n
 21  ES    13C-1,2,3,4,6,7,8-HpCDD    100.00   7.90e+07   1.10 y   37:47           -      0.48   y    n
 22  ES                   13C-OCDD    200.00   6.62e+07   0.89 y   40:43           -      0.20   y    n

 23  ES/RT        13C-2,3,7,8-TCDF    100.00   3.81e+08   0.80 y   28:23           -      1.47   y    n
 24  ES        13C-1,2,3,7,8-PeCDF    100.00   3.09e+08   1.58 y   32:34           -      1.19   y    n
 25  ES      13C-1,2,3,6,7,8-HxCDF    100.00   1.43e+08   0.52 y   34:46           -      0.87   y    n
 26  ES    13C-1,2,3,4,6,7,8-HpCDF    100.00   1.12e+08   0.47 y   36:58           -      0.68   y    n

 27  JS          13C-1,2,3,4-TCDD     100.00   2.60e+08   0.80 y   29:07    2.60e+06         -   n    n
 28  JS      13C-1,2,3,7,8,9-HxCDD    100.00   1.64e+08   1.28 y   35:35    1.64e+06         -   n    n

 29  CS           37Cl-2,3,7,8-TCDD    10.00   2.52e+07            29:26           -      0.97   y    n
 30  CS        13C-2,3,4,7,8-PeCDF    100.00   3.44e+08   1.58 y   32:59           -      1.32   y    n
 31  CS      13C-1,2,3,4,7,8-HxCDD    100.00   8.57e+07   1.26 y   35:18           -      0.52   y    n
 32  CS      13C-1,2,3,4,7,8-HxCDF    100.00   2.91e+08   0.53 y   34:50           -      1.78   y    n
 33  CS    13C-1,2,3,4,7,8,9-HpCDF    100.00   6.97e+07   0.44 y   38:10           -      0.43   y    n

 34  SS           37Cl-2,3,7,8-TCDD    10.00   2.52e+07            29:26           -      0.98   y    n
 35  SS        13C-2,3,4,7,8-PeCDF    100.00   3.44e+08   1.58 y   32:59           -      1.11   y    n
 36  SS      13C-1,2,3,4,7,8-HxCDD    100.00   8.57e+07   1.26 y   35:18           -      0.46   y    n
 37  SS      13C-1,2,3,4,7,8-HxCDF    100.00   2.91e+08   0.53 y   34:50           -      2.04   y    n
 38  SS    13C-1,2,3,4,7,8,9-HpCDF    100.00   6.97e+07   0.44 y   38:10           -      0.62   y    n

 39  Tot       Total Tetra-Furans       0.00   -             - n     -             -      1.07   y    n
 40  Tot       Total Tetra-Dioxins      0.00   -             - n     -             -      1.10   y    n
```

# Section 4

## System Performance

**Paradigm Analytical Labs**

# Section 4-4

# Continuing Calibrations

## Documentation for the Analysis
## of
## Polychlorinated Dibenzo-*p*-Dioxins & Dibenzofurans

DB -225

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                                                                                                    Page 1 of 1

   Run: 07FEB98     Analyte: M23_CONF   Cal: 225-07feb  Results:            Version: V3.5 17-APR-1997 11:14:34

                                                        07feb98d S4 07feb98d S5 07feb98d S6 07feb98d S7 07feb98d S8
                     Name Mean RRF     S. D.      %RSD    RRF#1    SD  RRF#2    SD  RRF#3    SD  RRF#4    SD  RRF#5    SD

             2,3,7,8-TCDF  0.9472    0.033      3.49 %     1.00   1.5   0.91  -1.3   0.94  -0.4   0.95   0.0   0.95   0.1
         13C-2,3,7,8-TCDF       -        -         - %        -     -      -     -      -     -      -     -      -     -
                   HxCDPE       -        -         - %        -     -      -     -      -     -      -     -      -     -
      QC CHK ION (Tetra)        -        -         - %        -     -      -     -      -     -      -     -      -     -
```

[m 8290 - 091498 m] ICal

1613 8/7

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OPUSquan    16-SEP-1998                 Page 1
                                                                                   Page 1 of 1

  Run: 14sep-crv  Analyte: m8290      Cal:           Results:           Version: V3.5 17-APR-1997 11:14:34

                                                         14sep98m S3 14sep98m S4 14sep98m S5 14sep98m S6 14sep98m S7
                      Name Mean RRF     S. D.      %RSD    RRF#1    SD   RRF#2   SD   RRF#3   SD   RRF#4   SD   RRF#5   SD

              2,3,7,8-TCDD  1.0257     0.030    2.96 %    1.08  1.7    1.02 -0.3    1.01 -0.6    1.00 -0.8    1.02 -0.1
           1,2,3,7,8-PeCDD  1.1457     0.026    2.26 %    1.15  0.3    1.16  0.7    1.16  0.5    1.15  0.3    1.10 -1.8
         1,2,3,4,7,8-HxCDD  0.8199     0.038    4.66 %    0.81 -0.2    0.80 -0.6    0.84  0.4    0.78 -1.1    0.88  1.5
         1,2,3,6,7,8-HxCDD  0.9128     0.059    6.50 %    0.87 -0.7    1.02  1.7    0.88 -0.6    0.91 -0.1    0.89 -0.4
         1,2,3,7,8,9-HxCDD  0.8982     0.042    4.65 %    0.85 -1.1    0.96  1.5    0.91  0.2    0.87 -0.6    0.90  0.0
       1,2,3,4,6,7,8-HpCDD  0.9131     0.008    0.87 %    0.91 -0.5    0.91 -0.9    0.91 -0.8    0.92  1.3    0.92  0.8
                      OCDD  1.0044     0.015    1.53 %    1.02  1.1    1.01  0.3    1.01  0.6    0.99 -0.6    0.98 -1.4

              2,3,7,8-TCDF  0.9992     0.013    1.33 %    1.01  1.1    0.98 -1.1    1.00 -0.3    0.99 -0.7    1.01  1.0
           1,2,3,7,8-PeCDF  0.8955     0.013    1.43 %    0.87 -1.6    0.89  0.0    0.90  0.4    0.91  1.1    0.90  0.2
           2,3,4,7,8-PeCDF  0.9204     0.029    3.13 %    0.93  0.4    0.93  0.2    0.93  0.4    0.94  0.7    0.87 -1.8
         1,2,3,4,7,8-HxCDF  0.9410     0.022    2.37 %    0.95  0.4    0.91 -1.3    0.97  1.1    0.95  0.6    0.92 -0.7
         1,2,3,6,7,8-HxCDF  1.1148     0.042    3.72 %    1.14  0.7    1.12  0.2    1.16  1.0    1.10 -0.4    1.05 -1.5
         2,3,4,6,7,8-HxCDF  1.0006     0.043    4.34 %    1.06  1.4    0.98 -0.5    1.02  0.5    0.98 -0.4    0.95 -1.1
         1,2,3,7,8,9-HxCDF  0.8709     0.026    3.03 %    0.87  0.1    0.83 -1.5    0.89  0.7    0.86 -0.4    0.90  1.0
       1,2,3,4,6,7,8-HpCDF  1.3737     0.012    0.86 %    1.37 -0.2    1.37 -0.5    1.39  1.7    1.36 -1.0    1.37  0.0
       1,2,3,4,7,8,9-HpCDF  1.1710     0.013    1.15 %    1.16 -0.8    1.16 -0.8    1.19  1.4    1.16 -0.6    1.18  0.8
                      OCDF  1.0873     0.020    1.85 %    1.06 -1.5    1.08 -0.5    1.09  0.3    1.11  1.0    1.10  0.7

          13C-2,3,7,8-TCDD  1.0598     0.030    2.86 %    1.04 -0.6    1.04 -0.8    1.05 -0.4    1.06  0.0    1.11  1.7
       13C-1,2,3,7,8-PeCDD  0.6999     0.054    7.67 %    0.66 -0.7    0.65 -0.8    0.68 -0.4    0.71  0.2    0.79  1.6
     13C-1,2,3,6,7,8-HxCDD  1.0514     0.030    2.83 %    1.04 -0.2    1.00 -1.6    1.06  0.2    1.08  0.9    1.07  0.8
   13C-1,2,3,4,6,7,8-HpCDD  0.8753     0.017    1.98 %    0.87 -0.5    0.87 -0.3    0.87 -0.1    0.86 -0.8    0.91  1.7
                  13C-OCDD  0.7701     0.038    4.93 %    0.74 -0.9    0.76 -0.4    0.75 -0.5    0.77  0.0    0.83  1.7

          13C-2,3,7,8-TCDF  1.3145     0.018    1.40 %    1.31 -0.1    1.30 -1.0    1.30 -0.7    1.32  0.3    1.34  1.6
       13C-1,2,3,7,8-PeCDF  1.1002     0.062    5.65 %    1.06 -0.7    1.06 -0.7    1.08 -0.4    1.10  0.0    1.     1.7
     13C-1,2,3,6,7,8-HxCDF  1.2071     0.034    2.84 %    1.17 -1.0    1.26  1.4    1.18 -0.9    1.22  0.3    1.     0.1
   13C-1,2,3,4,6,7,8-HpCDF  0.7878     0.017    2.21 %    0.77 -0.[illegible]  0.79  0.0    0.77 -1.0    0.80  0.5    0.     1.4

          13C-1,2,3,4-TCDD       -         -       - %       -    -       -    -       -    -       -    -            -
     13C-1,2,3,7,8,9-HxCDD       -         -       - %       -    -       -    -       -    -       -    -            -

          37Cl-2,3,7,8-TCDD  1.0350     0.042    4.05 %    1.01 -0.5    0.99 -1.1    1.03 -0.1    1.04  0.2    1.     1.5
        13C-2,3,4,7,8-PeCDF  1.0782     0.068    6.30 %    1.03 -0.3    1.03 -0.7    1.05 -0.4    1.09  0.2    1.     1.6
      13C-1,2,3,4,7,8-HxCDD  0.7931     0.042    5.29 %    0.75 -0.[illegible]  0.84  1.2    0.78 -0.3    0.76 -0.9    0.[illegible]3  1.0
      13C-1,2,3,4,7,8-HxCDF  0.9989     0.035    3.50 %    0.98 -0.7    0.98 -0.6    1.00  0.1    0.98 -0.5    1.06  1.7
    13C-1,2,3,4,7,8,9-HpCDF  0.6859     0.015    2.21 %    0.66 -1.4    0.69  0.1    0.68 -0.2    0.69  0.2    0.71  1.4

          37Cl-2,3,7,8-TCDD  0.9764     0.01[illegible]    [illegible].54 %    0.97 -0.5    0.95 -1.5    0.99  0.6    0.98  0.5    0.99  0.8
        13C-2,3,4,7,8-PeCDF  0.9797     0.009    0.90 %    0.97 -1.3    0.98 -0.4    0.98 -0.3    0.99  1.2    0.99  0.8
      13C-1,2,3,4,7,8-HxCDD  0.7552     0.054    7.15 %    0.72 -0.6    0.84  1.5    0.74 -0.3    0.70 -1.0    0.78  0.4
      13C-1,2,3,4,7,8-HxCDF  0.8281     0.038    4.53 %    0.83  0.1    0.78 -1.4    0.85  0.6    0.81 -0.6    0.87  1.2
    13C-1,2,3,4,7,8,9-HpCDF  0.8707     0.010    1.12 %    0.86 -1.1    0.87  0.1    0.89  1.6    0.86 -0.6    0.87 -0.1

        Total Tetra-Furans  0.9992     0.013    1.33 %    1.01  1.1    0.98 -1.1    1.00 -0.3    0.99 -0.7    1.01  1.0
       Total Tetra-Dioxins  1.0257     0.030    2.96 %    1.08  1.7    1.02 -0.3    1.01 -0.6    1.00 -0.8    1.02 -0.1
        Total Penta-Furans  0.9080     0.016    1.72 %    0.90 -0.3    0.91  0.2    0.92  0.5    0.93  1.1    0.88 -1.5
```

8290, 1613

CS

ES

SS

(23)

# Section 4

## System Performance

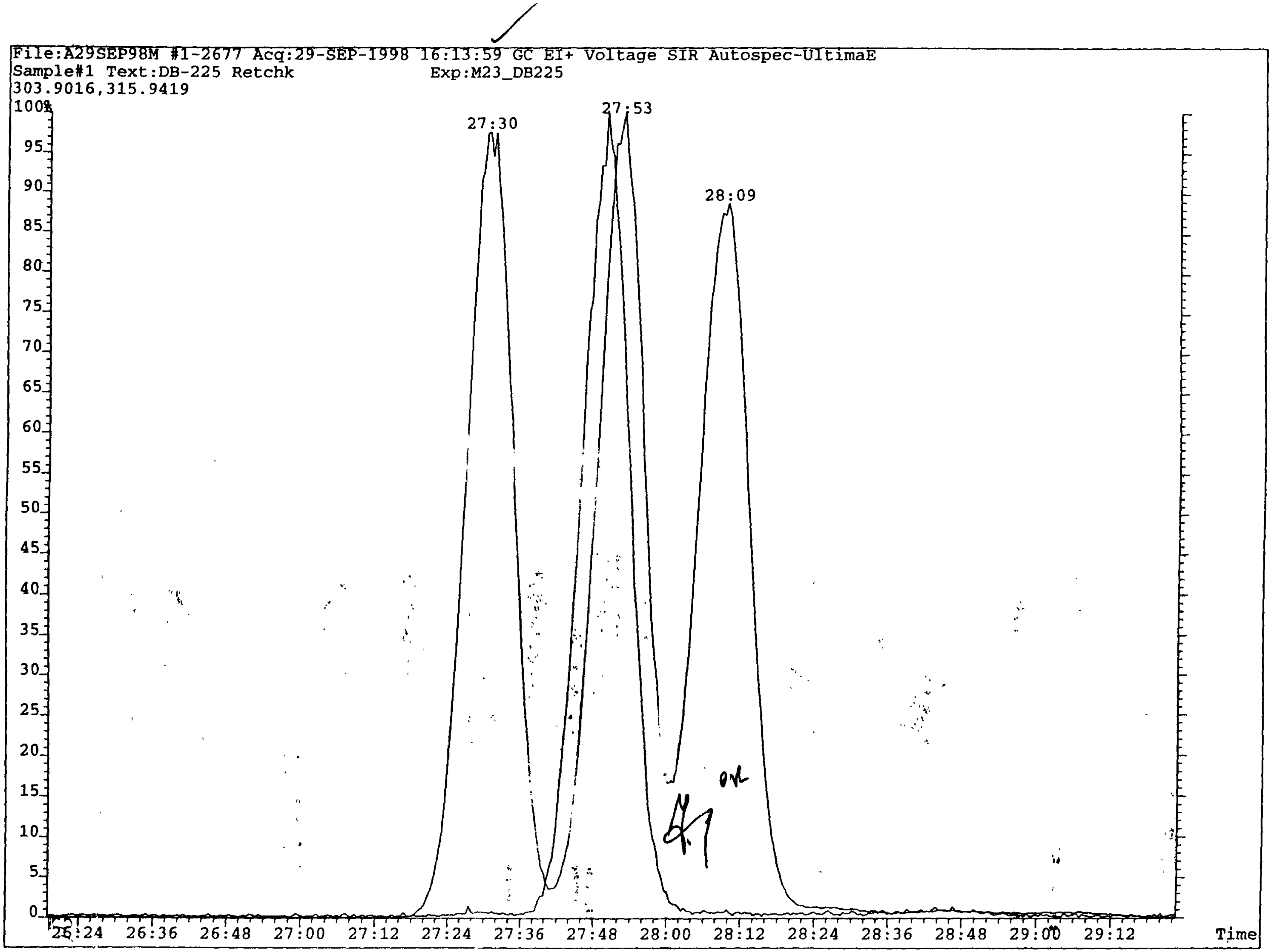
Paradigm Analytical Labs

## Section 4-3

# Initial Calibrations

(HP-5MS & DB-225 Columns)

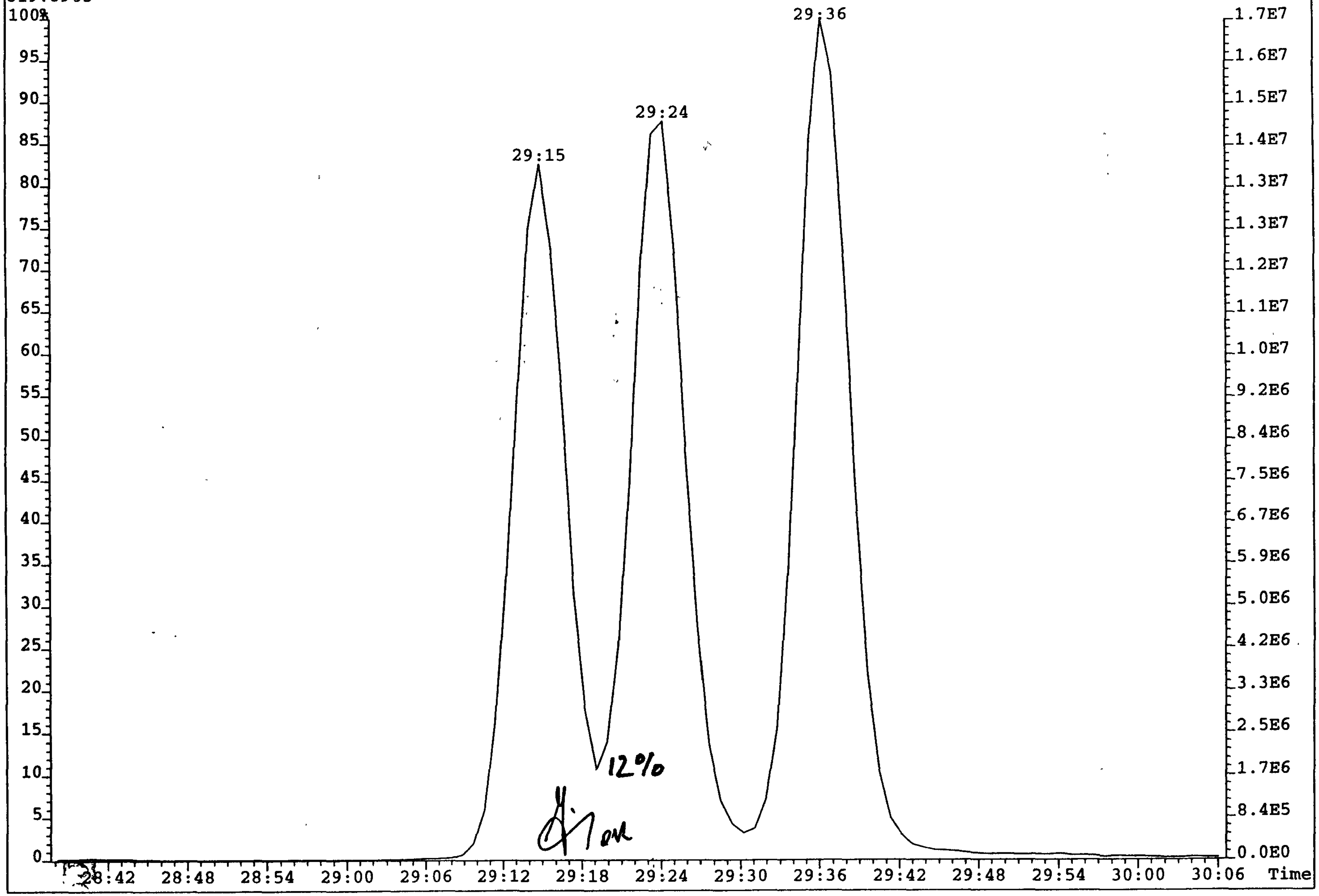
## Documentation for the Analysis
## of
## Polychlorinated Dibenzo-*p*-Dioxins & Dibenzofurans



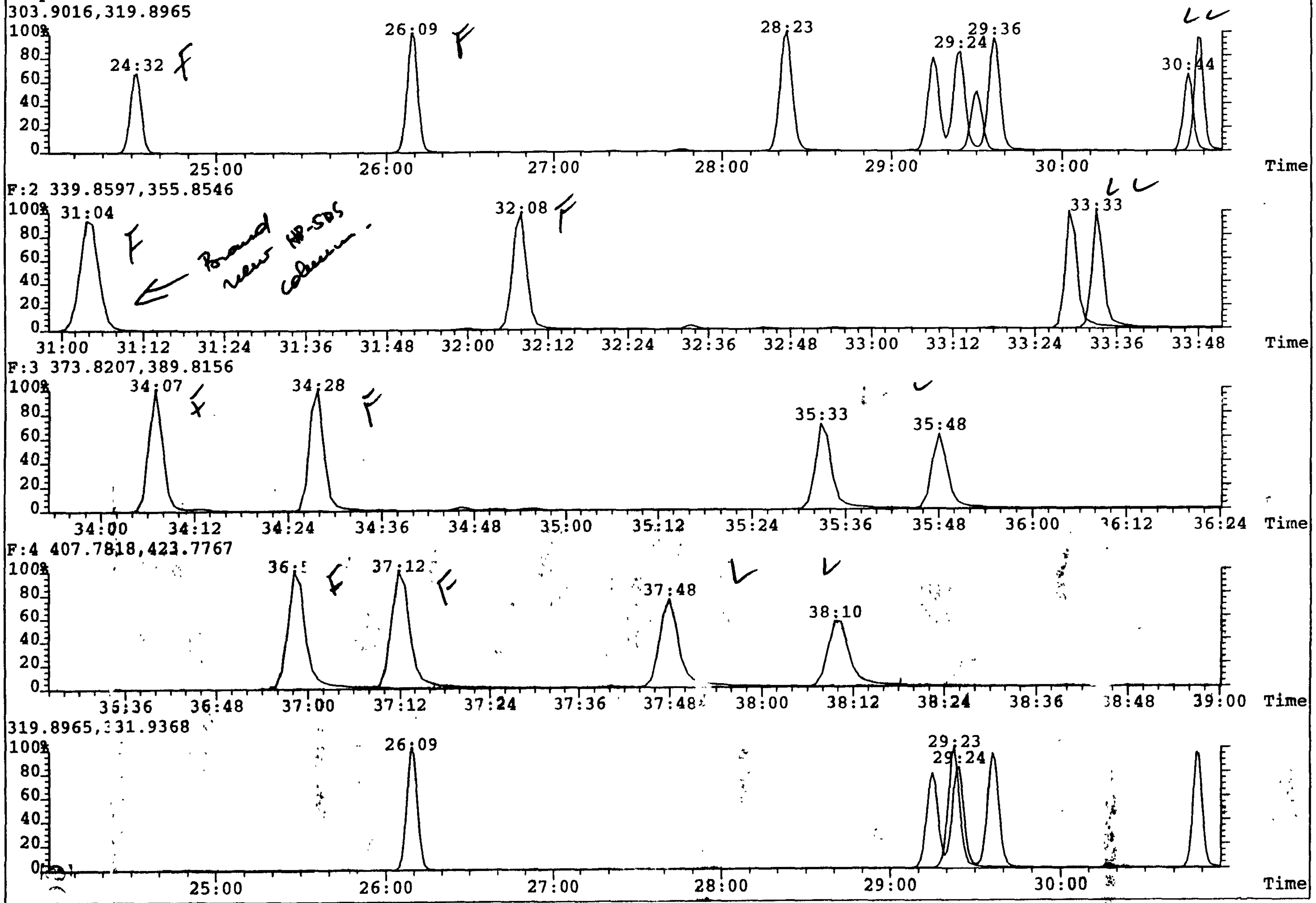
File:A29SEP98M #1-2677 Acq:29-SEP-1998 16:13:59 GC EI+ Voltage SIR Autospec-UltimaE
Sample#1 Text:DB-225 Retchk Exp:M23_DB225
303.9016,315.9419

ok

File: A26SEP98M  Acq: 26-SEP-1998 16:46:46  Exp: EXP_M23_DB5_OVATION Voltage SIR EI+  GC Autospec-UltimaE  Paradigm
Sample #1  Text: DB-5 Retchk  ALS #1
319.8965



File: A26SEP98M Acq: 26-SEP-1998 16:46:46 Exp: EXP_M23_DB5_OVATION Voltage SIR EI+ GC Autospec-UltimaE Paradigm
Sample #1 Text: DB-5 Retchk ALS #1



1

$T_s$ = Absolute stack temperature, °K (°R).

$$= 273 + t_s$$

Eq. 2-7

for metric.

$$= 460 + t_s$$

Eq. 2-8

for English.

$T_{std}$ = Standard absolute temperature, 293°K (528°R).

$v_s$ = Average stack gas velocity, m/sec (ft/sec).

$\Delta p$ = Velocity head of stack gas, mm $H_2O$ (in. $H_2O$).

3,600= Conversion factor, sec/hr.

18.0 = Molecular weight of water, g/g-mole (lb/lb-mole).

**5.2 Average Stack Gas Velocity.**

$$v_s = K_p C_p \left(\sqrt{\Delta p}\right)_{avg} \sqrt{\frac{T_{s(avg)}}{P_s M_s}}$$

Eq. 2-9

**5.3 Average Stack Gas Dry Volumetric Flow Rate.**

$$Q_{sd} = 3,600(1 - B_{ws}) v_s A \frac{T_{std}}{T_{s(avg)}} \frac{P_s}{P_{std}}$$

Eq. 2-10

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17. Ower, E. and R.C. Pankhurst. The Measurement of Air Flow, 4th Ed. London, Pergamon Press. 1966.

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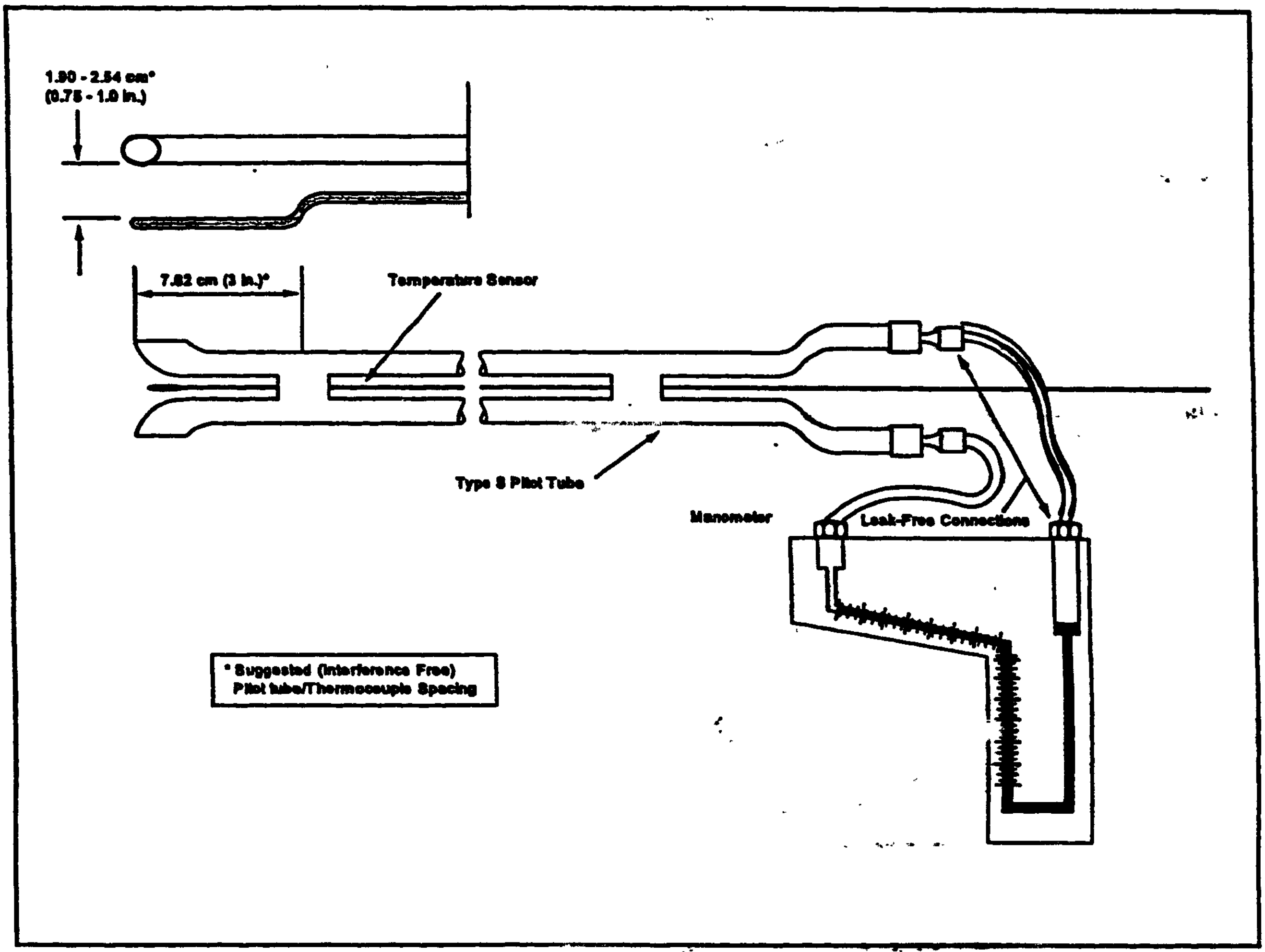
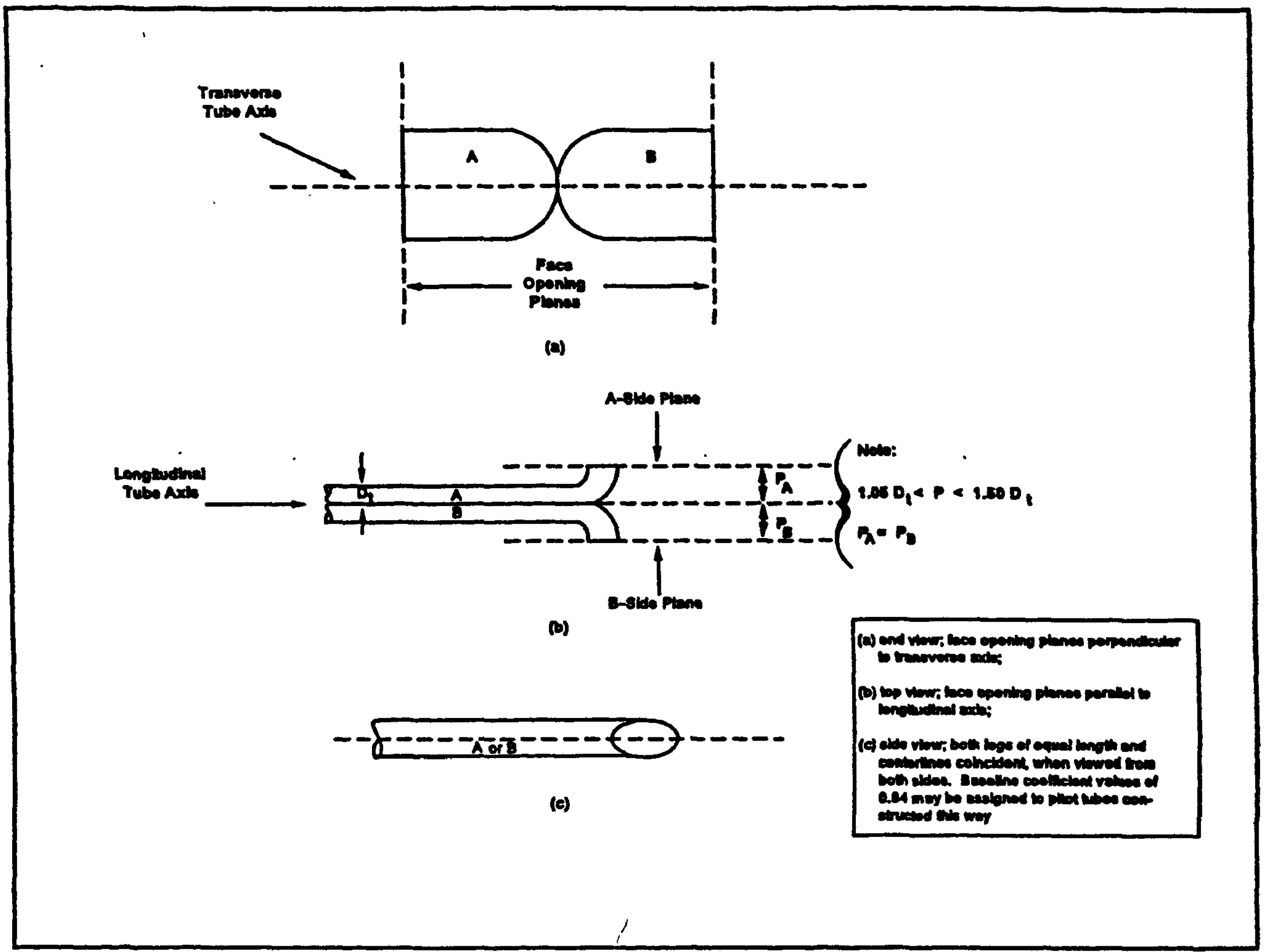


Figure 2-1. Type S pitot tube manometer assembly.



Note:

$1.05\ D_t < P < 1.50\ D_t$

$P_A = P_B$

(a) end view; face opening planes perpendicular to transverse axis;

(b) top view; face opening planes parallel to longitudinal axis;

(c) side view; both legs of equal length and centerlines coincident, when viewed from both sides. Baseline coefficient values of 0.84 may be assigned to pitot tubes constructed this way

Figure 2-2. Properly constructed Type S pitot tube.

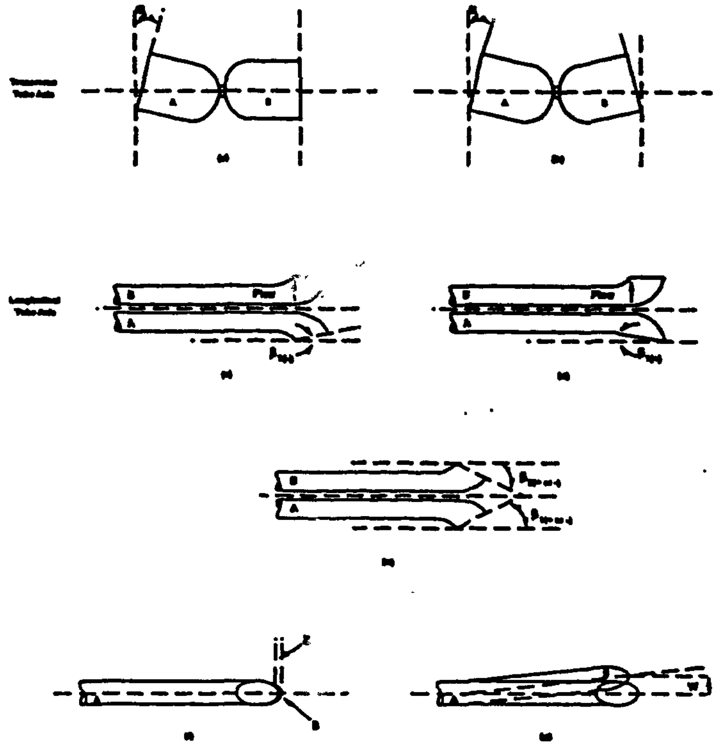


Figure 2-3. Types of face-opening misalignment that can result from field use or improper construction of Type S pitot tubes. These will not affect the baseline value of Cp(s) so long as $\alpha^1$ and $\alpha^2$ ≤10°, $\beta^1$ and $\beta^2$ ≤5°, z ≤0.32 cm (1/8 in.) and w ≤0.08 cm (1/32 in.) (citation 11 in Bibliography).

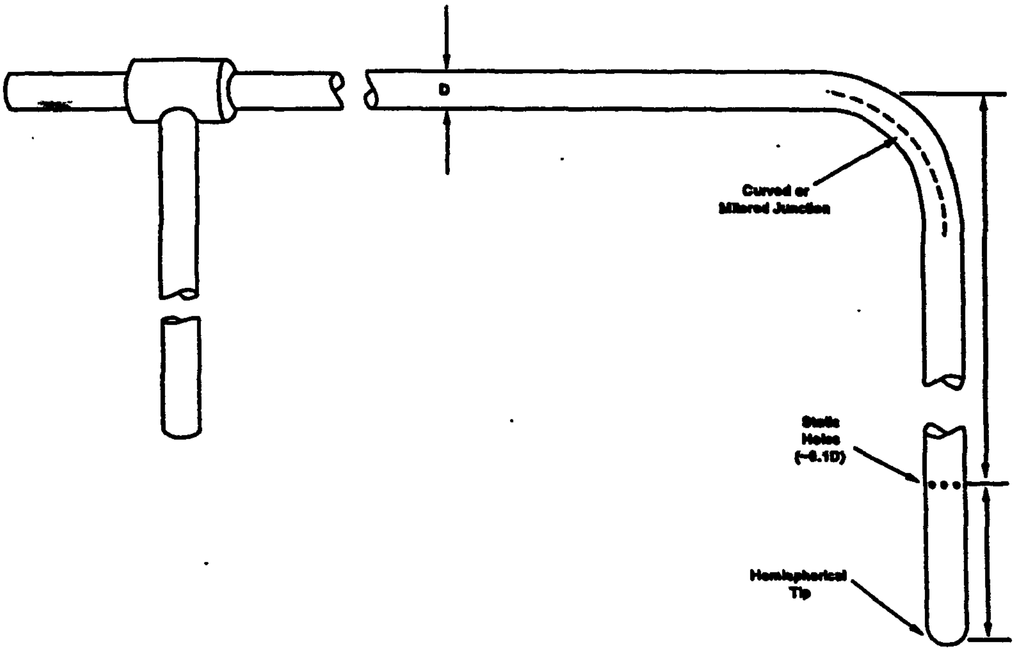


Figure 2-4. Standard pitot tube design specifications.

PLANT ______________________________
DATE ________ RUN NO. ______________STACK DIA. OR
DIMENSIONS, m (in.) _____ BAROMETRIC PRESS., mm Hg
(in. Hg) _____CROSS SECTIONAL AREA, $m^2$ ($ft^2$)________
OPERATORS __________________________
PITOT TUBE I.D. NO. ________________
AVG. COEFFICIENT, Cp = _____________
LAST DATE CALIBRATED _______________

SCHEMATIC OF STACK
CROSS SECTION

| Traverse Pt. No. | Vel. Hd., $\Delta p$ mm (in.) $H_2O$ | Stack Temperature | | $P_s$ mm Hg (in.Hg) | $(\Delta p)^{1/2}$ |
|---|---|---|---|---|---|
| | | $T_s$, °C (°F) | $T_s$, °K (°R) | | |
| | | | | | |
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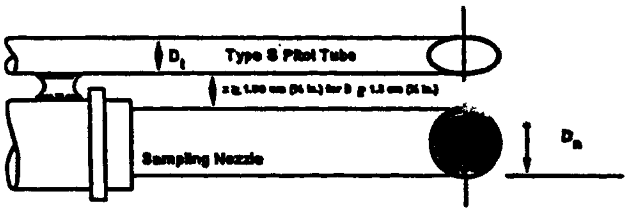
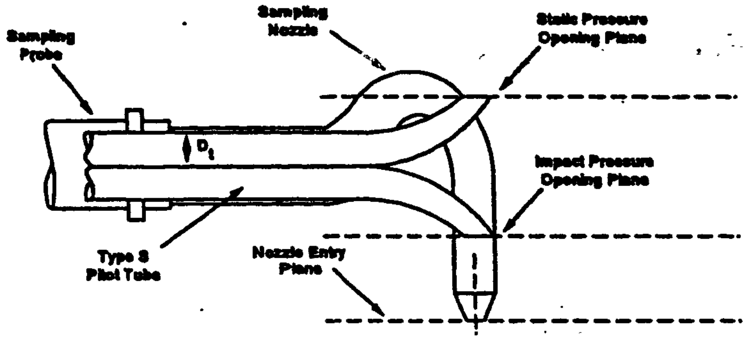
| | | | | | |
| | | Average | | | |

Figure 2-5. Velocity traverse data.



A. Bottom View; showing minimum pitot tube-nozzle separation.



B. Side View; to prevent pitot tube from interfering with gas flow streamlines approaching the nozzle, the static pressure opening plane of the pitot tube shall be even with or above the nozzle entry plane.

Figure 2-6. Proper pitot tube-sampling nozzle configuration to prevent aerodynamic interference; button-hook type nozzle; centers of nozzle and pitot opening aligned; $D_t$ between 0.48 and 0.95 cm (3/16 and 3/8 in.).

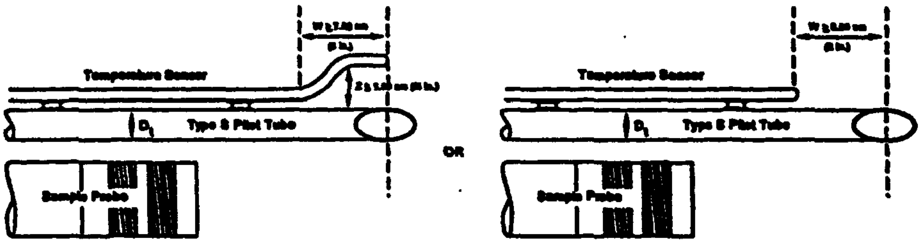


Figure 2-7. Proper thermocouple placement to prevent interference: $D_t$ between 0.48 and 0.95 cm (3/16 and 3/8 in.).

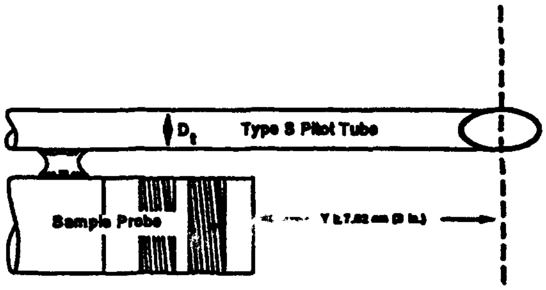


Figure 2-8. Minimum pitot-sample probe separation needed to prevent interference; $D_t$ between 0.48 and 0.95 cm (3/16 and 3/8 in.).

PITOT TUBE IDENTIFICATION NUMBER: ________ DATE: ____________CALIBRATED BY: ________________

| | "A" SIDE CALIBRATION | | | |
|---|---|---|---|---|
| RUN NO. | $\Delta P_{std}$ cm $H_2O$ (in $H_2O$) | $\Delta P_{(s)}$ cm $H_2O$ (in $H_2O$) | $C_{p(s)}$ | Deviation $C_{p(s)} - C_p(A)$ |
| 1 | | | | |
| 2 | | | | |
| 3 | | | | |
| | | $C_{p,avg}$ (SIDE A) | | |

| | "B" SIDE CALIBRATION | | | |
|---|---|---|---|---|
| RUN NO. | $\Delta P_{std}$ cm $H_2O$ (in $H_2O$) | $\Delta P_{(s)}$ cm $H_2O$ (in $H_2O$) | $C_{p(s)}$ | Deviation $C_{p(s)} - C_p(B)$ |
| 1 | | | | |
| 2 | | | | |
| 3 | | | | |
| | | $C_{p,avg}$ (SIDE B) | | |

$$\text{Average Deviation} = \sigma_{(A\,or\,B)} = \frac{\sum_{i=1}^{3} \left| C_{p(s)} - \bar{C}_{p(A\,or\,B)} \right|}{3} \leftarrow \text{Must Be} \le 0.01$$

$$\left| \bar{C}_p(\text{Side A}) - \bar{C}_p(\text{Side B}) \right| \leftarrow \text{Must Be} \le 0.01$$

Figure 2-9. Pitot tube calibration data.

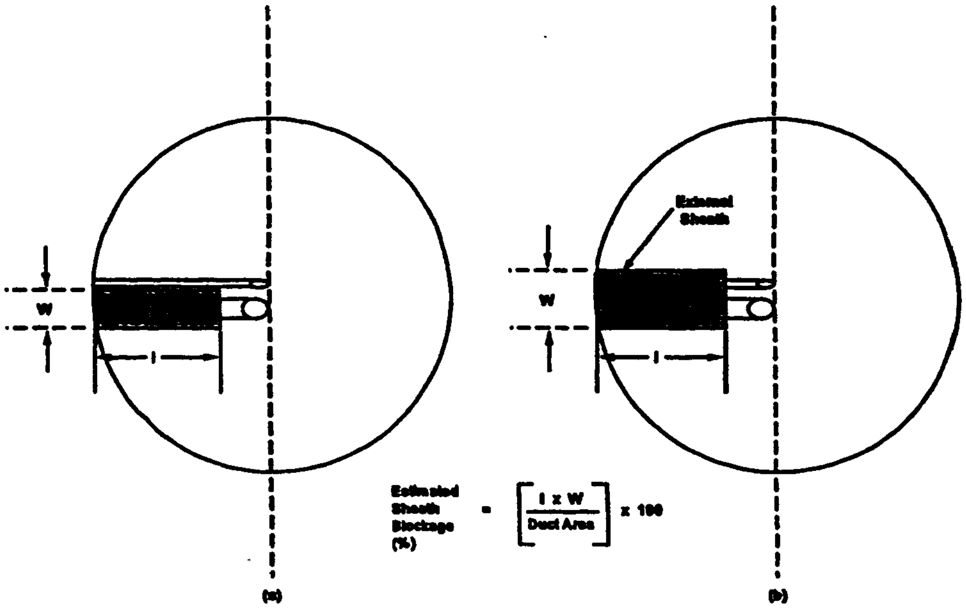


Figure 2-10. Projected-area models for typical pitot tube assemblies.

# Appendix G.3

## Sampling & Analysis Methods

## EPA Method 3A

EMISSION MEASUREMENT TECHNICAL INFORMATION CENTER
NSPS TEST METHOD

# Method 3A - Determination of Oxygen and Carbon Dioxide Concentrations in Emissions from Stationary Sources (Instrumental Analyzer Procedure)

## 1. APPLICABILITY AND PRINCIPLE

1.1 Applicability. This method is applicable to the determination of oxygen ($O_2$) and carbon dioxide ($CO_2$) concentrations in emissions from stationary sources only when specified within the regulations.

1.2 Principle. A sample is continuously extracted from the effluent stream: a portion of the sample stream is conveyed to an instrumental analyzer(s) for determination of $O_2$ and CQ concentration(s). Performance specifications and test procedures are provided to ensure reliable data.

## 2. RANGE AND SENSITIVITY

Same as in Method 6C, Sections 2.1 and 2.2, except that the span of the monitoring system shall be selected such that the average $O_2$ or $CO_2$ concentration is not less than 20 percent of the span.

## 3. DEFINITIONS

3.1 Measurement System. The total equipment required for the determination of the $O_2$ or $CO_2$ concentration. The measurement system consists of the same major subsystems as defined in Method 6C, Sections 3.1.1, 3.1.2, and 3.1.3.

3.2 Span, Calibration Gas, Analyzer Calibration Error, Sampling System Bias, Zero Drift, Calibration Drift, Response Time, and Calibration Curve. Same as in Method 6C, Sections 3.2 through 3.8, and 3.10.

3.3 Interference Response. The output response of the measurement system to a component in the sample gas, other than the gas component being measured.

## 4. MEASUREMENT SYSTEM PERFORMANCE SPECIFICATIONS

Same as in Method 6C, Sections 4.1 through 4.4.

## 5. APPARATUS AND REAGENTS

5.1 **Measurement System.** Any measurement system for $O_2$ or $CO_2$ that meets the specifications of this method. A schematic of an acceptable measurement system is shown in Figure 6C-1 of Method 6C. The essential components of the measurement system are described below:

5.1.1 **Sample Probe.** A leak-free probe of sufficient length to traverse the sample points.

5.1.2 **Sample Line.** Tubing to transport the sample gas from the probe to the moisture removal system. A heated sample line is not required for systems that measure the $O_2$ or $CO_2$ concentration on a dry basis, or transport dry gases.

5.1.3 **Sample Transport Line, Calibration Valve Assembly, Moisture Removal System, Particulate Filter, Sample Pump, Sample Flow Rate Control, Sample Gas Manifold, and Data Recorder.** Same as in Method 6C, Sections 5.1.3 through 5.1.9, and 5.1.11, except that the requirements to use stainless steel, Teflon, and nonreactive glass filters do not apply.

5.1.4 **Gas Analyzer.** An analyzer to determine continuously the $O_2$ or $CO_2$ concentration in the sample gas stream. The analyzer must meet the applicable performance specifications of Section 4. A means of controlling the [illegible] flow rate and a device for determining proper sample flow rate (e.g., precision rotameter, pressure gauge downstream of all flow controls, etc.) shall be provided at the analyzer. The requirements for measuring and controlling the analyzer for measuring and controlling the analyzer flow rate are not applicable if data are presented that demonstrate the analyzer is insensitive to flow variations over the range encountered during the test.

5.2 **Calibration Gases.** The calibration gases for $CO_2$ analyzers shall be $CO_2$ in $N_2$ or $CO_2$ in air. Alternatively, $CO_2/SO_2$, $O_2/SO_2$, or $O_2/CO_2/SO_2$ gas mixtures in $N_2$ may be used. Three calibration gases, as specified in Sections 5.3.1 through 5.3.4 of Method 6C, shall be used. For $O_2$ monitors that cannot analyze zero gas, a calibration gas concentration equivalent to less than 10 percent of the span may be used in place of zero gas.

## 6. MEASUREMENT SYSTEM PERFORMANCE TEST PROCEDURES

Perform the following procedures before measurement of emissions (Section 7).

6.1 **Calibration Concentration Verification.** Follow Section 6.1 of Method 6C, except if calibration gas analysis is required, use Method 3 and change the acceptance criteria for agreement among Method 3 results to 5 percent (or 0.2 percent by volume, whichever is greater).

6.2 **Interference Response.** Conduct an interference response test of the analyzer prior to its initial use in the field. Thereafter, recheck the measurement system if changes are made in the instrumentation that could alter the interference response (e.g., changes in the type of gas detector). Conduct the interference response in accordance with Section 5.4 of Method 20.

**6.3 Measurement System Preparation, Analyzer Calibration Error, Response Time, and Sampling System Bias Check.** Follow Sections 6.2 through 6.4 of Method 6C.

## 7. EMISSION TEST PROCEDURE

**7.1 Selection of Sampling Site and Sampling Points.** Select a measurement site and sampling points using the same criteria that are applicable to tests performed using Method 3.

**7.2 Sample Collection.** Position the sampling probe at the first measurement point, and begin sampling at the same rate as that used during the response time test. Maintain constant rate sampling (i.e., ±10 percent) during the entire run. The sampling time per run shall be the same as for tests conducted using Method 3 plus twice the average system response time. For each run, use only those measurements obtained after twice the response time of the measurement system has elapsed to determine the average effluent concentration.

**7.3 Zero and Calibration Drift Test.** Follow Section 7.4 of Method 6C.

## 8. QUALITY CONTROL PROCEDURES

The following quality control procedures are recommended when the results of this method are used for an emission rate correction factor, or excess air determination. The tester should select one of the following options for validating measurement results:

8.1 If both $O_2$ and CQ are measured using Method 3A, the procedures described in Section 4.4 of Method 3 should be followed to validate the $O_2$ and CQ measurement results.

8.2 If only $O_2$ is measured using Method 3A, measurements of the sample stream $_2$CO concentration should be obtained at the sample by-pass vent discharge using an Orsat or Fyrite analyzer, or equivalent. Duplicate samples should be obtained concurrent with at least one run. Average the duplicate Orsat or Fyrite analysis results for each run. Use the average $CO_2$ values for comparison with the$_2$O measurements in accordance with the procedures described in Section 4.4 of Method 3.

8.3 If only $CO_2$ is measured using Method 3A, concurrent measurements of the sample stream $CO_2$ concentration should be obtained using an Orsat or Fyrite analyzer as described in Section 8.2. For each run, differences greater than 0.5 percent between the Method 3A results and the average of the duplicate Fyrite analysis should be investigated.

## 9. EMISSION CALCULATION

9.1 For all $CO_2$ analyzers, and for $O_2$ analyzers that can be calibrated with zero gas, follow Section 8 of Method 6C, except express all concentrations as percent, rather than ppm.

9.2 For $O_2$ analyzers that use a low-level calibration gas in place of a zero gas, calculate the effluent gas concentration using Equation 3A-1.

$$C_{gas} = \frac{C_{ma} - C_{oa}}{C_m - C_o} (\overline{C} - C_m) + C_{ma} \qquad \text{Eq. 3A-1}$$

Where:

$C_{gas}$ = Effluent gas concentration, dry basis, percent.

$C_{ma}$ = Actual concentration of the upscale calibration gas, percent.

$C_{oa}$ = Actual concentration of the low-level calibration gas, percent.

$C_m$ = Average of initial and final system calibration bias check responses for the upscale calibration gas, percent.

$C_o$ = Average of initial and final system calibration bias check responses for the low level gas, percent.

$\overline{C}$ = Average gas concentration indicated by the gas analyzer, dry basis, percent.

## 10. BIBLIOGRAPHY

Same as in Bibliography of Method 6C.

# Appendix G.4

# Sampling & Analysis Methods

# EPA Method 23

6560-50

ENVIRONMENTAL PROTECTION AGENCY
40 CFR Part 60

[AD-FRL- ]
STANDARDS OF PERFORMANCE FOR NEW STATIONARY SOURCES
Appendix A , Test Method 23

AGENCY: Environmental Protection Agency (EPA).

ACTION: Proposed Rule.

SUMMARY: This rule amends Method 23, entitled "Determination of Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans from Stationary Sources," to correct existing errors in the method, to eliminate the methylene chloride rinse of the sampling train, and to clarify the quality assurance requirements of the method.

DATES: Comments. Comments must be received on or before ________________ (90 days after publication in the FEDERAL REGISTER].

Public Hearing. If anyone contacts EPA requesting to speak at a public hearing by ______________ (two weeks after publication in the FEDERAL REGISTER), a public hearing will be held on ______________ (four weeks after publication in the FEDERAL REGISTER), beginning at 10:00 a.m. Persons interested in attending the hearing should call Ms. Lala Cheek at (919) 541-5545 to verify that a hearing will be held.

Request to Speak at Hearing. Persons wishing to present oral testimony must contact EPA by ________________ (two weeks after publication in the FEDERAL REGISTER).

ADDRESSES: <u>Comments.</u> Comments should be submitted (in duplicate if possible) to Public Docket No. A-94-2 at the following address: U. S. Environmental Protection Agency , Air and Radiation Docket and Information Center, Mail Code: 6102, 401 M Street, SW, Washington, DC 20460. The Agency requests that a separate copy also be sent to the contact person listed below. The docket is located at the above address in Room M-1500 Waterside Mall (ground floor), and may be inspected from 8:30 a.m. to Noon and 1:00 to 3:00 PM, Monday through Friday. The proposed regulatory text and other materials related to this rulemaking are available for review in the docket or copies may be mailed on request from the Air Docket by calling <u>202-260-7548</u>. A reasonable fee may be charged for copying docket materials.

<u>Public Hearing.</u> If anyone contacts EPA requesting a public hearing, it will be held at EPA's Emission Measurement Laboratory, Research Triangle Park, North Carolina. Persons interested in attending the hearing or wishing to present oral testimony should notify Ms. Lala Cheek (MD-19), U.S. Environmental Protection Agency, Research Triangle Park, North Carolina 27711, telephone number (919) 541-5545.

Docket: A Docket, A-94-22, containing materials relevant to this rulemaking, is available for public inspection and copying between 8:30 a.m. and Noon and 1:00 and 3:00 p.m., Monday through Friday, in at EPA's Air Docket Section (LE-131), Room M-1500 Waterside Mall (ground floor) 401 M Street, S.W., Washington, D.C. 20460. A reasonable fee may be charged for copying.

FOR FURTHER INFORMATION CONTACT: Gary McAlister, Emission Measurement Branch (MD-19), Emissions, Monitoring, and Analysis Division, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina 27711, telephone (919) 541-1062.

SUPPLEMENTARY INFORMATION:

The proposed regulatory text of the proposed rule is not included in this Federal Register notice, but is available in Docket No. A-94-22 or by written or telephone request from the Air Docket (see ADDRESSES). If necessary, a limited number of copies of the Regulatory Text are available from the EPA contact persons designated earlier in this notice. This Notice with the proposed regulatory language is also available on the Technology Transfer Network (TTN), one of EPA's electronic bulletin boards. TTN provides information and technology exchange in various areas of air pollution control. The service is free except for the cost of the phone call. Dial (919) 541-5742 for up to a 14400 bps modem. If more information on TTN is needed, call the HELP line at (919) 541-5384.

## I. SUMMARY

Method 23 was promulgated along with the New Source Performance Standard for municipal waste combustors (Subpart Ea). As promulgated, the method contained some errors. This action would correct those errors and would clarify some of the existing quality assurance requirement In addition, the current procedure requires rinsing of the sampling train with two separate solvents which must be analyzed separately. Based on data the Agency has collected since promulgation of Method 23, we believe that one of these rinse steps and the resulting sample fraction can be eliminated. This could save as much as $2000 per test run in analytical costs.

## II. THE RULEMAKING

This rulemaking does not impose emission measurement requirements beyond those specified in the current regulations nor does it change any emission standard. Rather, the rulemaking would simply amend an existing test method associated with emission measurement requirements in the current regulations that would apply irrespective of this rulemaking.

## III. ADMINISTRATIVE REQUIREMENTS

## A. Public Hearing

A public hearing will be held, if requested, to discuss the proposed amendment in accordance with section 307(d)(5)of the Clean Air Act. Persons wishing to make oral presentations should contact EPA at the address given in the ADDRESSES section of this preamble. Oral presentations will be limited to 15 minutes each. Any member of the public may file a written statement with EPA before, during, or within 30 days after the hearing. Written statements should be addressed to the Air Docket Section address given in the ADDRESSES section of this preamble.

A verbatim transcript of the hearing and written statements will be available for public inspection and copying during normal working hours at EPA's Air Docket Section in Washington, DC (see ADDRESSES section of this preamble).

## B. Docket

The docket is an organized and complete file of all the information considered by EPA in the development of this rulemaking. The docket is a dynamic file, since material is added throughout the rulemaking development. The docketing system is intended to allow members of the public and industries involved to identify and locate documents readily so that they may effectively participate in the rulemaking process. Along with the statement of basis and purpose of the proposed and promulgated test method revisions and EPA responses to significant comments, the contents of the docket, except for interagency review materials, will serve as the record in case of judicial review [Section 307(d)(7)(A)].

## C. Executive Order 12291 Review

Under Executive Order 12291, EPA is required to judge whether a regulation is a "major rule" and, therefore, subject to the requirements of a regulatory impact analysis. This rulemaking does not impose emission measurement requirements beyond those specified in the current regulations, nor does it change any emission standard. The Agency has determined that this regulation would result in none of the adverse economic effects set forth in Section 1 of the Order as grounds for finding the regulation to be a "major rule." The Agency has, therefore, concluded that this regulation is not a "major rule" under Executive Order 12291.

## D. Regulatory Flexibility Act

The Regulatory Flexibility Act (RFA) of 1980 requires the identification of potentially adverse impacts of Federal regulations upon small business entities. The RFA specifically requires the completion of an analysis in those instances where small business impacts are possible. This rulemaking does not impose emission measurement requirements beyond those specified in the current regulations, nor does it change any emission standard. Because this rulemaking imposes no adverse economic impacts, an analysis has not been conducted.

Pursuant to the provision of 5 U.S.C. 605(b), I hereby certify that the promulgated rule will not have an impact on small entities because no additional costs will be incurred.

## E. Paperwork Reduction Act

This rule does not change any information collection requirements subject to Office of Management and Budget review under the Paperwork Reduction Act of 1980, 44 U.S.C. 3501 et seq.

## F. Statutory Authority

The statutory authority for this proposal is provided by sections 111 and 301(a) of the Clean Air Act, as amended: 42 U.S.C., 7411 and 7601(a).

LIST OF SUBJECTS

Air pollution control, municipal waste combustors, polychorinated dibenzo-p-dioxins, sources.

Date The Administrator

It is proposed that 40 CFR Part 60 be amended as follows:

1. The authority citation for Part 60 continues to read as follows: Authority: Clean Air Act (42 U.S.C. 7401 [et seq.], as amended by Pub. L 101-549).

2. Replace test Method 23 of Appendix A, with the following:

## Method 23 - Determination of Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans from Municipal Waste Combustors

### 1. APPLICABILITY AND PRINCIPLE

**1.1 Applicability.** This method is applicable to the determination of emissions of polychlorinated dibenzo-p-dioxins (PCDD's) and polychlorinated dibenzofurans (PCDF's) from municipal waste combustors. Calibration standards are selected for regulated emission levels for municipal waste combustors.

**1.2 Principle.** A sample is withdrawn isokinetically from the gas stream and collected in the sample probe, on a glass fiber filter, and on a packed column of adsorbent material. The sample cannot be separated into a particle and vapor fraction. The PCDD's and PCDF's are extracted from the sample, separated by high resolution gas chromatography (HRGC), and measured by high resolution mass spectrometry (HRMS).

## 2. APPARATUS

**2.1 Sampling.** A schematic of the sampling train is shown in Figure 23-1. Sealing greases shall not be used in assembling the train. The train is identical to that described in Section 2.1 of Method 5 of this appendix with the following additions:

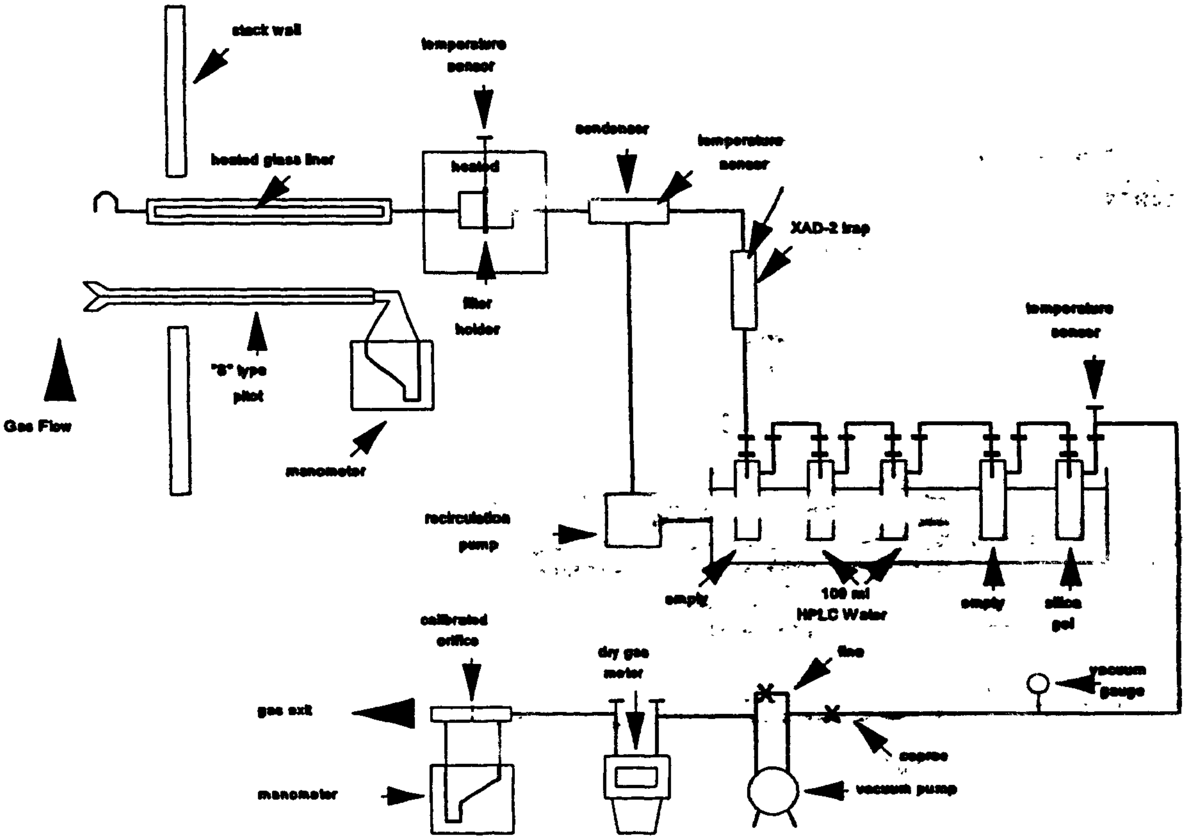


Figure 23.1 Sampling Train

**2.1.1 Nozzle.** The nozzle shall be made of nickel, nickel-plated stainless steel, quartz, or borosilicate glass.

**2.1.2 Sample Transfer Lines.** The sample transfer lines, if needed, shall be heat traced, heavy walled TFE (1/2 in. OD with 1/8 in. wall) with connecting fittings that are capable of forming leak-free, vacuum-tight connections without using sealing greases. The line shall be as short as possible and must be maintained at ≥120°C.

**2.1.1 Filter Support.** Teflon or Teflon-coated wire.

**2.1.2 Condenser.** Glass, coil type with compatible fittings. A schematic diagram is shown in Figure 23-2.

**2.1.3 Water Bath.** Thermostatically controlled to maintain the gas temperature exiting the condenser at ≤20°C (68°F).

**2.1.4 Adsorbent Module.** Glass container to hold up to 40 grams of resin adsorbent. A schematic diagram is shown in Figure 23-2. Other physical configurations of the water-jacketed resin trap/condenser assembly are acceptable. The connecting fittings shall form leak-free, vacuum tight seals. A coarse glass frit is included to retain the adsorbent in the water-jacketed sorbent module.

**2.1.5 Probe Liner.** The probe liner shall be made of glass and a Teflon ferrule or Teflon coated O-ring shall be used to make the seal at the nozzle end of the probe.

### 2.2 Sample Recovery.

**2.2.1 Fitting Caps.** Ground glass, Teflon tape, or aluminum foil (Section 2.2.6) to cap off the sample exposed sections of the train and sorbent module.

**2.2.2 Wash Bottles.** Teflon, 500-mL.

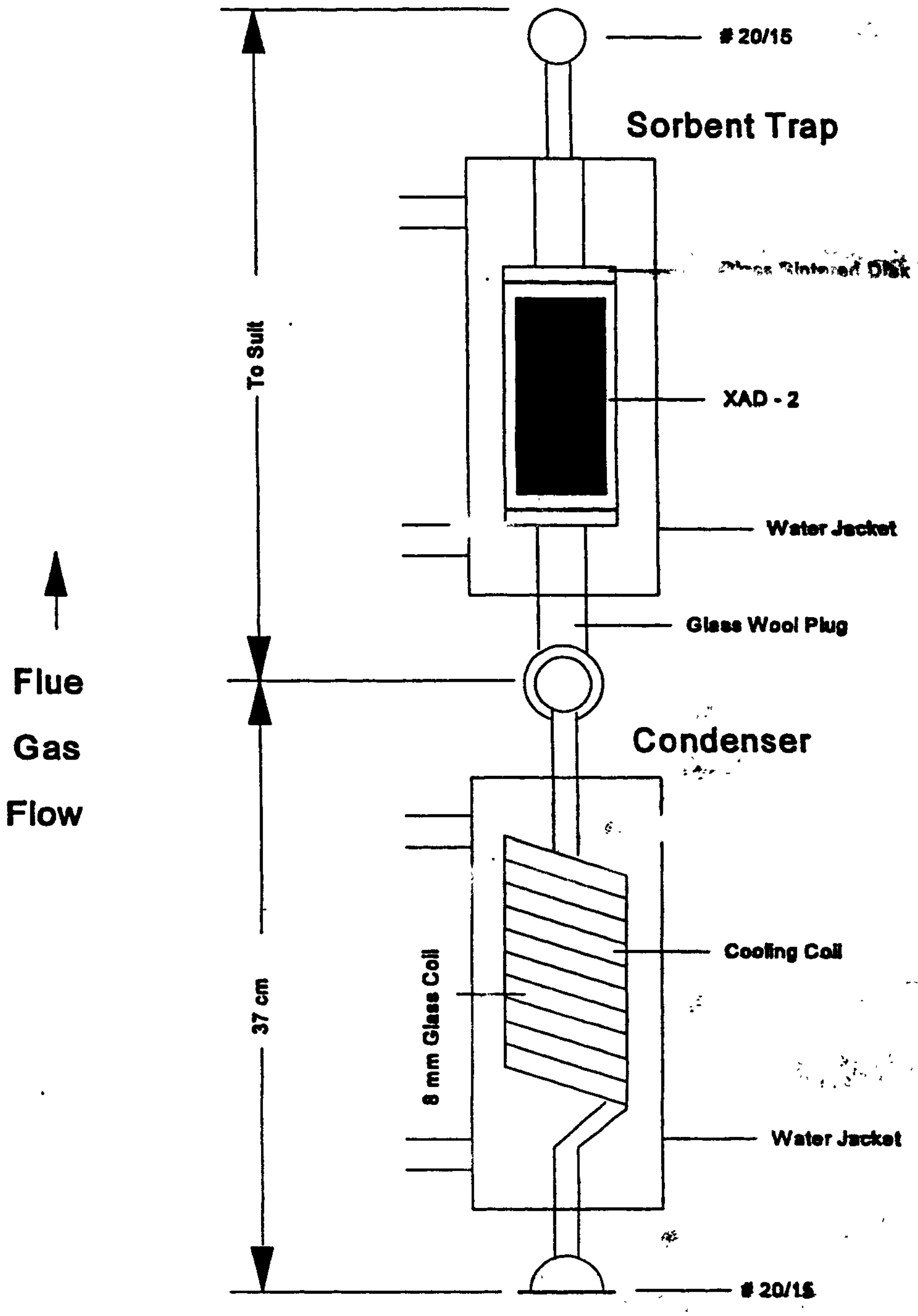


Figure 23.2 Condenser and Adsorbent Trap

**2.2.3 Probe Liner, Probe Nozzle, and Filter Holder Brushes.** Inert bristle brushes with precleaned stainless steel or Teflon handles. The probe brush shall have extensions of stainless steel or Teflon, at least as long as the probe. The brushes shall be properly sized and shaped to brush out the nozzle, probe liner, and transfer line, if used.

**2.2.4 Filter Storage Container.** Sealed filter holder, wide-mouth amber glass jar with Teflon-lined cap, glass petri dish, or Teflon baggie.

**2.2.5 Balance.** Triple beam.

**2.2.6 Aluminum Foil.** Heavy duty, hexane-rinsed (Do not use to wrap or ship filter samples, because it may react with particulate matter).

**2.2.7 Metal Storage Container.** Air tight container to store silica gel.

**2.2.8 Graduated Cylinder.** Glass, 250-mL with 2-mL graduations.

**2.2.9 Glass Sample Storage Containers.** Amber glass bottles for sample glassware washes, 500- or 1000-mL, with leak free Teflon-lined caps.

**2.3 Analysis.**

**2.3.1 Sample Containers.** 125- and 250-mL flint glass bottles with Teflon-lined caps.

2.3.2 **Test Tubes.** Glass.

2.3.3 **Soxhlet Extraction Apparatus.** Capable of holding 43 x 123 mm extraction thimbles.

2.3.4 **Extraction Thimble.** Glass, precleaned cellulosic, or glass fiber.

2.3.5 **Pasteur Pipettes.** For preparing liquid chromatographic columns.

2.3.6 **Reacti-vials.** Amber glass, 2-mL.

2.3.7 **Rotary Evaporator.** Buchi/Brinkman RF-121 or equivalent.

2.3.8 **Kuderna-Danish Concentrator Apparatus.**

2.3.9 **Nitrogen Evaporative Concentrator.** N-Evap Analytical Evaporator Model III or equivalent.

2.3.10 **Separatory Funnels.** Glass, 2-liter.

2.3.11 **Gas Chromatograph.** Consisting of the following components:

2.3.11.1 **Oven.** Capable of maintaining the separation column at the proper operating temperature ±10°C and performing programmed increases in temperature at rates of at least 40°C/min.

2.3.11.2 **Temperature Gauges.** To monitor column oven, detector, and exhaust temperatures ±1°C.

2.3.11.3 **Flow Systems.** Gas metering system to measure sample, fuel, combustion gas, and carrier gas flows.

**2.3.11.4 Capillary Columns.** A fused silica column, 60 x 0.25 mm inside diameter (ID), coated with DB-5 and a fused silica column, 30 m x 0.25 mm ID coated with DB-225. Other column systems may be substituted provided that the user is able to demonstrate, using calibration and performance checks, that the column system is able to meet the specifications of Section 6.1.2.2.

**2.3.12 Mass Spectrometer.** Capable of routine operation at a resolution of 1:10000 with a stability of ±5 ppm.

**2.3.13 Data System.** Compatible with the mass spectrometer and capable of monitoring at least five groups of 25 ions.

**2.3.14 Analytical Balance.** To measure within 0.1 mg.

## 3. REAGENTS

### 3.1 Sampling.

**3.1.1 Filters.** Glass fiber filters, without organic binder, exhibiting at least 99.95 percent efficiency (<0.05 percent penetration) on 0.3-micron dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM Standard Method D 2986-71 (Reapproved 1978) (incorporated by reference - see §60.17).

**3.1.1.1 Precleaning.** All filters shall be cleaned before their initial use. Place a glass extraction thimble and 1 g of silica gel and a plug of glass wool into a Soxhlet apparatus, charge the apparatus with toluene, and reflux for a minimum of 3 hours. Remove the toluene and discard it, but retain the silica gel. Place no more than 50 filters in the thimble onto the silica gel bed and top with the cleaned glass wool. Charge the Soxhlet with toluene and reflux for 16 hours. After extraction, allow the Soxhlet to cool, remove the filters, and dry them under a clean nitrogen ($N_2$) stream. Store the filters in a glass petri dishes and seal with Teflon tape.

**3.1.2 Adsorbent Resin.** Amberlite XAD-2 resin. Thoroughly cleaned before initial use. Do not reuse resin. If precleaned XAD-2 resin is purchased from the manufacturer, the cleaning procedure described in Section 3.1.2.1 is not required.

**3.1.2.1 Cleaning.** Procedure may be carried out in a giant Soxhlet extractor. An all-glass filter thimble containing an extra-coarse frit is used for extraction of XAD-2. The frit is recessed 10-15 mm above a crenelated ring at the bottom of the thimble to facilitate drainage. The resin must be carefully retained in the extractor cup with a glass wool plug and a stainless steel ring because it floats on methylene chloride. This process involves sequential extraction in the following order.

| Solvent | Procedure |
|---|---|
| Water | Initial Rinse: Place resin in a beaker, |

| | |
|---|---|
| | rinse once with HPLC water, and discard water. Refill beaker with water, let stand overnight, and discard water. |
| Water | Extract with HPLC water for 8 hours. |
| Methanol | Extract with methanol for 22 hours. |
| Methylene Chloride | Extract with methylene chloride for 22 hours. |
| Methylene Chloride | Extract with methylene chloride for 22 hours. |

#### 3.1.2.2 Drying.

**3.1.2.2.1 Drying Column.** Pyrex pipe, 10.2 cm ID by 0.6 m long, with suitable retainers.

**3.1.2.2.2 Procedure.** The adsorbent must be dried with clean inert gas. Liquid nitrogen from a standard commercial liquid nitrogen cylinder has proven to be a reliable source for large volumes of gas free from organic contaminants. Connect the liquid nitrogen cylinder to the column by a length of cleaned copper tubing, 0.95 cm ID, coiled to pass through a heat source. A convenient heat source is a water-bath heated from a steam line. The final nitrogen temperature should only be warm to the touch and not over 40°C. Continue flowing nitrogen through the adsorbent until all the residual solvent is removed. The flow rate should be sufficient to gently agitate the particles, but not so excessive as to cause the particles to fracture.

**3.1.2.3 Quality Control Check.** The adsorbent must be checked for residual methylene chloride ($MeCl_2$) as well as PCDDs and PCDFs prior to use. The analyst may opt to omit this check for precleaned XAD-2.

**3.1.2.3.1 $MeCl_2$ Residue Extraction.** Weigh a 1.0 g sample of dried resin into a small vial, add 3 mL of toluene, cap the vial, and shake it well.

**3.1.2.3.2 $MeCl_2$ Residue Analysis.** Inject a 2 $\mu$l sample of the extract into a gas chromatograph operated under the following conditions:

Column: 6 ft x 1/8 in stainless steel containing 10 percent OV-101™ on 100/120 Supelcoport.

Carrier Gas: Helium at a rate of 30 mL/min.

Detector: Flame ionization detector operated at a sensitivity of $4 \times 10^{-11}$ A/mV.

Injection Port Temperature: 250°C.

Detector Temperature: 305°C.

Oven Temperature: 30°C for 4 min; programmed to rise at 40°C/min until it reaches 250°C; return to 30°C after 17 minutes.

Compare the results of the analysis to the results from the reference solution. Prepare the reference solution by injecting

4.0 $\mu$l of methylene chloride into 100 mL of toluene. This corresponds to 100 $\mu$g of methylene chloride per g of adsorbent. The maximum acceptable concentration is 1000 $\mu$g/g of adsorbent. If the adsorbent exceeds this level, drying must be continued until the excess methylene chloride is removed.

**3.1.2.3.3 PCDD and PCDF Check.** Extract the adsorbent sample as described in Section 5.1. Analyze the extract as described in Section 5.3. If any of the PCDDs or PCDFs (tetra through hexa) are present at concentrations above the target detection limits (TDLs), the adsorbent must be recleaned by repeating the last step of the cleaning procedure. The TDLs for the various PCDD/PCDF congeners are listed in Table 1.

**3.1.2.4 Storage.** After cleaning, the adsorbent may be stored in a wide mouth amber glass container with a Teflon-lined cap or placed in glass adsorbent modules tightly sealed with glass stoppers. It must be used within 4 weeks of cleaning. If precleaned adsorbent is purchased in sealed containers, it must be used within 4 weeks after the seal is broken.

**3.1.3 Glass Wool.** Cleaned by sequential immersion in three aliquots of methylene chloride, dried in a 110°C oven, and stored in a methylene chloride-washed glass container with a Teflon-lined screw cap.

**3.1.4 Water.** Deionized distilled and stored in a methylene chloride-rinsed glass container with a Teflon-lined screw cap.

3.1.5 **Silica Gel.** Indicating type, 6 to 16 mesh. If previously used, dry at 175° C (350°F) for two hours. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used, subject to the approval of the Administrator.

3.1.6 **Chromic Acid Cleaning Solution.** Dissolve 20 g of sodium dichromate in 15 mL of water, and then carefully add 400 mL of concentrated sulfuric acid.

3.1.7 **HPLC Water.**

3.2 **Sample Recovery.**

3.2.1 **Acetone.** Pesticide quality.

3.2.2 **Toluene.** Pesticide quality.

3.3 **Analysis.**

3.3.1 **Potassium Hydroxide.** ACS grade, 2-percent (weight/volume) in water.

3.3.2 **Sodium Sulfate.** Granulated, reagent grade. Purify prior to use by rinsing with methylene chloride and oven drying. Store the cleaned material in a glass container with a Teflon-lined screw cap.

3.3.3 **Sulfuric Acid.** Reagent grade.

3.3.4 **Sodium Hydroxide.** 1.0 N. Weigh 40 g of sodium hydroxide into a 1-liter volumetric flask. Dilute to 1 liter with water.

3.3.5 **Hexane.** Pesticide grade.

3.3.6 **Methylene Chloride.** Pesticide grade.

3.3.7 **Benzene.** Pesticide grade.

3.3.8 **Ethyl Acetate.**

3.3.9 **Methanol.** Pesticide grade.

3.3.10 **Toluene.** Pesticide grade.

3.3.11 **Nonane.** Pesticide grade.

3.3.12 **Cyclohexane.** Pesticide Grade.

3.3.13 **Basic Alumina.** Activity grade 1, 100-200 mesh. Prior to use, activate the alumina by heating for 16 hours at 130°C. Store in a desiccator. Pre-activated alumina may be purchased from a supplier and may be used as received.

3.3.14 **Silica Gel.** Bio-Sil A, 100-200 mesh. Prior to use, activate the silica gel by heating for at least 30 minutes at 180°C. After cooling, rinse the silica gel sequentially with methanol and methylene chloride. Heat the rinsed silica gel at 50°C for 10 minutes, then increase the temperature gradually to 180°C over 25 minutes and maintain it at this temperature for 90 minutes. Cool at room temperature and store in a glass container with a Teflon-lined screw cap.

3.3.15 **Silica Gel Impregnated with Sulfuric Acid.** Combine 100 g of silica gel with 44 g of concentrated sulfuric acid in a screw capped glass bottle and agitate thoroughly. Disperse the solids with a stirring rod until a uniform mixture is obtained. Store the mixture in a glass container with a Teflon-lined screw cap.

3.3.16 **Silica Gel Impregnated with Sodium Hydroxide.** Combine 39 g of 1 N sodium hydroxide with 100 g of silica gel in a screw capped glass bottle and agitate thoroughly. Disperse solids with a stirring rod until a uniform mixture is obtained. Store the mixture in glass container with a Teflon-lined screw cap.

3.3.17 **Carbon/Celite.** Combine 10.7 g of AX-21 carbon with 124 g of Celite 545 in a 250-mL glass bottle with a Teflon-lined screw cap. Agitate the mixture thoroughly until a uniform mixture is obtained. Store in the glass container.

3.3.18 **Nitrogen.** Ultra high purity.

3.3.19 **Hydrogen.** Ultra high purity.

3.3.20 **Internal Standard Solution.** Prepare a stock standard solution containing the isotopically labelled PCDD's and PCDF's at the concentrations shown in Table 2 under the heading "Internal Standards" in 10 mL of nonane.

3.3.21 **Surrogate Standard Solution.** Prepare a stock standard solution containing the isotopically labelled PCDD's and PCDF's at the concentrations shown in Table 2 under the heading "Surrogate Standards" in 10 mL of nonane.

3.3.22 **Recovery Standard Solution.** Prepare a stock standard solution containing the isotopically labelled PCDD's and PCDF's at the concentrations shown in Table 2 under the heading "Recovery Standards" in 10 mL of nonane.

## 4. PROCEDURE

**4.1 Sampling.** The complexity of this method is such that, in order to obtain reliable results, testers and analysts should be trained and experienced with the procedures.

**4.1.1 Pretest Preparation.**

**4.1.1.1 Cleaning Glassware.** All glass components of the train upstream of and including the adsorbent module, shall be cleaned as described in Section 3A of the "Manual of Analytical Methods for the Analysis of Pesticides in Human and Environmental Samples." Special care shall be devoted to the removal of residual silicone grease sealants on ground glass connections of used glassware. Any residue shall be removed by soaking the glassware for several hours in a chromic acid cleaning solution prior to cleaning as described above.

**4.1.1.2 Adsorbent Trap.** The traps shall be loaded in a clean area to avoid contamination. They may not be loaded in the field. Fill a trap with 20 to 40 g of XAD-2. Follow the XAD-2 with glass wool and tightly cap both ends of the trap. Add 40 $\mu$l of the surrogate standard solution (Section 3.3.21) to each trap for a sample that will be split prior to analysis or 20 $\mu$l of the surrogate standard solution (Section 3.3.21) to each trap for samples that will not be split for analysis (Section 5.1). After addition of the surrogate standard solution, the trap must be used within 14 days. Keep the spiked sorbent under refrigeration until use.

4.1.1.3 **Sampling Train.** It is suggested that all components be maintained according to the procedure described in APTD-0576.

4.1.1.4 **Silica Gel.** Weigh several 200 to 300 g portions of silica gel in air tight containers to the nearest 0.5 g. Record the total weight of the silica gel plus container, on each container. As an alternative, the silica gel may be weighed directly in the fifth impinger just prior to sampling.

4.1.1.5 **Filter.** Check each filter against light for irregularities and flaws or pinhole leaks. Pack the filters flat in a clean glass container or Teflon baggie. Do not mark filter with ink or any other contaminating substance.

4.1.2 **Preliminary Determinations.** Same as Section 4.1.2 Method 5.

4.1.3 **Preparation of Sampling Train.**

4.1.3.1 During preparation and assembly of the sampling train, keep all train openings where contamination can enter, sealed until sampling is about to begin. Wrap sorbent module with aluminum foil to shield from radiant heat of sun light. (NOTE:

Do not use sealant grease in assembling the train.)

4.1.3.2 Place approximately 100 mL of water in the second and third impingers, leave the first and fourth impingers empty, and transfer approximately 200 to 300 g of preweighed silica gel from its container to the fifth impinger.

4.1.3.3 Place the silica gel container in a clean place for later use in the sample recovery. Alternatively, the weight of the silica gel plus the fifth impinger may be determined to the nearest 0.5 g and recorded.

4.1.3.4 Assemble the sampling train as shown in Figure 23-1.

4.1.3.5 Turn on the adsorbent module and condenser coil recirculating pump and begin monitoring the adsorbent module gas entry temperature. Ensure proper sorbent gas entry temperature before proceeding and before sampling is initiated. It is extremely important that the XAD-2 adsorbent resin temperature never exceed 50°C because thermal decomposition and breakthrough of surrogate standards will occur. During testing, the XAD-2 temperature must not exceed 20°C for efficient capture of the PCDD's and PCDF's.

4.1.4 **Leak-Check Procedure.** Same as Method 5, Section 4.1.4.

4.1.5 **Sampling Train Operation.** Same as Method 5, Section 4.1.5.

4.2 **Sample Recovery.** Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period. Seal the nozzle end of the sampling probe with Teflon tape or aluminum foil.

When the probe can be safely handled, wipe off all external particulate matter near the tip of the probe. Remove the probe from the train and close off both ends with aluminum foil. Seal off the inlet to the train with Teflon tape, a ground glass cap, or aluminum foil.

Transfer the probe and impinger assembly to the cleanup area. This area shall be clean and enclosed so that the chances of losing or contaminating the sample are minimized. Smoking, which could contaminate the sample, shall not be allowed in the cleanup area. Cleanup personnel shall wash their hands prior to sample recovery.

Inspect the train prior to and during disassembly and note any abnormal conditions, e.g., broken filters, colored impinger liquid, etc. Treat the samples as follows:

**4.2.1 Container No. 1.** Either seal the filter holder or carefully remove the filter from the filter holder and place it in its identified container. Do not place the filter in aluminum foil. Use a pair of cleaned tweezers to handle the filter. If it is necessary to fold the filter, do so such that the particulate cake is inside the fold. Carefully transfer to the container any particulate matter and filter fibers which adhere to the filter holder gasket, by using a dry inert bristle brush and a sharp-edged blade. Seal the container with Teflon tape.

**4.2.2 Adsorbent Module.** Remove the module from the train, tightly cap both ends, label it, and store it on ice for transport to the laboratory.

**4.2.3 Container No. 2.** Quantitatively recover material deposited in the nozzle, probe transfer lines, the front half of the filter holder, and the cyclone, if used, first, by brushing while rinsing three times with acetone and then, by rinsing the probe three times with toluene. Collect all the rinses in Container No. 2.

Rinse the back half of the filter holder three times with acetone. Rinse the connecting line between the filter and the condenser three times with acetone. Soak the connecting line with three separate portions of toluene for 5 minutes each. If using a separate condenser and adsorbent trap, rinse the condenser in the same manner as the connecting line. Collect all the rinses in Container No. 2 and mark the level of the liquid on the container.

**4.2.4 Impinger Water.** Measure the liquid in the first four impingers to within 1 mL by using a graduated cylinder or by weighing it to within 0.5 g by using a balance. Record the volume or weight of liquid present. This information is required to calculate the moisture content of the effluent gas. Discard the liquid after measuring and recording the volume or weight.

**4.2.5 Silica Gel.** Note the color of the indicating silica gel to determine if it has been completely spent and make a mention of its condition. Transfer the silica gel from the fifth impinger to its original container and seal.

## 5. ANALYSIS

All glassware shall be cleaned as described in Section 3A of the "Manual of Analytical Methods for the Analysis of Pesticides in Human and Environmental Samples." All samples must be extracted within 30 days of collection and analyzed within 45 days of extraction.

**5.1 Sample Extraction.** The analyst may choose to split the sample extract after the completion of sample extraction procedures. One half of the sample can then be archived. Sample preparation procedures are given for using the entire sample and for splitting the sample.

**5.1.1 Extraction System.** Place an extraction thimble (Section 2.3.4), 1 g of silica gel, and a plug of glass wool into the Soxhlet apparatus, charge the apparatus with toluene, and reflux for a minimum of 3 hours. Remove the toluene and discard it, but retain the silica gel. Remove the extraction thimble from the extraction system and place it in a glass beaker to catch the solvent rinses.

**5.1.2 Container No. 1 (Filter).** Transfer the contents directly to the glass thimble of the extraction system and extract them simultaneously with the XAD-2 resin.

**5.1.3 Adsorbent Cartridge.** Suspend the adsorbent module directly over the extraction thimble in the beaker (See Section 5.1.1). The glass frit of the module should be in the up position. Using a Teflon squeeze bottle containing toluene, flush the XAD-2 into the thimble onto the bed of cleaned silica gel. Thoroughly rinse the glass module catching the rinsings in the beaker containing the thimble. If the resin is wet, effective extraction can be accomplished by loosely packing the resin in the thimble. Add the XAD-2 glass wool plug to the thimble.

**5.1.4 Container No. 2 (Acetone and Toluene).** Concentrate the sample to a volume of about 1-2 mL using a Kuderna-Danish concentrator apparatus, followed by $N_2$ blow down at a temperature of less than 37°C. Rinse the sample container three times with small portions of methylene chloride and add these to the concentrated solution and concentrate further to near dryness. This residue contains particulate matter removed in the rinse of the sampling train probe and nozzle. Add the concentrate to the filter and the XAD-2 resin in the Soxhlet apparatus described in Section 5.1.1.

**5.1.5 Extraction.** For samples that are to be split prior to analysis add 40 µl of the internal standard solution (Section 3.3.20) to the extraction thimble containing the contents of the adsorbent cartridge, the contents of Container No. 1, and the concentrate from Section 5.1.4. Alternatively, 20 µl of the internal standard solution (Section 3.3.20) for samples that are not to be split prior to analysis. Cover the contents of the extraction thimble with the cleaned glass wool plug to prevent the XAD-2 resin from floating into the solvent reservoir of the extractor. Place the thimble in the extractor, and add the toluene contained in the beaker to the solvent reservoir. Add additional toluene to fill the reservoir approximately 2/3 full. Add Teflon boiling chips and assemble the apparatus. Adjust the heat source to cause the extractor to cycle three times per hour. Extract the sample for 16 hours. After extraction, allow the Soxhlet to cool. Transfer the toluene extract and three 10-mL rinses to the rotary evaporator. Concentrate the extract to approximately 10 mL. If decided to split the sample, store one half for future use, and analyze the other half according to the procedures in Sections 5.2 and 5.3. In either case, use a nitrogen evaporative concentrator to reduce the volume of the sample being analyzed to near dryness. Dissolve the residue in 5 mL of hexane.

### 5.2 Sample Cleanup and Fractionation.

The following sample cleanup and fractionation procedures are recommended. Alternative procedures may be utilized providing acceptable identification criteria (Section 5.3.2.5) and quantification criteria (Section 5.3.2.6) are met.

**5.2.1 Silica Gel Column.** Pack one end of a glass column, 20 mm x 230 mm, with glass wool. Add in sequence, 1 g silica gel, 2 g of sodium hydroxide impregnated silica gel, 1 g silica gel, 4 g of acid-modified silica gel, and 1 g of silica gel. Wash the column with 30 mL of hexane and discard. Add the sample extract, dissolved in 5 mL of hexane to the column with two additional 5-mL rinses. Elute the column with an additional 90 mL of hexane and retain the entire eluate. Concentrate this solution to a volume of about 1 mL using the nitrogen evaporative concentrator (Section 2.3.9).

**5.2.2 Basic Alumina Column.** Shorten a 25-mL disposable Pasteur pipette to about 16 mL. Pack the lower section with glass wool and 12 g of basic alumina. Transfer the concentrated extract from the silica gel column to the top of the basic alumina column and elute the column sequentially with 120 mL of 0.5 percent methylene chloride in hexane followed by 120 mL of 35 percent methylene chloride in hexane. Discard the first 120 mL of eluate. Collect the second 120 mL of eluate and concentrate it to about 0.5 mL using the nitrogen evaporative concentrator. Transfer this extract with hexane to "13 mL tubes".

**5.2.3 AX-21 Carbon/Celite 545 Column.** Remove the bottom 0.5 in. from the tip of a 2-mL disposable Pasteur pipette. Insert a glass fiber filter disk or glass wool plug in the top of the pipette 2.5 cm from the constriction. Add sufficient carbon/Celite™ mixture to form a 2 cm column (the 0.6 mL mark column. Top with a glass wool plug. In some cases AX-21 carbon fines may wash through the glass wool plug and enter the sample. This may be prevented by adding a celite plug to the exit end of the column. Pre-elute the column with 5 mL toluene, followed by 1 mL of a 50:50 methylene chloride/cyclohexane mixture, followed by 5 mL of hexane. Load in sequence, the sample extract in 1 mL hexane, 2x0.5 mL rinses in hexane, 2 mL of 50 percent methylene chloride in hexane and 2 mL of 50 percent benzene in ethyl acetate and discard the eluates. Invert the column and elute in the reverse direction with 13 mL of toluene. Collect this eluate. Concentrate the eluate in a nitrogen evaporator at 45°C to about 1 mL. Transfer the concentrate to a Reacti-vial using a toluene rinses and concentrate to near dryness (less than 20 $\mu$l) using a stream of $N_2$. Store extracts at room temperature, shielded from light, until the analysis is performed.

**5.3 Analysis.** Analyze the sample with a gas chromatograph coupled to a mass spectrometer (GC/MS) using the instrumental parameters in Sections 5.3.1 and 5.3.2. Immediately prior to analysis, add a 20 µl aliquot of the recovery standard solution from Table 2 to each sample. A 2 µl aliquot of the extract is injected into the GC. Sample extracts are first analyzed using the DB-5 capillary column to determine the concentration of each isomer of PCDD's and PCDF's (tetra-through octa-). If 2,3,7,8-TCDF is detected in this analysis, then analyze another aliquot of the sample in a separate run, using the DB-225 column to measure the 2,3,7,8 tetra-chloro dibenzofuran isomer. Other column systems may be used, provided that it can be demonstrated using calibration and performance checks that the column system is able to meet the specifications of Section 6.1.2.

**5.3.1 Gas Chromatograph Operating Conditions.** The recommended conditions are shown in Table 4.

**5.3.2 High Resolution Mass Spectrometer.**

**5.3.2.1 Resolution.** 10,000 resolving power or 100 ppm mass/mass.

**5.3.2.2 Ionization Mode.** Electron impact.

**5.3.2.3 Source Temperature 250°C.**

**5.3.2.4 Monitoring Mode.** Selected ion monitoring. A list of the various ions to be monitored is presented in Table 5.

**5.3.2.5 Identification Criteria.** The following identification criteria shall be used for the characterization of polychlorinated dibenzodioxins and dibenzofurans.

1. The integrated ion-abundance ratio (M/M+2 or M+2/M+4) shall be within 15 percent of the theoretical value. The acceptable ion-abundance ratio ranges (±15%) for the identification of chlorine-containing compounds are given in Table 6. If the ion-abundance ratio ranges are the outside those in Table 6, the source has the option of using the results if the concentration is determined using procedures in Section 9.3 or redoing the analysis to eliminate the unacceptable ion-abundance ratio.

2. The retention time for the analytes must be within 3 seconds of the corresponding $^{13}C$-labeled internal standard or surrogate standard.

3. The monitored ions, shown in Table 5 for a given analyte, shall reach their maximum within 2 seconds of each other.

4. The identification of specific isomers that do not have corresponding $^{13}C$-labeled standards is done by comparison of the relative retention time (RRT) of the analyte to the nearest internal standard retention time with reference (i.e., within 0.005 RRT units) to the comparable RRT's found in the continuing calibration.

5. The signal to noise ratio for all monitored ions must be greater than 2.5.

6. The confirmation of 2, 3, 7, 8-TCDF shall satisfy all of the above identification criteria.

7. Any PCDF coeluting (±2 s) with a peak in the corresponding PCDPE channel, of intensity 10% or greater compared to the analyte peak is evidence of a positive interference, the source may opt keep the value to calculate CDD/CDF concentration or conduct a complete reanalysis in an effort to remove or shift the interference. If a reanalysis is conducted, all values from the reanalyzed sample will be used for CDD/CDF concentration calculations.

8. Set the mass spectrometer lock channels as specified in Table 5. Monitor the quality control check channels specified in Table 5 to verify instrument stability during the analysis. If the signal varies by more than 25 percent from the average response, results for all isomers at corresponding residence time shall be invalid. The source has the options of conducting additional cleanup procedures on the other portion of the sample for split samples or diluting the original sample or following other procedures recommended by the Administrator. When a complete reanalysis is conducted, all concentration calculations shall be based on the reanalyzed sample.

**5.3.2.6 Quantification.** The peak areas for the two ions monitored for each analyte are summed to yield the total response for each analyte. Each internal standard is used to quantify the indigenous PCDD's or PCDF's in its homologous series. For example, the $^{13}C_{12}$-2,3,7,8-tetra chlorinated dibenzodioxin is used to calculate the concentrations of all other tetra chlorinated isomers. Recoveries of the tetra- and penta- internal standards are calculated using the $^{13}C_{12}$-1,2,3,4-TCDD. Recoveries of the hexa- through octa- internal standards are calculated using $^{13}C_{12}$-1,2,3,7,8,9-HxCDD. Recoveries of the surrogate standards are calculated using the corresponding homolog from the internal standard. When no peak is detected, the noise level, as measured by the intensity of the noise in a clear zone of the chromatogram, is used to calculate the detection limit. Tables 7, 8, and 9 summarize the quantification relationships for the unlabeled analytes, internal standards and surrogate standards, respectively.

## 6. CALIBRATION

Same as Method 5 with the following additions.

### 6.1 GC/MS System.

**6.1.1 Initial Calibration.** Calibrate the GC/MS system using the set of five standards shown in Table 3. The relative standard deviation for the mean response factor from each of the unlabeled analytes (Table 3) and of the internal and surrogate standards shall be less than or equal to the values in Table 6. The signal to noise ratio for the GC signal present in every selected ion current profile shall be greater than or equal to 10. The ion abundance ratios shall be within the control limits in Table 5.

### 6.1.2 Daily Performance Check.

**6.1.2.1 Calibration Check.** Inject 2 µl of solution Number 3 from Table 3. Calculate the relative response factor (RRF) for each compound and compare each RRF to the corresponding mean RRF obtained during the initial calibration. The analyzer performance is acceptable if the measured RRF's for the labeled and unlabeled compounds for the daily run are within the limits of the mean values shown in Table 10. In addition, the ion-abundance ratios shall be within the allowable control limits shown in Table 6.

**6.1.2.2 Column Separation Check.** Inject 2 µl of a solution of a mixture of PCDD's and PCDF's that documents resolution between 2,3,7,8-TCDD and other TCDD isomers. Resolution is defined as a valley between peaks that is less than 25 percent of the lower of the two peaks. Identify and record the retention time windows for each homologous series. Perform a similar resolution check on the confirmation column to document the resolution between

2,3,7,8 TCDF and other TCDF isomers.

**6.2 Lock Channels.** Set mass spectrometer lock channels as specified in Table 5. Monitor the quality control check channels specified in Table 5 to verify instrument stability during the analysis.

## 7. QUALITY CONTROL

**7.1 Sampling Train Collection Efficiency Check.** Add 40 µl of the surrogate standards in Table 2 for samples split for analysis or 20 µl of the surrogate standards for sample not split for analysis to the adsorbent cartridge of each train before collecting the field samples.

**7.2 Internal Standard Percent Recoveries.** A group of nine carbon-labeled PCDDs and PCDFs representing the tetra- through octachlorinated homologues, is added to every sample prior to extraction. The role of the internal standards is to quantify the native PCDD's and PCDF's present in the sample as well as to determine the overall method efficiency. Recoveries of the internal standards shall be between 40 to 130 percent for the tetra- through hexachlorinated compounds while the range is 25 to 130 percent for the hepta- and octachlorinated homologues.

**7.3 Surrogate Standard Recoveries.** The five surrogate compounds in Table 3 are added to the resin in the adsorbent sampling cartridge before the sample is collected. The surrogate recoveries are measured relative to the internal standards and are a measure of the sampling train collection efficiency. They are not used to measure the native PCDD's and PCDF's. All surrogate standard recoveries shall be between 70 and 130 percent. Poor recoveries for all the surrogates may be an indication of breakthrough in the sampling train. If the recovery of all standards is below 70 percent, the sampling runs must be repeated. As an alternative, the sampling runs do not have to be repeated if the final results are divided by the fraction of surrogate recovery (on a homolog group basis). Poor recoveries of isolated surrogate compounds should not be grounds for rejecting an entire set of samples.

7.4 **Toluene QA Rinse.** Report the results of the toluene QA rinse separately from the total sample catch. Do not add it to the total sample.

7.5 **Detection Limits.** Calculate the detection limits using the equation in Section 9.8. If the detection limits meet the Target Detection Limits (TDLs) in Table 1, then they are considered acceptable. If the TDLs are not met, the impact of the detection limits shall be calculated using the procedures in Section 9.9. If the maximum potential value of the sum of the summed detection limits is less then 50 percent of the emission standard, the detection limits are acceptable. If the value is greater than 50 percent of the emission standard, then the analysis and/or sampling and analysis must be repeated until acceptable detection limits are obtained.

## 8. QUALITY ASSURANCE

**8.1 Applicability.** When the method is used to analyze samples to demonstrate compliance with a source emission regulation, an audit sample must be analyzed, subject to availability.

**8.2 Audit Procedure.** Analyze an audit sample with each set of compliance samples. The audit sample contains tetra through octa isomers of PCDD and PCDF. Concurrently analyze the audit sample and a set of compliance samples in the same manner to evaluate the technique of the analyst and the standards preparation. The same analyst, analytical reagents, and analytical system shall be used both for the compliance samples and the EPA audit sample.

**8.3 Audit Sample Availability.** Audit samples will be supplied only to enforcement agencies for compliance tests. Audit samples may be obtained by writing:

Source Test Audit Coordinator (MD-77B)
Quality Assurance Division
Atmospheric Research and Exposure Assessment Laboratory
U.S. Environmental Protection Agency
Research Triangle Park, NC 27711

or by calling the Source Test Audit Coordinator (STAC) at (919)

541-7834. The audit sample request must be made at least 30 days prior to the scheduled compliance sample analysis.

8.4 **Audit Results.** Calculate the audit sample concentration according to the calculation procedure provided in the audit instructions included with the audit sample. Fill in the audit sample concentration and the analyst's name on the audit response form included with the audit instructions. Send one copy to the EPA Regional Office or the appropriate enforcement agency and a second copy to the STAC. The EPA Regional office or the appropriate enforcement agency will report the results of the audit to the laboratory being audited. Include this response with the results of the compliance samples in relevant reports to the EPA Regional Office or the appropriate enforcement agency.

## 9. CALCULATIONS

Same as Method 5, Section 6 with the following additions.

### 9.1 Nomenclature.

$A_{ai}$ = Integrated ion current of the noise at the retention time of the analyte.

$A_{cij}$ = Integrated ion current of the two ions characteristic of compound i in the jth calibration standard.

$A^*_{cij}$ = Integrated ion current of the two ions characteristic of the internal standard i in the jth calibration standard.

$A_{csi}$ = Integrated ion current of the two ions characteristic of surrogate compound i in the calibration standard.

$A_i$ = Integrated ion current of the two ions characteristic of compound i in the sample.

$A^*_i$ = Integrated ion current of the two ions characteristic of internal standard i in the sample.

$A_{rs}$ = Integrated ion current of the two ions characteristic of the recovery standard.

$A_{si}$ = Integrated ion current of the two ions characteristic of surrogate compound i in the sample.

$C_i$ = Concentration of PCDD or PCDF i in the sample, pg/$M^3$.

$C_T$ = Total concentration of PCDD's or PCDF's in the sample, pg/$M^3$.

DL = Detection limit, pg/sample.

$DL_{hs}$ = Detection limit for each homologous series, pg/sample.

$DL_{sum}$ = Sum of all isomers times the corresponding detection limit, ng/$m^3$.

$H_{ai}$ = Summed heights of the noise at the retention time of the analyte in the two analyte channels.

$m_{ci}$ = Mass of compound i in the calibration standard injected into the analyzer, pg.

$m^*_{ci}$ = Mass of labeled compound i in the calibration standard injected into the analyzer, pg.

$m^*_i$ = Mass of internal standard i added to the sample, pg.

$m_{rs}$ = Mass of recovery standard in the calibration standard injected into the analyzer, pg.

$m_s$ = Mass of surrogate compound in the sample to be analyzed, pg.

$m_{si}$ = Mass of surrogate compound i in the calibration standard, pg.

$RRF_i$ = Relative response factor for compound i.

$RRF_{rs}$ = Recovery standard response factor.

$RRF_s$ = Surrogate compound response factor.

$V_{m(std)}$ = Metered volume of sample run, dscm.

1000 = pg per ng.

### 9.2 Average Relative Response Factor.

$$RRF_i = \frac{1}{n}\sum_{j=1}^{n} \frac{A_{cij}\ m^*_{ci}}{A^*_{cij}\ m_{ci}} \qquad \text{Eq. 23-1}$$

### 9.3 Concentration of the PCDD's and PCDF's.

$$C_i = \frac{m_i^*\ A_i}{A_i^*\ RRF_i\ V_{m_{std}}} \qquad \text{Eq. 23-2}$$

### 9.4 Recovery Standard Response Factor.

$$RRF_{rs} = \frac{A_{ci}^{*}\, m_{rs}}{A_{rs}\, m_{ci}^{*}} \qquad \text{Eq. 23-3}$$

**9.5 Recovery of Internal Standards ($R^{*}$).**

$$R^{*} = \frac{A_{i}^{*}\, m_{rs}}{A_{rs}\, RF_{rs}\, m_{i}^{*}} \times 100\% \qquad \text{Eq. 23-4}$$

**9.6 Surrogate Compound Response Factor.**

$$RRF_{s} = \frac{A_{ci}^{*}\, m_{si}}{A_{csi}\, m_{ci}^{*}} \qquad \text{Eq. 23-5}$$

**9.7 Recovery of Surrogate Compounds ($R_s$).**

$$R_{s} = \frac{A_{si}\, m_{i}^{*}}{A_{i}^{*}\, RRF_{s}\, m_{s}} \times 100\% \qquad \text{Eq. 23-6}$$

**9.8 Detection Limit (DL).** The detection limit can be calculated based on either the height of the noise or the area of the noise using one of the two equations.

Detection limit using height for the DB-225 column. Three and one half times the height has been empirically determined to give area.

$$DL = \frac{2.5\ (3.5\ x\ H_{ai})\ m_i^*}{A_{ci}^*\ RRF_i} \qquad \text{Eq. 23-7}$$

Detection limit using height for the DB-5 column. Five times the height has been empirically determined to give area.

$$DL = \frac{2.5\ (5\ x\ H_{ai})\ m_i^*}{A_{ci}^*\ RRF_i} \qquad \text{Eq. 23-8}$$

Detection limit using area of the noise.

$$DL = \frac{2.5\ A_{ai}\ m_i^*}{A_{ci}^*\ RRF_i} \qquad \text{Eq. 23-9}$$

**9.9 Summed Detection Limits.** Calculate the maximum potential value of the summed detection limits. If the isomer (group of unresolved isomers) was not detected, use the value calculated for the detection limit in Section 9.8 above. If the isomer (group of unresolved isomers) was detected, use the value (target detection limit) from Table 1.

$$DL_{sum} = (13\ DL_{TCDD} + 16\ DL_{TCDF} + 12\ DL_{PeCDD} + 14\ DL_{PeCDF} + 7\ DL_{HxCDD} + 12\ DL_{HxCDF} + 2\ DL_{HpCDD} + 4\ DL_{HpCDF} + DL_{OCDD} + DL_{OCDF}) \ / \ 1000\ V_{m(std)} \qquad \text{Eq.23-10}$$

Note: The number of isomers used to calculate the summed detection limit represent the total number of isomers typically separated and not the actual number of isomers for each series.

**9.10 Total Concentration of PCDD's and PCDF's in the Sample.**

$$C_T = \sum_{i=1}^{n} C_i \qquad \text{Eq. 23-11}$$

Any PCDDs or PCDFs that are reported as not detected (below the DL) shall be counted as zero for the purpose of calculating the total concentration of PCDDs and PCDFs in the sample.

## 10. BIBLIOGRAPHY

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3. Thompson, J. R. (ed.). Analysis of Pesticide Residues in Human and Environmental Samples. U.S. Environmental Protection Agency. Research Triangle Park, NC. 1974.

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5. U.S. Environmental Protection Agency. Method 8290 - The Analysis of Polychlorinated Dibenzo-p-dioxin and Polychlorinated Dibenzofurans by High-Resolution Gas Chromatography/ High-Resolution Mass Spectrometry. In: Test Methods for Evaluating Solid Waste. Washington, DC. SW-846.

6. Personnel communications with R. L. Harless of U.S. EPA and Triangle Laboratory staff.

TABLE 23-1. TARGET DETECTION LIMITS (TDLs)

| ANALYTE | TDL (pg/Sample Train) |
|---|---|
| TCDD/TCDF | 50 |
| PeCDD/PeCDF | 250 |
| HxCDD/HxCDF | 250 |
| HpCDD/HpCDF | 250 |
| OCDD/OCDF | 500 |

TABLE 23-2. COMPOSITION OF THE SAMPLE FORTIFICATION AND RECOVERY STANDARDS SOLUTIONS*

| ANALYTE | CONCENTRATION (pg/$\mu$L) |
|---|---|
| Internal Standards | |
| $^{13}C_{12}$-2,3,7,8-TCDD | 100 |
| $^{13}C_{12}$-1,2,3,7,8-PeCDD | 100 |
| $^{13}C_{12}$-1,2,3,6,7,8-HxCDD | 100 |
| $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD | 100 |
| $^{13}C_{12}$-OCDD | 100 |
| $^{13}C_{12}$-2,3,7,8-TCDF | 100 |
| $^{13}C_{12}$-1,2,3,7,8-PeCDF | 100 |
| $^{13}C_{12}$-1,2,3,6,7,8-HxCDF | 100 |
| $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF | 100 |
| Surrogate Standards | |
| $^{37}Cl_4$-2,3,7,8-TCDD | 100 |
| $^{13}C_{12}$-1,2,3,4,7,8-HxCDD | 100 |
| $^{13}C_{12}$-2,3,4,7,8-PeCDF | 100 |
| $^{13}C_{12}$-1,2,3,4,7,8-HxCDF | 100 |
| $^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF | 100 |
| Recovery Standards | |

|  |  |
|---|---|
| $^{13}C_{12}$-1,2,3,4-TCDD | 100 |
| $^{13}C_{12}$-1,2,3,7,8,9-HxCDD | 100 |

[*] Calibration levels are specific for samples at the MWC compliance standard level.

TABLE 23-3. COMPOSITION OF THE INITIAL CALIBRATION SOLUTIONS

| COMPOUND | CONCENTRATIONS (pg/μl) | | | | |
|---|---|---|---|---|---|
| SOLUTION NO. | 1 | 2 | 3 | 4 | 5 |
| UNLABELED ANALYTES | | | | | |
| 2,3,7,8-TCDD | 0.5 | 1 | 5 | 50 | 100 |
| 2,3,7,8-TCDF | 0.5 | 1 | 5 | 50 | 100 |
| 1,2,3,7,8-PeCDD | 2.5 | 5 | 25 | 250 | 500 |
| 1,2,3,7,8-PeCDF | 2.5 | 5 | 25 | 250 | 500 |
| 2,3,4,7,8-PeCDF | 2.5 | 5 | 25 | 250 | 500 |
| 1,2,3,4,7,8-HxCDD | 2.5 | 5 | 25 | 250 | 500 |
| 1,2,3,6,7,8-HxCDD | 2.5 | 5 | 25 | 250 | 500 |
| 1,2,3,7,8,9-HxCDD | 2.5 | 5 | 25 | 250 | 500 |
| 1,2,3,4,7,8-HxCDF | 2.5 | 5 | 25 | 250 | 500 |
| 1,2,3,6,7,8-HxCDF | 2.5 | 5 | 25 | 250 | 500 |
| 1,2,3,7,8,9-HxCDF | 2.5 | 5 | 25 | 250 | 500 |
| 2,3,4,6,7,8-HxCDD | 2.5 | 5 | 25 | 250 | 500 |
| 1,2,3,4,6,7,8-HpCDD | 2.5 | 5 | 25 | 250 | 500 |
| 1,2,3,4,6,7,8-HpCDF | 2.5 | 5 | 25 | 250 | 500 |
| 1,2,3,4,7,8,9-HpCDF | 2.5 | 5 | 25 | 250 | 500 |
| OCDD | 5 | 10 | 50 | 500 | 1000 |
| OCDF | 5 | 10 | 50 | 500 | 1000 |
| INTERNAL STANDARDS | | | | | |
| $^{13}C_{12}$-2,3,7,8-TCDD | 100 | 100 | 100 | 100 | 100 |
| $^{13}C_{12}$-1,2,3,7,8-PeCDD | 100 | 100 | 100 | 100 | 100 |
| $^{13}C_{12}$-1,2,3,6,7,8-HxCDD | 100 | 100 | 100 | 100 | 100 |
| $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD | 100 | 100 | 100 | 100 | 100 |
| $^{13}C_{12}$-OCDD | 100 | 100 | 100 | 100 | 100 |

| | | | | | |
|---|---|---|---|---|---|
| $^{13}C_{12}$-2,3,7,8-TCDF | 100 | 100 | 100 | 100 | 100 |
| $^{13}C_{12}$-1,2,3,7,8-PeCDF | 100 | 100 | 100 | 100 | 100 |
| $^{13}C_{12}$-1,2,3,6,7,8-HxCDF | 100 | 100 | 100 | 100 | 100 |
| $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF | 100 | 100 | 100 | 100 | 100 |

TABLE 23-3. (Continued)

| COMPOUND | CONCENTRATION (pg/µl) | | | | |
|---|---|---|---|---|---|
| SOLUTION NO. | 1 | 2 | 3 | 4 | 5 |
| SURROGATE STANDARDS | | | | | |
| $^{37}Cl_4$-2,3,7,8-TCDD | 60 | 80 | 100 | 120 | 140 |
| $^{13}C_{12}$-2,3,4,7,8-PeCDF | 60 | 80 | 100 | 120 | 140 |
| $^{13}C_{12}$-1,2,3,4,7,8-HxCDD | 60 | 80 | 100 | 120 | 140 |
| $^{13}C_{12}$-1,2,3,4,7,8-HxCDF | 60 | 80 | 100 | 120 | 140 |
| $^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF | 60 | 80 | 100 | 120 | 140 |
| RECOVERY STANDARDS | | | | | |
| $^{13}C_{12}$-1,2,3,4-TCDD | 100 | 100 | 100 | 100 | 100 |
| $^{13}C_{12}$-1,2,3,7,8,9-HxCDD | 100 | 100 | 100 | 100 | 100 |

TABLE 23-4. RECOMMENDED GC OPERATING CONDITIONS

| Column Type | DB-5 | DB-225 |
|---|---|---|
| Length (m) | 60 | 30 |
| i.d. (mm) | 0.25 | 0.25 |
| Film Thickness (μm) | 0.25 | 0.25 |
| Carrier Gas | Helium | Helium |
| Carrier Gas Flow (mL/min) | 1-2 | 1-2 |
| Injection Mode | <-- splitless | --> |
| Valve Time (min) | 2.5 | 2.5 |
| Initial Temperature (° C) | 150 | 130 |
| Initial Time (min) | 0.5 | 2.5 |
| Rate 1 (deg. C/min) | 60 | 50 |
| Temperature 2 (deg. C) | 170 | 170 |
| Rate 2 (deg. C/min) | 3 | 4 |
| Final Temperature (deg. C) | 300 | 250 |

TABLE 23-5. ELEMENTAL COMPOSITIONS AND EXACT MASSES OF THE IONS MONITORED BY HIGH RESOLUTION MASS SPECTROMETRY FOR PCDD's AND PCDF's

| DESCRIPTOR NUMBER | ACCURATE MASS | ION TYPE | ELEMENTAL COMPOSITION | ANALYTE |
|---|---|---|---|---|
| 2 | 292.9825 | LOCK | $C_7F_{11}$ | PFK |
| | 303.9016 | M | $C_{12}H_4{}^{35}Cl_4O$ | TCDF |
| | 305.8987 | M+2 | $C_{12}H_4{}^{35}Cl_3Cl^{37}O$ | TCDF |
| | 315.9419 | M | ${}^{13}C_{12}H_4{}^{35}Cl_4O$ | TCDF(S) |
| | 317.9389 | M+2 | ${}^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO$ | TCDF(S) |
| | 319.8965 | M | $C_{12}H_4{}^{35}Cl_4O_2$ | TCDD |
| | 321.8936 | M+2 | $C_{12}H_4{}^{35}Cl_3{}^{37}ClO_2$ | TCDD |
| | 327.8847 | M | $C_{12}H_4{}^{37}Cl_4O_2$ | TCDD(S) |
| | 330.9792 | QC | $C_7F_{13}$ | PFK |
| | 331.9368 | M | ${}^{13}C_{12}H_4{}^{35}Cl_4O_2$ | TCDD(S) |
| | 333.9339 | M+2 | ${}^{13}C_{12}H_4{}^{35}Cl^{37}ClO_2$ | TCDD(S) |
| | 339.8597 | M+2 | $C_{12}H_3{}^{35}Cl_4{}^{37}ClO$ | PeCDF |
| | 341.8567 | M+4 | $C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$ | PeCDF |
| | 351.9000 | M+2 | ${}^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO$ | PeCDF(S) |
| | 353.8970 | M+4 | ${}^{13}C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$ | PeCDF(S) |
| | 355.8546 | M+2 | $C_{12}H_3{}^{35}Cl_3{}^{37}ClO_2$ | PeCDD |
| | 357.8516 | M+4 | $C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$ | PeCDD |
| | 367.8949 | M+2 | ${}^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$ | PeCDD(S) |
| | 369.8919 | M+4 | ${}^{13}C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$ | PeCDD(S) |
| | 375.8364 | M+2 | $C_{12}H_4{}^{35}Cl_5{}^{37}ClO$ | HxCDPE |
| | 409.7974 | M+2 | $C_{12}H_3{}^{35}Cl_6{}^{37}ClO$ | HpCPDE |
| 3 | 373.8208 | M+2 | $C_{12}H_2 35Cl_5{}^{37}ClO$ | HxCDF |
| | 375.8178 | M+4 | $C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O$ | HxCDF |
| | 383.8639 | M | ${}^{13}C_{12}H_2{}^{35}Cl_6O$ | HxCDF(S) |
| | 385.8610 | M+2 | ${}^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO$ | HxCDF(S) |
| | 389.8157 | M+2 | $C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$ | HxCDD |
| | 391.8127 | M+4 | $C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$ | HxCDD |
| | 392.9760 | LOCK | $C_9F_{15}$ | PFK |

| | | | | |
|---|---|---|---|---|
| | 401.8559 | M+2 | $^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$ | HxCDD(S) |
| | 403.8529 | M+4 | $^{13}C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O$ | HxCDD(S) |
| | 445.7555 | M+4 | $C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2O$ | OCDPE |
| | 430.9729 | QC | $C_9F_{17}$ | PFK |

TABLE 23-5. (Continued)

| DESCRIPTOR NUMBER | ACCURATE MASS | ION TYPE | ELEMENTAL DESCRIPTION | ANALYTE |
|---|---|---|---|---|
| | 407.7818 | M+2 | $C_{12}H^{35}Cl_6{}^{37}ClO$ | HpCDF |
| | 409.7789 | M+4 | $C_{12}H^{35}Cl_5{}^{37}Cl_2O$ | HpCDF |
| | 417.8253 | M | $^{13}C_{12}H^{35}Cl_7O$ | HpCDF(S) |
| | 389.8157 | M+2 | $C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$ | HxCDD |
| | 391.8127 | M+4 | $C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$ | HxCDD |
| | 392.9760 | LOCK | $C_9F_{15}$ | PFK |
| | 401.8559 | M+2 | $^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$ | HxCDD(S) |
| | 403.8529 | M+4 | $^{13}C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O$ | HxCDD(S) |
| | 445.7555 | M+4 | $C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2O$ | OCDPE |
| | 430.9729 | QC | $C_9F_{17}$ | PFK |
| | 407.7818 | M+2 | $C_{12}H^{35}Cl_6{}^{37}ClO$ | HpCDF |
| | 409.7789 | M+4 | $C_{12}H^{35}Cl_5{}^{37}Cl_2O$ | HpCDF |
| | 417.8253 | M | $^{13}C_{12}H^{35}Cl_7O$ | HpCDF(S) |
| | 419.8220 | M+2 | $^{13}C_{12}H^{35}Cl_6{}^{37}ClO$ | HpCDF(S) |
| | 423.7766 | M+2 | $C_{12}H^{35}Cl_6{}^{37}ClO_2$ | HpCDD |
| | 425.7737 | M+4 | $C_{12}H^{35}Cl_5{}^{37}Cl_2O_2$ | HpCDD |
| | 435.8169 | M+2 | $^{13}C_{12}H^{35}Cl_6{}^{37}ClO_2$ | HpCDD(S) |
| | 437.8140 | M+4 | $^{13}C_{12}H^{35}Cl_5{}^{37}Cl_2O_2$ | HpCDD(S) |
| | 479.7165 | M+4 | $C_{12}H^{35}Cl_7{}^{37}Cl_2O$ | NCPDE |
| | 430.9729 | LOCK | $C_9F_{17}$ | PFK |
| | 441.7428 | M+2 | $C_{12}{}^{35}Cl_7{}^{37}ClO$ | OCDF |
| | 443.7399 | M+4 | $C_{12}{}^{35}Cl_6{}^{37}Cl_2O$ | OCDF |
| | 457.7377 | M+2 | $C_{12}{}^{35}Cl_7{}^{37}ClO_2$ | OCDD |
| | 459.7348 | M+4 | $C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$ | OCDD |
| | 469.7779 | M+2 | $^{13}C_{12}{}^{35}Cl_7{}^{37}ClO_2$ | OCDD(S) |

| | | | | |
|---|---|---|---|---|
| | 471.7750 | M+4 | $^{13}C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$ | OCDD(S) |
| | 513.6775 | M+4 | $C_{12}{}^{35}Cl_8{}^{37}Cl_2O_2$ | DCDPE |
| | 442.9728 | QC | $C_{10}F_{17}$ | PFK |

The following nuclidic masses were used:
H = 1.007825 O = 15.994914 C = 12.000000 $^{35}$Cl = 34.968853
$^{13}$C = 13.003355 $^{37}$Cl = 36.965903 F = 18.9984
S = Labeled Standard
QC = Ion selected for monitoring instrument stability during the GC/MS analysis.

TABLE 23-6. ACCEPTABLE RANGES FOR ION-ABUNDANCE RATIOS OF PCDD's AND PCDF's

| Number of Chlorine Atoms | Ion Type | Theoretical Ratio | Control Limits | |
|---|---|---|---|---|
| | | | Lower | Upper |
| 4 | M/M+2 | 0.77 | 0.65 | 0.89 |
| 5 | M+2/M+4 | 1.55 | 1.32 | 1.78 |
| 6 | M+2/M+4 | 1.24 | 1.05 | 1.43 |
| 6[a] | M/M+2 | 0.51 | 0.43 | 0.59 |
| 7[b] | M?M+2 | 0.44 | 0.37 | 0.51 |
| 7 | M+2/M+4 | 1.04 | 0.88 | 1.20 |
| 8 | M+2/M+4 | 0.89 | 0.76 | 1.02 |

TABLE 23-7. UNLABELED ANALYTES QUANTIFICATION RELATIONSHIPS

| ANALYTE | INTERNAL STANDARD USED |
|---|---|
| 2,3,7,8-TCDD | $^{13}C_{12}$-2,3,7,8-TCDD |
| Other TCDD's | $^{13}C_{12}$-2,3,7,8-TCDD |
| | |
| 1,2,3,7,8-PeCDD | $^{13}C_{12}$-1,2,3,7,8-PeCDD |
| Other PeCDD's | $^{13}C_{12}$-1,2,3,7,8-PeCDD |
| | |
| 1,2,3,4,7,8-HxCDD | $^{13}C_{12}$-1,2,3,6,7,8-HxCDD |
| 1,2,3,6,7,8-HxCDD | $^{13}C_{12}$-1,2,3,6,7,8-HxCDD |
| 1,2,3,7,8,9-HxCDD | $^{13}C_{12}$-1,2,3,6,7,8-HxCDD |
| Other HxCDD's | $^{13}C_{12}$-1,2,3,6,7,8-HxCDD |
| | |
| 1,2,3,4,6,7,8-HpCDD | $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD |
| Other HpCDD's | $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD |
| | |
| OCDD | $^{13}C_{12}$-OCDD |
| | |
| 2,3,7,8-TCDF | $^{13}C_{12}$-2,3,7,8-TCDF |
| Other TCDF's | $^{13}C_{12}$-2,3,7,8-TCDF |
| | |
| 1,2,3,7,8-PeCDF | $^{13}C_{12}$-1,2,3,7,8-PeCDF |
| 2,3,4,7,8-PeCDF | $^{13}C_{12}$-1,2,3,7,8-PeCDF |
| Other PeCDF's | $^{13}C_{12}$-1,2,3,7,8-PeCDF |
| | |
| 1,2,3,4,7,8-HxCDF | $^{13}C_{12}$-1,2,3,6,7,8-HxCDF |
| 1,2,3,6,7,8-HxCDF | $^{13}C_{12}$-1,2,3,6,7,8-HxCDF |
| 1,2,3,7,8,9-HxCDF | $^{13}C_{12}$-1,2,3,6,7,8-HxCDF |
| 2,3,4,6,7,8-HxCDF | $^{13}C_{12}$-1,2,3,6,7,8-HxCDF |
| Other HxCDF's | $^{13}C_{12}$-1,2,3,6,7,8-HxCDF |
| | |
| 1,2,3,4,6,7,8-HpCDF | $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF |

| | |
|---|---|
| 1,2,3,4,7,8,9-HpCDF | $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF |
| | |
| OCDF | $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF |

## TABLE 23-8. INTERNAL STANDARDS QUANTIFICATION RELATIONSHIPS

| INTERNAL STANDARD | STANDARD USED DURING PERCENT RECOVERY DETERMINATION |
|---|---|
| $^{13}C_{12}$-2,3,7,8-TCDD | $^{13}C_{12}$-1,2,3,4-TCDD |
| $^{13}C_{12}$-1,2,3,7,8-PeCDD | $^{13}C_{12}$-1,2,3,4-TCDD |
| $^{13}C_{12}$-1,2,3,6,7,8-HxCDD | $^{13}C_{12}$-1,2,3,7,8,9-HxCDD |
| $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD | $^{13}C_{12}$-1,2,3,7,8,9-HxCDD |
| $^{13}C_{12}$-OCDD | $^{13}C_{12}$-1,2,3,7,8,9-HxCDD |
| | |
| $^{13}C_{12}$-2,3,7,8-TCDF | $^{13}C_{12}$-1,2,3,4-TCDD |
| $^{13}C_{12}$-1,2,3,7,8-PeCDF | $^{13}C_{12}$-1,2,3,4-TCDD |
| $^{13}C_{12}$-1,2,3,6,7,8-HxCDF | $^{13}C_{12}$-1,2,3,7,8,9-HxCDD |
| $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF | $^{13}C_{12}$-1,2,3,7,8,9-HxCDD |

## TABLE 23-9. SURROGATE STANDARDS QUANTIFICATION RELATIONSHIPS

| SURROGATE STANDARD | STANDARD USED DURING PERCENT RECOVERY DETERMINATION |
|---|---|
| $^{37}Cl_4$-2,3,7,8-TCDD | $^{13}C_{12}$-2,3,7,8-TCDD |
| $^{13}C_{12}$-2,3,4,7,8-PeCDF | $^{13}C_{12}$-1,2,3,7,8-PeCDF |
| $^{13}C_{12}$-1,2,3,4,7,8-HxCDD | $^{13}C_{12}$-1,2,3,6,7,8-HxCDD |
| $^{13}C_{12}$-1,2,3,4,7,8-HxCDF | $^{13}C_{12}$-1,2,3,6,7,8-HxCDF |
| $^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF | $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF |

## TABLE 23-10. MINIMUM REQUIREMENTS FOR INITIAL AND DAILY CALIBRATION RESPONSE FACTORS

| COMPOUND | RELATIVE RESPONSE FACTORS | |
|---|---|---|
| | INITIAL CALIBRATION (RSD) | DAILY CALIBRATION (% DIFFERENCE) |
| UNLABELED ANALYTES | | |
| 2,3,7,8-TCDD | 25 | 25 |
| 2,3,7,8-TCDF | 25 | 25 |
| 1,2,3,7,8-PeCDD | 25 | 25 |
| 1,2,3,7,8-PeCDF | 25 | 25 |
| 1,2,4,5,7,8-HxCDD | 25 | 25 |
| 1,2,3,6,7,8-HxCDD | 25 | 25 |
| 1,2,3,7,8,9-HxCDD | 25 | 25 |
| 1,2,3,4,7,8-HxCDF | 25 | 25 |
| 1,2,3,6,7,8-HxCDF | 25 | 25 |
| 1,2,3,7,8,9-HxCDF | 25 | 25 |
| 2,3,4,6,7,8-HxCDF | 25 | 25 |
| 1,2,3,4,6,7,8-HpCDD | 25 | 25 |
| 1,2,3,4,6,7,8-HpCDF | 25 | 25 |
| OCDD | 25 | 25 |
| OCDF | 30 | 30 |
| SURROGATE STANDARDS | | |
| $^{37}Cl_4$-2,3,7,8-TCDD | 25 | 25 |
| $^{13}C_{12}$-2,3,4,7,8-PeCDF | | |
| $^{13}C_{12}$-1,2,3,4,7,8-HxCDD | | |
| $^{13}C_{12}$-1,2,3,4,7,8-HxCDF | | |
| $^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF | | |

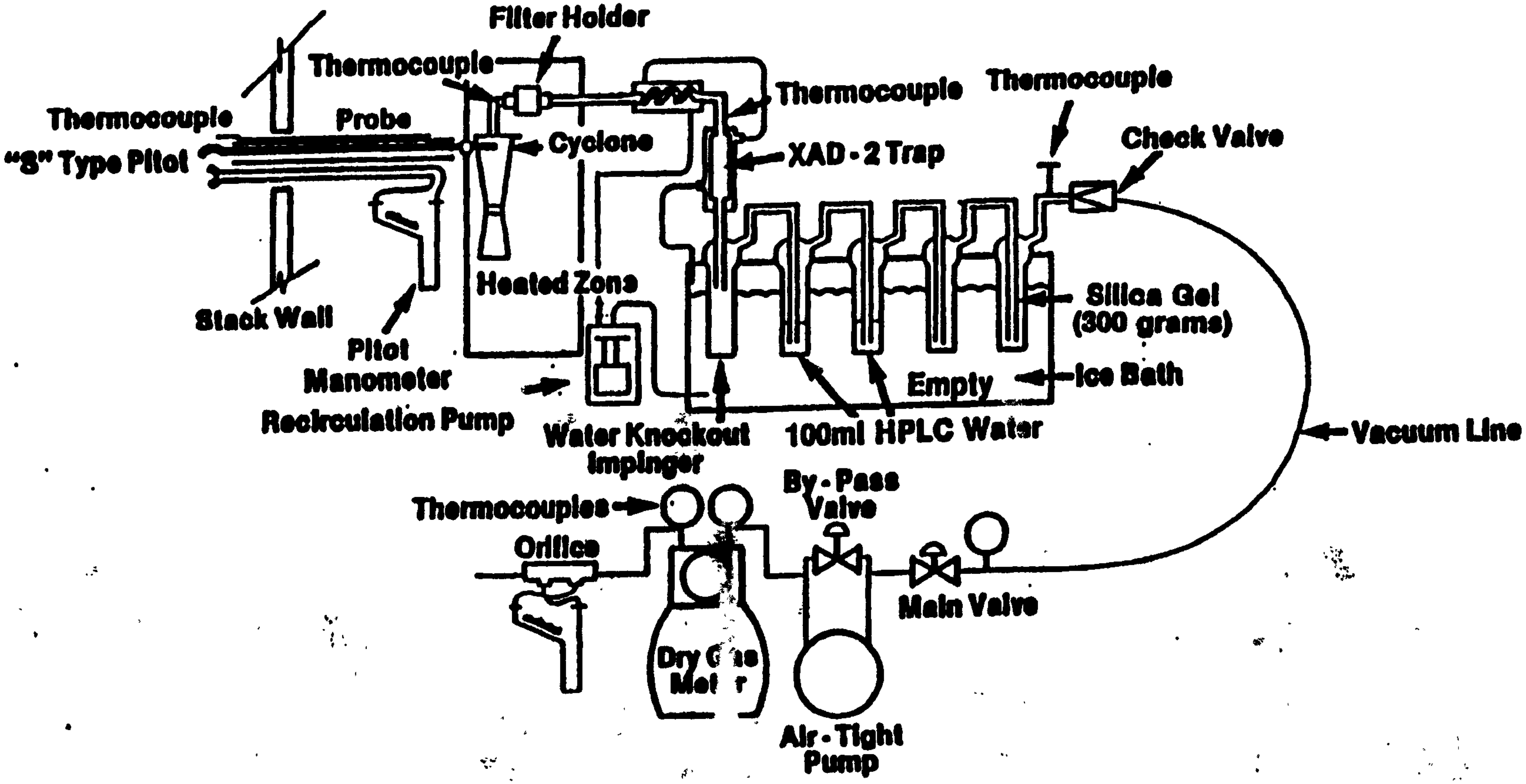

Figure 5-1. CDD/CDF Sampling Train Configuration

(Figure 2)

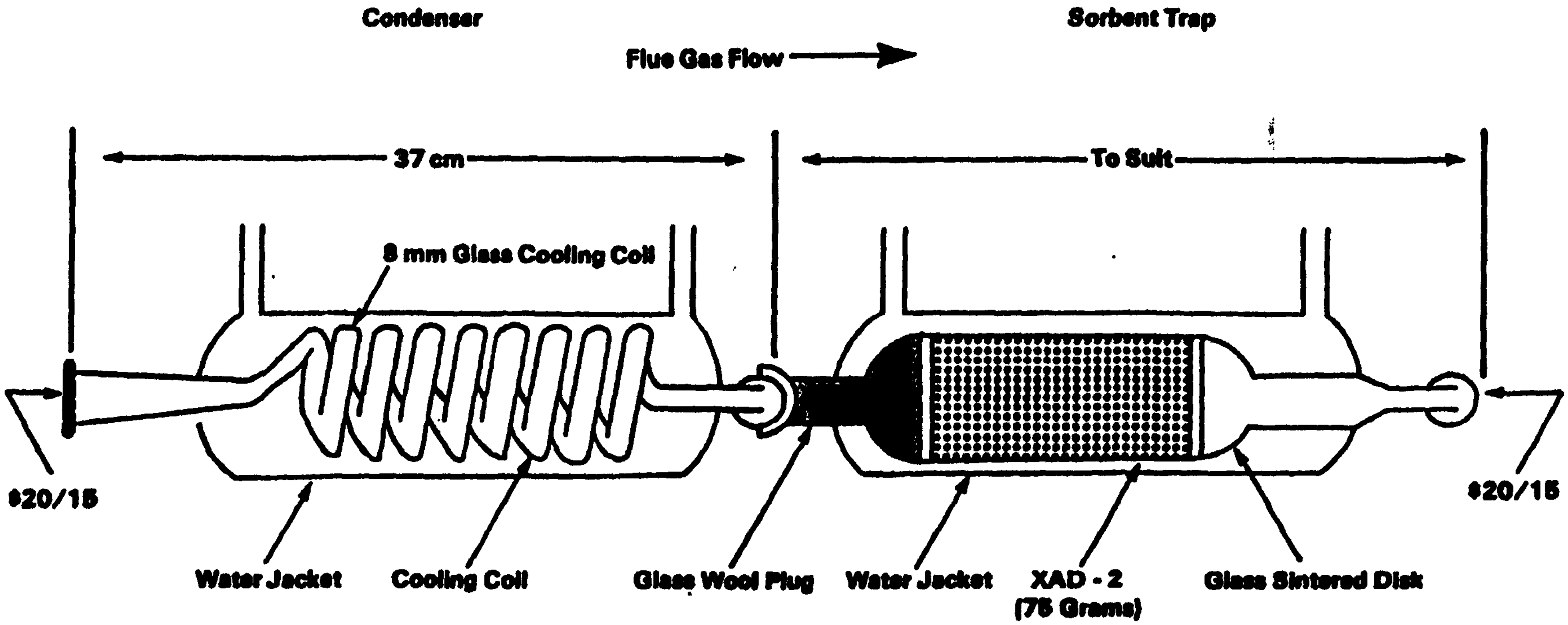

FIGURE 2. CONDENSER AND SORBENT TRAP FOR COLLECTION OF GASEOUS PCDDs AND PCDFs

# Appendix G.5

## Sampling & Analysis Methods

## EPA Method 25A

# METHOD 25A-DETERMINATION OF TOTAL GASEOUS ORGANIC CONCENTRATION USING A FLAME IONIZATION ANALYZER

## 1. Applicability and Principle

**1.1 Applicability.** This method applies to the measurement of total gaseous organic concentration of vapors consisting primarily of alkanes, alkenes, and/or arenes (aromatic hydrocarbons). The concentration is expressed in terms of propane (or other appropriate organic calibration gas) or in terms of carbon.

**1.2 Principle.** A gas sample is extracted from the source through a heated sample line, if necessary, and glass fiber filter to a flame ionization analyzer (FIA). Results are reported as volume concentration equivalents of the calibration gas or as carbon equivalents.

## 2. Definitions

**2.1 Measurement Systems.** The total equipment required for the determination of the gas concentration. The system consists of the following major subsystems:

**2.1.1 Sample Interface.** That portion of the system that is used for one or more of the following: sample acquisition, sample transportation, sample conditioning, or protection of the analyzer from the effects of the stack effluent.

**2.1.2 Organic Analyzer.** That portion of the system that senses organic concentration and generates an output proportional to the gas concentration.

**2.2 Span Value.** The upper limit of a gas concentration measurement range that is specified for affected source categories in the applicable part of the regulations. The span value is established in the applicable regulation and is usually 1.5 to 2.5 times the applicable emission limit. If no span value is provided, use a span value equivalent to 1.5 to 2.5 times the expected concentration. For convenience, the span value should correspond to 100 percent of the recorder scale.

**2.3 Calibration Gas.** A known concentration of a gas in an appropriate diluent gas.

**2.4 Zero Drift.** The difference in the measurement system response to a zero level calibration gas before and after a stated period of operation during which no unscheduled maintenance, repair, or adjustment took place.

**2.5 Calibration drift.** The difference in the measurement system response to a midlevel calibration gas before and after a stated period of operation during which no unscheduled maintenance, repair or adjustment took place.

**2.6 Response Time.** The time interval from a step change in pollutant concentration at the inlet to the emission measu[illegible] to the time at which 95 percent of the corresponding final value is reached as displayed on the recorder.

**2.7 Calibration Error.** The difference between the gas concentration indicated by the measurement system and the known concentration of the calibration gas.

## 3. Apparatus.

A schematic of an acceptable measurement system is shown in Figure 25A-1. The essential components of the measurement system are described below:

**3.1 Organic Concentration Analyzer.** A flame ionization analyzer (FIA) capable of meeting or exceeding the specifications in this method.

**3.2 Sample Probe.** Stainless steel, or equivalent, three-hole rake type. Sample holes shall be 4 mm in diameter or smaller and located at 16.7, 50, and 83.3 percent of the equivalent stack diameter. Alternatively, a single opening probe may be used so that a gas sample is collected from the centrally located 10 percent area of the stack cross-section.

**3.3 Sample Line.** Stainless steel or Teflon * tubing to transport the sample gas to the analyzer. The sample line should be heated, if necessary, to prevent condensation in the line.

**3.4 Calibration Valve Assembly.** A three way valve assembly to direct the zero and calibration gases to the analyzers is recommended. Other methods, such as quick-connect lines, to route calibration gas to the analyzers are applicable.

**3.5 Particulate Filter.** An in-stack or an out-of-stack glass fiber filter is recommended if exhaust gas particulate loading is significant. An out-of-stack filter should be heated to prevent any condensation.

* Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

**3.6 Recorder.** A strip-chart recorder, analog computer, or digital recorder for recording measurement data. The minimum data recording requirement is one measurement value per minute, Note: This method is often applied in highly explosive areas. Caution and care should be exercised in choice of equipment and installation.

## 4. Calibration and Other Gases.

Gases used for calibrations, fuel, and combustion air (if required) are contained in compressed gas cylinders. Preparation of calibration gases shall be done according to the procedure in Protocol No. 1, listed in Citation 2 of Bibliography. Additionally, the manufacturer of the cylinder should provide a recommended shelf life for each calibration gas cylinder over which the concentration does not change more than ±2 percent from the certified value. For calibration gas values not generally available (i.e., organics between 1 and 10 percent by volume), alternative methods for preparing calibration gas mixtures, such as dilution systems, may be used with prior approval of the Administrator.

Calibration gases usually consist of propane in air or nitrogen and are determined in terms of the span value. Organic compounds other than propane can be used following the above guidelines and making the appropriate corrections for response factor.

4.1 **Fuel.** A 40 percent $H_2$/60 percent $N_2$ gas mixture is recommended to avoid an oxygen synergism effect that reportedly occurs when oxygen concentration varies significantly from a mean value.

4.2 **Zero Gas.** High purity air with less than 0.1 parts per million by volume (ppmv) of organic material (propane or carbon equivalent) or less than 0.1 percent of the span value, whichever is greater.

4.3 **Low-level Calibration Gas.** An organic calibration gas with a concentration equivalent to 25 to 35 percent of the applicable span value.

4.4 **Mid-level Calibration Gas.** An organic calibration gas with a concentration equivalent to 45 to 55 percent of the applicable span value.

4.5 **High-level Calibration Gas.** An organic calibration gas with a concentration equivalent to 80 to 90 percent of the applicable span value.

## 5. Measurement System Performance Specifications

5.1 **Zero Drift.** Less than ±3 percent of the span value.

5.2 **Calibration Drift.** Less than ±3 percent of span value.

5.3 **Calibration Error.** Less than ±5 percent of the calibration gas value.

## 6. Pretest Preparations

6.1 **Selection of Sampling Site.** The location of the sampling site is generally specified by the applicable regulation or purpose of the test; i.e., exhaust stack, inlet line, etc. The sample port shall be located at least 1.5 meters or 2 equivalent diameters upstream of the gas discharge to the atmosphere.

6.2 **Location of Sample Probe.** Install the sample probe so that the probe is centrally located in the stack, pipe, or duct and is sealed tightly at the stack port connection.

**6.3 Measurement System Preparation.** Prior to the emission test, assemble the measurement system following the manufacturer's written instructions in preparing the sample interface and the organic analyzer. Make the system operable.

FIA equipment can be calibrated for almost any range of total organics concentrations. For high concentrations of organics [illegible] percent by volume as propane) modifications to most commonly available analyzers are necessary. One accepted method of equipment modification is to decrease the size of the sample to the analyzer through the use of a smaller diameter sample capillary. Direct and continuous measurement of organic concentration is a necessary consideration when determining any modification design.

**6.4 Calibration Error Test.** Immediately prior to the test series, (within 2 hours of the start of the test) introduce zero gas and high-level calibration gas at the calibration valve assembly. Adjust the analyzer output to the appropriate levels, if necessary. Calculate the predicted response for the low-level and mid-level gases based on a linear response line between the zero and high-level responses. Then introduce low-level and mid-level calibration gases successively to the measurement system. Record the analyzer responses for low-level and mid-level calibration gases and determine the differences between the measurement system responses and the predicted responses. These differences must be less than 5 percent of the respective calibration gas value. If not, the measurement system is not acceptable and must be replaced or repaired prior to testing. No adjustments to the measurement system shall be conducted [illegible] the calibration and before the drift check (Section 7.3). If adjustments are necessary before the completion of the test series, perform the drift checks prior to the required adjustments and repeat the calibration following the adjustments. If multiple electronic ranges are to be used, each additional range must be checked with a mid-level calibration gas to verify the multiplication factor.

**6.5 Response Time Test.** Introduce Zero gas into the measurement system at the calibration valve assembly. When the system output has stabilized, switch quickly to the high-level calibration gas. Record the time from the concentration change to the measurement system response equivalent to 95 percent of the step change. Repeat the test three times and average the results.

## 7. Emission Measurement Test Procedure

**7.1 Organic Measurement.** Begin sampling at the start of the test period, recording time and any required process information as appropriate. In particular, note on the recording chart periods of process interruption or cyclic operation.

**7.2 Drift Determination.** Immediately following the completion of the test period and hourly during the test period, reintroduce the zero and mid-level calibration gases, one at a time, to the measurement system at the calibration valve assembly. (Make no adjustments to the measurement system until after both the zero and calibration drift checks are made.) Record the analyzer response. If the drift values exceed the specified limits, invalidate the test results preceding the check and repeat the test following corrections to the measurement system. Alternatively, recalibrate the test measurement system as in Section 6.4 and report the results using both sets of calibration data (i.e., data determined prior to the test period and data determined following the test period).

## 8. Organic Concentration calculations

Determine the average organic concentration in terms of ppmv as propane or other calibration gas. The average shall be determined by the integration of the output recording over the period specified in the applicable regulation. If results are required in terms of ppmv as carbon, adjust measured concentrations using Equation 25A-1.

$$C_C = KC_{meas} \qquad \text{Eq. 25A-1}$$

Where:

$C_c$ = Organic concentration as carbon, ppmv.

$C_{meas}$= Organic concentration as measured, ppmv.

K = Carbon equivalent correction factor.

K = 2 for ethane.

K = 3 for propane.

K = 4 for butane.

K = Appropriate response factor for other organic calibration gases.

## 9. Bibliography

1. Measurement of Volatile Organic Compounds-Guideline Series. U.S. Environmental Protection Agency. Research Triangle Park, NC. Publication No. EPA-450/2-78-041. June 1978. p. 46-54.

2. Traceability Protocol for Establishing True Concentrations of Gases Used for Calibration and Audits of Continuous Source Emission Monitors (Protocol No. 1). U.S. Environmental Protection Agency,

Environmental Monitoring and Support Laboratory. Research Triangle Park, NC. June 1978.

3. Gasoline Vapor Emission Laboratory Evaluation-Part 2. U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards. Research Triangle Park, NC. EMB Report No. 75-GAS-6. August 1975.

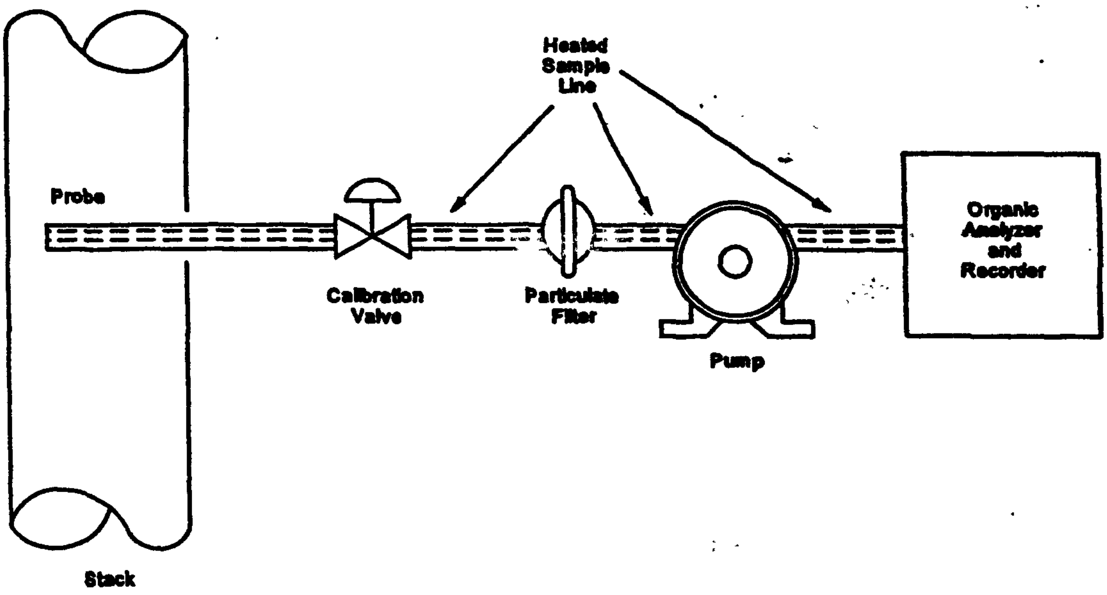

Figure 25A-1. Organic Concentration Measurement System.

# Appendix G.6

## Sampling & Analysis Methods

## EPA Proposed Method 322

# (PROPOSED) TEST METHOD 322 - MEASUREMENT OF HYDROGEN CHLORIDE EMISSIONS FROM PORTLAND CEMENT KILNS BY GFCIR

## 1.0 Applicability and Principle

1.1 Applicability. This method is applicable to the determination of hydrogen chloride (HCl) concentrations in emissions from portland cement kilns. This is an instrumental method for the measurement of HCl using an extractive sampling system and an infrared (IR) gas-filter correlation (GFC) analyzer. This method is intended to provide the cement industry with a direct interface instrumental method. A procedure for analyte spiking is included for quality assurance. This method is considered to be self-validating provided that the requirements in section 9 of this method are followed.

1.2 Principle. A gas sample is continuously extracted from a stack or duct over the test period using either a source-level hot/wet extractive subsystem or a dilution extractive subsystem. A nondispersive infrared gas filter correlation (NDIR-GFC) analyzer is specified for the measurement of HCl in the sample. The total measurement system is comprised of the extractive subsystem, the analyzer, and the data acquisition subsystem. Test system performance specifications are included in this method to provide for the collection of accurate, reproducible data.

1.3 Test System Operating Range. The measurement range (span) of the test system shall include the anticipated HCl concentrations of the effluent and spiked samples. The range should be selected so that the average of the effluent measurements is between 25 and 75 percent of span. If at any time during a test run, the effluent concentration exceeds the span value of the test system, the run shall be considered invalid.

## 2.0 Summary of Method

2.1 Sampling and Analysis. Kiln gas is continuously extracted from the stack or duct using either a source level, hot/wet extractive system, or an in-situ dilution probe or heated out-of-stack dilution system. The sample is then directed by a heated sample line maintained above 350°F to a GFC analyzer having a range appropriate to the type of sampling system. The gas filter correlation analyzer incorporates a gas cell filled with HCl. This gas cell is periodically moved into the path of an infrared measurement beam of the instrument to filter out essentially all of the HCl absorption wavelengths. Spectral filtering provides a reference from which the HCl concentration of the sample can be determined. Interferences are minimized in the analyzer by choosing a spectral band over which compounds such as $CO_2$ and $H_2O$ either do not absorb significantly or do not match the spectral pattern of the HCl infrared absorption.

2.2 Operator Requirements. The analyst must be familiar with the specifications and test procedures of this method and follow them in order to obtain reproducible and accurate data.

3.0 Definitions

3.1 Measurement System. The total equipment required for the determination of gas concentration. The measurement system consists of the following major subsystems:

3.1.1 Sample Interface. That portion of a system used for one or more of the following: sample acquisition, sample transport, sample conditioning, or protection of the analyzers from the effects of the stack gas.

3.1.2 Gas Analyzer. That portion of the system that senses the gas to be measured and generates an output proportional to its concentration.

3.1.3 Data Recorder. A strip chart recorder, analog computer, or digital recorder for recording measurement data from the analyzer output.

3.2 Span. The upper limit of the gas concentration measurement range displayed on the data recorder.

3.3 Calibration Gas. A known concentration of a gas in an appropriate diluent gas (i.e., $N_2$).

3.4 Analyzer Calibration Error. The difference between the gas concentration exhibited by the gas analyzer and the known concentration of the calibration gas when the calibration gas is introduced directly to the analyzer.

3.5 Sampling System Bias. The sampling system bias is the difference between the gas concentrations exhibited by the measurement system when a known concentration gas is introduced at the outlet of the sampling probe and the known value of the calibration gas.

3.6 Response Time. The amount of time required for the measurement system to display 95 percent of a step change in gas concentration on the data recorder.

3.7 Calibration Curve. A graph or other systematic method of establishing the relationship between the analyzer response and the actual gas concentration introduced to the analyzer.

3.8 Linearity. The linear response of the analyzer or test system to known calibration inputs covering the concentration range of the system.

3.9 Interference Rejection. The ability of the system to reject the effect of interferences in the analytical measurement processes of the test system.

4.0 Interferences

4.1 Sampling System Interferences. An important consideration in measuring HCl using an extractive measurement system is to ensure that a representative kiln gas sample is delivered to the gas analyzer. A sampling system interferant is a factor that inhibits an analyte from reaching the analytical instrumentation. Condensed water vapor is a strong sampling system interferant for HCl and other water soluble compounds.

"Cold spots" in the sampling system can allow water vapor in the sample to condense resulting in removal of HCl from the sample stream. The extent of HCl sampling system bias depends on concentrations of potential interferants, moisture content of the gas stream, temperature of the gas stream, temperature of sampling system components, sample flow rate, and reactivity of HCl with other species in the gas stream. For measuring HCl in a wet gas stream, the temperatures of the gas stream and sampling system components and the sample flow rate are of primary importance. In order to prevent problems with condensation in the sampling system, these parameters must be closely monitored.

4.1.1 System Calibration Checks. Performing these calibration checks where HCl calibration gas is injected through the entire system both before and after each test run demonstrates the integrity of the sampling system and capability of the analyzer for measuring this water soluble and otherwise unstable compound under ideal conditions (i.e., HCl in $N_2$).

4.1.2 Analyte Spiking Checks. For analyte spiking checks, HCl calibration gas is quantitatively added to the sample stream at a point upstream of the particulate filter and all other sample handling components both before and after each test run. The volume of HCl spike gas should not exceed 10 percent of the total sample volume so that the sample matrix is relatively unaffected. Successfully performing these checks demonstrates the integrity of the sampling system for measuring this water soluble and reactive compound under actual sample matrix conditions. Successfully performing these checks also demonstrates the adequacy of the interference rejection capability of the analyzer. (See section 9.3 of this method.)

4.2 Analytical Interferences. Analytical interferences are reduced by the GFC spectroscopic technique required by the method. The accuracy of HCl measurements provided by some GFC analyzers is known to be sensitive to the moisture content of the sample. This must be taken into account in order to acquire accurate results. These analyzers must be calibrated for the specific moisture content of the samples.

## 5.0 Safety

This method may involve sampling at locations having high positive or negative pressures, or high concentrations of hazardous or toxic pollutants, and cannot address all safety problems encountered under these diverse sampling conditions. It is the responsibility of the tester(s) to ensure proper safety and health practices, and to determine the applicability of regulatory limitations before performing this test method. Because HCl is a respiratory irritant, it is advisable to limit exposure to this compound.

## 6.0 Equipment and Supplies

Note: Mention of company or product names does not constitute endorsement by the U. S. Environmental Protection Agency.

6.1 Measurement System. Use any GFC measurement system for HCl that meets the specifications of this method. All sampling system components must be maintained above the kiln gas temperature, when possible, or at least 350°F. The length of sample transport line should be minimized and sampling rate should be as high as possible to minimize adsorption of HCl. The essential components of the measurement system are described in sections 6.1.1 through 6.1.12.

6.1.1 Sample Probe. Glass, stainless steel Hastalloy™, or equivalent, of sufficient length to traverse the sample points. The sampling probe shall be heated to a minimum of 350°F to prevent condensation. Dilution extractive systems must use a dilution ratio such that the average diluted concentrations are between 25 to 75 percent of the selected measurement range of the analyzer.

6.1.2 Calibration Valve Assembly. Use a heated, three-way valve assembly, or equivalent, for selecting either sample gas or introducing calibration gases to the measurement system or introducing analyte spikes into the measurement system at the outlet of the sampling probe before the primary particulate filter.

6.1.3 Particulate Filter. A coarse filter or other device may be placed at the inlet of the probe for removal of large particulate (10 microns or greater). A heated (Balston® or equivalent) filter rated at 1 micron is necessary for primary particulate removal, and shall be pla[illegible]y after the heated probe. The filter/filter holder shall be maintained at 350°F or a higher temperature. Additional filters at the inlet of the gas analyzer may be used to prevent accumulation of particulate material in the measurement system and extend the useful life of components. All filters shall be fabricated of materials that are nonreactive with HCl. Some types of glass filters are known to react with HCl.

6.1.4 Sample Transport Lines. Stainless steel or polytetrafluoroethylene (PTFE) tubing shall be heated to a minimum temperature of 350°F (sufficient to prevent condensation and to prevent HCl and $NH_3$ from combining into ammonium chloride in the sampling system) to transport the sample gas to the gas analyzer.

6.1.5 Sample Pump. Use a leak-free pump to pull the sample gas through the system at a flow rate sufficient to minimize the response time of the measurement system. The pump components that contact the sample must be heated to a temperature greater than 350°F and must be constructed of a material that is nonreactive to HCl.

6.1.6 Sample Flow Rate Control. A sample flow rate control valve and rotameter, or equivalent, must be used to maintain a constant sampling rate within ±10 percent. These components must be heated to a temperature greater than 350°F. (<u>Note</u>: The tester may elect to install a back-pressure regulator to maintain the sample gas manifold at a constant pressure in order to protect the analyzer(s) from over-pressurization, and to minimize the need for flow rate adjustments.)

6.1.7 Sample Gas Manifold. A sample gas manifold, heated to a minimum of 350°F, is used to divert a portion of the sample gas stream to the analyzer and the remainder to the by-pass discharge vent. The sample gas manifold should also include provisions for introducing calibration gases directly to the analyzer. The manifold must be constructed of material that is nonreactive to the gas being sampled.

6.1.8 Gas Analyzer. Use a nondispersive infrared analyzer utilizing the gas filter correlation technique to determine HCl concentrations. The analyzer shall meet the applicable performance specifications of section 8.0 of this method. (Note: Housing the analyzer in a clean, thermally-stable, vibration free environment will minimize drift in the analyzer calibration.) The analyzer (system) shall be designed so that the response of a known calibration input shall not deviate by more than ±3 percent from the expected value. The analyzer or measurement system manufacturer may provide documentation that the instrument meets this design requirement. Alternatively, a known concentration gas standard and calibration dilution system meeting the requirements of Method 205 of appendix M to part 51 of this chapter, "Verification of Gas Dilution Systems for Field Calibrations" (or equivalent procedure), may be used to develop a multi-point calibration curve over the measurement range of the analyzer.

6.1.9 Gas Regulators. Single stage regulator with cross purge assembly that is used to purge the CGA fitting and regulator before and after use. (This purge is necessary to clear the calibration gas delivery system of ambient water vapor after the initial connection is made, or after cylinder changeover, and will extend the life of the regulator.) Wetted parts are 316 stainless steel to handle corrosive gases.

6.1.10 Data Recorder. A strip chart recorder, analog computer, or digital recorder, for recording measurement data. The data recorder resolution (i.e., readability) shall be 0.5 percent of span. Alternatively, a digital or analog meter having a resolution of 0.5 percent of span may be used to obtain the analyzer responses and the readings may be recorded manually. If this alternative is used, the readings shall be obtained at equally-spaced intervals over the duration of the sampling run. For sampling run durations of less than 1 hour, measurements at 1-minute intervals or a minimum of 30 measurements, whichever is less restrictive, shall be obtained. For sampling run durations greater than 1 hour, measurements at 2-minute intervals or a minimum of 96 measurements, whichever is less restrictive, shall be obtained.

6.1.11 Mass Flow Meters/Controllers. A mass flow meter having the appropriate calibrated range and a stated accuracy of

±2 percent of the measurement range is used to measure the HCl spike flow rate. This device must be calibrated with the major component of the calibration spike gas (e.g., nitrogen) using an NIST traceable bubble meter or equivalent. When spiking HCl, the mass flow meter/controller should be thoroughly purged before and after introduction of the gas to prevent corrosion of the interior parts.

6.1.12 System Flow Measurement. A measurement device or procedure to determine the total flow rate of sample gas within the measurement system. A rotameter, or mass flow meter calibrated relative to a laboratory standard to within ±2 percent of the measurement value at the actual operating temperature, moisture content, and sample composition (molecular weight) is acceptable. A system which ensures that the total sample flow rate is constant within ±2 percent and which relies on an intermittent measurement of the actual flow rate (e.g., calibrated gas meter) is also acceptable.

6.2 HCl Calibration Gases. The calibration gases for the gas analyzer shall be HCl in $N_2$. Use at least three calibration gases as specified below:

6.2.1 High-Range Gas. Concentration equivalent to 80 to 100 percent of the span.

6.2.2 Mid-Range Gas. Concentration equivalent to 40 to 60 percent of the span.

6.2.3 Zero Gas. Concentration of less than 0.25 percent of the span. Purified ambient air may be used [illegible] the zero gas by passing air through a charcoal filter or through one or more impingers containing a solution of 3 percent $H_2O_2$.

6.2.4 Spike Gas. A calibration gas of known concentration (typically 100 to 200 ppm) used for analyte spikes in accordance with the requirements of section 9.3 of this method.

7.0 Reagents and Standards

7.1 Hydrogen Chloride. Hydrogen Chloride is a reactive gas and is available in steel cylinders from various commercial gas vendors. The stability is such that it is not possible to purchase a cylinder mixture whose HCl concentration can be certified at better than ±5 percent. The stability of the cylinder may be monitored over time by periodically analyzing cylinder samples. The cylinder gas concentration must be verified within 1 month prior to the use of the calibration gas. Due to the relatively high uncertainty of HCl calibration gas values, difficulties may develop in meeting the performance specifications if the mid-range and high-range calibration gases are not consistent with each other. Where problems are encountered, the consistency of the test gas standards may be determined: (1) by comparing analyzer responses for the test gases with the responses to additional certified calibration gas standards, (2) by reanalysis of the calibration gases in accordance with sections 7.2.1 or 7.2.2 of this method, or (3) by other procedures subject to the approval of EPA.

7.2 Calibration Gas Concentration Verification. There are two alternatives for establishing the concentrations of calibration gases. Alternative No. 1 is preferred.

7.2.1 Alternative No. 1. The value of the calibration gases may be obtained from the vendor's certified analysis within 1 month prior to the test. Obtain a certification from the gas manufacturer that identifies the analytical procedures and date of certification.

7.2.2 Alternative No. 2. Perform triplicate analyses of the gases using Method 26 of appendix A to part 60 of this chapter. Obtain gas mixtures with a manufacturer's tolerance not to exceed ±5 percent of the tag value. Within 1 month of the field test, analyze each of the calibration gases in triplicate using Method 26 of appendix A to part 60 of this chapter. The tester must follow all of the procedures in Method 26 (e.g., use midget impingers, heated Pallflex TX40H175 filter (TFE-glass mat), etc. if this analysis is performed. Citation 3 in section 13 of this method describes procedures and techniques that may be used for this analysis. Record the results on a data sheet. Each of the individual HCl analytical results for each calibration gas shall be within 5 percent (or 5 ppm, whichever is greater) of the triplicate set average; otherwise, discard the entire set and repeat the triplicate analyses. If the average of the triplicate analyses is within 5 percent of the calibration gas manufacturer's cylinder tag value, use the tag value; otherwise, conduct at least three additional analyses until the results of six consecutive runs agree within 5 percent (or 5 ppm, whichever is greater) of the average. Then use this average for the cylinder value.

7.3 Calibration Gas Dilution Systems. Sample flow rates of approximately 15 L/min are typical for extractive HCl measurement systems. These flow rates coupled with response times of 15 to 30 minutes will result in consumption of large quantities of calibration gases. The number of cylinders and amount of calibration gas can be reduced by the use of a calibration gas dilution system in accordance with Method 205 of appendix M to part 51 of this chapter, "Verification of Gas Dilution Systems for Field Instrument Calibrations." If this option is used, the tester shall also introduce an undiluted calibration gas approximating the effluent HCl concentration during the initial calibration error test of the measurement system as a quality assurance check.

## 8.0 Test System Performance Specifications

8.1 Analyzer Calibration Error. This error shall be less than ±5 percent of the emission standard concentration or ±1 ppm,(whichever is greater) for zero, mid-, and high-range gases.

8.2 Sampling System Bias. This bias shall be less than ±7.5 percent of the emission standard concentration or ±1.5 ppm (whichever is greater) for zero and mid-range gases.

8.3 Analyte Spike Recovery. This recovery shall be between

70 to 130 percent of the expected concentration of spiked samples calculated with the average of the before and after run spikes.

## 9.0 Sample Collection, Preservation, and Storage

9.1 Pretest. Perform the procedures of sections 9.1.1 through 9.1.3.3 of this method before measurement of emissions (procedures in section 9.2 of this method). It is important to note that after a regulator is placed on an HCl gas cylinder valve, the regulator should be purged with dry $N_2$ or dry compressed air for approximately 10 minutes before initiating any HCl gas flow through the system. This purge is necessary to remove any ambient water vapor from within the regulator and calibration gas transport lines; the HCl in the calibration gas may react with this water vapor and increase system response time. A purge of the system should also be performed at the conclusion of a test day prior to removing the regulator from the gas cylinder. Although the regulator wetted parts are corrosion resistant, this will reduce the possibility of corrosion developing within the regulator and extend the life of the equipment.

9.1.1 Measurement System Preparation. Assemble the measurement system by following the manufacturer's written instructions for preparing and preconditioning the gas analyzer and, as applicable, the other system components. Introduce the calibration gases in any sequence, and make all necessary adjustments to calibrate the analyzer and the data recorder. If necessary, adjust the instrument for the specific moisture content of the samples. Adjust system components to achieve correct sampling rates.

9.1.2 Analyzer Calibration Error. Conduct the analyzer calibration error check in the field by introducing calibration gases to the measurement system at any point upstream of the gas analyzer in accordance with sections 9.1.2.1 and 9.1.2.2 of this method.

9.1.2.1 After the measurement system has been prepared for use, introduce the zero, mid-range, and high-range gases to the analyzer. During this check, make no adjustments to the system except those necessary to achieve the correct calibration gas flow rate at the analyzer. Record the analyzer responses to each calibration gas. Note: A calibration curve established prior to the analyzer calibration error check may be used to convert the analyzer response to the equivalent gas concentration introduced to the analyzer. However, the same correction procedure shall be used for all effluent and calibration measurements obtained during the test.

9.1.2.2 The analyzer calibration error check shall be considered invalid if the difference in gas concentration displayed by the analyzer and the concentration of the calibration gas exceeds ±5 percent of the emission standard concentration or ±1 ppm, (whichever is greater) for the zero, mid-, or high-range calibration gases. If an invalid calibration is exhibited, cross-check or recertify the calibration gases, take corrective action, and repeat the analyzer calibration error check until acceptable performance is achieved.

9.1.3 Sampling System Bias Check. For nondilution extractive systems, perform the sampling system bias check by introducing calibration gases either at the probe inlet or at a calibration valve installed at the outlet of the sampling probe. For dilution systems, calibration gases for both the analyzer calibration error check and the sampling system bias check must be introduced prior to the point of sample dilution. For dilution and nondilution systems, a zero gas and either a mid-range or high-range gas (whichever more closely approximates the effluent concentration) shall be used for the sampling system bias check.

9.1.3.1 Introduce the upscale calibration gas, and record the gas concentration displayed by the analyzer. Then introduce zero gas, and record the gas concentration displayed by the analyzer. During the sampling system bias check, operate the system at the normal sampling rate, and make no adjustments to the measurement system other than those necessary to achieve proper calibration gas flow rates at the analyzer. Alternately introduce the zero and upscale gases until a stable response is achieved. The tester shall determine the measurement system response time by observing the times required to achieve a stable response for both the zero and upscale gases. Note the longer of the two times and note the time required for the measurement system to reach 95 percent of the step change in the effluent concentration as the response time.

9.1.3.2 For nondilution systems, where the analyzer calibration error test is performed by introducing gases directly to the analyzer, the sampling system bias check shall be considered invalid if the difference between the gas concentrations displayed by the measurement system for the sampling system bias check and the known gas concentration standard exceeds ±7.5 percent of the emission standard or ±1.5 ppm, (whichever is greater) for either the zero or the upscale calibration gases. If an invalid calibration is exhibited, take corrective action, and repeat the sampling system bias check until acceptable performance is achieved. If adjustment to the analyzer is required, first repeat the analyzer calibration error check, then repeat the sampling system bias check.

9.1.3.3 For dilution systems (and nondilution systems where all calibration gases are introduced at the probe), the comparison of the analyzer calibration error results and sampling system bias check results is not meaningful. For these systems, the sampling system bias check shall be considered invalid if the difference between the gas concentrations displayed by the analyzer and the actual gas concentrations exceed ±7.5 percent of the emission standard or ±1.5 ppm, (whichever is greater) for either the zero or the upscale calibration gases. If an invalid calibration is exhibited, take corrective action, and repeat the sampling system bias check until acceptable performance is achieved. If adjustment to the analyzer is required, first repeat the analyzer calibration error check.

### 9.2 Emission Test Procedures

9.2.1 Selection of Sampling Site and Sampling Points. Select a measurement site and sampling points using the same criteria that are applicable to Method 26 of appendix A to part 60 of this chapter.

9.2.2 Sample Collection. Position the sampling probe at the first measurement point, and begin sampling at the same rate as used during the sampling system bias check. Maintain constant rate sampling (i.e., ±10 percent) during the entire run. Field test experience has shown that conditioning of the sample system is necessary for approximately 1-hour prior to conducting the first sample run. This conditioning period should be repeated after particulate filters are replaced and at the beginning of each new day or following any period when the sampling system is inoperative. Experience has also shown that prior to adequate conditioning of the system, the response to analyte spikes and/or the change from an upscale calibration gas to a representative effluent measurement may be delayed by more than twice the normal measurement system response time. It is recommended that the analyte spikes (see section 9.3 of this method) be performed to determine if the system is adequately conditioned. The sampling system is ready for use when the time required for the measurement system to equilibrate after a change from a representative effluent measurement to a representative spiked sample measurement approximates the calibration gas response time observed in section 9.1.3.1 of this method.

9.2.3 Sample Duration. After completing the sampling system bias checks and analyte spikes prior to a test run, constant rate sampling of the effluent should begin. For each run, use only those measurements obtained after all residual response to calibration standards or spikes are eliminated and representative effluent measurements are displayed to determine the average effluent concentration. At a minimum, this requires that the response time of the measurement system has elapsed before data are recorded for calculation of the average effluent concentration. Sampling should be continuous for the duration of the test run. The length of data collection should be at least as long as required for sample collection by Method 26 of part 60 of this chapter. One hour sampling runs using this method have provided reliable data for cement kilns.

9.2.4 Validation of Runs. Before and after each run, or if adjustments are necessary for the measurement system during the run, repeat the sampling system bias check procedure described in section 9.1.3 of this method. (Make no adjustments to the measurement system until after the drift checks are completed.)

Record the analyzer's responses.

9.2.4.1 If the post-run sampling system bias for either the zero or upscale calibration gas exceeds the sampling system bias specification, then the run is considered invalid. Take corrective action, and repeat both the analyzer calibration error check procedure (section 9.1.2 of this method) and the sampling system bias check procedure (section 9.1.3 of this method) before repeating the run.

9.2.4.2 If the post-run sampling system bias for both the zero and upscale calibration gas are within the sampling system bias specification, then construct two 2-point straight lines, one using the pre-run zero and upscale check values and the other using the post-run zero and upscale check values. Use the slopes and y-intercepts of the two lines to calculate the gas concentration for the run in accordance with equation 1 of this method.

9.3 Analyte Spiking—Self-Validating Procedure. Use analyte spiking to verify the effectiveness of the sampling system for the target compounds in the actual kiln gas matrix. Quality assurance (QA) spiking should be performed before and after each sample run. The spikes may be performed following the sampling system bias checks (zero and mid-range system calibrations) before each run in a series and also after the last run. The HCl spike recovery should be within ±30 percent as calculated using equations 1 and 2 of this method. Two general approaches are applicable for the use of analyte spiking to validate a GFC HCl measurement system: (1) two independent measurement systems can be operated concurrently with analyte spikes introduced to one of the systems, or (2) a single measurement system can be used to analyze consecutively, spiked and unspiked samples in an alternating fashion. The two-system approach is similar to Method 301 of this appendix and the measurement bias is determined from the difference in the paired concurrent measurements relative to the amount of HCl spike added to the spiked system. The two-system approach must employ identical sampling systems and analyzers and both measurement systems should be calibrated using the same mid- and high-range calibration standards. The two-system approach should be largely unaffected by temporal variations in the effluent concentrations if both measurement systems achieve the same calibration responses and both systems have the same response times. (See Method 301 of this appendix for appropriate calculation procedures.) The single measurement system approach is applicable when the concentration of HCl in the source does not vary substantially during the period of the test. Since the approach depends on the comparison of consecutive spiked and unspiked samples, temporal variations in the effluent HCl concentrations will introduce errors in determining the expected concentration of the spiked samples. If the effluent HCl concentrations vary by more than ±10 percent (or ±5 ppm, whichever is greater) during the time required to obtain and equilibrate a new sample (system response time), it may be necessary to: (1) use a dual sampling system approach, (2) postpone testing until stable emission concentrations are achieved, (3) switch to the two-system approach [if possible] or, (4) rely on alternative QA/QC procedures. The dual-sampling system alternative uses two sampling lines to convey sample to the gas distribution manifold. One of the sample lines is used to continuously extract unspiked kiln gas from the source. The other sample line serves as the analyte spike line. One GFC analyzer can be used to alternately measure the HCl concentration from the two sampling systems with the need to purge only the components between the common manifold and the analyzer. This minimizes the time required to acquire an equilibrated sample of spiked or unspiked kiln gas. If the source varies by more than ±10 percent or ±5 ppm, (whichever is greater) during the time it takes to switch from the unspiked sample line to the spiked sample line, then the dual-sampling system alternative approach is not applicable. As a last option, (where no other alternatives can be used) a humidified nitrogen stream may be generated in the field which approximates the moisture content of the kiln gas. Analyte spiking into this humidified stream can be employed to assure that the sampling system is adequate for transporting the HCl to the GFC analyzer and that the analyzer's water interference rejection is adequate.

9.3.1 Spike Gas Concentration [illegible] The volume of HCl spike gas should not exceed 10 percent of the total sample volume (i.e., spike to total sample ratio of 1:10) to ensure that the sample matrix is relatively unaffected. An ideal spike concentration should approximate the native effluent concentration, thus the spiked sample concentrations would represent approximately twice the native effluent concentrations. The ideal spike concentration may not be achieved because the native HCl concentration cannot be accurately predicted prior to the field test, and limited calibration gas standards will be available during the field test. Some flexibility is available by varying the spike ratio over the range from 1:10 to 1:20. Practical constraints must be applied to allow the tester to spike at an anticipated concentration. Thus, the tester may use a 100 ppm calibration gas and a spike ratio of 1:10 as default values where information regarding the expected HCl effluent concentration is not available prior to the tests. Alternatively, the tester may select another calibration gas standard and/or lower spike ratio (e.g., 1:20) to more closely approximate the effluent HCl concentration.

9.3.2 Spike Procedure. Introduce the HCl spike gas mixture at a constant flow rate (±2 percent) at less than 10 percent of the total sample flow rate. (For example, introduce the HCl spike gas at 1 L/min (±20 cc/min) into a total sample flow rate of 10 L/min). The spike gas must be preheated before introduction into the sample matrix to prevent a localized condensation of the gas stream at the spike introduction point. A heated sample transport line(s) containing multiple transport tubes within the heated bundle may be used to spike gas up through the sampling system to the spike introduction point. Use a calibrated flow device (e.g., mass flow meter/controller) to monitor the spike flow rate. Use a calibrated flow device (e.g., rotameter, mass flow meter, orifice meter, or other method) to monitor the total sample flow rate. Calculate the spike ratio from the measurements of spike flow and total flow. (See equation 2 and 3 in section 10.2 of this method.)

9.3.3 Analyte Spiking. Determine the approximate effluent HCl concentrations by examination of preliminary samples. For single-system approaches, determine whether the HCl concentration varies significantly with time by comparing consecutive samples for the period of time corresponding to at least twice the system response time. (For analyzers without sample averaging, estimate average values for two to five minute periods by observing the instrument display or data recorder output.) If the concentration of the individual samples varies by more than ±10 percent relative to the mean value or ±5 ppm, (whichever is greater), an alternate approach may be needed.

9.3.3.1 Adjust the spike flow rate to the appropriate level relative to the total flow by metering spike gas through a calibrated mass flow meter or controller. Allow spike flow to equilibrate within the sampling system for at least the measurement system response time and a steady response to the spike gas is observed before recording response to the spiked gas sample. Next, terminate the spike gas flow and allow the measurement system to sample only the effluent. After the measurement system response time has elapsed and representative effluent measurements are obtained, record the effluent unspiked concentration. Immediately calculate the spike recovery.

9.3.3.2 If the spike recovery is not within acceptable limits and a change in the effluent concentration is suspected as the cause for exceeding the recovery limit, repeat the analyte spike procedure without making any adjustments to the analyzer or sampling system. If the second spike recovery falls within the recovery limits, disregard the first attempt and record the results of the second spike.

9.3.3.3 Analyte spikes must be performed before and after each test run. Sampling system bias checks must also be performed before and after each test run. Depending on the particular sampling strategy and other constraints, it may be necessary to compare effluent data either immediately before or immediately after the spike sample to determine the spike recovery. Either method is acceptable provided a consistent approach is used for the test program. The average spike recovery for the pre- and post-run spikes shall be used to determine if spike recovery is between 70 and 130 percent.

## 10.0 Data Analysis and Emission Calculations

The average gas effluent concentration is determined from the average gas concentration displayed by the gas analyzer and is adjusted for the zero and upscale sampling system bias checks, as determined in accordance with section 9.2.3 of this method. The average gas concentration displayed by the analyzer may be determined by integration of the area under the curve for chart recorders, or by averaging all of the effluent measurements. Alternatively, the average may be calculated from measurements recorded at equally spaced intervals over the entire duration of the run. For sampling run durations of less than 1-hour, average measurements at 2-minute intervals or less, shall be used. For sampling run durations greater than 1-hour, measurements at 2-minute intervals or a minimum of 96 measurements, whichever is less restrictive, shall be used. Calculate the effluent gas concentration using equation 1.

$$C_{gas} = \frac{(m_i + m_f)\left[\frac{(C_{avg} - b_c)}{m_c}\right] + (b_i + b_f)}{2} \qquad \text{(Eq. 1)}$$

where:

$b_c$ = Y-intercept of the calibration least-squares line.

$b_f$ = Y-intercept of the final bias check 2-point line.

$b_i$ = Y-intercept of the initial bias check 2-point line.

$C_{gas}$ = Effluent gas concentration, as measured, ppm.

$C_{avg}$ = Average gas concentration indicated by gas analyzer, as measured, ppm.

$m_c$ = Slope of the calibration least-squares line.

$m_f$ = Slope of the final bias check 2-point line.

$m_i$ = Slope of the initial bias check 2-point line.

The following equations are used to determine the percent recovery (%R) for analyte spiking:

$$\%R = (S_M/C_E) \times 100 \qquad \text{(Eq. 2)}$$

where:

$S_M$ = Mean concentration of duplicate analyte spiked samples (observed).

$C_E$ = Expected concentration of analyte spiked samples (theoretical).

$$C_E = C_S(Q_S/Q_T) + S_U(1-Q_S/Q_T) \qquad \text{(Eq. 3)}$$

where:

$C_S$ = Concentration of HCl spike gas (cylinder tag value).

$Q_S$ = Spike gas flow rate.

$Q_T$ = Total sample flow rate (effluent sample flow plus spike flow).

$S_U$ = Native concentration of HCl in unspiked effluent samples.

Acceptable recoveries for analyte spiking are ±30 percent.

## 11.0 Pollution Prevention

Gas extracted from the source and analyzed or vented from the system manifold shall be either scrubbed, exhausted back into the stack, or discharged into the atmosphere where suitable dilution can occur to prevent harm to personnel health and welfare or plant or personal property.

## 12.0 Waste Management

Gas standards of HCl are handled as according to the instructions enclosed with the materials safety data sheets.

## 13.0 References

1. Peeler, J.W., Summary Letter Report to Ann Dougherty, Portland Cement Association, June 20, 1996.

2. Test Protocol, Determination of Hydrogen Chloride Emissions from Cement Kilns (Instrumental Analyzer Procedure) Revision 4; June 20, 1996.

3. Westlin, Peter R. and John W. Brown. Methods for Collecting and Analyzing Gas Cylinder Samples. Source Evaluation Society Newsletter. 3(3):5-15. September 1978.

# APPENDIX H

# PROJECT PARTICIPANTS

# PROJECT PARTICIPANTS

| **Affiliation** | **Name** | **Responsibility** |
|---|---|---|
| USEPA | Joe Wood, ESD | Environmental Engineer |
| | Michael L. Toney, EMC | Work Assignment Manager |
| Pacific Environmental Services, Inc. | Franklin Meadows | Project Manager |
| | Michael D. Maret | Field Team Leader |
| | Gary Gay | Site Leader/Console Operator |
| | Paul Siegel | Sampling Technician |
| | Troy Abernathy | Sample Recovery |
| Atlantic Technical Services (PES Subcontractor) | Emil Stewart | Data Reduction |
| APCC, Ltd. (PES Subcontractor) | Aaron R. Christie | CEM Team Leader |
| | Peter Day | CEM Sampling Technician |
| Research Triangle Institute (EPA/ESD Contractor) | Cybele M. Brockmann | Process Coordinator |

# TECHNICAL REPORT DATA

Please read instructions on the reverse before completing

1. REPORT NO.
EPA-454/R-00-032

2.

3. RECIPIENT'S ACCESSION NO.

4. TITLE AND SUBTITLE
Final Report
Manual and Continuous Emissions Testing
Kiln No. 3 Scrubber Stack
Huron Lime
Huron, Ohio

5. REPORT DATE
July 2000

6. PERFORMING ORGANIZATION CODE

7. AUTHOR(S)
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8. PERFORMING ORGANIZATION REPORT NO.

9. PERFORMING ORGANIZATION NAME AND ADDRESS
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10. PROGRAM ELEMENT NO.

11. CONTRACT/GRANT NO.
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U.S. Environmental Protection Agency
Office of Air Quality Planning and Standards
Emissions, Monitoring and Analysis Division
Research Triangle Park, North Carolina 27711

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15. SUPPLEMENTARY NOTES

16. ABSTRACT

The United States Environmental Protection Agency (EPA) Emission Standards Division (ESD) is investigating the lime manufacturing industry to identify and quantify hazardous air pollutants (HAPs) emitted from lime kilns. ESD requested that EPA's Emissions, Monitoring and Analysis Division (EMAD) conduct the required testing. EMAD issued a work assignment to Pacific Environmental Services, Inc. (PES) to conduct a "screening" test to collect air emissions data as specified in the ESD test request. The primary objective of the testing program was to characterize HAP emissions from a rotary lime kiln located at the Huron Lime Company's Huron, Ohio facility. Based on the pollutant concentrations and emissionrates calculated from the results of the screening tests, the kiln may be selected by EPA for further testing.

The tests were conducted to quantify the controlled air emissions of polychlorinated dibenzo-*p*-dioxins and polyclorinated dibenzofurans (PCDDs/PCDFs) from the Kiln No. 3 scrubber stack. Concurrent with the PCDDs/PCDFs testing, sampling was conducted at the stack breeching to determine concentrations of oxygen ($O_2$), carbon dioxide ($CO_2$), and total hydrocarbons (THC).

During the testing program another EPA contractor monitored and recorded process and emission control system operating parameters.

This report consists of one volume totaling 440 pages.

17. KEY WORDS AND DOCUMENT ANALYSIS

| a. DESCRIPTIONS | b. IDENTIFIERS/OPEN ENDED TERMS | c. COASTI Field/Group |
|---|---|---|
| Dioxins/Furans<br>Hazardous Air Pollutants<br>Total Hydrocarbons<br>Wet Scrubber | | |

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