Analytical Services Division

Rocky Flats Environmental Technology Site

VERIFICATION AND VALIDATION GUIDELINES

FOR

SEMIVOLATILE ORGANICS

DA-SS02-v1

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1. PURPOSE AND INTRODUCTION

This procedure presents those data assessment steps which are unique to PSA Module SS02, Semivolatile 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-SS02, until replaced by a more recent version, is applicable to all versions of the PSA Module SS02.

This procedure for the data quality assessment of SS02 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 SS02 for Semivolatile 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 √ in the √ 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 √ 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 √ 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 SS02 for Semivolatile 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 SS02. 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 1D, COC record, sample preparation/extraction log.

Requirement Source: GR01 Exhibit B § 4.8 and SS02 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, &-e Follow instructions in DA GR01

Item 4-d If samples were not maintained at 4°C prior to receipt by the laboratory, do not qualify the sample results. Comment and assign the following code to all applicable samples **[703]**.

Item 4-f Technical requirements for sample holding times and sample preservation for SS02 are listed in the following table:

Table 3-1 Holding Time and Preservation Criteria

Matrix	Extraction Holding Time (maximum)	Analysis Holding Time (maximum)	Preservation
Water	7 days	40 days	Storage at 4°C
Soil	14 days	40 days	Storage at 4°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:

- 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]**.

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 SS02 Exhibit B § 2.7

Evaluation: *The following items 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 laboratory 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 2C/2D, sample preparation/extraction log, 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: SS02 Exhibit B § 2.8; SS02 Exhibit D § 2; GR01 Exhibit D.

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

Item 6-a-1 Check that Forms 2C/2D 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 for SW-846 if any surrogate recovery is outside the control limits (excludes dilutions). They are required for CLP if more than one base/neutral or acid surrogate is outside the control limits or if any surrogate has a recovery less than 10% (excludes dilutions and surrogates with advisory limits).

- 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

Surrogate Compounds	CLP-SOW		SW-846 8270B	
Base/Neutrals	Water	Soil	Water	Soil
Nitrobenzene-d5	35-114	23-120	35-114	23-120
2-Fluorobiphenyl	43-116	30-115	43-116	30-115
p-Terphenyl-d14	33-141	18-137	33-141	18-137
1,2-Dichlorobenzene-d4	16-110*	20-130*		
Acids				
Phenol-d5	10-110	24-113	--	--
Phenol-d6	--	--	10-94	24-113
2-Fluorophenol	21-110	25-121	21-100	25-121
2,4,6-Tribromophenol	10-123	19-122	10-123	19-122
2-Chlorophenol-d4	33-110*	20-130*	--	--
	*Advisory			

If surrogate recoveries for a sample fall outside the control limits, qualify as follows. Specify the fraction that is being qualified (base/neutral or acid):

- If the recovery of any two surrogates per fraction are greater than the control limits, estimate **[J 142]** positive results for that fraction. (Determine that high bias was not due to interference with the surrogate compound only.)
- If the recovery of any two surrogates per fraction are less than the control limits but greater than or equal to 10%, estimate **[J 142]** positive results and **[UJ 142]** non-detected results for that fraction.
- If the recovery of one surrogate is greater than the control limits and the recovery for another surrogate is are less than the control limits but greater than or equal to 10% (in the same fraction), estimate **[J 142]** positive results and **[UJ 142]** non-detected results for that fraction.
- 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 possible interferences or misidentification before qualifying the data.

- If the raw data confirms the surrogate recoveries, no further action is necessary.
- If the raw data indicates that a surrogate was misidentified , assign the reason code **[804]**. Use professional judgment to assign qualifiers based on the extent of the problems identified.

3.4. MS/MSD Recovery

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

Review Items: Deliverable Section Number 6: Forms 3C/3D, Forms 6B/6C, 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: SS02 Exhibit B § 2.8; SS02 Exhibit D § 2, 4; GR01 Exhibit D.

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

Item 6-a-2 Check that Forms 3C/3D 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 8270B* %R Limit
	Water	Soil	Water/Soil
Phenol	12-110[42]	26-90[35]	5-112 or lab limits
2-Chlorophenol	27-123[40]	25-102[50]	23-134 or lab limits
1,4-Dichlorobenzene	36-97[28]	28-104[27]	20-124 or lab limits
N-Nitroso-di-n-propylamine	41-116[38]	41-126[38]	D-230 or lab limits
1,2,4-Trichlorobenzene	39-98[28]	38-107[23]	44-142 or lab limits
4-Chloro-3-methylphenol	23-97[42]	26-103[33]	22-147 or lab limits
Acenaphthene	46-118[31]	31-137[19]	47-145 or lab limit
4-Nitrophenol	10-80[50]	11-114[50]	D-132 or lab limit
2,4-Dinitrotoluene	24-96[38]	28-89[47]	D-191 or lab limit
Pentachlorophenol	9-103[50]	17-109[47]	14-176 or lab limit
Pyrene	26-127[31]	35-142[36]	52-115 or lab limit
	Frequency: 1/20 samples		**Frequency: 1/20 samples**

* SW-846 allows the laboratory to calculate their own recovery limits. Use laboratory limits if provided.

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:

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

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

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. Discontinue validation and 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 5B, decafluorotriphenylphospine (DFTPP) 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 determined criteria are established using standard materials. These criteria must be met in all circumstances.

Requirement Sources: SS02 Exhibit B § 2.8; SS02 Exhibit D § 2; GR01 Exhibit D.

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

Item 6-a-4 Check that Form 5B 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 of the daily DFTPP instrument performance check.

- If the samples were analyzed outside the 12-hour 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 DFTPP ion abundance criteria are met:

Table 3-4 DFTPP Ion Abundance Criteria

m/z	CLP-SOW	SW-846 8270B
51	30-80% of m/z 198	30-60% of m/z 198
68	<2.0% of m/z 69	<2.0% of m/z 69
69	Mass 69 relative abundance	-- --
70	< 2.0% of mass 69	< 2.0% of mass 69
127	25.0-75.0% of mass 198	40-60% of mass 198
197	<1.0% of mass 198	<1% of mass 198
198	Base Peak, 100% relative abundance	Base Peak, 100% relative abundance
199	5.0 to 9.0% of mass 198	5 to 9% of mass 198
275	10.0-30.0% of mass 198	10-30% of mass 198
365	> 0.75% of mass 198	> 1% of mass 198
441	Present, but < mass 443	Present, but < mass 443
442	40.0-110.0% of mass 198	>40% of mass 198
443	15.0-24.0% of mass 442	17-23% of mass 442

- If mass assignment is in error (e.g., m/z 199 is assigned as the base peak), reject **[R 139]** all associated data.

If ion abundance criteria are not met, use to following guidelines:

- M/z 68, 79, 197, and 441 and the m/z ratios for 198/199 and 442/443 are critical. If any one of these m/z abundances or ratios is not met, reject **[R 139]** all associated data.
- M/z 51, 127, and 275 are not as critical. If any one of these m/z abundances is slightly outside criteria (e.g., m/z 275 at 40% with limits of 10-30%) but the remaining criteria are met, the deficiency is minor. At a minimum, comment and assign reason code **[139]** to all applicable data.
- The relative abundance of m/z 365 indicates suitable instrument zero adjustment. If the relative abundance for m/z 365 is zero, minimum detection limits may be affected. Reject **[R 139]** all applicable data.
- If m/z 365 is present but less than the minimum abundance criteria, the deficiency is not as serious. Use professional judgment to assess the severity of the problem. Either reject **[R 139]** the data or comment and assign reason code **[139]** to all applicable data.

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 8B/8C, 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: SS02 Exhibit B § 2.8; SS02 Exhibit D § 2; GR01 Exhibit D.

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

Item 6-a-5 Check that Forms 8B/8C are present for all calibrations associated with sample analyses and that all samples and blanks 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.

The IS compounds are: 1,4-dichlorobenzene-d4, naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12, and perylene-d12. CLP specifies these compounds; SW-846 recommends them.

- 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 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 verification 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 so, reject **[R 143]** affected non-detected results and estimate **[J 143]** affected positive results for compounds quantitated using that IS.

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

Item 6-a-5 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 of the data or to request revised 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 1B/1C, Forms 6B/6C, Forms 7B/7C, COC record, sample extraction/preparation logs, 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: SS02 Exhibit B § 2.8; SS02 Exhibit D § 2; GR01 Exhibit D.

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

Item 6-b-1 Check that Forms 1B/1C 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 Forms 1B/1C 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 items apply 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.

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

where:

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

RT_{is} = Retention time of nearest internal standard

- If the RRT results are not within ±0.06 RRT units of the standard RRT qualify the associated result as estimated **[J 143]**.

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

SW-846

◊ The characteristic ions from the reference mass spectrum (the three ions of greatest intensity or any ions over 30% relative intensity if fewer than three such ions occur) maximize within one scan of those in the sample mass spectrum.

◊ The relative intensities of these ions must agree within ±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.

CLP Method

◊ All ions present in the standard mass spectrum at a relative intensity greater than 10% must be present in the sample spectrum.

◊ The relative intensities of these ions must agree within ±20% between the sample and standard spectrum.

◊ Ions present at greater than 10% in the sample spectrum but not present in the standard spectrum must be considered (reviewed for possible background contamination or presence of coeluting compounds) and accounted for.

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 has occurred and that the data was qualified appropriately.

3.8. Compound Quantitation and RDL

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 1B/1C, Forms 6B/6C, COC record, sample preparation/extraction logs, sample chromatograms and quantitation reports.

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

Requirement Sources: SS02 Exhibit B § 2.8; SS02 Exhibit C; SS02 Exhibit D § 2, 4; and GR01 Exhibit D.

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

Item 6-b-1 Using the Line Item Code from the COC record, ensure that the detection limits reported on Form 1B/1C match the required detection limits (RDLs) listed in SSO2, Exhibit C. Note that dilutions, percent solids, and extraction steps will impact the final RDLs reported.

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

- If an adequate explanation is not found, qualify all results reported as non-detected above the RDL as rejected **[R 213]**.

Evaluate Forms 1B/1C to ensure that no "E" qualifiers are present. If "E" qualifiers are present, ensure that another Form 1B/1C with a diluted sample analysis is present in the data package.

- If not, 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 (EPA/NIST/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 reference spectra cannot be found do not continue validation. 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 Notifications. Assign the reason code **[801]** to all applicable data.

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:

Water and water-miscible waste:

$$\frac{ug}{L} = \frac{A_X \times I_S \times V_t \times DF}{A_{is} \times RRF \times V_0 \times V_i}$$

where:

A_X = Response of the characteristic ion for the analyte in the sample, area counts.

I_S = Amount of internal standard injected, ng.

V_t = Volume of total extract, uL.

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.

V_i = Volume of extract injected, uL.

Sediment/soil, sludge, and waste:

$$\frac{ug}{Kg} = \frac{A_X \times I_S \times V_t \times DF}{A_{is} \times RRF \times V_i \times W_S \times P}$$

where:

A_X, I_S, A_{is}, RRF = Same as in water and water-miscible waste above.

V_t = Volume of total extract, uL.

DF = Dilution factor.

V_i = Volume of extract injected, uL.

W_S = Weight of sample extract, 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 1F, sample extraction/preparation logs, 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 National Institute of Standards and Technology (NIST) mass spectral library search and the identifications assessed by the data reviewer.

Requirement Sources: SS02 Exhibit B § 2.8; SS02 Exhibit D § 2; GR01 Exhibit D.

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

Item 6-b-2 Check that Form 1F 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]** all applicable data.

Verify that all TIC results are qualified by the laboratory 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]**.

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]** all applicable data.

Verify that no semivolatile 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 volatile compounds reported as target analytes in the volatile fraction are not reported as TICs in the semivolatile 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.)

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

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-methy-2-pentanone, 4-methyl-2-penten-2-one, and 5,5-dimethyl-2(5H)-furanone.

Item 6-b-5 Evaluate the 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 ±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 my be changed to a non-specific results (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: Forms 6B/6C, Forms 7B/7C, 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 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: SS02 Exhibit B § 2.8; SS02 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 12 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 used for validation of samples are as follows:

Table 3-5 Initial Calibration Criteria

Method	# of Standards	Concentration	RRF Limit	%RSD Limit
CLP	5	20, 50, 80, 120, and 160 ng/uL	RRF > 0.05	30%
SW-846 8270B	5	Low near but above established MDL; others should define the range of the expected concentrations	RRF > 0.05	15%

- 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.

Continuing Calibration

Item 6-c-2

The continuing calibration must include all target compounds and surrogates, must be analyzed at the beginning of each 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 3-6 Continuing Calibration Criteria

Method	Concentration	RRF Limit	%D Limit
CLP	50 ng/uL	RRF > 0.05	25%
SW-846 8270B	Mid-level	RRF > 0.05	20%

- 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:*

Initial Calibration

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.

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

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

where:

X_i = Each individual value used to calculate the mean

$\overline{X}$ = 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.

Continuing Calibration

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)

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

Review Items: Deliverable Section 6: Form 3C.

Objective: Quality control check sample data are generated to provide information on the accuracy of the analytical method and laboratory performance in the event that the MS/MSD percent recoveries are outside control limits.

Requirement Sources: SS02 Exhibit B § 2.8; SS02 Exhibit D § 2; 4 GR01 Exhibit D.

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

Item 6-a-2

QC Check Sample Frequency

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 3C 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.

QC Check Sample Percent Recovery

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

Table 3-7 Quality Control Sample Percent Recovery Limits

Spiking Compound	%R Limit
Phenol	5-112
2-Chlorophenol	23-134
1,4-Dichlorobenzene	20-124
N-Nitroso-di-n-propylamine	D-230
1,2,4-Trichlorobenzene	44-142
4-Chloro-3-methylphenol	22-147
Acenaphthene	47-145
4-Nitrophenol	D-132
2,4-Dinitrotoluene	D-191
Pentachlorophenol	14-176
Pyrene	52-115

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]**.

Item 6-d-3 Check that Forms 1B/1C 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:

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

- If the % R values are not within 5% of the reported values assign the reason code **[804]** to all results. Data validation may proceed because data will not be qualified based on control sample recoveries.

3.12. Blanks

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

Review Items: Deliverable Section Number 6: Form 4B, Method Blank Forms 1B/1C, 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: SS02 Exhibit B § 2.8; SS02 Exhibit D § 2; 4; GR01 Exhibit D.

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

The following table summarizes the blank criteria:

Table 3-7: Blank Criteria

Method	Frequency	Criteria
CLP	Per matrix, per concentration level, for each extraction batch (not to exceed 20 samples)	No contaminants should be present in the blanks. The method blanks should be analyzed on each GC/MS system used to analyze that set of associated samples.
SW-846 8270B	A method blank should be extracted with each extraction batch, when there is a change in reagents, and following any concentrated sample that has saturated ions from a compound.	The blank samples should be carried through all stages of the sample preparation and measurement steps (i.e., the method blank should be analyzed on the same instrument as the samples).

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

- In 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 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 of 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: Report the value unqualified.
- If an associated method blank exhibits gross contamination, reject **[R 149]** positive results for the affected compounds.

  Note: 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.

- 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:

Semivolatile Extractables:

◊ Phthalate Esters

Item 6-d-2 Check that Forms 1B/1C 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 1F 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 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 through 6-e-3

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, SS05 Exhibit B §2.9, and requirements of base methods cited in SS05-A 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.

Item 6-e-3 If GPC is performed, verify sample results are adjusted to account for the GPC cleanup.

- If sample results are not adjusted to account for GPC cleanup, issue a Non-Compliance Notification and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing data and incorporate any deficiencies in the Non-Compliance Report. Cease validation until a new data package is received with adjusted results and return the SDP to ASD.I

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 4, and SS05 Exhibit B §2.12.

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-SS02 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, Semivolatile Organics, Module SS02-B, March 28, 1997.

6. REVISION HISTORY

- The first draft of DA-SS02 was prepared by QuantaLex Inc.
- Final drafting of DA-SS02-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.

Attachment 1: Data Quality Assessment Report Template

SS02

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

Action Items:

Comments:

Verification/Validation Signature_____ Date:_____

Reviewer Signature_____ Date:_____
(*Validation Only*)