ADDENDUM TO QUALITY ASSURANCE PROJECT PLAN FOR SUPPLEMENTAL RI/FS COLD CREEK SWAMP OPERABLE UNIT COLD CREEK/LEMOYNE SUPERFUND SITES

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Technology Development Laboratory Operations

QUALITY ASSURANCE MANUAL

Laboratory - Specific Attachment

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TD Lab Specific QA Attachment

TDL 1.0 INTRODUCTION

The Technology Development Laboratory (TDL) differs greatly from other IT Analytical Services (ITAS) Laboratories due to the wide variety of speciality services provided by the group. In addition to providing unique analytical services, (such as analysis of chlorinated dibenzo-p-dioxins and dibenzofurans) TDL performs treatability testing from bench scale testing up to full-size testing demonstrations. When needed, methodologies can be developed and tested for unique situations and matrices.

Due to the variety of TDL procedures, some routine ITAS Quality Assurance (QA) practices may not be readily applicable. On the other hand, many QA practices which are not implemented in a standard analytical laboratory will be necessary for QA in TDL. Many of these additional QA procedures are of a "project planning" nature and will follow a format similar to the IT Environmental Projects Group Engineering Operations QA Manual (QAM). These considerations make this Laboratory-Specific Attachment to the ITAS QAM both vital in its contribution to the QA program for TDL and unique in relation to other laboratory-specific attachments.

ITAS has developed a QA Program applicable to all analytical laboratories within IT Corporation (IT). The Program, as documented in the ITAS QAM, describes the activities to be undertaken by a laboratory so that data of acceptable quality are produced.

The ITAS Program recognizes that some QA activities must be detailed and adopted on a laboratory-specific basis. Also, certain practices described in the ITAS Program may require adoption by a laboratory for implementation because of necessary individual operations. To document individual laboratory QA practices, the ITAS Program requires the preparation of a laboratory-specific attachment to the ITAS QAM.

The TDL is part of ITAS and is located at 304 Directors Drive, Knoxville, Tennessee. The organization of TDL with respect to the rest of ITAS is shown in Figure 1.1.

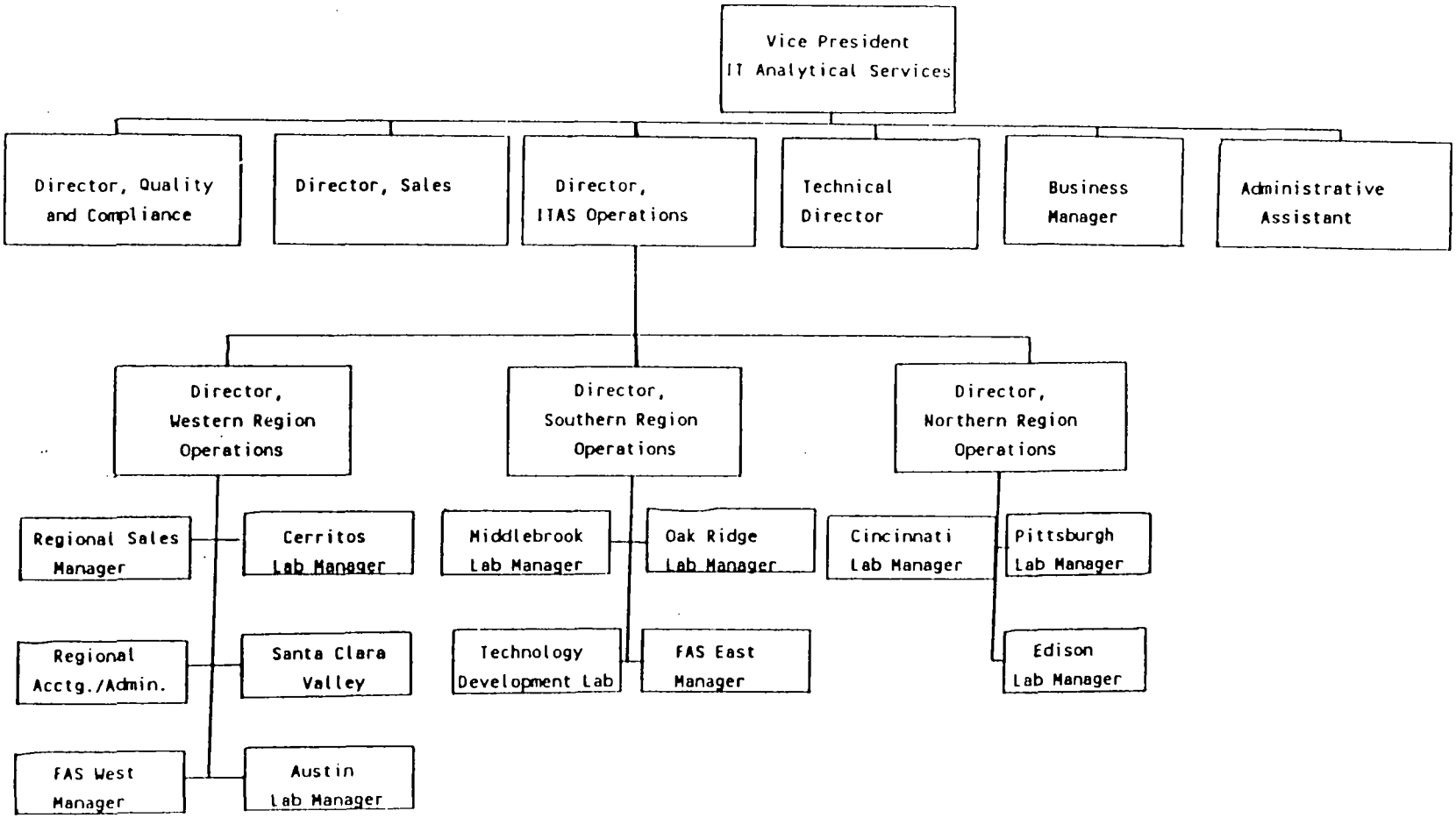
The manual contained herein is the laboratory-specific attachment for the ITAS TDL operations. As required by the ITAS QA Program, this attachment is a supplement to, and is incorporated with, the ITAS QAM to provide the QA Program for the TDL operations.

TDL 1.1 Format of the Attachment

The laboratory-specific attachment is formatted in the same manner as the ITAS QAM so that personnel in TDL operations can readily merge the requirements of the ITAS QAM and this document. Section headings for both documents are the same with the exception that this attachment uses the section prefix TDL so that attachment sections are clearly delineated. Section 17, Project Planning, has been added to this laboratory-specific attachment and has no counterpart in the ITAS QAM.

Since the attachment is not meant to be a self-standing document, the ITAS QAM must be included with the attachment to fully describe the laboratory-specific QA Program. In the event that the attachment describes an alternative means for implementation when compared with the manner prescribed in the ITAS QAM, the implementation described in this attachment shall prevail for TDL. Review and approval of the attachment, as prescribed by the ITAS QAM, demonstrates that the practices described in this manual are in accordance with the requirements of the ITAS QA Program.

Figure 1.1
ITAS Organizational Chart



TDL 2.0 ORGANIZATION

Organization responsibilities in TDL are unlike that of most ITAS labs. Many persons within TDL are responsible for duties which normally fall to several different people in larger analytical labs. Relationships of responsibilities for TDL are shown in Figure TDL 2-1.

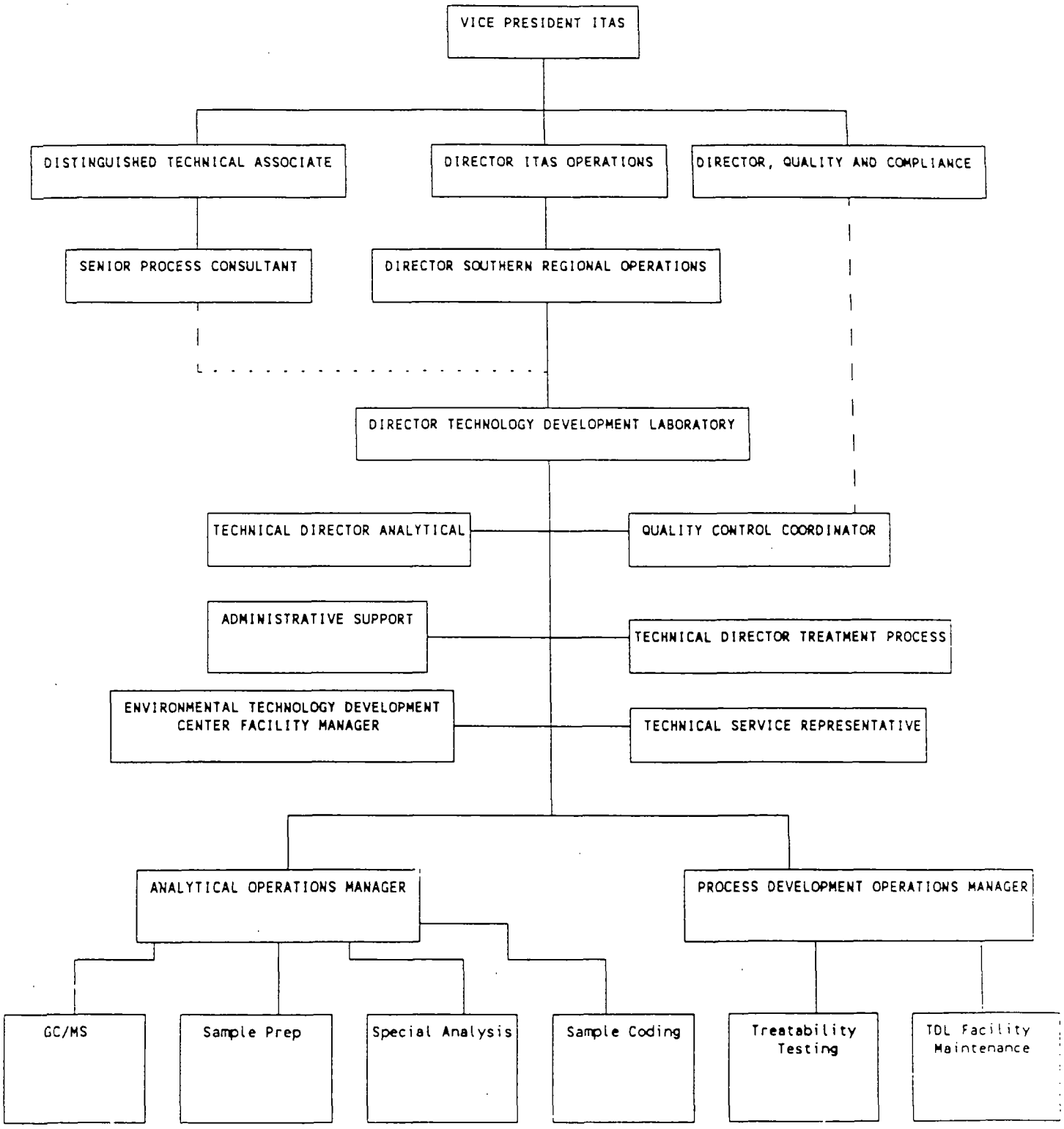
The quality-related responsibilities assigned to the laboratory manager and the operations manager, in the ITAS QA Manual, will be the responsibility of the Director of Technology Development Laboratory. TDL group leaders will take on responsibilities normally assigned to technical directors as well as those of group leaders. A listing of quality-related responsibilities follows:

- Director
  - Reports directly to Vice President, IT Analytical Services, or Regional Director Analytical Operations
  - Implements the Quality Assurance Program within the laboratory
  - Periodically determines the effectiveness of the Quality Assurance Program in the laboratory
  - Approves Laboratory-Specific Attachments to the Quality Assurance Manual and Project-Specific Manuals (QAPPs), SOPs, and revisions
  - Recommends to the Quality Assurance/Control Director, ITAS, changes in the Quality Assurance Program
  - Maintains current laboratory organization chart
  - Supervises laboratory participation in inter-laboratory accreditation and proficiency programs.
  - Prepares/Reviews Quality Assurance Project Plans*
- Operations Manager
  - Approves final issuance of laboratory reports*
  - Manages laboratory daily analytical and treatability testing operations
  - Supervises Quality Control activities performed as part of routine operations
  - Consults on development of experimental plans*

*Can be performed by Distinguished Technical Associate or Senior Process Consultant if designated by Director.

FIGURE 2.1

TECHNOLOGY DEVELOPMENT LABORATORY ORGANIZATIONAL CHART



- Reviews experimental plans for technical correctness and safety*
- Supervises the preparation and maintenance of laboratory records
- Oversees preventive maintenance program.
- Organizes and schedules the analytical testing program with consideration for sample-holding times
- Implements data verification procedures
- Assigns analysts for data processing and validation activities
- Reviews and approves all analytical data and submits to Director for issue
- Evaluates instrument performance and supervises instrument calibration and preventive maintenance programs
- Reports out-of-control or nonconforming situations to Director and Quality Control Coordinator as appropriate.

Technical Director

- Provides technical overview of laboratory activities
- Serves as an "in-house" consultant for the applicability of general Quality Control practices to specific needs
- Leads the training of analysts, engineers and technicians in laboratory operations and analytical procedures
- Evaluates analytical techniques, procedures, instrumentation and Quality Control procedures, and provide recommendations to the Laboratory Director
- Supervises the verification of software for data processing
- Recommends standards for purchasing instrumentation, equipment, reagents, gases, and chemicals
- Defines the instrument preventive maintenance schedule
- Defines the calibration program within the laboratory

- Quality Control Coordinator:

  - Prepares Quality Control standards, arranges insertion of Quality Control samples into the laboratory sample stream, and reviews the results

  - Performs statistical analyses utilizing results of QC samples analyses

  - Informs the Director and Group Leaders of data which lies outside of acceptable limits

  - Serves as the "focal point" for reporting and disposition of nonconformances; verifies corrective action

  - Resolves ongoing and recurring nonconformances within the Laboratory

  - Recommends corrective actions for resolution of nonconformances; verifies corrective action

  - Reviews statistical data to verify the laboratory is meeting stated Quality Control goals

  - Maintains current distribution lists for Laboratory-Specific Attachments and Project-Specific Manuals and Standard Operating Procedures

  - Closes findings and recommendations of Quality Assurance Audits

  - Notifies the Director of out-of-control situations

  - Stops production of data in a laboratory area where the review of quality control data or procedures shows significant problems

  - Reports nonconformances to the ITAS Director of Quality and Compliance or the Director, Quality Assurance, Environmental Project Group if the situation is not corrected within the Laboratory

  - Assists in the performance of Quality Assurance audits and performs Quality Control audits.

  - Establishes and supervises the laboratory Quality Assurance training program.

- Analysts/Engineers/Technicians

  - Performs procedures and data recording in accordance with accepted methods

  - Performs and documents calibration and preventive maintenance of instrumentation, as appropriate

  - Performs data processing and validation

  - Immediately reports out-of-control situations, instrument malfunction, calibration failure, or other nonconformances to the Group Leader and Quality Control Coordinator, as appropriate.

- Sample Coordinator

  - Oversees the log-in of all samples received, completion of chain-of-custody records, and maintenance of sample logs.

  - Supervises sample storage

TDL 3.0 STANDARD LABORATORY PRACTICE

Standard procedures as well as new or experimental procedures will conform to standard laboratory practice as much as possible in TDL. Exceptions will be noted on an individual basis in the project plan.

The basic steps of standard laboratory practice modified for TDL organization are outlined in Table TDL 3-1.

TABLE TDL 3-1

EXPERIMENTAL FLOW CHART

STEP NO.	DESCRIPTION	PERFORMED BY	DOCUMENTS USED OR PRODUCED
1	SAMPLE ARRIVES AT LABORATORY	Sample Coordinator	Chain-of-Custody Request for Analysis
2	SAMPLE(S) ARE UNPACKED AND INSPECTED AND PAPERWORK VERIFIED FOR COMPLETENESS. SAMPLES ARE NUMBERED AND PLACED IN STORAGE.	Sample Coordinator	Chain-of-Custody Request for Analysis Field Records
3	INTERNAL PAPERWORK INITIATED	Sample Coordinator	
4	SAMPLE(S) ARE SCHEDULED FOR EXPERIMENTATION	Operations Manager or Designated Project Manager	Sample Tracking Book, Analysis Schedule Form, Sample Tracking Sheets
5	SAMPLE(S) PROJECT FILE PREPARED	Administrative Support	Chain-of-Custody Request for Analysis Special Instructions from Client Price Quotes
6	SAMPLE(S) INFORMATION ENTERED INTO COMPUTER SYSTEM	Sample Coordinator	Computer Printout
7	EXPERIMENT PERFORMED	Chemists/Engineers	Sample Tracking Sheets, Logbook, Data Sheets, Maintenance Log.
8	RESULTS ANALYZED	Chemists/Engineers Chemists	Sample Tracking Sheets, Data Sheets

TABLE TDL 3-1 (continued)

EXPERIMENTAL FLOW CHART

STEP NO.	DESCRIPTION	PERFORMED BY	DOCUMENTS USED OR PRODUCED
9	DATA REVIEW/QC REVIEW	Chemists/Engineers Group Leader QA/QC Coordinator	QC Review Checklist
10	REPORT TO CLIENT	Lab Director Operations Manager	Final Report
11	RECORD MANAGEMENT	Administrative Support	All previously mentioned documents

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TDL 4.0 MATERIAL PROCUREMENT AND CONTROL

The quality of reagents, solvents, gases, water and laboratory vessels will be closely monitored due to the potential impact on experimental test results.

TDL 4.1 Air - Precautions are taken at the compressor to protect the quality of compressed air produced in-house. At the point of use, compressed air is passed through filters as needed depending upon the needs of the analyst or instrumentation.

TDL 4.2 Water - Deionized water is monitored to meet ASTM Type II water quality standards. A daily log book is kept to document requirements have been met.

TDL 4.3 Purchased Items (General) - Project Managers and Group Leaders are responsible for controlling and confirming quality in purchase orders at TDL. Individual project managers and group leaders are responsible for specifying the quality grade requirements for individual items with consent of the Quality Control Coordinator when necessary. The project manager will make the decision based on method requirements, project requirements and experience. The project manager is also responsible for verifying materials received meet requirements specified and overseeing proper storage after receipt.

Group leaders are responsible for overseeing purchase orders to verify suitable grades of materials are being ordered, received, labeled and stored. The group leader should see that certificates which come with certified materials are filed in the Quality Operations files. Materials should be removed when shelf life has expired.

TDL 4.4 Purchased Items and Services (Projects) - The control of purchased items and services shall, as appropriate to the specific project and each individual procurement, include:

- Prequalifications of subcontractor
- Bid evaluation
- Procurement source evaluation and selection
- Subcontractor performance verfication.

Control is provided by Purchasing, Project and Quality Assurance personnel. It shall be applied as necessary to verfity that an item or service conforms with the procurement documents. Items which do not meet Quality Assurance requirements shall not be accepted.

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TDL 5.0 SAMPLE HANDLING

Laboratory treatment tests and sample analyses are performed to produce data representative of conditions which existed at the time of sample collection or experimental testing. To provide representative samples for analysis, both field and laboratory procedures must be satisfactorily followed.

TDL 5.1 Field Collection and Shipment

To provide representative samples for testing or analysis, standard operating plans and project plans must be designed with that purpose in mind. The procedures must then be followed carefully to provide for sample integrity.

Prior to collecting samples, consideration must be given to the testing to be performed and the "hazard level" of the media being sampled so that proper sample containers and shipping containers can be assembled and the proper preservatives added to samples. In addition, field logs and record sheets, Chain of Custody forms, and Request for Analysis records must be assembled. All records required for documentation of field collection must be completed by the field team.

Field collection also includes uniquely identifying and labeling samples and packaging to preclude breakage during shipment. The interval between acquiring the sample and testing (holding time) should be monitored to assure representation of the sample.

High hazard (medium/high concentration) samples must be packaged according to DOT regulations. The ITAS Manual of Practice: "Sample Packaging and Shipment" should be used, in conjunction with 49 CFR, parts 100-177, when packaging hazardous samples. The manual also presents detailed instructions on packaging non-hazardous or environmental samples.

TDL 5.2 Chain of Custody/Request for Analysis

All samples shipped by TDL personnel or received at TDL should be accompanied by a Chain-of-Custody (example given in figure TDL 5-1) and a Request for Analysis (example given in figure TDL 5-2). Upon receipt, the sample coordinator will:

- Compare samples received against those listed on the Chain-of-Custody,
- Verify that sample holding times have not been exceeded,
- Note condition of samples upon receipt,
- Sign and date the Chain-of-Custody form and file with waybill, and,
- Initiate corrective measures for samples received without proper paperwork.

TDL 5.3 Receipt

After checking the samples and noting condition on the Chain-of-Custody, the Sample Coordinator enters all appropriate sample and project information in the sample tracking log book (example form given in figure TDL 5-3). The project manager is then informed of their location. As needed, laboratory blanks, duplicates and other QC samples are added to the project by the Sample Coordinator in accordance with the project manager's instructions.

TDL 5.4 Storage

Samples will be stored in laboratory refrigerators, freezers or properly ventilated areas for a period not to exceed six months after receipt, unless the client has made special arrangements for samples to be kept longer. Fate of samples should be agreed upon by the client at the onset of a project.

INTERNATIONAL TECHNOLOGY CORPORATION

Figure TDL 5-1

CHAIN-OF-CUSTODY RECORD

R/A Control No. _____

C/C Control No. A 93064

PROJECT NAME/NUMBER _____ LAB DESTINATION _____

SAMPLE TEAM MEMBERS _____ CARRIER/WAYBILL NO. _____

Sample Number	Sample Location and Description	Date and Time Collected	Sample Type	Container Type	Condition on Receipt (Name and Date)	Disposal Record No.

Special Instructions: _____

Possible Sample Hazards: _____

SIGNATURES: (Name, Company, Date and Time)

1. Relinquished By: _____

Received By: _____

2 Relinquished By: _____

Received By: _____

3. Relinquished By: _____

Received by: _____

4. Relinquished By: _____

Received By: _____

WHITE - To accompany samples
YELLOW - Field copy

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Figure TDL 5-2

INTERNATIONAL TECHNOLOGY CORPORATION

REQUEST FOR ANALYSIS

R/A Control No. B 96874

C/C Control No. _____

PROJECT NAME _____

PROJECT NUMBER _____

PROJECT MANAGER _____

BILL TO _____

PURCHASE ORDER NO. _____

DATE SAMPLES SHIPPED _____

LAB DESTINATION _____

LABORATORY CONTACT _____

SEND LAB REPORT TO _____

DATE REPORT REQUIRED _____

PROJECT CONTACT _____

PROJECT CONTACT PHONE NO. _____

Sample No.	Sample Type	Sample Volume	Preservative	Requested Testing Program	Special Instructions

TURNAROUND TIME REQUIRED: (Rush must be approved by the Project Manager)

Normal _____ Rush _____ (Subject to rush surcharge)

POSSIBLE HAZARD IDENTIFICATION: (Please indicate if sample(s) are hazardous materials and/or suspected to contain high levels of hazardous substances)

Nonhazard _____ Flammable _____ Skin Irritant _____ Highly Toxic _____ Other _____ (Please Specify)

SAMPLE DISPOSAL (Please indicate disposition of sample following analysis. Lab will charge for packing, shipping and disposal.)

Return to Client _____ Disposal by Lab _____

FOR LAB USE ONLY

Received By _____ Date/Time _____

WHITE - Original, to accompany samples
YELLOW - Field copy

TDL 5.5 Sample Tracking

Samples should be tracked from field personnel to the laboratory by means of a Chain-of-Custody record. If samples are sent from the TD laboratory to any other site, a Chain-of-Custody record will accompany the samples. The sample tracking log book (example shown in figure TDL 5-3) will show storage location of the sample as well as the project manager responsible for the sample while it is in the TDL. The samples fate will also be recorded in this tracking log book.

Documentation of Chain-of-Custody and Disposal (if any) will be kept in the project file for that sample.

TDL 5.6 Treatability Study Accountability

A daily account must be kept on the Treatability Study Monthly Accountability Form (an example of this form is in figure TDL 5-4) during Treatability Studies in order to maintain compliance with state and Federal guidelines. The project manager is responsible for keeping the form up to date and the TDL director or his designee conducts inspections to be sure compliance is maintained.

TDL 5.7 Initiation of Testing

Initiation of testing is the responsibility of the project manager. The project manager is informed by the sample coordinator when samples are received. The project manager initiates testing keeping in mind any holding times which need to be met.

TDL 5.8 Disposal

The types of waste handled and generated by TDL vary greatly and unlike most laboratories TDL handles large quantities at times for treatability studies. For these reasons, it is necessary for

Figure TDL 5-3

TECHNOLOGY DEVELOPMENT LABORATORY
SAMPLE TRACKING

Project Name						
Project Number						
TDL Sample #						
Client Sample #						
Sample Quantity						
Sample Matrix						
Date Received						
Date Due						
Storage Location						
Project Manager						
QA ID #						
Sample Fate						
Report Date						
Date Billed						

Sample Fate Codes:
DO - drummed and disposed of
SC - sent back to client
CE - completely used up or destroyed in extractions
TR - treated and rendered non-hazardous

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Figure TDL 5-4

TREATABILITY STUDY MONTHLY ACCOUNTABILITY FORM

CLIENT_____ PROJECT NO._____

EPA ID#_____ PROJECT NAME_____

PROJ. MGR._____ MONTH_____ 19_____

The following information must be continually updated and the original copy kept in the MASTER TREATABILITY FILE. This information must be kept for three (3) years after the study has been completed.

DAY OF MO.	AMOUNT OF AS RECEIVED WASTE ON SITE	AMOUNT OF WASTE TREATED TODAY	PROCESS TREATMENT RATE	AMOUNT OF RESIDUE ON SITE	AMOUNT OF RESIDUE RETURNED TO GENERATOR	AMT OF RESIDUE SHIPPED OUT FOR DISPOSAL	DATA ENTERED BY
01							
02							
03							
04							
05							
06							
07							
08							
09							
10							
11							
12							
13							
14							
15							
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25							
26							
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28							
29							
30							
31							

TDL 5.8 Disposal (continued)

TDL's Waste Disposal Coordinator to maintain a waste packaging area (pre-drumming) to handle sorting and packaging of waste to be sent off site. The Waste Disposal Coordinator completes a "Drum Information Sheet" (example of sheet is in figure TDL 5-5) which is filed in the TDL Quality/Operations files Disposal and date is also noted in the sample tracking log book under "sample fate".

Shipments are made at least once every three months. A copy of the certification of incineration (or other disposal technique) is kept in the Quality/Operations file and in the project file if necessary. When certification of incineration of a sample is required by the client, the Waste Disposal Coordinator sends a copy of the certification to the client.

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Figure TDL 5-5

DRUM INFORMATION SHEET

Drum No._____ Arrival Date_____ (N/A)_____

Drum Type_____ Gross Wt._____

Project Name_____

Project No._____

Recieved From_____

Shippers ID# _____

Chemical/Hazardous Material Name_____

Lab Pack ( )Y/N

Description:

Note: "Lab debris and consumables" refers to lab coats, gloves, rinsed glassware, sample jars with residues; disposable plastic sample cups, glass pipets and towels; rubber tubing, metal cans or other waste-contaminated articles; small volume (100g) stabilized waste samples combined with fly ash, cement or kiln dust.

DOT proper shipping name_____

_____

UN/NA NO._____ Hazard Class_____

EPA Waste Code No._____

Chemist/Drum Packer_____

Shipping
Destination_____

_____EPA ID #_____

Shipping Date_____

TDL 6.0 CALIBRATION PRACTICES

TDL 6.1 Calibration System

Each piece of equipment to be calibrated is assigned a unique identification number. Standard operating procedures are followed for each instrument used by the lab. Reference standards are used or calibration by an outside firm is documented. At a minimum, standards are checked against a standard that has been prepared independently or against a standard from an independent supplier. The frequency and acceptable performance tolerances follow those prescribed by the ITAS QAM, SOP, method or project plan being used, whichever is appropriate to the project.

Records are kept for periodic as well as operational calibrations. Calibrations which fall outside of acceptable limits result in corrective action.

TDL 6.2 Operational Calibration

Operational calibration is generally be performed as part of an analytical procedure or test method. Tables 6.1 and 6.2 give summaries from the ITAS QAM for instruments used by TDL.

TDL 6.3 Periodic Calibration

Periodic calibration is be performed for equipment such as balances, thermometers and ovens.

- Balances are calibrated to NBS traceable, class S weights once every three months. The acceptance criteria for an operational calibration to 0.01 gram shall be ±0.01g for 0 to 100g and ±0.1 percent of the applied weight over 100g.

- Reference thermometers are calibrated to an NBS traceable thermometer once every three years. Working thermometers are calibrated to a reference thermometer once every 12 months.

- Micropipettors are calibrated on a quarterly basis according to manufacturers' specifications using the calibration tool provided by the manufacturer.

Table 6.1. Summary of Operational Calibration Requirements

Instrument	Calibration Standards Used, Initial & Daily Minimum	Acceptance Limits	Corrective Actions	Reference
Atomic Absorption Spectrophotometer	Initial: 3 levels + blank Daily: 1 check standard (midrange) per 10 samples;	Linear regression correlation coefficient >0.995; ±5% of true value	Make new standards or establish new calibration curve	1,2
Gas Chromatograph	Initial: 3 levels + blank	Linear regression correlated coefficient >0.995; or 5 levels using point to point calibration with quadratic corr. coeff. >0.997	Make new standards, or	1
	Daily: 1 level of check standards	±20% of original curve	new calibration curve	
Ion Chromatograph	Initial: 3 levels ± blank Daily: 1 level of check standards every 20 samples (midrange)	±10% of original curve	Make new standards recalibrate	3

Table 6.1. Summary of Operational Calibration Requirements (cont'd)

Instrument	Calibration Standards Used, Initial & Daily Minimum	Acceptance Limits	Corrective Actions	Reference
pH Meter	Daily: 3 levels	±0.05 pH unit	Clean or replace electrode; recalibrate	1

References:
1) EPA SWL 846, "Test Methods for Evaluating Solid Waste"
2) USEPA Methods for Chemical Analysis of Water and Wastes, 1979
3) USEPA 300.0, 1984

Table 6.2. Summary of Periodic Calibration Requirements

Instrument	Calibration Standards Used, Initial & Daily Minimum	Acceptance Limits	Corrective Actions
Analytical Balance	Daily: Sensitivity	0.001 gm	Adjust, sensitivity
	Monthly: Reproducibility	Mean, Std. Dev. less than 0.1 mg	Service balance
	Monthly: Consistency	Difference less than 0.1 mg	Replace weights
	Quarterly: Class "S" weights Check	Difference less than 0.1 mg	Service balance
Thermometers	Calibrate in constant temperature baths at two temperatures against precision thermometers certified by NBS annually	$\pm$0.5°C	Tag and remove from service, replace
Pipettors	Gravimetric check quarterly	High volume (>100uL): $\leq$ 1.0% relative error and RSD Low volume (<100 uL): $\leq$ 2.0% relative error and RSD	Service or replacement

TDL 7.0 PREVENTIVE MAINTENANCE

TDL maintains many more instruments per chemist than most analytical laboratories in order to have availability of many different types of instrumentation. Instruments are not used on a daily basis, but rather on a project specific basis. Therefore, it is necessary to have different modes of operation into which each piece of equipment can be classified. The three classifications follow:

1) Operational - the equipment can be used for analysis or testing without further preparations. It is currently following an operational preventive maintenance schedule (see Figure 7-1) which is posted on the instrument. Details of individual preventive maintenance schedules are discussed in TDL laboratory standard operating procedures.

2) Stand By - the equipment is following a less rigorous stand by preventive maintenance schedule, which is posted on the instrument. Calibration or minor maintenance will be required before the equipment can be used for analysis or testing.

3) Shut Down - the equipment is labeled as being "shut down". No preventive maintenance is performed and a start up maintenance is required before the equipment can be used.

Figure 7-1

Month Year 198__

Preventative Maintenance Schedule

1. Replace Septa	(A)
2. Perform Column Maintenance	(F)
3. Replace Column	(F)
4. Clean Sorce	(C)(F)
5. Replace Injection Port Liner	(F)
6. Clean Poles	(C)(F)
7. Clean Fans	(B)
8. Printer Maintenance	(B)
9. Printer Ribbon Replacement	(F)
10. Retune	(C)(F)
11. Dip Rods	(C)(F)
12. Change Mechanical Pump Oil	(E)
13. Change Turbo Pump Oil	(D)
14. Change Water Filter	(D)
15. Change Gas Line Filter	(F)
16. Cool Flow Maintenace	(E)
17. Calibrate	(C)(F)
18. Other	(F)
19.	
20.	
21.	
22.	
23.	
24.	
25.	
26.	

1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31

Scheduled Times For Preventative Maintenance

(A) Daily
(B) Monthly
(C) Quarterly (January, April, July, October)
(D) Annually (January)
(E) Biannually(January, July)
(F) As Needed

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TDL 8.0 ANALYSIS OF QUALITY CONTROL SAMPLES

TDL 8.1 Frequency of QC Samples

The minimum frequency for analysis of QC samples in TDL is 15%, that is; a blank, a spike and a duplicate or spike duplicate out of every 20 samples.

TDL 8.2 Verification Samples

Verification samples are used when available and appropriate to assure levels of concentration or qualitative accuracy. These verification standards (ex. NBS or EPA standards) may not be appropriate for experimental procedures, but are used wherever needed for calibration prior to analytical procedures or certain types of experimental procedures.

TDL 8.3 QC Levels

Level I -- ITAS standard practice. Use available analytical procedures. Fifteen percent QC samples (blank/spike and duplicate for every 20 samples). QC samples may not be performed for a specific project, but as part of compiled sets of samples. QC data are not reported as part of analytical results.

Level II - Use available analytical (reference) methods. Fifteen percent QC samples minimum (blank/spike/duplicate) per 20 samples. QC samples are project or client-specific. QC summary report is included as part of the analytical results reported. No raw data are included.

Level III - The highest level requires use of referenced regulatory procedures, and/or established/verified procedures using confirmatory type techniques (i.e., GC/MS dual column GC). Method blank plus two other QC samples (duplicate, spike or matrix spike duplicate pairs) minimum per 20 samples of each matrix. A QC summary report is supplied with supporting data. Where applicable, this level is the USEPA Contract Laboratory Program (CLP) package.

Project-specific requirements must be defined in a QA Project Plan or Work Plan. Project specific documentation should be submitted to the laboratory before beginning work. Project requirements for QC samples cannot be less than Level I.

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TDL 9.0 ANALYTICAL PROCEDURES

Analytical procedures in TDL will generally fall into four categories: Standard and non-standard analytical procedures, and standard and non-standard experimental procedures. The standard methods follow an accepted method documented in TDL standard operating procedures (SOPs). The non-standard methods go through a predetermined process of decision making, however, the actual procedure is decided upon on a project specific basis.

TDL 9.1 Standard Analytical Procedures

Standard analytical procedures performed by TDL follow TDL SOPs. The SOPs are based on prior experience with approved methods or modifications thereof (such as EPA methods 608 and 8080 for PCB's in water and soil).

TDL 9.2 Non-Standard Analytical Procedures

Analyses which have no previously implemented method prescribed to them fall under the non-standard category. Methods and procedures are compiled from previous knowledge or experience on a project specific basis. Standard Operating Procedures are developed during these analysis for future use.

TDL 9.3 Standard Experimental Procedures

Experimental procedures which can follow a SOP are considered standard. A SOP is followed for each different type of experimental procedure.

TDL 9.4 Non-Standard Experimental Procedures

The procedure for each non-standard experimental procedure is determined and defined for each project by the project manager. Newly developed procedures are documented as SOPs.

TDL 9.0 ANALYTICAL PROCEDURES

Analytical procedures in TDL will generally fall into four categories: Standard and non-standard analytical procedures, and standard and non-standard experimental procedures. The standard methods follow an accepted method documented in TDL standard operating procedures (SOPs). The non-standard methods go through a predetermined process of decision making, however, the actual procedure is decided upon on a project specific basis.

TDL 9.1 Standard Analytical Procedures

Standard analytical procedures performed by TDL follow TDL SOPs. The SOPs are based on prior experience with approved methods or modifications thereof (such as EPA methods 608 and 8080 for PCB's in water and soil).

TDL 9.2 Non-Standard Analytical Procedures

Analyses which have no previously implemented method prescribed to them fall under the non-standard category. Methods and procedures are compiled from previous knowledge or experience on a project specific basis. Standard Operating Procedures are developed during these analysis for future use.

TDL 9.3 Standard Experimental Procedures

Experimental procedures which can follow a SOP are considered standard. A SOP is followed for each different type of experimental procedure.

TDL 9.4 Non-Standard Experimental Procedures

The procedure for each non-standard experimental procedure is determined and defined for each project by the project manager. Newly developed procedures are documented as SOPs.

TDL 10.0 DATA VERIFICATION

TDL 10.1 QC Data

Quality control samples are inserted into the sample stream at a rate of not less than one spike, one duplicate and one blank for every twenty samples in a given matrix (referred to as a batch). Accuracy and precision calculated from these QC samples must meet the requirements prescribed by the method, SOP or Project Plan. If QC samples do not meet the QC goals, they will be redone or a nonconformance will be filed with the QC Coordinator. This means of evaluating QC data assures verification of procedures.

TDL 10.2 Data Validation

Validation of data takes place on two levels. The first level of validation is to unveil problems as soon as possible during an ongoing project. The second level is to keep unvalidated results from being reported to the clients.

The first level consists of either review by a project manager or peer. This review is covers the following three items:

1. Are QC results are acceptable?
2. Check for outliers
3. Re-compute calculations at a rate of at least 20%

The second level is the independent review which must be done before the final report goes out. All results prior to the independent review are considered "draft" or "preliminary".

Data validation on both levels is documented by signing and dating the draft or hard copy of the data and indicating "project manager review" or "independent final review." For longer, more formal projects, a Review Documentation form (figure 10-1 is an example of one) should be completed and filed in the appropriate project file.

FIGURE 10-1

To_____ Date_____

From_____

Please review the document described below for the items indicated. This document is (a) attached, (b) already in your possession. Following your review, place your signature and the date in the spaces provided, provide your review comments or conclusions, send the original of the form to Central Files, and provide me with a copy of the form with the document reviewed (a) attached, or (b) retained by you unless marked with comments, by _____ _____. date (and time, if applicable)

Charge No. for review time _____

REVIEW DOCUMENTATION

Project No. _____ Project Name _____

Document title and, if applicable, subject, revision no., draft no., or other description necessary for specific identification _____

Date document was prepared_____
Document preparer or originator_____

Please review this document for _____

Reviewer_____

Date Reviewed _____

Review Comments or Conclusions

TDL 10.3 Verification of Software

If computer software is used to acquire, process, or report data, it is necessary to demonstrate that the software correctly performs its intended function.

Software verification can be documented by comparing its performance against known results and filing both results in the Quality/Operations files. The standard operating procedure for software verification can be found in the ITAS omnibus SOP manual.

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TDL 11.0 REPORT PREPARATION

TDL 11.1 Types of Report

1. Letter Report or Letter of Analysis

   - Usually written for a project that lasts less than two weeks in the lab.
   - Simply describes the experiment and gives the results.

2. Project Report

   - More formal report, for projects lasting more than two weeks.
   - Contains a table of contents.
   - Results are given in tabular form.
   - QA procedures and results are given, if applicable.

3. Full Research Report

   - Written for a large project which follows a quality assurance project plan or work plan.
   - Document stands alone, all raw data are included.
   - The format follows the outline in Figure 11-1 which shows the sections in order, with suggested topics which would fall under each section. (Suggestions are also made under the "comments" column as to the length of each section.)

FIGURE 11-1

Final Report Outline	Comments
1. Executive Summary	1 page
Statement of Scope and Purpose	• Identify waste source • Technologies evaluated (why? regulatory driven?) • Goals for project (cleanest? cheapest? cleanup criteria?) • Test results (Key data) • Conclusion • Recommendation
2. Introduction	3-4 pages
Statement of Scope and Purpose	Concise Statement of Scope and purpose
Background Ex. Literature Search Results	Rationale for the project Very brief in introduction.
Approach	Brief and concise, generalized description of activities to provide enough background for reader to relate to and comprehend results in later section.
Environmental Compliance	Compliance requirements for project such as special permits or treatability study exemption.
3. Experimental Methods	4+ pages test, tables and figures Include sample prep, preservation and analysis method (method no. if possible) and QA/QC results (unless they are significant enough to be a section of their own - See 5)

- Approach - justification if needed information to be gained
- Setup - physical description of experiment including sizes, types and vendor names and a photo or figure of setup
- Feed Preparation (where applicable) - was it filtered, decanted or composited?

4. Test Results

7-10 pages text, tables and figures

- Initial analysis
- Results for each technology tested

5. QA/QC Results (if part of QAPP)

1-3 pages

Tables showing results and statistical analysis performed

6. Process Design (if needed)

- Design Basis including experimental results
- Process Design including figures, flow diagrams and physical description of the process
- Economics

7. Summary and Conclusions

2+ pages

Summary of Results

Provides more details than in Section 1.

Conclusions

Shows relevant scientific thoughts to tie-in the above sections, and the study.

8. Recommendations	1+ pages Provides a detailed discussion of further research areas based on information gaps identified in this study, and considerations for future research.
Appendices	May include permits, copies of actual analytical methods, certificates of analysis, preliminary data, etc.

TDL 11.2 Method of Preparation

For each report the project manager, or designated representative, shall:

- Determine the content of the report based on the scope of the project, client requirements and any regulatory requirements that might exist.
- Determine the report format and type (from the list in TDL 11.1).
- Assign personnel to prepare the items required for the report.
- Assign personnel as needed to review the work prepared for the report.

TDL 11.3 Review

The project manager is responsible for initiation of the report. The report can be written by other technical personnel on the project and reviewed by the project manager, or can be written by the project manager and must be reviewed by peers.

The independent review by either the TD Lab Director or the Senior Process Consultant must then take place before the final report can be released.

TDL 12.0 RECORDS MANAGEMENT

Records are maintained that include documents which demonstrate the testing performance of the laboratory.

Laboratory records are maintained in two broad categories:

Project Files

- Documents which are specific to a project or a group of samples within an ongoing project, such as chain-of-custody and raw analytical data.

Quality/Operation Files

- Documents which demonstrate overall laboratory operation, such as instrument log books and control charts. These records will directly affect the data for a specific project, but in general their applicability is not limited to one project.

All laboratory records from time of sample receipt through reporting and disposal must be available and stored in a manner that safeguards their integrity from tampering or physical damage and loss. Any documentation that bears on the reported results must be available if requested by the client or other entitled individuals. This includes operational and project-specific data. Data may be stored in "real-time" as it is produced, or filed in a manner

allow prompt retrieval and assembly into a complete project file as described in the following section.

TDL 12.1 Project Records

Separate record packages are maintained for each project. Filing of records for a specific project shall be by the unique project identification number assigned by the laboratory for that project. Within a project file, categories of information are filed separately.

Figure 12-1 presents the categories used within a TDL project file. It is not expected that all categories will be applicable for every project. However, in all project files, specific information will be filed in accordance with the category designation provided. Each project file shall have an index that lists each record unit within the project file. A copy of this index page with the sections which apply to the specific project circled is located at the beginning of each project file.

TDL Project Index

TDL Project Mgr._____ Proj. Mgr._____

Project Name _____ Project Number_____

Date Began _____

A Project Index

B Correspondence (including contracts, proposals and permits)

C Project Plans (QAPPs, work plans, operating plans, Health & Safety Plans)

D Chain-of-Custody

E Field Records

F Request-for-Analysis

G Calibration Records

H Analytical Data (copies of log book pages,log book pages, chromatograms)

I Experimental Data (operating log sheets)

J QC Samples

K Data Reports

L Project Specific Requirements

M Nonconformances

N QA Plans

O Miscellaneous

P Method Description

Q Work Order Records

R Subcontractor Records

S Engineering Calculations or Process Designs

T Compliance Records (treatability study, inspection logs)

U Vendor Files

Comments _____

_____

_____

_____

TDL 12.2 Quality/Operations Records

Other records, which do not necessarily pertain to a particular project, are filed as the quality/operations records. An example of the list of records kept by TDL is given in Figure 12-2.

Many of the laboratory operations records are in daily use and are not filed in a central location until they are completed. Other files are kept in a central location for easy access to all personnel.

TDL 12.3 Record Control

The TDL secretary is responsible for the records management system. This person shall:

- Initiate new project files including the project index.
- Add new records to existing files, initiate new files within a category, and update the index.
- Assist laboratory personnel in withdrawing and returning records.

FIGURE 12-2

TDL Quality and Operations Files

Stock Solution Data Sheets (by date received)
Periodic Calibrations (pipetters, balances and thermometers)
Material Safety Data Sheets
Extraction Notebooks
Preventive Maintenance Schedules
Maintenance Logs
Run Logs for items on the Nelson System
Training Records, Resumes, & Certificates
TDL Job Descriptions
Organizational Charts
Signature Initial Record
Nonconformance Memos (monthly file)
Monthly QC reports
Monthly Surveillance Report
Audit Reports
Performance Evaluations
Control Charts
Master Copy and Extra Copies of TDL SOP's & QA Manual
Distribution Lists for TDL SOP's & Lab Specific QA
Contents of Drums/Disposal logs
Radiological Material License File for EC Detectors
Treatability Study Files
Tailgate Safety Meeting Records
Monthly Safety Inspection Records
Computer Software Verifications
Certifications
Material Procurement & Control
Temperature Logs
Water Quality Logs
Master Sample Log

TDL 13.0 NONCONFORMANCES AND CORRECTIVE ACTION

A nonconformance is a deficiency in procedure sufficient to render the quality of an item unacceptable or indeterminate or any event which is beyond the limits documented and established for laboratory operation. Non-conformances may be caused by non-Laboratory operations (e.g., field collection paperwork not complete) and be identified at the laboratory.

Nonconformances may include (but are not limited to) the following:

- Sample receiving documentation not correct
- Sample condition on receipt not acceptable
- Sample holding time exceeded
- Sample storage conditions outside criteria
- Incorrect sample preparation/analysis procedures used
- QC sample data (blank, spike, duplicate, surrogates, etc.) outside limits
- Calibration requirements not met
- Data recording errors, transcription errors, or failure to document
- Data validation errors
- A recovery or relative percent difference result that is out of control (e.g., more than three standard deviations from the weighted mean (for that month)
- Relative standard deviation for response factors greater than accepted limits
- Positive blanks
- Any situation or result which might affect the quality of data

A corrective action is an appropriate measure applied to correct a deficiency and minimize the possibility of recurrence.

Corrective action will include, but not necessarily be limited to:

- Recalibration of instruments, using freshly prepared calibration standards
- Reanalysis of samples
- Replacement of lots of solvent or other reagents that give unacceptable blank values.
- Additional training of laboratory personnel in correct implementation of sample preparation and analytical methods
- Reassignment of personnel, if necessary, to improve the overlap between operator skills and method requirements
- Communication with the clients to determine the appropriate action (e.g., insufficient sample remaining for reanalysis).

TDL 13.1 Procedure for Documenting Nonconformance and Corrective Action

Any laboratory employee noticing a deficiency suspected of being a nonconformance shall report the deficiency to the responsible supervisor and to the QCC on a nonconformance memo form.

The Quality Control Coordinator shall maintain a log of nonconformances that includes a description of the problem and corrective action, and lists the responsible manager, the affected projects and sample numbers, and initial and closeout dates.

Once the corrective action has been taken, the QCC shall verify, through a special surveillance or audit, that the problem has been corrected. The QCC shall document that the corrective action has been completed satisfactorily (closed out) by signing the nonconformance memo.

The QCC shall file a copy of all records pertaining to the nonconformance with the nonconforming item records (i.e., specific project files or procurement files). The nonconformance log maintained by the QCC shall contain a reference to the file location.

TDL 13.2 Variances

Variances from standard approved operational procedures and plans shall be documented in a Variance form. It is recognized that procedures such as work plans cannot be prepared which properly foresee all conditions encountered during a field program. The project manager shall initiate and maintain the variances for each project. All items recorded on the Variance form require the approval of the Project Manager's supervisor and the Quality Control Coordinator. The Variance form shall contain: date and nature of the variance, applicable document, and IT personnel initiating the variance. In no case shall a subcontractor initiate a variance. If a variance is proposed by the client, it shall be so recorded.

The Project Manager shall keep a copy of all variances with the project file. The Project Manager's supervisor shall review copies of the forms and, when in agreement, indicate approval by signing and dating each variance. The copy shall be forwarded to the Quality Control Coordinator for review, signing, and dating and then returned to the Project Manager for inclusion in the project files. Originals of the Variance form shall be kept on site (when appropriate) until the field work is complete.

Variances to approved procedures and plans will be approved prior to implementing the deviating action.

TDL 14.0 QA/QC AUDITS

In-house audits (often called surveillances) are performed on a monthly basis by the Quality Control Coordinator. They are conducted on behalf of the laboratory management to verify that the Quality Assurance Program is implemented and functioning on a daily basis.

The QCC schedules the audit and sends a memo to the TDL staff announcing the date and scope of the audit. This is done at least one week prior to the audit. The laboratory manager discusses the scope of the audit and has a post audit review with the QCC.

Results of the in-house audits are reported to the Laboratory Manager and the ITAS Director of QA & Compliance. Appropriate personnel are informed of actions needing to be performed as a result of in-house audit findings. Audits are filed in the laboratory Quality/Operations files.

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TDL 15.0 QUALITY REPORTS TO MANAGEMENT

The Quality Control Coordinator is responsible for providing monthly reports to the management indicating the effectiveness of the Quality Assurance Program. These reports are written to the ITAS Director of Quality Assurance with copies sent to the Operations Managers and Laboratory Director. The report contains those sections from the following list which are appropriate to that month's activities:

- Internal audits, performance evaluations, audit information in general.
- Certification information.
- Nonconformance/corrective action summary.
- Summary of monthly QC data.
- New or revised laboratory SOPs.
- Personnel changes or training.
- Instrumentation changes, additions, deletions.
- New test methods.
- Training courses attended by lab staff.
- Miscellaneous information.
- QAPPs received or written.

The QCC Monthly Report Format is attached. All sections are included each month with appropriate information or comments such as "no external audits this month".

QCC MONTHLY REPORT FORMAT

1. AUDITS

- INTERNAL AUDIT/SURVEILLANCE(S)
- EXTERNAL AUDITS(S)
- SUBCONTRACTOR AUDIT(S)

2. PERFORMANCE EVALUATION SAMPLES

- PE SAMPLES IN-HOUSE
- PE RESULTS SUBMITTED/PENDING
- PE RESULTS RECEIVED

3. CERTIFICATIONS

- RECEIVED
- INITIATED

4. NONCONFORMANCE SUMMARY

- INCLUDE A COPY OF YOUR NONCONFORMANCE LOG OR TABULATE BY AREA (PREFERRED)

5. HOLDING TIME VIOLATION SUMMARY

- SEPARATE FROM ITEM 4.0
- TABULATE AS FOLLOWS:

DEPARTMENT	PARAMETER	# OF SAMPLES	EXPLANATION/ CORRECTIVE ACTION	% OF TOTAL SAMPLES ANALYZED

- INDICATE LABORATORY-CONTROLLABLE VS. OUTSIDE OF LAB CONTROL

6. QC DATA/CONTROL CHART SUMMARY

- DESCRIBE OUT-OF-CONTROL DATA POINTS, TRENDS OBSERVED, CORRECTIVE ACTIONS

7. SOPs ISSUED/REVISED

8. TRAINING RECEIVED/CONDUCTED

9. QAPPs RECEIVED/REVIEWED

10. MISCELLANEOUS

TDL 16.0 TRAINING

Training and qualifications of all personnel is documented with resumes which include academic credentials, employment history, experience and professional registrations. An Individual Training record, example given in figure 16-1, is kept for each employee showing on-the- job training and courses taken as well. Each employee has a Personnel Qualification Record, similar to the one shown in figure 16-2, signed by his or her supervisor indicating the procedures which he/she is qualified to perform. Specific training procedures and deocumentation are covered in laboratory SOPs.

TDL 16.1 Personnel Training and Qualification

All quality related activities performed by IT shall be accomplished by personnel qualified on the basis of education, experience, and training.

TDL 16.1.1 Project Staff Training

General training in the reauirements of the Quality Assurance Program is required of all personnel. Formal training sessions are conducted and documented by the Quality Control Coordinator. The training program shall address: regulatory requirements (as appropriate); basic Quality control practices, including checks and balances inherent in the system; responsibilities of the Project Staff; responsibilities of Quality Assurance personnel; and the performance of audits.

New technical employees are given training in Quality Assurance practices and policies. The training must take place soon after the new employee begins work at IT, preferably the first week of employment. It is the joint responsibility of the Laboratory Director and the Quality Control Coordinator to implement this training. New employee training is documented by the Quality Control Coordinator.

Quality Assurance training documentation includes the name(s) of those trained, date(s) of training, and topics covered in training. The documentation is maintained in the Quality/Operations files.

For projects requiring a departure from standard Quality Level I practices, the Project Manager is responsible for reviewing the revised practices with members of the Project Staff. At the request of the Project Manager, this will be done by the Quality Control Coordinator or senior members of the Project Staff. Project-specific reviews shall include contractual and regulatory reuqirements, amendments of standard practice adopted for the project, project-specific procedures, and project-specific Quality Assurance documents. As necessary, Quality Assurance documents or summaries will be issued to the Project Staff. The review is documented by a memorandum to the project file. The documentation includes the date of the review, the topics discussed, and the names of the participating Project Staff.

TDL 16.1.2 Project Staff Qualifications

Project Staffs are primarily composed of professional personnel who are engineers or scientists. Such personnel shall be assigned duties within the capabilities of their education and experience by the Laboratory Director or Project Manager. The Project Manager shall be appointed by the Laboratory Director. Qualifications of all professional personnel shall be documented by resumes which include academic credentials, employment hisotry, experience, and professional registrations. Resumes are maintained in the Quality/Operations files.

Technicians and support personnel performing a technical function for a project are qualified through experience which is indicated in their resumes. These personnel shall also be assigned by the

Laboratory Director or Project Manager based on their capabilities. Technicians and support personnel shall be supervised in their activities by experienced personnel until the Project Manager approves independent performance of their duties.

If subcontractors are used by IT on a project, the Project Manager is responsible for establishing necessary qualification of subcontractor personnel and verifying their capability. Qualifications shall be established in the procurement documents.

If a project requires personnel with specific certifications (such as engineering registration in a given state or technical certification by a state or national organization), personnel meeting the necessary qualifications will be provided by IT.

TDL 16.1.3 Quality Assurance Personnel, Training and Qualifications

Quality Assurance personnel qualifications shall be documented in resumes as for professional personnel.

If audits are required for projects governed by USNRC Quality Assurance requirements (10 CFR 50, Appendix B), lead auditors shall be quialified in accordance with ANSI NQA-1, Supplement 2S-3. Qualifications shall be performed by the Director of ITAS Quality Assurance.

The auditor responsible for conducting a Quality Assurance audit is designated the Lead Auditor. Lead Auditors shall have the expertise necessary to direct all phases of the audit.

Lead Auditors for nuclear-related projects (10 CFR 50, Appendix B requirements applied) shall meet several training and experience requirements for certification, as outlined below. First, they shall obtain a minimum of ten credits using the certification system presented in ASI NQA-1, Appendix 2A-3.

Lead Auditors shall be capable of communicating effectively, in writing and orally. In addition, Lead Auditors shall have participated in a minimum of five Quality Assurance audits within the three-year period immediately prior to initial certification. One of these audits shall have been for a nuclear-related project and must have taken place within the 12-month period prior to initial certification.

Finally, Lead Auditors must undertake and pass a formal examination. This examination may be either written or oral, and should cover applicable aspects of the IT Quality Assurance Program, regulatory requirements, aned Quality Assurance auditing. The Director of ITAS Quality Assurance is responsible for preparing and administering the examination and evaluating examination results. Evidence of the examination shall be documented in the training files.

Continued proficiency of Lead Auditors shall be maintained through active participation in Quality Assurance audits and the preparation and review of Quality Assurance documents.

If a previously certified Lead Auditor fails to maintain proficiency for two consecutive years, formal requalification, by the Director of ITAS QA shall be required. The requalification will consist of:

- Reexamination
- Satisfactory participation in at least one Quality Assurance audit of a nuclear-related project,
- Additional training and/or experience as deemed necessary by the Director, Quality Assurance and Discipline Management.

Formal certification and the annual evaluation of nuclear Lead Auditors shall be documented and maintained by the Director, ITAS Quality Assurance.

TDL 16.1.4 Subcontractor Personnel, Training and Qualifications

If specific qualifications are required of subcontractor personnel, it is the responsibility of the Project Manager to impose such qualification requirements upon the subcontractor through procurement documents, specifications, and other means, and to verify fulfillment. Necessary qualifications may be due to accepted standard of practice, regulation (such as engineering registration), technician registration and/or certification, or craft requirements (such as welder certification).

As appropriate for a specific project, the project manager is responsible for the training of subcontractor personnel with regard to IT quality-related requirements and documenting this training.

Figure 16 - 2

PERSONNEL QUALIFICATION RECORD

NAME: _____ HIRE DATE: _____

TITLE: _____ SUPERVISOR: _____

PROCEDURE NAME, NUMBER	QUALIFY DATE	RENEW BY	APPROVAL

TRAINING SESSION	DATE	APPROVAL

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TDL 17.0 PROJECT PLANNING

This section presents quality-related activities that are appropriate during the project planning stage. This section also includes a discussion of proposal preparation as part of project planning. The selection of activities for implementation, and needed degree of implementation, is dependent upon the needs of the specific project. The means for implementing several topics are included.

Project planning will fall into two types:

1) Large (Research) Projects requiring a full Quality Assurance Project Plan or at least a QAPP form (example of form in Figure TDL 17-1).

2) Short Projects typically requiring less than 200 hours total effort and usually lasting less than two weeks in the lab. The short project requires no QAPP, however, the short project form (like the one shown in Figure 17-2) must be completed.

Figure TDL 17-1
TDL QAPP FORM

Project Name _____

Project Number _____

Client Contact _____

Phone _____

Address _____

Send report to same as above/other _____

Due Date _____

TDL Project Manager (filling out this form) _____

Reviewed By _____

QC Approval _____

List of Subcontractors _____

1) Project Description (include anticipated start and completion dates, brief description of work plan and intended use of acquired data)

_____

_____

_____

_____

_____

_____

2) Project Organization/Responsibilty (list of persons involved, their title and responsibility for this project-may attach org. chart)

_____

_____

_____

_____

3) QA Objectives (include minimum no. of blks, dups, spikes and precision, accuracy and completeness specifications)

_____

_____

_____

Figure TDL 17-1 (Cotinued)

4) Sampling Plan (when and how taken, container requirements storage and preservative and purpose formal or screening)

5) Sample Custody (what forms needed C of C, R for A etc.)

6) Calibrations (requirements and frequency)

7) Analytical Procedures (list method nos. when possible)

8) Data Reduction (List calculations to be performed using A,B,C and X,Y,Z)

9) Surveillance or Audit (by whom prior to start, during or after completion)

10) Preventive Maintenance (list instruments and frequency)

Figure TDL 17-2
TDL Short Project Form

Project Name _____

Project Number _____

Client Contact _____

Phone _____

Address _____

Send Report to same as above/other _____

Due Date _____

TDL Project Manager (filling out this form) _____

Other TDL Personnel Involved _____

TDL Sample Numbers _____

Lab Notebook Reference Pages (book:pages) _____

Computer Data File Reference analyses: _____ report: _____

List of Subcontractors _____

Brief Description of Work Plan _____

TDL 17.1 Project Preparation

The Quality Assurance activities required during a project must be determined and implemented during the planning stage of a project. Quality must be "built-in" from the beginning. Several key decisions must be made during planning, the first being the selection of quality level as discussed in Section TDL 17.2.

However, defining the quality level does not provide all of the detail needed to plan the project. Questions of specific actions to be taken during the project must be addressed. Following are typical questions that should be asked during planning. The list is representative, not inclusive:

- What is the problem? Why is IT involved?
- What do we need to know about the site, or problem?
  - Contaminants present
  - Identification and extent of contaminants
  - Ground water regime
  - Sources of the contanimants and likelihood of unknown contaminants
  - Means for in situ or on-site treatment
  - General chemistry of the waste stream or contaminated soil and water
  - Potential for concentrating contaminants for off-site disposal or treatment
- What is the end product of our work?
  - Preparation of a regulatory submittal
  - Report to client characterizing the site and/or containing recommendations for remediation
  - Report presenting designs or results of technology development
  - Specifications/drawings for direct implementation of remediation
- How can we determine the data we need to know?
  - Collection and analysis of samples

- Bench scale prototype testing
- Geophysical surveys
- Theoretical modeling

- If data is needed, what kind of samples do we need; how are the samples obtained; how will samples be analyzed?
- Do we have the equipment and materials required?
- Do we have needed data sheets; should they be changed or new ones prepared?
- What will the data be used for?
  - Baseline site characterization
  - Ongoing monitoring to verify regulatory compliance
  - Comparison of alternative treatment technologies
  - Remedial design and construction
  - Litigation
- How "good" must the data be?
  - Are there regulatory criteria?
  - What accuracy and precision are required?
  - Are the results to be used for comparative or absolute calculations
  - What are the capabilities of existing analytical procedures?
- What is IT's exposure?
  - Are we developing criteria or implementing criteria development by others (including regulation)?
  - Are we originating design/remediation?
  - Is it expected that we will be required to demonstrate our performance through documentation to the client?
- Who will do the work; are qualified people in IT; are they available?
- What controls are needed for subcontractor services?
- Do we have needed computer software; has it been documented and verified?
- Is peer review planned; who will do it; are they available?
- Finally, has planning addressed the proposal prepared by IT, and included contractual/regulatory demands? Does planning provide the means for satisfying our contract?

TDL 17.2 Selection of Quality Level

Three levels of application of the Quality Assurance Program are defined for use in treatability and engineering projects. See Section 8.3 of this QAM for Analytical QC levels. The three quality levels are:

- Level I - The "standard" IT quality requirements are applied. There is minimal involvement of Quality Assurance personnel during the course of work, generally auditing is not performed.
- Level II - Specifically detailed QC procedures are implemented and the results of QC activities reported.
- Level III - The maximum application of Quality Assurance practices and substantial participation of Quality Control personnel.

If the Project Manager does not select a different level, Level I shall be applied. If Level II or III is selected, a specific implementing Quality Assurance Project Plan is prepared.

The level selected defines the intensity of application of Quality Assurance functions. The major difference between Levels I and II is the required formal audits.

The level selection process must occur during the project proposal preparation stage so that the cost of implementation and schedule considerations can be included. At the latest, the selection shall be made during the planning stage of a project. The level selection is documented by a TDL short or QAPP form stating the project quality level selected.

To select a project quality level, the following points shall be evaluated:

- Institutional needs that control the end use of project information and results (i.e., what must the project satisfy?)

- Criteria which govern the performance of the project (such as regulations, contract requirements, and IT policies)
- State of knowledge necessary to satisfactorily complete the project (i.e., what is the degree of technology required?)
- Project complexity.

Start by defining a general review of project objectives. Consider if the performance of project activities and end use of data and results are controlled by government regulations or expected legal action. Regulations promulgated by th USNRC mandate the adoption of Level II with a full Quality Assurance Program for projects regulated by USNRC. In a similar fashion, federal (e.g., U.S. EPA) and state environmental agencies often mandate through their regulations the use of a formal Quality Assurance Program for a project, so that Level II implementation is appropriate. Projects controlled by consent decrees or impending legal proceedings should be considered for Level III implementation because of the expected need to demonstrate performance.

The project control needed is a function of the expected magnitude of IT liability, industry, or public exposure; the future technical use of data generated; and the objectives of a research and development effort. In addition, the Project Manager or other IT management may require Level II commitment (e.g., formal audits) in any project to serve as a management review tool.

Once the appropriate control category has been determined, the degree of technology required to satisfactorily complete a project shall be defined.

The final determination which must be made prior to selection of the project quality level is the degree of project complexity.

If the project is amended from Level I practice, the following courses of action shall be taken:

- If the project is to be conducted at Quality Level II, the appropriate Quality Assurance Officer shall prepare a Quality Assurance Outline or Quality Assurance Project Plan for the project listing all Quality Assurance activities which exceed Level I and the means for implementation. This could include preparation of work plans, project-specific Quality Assurance Plans, audits to be performed, and formal subcontracting practices. The outline on a Quality Assurance Project Plan shall be reviewed and approved by the Director, Quality Assurance and Discipline Management.

- If Level I practices must be increased, the appropriate Quality Control Coordinator shall prepare, in concert with the Project Manager, a Project Quality Control document for distribution to the affected Project Staff stating the changes.

- Work plans, sampling protocols, and Quality Assurance plans, developed for a specific project, shall be approved.

- Regulatory and client requirements which affect established Quality Assurance practices shall be discussed by the Project Committee and addressed in the form of the documents discussed above.

- If a nonconformance is not resolved to the satisfaction of the appropriate Quality Assurance Officer, the reporting and resolution mechanism shall be instituted.

- If a request for corrective action resulting from an audit is not adequately resolved, a reporting and a resolution mechanism shall be instituted.

TDL 17.3 Subcontractor Procurement

Outside testing services will not be used to bypass quality assurance requirements. The control of procurement documents and resulting subcontractor furnished quality-related items and/or services shall be based on the effect an item or service will have on project requirements and needed results. Subcontractors may be required by IT for: the purpose of providing a service under direct IT supervision (such as drilling); or performing activities independent of IT (such as for specialty testing, supplying equipment, or detail design engineering). The degree of independence of the subcontractor's service must be considered in determining the procurement controls needed. As a general comment, the more independent a subcontractor is of IT supervision, the

stringent should be the Quality Assurance requirements placed upon them.

The procurement process shall be adequately documented. The documentation shall include, as appropriate:

- Requests for proposal
- Specifications and scopes of work
- Quotes (proposals) submitted by subcontractors
- Telephone conversation documentation
- Meeting notes
- Purchase order requisition
- Purchase orders
- Prequalification forms
- Bid reviews
- Bid comparisons
- Change order documentation

TDL 17.3.1 Procurement Document Control

Procurement documents issued by IT; including bid requests, purchase orders, and contracts; shall be prepared, reviewed, and approved as a joint effort of Purchasing, Project, and Quality Assurance personnel. The extent of each group's involvement is a function of the particular project and the item or service procured. Determination of necessary involvement shall be made by the Project Manager. Project procurement documents require review and approval by the Quality Control Coordinator.

Changes in a procurement document shall be subject to the same degree of control as was utilized in the preparation of the original document.

TDL 17.3.1.1 Procurement Document Content

Procurement documents shall state applicable requirements for personnel qualification, technical performance, quality, acceptability, and documentation.

The documents shall, as appropriate, specify the following technical requirements:

- General requirements (scope of work)
- Pertinent codes and standards
- Material composition and/or physical and chemical requirements
- Quantity and scheduling requirements
- Work procedures
- Testing and calibration requirements
- Performance and/or accept/reject criteria
- Reporting requirements.

Procurement documents specify the scope of work in sufficient detail so that it is reasonable to expect that confusion and misunderstandings are eliminated.

Technical requirements shall be included in the procurement documents or referenced to specific drawings, specifications, procedures, regulations, or codes (along with specific revision numbers and issue dates) that describe the items or services to be furnished.

Purchased items and services shall be controlled by invoking appropriate quality-related requirements and elements of the appropriate IT Quality Assurance Program in the procurement documents. The requirements which a subcontractor must satisfy will vary depending on the purpose of the procurement and the degree of subcontractor independence. IT's right to stop work for quality problems should be clearly stated.

The responsibility for compliance with applicable quality requirements and elements of the appropriate IT Quality Assurance Program can be delegated to the subcontractor in the procurement documents, or can be retained by IT. If the responsibility is

delegated, the subcontractor must have a documented Quality Assurance Program suitable for the needs of the project. If responsibility for compliance is retained, IT shall use the procurement documents to "pass down" the performance of specific quality requirements to the subcontractor. The "pass down" provides appropriate requirements which can be met without requiring the subcontractor to have their own Quality Assurance Program.

To verify acceptability, the procurement documents shall provide for IT access to subcontractors' facilities, work areas, and records for auditing and inspection. This may include prequalification (example in figure TDL 17-3) or audits by Quality Assurance personnel, a quality check by Project personnel prior to beginning work or inspections and tests by Project personnel during the performance of work.

Documentation required by IT to provide evidence that materials, equipment, and services are of specified quality shall be identified by the procurement documents. The right of IT to review and approve this documentation prior to acceptance of an item or service should be stated. Instructions for the preparation, control, retention, and disposition of documentation must also be included or referenced.

Required documentation can include:

- Quality Assurance Project Plan
- Procedures and drawings
- Equipment operating manuals
- Test and work plans and logs
- Test and analysis data, results, and verifications
- Calibration records
- Personnel qualifications
- Material certificates of conformance
- Nonconformance records
- Drawing and calculation checkprints
- Computer program verification records

In addition to the above; subcontractor submittals or nonconformance, work progress, results, and other deliverables shall be specified in the procurement documents, as appropriate.

INTERNATIONAL TECHNOLOGY CORPORATION

Figure TDL 17-3

Contractor Prequalification Form

BACKGROUND

1 Your company name and division or operation group. _____

_____

2. Your company address. _____

_____

3. Name(s) of persons to contact for additional information, including addresses and phone numbers.

Name	Address	Phone Number

4 Describe your type of work.

_____

_____

5. Describe assignment for this project. _____

_____

6. List key personnel planned for this project. Please list names, expected positions, and safety performance on last three projects worked on.

Name	Position	Safety Performance

HEALTH AND SAFETY PRACTICES

1 Do you have a written safety program? Yes _____ No _____

2 Do you have an orientation program for new hires? Yes _____ No _____
If yes, does it include instruction on the following?

	Yes	No		Yes	No
a Head protection	_____	_____	i. Fire protection	_____	_____
b Eye protection	_____	_____	j First aid facilities	_____	_____
c Hearing protection	_____	_____	k Emergency procedures	_____	_____
d Respiratory protection	_____	_____	l Toxic substances	_____	_____
e Safety belts and lifeline	_____	_____	m Trenching and excavation	_____	_____
f Scaffolding	_____	_____	n Signs, barricades, flagging	_____	_____
g Perimeter guarding	_____	_____	o Electrical safety	_____	_____
h Housekeeping	_____	_____	p. Rigging and crane safety	_____	_____

3 Do you have a development program for all newly hired or promoted supervisors? Yes _____ No _____
If yes, does it include instruction on the following?

	Yes	No		Yes	No
a Safe work practices	_____	_____	e First aid procedures	_____	_____
b Safety supervision	_____	_____	f Accident investigation	_____	_____
c "Tailgate" meetings	_____	_____	g Fire protection	_____	_____

Figure TDL 17-3
(Continued)

HEALTH AND SAFETY PRACTICES (continued)

4 Do you hold "tailgate" safety meetings? Yes _____ No _____

How often? _____

5 Do you hold site safety meetings for field supervisors? Yes _____ No _____

How often? _____

6 Do you conduct project safety inspections? Yes _____ No _____

If yes, who conducts this inspection (title)? _____

And how often? _____

MEDICAL PRACTICES

1 Do you provide preemployment medical exams? Yes _____ No _____

Describe content and pass/fail criteria. _____

2. Do you conduct periodic/update medical exams? Yes _____ No _____

How often? _____

Describe content and pass/fail criteria. _____

3. Do you conduct postemployment/termination medical exams? Yes _____ No _____

Describe content. _____

ACCIDENT REPORT RESULTS

1 Please use your last year's OSHA No. 200 Log to fill in:

a Number of injuries and illnesses _____

b Number of lost workday cases _____

c Number of restricted workday cases _____

d Number of cases with medical attention only _____

e Number of fatalities _____

2 Employee hours worked last year (do not include any nonwork time, even though paid) _____ hours

ACCIDENT REPORTING MECHANISMS

1 Are accident reports (OSHA 200) and report summaries sent to the following? How often?

	No	Yes	Monthly	Quarterly	Annually		No	Yes	Monthly	Quarterly	Annually
Field Superintendent	___	___	_____	_____	_____	President of Firm	___	___	_____	_____	_____
Vice President of Operations	___	___	_____	_____	_____						

2 How are accident records and accident summaries kept? How often are they reported?

	No	Yes	Monthly	Annually		No	Yes	Monthly	Annually
Accidents totaled for the entire company	___	___	_____	_____	Subtotaled by manager	___	___	_____	_____
Accidents totaled by project	___	___	_____	_____	Subtotaled by foreman	___	___	_____	_____

3 How are the costs of individual accidents kept? How often are they reported?

	No	Yes	Monthly	Annually		No	Yes	Monthly	Annually
Costs totaled for entire company	___	___	_____	_____	Subtotaled by manager	___	___	_____	_____
Costs totaled by					Subtotaled by				

Figure TDL 17-3
(Continued)

INSURANCE INFORMATION

1 List your firm's Interstate Experience Modification Rate for the three most recent years.

19____ _____ 19____ _____ 19____ _____

2. Describe the limits of your worker's compensation insurance coverage.

_____

_____

_____

3 Describe the limits of your comprehensive general liability insurance.

_____

_____

_____

4 Can you arrange for International Technology Corporation to be a named insured on your comprehensive general liability insurance for this project?

Yes _____ No _____

QUALITY ASSURANCE

1 Do you have an internal Quality Assurance Program applicable to your work activities?

Yes _____ No _____

2. Is the program documented in a QA manual and/or written procedures?

Yes _____ No _____

3 Provide a brief description of the QA Program and applicable regulations (such as NRC, EPA) which it meets

_____

_____

_____

_____

_____

4 List governing standards (such as ANSI, ASTM, EPA) which have been adopted for testing, analysis, and work performance.

_____

_____

_____

_____

5. Have you previously been required to contract to have and implement a QA Program?

Yes _____ No _____

If yes, cite examples. _____

_____

_____

_____

6 Have you previously been included under a client's QA Program?

Yes _____ No _____

If yes, did you directly implement part of their QA Program?

Yes _____ No _____

Describe participation. _____

_____

_____

_____

Figure TDL 17-3
(Continued)

QUALITY ASSURANCE (continued)

7 Will you provide IT personnel with access to your facilities/operations for the purpose of prequalification and in-process audits?

Yes _____ No _____

If no, explain _____

8 Which of the following QA practices do you normally perform:

	Yes	No
a. Design Reviews (such as checking, data review, peer review)	_____	_____
b. Calibration	_____	_____
c Preventive Maintenance	_____	_____
d In-process Inspection	_____	_____
e In-process Testing	_____	_____
f Formal Training	_____	_____
g. Personnel Certification	_____	_____
h Corrective Action (identification, reporting, resolution)	_____	_____
i Record Maintenance	_____	_____
j Audits	_____	_____

9 If the service to be provided to International Technology Corporation involves laboratory analyses, list the proficiency and certification programs in which you participate and provide the results of the most recent sample round.

_____

10 If you are supplying materials or equipment, are you prepared to provide certifications and/or test results?

Yes _____ No _____

Describe the industry standards which generally apply.

_____

11 List applicable certifications of key personnel (or include in attached resumes).

_____

12 Will you provide applicable quality-related records if requested by International Technology Corporation such as fabrication drawings, as-built drawings, calibration records, operation logs, QA sample results?

Yes _____ No _____

Finally, procurement documents shall require subcontractors to incorporate applicable requirements in subtier procurements.

TDL 17.4.1.2 Procurement Document Review and Approval

Procurement documents shall be reviewed to verify that applicable IT, regulatory, and client requirements (performance, quality, acceptability, and documentation) have been incorporated. The reviews shall also verify that changes in the documents, as a result of bid evaluations or precontract negotiations, have been made. Reviews of bid requests and purchase orders shall be performed prior to their release. Reviews of contracts shall be performed prior to contract award. Purchasing, Project, and Quality Assurance personnel shall participate, as necessary. The Quality Control Coordinator will review all technical purchase order requisitions. Documentation of the review shall be by signing the document draft, or by signing a routing stamp on the document.

Contract and subcontractor service purchase order approval shall be indicated as required by IT purchasing policies. Bid requests shall be approved by the Project Manager and/or responsible Laboratory Director or designated Project Director for a specific project. Copies of all contracts, bid requests, and purchase orders for project services and review copies shall be maintained as records for the project.

TDL 17.5 Peer Review

This Quality Assurance Program provides controls for the formal verification (checking) of documents such as calculations and the presentation of information in the form of drawings, logs, and tables. Review and necessary approvals are also cited for quality related documents. However, quite often during the course of a project or proposal, verification of technical decisions and concepts such as interpretation of data and the evaluation of results is required so that the project or proposal can proceed on a

sound conceptual basis. The review of concept, or approach, may be needed for the following:

- During the planning stage have appropriate steps been taken to meet the goals of the project? This could include:
  - Does the sampling plan provide for the number, type, volume, etc. of samples anticipated to provide needed data?
  - Are analytical procedures adequate to provide data of sufficient detection, accuracy, precision, completeness for the project needs?
  - Are appropriate computation methods to be used during design?
- Are data of sufficient quality and properly interpreted so that conclusions can be justified and demonstrated?
- If design parameters are assumed, are they reasonable for the computations performed? What is the effect of variation of the assumptions upon results?
- Do the results presented by IT in the format of a report, or other document (Section 9.3), adequately present the work performed and the conclusions reached? Do the results fulfill the objectives of the project?

The mechanism to be used in the Technology Development Laboratory for such verification is peer review.

The need for peer review is dependent upon the scope of the individual project. The Laboratory Director and Project Manager shall determine during the planning stage of a project if peer review will be implemented, the points in the project when the review will be performed, and the individual(s) who will perform the review. The extent and importance of peer review for a specific project should be based on:

- Technical complexity of the work
- Experience of project personnel (peer review should also be a learning/teaching mechanism)
- Difficulty of implementing and fulfilling the methods/procedures to be used in the project

3 4 0264

- Complexity of the logistics required to manage the project
- Effect upon project schedule and succeeding project stages if part of the work does not meet project goals so that either project goals/objectives must be revised or the work repeated
- Potential for liability.

For peer review to be effective, the schedule for the reviews must be estimated and included as part of the project schedule, and time must be allowed for the reviews. The selected peer reviewers should be notified of their expected participation as soon as possible.

A peer reviewer should be selected based on the following:

- The reviewer must be independent of the project. The reviewer must be sufficiently informed concerning the project, but should not be making the decisions which determine the course of the project. It is intended that peer review be an "outside" overview of the project.
- The reviewer must be a person knowledgeable in the area of work, preferably a senior technical person. Peer reviewers can be part of the IT organization, or outside consultants or may sign off on project documents.

At the conclusion of a peer review, the reviewer(s) shall prepare a report to the Laboratory Director, Project Manager, and Quality Control Coordinator or may sign off on project documents.

ds/CP25

INTERNATIONAL TECHNOLOGY CORPORATION

QUALITY ASSURANCE MANUAL
IT ANALYTICAL SERVICES

Prepared by:

Paul E. Mills
Director, Quality
Assurance/Control
Analytical Services

Approved for Issue:

Raul A. Deju
Senior Vice President
Environmental Projects Group

Brad S. Figley
Vice President
Analytical Services

David E. Troxell
Director, Quality Assurance
and Discipline Management
Environmental Projects Group

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INTERNATIONAL TECHNOLOGY CORPORATION

STATEMENT OF MANAGEMENT POSITION

IT Corporation is committed to providing quality services for environmental management; services which meet the needs of our clients, satisfy regulatory requirements, and are commensurate with the current state of the art.

To satisfy our clients' quality objectives, to meet regulatory requirements, and to comply with IT Corporate-wide requirements, IT Analytical Services (ITAS) has adopted a comprehensive Quality Assurance Program. The principles and practices of the Program apply to every employee at every level within ITAS; they are fundamental to the way we do business and to the services we provide.

The Quality Assurance Manual is an overall statement of Program policy. This Manual provides guidance to ITAS personnel in fulfilling their responsibilities, and serves as a statement to external parties of ITAS' commitment to quality.

Implementation of the Program is the responsibility of all ITAS personnel. Management at every level has the commitment, duty, and authority to insist that these responsibilities are met, and that the principles and practices of the Program are followed and implemented.

Quality Assurance Coordinators are assigned in all ITAS laboratories to see that the Program is implemented on a day-to-day basis as intended. Each Coordinator has the authority and duty to stop work if and when necessary to satisfy Program requirements.

To verify that the Quality Assurance Program is successfully implemented, the laboratories are independently audited by the ITAS Director of Quality Assurance/Control, who is directly responsible to the Vice President, Analytical Services. In addition, the laboratories are subject to audits by the Environmental Projects Group Director of Quality Assurance and Discipline Management, and by various regulatory authorities and other outside agencies.

Brad S. Figley
Vice President
Analytical Services
February 1, 1988

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INTERNATIONAL TECHNOLOGY CORPORATION

Dated: August 1, 1984
Revision No.: 3
Date Revised: June 17, 1988

MANUAL OF STANDARD OPERATING PROCEDURES SPECIAL ANALYSIS LABORATORY IT ANALYTICAL SERVICES KNOXVILLE

Prepared by:

IT SAL Staff

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IT ANALYTICAL SERVICES
TECHNOLOGY DEVELOPMENT/
SPECIAL ANALYSIS LABORATORY/PC 4622

- **LABORATORY DIRECTOR** — Richard Miller (G)
  - **TECHNICAL DIRECTOR** — (Vacant) (R)
  - **QC COORDINATOR** — Patricia Carswell (R)
  - **H&S COORDINATOR** — Rich Miller (Acting) (R)
    - M. Krstich, Env. Comp
  - **ACCOUNTING MANAGER** — (Vacant) (R)
  - **SALES MANAGER** — Joseph McLaughlin (R)
  - **LAB SYSTEMS MANAGER** — Bruce Wagner (G)
    - **LIMS OPERATION** (R)
      - (Bruce Wagner), Acting LIMS Manager
    - **ADMIN & SAMPLE CONTROL** (R)
      - M. Whipple, Sample Control Clerk
      - D. Smith, Team Leader
        - Susan Oxendine, Secretary (Temp)
        - R. Ford, Secretary
    - **REPORT & PROPOSAL PROD** (R)
      - (D. Smith), Team Leader
        - Q. Clark Smith, Specialist
  - **ENV TECH DEV CENTER FACILITY MANAGER** — Thomas Geisler (G)
    - C. Lauer, Chemist
    - R. Cole, Chemist
    - D. Edde, Secretary (Temp)
  - **PROCESS DEVELOPMENT GROUP MANAGER** — Edward Alperin (G)
    - **TREATABILITY TESTING** (Y)
      - E. Stine, Chemist
      - C. Morren, Chemist
      - A. Groen, Chemist
      - D. Handly, Chemist
      - K. Sadler, Engineer
      - (Vacant), Engineer
      - C. Morgan, Technician
      - K. Ferguson, Facilities Technician
  - **SPECIAL ANALYSIS GROUP MANAGER** — Duane Root (G)
    - N. Conrad, Project Manager
    - D. McClure, Project Manager
    - C. Pudelek, Project Manager
    - **SAMPLE PREP** (R)
      - D. McNeil, Chemist
      - J. Brewer, Technician
      - J. Hendrickson, Technician
    - **SPECIAL ANALYSIS** (Y)
      - W. Li, Chemist
      - P. Parsley, Chemist
      - (Vacant), Chemist
      - D. Demott, Technician Part Time
    - **GC/MS** (Y)
      - B. Hall, Chemist
      - N. Meehan, Chemist
      - D. Thal, Chemist
      - (Vacant), Chemist

Exempt Employees	24
Non Exempt Employees	6
Hourly Employees	0
TOTAL EMPLOYEES:	30
Full Time Equivalent	30

Approval: RMiller Date: 1/8/91

DUANE K. ROOT

Professional Qualifications

Dr. Root is a project chemist and the analytical manager for the Technology Development Group. He has extensive experience, both professional and academic, in analytical chemistry. His areas of specialty include chromatographic methods of separation and electrochemical principles as they apply to synthesis and electroanalytical techniques. His process development experience includes process conceptualization, fundamental experimentation, overall evaluation, and scale-up.

Education

Ph.D., Analytical Chemistry, Texas Tech University, Lubbock, Texas; 1980
B.S., Chemistry, University of Michigan, Ann Arbor, Michigan; 1973

Experience and Background

1989 - Present — Analytical Manager, Technology Development Laboratory, IT Corporation Knoxville, Tennessee. Responsible for the management and operation of the following:

- Special Analytical Group which is responsible for analytical method development, and performing non-routine analyses involving gas chromoatography (GC)/mass spectrometer (MS), liquid chromotograph (LC), instrument control (IC), ultraviolet/visible/infrared (UV/VIS/IR) spectrophotometric and atomic absorption (AA) methodologies.

- GC/MS Analytical Group which performs analyses by either low resolution or high resolution mass spectrometry for chlorinated and unchlorinated dioxin and furan compounds as well as polychlorinated biphenyls (PCBs).

1984 - 1989 — Chemist, Technology Development Laboratory, IT Corporation, Knoxville, Tennessee. Responsible for the development of new technology, applying existing technology to new applications, and providing technical support to operating divisions. Experience includes:

- Project leader for the development and evaluation of technology for the chemical detoxification of PCB and chlorinated dioxin contaminated fluids and solids. This work ranged from bench-top experiments to scale-up process evaluation.

- Evaluation of the use of UV radiation for photolytic decomposition of chlorinated dioxins and associated compounds.

- Evaluation of dye sensitized photo-oxidation of organic constituents in aqueous samples.

- Development of analytical methods for the analysis of environmental contaminants.

- Supervision of analyses and the establishment of quality assurance/quality control standards for the group.

1980 - 1984 **Project Leader, Dow Chemical Company, Plaquemine, Louisiana.** Responsible for research and development of electrochemical processes. This included development of electrochemical systems for energy applications, such as fuel cells and batteries, and process applications from chemical production to wastewater treatment. In addition, provided expertise for analytical applications of electrochemical methods.

- Major project research was the development of high surface area, gas diffusion cathodes for the reduction of oxygen. Other research experience included development and characterization of polymer composite - membrane type electrodes, membrane/electrode composites, solid polymer electrolyte systems, and polymer modified electrodes.

1974 - 1976 **Environmental Chemist, Clayton Environmental Consultants, Southfield, Michigan**. Responsibilities included specialized analyses of, and analytical methods development for, organic and inorganic contaminants of environmental samples. Responsibilities also included sampling methods development, field sampling, and establishing field laboratories for on-site analyses.

Professional Affiliations

American Chemical Society
The Electrochemical Society

Publications

D. K. Root, "Electrochemical Behavior of Zinc(II) Tetraphenylporphyrin in the Presence of Pyridine. The Mechanism and Kinetics of the Peripheral Substitution Reaction Between the Cation Radical and Pyridine," (Revision pending - J. Inorg. Chem.).

D. K. Root, 1982, "Electrocarboxylation of Imines. Preparation of N-Substituted a-Amino Acids." J. Electrochem. Soc., 129, 1231.

D. K. Root, 1978, "Reductive Alkylation of Phenazine. Electrochemical Preparation of 5,10-Dihydro-5,10-Dimethyl and 5,10-Dihydro-5, 10-Diethyl-Derivatives," J. Org. Chemical, 43, 778.

PATTI B. CARSWELL

Professional Qualifications

Ms. Carswell is a chemist at the Technology Development Laboratory with six years of experience in analytical chemistry. Her analytical experience includes work with Polychlorinated Biphenyls (PCBs), Polycyclic Aromatic Hydrocarbons (PAHs), and Total Recoverable Petroleum Hydrocarbons (TRPH). Her responsibilities have included method development, sample extraction, and analysis. Her analytical capabilities have included gas chromatography/flame ionization detector/electron capture detectors/thermal conductivity detector/atomic emission detector (GC/FID/ECD/TCD/AED), liquid chromatography (HPLC), and infrared spectrophotometry (IR).

Education

B. A. Chemistry, Carson–Newman College, Jefferson City, Tennessee, 1981.

B. A. Biology, Carson–Newman College, Jefferson City, Tennessee, 1981.

East Tennessee State University, Johnson City, Tennessee, 1975–1979.

Walters State Community College, Morristown, Tennessee, Summer sessions 1977–1979.

Experience and Background

1990 – Present — Quality Control Coordinator, Technology Development Laboratory, IT Corporation, Knoxville, Tennessee.

Responsible for:

- Monitoring laboratory quality assurance activities to determine conformance with corporate policy and procedures.
- Advising and training of staff in quality assurance issues.
- Reviewing work proposals and QAPPs for quality assurance aspects.
- Reviewing data and all reports produced in the Technology Development Laboratory.
- Maintaining quality and operations files.

1986 – 1990 — Chemist, I.T. Corporation, Technology Development Laboratory, Knoxville, Tennessee.

- Responsible for routine extractions/analyses by standard U.S. Environmental Protection Agency (EPA) protocols.
- Performed analytical method development on routine and non–routine analyses.
- Provided analytical support for pilot– and bench–scale processes.
- Responsible for compliance of Tennessee Radioactive Material License in regard to Electron Capture Detectors.

1984 – 1986 Laboratory Technician, Great Lakes Chemical Corporation, Newport, Tennessee. Was responsible for testing all samples received on shift, including in–process testing and final controls on finished products. Experience included GC, HPCL, IR, and wet chemistries.

Was responsible for supervision of laboratory in lab manager's absence and in start–up process situations.

1982 – 1984 Cardiac Testing Assistant, Morristown–Hamblen Hospital, Morristown, Tennessee. Responsibilities included performing Echocardiograms, Thallium Stress Tests, EEGs, Phonocardiograms and Holter Monitor Scanning.

Professional Association

American Chemical Society

WALTER W. LI

Professional Qualifications

Mr. Li is an organic/analytical chemist and Technical Associate with experience in extraction, analysis, and quantitation of various environmental pollutants in a wide range of matrices. His experience includes work with dioxins/furans, polynuclear aromatic hydrocarbons (PAHs), pesticides, herbicides, polychlorinated biphenyls (PCBs), and organometallic analyses such as tributyltin and methyl mercury. He is currently with the Technology Development Laboratory and is responsible for developing extraction and instrument analytical procedures for analyses of compounds where no formal or standard methods exist or apply. In addition, his analytical capabilities include gas chromatography/flame ionization/electron capture/atomic emission detectors (GC/FID/EC/AED), gas chromatography/mass spectrometry (GC/MS), high pressure liquid chromatography (HPLC), and atomic absorption (AA). Mr. Li has also has worked in university and corporate settings involving organic reactions in molten salt media and the recovery of petroleum products from waste rubber.

Education

B.A., Chemistry, Emory University, Atlanta, Georgia; 1978
Graduate work, University of Tennessee, Knoxville, Tennessee; 1978-1980

Experience and Background

1987 - Present — Analytical Specialist, Analytical Services/Technology, Technical Development Laboratory, IT Corporation, Knoxville, Tennessee. Responsible for instrumental, and analytical procedural method development/modification and trouble shooting problems with current methods.

- Analytical support for pilot- and bench-scale processes and other project development work
- Responsible for routine analyses by standard U.S. Environmental Protection Agency (EPA) protocols
- Responsible for some quality assurance/quality control (QA/QC) duties.

1983 - 1987 — Supervisor of Special Analysis Extraction Laboratory, Technology Development Laboratory, IT Corporation, Knoxville, Tennessee. Was responsible for supervising and scheduling work for extraction of dioxin/furans from various environmental matrices. Other duties included:

- Method development/modification
- Creating and maintaining analytical spiking solutions and stock standards
- GC/MS analysis
- Training new employees for working in the high-hazard laboratory.

1980 - 1983 Analytical Chemist, Technology Development Laboratory, IT Corporation, Knoxville, Tennessee. Was responsible for GC/MS analysis and extraction of dioxin in soil and water and Liqid Chromatography (LC) analysis of PAHs using ultraviolet (UV)/fluorescence detectors. Other duties included Total Organic Halogens (TOX) analysis of water, GC analysis of pesticides, and National Institute of Occupational Safety and Health (NIOSH) air samples. Also assigned to on-site work in a mobile laboratory analyzing water and soil samples by GC/FID for perchloroethylene, styrene, and vinyl chloride.

1980 Analytical Chemist, IT Enviroscience, Knoxville, Tennessee. responsible for extraction and analysis of trace contaminants in environmental samples. Experience included GC/FID/EC work for analysis of pesticides, herbicides and PCBs, LC/UV/fluorescence work for analysis of PAHs, method development work, and Resource Conservation and Recovery Act (RCRA) analyses. Extraction of air, water, and soil samples were performed using various internal, U.S. Environmental Protection Agency (EPA), and other published methods.

1979 - 1980 Research Assistant, University of Tennessee, Knoxville, Tennessee. Participated in research work on U.S. Department of Energy (DOE) grant involving organic reactions in molten salt media.

1979 Research assistant, Ford Motor Company, Dearborne, Michigan. Involved in research work on DOE grant involving pyrolysis of scrap rubber in the presence of molten salt catalysts for the recovery of petroleum products.

1979 Technician, University of Tennessee, Knoxville, Tennessee. Measured conductivities through various molten salt mixtures.

Registrations

American Chemical Society (ACS)
IT Corporation Technical Associate

Publications

Larsen, J. W., Li, W., Weisen, R., I and EC Process Design and Development, Vol. 23, p. 648, 1989.

INTERNATIONAL TECHNOLOGY CORPORATION

ANALYTICAL SERVICES

LABORATORY REPORT

Gregory E. Johnson
E.A. Engineering, Science, and Technology
15 Loveton Circle
Sparks, MD 21152

January 24, 1991

TDL Project Number: 482826

Client Project ID: Methyl Mercury Method Validation

I. Introduction

The Technology Development Laboratory (TDL) of IT Corporation has experience analyzing sediment, water and fish samples for monomethyl mercury from a project conducted for the USEPA SMO (SAS #5003A) in November, 1989. Based on the work conducted for that project we have defined our procedures for the analysis of sediments and waters and have conducted instrument calibration and detection limit studies. The method description for sediments and waters, QA/QC activities, and results of instrument calibration and the detection limit study are described herein.

A standard and validated method for analysis for soil, sediment and water samples has not been published. The procedures we have used are modifications from a published method for fish and shellfish: AOAC Official Methods of Analysis 25.146-25.152, "Mercury (Methyl) in Fish and Shellfish Gas Chromatographic Method".

Reviewed and Approved:

Duane K. Root
Analytical Operations Manager
qhc-s\WWL048

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304 DIRECTORS DRIVE
KNOXVILLE, TENNESSEE

II. Analytical Results/Methodology

Water Samples

1. Ten (10) mls of sample is placed into a 40 ml VOA vial with TFE lined cap and acidified with 1 ml of 1N HCl/5% NaCl solution. The sample is then extracted with 3-5 mls of benzene by hand shaking the sample for 15 seconds. After settling, the benzene is pipetted off and saved. An additional extraction is performed with benzene and the benzene layers are combined. The total volume of the extract is recorded and used in calculating the concentrations of methyl mercury.

Sediment Samples

1. Four (4) grams of sample is weighed into a 40 ml VOA vial with TFE lined cap and extracted with 30 mls of acetone by hand shaking the sample for 15 seconds. The sample is then centrifuged at 1500 rpm for 10 minutes and the acetone is decanted off and discarded. This step is repeated 2 more times.

. Thirty (30) mls of benzene is added to the acetone washed sample and shaken by hand for 30 seconds. The sample is then centrifuged and the benzene is decanted off and discarded.

3. Four (4) mls of the HCl solution (1:1, v:v) is added to the prewashed sample and shaken for 30 seconds. Then 5 mls of benzene is added, and the sample is shaken by hand for 2 minutes. After centrifugation, the benzene is pipetted off and saved. Two more extractions are repeated with 5 mls of benzene each. The total volume of the extracts is recorded and used in calculating the concentrations of methyl mercury.

Samples may be run using the total volume from extraction, or concentrated to a lower volume by nitrogen gas blowdown.

Instrumentation - A Hewlett Packard 5890 gas chromatograph (GC) equipped with an electron capture detector is used for the analysis. The GC column is a Restek 30 meter megabore Rtx-5. The GC column requires conditioning prior to sample or standard analysis with a solution of 1 mg/ml mercuric chloride in benzene.

GC Conditions are:

Injector Temp.: 210°C
Detector Temp.: 270°C
Program: Initial temperature of 110°C for 5 minutes and then ramp to 115°C at 10 degrees per minute and hold for 2 minutes.
Flows: Helium carrier was at 7 mls/min + 25 mls/min makeup gas

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II. Analytical Results/Methodology (continued)

Data is collected and processed using the PE/Nelson Turbochrome II Analytical Chromatography System.

If the samples are concentrated down to a 1:1 ratio of solvent to starting sample weight, the interferences in the sample may be significant. These interferences may not interfere with the methyl mercury, but they have an impact on column lifetime. If very low detection limits are required, the samples can be concentrated to a 1:1 solvent to sample weight ratio, but this will also increase the maintenance on the column. Our instrumentation seems to respond reliably at a level of 5 ppb. Below that level, and with large amounts of interferences, the response and peak shape are more visibly affected.

Performance - A calibration of the instrument was performed using standards of monomethyl mercury chloride in benzene at five concentration levels. Results of this calibration are shown in Appendix A along with the associated chromatograms. Daily standards were also analyzed to verify instrument calibration. The response from these standards were within 20% of the initial calibration. See Appendix B.

A detection limit study was also completed using the low standard. The standard was run 5 times for the study. From the results of the analyses, a method detection limit (MDL) and a method quantification limit (MQL) were calculated. See Appendix C.

III. Quality Control

Sample Holding Times - Holding times will be 10 days for the extraction and 30 days for the analysis. This is based on what was defined for previous work for USEPA (SAS #5003A).

Method Blank - A method blank will be analyzed with each batch of samples extracted, at a minimum rate of 1 out of 20 samples analyzed.

Matrix Spike - A matrix spike sample will be analyzed at a minimum rate of 1 out of 20 samples analyzed.

Matrix Spike Duplicate - A matrix spike duplicate sample will be analyzed at a minimum rate of 1 out of 20 samples analyzed.

Initial Calibration - An initial calibration curve will be generated by running at least a three point curve. The RSD from the standard responses from the initial calibration will be less than 25%.

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III. Quality Control (continued)

Continuing Calibration - During the actual sample analysis, a mid level standard will be run and must meet within 25% of the calibration. Since the gas chromatography system will need constant reconditioning with the mercuric chloride solution, response verification standards (RVS's) will be run interspersed with the samples to insure the retention times and response are not degrading. The frequency of RVS analyses will be determined by the analyst's assessment of the interferences with the samples run, but will be a minimum of one every twelve hours. If the RVS response fails to meet within 25% of the calibration, the column will be reconditioned and the analysis of the RVS will be repeated. If the RVS fails to meet calibration after reconditioning, then a new (initial) calibration will be performed. In any case, an analysis will be repeated if it is not confirmed by RVS's meeting calibration before and after the analysis.

In many cases where the sample matrix interferences are so large, column maintenance (cutting off a section from the front end and changing the injector sleeve) could also be frequent. In these cases, a RVS will be run and as long as the response is within the continuing calibration parameters, the initial calibration will still be used. The new retention time will be noted so that subsequent runs will be correctly identified.

Detection Limits - Interferences will also determine the final detection limit of each sample since dilutions of the final extract may be necessary.

Method Modifications - Since this method is not a published and approved method, some modifications to the described scheme may be necessary. Steps such as adding more acid if the sample is not fully saturated, or if emulsions form, other steps or more benzene may be needed. These will be documented and non conformances will be filed.

APPENDIX A
INITIAL CALIBRATION SUMMARY

APPENDIX A
Page 1 of 1
Gregory E. Johnson
Methyl Mercury Method Validation
Date: January 24, 1991
TDL Project No.: 482826

IT ANALYTICAL SERVICES
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KNOXVILLE, TENNESSEE

MONOMETHYL MERCURY CHLORIDE

INITIAL CALIBRATION SUMMARY

Run Date: December 7, 1990

LEVEL	CONCENTRATION (ppb)	RESPONSE FACTOR
1	5	1.37×10^{-4}
2	15	1.32×10^{-4}
3	50	1.15×10^{-4}
4	151	1.02×10^{-4}
5	301	9.89×10^{-5}

Mean RF = 1.17×10^{-4}
RSD = 14.7%

Sample File : C:\2700\DATA\EAHG1P.smp
Created on : on : 12/4/90 12:51
Edited on : on : 12/8/90 16:00
Number Of Times Edited : 0

Sample Description :
Default Injection Volume = 1.0000 ul
An External Standard Calibration Will Be Used
Unknown Peaks Will Use The Response Factor Of The Nearest Neighbor

Component Information :
MMHgCl
Retention Time : 4.550 min Search Window: 5 sec. 5 %
Reference Component: (Find Largest Peak)
Group Name :
✓ Calibrating Area versus Amount Using a Pt. to Pt. Fit
✓ Curve Will Be Forced Through The Origin
Amounts Will Not Be Scaled Prior To The Regression
Weighting Factor For the Regression: 1
Calibration Levels:

	Level Name	Amount	Area	Height	ISTD Resp.	ISTD Amt.	# Replicates
1.37×10^{-4}	1	5.0000	76578.59	3200.53	----------	----------	1
1.52×10^{-4}	2	15.0000	113711.50	24990.66	----------	----------	1
1.5×10^{-4}	3	30.0000	434789.00	91084.16	----------	----------	1
1.02×10^{-4}	4	151.0000	1479726.25	297180.63	----------	----------	1
9.99×10^{-5}	5	701.0000	7042735.00	553695.81	----------	----------	1

✓1 CKK 12/20/90

$\bar{r} = 1.17 \times 10^{-4}$
%RSD = 14.6%

Point to Point Through Zero

Calibration Replicate Listing:
Component: MMHgCl

Level : 1

Retention	Area	Height	Amount	ISTD Response	ISTD Amount	Date/Time		File
4.183	78498.00	9526.29	5.0000	----------	----------	12/7/90	14:30	EAHG052
4.200	74579.00	7872.77	5.0000	----------	----------	12/7/90	14:33	EAHG053

Level : 2

Retention	Area	Height	Amount	ISTD Response	ISTD Amount	Date/Time		File
4.217	117389.00	25598.31	15.0000	----------	----------	12/7/90	14:30	EAHG054
4.217	109934.00	24203.00	15.0000	----------	----------	12/7/90	14:33	EAHG055

Level : 3

Retention	Area	Height	Amount	ISTD Response	ISTD Amount	Date/Time		File
4.233	444047.00	94487.48	30.0000	----------	----------	12/7/90	14:30	EAHG056
4.233	425531.00	87684.85	30.0000	----------	----------	12/7/90	14:33	EAHG057

Level : 4

Retention	Area	Height	Amount	ISTD Response	ISTD Amount	Date/Time		File
4.250	1442291.00	279111.34	151.0000	----------	----------	12/7/90	14:30	EAHG058

4.250	1517161.50	295249.44	151.0000	----------	----------	12/7/90	14:33	EAHG059

Level 3

Retention	Area	Height	Amount	ISTD Response	ISTD Amount	Date/Time		File
4.233	3042735.00	553695.81	301.0000	----------	----------	12/7/90	14:30	EAHG061 } same file
4.233	3042735.00	553695.81	301.0000	----------	----------	12/7/90	14:33	EAHG061 }
4.267	3108172.00	602328	301.24			12/7/90	14:17	EAHG060

Retention time over ~~10~~ 9 runs DKR 12/20/90

$\bar{X}$ = 4.235 mins σ = 0.0327 min

% RSD = 0.77%

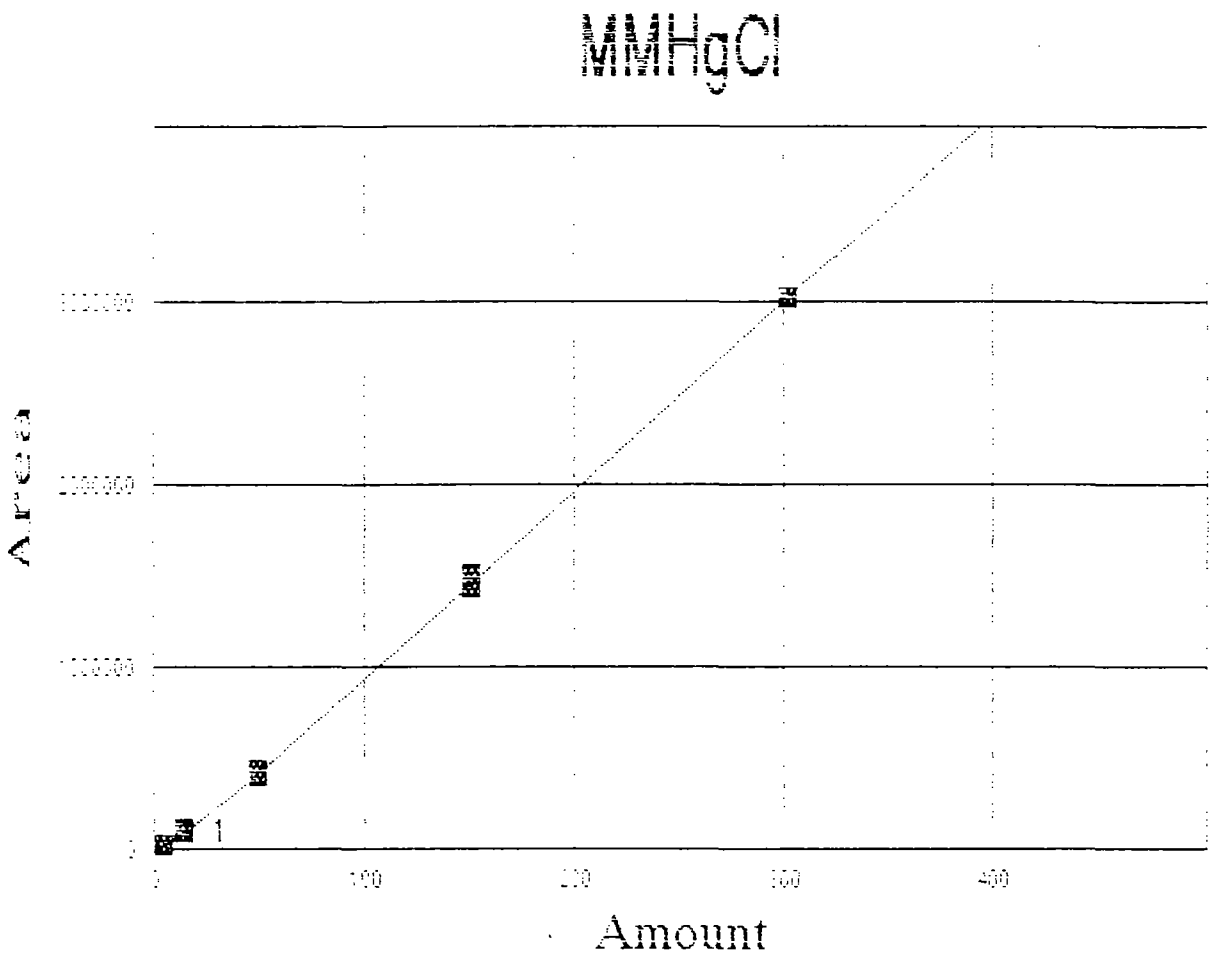
EAHG060 NOT USed

QC DKR 12/20/90

Av area { EAHG061 / EAHG060 = 3075453.5 R_f = 9.79x10^{-5} %RSD still 15% & $\bar{X}$ R_f still 1.17x10^{-4}

[illegible] used only EAHG061 in calibration [illegible] 12/21/90
with duplicate [illegible] for other + [illegible]

Curve #1 : Point to Point Fit - Forced Through the Origin
Weighting Factor = ... to Weighting



CALPLOT Data Lists For Sample File: "C: [illegible] DATA\EAHG1P.SMP"
Date: [illegible] Time: [illegible]

3 4 0298

Data Lists for Calculated Values for Each Plot
[illegible] Forced Through the Origin
Weighting Factor = 1.0 (No Weighting)

Level Name	Observed X-Value	Calculated X-Value	Delta	%Diff.	Observed Y-Value	Calculated Y-Value	Delta	%Diff.
1	5.0000	5.2533	-0.2533	-5.066	38498.0000	36538.5000	1959.5000	5.090
1	5.0000	4.7319	0.2681	5.362	34579.0000	36538.5000	-1959.5000	-5.667
2	15.0000	15.4339	-0.4339	-2.893	117889.0000	113911.5000	3977.5000	3.374
2	15.0000	14.4859	0.5141	3.427	109934.0000	113911.5000	-3977.5000	-3.618
3	50.0000	50.3948	-0.3948	-0.790	444047.0000	434789.0000	9258.0000	2.085
3	50.0000	49.9902	0.0098	0.020	425531.0000	434789.0000	-9258.0000	-2.176
4	151.0000	147.3816	3.6184	2.396	1442291.0000	1479726.2500	-37435.2500	-2.596
4	151.0000	154.5926	-3.5926	-2.379	1517161.5000	1479726.2500	37435.2500	2.468
5	301.0000	301.0000	0.0000	0.000	3042735.0000	3042735.0000	0.0000	0.000
5	301.0000	301.0000	0.0000	0.000	3042735.0000	3042735.0000	0.0000	0.000

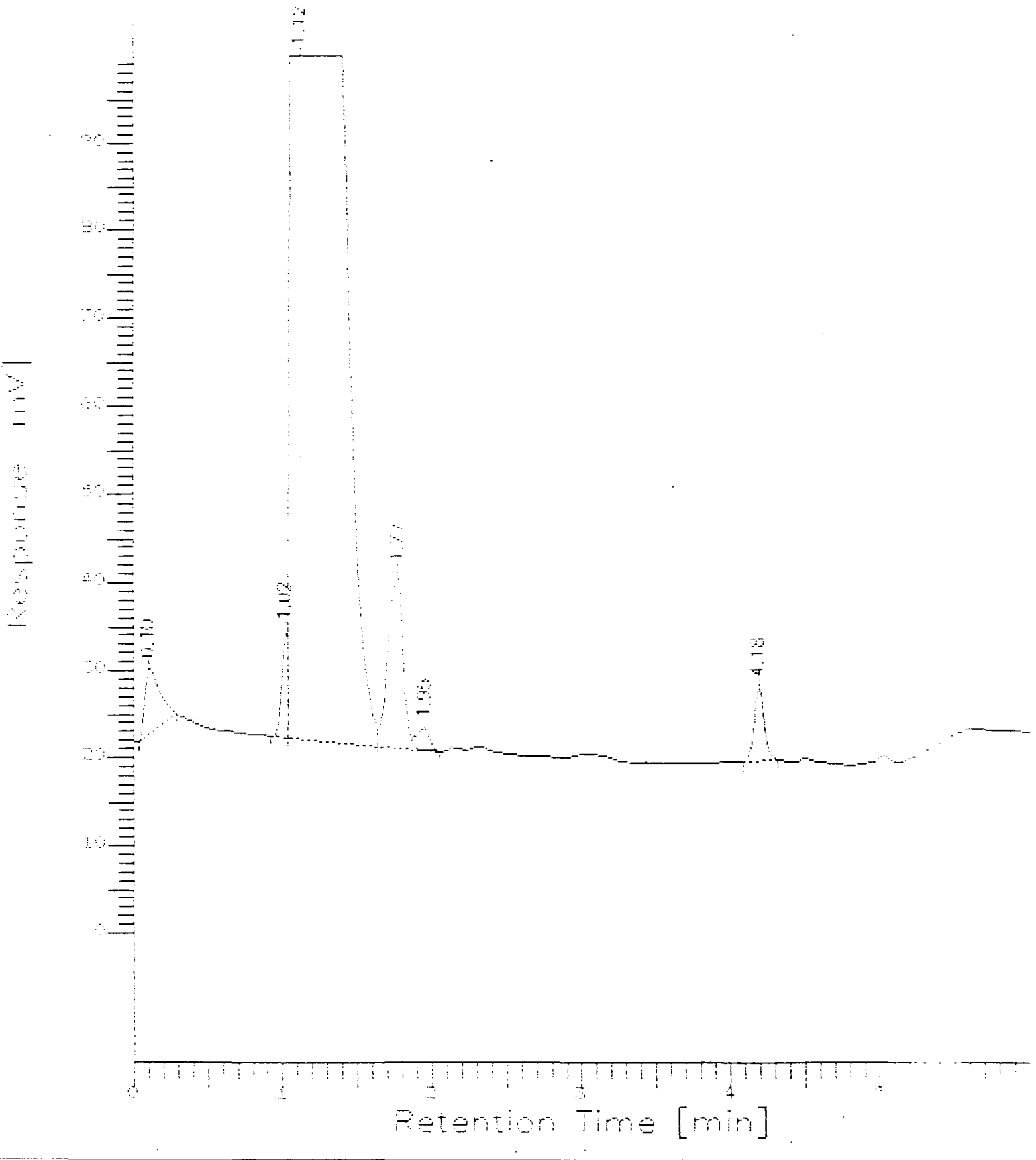
Sample ... 264:06-mmHg STD IC 5ppb

DEFAULT REPORT

...	...	...	...	...	...	...	...	...
1		...	...	...	...	...	...	...
2		...	...	...	...	...	...	...
3		...	...	...	...	...	...	...
4		...	...	...	...	...	...	...
5		...	...	...	...	...	...	...
6		...	...	...	...	...	...	...
			9548966	1019498	9.55	9.55		

Chromatogram

FileName : C:\CHEM32\DATA\GAH0052.raw
Start Time : 0.00 min
Scale Factor: 1
End Time : 5.00 min
Plot Offset: 0 mV
Date : 12/7/90 13:09
Low Point : 0.00 mV
Plot Scale: 100 mV
Page 1 of 1
High Point : 100.00 mV



Sample Name : 664:06 - MMH_2 STD II @ 5ppb Time : 12/7/90 13:17
Acq. Number : 52 Study :
Operator : DWL

Interface # : 4 Channel : 0 A/D mV Range : 1000
AutoSampler : Hewlett-Packard 7673A
Rack/Vial : 0/1

Data Acquisition Time: 12/7/90 13:00
Delay Time : 0.00 min.
End Time : 6.00 min.
Sampling Rate : 1.0000 pts/sec

Raw Data File : C:\2700\DATA\EAHG055.raw
Result File : C:\2700\DATA\EAHG055.rst
Instrument File: C:\2700\DATA\EAHG.ins
Process File : C:\2700\DATA\EAHG.prc
Sample File : C:\2700\DATA\EAHG.smp
Sequence File : C:\2700\DATA\EAHG.seq

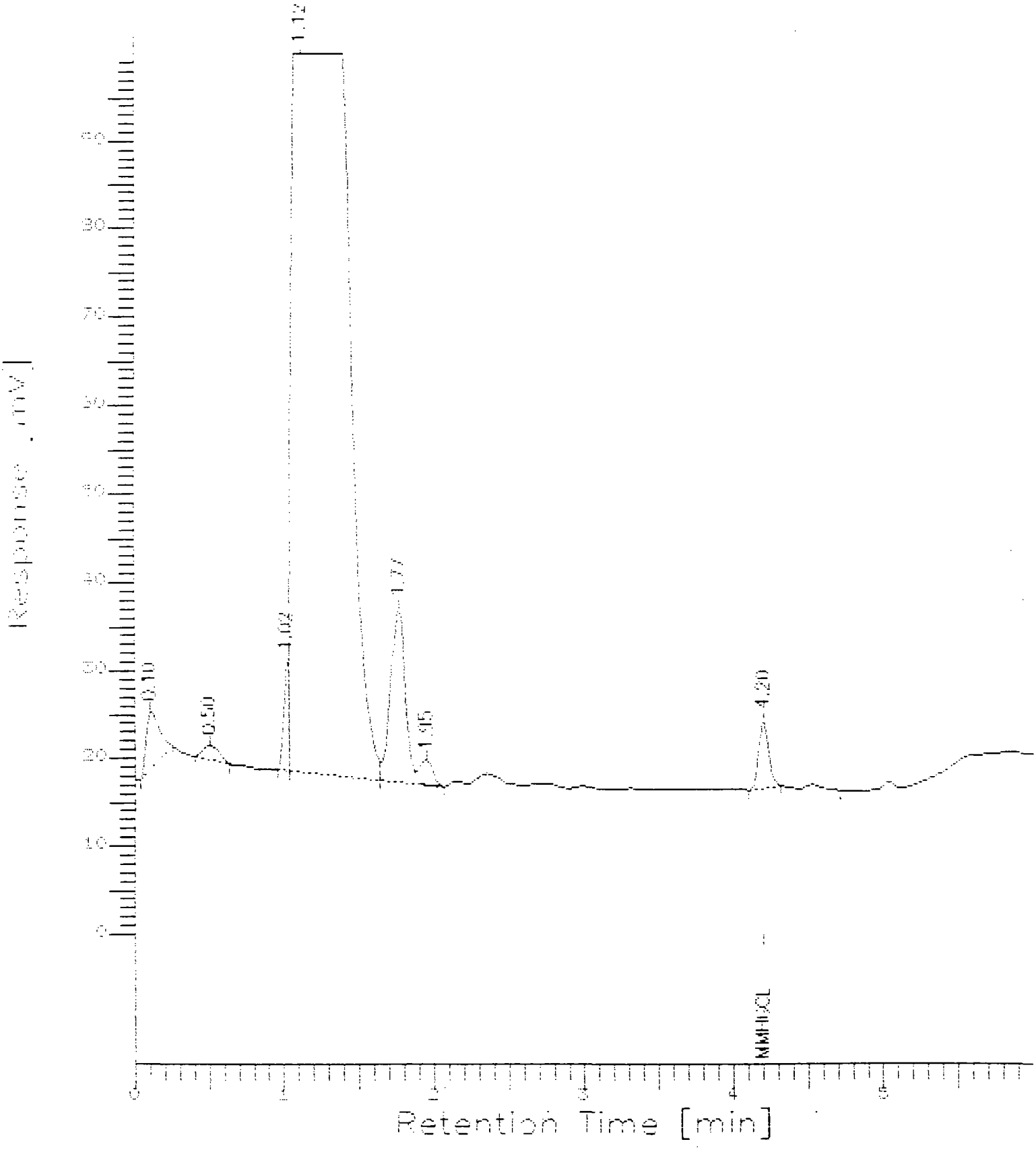
Inj. Volume : 1 ul Area Reject : 0.00
Sample Amount : 1.0000

DEFAULT REPORT

Peak #	Component Name	Ret Time (min)	Area (uV-sec)	Height (uV)	SL	RAW AMOUNT PPB	CORRECTED AMOUNT PPB	Area/ Amount
1		0.100	42109	6665	BB	4.08	4.08	10318.07
2		0.500	11569	1657	BB	1.12	1.12	10318.07
3		1.017	78836	12529	BV	7.64	7.64	10318.07
4		1.117	9272100	972219	VV	898.63	898.63	10318.07
5		1.767	135931	19385	VE	13.17	13.17	10318.07
6		1.950	14432	2943	EB	1.40	1.40	10318.07
7	MMHoCl	4.200	36579	7873	BB	3.55	3.55	10318.07
			9547255	1023791		925.39	925.39	

FileName : C: 2720\DATA\SAHG053.raw
Start Time : 0.00 min End Time : 5.00 min
Scale Factor: 1 Plot Offset: 0 mV

Date : 12/7/90 13:17 Page 1 of 1
Low Point : 0.00 mV High Point : 100.00 mV
Plot Scale: 100 mV



Sample Name : 664:06-MMHg STD III @ 15 ppb Time : 12/7/90 13:26
Vial Number: 54 Study :
Operator : MWL

Interface # : 4 Channel : A A/D mV Range : 1000
AutoSampler : Hewlett-Packard 7673A
Rack/Vial : 0/2

Data Acquisition Time: 12/7/90 13:08
Delay Time : 0.00 min.
End Time : 6.00 min.
Sampling Rate : 1.0000 pts/sec

Raw Data File : C:\2700\DATA\EAHG054.raw
Result File : C:\2700\DATA\EAHG054.rst
Instrument File: C:\2700\DATA\EAHG.ins
Process File : C:\2700\DATA\EAHG.prc
Sample File : C:\2700\DATA\EAHG.smp
Sequence File : C:\2700\DATA\EAHG.seq

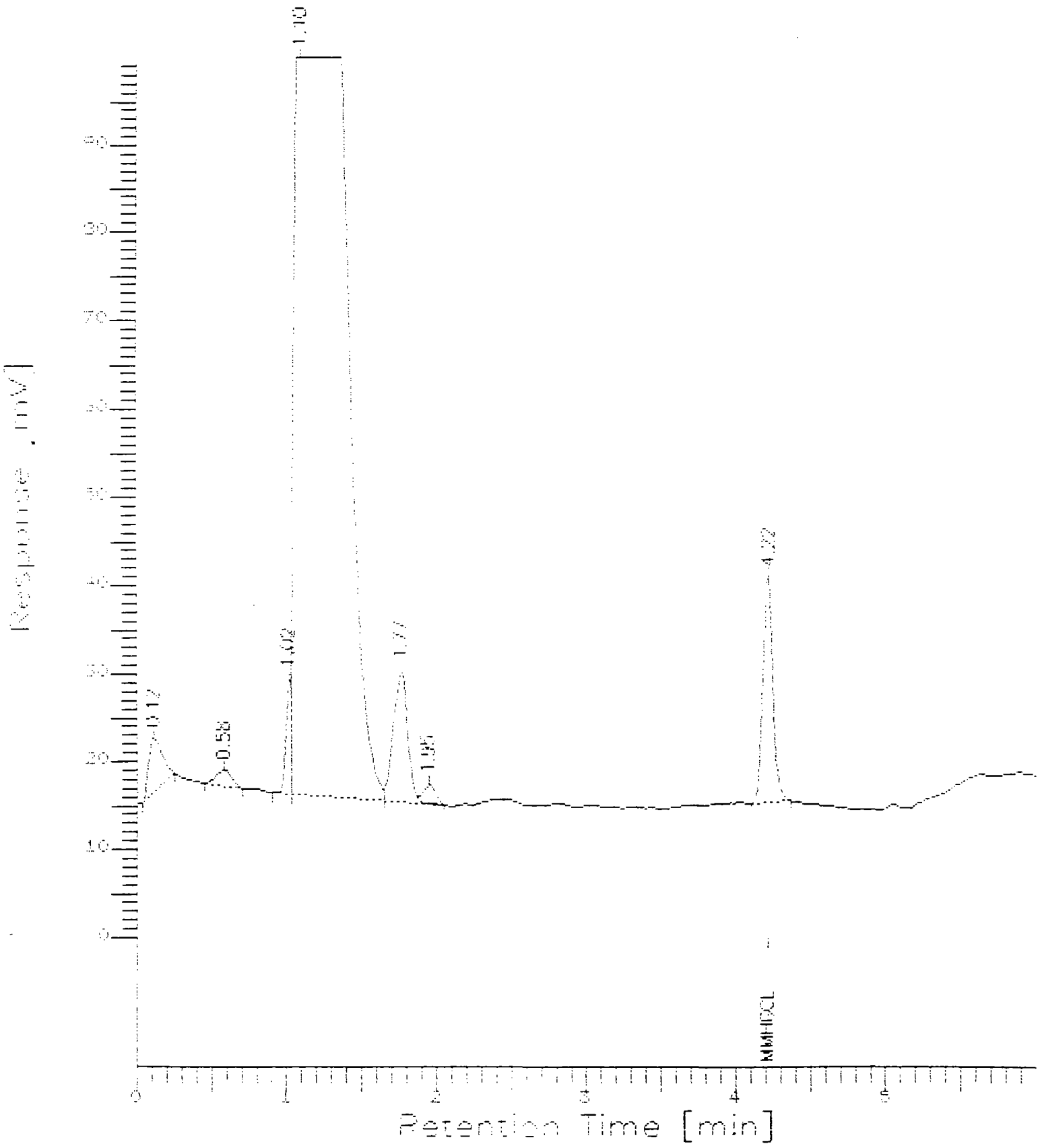
Inj. Volume : 1 ul Area Reject : 0.00
Sample Amount : 1.0000

DEFAULT REPORT

Peak #	Component Name	Ret Time (min)	Area (uV-sec)	Height (uV)	BL	RAW AMOUNT PPB	CORRECTED AMOUNT PPB	Area/ Amount
1		0.117	40535	6117	BB	3.93	3.93	10318.07
2		0.567	12380	1772	BB	1.20	1.20	10318.07
3		1.017	37156	13290	BV	3.86	3.86	10318.07
4		1.100	3252544	974360	VV	302.72	302.72	10318.07
5		1.767	98204	15026	VE	9.52	9.52	10318.07
6		1.950	10978	2228	EB	1.05	1.05	10318.07
7	MMHg(I)	4.217	117689	25598	BB	11.43	11.43	10318.07
			9501585	1038331		333.64	333.64	

Chromatogram

FileName : D:\2700\DATA\EAH0054.raw
Start Time : 0.00 min End Time : 5.00 min
Scale Factor: 0 Plot Offset: 0 mV
Data : 12/7/99 13:26 Page 1 of 1
Low Point : 0.00 mV High Point : 100.00 mV
Plot Scale: 100 mV



3 4 060

Sample Name : E64:06-mmHg STD III 15pp6 Time : 12/7/90 13:35
Vial Number: 55 Study :
Operator : WWL

Interface # : 4 Channel : A A/D mV Range : 1000
AutoSampler : Hewlett-Packard 7673A
Rack/Vial : 0/2

Data Acquisition Time: 12/7/90 13:17
Delay Time : 0.00 min.
End Time : 6.00 min.
Sampling Rate : 1.0000 pts/sec

Raw Data File : C:\2700\DATA\EAH6055.raw
Result File : C:\2700\DATA\EAH6055.rst
Instrument File: C:\2700\DATA\EAH6.ins
Process File : C:\2700\DATA\EAH6.src
Sample File : C:\2700\DATA\EAH6.smp
Sequence File : C:\2700\DATA\EAH6.seq

Inj. Volume : 1 ul Area Reject : 0.00
Sample Amount : 1.0000

DEFAULT REPORT

Peak #	Component Name	Ret Time (min)	Area (uV-sec)	Height (uV)	BL	RAW AMOUNT PPB	CORRECTED AMOUNT PPB	Area/ Amount
1		0.817	35231	5591	BB	3.41	3.41	10318.07
2		0.567	14440	2090	BB	1.40	1.40	10318.07
3		1.117	3300932	976215	BV	304.50	304.50	10318.07
4		1.767	102336	14222	VB	9.92	9.92	10318.07
5		1.967	13242	3424	BB	1.28	1.28	10318.07
6	mmHg(1)	4.017	109934	24203	BB	10.66	10.66	10318.07
			8576114	1024745		831.17	831.17	

[redacted]

FileName : I:\2700\DATA\E0HG055.raw
Start Time : 0.00 min End Time : 5.00 min
Scale Factor: 0 Plot Offset: 0 mV

Date : 12/7/90 12:35
Low Point : 0.00 mV High Point : 100.00 mV
Plot Scale: 100 mV

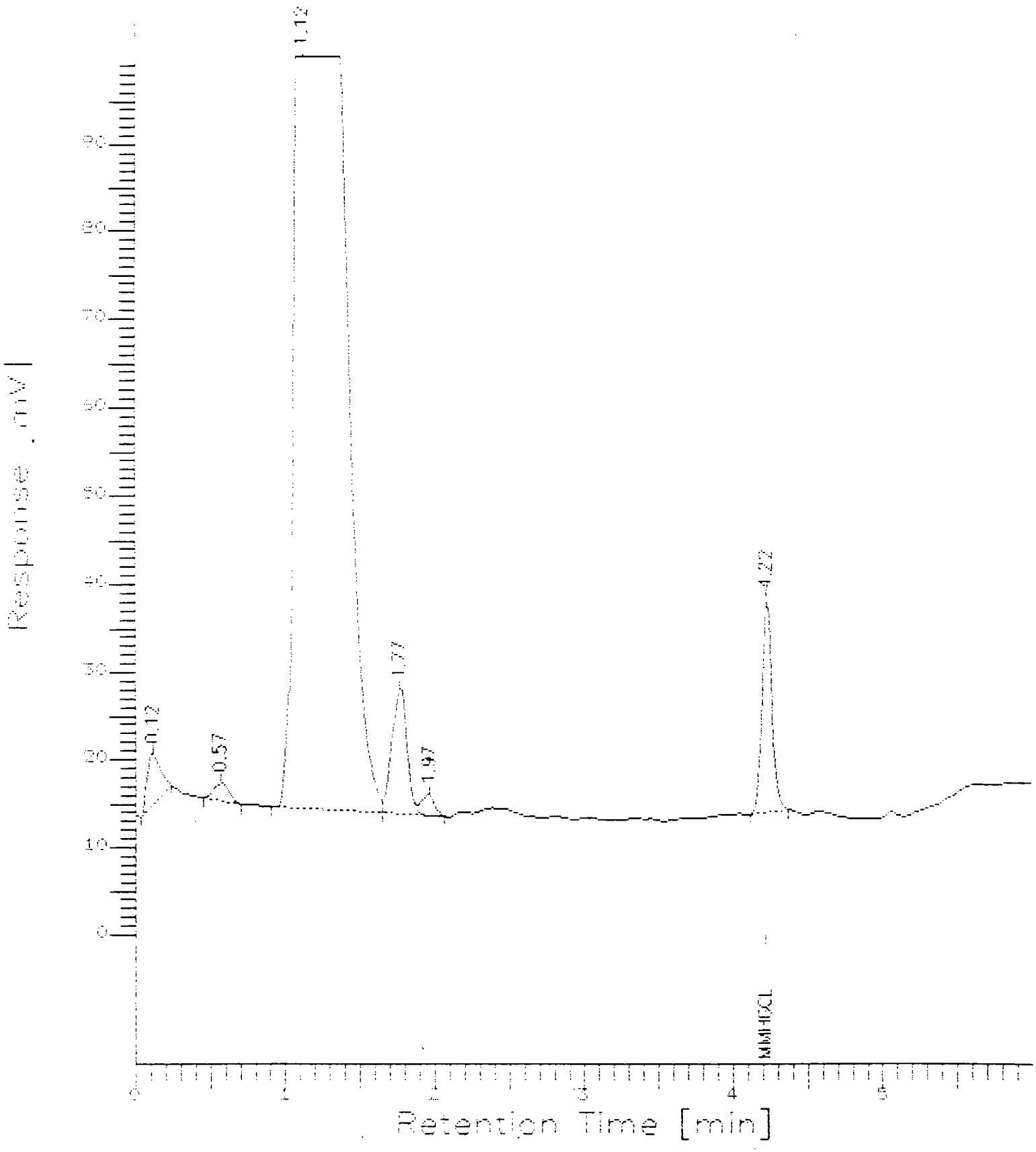


Table Name : 56+Use-MMHG STD 1V 50ppb Time : 12/7/90 13:43
Table Number: 56 Study :
Operator : WWL

Interface # : 4 Channel : A A/D mV Range : 1000
AutoSampler : Hewlett-Packard 7673A
Rack/Vial : 0/2

Data Acquisition Time: 12/7/90 13:25
Delay Time : 0.00 min.
End Time : 6.00 min.
Sampling Rate : 1.0000 pts/sec

Raw Data File : C:\2700\DATA\EAHG056.raw
Result File : C:\2700\DATA\EAHG056.rst
Instrument File: C:\2700\DATA\EAHG.ins
Process File : C:\2700\DATA\EAHG.prc
Sample File : C:\2700\DATA\EAHG.smp
Sequence File : C:\2700\DATA\EAHG.seq

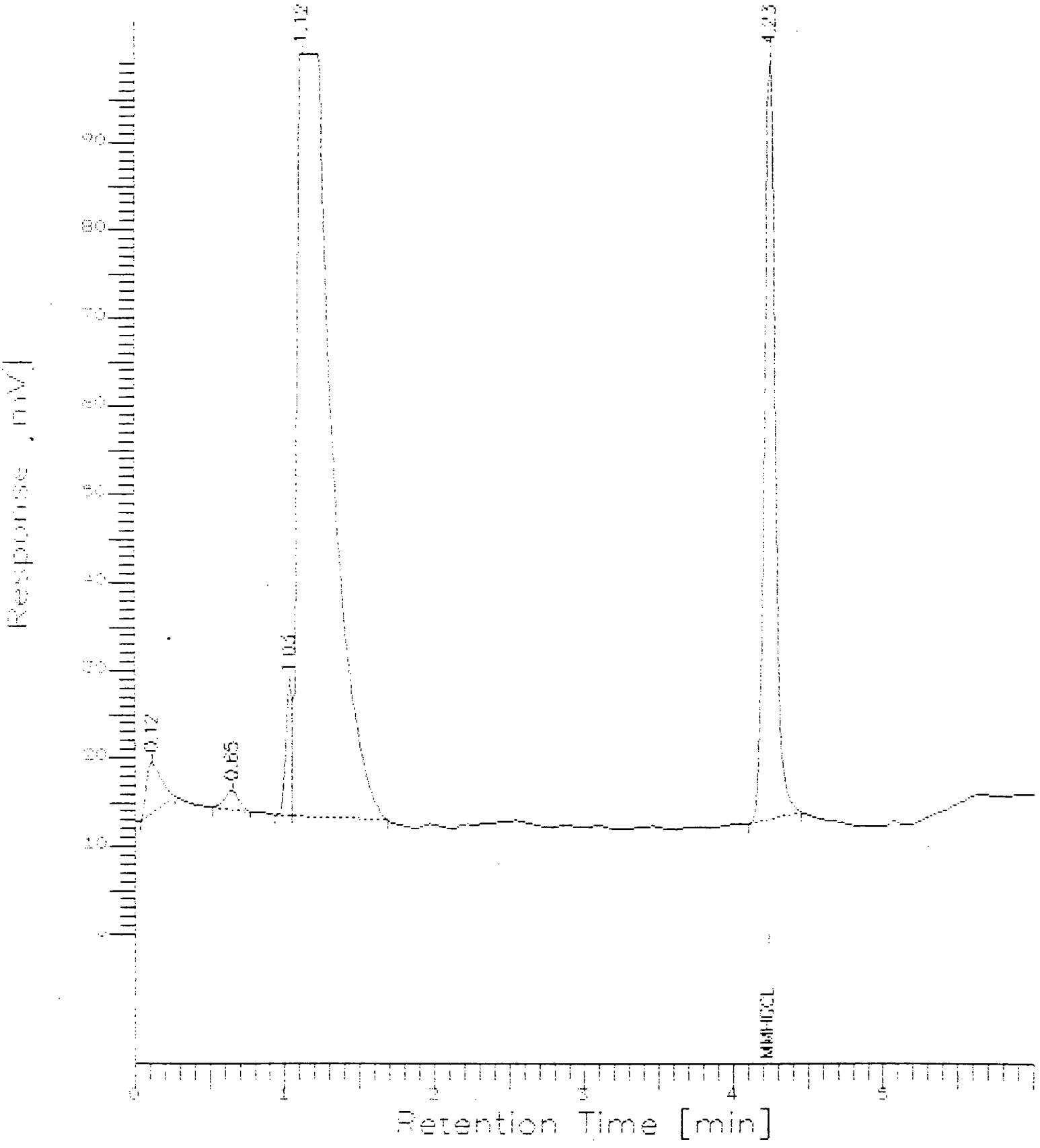
Inj. Volume : 1 ul Area Reject : 0.00
Sample Amount : 1.0000

DEFAULT REPORT

Peak #	Component Name	Ret Time (min)	Area (uV-sec)	Height (uV)	BL	RAW AMOUNT PPB	CORRECTED AMOUNT PPB	Area/ Amount
1		0.117	39925	5583	BB	3.87	3.87	10318.07
2		0.650	14366	2124	BB	1.39	1.39	10318.07
3		1.033	50311	14977	BV	4.88	4.88	10318.07
4		1.117	3309842	763852	VB	320.78	320.78	10318.07
5	MMHg01	4.233	444047	94484	BB	43.04	43.04	10318.07
			7858490	921019		373.36	373.36	

FileName : C:\2700\DATA\EAH0056.raw
Start Time : 0.00 min End Time : 6.00 min
Scale Factor: 0 Plot Offset: 0 mV

Date : 12/7/90 10:44
Low Point : 0.00 mV High Point : 100.00 mV



3 4 030

Sample Name : ea4:06-MMHG STD IV 5ppb Time : 12/7/90 13:52
Sample Number: 97 Stuck :
Operator : WWL

Interface # : 1 Channel : A A/D mV Range : 1000
AutoSampler : Hewlett-Packard 7673A
Rack/Vial : 0/3

Data Acquisition Time: 12/7/90 13:34
Delay Time : 0.00 min.
End Time : 6.00 min.
Sampling Rate : 1.0000 pts/sec

Raw Data File : C:\2700\DATA\EAHG057.raw
Result File : C:\2700\DATA\EAHG057.rst
Instrument File: C:\2700\DATA\EAHG.ins
Process File : C:\2700\DATA\EAHG.prc
Sample File : C:\2700\DATA\EAHG.smp
Sequence File : C:\2700\DATA\EAHG.seq

Inj. Volume : 1 ul Area Reject : 0.00
Sample Amount : 1.0000

DEFAULT REPORT

Peak #	Component Name	Ret Time (min)	Area (uV-sec)	Height (uV)	BL	RAW AMOUNT PPB	CORRECTED AMOUNT PPB	Area/ Amount
1		0.117	34863	3245	BB	3.38	3.38	10318.07
2		0.633	14560	2171	BB	1.41	1.41	10318.07
3		1.033	48808	14775	BV	4.73	4.73	10318.07
4		1.117	3308482	728920	VB	320.65	320.65	10318.07
5	MMHgCl	4.233	425531	57685	BB	41.24	41.24	10318.07
			3832243	878696		371.41	371.41	

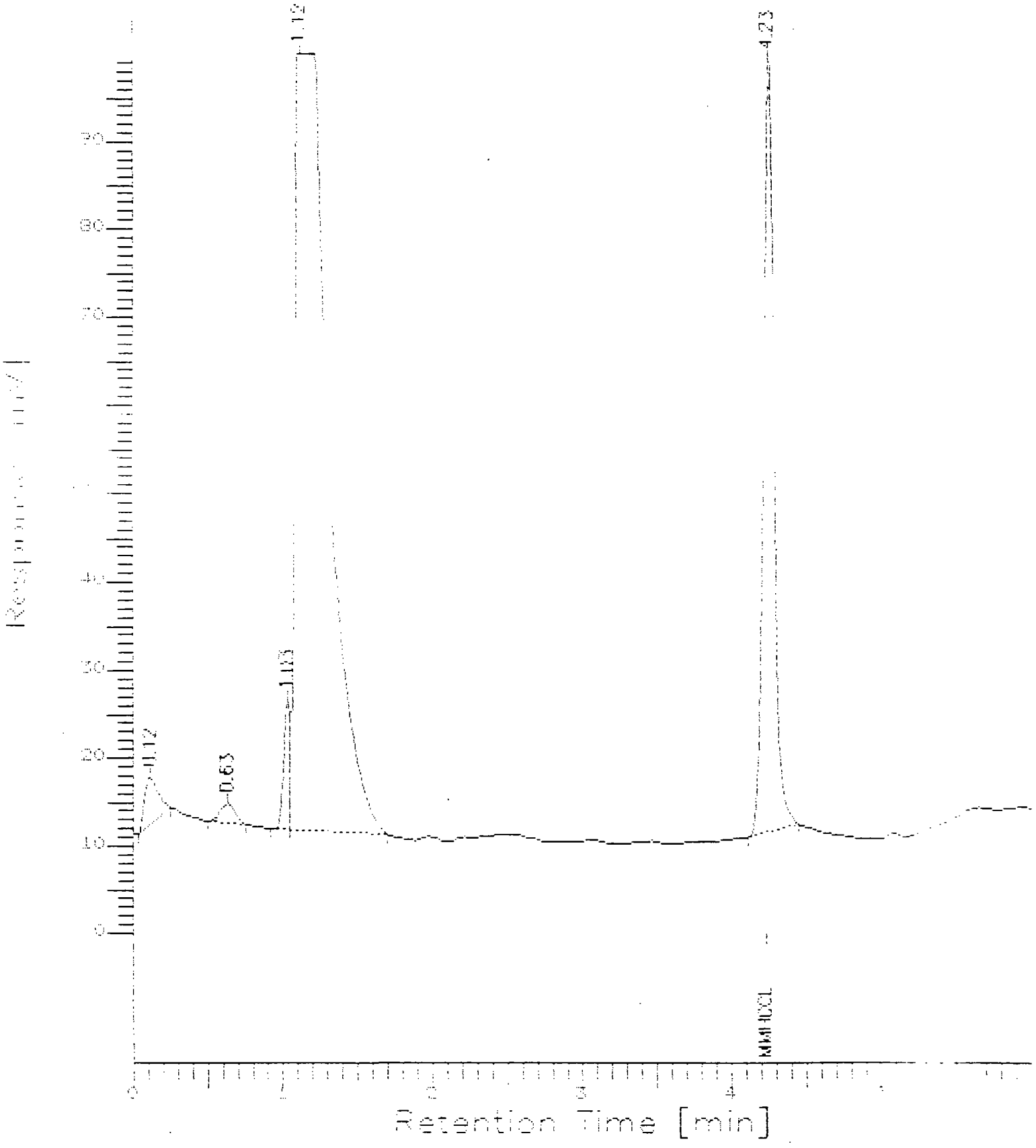
3 4 03:1)

Chromatogram

FileName : C:\2700\DATA\EAH6057.raw
Start Time : 0.00 min End Time : 5.00 min
Scale Factor: 0 Plot Offset: 0 mV

Date : 12/7/90 13:52
Low Point : 0.00 mV
Plot Scale: 100 mV

High Point : 100.00 mV



==

Sample Name : c64;08-MMHG STD V 151/p26 Time : 12/7/90 14:00
Sample Number: 08 Study :
Operator : JWL

Interface # : 4 Channel : A A/D mV Range : 1000
AutoSampler : Hewlett-Packard 7673A
Rack/Vial : 0/4

Data Acquisition Time: 12/7/90 13:42
Delay Time : 0.00 min.
End Time : 6.00 min.
Sampling Rate : 1.0000 pts/sec

Raw Data File : C:\2700\DATA\EAHG058.raw
Result File : C:\2700\DATA\EAHG058.rst
Instrument File: C:\2700\DATA\EAHG.ins
Process File : C:\2700\DATA\EAHG.prc
Sample File : C:\2700\DATA\EAHG.smp
Sequence File : C:\2700\DATA\EAHG.seq

Inj Volume : 1 ul Area Reject : 0.00
Sample Amount : 1.0000

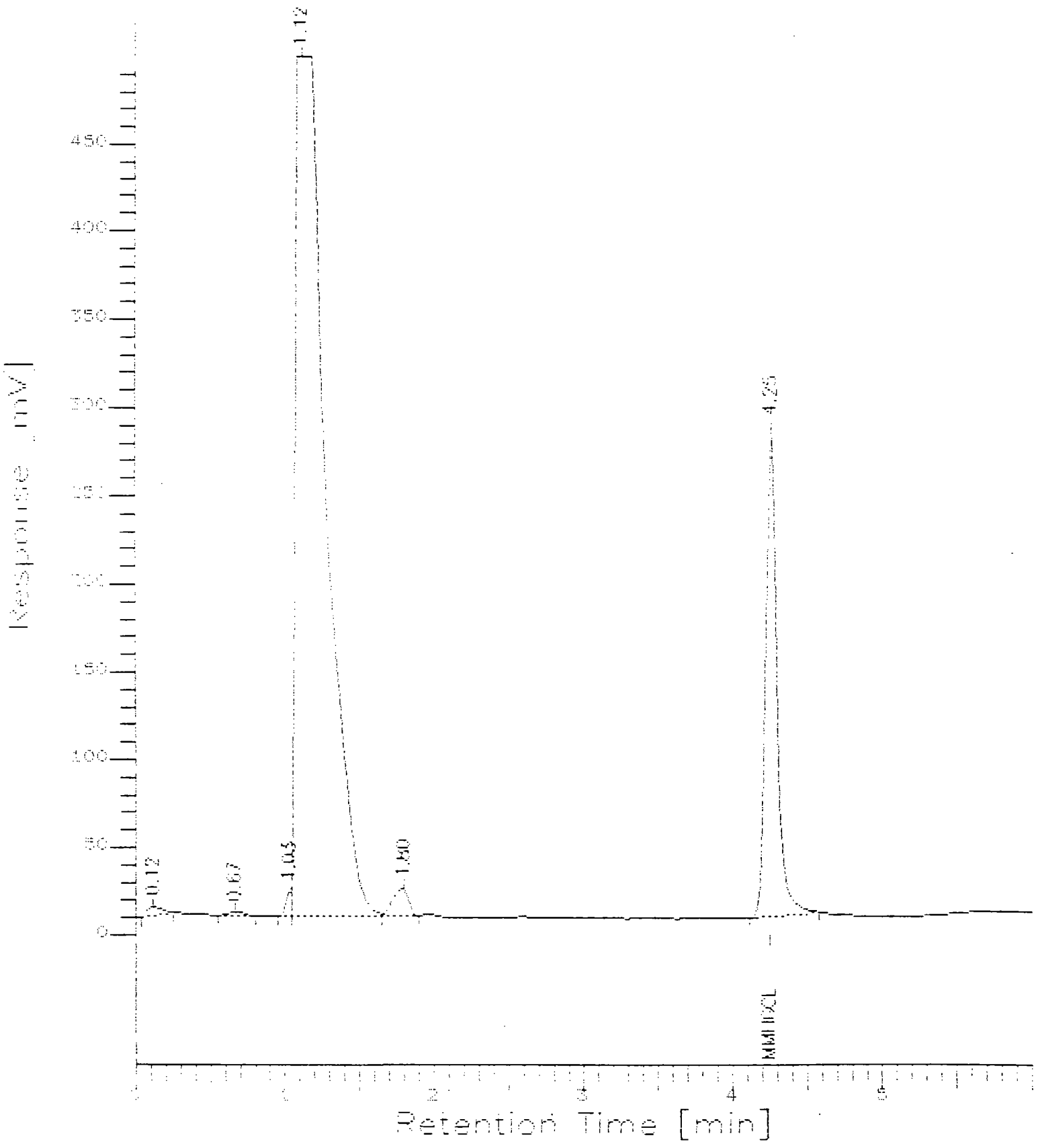
==

DEFAULT REPORT

Peak #	Component Name	Ret Time [min]	Area [uV-sec]	Height [uV]	BL	RAW AMOUNT PPB	CORRECTED AMOUNT PPB	Area/ Amount
1		0.117	34773	5130	BB	3.37	3.37	10318.07
2		0.667	15281	2243	BB	1.48	1.48	10318.07
3		1.033	48206	14316	BV	4.67	4.67	10318.07
4		1.117	9355483	979571	VV	906.71	906.71	10318.07
5		1.300	109498	15547	VB	10.61	10.61	10318.07
6	MMHgCl	4.250	1442291	279112	BB	139.78	139.78	10318.07
			11005531	1295919		1066.63	1066.63	

Chromatogram

FileName : C:\E700\DATA\EAM0058.raw
Date : 12/7/90 14:00
Page 1 of 1
Start Time : 0.00 min
End Time : 6.00 min
Low Point : 0.00 mV
High Point : 500.00 mV
Scale Factor: 0
Plot Offset: 0 mV
Plot Scale: 500 mV



3 4 0313

Sample Name : 30415-MMHG STD : 157ppb   Time : 12/7/90 14:09
Sample Number : 39   Cycle :
Operator : MHL

Interface # : 4   Channel : 4   A/D mV Range : 1000
AutoSampler : Hewlett-Packard 7673A
Rack/Vial : [illegible]

Data Acquisition Time : 12/7/90 13:51
Delay Time : 0.00 min.
End Time : 5.00 min.
Sampling Rate : 1.5000 pts/sec

Raw Data File : C:\2700\DATA\EAHG039.raw
Result File : C:\2700\DATA\EAHG039.rst
Instrument File : C:\2700\DATA\EAHG.ins
Process File : C:\2700\DATA\EAHG.prc
[illegible] File : C:\2700\DATA\EAHG.smp
Sequence File : C:\2700\DATA\EAHG.seq

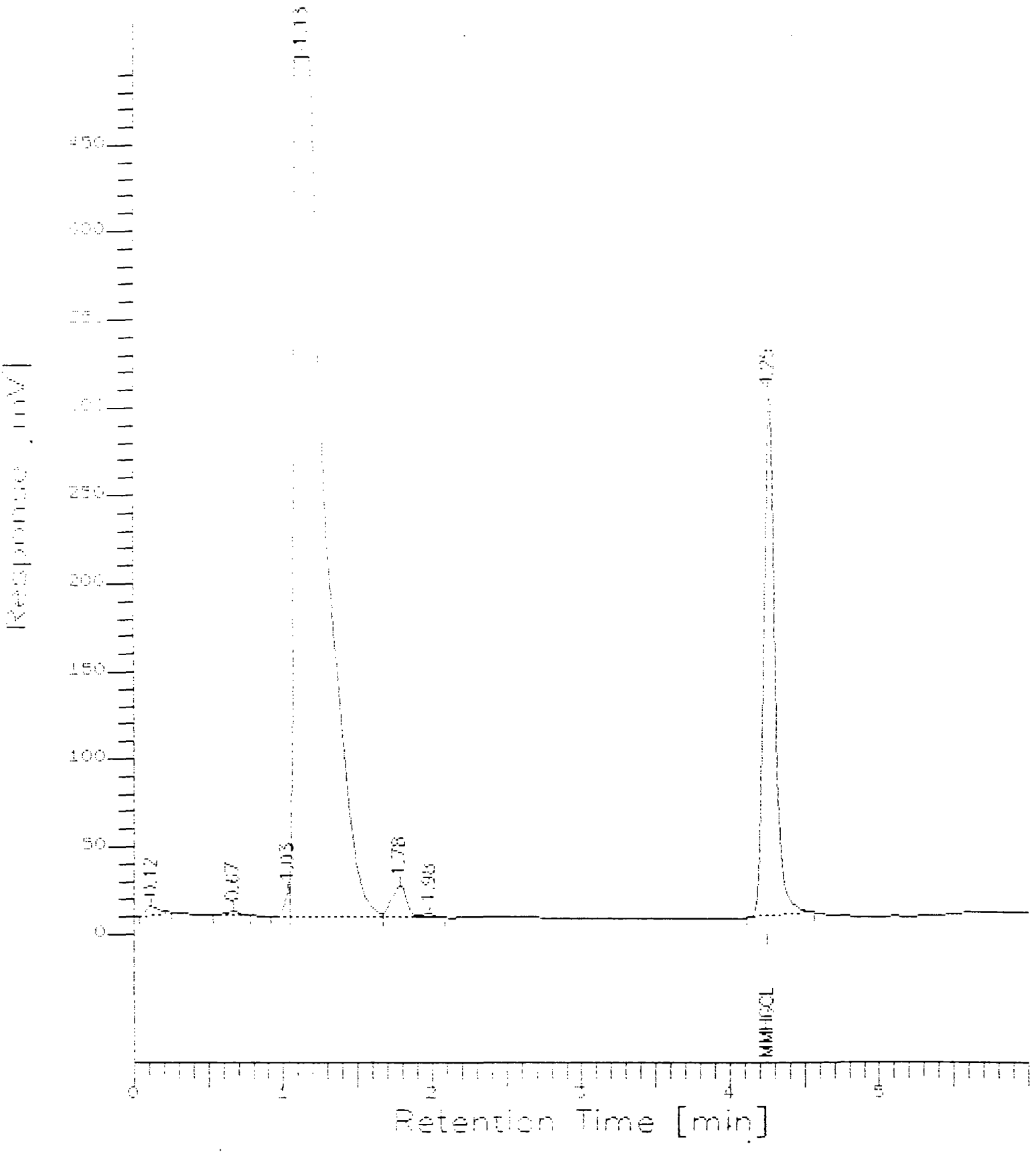
Inj Volume : 1 ul   Area Reject : 0.00
Sample Amount : 1.0000

DEFAULT REPORT

Peak #	Component Name	Ret Time [min]	Area [uV-sec]	Height [uV]	BL RAW AMOUNT PPB	CORRECTED AMOUNT PPB	Area/ Amount
1		[illegible]	33794	5059.35	3.28	3.28	10318.07
2		0.657	14854	2155.38	1.44	1.44	10318.07
3		1.0[illegible]	44271	17955.87	4.29	4.29	10318.07
4		1.177	9515547	950339.99	922.32	922.32	10318.07
5		1.757	126290	17733.95	12.24	12.24	10318.07
6		1.987	13293	2478.55	1.29	1.29	10318.07
7	**HgCl	4.251	1517162	135249.58	147.04	147.04	10318.07
			11266220	1317049	1091.89	1091.89	

Chromatogram

FileName : C:\CTIO\DATA\2HH0059.raw
Start Time : 0.00 min
Scale Factor: 1
End Time : 6.00 min
Plot Offset: 0 mV
Date : 12/7/99 14:09
Low Point : 0.00 mV
Plot Scale: 500 mV
Page 1 of 1
High Point : 510.00 mV



3 4 031b

Sample Name : 864:06-MMHG STD #1 30/1/96 Time : 12/7/90 14:17
Sample Number: 60 Study :
Operator : WWL

Interface # : 4 Channel : A A/D mV Range : 1000
AutoSampler : Hewlett-Packard 7673A
Rack/Vial : 0/5

Data Acquisition Time: 12/7/90 13:59
Delay Time : 0.00 min.
End Time : 6.00 min.
Sampling Rate : 1.0000 pts/sec

Raw Data File : C:\2700\DATA\EAHG060.raw
Result File : C:\2700\DATA\EAHG060.rst
Instrument File: C:\2700\DATA\EAHG.ins
Process File : C:\2700\DATA\EAHG.prc
Sample File : C:\2700\DATA\EAHG.smp
Sequence File : C:\2700\DATA\EAHG.seq

Inj. Volume : 1 ul Area Reject : 0.00
Sample Amount : 1.0000

DEFAULT REPORT

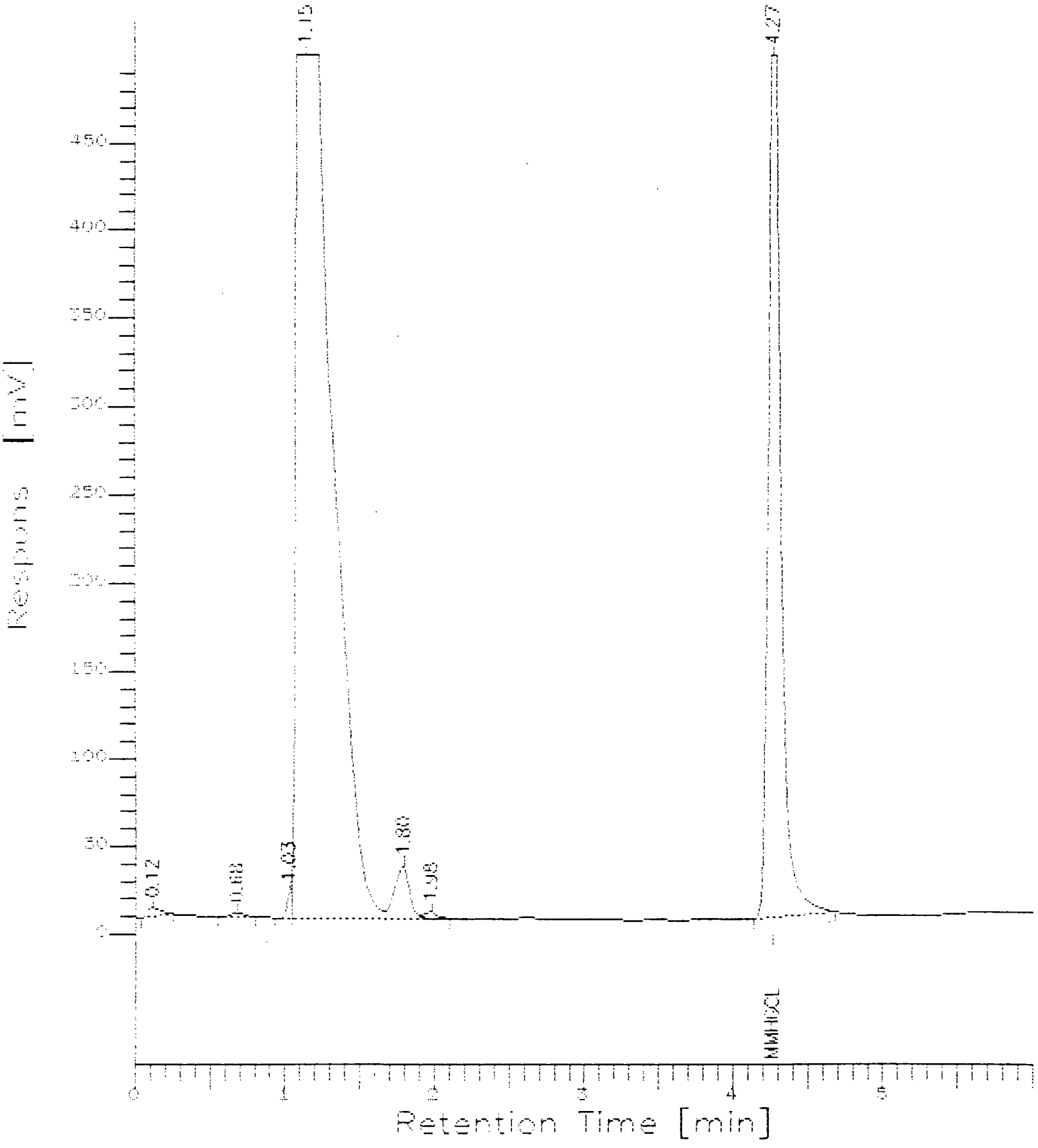
Peak #	Component Name	Ret Time [min]	Area [uV-sec]	Height [uV]	BC	RAW AMOUNT PPB	CORRECTED AMOUNT PPB	Area/ Amount
1		0.117	33324	4939	BB	3.23	3.23	10318.07
2		0.680	15917	2226	BB	1.54	1.54	10318.07
3		1.033	44664	15112	BV	4.33	4.33	10318.07
4		1.150	11675696	991102	VV	1131.59	1131.59	10318.07
5		1.300	307032	70265	VE	29.07	29.07	10318.07
6		1.983	19994	3663	EB	1.93	1.93	10318.07
7	*MMPG1	4.267	7108172	662328	BB	701.24	701.24	10319.07
			15104698	1639734		1463.91	1463.91	

Chromatogram 3 4 0316

FileName : C:\2700\DATA\EAHG060.raw
Start Time : 0.00 min End Time : 6.00 min
Scale Factor: 0 Plot Offset: 0 mV

Date : 12/7/90 14:18
Low Point : 0.00 mV High Point : 500.00 mV

Page 1 of 1



Sample Name : 6e4:06-MMHG STD VI 30/526 Time : 12/7/90 14:26
Sample Number: 61 Study :
Operator : WWL

Interface # : 4 Channel : A A/D mV Range : 1000
AutoSampler : Hewlett-Packard 7673A
Rack/Vial : 0/5

Data Acquisition Time: 12/7/90 14:08
Delay Time : 0.00 min.
End Time : 6.00 min.
Sampling Rate : 1.0000 pts/sec

Raw Data File : C:\2700\DATA\EAHG061.raw
Result File : C:\2700\DATA\EAHG061.rst
Instrument File: C:\2700\DATA\EAHG.ins
Process File : C:\2700\DATA\EAHG.prc
Sample File : C:\2700\DATA\EAHG.smp
Sequence File : C:\2700\DATA\EAHG.seq

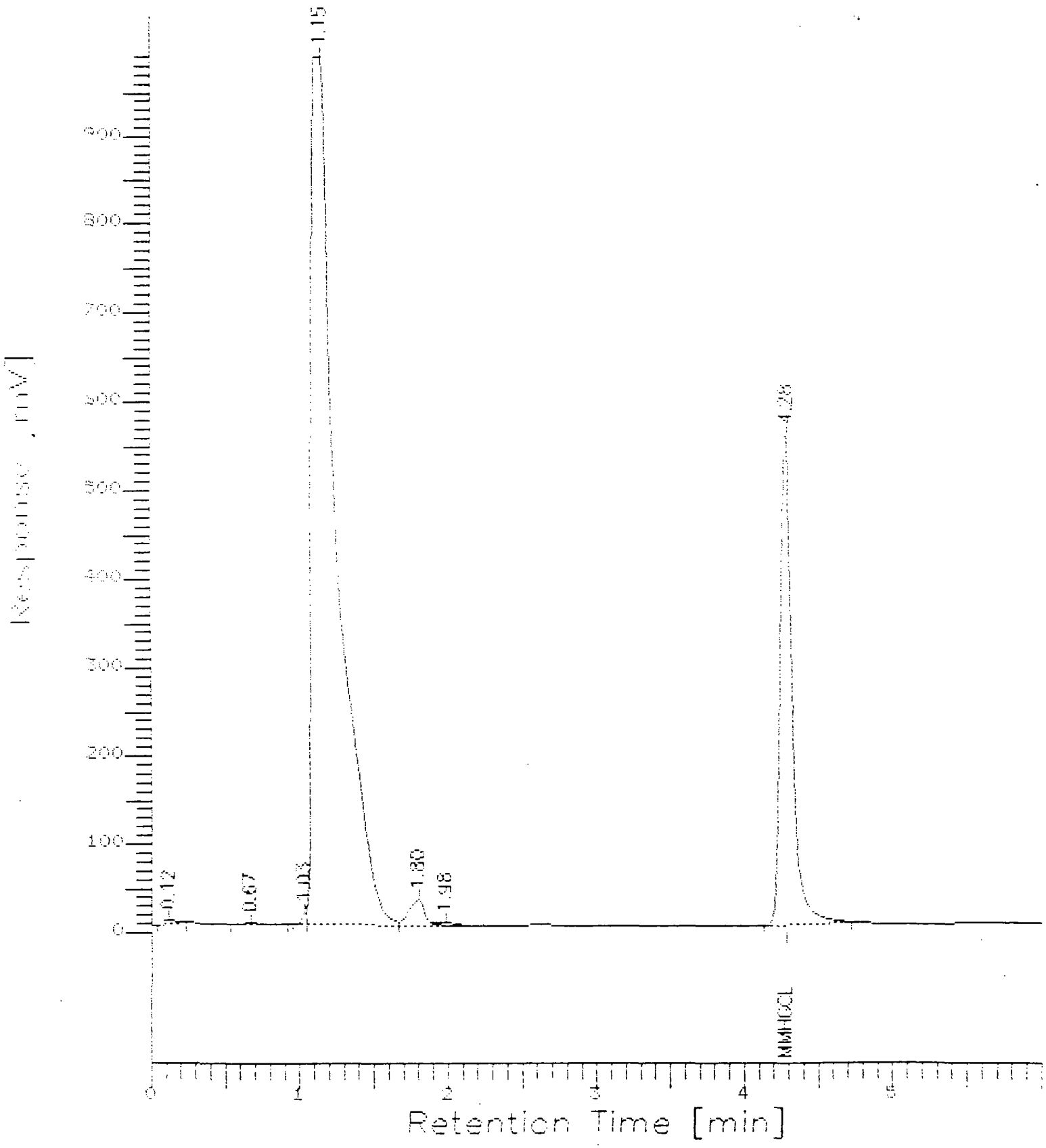
Inj. Volume : 1 ul Area Reject : 0.00
Sample Amount : 1.0000

DEFAULT REPORT

Peak #	Component Name	Ret Time (min)	Area (uV-sec)	Height (uV)	BL	RAW AMOUNT PPB	CORRECTED AMOUNT PPB	Area/ Amount
1		0.117	30559	4824	BB	2.96	2.96	10318.07
2		0.667	16086	2274	BB	1.56	1.56	10318.07
3		1.033	42501	14691	BV	4.12	4.12	10318.07
4		1.150	11747272	981472	VV	1138.52	1138.52	10318.07
5		1.800	209891	29503	VE	20.34	20.34	10318.07
6		1.983	20553	3666	EB	1.99	1.99	10318.07
7	MMHg01	4.283	3042735	553696	BB	294.89	294.89	10318.07
			15109597	1590127		1464.38	1464.38	

Chromatogram

FileName : I:\2700\DATA\EAH6061.raw
Date : 12/7/99 14:26
Page 1 of 1
Start Time : 0.00 min
End Time : 6.00 min
Low Point : 0.00 mV
High Point : 1000.00 mV
Scale Factor: 0
Plot Offset: 0 mV
Plot Scale: 1000 mV



APPENDIX B
CONTINUING CALIBRATION SUMMARY

APPENDIX B
Page 1 of 1
Gregory E. Johnson
'ethyl Mercury Method Validation
Date: January 24, 1991
TDL Project No.: 482826

IT ANALYTICAL SERVICES
304 DIRECTORS DRIVE
KNOXVILLE, TENNESSEE

MONOMETHYL MERCURY CHLORIDE

CONTINUING CALIBRATION SUMMARY

RUN DATE	LEVEL	CONCENTRATION (ppb)	% RECOVERY[1]
12/07/90	2	15	107
12/10/90	1	5	86
12/10/90	2	15	112
12/11/90	1	5	100

[1]Must be 75-125%.

Sample Name : 684:06-MMHG STDIII @15ppb Time : 12/7/90 14:29
Sample Number: 79 Level 2 Study :
Operator : WWL 3 4 0321

nterface # : 4 Channel : A A/D mV Range : 1000
AutoSampler : Hewlett-Packard 7673A
Rack/Vial : 3/10

Data Acquisition Time: 12/7/90 20:22
Delay Time : 0.00 min.
End Time : 6.00 min.
Sampling Rate : 1.0000 pts/sec

Raw Data File : C:\2700\DATA\EAHG079.raw
Result File : C:\2700\DATA\EAHG079.rst
Instrument File: C:\2700\DATA\EAHG.ins
Process File : C:\2700\DATA\EAHG.prc
Sample File : C:\2700\DATA\EAHG1P.smp
Sequence File : C:\2700\DATA\EAHG.seq

Inj. Volume : 1 ul Area Reject : 0.00
Sample Amount : 1.0000

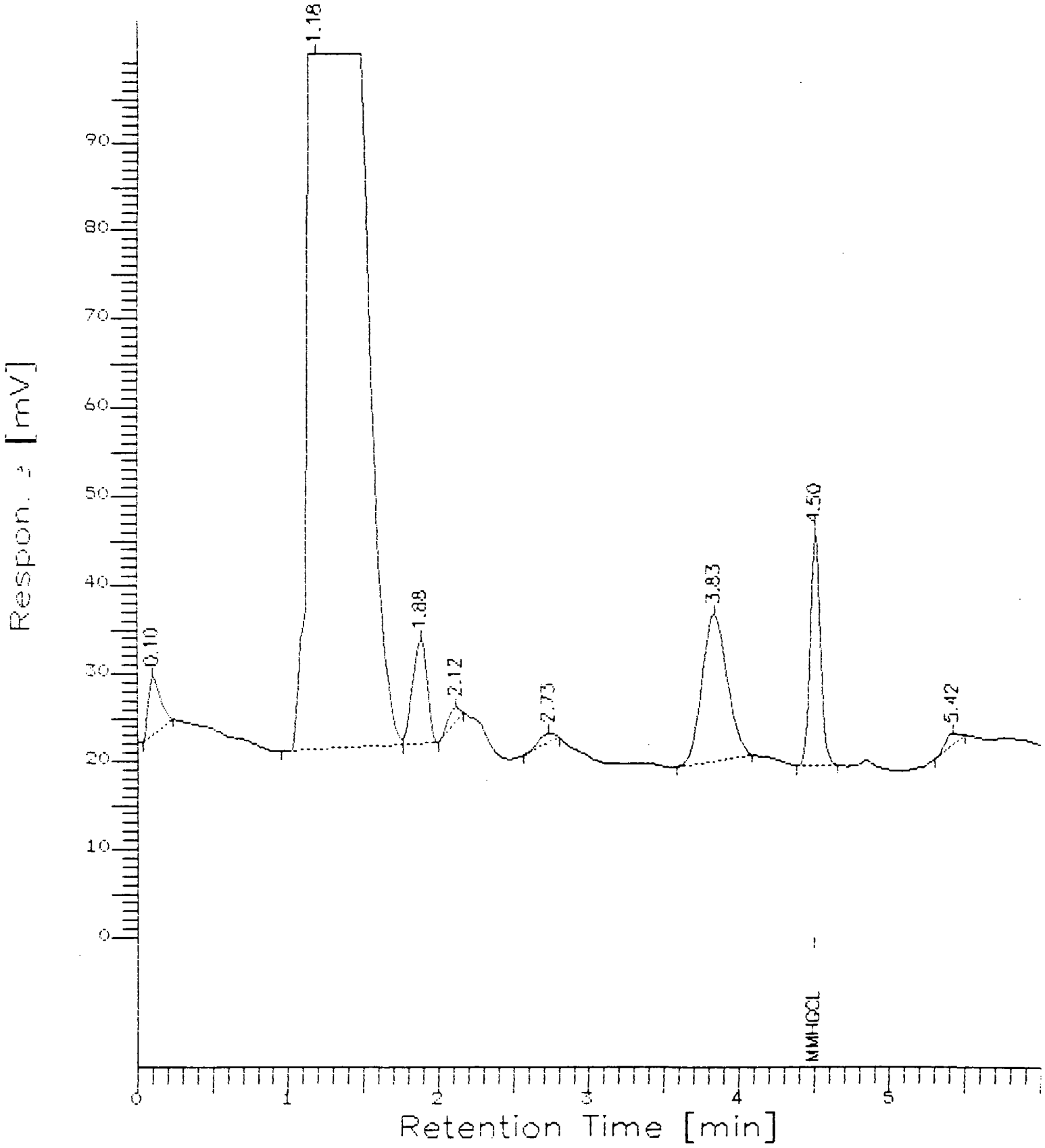
DEFAULT REPORT

Peak	Component Name	Ret Time [min]	Area [uV-sec]	Height [uV]	BL	RAW AMOUNT PPB	CORRECTED AMOUNT PPB	Area/ Amount	
1		0.150	76592	6530	BB	4.76	4.76	7695.71	
2		1.183	8567191	969058	BV	1113.24	1113.24	7695.71	
3		1.883	79039	11747	VB	10.27	10.27	7695.71	
4		2.117	11367	1644	BB	1.48	1.48	7695.71	
5		2.733	9891	1145	BB	1.29	1.29	7695.71	107%
6		3.833	192081	16573	BB	25.09	25.09	7695.71	
7	MMHgCl	4.500	123403	26259	BB	16.04	16.04	7695.71	
8		5.417	10168	1324	BB	1.32	1.32	7695.71	
			9030732	1034279		1173.48	1173.48		

Chromatogram 3 4 0322

FileName : C:\2700\DATA\EAHG079.raw
Date : 12/9/90 14:29

Start Time : 0.00 min
End Time : 6.00 min
Low Point : 0.00 mV
High Point : 100.00 mV
Scale Factor: 0
Plot Offset: 0 mV
Plot Scale: 100 mV



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Sample Name : 664:06-MMHG STD II @ 5ppb Time : 12/18/90 9:15
Sample Number: 95 Level 1 Study :
Operator : WWL

Interface # : 4 Channel : A A/D mV Range : 1000
AutoSampler : Hewlett-Packard 7673A
Rack/Vial : 0/1

Data Acquisition Time: 12/10/90 13:21
Delay Time : 0.00 min.
End Time : 6.00 min.
Sampling Rate : 1.0000 pts/sec

Raw Data File : EAHG095.RAW
Result File : E:\TEMP\~ERS3B0E.rst
Instrument File: C:\2700\DATA\EAHG.ins
Process File : C:\2700\DATA\EAHG.prc
Sample File : C:\2700\DATA\EAHG1P.smp
Sequence File : C:\2700\DATA\default.seq

Inj. Volume : 1 ul Area Reject : 0.00
Sample Amount : 1.0000

Noise Threshold: 300 Area Threshold : 100 Bunch Factor: 1
Dilution Factor: 1.00 Multiplier : 1.00
Divisor : 1.0000 Additive Constant: 0.00

Instrument Conditions:

Total Number of Peaks Detected: 5

==========

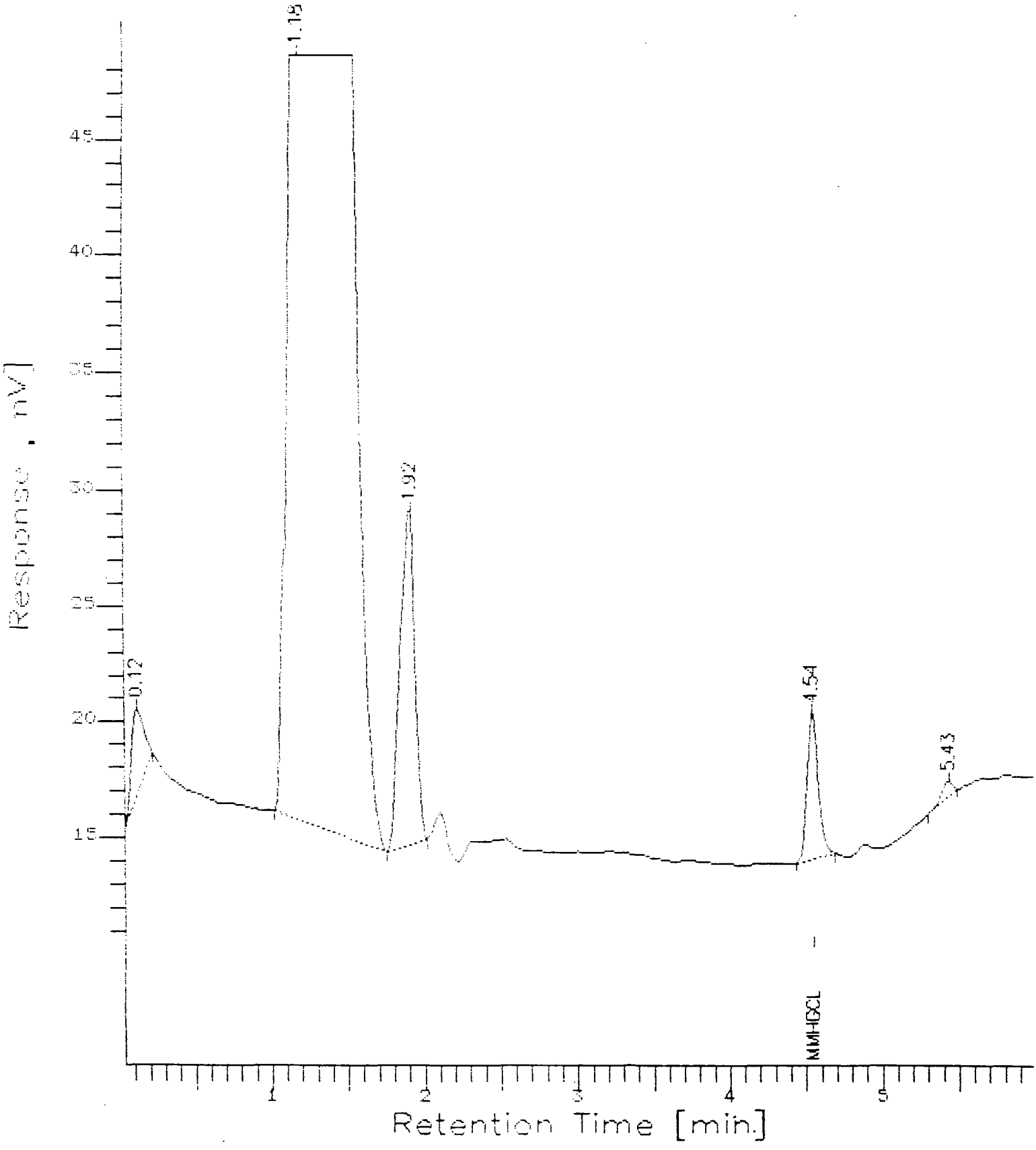
EA ENGINEERING METHYL MERCURY REPORT

Peak #	Component Name	Ret Time [min]	Area [uV-sec]	Height [uV]	BL	RAW AMOUNT PPB	CORRECTED AMOUNT PPB	Area/ Amount
1		0.117	22510	3719	BB	3.08	3.08	7307.70
2		1.183	9403310	974937	BB	1286.77	1286.77	7307.70
3		1.917	98812	14357	BB	13.52	13.52	7307.70
4	MMHgCl	4.541	31437	6470	MM	4.30	4.30	7307.70
5		5.433	3456	575	BB	0.47	0.47	7307.70
			9559525	1000058		1308.14	1308.14	

$\frac{4.3}{5} = 86\%$

Chromatogram 3 4 0324

FileName : EAH8095.RAW
Start Time : 0.03 min
Scale Factor: 3
End Time : 5.98 min
Plot Offset: 11 mV
Date : 12/18/90 9:16
Low Point : 10.77 mV
Plot Scale: 38 mV
Page 1 of 1
High Point : 48.59 mV



```
==========================================================================
 mple Name   : 664:06-MMHG STD III @ 15ppb Time          : 12/18/90  9:05
 ample Number: 112  Level 2                Study         :
Operator     : WWL

Interface #  :   4       Channel : A     A/D mV Range : 1000
AutoSampler  : Hewlett-Packard 7673A
Rack/Vial    : 0/7

Data Acquisition Time: 12/10/90  18:29
Delay Time   : 0.00   min.
End Time     : 5.00   min.
Sampling Rate  : 1.0000   pts/sec

Raw Data File  : EAHG112.RAW
Result File    : E:\TEMP\~ERS0226.rst
Instrument File: C:\2700\DATA\EAHG.ins
Process File   : C:\2700\DATA\EAHG.prc
Sample File    : C:\2700\DATA\EAHG1P.smp
Sequence File  : C:\2700\DATA\default.seq

Inj. Volume    : 1          ul    Area Reject      : 0.00
Sample Amount  : 1.0000

Noise Threshold: 300          Area Threshold   : 100      Bunch Factor: 1
Dilution Factor: 1.00         Multiplier       : 1.00
 visor         : 1.0000       Additive Constant: 0.00

Instrument Conditions:

Total Number of Peaks Detected: 7
==========================================================================
```

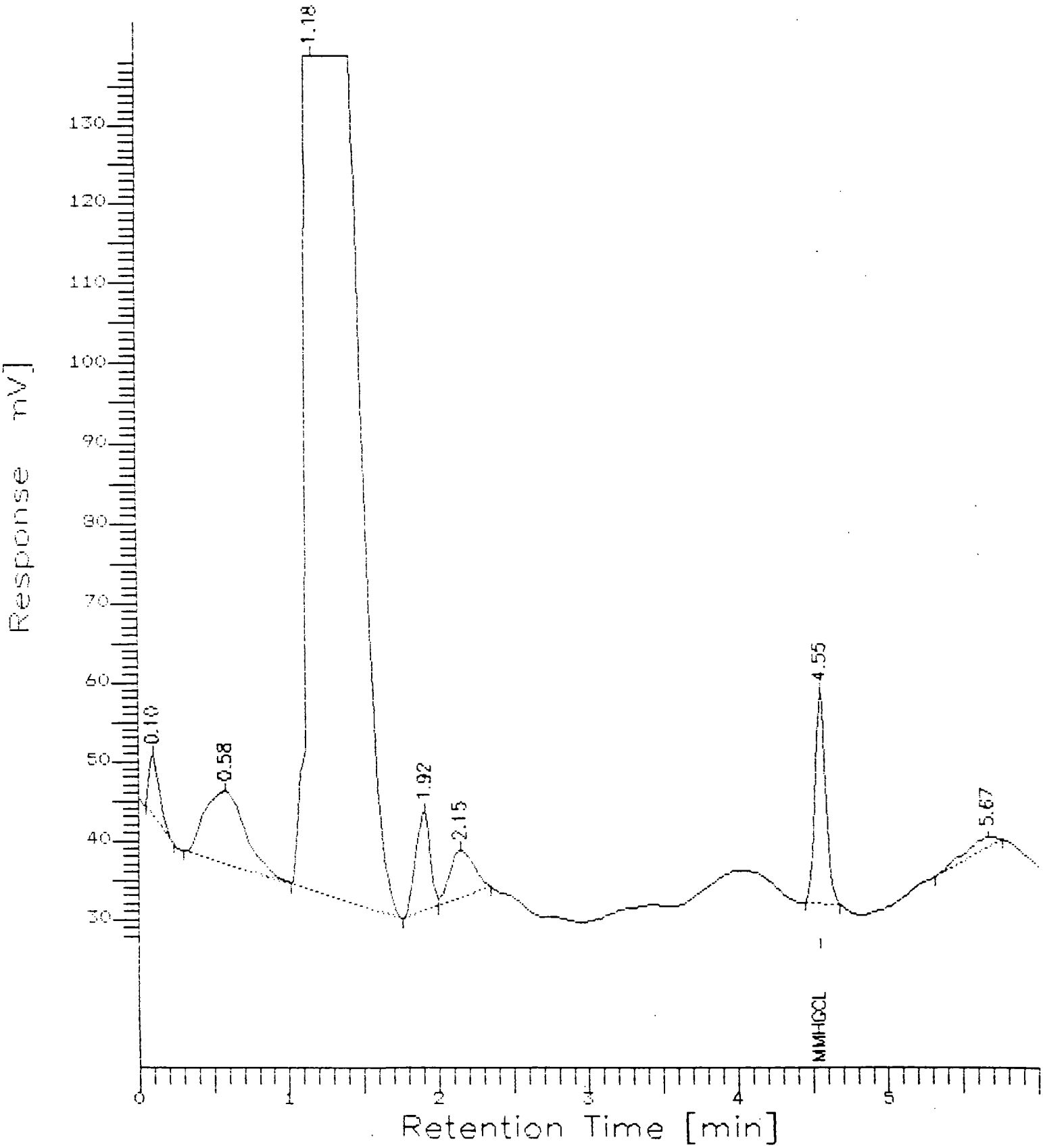
EA ENGINEERING METHYL MERCURY REPORT

Peak #	Component Name	Ret Time [min]	Area [uV-sec]	Height [uV]	BL	RAW AMOUNT PPB	CORRECTED AMOUNT PPB	Area/ Amount	
1		0.100	32941	7693	BB	4.24	4.24	7766.49	
2		0.583	169001	9147	BV	21.76	21.76	7766.49	
3		1.183	8505218	957220	VB	1095.12	1095.12	7766.49	
4		1.917	85649	12542	BV	11.03	11.03	7766.49	
5		2.150	68125	5967	VB	8.77	8.77	7766.49	
6	MMHgCl	4.554	130828	27000	MM	16.85	16.85	7766.49	$\frac{16.85}{15} = 112\%$
7		5.667	24172	1296	BB	3.11	3.11	7766.49	
			9015932	1020865		1160.88	1160.88		

Chromatogram

FileName : EAH6112.RAW
Start Time : 0.00 min
Scale Factor: 0
End Time : 6.00 min
Plot Offset: 28 mV
Date : 12/18/90 9:06
Low Point : 27.65 mV
Plot Scale: 111 mV

High Point : 138.95 mV



Sample Name : 664.06-mMHg STD II @ 5ppb Level 1 Time : 12/9/90 17:03
Sample Number: 135 Study :
Operator : WWL

3 4 0397

Interface # : 4 Channel : A A/D mV Range : 1000
AutoSampler : Hewlett-Packard 7673A
Rack/Vial : 0/1

Data Acquisition Time: 12/11/90 16:28
Delay Time : 0.00 min.
End Time : 6.00 min.
Sampling Rate : 1.0000 pts/sec

Raw Data File : C:\2700\DATA\EAHG135.raw
Result File : C:\2700\DATA\EAHG135.rst
Instrument File: C:\2700\DATA\EAHG.ins
Process File : C:\2700\DATA\EAHG.prc
Sample File : C:\2700\DATA\EAHG1P.smp
Sequence File : C:\2700\DATA\EAHG.seq

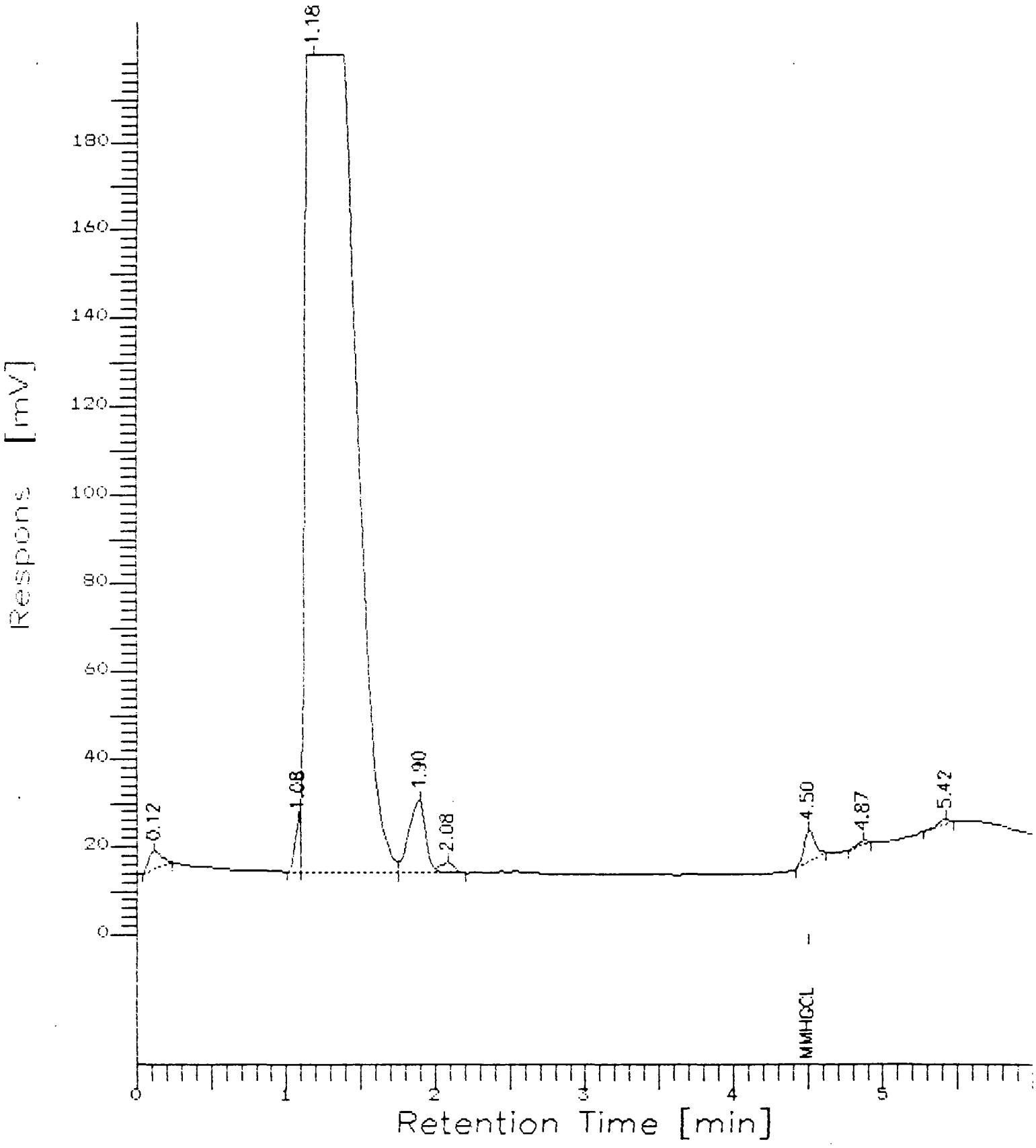
Inj. Volume : 1 ul Area Reject : 0.00
Sample Amount : 1.0000

EA ENGINEERING METHYL MERCURY REPORT

Peak #	Component Name	Ret Time [min]	Area [uV-sec]	Height [uV]	BL	RAW AMOUNT PPB	CORRECTED AMOUNT PPB	Area/ Amount	
1		0.117	25200	3949	BB	3.45	3.45	7307.70	
2		1.083	37033	11763	BV	5.07	5.07	7307.70	
3		1.183	10019235	975947	VV	1371.05	1371.05	7307.70	
4		1.900	127776	16668	VE	17.49	17.49	7307.70	
5		2.083	13948	2413	EB	1.91	1.91	7307.70	100%
6	MMHgCl	4.500	36479	7112	BB	4.99	4.99	7307.70	
7		4.867	5330	968	BB	0.73	0.73	7307.70	
8		5.417	6333	1041	BB	0.87	0.87	7307.70	
			10271333	1019860		1405.55	1405.55		

Chromatogram 3 4 0328

FileName : C:\2700\DATA\EAHG135.raw
Start Time : 0.00 min
End Time : 6.00 min
Scale Factor: 0
Plot Offset: 0 mV
Date : 12/9/90 17:03
Low Point : 0.00 mV
Plot Scale: 200 mV
Page 1 of 1
High Point : 200.00 mV



APPENDIX C
DETECTION LIMIT AND QUANTIFICATION LIMIT USING THE LOW STANDARD (5 ppb)

APPENDIX C
Page 1 of 1
Gregory E. Johnson
ethyl Mercury Method Validation
Date: January 24, 1991
TDL Project No.: 482826

IT ANALYTICAL SERVICES
304 DIRECTORS DRIVE
KNOXVILLE, TENNESSEE

DETECTION LIMIT AND QUANTIFICATION LIMIT

USING THE LOW STANDARD (5 ppb)

MONOMETHYL MERCURY CHLORIDE

DATA FILE	AREA
EAHG065	37900
EAHG066	37964
EAHG067	35842
EAHG068	35819
EAHG069	34346

Mean = 36374.2

σ = 1546.0

% RSD = 4.3% (0.043)

σ (ppb) = 0.043 X 5 ppb = 0.215 ppb

3X for MDL = 0.645 ppb

5X for MQL = 1.075 ppb

Sample Name : 664:06-MMHG II REP1 Time : 1/24/91 14:21
Sample Number: 05 Study :
Operator : MWL

Interface # : 4 Channel : A A/D mV Range : 1000
AutoSampler : Hewlett-Packard 7673A
Rack/Vial : 0/1

Data Acquisition Time: 12/7/90 16:31
Delay Time : 0.00 min.
End Time : 6.00 min.
Sampling Rate : 1.0000 pts/sec

Raw Data File : C:\2700\DATA\EAHG05.raw
Result File : C:\TEMP\EP03F3E.rst
Instrument File: C:\2700\DATA\EAHG.ins
Process File : C:\2700\DATA\EAHG.prc
Sample File : C:\2700\DATA\EAHG1P.smp
Sequence File : C:\2700\DATA\default.seq

Inj. Volume : 1 ul Area Reject : 0.00
Sample Amount : 1.0000

Noise Threshold: 500 Area Threshold : 100 Summ Factor: 1
Dilution Factor: 1.00 Multiplier : 1.00
Divisor : 1.0000 Additive Constant: 0.00

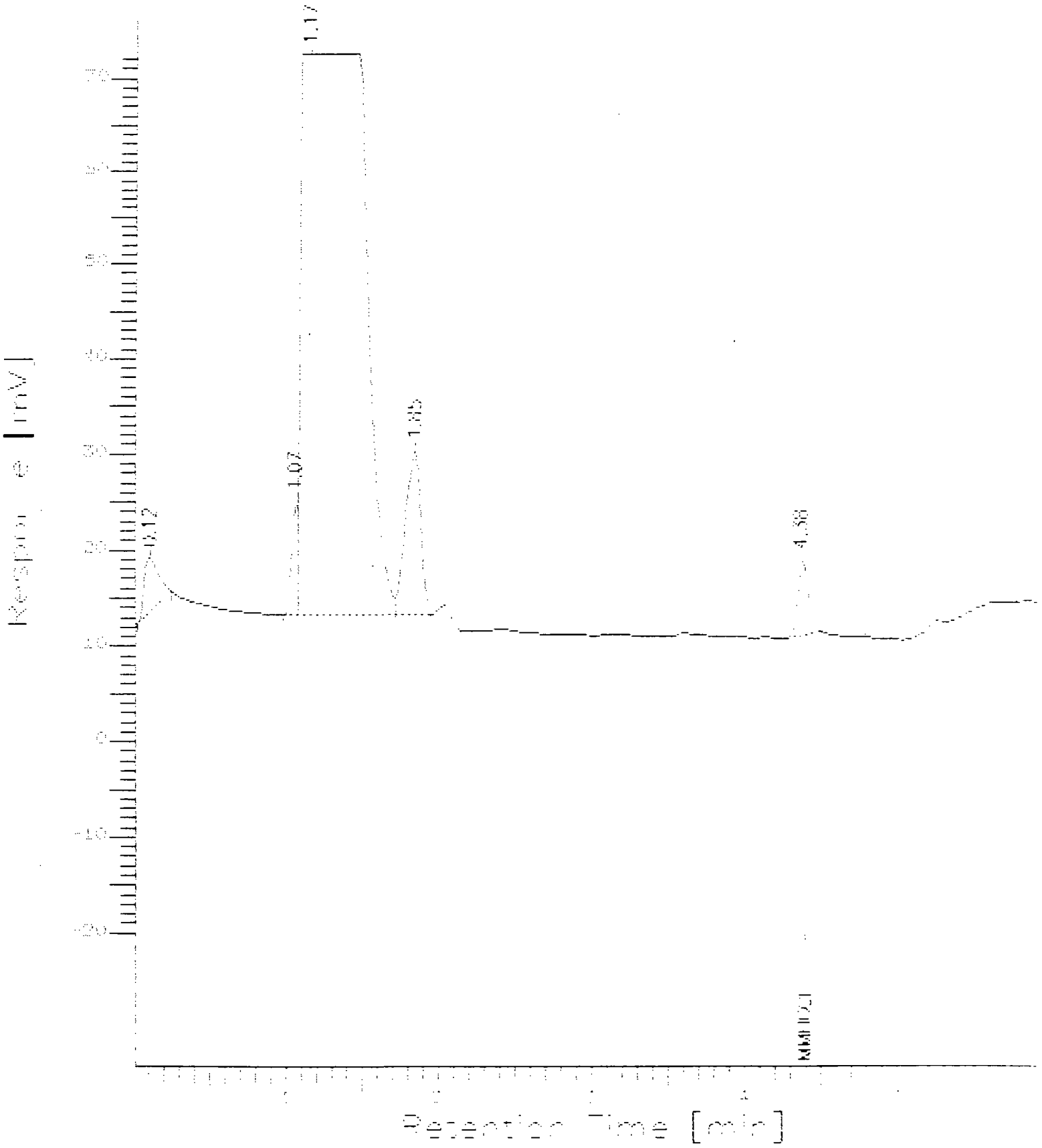
Instrument Conditions:

Total Number of Peaks Detected: 5

EA ENGINEERING METHYL MERCURY REPORT

Peak #	Component Name	Ret Time [min]	Area [uV-sec]	Height [uV]	BL	RAW AMOUNT ppb	CORRECTED AMOUNT ppb	Area/ Amount
1		1.117	37642	5574	BB	5.14	5.14	7322.31
2		1.367	39606	11879	BV	5.41	5.41	7322.31
3		1.167	9469021	977328	VV	1292.18	1293.19	7322.31
4		1.950	116491	17166	VB	15.91	15.91	7322.31
5	MMHgCl	4.333	37900	7688	BB	5.18	5.18	7322.31
			9700660	1019825		1324.81	1324.81	

FileName : D:\2700\DATA\EA450b5.raw
Start Time : 0.00 min End Time : 6.00 min
Scale Factor: 0 Plot Offset: -20 mV
Date : 1/24/91 14:02 Page 1 of 1
Low Point : -20.25 mV High Point : 71.75 mV
Plot Scale: 92 mV



Sample Name : p4/06-MMHG II REP2 Time : 1/04/91 14:04
Sample Number: 66 Study :
Operator : AWL

Interface # : 4 Channel : A A/D mV Range : 1000
AutoSampler : Hewlett-Packard 7673A
Rack/Vial : 0/1

Data Acquisition Time: 12/7/90 16:40
Delay Time : [illegible] min.
End Time : [illegible] min.
Sampling Rate : 1.0000 pts/sec

Raw Data File : C:\2700\DATA\EAHG066.raw
Result File : F:\TEMP\SAS2E3C.ret
Instrument File : C:\2700\DATA\EAHG.ins
Process File : C:\2700\DATA\EAHG.prc
Sample File : C:\2700\DATA\EAHG1P.smp
Sequence File : C:\2700\DATA\default.seq

Inj. Volume : [illegible] ul Area Reject : 0.00
Sample Amount : 1.0000

Noise Threshold: 210 Area Threshold : 100 Bunch Factor: 1
Dilution Factor: 1.00 Multiplier : 1.00
Divisor : 1.0000 Additive Constant: 0.00

Instrument Conditions:

Total Number of Peaks Detected: 8

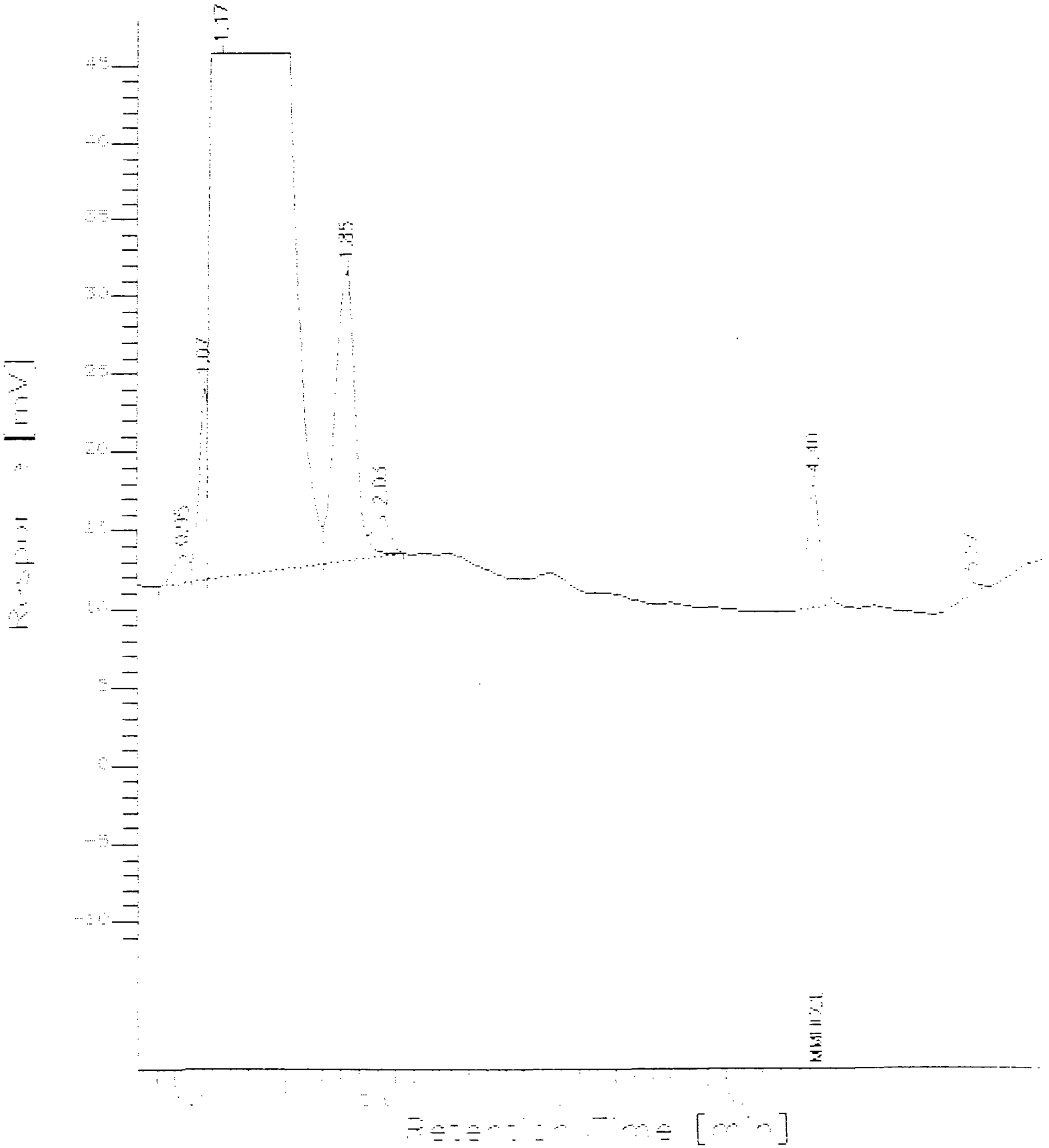
EA ENGINEERING METHYL MERCURY REPORT

Peak #	Component Name	Ret Time [min]	Area [uV-sec]	Height [uV]	BL	RAW AMOUNT PPB	CORRECTED AMOUNT PPB	Area/ Amount
1		0.100	32215	5293	BB	4.40	4.40	7322.97
2		0.950	11699	1757	BV	1.60	1.60	7322.97
3		1.067	41649	12354	VV	5.69	5.69	7322.97
4		1.167	9474122	978562	VV	1293.76	1293.76	7322.97
5		1.350	141182	18563	VE	19.28	19.28	7322.97
6		2.033	12489	2206	EB	1.71	1.71	7322.97
7	MMHgCl	4.400	37964	7901	BB	5.18	5.18	7322.97
8		5.267	4074	731	BB	0.56	0.56	7322.97
			9755392	1027475		1332.16	1332.16	

Chromatogram

FileName : C:\2700\DATA\EAPB056.raw
Start Time : 0.70 min
Scale Factor: 0
End Time : 5.70 min
Plot Offset: -11 mV
Date : 1/24/91 14:04
Low Point : -11.04 mV
Plot Scale: 57 mV

High Point : 45.79 mV



3 4 0335

Sample Name : [illegible] II [illegible]   [illegible] : [illegible]
Sample Number : [illegible]   Study :
Operator : [illegible]

Interface # : 4   Channel : [illegible]   [illegible] Range : [illegible]
AutoSampler : Hewlett-Packard [illegible]
[illegible] Vial : 0/1

Data Acquisition Time: 12/[illegible]/[illegible] [illegible]
[illegible] : [illegible] min.
[illegible] : [illegible] min.
[illegible] Rate : [illegible] pts/sec

[illegible]
[illegible]
Instrument File : [illegible]
[illegible]
Sample File : [illegible]
[illegible]

[illegible] : [illegible] ul   Area Reject : [illegible]
Sample Amount : 1.0000

[illegible] Threshold: [illegible]   Area Threshold : 100   Bunch Factor : [illegible]
Dilution Factor: 1.00   Multiplier : 1.00
Divisor : 1.0000   Additive Constant: 0.00

Instrument Conditions:

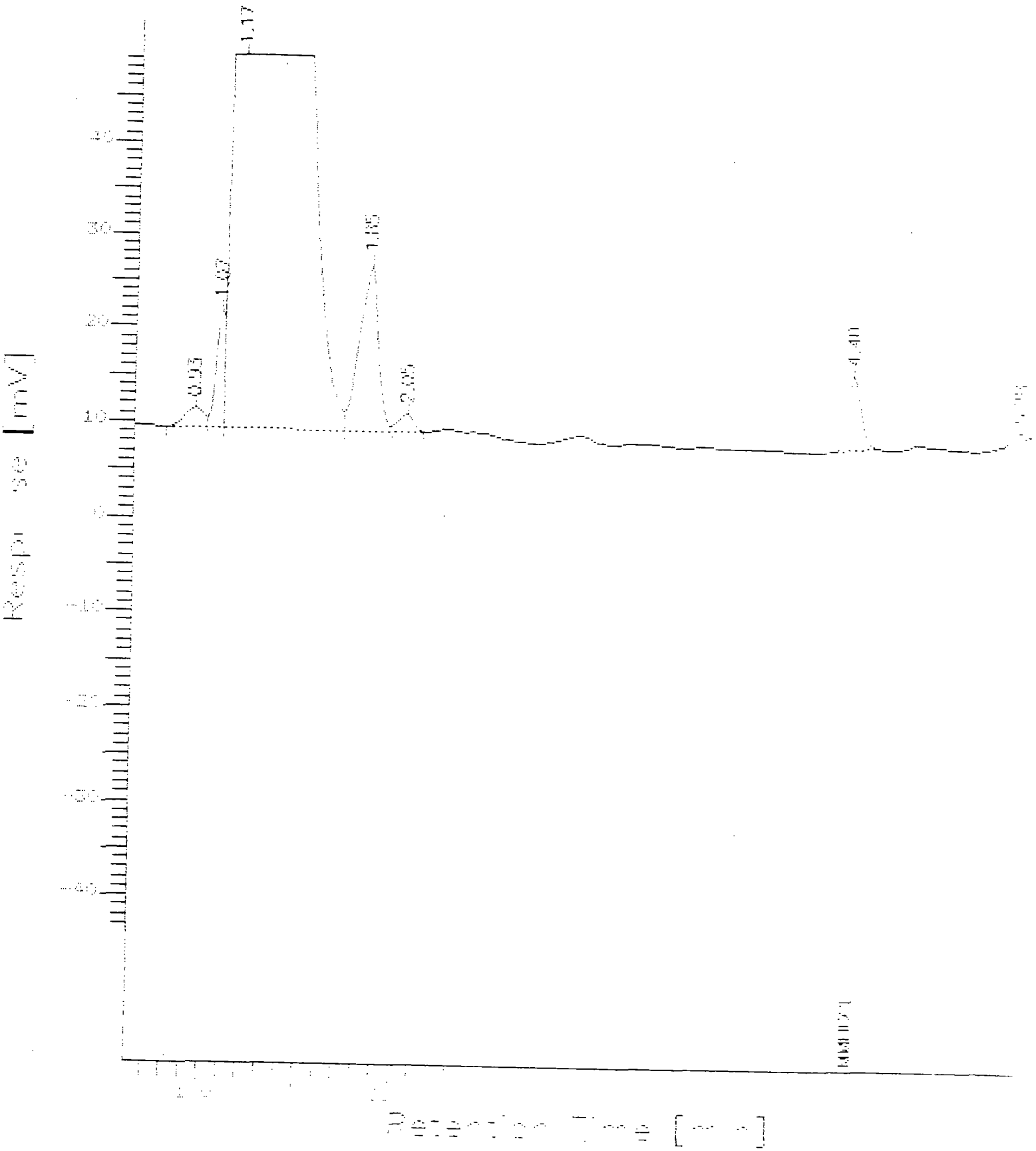
Total Number of Peaks Detected: [illegible]

EA ENGINEERING METHYL MERCURY REPORT

Peak #	Component Name	Ret Time [min]	Area [uV-sec]	Height [uV]	BL	RAW AMOUNT PPB	CORRECTED AMOUNT PPB	Area/ Amount
1		0.117	30318	4624	BB	4.15	4.15	7307.70
2		0.733	15060	2044	BV	2.06	2.06	7307.70
3		1.067	41027	12613	VV	5.61	5.61	7307.70
4		1.167	9445734	981165	VV	1292.57	1292.57	7307.70
5		1.850	120186	17928	VV	16.45	16.45	7307.70
6		2.050	9462	1836	VB	1.30	1.30	7307.70
7	MMHgCl	4.400	35842	7915	BB	4.91	4.91	7307.70
8		5.283	4253	757	BB	0.58	0.58	7307.70
			9701890	1028753		1327.62	1327.62	

FileName : C:\2700\DATA\EAHG0o7.raw
Start Time : 0.62 min End Time : 5.73 min
Scale Factor: 0 Plot Offset: -44 mV

Data : 1/24/91 14:15 Page 1 of 1
Low Point : -45.77 % Upper Point : 15.23 %
Plot Scale: 60 mV



Sample Name : 664:16-MMHG II REP4 Time : 1/24/91 14:06
Sample Number: 66 Study :
Operator : WWL

Interface # : 4 Channel : A A/D mV Range : 1.000
AutoSampler : Hewlett-Packard 7673A
Rack/Vial : 0/1

Data Acquisition Time: 12/7/90 22:37
Delay Time : 0.00 min.
Run Time : 6.00 min.
Sampling Rate : 1.0000 pts/sec

Raw Data File : C:\2700\DATA\EAHG0663.raw
Result File : E:\TEMP*.RES2219.rst
Instrument File: C:\2700\DATA\EAHG.ins
Process File : C:\2700\DATA\EAHG.pro
Sample File : C:\2700\DATA\EAHG1F.smp
Sequence File : C:\2700\DATA\default.seq

Inj. Volume : 1 µl Area Reject : 0.00
Sample Amount : 1.0000

Noise Threshold: 300 Area Threshold : 100 Dunon Factor: 1
Dilution Factor: 1.00 Multiplier : 1.00
Divisor : 1.0000 Additive Constant: 0.00

Instrument Conditions:

Total Number of Peaks Detected: 6

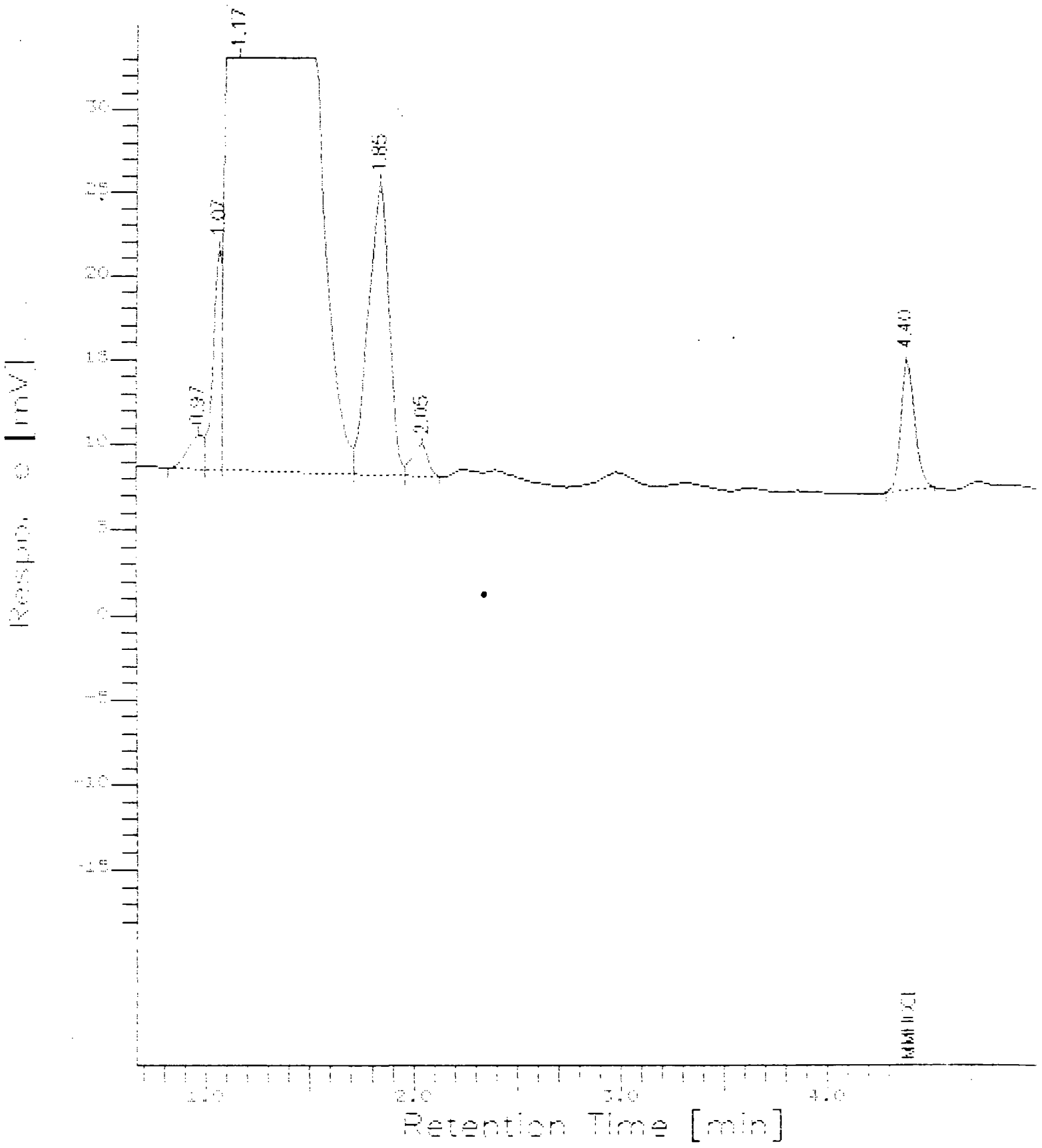
EA ENGINEERING METHYL MERCURY REPORT

Peak #	Component Name	Ret Time [min]	Area [uV-sec]	Height [uV]	BL	RAW AMOUNT PPB	CORRECTED AMOUNT PPB	Area/ Amount
1		0.117	26612	4330	BB	3.64	3.64	7307.70
2		0.967	12402	2042	BV	1.70	1.70	7307.70
3		1.067	43993	13182	VV	6.02	6.02	7307.70
4		1.167	9368698	962045	VV	1282.03	1282.03	7307.70
5		1.350	119207	17440	VV	16.31	16.31	7307.70
6		2.050	9670	1860	VB	1.32	1.32	7307.70
7	MMHgCl	4.400	35819	7722	BB	4.90	4.90	7307.70
8		5.283	4490	876	BB	0.61	0.61	7307.70
			9620881	1029497		1316.54	1316.54	

Chromatogram 3 4 0338

FileName : C:\2700\DATA\EAH6068.raw
Start Time : 0.37 min
Scale Factor: 0
End Time : 5.07 min
Plot Offset: -19 mV
Date : 1/24/91 14:06
Low Point : -18.77 mV
Plot Scale: 52 mV

High Point : 33.07 mV



Sample Name : 664:06-MMHG II REP5 Time : 1/24/91 14:07
Sample Number: 69 Study :
Operator : WWL

Interface # : 4 Channel : A A/D mV Range : 1000
AutoSampler : Hewlett-Packard 7673A
Rack/Vial : 0/1

Data Acquisition Time: 12/7/90 17:08 3 4 0339
Delay Time : 0.00 min.
End Time : 6.00 min.
Sampling Rate : 1.0000 pts/sec

Raw Data File : C:\2700\DATA\EAHG069.raw
Result File : S:\TEMP\MERGED0.rst
Instrument File: C:\2700\DATA\EAHG.ins
Process File : C:\2700\DATA\EAHG.prc
Sample File : C:\2700\DATA\EAHG1P.smp
Sequence File : C:\2700\DATA\default.seq

Inj. Volume : 1 ul Area Reject : 0.00
Sample Amount : 1.0000

Noise Threshold: 300 Area Threshold : 100 Bunch Factor: 1
Dilution Factor: 1.00 Multiplier : 1.00
Divisor : 1.0000 Additive Constant: 0.00

Instrument Conditions:

Total Number of Peaks Detected: 7

EA ENGINEERING METHYL MERCURY REPORT

Peak #	Component Name	Ret Time [min]	Area [uV-sec]	Height [uV]	BL	RAW AMOUNT ppb	CORRECTED AMOUNT ppb	Area/ Amount
1		0.117	30760	4558	BB	4.21	4.21	7307.70
2		1.067	38959	12537	BV	5.33	5.33	7307.70
3		1.167	9352669	983594	VV	1279.84	1279.84	7307.70
4		1.850	111377	16439	VB	15.24	15.24	7307.70
5		2.300	18432	1243	BB	2.52	2.52	7307.70
6	MMHgCl	4.417	34346	7298	BB	4.70	4.70	7307.70
7		5.283	5324	1049	BB	0.73	0.73	7307.70
			9591866	1026818		1312.57	1312.57	

Chromatogram 3 4 0340

FileName : C:\2700\DATA\EAHG069.raw
Start Time : 0.82 min End Time : 4.88 min
Scale Factor: 0 Plot Offset: -20 mV

Date : 1/24/91 14:07
Low Point : -20.35 mV High Point : 52.22 mV
Plot Scale: 73 mV