



VERIFICATION AND VALIDATION GUIDELINES

FOR

VOLATILE ORGANICS

DA-SS01-v1

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1. PURPOSE AND INTRODUCTION

This procedure presents those data assessment steps which are unique to PSA Module SS01, Volatile Organics. This procedure is to be used in conjunction with the general guideline for data verification and validation, DA-GR01.

The purpose of this procedure is to provide guidance in the completion of Data Review Checklist (DRC) Examination, Data Verification, and Data Validation activities as part of the Rocky Flats Environmental Technology Site (RFETS) Analytical Services Division Data Assessment Program. The Data Assessment Program is described in the Kaiser-Hill Analytical Services Division Procedure ASD-001, Performance Assurance Data Assessment Program.

This version of DA-SS01, until replaced by a more recent version, is applicable to all versions of the PSA Module SS01.

This procedure for the data quality assessment of SS01 Sample Data Packages is organized into the following Sections:

- DRC Examination Instructions
- Verification and Validation Instructions
- Data Quality Assessment Report Preparation
- References
- Revision History
- Attachments

2. DATA REVIEW CHECKLIST (DRC) EXAMINATION INSTRUCTIONS

The instructions contained in this section are specific to PSA Module SS01 for Volatile Organics analyses. The instructions in this section are to be used in conjunction with the general instructions for DRC Examination found in Analytical Services Procedure DA-GR01.

2.1. Examination of NA Replies:

Several items in the DRC Checklist may be marked as NA, indicating that the item was not applicable to the analysis performed or to the data package. For the following items, enter \checkmark in the \checkmark column of the DRC to indicate that the NA response is not verified but accepted:

Table 2-1 Non Applicable DRC Items

Section 1 Items	Section 3 Items	Section 4 Items	Section 5 Items
1-d	3-b	4-b	5-b
		4-e	5-c
			5-d
			5-e
			5-f
			5-g

2.1.1. For all other items with marked NA in the Reply column, enter X in the \checkmark column to indicate that the verification is required for this item.

2.2. Examination of the Sample Narrative

Read the sample narrative for information which indicates additional items to be verified. Items to check include statements about data qualifiers, blank contamination, or sample handling problems.

2.2.1. If the narrative states that B or E flags are present, enter X in the \checkmark column of item 6-b-7 to indicate that verification is required for this item due to information provided in the Narrative.

3. VERIFICATION AND VALIDATION INSTRUCTIONS

The instructions contained in this section are specific to PSA Module SS01 for Volatiles Organics analyses. The instructions in this section are to be used in conjunction with the general instructions for DRC Examination found in Analytical Services Procedure DA-GR01. The remainder of this section includes specific instructions for performing verification and validation activities for Sample Data Packages generated under PSA Module SS01. Each section corresponds to a DRC Checklist element that may contain multiple item numbers. These item numbers are referenced within each section of this procedure.

3.1. Chain of Custody, Holding Times, and Sample Preservation

DRC Items 4-a through 4-g

Review Items: Deliverable Section Number 4; Deliverable Section Number 6: Form 1A, COC record, sample preparation/extraction log (medium level only).

Requirement Source: GR01 Exhibit B § 4.8 and SS01 Exhibit D § 3.

Objective: The objective is to ascertain the validity of results based on the holding time and preservation of the sample and to check that Sample COC documentation is included in the sample data package (SDP).

NOTE: The holding time is based on the date when collection was completed, rather than verified time of sample receipt (VTSR).

Evaluation: *The following steps apply to both verification and validation:*

Items 4-a ,b, c, and-e Follow instructions in DA GR01

Item 4-d If samples which were not acid-preserved were not maintained at 4° C prior to receipt by the laboratory, do not qualify the sample results. However, assign the reason code [703] to all applicable samples.

Item 4-f Technical requirements for sample holding times and sample preservation for SS01 are listed in the following table:

Table 3-1 Holding Time and Preservation Criteria

Method	Holding Time (maximum)	Preservation
CLP-SOW	14 days	All water samples preserved with acid; Storage at 4 ^o C
	7 days	All water samples unpreserved
	14 days	Storage at 4 ^o C All soil samples Storage at 4 ^o C
Method 524.2 & SW846 Method 8260	14 days	All samples preserved with HCl or ascorbic acid; Storage at 4 ^o C
	7 days	All water samples unpreserved
	14 days	Storage at 4 ^o C All soil samples Storage at 4 ^o C

Determine the actual analysis and preparation holding times by comparing the preparation and analysis dates on the raw data and the sample collection date on the COC. If the actual holding time is greater than the maximum allowable holding time, record the appropriate qualification and reason codes on the electronic deliverable and in the Data Quality Assessment Report as determined from the following:

- If water volatile samples are not acid preserved and the analysis holding time exceeds seven days but is less than or equal to 14 days, qualify all positive and non-detected results for aromatic compounds as estimated (J). Assign code [**J 101**] if the holding time violation is attributed to the laboratory. If the holding time violation is not attributed to the laboratory, assign code [**J 701**].
- Qualify all positive results as estimated (J) if the actual holding time was greater than the maximum holding time. Assign code [**J 101**] if the holding time violation is attributed to the laboratory. If the holding time violation is not attributed to the laboratory, assign code [**J 701**].
- Qualify all non-detected results as estimated (J) if the actual holding time was greater than the maximum holding time but less than two times the maximum holding time. Assign code [**J 101**] if the holding time violation is attributed to the laboratory. If the holding time violation is not attributed to the laboratory, assign code [**J 701**].
- Qualify all non-detects as rejected (R) if the actual holding time was greater than two times the maximum holding time. Assign reason code [**R 102**] if the hold time violation is attributed to the lab. If the hold-time violation is not attributed to the laboratory, assign reason code [**R 702**].

Note: For aromatic compounds grossly exceeded is determined by the 14 rather than seven day holding time.

Item 4-g If documentation specifically indicates samples were not properly preserved after sample receipt, but prior to analysis, initiate a Non-Compliance Notification and qualify all results as estimated [**J 201**].

3.2. Sample Data Package Narrative

DRC Items 5-a through 5-g

- Review Items:** Deliverable Section Number 5: sample case narrative.
- Objective:** Review the narrative for compliance to requirements and for information useful for validation of data.
- Requirement Source:** GR01 Exhibit B § 4.9 and SS01 Exhibit B § 2.7
- Evaluation:** *The following steps apply to both verification and validation:*
- Check that the SDP Narrative is present and that each Item 5-a through Item 5-d are compliant.
 - If any of the following items are non-compliant, do not qualify the results. Comment and include the reason code **[805]**.
 - Item 5-a** Method reference numbers and revisions.
 - Item 5-b** Descriptions of matrix interferences.
 - Item 5-c** Description of required dilutions.
 - Item 5-d** Explanations of any QC deficiencies, missed holding times, or inability to achieve the required detection limits (RDLs).
 - Item 5-e** Reasons for reanalysis, reanalysis Analytical Batch Identifications Numbers, and a synopsis of the reanalysis Analytical Batch QC Assessment.
 - Item 5-f** Explanations and descriptions of all deviations from routine protocols, including deviations from approved standard operating procedures (SOPs), detection limit modifications, etc. If it was necessary to contact the CTR for instructions due to the nature of the deviation, the laboratory shall document those instructions in the narrative.
 - Item 5-g** Explanations for each item marked "N" on the Data Review Checklist.

3.3. System Monitoring Compound (Surrogate) Recovery

DRC Items 6-a-1, and 6-b-3

- Review Items:** Deliverable Section Number 6: Forms 2A/2B, sample preparation/extraction log (medium level only), sample chromatograms and quantitation reports.
- Objective:** Laboratory performance on individual samples is established by means of spiking samples with surrogate compounds prior to extraction and analysis to determine surrogate spike recoveries. The evaluation of the 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 analytes. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the review and

validation of data based on specific sample results are frequently subjective and demand analytical experience and professional judgment.

Requirement Sources: SS01 Exhibit B § 2.8; SS01 Exhibit D § 2; GR01 Exhibit D.

Evaluation: *The following steps apply to both verification and validation:*

Item 6-a-1 Check that Forms 2A/2B are present.

- If not provided, issue a Non-Compliance Notification to request the missing data. Do not qualify any data. Comment and assign reason code [801] to all applicable data.

Check that surrogate recoveries are reported for all sample, spike, and blank analyses.

- If not provided, issue a Non-Compliance Notification to request the missing data. Do not qualify any data. Comment and assign reason code [803] to all applicable data.

Reanalysis and reextraction/reanalysis are required by all methods if any surrogate recovery is outside the control limits (excludes dilutions).

- If appropriate reanalyses or reextractions were not performed, comment and assign reason code [142] to all applicable data.
- If appropriate reanalyses/reextractions were performed, determine whether the original analysis or the reanalysis/reextraction is to be reported and provide an explanation in the data quality assessment report.

Check that the surrogate percent recoveries (%R) are within the following limits:

Table 3-2 Surrogate Control Limits

CLP-SOW			524.2	SW-846 8260A		
Toluene-d8 (TOL) Bromofluorobenzene (BFB), 1,2-Dichloroethane d4 (DCA)			4-Bromofluorobenzene 1,2-Dichlorobenzene-d4	4-Bromofluorobenzene (BFB), Toluene-d8 (TOL) Dichloroethane d4 (DCA) Dibromofluoromethane (DBF).		
Limits:			Limits:	Limits:		
	Water	Soil		Water	Soil	
TOL:	88-110	84-138	80-120	BFB:	86-115	74-121
BFB:	86-115	59-113		TOL:	88-110	81-117
DCA:	76-114	70-121		DCA:	80-120	80-120
				DBF:	86-118	80-120

If surrogate recoveries for a sample fall outside the control limits, qualify as follows:

- If the recovery of any surrogate is greater than the control limits, estimate [J 142] positive results. (Determine that high bias was not due to interference with the surrogate compound only.)
- If the recovery of any surrogate is less than the control limits but greater than or equal to 10%, estimate [J 142] positive results and [UJ 142] non-detected results.

- If the recovery of any one surrogate is less than 10% in either fraction, estimate [J 142] positive results and reject [R 142] non-detected results for that fraction.

Dilutions

- If no surrogate recovery is reported due to dilution and an undiluted analysis was provided, use professional judgment to evaluate the percent recoveries in the undiluted analysis to qualify the results being reported from the diluted analysis.

If no surrogate recovery is reported due to dilution and an undiluted analysis was not provided, determine if the dilution factor was high enough to justify the surrogates being diluted out.

- If so, estimate [J 142] positive results and [UJ 142] non-detected results. If not, estimate [J 142] positive results and reject [R 142] non-detected results.

Evaluation: *The following steps apply to validation only:*

Item 6-b-3 Check chromatograms and quantitation reports to evaluate the recoveries. Verify at least one surrogate recovery per sample.

- If calculated recoveries are not within 5% of reported result, issue a Non-Compliance Notification and assign reason code [803] to all applicable data.. Cease validation until a new data package is received. Inspect all other SDP deliverables for missing data, incorporate any deficiencies in the Non-Compliance Report, and return the SDP to ASD.

Item 6-a-1 If the %R values are not within the control limits, check the raw data for interferences or misidentification before qualifying the data.

- If raw data confirms % R, no action is required.
- If raw data indicates misidentification, assign reason code [804]. Use professional judgment to assign a qualifier based on the severity of the problem.

3.4. MS/MSD Recovery (CLP & SW-846)

DRC Items 6-a-2, and 6-d-3

Review Items: Deliverable Section Number 6: Forms 3A/3B, Form 6A, MS/MSD chromatograms and quantitation reports.

Objective: These data 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.

Requirement Sources: SS01 Exhibit B § 2.8; SS01 Exhibit D § 2, 4; GR01 Exhibit D.

Evaluation: *The following steps apply to both verification and validation:*

Item 6-a-2 Check that Forms 3A/3B are present and that MS/MSD analyses were performed at the required frequency.

- If not, issue a Non-Compliance Notification to request the missing data. Do not qualify any data. Comment and assign reason code [801] to all applicable data.

Check that the MS/MSD percent recoveries (%R) and relative percent differences (RPD) are within the following limits:

Table 3-3 MS/MSD Frequency and Control Limits

Spiking Compound	CLP-SOW %R Limits[RPD Limit]		SW-846 8260A %R Limit
	Water	Soil	
1,2-Dichloroethene	61-145[14]	59-172[22]	Not Provided. Use CLP.
Trichloroethene	71-120[14]	62-137[24]	
Benzene	76-127[11]	66-142[21]	
Toluene	76-125[13]	59-139[21]	
Chlorobenzene	75-130[13]	60-133[21]	
	Frequency: 1/20 samples		

No action is taken on MS/MSD or matrix duplicate data alone to qualify an entire batch. However, using informed professional judgment the data Reviewer may use the MS/MSD results in conjunction with other QC criteria and determine the need for some qualification of data.

Evaluation: *The following item applies to validation only:*

Item 6-d-3 Calculate at least one percent recovery and one RPD value in the MS/MSD data using the following calculations:

$$\%R = \frac{\text{Found_Value}}{\text{True_Value}} \times 100$$

$$RPD = \frac{|D_1 - D_2|}{\left(\frac{D_1 + D_2}{2}\right)} \times 100$$

where:

D_1 = MS Concentration.

D_2 = MSD Concentration.

- If the %R or % RPD values cannot be verified within 5%, discontinue validation. Inspect all other SDP deliverables for other missing or incomplete information. Issue a Non-Conformance Notification for all noted deficiencies and assign reason code [803] to all applicable data.. Return the SDP to ASD with the Non-Compliance Notification.

3.5. Instrument Performance Check

DRC Items 6-a-4, and 6-d-1

Review Items: Deliverable Section Number 6: Form 5A, bromofluorobenzene (BFB) bar graph spectrum, mass listing, and RIC.

Objective: Instrument performance checks (tuning) are performed to ensure mass resolution, identification, and to some extent, sensitivity. These criteria are not sample specific. Conformance is established by adherence to acceptance criteria using standard

reference materials. These criteria must be met in all circumstances.

Requirement Sources: SS01 Exhibit B § 2.8; SS01 Exhibit D § 2; GR01 Exhibit D.

Evaluation: The following steps apply to both verification and validation:

Item 6-a-4 Check that Form 5A is present for the all calibrations and that all samples are included.

- If not, issue a Non-Compliance Notification to request the missing data. Do not qualify any data. Comment and assign reason code [801] to all applicable data.

Verify that the sample analyses occurred within 12 hours (8 hours for 524.2) of the daily BFB instrument performance check.

- If the samples were analyzed outside the time limit, use professional judgment to qualify the data based upon the severity of the problem. At a minimum, comment and assign reason code [139] to all applicable data.

Check that the following BFB ion abundance criteria are met:

Table 3-4 BFB Ion Abundance Criteria

m/z	CLP-SOW	524.2	SW-846 8260A
50	8.0-40.0% of m/z 95	15-40% of m/z 95	15-40% of m/z 95
	30.0-66.0% of m/z 95	30-80% of m/z 95	30-60% of m/z 95
	Base peak. 100% Relative Abundance	Base peak	Base peak
	5.0-9.0% of m/z 95	5-9% of m/z 95	5-9% of m/z 95
	< 2% of m/z 174	< 2% of m/z 174	< 2% of m/z 174
	> 50.0-120.0% of m/z 95	> 50% of m/z 95	> 50% of m/z 95
	4.0-9.0% of m/z 174	5-9% of m/z 174	5-9% of m/z 174
	93.0-101.0% of m/z 174	> 95% but < 101% of m/z 174	> 95% but < 101% of m/z 174
	5.0-9.0% of m/z 176	5-9% of m/z 176	5-9% of m/z 176

- If mass assignment is in error (e.g., m/z 96 is assigned as the base peak), reject [R 139] all associated data.
- If ion abundance criteria are not met, reject all associated results [R 139].

Evaluation: *The following item applies to validation only:*

Item 6-d-1 Verify from the raw data that the mass assignment is correct and that the mass listing is normalized to the correct base peak. Verify that the mass calibration is correct and that there are not transcription errors. Compare the mass listings submitted in the raw data to the reported relative abundances. Recalculate two m/z ratios. If possible, verify that spectra were generated using appropriate background subtraction techniques.

- If any problems are found, issue a Non-Compliance Notification to request the missing data. Do not qualify any data. Comment and assign reason code [803] to all applicable data.

3.6. Internal Standard Area and RT Summary

DRC Item 6-a-5

Review Items: Deliverable Section Number 6: Forms 8A, calibration quantitation reports, sample chromatograms and quantitation reports.

Objective: Internal standard (IS) performance criteria ensure that the GC/MS sensitivity and response are stable for every analytical run.

Requirement Sources: SS01 Exhibit B § 2.8; SS01 Exhibit D § 2; GR01 Exhibit D.

Evaluation: *The following steps apply to both verification and validation:*

- Item 6-a-5** Check that Form 8A is present for all calibrations associated with sample analyses and that all samples and blanks are included.
- If discrepancies are found, issue a Non-Compliance Notification to request the missing data. Do not qualify any data. Comment and assign reason code **[801]** to all applicable data.
 - If the IS compounds vary from the method, use professional judgment to assess the impact on the data. At a minimum, comment and assign reason code **[143]** to all applicable data. The following summarizes the IS compounds required:

Table 3-5 Internal Standard Control Compounds

CLP-SOW	524.2	SW-846 8260A
Bromochloromethane	Fluorobenzene	Fluorobenzene
1,4-Difluorobenzene		2-Bromo-1-chloropropane
Chlorobenzene-d5		1,4-Dichlorobenzene-d4

The IS area count in the samples and blanks must not differ by more than a factor of two (-50% to +100%) from the area count measured in the associated calibration standard. If so, qualify as follows:

- If the area count for any IS in a sample is above the acceptance limits (+100%), estimate **[J 143]** positive results for compounds quantitated using that IS.
- If the area count for any IS in a sample is below the acceptance limits (-50%), estimate **[J 143]** positive results and **[UJ 143]** non-detected results for compounds quantitated using that IS.
- If the area count for any IS in a sample is extremely low (i.e., less than 50% of the lower control limit), or if instrument sensitivity exhibits a major abrupt drop off, reject **[R 143]** non-detected results and estimate **[J 143]** positive results for compounds quantitated using that IS.

Evaluation: *The following item applies to validation only:*

Item 6-a-5 The IS retention time in the continuing calibration standard must not vary by more than ± 30 seconds from the last daily calibration standard.

- If ± 30 seconds is exceeded, reject [R 143] affected non-detected results and estimate [J 143] affected positive results for compounds quantitated using that IS.

Verify that area counts and retention times are correctly transcribed from the sample and standard quantitation reports onto the Forms 8B/8C.

- Depending upon the magnitude of the problem, issue a Non-Compliance Notification to request clarification or explanation of the data. Comment and assign reason code [804] to all applicable data. Alternatively, use the times and area counts from the quantitation reports.

The IS retention time in the continuing calibration standard must not vary by more than ± 30 seconds from the last daily calibration standard.

- If so, examine the chromatogram for evidence of false positives or false negatives. Use professional judgment to qualify the data based upon the chromatogram and magnitude of the shift.
- Reject [R 143] affected non-detected results and estimate [J 143] affected positive results for compounds quantitated using that IS.

3.7. Sample Results (Target Identification)

DRC Items 6-b-1, 6-b-3 through 6-b-5

Review Items: Deliverable Section Number 6: Form 1A, Form 6A, Form 7A, COC record, sample extraction/preparation logs (medium level only), and sample chromatograms and quantitation reports.

Objective: Qualitative criteria for compound identification have been established to minimize the number of false positives (reporting a compound present when it is not) and false negatives (not reporting a compound that is present).

Requirement Sources: SS01 Exhibit B § 2.8; SS01 Exhibit D § 2; GR01 Exhibit D.

Evaluation: *The following steps apply to both verification and validation:*

Item 6-b-1 Check that Form 1A is present for each sample.

- If not provided, issue a Non-Compliance Notification to request the missing data. Do not qualify any data. Comment and assign reason code [801] to all applicable data.

Check that significant figures and flagging protocol are as specified in the latest version of CLP.

- If significant problems exist, issue a Non-Compliance Notification to request the missing data. Do not qualify the data. Comment and assign reason code [803] to all applicable data.

Evaluate Form 1A to ensure that no "B" qualifiers are present.

- If "B" qualifiers are present, proceed with the qualification specified under Blanks.

Ensure that blank contamination is addressed in the SDP Narrative.

- If not addressed, do not qualify the results. Comment and include the reason code [805].

Evaluation: *The following item applies to validation only:*

Item 6-b-3 Verify that all significant peaks on the chromatogram are accounted for as target compounds, TICs, surrogates, and internal standards.

- In the event a target compound, surrogate, or internal standard are misidentified, correct the EDD and the specific data point on the Form 1 (initial and date) and assign the reason code [804].
- In the event a TIC is misidentified, comment in the Data Quality Assessment Report.

Relative Retention Time

Item 6-b-4 Confirm positive results by reviewing the relative retention times (RRTs) for positive results on the sample integration reports to those for the associated calibration standard. The RRTs must be within ± 0.06 RRT units of the standard RRT for CLP and SW-846. The GC retention time of the sample component should be within 3 standard deviations of the mean retention time of the compound in the calibration mixture for 524.2.

$$RRT = \left(\frac{RT_{com}}{RT_{is}} \right)$$

where:

RT_{com} = Retention time of the compound of interest

RT_{is} = Retention time of nearest internal standard

- If results are not within ± 0.06 RRT units, or 3 standard deviations (524.2), qualify the associated results as estimated, [J 143].

Item 6-b-5 Mass spectra of the sample compound and a laboratory-generated standard must match as follows:

CLP & 524.2

- ◇ All ions present in the standard mass spectra at relative intensity greater than 10.0 percent (most abundant ion in the spectrum equals 100.0 percent) must be present in the sample spectrum.
- ◇ The relative intensities of these ions must agree within $\pm 20\%$ between the sample and standard spectrum.
- ◇ Ions greater than 10.0 percent in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. Favor false positives.

SW-846

- ◇ The intensities of characteristic ions maximize the same scan or within one scan of each other in the standard versus the sample.

- ◇ The relative intensities of these ions must agree within $\pm 30\%$ between the sample and standard spectrum.
- ◇ Structural isomers (very similar mass spectra) should be identified separately if the height of the valley between the isomer peaks is less than 25% of the sum of the two peaks. Otherwise, structural isomers are identified as isomeric pairs.

The application of qualitative criteria for GC/MS analysis compounds requires professional judgment.

- If it is determined that an incorrect false positive exists, the result should be reported as non-detected [**U 145**].
- If it is determined that a false negative exists, reject [**R 145**] the non-detected result if the unreported positive result exceeds the RDL.

Be aware of situations (e.g., high concentration samples preceding low concentration samples) when sample carry-over is a possibility. Use professional judgment to determine if carry-over occurred and that the data was qualified appropriately.

3.8. Compound Quantitation and RDLs

DRC Items 6-b-1, 6-b-3, 6-b-4, 6-b-6 and 6-b-7

Review Items: Deliverable Section Number 6: Form 1A, Form 6A, COC record, sample preparation/extraction logs (medium level only), sample chromatograms and quantitation reports.

Objective: The objective is to ensure that the reported quantitation results and detection limits are accurate.

Requirement Sources: SS01 Exhibit B § 2.8; SS01 Exhibit C; SS01 Exhibit D § 2, 4; and GR01 Exhibit D.

Evaluation: *The following items apply to both validation and verification:*

Item 6-b-1: Using the Line Item Code from the COC record, ensure that the detection limits reported on Form 1A match the required detection limits (RDLs) listed in SS01, Exhibit C. Note that dilutions, percent solids, and extraction steps will impact the final RDLs reported.

If RDLs on Form 1A are not those required by the Line Item Code requested, check the RIN file for additional information, which may explain the deviation.

- If an explanation is not found, comment and qualify all results reported as [**R 213**].

Evaluate Form 1A to ensure that no “E” qualifiers are present. If “E” qualifiers are present, ensure that another Form 1A with a diluted sample analysis is present in the data package.

- If not, comment and estimate [**J 148**] the positive “E” result.

Ensure that required dilutions are addressed in the SDP Narrative.

- If not addressed, do not qualify the results. Comment and include the reason code **[805]**.
- If the diluted sample analysis fails to keep the response of the major constituents in the upper half of the calibration range, use professional judgment to qualify the data. At a minimum, comment and assign reason code **[148]** to all applicable data.

Evaluation: *The following items apply to validation only:*

- Item 6-b-4** Verify that compound quantitation, as well as the adjustment of the MDLs, is performed according to the method. Target compound quantitation should be performed using the internal standard associated by the method.
- If not, use professional judgment to qualify the data.
- Ensure that the same internal standard was used in the standard as in the sample.
- If a different internal standard was used in the sample than in the standard, do not qualify any data. Issue a Non-Compliance Notification to request the corrected data, comment, and assign reason code **[803]** to all applicable data.
- Item 6-b-3** Target compounds should be quantitated from the primary ion listed in the method unless the primary ion has interference from the sample.
- If a secondary quantitation ion was used, verify that the standard was also quantitated from the same secondary quantitation ion.
- If a different quantitation ion was used in the sample than in the standard, do not qualify any data. Issue a Non-Compliance Notification to request the corrected data, comment, and assign reason code **[804]** to all applicable data.
- Item 6-b-6** Verify that acceptable (NIST/EPA/MSDC) Mass Spectral Library Searches are provided for all sample identifications.
- If reference spectra are not provided, but applicable reference spectra can be found elsewhere in the data package, comment and issue a Non-Compliance Notification and assign the reason code **[802]** to all applicable data.
 - If acceptable reference spectra cannot be found, discontinue validation. Inspect all other SDP deliverables for any other missing or incomplete information. Issue a Non-Conformance Notification for all noted deficiencies and assign reason code **[801]** to all applicable data. Return the SDP to ASD with the Non-Compliance Notification.
- Item 6-b-7** Verify that all B and E qualifiers are explained in the case narrative.
- If not, comment and assign the reason code **[805]** to all applicable data.

Calculations

- Item 6-b-4** Examine the raw data to verify the correct calculation of one positive result per sample. Quantitation reports, chromatograms, sample preparation/extraction logs, dilutions, and cleanups are compared to the reported sample results.

- If significant problems exist, issue a Non-Compliance Notification to request clarification of the data or receipt of missing or additional data. Do not qualify the data. Comment and assign reason code **[803]** to all applicable data.

Calculate using the following equations:

Purgeables:

Water and water-miscible waste:

$$\frac{ug}{L} = \frac{A_x \times I_s \times DF}{A_{is} \times RRF \times V_0}$$

where:

- A_x = Response of the characteristic ion for the analyte in the sample, area counts.
- I_s = Amount of internal standard injected, ng.
- DF = Dilution factor.
- A_{is} = Response of the characteristic ion for the internal standard, area counts.
- RRF = Response factor for the analyte from the appropriate calibration standard.
- V_0 = Volume of water purged, mL.

Sediment/soil, sludge, and waste:

Medium Level

$$\frac{ug}{Kg} = \frac{A_x \times I_s \times V_t \times DF}{A_{is} \times RRF \times V_i \times W_s \times P}$$

Low-Level

$$\frac{ug}{Kg} = \frac{A_x \times I_s \times DF}{A_{is} \times RRF \times W_s \times P}$$

where:

- A_x, I_s, A_{is}, RRF = Same as in water and water-miscible waste above.
- V_t = Volume of total extract, uL.
- V_i = Volume of extract added for purging, uL.
- W_s = Weight of sample extracted or purged, g.
- P = Percent Solids/100.

TICs only:

- A_x = Total response of the TIC in the sample, area counts.
- A_{is} = Total response of the nearest internal standard free of interference, area counts.
- RRF = One (1.0).

- If the RRF values are not verified within 5%, Issue a Non-Compliance Notification and stop the validation process. Inspect all other SDP deliverables for any other missing or revised information needed for data assessment, assign reason code **[803]** to all applicable data, and return the SDP to ASD with a Non-Compliance Notification.

3.9. Tentatively Identified Compounds (TICs)

DRC Items 6-b-2, 6-b-5, 6-b-6, and 6-d-2

Review Items: Deliverable Section Number 6: Form 1E, sample extraction/preparation logs (medium level only), and sample chromatograms and quantitation reports.

Objective: Peaks not identified as target compounds, surrogates, or internal standards are potential TICs. TICs must be qualitatively identified by a NIST/EPA/MSDC mass spectral library search and the identifications assessed by the data reviewer.

Requirement Sources: SS01 Exhibit B § 2.8; SS01 Exhibit D § 2; GR01 Exhibit D.

Evaluation: *The following items apply to both verification and validation:*

Item 6-b-2 Check that Form 1E is present for each sample.

- If not provided, issue a Non-Compliance Notification to request the missing data. Do not qualify any data. Comment and assign reason code **[801]** to all applicable data.

Verify that all TIC results are qualified by the laboratories as “**NJ**”.

- If TIC results are not qualified as “**NJ**” by the laboratory, “**NJ**” shall be applied to all applicable results by the data assessor with reason code **[804]**.

Evaluation: *The following items apply to validation only:*

Item 6-b-2 Verify that the alkane series are properly identified and reported as series (e.g., C5-C9 as a single entry along with the estimate for the total concentration of the series).

- If not, issue a Non-Compliance Notification to request the corrected data, comment, and assign reason code **[803]** to all applicable data.

Verify that no volatile target compounds are mistakenly reported as TICs.

- If so, the quantitation is in error due to the use of total area. Do not qualify any data. Issue a Non-Compliance Notification to request the corrected data, comment, and assign reason code **[226]** to all applicable data.

Verify that semivolatile compounds reported as target analytes in the semivolatile fraction are not also reported as TICs in the volatile fraction.

- If so, reject **[R 199]** the TIC.

Note: If a TIC result in the sample is not sufficiently above the level in the blank, the TIC result should not be reported. (Generally, the 10X blank rule is applied here.)

Item 6-b-6 and 6-d-2

Verify that the laboratory has generated a NIST/EPA/MSDC mass spectral library search for all required peaks in the chromatograms for samples and blanks.

- If not, issue a Non-Compliance Notification to request the corrected data, comment, and assign reason code **[803]** to all applicable data.
- When a compound is not found in any blanks but is a suspected common laboratory artifact/contaminant, reject **[R 199]** the TIC. Examples:
 - ◊ Common contaminants: CO₂ (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 less than 100ug/L or 4000 ug/Kg.
 - ◊ Solvent preservative cyclohexene (methylene chloride preservative). Related by-products cyclohexanone, cyclohexenone, cyclohexanol, cyclohexenol, chlorocyclohexene, and chlorocyclohexanol.
 - ◊ Aldol reaction products of acetone including: 4-hydroxy-4-methyl-2-pentanone, 4-methyl-2-penten-2-one, and 5,5-dimethyl-2(5H)-furanone.

Item 6-b-5 Evaluate the TIC Spectra using the following guidance:

- ◊ All ions present in the standard mass spectrum at a relative intensity greater than 10% should be present in the sample spectrum.
- ◊ The relative intensities of these ions must agree within $\pm 20\%$ between the sample and reference spectrum.
- ◊ Molecular ions present in the reference spectrum should be present in the sample spectrum.
- ◊ Ions present in the sample spectrum but not present in the reference spectrum must be reviewed for possible background contamination, interference, or coelution of additional TIC or target compounds.
- When the above criteria are not met but the identification is correct in the technical judgment of the data reviewer or mass spectral interpretation specialist, report the TIC as is.
- If the identification is uncertain in the data reviewer's judgment, or there are extenuating factors affecting compound identification, the TIC result may be reported as "unknown" or changed to an appropriate identification.
- If more than one possible match exists, the TIC may be reported as "either compound X or compound Y."
- If isomer specificity is in question, the TIC result may be changed to a non-specific result (e.g., 1,3,5-trimethyl benzene to trimethyl benzene isomer).
- If identification is uncertain but other samples have a TIC of similar RRT and the same ions, identification information may be inferred.

Examine blank chromatograms to verify that sample TICs are not found in the blanks. Be aware that TICs at low levels in the samples may also be present in the blanks at levels below 10% of the internal standard height. This is particularly likely for common laboratory artifacts and contaminants.

- If a TIC identification is changed for any of the above reasons, add the reason code [226] to all affected data points.

Examine the raw data to verify the correct calculation of one TIC result per sample. Use the equation in Section 3.8.

- If significant problems exist, issue a Non-Compliance Notification to request the information necessary to resolve the discrepancy. Do not qualify the data. Comment and assign reason code [803] to all applicable data.

3.10. Calibration

DRC Items 6-c-1 through 6-c-4,

Review Items: Deliverable Section Number 6: Form 6A, Form 7A, sample and standard chromatograms and quantitation reports.

Objective: Compliance 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 of producing a linear calibration curve. Continuing calibration establishes the 8 or 12-hour relative response factors on which the quantitations are based and checks satisfactory performance of the instrument on a day-to-day basis.

Requirement Sources: SS01 Exhibit B § 2.8; SS01 Exhibit D § 2; GR01 Exhibit D.

Evaluation: *The following items apply to both verification and validation:*

Initial Calibration

Item 6-c-1 The initial calibration must include all target compounds and surrogates, must be performed within 8 (524.2) or 12 (others) hours of the associated instrument performance check, and must be performed at the beginning of the sequence or when continuing calibration criteria are not met. The acceptance criteria for samples are as follows:

Table 3-6 Initial Calibration Criteria

Method	%RSD
CLP	25%
8260	25%
524.2	30%

- If an inappropriate number of standards or inappropriate concentration levels are analyzed, use professional judgment to assess the impact on the data. At a minimum, comment and assign reason code [168] to all applicable data.
- Estimate [J 140] positive results and [UJ 140] non-detected results for those compounds whose %RSDs exceed the criteria in the associated initial calibration.

- Estimate [J 140] positive results for those compounds whose RRFs are less than 0.05.
- Reject [R 140] non-detected results for those compounds whose RRFs are less than 0.05.

Evaluation: *The following items apply to validation only:*

Item 6-c-3 Target compounds should be quantitated from the primary ion listed in the method unless the primary ion has interference from the sample.

If a secondary quantitation ion was used, verify that the standard was also quantitated from the same secondary quantitation ion.

- If a different quantitation ion was used in the sample than in the standard, do not qualify any data. Issue a Non-Compliance Notification to request an explanation or revised narrative, and assign reason code [803] to all applicable data.

Item 6-c-4 Check the raw data and verify at least one RRF per calibration standard. Recalculate at least one average RRF and %RSD:

$$RRF = \frac{A_x \times C_{is}}{A_{is} \times C_x}$$

where:

A_x = Response for the characteristic ion for the analyte to be measured, units area counts.

C_{is} = Concentration of the internal standard, ug/L.

A_{is} = Response for the characteristic ion for the internal standard, units area counts.

C_x = Concentration of the analyte to be measured, ug/L.

$$\%RSD = \frac{SD}{\bar{X}} \times 100$$

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

where:

X_i = Each individual value used to calculate the mean

\bar{X} = The mean of n values

n = The total number of values

- If the RRF for the %RSD cannot be verified to within 0.5%, issue a Non-Conformance Notification to obtain corrected values and discontinue validation. Assign reason code [803] to all associated data. Inspect all other SDP deliverables for missing data, incorporate any deficiencies in the Non-Compliance Report, and return the SDP to ASD.

Evaluation: *The following items apply to both verification and validation:*

Continuing Calibration

Item 6-c-2: The continuing calibration must include all target compounds and surrogates, and must be analyzed at the beginning of each 8 or 12-hour analysis period following the instrument performance check and prior to the sample and blank analyses. The acceptance criteria used for validation of samples are as follows:

Table 3-7 Continuing Calibration Criteria

Method	%D
CLP	25%
8260	25%
524.2	30%

- If the continuing calibration frequency criteria are not met or if inappropriate concentration levels are analyzed, use professional judgment to assess the impact on the data. At a minimum, comment and assign reason code [168] to all applicable data.
- Estimate [J 141] positive results and [UJ 141] non-detected results for those compounds whose %Ds exceed the criteria in the continuing calibration.
- Estimate [J 141] positive results for those compounds whose RRFs are less than 0.05.
- Reject [R 141] non-detected results for those compounds whose RRFs are less than 0.05.

Evaluation: *The following items apply to validation only:*

Item 6-c-3 Target compounds should be quantitated from the primary ion listed in the method unless the primary ion has interference from the sample.

If a secondary quantitation ion was used, verify that the standard was also quantitated from the same secondary quantitation ion.

- If a different quantitation ion was used in the sample than in the standard, do not qualify any data. Issue a Non-Compliance Notification to request an explanation or revised narrative, and assign reason code [803] to all applicable data.

Item 6-c-4 Recalculate at least one daily RRF and %D:

$$\%D = \frac{R_1 - R_2}{R_1} \times 100$$

where:

R_1 = Calibration factor from first analysis.

R_2 = Calibration factor from subsequent analysis.

- If calculated results are not verifiable to within 5%, initiate a Non-Conformance Notification, cease validation until revised calculations are obtained. Inspect all other SDP deliverables for missing data, incorporate any deficiencies in the Non-

Compliance Report, assign reason code [803] to all applicable data, and return the SDP to ASD.

3.11. Quality Control Check Samples (SW-846, 524.2)

DRC Items 6-a-2 and 6-d-3,

Review Items: Deliverable Section 6: Form 3A.

Objective: Quality control check sample (also known as laboratory fortified blank [LFB]) data are generated to provide information on the accuracy of the analytical method and laboratory performance.

Requirement Sources: SS01 Exhibit B § 2.8; SS01 Exhibit D § 2; 4 GR01 Exhibit D.

Evaluation: *The following items apply to both verification and validation:*

Item 6-a-2

QC Check Sample Frequency

SW-846 8260A

If the percent recovery for any compound in the MS/MSD analysis is outside the control limits, verify that a quality control check sample was analyzed containing that compound. The quality control check sample percent recoveries should be summarized on Form 3A and supporting data should be provided.

- If not, issue a Non-Compliance Notification to request the missing data. Do not qualify any data. Comment and assign reason code [803] to all applicable data.

Method 524.2

Method 524.2 analyses: One LFB is required with each batch of samples processed within a working shift (up to 20 samples).

- If the frequency requirement is not met, do not qualify the data, however, assign reason code [168] to the data.
- If LFB was not analyzed, qualify all results as [J 168].

QC Check Sample Percent Recovery

Check that the quality control check sample/LFB percent recoveries (%R) are within the following limits:

Table 3-8 Quality Control Sample Percent Recovery Limits

SW-846 8260A		524.2
1,2-Dichloroethene	61-145	All compounds within 80-120
Trichloroethene	71-120	
Benzene	76-127	
Toluene	76-125	
Chlorobenzene	75-130	

SW-846 8260A

No action is taken on quality control sample data alone to qualify an entire batch. However, using informed professional judgment the data reviewer may use the quality control sample results in conjunction with other QC criteria and determine the need for some qualification of data. Do not qualify the data. However, assign the reason code [110].

Method 524.2 only

- If any target compound is absent from the LFB analyses, estimate [J] positive results and reject [R 110] non-detected results for that compound in the associated samples.
- If the recovery of any compound is greater than the recovery limits in both the LFB, estimate [J 110] positive results for that compound in the associated samples.
- If the recovery of any compound is less than the recovery limits but greater than 10% in the LFB, estimate [J 110] all positive and non-detected results for that compound in the associated samples.
- If the recovery of any compound is less than 10% in the LFB, estimate [J 110] positive results and reject [R 110] non-detected results for that compound in the associated samples.

Item 6-d-3 Check that Form 1A is present for all MS/MSD analyzed.

- If not provided, issue a Non-Compliance Notification to request the missing data. Do not qualify any data. Comment and assign reason code [801] to all applicable data.

Evaluation: *The following item applies to validation only:*

Item 6-d-3 Verify that all significant peaks on the chromatogram are accounted for as target compounds, TICs, surrogates, and internal standards.

- In the event a target compound, surrogate, or internal standard are misidentified, correct the EDD and the specific data point on the Form 1 (initial and date) and assign the reason code [804].
- In the event a TIC is misidentified, comment in the Data Quality Assessment Report.

Calculate at least one percent recovery in the quality control sample data using the following calculation:

$$\%R = \frac{\text{Found_Value}}{\text{True_Value}} \times 100$$

- If calculated results are not verifiable to within 5%, initiate a Non-Conformance Notification, cease validation until revised calculations are obtained. Inspect all other SDP deliverables for missing data, incorporate any deficiencies in the Non-Compliance Report, assign reason code [803] to all applicable data, and return the SDP to ASD.

3.12. **Blanks**

DRC Item 6-a-3, and 6-d-1 and 6-d-2

Review Items: Deliverable Section Number 6: Form 4A, Method Blank Form 1A, chromatograms and quantitation reports.

Objective: The assessment of laboratory blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of laboratory blanks apply to any blank associated with the samples (e.g., method, instrument, trip, or equipment blanks). If problems with any blank exist, all data associated with the method blank 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.

Requirement Sources: SS01 Exhibit B § 2.8; SS01 Exhibit D § 2; 4; GR01 Exhibit D.

Evaluation: *The following items apply to both verification and validation:*

Item 6-a-3 Verify that Method Blank Summary Forms (4A) are present.

- If not provided, issue a Non-Compliance Notification to request the missing data. Do not qualify any data. Comment and assign reason code **[801]** to all applicable data.

Item 6-d-1 The following table summarizes the blank criteria:

Table 3-9 Blank Criteria

Method	Type and Frequency	Criteria
CLP	Method Blank: once every 12 hours Storage Blank: once per SDG Instrument Blank: after saturated ions in a sample	No contaminants should be present in the blanks. The method blanks should be analyzed after the calibration standards
524.2	Method Blank: 1 per batch of samples processed as a group within a work shift	No target compounds at or above MDL
SW-846 8260A	Method Blank: once per batch (up to 20 samples), before processing any samples, when there is a change in reagents, and following any concentrated sample that has saturated ions from a compound.	No interferences at or above MDL The blank should be carried through all stages of sample preparation and measurement (i.e., extraction blanks should be analyzed on each instrument used for sample analyses)

If more than one blank is associated with a sample, qualification should be based upon comparison of the blank with the highest level of contamination.

- If the proper blanks were not analyzed at the appropriate frequency, determine the severity of the problem and its effect on the data using professional judgment. At a minimum, comment and assign reason code **[168]** to all applicable data.

If a target compound is found at any concentration in the blanks but not in the samples, no action is taken.

If a target compound is found in the blanks at any concentration and is also found in the sample, apply the following:

- If the sample concentration is less than 5 times the blank concentration (10 times for common contaminants) and less than or equal to the RDL: Report the RDL followed by [U 149].
- If the sample concentration is less than or equal to 5 times the blank concentration (10 times for common contaminants) and greater than the RDL: Report the value followed by [U 149].
- If the sample concentration is greater than 5 (10 times for common contaminants) times the blank concentration and greater than the RDL do not qualify the reported value.
- If an associated method blank was not analyzed for the samples, estimate [J 149] positive results.

The common contaminants according to the National Functional Guidelines for Organic Data Review (2/94) are:

Purgeables:

- ◇ Acetone
- ◇ 2-Butanone
- ◇ Methylene chloride

Item 6-d-2 Check that Form 1A is present for each blank.

- If not provided, issue a Non-Compliance Notification to request the missing data. Do not qualify any data. Comment and assign reason code [801] to all applicable data.

Check that Form 1E is present for each blank.

- If not provided, issue a Non-Compliance Notification to request the missing data. Do not qualify any data. Comment and assign reason code [801] all applicable data.

Evaluation: *The following item applies to validation only:*

Item 6-d-2 If an associated method blank exhibits gross contamination, reject [R 149] positive results for the affected compounds. The Functional Guidelines define gross contamination as saturated peaks. Professional judgment must be used to assess the impact the contamination has on the associated samples and which compounds are considered affected.

Verify that all significant peaks on the chromatogram are accounted for as target compounds, TICs, surrogates, and internal standards.

- In the event a target compound, surrogate, or internal standard are misidentified, correct the EDD and the specific data point on the Form 1 (initial and date) and assign the reason code [804].
- In the event a TIC is misidentified, comment in the Data Quality Assessment Report.

Recalculate one positive result per blank. Review the chromatograms and quantitation reports to evaluate blank results.

Note: The Reviewer must consider the weights, volumes, percent solids, and dilution factors when applying the 5x and 10x rules. These

factors must be accounted for so that an actual comparison of the contamination is made. The Reviewer should be particularly aware of sample results which undiluted exceed the action level, but fall within the action level as a result of the subsequent dilution.

- If the calculated result do not agree within 5% or if a compound was misidentified, comment and assign reason code **[804]** to all applicable data. Review all other positive blank results.

3.13. Sample Preparation Raw Data

DRC Items 6-e-1 and 6-e-2

Review Items: Deliverable Section Number 6-e.

Objective: To check that sample preparation raw data deliverable requirements have been met and that raw data are present in a form suitable for validation and retention.

Requirement Sources: GR01 Exhibit B § 4.11, GR01 Exhibit F § 4, SS01 Exhibit B §2.9, and requirements of base methods cited in SS01 Exhibit D Section 2.

Evaluation: *The following items apply to validation activities only:*

Item 6-e-1 Check that preparation raw data (benchsheets and/or preparation logs) are included for all analyses performed and include the following:

- ◇ Analytical Batch identifier
- ◇ Date of preparation
- ◇ Identifiers for all samples, sample duplicates, and spikes
- ◇ Identifiers for at least one preparation blank and lab control sample
- ◇ For aqueous samples initial and final volumes for all samples and QC samples
- ◇ For solids and non-aqueous liquids reported by weight, initial weights and final volumes for all samples and QC samples
- ◇ For samples reported by weight, balance identifiers with dates of use.
- ◇ Dated signatures for at least one analyst and one reviewer

Check this item as complete if raw data were sufficient to perform calculations for all previous items.

- Omissions or errors which do not affect your ability to review the data shall be documented and with reason code **[804]**.
- Other omissions or errors shall be documented for inclusion in a Non-Compliance Notification. Inspect all other SDP deliverables for any other missing or revised information needed for data assessment and return the SDP to ASD with Non-Compliance Notification. Assign reason code **[803]** to all applicable data.

- Item 6-e-2** Verify that instrument run logs are available for all analytical sequences.
- Omissions or errors which do not affect your ability to review the data shall be documented and with reason code **[804]**.
 - Other omissions or errors shall be documented for inclusion in a Non-Compliance Notification. Inspect all other SDP deliverables for any other missing or revised information needed for data assessment and return the SDP to ASD with Non-Compliance Notification. Assign reason code **[803]** to all applicable data.

3.14. **Electronic Data Deliverable (EDD)**

DRC Items 7-a through 7-c

Review Items: Deliverable Section Number 10.

Objective: To ensure that electronically-reported data are accurate.

Requirement Sources: GR01 Exhibit B and GR02.

Evaluation: *The following items apply to both verification and validation:*

Item 7-a through 7-c

See DA-GR01 for evaluation.

4. DATA QUALITY ASSESSMENT REPORT PREPARATION

Prepare a Data Quality Assessment Report according to the General Data Assessment guidelines presented in DA-GR01. A Data Quality Assessment Report template for DV-SS01 is presented as Attachment 1.

5. REFERENCES

- USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, February 1994.
- Reason Codes for Data Assessment, Analytical Services Document
- Statement of Work for Analytical Measurements, General Laboratory Requirements, Module GR01-B.1, June 2, 1997.
- Statement of Work for Analytical Measurements, Volatile Organics, Module SS01-B, March 28, 1997.

6. REVISION HISTORY

- The first draft of DA-SS01 was prepared by QuantaLex Inc.
- Final drafting of DA-SS01-v1 was completed by Ed Brovsky of Kaiser-Hill Analytical Services on December 3, 1997. This revision included: formatting for consistency with DA-GR01-v1, formatting to separate evaluation and action criteria, inclusion of new and revised reason codes, corrections and additions of evaluation and action criteria, and general editing.

Effective Date:
December 3, 1997

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Attachment 1: Data Quality Assessment Report Template

SS01

**Data Quality Assessment Report
Rocky Flats Environmental Technology Site**

RIN Number	Analytical Method/PSA Line Item	Validation Level
Analytical Laboratory	Assessment Performed by	Number of Samples/ Matrix.

Sample Numbers: _____

Quality Control Element	Reviewed	Non-Compliance Identified
General (Cover Page, Table of Contents, DRC Checklist, Narrative)		
Chain of Custody		
Holding Times		
Sample Preservation		
Surrogate Recovery		
Matrix Spike/Matrix Spike Duplicate		
Instrument Performance Check		
Internal Standards		
Sample Results		
Tentatively Identified Compounds		
Calibration		
Quality Control Check Samples		
Blanks		
Other QC		

- Y Item was reviewed or non-compliance was identified
- N Item was not reviewed or non-compliance was not identified
- N/A Item is not applicable to the Line Item

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Action Items:

Comments:

Verification/Validation Signature _____

Date: _____

Reviewer Signature _____

Date: _____

(Validation Only)

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