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DATAVAL<sup>®1992</sup> : A COMPUTER PROGRAM  
FOR THE VALIDATION OF  
ANALYTICAL DATA FOLLOWING  
ENVIRONMENTAL PROTECTION AGENCY  
REQUIREMENTS

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## CONTENTS

FIGURES .....	vii
TABLES .....	vii
ACRONYMS AND INITIALIZATIONS .....	ix
ABSTRACT .....	xi
1. INTRODUCTION .....	1
2. DATA VALIDATION GUIDELINES .....	2
3. PROGRAM DESCRIPTION .....	3
3.1 PACKAGE IDENTIFICATION .....	7
3.2 MENUS .....	7
3.2.1 Main Menu .....	10
3.2.2 SAMPLES .....	10
3.2.3 MB .....	12
3.2.4 LAB .....	15
3.2.5 REPORTS .....	17
3.2.6 FINAL .....	17
3.2.7 PRINT .....	17
3.2.8 DELETE .....	22
3.2.9 MISC .....	22
4. REPORTS .....	28
4.1 HOLDING TIMES REPORT .....	30
4.2 INSTRUMENT PERFORMANCE REPORTS .....	33
4.2.1 Tuning Report .....	33
4.2.2 P_P Instrument Performance Report .....	36
4.3 CALIBRATION REPORTS .....	38
4.3.1 VOL, BNA, and H_A .....	38
4.3.2 P_P .....	41
4.3.3 PHC .....	44
4.3.4 MET .....	45

4.3.5 ANI .....	48
4.4 BLANKS REPORT .....	49
4.5 SURROGATE RECOVERY .....	52
4.6 SPIKE REPORTS .....	53
4.6.1 MS/MSD Report .....	56
4.6.2 Blank Spike Report .....	58
4.6.3 MS Report .....	58
4.7 FIELD DUPLICATES REPORT .....	60
4.8 INTERNAL STANDARDS REPORT .....	60
4.9 LAB DUPLICATES REPORT .....	63
4.10 SPECIAL REPORTS FOR MET AND ANI ANALYSES .....	63
4.10.1 ICP Interference Check Sample Report .....	63
4.10.2 LCS Report .....	65
4.10.3 ICP Serial Dilution Report .....	65
4.10.4 Furnace AA QC Report .....	69
4.11 CONTAMINATION REPORT .....	69
4.11.1 Special Calibration Report for PHC .....	72
4.12 SYSTEM REVIEW REPORT .....	73
4.13 OVERALL ASSESSMENT REPORT .....	73
4.14 FINAL SUMMARY REPORT .....	75
5. COVER SHEETS .....	75
6. PACKAGE DEFICIENCIES SUMMARY .....	80
7. PROGRAM TESTING .....	83
REFERENCES .....	85
GLOSSARY .....	86

APPENDIX A	REPORTS REQUIRED FOR VALIDATION LEVEL
APPENDIX B	DATA QUALIFIER Q FLAGS
APPENDIX C	ELECTRONIC TRANSFER DATA BASES
APPENDIX D	PROGRAM FLAGGING LOGIC

APPENDIX E	TEST PACKAGE REPORTS
APPENDIX F	EQUATIONS USED FOR VALIDATING DATA
APPENDIX G	NONCONFORMANCE REPORT AND PROCEDURE



## FIGURES

1.	Package selection menu	8
2.	Package ID input screen	9
3.	Main menu	11
4.	SAMPLES menu	13
5.	MB menu	14
6.	LAB menu	16
7.	REPORTS menu, SDG selection menu	18
8.	REPORTS menu, analysis type selection screen	19
9.	FINAL menu	20
10.	PRINT menu	21
11.	DELETE screen	23
12.	MISC menu	24
13.	Analysis type selection screen for editing CAS libraries	25
14.	EPA analysis method codes screen	27
15.	VOL report selection menu	31
16.	Holding times reports for H_A and BNA	32
17.	Tuning reports for VOL and BNA	35
18.	P_P instrument performance reports	37
19.	Initial calibration reports for VOL, BNA, and H_A	40
20.	Continuing calibration reports for VOL, BNA, and H_A	42
21.	Initial and continuing calibration for P_P	43
22.	PHC calibration report	46
23.	Calibration reports for MET	47
24.	ANI calibration report	50
25.	VOL blanks report, pages 1 and 2	51
26.	Surrogate recovery report for BNA	54
27.	Surrogate recovery report for P_P	55
28.	MS/MSD report for ANI	57
29.	Blank spike and MS reports for VOL	59

30.	Field duplicates report for VOL .....	61
31.	Internal standards report for BNA .....	62
32.	Lab duplicates report for BNA .....	64
33.	ICP interference check sample report for MET .....	66
34.	Laboratory control samples report for ANI .....	67
35.	ICP serial dilution report for MET .....	68
36.	Furnace atomic absorption QC report for MET .....	70
37.	Contamination report for BNA .....	71
38.	Contamination report, including level D, for PHC .....	74
39.	Final summary report .....	76
40.	Final flags table .....	77
41.	Cover sheet for organics analyses .....	78
42.	Cover sheet for inorganics analyses .....	79
43.	Package Deficiencies Summary, page 1 .....	81
44.	Package Deficiencies Summary, page 2 and 3 .....	82
45.	Package Deficiencies Summary, page 4 .....	84

## TABLES

1.	DATAVAL analyses and EPA analysis methods .....	6
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## ACRONYMS AND INITIALIZATIONS

AA	atomic absorption
ANI	anions analysis
BFB	bromofluorobenzene
BNA	base/neutral/acid analysis
CAS	Chemical Abstract Service
CCV	continuing calibration verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CF	calibration factor
CLP	Contract Laboratory Program
CRDL	contract-required detection limit
CRQL	contract-required quantitation limit
DBC	dibutylchloroendate
DFTPP	decafluorotriphenylphosphine
DL	laboratory duplicate
DOE	Department of Energy
DSD	data set deliverables
EPA	Environmental Protection Agency
ER	equipment rinsate
FB	field blank
GC	gas chromatograph
H_A	halocarbons and aromatic hydrocarbons analysis
HAZWRAF	Hazardous Waste Remedial Actions Program
HT	holding times
ICP	inductively coupled plasma
ICS	interference check sample
ICV	initial calibration verification
ID	identification
IDL	instrument detection limit

IS	internal standard
LCS	laboratory control sample
MB	method blank
MET	metals analysis
MS	matrix spike
MS/MSD	matrix spike/matrix spike duplicate
NPL	National Priorities List
ORNL	Oak Ridge National Laboratory
P_P	pesticides and polychlorinated biphenyls analysis
PAG	Pollutant Assessments Group
PCB	polychlorinated biphenyl
PHC	petroleum hydrocarbons analysis
QC	quality control
RE	re-extraction
RPD	relative percentage difference
RRF	relative response factor
RRT	relative response time
RSD	relative standard deviation
RT	retention time
SDG	sample delivery group
SR	soil sample replicate
ST	soil sample triplicate
TAL	target analyte list
TB	trip blank
TC	target compounds
TCL	Target Compound List
TIC	Tentatively Identified Compounds
VOL	volatile organic compounds analysis
WR	water sample duplicate
WT	water sample triplicate
%D	percent difference
%R	percent recovery

## ABSTRACT

A computer program, DATAVAL, has been developed by personnel at the Oak Ridge National Laboratory Pollutant Assessments Group in Grand Junction, Colo., for the validation of analytical chemical data issuing from the U. S. Environmental Protection Agency's (EPA) Contract Laboratory Program. The program accommodates the electronic transfer of: 1) sample data that have been entered into a computer in the field and 2) sample results in the prescribed format from an analytical laboratory. DATAVAL then guides the validator through the EPA validation procedures, performs calculations and evaluations of the data, and prints reports in the required format. The validation process is rendered less time-consuming and error-prone. A description of the program and details of the data validation methods used are presented in this report.



## 1. INTRODUCTION

The Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) provides procedures for the identification, evaluation, and remediation of former hazardous waste disposal sites. During these activities, the analysis of soil, water, and waste samples may be performed. Under certain circumstances, the quality of the results of these analyses must be verified and validated. A computer program has been developed by personnel at the Oak Ridge National Laboratory (ORNL) in Grand Junction, Colo., for the validation of analytical chemical data. This program, DATAVAL, is used in support of the ORNL Pollutant Assessment Group's (PAG) site-characterization monitoring programs. Originally developed for the Arizona Air National Guard Project, Tucson, Ariz., in March 1990, the program has subsequently been used for several other projects. This report gives a description of the data validation process employed in DATAVAL, including the calculations used and the structure required for input and output of data.

Data validation, as described herein, is a technical review and evaluation of analytical data generated through the U.S. Environmental Protection Agency's (EPA) Contract Laboratory Program (CLP) and is performed according to EPA guidelines. Based on specific quality control (QC) criteria, the validation process provides information on the analytical limitations of data. The initial procedure used by ORNL involved reviewing the data, taking hand-written notes, verifying calculations by calculator, and transcribing notes into a report. This transcription introduced errors into the process that were often difficult to find and time-consuming to correct. It became obvious that a computer program was needed that could: 1) guide a data validator through the EPA validation procedures, 2) perform calculations and evaluations of the data, and 3) print reports in the required format. Such a program has been written. DATAVAL is set up to follow the sequence of steps for data validation outlined in EPA guidelines, thus reducing errors of omission.

An IBM®-compatible computer with a hard drive, using MS-DOS® version 3.3 operating system, is required to run the program. A laser jet printer is also required; a math coprocessor is desirable. The computer program consists of DATAVAL.EXE and 207 associated data files, all of which reside in the same directory. Compiled by Clipper® version 5.01 and linked with Blinker® version 1.5, the source code used to develop DATAVAL.EXE is listed in *DATAVAL: Data Validation Source Code, Version 1.0A*, ORNL/TM-12377.

## 2. DATA VALIDATION GUIDELINES

DATAVAL is based on EPA requirements for the validation of analytical data for organic and inorganic compounds (U.S.EPA 1988a, 1988b, 1991a) with additional outlines and guidance taken from the U.S. Department of Energy (DOE) document, *Quality Control Requirements for Field Methods* (U.S.DOE 1990), prepared by the Hazardous Waste Remedial Actions Program (HAZ-WRAP) Support Contractor Office, Martin Marietta Energy Systems, Inc., Oak Ridge, Tenn. The requirements in these documents are explicit and include the following: 1) the logical sequence to be followed in data validation, 2) the number and types of calculations required, 3) the equations for evaluating the data and the limits with which the data have to comply, 4) the required reports for each level of validation, and 5) the logic upon which decisions concerning the validity of the data are made. DATAVAL is designed to meet the requirements of these documents and ease the task of the validator by providing calculations and structure. The validator interfaces with the program by reviewing forms, inserting data electronically or manually, and making decisions other than those made by the program.

The EPA has identified five general levels of analytical options to support data collection based on the type of site to be investigated, the level of accuracy and precision required, and the intended use of the data (U.S.EPA 1987).

HAZWRAP defines levels A, B, C, D, and E to correspond to EPA levels I, II, III, IV, and V (U.S.DOE 1990). DATAVAL is designed to execute level B (II), level C (III), and level D (IV) validations. Level E (V) may be used in the future.

The level of validation required is determined by several considerations:

- Is rapid turnaround of sample results in the field needed?  
If so, use level B.
- Are the data from a National Priorities List (NPL) site?  
If so, use level D.
- Is the site located near a populated area?  
If so, use level C if the site is not on the NPL and is not likely to undergo litigation.
- Is there a possibility of litigation?  
If so, use level D.

The validation reports required for each of these levels of validation are outlined in Appendix A.

Analytical results arrive from the laboratory flagged with any of several data qualifiers or Q flags. During the validation process, flags may be added or changed. These flags are defined in Appendix B and will be referred to throughout this report. All compounds without data qualifiers should be considered to be present in the sample at the concentration given.

### 3. PROGRAM DESCRIPTION

DATAVAL is structured to produce a number of reports that cover all the requirements for data review. The following terms, used throughout this document, are defined to clarify the relationship between reports produced by

DATAVAL. Additional definitions may be found in the Glossary.

**Data Set Deliverables (DSD):** the forms, charts, narratives, and results from the analytical laboratory that pertain to the group of data being validated. For level C validation, this includes all CLP forms, case narratives, control charts, and calibration curve data. Level D validation requires, in addition, all data results. Level B requirements are less than level C, consisting basically of forms, charts, and data pertaining to calibrations, blanks, and duplicates.

**Package:** a validation final report that relates to a specific period of sampling, such as soil samples taken during monitor well installation or water samples taken during the first round of water sampling.

**Packet:** a group of reports for one specific analysis included within a package.

**Project:** the name of the facility at which the samples were collected, such as a military base or federal facility. A project may require several different rounds of sampling and, therefore, several different packages.

**Report:** a specific requirement within the data validation package, such as a tuning report for volatile organic compounds analysis or a matrix spike/matrix spike duplicate report for petroleum hydrocarbons analysis. Different analyses have different requirements for the same report. The tuning report for the volatile organic compounds analysis is slightly different from the tuning report for the semivolatile organic compounds analysis.

Seven analyses may be validated by the program:

1. Volatile organic compounds (VOL)
2. Semivolatile organic compounds (BNA, for base/neutral/acid analysis)
3. Pesticides and polychlorinated biphenyls (P\_P)

4. Metals (MET)
5. Petroleum hydrocarbons (PHC)
6. Halocarbons and aromatic hydrocarbons (H\_A)
7. Anions (ANI).

OL, BNA, P\_P, and MET analyses are covered by EPA and HAZWRAP guidelines (U.S.EPA 1988a; U.S.EPA 1988b; U.S.DOE 1990). Data validation requirements for the PHC analysis are set forth by HAZWRAP (U.S.DOE 1990). H\_A and ANI validations were established for specific projects. H\_A validation closely follows that for BNA; ANI validation includes some requirements from both VOL and MET. Table 1 correlates the various DATAVAL analyses to the EPA analysis methods used by the analytical laboratory.

During the analysis of samples and the validation of results, samples are often referred to by matrix and by type. Samples may occur as either soil samples or water samples, soil or water being the matrix. QC sample types are: method blank (MB), field blank (FB), trip blank (TB), equipment rinsate (ER), matrix spike (MS), matrix spike/matrix spike duplicate (MS/MSD), sample reextraction (RE), laboratory duplicate (DL), water sample duplicate (WR), water sample triplicate (WT), soil sample replicate (SR), and soil sample triplicate (ST).

One of the most useful features of DATAVAL is the CAS (for Chemical Abstract Service) or target compound (TC) libraries. These have been established to coincide with EPA listings of the pollutants or environmental contaminants to be determined during the analysis of environmental samples. For all organics analysis, a target compound list (TCL) and contract required quantitation limits (CRQLs) have been set (U.S.EPA 1991b). Likewise, for inorganics analysis, a target analyte list (TAL) and CRQLs have been set (U.S.EPA 1990). In addition, a tentatively identified compounds (TIC) list has been established for organic compounds. All of these lists can be and are revised by the EPA on occasion. Because of their importance to data validation, these lists have been incorporated into TC libraries, which function as the backbone of DATAVAL.

Table 1. DATAVAL analyses and EPA analysis methods

Analysis	EPA Analysis Method	
	Water	Soil
VOL	624	8240
BNA	625	8250
P_P	608	8080
PHC (extractables)	see U.S. DOE 1990	
PHC (total)	418.1	418.1
MET	200.7	6010
	206.2	7060
	245.1	7470
	etc., depending on the analyte	
ANI	429	
H_A	601	8010

Note: The methods listed are an example of the many methods listed for some of these analyses. The list is not complete but provides a fairly good sampling of the methods used.

### **3.1 PACKAGE IDENTIFICATION**

Throughout the data validation process, packages are kept unique by a series of identification steps executed during program start-up. DATAVAL begins by presenting a package selection screen, listing existing packages and the option for creating a new package (Fig. 1). A new package is identified by the name or abbreviation of the project, along with a notation for the round of sampling (Fig. 2). For example, for a project at the Fallon Naval Air Station with a round of sampling in July, the new package might be identified as FNAS/July. At this point, the level of validation (B, C, or D) is identified for the package. The package identification (ID) name is necessary in order to get into the correct report data fields. Data are stored in the program under data fields that can only be opened by the correct package ID.

### **3.2 MENUS**

DATAVAL includes a menu system that is arranged in the logical sequence of the validation process and serves as a guide through the steps of data validation. The system is keyed to select different reports, keyed to the type of analysis, and keyed to the level of validation. The menus also allow the validator the flexibility to follow any sequence of data validation.



```
* * * * *  
*  
*          CHEMICAL DATA VALIDATION REPORT GENERATOR          *  
*  
*  
*          Package Name: TEST                                     *  
*          Package Code: 1                                       *  
*          Project Name: PROGRAM TEST                             *  
*          Laboratory:  ORNL                                       *  
*  
*          Validation Analyst's Name:  DENNIS MARTY              *  
*          Beginning Date of Validation: 09/23/92                *  
*          Validation Level (B,C,D,E,II,III,IV,V):  D           *  
*  
*  
*          Enter data. [Esc] to abort changes and select a different package. *  
*  
*  
* * * * *
```

Fig. 2. Package ID input screen.

### 3.2.1 Main Menu

The main menu allows access to generalized areas of the program and appears across the top of the screen, as shown in Fig. 3. These entries represent the following:

- **SAMPLES:** Sample data base (enter, edit)
- **MB:** Method blank data base (enter, edit)
- **LAB:** Lab data base (enter, edit)
- **REPORTS:** Report input
- **FINAL:** Final report edit
- **PRINT:** Print final report
- **DELETE:** Clear data from all packages
- **MISC:** TCL, TIC, and EPA method code data bases (enter, edit)

This menu permits the validator to load the field data base into the sample data base, load analytical results into the lab data base, switch between sample packets, and edit and print a final report. The desired entry is highlighted using the arrow keys and selected by pressing Enter. Each of these areas of the program is discussed below along with the menu(s) that appear when an entry is selected.

### 3.2.2 SAMPLES

Several data bases are accessed during the course of the program. The sample data base, entered using this option, interfaces with both the field data base and the lab data base.



All data that describe and document samples and sampling conditions during a sampling trip are kept in a portable computer in the field. These include: (1) sample field number, (2) sample matrix, (3) sample date, (4) sample type, and (5) requested analyses. At the end of the trip, this ASCII-formatted field data base is downloaded to the sample data base located in DATAVAL, simplifying the data validation process and increasing the efficiency of the program by reducing data entry time and the possibility of errors. In the SAMPLES menu, shown in Fig. 4, the AUTO option is used to transfer data electronically. See Appendix C for a description of the fields used by the program for electronic transfer of data.

Provision has been made for entering data into the sample data base manually. At the main menu, the validator selects the SAMPLES option, followed by the MANUAL option. He then enters a number and the sample type and chooses an analyses type for that sample. The following are entered for each analysis type selected: 1) sample matrix, 2) sample date, 3) SDG, 4) extraction date, 5) analysis date, 6) analysis time, and 7) dilution. Data can be entered from chain-of-custody reports or field logbooks before the arrival of the DSD. SDG numbers, extraction dates, analysis dates and times, and dilutions are skipped and added into the program when the DSD arrives. These data are added by using either the MANUAL option, as described above, or the BROWSE option in the SAMPLES menu selection.

Data from the sample data base are combined with analytical results from the lab data base when this second data base is created by entering the lab data base with the same package ID that has been given to the sample data base.

### **3.2.3 MB**

The MB data base is created automatically when data are entered into the lab data base electronically (see section below). However, MB data may be entered manually into the data base when necessary. The MB menu is shown in Fig. 5.





Method blank information is correlated to samples, and the program accesses the MB data base to make decisions about contaminants in other parts of the program. Since specific method blanks may be used for more than one SDG, a separate data base was set up.

### 3.2.4 LAB

Analytical results from the laboratory may be introduced into the DATAVAL lab data base electronically by transfer of data from a floppy disk. When the lab data base is created electronically, the sample data base is updated, and method blank data are entered in to the MB data base. The AUTO option in the LAB menu is used to make this electronic transfer from disk (Fig. 6). See Appendix C for a description of the fields used by the program for electronic transfer of data.

For entering data manually into the lab data base, Form I for all samples is reviewed to locate analysis dates, analytes detected, concentrations, and Q flags. From the main menu, the LAB option is selected, and all applicable SDG numbers appear. Upon selecting an SDG number, an analysis selection screen appears, and an analysis is selected. A list of all samples within that SDG with that analysis appears. If contamination in a sample is indicated on the DSD form, the TC library for that analysis is entered by pressing the F10 key. Analytes detected are selected from the library for that sample, and their concentrations are entered. Q flags are also entered at this time if needed. These steps are repeated until all analytes have been entered.

Compounds that do not appear in a TC library may be added by highlighting and choosing NEW at the bottom of the TCL or TIC list to produce a screen for adding new compounds. The ESC key returns the validator to the sample number selection screen, and the steps above are repeated until all samples have been entered. The ESC key returns the validator to the analysis type selection screen so that a new analysis can be selected along with the appropriate TC library. This is repeated until all requested analyses have been entered. Using the ESC key to



step back one screen at a time, a new SDG number can be selected, and all the above steps repeated for that group. This sequence is repeated until all samples have been entered into the program.

The logic used within DATAVAL to insert flags into this data base as validation proceeds is based on EPA requirements. See Appendix D for a description of DATAVAL flagging logic.

### **3.2.5 REPORTS**

It is in the REPORTS option that most of the actual data validation takes place. Choosing the REPORTS option at the main menu presents a screen for SDG selection (Fig. 7). This screen is followed by one for analysis type selection (Fig. 8) and then by a screen listing all the different types of reports for the analysis chosen. These reports are discussed in detail in Sect. 4.

### **3.2.6 FINAL**

This part of the program allows the validator to edit and print the final summary report, along with cover sheets and a report of package deficiencies needed for the final report presentation copy (Fig. 9). The summary report, which includes all sample contamination data and laboratory and validation flags, is discussed in Sect. 4.13. The cover sheets and the Package Deficiencies Summary are described in Sect. 5 and 6.

### **3.2.7 PRINT**

All the individual reports produced by DATAVAL and included in the final report presentation copy are printed, by analysis, using this option (Fig. 10).



```
*****
*
*
*          CHEMICAL DATA VALIDATION REPORT GENERATOR
*
*  Esc  SAMPLES  MB  LAB  REPORTS  FINAL  PRINT  DELETE  MISC
*
*
*          ANALYSIS TYPE SELECTION
*
*          1. VOL
*          2. BNA
*          3. PHC
*          4. H A
*          5. P P
*          6. MET
*          7. ANI
*          8.
*
*          Select analysis type. [Esc] to abort.
*
*
*          Develop the individual reports.
*
*
*
*****
```

Fig. 8. REPORTS menu, analysis type selection screen.



```
* * * * *  
*  
*          CHEMICAL DATA VALIDATION REPORT GENERATOR          *  
*  
*   Esc  SAMPLES  MB  LAB  REPORTS  FINAL  PRINT  DELETE  MISC  *  
*                                     ----- *  
*                                     Esc *  
*                                     ANALYSIS *  
* * * * *  
*  
*                                     Return to main menu. *  
* * * * *  
* * * * *
```

Fig. 10. PRINT menu.

### 3.2.8 DELETE

The DELETE option is used to delete all data from all packages (Fig. 11.).

### 3.2.9 MISC

The MISC option contains: 1) access to the CAS libraries and the EPA analysis method codes and 2) means for editing menus for validation level and report forms for column headings (Fig. 12).

As described earlier, the CAS libraries are made up of TCL and TAL compounds and TIC. A separate library exists for each of the seven analysis types; for editing, these libraries are entered from an analysis type selection screen (Fig. 13). Each library contains the following:

1. compound name: the compounds or elements covered by that analysis,
2. CAS number: each compound's Chemical Abstract Service number,
3. TCL or TIC designation: classification of each compound or element as being a TCL compound (this includes TAL compounds) or a TIC,
4. limits: CRQL according to sample matrix (for TCL only),
5. analysis method: EPA analysis method number according to sample matrix,
6. units: default concentration units according to sample matrix.

The program accesses the correct library according to the analysis that is currently logged into the report menu. Compounds in the library may then be highlighted for entry into the report.

```
* * * * *  
*  
*  
*          CHEMICAL DATA VALIDATION REPORT GENERATOR          *  
*   Esc  SAMPLES  MB  LAB  REPORTS  FINAL  PRINT  DELETE  MISC  *  
*  
*  
*          All data from all packages will be deleted (Y/N): N  *  
*  
*  
*  
*  
*  
*  
*  
*  
*  
*          Delete all validation packages.                       *  
*  
*  
* * * * *
```

Fig. 11. DELETE screen.

```
* * * * *  
*  
*          CHEMICAL DATA VALIDATION REPORT GENERATOR          *  
*  Esc  SAMPLES  MB  LAB  REPORTS  FINAL  PRINT  DELETE  MISC  *  
*                                     ----- *  
*                                     Esc *  
*                                     CAS *  
*                                     EPA *  
*                                     MENUS *  
*                                     FORMS *  
* * * * *  
*  
*                                     Return to main menu. *  
* * * * *  
* * * * *
```

Fig. 12. MISC menu.



Fields within the libraries designate various limiting parameters and are self-descriptive. Examples of these field names are:

DLO:       % difference low limit  
WMSRLO: water matrix MS/MSD % recovery low limit  
SCRQL:     soil matrix contract required quantitation limit  
WEHT:      water matrix extraction holding time.

DATAVAL is designed to look for these limits when a comparison is made between a calculated number and a limit range, using up to two indices (compound name and analysis type) for the determination of the correct location of the limit. The program includes computer instructions for each report that select the correct limits from the TC library and make a comparison of a calculated number and the limits.

If a new laboratory procedure or contract changes the validation limits, those limits can be edited with the MISC menu selection. Either single limits or large groups of limits can be selected and changed. New compounds can be added to the TCL list, by analysis, using this menu. TIC additions are made during the DSD input, when sample contamination is added to the program. An input line at the bottom of the data base table (labeled NEW) is used to add new compounds to the TIC list.

A dynamic part of DATAVAL, the TC libraries provide flexibility for additions and modifications without the need for time-consuming programming changes.

Also included in the MISC option is a data base of EPA analysis method codes that may be edited. These codes are generated by the analytical laboratories and are seen by DATAVAL only when data are transferred electronically from a laboratory. This data base allows these codes to be correlated to DATAVAL analysis codes (VOL, ANI, MET, etc.). An example of these codes may be seen in Fig. 14.



#### 4. REPORTS

At the heart of DATAVAL are the reports that are generated for each analysis. To accomplish the review of sample data, the EPA requires that a number of aspects of each analysis be checked. For example, the following is the list of requirements for the volatile organic analysis (U.S.EPA 1991a):

1. Technical holding times
2. Gas chromatograph/mass spectrometer (GC/MS) tuning
3. Initial calibration
4. Continuing calibration
5. Blanks
6. Surrogate recovery
7. Matrix spikes/matrix spike duplicates
8. Field duplicates
9. Internal standards
10. TCL compound identification
11. Compound quantitation and reported detection limits
12. Tentatively identified compounds
13. System performance
14. Overall assessment of data.

DATAVAL is structured in a report format in order to accomplish these various checks. Appendix A lists the required reports by analysis and level of validation. In addition to specific formats according to analysis and report type, all reports have a Comments section for additional remarks that may not be covered by the structured report form.

Analytical laboratories submitting data as part of the CLP are required by the EPA to use standardized data reporting forms. DATAVAL has been designed so that the reports issued by the program correspond closely to these forms. Form numbers do not necessarily agree between organics analysis and inorganics analysis. As an example, Form II in the organics DSD has several names:

Form II VOA-1 Water Volatile System Monitoring Compound Recovery  
Form II VOA-2 Soil Volatile System Monitoring Compound Recovery  
Form II SV-1 Water Semivolatile Surrogate Recovery  
Form II SV-2 Soil Semivolatile Surrogate Recovery  
Form II PEST-1 Water Pesticide Surrogate Recovery  
Form II PEST-2 Soil Pesticide Surrogate Recovery

All of these variations of Form II are used for the Surrogate Recovery reports issuing from DATAVAL for VOL, BNA, P\_P, PHC, and H\_A. On the other hand, Form II arriving with the inorganics deliverables has the following names:

Form II (PART 1) - IN Initial and Continuing Calibration Verification  
Form II (PART 2) - IN CRDL Standard for AA and ICP

These are used for the calibration reports DATAVAL produces for MET and ANI.

It should be noted that a given form (e.g., Form II SV-1) may appear with various revision numbers and, therefore, slightly different formats. This stems from the fact that there are now several revisions of the EPA's CLP statement of work documents, both for organics and inorganics analysis. DATAVAL has been made as flexible as possible to accommodate this lack of standardization.

Much of what is contained on these forms is entered into the lab data base or TC libraries of DATAVAL either electronically or manually before actual data validation begins. However, the validator produces each report with the corresponding form in hand, using information on the form as he interacts with the program.

To enter a report menu, REPORTS is selected from the main menu and the SDG selection menu is displayed (Fig. 7). Upon selecting an SDG, the various analyses for that group are displayed (Fig. 8). Picking an analysis delivers the validator into a report selection menu; as an example, the menu for VOL analysis is shown in Fig. 15. These reports correspond closely to the EPA requirements for data validation of the VOL analysis, and many are the same or similar for the various analyses. The validator chooses a report and interacts with DATAVAL as described below. An example of the type of report is shown with each description. A test package showing all reports produced by DATAVAL may be seen in Appendix E.

#### 4.1 HOLDING TIMES REPORT

The holding times (HT) report is generated automatically by DATAVAL as soon as all data are entered into both the lab data base and the sample data base. The purpose of the report is to check the time between sampling and analysis and determine if it is within limits. Figure 16 shows examples of HT reports for BNA and H\_A. These reports differ in that not all samples required both analyses.

The program calculates the time to extraction (if applicable) and the time to analysis and compares these to the EPA and HAZWRAP limit requirements found in the TC library for that analysis (U.S.EPA 1988a, 1988b; U.S.DOE 1990). HT limits have been established by the EPA for water matrix samples only. HAZWRAP has limits for both water and soil samples. If no other guidelines are available, limits for water samples are applied to soil samples by the program. DATAVAL then indicates, true or false (T/F), if these limits have been met. For VOL, BNA, H\_A, P\_P, and PHC, the validator examines sample records to



PROJECT: PROGRAM TEST  
 ANALYSIS: BNA - HOLDING TIMES  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92  
 DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

SAMPLE NUMBER	SAMPLE TYPE	MATRIX	SAMPLE DATE	EXTRACTION DATE	ANALYSIS DATE	EXTRACTION DAYS	EXTRACTION ACCEPTABLE	ANALYSIS DAYS	ANALYSIS ACCEPTABLE
1000		S	09/10/92	09/14/92	09/15/92	4	T	1	T
1000	DL	S	09/10/92	09/15/92	09/16/92	5	T	1	T
1001	SR	S	09/10/92	09/11/92	09/15/92	1	T	4	T
1002		W	09/10/92	09/15/92	09/18/92	5	T	3	T
1002	DL	W	09/10/92	09/20/92	09/21/92	10	F	1	T
1003	WR	W	09/10/92	09/15/92	09/18/92	5	T	3	T
1004	MS	W	09/10/92	09/18/92	09/22/92	8	F	4	T
1005	ER	W	09/10/92	09/18/92	09/22/92	8	F	4	T
1006	FB	W	09/10/92	09/25/92	09/27/92	15	F	2	T

PROJECT: PROGRAM TEST  
 ANALYSIS: H A - HOLDING TIMES  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92  
 DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

SAMPLE NUMBER	SAMPLE TYPE	MATRIX	SAMPLE DATE	EXTRACTION DATE	ANALYSIS DATE	EXTRACTION DAYS	EXTRACTION ACCEPTABLE	ANALYSIS DAYS	ANALYSIS ACCEPTABLE
1002		W	09/10/92		09/15/92			5	F
1003	WR	W	09/10/92		09/22/92			12	F
1004	MS	W	09/10/92		09/22/92			12	F
1005	ER	W	09/10/92		09/22/92			12	F
1006	FB	W	09/10/92		09/27/92			17	F

Fig. 16. Holding times report for H\_A and BNA.

determine if samples were properly preserved. For MET and ANI, digestion and/or distillation logs must be examined to determine if samples were preserved at the proper pH.

The HT report is used during the data validation final edit (Sect. 4.14) to aid in determining if any results should be flagged as estimated (J) and sample quantitation limits as estimated (UJ) or as unusable (R).

## 4.2 INSTRUMENT PERFORMANCE REPORTS

For VOL and BNA analyses that are performed using a GC/MS, instrument performance is checked by the laboratory to ensure mass resolution, identification, and, to some degree, sensitivity of the instrument to TCs. This procedure is called tuning, and the results, which must be verified, are reported on Form V in the DSD.

Pesticides and polychlorinated biphenyls (PCBs) are determined in samples using a gas chromatography (GC) analysis, designated P\_P in DATAVAL. Instrument performance criteria ensure that adequate chromatographic resolution and instrument sensitivity are achieved by the chromatographic system. P\_P instrument performance results are found on Form IX (Pesticide/PCB Standards Summary) and Form VIII in the DSD (U.S.EPA 1989).

EPA guidelines (1988a) are used for validating data.

### 4.2.1 Tuning Report

At the Tuning Report screen for VOL or BNA, the validator enters the Lab File ID number from Form V for that particular tuning and the lower limit number for expanded criteria, if applicable (see below). The % relative abundance and % ion abundance for certain ions are also entered.

The process is a two-step verification, requiring the validator to first check

Form V for the following:

- are all % relative ion abundances within limits (U.S.EPA 1988a)?  
(reported in the FORM column of the report, Fig. 17, Y/N)
- is the form completed for each 12-h period during which samples were analyzed?  
(reported in COMMENTS:)
- are there transcription errors?  
(reported in COMMENTS:)
- is the number of significant digits correct?  
(reported in COMMENTS:)
- do spectra contain sharp peaks (level D only) and have appropriate background subtraction techniques been used?  
(reported in the SPEC column, Y/N)

Step two requires the program to calculate one particular % ion abundance value for each 12-h period reported. For the VOL analysis, using the compound bromofluorobenzene (BFB), the program calculates the % ion abundance of the relative abundance of m/e 176 (ratio of mass to charge of ion) to the relative abundance of m/e 174 [Appendix F, Eq. (1)]. This % abundance must fall within the range  $95.0\% < X < 101.0\%$  (U.S.EPA 1988a). For the BNA analysis, the program calculates the % ion abundance of the relative abundance of m/e 443 to m/e 442 for the compound decafluorotriphenylphosphine (DFTPP) [Appendix F, Eq. (2)]. This % abundance must fall within the range  $17.0\% < X < 23.0\%$  (U.S.EPA 1988a). The report shows the program-calculated % ion abundance, the laboratory-calculated % ion abundance, calculation errors (T/F), and out-of-limit indicator (T/F). Examples of VOL and BNA tuning reports are shown in Fig. 17.

PROJECT: PROGRAM TEST  
 ANALYSIS: VOL - TUNING  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #: 1000

DATE: 09/24/92

DATA VALIDATION LEVEL: D  
 ENDING SAMPLE #: 1007

SDG	LAB ID NUMBER	COMPOUND	EXP	FORM	SPEC	176 m/z RELATIVE ABUN	174 m/z RELATIVE ABUN	CALC % ABUN	LAB % ABUN	CALC ERROR	LIMIT
1000	12345	BFB		Y	Y	73.20	76.20	96.06	96.10	F	T
1002	EBF80305	BFB		Y	Y	78.20	78.40	99.74	99.70	F	T
1002	EBF80306	BFB	Y	Y	Y	78.40	78.80	99.49	99.40	F	T

Comments:

PROJECT: PROGRAM TEST  
 ANALYSIS: BNA - TUNING  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #: 1000

DATE: 09/24/92

DATA VALIDATION LEVEL: D  
 ENDING SAMPLE #: 1007

SDG	LAB ID NUMBER	COMPOUND	EXP	FORM	SPEC	443 m/z RELATIVE ABUN	442 m/z RELATIVE ABUN	CALC % ABUN	LAB % ABUN	CALC ERROR	LIMIT
1000	CV03182	DFTPP		Y	Y	9.30	48.70	19.10	18.80	F	T
1002	DV0304	DFTPP		Y	Y	14.30	72.50	19.72	19.70	F	T

Comments:

Fig. 17. Tuning reports for VOL and BNA.

The lower limit number is used as a key to identify instances where the laboratory uses expanded criteria for the tuning limits, a situation where laboratory instruments that cannot meet the ranges listed are allowed by the CLP to use larger ranges.. For VOL and BNA analyses, the ion abundance lower limit criteria are found under m/e 50 and m/e 51. Once the lower limit is inserted into the program, DATAVAL checks the expanded criteria limits against EPA guidelines (U.S.EPA 1988a). For example, if for the VOL analysis, the 50 m/e % relative abundance value is less than 15, expanded criteria were used, and the program documents the acceptance of expanded criteria in the EXP column (Y for expanded) of the report.

#### **4.2.2 P\_P Instrument Performance Report**

Instrument performance for the P\_P analysis is covered by two reports, Instrument Performance and Cal and Calibration Percent Breakdown (Fig. 18). These are in addition to the calibration reports described in Sect. 4.3.2. Besides reviewing the EPA forms, the validator must also review actual chromatograms to ascertain the validity of the data.

From Form X the validator enters the EPA Sample No., the Instrument ID, and the retention time (RT) of dichlorodiphenyltrichloroethane (4-4'-DDT) on the GC column. The program checks the RT, which must be 12 min or greater, and indicates in the Comments section if results are invalid. Form VII (U.S.EPA 1991a) or Form IX (U.S.EPA 1988a) of the DSD is then reviewed by the validator for pesticide standards RT windows: these windows must be present and the standards must fall within these windows. If missing, the laboratory is contacted to send this information.

Next, the degradation of DDT and endrin are checked. The breakdown of neither may exceed 20% nor may the breakdown of the two combined exceed 20% (U.S.EPA 1988a). The presence of endrin aldehyde and/or endrin ketone in the

PROJECT: PROGRAM TEST DATE: 09/24/92  
 ANALYSIS: P P - INSTRUMENT PERFORMANCE & CAL  
 REVIEWER: DENNIS MARTY DATA VALIDATION LEVEL: D  
 BEGINNING SAMPLE #: 1000 ENDING SAMPLE #: 1007

## DOT RESPONSE TIME

SDG	INSTRUMENT ID NUMBER	CAL DATE	CAL TIME	EPA SAMPLE NUMBER	RT	LIMIT	ADEQUATE SEPARATION
1002	V3700-F	09/18/92	1540	INDA	13.58	Y	Y
1002	V3700-F	09/18/92	1800	INDA(TEST)	10.91	N	N

Comments:

PROJECT: PROGRAM TEST DATE: 09/24/92  
 ANALYSIS: P P - CALIBRATION PERCENT BREAKDOWN  
 REVIEWER: DENNIS MARTY DATA VALIDATION LEVEL: D  
 BEGINNING SAMPLE #: 1000 ENDING SAMPLE #: 1007

SDG	INSTRUMENT NUMBER	CAL DATE	PERCENT BREAKDOWN DDT	LIMIT DDT	PERCENT BREAKDOWN ENDRIN	LIMIT ENDRIN	PERCENT BREAKDOWN COMBINED	LIMIT COMBINED
1002	V3700-F	09/24/92	0.00	T	***		***	

Comments:

Fig. 18. P\_P instrument performance reports.

chromatogram or, for DDT, the presence of dichlorodiphenyldichloroethane (DDD) and/or dichlorodiphenylethane (DDE) is evidence of this degradation. A combined percent breakdown must be calculated if there is evidence of a peak at the RT of endrin aldehyde/DDD. Peak areas for these various compounds, listed as calibration factors (CFs) on Form VI, are entered into the program, and DATAVAL performs the calculations [Appendix F, Eq. (3), Eq. (4)]. DATAVAL inserts T/F in the LIMIT column for whether or not the results were within limits.

A final check is made by noting on Form X the percent difference (%D) in RT for dibutylchloroendate (DBC), if given, in all standards and samples. The guidelines are:  $\leq 2.0\%$  for packed column analysis,  $\leq 0.3\%$  for capillary column analysis, and  $\leq 1.5\%$  for wide-bore capillary column analysis (U.S.EPA 1988a). These results are entered into the program as numerical answers or as direct T/F answers. If numerical answers are given, DATAVAL calculates the %D between the RT of DBC in the initial standard and the RT of DBC in a subsequent analysis [Appendix F, Eq. (5)] and supplies the correct statements to the report.

### **4.3 CALIBRATION REPORTS**

Calibration of an instrument ensures that the instrument is capable of producing a linear calibration curve and acceptable qualitative and quantitative results. Calibration reports, which include both initial and continuing calibrations, are required for all analyses and vary somewhat depending upon the analysis.

#### **4.3.1 VOL, BNA, and H\_A**

Initial and continuing calibration reports for the analyses of volatile organic compounds, semivolatile organic compounds, and volatile halocarbon and aromatic compounds are identical.

### Initial Calibration Report

For the initial calibration report, the validator checks the relative response factors (RRFs), the average RRF, and the percent relative standard deviations (%RSDs) for all compounds on Form VI in the DSD. After entering the calibration date, the validator calls up the TC library for that analysis. Any compound on Form VI with an RRF less than 0.05 (or close to this value) or a %RSD greater than 30% (or close to this value) is highlighted in the TC library and thus entered into the initial calibration report. For those compounds picked, the validator enters RRF1 to RRF5 data and average RRF data (RRFI, Fig. 19) into the validation report from Form VI. The program calculates the mean RRF and %RSD for these compounds and compares the results to the limits, 0.05 and 30% (U.S.EPA 1988a), in the TC library [Appendix F, Eqs. (6), (7), (8)]. Any compounds outside the limits are flagged by the program in the lab data base. Whether or not the %RSD was within limits is flagged automatically (T/F) by the program in the CHK %RSD column in the report.

If all data appear to be within limits, the validator randomly selects from the TC library at least two TCL compounds for use in verifying the laboratory calculations. These compounds will also appear on the report. The validator must then enter into the initial calibration report (T/F) whether or not the DATAVAL average RRF calculation result (RRFC) is greater than 0.05 (CHKC column). With time and experience, validators can scan Form VI and choose questionable calculations. For the %RSD to be less than 30%, the range of RRFs must be fairly close. A compound with a number well outside the range of the other numbers in its group of RRFs may have a %RSD greater than 30% and should be checked. A compound with a group of wide-ranging RRFs is another candidate for calculation errors.

PROJECT: PROGRAM TEST  
 ANALYSIS: VOL - INITIAL CALIBRATION  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92

DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

CAL DATE	COMPOUND	SDG	RRF1	RRF2	RRF3	RRF4	RRF5	RRFC	CHKC	RRF1	%RSD	CHK	%RSD
09/20/92	2-BUTANONE	1002	0.173	0.215	0.205	0.219	0.225	0.207	T	0.207	9.9	T	
09/20/92	CARBON DISULFIDE	1000	1.259	1.548	1.639	1.730	1.706	1.576	T	1.576	12.1	T	
09/20/92	ETHYLBENZENE	1000	0.415	0.464	0.453	0.465	0.445	0.448	T	0.448	4.6	T	
09/20/92	TRANS-1,3-DICHLOROPROPENE	1002	0.221	0.287	0.327	0.383	0.417	0.327	T	0.327	23.7	T	

Comments:

PROJECT: PROGRAM TEST  
 ANALYSIS: BNA - INITIAL CALIBRATION  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92

DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

CAL DATE	COMPOUND	SDG	RRF1	RRF2	RRF3	RRF4	RRF5	RRFC	CHKC	RRF1	%RSD	CHK	%RSD
09/18/92	BENZYL ALCOHOL	1000	1.320	1.353	1.385	1.290	1.255	1.321	T	1.321	3.9	T	
09/18/92	DIBENZOFURAN	1000	1.924	1.885	1.898	1.749	1.608	1.813	T	1.813	7.3	T	
09/20/92	BENZO(K)FLUORANTHENE	1002	1.981	1.702	1.248	1.103	1.169	1.441	T	1.441	26.6	T	
09/20/92	BENZOIC ACID	1002	0.000	0.165	0.234	0.251	0.229	0.220	T	0.220	17.2	T	

Comments:

PROJECT: PROGRAM TEST  
 ANALYSIS: H A - INITIAL CALIBRATION  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92

DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

CAL DATE	COMPOUND	SDG	RRF1	RRF2	RRF3	RRF4	RRF5	RRFC	CHKC	RRF1	%RSD	CHK	%RSD
09/18/92	BROMOMETHANE	1000	1.125	1.135	1.142	1.151	1.181	1.147	T	1.148	1.9	T	
09/18/92	ETHYL BENZENE	1000	0.892	0.915	0.952	0.991	1.205	0.991	T	1.058	12.7	T	
09/20/92	CARBON TETRACHLORIDE	1002	1.450	1.520	1.485	1.602	1.750	1.561	T	1.559	7.7	T	

Comments:

Fig. 19. Initial calibration reports for VOL, BNA, and H\_A.

### Continuing Calibration Report

These calibrations are usually performed at the end of each run of samples or every 8-h period, whichever is less. The criteria are that RRFs must be  $\geq 0.05$  and %Ds between initial and continuing calibration average RRF values must be  $\leq 25\%$  (U.S.EPA 1988a). Compounds from Form VII are picked in the TC library for entry into the continuing calibration report. Those compounds with average RRFs  $\leq 0.05$  and/or %D  $\geq 25\%$  are entered along with the laboratory values for initial calibration average RRF (RRFI on the report, Fig. 20) and continuing calibration average (RRFC on the report). DATAVAL calculates the %D between these two values [Appendix F, Eq. (9)]. The program indicates whether or not the compounds are within the limits in the LIMIT column (T/F) and enters flags into the lab data base if necessary.

Several other compounds with either good or questionable values are selected for input into the report. Good values illustrate that the instrument is working for the majority of compounds; questionable values are included to verify laboratory calculations.

### 4.3.2 P\_P

#### Initial and Continuing Calibration

Initial calibration for level C validation is similar to VOL, BNA, and H\_A. However, the initial calibration for level D validation differs in that the %RSD of CFs for aldrin, endrin, DDT, and DBC must not exceed 10% (U.S. EPA 1988a). These CFs are entered into the program from Form VIII, and DATAVAL calculates and checks the %RSD against the required limit [Appendix F, Eqs. (6), (7), and (8), where  $X$  is CF]. If initial calibration requirements are not met, DATAVAL inserts F in the COMP column of the report (Fig. 21). CFs are shown in the LOW, MEDIUM, and HIGH columns of the report.

PROJECT: PROGRAM TEST  
 ANALYSIS: VOL - CONTINUING CALIBRATION  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92  
 DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

CAL DATE	TIME	COMPOUND	SDG	RRF1	RRFC	% D	LIMITS
09/24/92	0030	1,1,1-TRICHLOROETHANE	1000	0.783	0.334	57.3	F
09/24/92	0030	BROMODICHLOROMETHANE	1000	0.526	0.365	30.6	F
09/24/92	0030	BROMOFORM	1000	0.539	0.272	49.5	F
09/24/92	0030	BROMOMETHANE	1000	0.795	1.022	-28.6	F
09/24/92	0030	CARBON DISULFIDE	1000	1.576	0.984	37.6	F
09/24/92	0030	CIS-1,3-DICHLOROPROPENE	1000	0.396	0.297	25.0	F
09/24/92	0030	VINYL ACETATE	1000	0.556	0.354	36.3	F
09/24/92	0045	TRANS-1,3-DICHLOROPROPENE	1002	0.327	0.233	28.7	F
09/24/92	0045	TRICHLOROETHENE	1002	0.444	0.333	25.0	T

Comments:

PROJECT: PROGRAM TEST  
 ANALYSIS: BNA - CONTINUING CALIBRATION  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92  
 DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

CAL DATE	TIME	COMPOUND	SDG	RRF1	RRFC	% D	LIMITS
09/24/92	0000	2-NITROANILINE	1000	0.604	0.430	28.8	F
09/24/92	0000	BENZO(G,H,I)PERYLENE	1000	1.417	0.480	66.1	F
09/24/92	0000	BENZOIC ACID	1000	0.269	0.119	55.8	F
09/24/92	0000	BENZOIC ACID	1002	0.220	0.147	33.2	F
09/24/92	0000	BIS(2-CHLOROISOPROPYL)ETHER	1002	3.349	2.478	26.0	F
09/24/92	0000	DIBENZ(A,H)ANTHRACENE	1000	1.250	0.700	44.0	F
09/24/92	0000	INDENO(1,2,3-CD)PYRENE	1000	1.631	0.744	54.4	F
09/24/92	0000	N-NITROSO-DI-N-PROPYLAMINE	1002	1.164	0.797	31.5	F

Comments:

PROJECT: PROGRAM TEST  
 ANALYSIS: H A - CONTINUING CALIBRATION  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92  
 DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

CAL DATE	TIME	COMPOUND	SDG	RRF1	RRFC	% D	LIMITS
09/24/92	0000	TETRACHLOROETHENE	1000	1.123	1.215	-8.2	T
09/24/92	0000	TRANS-1,2-DICHLOROETHENE	1000	0.892	1.254	-40.6	F
09/24/92	1120	DICHLORO(1)FLUOROMETHANE	1002	1.250	0.895	28.4	F

Comments:

Fig. 20. Continuing calibration reports for VOL, BNA, and H\_A

PROJECT: PROGRAM TEST  
 ANALYSIS: P P - INITIAL CALIBRATION  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #: 1000

DATE: 09/24/92  
 DATA VALIDATION LEVEL: D  
 ENDING SAMPLE #: 1007

SDG	MIXTURE	COMPOUND	DATE	TIME	LOW	MEDIUM	HIGH	AVERAGE	% RELATIVE STANDARD DEVIATION	COMP
1002	A	GAMMA-BHC (LINDANE)	09/18/92	2200	1000	1750	2250	1667	37.7	F

PROJECT: PROGRAM TEST  
 ANALYSIS: P P - CONTINUING CALIBRATION  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #: 1000

DATE: 09/24/92  
 DATA VALIDATION LEVEL: D  
 ENDING SAMPLE #: 1007

SDG	MIXTURE	COMPOUND	DATE	TIME	R1	R2	PERCENT DIFFERENCE	LIMIT	QUANTITATED/ CONFIRMED
1002	A	HEPTACHLDR	09/24/92	1000	1125	1178	-4.7	T	q

Comments:

Fig. 21. Initial and continuing calibration reports for P\_P.

Continuing calibration requires that DATAVAL calculate the %D between initial CFs and subsequent CFs (R1 and R2 on the report) for approximately 10% of the reported values [Appendix F, Eq. (10)]. For P\_P analysis, either a GC column providing quantitative determinations or one for only qualitative confirmation may be used. This is indicated in the QUANTITATED/CONFIRMED column on the report. The limits are that the %D must be  $\leq 15\%$  for compounds being quantitated and  $\leq 20\%$  for compounds being confirmed (U.S.EPA 1988a).

Several compounds from the TC library, along with their respective CFs from Form IX, are entered into the program. The calculations and comparisons to the required limits are completed by the program and the results entered into the LIMIT column. Examples of initial and continuing calibration reports for P\_P are shown in Fig. 21.

### 4.3.3 PHC

Petroleum hydrocarbon compounds are included, under EPA guidelines, in the VOL analysis. Calibration reports for data from samples analyzed according to EPA method 8015 (U.S.EPA 1986) are identical to those outlined above for volatile and semivolatile compounds. HAZWRAP, however, has separate level C data validation requirements for results from samples analyzed for petroleum hydrocarbons using EPA method 418.1 (U.S.DOE 1990). In this case, the laboratory performs a three- to five-point standard curve bracketing sample concentration, using the method of least squares to plot concentration (amount) vs response (area).

In order to determine the type of curve to use for quantification (linear or quadratic), the laboratory calculates the %RSD of the response factors (areas of the peaks). If the %RSD is less than 20%, the linear curve is used. If the %RSD exceeds 20%, a quadratic curve is used. These limits are contained on QC sheets provided in the DSD by the laboratory. The correlation coefficient must fall within  $\pm 0.995$  in order for the calibration to be acceptable (U.S.DOE 1990).

The data for this calibration check are found in the DSD for PHC analysis on a form designated as a calibration summary. After entering the calibration date, the validator chooses the compounds used for calibration from the TC library and enters data pairs from the calibration summary. DATAVAL calculates the correlation coefficient and the %RSD [Appendix F, Eqs. (14) and (8)] and determines the curve type to use for calibration. The program performs a linear or quadratic curve fit of the data and reports the resulting constants as NUM1 and NUM2 [Appendix F, Eqs. (6), (11), (12), (13), and (28)]. The validator compares these to corresponding numbers reported by the laboratory. The LIMIT column of the report shows a Y/N answer to whether or not the correlation coefficient falls within the HAZWRAP limits (Fig. 22). The program also flags compounds in the lab data base that are affected by out-of-limits correlation coefficients.

#### 4.3.4 MET

Calibration reports for the analysis of metals require both initial and continuing calibration checks and cover four analyses: 1) mercury (Hg) analysis, 2) cyanide (CN<sup>-</sup>) analysis, 3) atomic absorption analysis (AA), and 4) inductively coupled plasma (ICP) analysis. The initial calibration report called "CAL (Curve Validation)" on the MET report selection menu, requires that a calibration curve be established for each of these analyses by running standards and blanks. For Hg and CN<sup>-</sup>, data pairs for these curves are found on Form II, Part 1, under Initial Calibration, True and Found. At least four data pairs from these forms are entered into DATAVAL, which then calculates the correlation coefficients and compares them to the limit,  $\geq 0.995$  (U.S.EPA 1988b) [Appendix F, Eqs. (6), (11), (14)]. The curve type (Hg or CN<sup>-</sup>), correlation coefficient, and limit evaluation (LIMIT column, T/F) are printed on the validation report (Fig. 23).

PROJECT: PROGRAM TEST  
 ANALYSIS: PHC - CALIBRATION  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #: 1100

DATE: 09/28/92  
 DATA VALIDATION LEVEL: D  
 ENDING SAMPLE #: 1103

SDG	METHOD	COMPOUND	DATE	TIME	CORR CORFF.	LIMIT	NUM1	NUM2	%RSD
1100	HBHC	HBPC (TO JP-5 JET FUEL)	09/28/92	1000	0.99232	N	582.0872	-0.1819	76.4
1100	LBHC	BENZENE	09/28/92	1000	0.99986	Y	*****	64.3309	79.0

If %RSD <= 20%, then a linear data fit ( $y=mx+b$ ) is employed, with  $m = \text{NUM1}$  and  $b = \text{NUM2}$ .  
 If %RSD > 20%, then a quadratic fit ( $y=a+bx+cx^2$ ) is employed, with  $a = 0$ ,  $b = \text{NUM1}$  and  $c = \text{NUM2}$ .

Comments:

Fig. 22. PHC calibration report.

PROJECT: PROGRAM TEST  
 ANALYSIS: MET - CAL (Curve Validation)  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92  
 DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

SDG	CURVE TYPE	CORRELATION COEFFICIENT	LIMIT
1002	AA	0.99663	T
1002	ICP	0.99959	T
1002	MERCURY	0.98917	F

Comments:

PROJECT: PROGRAM TEST  
 ANALYSIS: MET - CALIBRATION  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92  
 DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

SDG	COMPOUND TYPE	COMPOUND	TRUE CONCENTRATION	FOUND CONCENTRATION	LAB PERCENT RECOVERY	CAL PERCENT RECOVERY	COMP	LIMIT
1002	ICP	ARSENIC	4000.00	3778.50	94.50	94.46	T	T
1002	ICP	CALCIUM	40000.00	41733.22	104.30	104.33	T	T

Comments:

Fig. 23. Calibration reports for MET.

For AA and ICP analyses, the validator enters five data pairs from Form II, Part 2, for any element from the CRDL (contract-required detection limit) column, True and Found. As above, the program calculates the correlation coefficients and shows the evaluation on the report.

Continuing calibration (CALIBRATION on the MET report selection menu) requires a calculation of percent recovery (%R). Several analytes are chosen from Form II, Part 1 - both those with results outside the limits and those with good results - and are entered into the program along with analysis type (M column on the form) and the initial and continuing recoveries for each analyte. Values for Hg and CN<sup>-</sup>, if present, are also entered. DATAVAL calculates the %R for these analytes and flags results appropriately [Appendix F, Eq. (15)]. Analytical results must fall within the control limits or 90 to 110 %R for all analytes except mercury and cyanide. The limits for mercury and cyanide are 80 to 120 %R and 85 to 115 %R. Analysis type, initial and continuing analyte concentrations (TRUE and FOUND), and %R (both laboratory-calculated and DATAVAL-calculated) are shown on the calibration report (Fig. 23). If the laboratory and DATAVAL agree on the calculation, T is shown in the COMP column. Whether or not analytes are within limits is shown in the LIMITS column (T/F).

#### 4.3.5 ANI

Data validation for the analysis of anions is not included in EPA or HAZWRAP guidelines. The ANI reports were added to DATAVAL at the request of HAZWRAP for a particular project. Formatting for these reports is most similar to that of VOL and BNA reports, although the calibration report for ANI is more like the continuing calibration report for MET. All limits used for ANI reports are specified on forms provided by the analytical laboratory in the DSD.

Calibration for ANI requires a calculation of relative percent difference (RPD) between true concentrations and found (the analytical result) concentrations [Appendix F, Eq. (16)]. Several analytes are chosen from the laboratory-provided form, both those with results outside the limits and those with good results. These are entered into the program along with compound type (M column on the laboratory report) and the true and found concentrations for each analyte. DATAVAL calculates the RPD for these analytes and flags results appropriately. Whether or not analytes are within limits is shown on the report in the LIMITS column (T/F) (Fig. 24). The COMP column indicates whether the laboratory-calculated and DATAVAL-calculated RPDs are the same.

#### 4.4 BLANKS REPORT

Laboratory and field blank analyses are performed to determine the existence and magnitude of contamination due to laboratory or field activities. Blank reports give these results for all the different types of blanks run for each type of analysis. The report consists of two pages: one for contaminated blanks and one for blanks with no contamination. An example of each page for VOL analysis is shown in Fig. 25.

Blank information found in the HT report (TB and FB) is automatically inserted into the blanks report by the program. Method blank contamination can be inserted either electronically or manually. Data from Form III are used for MET and ANI analyses; data for all other analyses are reported on Form IV.

DATAVAL sorts all blanks with contamination, locates samples associated with contaminated blanks, determines the logic that applies for flagging, and flags the analytical results. The program also determines that the matrix is the same for the blank and its associated samples. RTs are reported for any TIC where there is

PROJECT: PROGRAM TEST  
ANALYSIS: ANI - CALIBRATION  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #: 1000

DATE: 09/24/92  
DATA VALIDATION LEVEL: D  
ENDING SAMPLE #: 1007

SDG	COMPOUND TYPE	COMPOUND	TRUE CONCENTRATION	FOUND CONCENTRATION	LAB RPD	CALCULATED RPD	COMP	LIMIT
1002	ICP	FLUORIDE	11.00	10.00	9.52	9.52	T	F

Comments:

Fig. 24. ANI calibration report.

PROJECT: PROGRAM TEST  
 ANALYSIS: VOL - BLANKS  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92  
 DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

BLANK NUMBER	SAMPLE TYPE	COMPOUND	RT	TCL or TIC	CONCENTRATION	UNITS	QCODE
VOL2MB	MB	1,1,1-TRICHLOROETHANE		TCL	10.00	µg/kg	J
VOL1MB	MB	1,1,1-TRICHLOROETHANE		TCL	5.00	µg/L	J

Comments:

PROJECT: PROGRAM TEST  
 ANALYSIS: VOL - BLANKS  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92  
 DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

BLANK NUMBER	SAMPLE TYPE	SDG	MATRIX
1006	FB	1002	W
1007	TB	1002	W
VOL2MB	MB	1000	S
VOL1MB	MB	1002	W

Comments:

Fig. 25. VOL blanks report, pages 1 and 2.

a chance of confusing two or more compounds. Laboratory-reported Q codes are shown on the report.

When the program finds a contaminant compound in both a sample and its associated blank, the 5X/10X rule is applied for VOL, BNA, PHC, and H\_A analyses and the 5X rule for P\_P, MET, and ANI (U.S.EPA 1988a, 1988b). Contaminant compounds are divided into two groups: regular compounds and a group of 5 compounds that are identified as common laboratory contaminants. EPA guidelines state that a contaminant compound should not be reported as such unless the concentration of the compound in the sample exceeds 10 times the amount in any blank for the common laboratory contaminants or 5 times the amount for other compounds. Compounds and their multipliers are provided by the appropriate TC library. Both the compound name and the RT must match between sample and blank. DATAVAL multiplies the blank contaminant concentration by 5 for regular compounds or by 10 if the compound is in the common laboratory contaminant group and compares the result to the sample concentration [Appendix F, Eqs. (17) and (18)]. If the sample concentration is greater than this calculated value, no flags are applied to the sample. However, if the sample concentration is less than this value, appropriate flags are inserted into the results according to the flagging logic.

#### **4.5 SURROGATE RECOVERY**

Individual samples and method blanks for VOL, BNA, PHC, and P\_P analysis are spiked with surrogate compounds prior to sample preparation to establish laboratory performance. Laboratory control samples (LCSs) may also be spiked for surrogate recovery check. The evaluation of the results of these surrogate spikes is often subjective, requiring experience and professional judgement.

For VOL, BNA, and PHC, the validator reviews both raw data and Form II of the DSD and answers the following questions, true or false, posed by

DATAVAL in the surrogate recovery report (Fig. 26):

- 1) Were recoveries on Form II verified?
- 2) Were all recoveries  $\geq 10\%$ ?
- 3) Was surrogate recovery a problem?
- 4) If 3) is T, is there evidence of purging, reinjection, or re-extraction?
- 5) Were there two blanks with surrogates outside criteria?
- 6) Were there two or more analyses for a fraction?

If an F appears on the report for question 2, the validator investigates compounds related to the surrogate and decides whether to flag sample results.

A different, smaller report is used for P\_P (Fig. 27). The following questions are answered:

- 1) Were recoveries on Form II verified?
- 2) If recoveries are not verified, is there evidence of interference?

These reports are then used during the final edit to flag results (Sect. 4.14).

#### 4.6 SPIKE REPORTS

Spike data (MS/MSD, MS, and blank spike) are generated to indicate the long-term precision and accuracy of the analytical method for various matrices; they cannot be used by themselves to evaluate the precision and accuracy of the individual samples. The results of the reports on this data are used in conjunction with other quality criteria in the final edit to determine the need for qualification of the data (Sect. 4.14).

PROJECT: PROGRAM TEST  
 ANALYSIS: BNA - SURROGATE RECOVERY  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #: 1000

DATE: 09/24/92  
 DATA VALIDATION LEVEL: D  
 ENDING SAMPLE #: 1007

SDG	QUESTION 1	QUESTION 2	QUESTION 3	QUESTION 4	QUESTION 5	QUESTION 6
1000	T	T	F		F	F
1002	T	T	F		F	F

Question 1) Were recoveries on form III verified?  
 Question 2) Were all recoveries  $\geq 10\%$ ?  
 Question 3) Was surrogate recovery a problem?  
 Question 4) If 3) is 1, is there evidence of purging, reinjection, or re-extraction?  
 Question 5) Were there two blanks with surrogates outside criteria?  
 Question 6) Were there two or more analyses for a fraction?

Comments:

Fig. 26. Surrogate recovery report for BNA.

PROJECT: PROGRAM TEST  
ANALYSIS: P P - SURROGATE RECOVERY  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92

DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

---

SDG	QUESTION 1	QUESTION 2
1002	T	

Question 1) Recoveries on form II were verified?  
Question 2) If recoveries are not verified, there is evidence of interference?

Comments:

Fig. 27. Surrogate recovery report for P\_P.

Spike report data are taken from Form III in the DSD for VOL, BNA, P\_P, and PHC. This form may contain both MS and MSD data or MS data only, depending on what the laboratory reports. QC limits for each spike are included on the form and are entered into TC libraries for each packet. MET data are reported on Form V. Because laboratories often confuse the forms used for spike sample reporting, the validator may have to pick which of the following reports best fits the data reported.

The type of spike report is chosen from the menu, and spike compound names are entered into the report from the TC library.

#### 4.6.1 MS/MSD Report

The following information is entered into the program from Form III:

- EPA sample number
- compound to be checked
- spike concentration added (the same for both MS and MSD)
- MS concentration found (matrix spike on the report)
- MSD concentration found (MSD on the report)
- RPD (from the laboratory).

DATAVAL calculates the %R for the MS and MSD [Appendix F, Eqs. (19) and (20)] and verifies (T/F) that these are within limits (columns MS VER and MSD VER). QC limits are shown on Form III. See Fig. 28 for an example of a MS/MSD report. DATAVAL flags the results associated with these samples if necessary. The program then calculates the RPD (CAL RPD) between MS and MSD recoveries [Appendix F, Eq. (21)], compares the result to the laboratory-reported RPD, and records whether or not the two values are the same (RPD VER, T/F).

PROJECT: PROGRAM TEST  
 ANALYSIS: ANI - MS/MSD  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92

DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

SAMPLE NUMBER	SAMP TYPE	SDG	COMPOUND	SPIKE ADDED	SAMPLE RESULT	MATRIX SPIKE	MSD	MS %	MS VER	MSD %	MSD VER	CAL RPD	RPD VER
1004	MS	1002	NITRATE, AS N	2.20	0.80	2.50	1.20	77.27	T	18.18	F	70	F

Comments:

Fig. 28. MS/MSD report for ANL

Even though an MS/MSD report form exists for MET, spike reports for this analysis always take the form of MS reports (Sect. 4.6.3).

#### 4.6.2 Blank Spike Report

An abbreviated form of the MS/MSD report, the blank spike report is used when only MS or blank spike data are reported by the laboratory. The following information is entered into the program from Form III or whatever other form may have been sent by the laboratory:

- EPA sample number
- spike concentration added
- blank spike concentration found (blank spike on the report, Fig. 29)

DATAVAL calculates the %R for the blank spike [Appendix F, Eq. (22)] and verifies (T/F) that these are within limits.

#### 4.6.3 MS Report

Although the format for this report is the same as the two previous reports, the input headings are different: Spike Sample Result (SSR), Sample Result (SR), and Spike Added (SA). See Fig. 29 for an example of an MS report. DATAVAL calculates the %R of the matrix spike [Appendix F, Eq. (23)] and reports (T/F) whether or not the results are within the limits found in the TC library. These limits are found either on the form sent by the laboratory or are taken from the EPA guidelines for inorganics analysis (U.S.EPA 1988b). In addition, the program verifies that the field blank was not used for the spike analysis.

PROJECT: PROGRAM TEST  
 ANALYSIS: VOL - BLANK SPIKE  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92

DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

SAMPLE NUMBER	SAMP TYPE	SDG	COMPOUND	SPIKE ADDED	SAMPLE CONCENTRATION	BLANK SPIKE	LAB BS % R	CAL % R	LIMIT
1000		1000	TRICHLOROETHENE	64.1000	0.0000	59.7000	93.0000	93.1	T

Comments:

PROJECT: PROGRAM TEST  
 ANALYSIS: VOL - MS  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92

DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

SAMPLE NUMBER	SAMP TYPE	SDG	COMPOUND	SSR	SR	SA	CAL % R	LIMIT
1000		1000	TOLUENE	58.5000	0.0000	64.10	91.3	T

Comments:

Fig. 29. Blank spike and MS reports for VOL.

#### 4.7 FIELD DUPLICATES REPORT

Duplicate sample analyses are used as indicators of laboratory precision based on each sample matrix. Field blanks cannot be used for this analysis; the program verifies this and indicates in the comments of the report if field blanks were used (Fig. 30).

At the FIELD DUPLICATES REPORT screen, the validator picks a sample number(s) and duplicate number(s). The program lists all the compounds found in both the sample and its duplicate. The validator picks several of these compounds for the report. RTs are reported for any TIC where there is a chance of confusing two or more compounds. DATAVAL calculates the RPD between the sample and the duplicate [Appendix F, Eq. (24)]. The validator's observations regarding the results appear in the comments section of the report.

#### 4.8 INTERNAL STANDARDS REPORT

GC/MS instruments require the use of an internal standard (IS) to determine that sensitivity and response are stable during every analytical run. IS reports are generated for VOL, BNA, and H\_A analyses.

The validator enters the Lab File ID from Form VIII and IS compound names from the TC library. He reviews Form VIII for the following and enters responses (T/F):

1. IS area counts for samples and blanks are within -50% to +100% of the associated calibration standard;
2. RTs of internal standards in samples and blanks are within  $\pm 30$  sec of the RT of the associated calibration standard.

DATAVAL flags any data as necessary. An example of an IS report is shown in Fig. 31.

PROJECT: PROGRAM TEST  
ANALYSIS: VOL - FIELD DUPLICATES  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

SDG	SAMPNUM	SAMPTYPE	DUPNUM	DUPTYPE	COMPOUND	RT	SAMP CON	DUP CON	RPD
1000	1000		1001	SR	1,1,1-TRICHLOROETHANE		100.00	135.00	29.79
1000	1000		1001	SR	2-BUTANONE		50.00	85.00	51.85
1000	1000		1001	SR	CARBON DISULFIDE		2500.00	2700.00	7.69
1002	1002		1003	WR	VINYL ACETATE		1500.00	1700.00	12.50

Comments:

Fig. 30. Field duplicates report for VOL.

PROJECT: PROGRAM TEST  
ANALYSIS: BNA - INTERNAL STANDARDS  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #: 1000

DATE: 09/24/92  
DATA VALIDATION LEVEL: D  
ENDING SAMPLE #: 1007

SDG	FORM NUMBER	DATE	TIME	COMPOUND	SAMPLE NUMBER	SAMPLE TYPE	AREA COUNTS	RETENTION TIME
1000	CG0329	09/24/92	0000	ACENAPHTHENE-d10	1000		T	T
1002	CV03222	09/24/92	0000	ACENAPHTHENE-d10	1002		T	T

Comments:

Fig. 31. Internal standards report for BNA.

#### **4.9 LAB DUPLICATES REPORT**

On occasion, a laboratory will send analytical results for diluted duplicates of samples. These diluted duplicates are most likely run because of high concentrations of compounds in the original sample. High concentrations can cause difficulties with the analysis, and the results are often suspect. When a package contains these results of diluted laboratory duplicates, the validator enters into this report the original sample number, the duplicate number, and the compounds associated with these samples. DATAVAL calculates [Appendix F, Eq. (24)] and reports the RPD for each compound (Fig 32). RTs are reported for any TIC where there is a chance of confusing two or more compounds.

There are no EPA or HAZWRAP requirements for this report; it is included in the package for the benefit of the project manager.

#### **4.10 SPECIAL REPORTS FOR MET AND ANI ANALYSES**

Four reports are used for MET and ANI analyses only:

1. ICP interference check sample (ICS) report;
2. LCS report;
3. ICP serial dilution report;
4. Furnace AA QC report (for MET only).

##### **4.10.1 ICP Interference Check Sample Report**

The ICS verifies the laboratory's interelement and background correction factors. Both an initial and final check are made. For this report, the validator must first review laboratory data to verify that the check sample (solution AB) was run at the beginning and end of each sample analysis run or a minimum of twice

PROJECT: PROGRAM TEST  
ANALYSIS: BNA - LAB DUPLICATES  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #: 1000

DATE: 09/24/92

DATA VALIDATION LEVEL: D  
ENDING SAMPLE #: 1007

SDG	SAMPNUM	SAMPTYPE	DUPNUM	DUPTYPE	DILUTION	COMPOUND	RT	SAMP CON	DUP CON	RPD
1000	1000		1000	DL	10.00	BIS(2-CHLORDETHOXY)METHANE		1750.00	1900.00	8.22
1002	1002		1002	DL	50.00	N-NITROSO-DI-N-PROPYLAMINE		50.00	42.00	17.39

Comments:

Fig. 32 Lab duplicates report for BNA.

per 8-h working shift, whichever is more frequent. Next, the validator picks several samples at random and enters the laboratory results for those samples from Form IV. DATAVAL calculates the %R [Appendix F, Eq. (25)] and indicates whether the result falls within  $\pm 20\%$  of the true value (U.S.EPA 1988b). This is shown in the INIT LIMIT and FIN LIMIT columns of the report (Fig. 33). The program also flags any data that do not meet the criteria in the lab data base.

#### 4.10.2 LCS Report

The LCS serves as a monitor of the overall performance of all steps in the analysis, including sample preparation. For this report, the validator must first review Form VII to verify that LCS results fall within %R control limits (U.S.EPA 1988b). Analyte names (from the TC library) and values for LCS "found" and LCS "true" are entered into the program. DATAVAL then calculates one or more %Rs to verify laboratory calculations [Appendix F, Eq. (15)]. The program indicates (T/F) in the COMP column on the report (Fig. 34) whether or not the laboratory-calculated %R matches the DATAVAL-calculated %R. The results are compared to EPA limits and data flagged where appropriate.

#### 4.10.3 ICP Serial Dilution Report

Serial dilution of samples analyzed by ICP indicates if significant physical or chemical interferences exist due to sample matrix. Analyte names from the TC library and sample and serial dilution results from Form IX are entered into the report. DATAVAL calculates the %D between the initial sample (I) and the serial dilution (S) [Appendix F, Eq. (26)]; the two must agree within 10% (U.S.EPA 1988b). The program then compares the result to the laboratory-calculated %D and displays the results of this comparison in the COMP column in the report (Fig. 35). The validator must use professional judgement to qualify (flag) the data if evidence of negative interference is found.

PROJECT: PROGRAM TEST  
 ANALYSIS: MET - ICP INTERFERENCE  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #: 1000

DATE: 09/24/92  
 DATA VALIDATION LEVEL: D  
 ENDING SAMPLE #: 1007

SDG	COMPOUND	SOLUTION	TRUE CON	INITIAL FOUND	CAL INITIAL PERCENT RECOVERY	INIT LIMIT	FINAL FOUND	CAL FINAL PERCENT RECOVERY	FIN LIMIT
1002	ALUMINUM	A	500000.00	493941.00	98.8	T	494479.00	98.9	T
1002	MAGNESIUM	AB	491000.00	494219.60	100.7	T	484706.80	98.7	T

Comments:

Fig. 33. ICP interference check sample report for MET.



PROJECT: PROGRAM TEST  
ANALYSIS: MET - ICP SERIAL DILUTION  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #: 1000

DATE: 09/24/92  
DATA VALIDATION LEVEL: D  
ENDING SAMPLE #: 1007

SDG	COMPOUND	EPA SAMPLE NUMBER	INITIAL SAMPLE RESULT	SERIAL DILUTION RESULT	LAB PERCENT DIFFERENCE	CALCULATED PERCENT DIFFERENCE	LAB VS CAL COMPARISON	LIMITS
1002	LITHIUM HYDRIDE	12430F	50.00	57.00	14.0	14.00	T	F

Comments:

Fig. 35. ICP serial dilution report for MET.

#### 4.10.4 Furnace AA QC Report

In order to establish the precision and accuracy of the individual analytical determinations, duplicate injections and furnace post-digestion spikes are run on the furnace AA. Data for this report are taken from Form II (Part 2) for inorganics analysis, "CRDL Standard for AA and ICP." Using the "True" and "Found" concentrations for a given analyte, DATAVAL calculates the %R [Appendix F, Eq. (15)] and compares the result to the laboratory-calculated result. Whether or not the two compare is indicated in the COMP column of the report (Fig. 36). The LIMIT column indicates if the program-calculated %R is within the EPA requirements of  $\geq 85\%$  and  $\leq 115\%$  (U.S.EPA 1988b).

Often, results for duplicate injections of these standards are shown on additional copies of Form II. For a given analyte, several "Found" values are entered into the program, which then calculates a %RSD [Appendix F, Eqs. (6), (7), and (8)]. The result of this calculation and whether or not it is within the limits of  $\pm 20\%$  is shown on the report above the Comments section. This information is used for qualifying data during the final edit (Sect. 4.14).

#### 4.11 CONTAMINATION REPORT

Following all the data validation activities listed above, a final contamination report for each analysis is issued that collects and summarizes all previous validation actions. No input is required from the validator. The report consists of two pages for each analysis. The first page lists all TCL compounds and TIC detected, along with their concentrations. RTs are reported for any TIC where there is a chance of confusing two or more compounds. The second page shows the number of samples in which a particular compound has occurred and its high, low, and mean concentrations. This information is provided to project managers for such purposes as risk assessment. Figure 37 shows the two pages of the calibration report for the BNA analysis.



PROJECT: PROGRAM TEST  
 ANALYSIS: BNA - CONTAMINATION REPORT  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #: 1000

DATE: 09/24/92

DATA VALIDATION LEVEL: D  
 ENDING SAMPLE #: 1007

SAMPLE NUMBER	SAMPLE TYPE	SAMPLE DILUTION	SQG	COMPOUND	RT	TCL/ITC	CONCENTRATION	UNITS	Q FLAG
1000		1.00	1000	BIS(2-CHLOROETHOXY)METHANE		TCL	1750.00	µg/kg	
1000	DL	1.00	1000	BIS(2-CHLOROETHOXY)METHANE		TCL	1900.00	µg/kg	
1001	SR	1.00	1000	BIS(2-CHLOROETHOXY)METHANE		TCL	2200.00	µg/kg	
1002		1.00	1002	N-NITROSO-DI-N-PROPYLAMINE		TCL	50.00	µg/L	
1002	DL	1.00	1002	N-NITROSO-DI-N-PROPYLAMINE		TCL	42.00	µg/L	

PROJECT: PROGRAM TEST  
 ANALYSIS: BNA - CONTAMINATION REPORT  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #: 1000

DATE: 09/24/92

DATA VALIDATION LEVEL: D  
 ENDING SAMPLE #: 1007

TCL/ITC	COMPOUND	RT	MATRIX	NUMBER OF SAMPLES	HIGH CON	LOW CON	MEAN CON	IDL
TCL	BIS(2-CHLOROETHOXY)METHANE		S	3	2200.00	1750.00	1950.00	350.00
TCL	N-NITROSO-DI-N-PROPYLAMINE		W	2	50.00	42.00	46.00	10.00

Fig. 37. Contamination report for BNA.

The contamination report fulfills EPA and HAZWRAP requirements for special reports for several of the analyses (U.S.EPA 1988a, 1988b; U.S.DOE 1990):

- Target compound identification (VOL, BNA, PHC)
- Tentatively identified compounds (VOL, BNA, PHC)
- Sample result verification (MET)
- Compound identification (P\_P).

Examples of contamination reports for all seven analyses may be seen in Appendix E.

#### **4.11.1 Special Calibration Report for PHC**

Level D validation for petroleum hydrocarbon compounds requires a special calibration report to accompany the contamination report, in addition to other calibration reports (U.S.EPA 1988a). This report can indicate a growing instrument problem that could affect later samples. DATAVAL inserts this report format into the menu when level D is entered into the project identification.

Analytical instrument numbers and calibration dates and times are entered into the report, and DATAVAL compares each sample to the calibration report that immediately precedes the run. The relative response time (RRT) for each compound is listed on the report and must be within  $\pm 0.06$  of the calibration RRT (U.S.EPA 1988a). Compounds outside these limits are flagged in the table of the report and an explanation given at the bottom of the table. A compound may also be flagged as missing an interval of calibration, a situation where sample results are not enclosed by calibration results (initial and continuing calibrations). This usually occurs when the laboratory has neglected to send all calibration results.

The program then runs a 95% confidence interval check to detect possible input errors by the validator and to check for calibrations that are out of line with previous calibrations [Appendix F, Eqs. (6), (7), and (27) where  $X$  is RRT] (Ott 1988). Data outside the 2-sigma interval are flagged on the table and an explanation given at the bottom of the table.

Both Level C and Level D contamination reports for PHC are shown in Fig. 38.

#### **4.12 SYSTEM REVIEW REPORT**

EPA guidelines require that the validator evaluate the ongoing performance of the GC/MS system for VOL, BNA, H\_A, and PHC analyses using instrument performance indicators (U.S.EPA 1988a). This report is accessed at the report selection menu of each of the individual analyses. The validator inserts comments into the report indicating the level of performance for the system. A number of indicators that can supply insight into problems that may be building during a sample run may be used to make this evaluation. An example might be an initial calibration performed days or weeks before subsequent continuing calibrations.

#### **4.13 OVERALL ASSESSMENT REPORT**

Comments on the validity of the overall data package for VOL, BNA, H\_A, PHC, and P\_P analyses make up this report. When several QC criteria are out of specification and since these factors are often additive, it is the responsibility of the validator to inform users of questionable data quality and of data limitations to assist in avoiding inappropriate use of the data.

PROJECT: PROGRAM TEST  
 ANALYSIS: PHC - CONTAMINATION REPORT  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #: 1000

DATE: 09/25/92  
 DATA VALIDATION LEVEL: C  
 ENDING SAMPLE #: 1007

SAMPLE NUMBER	SAMPLE TYPE	SAMPLE DILUTION	SDG	COMPOUND	RT	TCL/TIC	CONCENTRATION	UNITS	D FLAG
1000		1.00	1000	BENZENE		TCL	200.00	mg/kg	
1000		1.00	1000	HBPC (TO JP-5 JET FUEL)		TCL	1500000.00	mg/kg	
1000	DL	1.00	1000	BENZENE		TCL	750.00	mg/kg	
1000	DL	1.00	1000	HBPC (TO JP-5 JET FUEL)		TCL	2750000.00	mg/kg	
1001	SR	1.00	1000	BENZENE		TCL	900.00	mg/kg	
1001	SR	1.00	1000	HBPC (TO JP-5 JET FUEL)		TCL	1750000.00	mg/kg	
1002		1.00	1002	ETHYL BENZENE		TCL	2000.00	µg/L	
1002		1.00	1002	MP XYLENE		TCL	20.00	µg/L	
1002		1.00	1002	O XYLENE		TCL	100.00	µg/L	

PROJECT: PROGRAM TEST  
 ANALYSIS: PHC - CONTAMINATION REPORT  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #: 1000

DATE: 09/25/92  
 DATA VALIDATION LEVEL: C  
 ENDING SAMPLE #: 1007

TCL/TIC	COMPOUND	RT	MATRIX	NUMBER OF SAMPLES	HIGH CON	LOW CON	MEAN CON	IDL
TCL	BENZENE		S	3	900.00	200.00	616.67	0.05
TCL	ETHYL BENZENE		W	1	2000.00	2000.00	2000.00	NA
TCL	HBPC (TO JP-5 JET FUEL)		S	3	2750000.00	1500000.00	2000000.00	10.00
TCL	MP XYLENE		W	1	20.00	20.00	20.00	NA
TCL	O XYLENE		W	1	100.00	100.00	100.00	NA

PROJECT: PROGRAM TEST  
 ANALYSIS: PHC - CONTAMINATION REPORT (SRV)  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #: 1000

DATE: 09/24/92  
 DATA VALIDATION LEVEL: D  
 ENDING SAMPLE #: 1007

## STANDARDS DATA

SDG	INST #	DATE	TIME	BENZENE	TOLUENE	D1 IST/CL BENZENE	ETHYL BENZENE	MP XYLENE	O XYLENE	D1 SUR/CL BENZENE	SOLVENT PEAK	D2 IST/CL BENZENE	D2 SUR/CL BENZENE
1002	1254	09/15/92	1430	1.14	2.45	2.98	3.41	3.79	7.24	9.82	0.91	2.75	8.56
1002	1254	09/15/92	1540	1.15	2.47	2.92	3.51	3.79	7.30	9.84	0.90	2.76	8.60
1002	1254	09/15/92	1640	1.10	2.42	3.01	3.39	3.77	7.19	9.86	0.88	2.76	8.51
1002	1254	09/15/92	1740	1.11	2.47	2.95	3.39	* 2.74	7.25	9.85	0.94	2.75	8.60
1002	1254	09/15/92	1840	1.15	2.48	2.99	3.41	3.80	7.20	9.80	0.95	2.78	8.62
1002	1254	09/15/92	1940	1.11	2.43	3.02	3.38	3.83	7.25	9.83	0.95	2.76	8.65
AVERAGE				1.13	2.45	2.98	3.42	3.62	7.24	9.83	0.92	2.76	8.59
SIGMA				0.02	0.02	0.04	0.05	0.43	0.04	0.02	0.03	0.01	0.05

\* = RRT outside of average ± 2 sigma.

Comments:

PROJECT: PROGRAM TEST  
 ANALYSIS: PHC - CONTAMINATION REPORT (SRV)  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #: 1000

DATE: 09/24/92  
 DATA VALIDATION LEVEL: D  
 ENDING SAMPLE #: 1007

## SAMPLE DATA

SDG	SAMPLE #	INST #	DATE	TIME	BENZENE	TOLUENE	D1 IST/CL BENZENE	ETHYL BENZENE	MP XYLENE	O XYLENE	D1 SUR/CL BENZENE	SOLVENT PEAK	D2 IST/CL BENZENE	D2 SUR/CL BENZENE
1002	1002	1254	09/15/92	1530	1.11					7.29				2.77
1002	1003	1254	09/15/92	1730	* 1.24			3.38				0.89	2.77	8.52
1002	1004	1254	09/15/92	1630			* 2.31							8.62
1002	1005	1254	09/15/92	1830	1.17			* 3.04						
1002	1006	1254	09/15/92	1930	1.20	2.44	3.02	3.38	3.81	7.25	* 9.68	0.90	2.80	8.60

\* = sample compound RRT outside of ±0.06 RRT calibration.  
 \* = sample does not have an interval of calibration.

Comments:

Fig. 38. Contamination report, including level D, for PHC.

#### 4.14 FINAL SUMMARY REPORT

The final summary report is edited and printed from the FINAL option on the main menu. Part of a final summary report is shown in Fig. 39. The validator enters the SUMMARY option and reviews all detected compounds. When an F flag appears with a compound, the validator consults the final flag table (Fig. 40) to determine the appropriate Q flag to insert for that compound. The field above the F code on the table is used to correlate F codes to Q codes. For any F flags that do not have fields that correlate to the final flags table, the validator must review data from various reports such as surrogate recovery and spike reports to determine the Q flag to apply. The Q codes chosen are those that appear in the Final Code column on the report (Fig. 39). The BR flag, "unusable due to blank contamination," is the only one the validator may not override.

Following this editing process, the final summary report is printed.

### 5. COVER SHEETS

Upon completion of data validation, each package is issued with two cover sheets, one for organics (Fig. 41) and one for inorganics (Fig. 42) analyses. These are generated from the FINAL option of the main menu. All analyses performed for that package are shown on the cover sheets; codes indicating the condition of the data are entered for each analysis. There are also three areas for comments that may be utilized by the validator.

PROJECT: PROGRAM TEST  
 Final Summary  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92

DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

SAMPLE NUMBER	SAMPLE TYPE	COMPOUND	RT	TCL/TIC	MATRIX	ANALYSIS TYPE	CONCENTRATION	UNITS	Q CODE	FINAL CODE
1000		1,1,1-TRICHLOROETHANE		TCL	S	VOL	100.00	µg/kg	J	J
1000		1,2-DICHLOROBENZENE		TCL	S	H_A	200.00	µg/kg		
1000		2-BUTANONE		TCL	S	VOL	50.00	µg/kg	J	J
1000		ARSENIC		TCL	S	MET	200.00	mg/kg		
1000		BENZENE		TCL	S	PHC	200.00	mg/kg		
1000		BIS(2-CHLOROETHOXY)METHANE		TCL	S	BNA	1750.00	µg/kg		
1000		CARBON DISULFIDE		TCL	S	VOL	2500.00	µg/kg		J
1000		CHROMIUM		TCL	S	MET	10.00	mg/kg	J	J
1000		ENDRIN KETONE		TCL	S	P_P	50.00	µg/kg		BR
1000		HBPC (TO JP-5 JET FUEL)		TCL	S	PHC	1500000.00	mg/kg		
1000	DL	1,1,1-TRICHLOROETHANE		TCL	S	VOL	125.00	µg/kg		J
1000	DL	1,2-DICHLOROBENZENE		TCL	S	H_A	225.00	µg/kg		
1000	DL	2-BUTANONE		TCL	S	VOL	75.00	µg/kg		
1000	DL	ARSENIC		TCL	S	MET	180.00	mg/kg		
1000	DL	BENZENE		TCL	S	PHC	750.00	mg/kg		
1000	DL	BENZENE		TCL	S	VOL	25.00	µg/kg		
1000	DL	BIS(2-CHLOROETHOXY)METHANE		TCL	S	BNA	1900.00	µg/kg		
1000	DL	CARBON DISULFIDE		TCL	S	VOL	3000.00	µg/kg		J
1000	DL	CHROMIUM		TCL	S	MET	12.00	mg/kg	J	J
1000	DL	ENDRIN KETONE		TCL	S	P_P	35.00	µg/kg		BR
1000	DL	HBPC (TO JP-5 JET FUEL)		TCL	S	PHC	2750000.00	mg/kg		
1001	SR	1,1,1-TRICHLOROETHANE		TCL	S	VOL	135.00	µg/kg		J
1001	SR	1,2-DICHLOROBENZENE		TCL	S	H_A	320.00	µg/kg		
1001	SR	2-BUTANONE		TCL	S	VOL	85.00	µg/kg		
1001	SR	ARSENIC		TCL	S	MET	325.00	mg/kg		
1001	SR	BENZENE		TCL	S	PHC	900.00	mg/kg		
1001	SR	BIS(2-CHLOROETHOXY)METHANE		TCL	S	BNA	2200.00	µg/kg		
1001	SR	CARBON DISULFIDE		TCL	S	VOL	2700.00	µg/kg		J
1001	SR	CHROMIUM		TCL	S	MET	16.00	mg/kg	J	J
1001	SR	ENDRIN KETONE		TCL	S	P_P	32.00	µg/kg		BR
1001	SR	HBPC (TO JP-5 JET FUEL)		TCL	S	PHC	1750000.00	mg/kg		
1002		2-CHLOROETHYL VINYL ETHER		TCL	W	H_A	150000.00	µg/L		
1002		4,4'-DDD		TCL	W	P_P	75.00	µg/L		
1002		4,4'-DDE		TCL	W	P_P	20.00	µg/L		
1002		4,4'-DDT		TCL	W	P_P	50.00	µg/L		R
1002		CHLORIDE		TCL	W	ANI	25.00	mg/L		
1002		ETHYL BENZENE		TCL	W	PHC	2000.00	µg/L		
1002		FLUORIDE		TCL	W	ANI	30.00	mg/L		J
1002		MERCURY		TCL	W	MET	1200.00	µg/L		J
1002		MP XYLENE		TCL	W	PHC	20.00	µg/L		
1002		N-NITROSO-DI-N-PROPYLAMINE		TCL	W	BNA	50.00	µg/L		J
1002		O XYLENE		TCL	W	PHC	100.00	µg/L		
1002		SILVER		TCL	W	MET	100.00	µg/L		
1002		VINYL ACETATE		TCL	W	VOL	1500.00	µg/L		
1002		ZINC		TCL	W	MET	500.00	µg/L		
1002	DL	N-NITROSO-DI-N-PROPYLAMINE		TCL	W	BNA	42.00	µg/L		J
1002	DL	VINYL ACETATE		TCL	W	VOL	1250.00	µg/L		
1003	WR	VINYL ACETATE		TCL	W	VOL	1700.00	µg/L		

Fig. 39. Final summary report.

## FINAL FLAGS

VOL/BNA/PHC/H A		
FIELD	RESULT TO FLAG	FINAL Q CODE
RRFCCI	F	J
PRSDCI	F	J
PDCC	F	J
CCCP	F	J
MET/ANI		
FIELD	RESULT TO FLAG	FINAL Q CODE
CCCM	F	J OR UJ
NOTE: IF RESULT IS: 1. > IDL THEN J, OR 2. < IDL THEN UJ		
CPRCM2	F	J
CPRLCS	F	J
CPRICPI	F	J
CPDICPSD	F	J

Fig. 40. Final flags table.

REGION: \_\_\_\_\_

## ORGANIC REGIONAL DATA ASSESSMENT

VALIDATION LEVEL: D

CASE NO.: \_\_\_\_\_

SITE: PROGRAM TEST

NUMBER OF SAMPLES/MATRIX

WATER: 38 SOIL: 15

LABORATORY: ORNL

REVIEWER (IF NOT ESD): \_\_\_\_\_

REVIEWER'S NAME: DENNIS MARTY

COMPLETION DATE: 09/24/92

SAMPLES START #: 1000

END #: 1007

## DATA ASSESSMENT SUMMARY

	VOA	BNA	PEST	H&A	ANIONS
1. HOLDING TIMES-----	0	0	0	0	0
2. GC/MS TUNE/INSTR. PERFORM-----	0	0			
3. CALIBRATIONS-----	0	0	0	0	0
4. BLANKS-----	0	0	0	0	0
5. SURROGATES-----	0	0	0	0	0
6. MATRIX SPIKE/DUP.-----	0	0	0	0	0
7. OTHER QC-----	0	0	0	0	0
8. INTERNAL STANDARDS-----	0	0	0	0	0
9. COMPOUND IDENTIFICATION-----	0	0	0	0	0
10. SYSTEM PERFORMANCE-----	0	0	0	0	0
11. OVERALL ASSESSMENT-----	0	0	0	0	0

0 = DATA HAD NO PROBLEMS/OR QUALIFIED DUE TO MINOR PROBLEMS.

M = DATA QUALIFIED DUE TO MAJOR PROBLEMS.

Z = DATA UNACCEPTABLE.

X = PROBLEMS, BUT DO NOT AFFECT DATA.

ACTION ITEMS: THIS IS A TEST SET OF DATA FOR THE PROGRAM.AREAS OF CONCERN:NOTABLE PERFORMANCE:

Fig. 41. Cover sheet for organics analyses.

REGION: \_\_\_\_\_

## INORGANIC REGIONAL DATA ASSESSMENT

VALIDATION LEVEL: D

CASE NO.: \_\_\_\_\_

LABORATORY: ORNL

SITE: PROGRAM TEST

NUMBER OF SAMPLES/MATRIX

WATER: 38 SOIL: 15

REVIEWER (IF NOT ESD): \_\_\_\_\_

REVIEWER'S NAME: DENNIS MARTY

COMPLETION DATE: 09/24/92

SAMPLES START #: 1000

END #: 1007

## DATA ASSESSMENT SUMMARY

	ICP	AA	HG	CYANIDE
1. HOLDING TIMES -----	0	0	0	
2. CALIBRATIONS -----	0	0	0	
3. BLANKS -----	0	0	0	
4. ICS -----	0			
5. LCS -----	0	0		
6. DUPLICATE ANALYSIS -----	0	0	0	
7. MATRIX SPIKE -----	0	0	0	
8. MSA -----		0		
9. SERIAL DILUTION -----	0			
10. SAMPLE VERIFICATION -----	0	0	0	
11. OTHER QC -----	0	0	0	
12. OVERALL ASSESSMENT -----	0	0	0	

0 = DATA HAD NO PROBLEMS/OR QUALIFIED DUE TO MINOR PROBLEMS.

M = DATA QUALIFIED DUE TO MAJOR PROBLEMS.

Z = DATA UNACCEPTABLE.

X = PROBLEMS, BUT DO NOT AFFECT DATA.

ACTION ITEMS:AREAS OF CONCERN:NOTABLE PERFORMANCE:

Fig. 42. Cover sheet for inorganics analyses.

## 6. PACKAGE DEFICIENCIES SUMMARY

The Package Deficiencies Summary outlines all the problem areas discovered during data validation for a specific package. This summary report lists the total number of samples in the package, the number of samples analyzed by each analysis type, QC samples as a percentage of the total number of samples, and several sections delineating deficiencies.

The first section names problem areas and lists the number of out-of-limit events for each area. The second section documents the ratio of the number of detected compounds for which data qualifiers have been changed to the total number of detected compounds. Both of these sections are on page 1 of the Summary (Fig. 43). This is followed by a third section (page 2, Fig. 44) containing notes that can be manually entered regarding the following:

1. Missing chain-of-custody forms
2. Illegible information (e.g., on laboratory report forms)
3. Information missing from laboratory report
4. Missing QC information
5. Transcription errors
6. Logbook errors
7. Request for analysis problems.

These problems, which may be detected by the validator while reviewing a data package, may have a direct bearing on the results.

The fourth section (page 3, Fig. 44) documents the nonconformance of calibrations for organics analyses, according to the requirements in the CLP functional guidelines (U.S.EPA 1991a). For example, for the VOL analysis, the report will list the number of compounds in nonconformance if the %RSDs of

PROJECT: PROGRAM TEST  
 LABORATORY: ORNL  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92  
 DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

PACKAGE DEFICIENCIES SUMMARY

Total number of samples: 8

Analysis Type	Number of Analyses
VOL - Volatiles:	8
BNA - Semivolatiles:	7
PHC - Petroleum Hydrocarbons:	7
MET - Metals:	7
P_P - Pesticides:	7
ANI - Anions:	5
H_A - Halocarbons and Aromatics:	5

Sample Type	QC Samples as a % of Total Samples
Duplicate QC water samples:	16.67%
Duplicate QC soil samples:	50.00%
MS/MSD QC water samples:	16.67%
MS/MSD QC soil samples:	0.00%

Problem	Number of Problems
1. Holding Times exceeded:	
A. Extraction Holding Times exceeded:	1
B. Analysis Holding Times exceeded:	1
2. Tuning problems (VOL & BNA):	0
3. Initial Calibration:	0
4. Continuing Calibration:	8
5. Surrogate Recovery outside of limits (level IV or D):	0
6. Method Blank contamination:	3
7. Trip Blank or Field Blank contamination:	0
8. MS/MSD Recovery:	0
9. Matrix Spike:	0
10. Blank Spike:	1
11. Internal Standards:	0
12. PHC Calibration:	0
13. Field Duplicate:	3
14. PEST/PCB Continuing Calibrations:	0
15. PEST/PCB Instrument Performance:	0
16. Metals Curve Validation:	1
17. Metals Calibration:	1
18. Laboratory Control Samples:	0
19. ICP Interference:	0
20. ICP Serial Dilution:	0
21. PHC Sample Result Verification:	0

Ratio of detects with changed flags to total detects: 13 / 48

Fig. 43. Package Deficiencies Summary, page 1.

PROJECT: PROGRAM TEST  
 LABORATORY: ORNL  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92  
 DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

PACKAGE DEFICIENCIES SUMMARY

Chain of Custody Missing: THIS IS A TEST SET FOR THIS SOFTWARE PACKAGE.

Illegible Information: THIS IS A TEST SET FOR THIS SOFTWARE PACKAGE.

Missing Information (Lab Report):

Missing QC Information:

Transcription Errors:

Logbook Problems:

Request for Analysis Problems:

PROJECT: PROGRAM TEST  
 LABORATORY: ORNL  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92  
 DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

PACKAGE DEFICIENCIES SUMMARY

NONCONFORMANCE CALIBRATIONS FOR ORGANIC ANALYSES

ANALYSIS TYPE	CAL TYPE	DATE	TIME	NUMBER OF COMPOUNDS		
				%RSD>40%	%RSD>30%	%RSD>25%
BNA	CONT	09/24/92	0000	4		7
H_A	CONT	09/24/92	0000	1		
VOL	CONT	09/24/92	0030	2		7

Fig. 44. Package Deficiencies Summary, pages 2 and 3.

more than two compounds exceed the limit of 30%. These nonconformance problems cannot be corrected on the current package but may be used for corrective purposes for future data sets.

The final section (page 4, Fig. 45) is an optional report that indicates any elevated detection limits that occurred during analysis. The program collects laboratory detection limits during the electronic transfer of laboratory data and reports these along with the contract-required detection limits extracted from the TC libraries (U.S.EPA 1989; U.S.EPA 1990). LIDL on the report is the laboratory-reported instrument detection limit; SIDL and WIDL are the contract-required detection limits for soil and water. An explanation from the laboratory, usually found in the case narrative of the DSD, may be shown in the comments section of the report. This report can be printed as part of the Package Deficiencies Summary or can be printed separately at a later date.

A procedure has been established for documenting and reporting a nonconformance problem to the responsible laboratory. This procedure and report may be seen in Appendix G. If possible, the laboratory will correct the nonconformance problem and resubmit data that will allow DATAVAL to revalidate that specific package.

## 7. PROGRAM TESTING

Several sets of data have been validated both manually and using DATAVAL in order to check the accuracy and integrity of the program. A test data set has been developed to bring into the validation process a number of the more obscure problems encountered during various validation projects. During validation of this test data, the program has performed well, catching situations that required hours of manual checking and that could have been easily overlooked. The program will continue to be evaluated for integrity during subsequent validation projects.

PROJECT: TEST

DATE:01/12/93

REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000DATA VALIDATION LEVEL:C  
ENDING SAMPLE #:1003

## Package Deficiencies Summary

## Instrument Detection Limit Report

ANALYSIS TYPE	COMPOUND	SAMPLE NUMBER	MATRIX	LIDL	SIDL	WIDL	CONCENTRATION	UNITS
BNA	1,2,4-TRICHLOROBENZENE	1000	W	100.00		10.00	200.00	µg/L
BNA	1,2-DICHLOROBENZENE	1000	W	25.00		10.00	500.00	µg/L
VOL	ACETONE	1000	W	100.00		10.00	5000.00	µg/L
VOL	CARBON DISULFIDE	1002	W	8.00		5.00	50.00	µg/L

Comments:

Fig. 45. Package Deficiencies Summary, page 4.

## REFERENCES

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## GLOSSARY

**Analyte** - the compound, element, or ion an analysis seeks to determine; the element of interest.

**Blank** - see Method Blank

**Calibration** - for MET and ANI analyses, the establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. Calibration standards must be prepared using the same type of acid or concentration of acids as used in sample preparation.

**Calibration Standards** - for MET and ANI analyses, a series of known standard solutions used by the analyst for calibration of the instrument, i.e., preparation of the analytical curve

**Continuing Calibration** - analytical standard run every 12 h to verify the calibration of the GC/MS system for VOL, BNA, H\_A, P\_P, and PHC analyses. For MET and ANI analyses, analytical standard run every 10 analytical samples or every 2 h, whichever is more frequent, to verify the calibration of the analytical system.

**Contract-Required Detection Limit (CRDL)** - minimum level of detection acceptable under the EPA CLP Inorganics Analysis Statement of Work (U.S.EPA 1990)

**Contract-Required Quantitation Limit** - the concentration of an analyte in a sample equivalent to the concentration of the lowest calibration standard analyzed for that analyte. This is associated with volatile and semivolatile organics analyses.

**Correlation Coefficient** - a number ( $r$ ) that indicates the degree of dependence between two variables (concentration vs absorbance) determined on the basis of the least squares line. The more dependent they are, the closer the value to one.

**Duplicate** - a second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method

**Field Blank** - any sample submitted from the field identified as a blank

**Holding Time** - for VOL, H\_A, PHC, MET, and ANI, the elapsed time, expressed in days, from the date of collection of the sample until the date of its analysis

$$\text{holding time} = (\text{sample analysis date}) - (\text{sample collection date})$$

For BNA and P\_P, holding time is related to the date of extraction

$$\text{extraction holding time} = (\text{sample extraction date}) - (\text{sample collection date})$$

$$\text{analysis holding time} = (\text{sample analysis date}) - (\text{sample extraction date})$$

**Initial Calibration** - analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the mass spectrometer or electron capture detector to the target compounds.

**Internal Standards** - compounds added to every standard, blank, matrix spike, matrix spike duplicate, sample (for VOL), and sample extract (for BNA) at a known concentration, prior to analysis. Internal standards are used as the basis for quantitation of the target compounds.

**Laboratory Control Sample (LCS)** - a control sample of known composition.

Aqueous and solid laboratory control samples are analyzed using the same sample preparation, reagents, and analytical methods employed for the EPA samples received.

**m/e (m/z)** - mass to charge ratio

**Matrix:** the predominant material of which the sample to be analyzed is composed. For the purpose of this document, a sample matrix is either water or soil/sediment. Matrix is not synonymous with phase (liquid or solid).

**Matrix Spike:** aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery

**Matrix Spike Duplicate** - a second aliquot of the same matrix as the matrix spike that is spiked in order to determine the precision of the method

**Method Blank** - an analytical control consisting of all reagents, internal standards, and surrogate standards that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.

**Percent Difference (%D)** - as used in this report and elsewhere to compare two values, the direction and the magnitude of the comparison; the percent difference may be either negative, positive, or zero

**Relative Percent Difference (RPD)** - as used in this report and elsewhere to compare two values, the relative percent difference is based on the mean of the two values and is reported as an absolute value, i.e., always expressed as a positive number or zero

**Relative Response Factor (RRF)** - a measure of the relative mass spectral response of an analyte compared to its internal standard. Relative response factors are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples.

**Sample** - a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number

**Sample Delivery Group (SDG)** - a unit used to identify a group of samples for delivery. An SDG is a group of 20 or fewer field samples, received over a period of up to 14 calendar days (7 calendar days for 14-day data turnaround contracts). Data from all samples in an SDG are due concurrently. At the discretion of the laboratory, samples may be assigned to an SDG by matrix, i.e., all soil samples in one SDG, all water samples in another).

**Sample Number (EPA Sample Number)** - a unique identification number designated by the EPA for each sample

**Semivolatile Compounds** - compounds amenable to analysis by extraction of the sample with an organic solvent. Used synonymously with base/neutral/acid (BNA) compounds.

**Surrogates (Surrogate Standard)** - for VOL, BNA, H\_A, PHC, and P\_P, compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard; used to evaluate analytical efficiency by measuring recovery. Surrogates are brominated, fluorinated, or isotopically labelled compounds not expected to be detected in environmental media.

**Target Compound List (TCL)** - a list of compounds designated for analysis by the EPA in Exhibit C of the CLP Statement of Work (U.S.EPA 1991b)

**Tentatively Identified Compounds (TIC)** - compounds detected in samples that are not target compounds, internal standards, system monitoring compounds, or surrogates. Up to 30 peaks (those greater than 10% of peak areas or heights of nearest internal standards) are subjected to mass spectral library searches for tentative identification.

**Tuning** - the GC/MS system used for analysis of volatile organic compounds must be tuned, using suitable compounds, to meet the manufacturer's specifications. Tuning and performance criteria are established to ensure mass resolution, identification and, to some degree, sensitivity. The results of this activity are provided on the tuning report.

**Volatile Compounds** - compounds amenable to analysis by the purge and trap technique.

Sources: U.S.EPA 1990, 1991b.

**APPENDIX A**

**REPORTS REQUIRED FOR VALIDATION LEVELS**



**APPENDIX A**  
**REPORTS REQUIRED FOR VALIDATION LEVELS**

Listed below are the data validation reports, by analysis, required for each validation level.

The lab duplicates report is an informational report included for every analysis at all levels. Field duplicates are a requirement in the field testing program but are not required for levels B (II) and C (III) validations. Since the duplicate samples are taken in the field, the field duplicates report is completed for these levels. Two reports included for all analyses at every level as a summary of the validation package are the contamination report and the system review.

**LEVEL B or II**

**VOL (GC/MS)**

Holding Times  
Calibration  
Blanks  
MS/MSD, MS, or BS  
Overall Assessment

**VOL (GC)**

Holding Times  
Calibrations  
Blanks  
MS/MSD, MS, or BS  
Overall Assessment

**BNA**

Holding Times  
Calibrations  
Blanks  
MS/MSD, MS, or BS  
Overall Assessment

**PHC**

Holding Times  
Calibrations  
Blanks  
MS/MSD, MS, or BS  
Overall Assessment

**P\_P**

Holding Times  
Instrument Performance  
Calibrations  
Blanks  
MS/MSD, MS, or BS  
Overall Assessment

**H\_A**

Holding Times  
Calibrations  
Blanks  
MS/MSD, MS, or BS  
Overall Assessment

**MET, ANI**

Holding Times

Calibrations

Blanks

MS/MSD, MS, or BS

**LEVEL C OR III**

**VOL (GC/MS)**

Holding Times

Tuning

Calibrations

Blanks

Surrogate Recovery

MS/MSD, MS, or BS

Internal Standards

Overall Assessment

**VOL (GC)**

Holding Times

Calibrations

Blanks

Surrogate Recovery

MS/MSD, MS, or BS

Overall Assessment

**BNA**

**Holding Times**  
**Tuning**  
**Calibrations**  
**Blanks**  
**Surrogate Recovery**  
**MS/MSD, MS, or BS**  
**Internal Standards**  
**Overall Assessment**

**PHC**

**Holding Times**  
**Calibrations**  
**Blanks**  
**Overall Assessments**

**P\_P**

**Holding Times**  
**Instrument Performance**  
**Calibrations**  
**Blanks**  
**Overall Assessment**

**H\_A**

**Holding Times**  
**Calibrations**  
**Blanks**  
**MS/MSD, MS, or BS**  
**Overall Assessment**

MET, ANI

Holding Times

Calibrations

Blanks

MS/MSD, MS, or BS

LEVEL D OR IV

VOL & BNA

Holding Times

Tuning

Calibrations

Blanks

Surrogate Recovery

MS/MSD, MS, or BS

Field Duplicates

Internal Standards

Overall Assessment

P\_P

Holding Times

Instrument Performance

Calibrations

Blanks

Surrogate Recovery

MS/MSD, MS, or BS

Field Duplicates

Overall Assessment

PHC

Holding Times  
Calibration  
Blanks  
Surrogate Recovery  
MS/MSD, MS, or BS  
Field Duplicates  
Overall Assessment

H\_A

Holding Times  
Calibrations  
Blanks  
Surrogate Recovery  
MS/MSD, MS, BS  
Internal Standards  
Field Duplicates  
Overall Assessment

MET & ANI

Holding Times  
Calibrations  
Blanks  
ICP Interference  
Laboratory Control Samples  
Furnace Atomic Absorption QC  
ICP Serial Dilution  
Field Duplicates  
MS/MSD, MS, BS

**APPENDIX B**

**DATA QUALIFIER Q FLAGS**



APPENDIX B  
DATA QUALIFIER DEFINITIONS  
Q FLAGS

The following are the codes used to qualify data, both in the DSD and by DATAVAL and the validator.

- U The analyte was analyzed for but not detected above the level of the associated value. This value is either the sample quantitation limit or the sample detection limit.
- J The analyte was positively identified. The associated value is the approximate concentration.
- N The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification."
- NJ The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
- UJ The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
- R The data are unusable due to serious deficiencies in the ability to analyze the sample.
- B The analyte was detected in the associated blank.
- BR The data are unusable due to blank contamination.



**APPENDIX C**

**ELECTRONIC TRANSFER DATA BASES**



APPENDIX C  
ELECTRONIC TRANSFER DATA BASES

SAMPLE DATA BASES

Field Name	Description	Data Base of Origin	Size of Field <sup>a</sup>
1. SAMPNUM	Sample number	FIELD	8
2. SAMPTYPE	Sample type	FIELD	3
3. ANALTYPE	Analysis type	FIELD	3
4. SAMPDATE	Sample date	FIELD	8
5. EXTDATE	Extraction date	LAB	8
6. ANALDATE	Analysis date	LAB	8
7. ANALTIME	Analysis time	LAB	5
8. MATRIX	Sample matrix	FIELD	1
9. COMPOUND	Contamination	LAB	3 5
10. RT	Response time	LAB	6
11. TCL_TIC	Compound list	LAB	3
12. CON	Concentration	LAB	13 2
13. UNITS	Conc. units	LAB	5
14. QCODE	Lab Q code	LAB	3
15. MBNUM	Associated method blank	LAB	15

<sup>a</sup> The first number indicates whole number characters; the second number indicates places beyond the decimal.

## METHOD BLANK DATA BASES

Field Name	Description	Data Base of Origin	Size of Field <sup>a</sup>
1. MBNUM	Method blank number	LAB	15
2. ANALDATE	Analysis date	LAB	8
3. ANALTYPE	Type of analysis	LAB	3
4. COMPOUND	Contamination	LAB	3 5
5. RT	Response time	LAB	6
6. TCL_TIC	Compound list	LAB	3
7. CON	Concentration	LAB	13 2
8. UNITS	Conc. units	LAB	5
9. MATRIX	Sample matrix	LAB	1

<sup>a</sup> The first number indicates whole number characters; the second number indicates places beyond the decimal.

**APPENDIX D**

**PROGRAM FLAGGING LOGIC**



APPENDIX D  
PROGRAM FLAGGING LOGIC

VOL, BNA, PHC, H&A

- I. Holding times
  - A. Professional judgement is used for flags.
- II. Tuning
  - A. If mass calibration is in error, classify all associated data as Unusable (R).
  - B. Professional judgement is used if ion abundance is outside of limits.
- III. Calibrations
  - A. Initial calibration
    - 1. If compound average RRF  $< 0.05$ , then:
      - a. flag positive results as Estimated (J);
      - b. flag non-defects as Unusable (R).
    - 2. If compound has % RSD  $> 30\%$ , then:
      - a. flag positive results as Estimated (J);
      - b. non-detects may be qualified using professional judgement.
  - B. Continuing calibration
    - 1. If compound has average RRF  $< 0.05$ , then:
      - a. flag positive results as Estimated (J);
      - b. flag non-detects as Unusable (R).
    - 2. If compound has %D  $> 25\%$ , then:
      - a. flag positive results as Estimated (J);
      - b. non-detects may be qualified using professional judgement.

Note: These flags are for compounds from samples that are run by the particular instrument corresponding to the particular calibration.

IV. Blanks

A. Blank contamination

1. Flag all associated sample data according to the following guidelines:
  - a. compound is found in the blank but not in the sample, no action required;
  - b. compound is found in the blank and also in the sample:

Case 1. If sample result is greater than the CRQL but less than the amount for the 5x/10x rule, then the results are flagged as Non-detect (U). The lab would report this as a positive result (i.e., for 60, the flag would be 60U).

Case 2. If sample result is less than CRQL and is also less than the required amount by the 5X/10X rule, then the results are flagged as non-detects (i.e., 5J would be flagged 5U).

Case 3. If sample result is greater than the required amount (5x/10x), then the result is left as a positive detect.

**P\_P**

I. Blanks

- A. Use Case 1 from the VOL procedure.
- B. Use Case 3 from the VOL procedure.

II. Surrogate recovery

- A. If low recoveries are obtained, flag associated positive results and quantitation limits as Estimated (J).
- B. If high recoveries are obtained, professional judgement should be used.

**MET, ANI**

- I. Holding times
  - A. If criteria for holding times are not met, qualify all results > IDL as Estimated (J) and results < IDL as Estimated (UJ).
  - B. If holding times are exceeded, the validator must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. The expected bias would be low, and the validator may determine that results < IDL are Unusable (R).
  - C. Due to the limited information concerning holding times for soil samples, it is left to the discretion of the validator whether to apply water holding time criteria to soil samples. If the data are qualified when water holding time criteria are applied to soil samples, it must be clearly documented in the review.
- II. Calibrations
  - A. If the minimum number of standards were not used for initial calibration or if the instrument was not calibrated daily and each time that the instrument was set up, qualify the data as Unusable (R).
  - B. If the correlation coefficient is < 0.995, qualify results > IDL as Estimated (J) and results < IDL as Estimated (UJ).
  - C. If the mid-range CN<sup>-</sup> standard was not distilled, qualify all associated results as Estimated (J).

D. If the ICV or CCV %R falls outside the acceptance windows, use professional judgement to qualify all associated data. If possible, indicate the bias in the review. The following guidelines are recommended:

1. If the ICV or CCV %R falls outside the acceptance windows but within the ranges 75-89% or 111%-125% (CN<sup>-</sup>, 70-84% or 116-130%; Hg, 65-79% or 121-135%), qualify results > IDL as Estimated (J).
2. If the ICV or CCV %R is within the range of 111-125% (CN<sup>-</sup>, 116-130%; Hg, 121-135%), results < IDL are acceptable.
3. If the ICV or CCV %R is 75-89% (CN<sup>-</sup>, 70-84%; Hg, 65-79%), qualify results < IDL as Estimated (UJ).
4. If the ICV or CCV %R is < 75%, (CN<sup>-</sup>, < 70%; Hg, < 65%), qualify all positive results as Unusable (R).
5. If the ICV or CCV %R is > 125%, (CN<sup>-</sup>, > 130%; Hg, > 135%), qualify results > IDL as Unusable (R); results < IDL are acceptable.

**APPENDIX E**

**TEST PACKAGE REPORTS**



PROJECT: PROGRAM TEST  
 LABORATORY: ORNL  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92  
 DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

PACKAGE DEFICIENCIES SUMMARY

Total number of samples: 8

Analysis Type	Number of Analyses
VOL - Volatiles:	8
BNA - Semivolatiles:	7
PHC - Petroleum Hydrocarbons:	7
MET - Metals:	7
P_P - Pesticides:	7
ANI - Anions:	5
H_A - Halocarbons and Aromatics:	5

Sample Type	QC Samples as a % of Total Samples
Duplicate QC water samples:	16.67%
Duplicate QC soil samples:	50.00%
MS/MSD QC water samples:	16.67%
MS/MSD QC soil samples:	0.00%

Problem	Number of Problems
1. Holding Times exceeded:	
A. Extraction Holding Times exceeded:	1
B. Analysis Holding Times exceeded:	1
2. Tuning problems (VOL & BNA):	0
3. Initial Calibration:	0
4. Continuing Calibration:	8
5. Surrogate Recovery outside of limits (level IV or D):	0
6. Method Blank contamination:	3
7. Trip Blank or Field Blank contamination:	0
8. MS/MSD Recovery:	0
9. Matrix Spike:	0
10. Blank Spike:	1
11. Internal Standards:	0
12. PHC Calibration:	0
13. Field Duplicate:	3
14. PEST/PCB Continuing Calibrations:	0
15. PEST/PCB Instrument Performance:	0
16. Metals Curve Validation:	1
17. Metals Calibration:	1
18. Laboratory Control Samples:	0
19. ICP Interference:	0
20. ICP Serial Dilution:	0
21. PHC Sample Result Verification:	0

Ratio of detects with changed flags to total detects: 13 / 48

PROJECT: PROGRAM TEST  
LABORATORY: ORNL  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

---

PACKAGE DEFICIENCIES SUMMARY

Chain of Custody Missing: THIS IS A TEST SET FOR THIS SOFTWARE PACKAGE.

Illegible Information: THIS IS A TEST SET FOR THIS SOFTWARE PACKAGE.

Missing Information (Lab Report):

Missing QC Information:

Transcription Errors:

Logbook Problems:

Request for Analysis Problems:

PROJECT: PROGRAM TEST  
LABORATORY: ORNL  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

## PACKAGE DEFICIENCIES SUMMARY

## NONCONFORMANCE CALIBRATIONS FOR ORGANIC ANALYSES

ANALYSIS TYPE	CAL TYPE	DATE	TIME	NUMBER OF COMPOUNDS		
				%RSD>40%	%RSD>30%	%RSD>25%
BNA	CONT	09/24/92	0000	4		7
H_A	CONT	09/24/92	0000	1		
VOL	CONT	09/24/92	0030	2		7

REGION: \_\_\_\_\_

**ORGANIC REGIONAL DATA ASSESSMENT**

VALIDATION LEVEL: D

CASE NO.: \_\_\_\_\_

SITE: PROGRAM TEST  
NUMBER OF SAMPLES/MATRIX

LABORATORY: ORNL

WATER: 38 SOIL: 15  
REVIEWER (IF NOT ESD): \_\_\_\_\_  
REVIEWER'S NAME: DENNIS MARTY  
COMPLETION DATE: 09/24/92

SAMPLES START #: 1000  
END #: 1007

**DATA ASSESSMENT SUMMARY**

	VOA	BNA	PEST	H&A	ANIONS
1. HOLDING TIMES-----	O	O	O	O	O
2. GC/MS TUNE/INSTR. PERFORM-----	O	O	O	O	O
3. CALIBRATIONS-----	O	O	O	O	O
4. BLANKS-----	O	O	O	O	O
5. SURROGATES-----	O	O	O	O	O
6. MATRIX SPIKE/DUP.-----	O	O	O	O	O
7. OTHER QC-----	O	O	O	O	O
8. INTERNAL STANDARDS-----	O	O	O	O	O
9. COMPOUND IDENTIFICATION-----	O	O	O	O	O
10. SYSTEM PERFORMANCE-----	O	O	O	O	O
11. OVERALL ASSESSMENT-----	O	O	O	O	O

- O = DATA HAD NO PROBLEMS/OR QUALIFIED DUE TO MINOR PROBLEMS.
- M = DATA QUALIFIED DUE TO MAJOR PROBLEMS.
- Z = DATA UNACCEPTABLE.
- X = PROBLEMS, BUT DO NOT AFFECT DATA.

ACTION ITEMS: THIS IS A TEST SET OF DATA FOR THE PROGRAM.

AREAS OF CONCERN:

NOTABLE PERFORMANCE:

REGION: \_\_\_\_\_

INORGANIC REGIONAL DATA ASSESSMENT

VALIDATION LEVEL: D

CASE NO.: \_\_\_\_\_

SITE: PROGRAM TEST  
NUMBER OF SAMPLES/MATRIX

LABORATORY: ORNL

WATER: 38 SOIL: 15

REVIEWER (IF NOT ESD): \_\_\_\_\_

REVIEWER'S NAME: DENNIS MARTY

COMPLETION DATE: 09/24/92

SAMPLES START #: 1000

END #: 1007

DATA ASSESSMENT SUMMARY

	ICP	AA	HG	CYANIDE
1. HOLDING TIMES -----	0	0	0	
2. CALIBRATIONS -----	0	0	0	
3. BLANKS -----	0	0	0	
4. ICS -----	0			
5. LCS -----	0	0		
6. DUPLICATE ANALYSIS -----	0	0	0	
7. MATRIX SPIKE -----	0	0	0	
8. MSA -----	0	0		
9. SERIAL DILUTION -----	0			
10. SAMPLE VERIFICATION -----	0	0	0	
11. OTHER QC -----	0	0	0	
12. OVERALL ASSESSMENT -----	0	0	0	

- O = DATA HAD NO PROBLEMS/OR QUALIFIED DUE TO MINOR PROBLEMS.
- M = DATA QUALIFIED DUE TO MAJOR PROBLEMS.
- Z = DATA UNACCEPTABLE.
- X = PROBLEMS, BUT DO NOT AFFECT DATA.

ACTION ITEMS:

AREAS OF CONCERN:

NOTABLE PERFORMANCE:

PROJECT: PROGRAM TEST  
 Final Summary  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92  
 DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

SAMPLE NUMBER	SAMPLE TYPE	COMPOUND	RT	TCL/TIC	MATRIX	ANALYSIS TYPE	CONCENTRATION	UNITS	Q CODE	FINAL CODE
1000		1,1,1-TRICHLOROETHANE		TCL	S	VOL	100.00	µg/kg	J	J
1000		1,2-DICHLOROBENZENE		TCL	S	H_A	200.00	µg/kg		
1000		2-BUTANONE		TCL	S	VOL	50.00	µg/kg	J	J
1000		ARSENIC		TCL	S	MET	200.00	mg/kg		
1000		BENZENE		TCL	S	PHC	200.00	mg/kg		
1000		BIS(2-CHLOROETHOXY)METHANE		TCL	S	BNA	1750.00	µg/kg		
1000		CARBON DISULFIDE		TCL	S	VOL	2500.00	µg/kg		J
1000		CHROMIUM		TCL	S	MET	10.00	mg/kg	J	J
1000		ENDRIN KETONE		TCL	S	P_P	50.00	µg/kg		BR
1000		HBPC (TO JP-5 JET FUEL)		TCL	S	PHC	1500000.00	mg/kg		
1000	DL	1,1,1-TRICHLOROETHANE		TCL	S	VOL	125.00	µg/kg		J
1000	DL	1,2-DICHLOROBENZENE		TCL	S	H_A	225.00	µg/kg		
1000	DL	2-BUTANONE		TCL	S	VOL	75.00	µg/kg		
1000	DL	ARSENIC		TCL	S	MET	180.00	mg/kg		
1000	DL	BENZENE		TCL	S	PHC	750.00	mg/kg		
1000	DL	BENZENE		TCL	S	VOL	25.00	µg/kg		
1000	DL	BIS(2-CHLOROETHOXY)METHANE		TCL	S	BNA	1900.00	µg/kg		
1000	DL	CARBON DISULFIDE		TCL	S	VOL	3000.00	µg/kg		J
1000	DL	CHROMIUM		TCL	S	MET	12.00	mg/kg	J	J
1000	DL	ENDRIN KETONE		TCL	S	P_P	35.00	µg/kg		BR
1000	DL	HBPC (TO JP-5 JET FUEL)		TCL	S	PHC	2750000.00	mg/kg		
1001	SR	1,1,1-TRICHLOROETHANE		TCL	S	VOL	135.00	µg/kg		J
1001	SR	1,2-DICHLOROBENZENE		TCL	S	H_A	320.00	µg/kg		
1001	SR	2-BUTANONE		TCL	S	VOL	85.00	µg/kg		
1001	SR	ARSENIC		TCL	S	MET	325.00	mg/kg		
1001	SR	BENZENE		TCL	S	PHC	900.00	mg/kg		
1001	SR	BIS(2-CHLOROETHOXY)METHANE		TCL	S	BNA	2200.00	µg/kg		
1001	SR	CARBON DISULFIDE		TCL	S	VOL	2700.00	µg/kg		J
1001	SR	CHROMIUM		TCL	S	MET	16.00	mg/kg	J	J
1001	SR	ENDRIN KETONE		TCL	S	P_P	32.00	µg/kg		BR
1001	SR	HBPC (TO JP-5 JET FUEL)		TCL	S	PHC	1750000.00	mg/kg		
1002		2-CHLOROETHYL VINYL ETHER		TCL	W	H_A	150000.00	µg/L		
1002		4,4'-DDD		TCL	W	P_P	75.00	µg/L		
1002		4,4'-DDE		TCL	W	P_P	20.00	µg/L		
1002		4,4'-DDT		TCL	W	P_P	50.00	µg/L		R
1002		CHLORIDE		TCL	W	ANI	25.00	mg/L		
1002		ETHYL BENZENE		TCL	W	PHC	2000.00	µg/L		
1002		FLUORIDE		TCL	W	ANI	30.00	mg/L		J
1002		MERCURY		TCL	W	MET	1200.00	µg/L		J
1002		MP XYLENE		TCL	W	PHC	20.00	µg/L		
1002		N-NITROSO-DI-N-PROPYLAMINE		TCL	W	BNA	50.00	µg/L		J
1002		O XYLENE		TCL	W	PHC	100.00	µg/L		
1002		SILVER		TCL	W	MET	100.00	µg/L		
1002		VINYL ACETATE		TCL	W	VOL	1500.00	µg/L		
1002		ZINC		TCL	W	MET	500.00	µg/L		
1002	DL	N-NITROSO-DI-N-PROPYLAMINE		TCL	W	BNA	42.00	µg/L		J
1002	DL	VINYL ACETATE		TCL	W	VOL	1250.00	µg/L		
1003	WR	VINYL ACETATE		TCL	W	VOL	1700.00	µg/L		

PROJECT: PROGRAM TEST  
ANALYSIS: ANI - HOLDING TIMES  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

SAMPLE NUMBER	SAMPLE TYPE	MATRIX	SAMPLE DATE	EXTRACTION DATE	ANALYSIS DATE	EXTRACTION DAYS	EXTRACTION ACCEPTABLE	ANALYSIS DAYS	ANALYSIS ACCEPTABLE
1002		W	09/10/92		09/15/92			5	T
1003	WR	W	09/10/92		09/22/92			12	T
1004	MS	W	09/10/92		09/22/92			12	T
1005	ER	W	09/10/92		09/22/92			12	T
1006	FB	W	09/10/92		09/27/92			17	T

PROJECT: PROGRAM TEST  
ANALYSIS: ANI - CALIBRATION  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #: 1000

DATE: 09/24/92  
DATA VALIDATION LEVEL: D  
ENDING SAMPLE #: 1007

SDG	COMPOUND TYPE	COMPOUND	TRUE CONCENTRATION	FOUND CONCENTRATION	LAB RPD	CALCULATED RPD	COMP	LIMIT
1002	ICP	FLUORIDE	11.00	10.00	9.52	9.52	T	F

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: ANI - BLANKS  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92

DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

BLANK NUMBER	SAMPLE TYPE	COMPOUND	RT	TCL or TIC	CONCENTRATION	UNITS	RCODE
ANI100	MB	NITRATE, AS N		TCL	10.00	mg/L	

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: ANI - BLANKS  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

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BLANK NUMBER	SAMPLE TYPE	SDG	MATRIX
1006	FB	1002	W
AN11MB	MB	1002	W

Comments:

PROJECT: PROGRAM TEST  
 ANALYSIS: ANI - MS/MSD  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92

DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

SAMPLE NUMBER	SAMP TYPE	EDG	COMPOUND	SPIKE ADDED	SAMPLE RESULT	MATRIX SPIKE	MSD	MS %	MS VER %	MSD %	MSD VER %	CAL RPD	RPD VER
1004	MS	1002	NITRATE, AS N	2.20	0.80	2.50	1.20	77.27	T	18.18	F	70	F

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: ANI - MS  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

SAMPLE NUMBER	SAMP TYPE	SDG	COMPOUND	SSR	SR	SA	CAL SR	LIMIT
1004	MS	1002	PHOSPHATE	10.0000	1.0000	8.90	101.1	T

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: ANI - BLANK SPIKE  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

SAMPLE NUMBER	SAMP TYPE	SDG	COMPOUND	SPIKE ADDED	SAMPLE CONCENTRATION	BLANK SPIKE	LAB BS X R	CAL X R	LIMIT
1004	MS	1002	FLUORIDE	12.0000	2.5000	8.0000	85.0000	45.8	F

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: ANI - LABORATORY CONTROL SAMPLES  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #: 1000

DATE: 09/24/92  
DATA VALIDATION LEVEL: D  
ENDING SAMPLE #: 1007

SDG	LAB ID NUMBER	COMPOUND	WATER / SOIL	TRUE CONCENTRATION	FOUND CONCENTRATION	LAB PERCENT RECOVERY	CAL PERCENT RECOVERY	COMP	LIMIT
1002	AB4238	FLUORIDE	W	10.00	9.20	92.00	92.00	T	T

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: ANI - ICP INTERFERENCE  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92

DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

SDG	COMPOUND	SOLUTION	TRUE CON	INITIAL FOUND	CAL INITIAL PERCENT RECOVERY	INIT LIMIT	FINAL FOUND	CAL FINAL PERCENT RECOVERY	FIN LIMIT
1002	NITRATE, AS N	AB	15.00	12.80	85.3	T	13.90	92.7	T

Comments:



PROJECT: PROGRAM TEST  
ANALYSIS: ANI - ICP SERIAL DILUTION  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92

DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

SDG	COMPOUND	EPA SAMPLE NUMBER	INITIAL SAMPLE RESULT	SERIAL DILUTION RESULT	LAB PERCENT DIFFERENCE	CALCULATED PERCENT DIFFERENCE	LAB VS CAL COMPARISON	LIMITS
1002	SULFATE	2389A	25.00	27.00	7.2	8.00	F	T

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: ANI - CONTAMINATION REPORT  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

SAMPLE NUMBER	SAMPLE TYPE	SAMPLE DILUTION	SDG	COMPOUND	RT	TCL/TIC	CONCENTRATION	UNITS	Q	FLAG
1002		1.00	1002	CHLORIDE		TCL	25.00	mg/L		
1002		1.00	1002	FLUORIDE		TCL	30.00	mg/L		

PROJECT: PROGRAM TEST  
ANALYSIS: ANI - CONTAMINATION REPORT  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92

DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

TCL/ITC	COMPOUND	RT	MATRIX	NUMBER OF SAMPLES	HIGH CON	LOW CON	MEAN CON	IDL
TCL	CHLORIDE		W	1	25.00	25.00	25.00	0.50
TCL	FLUORIDE		W	1	30.00	30.00	30.00	0.50

PROJECT: PROGRAM TEST  
ANALYSIS: ANI - OVERALL ASSESSMENT  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

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Comments:

SDG: 1002 NO PROBLEMS.

PROJECT: PROGRAM TEST  
 ANALYSIS: BNA - HOLDING TIMES  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92  
 DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

SAMPLE NUMBER	SAMPLE TYPE	MATRIX	SAMPLE DATE	EXTRACTION DATE	ANALYSIS DATE	EXTRACTION DAYS	EXTRACTION ACCEPTABLE	ANALYSIS DAYS	ANALYSIS ACCEPTABLE
1000		S	09/10/92	09/14/92	09/15/92	4	T	1	T
1000	DL	S	09/10/92	09/15/92	09/16/92	5	T	1	T
1001	SR	S	09/10/92	09/11/92	09/15/92	1	T	4	T
1002		W	09/10/92	09/15/92	09/18/92	5	T	3	T
1002	DL	W	09/10/92	09/20/92	09/21/92	10	F	1	T
1003	WR	W	09/10/92	09/15/92	09/18/92	5	T	3	T
1004	MS	W	09/10/92	09/18/92	09/22/92	8	F	4	T
1005	ER	W	09/10/92	09/18/92	09/22/92	8	F	4	T
1006	FB	W	09/10/92	09/25/92	09/27/92	15	F	2	T

PROJECT: PROGRAM TEST  
ANALYSIS: BNA - TUNING  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

SDG	LAB ID NUMBER	COMPOUND	EXP	FORM	SPEC	443 R/Z RELATIVE ABUN	442 R/Z RELATIVE ABUN	CALC 2 ABUN	LAB 2 ABUN	CALC ERROR	LIMIT
1000	CV031&2	DFTPP		Y	Y	9.30	48.70	19.10	18.80	F	T
1002	DV0304	DFTPP		Y	Y	14.30	72.50	19.72	19.70	F	T

Comments:

PROJECT: PROGRAM TEST  
 ANALYSIS: BNA - INITIAL CALIBRATION  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92  
 DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

CAL DATE	COMPOUND	SDG	RRF1	RRF2	RRF3	RRF4	RRF5	RRFC	CMKC	RRFI	RRSD	CHK	RRSD
09/18/92	BENZYL ALCOHOL	1000	1.320	1.353	1.385	1.290	1.255	1.321	T	1.321	3.9	T	
09/18/92	DIBENZOFURAN	1000	1.924	1.885	1.898	1.749	1.608	1.813	T	1.813	7.3	T	
09/20/92	BENZO(K)FLUORANTHENE	1002	1.981	1.702	1.248	1.103	1.169	1.441	T	1.441	26.6	T	
09/20/92	BENZOIC ACID	1002	0.000	0.165	0.234	0.251	0.229	0.220	T	0.220	17.2	T	

Comments:

PROJECT: PROGRAM TEST  
 ANALYSIS: BNA - CONTINUING CALIBRATION  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92

DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

CAL DATE	TIME	COMPOUND	SDG	RRFI	RRFC	% D	LIMITS
09/24/92	0000	2-NITROANILINE	1000	0.604	0.430	28.8	F
09/24/92	0000	BENZO(G,H,I)PERYLENE	1000	1.417	0.460	66.1	F
09/24/92	0000	BENZOIC ACID	1000	0.269	0.119	55.8	F
09/24/92	0000	BENZOIC ACID	1002	0.220	0.147	33.2	F
09/24/92	0000	BIS(2-CHLOROISOPROPYL)ETHER	1002	3.349	2.478	26.0	F
09/24/92	0000	DIBENZ(A,H)ANTHRACENE	1000	1.250	0.700	44.0	F
09/24/92	0000	INDENO(1,2,3-CD)PYRENE	1000	1.631	0.744	54.4	F
09/24/92	0000	N-NITROSO-DI-N-PROPYLAMINE	1002	1.164	0.797	31.5	F

Comments:

PROJECT: PROGRAM TEST  
 ANALYSIS: BNA - SURROGATE RECOVERY  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92

DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

SDG	QUESTION 1	QUESTION 2	QUESTION 3	QUESTION 4	QUESTION 5	QUESTION 6
1000	T	T	F		F	F
1002	T	T	F		F	F

Question 1) Were recoveries on form [1] verified?  
 Question 2) Were all recoveries >= 10%?  
 Question 3) Was surrogate recovery a problem?  
 Question 4) If 3) is 1, is there evidence of purging, reinjection, or re-extraction?  
 Question 5) Were there two blanks with surrogates outside criteria?  
 Question 6) Were there two or more analyses for a fraction?

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: BNA - BLANKS  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92

DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

BLANK NUMBER	SAMPLE TYPE	COMPOUND	RT	TCL or TIC	CONCENTRATION	UNITS	GCODE
BNA2MB	MB	BIS(2-CHLOROETHOXY)METHANE		TCL	5.00	µg/kg	J
BNA1MB	MB	BIS(2-CHLOROETHOXY)METHANE		TCL	25.00	µg/L	J

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: BNA - BLANKS  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/52

DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

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BLANK NUMBER	SAMPLE TYPE	SDG	MATRIX
1006	FB	1002	W
BNA2MB	MB	1000	S
BNA1MB	MB	1002	W

Comments:

PROJECT: PROGRAM TEST  
 ANALYSIS: BNA - MS/MSD  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #: 1000

DATE: 09/24/92

DATA VALIDATION LEVEL: D  
 ENDING SAMPLE #: 1007

SAMPLE NUMBER	SAMP TYPE	SDG	COMPOUND	SPIKE ADDED	SAMPLE RESULT	MATRIX SPIKE	MSD	MS %	MS VER	MSD %	MSD VER	CAL RPD	RPD VER
1000		1000	PENTACHLOROPHENOL	100.00	25.00	85.00	90.00	60.00	T	65.00	T	-6	T
1000		1000	PYRENE	50.00	0.00	25.00	30.00	50.00	T	60.00	T	-18	T
1004	MS	1002	PENTACHLOROPHENOL	8490.00	0.00	4500.00	4940.00	53.00	T	58.19	T	-9	T
1004	MS	1002	PHENOL	200.00	0.00	51.40	58.70	25.70	T	29.35	T	-13	T

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: BNA - MS  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #: 1000

DATE: 09/24/92  
DATA VALIDATION LEVEL: D  
ENDING SAMPLE #: 1007

SAMPLE NUMBER	SAMP TYPE	SDG	COMPOUND	SSR	SR	SA	CAL SR	LIMIT
1000		1000	PYRENE	125.0000	10.0000	80.00	143.8	F
1004	MS	1002	2-CHLOROPHENDL	133.0000	0.0000	200.00	66.5	T

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: BNA - BLANK SPIKE  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

SAMPLE NUMBER	SAMP TYPE	SDG	COMPOUND	SPIKE ADDED	SAMPLE CONCENTRATION	BLANK SPIKE	LAB BS % R	CAL % R	LIMIT
1000		1000	1,2,4-TRICHLOROBENZENE	3330.0000	0.0000	2020.0000	61.0000	60.7	T
1000		1000	4-NITROPHENOL	6660.0000	0.0000	4130.0000	62.0000	62.0	T
1004	MS	1002	4-NITROPHENOL	200.0000	0.0000	65.0000	32.0000	32.5	T
1004	MS	1002	N-NITROSO-DI-N-PROPYLAMINE	100.0000	0.0000	60.6000	61.0000	60.6	T

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: BNA - FIELD DUPLICATES  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92

DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

SDG	SAMPNUM	SAMPTYPE	DUPNUM	DUPTYPE	COMPOUND	RT	SAMP CON	DUP CON	RPD
1000	1000		1001	SR	BIS(2-CHLOROETHOXY)METHANE		1750.00	2200.00	22.78

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: BNA - LAB DUPLICATES  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #: 1000

DATE: 09/24/92  
DATA VALIDATION LEVEL: D  
ENDING SAMPLE #: 1007

SDG	SAMPNUM	SAMPTYPE	DUPNUM	DUPTYPE	DILUTION	COMPOUND	RT	SAMP CON	DUP CON	RPD
1000	1000		1000	DL	10.00	BIS(2-CHLOROETHOXY)METHANE		1750.00	1900.00	8.22
1002	1002		1002	DL	50.00	N-NITROSO-DI-N-PROPYLAMINE		50.00	42.00	17.39

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: BNA - INTERNAL STANDARDS  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #: 1000

DATE: 09/24/92

DATA VALIDATION LEVEL: D  
ENDING SAMPLE #: 1007

SDG	FORM NUMBER	DATE	TIME	COMPOUND	SAMPLE NUMBER	SAMPLE TYPE	AREA COUNTS	RETENTION TIME
1000	CG0329	09/24/92	0000	ACENAPHTHENE-d10	1000		T	T
1002	CV03222	09/24/92	0000	ACENAPHTHENE-d10	1002		T	T

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: BNA - CONTAMINATION REPORT  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

SAMPLE NUMBER	SAMPLE TYPE	SAMPLE DILUTION	SDG	COMPOUND	RT	TCL/TIC	CONCENTRATION	UNITS	Q FLAG
1000		1.00	1000	BIS(2-CHLOROETHOXY)METHANE		TCL	1750.00	µg/kg	
1000	DL	1.00	1000	BIS(2-CHLOROETHOXY)METHANE		TCL	1900.00	µg/kg	
1001	SR	1.00	1000	BIS(2-CHLOROETHOXY)METHANE		TCL	2200.00	µg/kg	
1002		1.00	1002	N-NITROSO-DI-N-PROPYLAMINE		TCL	50.00	µg/L	
1002	DL	1.00	1002	N-NITROSO-DI-N-PROPYLAMINE		TCL	42.00	µg/L	

PROJECT: PROGRAM TEST  
ANALYSIS: BNA - CONTAMINATION REPORT  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #: 1000

DATE: 09/24/92  
DATA VALIDATION LEVEL: D  
ENDING SAMPLE #: 1007

TCL/ TIC	COMPOUND	RT	MATRIX	NUMBER OF SAMPLES	HIGH CON	LOW CON	MEAN CON	IDL
TCL	BIS(2-CHLOROETHOXY)METHANE		S	3	2200.00	1750.00	1950.00	350.00
TCL	N-NITROSO-DI-N-PROPYLAMINE		W	2	50.00	42.00	46.00	10.00

PROJECT: PROGRAM TEST  
ANALYSIS: BNA - SYSTEM REVIEW  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/82  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

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Comments:

SDG: 1000 NO PROBLEMS.

SDG: 1002 NO MAJOR PROBLEMS WITH THE SYSTEM.

PROJECT: PROGRAM TEST  
ANALYSIS: BNA - OVERALL ASSESSMENT  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92

DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

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Comments:

SDG: 1000 NO PROBLEMS.

SDG: 1002 NO PRBLEMS MAJOR PROBLEMS WITH THE DATA.

PROJECT: PROGRAM TEST  
ANALYSIS: H A - HOLDING TIMES  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #: 1000

DATE: 09/24/92  
DATA VALIDATION LEVEL: D  
ENDING SAMPLE #: 1007

SAMPLE NUMBER	SAMPLE TYPE	MATRIX	SAMPLE DATE	EXTRACTION DATE	ANALYSIS DATE	EXTRACTION DAYS	EXTRACTION ACCEPTABLE	ANALYSIS DAYS	ANALYSIS ACCEPTABLE
1002		W	09/10/92		09/15/92			5	F
1003	WR	W	09/10/92		09/22/92			12	F
1004	MS	W	09/10/92		09/22/92			12	F
1005	ER	W	09/10/92		09/22/92			12	F
1006	FB	W	09/10/92		09/27/92			17	F

PROJECT: PROGRAM TEST  
 ANALYSIS: H A - INITIAL CALIBRATION  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #: 1000

DATE: 09/24/92

DATA VALIDATION LEVEL: D  
 ENDING SAMPLE #: 1007

CAL DATE	COMPOUND	SDG	RRF1	RRF2	RRF3	RRF4	RRF5	RRFC	CHKC	RRF1	XRSD	CHK XRSD
09/18/92	BROMOMETHANE	1000	1.125	1.135	1.142	1.151	1.181	1.147	T	1.148	1.9	T
09/18/92	ETHYL BENZENE	1000	0.892	0.915	0.952	0.991	1.205	0.991	T	1.058	12.7	T
09/20/92	CARBON TETRACHLORIDE	1002	1.450	1.520	1.485	1.602	1.750	1.561	T	1.559	7.7	T

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: H A - CONTINUING CALIBRATION  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

CAL DATE	TIME	COMPOUND	SDG	RRFI	RRFC	% D	LIMITS
09/24/92	0000	TETRACHLOROETHENE	1000	1.123	1.215	-8.2	T
09/24/92	0000	TRANS-1,2-DICHLOROETHENE	1000	0.892	1.254	-40.6	F
09/24/92	1120	DICHLORODIFLUOROMETHANE	1002	1.250	0.895	28.4	F

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: H A - LAB DUPLICATES  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #: 1000

DATE: 09/24/92  
DATA VALIDATION LEVEL: D  
ENDING SAMPLE #: 1007

SDG	SAMPNUM	SAMPTYPE	DUPNUM	DUPTYPE	DILUTION	COMPOUND	RT	SAMP CON	DUP CON	RPD
1000	1000		1000	DL	10.00	1,2-DICHLOROBENZENE		200.00	225.00	11.76

Comments:

PROJECT: PROGRAM TEST  
 ANALYSIS: H A - SURROGATE RECOVERY  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92

DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

SDG	QUESTION 1	QUESTION 2	QUESTION 3	QUESTION 4	QUESTION 5	QUESTION 6
1000	T	T	F		F	F
1002	T	T	F		F	F

Question 1) Were recoveries on form [1] verified?  
 Question 2) Were all recoveries >= 10%?  
 Question 3) Was surrogate recovery a problem?  
 Question 4) If 3) is 1, is there evidence of purging, reinjection, or re-extraction?  
 Question 5) Were there two blanks with surrogates outside criteria?  
 Question 6) Were there two or more analyses for a fraction?

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: H A - BLANKS  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #: 1000

DATE: 09/24/92  
DATA VALIDATION LEVEL: D  
ENDING SAMPLE #: 1007

BLANK NUMBER	SAMPLE TYPE	COMPOUND	RT	TCL or TIC	CONCENTRATION	UNITS	QCODE
HA1NB	NB	CHLOROBENZENE		TCL	15.00	µg/L	
HA2NB	NB	METHYLENE CHLORIDE		TCL	10.00	µg/L	

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: H A - BLANKS  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

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BLANK NUMBER	SAMPLE TYPE	SDG	MATRIX
1006	FB	1002	W
HA1MB	MB	1002	W
HA2MB	MB	1002	W

Comments:

PROJECT: PROGRAM TEST  
 ANALYSIS: H A - MS/MSD  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #: 1000

DATE: 09/24/92

DATA VALIDATION LEVEL: D  
 ENDING SAMPLE #: 1007

SAMPLE NUMBER	SAMP TYPE	SDG	COMPOUND	SPIKE ADDED	SAMPLE RESULT	MATRIX SPIKE	MSD	MS %	MS VER	MSD %	MSD VER	CAL RPS	RPD VER
1000		1000	1,1-DICHLOROETHENE	100.00	0.00	56.00	62.00	56.00	F	62.00	F	-10	T
1000		1000	BENZENE	250.00	75.00	88.00	90.00	5.20	F	6.00	F	-2	T
1004	MS	1002	TRICHLOROETHENE	100.00	0.00	75.00	68.00	75.00	T	68.00	F	10	F

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: H A - MS  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #: 1000

DATE: 09/24/92  
DATA VALIDATION LEVEL: D  
ENDING SAMPLE #: 1007

SAMPLE NUMBER	SAMP TYPE	SDG	COMPOUND	SSR	SR	SA	CAL SR	LIMIT
1000		1000	BENZENE	120.0000	0.0000	200.00	60.0	F
1004	MS	1002	TOLUENE	500.0000	125.0000	350.00	107.1	T

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: H A - BLANK SPIKE  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #: 1000

DATE: 09/24/92  
DATA VALIDATION LEVEL: D  
ENDING SAMPLE #: 1007

SAMPLE NUMBER	SAMP TYPE	SDG	COMPOUND	SPIKE ADDED	SAMPLE CONCENTRATION	BLANK SPIKE	LAB BS	CAL	LIMIT
1000		1000	TOLUENE	200.0000	10.0000	140.0000	95.0000	65.0	F
1004	MS	1002	BENZENE	200.0000	20.0000	120.0000	110.0000	50.0	F

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: H A - FIELD DUPLICATES  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

SDG	SAMPNUM	SAMPTYPE	DUPNUM	DUPTYPE	COMPOUND	RT	SAMP CON	DUP CON	RPD
1000	1000		1001	SR	1,2-DICHLOROBENZENE		200.00	320.00	46.15

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: H A - INTERNAL STANDARDS  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #: 1000

DATE: 09/24/92  
DATA VALIDATION LEVEL: D  
ENDING SAMPLE #: 1007

SDG	FORM NUMBER	DATE	TIME	COMPOUND	SAMPLE NUMBER	SAMPLE TYPE	AREA COUNTS	RETENTION TIME
1000	CV03224	09/24/92	0000	DICHLORO(1)FLUOROMETHANE	1000		T	T
1002	CD091241	09/24/92	1100	BENZENE	1002		T	T

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: H A - CONTAMINATION REPORT  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

SAMPLE NUMBER	SAMPLE TYPE	SAMPLE DILUTION	SDG	COMPOUND	RT	TCL/ TTL	CONCENTRATION	UNITS	FLAG
1000		1.00	1000	1,2-DICHLOROBENZENE		TCL	200.00	µg/kg	
1000	DL	1.00	1000	1,2-DICHLOROBENZENE		TCL	225.00	µg/kg	
1001	SR	1.00	1000	1,2-DICHLOROBENZENE		TCL	320.00	µg/kg	
1002		1.00	1002	2-CHLOROETHYL VINYL ETHER		TCL	150000.00	µg/L	

PROJECT: PROGRAM TEST  
ANALYSIS: H A - CONTAMINATION REPORT  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #: 1000

DATE: 09/24/92  
DATA VALIDATION LEVEL: D  
ENDING SAMPLE #: 1007

TCL/ IDL	COMPOUND	RT	MATRIX	NUMBER OF SAMPLES	HIGH CON	LOW CON	MEAN CON	IDL
TCL	1,2-DICHLOROBENZENE		S	3	320.00	200.00	248.33	NA
TCL	2-CHLOROETHYL VINYL ETHER		W	1	150000.00	150000.00	150000.00	NA

PROJECT: PROGRAM TEST  
ANALYSIS: H A - SYSTEM REVIEW  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

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Comments:

SDG: 1000 NO MAJOR PROBLEMS.

SDG: 1002 NO MAJOR PROBLEMS.

PROJECT: PROGRAM TEST  
ANALYSIS: H A - OVERALL ASSESSMENT  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

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Comments:

SDG: 1000 NO MAJOR PROBLEMS.

SDG: 1002 NO PROBLEMS WITH THE DATA.

PROJECT: PROGRAM TEST  
ANALYSIS: MET - HOLDING TIMES  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

SAMPLE NUMBER	SAMPLE TYPE	MATRIX	SAMPLE DATE	EXTRACTION DATE	ANALYSIS DATE	EXTRACTION DAYS	EXTRACTION ACCEPTABLE	ANALYSIS DAYS	ANALYSIS ACCEPTABLE
1000		S	09/10/92		09/15/92			5	T
1000	DL	S	09/10/92		09/16/92			6	T
1001	SR	S	09/10/92		09/15/92			5	T
1002		W	09/10/92		09/15/92			5	T
1003	WR	W	09/10/92		09/22/92			12	T
1004	MS	W	09/10/92		09/22/92			12	T
1005	ER	W	09/10/92		09/22/92			12	T
1006	FB	W	09/10/92		09/27/92			17	T

PROJECT: PROGRAM TEST  
ANALYSIS: MET - CAL (Curve Validation)  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92

DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

SDG	CURVE TYPE	CORRELATION COEFFICIENT	LIMIT
1002	AA	0.99663	T
1002	ICP	0.99959	T
1002	MERCURY	0.98917	F

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: MET - CALIBRATION  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

SDG	COMPOUND TYPE	COMPOUND	TRUE CONCENTRATION	FOUND CONCENTRATION	LAB PERCENT RECOVERY	CAL PERCENT RECOVERY	COMP	LIMIT
1002	ICP	ARSENIC	4000.00	3778.50	94.50	94.46	T	T
1002	ICP	CALCIUM	40000.00	41733.22	104.30	104.33	T	T

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: MET - BLANKS  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

BLANK NUMBER	SAMPLE TYPE	COMPOUND	RT	TCL or TIC	CONCENTRATION	UNITS	QCODE
NET2MB	MB	ARSENIC		TCL	50.00	mg/kg	
NET1MB	MB	ARSENIC		TCL	15.00	µg/L	

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: MET - BLANKS  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #: 1000

DATE: 09/24/92  
DATA VALIDATION LEVEL: D  
ENDING SAMPLE #: 1007

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BLANK NUMBER	SAMPLE TYPE	SDG	MATRIX
1006	FB	1002	W
MET2MB	MB	1000	S
MET1MB	MB	1002	W

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: MET - BLANK SPIKE  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92

DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

SAMPLE NUMBER	SAMP TYPE	SDG	COMPOUND	SPIKE ADDED	SAMPLE CONCENTRATION	BLANK SPIKE	LAB MS % R	CAL % R	LIMIT
1004	MS	1002	BORON	100.0000	0.0000	92.0000	92.0000	92.0	T

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: MET - LABORATORY CONTROL SAMPLES  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

SDG	LAB ID NUMBER	COMPOUND	WATER /SOIL	TRUE CONCENTRATION	FOUND CONCENTRATION	LAB PERCENT RECOVERY	CAL PERCENT RECOVERY	COMP	LIMIT
1002	LCSW-E2039	ANTIMONY	W	4000.00	3834.70	95.90	95.87	T	T
1002	LCSW-E2039	CALCIUM	W	20000.00	21747.90	108.70	108.74	T	T

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: MET - ICP INTERFERENCE  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92

DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

SDG	COMPOUND	SOLUTION	TRUE CON	INITIAL FOUND	CAL INITIAL PERCENT RECOVERY	INIT LIMIT	FINAL FOUND	CAL FINAL PERCENT RECOVERY	FIN LIMIT
1002	ALUMINUM	A	500000.00	493941.00	98.8	T	494479.00	98.9	T
1002	MAGNESIUM	AB	491000.00	494219.60	100.7	T	484706.80	98.7	T

Comments:



PROJECT: PROGRAM TEST  
ANALYSIS: MET - ICP SERIAL DILUTION  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #: 1000

DATE: 09/24/92

DATA VALIDATION LEVEL: D  
ENDING SAMPLE #: 1007

SDG	COMPOUND	EPA SAMPLE NUMBER	INITIAL SAMPLE RESULT	SERIAL DILUTION RESULT	LAB PERCENT DIFFERENCE	CALCULATED PERCENT DIFFERENCE	LAB VS CAL COMPARISON	LIMITS
1002	LITHIUM HYDRIDE	1243DF	50.00	57.00	14.0	14.00	T	F

Comments:

PROJECT: PROGRAM TEST  
 ANALYSIS: MET - CONTAMINATION REPORT  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92  
 DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

SAMPLE NUMBER	SAMPLE TYPE	SAMPLE DILUTION	SDG	COMPOUND	RT	TCL/TIC	CONCENTRATION	UNITS	Q FLAG
1000		1.00	1000	ARSENIC		TCL	200.00	mg/kg	
1000		1.00	1000	CHROMIUM		TCL	10.00	mg/kg	J
1000	DL	1.00	1000	ARSENIC		TCL	180.00	mg/kg	
1000	DL	1.00	1000	CHROMIUM		TCL	12.00	mg/kg	J
1001	SR	1.00	1000	ARSENIC		TCL	325.00	mg/kg	
1001	SR	1.00	1000	CHROMIUM		TCL	16.00	mg/kg	J
1002		1.00	1002	MERCURY		TCL	1200.00	µg/L	
1002		1.00	1002	SILVER		TCL	100.00	µg/L	
1002		1.00	1002	ZINC		TCL	500.00	µg/L	

PROJECT: PROGRAM TEST  
ANALYSIS: MET - CONTAMINATION REPORT  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #: 1000

DATE: 09/24/92  
DATA VALIDATION LEVEL: D  
ENDING SAMPLE #: 1007

TCL/ TIC	COMPOUND	RT	MATRIX	NUMBER OF SAMPLES	HIGH CON	LOW CON	MEAN CON	TDL
TCL	ARSENIC		S	3	325.00	180.00	235.00	NA
TCL	CHROMIUM		S	3	16.00	10.00	12.67	NA
TCL	MERCURY		W	1	1200.00	1200.00	1200.00	NA
TCL	SILVER		W	1	100.00	100.00	100.00	5.00
TCL	ZINC		W	1	500.00	500.00	500.00	5.00

PROJECT: PROGRAM TEST  
ANALYSIS: MET - OVERALL ASSESSMENT  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

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Comments:

SDG: 1002 NO SYSTEM PROBLEMS FOUND.

PROJECT: PROGRAM TEST  
 ANALYSIS: PHC - HOLDING TIMES  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92

DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

SAMPLE NUMBER	SAMPLE TYPE	MATRIX	SAMPLE DATE	EXTRACTION DATE	ANALYSIS DATE	EXTRACTION DAYS	EXTRACTION ACCEPTABLE	ANALYSIS DAYS	ANALYSIS ACCEPTABLE
1000		S	09/10/92		09/15/92			5	T
1000	DL	S	09/10/92		09/16/92			6	T
1001	SR	S	09/10/92		09/15/92			5	T
1002		W	09/10/92		09/15/92			5	T
1003	WR	W	09/10/92		09/15/92			5	T
1004	MS	W	09/10/92		09/15/92			5	T
1005	ER	W	09/10/92		09/15/92			5	T
1006	FB	W	09/10/92		09/15/92			5	T

PROJECT: PROGRAM TEST  
ANALYSIS: PHC - INITIAL CALIBRATION  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #: 1000

DATE: 09/25/92

DATA VALIDATION LEVEL: C  
ENDING SAMPLE #: 1007

CAL DATE	COMPOUND	SDG	RRF1	RRF2	RRF3	RRF4	RRF5	RRFC	CHKC	RRF1	XRSD	CHK XRSD
09/15/92	BENZENE	1000	0.125	0.127	0.128	0.125	0.126	0.126	T	0.126	1.0	T
09/15/92	TOLUENE	1000	1.145	1.148	1.149	1.150	1.153	1.149	T	1.149	0.3	T

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: PHC - CONTINUING CALIBRATION  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/25/92  
DATA VALIDATION LEVEL:C  
ENDING SAMPLE #:1007

CAL DATE	TIME	COMPOUND	SDG	RRFI	RRFC	% D	LIMITS
09/20/92	1547	MP XYLENE	1000	1.587	0.957	39.7	F
09/20/92	1547	TOLUENE	1000	1.148	1.025	10.7	T

Comments:

PROJECT: PROGRAM TEST  
 ANALYSIS: PHC - CALIBRATION  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #: 1100

DATE: 09/28/92

DATA VALIDATION LEVEL: D  
 ENDING SAMPLE #: 1103

SDG	METHOD	COMPOUND	DATE	TIME	CORR CORFF.	LIMIT	NUM1	NUM2	XRSD
1100	HBHC	HBPC (TO JP-5 JET FUEL)	09/28/92	1000	0.99232	N	582.0872	-0.1819	76.4
1100	LBHC	BENZENE	09/28/92	1000	0.99986	Y	*****	64.3309	79.0

If XRSD <= 20%, then a linear data fit ( $y=mx+b$ ) is employed, with  $m = \text{NUM1}$  and  $b = \text{NUM2}$ .  
 If XRSD > 20%, then a quadratic fit ( $y=a+bx+cx^2$ ) is employed, with  $a = 0$ ,  $b = \text{NUM1}$  and  $c = \text{NUM2}$ .

Comments:

PROJECT: PROGRAM TEST  
 ANALYSIS: PHC - SURROGATE RECOVERY  
 REVIEWER: DENNIS NARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92  
 DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

SDG	QUESTION 1	QUESTION 2	QUESTION 3	QUESTION 4	QUESTION 5	QUESTION 6
1002	T	T	F		F	F

Question 1) Were recoveries on form III verified?  
 Question 2) Were all recoveries  $\geq 10\%$ ?  
 Question 3) Was surrogate recovery a problem?  
 Question 4) If 3) is 1, is there evidence of purging, reinjection, or re-extraction?  
 Question 5) Were there two blanks with surrogates outside criteria?  
 Question 6) Were there two or more analyses for a fraction?

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: PHC - BLANKS  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #: 1000

DATE: 09/24/92

DATA VALIDATION LEVEL: D  
ENDING SAMPLE #: 1007

BLANK NUMBER	SAMPLE TYPE	COMPOUND	RT	TCL or TIC	CONCENTRATION	UNITS	QCODE
PHC2ND	MB	BENZENE		TCL	10.00	mg/kg	
PHC1MB	MB	BENZENE		TCL	25.00	µg/L	

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: PHC - BLANKS  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92

DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

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BLANK NUMBER	SAMPLE TYPE	SDG	MATRIX
1006	FB	1002	W
PHC2MB	MB	1000	S
PHC1MB	MB	1002	W

Comments:

PROJECT: PROGRAM TEST  
 ANALYSIS: PHC - MS/MSD  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92  
 DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

SAMPLE NUMBER	SAMP TYPE	SDG	COMPOUND	SPIKE ADDED	SAMPLE RESULT	MATRIX SPIKE	MSD	MS %	MS VER	MSD %	MSD VER	CAL RPD	RPD VER
1004	MS	1002	JPS JET FUEL	35.00	0.00	41.00	35.00	117.14	F	100.00	F	16	F
1004	MS	1002	TOLUENE	1.80	0.00	1.20	1.70	66.67	F	94.44	F	-34	T

Comments:

PROJECT: PROGRAM TEST  
 ANALYSIS: PHC - CONTAMINATION REPORT  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/25/92

DATA VALIDATION LEVEL:C  
 ENDING SAMPLE #:1007

SAMPLE NUMBER	SAMPLE TYPE	SAMPLE DILUTION	SDG	COMPOUND	RT	TCL/TIC	CONCENTRATION	UNITS	Q FLAG
1000		1.00	1000	BENZENE		TCL	200.00	mg/kg	
1000		1.00	1000	MBPC (TO JP-5 JET FUEL)		TCL	1500000.00	mg/kg	
1000	DL	1.00	1000	BENZENE		TCL	750.00	mg/kg	
1000	DL	1.00	1000	MBPC (TO JP-5 JET FUEL)		TCL	2750000.00	mg/kg	
1001	SR	1.00	1000	BENZENE		TCL	900.00	mg/kg	
1001	SR	1.00	1000	MBPC (TO JP-5 JET FUEL)		TCL	1750000.00	mg/kg	
1002		1.00	1002	ETHYL BENZENE		TCL	2000.00	µg/L	
1002		1.00	1002	MP XYLENE		TCL	20.00	µg/L	
1002		1.00	1002	O XYLENE		TCL	100.00	µg/L	

PROJECT: PROGRAM TEST  
 ANALYSIS: PHC - CONTAMINATION REPORT  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/25/92

DATA VALIDATION LEVEL:C  
 ENDING SAMPLE #:1007

TCL/ TIC	COMPOUND	RT	MATRIX	NUMBER OF SAMPLES	HIGH CON	LOW CON	MEAN CON	IDL
TCL	BENZENE		S	3	900.00	200.00	616.67	0.05
TCL	ETHYL BENZENE		W	1	2000.00	2000.00	2000.00	NA
TCL	HBPC (TO JP-5 JET FUEL)		S	3	2750000.00	1500000.00	2000000.00	10.00
TCL	MP XYLENE		W	1	20.00	20.00	20.00	NA
TCL	O XYLENE		W	1	100.00	100.00	100.00	NA

PROJECT: PROGRAM TEST  
 ANALYSIS: PHC - CONTAMINATION REPORT (SRV)  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92  
 DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

## STANDARDS DATA

SDG	INST #	DATE	TIME	BENZENE	TOLUENE	01 1ST/ BENZENE	ETHYL BENZENE	MP XYLENE	O XYLENE	01 SUR/ BENZENE	SOLVENT PEAK	02 1ST/ BENZENE	02 SUR/ BENZENE
1002	1254	09/15/92	1430	1.14	2.45	2.98	3.41	3.79	7.24	9.82	0.91	2.75	8.56
1002	1254	09/15/92	1540	1.15	2.47	2.92	3.51	3.79	7.30	9.84	0.90	2.76	8.60
1002	1254	09/15/92	1640	1.10	2.42	3.01	3.39	3.77	7.19	9.86	0.88	2.76	8.51
1002	1254	09/15/92	1740	1.11	2.47	2.95	3.39	* 2.74	7.25	9.85	0.94	2.75	8.60
1002	1254	09/15/92	1840	1.15	2.48	2.99	3.41	3.80	7.20	9.80	0.95	2.78	8.62
1002	1254	09/15/92	1940	1.11	2.43	3.02	3.38	3.83	7.25	9.83	0.95	2.76	8.65
AVERAGE				1.13	2.45	2.98	3.42	3.62	7.24	9.83	0.92	2.76	8.59
SIGMA				0.02	0.02	0.04	0.05	0.43	0.04	0.02	0.03	0.01	0.05

\* \* RRT outside of average  $\pm 2$  sigma.

Comments:

PROJECT: PROGRAM TEST  
 ANALYSIS: PHC - CONTAMINATION REPORT (SRV)  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92  
 DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

## SAMPLE DATA

SDG	SAMPLE #	INST #	DATE	TIME	BENZENE	TOLUENE	D1 1ST/ CL BENZENE	ETHYL BENZENE	MP XYLENE	O XYLENE	D1 SUR/ CL BENZENE	SOLVENT PEAK	D2 1ST/ CL BENZENE	D2 SUR/ CL BENZENE
1002	1002	1254	09/15/92	1530	1.11					7.29			2.77	
1002	1003	1254	09/15/92	1730	* 1.24			3.38				0.89	2.77	8.52
1002	1004	1254	09/15/92	1630		* 2.31								8.62
1002	1005	1254	09/15/92	1830	1.17		* 3.04							
1002	1006	1254	09/15/92	1930	1.20	2.44	3.02	3.38	3.81	7.25	* 9.68	0.90	2.80	8.60

\* # sample compound RRT outside of  $\pm 0.06$  RRT calibration.  
 \* # sample does not have an interval of calibration.

Comments:

E-79

PROJECT: PROGRAM TEST  
ANALYSIS: PHC - SYSTEM REVIEW  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1900

DATE:09/24/92

DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

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Comments:

SDG: 1002 NO SYSTEM PROBLEMS FOUND.

PROJECT: PROGRAM TEST  
ANALYSIS: PHC - OVERALL ASSESSMENT  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

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Comments:  
SDG: 1002 NO PROBLEMS.

PROJECT: PROGRAM TEST  
 ANALYSIS: P P - HOLDING TIMES  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92

DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

SAMPLE NUMBER	SAMPLE TYPE	MATRIX	SAMPLE DATE	EXTRACTION DATE	ANALYSIS DATE	EXTRACTION DAYS	EXTRACTION ACCEPTABLE	ANALYSIS DAYS	ANALYSIS ACCEPTABLE
1000		S	09/10/92	09/12/92	09/15/92	2	T	3	T
1000	DL	S	09/10/92	09/15/92	09/16/92	5	T	1	T
1001	SR	S	09/10/92	09/11/92	09/15/92	1	T	4	T
1002		W	09/10/92	09/15/92	09/18/92	5	T	3	T
1003	WR	W	09/10/92	09/15/92	09/18/92	5	T	3	T
1004	MS	W	09/10/92	09/20/92	09/22/92	10	F	2	T
1005	ER	W	09/10/92	09/20/92	09/22/92	10	F	2	T
1006	FB	W	09/10/92	09/28/92	09/30/92	18	F	2	T



PROJECT: PROGRAM TEST  
ANALYSIS: P P - INITIAL CALIBRATION  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/25/92  
DATA VALIDATION LEVEL:C  
ENDING SAMPLE #:1007

SDG	COMPOUND	DATE	TIME	RRF1	RRF2	RRF3	RRF4	RRF5	AVERAGE RRF	% RELATIVE STANDARD DEVIATION	COMP
1000	4,4'-DDD	09/10/92	1125	1140000	1120000	1250000	1650000	1450000	1322000	17.0	F
1000	AROCLOR-1248	09/10/92	1125	1500	1400	1470	1600	1700	1534	7.7	T

PROJECT: PROGRAM TEST  
ANALYSIS: P P - CONTINUING CALIBRATION  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/25/92  
DATA VALIDATION LEVEL:C  
ENDING SAMPLE #:1007

SDG	COMPOUND	DATE	TIME	R1	R2	PERCENT DIFFERENCE	LIMIT	QUANTITATED/ CONFIRMED
1000	DIELDRIN	09/25/92	2250	11540	12870	-11.5	T	Q
1000	HEPTACHLOR	09/25/92	2250	8750000	7020000	19.8	T	C

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: P P - INITIAL CALIBRATION  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

SDG	MIXTURE	COMPOUND	DATE	TIME	LOW	MEDIUM	HIGH	AVERAGE	% RELATIVE STANDARD DEVIATION	COMP
1002	A	GAMMA-BHC (LINDANE)	09/18/92	2200	1000	1750	2250	1667	37.7	F

PROJECT: PROGRAM TEST  
ANALYSIS: P P - CONTINUING CALIBRATION  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #: 1000

DATE: 09/24/92  
DATA VALIDATION LEVEL: D  
ENDING SAMPLE #: 1007

SDG	MIXTURE	COMPOUND	DATE	TIME	R1	R2	PERCENT DIFFERENCE	LIMIT	QUANTITATED/CONFIRMED
1002	A	HEPTACHLOR	09/24/92	1000	1125	1178	-4.7	T	Q

Comments:



PROJECT: PROGRAM TEST  
ANALYSIS: P P - BURROGATE RECOVERY  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

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SDG	QUESTION 1	QUESTION 2
1002	T	

Question 1 } Recoveries on form 11 were verified?  
Question 2 } If recoveries are not verified, there is evidence of interference?

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: P P - BLANKS  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

BLANK NUMBER	SAMPLE TYPE	COMPOUND	RT	TCL or TIC	CONCENTRATION	UNITS	QC CODE
PP2MB	MB	ENDRIN KETONE		TCL	25.00	µg/kg	
PP1MB	MB	4,4'-DDD		TCL	5.00	µg/L	J

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: P P - BLANKS  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92

DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

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BLANK NUMBER	SAMPLE TYPE	SDG	MATRIX
1006	FB	1002	W
PP2MB	MB	1000	S
PP1MB	MB	1002	W

Comments:

PROJECT: PROGRAM TEST  
 ANALYSIS: P P - MS/MSD  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #: 1000

DATE: 09/24/92

DATA VALIDATION LEVEL: D  
 ENDING SAMPLE #: 1007

SAMPLE NUMBER	SAMP TYPE	SDG	COMPOUND	SPIKE ADDED	SAMPLE RESULT	MATRIX SPIKE	MSD	MS %	MS VER	MSD %	MSD VER	CAL RPO	RPO VER
1004	MS	1002	DIELDRIN	0.20	0.00	0.26	0.31	130.00	F	155.00	F	-18	T

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: P P - MS  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #: 1000

DATE: 09/24/92  
DATA VALIDATION LEVEL: D  
ENDING SAMPLE #: 1007

SAMPLE NUMBER	SAMP TYPE	SDG	COMPOUND	SSR	SR	SA	CAL SR	LIMIT
1004	MS	1002	DIELDRIN	0.5000	0.0000	0.20	250.0	F

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: P P - BLANK SPIKE  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

SAMPLE NUMBER	SAMP TYPE	SDG	COMPOUND	SPIKE ADDED	SAMPLE CONCENTRATION	BLANK SPIKE	LAB BS Z R	CAL Z R	LIMIT
1004	MS	1002	DIELDRIN	0.2000	0.0000	0.2600	130.0000	130.0	F

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: P P - CONTAMINATION REPORT  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

SAMPLE NUMBER	SAMPLE TYPE	SAMPLE DILUTION	SDG	COMPOUND	RT	TCL/TIC	CONCENTRATION	UNITS	Q FLAG
1000		1.00	1000	ENDRIN KETONE		TCL	50.00	µg/kg	
1000	DL	1.00	1000	ENDRIN KETONE		TCL	35.00	µg/kg	
1001	SR	1.00	1000	ENDRIN KETONE		TCL	22.00	µg/kg	
1002		1.00	1002	4,4'-DDD		TCL	75.00	µg/L	
1002		1.00	1002	4,4'-DDE		TCL	20.00	µg/L	
1002		1.00	1002	4,4'-DDT		TCL	50.00	µg/L	

PROJECT: PROGRAM TEST  
ANALYSIS: P P - CONTAMINATION REPORT  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #: 1000

DATE: 09/24/92  
DATA VALIDATION LEVEL: D  
ENDING SAMPLE #: 1007

TCL/ YTC	COMPOUND	RT	MATRIX	NUMBER OF SAMPLES	HIGH CON	LOW CON	MEAN CON	IDL
TCL	4,4'-DDD		W	1	75.00	75.00	75.00	0.10
TCL	4,4'-DDE		W	1	20.00	20.00	20.00	0.10
TCL	4,4'-DDT		W	1	50.00	50.00	50.00	0.10
TCL	ENDRIN KETONE		S	3	50.00	32.00	39.00	17.00

PROJECT: PROGRAM TEST  
ANALYSIS: P P - OVERALL ASSESSMENT  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

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Comments:

SDG: 1002 NO PROBLEMS.

PROJECT: PROGRAM TEST  
ANALYSIS: VOL - HOLDING TIMES  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92

DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

SAMPLE NUMBER	SAMPLE TYPE	MATRIX	SAMPLE DATE	EXTRACTION DATE	ANALYSIS DATE	EXTRACTION DAYS	EXTRACTION ACCEPTABLE	ANALYSIS DAYS	ANALYSIS ACCEPTABLE
1000		S	09/10/92		09/15/92			5	T
1000	DL	S	09/10/92		09/16/92			6	T
1001	SR	S	09/10/92		09/15/92			5	T
1002		W	09/10/92		09/15/92			5	T
1002	DL	W	09/10/92		09/17/92			7	T
1003	WR	W	09/10/92		09/22/92			12	T
1004	MS	W	09/10/92		09/22/92			12	T
1005	ER	W	09/10/92		09/22/92			12	T
1006	FB	W	09/10/92		09/27/92			17	F
1007	TB	W	09/10/92		09/21/92			11	T

PROJECT: PROGRAM TEST  
ANALYSIS: VOL - TUNING  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92

DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

SCG	LAB ID NUMBER	COMPOUND	EXP	FORM	SPEC	176 m/z RELATIVE ABUN	174 m/z RELATIVE ABUN	CALC % ABUN	LAB % ABUN	CALC ERROR	LIMIT
1000	12345	BFB		Y	Y	73.20	76.20	96.06	96.10	F	T
1002	EBFB0305	BFB		Y	Y	78.20	78.40	99.74	99.70	F	T
1002	EBFB0306	BFB	Y	Y	Y	78.40	78.80	99.49	99.40	F	T

Comments:

PROJECT: PROGRAM TEST  
 ANALYSIS: VOL - INITIAL CALIBRATION  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #: 1000

DATE: 09/24/92  
 DATA VALIDATION LEVEL: D  
 ENDING SAMPLE #: 1007

CAL DATE	COMPOUND	SDG	RRF1	RRF2	RRF3	RRF4	RRF5	RRFC	CHKC	RRF1	XRSD	CHK	XRSD
09/20/92	2-BUTANONE	1002	0.173	0.215	0.205	0.219	0.225	0.207	T	0.207	9.9	T	
09/20/92	CARBON DISULFIDE	1000	1.259	1.548	1.639	1.730	1.706	1.576	T	1.576	12.1	T	
09/20/92	ETHYLBENZENE	1000	0.415	0.464	0.453	0.465	0.445	0.448	T	0.448	4.6	T	
09/20/92	TRANS-1,3-DICHLOROPROPENE	1002	0.221	0.287	0.327	0.383	0.417	0.327	T	0.327	23.7	T	

Comments:

## E-100

PROJECT: PROGRAM TEST  
ANALYSIS: VOL - CONTINUING CALIBRATION  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

CAL DATE	TIME	COMPOUND	SDG	RRFI	RRFC	% D	LIMITS
09/24/92	0030	1,1,1-TRICHLORETHANE	1000	0.783	0.334	57.3	F
09/24/92	0030	BROMODICHLOROMETHANE	1000	0.526	0.365	30.6	F
09/24/92	0030	BROMOFORM	1000	0.539	0.272	49.5	F
09/24/92	0030	BROMOMETHANE	1000	0.795	1.022	-28.6	F
09/24/92	0030	CARBON DISULFIDE	1000	1.576	0.984	37.6	F
09/24/92	0030	CIS-1,3-DICHLOROPROPENE	1000	0.396	0.297	25.0	F
09/24/92	0030	VINYL ACETATE	1000	0.556	0.354	36.3	F
09/24/92	0045	TRANS-1,3-DICHLOROPROPENE	1002	0.327	0.233	28.7	F
09/24/92	0045	TRICHLORoETHENE	1002	0.444	0.333	25.0	T

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: VOL - SURROGATE RECOVERY  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92

DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

SDG	QUESTION 1	QUESTION 2	QUESTION 3	QUESTION 4	QUESTION 5	QUESTION 6
1000	T	T	F		F	F
1002	T	T	F		F	F

Question 1) Were recoveries on form 111 verified?  
Question 2) Were all recoveries  $\geq 10\%$ ?  
Question 3) Was surrogate recovery a problem?  
Question 4) If 3) is Y, is there evidence of purging, reinjection, or re-extraction?  
Question 5) Were there two blanks with surrogates outside criteria?  
Question 6) Were there two or more analyses for a fraction?

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: VOL - BLANKS  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

BLANK NUMBER	SAMPLE TYPE	COMPOUND	RT	TCL or TIC	CONCENTRATION	UNITS	GCODE
VOL2MB	MB	1,1,1-TRICHLOROETHANE		TCL	10.00	µg/kg	J
VOL1MB	MB	1,1,1-TRICHLOROETHANE		TCL	5.00	µg/L	J

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: VOL - BLANKS  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

BLANK NUMBER	SAMPLE TYPE	SDG	MATRIX
1006	FB	1002	W
1007	TB	1002	W
VOL2NB	NB	1000	S
VOL1NB	NB	1002	W

Comments:

PROJECT: PROGRAM TEST  
 ANALYSIS: VOL - MS/MSD  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #: 1000

DATE: 09/24/92

DATA VALIDATION LEVEL: D  
 ENDING SAMPLE #: 1007

SAMPLE NUMBER	SAMP TYPE	SDG	COMPOUND	SPIKE ADDED	SAMPLE RESULT	MATRIX SPIKE	MSD	MS %	MS VER	MSD %	MSD VER	CAL RPD	RPD VER
1000		1000	1,1-DICHLOROETHENE	64.10	0.00	64.70	65.90	100.94	T	102.81	T	-2	T
1000		1000	BENZENE	64.10	0.00	54.50	55.00	85.02	T	85.80	T	-1	T
1004	MS	1002	CHLOROBENZENE	64.10	0.00	60.40	60.30	94.23	T	94.07	T	0	T

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: VOL - MS  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

SAMPLE NUMBER	SAMP TYPE	SDG	COMPOUND	SSR	SR	SA	CAL AR	LIMIT
1000		1000	TOLUENE	58.5000	0.0000	64.10	91.3	T

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: VOL - BLANK SPIKE  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

SAMPLE NUMBER	SAMP TYPE	SDG	COMPOUND	SPIKE ADDED	SAMPLE CONCENTRATION	BLANK SPIKE	LAB BS % R	CAL % R	LIMIT
1000		1000	TRICHLOROETHENE	64.1000	0.0000	59.7000	93.0000	93.1	T

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: VOL - FIELD DUPLICATES  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

SDG	SAMPNUM	SAMPTYPE	DUPNUM	DUPTYPE	COMPOUND	RT	SAMP CON	DUP CON	RPD
1000	1000		1001	SR	1,1,1-TRICHLOROETHANE		100.00	135.00	29.79
1000	1000		1001	SR	2-BUTANONE		50.00	85.00	51.85
1000	1000		1001	SR	CARBON DISULFIDE		2500.00	2700.00	7.69
1002	1002		1003	WR	VINYL ACETATE		1500.00	1700.00	12.50

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: VOL - LAB DUPLICATES  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

SDG	SAMPNUM	SAMPTYPE	DUPNUM	DUPTYPE	DILUTION	COMPOUND	RT	SAMP CON	DUP CON	RPD
1000	1000		1000	DL	10.00	1,1,1-TRICHLOROETHANE		100.00	125.00	22.22
1000	1000		1000	DL	10.00	2-BUTANONE		50.00	75.00	40.00
1000	1000		1000	DL	10.00	CARBON DISULFIDE		2500.00	3000.00	18.18
1002	1002		1002	DL	100.00	VINYL ACETATE		1500.00	1250.00	18.18

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: VOL - INTERNAL STANDARDS  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #: 1000

DATE: 09/24/92  
DATA VALIDATION LEVEL: D  
ENDING SAMPLE #: 1007

SIG	FORM NUMBER	DATE	TIME	COMPOUND	SAMPLE NUMBER	SAMPLE TYPE	AREA COUNTS	RETENTION TIME
1000	ES03122	09/24/92	0030	BROMOCHLOROMETHANE	1000		T	T
1000	ES03122	09/24/92	0030	BROMOCHLOROMETHANE	1000	DL	T	T
1002	ES03122	09/24/92	0000	1,4-DIFLUOROBENZENE	1002		T	F
1002	ES03122	09/24/92	0000	1,4-DIFLUOROBENZENE	1005	ER	F	T

Comments:

PROJECT: PROGRAM TEST  
 ANALYSIS: VOL - CONTAMINATION REPORT  
 REVIEWER: DENNIS MARTY  
 BEGINNING SAMPLE #:1000

DATE:09/24/92

DATA VALIDATION LEVEL:D  
 ENDING SAMPLE #:1007

SAMPLE NUMBER	SAMPLE TYPE	SAMPLE DILUTION	SDG	COMPOUND	RT	TCL/TIC	CONCENTRATION	UNITS	Q FLAG
1000		1.00	1000	1,1,1-TRICHLOROETHANE		TCL	100.00	µg/kg	J
1000		1.00	1000	2-BUTANONE		TCL	50.00	µg/kg	J
1000		1.00	1000	CARBON DISULFIDE		TCL	2500.00	µg/kg	
1000	DL	1.00	1000	1,1,1-TRICHLOROETHANE		TCL	125.00	µg/kg	
1000	DL	1.00	1000	2-BUTANONE		TCL	75.00	µg/kg	
1000	DL	1.00	1000	BENZENE		TCL	25.00	µg/kg	
1000	DL	1.00	1000	CARBON DISULFIDE		TCL	3000.00	µg/kg	
1001	SR	1.00	1000	1,1,1-TRICHLOROETHANE		TCL	135.00	µg/kg	
1001	SR	1.00	1000	2-BUTANONE		TCL	85.00	µg/kg	
1001	SR	1.00	1000	CARBON DISULFIDE		TCL	2700.00	µg/kg	
1002		1.00	1002	VINYL ACETATE		TCL	1500.00	µg/L	
1002	DL	1.00	1002	VINYL ACETATE		TCL	1250.00	µg/L	
1003	WR	1.00	1002	VINYL ACETATE		TCL	1700.00	µg/L	

Comments:

PROJECT: PROGRAM TEST  
ANALYSIS: VOL - CONTAMINATION REPORT  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

TCL/ TIC	COMPOUND	RT	MATRIX	NUMBER OF SAMPLES	HIGH CON	LOW CON	MEAN CON	IDL
TCL	1,1,1-TRICHLOROETHANE		S	3	135.00	100.00	120.00	6.00
TCL	2-BUTANONE		S	3	85.00	50.00	70.00	13.00
TCL	BENZENE		S	1	25.00	25.00	25.00	6.00
TCL	CARBON DISULFIDE		S	3	3000.00	2500.00	2733.33	6.00
TCL	VINYL ACETATE		W	3	1700.00	1250.00	1483.33	10.00

PROJECT: PROGRAM TEST  
ANALYSIS: VOL - SYSTEM REVIEW  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

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Comments:

SDG: 1000 PROBLEMS EXIST WITH THE CONTINUING CALIBRATIONS.

SDG: 1002 NO MAJOR PROBLEMS WITH THE SYSTEM.

PROJECT: PROGRAM TEST  
ANALYSIS: VOL - OVERALL ASSESSMENT  
REVIEWER: DENNIS MARTY  
BEGINNING SAMPLE #:1000

DATE:09/24/92  
DATA VALIDATION LEVEL:D  
ENDING SAMPLE #:1007

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Comments:

SDG: 1000 THE MAJORITY OF THE DATA APPEARS TO BE GOOD.

SDG: 1002 MOST DATA APPEARS TO BE GOOD.



**APPENDIX F**

**EQUATIONS USED FOR VALIDATING DATA**



## APPENDIX F

## EQUATIONS USED FOR VALIDATING DATA

TUNING

$$\% \text{ Ion Abundance} = \frac{\% \text{ relative abundance of } m/z \ 176}{\% \text{ relative abundance of } m/z \ 174} \times 100 \quad (1)$$

$$\% \text{ Ion Abundance} = \frac{\% \text{ relative abundance of } m/z \ 443}{\% \text{ relative abundance of } m/z \ 442} \times 100 \quad (2)$$

PERCENT BREAKDOWN OF ENDRIN

$$\% \text{ Breakdown} = \frac{\text{Degradation peak area(aldehyde + ketone)}}{\text{Peak area(endrin + aldehyde + ketone)}} \times 100 \quad (3)$$

PERCENT BREAKDOWN OF DDT

$$\% \text{ Breakdown} = \frac{\text{Total DDT Degradation peak area(DDE + DDD)}}{\text{Total DDT peak area(DDT + DDE + DDD)}} \times 100 \quad (4)$$

PERCENT DIFFERENCE IN RESPONSE TIMES

$$\%D = \frac{RT_i - RT_s}{RT_i} \times 100 \quad (5)$$

where

$RT_i$  = retention time of DBC in the initial standard

$RT_s$  = retention time of DBC in a subsequent analysis

MEAN OF X VALUES

$$\bar{X} = \sum_{i=1}^n \frac{X_i}{n} \quad (6)$$

STANDARD DEVIATION OF X VALUES

$$\sigma = \sqrt{\sum_{i=1}^n \frac{(X_i - \bar{X})^2}{n-1}} \quad (7)$$

PERCENT RELATIVE STANDARD DEVIATION

$$\%RSD = \frac{\sigma}{\bar{X}} \times 100 \quad (8)$$

CONTINUING CALIBRATIONSPercent Difference

$$\%D = \frac{\overline{RRF}_I - RRF_F}{\overline{RRF}_I} \times 100 \quad (9)$$

where

$\overline{RRF}_I$  = average relative response factor from initial calibration

$RRF_F$  = relative response factor from continuing calibration standard

PERCENT DIFFERENCES BETWEEN RECOVERIES, P\_P

$$\%D = \frac{R_1 - R_2}{R_1} \times 100 \quad (10)$$

where

$R_1$  = calibration factor from first analysis

$R_2$  = calibration factor from subsequent analysis

MEAN OF Y VALUES

$$\bar{y} = \frac{\sum_{i=1}^n y_i}{n} \quad (11)$$

SLOPE OF LINE

$$m = \frac{\sum_{i=1}^n (x \times y) - \frac{\sum_{i=1}^n (x) \times \sum_{i=1}^n (y)}{n}}{n} \quad (12)$$

Y-INTERCEPT OF THE LINE

$$b = \bar{y} - m \times \bar{x} \quad (13)$$

CORRELATION COEFFICIENT

$$r = \frac{\sum_{i=1}^n (xy) - \frac{(\sum_{i=1}^n x) \times (\sum_{i=1}^n y)}{n}}{\sqrt{(\sum_{i=1}^n x^2 - \frac{(\sum_{i=1}^n x)^2}{n}) \times (\sum_{i=1}^n y^2 - \frac{(\sum_{i=1}^n y)^2}{n})}} \quad (14)$$

PERCENT RECOVERY

$$\%R = \frac{\text{Found}}{\text{True}} \times 100 \quad (15)$$

where

*Found* = concentration of analyte measured in the solution

*True* = concentration of analyte in the solution

RELATIVE PERCENT DIFFERENCE

$$RPD = \frac{|(\text{True}) - (\text{Found})|}{\frac{(\text{True}) + (\text{Found})}{2}} \quad (16)$$

where

*True* = concentration of analyte in the solution

*Found* = concentration of analyte measured in the solution

**BLANK LIMITS****Target Compound List Limits**

$$\text{Limit} = 5 \times (\text{concentration level in blank}) \quad (17)$$

**Common Lab Contaminants Limits**

$$\text{Limit} = 10 \times (\text{concentration level in blank}) \quad (18)$$

**MS RECOVERY**

$$\%R = \frac{MS - SR}{SA} \times 100 \quad (19)$$

where

- MS* = matrix spike
- SR* = sample result
- SA* = spike added

**MSD RECOVERY**

$$\%R = \frac{MSD - SR}{SA} \times 100 \quad (20)$$

where

- MSD* = matrix spike duplicate
- SR* = sample result
- SA* = spike added

**RELATIVE PERCENT DIFFERENCE IN MS/MSD RECOVERIES**

$$RPD = \frac{(MS - MSD)}{\left(\frac{MS + MSD}{2}\right)} \times 100 \quad (21)$$

**BLANK SPIKE RECOVERY**

$$\%R = \frac{BS - SC}{SA} \times 100 \quad (22)$$

where

*BS* = blank spike concentration found

*SC* = sample concentration

*SA* = spike added

**MS RECOVERY**

$$\%R = \frac{(SSR - SR)}{SA} \times 100 \quad (23)$$

where

*SSR* = spiked sample result

*SR* = sample result

*SA* = spike added

**FIELD AND LAB DUPLICATES RELATIVE PERCENT DIFFERENCE**

$$RPD = \frac{|S - D|}{(S + D)/2} \times 100 \quad (24)$$

where

*S* = sample concentration

*D* = duplicate concentration

**INTERFERENCE CHECK SAMPLE****Percent Recovery**

$$\%R = \frac{\text{Found(AB)}}{\text{True(AB)}} \times 100 \quad (25)$$

where

*Found* = concentration of analyte measured in solution AB

*True* = concentration of analyte in solution AB

**ICP SERIAL DILUTION PERCENT DIFFERENCE**

$$\%D = \frac{|I-S|}{I} \times 100 \quad (26)$$

where

*I* = initial sample result

*S* = serial dilution sample result

**CONFIDENCE INTERVAL TEST**

$$I = (\bar{X} \pm 2\sigma) \quad (27)$$

where

*I* = 95% confidence interval

$\bar{X}$  = average relative response time

**PHC CALIBRATION**

$$\begin{aligned} \sum y &= b\sum x + c\sum x^2 \\ \sum xy &= b\sum x^2 + c\sum x^3 \\ \sum x^2y &= b\sum x^3 + c\sum x^4 \end{aligned} \quad (28)$$

where

*b* and *c* = constants determined by simultaneously solving the equations above

Sources: U.S.EPA 1988a, 1988b; Ott 1988; Spiegel 1961



**APPENDIX G**

**NONCONFORMANCE REPORT AND PROCEDURE**



**PROCEDURES FOR CORRECTION OF NONCONFORMANCES**

Items that generate nonconformance flags during the data validation process fall into three categories:

1. Nonconformance items that can be corrected. Correction requires the completion of a nonconformance report form that will be sent to the laboratory, along with a request for appropriate corrective action and response.
2. Nonconformance items for which corrections have been received and the flag is the result of the correction.
3. Nonconformance items for which no correction is possible (e.g., holding time exceeded, incorrect instrument calibration, etc.).

Nonconformances shall be handled through current quality assurance policies and guidelines. Specifically, a nonconformance report describing the problem and requesting actions and resolutions will be completed for each nonconformance. This report will be reviewed and approved by the QA/QC officer. A nonconformance response form will be attached to the nonconformance report. This response form shall be completed by the laboratory responsible for creating the nonconformance error and returned to the initiator within 30 days of receipt of the nonconformance report. The root cause of the problem, extent of condition, corrective actions, and actions to prevent recurrence are all required to be listed on the response form. Once the form has been returned to the initiator, the QA/QC officer shall review all data on the nonconformance report and determine whether or not the response is acceptable. The QA/QC officer shall check the appropriate box on the nonconformance form regarding acceptance or non-acceptance of the response and sign and date the form. Non-acceptance of the resolution shall require further investigation, root-cause analysis, and response from the person or persons responsible for the nonconformance until such corrective action and root-cause analysis is deemed acceptable by the QA/QC officer.

**NONCONFORMANCE RESPONSE AND EVALUATION**

- 1) Initiate and prepare nonconformance report.
- 2) Submit nonconformance report to QA/QC officer for review and concurrence.
- 3) Submit nonconformance report and nonconformance response form to laboratory.
- 4) Obtain response from responsible laboratory within thirty (30) calendar days after receipt of nonconformance report.
- 5) If a response is not received within thirty (30) calendar days of the nonconformance report transmittal date, call and document phone conversations to responsible laboratory. If the telephone effort is unsuccessful and the response is not received within the newly agreed-upon completion date, prepare a follow-up letter and send to the responsible organization management. Send copies to personnel on distribution list. If a response to the letter is not received within the allotted time and if all actions are unsuccessful, consideration will be given to initiate stop work action.
- 6) Once the response is received, the QA/QC officer evaluates the response and corrective actions for acceptability. The response evaluations may be dispositioned as: accepted, accepted with modifications, or rejected.
- 7) When the response to a nonconformance report is accepted but requires verification prior to closeout, the nonconformance report remains open subject to verification.
- 8) If the response to the nonconformance report includes satisfactory documentation to provide verification of corrective action implementation, the nonconformance is deemed acceptable by the QA/QC officer and the report closed within fifteen (15) calendar days after receipt.
- 9) If the response to a nonconformance report is not accepted the QA/QC officer notifies the initiator of the nonconformance and provides such reasons for rejection within fifteen (15) calendar days.

- 10) The initiator of the nonconformance report prepares a response letter to the responsible party within fifteen (15) calendar days of receipt of the decision of the QA/QC officer, noting the results of the evaluation and discussing any additional information that may be required.
- 11) The nonconformance report is closed out upon completion and acceptance of the corrective actions by letter. A copy of the closure action shall be provided to all responsible parties. Copies of all nonconformance reports and dispositions shall be documented, filed, and maintained.



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