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e. Initial Calibration Response Factors (Form 3b)
f. Initial Calibration Ion Abundance Ratios (Forms 3c and 3d)
g. Calibration Verification (Forms 4a and 4b)
h. Retention Time Windows (Form 5)
i. Sample Relative Retention Times (Form 6a and 6b)
j. Initial precision and recovery (Forms 7a and 7b)
k. Ongoing precision and recovery (Forms 8a and 8b)
l. MS/MSD results and spiking level summary
m. Selected Ion Current Profiles (SICPs) for the Initial Calibration (5 concentrations run one time)
n. Mass spectrometer resolution demonstration SICPs for each analysis shift
o. GC resolution demonstration SICPs for each analysis shift
p. SICPs for one calibration standard for each analysis shift
q. SICPs for each sample and blank run

The GC/MS displays for the PCDD/PCDF analyses must include the standard and sample SICP chromatograms as specified in the analytical method with the date and time of analysis; the file name; sample number; and instrument I.D. number. The SICP mass chromatograms must also have the quantitation ion and confirmation ion displayed, integrated area and peak height listed for all peaks 2.5 times above background, and all peaks must show retention time at the maximum height.

The data packages for the radiochemical analyses for lead^{210}, cesium^{137}, and beryllium^{7} will include, as a minimum, a case narrative; printed report of analytical results with sample number, sample description, sample type, pCi/g dry, error estimates, data sheets for each sample analysis; detector calibrations; detector performance checks; blank analyses; lab replicate analyses; recovery factors; and calculated lower limits of detection for detects.

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Laboratory data qualifiers will be provided and defined by the analytical laboratory.

Electronic deliverable requirements will be provided to the laboratory if such deliverables are to be required of the laboratory. All electronic deliverables must match hard copy. An example format is shown in Table 8-1.

8.2 INDEPENDENT DATA REVIEW PROCESS

The second level of review and validation will be performed by a designee of the Contractor QA/QC Officer whose function is to provide an independent review of the data package and will include a review of laboratory performance criteria (Section 8.2.1) and sample specific criteria (Section 8.2.2).

In accordance with Section XI, Paragraph 71 of the AOC, the QA/QC methodologies specified in this QAPP are based on those set forth in SW-846. In addition, in accordance with Section B.3.c.11 of the SOW (Appendix 1 to the AOC) the QAPP was prepared taking into consideration the "Region II CERCLA Quality Assurance Manual", which contains Standard Operating Procedures (Region II SOPs) for use in an independent review of the laboratory data quality. The specific Region II SOPs listed below will be used for validation of sample data. Copies of the Region II SOPs are contained in Attachment 1 to the QAPP.

For volatile organics, semivolatile organics and pesticide/PCB data:

CLP Organics Data Review and Preliminary Review SOP HW-6, Revision 8, 1/92,

For metals and cyanide data:

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Evaluation of Metals Data for the Contract Laboratory Program SOP HW-2, Revision 11, 1/92,

and for the PCDD/PCDF data:

Polychlorinated Dibenzodioxins/Polychlorinated Dibenzofurans Data Review SOP No. 11, Revision 2, June 1993.

However, these SOPs are specifically designed for the QA/QC methodologies and analytical methods specified in the EPA Contract Laboratory Program Statements of Work rather than those in SW-846. Therefore, the Quality Control limits specified in the analytical methods will replace the Control limits in the SOPs for validation. If there is a quality control sample or measure specified in the SOPs that is not required when using the analytical methods, the response by the validator in completing the SOP checklist will be "not applicable". If a validation question in the Region II SOP checklist refers to similar processes (in CLP versus the analytical method) with somewhat differing protocols, such as calibration requirements, the validation question will be evaluated and answered with respect to the analytical method requirements in place of responding "not applicable".

In completing the validation checklists contained within the Region II SOPs, the questions on whether or not a Sample Management Office (SMO) checklist, an SMO Contract Compliance Screening (CCS) checklist, Record of Communication, Trip Report, Contract Non-Compliance - SMO Report or CLP Re-Analysis Request/Approval Record are included are considered "not applicable". The "Case Number and/or SAS number" shall refer to the OCC designation for the Sample Delivery Group analyzed. The sample Chain-of-Custody forms shall serve as the Traffic Report Forms.

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Upon completion of the data validation; the data packages, the completed assessment checklists, the telephone record log, data summary sheets, data assessment narratives, and the data assessment checklists shall be filed in the OCC files rather than mailed to the Regional Sample Control Center, the Regional DPO, Sample Management Office (SMO), the laboratory, the Monitoring and Management Branch or any of the specified EPA addresses listed in the Region II SOPs.

Validation of sample data from SW-846 methods for which no Region II SOPs are applicable and for sample data from non-SW-846 methods will be as specified in Sections 8.2.1 and 8.2.2.

The validation results will be utilized to provide an evaluation of the overall laboratory performance and will allow evaluation of:

- Accuracy through evaluation of spike sample recoveries, LCS analyses, and surrogate spike recoveries where specified in Section 3.0

- Precision of analysis through evaluation of matrix duplicate or matrix spike duplicate analytical results

- Representativeness through adherence to sampling procedures described in Section 4.0 of this QAPP and in respective sections of the FSP and field SOPs

- Comparability through evaluation of sample-specific reporting limits, units of measure, and adherence to specified analytical methodology

- Completeness through evaluation of the overall field completeness and the overall analytical completeness

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- Problems with analyses noted in the Case Narrative

As part of the data validation process, the following validation qualifiers and their meanings will be used:

U Non-detect - The analyte was analyzed for, but was not detected above the reported sample quantitation limit.

J Estimated value - The analyte was positively identified; but the associated numerical value is the approximate concentration of the analyte in the sample.

NJ The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.

UJ Estimated non-detect - The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.

R The sample results are rejected. Due to significant QA/QC problems the analysis is invalid and provides no information as to whether the analyte is present or not. Once the data are flagged with R, any further review or consideration is unnecessary.

If no determination of the overall bias of a result qualified as estimated can be made, the result will be flagged with J. If the data reviewer can determine the overall bias

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for sample data qualified as estimated, the data reviewer will qualify the sample result as either an estimated minimum value (JL) or an estimated maximum value (JH).

8.2.1 Laboratory Performance Criteria

Initially, 100 percent of the data packages from each laboratory for each analysis type will be reviewed for the following laboratory performance criteria:

- Initial Calibrations
- Continuing Calibration Verifications
- Laboratory Control Samples, if applicable
- Result Calculations

Ten percent of the data on the summary forms for each data package will be checked back to the raw data for potential calculation errors, transcription errors and data transfer errors. If significant errors are found, an additional ten percent of the data type for which errors were found will be checked. If similar errors are still found, 100 percent of the data type for which the error was found will be checked.

If the initial validation efforts indicate that no significant problems are being encountered with respect to the laboratory performance criteria for a given laboratory, OCC may request of EPA that review of these criteria on the remaining data not be required.

The Region II SOPs will be used for validation of the laboratory performance criteria for the volatile organics, semivolatile organics, pesticide/PCBs, metals and cyanide, and PCDDs/PCDFs analyses as discussed in Section 8.2 above. Validation and qualification of the laboratory performance criteria for the chlorinated herbicides analyses, total

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extractable petroleum hydrocarbons analyses, radiochemical analyses and analyses of other non-SW-846 analyses are specified below.

The validation protocols for PCDDs/PCDFs in Region II SOP No. 11 require that an EPA supplied PE sample containing 2,3,7,8-TCDD and the tetra- through octachloro dioxins and furans, an EPA supplied PE interference fortified blank, and an EPA supplied PE blank (i.e., field blank consisting of uncontaminated sand or soil) be submitted to the laboratory for analysis at the frequencies specified in Table 9-2. In order for these PE samples to be available for submittal to the laboratory by the field sampling team, EPA will be provided a minimum of two weeks notice of the need for the PE samples. If the PE samples are unavailable from EPA within two weeks of notification, a laboratory control sample containing 2,3,7,8-TCDD and the tetra- through octachloro dioxins and furans will be substituted for the PE sample, the laboratory method blank will be substituted for the field blank, and the PE interference fortified blank will not be required. The Region II SOP No. 11 will be modified accordingly during validation.

8.2.1.1 Chlorinated Herbicides

The chlorinated herbicide analyses will undergo validation for the criteria listed below. Any significant problems identified in the case narrative will be evaluated during the validation process.

1. Calibration - Raw data and summary sheets for both initial and continuing calibrations will be included within the data packages. All standard retention times must fall within the retention time windows established per the analytical method. If the retention time criteria are not met for a standard, beginning with the samples following the last in-control standard, check to see if the chromatograms contain peaks within an expanded window surrounding

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the expected retention times. If no peaks are found, and the surrogate (s) is visible, non-detects are valid. If peaks are present and cannot be identified through "pattern recognition" or a consistent shift in standard retention times, reject all affected compound results.

In the initial calibration, the Relative Standard Deviation (RSD) for each analyte's calibration factors will be reviewed against a limit of # 30 percent. Associated sample data for those analytes with %RSD > 30 will be qualified as estimated. Acceptable calibration verification (continuing calibration) is determined by satisfactory analyses of standard calibration mixtures for the following two criteria: Retention times for analytes contained in the respective standard solutions are within the appropriate retention time windows and the %D between the true analyte concentrations and the measured analyte concentrations are less than or equal to 25.0 percent. In the case where a standard solution analysis does not satisfy the specified control criteria, data following the last in-control standard to the next in-control standard are potentially affected. Associated sample data for detected analytes will be qualified as estimated if the reported quantitation was performed on a column which was out of control. For associated non-detect sample data, sample data will be qualified as estimated if the reported quantitation was performed on a column which was out of control and has a potentially low bias. Calibration factors and %RSDs for the calibrations in ten percent of the data packages will be checked back to the raw data.

2. Laboratory Control Sample - The LCS data will be reviewed against limits of 80 to 120 percent. If the percent recovery is $ 50% and less than 80%, qualify associated data as estimated. If the percent recovery is > 120%, qualify associated positive data as estimated. Reject associated sample data if the percent recovery is < 50%.

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3. Compound Quantitation and Reported Detection Limits - Data for one or more detected target analytes for samples in the data packages will be recalculated from the raw data to check for calculation and transcription errors.

8.2.1.2 Total Extractable Petroleum Hydrocarbons

The TEPH analyses will undergo validation for the criteria listed below. Any significant problems identified in the case narrative will be evaluated during the validation process.

1. Calibration - Raw data and summary sheets for both initial and continuing calibrations will be included within the data packages. In the initial calibration, the RSD for the calibration factors will be reviewed against a limit of # 30 percent. Associated sample data for those analytes with %RSD > 30 will be qualified as estimated.

   The %D between the true concentration and the measured concentration in the CCV standard will be reviewed against a limit of ± 25 percent. In the case where a standard solution analysis does not satisfy the specified criteria, data following the last in-control standard to the next in-control standard are potentially affected. Associated detected sample data will be qualified as estimated and associated non-detected sample data will be qualified as estimated if a low bias is determined to be present. Calibration factors and %RSD for the calibrations in ten percent of the data packages will be checked back to the raw data.

2. Compound Quantitation and Reported Detection Limits - Data for one or more detected target analytes per sample for ten percent of the data packages

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will be recalculated from the raw data to check for calculation and transcription errors.

8.2.1.3 Radiochemical Data (^{210}Pb, ^{137}Cs, ^{7}Be)

Data validation will consist of the following as defined by the analytical methodologies:

1. Detector Calibrations - Calibration documentation for each detector used including identification of detection, analyst's initials, date and time of calibration, name and activity and date certified of NBS traceable standard used to check detector calibration, counts per minute (C/M) observed, time of count, and calculated efficiencies will be reviewed.

2. Detector Performance Checks - Performance check results for each detector used including copies of control charts and logs comprising check source results and allowable limits for efficiency control for both the alpha and gamma spectrometry systems and blank results for background control for the alpha system will be evaluated for compliance with the method.

8.2.1.4 Other Analytes

Data validation will consist of the applicable portions of the following as defined by QA/QC sample specifications contained in this QAPP and the analytical methodology:

- Evaluation of spike recoveries (control sample spikes) and duplicate analysis precision (laboratory duplicates) with data outside of the accuracy and precision limits specified in the analytical methods or Section 4 qualified as estimated, ⅠJⅠ (or rejected, ⅠRⅠ, if in the professional judgment of the validator the data are unusable).

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