INNOVATIVE APPROACHES TO DATA VALIDATION

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION III

JUNE 1995

INNOVATIVE APPROACHES FOR
VALIDATION OF ORGANIC AND INORGANIC DATA-
STANDARD OPERATING PROCEDURES
JUNE 1995

U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION III
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NOTICE

The procedures set forth herein are intended as guidance. They do not constitute rulemaking by the Agency and may not be relied on to create a substantive or procedural right enforceable by any other person. The Government may take action that is at variance with the procedures in this manual.

INTRODUCTION

There are five levels of data validation within this manual approach. The levels consist of three (3) organic review procedures (M-1, M-2, M-3) and two inorganic review procedures (IM-1, IM-2). (The term "level" is perhaps misleading because it implies succession, which is not the case with this or any other level presented in this document. The "levels" are independent of each other.) M-3 and IM-2 review procedures represent full validation as described in the EPA National Functional Guidelines for Data Review. Should the intended data use dictate review by the protocols presented in the National Functional Guidelines for Data Review (IM-2 and M-3) refer to the most recent version of Region III Modifications to the National Functional Guidelines. The remaining data review levels are described in detail in this document. The SOPs describing how to apply these levels to a data-package are in Appendices A through D. All procedures require full CLP or CLP equivalent data package deliverables.

In general, organic Level M-1's emphasis is on reviewing positive (detected) data. The primary question asked is whether or not a compound is present. If it is, the next question addressed is whether the compound is potentially from field or laboratory-induced contamination. If the answer is negative, then the presence of the compound is considered confirmed, and the reported concentrations are considered usable for some predefined data uses. If information regarding data quality and usability is required, then the emphasis is shifted accordingly to an evaluation of data quality parameters, false negatives, and detection limits (Level M-2 or IM-1). If legally defensible data are necessary, then a full, CLP-equivalent data validation is performed (Level M-3 or IM-2). There is a definite focus at every step of the process. This allows for a clear differentiation in the levels of data validation.

For guidance purposes, general data use categories and suggested levels of review are provided in Table 1-3. However, it is important to note that the selected level of review will be specific to the intended data use and specific project objectives.

Standard Operating Procedures

SOPs have been developed for Levels M-1, M-2, and IM-1 and are in the following sections of this document:

- Volatiles (Appendix A)
- Semivolatiles (Appendix B)
- Pesticides/PCB (Appendix C)
- Metals and Cyanide (Appendix D)

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B.   Validation of Semivolatile Data (Levels M1 and M2)

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C-1. Manual Level M1 (for Pesticides/PCBs)

C-2. Manual Level M2 (for Pesticides/PCBs)

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Attachment to Appendix C-2

D.   Validation of Inorganic Analyte Data (Level M1)

Glossary of Acronyms and Terms

Acronyms

%D	percent difference
%R	percent recovery of spiked amounts of analytes
AA	atomic absorption
AOB	analytical operations branch
APO	administrative project officer
BPB	Bromofluorobenzene (volatile instrument performance check)
BNA	base/neutral/acid
CADRE	computer aided data review and evaluation
CARD	CLP analytical results database
CCB	continuing calibration blank
CCS	contract compliance screening
CCV	continuing calibration verification
CF	calibration factor
CLP	contract laboratory program
CLP RAS	contract laboratory program routine analytical services
CRDL	contract required detection limit
CRQL	computer required quantitation limit
CSF	complete SDG file
CV	Coefficient of Variation
DAS	delivery of analytical services
DFTPP	Decafluorotriphenylphosphine (semivolatile instrument performance check)
DPO	deputy project officer
DQOs	data quality objectives
DV	data validation
ECD	electron-capture detector
EICP	extracted ion current profile
EMSL-LV	Environmental Monitoring Support Laboratory--Las Vegas
EPA	United States Environmental Protection Agency
GC	gas chromatography
GC/EC	gas chromatography/electron capture
GC/MS	gas chromatography/mass spectra
GPC	gel Permeation Chromatography
HAZRAP	Hazardous Waste Remedial Actions Program
ICB	initial calibration blank
ICP	inductively coupled plasma
ICS	interface check sample
ICV	initial calibration verification
IDL	instrument detection limits
IRDA	Inorganic Regional Data Assessment
IS	internal standards
LCS	library control sample

MCL	maximum contamination limit
ml	milliliter
MS/MSD	matrix spike/matrix spike duplicates
MSA	Method of Standard Addition.
m/z	the ratio of mass (m) to charge (z) of ions measured by GC/MS
NEESA	Naval Energy and Environmental Support Activity
NIST	National Institute of Standards and Technology
OADS	organic analysis data sheet (Form I)
ORDA	Organic Regional Data Assessment
PB	preparation blank
PC	personal computer
PCB	polychlorinated biphenyls
PEST/PCB	pesticide/polychlorinated biphenyls
PRP	potentially responsible party
QA/QC	quality assurance/quality control
QAPjP	quality assurance project plan
QC	quality control
QL	qualitative
QN	quantitative
RAS	routine analytical services
RIC	reconstructed ion chromatogram
RI/FS	remedial investigation/feasibility study
RPD	relative percent difference
RPM	remedial project manager
RRF	relative response factor
RRT	relative retention time
RSD	relative standard deviation
RT	retention time
SAS	special analytical services
SDG	sample delivery group
SMC	system monitoring compound
SMO	sample management office
SOP	standard operating procedure
SOW	statement of work
SVOA	semivolatile organic analyte
TAL	target analyte list
TCL	target compound list
TIC	tentatively identified compound
TPH	total petroleum hydrocarbons
TPO	technical project officer
VOA	volatile organic analytes

TERMS

Associated Samples

Any sample related to a particular QC analysis.
For example:
- For ICV, all samples run under the same calibration curve.
- For duplicate RPD, all SDG samples digested/ distilled of the same matrix.

Calibration Curve

A plot of absorbance versus concentration of standards.

Case

A finite, usually predetermined number of samples collected over a given time period for a particular site. A Case consists of one or more Sample Delivery Group(s).

Appendix A
Validation of Volatile Organic Analyte Data

- Subappendix A-1 covers Level M1

- Subappendix A-2 covers Level M2

Appendix A-1
Validation of Volatile Organic Analyte Data
Manual Level M1

1. Purpose and Applicability

This procedure provides step-by-step instructions to manually validate the volatile organic analyte (VOA) data using the manual innovative data validation approach at Level M1.

The procedure is applicable to the VOA data obtained using the Contract Laboratory Program Statement of Work (CLP SOW). Hard copy data conforming to the CLP SOW specifications are essential in order to carry out the procedure.

Data validated using this procedure are considered usable for the following types of purposes; however, the data users must decide on a case-by-case basis whether the procedure is suitable for their intended data uses. The suggested data uses are:

- Oversight of activities led by other parties

- Comparison to action levels

- Initial site investigation

- Contamination sources

2. Quality Control Measures Checked

Table M1-VOA-1 highlights the quality control (QC) indicators evaluated under this data validation procedure.

3. Procedure

The following subsections describe for each of the QC indicators the acceptance criteria, location and retrieval of QC data, evaluation of the QC data, actions taken in the event the QC acceptance criteria are exceeded, and documentation of the QC violations in a standardized report form.

Table M1-VOA-1 QC CHECKLIST FOR LEVEL M1 CLP RAS VOLATILE ORGANICS			
QC MEASURES	Manual		
	M1	M2	M3
Action Level Notification	X	X	X
Instrument Tune		X	X
Initial Calibration (RRF)		X	X
Initial Calibration (%RSD)		X	X
Continuing Calibration (RRF)		X	X
Continuing Calibration (%D)		X	X
Laboratory Blank	X	X	X
MS/MSD (%R, RPD)		X	X
Internal Standard Area		X	X
Field Blank	X	X	X
Sample Paperwork	X	X	X
Holding Time		X	X
Retention Time	X	X	X
Surrogate Recovery		X	X
Dilution Factor		X	X
Moisture Content			X
Mass Spectra	X		X
Chromatograms	X	X	X
Raw Data			X

Reporting requirements for Level M1 are:

- Hand annotate the Form I's, including
  - Data validation qualifiers
  - Sample identification number
  - Sampling location

- Provide a narrative that includes
  - A statement that defines the level of the data review, i.e., M1
  - Major problems associated with analysis

- Include the following attachments
  - List of data validation qualifiers
  - Support documentation including forms that support assigning data qualifiers
  - Chain of custody form

3.1 Action Level Notification

The purpose behind action level notification is to make the EPA Remedial Project Officer (RPM) or the Site Project Officer (SPO) aware of the potential human health risk at the site. In accordance with the Region III Hazardous Waste Division policy, the EPA RPM or SPO must be promptly notified of any contaminant exceeding the established action level or the 10-day health advisory limit. The data for contaminants exceeding the action levels must be validated as a top priority and reported to the RPM or SPO. Validation of the rest of the data may then be completed normally.

3.1.1 Acceptance Criteria

EPA's Office of Solid Waste and Emergency Response has established 10-day health advisory limits or the action levels for several organic compounds and elements of special health risk concern based on the Safe Drinking Water Act. The volatile organic compounds and their 10-day health advisory limits are listed in Table M1-VOA-2. The criteria for action level notification are as follows:

- The contaminant concentration must be equal to or above the established 10-day health advisory limits.

- Data for contaminants exceeding the action levels must be validated as a top priority.

- The following EPA personnel must be notified of the action level exceedances:

- EPA RPM or SPO

- EPA Section Chiefs:

  - Site Investigation (SI)

  - Remedial

  - Enforcement

  - RCRA

- EPA Section Toxicologists:

  - Enforcement

  - Superfund

  - RCRA

- The remaining data validation should be completed per normal procedures.

- Any special instructions from the Hazardous Waste Division should be followed.

- Records should be kept of the data review, action level notification and any follow-up instructions and actions.

Table M1-VOA-2 VOLATILE ORGANIC ANALYTES AND ACTION LEVELS			
Compound	Action Level*	Compound	Action Level*
Benzene	233	Carbon tetrachloride	160
Chlorobenzene	1,800	1,2-Dichloroethane	740
1,1-Dichloroethylene	1,000	cis-1,2-Dichloroethylene	1,000
trans-1,2-Dichloroethylene	2,720	Dichloromethane (methylene chloride)	1,500
1,2-Dichloropropane	90	Ethylbenzene	2,100
Methylethyl ketone (MEK, 2-butanone)	7,500	Styrene	20,000
Tetrachloroethylene	34,000	Toluene	6,000
1,1,1-Trichloroethane	35,000	Vinyl chloride	2,600
Xylenes	7,800	–	–
*All units are ug/l.			

3.1.2 Review Items

All data required to perform the complete Level M1 validation, as detailed in the following sections, are necessary for carrying out action level notification. The location of the data and their retrieval procedures are also discussed below.

3.1.3 Evaluation Procedure

The evaluation process preceding action level notification will primarily consist of comparing the results on Form I's with the action levels presented in Table M1-VOA-2. Following the identification of the contaminants exceeding the action levels, focused data validation should be performed using the criteria, and procedures described in the appropriate sections below.

3.1.4 Action

The action resulting from focused data validation will be the notification of action level exceedance to the personnel identified above in Section 3.1.1.

3.1.5 Reporting

Copies of Form I's can be used to highlight the contaminants above the action levels. The findings of the focused validation can be summarized in a memorandum, and the data qualifiers resulting from focused validation may be written on the Form I's. The marked up forms should be clarified that they represent validation of only the contaminants exceeding the action levels, and not all data.

3.2 Evaluation of Mass Spectra For Detected Compounds

The primary QC indicator checked in Level M1 is the mass spectra for the detected compounds. This indicator pertains to evaluating a compound's presence by matching its mass spectrum with a standard (known) mass spectrum for the compound. No assessment is made of the reported quantity of the compound or any quantitative quality control indicators that could lend some uncertainty to the reported value.

3.2.1 Acceptance Criteria

The acceptance criteria for mass spectral matching are given in the CLP Functional Guidelines and are as follows:

- All ions present in the standard (known) spectrum at a relative intensity greater than 10 percent must be present in the sample mass spectrum.

- The relative intensities of the qualifying ions (those above 10 percent relative intensity) must be within +/-20 percent between the standard and sample spectra, for example, an ion with a relative abundance of 50 percent in the standard spectrum must be present between a relative abundance of 30 and 70 percent in the sample spectrum).

- Ions present in the sample above 10 percent relative abundance must be accounted for.

3.2.2 Review Items

Form I's for each field sample, field blanks, and laboratory blanks included in a Sample Delivery Group (SDG) are necessary to compile a list of the detected compounds. Mass spectra are then necessary for each of the detected compounds in a sample. The required mass spectra include both the sample spectra as well as the standard spectra.

With respect to the blanks, it may be beneficial to evaluate the blanks before the sample mass spectra are evaluated. If a compound is found to be a common contaminant, i.e., it is present in any one of the blanks and also in the sample, the concentration in the sample should be evaluated with respect to the highest blank contamination using the 5 (or 10)

times criteria before proceeding with the elaborate mass spectral evaluations. The effort required for the sample mass spectral evaluation may be significantly reduced by performing the blank evaluation first. The mass spectra for the blanks; however, should be evaluated to ensure that the contaminants were identified properly.

All sample Form I's are generally located together in front of the data package. Copies of the Form I's can also be found in the detailed sample data package organized by sample; i.e., the Form I and its substantiating raw data for a sample are placed together. The raw data include a quantitation report and mass spectra for each detected compounds in that sample are. A standard (known) mass spectrum, and two sample mass spectra (one unaltered and another background-subtracted) are generally provided. The presentation of the mass spectra differs for different instrument manufacturers, but information necessary for evaluating mass spectra is always provided in some form. Hands-on experience with different brands of mass spectrometers, although not essential, can be helpful in interpreting the information.

Copies should be made of the Form I's, preferably from the summary data package, if included. Otherwise, the forms can be pulled out from the raw sample data package. All detects should then be highlighted with a marker or other convenient means. The raw data should then be tagged for every sample for evaluating the mass spectra and the chromatograms (reconstructed ion current profiles).

3.2.3 Evaluation of Mass Spectra

Compare the highlighted Form I's with the tagged raw sample data. Verify that sample results are present for all of the samples listed on the chain-of-custody form. Verify that raw data are provided for every sample, and a Form I exists for every sample represented by raw data. Verify on a sample-by-sample basis that mass spectra are provided for every detected (highlighted) compound reported on the Form I's. A cross verification should then be performed that all target compounds for which mass spectra are provided are also reported as detected on Form I's.

Mass spectral comparison routines based on the evaluation criteria presented earlier (presence of major ions and their relative intensities) are built into the instrument software; therefore, performed automatically. A listing of ions and their relative intensities is not produced; rather a score based on the extent of the fit of all criteria is produced. The score is based on a basis of 100 or 1000 for a perfect match of the presence of major ions and their relative abundances. The score is printed on the quantitation report as a "q" value. A score upward of 60 percent of the maximum (upward of 60 or 600) is generally considered acceptable for positive identification for a compound.

Visual comparison of a mass spectrum involves looking for the base ion (the mass fragment with the highest intensity), the parent ion (mass fragment equal to the molecular weight of the compound) and other characteristic ions representing removal of one or more functional

groups (such as -CH$_3$, -CH$_2$-, -Cl, -OH, or a combination thereof) from the parent ion, the base ion or other ions. The removal of functional groups is often successive and produces a fingerprint pattern for a type of compound; i.e., straight-chain hydrocarbons produce a characteristic envelope of mass fragments separated by 14 mass units. Similarly, aromatic compounds produce a peculiar fingerprint. The base ion represents the most stable mass fragment; therefore, it is always present. However, a parent ion often is unstable and may not exist at all or may exist at a low abundance or relative intensity.

Compare the standard (known) mass spectrum with the background-subtracted sample spectrum for the presence of the base, parent and other characteristic ions. Although background-subtracted and unaltered mass spectra are generally provided, the former type of spectra are much cleaner looking due to the subtraction of column bleed or other broad-based interferences; therefore, better suited for comparison. Generally, the presence of major ions and overall matching of the fingerprint-pattern between the standard and the sample spectra can be considered satisfactory. The comparison is rather subjective, and requires a trained-eye to deduce the information.

Interference still may be present in a background-subtracted mass spectrum due to co-eluting compounds (as compared to column bleed or broad-based interferences). Unless the interfering compound is an isomer or an analog of the target compound in question, the fingerprints produced by the target compound and the interfering compounds can be quite different. Inseparable isomers reflect a limitation of the gas chromatography without any recourse. Such isomers are reported as "total", for example, total xylenes (combination of ortho-, meta- and para-xylenes). Analogous compounds generally have different retention times; therefore, may not be of much concern. Thus, co-eluting compounds do not pose any problems in evaluating the mass spectra.

Seldom does a CLP laboratory incorrectly interpret a mass spectrum, but there is a subjective element to mass spectral interpretation. While working within the CLP guidelines, the laboratories do have their own reporting practices at the limit of the instrument's sensitivity. Most of the mass spectral identification problems occur near the limit of detection where the differences in the relative intensities are not easily discernible. Special attention should be paid in evaluating mass spectra for compounds detected at low concentrations. In addition, oxygenated compounds such as acetone and 2-butanone produce poor mass spectra. For such compounds, the standard spectrum may show the presence of the parent ion, but the same may not be true of the sample spectrum. Professional judgment should be exercised in evaluating such mass spectra.

Rearrangements and other side-reactions often occur inside a mass spectrometer. These phenomena produce mass fragments that are not easily accountable from the structure of the parent compound.

3.2.4 Action

In the event the mass spectrum of a detected compound does not at all resemble the standard spectrum or has extremely poor matching, the compound should be considered undetected. In this case the detected value should be changed to the CRDL for that sample and flagged as undetected, "U." (NOTE: This action is in contrast to the EPA Functional Guidelines which recommend rejection of data. It is felt that an outright rejection of data is not justified. If the mass spectrum produced at a target compound's retention time does not match the standard spectrum, a conclusion can be drawn that the target compound is not present and the mass spectrum may be due to something else. In such a case the target compound should be considered as undetected and data should not be rejected. Make sure that the target compound in question does produce a good mass spectrum by inspecting the standard mass spectrum from the calibration and the fit score on the quantitation report for the daily or continuing calibration. Data for undetected compounds are usable for many purposes such as risk assessment; therefore, rejection of data is not beneficial to a project. The conflict of improper characterization should be brought up and rectified with the laboratory.)

If there is some evidence of the compound's presence (as determined by visual matching of the base ion, parent ion and fingerprint pattern despite a poor fit score), the compound should be considered tentatively identified. The reported value should not be altered in this case; however, a data qualifier code, "N", should be appended to the data point to denote tentative identification.

In both of the above cases, evaluation of additional information such as the retention time and frequency of detection in other field samples is warranted. Previous site history is also an important evaluation; however, information essential to perform such an evaluation (in contrast to 'validation') may not be easily available to a data validator. Nonetheless, it is incumbent upon a data validator to make appropriate recommendations to the project manager or the remedial officer.

3.2.5 Reporting

The most convenient form of reporting the mass spectral evaluation is through a brief memorandum to appropriate authorities. The highlighted Form I's and the mass spectra in question should be appended to the memorandum in support of the conclusions. Also fill out and attach the Mass Spectral Evaluation Form, as depicted in Table M1-VOA-3, to the report as a record of what was done. Record the date(s) of analyses. Field sample numbers should be transcribed from the chain-of-custody in the Sample Identifier column. Notations may be placed under the "MS" column for each sample to describe the mass spectral evaluation. The following notations are suggested:

- X—Acceptable mass spectrum.

- N—Tentative identification. Partial proof of a compound's presence, but all identification criteria not met.

- U—No mass spectral match. Using professional judgment, the compound is considered undetected.

- R—No mass spectral match. Using professional judgment, the compound data are rejected from further use.

3.3 Evaluation of Chromatograms

The purpose behind evaluating the chromatograms is to get an idea regarding potential false negatives, and gross analytical errors. Checking for positive data as described under mass spectral evaluation does not offer any insight into data that are not reported (i.e., reported as nondetects). Laboratory error or gross interference from other compounds could be the reasons for erroneous reporting. The gas chromatograms or the reconstructed ion current (RIC) profiles are the primary tools used for the false negative evaluation under Level M1.

3.3.1 Acceptance Criteria

There are no EPA-established criteria for evaluating chromatograms for false negatives. The criteria used for evaluating chromatograms are based on good laboratory and scientific practices, and these are not hard and fast requirements. The suggested evaluation criteria are:

- There should not be any significant peaks in the chromatograms that are not accounted for as TCLs or TICs. Significant peaks are those with a minimum peak height of 10 percent of the closest internal standard.

- The chromatograms should ideally have base-line resolution between adjacent peaks. Also, there should not be broad (unresolved) envelopes in the chromatograms.

- There should not be abrupt shifts in the baseline.

- There should not be peak tailing or sharp rise in the peak fronts.

Table M1-VOA-3
VOA MASS SPECTRAL EVALUATION SUMMARY

Dates Analyzed:	Sample Identifier:	Comments on Chromatogram
	1.	1.
	2.	2.
	3.	3.
	4.	4.
Instrument ID:	5.	5.
	6.	6.
	7.	7.
	8.	8.
	9.	9.
	10.	10.

SPCC(#), CCC(*) Aromatic (AR) Compound:	Sample Identifier																			
	1		2		3		4		5		6		7		8		9		10	
	MS	RT	MS	RT	MS	RT	MS	RT	MS	RT	MS	RT	MS	RT	MS	RT	MS	RT	MS	RT
Chloromethane (#)																				
Bromomethane																				
Vinyl Chloride (*)																				
Chloroethane																				
Methylene Chloride																				
Acetone																				
Carbon Disulfide																				
1,1-Dichloroethene (*)																				
1,1-Dichloroethane (#)																				
1,2-Dichloroethene (total)																				
Chloroform (*)																				
1,2-Dichloroethane																				
2-Butanone																				
1,1,1-Trichloroethane																				
Carbon Tetrachloride																				
Bromodichloromethane																				
1,2-Dichloropropane (*)																				
cis-1,3-Dichloropropene																				
Trichloroethene																				
Dibromochloromethane																				
1,1,2-Trichloroethane																				
Benzene AR																				
trans-1,3-Dichloropropene																				
Bromoform (#)																				
4-Methyl-2-Pentanone																				
2-Hexanone																				
Tetrachloroethene																				
1,1,2,2-Tetrachloroethane (#)																				
Toluene (*)(AR)																				
Chlorobenzene (#)(AR)																				
Ethylbenzene (*)(AR)																				
Styrene (AR)																				
Xylenes (total) (AR)																				

3.3.2 Review Items

Chromatograms, or the RIC profiles for each sample are necessary for the evaluation. The RIC profiles can be found in the front of the raw data package for each sample. The quantitation report for each sample is also necessary to retrieve either the retention times or information regarding scan numbers to compare with the peaks on the chromatograms.

3.3.3 Evaluation of Chromatograms

Visually inspect the chromatograms for all peaks that appear to be at least 10 percent in height of the nearest internal standard. Compare that the scan number or the retention time that appears on the x-axis of the RIC profile with that listed on the quantitation report for the TCLs or the library search record for the TICs. Make sure that all significant peaks are accounted for.

Also observe the RIC profile for peak resolution between adjacent peaks. Poor peak-to-peak resolution is indicative of degrading performance of the gas chromatographic column. The values obtained from a degrading system are prone to be inaccurate. Obviously certain isomeric or homologous compounds are difficult to separate. But generally, there should be at least 90 percent valley between the neighboring peaks.

Inspect the RIC profile for broad, unresolved envelopes. These are generally indicative of outside interference from a series of homologous compounds such as straight-chain hydrocarbons. Especially, assess the interference with the internal standards and surrogates under the envelope using the expected area counts. The standard values that are far from the expected values may be indicative of potential problems with the TCL detection or quantification.

Inspect the RIC profile for abrupt shifts in the baseline. Such shifts are indicative of problems with instrument sensitivity or leakage in the system. The area counts obtained from shifted baseline are inaccurate, or even the detection of a TCL at low concentration may be missed.

Rapid peak rising or peak tailing indicate problems with the gas chromatographic column, such as depleted stationary phase on the column, decomposition of the stationary phase or creation of active sites. Again, a visual inspection of the RIC profile will yield information on the shape of the peak.

3.3.4 Action

Professional discretion must be used when evaluating and qualifying data based on the chromatographic evaluations. An experienced chemist can generally infer the magnitude and the frequency of the problem from the RIC profile. If the problem appears to be

systematic, then it should be brought to the laboratories attention and resolved. Intermittent problems may or may not require any action. The following guidelines are suggested when acting on RIC profile observations:

- Any unaccounted TCL peak with area equivalent to or greater than the lowest reportable limit for the sample must be brought to the laboratory's attention and resolved. Any unaccounted non-TCL (i.e., TIC) peak with an area equal to or greater than 10 percent area of the nearest internal standard must also be resolved with the laboratory. TICs with less than 10 percent area are not required to be reported according to the CLP-RAS protocols. In the event, the discrepancy cannot be resolved with the laboratory, the problems should be documented and brought to the attention of the CLP-TPO, the RPM and the SM. The data for unreported TCL or TICs may be considered unusable until the problems are resolved.

- If a peak resolution problem is evident for the samples, and appears to be systematic (i.e., present in all calibration samples, QC samples, and field samples and increasing as the run progresses), additional QC measures such as the continuing calibration percent difference (%D), and internal and surrogate standard recoveries in the vicinity of the affected peaks should be evaluated to determine if the peak resolution problem could affect detection or quantification. If determined so, the positive data may be qualified as estimated, "J." Negative data may also be qualified as estimated, "UJ" if the ability to detect at low concentrations is also deemed to be jeopardized by poor resolution of adjacent peaks.

- Broad envelopes of homologous compounds could interfere with quantification or even detection. If the interference is evident from the recoveries of the internal and surrogate standards in the vicinity of the envelope, associated compounds may also be interfered with. Using professional discretion, the positive and negative data may be considered as estimated, "J" and "UJ", respectively. If the project objectives cannot be met with the estimated data, alternative sample preparation and cleanup procedures may need to be developed and specified. The recommended solution should be brought to the attention of the RPM, the SM and the TPO.

- Discrete shifts in the baseline in the middle of a run are indicative of intermittent problems. If the shift is due to leakage or change in the system pressure, the positive as well as negative data may be considered estimated ("J" and "UJ," respectively). The problem could also be due to some fluctuation in the instrument electronics which may lead to drastic changes in the sensitivity of the instrument to detect the compounds. As a note, professional judgment should be exercised in determining the severity of the

problem. For example, the magnitude of a drop in the baseline below the zero line may not be estimated and could be very significant. On the other hand, a drop that yields a baseline still above zero can be put in a perspective with the original baseline and a general appearance of the entire RIC profile.

- The problems with peak symmetry are indicative of system degradation, and should be brought to the attention of the laboratory for a corrective action. Professional judgment should be used when and if qualifying any data due to unsymmetrical peaks. First the problem should be defined in terms of persistence throughout the chromatogram and also from sample to sample. Additionally, the shapes and area counts for the internal and surrogate standards should be evaluated to see if the problem could have affected compound detection and/or quantification. Data qualification may be uncalled for if the standard area counts are acceptable.

3.3.5 Reporting

The Mass Spectral Evaluation Form (Table M1-VOA-3) presented earlier may be used to note any brief comments on the chromatographic evaluations. The comments may be noted against each sample identifier. For more descriptive comments, a separate sheet may be used.

3.4 Evaluation of Retention Times

While not unequivocal in identifying a compound, the retention times are quite helpful in confirming the presence of a detected compound. Matching of mass spectrum and retention time of sample with those of a standard yields higher credibility and confidence level to the detection in the sample. On the other hand, not matching the retention time may or may not invalidate the detection. If a mass spectral match is made beyond any doubt, poor matching of the retention time may not have adverse impact on the detection. If mass spectral matching is unacceptable or only partial, and the retention times do not match, then a strong doubt can be cast on a compound's presence.

3.4.1 Acceptance Criteria

The criteria for retention times are specified in the EPA's functional guidelines as follows:

The relative retention times (RRTs) must be within +/-0.06 RRT units of the applicable internal standard RRT.

3.4.2 Review Items

Quantitation reports for the sample and continuing calibration are required for the evaluation. These are located in the raw data for samples and standards. A copy of the continuing calibration quantitation reports may be made or the reports pulled out from the raw data to facilitate a comparison with the sample quantitation reports.

3.4.3 Evaluation Procedures

For the detected compounds, determine the relative retention time for the compounds by dividing their retention times with the retention time of their associated internal standard in the samples as well as in the applicable continuing (or initial) calibrations. The sample RRTs must fall in range of standard RRT +/- 0.06 units.

3.4.4 Action

Action for retention time evaluation requires professional discretion. Action taken must be based on other data such as mass spectra and not on retention times alone. The following actions are suggested for several potential situations.

- Acceptable matching of the mass spectra and the RRTs—No action suggested.

- Acceptable matching of the mass spectra but poor matching of the RRTs— No action suggested, but the cause of retention time shift may be investigated. Check to see if similar discrepancies are observed in other parts of the chromatogram. Often high concentration of a compound will cause shifts in the retention times for other compounds in the nearby eluting region, but shifts in retention times in other areas may not be observed. Shifts all through the chromatogram may be indicative of an erratic system, such as flow rate fluctuations, poor temperature regulation, restriction or leakage in the system. There are likely chances that the sample values in such conditions may be inaccurate. Positive data may be qualified as estimated, "J," if deemed essential.

3.4.5 Reporting

The M1-VOA-MS Form (Table M1-VOA-3) presented earlier may be used to record any problems in the RRT matching. For the detected compounds in each sample, the calculated RRTs may be recorded under the "RT" column. A data qualifier code may be added to the values exceeding 0.06 RRT and requiring qualification, such as "0.15J," for a compound with a difference of 0.15 RRT units and where a professional judgment to estimate the data is exercised.

3.5 Evaluation of Blanks

Laboratory blanks and field blanks have a profound impact on false positives reported in samples; i.e., compounds reported as positive detects but not originating from the samples themselves. Cross contamination from the sampling equipment, incidental contamination from the field conditions or contamination from the laboratory equipment or general environmental are likely sources of false positives in the samples.

3.5.1 Acceptance Criteria

Criteria for blank evaluation are specified in the EPA's functional guidelines. In addition, Region III has some additional requirements modifying the guidance. The acceptance criteria for blanks apply equally to any type of blanks associated with either sampling or analysis, such as trip blanks, rinsate blanks, field-or bottle blanks, and laboratory method blanks. While there are several criteria for evaluating the blanks, the only criterion applicable to Level M1 is the comparison of the blank and sample concentrations. This criterion is:

> For common contaminants, such as methylene chloride, acetone and 2-butanone, the sample concentration must be minimally 10 times the blank concentration to be considered a positive detect. Other contaminants must be present in the sample at or above 5 times the blank concentration. The blank with the highest concentration must be used (this is, if the laboratory blank has the highest concentration of methylene chloride, and the equipment rinsate blank has the highest concentration of acetone, both of these blanks must be used to qualify the respective contaminants). All blanks should be checked and the highest concentration of contaminants in any blank should be used for data qualification.

3.5.2 Review Items

Data requirements and data retrieval procedures for blanks are the same as those for the field samples because the blanks as well as field samples are validated similarly. Form I's, mass spectra, chromatograms, quantitation reports, etc., are essential for performing a validation of the blanks first.

3.5.3 Evaluation Procedure

Validate the blanks the same as the field samples. detailed validation procedures are described above under appropriate sections. Use the validated blank results for a comparison with the sample results. Make certain that the samples and blanks are evaluated on the same basis of sample weight or volume, dilution factors, moisture content, etc. Use the 5 (or 10) times the highest blank concentrations for qualifying the sample data.

3.5.4 Action

If the sample concentrations do not meet the criteria of 5 (or 10) times the blank concentration, the sample results should be considered essentially undetected (or as not detected substantially above the levels reported in the blanks); therefore, flagged "B" in accordance with the Region III data validation guidelines.

3.5.5 Reporting

Form I's may be used to write the "B" data qualifier for the data not meeting the blank criteria.

3.6 Sample Paperwork

The purpose for evaluating the sample paperwork is to determine that the samples being validated are indeed the ones taken from the site, and have not been tampered with. Accurate sample identity is of paramount importance in substantiating the sample data. Without unequivocal sample identity and chain-of-custody procedures, the sample data may not be defensible or enforceable.

Under the current CLP contracts, the original paperwork (i.e., the purge package or the administrative record) is included in the data package from the laboratory. It is assumed that the data validator is not privy to the original paperwork; therefore, the evaluation criteria and procedures described below apply only to the documents that are generally included in the data validation package. These documents are the chain-of-custody forms and Region III Shipping Record.

3.6.1 Acceptance Criteria

Criteria for acceptability or authenticity of the sampling paperwork, document control and chain-of-custody have been established by the National Enforcement Investigations Center (NEIC), in support of the CLP. Overall criteria are too numerous and subjective to be discussed here, but the criteria that apply to data validation are as follows:

- The chain-of-custody form should be properly and completely filled out including the sampler signatures, date and time of sampling, sampling station identification, analyses requested, traffic numbers, tag numbers, etc. These data are minimally required to confirm the authenticity of the sample and its data.

- The chain-of-custody must be maintained at all times. The custody transfers between different parties must be signed and dated.

3.6.2 Review Items

A copy of the chain-of-custody form is essential to confirm the identity of the samples. In addition, the Region III Shipping Record is essential to identify the field QC samples. The chain-of-custody and Shipping Record are generally located in front of the data package.

3.6.3 Evaluation Procedure

Ensure that the chain-of-custody form was signed and dated by the samplers, and a time and date were entered for sample collection. The laboratory copy of the chain-of-custody must have the signature of the laboratory sample custodian. Any errors on the form should have been crossed out with a single line through the entry. Verify that all collected samples have unique station identification, traffic numbers and sample tag numbers. Ensure that the Region III Shipping Record correctly reflects the information on the chain-of-custody.

3.6.4 Action

The action to be taken in qualifying the data is highly dependent on the nature of the problem. Some errors in paperwork are practically unavoidable in real situations. An effort should be made to reconcile the differences by cross checking the field notebooks against the sampling paperwork. Occasionally, the samplers may forget to sign the chain-of-custody; however, the field notebooks may amply describe the sampling event. Problems are also inevitable in noting or cross-referencing sample tag numbers and traffic numbers. Generally, there are several alternate sources of information to substantiate or refute the problem.

3.6.5 Reporting

Any discrepancies found in the paperwork must be immediately brought to the attention of the EPA RPM or SPO. Clearly define the problems in a memorandum to the responsible parties. Attach marked copies of the chain-of-custody forms to substantiate the findings.

Appendix A-2
Validation of Volatile Organic Analyte Data
Manual Level M2

1. Purpose and Applicability

This procedure provides step-by-step instructions to manually validate the volatile organic analyte (VOA) data using the manual innovative data validation approach at Level M2. This approach focuses on the use of information contained on the CLP forms and a review of chromatograms as summarized in Table M2-VOA-1. The procedures are based on modifications to Regions III's National Functional Guidelines for Organic Data Review.

The procedure is applicable to the VOA data obtained using the Contract Laboratory Program Statement of Work (CLP SOW). Hard copy data conforming to the CLP SOW specifications are essential in order to carry out the procedure.

Data validated using this procedure are considered usable for the following types of purposes; however, the data users must decide on a case-by-case basis whether the procedure is suitable for their intended data uses. The suggested data uses are:

- Oversight of activities led by other parties

- Comparison to action levels

- Initial site investigation

- Contamination sources

- Nature and extent of contamination

- Preliminary risk assessment

- Risk assessment with known high levels of toxics

- Feasibility study

- Preliminary design

- Treatability study

- Initial cleanup verification

Table M2-VOA-1
QC CHECKLIST FOR LEVEL M2
CLP RAS VOLATILE ORGANICS

QC MEASURES	Manual			Combined		
	M1	M2	M3	C1	C2	C3
Action Level Notification	X	X	X	X	X	X
Instrument Tune		X	X	X	X	X
Initial Calibration (RRF)		X	X	X	X	X
Initial Calibration (%RSD)		X	X	X	X	X
Continuing Calibration (RRF)		X	X	X	X	X
Continuing Calibration (%D)		X	X	X	X	X
Laboratory Blank	X	X	X	X	X	X
MS/MSD (%R,RPD)		X	X	X	X	X
Internal Standard Area		X	X	X	X	X
Field Blank	X	X	X	X	X	X
Sample Paperwork	X	X	X		X	X
Holding Time		X	X	X	X	X
Retention Time	X	X	X	X	X	X
Surrogate Recovery		X	X	X		X
Dilution Factor		X	X	X		X
Moisture Content		X	X	X		X
Mass Spectra	X		X		X	X
Chromatograms	X	X	X		X	X
Raw Data			X			X

2. Quality Control Measures Checked

Table M2-VOA-1 highlights the quality control (QC) indicators evaluated under this data validation procedure.

3. Procedure

The following subsections describe for each of the QC indicators the acceptance criteria, data requirements and retrieval of QC data, evaluation of the QC data, actions taken in the event the QC acceptance criteria are exceeded, and documentation of the QC violations in a standardized report form.

The volatile data requirements to be checked are listed below:

3.1   Action Level Notification

3.2   Technical Holding Times (CCS - Contractual holding times only)

3.3   GC/MS Instrument Performance Check (CCS)

3.4   Initial Calibration (CCS)

3.5   Continuing Calibration (CCS)

3.6   Blanks

3.7   System Monitoring Compounds (CCS)

3.8   Matrix Spike/Matrix Spike Duplicate

3.9   Internal Standards

3.10   Reported Contract Required Quantitation Limits (CRQLs)

3.11   Tentatively Identified Compounds

Two forms have been developed to assist in the performance and documentation of implementing Level M2. The first form, M2-VOA-QUAL, summarizes holding time, calibrations, blanks, surrogates, and internal standards. The second form, M2-VOA-SPK, summarizes surrogate and matrix spike quality control checks. These forms are presented in Table M2-VOA-2 and Table M2-VOA-3.

Reporting requirements for Level M2 are:

- Hand annotate the Form I's, including
  - Data validation qualifiers
  - Sample identification number
  - Sampling location

- Provide a narrative that includes
  - A statement that defines the level of the data review, ie. M2
  - Major and minor problems associated with the analysis
  - Highlight issues that may have affected detection limits

- Include the following attachments
  - List of data validation qualifiers
  - Support documentation including forms that support assigning data qualifiers
  - Chain of custody form
  - Samples affected by calibration should be listed on the appropriate calibration forms

The data qualifiers assigned in this review are as follows:

Codes Relation to Identification (Confidence concerning presence or absence of compounds)

U   =   Not detected. The associated number indicates approximate sample concentration necessary to be detected.

(NO CODE) =   Confirmed identification.

B   =   Not detected substantially above the level reported in laboratory or field blanks.

R   =   Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.

N   =   Tentative identification. Consider present. Special methods may be needed to confirm its presence or absence in future sampling efforts.

Case No.._____
SDG No.:_____
Data Reviewer:_____
Site:_____

Procedure No.: M2-VOA
Revision: 1
Date: 06/30/1995
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Table M2-VOA-2. VOA Qualifier Summary (Calibrations, Blanks, Holding Time, Surrogates, Internal Standards)

Date(s) Analyzed:

Samples Analyzed Within 12 Hours of Tune? __Yes __No

Instrument ID:

Tune OK? __Yes __No

Method Blank ID:

Sample Identifier:	Hold Time Out, days		Standards: (<,>)					
			Surrogates			ISTDs		
	AR	All	S1	S2	S3	IS1	IS2	IS3
1.								
2.								
3.								
4.								
5.								
6.								
7.								
8.								
9.								
10.								

Date: _____   Time: _____   _____

Compound: SPCC (#) CCC(•) Aromatic (AR)	Initial Cal.		Continuing Cal.		Blanks		Qualifiers
	RRF<.05	%RSD>30	RRF<.05	%D>25	Meth.	Trip	(+/-)
Chloromethane (#)							
Bromomethane							
Vinyl Chloride (•)							
Chloroethane							
Methylene Chloride							
Acetone							
Carbon Disulfide							
1,1-Dichloroethene (•)							
1,1-Dichloroethane (#)							
1,2-Dichloroethene (total)							
Chloroform (•)							
1,2-Dichloroethane							
2-Butanone							
1,1,1-Trichloroethane							
Carbon Tetrachloride							
Bromodichloromethane							
1,2-Dichloropropane (•)							
cis-1,3-Dichloropropene							
Trichloroethene							
Dibromochloromethane							
1,1,2-Trichloroethane							
Benzene AR							
trans-1,3-Dichloropropene							
Bromoform (#)							
4-Methyl-2-Pentanone							
2-Hexanone							
Tetrachloroethene							
1,1,2,2-Tetrachloroethane (#)							
Toluene (•)(AR)							
Chlorobenzene (#)(AR)							
Ethylbenzene (•)(AR)							
Styrene (AR)							
Xylenes (total) (AR)							

Qualifier column notes (right side): I S # 1 ; I S # 2 ; I S # 3

Reported as: RT   (μg/kg, μg/L)

TICs Reported In Blank(s):

Case No.:_____
SDG No.:_____
Data Reviewer:_____
Site:_____

Procedure No.: M2-VOA
Revision: 1
Date: 06/30/1995
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Table M2-VOA-3. VOA Surrogate and Matrix Spike Quality Control Summary

Surrogate Data Summary

Sample Identifier:	Aqueous Sample Recoveries, %R			Soil Sample Recovery, %R			Qualifiers (+/-)
	S1	S2	S3	S1	S2	S3	
(Acceptance Range, %R):	88-110	86-115	76-114	84-138	59-113	70-121	
1.							
2.							
3.							
4.							
5.							
6.							
7.							
8.							
9.							
10.							
11.							
12.							
13.							

S1 = Toluene-d8, S2 = Bromofluorobenzene, S3 = 1,2-dichloroethane-d4

MS/MSD Data Summary

Spike Compound:Aromatic (AR)	SPCC (#) CCC (*)	Matrix Spike, Recovery, %R		Matrix Spike Duplicate, Recovery, %R		MS/MSD Precision, RPD		Qualifiers (+/-)
		Range	Actual	Range	Actual	Range	Actual	
AQUEOUS SAMPLES								
1,1-Dichloroethene	(*)	61-145		61-145		14		
Trichloroethene		71-120		71-120		14		
Benzene	AR	76-127		76-127		11		
Toluene	(*)(AR)	76-125		76-125		13		
Chlorobenzene	(#)(AR)	75-130		75-130		13		
SOIL SAMPLES								
1,1-Dichloroethene	(*)	59-172		59-172		22		
Trichloroethene		62-137		62-137		24		
Benzene	AR	66-142		66-142		21		
Toluene	(*)(AR)	59-139		59-139		21		
Chlorobenzene	(#)(AR)	60-133		60-133		21		

Codes Related to Quantitation (Can be used for both positive results and sample quantitation limits):

J $=$ Analyte present. Reported value may not be accurate or precise.

K $=$ Analyte present. Reported value may be biased high. Actual value is expected lower.

L $=$ Analyte present. Reported value may be biased low. Actual value is expected to be higher.

UJ $=$ Not detected, quantitation limit may be inaccurate or imprecise.

UL $=$ Not detected, quantitation limit is probably higher.

Other Codes

Q $=$ No analytical result.

* $=$ Results reported from diluted analysis.

3.1 Action Level Notification

The purpose behind action level notification is to make the EPA Remedial Project Officer (RPM) or the Site Project Officer (SPO) aware of the potential human health risk at the site. In accordance with the Region III Hazardous Waste Division policy, the EPA RPM or SPO must be promptly notified of any contaminant exceeding the established action level or the 10-day health advisory limit. The data for contaminants exceeding the action levels must be validated as a top priority and reported to the RPM or SPO. Validation of the rest of the data may then be completed normally.

3.1.1 Acceptance Criteria

EPA's Office of Solid Waste and Emergency Response has established 10-day health advisory limits or the action levels for several organic compounds and elements of special health risk concern based on the Safe Drinking Water Act. The volatile organic compounds and their 10-day health advisory limits are listed in Table M2-VOA-4. The criteria for action level notification are as follows:

- The contaminant concentration must be equal to or above the established 10-day health advisory limits.

- Data for contaminants exceeding the action levels must be validated as a top priority.

- The following EPA personnel must be notified of the action level exceedances:

    - EPA RPM or SPO

    - EPA Section Chiefs:

        - Site Investigation (SI)

        - Remedial

        - Enforcement

        - RCRA

    - EPA Section Toxicologists:

        - Enforcement

        - Superfund

        - RCRA

- The remaining data validation should be completed per normal procedures.

- Any special instructions from the Hazardous Waste Division should be followed.

- Records should be kept of the data review, action level notification and any follow up instructions and actions.

Table M2-VOA-4 VOLATILE ORGANIC ANALYTES AND ACTION LEVELS			
Compound	Action Level*	Compound	Action Level*
Benzene	233	Carbon tetrachloride	160
Chlorobenzene	1,800	1,2-Dichloroethane	740
1,1-Dichloroethylene	1,000	cis-1,2-Dichloroethylene	1,000
trans-1,2-Dichloroethylene	2,720	Dichloromethane (methylene chloride)	1,500
1,2-Dichloropropane	90	Ethylbenzene	2,100
Methylethyl ketone (MEK, 2-butanone)	7,500	Styrene	20,000
Tetrachloroethylene	34,000	Toluene	6,000
1,1,1-Trichloroethane	35,000	Vinyl chloride	2,600
Xylenes	7,800	--	--
*All units are ug/l.			

3.1.2 Review Items

All data required to perform the complete Level M2 validation, as detailed in the following sections are necessary for carrying out action level notification. The location of the data and their retrieval procedures are also discussed below.

3.1.3 Evaluation Procedure

The evaluation process preceding action level notification will primarily consist of comparing the results on Form I's with the action levels presented in Table M2-VOA-4. Following the identification of the contaminants exceeding the action levels, focused data validation should be performed using the criteria, and procedures described in the appropriate sections below.

3.1.4 Action

The action resulting from focused data validation will be the notification of action level exceedance to the personnel identified above in Section 3.1.1.

3.1.5 Reporting

Copies of Form I's can be used to highlight the contaminants above the action levels. The findings of the focused validation can be summarized in a memorandum, and the data qualifiers resulting from focused validation may be written on the Form I's. The marked up forms should be clarified that they represent validation of only the contaminants exceeding the action levels and not all data.

3.2 Technical Holding Times

The objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of analysis.

3.2.1 Acceptance Criteria

Technical requirements for sample holding times have only been established for water matrices. The holding times for soils (and other non-aqueous matrices such as sediments, oily wastes, and sludge) are currently under investigation. In Region III, a 14 day holding time will be applied to all non-aqueous samples. When soil holding time criteria are established and available, the procedure for qualifying soil samples will be re-evaluated.

The holding time criteria for water samples, as stated in the current 40 CFR Part 136 (Clean Water Act) is as follows:

> For non-aromatic volatile compounds in cooled (@ 4°C) water samples, the maximum holding time is 14 days from sample collection.

> Maximum holding times for purgeable aromatic hydrocarbons in cooled (@ 4°C ± 2°C), acid-preserved (pH 2 or below) water samples are 14 days from sample collection.

> Water samples that have not been maintained at 4°C (± 2°C) and/or preserved to a pH of 2 or below should be analyzed within 7 days from sample collection. If insufficient ice is used to ship samples, the laboratory may receive samples with no ice left in the cooler. Under these circumstances, the temperature of the samples may exceed 4°C.

It is further required that volatile compounds in properly preserved non-aqueous samples be analyzed within 14 days of sample collection for all volatile compounds.

3.2.2 Review Items

Form I VOA, EPA Sample shipping log and/or chain-of-custody.

3.2.3 Evaluation Procedure

Technical holding times are established by comparing the sampling dates on the EPA Sample Traffic Report with dates of analysis on Form I VOA. Examine the sample records to determine if samples were preserved. If it is indicated that there were problems with the samples, then the integrity of the sample may have been compromised and professional judgment should be used to evaluate the effect of the problem on the sample results.

3.2.4 Action

1. If technical holding times are exceeded, qualify the sample results as follows. (Also see Table M2-VOA-5).

   If there is no evidence that the samples were properly preserved and the technical holding times exceeded 7 days, qualify positive results with "L" and sample quantitation limits with "UL" for aromatic compounds. Use professional judgment to determine if and how non-aromatic volatile compounds should also be qualified.

   If the samples were properly preserved but the technical holding times exceeded 14 days, qualify all positive results with "L" and all sample quantitation limits with "UL."

Table M2-VOA-5 QUALIFICATION OF VOLATILE ANALYTES BASED ON TECHNICAL HOLDING TIMES			
Matrix	Preserved	> 7 Days	14 Days
Water	No	All Aromatics*	All Compounds
Water	Yes	None	All Compounds
Non-aqueous	No/Yes	None	All Compounds
*Reviewer should use professional judgment to determine if data for additional compounds require qualification.			

2. If technical holding times are grossly exceeded (e.g., by greater than two times the required time for volatiles) either on the first analysis or upon re-analysis, the reviewer must use professional judgment to determine the reliability of the data and the effects of additional storage on the sample results. Should the reviewer determine that qualification is necessary, non-detected volatile target compounds

may be qualified unusable (R). Positive results are considered bias low and are qualified with "L".

3. When there are other quality control problems in conjunction with exceeded holding times (such as suspected laboratory contamination), the reviewer should follow the hierarchy of qualifiers. In particular, if for any reason the reviewer doubts the presence of a compound, the data summary should display only the "B" or "R" qualifier and not the "L" qualifier. This is because no net direction of bias can be inferred under these conditions. When results are reported by the laboratory as below the CRQL, the "L" qualifier is used over the "J" qualifier.

4. Customized Qualifier Summary Forms should be filled to show holding time performance (one per 12-hour period per instrument).

3.3 GC/MS Instrument Performance Check

Gas chromatograph/mass spectrometer (GC/MS) instrument performance checks (formerly referred to as tuning) are performed to ensure mass resolution, identification, and to some degree, sensitivity. These criteria are not sample specific. Conformance is determined using standard materials, therefore, these criteria should be met in all circumstances.

3.3.1 Acceptance Criteria

The analysis of the instrument performance check solution must be performed at the beginning of each 12-hour period during which samples or standards are analyzed. The instrument performance check, bromofluorobenzene (BFB) for volatile analysis, must meet the ion abundance criteria given below.

Bromofluorobenzene (BFB)

m/z	Ion Abundance Criteria
50	8.0 - 40.0% of m/z 95
75	30.0 - 66.0% of m/z 95
95	Base peak, 100% relative abundance
96	5.0 - 9.0% of m/z 95
173	Less than 2.0% of m/z 174
174	50.0 - 120.0% of m/z 95
175	4.0 - 9.0% of mass 174
176	93.0 - 101.0% of m/z 174
177	5.0 - 9.0% of m/z 176

NOTE: All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120 percent that of m/z 95.

3.3.2 Review Items

Form V VOA.

3.3.3 Evaluation Procedure

1. Compare the data presented for each Instrument Performance Check (Form V VOA) with each mass listing submitted to ensure the following:

    - Form V VOA is present and completed for each 12-hour period during which samples were analyzed.

    - The appropriate number of significant figures has been reported (number of significant figures given for each ion in the ion abundance criteria column) and that rounding is correct. (See SOW for requirements).

2. Verify that the mass assignment is correct and that the mass listing is normalized to m/z 95.

3. Verify that the ion abundance criteria was met. The criteria for m/z 173, 176, and 177 are calculated by normalizing to the specified m/z.

NOTE: All instrument conditions must be identical to those used in the sample analysis.

3.3.4 Action

1. If the laboratory has made minor transcription errors which do not significantly affect the data, the data reviewer should make the necessary corrections on a copy of the form.

2. If the laboratory has failed to provide the correct forms or has made significant transcription or calculation errors, the Region's designated representative should contact the laboratory and request corrected data. If the information is not available then the reviewer must use professional judgment to assess the data. This should be noted on the ORDAS form.

3.   If mass assignment is in error (such as m/z 96 is indicated as the base peak rather than m/z 95), classify all associated data as unusable (R).

4.   If ion abundance criteria are not met, professional judgment may be applied to determine to what extent the data may be utilized. Guidelines to aid in the application of professional judgment to this topic are discussed as follows:

The most important factors to consider are the empirical results that are relatively insensitive to location on the chromatographic profile and the type of instrumentation. Therefore, the critical ion abundance criteria for BFB are the m/z 95/96, 174/175, 174/176, and 176/177 ratios. The relative abundances of m/z 50 and 75 are of lower importance.

5.   If the reviewer has reason to believe that instrument performance check criteria were achieved using techniques other than those described in II.D.4, then additional information on the instrument performance checks should be obtained. If the techniques employed are found to be at variance with the contract requirements, the performance and procedures of the laboratory may merit evaluation. Concerns or questions regarding laboratory performance should be noted for TPO action in the ORDAS form. For example, if the reviewer has reason to believe that an inappropriate technique was used to obtain background subtraction (such as background subtracting from the solvent front or from another region of the chromatogram rather than the BFB peak), then this should be noted for TPO action in the ORDAS form.

3.4 Initial Calibration

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for compounds on the volatile target compound list (TCL). Initial calibration demonstrates that the instrument is capable of acceptable performance in the beginning of the analytical run and of producing a linear calibration curve.

3.4.1 Acceptance Criteria

1.   Initial calibration standards containing both volatile target compounds and system monitoring compounds are analyzed at concentrations of 10, 20, 50, 100, and 200 ug/L at the beginning of each analytical sequence or as necessary if the continuing calibration acceptance criteria are not met. The initial calibration (and any associated samples and blanks) must be analyzed within 12 hours of the associated instrument performance check.

2. Separate initial calibrations must be performed for water samples (or medium level soil samples) and for low level soil samples. The calibration for water samples and medium level soil samples is performed with an unheated purge and the calibration for low level soil samples is performed with a heated purge.

3. Initial calibration standard Relative Response Factors (RRFs) for volatile target compounds and system monitoring compounds (surrogates) must be greater than or equal to 0.05. (Contractual initial calibration RRF criteria are listed in Appendix A).

4. The Percent Relative Standard Deviation (%RSD) from the initial calibration must be less than or equal to 30.0% for all compounds. (Contractual calibration %RSD criteria are listed in Appendix A).

3.4.2 Review Items

Form VI VOA and chromatograms.

3.4.3 Evaluation Procedures

1. Verify that the correct concentration of standards were used for the initial calibration (i.e., 10, 20, 50, 100, and 200 ug/L for water).

2. Verify that the correct initial calibration was used for water and medium level soil samples (i.e., unheated purge) and for low level soil samples (i.e., heated purge).

3. If any sample results were calculated using an initial calibration, verify that the correct standard (i.e., the 50 ug/L standard) was used for calculating sample results and that the samples were analyzed within 12 hours of the associated instrument performance check.

4. Evaluate the initial calibration RRFs and RRFs for all volatile target compounds and system monitoring compounds (surrogates):

    Verify that for all volatile target compounds and system monitoring compounds, the initial calibration RRFs are greater than or equal to 0.05.

NOTE: Because historical performance data indicate poor response and/or erratic behavior, the volatile compounds listed below have no contractual maximum %RSD criteria. Contractually they must meet a minimum RRF criterion of 0.01; however, for data review purposes, the "greater than or equal to 0.05" criterion is applied to all volatile compounds.

Volatile Target Compounds Exhibiting Poor Response

- Acetone
- 2-Butanone
- Carbon disulfide
- Chloroethane
- Chloromethane
- 1,2-Dichloroethene (total)
- 1,2-Dichloropropane
- 2-Hexanone
- Methylene chloride
- 4-Methyl-2-pentanone
- **Toluene-d8**
- **1,2-Dichloroethane-d4**

NOTE: Compounds in bold are system monitoring compounds.

5. Evaluate the %RSD for all volatile target compounds and system monitoring compounds:

a. Verify that all volatile target compounds have a %RSD of less than or equal to 30.0%. The contractual criteria for an acceptable initial calibration specifies that up to any 2 volatile target compounds may fail to meet minimum RRF or maximum %RSD as long as they have RRFs that are greater than or equal to 0.010, and %RSD of less than or equal to 40.0%. For data review purposes, however, all compounds must be considered for qualification when the %RSD exceeds the ± 30.0% criterion.

b. If the %RSD is greater than 30.0%, then the reviewer should use professional judgment to determine the need to check the points on the curve for the cause of the non-linearity. This is checked by eliminating either the high point or the low point and recalculating the %RSD.

3.4.4 Action

1. All volatile target compounds, including the "poor performers" listed above (excluding the system monitoring compounds) will be qualified using the following criteria:

a. If the %RSD is greater than 30.0% and all initial calibration RRFs greater than or equal to 0.05, qualify positive results with "J". Non-detects are not qualified. When the %RSD is grossly exceeded (i.e., > 50%) use professional judgment for qualifying non-detects as "UJ".

b.   If any initial calibration RRF is less than 0.05, qualify positive results that have acceptable mass spectral identification with "L", and non-detected analytes as unusable, "R".

2. At the reviewer's discretion, a more in-depth review to minimize the qualification of data can be accomplished by considering the following:

a.   If any of the required volatile compounds have a %RSD greater than 30.0%, and if eliminating either the high or the low point of the curve does not restore the %RSD to less than or equal to 30.0%:

   i.   Qualify positive results for that compound(s) with "J".

   ii.   No qualifiers are needed for volatile target compounds that were not detected. If the %RSD is grossly exceeded (i.e., >50%), professional judgment is used to qualify non-detects with "UJ".

b.   If the high point of the curve is outside of the linearity criteria (e.g., due to saturation):

   i.   No qualifiers are required for positive results in the linear portion of the curve.

   ii.   Qualify positive results outside of the linear portion of the curve with a "J".

   iii. No qualifiers are needed for volatile target compounds that were not detected. If the %RSD is grossly exceeded (i.e., >50%, professional judgment is used to qualify non-detects with "UJ".

c.   If the low end of the curve is outside of the linearity criteria:

   i.   No qualifiers are required for positive results in the linear portion of the curve.

   ii.   Qualify low level positive results in the area of non-linearity with "J".

   iii. No qualifiers are needed for volatile target compounds that were not detected. If the %RSD is grossly exceeded (i.e., >50%), professional judgment is used to qualify non-detects with "UJ".

NOTE: If a, b, or c options are used, a description of the process must be clearly stated in the data review narrative.

3. If the laboratory has failed to provide adequate calibration information, the designated representative should contact the laboratory and request the necessary information. If the information is not available, the reviewer must use professional judgment to assess the data.

4. The potential effects on the data due to unacceptable calibration criteria should be noted in the data review narrative.

5. If calibration criteria are exceeded, this should be noted on the ORDAS.

3.5 Continuing Calibration

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data. Continuing calibration establishes the 12-hour relative response factors on which the quantitations are based and checks satisfactory performance of the instrument on a day-to-day basis.

3.5.1 Acceptance Criteria

1. Continuing calibration standards containing both target compounds and system monitoring compounds are analyzed at the beginning of each 12-hour analysis period following the analysis of the instrument performance check and prior to the analysis of the method blank and samples. The continuing calibration may either be a part of the initial calibration or run independently on another 12-hour analysis period.

2. The continuing calibration RRF for volatile target compounds and system monitoring compounds must be greater than or equal to 0.05.

3. The percent difference (%D) between the initial calibration RRF and the continuing calibration RRF must be within $\pm$ 25.0%.

3.5.2 Review Items

Review Items: Form VII VOA and chromatograms.

3.5.3 Evaluation Procedure

1. Verify that the continuing calibration was run at the required frequency and that the continuing calibration was compared to the correct initial calibration.

2. Evaluate the continuing calibration RRF for all volatile target compounds and system monitoring compounds:

    Verify that all volatile compounds and system monitoring compounds meet the RRF specifications.

    NOTE: Because historical performance data indicate poor response and/or erratic behavior, the compounds listed in Section 3.4.3, above, have no contractual maximum %D criteria. Contractually they must meet a minimum RRF criterion of 0.01; however, for data review purposes, the "greater than or equal to 0.05" criterion is applied to all volatile compounds.

3. Evaluate the %D between initial calibration RRF and continuing calibration RRF for one or more compound(s):

    Verify that the %D is within ± 25.0% for all volatile target compounds and system monitoring compounds. Note those compounds which have a %D outside the ± 25.0% criterion. The contractual criteria for an acceptable continuing calibration specifies that up to any 2 volatile target compounds may fail to meet minimum RRF or maximum %D as long as they have RRFs that are greater than or equal to 0.010, and %D of less than or equal to 40.0%. For data review purposes, however, all compounds must be considered for qualification when the %D exceeds the ± 25.0% criterion.

3.5.4 Action

1. The reviewer should use professional judgment to determine if it is necessary to qualify the data for any volatile target compound. If qualification of data is required, it should be performed using the following guidelines:

    a.   If the %D is outside the ± 25.0% criterion and the continuing calibration RRF is greater than or equal to 0.05, qualify positive results with "J".

    b.   If the %D is outside the ± 25.0% criterion and the continuing calibration RRF is greater than or equal to 0.05, no qualification of non-detected volatile target compounds is necessary. If the %D is grossly exceeded (>50%), professional judgment may be used to qualify non-detects with "UJ".

    c.   If the continuing calibration RRF is less than 0.05, qualify positive results that have acceptable mass spectral identifications with "L" or use professional judgment and include justification for not qualifying the data in the data review narrative.

d. If the continuing calibration RRF is less than 0.05, qualify non-detected volatile target compounds as unusable, "R".

2. If the laboratory has failed to provide adequate calibration information, the designated representative should contact the laboratory and request the necessary information. If the information is not available, the reviewer must use professional judgment to assess the data.

3. The potential effects on the data due to unacceptable calibration criteria should be noted in the data review narrative.

4. If calibration criteria are exceeded, this should be noted in the ORDAS.

3.6. Blanks

The purpose of laboratory (or field) blank analysis is to determine the existence and magnitude of contamination resulting from laboratory (or field) activities. The criteria for evaluation of blanks apply to any blank associated with the samples (e.g., methods blanks, instrument blanks, trip blanks, and equipment blanks). If problems with any blank exist, all associated data must be carefully evaluated to determine whether or not there is an inherent variability in the data, or if the problem is an isolated occurrence not affecting other data.

3.6.1 Acceptance Criteria

1. No contaminants should be found in the blanks.

2. A method blank analysis must be performed after the calibration standards and once for every 12-hour time period beginning with the injection of BFB.

3. The method blank must be analyzed on each GC/MS system used to analyze samples for each type of analysis, i.e., unheated purge (water and medium level soil) and heated purge (low level soil).

4. An instrument blank should be analyzed after any sample that has exceeded the initial calibration range for any given compound to check that the blank is free of interference and the system is not contaminated.

3.6.2 Review Items

Form I VOA, Form IV VOA and chromatograms.

3.6.3 Evaluation Procedures

1. Review the results of all associated blanks on the forms and chromatograms to evaluate the presence of target and non-target compounds in the blanks.

2. Verify that a method blank analysis has been reported per matrix, per concentration level for each 12-hour time period on each GC/MS system used to analyze volatile samples. The reviewer can use the Method Blank Summary (Form IV VOA) to identify the samples associated with each method blank.

3. Verify that the instrument blank analysis has been performed following any sample analysis where a target analyte(s) is reported at high concentration(s).

3.6.4 Action

If the appropriate blanks were not analyzed with the frequency described in Criteria 2, 3, and 4, then the data reviewer should use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. The situation should be noted for TPO action on the ORDAS form.

Action regarding unsuitable blank results depends on the circumstances and origin of the blank. Positive sample results should be reported and qualified "B", if the concentration of the compound in the sample is less than or equal to 10 times (10x) the amount in any blank for the common volatile laboratory contaminants (methylene chloride, acetone, and 2-butanone), or 5 times (5x) the amount for other volatile target compounds. In situations where more than one blank is associated with a given sample, qualification should be based upon a comparison with the blank having the highest concentration of a contaminant. The results must not be corrected by subtracting any blank value.

For qualification purposes, consider all blanks in a case associated with all samples.

Field blanks measure contamination introduced not only in the field but also from the laboratory. In general, evaluation of the impact on specific sample results is handled the same as with laboratory blanks. The reviewer should use caution in attributing contamination to the field as opposed to laboratory sources. However, when field-introduced contamination is suspected, it is helpful for the reviewer to consult the sampling group to identify possible sources and prevent future reoccurrences. Verified field sources of contamination should be noted in the data review narrative. If a field blank has the highest concentration of a contaminant, then all samples in the associated case are qualified "B", using the 5x and 10x rule. Other field blanks associated with the case are not qualified.

Specific actions are as follows:

1. If a volatile compound is found in a blank but <u>not</u> found in the sample, no action is taken. If the contaminants found are volatile target compounds (or interfering non-target compounds) at significant concentrations above the CRQL, then this should be noted for TPO action in the ORDAS.

2. Any volatile compound detected in the sample (other than the common volatile laboratory contaminants), that was also detected in any associated blank, is qualified "B", when the sample concentration is less than five times (5x) the blank concentration. For common volatile laboratory contaminants, the results are qualified "B", when the sample concentration is less than 10 times (10x) the blank concentration.

3. The reviewer should note that blanks may not involve the same weights, volumes, or dilution factors as the associated samples. These factors must be taken into consideration when applying the "5x" and "10x" criteria, such that a comparison of the total amount of contamination is actually made.

   Additionally, there may be instances where little or no contamination was present in the associated blanks, but qualification of the sample is deemed necessary. If the reviewer determines that the contamination is from a source other than the sample, he/she should qualify the data. Contamination introduced through dilution water is one example. Although it is not always possible to determine, instances of this occurring can be detected when contaminants are found in the diluted sample result but are absent in the undiluted sample result. Since both results are not routinely reported, it may be impossible to verify this source of contamination.

4. If gross contamination exists (i.e., saturated peaks by GC/MS), all affected compounds in the associated samples should be qualified as unusable (R) due to interference. This should be noted for TPO action in the ORDAS if the contamination is suspected of having an effect on the sample results.

5. If inordinate numbers of other target compounds are found at low levels in the blank(s), it may be indicative of a problem and should be noted for TPO action in the ORDAS form.

6. The same consideration given to the target compounds should also be given to Tentatively Identified Compounds (TICs), which are found in both the sample and associated blank(s). (See VOA Section XIII for TIC guidance.)

7. If an instrument blank was not analyzed following a sample analysis which contained an analyte(s) at high concentration(s), sample analysis results after the high concentration sample must be evaluated for carryover. Professional judgment should be used to determine if instrument cross-contamination has affected any positive compound identification(s). If instrument cross-contamination is suggested, then this should be noted for TPO action if the cross-contamination is suspected of having an effect on the sample results. Sample results which are possible artifacts of carry-over should be flagged as unusable (R).

8. When there is convincing evidence that contamination is restricted to a particular instrument, matrix, or concentration level, the 5x/10x rule will only be applied to compare contaminated blanks to certain associated samples (as opposed to all samples in the case). Some examples are as follows:

   Column bleed (siloxanes) may be localized to a particular instrument.

   Methanol extractions in the medium soil volatile analysis protocol can give rise to contaminants that are not seen in the low-level aqueous analyses.

   Common laboratory contaminants, such as methylene chloride, are generally too unpredictable to safely assume contamination is restricted to a particular instrument, matrix, or concentration level.

9. For benzene and/or toluene, the reviewer may identify that the observed laboratory contamination is attributable to a specific, regular, and predictable process (such as trap bleed), which results in a constant 1 or 2 ppb instrument level concentration in all runs (both samples and blanks). In this situation, the reviewer may want to consider and flag certain results as tentatively identified, "N", as opposed to "B", if the sample instrument level is clearly greater than the consistent level of contamination detected in blanks and other samples. (This particular situation supersedes the 5x/10x rule.)

10. The following are examples of applying the blank qualification guidelines. Certain circumstances may warrant deviations from these guidelines. Any deviations must be clearly stated in the data review narrative.

Example 1: Sample result is greater than the Contract Required Quantitation Limit (CRQL), but is less than the 5x or 10x multiple of the blank result.

	Rule	
	10x	5x
Blank Result	7	7
CRQL	5	5
Sample Result	60	30
Final Sample Result	60B	30B

In the example for the "10x" rule, sample results less than 70 (or 10 x 7) would be qualified "B". In the case of the "5x" rule, sample results less than 35 (or 5 x 7) would be qualified "B".

Example 2: Sample result is less than the CRQL, and is also less than the 5x or 10x multiple of the blank result.

	Rule	
	10x	5x
Blank Result	6	6
CRQL	5	5
Sample Result	4J	4J
Final Sample Result	4B	4B

Note that data are reported as 4B, indicating that the qualitative presence is not confirmed.

Example 3: Sample result is greater than the 5x or 10x multiple of the blank result.

	Rule	
	10x	5x
Blank Result	10	10
CRQL	5	5
Sample Results	120	60
Final Sample Result	120	60

For both the "10x" and "5x" rules, sample results exceeded the adjusted blank result of 100 (or 10 x 10) and 50 (or 5 x 10), respectively.

3.7 System Monitoring Compounds (Surrogate Spikes)

Laboratory performance on individual samples is established by means of spiking activities. All samples are spiked with system monitoring compounds (formerly referred to as surrogates) prior to sample purging. The evaluation of the results of these system monitoring compounds is not necessarily straightforward. The sample itself may produce effects due to such factors as interferences and high concentrations of analytes. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the evaluation and review of data based on specific sample results is frequently subjective and demands analytical experience and professional judgment. Accordingly, this section consists primarily of guidelines, in some cases with several optional approaches suggested.

3.7.1 Acceptance Criteria

1. Three system monitoring compounds (1,2-dichloroethane-d4, bromofluorobenzene, and toluene-d8) are added to all samples and blanks to measure their recovery in environmental samples and blank matrices.

2. Recoveries for system monitoring compounds in volatile samples and blanks must be within the limits specified in Appendix A and the SOW.

3.7.2 Review Items

Form II VOA and chromatograms.

3.7.3 Evaluation Procedures

1. Check chromatograms to verify the recoveries on the System Monitoring Compound Recovery Form—Form II VOA.

2. The following should be determined from the System Monitoring Compound Recovery form(s):

   a. If any system monitoring compound(s) in the volatile fraction is out of specification, there should be a reanalysis to confirm that the non-compliance is due to sample matrix effects rather than laboratory deficiencies.

   NOTE: When there are unacceptable system monitoring compound recoveries followed by successful analyses, the laboratories are required to report only the successful run.

b.   The laboratory failed to perform acceptably if system monitoring compounds are outside criteria with no evidence of re-analysis. Medium soils must first be re-extracted prior to re-analysis when this occurs.

c.   Verify that no blanks have system monitoring compounds outside the criteria.

3. Any time there are two or more analyses for a particular sample, the reviewer must determine which are the best data to report. Considerations should include but are not limited to:

a.   System monitoring compound recovery (marginal versus gross deviation).

b.   Technical holding times.

c.   Comparison of the values of the target compounds reported in each sample analysis.

d.   Other QC information, such as performance of internal standards.

3.7.4 Action

Data are qualified based on system monitoring compounds results if the recovery of any volatile system monitoring compound is out of specification. For system monitoring compound recoveries out of specification, the following approaches are suggested based on a review of all data from the package, especially considering the apparent complexity of the sample matrix. (Also, see Table M2-VOA-6, below).

1. If a system monitoring compound in the volatile sample has a recovery greater than the upper acceptance limit:

a.   Detected volatile target compounds are qualified "J".

b.   Results for non-detected volatile target compounds should be qualified "UJ"

2. If a system monitoring compound in the volatile sample has a recovery greater than or equal to 10% but less than the lower acceptance limit:

a.   Detected volatile target compounds are qualified "J".

b.   For non-detected volatile target compounds, the sample quantitation limit is qualified as approximated, "UJ".

3. If a system monitoring compound in a volatile sample shows less than 10% recovery:

    a.   Detected volatile compounds are qualified "L".

    b.   Non-detected volatile target compounds are qualified as unusable, "R".

4. If two or three system monitoring compounds in the volatile sample have recoveries outside acceptance limits, refer to Table M2-VOA-6.

5. In the special case of a blank analysis with system monitoring compounds out of specification, the reviewer must give special consideration to the validity of associated sample data. The basic concern is whether the blank problems represent an isolated problem with the blank alone, or whether there is a fundamental problem with the analytical process. For example, if one or more samples in the batch show acceptable system monitoring compound recoveries, the reviewer may choose to consider the blank problem to be an isolated occurrence. However, even if this judgment allows some use of the affected data, analytical problems should be noted for TPO action on the ORDAS. Also note if there are potential contractual problems associated with the lack of reanalysis of samples that were out of specification.

6. Whenever possible, potential effects of the data resulting from system monitoring recoveries not meeting the advisory limits should be noted in the data review narrative.

7. Positive results for compounds already flagged for blank contamination, "B", will not need a separate flag for system monitoring compound recoveries. However, these situations should be addressed in the data review narrative and the support documentation.

Table M2-VOA-6 QUALIFICATION OF VOLATILE ANALYTES BASED ON SYSTEM MONITORING COMPOUND RECOVERIES					
	1 or more < 10%	1 High/Low	2 or 3 High/Low	2 or 3 All Low	2 or 3 All High
Detected Analytes	L	J	J	L	K
Non-Detected Analytes	R	UJ	UJ	UL	None

8. When dilutions are performed which prevent detection of system monitoring compounds, the data review narrative and support documentation should indicate that extraction efficiency/method accuracy cannot be verified.

9. When both the initial analysis and the reanalysis have system monitoring compound recoveries outside of criteria, the data summary form should normally contain the highest concentration obtained for each compound detected, provided that system monitoring compound recoveries in the analysis being reported do not suggest a high bias. However, if a demonstrated laboratory contaminant is detected in one analysis but not in the other, the negative result may be more appropriate to report.

When the reanalysis of a sample is within the system monitoring compound recovery criteria, the laboratory is required to provide only data for the acceptable analysis. If both sets of data are provided, and if a compound was detected in the initial analysis but not in the reanalysis, then the positive result should be reported (provided the compound is not a demonstrated laboratory contaminant). The reported result should be flagged as estimated (J), due to possible sample inhomogeneity.

3.8 Matrix Spike/Matrix Spike Duplicate

Data for matrix spike/matrix spike duplicates (MS/MSD) are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. These data alone cannot be used to evaluate the precision and accuracy of individual samples. However, when exercising professional judgment, this data should be used in conjunction with other available QC information.

3.8.1 Acceptance Criteria

1. Matrix spike (MS) and matrix spike duplicate (MSD) samples are analyzed at a frequency of one MS and MSD per 20 samples of similar matrix.

2. Spike recoveries should be within the advisory limits provided on Form III VOA-1 and VOA-2.

3. Relative percent difference (RPD) between MS and MSD recoveries must be within the advisory limits provided on Form III VOA-1 and VOA-2.

3.8.2 Review Items

Form III VOA-I and VOA-2, chromatograms.

3.8.3 Evaluation Procedures

1. Verify that MS and MSD samples were analyzed at the required frequency and that results are provided for each sample matrix.

2. Inspect results for the MS/MSD Recovery on Form III VOA-1 and VOA-2 and verify that the results for recovery and RPD are within the advisory limits.

3. Check that the matrix spike recoveries and RPDs were calculated correctly.

4. Compare %RSD results of non-spiked compounds between the original result, MS, and MSD.

3.8.4 Action

1. No action is taken on MS/MSD data alone. However, using informed professional judgment, the data reviewer may use the MS and MSD results in conjunction with other QC criteria to determine the need for some qualification of the data.

2. The data reviewer should first try to determine to what extent the results of the MS/MSD affect the associated data. This determination should be made with regard to the MS/MSD sample itself as well as specific analytes for all samples associated with the MS/MSD.

3. In those instances where it can be determined that the results of the MS/MSD affect only the sample spiked, then qualification should be limited to this sample alone. However, it may be determined through the MS/MSD results that a laboratory is having a systematic problem in the analysis of one or more analytes, which affects all associated samples.

4. The reviewer must use professional judgment to determine the need for qualification of positive results of non-spiked compounds.

5. When non-spiked compounds are present in either the MS or MSD results, a table in the data review narrative is constructed showing original (unspiked) sample results for non-spiked compounds, non-spiked compounds present in the MS and MSD and the calculated %RSD.

   NOTE: If a field blank was used for the MS/MSD, a statement to that effect must be included on the ORDAS and noted for the TPO.

3.9 Internal Standards

Internal Standards (IS) performance criteria ensures that GC/MS sensitivity and response are stable during each analysis.

3.9.1 Acceptance Criteria

1. Internal standard area counts must not vary by more than a factor of two (-50% to +100%) from the associated calibration standard.

2. The retention time of the internal standard must not vary more than ±30 seconds from the retention time of the associated calibration standard.

3.9.2 Data Requirements and Data Retrieval

Form VII VOA and chromatograms.

3.9.3 Evaluation Procedures

1. Check the internal standard retention times and areas reported on the Internal Standard Area Summary (Form VIII VOA).

2. Verify that all retention times and IS areas are within criteria.

3. If there are two analyses for a particular fraction, the reviewer must determine which are the best data to report. Considerations should include:

    a. Magnitude and direction of the IS area shift.

    b. Magnitude and direction of the IS retention time shift.

    c. Technical holding times.

    d. Comparison of the values of the target compounds reported in each fraction.

    c. Other QC.

3.9.4 Action

1. If an IS area count for a sample or blank is outside -50% or +100% of the area for associated standard, then:

    a. Positive results for compounds quantitated using that IS should be qualified with "J".

    b. Non-detected compounds quantitated using an IS area count greater than ± 100% should be qualified "UJ".

c. Non-detected compounds quantitated using an IS area count less than 50% are reported as the associated sample quantitation limit and qualified with "UJ".

d. If extremely low area counts are reported, or if performance exhibits a major abrupt drop-off then a severe loss of sensitivity is indicated. Non-detected target compounds should then be qualified as unusable, "R".

2. If an IS retention time varies by more than 30 seconds:

The chromatographic profile for that sample must be examined to determine if any false positives or negatives exist. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for that sample fraction. Positive results should not need to be qualified as "R", if the mass spectral criteria are met.

3. If the internal standards performance criteria are grossly exceeded, then this should be noted for TPO action in the ORDAS. Potential affects on the data resulting from unacceptable internal standard performance should be noted in the data review narrative.

3.10 Reported CRQLs

The objective is to ensure that the Contract Required Quantitation Limits (CRQLs) are accurate.

3.10.1 Acceptance Criteria

1. The adjustment of the CRQLs must be calculated according to the correct equation to account for dilution and moisture content as appropriate.

3.10.2 Review Items

Form I VOA and chromatograms.

3.10.3 Evaluation Procedures

1. Verify that the CRQLs have been adjusted to reflect all sample dilutions and dry weight factors that are not accounted for by the method.

3.10.4 Action

1. If any discrepancies are found, the laboratory may be contacted by the designated representative to obtain additional information that could resolve any differences. If a

discrepancy remains unresolved, the reviewer must use professional judgment to decide which value is the best value. Under these circumstances, the reviewer may determine qualification of data is warranted. A description of the reasons for data qualification and the qualification that is applied to the data should be documented in the data review narrative and in the document support.

2. The reviewer must assure that any results in error by more than 10 percent are identified and corrected on the sample data summary. If laboratory resubmission is not performed, the reviewer should document his/her changes to the data in the narrative and support documentation. Calculation errors should also be noted on the ORDAS.

3. Numerous or significant failures to properly evaluate and adjust CRQLs should be noted for TPO action on the ORDAS.

3.11 Tentatively Identified Compounds

Chromatographic peaks in volatile fraction analyses that are not target analytes, system monitoring compounds or internal standards are potential tentatively identified compounds (TICs).

3.11.1 Acceptance Criteria

For each sample, the laboratory must conduct a mass spectral search of the NIST library and report the possible identity for the 10 largest volatile fraction peaks which are not system monitoring compounds, internal standards, or target compounds, but which have an area or height greater than 10 percent of the area or height of the nearest internal standard. TIC results are reported for each sample on the Organic Analyses Data Sheet (Form I VOA-TIC).

> NOTE: Since the SOW revision of October 1986, the CLP does not allow the laboratory to report as tentatively identified compounds any target compound which is properly reported in another fraction. For example, late eluting volatile target compounds should not be reported as semivolatile TICs.

3.11.2 Review Items

Form I VOA-TIC chromatograms.

3.11.3 Evaluation Procedures

1. Blank chromatograms should be examined to verify that TIC peaks present in samples are not found in blanks. When a low-level non-target compound that is a common

artifact or laboratory contaminant is detected in a sample, a thorough check of blank chromatograms may require looking for peaks which are less than 10 percent of the internal standard height, but present in the blank chromatogram at a similar relative retention time.

2. The reviewer should be aware of common laboratory artifacts/contaminants and their sources (e.g., aldol condensation products, solvent preservatives, and reagent contaminants). These may be present in blanks and not reported as sample TICs.

    Examples:

    a.   Common laboratory contaminants: CO_2 (m/z 44), siloxanes (m/z 73), diethyl ether, hexane, certain freons (1,1,2-trichloro-1,2,2-trifluoroethane or fluorotrichloromethane), and phthalates at levels less than 100 ug/L or 4000 ug/Kg.

    b.   Solvent preservatives such as cyclohexene which is a methylene chloride preservative. Related by-products include cyclohexanone, cyclohexenone, cyclohexanol, cyclohexenol, chlorocyclohexene, and chlorocyclohexanol.

    c.   Aldol condensation reaction products of acetone include: 4-hydroxy-4-methyl-2-pentanone, 4-methyl-2-penten-2-one, and 5,5-dimethyl-2(5H)-furanone.

3. Occasionally, a target compound may be identified in the proper analytical fraction by non-target library search procedures, even though it was not found on the quantitation list. If the total area quantitation method was used, the reviewer should request that the laboratory recalculate the result using the proper quantitation ion. In addition, the reviewer should evaluate other sample chromatograms and check library reference retention times on quantitation lists to determine whether the false negative result is an isolated occurrence or whether additional data may be affected.

4. Target compounds could be identified in more than one fraction. Verify that quantitation is made from the proper fraction.

5. TIC concentration should be estimated assuming a RRF of 1.0.

3.11.4 Action

1. All TIC results should be qualified "J", estimated concentration, on the laboratory Form I-TICs.

2. General actions related to the review of TIC results are as follows:

If all contractually required peaks were not library searched and quantitated, the designated representative could request these data from the laboratory.

3. Blank Results

    Form I-TIC which contains sample results that are questioned by laboratory results, should be flagged "B" and a line drawn through these data for emphasis (initialed and dated), on the Form I-TIC that is included in the validation report.

    To be considered questionable, a sample TIC concentration must be within 10 times the concentration of one of the blank results. If different volumes/weights are used, the total amount of compound in the extract must be compared for sample versus blank. For VOA data, an instrument level comparison is used unless the contamination is proven to originate during sample storage (before preparation/analysis). In general, blanks analyzed within the same case, by the same lab, may be cross-applied to either soil or water samples extracted or analyzed on other days.

    To question a sample result, only presumptive evidence for the presence of the compound in the blank is necessary. The presence of the TIC in the blank is suggested in any of the following situations:

    All blank results must be attached in the support documentation section of the data review.

4. When a compound is not found in any blanks, but is a suspected artifact of common laboratory contaminant, the result may be qualified as unusable, "R", and a line drawn through the result (initialed and dated) on a copy of the Form I-TIC that is included in the validation report.

5. Physical constants, such as boiling point, may be factored into professional judgment of TIC results.

6. Failure to properly evaluate and report TICs should be noted for TPO action on the ORDAS form.

Appendix B
Validation of Semivolatile Organic Analyte Data

- Subappendix B-1 covers Level M1

- Subappendix A-2 covers Level M2

Appendix B
Validation of Semivolatile Organic Analyte Data

- Subappendix B-1 covers Level M1

- Subappendix A-2 covers Level M2

Appendix B-1
Validation of Semivolatile Organic Analyte Data Manual Level M1

1. Purpose and Applicability

This procedure provides step-by-step instructions to manually validate the semivolatile organic analyte (SVOA) data using the manual innovative data validation approach at Level M1.

The procedure is applicable to the SVOA data obtained using the Contract Laboratory Program Statement of Work (CLP SOW). Hard copy data conforming to the CLP SOW specifications are essential in order to carry out the procedure.

Data validated using this procedure are considered usable for the following types of purposes; however, the data users must decide on a case-by-case basis whether the procedure is suitable for their intended data uses. The suggested data uses are:

- Oversight of activities led by other parties

- Comparison to action levels

- Initial site investigation

- Contamination sources

2. Quality Control Measures Checked

Table M1-SVOA-1 highlights the quality control (QC) indicators evaluated under this data validation procedure.

3. Procedure

The following subsections describe for each of the QC indicators the acceptance criteria, location and retrieval of QC data, evaluation of the QC data, actions taken in the event the QC acceptance criteria are exceeded, and documentation of the QC violations in a standardized report form.

Table M1-SVOA-1 QC CHECKLIST FOR LEVEL M1 CLP RAS SEMIVOLATILE ORGANICS			
QC MEASURES	Manual		
	M1	M2	M3
Action Level Notification	X	X	X
Instrument Tune		X	X
Initial Calibration (RRF)		X	X
Initial Calibration (%RSD)		X	X
Continuing Calibration (RRF)		X	X
Continuing Calibration (%D)		X	X
Laboratory Blank	X	X	X
MS/MSD (%R, RPD)		X	X
Internal Standard Area		X	X
Field Blank	X	X	X
Sample Paperwork	X	X	X
Holding Time		X	X
Retention Time	X	X	X
Surrogate Recovery		X	X
Dilution Factor		X	X
Moisture Content			X
Mass Spectra	X		X
Chromatograms	X	X	X
Raw Data			X

Reporting requirements for Level M1 are:

- Hand annotate the Form I's, including
  - Data validation qualifiers
  - Sample identification number
  - Sampling location

- Provide a narrative that includes
  - A statement that defines the level of the data review, i.e., M1
  - Major problems associated with analysis

- Include the following attachments
  - List of data validation qualifiers
  - Support documentation including forms that support assigning data qualifiers
  - Chain of custody form

3.1 Action Level Notification

The purpose behind action level notification is to make the EPA Remedial Project Officer (RPM) or the Site Project Officer (SPO) aware of the potential human health risk at the site. In accordance with the Region III Hazardous waste division policy, the EPA RPM or SPO must be promptly notified of any contaminant exceeding the established action level or the 10-day health advisory limit. The data for contaminants exceeding the action levels must be validated as a top priority and reported to the RPM or SPO. Validation of the rest of the data may then be completed normally.

3.1.1 Acceptance Criteria

EPA's Office of Solid Waste and Emergency Response has established 10-day advisory limits or the action levels for several organic compounds and elements of special health risk concerns based on the Safe Drinking Water Act. The semivolatile organic compounds and their 10-day health advisory limits apply only to aqueous samples and are listed in Table M1-SVOA-2. The criteria for action level notification are as follows:

- The contaminant concentration must be equal to or above the established 10-day health advisory limits.

- Data for contaminants exceeding the action levels must be validated as a top priority.

- The following EPA personnel must be notified of the action level exceedances:

  - EPA RPM or SPO

  - EPA Section Chiefs:

    - Site Investigation (SI)

    - Remedial

    - Enforcement

    - RCRA

  - EPA Section Toxicologists:

    - Enforcement

    - Superfund

    - RCRA

- The remaining data validation should be completed per normal procedures.

- Any special instructions from the Hazardous Waste Division should be followed.

- Records should be kept of the data review, action level notification and any follow up instructions and actions.

Table M1-SVOA-2 SEMIVOLATILE ORGANIC ANALYTES AND ACTION LEVELS			
Compound	Action Level*	Compound	Action Level*
1,3-Dichlorobenzene	8,930	1,4-Dichlorobenzene	10,700
Pentachlorophenol	300	---	---
*All units are ug/l.			

3.1.2 Review Items

All data required to perform Level M1 validation, as detailed in the following sections are necessary for carrying out action level notification. The location of the data and their retrieval procedures are also discussed below.

3.1.3 Evaluation Procedure

The evaluation process preceding action level notification will primarily consist of comparing the results on Form I's with the action levels presented in Table M1-SVOA-2. Following the identification of the contaminants exceeding the action levels, focused data validation should be performed using the criteria, and procedures described in the appropriate sections below.

3.1.4 Action

The action resulting from focused data validation will be the notification of action level exceedance to the personnel identified above in Section 3.1.1.

3.1.5 Reporting

Copies of Form I's can be used to highlight the contaminants above the action levels. The findings of the focused validation can be summarized in a memorandum, and the data qualifiers resulting from focused validation may be written on the Form I's. The marked up forms should be clarified that they represent validation of only the contaminants exceeding the action levels, and not all data.

3.2 Evaluation of Mass Spectra for the Detected Compounds

The primary QC indicator checked in Level M1 is the mass spectra for the detected compounds. This indicator pertains to evaluating a compound's presence by matching its mass spectrum with a standard (known) mass spectrum for the compound. No assessment is made of the reported quantity of the compound or any quantitative quality control indicators that could lend some uncertainty to the reported value.

3.2.1 Acceptance Criteria

The acceptance criteria for mass spectral matching are given in the CLP Functional Guidelines and are as follows:

- All ions present in the standard (known) spectrum at a relative intensity greater than 10 percent must be present in the sample mass spectrum

- The relative intensities of the qualifying ions (those above 10 percent relative intensity) must be within +/-20 percent between the standard and sample spectra, for example, an ion with a relative abundance of 50 percent in the standard spectrum must be present between a relative abundance of 30 and 70 percent in the sample spectrum)

- Ions, present in the sample above 10 percent relative abundance must be accounted for.

3.2.2 Review Items

Form I's for each field sample, field blanks, and laboratory blanks included in a Sample Delivery Group (SDG) are necessary to compile a list of the detected compounds. Mass spectra are then necessary for each of the detected compounds in a sample. The required mass spectra include both the sample spectra as well as the standard spectra.

With respect to the blanks, it may be beneficial to evaluate the blanks before the sample mass spectra are evaluated. If a compound is found to be a common contaminant, i.e., it is present in any one of the blanks and also in the sample, the concentration in the sample should be evaluated with respect to the highest blank contamination using the 5 (or 10) times criteria before proceeding with the elaborate mass spectral evaluations. The effort required for the sample mass spectral evaluation may be significantly reduced by performing the blank evaluation first. The mass spectra for the blanks; however, should be evaluated to ensure that the contaminants were identified properly.

All sample Form I's are generally located together in front of the data package. Copies of the Form I's can also be found in the detailed sample data package organized by sample; i.e., the Form I and its substantiating raw data for a sample are placed together. The raw data include a quantitation report and mass spectra for each detected compounds in that sample are. A standard (known) mass spectrum, and two sample mass spectra (one unaltered and another background-subtracted) are generally provided. The presentation of the mass spectra differs for different instrument manufacturers, but information necessary for evaluating mass spectra is always provided in some form. Hands-on experience with different brands of mass spectrometers, although not essential, can be helpful in interpreting the information.

Copies should be made of the Form I's, preferably from the summary data package, if included. Otherwise, the forms can be pulled out from the raw sample data package. All detects should then be highlighted with a marker or other convenient means. The raw data should then be tagged for every sample for evaluating the mass spectra and the chromatograms (reconstructed ion current profiles).

3.2.3 Evaluation of Mass Spectra

Compare the highlighted Form I's with the tagged raw sample data. Verify that raw data are provided for every sample, and a Form I exists for every sample represented by raw data. Verify on a sample-by-sample basis that mass spectra are provided for every detected (highlighted) compound reported on the Form I's. A cross verification should then be performed that all target compounds for which mass spectra are provided are also reported as detected on Form I's.

Mass spectral comparison routines based on the evaluation criteria presented earlier (presence of major ions and their relative intensities) are built into the instrument software; therefore, performed automatically. A listing of ions and their relative intensities is not produced; rather a score based on the extent of the fit of all criteria is produced. The score is based on a basis of 100 or 1000 for a perfect match of the presence of major ions and their relative abundances. The score is printed on the quantitation report as a "q" value. a score upward of 60 percent of the maximum (upward of 60 or 600) is generally considered acceptable for positive identification for a compound.

Visual comparison of a mass spectrum involves looking for the base ion (the mass fragment with the highest intensity), the parent ion (mass fragment equal to the molecular weight of the compound) and other characteristic ions representing removal of one or more functional groups (such as $-CH_3$, $-CH_2-$, $-Cl$, $-OH$, or a combination thereof) from the parent ion, the base ion or other ions. The removal of functional groups is often successive and produces a fingerprint pattern for a type of compound; i.e., straight-chain hydrocarbons produce a characteristic envelope of mass fragments separated by 14 mass units. Similarly, aromatic compounds produce a peculiar fingerprint. The base ion represents the most stable mass fragment; therefore, it is always present. However, a parent ion often is unstable and may not exist at all or may exist at a low abundance or relative intensity.

Compare the standard (known) mass spectrum with the background-subtracted sample spectrum for the presence of the base, parent and other characteristic ions. Although background-subtracted and unaltered mass spectra are generally provided, the former type of spectra are much cleaner looking due to the subtraction of column bleed or other broad-based interferences; therefore, better suited for comparison. Generally, the presence of major ions and overall matching of the fingerprint pattern between the standard and the sample spectra can be considered satisfactory. The comparison is rather subjective, and requires a trained-eye to deduce the information.

Interference still may be present in a background-subtracted mass spectrum due to coeluting compounds (as compared to column bleed or broad-based interferences). Unless the interfering compound is an isomer or an analog of the target compound in question, the fingerprints produced by the target compound and the interfering compounds can be quite

different. Inseparable isomers reflect a limitation of the gas chromatography without any recourse. Concentrations for such isomers may be reported either as all contribution from one isomer or the value may be divided equally between the two isomers. For example, difficulties are often seen in separating benzo(b)fluoranthene and benzo(k)fluoranthene by gas chromatography. These compounds also produce similar mass spectra; therefore, it is quite difficult to tell them apart. Homologous compounds generally have different retention times; therefore, may not be of much concern. Thus, coeluting compounds do not pose significant problems when evaluating the mass spectra.

Seldom does a CLP laboratory incorrectly interpret a mass spectrum, but there is a subjective element to mass spectral interpretation. While working within the CLP guidelines, the laboratories do have their own reporting practices at the limit of the instrument's sensitivity. Most of the mass spectral identification problems occur near the limit of detection where the differences in the relative intensities are not easily discernible. Special attention should be paid in evaluating mass spectra for compounds detected at low concentrations.

Rearrangements and other side-reactions often occur inside a mass spectrometer. These phenomena produce mass fragments that are not easily accountable from the structure of the parent compound. While it is desirable that a data validator possess the knowledge to interpret complex mass spectra, for the routine CLP analyses, such expertise is unnecessary.

3.2.4 Action

In the event the mass spectrum of a detected compound does not at all resemble the standard spectrum or has extremely poor matching, the compound should be considered undetected. In this case the detected value should be changed to the CRDL for that sample and flagged as undetected, "U." (NOTE: This action is in contrast to the EPA Functional Guidelines which recommend rejection of data. It is felt that an outright rejection of data is not justified. If the mass spectrum produced at a target compound's retention time does not match the standard spectrum, a conclusion can be drawn that the target compound is not present and the mass spectrum may be due to something else. In such a case the target compound should be considered as undetected and data should not be rejected. Make sure that the target compound in question does produce a good mass spectrum by inspecting the standard mass spectrum from the calibration and the fit score on the quantitation report for the daily or continuing calibration. Data for undetected compounds are usable for many purposes such as risk assessment; therefore, rejection of data is not beneficial to a project. The conflict of improper characterization should be brought up and rectified with the laboratory.)

If there is some evidence of the compound's presence (as determined by visual matching of the base ion, parent ion and fingerprint pattern despite a poor fit score), the compound

should be considered tentatively identified. The reported value should not be altered in this case; however, a data qualifier code, "N", should be appended to the data point to denote tentative identification.

In both of the above cases, evaluation of additional information such as the retention time and frequency of detection in other field samples is warranted. Previous site history is also an important evaluation; however, information essential to perform such an evaluation (in contrast to 'validation') may not be easily available to a data validator. Nonetheless, it is incumbent upon a data validator to make appropriate recommendations to the project manager or the remedial officer.

3.2.5 Reporting

The most convenient form of reporting the mass spectral evaluation is through a brief memorandum to appropriate authorities. The highlighted Forms Is and the mass spectra in question should be appended to the memorandum in support of the conclusions. Also fill out and attach the Mass Spectral Evaluation Form, as depicted in Table M1-SVOA-3, to the report as a record of what was done. Record the date(s) of analyses. Field sample numbers should be transcribed from the chain-of-custody in the Sample Identifier column. Notations may be placed under the "MS" column for each sample to describe the mass spectral evaluation. The following notations are suggested:

- X—Acceptable mass spectrum.

- N—Tentative identification. Partial proof of a compound's presence, but all identification criteria not met.

- U—No mass spectral match. Using professional judgment, the compound is considered undetected.

- R—No mass spectral match. Using professional judgment, the compound data are rejected from further use.

3.3 Evaluation of Chromatograms

The purpose behind evaluating the chromatograms is to get an idea regarding potential false negatives, and gross analytical errors. Checking for positive data as described under mass spectral evaluation does not offer any insight into data that are not reported (i.e., reported as nondetects). Laboratory error or gross interference from other compounds could be the reasons for erroneous reporting. The gas chromatograms or the reconstructed ion current (RIC) profiles are the primary tool used for the false negative evaluation under Level M1.

Case No.:_____

SDG No.:_____

Data Reviewer:_____

Site:_____

Procedure No.: M1-SVOA

Revision: 1

Date: 06/30/1995

Table M1-SVOA-3
SVOA Mass Spectral Evaluation Summary

Dates Analyzed:	Sample Identifier:		Comments on Chromatograms
	1.	1.	
	2.	2.	
	3.	3.	
	4.	4.	
Inst. ID:	5.	5.	
	6.	6.	
	7.	7.	
	8.	8.	
	9.	9.	
	10.	10.	

Compound:	SPCC (#) CCC(*) Acidic (A) Base/Neutral (BN)	1 MS	1 RT	2 MS	2 RT	3 MS	3 RT	4 MS	4 RT	5 MS	5 RT	6 MS	6 RT	7 MS	7 RT	8 MS	8 RT	9 MS	9 RT	10 MS	10 RT
Phenol	(*)(A)																				
bis(2-Chloroethyl)ether	(BN)																				
2-Chlorophenol	(A)																				
1,3-Dichlorobenzene	(BN)																				
1,4-Dichlorobenzene	(*)(BN)																				
1,2-Dichlorobenzene	(BN)																				
2-Methylphenol	(A)																				
2,2'-oxybis(1-Chloropropane)	(BN)																				
4-Methylphenol	(A)																				
N-Nitroso-di-n-propylamine	(BN)(#)																				
Hexachloroethane	(BN)																				
Nitrobenzene	(BN)																				
Isophorone	(BN)																				
2-Nitrophenol	(A)(*)																				
2,4-Dimethylphenol	(A)																				
bis(2-Chloroethoxy)methane	(BN)																				
2,4-Dichlorophenol	(A)																				
1,2,4-Trichlorobenzene	(BN)																				
Naphthalene	(BN)																				
4-Chloroaniline	(BN)																				
Hexachlorobutadiene	(BN)(*)																				
4-Chloro-3-methylphenol	(A)(*)																				
2-Methylnaphthalene	(BN)																				
Hexachlorocyclopentadiene	(BN)(#)																				
2,4,6-Trichlorophenol	(A)(*)																				
2,4,5-Trichlorophenol	(A)																				
2-Chloronaphthalene	(BN)																				
2-Nitroaniline	(BN)																				
Dimethylphthalate	(BN)																				
Acenaphthylene	(BN)																				
2,6-Dinitrotoluene	(BN)																				
3-Nitroaniline	(BN)																				
Acenaphthene	(*)(BN)																				
2,4-Dinitrophenol	(A)(#)																				
4-Nitrophenol	(A)(#)																				

SPCC (f) CCC(*) Acidic (A) Compound: Base/Neutral (BN)	Sample Identifier																			
	1		2		3		4		5		6		7		8		9		10	
	MS	RT	MS	RT	MS	RT	MS	RT	MS	RT	MS	RT	MS	RT	MS	RT	MS	RT	MS	RT
Dibenzofuran (BN)																				
2,4-Dinitrotoluene (BN)																				
Diethylphthalate (BN)																				
4-Chlorophenyl-phenylether (BN)																				
Fluorene (BN)																				
4-Nitroaniline (BN)																				
4,6-Dinitro-2-methylphenol (A)																				
N-Nitrosodiphenylamine (BN)(*)																				
4-Bromophenyl-phenylether (BN)																				
Hexachlorobenzene (BN)																				
Pentachlorophenol (A)(*)																				
Phenanthrene (BN)																				
Anthracene (BN)																				
Carbazole (BN)																				
Di-n-Butylphthalate (BN)																				
Fluoranthene (BN)(*)																				
Pyrene (BN)																				
Butylbenzylphthalate (BN)																				
3,3'-Dichlorobenzidine (BN)																				
Benzo(a)anthracene (BN)																				
bis(2-ethylhexyl)phthalate (BN)																				
Chrysene (BN)																				
Di-n-Octylphthalate (BN)(*)																				
Benzo(b)fluoranthene (BN)																				
Benzo(k)fluoranthene (BN)																				
Benzo(a)pyrene (BN)(*)																				
Indeno(1,2,3-cd)pyrene (BN)																				
Dibenz(a,h)anthracene (BN)																				
Benzo(g,h,i)perylene (BN)																				

3.3.1 Acceptance Criteria

There are no EPA-established criteria for evaluating chromatograms for false negatives. The criteria used for evaluating chromatograms are based on good laboratory and scientific practices, and these are not hard and fast requirements. The suggested evaluation criteria are:

- There should not be any significant peaks in the chromatograms that are not accounted for as TCLs or TICs. Significant peaks are those with a minimum peak height of 10 percent of the closest internal standard.

- The chromatograms should ideally have base-line resolution between adjacent peaks. Also, there should not be broad (unresolved) envelops in the chromatograms.

- There should not be abrupt shifts in the baseline.

- There should not be peak tailing or sharp rise in the peak fronts.

3.3.2 Review Items

Chromatograms, or the RIC profiles for each sample are necessary for the evaluation. The RIC profiles can be found in the front of the raw data package for each sample. The quantitation report for each sample is also necessary to retrieve either the retention times or information regarding scan numbers to compare with the peaks on the chromatograms.

3.3.3 Evaluation of Chromatograms

Visually inspect the chromatograms for all peaks that appear to be at least 10 percent height of the nearest internal standard. Compare that the scan number or the retention time that appears on the x-axis of the RIC profile with that listed on the quantitation report for the TCLs or the library search record for the TICs. Make sure that all significant peaks are accounted for.

Also observe the RIC profile for peak resolution between adjacent peaks. Poor peak-to-peak resolution is indicative of degrading performance of the gas chromatographic column. The values obtained from a degrading system are prone to be inaccurate. Obviously certain isomeric or homologous compounds are difficult to separate. But generally, there should be at least 90 percent valley between the neighboring peaks.

Inspect the RIC profile for broad, unresolved envelops. These are generally indicative of outside interference from a series of homologous compounds such as straight-chain hydrocarbons. Especially, assess the interference with the internal standards and surrogates under the envelop using the expected area counts. The standard values that are far from the expected values may be indicative of potential problems with the TCL detection or quantification.

Inspect the RIC profile for abrupt shifts in the baseline. Such shifts are indicative of problems with instrument sensitivity or leakage in the system. The area counts obtained from shifted baseline are inaccurate, or even the detection of a TCL at low concentration may be missed.

Rapid peak rising or peak tailing indicate problems with the gas chromatographic column, such as depleted stationary phase on the column, decomposition of the stationary phase or creation of active sites. Again, a visual inspection of the RIC profile will yield information on the shape of the peak.

3.3.4 Action

Professional discretion must be used when evaluating and qualifying data based on the chromatographic evaluations. An experienced chemist can generally infer the magnitude and the frequency of the problem from the RIC profile. If the problem appears to be systematic, then it should be brought to the laboratories attention and resolved. Intermittent problems may or may not require any action. The following guidelines are suggested when acting on RIC profile observations:

- Any unaccounted TCL peak with area equivalent to or greater than the lowest reportable limit for the sample must be brought to the laboratory's attention and resolved. Any unaccounted non-TCL (i.e., TIC) peak with an area equal to or greater than 10 percent area of the nearest internal standard must also be resolved with the laboratory. TICs with less than 10 percent area are not required to be reported according to the CLP-RAS protocols. In the event, the discrepancy cannot be resolved with the laboratory, the problems should be documented and brought to the attention of the CLP-TPO, the RPM and the SM. The data for unreported TCL or TICs may be considered unusable until the problems are resolved.

- If a peak resolution problem is evident for the samples, and appears to be systematic (i.e., present in all calibration samples, QC samples, and field samples and increasing as the run progresses, additional QC measures such as the continuing calibration percent difference (%D), and internal and surrogate standard recoveries in the vicinity of the affected peaks should be

evaluated to determine if the peak resolution problem could affect detection or quantification. If determined so, the positive data may be qualified as estimated, "J." Negative data may also be qualified as estimated, "UJ" if the ability to detect at low concentrations is also deemed to be jeopardized by poor resolution of adjacent peaks.

- Broad envelops of homologous compounds could interfere with quantification or even detection. If the interference is evident from the recoveries of the internal and surrogate standards in the vicinity of the envelop, associated compounds may also be interfered with. Using professional discretion, the positive and negative data may be considered as estimated, "J" and "UJ", respectively. If the project objectives cannot be met with the estimated data, alternative sample preparation and cleanup procedures may need to be developed and specified.

- Discrete shifts in the baseline in the middle of a run are indicative of intermittent problems. If the shift is due to leakage or change in the system pressure, the positive as well as negative data may be considered estimated ("J" and "UJ, respectively). The problem could be also due to some fluctuation in the instrument electronics which may lead to drastic changes in the sensitivity of the instrument to detect the compounds. As a note, professional judgment should be exercised in determining the severity of the problem. For example, the magnitude of a drop in the baseline below the zero line may not be estimated and could be very significant. On the other hand, a drop that yields a baseline still above zero can be put in a perspective with the original baseline and a general appearance of the entire RIC profile.

- The problems with peak symmetry are indicative of system degradation, and should be brought to the attention of the laboratory for a corrective action. Professional judgment should be used when and if qualifying any data due to unsymmetrical peaks. First the problem should be defined in terms of persistence throughout the chromatogram and also from sample to sample. Additionally, the shapes and area counts for the internal and surrogate standards should be evaluated to see if the problem could have affected compound detection and/or quantification. Data qualification may be uncalled for if the standard area counts are acceptable.

3.3.5 Reporting

The Mass Spectral Evaluation Form (Table M1-SVOA-3) may be used to note any brief comments on the chromatographic evaluations. The comments may be noted against each sample identifier. For more descriptive comments, a separate sheet may be used.

3.4 Evaluation of Retention Times

While not unequivocal in identifying a compound, the retention times are quite helpful in confirming the presence of a detected compound. Matching of mass spectrum and retention time of sample with those of a standard yields higher credibility and confidence level to the detection in the sample. On the other hand, not matching the retention time may or may not invalidate the detection. If a mass spectral match is made beyond any doubt, poor matching of the retention time may not have adverse impact on the detection. If mass spectral matching is unacceptable or only partial, and the retention times do not match, then a strong doubt can be cast on a compound's presence.

3.4.1 Acceptance Criteria

The criteria for retention times are specified in the EPA's functional guidelines as follows:

> The relative retention times (RRTs) must be within +/-0.06 RRT units of the applicable internal standard RRT.

3.4.2 Review Items

Quantitation reports for the sample and continuing calibration are required for the evaluation. These are located in the raw data for samples and standards. A copy of the continuing calibration quantitation reports may be made or the reports pulled out from the raw data to facilitate a comparison with the sample quantitation reports.

3.4.3 Evaluation of Retention Times

For the detected compounds, determine the relative retention time for the compounds by dividing their retention times with the retention time of their associated internal standard in the samples as well as in the applicable continuing (or initial) calibrations. The sample RRTs must fall in range of standard RRT +/- 0.06 units.

3.4.4 Action

Action for retention time evaluation requires professional discretion. Action taken must be based on other data such as mass spectra and not on retention times alone. The following actions are suggested for several potential situations.

- Acceptable matching of the mass spectra and the RRTs--No action suggested.

- Acceptable matching of the mass spectra but poor matching of the RRTs--No action suggested, but the cause of retention time shift may be investigated. Check to see if similar discrepancies are observed in other parts of the chromatogram. Often high concentration of a compound will cause shifts in the retention times for other compounds in the nearby eluting region, but shifts in retention times in other areas may not be observed. Shifts all through the chromatogram may be indicative of an erratic system, such as flow rate fluctuations, poor temperature regulation, restriction or leakage in the system. There are likely chances that the sample values in such conditions may be inaccurate. Positive data may be qualified as estimated, "J", if deemed essential.

3.4.5 Reporting

The form presented earlier in Table M1-SVOA-3 may be used to record any problems in the RRT matching. For the detected compounds in each sample, the calculated RRTs may be recorded under the "RT" column. A data qualifier code may be added to the values exceeding 0.06 RRT and requiring qualification, such as "0.15J", for a compound with a difference of 0.15 RRT units and where a professional judgment to estimate the data is exercised.

3.5 Evaluation of Blanks

Laboratory blanks and field blanks have a profound impact on false positives reported in samples; i.e., compounds reported as positive detects but not originating from the samples themselves. Cross contamination from the sampling equipment, incidental contamination from the field conditions or contamination from the laboratory equipment or general environmental are likely sources of false positives in the samples.

3.5.1 Acceptance Criteria

Criteria for blank evaluation are specified in the EPA's Functional Guidelines. In addition, Region III has some additional requirements modifying the guidance. The acceptance criteria for blanks apply equally to any type of blanks associated with either sampling or analysis, such as trip blanks, rinsate blanks, field or bottle blanks, laboratory method blanks. While there are several criteria for evaluating the blanks, the only criterion applicable to Level M1 is the comparison of the blank and sample concentrations. This criterion is as follows:

> For common contaminants, such as the phthalate esters, the sample concentration must be minimally 10 times the blank concentration to be considered a positive detect. Other contaminants must be present in the

sample at or above 5 times the blank concentration before they can be considered significant detects. The blank with the highest concentration must be used (this is, if the laboratory blank has the highest concentration of naphthalene, and the equipment rinsate blank has the highest concentration of 2-nitroaniline, both of these blanks must be used to qualify the respective contaminants).

3.5.2 Review Items

Data requirements and data retrieval procedures for blanks are the same as those for the field samples because the blanks as well as field samples are validated similarly. Form I's, mass spectra, chromatograms, quantitation reports, etc., are essential for performing a validation of the blanks first.

3.5.3 Blank Evaluation Procedure

Validate the blanks same as the field samples. Detailed validation procedures are described above under appropriate sections. Use the validated blank results for a comparison with the sample results. Make certain that the samples and blanks are evaluated on the same basis of sample weight or volume, dilution factors, moisture content, etc. Use the 5 (or 10) times the highest blank concentrations for qualifying the sample data.

3.5.4 Action

If the sample concentrations do not meet the criteria of 5 (or 10) times the blank concentration, the sample results should be considered essentially undetected (or as not detected substantially above the levels reported in the blanks); therefore, flagged "B" in accordance with the Region III data validation guidelines.

3.5.5 Reporting

Form I's may be used to write the "B" data qualifier for the data not meeting the blank criteria.

3.6 Sample Paperwork

The purpose for evaluating the sample paperwork is to determine that the samples being validated are indeed the ones taken from the site, and have not been tampered with. Accurate sample identity is of paramount importance in substantiating the sample data. Without unequivocal sample identity and chain-of-custody procedures, the sample data may not be defensible or enforceable.

Under the current CLP contracts, the original paperwork (i.e., the purge package or the administrative record) is included in the data package from the laboratory. It is assumed that the data validator is not privy to the original paperwork; therefore, the evaluation criteria and procedures described below apply only to the documents that are generally included in the data validation package. These documents are the chain-of-custody forms and Region III Shipping Record.

3.6.1 Acceptance Criteria

Criteria for acceptability or authenticity of the sampling paperwork, document control and chain-of-custody have been established by the National Enforcement Investigations Center (NEIC), in support of the CLP. Overall criteria are too numerous and subjective to be discussed here, but the criteria that apply to data validation are:

- The chain-of-custody form should be properly and completely filled out including the sampler signatures, date and time of sampling, sampling station identification, analyses requested, traffic numbers, tag numbers, etc. These data are minimally required to confirm the authenticity of the sample and its data.

- The chain-of-custody must be maintained at all times. The custody transfers between different parties must be signed and dated.

3.6.2 Review Items

A copy of the chain-of-custody form originated in the field and that returned from the laboratory with the data are essential to confirm the identity of the samples. In addition, the Region III Shipping Record is essential to identify the field QC samples. The chain-of-custody and Shipping Record are generally located in front of the data package.

3.6.3 Evaluation Procedure

Ensure that the chain-of-custody form was signed and dated by the samplers, and a time and date were entered for sample collection. The laboratory copy of the chain-of-custody must have the signature of the laboratory sample custodian. Any errors on the form should have been crossed out with a single line through the entry. Verify that all collected samples have unique station identification, traffic numbers and sample tag numbers. Ensure that the Region III Shipping Record correctly reflects the information on the chain-of-custody.

3.6.4 Action

The action to be taken in qualifying the data is highly dependent on the nature of the problem. Some errors in paperwork are practically unavoidable in real situations. An effort should be made to reconcile the differences by cross checking the field notebooks against the sampling paperwork. Occasionally, the samplers may forget to sign the chain-of-custody; however, the field notebooks may amply describe the sampling event. Problems are also inevitable in noting or cross-referencing sample tag numbers and traffic numbers. Generally, there are several alternate sources of information to substantiate or refute the problem.

3.6.5 Reporting

Any discrepancies found in the paperwork must be immediately brought to the attention of the EPA RPM. Clearly define the problems in a memorandum to the responsible parties. Attach marked copies of the chain-of-custody forms to substantiate the findings.

Appendix B-2
Validation of Semivolatile Organic Analyte Data
Manual Level M2

1. Purpose and Applicability

This procedure provides step-by-step instructions to manually validate the semivolatile organic analyte (SVOA) data using the manual innovative data validation approach at Level M2. This approach focuses on the use of information contained on the CLP forms and a review of chromatograms as summarized in Table M2-SVOA-1. The procedures are based on modifications to Regions III's National Functional Guidelines for Organic Data Review.

The procedure is applicable to the SVOA data obtained using the Contract Laboratory Program Statement of Work (CLP SOW). Hard copy data conforming to the 1990 CLP SOW specifications are essential in order to carry out the procedure.

Data validated using this procedure are considered usable for the following types of purposes; however, the data users must decide on a case-by-case basis whether the procedure is suitable for their intended data uses. The suggested data uses are:

- Oversight of activities led by other parties

- Comparison to action levels

- Initial site investigation

- Contamination sources

- Nature and extent of contamination

- Preliminary risk assessment

- Risk assessment with known high levels of toxics

- Feasibility study

- Preliminary design

- Treatability study

- Initial cleanup verification

Table M2-SVOA-1 QC CHECKLIST FOR LEVEL M2 CLP RAS SEMIVOLATILE ORGANICS			
QC MEASURES	M1	Manual M2	M3
Action Level Notification	X	X	X
Instrument Tune		X	X
Initial Calibration (RRF)		X	X
Initial Calibration (%RSD)		X	X
Continuing Calibration (RRF)		X	X
Continuing Calibration (%D)		X	X
Laboratory Blank	X	X	X
MS/MSD (%R, RPD)		X	X
Internal Standard Area		X	X
Field Blank	X	X	X
Sample Paperwork	X	X	X
Holding Time		X	X
Retention Time	X	X	X
Surrogate Recovery		X	X
Dilution Factor		X	X
Moisture Content		X	X
Mass Spectra	X		X
Chromatograms	X	X	X
Raw Data			X

2. Quality Control Measures Checked

Table M2-SVOA-1 highlights the quality control (QC) indicators evaluated under this data validation procedure.

3. Procedure

The following subsections describe for each of the QC indicators the acceptance criteria, location and retrieval of QC data, evaluation of the QC data, actions taken in the event the QC acceptance criteria are exceeded, and documentation of the QC violations in a standardized report form.

The semivolatile data requirements to be checked are listed below:

3.1 Action Level Notification

3.2 Technical Holding Times (CCS - Contractual holding times only)

3.3 GC/MS Instrument Performance Check (CCS)

3.4 Initial Calibration (CCS)

3.5 Continuing Calibration (CCS)

3.6 Blanks (CCS)

3.7 Surrogate Spikes (CCS)

3.8 Matrix Spikes/Matrix Spike Duplicates

3.9 Internal Standards (CCS)

3.10 Reported Contract Required Quantitation Limits (CRQLs)

3.11 Tentatively Identified Compounds

Two forms have been developed to assist in the performance and documentation of implementing Level M2. The first form, M2-SVOA-QUAL, summarizes holding time, calibrations, blanks, surrogates, and internal standards. The second form, M2-SVOA-SPK, summarizes surrogate and matrix spike quality control checks. These forms appear on the following pages as Table M2-SVOA-2 and Table M2-SVOA-3.

Reporting requirements for Level M2 are as follows:

- Hand annotate the Form I's, including
    - Data validation qualifiers
    - Sample identification number
    - Sampling location

- Provide a narrative that includes
    - A statement that defines the level of the data review, i.e., M2
    - Major and minor problems associated with the analysis
    - Highlight issues that may have affected detection limits

- Include the following attachments
    - List of data validation qualifiers
    - Support documentation including forms that support assigning data qualifiers
    - Chain of custody form
    - Samples affected by calibration should be listed on the appropriate calibration forms

The data qualifiers assigned in this review are as follows:

Codes Relating To Identification (Confidence concerning presence or absence of compounds)

U = Not detected. The associated number indicates approximate sample concentration necessary to be detected.

(NO CODE) = Confirmed identification.

B = Not detected substantially above the level reported in laboratory or field blanks.

R = Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.

N = Tentative identification. Consider present. Special methods may be needed to confirm its presence or absence in future sampling efforts.

Codes Related To Quantitation (can be used for both positive results and sample quantitation limits):

J = Analyte present. Reported value may not be accurate or precise.

Case No.:_____

SDG No.:_____

Data Reviewer:_____ 6

Site:_____

Procedure No.: M2-SVOA

Revision: 1

Date: 06/30/1995

Table M2-SVOA-2. SVOA Qualifier Summary (Calibrations, Blanks, Holding Time, Surrogates, Internal Standards)

		Hold Time Out, days		Standards: (<,>)															
				Surrogate								Internal (IS)					
Analysis Date(s):	Sample Identifier:	Ex	An	1	2	3	4	5	6	7	8	1	2	3	4	5	6
Analyzed Within 12 Hours of Tune? __Yes__No	1.																
Inst. ID:	2.																
	3.																
Tune OK? __Yes__No	4.																
MBlnk ID:	5.																
	6.																
Ext. Dates:	7.																
	8.																
	9.																
	10.																

Date:_____ Time:_____

Compound:	SPCC (#) CCC(*) Acidic (A) Base/Neutral (BN)	Initial Cal.		Continuing Cal.		Blanks		Qualifiers	
		RRF<.05	%RSD>30	RRF<.05	%D>25	Lab	Field	(+/-)	
Phenol	(*)(A)								
bis(2-Chloroethyl)ether	(BN)								
2-Chlorophenol	(A)								
1,3-Dichlorobenzene	(BN)								I
1,4-Dichlorobenzene	(*)(BN)								S
1,2-Dichlorobenzene	(BN)								#
2-Methylphenol	(A)								1
2,2'-oxybis(1-Chloropropane)	(BN)								
4-Methylphenol	(A)								
N-Nitroso-di-n-propylamine	(BN)(#)								
Hexachloroethane	(BN)								
Nitrobenzene	(BN)								
Isophorone	(BN)								
2-Nitrophenol	(A)(*)								
2,4-Dimethylphenol	(A)								I
bis(2-Chloroethoxy)methane	(BN)								S
2,4-Dichlorophenol	(A)								#
1,2,4-Trichlorobenzene	(BN)								2
Naphthalene	(BN)								
4-Chloroaniline	(BN)								
Hexachlorobutadiene	(BN)(*)								
4-Chloro-3-methylphenol	(A)(*)								
2-Methylnaphthalene	(BN)								
Hexachlorocyclopentadiene	(BN)(#)								
2,4,6-Trichlorophenol	(A)(*)								
2,4,5-Trichlorophenol	(A)								
2-Chloronaphthalene	(BN)								
2-Nitroaniline	(BN)								I
Dimethylphthalate	(BN)								S
Acenaphthylene	(BN)								#
2,6-Dinitrotoluene	(BN)								3
3-Nitroaniline	(BN)								
Acenaphthene	(*)(BN)								
2,4-Dinitrophenol	(A)(#)								
4-Nitrophenol	(A)(#)								

Case No.:_____

SDG No.:_____

Data Reviewer:_____

Site:_____

Procedure No.: M2-SVOA

Revision: 1

Date: 06/30/1995

Table M2-SVOA-2
(contd.)

Analysis Date:

Instrument ID:

Date: _____   Time: _____   _____

Compound: SPCC (#) CCC(*) Acidic (A) Base/Neutral (BN)	Initial Cal.		Continuing Cal.		Blanks		Qualifiers
	RRF<.05	%RSD>30	RRF<.05	%D>25	Lab	Field	(+/-)
Dibenzofuran (BN)							
2,4-Dinitrotoluene (BN)							
Diethylphthalate (BN)							
4-Chlorophenyl-phenylether (BN)							
Fluorene (BN)							I
4-Nitroaniline (BN)							S
4,6-Dinitro-2-methylphenol (A)							#
N-Nitrosodiphenylamine (BN)(*)							4
4-Bromophenyl-phenylether (BN)							
Hexachlorobenzene (BN)							
Pentachlorophenol (A)(*)							
Phenanthrene (BN)							
Anthracene (BN)							
Carbazole (BN)							
Di-n-Butylphthalate (BN)							
Fluoranthene (BN)(*)							
Pyrene (BN)							
Butylbenzylphthalate (BN)							I
3,3'-Dichlorobenzidene (BN)							S
Benzo(a)anthracene (BN)							#
bis(2-ethylhexyl)phthalate (BN)							5
Chrysene (BN)							
Di-n-Octylphthalate (BN)(*)							
Benzo(b)fluoranthene (BN)							I
Benzo(k)fluoranthene (BN)							S
Benzo(a)pyrene (BN)(*)							#
Indeno(1,2,3-cd)pyrene (BN)							6
Dibenz(a,h)anthracene (BN)							
Benzo(g,h,i)perylene (BN)							

Reported as: RT   (µg/kg, µg/L)

TICs Reported In Blank(s):

Case No.:_____

SDG No.:_____

Data Reviewer:_____

Site:_____

Procedure No.: M2-SVOA

Revision: 1

Date: 06/30/1995

Table M2-SVOA-3. SVOA Soil Surrogate and Matrix Spike Quality Control Summary

Surrogate Data Summary

Sample Identifier:	Soil Sample Recoveries, %R								Qualifiers (+/-)
	S1	S2	S3	S4	S5	S6	S7	S8	
(Acceptance Range, %R):	23-120	30-115	18-137	24-113	25-121	19-122	20-130	20-130	

S1 = Nitrobenzene-d5, S2 = 2-fluorobiphenyl, S3 = terphenyl-d14, S4 = phenol-d5, S5 = 2-fluorophenol, S6 = 2,4,6-tribromophenol, S7 = 2-chlorophenol-d4, S8 = 1,2-dichlorobenzene-d4.

MS/MSD Data Summary

Spike Compound: SPCC (#) CCC (*) Aromatic (AR)	Matrix Spike, Recovery, %R		Matrix Spike Duplicate, Recovery, %R		MS/MSD Precision, RPD		Qualifiers (+/-)
	Range	Actual	Range	Actual	Range	Actual	
SOIL SAMPLES							
Phenol	26-90		26-90		35		
2-Chlorophenol	25-102		25-102		50		
1,4-Dichlorobenzene	28-104		28-104		27		
N-Nitroso-di-n-propylamine	41-126		41-126		38		
1,2,4-Trichlorobenzene	38-107		38-107		23		
4-Chloro-3-methylphenol	26-103		26-103		33		
Acenaphthene	31-137		31-137		19		
4-Nitrophenol	11-114		11-114		50		
2,4-Dinitrotoluene	28-89		28-89		47		
Pentachlorophenol	17-109		17-109		47		
Pyrene	35-142		35-142		36		

Case No.:_____
SDG No.:_____
Data Reviewer:_____
Site:_____

Procedure No.: M2-SVOA
Revision: 1
Date: 06/30/1995

Table M2-SVOA-3. SVOA Aqueous Surrogate and Matrix Spike Quality Control Summary

Surrogate Data Summary

Sample Identifier:	Aqueous Sample Recoveries, %R	
	S1	S2	S3	S4	S5	S6	S7	S8	Qualifiers (+/-)
(Acceptance Range, %R):	35-114	43-116	33-141	10-110	21-110	10-123	33-110	16-110	
1.									
2.									
3.									
4.									
5.									
6.									
7.									
8.									
9.									
10.									

S1 = Nitrobenzene-d5, S2 = 2-fluorobiphenyl, S3 = terphenyl-d14, S4 = phenol-d5, S5 = 2-fluorophenol, S6 = 2,4,6-tribromophenol, S7 = 2-chlorophenol-d4, S8 = 1,2-dichlorobenzene-d4. NOTE: Surrogates S7 and S8 have advisory limits only.

MS/MSD Data Summary

Spike Compound: Aromatic (AR)	SPCC (#) CCC (*)	Matrix Spike, Recovery, %R		Matrix Spike Duplicate, Recovery, %R		MS/MSD Precision, RPD		Qualifiers (+/-)
		Range	Actual	Range	Actual	Range	Actual	
AQUEOUS SAMPLES								
Phenol		12-110		12-110		42		
2-Chlorophenol		27-123		27-123		40		
1,4-Dichlorobenzene		36-97		36-97		28		
N-Nitroso-di-n-propylamine		41-116		41-116		38		
1,2,4-Trichlorobenzene		39-98		39-98		28		
4-Chloro-3-methylphenol		23-97		23-97		42		
Acenaphthene		46-118		46-118		31		
4-Nitrophenol		10-80		10-80		50		
2,4-Dinitrotoluene		24-96		24-96		38		
Pentachlorophenol		9-103		9-103		50		
Pyrene		26-127		26-127		31		

K   = Analyte present. · Reported value may be biased high. Actual value is
         expected lower.

L . = Analyte present. Reported·value may be biased low. Actual value is
          expected to be higher.

UJ   = Not detected, quantitation limit may be inaccurate or imprecise.

UL   = Not detected, quantitation limit is probably higher.

Other Codes

Q · = No analytical result. ·

*   = Results reported from diluted analysis.

3.1 Action Level Notification

The purpose behind action level notification is to make the EPA Remedial Project Officer
(RPM) or the Site Project Officer (SPO) aware of the potential human health risk at the
site. In accordance with the Region III Hazardous waste division policy, the EPA RPM or
SPO must be promptly notified of any contaminant exceeding the established action level or
the 10-day health advisory limit. The data for contaminants exceeding the action levels
must be validated as a top priority and reported to the RPM or SPO. Validation of the rest
of the data may then be completed normally.

3.1.1 Acceptance Criteria

EPA's Office of Solid Waste and Emergency Response has established 10-day advisory
limits or the action levels for several organic compounds and elements of special health risk
concerns based on the Safe Drinking Water Act. The semivolatile organic compounds and
their 10-day health advisory limits apply only to aqueous samples and are listed in Table
M2-SVOA-4. The criteria for action level notification are as follows:

- The contaminant concentration must be equal to or above the established 10-
  day health advisory limits.

- Data for contaminants exceeding the action levels must be validated as a top
  priority.

- The following EPA personnel must be notified of the action level
  exceedances:

- EPA RPM or SPO

- EPA Section Chiefs:

  - Site Investigation (SI)

  - Remedial

  - Enforcement

  - RCRA

- EPA Section Toxicologists:

  - Enforcement

  - Superfund

  - RCRA

- The remaining data validation should be completed per normal procedures.

- Any special instructions from the Hazardous Waste Division should be followed.

- Records should be kept of the data review, action level notification and any follow up instructions and actions.

Table M2-SVOA-4 SEMIVOLATILE ORGANIC ANALYTES AND ACTION LEVELS			
Compound	Action Level*	Compound	Action Level*
1,3-Dichlorobenzene	8,930	1,4-Dichlorobenzene	10,700
Pentachlorophenol	300	---	---
*All units are ug/l.			

3.1.2 Review Items

All data required to perform Level M2 validation, as detailed in the following sections, are necessary for carrying out action level notification. The location of the data and their retrieval procedures are also discussed below.

3.1.3 Evaluation Procedure

The evaluation process preceding action level notification will primarily consist of comparing the results on Form I's with the action levels presented in Table M2-SVOA-4. Following the identification of the contaminants exceeding the action levels, focused data validation should be performed using the criteria and procedures described in the appropriate sections below.

3.1.4 Action

The action resulting from focused data validation will be the notification of action level exceedance to the personnel identified above in Section 3.1.1.

3.1.5 Reporting

Copies of Form I's can be used to highlight the contaminants above the action levels. The findings of the focused validation can be summarized in a memorandum, and the data qualifiers resulting from focused validation may be written on the Form I's. The marked up forms should be clarified that they represent validation of only the contaminants exceeding the action levels, and not all data.

3.2 Technical Holding Times

The objective is to ascertain the validity of results based on the holding time of the sample from <u>time of collection</u> to time of sample extraction and analysis.

3.2.1 Acceptance Criteria

Technical requirements for sample holding times have only been established for water matrices. The holding times for soils (and other non-aqueous matrices such as sediments, oily wastes, and sludge) are currently under investigation. When the results are available they will be incorporated into the data evaluation process. Additionally, results of holding time studies will be incorporated into the data review criteria as the studies are conducted and approved.

The holding time criteria for water samples, as stated in the current 40 CFR Part 136 (Clean Water Act) is as follows:

> For semivolatile compounds in cooled (@ 4°C) water samples the maximum holding time is 7 days from sample collection to extraction and 40 days from sample extraction to analysis.

It is further required that semivolatile compounds in properly preserved non-aqueous samples be extracted within 7 days from sample collection and the extracts analyzed within 40 days from sample extraction.

3.2.2 Review Items

Form I SV-1 and SV-2, EPA Sample Shipping Log and/or chain-of-custody.

3.2.3 Evaluation Procedures

Technical holding times for sample extraction are established by comparing the sampling date on the EPA Sample Traffic Report with the dates of extraction on Form I SV-1 and SV-2. To determine if the samples were analyzed within the holding time after extraction, compare the dates of extraction on the sample extraction sheets with the dates of analysis on Form I SV-1 and SV-2.

Verify that the C.O.C. indicates that the samples were received intact and iced. If the samples were not iced or there were any problems with the samples upon receipt, then discrepancies in the sample condition could affect the data.

3.2.4 Action

1. If technical holding times are exceeded, flag all positive results as estimated "J" and sample quantitation limits as estimated "UJ" and document that holding times were exceeded. However, please note that some extractable compounds are extremely persistent in the environment (e.g., PAHs) in non-aqueous matrices and would not be expected to degrade significantly during sample storage. The reviewer must use professional judgment in the application of data qualifiers to those compounds in non-aqueous matrices.

2. If in the professional judgment of the data reviewer a loss of semivolatile compound(s) is evident due to exceeding the holding time criteria, the affected positive results or the associated quantitation limits may be qualified as biased low, "L" or "UL" respectively. The narrative must contain the reviewer's justification for qualification of the compound results as biased low.

3.   If technical holding times are grossly exceeded (greater than 2 times the required technical holding time), either on the first analysis or upon re-analysis, the reviewer must use professional judgment to determine the reliability of the data and the effects of additional storage on the sample results. The reviewer may determine that positive results or the associated quantitation limits are approximates and should be qualified with "J" or "UJ", respectively. The reviewer may determine that non-detect data are unusable (R).

4.   Because of limited information concerning holding times for non-aqueous samples, it is recommended that a comment in the data review narrative be included to state that aqueous holding times were applied.

5.   Whenever possible, the reviewer should comment on the effect of exceeding the holding time on the resulting data in the data review narrative.

6.   When contractual and/or technical holding times are exceeded, this should be noted on the ORDAS form.

7.   The reviewer should also be aware of the scenario in which the laboratory has exceeded the technical holding times, but met contractual holding times. In this case, the data reviewer should notify the Regional TPO (where samples were collected) and/or RSCC that shipment delays may have occurred so that the field problem can be corrected. The reviewer may pass this information on to the Regional TPO on the ORDAs, but should explain that contractually the laboratory met the requirements.

8.   When there are other quality control problems in conjunction with exceeded holding times (such as suspected laboratory contamination), the reviewer should follow the hierarchy of qualifiers. In particular, if for any reason the reviewer doubts the presence of a compound, the data summary should display only the "B" or "R" qualifier, and not the "L" qualifier. This is because no net direction of bias can be inferred under these conditions.

3.3 GC/MS Instrument Performance Check

Gas chromatograph/mass spectrometer (GC/MS) instrument performance checks (formerly referred to as tuning) are performed to ensure mass resolution, identification and, to some degree, sensitivity. These criteria are not sample specific. Conformance is determined using standard materials, therefore, these criteria should be met in all circumstances.

3.3.1 Acceptance Criteria

The analysis of the instrument performance check solution must be performed at the beginning of each 12-hour period during which samples or standards are analyzed. The instrument performance check, decafluorotriphenylphosphine (DFTPP) for semivolatile analysis, must meet the ion abundance criteria given below.

Decafluorotriphenylphosphine (DFTPP)

m/z	ION ABUNDANCE CRITERIA
51	30.0 - 80.0% of m/z 198
68	Less than 2.0% of m/z 69
69	Present
70	Less than 2.0% of m/z 69
127	25.0 - 75.0% of m/z 198
197	Less than 1.0% of m/z 198
198	Base peak, 100% relative abundance
199	5.0 - 9.0% of m/z 198
275	10.0 - 30.0% of m/z 198
365	Greater than 0.75% of m/z 198
441	Present, but less than m/z 443
442	40.0 - 110.0% of m/z 198
443	15.0 - 24.0% of m/z 442

NOTE: All ion abundances must be normalized to m/z 198, the nominal base peak, even though the ion abundances of m/z 442 may be up to 110 percent that of m/z 198.

3.3.2 Review Items

Form V SV.

3.3.3 Evaluation Procedures

1.   Compare the data presented on each GC/MS Instrument Performance Check (Form V SV) with each mass listing submitted and ensure the following:

    a.   Form V SV is present and completed for each 12-hour period during which samples were analyzed.

b.   The appropriate number of significant figures has been reported (number of significant figures given for each ion in the ion abundance criteria column) and that rounding is correct.

c.   The laboratory has not made any calculation errors.

2.   Verify that the ion abundance criteria were met. The criteria for m/z 68, 70, 441, and 443 are calculated by normalizing to the specified m/z.

3.   If possible, verify that spectra were generated using appropriate background subtraction techniques. Since the DFTPP spectrum is obtained from chromatographic peaks that should be free from coelution problems, background subtraction should be done in accordance with the following procedure. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged and background subtraction must be accomplished using a single scan prior to the elution of DFTPP.

NOTE: All instrument conditions must be identical to those used in the sample analysis. Background subtraction actions resulting in spectral distortions for the sole purpose of meeting the contract specifications are contrary to the quality assurance objectives and are therefore unacceptable.

3.3.4 Action

1.   If the laboratory has made minor transcription errors which do not significantly affect the data, the data reviewer should make the necessary corrections on a copy of the form.

2.   If the laboratory has failed to provide the correct forms or has made significant transcription or calculation errors, the Region's designated representative should contact the laboratory and request corrected data. If the information is not available, then the reviewer must use professional judgment to assess the data. The Regional TPO should be notified by noting the problem(s) on the ORDAS.

3.   If mass assignment is in error (such as m/z 199 is indicated as the base peak rather than m/z 198), classify all associated data as unusable, (R).

4.   If ion abundance criteria are not met, professional judgment may be applied to determine to what extent the data may be utilized. Guidelines to aid in the application of professional judgment in evaluating ion abundance criteria are discussed as follows:

a.   Some of the most critical factors in the DFTPP criteria are the non-instrument specific requirements that are also not unduly affected by the location of the spectrum on the chromatographic profile. The m/z ratios for 198/199 and 442/443 are critical. These ratios are based on the natural abundances of carbon 12 and carbon 13 and should always be met. Similarly, the relative abundances for m/z 68, 70, 197, and 441 indicate the condition of the instrument and the suitability of the resolution adjustment and are very important. Note that all of the foregoing abundances relate to adjacent ions; they are relatively insensitive to differences in instrument design and position of the spectrum on the chromatographic profile.

b.   For the ions at m/z 51, 127, and 275, the actual relative abundance is not as critical. For instance, if m/z 275 has 40% relative abundance (criteria: 10.0-30.0%) and other criteria are met, then the deficiency is minor.

c.   The relative abundance of m/z 365 is an indicator of suitable instrument zero adjustment. If relative abundance for m/z 365 is zero, minimum detection limits may be affected. On the other hand, if m/z 365 is present, but less than the 0.75% minimum abundance criteria, the deficiency is not as serious.

5.   Decisions to use analytical data associated with DFTPP instrument performance checks not meeting contract requirements should be clearly noted in the data review narrative.

6.   If the reviewer has reason to believe that instrument performance check criteria were achieved using techniques other than those specified in the SOW and in subparagraph a. above, additional information on the DFTPP instrument performance checks should be obtained. If the techniques employed are found to be at variance with contract requirements, the procedures of the laboratory may merit evaluation. Concerns or questions regarding laboratory performance should be noted for TPO action on the ORDAS. For example, if the reviewer has reason to believe that an inappropriate technique was used to obtain background subtraction (such as background subtracting from the solvent front or from another region of the chromatogram rather than the DFTPP peak), then this should be noted for TPO action on the ORDAS.

3.4 Initial Calibration

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for compounds on the semivolatile Target Compound List (TCL). Initial calibration

demonstrates that the instrument is capable of acceptable performance in the beginning of the analytical run and of producing a linear calibration curve.

3.4.1 Acceptance Criteria

1. Initial calibration standards containing both semivolatile target compounds and surrogates are analyzed at concentrations of 20, 50, 80, 120, and 160 ug/L at the beginning of each analytical sequence or as necessary if the continuing calibration acceptance criteria are not met. The initial calibration (and any associated samples and blanks) must be analyzed within 12 hours of the associated instrument performance check.

2. Minimum Relative Response Factor (RRF) criteria must be greater than or equal to 0.05. Contractual RRF criteria are listed in Appendix A.

3. The Percent Relative Standard Deviations (%RSD) for the RRFs in the initial calibration must be less than or equal to 30%.

3.4.2 Review Items

Form VI SV-1 and SV-2 and chromatograms.

3.4.3 Evaluation Procedures

1. Verify that the correct concentration of standards were used for the initial calibration (i.e., 20, 50, 80, 120, and 160 ug/L). For the eight compounds with higher CRQLs, only a four-point initial calibration is required (i.e., 50, 80, 120, and 160 ug/L).

2. If any sample results were calculated using an initial calibration, verify that the correct standard (i.e., the 50 ppb standard) was used for calculating sample results and that the samples were analyzed within 12 hours of the associated instrument performance check.

3. Evaluate the RRFs for all semivolatile target compounds and surrogates:

   Verify that all semivolatile target compounds and surrogates have RRFs that are greater than or equal to 0.05. If problems are suspected with low response factor or compound identification, also check elution order.

   NOTE: Because historical performance data indicate poor response and/or erratic behavior, the semivolatile compounds listed above have no

contractual maximum %RSD criteria. Contractually they must meet a minimum RRF criteria of 0.01; however, **for data review purposes, the "greater than or equal to 0.05" criterion is applied to all semivolatile compounds.**

Semivolatile Target Compounds Exhibiting Poor Response

- 2,2'-oxybis(1-Chloropropane)
- 4-Chloroaniline
- Hexachlorobutadiene
- Hexachlorocyclopentadiene
- 2-Nitroaniline
- Dimethylphthalate
- 3-Nitroaniline
- 2,4-Dinitrophenol
- 4-Nitrophenol
- Carbazole
- Diethylphthalate
- 4-Nitroaniline
- 4,6-Dinitro-2-methylphenol
- N-Nitrosodiphenylamine
- Di-n-butylphthalate
- Butylbenzylphthalate
- 3-3'-Dichlorobenzidine
- bis(2-Ethylhexyl)phthalate
- Di-n-octylphthalate

4.   Evaluate the %RSD for all semivolatile target compounds and surrogates.

a.   Verify that all semivolatile target compounds have a %RSD of less than or equal to 30%. The contractual criteria for an acceptable initial calibration specifies that up to any 4 semivolatile target compounds may fail to meet minimum RRF or maximum %RSD as long as they have RRFs that are greater than or equal to 0.010, and %RSD of less than or equal to 40.0%. For data review purposes, however, all compounds must be considered for qualification when the %RSD exceeds the ± 30.0% criterion.

b.   If the %RSD is greater than 30.0%, then the reviewer should use professional judgment to determine the need to check the points on the curve for the cause of the non-linearity. This is checked by eliminating either the high point or the low point and recalculating the %RSD.

3.4.4 Action

1. All semivolatile target compounds, including the 19 "poor performers," listed above, will be qualified using the following criteria:

   a. If the %RSD is greater than 30.0% and the RRF is greater than or equal to 0.05, qualify positive results with "J", and non-detected semivolatile target compounds using professional judgment.

   b. If the RRF is less than 0.05, qualify positive results that have acceptable mass spectral identification with "J" using professional judgment, and non-detects as unusable (R).

2. At the reviewer's discretion, a more in-depth review to minimize the qualification of data can be accomplished by considering the following:

   a. If any of the required semivolatile compounds have a %RSD greater than 30.0%, and if eliminating either the high or the low point of the curve does not restore the %RSD to less than or equal to 30.0%:

      i. Qualify positive results for that compound(s) with "J".

      ii. Qualify non-detected semivolatile target compounds based on professional judgment.

   b. If the high point of the curve is outside of the linearity criteria (e.g. due to saturation):

      i. No qualifiers are required for positive results in the linear portion of the curve.

      ii. Qualify positive results outside of the linear portion of the curve with "J".

      iii. No qualifiers are needed for non-detected target compounds.

   c. If the low end of the curve is outside of the linearity criteria:

      i. No qualifiers are required for positive results in the linear portion of the curve.

      ii. Qualify low level positive results in the area of non-linearity with "J".

      iii.   Qualify non-detected semivolatile target compounds using professional judgment.

3. If the laboratory has failed to provide adequate calibration information, the designated representative should contact the laboratory and request the necessary information. If the information is not available, the reviewer must use professional judgment to assess the data.

4. Whenever possible, the potential effects on the data resulting from a failure to meet calibration criteria should be noted in the data review narrative.

5. If calibration criteria are grossly exceeded, this should be noted for TPO action on the ORDAS.

6. When it is suspected that relative response factors were incorrectly generated from misidentified peaks or incorrect area measurements, the laboratory should be contacted to requantitate these RRFs and associated sample results. The ORDAS should identify affected results and document the cause of the reviewer's suspicions. In addition, a CLP telephone log must be completed.

7. Positive results for compounds flagged for blank contamination (B) will not need a separate flag (J) in the data summary form for minimum RRF, %RSD, or %D outside criteria. However, these situations should be addressed in the data review narrative and issues pertaining to noncompliance should be documented on the ORDAS.

3.5 Continuing Calibration

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for semivolatile target compounds. Continuing calibration establishes the 12-hour relative response factors on which the quantitations are based and checks satisfactory performance of the instrument on a day-to-day basis.

3.5.1 Acceptance Criteria

1. Continuing calibration standards containing both target compounds and surrogates are analyzed at the beginning of each 12-hour analysis period following the analysis of the instrument performance check and prior to the analysis of blanks and samples.

2. The minimum Relative Response Factors (RRF) for semivolatile target compounds and surrogates must be greater than or equal to 0.05.

3. The percent difference (%D) between the initial calibration RRF and the continuing calibration RRF must be within ± 25.0% for all target compounds.

3.5.2 Review Items

Form VII SV-1 and SV-2 and chromatograms.

3.5.3 Evaluation Procedures

1. Verify that the continuing calibration was run at the required frequency and that the continuing calibration was compared to the correct initial calibration.

2. Evaluate the continuing calibration RRF for all semivolatile target compounds and surrogates.

> Verify that all semivolatile target compounds and surrogates have RRFs within specifications.

> NOTE: Because historical performance data indicate poor response and/or erratic behavior, the compounds listed in Section 3.4.3 have no contractual maximum %D criteria. Contractually they must meet a minimum RRF criterion of 0.01; however, for data review purposes, the "greater than or equal to 0.05" criterion is applied to all semivolatile compounds.

3. Evaluate the %D between initial calibration RRF and continuing calibration RRF for one or more semivolatile compounds.

> Verify that the %D is within the ± 25.0% criterion, for all semivolatile target compounds and surrogates. Note those compounds which have a %D outside the ± 25.0% criterion. The contractual criteria for an acceptable continuing calibration specifies that up to any 4 semivolatile target compounds may fail to meet minimum RRF or maximum %D as long as they have RRFs that are greater than or equal to 0.010, and %D of less than or equal to 40.0%. For data review purposes, however, all compounds must be considered for qualification when the %D exceeds the ± 25.0% criterion.

3.5.4 Action

1. The reviewer should use professional judgment to determine if it is necessary to qualify the data for any semivolatile target compound. If qualification of data is required, it should be performed using the following guidelines:

a. If the %D is outside the ± 25.0% criterion and the continuing calibration RRF is greater than or equal to 0.05, qualify positive results "J".

b. If the %D is outside the ± 25.0% criterion and the continuing calibration RRF is greater than or equal to 0.05, qualify non-detected semivolatile target compounds based on professional judgment.

c. If the continuing calibration RRF is less than 0.05, qualify positive results that have acceptable mass spectral identification with "J" or use professional judgment.

d. If the continuing calibration RRF is less than 0.05, qualify non-detected semivolatile target compounds as unusable (R).

2. If the laboratory has failed to provide adequate calibration information, the designated representative should contact the laboratory and request the necessary information. If the information is not available, the reviewer must use professional judgment to assess the data.

3. Whenever possible, the potential effects on the data resulting from a failure to meet calibration criteria should be noted in the data review narrative.

4. If calibration criteria are grossly exceeded, this should be noted for TPO action on the ORDAS.

5. When it is suspected that relative response factors were incorrectly generated from misidentified peaks or incorrect area measurements, the laboratory should be contacted to requantitate these RRFs and associated sample results. The ORDAS should identify affected results and document the cause of the reviewer's suspicions. In addition, a CLP telephone log must be completed.

6. Positive results for compounds flagged for blank contamination (B) will not need a separate flag (J) in the data summary form for minimum RRF, %RSD, or %D outside criteria. However, these situations should be addressed in the data review narrative and issues pertaining to noncompliance should be documented on the ORDAS.

3.6 Blanks

The purpose of laboratory (or field) blank analyses is to determine the existence and magnitude of contamination problems resulting from laboratory (or field) activities. The criteria for evaluation of blanks apply to any blank associated with the samples (e.g.,

method blanks, instrument blanks, trip blanks, and equipment blanks). If problems with any blank exist, all associated data must be carefully evaluated to determine whether or not there is an inherent variability in the data, or if the problem is an isolated occurrence not affecting other data.

3.6.1 Acceptance Criteria

1. No contaminants should be found in the blanks.

2. The method blank must be analyzed on each GC/MS system used to analyze that specific group or set of samples.

3.6.2 Review Items

Form I SV-1 and SV-2, Form IV SV and chromatograms.

3.6.3 Evaluation Procedures

1. Review the results of all associated blanks, Form I SV-1 and SV-2, and chromatograms to evaluate the presence of target and non-target compounds in the blanks.

2. Verify that a method blank analysis has been reported per matrix, per concentration level, for each extraction batch and for each GC/MS system used to analyze semivolatile samples. The reviewer can use the Method Blank Summary (Form IV SV) to assist in identifying samples associated with each method blank.

3.6.4 Action

If the appropriate blanks were not analyzed with the frequency described above, then the data reviewer should use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. The situation should be noted for TPO action on the ORDAS.

Action in the case of unsuitable blank results depends on the circumstances and origin of the blank. Positive sample results should be reported unless the concentration of the compound in the sample is less than or equal to 10 times (10x) the amount in any blank for the common phthalate contaminants, or 5 times the amount for other compounds. In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank* having the highest concentration of a contaminant. The results must not be corrected by subtracting any blank value.

For qualification purposes, to determine the highest concentration of a contaminant, consider all blanks in a case associated with all samples.

Field blanks measure contamination introduced not only in the field but also from the laboratory. In general, evaluation of the impact on specific sample results is handled as with laboratory blanks. The reviewer should use caution in attributing contamination to the field as opposed to laboratory sources. However, when field-introduced contamination is suspected, it is helpful for the reviewer to consult the sampling group to identify possible sources and prevent future reoccurrences. Verified field sources of contamination should be noted in the data review narrative. If a field blank has a highest concentration of a contaminant, then all samples in the associated case are qualified "B", using the 5x and 10x rule. Other field blanks associated with the case are not qualified.

Specific actions are as follows:

1. If a semivolatile compound is found in a blank but __not__ found in the sample, no action is taken. If the contaminants found are volatile target compounds (or interfering non-target compounds) at significant concentrations above the CRQL, then this should be noted for TPO action on the ORDAS.

2. Any semivolatile compound detected in the sample (other than the common phthalate contaminants), that was also detected in any associated blank, is qualified "B" if the sample concentration is less than five times (5x) the blank concentration. For phthalate contaminants, the results are qualified "B" when the sample result is less than 10x the blank concentration.

   In using the 5x/10x rule to compare blank results to sample results which were calculated using different weights, volumes, or dilution factors, the reviewer must choose between comparing the levels detected with the instrument, the total amount of compound (ug of contamination) present in the extracts, or the final concentration of the contaminant in the sample aliquots. Often, more than one approach will be acceptable and will yield the equivalent flagging of sample results.

   a. Comparisons involving sample dry weight correction factors, but with all other calculation factors the same for sample versus blank:

      In this case, the reviewer can compare the __wet weight__ concentrations, instrument levels, or the total amount of compound (ug of contaminant) in the extracts. All of these approaches will be acceptable and will yield equivalent flagging of sample results.

   b. When the sample has a smaller initial aliquot size than the blank (purge or extraction weight/volume), but all other calculation factors beyond this

analytical step are identical (i.e., same final extract volumes, injection volumes, and extract dilution factors for sample versus blank):

- In this case, it is acceptable and equivalent to compare either instrument levels, the total amount of compound (ug of contaminant) in the extracts, or the concentration of contaminant in the extracts.

- Final concentrations of sample versus blank should not be compared.

c.   When the sample has a larger final extract volume or a greater dilution factor than the blank:

- If the laboratory contaminant may have been introduced after or during the sample dilution step, then a direct comparison of instrument levels is appropriate. For example, comparing the instrument level result for a water sample that was diluted 1:100 prior to injection would take into account possible laboratory contamination of the syringe, instrument, or dilution solvent.

- On the other hand, if it is highly probable that the contamination originated before the dilution step, then it is more appropriate to calculate and compare the total amount of compound (ug of contaminant) present in the undiluted extract of the sample versus the blank. For example, a BNA extract diluted 1:100 prior to injection may only be subject to phthalate contamination prior to the dilution step (i.e., during extraction/concentration).

- If the results of a dilution run are to be flagged (B) because of blank contamination, the reviewer should attempt to determine whether an undiluted run was also performed. If so, the undiluted run may be used to verify the presence of a compound detected at levels too high to be questioned or, conversely, to prove that a compound was actually not present at levels multiplied by a dilution factor.

The reviewer should note that blanks may not involve the same weights, volumes, or dilution factors as the associated samples. These factors must be taken into consideration when applying the "5x" and "10x" criteria, such that a comparison of the total amount of contamination is actually made.

Additionally, there may be instances where little or no contamination was present in the associated blanks, but qualification of the sample was deemed necessary. Contamination introduced through dilution is one example. Although it is not always possible to determine, instances of this occurring can be detected when contaminants are found in the diluted sample result, but are absent in the undiluted

sample result. Since both results are not routinely reported, it may be impossible to verify this source of contamination. However, if the reviewer determines that the contamination is from a source other than the sample, he/she should qualify the data. An explanation of the rationale used for this determination should be provided in the narrative accompanying the Regional Data Assessment Summary.

3. If gross contamination exists (i.e., saturated peaks by GC/MS), all affected compounds in the associated samples should be qualified as unusable (R), due to interference. This should be noted for TPO action if the contamination is suspected of having an effect on the sample results.

4. If inordinate amounts of other target compounds are found at low levels in the blank(s), it may be indicative of a problem and should be noted for TPO action.

5. The same consideration given to the target compounds should also be given to Tentatively Identified Compounds (TICs) which are found in both the sample and associated blank(s). (See Section 3.11 for TIC guidance.)

6. If an instrument blank was not analyzed following a sample analysis which contained an analyte(s) at high concentration(s), sample analysis results after the high concentration sample must be evaluated for carryover. Professional judgment should be used to determine if instrument cross-contamination has affected any positive compound identification(s). If instrument cross-contamination is suggested, then this should be noted for TPO action if the cross-contamination is suspected of having an effect on the sample results.

7. Blanks or samples run after a matrix spike or standard should be carefully examined to determine the occurrence of instrument or syringe carry-over. Since the efficiency of sample transfer can vary dramatically according to apparatus and operator techniques, professional judgment should be used in each case to determine whether sample or blank results are attributable to carry-over. Some common examples are as follows:

• Zero to one percent syringe carry-over occasionally in BNA runs.

• Higher percentages of carry-over following BNA runs that are saturated.

Sample results which are possible artifacts of carry-over should be flagged as unreliable (R).

8. When there is convincing evidence that contamination is restricted to a particular instrument, matrix, or concentration level, the 5X/10X rule will only be applied to compare contaminated blanks to certain associated samples (as opposed to all samples in the case). Some examples are as follows:

- Column bleed (siloxanes) may be localized to a particular instrument.

- Common laboratory contaminants, such as methylene chloride and phthalates, are generally too unpredictable to safely assume contamination is restricted to a particular instrument, matrix, or concentration level.

The following are examples of applying the blank qualification guidelines. Certain circumstances may warrant deviations from these guidelines.

Example 1: Sample result is greater than the Contract Required Quantitation Limit (CRQL), but is less than the 5x or 10x multiple of the blank result.

	Rule	
	10x	5x
Blank Result	7	7
CRQL	5	5
Sample Result	60	30
Qualified Sample Result	60B	30B

In the example for the "10x" rule, sample results less than 70 (or 10 x 7) would be qualified "B". In the case of the "5x" rule, sample results less than 35 (or 5 x 7) would be qualified "B".

Example 2: Sample result is less than CRQL, and is also less than the 5x or 10x multiple of the blank result.

	Rule	
	10x	5x
Blank Result	6	6
CRQL	5	5
Sample Result	4J	4J
Qualified Sample Result	4B	4B

Note that data are reported as 4B, indicating that the qualitative presence is not confirmed.

<u>Example 3</u>: Sample result is greater than the 5x or 10x multiple of the blank result.

<u>Rule</u>

	<u>10x</u>	<u>5x</u>
Blank Result	10	10
CRQL	5	5
Sample Result	120	60
Qualified Sample Result	120	60

For both the "10x" and "5x" rules, sample results exceeded the adjusted blank results of 100 (or 10x10) and 50 (or 5x10), respectively.

3.7 Surrogate Spikes

Laboratory performance on individual samples is established by means of spiking activities. All samples are spiked with surrogate compounds prior to sample preparation. The evaluation of the results of these surrogate spikes is not necessarily straightforward. The sample itself may produce effects because of such factors as interferences and high concentrations of analytes. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the evaluation and review of data based on specific sample results is frequently subjective and demands analytical experience and professional judgment. Accordingly, this section consists primarily of guidelines, in some cases with several optional approaches suggested.

3.7.1 Acceptance Criteria

1. Surrogate spikes, 4 acid compounds (3 required and 1 advisory) and 4 base/neutral compounds (3 required and 1 advisory) are added to all samples and blanks to measure their recovery in sample and blank matrices.

2. Surrogate spike recoveries for semivolatile samples and blanks must be within the limits specified on Form II SV-1 and SV-2.

3.7.2 Review Items

Form II SV-1 and SV-2 and chromatograms.

3.7.3 Evaluation Procedures

1.   Review chromatograms to check the surrogate spike recoveries on the Surrogate Recovery Form II SV-1 and SV-2.

2.   The following should be determined from the Surrogate Recovery form(s):

   a.   If any two base/neutral or acid surrogates are out of specification, or if any one base/neutral or acid extractable surrogate has a recovery of less than 10%, then there should be a reanalysis to confirm that the non-compliance is because of sample matrix effects rather than laboratory deficiencies.

   NOTE: When there are unacceptable surrogate recoveries followed by successful re-analyses, the laboratories are required to report only the successful run.

   b.   The laboratory has failed to perform satisfactorily if surrogate recoveries are out of specification and there is no evidence of reinjection of the extract, or reextraction and reanalysis (if reinjection fails to resolve the problem).

   c.   Verify that no blanks have surrogates recoveries outside the criteria.

3.   Any time there are two or more analyses for a particular fraction the reviewer must determine which are the best data to report. Considerations should include but are not limited to:

   a.   Surrogate recovery (marginal versus gross deviation).

   b.   Technical holding times.

   c.   Comparison of the values of the target compounds reported in each fraction.

   d.   Other QC information, such as performance of internal standards.

4.   When both the initial analysis and the reanalysis have surrogate recoveries outside of criteria, the data summary should normally contain the highest concentration obtained for each compound detected, provided that surrogate recoveries in the analysis being reported do not suggest a high bias. However, if a demonstrated laboratory contaminant is detected in one analysis but not the other, the negative result may be more appropriate to report.

   When the reanalysis of a fraction is within surrogate recovery criteria, the laboratory is required to provide only data for the acceptable analysis. If both sets of data are provided, and if a compound was detected in the initial analysis but not

the reanalysis, then the positive result should be reported (provided the compound is not a demonstrated laboratory contaminant). The reported result should be flagged as estimated (J), due to possible sample inhomogeneity.

5. If advisory surrogates are outside established criteria, professional judgment will be used in qualifying the sample results. If the results are outside the criteria, then qualification would only affect similar target compounds.

3.7.4 Action

Data are not qualified with respect to surrogate recovery unless two or more semivolatile surrogates, within the same fraction (base/neutral or acid fraction), are out of specification. For surrogate spike recoveries out of specification, the following approaches are suggested based on a review of all data from the case, especially considering the apparent complexity of the sample matrix.

NOTE: These actions apply to all surrogates, except for "advisory" surrogates. Professional judgment should be used in qualifying sample results based on advisory surrogate recoveries. Qualification based on advisory surrogate recoveries should be applied to similar compounds in the sample only. Specify in the narrative any actions taken based on advisory surrogate recovery.

1. If two or more surrogates in either semivolatile fraction (base/neutral or acid fraction) have a recovery greater than the upper acceptance limit (UL):

   a. Specify the fraction that is being qualified; i.e. acid, base/neutral, or both.

   b. Detected semivolatile target compounds are qualified biased high, "K".

   c. Results for non-detected semivolatile target compounds should not be qualified.

2. If two or more surrogates in either semivolatile fraction have a recovery greater than or equal to 10% but less than the lower acceptance limit (LL):

   a. Specify the fraction that is being qualified, i.e. acid, base/neutral, or both.

   b. Detected semivolatile target compounds are qualified biased low, "L".

   c. For non-detected semivolatile target compounds, the sample quantitation limit is qualified as biased low, "UL".

3. If any surrogate in either semivolatile fraction show less than 10% recovery:

   a. Specify the fraction that is being qualified, i.e. acid, base/neutral, or both.

   b. Detected semivolatile target compounds are qualified biased low, "L".

   c. Non-detected semivolatile target compounds may be qualified as unusable (R). (If advisory surrogate limits are not met, use professional judgment to qualify non-detected compounds).

Table M2-SVOA-5 QUALIFICATION OF SEMIVOLATILE ANALYTES BASED ON SURROGATE RECOVERIES				
	2 or 3 All High	2 or 3 All Low	2 or 3 Mixed High/Low	1 or More < 10% Rec.
Detected analytes	K	L	J	L
Non-detected analytes	None	UL	UJ	R

4. If two or more surrogate recoveries in either semivolatile fraction (base/neutral or acid fraction) are outside surrogate recovery limits, and one of the recoveries is below the lower limit (but > 10%) and the other recovery is above the upper limit:

   a. Specify the fraction that is being qualified, i.e., acid, base/neutral, or both.

   b. Detected semivolatile target compounds are qualified as estimated, "J".

   c. Non-detected semivolatile target compounds are qualified as estimated, "UJ".

5. In the special case of a blank analysis with surrogates out of specification, the reviewer must give special consideration to the validity of associated sample data. The basic concern is whether the blank problems represent an isolated problem with the blank alone, or whether there is a fundamental problem with the analytical process. For example, if one or more samples in the batch show acceptable surrogate recoveries, the reviewer may choose to consider the blank problem to be an isolated occurrence. However, even if this judgment allows some use of the affected data, analytical problems should be noted for TPO action. Also note if there are potential contractual problems associated with the lack of re-analysis of samples that were out of specification.

6. Whenever possible, the potential effects of the data resulting from surrogate recoveries not meeting the advisory limits should be noted in the data review narrative.

7. Positive results for compounds already flagged for blank contamination will not need a separate flag for surrogate recoveries. However, these situations should be addressed in the narrative or the support documentation.

8. When dilutions are performed which prevent detection of BNA surrogate compounds, the narrative or support documentation should indicate that extraction efficiency/method accuracy cannot be verified.

9. Although semivolatile surrogate recoveries cannot usually be correlated with specific analytes, in the following cases specific action will be allowed based upon a particular surrogate:

   a. When a semivolatile surrogate is the deuterated analog of a TCL analyte (for example, d_5-phenol and phenol), a low recovery for the surrogate can be used to flag positive results and quantitation limits as biased low for the undeuterated analog. (This applies even if no other surrogates are outside criteria or if other surrogates are biased high instead of low.)

   b. When d_{12}-terphenyl is biased low, positive results and quantitation limits for the heavier polyaromatic hydrocarbons (those which elute starting with fluorathene) can be considered as biased low. (This applies even if no other surrogates are outside criteria or if other surrogates are biased high instead of low.)

   c. When 2,4,6-tribromophenol is biased low, positive results and quantitation limits for trichlorophenols and pentachlorophenol can be considered as biased low. (this applies even if no other surrogates are outside criteria or if other surrogates are biased high instead of low.)

3.8 Matrix Spikes/Matrix Spike Duplicates

Data for matrix spikes/matrix spike duplicates (MS/MSD) are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. These data alone cannot be used to evaluate the precision and accuracy of individual samples. However, when exercising professional judgment, this data should be used in conjunction with other available QC information.

3.8.1 Acceptance Criteria

1. Matrix spike and matrix spike duplicate samples are analyzed at frequency of one MS and MSD per 20 samples of similar matrix.

2. Matrix spike and matrix spike duplicate recoveries should be within the advisory limits established on Form III SV-1 and SV-2.

3. The Relative Percent Differences (RPDs) between matrix spike and matrix spike duplicate recoveries should be within the advisory limits listed on Form III SV-1 and SV-2.

3.8.2 Data Requirements and Data Retrieval

Form III SV-1 and SV-2 and chromatograms.

3.8.3 Evaluation Procedures

1. Verify that MS and MSD samples were analyzed at the required frequency and that results are provided for each sample matrix.

2. Inspect results for the MS/MSD Recovery on Form III SV-1 and SV-2 and verify that the results for recovery and RPD are within the advisory limits.

3. Compare results (%RSD) of non-spiked compounds between the original result, MS, and MSD.

3.8.4 Action

1. No action is taken on MS/MSD data alone. However, using informed professional judgment the data reviewer may use the matrix spike and matrix spike duplicate results in conjunction with other QC criteria and determine the need for some qualification of the data.

2. The data reviewer should first try to determine to what extent the results of the MS/MSD affect the associated data. This determination should be made with regard to the MS/MSD sample itself as well as specific analytes for all samples associated with the MS/MSD.

3. In those instances where it can be determined that the results of the MS/MSD affect only the sample spiked, then qualification should be limited to this sample alone. However, it may be determined through the MS/MSD results that a laboratory is

having a systematic problem in the analysis of one or more analytes, which affects all associated samples.

4.   The reviewer must use professional judgment to determine the need for qualification of positive results of non-spiked compounds.

NOTE: If a field blank was used for the MS/MSD, a statement to that effect must be included for TPO action on the ORDAS.

5.   When extremely low % recoveries are noted, qualify data for all affected compounds using professional judgment.

6.   When non-spiked compounds are present in either the MS or MSD results, a table in the data review narrative is constructed showing original (unspiked) sample results for non-spiked compounds, non-spiked compounds present in the MS and MSD and the calculated %RSD.

3.9 Internal Standards

Internal Standards (IS) performance criteria ensure that GC/MS sensitivity and response are stable during every analytical run.

3.9.1 Acceptance Criteria

1.   Internal standard area counts for samples and blanks must not vary by more than a factor of two (- 50% to + 100%) from the associated calibration standard.

2.   The retention time of the internal standards in samples and blanks must not vary by more than $\pm$ 30 seconds from the retention time of the associated calibration standard.

3.9.2 Review Items

Form VIII SV-1 and SV-2 and chromatograms.

3.9.3 Evaluation Procedures

1.   Check raw data (e.g., chromatograms and quantitation lists) for samples and blanks to verify the internal standard retention times and areas reported on the Internal Standard Area Summary (Forms VIII SV-1, VIII SV-2).

2.   Verify that all retention times and IS areas are within the required criteria.

3. If there are two analyses for a particular fraction, the reviewer must determine which are the best data to report. Considerations should include:

    a.   Magnitude and direction of the IS area shift.

    b.   Magnitude and direction of the IS retention time shift.

    c.   Technical holding times.

    d.   Comparison of the values of the target compounds reported in each fraction.

3.9.4 Action

1. If an IS area count for a sample or blank is outside - 50% or + 100% of the area for the associated standard:

    a.   Positive results for compounds quantitated using that IS should be qualified with "J".

    b.   Non-detected compounds quantitated using an IS area count greater than +100% should be qualified with "UJ".

    c.   Non-detected compounds quantitated using an IS area count less than 50% are reported as the associated sample quantitation limit and qualified with "UJ".

    d.   If extremely low area counts are reported, or if performance exhibits a major abrupt drop-off, then a severe loss of sensitivity is indicated. Non-detected target compounds should then be qualified as unusable (R).

2. If an IS retention time varies by more than 30 seconds:

The chromatographic profile for that sample must be examined to determine if any false positives or negatives exist. For shifts of a large magnitude, the reviewer may consider partial or total rejection (R) of the data for that sample fraction. Positive results should not need to be qualified with "R" if the mass spectral criteria are met.

3. If the internal standards performance criteria are grossly exceeded, then this should be noted for TPO action. Potential effects on the data resulting from unacceptable internal standard performance should be noted in the data review narrative.

3.10 Reported CRQLS

The objective is to ensure that the reported quantitation results and Contract Required Quantitation Limits (CRQLs) for semivolatile target compounds are accurate.

3.10.1 Acceptance Criteria

1. The CRQL must be calculated according to the correct equation and account for moisture content and dilution factor as appropriate.

3.10.2 Review Items

Form I SV-1 and SV-2 and chromatograms.

3.10.3 Evaluation Procedures

1. Verify that the CRQLs have been adjusted to reflect all sample dilutions, concentrations, splits, clean-up activities, and dry weight factors that are not accounted for by the method.

3.10.4 Action

1. If there are any discrepancies found, the laboratory may be contacted by the designated representative to obtain additional information that could resolve any differences. If a discrepancy remains unresolved, the reviewer must use professional judgment to decide which value is the best value. Under these circumstances, the reviewer may determine qualification of data is warranted. Decisions made on data quality should be included in the data review narrative. A description of the reasons for data qualification and the qualification that is applied to the data should be documented in the data review narrative.

2. Numerous or significant failures to properly evaluate and adjust CRQLs should be noted for TPO action.

3. The reviewer must assure that any results in error by more than 10 percent are identified and corrected on the sample data summary. If laboratory resubmission is not performed, the reviewer should document his/her changes to the data in the narrative or support documentation. Calculation errors should also be noted on the ORDA.

3.11 Tentatively Identified Compounds

Chromatographic peaks in semivolatile fraction analyses that are not target analytes, surrogates, or internal standards are potential tentatively identified compounds (TICs). TICs must be qualitatively identified by a National Institute of Standards and Technology (NIST) mass spectral library search and the identifications assessed by the data reviewer.

3.11.1 Acceptance Criteria

For each sample, the laboratory must conduct a mass spectral search of the NIST library and report the possible identity for the 20 largest semivolatile fraction peaks which are not surrogate, internal standard, or target compounds, but which have area or height greater than 10 percent of the area or height of the nearest internal standard. TIC results are reported for each sample on the Organic Analyses Data Sheet (Form I SV-TIC).

NOTE: Since the SOW revision of October 1986, the CLP does not allow the laboratory to report as tentatively identified compounds any target compound which is properly reported in another fraction. For example, late eluting volatile target compounds should not be reported as semivolatile TICs.

3.11.2 Review Items

Form I SV-TIC, chromatograms.

3.11.3 Evaluation Procedures

1. Guidelines for tentative identification are as follows:

   Ensure that TIC results are reported on Form I's.

2. Blank chromatograms should be examined to verify that TIC peaks present in samples are not found in blanks. When a low-level non-target compound that is a common artifact or laboratory contaminant is detected in a sample, a thorough check of blank chromatograms may require looking for peaks which are less than 10 percent of the internal standard height, but present in the blank chromatogram at a similar relative retention time.

3. The reviewer should be aware of common laboratory artifacts/contaminants and their sources (e.g., aldol condensation products, solvent preservatives, and reagent contaminants). These may be present in blanks and not reported as sample TICs.

   Examples:

a.   Common laboratory contaminants: CO_2 (m/z 44), siloxanes (m/z 73), diethyl ether, hexane, certain freons (1,1,2-trichloro-1,2,2-trifluoroethane or fluoro-trichloromethane), and phthalates at levels less than 100 ug/L or 4000 ug/Kg.

b.   Solvent preservatives, such as cyclohexene which is a methylene chloride preservative. Related by-products include cyclohexanone, cyclohexenone, cyclohexanol, cyclohexenol, chlorocyclohexene, and chlorocyclohexanol.

c.   Aldol reaction products of acetone include: 4-hydroxy-4-methyl-2-pentanone, 4-methyl-2-penten-2-one, and 5,5-dimethyl-2(5H)-furanone.

4.   Occasionally, a target compound may be identified as a TIC in the proper analytical fraction by non-target library search procedures, even though it was not found on the quantitation list. If the total area quantitation method was used, the reviewer should request that the laboratory recalculate the result using the proper quantitation ion. In addition, the reviewer should evaluate other sample chromatograms and check library reference retention times on quantitation lists to determine whether the false negative result is an isolated occurrence or whether additional data may be affected.

5.   Target compounds may be identified in more than one fraction. Verify that quantitation is made from the proper fraction.

3.11.4 Action

1.   All TIC results should be qualified "J", estimated concentration on the Laboratory Form I-TICs.

2.   Blank Results

Form I-TIC which contain sample results that are questioned by blank results, should be flagged "B" and a line drawn through these data for emphasis (initialed and dated).

To be considered questionable, a sample TIC concentration must be within 10 times the concentration of one of the blank results. If different volumes/weights are used, the total amount of compound in the extract must be compared for sample versus blank. In general, blanks analyzed within the same case, by the same lab, may be cross-applied to either soil or water samples extracted or analyzed on other days.

All blank results must be attached in the support documentation section of the data review.

3. When a compound is not found in any blanks, but is a suspected artifact of common laboratory contamination, the reviewer should cross off the reported TIC result on the copy of the Form I-TIC and note the reason(s) in the narrative.

4. Physical constants, such as boiling point, may be factored into professional judgment of TIC results.

5. Failure to properly evaluate and report TICs should be noted for TPO action.

Appendix C-1
Validation of Pesticides and PCB Analyte Data
Manual Level M1

1. Purpose and Applicability

This procedure provides step-by-step instructions to manually validate pesticide and polychlorinated biphenyl (pest/PCBs) data using the manual innovative data validation approach at Level M1.

The procedure is applicable to the Pesticide/PCB data obtained using the Contract Laboratory Program Statement of Work (CLP SOW). Hard copy data conforming to the applicable CLP SOW specifications are essential in order to carry out the procedure.

Data validated using this procedure are considered usable for the following types of purposes; however, the data users must decide on a case-by-case basis whether the procedure is suitable for their intended data uses. The suggested data uses are:

- Oversight of activities led by other parties

- Action level comparison

- Initial site investigation

- Contamination sources

2. Quality Control Measures Checked

Table M1-PEST-1 highlights the quality control (QC) indicators evaluated under this data validation procedure.

3. Procedure

The following subsections describe for each of the QC indicators the acceptance criteria, location and retrieval of QC data, evaluation of the QC data, actions taken in the event the QC acceptance criteria are exceeded, and documentation of the QC violations in a standardized report form.

Table M1-PEST-1 QC CHECKLIST FOR LEVEL M1 CLP RAS PESTICIDES/POLYCHLORINATED BIPHENYLS			
QC MEASURES	**Manual**		
	M1	**M2**	**M3**
Action Level Notification	X	X	X
GC/ECD Performance Check		X	X
Initial Calibration (CF)		X	X
Initial Calibration (%RSD)		X	X
Continuing Calibration (RPD)		X	X
Laboratory Blank	X	X	X
MS/MSD (%R, RPD)		X	X
Field Quality Control (dup., blnk., PE samp.)	X	X	X
Sample Paperwork	X	X	X
Holding Time		X	X
Retention Time	X	X	X
Surrogate Recovery		X	X
Dilution Factor		X	X
Moisture Content			X
Pesticide Cleanup Checks		X	
Chromatograms	X	X	X
Raw Data			X

Reporting requirements for Level M1 are:

- Hand annotate the Form I's, including
    - Data validation qualifiers
    - Sample identification number
    - Sampling location

- Provide a narrative that includes
    - A statement that defines the level of the data review, i.e., M1
    - Major problems associated with analysis

- Include the following attachments
    - List of data validation qualifiers
    - Support documentation including forms that support assigning data qualifiers
    - Chain of custody form

3.1 Action Level Notification

The purpose behind action level notification is to make the EPA Remedial Project Officer (RPM) or the Site Project Officer (SPO) aware of the potential human health risk at the site. In accordance with the Region III Hazardous Waste Division policy, the EPA RPM or SPO must be promptly notified of any contaminant exceeding the established action level or the 10-day health advisory limit. The data for contaminants exceeding the action levels must be validated as a top priority and reported to the RPM or SPO. Validation of the rest of the data may then be completed normally.

3.1.1 Acceptance Criteria

Region III Hazardous Waste Division has established 10-day advisory limits or the action levels for several organic compounds and elements of special health risk concern. The pesticide organic compounds and their 10-day health advisory limits apply only to aqueous samples and are listed in Table M1-PEST-2. The criteria for action level notification are as follows:

- The contaminant concentration must be equal to or above the established 10-day health advisory limits.

- Data for contaminants exceeding the action levels must be validated as a top priority.

- The following EPA personnel must be notified of the action level exceedances:

EPA RPM or SPO

EPA Section Chiefs:

—   Site Investigation (SI)

—   Remedial

—   Enforcement

—   RCRA

EPA Section Toxicologists:

—   Enforcement

—   Superfund

—   RCRA

- The remaining data validation should be completed per normal procedures.

- Any special instructions from the Hazardous Waste Division should be followed.

- Records should be kept of the data review, action level notification and any follow-up instructions and actions.

Table M1-PEST-2 PESTICIDES AND POLYCHLORINATED BIPHENYLS AND ACTION LEVELS			
Compound	Action Level*	Compound	Action Level*
Chlordane	63	Endrin	5
Heptachlor	10	Lindane	1,200
Methoxychlor	2,000	Toxaphene	80
*All units are ug/l.			

3.1.2 Review Items

All data required to perform the complete Level M1 validation, as detailed in the following sections are necessary for carrying out action level notification. The location of the data and their retrieval procedures are also discussed below.

3.1.3 Evaluation Procedure

The evaluation process preceding action level notification will primarily consist of comparing the results on Form I's with the action levels presented in Table M1-PEST-2. Following the identification of the contaminants exceeding the action levels, focused data validation should be performed using the criteria, and procedures described in the appropriate sections below.

3.1.4 Action

The action resulting from focused data validation will be the notification of action level exceedance to the personnel identified above in Section 3.1.1.

3.1.5 Reporting

Copies of Form I's can be used to highlight the contaminants above the action levels. The findings of the focused validation can be summarized in a memorandum, and the data qualifiers resulting from focused validation may be written on the Form I's. The marked up forms should be clarified that they represent validation of only the contaminants exceeding the action levels, and not all data.

3.2 Evaluation of Retention Times

Retention times are the only tool (in the event that a mass spectral analysis was not required) that allow for the identification of the pesticides and PCBs. While a retention time from a single column is not an unequivocal proof of a compound's presence, if the retention time of the suspected compound on a second column also matches that of a standard, then the compound's presence is deemed confirmed. There is reasonable probability that a non-target compound may have the same retention time as a target compound on one gas chromatographic column, but the probability of the two compounds having same retention times also on a second column is indeed very remote. For this reason, the methods utilizing non-specific detectors require that the analyses be performed under two separate sets of chromatographic conditions

3.2.1 Acceptance Criteria

The retention time acceptance criteria have been established by the EPA and Region III as follows:

> The retention times of both of the surrogates, matrix spikes, and reported compounds in each sample must be within the calculated retention time windows on both columns. The acceptable windows are ±0.05 minutes for compounds eluting before heptachlor epoxide and ±0.07 minutes for compounds eluting after and including heptachlor epoxide. The retention time for the surrogate tetrachloro-m-xylene (TCX) must be within ±0.05 minutes of the mean retention time determined from the initial calibration and that for decachlorobiphenyl (DCB) must be within ±0.10 minutes of the mean retention time determined from the initial calibration.

3.2.2 Review Items

The instrument level printouts or the quantitation reports are required to obtain the retention times for the detected compounds. These are included in the raw data sections of the data package for the calibrations as well as the samples. Additionally, Forms VIII and X would be helpful in substantiating and documenting any discrepancies. It also may be helpful to use a copy of the necessary calibration quantitation reports for a comparison with the sample retention times.

3.2.3 Evaluation Procedures

Note: It is important to emphasize that the evaluation of the retention times goes hand-in-hand with the chromatographic evaluations. During the evaluation of any one of these QC measures, a substantial use of the other QC measure is involved. Therefore, it may be beneficial to carry out these two evaluations together.

- Review the quantitation reports and chromatograms for retention times in the standards and samples. Use Forms I, VIII and X as additional tools for documenting and confirming the accuracy of the reported data. Confirm reported detected analytes by comparing the sample chromatograms to the tabulated results and verifying peak measurements and retention times. Acceptable ranges for the pesticides and PCBs are presented in Table M1-PEST-3. Ensure that the sample retention times are within these ranges for the compounds reported as detected and confirmed by the second column analysis. Perform a similar evaluation of the associated blank data for a confirmation of the reported laboratory contaminants, if any.

- For multi-component target compounds (Toxaphene and Aroclors), the retention times and relative peak height ratios of major component peaks should be compared against the appropriate standard chromatograms.

3.2.4 Action

If the qualitative criteria for both columns were not met, all target compounds that are reported detected should be considered nondetected. The reviewer may need to use the qualifiers that are specific to pesticides. The reviewer should use professional judgment to assign an appropriate quantitation limit using the following guidance.

- If the misidentified peak was sufficiently outside the target pesticide retention time window, then the reported values may be a false positive and should be replaced with the sample CRQL value.

- If the misidentified peak poses an interference with potential detection of a target peak, the reported value should be considered and qualified as unusable (R).

- If the data reviewer identifies a peak in both GC column analyses that falls within the appropriate retention time window, but was reported as a nondetect, then the compound may be a false negative. Professional judgment should be used to decide if the compound should be included.

    All conclusions made regarding target compound identification should be included in the data review narrative.

3.2.5 Reporting

Include one or more Forms M1-PEST-WNDW (example provided by Table M1-PEST-4) to represent the acceptance windows for the retention times based on the calibrations. Record any deviations of the retention times on the Form M1-PEST-RT for the detected compounds in each sample. Include these documents in the report for Level M1 data validation results. If the data need qualification, enter appropriate qualifier code on the sample Form I's, and attach these to the report.

3.3 Evaluation of Chromatograms For Detected Compounds

The purpose behind evaluating the chromatograms is to get an idea regarding potential false negatives, and gross analytical errors. Some idea as to the false positives may also be derived by checking the chromatograms. Evaluation of the positive data as described under

Dates Analyzed: Instrument ID:

		Primary Column		Secondary Column	
Compound	Retention Time Window in Minutes	Std. Ret. Time	Range	Std. Ret. Time	Range
alpha-BHC	±0.05				
beta-BHC	±0.05				
delta-BHC	±0.05				
gamma-BHC (Lindane)	±0.05				
Heptachlor	±0.05				
Heptachlor epoxide	±0.07				
Endosulfan I	±0.07				
Dieldrin	±0.07				
4,4'-DDE	±0.07				
Endrin	±0.07				
Endosulfan II	±0.07				
4,4'-DDD	±0.07				
Endosulfan sulfate	±0.07				
4,4'-DDT	±0.07				
Methoxychlor	±0.07				
Endrin ketone	±0.07				
Endrin aldehyde	±0.07				
alpha-Chlordane	±0.07				
gamma-Chlordane	±0.07				
Toxaphene	±0.07				
Aroclor 1016	±0.07				
Aroclor 1221	±0.07				
Aroclor 1232	±0.07				
Aroclor 1242	±0.07				
Aroclor 1248	±0.07				
Aroclor 1254	±0.07				
Aroclor 1260	±0.07				
Tetrachloro-m-xylene (Surr.)	±0.05				
Decachlorobiphenyl (Surr.)	±0.10				

Table M1-PEST-3
Pesticides/Polychlorinated Biphenyls Retention time Windows

Case No.:_____
SDG No.:_____
Data Reviewer:_____
Site:_____

Procedure No.: M1-PEST
Revision: 1
Date: 06/30/1995
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Table M1-PEST-4
PEST/PCB Retention Time Evaluation Summary

Dates Analyzed:	Sample Identifier:		Comments	
	1.		1.	
	2.		2.	
	3.		3.	
	4.		4.	
Instrument ID:	5.		5.	
	6.		6.	
Column (1):	7.		7.	
	8.		8.	
Column (2):	9.		9.	
	10.		10.	

Sample Identifier & Retention Times for Primary Column (1) and Secondary Column (2)

Compound:		1		2		3		4		5		6		7		8		9		10	
		(1)	(2)	(1)	(2)	(1)	(2)	(1)	(2)	(1)	(2)	(1)	(2)	(1)	(2)	(1)	(2)	(1)	(2)	(1)	(2)
alpha-BHC																					
beta-BHC																					
delta-BHC																					
gamma-BHC (lindane)																					
Heptachlor																					
Heptachlor epoxide																					
Endosulfan I																					
Dieldrin																					
4,4'-DDE																					
Endrin																					
Endosulfan II																					
4,4'-DDD																					
Endosulfan sulfate																					
4,4'-DDT																					
Methoxychlor																					
Endrin ketone																					
Endrin aldehyde																					
alpha-chlordane																					
gamma-chlordane																					
Toxaphene	Peak 1																				
	Peak 2																				
	Peak 3																				
Aroclor 1016	Peak 1																				
	Peak 2																				
	Peak 3																				
Aroclor 1221	Peak 1																				
	Peak 2																				
	Peak 3																				
Aroclor 1232	Peak 1																				
	Peak 2																				
	Peak 3																				
Aroclor 1242	Peak 1																				
	Peak 2																				
	Peak 3																				
Aroclor 1248	Peak 1																				
	Peak 2																				
	Peak 3																				
Aroclor 1254	Peak 1																				
	Peak 2																				
	Peak 3																				
Aroclor 1260	Peak 1																				
	Peak 2																				
	Peak 3																				

mass spectral evaluation does not offer any insight into data that are not reported (i.e., reported as nondetects). Laboratory error or gross interference from other compounds could be the reasons for erroneous reporting. The gas chromatographic profiles are the primary tool used for the false negative evaluation under Level M1.

3.3.1 Acceptance Criteria

There are no EPA-established quantitative criteria for evaluating chromatograms for false negatives. The criteria used for evaluating chromatograms are based on good laboratory and scientific practices, and these are not hard and fast requirements. The suggested evaluation criteria are as follows:

- Chromatograms must display single component pesticides detected in the sample and the largest peak of any multi-component analyte detected in the sample at less than full scale.

- If an extract must be diluted, chromatograms must display single component pesticides between 10 and 100 percent of full scale, and multicomponent analytes between 25 and 100 percent of full scale.

- For any sample, the baseline of the chromatogram must return to below 50 percent of full scale before the elution time of alpha-BHC, and also return to below 25 percent of full scale after the elution time of alpha-BHC and before the elution time of decachlorobiphenyl.

- If a chromatogram is replotted electronically to meet these requirements, the scaling factor used must be displayed on the chromatogram, and both the initial chromatogram and the replotted chromatogram must be submitted in the data package.

- There should not be any significant peaks in the chromatograms that are accounted for as TCLs. Significant peaks are those with a minimum peak height of 10 percent of the full scale deflection.

- The chromatograms should ideally have base-line resolution between adjacent peaks. Also, there should not be broad (unresolved) envelops in the chromatograms.

- There should not be abrupt shifts in the baseline.

- There should not be peak tailing or sharp rise in the peak fronts.

3.3.2 Review Items

Chromatograms for each sample and standard calibrations are necessary for the evaluation. These can be found in the front of the raw data package for each sample. The quantitation report for each sample is also necessary to retrieve the retention times since the chromatographic profile from a single column can be inconclusive. A copy of the standard chromatograms from both chromatographic columns may be very helpful in visually comparing the sample traces for fingerprint features.

3.3.3 Evaluation Procedures

Note: It is important to emphasize that the evaluation of the chromatograms goes hand-in-hand with the retention time evaluations. During the evaluation of any one of these QC measures, a substantial use of the other QC measure is involved. Therefore, it may be beneficial to carry out these two evaluations together.

- Visually inspect the primary and the secondary column chromatograms for all peaks that appear to be at least 10 percent of the full scale deflection. Compare the retention time of the suspect compound on the primary column with that of the standard. If the retention time is close to the acceptance range, then check the retention time on the secondary column. If there is clear disagreement in the retention time the compound is likely not a target compound and should not have been reported.

- If multicomponent target compounds exhibit marginal pattern-matching quality, professional judgment should be used to establish whether the differences are due to environmental "weathering" (i.e., degradation of the earlier eluting peaks relative to the later eluting peaks). If the presence of a multicomponent pesticide is strongly suggested, results should be reported as presumptively present (N).

- If an observed pattern closely matches more than one Aroclor, professional judgment should be used to decide whether the neighboring Aroclor is a better match, or if multiple Aroclors are present.

- If GC/MS confirmation was required but not performed, the reviewer should report this for TPO action.

- Also observe the chromatogram traces for peak resolution between the adjacent single component peaks. Poor peak-to-peak resolution is indicative of degrading performance of the gas chromatographic column. The values obtained from a degrading system are prone to be inaccurate. Generally,

there should be at least 90 percent valley between the neighboring peaks. This criteria is important for detectors such as the electron capture detector (ECD) that do not allow for unequivocal identification. Poor peak resolution between the adjacent peaks may results in estimated quantitation of both for both compounds.

- Inspect the ECD profile for broad, unresolved envelops. These are generally indicative of outside interference from homologous compounds. Such envelops can mask the target peaks or interfere with accurate quantitation of the peaks.

- Inspect the ECD profile for abrupt shifts in the baseline. Such shifts are indicative of problems with instrument sensitivity or leakage in the system. The area counts obtained from shifted baseline are inaccurate, or even the detection of a TCL at low concentration may be missed.

- Rapid peak rising or peak tailing indicate problems with the gas chromatographic column, such as depleted stationary phase on the column, decomposition of the stationary phase or creation of active sites. Again, a visual inspection of the ECD profile will yield information on the shape of the peak.

3.3.4 Action

Professional discretion must be used when evaluating and qualifying data based on the chromatographic evaluations. An experienced chemist can generally infer the magnitude and the frequency of the problem from the ECD profile and fingerprints. If the problem appears to be systematic, then it should be brought to the laboratory's attention and resolved. Intermittent problems may or may not require any action. The following guidelines are suggested when acting on ECD profile observations:

- Any unaccounted TCL peak (as confirmed by the retention times on both columns) with area equivalent to or greater than the lowest reportable limit for the sample must be brought to the laboratory's attention and resolved. In the event, the discrepancy cannot be resolved with the laboratory, the problems should be documented and brought to the attention of the CLP-TPO, the RPM and the SM. The data for unreported TCLs may be considered unusable until the problems are resolved.

- If a peak resolution problem is evident for the samples, and appears to be systematic (i.e., present in all calibration samples, QC samples, and field samples and increasing as the run progresses, additional QC measures such

as the continuing calibration percent difference (%D), and surrogate standard recoveries in the vicinity of the affected peaks should be evaluated to determine if the peak resolution problem could affect detection or quantitation. If determined so, the positive data may be qualified as estimated, "J." Negative data may also be qualified as estimated, "UJ" if the ability to detect at low concentrations is also deemed to be jeopardized by poor resolution of adjacent peaks.

Broad envelopes of homologous compounds could interfere with quantitation or even detection. If the interference is evident from the recoveries of the surrogate standards in the vicinity of the envelop, associated compounds may also be interfered with. Using professional discretion, the positive and negative data may be considered as estimated, "J" and "UJ", respectively. If the project objectives cannot be met with the estimated data, alternative sample preparation and cleanup procedures may need to be developed and specified. The recommended solution should be brought to the attention of the RPM, the SM and the TPO.

Discrete shifts in the baseline in the middle of a run are indicative of intermittent problems. If the shift is due to leakage or change in the system pressure, the positive as well as negative data may be considered estimated ("J" and "UJ, respectively). The problem could be also due to some fluctuation in the instrument electronics which may lead to drastic changes in the sensitivity of the instrument to detect the compounds. As a note, professional judgment should be exercised in determining the severity of the problem. For example, the magnitude of a drop in the baseline below the zero line may not be estimated and could be very significant. On the other hand, a drop that yields a baseline still above zero can be put in a perspective with the original baseline and a general appearance of the entire RIC profile.

The problems with peak symmetry are indicative of system degradation, and should be brought to the attention of the laboratory for a corrective action. Professional judgment should be used when and if qualifying any data due to unsymmetrical peaks. First the problem should be defined in terms of persistence throughout the chromatogram and also from sample to sample. Additionally, the shapes and area counts for the surrogate standards should be evaluated to see if the problem could have affected compound detection and/or quantitation. Data qualification may be uncalled for if the standard area counts are acceptable.

3.3.5 Reporting

Keeps notes of the problems and substantiate them with copies of both chromatograms and other pertinent laboratory paperwork. Include these items in the overall report for Level M1 data validation. For more descriptive comments, a separate sheet may be used.

3.4 Evaluation of Blanks

The purpose of laboratory (or field) blank analyses is to determine the existence and magnitude of contamination problems resulting from laboratory (or field) activities. The criteria for evaluation of laboratory blanks apply to any blank associated with the samples (e.g., method blanks, instrument blanks, and sulfur cleanup blanks). If problems exist with any type of blank, all associated data must be carefully evaluated to determine whether or not there is an inherent variability in the data, or if the problem is an isolated occurrence not affecting other data.

Laboratory blanks and field blanks have a profound impact on false positives reported in samples; i.e., compounds reported as positive detects but not originating from the samples themselves. Cross contamination from the sampling equipment, incidental contamination from the field conditions or contamination from the laboratory equipment or general environmental are likely sources of false positives in the samples.

3.4.1 Acceptance Criteria

Criteria for blank evaluation are specified in the EPA's functional guidelines. In addition, Region III has some additional requirements modifying the guidance. The acceptance criteria for blanks apply equally to any type of blanks associated with either sampling or analysis, such as trip blanks, rinsate blanks, field or bottle blanks, laboratory method blanks. While there are several criteria for evaluating the blanks, the only criteria applicable to Level M1 is the comparison of the blank and sample concentrations. These criteria are as follows:

> No contaminants should be present in the blanks.

> Frequency of Blank analyses:

>> Method Blanks—A method blank analysis must be performed for each 20 samples of similar matrix in each sample delivery group (SDG) or whenever a sample extraction procedure is performed.

>> Instrument Blanks—An acceptable instrument blank must be run at least once every 12 hours and immediately prior to the analysis of

either the performance evaluation mixture or Individual Standard Mixtures A and B, depending on the place in the analysis sequence.

Sulfur Cleanup Blanks—A sulfur cleanup blank must be analyzed whenever part of a set of samples extracted together requires sulfur cleanup. If the entire set of samples associated with a method blank requires sulfur cleanup, then the method blank also serves the purpose of a sulfur blank and no separate sulfur blank is required.

3.4.2 Review Items

Data requirements and data retrieval procedures for blanks are the same as those for the field samples because the blanks as well as field samples are validated similarly. Form I's, mass spectra, chromatograms, quantitation reports, etc., are essential for performing a validation of the blanks first.

3.4.3 Evaluation Procedure

Validate the blanks same as the field samples. Detailed validation procedures are described above under appropriate sections. Use the validated blank results for a comparison with the sample results. Make certain that the samples and blanks are evaluated on the same basis of sample weight or volume, dilution factors, moisture content, etc. Use the 5 (or 10) times the highest blank concentrations for qualifying the sample data. More specifically, the blank data evaluation procedures are as follows:

- Review the results of all associated blanks, Form I PEST, and Form IV PEST, and chromatograms to evaluate the presence of TCL pesticides.

- Verify that method blank analysis has been reported per SDG, per matrix, per concentration level, for each GC system used to analyze samples, and for each extraction batch.

- Verify that the method blank analyses do not contain any target pesticide or Aroclor/Toxaphene at greater than its Contract Required Quantitation Limits (CRQL).

- For the surrogates in each method blank, verify that the observed retention times are within the appropriate retention time windows calculated from the initial calibration.

- Verify that the instrument blank analysis has been performed every 12 hours as part of the continuing calibration and following a sample analysis which

contains an analyte(s) at high concentration(s), and that the instrument blanks do not contain any target analytes above one-half the CRQL, assuming that the material in the instrument resulted from the extraction of a 1-L water sample.

Verify that the sulfur cleanup blanks were analyzed at the required frequency and that they do not contain any target compound above the CRQL, assuming that the material in the instrument resulted from the extraction of a 1-L water sample. If a separate sulfur cleanup blank was prepared, one version of Form IV PEST should be completed associating all the samples with the method blank, and a second version of Form IV PEST should be completed listing only those samples associated with the separate sulfur cleanup blank.

3.4.4 Action

If the appropriate blanks were not analyzed with the frequency described in Criteria 3, 4, and 5, then the data reviewer should use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. The situation should be brought to the attention of the TPO.

Action in the case of unsuitable blank results depends on the circumstances and the origin of the blank. Detected compound results should be reported unless the concentration of the compound in the sample is less than or equal to 5 times (5x) the amount in the blank. In instances where more than one blank is associated with a given sample, qualification should be based on a comparison with the associated blank having the highest concentration of a contaminant. The results must not be corrected by subtracting the blank value.

Specific actions are as follows:

- If a target pesticide or Aroclor/Toxaphene is found in the blank but **not** found in the sample(s), no qualification is required. If the contaminant(s) is found at level(s) significantly greater than the CRQL, then this should be noted in the data review narrative.

- Any pesticide or Aroclor/Toxaphene detected in the sample, that was also detected in any associated blank, is qualified if the sample concentration is less than five times (5x) the blank concentration. The quantitation limit may also be elevated. Typically, the sample CRQL is elevated to the concentration found in the sample. The reviewer should use professional judgment to determine if further elevation of the CRQL is required.

- The reviewer should note that analyte concentrations calculated for method blank may not involve the same weights, volumes, or dilution factors as the associated samples. These factors must be taken into consideration when applying the "5x" criteria, such that a comparison of the total amount of contamination is actually made.

- In addition, there may be instances when little or no contamination was present in the associated blanks, but qualification of the sample was deemed necessary. Contamination introduced through dilution is one example. Although it is not always possible to determine, instances of this occurring can be detected when contaminants are found in the diluted sample result, but absent in the undiluted sample result. Since both results are not routinely reported, it may be possible to verify this source of contamination. However, if the reviewer determines that the contamination is from a source other than the sample, he/she should qualify the data. In this case, the "5x" rule does not apply; the sample value should be reported as a nondetected target compound, "U."

- If gross contamination exists (i.e., saturated peaks), all affected compounds in the associated samples should be qualified as unusable (R), due to interference. This should be noted in the data review narrative if the contamination is suspected of having an effect on the sample results.

- If inordinate amounts of other target pesticides or Aroclors/Toxaphene are found at low levels in the blank(s), it may be indicative of a problem at the laboratory and should be noted in the data review narrative.

- If an instrument blank was not analyzed following a sample analysis which contained an analyte(s) at high concentration(s), sample analysis results after the high concentration sample must be evaluated for carryover. Professional judgment should be used to determine if instrument cross-contamination has effected any positive compound identification(s), and if so, detected compound results should be qualified. If instrument cross-contamination is suggested, then this should be noted in the data review narrative if the cross-contamination is suspected of having an effect on the sample results.

The following are examples of applying the blank qualification guidelines. Certain circumstances may warrant deviations from these guidelines:

Example 1: Sample result is greater than the CRQL, but is less than the 5x multiple of the blank result.

	5x
Blank Result	1.0
CRQL	0.5
Sample Result	4.0
Qualified Sample Result	4.0B

In this case, sample results less than 5.0 (or 5 x 1.0) would be qualified as nondetected target compounds.

Example 2: Sample result is less than the CRQL, and is also less than the 5x multiple of the blank result.

	5x
Blank Result	1.0
CRQL	0.5
Sample Result	0.4J
Qualified Sample Result	0.4B

Example 3: Sample result is greater than the 5x multiple of the blank result.

	5x
Blank Result	1.0
CRQL	0.5
Sample Result	10.0
Qualified Sample Result	10.0

In this case, the sample result exceeded the adjusted blank result (5x 1.0) and the sample result is not qualified.

● Record blank contaminants on QSFs.

If the sample concentration do not meet the criteria of 5 (or 10) times the blank concentration, the sample results should be considered essentially undetected (or as not detected substantially above the levels reported in the blanks); therefore, flagged ''B'' in accordance with the Region III data validation guidelines.

3.4.5 Reporting

Form I's may be used to write the "B" data qualifier for the data not meeting the blank criteria. Additional discrepancies may be included in the overall report for the Level M1 validation.

3.5 Sample Paperwork

The purpose for evaluating the sample paperwork is to determine that the samples being validated are indeed the ones taken from the site, and have not been tampered with. Accurate sample identity is of paramount importance in substantiating the sample data. Without unequivocal sample identity and chain-of-custody procedures, the sample data may not be defensible or enforceable.

Under the current CLP contracts, the original paperwork (i.e., the purge package or the administrative record) is included in the data package from the laboratory. It is assumed that the data validator is not privy to the original paperwork; therefore, the evaluation criteria and procedures described below apply only to the documents that are generally included in the data validation package. These documents are the chain-of-custody forms and Region III Shipping Record.

3.5.1 Acceptance Criteria

Criteria for acceptability or authenticity of the sampling paperwork, document control and chain-of-custody have been established by the National Enforcement Investigations Center (NEIC), in support of the CLP. Overall criteria are too numerous and subjective to be discussed here, but the criteria that apply to data validation are:

- The chain-of-custody form should be properly and completely filled out including the sampler signatures, date and time of sampling, sampling station identification, analyses requested, traffic numbers, tag numbers, etc. These data are minimally required to confirm the authenticity of the sample and its data.

- The chain-of-custody must be maintained at all times. The custody transfers between different parties must be signed and dated.

3.5.2 Review Items

A copy of the chain-of-custody form originated in the field and that returned from the laboratory with the data are essential to confirm the identity of the samples. In addition,

the Region III Shipping Record is essential to identify the field QC samples. The chain-of-custody and Shipping Record are generally located in front of the data package.

3.5.3 Evaluation Procedure

Ensure that the chain-of-custody form was signed and dated by the samplers, and a time and date were entered for sample collection. The laboratory copy of the chain-of-custody must have the signature of the laboratory sample custodian. Any errors on the form should have been crossed out with a single line through the entry. Verify that all collected samples have unique station identification, traffic numbers and sample tag numbers. Ensure that the Region III Shipping Record correctly reflects the information on the chain-of-custody.

3.5.4 Action

The action to be taken in qualifying the data is highly dependent on the nature of the problem. Some errors in paperwork are practically unavoidable in real situations. An effort should be made to reconcile the differences by cross checking the field notebooks against the sampling paperwork. Occasionally, the samplers may forget to sign the chain-of-custody; however, the field notebooks may amply describe the sampling event. Problems are also inevitable in noting or cross-referencing sample tag numbers and traffic numbers. Generally, there are several alternate sources of information to substantiate or refute the problem.

3.5.5 Reporting

Any discrepancies found in the paperwork must be immediately brought to the attention of the EPA RPM or SPO. Clearly define the problems in a memorandum to the responsible parties. Attach marked copies of the chain-of-custody forms to substantiate the findings.

Appendix C-2

Validation of Pesticide/PCB Organic Analyte Data Manual Level M2

1. Purpose and Applicability

This procedure provides step-by-step instructions to manually validate the pesticide analyte organic data using the manual innovative data validation approach at Level M2. This approach focuses on the use of information contained on the CLP forms and a review of chromatograms as summarized in Table M2-PEST-1.

The procedure is applicable to the Pesticide/PCB obtained using the Contract Laboratory Program Statement of Work (CLP SOW). Hard copy data conforming to the applicable CLP SOW specifications are essential in order to carry out the procedure.

Data validated using this procedure are considered usable for the following types of purposes; however, the data users must decide on a case-by-case basis whether the procedure is suitable for their intended data uses. The suggested data uses are:

- Oversight of activities led by other parties

- Comparison to action levels

- Initial site investigation

- Contamination sources

- Nature and extent of contamination

- Preliminary risk assessment

- Risk assessment with known high levels of toxics

- Feasibility study

- Preliminary design

- Treatability study

- Initial cleanup verification

Table M2-PEST-1 QC CHECKLIST FOR LEVEL M2 CLP RAS PESTICIDES-POLYCHLORINATED BIPHENYLS			
	Manual		
QC MEASURES	M1	M2	M3
Action Level Notification	X	X	X
GC/ECD Performance Check		X	X
Initial Calibration (CF)		X	X
Initial Calibration (%RSD)		X	X
Continuing Calibration (RPD)		X	X
Laboratory Blank	X	X	X
MS/MSD (%R, RPD)		X	X
Field Quality Control (dup., blnk., PE samp.)	X	X	X
Sample Paperwork	X	X	X
Holding Time		X	X
Retention Time	X	X	X
Surrogate Recovery		X	X
Dilution Factor		X	X
Moisture Content		X	X
Pesticide Cleanup Checks		X	
Chromatograms	X	X	X
Raw Data			X

2. Quality Control Measures Checked

Table M2-PEST-1 highlights the quality control (QC) indicators evaluated under this data validation procedure.

3. Procedure

The following subsections describe for each of the QC indicators the acceptance criteria, location and retrieval of QC data, evaluation of the QC data, actions taken in the event the QC acceptance criteria are exceeded, and documentation of the QC violations in a standardized report form.

The pesticides data requirements to be checked are listed below:

3.1   Action Level Notification

3.2   Technical Holding Times (CCS—Contractual holding times only)

3.3   GC/ECD Instrument Performance Check

3.4   Initial Calibration (CCS)

3.5   Continuing Calibration (CCS)

3.6   Blanks

3.7   Surrogate Spikes (CCS)

3.8   Matrix Spikes/Matrix Spike Duplicates

3.9   Pesticide Cleanup Checks

3.10   Reported Contract Required Quantitation Limits (CRQLs)

Two forms have been developed to assist in the performance and documentation of implementing Level M2. The first form, M2-PEST-QUAL, summarizes holding time, calibrations, blanks, surrogates, and internal standards. The second form, M2-PEST-SPK, summarizes surrogate and matrix spike quality control checks. These forms are presented on the following pages as Tables M2-PEST-2 and M2-PEST-3.

Reporting requirements for Level M2 are as follows:

- Hand annotate the Form I's, including:
  - Data validation qualifiers
  - Sample identification number
  - Sampling location

- Provide a narrative that includes:
  - A statement that defines the level of the data review, i.e., M2
  - Major and minor problems associated with the analysis
  - Highlight issues that may have affected detection limits

- Include the following attachments:
  - List of data validation qualifiers
  - Support documentation including forms that support assigning data qualifiers
  - Chain of custody form
  - Samples affected by calibration should be listed on the appropriate calibration forms

Data qualifiers assigned in this review are as follows:

Codes Relating to Identification (Confidence Concerning Presence or Absence of Compounds):

U   =   Not detected. The associated number indicates approximate sample concentration necessary to be detected.

(NO CODE) =   Confirmed identification.

B   =   Not detected substantially above the level reported in laboratory or field blanks.

R   =   Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.

N   =   Tentative identification. Consider present. Special methods may be needed to confirm its presence or absence in future sampling efforts.

Codes Related to Quantitation (can be used for both positive results and sample quantitation limits):

J   =   Analyte present. Reported value may not be accurate or precise.

Method 608/8080
Case No:_____
SDG No:_____

Procedure No.: M2-PEST
Revision: 1
Date: 06/30/1995
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TABLE M2-PEST-2
PESTICIDE/PCBS QUALIFIER SUMMARY (CALIBRATIONS, BLANKS, HOLDING TIME, SURROGATES, INTERNAL STANDARDS)

Date Analyzed:

Instrument ID:

Method Blank ID:

Extraction Date:

	Matrix	Sample Identifier:	Hold Time Out, days Ext.	Anal.	Last IND Standard before Analysis 1	2	3	4	5	6

Column: Compound:	Primary Secondary	Init. Cal. %RSD>10	Calibrations: Continuing, %D>15% or 20% 1	2	3	4	5	6	Blank	Qualifiers (+/-)
	Cont. Cal. date/month Time									
alpha-BHC										
beta-BHC										
delta-BHC										
gamma-BHC (Lindane)										
Heptachlor										
Aldrin										
Heptachlor epoxide										
Endosulfan I										
Dieldrin										
4,4'-DDE										
Endrin										
Endosulfan II										
4,4'-DDD										
Endosulfan sulfate										
4,4'-DDT										
Methoxychlor										
Endrin ketone										
alpha-chlordane										
gamma-chlordane										
Toxaphene										
Aroclor-1016 (PCB-1016)										
Aroclor-1221 (PCB-1221)										
Aroclor-1232 (PCB-1232)										
Aroclor-1242 (PCB-1242)										
Aroclor-1248 (PCB-1248)										
Aroclor-1254 (PCB-1254)										
Aroclor-1260 (PCB-1260)										
Dibutylchlorendate (DBC)	Surr.									

Validation Criteria:	Primary Column		Secondary Column
Detected compounds	%D < 15	and	%D < 20
Undetected compounds	%D < 20	or	%D < 20

Method 608/8080

Case No:_____

SDG No:_____

Procedure No.: M2-PEST
Revision: 1
Date: 06/30/1995
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Table M2-PEST-3
PESTICIDE/PCBS SURROGATE AND MATRIX SPIKE QUALITY CONTROL
SUMMARY

Surrogate Data Summary

| Sample Identifier: | Surrogate Recoveries, %R | | |
| | Aqueous | Soil | |
(Acceptance Range, %R):	(24-154)	(20-150)	Qualifiers (+/-)

Surrogate = Dibutylchlorendate (DBC)

MS/MSD Data Summary

| Spike Compound: | Matrix Spike, Recovery, %R | | Matrix Spike Duplicate, Recovery, %R | | MS/MSD Precision, RPD | | Qualifiers (+/-) |
	Range	Actual	Range	Actual	Range	Actual	
AQUEOUS SAMPLES							
gamma-BHC (Lindane)	56-123		56-123		14		
Heptachlor	40-131		40-131		20		
Aldrin	40-120		40-120		22		
Dieldrin	52-126		52-126		18		
Endrin	56-121		56-121		21		
4.4'-DDT	38-127		38-127		27		
SOIL SAMPLES							
gamma-BHC (Lindane)	46-127		46-127		50		
Heptachlor	35-130		35-130		31		
Aldrin	34-132		34-132		43		
Dieldrin	31-134		31-134		38		
Endrin	42-139		42-139		45		
4.4'-DDT	23-134		23-134		50		

K   =   Analyte present. Reported value may be biased high. Actual value is expected lower.

L   =   Analyte present. Reported value may be biased low. Actual value is expected to be higher.

UJ   =   Not detected, quantitation limit may be inaccurate or imprecise.

UL   =   Not detected, quantitation limit is probably higher.

Other Codes

Q   =   No analytical result.

*   =   Results reported from diluted analysis.

3.1 Action Level Notification

The purpose behind action level notification is to make the EPA Remedial Project Officer (RPM) or the Site Project Officer (SPO) aware of the potential human health risk at the site. In accordance with the Region III Hazardous Waste Division policy, the EPA RPM or SPO must be promptly notified of any contaminant exceeding the established action level or the 10-day health advisory limit. The data for contaminants exceeding the action levels must be validated as a top priority and reported to the RPM or SPO. Validation of the rest of the data may then be completed normally.

3.1.1 Acceptance Criteria for Action Level Notification

EPA's Office of Solid Waste and Emergency Response has established 10-day advisory limits or the action levels for several organic compounds and elements of special health risk concerns based on the Safe Drinking Water Act. The pesticide organic compounds and their 10-day health advisory limits apply only to aqueous samples and are listed in Table M2-PEST-4. The criteria for action level notification are as follows:

- The contaminant concentration must be equal to or above the established 10-day health advisory limits.

- Data for contaminants exceeding the action levels must be validated as a top priority.

- The following EPA personnel must be notified of the action level exceedances:

--   EPA RPM or SPO

--   EPA Section Chiefs:
  -    Site Investigation (SI)
  -    Remedial
  -    Enforcement
  -    RCRA

  EPA Section Toxicologists:
  -    Enforcement
  -    Superfund
  -    RCRA

- The remaining data validation should be completed per normal procedures.

- Any special instructions from the Hazardous Waste Division should be followed.

- Records should be kept of the data review, action level notification and any follow up instructions and actions.

Table M2-PEST-4 PESTICIDES AND POLYCHLORINATED BIPHENYLS AND ACTION LEVELS			
Compound	Action Level*	Compound	Action Level*
Chlordane	63	Endrin	5
Heptachlor	10	Lindane	1,200
Methoxychlor	2,000	Toxaphene	80
*All units are ug/l.			

3.1.2 Data Requirements and Retrieval of Data

All data required to perform Level M2 validation, as detailed in the following sections, are necessary for carrying out action level notification. The location of the data and their retrieval procedures are also discussed below.

3.1.3 Evaluation Procedure

The evaluation process preceding action level notification will primarily consist of comparing the results on Form I's with the action levels presented in Table M2-PEST-4. Following the identification of the contaminants exceeding the action levels, focused data validation should be performed using the criteria, and procedures described in the appropriate sections below.

3.1.4 Action

The action resulting from focused data validation will be the notification of action level exceedance to the personnel identified above in Section 3.1.1.

3.1.5 Reporting

Copies of Form I's can be used to highlight the contaminants above the action levels. The findings of the focused validation can be summarized in a memorandum, and the data qualifiers resulting from focused validation may be written on the Form I's. The marked up forms should be clarified that they represent validation of only the contaminants exceeding the action levels, and not all data.

3.2 Technical Holding Times

The objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample extraction and analysis.

3.2.1 Acceptance Criteria

Technical requirements for sample holding times have only been established for water matrices. The holding times for soils are currently under investigation. When the results are available they will be incorporated into the data evaluation process. In addition, results of holding time studies will be incorporated into the data review criteria as the studies are conducted and approved. The maximum holding time, as stated in the current 40 CFR Part 136, for pesticides and Aroclors in cooled (@ 4°C) water samples is 7 days from sample collection to extraction and 40 days from sample extraction to analysis. It is recommended that pesticides and Aroclors in soil samples be extracted within 7 days of sample collection.

The contractual holding times, which differ from the technical holding times, state that extraction of water samples by separatory funnel must be completed within 5 days of validated time of sample receipt (VTSR), extraction of water samples by continuous liquid-liquid extraction procedures must be started within 5 days of VTSR, and soil/sediment samples are to be extracted within 10 days of VTSR. Also, contractually both water and

soil sample extracts must be analyzed within 40 days of extraction. However, the contractual delivery due date is either 14 days or 35 days after receipt in the laboratory of the last sample in the SDG, depending on the contract.

3.2.2 Review Items

Form I PEST, EPA Sample Traffic Report, and/or chain-of-custody.

3.2.3 Evaluation Procedure

Technical holding times for sample extraction are established by comparing the sample collection date on the EPA Sample Traffic Report with the dates of extraction on Form I PEST. To determine if the samples were analyzed within the holding time after extraction, compare the dates of extraction on Form I PEST.

Verify that the traffic report indicates that the samples were received intact and iced. If the samples were not iced or there were any problems with the samples upon receipt, then discrepancies in the sample condition could affect the data.

3.2.4 Action

1. If technical holding times are exceeded, qualify all detected compound results as estimated "J" and sample quantitation limits as estimated "UJ," except for PCBs which are not expected to degrade significantly during storage.

2. If technical holding times are grossly exceeded, either on the first analysis or upon reanalysis, the reviewer must use professional judgment to determine the reliability of the data and the effect of additional storage on the sample results. The reviewer may determine that detected compound results or the associated quantitation limits are approximates and should be qualified with "J" or "UJ," respectively. The reviewer may determine that nondetected target compound data are unusable (R).

3. Due to limited information concerning holding times for soil samples, water holding time criteria should be applied.

4. The reviewer should also be aware of the scenario in which the laboratory has exceeded the technical holding times, but met contractual holding times. In this case, the data reviewer should notify the Regional TPO (where samples were collected) and/or RSCC that shipment delays have occurred so that the field problem can be corrected. The reviewer may pass this information on to the laboratory's TPO, but should explain that contractually the laboratory met the requirements.

3.3 GC/ECD Instrument Performance Check

Performance checks on the gas chromatograph with electron capture detector (GC/ECD) system are performed to ensure adequate resolution and instrument sensitivity. These criteria are not sample specific. Conformance is determined using standard materials; therefore, these criteria should be met in all circumstances.

3.3.1 Acceptance Criteria

1.   Resolution Check Mixture

a.   The Resolution Check Mixture must be analyzed at the beginning of every initial calibration sequence, on each GC column, and instrument used for analysis. The Resolution Check Mixture contains the following pesticides and surrogates:

- gamma-Chlordane
- Endosulfan I
- 4,4'-DDE
- Dieldrin
- Endosulfan sulfate
- Endrin ketone
- Methoxychlor
- Tetrachloro-m-xylene
- Decachlorobiphenyl

b.   The depth of the valley between two adjacent peaks in the Resolution Check Mixture must be greater than or equal to 60.0 percent of the height of the shorter peak.

2.   Performance Evaluation Mixtures

a.   The Performance Evaluation Mixture (PEM) must be analyzed at the beginning (following the resolution check mixture) and at the end of the initial calibration sequence. The PEM must also be analyzed at the beginning of every other 12-hour analytical period. The PEM contains the following pesticides and surrogates.

- gamma-BHC
- alpha-BHC
- 4,4'-DDT
- beta-BHC

- Endrin
- Methoxychlor
- Tetrachloro-m-xylene
- Decachlorobiphenyl

b.   The resolution of adjacent peaks for the PEM injections in each calibration (initial and continuing) must be 100 percent for both GC columns.

c.   The absolute retention times of each of the single component pesticides and surrogates in all PEM analyses must be within the specific retention time windows centered around the mean retention times determined from the three-point initial calibration using the individual Standard Mixtures. A list of the retention time windows is found in Attachment 1, Table 1.

For example, for a given pesticide the mean retention time is first determined from the initial calibration and found to be 12.69 minutes. The retention time window for this pesticide is ±0.05 minutes. Therefore, the calculated retention time window would range from 12.64 to 12.74 minutes.

d.   The relative percent difference (RPD) between the calculated amount and the true amount for each of the single component pesticides and surrogates in the PEM analyses must be less than or equal to 25.0 percent.

e.   The percent breakdown is the amount of decomposition that 4,4'-DDT and Endrin undergo when analyzed on the GC column. For Endrin, the percent breakdown is determined by the presence of Endrin aldehyde and/or Endrin ketone in the GC chromatogram. For 4,4'-DDT, the percent breakdown is determined from the presence of 4,4'-DDD and/or 4,4'-DDE in the GC chromatogram. The equations used to verify these calculations are provided in Attachment 1, Equations A and B.

   i.   The percent breakdown for both 4,4'-DDT and Endrin in each PEM must be less than or equal to 20.0 percent for both GC columns.

   ii.   The combined percent breakdown for 4,4'-DDT and Endrin in each PEM must be less than or equal to 30.0 percent for both GC columns.

3.3.2 Review Items

Form VI PEST-4, Form VII PEST-1 Form VIII PEST, and chromatograms.

3.3.3 Evaluation Procedure

1. Resolution Check Mixture

    a. Verify from the Form VIII PEST that the resolution check mixture was analyzed at the beginning of the initial calibration sequence on each GC column and instrument used for analysis.

    b. Check the resolution check mixture data and Form VI PEST-4 to verify that the resolution criterion between two adjacent peaks for the required compounds is less than or equal to 60 percent.

2. Performance Evaluation Mixture

    Verify from the Form III PEST that the Performance Evaluation Mixture (PEM) was analyzed at the proper frequency and position sequence.

    a. Check the PEM data from the initial and continuing calibrations to verify that the resolution between adjacent peaks is 100 percent on both GC columns.

    b. Check the PEM data from the initial and continuing calibrations and Form VII PEST-1 to verify that the absolute retention times for the pesticides in each analysis are within the calculated retention time windows based on the mean RT from the three-point initial calibration using equations and examples found in Attachment 1, Table 1.

    d. Verify that the relative percent difference (RPD) between the calculated amount and the true amount for each of the pesticides and surrogates is less than or equal to 25.0 percent.

    e. Verify that the individual breakdowns for 4,4'-DDT and Endrin are less than or equal to 20.0 percent, and that the combined breakdown is less than or equal to 30.0 percent.

3.3.4 Action

1. Resolution Check Mixture: If resolution criteria are not met the quantitative results may not be accurate due to inadequate resolution. Detected target compounds that were not adequately resolved should be qualified with "J." Qualitative identifications may also be questionable if coelution exists. Nondetects with retention times in the region of coelution may not be valid, depending on the extent

of the problem. Professional judgment should be used to determine the need to qualify data as unusable (R).

2. Performance Evaluation Mixture Retention Times: Retention time windows are used in qualitative identification. If the retention times of the pesticides in the PEM do not fall within the retention time windows, the associated sample results should be carefully evaluated. All samples injected after the last in-control standard are potentially affected. It should be noted for TPO action if the PEM retention time criteria are grossly exceeded.

    a. For the affected samples, check to see if the sample chromatograms contain any peaks that are close to the expected retention time window of the pesticide of interest. If no peaks are present either within or close to the retention time window of the deviant target pesticide compound, then there is usually no effect on the data (i.e., nondetected values can be considered valid). Sample data that are potentially affected by standards not meeting the retention time windows should be noted in the data review narrative.

    b. If the affected sample chromatograms contain peaks which may be of concern (i.e., above the CRQL and either close to or within the expected retention time window of the analyte of interest), then the reviewer should determine the extent of the effect on the data and may choose to qualify detected target compounds "N" and nondetected target compounds "UJ." In some cases, additional effort by the reviewer may be necessary to determine if sample peaks represent the compounds of interest, for example:

        i. The reviewer can examine the data package for the presence of three or more standards containing the pesticide of interest that were run within a 72-hour period during which the sample was analyzed.

        ii. If three or more such standards are present, the mean and standard deviation of the retention time window can be reevaluated.

        iii. If all standards and matrix spikes fall within the revised window, the valid positive or negative sample results can be determined using this window.

        iv. The narrative should identify the additional efforts taken by the reviewer and the resultant impact on data usability. In addition, the support documentation should contain all calculations and comparisons generated by the reviewer.

c. If the reviewer cannot do anything with the data to resolve the problem of concern, all positive results and quantitation limits should be qualified "R."

3. Performance Evaluation Mixture Resolution: If PEM resolution criteria are not met then the quantitative results may not be accurate due to inadequate resolution. Positive sample results for compounds that were not adequately resolved should be qualified with "J." Qualitative identifications may be questionable if coelution exists. Nondetected target compounds that elute in the region of the coelution may not be valid depending on the extent of the coelution problem. Professional judgment should be used to qualify data as unusable (R).

4. If RPD criteria are not met, qualify all associated positive results generated during the analytical sequence with "J" and the sample quantitation limits for nondetected target compounds with "UJ."

5. 4,4'-DDT/Endrin Breakdown:
   a. If 4,4'-DDT breakdown is greater than 20.0 percent:

      i. Qualify all positive results for DDT as "L" biased low. If DDT was not detected, but DDD and DDE are detected, then qualify the quantitation limit for DDT as unusable (R).

      ii. Qualify positive results for DDD and/or DDE as presumptively present at an estimated quantity (N).

   b. If Endrin breakdown is greater than 20.0 percent:

      i. Qualify all positive results for Endrin with "J." If Endrin was not detected, but Endrin aldehyde and Endrin ketone are detected, then qualify the quantitation limit for Endrin as unusable (R).

      ii. Qualify positive results for Endrin ketone as presumptively present at an approximated quantity (N).

   c. If the combined 4,4'-DDT and Endrin breakdown is greater than 30.0 percent:

      i. Qualify all positive results for DDT and Endrin with "J." If Endrin was not detected, but Endrin aldehyde and Endrin ketone are detected, then qualify the quantitation limit for Endrin as unusable (R). If DDT was not detected, but DDD and DDE are detected, then qualify the quantitation limit for DDT as unusable (R).

ii. Qualify positive results for Endrin ketone as presumptively present at an estimated quantity (N). Qualify positive results for DDD and/or DDE as presumptively present at an approximated quantity (N).

6. Potential effects on the sample data resulting from the initial calibration problems should be noted in the data review narrative.

3.4 Initial Calibration

Compliance requirements for satisfactory initial calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for pesticide and Aroclor target compounds. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analytical sequence and of producing a linear calibration curve.

3.4.1 Acceptance Criteria

1. Individual Standard Mixtures

   a. Individual Standard Mixtures A and B (containing all of the single component pesticides and surrogates) must be analyzed at low, midpoint, and high levels during the initial calibration, on each GC column and instrument used for analysis.

   b. The resolution between any two adjacent peaks in the midpoint concentration of Individual Standard Mixtures A and B in the initial calibration must be greater than or equal to 90.0 percent on both columns.

   c. The absolute retention times of each of the single component pesticides and surrogates are determined from three-point initial calibration using the Individual Standard Mixtures. A list of the retention time windows are included in Attachment 1, Table 1.

   d. At least one chromatogram from each of the Individual Standard Mixtures A and B must yield peaks that give recorder deflections between 50 to 100 percent of full scale.

   e. The concentrations of the low, medium, and high level standards containing all of the single component pesticides and surrogates (Individual Standard Mixtures A and B) must meet the following criteria on both GC columns.

      The low point corresponds to the CRQL for each analyte. The midpoint concentration must be four times the low point. The high point must be at least 16 times the low point, but a higher concentration may be chosen.

f. The Percent Relative Standard Deviation (%RSD) of the calibration factors for each of the single component pesticides and surrogates in the initial calibration on both columns for Individual Standard Mixtures A and B must be less than or equal to 20.0 percent, except as noted below. For the two surrogates, the %RSD must be less than or equal to 30.0 percent. Up to two single component target pesticides (other than the surrogates) per column may exceed the 20.0 percent limit but the %RSD must be less than or equal to 30.0 percent. Calculation is included in Attachment 1, Equation D.

Note: Either peak area or peak height may be used to calculate the calibration factors that are, in turn, used to calculate %RSD. However, the type of peak measurement used to calculate each calibration factor for a given compound must be consistent. For example, if peak area is used to calculate the low point calibration factor for endrin, then the mid- and high-point calibration factors for endrin must also be calculated using peak area.

2. Multi-Component Target Compounds

a. The multi-component target compounds (the 7 Aroclor and Toxaphene) must each be analyzed separately at a single concentration level during the initial calibration sequence. The analysis of the multi-component target compounds must also contain the pesticide surrogates.

b. For each multi-component analyte, the retention times are determined for three to five peaks. A retention time window of ±0.07 minutes is used to determine retention time windows for all multi-component analyte peaks.

c. Calibration factor data must be determined for each peak selected from the multi-component analysis.

3.4.2 Review Items

Form VI PEST-1, 2, 3, and 4, Form VII PEST-1, Form VIII PEST, and chromatograms.

3.4.3 Evaluation Procedures

1. Individual Standard Mixtures

a. Verify from the Form VIII PEST that the Individual Standard Mixtures A and B were analyzed at the proper frequency on each GC column and instrument used for analysis.

2. Multi-Component Target Compounds

   a. Verify from the Form VIII PEST that each of the multi-
       component target compounds were analyzed at the required
       frequency.

   b. Check the data for the multi-component target compounds and
       Form PEST VI-3 to verify that a least three peaks were used for
       calibration and that retention time and calibration factor data are
       available for each peak.

3.4.4 Action

1. If the initial calibration sequence was not followed as required, then professional
    judgment must be used to evaluate the effect of the non-compliance on the sample
    data. If the requirements for the initial calibration sequence were not met, then this
    should be noted for TPO action. If the non-compliance has a potential effect on the
    data, then the data should be qualified according to the professional judgment of the
    reviewer and this should be noted in the data review narrative.

2. If resolution criteria are not met, then the quantitative results may not be accurate
    due to peak overlap and lack of adequate resolution. Positive sample results for
    compounds that were not adequately resolved should be qualified with "J."
    Qualitative identifications may be questionable if coelution exists. Nondetected
    target compounds that elute in the region of coelution may not be valid depending
    on the extent of the coelution problem. Professional judgment should be used to
    qualify data as unusable (R).

3. If the %RSD linearity criteria are not met for the compound(s) being quantified,
    qualify all associated positive quantitative results with "J" and the sample
    quantitation limits for nondetected target compounds with "UJ."

4. Potential effects on the sample data due to problems with calibration should be
    noted in the data review narrative. If the data reviewer has knowledge that the
    laboratory has repeatedly failed to comply with the requirements for frequency,
    linearity, retention time, or resolution, the data reviewer should notify the TPO.

3.5. Continuing Calibration

Compliance requirements for satisfactory instrument calibration are established to ensure
that the instrument is capable of producing acceptable qualitative and quantitative data.

Continuing calibration checks and documents satisfactory performance of the instrument over specific time periods during sample analysis. To verify the calibration and evaluate instrument performance, continuing calibration is performed, consisting of the analyses of instrument blanks, the PEM, and the midpoint concentration of Individual Standard Mixtures A and B.

3.5.1 Acceptance Criteria

1. An instrument blank and the PEM must bracket one end of a 12-hour period during which samples are analyzed, and a second instrument blank and the midpoint concentration of Individual Standard Mixtures A and B must bracket the other end of the 12-hour period.

2. The resolution between any two adjacent peaks in the midpoint concentration of Individual Standard Mixtures A and B must be greater than or equal to 90.0 percent.

3. The absolute retention time for each single component pesticide and surrogate in the midpoint concentration of Individual Standard Mixtures A and B must be within the retention time windows determined from the initial calibration.

4. The RPD between the calculated amount and the true amount for each of the pesticides and surrogates in the midpoint concentration of the Individual Standard Mixtures A and B must not exceed 25.0 percent.

3.5.2 Review Items

Form VII PEST-1 and 2, Form VIII PEST, and chromatograms.

3.5.3 Evaluation Procedure

1. Check the Form VIII PEST to verify that the instrument blanks, PEMs, and Individual Standard Mixtures were analyzed at the proper frequency and that no more than 12 hours was elapsed between continuing calibration brackets in an ongoing analytical sequence.

2. Check the data for the midpoint concentration of Individual Standard Mixtures A and B to verify that the resolution between any two adjacent peaks is greater than or equal to 90.0 percent.

3. Check the data for each of the single component pesticides and surrogates in the midpoint concentration of Individual Standard Mixtures A and B and Form VII

PEST-2 to verify that the absolute retention times are within the appropriate retention time windows.

4. Check the data from the midpoint concentration of Individual Standard Mixtures A and B and Form VII PEST-2 to verify that the RPD between the calculated amount and the true amount for each of the pesticides and surrogates is less than or equal to 25.0 percent.

3.5.4 Action

1. If the continuing calibration sequence was not followed as required, then professional judgment must be used to evaluate the effect of the noncompliance on the sample data. If the requirements for the continuing calibration sequence were not met, then this should be noted for TPO action. If the noncompliance has a potential effect on the data, then the data should be qualified according to the professional judgment of the reviewer and this should be noted in the data review narrative.

2. If resolution criteria are not met than the quantitative results may not be accurate due to inadequate resolution. Positive sample results for compounds that were not adequately resolved should be qualified with "J." Qualitative identification may be questionable if coelution exists. Nondetected target compounds that elute in the region of coelution may not be valid depending on the extent of the coelution problem. Professional judgment should be used to qualify data as unusable (R).

3. Retention time windows are used in qualitative identification. If the standards do not fall within the retention time windows, the associated sample results should be carefully evaluated. All samples injected after the last in-control standard are potentially effected.

    a. For the affected samples, check to see if the sample chromatograms contain any peaks that are close to the expected retention time window of the pesticide of interest. If no peaks are present either within or close to the retention time window of the deviant target pesticide compound, then nondetected values can be considered valid. Sample data that is potentially affected by the standards not meeting the retention time windows should be noted in the data review narrative. If the retention time window criteria are grossly exceeded, then this should be noted for TPO action.

    b. If the affected sample chromatograms contain peaks which may be of concern (i.e., above the CRQL and either close to or within the expected retention time window of the pesticide of interest), then the reviewer should

follow the guidelines provided in Section 3.3 to determine the extent of the effect on the data.

4. If the RPD is greater than 25 percent for the compound(s) being quantified, qualify all associated positive quantitative results with "J" and the sample quantitation limits for nondetects with "UJ."

5. Potential effects on the sample data due to problems with calibration should be noted in the data review narrative. If the data reviewer has knowledge that the laboratory has repeatedly failed to comply with the requirements for frequency, linearity, retention time, resolution, or DDT/Endrin breakdown, the data reviewer should notify the TPO.

3.6 Blanks

The purpose of laboratory (or field) blank analyses is to determine the existence and magnitude of contamination problems resulting from laboratory (or field) activities. The criteria for evaluation of laboratory blanks apply to any blank associated with the samples (e.g., method blanks, instrument blanks, and sulfur cleanup blanks). If problems with any blank exist, all associated data must be carefully evaluated to determine whether or not there is an inherent variability in the data, or if the problem is an isolated occurrence not affecting other data.

3.6.1 Acceptance Criteria

1. No contaminants should be present in the blanks.

2. Frequency:

   a. Method Blanks—A method blank analysis must be performed for each 20 samples of similar matrix in each sample delivery group (SDG) or whenever a sample extraction procedure is performed.

   b. Instrument Blanks—An acceptable instrument blank must be run at least once every 12 hours and immediately prior to the analysis of either the performance evaluation mixture or Individual Standard Mixtures A and B, depending on the place in the analysis sequence.

   c. Sulfur Cleanup Blanks—A sulfur cleanup blank must be analyzed whenever part of a set of samples extracted together requires sulfur cleanup. If the entire set of samples associated with a method blank requires sulfur cleanup, then the method blank also serves the purpose of a sulfur blank and no separate sulfur blank is required.

3.6.2 Review Items

Form I PEST, Form IV PEST, and chromatograms.

3.6.3 Evaluation Procedure

1. Review the results of all associated blanks, Form I PEST, Form IV PEST, and chromatograms to evaluate the presence of TCL pesticides.

2. Verify that method blank analysis has been reported per SDG, per matrix, per concentration level, for each GC system used to analyze samples, and for each extraction batch.

3. Verify that the method blank analyses do not contain any target pesticide or Aroclor/Toxaphene at greater than its Contract Required Quantitation Limits (CRQL).

4. For the surrogates in each method blank, verify that the observed retention times are within the appropriate retention time windows calculated from the initial calibration.

5. Verify that the instrument blank analysis has been performed every 12 hours as part of the continuing calibration and following a sample analysis which contains an analyte(s) at high concentration(s), and that the instrument blanks do not contain any target analytes above one-half the CRQL, assuming that the material in the instrument resulted from the extraction of a 1-L water sample.

6. Verify that the sulfur cleanup blanks were analyzed at the required frequency and that they do not contain any target compound above the CRQL, assuming that the material in the instrument resulted from the extraction of a 1-L water sample. If a separate sulfur cleanup blank was prepared, one version of Form IV PEST should be completed associating all the samples with the method blank, and a second version of Form IV PEST should be completed listing only those samples associated with the separate sulfur cleanup blank.

3.6.4 Action

If the appropriate blanks were not analyzed with the frequency described in Criteria 3, 4, and 5, then the data reviewer should use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. The situation should be brought to the attention of the TPO.

Action in the case of unsuitable blank results depends on the circumstances and the origin of the blank. Detected compound results should be reported and qualified "B" if the concentration of the compound in the sample is less than or equal to 5 times (5x) the amount in the blank. In instances where more than one blank is associated with a given sample, qualification should be based on a comparison with the associated blank having the highest concentration of a contaminant. The results must not be corrected by subtracting the blank value.

Specific actions are as follows:

1. If a target pesticide or Aroclor/Toxaphene is found in the blank but not found in the sample(s), no qualification is required. If the contaminant(s) is found at level(s) significantly greater than the CRQL, then this should be noted in the data review narrative.

2. Any pesticide or Aroclor/Toxaphene detected in the sample, that was also detected in any associated blank, is qualified if the sample concentration is less than five times (5x) the blank concentration. The quantitation limit may also be elevated. Typically, the sample CRQL is elevated to the concentration found in the sample. The reviewer should use professional judgment to determine if further elevation of the CRQL is required.

The reviewer should note that analyte concentrations calculated for method blank may not involve the same weights, volumes, or dilution factors as the associated samples. These factors must be taken into consideration when applying the "5x" criteria, such that a comparison of the total amount of contamination is actually made.

In addition, there may be instances when little or no contamination was present in the associated blanks, but qualification of the sample was deemed necessary. Contamination introduced through dilution is one example. Although it is not always possible to determine, instances of this occurring can be detected when contaminants are found in the diluted sample result, but absent in the undiluted sample result. Since both results are not routinely reported, it may be possible to verify this source of contamination. However, if the reviewer determines that the contamination is from a source other than the sample, he/she should qualify the data. In this case, the "5x" rule does not apply; the sample value should be reported and qualified "B" and a note should be added to the narrative.

3. If gross contamination exists (i.e., saturated peaks), all affected compounds in the associated samples should be qualified as unusable (R), due to interference. This should be noted in the data review narrative if the contamination is suspected of having an effect on the sample results.

4.   If inordinate amounts of other target pesticides or Aroclors/Toxaphene are found at low levels in the blank(s), it may be indicative of a problem at the laboratory and should be noted in the data review narrative.

5.   If an instrument blank was not analyzed following a sample analysis which contained an analyte(s) at high concentration(s), sample analysis results after the high concentration sample must be evaluated for carryover. Professional judgment should be used to determine if instrument cross-contamination has effected any positive compound identification(s), and if so, detected compound results should be qualified. If instrument cross-contamination is suggested, then this should be noted in the data review narrative if the cross-contamination is suspected of having an effect on the sample results.

The following are examples of applying the blank qualification guidelines. Certain circumstances may warrant deviations from these guidelines:

Example 1:   Sample result is greater than the CRQL, but is less than the 5x multiple of the blank result.

	5x
Blank Result	1.0
CRQL	0.5
Sample Result	4.0
Qualified Sample Result	4.0B

In this case, sample results less than 5.0 (or 5 x 1.0) would be qualified as nondetected target compounds.

Example 2:   Sample result is less than the CRQL, and is also less than the 5x multiple of the blank result.

	5x
Blank Result	1.0
CRQL	0.5
Sample Result	0.4J
Qualified Sample Result	0.4B

Example 3:   Sample result is greater than the 5x multiple of the blank result.

	5x
Blank Result	1.0
CRQL	0.5
Sample Result	10.0
Qualified Sample Result	10.0

In this case, the sample result exceeded the adjusted blank result (5x 1.0) and the sample result is not qualified.

3.7 Surrogate Spikes

Laboratory performance on individual samples is established by means of spiking samples prior to extraction and analysis to determine surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample extraction. The evaluation of the recovery results of these surrogate spikes is not necessarily straightforward. The sample itself may produce effects due to such factors as interferences and high concentrations of target and/or nontarget analytes. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the evaluation and review of data based on specific sample results is frequently subjective and demands analytical experience and professional judgment. Accordingly, this section consists primarily of guidelines, in some cases with several optional approaches suggested.

3.7.1 Acceptance Criteria

1.   Two surrogate spikes, tetrachloro-m-xylene and decachlorobiphenyl, are added to all samples, Individual Standard Mixtures, PEMs, blanks, and matrix spikes to measure their recovery in sample and blank matrices.

2.   The advisory limits for recovery of the surrogates tetrachloro-m-xylene (TCX) and decachlorobiphenyl (DCB) are 60 to 150 percent for both water and soil samples.

3.   The retention times of both of the surrogates in the PEM, Individual Standard Mixtures, and samples must be within the calculated retention time windows. TCX must be within ±0.05 minutes, and DCB must be within ±0.10 minutes of the mean retention time determined from the initial calibration.

3.7.2 Review Items

Form II PEST, Form VIII PEST, and chromatograms.

3.7.3 Evaluation Procedures

1. Verify that the recoveries on the Surrogate Recovery Form II PEST are accurate and within the advisory limits and that the retention times on the Pesticide Analytical Sequence Form VIII PEST are accurate and within the retention time limits.

2. If low surrogate recoveries are observed, the reviewer should investigate whether the low recoveries were a result of sample dilution.

3. In the special case of a blank analysis with surrogates out of specification, the reviewer must give special consideration to the validity of associated sample data. The basic concern is whether the blank problems represent an isolated problem with the blank alone, or whether there is a fundamental problem with the analytical process. For example, if one or more samples in the batch show acceptable surrogate recoveries, the reviewer may choose to consider the blank problem to be an isolated occurrence.

3.7.4 Action

1. If surrogate spike recoveries are outside of advisory limits, the following guidance is suggested. Professional judgment must be used in applying these criteria.

    a. If low recoveries (i.e., between 10 and 60 percent) are obtained, f this may be an indication of a low bias in sample results and associated detected compound data should be qualified "L" and quantitation limits "UL."

    b. If high recoveries (i.e, greater than 150 percent) are obtained, this may be an indication of a high bias due to co-eluting interferences. Qualify associated detected compound data with "K", nondetected analytes do not require qualification.

    c. If either pesticide surrogate recovery is reported as between zero percent and 10 percent, the reviewer should examine the sample chromatogram to assess the qualitative validity of the analysis. If low surrogate recoveries are found to be due to sample dilution, then professional judgment should be used to determine if the resulting data should be qualified. If sample dilution is not a factor, then detected target compounds may be qualified "L" and nondetected target compound results should be qualified unusable (L).

    d. If zero pesticide surrogate recovery is reported, the reviewer should examine the sample chromatogram to determine if the surrogate may be present, but

slightly outside its retention time window. If this is the case, in addition to assessing surrogate recovery for quantitative bias, the overriding consideration is to investigate the qualitative validity of the analysis. If the surrogate is not present, qualify all nondetected target compounds as unusable (R).

2.   If surrogate retention times in PEMs, individual standards, and samples are outside of the retention time limits, qualification of the data is left up to the professional judgment of the reviewer. Refer to Section 3.3.4.2 for more guidance.

3.   Extreme or repeated analytical problems with surrogate recoveries should be noted for TPO action.

4.   Potential effects of the data resulting from surrogate recoveries not meeting the advisory limits should be noted in the data review narrative.

3.8 Matrix Spikes/Matrix Spike Duplicates

Data for matrix spikes (MS) and matrix spike duplicates (MSD) are generated to determine long-term precision and accuracy of the analytical method on various matrices. These data alone cannot be used to evaluate the precision and accuracy of individual samples. However, when exercising professional judgment, MS/MSD data should be used in conjunction with information on other deficiencies.

3.8.1 Acceptance Criteria

1.   Matrix spikes (MS) and matrix spike duplicate (MSD) samples are analyzed at a frequency of at lest one MS and MSD per 20 samples of each matrix.

2.   Matrix spike recoveries should be within the advisory limits provided on Form III PEST-1 and PEST-2 and in Attachment 1, Table 2.

3.   Relative percent difference (RPD) between MS and MSD recoveries must be within the advisory limits provided on Form III PEST-1 and PEST-2 and in Attachment 1, Table 2.

3.8.2 Review Items

Form III PEST-1 and PEST-2, and chromatograms

3.8.3 Evaluation Procedures

1.   Verify that MS and MSD samples were analyzed at the required frequency and that results are provided for each sample matrix.

2.   Inspect results for the MS/MSD Recovery on Form III PEST-1 and PEST-2 and verify that the results for recovery and RPD are within the advisory limits.

4.   Check that the matrix spike recoveries and RPD were calculated correctly.

5.   Compare %RSD results of nonspiked compounds between the original result, MS, and MSD.

3.8.4 Action

1.   No action is taken on MS/MSD data alone. However, using informed professional judgment the data reviewer may use the MS and MSD results in conjunction with other QC criteria and determine the need for some qualification of the data.

2.   The data reviewer should first try to determine to what extent the results of the MS/MSD affect the associated sample data. This determination should be made with regard to the MS/MSD sample itself, as well as specific analytes for all sample associated with the MS/MSD.

3.   In those instances where it can be determined that the results of the MS/MSD affect only the sample spiked, then qualification should be limited to this sample alone. However, it may be determined through the MS/MSD results that a laboratory is having a systematic problem in the analysis of one or more analytes, which affects all associated samples. For example, if the recoveries for MS and MSD are consistently low for both water and soil samples, this could be indicative of a systematic problem in the laboratory and recoveries should be examined in all associated samples.

4.   The reviewer must use professional judgment to determine the need for qualification of positive results of nonspiked compounds.

    NOTE: If a field blank was used for the MS/MSD, a statement to that effect must be included for the TPO.

3.9 Pesticide Cleanup Checks

Pesticide cleanup procedures are utilized to remove matrix interferences from sample extracts prior to analysis. The use of the Florisil cartridge cleanup procedure significantly reduces matrix interferences caused by polar compounds. Gel permeation chromatography (GPC) is used to remove high molecular weight contaminants that can interfere with the analysis of target analytes. Pesticide cleanup procedures are checked by spiking the

cleanup columns and cartridges, and verifying the recovery of pesticides through the cleanup procedure.

3.9.1 Acceptance Criteria

1. Florisil Cartridge Cleanup

   a. Florisil cartridges must be used for the cleanup of all sample extracts.

   b. Every lot number of Florisil cartridges used for sample cleanup must be checked by spiking with 2,4,5-trichlorophenol and the midpoint concentration of Individual Standard Mixture A.

   c. The lot of Florisil cartridges is acceptable if the recoveries for all of the pesticides and surrogates in Individual Standard Mixture A are within 80 to 120 percent, if the recovery of 2,4,5-trichlorophenol is less than 5 percent, and if no peaks interfering with the target analytes are detected.

2. Gel Permeation Chromatography (GPC)

   a. GPC is used for the cleanup of all soil sample extracts and for water sample extracts that contain high molecular weight components that interfere with the analysis of the target analytes.

   b. At least once every 7 days, the calibration of the GPC units must be checked by spiking with two check mixtures: the matrix spiking solution and a mixture of 0.2 ug/ml Aroclors 1016 and 1260.

   c. The GPC calibration is acceptable if the recovery of the pesticides in the matrix spiking solution are within 80 to 110 percent, and the Aroclor patterns should match those generated for previously run standards.

   d. A GPC blank must be analyzed after each GPC calibration and is acceptable if the blank does not exceed one-half the CRQL for any target analytes.

3.9.2 Review Items

Form IX PEST-1 and 2 and chromatograms

3.9.3 Evaluation Procedure

1. Florisil Cartridge Check

Check the data from the Florisil cartridge solution analyses and the Form IX PEST-1 and recalculate some of the percent recoveries to verify that the percent recoveries of the pesticides and surrogates in Individual Standard Mixture A are within 80 to 120 percent, the recovery of 2,4,5-trichlorophenol is less than 5 percent, and no interfering peaks are present. Compare the raw data to the reported results and verify that no calculation or transcription errors have occurred.

2. Gel Permeation Chromatography (GPC)

Check the data from the GPC calibration check analyses and the Form IX PEST-2 and recalculate some of the percent recoveries to verify that the percent recoveries of the pesticides in the matrix spike solution are within 80 to 110 percent and that the Aroclor patterns are similar to those of previous standards. Check to make sure that no transcription errors have occurred.

3.9.4 Action

1. If Florisil Cartridge Check criteria are not met, the raw data should be examined for the presence of polar interferences and professional judgment should be used to qualifying the data. If a laboratory chooses to analyze samples under an unacceptable Florisil Cartridge Check, then the TPO should be notified.

2. If Gel Permeation Criteria are not met, the raw data should be examined for the presence of high molecular weight contaminants and professional judgment should be used in qualifying the data. If a laboratory chooses to analyze samples under unacceptable Gel Permeation Criteria, then the TPO should be notified.

3. If zero recovery was obtained for the pesticide compounds and surrogates during either check, then the nondetected target compounds may be suspect and the data may be qualified unusable (R).

4. If high recoveries (i.e, greater than 120 percent) were obtained for the pesticides and surrogates during either check, use professional judgment to qualify detected target compounds as biased high (K). Nondetected target compounds do not require qualification.

5. Potential effects on the sample data result from the pesticide cleanup analyses not yielding acceptable results should be noted in the data review narrative.

3.10 Reported CRQLS

The objective is to ensure that contract required quantitation limits (CRQLs) are accurate.

3.10.1 Acceptance Criteria

The adjustment of the CRQL, must be calculated according to the equations provided in Attachment 1, Equations N and O.

3.10.2 Review Items

Form 1 PEST, Form X PEST-1 and PEST-2, and chromatograms.

3.10.3 Evaluation Procedure

1.   Verify that the CRQLs have been adjusted to reflect all sample dilution, concentrations, splits, cleanup activities, and dry weight factors that are not accounted for by the method.

3.10.4 Action

1.   If there are any discrepancies found, the laboratory may be contacted by the designated representative to obtain additional information that could resolve any differences. If a discrepancy remains unresolved, the reviewer must decide which value is the best value. Under these circumstances, the reviewer may determine if qualification of the data is warranted. A description of the reasons for data qualification and the qualification that is applied to the data should be documented in the data review narrative.

Attachment 1 to Appendix C-2
Multi-Media, Multi-Concentration
Contractual Requirements and Equations
for Pesticide Data Review

GC/ECD Instrument Performance Check

Check the Performance Evaluation Mixture calculations using equations II.2, II.3, and II.4 to ensure correct calculation of DT and Endrin breakdown. The breakdown of DDT and Endrin in both of the PEM injections must be less than 20.0 percent, and the combined breakdown of DDT and Endrin must be less than 30.0 percent.

$$\% \; Breakdown \; DDT = \frac{Amount \; found \; in \; ng \; (DDD+DDE) \; x \; 100}{Amount \; in \; ng \; of \; DDT \; injected} \qquad (A)$$

$$\% \; Breakdown \; Endrin =$$

$$\frac{Amount \; found \; in \; ng \; (Endrin \; aldehyde \; + \; Endrin \; ketone) \times 100}{Amount \; of \; Endrin \; injected \; in \; ng} \qquad (B)$$

$$Combined \; \% \; Breakdown \; = \; \% \; Breakdown \; DDT \; + \; \% \; Breakdown \; Endrin \qquad (C)$$

All peaks in both the injections of the Performance Evaluation Mixture must be 100 percent resolved on both columns. The relative percent difference of the calculated amount and the true amount for each of the single component pesticides and surrogates in the PEMs must be less than or equal to 25.0 percent using equation D.

$$RPD = \frac{|C_{nom} - C_{calc}|}{C_{nom}} \times 100 \qquad (D)$$

Where:

C_{nom} = True concentration of each analyte

C_{calc} = Calculated concentration of each analyte from the analysis of the standard

Initial Calibration

Retention time windows for each analyte and surrogate are calculated using Table 1. Windows are centered around the mean absolute retention time for the analyte established during the initial calibration. For example, for a given pesticide the mean retention time is first determined from the initial calibration and found to be 12.69 minutes. The retention time window for this pesticide is ± 0.05 minutes. Therefore, the calculated retention time window would range from 12.64 to 12.74 minutes.

Table 1 RETENTION TIME WINDOWS FOR PESTICIDE TARGET COMPOUNDS	
Pesticide Compounds	**Retention Time Windows in Minutes**
alpha-BHC	± 005
beta-BHC	± 0.05
gamma-BHC	± 0.05
delta-BHC	± 0.05
Heptachlor	± 0.05
Aldrin	± 0.05
alpha-Chlordane	± 0.07
gamma-Chlordane	± 0.07
Heptachlor epoxide	± 0.07
Dieldrin	± 0.07
Endrin	± 0.07
Endrin aldehyde	± 0.07
Endrin ketone	± 0.07
DDD	± 0.07
DDE	± 0.07
DDT	± 0.07
Endosulfan I	± 0.07
Endosulfan II	± 0.07
Endosulfan sulfate	± 0.07
Methoxychlor	± 0.07
Aroclors	± 0.07
Toxaphene	± 0.07
Tetrachloro-m-xylene	± 0.05
Decachlorobiphenyl	± 0.10

The %RSD of the calibration factors for each single component target compound must be less than or equal to 20.0 percent. The %RSD for the two surrogates must be less than or equal to 30.0 percent. Up to two single component target compounds per column may exceed the 20.0

$$RPD = \frac{|C_{nom} - C_{calc}|}{C_{nom}} \times 100 \qquad (I)$$

Where:

C_{nom} = True concentration of each analyte

C_{calc} = Calculated concentration of each analyte from the analysis of the standard

Surrogate Spikes

The advisory limits for recovery of tetrachloro-m-xylene (TCX) and decachlorobiphenyl (DCB) are 60 to 150 percent for both water and soil samples. The surrogate percent recovery is calculated using equation J. The retention times of both surrogates must be within the calculated retention time windows, i.e., TCX must be within ±0.05 minutes of the mean retention time determined from the initial calibration and DCB must be within ±0.10 minutes of the mean retention time determined from the initial calibration.

$$Surrogate\ Percent\ Recovery = \frac{Q_d}{Q_a} \times 100 \qquad (J)$$

Where:

Q_d = Quantity determined by analysis

Q_a = Quantity added to sample/blank

Matrix Spikes/Matrix Spike Duplicate

The matrix spike/matrix spike duplicate recovery and RPD requirements are listed in Table 2. The matrix spike recoveries and RPD are calculated using equations K and L.

Table 2 MS/MSD CONTRACTUAL REQUIREMENTS				
Compound	% Recovery Water	RPD Water	% Recovery Soil	RPD Soil
gamma-BHC (Lindane)	56-123	15	46-127	50
Heptachlor	40-131	20	35-130	31
Aldrin	40-120	22	34-132	43
Dieldrin	52-126	18	31-134	38
Endrin	56-121	21	42-139	45
4,4'-DDT	38-127	27	23-134	50

percent limit for %RSD, but those compounds must have a %RSD of less than or equal to 30.0 percent. Calibration factors are calculated using equations G and H and the %RSD is calculated using equations E and F.

$$\%RSD = \frac{StandardDeviation}{Mean} \; x \; 100 \tag{E}$$

where:

$$Standard\ Deviation\ -\ \left|\frac{\sum\limits_{i=1}^{n} (x_i - \bar{x})^2}{n-1}\right|^{1/2} \tag{F}$$

Where:

x_i = each individual value used to calculate the mean
x = the mean of n values
n = the total number of values

$$CF = \frac{Peak\ Area\ (\sqrt{}\ Height)\ of\ the\ Standard}{Mass\ injected\ (ng)} \tag{G}$$

$$\overline{CF} = \sum\limits_{i=1}^{n} \frac{CF_i}{n} \tag{H}$$

Where:

CF	=	Mean calibration factor of n values
CF_i	=	i^{th} calibration factor
n	=	Total number of values

Continuing Calibration

The retention time (RT) for each target compound and surrogate must be within RT window as calculated above using the mean absolute RT established during the three-point initial calibration. The relative percent difference of the calculated amount and the true amount for each of the compounds in the mid-point concentration of the Individual Standard Mixtures must be less than or equal to 25.0 percent using equation I.

$$Spike\ Recovery = \frac{SSR - SR}{SA} \times 100 \qquad\qquad (K)$$

Where:

SSR = Spike sample result
SR = Sample result
SA = Spike added

$$RPD = \frac{|MSR - MSDR|}{1/2\ (MSR + MSDR)} \times 100 \qquad\qquad (L)$$

Where:

RPD = Relative percent difference
MSR = Matrix spike recovery
MSDR = Matrix spike duplicate recovery

The vertical bars in the formula above indicate the absolute value of the difference, hence RPD is always expressed as a positive value.

Pesticide Cleanup Check

Every lot number of Florisil cartridges used for sample cleanup must be checked by spiking with 2,4,5-trichlorophenol and the midpoint concentration of Individual Standard Mixture A. The recoveries for all of the pesticides and surrogates in Individual Standard Mixture A must be within 80 to 120 percent, the recovery off 2,4,5-trichlorophenol must be less than 5 percent, and no peaks must interfere with the target analytes. Percent recovery is determined using equation M.

$$Percent\ Recovery = \frac{Q_d}{Q_a} \times 100 \qquad\qquad (M)$$

Where:

Q_d = Quantity determined by analysis
Q_a = Quantity added to sample/blank

The gel permeation chromatography (GPC) apparatus must be calibrated every 7 days. The calibration is acceptable if the recovery of each single component analyte is within 80 to 110 percent and the Aroclor patterns match patterns previously generated by standards.

Reported CRQLs

The CRQL of the single component pesticides is calculated using equations N and O, as appropriate.

CRQL for waters:

$$Sample\ CRQL = Reference\ CRQL\ x\ Df \qquad (N)$$

$$Df \quad = \quad Dilution\ factor$$

CRQL for soils/sediments (dry weight basis):

$$Sample\ CRQL = Reference\ CRQL\ x\ \frac{Df}{D} \qquad (O)$$

$$D \quad = \quad \frac{100 - \%\ moisture}{100}$$

Appendix D
Validation of Inorganic Analyte Data

- Level IM-1

VALIDATION OF TARGET ANALYTE LIST METALS AND CYANIDE DATA MANUAL APPROACH IM-1

1. PURPOSE AND APPLICABILITY

This procedure provides instructions to manually validate the target analyte list (TAL) metals and cyanide data using a manual innovative data validation approach that is based on the EPA's National Functional Guidelines for Data Review and EPA Region III's Modifications to the National Functional Guidelines for Data Review. Specifically, the approach is based on the use of quality control (QC) information contained on the laboratory QC summary forms, and does not utilize the raw data. The information that is obtained from the QC summary forms is indicated on Table IM-1-INORG-QC. This procedure is applicable to the TAL metal and cyanide data obtained using the Contract Laboratory Program Statement of Work (CLP SOW).

Data validated using this procedure are considered usable for the following types of purposes; however, the data users must decide on a case-by-case basis whether the procedure is suitable for their intended data uses. The suggested data uses are:

- Oversight of activities led by other parties

- Comparison to action levels

- Initial site investigation

- Contamination sources

- Nature and extent of contamination

- Preliminary risk assessment

- Risk assessment with known high levels of toxics

- Feasibility study

- Preliminary design

- Treatability study

- Initial cleanup verification

2. QUALITY CONTROL MEASURES CHECKED

Table IM-1-INORG-QC highlights the quality control (QC) indicators evaluated under this data validation procedure. These measures fall under one or more data validation steps highlighted below.

2.1 PROCEDURES

Data validation is performed primarily with respect to the technical data quality criteria; however, there are certain contractual criteria that may reflect on a participating laboratory's compliance with the terms and conditions of the program and future audits of the laboratory. The term "CCS" next to a validation step indicates that there is a contractual criteria in addition to the technical criteria. It is incumbent upon a data validator to point out the contractual deficiencies to the laboratory's CLP Technical Project Officer (TPO) for clarifications and corrective action. The data validation steps are as follows:

- Action Level Notification

- Technical Holding Times (CCS - Contractual holding times only)

- Calibration

  - Initial (CCS)

  - Initial and Continuing Calibration Verification (CCS)

  - CRDL Standards

- Blanks

  - Initial Calibration Blank (CCS)

  - Continuing Calibration Blank (CCS)

  - Preparation Blank (CCS)

  - ICP Interference Check Sample (CCS)

  - Laboratory Control Sample (CCS)

  - Duplicate Sample (CCS)

Table IM-1-INORG-QC QC CHECKLIST FOR LEVEL IM-1 CLP RAS TARGET ANALYTE LIST INORGANICS		
QC MEASURES	IM-1	M2
Action Level Notification	X	X
Holding Times	X	X
Initial Calibration	X	X
Initial Calibration Verification	X	X
Continuing Calibration Verification	X	X
Instrument Blanks	X	X
Laboratory Blank	X	X
ICP Interference Check	X	X
Laboratory Control Sample	X	X
Duplicate Precision	X	X
Matrix Spike Recovery	X	X
Furnace QC (MSA)	X	X
ICP Serial Dilution	X	X
Field Duplicates	X	X
Reporting Limit Verification	X	X
Sample Paperwork	X	X
Raw Data		X

- Matrix Spike Sample (CCS)

- Graphite Furnace Atomic Absorption QC (CCS)

- Method of Standard Addition (MSA)

- ICP Serial Dilution (CCS)

- Field Duplicates (if included)

- Reporting Limit Verification (IDLs, linear range, dilution factors and moisture content)

- Sample Paperwork

2.2 REPORTING REQUIREMENTS

Several forms have been developed to assist in the performance evaluation and in keeping track of the data quality qualifiers. The first form, IM-1-INORG-HT, summarizes the holding times. The second form, IM-1-INORG-CAL allows documentation of the calibration and blank QC violations. The third form, IM-1-INORG-SPK is used for the spike recoveries, duplicate precision, and control sample analyses data. The fourth form, IM-1-INORG-QL is used for summarizing all qualifiers for the samples. The EPA Region III Inorganic Regional Data Assessment form is used by the data validator to summarize contractual deficiencies for the laboratory's CLP Technical Project Officer (TPO). All forms are appended at the back of this SOP.

A memorandum describing those elements that were outside of established QC criteria, the actions which were taken and the impact on data usability must be prepared with substantiating documentation. The report and the supporting documentation should include the following:

1. Hand annotated Form 1s with
   a. data validation qualifiers
   b. sample identification number
   c. sampling location

2. A narrative description with
   a. a statement that defines the level of the data review, i.e., IM-1
   b. major and minor problems associated with the analysis
   c. issues that may have affected detection limits

3. The following attachments
   a. list of data validation qualifiers
   b. documentation which includes forms that support the assigned data qualifiers. Samples affected by noncompliant quality control measures should be listed on these forms.
   c. chain of custody form(s)

GLOSSARY OF DATA QUALIFIER CODES (INORGANIC)

The codes described below are those recommended in the EPA's national Functional Guidelines and the Region III's modifications.

Codes Relating to Identification
(confidence concerning presence or absence of analytes):

U = Not detected. The associated number indicates approximate sample concentration necessary to be detected.

(NO CODE) = Confirmed identification.

B = Not detected substantially above the level reported in laboratory or field blanks.

R = Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.

Codes Relating to Quantitation
(can be used for both positive results and sample quantitation limits):

J = Analyte present. Reported value may not be accurate or precise.

K = Analyte present. Reported value may be biased high. Actual value is expected lower.

L = Analyte present. Reported value may be biased low. Actual value is expected to be higher.

UJ = Not detected, quantitation limit may be inaccurate or imprecise.

UL = Not detected, quantitation limit is probably higher.

Other Codes

Q = No analytical result.

* = Results reported from diluted analysis.

The following subsections describe for each of the QC indicators the acceptance criteria, location and retrieval of QC data, evaluation of the QC data, actions taken in the event the QC acceptance criteria are exceeded, and documentation of the QC violations in a standardized report form.

2.3 ACTION LEVEL NOTIFICATION

The purpose behind action level notification is to make the EPA Remedial Project Officer (RPM) or the Site Project Officer (SPO) aware of the potential human health risk at the site. In accordance with the Region III Hazardous Waste Division policy, the EPA RPM or SPO must be promptly notified of any contaminant exceeding the established action level or the 10-day health advisory limit. The data for contaminants exceeding the action levels must be validated as a top priority and reported to the RPM or SPO, as soon as possible. Validation of the rest of the data may then be completed within the normal time frame.

2.3.1 Acceptance Criteria

EPA's Office of Solid Waste and Emergency Response has established 10-day advisory limits and action levels for several organic compounds and elements of special health risk concerns based on the Safe Drinking Water Act. The target analytes and their 10-day health advisory limits are listed in Table IM-1-INORG-AL. The criteria for action level notification are as follows:

- The contaminant concentration must be equal to or above the established 10-day health advisory limits.

- Data for contaminants exceeding the action levels must be validated as a top priority.

- The following EPA personnel must be notified of the action level exceedances:

    - EPA RPM or SPO
    - EPA Section Chiefs

- The remaining data validation should be completed per normal procedures.

- Any special instructions from the Hazardous Waste Division should be followed.

- Records should be kept of the data review, action level notification and any follow up instructions and actions.

Table IM-1-INORG-AL TARGET ANALYTE LIST METALS AND ACTION LEVELS			
Compound	Action Level[a]	Compound	Action Level[a]
Arsenic	50	Cadmium	40
Chromium	1000	Lead	15[b]
Nickel	1000	Cyanide	200
[a] All units are ug/l.			
[b] Lead also has an action level of 500 mg/kg (ppmw) for soil samples.			

2.3.2 Data requirements and Retrieval of Data

All forms required to perform Level IM-1 validation, as detailed in the following sections are necessary for carrying out action level notification.

2.3.3 Evaluation Procedure

The evaluation process preceding action level notification will primarily consist of comparing the results on Form Is with the action levels presented in Table IM-1-INORG-AL. Following the identification of the contaminants exceeding the action levels, focused data validation should be performed using the criteria, and procedures described in the appropriate sections below.

2.3.4 Action

The action resulting from focused data validation will be the notification of action level exceedance to the personnel identified above in Section 2.3.1. Copies of Form Is can be used to highlight the contaminants above the action levels. The findings of the focused validation can be summarized in a memorandum, and the data qualifiers resulting from focused validation may be written on the Form Is. The marked up forms should state that they represent validation of only the contaminants exceeding the action levels, and not all data.

2.4 HOLDING TIMES

The primary objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample extraction and analysis. The secondary objective is also to verify compliance with the contractual extraction and analysis holding times from the verified time of sample receipt (VTSR) at the laboratory.

2.4.1 Acceptance Criteria

Technical requirements for sample holding times have only been established for water matrices. The following holding times (from the time of sample collection) and preservation requirements were established under 40 CFR 136 (Clean Water Act) and are found in Volume 49, Number 209 of the Federal Register, page 43260, issued on October 26, 1984.

- Metals: 6 months; preserved to pH < 2
- Mercury: 28 days; preserved to pH < 2
- Cyanide: 14 days; preserved to pH > 12

Contractual holding times have been established by the CLP for the water and soil/sediment samples. The times are counted from the time of sample receipt at the laboratory, and are as follows:

- Metals: 180 days
- Mercury: 26 days
- Cyanide: 12 days

Generally, the holding times are calculated using the dates only and not the times.

2.4.2 Data Requirements and Retrieval of Data

Forms 1, 13, 14, and EPA Traffic Reports and Sample Shipping Logs.

2.4.3 Evaluation Procedure

Technical holding times are established by comparing the sampling date on the EPA Sample Traffic Report with the dates of sample preparation/extraction and analysis found on the extraction and instrument run logs (Forms 13 and 14, respectively).

Technical Holding Time (days) = Analysis Date - Sampling Date

Contractual holding times are established by comparing the sample receipt date on Form Is with the sample preparation/extraction and analysis dates on the extraction and run logs (Forms 13 and 14, respectively).

Contractual Holding Time (days) = Analysis Date - Receipt Date

2.4.4 Action

The following actions are required only for the violations of the technical holding times. If contractual holding times are exceeded, then the TPO must be notified of the violation.

1. If 40 CFR 136 criteria for technical holding times and preservation are not met, qualify all results > Instrument Detection Limit (IDL) as biased low (L) and the results < IDL as estimated detection limits biased low (UL).

2. If holding times are exceeded by two times (2X) the criteria for mercury and cyanide, qualify the non-detected results (results < IDL) as unusable (R).

3. If there are gross violations of the holding times for the metals, use professional judgment to determine the reliability of the data. A low bias would be expected for significantly longer holding times, and the reviewer may reject the non-detected data (results < IDL) as unusable (R).

4. Although the technical holding times for soil samples have not been established, apply the water holding time criteria to the soil samples. If the soil sample data are qualified using the water holding time criteria, this must be clearly documented in the narrative report.

2.5 CALIBRATION

Requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration is still valid.

Standards at concentrations near the lower limit of detection are also required to be run to determine the linearity of the instrument.

2.5.1 Acceptance Criteria

1. Initial Calibration. Instruments must be calibrated daily and each time the instrument is set up. Specific requirements for each type of analysis are as follows:

    - ICP Analysis

        - A blank and at least one standard must be used in establishing the analytical curve.

- Atomic Absorption Analysis (AA)

  - A blank and at least three standards, one of which must be at the Contract Required Detection Limit (CRDL), must be used in establishing the analytical curve.

- Mercury Analysis

  - A blank and at least four standards must be used in establishing the analytical curve.
  - The correlation coefficient must be ≥ 0.995. (This is a technical criterion and not a contractual one.)

- Cyanide Analysis

  - A blank and at least three standards must be used in establishing the analytical curve.
  - The correlation coefficient must be ≥ 0.995. (This is a technical criterion and not a contractual one.)

2. Initial and Continuing Calibration Verification (ICV and CCV).

- Analysis results must fall within the control limits of 90 to 110 percent Recovery (%R) of the true value for all analytes except mercury and cyanide.

- Analysis results for mercury must fall within the control limits of 80 to 120%R.

- Analysis results for cyanide must fall within the control limits of 85 to 115%R.

3. CRDL Standards for ICP (CRI) and AA (CRA).

- A CRI must be run at a concentration of 2X CRDL, or 2X the IDL, whichever is greater, for each ICP analyte (except Al, Ba, Ca, Fe, Mg, Na and K) at the beginning and end of each sample run or a minimum of twice per 8 hours.

- A CRA must be run at a concentration equal to the CRDL, or the IDL, whichever is greater, at the beginning of each sample run.

- The CRDL standard recoveries should be between 90-110% of the true values.

2.5.2 Data Requirements and Retrieval of Data

Forms 1, 2A, 2B and 14.

2.5.3 Evaluation Procedure

1. Verify that the instrument was calibrated daily and each time the instrument was set up using the correct number of standards and blank.

2. Recalculate one or more of the ICV and CCV %R per type of analysis (ICP, GFAA, etc.) using the following equation and verify that the recalculated value agrees with the laboratory reported values on Form 2A. Due to possible rounding discrepancies, allow results to fall within 1 percent of the contract windows (e.g., 80 to 111 percent).

$$\%R = \frac{Found}{True} \times 100$$

where:

Found = Concentration (in ug/L) of each analyte measured in the analysis of the ICV or CCV solution

True = Concentration (in ug/L) of each analyte in the ICV or CCV source

3. Verify from the run log (Form 14) that the CRAs and CRIs were run at the required frequency.

4. Verify from Form 2B that the CRIs were at 2X CRDL, or 2X IDL, whichever was greater, and the CRAs were at the CRDL, or the IDL, whichever was greater.

2.5.4 Action

1. If the minimum number of standards as defined above were not used for initial calibration, use professional judgment in qualifying the data. However, if the instrument was not calibrated daily and each time it was set up, qualify the data as unusable (R). Document the noncompliance with the calibration requirements in the narrative and document on the EPA Region III Inorganic Regional Data Assessment Form.

2.   If the ICV or CCV %R falls outside the acceptance windowns, using the following guidelines for qualifying the data.

Percent Recovery (%R)			Qualifier	
Metals	Cyanide	Mercury	> IDL	< IDL
90-110	85-115	80-120	None	None
75-89	70-84	65-79	L	UL
>110	>115	>120	K	None
<75	<70	<65	L	R

- If the ICV or CCV %R falls outside the acceptance windows, but within the ranges of 75 to 89 percent (metals) or 70-84(CN) or 65-79(Hg), qualify results > IDL as biased low (L), and results < IDL as biased low (UL).

- If the ICV or CCV %R is >110 (metals) or >115 (CN) or >120 (Hg), results < IDL are acceptable. Results > IDL should be qualified as biased high (K).

- If the ICV or CCV %R is <75 percent (metals) or <70 percent (CN) or <65 percent (Hg), qualify all positive results as unusable (R).

3.   Please be advised, there are no National Functional Guidelines to qualify the data based on the CRDL analyses; however, EPA Region III has developed specific guidance to qualify the data.

- If the recovery for the CRI or CRA is >110% and the reported sample results are >IDL, but < 2X CRDL, qualify the data as biased high (K).

- If the recovery for the CRI or CRA is between 50-89%, qualify results > IDL, but < 2X CRDL as biased low. Qualify results < IDL as biased low (UL).

- If the recovery for an element is <50%, qualify the results > IDL, but < 2X CRDL as biased extremely low (L). Qualify results < IDL as unusable (R).

2.6 BLANKS

Blank results are assessed to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks applies to any blank associated with the samples. If problems with any blank exist, all data associated with the Case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the Case, or if the problem is an isolated occurrence not affecting other data.

2.6.1 Acceptance Criteria

No contaminants should be in the blank(s) at concentrations, > IDL.

2.6.2 Data Requirements and Retrieval of Data

Forms 1 and 3.

2.6.3 Evaluation Procedure

Review the results reported on the Blank Summary (Form 3) for all blanks. Note that the instrument blanks serve two purposes: One, to determine any sample carryover, and second, to determine the shift in the instrument baseline. It is common that the instrument baseline may shift upward or downward from the calibration reference point (zero concentration standard) during a sample run. This shift is not necessarily indicative of any carryover from the previous sample. Negative blank results are common indicating a shift in the baseline. Professional judgment should be used when qualifying data based on the instrument blanks.

2.6.4 Action

Action in the case of unsuitable blank results depends on the circumstances and origin of the blank.

- Sample results > IDL but < five times the amount in any blank should be qualified as (B).

- Any blank with a negative result whose absolute value is > CRDL must be carefully evaluated to determine its effect on the sample data.

- Qualify the field blanks in a manner similar to that for samples. A field blank may not be used to qualify another field blank.

When multiple field blanks are present in a SDG, use the highest detected concentration for each analyte in the field and laboratory blanks when qualifying the sample data.

Note: The blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. In particular, soil sample results reported on Form 1 will not be on the same basis (units, dilution) as the calibration blank data reported on Form 3.

2.7 ICP INTERFERENCE CHECK SAMPLE

The ICP Interference Check Sample (ICS) verifies the contract laboratory's interelement and background correction factors.

2.7.1 Acceptance Criteria

1.   An ICS must be run at the beginning and end of each sample analysis run (or a minimum of twice per 8-hour working shift, whichever is more frequent).

2.   Results for the ICS solution AB analysis must fall within the control limits of ± 20 percent of the true value.

2.7.2 Data Requirements and Retrieval of Data

Forms 1 and 4

2.7.3 Evaluation Procedure

1.   Verify at random the reported %Rs for the Solution AB using the true and found values. Use the following equation:

$$\text{ICS \%R} = \frac{\text{Found} \cdot \text{Solution AB}}{\text{True Solution AB}} \times 100$$

where:
   Found Solution AB   = Concentration (in μg/l) of each analyte measured in the analysis of solution AB

   True Solution AB   = Concentration (in μg/l) of each analyte in solution AB

2.   Check the results with an absolute value > IDL for those analytes which are not present in the ICS solution.

2.7.4 Action

1. For samples with concentrations of aluminum, calcium, iron, and magnesium which are comparable to or > their respective levels in the Interference Check Sample:

    - If the ICS recovery for an element is > 120 percent and the sample results are < IDL, this data is acceptable for use.

    - If the ICS recovery for an element is > 120 percent and the sample results are > IDL, qualify the affected data as biased high (K).

    - If the ICS recovery for an element falls between 50 and 79 percent and the sample results are > IDL, qualify the affected data as biased low (L).

    - If sample results are < IDL, and the ICS recovery for that analyte falls within the range of 50 to 79 percent, the possibility of false negatives may exist. Qualify the data for these samples as detection limits biased low (UL).

    - If ICS recovery results for an element is < 50 percent, qualify results > IDL as biased low (L), and results < IDL as unusable (R).

2. If results > IDL are observed for elements which are not present in the ICS solution, the possibility of false positives exists. An evaluation of the associated sample data for the affected elements should be made. For samples with comparable or higher levels of interferents and with analyte concentrations that approximate those levels found in the ICS (false positives), qualify sample results > IDL as biased high (K).

3. If negative results are observed for elements that are not present in the EPA ICS solutions, and their absolute value is > IDL, the possibility of false negatives in the samples may exist. If the absolute value of the negative results is > IDL, an evaluation of the associated sample data should be made. For samples with comparable or higher levels of interferents, qualify results for the affected analytes < IDL as biased low (UL), qualify results for the affected analytes > IDL as biased low (L).

4. In general, the sample data can be accepted if the concentrations of aluminum, calcium, iron, and magnesium in the sample are found to be < or equal to their respective concentrations in the ICS. If these elements are present at concentrations > the level in the ICS, or other elements are present at concentrations > the level in the ICS, or other elements are present in the sample at > 10 mg/L, the reviewer should investigate the possibility of other interference effects by using the Table found in the most recent version of the SOW. These analyte concentration

equivalents presented in the table should be considered only as estimated values, since the exact value of any analytical system is instrument specific. Therefore, estimate the concentration produced by an interfering element. If the estimate is >2X CRDL and also >10 percent of the reported concentration of the affected element, qualify the affected results as biased high (K).

2.8 LABORATORY CONTROL SAMPLE

The laboratory control sample (LCS) serves as a monitor of the overall performance of all steps in the analysis, including the sample preparation.

2.8.1 Acceptance Criteria

1. All aqueous LCS results must fall within the control limits of 80 to 120%R, except Sb and Ag which have no control limits.

2. All solid LCS results must fall within the control limits established by the EPA. This information is available from EMSL/LV.

2.8.2 Data Requirements and Retrieval of Data

Forms 1 and 7.

2.8.3 Evaluation Procedure

1. Review Form VII and verify that results fall within the control limits.

2. Randomly verify the reported recoveries on Form VII using the following equation:

$$LCS \ \%R = \frac{LCS \ Found}{LCS \ True} \times 100$$

where:
LCS Found = Concentration (in $\mu g/l$ for aqueous; mg/kg for solid) of each analyte measured in the analysis of LCS solution

LCS True = Concentration (in $\mu g/l$ for aqueous; mg/kg for solid) of each analyte in the LCS source

2.8.4 Action

1. Aqueous LCS:

- If the LCS recovery for any analyte falls within the range of 50 to 79 percent, qualify results > IDL as biased low (L). If the LCS recovery is > 120 percent, qualify results > IDL as biased high (K).

- If results are < IDL and the LCS recovery is > 120 percent, the data are acceptable.

- If results are < IDL and LCS recovery falls within the range of 50 to 79 percent, qualify the data for the affected analytes as biased low (UL).

- If LCS recovery results are < 50 percent, qualify results > IDL as biased low (L), and results < IDL as unusable (R).

2. Solid LCS:

- If the solid LCS recovery for any analyte is below the lower acceptable control limit, qualify all sample results > IDL as biased low (L). If the LCS recovery is above the upper control limit, qualify the results > IDL as biased high (K).

- If the LCS results are lower than the control limits, qualify all sample results < IDL as detection limits biased low (UL).

- If the LCS results are higher than the control limits and the sample results are < IDL, the data are acceptable.

2.9 DUPLICATE SAMPLE ANALYSIS

Duplicate analyses are used to determine the laboratory precision for each sample matrix.

2.9.1 Acceptance Criteria

1. Samples identified as field blanks cannot be used for duplicate sample analysis.

2. A control limit of ± 20 percent (35 percent for soil) for the Relative Percent Difference (RPD) shall be used for sample values >5X CRDL.

3. A control limit of $\pm$ CRDL ($\pm 2X$ CRDL for soil) shall be used for sample values < five times CRDL, including the case when only one of the duplicate sample values is < five times CRDL.

2.9.2 Data Requirements and Retrieval of Data

Forms 1 and 6.

2.9.3 Evaluation Procedure

1. Review Form 6 and verify that results fall within the control limits.

2. Recalculate one or more RPD using the following equation to verify that results have been correctly reported on Form 6.

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

where:

S = First sample value (original)
D = Second sample value (duplicate)

3. Verify that the field blank was not used for duplicate analysis.

2.9.4 Action

1. If duplicate analysis results for a particular analyte fall outside the appropriate control windows, qualify results > IDL, for that analyte in all associated samples of the same matrix as estimated (J), and results < IDL as estimated (UJ).

2. If the field blank was used for duplicate analysis, all other QC data must be carefully checked and professional judgement exercised when evaluating the data. Document this information on the EPA Region III Inorganic Regional Data Assessment Form.

2.10 MATRIX SPIKE SAMPLE ANALYSIS

The matrix spike sample analysis provides information about the effect of each sample matrix on the digestion and measurement methodology.

2.10.1 Acceptance Criteria

1. Samples identified as field blanks cannot be used for spiked sample analysis.

2. Spike recovery (%R) must be within the limits of 75 to 125 percent. However, spike recovery limits do not apply when sample concentration exceeds the spike concentration by a factor of four or more.

3. If the matrix spike recovery limits are not met and the sample concentration does not exceed 4X the spike added, a post-digestion spike must be performed for the ICP, CN and flame AA analyses. This criteria does not apply to silver. The post digestion spike must be performed at 2X the sample concentration or 2X CRDL, whichever is greater.

2.10.2 Data Requirements and Retrieval of Data

Forms 1, and 5A and 5B.

2.10.3 Evaluation Procedure

1. Review Form 5A and verify that results fall within the specified limits.

2. Recalculate one or more %R using the following equation to verify that results were correctly reported on Form 5A.

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

where:
SSR = Spiked sample result
SR = Sample result
SA = Spike added

3. Verify that the field blank was not used for spike analysis.

4. Verify that a post-digestion spike was performed when required, and at specified spike concentrations.

2.10.4 Action

1. If the spike recovery is > 125 percent and the reported sample results are < IDL, the data is acceptable for use.

2. If the spike recovery is > 125 percent and the sample results are > IDL, qualify the data for these samples as biased high (K).

3. If the spike recovery is <75 percent and the sample results are > IDL, qualify the data for these samples as biased low (L).

4. If the spike recovery falls within the range of 30 to 74 percent and the sample results are < IDL, qualify the data for these samples as detection limits biased low (UL).

5. If spike recovery results fall < 30 percent and the sample result are < IDL, qualify the data for these samples as unusable (R). If the results are > IDL, qualify the data as biased extremely low (L).

6. If the field blank was used for matrix spike analysis, all other QC data must be carefully checked and professional judgement exercised when evaluating the data. Report this information on the EPA Region III Inorganic Regional Data Assessment form.

7. If a post-digestion spike was not performed when required, or the recoveries fall outside the matrix spike recovery limits, document this in the narrative. (The post-digestion recovery data are not used for data qualification as acceptable recovery limits have not been established by the EPA yet.)

2.11 FURNACE ATOMIC ABSORPTION QC

Duplicate injections and post-digestion/analytical spikes are required for each element and sample analyzed by the furnace atomic absorption technique. Additionally, a reanalysis by the method of standard addition (MSA) is required for samples meeting certain conditions specified in the most recent version of the SOW. The post-digestion/analytical spike recoveries for the GFAA metals are reported on Form 14 under the %R column. The correlation coefficient (r) can be found on Form 8 for samples requiring MSA. The flag "+" is placed on Form 1s for samples and analytes with r <0.995.

2.11.1 Acceptance Criteria

1. The post-digestion/analytical spike recovery for the GFAA metals must be within 85-115%.

3. The correlation coefficient (r) for the samples requiring reanalysis by the MSA must be 0.995 or better using the ordinary least squares linear regression. (A formula for linear regression can be found in the SOW, or some computer software applications have the linear regression function built-in.)

2.11.2 Data Requirements and Retrieval of Data

Forms 1, 8 and 14.

2.11.3 Evaluation Procedure

1. Evaluate the %Rs from Form 14 for a comparison with the analytical spike recovery requirement of 85-115%.

2. Verify by random recalculation that the r values reported on Form 8 are accurate and equal to or greater than 0.995.

2.11.4 Action

1. If the analytical spike recovery is less than 85%, but greater than 40%, qualify results > IDL as biased low, (L), and results < IDL as biased low (UL).

2. If the analytical spike recovery is greater than 115%, qualify results > IDL as biased high (K), sample results < IDL are acceptable.

3. If the analytical spike recovery is less than 10% and the results are < IDL, the data should be qualified usable (R). Sample results > IDL should be qualified biased extrememly low (L).

4. If the MSA correlation coefficient is < 0.995, qualify the data as estimated (J).

2.12 ICP SERIAL DILUTION

The serial dilution determines whether significant physical or chemical interferences exist due to sample matrix.

2.12.1 Evaluation Criteria

If the analyte concentration is sufficiently high (concentration in the original sample is minimally a factor of 50 above the IDL), an analysis of a 5-fold dilution must agree within 10 percent Difference (%D) of the original results.

2.12.2 Data Requirements and Retrieval of Data

Forms 1 and 9.

2.12.3 Evaluation Procedure

1. Recalculate at random the %Ds using the following equation to verify the dilution analysis results reported on Form 9.

$$\%D = \frac{|I-S|}{I} \times 100$$

where:

I = Initial sample result
S = Serial dilution result (instrument reading X 5)

2. Determine whether there is negative interference. Generally, a diluted sample should offer lesser interference; hence, more accurate and higher reported values. However, it is also possible to obtain lower values for the diluted sample, indicating a potential for negative interference. The apparent negative interference may be related to sample concentrations in the less accurate regions of the ICP instrumentation i.e., near the IDL or the upper limit of the linear range. Carefully evaluate if the negative interference is real.

2.12.4 Action

1. When the diluted sample results are outside the range of 90-110% of the original sample result, qualify the associated data as estimated (J).

2. If evidence of negative interference is found, use professional judgement to qualify the data.

2.13 FIELD DUPLICATES

Field duplicate samples may be taken and analyzed as a indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which measure only laboratory performance. It is also expected that soil duplicate results will have greater variance than water matrices due to difficulties associated with collection identical field samples.

2.13.1 Acceptance Criteria

There are no review criteria for field duplicate analyses comparability.

2.13.2 Data Requirements and Retrieval of Data

Form 1.

2.13.3 Evaluation Procedure

Samples which are field duplicates should be identified using EPA Sample Traffic Reports or sample field sheets. The reviewer should compare the results reported for each sample and calculate the Relative Percent Difference (RPD), if appropriate.

2.13.4 Action

Any evaluation of the field duplicates should be provided with the narrative report. At the reviewer's discretion, a table listing the RPDs between the original and the duplicate samples may be prepared.

2.14 REPORTING LIMIT VERIFICATION

Reporting limit verification is performed to verify that the CRDLs were met; to ensure that the reported quantitation results were accurate, and to ensure that the ICP data were not reported beyond the upper linear range of the instrument.

The positive results themselves are not verified for accuracy, as this would require the use of raw data. However, the undetected values are checked for proper application of dilution factors and moisture content normalization. Indirectly, a check on the reporting limits would suggest that the sample results have been properly adjusted for the dilution factors and moisture content.

2.14.1 Acceptance Criteria

Minimally, the laboratory should meet the basic CRDLs specified in the most recent version of SOW. This implies that the laboratory's IDLs must be equal to or less than the CRDLs. The laboratory has a choice of using the ICP or AA instrumentation for analyses of metals; however, the sample results for As, Pb, Se and Tl must be greater than 5X IDL. Otherwise, AA instrumentation must be used.

ICP data must not be reported beyond the established linear range without sample dilution. The linear range for each ICP metal is established on a quarterly basis.

Furthermore, the dilution factor and moisture content correction must be made to the sample results, as applicable. This may apparently raise the reporting limit above the CRDLs for some analytes.

2.14.2 Data Requirements and Retrieval of Data

Forms 1, 10 and 12.

2.14.3 Evaluation Procedure

1.   Verify from Form 10 that the IDLs reported for each analyte and instrument meet the CRDLs at a minimum. There may be several Form 10s for the ICP, AA, Hg and CN analytes.

2.   Verify that the linear ranges for the ICP metals are reported on Form 12, and no undiluted sample data are reported above the linear ranges.

3.   Verify on the Form 1s that the CRDLs have been adjusted for any dilution factors and moisture content, as applicable.

4.   Verify that the sample results are >5X ICP IDL, if ICP analysis results are used for As, Tl, Se, or Pb.

2.14.4 Action

If there are any discrepancies found, the laboratory may be contacted by the designated representative to obtain additional information that could resolve any differences. If a discrepancy remains unresolved, the reviewer may determine qualification of the data is warranted.

2.15 SAMPLE PAPERWORK

The purpose for evaluating the sample paperwork is to determine that the samples being validated are indeed the ones taken from the site, and have not been tampered with. Accurate sample identity is of paramount importance in substantiating the sample data. Without unequivocal sample identity and chain-of-custody procedures, the sample data may not be defensible or enforceable.

Under the current CLP contracts, the original paperwork (i.e., the purge package or the administrative record) is included in the data package from the laboratory. It is assumed that the data validator is not privy to the original paperwork; therefore, the evaluation criteria and procedures described below apply only to the documents that are generally

included in the data validation package. These documents are the chain-of-custody forms and Region III Shipping Record.

2.15.1 Acceptance Criteria

Criteria for acceptability or authenticity of the sampling paperwork, document control and chain-of-custody have been established by the National Enforcement Investigations Center (NEIC), to support the CLP. Overall criteria are too numerous and subjective to be discussed here, but the criteria that apply to data validation are:

- The chain-of-custody form should be properly and completely filled out including the sampler signatures, date and time of sampling, sampling station identification, analyses requested, traffic numbers, tag numbers, etc. These data are minimally required to confirm the authenticity of the sample and its data.

- The chain-of-custody must be maintained at all times. The custody transfers between different parties must be signed and dated.

2.15.2 Data Requirements and Retrieval of Data

A copy of the chain-of-custody form is essential to confirm the identity of the samples. The Region III Shipping Record is required to identify the field QC samples. The chain-of-custody form and Shipping Record are generally located in front of the data package.

2.15.3 Evaluation Procedure

Ensure that the chain-of-custody form was signed and dated by the samplers, and a time and date were entered for sample collection. The laboratory copy of the chain-of-custody must have the signature of the laboratory sample custodian. Any errors on the form should have been crossed out with a single line through the entry. Verify that all collected samples have unique station identification, traffic numbers and sample tag numbers. Ensure that the Region III Shipping Record correctly reflects the information on the chain-of-custody.

2.15.4 Action

The action to be taken in qualifying the data is highly dependent on the nature of the problem. Some errors in paperwork are practically unavoidable in real situations. An effort should be made to reconcile the differences by cross checking the field notebooks against the sampling paperwork. Occasionally, the samplers may forget to sign the chain-of-custody; however, the field notebooks may amply describe the sampling event. Problems are also inevitable in noting or cross-referencing sample tag numbers and traffic

numbers. Generally, there are several alternate sources of information to substantiate or refute the problem.

Any discrepancies found in the paperwork must be immediately brought to the attention of the EPA RPM or SPO. Clearly define the problems in a memorandum to the responsible parties. Attach marked copies of the chain-of-custody forms to substantiate the findings.

Case No.:_____ _____
SDG No.:_____
Data Reviewer/Date:_____/_____
Site:_____

Procedure No.: 1M-1-INORG
Revision: 2
Date: 06/30/1995

	Sample		ICP (180 Days)			AA (180 Days)			Hg (28 Days)			CN (14 Days)		
Number	Date Coll.	Date Recd. Lab.	Date Prep. Anal.	Hold. Time, Days	Qual.	Date Prep. Anal.	Hold. Time, Days	Qual.	Date Prep. Anal.	Hold. Time, Days	Qual.	Date Prep. Anal.	Hold. Time, Days	Qual.
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Table 1M-1-INORG-HT Holding Time Summary

Table IM-1-INORG-CAL.   TAL Metals and Cyanide Calibration and Blank Qualifier Summary

ICP Instrument ID:
Instrument Calibrated? __Yes __No
ICP Date Started:

ICP Date Ended:

AA Instrument IDs: As_____ Pb___
Se_____ Tl_____
Instruments Calibrated?

AA Date Started:

AA Date Ended:

Sample Identifier:	Sample Identifier:
1.	11.
2.	12.
3.	13.
4.	14.
5.	15.
6.	16.
7.	17.
8.	18.
9.	19.
10.	20.

Analyte	(Criteria)	ICV 90-110	CCV 90-110	CRDL	Qual.	ICB <IDL	CCB <IDL	PB <IDL	Field Bl. <IDL	Qual.
Aluminum	Al									
Antimony	Sb									
Arsenic	As									
Barium	Ba									
Beryllium	Be									
Cadmium	Cd									
Calcium	Ca									
Chromium	Cr									
Cobalt	Co									
Copper	Cu									
Iron	Fe									
Lead	Pb									
Magnesium	Mg									
Manganese	Mn									
Mercury	Hg									
Nickel	Ni									
Potassium	K									
Selenium	Se									
Silver	Ag									
Sodium	Na									
Thallium	Tl									
Vanadium	V									
Zinc	Zn									
Cyanide	CN									

* The ICV and CCV criteria: Hg = 80-120% R, CN = 85-115% R.

Case No.:_____

SDG No.:_____

Data Reviewer/Date:_____ /_____

Site:_____

Procedure No.: IM-1-INORG

Revision: 2

Date: 06/30/1995

Table IM-1-INORG-SPK. TAL Metals and Cyanide Matrix Spike, Duplicate and ICP Serial Dilution Quality Control Summary

Analyte (Criteria):	Matrix Spike/Duplicate			ICP Ser. Dil.		Post Digest./Anal. Spike			LCS Recovery	
	Rec., %R	RPD				ICP	AA			
	75-125	25 (35)*	Quals.	+/-10%	Quals.	75-125%	85-115%	Quals.	80-120%*	Quals.
Aluminum Al										
Antimony Sb										
Arsenic As										
Barium Ba										
Beryllium Be										
Cadmium Cd										
Calcium Ca										
Chromium Cr										
Cobalt Co										
Copper Cu										
Iron Fe										
Lead Pb										
Magnesium Mg										
Manganese Mn										
Mercury Hg										
Nickel Ni										
Potassium K										
Selenium Se										
Silver Ag										
Sodium Na										
Thallium Tl										
Vanadium V										
Zinc Zn										
Cyanide CN										

Note: See the SOP for the exact criteria under special situations.

*There are no criteria at the present time to qualify the ICP metals or Cyanide based on the post-digestion spike recoveries.

* The precision RPD for soil samples is 35.

*The range of 80-120% R is used only for the aqueous LCSs. Solid LCS samples have specific recovery ranges for each analyte.

Case No.:_____ _____
SDG No.:_____
Data Reviewer/Date:_____ / _____
Site:_____

Procedure No.: IM-1-INORG
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Table IM-1-INORG-QL. Summary of Data Qualifiers

Sample Number	Analyte																							
	Al	Sb	As	Ba	Be	Cd	Ca	Cr	Co	Cu	Fe	Pb	Mg	Mn	Hg	Ni	K	Se	Ag	Na	Tl	V	Zn	CN

Table IM-1-INORG-IRDA
EPA Region III
Inorganic Regional Data Assessment

Case No. _____   Site _____

Laboratory _____   Number of Samples/Matrix _____

SDG No. _____   Reviewer (if not CRL) _____

SOW No. _____   Reviewer's Name _____

TPO: Action _____ FYI _____   Completion Date _____

Data Assessment Summary

	ICP	AA	Hg	Cyanide
Holding Times	_____	_____	_____	_____
Calibrations	_____	_____	_____	_____
Blanks	_____	_____	_____	_____
ICS	_____			
LCS	_____			
Duplicate Analysis	_____	_____	_____	_____
Matrix Spike	_____	_____	_____	
Post Digestion/Analytical Spike	_____	_____		
MSA		_____		
Serial Dilution	_____			
Field Duplicates	_____	_____	_____	_____
Reporting Limit Verification	_____	_____	_____	_____
Sample Paperwork	_____	_____	_____	_____

    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: _____

_____

_____