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CNC CHARLESTON  
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MINUTES AND AGENDA FROM RESTORATION ADVISORY BOARD COMMUNITY  
RELATIONS SUBCOMMITTEE MEETING DATED 10 OCTOBER 1995 CNC  
CHARLESTON SC  
10/10/1995  
CNC CHARLESTON

# COMMUNITY RELATIONS SUBCOMMITTEE MEETING AGENDA

DATE: TUESDAY, 10 OCTOBER 1995  
TIME: 4:00PM  
PLACE: NH-51 CONFERENCE ROOM

## AGENDA ITEMS

- STATUS OF COMMUNITY RELATIONS PLAN *CRP*  
*Hard out on + Poster. Incorporate change, Get out by produce in Memphis, NHP RAB 14 Nov 95*
- ~~FACT SHEET FOR~~ INVESTIGATION RESULTS
- FACT SHEET FOR OTHER ENVIRONMENTAL CONCERNS
- SUBCOMMITTEE REPORT FOR RAB MEETING
- OTHER TOPICS

- FACT SHEET ON LEASING PROCESS, To be mailed  
*by ENSA to CRP members.*

- *Need <sup>Further info</sup> on Leasing Process*
- *FACT sheet Leasing Process*

- *I needly RAB for when they been releasable*  
*available + releasable*

- *Issue for CR Sub Com*  
*to address, question, suggestion,*

## COMMUNITY RELATIONS SUBCOMMITTEE MEETING AGENDA

DATE: TUESDAY, 10 OCTOBER 1995  
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### AGENDA ITEMS

- STATUS OF COMMUNITY RELATIONS PLAN
- FACT SHEET FOR INVESTIGATION RESULTS
- FACT SHEET FOR OTHER ENVIRONMENTAL CONCERNS
- SUBCOMMITTEE REPORT FOR RAB MEETING
- OTHER TOPICS
  - Community Relations Plan to be complete and ready for distribution at the next RAB meeting 14 Nov 95
  - Community Relation Subcommittee saw the need for further information concerning the leasing process.
  - Started work on a FACT SHEET for the Leasing Process.
  - Working on format for a handout and poster for <sup>the RAB</sup> investigation results. The poster and handout will be distributed when investigation results are available and releasable.
  - The Community Relations Subcommittee is asking the RAB for ~~input~~ issues, questions and suggestions for the CR Subcommittee <sup>to address</sup>. We would like there to be given to us verbally tonight if you have such concerns.

# ISSUES, Questions, Suggestions From RAB

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1. Using EnSafe for community outreach. (Doyle EPA)  
(Enure Justice) can also be addressed.  
Getting the word out to the committee.
2. Meeting location determine location for RAB.  
(Chekoree church location) (Daytime meeting)  
move back closer to community.
- ~~3.~~ Using something as a drawing Lunch, Drop in etc
3. More information out to people in the immediate area.
4. Going out to different community groups talking about the MB Cleanup. Get them Meeting announcement



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION IV

345 COURTLAND STREET, N.E.  
ATLANTA, GEORGIA 30365

4WD-FFB

October 13, 1995

HAND DELIVERY

Daryle L. Fontenot, P.E.  
Department of the Navy  
Southern Division  
Naval Facilities Engineering Command  
P.O. Box 190010  
North Charleston, S.C. 29419-9010

SUBJ: Data Validation Review and Laboratory Audits for Naval  
Base Charleston

Dear Mr. Fontenot:

The U.S. Environmental Protection Agency (EPA) has completed the data validation review and laboratory audits for Naval Base Charleston. The purpose of these activities was to establish confidence in the procedures used, analytical data generated, and in the data validation program. As a result of this review, EPA has determined that confidence can be placed in these data.

Specific comments are identified in the enclosures. If you have any questions, please call me at (803) 743-9985, or (404) 347-3555, Voice Mail Extension 2061.

Sincerely,

A handwritten signature in cursive script that reads "Doyle T. Brittain".

Doyle T. Brittain  
Senior Remedial Project Manager

Enclosure

cc: Bobby Dearhart, CNSY  
Joe Bowers, SCDHEC  
Jeannie Olano, SCDHEC  
Ann Ragan, SCDHEC

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
REGION IV  
ENVIRONMENTAL SERVICES DIVISION  
960 COLLEGE STATION RD.  
ATHENS, GA 30605-2720

August 17, 1995

MEMORANDUM

**SUBJECT:** Data Validation Review and Laboratory Audits for  
Naval Base Charleston, Charleston, SC

**FROM:** Gary Bennett, Acting Chief *Gary Bennett*  
Laboratory Evaluation and  
Quality Assurance Section

**TO:** Jon D. Johnston, Chief  
Federal Facilities Branch  
Waste Management Division

As you requested in your May 8, 1995 memoranda, staff of the Laboratory Evaluation and Quality Assurance Section (LEQAS) have completed a number of on-site laboratory audits and a review of the data validation service involved in the RCRA RFI at Naval Base Charleston (NBC). LEQAS will issue detailed reports on each of the individual on-site evaluations within the next 45 days. However, since you indicated that these evaluations were a high priority, this memorandum provides a synopsis of our evaluations and conclusions.

After receipt of your memoranda, a follow-up meeting was held with Mr. Doyle Brittain, Senior Remedial Project Manager for NBC, on May 28 to further define the goals of the requested on-site visits. Mr. Brittain indicated that the primary goal of the on-site audits was to determine if there were any irregularities associated with the generation and validation of the NBC RFI data and to determine if the data were of suitable quality for decision making at the facility. Subsequently, telephone calls were made to Ms. Tina Cantwell of EnSafe / Allen & Hoshall, the Navy's prime RFI contractor, to establish a schedule for visiting the facilities. Ms. Cantwell was very cooperative in arranging visits to each of Ensafe's subcontractors involved in the NBC RFI.

The following is a schedule and summary of our findings at each of the facilities evaluated:

**July 11, 1995 - Validata, Norcross, GA** - Validata is a third party data validation service under contract to Ensafe to perform validation of all NBC RFI data. LEQAS had performed data validations on a portion of the NBC RFI data submitted by Savannah Laboratories and Pace Laboratories/Indianapolis

on samples collected from August - December, 1994 (see March 16 memo from John McConney, LEQAS, to Doyle Brittain). Since Validata had also reviewed the same data, the LEQAS data review can be considered an overview of the technical quality of Validata's services. No significant discrepancies or deficiencies were noted with the data during the LEQAS review.

Because the technical quality of Validata's services had been assessed through the LEQAS independent data validation, the on-site visit to Validata consisted of an administrative review to determine the company's policies and procedures. The company has three full time employees, but the remainder of it's employees also perform laboratory work for other firms while doing part-time data validation for Validata. According to stated company policy, employees are not allowed to review data from their own laboratory. Validata uses EPA data review guidelines for the normal target compound and target analyte constituents. However, for the validation of dioxin/furan data, for which no EPA data review guidelines exist, the company did not have a formal data review standard operating procedure.

**July 19, 1995 - Savannah Laboratories, Savannah, Georgia -** An on-site evaluation and data audit was performed at this laboratory. NBC RFI data from this lab was also reviewed by LEQAS in the set of data provided by the Navy and no significant problems were noted. One minor deficiency was noted during the on-site visit in TCLP Method 1311; no deviations were noted during the data audit. Based on our evaluation, this laboratory has provided quality data in support of the RFI.

**July 25, 1995 - Compuchem Laboratories, Research Triangle Park, North Carolina -** An on-site data audit for NBC RFI data was performed at this lab. Compuchem is an EPA Contract Laboratory Program (CLP) firm and receives a significant amount of oversight from the Agency. CLP data packages are routinely reviewed by LEQAS and the lab receives quarterly blind performance evaluation samples, as well as routine on-site visits from the Region 4 CLP Technical Project Officer, Mr. Tom Bennett. Because of this technical oversight, the on-site visit was limited to a data audit. No deviations or irregularities were noted in the NBC RFI data. Based on our evaluation, this laboratory has provided quality data in support of the RFI.

**July 25, 1995 - PACE Laboratories, Indianapolis, Indiana -** An on-site evaluation and data audit was performed at this laboratory. This laboratory provided dioxin/furan data for the RFI. A review of this laboratory's data by Validata resulted in questions about the validity of the data with several analytical issues discussed. A series of exchanges between the validation service, the lab, and Ensaf resolved

these questions. Our on-site visit revealed no major irregularities with the analytical procedures or the data generated. Though there is room for improvement, the deficiencies noted during the audit do not seriously impact data quality. Based on our evaluation, the laboratory provided reliable data for the RFI.

**July 27, 1995** - PACE Laboratories, Hampton, New Hampshire - PACE is an EPA Contract Laboratory Program (CLP) firm and receives a significant amount of oversight from the Agency. CLP data packages are routinely reviewed by LEQAS and the lab receives quarterly blind performance evaluation samples, as well as routine on-site visits from the Region 1 CLP Technical Project Officers, Ms. Deb Szaro and Ms. Moira Lataille. No deviations or irregularities were noted in the NBC RFI data. Based on our evaluation, this laboratory has provided quality data in support of the RFI.

To summarize our findings, we found no irregularities at any of the laboratories we visited. The data we examined was generated using the appropriate analytical methods with acceptable quality control procedures and was well-documented. The issues raised by Validata for the RFI dioxin/furan data appeared to be due to incomplete documentation received with the data packages. Most of the missing documentation was subsequently provided by the laboratory. Since a third party data validation service has no direct control over the contractual arrangements for deliverables made between the prime contractor and the laboratory, it is understandable that complications may arise when validating non-standard analyses such as dioxins/furans.

Please contact me at 706/546-3287 if you have any questions or comments.

cc: John Marlar, ESD  
Doyle Brittain, WMD  
Tom Bennett, ESD



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345 COURTLAND STREET, N.E.  
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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
REGION IV  
ENVIRONMENTAL SERVICES DIVISION  
960 COLLEGE STATION RD.  
ATHENS, GA 30605-2720

August 17, 1995

MEMORANDUM

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Naval Base Charleston, Charleston, SC

**FROM:** Gary Bennett, Acting Chief  
Laboratory Evaluation and  
Quality Assurance Section

*Gary Bennett*

**TO:** Jon D. Johnston, Chief  
Federal Facilities Branch  
Waste Management Division

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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
REGION IV  
ENVIRONMENTAL SERVICES DIVISION  
960 COLLEGE STATION RD.  
ATHENS, GA 30605-2720

August 30, 1995

MEMORANDUM

**SUBJECT:** Data Validation Review and Laboratory Audits for  
Naval Base Charleston, Charleston, SC

**FROM:** Gary Bennett, Acting Chief *G. Bennett*  
Laboratory Evaluation and  
Quality Assurance Section

**TO:** Jon D. Johnston, Chief  
Federal Facilities Branch  
Waste Management Division

As you requested in your May 8, 1995 memoranda, staff of the Laboratory Evaluation and Quality Assurance Section (LEQAS) conducted a number of on-site laboratory audits and a review of the data validation service involved in the RCRA RFI at Naval Base Charleston (NBC). My memo of August 18 provided a synopsis of the findings at each of the facilities evaluated. Enclosed are detailed reports on each of the individual on-site evaluations conducted.

Please contact me at 706/546-3287 if you have any questions or comments.

Enclosures

cc: Doyle Brittain, WMD w/ enclosures

## ADMINISTRATIVE REVIEW

Validata Chemical Services, Inc.  
P.O. Box 930422  
Norcross, GA 30093

### INTRODUCTION

An administrative review was conducted at Validata Chemical Services, Inc. (Validata), Norcross, GA, on July 11, 1995. This audit was conducted by the U.S. Environmental Protection Agency, (EPA) Region IV, Environmental Services Division, Laboratory Evaluation & Quality Assurance (LEQA) Section. The audit team consisted of Gary Bennett, John McConney and Yolanda Brown. The purpose of the audit was to review and assess the adequacy of the data review process and to evaluate the company's staffing/qualifications, procedures, management review process and effectiveness. The audit was conducted with Kevin Harmon, Client Services Director, Validata and Tony Hunt, Charleston Naval Base Project Manager, Southern Division.

### FINDINGS

#### A. Staffing/Qualifications

At the time of this audit, Validata had a staff of seventeen Chemists. The staff is composed of three full-time Chemists and fourteen part-time Chemists, who are on-call when needed. All personnel work out of their homes and have a computer, in which the review document is generated. All personnel, with the exception of one, have a minimum of a B.S. degree and background in analytical CLP work. The non-degreed employee has a strong background in Solid Waste 846 Methods. Each employee has completed in-house training in the validation procedures. The training is designed to cover the full range validation process required to effectively review laboratory data. The company's stated policy is that no part-time employee may review data from a laboratory where that person holds employment.

#### B. Procedures

Validata developed and follows a set of Standard Operating Procedures (SOP's). The SOP's are in a checklist format for volatiles, semivolatiles, inorganics, herbicides and pesticides. The checklists are composed of the guidance within the EPA's National Functional Guidelines. An abbreviated dioxin/furan checklist which followed the major requirements of EPA Method 8290 was also used. Validata's SOP's incorporate a three step validation process: Validation, Validation Check by a Senior Chemist and the Final Check.

### C. Management Reviews

The management reviews consist of an overview (by a Senior Chemist) and a second overview (by Kevin Harmon) of the data package. This process is utilized to provide two additional reviews/overviews by management to insure a quality product has been produced by the laboratory. The data must be of suitable quality for decision making purposes by the Naval Base Charleston officials as specified in the Quality Assurance Project Plans for the site.

### D. Effectiveness

The quality of Validata's data validation process had previously been examined by EPA's LEQA Section by review of previously validated data. No major discrepancies were noted during this overview.

## **RECOMMENDATIONS**

1. Maintain a central file for all data review projects. Presently, each Chemist is maintaining the file copy of all projects they work on at their home. It was recommended that a central file be maintained at the Validata main office, which will enable enhanced control of the company's records. It was noted that the central office does maintain a computer file for all projects.
2. Develop a comprehensive standardized data review checklist for dioxin/furan validation. This checklist should include necessary measures to take when quality control criteria are exceeded and should reference specific Method 8290 requirements.

## **CONCLUSIONS**

In conclusion, the review of the procedures utilized by Validata for the process of data validation of samples for the Charleston Naval Base project appear to be effective in the determination of data quality.

**EXECUTIVE SUMMARY**  
**LABORATORY EVALUATION REPORT**  
**Savannah Laboratories**  
**5102 LaRoche Avenue**  
**Savannah, Georgia 31416**

**INTRODUCTION**

On July 19, 1995, an on-site evaluation was conducted at Savannah Laboratories, Savannah, Georgia. The evaluation was performed at the request of the Federal Facilities Branch, Waste Management Division, USEPA, Region IV, and was related to analytical services the laboratory provided for a RCRA Facility Investigation (RFI) at the US Navy's Charleston Naval Base. The evaluation was conducted by Gary Bennett and Yolanda Brown of the Laboratory Evaluation/Quality Assurance Section (LEQAS), Region IV Environmental Services Division.

The purpose of the evaluation was to assess the laboratory's capability to perform both inorganic and organic analyses and to perform a data audit of the Charleston Naval Base RFI data. The findings of this evaluation are based upon information provided by the laboratory as well as the personal observations of the evaluators.

**FINDINGS/RECOMMENDATIONS**

Details of all findings are contained in the attached laboratory evaluation guidelines checklist. The following is a summary of the most notable findings of this evaluation, and when appropriate, a recommendation for improvement or correction of any deficiencies noted.

General Information

The laboratory has been in existence for approximately fifteen years and employs a total of 157 personnel. The laboratory operates on a 24 hour basis with three shifts. The laboratory had a written Quality Assurance (QA) Plan and Standard Operating Procedure (SOP) in place at the time of the visit.

Facilities, Equipment and Personnel

The facilities included office, laboratory, and sample receipt areas. The laboratory facility contained approximately 30,000 square feet. A new laboratory is being constructed adjacent to the current building and will be ready for occupancy later this year. Portions of the laboratory's operation will be moved into the new building. Sample receipt and preparation areas were segregated from instrumental analysis areas. All office and laboratory space was clean and orderly. The laboratory contained the appropriate instrumentation and

equipment to perform all the analyses evaluated. All personnel interviewed were cooperative and appeared to be competent in their areas of expertise.

SECTION I. SAMPLE RECEIPT AND STORAGE

Recommendations: None

GENERAL OBSERVATIONS FOR ORGANICS - SECTIONS II.- VI.

Recommendations: None

SECTION VII. SAMPLE PREPARATION - INORGANIC

Recommendations: None

SECTION VIII. METALS ANALYSIS

Recommendations: None

SECTION IX. CYANIDE ANALYSIS

Recommendations: None

SECTION X -XI. TCLP EXTRACTION FOR METALS, EXTRACTABLES, AND PESTICIDES, AND VOLATILES

Observations: Samples to be analyzed for extractables, pesticides and metals were extracted in plastic extraction bottles.

**Recommendations: Samples to be analyzed for extractables and pesticides should be extracted in glass or teflon containers.**

SECTION XII. FILE AUDIT

Recommendations: None.

LABORATORY EVALUATION AND QUALITY ASSURANCE SECTION  
U.S. EPA REGION IV  
ATHENS, GEORGIA

Lab Name: Savannah Laboratories  
Address: 5102 LaRoche Avenue, Savannah, GA 31416  
Date of Evaluation: July 19, 1995

INTRODUCTION

- Purpose of Evaluation - The purpose of the lab audit is to assess the laboratory's overall practices and functions in performing analyses. This is accomplished by interviews with laboratory personnel and by personal observations of EPA, Region IV lab evaluators. If problem areas are identified, an effort will be initiated by the evaluators to furnish recommendations for improvement or correction, with the overall goal being to assure that the Agency receives data appropriate for the projects concerned. The product of the evaluation will be a report of all the information received. This evaluation process does not serve as a certification program and as such will not result in any "list" of approved laboratories.

- Scope of Evaluation - The evaluation will include, but is not limited to: information on personnel qualifications, facilities and equipment, sample custody, sample prep and analysis, data reduction, quality assurance/quality control, and records of all processes.

PERSONNEL CONDUCTING THE EVALUATION

Name/Organization Yolanda Brown, LEQA

Name/Organization Gary Bennett, LEQA

Organization Code:

LEQA = Laboratory Evaluation and Quality Assurance Section of  
USEPA, Region IV, Environmental Services Division

GENERAL INFORMATION

How long has the laboratory been in operation? 15 years

Name of Laboratory Manager Janette Long  
BS/Chemistry, 19 years experience

Name of Quality Assurance Coordinator (education/experience)  
Wayne Robbins  
BS/Chemistry, 12 years experience

Total number of personnel 157 (Obtain organizational chart and employee list if possible)

Total Technical Personnel 157  
Total Clerical Personnel \_\_\_\_\_

Total area of facility	<u>30,000</u>	sq. ft.
Laboratory area	<u>21,000</u>	sq. ft.
Offices area	<u>9,000</u>	sq. ft.

Is there a written Standard Operating Procedure for all laboratory operations? x yes \_\_\_\_\_ no

Is there a separate written Quality Assurance Manual for all Laboratory operations? x yes \_\_\_\_\_ no

Date of preparation 12/94

Are there files maintained on each project? x yes \_\_\_ no

Are files/records maintained such that ALL information for each project is available for future reference? x yes \_\_\_ no  
Exceptions: \_\_\_\_\_

How long are these files maintained? \_\_\_\_\_

Is there a laboratory information management (LIMS) system? x yes \_\_\_ no

SECTION I. SAMPLE RECEIPT AND STORAGE AREA

YES NO

Is there a person designated for receipt and storage responsibility?

x \_\_\_\_\_Name Gloria Fullwood Title Dep. Custody Manager1. Is there a sample receipt section in the SOP? x \_\_\_\_\_  
If so, is it available in the sample receipt area? x \_\_\_\_\_2. Is sample custody maintained and documented? x \_\_\_\_\_3. a. Is a sample log maintained? x \_\_\_\_\_b. Are unique numbers assigned to each sample? x \_\_\_\_\_4. a. Is sample container integrity verified? x \_\_\_\_\_b. Are samples monitored for correct preservation? x \_\_\_\_\_Checked in sample analysis area, containers are not opened in sample receipt area.

How are samples that are improperly preserved and/or received in inappropriate containers handled?

Noted on custody excursion sheet, given to project manager for resolution with client.5. Are sample labels and chain of custody records cross checked? x \_\_\_\_\_6. a. Are adequate facilities for sample storage available? x \_\_\_\_\_b. Are Volatiles kept separate from others? x \_\_\_\_\_c. Are cold storage temperatures routinely checked and recorded? x \_\_\_\_\_d. Are temperature excursions noted, with appropriate action taken? x \_\_\_\_\_7. Are all sample receipt procedures/documents consistent with the SOP? x \_\_\_\_\_

Comments:

8. Is sample age monitored and communicated to analysts? x \_\_\_\_\_

SECTION I. - COMMENTS:



11. Are extracts properly stored in refrigerated areas?   x
12. Are extracts stored separately from standards?   x
13. Is the appropriate extraction section of the SOP available to analysts?   x
14. Do analysts monitor and maintain lot numbers of:
- a. solvents   x
  - b. spiking solutions   x
  - c. clean-up adsorbents (alumina, florisil, etc)   x

SECTION II. - COMMENTS:

- Soil samples extracted with sonication.
- Spike & surrogate standards prepared in extraction area.
- Spike & surrogate standards analyzed prior to use with samples.

## SECTION III. STANDARDS PREPARATION - ORGANICS

	YES	NO
Is a person designated in charge of standard prep?	<u>  x  </u>	_____
Name <u>  Bernard Kirkland  </u> Title <u>  Chemist  </u>		
Education <u>  BS - Chemistry  </u> Experience <u>  4 years  </u>		
1. Are stock standards obtained from certified sources?	<u>  x  </u>	_____
2. Are volatile and semivolatile solutions kept in separate areas?	<u>  x  </u>	_____
3. Are standards properly kept under refrigeration?	<u>  x  </u>	_____
4. a. Are log books of standard preparations maintained?	<u>  x  </u>	_____
b. Does the log book contain:		
- reference to stock standard used in prep?	<u>  x  </u>	_____
- identity of person preparing standard?	<u>  x  </u>	_____
- date of preparation?	<u>  x  </u>	_____
- analyte concentrations?	<u>  x  </u>	_____
5. Are working standards properly labeled with:		
a. analyte concentrations	<u>  x  </u>	_____
b. date of preparation	<u>  x  </u>	_____
6. Are standards prepared fresh at an acceptable frequency?	<u>  x  </u>	_____
7. Does the SOP contain a section on Standards Prep? If so, is it posted within the prep area?	<u>  x  </u> <u>  x  </u>	_____ _____
8. Are primary standards traceable to references?	<u>  x  </u>	_____

## SECTION III. - COMMENTS:

- All neat standards have certificates on file.

## SECTION IV. - VOLATILES ANALYSIS (VOAs)

YES NO

Is a person designated in charge of VOA analyses?   x       Name   Myron Young   Title   Chemist  Education   BS/Chemistry   Experience   10 years  1. Are VOAs analyzed by EPA methods/GC/MS?   x       By method: 624/CLP  
RCRA 8260If not, list method and general technique used:  
Analyses are based on RCRA method 8260 or CLP SOW if requested

2. List instrumentation used for VOA analysis:

  2 HP 5872, 2 HP 5971, 2 HP 5970  Is this instrumentation dedicated to VOAs?   x       3. Are methods (SOPs) available to analysts in the instrumentation area?   x       4. Is the VOA analysis area segregated from other sections of the laboratory?   x       5. a. Are analysis log books used and kept at the instrument?   x       b. Do they include all injections/analyses made on the instrument?   x       c. Are holding times monitored by analysts?   x       

d. Does the record include:

- analyst(s) name   x       - date of analysis   x       - time of analysis   x       e. Are the raw data (chromatograms,.etc) labeled so as to be able to relate to the log book?   x

## SECTION IV. - CONTINUED

	YES	NO
6. Are instrument maintenance logs used?	<u>x</u>	___
7. Is the GC/MS properly tuned (at least every 12 hours) with BFB? <u>12 hrs for 8260, 24 hrs for 625</u>	<u>x</u>	___
8. a. Is there an initial standard calibration curve?	<u>x</u>	___
b. Are there continuing calibration standards analyzed at an acceptable frequency (at least every 12 hours)?	<u>x</u>	___
c. Do the analysts monitor requirements for %RSD for the RRFs and for minimum average RRFs?	<u>x</u>	___
d. Is there documentation of all these calibrations?	<u>x</u>	___
e. Is the SOP clear as to what constitutes acceptable calibrations and for appropriate corrective actions, when needed?	<u>x</u>	___
9. Does the SOP clearly define acceptable blanks and note corrective actions, if needed?	<u>x</u>	___
If there are trace blank contaminants that are also in samples, how are they accounted for in the final reported data? <u>Compounds are flagged in final data report.</u>		
10. Are efforts made to identify non-target analytes?	<u>x</u>	___
11. Are all raw data maintained, either on hard copy or magnetic tape?	<u>x</u>	___
12. Does the lab have the necessary equipment to do low level soil work, ie., heated purge and trap?	<u>x</u>	___
If so, is there a separate calibration for the heated analysis?	<u>x</u>	___
13. Are internal standards/surrogates used to monitor instrument/analysis performance?	<u>x</u>	___
If so, do the analysts perform and document corrective action when the criteria are exceeded?	<u>x</u>	___
14. Is there any secondary review of all documents/data by any person other than the one generating the information?	<u>x</u>	___

15. Is there a system of data qualifiers (or other means) to denote reported data that have not met all QC criteria, ie., holding times exceeded, surrogates out, internal standards out, etc.?

  x   \_\_\_\_\_

SECTION IV. - COMMENTS:

## SECTION V. - SEMIVOLATILES (SVs)

	YES	NO
Is a person designated in charge of SVs?	<u>  x  </u>	<u>      </u>
Name <u>  Bernard Kirkland  </u> Title <u>  Department Manager  </u>		
Education <u>  BS Chemistry  </u> Experience <u>  4 years  </u>		
1. Are SVs analyzed by EPA methods (GC/MS)? By method: 625/CLP RCRA 8270	<u>  x  </u>	<u>      </u>
2. List instrumentation used for SV analysis: <u>  4-HP 5970, 1-HP 5971 MSD, 1-HP 5972  </u>		
3. Are methods (SOPs) available to analysts in the instrumentation area?	<u>  x  </u>	<u>      </u>
4. a. Are analysis log books used and kept at the instrument?	<u>  x  </u>	<u>      </u>
b. Do they include all injections made on the instrument?	<u>  x  </u>	<u>      </u>
c. Are holding times monitored by analysts?	<u>  x  </u>	<u>      </u>
d. Does the record include:		
- analyst(s) name	<u>  x  </u>	<u>      </u>
- date of analysis	<u>  x  </u>	<u>      </u>
- time of analysis	<u>  x  </u>	<u>      </u>
e. Are the raw data (chromatograms, etc) labeled so as to be able to relate to the log book?	<u>  x  </u>	<u>      </u>
5. Are instrument maintenance logs used?	<u>  x  </u>	<u>      </u>
6. Is the GC/MS properly tuned (at least once every 12 hours) with DFTPP?	<u>  x  </u>	<u>      </u>
7. a. Is there an initial standard calibration curve?	<u>  x  </u>	<u>      </u>
b. Are there continuing calibration standards analyzed at an acceptable frequency (at least every 12 hours)?	<u>  x  </u>	<u>      </u>
c. Do analysts monitor requirements for %RSD for RFs and for minimum RRFs?	<u>  x  </u>	<u>      </u>
d. Is there documentation of all calibrations?	<u>  x  </u>	<u>      </u>

e. Is the SOP clear as to what constitutes acceptable calibrations and for appropriate corrective actions when needed?   x       

8. Does the SOP clearly define acceptable blanks and note corrective actions, if needed?   x       

If there are trace blank contaminants in the samples and blanks how are they accounted for in the final reported data? Sample results qualified in final data report.

9. Are efforts made to identify non-target analytes?   x       

10. Are all raw data maintained, either on hard copy or magnetic tape?   x       

11. Are internal standards/surrogates used to monitor instrument/ analysis performance?   x       

If so, do the analysts perform and document corrective actions when the criteria are exceeded?   x       

12. Is there any secondary review of all documents/data by any person other than the one generating the information?   x       

13. Is there a system of data qualifiers (or other means) to denote reported data that have not met all QC criteria, ie., holding times exceeded, surrogates out, internal standards, out, etc.?   x       

#### SECTION V. - COMMENTS:

- CLP samples batched/analyzed separately from 8270 samples.



## SECTION VI. - Pests/PCBs - continued

- |  | YES      | NO  |
|--|----------|-----|
| 7. Are retention time windows used for qualitative decisions?  | <u>x</u> | ___ |
| 8. Is corrective action required when a continuing calibration standard falls outside these established windows?   | <u>x</u> | ___ |
| 9. Are calibration factors used to monitor quantitative stability of the standards?  | <u>x</u> | ___ |
| 10. Are analysts sensitive to chromatography characteristics (peak shape, resolution, etc.)  | <u>x</u> | ___ |
| 11. Is there a surrogate compound used?<br>If so, please list <u>DBC, TCX, BCB</u>   | <u>x</u> | ___ |
| Any criteria/action limits for the surrogate?<br>-Updated annually   | <u>x</u> | ___ |
| 12. a. Are at least 2 columns used for qualitative analysis?   | <u>x</u> | ___ |
| b. Do the analysts use any quantitative criteria to aid in making qualitative decisions (ie., the difference in quantitation between the primary and confirmatory columns)?                        | <u>x</u> | ___ |
| 13. Does the SOP clearly define acceptable blanks and note corrective actions, if needed?  | <u>x</u> | ___ |
| If there are trace blank contaminants in the samples and blanks how are they accounted for in the final reported data? <u>If target analyte detected in blank samples samples are reextracted.</u> |          |     |
| 14. Is there any secondary review of all documents/data by any person other than the one generating the information?   | <u>x</u> | ___ |
| 15. Is there a system of data qualifiers (or other means) to denote reported data that have not met all QC criteria, ie., holding times exceeded, surrogate out, internal standards out, etc.      | <u>x</u> | ___ |

SECTION VII. SAMPLE PREPARATION- INORGANIC

YES NO

Is a designated person in charge of metals prep? x     Name Daphne BryantEducation/Experience B.S. Biology/ 12 years1. Is sample prep area free of dust and other sources of contamination (metal containers, spatulas, etc)? x     2. Is metal free water available for cleaning glassware , preparing standards, dilutions, etc.? x       
SOURCE Continental Deionization System3. Is water purification system properly monitored? x       
How? pH and resistance monitored4. Does SOP contain glassware cleaning instructions? x       
Is SOP posted in wash area? x     5. Is acid rinse used for glassware? x       
Is metals prep glassware kept segregated? x     6. Is sample volume appropriate to sample matrix? x       
Water 100 ml      Final volume 100 ml (Method 3010)  
Soil 1 g      Final volume 100 ml (Method 3050)Is suitable volumetric glassware used to transfer sample to beaker? Type 100 grad cylinder x     7. Are appropriate acids added to samples for digestion? x       
Acid supplier/grade Baker Trace Metal Grade  
Acid type/volume    ICP      AAS      GFAAS  
HCl                    x                      
HNO3                  x                x8. Are samples evaporated gently, not allowed to boil? x       
Mercury samples digested @ 95 deg. in water bath? x     9. Are samples filtered after digestion, or settled/ decanted after digestion? Decanted x     10. Are digestates stored in proper containers? x       
Plastic x Glass      Reusable      Disposable     11. Are stock standards adequate? x       
Purchased x Prepared in-house      Conc.     Are purchased stock standards certified? x     Bottles dated when prepared/received? x

## SECTION VII. - Inorganic prep - CONTINUED

YES NO

12. Are calibration standards prepared at correct freq.?  x  \_\_\_  
Daily  x  Weekly \_\_\_ Other  Monthly for ICP

Is logbook or record of standard prep maintained?  x  \_\_\_  
 Thorough logbook on standard preparation.

13. Do calibration standards & samples have same acid concentration?  x  \_\_\_

14. Are method blanks, matrix spikes, matrix duplicates digested concurrently with sample batch?  x  \_\_\_

SECTION VII. - COMMENTS:

## SECTION VIII. - METALS ANALYSIS

YES NO

Is a designated person in charge of ICP analysis?   x       Name   Ernie Walton   Title   Department Mgr  Education/Experience   MS Chemistry/11 yrs  Is a designated person in charge of AAS/GFAAS analysis?   x       Name   same   Title                     Education/Experience                     Is a designated person in charge of cold vapor/hydride analysis?   x       Name   Daphne Bryant   Title                                     Education/Experience                                     A. ICP1. Are ICP metals analyzed according to EPA Method 200.7 or 6010?   x         
If not list method and/or general technique used:  
  

List instrumentation used for ICP analysis:

  (2) TJA simul ICP; 1 TJA Trace; 4 TJA GFAA  2. Are methods (SOPs) available to analysts in the instrumentation area?   x       3. Are run logs kept by the analysts?        x    
**Instrument output serves as run log**4. Are holding times monitored by the analysts?   x       5. Does the ICP provide hard copy output?   x       6. Is the ICP calibrated at the correct frequency?   x         
(Calibrate daily, 1/24 hrs, or each time instrument set up)

## SECTION VIII. - Metals Analysis - CONTINUED

	YES	NO
7. Are proper number of calibration standards used?	<u>  x  </u>	_____
8. Is proper initial cal. verification std. used? ( Certified and analyzed at correct frequency? ) <u>Uses ICV standard from a separate source than cal. stds.</u>	<u>  x  </u>	_____
9. Have interelement corrections for spectral inter- ferences been programmed into the ICP? Is manual correction required?	<u>  x  </u>	_____
How often are these IEC factors updated?		<u>  x  </u>
10. Is an interelement check standard (ICS) analyzed on a daily basis to check the IECs ? <u>Control limits are + PQL</u>	<u>  x  </u>	_____
11. Are continuing calibration check standards analyzed at the correct frequency?	<u>  x  </u>	_____
12. Is an auto-sampler used?	<u>  x  </u>	_____
13. Are multiple exposures used for analysis? <u>2 exposures per sample.</u>	<u>  x  </u>	_____
14. What is integration time for exposure? <u>10 sec /exposure</u>	<u>  x  </u>	_____
15. Is instrument's linear range determined? How? <u>Analyzes series of standards.</u>	<u>  x  </u>	_____
16. Sample diluted when linear range exceeded?	<u>  x  </u>	_____
<u>B. AAS/GFAAS (includes mercury cold vapor)</u>		
1. Are AAS/GFAAS metals analyzed according to 200 series or 7000 series EPA methods? If not, list method and general technique used: _____	<u>  x  </u>	_____
List instrumentation used for AAS/GFAAS analysis: <u>TJA &amp; PE GFAAS</u>		
2. Are methods (SOPs) available to analysts in the instrumentation area?	<u>  x  </u>	_____
3. Are run logs kept by the analysts?	<u>  x  </u>	_____

## SECTION VIII. - Metals Analysis - CONTINUED

YES NO

4. Are holding times monitored by the analyst?  
(esp. mercury - 28 days)   x
5. Does the AAS/GFAAS provide hard copy output?  
Analysts' initials, date, and time included  
on hard copy?   x         
  x
6. Are instruments calibrated with correct number of  
standards and at correct frequency? Is calibration  
range adequate and within instrument's linear range?   x
- Comments: 4 stds + blank for calib. Must have cc >.995  
Daily calibration.
7. Is calibration standard at or near MQL analyzed?   x
8. Are proper initial and continuing cal. verification  
standards used? ( check type & frequency )   x
9. Are L'vov platforms used in the furnace tubes?  
For Pb & Tl only   x
10. Are multiple burns used and averaged?   x
11. Are analytical spikes used for GFAAS?   x         
If yes, what control limits are used?  
If CLP protocol requested; MS/MSD analyzed 10% freq.
12. Are element specific instrument conditions recorded?   x
13. Are methods of standard addition ever used for  
analysis? When? On CLP or TCLP when req'd by method.   x
14. Dilutions performed when samples exceed calibration  
range?   x

## SECTION VIII. - COMMENTS:

-PQLs are verified by analyzing standards daily; MDLs  
determined annually.

SECTION IX. CYANIDE

- Not evaluated

SECTION X. TCLP EXTRACTION FOR METALS, EXTRACTABLES, AND PESTICIDES

- |   |              |              |
|---|--------------|--------------|
| 1. Does the laboratory have an appropriate rotary agitation apparatus (30 +/- 2 RPM required)?  | <u>  x  </u> | <u>    </u>  |
| 2. Are extraction vessels appropriate for intended analytes (no plastic for organics)?<br><u>Plastic containers used for organic extractions.</u>   | <u>    </u>  | <u>  x  </u> |
| 3. Is pH of extraction fluid checked after fluid preparation? (fluid #1 = 4.93; fluid #2 = 2.88)  | <u>  x  </u> | <u>    </u>  |
| 4. Has percent solids of sample been determined?  | <u>  x  </u> | <u>    </u>  |
| 5. Are samples containing less than 0.5 % not subjected to an extraction?   | <u>  x  </u> | <u>    </u>  |
| 6. Has lab determined if particle size reduction is required? (sample must pass thru 9.5 mm sieve)  | <u>  x  </u> | <u>    </u>  |
| 7. Is the sample evaluated to determine the correct extraction fluid?<br>NOTE: 5g of sample + 96.5 mL of DI water, stir for 5 min. If pH < 5.0 use fluid #1. If pH > 5.0 add 3.5 ml 1 N HCl, stir, heat to 50 deg C, hold for 10 min. Cool, if pH < 5.0 use fluid # 1, if pH > 5.0 use fluid # 2. | <u>  x  </u> | <u>    </u>  |
| 8. Is extraction fluid = 20 times the weight of sample used? (percent solids must be factored into this calculation)  | <u>  x  </u> | <u>    </u>  |
| 9. Is sample extracted for 18 +/-2 hours?   | <u>  x  </u> | <u>    </u>  |
| 10. Are glass fiber filters with pore sizes of 0.6-0.8 um used for extract filtration? <u>0.7 um filter</u>   | <u>  x  </u> | <u>    </u>  |
| 11. Are filters used in metals analysis acid washed? (1 N nitric w/ three DI water rinses)  | <u>  x  </u> | <u>    </u>  |
| 12. Are matrix spikes performed for each waste type?  | <u>  x  </u> | <u>    </u>  |
| 13. Are spike compounds added after extraction but before preservation?   | <u>  x  </u> | <u>    </u>  |
| 14. Are holding times as stated in the method, section 8.4 observed?  | <u>  x  </u> | <u>    </u>  |

SECTION X. - COMMENTS:

SECTION XI. TCLP ZERO HEADSPACE EXTRACTION (ZHE) FOR VOLATILES

**-Not evaluated**

## SECTION XII. - FILE AUDIT

	YES	NO
1. a. Does the file contain a record of sample receipt?	<u>  x  </u>	_____
b. Is the chain of custody/log book properly recorded to clearly indicate date of receipt and person receiving samples?	<u>  x  </u>	_____
2. a. Are the records of extraction/preparation in the file or available?	<u>  x  </u>	_____
b. Are the records of extraction/prep properly recorded with date of extraction, person performing the analysis, and the technique used?	<u>  x  </u>	_____
c. Are the records clear for wts./volumes extracted or digested, final volumes of sample, etc.	<u>  x  </u>	_____
d. If soil sediments were analyzed, is there a % moisture determination, properly documented and reported along with the data?	<u>  x  </u>	_____
3. a. Is the sample analysis (instrumental) information in the file or available?	<u>  x  </u>	_____
b. Are instrument run logs properly recorded and included for ALL sample analyses (be sure to note any reanalyses)?	<u>  x  </u>	_____
c. Is documentation of all calibrations available?	<u>  x  </u>	_____
Were calibrations properly performed?	<u>  x  </u>	_____
Were corrective actions, if indicated, properly performed?	<u>  x  </u>	_____
d. Were proper Quality Control analyses performed and is the data available for:		
- Blanks (1 per batch of extraction/prep)	<u>  x  </u>	_____
- Surrogates	<u>  x  </u>	_____
- Matrix Spikes (1 per 20 samples)	<u>  x  </u>	_____

## SECTION XII. - File Audit - CONTINUED

YES NO

- e. Are raw quan lists, chromatograms, strip charts, etc., available for both samples and standard?

x \_\_\_\_\_List any not available: N/A

- f. Are the raw data properly labeled so as to to be able to trace to the samples and other documentation (ie., sample #, dates, times, etc.)

x \_\_\_\_\_

4. If quality control measures were outside criteria, did the laboratory take appropriate measures?

x \_\_\_\_\_List criteria excursions and action taken:  
No excursions noted.

5. a. Does the final reported data have a narrative to describe any problems in the analyses?

x \_\_\_\_\_

- b. Are all significant problems indicated by the raw data discussed in the narrative?

x \_\_\_\_\_

6. Does a spot check of the raw data to the final reports reflect accurate transcription?

x \_\_\_\_\_

7. Do all dates and times follow a logical pattern?

x \_\_\_\_\_List discrepancies: None

**EXECUTIVE SUMMARY**  
**DATA AUDIT REPORT**  
**Compuchem Laboratories**  
**3306 Chapel Hill/Nelson Hwy.**  
**Research Triangle Park, NC 27709-4998**

**INTRODUCTION**

On July 25, 1995, a data audit was conducted at Compuchem Laboratories, Research Triangle Park, North Carolina. The evaluation was performed at the request of the Federal Facilities Branch, Waste Management Division, USEPA, Region IV, and was related to analytical services the laboratory provided for a RCRA Facility Investigation (RFI) at the US Navy's Charleston Naval Base. The evaluation was conducted by Gary Bennett of the Laboratory Evaluation/Quality Assurance Section (LEQAS), Region IV Environmental Services Division.

Compuchem holds both organic and inorganic analytical contracts in EPA's Contract Laboratory Program (CLP). CLP laboratories are subject to extensive oversight by EPA, including on-site audits by the EPA Region IV CLP Technical Project Officer (Mr. Tom Bennett), evaluation of laboratory results from the CLP quarterly blind performance evaluation program, and screening of the lab's CLP data packages for contractual compliance. Because of EPA Region IV's contacts with, and knowledge of Compuchem Environmental Corporation, the visit on July 25 was focused strictly on the documentation related to specific samples analyzed for the Naval Base Charleston RFI. The documentation provided by Compuchem was compared to the laboratory's Quality Assurance Plan (QAP) as provided in the Naval Base Charleston "Final Comprehensive Quality Assurance Manuals, Revision No:01", dated May 19, 1995.

The findings of this evaluation are based upon a review of the laboratory's data for 69 samples analyzed for volatile organic compounds, semivolatile organic compounds, pesticides/PCBs, and metals. These samples were received over a three week time period from March 14 to April 8, 1995 and were included in Compuchem's Sample Delivery Group Number 30215. Specific sample numbers for the data reviewed are on file with the LEQAS.

## **FINDINGS/RECOMMENDATIONS**

Details of the findings are contained in the attached laboratory evaluation guidelines checklist, Section XII. The data packages examined at Compuchem contained records of sample receipt, instrument calibration, instrument output and quality control checks. The packages were complete, comprehensive, and provided sufficient information to enable the auditor to recreate all sample results. Sample results were in accordance with the laboratory's QAP provided in Naval Base Charleston "Final Comprehensive Quality Assurance Manuals, Revision No:01". No recommendations were required based on the data reviewed at the laboratory.

LABORATORY EVALUATION AND QUALITY ASSURANCE SECTION  
U.S. EPA REGION IV  
ATHENS, GEORGIA

Lab Name: Compuchem Environmental Corporation  
Address: 3306 Chapel Hill/Nelson Hwy, Research Triangle Park,  
North Carolina 27709-4998  
Date of Evaluation: July 19, 1995

INTRODUCTION

- Purpose of Evaluation - The purpose of the lab audit is to assess the laboratory's overall practices and functions in performing analyses. This is accomplished by interviews with laboratory personnel and by personal observations of EPA, Region IV lab evaluators. If problem areas are identified, an effort will be initiated by the evaluators to furnish recommendations for improvement or correction, with the overall goal being to assure that the Agency receives data appropriate for the projects concerned. The product of the evaluation will be a report of all the information received. This evaluation process does not serve as a certification program and as such will not result in any "list" of approved laboratories.

- Scope of Evaluation - The evaluation will include, but is not limited to: information on personnel qualifications, facilities and equipment, sample custody, sample prep and analysis, data reduction, quality assurance/quality control, and records of all processes.

PERSONNEL CONDUCTING THE EVALUATION

Name/Organization Gary Bennett, LEQA

Organization Code:

LEQA = Laboratory Evaluation and Quality Assurance Section of  
USEPA, Region IV, Environmental Services Division

## SECTION XII. - FILE AUDIT

	YES	NO
1. a. Does the file contain a record of sample receipt?	<u>  x  </u>	_____
b. Is the chain of custody/log book properly recorded to clearly indicate date of receipt and person receiving samples? <u>For the samples reviewed, there was chain of custody form which was not signed and dated.</u>	<u>  x  </u>	_____
2. a. Are the records of extraction/preparation in the file or available?	<u>  x  </u>	_____
b. Are the records of extraction/prep properly recorded with date of extraction, person performing the analysis, and the technique used?	<u>  x  </u>	_____
c. Are the records clear for wts./volumes extracted or digested, final volumes of sample, etc.	<u>  x  </u>	_____
d. If soil sediments were analyzed, is there a % moisture determination, properly documented and reported along with the data?	<u>  x  </u>	_____
3. a. Is the sample analysis (instrumental) information in the file or available?	<u>  x  </u>	_____
b. Are instrument run logs properly recorded and included for ALL sample analyses (be sure to note any reanalyses)?	<u>  x  </u>	_____
c. Is documentation of all calibrations available?	<u>  x  </u>	_____
Were calibrations properly performed?	<u>  x  </u>	_____
Were corrective actions, if indicated, properly performed?	<u>  x  </u>	_____
d. Were proper Quality Control analyses performed and is the data available for:		
- Blanks (1 per batch of extraction/prep)	<u>  x  </u>	_____
- Surrogates	<u>  x  </u>	_____
- Matrix Spikes (1 per 20 samples)	<u>  x  </u>	_____

## SECTION XII. - File Audit - CONTINUED

YES NO

- e. Are raw quan lists, chromatograms, strip charts, etc., available for both samples and standard?   x

List any not available:   N/A  

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- f. Are the raw data properly labeled so as to to be able to trace to the samples and other documentation (ie., sample #, dates, times, etc.)   x

4. If quality control measures were outside criteria, did the laboratory take appropriate measures?   x

List criteria excursions and action taken:  
For the pesticide analyses, there was one surrogate outside control limits. This was noted in the final data report.

5. a. Does the final reported data have a narrative to describe any problems in the analyses?   x

- b. Are all significant problems indicated by the raw data discussed in the narrative?   x

6. Does a spot check of the raw data to the final reports reflect accurate transcription?   x

7. Do all dates and times follow a logical pattern?   x

List discrepancies:   None  

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Report of an On-Site Evaluation of  
**Pace Environmental Laboratories**  
**New England - NH Laboratory**

August 15, 1995

by

**John McConney**

**U.S. Environmental Protection Agency**  
**Environmental Services Division**  
**960 College Station Road**  
**Athens, Georgia 30613**

## **INTRODUCTION**

On July 27, 1995, an on-site laboratory evaluation was conducted at the Pace Environmental Laboratory, New England-NH, Hampton, NH. The on-site evaluation was conducted by John McConney of the U.S. EPA Region IV Environmental Services Division at the request of the U.S. EPA Region IV Waste Management Division, Federal Facilities Branch.

The purpose of the evaluation was to assess the laboratory's capability to perform volatile, semivolatile and pesticide/PCBs analyses. The findings of this evaluation are based upon information provided by the laboratory as well as the personal observations of the evaluator.

## **GENERAL INFORMATION**

The Pace Environmental Laboratory has been in operation since 1983 and employs 53 personnel. The laboratory had a Standard Operating Procedure (SOP) manual in place. Copies of relevant portions of the SOP were kept throughout the laboratory. The laboratory has a Quality Assurance (QA) Plan in place.

## **FACILITIES, EQUIPMENT AND PERSONNEL**

Laboratory space was adequate to house personnel and equipment. All personnel interviewed were cooperative and appeared to be competent in their areas of expertise. The laboratory contained all the instrumentation and equipment needed to perform the analyses evaluated.

## **FINDINGS/RECOMMENDATIONS**

Details of all findings are contained in the attached laboratory evaluation guidelines checklist. The following is a summary of the most notable findings of this evaluation, and when appropriate, a recommendation for improvement or correction of any deficiencies noted.

### Section I. Sample Receipt and Storage Area

**OBSERVATION:** The laboratory is currently checking samples for chemical preservation by using a disposable pipette to obtain an aliquot of the sample.

**RECOMMENDATION:** The laboratory should pour a small amount of the sample into another container and test this aliquot with pH paper. This will eliminate the possibility of contamination being introduced into the sample.

SECTION II. SAMPLE PREPARATION - ORGANIC ANALYSIS

OBSERVATION: The laboratory does not monitor the effectiveness of the water purification system.

**RECOMMENDATION: The laboratory should institute a regular series of check blanks to monitor the water purification system. This results of these check blanks should be kept on file to demonstrate that the water purification system is operating correctly.**

OBSERVATION: During the audit it was noted that the laboratory extraction SOP had extensive hand-written notes in the margin. Laboratory personnel explained that the SOP was in the process of revision.

**RECOMMENDATION: The laboratory should complete the revision as soon as possible so that the SOP will be complete and up to date.**

SECTION IV: VOLATILES ANALYSIS (VOAs)

OBSERVATION: The laboratory is not noting the injection time in the analysis run log.

**RECOMMENDATION: The laboratory should note the injection time in the analysis run log.**

SECTION V: SEMIVOLATILES (SVs)

OBSERVATION: The laboratory is not noting the injection time in the analysis run log.

**RECOMMENDATION: The laboratory should note the injection time in the analysis run log.**

SECTION VI: PESTICIDES/PCBs (PEST/PCB)

OBSERVATION: The laboratory is not noting the injection time in the analysis run log.

**RECOMMENDATION: The laboratory should note the injection time in the analysis run log.**

SECTION VII: FILE AUDIT

OBSERVATION: The QA Manual specifies a QC Limit of 40 percent difference for the semivolatile calibration check compounds. However, the method specifies a QC Limit of 30 percent

difference.

**RECOMMENDATION:** The laboratory should revise the QA Manual to be in accordance with the method requirements.

**OBSERVATION:** The SOP for the non-CLP data review specifies the data review criteria but does not specify the process.

**RECOMMENDATION:** The SOP for the non-CLP data review should be revised to include the data review process in addition to the criteria.

### **CONCLUSIONS**

The analyses performed by the Pace Environmental Laboratory were for the most part in accordance with the methodology requirements. Though there is room for improvement, the deficiencies noted above do not seriously impact the overall data quality. The laboratory appeared to be providing reliable data for the required analyses.

LABORATORY EVALUATION GUIDELINES  
LABORATORY EVALUATION AND QUALITY ASSURANCE SECTION  
U.S. EPA REGION IV  
ATHENS, GEORGIA  
Organic Analyses

Lab Name: Pace Environmental Laboratories  
Address: 1 Lafayette Road, P.O. Box 2130, Hampton, NH 03843

INTRODUCTION

- Purpose of Evaluation - The purpose of the lab audit is to assess the laboratory's overall practices and functions in performing analytical analyses. This is accomplished by interviews with laboratory personnel and by personal observations of EPA, Region IV lab evaluators. If problem areas are identified, an effort will be initiated by the evaluators to furnish recommendations for improvement or correction, with the overall goal being to assure that the Agency receives data appropriate for the projects concerned. The product of the evaluation will be a report of all the information received. This evaluation process does not serve as a certification program and as such will not result in any "list" of approved laboratories.

- Scope of Evaluation - The evaluation will include, but is not limited to: information on personnel qualifications, facilities and equipment, sample custody, sample prep and analysis, data reduction, quality assurance/quality control, and records of all processes.

PERSONNEL CONDUCTING THE EVALUATION

Name/Organization John P. McConney, LEQA

Name/Organization \_\_\_\_\_

Name/Organization \_\_\_\_\_

Name/Organization \_\_\_\_\_

Organization Codes:

LEQA = Laboratory Evaluation and Quality Assurance Section of  
USEPA, Region IV, Environmental Services Division  
OCS = Organic Chemistry Section of the USEPA, Region IV,  
Environmental Services Division  
ICS = Inorganic Chemistry Section of the USEPA, Region IV,  
Environmental Services Division



SECTION I. SAMPLE RECEIPT AND STORAGE AREA

	YES	NO
Is there a person designated for receipt and storage responsibility?	<u>X</u>	___
Name <u>Kathy Lawson</u> Title <u>Sample Custodian</u>		
Education <u>BA</u> Experience <u>5 yrs</u>		
1. Is there a sample receipt section in the SOP? If so, is it available in the sample receipt area?	<u>X</u> <u>X</u>	___ ___
2. Is sample custody maintained and documented?	<u>X</u>	___
3. a. Is a sample log maintained? b. Are unique numbers assigned to each sample?	<u>X</u> <u>X</u>	___ ___
4. a. Is sample container integrity verified? b. Are samples monitored for correct preservation? c. Is sample temperature checked?	<u>X</u> <u>X</u> <u>X</u>	___ ___ ___
5. Are sample labels and chain of custody records cross checked?	<u>X</u>	___
6. a. Are adequate facilities for sample storage available? b. Are volatiles kept separate from others? c. Are cold storage temperatures routinely checked and recorded? d. Are temperature excursions noted, with appropriate action taken?	<u>X</u> <u>X</u> <u>X</u> <u>X</u>	___ ___ ___ ___
7. Are all sample receipt procedures/documents consistent with the SOP? Comments:	<u>X</u>	___
8. Is sample age monitored and communicated to analysts?	<u>X</u>	___

- |     |   |             |             |
|-----|---|-------------|-------------|
| 9.  | Are sample containers provided to clients?                          | <u>X</u>    | <u>    </u> |
|     | If so,  |             |             |
| a.  | Are these sample containers cleaned by the laboratory?              | <u>    </u> | <u>X</u>    |
| b.  | Is the cleaning protocol comparable to Region IV protocol?          |             | NA          |
| c.  | Are there QC checks in place?                                       | <u>X</u>    | <u>    </u> |
| d.  | Are these sample containers obtained from a commercial vendor?      | <u>X</u>    | <u>    </u> |
| e.  | Does this vendor have QC checks?                                    | <u>X</u>    | <u>    </u> |
| f.  | Are preservatives added to the sample containers prior to shipping? | <u>    </u> | <u>X</u>    |
| g.  | Are trip blanks placed in the coolers?                              | <u>X</u>    | <u>    </u> |
| h.  | Temperature blank placed in the coolers?                            | <u>X</u>    | <u>    </u> |
| 10. | Does the laboratory clean sampling equipment for clients?           | <u>    </u> | <u>X</u>    |
|     | If so, is the cleaning protocol comparable to Region IV protocol?   |             | NA          |

## SECTION I. - COMMENTS:

The laboratory is checking the samples for chemical preservation by using a pipette to transfer an aliquot of the sample to another container. The SOP is vague about the procedure to be used for checking sample pH. The SOP reads "Check all water samples with pH paper to see whether samples have been preserved."

## SECTION II. SAMPLE PREPARATION - ORGANICS ANALYSIS

	YES	NO
Is a person designated in charge of extractions?	<u>X</u>	___
Name <u>Jim Holst</u> Title <u>Extraction Supervisor</u>		
Education <u>BS</u> Experience <u>3.5 yrs</u>		
1. a. Is sample age noted by extraction personnel?	<u>X</u>	___
b. Are samples outside recommended holding times noted?	<u>X</u>	___
2. Is hood space adequate for the prep area?	<u>X</u>	___
3. Are separate areas used for standard prep and sample prep?	___	<u>X</u>
4. Are glassware clean-up procedures documented and available to prep analyst?	<u>X</u>	___
5. a. What type of water purification system is used for the organics? <u>Mil Q Plus</u>		
b. Is the system properly maintained and monitored for contamination?(i.e. check blanks)	___	<u>X</u>
6. Are solvent storage areas located in such a way as to prevent possible contamination?	<u>X</u>	___
7. Is prep for volatiles separate from other organic extractions?	___	___
8. If problems with blanks occur, is the extraction lab informed?	<u>X</u>	___
How is this information communicated?		
<u>daily meetings</u>		
<u>corrective action reports</u>		
9. a. Are extraction log books maintained?	<u>X</u>	___
b. Are details(bench notes,etc) kept in the book?	<u>X</u>	___
10. a. Are analytical balances available and checked by qualified personnel periodically?	<u>X</u>	___
b. Are balances checked using Class S weights at least once per month?	<u>X</u>	___
Are results documented?	<u>X</u>	___

- |   |          |     |
|---|----------|-----|
| 11. Are extracts properly stored in refrigerated areas?                     | <u>X</u> | ___ |
| 12. Are extracts stored separately from standards?                          | <u>X</u> | ___ |
| 13. Is the appropriate extraction section of the SOP available to analysts? | <u>X</u> | ___ |
| 14. Do analysts monitor and maintain lot numbers of:                        |          |     |
| a. solvents   | <u>X</u> | ___ |
| b. spiking solutions  | <u>X</u> | ___ |
| c. clean-up adsorbents (alumina, florisil, etc)                             | <u>X</u> | ___ |

SECTION II. - COMMENTS:



## SECTION IV. - VOLATILES ANALYSIS (VOAs)

YES NO

Is a person designated in charge of VOA analyses?  
 Person Interviewed:

\_\_\_ X

Name Angie Richard Title GC/MS Analyst

Education BA Experience 2 yrs

1. Are VOAs analyzed by EPA methods/GC/MS?  
 By method: RCRA 8240

X \_\_\_

If not, list method and general technique used:

\_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

2. List instrumentation used for VOA analysis:  
4 HP 5970

\_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

Is this instrumentation dedicated to VOAs?

X \_\_\_

3. Are methods (SOPs) available to analysts in the  
 instrumentation area?

X \_\_\_

4. Is the VOA analysis area segregated from other  
 sections of the laboratory?

\_\_\_ X

5. a. Are analysis log books used and kept at the  
 instrument?

X \_\_\_

b. Do they include all injections/analyses made  
 on the instrument?

X \_\_\_

c. Are holding times monitored by analysts?

X \_\_\_

d. Does the record include:

- analyst(s) name
- date of analysis
- time of analysis

X \_\_\_

X \_\_\_

\_\_\_ X

e. Are the raw data (chromatograms,.etc) labeled  
 so as to be able to relate to the log book?

X \_\_\_

## SECTION IV. - CONTINUED

	YES	NO
6. Are instrument maintenance logs used?	<u>X</u>	___
7. Is the GC/MS properly tuned (at least every 12 hours) with BFB?	<u>X</u>	___
8. a. Is there an initial standard calibration curve?	<u>X</u>	___
b. Are there continuing calibration standards analyzed at an acceptable frequency (at least every 12 hours)?	<u>X</u>	___
c. Do the analysts monitor requirements for %RSD for the RRFs and for minimum average RRFs?	<u>X</u>	___
d. Is there documentation of all these calibrations?	<u>X</u>	___
e. Is the SOP clear as to what constitutes acceptable calibrations and for appropriate corrective actions, when needed?	<u>X</u>	___
9. Does the SOP clearly define acceptable blanks and note corrective actions, if needed?	<u>X</u>	___
<p>If there are trace blank contaminants that are also in samples, how are they accounted for in the final reported data? <u>the laboratory assigns data qualifiers during secondary data review</u></p>		
10. Are efforts made to identify non-target analytes? <u>done only on client request</u>	<u>X</u>	___
11. Are all raw data maintained, either on hard copy or magnetic tape?	<u>X</u>	___
12. Does the lab have the necessary equipment to do low level soil work, i.e., heated purge and trap?	<u>X</u>	___
If so, is there a separate calibration for the heated analysis?	<u>X</u>	___
13. Are internal standards/surrogates used to monitor instrument/analysis performance?	<u>X</u>	___
If so, do the analysts perform and document corrective action when the criteria are exceeded?	<u>X</u>	___

14. Is there any secondary review of all documents/data by any person other than the one generating the information? X \_\_\_\_\_
15. Is there a system of data qualifiers (or other means) to denote reported data that have not met all QC criteria, i.e., holding times exceeded, surrogates out, internal standards out, etc.? X \_\_\_\_\_
16. Are matrix spike/matrix spike duplicate samples analyzed? X \_\_\_\_\_  
What is the frequency? per batch  
What are the QC limits? per method requirements

SECTION IV. - COMMENTS:

## SECTION V. - SEMIVOLATILES (SVs)

	YES	NO
Is a person designated in charge of SVs?	<u>X</u>	___
Name <u>Liane Hall</u> Title <u>Senior Analyst</u>		
Education <u>MS</u> Experience <u>7 yrs</u>		
1. Are SVs analyzed by EPA methods (GC/MS)? By method: <u>RCRA 8270</u>	<u>X</u>	___
If not, list method and briefly describe technique: _____ _____ _____ _____		
2. List instrumentation used for SV analysis: <u>2 HP 5870</u> _____ _____ _____		
3. Are methods (SOPs) available to analysts in the instrumentation area?	<u>X</u>	___
4. a. Are analysis log books used and kept at the instrument?	<u>X</u>	___
b. Do they include all injections made on the instrument?	<u>X</u>	___
c. Are holding times monitored by analysts?	<u>X</u>	___
d Does the record include:		
- analyst(s) name	<u>X</u>	___
- date of analysis	<u>X</u>	___
- time of analysis	___	<u>X</u>
e. Are the raw data (chromatograms, etc) labeled so as to be able to relate to the log book?	<u>X</u>	___
5. Are instrument maintenance logs used?	<u>X</u>	___
6. Is the GC/MS properly tuned (at least once every 12 hours) with DFTPP?	<u>X</u>	___

SECTION V. - COMMENTS:



## SECTION VI. - Pests/PCBs - continued

- |  | YES      | NO  |
|--|----------|-----|
| 7. Are retention time windows used for qualitative decisions?  | <u>X</u> | ___ |
| 8. Is corrective action required when a continuing calibration standard falls outside these established windows?   | <u>X</u> | ___ |
| 9. Are calibration factors used to monitor quantitative stability of the standards?  | <u>X</u> | ___ |
| 10. Are analysts sensitive to chromatography characteristics (peak shape, resolution, etc.)  | <u>X</u> | ___ |
| 11. Is there a surrogate compound used?<br>If so, please list <u>as per method</u>   | <u>X</u> | ___ |
| Any criteria/action limits for the surrogate?  | <u>X</u> | ___ |
| 12. Are at least 2 columns used for qualitative analysis?  | <u>X</u> | ___ |
| 13. Does the SOP clearly define acceptable blanks and note corrective actions, if needed?  | <u>X</u> | ___ |
| If there are trace blank contaminants in the samples and blanks how are they accounted for in the final reported data? <u>trace contaminants are not allowed</u>                               |          |     |
| _____  |          |     |
| _____  |          |     |
| 14. Is there any secondary review of all documents/data by any person other than the one generating the information?   | <u>X</u> | ___ |
| 15. Is there a system of data qualifiers (or other means) to denote reported data that have not met all QC criteria, i.e., holding times exceeded, surrogate out, internal standards out, etc. | <u>X</u> | ___ |
| 16. Are matrix spike/matrix spike duplicate samples analyzed?<br>What is the frequency? <u>per batch</u><br>What are the QC limits? <u>per method requirements</u>                             | <u>X</u> | ___ |

SECTION VI - COMMENTS:

## SECTION VII. - FILE AUDIT

	YES	NO
1. a. Does the file contain a record of sample receipt?	<u>X</u>	___
b. Is the chain of custody/log book properly recorded to clearly indicate date of receipt and person receiving samples?	<u>X</u>	___
2. a. Are the records of extraction/preparation in the file or available?		<u>available</u>
b. Are the records of extraction/prep properly recorded with date of extraction, person performing the analysis, and the technique used? <u>* but not in project file</u>	<u>X</u> *	___
c. Are the records clear for wts./volumes extracted or digested, final volumes of sample, etc.	<u>X</u>	___
d. If soil sediments were analyzed, is there a % moisture determination, properly documented and reported along with the data?		NA
3. a. Is the sample analysis (instrumental) information in the file or available?	<u>X</u>	___
b. Are instrument run logs properly recorded and included for ALL sample analyses (be sure to note any reanalyses)?	<u>X</u>	___
c. Is documentation of all calibrations available?	<u>X</u>	___
Were calibrations properly performed?		<u>apparently</u>
Were corrective actions, if indicated, properly performed?		NA
d. Were proper Quality Control analyses performed and is the data available for:		
- Blanks (1 per batch of extraction/prep)	<u>X</u>	___
- Surrogates	<u>X</u>	___
- Matrix Spikes (1 per 20 samples) <u>used blank spikes</u>	<u>X</u>	___

## SECTION XII. - File Audit - CONTINUED

YES NO

- e. Are raw quan lists, chromatograms, strip charts, etc., available for both samples and standard?  X

List any not available: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

- f. Are the raw data properly labeled so as to to be able to trace to the samples and other documentation (i.e., sample #, dates, times, etc.)  X

4. If quality control measures were outside criteria, did the laboratory take appropriate measures? NA

List criteria excursions and action taken:  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

5. a. Does the final reported data have a narrative to describe any problems in the analyses?  X

- b. Are all significant problems indicated by the raw data discussed in the narrative?  X

6. Does a spot check of the raw data to the final reports reflect accurate transcription?  X

7. Do all dates and times follow a logical pattern?  X

List discrepancies: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

SECTION XII. - COMMENTS:

Report of an On-Site Evaluation of

**Pace Environmental Laboratories  
Indianapolis, IN**

August 10, 1995

by

**John McConney**

U.S. Environmental Protection Agency  
Environmental Services Division  
960 College Station Road  
Athens, Georgia 30613

## INTRODUCTION

On July 25, 1995, an on-site laboratory evaluation was conducted at the Pace Environmental Laboratory, Indianapolis, Indiana. The on-site evaluation was conducted by John McConney of the U.S. EPA Region IV Environmental Services Division at the request of the U.S. EPA Region IV Waste Management Division, Federal Facilities Branch.

The purpose of the evaluation was to assess the laboratory's capability to perform polychlorinated dibenzo-p-dioxin and polychlorinated dibenzo-p-furan high resolution analyses. The findings of this evaluation are based upon information provided by the laboratory as well as the personal observations of the evaluator.

## GENERAL INFORMATION

The Pace Environmental Laboratory employs 25 personnel, five of which are directly involved with dioxins/furans extraction and analyses. The laboratory had a Standard Operating Procedure (SOP) manual in place. Copies of relevant portions of the SOP were kept throughout the laboratory. The laboratory has a Quality Assurance (QA) Plan in place.

## FACILITIES, EQUIPMENT AND PERSONNEL

Laboratory space was adequate to house personnel and equipment. All personnel interviewed were cooperative and appeared to be competent in their areas of expertise. The laboratory contained all the instrumentation and equipment needed to perform the analyses evaluated.

## FINDINGS/RECOMMENDATIONS

Details of all findings are contained in the attached laboratory evaluation guidelines checklist. The following is a summary of the most notable findings of this evaluation, and when appropriate, a recommendation for improvement or correction of any deficiencies noted. Some of these observations are based on a review of file data; the laboratory has already taken steps to correct some of the deficiencies that are noted below.

### Section I. Sample Receipt and Storage Area

**OBSERVATION:** The laboratory did not have a fume hood in the sample receipt area.

**RECOMMENDATION:** The laboratory should install a fume hood in the sample receipt area. This will allow the laboratory to open

**RECOMMENDATION:** The laboratory should analyze a complete GC Column Performance Check Solution at the beginning of each 12-hour shift to verify the switching times. The SICPs from the GC check solution analysis should be included in the data package.

**OBSERVATION:** The laboratory is using an incorrect quality control (QC) limit for the matrix spike/matrix spike duplicate (MS/MSD). The laboratory is using 25 percent as the QC limit for the agreement between the MS and MSD. According to the specifications of Method 8290, paragraph 8.3.6.4, the QC limit for the agreement is 20 percent.

**RECOMMENDATION:** The laboratory should use 20 percent as the QC limit for the agreement between the MS and MSD.

**OBSERVATION:** The documentation provided by the laboratory does not meet the specifications of Method 8290, paragraph 8.2.1.2, which require the first and last eluters in the GC Column Performance Check Solution to be labeled on the SICP.

**RECOMMENDATION:** The laboratory should label the first and last eluters in the GC Column Performance Check Solution on the SICP as appropriate.

**OBSERVATION:** The laboratory is not providing the SICPs of the lock mass ions.

**RECOMMENDATION:** While not required in Method 8290, the laboratory should provide the SICPs of the monitored lock mass ions.

## **CONCLUSIONS**

The analyses performed by the Pace Environmental Laboratory were for the most part in accordance with the methodology requirements. Though there is room for improvement, the deficiencies noted above do not seriously impact the overall data quality. The laboratory appeared to be providing reliable data for the required analyses.

LABORATORY EVALUATION GUIDELINES  
LABORATORY EVALUATION AND QUALITY ASSURANCE SECTION  
U.S. EPA REGION IV  
ATHENS, GEORGIA  
PCDD/PCDF Analyses

Lab Name: Pace - Indianapolis, IN  
Address: 7726 Moller Road, Indianapolis, IN 46268

INTRODUCTION

- Purpose of Evaluation - The purpose of the lab audit is to assess the laboratory's overall practices and functions in performing analytical analyses. This is accomplished by interviews with laboratory personnel and by personal observations of EPA, Region IV lab evaluators. If problem areas are identified, an effort will be initiated by the evaluators to furnish recommendations for improvement or correction, with the overall goal being to assure that the Agency receives data appropriate for the projects concerned. The product of the evaluation will be a report of all the information received. This evaluation process does not serve as a certification program and as such will not result in any "list" of approved laboratories.

- Scope of Evaluation - The evaluation will include, but is not limited to: information on personnel qualifications, facilities and equipment, sample custody, sample prep and analysis, data reduction, quality assurance/quality control, and records of all processes.

PERSONNEL CONDUCTING THE EVALUATION

Name/Organization John P. McConney

Name/Organization \_\_\_\_\_

Name/Organization \_\_\_\_\_

Name/Organization \_\_\_\_\_

Organization Codes: x

LEQA = Laboratory Evaluation and Quality Assurance Section of USEPA, Region IV, Environmental Services Division  
OCS = Organic Chemistry Section of the USEPA, Region IV, Environmental Services Division  
ICS = Inorganic Chemistry Section of the USEPA, Region IV, Environmental Services Division

GENERAL INFORMATIONHow long has the laboratory been in operation? 10-11 yrs

Name of Laboratory Manager

Stephen A. Barnett

Name of Quality Assurance Coordinator (education/experience) \_\_\_\_\_

Julia Tillman, BS, 3 yrs QA/QC with 8.5 yrs total

Does QA Coordinator report directly to senior management?

X yes \_\_\_ noTotal number of personnel 25 (Obtain organizational chart and employee list if possible)Total Technical Personnel 15Total Clerical Personnel 10PCDD extraction Personnel 2PCDD HRGC/HRMS analysis Personnel 2Total area of facility 13000 sq. ft.Laboratory area 7300 sq. ft.Offices area 5700 sq. ft.Is there a laboratory information management (LIMS) system? X yes \_\_\_ noComments: LIMS handles mostly sample receipt and billing; spreadsheets are also used

Is there a health and safety training program in place?

X yes \_\_\_ no

Comments:

Quality Assurance Plan/Standard Operating ProceduresIs there a written Standard Operating Procedure for all laboratory operations? X yes \_\_\_ noDate of initial preparation varied by sectionAny revisions? revised annuallyAuthor(s)? Julia Tillman, et.al.

Comments:

Is there a separate written Quality Assurance Manual for all  
Laboratory operations?  yes  no

Date of latest revision 1/95

Comments:

The laboratory is certified for High Resolution PCDD analyses by Region V and by the Army Corp. Reportedly, audits were included as part of the certification process.

SECTION I. SAMPLE RECEIPT AND STORAGE AREA

YES NO

Sample Custodian

Name Kim Vannoy Title Sample CustodianEducation BS Experience 1 yr

1. How is sample custody maintained and documented?  
Chain-of-Custody (COC) is checked in and internal  
paperwork is generated - the LIMS is used for this
- 
2. Is there a sample receipt section in the SOP? X \_\_\_  
 If so, is it available in the sample receipt area? X \_\_\_
3. a. Is a sample log maintained? X \_\_\_  
 b. Are unique numbers assigned to each sample? X \_\_\_
4. a. Is sample container integrity verified? X \_\_\_  
 b. Is sample temperature checked on receipt? X \_\_\_
- How are samples that are improperly preserved  
 and/or received in inappropriate containers  
 handled?  
condition is noted on form and project  
manager is notified - he notifies client  
if necessary
5. Are sample labels and chain of custody records  
 cross checked? X \_\_\_  
 Is there secondary review of logbooks? X \_\_\_
6. a. Are adequate facilities for sample storage  
 available? X \_\_\_  
 b. Are there segregated storage areas for dioxin  
 samples? X \_\_\_  
 c. Are cold storage temperatures checked daily  
 and recorded (2 to 6°C) ? X \_\_\_  
 d. Are fish and adipose samples stored separately  
 with maximum temperature of less than -20°C? \_\_\_ X  
 e. Are temperature excursions noted, with  
 appropriate action taken? X \_\_\_  
 f. Are samples stored in the dark? X \_\_\_
7. Are all sample receipt procedures/documents  
 consistent with the SOP? X \_\_\_
8. Is sample age monitored and communicated to  
 analysts? X \_\_\_



## SECTION II. SAMPLE PREPARATION

YES NO

Extraction Supervisor

Name Neil Stock Title Group LeaderEducation BS Experience 4 yrs

Other extraction personnel

Name Steven EdwardsEducation BS Experience 2.5 yrsName Michele JacksonEducation BS Experience < 1 yr

Name \_\_\_\_\_

Education \_\_\_\_\_ Experience \_\_\_\_\_

Name \_\_\_\_\_

Education \_\_\_\_\_ Experience \_\_\_\_\_

What matrices are extracted at the facility?

water, soil/sediment, fish, ash, air, still bottom,  
sludge, but not human adipose tissue

Is the laboratory maintained in a clean and organized manner?

mostly clean and well-organized

1. a. Is sample age noted by extraction personnel? X \_\_\_\_\_  
 b. Are samples outside recommended holding times noted? X \_\_\_\_\_
2. Is hood space adequate for the prep area? X \_\_\_\_\_  
 Is the flow periodically checked and recorded? X \_\_\_\_\_
3. Is there is an area devoted to dioxin extraction? X \_\_\_\_\_  
 Does the extraction area have adequate workspace? X \_\_\_\_\_  
 Are the laboratory benches made of impervious materials? X \_\_\_\_\_
4. Is glassware reused? X \_\_\_\_\_  
 Are glassware clean-up procedures documented and available to prep analyst? X \_\_\_\_\_  
 (rinsed with last solvent, washed and rinsed with water, rinsed with acetone and hexane, capped and stored covered)  
the laboratory is using toluene in place of hexane
5. a. What type of water purification system is used at the laboratory? millipore system - reverse osmosis with three scrubbing beds

- b. Is the system properly maintained and monitored for contamination? (i.e. check blanks) daily X \_\_\_
- c. Are the results of check blanks maintained on file? X \_\_\_
6. Are solvent storage areas vented or located in such a way as to prevent possible laboratory contamination? X \_\_\_
7. Are fish samples blended at the laboratory? X \_\_\_  
SS meat grinder with 3-5 mm hole size inner plate? X \_\_\_
8. If problems with blanks occur, is the extraction lab informed? X \_\_\_
- How is this information communicated?  
use regular check forms that are given to  
and kept in extraction laboratory
- 
9. a. Are extraction log books maintained? X \_\_\_  
b. Are details (bench notes, etc) kept in the book? X \_\_\_  
c. Are spiking solution lot numbers recorded? X \_\_\_
10. a. Are analytical balances located away from drafts and areas subject to temperature changes? See Note 1  
b. Are balances checked using Class S weights at least once per month? X \_\_\_  
Are results documented? X \_\_\_
11. Are extracts properly stored in sealed vials in the dark at ambient temperature prior to analysis? X \_\_\_
12. Is the appropriate dioxin extraction section of the SOP available to analysts? X \_\_\_
13. Do analysts monitor and maintain lot numbers of:  
a. solvents See Note 2  
b. clean-up adsorbents (alumina, silica, carbon) X \_\_\_  
c. Are these used in a first-in, first-out basis? X \_\_\_
14. Are the purity and reactivity of the analytical reagents verified before use? X \_\_\_  
AX-21/Celite checks on file? (8290 requirement) X \_\_\_
15. What is the final extract volume? 20 uL

## SECTION II. - COMMENTS:

Note 1: balance is located inside of a hood - this is a good location for safety but may subject the balance to drafts and temperature changes.

Note 2: Lot numbers are noted but it may be difficult to trace some solvents.

## SECTION III. STANDARDS PREPARATION

	YES	NO
Is a person designated in charge of standard prep?	<u>      </u>	<u>  X  </u>
Name _____ Title _____		
Education _____ Experience _____		
1. Are commercially prepared standard mixes used? If not, are stock standards obtained from certified sources?	<u>  X  </u>	<u>      </u>
2. Are separate areas used for standard prep and sample prep?		NA
3. Are standards properly stored in the dark?	<u>  X  </u>	<u>      </u>
4. a. Are log books of spiking/calibration standards preparation and tracking maintained?		NA
b. Does the log book contain:		
- reference to stock standard used in prep?		NA
- identity of person preparing standard?		NA
- date of preparation?		NA
- analyte concentrations?		NA
5. Are working standards properly labeled with:		
a. analyte concentrations	<u>  X  </u>	<u>      </u>
b. date of preparation	<u>  X  </u>	<u>      </u>
6. Are standards prepared fresh at an acceptable frequency?	<u>  X  </u>	<u>      </u>
7. Are standards stored separately from extracts?	<u>  X  </u>	<u>      </u>
8. Does the SOP contain a section on Standards Prep? If so, is it posted within the prep area?		NA NA
9. Are primary standards traceable to EPA references?	<u>  X  </u>	<u>      </u>
10. How does the laboratory insure that the solutions are prepared or received at appropriate concentrations?		
<u>comparison with previous standard lot</u>		

## SECTION III. - COMMENTS:

The laboratory is using commercially prepared internal standard solution (sample fortification solution) and recovery standard solution. These are diluted as appropriate. Instrument calibration standards are also commercially prepared.

## SECTION IV. - HRGC/HRMS ANALYSIS

YES NO

## HRGC/HRMS Supervisor

Name Don Eickhoff Title Group Leader  
 Education BS Experience 5 yrs

## Other HRGC/HRMS personnel

Name Melanie Finn  
 Education still working on degree Experience 3 yrs

Name \_\_\_\_\_  
 Education \_\_\_\_\_ Experience \_\_\_\_\_

- |    |   |          |          |
|----|---|----------|----------|
| 1. | Is method 8290 used?  | <u>X</u> | _____    |
|    | Is method 1613 used?  | <u>X</u> | _____    |
|    | Any other methods used? <u>not for high-resolution</u>  |          | _____    |
| 2. | List instrumentation used for analysis:<br><u>2 VG Autospec</u>   |          | _____    |
| 3. | Are methods (SOPs) available to analysts in the instrumentation area? <u>copy of 1613 was in lab but copy of 8290 was not kept in lab</u> | _____    | <u>X</u> |
| 4. | a. Are analysis log books used and kept at the instrument?  | <u>X</u> | _____    |
|    | b. Do they include all injections/analyses made on the instrument?  | <u>X</u> | _____    |
|    | c. Does the record include:   |          |          |
|    | - analyst(s) name   | <u>X</u> | _____    |
|    | - date of analysis  | <u>X</u> | _____    |
|    | - time of analysis  | <u>X</u> | _____    |
|    | - laboratory filename and/or client filename  | <u>X</u> | _____    |
|    | d. Are the raw data (SICPs, etc) labeled so as to be able to be related to the log book?  | <u>X</u> | _____    |
| 5. | Are instrument maintenance logbooks kept?   | <u>X</u> | _____    |
| 6. | Does the laboratory perform regular preventative maintenance?   | <u>X</u> | _____    |
|    | Is a maintenance schedule available?  | _____    | <u>X</u> |
|    | <u>maintenance is done as needed</u>  |          |          |
| 7. | Does the laboratory have contracts with manufacturers for maintenance?  | <u>X</u> | _____    |
| 8. | Are all raw data maintained, either on hard copy or magnetic tape?  | <u>X</u> | _____    |

## Section IV (cont.)

	YES	NO
A. MASS RESOLUTION CHECK		
1. Is the HRGC/HRMS properly tuned (every 12 hours) with PFK?	<u>X</u>	___
2. Is the 12-hour time period monitored?	<u>X</u>	___
3. Is the exact mass of m/z 380.9760 checked? (within 5 ppm - 380.9741 to 380.9779)	<u>X</u>	___
4. Is this properly documented? (scale calibrated and peak width on hardcopy) <u>scale was not calibrated</u>	___	<u>X</u>
5. Are these requirements included in the SOP?	<u>X</u>	___
B. GC COLUMN PERFORMANCE CHECK		
1. Analyzed at beginning of 12-hour shift?	<u>X</u>	___
2. Is the 2378-TCDD resolution checked? ( $\leq 25\%$ )	<u>X</u>	___
3. Is the 1289-TCDD and 13468-PeCDF switching time checked? (allowable tolerance $> 10$ sec.)	<u>X</u>	___
4. Eight homologue retention time windows determined? (are these explicitly recorded?)	<u>X</u>	<u>X</u>
5. Is this properly documented? <u>first and last eluters not labeled</u>	___	<u>X</u>
6. Are these requirements included in the SOP?	___	<u>X</u>
C. INITIAL CALIBRATION		
1. Analyzed at proper frequency?	<u>X</u>	___
2. Concentrations as per 8290 or 1613?	<u>both</u>	___
3. Are the following parameters checked? Signal-to-noise ratio $\geq 2.5$ , % RSD $\leq 20$ or 30, ion abundance ratio within limits	<u>X</u>	___
4. Is this properly documented?	<u>X</u>	___
5. Are these requirements included in the SOP?	<u>X</u>	___

## Section IV (cont.)

	YES	NO
D. CONTINUING CALIBRATIONS		
1. Analyzed at proper frequency? (beginning and ending calibration each 12-hrs)	<u>X</u>	___
2. Correct concentration used (cc-3 solution)?	<u>X</u>	___
3. Are the following parameters checked? Signal-to-noise ratio $\geq 2.5$ , % D or RPD $\leq 20$ or 30, ion abundance ratio within limits	<u>X</u>	___
4. Ending calibration - %RPD checked? If it fails, are proper measures carried out? <u>%D is checked; the lab knows the corrective measures but has never had to use them</u>	___ <u>X</u>	<u>X</u> ___
5. Is this properly documented?	<u>X</u>	___
6. Are these requirements included in the SOP?	<u>X</u>	___
E. METHOD BLANKS		
1. Analyzed at proper frequency? (immediately after each Ical or Ccal) <u>lab is analyzing blank only once</u>	___	<u>X</u>
2. Does the SOP clearly define acceptable blanks and note corrective actions, if needed?  If there are trace blank contaminants that are also in samples, how are they accounted for in the final reported data? <u>report results out and let end user make decision</u>	___	<u>X</u>
F. MATRIX SPIKE/MATRIX SPIKE DUPLICATE		
1. Analyzed at proper frequency? (one set per SDG/batch)	<u>X</u>	___
2. Does the SOP clearly define acceptable RPD? <u>the lab has defined a RPD of 25% but 8290 specifies a RPD of 20%</u>	<u>X</u>	___
3. Is this properly documented?	<u>X</u>	___

## Section IV (cont.)

	YES	NO
G. SAMPLE ANALYSIS		
1. Injection volume used?	<u>2 ul</u>	
2. Analyzed with same instrument settings as associated calibration/performance check samples?	<u>X</u>	<u>    </u>
3. Internal standard recovery monitored? (40% -135%)	<u>X</u>	<u>    </u>
4. Proper Identification criteria used? (retention time, s/n ratio, ion abundance ratio, current response max)	<u>X</u>	<u>    </u>
5. Are EMPCs reported?	<u>X</u>	<u>    </u>
6. Are PCDPEs monitored? hardcopy provided?	<u>X</u>	<u>    </u>
7. Are the lockmass ions continuously monitored? <u>provided documentation does not include SICPs</u>	<u>X</u>	<u>    </u>
8. Dilutions properly performed?	<u>X</u>	<u>    </u>
9. Is 2378-TCDF properly confirmed? <u>done only on client request</u>	<u>X</u>	<u>    </u>
10. Is all of this properly documented?	<u>X</u>	<u>    </u>
11. Are these requirements included in the SOP? <u>SOP is vague and references 8290 which was not in lab at time of audit</u>		<u>see note</u>
H. DATA REDUCTION/REVIEW		
1. Is there any secondary review of all documents/data by any person other than the one generating the information? <u>data review SOP was not kept in lab</u>	<u>X</u>	<u>    </u>
2. Is there a system of data qualifiers (or other means) to denote reported data that have not met all QC criteria, i.e., holding times exceeded, surrogates out, internal standards out, etc.? <u>only qualifier used is J for &lt; DL</u>	<u>    </u>	<u>X</u>
SECTION IV. - COMMENTS: <u>instrument software autoamatically checks S/N ratio; the S/N ratio is not printed on data forms</u>		

## SECTION V. - FILE AUDIT

Are there files maintained on each project? X yes \_\_\_\_\_ no

Are files/records maintained such that ALL information for each project is available for future reference? X yes \_\_\_\_\_ no

	YES	NO
1. a. Does the file contain a record of sample receipt?	<u>X</u>	_____
b. Is the chain of custody/log book properly recorded to clearly indicate date of receipt and person receiving samples?	<u>X</u>	_____
2. a. Are the records of extraction/preparation in the file or available?	<u>X</u>	_____
b. Are the records of extraction/prep properly recorded with date of extraction, person performing the analysis, and the technique used?	<u>X</u>	_____
c. Are the records clear for wts./volumes extracted or digested, final volumes of sample, etc.	<u>X</u>	_____
d. If soil sediments were analyzed, is there a % moisture determination, properly documented and reported along with the data?	_____	<u>X</u>
3. a. Is the sample analysis (instrumental) information in the file or available?	<u>X</u>	_____
b. Are instrument run logs properly recorded and included for ALL sample analyses (be sure to note any reanalyses)?	_____	<u>X</u>
c. Is documentation of all calibrations available?	<u>X</u>	_____
Were calibrations properly performed?	_____	<u>X</u>
<u>sequence was different than "typical"</u>		
<u>sequence presented in method - windows</u>		
<u>are only determined during Ical, not for</u>		
<u>every 12-hour shift as specified in 8290</u>		
Were corrective actions, if indicated, properly performed?		NA

7. Do all dates and times follow a logical pattern?  X     

List discrepancies: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

SECTION XII. - COMMENTS: