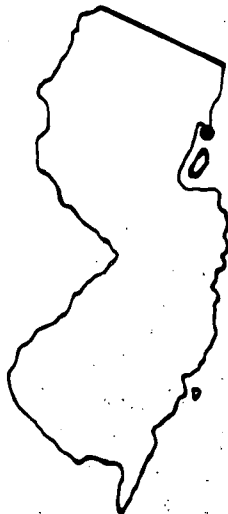


**THE NEW JERSEY DEPARTMENT
OF ENVIRONMENTAL PROTECTION**

**FEASIBILITY STUDY
SYNCON RESINS SITE**

**KEARNY
HUDSON COUNTY
NEW JERSEY**



**QUALITY ASSURANCE
PROJECT MANAGEMENT PLAN**

EBASCO

332094



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EBASCO SERVICES INCORPORATED

CONTROL DOCUMENT NO. Q-109

ASSIGNED TO Mr Russell Trice, Site Manager

Hazardous Site Mitigation Admin.

NJDEP

428 East State St, Trenton, NJ 08625

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EBASCO SERVICES INCORPORATED
QUALITY ASSURANCE PROJECT MANAGEMENT PLAN
FOR
FEASIBILITY STUDY
AT THE SYNCON RESINS SITE
KEARNY, HUDSON COUNTY, N.J.

Approved by:

<u>NAME</u>
<u>T GRANGER</u>
<u>J GUSHUE</u>
<u>R TRICE</u>

<u>TITLE</u>	<u>DATE</u>
EBASCO PROJECT MANAGER	5/13/85
EBASCO QUALITY ASSURANCE OFFICER	5/13/85
NJDEP PROJECT OFFICER	5/14/85

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List of all individuals receiving a copy of the Quality Assurance Project Management Plan.

Syncon Resins Project Manager, Ebasco Services Incorporated
Syncon Resins Site Manager, Ebasco Services Incorporated
Syncon Resins Quality Assurance Officer, Ebasco Services Incorporated
Syncon Resins Project Manager, Stabler-Reutter, Inc.
Syncon Resins Project Manager, VEP Associates, Inc.
Syncon Resins Project Manager, Empire Soils Investigations Inc.
Syncon Resins Project Manager, US Testing Company, Inc.

Syncon Resins Site Project Officer, State of New Jersey,
Department of Environmental Protection, Division of Waste Management
Syncon Resins Site Quality Assurance Officer, State of New Jersey,
Department of Environmental Protection, Division of Waste Management
Syncon Resins Site Project Officer, U.S. Environmental Protection
Agency-Region II
Syncon Resins Site Project Quality Assurance Officer, U.S. Environmental
Protection Agency-Region II

3. PROJECT DESCRIPTION

The Syncon Resins site encompasses about 15 acres along the Passaic River in an industrial area of Kearny, New Jersey. Syncon, which ceased operations in 1981, produced resin carriers for pigments, paints and varnish products, much of the time apparently by reprocessing off-specification resins from other manufacturers. While the over 12,000 drums of resins, solvents and other unknown substances previously stored on site have been removed from the site, the site still contains 144 storage tanks, two lagoons and five suspected underground storage tanks. A variety of hazardous substances were handled at the Syncon Resins site including solvents, waste oil, corrosives, organic liquids, solids, acids, alkalies, ketones, inorganic liquid and solids. Soil, shallow groundwater and surface water samples indicate the presence of various pollutants including toluene, xylene, PCBs, heavy metals, pesticides and cyanide.

The major concern remaining at the site appears to be the tanks and vessels of unknown characterization. Accordingly, the Ebasco approach includes a comprehensive program for identification and characterization of the hazard associated with these facilities. Ebasco will implement this program and document the findings in a report. This report will present recommendations on methods of removing the waste from the tanks, tank decontamination procedures and final disposal of the materials contained in the tanks and vessels.

A second area of concern is indicated by the strong odors reported throughout the site even after removal of the bulk of the 55-gallon drums. The emission of volatiles, and potentially the build-up of volatiles in quiescent areas of onsite structures, may pose a hazard to onsite workers during site investigation as well as offsite populations being exposed to these contaminants. This will require careful evaluation of the site and structures

through initial inspection and measurements made by Ebasco air quality personnel and by the Ebasco Health and Safety Officer who will advise project management and develop procedures and policies for site workers to ensure adequate protection and safe working practices. Ebasco's proposed air quality investigation will then be initiated to determine the extent and nature of possible air contamination for the site. After completion of this activity, Ebasco will present results and recommendations to the NJDEP for their decision as to the need to perform an investigation to determine the nature and extent of offsite air contamination.

The priority assigned these two issues is due to the imminent hazard presented by the site conditions as well as anticipated circumstances associated with the more conventional concern with groundwater and surface water contamination from hazardous waste sites. The Ebasco project teams will identify and assess any threat to public health and environment posed by the site due to these concerns and will develop sound remedial action alternatives to mitigate the threat. However, for the Syncon Resins site, the industrial nature of the area, the distance to the nearest residence, the distance and direction to the nearest known potable water source and the otherwise already deteriorated conditions of the Passaic River, reduce the relative urgency for resolving these issues.

In addition to the technical problems to be addressed, the issue of public concern and community relations will be addressed. Community relations are an integral component of a successful project effort, and Ebasco will assist NJDEP in the interaction between concerned public groups, interested local officials, agencies, and the NJDEP.

4. PROJECT ORGANIZATION AND RESPONSIBILITY

Conduct of the Syncon Resins Site Feasibility Study requires a project organization for efficient information and work order flow, to meet Health and Safety and Quality Assurance goals and to achieve the aims of the project.

The overall project organization for the Ebasco Team is shown on Figure 4-1. Key personnel and responsibilities are given in Tables 4-1 and 4-2.

The Project Manager is the overall manager of technical and administrative activities and is responsible for coordinating and scheduling of all project activities.

The Quality Assurance Officer is responsible for development and implementation of the Quality Assurance Project Management Plan (QAPMP), and conducts systems and performance audits and data quality reviews.

The Health and Safety Officer is responsible for advising the Project Staff on health and safety issues, conducting health and safety training sessions and monitoring the effectiveness of the health and safety program conducted in the field.

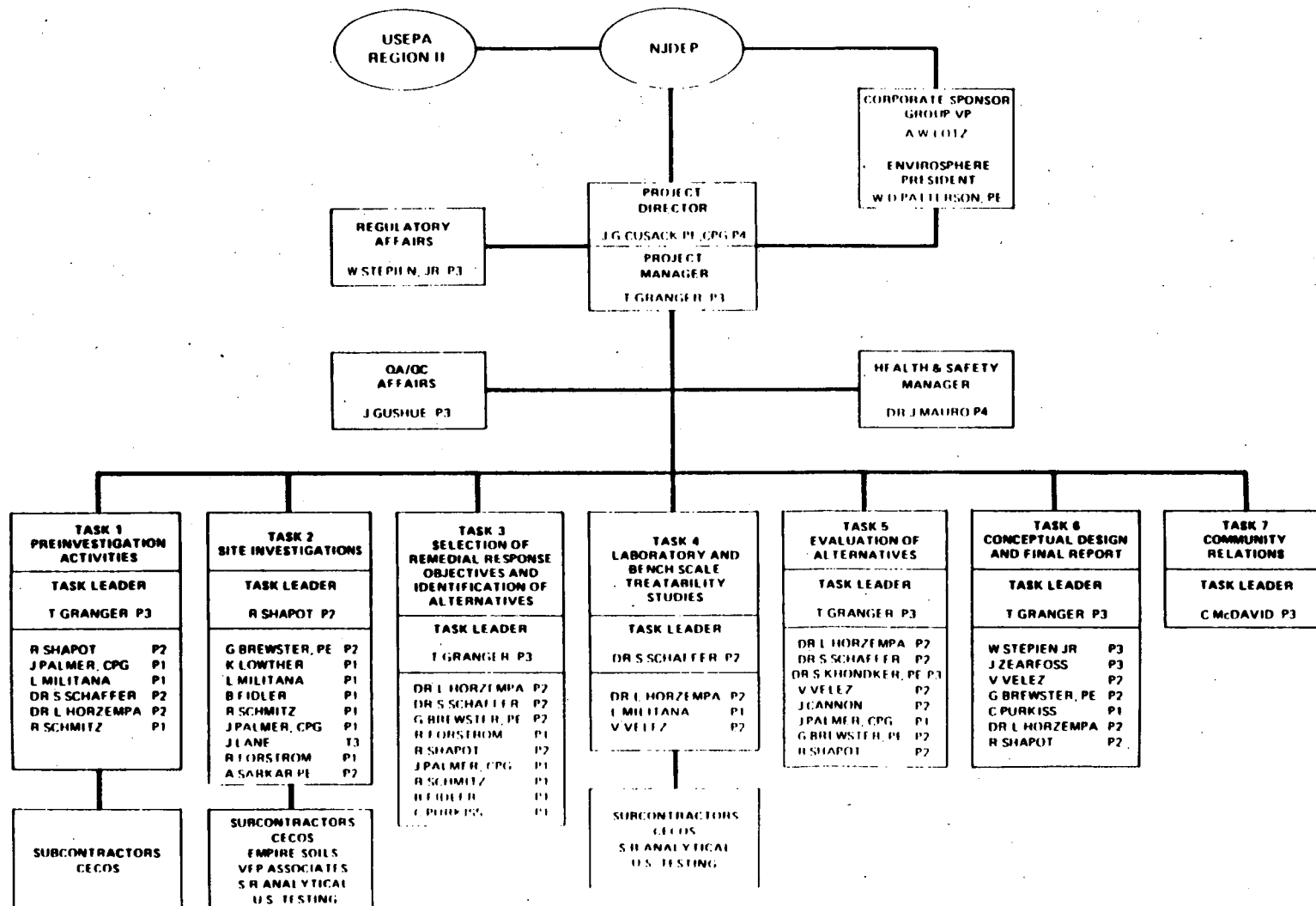
The Task Leaders are responsible for performance of their task(s) in conformance with the responsibilities delineated in the Contract between Ebasco and NJDEP. They will also ensure the soundness of the technical approach used by field personnel for collecting water, soil, waste, dust and air samples, and for reviewing the results of field activities to evaluate the integrity of the samples collected. Task Leaders, or an assigned task staff member, are also responsible for the Quality Control of sampling operations and data processing activities.

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Surveying, drilling, task sampling and laboratory analyses are the responsibilities of the assigned subcontractors. Quality Control of these operations is the responsibility of the subcontractor's supervisor, except for S-R Analytical for which the Laboratory Manager will have that responsibility.

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SYNCON RESINS SITE PROJECT ORGANIZATION



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TABLE 4-1
PROJECT PERSONNEL

<u>EBASCO</u>	<u>PERSONNEL</u>
PROJECT MANAGER	T GRANGER
HEALTH & SAFETY OFFICER	L MILITANA
REGULATORY AFFAIRS	W STEPIEN, JR
QUALITY ASSURANCE OFFICER	J GUSHUE
TASK 2 LEADER - SITE INVESTIGATIONS	R SHAPOT
TASK 4 LEADER - LABORATORY AND BENCH SCALE TREATABILITY STUDIES	DR. S SCHAFER
TASK 7 LEADER - COMMUNITY RELATIONS	C McDAVID
<u>SUBCONTRACTORS</u>	<u>PERSONNEL</u>
EMPIRE SOILS INVESTIGATIONS INC.	D ANDERSON
VEP ASSOCIATES (SURVEYING)	L HOYT
S-R ANALYTICAL INC. PROJECT MANAGER	C WARD
CECOS INC.	C MYERS
US TESTING COMPANY, INC.	E PATXOT

TABLE 4-2
RESPONSIBILITIES OF KEY INDIVIDUALS

<u>EBASCO</u>	<u>PERSONNEL</u>
OVERALL PROJECT COORDINATION	T GRANGER
OVERALL QA	J GUSHUE
SAMPLING OPERATIONS	R SHAPOT
SAMPLING QC	B FIDLER
LABORATORY ANALYSES	C WARD (S-R ANALYTICAL) A TORDINI (US TESTING)
LABORATORY QC	I LAMBERT (S-R ANALYTICAL) E PATXOT (US TESTING)
DATA PROCESSING ACTIVITIES	L HORZEMPA
DATA PROCESSING QC	S SCHAFER
DATA QUALITY REVIEW	J GUSHUE
PERFORMANCE AUDITING	J GUSHUE
SYSTEMS AUDITING	J GUSHUE
SURVEYING OPERATIONS	L HOYT
DRILLING OPERATIONS	D ANDERSON
TANK SAMPLING	C MYERS

5. QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA IN TERMS OF PRECISION, ACCURACY, COMPLETENESS, REPRESENTATIVENESS, AND COMPARABILITY

The quality assurance objective for measurement data is to ensure that environmental monitoring data of known and acceptable quality are provided. Field and laboratory data will be used for site assessments and hazard determinations, remedial investigations, engineering feasibility studies, community relations programs, and support of enforcement and cost recovery proceedings.

The quality assurance objective for requested data on the analytical results of the environmental samples collected will include:

Precision: The laboratory objective for precision is to equal or exceed the precision demonstrated for these analytical methods on similar samples, and shall be within the established control limits for the methods, as published by the Environmental Protection Agency (EPA).

Accuracy: The laboratory objective for accuracy is to equal or exceed the accuracy demonstrated for these analytical methods for each media (soil, water, air) of interest on similar samples, and shall be within the established control limits for the methods as published by EPA.

Representativeness: The representativeness of the data from the sampling sites depends on the sampling procedures. The representativeness of the analytical data is a function of the procedures used in processing the samples. The objective for representativeness is to provide data of the same high quality as other analyses of similar samples using the same methods during the same time period within the laboratory. Representativeness can be determined for this objective by a comparison of the quality control data for

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these samples against other data for similar samples analyzed at the same times.

Comparability: The results of these analyses can be compared with other analyses by other laboratories, because the objectives of the laboratory for comparability are: to demonstrate traceability of standards to National Bureau of Standards (NBS) or EPA sources; to use standard methodology; to apply appropriate levels of quality control within the context of the Laboratory Quality Assurance Program; and to participate in interlaboratory studies to document laboratory performance. By using traceable standards and standard methods, the analytical results can be compared to other laboratories operating similarly. The QA Program documents internal performance, and the interlaboratory studies document performance compared to other analysis at other locations.

Completeness: The completeness of an analysis is documented by including in the report sufficient information to allow the data user to assess the quality of the results. The information delivered will conform to current NJDEP data reporting requirements.

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6. SAMPLING PROCEDURES

6.1 General

The purpose of sampling is to obtain specimens that represent the situation being studied. Specific protocols for sampling have been developed for site investigations and are discussed in the Field Sampling Plan (FSP) for the Syncon Resins Site Feasibility. These protocols describe procedures for acquiring samples that best represent the environmental matrix, whether it be soil, sludge, water, or air. The trace level contamination of samples from external sources will be controlled through proper selection of sampling equipment as well as following good sampling techniques. Great care is required while using measuring devices, sampling devices, tubing or transfer pipes that come in contact with the matrix to be analyzed.

Appropriate randomization schemes will be used to obtain an unbiased representative of the population of interest. This means that the data acquired from the sample is related in all probability to all other samples that could be selected from the target population under specified conditions. Each sampling program has been planned in detail in the FSP. The sampling program in the FSP includes:

- o reasons for choosing sampling sites
- o description of sampling sites
- o number of samples
- o sampling methodologies
- o labeling requirements
- o container preparation
- o field blank preparation

- o sample preservation
- o storage, and
- o instructions for transport to the laboratory

The following discussion provides a generalized description of sampling procedures along with pertinent references.

6.2 Sampling Procedures

Sampling Procedures are discussed in detail in the FSP.

6.3 Sample Volume

The sample volume will depend on: a) the number of analyses to be performed, or b) the concentrations of the contaminants of interest.

The volume of samples for water, soil, air, and waste are established in the FSP.

6.4 Blanks

The following blanks will be collected and analyzed:

6.4.1 Field Blanks

On each day of sampling a field blank will be collected for each matrix sampled. This will indicate whether possible contamination has occurred with the sampling equipment. Soil field blanks will be subjected to volatile organic analysis only and, air and water field blanks will be subjected to priority organic contaminant analysis unless an exception is made to this requirement in the Field Sampling Plan.

SYN 001 1786

6.4.2 Trip Blanks

For each shipment of samples, trip blanks will be provided. Trip blanks for air, water, and soil samples will be analyzed for all priority pollutant organic compounds. This testing will be particularly valuable for methylene chloride quality control.

6.4.3 Method Blanks

To ensure that no contamination occurs during laboratory workup, The Analytical Laboratory will routinely process a method blank with all analytical runs.

6.5 Duplicates

6.5.1 Water/Soil/Sediment

Five percent of the water/soil/sediment analysis will be duplicated to indicate the precision of the methods used. Both water and soil duplicates will be analyzed for full priority pollutant analysis and forward library search.

6.5.2 Air Samples

At least ten percent of the gas and particulate phase analysis will be duplicated. Tenax and charcoal tubes will be analyzed by the gas phase analysis.

6.6 Spiked Samples

Five percent of the soil and water samples will be spiked with known quantities of the substances of interest before analysis to determine the percent recovery and indicate the general accuracy of the methods used.

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6.7 Split Samples

Whenever requested by the NJDEP Field Representative, samples will be split.

6.8 Sample Preservation

6.8.1 Objective

It is important to maintain the integrity of the samples from the time of collection until the analyses are performed. The samples should therefore be preserved during transportation and storage to prevent or retard degradation or modification of chemicals in samples.

6.8.2 Procedures

The preservation, holding times and the containers to be used will conform to the information given in Guidelines Establishing Test Procedures for the Analysis of Pollutants under the Clean Water Act, 40 CFR 136, October 26, 1984 (Table 1) and the Field Sampling Plan. The procedures for cleaning the glass and plastic containers and their caps are given in the Operating Procedures of the Analytical Laboratory.

TABLE 1 - REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Parameter No./Name	Container (1)	Preservation (2,3)	Maximum Holding Time (4)
INORGANIC TESTS:			
Acidity	P,G	Cool, 4°C	14 days
Alkalinity	P,G	Cool, 4°C	14 days
Ammonia	P,G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Biochemical Oxygen Demand	P,G	Cool, 4°C	48 hours
Bromide	P,G	None required	28 days
Biochemical Oxygen Demand, Carbonaceous	P,G	Cool, 4°C	48 hours
Chemical Oxygen Demand	P,G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Chloride	P,G	None required	28 days
Chlorine, Total Residual	P,G	None required	Analyze immediately
Color	P,G	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination	P,G	Cool, 4°C, NaOH to pH>12, 0.6g ascorbic acid(5)	14 days(6)
Fluoride	P	None required	28 days
Hardness	P,G	HNO ₃ to pH<2, H ₂ SO ₄ to pH<2	6 months
Hydrogen Ion (pH)	P,G	None required	Analyze immediately
Kjeldahl and Organic Nitrogen	P,G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Nitrate	P,G	Cool, 4°C	48 hours
Nitrate-Nitrite	P,G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Nitrite	P,G	Cool, 4°C	48 hours
Oil and Grease	G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Organic Carbon	P,G	Cool, 4°C, HCl or H ₂ SO ₄ to pH<2	28 days
Orthophosphate	P,G	Filter immediately, Cool, 4°C	48 hours
Oxygen, Dissolved-Probe	G Bottle and top	None required	Analyze immediately
Oxygen, Dissolved-Winkler	G Bottle and top	Fix on site and store in dark	8 hours
Phenols	G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Phosphorus (elemental)	G	Cool, 4°C	48 hours
Phosphorus, Total	P,G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Residue, Total	P,G	Cool, 4°C	7 days
Residue, Filterable	P,G	Cool, 4°C	48 hours
Residue, Nonfilterable (TSS)	P,G	Cool, 4°C	7 days
Residue, Settleable	P,G	Cool, 4°C	48 hours
Residue, Volatile	P,G	Cool, 4°C	7 days
Silica	P	Cool, 4°C	28 days
Specific Conductance	P,G	Cool, 4°C	28 days
Sulfate	P,G	Cool, 4°C	28 days
Sulfide	P,G	Cool, 4°C, add zinc acetate plus sodium hydroxide to pH>9	7 days
Sulfite	P,G	None required	Analyze immediately
Surfactants	P,G	Cool, 4°C	48 hours
Temperature	P,G	None required	Analyze immediately
Turbidity	P,G	Cool, 4°C	48 hours
METALS: (7)			
Chromium VI	P,G	Cool, 4°C	24 hours
Mercury	P,G	HNO ₃ to pH<2	28 days
Metals, except Chromium VI and Mercury	P,G	HNO ₃ to pH<2	6 months

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TABLE 1 - REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES (Continued)

Parameter No./Name	Container ⁽¹⁾	Preservation ^(2,3)	Maximum Holding Time ⁽⁴⁾
ORGANIC TESTS: ⁽⁸⁾			
Purgeable Halocarbons	G, Teflon-lined septum	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾	14 days
Purgeable Aromatic Hydrocarbons	G, Teflon-lined septum	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾ , HCl to pH(2)(9)	14 days
Acrolein and Acrylonitrile	G, Teflon-lined septum	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾ , adjust pH to 4-5(10)	14 days
Phenols ⁽¹¹⁾	G, Teflon-lined cap	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾	7 days until extraction, 40 days after extraction
Benzidines ⁽¹¹⁾	G, Teflon-lined cap	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾	7 days until extraction(13)
Phthalate Esters ⁽¹¹⁾	G, Teflon-lined cap	Cool, 4°C	7 days until extraction, 40 days after extraction
Nitrosamines ^(11,14)	G, Teflon-lined cap	Cool, 4°C, store in dark, 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾	7 days until extraction, 40 days after extraction
PCBs ⁽¹¹⁾ Acrylonitrile	G, Teflon-lined cap	Cool, 4°C	7 days until extraction, 40 days after extraction
Nitroaromatics and Isophorone ⁽¹¹⁾	G, Teflon-lined cap	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾ , store in dark	7 days until extraction, 40 days after extraction
Polynuclear Aromatic Hydrocarbons ⁽¹¹⁾	G, Teflon-lined cap	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾ , store in dark	7 days until extraction, 40 days after extraction
Halothane ⁽¹¹⁾	G, Teflon-lined cap	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾	7 days until extraction, 40 days after extraction
Chlorinated Hydrocarbons ⁽¹¹⁾	G, Teflon-lined cap	Cool, 4°C	7 days until extraction, 40 days after extraction
TCDF ⁽¹¹⁾	G, Teflon-lined cap	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾	7 days until extraction, 40 days after extraction
PESTICIDES TESTS:			
Pesticides ⁽¹¹⁾	G, Teflon-lined cap	Cool, 4°C, pH 5-9(15)	7 days until extraction, 40 days after extraction
RADIOLOGICAL TESTS:			
I-5 Alpha, beta and radium	P, G	HNO ₃ to pH(2)	6 months

TABLE 1 Notes

(1) Polyethylene (P) or Glass (G).

(2) Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

(3) When any sample is to be shipped by common carrier or sent through the United States Mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172).

(4) Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer time, and has received a variance from the Regional Administrator.

(5) Should only be used in the presence of residual chlorine.

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TABLE 1 Notes (Continued)

(6) Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

(7) Samples should be filtered immediately on-site before adding preservative for dissolved metals.

(8) Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

(9) Sample receiving no pH adjustment must be analyzed within seven days of sampling.

(10) The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.

(11) When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re: the requirement for thiosulfate reduction of residual chlorine) and footnotes 12, 13 (re: the analysis of benzidine).

(12) If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine.

(13) Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.

(14) For the analysis of diphenylnitrosamine add 0.008% $\text{Na}_2\text{S}_2\text{O}_3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.

(15) The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $\text{Na}_2\text{S}_2\text{O}_3$.

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7. SAMPLE CUSTODY

The history of each sample and its handling is documented from its collection through all transfers of custody until it is transferred to an analytical laboratory. Internal laboratory records then document the custody of the sample through its final disposition.

7.1 Sample

A "sample" is physical evidence collected from a facility or the environment. An essential part is the control of this evidence (i.e., sample) gathered from the facility or environment. To accomplish this, the following sample identification and chain-of-custody procedures will be followed.

7.2 Sample Identification

The method of identification of a sample depends on the type of measurement or analysis performed. When in situ measurements are made, the data are recorded directly in logbooks or Field Data Records (FDRs), with identifying information (project code, station numbers, station location, date, time, samplers), field observations, and remarks. Examples of in situ measurements include pH, temperature, conductivity, flow measurement, continuous air monitoring, and stack gas analysis.

Samples, other than in situ measurements, are identified by a sample label (see Figure 7-1).

These samples are removed and transported from the sample location to a laboratory or other location for analysis. Before removal, however, a sample is often separated into portions depending upon the analyses to be

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performed. Each portion is preserved in accordance with the Field Sampling Plan. The sample container is identified by a sample label. The information recorded on the sample label includes:

Project Name - Syncon Resins Site Feasibility Study

Sample Location - The sampling station description

Date -

Time - A four-digit number indicating the 24-hour time of collection - for example: 0954 is 9:54 am and 1629 is 4:29 pm

Type of Analysis -

Preservation Notes -

Sampling Technician- Name of the sampler

Media -

Sample Type -

Laboratory No. -

Remarks - Pertinent observations of the samplers

Lab # - May be completed by the receiving laboratory

The sample label contains an appropriate place for designating the sample as a grab or a composite and identifying the type of sample (air, water, sludge, etc.) collected for analyses. When used for air samples, the sampler may use the remarks section to designate the sequence number to identify the sample. The sample labels are attached to each sample or container.

After collection, separation, identification, and preservation, the sample is maintained under chain-of-custody procedures until it is in the custody of the Analytical Laboratory.

If the composite or grab sample is to be split, it is aliquoted into similar sample containers. Identical information is completed on the label attached to each split and one of these is marked "Split." In a similar fashion, labels will be marked for "Blank" or "Duplicate" samples.

7.3 Sample Preservation

The sample preservation is important to prevent or retard the degradation or modification of chemicals in samples during transportation and storage. The Field Sampling Plan (FSP) describes the preservation procedures for various compounds of interest.

7.4 Chain-of-Custody Procedures

The samples collected during a site investigation must be traceable from the time the samples are collected until they or their derived data are used in the final report. In order to maintain and document sample possession, the following chain-of-custody procedures is implemented.

7.4.1 Field Custody Procedures

- (a) Samples are collected as described in the Field Sampling Plan.
- (b) The field sampler is personally responsible for the care and custody of the samples collected until they are properly transferred or dispatched.
- (c) During sampling, blank samples will be prepared, as established in the Field Sampling Plan as appropriate (with and without preservatives), in the same type of containers used to hold the samples.

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- (d) Logbooks and other records are signed and dated.
- (e) When photographs are taken of the sampling as part of the documentation procedure, the name of the photographer, date, time, site location and site description are entered sequentially in the logbook as photos are taken. Once developed the photographic prints shall be serially numbered corresponding to the logbook descriptions.
- (f) Sample labels shall be completed for each sample, using waterproof ink unless prohibited by weather conditions, e.g., a logbook notation would explain that a pencil was used to fill out the sample label because a ballpoint pen would not function in freezing weather.
- (g) The Field Operations Leader determines whether proper custody procedures were followed during the field work and decides if additional samples are required.

7.4.2 Transfer of Custody and Shipment

- (a) Samples are accompanied by a Chain-of-Custody Record (see Figure 7-2). When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the Record. This Record documents sample custody transfer from the sampler, often through another person, to the analyst in the laboratory.
- (b) Samples will be packaged properly for shipment and dispatched to the appropriate laboratory for analysis, with a separate custody record accompanying each shipment (e.g., one for each field laboratory, one for samples shipped, driven, or

otherwise transported to the laboratory). Shipping containers will be padlocked or sealed for shipment to the laboratory. The method of shipment, courier name(s), and other pertinent information is entered in the "Remarks" section on the custody record.

- (c) Whenever samples are split with a source or government agency, a separate Receipt for Samples form (see Figure 7-3) is prepared for those samples and marked to indicate with whom the samples are being split. The person relinquishing the samples to the facility or agency shall request the signature of a representative of the appropriate party acknowledging receipt of the samples. If a representative is unavailable or refuses to sign, this is noted in the "Received by" space. When appropriate, as in the case where the representative is unavailable, the custody record should contain a statement that the samples were delivered to the designated location at the designated time.
- (d) All shipments will be accompanied by the Chain-of-Custody Record identifying its contents. The original Record will accompany the shipment, and the copy will be retained by the Field Operations Leader.
- (e) If sent by mail, the package will be registered with return receipt requested. If sent by common carrier or air, proper documentation should be maintained.

7.4.3 Receipt for Samples Form

A completed Receipt for Samples form (see Figure 7-3) shall be completed whenever splits are provided. This form must be completed and a copy given to the owner, operator, or agent-in-charge even if the offer for split samples is declined. The original is retained by the laboratory.

7.4.4 Laboratory Custody Procedures

- (a) A designated sample custodian accepts custody of the shipped samples and verifies that the information on the sample labels matches that on the Chain-of-Custody Records. Pertinent information as to shipment, pickup, courier, etc., is entered in the "Remarks" section. The custodian then enters the sample label data into a bound logbook which is arranged by project code and station number.

The laboratory custodian will use the sample label number or assign a unique laboratory number to each sample label and will assure that all samples are transferred to the proper analyst or stored in the appropriate secure area.

- (b) The laboratory custodian distributes samples to the appropriate analysts. Laboratory personnel are responsible for the care and custody of samples from the time they are received until the sample is exhausted or returned to the custodian.

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- (c) When sample analyses and necessary quality assurance checks have been completed in the laboratory, the unused portion of the sample and the sample container must be disposed of properly. All identifying tags, data sheets, and laboratory records shall be retained as part of the permanent documentation. Samples received by the laboratory will be retained until analyses and quality assurance checks are completed.

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FIGURE 7-1

SAMPLE LABEL

EBASCO

PROJECT NAME: FEASIBILITY STUDY - SYNCON RESINS SITE

FIELD SAMPLE #: _____

SAMPLE LOCATION: _____

DATE: _____ TIME _____

TYPE OF ANALYSIS: _____

PRESERVATION NOTES: _____

SAMPLING TECHNICIAN: _____

LAB #: _____

MEDIA: _____ SAMPLE TYPE: _____

REMARKS: _____

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8. CALIBRATION PROCEDURES AND FREQUENCY

The calibration procedures and frequency of calibration for monitoring equipment is included in the Field Sampling Plan section.

9. ANALYTICAL PROCEDURES

9.1 Background

For each measurement parameter, including all pollutant measurement systems, as far as possible Standard Operating Procedures (SOP) approved by EPA will be used when available.

Field samples taken for laboratory analysis will be analyzed by the Analytical Laboratory. The analyses will be performed in conformance with EPA and State of New Jersey requirements. The methods implemented will be approved by NJDEP.

The analytical methods both qualitative and quantitative which are not officially approved by EPA will be reviewed and approved by the responsible Ebasco Task Leader prior to use in project activities and the NJDEP will be notified. The methods will be submitted in a format which will describe in detail the exact procedures and materials required to analyze the samples. The following items will be included in the procedure:

- o Medium of Application (i.e., water, soil, air)
- o Principle of Method
- o Sample size requirements
- o Detection limits
- o Interferences and Corrective Measures
- o Apparatus (including instrumental parameters)
- o Reagents
- o Calibration procedure
- o Sample preparation (i.e., extraction, digestion)

- o Diagrams or tables that describe the method
- o Step-by-step analytical procedure
- o Details of calculation
- o Quality Control requirements (i.e., blanks, spikes, duplicates)
- o Report requirements
- o Reference

Data will be included, if appropriate, to support the limitations and the applicability of the method.

If at any time a change in the documented procedure is required, then the Responsible Ebasco Task Leader will examine and evaluate the significance of the change. If the change/modification is determined to be significant, the Ebasco Quality Assurance Officer will then require additional precision, accuracy and detection limit data either to demonstrate that the previous estimates of the limitations are still valid or to develop the necessary data for accuracy describing the new method. EPA guidelines for acceptance of alternative methods will be followed.

9.2 Specific Analytical Chemical Procedures

The detailed analytical chemical procedures employed are included in the operations procedures manuals of the Analytical Laboratories contracted for the Project. The relevant procedures employed on this project are reproduced in Appendix A.

9.3 Test Methods

9.3.1 Priority Pollutants

The Analytical Laboratory procedures include EPA test methods for the National Pollutant Discharge Elimination System priority pollutants, or appropriate state-of-the-art modifications of these tests. The firm's library search program provides identification and analysis of additional compounds according to specific chemical testing needs.

9.3.2 Organic Compounds

The library search package at the Analytical Laboratory analysis of organic compounds using gas chromatograph/mass spectrometer (GC/MS) techniques and follows EPA's advanced method 624, in which purged and trapped volatile compounds are verified using stable isotope-labeled internal standards. For analysis of compounds extractable in basic or acidic media, EPA's Method 625 is used with each analysis performed on fused silica capillary columns.

9.3.3 Trace Metals

Atomic absorption (AA) or inductively coupled plasma (ICP) emission techniques are used at the Analytical Laboratory to analyze trace metals. The EPA protocol for AA, graphite furnace or flame techniques are employed, as appropriate. Cold vapor AA procedures are used to analyze mercury. Phenols and cyanide levels are determined through EPA distillation and colorimetric procedures.

9.3.4 Use of Methylene Chloride

The use of methylene chloride is prohibited for cleaning of any collection or storage container or device.

9.4 Control of Testing

Quality Assurance at the laboratory is reinforced by a special quality control staff. Data validation is ensured through a series of detailed steps. Each test procedure is fully examined and documented in trial runs. Each procedure is also routinely verified through use of replicate analysis and internal and external reference standards. Through a variety of "blank" runs, checks are made on all reagents, glassware, and other supplies that may artificially contaminate a sample. Recoveries are monitored through use of surrogates or compounds chemically equivalent to the compounds to be extracted from the sample.

9.5 EXTENT OF TESTING PROGRAM

The Analytical Laboratory's testing programs is designed to meet the requirements of Federal and State environmental permits and regulations. The analyses can determine the chemical content of groundwater and surface water, sediment, air solid and liquid wastes, effluent discharges, drinking water, and soils samples. The specific testing programs include the following:

- o National Pollutant Discharge Elimination System Tests
 - Priority Pollutants
 - Permit Pollutants
- o Hazardous Wastes Tests
 - EP Toxicity Tests
- o Groundwater Tests
 - Interim primary drinking water standards
 - Trihalomethane compounds

- Secondary drinking water standards
 - Suggested no adverse response level contaminants
- o Polychlorinated Biphenyls Tests
 - o Superfund Site Tests

In addition, the Analytical Laboratory is capable of analyzing for polychlorinated dibenzodioxins and other highly hazardous compounds.

9.6 Limit of Detection

Limits of detection for the Analytical Laboratory laboratories are described in the Standard Operating Procedures for the Analytical Laboratory.

9.7 Instrument Condition

Calibration standards will be used by the Analytical Laboratory to demonstrate that the performance of the instrument does not cause unnecessary error in the analyses. This calibration will indicate instrument stability and sensitivity. Depending upon the type of instrumental technique being used, it may be necessary to check instrument stability or sensitivity at certain intervals during the analysis.

10. DATA REDUCTION, VALIDATION AND REPORTING

10.1 Data Reduction

10.1.1 Definition

Data reduction frequently includes computation of summary statistics and their standard errors, confidence intervals, test of hypothesis relative to the parameters, and model validation. Procedures address the reliability of computations and the overall correctiveness of the data reduction.

10.1.2 References

The following references will be used for analytical and data reduction procedures;

- o EPA-600/4-79-020, "Methods for Chemical Analysis of Water and Wastes, Section 200; Metals."
- o 44 FR 69559 Appendix IV, Optical Emission Spectrometric Method for Trace Element Analysis of Water and Wastes.
- o EPA-600/4-79-019, March 1979, "Handbook for Analytical Quality Control in Water and Wastewater Laboratories," Chapter 7, Data Handling and Reporting."

10.1.3 Data Collection

The data are collected at the site or in the laboratory. The samples collected at the site are analyzed in the laboratory following standard procedures. The data collected at the site may include pH, temperature,

conductivity, dissolved oxygen, etc. and are recorded in log books. In addition, water, soil, air, and solid waste samples are collected and transported to the Analytical Laboratory for analyses of the contaminants of interest.

10.1.4 Data Usage

The data generated at site and/or in the laboratory will be used to satisfy the individual task requirements. The equations and the typical calculation sequence which should be followed to reduce the data in the acceptable format will be described in the task reports.

10.1.5 Auxiliary Data

Auxiliary data produced for internal records and not reported to clients as part of the analytical data includes the following: laboratory worksheets, laboratory notebooks, sample tracking system forms, instrument logs, standards records, maintenance records, calibration records, and associated quality control. These sources are available for inspection during audits, and to determine the validity of data.

10.2 DATA VALIDATION

(a) Validation will be performed when reviewing the data to assure the following:

- o Sampling procedures are employed at site as indicated in Section 6.
- o The analytical procedures are followed in the laboratory as indicated in Section 9.

- o The test results are within the control limits.
 - o The data and calculations are checked by the supervisor who was not involved in the performance of sampling, analysis or data reduction.
 - o A final review of the data by the laboratory manager for correctness and validity of the data.
 - o The degree of agreement between samples and duplicates will be one of the most significant indicators of data validity.
- (b) Documentation used to validate precision and accuracy of the measured parameters and to support the representativeness, comparability, and completeness of the work include:
- o Description of the calibration of methods and instruments.
 - o Description of routine instrument checks (noise levels, drift, linearity, etc.).
 - o Documentation on traceability of instrument standards, samples, and data.
 - o Documentation on analytical methodology and QC methodology.
 - o Description of applicable performance audits with appropriate audit materials.
 - o Description of the control for interference contaminants in analytical methods (use of reference blanks and check standards for method accuracy and precision).

- o Description of levels of routine maintenance to ensure analytical reliability.
 - o Documentation on sample preservation and transport.
- (c) The Ebasco Quality Assurance Officer is responsible for final data validation, which may require review of the documentation listed in (b) above.

10.3 Reporting

10.3.1 Contents of Report

- (a) The results of laboratory analysis will be reported to the Project Manager within 30 calendar days from the arrival of the samples at the laboratory. The laboratory report will conform to the reporting format identified in the RFP. Data reporting forms have been included in this QAPMP section. As a minimum, the laboratory report will contain the following information for each environmental and waste sample submitted:

- o Laboratory review page, which includes the lab name, certification number, lab supervisor name and signature, and certifying statement.

- o Table of contents listing all major sections of deliverable documents.
- o Laboratory chronicle presenting the dated sequence of sample movement through the laboratory for analysis (and reanalysis, if required). This chronicle will address receipt/refrigeration of the sample, preparation by fraction and re-extraction (if necessary), analysis, Section Supervisor review and approval with signature, and QA/QC Officer review and approval with signature.
- o Methodology review citing the approved SOPs and including a brief narrative outlining the essential points of each methodology used.

In addition, the following forms will be included in the report package:

- o GC/MS/DS Tune Summary Forms
- o Targetted Analyte Calibration Forms
- o Analyte Summary QC/Surrogate Summary Forms
- o Non-Targetted Summary Forms

Surrogate recovery achievement will conform to either current IFB (WA83-A198) forms with water/soil matrices or in-house determined R₊₃s as defined by EPA-600/4-82-057, July 1982 protocols, wherein a minimum of two surrogates per fraction will be used (1 surrogate for pesticides/PCBs and 2,3,7,8 - TCDD analysis). In either case, criteria of achievement for surrogates will be

rigorously followed and will be directly related to an IFB preassessment (screen) of the samples.

Supportive raw data will also be included with the reporting package (i.e., chromatograms for tune compounds, raw data for method blanks, field blanks, matrix spikes, and matrix duplicates). SOP verification of calibration curves will be followed, and the results of the calibration check will be incorporated into the reporting package.

In the area of data system outputs, all actual data output, along with handwritten decision-making by the mass spectroscopist on the data output sheet, will be submitted with the reporting package. Software used will be defined as to type and source, and the method of quantification will be shown.

Verification of qualitative identifications of positive targetted analyses will be performed by one of the following methods:

- o Standard spectrum versus sample spectrum, or
- o Mass chromatographs for characteristic and secondary ions versus RIC showing maxima +1 scan (presentation must be within a limited window near the expected RT of the analytes).

In addition, an NBS search of non-targetted compound spectra will be performed and presented with the three best matches.

A separate Non-Conformance Summary Report will accompany the reporting package. This report, in narrative and tabular form, will present all data falling outside of the QA criteria specified and approved in the QA plan. For surrogates outside of control limits, directions presented in IFB (WA83-A198) Exhibit E, p. 8 of 63, will be followed.

For specific analyses, the following additional reporting requirements apply as follows:

Gas Chromatography or High Performance Liquid Chromatography (HPLC)

1. Chromatograms, appropriately labelled with time, run, analyst, instrument, temperature, column and detector. Also internal standards, surrogates and identifiable peaks should be labelled. Chromatograms are required for standards and test runs.
2. For Qualitative Verifications - calculations of Relative Retention Times.
3. Copies of calibration curves used to establish linear detector response to the compound of interest.
4. Copy of one instrument/reagent blank per eight hour shift.

Gas Chromatography/Mass Spectrometry (GC/MS)

1. One copy of DFTPP and BFB mass spectra for each 8 hour shift to demonstrate tuning.

2. Copies of standards mass spectra.
3. Copies of sample mass spectra.
4. One copy of replicate and matrix spike spectra per twenty (20) samples.
5. One copy of blank spectra per eight (8) hour shift.

Atomic Absorption Spectrophotometry

1. Duplicate results (one per 10 analyses).
2. Spiked sample results (one per 20 analyses, and at least one per matrix).
3. Calibration curve results including blank (one per hour of continuous sample analysis).

Spectrophotometer/Colorimeter Methods

1. Calibration curve results, including blank (one per day of analysis).
2. Duplicate results (one per 10 analyses).

3. Spike results (one per 20 samples).

- (b) Each parameter tested will include: name of parameter, USEPA storet number, if there is any, USEPA or other approved testing procedure references, results of analysis, and the units of the reported results.

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11. INTERNAL QUALITY CONTROL CHECKS

11.1 Quality Control Checks

Quality assurance auditors will be responsible for ensuring that regular internal quality control checks are conducted on the generation of environmental monitoring data. These checks will include usage of the following:

- o Possession and use of the latest approved procedure(s), standards and/or Project Specific Instruction(s);
- o Conformance with appropriate procedures, standards and instructions;
- o Thoroughness of the performance;
- o Identification and completeness of paperwork generated during performance and should include;
 - Project number and/or name
 - Task description
 - Name of performer
 - Date(s) of performance
 - Page number and total number of pages, if more than one sheet;
 - All blank titled spaces of forms have been considered, and
 - Total presentation is legible and reproducible.
 - Data entries, calculations and results are within scope of reason
 - Plots, charts, data summaries, graphs, etc., are precise, and parameters are clearly defined
 - Input data has been approved for use by original department, has been accurately transcribed, and is properly referenced.

11.2 Acceptance Criteria

The following acceptance criteria shall be considered if pertinent to the specific activity (task):

- o Appropriate forms, logs, or formats have been utilized
- o Equipment utilized has been referenced
- o Equipment utilized meets specifications

Other acceptance criteria are incorporated into the technical procedures which describe the performance and documentation of a specific activity (task).

11.3 Acceptance Documentation

The checker shall indicate his acceptance of the task performance and resultant paperwork by signing (or initialing) and dating the appropriate form or space provided.

Differences between the checker(s) and task performer(s) shall be discussed and resolved. If agreement cannot be reached, the differences shall be brought to the attention of succeeding higher levels of management until resolution is achieved.

11.4 Check Frequency

Undocumented checks (surveillance) may be performed, as assigned, during the activity.

A check of documentation shall be performed at the completion of the task.

11.5 Documentation

There are no documents generated in compliance with this procedure; the checking function is documented on material generated in compliance with the applicable procedures for the specific task.

11.6 Analytical Laboratory

The internal quality control procedures are described in Appendix A.

11.7 Field Sampling

11.7.1 Procedure

The procedure for the use of blanks, duplicate samples, spiked samples, and surrogate recoveries has been established for assessing data precision and accuracy and is included in Section 14.0.

11.7.2 Corrective Action

If field procedures are inadequate, the immediate corrective action will be to insure proper, approved procedures are implemented. If samples have been collected, these samples may be discarded and new samples taken. If samples have been sent for analysis, the laboratory will be contacted to terminate analysis.

11.7.3 Contamination

If sample results indicate unacceptable contamination of field or trip blanks, sampling and analysis may be reinitiated. This decision will be made by the Project Manager after consultation with the NJDEP Project Officer.

12. PERFORMANCE AND SYSTEMS AUDITS

12.1 Purpose

The purpose of this procedure is to assure that the surveillance of procured services activities is performed to the extent necessary to verify compliance with the requirements stated in the procurement documents.

12.2 System Audits

The performance of the QA system will be audited as applicable to each field and laboratory procedure. (Follow-up audits will be conducted as needed to confirm corrective actions required.) All system audit reports will be submitted to NJDEP for review.

12.2.1 Analytical Laboratories

The Ebasco QA Officer will audit the Analytical Laboratories.

The system audits performed at the Analytical Laboratories are described in Appendix A.

12.2.2 Field Sampling

Prior to the start of project activities, or shortly after systems are operational, the QA Officer will conduct a system audit covering, as a minimum, the following onsite activities.

- o Organization and Responsibility in order to determine whether the quality assurance organization is operational.
- o The collection of samples to assure that written procedures are available and are being followed.

- o Chain-of-Custody procedures to assure that the appropriate steps have been followed in the traceability of sample origin in order to maintain the integrity of samples and credibility of results obtained therefrom.
- o Operational procedures to assure that the appropriate QC checks are being made in the field and records are maintained of these checks.
- o Equipment to determine whatever the specified equipment is available and in working order.
- o Training to assure that sampling crews are adequately trained.
- o Records to assure that recordkeeping procedures are operational.
- o Corrective action to verify that the appropriate chain of command is followed in responding to out-of-control situations and are properly reported.

12.3 Surveillance

12.3.1 Constant Surveillance

Constant surveillance of a contractor during field sampling and testing shall be performed by the Task Leader or other qualified Ebasco supervisor.

12.3.2 Periodic Surveillance

Periodic surveillance shall be performed during all Ebasco project activities by qualified personnel approved by the Project Manager.

Onsite periodic surveillance and acceptance of the services performed shall be documented by signature and date on the documents generated, by

the person assigned by the Project Manager. Such documents include forms, logs, maps, charts, drawings, test results, checklists, computer printouts, test evaluations, or in the case of fabrication, inspection reports, etc.

12.4 Performance Audit

12.4.1 Analytical Laboratories

Performance audits of the Analytical Laboratory will be conducted by the Quality Assurance Officer early in the analytical process to ensure that practices are in conformance with Standard Operating Procedures (SOP's), laboratory QA/QC is fully implemented, and Good Laboratory Practices (GLP) are in effect. Commercial or EMSL, Cincinnati performance evaluation samples will be provided to the laboratory as part of the performance audit. Each of the chemical analytical methods to be used will be assessed by the results of QA check sample analyses following the contract SOP's. Radiation check samples provided by EPA, Las Vegas will be submitted to the laboratory for analysis. A data certification audit will be conducted at the end of analyses prior to final data acceptance by Ebasco. Follow-up audits may be conducted as required to verify the implementation of required corrective actions.

12.4.2 Field Investigation

Performance audits of the field investigation will be conducted by the Quality Assurance officer to ensure compliance with the Quality Assurance Project Management Plan. During these audits the QA officer will review surveillance documentation and other evidence of supervision. Using the Field Sampling Plan as the basis, the QA officer will also confirm performance of equipment maintenance and calibration; compliance with drilling sampling and testing protocols; and sample handling and preservation. The QA officer will be responsible to audit the

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performance of each major class of field activity, e.g., soil augering; water well sampling; drum sampling; air monitoring; split-spoon sampling; surface water testing; etc.

12.5 Resolution of Discrepancies

If there are any discrepancies, deficiencies or indeterminate results, the individual performing the surveillance shall take the necessary action to require appropriate corrective actions to be taken and assure that corrective actions are completed prior to documenting acceptance of the services performed. If resolution cannot be reached, work shall be stopped by the individual performing the surveillance and the problems shall be brought to the attention of the Project Manager or delegated representative, to attain resolution.

The Project Manager or delegated representative, shall evaluate the problems, provide solutions and verify implementation of solutions prior to allowing the activity to resume.

12.6 Nonconformance/Stop-Work

Nonconformance/Stop-Work shall be handled in conformance with Section 15 (Corrective Action).

13. PREVENTIVE MAINTENANCE

13.1 General

Program team members must be able to respond rapidly to a variety of incidents, confidently using both routine as well as incident-specific equipment. Because of the unpredictable nature of these events, personnel must rely on the current state of preparedness of their equipment. To this end, preventive maintenance of equipment is critically important, especially when considering the potential personal hazard associated with their tasks. As part of general good field practices, all program members should be cognizant of preventive maintenance procedures in order to minimize chance of toxic exposure or downtime of field equipment and the wasteful and possibly misleading generation of spurious data caused by the lack of timely preventive maintenance.

13.2 Objective

The objective of the preventive maintenance program for sampling and analytical equipment is to avoid generating spurious environmental measurements that could endanger site personnel, lead to inappropriate remedial responses, or impede an enforcement action.

13.2 Sampling and Analytical Equipment

Depending on the media involved and the intended purpose, a wide variety of equipment is available for sampling and analytical activities. Because of the possible immediate-response nature of the field program, the reliance placed on such equipment to assist in evaluating the appropriate level of protection, and the use of environmental

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measurements to support enforcement cases, all sampling and analytical equipment, whether electronic, mechanical, chemical, or otherwise, shall be maintained at its proper functional status. All sampling and analytical equipment for air, groundwater, surface water, solids, and sediments shall be maintained to manufacturer's specifications and in operational condition. Routine preventive maintenance as well as per use inspections and checkout should be conducted to assure proper operation of the various pieces of equipment.

Preventive maintenance for field equipment and frequencies of maintenance are presented in Section VI of the Field Sampling Plan. Maintenance and frequency of maintenance for laboratory analytical equipment is located in Appendix A, Section II of the QAPMP. The laboratory will rely on redundancy of instrumentation to accommodate equipment breakdowns.

13.3 Support Equipment

Support equipment is defined as all equipment not previously discussed that will at some point be required for completing an environmental monitoring or measurement task. This equipment may include safety devices, storage and transportation containers, wind indicators, cameras, and communications gear. Support equipment for preventive maintenance purposes should be periodically inspected to maintain the performance standards necessary for proper and efficient execution of all tasks and responsibilities. Appropriate and sufficient replacement parts or equipment should be available for all of these categories of equipment so that sampling and monitoring tasks are not substantively impeded or delayed.

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13.4 Laboratory Analytical Equipment

Normal preventive maintenance procedures are employed by the Analytical Laboratory as described in Appendix A. Supplement 1 to Appendix A of the QAPMP summarizes the laboratory instrumentation, along with frequency and type of maintenance performed on each instrument.

13.5 FIELD ANALYTICAL INSTRUMENTS

The field analytical procedures include measurement of pH, temperature, conductivity and organic vapor in air. Preventive maintenance procedures for this equipment are detailed in the FSP.

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14. SPECIFIC ROUTINE PROCEDURES TO ASSESS
DATA PRECISION, ACCURACY AND COMPLETENESS

14.1 Definition of the Terms

14.1.1 Samples

A group of units or portion of material, taken from a larger collection of units or quantity of material, which serves to provide information that can be used as a basis for judging the quality of the larger quantity as a basis for action on the larger quantity.

14.1.2 Data Quality

The totality of features and characteristics of data that bears on its ability to satisfy a given purpose. The characteristics of major importance are accuracy, precision, completeness, representativeness and comparability.

14.1.3 Accuracy

The degree of agreement of a measurement (or an average of measurements of the same thing), X , with an accepted reference or true value, T , is usually expressed as the difference as a percentage of the reference or true value, $100 (X-T)/T$. This measure is also known as the Percent Recovery (PR), or recovery efficiency. Accuracy is a measure of the bias in a system.

14.1.4 Bias

The error in a method which systematically distorts results. The term is used interchangeably with accuracy in that bias is a measure of inaccuracy.

14.1.5 Relative Error

The mean error of a series of test results as percentage of the true result.

14.1.6 Precision

The degree of mutual agreement among individual measurements. Relative to a method of test, precision is the degree of mutual agreement among individual measurements made under prescribed, like conditions.

14.1.7 Precision Data

Measurements which relate to the variation among the test results themselves, i.e., the scatter or dispersion of a series of test results, without assumption of any prior information. The following measures apply:

- (a) Standard Deviation (δ). The square root of the variance:

$$\delta = \sqrt{\frac{\sum_{i=1}^{i=n} (X_i - \mu)^2}{n}}$$

- (b) Unbiased standard deviation, estimate of universe(s):

$$s = \sqrt{\frac{\sum_{i=1}^n (X_i - \bar{X})^2}{n-1}}$$

- (c) Coefficient of variance (V). The ratio of the standard deviation of a set of numbers, n, to their average, X, expressed as a percentage:

$$V = \frac{s}{\bar{X}}$$

- (d) Relative Percent Difference (RPD). RPD is an average used to compare duplicate analyses. It may be calculated as:

$$RPD = 2 \frac{(C_1 - C_2)}{(C_1 + C_2)} \times 100\%$$

where C_1 and C_2 are the concentrations found in two separate aliquotes of the same sample.

14.1.8 Completeness

A measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct normal conditions.

At the end of a project or specified time period, completeness may be calculated as:

$$\text{Completeness \%} = \frac{\text{Number of Valid Data Acquired}}{\text{Total Number of Values Planned}} \times 100$$

14.1.9 Representativeness

The degree to which data accurately and precisely represent a

characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition.

14.1.10 Comparability

The confidence with which one data set can be compared to another.

14.2 Fieldwork

14.2.1 Sampling

The sampling consists of a single collection cycle in the field for subsequent chemical analysis in an analytical laboratory. There is no opportunity to make routine assessments of accuracy, precision, or completeness in the course of the field sampling.

14.2.2 Blank Samples

The assessment of field sampling for inadvertent contamination will consist of the inclusion of a 1 trip blank per shipment and 1 field blank per sample media per day of sampling. The blanks will contribute to accuracy of identification by checking for compounds inadvertently introduced into the samples during shipment or from contaminated sampling equipment.

14.2.3 Duplicate Samples

At least one duplicate field sample will be taken for each media sampled (air, water, soil) for each sampling program, where soil or water sample numbers exceed 20, duplicates will be at the approximate rate of 5%. For air samples the duplication rate will be approximately 10%. Duplicate samples are used to estimate the precision of results reported by the

laboratory. The Relative Percent Difference (RPD) for duplicates will be compared with the laboratory's RPD control limits for a specific analyte as a data acceptance test.

14.2.4 Spiked Samples

Environmental samples which will be specified as "to be spiked" by the laboratory will be taken for each media (air, water, soil) at the rate of at least one per twenty samples. Spikes will consist of dosing a known quantity of analyte (s) into an environmental sample whose native or background analyte concentrations must also be determined. The results of such spiking may be used to estimate the accuracy of data reported by the laboratory. The Percent Recovery (PR) for spiked samples will be compared with the laboratory's PR control charts for specific analytes as a data acceptance test.

14.3 Laboratory Analysis

Assessments of the precision, accuracy, and completeness of data are made by the Analytical Laboratory. The methods for making these assessments are prescribed in the Standard Operating Procedures of the Analytical Laboratory described in Appendix A. These procedures specify the processing of blanks, duplicates, and spikes at the approximate rate of 1 in every 10 samples. Surrogate standards are used with each sample processed by gas chromatograph/mass spectrograph. Additionally, the Analytical Laboratory monitors their quality control data to ensure that it is within the established control limits for the methods.

14.4 Procedure Validation

When analytical methods are developed or a laboratory is to be certified to perform a method, the data necessary to characterize the method or to

demonstrate a laboratory's ability to perform the method must be submitted to the responsible Ebasco Task Leader and the Ebasco Quality Assurance Officer prior to implementation.

These data will include estimates of the standard deviation, percent recovery, and coefficient of variation at target concentrations established by the Quality Assurance Officer.

14.5 Review of Data

When sample analysis data are received from the Analytical Laboratory it will be reviewed by the Quality Assurance Officer, and the accuracy and precision achieved will be compared to established control limits.

15. CORRECTIVE ACTION

15.1 Nonconformance Report

A Nonconformance Report (NCR) will be issued for each nonconforming condition identified i.e., when objectives for precision, accuracy, completeness representatives or comparability are not satisfied, or when unacceptable procedural practices or conditions are identified.

The Nonconformance Report will fully describe the conditions requiring corrective action, will indicate the nature of the corrections required, and will specify a schedule for compliance. The final authority for issuance of an NCR rests with the Quality Assurance Officer through the Project Manager.

15.2 Corrective Action

Upon the issuance of an NCR, it will be delivered to a responsible officer of the laboratory or organization involved. The NCR will provide space for the responsible individual to indicate the nature of the corrective action taken, and will require appropriate documentation of such action. After the NCR has been reviewed and the corrective action is acceptable, the Quality Assurance Officer will sign the NCR to this effect and inform the involved parties that the NCR has been satisfactorily resolved.

15.3 Stop-Work Order

If corrective actions are insufficient, or resolution cannot be reached, or results of prior work are indeterminate, work may be stopped by a Stop-Work Order. The Stop-Work Order can only be authorized by the Project Manager, or Ebasco Quality Assurance Officer in writing. The NJDEP shall be alerted that a Stop-Work Order has been issued.

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15.4 Stop-Work Corrective Action

The conditions for which the Stop-Work Order was issued will be described in sufficient detail to allow proper evaluation of the problems and to effect proper corrective action. Documentation of discussions, telecoms, or correspondence which describe the actions taken to evaluate the problems, provide solutions, and verify implementation of solutions shall be attached to the Stop-Work Order and fully referenced in the appropriate spaces. Work shall not continue until the Stop-Work Order has been rescinded by the individual that authorized the stop work.

15.5 Cause and Action to Prevent Recurrence

The Ebasco Quality Assurance Officer, shall track the NCRs, analyze the corrective actions required, and take the necessary steps to resolve the causes of the nonconforming conditions in order to prevent recurrence.

16. QUALITY ASSURANCE REPORTS TO MANAGEMENT

16.1 Frequency

At the conclusion of each laboratory certification, field or laboratory audit or data validation, the Quality Assurance Officer will prepare and provide a comprehensive quality assurance report to the Project Manager, the NJDEP, the procured service organization and other appropriate project personnel on the performance of the Quality Assurance Program.

16.2 Contents

The reports to management will contain:

- o Results of system and performance audits conducted,
- o An assessment of the accuracy of measurement data, including its precision, completeness, representativeness, and comparability,
- o A listing of the Nonconformances issued during the period, related corrective actions undertaken, and an assessment of the results of these actions, and
- o Identification of significant Quality Assurance problems and recommended solutions.

Appendix A
Review No. 0
Date Dec. 7, 1984
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APPENDIX A
ANALYTICAL LABORATORY PROCEDURES*



ANALYTICAL INC.

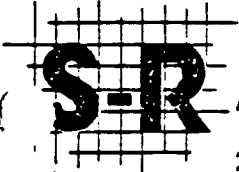
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S-R ANALYTICAL, INC.

CHERRY HILL, NEW JERSEY

STANDARD OPERATING PROCEDURES MANUAL

SYN 001 1836



S-R ANALYTICAL INC.

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April 22, 1985

DISCLAIMER

S-R Analytical, Inc. (S-R) has endeavored to incorporate the WA83A198 modifications into S-R's Standard Operating Procedures (SOP) Manual where applicable. An overview of the technical changes incorporated by this "New Format" methodology is also delineated on pages 35 through 41 section B of the SOP Manual.

It should be noted however that S-R's Quality Assurance Manual contained in Section A is designed to provide a general approach toward laboratory quality control and quality assurance, in the absence of contract-required or client-specific guidelines. There may be minor conflicts therefore between recommendations made in the Quality Assurance Manual and requirements of the WA83A198 format. Where these occur, the WA83A198 format will control for the analysis of all samples submitted under the Syncon Resins project with the prime contractor, Ehasco/Envirosphere.

Catherine M. Ward

Catherine M. Ward
Project Manager

CMW/mb

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STANDARD OPERATING PROCEDURES MANUAL

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. ANALYTICAL METHODS	B
. EXAMPLE DATA REPORT	C

FOREWARD

This document was prepared by S-R Analytical, Inc. (S-R) and contains information pertaining to all levels of the current Quality Assurance Program (QAP). This manual is periodically updated, as required, by S-R's Management. Any questions concerning this information should be directed to any of the people listed below:

Prepared December, 1980 William J. Ziegler, Laboratory Manager
Revised September, 1981 William J. Ziegler, Laboratory Manager
Revised February, 1982 William M. Hartman, Quality Assurance Manager
Revised June, 1982, William M. Hartman, Quality Assurance Manager
Revised November, 1984, Catherine M. Ward, Project Manager

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SECTION A

THE LABORATORY QUALITY ASSURANCE PROGRAM

S-R ANALYTICAL, INC.

Quality Assurance Program

QUALITY ASSURANCE PROGRAM

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I. QUALITY ASSURANCE PROGRAM, OVERVIEW

1. MANAGEMENT STRUCTURE

It is the philosophy of the management of S-R Analytical (S-R) that there should be no compromises made in the level of effort taken to provide a finished product of the highest caliber. In order to assure the attainment of this goal, the management as well as the quality assurance personnel of S-R have developed a Quality Assurance Program (QAP) which exceeds all of the known regulatory quality assurance requirements for routine sampling and environmental analyses. S-R also follows rigorous data review and evaluation protocols to assure the correctness of the final product. A typical summary of these procedures is as follows:

- . Rigorous sample and standard preparation
- . Calibration and/or standardization
- . Analyses including QA/QC samples
- . Analysis calculations
- . QA/QC checks - includes internal, external, and standard reference material (SRM) spiking procedures
- . Calculation and notebook review by Supervisor and/or Laboratory Manager
- . Independent data audit
- . Report preparation - author
- . Report review - Laboratory Manager and/or Quality Assurance Manager
- . Typing - typist proofreading
- . Office Manager Review - editorial accuracy
- . Author Review - technical accuracy
- . Laboratory Manager review
- . Corrections made
- . Management Review - verify internal consistency, and all interpretations and/or conclusions for correctness
- . Reproduction and Issuance of final report

The implementation of the above procedures both minimizes the occurrence of inaccuracies as well as provides a means for identifying and correcting problems as they occur. This decision making process is also delineated in a flow chart in Appendix F.

The structure of the administrative group of S-R is displayed in Figure 1. Figure 2 shows the organization of the Laboratory Services Department. The Quality Assurance Manager reports directly to the General Manager. This organization allows the QA Manager to function independently of the workload.

The Quality Assurance Manager, the Laboratory Manager, and the Section Supervisor function as a team when necessary to correct any problems causing, or which may lead to, a reduction in the high level of quality required by the management of S-R Analytical, Inc. The resumes of current personnel may be found in Appendix G.

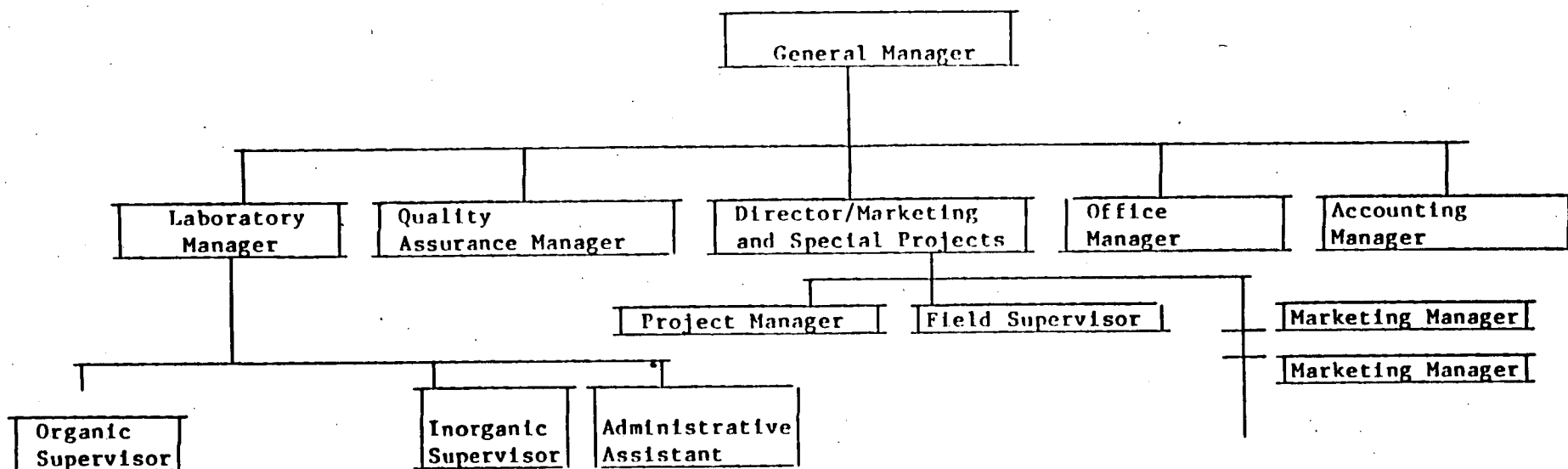
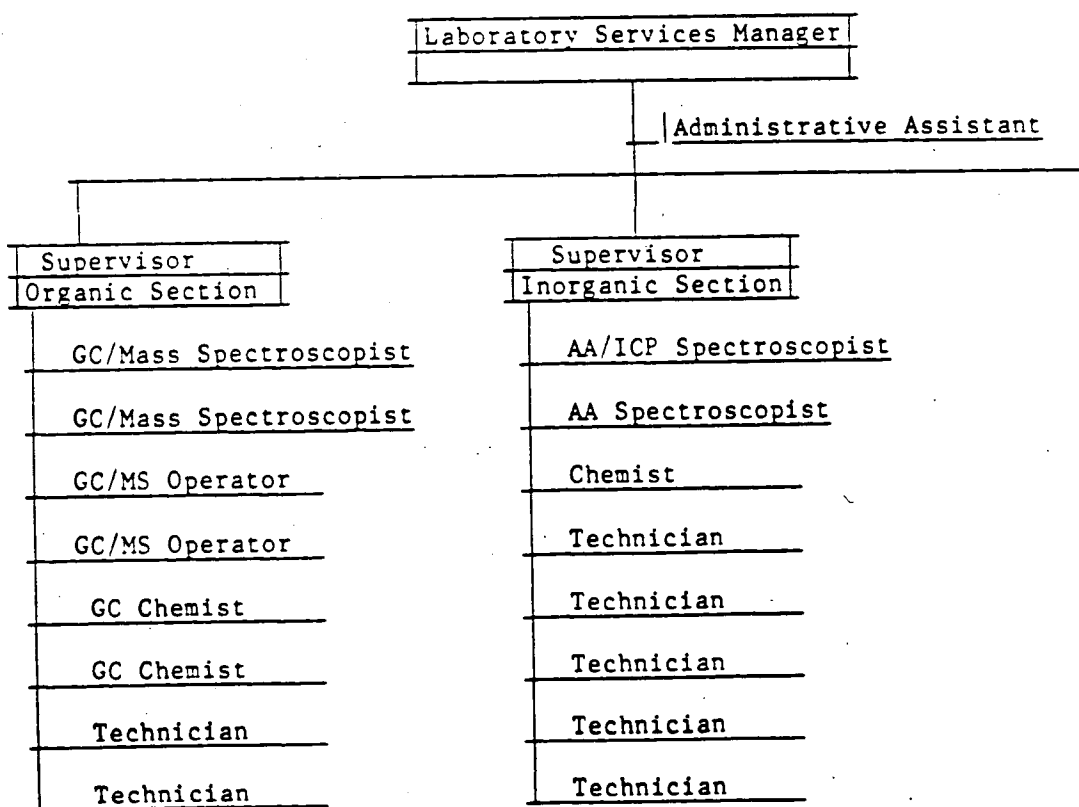


FIGURE 1

FIGURE 2

S-R LABORATORY SERVICES DEPARTMENT

2. FUNCTION OF QUALITY ASSURANCE MANAGER

The primary function of the Quality Assurance Manager is to identify conditions which may lead to a reduction in analytical quality.

The Quality Assurance Manager reports directly to the General Manager on all aspects of analytical quality. Together with the General Manager, the Quality Assurance Manager acts upon problems which may lead to a quality reduction.

The Quality Assurance Manager is also responsible for monitoring the correctness of all routine internal quality control measurements (reagent blank, duplicates, and spiked sample analysis) with independently prepared, externally (blind) spiked samples, synthetically prepared samples, and Standard Reference Materials (SRMs). The Quality Assurance Manager is directly responsible for overseeing the validation of all new methods for S-R. The Quality Assurance Manager also acts as an independent audit of the raw analytical data being generated throughout the laboratory.

3. SAMPLING AND SAMPLE INTEGRITY

(A) General

There are numerous sources of variability associated with any analytical technique, but before a sample even arrives at the laboratory there are sources of error associated with sample collection and preservation techniques. The main goal of sample collection is to obtain a sample that is truly representative of the system under study. There are many factors that affect the sample collection of representative samples some of which are:

- . Sampling source
- . Sampling technique
- . Physical state of the system at the time of sampling (static vs. dynamic)

If the sample is not truly representative of the system under study, then even the most rigorous analytical methods or the most sophisticated instrumentation will fail to yield meaningful results that characterize the system.

Quality Assurance Program

Often the physical and chemical properties of the specific parameters to be determined will affect the results obtained on a given sample. These factors can cause results to vary from what is expected at the time of sampling. In addition, they can vary greatly within a given family of pollutants; an example of this would be the base-neutral extractable organic compounds. The Laboratory Services Department will provide information when possible concerning the properties of any compounds that are being considered. Some of the properties which may adversely affect sample integrity are listed below:

- | | |
|-------------------------|---------------------|
| . Solubility properties | . Phase separation |
| . Cross reaction | . Decomposition |
| . Degradations | . Transition points |
| . Polymerization | . Density |

If the system to be sampled is not uniform in nature, as in some lakes or lagoons with stratified layers, or chemical storage tanks, then multiple samples should be taken. When possible, the number of samples which are taken should be derived from a statistical evaluation of the system under study.

(B) Preservation and Sample Integrity

Prior to the initiation of analysis, temperature, chemicals, and time are used to maintain samples as closely as possible to the physical and chemical state they existed in the sample sources.

Preservation of samples is not always possible. This is evident in the case of samples containing a hydrocarbon matrix that is not miscible with the inorganic preservatives that are used. If possible, all samples should be reduced to a temperature of approximately 4°C until delivery to the lab. Preservatives for specific priority pollutants analysis are outlined in Table 1. A more comprehensive table of preservations can be found in Appendix A of the Stablax-Reutter Quality Assurance Manual. All of the containers and preservatives listed below are available from the Laboratory Service Department. Keep in mind that there are special cleaning precautions that must be followed before a sample container can be used.

Aqueous samples are collected in accordance with the following publication:

- . Field Procedures Manual for Water
Data Acquisition, Office of Quality Assurance,
Division of water Resources, NJDEP, 1983 edition.

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Table I

Quantity of Sample and Preservatives for Priority PollutantParameters in Water, Wastewater and Other Aqueous Based Matrices

<u>Priority Pollutants Class*</u>	<u>Size of Sample</u>	<u>Sample Container</u>	<u>Preservations</u>
Acid Extractable	1 liter	glass*	Maintain at Approx. 4°C
Base Neutrals	2 liter	glass*	Maintain at Approx. 4°C
Pesticides	1 liter	glass	Maintain at Approx. 4°C
Polychlorinated Biphenyls	1 liter	glass	Maintain at Approx. 4°C
Purgeable Organics	80 ml	glass**	Maintain at Approx. 4°C
Metals	500 ml	nalgene	HNO ₃ pH <2
Cyanides	250 ml	nalgene	NaOH pH <12

* Amber glass is recommended when the containers will be exposed to light for a prolonged period of time.

** At least two (2) glass vials (40 mls) with Teflon lined screw caps.

Samples containing organic hazardous wastes, soil, sludge, oil or any heavy hydrocarbon matrix cannot be chemically preserved with procedures designed for aqueous systems.

In samples containing a heavy hydrocarbon matrix, the amount of sample that can be used in the analysis is greatly reduced, due to interferences associated with the matrix. Therefore, only 100 grams of sample are routinely needed for the conduct of a complete priority pollutant analysis.

The Laboratory Services Department strongly recommends that the following preservation steps be taken for non-aqueous of samples.

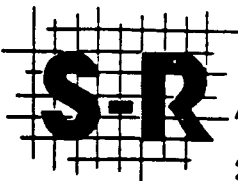
- The sample should be taken in amber glass since many priority pollutants are photosensitive.
- Maintain samples at a temperature less than 4°C.
- Deliver the samples to the laboratory within 24 hours of sampling.
- Keep the air space in the sample container at a minimum.

Quality Assurance Program

(C) Sample Collection and Documentation

The main goal of sample collection is to obtain a sample which is truly representative of the system under study. When a statistical evaluation of the system cannot be made, the Field Sampling Supervisor must intuitively determine where and how to best represent the system. However, in order to substantiate the decision of how, when, where, and why he chose to take the particular sample(s), the Field Sampling Supervisor must clearly document all information pertinent to this decision such that it can be reviewed and evaluated by S-R management. This information is recorded on an S-R Field Data Sheet (Figure 3), as well as in a Field Notebook.

Samples requiring custody control at all times are subjected to detailed chain of custody procedures. These procedures include the documentation of anyone who has custody or control of the sample by requiring a signature and reason for control before the release of any portion of the sample (See Figure 4). Sample container preparation is recorded in a laboratory notebook. The sample containers are prepared, closed and permanently sealed with unique, serialized S-R Chain of Custody Label (Coded A) made from UL listed, tamperproof paper. A corresponding, unique label is provided (Coded B) with every bottle and such that the sampler may reseal the bottle after collection, thereby preventing anyone from opening the container prior to its receipt by the Laboratory Services Department. The detailed procedures for chain of custody and bottle cleaning are located in Appendices B and D.



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CHAIN OF CUSTODY RECORD

Name of Unit and Address						
Number	Item	Description of Samples				
		Remarks:				
Person Assuming Responsibility for Samples:					Time	Date
Number	Relinquished by:	Received by:	Time	Date	Reason for Change of Custody	
Number	Relinquished by:	Received by:	Time	Date	Reason for Change of Custody	
Number	Relinquished by:	Received by:	Time	Date	Reason of Change of Custody	
Number	Relinquished by:	Received by:	Time	Date	Reason of Change of Custody	

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Quality Assurance Program

CHAIN OF CUSTODY				
Sample No.	Date	Relinquished by	Received by	Purpose of Change of Custody
		Name, Position	Name, Position	
		Signature	Signature	
		Name, Position	Name, Position	
		Signature	Signature	
		Name, Position	Name, Position	
		Signature	Signature	
		Name, Position	Name, Position	
		Signature	Signature	
		Name, Position	Name, Position	
		Signature	Signature	
		Name, Position	Name, Position	
		Signature	Signature	
		Name, Position	Name, Position	
		Signature	Signature	
		Name, Position	Name, Position	
		Signature	Signature	
		Name, Position	Name, Position	
		Signature	Signature	

Figure 4

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Quality Assurance Program

3. ANALYTICAL TECHNIQUES FOR ORGANIC PRIORITY POLLUTANTS

The following outline summarizes the various organic sample preparation techniques and instrumental methods used by the Laboratory Services Department in Organic Priority Pollutant analysis. Most of these methods are based on the 600 Series Methods published in the Federal Register, Vol. 44, No. 233, December 3, 1979, 1982 Revision. In case of solid samples or samples with a hydrocarbon matrix, modifications to the 600 Series methods must be made. The modifications used by Stablax-Reutter were developed based on our own research, as well as correspondence with various experts in the field from EPA, in universities, and from various articles published in technical journals. Specific references and details on any of the techniques discussed can be provided upon request. Several sample preparation procedures used by Stablax-Reutter for solid samples, or liquid hydrocarbon matrices can be found in the following publication:

- . EPA - Test Methods for Evaluating Solid Wastes - Physical/Chemical Methods-SWA846-Revision, July 1982.

Additional details on these procedures are contained in the GC Section Operations Manual, and can be furnished upon request.

Items discussed in this section are as follows:

- . Purgeable halogenated and aromatic organic compounds
- . Pesticides and PCBs
- . Acid extractables and base/neutral extractables
- . Confirmation of results
- . Listing of instrumentation

(A) Purgeable Halogenated and Aromatic Organic Compounds

- . Isolation Technique - static headspace, purge and trap with CDS instrument or Tekmar LSC-1, thermal desorption purge and trap
- . GC Columns primary - 1% SP-1000 on 60/80 mesh Carbopack B with 5 cm pre-column 5% SP-1000 on 80/100 mesh Supelcoport.
confirmatory - n-octane on 100/120 mesh Poracil C
- . GC Detectors - Hall, ECD, PID, FID, GC/MS.
Note: with capability for Hall /PID split and FID/ECD split.

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Quality Assurance Program

Water and wastewater samples can usually be analyzed by purge and trap following EPA Methods 601, 602, and 603. The Laboratory Services Department uses the Tekmar LSC-1 or the CDS for this type of analysis. Soils, sludges, oils, and chemical wastes cannot be handled in this manner. For these types of samples either static headspace analysis, or thermal desorption purge and trap is used.

In static headspace analysis 0.25 to 1.00 gram of sample is placed in a 60 ml vial, and the vial is sealed with a teflon lined cap. The vial is then heated to 60°C for 30 minutes, and a sample of the headspace above the sample is removed and injected into the GC. However, a serious problem with static headspace analysis arises when the headspace becomes saturated with a given analyte. When this occurs a "greater than" result must be reported.

In thermal desorption purge and trap, the sample is placed in a sealed vessel and is sparged with helium gas. The volatile components are collected onto a tenax column which is then heated and desorbed into the GC or GC/MS. The sealed vessel can be heated to any temperature specified; usually 60°C is used. The Laboratory Services Department uses the CDS thermal desorption purge and trap device. The Tekmar device is also available for this type of analysis.

(B) Pesticides and PCBs

- Type of Extraction - Liquid/liquid extraction with solvent, continuous soxhlet extraction with Methylene Chloride sonication with Methylene Chloride
- Clean-up Solvent - 6% diethyl ether in petroleum ether
15% diethyl ether in petroleum ether
- Clean-up Columns - Florisil, 60/100 mesh in 22 x 300 mm glass glass columns with stopcocks, columns charged by weight
- GC Columns
 - primary - 1.5% SP-2250/1.95% SP-2401 on 100/120 mesh Supelcoport
 - confirmatory - 4% SE-30/6% SP-2401 on 100/120 mesh Supelcoport
- GC Detectors - Hall/PID split for PCBs, Hall and ECD for PCBs, ECD for pesticides, GC/MS for confirmatory work.

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Quality Assurance Program

PCBs are difficult analytes to characterize because of the multiplicity of peaks that must be determined. In samples with a hydrocarbon matrix, it is difficult to determine PCBs against other interfering peaks. The following considerations should be kept in mind.

- . Electron capture detection (ECD) is the most sensitive detector for PCBs, but it is not the most selective. For clean water or wastewater samples, in which extremely low detection limits are required, ECD should be used. However, in samples containing an organic matrix, ECD is not recommended. The ECD will respond to any species with a high affinity for electrons. This includes not only halogens, but also oxygenated and sulfur groups commonly found in oil. These groups will give an ECD response, and can either mask PCB patterns, or lead to false positives. Cleanup techniques may be used which minimize positive interferences.
- . The Hall detector is the most selective detector for halogenated organics. Interferences that affect an ECD response usually do not appear using a Hall detector. The Hall detector is actually a high temperature reaction vessel in which halogenated groups are split off of organic molecules and react to form HCl, HBr, HI. This reaction product is then fed to a conductivity detector which responds only to these species. The result is a truly halogen specific detector. PCB patterns are very distinct and well defined using a Hall detector.
- . Gas Chromatography/Mass Spectroscopy (GC/MS) is specific for PCBs as long as the proper target ions are used in the selective ion monitoring program. The problem with GC/MS is that it responds (like an FID) to every hydrocarbon species present. Quantitative work is nearly impossible, but qualitative confirmations are possible. The library search program used must be able to distinguish peaks originating from hydrocarbon constituents. This kind of search is time consuming, but without it a false negative conclusion may be made. The key is to search for mono-, di-, tri-, tetra- and penta- chlorinated biphenyl fragments using the following target ions; 224, 260, 294, 330. Searching for a given Arochlor can lead to a false negative due to the hydrocarbon matrix, which can mask the Arochlor.
- . The photoionization detector (PID) is another type of detector that the Laboratory Services Department can utilize for PCB characterization. The PID is specific for aromatic groups. The PID is used in conjunction with the Hall detector in an effluent split mode. This PID/Hall split further aids in identifying and characterizing PCBs.

Quality Assurance Program

(C) Acid Extractables and Base/Neutral Extractables. Acid Extractables

- Type of Extraction - Sample is acidified and extracted with methylene chloride.
- GC Columns - 1% SP-1240 DA on 100/120 mesh Supelcoport
 - SE54 fused silica wide bore capillary
 - 5% OV-17 on Chromosorb W-AW-DMCS 80/100 mesh
- GC Detectors - FID, PID, GC/MS

. Base/Neutral Extractables

- Type of Extraction - Sample is treated with NaOH and extracted with methylene chloride
- GC Columns - 3% SP-2250 DB on 100/120 mesh Supelcoport
 - 1% SP-2250 on 100/120 mesh Supelcoport
 - SE54 Fused silica wide bore capillary
- GC Detectors - FID, GC/MS

For water and wastewater samples, acid extractables and base/neutrals can be analyzed by S-R as described in the EPA Methods 604, 606, 607, and 609 thru 612 and 625. For solid samples or hydrocarbon matrices one to ten grams of sample is weighed directly into a centrifuge tube. An aliquot of an acid or base solution is added and the sample mixture is agitated. Then an equal volume of methylene chloride is added and the mixture is again agitated. The mixture is separated by centrifugation, and the methylene chloride layer is separated. Two more aliquots of methylene chloride are added and the separation repeated. The combined methylene chloride extracts are then concentrated using Kuderna Danish glassware. These analyses are conducted only by GC/MS.

Quality Assurance Program

(D) Confirmation of Results

Gas chromatography is an extremely useful tool for the analysis of organic compounds. However, GC is not capable of unequivocally identifying a particular analyte. Any species which elutes from the column at the same time as the analyte will cause the calculated concentration to be high. Where significant decisions hinge on the results of a GC analysis, confirmation is advisable. Confirmation can be accomplished in either of two ways:

. GC Confirmation

The presence of the analyte of interest can be confirmed by GC using a second column of significantly different polarity. Use of a second column changes the retention times of the eluting compounds. If interferences were present in the original analysis, a significant change in the analytical result will be observed. If different results are obtained the lower result should be considered most valid since interferences always cause the apparent concentration to increase.

. GC/MS Confirmation

When a Mass Spectrometer/Data System is used as the GC detector (GC/MS/DS) it is possible in nearly all cases to unequivocally confirm the presence or absence of a particular compound. When the GC/MS/DS has been calibrated with the compound(s) of interest (Priority Pollutants) quantitative analysis is possible. Some analytes such as PCBs, are really a mixture of compounds and in general, it is not possible to perform quantitative analyses for these materials on GC/MS. Their presence or absence can, however, be confirmed by GC/MS/DS. If present, concentrations can be determined using GC with the appropriate detector.

(E) List of Major Instrumentation

- . UV/visible spectrophotometer - Perkin Elmer Model 550
- . Visible spectrophotometer - Bausch & Lomb Spectronic 20
- . Atomic Absorption Spectrophotometer - IL Model 257
- . Atomic Absorption Spectrophotometer - IL Model Video 22
- . Inductively Coupled Argon Plasma Emission Spectrometer - ARL Model 35000
- . Technicon Auto Analyzer with manifold for Phenols, Cyanides, Chloride and Fluoride.
- . Dionex Ion Chromatograph

Quality Assurance Program

- . Gas Chromatograph - Tracor Model 560 with Hall detector and PID which can be used in a split mode. Capillary columns can be used.
- . Gas Chromatograph - Tracor Model 560 with Hall detector and PID which can be used in a split mode. The instrument is also equipped with an autosampler.
- . Gas Chromatograph - Tracor Model 560 with Hall detector and linearized electron capture detector. ECD has capillary column capability.
- . Gas Chromatograph - Perkin Elmer Model 3920 with FID, ECD and Flame Photometric Detector
- . Gas Chromatograph - Perkin Elmer Model Sigma II with FID, ECD, and Nitrogen/Phosphorus detector. The FID and ECD can be used in a split mode. Capillary columns can also be used.
- . CDS purge and trap device that can handle both solids and liquids.
- . GC/MS/DS - Finnigan OWA 1020 - equipped with 9 track tape for archival storage as well as a Control Data Nova 4 computer having an NBS library of 35,000 compounds for qualitative work.
- . GC/MS/DS - Finnigan 5100 (2) - equipped with updated 38,700 compound NBS library with Super-Incos software, magnetic tape storage, dual-purpose capillary and packed column injector and split/splitless operation in capillary mode. Quadrupole MS.
- . Bomb Calorimeter - Parr Model 1241
- . Fluorescence Spectrophotometer
- . Hewlett-Packard Model 3353B Data System with 5 channels
- . Tekmar LC 1 Purge and Trap device (2)
- . Total Organic Carbon Analyzer - Oceanographics International Model 524 The instrument has capability to analyze both solids and liquids
- . Wilks Miran 1A Infrared spectrophotometer
- . Perkin Elmer Model 257 Infrared Spectrophotometer

In addition, the Laboratory is equipped with various pH meters, selective ion meters and electrodes, conductivity meters, turbidimeter, and physical testing devices such as flash point tester, viscometers, compressive strength tester, etc.

No analytical methods are used unless:

- . The method has been proven valid through the use of external and SRM QA checks. The validity will be assessed by the QA Manager, Laboratory Manager, and the appropriate supervisor.
- . The analyst is fully familiar with the preparatory techniques and analytical equipment to be used.
- . The method is fully and permanently documented in published source, laboratory notebook and/or in the Quality Assurance file.

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II. WORKING DOCUMENT OF THE QUALITY ASSURANCE PROGRAM

The QA described in this document is in excess of the minimum requirements that must be followed by all laboratory personnel in order to maintain S-R Analytical compliance with EPA and state guidelines.

As an EPA laboratory certified under the Safe Drinking Water Act and Clean Water Act and NIOSH, S-R Analytical, Inc. is committed to maintain a rigid quality control program in accordance with criteria set forth in the following:

- EPA Handbook for Analytical Quality Control in Water and Wastewater, March, 1979
- EPA Manual for the Interim Certification of Laboratories Involved in Analyzing Public Drinking Water Supplies, May, 1978
- Standard Methods for the Examination of Water and Wastewater, 14th edition
- Water Laboratory Certification Program, New Jersey State Department of Health, 1978
- Manual for Analytical Quality Control for Pesticides and related compounds in Human and Environmental media, Jan., 1979.
- Regulations governing laboratory certification and standards of performance, (N.J.A.C. 57:18)

This section is devoted to a discussion of procedures used to maintain the production of reliable and accurate data. The information is categorized as follows:

- . Sample identity and control
- . Notebooks
- . Training and education
- . Modification of procedures
- . Internal quality control
- . External quality control
- . Instrumentation
- . Equipment
- . Reporting of analytical results
- . Microbiology

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1. SAMPLE IDENTITY AND CONTROL

All samples submitted to the laboratory will be properly preserved for whatever analyses required, and must be in the proper type of sample container. A copy of the required preservation techniques from the EPA manual on Methods for the Chemical Analysis of Water and Wastes, March 1979, is included in the Appendix for reference.

All samples will be given an S-R identification number at the time of submittal, and the appropriate log-in forms will be completed. The sample will be clearly labeled with the client name, date of submittal, preservatives added, S-R ID number, and any other pertinent information supplied by the client. All log-in forms must be reviewed with the Laboratory Manager, before the information is entered into the computer. The Field Data Sheet(s) will be filled out in detail in order to substantiate how, what, where, and why the sample(s) was taken. The sample designation which appears on the Data Sheet is to be exactly the same as on the sample container.

When required by a client or regulatory agency, all S-R chain of custody procedures will be followed.

The procedure for and examples of the appropriate forms for logging in samples are presented in Appendix C. Also included are reproductions of container labels used.

Samples are to be submitted for analysis within the recommended holding times. While awaiting analysis and during analysis, samples are to be stored at 4°C in the walk-in refrigeration unit.

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2. Notebooks

All laboratory data, including preparation of solutions and standards, preservation of samples, instrumental analog and/or digitized printouts or recorder tracings, etc., is to be entered into the laboratory data file. The code number on each digital chromatogram printout must be crossed referenced in the notebook or on the analog chromatogram. All sample calculations and quality control/quality assurance data will be clearly documented in the notebook. Each page of the notebook will be dated and signed at the end of each day.

All laboratory notebooks are to be reviewed with the Laboratory Manager and/or the respective Supervisor on a weekly basis.

The Laboratory Manager and/or Supervisors will sign and witness each page of the notebook.

The following is the instruction to all personnel assigned Laboratory Notebooks.

INSTRUCTIONS FOR UTILIZING LABORATORY NOTEBOOKS

In addition to providing a complete record of laboratory work which can be understood and repeated, the notebook has been designed to afford maximum legal value. Several practices must be followed to give the notebook value as a legal document in possible litigation:

- Enter all data directly into this book; it is permanently bound with numbered pages so that pages can not be substituted or deleted. Do not record data elsewhere for transfer into the book. Write in ink. Never make erasures. Thus the integrity of the record will not be in question.
- Record sufficient information. All procedures, reagents, apparatus, sketches, condition, references, etc., should be entered into the book as the work is done. The purpose and significance of the experiments as well as observations, results, and conclusions should be made clear. What may seem trivial at the time may later prove of critical importance. Your entries should be clear and complete enough for someone else who is "skilled in the art" to read and comprehend what has been accomplished.

Avoid sweeping negative statements, e.g.: "This procedure is worthless, " which could later limit the scope of your claims.

- Not only is the conception of a method important, but so is the diligence shown in making a working model or demonstrating that the idea works --"reducing to practice." These two elements of a method, conception and reduction to practice, must be corroborated by a witness. The records of the analyst(s) are not enough. Thus, each page of the notebook should be read, witnessed, and dated (daily, if possible) by someone who is competent to understand it, but who does not

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claim, to be a co-author. Charts, tables, etc., should be complete, and lines should be drawn through any blank spaces prior to witnessing. It may be wise to perform key experiments in front of one or more witnesses. Spectra, charts, gas chromatographs, digitized computer printouts etc., should be signed, dated, witnessed, and if they can not be permanently attached to the notebook, they should be referred to with an entry in the book and kept permanently on file. Access to the sealed folder containing all spectra, charts, gas chromatograms, and digitized computer printouts can be only obtained if specified material is released by signature. Dates and witnesses can establish your priority of method development.

- To delete an entry, draw a line through it so that it is still legible. Corrections should be made adjacent to the deleted entry, and they should be initialed and dated by you and the corroborating witness. Changes made after the page has been witnessed should also be initialed and dated by you and the witness. Always use the current date.
- The notebook and its contents are to be considered confidential and of great value. Exercise every care in preserving them. Report the loss or theft of a notebook to your Group Leader and/or Laboratory Manager immediately.
- Index the contents and return each book as completed (or when not in use) for filing.
- New ideas must be recorded and witnessed as they occur to establish priority of invention. Even ideas which do not pertain to the project at hand should be documented in the book.

Keep your research records as if each project were to be patented or in litigation. Even though the work contained in the book may not result in a litigation or patent application, observation of these practices will provide a clear record for reports, publication, or future reference:

Instructions Read and Understood by _____

Dated _____

3. TRAINING AND EDUCATION

The Laboratory Manager is directly responsible for the training of all personnel in the Laboratory Services Department. No one, with the exception of the Supervisors, is to take training of other personnel upon themselves without first consulting the Laboratory Manager.

Training of personnel is accomplished in the following manner:

- . Direct supervision by Supervisors, Chemists, Laboratory Manager, and/or Quality Assurance Manager.
- . On the job training with intermittent assistance from superiors.
- . Enrollment in seminars or courses such as with the National Bureau of Standards, American Chemical Society.
- . Reading and studying of established methods.

Education is encouraged through a tuition reimbursement program for all personnel. Courses must be related to the type of work required by the Company. In addition, technical personnel are to avail themselves of the technical journals circulated in order to keep current on new sampling and analysis technology.

4. MODIFICATION OF PROCEDURES

All analytical methods currently utilized for routine sample processing in the S-R laboratory department are considered Standard Methods. It is understood that no changes whatsoever may be arbitrarily made in these standard methods. This includes all the preparatory techniques as well as the instrumental conditions of analysis. All these preparatory techniques and instrumental conditions should be fully documented - either in a published method i.e. Standard Methods, 14th; in a bound laboratory notebook; or permanently filed in the Quality Assurance File.

There are several reasons for requiring standardized analytical techniques. The Standard Method acts as a guide such that several different analysts may use the techniques described in the method and arrive at comparable results. When all analytical conditions are held constant, the Standard Method may be subjected to statistical evaluation and meaningful precision and accuracy data generated.

When an analyst wishes to modify an existing technique because of the potential gain in the efficiency, economy, the precision and/or accuracy of a method, the laboratory management must be consulted and a validation technique agreed upon.

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5. INTERNAL QUALITY CONTROL

- . Duplicate Determinations - a duplicate or duplicate spike analysis on one out of every 10 (20 for certain parameters) samples is to be done for every parameter determined. If the quantity of samples is less than 10, one duplicate will be done. The results of the duplicate analyses are to be documented on control charts. Each test is to have a separate document showing the % deviation in the duplicate results. A separate table is to be attached to each chart showing notebook references and dates for each duplicate analysis. Upper and lower control limits may be periodically determined by statistical methods and posted on all control charts.
- . Spiked Sample Determinations - one sample out of every 10 (20 for certain parameters) to be spiked with a specified quantity of each analyte to be determined. The Lab Manager should be consulted as to the quantity of analyte to spike to a given sample. The % recoveries of all spikes are to be documented in the notebooks, and reported on control charts (with certain exceptions). A separate table is to be attached to each control chart for each test showing the notebook references and dates of each spiked analysis. Upper and lower control limits are to be periodically determined by statistical methods and posted on all control.
- . Complete Reagent Blank - A complete reagent blank analysis should be conducted at a frequency equivalent to about 10% of the total sample load.
- . The Department Supervisors will be responsible for control charts.

6. EXTERNAL QUALITY CONTROL

- . S-R Analytical will participate proficiency testing to the extent required by applicable regulatory agencies.

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7. DATA REVIEW

Before any analytical data is transcribed to a test report or verbally transmitted to a client, all information pertinent to the analysis is to be received by the respective Supervisor. Examples of this are:

- . Signed notebook page(s)
- . Standard graphs(s)
- . Recorder plot (for automatic analyzer, ion chromatograph, gas chromatograph, or atomic absorption spectrometer.)
- . Data System, Integrator, ICP, or GC/MS/DS printout.
- . Infrared Spectrum Traces

The Supervisor will spot check this information for the correctness of analytical calculations as well as any interpretations(s) and/or assumptions contained in the data. If all of this information is deemed correct, the Supervisor should sign the respective notebook pages. If there is a problem with the data package, the respective Supervisor should review this information with the analyst. If the data package is inconclusive or unacceptable, the work should be repeated. At this point, the analyst should be provided with a specific set of instructions aimed at preventing the re-development of additional inconclusive or unacceptable data packages.

9. INSTRUMENTATION

. Analytical Balance

- When the balance is not in use, all weights are to be returned to 0, and the balance left on the arrested position and cleaned thoroughly.
- The balance is to be certified and checked once a year by a balance servicing company.
- The balance is to be checked once per month with class S weights, over the range of 5 mgs to 100 grams.

. pH Meter

- The meter is to be standardized against two buffers that bracket the pH of the sample. The buffers used to standardize the pH meter must be documented in the lab notebook and a separate calibration log book.
- Keep electrodes immersed in water when not in use, and filled with filling solution.

. Conductivity Meter

- The cell constant is to be determined monthly with standard 0.01M KCl solution.
- Twice per year the performance of the conductivity cell will be checked against five (5) standards as shown in Table 205:I, pg. 73, Standard Methods for the Examination of Water and Wastewater, 14th edition.

. Turbidimeter

- Formazin Standards of 4 and 40 NTU are to be prepared as described on page 133 and 134 of Standard Methods for the Examination of Water and Wastewater, 14th edition.
- Samples of turbidity greater than 40 NTU are to be diluted into the range of the standards before analysis.

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9. INSTRUMENTATION (Continued). Spectrophotometer

- A quarterly calibration of the PE 550, UV visible spectrophotometer and/or the Bausch & Lomb Spectronic 20 shall be done for the following determinations:

- Nitrates
- Phosphates
- Boron
- Sulfide
- Nitrite
- Sulfate
- Hexavalent Chromium
- MBAS

Each calibration curve is to consist of 5 standards and a blank.

- Each time a set of analyses are done, a calibration curve is generated a mid and high level standard. The absorbance values for the standards must be consistent with previous curves. If they are not, the Lab Manager is to be notified.

. Auto Analyzer

- Five (5) standards and a blank are to be run at the beginning and end of the entire daily workload.
- Each rack shall contain a mid range standard at the beginning and end of each set of samples.

. Atomic Absorption Spectrophotometer

- The AAS is to be calibrated quarterly with five standards for each metal analyzed, everytime the analysis is determined. The absorbance values for the standards must be consistent with previous curves. If they are not the Lab Manager is to be notified.

9. INSTRUMENTATION (Continued)

. Atomic Absorption Spectrophotometer (Continued)

- Each set of samples should be aspirated twice.
- At the end of a given set of analysis the standards should be run, and the measured concentration recorded in the notebook.
- All ionization and chemical interferences must be eliminated or minimized when determining metals by AAS.

9. INSTRUMENTATION (Continued). Gas Chromatography

Gas Chromatography is one of the primary tools used to identify and quantify organic compounds in environmental media. In order to facilitate the interpretation of the analytical data developed and to insure the precision and accuracy of each analytical technique, the following will be adhered to:

- All useful gas chromatograms should be kept and filed. Each chromatogram should be referenced into a lab notebook where calculations based on a recent standard are recorded with all pertinent Quality Control information.
- All instrumental conditions should be recorded in the laboratory notebook. These conditions should not deviate from established methods.
- Standard injection volume should equal sample injection volume. Syringes should be checked to assure they are leak-resistant at column inlet pressures. (When possible, injection volumes should be in the 3-8 microliter range). Solvent flush techniques should be used whenever variable injection volumes are required.
- GC oven temperature should be monitored periodically either with an NBS traceable thermometer or by the relative retention time of p,p'-DDT versus Aldrin.
- Only high purity carrier gases are to be used.
- Septa should be changed when ever needed, but at least once daily when GCs are in use.
- GC columns and/or septa should not be removed or changed while oven is hot and/or while a positive inlet pressure remains.

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9. INSTRUMENTATION (Continued). Gas Chromatography (Continued)

The following information pertains to the operation of the flame-photometric detector and the electron-capture detector.

- Flame Photometric Detector (FPD)

- .. FPD flame conditions should be optimized for the exact column used for pesticide residue analysis before column evaluation begins (See Perkin-Elmer Manual for optimization procedures).
- .. Do not use re-silanized (Rejuv-8) columns with FPD. Column bleed causes permanent fogging of quartz heat shield.
- .. Linear range of FPD in phosphorous mode should be determined for each compound of interest. Standards for quantification should fall well within this linear range.
- .. Linear range of FPD in sulfur mode should be determined most carefully since the plot of peak area vs. concentration is an exponential equation. At least five standards could be used to determine the linear range. Standards for quantification should fall well within this range.

- Electron-Capture Detector (ECD)

The linear range of the ECD is generally in the range of 5 picograms to 1000 picograms of chlorinated pesticidal material. The linear range of the detector for each chlorinated compound of interest should be determined carefully. The standards used for quantification should fall well within this range.

- .. The breakdown of pp'DDT into its metabolites as well as the breakdown of Endrin, should be monitored closely to determine the reactivity of the column with these pesticidal constituents. If the ratio of the peak area of the breakdown products of DDT equals 3-5% or exceeds of the total and/or 10% for Endrin, the column should not be used for pesticide residue analysis. Changing the glass wool plug or treating the column with a silanizing agent may solve this problem.
- .. A standing detector current profile should be taken whenever a column is changed in the gas chromatograph. A change in the standing current profile is indicative of detector aging or contamination as well as unusual column deterioration or carrier gas impurities.

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9. INSTRUMENTATION (Continued). Gas Chromatography- Electron-Capture Detector (ECD)

- .. The ECD detector should be operated at a temperature at least 50°C higher than the highest column temperature to prevent the accumulation of residues. Routinely a detector temperature of 350°C is recommended.
- .. Do not change the range of the ECD detector while running a set of samples and/or standards. Set range at desired level of sensitivity (but within Linear Response Range) and maintain at this setting.

- Hall Detector

- .. A reaction tube temperature of at least 800°C for routine work and 900°C for PCB analyses must be maintained.
- .. Both hydrogen flow rates and electrolytic solvent flow rates must be optimized before use. These rates may not be changed in the middle of analysis.
- .. Reaction tubes and ion exchange resins must be changed periodically to prevent excessive baseline noise. (2 weeks-2 months depending on work load).
- .. Conductivity cell must be soaked in an ultrasonic phosphoric acid bath periodically (when previously mentioned steps do not reduce baseline noise).

. Gas Chromatography/Mass Spectrometry

Gas Chromatography/Mass Spectrometry is the most sensitive and reliable tool used to identify and quantify organic compounds in environmental media. In order to facilitate the interpretation of the analytical data developed and to insure the precision and accuracy of each analytical technique, the following will be adhered to:

- All useful quantitation printouts should be kept and filed. Printouts for each job should be kept in a file folder along with copies of the printouts for the daily standard(s) and spike(s).

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- Standard injection volume should equal sample injection volume. Syringes should be checked to assure they are leak-resistant at column inlet pressures. (When possible, injection volumes should be in the 2-4 microliter range for capillary columns, 5-10 microliter range for packed columns).
- GC oven temperature should be monitored periodically with an NBS traceable thermometer.
- Only high purity carrier gases are to be used.
- Septa should be changed when ever needed, but at least once daily.
- GC columns and/or septa should not be removed or changed while oven is hot and/or while a positive inlet pressure remains.
- The GC/MS should be tuned at the beginning of each 12 hour period with either 4-Bromofluorobenzene (BFB) for volatiles, or decafluoro-triphenylphosphine (DFTPP) for acid extractables and/or base/neutral extractables. The GC/MS should provide a mass spectrum in accordance with the specifications set forth in EPA methods 624 and 625 when 50 ng of either material is injected.
- A calibration curve, consisting of three points spaced over the linear range of the detector, should be acquired. The response factors should be entered into the appropriate response lists, and all samples should be quantitated based upon this curve
- The GC/MS manifold should be operated at 75°C at all times.
- The jet separator oven/transfer line should be operated at 235°C for packed column work, 290°C for capillary column work.
- The linear range of the GC/MS is generally in the range of .01 to 2 micrograms on the 1020, and .01 to 5 micrograms on the 5100's. For volatiles, 1-200 ng for base neutrals and 5-300 ng for acid extractables on the 5100's are the linear ranges.

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10. EQUIPMENT. Thermometer Calibrations

- Once a year the thermometers in the incubator, BOD chambers, walk-in refrigerators, and ovens are to be calibrated against an NBS thermometer, and documented in the QC book.
- Incubator, walk-in and refrigerator temperatures are to be documented daily in the QC book.
- BOD chamber temperatures are to be documented in the notebooks whenever BODs are put in operation or taken out of operation.
- Oven temperatures are to be documented in the notebook whenever solids determinations are done.

. Deionized Water

- Monthly checks of pH, specific conductance and residual chlorine are to be done on the laboratory deionized water, and documented in the QC book.
- Once per year a complete heavy metals determination and suitability test is to be performed on the laboratory deionized water supply.

. Glassware

- Only Class A volumetric glassware is to be used in the laboratory.
- For trace metals and organics in drinking water samples, a special set of glassware will be reserved for sample preparation.
- Borosilicate glass or polyethylene bottles are to be used for reagents and solutions. No metal lids should be used.
- Samples or solutions to be analyzed for boron, silica or fluoride should not be stored in borosilicate glass.
- Glassware to be used in metal determinations should be rinsed with 1:1 nitric acid:water.
- Glassware to be used in trace organic analysis should be rinsed with chromic acid solution followed by distilled water, and then rinsed with pesticide grade acetone, hexane, and oven dried.
- In phosphate analyses, all glassware must be cleaned with phosphate-free detergent, and acid rinsed with 1:1 H₂SO₄:water solution.
- Glassware for ammonia determinations must be rinsed with ammonia-free water, prepared by passing the deionized water through a cation exchanger.
- All other glassware is to be cleaned thoroughly with phosphate-free detergent, and rinsed with deionized water.

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11. REPORTING OF ANALYTICAL RESULTS

In order to assure a uniform reporting system, Stablex-Reutter adheres to the following system:

All results are reported to three significant figures if above 1% (is XX.X;X.XX); to two significant figures if below 1% (is 0.XX;0.0XX). At the parts per million level, results are reported to only two significant figures (is XX0;XX;0.XX). In all cases the figures reported should be significant; that is, a variation of no more than ± 5 in the last significant figure reported.

It is good practice to retain no more figures than is significant in all preliminary calculations, and have one extra figure recorded in the notebooks, with the final reports having the correct number of significant figures.

All calculations are to be maintained in the notebooks.

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12. MICROBIOLOGY

The following tests or measurements with the noted frequencies are to be conducted on the Laboratory water used in microbiological analyses.

- . Annual - Laboratory Pure Water
 - Suitability, metals
- . Monthly - D.I. water - conductance, pH
- . Daily - Incubator temperatures (logged in book)

Two types of bacteriological analyses are conducted by the Laboratory Services Department:

- Membrane Filtration (MF) Analysis
- Most Probable Number (MPN) Analysis for Coliforms

(A) Membrane Filtration Analysis (MF)

- . Each Occurrence
 - Media - 1) pH (logged in book)
2) (+) or (-) control (logged in book)
 - Confirmation - all (+) Total Coliform analyses confirmed through Lauryl Tryptose and Brilliant Green Lactose Bile series
 - Autoclave
 - .. Record time of sterilization cycle
 - .. Pressure
 - .. Temperature
 - .. Place sterile indicator in autoclave
 - . Use of prepared refrigerated media - before use, incubate at 35°C for 24 hours.
 - Media
 - .. Date media upon receipt and refrigerate
 - .. Open bottles expired after six months
 - .. Sealed bottles expired after one year
 - Prepared media
 - .. Total Coliform agar (M-Endo) - prepared plates good for 48 hours.
 - .. Fecal Coliform agar (MFC) - prepared plates good for 72 hours.
 - .. Fecal Streptococcus agar (KFC) - prepared plates good for 90 days.

All the above conditions apply when media is kept refrigerated and dark.

12. MICROBIOLOGY (Continued). Filters

- Only certified membrane filters are to be used
- Certification slips (which also contain lot #'s) are to be retained and placed in file

. Analyses

- At least one (1) analysis per month should be a side by side MPN vs filter technique. The MPN series should be 15 tubes (5-10 mls, 5-1 ml, 5-0.1 ml). Results to be recorded side by side in notebook.
- Counting - only colonies that produce characteristic green sheen are to be counted. If a positive result occurs, it is to be confirmed via the Lauryl Tryptose-BGLB series. If more than 5 colonies, at least 5 of the colonies should be confirmed. If a colony producing anything other than the green sheen, i.e., pink or red, this colony or colonies should be subjected to Lauryl Tryptose fermentation test.
- Reporting
 - .. When using filter membrane if no colonies grow report 0 colonies/100 ml

(B) MPN (Most Probable Number) Analysis for Coliforms

- . Sample Preservation - all samples for Coliform analysis are to be collected in sterile bottles that contain a satisfactory dechlorinating agent (e.g., Sodium thiosulfate $\text{Na}_2\text{S}_2\text{O}_3$). The samples should be analyzed as soon as possible after receipt in the laboratory. Samples may not exceed a 24 hour time limit. Samples can be stored up to 24 hours at 4°C.
- . Analysis - Samples are to be analyzed according to procedures set forth in Method 908, "Multiple Tube Fermentation Technic for Members of the Coliform Group", Standard Methods for the Examination of Water and Wastewater, 15th edition.

All pipets, fermentation tubes, lauryl tryptose broth and dilution water are to be checked for sterility prior to analysis with appropriate indicators. Records are to be kept in laboratory notebooks on all sterility checks.

Samples are to be subjected to a series of dilutions. The volume of the dilution is dependent on the nature of the sample. Normally a five tube multiple dilution series of 10.0 mls, 0.1 mls and 0.01 mls will be used for each sample.

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10. MICROBIOLOGY (Continued)(B) MPN (Most Probable Number) Analysis for Coliforms

- . All sample tubes are to be checked after an incubation period of 24 hours at $35 \pm 0.5^{\circ}\text{C}$ for turbidity and/or gas formation. Samples showing positive (+) gas formation after 24 hours are to be considered a positive result. Samples are then rechecked after an additional 24 hours at $35 \pm 0.5^{\circ}\text{C}$.
- . After a total of 48 hours at $35 \pm 0.5^{\circ}\text{C}$ all positive tubes (turbidity and/or gas formation) are to be subjected to a confirmatory test with Brilliant Green Lactose Bile Broth.
- . Data and Results - all pertinent data is to be recorded in laboratory notebooks. This data must include sample designations, tube numbers and dilution volumes as well as analysis initiation times and the times, dates and results for all incubation periods.
- . Reporting:
 - All results are to be reported as the MPN Index/100 mls.
 - If no colonies grow report <2 colonies/100 mls

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13. BIBLIOGRAPHY

Summary of method manuals and publications currently used by S-A Analytical, Inc.

- Sampler and Sampling Procedures for Hazardous Waste Streams, EPA 600/2-80-018, January, 1980.
- Manual for the Interim Certification of Laboratories Involved in Analyzing Public Drinking Water Suppliers (Criteria and Procedures), EPA 600-8-78-008, May, 1978.
- Safety Manual for Hazardous Waste Site Investigations, Draft, September, 1979.
- Manual for Analytical Quality Control for Pesticides and Related Compounds in Human and Environmental Samples, EPA 600/1-79-008, January, 1979.
- Handbook for Analytical Quality Control in Water and Wastewater Laboratories, EPA 600/4-79-019, March, 1979.
- Standard Methods for the Examination of Water and Wastewater, 14th edition, 1980, APHA-AWWA-WPCF.
- Official Methods of Analysis of the Association of Official Analytical Chemists, 12th edition, 1975.
- Atomic Absorption Newsletter.
- Metals Handbook, Volume 1, 8th edition, American Society for Testing Materials.
- Manuals of Analytical Methods for the Analysis of Pesticides in Human and Environmental Samples, EPA 600/8-80-038.
- 1982 ASTM Standard (all parts applicable to S-R).
- Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020, March, 1979.
- Food and Drug Administration, Pesticide Analytical Manual, Volume I and Volume II (parts 1-3), August 1977.
- Federal Register, Vol. 44, No. 233, July, 1982, Revision.
- Federal Register, Vol. 45, No. 98, May 19, 1980.

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APPENDIX

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APPENDIX A

RECOMMENDATION FOR SAMPLING AND PRESERVATION OF SAMPLES ACCORDING TO MEASUREMENT (1)

<u>Measurement</u>	<u>Vol. Req. (ml)</u>	<u>Container (2)</u>	<u>Preservatives</u>	<u>Holding Time (3)</u>
<u>Physical Properties</u>				
Color	50	P,G	Cool, 4°C	24 Hrs.
Conductance	100	P,G	Cool, 4°C	24 Hrs. (4)
Hardness	100	P,G	Cool, 4°C HNO ₃ to pH <2	6 Mos. (5)
Odor	200	G only	Cool, 4°C	24 Hrs.
pH	25	P,G	Det. on site	6 Hrs.
<u>Residue</u>				
- Filterable	100	P,G	Cool, 4°C	7 Days
- Non-Filterable	100	P,G	Cool, 4°C	7 Days
- Total	100	P,G	Cool, 4°C	7 Days
- Volatile	100	P,G	Cool, 4°C	7 Days
Settleable Matter	1000	P,G	None Req.	24 Hrs.
Temperature	1000	P,G	Det. on site	No Holding
Turbidity	100	P,G	Cool, 4°C	7 Days
<u>Metals</u>				
Dissolved	200	P,G	Filter on site HNO ₃ to pH <2	6 Mos. (5)
Suspended	200		Filter on site	6 Mos.
Total	100	P,G	HNO ₃ to pH <2	6 Mos. (5)

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<u>Measurement</u>	<u>Req. (ml)</u>	<u>Container (2)</u>	<u>Preservative</u>	<u>Holding Time</u>
Mercury , Dissolved	100	P,G	Filter on site HNO ₃ to pH <2	38 Days (Glass 13 Days)
Mercury, Total	100	P,G	HNO ₃ to pH <2	(Hard Plastic)
<u>Inorganics, Non-Metallics</u>				
Acidity	100	P,G	None Req.	24 Hrs.
Alkalinity	100	P,G	Cool, 4°C	24 Hrs.
Bromide	100	P,G	Cool, 4°C	24 Hrs.
Chloride	50	P,G	None Req.	7 Days
Chlorine	200	P,G	Det. on site	No Holding
Cyanides	500	P,G	Cool 4°C NaOH to pH 12	24 Hrs.
Fluoride	300	P,G	None Req.	7 Days
Iodide	100	P,G	Cool, 4°C	24 Hrs.
Nitrogen				
- Ammonia	400	P,G	Cool, 4°C H ₂ SO ₄ to pH <2	24 Hrs.
- Kjeldahl, Total	500	P,G	Cool, 4°C H ₂ SO ₄ to pH <2	24 Hrs. (b)
- Nitrate plus Nitrite	100	P,G	Cool, 4°C H ₂ SO ₄ to pH <2	24 Hrs. (c)
- Nitrate	100	P,G	Cool, 4°C	24 Hrs.
- Nitrite	50	P,G	Cool, 4°C	48 Hrs.

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<u>Measurement</u>	<u>Vol. Req. (ml)</u>	<u>Container (2)</u>	<u>Preservatives</u>	<u>Holding Time</u>
Dissolved Oxygen				
- Probe	300	G only	Det. on site	No Holding
- Winkler	300	G only	Fix on site	4-8 Hours
Phosphorous				
- Ortho-phosphate Dissolved	50	P,G	Filter on site Cool, 4°C	24 Hrs.
- Hydrolyzable	50	P,G	Cool, 4°C H ₂ SO ₄ to pH <2	24 Hrs.(6)
- Total	50	P,G	Cool, 4°C H ₂ SO ₄ to pH <2	24 Hrs.(6)
- Total Dissolved	50	P,G	Filter on site Cool, 4°C H ₂ SO ₄ to pH <2	24 Hrs.(6)
Silica	50	P only	Cool, 4°C	7 Days
Sulfate	50	P,G	Cool, 4°C	7 Days
Sulfide	500	P,G	2 ml zinc acetate	24 Hrs.
Sulfite	50	P,G	Det. on site	No Holding
<u>Organics</u>				
BOD	1000	P,G	Cool, 4°C	24 Hrs.
COD	50	P,G	H ₂ SO ₄ to pH <2	7 Days(6)
Oil and Grease	1000	G only	Cool, 4°C H ₂ SO ₄ of HCL to pH <2	24 Hrs.
Organic carbon	25	P,G	Cool, 4°C H ₂ SO ₄ of HCL to pH <2	24 Hrs.
Phenolics	500	G only	Cool, 4°C H ₃ PO ₄ to pH <4 1.0 g CuSO ₄ /l	24 Hrs.
MBAS	250	P,G	Cool, 4°C	24 Hrs.

Quality Assurance Program

<u>Measurement</u>	<u>Vol. Req. (ml)</u>	<u>Container (2)</u>	<u>Preservative</u>	<u>Holding Time</u>
NTA	50	P,G	Cool, 4°C	24 Hrs.

1. More specific instructions for preservation and sampling are found with each procedure as detailed in the EPA Manual of Chemical Analysis of Water and Wastes. A general discussion on sampling water and industrial wastewater may be found in ASTM, Part 31, p. 72-82 (1976) Method D-3370.
2. Plastic (P) or Glass (G). For metals, polyethylene with a polypropylene cap (no liner) is preferred.
3. It should be pointed out that holding times listed above are recommended for properly preserved samples based on currently available data. It is recognized that for some sample types, extension of these times may be possible while for other types, these times may be too long. Where shipping regulations prevent the use of the proper preservation technique or the holding time is exceeded, such as the case of a 24-hour composite, the final reported data for these samples should indicate the specific variance.
4. If the sample is stabilized by cooling, it should be warmed to 25°C for reading, or temperature correction made and results reported at 25°C.
5. Where HNO₃ cannot be used because of shipping restrictions, the sample may be initially preserved by icing and immediately shipped to the laboratory. Upon receipt in the laboratory, the sample must be acidified to a pH <2 with HNO₃ (normally 3 ml 1:1 HNO₃/liter is sufficient). At the time of analysis, the sample container should be thoroughly rinsed with 1:1 HNO₃ and the washings added to the sample (volume correction may be required).
6. Data obtained from National Enforcement Investigations Center, Denver, Colorado, support a four-week holding time for this parameter in Sewerage Systems. (SIC4952)

APPENDIX B

Procedure No. 101

Title: Chain of Custody

Date: January 1983

Scope of Application

Chain of Custody ensures that sample integrity and traceability is maintained from the moment of sampling until storage after analysis. It is applicable to all X-029 samples.

Summary of Method

A description of all samples and a chronological record of the person who takes custody of these samples is recorded on a Chain of Custody Record.

Apparatus

- Stablex-Reutter Chain of Custody Record Form No. 83-10 (Exhibit I)
- Black of blue nonerasable pen

Procedure

1. The sampler is to fill in Section I with the clients name and address. If this is proprietary, leave blank.
2. Section II is filled out as each sample is taken. Record the number of the sample, the container type and a description and/or designation of the sample. If there are any safety concerns or any other pertinent data, fill in the "Remarks" section.
3. Section III is filled out by the person who takes custody of the sample from the sampling team and delivers them to Stablex-Reutter, Inc. (S-R).
4. Section IV is completed as custody of the sample is initiated and relinquished by all those who subsequently handle the sample. FILL IN ALL THE REQUIRED INFORMATION. Names must be printed and then signed.
5. In the case of interlaboratory custody changes, Group Leaders can sign the samples out for extended periods for multiple analysis. Individual analysts do not have to receive and relinquish via the Chain of Custody form. However, the Group Leader is responsible for the samples handled by his/her group and must maintain strict control over the proper care, handling and storage of the samples.

APPENDIX C

Procedure No. 102

Title: Log In Procedure

Date: August 23, 1984

Scope and Application

This procedure ensures that samples delivered to the laboratory by S-R sampling personnel or by outside personnel are quickly and accurately registered into the laboratory data system.

Method

1. Assign sample number in book and initiate customer order (C.O.) with job number and client name.
2. Note temperature of samples on arrival; note on the customer order.
3. Date listed should be date sample was taken or date of arrival, not log-in date.
4. Marketing personnel should submit paperwork for each sample prior to or coinciding with its arrival. Check paperwork for the following information:
 - . Client Name
 - . Priority
 - . Analysis Requested
5. Based on appearance and/or physical tests, record matrix:
 1. Aqueous
 2. Non-Aqueous
 3. Solid
6. List any safety concerns, if known.
7. List bottle types .
8. Check samples for proper preservatives. If samples arrive unpreserved, divide the samples into appropriate aliquots and preserve. Note this on the customer order.
9. Check the chain of custody against the samples and sign it.
10. List sample designations (and descriptions) and corresponding S-R #.
11. Circle appropriate analysis and write in the number of samples to be analyzed.
12. Note any information that could have bearing on the analysis. (eg., small sample volume, sample not properly preserved, etc.)
13. Prepare label for each individual bottle with the S-R number. Assign a place in the walk-in for the sample(s) using the orange walk-in log book and the following system of sample labelling:
 - . yellow sticker - normal handling
 - . red sticker - do not throw out sample

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14. If samples are to be analyzed for pH notify section supervisor and leave the unpreserved portion of the sample to equilibrate to room temperature.
15. Bring C.O. to Marketing for approval or answers to questions.
16. Prepare client folder with S-R# and client name designated.
17. After Marketing has approved the C.O. with the salesman's initials, make copies of the C.O. and distribute as follows:
 - . original - to client folder
 - . 1 copy - to Supervisor of Organic Section - only if the following analyses groups are circled:

GC/MS
GC
O & G
TOC
 - . 1 copy - to supervisor of inorganic section (if any other analysis or group analyses are circled)
 - . 1 copy - to the Laboratory Manager
 - . 1 copy - to the Project Manager (if applicable)
18. Supervisors must be notified as soon as possible so that they can schedule work. If a sample has a priority of 72 hours or less, notify the supervisors even before the paperwork is complete. If the sample requires BOD, pH or another "immediate" analysis, notify the inorganic supervisor.
19. All information that arrives with the samples should be kept in the client folder along with the C.O.
20. The samples are then assigned a location in the walk-in and are labelled accordingly.
21. Each Friday, check the test report log (see the office manager) for those reports that have been mailed that week. Make a list of the S-R number and client names.
22. Remove those samples from the walk-in and put in the storage room. Update the walk-in log book accordingly.

S-R No. _____

Date Submitted _____ Date Due _____

Customer: _____

Report Address _____

Contact: _____

Salesperson: _____

Sampled By _____ Sample Qty _____ Matrix _____ Code _____

S-R _____ AQ 01
Client _____ ORG 02
_____ SOLID 03
_____ OTHER _____
_____ TOTAL

Applicable Regulations/Agency: X029;EPA;
SDWA 1,2&WW;NEW FORMAT;ECRA;OSHA;DEP

Sample Disposition - Bottles

Received properly preserved

Quantity | Type YES NO DONE AT LAB

Sample Designation

S-R No. | Client Description

Metals	DEP/EPA Holding Times	General Methods	DEP/EPA Holding Times
Prep	6M	BOD	6H/48H
AS	6M	COD	7D/28D
SE	6M	PHEN	24H/28D
SB	6M	TS	7D
HG	38D(G)13D(P)	TDS	7D
BA	6M	TSS	7D
BE	6M	TVS	7D
CD	6M	ALKAL	24H/14D
CA	6M	CHLORIDE	7D/28D
CR	6M	HARDNESS	7D/6M
CU	6M	NITRATE	24H/28D
FE	6M	NH ₃ -D	24H/28D
PB	6M	TKN	7D/28D
MG	6M	PO ₄	24H/48H
MN	6M	T PHOS	7D/28D
NI	6M	SO ₄	7D/28D
K	6M	FLUORIDE	7D/28D
SI	6M	CN-D	24H/14D
AG	6M	CN-A	24H/14D
NA	6M	REACT	24H*
TL	6M	FLASHPT	14D*
ZN	6M	pH	NONE
CR-6	24H	SULFIDE	24H
PP MET		T SUL	-
SDWA/RCRA		VISC	-
EP PROC	14D	ASH	-
		SP. GRAV	-
		SP. COND	24H/28D
		COLOR	24H/48H
		BS+W	-
		H ₂ O-DS	-
		API	-
		BTU	-
		TURB	7D/48H
		ASB PC	-
		ASB XRD	-

GC/MS

M624 14D
M625-AE 7D**
M625-BN 7D**
NBS-VOL 14D
NBS-NONVOL 7D

GC/Organic

HERB 3D/7D**
PCBs 3D/7D**
SDW-PEST 3D/7D**
PP-PEST 3D/7D**
PETHCS-GC 24H/28D*
PETHCS-IR 24H/28D*
TOX 14D*
TOC 24H/28D
O+G 24H/28D

Bacteriology

T COLI-MF 6H(WW) &
F COLI-MF 30H(FW)

Other Analysis

Special Instruction/Concerns

* Best Estimate
** Extractions can be
held longer

PROTECT COST

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APPENDIX D

Cleaning Procedure for Sample Containers

A. Preliminary

1. Only glassware that is without scratches, stains or chips shall be used. Plasticware, likewise, should have no markings, stains or scratches.
2. All glassware is thoroughly washed with a hot solution of a non-phosphate detergent and rinsed several times with tap water and then deionized water.
3. This procedure is sufficient for analyses that do not require preservation or trace analysis of pesticides, base/neutrals or acid-extractable compounds.

B. Additional Preparation of Sample Container for Purgeable Organic Analysis (Aqueous)

1. After a 40 ml glass vial with screw cap is detergent washed and rinsed with water, (both tap and deionized) it is then dried at 105°C for one hour.
2. A teflon-faced silicone septum is taken through the same procedure and after cooling, the septum is placed in the cap with the teflon side facing the interior of the vial.

NOTE: Due to the possibility of diffusion of organics through the seal to contaminate the vial contents, a second vial should be prepared and filled with distilled water. This vial constitutes a sample blank and should be carried through the sampling and handling protocol.

C. Additional Preparation of Sample Container for Pesticide Analysis (Aqueous)

1. The initial wash should be performed on a 1 liter (Wheaton) glass bottle with teflon lined cap, but performed in a high temperature (50°C) bath of detergent in water.
2. After rinsing with tap water and distilled water, the bottle should next be rinsed with aliquots of acetone and hexane.

D. Additional Preparation of Sample Container for Metals Analysis (Aqueous)

1. The sample bottle may be of borosilicate glass, linear polyethylene, propylene or teflon, 1 liter size.
2. After the initial detergent wash and rinse with tap and deionized water, the bottle is then rinsed with aliquots of 1:1 nitric acid, tap water, 1:1 hydrochloric acid, tap water and then distilled deionized water, in that order.

NOTE: Preservation of the sample occurs at the time of sampling. Appropriate preservations will be supplied by S-R upon request.

Cleaning Procedure
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E. Preparation of Sample Container for Cyanide Analysis (Aqueous)

1. The container should be of brown borosilicate glass, preferably 1 liter volume.
2. After the detergent wash, the container should be rinsed with tap water, 1N NaOH. Then distilled deionized water.

NOTE: Preservation of the sample occurs at the time of sampling. Appropriate preservatives will be supplied by S-R upon request.

F. Additional Preparation of Sample Container for Phenol Analysis (Aqueous)

1. The container should be of brown, borosilicate glass, and may be as small as 40 ml.
2. After the detergent wash, the container should be rinsed with tap water then a solution of copper sulfate-phosphoric acid. The final rinse is with distilled, deionized water.

NOTE: Preservation of the sample occurs at the time of sampling. Appropriate preservatives will be supplied

G. Additional Preparation of Sample Container for Oil and Grease. TOC Analysis (Aqueous)

1. The container should be borosilicate glass, preferably 1 liter volume.
2. After the initial detergent wash, the bottle is rinsed with tap water, 1:1 hydrochloric acid, then distilled, deionized water, in that order.

NOTE: Preservation of the sample occurs at the time of sampling. Appropriate preservatives will be supplied by S-R upon request.

H. Additional Preparation of Sample Container for Ammonia, Nitrogen and COD Analysis (Aqueous)

1. The container may be borosilicate glass, linear polyethylene or propylene, preferably 250 ml volume.
2. After the initial detergent wash, the container is rinsed with tap water, 1:1 sulfuric acid and distilled, deionized water, in that order.

NOTE: Preservation of the sample occurs at the time of sampling. Appropriate preservatives will be supplied by S-R upon request.

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I. Additional Preparation of Sample Container for Bacteria Analysis (Aqueous)

1. The container should be linear polyethylene or propylene, preferably 300 ml size, capable of withstanding sterilization temperatures.
2. After the initial detergent, rinse thoroughly with tap water, distilled, deionized water and dilute nitric acid, in that order. (This last rinse will remove any possible heavy metal contamination).
3. Perform a final rinse with distilled, deionized water and dry by draining.
4. Add sodium thiosulfate in an amount sufficient to provide a concentration of approximately 100 mg/l in the sample if the sample is expected to contain residual chlorine.
5. Cap the bottle and wrap neck and top with aluminum foil.
6. Sterilize the bottle in a dry, hot-air sterilizer at a minimum of 170°C for at least 1 hour.

J. Sample Container Closure (not including 40 ml vials)

1. Upon obtaining samplers, the sampler (EPA representative) shall hold the container upright and replace the lid with clockwise turns of the wrist until the lid is tightly attached.
2. Chain of Custody tape should then be affixed over the lid of the bottle such that the tape must be broken in order to open the container.

K. Sample Container Closure (40 ml vials)

1. The sampler (EPA representative) should endeavor to close the vial while still in contact with the sample in order to ensure that the bottle is sealed without an airspace.
2. Re-attach the lid with the same clockwise effort as delineated above. Once tightly capped, insert the bottle to check for air bubbles. If none are present, seal the vial.
3. Chain of Custody tape is affixed to cap so the the tape is ripped upon opening on the vial.

APPENDIX E

Cleaning of Equipment to be Used in Sampling

A. List of Equipment

1. Open Tube (Thief) Sampler
2. Coliwasa Sampler
3. Kemmerer Bottle Sampler
4. Weighted Bottle Sampler
5. Wheaton Grab Sampler
6. Bacon Bomb Sampler
7. Ponar Dredge Sampler
8. Peterson Dredge Sampler
9. Soil Coring Device Sampler
10. Vehimeyer Sampler
11. Split Spoon (Split Barrel) Sampler
12. Grain Sampler
13. Trier Sampler
14. Waste Pile Sampler
15. Bailer
16. Silver Bullet Sampler
17. Pond Sampler
18. Surface Skimmer Sampler
19. Submersible Sampler
20. Trowel Sampler
21. Wire Rope Sampler

B. Dismantling and Cleaning Procedure

1. Prior to cleaning, the samplers should be dismantled as much as possible, according to manufacturer's specifications.

NOTE: Rubber or plastic gloves should be worn in performing all dismantling and cleaning steps until rinsing.

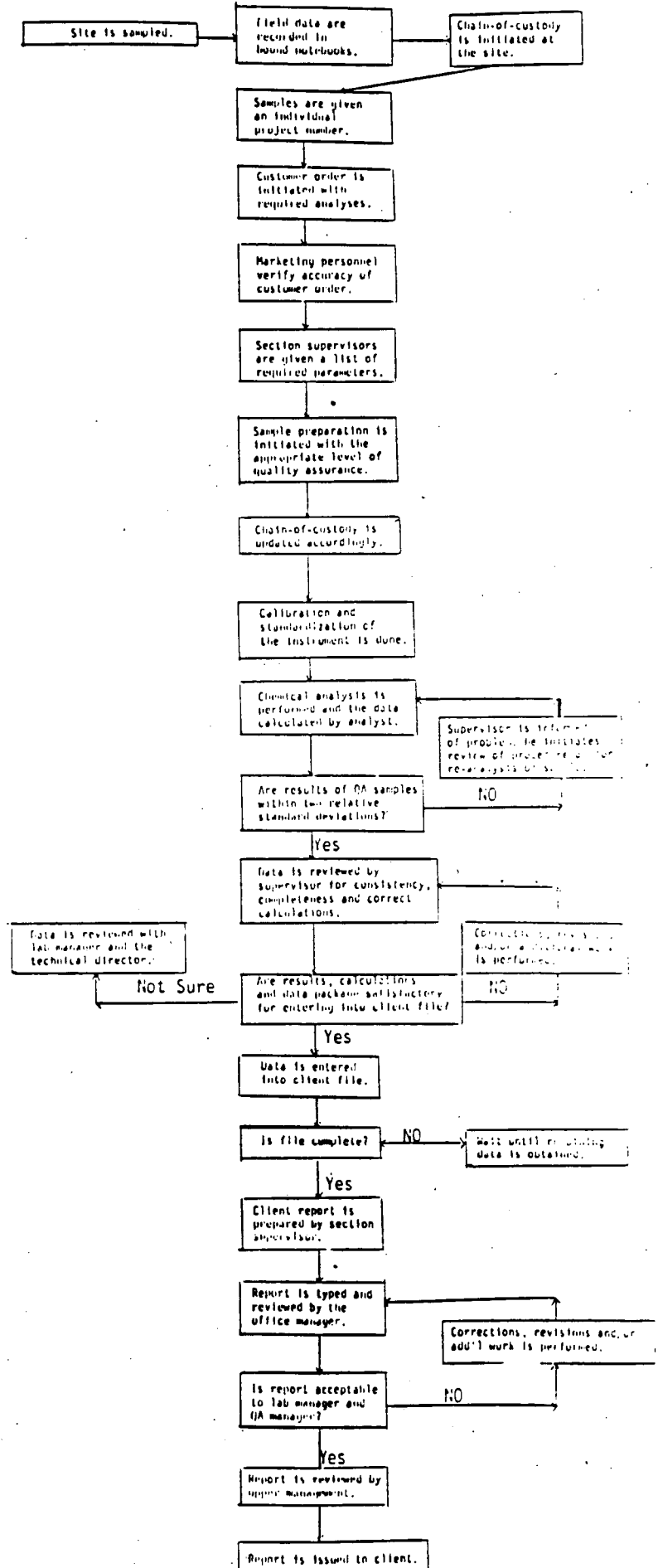
2. Any dried residue or mud should be physically removed by rubbing with paper towels or by hand.
3. Any organic film or residue may be removed by initial rinsing with acetone or other solvent.

NOTE: Manufacturer's specifications should be checked for compatibility of chosen solvent with sampler material.

4. The disassembled sampler is then washed thoroughly in a hot detergent solution and rinsed with tap water, distilled deionized water and then acetone, in that order.
5. The sampler is then re-assembled and the openings wrapped/sealed with sterilized aluminum foil to prevent contamination.

LABORATORY FLOW CHART

APPENDIX F



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Stablex-Reutter Inc.

RESUME

William P. Dolan
General Manager

EDUCATION

B.S. in Chemistry, 1967, University of Maryland, College Park, MD
A.A.S. in Chemical Technology, 1959, State University of New York Farmingdale, NY

EXPERIENCE

Mr. Dolan is responsible for developing client contacts, project administration, proposal strategies and the overall marketing effort. Mr. Dolan is responsible for expanding the capabilities and services of the laboratory department. He is a specialist in the sampling, handling, disposal and analysis of toxic and hazardous industrial wastes. He is responsible for the preparation of proposals to industry and government and performs project management tasks of all laboratory contracts.

Mr. Dolan has experience in the following areas of industrial chemistry and environmental science:

- | | |
|------------------------|-------------------------|
| . Inorganic analyses | . Failure analysis |
| . Metallurgy | . Food analysis |
| . Corrosion analysis | . Air pollution testing |
| . Wastewater analyses | . Industrial hygiene |
| . Water chemistry | . Quality control |
| . Toxic waste analysis | . Pigment chemistry |

Previously, Mr. Dolan was with John G. Reutter Associates of Camden, NJ. He guided the growth of a one man basic wastewater analysis laboratory into a multidisciplinary laboratory recognized as a leader in hazardous waste sampling and analysis. He has managed numerous projects involving the sampling of abandoned hazardous waste sites, compositing and compatibility of wastes, analyses of wastes for heavy metals, priority pollutants, physical parameters and conducted leachate tests of the wastes. Mr. Dolan has coordinated many projects under contract to the State of New Jersey Department of Environmental Protection Division of Hazard Management. Mr. Dolan managed an industrial hygiene analysis program for ethylene thiourea using air sampling and urine analysis for monitoring under contract with a major chemical company. He supervised a large sample volume contract with the City of New York involving projects with three thousand samples per project. He managed a project with a major chemical company in providing specialized analyses for trace amounts of halogenated hydrocarbons in effluent discharge to a river. This project involved the development of new analysis protocols.

Prior to joining John G. Reutter Associates Mr. Dolan was with Booz, Allen & Hamilton in Florham Park, New Jersey as a Research Associate.

Stablex-Reutter Inc.

William P. Dolan
Page 2

He conducted a major odor evaluation study for a medium sized wastewater treatment plant in Gloversville-Amsterdam, New York. This study utilized dispersion modeling to predict the travel of odorous emission from the plant. Air pollution control recommendations were made. Mr. Dolan also developed techniques for the sampling and analysis of containerized PVC pellets for residual vinyl chloride monomer. This work was performed for a major Ohio based supplier to the automotive industry.

At Rossnagel & Associates in Cherry Hill, NJ Mr. Dolan was engaged as the Laboratory Manager. He participated in numerous air pollution emission tests and coordinated stack testing projects with up to five stack test teams on-site acting in concert. He expanded and developed the laboratory into wastewater analysis, industrial hygiene and hazardous wastes.

Previously Mr. Dolan was with E.I. Dupont de Nemour's Marshall Laboratory in Philadelphia, PA as a Chemist. He conducted studies on the dispersion of pigments into organic media. This work included the development of a rapid test for measuring the coloring strength of dispersed pigments.

Additionally Mr. Dolan was with the USDA, Agricultural Research Service in Beltsville, Maryland where he did developmental work on methods of analysis for aflatoxins and proteins in food grains.

As a Chemist and Metallurgist with Fairchild Camera and Instrument Corporation, Mr. Dolan performed chemical and physical analyses of ferrous and non-ferrous alloys. Physical tests included tensile and yield strengths, hardness, metallographic examination and failure analyses. He also had the responsibility to manage and treat the discharge of electroplating wastewater from an 80 tank facility.

PROFESSIONAL AFFILIATIONS

American Chemical Society
American Society for Testing and Materials
Chromatography Forum
Air Pollution Control Association

PUBLICATIONS

"Odor Panels" Chapter 5, Industrial Odor Technology assessment, Ann Arbor Science Publishers, Inc., 1975.

"Gas Chromatographic Analysis of Organic Emissions," Chapter 7, same book as above.

"Rapid Semi-Quantitative Analysis of Aflatoxin in Corn" Journal of Official Analytical Chemists, 1969.

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RESUME

Catherine Ward
Project Manager

EDUCATION

B.S. in Biology, 1980, St. Joseph University
J.D. Candidate, Rutgers University Law School

EXPERIENCE

Mrs. Ward has over five years of lab chemistry experience related to public health and environmental analysis for identification of chemical substances. Major projects have included the following areas of industrial chemistry and environmental science: inorganic analyses, wastewater analyses, water chemistry, toxicity testing, and toxic waste analysis. Mrs. Ward has had operational responsibilities for all inorganic analyses in the Laboratory and experience in many categories.

- . Supervision of a staff of chemists and technicians conducting wet chemical analyses, atomic absorption and ICP spectroscopy.
- . Performance of detailed chemical and physical analyses of a wide variety of industrial waste liquids, sludges and solids. She is responsible for thoroughly characterizing wastes, assigning hazard codes and recommending safe handling procedures.
- . Performance and evaluation of leachate tests on treated and untreated waste materials including the EPA-extraction procedure, Illinois Leachate Test, ASTM Methods A and B Shake Test, Japanese Leachate Test, and other leaching protocols.
- . Evaluation of toxic and hazardous wastes streams in accordance with the EPA Guidelines and Regulations for identification and listing of hazardous waste.

Mrs. Ward has been responsible for many of the activities of the Laboratory Department, including:

- . Care and maintenance of the laboratory instrumentation.
- . Quality control and quality assurance of the Laboratory.
- . Development of laboratory safety program

Mrs. Ward also was the in-house co-ordinator/construction manager for S-R's relocation to Cherry Hill, NJ, which involved the administration of over \$160,000.

PROFESSIONAL AFFILIATIONS

ASTM
Society of Applied Spectroscopy

SYN 001 1894

RESUME

James W. Shearard Jr.
Director of Marketing and
Special Projects

EDUCATION

B.A. in Environmental Studies, 1975, Colby College
M.B.A., 1983, Rutgers University

EXPERIENCE

Mr. Shearard has the primary responsibility of managing all Stablex-Reutter (S-R) projects with the State of New Jersey Department of Environmental Protection. He has coordinated projects with the following State agencies:

- . Division of Waste Management
- . Division of Criminal Justice
- . Bureau of Air Pollution Control
- . Bureau of Groundwater Management

Project duties, as key liaison contact for these agencies, included:

- . Contract preparation, pricing, and invoicing
- . Scheduling of S-R Hazardous Response Sampling Team
- . Coordination of all analytical work
- . Scheduling 48 hour service for reporting of analytical results
- . Maintaining telephone report log for reporting of results
- . Design and implementation of various Chain of Custody formats
- . Commitment of S-R personnel for project completion

Prior to joining Stablex-Reutter, Mr. Shearard was with Technological Resources, Inc. (TRI) (Campbell Soup) as Coordinator of Environmental Programs and Analytical Services. In this position he provided project control for the engineering and analysis services offered by TRI to government and food industries. Mr. Shearard worked with projects utilizing landframing techniques as a means of treatment and disposal for waste treatment plant effluents and sludges.

Resume

Ian C. Lambert
Laboratory Manager

Education

B. S. in Chemistry, 1976 Rutgers University

Experience

Mr. Lambert is responsible for coordinating all laboratory activities, planning, project management, providing technical expertise and ensuring the overall quality of all laboratory work. He is responsible for providing the staffing and training for expanding capabilities and providing the equipment to maintain state of the art analytical capability. He is responsible for reviewing all test reports, trouble shooting problem analyses and directing the development of new methods.

Mr. Lambert has experience in the following areas of chemistry:

- . Inorganic Analyses
 - Flame Atomic Absorption
 - Graphite Furnace Atomic Absorption
 - Flameless Mercury and Hydride Generation
 - Atomic Emission Spectroscopy
- . Organic Analysis
 - GC-FID, EC, FPD, TC
 - GC/MS/DS
 - Infrared Spectroscopy
 - Ultraviolet Spectroscopy
- . Toxic Waste Analysis
- . Bacteriology
- . Air Pollution Testing
- . Industrial Hygiene Monitoring and Analysis
- . Quality Control/Quality Assurance

Mr. Lambert was previously with Johnson Matthey Inc. of Wayne, PA. As Division Quality Control Manager he was responsible for directing a 35 member laboratory in maintaining the quality of automobile components manufactured for major American and Japanese automobile manufacturers. Analytical capabilities were DCP, XRF, XRD and SEM.

Prior to Johnson Matthey Inc., Mr. Lambert was with John G. Reutter Associates of Camden, N.J. He was responsible for helping guide the growth of a one man laboratory into a multidisciplinary analytical laboratory specializing in every kind of waste sampling and analysis. He has managed projects including sampling of hazardous waste sites, sampling of worker atmospheres, emission stacks, wastewater, estuarine and ocean monitoring.

Professional Affiliations

American Chemical Society
American Society for Testing and Material
American Industrial Hygiene Association
Chromatography Forum

RESUME

Michael Shmookler
Supervisor, Organic Section

EDUCATION

B.S. in Chemistry, 1975 Saint Joseph's College, Philadelphia, PA
M.S. in Chemistry, 1978 Saint Joseph's College, Philadelphia, PA
Ph.D. in Chemistry, 1984 Drexel University, Philadelphia, PA

EXPERIENCE

Dr. Shmookler's responsibilities include supervision of the organic chemistry section of Stablex-Reutter, Inc. Instrumentation under his auspices include:

- . (5) - Gas chromatographs with a total of six different detectors (FID, FPD, ECD, NPD, PID, HECD)
- . (1) - GC/MS/DS - Finnigan Model 1020 Organics in Water Analyzer
- . (2) - GC/MS/DS - Finnigan Model 5100
- . (1) - Infrared Spectrophotometer
- . (1) - Ion Chromatograph
- . (1) - TOC Analyzer

Duties also include methods development for analyzing trace contaminants in samples with complex matrices.

Dr. Shmookler conducted his Ph.D. research in the environmental analysis of air samples for trace levels of pollutants by gas chromatography.

In addition to supervision of chemists and spectroscopists, Dr. Shmookler also prepares project reports and provides proposal information.

PROFESSIONAL AFFILIATIONS

American Chemical Society
Delaware Valley Chromatography Forum
American Chemical Society, Philadelphia Section
Philadelphia Analytical Topical Group
Phi Lambda Upsilon

Mr. Shmookler is currently an evening college faculty member at Drexel University.

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RESUME

William A. Fithian
Inorganic Chemist

EDUCATION

B.S. in Chemistry, 1982, Glassboro State College, Glassboro, NJ.
B.A. in Geology, 1982, Glassboro State College, Glassboro, NJ.

EXPERIENCE AT S-R

Mr. Fithian has extensive experience in selected wet chemical analyses and is well versed in all EPA sample preparation protocols for the trace analysis of metals in aqueous and non-aqueous matrices. Mr. Fithian has operated and maintained two Atomic Absorption Spectrophotometers including one A.A. Graphite Furnace and also has operated and maintained an Ion Chromatograph. His past responsibilities include the quality control of metal analyses and all anion chromatography analyses and he ensured that certain safety standards were practiced and upheld in the laboratory.

CURRENT RESPONSIBILITIES

Mr. Fithian is responsible for the operation of one GC/MS along with the maintenance of all three GC/MS operated by S-R. Also, he is responsible for the Quality Control of the Volatiles of the EP Method 624 and Acid Extractable Base/Neutral Compounds by EP Method 625.

He has attended a Finnigan operators course for a better understanding of the Finnigan 5100 GC/MS.

RESUME

NAME: Charles Corcoran

TITLE: Analytical Chemist

EDUCATION OR DEGREE:

B.S. Chemistry, North Adams State College
North Adams, MA

PREVIOUS EXPERIENCE IN FIELD:

Century Laboratories
Analysis by GC/MS Volatiles, AE/BN
Dioxin (in addition to screen)
Analysis by GC for PCB, Pesticides, Herbicides
Volatiles
Analysis and method development for HPLC (Polynuclear
Aromatics, Sugars, Detergents)

EXPERIENCE AT S-R:

GC Analysis and Miscellaneous Pesticides, PCBs, Herbicides,
Pethydrocarbons, Other Analysis, GC/MS Analysis, Volatiles AE/BN
Also IR, TOC, Prep. Work (Limited)

CURRENT RESPONSIBILITIES:

GC/MS & GC operator
Analysis of Samples, Data Reduction

Member: American Chemical Society
Delaware Valley Chromatography Forum

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NAME: Debora Vogt

TITLE: GC/MS Technician

EDUCATION OR DEGREE:

A.A. in chemistry Delaware County Community College
Pursuing a B.S. (3rd year) in Chemistry, Widner University.

PREVIOUS EXPERIENCE IN FIELD:

EXPERIENCE AT S-R:

Mrs. Vogt's previous responsibilities have been for the routine organic quantitative analysis of environmental samples for trace levels of hydrocarbons. She is well versed in all EPA sample preparation and extraction protocols. She has operated four gas chromatographs with splitters and multidetectors plus a Hewlett - Packard Data System.

CURRENT RESPONSIBILITIES:

Mrs. Vogt is currently a GC/MS operator responsible for volatiles by EP Method 624.

RESUME

Lee F. Cramer
GC/MS Operator Microbiology Supervisor

EDUCATION

B.S. Biological Science, Drexel University
Graduate studies in microbiology, Hahnemann Medical College

EXPERIENCE AT S-R

Mr. Cramer's primary function is to perform the detailed extraction procedures on waste samples for ultimate analysis by QC and GC/MS. He has worked with numerous extraction protocols on a variety of wastes and wastewaters.

PREVIOUS EXPERIENCE IN THE FIELD

Previously Mr. Cramer was with Standard Branches as a Quality Control Laboratory Technicians. His duties included peroxide and dilatometer tests on vegetable oils, vitamin A assays, tests on keeping qualities of margarine, penetrometer tests on shortening, and occasionally assisting with production line monitoring of margarine for oil, water, salt and solids content.

At the Philadelphia Water Department, Mr. Cramer was an Industrial Wastes Chemist. His duties included testing industrial wastes for chemical and biological oxygen demands, and for acidity/alkalinity, nitrate and chloride contents. He also tested Delaware River water samples for biological oxygen demand, and tested garbage disposal units to determine acceptability according to city ordinances.

Mr. Cramer was a Microbiology Technician at the Smith, Kline and French Laboratories in Philadelphia, PA. His duties included screening for chemicals of possible pharmaceutical interest, chiefly by supplying promising substrates in the growth media of several hundreds of microorganisms and using thin layer chromatography techniques to separate metabolic products.

CURRENT RESPONSIBILITIES

Mr. Cramer is currently responsible for the operation of a GC/MS analysis for Volatiles by EP Method 624. He also is the supervisor for any microbiological analyses performed at S-R.

SYN 001 1901

NAME: Edward Palmer

TITLE: Chemist

EDUCATION OR DEGREE:

B.S. in Chemistry, 1983, St. Francis College

PREVIOUS EXPERIENCE IN FIELD:

Mr. Palmer has experience in physical organic chemistry by working for Argon National Laboratories.

EXPERIENCE AT S-R:

CURRENT RESPONSIBILITIES:

Mr. Palmer is a GC operator responsible for all GC operations in the organic laboratory.

SYN 001 1902

NAME: Howard Whaley

TITLE: Laboratory Technician

EDUCATION OR DEGREE:

Pursuing a degree (3 years) in Chemistry at Rutgers University

PREVIOUS EXPERIENCE IN FIELD:

Mr. Whaley was a laboratory technician at Campbell Soup Co.
performing the following analyses:

- Organic Prep Work
- GC Work
- EDB Analysis
- Total Fat Analysis
- Pesticides
- Chlorinated Pesticides

EXPERIENCE AT S-R:

Mr. Whaley was in charge of Organic Prep.

CURRENT RESPONSIBILITIES:

Mr. Whaley is responsible for operation of the GC/MS.

NAME: Donna McDonough

TITLE: Chemist

EDUCATION OR DEGREE:

B.S. in Environmental Science, 1983, Cook College

PREVIOUS EXPERIENCE IN FIELD:

EXPERIENCE AT S-R:

Miss McDonough has experience in doing organic extractions for GC and GC/MS analysis. These include Purgeable Organics, Polychlorinated Biphenyls, Pesticides and Non-Volatile Pollutants. Also, she is experienced in Total Organic Carbon Preparation.

CURRENT RESPONSIBILITIES:

Miss McDonough is responsible for analyzing for:

Pesticides

PCBs

Herbicides

Petroleum Hydrocarbons by GC

Total Organic Carbon Analysis

NAME: Cecily Mamrol

TITLE: Laboratory Technician

EDUCATION OR DEGREE:

Pursuing a degree (3rd year) in Environmental Science
at Drexel University

PREVIOUS EXPERIENCE IN FIELD:

Miss Mamrol is experienced as a laboratory technician for
the USDA. She is knowledgeable on the following analyses:

HPLC
TLC
Extraction
Supervision of Cultures

EXPERIENCE AT S-R:

Miss Mamrol is experienced in working on the GC in the Mobile
Wet Lab, phenols, reactivity, TOC's, compatabilities, and
organic preparation.

CURRENT RESPONSIBILITIES:

Currently Miss Mamrol performs the analysis for phenols along with
using the TOC analyzer and TOC sealer.

NAME: Andrea Pague

TITLE: Laboratory Technician

EDUCATION OR DEGREE:

B. S. in Chemistry, 1984, Dickenson College

PREVIOUS EXPERIENCE IN FIELD:

None

EXPERIENCE AT S-R:

CURRENT RESPONSIBILITIES:

Miss Pague is responsible for the following laboratory analyses:

Prep for the following tests:

Pesticides
PCB
Oil and Grease
Petroleum Hydrocarbons
Volatiles
TOC

She is experienced in using the following instruments:

TOC Analyzer
Nuclear Magnetic Residual

NAME: Annette Olsen

TITLE: Laboratory Technician

EDUCATION OR DEGREE:

B.S. in Environmental Health, 1982, Indiana University, P.A.
M.S. in Industrial Hygiene, 1984, Drexel University

PREVIOUS EXPERIENCE IN FIELD:

Miss Olsen previously was employed with Sun Information Systems where she was responsible for Material Safety Data Sheets.

EXPERIENCE AT S-R:

Miss Olsen has previously been involved in the analysis of:

- Oil and Grease
- TOC
- Petroleum Hydrocarbons

CURRENT RESPONSIBILITIES:

Miss Olsen is currently responsible for the following analyses:

- SDW Pesticides
- Priority Pollutants
- Standards

SYN 001 1907

NAME: Patricia Twaddell

TITLE: Technician

EDUCATION OR DEGREE:

B.S. in Biology, 1984, Lynchburg College

PREVIOUS EXPERIENCE IN FIELD:

EXPERIENCE AT S-R:

CURRENT RESPONSIBILITIES:

Miss Twaddell is responsible for Oil and Grease analysis and also the preparatory work for:

- . Pesticides
- . Herbicides
- . Acid Extractables
- . PCBs
- . Base/Neutral Extractable
- . Volatiles

RESUME

Joseph P. McLaughlin
Inorganic Section Supervisor

EDUCATION

B.S. Entomology/Applied Ecology, 1979 University of Delaware
M.S. Environmental Science, 1982 Drexel University

EXPERIENCE

Mr. McLaughlin is responsible for all the activities of the inorganic section, including wet chemistry techniques, compatability evaluation of solid waste, atomic absorption and ICP techniques. His duties are listed below:

- . Scheduling of all client work
- . Maintaining S-R's QC/QA policy
- . Supervising three (3) chemists, five (5) technicians and three (3) assistants
- . Training of new personnel
- . Preparing test reports
- . Introducing new analytical methods into the laboratory

In addition to analytical techniques, Mr. McLaughlin is familiar with sampling methods and the State and Federal Regulations concerning potable, wastewater and solid waste/soil samples.

Prior to joining Stablex-Reutter, Inc., he held the position of Laboratory Manager with Brandt Associates Inc., Newark, DE. where he was responsible for overall technical supervision and quality control of the microbiological and inorganic chemistry sections specializing in water quality analyses. With laboratories in two locations, Mr. McLaughlin acted as the technical liason/coordination between them. His duties also included all customer service functions and technical services initiated at his location.

PROFESSIONAL AFFILIATIONS

American Society for Microbiology
American Water Works Association

SYN 001 1909

RESUME

Marian Murphy
Chemist

EDUCATION

B.S. in Chemistry, West Virginia State College Institute, WV.

PREVIOUS EXPERIENCE

Mrs. Murphy has seven years experience at Publicker Industries in quality control testing of ethanol and other organic solvents, including crude ethylether for ACS, USP and NF Specifications. She has also analyzed grain and raw sugar samples for sugar and starch content as well as Kjeldahl Nitration, fiber and fats testing. she is also experienced in acid and antifreeze testing.

EXPERIENCE AT S-R

- . Supervision of three chemists conducting wet chemistry, atomic absorption, ICP Spectroscopy and Ion Chromatography.
- . She is responsible for care and maintenance of inorganic instruments and chemical and physical inventory for inorganic analysis.
- . Training laboratory personnel.
- . Introduction of new methods of analysis to the laboratory.
- . Development of laboratory safety program.
- . Operating Ion Chromatography.
- . AA Work

CURRENT RESPONSIBILITIES

Mrs. Murphy is responsible for inorganic analyses in the laboratory. Her responsibilities include:

- . Operating Ion Chromatography
- . Operating the Atomic Absorption Spectrophotometer
- . Introduction of new methods of analysis to the laboratory
- . Training laboratory personnel

SYN 001 1910

NAME: Maria Rodrigues

TITLE: Laboratory Assistant

EDUCATION OR DEGREE:

PREVIOUS EXPERIENCE IN FIELD:

None

EXPERIENCE AT S-R:

CURRENT RESPONSIBILITIES:

Maria is responsible for the cleaning and sterilization of all glassware in the laboratory.

RESUME

Michael S. Kenney
AA/ICP Spectroscopist

EDUCATION

B.S. Marine Biology, 1981 University of North Carolina
B.A. Chemistry, 1981 University of North Carolina

EXPERIENCE

Mr. Kenney is responsible for the preparation and analysis of potable water, wastewater and soil/solid waste samples for heavy metals content. He is familiar with EPA and Standard Methods for the handling, digesting and analysis of these samples, using the following technique:

- . Atomic Absorption
 - Direct Aspiration
 - Hydride Generation
 - Cold Vapor Generation
- . Inductively Coupled Argon Plasma Emission

His duties include the training and supervision of two (2) AA technicians as well as the scheduling of all heavy metals analysis. Mr. Kenney makes "first-line" Quality Assurance decisions and is responsible for bringing concerns to notice of the Inorganic Section Supervisor.

Prior to joining S-R, he was a chemist with Interstate Sanitation Commission in New York City, where he was involved with analyzing municipal and industrial effluents for compliance with NPDES permits and determining wasteload capacities of the receiving waters within the Interstate Sanitation District.

SYN 001 1912

RESUME

Luis A. deAndino
Environmental Chemist

EDUCATION

M.S. Environmental Science, 1982 Drexel University
B.S. Environmental Management, 1979 University of Puerto Rico

EXPERIENCE

Mr. deAndino is responsible for analyzing samples for heavy metals content, using the following atomic absorption/emission methods:

- . Direct Aspiration
- . Hydride Techniques
- . Cold Vapor Technique
- . Graphite Furnace

Mr. de Andino is also the On-Site Supervisor for S-R's Mobile Laboratory, recently spending eight weeks at a hazardous waste site coordinating and performing analysis. His duties included supervising two chemists and analyzing soil and charcoal tube samples for the presence of solvents, EP Toxicity (heavy metals) and Total Organic Halogens.

Mr. deAndino was employed as a temporary full-time chemist for D'Appolonia Waste Isolation Inc., at an on-site mobile laboratory. Duties included RCRA leachate preparation (for EP metals), characterization/compatibility studies of drum samples, TOX analysis of soil and aqueous samples, purge and trap, GC analysis, volatile organics and GC analysis of dosimeters and personnel safety monitors.

As a laboratory technician at the Center for Energy and Environment Research, Mr. de Andino assisted in the start-up, operation and maintenance of a Magnetic Separation Pilot Plant. This plant was "state of the art" technology for treatment of sewage, industrial and pharmaceutical wastes. Before and after filtration run samples were collected and analyzed by Mr. de Andino for a variety of wet chemical tests. He also participated in waste water effluent sampling.

SYN 001 1913

NAME: Maria Stanik

TITLE: Technician

EDUCATION OR DEGREE:

B.S. in Biology, 1983, Delaware Valley College of
Science and Agriculture

PREVIOUS EXPERIENCE IN FIELD:

EXPERIENCE AT S-R:

CURRENT RESPONSIBILITIES

Miss Stanik is responsible for the following laboratory
analyses:

Cyanide
Total Sulfur
Heat of Combustion
Ammonia
Phenols
Flashpoint
Total Nitrogen

She is also experienced in using the A.A. Spectrophotometer.

SYN 001 1914

NAME: Linda Hammond

TITLE: Laboratory Assistant

EDUCATION OR DEGREE:

Pursuing a degree (2nd year) in Biology at Drexel University,
Philadelphia, PA.

PREVIOUS EXPERIENCE IN FIELD:

None

EXPERIENCE AT S-R:

Miss Hammond has experience in the following laboratory analyses:

phenol	chloride
phosphate	reactivity
BS & W	pH
TOC	viscosity
COD	

Linda also has experience on the following equipment:

TOC Analyzer
pH Meter
UV-Vis. Spectrometer

CURRENT RESPONSIBILITIES:

Miss Hammond is responsible for all sample log-in procedures which include:

assigning sample designations
maintaining chain of custody forms
disbursal of notifications to departments
of necessary analyses for each sample

She is also responsible for purchasing in the laboratory and for cyanide on analyses.

SYN 001. 1915

NAME: William Schmidt

TITLE: Laboratory Technician

EDUCATION OR DEGREE:

Pursuing a degree (4th year) in Biology at
Drexel University, Philadelphia, PA

PREVIOUS EXPERIENCE IN FIELD:

Mr. Schmidt has had experience working as a Station
Chemist on a ^S/₂O₂ Scrubber at the Philadelphia Electric
Company and in microbiology testing on Sewage Water
for the Philadelphia Water Department.

EXPERIENCE AT S-R:

Mr. Schmidt has worked on S-R's Mobile Lab doing EP
Toxicity Extractions, GC work, and Wet Lab work.

CURRENT RESPONSIBILITIES:

Mr. Schmidt is responsible for the following analyses:

Colormetric Methods
Tycrameter Methods

He aslo operates a Spectrophotometer.

SYN 001 1916

NAME: Joseph Walker

TITLE: Laboratory Technician

EDUCATION OR DEGREE:

B.A. in Biology, 1977, Rutgers University

PREVIOUS EXPERIENCE IN FIELD:

Mr. Walker has experience in BOD's which he analyzed for the State of New Jersey.

EXPERIENCE AT S-R:

CURRENT RESPONSIBILITIES:

Mr. Walker is responsible for the following laboratory analyses:

Solids	Sulfate
BOD	pH
COD	EP
Nitrate	Chrome + 6

He is also experienced on the following equipment:

pH meter
BOD meter
Specrophotometer

SYN 001 1917

NAME: Jennifer Wong

TITLE: Laboratory Technician

EDUCATION OR DEGREE:

Pursuing a degree (3rd year) at Drexel University

PREVIOUS EXPERIENCE IN FIELD:

None

EXPERIENCE AT S-R:

CURRENT RESPONSIBILITIES:

Miss Wong is responsible for the following analyses:

Metal Digestion
Analysis for Metals
Wet Chemistry

She is also knowledgeable on the Atomic Absorption Spectrophotometer.

NAME: Nicholas Sodano

TITLE: Laboratory Technician

EDUCATION OR DEGREE:

B.S. Marine Science, 1981, Stockton State College

PREVIOUS EXPERIENCE IN FIELD:

Mr. Sodano has acquired the following experience through working for the New Jersey Department of Fish and Game:

Field Sampling
Creol Surveys
Selenity
Oxygen Disbursal of Water Sample

EXPERIENCE AT S-R:

CURRENT RESPONSIBILITIES:

Mr. Sodano is responsible for the digestion of various samples to release metals into element form for analyses.

SYN 001 1919

Stablex-Reutter Inc.

RESUME

John M. Kelly
Field Team Leader

EDUCATION

A.A.S. in Biology, Camden County Community College, New Jersey
B.A. Candidate in Biology and Business, Rutgers University, Camden, NJ

EXPERIENCE

Mr. Kelly has conducted a wide variety of field sampling and testing projects. He is experienced in the conduct of stack testing and has conducted tests for:

- | | |
|----------------------|------------------------|
| . Particulates | . Chlorinated solvents |
| . Oxides of sulfur | . Flue gases |
| . Oxides of nitrogen | . Metal dusts |

In addition, Mr. Kelly is experienced in all phases of industrial hygiene sampling and analysis.

He has taken the lead in the development of safety protocols for the S-R Hazardous Waste Field Sampling Team. Mr. Kelly leads this team and conducts hazardous waste sampling, compatibility testing and compositing.

He has performed many phases of wet chemistry analysis including atomic absorption and gas chromatography.

He has designed sampling equipment to suit the application for the retrieval of representative samples.

SECTION B

ANALYTICAL METHODS

S-R ANALYTICAL, INC.

SECTION B

ANALYTICAL METHODS

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C. Supplement No. 1

- Instrument and Maintenance Schedule
- Surrogate Recovery Control Limits under the EPA/NJDEP Program
- Sub-contractor review
- Extraction Technique for Volatile Organics in Air collected on charcoal
- Compatibility Testing

TITLE: Total Cyanide (Ligand Displacement Method)

REFERENCE: "Analytical Chemistry", Vol. 53, pp. 2254-2258 (1981).

SCOPE AND APPLICATION

This method is used to determine the concentration of cyanide in aqueous and soil/solid waste matrices. The method will detect inorganic cyanides that are present either as simple salts or complex radicals.

SUMMARY OF METHOD

A 1/2 hour reflux distillation is used at a pH of 4.5 in the presence of sequestering agents and lead (II). These agents displace cyanide from the stable CN-complexes. The pH and the presence of lead alienate interferences normally encountered in cyanide analysis. The liberated CN-gas is trapped in a scrubber solution of 1.25 N NaOH. This solution is analyzed by either specific ion electrode or automated colorimetric methods.

SAMPLE HANDLING AND PRESERVATION

Soil and solid waste samples are collected in a brown glass container and maintained at 4°C until analysis. Aqueous samples are collected in brown glass containers and preserved with 10N NaOH sufficient to raise the pH of the sample greater than 10. The containers are maintained at 4°C until analysis. The holding time for aqueous samples is 24 hours. For soil samples the holding time is 14 days.

APPARATUS

- Heating mantle or burner
- 1 liter, two-neck distillation flask equipped with clear seal joints
- inlet tube
- straight condenser with side arm
- absorber tube
- gas fritted bubbler
- low vacuum source
- Auto analyzer II equipped with a 578 nm filter for the colorimeter and cyanide manifold
- specific ion meter equipped with reference electrode
- cyanide specific ion electrode

Reagents

- 1,000 ppm Cyanide Stock Solution - Dissolve approximately 2 grams KOH and 2.51 grams KCN in liter distilled, deionized water. Standardize 25 ml of this solution against standard AgNO_3 titrant to the first change in color from canary yellow to a salmon hue. (Use a 20% solution of paradimethylaminobenzalrhodanine in acetone as the indicator.) Check titer weekly.
- Standard Cyanide Solutions - Dilute the stock solution to make up 0.1, 1.0, 5.0, 10, 100 ppm standards. Prepare the standards daily, using 0.1N NaOH.
- Scrubber Solution - 50 milliliters of 1.25 N NaOH
- Lead Acetate Solution (0.24M): Dilute 91.04 g lead acetate to liter with deionized water.
- Tiron Solution (0.64M): Dilute 201 g Tiron to 1 liter with deionized water.
- Tetraethylenepentaamine (TEP) Solution (1.32M): Adjust the pH of 250 g TEP to 4.5 with conc. HCl and dilute to 1 liter.
- Acetate Buffer (11.7N) with pH 4.5
- Chloramine T Solution: Dissolve 1.0 g powder in 100 ml deionized water. Prepare weekly and store in refrigerator.
- Pyridine-Barbituric Acid Reagent: Place 15 g barbituric acid in a 250 ml volumetric flask. Add just enough water to wash the sides of the flask and wet the barbituric acid. Add 15 ml con. HCl, mix and cool to room temperature. Dilute to 250 ml with deionized water.
- Sodium Dihydrogen Phosphate (1M): Dissolve 138 g $\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$ in 1 liter deionized water. Refrigerate.
- Acetic Acid Solution: Add 672 ml glacial acetic acid to 500 ml deionized water and adjust the pH of the solution to 4.5 with 10N NaOH. Dilute to 1 liter.

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PROCEDURE

A. REFLUX DISTILLATION

1. Add a representative aliquot (500 ml if aqueous, 10 g if soil/solid waste) of sample to the distillation flask. If the sample aliquot is less than 500 ml, add deionized water to dilute to 500 mls.
2. Add 50 mls of 1.25 N NaOH to gas scrubber.
3. Connect the apparatus and turn on the vacuum source. Adjust the flow rate such that the air flow through the inlet tube is at least 3 bubbles per sec.
4. Add the following to the distillation flask:
 - a. 10 ml of the lead acetate solution
 - b. 10 ml of the TIRON solution
 - c. 5 ml of the TEP solution
 - d. 10 ml of the acetate buffer
5. Between additions, rinse the air inlet tube with deionized water and allow some time for the air flow to mix the flask contents.
6. Add boiling chips to the flask.
7. Heat the solution to boiling - it may be necessary to readjust the air flow rate during heating in order to prevent the solution from backing up into and overflowing from the air inlet tube. (If this occurs anyway, the distillation should be stopped and a new aliquot of sample taken.)
8. After refluxing for 30 minutes, turn off the heat but continue the aeration for 15 minutes.
9. Transfer the scrubber solution quantitatively to a 100.0 ml volumetric flask and dilute to volume with deionized water. Transfer to a plastic, capped container to await analysis.
10. Check scrubber solution for presence of sulfide using lead acetate paper.
11. For each set of distillations, distill deionized water along with the reagents. Distill a duplicate every 10 samples. Spike every 10th sample and distill.

B. QUANTITATIVE ANALYSIS

Specific Ion Electrode Method

1. Standardize the electrodes according to the manufacturer's manual.
2. Develop a calibration curve using 5 standards and a blank.
3. Place the cyanide electrode and reference electrode into each of the sample scrubber solution, allowing 30-60 seconds for the probes to equilibrate.
4. Record the millivolts.
5. Rinse or wipe the electrodes in between solutions.
6. Chart millivolts vs. concentration units and calculate the parts per million of cyanide in each sample solution according to the graph.
7. Calculate final units by multiplying the concentration of the scrubber solution by the factor entailed in the original sample size.

Automated Colorimetric Method

NOTE: This method also contains a distillation step so the aqueous samples may be distilled and analyzed by this method. Soil/solid waste samples must at least be distilled by the reflux method.

1. Prepare the auto analyzer for analysis according to the manufacturer's instructions.
2. Place the standards in the sampling cups, filling every other cup with deionized water only.
3. After the standards have been analyzed, determine qualitatively if they are linear. if so, proceed with the sample analysis.
4. Fill alternate cups with aqueous samples or if the samples are soil/solid waste, with the scrubber solutions resulting from the reflux method.
5. Re-analyze a standard (or blank) after every tenth sample.
6. Chart the recorder response vs. known concentration (ppm) for the calibration curve.
7. Using the response obtained for each sample, calculate the CN-concentration from the graph.
8. If required, make the appropriate calculations to account for the original sample size, if the sample was refluxed.

PRECISION AND ACCURACY

S-R has generated data internally that supports the following precision and accuracy statements for cyanide:

<u>Matrix</u>	<u>PRECISION</u>	<u>ACCURACY</u>	
	<u>% RSD</u>	<u>% P</u>	<u>%P + 2S</u>
aqueous	13	90	61 - 119
soil/solid	16	89	53 - 125

TITLE: Total Phenol

REFERENCE: Standard Methods for the Examination of Water and Wastewater, 14th edition, EPA Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Technical Auto Analyzer Methods Manual

SCOPE AND APPLICATION

This method is applicable to aqueous soil/solid waste matrices at levels of 1 ug/l to 500 ug/l.

SUMMARY OF METHOD

The manual method entails a distillation and chloroform extraction of the sample and colorimetric determination at 460 nm. The automated method is based on the distillation of phenol and subsequent reaction of the distillate with alkaline ferricyanide and 4-aminoantipyrine to form a red complex which is measured at 505 or 520 nm.

SAMPLE HANDLING AND PRESERVATION

The preservatives and holding times for aqueous samples are dependent on who will ultimately be using the analytical data:

- . For aqueous and soil/solid waste matrices EPA recommends acidifying the sample with sulfuric acid with a holding time of 28 days.
- . For potable and wastewater, NJDEP requires preservation by copper sulfate/phosphoric acid and a holding time of 24 hours.

No preservatives are required for soil/solid waste matrices. All samples should be maintained at 4°C until the time of analysis.

APPARATUS

- Technicon Auto Analyzer II including:
 - . Phenol Manifold
 - . proportioning pump
 - . heating bath with distillation coil
 - . distillation head
 - . colorimeter with 50 mm flow cell and 505 or 520 nm filter
 - . recorder
 - . disposable sample cups

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APPARATUS (CONT'D)

- Distillation apparatus: 250 ml flask and condenser (Graham).
- Separatory funnel, 250 ml
- Spectrophotometer

REAGENTS

A. Manual

- pH Sticks: to cover 4.0 pH
- Copper Sulfate Solution: Dissolve 100g $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ in distilled water and dilute to 1 liter.
- Phosphoric Acid (1 + 9)
- Conc. Ammonium Hydroxide
- Ammonium Chloride Solution: Dissolve 50g of NH_4Cl in distilled, deionized water and dilute to 1 liter.
- Aminoantipyrine Solution: Dissolve 2.0g of 4-aminoantipyrine in water and dilute to 100ml. Prepare a fresh solution daily.
- Potassium Ferricyanide Solution: Dissolve 8.0g $\text{K}_3\text{Fe}(\text{CN})_6$ in distilled water and dilute to 100ml. Prepare fresh weekly.
- Chloroform
- Sodium Sulfate, Anhydrous

REAGENTS (CONT'D)

B. Automated

- Distillation reagent: add 100 ml of conc. phosphoric acid (85% H_3PO_4) to 800 ml of deionized water, cool and dilute to 1 liter.
- Buffered potassium ferricyanide: Dissolve 1.0 g potassium ferricyanide, 3.1 g boric acid and 3.75 g potassium chloride in 800 ml deionized water. Adjust to pH of 10.3 with 1N solution hydroxide and dilute to 1 liter. Add 0.5 ml of Brij-35. Prepare fresh weekly.
- Sodium hydroxide (1N): Dissolve 40 g NaOH in 500 ml deionized water, cool and dilute to 1 liter.
- 4-Aminoantipyrine: Dissolve 1.0 g of 4-aminoantipyrine in 800 ml of deionized water and dilute to 1 liter. Prepare fresh daily.
- Ferrous ammonium sulfate: Dissolve 1.1 g ferrous ammonium sulfate in 500 ml deionized water containing 1 ml H_2SO_4 and dilute to 1 liter with freshly boiled and cooled deionized water.
- Stock phenol solution: Dissolve 1.0 g phenol in 500 ml of deionized water and dilute to 1 liter. Add 1 g CuSO_4 and 0.5 ml conc. H_3PO_4 as preservative. (1 ml = 1.0 mg phenol.)
- Standard phenol solution: Dilute 10.0 ml of stock phenol solution to 1 liter. (1.0 ml = 0.01 mg phenol.)
- Standard phenol solution B: Dilute 100.0 ml of standard phenol solution A to 1 liter. (1.0 ml = 0.001 mg phenol.)
- Daily Standards: Dilute 1.0, 2.0, 5.0, 10.0 and 20.0 mls of standard phenol solution B in 100.0 ml volumetrics using deionized water. Add 0.1 g CuSO_4 and 2 drops of conc. H_3PO_4 to each flask prior to diluting to 100.0 mls.

PROCEDURE

A. Manual

1. If the sample is aqueous and preserved, transfer 100 ml into the distillation flask. If the sample is soil/solid waste, measure 10 g into a beaker and add 500 ml water. Adjust the pH to 4.0 with 1+9 H_3PO_4 and 5 ml CuSO_4 solution. Then transfer the mixture to the distillation flask. Use a 100 ml graduate cylinder as the receiving vessel.
2. Distill approx. 75 ml of the sample then stop the distillation. When the boiling stops, add 25 ml DI water.
3. Transfer the distillate, after cooling, to a 250 ml beaker.
4. Prepare a 100 ml distilled blank and a series of 100 ml phenol standards: 5, 10, 20, 30, 40 and 50 ug phenol.
5. Treat sample, blank and standard as follows: Add 25 ml NH_4Cl solution and adjust to $\text{pH } 10.0 \pm 0.2$ using conc. NH_4OH .
6. Transfer to a 250 ml separatory funnel, add 1.0 ml aminoantipyrine solution, mix well, add 1.0 ml potassium ferricyanide solution, mix well and allow the color to develop for 3 minutes. The solution should be clear and light yellow.
7. Extract immediately with CHCl_3 , using 10 ml. Shake the sep funnel at least 10 times. Let the CHCl_3 settle, shake again 10 times and again let it settle.
8. Filter the chloroform extracts through filter paper containing a 5 g layer of anhydrous Na_2SO_4 . Collect the dried extracts in clean cells.
9. Read the absorbance of the standards, blank and samples at 460 nm. Plot absorbance versus ug phenol for the calibration curve and calculate sample concentrations from the curve.

. third

PROCEDURE

B. Automated

1. Set up the manifold according to the manufacturer's manual.
2. Allow colorimeter and recorder to warm up for 30 minutes. Generate a baseline with all reagents, feeding deionized water through the sample line. Use polyethylene tubing for the sample line.
3. Place phenol standards in samplers in order of decreasing concentration, filling alternate sample cups with deionized water in order to guarantee a return to baseline.
4. Load the remaining sample cups with aqueous samples or aqueous phases of soil/solid waste samples. Instead of every fifth sample, place a standard or a blank in the sample cup. Every tenth sample analyze a duplicate and a spike of that sample.
5. Generate a standard curve by plotting peak heights of standards vs. concentration values.
6. Calculate sample values by comparing sample peak heights with standards.

PRECISION AND ACCURACY

1. S-R has generated the following internal precision and accuracy data:

<u>Matrix</u>	<u>Accuracy</u>		<u>Precision</u>
	<u>% P</u>	<u>% P + 2S</u>	<u>% RSD</u>
aqueous	99	78-120	92
soil/solid waste	86	48-124	27

SYN
001
1932

TITLE: Preparation of Aqueous Samples for General Metals Analysis

REFERENCE: EPA Methods for the Chemical Analysis of Water and Wastes, EPA-600/4-79-020

SCOPE AND APPLICATION

This method is applicable to all aqueous samples, including potable water, wastewater and leachates, to be analyzed for the following metals:

Antimony	Lead	Iron
Barium	Nickel	Magnesium
Beryllium	Silver	Manganese
Cadmium	Thallium	Potassium
Chromium	Zinc	Sodium
Copper	Calcium	

SUMMARY OF METHOD

The samples are acid digested with nitric acid and diluted to a final volume of 100 ml.

SAMPLE HANDLING AND PRESERVATION

The samples should be collected in glass or plastic containers and acidified with HNO_3 . Containers should be refrigerated at 4°C until preparation. The holding times for the samples prior to preparation, is six months.

INTERFERENCES

Organic materials present in the samples will not totally break down without the addition of other reagents such as sulfuric acid or perchloric acid. If organics are present to a substantial degree, an alternate digestion method should be used.

APPARATUS

- 250 ml glass beakers
- watchglasses
- hot plate
- 100.0 ml volumetric flasks
- graduate cylinders
- 0.45 μm filters (12.5 in diameter)
- glass or plastic funnels

REAGENTS

- Conc. Nitric Acid
- Spiking Solutions: 1000 ppm solutions of the metals being analyzed for. (The amounts to be added will depend on the expected concentration of these metals in the samples)
- Internal Reference Standard: 100.0 ppm solution of yttrium, in 3% HNO_3

PROCEDURE

1. Transfer a representative aliquot (100 ml) of sample to an acid-rinsed beaker. If concentration of the sample is required, use an aliquot of at least 200 mls.
2. Add 5 mls conc. HNO_3 and a predetermined volume of the spiking solution, if required.
3. Place the beaker on the hot plate and allow the sample to evaporate to near dryness, making sure the sample does not boil.
4. Cool the beaker and add another 5 ml acid.
5. Cover the beaker with a watchglass and return to the hot plate. Increase the heat until a gentle refluxing action occurs.
6. Continue heating, adding more acid as required, until a light-colored, wet residue is obtained. This indicates the digestion is complete.
7. Cool and add 1-2 ml conc. nitric acid and warm the beaker slightly to dissolve the residue.
8. Wash down the beaker walls and watchglass with distilled, deionized water and filter the sample to remove silicates and other insoluble materials that could clog the atomizer of the AA or ICAP instruments.
9. Dilute to a final volume of 100.0 mls.
10. NOTE: Every tenth sample digested is to be spiked for those metals believed to present and at the appropriate level. Every tenth sample is also to be digested in duplicate. A method blank should be digested every day.
11. Analyze the digestions by atomic absorption or atomic emission spectrometry using either flame AA or inductively coupled argon plasma.

TITLE: Preparation of Aqueous Samples for Arsenic and Selenium Analysis

REFERENCE: Standard Methods, for the Examination of Water and Wastewater,
14th edition

SCOPE AND APPLICATION

This method is applicable to all aqueous samples, including potable water, wastewater and leachates to be analyzed for arsenic and selenium.

SUMMARY OF METHOD

The samples are acid digested with nitric, sulfuric and hydrochloric acid and diluted to a final volume of 100.0 ml.

SAMPLE HANDLING AND PRESERVATION

The samples should be collected in glass or plastic containers and acidified with HNO_3 . Containers should be refrigerated at 4°C until preparation. The holding time for the samples prior to preparation is six months.

INTERFERENCES

Organic materials present in the sample may require the addition of perchloric acid in order to break down. If there is a significant amount of organic material, an alternate digestion technique should be used.

APPARATUS

- 250 ml glass beakers
- watchglasses
- hot plates
- 100.0 ml volumetric flasks
- graduate cylinders
- glass or plastic funnels

REAGENTS

- Conc. nitric acid
- Sulfuric acid (18N): Add 500 ml conc. H_2SO_4 to 200 ml deionized water in a 1 liter volumetric. Allow solution to cool, then dilute to one liter.
- Conc. hydrochloric acid
- Arsenic and selenium spiking solutions (1000 ppm): obtain from a chemical supply house.

PROCEDURE

1. Transfer a representative aliquot (100 ml) of sample to an acid-rinsed beaker. If concentration of the sample is required, use an aliquot of at least 200 mls.
2. Add 10 ml conc. nitric acid and 12 ml 18N sulfuric acid and place the beaker on the hot plate.
3. Evaporate the sample to SO_3 (dense white) fumes, usually at an approximate volume of 20 ml.
4. Maintain oxidizing conditions at all times by adding small amounts of nitric acid whenever the red-brown NO_2 fumes disappear.
5. Cool and add 40 ml conc. hydrochloric acid and rinse the beaker walls with distilled, deionized water.
6. Bring to a final volume of 100.0 ml.
7. NOTE: Every tenth sample digested is to be spiked for arsenic and selenium at the appropriate level. Every tenth sample is also to be digested in duplicate. A method blank should be digested every day.
8. Analyze the digestions by atomic absorption spectrometry using the hydride generation technique.

TITLE: Preparation of Aqueous Samples for Mercury Analysis

REFERENCE: EPA Methods for Chemical Analysis of Water and Wastes, EPA-600/
4-79-020

SCOPE AND APPLICATION

This method is applicable to all aqueous samples, including potable water, wastewater and leachates to be analyzed for mercury.

SUMMARY OF METHOD

The samples are digested with acids and oxidizing reagents at 90°C for two hours. After cooling, reducing agents are added to the samples, first to convert all mercury present to elemental Hg, then to mercury vapor.

SAMPLE HANDLING AND PRESERVATION

The samples may be collected in glass or plastic containers and acidified with nitric acid. Prior to digestion, the samples should be kept at 4°C. If collected in glass, the holding time is 38 days. If collected in plastic, the holding time is 13 days. Once digested, analysis of the digestions must occur within 24 hours.

INTERFERENCES

Chloride and sulfide interferences are eliminated by the addition of excess potassium permanganate.

APPARATUS

- 250 ml glass beakers
- graduate cylinder
- 100.0 ml volumetric flasks
- glass or plastic funnels
- 100 ml plastic container with lids

REAGENTS

- Conc. Sulfuric Acid (reagent grade)
- Sulfuric Acid (0.5N): Dilute 14 ml conc. H_2SO_4 to one liter with deionized, distilled water.
- Conc. Nitric Acid (reagent grade)
- Sodium Chloride-Hydroxylamine Hydrochloride Solution: Dissolve 12 g of sodium chloride in distilled, deionized water and dilute to 100 ml.
- Potassium Permanganate Solution (5%): Dissolve 5 g KMnO_4 in 100 ml deionized, distilled water.
- Potassium Persulfate Solution: Dissolve 5 g KSO_4 in 100 ml deionized, distilled water.
- Stock Mercury Solution (1000 ppm): Obtain from a chemical supply company or make by dissolving 0.1354 g mercuric chloride in 75 ml distilled, deionized water. Add 10 ml HNO_3 and dilute to 100.0 ml. (1.0 ml = 1.0 mg)
- Standard Mercury Solution: Make successive dilutions of the stock in order to obtain a working standard of 0.1 ppm in 0.15% HNO_3 . Prepare fresh daily.

PROCEDURE

1. Transfer representative aliquots of sample (50 ml) to the Erlenmeyer flasks.
2. Add 5 ml conc. H_2SO_4 and 2.5 ml conc. HNO_3 , mixing after each addition.
3. Add 15 ml KMnO_4 solution to each flask. If interferences are present, excess permanganate solution may be required - the samples must remain purple throughout the digestion.
4. Add 8 ml of potassium persulfate solution.
5. Heat the flasks on a hot plate for two hours at 90°C .

PROCEDURE (CONT'D)

6. Cool and add 6 ml of sodium chloride-hydroxylamine hydrochloride to reduce the excess permanganate (more may be required), and dilute to 100.0 ml in a volumetric flask.
7. Transfer the digestions to plastic containers and seal.
8. NOTE: Digest several 0.1 ppm standards in the same manner as above and digest one blank for each hot plate used. Every tenth sample should be digested in duplicate and also spiked and digested.
9. Analyze the digestions by atomic absorption spectrometry, by the flameless, cold-vapor technique.

SYN 001 1939

TITLE: Preparation of Soil/Solid Waste Samples for Metals Analysis

REFERENCE: EPA Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, July, 1982 revision

SCOPE AND APPLICATION

This method is an acid digestion procedure used to prepare soil/solid waste matrices to be analyzed for the following metals:

Antimony	Copper	Zinc
Arsenic	Lead	Calcium
Barium	Nickel	Iron
Beryllium	Selenium	Magnesium
Cadmium	Silver	Manganese
Chromium	Thallium	Potassium
		Sodium

SUMMARY OF METHOD

The samples are digested with nitric acid and hydrogen peroxide and refluxed with nitric acid.

SAMPLE HANDLING AND PRESERVATION

The samples should be collected in glass or plastic containers and kept at 4°C until digestion.

INTERFERENCES

Some matrices will not be amenable to this method of digestion so an alternate one should be used.

APPARATUS

- 125 ml conical Phillips beakers
- watchglasses
- drying oven
- thermometer, 0°-200°C
- No. 42 filter paper

REAGENTS

- conc. nitric acid
- nitric acid (1+1)
- 30% hydrogen peroxide
- Deionized, distilled water

PROCEDURE

1. Weigh out a 1.0 g aliquot of sample and place in flask.
2. Add 10 ml 1+1 HNO_3 . Swirl flask to mix contents and cover with a watchglass.
3. Heat the sample at 95°C and reflux for 10 min.
4. Allow the sample to cool, add 5 ml of conc. HNO_3 , replace the watchglass and reflux for 30 min. DO NOT ALLOW THE SAMPLE TO GO DRY.
5. After the evaporation, cool the sample and add 2 ml water and 3 ml of 30% H_2O_2 . Return the beaker to the hot plate for warming. Heat until the reaction subsides, then cool the beaker.
6. Continue to add 30% H_2O_2 in 1 ml aliquots with warming until the reaction is minimal or until the appearance of the sample is unchanged. DO NOT ADD MORE THAN 10 ml H_2O_2 .
7. Cool the sample and add 5 ml of 1+1 nitric acid and 10 ml of water, return the covered beaker to the hot plate and heat for another 10 min.
8. After cooling, filter the digested sample through No. 42 filter paper into a 100 ml volumetric flask. Dilute to 100.0 ml with water.
9. Note: Every tenth sample digested is to be spiked for those metals being analyzed and at the appropriate level. Every tenth sample is also to be digested in duplicate. A method blank should be digested every day.
10. Analyze the digestions by atomic absorption or atomic emission spectrometry using either flame AA or inductively coupled argon plasma.

TITLE: Preparation of Soil/Solid Waste Samples for Mercury Analysis

REFERENCE: EPA Methods for Chemical Analysis of water and wastes, EPA-600/
4-79-020

SCOPE AND APPLICATION

This method is applicable to soil/solid waste samples to be analyzed for Mercury.

SUMMARY OF METHOD

The samples are digested with acids and oxidizing reagents at 90°C for thirty minutes. After cooling, reducing agents are added to the samples, first to convert all mercury present to elemental mercury and then to mercury vapor.

SAMPLE HANDLING AND PRESERVATION

The samples may be collected in glass or plastic and should be kept at 4°C prior to digestion. Once digested, analysis must occur within 24 hours.

INTERFERENCES

Interferences from chloride, sulfide and organic compounds are eliminated by the addition of excess potassium permanganate.

APPARATUS

- 250 ml Erlenmeyer flasks
- graduate cylinder
- 100.0 ml volumetric flasks
- plastic or glass funnels
- 100 ml plastic containers with lids

SYN 001 1942

TITLE: Analysis for General Metals Analysis by Direct Aspiration
Atomic Absorption/ Emission Spectrometry

REFERENCE: Standard Methods for the Examination of Water and Wastewater, 15th edition, EPA Test Methods for the Analysis of Solid Waste, Physical/ Chemical Methods, SW846, July, 1982 revision, EPA Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020

SCOPE AND APPLICATION

This method is a general atomic absorption procedure applicable to the determination of the following metals in aqueous and soil/solid waste matrices:

Barium	Copper	Nickel
Beryllium	Iron	Potassium
Cadmium	Lead	Silver
Calcium	Manganese	Sodium
Chromium	Magnesium	Thallium
		Zinc

SUMMARY OF METHOD

Prior to application of this method, samples must have been digested by a method specific to the sample matrix. A representative aliquot of this digestion is aspirated into an air/acetylene flame. The resulting change in absorption of hollow cathode radiation will be proportional to the metal concentration.

INTERFERENCE

Nonspecific absorption and light scattering can be significant at the analytical wavelength. Background correction is required. Samples and standards should be monitored for viscosity differences that may alter the aspiration rate. Some metals will require special precautions to be taken in order to suppress ionization or chemical interferences:

<u>Metal</u>	<u>Interferences</u>	<u>Corrective Measure</u>
AL	Ionization; depressed by Si	2000 ppm K, standard addition
Ba	Ionization	2000 ppm K
Ca	Ionization	2000 ppm K
	Si, Al, PO ₄ & SO ₄	La or Sr, 0.1 to 1% W/V
Cr	High Co, Fe & Ni	2% W/V NH ₄ Cl
Cu	High Zn/Cu ratio, depression	N ₂ O Flame
Fe	Si	2000 ppm Ca
K	Ionization	1000 ppm Cs
Mg	Al & Si, depression	Sr or La, 1000 ppm
Mn	Si	200 ppm Ca
Na	Ionization	1000 ppm K
Pb	PO ₄ , CO ₃ , I, F when 10 x Pb	0.1 M EDTA
Sr	Ionization	2000 ppm K
	Si, Al, Ti, PO ₄ & SO ₄	1 % La

SYN 001 1943

REAGENTS

- Aqua regia: Prepare immediately before use by carefully adding three volumes of conc. hydrochloric acid to one volume of conc. nitric acid.
- Sulfuric acid (0.5N): Dilute 14 ml of conc. sulfuric acid to one liter.
- Sodium chloride - hydroxylamine hydrochloride solution: Dissolve 12 g of sodium chloride and 12 g of hydroxylamine hydrochloride in deionized, distilled water and dilute to 100 ml.
- Potassium permanganate solution (5%): Dissolve 5 g of KMnO_4 in 100 ml distilled, deionized water.
- Stock mercury solution (1000 ppm): Obtain from a chemical supply company or make by dissolving 0.1354 g mercuric chloride in 75 ml distilled, deionized water. Add 10 ml HNO_3 and dilute to 100.0 ml (1.0 ml = 1.0 mg).
- Standard mercury solution: Make successive dilutions of the stock in order to obtain a working standard of 0.1 ppm in 0.15% HNO_3 . Prepare fresh daily.

PROCEDURE

1. Weigh 1.0 g portions of sample and place in Erlenmeyer flasks.
2. Add 5 ml deionized, distilled water and 5 ml aqua regia.
3. Heat for two minutes on a 90°C hotplate.
4. Cool and add 50 ml deionized, distilled water and 15 ml potassium permanganate solution to each sample flask.
5. Mix thoroughly and heat for 30 minutes at 90°C.
6. Cool and add 6 ml of sodium chloride-hydroxylamine hydrochloride to reduce excess permanganate (more may be required).
7. Dilute sample to 100.0 ml in a volumetric flask using deionized, distilled water.
8. NOTE: Digest several 0.1 ppm standards in the same manner as above and digest one blank for each hotplate used. Every tenth sample should be digested in duplicate and also spiked and digested.
9. Analyze the digestions by atomic absorption spectrometry, using the flameless, cold-vapor technique.

APPARATUS

- Atomic Absorption Spectrophotometer: single or dual channel, double beam instrument, with a grating monochromator, photomultiplier detector, adjustable slits and background correction.
- Hollow cathode lamp: specific to the metals
- Burner heads: specific to the flame being used (air/acetylene or nitrous oxide/acetylene)

REAGENTS

- ASTM Type II Water
- Conc. Nitric Acid: preferably analyzed for trace elements
- Stock solution (1000 mg/l): Obtained from either Spex Industries, Fisher Scientific or Alpha Products. Standards may also be made in-house by referring to EPA Analysis of Solid Waste - Physical/Chemical Methods, SW846, July, 1982, Revision. All stock solutions should be checked periodically for strength and should be discarded prior to the manufacturer's expiration date. ALIQUOTS SHOULD NEVER BE PIPETTED DIRECTLY FROM THE BOTTLE.
- Working Standards: Prepare dilutions of the stock solutions in 100.0 ml volumetrics to be used as calibration standards. All standards should be prepared in 3% HNO₃ with any solutions to correct interferences being added prior to dilution to 100.0 mls. The standards should be made in accordance with the linear range of that element:

METALCALIBRATION STANDARDS, ppm

Barium	1.0, 2.0, 2.5, 3.0, 4.0
Beryllium	0.100, 0.125, 0.150, 0.175, 0.200
Cadmium	0.25, 0.50, 0.75, 1.00, 1.50
Chromium	0.50, 1.0, 1.5, 2.0, 3.0
Copper	0.50, 0.70, 1.0, 1.5, 2.0
Iron	0.50, 1.0, 1.5, 2.0, 3.0
Lead	1.0, 2.0, 4.0, 7.0, 10
Manganese	0.2, 0.5, 1.0, 1.5, 2.0
Nickel	0.5, 1.0, 1.5, 2.0, 3.0
Silver	0.3, 0.7, 1.0, 1.5, 2.0
Thallium	1.0, 2.5, 5.0, 10, 15
Zinc	0.10, 0.25, 0.50, 0.75, 1.0
Calcium	0.5, 1.0, 1.5, 2.0, 2.5
Magnesium	0.1, 0.15, 0.2, 0.3, 0.4
Sodium	0.1, 0.2, 0.3, 0.4, 0.5
Potassium	0.25, 0.50, 0.75, 1.0, 1.5

NOTE: CALIBRATION STANDARDS AND A BLANK SHOULD BE PREPARED DAILY.

REAGENTS (CONT'D)

- Air: Cleaned and dried through a suitable filter to remove oil, water and other foreign substances. The desiccant in the compressor should be checked twice weekly for saturation.
- Acetylene: High purity grade. Cylinders must be replaced before the tank pressure reaches 50 psig to prevent acetone from damaging the instrument tubing.
- Nitrous Oxide

PROCEDURE

1. Set up the instrument in accordance with the manufacturer's methods manual, using the following wavelengths and flame conditions:

<u>ELEMENT</u>	<u>METHOD DETECTION</u>		<u>FLAME CONDITIONS</u>
	<u>LIMIT, ug/l</u>	<u>WAVELENGTH, nm</u>	
Barium	0.087	553.6	nitrous oxide/acetylene - fuel
Beryllium	0.010	234.9	nitrous oxide/acetylene - fuel
Cadmium	0.004	228.8	air/acetylene - oxidizing
Chromium	0.026	357.9	nitrous oxide/acetylene - fuel
Copper	0.050	324.7	air/acetylene - oxidizing
Iron	0.050	248.3	air/acetylene - oxidizing
Lead	0.043	283.3	air/acetylene - oxidizing
Manganese	0.020	279.5	air/acetylene - oxidizing
Nickel	0.036	232.0	air/acetylene - oxidizing
Silver	0.009	328.1	air/acetylene - oxidizing
Thallium	0.10	276.8	air/acetylene - oxidizing
Zinc	0.010	213.9	air/acetylene - oxidizing
Calcium	0.050	422.7	air/acetylene - oxidizing
Magnesium	0.010	285.2	air/acetylene - oxidizing
Potassium	0.050	766.5	air/acetylene - oxidizing
Sodium	0.010	589.0	air/acetylene - oxidizing

2. Construct a calibration curve by aspirating each standard plus a blank and plotting the concentrations of standards against the absorbances. Allow the baseline to return to zero after each standard is aspirated.
3. Aspirate each sample individually, using an appropriate integration time as determined by the manufacturer's recommendations and the sensitivity of the element. Generally, longer integration times are required for "jumpy" elements.

Envirosphere Company
March 25, 1985
Page 2

Equipment/Instrument

Gas Chromatographs

Gas Chromatography/Mass Spectrometry

Maintenance Schedule

In addition to the guidelines described on pages 29 through 31 of the OA/OC Section of the S-R SOP, the following is performed: All gas flows are checked on alternate days. Once each month, injector ports, screens and fans are cleaned. Each detector is also dismantled and cleaned (the ECD detector is "baked out" prior to cleaning with Freon).

The following maintenance is performed:

Monthly-The disk drive prefilter is cleaned, the source is cleaned and the filament is changed.

Every 3 months-The furnace filter and the forepump oil is changed.

Every 6 months-The disk drive absolute filter and the turbo pump oil is changed.

All maintenance is performed on a Friday, with each instrument assigned to a specific one, for example:

Model 1020-First Friday of each month

Model 5100A-Second Friday

Model 5100B-Third Friday

Model 5100C-Fourth Friday

Should you require additional information, please don't hesitate to call.

Sincerely,

S-R ANALYTICAL, INC.

Catherine M. Ward

Catherine M. Ward
Project Manager

CMW/aso

SYN 001 1947

MEMORANDUM

TO: Dr. Thomas Stevenson
RE: Spare Parts Inventory
DATE: March 26, 1985
PREPARED BY: Catherine M. Ward

S-R has little need for spare parts storage and instead relies on redundancy of instrumentation to accommodate breakdowns in the following sections:

Gas Chromatographs

There are currently six (6) gas chromatographs in use, with another scheduled to arrive within four (4) weeks. All the instruments are made by one (1) of only two (2) manufacturers so the detectors and the majority of parts are interchangeable. In addition, the instruments are covered under a service contract with TR Associates, Lewisburg, PA, (717) 523-6002, who will respond within 48 hours in an "emergency" situation.

Gas Chromatographs/Mass Spectrometers

There are currently three (3) Finnegan mass spectrometers at our Cherry Hill facility. Another Finnegan is expected to arrive in mid-April. The instruments are protected from electrical variations, brown outs and blackouts (for 20 minutes) by an Uninterrupted Power Supply (UPS) system which minimizes downtime significantly. Because the instruments are obtained from a single manufacturer, parts may be exchanged between instruments if necessary but S-R has been able to almost exclusively rely on the service contract with Finnegan. Service and parts arrive within 48 hours.

Atomic Absorption/Emission Spectrophotometers

S-R has three (3) atomic absorption units (AA's) and one (1) inductively coupled argon plasma (ICP) unit. The AA's are serviced by the manufacturer which is located within a two hour drive and the ICP is serviced under a contract with its manufacturer, Applied Research Laboratory (ARL).

SURROGATE RECOVERY CONTROL LIMITS

The following limits appeared in the NJDEP WA83A198 document:

QUALITY CONTROL LIMITS FOR PERCENT RECOVERY

<u>Fraction</u>	<u>Surrogate</u>	<u>Water</u>	<u>Soil</u>
Volatile	toluene-d ₈	86-119	69-127
Volatile	4-bromofluorobenzene (HFB)	85-121	61-122
Volatile	1,2-Dichloroethane-d ₄	77-120	64-129
Base/Neutral	nitrobenzene-d ₅	41-120	24-115
Base/Neutral	2-fluorobiphenyl	44-119	37-120
Base/Neutral	terphenyl-d ₁₄	33-128	28-133
Acid	phenol-d ₅	15-96	20-106
Acid	2-fluorophenol	23-107	24-111
Acid	2,4,6-tribromophenol	20-105	11-102

SURROGATE ADVISORY LIMITS FOR PERCENT RECOVERY BASED ON SINGLE LABORATORY DATA

<u>Fraction</u>	<u>Surrogate</u>	<u>Water*</u>	<u>Soil*</u>
Pesticide	dibutylchlorodate	67-114	0-205
Dioxin	1,2,3,4-TCDD	23-148	18-128

The following limits appear in the EPA-CLP document (IFHWAR5-J176, J177, J178), January, 1985 revision:

CONTRACT REQUIRED SURROGATE SPIKE RECOVERY LIMITS

<u>Fraction</u>	<u>Surrogate Compound</u>	<u>Low/Medium Water</u>	<u>Low/Medium Soil/Sediment</u>
VOA	toluene-d ₈	86-119	50-160
VOA	4-bromofluorobenzene	85-121	50-160
VOA	1,2-dichloroethane-d ₄	77-120	50-160
HNA	nitrobenzene-d ₅	41-120	20-140
HNA	2-fluorobiphenyl	44-119	20-140
HNA	p-terphenyl-d ₁₄	33-128	20-140
HNA	phenol-d ₅	15-103	20-140
HNA	2-fluorophenol	23-121	20-140
HNA	2,4,6-tribromophenol	10-130	10-140
Pesticide	dibutylchlorodate	(48-136*)	(20-150)*

SYN 001 1949

*Advisory limits only.

RECOVERIES OBTAINED BY SPIKING TENAX TUBES:

BENZENE

<u>Test Report</u>	<u>Volume of Spike Added, ug</u>	<u>% Recovery</u>
10074	2	96
10074	2	75
10074	6	97
9722	2	96
9748	2	93
9748	6	97

TOLUENE

<u>Test Report</u>	<u>Volume of Spike Added, ug</u>	<u>% Recovery</u>
10074	2	110
10074	2	72
10074	6	95
9722	2	94
9748	2	95
9748	6	95

XYLENES

<u>Test Report</u>	<u>Volume of Spike Added, ug</u>	<u>% Recovery</u>
10074	2	114
10074	2	99
10074	6	113
9722	2	106
9748	2	130
9748	6	113

SIN 001 1950

RECOVERIES OBTAINED BY SPIKING TENAX TUBES (CONT'D)

Test Report No. SR10514

<u>Constituent</u>	<u>Amount of Spike, μg</u>	<u>% Recovery</u>
Methylene chloride	1.0	145
1,1-Dichloroethylene	1.0	134
1,1-Dichloroethane	1.0	121
trans-1,2-Dichloroethylene	1.0	110
Chloroform	1.0	117
1,2-Dichloroethane	1.0	112
1,1,1-Trichloroethane	1.0	110
Carbon tetrachloride	1.0	118
Bromodichloromethane	1.0	107
1,2-Dichloropropane	1.0	115
trans-1,3-Dichloropropene	1.0	109
Trichloroethylene	1.0	118
Dibromochloromethane	1.0	118
Benzene	1.0	105
1,1,2-Trichloroethane	1.0	122
cis-1,3-Dichloropropene	1.0	108
2-Chloroethyl vinyl ether	1.0	133
Bromoform	1.0	122
1,1,2,2-Tetrachloroethane	1.0	129
Tetrachloroethylene	1.0	118
Toluene	1.0	108
Chlorobenzene	1.0	115
Ethyl benzene	1.0	106

ANALYTICAL INC.

PROCEDURE NO.: 110

TITLE: Sub-contractor Review

DATE: April 1, 1985

SCOPE AND APPLICATION

This procedure delineates the process whereby S-R Analytical, Inc. (S-R) determines the ability of a sub-contractor to perform according to S-R's standards.

Method

1. Once it is clear that S-R will be responsible for data generation that can not occur in-house, a sub-contractor should be located, preferably through references.
2. The following information should be obtained in an initial call with the sub-contractor:
 - . State Certifications
 - . Federal Certifications
 - . The Sub-contractor's Rotice Quality Control/Quality Assurance Program
3. Depending on the parameter to be analyzed, a site visit should be arranged. The Laboratory Manager and/or the Quality Assurance Manager from S-R should be among the people attending.
4. During the site visit, S-R personnel should obtain the following information:
 - . The size of the laboratory space dedicated to the analysis of interest
 - . The education, experience and training of the people who will be performing the analysis
 - . The general appearance of the laboratory (cleanliness, safety, etc.)
 - . The make and model of instruments to be used in the analysis
 - . The sample storage capability (refrigerated and non-refrigerated)
 - . Copies of the laboratory's analytical procedures.

Method (CONT'D)

5. Upon return to S-R, a report of the visit should be prepared.
6. Proficiency samples should then be submitted to the proposed sub-contractor. Although the lab may be told that these are known spikes, the lab should provide results in the same time period that actual sample results will be expected to be generated.
7. Only if the proficiency samples are accurately analyzed should S-R consider using the sub-contractor.

TITLE: Extraction Technique for Volatile Organics in Air
Collected on Charcoal

REFERENCE: S-R Analytical Protocol

SCOPE AND APPLICATION

This extraction technique applies to all charcoal tubes to be analyzed for volatile organics by GC or GC/MS techniques.

SAMPLE HANDLING AND PRESERVATION

After sampling, and after extraction until analysis, the charcoal tubes and extracts, respectively are kept in a freezer. EXTRACTION and ANALYSIS should be performed within fourteen (14) days. Extraction should be performed just prior to analysis.

APPARATUS AND REAGENTS

- 10 ml glass vials with screw cap lids
- 500 ml syringe
- Methanol: pesticide grade.
- tweezers

PROCEDURE

1. The tip of the glass charcoal tube is broken at the tapered end using tweezers and all charcoal to be front of the fritted disc is transferred to a glass vial containing 500 ul methanol. An aliquot of the standard containing d8-toluene and 4-bromofluorobenzene is added. This vial is labelled with the sample number and "A".
2. The contents of the glass tube after the fritted disc are quantitatively transferred to another glass vial containing methanol. The surrogate is then added to the vial, which is labelled with the sample number and "B".
3. Close the vials and shake them to mix the contents.
4. Allow the vials to sit in a freezer for no more than one hour.
5. Prior to analysis, allow the vial to come to room temperature before removing an aliquot of extract for injection into the GC/MS samples purge vessel. The "A" portion should be analyzed first unless both portions are requested to be analyzed by the client.

Note: Analysis should be performed as soon as possible after extraction.

The following data represents recoveries obtained from spiking charcoal tubes with known amounts of volatile organics:

<u>Constituent</u>	<u>Amount of Spike, ug</u>	<u>% Recovery</u>
chloromethane	1.0	110
bromomethane	1.0	122
vinyl chloride	1.0	100
chloroethane	1.0	103
methylene chloride	1.0	95
ethene, 1,1-dichloro	1.0	104
ethane, 1,1-dichloro	1.0	108
1,2-trans-dichloroethene	1.0	112
chloroform	1.0	104
ethane, 1,2-dichloro-	1.0	76
ethane, 1,1,1-trichloro-	1.0	110
carbon tetrachloride	1.0	89
bromodichloromethane	1.0	109
propane, 1,2-dichloro-	1.0	102
1,3-trans-dichloropropene	1.0	93
trichloroethylene	1.0	97
chlorodibromomethane	1.0	94
benzene	1.0	97
ethane, 1,1,2-trichloro-	1.0	56
1,3-cis-dichloropropene	1.0	130
2-chloroethylvinylether	1.0	90
bromoform	1.0	97
ethane, 1,1,2,2-tetrachloro-	1.0	96
ethene, tetrachloro-	1.0	99
toluene	1.0	116
chlorobenzene	1.0	145
ethylbenzene	1.0	157

<u>Constituent</u>	<u>Amount of Spike, ug</u>	<u>% Recovery</u>
1,1-dichloroethane	1.0	87
Chloroform	1.0	96
1,1,1-trichloroethane	1.0	78
1,2-dichloropropane	1.0	99
Trans-1,3-dichloropropene	1.0	62
Trichloroethylene (TCE)	1.0	101
Benzene	1.0	76
Dibromochloromethane	1.0	120
1,1,2-trichloroethane	1.0	129
cis-1,3-dichloropropene	1.0	100
Tetrachloroethylene	1.0	86
Toluene	1.0	90
Chlorobenzene	1.0	100

104

TITLE: Compatibility Testing (Physical, Qualitative)

REFERENCE: S-R Analytical In-House Method

SCOPE AND APPLICATION

This series of tests determines the physical characteristics of unknown solid waste samples in order to classify and group similar samples for the purpose of compositing. These composites facilitate subsequent quantitative analysis and disposal.

SAMPLING HANDLING

The samples analyzed should be considered hazardous and handled with extreme care, using all safety precautions. ALL WORK SHOULD BE DONE IN A HOOD. USE ONLY SMALL (ABOUT 1 GRAM) ALIQUOTS OF SAMPLE.

PROCEDURE

1. H₂O Solubility/Reactivity

To 5 grams of sample in a 125 ml wide-mouth bottle slowly add 50 ml deionized water. Note reactivity and solubility. If insoluble, save for pH, redox potential, cyanide, sulfide. Record as soluble, partially soluble or insoluble for water solubility, and positive or negative or negative for reactivity.

2. Hexane Solubility

Transfer a small aliquot of sample (approx. one gram) to a 10 ml screwcap vial. Mark level of material in vial. Slowly add 5 ml hexane by means of a Repipet. Cap vial and shake vigorously; allow contents to settle. Report as soluble, partially soluble, or insoluble.

3. Redox Potential

Measure redox potential using at Pt/Ag/AgCl combination electrode according to the manufacturer's instructions. Measure aqueous samples directly and the leachates of non-aqueous samples. Record redox potential in mv.

4. pH

Check pH of aqueous leachates with wide range (0-14) pH test strips. If pH is less than 4, confirm pH with 0-6 test strips. If pH is greater than 10, confirm pH with 11-14 test strips. Record pH in units.

SECTION C

EXAMPLE DATA REPORT

S-R ANALYTICAL, INC.



ANALYTICAL INC.

28 Springdale Rd. Cherry Hill, NJ. 08003
(609) 751-1122 (215) 923-2068

Analytical Data Report Package

for

XYZ Corporation
1 Industrial Plaza
Washington, D.C. 09154

Project: Waste Landfill

<u>Client Designation #</u>	<u>S-R Sample #</u>	<u>Sample Location</u>	<u>Matrix</u>	<u>Date & Time of Sampling</u>	<u>Sampled By</u>
XYZ-1	ABCD-1	MW 1	Aqueous	6/10/84 0710	S-R

Lab Name Stablex-Reutter, Inc.

Certification # NJ 04012

Project/Laboratory Manager Signature Catherine M. Ward

Name Catherine M. Ward

PROFESSIONAL LABORATORY/CONSULTING SERVICES

SYN 001 1958

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LEGEND

A. Definition of Terms

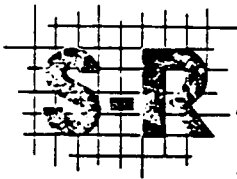
ND	Not Detected (This includes compounds analyzed by GC/MS/DS that did not pass the EPA peak-match criteria)
BMDL	Below Method Detection Limit (The compound was detected but at levels too low to be accurately quantified)
%RPD	Relative Percent Difference (Computed as follows: $\frac{\text{Difference between Replicates}}{\text{Average of Replicates}} \times 100$)

B. Footnotes

1. The method detection limits referenced for volatile organics are those recommended in EPA Method 624, Federal Register, Vol. 44, No. 233, December 3, 1979.

I CHAIN OF CUSTODY/ NOTEBOOK ACCOUNTABILITY

SYN 001 1961



ANALYTICAL INC.

28 Springdale Rd., Cherry Hill, NJ. 08003
(609) 751-1122 (215) 923-2068

CHAIN OF CUSTODY RECORD

Name of Unit and Address						
Number	Item	Description of Samples				
		Remarks:				
Person Assuming Responsibility for Samples:					Time	Date
Number	Relinquished by:	Received by:	Time	Date	Reason for Change of Custody	
Number	Relinquished by:	Received by:	Time	Date	Reason for Change of Custody	
Number	Relinquished by:	Received by:	Time	Date	Reason of Change of Custody	
Number	Relinquished by:	Received by:	Time	Date	Reason of Change of Custody	

SYN 001 1962

[illegible]

SYN 001 1963

II METHODOLOGY REVIEW

N.J.D.E.P. PROJECTS - PROPOSED INTERIM PROTOCOL

GC/MS/DS STANDARD OPERATION PROCEDURE OVERVIEW

I. PURGEABLE ORGANICS

A. Holding Times

1. Aqueous samples will be analyzed within 14 days of sample receipt.
2. Soil/solid waste samples will be analyzed within 14 days of sample receipt.

B. Preparation Procedure

1. None for aqueous. An aliquot of sample is added to a 35 ml purge vessel along with internal reference standard and surrogates.
2. Soil/solid waste samples will be prepared as follows:

A known weight of sample is added to a screwcap test tube with 10 ml of methanol. The tube is sealed, agitated and allowed to sit in a freezer for no less than 1 hour. An aliquot of the methanol extract is then transferred to a 35 ml purge vessel along with 30 ml of DI Water, and an internal reference standard and surrogates added for recovery purposes.

C. Analysis

1. Aqueous and soil/solid waste samples will be analyzed according to Method 624, Test Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, June, 1982 revision.
2. Detection Limits for aqueous samples will be reported in accordance with the December 3, 1979 version of Method 624. Soil/solid waste limits will be 0.33 ppm.

D. Tune

1. The tune of the GC/MS/DS instruments will be checked just prior to the start of each 12 hour shift. They will be retuned if required.
2. The tune will be performed with + 1 scans of the apex of the peak of the tuning compound (BFB).

E. Calibration

1. An initial 3-point calibration curve will be generated and a calibration check will suffice in subsequent shifts as long as the percent change of each of the check compounds is less than 25% using the following compounds :

vinyl chloride	toluene
1,1-dichloroethene	bromodichloromethane
chloroform	

2. The lowest calibration standard will be prepared at a level of 0.5 micrograms.

F. Internal Standards

- The following compounds will be used:
 - 1,4-dichlorobutane
 - 2-bromo, 1-chloropropane
 - bromochloromethane

G. Surrogates

1. The following surrogate compounds will be used:
 - d8-toluene
 - 4-bromofluorobenzene
2. The control limits will be those listed in Exhibit B (pgs. 22 and 23 of 42).
3. NJDEP recognizes that up to 15% of the surrogate recoveries are expected to fall outside the control limits without adversely affecting the overall data. If a surrogate recovery does fall outside the control limits, the sample will be reanalyzed to demonstrate that the same surrogate falls outside in the same direction.
4. The surrogates will be added at a level of 50 ppb for aqueous samples and 5 ppm for soil/solid waste samples.

H. Quality Control

1. One sample in twenty will be analyzed in duplicate. One sample in twenty will be spiked. If the spike recoveries or duplicate RSD's should fall outside the control limits generated by S-R but the surrogate recoveries are within the established limits, then no re-analysis will be done.
2. A method blank will be prepared with every group of samples received on a given day within a given matrix. The prepared method blank will be analyzed every twenty samples or more frequently if there is a positive result obtained from analysis of the trip or field blanks.

II. SEMIVOLATILE ORGANICS (Acid Extractables, Base/Neutral Extractables)

A. Holding Times

1. Aqueous samples will be prepared within 7 days of sample receipt. Analysis will be done within 40 days of sample preparation.
2. Soil/solid waste samples will be prepared within 7 days of sample receipt. Analysis will be done within 40 days of sample preparations.

B. Preparation Procedure

1. Aqueous samples will be prepared according to the following extraction procedure:

A known volume of sample is adjusted with 6 M NaOH to $\text{pH} \geq 11$. The sample is extracted three times with pesticide-grade methylene chloride and the extracts combined in a Kuderna-Danish (K-D) apparatus. The sample is then adjusted with 6M HCl to a $\text{pH} \leq 2$ and extracted three more times with methylene chloride. These extracts are combined in a second K-D apparatus. Both sets of extracts are then evaporated over a hot water bath to a final volume of 5 milliliters and recombined just prior to injection.

2. Soil/solid waste samples will be prepared according to Method 3550, SW846, Methods for the Analysis of Solid Waste, Physical/Chemical Methods, July, 1982 revision.
3. Soil/solid waste samples to be screened for dioxin will be prepared by splitting the extract obtained by the above procedures and boiling half of it down to a final volume of 0.2 milliliters.

C. Analysis

1. Aqueous and soil/solid waste samples will be analyzed according to Method 625, Test Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, July, 1982, revision.
2. Detection limits will be reported in accordance with the December 3, 1979 version of Method 625. Soil/solid waste detection limits will be 0.33 ppm for acid extractables and 0.33 ppm for base/neutral extractable compounds.

D. Tune

1. The tune of the GC/MS/DS instruments will be checked just prior to the start of each 12 hour shift. They will be retuned if required.
2. The tune will be performed on the apex of the peak of the tuning compound (DFTPP).

E. Calibration

1. An initial three-point calibration curve will be generated and a calibration check will suffice in subsequent shifts as long as the specified target compounds have been attained. These compounds are

phenol
1,4-Dichlorobenzene
2-Nitrophenol
Bis (2-chloroethoxy) methane
4-Chloro-m-cresol
Hexachlorocyclopentadiene
2,4,6-Trichlorophenol
Acenaphthene
Di-n-octylphthalate
Benzo (a) pyrene

If one or two compounds fall out, the curve does not have to be re-generated if the individual differences do not exceed 50% and the CCC mean is within 25%, , but the data must be qualified on the report. If three compounds fall outside the 25% control limit, NJDEP will entertain discussion on continuing the analysis without calibration, as long as the outliers are within 50%.

2. The lowest calibration standard will be prepared at a level of 20 nanograms.

F. Internal Standards

- The following compound will be used:

dl0-Anthracene

SYN 001 1968

G. Surrogates

1. The following surrogate compounds will be used and the control limits attained:

<u>Compound</u>	<u>Control Limit</u>	
	<u>Aqueous</u>	<u>Soil/Solid Waste</u>
d5-Nitrobenzene	41-120	24-115
2-Fluorobiphenyl	44-119	37-120
d5 -Phenol	15-96	20-106
2-Fluorophenol	23-107	24-111
1,2,3,4-TCDD (if available)	NA	NA

NA - Not Available

3. NJDEP recognizes that up to 15% of the surrogate recoveries are expected to fall outside the control limits without adversely affecting the overall data. If a surrogate recovery does fall outside the control limits, the sample will be reanalyzed to demonstrate that the same surrogate falls outside in the same direction.
4. The surrogates and internal reference standards will be added at a level of 50 ppb for aqueous samples and ppm for soil/solid waste samples. The internal reference standard will be added at a level of 50 ppb for aqueous samples and ppm for soil/solid waste samples.

H. Quality Control

1. One sample in twenty will be analyzed in duplicate. One sample in twenty will be spiked. If one or two of the spike recoveries and duplicate RSD's should fall outside the control limits generated by S-R, but the surrogate recoveries are within the established limits, then no re-analysis will be done.
2. A method blank will be prepared with every group of samples received on a given day within a given matrix. The prepared method blank will be analyzed every twenty samples or more frequently if there is a positive result obtained from analysis of the trip or field blank.

III QUANTITATIVE RESULTS AND QUALITY ASSURANCE DATA

A. ORGANIC DATA

SYM 001 1970

E. Surrogates

1. The following surrogate compound will be used and the control limits attained.

<u>Control Limits</u>		
<u>Compound</u>	<u>Aqueous</u>	<u>Soil/Solid Waste</u>
Dibutyl chlorendate	67 - 114*	0 - 205*

* For advisory purposes only.

2. The surrogates will be added at a level of 50 ppb for aqueous samples and 5 ppm for soil/solid waste samples.

F. Quality Control

1. One sample in twenty will be analyzed in duplicate. One sample in twenty will be spiked.
2. A method blank will be prepared with every group of samples received on a given day within a given matrix. The prepared method blank will be analyzed every twenty samples or more frequently if there is a positive result obtained from analysis of the trip or field blanks.

IV. RE-ANALYSIS

- If a re-analysis should be requested by NJDEP or required by some defect in the original data, NJDEP recognizes that it may occur outside the holding times but will not discard the data on that basis. Every effort will be made, however to remain within the holding times for reanalysis.

SYN 001 1971

III. SEMIVOLATILE ORGANICS (Pesticides and Polychlorinated Biphenyls)

A. Holding Times

1. Aqueous and soil/solid waste samples will be prepared within 7 days of sample receipt. Analysis will be done within 40 days of sample preparation.

B. Preparation Procedure

1. Aqueous samples will be prepared according to the following extraction procedure:

A known volume of sample is adjusted with 6 M NaOH to $\text{pH} \geq 11$. The sample is extracted three times with pesticide-grade methylene chloride and the extracts combined in a Kuderna-Danish (K-D) apparatus. The sample is then adjusted with 6M HCl to a $\text{pH} \leq 2$ and extracted three more times with methylene chloride. Both sets of extracts are then evaporated over a hot water bath to a final volume of 10 milliliters and recombined just prior to injection.

2. Soil/solid waste samples will be prepared according to Method 3550, SW846, Methods for the Analysis of Solid Waste, Physical/Chemical Methods, July, 1982 revision.

C. Analysis

1. Aqueous and soil/solid waste samples will be analyzed according to EPA Method 608, Organochlorine Pesticides and PCB's, Federal Register, Vol. 44, No. 233, December 3, 1979.
2. Detection limits for aqueous samples will be reported in accordance with the procedure listed above. Soil/solid waste detection limits will be 5 ppm.

D. Calibration

1. The GC/ECD will be calibrated with an initial 3-point calibration and a calibration check will suffice in subsequent shifts.

GC/MS STANDARD DECAFLUOROTRIPHENYLPHOSPHINE (DFTPP) TUNE

CRITERIA FOR SEMIVOLATILES, 50ng LEVEL OR LESS

- Lab Sample No. ABCD -1

- Instrumentation (Manufacturer/Model): Finnigan/5100

°GC Finnigan 9611

*Serial Number - MS 13037-1083

°MS Finnigan 5100

°Interface (GC/MS) (Direct) x

°Column:

(OSCI)

(Other/Specify)

(Other/Specify) _____ 20°/minute to _____
 _____ 290°, final h _____
 _____ 15 minutes _____

*Scan(s) Background Subtraction --

°Enhanced (S15B2NOT) --

Other

- DFTPP Acquisition (50ng)

°Date: 5/8/84

°Time: 16:15

° Scan No: 463

°Scan(s): 462-464

(Apex of Eluting Peak)

(Employed, + 1 scan
from Apex)

- Analyst WILLIAM A. FITHIAN

(Signature)

IFICATIONS ACHIEVED

m/e	Ion Abundance Criteria	% Relative Abundance 198 m/z (base peak)	% Relative Abundance of Required Ion
51	30 - 60% of base mass	32.07	
68	Less than 2% of mass 69	0.58	1.47
69	Mass 69 relative abundance	39.57	
70	Less than 2% of mass 69	0	0
127	40 - 60% of mass 198	42.50	
197	Less than 1% of mass 198	0.41	
198	Base peak, 100% relative abundance	100	
199	5 - 9% of mass 198	7.99	
275	10 - 30% of mass 176	23.97	
365	Greater than 1% of mass 198	2.10	
441	Less than mass 443	11.10	
442	Greater than 40% of mass 198	67.39	
443	17 - 23% of mass 442	14.65	21.74

(1) Value is % of mass 69.

(2) Value is % of mass 442.

DFTPP Performance Results:

X__ The DFTPP performance results were reviewed and found to be within the specified criteria.

APPROVAL :

Supervisor GC/MS Group Signature Michael Smolish

(Print/Typed) Michael Shmookler

SYN 001 1973

GC/MS STANDARD DECAFLUOROTRIPHENYLPHOSPHINE (DFTPP) TUNE

CRITERIA FOR VOLATILES, 50ng LEVEL OR LESS

- Lab Sample No. ABCD-1
 - Instrumentation (Manufacturer/Model): Finnigan/5100
 °GC Finnigan 9611
 °MS Finnigan 5100
 °Interface (GC/MS) (Direct) x
 (OSCI) _____
 (Other/Specify) _____
 °Serial Number - MS 13243-0684
 °Column: _____
 (FSCC) 30m DB-5 Temp Program Used 150°/1 minute
 20°/minute to
 225°; final h
 15 minutes
 - DFTPP Acquisition (50ng) °Scan(s) Background Subtraction ---
 °Date: 6/25/84 °Time: 11:30
 °Scan No: 720 °Scan(s): 719-722
 (Apex of Eluting (Employed, + 1 scan
 Peak) from Apex)
 - Analyst WILLIAM A. FITHIAN (Signature) William A. Fithian Other _____

SPECIFICATIONS ACHIEVED

m/e	Ion Abundance Criteria	% Relative Abundance 198 m/z (base peak)	% Relative Abundance of Required Ion
51	30 - 60% of base mass	41.67	
68	Less than 2% of mass 69	0	0
69	Mass 69 relative abundance	45.57	
70	Less than 2% of mass 69	0.29	0.64
127	40 - 60% of mass 198	57.62	
197	Less than 1% of mass 198	0	
198	Base peak, 100% relative abundance	100	
199	5 - 9% of mass 198	6.74	
275	10 - 30% of mass 176	16.73	
365	Greater than 1% of mass 198	2.01	
441	Less than mass 443	7.89	
442	Greater than 40% of mass 198	51.15	
443	17 - 23% of mass 442	9.65	18.87

(1) Value is % of mass 69.

(2) Value is % of mass 442.

DFTPP Performance Results:

___ The DFTPP performance results were reviewed and found to be within the specific criteria.

APPROVAL:

Supervisor GC/MS Group Signature Michael Shmookler

(Print/Typed) Michael Shmookler

SYN
001
1974

Initial Calibration Data - Semivolatile HSL Compounds

Instrument Identifier Finnegan 5100A Calibration Date 5/8/84

COMPOUNDS	RRF 20	RRF 40	RRF 60	$\overline{\text{RRF}}$
N-nitrosodimethylamine	0.01	0.02	0.03	0.02
bis (2-chloroethyl) ether	0.28	0.29	0.30	0.29
2-chlorophenol	0.23	0.24	0.25	0.24
phenol	0.28	0.29	0.30	0.29
1,3-dichlorobenzene	0.38	0.39	0.40	0.39
1,4-dichlorobenzene	0.39	0.40	0.41	0.40
1,2-dichlorobenzene	0.41	0.42	0.43	0.42
bis (2-chloroisopropyl) ether	0.24	0.25	0.26	0.25
hexachloroethane	0.15	0.16	0.17	0.16
N-nitroso-di-n-propylamine	0.24	0.25	0.26	0.25
nitrobenzene	0.23	0.24	0.25	0.24
isophorone	0.50	0.51	0.52	0.51
2-nitrophenol	0.12	0.13	0.14	0.13
2,4-dimethylphenol	0.17	0.18	0.19	0.18
bis (2-chloroethoxy) methane	0.28	0.29	0.30	0.29
2,4-dichlorophenol	0.24	0.25	0.26	0.25
1,2,4-trichlorobenzene	0.40	0.41	0.42	0.41
naphthalene	0.88	0.89	0.90	0.89
hexachlorobutadiene	0.28	0.29	0.30	0.29
4-chloro-m-cresol	0.20	0.21	0.22	0.21
hexachlorocyclopentadiene	0.01	0.2	0.03	0.02
2,4,6-trichlorophenol	0.21	0.22	0.23	0.22
2-chloronaphthalene	0.70	0.71	0.72	0.71
acenaphthylene	0.83	0.84	0.85	0.84
dimethyl phthalate	0.79	0.80	0.81	0.80
2,6-dinitrotoluene	0.17	0.18	0.19	0.18
acenaphthene	0.62	0.63	0.64	0.63
2,4-dinitrophenol	0.01	0.02	0.03	0.02
2,4-dinitrotoluene	0.17	0.18	0.19	0.18
4-nitrophenol	0.02	0.03	0.04	0.03
fluorene	0.65	0.66	0.67	0.66
4-chlorophenylphenylether	0.15	0.16	0.17	0.16
diethyl phthalate	0.73	0.74	0.75	0.74
4,6-dinitro-o-cresol	0.07	0.08	0.09	0.08
diphenylamine	0.07	0.08	0.09	0.08
azobenzene	0.24	0.25	0.26	0.25
4-bromophenyl-phenyl ether	0.29	0.30	0.31	0.30
Hexachlorobenzene	0.47	0.48	0.49	0.48

RRF - Response Factor (subscript is the amount in nanograms)

 $\overline{\text{RRF}}$ - Average Response Factor

SYN 001 1975

Initial Calibration Data - Semivolatile HSL Compounds

Instrument Identifier Finnegan 5100A Calibration Date 5/8/84

COMPOUNDS	RRF 20	RRF 40	RRF 60	$\overline{\text{RRF}}$
pentachlorophenol	0.09	0.10	0.11	0.10
phenanthrene	0.78	0.79	0.80	0.79
anthracene	0.78	0.79	0.80	0.79
dibutyl phthalate	0.97	0.98	0.99	0.98
floranthene	0.83	0.84	0.85	0.86
pyrene	0.83	0.84	0.85	0.86
benzidine	0.14	0.15	0.16	0.15
butyl benzyl phthalate	0.73	0.74	0.75	0.74
benzo(a)anthracene	0.17	0.18	0.19	0.18
chrysene	0.17	0.18	0.19	0.18
3,3-dichlorobenzidine	0.00	0.01	0.02	0.01
bis (2-ethylhexyl) phthalate	0.19	0.20	0.21	0.20
di-n-octyl phthalate	0.73	0.74	0.75	0.74
benzo (b) fluoranthene	0.00	0.01	0.02	0.01
benzo (k) fluoranthene	0.04	0.05	0.06	0.05
benzo (a) pyrene	0.02	0.03	0.04	0.03
indeno (1,2,3-cd) pyrene	0.01	0.02	0.03	0.02
dibenzo (a,h) anthracene	0.003	0.004	0.005	0.004
benzo (g,h,i) perylene	0.01	0.02	0.03	0.02

RRF - Response Factor (subscript is the amount in nanograms)

 $\overline{\text{RRF}}$ - Average Response Factor

SYN 001 1976

Initial Calibration Data - Volatile HSL Compounds

Instrument Identifier Finnegan 5100B Calibration Date 6/25/84

COMPOUNDS	RRF 100	RRF 1000	RRF 1500	RRF
chloromethane	52	53	54	53
bromomethane	110	111	112	111
dichlorodifluoromethane	86	87	88	87
vinyl chloride	53	54	55	54
chloroethane	26	27	28	27
methylene chloride	38	39	40	39
trichlorofluoromethane	42	43	43	42
ethene, 1,1-dichloro-	38	39	39	38
ethane, 1,1-dichloro-	14	15	16	15
1,2-trans-dichloroethene	39	40	41	40
chloroform	57	58	59	58
ethane, 1,2-dichloro-	14	15	16	15
ethane, 1,1,1-trichloro-	43	44	45	44
carbon tetrachloride	14	15	16	15
bromodichloromethane	35	36	37	36
propane, 1,2-dichloro-	21	22	23	22
1,3-trans-dichloropropene	32	33	34	33
trichloroethylene	41	42	43	42
chlorodibromomethane	20	21	22	21
benzene	101	102	103	102
ethane, 1,1,2-trichloro-	16	17	18	17
1,3-cis-dichloropropene	12	13	14	13
2-chloroethyl vinyl ether	2.9	3.9	4.9	3.9
bromoform	8.2	9.2	10.2	9.2
ethane, 1,1,2,2-tetrachloro-	10	11	12	11
ethene, tetrachloro-	65	66	67	66
toluene	70	71	72	71
chlorobenzene	45	46	47	46
ethylbenzene	8.7	9.7	10.7	9.7

RRF - Response Factor (subscript is the amount of nanograms)

RRF - Average Response Factor

SYN
001
1977

Initial Calibration Data - Pesticides/Polychlorinated HSL Compounds

Instrument Identifier Tracor #3 Calibration Date 5/15/84

COMPOUNDS	RRF 20	RRF 40	RRF 60	RRF
aldrin	0.71	0.71	0.75	11.9
alpha BHC	0.81	0.81	0.81	12.5
beta BHC	0.75	0.76	0.76	8.7
gamma BHC	0.45	0.45	0.43	7.5
delta BHC	0.38	0.36	0.37	6.0
chlordan	0.82	0.81	0.82	7.5
dieldrin	0.67	0.64	0.65	10.4
p,p'-DDD	0.37	0.36	0.37	5.8
p,p'-DDE	0.68	0.66	0.63	10.6
p,p'-DDT	0.55	0.59	0.55	8.6
Endosulfan I	0.65	0.65	0.62	8.0
Endosulfan II	0.87	0.84	0.88	9.5
Endosulfan Sulfate	0.75	0.73	0.72	10.1
Endrin	0.32	0.31	0.32	5.0
Endrin Aldehyde	0.51	0.51	0.51	5.6
Heptachlor	0.41	0.40	0.41	6.4
Heptachlor Epoxide	0.55	0.50	0.58	8.6
Toxaphene	0.80	0.79	0.82	6.7
Aroclor 1016	0.55	0.55	0.55	9.8
Aroclor 1221	0.97	0.97	0.97	11.2
Aroclor 1232	0.86	0.86	0.88	10.5
Aroclor 1242	0.73	0.74	0.73	5.8
Aroclor 1248	0.42	0.42	0.44	6.7
Aroclor 1254	0.66	0.66	0.66	7.9
Aroclor 1260	0.51	0.50	0.54	8.8

RRF - Response Factor (subscript is amount in nanograms)

RRF - Average Response Factor

SYN 001 1978

Calibration Check - Semivolatile HSL Compounds

Instrument Identifier Finnegan 5100Calibration Date 5/8/84Standard File (RUN) 62458Date 5/8/84-Time 1650

Maximum % D for CCC is 25

COMPOUNDS	$\overline{\text{RRF}}$	RRF	% D	CCC
N-nitrosodimethylamine	0.02	0.01	50	
bis (2-chloroethyl) ether	0.29	0.28	3.4	
2-chlorophenol	0.24	0.23	4.2	
phenol	0.29	0.28	3.4	*
1,3-dichlorobenzene	0.39	0.38	2.6	
1,4-dichlorobenzene	0.40	0.39	2.5	*
1,2-dichlorobenzene	0.42	0.41	2.4	
bis (2-chloroisopropyl) ether	0.25	0.24	4.0	
hexachloroethane	0.16	0.15	6.2	
N-nitroso-di-n-propylamine	0.25	0.24	4.0	
nitrobenzene	0.24	0.23	4.2	
isophorone	0.51	0.50	2.0	
2-nitrophenol	0.13	0.12	7.7	*
2,4-dimethylphenol	0.18	0.17	5.6	
bis (2-chloroethoxy) methane	0.29	0.28	3.4	*
2,4-dichlorophenol	0.25	0.25	4.0	
1,2,4-trichlorobenzene	0.41	0.40	2.4	
naphthalene	0.89	0.88	1.1	
hexachlorobutadiene	0.28	0.27	3.4	
4-chloro-m-cresol	0.21	0.20	4.8	*
hexachlorocyclopentadiene	0.02	0.01	0	*
2,4,6-trichlorophenol	0.22	0.21	4.5	*
2-chloronaphthalene	0.71	0.70	1.4	
acenaphthylene	0.84	0.83	1.2	
dimethyl phthalate	0.80	0.79	1.2	
2,6-dinitrotoluene	0.18	0.17	5.6	
acenaphthene	0.63	0.62	1.6	*
2,4-dinitrophenol	0.02	0.01	50	
2,4-dinitrotoluene	0.18	0.17	5.6	
4-nitrophenol	0.03	0.02	33	
fluorene	0.66	0.65	1.5	
4-chlorophenylphenylether	0.16	0.15	6.2	
diethyl phthalate	0.74	0.73	1.4	
4,6-dinitro-o-cresol	0.08	0.07	12	
diphenylamine	0.08	0.07	12	
azobenzene	0.25	0.24	4.0	
4-bromophenyl-phenyl ether	0.30	0.29	3.3	
Hexachlorobenzene	0.48	0.47	2.1	

 $\overline{\text{RRF}}$ - Average Response Factor from initial calibration form

RRF - Response Factor from daily standard file

% D - Percent Difference

CCC - Calibration Check Compounds (Those compounds flagged with an * must be achieved)

SYN 001 1979

Calibration Check - Semivolatile HSL Compounds (CONT'D)

Instrument Identifier Finnegan 5100 A Calibration Date 5/8/84Standard File (RUN) 62458 Date 5/8/84 Time 1650

Maximum % D for CCC is 25

COMPOUNDS	RRF	RRF	% D	CCC
pentachlorophenol	0.10	0.11	10	
phenanthrene	0.79	0.78	1.3	
anthracene	0.79	0.78	1.3	
dibutyl phthalate	0.98	0.99	1.0	
floranthene	0.84	0.85	1.2	
pyrene	0.84	0.85	1.2	
benzidine	0.15	0.14	6.7	
butyl benzyl phthalate	0.74	0.75	1.4	
benzo(a)anthracene	0.18	0.17	5.6	
chrysene	0.18	0.17	5.6	
3,3-dichlorobenzidine	0.01	0.00	100	
bis (2-ethylhexyl) phthalate	0.20	0.19	5.0	
di-n-octyl phthalate	0.74	0.75	1.4	*
benzo (b) fluoranthene	0.01	0.00	100	
benzo (k) fluoranthene	0.05	0.04	20	
benzo (a) pyrene	0.03	0.02	0	*
indeno (1,2,3-cd) pyrene	0.02	0.01	0	
dibenzo (a,h) anthracene	0.004	0.003	25	
benzo (g,h,i) perylene	0.02	0.01	50	

RRF - Average Response Factor from initial calibration form

RRF - Response Factor from daily standard file

% D - Percent Difference

CCC - Calibration Check Compounds (Those compounds flagged with an * must be achieved)

SYN 001 1980 0861

Calibration Check - Volatile HSL Compounds

Instrument Identifier Finnigan 5100 B Calibration Date 6/25/84
 Standard File (RUN) ABSTD625 Date 6/25/84 Time 0950

Maximum % D for CCC is 25

COMPOUNDS	<u>RRF</u>	RRF	% D	CCC
chloromethane	53	54	1.8	
bromomethane	111	112	0.9	
dichlorodifluoromethane	87	88	1.1	
vinyl chloride	54	55	1.8	*
chloroethane	27	28	3.7	
methylene chloride	39	40	2.6	
trichlorofluoromethane	43	44	2.3	
ethene, 1,1,-dichloro-	39	40	2.6	*
ethane, 1,1-dichloro-	15	16	6.7	
1,2-trans-dichloroethene	40	41	2.5	
chloroform	58	59	1.7	*
ethane, 1,2-dichloro-	15	16	6.7	
ethane, 1,1,1-trichloro-	44	45	2.2	
carbon tetrachloride	15	16	6.7	
bromodichloromethane	36	37	2.8	*
propane, 1,2-dichloro-	22	23	4.5	
1,3-trans-dichloropropene	33	34	3.0	
trichloroethylene	42	43	2.3	
chlorodibromomethane	21	22	4.8	
benzene	102	103	1.0	
ethane, 1,1,2--trichloro-	17	18	5.9	
1,3-cis-dichloropropene	13	14	7.7	
2-chloroethyl vinyl ether	3.9	4.9	2.6	
bromoform	9.2	10.2	1.1	
ethane, 1,1,2,2-tetrachloro-	11	12	9.1	
ethene, tetrachloro-	66	67	1.5	
toluene	71	72	1.4	*
chlorobenzene	47	48	2.1	
ethylbenzene	9.7	10.7	1.0	

RRF - Average Response Factor from initial calibration Form VII

RRF - Response Factor from daily standard file

% D - Percent Difference

CCC - Calibration Check Compounds (Those compounds with an * must be achieved.)

EXN
001
1981

Calibration Check - Pesticide/Polychlorinated Biphenyl HSL Compounds

Instrument Identifier Tractor #3 Calibration Date 5/15/84
 Standard File (RUN) Pestck 1 Date 7/15/84 Time 1440

Maximum % D for CCC is 25

COMPOUNDS	<u>RRF</u>	RRF	% D
aldrin	11.0	10	9.0
alpha BHC	12.6	13.6	7.9
beta BHC	8.7	7.7	11
gamma BHC	7.5	6.5	13
delta BHC	6.0	5.0	17
chlordane	7.5	8.5	13
dieldrin	10.4	11.4	9.6
p,p'-DDD	5.8	6.8	9.4
p,p'-DDE	10.6	11.6	12
p,p'-DDT	8.6	9.5	12
Endosulfan I	8.0	9.0	10
Endosulfan II	9.5	10.5	9.9
Endosulfan Sulfate	10.1	11.1	20
Endrin	5.0	6.0	18
Endrin Aldehyde	5.6	6.6	16
Heptachlor	6.4	7.4	12
Heptachlor Epoxide	8.6	9.6	15
Toxaphene	6.7	7.7	10
Aroclor 1016	9.8	10.8	8.9
Aroclor 1221	11.2	12.2	9.5
Aroclor 1232	10.5	11.5	9.5
Aroclor 1242	5.8	6.8	17
Aroclor 1248	6.7	7.7	15
Aroclor 1254	7.9	8.9	13
Aroclor 1260	8.8	9.8	11

RRF - Average Response Factor from initial calibration from

RRF - Response Factor from daily standard file

% D - Percent Difference

SYN 001 1982

GC And GC/MS Surrogate Recovery Data

Sample No. ABCD -1 Lab Control Number EPA5 Sample Matrix Aqueous

COMPOUND	CONCENTRATION ADDED TO SAMPLE MATRIX (Nanograms)	% REC'Y	CONTROL LIMITS (R + 3S) (Laboratory generated)		QUALIFIED
			LOWER	UPPER	
<u>Volatile Fraction - GC/MS</u> (2 required)					
1) D8-Toluene	158	102	86	119	
2) BFB	202	103	85	121	
<u>Acid Fraction - GC/MS</u> (2 required)					
1) 2-Fluorophenol	124	68	24	111	
2) D5-Phenol	105	60	20	101	
<u>Base/Neutral Fraction - GC</u> (2 required)					
1) 2-Fluorobiphenyl	200	82	44	119	
2) D5-Nitrobenzene	264	70	24	115	
<u>Organochlorine Pesticide/PCB Fraction</u> (1 required)					
1) Dibutyl chlorendate	180	NA	0*	205*	
<u>2, 3, 7, 8 - TCDD - GC/MS</u>					
1) ³⁷ Cl ₄ Isotope 2, 3, 7, 8, - TCDD m.w. 328 (Require)	190	NA	18*	128*	

SYN 001

* Advisory Limits set by EPA.

SYN 001 1983

Client/Project XYZ Corp.Analysis SemivolatilesClient Designation # XYZ-1 Sample Matrix AqueousDate Completed 7/1/84S-R Sample # ABCD-1 Time 1800 to 1850Duplicate Sample No. ABCD-1Spike Sample No. ABCD-4

PARAMETER	RESULTS		QC REPLICATE			QC BLANK	STDS/CAL. CHECK				QC MATRIX SPIKE		
	CONCENTRATION		CONCENTRATION			BLANK	TRUE VALUE	REPORTED	Z REC'Y	CONTROL LIMIT	SAMPLE	SPIKE ADDED	Z REC'Y
	SAMPLE	MDL	FIRST	SECOND	% RPD								
bis (2-chloroethyl) ether	ND	10	--	--	--	ND	50	33	66	72 + 74	ND	30	84
bis (2-chloroisopropyl) ether	ND	10	--	--	--	ND	50	65	130	71 + 40	ND	30	80
4-bromophenyl phenyl ether	ND	10	--	--	--	ND	50	40	80	75 + 40	ND	30	65
4-chlorophenyl phenyl ether	ND	10	--	--	--	ND	50	44	88	45 + 22	ND	30	54
bis (2-chloroethoxy) methane	ND	10	--	--	--	ND	50	20	40	82 + 148	ND	30	70
dimethylphthalate	ND	10	--	--	--	ND	50	15	30	35 + 72	ND	30	88
dibutyl phthalate	ND	10	--	--	--	ND	50	66	132	93 + 102	ND	30	50
bis (2-ethylhexyl) phthalate	ND	10	--	--	--	ND	50	80	160	82 + 126	ND	30	43
butyl benzyl phthalate	ND	10	14	16	13	ND	50	70	140	74 + 86	ND	30	32
dioctyl phthalate	14	10	--	--	--	ND	50	52	104	89 + 64	ND	30	91
diethyl phthalate	ND	10	--	--	--	ND	50	55	110	48 + 56	ND	30	105
hexachlorobenzene	ND	10	--	--	--	ND	50	60	120	71 + 44	ND	30	30
1,2-dichlorobenzene	ND	10	--	--	--	ND	50	33	66	62 + 56	ND	30	120
1,3-dichlorobenzene	BMDL	10	--	--	--	ND	50	45	90	54 + 48	ND	30	88
1,4-dichlorobenzene	ND	10	--	--	--	ND	50	61	122	63 + 70	ND	30	70
1,2,4-trichlorobenzene	ND	10	--	--	--	ND	50	52	104	69 + 52	ND	30	55
nitrobenzene	ND	10	--	--	--	ND	50	15	30	82 + 108	ND	30	76
2,4-dinitrotoluene	ND	10	--	--	--	ND	50	28	56	79 + 68	ND	30	77
2,6-dinitrotoluene	ND	10	--	--	--	ND	50	56	112	79 + 50	ND	30	40
benzidine	ND	10	--	--	--	ND	50	29	58	63 + 110	ND	30	99
3,3'-dichlorobenzidine	ND	10	--	--	--	ND	50	44	88	143 + 280	ND	30	55
N-nitrosodipropyl amine	ND	10	--	--	--	ND	50	10	20	76 + 90	ND	30	140
N-nitrosodiphenyl amine	ND	10	--	--	--	ND	50	48	96	86 + 36	ND	30	87
N-nitrosodimethyl amine	ND	10	--	--	--	ND	50	45	90	70 + 25	ND	30	66
benzo (b) fluoranthene	ND	10	--	--	--	ND	50	30	60	41 + 42	ND	30	67
phenanthrene	ND	10	--	--	--	ND	50	19	38	76 + 44	ND	30	45
fluoranthene	ND	10	--	--	--	ND	50	20	40	80 + 52	ND	30	32
benzo (k) fluoranthene	ND	10	--	--	--	ND	50	15	30	47 + 54	ND	30	50
benzo (a) pyrene	ND	10	--	--	--	ND	50	40	80	43 + 42	ND	30	42
Units	(ppb)	(ppb)	(ppb)	(ppb)		(ppb)	(ppb)	(ppb)			(ppb)	(ug)	

QUANTITATIVE RESULTS AND QUALITY ASSURANCE DATA

Client/Project XYZ CORP.
 Client Designation # XYZ-1 Sample Matrix Aqueous
 S-R Sample # ABCD-1 Time 1800 to 1850

Analysis Semivolatiles
 Date Completed 7/1/84
 Duplicate Sample No. ABCD-1
 Spike Sample No. ABCD-4

PARAMETER	RESULTS		QC REPLICATE			QC BLANK	STDS/CAL. CHECK				QC MATRIX SPIKE		
	CONCENTRATION		CONCENTRATION			BLANK	TRUE VALUE	REPORTED	Z REC'Y	CONTROL LIMIT	SAMPLE	SPIKE ADDED	Z REC'Y
	SAMPLE	MDL	FIRST	SECOND	Z RPD								
indeno (1,2,3-c,d) pyrene	ND	25	--	--	--	ND	100	75	75	81+86	ND	30	85
benzo (g,h,i) perylene	ND	25	--	--	--	ND	100	87	87	68+80	ND	30	90
naphthalene	27	10	27	32	17	ND	100	95	95	77+70	ND	30	65
2-chloronaphthalene	ND	25	--	--	--	ND	100	40	40	79+54	ND	30	78
fluorene	ND	25	--	--	--	ND	100	42	42	80+40	ND	30	54
anthracene	ND	25	--	--	--	ND	100	54	54	76+44	ND	30	87
pyrene	ND	25	--	--	--	ND	100	85	85	80+46	ND	30	77
benzo (a) anthracene	ND	25	--	--	--	ND	100	34	34	75+56	ND	30	88
chrysene	ND	25	--	--	--	ND	100	105	105	75+26	ND	30	50
dibenzo (a,h) anthracene	ND	25	--	--	--	ND	100	140	140	70+80	ND	30	43
isophorone	ND	10	--	--	--	ND	100	87	87	77+44	ND	30	95
hexachloroethane	ND	25	--	--	--	ND	100	95	95	52+52	ND	30	24
hexachlorocyclopentadiene	ND	25	12	13	8.0	ND	100	88	88	12+24	ND	30	8.0
azobenzene	ND	25	--	--	--	ND	100	74	74	63+110	ND	30	88
acenaphthalene	ND	25	--	--	--	ND	100	66	66	82+46	ND	30	70
hexachlorobutadiene	ND	25	--	--	--	ND	100	63	63	48+56	ND	30	60
phenol	ND	25	--	--	--	ND	100	62	62	70+40	ND	100	44
2-chlorophenol	ND	25	--	--	--	ND	100	70	70	70+23	ND	100	32
2,4-dichlorophenol	ND	25	--	--	--	ND	100	44	44	74+24	ND	100	80
2,4,6-trichlorophenol	ND	25	--	--	--	ND	100	23	23	77+20	ND	100	75
pentachlorophenol	ND	25	--	--	--	ND	100	80	80	40+26	ND	100	66
4-chloro-3-methylphenol	ND	25	--	--	--	ND	100	55	55	50+29	ND	100	87
2,4-dimethylphenol	ND	25	--	--	--	ND	100	75	75	64+25	ND	100	50
2-methyl-4,6-dinitrophenol	ND	250	--	--	--	ND	300	200	67	83+18	ND	300	54
2-nitrophenol	60	25	60	59	1.7	ND	100	66	66	75+25	ND	100	33
4-nitrophenol	ND	25	--	--	--	ND	100	5	5	41+20	ND	100	84
2,4-dinitrophenol	ND	250	--	--	--	ND	300	350	350	78+21	ND	300	75
Units	(ppb)	(ppb)	(ppb)	(ppb)		(ppb)	(ppb)	(ppb)			(ppb)	(ug)	

Client/Project XYZ CORP.
 Client Designation # XYZ-1 Sample Matrix Aqueous
 S-R Sample # ABCD-1 Time 1030 to 1115

Analysis Volatiles
 Date Completed 6/25/84
 Duplicate Sample No. ABCD-2

PARAMETER	RESULTS		QC REPLICATE			QC BLANK	STDS/CAL. CHECK				QC MATRIX SPIKE		
	CONCENTRATION		CONCENTRATION			BLANK	TRUE VALUE	REPORTED	% REC'Y	CONTROL LIMIT	SAMPLE	SPIKE ADDED	% REC'Y
	SAMPLE	MDL	FIRST	SECOND	% RPD								
chloromethane	ND	10	--	--	--	ND	50	47	94	111+76	ND	100	87
bromomethane	ND	10	--	--	--	ND	50	50	100	94+32	ND	100	88
dichlorodifluoromethane	ND	10	--	--	--	ND	50	51	102	88+24	ND	100	85
vinyl chloride	ND	10	--	--	--	ND	50	48	96	108+47	ND	100	42
chloroethane	ND	10	--	--	--	ND	50	43	86	98+27	ND	100	90
methylene chloride	ND	10	--	--	--	ND	50	35	70	101+23	ND	100	82
trichlorofluoromethane	ND	10	--	--	--	ND	50	60	120	95+34	ND	100	110
ethene, 1,1-dichloro	ND	10	--	--	--	ND	50	47	94	100+28	ND	100	79
ethane, 1,1-dichloro	ND	10	18	19	5.4	ND	50	39	78	97+26	ND	100	96
1,2-trans-dichloroethene	ND	10	--	--	--	ND	50	52	104	118+38	ND	100	95
chloroform	ND	10	--	--	--	ND	50	58	116	107+22	ND	100	97
ethane, 1,2-dichloro-	ND	10	--	--	--	ND	50	38	76	113+37	ND	100	98
ethane, 1,1,1-trichloro-	ND	10	--	--	--	ND	50	42	84	108+27	ND	100	110
carbon tetrachloride	ND	10	--	--	--	ND	50	41	82	97+29	ND	100	105
bromodichloromethane	ND	10	--	--	--	ND	50	55	110	110+21	ND	100	85
propane, 1,2-dichloro-	ND	10	--	--	--	ND	50	43	86	103+21	ND	100	93
1,3-trans-dichloropropene	ND	10	20	15	28	ND	50	44	88	106+14	ND	100	92
trichloroethylene	ND	10	--	--	--	ND	50	58	116	99+30	ND	100	99
chlorodibromomethane	ND	10	--	--	--	ND	50	50	100	109+34	ND	100	76
benzene	ND	10	--	--	--	ND	50	37	74	105+17	ND	100	110
ethane, 1,1,2-trichloro-	ND	10	--	--	--	ND	50	56	112	108+22	ND	100	84
1,3-cis-dichloropropene	ND	10	--	--	--	ND	50	47	94	109+23	ND	100	95
2-chloroethyl vinyl ether	ND	10	--	--	--	ND	50	44	88	109+19	ND	100	78
bromoform	ND	10	--	--	--	ND	50	50	100	116+39	ND	100	92
ethane, 1,1,2,2-tetrachloro-	ND	10	--	--	--	ND	50	47	94	111+26	ND	100	120
ethene, tetrachloro-	ND	10	--	--	--	ND	50	48	96	100+29	ND	100	77
toluene	ND	10	--	--	--	ND	50	51	102	98+39	ND	100	105
chlorobenzene	ND	10	--	--	--	ND	50	40	80	93+40	ND	100	88
ethylbenzene	ND	10	--	--	--	ND	50	41	82	100+43	ND	100	87
acrolein	ND	100	--	--	--	ND	200	140	70	75+24	ND	200	65
acrylonitrile	ND	100	--	--	--	ND	200	235	118	91+45	ND	200	87
Units	(ppb)	(ppb)	(ppb)	(ppb)		(ppb)	(ppb)	(ug)			(ppb)	(ug)	

QUANTITATIVE RESULTS AND QUALITY ASSURANCE DATA

Client/Project XYZ CORP. Analysis Pesticides/PCBs

Client Designation # XYZ -1 Sample Matrix Aqueous Date Completed 7/15/ 84

S-R Sample # ABCD-1 Time 1540 to 1635 Duplicate Sample No. ABCD-2

PARAMETER	RESULTS		QC REPLICATE			QC BLANK	STDS/CAL. CHECK				QC MATRIX SPIKE		
	CONCENTRATION		CONCENTRATION			BLANK	TRUE VALUE	REPORTED	% REC'Y	CONTROL LIMIT	SAMPLE	SPIKE ADDED	% REC'Y
	SAMPLE	MDL	FIRST	SECOND	% RPD								
Aldrin	ND	1.0	--	--	--	ND	25.7	16	62	92+81	ND	25.7	85
alpha BHC	ND	1.0	--	--	--	ND	12.8	14	110	77+49	ND	12.8	91
beta BHC	ND	5.0	--	--	--	ND	5.9	5.6	95	94+94	ND	5.9	44
gamma BHC	ND	4.0	--	--	--	ND	64	62	98	89+47	ND	14	85
delta BHC	ND	5.0	--	--	--	ND	70	66	94	84+30	ND	70	87
Chlordane	ND	10	--	--	--	ND	85	75	88	80+40	ND	85	65
Dieldrin	ND	5.0	--	--	--	ND	61.4	45	73	84+47	ND	61.4	70
p,p'-DDD	ND	5.0	--	--	--	ND	61.1	45	74	87+33	ND	61.1	88
p,p'-DDE	ND	5.0	--	--	--	ND	61.1	60	98	87+30	ND	61.7	76
p,p'-DDT	ND	5.0	--	--	--	ND	17	15	113	91+30	ND	17	65
Endosulfan I	ND	10	--	--	--	ND	87	90	97	80+20	ND	87	45
Endosulfan II	ND	10	--	--	--	ND	95	90	106	81+23	ND	95	99
Endosulfan Sulfate	ND	10	--	--	--	ND	24.3	23	106	95+32	ND	24.3	108
Endrin	ND	0.4	--	--	--	ND	61.5	60	98	90+32	ND	61.5	87
Endrin Aldehyde	ND	10	--	--	--	ND	12.8	11.2	87	85+82	ND	12.8	47
Heptachlor	ND	1.0	--	--	--	ND	23	19.5	70	81+50	ND	23	58
Heptachlor Epoxide	ND	5.0	--	--	--	ND	18	16	89	79+30	ND	18	65
Toxaphene	ND	5.0	--	--	--	ND	17.6	17.4	99	95+34	ND	17.6	99
rochlor 1016	ND	5.0	--	--	--	ND	17.9	18.7	104	95+34	ND	17.9	66
rochlor 1221	ND	5.0	--	--	--	ND	84	80	95	95+34	ND	84	87
rochlor 1232	ND	5.0	--	--	--	ND	91	92	101	95+34	ND	91	45
rochlor 1242	ND	5.0	--	--	--	ND	15.7	13	83	95+34	ND	15.7	105
rochlor 1248	ND	5.0	--	--	--	ND	85.3	81	95	95+34	ND	85.3	110
rochlor 1254	ND	5.0	8.5	8.9	4.6	ND	44	42	105	95+34	ND	44	105
rochlor 1260	ND	5.0	--	--	--	ND	56	50	114	95+34	ND	56	88
Units	(ppb)	(ppb)	(ppb)	(ppb)		(ppb)	(ppb)	(ppb)			(ppb)	(ppb)	

1987 001 NXS

SUMMARY OF NBS (38,700+ analyte version, April '82)

LIBRARY SEARCH RESULTS OF NONTARGETED PEAKS WITH
ESTIMATED CONCENTRATION OF TENTATIVELY IDENTIFIED COMPOUNDSANALYTICAL FRACTION: ACID EXTRACTABLE

DATA FILE NAME: ABCD1

SAMPLE # ABCD-1

ITEM	SCAN NUMBER	COMPOUND NAME/m.w.	MATCH FACTOR FIT	ASSESSMENT ¹			ESTIMATED ² CONC. (ug/kg)
				RS	ISO	UK	
1	1975	1-Ethenyl-3-methylene-					
2		cyclopentene	859	X			22
3	879	1,3,5-Trimethyl Benzene	904		X		1.2
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							

SPECTROSCOPIST _____

DATE 5/8/84

- (1) RS - Reasonable Identification
 ISO - Isomer or Similar Compound
 UK - Unknown, not in NBS Library

- (2) Calculated versus nearest eluting internal standard as a sample ratio/proportion

SYN 101 1988

SUMMARY OF NBS (38,700+ analyte version, April '82)

LIBRARY SEARCH RESULTS OF NONTARGETED PEAKS WITH
ESTIMATED CONCENTRATION OF TENTATIVELY IDENTIFIED COMPOUNDSANALYTICAL FRACTION: BASE/NEUTRAL

DATA FILE NAME: ABCD-1

SAMPLE #: ABCD-1

ITEM	SCAN NUMBER	COMPOUND NAME/m.w.	MATCH FACTOR FIT	ASSESSMENT ¹			ESTIMATED ² CONC. (ug/kg)
				RS	ISO	UK	
1							
2		1,1,2-Trichloro-1,2,2-					
3	650	Trifluoroethane	910	X			15.8
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							

SPECTROSCOPIST _____

DATE 6/25/84

- (1) RS - Reasonable Identification
 ISO - Isomer or Similar Compound
 UK - Unknown, not in NBS Library

- (2) Calculated versus nearest eluting internal standard as a simple ratio/proportion

SYN 001 1989

III QUANTITATIVE RESULTS AND QUALITY ASSURANCE DATA

B. INORGANIC DATA

SYN 001 1990

Initial Calibration Data and Calibration Check - Metals

Lab Sample No. ABCD-1Instrument IL Video 22 Calibration Date 8/15/84

ELEMENTS	Initial Calibration Data				Calibration Check		
	Control Limit	Actual Value	Found	% RSD	Actual Value	Found	% RSD
Antimony	84+76	1,000	850	16	1,000	950	5.1
Arsenic	94+19	1,000	870	14	1,000	870	14
Barium	106+24	1,000	930	7.3	1,000	990	1.0
Beryllium	91+18	1,000	940	6.2	1,000	880	13
Cadmium	98+11	1,000	990	1.0	1,000	790	23
Chromium	107+22	1,000	1,000	0	1,000	850	16
Copper	96+14	1,000	870	14	1,000	950	5.1
Lead	101+19	1,000	910	9.4	1,000	870	14
Mercury	97+28	10	10.6	5.8	10	8.7	14
Nickel	94+22	1,000	990	1.0	1,000	950	5.1
Selenium	89+24	1,000	850	16	1,000	930	7.3
Silver	84+52	1,000	910	9.4	1,000	980	2.0
Thallium	92+24	1,000	900	10	1,000	920	8.3
Zinc	36+14	1,000	890	12	1,000	890	12
Units		(ppb)	(ppb)		(ppb)	(ppb)	

SYN 001 1991

Initial Calibration Data and Calibration Check - Cyanide and Phenol

Lab Sample No. ABCD-1Instrument PE 550 UV/VIS Spec Calibration Date 6/11/84

ELEMENTS	Initial Calibration Data				Calibration Check		
	Control Limit	Actual Value	Found	% RSD	Actual Value	Found	% RSD
Cyanide	90 + 29	1,000	990	1.0	20	18	10
Phenol	99 + 21	2,000	1,890	5.6	40	41	2.5
Units		(ppb)			(ppb)	(ppb)	

SYN 001 1992

QUANTITATIVE RESULTS AND QUALITY ASSURANCE DATA

Client/Project XYZ CORP. Analysis Heavy Metals

Client Designation # XYZ-1 Sample Matrix Aqueous Date Completed 8/30/84

S-R Sample # ABCD-1 Time 1010 to 1950 Duplicate Sample No. ABCD-1

Spike Sample No. ABCD-4

PARAMETER	RESULTS		QC REPLICATE			QC BLANK	STDS/CAL. CHECK				QC MATRIX SPIKE		
	CONCENTRATION		CONCENTRATION			BLANK	TRUE VALUE	REPORTED	Z REC'Y	CONTROL LIMIT	SAMPLE	SPIKE ADDED	Z REC'Y
	SAMPLE	MDL	FIRST	SECOND	% RPD								
Antimony	ND	2.0	--	--	--	ND	100	97	97	84+76	ND	500	87
Arsenic	ND	2.0	--	--	--	ND	100	85	85	94+19	ND	500	88
Barium	ND	88	--	--	--	ND	100	79	79		ND	500	89
Beryllium	ND	10	--	--	--	ND	100	100	100	91+18	ND	500	96
Cadmium	ND	4.0	--	--	--	ND	100	105	105	98+11	ND	500	95
Chromium	51	26	51	56	9.3	ND	100	95	95	107+22	28	500	76
Copper	75	50	75	73	2.7	ND	100	96	96	96+14	ND	500	95
Lead	87	43	87	80	8.4	ND	100	90	90	101+19	ND	500	105
Mercury	ND	1.0	--	--	--	ND	10	8.5	8.5	97+28	ND	10	98
Nickel	ND	36	--	--	--	ND	100	95	95	94+22	ND	500	110
Selenium	ND	1.0	--	--	--	ND	100	76	76	89+24	ND	500	87
Silver	ND	9.0	--	--	--	ND	100	85	85	84+52	ND	500	95
Thallium	ND	100	--	--	--	ND	100	96	96	92+24	ND	500	108
Zinc	18	10	18	23	24	ND	100	99	99	86+14	25	500	87
Units	(ppb)	(ppb)	(ppb)	(ppb)		(ppb)	(ppb)	(ppb)			(ppb)	(ug)	

1996T 100 WAS

QUANTITATIVE RESULTS AND QUALITY ASSURANCE DATA

Client/Project XYZ CORP.
 Client Designation # XYZ -1 Sample Matrix Aqueous
 S-R Sample # ABCD-1 Time 1215 to 1320

Analysis Cyanide, Phenol
 Date Completed 6/11/84
 Duplicate Sample No. ABCD-2
 Spike Sample No. ABCD-4

PARAMETER	RESULTS		QC REPLICATE			QC BLANK	STDS/CAL. CHECK				QC MATRIX SPIKE		
	CONCENTRATION		CONCENTRATION										
	SAMPLE	MDL	FIRST	SECOND	% RPD	BLANK	TRUE VALUE	REPORTED	% REC'Y	CONTROL LIMIT	SAMPLE	SPIKE ADDED	% REC'Y
Cyanide	ND	10	--	--	--	ND	50	48	96	90+29	ND	25	90
Phenol	11	10	11	13	17	ND	100	110	110	99+21	ND	30	87
Units	(ppb)	(ppb)	(ppb)	(ppb)		(ppb)	(ppb)	(ppb)			(ug)	(ug)	

IV SUPPORTING DOCUMENTS
A. DFTPP TUNE PRINTOUTS

MID. Mass. List

06/25/84 11:30:00 + 10:09

Data: DFTPP625 # 720

Cali: DFTPP625 # 2

Base m/z: 198

RIC: 36765600.

Sample: DFTPP 6/25/84

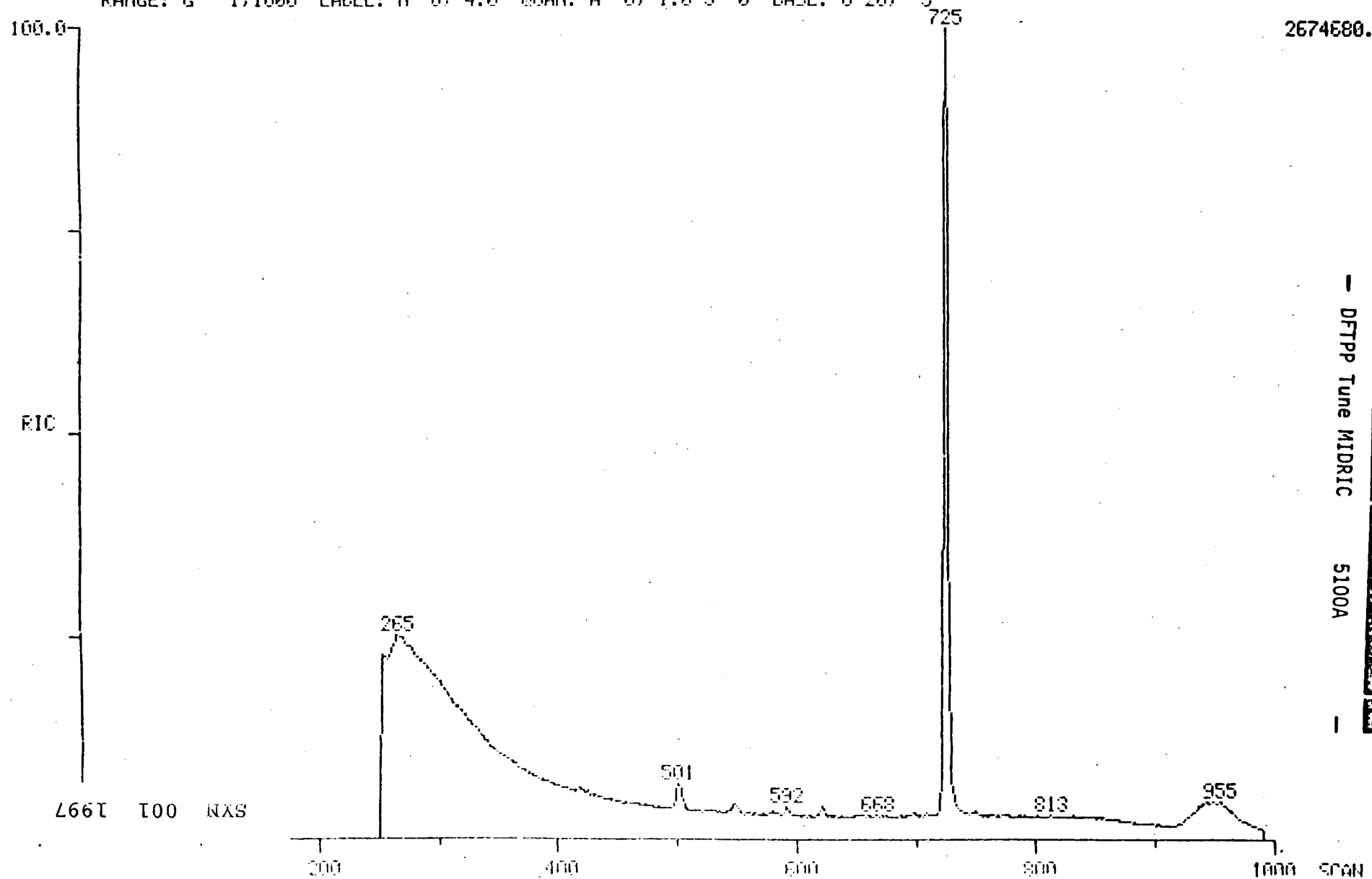
ds.: EI

#719 to #722 Summed

45 0.00 Minima		Min inten: 10304.					
445 #	0 Maxima						
Mass	% RA	Mass	% RA	Mass	% RA	Mass	% RA
50	11.15	122	0.92	178	0.35	245	1.01
51	41.67	123	1.59	179	3.16	246	1.48
52	2.14	124	0.71	180	2.24	247	0.33
56	0.99	125	0.79	181	1.13	249	0.33
57	2.50	127	57.62	184	0.32	253	0.30
61	0.46	128	4.46	185	1.66	255	35.64
62	0.55	129	22.41	186	13.23	256	5.22
63	1.59	130	1.95	187	3.89	257	0.44
64	0.23	131	1.09	188	0.45	258	1.96
65	0.80	132	0.26	189	0.67	259	0.34
69	45.57	133	0.26	191	0.50	265	0.81
70	0.29	134	0.61	192	1.05	273	1.13
73	0.51	135	1.94	193	1.16	274	3.46
74	3.94	136	0.81	194	0.30	275	16.73
75	6.48	137	1.02	196	2.41	276	2.26
76	2.38	138	0.25	198	100.00	277	1.53
77	47.16	140	0.27	199	6.74	278	0.27
78	3.43	141	2.38	200	0.51	281	0.27
79	2.78	142	1.00	201	0.56	285	0.28
80	2.22	143	0.74	203	0.54	293	0.40
81	3.56	145	0.23	204	2.73	296	4.18
82	0.94	146	0.46	205	4.63	297	0.59
83	0.90	147	1.45	206	19.22	303	0.53
85	1.49	148	2.66	207	3.34	314	0.26
86	0.92	149	0.68	208	0.79	315	0.51
87	0.51	150	0.27	209	0.33	316	0.31
88	0.31	151	0.43	210	0.41	323	1.45
91	0.85	152	0.26	211	1.02	324	0.31
92	0.82	153	0.83	215	0.26	327	0.32
93	5.38	154	0.66	216	0.45	334	0.97
94	0.42	155	1.46	217	4.91	335	0.31
96	0.43	156	2.26	218	0.67	346	0.34
98	4.27	157	0.51	219	0.55	352	0.47
99	3.52	158	0.47	221	4.21	353	0.37
100	0.53	159	0.40	223	1.12	354	0.63
101	2.11	160	0.81	224	9.67	365	2.01
103	0.67	161	1.35	225	2.50	366	0.32
104	1.19	162	0.42	226	0.32	372	0.86
105	1.25	165	0.84	227	4.07	373	0.26
106	0.49	166	0.86	228	0.64	383	0.25
107	17.51	167	5.39	229	0.85	402	0.39
108	2.56	168	2.68	231	0.39	403	0.48
110	35.82	169	0.51	234	0.27	421	0.43
111	5.14	171	0.23	235	0.33	422	0.50
112	0.66	172	0.40	236	0.28	423	3.10
113	0.28	173	0.57	237	0.31	424	0.65
116	0.84	174	1.07	241	0.26	441	7.89
117	12.72	175	1.82	242	0.50	442	51.15
118	1.00	176	0.57	243	0.56	443	9.65
119	0.37	177	0.96	244	7.18	444	0.95

MIDRIC
10/16/84 12:25:00
SAMPLE: DFTPP 10/16/84
CONDOS.: EI
RANGE: G 1.1000 LABEL: N 0. 4.0 QUAN: A 0. 1.0 J 0 BASE: U 20. 3

DATA: DFTPP1016 #724 SCANS 1 TO 1000
CALI: DFTPP1016 #2



MID Mass List
06/01/84. 8:17:00 + 10:09
Sample: DFTPP 6/1/84
Conds.: EI

Data: DFTPP601 # 719
Call: DFTPP601 # 2

Base m/z: 198
RIC: 6111230.

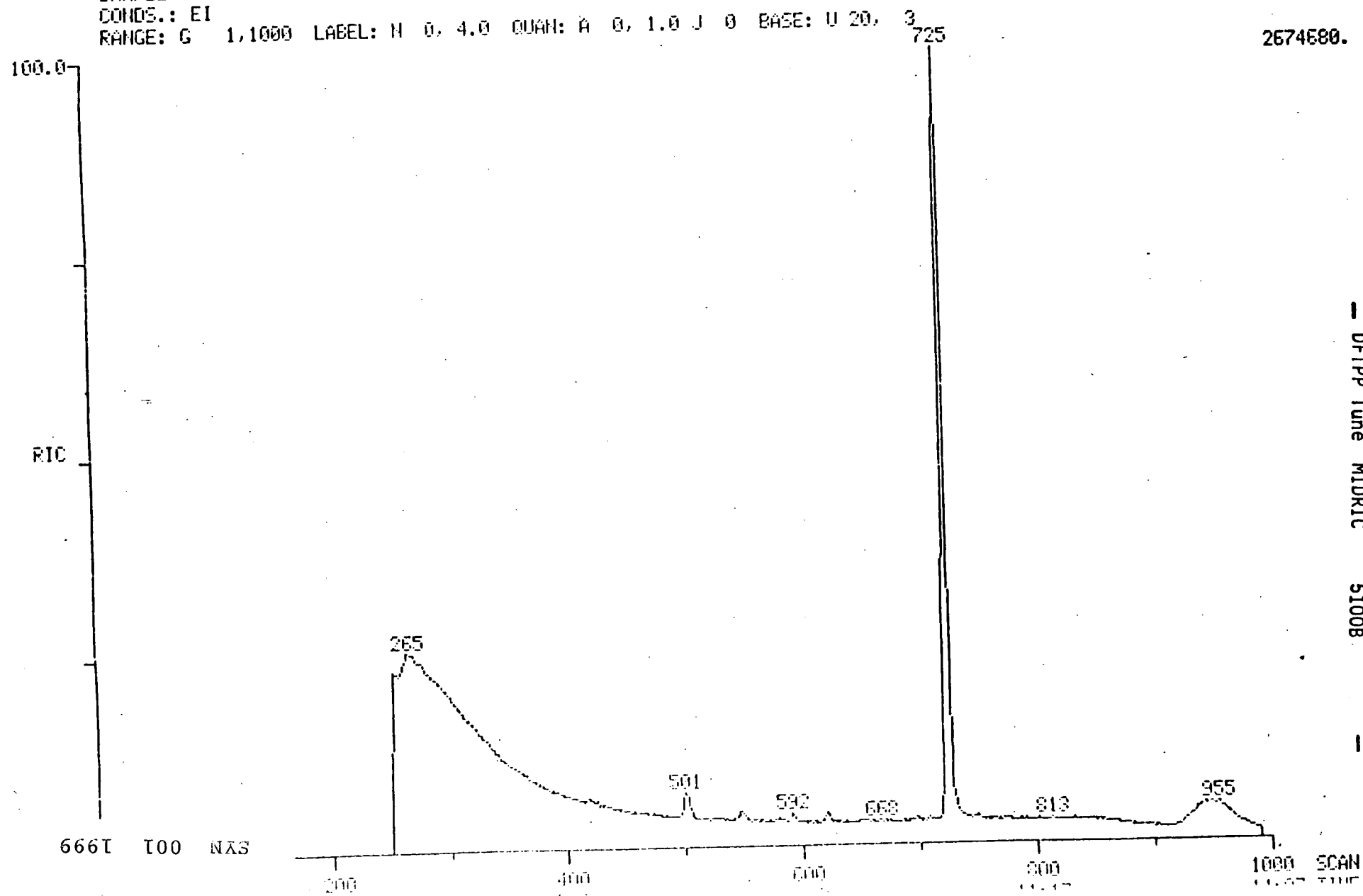
DFTPP Tune Mass List 5100B

45	0.00 Minima	Min inten: 205					
445 #	0 Maxima						
Mass	% RA	Mass	% RA	Mass	% RA	Mass	% RA
47	1.46	117	13.34	185	1.63	249	0.45
48	0.78	118	1.02	186	12.05	253	0.55
49	4.34	122	0.79	187	3.73	255	49.40
50	16.70	123	1.47	188	0.46	256	7.45
51	52.09	124	0.67	189	0.70	257	0.58
52	2.72	125	0.76	191	0.46	258	2.65
55	0.40	127	44.70	192	1.25	259	0.46
56	1.15	128	3.60	193	1.03	265	1.11
57	2.54	129	19.51	194	0.35	271	0.36
61	0.53	130	1.88	195	0.31	272	0.35
62	0.72	131	0.44	196	2.37	273	1.57
63	1.57	134	0.65	198	100.00	274	4.81
65	0.74	135	1.69	199	6.60	275	26.34
69	45.30	136	0.70	200	0.59	276	3.78
70	0.36	137	0.74	201	0.48	277	1.97
73	0.54	140	0.32	202	0.31	278	0.40
74	4.67	141	2.44	203	0.76	285	0.42
75	7.11	142	0.83	204	3.09	293	0.52
76	2.92	143	0.64	205	5.58	296	5.43
77	42.31	146	0.49	206	22.99	297	0.76
78	2.81	147	1.36	207	3.26	303	0.72
79	2.69	148	3.01	208	0.75	314	0.45
80	2.36	149	0.70	209	0.38	315	0.71
81	3.15	151	0.43	210	0.53	316	0.49
82	0.91	152	0.32	211	1.12	321	0.32
83	0.73	153	0.76	212	0.31	323	2.06
84	2.05	154	0.66	215	0.32	324	0.46
85	0.58	155	1.57	216	0.60	327	0.49
86	1.97	156	2.05	217	5.95	334	1.41
87	0.50	157	0.48	218	0.81	335	0.43
88	0.49	158	0.54	221	3.06	341	0.35
91	0.85	159	0.48	222	0.94	346	0.44
92	0.89	160	0.82	223	1.53	352	0.64
93	5.68	161	1.29	224	11.88	353	0.51
94	0.55	162	0.42	225	3.07	354	0.89
96	0.21	165	0.85	226	0.47	365	2.76
98	4.56	166	0.90	227	5.00	366	0.44
99	3.15	167	4.76	228	0.73	372	1.33
100	0.44	168	2.61	229	1.05	373	0.46
101	1.96	169	0.49	231	0.55	383	0.39
103	0.50	172	0.46	234	0.43	402	0.65
104	1.11	173	0.61	235	0.46	403	0.76
105	1.10	174	1.03	236	0.37	404	0.30
106	0.59	175	1.84	237	0.49	421	0.86
107	14.22	176	0.55	241	0.34	422	0.84
108	2.13	177	0.89	242	0.70	423	5.70
109	0.64	178	0.54	243	0.80	424	1.08
110	21.49	179	3.52	244	10.13	441	15.37
111	3.56	180	2.32	245	1.32	442	92.69
112	0.59	181	1.29	246	1.72	443	17.54
116	1.19	184	0.43	247	0.40	444	1.63

MIDRIC
10/16/84 12:25:00
SAMPLE: DFTPP 10/16/84
CONDOS.: EI
RANGE: G 1,1000 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3

DATA: DFTPP1016 #724 SCANS 1 TO 1000
CALI: DFTPP1016 #2

2674680.



IV SUPPORTING DOCUMENTS

B. GC/MS AND GC CALIBRATION PRINTOUTS

Data: EBSTD5809.TI

08/09/84 19:00:00

Sample:

- CAL BLK

- Semivolatile

Page 30

Prepared by:

Analyst:

AMOUNT=AREA * REF.AMNT/(REF.AREA)* RESP.FACT)

Resp. fac. from Library Entry

NO	NAME
1	D10-ANTHRACENE (I. S.)
2	PHENANTHRENE
3	ACRIDINE
4	ANTHRACENE
5	PYRENE
6	CHRYSENE
7	BENZO-A-PYRENE
8	PHENOL
9	O-CRESOL
10	M, P-CRESOL
11	NAPHTHALENE

No	m/z	Scan	Time	Ref	RRT	Meth	Area(Hght)	Amount	%Tot
1	188	1308	27:47	1	1.000	A BB	174960.	40.000 NG/ML	81.53
2	178	1304	27:42	1	0.997	A BV	32416.	0.736 NG	1.50
3	179	1318	28:00	1	1.008	A BB	17360.	0.957 NG	1.95
4	178	1312	27:52	1	1.003	A VV	32016.	2.217 NG	4.52
5	NOT FOUND								
6	228	1849	39:16	1	1.414	A BB	26768.	1.469 NG	2.99
	NOT FOUND								
	66	552	11:43	1	0.422	A BB	1440.	0.890 NG	1.81
9	77	638	13:33	1	0.488	A BB	1472.	1.423 NG	2.90
10	77	658	13:59	1	0.503	A BB	2080.	0.602 NG	1.23
11	75	796	16:54	1	0.609	A BB	832.	0.769 NG	1.57

SYN 001 2001

MIDRIC
02/09/84 19:00:00

DATA: EBSTD5809 #1
CALI: EBSTD5809 #2

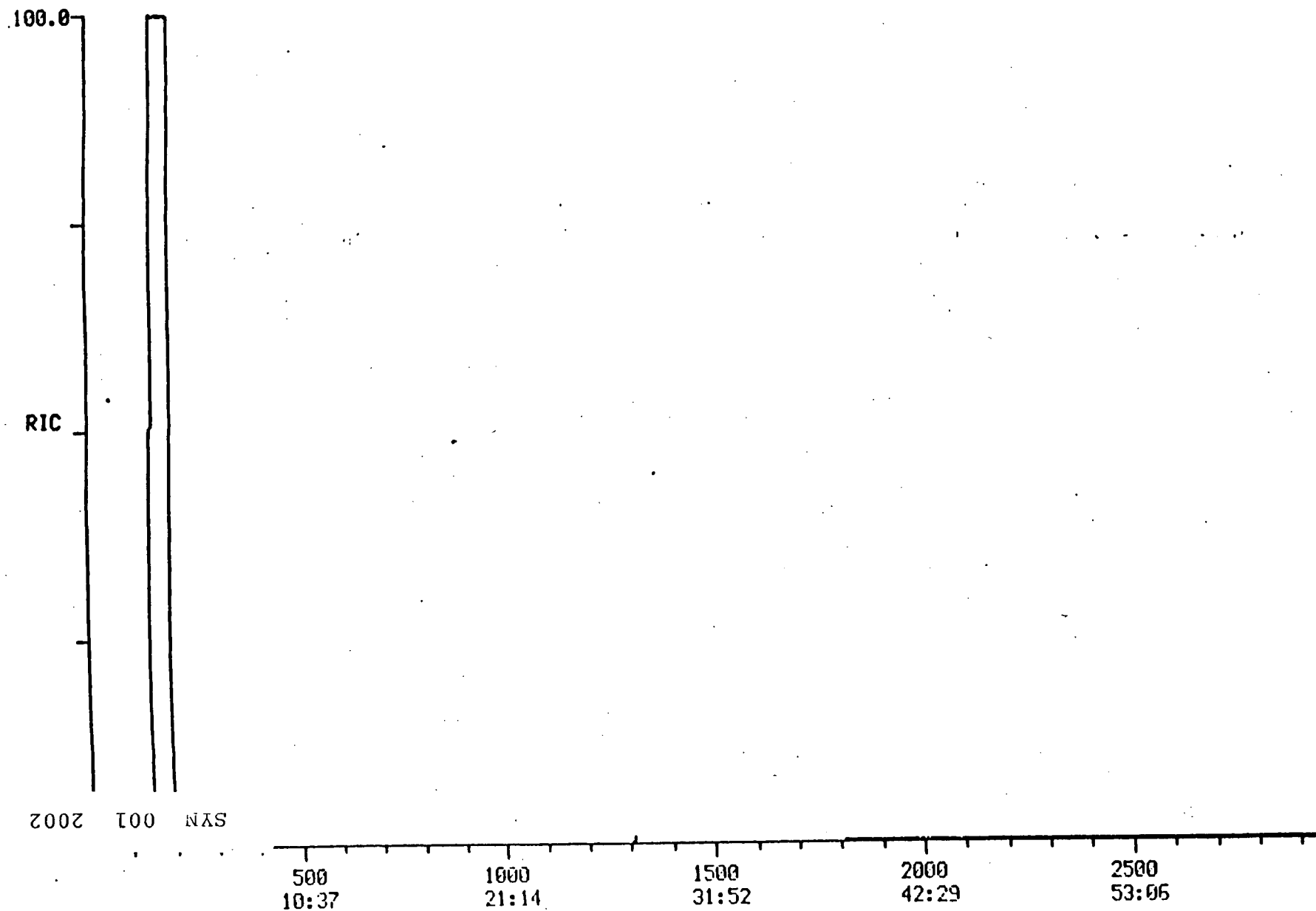
SCANS 1 TO 2950

SAMPLE:

COND5.:

RANGE: G 1, 3 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3

16760800.



Data: EBSTD10809.TI
08/09/84 17:49:00

- CAL STD No. 1 - Semivolatile

Page 32

Sample:

Submitted by:

Analyst:

AMOUNT=AREA * REF.AMNT/(REF.AREA)* RESP.FACT)

Resp. fac. from Library Entry

NO NAME

- 1 D10-ANTHRACENE (I. S.)
- 2 PHENANTHRENE
- 3 ACRIDINE
- 4 ANTHRACENE
- 5 PYRENE
- 6 CHRYSENE
- 7 BENZO-A-PYRENE
- 8 PHENOL
- 9 O-CRESOL
- 10 M, P-CRESOL
- 11 NAPHTHALENE

No	m/z	Scan	Time	Ref	RRT	Meth	Area(Hght)	Amount	%Tot
1	188	1311	27:51	1	1.000	A BB	206944.	40.000 NG/ML	60.15
2	178	1306	27:44	1	0.996	A BV	85760.	1.645 NG	2.47
3	179	1321	28:04	1	1.008	A VB	47680.	2.223 NG	3.34
4	178	1314	27:55	1	1.002	A VV	76416.	4.474 NG	6.73
5	100	1580	33:34	1	1.205	A BB	1120.	1.495 NG	2.25
6	228	1880	39:56	1	1.434	A BB	54304.	2.519 NG	3.79
7	252	2415	51:18	1	1.842	A BV	33392.	5.288 NG	7.95
	66	556	11:49	1	0.424	A BB	5360.	2.801 NG	4.21
	77	640	13:36	1	0.488	A BB	3472.	2.838 NG	4.27
10	77	660	14:01	1	0.503	A BB	7840.	1.919 NG	2.89
11	75	797	16:56	1	0.608	A BB	1664.	1.300 NG	1.95

SYN 001 2003

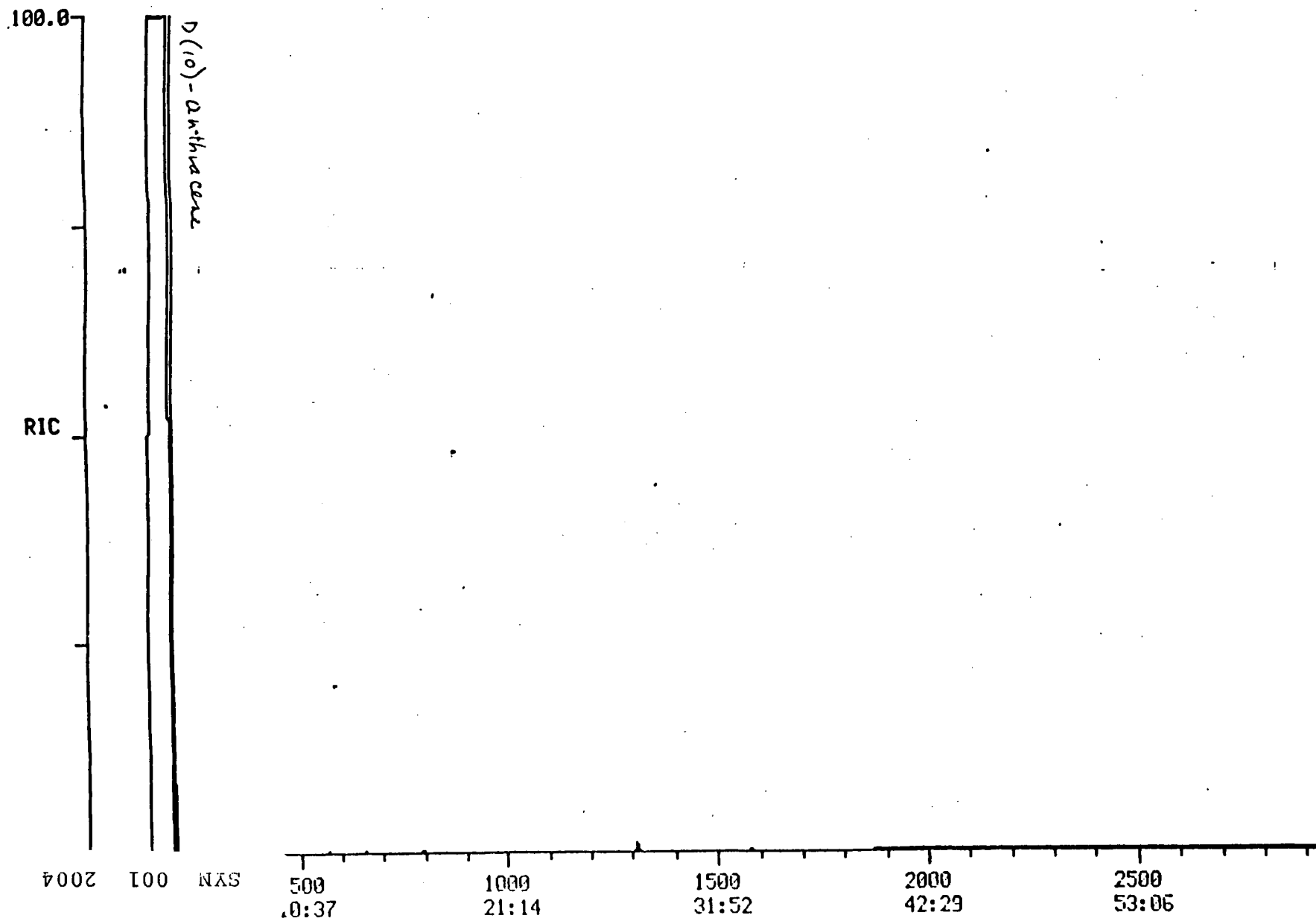
MIDRIC
08/09/84 17:49:00
SAMPLE:
CONDS.:
RANGE: G 1, 3

DATA: EBSTD10309 #1
CALI: EBSTD10809 #2

SCANS 1 TO 2950

LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3

16760800.



Data: EBSTD20809.TI

08/09/84 21:24:00

Sample:

Analyzed by:

Analyst:

AMOUNT=AREA * REF.AMNT/(REF.AREA)* RESP.FACT)

Resp. fac. from Library Entry

NO NAME
1 D10-ANTHRACENE (I.S.)
2 PHENANTHRENE
3 ACRIDINE
4 ANTHRACENE
5 PYRENE
6 CHRYSENE
7 BENZO-A-PYRENE
8 PHENOL
9 O-CRESOL
10 M, P-CRESOL
11 NAPHTHALENE

No	m/z	Scan	Time	Ref	RRT	Meth	Area(Hght)	Amount	%Tot
1	188	1306	27:44	1	1.000	A BV	376384.	40.000 NG/ML	12.53
2	178	1302	27:39	1	0.997	A BV	1601120.	16.889 NG	5.29
3	179	1315	27:56	1	1.007	A VB	958020.	24.557 NG	7.69
4	178	1309	27:48	1	1.002	A VV	1542940.	49.671 NG	15.56
5	100	1549	32:54	1	1.186	A*BB	37120.	27.239 NG	8.53
6	228	1822	38:42	1	1.395	A BB	947920.	24.174 NG	7.57
	252	2359	50:06	1	1.806	A BB	450496.	39.226 NG	12.29
	66	557	11:50	1	0.426	A BB	83904.	24.109 NG	7.55
9	77	640	13:36	1	0.490	A VB	74725.	33.589 NG	10.52
10	77	660	14:01	1	0.505	A BV	178031.	23.960 NG	7.51
11	75	797	16:56	1	0.610	A BB	36688.	15.760 NG	4.94

SYN 001 2005

MIDRIC
08/09/84 21:24:00

DATA: EBSTD20809 #1
CALI: EBSTD20309 #2

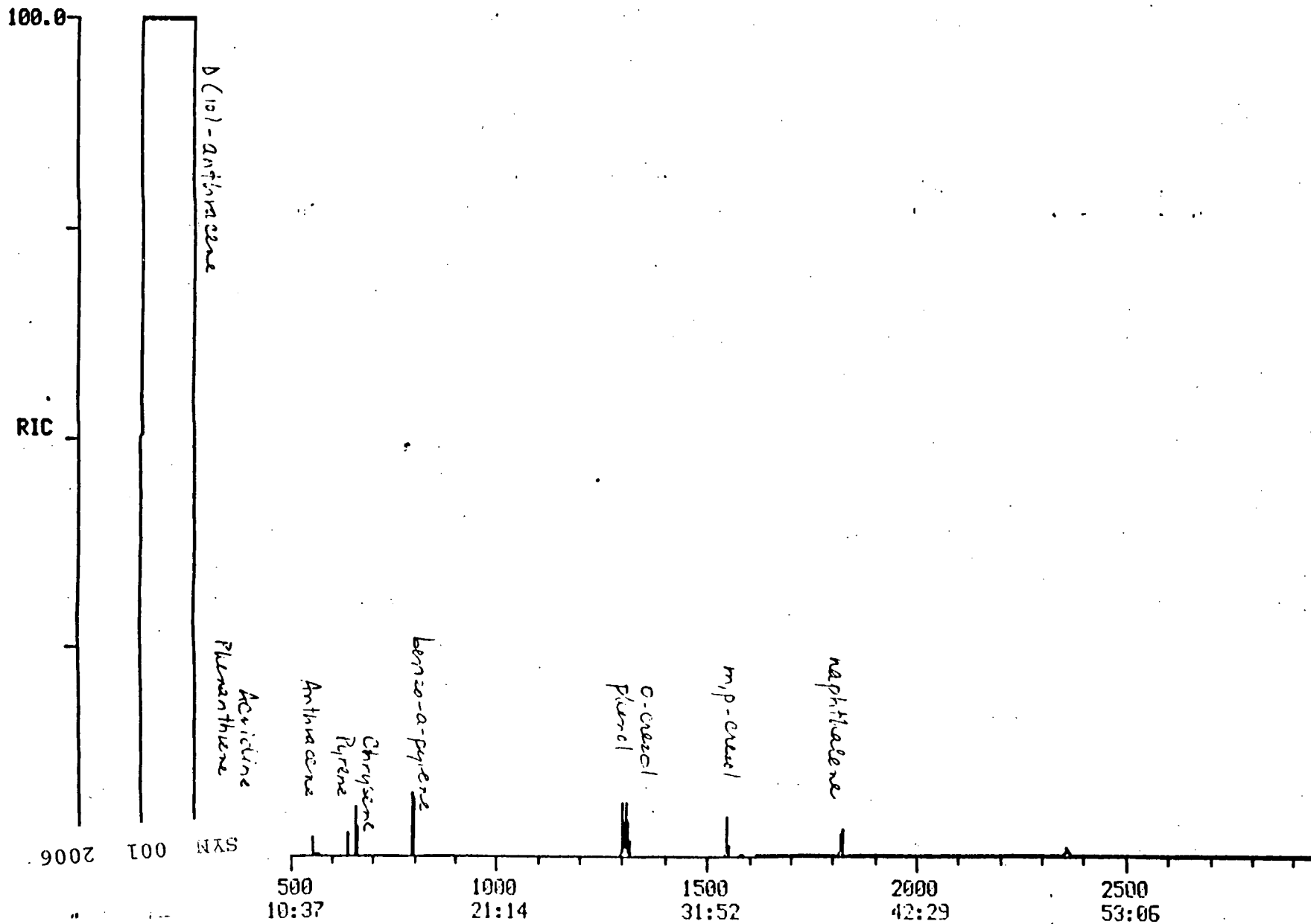
SCANS 1 TO 2950

SAMPLE:
CONDS.:

RANGE: G 1,2950 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3

16760800.

100 FINISHED, 11 FOUND
FINISHED AT: 8/10/84 12:02:37



Data: EBSTD809.T1

08/09/84 16:12:00

Sample: EBASCO STANDARD 8/9/84

Submitted by: SR

Analyst: WAF

- CAL STD No. 3 - Semivolatile -

AMOUNT=AREA * REF. AMNT/(REF. AREA)* RESP. FACT)
Resp. fac. from Library Entry

NO NAME
1 D10-ANTHRACENE (I. S.)
2 PHENANTHRENE
3 ACRIDINE
4 ANTHRACENE
5 PYRENE
6 CHRYSENE
7 BENZO-A-PYRENE
8 PHENOL
9 O-CRESOL
10 M, P-CRESOL
11 NAPHTHALENE

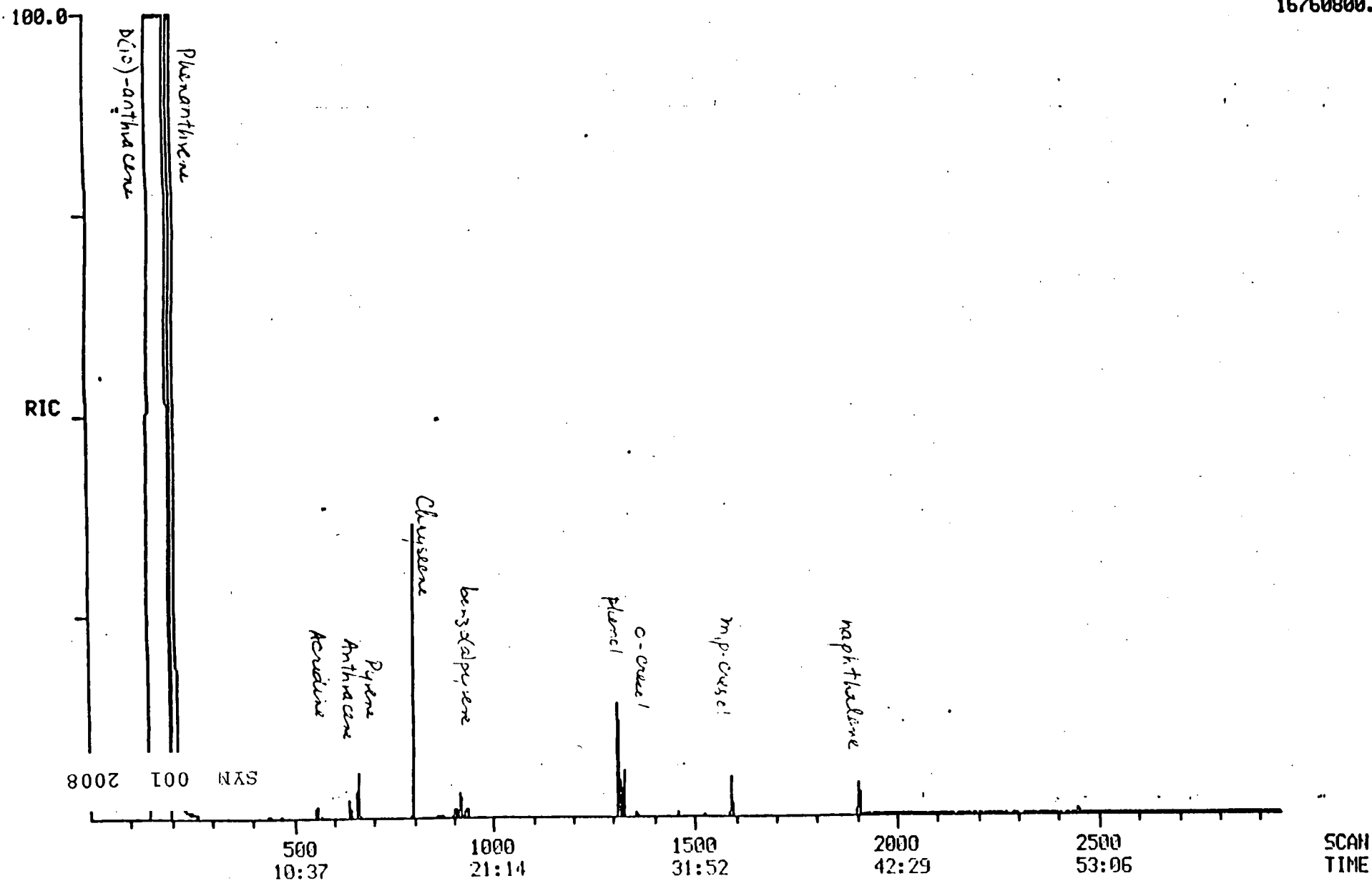
No	m/z	Scan	Time	Ref	RRT	Meth	Area(Hght)	Amount	%Tot
1	188	1315	27:56	1	1.000	A BB	150912.	40.000 NG/ML	4.76
2	178	1311	27:51	1	0.997	A BV	3040840.	80.000 NG	9.52
3	179	1327	28:11	1	1.009	A VB	1251340.	80.000 NG	9.52
4	178	1319	28:01	1	1.003	A VV	996400.	80.000 NG	9.52
5	100	1592	33:49	1	1.211	A BB	43712.	80.000 NG	9.52
6	228	1906	40:29	1	1.449	A BB	1257790.	80.000 NG	9.52
	252	2448	52:00	1	1.862	A BB	368387.	80.000 NG	9.52
	66	560	11:54	1	0.426	A BB	111632.	80.000 NG	9.52
9	77	642	13:38	1	0.488	A BB	71360.	80.000 NG	9.52
10	77	663	14:05	1	0.504	A BB	238336.	80.000 NG	9.52
11	75	799	16:58	1	0.608	A BB	74672.	80.000 NG	9.52

MIDRIC
03/09/84 16:12:00
SAMPLE: EBASCO STANDARD 8/9/84
CONDS.: EI
RANGE: G 1,2950 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3

DATA: EBSTD809 #1
CALI: EBSTD809 #2

SCANS 1 TO 2950

16760800.



a: BLANKTB11.TI

11/84 14:55:00

ed by:

Analyst:

JUNT=AREA * REF.AMNT/(REF.AREA)* RESP.FACT)

sp. fac. from Library Entry

J NAME

1 1,4 DICHLOROBUTANE (I. S.)

2 BENZENE

3 TOLUENE

4 O-XYLENE

5 M, P-XYLENE

J	m/z	Scan	Time	Ref	RRT	Meth	Area(Hght)	Amount	%Tot
1	55	351	29:45	1	1.000	A BV	421395.	44.000 NG/ML	99.83
2	78	284	24:04	1	0.809	A BB	21656.	0.021 UG	0.05
3	92	364	30:51	1	1.037	A VB	20820.	0.023 UG	0.05
4	106	481	40:46	1	1.370	A BB	7628.	0.008 UG	0.02
5	91	499	42:17	1	1.422	A VB	29816.	0.021 UG	0.05

MIDRIC
08/11/84 14:55:00

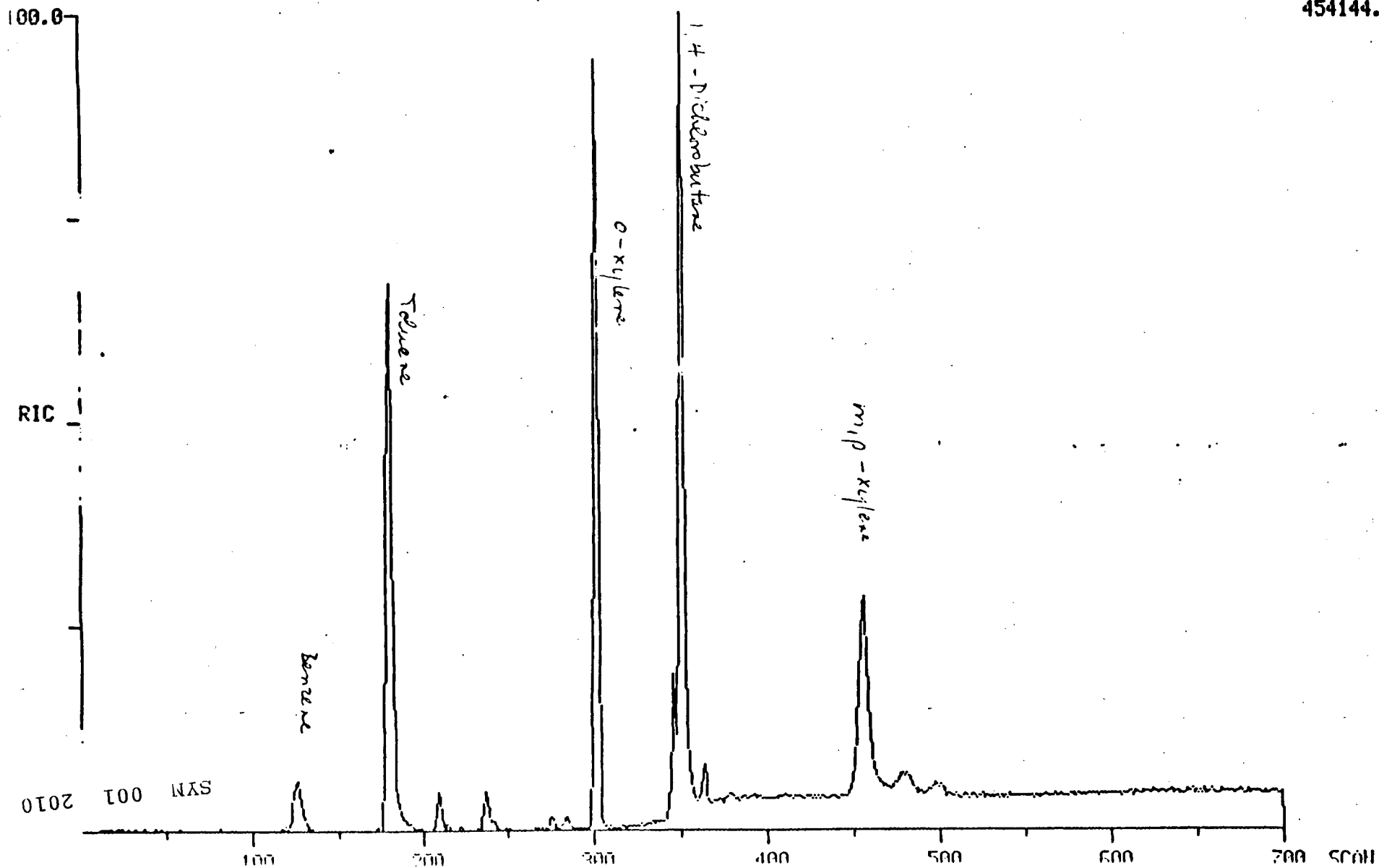
DATA: BLANKT811 #1
CALI: BLANKT811 #2

SCANS 1 TO 700

SAMPLE:
CONDS.:

RANGE: G 1, 1 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3

454144.



Data: EBSTD2T811.TI

- CAL STD No.1 - Volatile

08/11/84 11:11:00

Sample:

ttd by:

Analyst:

AMOUNT=AREA * REF.AMNT/(REF.AREA)* RESP.FACT)

resp. fac. from Library Entry

NO NAME

1 1,4 DICHLOROBUTANE (I. S.)

2 BENZENE

3 TOLUENE

4 O-XYLENE

5 M, P-XYLENE

No	m/z	Scan	Time	Ref	RRT	Meth	Area(Hght)	Amount	%Tot
1	55	351	29:45	1	1.000	A BB	515257.	44.000 NG/ML	93.57
2	78	284	24:04	1	0.809	A BV	747048.	0.585 UG	1.24
3	92	364	30:51	1	1.037	A VB	651888.	0.594 UG	1.26
4	106	482	40:51	1	1.373	A BV	679820.	0.614 UG	1.31
5	91	499	42:17	1	1.422	A VB	2133130.	1.229 UG	2.61

MIDRIC
08/11/84 11:11:00

DATA: ERSTD2T811 #1
CALI: ERSTD2T811 #2

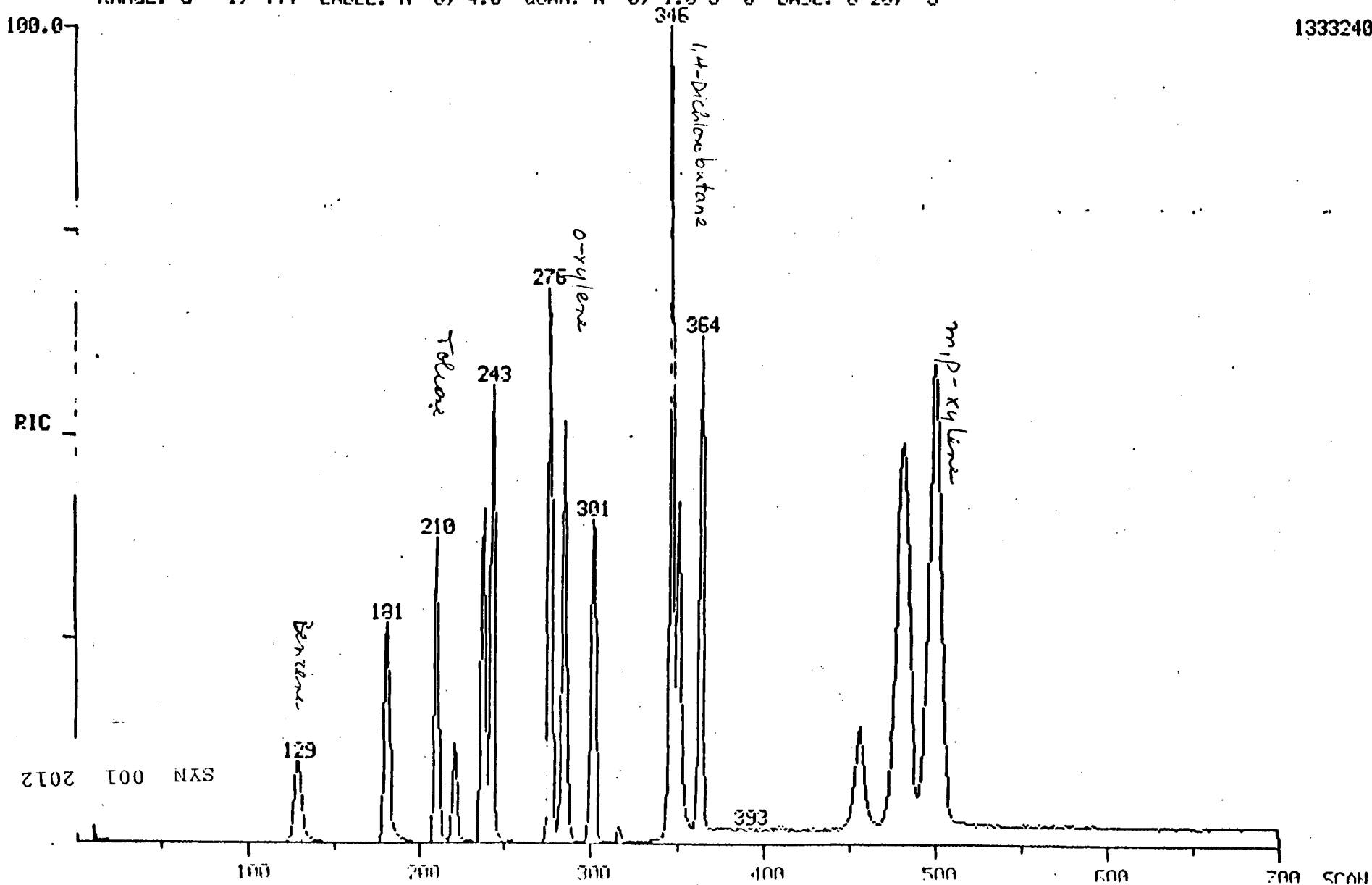
SCANS 1 TO 700

SAMPLE:

CONDS.:

RANGE: G 1, 444 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3

1333240.



ta: EBSTD3T811.TI

/11/84 12:31:00

nple:

ated by:

Analyst:

CAL STD NO. 2 VOLATILE

Page 42

NT=AREA * REF.AMNT/(REF.AREA)* RESP.FACT)

sp. fac. from Library Entry

J NAME

1 1,4 DICHLOROBUTANE (I.S.)

2 BENZENE

3 TOLUENE

4 O-XYLENE

5 M,P-XYLENE

J	m/z	Scan	Time	Ref	RRT	Meth	Area(Hght)	Amount	%Tot
1	55	351	29:45	1	1.000	A BB	581313.	44.000 NG/ML	89.34
2	78	284	24:04	1	0.809	A BV	1509910.	1.047 UG	2.13
3	92	364	30:51	1	1.037	A VB	1312000.	1.059 UG	2.15
4	106	482	40:51	1	1.373	A BV	1291350.	1.034 UG	2.10
5	91	499	42:17	1	1.422	A VB	4136200.	2.112 UG	4.29

SYN 001 2013

MIDRIC
08/11/84 12:31:00

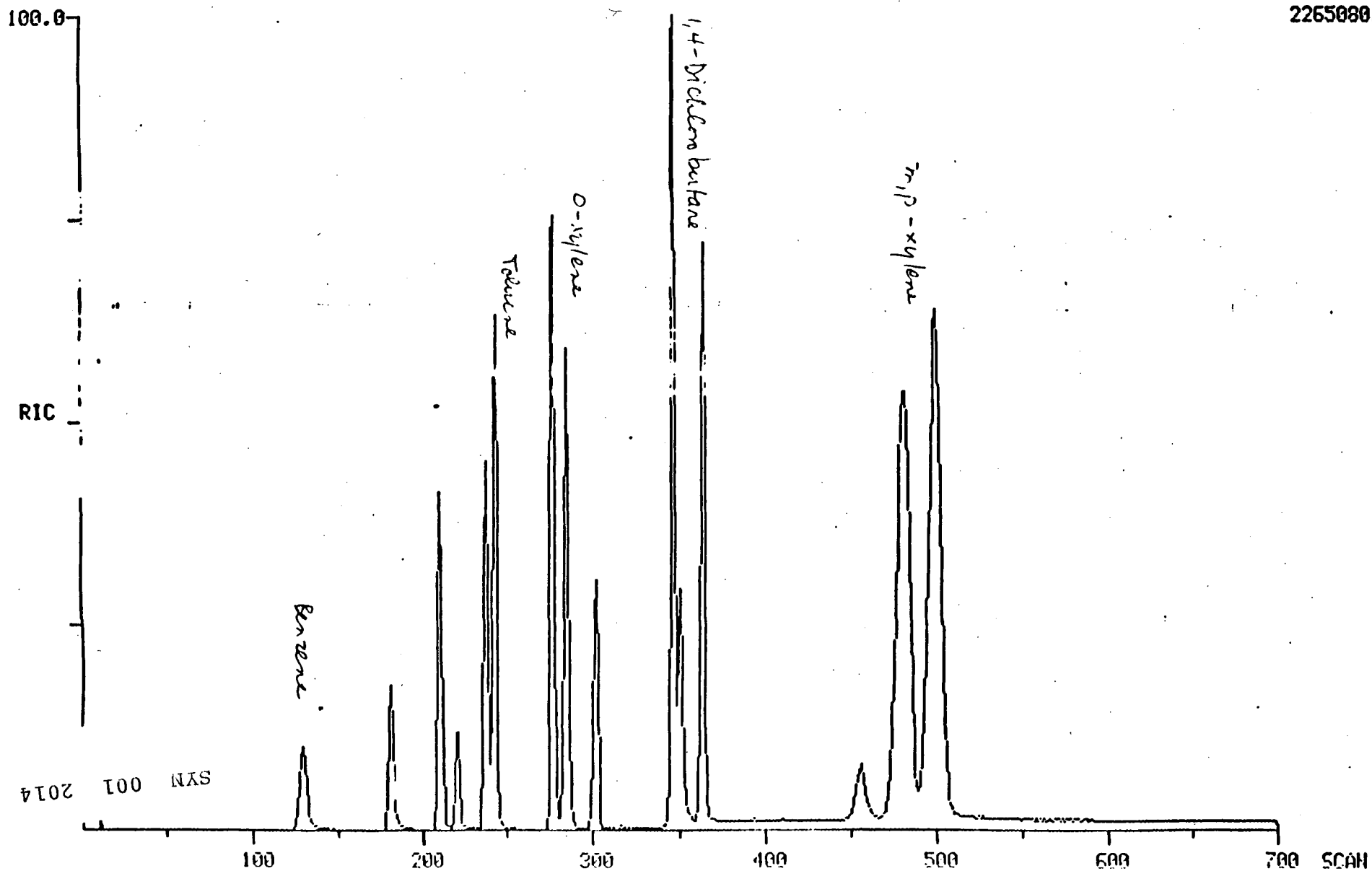
DATA: EBSTD3T811 #1
CALI: EBSTD3T811 #2

SCANS 1 TO 700

SAMPLE:
CONDS.:

RANGE: G 1, 1 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3

2265080.



File: E BSPKT811.TI

Date: 11/11/84 13:43:00

Sample:

CAL STD No. 3 - Volatile

Prepared by:

Analyst:

Amount = AREA * REF. AMNT / (REF. AREA) * RESP. FACT

sp. fac. from Library Entry

0 NAME

1 1,4 DICHLOROBUTANE (I. S.)

2 BENZENE

3 TOLUENE

4 O-XYLENE

5 M, P-XYLENE

No	m/z	Scan	Time	Ref	RRT	Meth	Area(Hght)	Amount	%Tot
1	55	351	29:45	1	1.000	A BB	440668.	44.000 NG/ML	90.20
2	78	284	24:04	1	0.809	A BV	1056550.	0.967 UG	1.98
3	92	364	30:51	1	1.037	A VB	917392.	0.977 UG	2.00
4	106	482	40:51	1	1.373	A BV	887106.	0.937 UG	1.92
5	91	500	42:23	1	1.425	A VB	2823360.	1.902 UG	3.90

SYN 001 2015

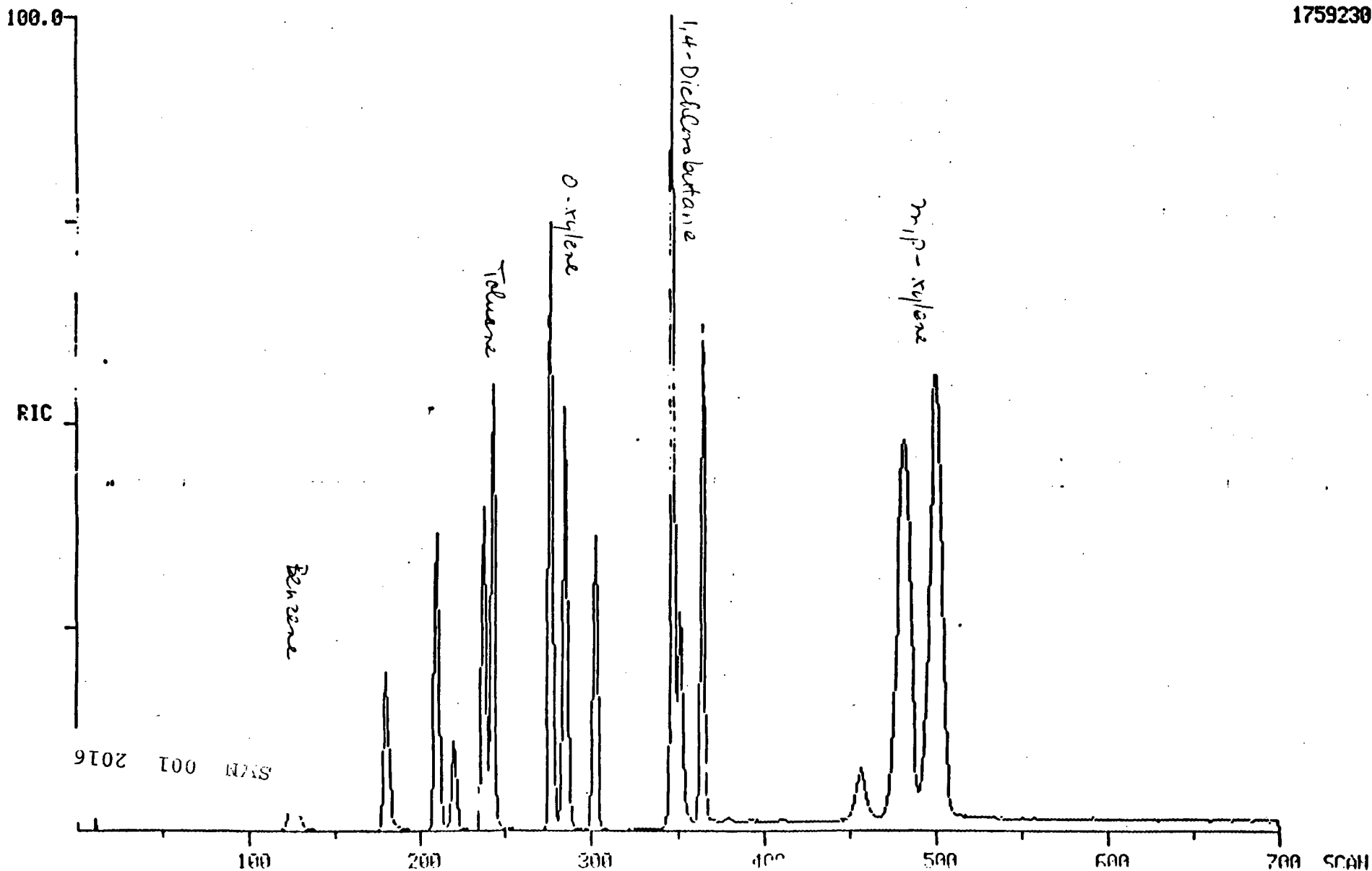
SAMPLE:
CONDS.:

DATA: EBSFKT811 #1
CALI: EBSFKT811 #2

SCANS 1 TO 700

RANGE: G 1, 1 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3

1759230.

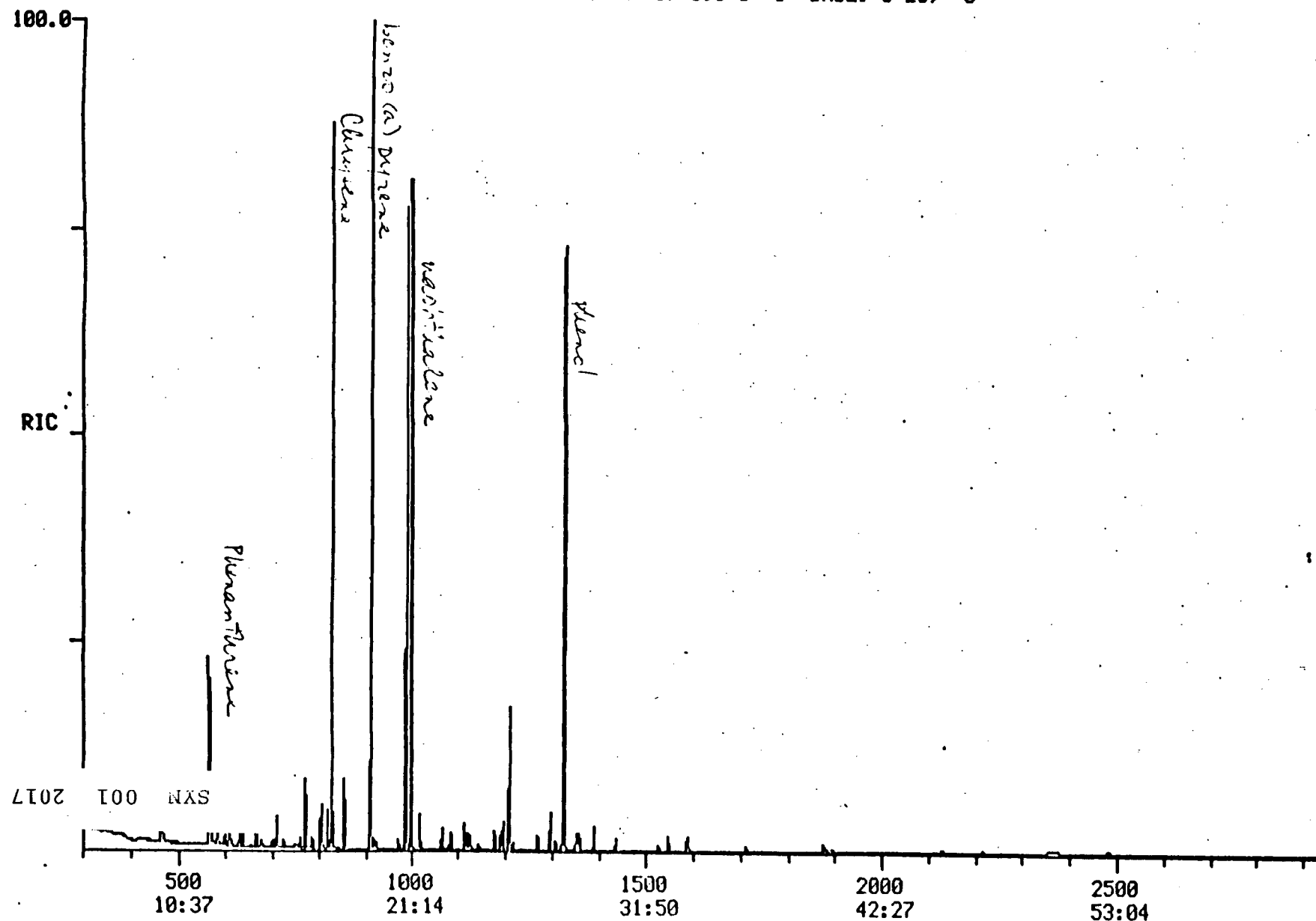


MIDRIC
06/01/84 8:53:00
SAMPLE: AE/BN STANDARD 6/1/84
CONDS.: EI
RANGE: G 1,2950 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3

DATA: ABSTD601 #1
CALI: ABSTD601 #2

SCANS 300 TO 2950

2162680.

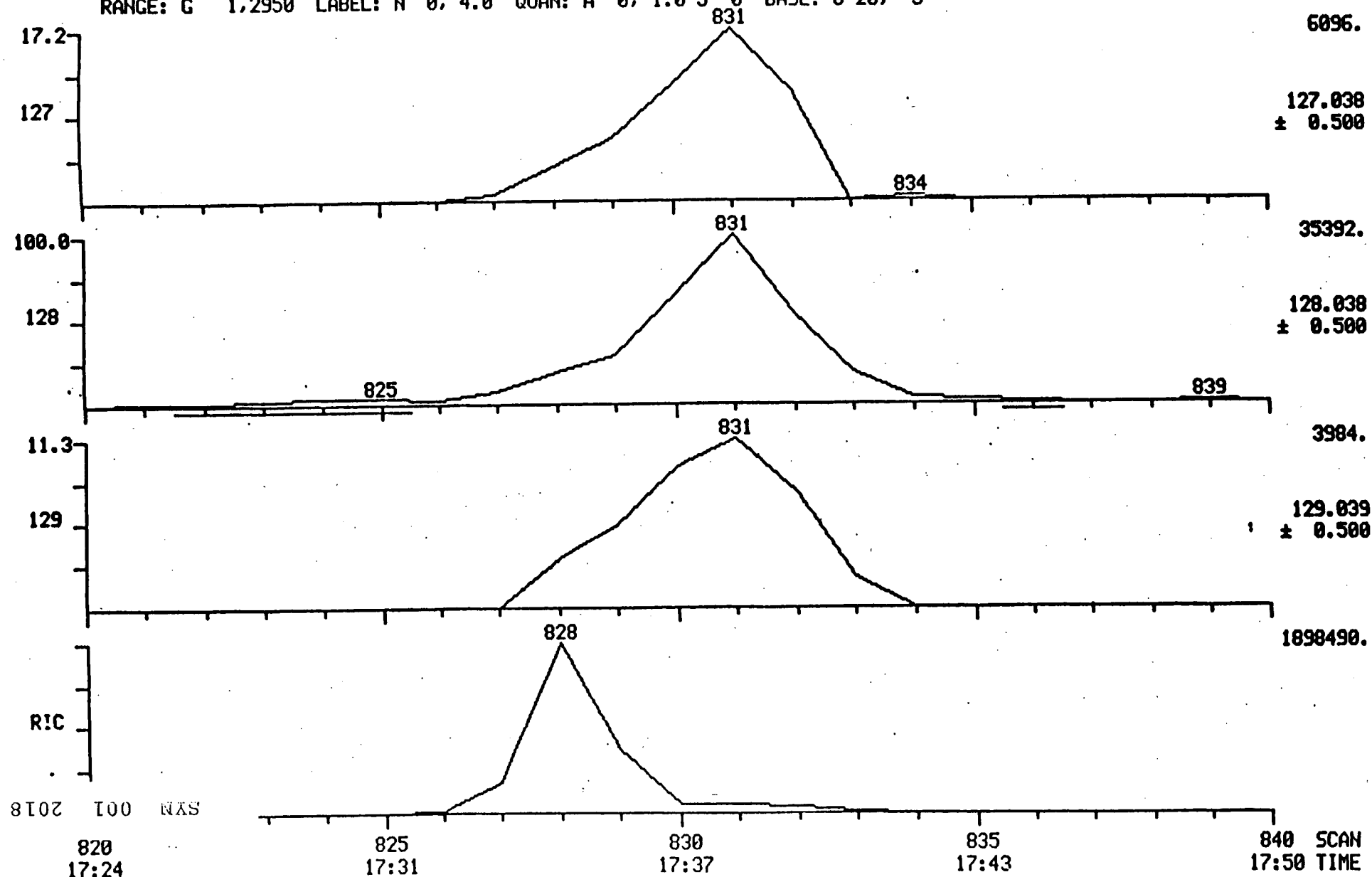


- CAL CHECK/MIDRIC - Semi-volatile -

MIDRIC+MASS CHROMATOGRAMS
06/01/84 8:53:00
SAMPLE: AE/BN STANDARD 6/1/84
CONDS.: EI
RANGE: G 1.2950 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3

DATA: ABSTD001 #1
CALI: ABSTD001 #2
[NAPHTHALENE]

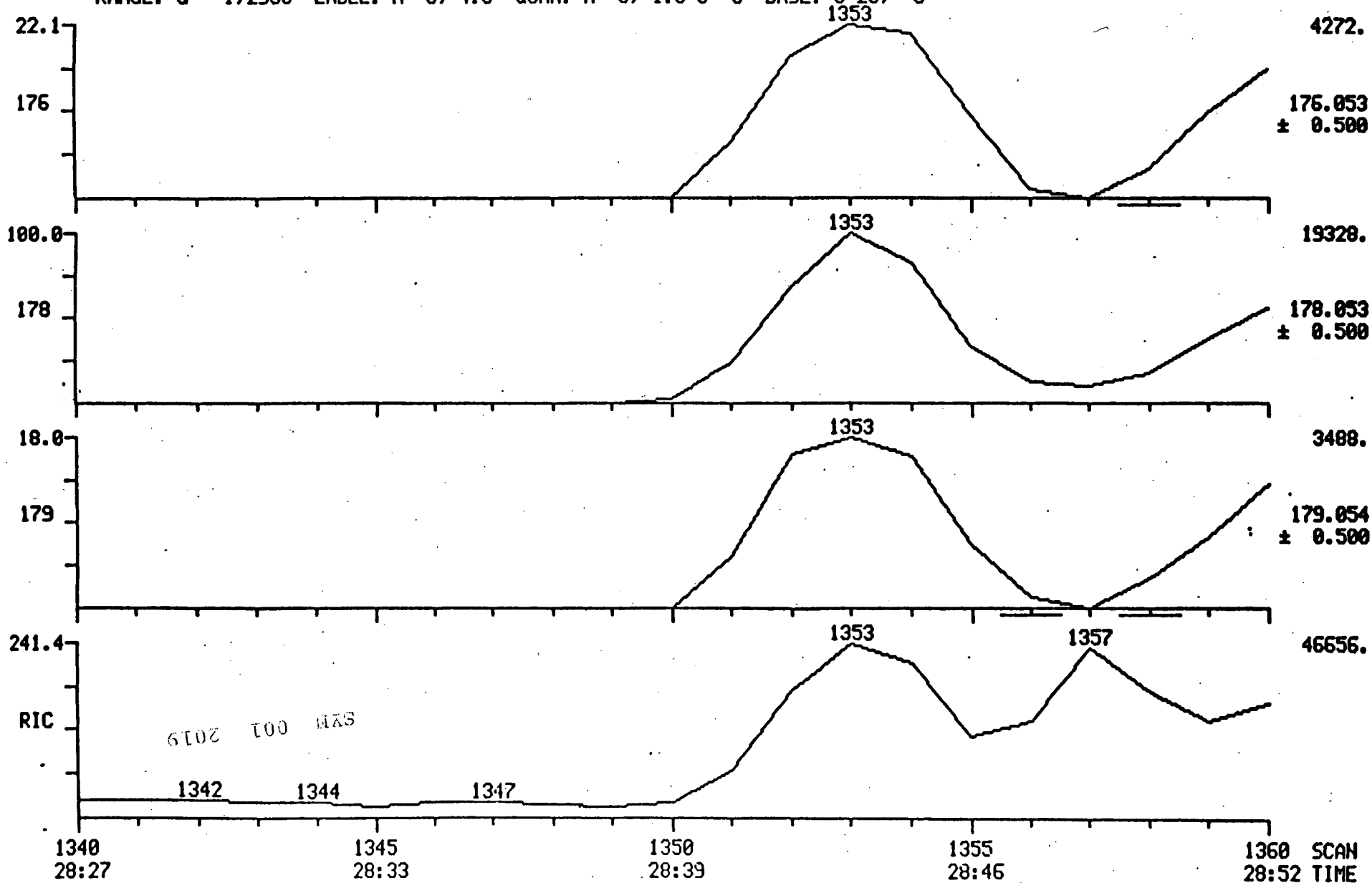
SCANS 820 TO 840



MIDRIC+MASS CHROMATOGRAMS
06/01/84 8:53:00
SAMPLE: AE/BN STANDARD 6/1/84
CONDS.: EI
RANGE: G 1,2950 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3

DATA: ABSTD601 #1
CALI: ABSTD601 #2
[PHENANTHRENE]

SCANS 1340 TO 1360

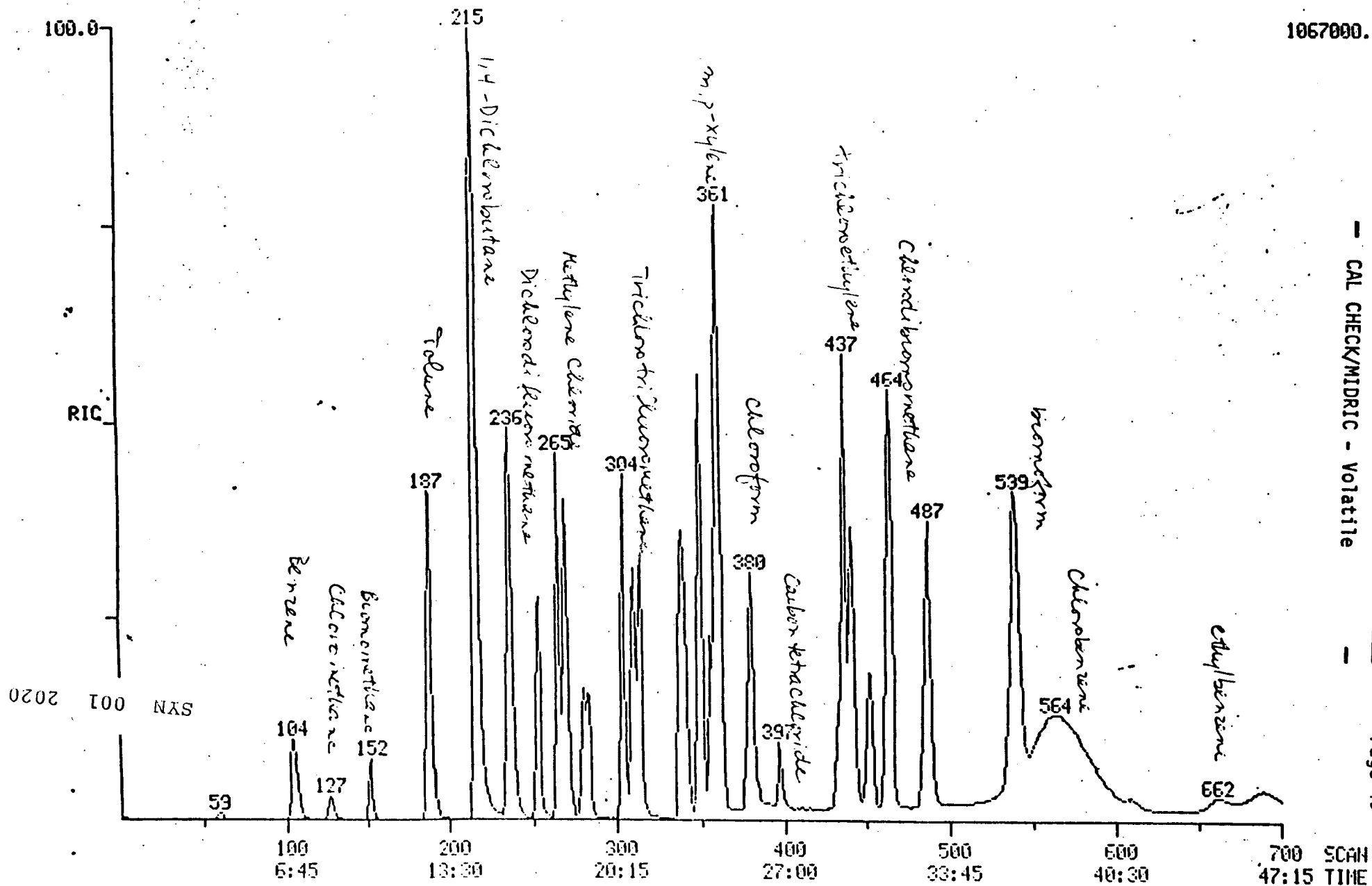


RIC
05/09/84 16:48:00
SAMPLE:

DATA: VOLSTD5088

SCANS 1 TO 700

1067000.



MIDRIC
06/26/84 19:55:00

DATA: BLANK626 #1

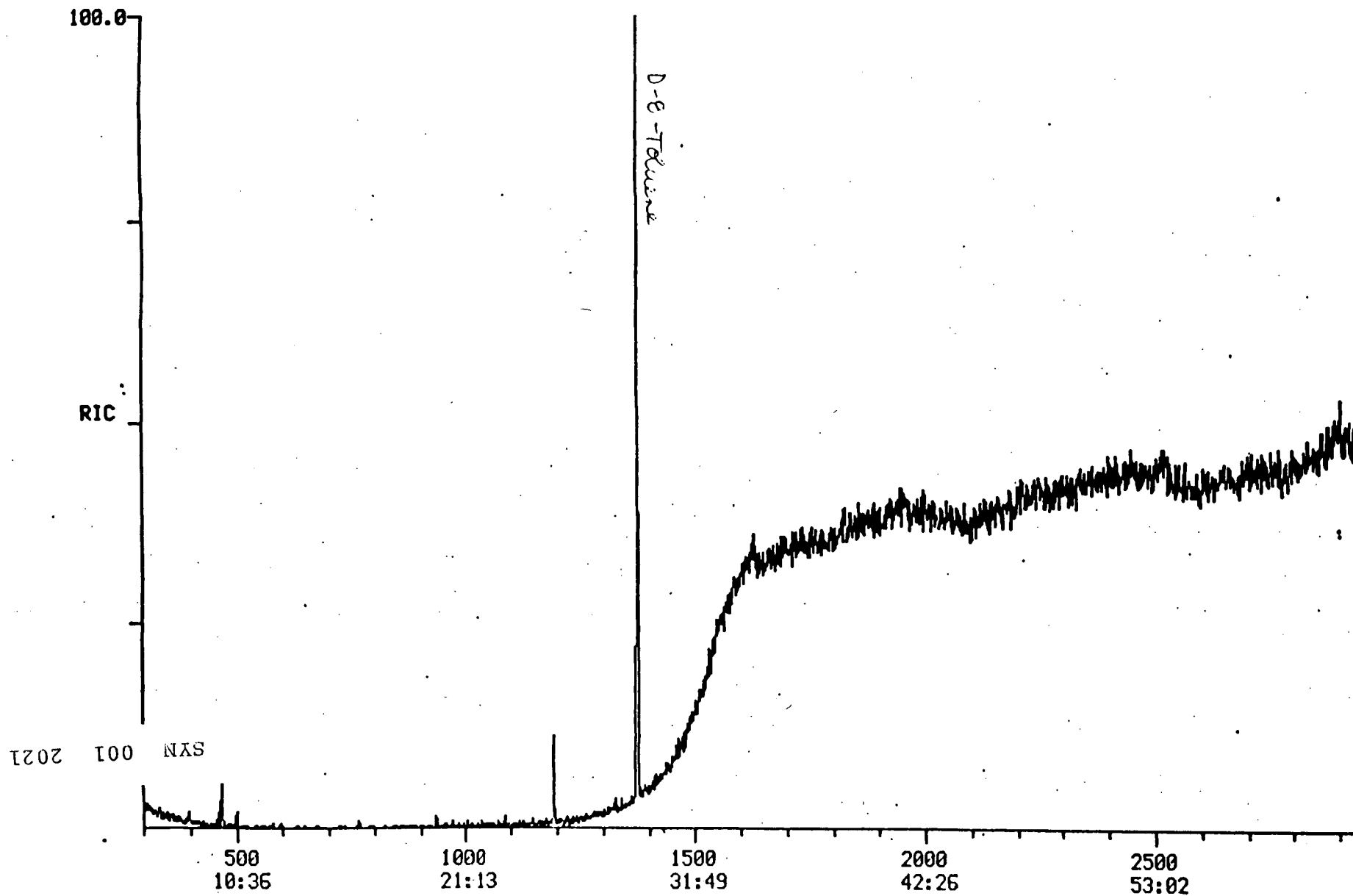
SCANS 300 TO 2950

SAMPLE:

CONDS.:

RANGE: G 1.2950 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3

105856.



- CAL CHECK/MASS SPECTRA - Volatile -

Sample Number : Sample + Spike
 Client Name : XYZ
 Date Analyzed : 5/15/84
 Instrument (Circle One) : 1020 5100
 Prep Factor : 1

Analyst : WAF
 Conditions (Circle One) EI CI
 Comments : 30 µl injected

Name	RESULTS			3-PEAK MATCH	SPIKE (UG)		
	PPB	PPM	UG		ACTUAL	THEO.	% RE
0.10-ANTHRACENE (IS)					20	20	100
BIS(2-CHLOROETHYL) ETHER		< 10			42	50	84
BENZENE, 1,4-DICHLORO- (1,4-DICHLOROBENZENE)					35	50	70
BIS(2-CHLOROETHOXY) ETHER					25	50	50
BENZENE, NITRO (NITROBENZENE)					35	50	70
ACENAPHTHYLENE					28	50	56
DIETHYLENETHALATE					48	50	96
2,4-DINITROTOLUENE					44	50	88
4-BROMOBENZOYL PHENYL ETHER					34	50	68
DIETHYLENETHALATE					39	50	78
2,3-DICHLOROBENZOTRINE					42	50	84
BIS(2-ETHYLHEXYL) ETHALATE					42	50	84
BENZO(a)FLUORANTHENE					36	50	72
DE-NITROBENZENE (SURROGATE)							
N-NITROBENZOTRISOPYL AMINE							
ETHANE, HEXACHLORO (HEXACHLOROETHANE)							
ISOPHORENE							
1,2,4-TRICHLOROBENZENE							
HEXACHLOROCYCLOPENTADIENE							
CHLORONAPHTHYLENE							
3-DIBENZYL HYDRAZINE							
N-NITRODIBENZYL AMINE							
PHENANTHRENE							
FLUORANTHENE							
BUTYLENETHALATE		14		YES			
N-NITRODIBENZYL AMINE		< 10					
4-CHLOROBENZOYL PHENYL ETHER							
BENZOTRINE							
DI-N-ETHYLENETHALATE							
BENZO(a)FLUORANTHENE							
BENZO(a)PYRENE							
INDENO(1,2,3-CD)PYRENE							
BENZO(b)FLUORENE							
2-FLUOROBIPHENYL (SURROGATE)					116	120	97
BENZENE, 1,2-DICHLORO- (1,2-DICHLOROBENZENE)	6.1			NO			
BENZENE, 1,2-DICHLORO- (1,2-DICHLOROBENZENE)	< 10						
BIS(2-CHLOROETHOXY) METHANE	< 10						
NAPHTHYLENE	21			YES			
ACENAPHTHYLENE, 1,2-DIHYDRO- (ACENAPHTHYLENE)	< 10						
2,4-DINITROTOLUENE							
CH-FLUORENE							
DIETHYLENETHALATE							
BENZENE, HEXACHLORO- (HEXACHLOROBENZENE)							
ANTHRACENE							
PYRENE							
BENZO(a)ANTHRACENE							
CHRYSENE							
DEBENZO(a,h)ANTHRACENE							
1,2,3,4,5-PENTACHLORO-1,2,3,4,5-HEXACHLORO-							
20-NAPHTHYLENE (SURROGATE)							
NOL		< 25					
1,2-DICHLOROBENZENE					130	122	106
2-NITROBENZENE					53	58	91
4-DIMETHYLPHENOL					47	43	110
4-DICHLOROPHENOL					44	50	88
4-CHLORO-2-METHYLPHENOL (P-CHLORO-M-CRESOL)					32	50	64
2,4,6-TRICHLOROPHENOL					38	50	76
2,4-DINITROPHENOL					47	50	94
4-NITROBENZENE					46	50	92

SYN 001 2025

Sample Number : Blank
 Client Name : XY2
 Date Analyzed : 5/15/84
 Instrument (Circle One) : 1020... 5100
 Prep Factor :

Analyst : WAF
 Conditions (Circle One) : EI CI
 Comments : 30 ml injected

No.	Name	RESULTS			3-PEAK MATCH	SPIKE (UG)		
		PPB	PPM	UG		ACTUAL	THEO.	% RE
1	DIO-ANTHRACENE (IS)					20	20	100
2	BIS(2-CHLOROETHYL)ETHER			<10			50	
3	BENZENE, 1,4-DICHLORO- (1,4-DICHLOROBENZENE)						50	
4	BIS(2-CHLOROETHOXY)ETHER						50	
5	BENZENE, NITRO (NITROBENZENE)						50	
6	ACENAPHTHYLENE						50	
7	DIMETHYLENTHALATE						50	
8	2,4-DINITROTOLUENE						50	
9	4-BROMOBENZOYL PHENYL ETHER						50	
0	DIMETHYLENTHALATE						50	
1	3,3'-DICHLOROBENZOTINE						50	
2	BIS(2-ETHYLHEXYL)THALATE						50	
3	BENZO(a)FLUORANTHENE						50	
4	DE-NITROBENZENE (SURROGATE)							
5	N-NITROSODIETHANOLAMINE			<10				
6	ETHANE, HEXACHLORO (HEXACHLOROETHANE)							
7	ISOPHORENE							
8	1,2,4-TRICHLOROBENZENE							
9	HEXACHLOROCYCLOPENTADIENE							
	-CHLORONAPHTHALENE							
	2-DIPHENYLHYDRAZINE							
	N-NITROSODIPHENYLAMINE							
	PHENANTHRENE							
2	FLUORANTHENE							
3	DIMETHYLENTHALATE							
4	N-NITROSODIMETHYLAMINE							
5	4-CHLOROBENZOYL PHENYL ETHER							
6	BENZOTINE							
7	DI-N-ETHYLENTHALATE							
8	BENZO(k)FLUORANTHENE							
9	BENZO(a)PYRENE							
0	INDENO(1,2,3-cd)PYRENE							
1	BENZO(b)FLUORENE							
2	2-FLUOROBIPHENYL (SURROGATE)					98	120	82
3	BENZENE, 1,3-DICHLORO- (1,3-DICHLOROBENZENE)			<10				
4	BENZENE, 1,2-DICHLORO- (1,2-DICHLOROBENZENE)							
5	BIS(2-CHLOROETHOXY)METHANE							
6	NAPHTHALENE							
7	ACENAPHTHYLENE, 1,2-DIHYDRO- (ACENAPHTHALENE)							
8	2,4-DINITROTOLUENE							
9	PH-FLUORENE							
0	DIETHYLENTHALATE							
1	BENZENE, HEXACHLORO- (HEXACHLOROBENZENE)							
2	ANTHRACENE							
3	PYRENE							
4	BENZO(a)ANTHRACENE							
5	DIBENZO(a,h)ANTHRACENE							
6	1,2,3,4,5,6-HEXACHLORO-							
7	OP-NAPHTHALENE (SURROGATE)							
8	PHENOL			<25				
9	CHLOROPHENOL							
0	--NITROPHENOL							
1	2,4-DIMETHYLPHENOL							
2	2,4-DICHLOROPHENOL							
3	4-CHLORO-2-METHYLPHENOL (P-CHLORO-M-CRESOL)							
4	2,4,6-TRICHLOROPHENOL							
5	2,4-DINITROPHENOL			<250				
6	4-NITROPHENOL			<250				
7	2-METHYL-4,6-DINITROPHENOL			<250				

SYN 001 2026

Sample Number : Duplicate
 Client Name : XYZ
 Date Analyzed : 5/15/84
 Instrument (Circle One) : 1020 5100
 Prep Factor :

Analyst : WAF
 Conditions (Circle One) EI CI
 Comments : 30 ml ~~BE~~ injected

No.	Name	RESULTS			3-PEAK MATCH	SPIKE (UG)		
		PPB	PPM	UG		ACTUAL	THEO.	% RE
1	DIO-ANTHRACENE (TS)					20	20	
2	BIS(2-CHLOROETHYL) ETHER			<10			50	
3	BENZENE, 1,4-DICHLORO- (1,4-DICHLORO BENZENE)						50	
4	BIS(2-CHLOROISOPROPYL) ETHER						50	
5	BENZENE, NITRO (NITROBENZENE)						50	
6	ACENAPHTHYLENE						50	
7	DIMETHYL PHTHALATE						50	
8	2,4-DINITROTOLUENE						50	
9	4-BROMOPHENYL PHENYL ETHER						50	
10	DIETHYL PHTHALATE						50	
11	3,3'-DICHLORO BENZIDINE						50	
12	BIS(2-ETHYLHEXYL) PHTHALATE						50	
13	BENZO(E) FLUORANTHENE						50	
14	DE-NITROBENZENE (SURROGATE)							
15	N-NITROSODIMETHYL AMINE			<10				
16	ETHANE, HEXACHLORO (HEXACHLOROETHANE)							
17	ISOPHOBONE							
18	1,2,4-TRICHLORO BENZENE							
19	HEXACHLOROCYCLOPENTADIENE			13				
20	2-CHLORONAPHTHALENE			<10				
21	2-DIPHENYL HYDRAZINE							
22	-NITROSODIPHENYL AMINE							
23	PHENANTHRENE							
24	FLUORANTHENE							
25	BUTYLENYL PHTHALATE							
26	N-NITROSODIMETHYL AMINE							
27	4-CHLORO PHENYL PHENYL ETHER							
28	BENZIDINE							
29	DI-N-OCTYL PHTHALATE							
30	BENZO(K) FLUORANTHENE							
31	BENZO(A) PYRENE							
32	INDENO(1,2,3-CD) PYRENE							
33	BENZO(GHI) PERYLENE							
34	2-FLUORODIPHENYL (SURROGATE)						120	
35	BENZENE, 1,3-DICHLORO- (1,3-DICHLORO BENZENE)			6.7	NO			
36	BENZENE, 1,2-DICHLORO- (1,2-DICHLORO BENZENE)			<10				
37	BIS(2-CHLOROETHOXY) METHANE			<10				
38	NAPHTHALENE			32				
39	ACENAPHTHYLENE, 1,2-DIHYDRO- (ACENAPHTHALENE)			<10				
40	2,4-DINITROTOLUENE							
41	9H-FLUORENE							
42	DIETHYL PHTHALATE							
43	BENZENE, HEXACHLORO- (HEXACHLORO BENZENE)							
44	ANTHRACENE							
45	PYRENE							
46	BENZO(A) ANTHRACENE							
47	CHRYSENE							
48	DEBENZO(A,H) ANTHRACENE							
49	1,3-PHTHALDIENE, 1,1,2,3,4,4-HEXACHLORO-							
50	OP-NAPHTHALENE (SURROGATE)						122	
51	PHENOL			<25			58	
52	2-CHLOROPHENOL			<25			49	
53	NITROPHENOL			59	YES		43	
54	4-DIMETHYLPHENOL			<25			50	
55	2,4-DICHLOROPHENOL						50	
56	4-CHLORO-2-METHYLPHENOL (P-CHLORO-M-CRESOL)						50	
57	2,4,6-TRICHLOROPHENOL						50	
58	2,4-DINITROPHENOL			<25			50	
59	4-NITROPHENOL			<25			50	
60	2-METHYL-4,6-DINITROPHENOL			<25			50	
61	PENTACHLOROPHENOL			<25			51	
62	2-FLUOROPHENOL (SURROGATE)						123	

SYN 001 2027

MLC

06/01/84 15:47:00

SAMPLE: 9581-2 AE/BN DUP REDD 1:10 DIL

CONDS.: EI

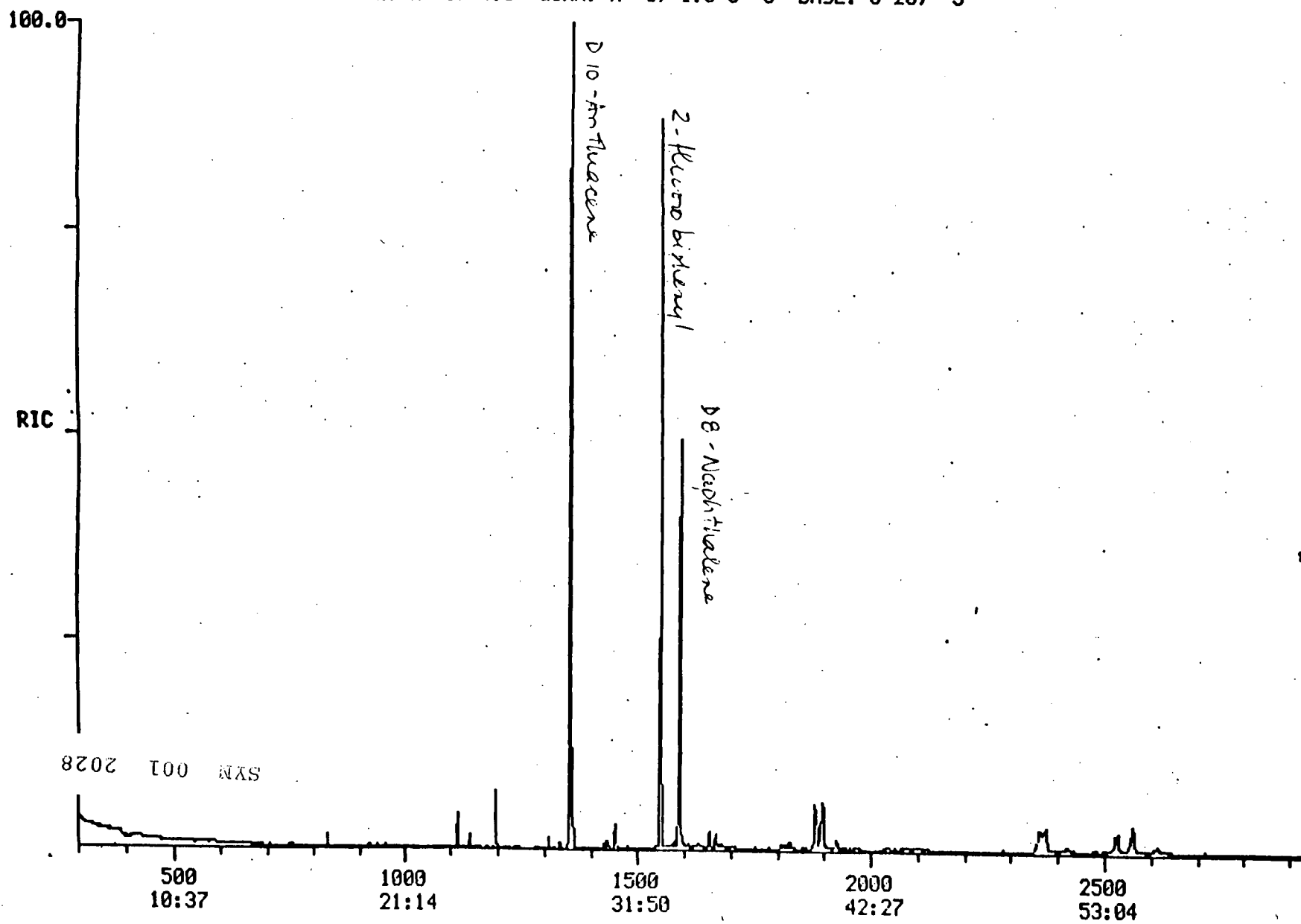
RANGE: G 1,2950 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3

DATA: 95812ABDR #1

CALI: 95812ABDR #2

SCANS 300 TO 2950

4562940.



SEMIVOL. QUANT./MIDRIC - SAMPLE

MIDRIC+MASS CHROMATOGRAMS

DATA: 95812ABDR3 #1

SCANS 830 TO 850

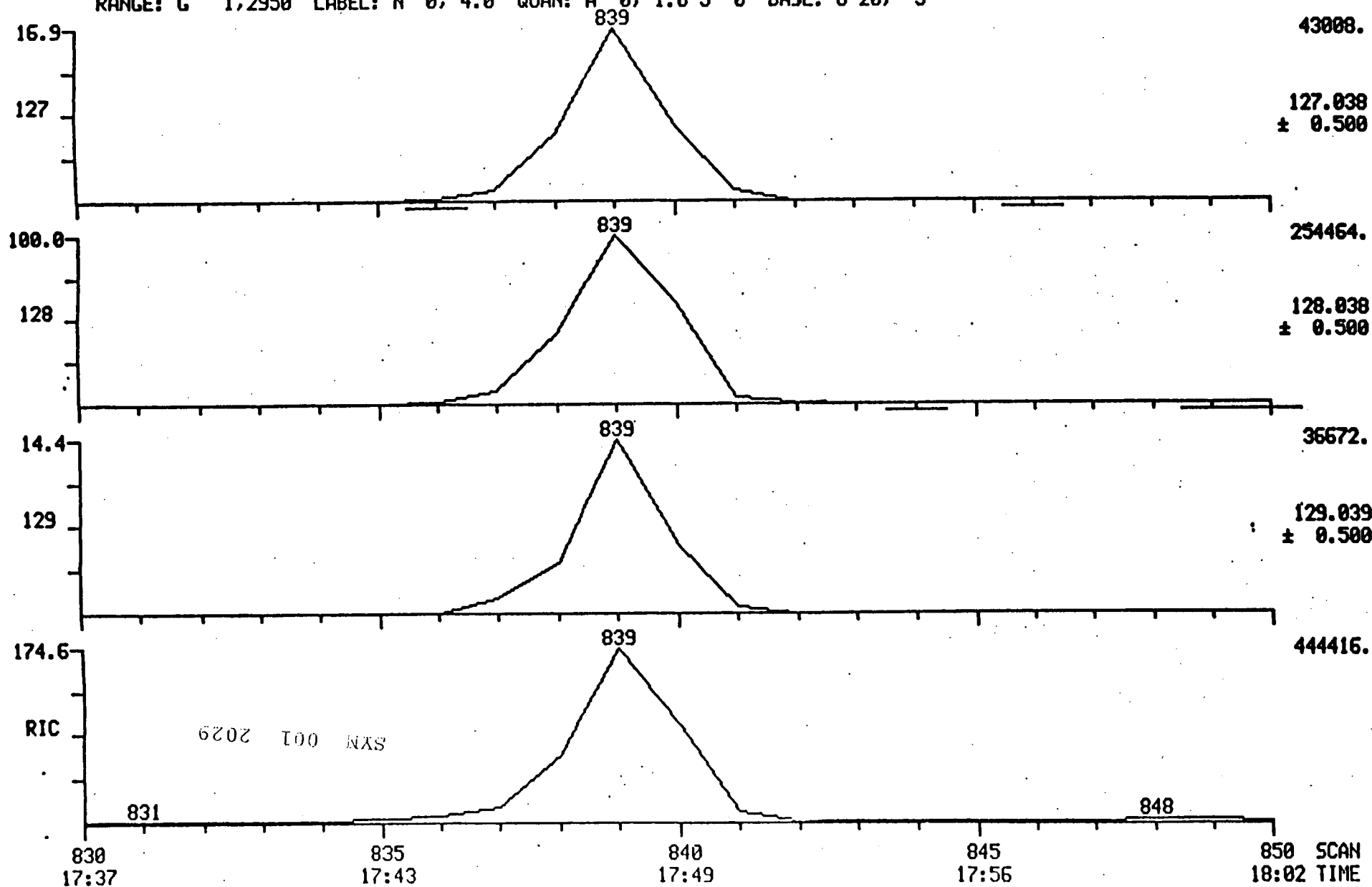
06/25/84 15:48:00

CALI: 95812ABDR3 #2

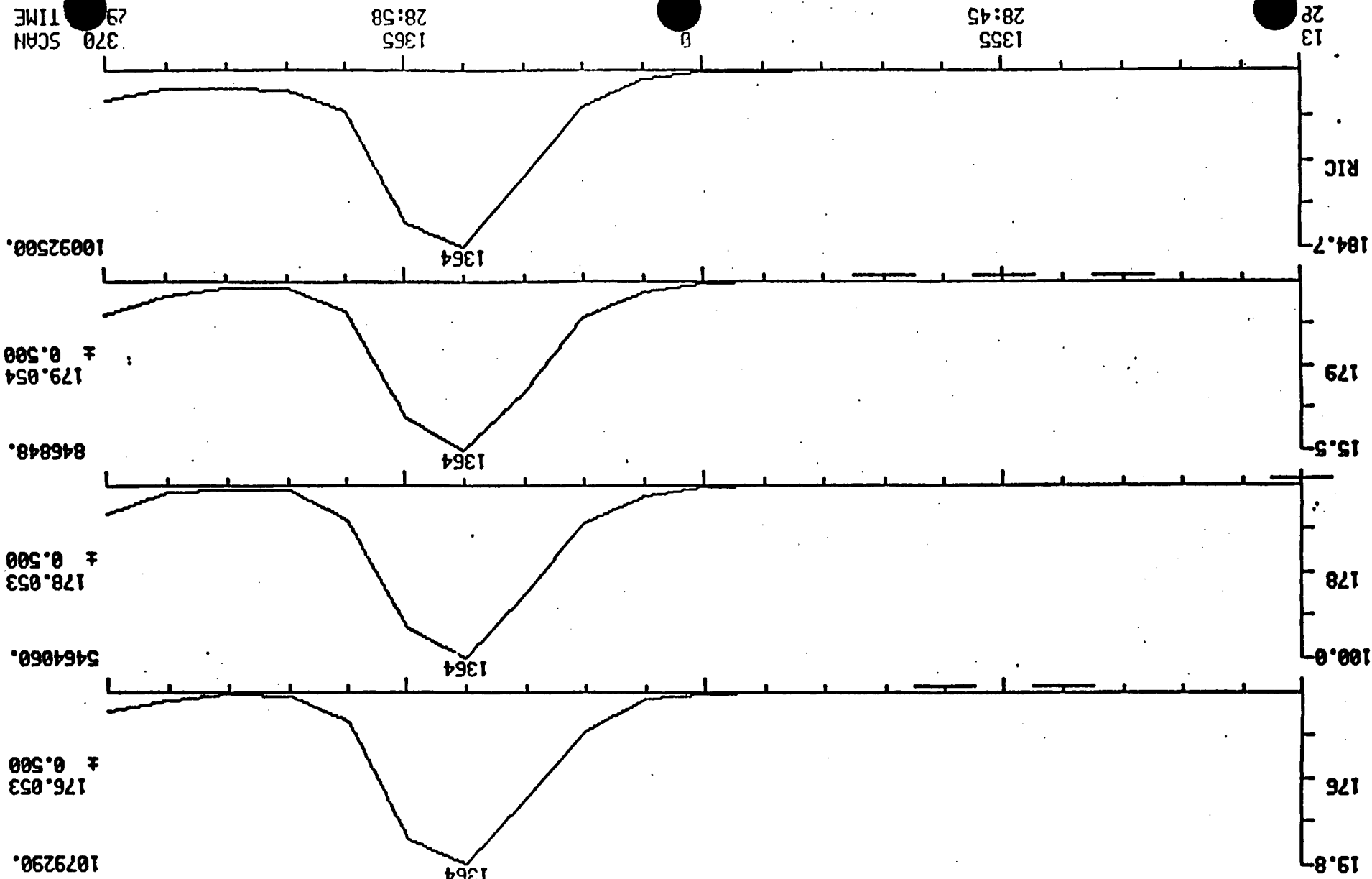
SAMPLE: 9581-2 AE/BN REDO#3 (1:10 DIL) [NAPHTHALENE]

CONDS.: EI

RANGE: G 1.2950 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3



MIDRIC+MASS CHROMATOGRAMS
 06/25/84 15:48:00
 SAMPLE: 9581-2 AE/BN REDO#3 (1:10 DIL) [PHENANTHRENE]
 COND.: EI
 RANGE: C 1.2950 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3
 DATA: 95812ABDR3 #1
 CALI: 95812ABDR3 #2
 SCANS 1350 TO 1370



SYN 001 2030

MIL

06/01/84 15:47:00

DATA: 95812ABDR #.

SCANS 300 TO 2950

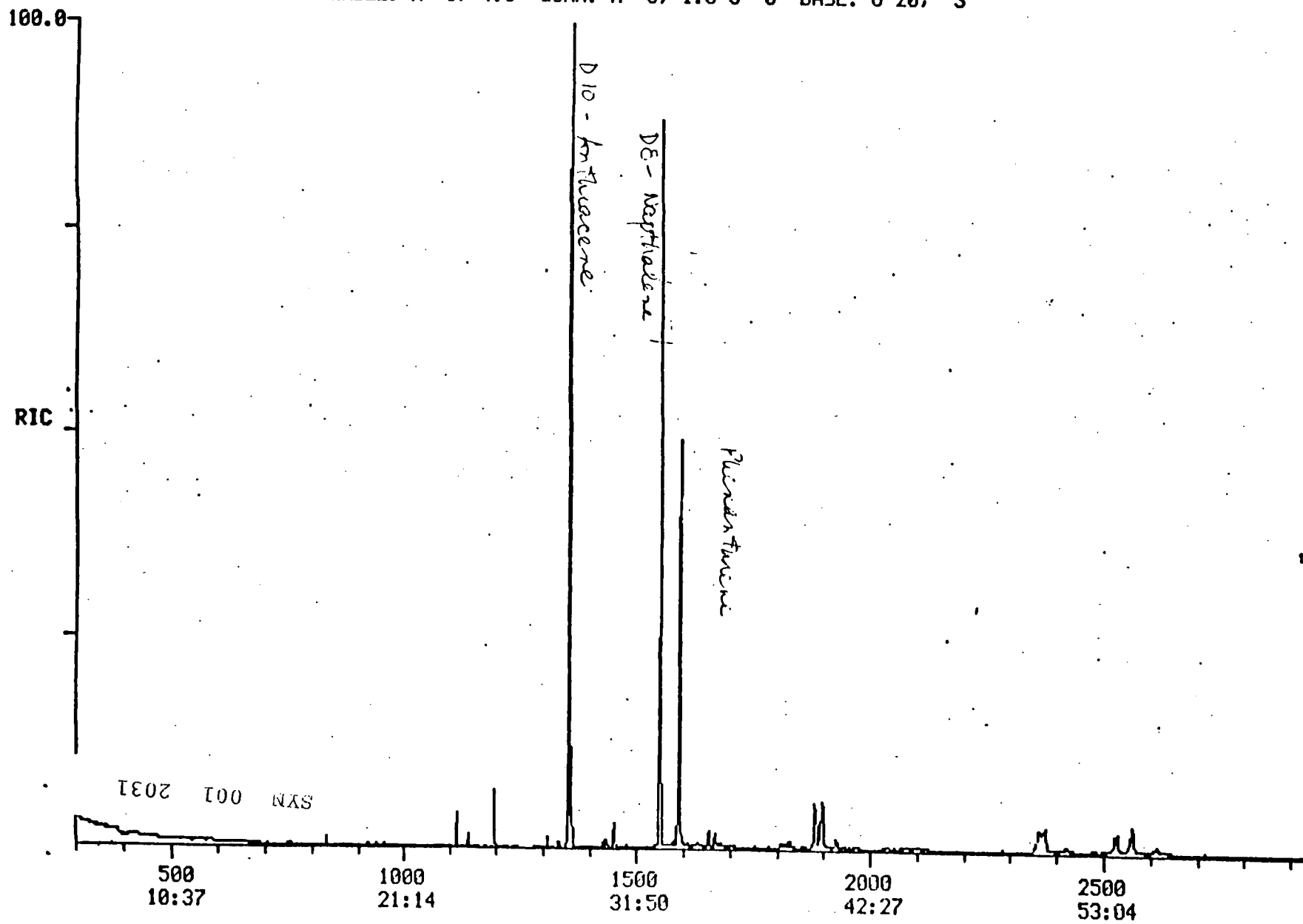
CALI: 95812ABDR #2

SAMPLE: 9581-2 AE/BN DUP REDO 1:10 DIL

CONDS.: EI

RANGE: G 1,2950 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3

4562940.



SEMIVOL./MIDRIC - DUPLICATE

MIDRIC+MASS CHROMATOGRAMS

06/25/84 15:48:00

SAMPLE: 9581-2 AE/BN REDO#3 (1:10 DIL) [NAPHTHALENE]

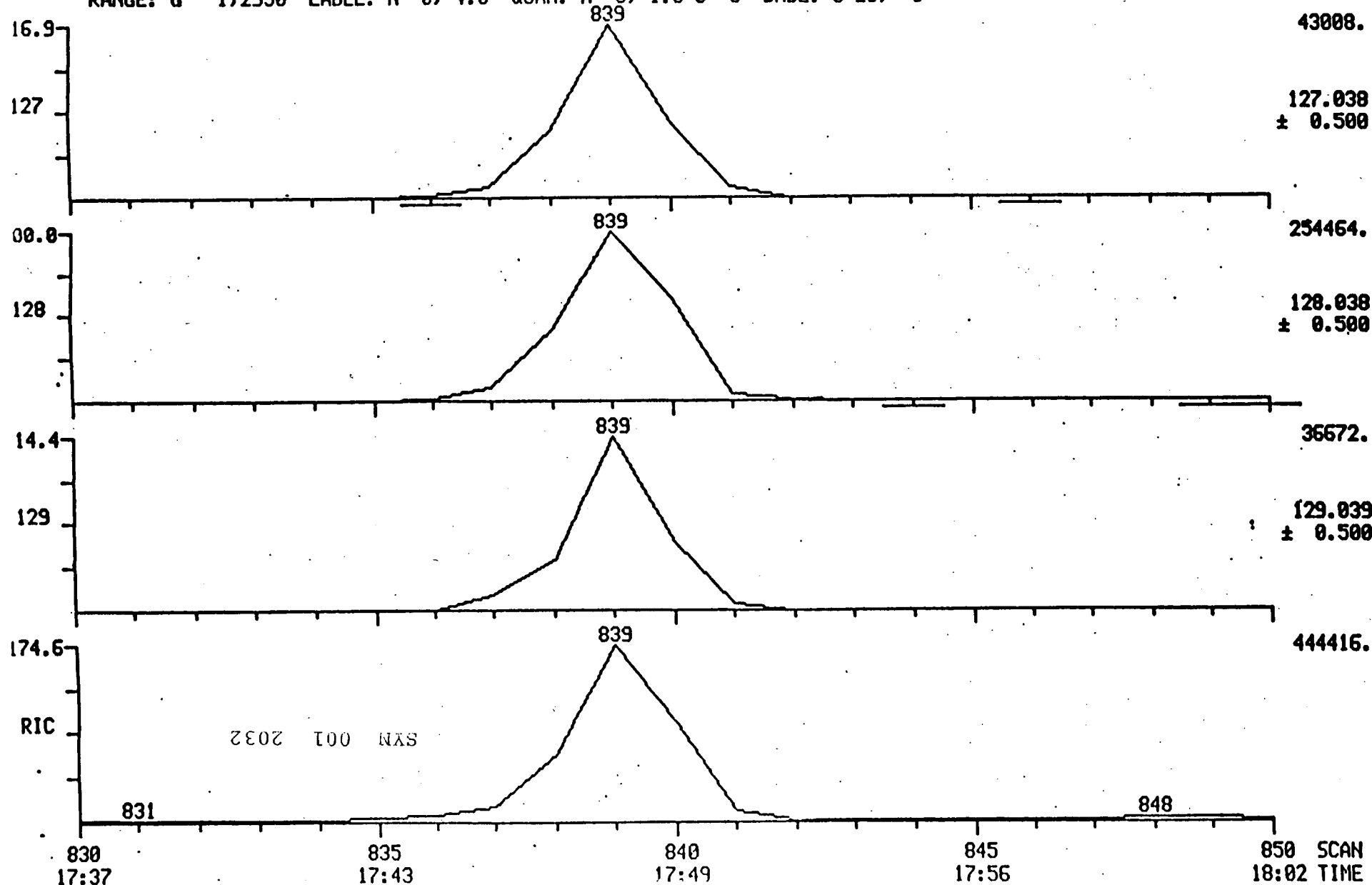
CONDS.: EI

RANGE: G 1.2950 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3

DATA: 95812ABDR3 #1

SCANS 830 TO 850

CALI: 95812ABDR3 #2



SEMI-VOL. MASS SPECTRA - DUPLICATE

MIDRIC+MASS CHROMATOGRAMS

DATA: 95812ABDR3 #1

SCANS 1350 TO 1370

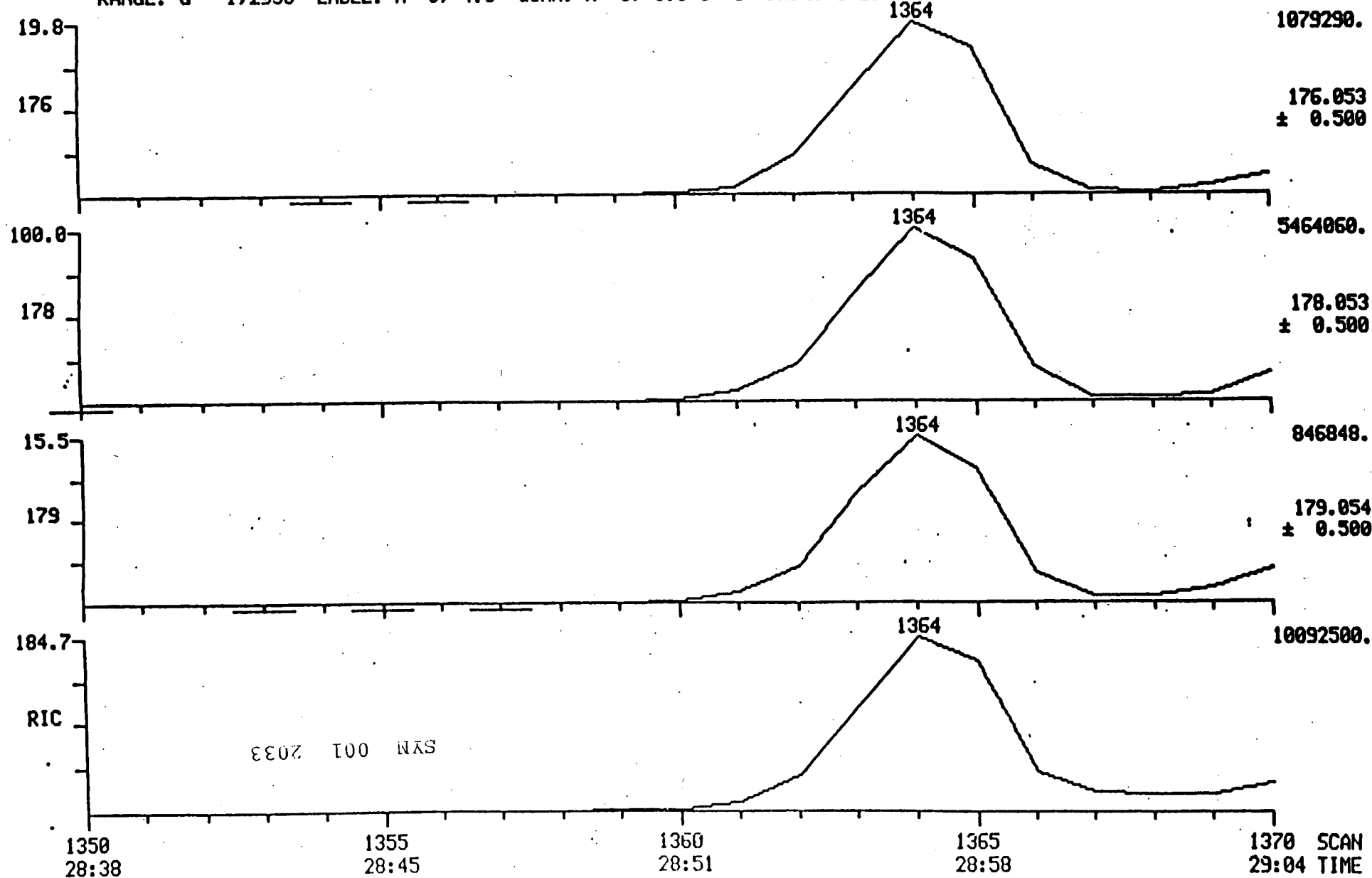
06/25/84 15:48:00

CALI: 95812ABDR3 #2

SAMPLE: 9581-2 AE/BN REDON3 (1:10 DIL) [PHENANTHRENE]

CONDS.: EI

RANGE: G 1,2950 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3

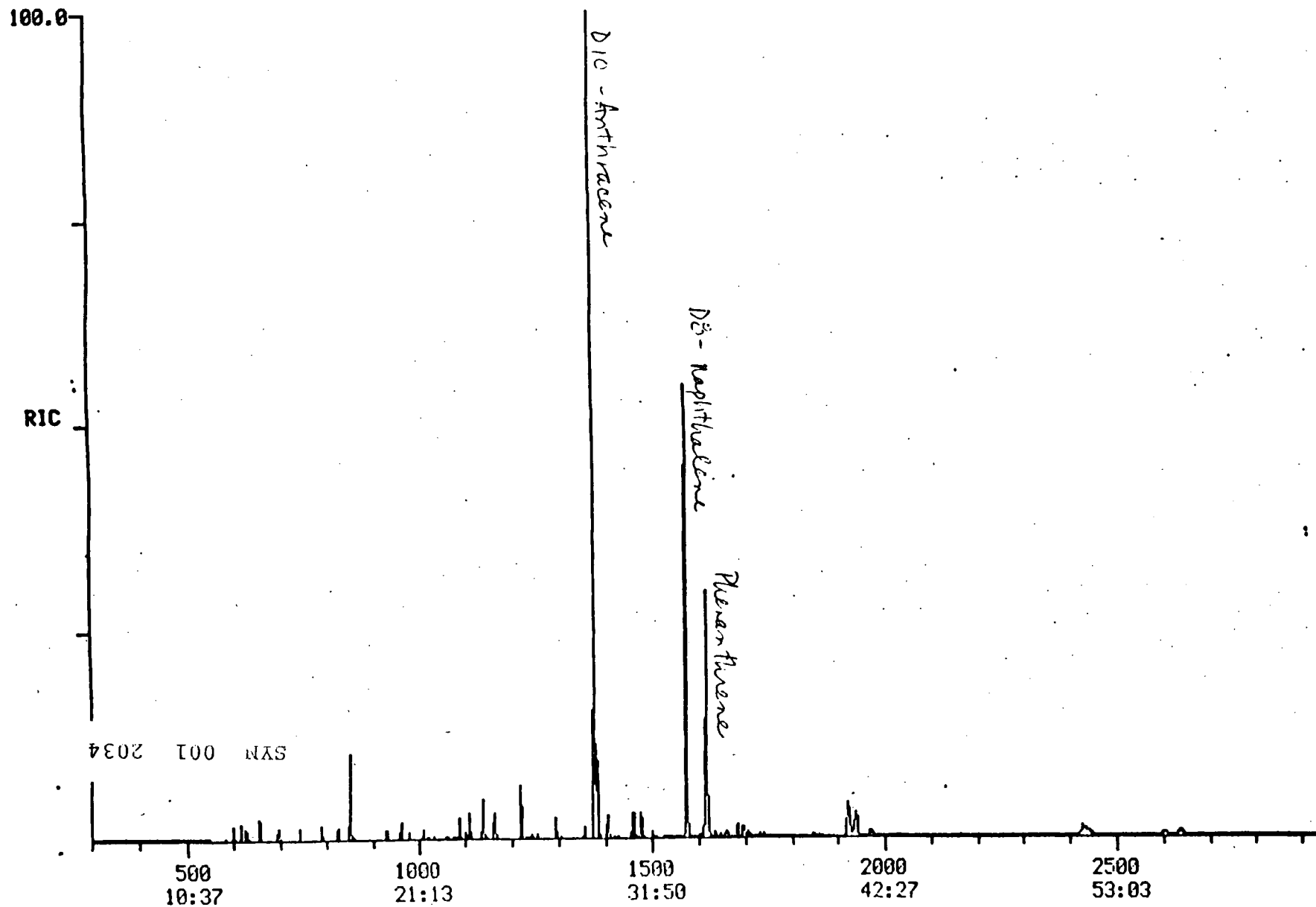


MIDRIC
05/11/84 20:06:00
SAMPLE: 9581-2 AE/BN SPIKE 1:10
CONDS.: EI
RANGE: G 1,2950 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3

DATA: 95812ABS #1
CALI: 95812ABS #2

SCANS 300 TO 2950

5914620.



SEMIVOL. QUANT./MIDRIC - SPIKE

MIDRIC+MASS CHROMATOGRAMS

06/01/84 15:47:00

SAMPLE: 9581-2 AE/BN DUP REDO 1:10 DIL [NAPHTHALENE]

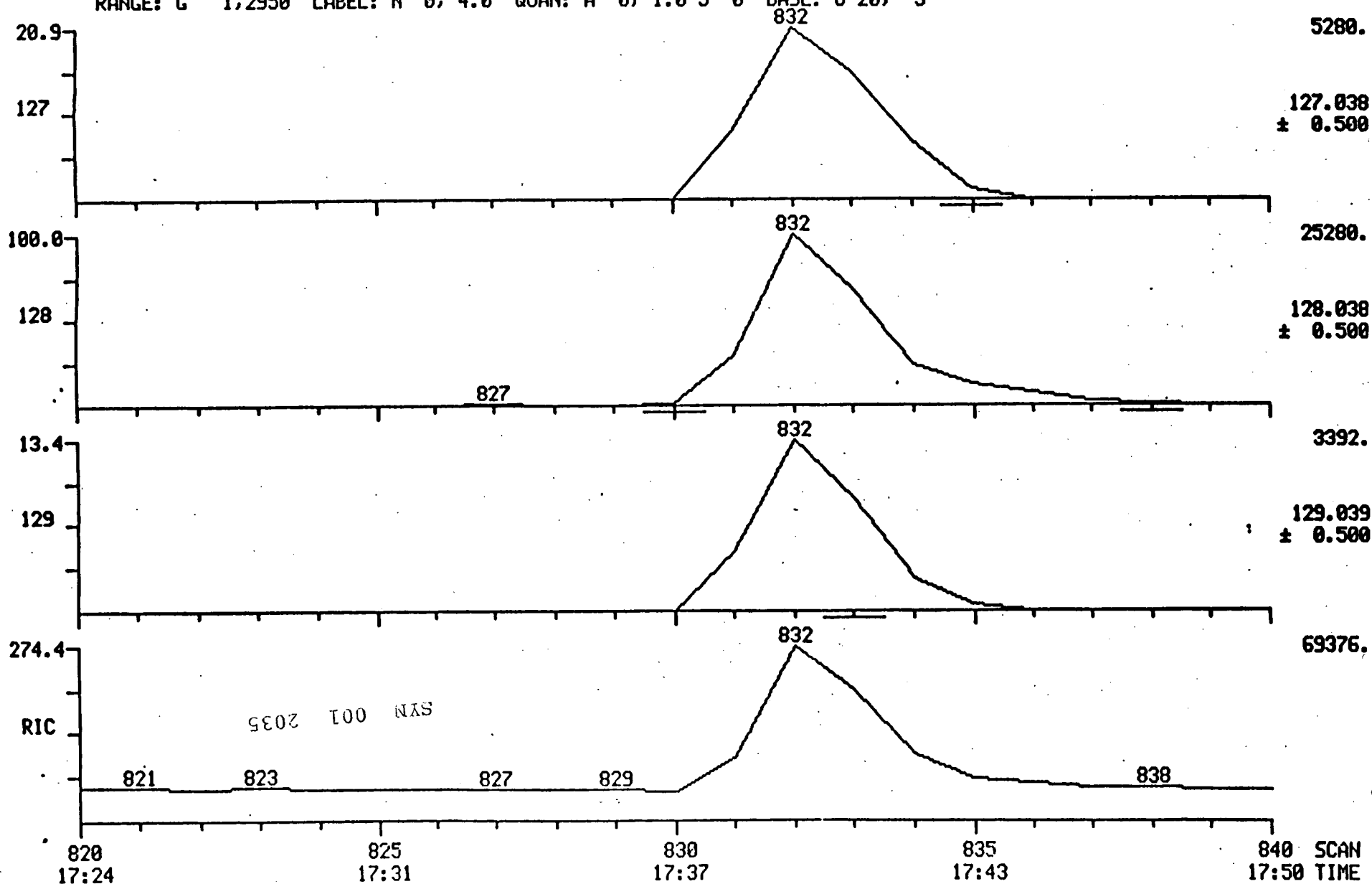
CONDS.: EI

RANGE: G 1,2950 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3

DATA: 95812ABDR #1

CALI: 95812ABDR #2

SCANS 820 TO 840



MIDRIC+MASS CHROMATOGRAMS

DATA: 95812ABDR #1

SCANS 1340 TO 1360

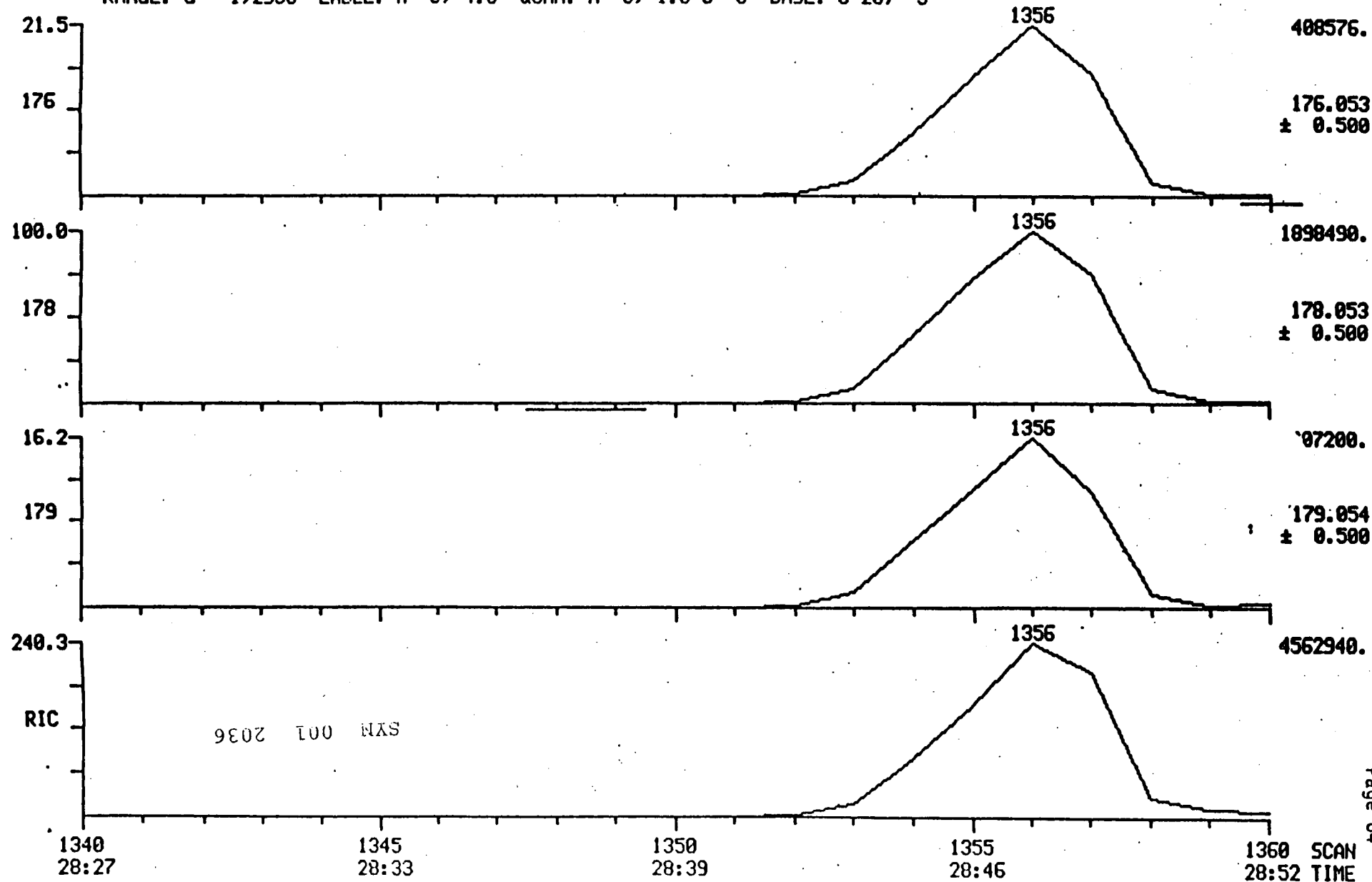
06/01/84 15:47:00

CALI: 95812ABDR #2

SAMPLE: 9581-2 AE/BN DUP REDO 1:10 DIL [PHENANTHRENE]

CONDS.: EI

RANGE: G 1,2950 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3



Sample Number : Blank Analyst : WAF
 Sample Name : XYZ Prep Factor : 1
 Date Analyzed : 6/25/84 Amount Purged : 30 ml
 Instrument (Circle One): 1020 5100A 5100B Calculation Factor: _____
 Comments : _____

No.	NAME	RESULTS			THREE-PEAK CRITERION	
		PPB/	PPM	UG	YES/NO	CODE*/COMMENTS
1	Chloromethane		<10			
2	Bromomethane					
3	Vinyl Chloride					
4	Chloroethane					
5	Methylene Chloride					
6	1,1-Dichloroethene					
7	1,1-Dichloroethane					
8	Trans-1,2-Dichloroethene					
9	Chloroform					
10	1,2-Dichloroethane					
11	1,1,1-Trichloroethane					
12	Carbon Tetrachloride					
13	Bromodichloromethane					
14	1,2-Dichloropropane					
	Trans-1,3-Dichloropropene					
	Trichloroethene (TCE)					
	Benzene					
18	Dibromochloromethane					
19	1,1,2-Trichloroethane					
20	Cis-1,3-Dichloropropene					
21	2-Chloroethyl Vinyl Ether					
22	Bromoform					
23	1,1,2,2-Tetrachloroethane					
24	Tetrachloroethene					
25	Toluene					
26	Chlorobenzene					
27	Ethyl Benzene					
28	Xylenes, total					
29	Dichlorodifluoromethane					
30	Trichlorofluoromethane					
31	Acrolein		<10			
32	Acrylonitrile		<10			

SYN 001 2037

SURROGATE RECOVERY DATA

NO.	SURROGATE	AMOUNT SPIKED (NG)	% RECOVERY	ACCEPTABLE LIMITS
1	D8-Phenol	258	85	
	BFB	120	76	

Three-Peak Codes:

1. Ions Missing

Sample Number : Sample Analyst : WAF
 Client Name : XYZ Prep Factor : -
 : Analyzed : 6/25/84 Amount Purged : 30 ml
 Instrument (Circle One): 1020 5100A 5100B Calculation Factor: -
 Comments :

No.	NAME	RESULTS			THREE-PEAK CRITERION	
		PPB	PPM	UG	YES/NO	CODE*/COMMENTS
1	Chloromethane		<10			
2	Bromomethane					
3	Vinyl Chloride					
4	Chloroethane					
5	Methylene Chloride					
6	1,1-Dichloroethene					
7	1,1-Dichloroethane					
8	Trans-1,2-Dichloroethene					
9	Chloroform					
10	1,2-Dichloroethane					
11	1,1,1-Trichloroethane					
12	Carbon Tetrachloride					
13	Bromodichloromethane					
14	1,2-Dichloropropane					
15	Trans-1,3-Dichloropropene					
16	Trichloroethene (TCE)					
17	Benzene					
18	Dibromochloromethane					
19	1,1,2-Trichloroethane					
20	Cis-1,3-Dichloropropene					
21	2-Chloroethyl Vinyl Ether					
22	Bromoform					
23	1,1,2,2-Tetrachloroethane					
24	Tetrachloroethene					
25	Toluene					
26	Chlorobenzene					
27	Ethyl Benzene					
28	Xylenes, total					
29	Dichlorodifluoromethane					
30	Trichlorofluoromethane					
31	Acrolein		<100			
32	Acrylonitrile		<10			

SYN 001 2038

SURROGATE RECOVERY DATA

NO.	SURROGATE	AMOUNT SPIKED (NG)	% RECOVERY	ACCEPTABLE LIMITS
1	D8-Phenol	258	95	
2	BFB	120	85	

* Three-Peak Codes:

1. Ions Missing
2. All ions present but not within 20%. Compound probably present.
3. All ions present but not within 20%. Compound probably not present.

Sample Number : Duplicate Analyst : WAF
 Client Name : XYZ Prep Factor : -
 Analyzed : 6/25/84 Amount Purged : 30 ml
 Instrument (Circle One): 1020 5100A 5100B Calculation Factor: _____
 Comments : _____

No.	NAME	RESULTS			THREE-PEAK CRITERION	
		PPB	PPM	UG	YES/NO	CODE*/COMMENTS
1	Chloromethane		<10			
2	Bromomethane					
3	Vinyl Chloride					
4	Chloroethane					
5	Methylene Chloride					
6	1,1-Dichloroethene					
7	1,1-Dichloroethane					
8	Trans-1,2-Dichloroethene					
9	Chloroform					
10	1,2-Dichloroethane					
11	1,1,1-Trichloroethane					
12	Carbon Tetrachloride					
13	Bromodichloromethane					
14	1,2-Dichloropropane					
15	Trans-1,3-Dichloropropene					
16	Trichloroethene (TCE)					
17	Benzene					
18	Dibromochloromethane					
19	1,1,2-Trichloroethane					
20	Cis-1,3-Dichloropropene					
21	2-Chloroethyl Vinyl Ether					
22	Bromoform					
23	1,1,2,2-Tetrachloroethane					
24	Tetrachloroethene					
25	Toluene					
26	Chlorobenzene					
27	Ethyl Benzene					
28	Xylenes, total					
29	Dichlorodifluoromethane					
30	Trichlorofluoromethane					
31	Acrolein		<100			
32	Acrylonitrile		<100			

SYN 001 2039

SURROGATE RECOVERY DATA

NO.	SURROGATE	AMOUNT SPIKED (NG)	% RECOVERY	ACCEPTABLE LIMITS
1	D8-Phenol	250	110	
2	BFB	120	95	

Three-Peak Codes:

1. Ions Missing

2. All ions present but not within 20%. Compound probably present.

EPA Method 624 Purgeable Organics by GC/MS

Spike Recovery Data

Job Number : ABCD Instrument (Circle One): 1020 5100A 5100B
 Sample Spiked : Sample + Spike Analyst : WAF
 Date Analyzed : Agulons Matrix : —

No.	NAME	Amount Spiked (ug)	% Recovery	Recovery Limits
1	Chloromethane	100	87	111 ± 76
2	Bromomethane		85	94 ± 32
3	Vinyl Chloride		85	108 ± 47
4	Chloroethane		42	98 ± 27
5	Methylene Chloride		70	101 ± 23
6	1,1-Dichloroethene		75	100 ± 28
7	1,1-Dichloroethane		92	91 ± 26
8	Trans-1,2-Dichloroethene		85	116 ± 38
9	Chloroform		93	107 ± 22
10	1,2-Dichloroethane		92	113 ± 37
11	1,1,1-Trichloroethane		94	108 ± 27
12	Carbon Tetrachloride		76	97 ± 29
13	Bromodichloromethane		110	110 ± 21
14	1,2-Dichloropropane		84	103 ± 21
	Trans-1,3-Dichloropropene		95	106 ± 14
	Trichloroethene (TCE)		78	92 ± 30
	Benzene		92	105 ± 17
18	Dibromochloromethane		120	109 ± 32
19	1,1,2-Trichloroethane		77	108 ± 22
20	Cis-1,3-Dichloropropene		105	109 ± 23
21	2-Chloroethyl Vinyl Ether		88	104 ± 19
22	Bromoform		87	116 ± 39
23	1,1,2,2-Tetrachloroethane		89	111 ± 26
24	Tetrachloroethene		110	100 ± 24
25	Toluene		105	98 ± 37
26	Chlorobenzene		96	93 ± 40
27	Ethyl Benzene		95	100 ± 43
28	Xylenes, total		97	95 ± 30
29	Dichlorodifluoromethane		110	87 ± 19
30	Trichlorofluoromethane		99	95 ± 34
31	Acrolein	200	65	75 ± 2.1
32	Acrylonitrile	200	87	91 ± 45

SYN 001 2040

SURROGATE RECOVERY DATA

NO.	SURROGATE	AMOUNT SPIKED (NG)	% RECOVERY	ACCEPTABLE LIMITS
1	D8-Phenol	150	98	
	BFB	105	75	

MIDRIC
05/14/84 10:21:00

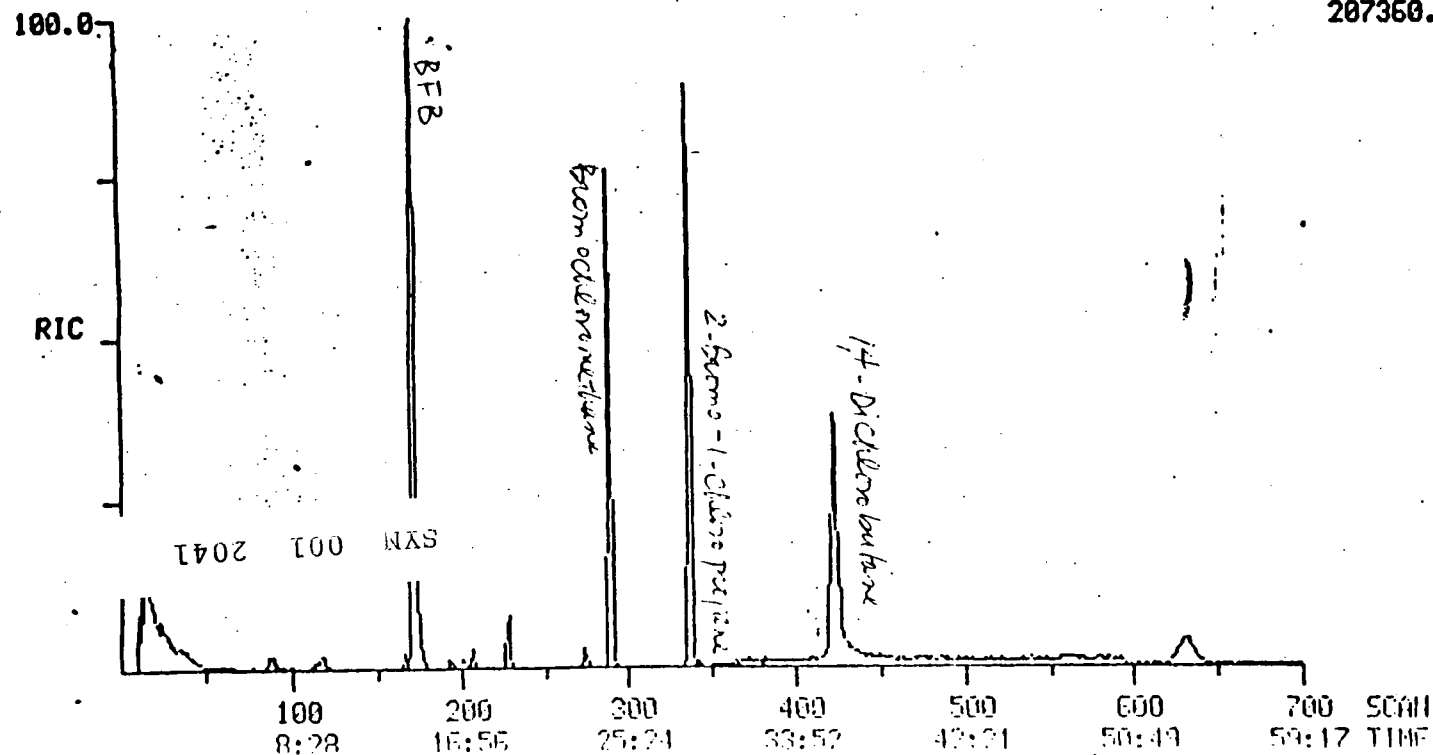
SAMPLE:
CONDS.:

RANGE: G 1, 1 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3

DATA: BLANK514 #1
CALI: BLANK514 #2

SCANS 1 TO 700

207360.

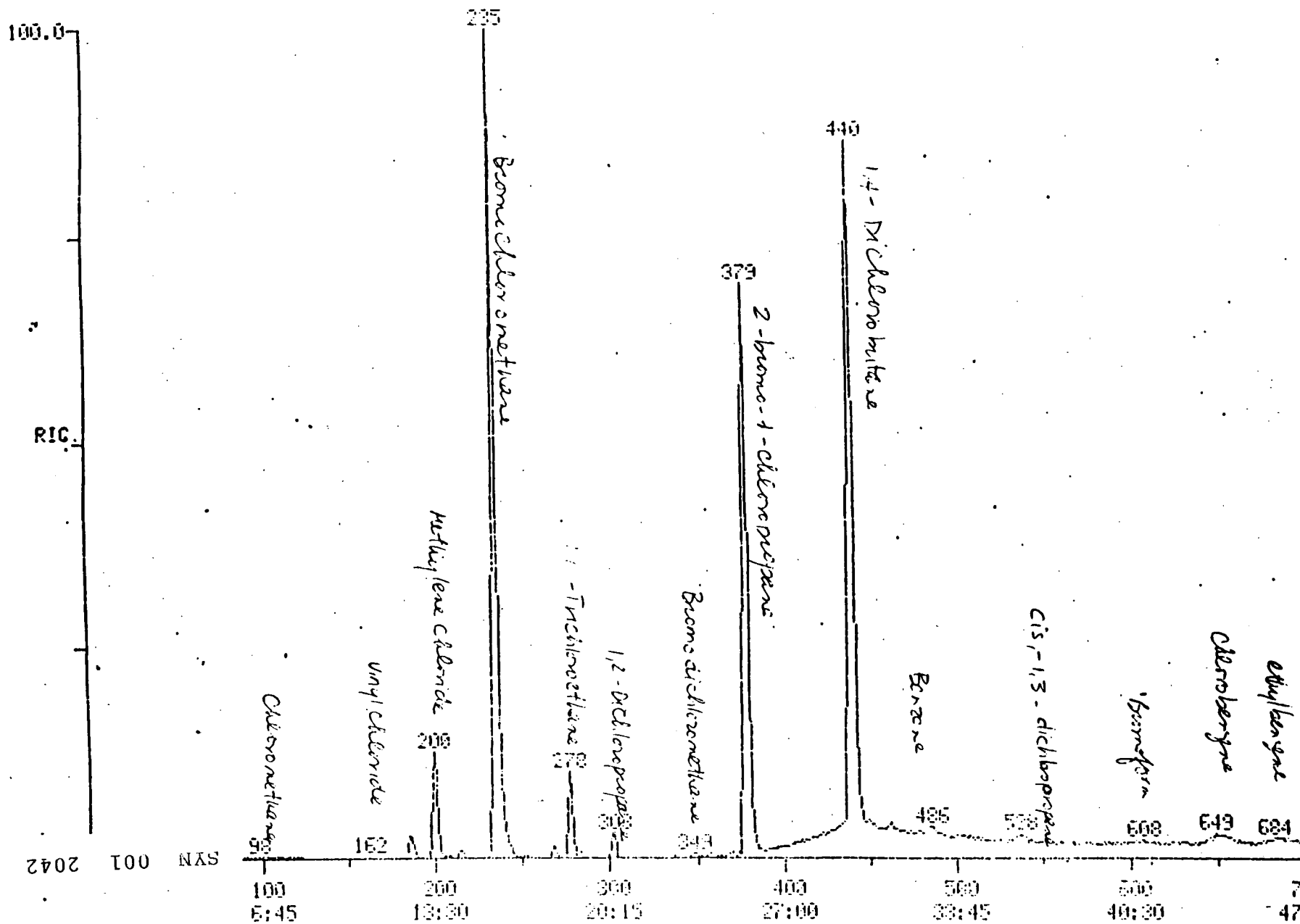


RIC
05/08/84 22:00:00
SAMPLE:

DATA: 958110

SCANS 1 TO 2

265720.



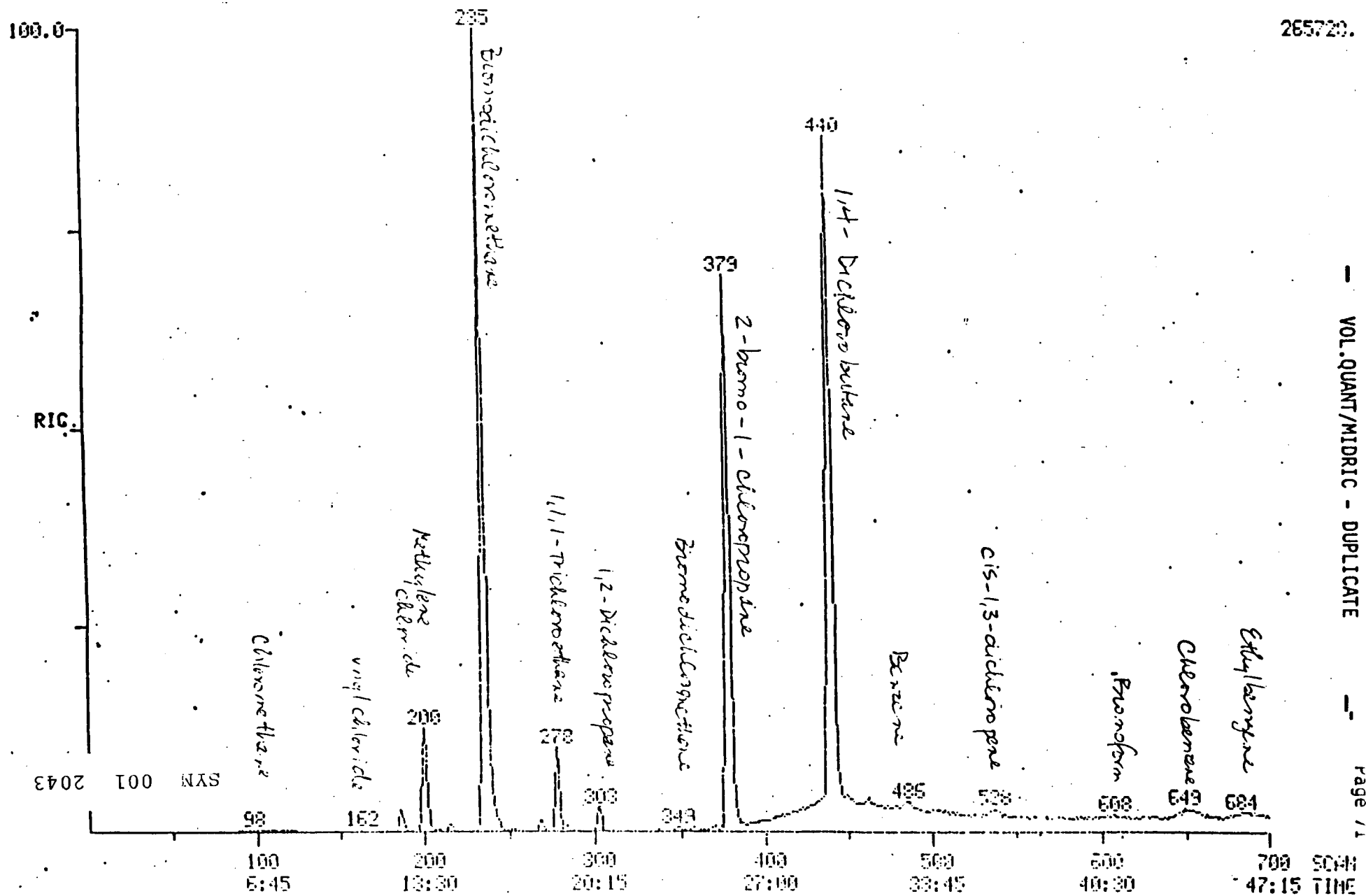
VOL. QUANT/MIDRIC - SAMPLE

RIC
05/03/84 22:00:00
SAMPLE:

DATA: 958110

SCANS 1 700

265720.

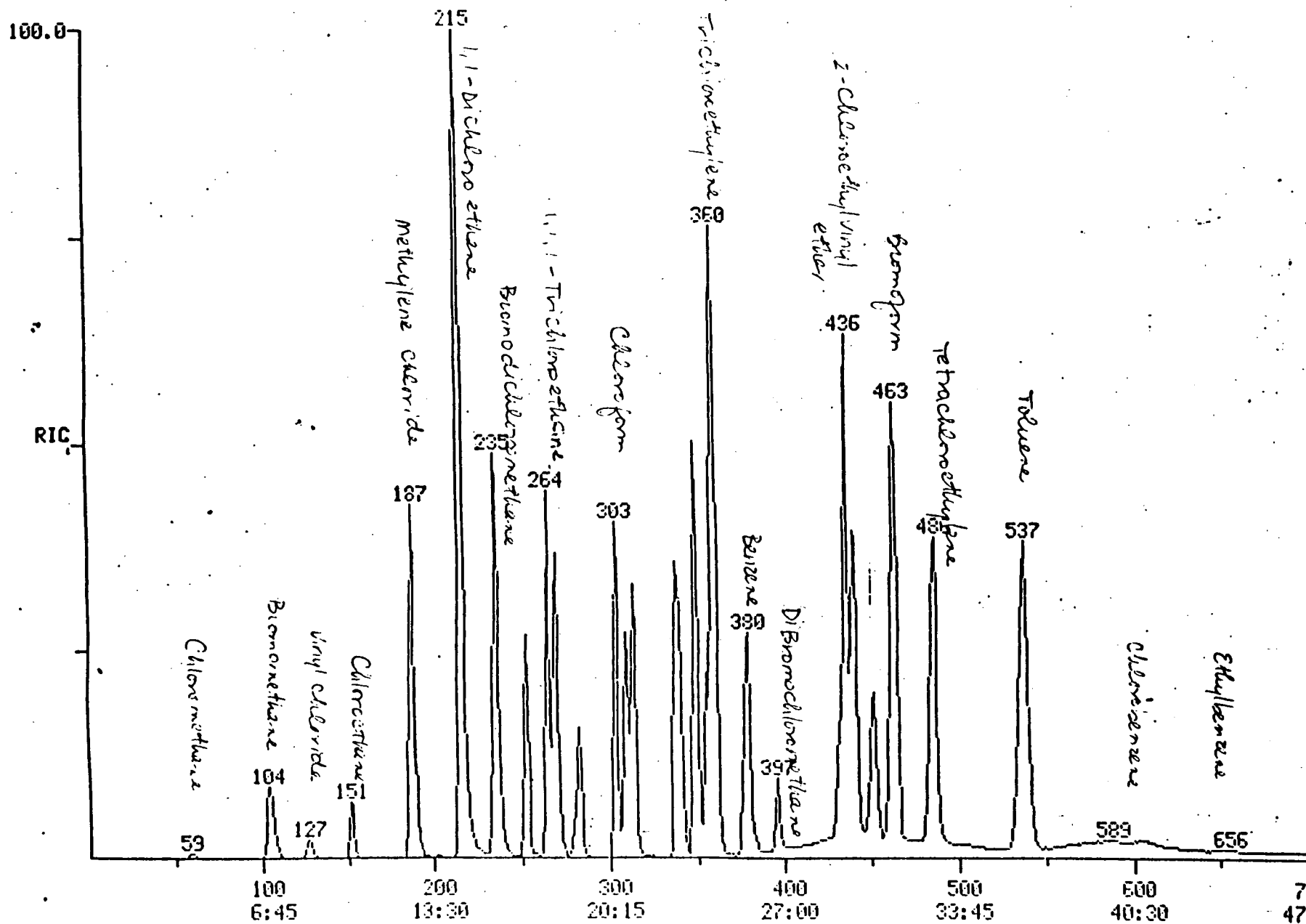


RIC
05/09/94 20:47:00
SAMPLE:

DATA: 9581105

SCANS 1 TC 00

929792.

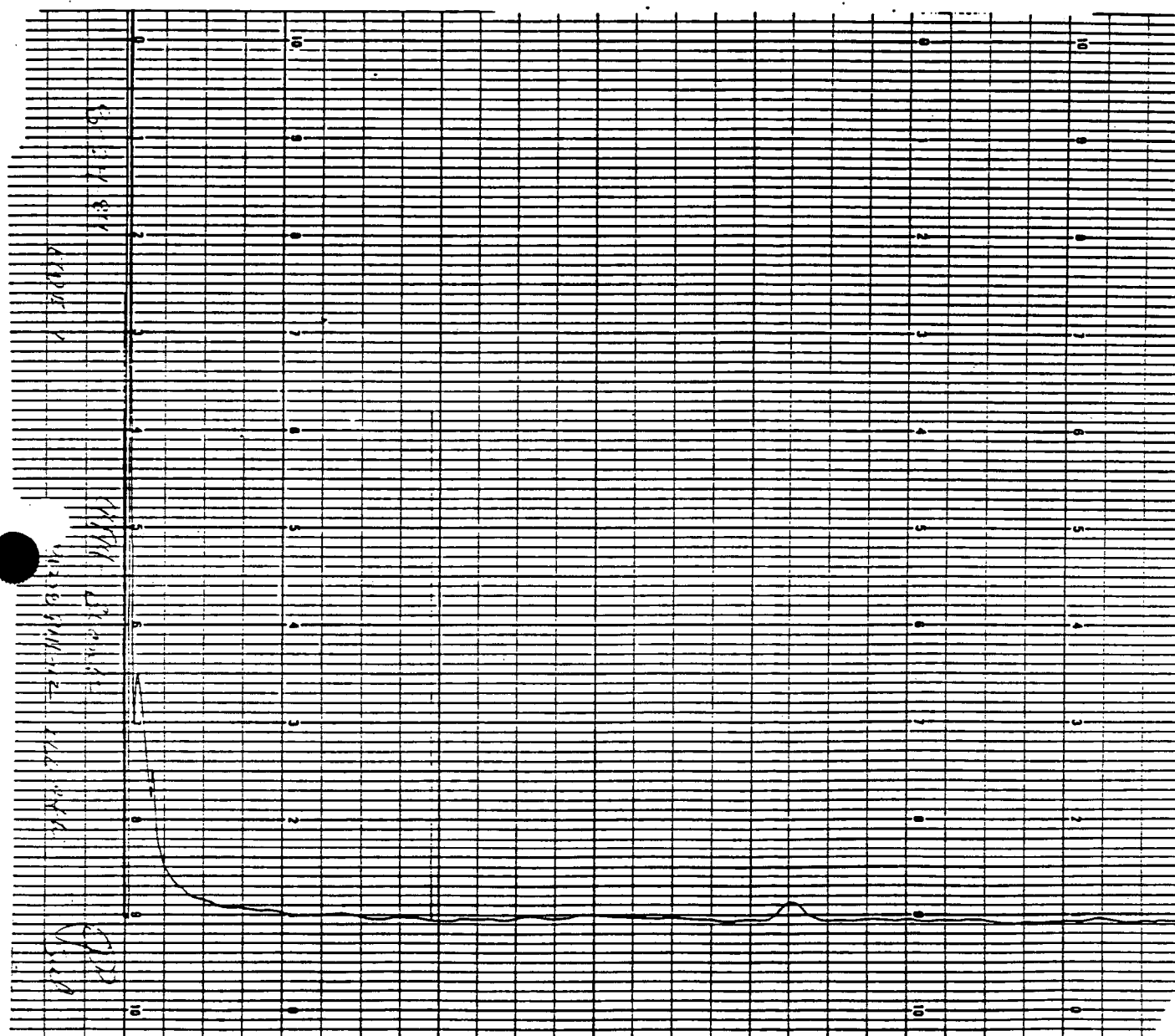


VOL. QUANT/MIDRIC - SPIKE

IV SUPPORTING DOCUMENTS

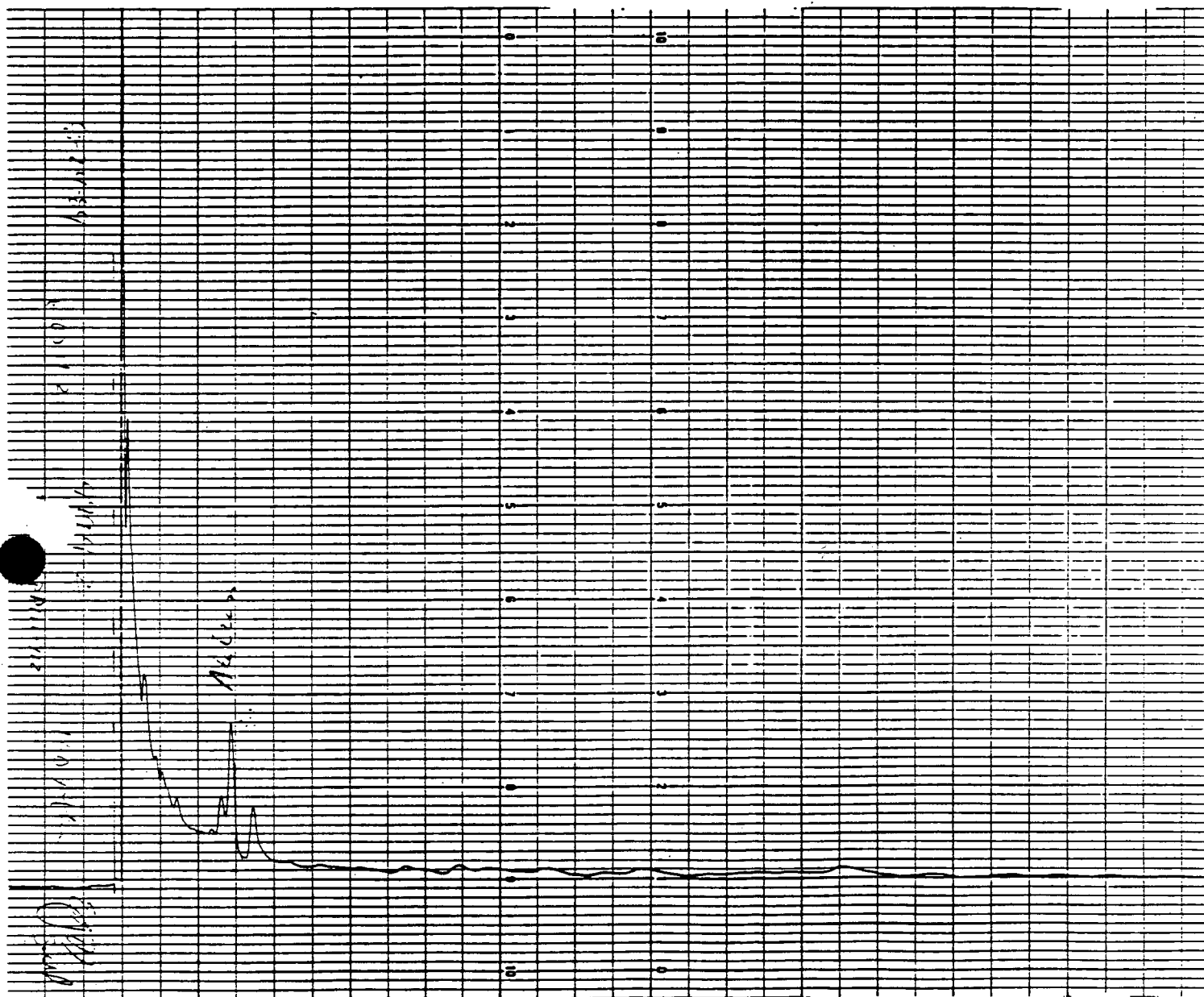
C.. GC CHROMATOGRAMS

SYN 001 2045

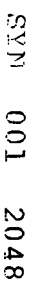


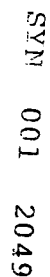
SYN 001 2046

PEST. QUANT. - SAMPLE



SYN 001 2047

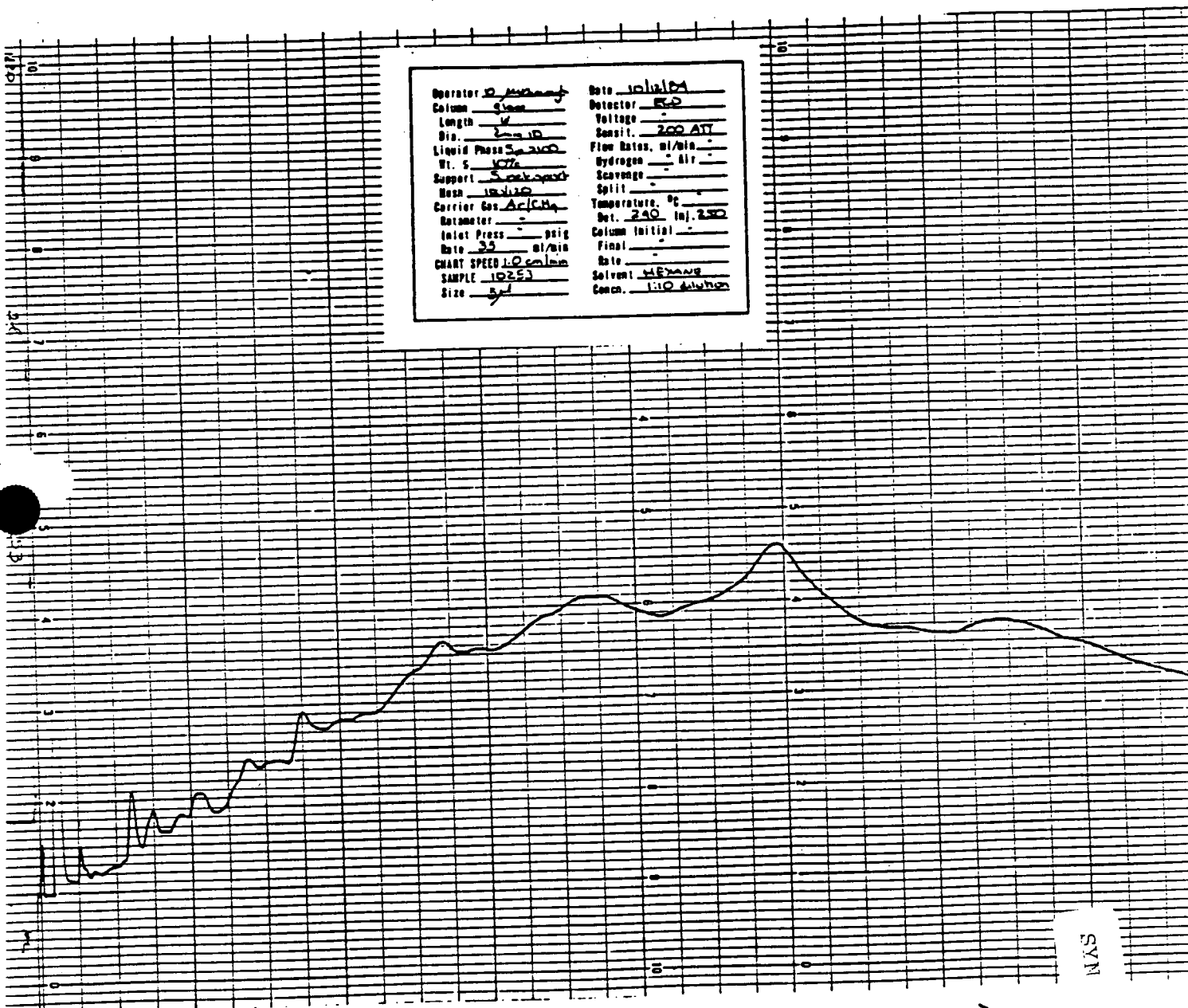




Operator <u>R. M. Smith</u>	Date <u>10/1/61</u>
Column <u>9' x 1/8"</u>	Detector <u>ECG</u>
Length <u>2mm ID</u>	Voltage <u>-</u>
Wt. <u>100m</u>	Sensit. <u>100 ATT</u>
Liquid Phase <u>SO 2400</u>	Flow Rates, ml/min <u>-</u>
Support <u>Supelco</u>	Hydrogen <u>-</u> Air <u>-</u>
Mesh <u>100/120</u>	Scavenger <u>-</u>
Carrier Gas <u>Ar 100</u>	Split <u>-</u>
Rotameter <u>-</u>	Temperature, °C <u>-</u>
Inlet Press. <u>-</u> psig	Det. <u>250</u> Inj. <u>250</u>
Rate <u>3.5</u> ml/min	Column Initial <u>-</u>
CHART SPEED <u>10 cm/min</u>	Final <u>-</u>
SAMPLE <u>10253-blank</u>	Rate <u>-</u>
Size <u>5μ</u>	Solvent <u>HEXANE</u>
	Concn. <u>-</u>

SYN 001 2050

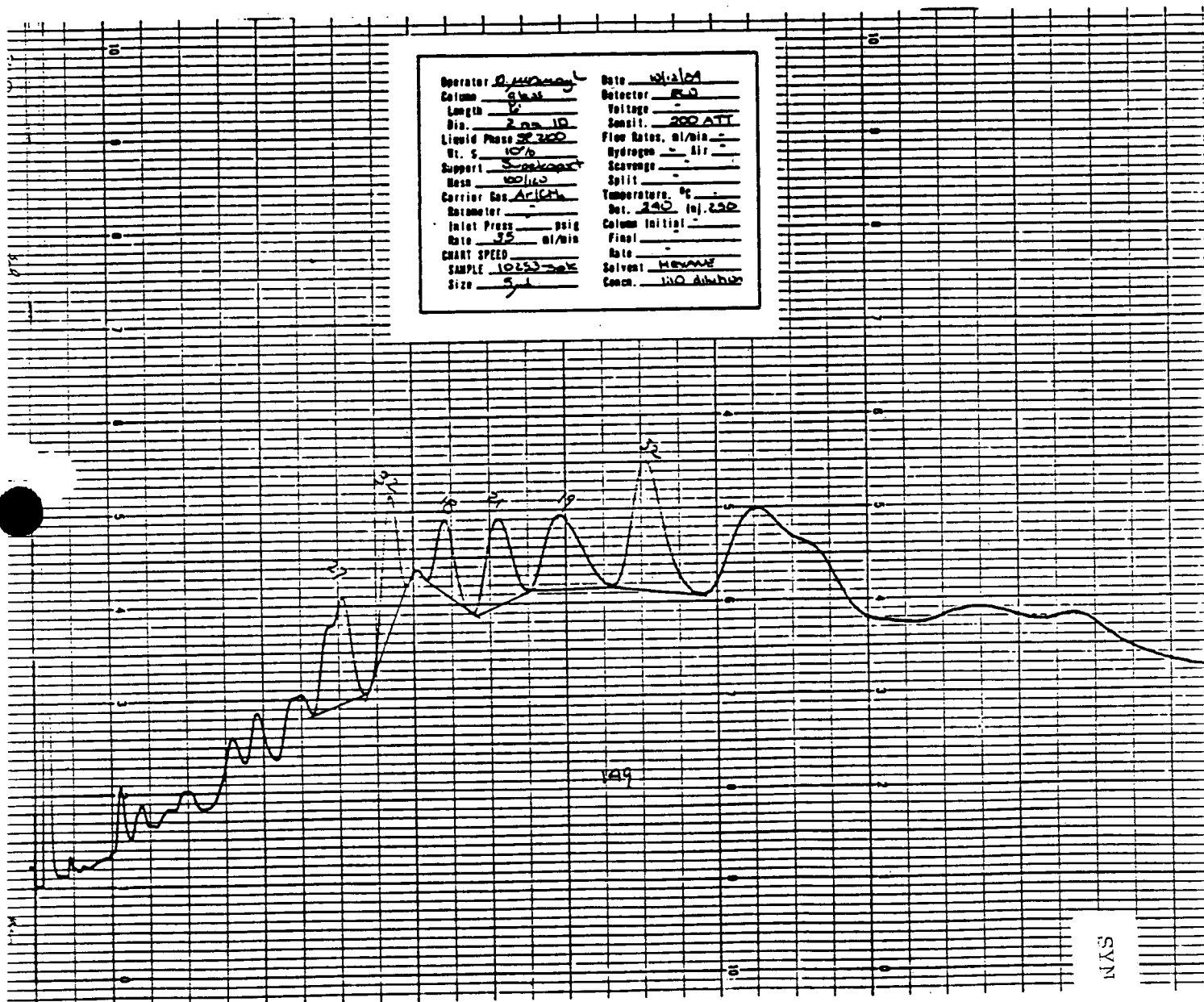
PCB QUANT - SAMPLE

SYM
001
2051

SYN 601 2052

PCB QUANT - SPIKE

Operator <u>B. H. H. H.</u>	Date <u>10/2/64</u>
Column <u>5' x 1/8"</u>	Detector <u>FD</u>
Length <u>5'</u>	Voltage <u>200 ATT</u>
Dia. <u>2 mm ID</u>	Flow Rates, ml/min <u>-</u>
Liquid Phase <u>SE 30</u>	Hydrogen <u>Air</u>
Wt. % <u>10%</u>	Scavenger <u>-</u>
Support <u>Supracarb</u>	Split <u>-</u>
Mesh <u>100/120</u>	Temperature, °C <u>-</u>
Carrier Gas <u>Ar/CH₄</u>	Det. <u>240</u> (adj. <u>230</u>)
Barometer <u>-</u>	Column Initial <u>-</u>
Inlet Press <u>-</u> psig	Final <u>-</u>
Rate <u>35</u> ml/min	Rate <u>-</u>
CHART SPEED <u>-</u>	Solvent <u>Hexane</u>
SAMPLE <u>10223-54K</u>	Concn. <u>100 Aibuhub</u>
Size <u>5-1</u>	



Archlor 1260

SYN 001 2053

IV SUPPORTING DOCUMENTS

E. NBS SEARCH PRINTOUTS

SYN 001 2054

Quantitation Report

File: FLSRG

Data: 99381TV.TI
08/11/84 18:38:00

NBS SEARCH RESPONSE LIST

le:

mitted by:

Analyst:

AMOUNT=AREA * REF.AMNT/(REF.AREA)* RESP.FACT)

Resp. fac. from Library Entry

NO	NAME
1	METHANE, DICHLORO-
2	METHANE, DICHLORO-
3	UNKNOWN
4	METHANOL, TRIFLUORO-, COMPD. WITH N,N-DIMETHYLMETHANAMINE (1:1)
5	UNKNOWN
6	METHANE, BROMOCHLORO-
7	METHANE, BROMOCHLORO-
8	UNKNOWN
9	METHANE, TRICHLORO-
10	ETHANE, 1,1,2-TRICHLORO-1,2,2-TRIFLUORO-
11	ETHANE, 1,1,2-TRICHLORO-1,2,2-TRIFLUORO-
12	UNKNOWN
13	UNKNOWN
14	UNKNOWN
15	NOT IDENTIFIED
16	UNKNOWN
17	UNKNOWN
18	METHANE, TETRACHLORO-
	METHANE, TETRACHLORO-
	NOT IDENTIFIED
19	NOT IDENTIFIED
20	UNKNOWN
21	ETHENE, TRICHLORO-
22	1,5-HEXADIEN-3-YNE
23	1,5-HEXADIEN-3-YNE
24	SILANE, DIMETHOXYDIMETHYL-
25	NOT IDENTIFIED
26	NOT IDENTIFIED
27	NOT IDENTIFIED
28	NOT IDENTIFIED
29	ETHENE, TETRACHLORO-
30	ETHENE, TETRACHLORO-
31	UNKNOWN
32	BENZENE, (TRIFLUOROMETHYL)-
33	1-PROPANOL, 2,2-DIMETHYL-, PROPANOATE
34	UNKNOWN
35	NOT IDENTIFIED
36	NOT IDENTIFIED
37	BENZENEMETHANOL, 3,5-DIMETHYL-
38	SULFOXIDE, METHYL PHENETHYL
39	CYCLOTRISILOXANE, HEXAMETHYL-

SYN 001 2055

NBS SEARCH FIT LIST

DATA FILE: 99381TV

LIBRARY SEARCHED: LIBRARYNB

ENTRY	SCAN	PURITY	FIT	# LIB ENTRIES WITH FIT > 850	# OF SATURATED PEAKS IN SCAN
1	126	975	984	2	0
2	141	267	917	2	0
3	153	299	949	1	0
4	161	68	936	3	0
5	173	764	1000	3	0
6	179	755	971	1	0
7	181	904	981	1	0
8	202	520	996	2	0
9	210	949	964	2	0
10	215	880	965	2	0
12	220	160	997	1	0
14	222	87	992	2	0
15	232	3	29	0	0
16	237	132	992	3	0
18	242	787	938	2	0
20	252	3	29	0	0
21	263	3	24	0	0
22	268	3	19	0	0
23	276	906	988	3	0
24	282	753	937	7	0
25	284	897	950	8	0
26	287	907	976	2	0
27	302	3	71	0	0
28	315	3	32	0	0
29	341	3	46	0	0
31	346	847	983	1	0
33	350	91	999	1	0
34	353	569	942	4	0
35	356	726	873	2	0
36	364	17	987	9	0
37	377	3	78	0	0
38	379	3	78	0	0
39	397	396	850	1	0
40	410	361	941	9	0
41	455	759	956	3	0

SYN 001 2056

RESPONSE/AREA COUNT LIST

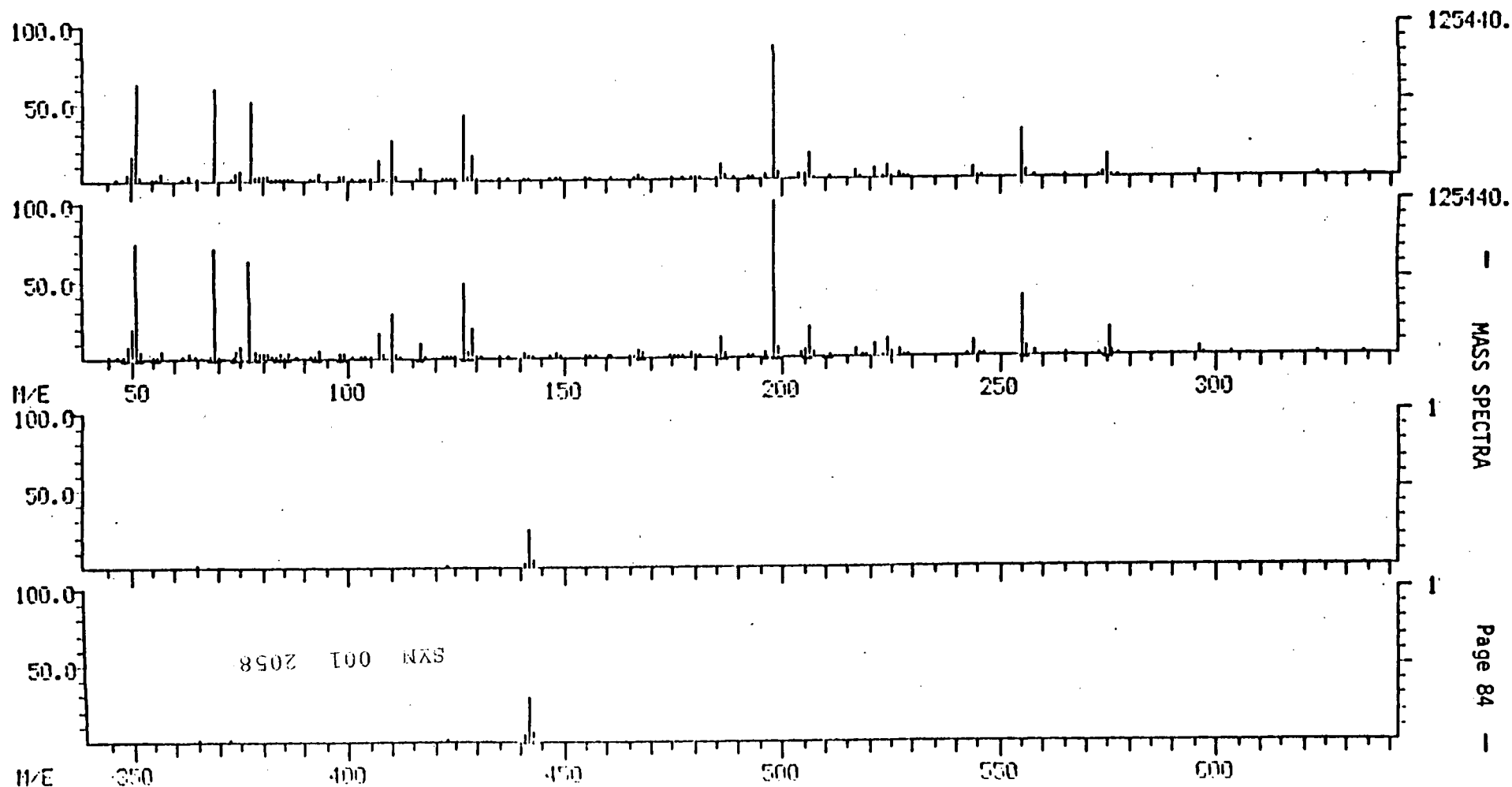
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0	TOT	126	10:41	9	0.600	A BB	21285300.	135.636	33.83
2	TOT	142	12:02	9	0.676	A BB	12896.	0.082	0.02
3	NOT FOUND								
4	TOT	161	13:39	9	0.767	A BB	39055.	0.249	0.06
5	TOT	172	14:35	9	0.819	A BB	704670.	4.490	1.12
6	TOT	181	15:20	9	0.862	A VB	1189580.	7.580	1.89
7	TOT	181	15:20	9	0.862	A BB	1215190.	7.744	1.93
8	TOT	202	17:07	9	0.962	A BB	12724.	0.081	0.02
9	TOT	210	17:48	9	1.000	A BB	15693000.	100.000	24.95
10	TOT	210	17:48	9	1.000	A BV	7003380.	44.627	11.13
11	TOT	215	18:13	9	1.024	A VB	3481820.	22.187	5.53
12	TOT	215	18:13	9	1.024	A BV	13132.	0.084	0.02
13	TOT	220	18:39	9	1.048	A VB	51120.	0.326	0.08
14	TOT	220	18:39	9	1.048	A VB	51120.	0.326	0.08
15	TOT	232	19:40	9	1.105	A BB	170016.	0.000	0.00
16	TOT	232	19:40	9	1.105	A BB	1068.	0.007	0.00
17	TOT	237	20:05	9	1.129	A BB	77456.	0.494	0.12
18	TOT	237	20:05	9	1.129	A BV	53648.	0.342	0.09
19	TOT	242	20:31	9	1.152	A VB	192896.	1.229	0.31
20	TOT	252	21:21	9	1.200	A BB	129736.	0.000	0.00
21	TOT	263	22:17	9	1.252	A BB	187776.	0.000	0.00
22	NOT FOUND								
23	TOT	276	23:23	9	1.314	A BB	1344920.	8.570	2.14
24	TOT	284	24:04	9	1.352	A VB	1968510.	12.544	3.13
5	TOT	284	24:04	9	1.352	A VB	1968160.	12.542	3.13
26	TOT	287	24:19	9	1.367	A BB	1438090.	9.164	2.29
27	TOT	302	25:36	9	1.438	A BB	4620230.	0.011	0.00
28	TOT	315	26:42	9	1.500	A BB	85208.	0.000	0.00
29	TOT	341	28:54	9	1.624	A BB	15840.	0.000	0.00
30	TOT	346	29:19	9	1.648	A BB	197184.	0.000	0.00
31	TOT	346	29:19	9	1.648	A BV	562248.	3.583	0.89
32	TOT	350	29:40	9	1.667	A VB	528320.	3.367	0.84
33	TOT	350	29:40	9	1.667	A BB	322440.	2.055	0.51
34	TOT	353	29:55	9	1.681	A BB	947896.	6.040	1.51
35	TOT	356	30:10	9	1.695	A VB	1571230.	10.012	2.50
36	TOT	364	30:51	9	1.733	A BB	62000.	0.395	0.10
37	TOT	379	32:07	9	1.805	A BB	116744.	0.000	0.00
38	TOT	379	32:07	9	1.805	A BB	124557.	0.000	0.00
39	TOT	397	33:39	9	1.890	A BB	101232.	0.645	0.16
40	TOT	410	34:45	9	1.952	A BB	179917.	1.146	0.29
41	TOT	455	38:34	9	2.167	A BB	834922.	5.320	1.33

SYN 001 2057

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09/10/84 12:07:00 + 12:40
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CONDS.:
GC TEMP: 225 DEG. C -
ENHANCED (S 150 211 0T)

DATA: DFTPPA910 H836
CALI: BLANKD10A H2

BASE M/Z: 15 198
RIC: 917503.7 1114110.



MID LIBRARY SEARCH
 09/10/84 12:07:00 + 12:40
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 CONDS.:
 ENHANCED (S 150 211 0T)

DATA: DETPPA910 # 896
 CALI: BLANK913A # 2

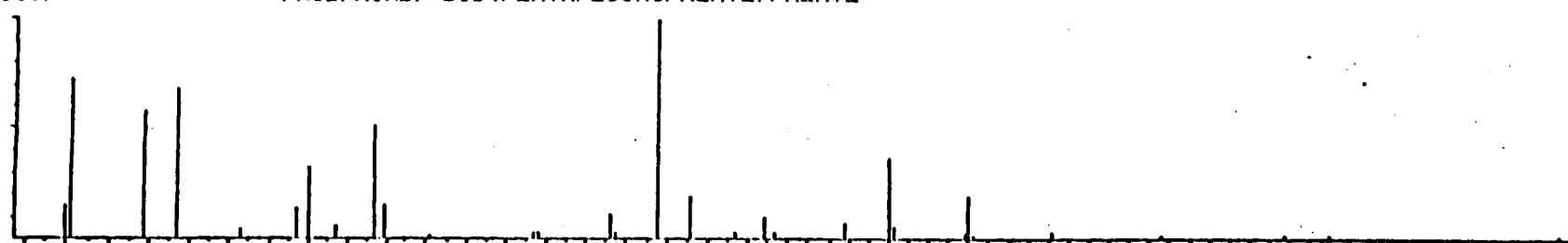
CASE N/Z: 195
 RIC: 842751.



C18.H5.F10.P

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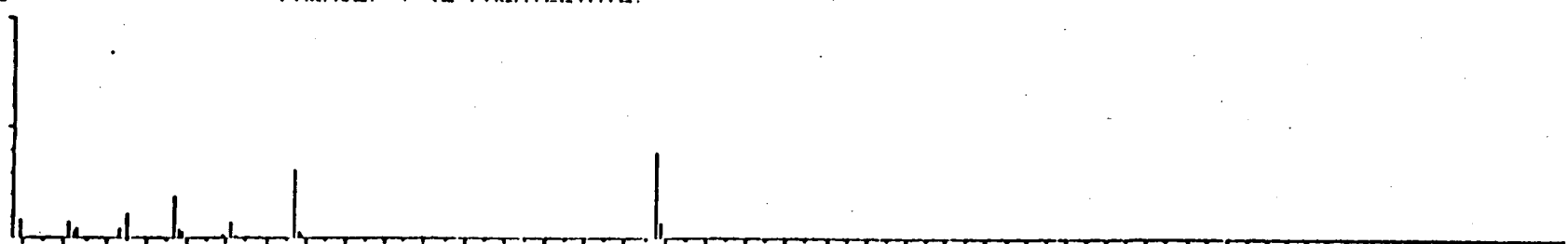
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 B PK 198
 PAIR 1
 III 35196
 PUR 806



C14.H14.0

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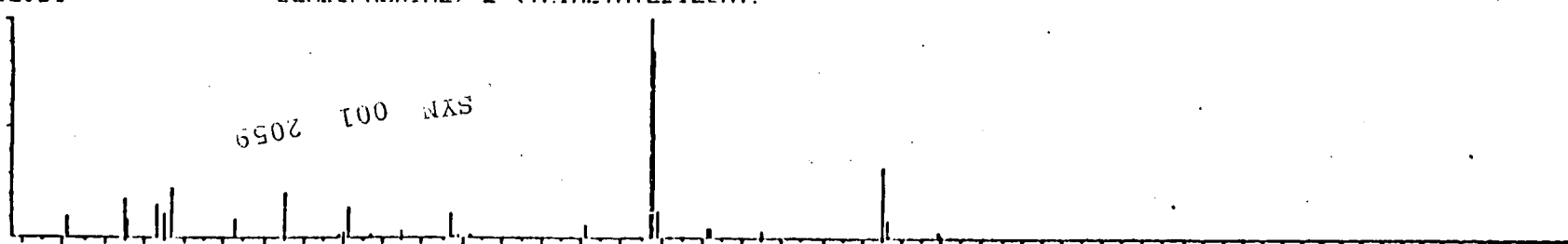
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 PAIR 2
 III 15176
 PUR 202



C16.H18.02.SI

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 B PK 197
 PAIR 3
 III 24101
 PUR 194



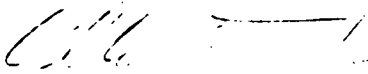
M/Z

50 100 150 200 250 300 350 400

STATEMENT OF QUALIFICATIONS

Prepared for
Envirosphere Company
2 World Trade Towers
90th Floor
New York, NY 10048

April 16, 1985


Allan H. Tordini

SYN 001 2060

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SYN 001 2061

1.0 INTRODUCTION

United States Testing Company, Inc. (USTC) is pleased to submit its Statement of Qualifications to Envirosphere for Environmental Analytical Laboratory Services.

USTC has supplied precise, scientific data to government and industry for more than a century. USTC maintains laboratories in Los Angeles, California; Richland, Washington; Tulsa, Oklahoma; Rochelle, Illinois; Memphis, Tennessee; Reading, Pennsylvania and in Hoboken, New Jersey, the home office. In addition, the company maintains mobile facilities across the nation. For example, USTC's Quality Assurance Services Division has established and manned with technical surveillance employees, mobile laboratory facilities in 43 nuclear and fossil fuel construction sites. Many of these mobile laboratory facilities provide quality assurance evaluations for periods in excess of five years during the construction of large power plants.

The Environmental Chemistry Group has provided water, wastewater and solid and hazardous waste analytical services for more than a decade. The laboratories in Hoboken, New Jersey are certified by the State of New Jersey under Federal EPA regulations and are under contract with the U. S. Environmental Protection Agency for the analysis of soil, water and waste samples from "Superfund" sites for organic and inorganic pollutants.

2.0 CAPABILITIES AND FACILITIES

USTC has one of the most advanced and complete analytical facilities in the United States for the analysis of Priority Pollutants and other environmentally significant contaminants. The Environmental Analysis Laboratory is located in the five-story, 120,000 square-foot central laboratories building, in Hoboken, New Jersey.

All analytical procedures are performed in strict accordance with protocols specified by the EPA, State, local authorities or by the client. Use of the best analytical techniques and State-of-the-Art equipment enables USTC to provide precise and accurate results. Furthermore, USTC maintains a Quality Assurance program which continually monitors all instrumentation, personnel and data.

2.1 FACILITIES

The Environmental Analysis Laboratory of USTC has the following general facilities available:

- * 14,000 square feet of laboratory space
- * 50 cubic feet of -8 C storage
- * 700 cubic feet of 4 C storage
- * 95 linear feet of hood space

- * 400 linear feet of bench space
- * 2000 square feet of storage space
- * limited access laboratory for special extractions
- * separate sample storage, processing, extraction and analysis areas to minimize cross-contamination

2.2 INSTRUMENTATION

The laboratory has all instrumentation in-house to perform a full scope of Environmental Analyses. In the sections which follow, the major equipment owned by the Environmental Chemistry Division is presented.

2.2.1 INSTRUMENTATION AVAILABLE FOR ORGANIC ANALYSES

Gas Chromatography/Mass Spectroscopy

- * The laboratory owns nine (9) Finnigan GC/MS instruments (2 OWA 30 and 7 1020).
- * 9-Track data tape capability for all GC/MS
- * Capillary Column capability for all GC/MS
- * 2 stand-alone data systems for off-line accessibility of data
- * Autosampler capability for extracts and both water and soil purgeables

Gas Chromatography

- * The laboratory owns the following electron capture gas chromatographic equipment, used for the analysis of Pesticides/PCB's:

Hewlett Packard Model 5840 with 5840A recording integrator, microprocessor instrument control and autosampler

Shimadzu Model GC 9A with dual capillary column capability, recording integrator, microprocessor instrument control and autosampler

Shimadzu model GC 9A with dual packed columns, dual electron capture detectors, recording integrator, microprocessor control and autosampler

Additional Gas Chromatography instrumentation available is as follows:

Hewlett Packard Model 5730

Flame Ionization Detector
Electron Capture Detector
Capillary Capability
Recording Integrator
Microprocessor Control

Additional Gas Chromatography Instrumentation (con't)

Two Hewlett Packard Model 5750

Flame Ionization Detector
Electron Capture Detector
Recording Integrator

Varian Model 2700

Dual Flame Detector
Recording Integrator

Varian Model 1400

Thermal Conductivity Detector
Strip Chart Recorder

Shimadzu Model GC 9A

Flame Ionization Detector
Capillary Column
Microprocessor Control
Autosampler

Data Processing Equipment

In addition to data systems dedicated to analytical instrumentation, the laboratory owns four IBM PC/XT systems for data management and quality control purposes. Final written reports of data will be generated by these systems, thereby reducing the likelihood of calculation or transcription errors

The laboratory has an extensive inventory of extraction glassware, and other support equipment and supplies necessary for the extraction and analysis of environmental samples.

2.2.2. INSTRUMENTATION AVAILABLE FOR INORGANIC ANALYSES

Jarrell Ash Model ICAP 9000 Inductively Coupled Plasma Spectroscopic

Autosampler
Microprocessor Control

In addition, the laboratory owns Atomic Absorption Spectrometers (flame and furnace), High Performance Liquid Chromatographs, Infrared Spectrometers, Ultraviolet and Visible Spectroscopes, and Total Organic Carbon Analyzers.

2.3 MAINTENANCE SCHEDULES

USTC's Quality Assurance program includes continual monitoring of instrument response to permit identification of the need for routine instrument maintenance.

2.3.1 GC/MS MAINTENANCE

Area responses are monitored for initial and continuing calibration standards, and significant decreases in these responses indicate the need to clean or replace the ion source. This is cleaned at a minimum of bi-weekly, and more frequently should the need arise.

In addition, routine maintenance is performed weekly. Routine maintenance includes cleaning and/or changing the injection port liner, changing the injection port septum, and removing the top few inches of the capillary column.

Non-routine maintenance is performed as required, and the instruments are covered by service contracts or under warranty. In addition to the generally available manufacturer's service, USTC uses several local consultants as needed to reduce down-time due to instrumental problems.

2.3.2. GC MAINTENANCE

As for GC/MS, area responses are monitored for standards, and decreasing responses are considered an indication of the need to clean the detector. Loss of resolution in standards is used to indicate the need to clean or re-pack packed columns, or to remove the top few inches of capillary columns.

Routine maintenance is performed weekly, and includes changing injection port septa, cleaning or changing injection port liners, etc.

Non-routine maintenance is performed as required, and the instruments are covered by service contracts or under warranty. In addition to the generally available manufacturer's service, USTC uses several local consultants as needed to reduce down-time due to instrumental problems.

2.3.3 ICP and AA MAINTENANCE

The plasma torch from the ICP is cleaned of solids build-up periodically, at a minimum of once per week, and more often if the need is indicated.

3.0 PERSONNEL

The Environmental Chemistry Division consists of a technical staff of 27 individuals and a non-secretarial support staff (data review, QC) of four. Figure 3.1 shows the management structure of the Division. Resumes of key personnel are presented in this section.

8

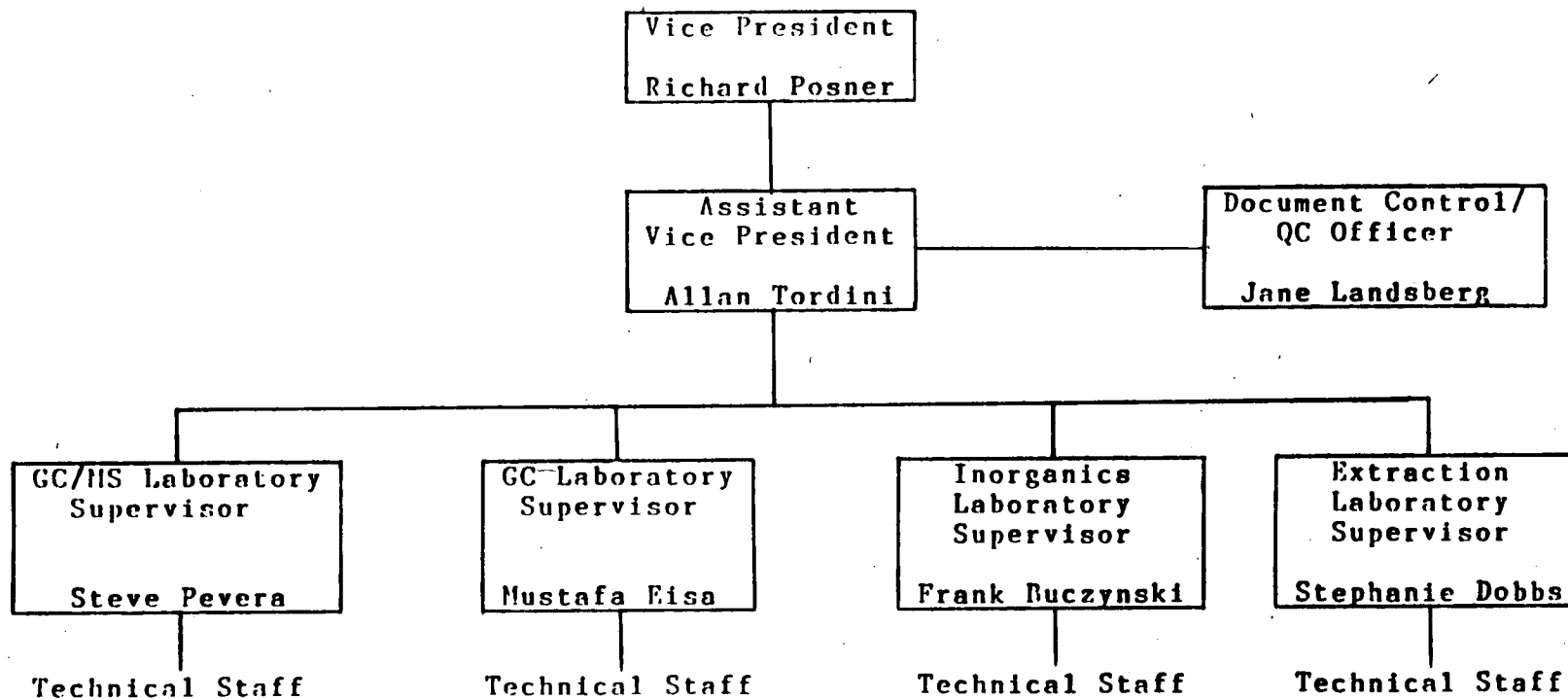


Figure 3.1 Management structure.

R E S U M E

RICHARD POSNER

POSITION

Vice President

RESPONSIBILITIES

Total responsibility for Divisional performance in the analysis of samples submitted for organic and inorganic analysis. Accountable to the United States Testing Company, Inc.

EDUCATION

Ph.D candidate, City University of New York. MAJOR: Chemistry

M.S., Stevens Institute of Technology. MAJOR: Analytical Chemistry

B.S., City University of New York. MAJOR: Chemistry

PROFESSIONAL AFFILIATIONS

Member - ASTM committee D.19

Member - Water Pollution Control Federation

MANAGEMENT EXPERIENCE

1981 - Present, Vice President of Metals and Environmental Chemistry Division, United States Testing Company, Inc.

In this position, Mr. Posner has the authority and responsibility for the technical operation and direction of the laboratory staff. He is responsible for the creation of the GC/MS capabilities within United States Testing Company, Inc. Mr. Posner recruited the staff and organized the GC/MS laboratory to enable USTC to offer these services, and to qualify for participation in the USEPA Contract Laboratory Program.

The company's participation in this program has expanded beyond routine analytical services under several "IFB" contracts to include special analytical services for analysis of TCDD and other non-routine parameters. Under Mr. Posner's direction, USTC has also qualified for and been awarded several bid lots on a contract for analysis of 2,3,7,8-TCDD in soil samples collected from superfund sites.

1978 to 1981 - Assistant Vice President of
Metals and Environmental Chemistry Division,
United States Testing Company, Inc.

Mr. Posner was responsible for the management of the laboratory, and was largely responsible for the development of USTC's Environmental Laboratory. Under his management, the laboratory grew from a small group performing rudimentary wet chemical analyses to a major source of environmental analyses in the U.S.

TECHNICAL
EXPERIENCE

As a former GC/MS operator, Mr Posner has ample experience in the analysis and interpretation of data from environmental samples. Upon USTC's qualification for EPA's Dioxin program, Mr. Posner personally developed the company's program for fulfilling all EPA requirements, including data review guidelines.

PUBLICATIONS

The Analysis of Nitrite in Bacon by Ion Chromatography: Rocky Mountain Symposium, August 1978.

Altering the Dynamic Range of Ion Chromatography Column by Prior Chemical Treatment of Samples: Ion Chromatography Analysis of Environmental Pollutants, Volume 2, 1979.

The Analysis of Cations by Ion Chromatography, From Cements to Vitamins: Rocky Mountain Chromatography Symposium, August, 1980.

The Analysis of Portland Cements by Ion Chromatography: Pittsburgh Conference, 1980.

The Analysis of Ascorbic Acid by Graphite Furnace Atomic Absorption Spectroscopy: Pittsburgh Conference, 1980.

The Analysis of Formaldehyde (as Formate) by Ion Exclusion Chromatography: Pittsburgh Conference, 1981.

The Analysis for the Assay of Barium Sulfate by Anion Ion Chromatography: Rocky Mountain Symposium, 1981.

CERTIFICATIONS

ACS Certification

Instrumentation Laboratories Atomic Absorption Course

Instrumentation Laboratories Graphite Furnace Course

University of Pennsylvania (Wharton Business School) Special Course in Scientific Management.

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R E S U M E

ALLAN TORDINI

POSITION

Manager Environmental Chemistry

RESPONSIBILITIES

Responsible for direct management of analyses performed by the Environmental Chemistry Division of USTC. Accountable to the Divisional Vice President. Includes overall QC responsibility and ensuring adherence to QC specifications. Also responsible for final data review prior to reporting.

EDUCATION

M.S., Rutgers University.
MAJOR: Environmental Science

B.S., Dickinson College. MAJOR: Biology

PROFESSIONAL AFFILIATIONS

Member - ASTM Committees D.19 and E.47

Member - Society of Environmental
Toxicology and Chemistry

Member - Water Pollution Control
Federation

MANAGEMENT EXPERIENCE

1982 - Present, Manager of
Environmental Chemistry, United States
Testing Co., Inc.

Mr. Tordini is responsible for the overall management of the Environmental Chemistry Division. This includes development and implementation of QA systems for the analysis of environmental samples for TCDD and other routine and non-routine parameters.

TECHNICAL
EXPERIENCE

Mr. Tordini has been responsible for the technical management of the Environmental Chemistry Division since 1982. Under his management the division is successfully performing analysis of samples submitted by USEPA for priority pollutants, TCDD, Pesticides and inorganic pollutants by various methods. The methods include 624 and 625 (and contractual modifications), 1624 and 1625 (isotope dilution) and selective ion monitoring by GC/MS. He is ultimately responsible for the validity of data from these analyses and performs final data review prior to reporting of results.

PUBLICATIONS

Schwab, G.M., Tordini, A.M., et al 1981. Feasibility Study for a National Comprehensive Water Quality Monitoring Program. EPA Contract No. 68-03-2909 (NTIS publication pending).

R E S U M E

STEVE PEVERA

POSITION

Laboratory Supervisor

RESPONSIBILITIES

Responsible for overall supervision of activities in USTC's Environmental Analysis Laboratory. Includes management of personnel performing routine extraction and analysis of soil, water and waste samples for Purgeable and Extractable Priority Pollutants, Pesticides, Herbicides, Dioxin and other parameters. Accountable to the Divisional Vice President.

EDUCATION

Graduate Courses, Brooklyn Polytech.
MAJOR: X-ray Chrystallography

B. S., Saint John's University
MAJOR: Chemistry

MANAGEMENT EXPERIENCE

1980 to Present - Laboratory Supervisor

In this position Mr. Pevera is responsible for the management of the personnel in the Environmental Laboratory. This includes supervision of all analyses performed under EPA contract and for commercial clients. He previously served as manager of the USTC Metals and Water Laboratory. In this position he oversaw the activities of chemists performing gas chromatography and classical wet chemical analyses as per EPA Methods.

TECHNICAL
EXPERIENCE

Mr. Pevera has broad experience in Analytical Environmental Chemistry including two years in TCDD extractions. Among the instruments with which Mr. Pevera has 5 or more years' experience are:

Gas Chromatograph
Mass Spectrometers
Atomic Absorption Spectrometers
Electron Microscope

R E S U M E

SEYED DASTGHEYB

POSITION

GC/MS Operator

RESPONSIBILITIES

Responsible for analysis of Acid/Base/Neutral compounds by capillary column GC/MS in accordance with specified methods. Accountable to Laboratory Supervisor.

EDUCATION

Ph. D. candidate, Rutgers University

M.S., Montclair State College. MAJOR: Analytical Chemistry.

B.S., Montclair State College. MAJOR: Chemistry

PROFESSIONAL AFFILIATIONS

Member - American Chemical Society

Member - Montclair Science Club

TECHNICAL EXPERIENCE

1983 - Present, GC/MS Operator, United States Testing Co., Inc.

Responsible for the operation of a Mass Spectrometer/Gas Chromatograph Micro Analytical System for analysis of extractable organics and the use of radioactive isotope tagging techniques for qualitative and quantitative analysis of Volatiles and Extractables in wastewaters. Previous experience with techniques outlined in ASTM, EPA and USP manuals for analysis of organic and inorganic constituents.

RESEARCH

Development of technique using AAGF, GC, IR for Determination of Toxic Substances in Fish Liver.

R E S U M E

MOHAMMAD AMIRSOLEYMANI

POSITION

GC/MS Operator

RESPONSIBILITIES

Responsible for the analysis of samples for extractable organic compounds in accordance with specified methods. Accountable to Laboratory Supervisor.

EDUCATION

Ph.D. Candidate, City University
MAJOR: Organic Chemistry

M.S., Long Island University
MAJOR: Organic Chemistry

B.S., Razi University, MAJOR: Chemistry

TECHNICAL EXPERIENCE

1981 to Present, Mass Spectroscopist,
United States Testing Co., Inc.

Responsible for analysis of water, waste water and solid waste samples by EPA methods. Mr. Amirsoleymani's instrumentation experience includes GC/MS, GC, Atomic Absorption Spectrophotometry and Infrared Scanning Spectrophotometry.

RESEARCH

Inhibition of Urease by Hydroxamic Acid

R E S U M E

DOREEN HACKETT

POSITION

GC/MS Operator

RESPONSIBILITIES

Responsible for analysis of environmental samples by specified methods. Accountable to Laboratory Supervisor.

EDUCATION

B.S. Hunter College (CUNY) MAJOR:
Environmental Chemistry, 1980

TECHNICAL EXPERIENCE

1983 - Present, Mass Spectroscopist
United States Testing Co., Inc.

Responsible for analysis of environmental samples for priority pollutants by GC/MS for commercial and governmental clients. Analytical techniques used include purge and trap, selective ion monitoring, isotope dilution and conventional capillary column analysis for volatile and extractable organics. Also responsible for operation of GC/MS for analysis of samples for TCDD.

1981 -1983, Gas Chromatographer

Responsible for the gas chromatographic analysis of volatile compounds by purge and trap and extractable pesticides and herbicides by GC/EC. Included appropriate quality control and data review responsibilities

1980 - 1981, Chemist

Responsible for water, wastewater and sludge analysis using EPA methods. Instrumentation included: Inductive Coupled Plasma, Atomic Absorption, Graphite Furnace, Infrared Spectrophotometer, Ion Chromatograph, and Total Organic Carbon Analyzer.

SYN 001 2079

R E S U M E

FRANK BUCZYNSKI

POSITION

Inorganic Laboratory Supervisor

RESPONSIBILITIES

Responsible for supervision of metals by ICP and flame and flameless AA. Accountable to Laboratory Supervisor.

EDUCATION

B.A. Queens College (CUNY)
MAJOR: Chemistry, 1978

TECHNICAL EXPERIENCE

1983 - Present, Inorganic Lab Supervisor,
United States Testing Co., Inc.

Mr. Buczynski has been supervising USTC's participation in the EPA Contract Laboratory Program for inorganic analyses since 1983. He was responsible for the installation of a new ICP instrument and two additional furnace AA systems. He functions as the primary liason between USTC and EPA on contractual matters for all inorganic contracts.

1980 -1983, Laboratory Supervisor at a
Commercial Laboratory

Mr Buczynski supervised a commercial laboratory performing instrumental inorganic analyses and conventional wet analyses on environmental samples. He was in part responsible for the laboratorie's entrance into the EPA Contract Laboratory Program, and performed all ICP analyses under the resulting contracts.

SYN 001 2080

R E S U M E

MUSTAFA H. EISA

POSITION

GC Operator

RESPONSIBILITIES

Responsible for analysis of Pesticides/Pcb's using GC, by EPA method 8150. Accountable to Laboratory Supervisor.

EDUCATION

Currently pursuing B.S., Jersey City State College. MAJOR: Chemistry

TECHNICAL
EXPERIENCE

1980 to Present, Gas Chromatographer
United States Testing Co., Inc.

Mr. Eisa has been analyzing environmental samples for pesticides/PCB's and herbicides for more than three years. His responsibilities have included preparation and analysis of soil, oil, water and atmospheric samples for pesticides, herbicides and other environmental contaminants.

R E S U M E

STEPHANIE DOBBS

POSITION

Extraction Laboratory Supervisor

RESPONSIBILITIES

Responsible for overall supervision of activities in USTC's organic extraction laboratory. Direct performance of non-routine extractions, sample preparation and clean-up. Under Ms. Dobbs supervision, the laboratory personnel extract samples in accordance with EPA Contract Laboratory Program protocols in a manner which permits adherence to Q.C. criteria for matrix and surrogate spike recoveries and duplicate analyses. Accountable to Laboratory Supervisor.

EDUCATION

M.S., New York University
MAJOR: Public Administration

B.S., St. John's University
MAJOR: Toxicology

EXPERIENCE

1983 - Present, Extraction Laboratory
Supervisor, United States Testing Co., Inc.

Responsible for overall supervision of activities in USTC organic extraction laboratory. Ms. Dobbs supervises a staff of 5 extraction chemists in the preparation of samples for analysis for acid and base/neutral extractables, TCDD, herbicides and pesticides.

1982 - 1983 Extraction Laboratory Supervisor
for Commercial Laboratory

Responsible for overall supervision of organic extractions for a commercial laboratory involved in the EPA Contract Laboratory Program.

RESUME

JANE LANDSBERG

POSITION

Document Control Officer

RESPONSIBILITIES

Ensuring adherence to chain-of-custody and sample and data documentation requirements. Includes logging in of samples, maintenance of sample tracking records, documenting traceability of standards, establishing job-file inventories and compiling final reports. Accountable to Division Manager.

EDUCATION

Currently pursuing B.S., Montclair State College. MAJOR: Chemistry

EXPERIENCE

1983 - Present, Document Control Officer for EPA Contracts 68-01-6726, 68-01-6780 and 68-01-6963.

In this position Ms. Landsberg is responsible for ensuring the laboratory's compliance with all contractual requirements for documentation and chain-of-custody. She was responsible for the development of USTC's Standard Operating Procedures for standards traceability, sample tracking, file documentation and instrumentation logs.

4.0 QUALITY ASSURANCE

USTC has an ongoing QA/QC program which meets all requirements of the EPA Contract Laboratory Program. Quality Assurance measures include the addition of surrogate compounds to all samples to be analyzed for Organic Priority Pollutants. In addition, with each batch of samples submitted to USTC (or with every 20 samples, whichever is greater) one matrix spike, one duplicate matrix spike (or duplicate sample analysis for metals) and one reagent blank are analyzed.

The surrogate compounds used and the expected recovery limits for each are given in Table 4.1

TABLE 4.1 Surrogates and Recovery Limits

<u>Surrogate</u>	<u>Water Recovery limits</u>	<u>Sediment Recovery limits</u>
Toluene-d8	86-119%	50-160%
4-Bromofluorobenzene	85-121%	50-160%
1,2-dichloroethane-d4	77-120%	50-160%
Nitrobenzene-d5	41-120%	20-140%
2-Fluorobiphenyl	44-119%	20-140%
p-Terphenyl-d14	33-123%	20-150%
Phenol-d5	15-103%	20-140%
2-Fluorophenol	23-121%	20-140%
2,4,6-Tribromophenol	10-130%	10-140%
Dibutylchlorendate	48-136%	20-150%

Limits for Dibutylchlorendate are considered advisory only, and recoveries outside of the expected limits do not require re-analysis of the sample. All other surrogate recovery limits are considered mandatory, and samples in which recoveries are outside of these limits are re-analyzed.

The compounds/elements which are spiked into samples, along with expected recovery limits are presented in Table 4.2.

Table 4.2 Spikes and Recovery Limits

<u>Compound</u>	<u>Water Recovery Limit</u>	<u>Sediment Recovery Limit</u>
1,1-Dichloroethane	61-145	59-172
Trichloroethene	71-120	62-137
Chlorobenzene	75-130	60-133
Toluene	76-125	59-139
Benzene	76-127	66-142
1,2,4-Trichlorobenzene	39-98	38-107
Acenaphthene	46-118	31-137
2,4-Dinitrotoluene	24-96	28-89
Di-n-butyl Phthalate	11-117	29-135
Pyrene	26-127	35-142
N-Nitroso-di-n-propylamine	41-116	41-126
1,4-Dichlorobenzene	36-97	28-104
Pentachlorophenol	9-103	17-109
Phenol	12-89	26-90
2-Chlorophenol	27-123	25-102
4-Chloro-3-Methylphenol	23-97	26-103
4-Nitrophenol	10-80	11-114
Lindane	56-123	46-127
Heptachlor	40-131	35-130
Aldrin	40-120	34-132
Dieldrin	52-126	31-134
Endrin	56-121	42-139
4,4'-DDT	38-127	23-134
All Metals	75-125	75-125

Expected precision, as measured by relative percent difference (RPD) is as listed in Table 4.3.

Table 4.3 Expected Precision

<u>Compound</u>	<u>Water RPD</u>	<u>Soil RPD</u>
1,1-Dichloroethane	14	22
Trichloroethene	14	24
Chlorobenzene	13	21
Toluene	13	21
Benzene	11	21
1,2,4-Trichlorobenzene	28	23
Acenaphthene	31	19
2,4-Dinitrotoluene	38	47
Di-n-butyl Phthalate	40	47
Pyrene	31	36
N-Nitroso-di-n-propylamine	38	38
1,4-Dichlorobenzene	28	27
Pentachlorophenol	50	47
Phenol	42	35
2-Chlorophenol	40	50
4-Chloro-3-Methylphenol	42	33
4-Nitrophenol	50	50
Lindane	15	50
Heptachlor	20	31
Aldrin	22	43
Dieldrin	18	38
Endrin	21	45
4,4'-DDT	27	50
All Metals	20	20

4.1 CALIBRATION PROCEDURES

All instruments are calibrated prior to analysis of any samples. The sections that follow describe calibration procedures used for each instrument.

4.1.1 GC/MS

Initially, the instruments are tuned to EPA specifications for spectral abundances for Bromofluorobenzene (Volatiles) or Decafluorotriphenylphosphine (ABN's). Once tuned, the instruments are calibrated by analyzing five concentrations of all contaminants of interest, spanning a range of 10-200 ug/l. Response factors for each contaminant, relative to an internal standard, are calculated, and must meet criteria prior to analysis of samples.

Once calibration is complete, it is verified every 12 hours by the analysis of a single mid-range standard. Again, response factors are calculated, and must meet criteria prior to further analysis of samples.

4.1.2 GC

Pesticides are analyzed by the external standard method. One standard of all compounds of interest is analyzed each 24 hours, with periodic calibration checks analyzed throughout the 24 hour period.

4.1.3 INDUCTIVELY COUPLED PLASMA

The ICP spectrometer is calibrated at the beginning of each 24 hour period by analysis of standards of known concentration. Calibration is verified by analysis of an EPA QC sample of known concentration immediately after calibration and after each 10 samples are analyzed. In addition, at the beginning and end of each shift, an interfering element calibration verification is performed. This consists of the analysis of a solution containing low concentrations of parameters of interest in the presence of high concentrations of interfering elements.

4.1.4 ATOMIC ABSORPTION SPECTROSCOPE

The AA spectrometer is calibrated using standards of known concentration and verified by the analysis of EPA QC standards of known concentration. Calibration is verified after analysis of every 10 samples by analysis of a known standard.

4.2 ANALYTICAL PROCEDURES

All procedures where applicable, are those currently required for analyses being performed for USEPA, under the USEPA Contract Laboratory Program (CLP). The procedures can be found in the following documents:

Organic Analyses: Solicitation WA 84 A267, issued by USEPA, resulting in contract 68-01-6963.

Inorganic Analyses: Solicitation WA 84 J091, issued by USEPA, resulting in contract 68-01-6631.

4.3 Standard Operating Procedures

USTC has established Standard Operating Procedures (SOP's) for all aspects of the analyses of interest, including sample tracking, chain of custody, data review and documentation control. The SOP's applicable to this project are presented in this section. These are presented only as generic examples. If necessary, additional SOP's, specific to this project, will be developed.

Standard Operating Procedures for Chain of Custody

A. Sample Receipt

All samples delivered to USTC are to be accepted by the Receiving Department in the following manner:

- I Sample container is removed from delivery vehicle and brought into the receiving area.
- II Sample container is checked for any obvious damage.
- III Delivery receipt, driver manifest and/or bill of lading are checked for accuracy and signed by receiving personnel noting any discrepancies or obvious damage.
- IV A numbered USTC Receiving Report is assigned and completed by the receiving personnel. The following information must be entered on the Receiving Report:
 - date of receipt
 - time of receipt
 - name of individual to whom samples are to be released (Sample Custodian)
 - department to which samples are to be delivered
 - received from:
 - shipped from:
 - delivered by:
 - number of containers in shipment
 - description of materials
 - signature of receiving clerk

See Figure 4.1 for an example of a properly completed Receiving Report.

Figure 4.1 Receiving report.

RECEIVING REPORT									
PURCHASE ORDER NO.		FOR DEPARTMENT CHEMISTRY				NO. * 15869			
RECEIVED FROM U.S. EPA									
SHIPPED FROM LAS VEGAS, NV									
DELIVERED BY FEDERAL						CHARGES PAID \$			
FREIGHT ☐		EXPRESS ☒		PARCEL POST ☐		UNITED PARCEL ☐		DELIVERED ☐	
PICK UP ☐		CHARGES COLLECT ☐							
CARRIED BY		WEIGHT		PARTIAL ☐ COMPLETE ☐		INVOICE NO.		DATE OF INVOICE / /	
QUANTITY		DESCRIPTION OF MATERIALS						WEIGHT ENTERED	
		DOLE - SAMPLES							
		FOR							
		A. TOEDINI							
BAGS		CASES		PACKAGES		CRATES		BUNDLES	
REMARKS									
DATE 10/1/83 RECEIVING CLERK James Sandberg									
TIME 10:00 AM									

SYN 001 2090

- V. Delivery is recorded in Receiving Dept. delivery log noting date and time of delivery, mode of shipment, who the sample is from, who the sample is consigned to, and the number of the Receiving Report assigned.
- VI. If custody of cooler is transferred from the person receiving shipment to one or more intermediates prior to delivery to the Sample Custodian, a Chain of Custody Sheet must be completed, and must reflect every change of custody. An example of the Chain of Custody Sheet is shown in Figure 4.2.
- VII. Sample container is delivered directly to the Sample Custodian or designated alternate who will sign the Receiving Report, and Chain of Custody Sheet (if present), return one copy to receiving personnel and retain the remaining copy for the permanent case file.
- VIII. Receiving personnel will then file the copy of the Receiving Report signed by the Sample Custodian in the numerical filing system in the Receiving Dept. office. If custody of the cooler was transferred prior to delivery to the Sample Custodian and a Chain of Custody form was used, then a copy of this should be attached to the Receiving report.
- IX. The sample container is now the responsibility of the Sample Custodian or designated alternate. This individual must log the sample in and open the case file.

CHAIN OF CUSTODY RECORD FOR SAMPLE RECEIPT

Receiving report # _____

Airbill # _____

of coolers in shipment _____

Shipment accepted by:	Date/Time	Rec'd by	Date/Time
Relinquished by:	Date/Time	Rec'd by	Date/Time
Relinquished by:	Date/Time	Rec'd by	Date/Time
Relinquished by:	Date/Time	Rec'd by	Date/Time
Relinquished by:	Date/Time	Rec'd by	Date/Time
Relinquished by:	Date/Time	Rec'd by	Date/Time

Figure 4.2

B. Log-In

Upon notification of sample receipt, the Sample Custodian will sign internal Receiving Report, return pink copy to Receiving Dept., retain remaining copies for case file, and proceed with sample inspection and log-in as follows:

- I. Examine the shipping container(s) and record in the Sample Receipt Log:
 - A. Presence/absence of custody seal(s) on the shipping container(s)
 - B. Condition of custody seal(s)
- II. Open shipping container(s), remove the enclosed sample documents, and record in the Sample Receipt Log:
 - A. Presence/absence of Chain of Custody Record(s)
 - B. Presence/absence of SMO forms
 - C. Presence/absence of airbills and/or bills of lading documenting shipment of samples
- III. Remove sample containers and record in Sample Receipt Log:
 - A. Condition of samples (intact, broken, leaking, etc.)
 - B. Presence/absence of sample tags

If sample tags are present:

- C. Record sample tag numbers
- D. Compare with Chain of Custody Record(s)
 1. Record agreement/disagreement or
 2. Record the fact that sample tag numbers are not listed on Chain of Custody Record(s)

IV. Compare the following documents to verify agreement of information:

- A. Chain of Custody Record(s)
- B. Sample Tag(s)
- C. SMO form(s)
- D. Airbill(s) or Bill(s) of Lading

Record agreement and/or any discrepancies. If discrepancies are found, contact SMO for clarification and notify appropriate lab personnel.

- V. If all samples recorded on the Chain of Custody Record were received by the lab and no problems were observed with sample shipment, sign the Chain of Custody Record in the "received for laboratory by" box of the document. If problems are noted, sign for shipment and record problems in remarks box. Call SMO immediately at (703) 557-2490 to resolve any problems.

Figure 4.3 presents an example of a properly completed sample log sheet.

VI. Sample Numbering

An internal numbering system shall be used for identification of all samples as follows:

Each case will be assigned a project number of the 77000 series. These shall be assigned sequentially upon receipt (77000, 77001, etc.).

Each sample shall be assigned an identifying number within the assigned project number sequence, i.e., sample #1 of project 77000 will be identified as 77000-1, etc.

Internal sample numbers are assigned by the Sample Custodian and are recorded in the Sample Receipt Log alongside the corresponding EPA sample numbers.

Assigned numbers are then stamped on labels and affixed to each sample container.

Properly labeled sample containers are placed in the secure storage area.

11 SIGNATURE: Jane J. J. J.
11 APPROPRIATE RESPONSE

DOCUMENT CONTROL : 1111-06 04

My Seal

-of-Custody

: says

Tag Numbers

• 1800S

(present) absent
(intact) not intact

present/absent

present absent

(listed) not listed on chain-of-custody

present/absent

CASE NUMBER 1111

AIRBILL NUMBER 1234569870

QC Report Number 13

[illegible]

560Z 100 NXS

Figure 4.3 Sample log sheet.

C. Storage

Once all samples are logged in, they are stored in a secure short-term storage area. Shelves are to be clearly labeled, and location information is recorded on the Analysis Instruction Sheet for the appropriate laboratory personnel by the Sample Custodian upon completion of sample log-in and labeling procedure.

If a sample is removed from this area it must be indicated on the Sample Tracking Sheet along with the initials of the person now in custody of the sample, date and time of removal, and reason for removal. All samples must be returned to this area at the end of each working day and the date and time recorded. See Figure 4 for an example of a Sample Tracking Record.

Samples are held in this area for a period of approximately 30 days following receipt.

Once extracted, samples will be moved to the long-term storage area. This is a secure area with limited access, where samples will be held when the need for repeated access is no longer anticipated. Sample tracking records will be maintained for samples in this storage area.

All sample extracts will be stored in the laboratory in a secure area.

SOP for Safe Handling of TCDD Standards and Wastes

Safe handling practices must be observed during all aspects of sample and standard preparation, storage, and disposal.

A. Personnel Practices

1. Protective Clothing: A fully fastened laboratory coat is to be worn in the laboratory area in which TCDD is being used. Clean clothing will be provided weekly and should not be worn outside the laboratory area. Clothing overtly contaminated by TCDD should be removed immediately and disposed of or decontaminated prior to laundering. Disposable Latex or PVC gloves are to be worn when handling TCDD samples or standards. Disposable gloves are to be discarded after each use and immediately after overt contact with chemical TCDD.
2. Eye Protection: Safety goggles or glasses will be made available and must be used in the laboratory work area.
3. Eating, Drinking, and Smoking: There is to be no eating, drinking, smoking, chewing of gum or tobacco, application of cosmetics, or storage of utensils, food, or food containers in laboratory areas where TCDD samples or standards are used or stored.
4. Pipetting: Mechanical pipetting aids must always be used for all pipetting procedures. Oral pipetting is prohibited.
5. Personal Hygiene: All personnel should wash their hands immediately after the completion of any procedure in which TCDD has been used and when they leave the laboratory. Immediately after an exposure to TCDD, personnel should wash or, if appropriate, shower the affected area.

E. Operational Practices

1. Work Area Identification: Each entrance to the TCDD laboratory must have affixed to it a sign with the following warning:

TCDD LABORATORY
AUTHORIZED PERSONNEL ONLY

2. Access Control: The TCDD laboratory may be entered only by persons authorized by the Project Manager. The laboratory will be locked, and only authorized personnel will have keys. Potential problems and hazards that may be encountered in the laboratory should be reviewed with maintenance and emergency personnel prior to their being needed. Access doors to work areas should be kept closed at all times.
3. Work Surfaces: All work surfaces (bench tops, wood floors, etc.) on which TCDD samples or standards are used should be covered with stainless steel or plastic trays, dry absorbent plastic backed paper or other impervious material. The protective surfaces should be decontaminated or disposed of after the procedure involving TCDD has been completed.
4. Use of Primary Containment Equipment: Procedures involving solid or liquid samples that may result in the generation of aerosols should only be conducted in a chemical fume hood, a glove box, or other suitable containment equipment. Examples of aerosol producing procedures are the opening of closed vessels, transfer operations, weighing or blending.
5. Use of Analytical Instrumentation: Vapors or aerosols produced by analytical instruments, including GC/MS exhausts, are captured through local exhaust ventilation or scrubbed through a carbon filter. When a sample is removed from the analytical instrument, it is to be placed in a tightly stoppered sample tube or otherwise safeguarded from contaminating the laboratory. Any analytical equipment that becomes overtly contaminated should not be used again until it has been decontaminated.
6. Use of Respirators as Personal Protective Devices: A respirator will be provided for all personnel who enter areas where TCDD samples are extracted. Respirators must be worn when working with dry or dusty samples. The respirators will be selected in accordance with the requirements of the National Institute for Occupational Safety and Health (NIOSH) under the provisions of 30 CFR Part 11. NIOSH-approved carbon canisters and particulate filters will be used on the respirators, and each individual will have a specific respirator assigned to him/her. All respirators will be fit-tested prior to use.

7. Storage and Identification of Stock Quantities: Stock quantities of TCDD are stored in a specific storage cabinet that is locked at all times. This storage area is located within the laboratory work area. The storage area has affixed to it a sign which will read as follows:

TCDD STANDARDS
AUTHORIZED PERSONNEL ONLY

A listing of stock quantities of TCDD stored within the storage area will be maintained. The inventory must include the quantities of TCDD acquired and the dates of acquisition. Storage vessels containing stock quantities must have affixed to them labels with an appropriate warning such as: CAUTION: TCDD.

8. Working Quantities: Working quantities of TCDD standards, samples, etc., present in the work area should be kept to a minimum. Quantities should not exceed the amounts required for use in one week. This does not include amounts stored in a specific TCDD storage area or cabinet that is located within the laboratory work area. Storage vessels containing working quantities should have affixed to them labels with an appropriate warning such as: CAUTION: TCDD.
9. Laboratory Transport: Storage vessels containing TCDD standards or samples that are to be moved from one site to another (i.e. storage area to work area) must first be placed in an unbreakable outer container. Contaminated materials which are to be transferred from work areas to disposal areas should first be placed in a closed plastic bag or other suitable impermeable and sealed primary container. The primary container should be placed in a durable outer container before being transported. The outer container should be labeled with an appropriate warning such as: CAUTION: TCDD.
10. Housekeeping: General housekeeping procedures which suppress the formation of aerosols such as the use of a wet mop or a vacuum cleaner equipped with a HEPA filter (to remove particulates) should be used. Dry sweeping and dry mopping should not be used because of the hazard of aerosol formation.
11. Disposal: All waste produced in the TCDD laboratory shall be deposited in a 55-gallon drum, which will be disposed of at a facility capable of handling TCDD wastes. Disposable gloves, glassware, paper towels, spent absorbents, etc. should all be disposed of in this drum. The drum will be labeled and transported in accordance with applicable regulations.

C. Facility Specifications

1. Handwashing Facility: A handwashing facility will be available within the work area.
2. Shower Facility: A shower facility is located on the 5th floor of USTC. The shower facility is available and readily accessible at all times. This does not replace the emergency shower, which will be in the Extraction Laboratory.
3. Eye Wash Facility: An emergency eye wash facility will be located in each laboratory. It will be designed to wash both eyes at the same time with a continuous stream of potable water.
4. Exhaust Air from Primary Containment Equipment: The exhaust air from the glove box is treated by HEPA filtration. All exhaust air from primary containment equipment is discharged to the outdoors so that the possibility of entry into the building's air supply is minimized.
5. Exhaust Ventilation: A mechanical exhaust ventilation system is provided for controlling air movement. The movement of air should be from areas of lower contamination potential to areas of higher contamination potential (i.e. from entry corridors to the laboratory). This directional air flow is achieved by the draw from fume hoods in the TCDD laboratory. The exhaust air from laboratory areas will be discharged outdoors so that the possibility of entry into the building's air supply is minimized. Exhaust air from laboratory areas which is not derived from primary containment equipment can be discharged to the outdoors without being treated.

D. Work Area Monitoring Program

Wipe tests will be performed bi-monthly to determine cleanliness of work surfaces and tools. Wipe tests are to be performed by wetting a piece of filter paper with isooctane or other suitable solvent and wiping the area in question. Each wipe will subsequently be evaluated and analyzed by GC/EC. Any peaks which would indicate an amount of TCDD greater than 1 ug should be confirmed by GC/MS. If any wipe test indicates concentrations greater than 1 ug/wipe, the source of the contamination must be identified and corrected.

E. Sample and Extract Storage Area

1. Samples and Extracts: All current samples and extracts covered by this contract will be stored in the secure minimum-admittance lab designated for TCDD sample work-up only. Prior to, and following sample extraction, raw samples will be stored in the sealed, clearly labeled paint cans in which they are received. These paint cans will remain in the TCDD lab until the analysis has been completed and all results have been submitted. After submission of results, due to the bulky nature and number of paint cans expected, the raw samples will be removed to a secure area in the basement of USTC building until notification is received from the EPA that samples may be disposed of.

All extracts will be stored in clearly labeled glass vials in the refrigerator located in the TCDD lab. Storage space is not expected to be a problem with sample extracts, so they will remain in this area until EPA personnel request the disposal or return of extracts. The standard operating procedure for chain of custody of all samples (raw and extracted) must be adhered to at all times.

SOP for Security Procedures for Lab & Samples

1. USTCO maintains a posted guard 24 hours a day and 365 days per year.
2. Access to the building by employees is controlled and monitored by color coded picture identification cards.
3. Visitors must sign in upon entering at the guard stand (front entrance) and then again at the reception desk on the fourth floor, directly off the elevator access area. Visitors are assigned ID badges and are then escorted to their authorized destinations.
4. All laboratory doors have secure locks.
5. Sample receipt areas are secure and have limited access.
6. Chain of Custody procedures (See SOP) are followed.
7. Samples are stored prior to and during the analysis in the extraction laboratory. After analysis, the samples are stored in the basement. Access to this room is by authorized personnel only.

STANDARD OPERATING PROCEDURE FOR DOCUMENT CONTROL

To ensure that all documents relating to each EPA case are compiled in one secure location, the following procedure has been implemented:

I Case File Folders

- A) At the time of sample receipt the Sample Custodian will set up a file folder for each submitted case. Each folder will be clearly labeled with the USTC project number and the SMO case number.
- B) All documents, sample tags, SMO forms and laboratory generated data for each case will be placed in the individual file folder established for that case.
- C) Documents such as sample tags, traffic reports, airbills, notebook pages, etc. will be arranged by document type. The actual data package original will also be contained in the file.
- D) All current case files are kept in the office of the Sample Custodian. Once the deliverables have been distributed, the files may be moved to a secure storage area outside of the Sample Custodian's Office.

II Document Numbering & Inventory

- A) All documents in each case file will be assigned a serialized number (document control #) associating it with the case and region, e.g., SMO case # 2560 - Region III - Serialized document # 01.
- B) The serialized numbers will be assigned by document type, and the document control # is recorded on each document or set of documents.
- C) A document inventory sheet is included in each case file. The assigned document control number and number of pieces of each is listed next to the document or section description.
- D) Document numbering and inventory must be completed before submission of the Document Control and Chain of Custody Packages to NEIC's Contractor Evidence Audit Team, Techlaw.
- E) In the event all case documents are sent to the EPA, USTC will retain a copy of the document inventory list for that case.

Standard Operating Procedure
for Data Review

Organic

1. All data generated under this contract will undergo a 3 tier review system.

2. Tier I

Laboratory Data Review

As data is generated by the analyst it will be verified to ensure that the data meets the following contract criteria:

- A. Calibration is within specification.
- B. Calibration verification is within specification
- C. Instrument tuning criteria are met

3. Tier II

Laboratory Data Review

Data from the laboratory is reviewed by a data reviewer to verify that the following contract criteria are met:

- A. Reagent blank is negative.
- B. Verify calibration specifications have been met.
- C. Calculate recoveries and RPD for duplicate and spiked samples.
- D. Check calculations of final results and ensure that all reporting and QC forms are properly completed.

4. Tier III

Laboratory Data Review

Final review is performed by the individual preparing the data package as follows:

- A. Check all forms for completeness and accuracy
- B. Verify EPA and USTCO identification numbers. If necessary, prepare a cross-index table.
- C. Check all data for significant figures.
- D. Verify that all required deliverables are contained in final package - check against deliverables index.

The Project Manager will spot-check data packages to ensure that the data review SOP is adequate and that the deliverables are in compliance with contract specifications, or that extenuating circumstances resulting in packages not in compliance are covered in the case narrative.

5.0 RELATED EXPERIENCE

USTC has performed GC/MS analyses on many projects of similar magnitude to this program. Following are brief descriptions of some of the major projects in which USTC has participated.

USEPA Contract 68-01-6935

USTC has two lots under this contract, for the analysis of soil samples for 2,3,7,8-TCDD. The contractual obligation is for a maximum of 200 samples per month per lot, or a total monthly obligation of 400 samples.

USEPA Contracts 68-01-6727, 68-01-6780 and 68-01-6963

These three contracts are all for analysis of soil, water, and waste samples collected from Superfund sites for organic contaminants by GC/MS and GC. The combined monthly sample volume generated by these contracts is 240 samples. Due to the large number of samples requiring analysis, USEPA has occasionally requested that USTC accept more than its contractual obligation of 240 samples per month.

Soil, Water and Leachate Analyses at Major Superfund Sites in New Jersey

USTC is involved as an independent laboratory in three major Superfund Sites in New Jersey. Samples of various matrices have been analyzed over the past two years for 2,3,7,8-TCDD, Priority Pollutants (organic and inorganic), and miscellaneous conventional parameters. Sampling events generally result in groups of 100 - 200 samples.

Analysis of Effluent and Process Samples for Organic Contaminants by Isotope Dilution GC/MS

Under Special Analytical Services Contracts with USEPA, USTC has analyzed more than 4000 effluent and process water samples from various industries over the past year. These analyses were performed for USEPA's Effluent Guidelines Division, in support of development of Effluent Limitations. All analyses were performed by EPA Methods 1624 and 1625 (Isotope Dilution).

Special Analytical Services for USEPA

USTC, as a participant in the Contract Laboratory Program, performs special analytical services not covered by the standard contract. Among the types of special services performed are analysis of soil and water samples for tetra through octa classes of chlorinated dibenzo dioxins and furans, analysis of high volume air filters for extractable organics, analysis of sorbent tube air samples for volatile organics and analysis of various tissues for organic and inorganic contaminants.

It should be noted that USTC is the only laboratory in the U.S. which has been approved by EPA to perform analyses by all four of the major analytical methods used in Environmental Evaluation today:

- * 2,3,7,8-TCDD by Selective Ion Monitoring
- * Organic Pollutants by GC/MS
- * Inorganic Pollutants by ICP and AA
- * Organic Pollutants by Isotope Dilution

6.0 REPORTING

All final data reports for samples analyzed for organic contaminants are currently computer generated. An example of a report of Volatile Organics is presented in Figure 6.1. In addition, calibration and calibration verification reports are generated directly from the analytical data systems, as are quantitation reports and all raw GC/MS data.

Currently, data is manually entered prior to report generation. By May 1, 1985, however, it is anticipated that a direct link will be made between the analytical data systems and the system generating final reports. This will eliminate the need for manual data input, thereby reducing both the time required to generate a report and the likelihood of transcription errors.

All data is reported in the standardized format required by the EPA Contract Laboratory Program

Organics Analysis Data Sheet
(Page 1)

LABORATORY NAME: UNITED STATES TESTING CO, INC
LAB SAMPLE ID NO: 75678-96
SAMPLE MATRIX: WATER
DATA RELEASE AUTHORIZED BY _____

CASE NO: 1234
QC REPORT NO: 45
CONTRACT NO: 68-01-6563
DATE SAMPLE RECEIVED: 12/12/84

VOLATILE COMPOUNDS

CONCENTRATION: LGM
DATE EXTRACTED/PREPARED: 12/14/84
DATE ANALYZED: 12/14/84
CONC/DIL FACTOR: NA pH 5.8
PERCENT MOISTURE NA
PERCENT MOISTURE (DEDUCTED) NA

CASE NUMBER	ug/l	CASE NUMBER	ug/l
74-22-3	10 U	75-34-5	5 U
75-01-4	10 U	75-67-5	5 U
75-00-3	10 U	10061-02-6	5 U
75-03-2	10 U	79-01-6	5 U
67-64-1	5 U	124-48-1	5 U
75-15-0	5 U	75-00-5	5 U
75-35-4	5 U	71-43-2	5 U
75-35-4	5 U	10061-01-5	5 U
158-81-3	5 U	110-75-6	10 U
67-68-3	5 U	75-25-2	55
107-05-2	5 U	591-76-6	10 U
76-53-3	10 U	108-11-1	10 U
71-55-6	5 U	127-18-4	5 U
56-23-5	5 U	108-62-3	5 U
108-05-4	10 U	108-50-7	5 U
75-27-4	5 U	100-41-4	5 U
		100-42-5	5 U
			TOTAL XYLENES

Value If the result is a value greater than or equal to the detection limit, report the value.

0 This flag applies to pesticide parameters where the identification has been confirmed by GC/MS.

- Indicates compound was analyzed for but not detected. The number is the minimum attainable detection limit for the sample.

5 Analyte was found in the blank as well as the sample. Indicates possible/credible blank contamination.

Figure 6.1 Volatile reporting sheet.

7.0 CERTIFICATIONS

USTC is certified by the State of New Jersey in all categories for environmental analyses. The state certification number is 09370.

Although USEPA does not certify laboratories, USTC has qualified to participate in the Contract Laboratory Program for analysis of Organics, Inorganics and Dioxin. In order to continue participation in these programs, USTC is subject to quarterly inspections and audits by EPA, and the analysis of quarterly performance evaluation samples.

USTC is located in a labor surplus area (Hoboken, NJ).