State of Connecticut

Department of Energy and Environmental Protection

Recommended Reasonable Confidence Protocols

Quality Assurance and Quality Control Requirements

Pesticides by Method 8081, SW-846

Version ~~2~~3.0

Month 2023

Written by the Connecticut ~~DEP~~DEEP QA/QC Workgroup

| Revision | Comments | Date |
|---|---|---|
| 1.0 | First version for publication | 7/11/2005 |
| 2.0 | Final version based upon public comments | July 2006 |
| 3.0 | Updates to reflect CAM method updates to improve consistency between different states. | Month 2023 |

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## ACRONYM LIST

| ACRONYM | DEFINITION |
|---|---|
| BHC | Benzene hexachloride |
| CASN | Chemical Abstracts Service Number |
| CCV | Continuing calibration verification |
| CF | Calibration factor |
| %D | Percent difference or percent drift |
| DCB | Decachlorobiphenyl |
| DDD | Dichlorodipheyldichloroethane |
| DDE | Dichlorodiphenylethane |
| DDT | Dichlorodiphenyltrichloroethane |
| DEEP | CT Department of Energy and Environmental Protection |
| ECD | Electron capture detector |
| ELCD | Electrolytic conductivity detector |
| GC | Gas chromatograph |
| GC/MS | Gas chromatograph/mass spectrometry |
| ICV | Initial calibration verification |
| LCS | Laboratory control sample |
| LLOQ | Lower limit of quantitation |
| MS | Matrix spike |
| MSD | Matrix spike duplicate |
| MSE | Microscale solvent extraction |
| NA | Not applicable |
| NAPL | Non-aqueous phase liquid |
| PCBs | Polychlorinated biphenyls |
| PFE | Pressurized fluid extraction |
| PTFE | Polytetrafluoroethylene |
| QA | Quality assurance |
| QC | Quality control |
| r/r2 | Correlation coefficient/determination |
| RCP | Reasonable Confidence Protocol |
| RF | Response factor |
| RL | Reporting limit |
| RPD | Relative percent difference |
| RSR/RSRs | Remediation Standard Regulations |
| %RSD | Percent relative standard deviation |
| SPE | Solid-phase extraction |
| TCMX | Tetrachloro-m-xylene |
| µg/kg | micrograms per kilogram |
| µg/L | micrograms per liter |

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# *1.0 Quality Assurance and Quality Control Requirements for SW-846 Method 8081*

## *1.1 Method Overview*

Method 8081 is gas chromatography ("GC") procedure used to determine chlorinated pesticides in a variety of matrices including waters, soils, sediments, wastes, etc. This procedure requires an experienced GC analyst familiar with the QA/QC requirements of the method. The sample introduction procedure requires the use of a solvent extraction procedure. (see Table 1.0 for applicable extraction methods). ~~All method references are to the latest promulgated version of the method found in Test Methods for Evaluating Solid Waste, SW-846.~~

Open-tubular, capillary columns are employed with electron capture detectors ("ECD") or electrolytic conductivity detectors ("ELCD"). When compared to packed columns, these fused-silica, open-tubular columns offer improved resolution, better selectivity, increased sensitivity, and faster analysis. The target analytes may be determined with ~~either a single or~~a dual-column chromatographic system. The method also may be applied to other matrices such as oils and wipe samples, if appropriate sample extraction procedures are employed.

All method references are to the latest promulgated version of the method found in Test Methods for Evaluating Solid Waste, SW-846.

## *1.2 Summary of SW-846 Method 8081*

### 1.2.1 Sample Extraction and Cleanup

Samples for analysis by SW-846 Method 8081 require extraction by one of the following methods. The use of a hydrophilic solvent mixture, either 1:1 Acetone/Hexane or 1:1 Acetone/Methylene chloride, is recommended for soil and sediment samples.

**Table 1.0: Extraction Methods**

| SW-846 Method | Matrix | Description |
|---|---|---|
| 3542 | Air Samples | Extraction of Analytes Collected Using a Modified Method 5 Sampling Train |
| ~~3510C~~3510 | Aqueous | Separatory Funnel Liquid-Liquid Extraction |
| ~~3520C~~3520 | Aqueous | Continuous Liquid-Liquid Extraction |
| 3511 | Aqueous | Organic Compounds in Water by Microextraction |
| 3535 | Aqueous | Solid-Phase Extraction ("SPE") |
| ~~3540C~~3540 | Soil/Sediment | Soxhlet Extraction |
| 3541 | Soil/Sediment | Automated Soxhlet Extraction |
| ~~3545A~~3545 | Soil/Sediment | Pressurized Fluid Extraction ("PFE") |
| 3546 | Soil/Sediment | Microwave Extraction |
| 3570 | Soil/Sediment | Microscale Solvent Extraction ("MSE") |
| ~~3550C~~3550 | Contaminated Solids[1] | Ultrasonic Extraction |
| ~~3580A~~3580 | NAPL | Solvent Dilution |

[1]Sonication may only be used for the extraction of highly contaminated (free product) non-soil/sediments (debris). Any other use of ultrasonic extraction is not allowed.
[2]Air drying of samples is not allowed.

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Extracts may be cleaned up, as required, by any of the following methods prior to gas chromatography/mass spectrometry ("GC/MS") analysis by SW-846 Method 8081.

**Table 2.0: Extraction Clean-up Methods**

| SW-846 Method | Description |
|---|---|
| 3600 | General Cleanup Selection |
| 3610 | Alumina |
| 3620 | Florisil |
| 3630 | Silica Gel |
| 3640 | Gel Permeation Chromatography |
| 3660 | Sulfur Cleanup |

### 1.2.2 GC Analysis

The chlorinated pesticides are extracted from the sample using the appropriate method. The solvent extract is concentrated and then aliquots are injected onto the GC column in the gas chromatograph. The ~~gas chromatograph (~~GC~~)~~ oven is temperature programmed to facilitate separation of the analytes which are then detected by an ECD or ELCD interfaced to the column

Preliminary identification of target analytes is accomplished by comparing the retention time of the chromatographic peaks of the sample to known pesticides analyzed under the exact same conditions. Confirmation is accomplished either by analysis of the same extract on a dissimilar column, again comparing the retention times of the chromatographic peaks of the sample to known pesticides analyzed under the exact same conditions, or by using at least one other independent qualitative technique such as GC/MS. Quantitation is accomplished by constructing a minimum 5-point calibration curve of pesticide concentration versus peak area. ~~Confirmation~~Identification of pesticides on a single column must be confirmed on a second column or must be supported by at least one other independent qualitative technique. Although a dual-column option may satisfy this requirement, due caution should be exercised when highly contaminated samples are processed or during times of high sample throughput. Dual column c~~C~~onfirmation is not required in the case where pesticides are not detected above their specific reporting limit/lower limit of quantitation ("RL/LLOQ").

## *1.3 Method Interferences*

Refer to SW-846 Methods 3500 ~~(Sec. 3.0, in particular),~~ 3600, and 8000 for a detailed discussion of interferences. Interferences co-extracted from the samples will vary considerably from matrix to matrix. While general cleanup techniques are referenced or provided as part of this method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation. Sources of interference in this method can be grouped into four broad categories.

- Contaminated solvents, reagents, or sample processing hardware;
- Contaminated GC carrier gas, parts, column surfaces, or detector surfaces;
- Non-target compounds simultaneously extracted from the sample matrix which cause a detector response; and
- Co-elution of target analytes

An in-depth discussion of the causes and corrective actions for all of these interferences is beyond the scope of this guidance document. A brief discussion of the more prevalent interferences is presented below.

### 1.3.1 Chemical Contaminants

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Major contaminant sources for SW-846 Method 8081 include, but are not limited to, plastics, impurities in laboratory chemicals, contaminated laboratory ware, etc. The use of non-polytetrafluoroethylene ("PTFE") thread sealants, plastic tubing, or flow controllers with rubber components should be avoided, since such materials may contaminate the analytical system.

Analysis of blanks provides information about the presence of contaminants. When potential interfering peaks or high levels of target compounds are detected in blanks, the laboratory should try and find the source of the contamination and eliminate it. **Subtracting blank values from sample results is not permitted.** Any method blank exceedances should be fully documented in the laboratory report narrative.

Interferences by phthalate esters introduced during sample preparation can pose a major problem in chlorinated pesticide determinations by SW-846 Method ~~8081A.~~ 8081. Common flexible plastics contain varying amounts of phthalate esters, as plasticizers, which are easily extracted or leached from such materials during laboratory operations. Interferences from phthalate esters can best be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination. These materials may be removed prior to analysis using Method 3640 (Gel Permeation Cleanup) or Method 3630 (Silica Gel Cleanup).

### 1.3.2 Cross-contamination/ Carryover

Cross-contamination can occur when any sample is analyzed immediately after a sample containing high concentrations of pesticides or other compounds which cause a detector response, such as ~~PCB's.~~polychlorinated biphenyls ("PCBs"). Syringes on the autosampler may also become contaminated in the same manner. If a high sample is inadvertently analyzed, the system must be demonstrated to be clean by analysis of solvent blanks. Laboratories should be aware that carryover from high boiling point compounds may not appear until a later run (ghost peaks).

### 1.3.3 Sulfur Interferences

The presence of elemental sulfur (S) will result in broad peaks that interfere with the detection of early-eluting chlorinated pesticides. Sulfur contamination should be expected with sediment samples and can be removed ~~through the use of~~using SW-846 Method 3660.

### 1.3.4 Co-elution

As described in ~~Section 3.8 and 3.9 of~~ SW-846 Method 8081, co-elution among the many target analytes or other compounds can cause interference problems. The GC analyst should experiment with varying chromatographic conditions to obtain the most efficient compound separation.

### 1.3.5 Special Precautions

DDT and endrin are easily degraded in the injection port. Breakdown occurs when the injection port liner is contaminated with high boiling residue from sample injection or when the injector contains metal fittings. The potential for DDT and endrin breakdown should be evaluated before samples are analyzed and at the beginning of each 12-hour shift as described in ~~Section 8.4.6 of~~ SW-846 Method 8081.

## *1.4 Quality Control Requirements for SW-846 Method 8081*

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## 1.41.1 Reporting Limits/Lower Limits of Quantitation for SW-846 Method 8081

The reporting limit ("RL"), or lower limit of quantitation ("LLOQ"), for a compound is dependent on the concentration of the lowest non-zero standard in the initial calibration, sample weight/volume, extraction procedure, and moisture content. Table 3.0 lists approximate RL/LLOQs for various matrices utilizing a GC with an electrolytic conductivity detector ("GC/ECD"). Electrolytic conductivity detectors will have slightly higher ~~reporting limits~~RL/LLOQs. Solid matrices in this table assume 100% solids.

**Table 3~~1~~.0: Typical Reporting Limits / Lower Limits of Quantitation**

| Matrix | Typical Reporting Limit |
|---|---|
| Water | 0.05 to 0.5 µg/L |
| Soil | 1.7 to 17 µg/kg |

Moisture content of soils and sediments will also raise the RL/LLOQ, as all results must be reported on a dry weight basis for these two matrices. Sample dilution or lower sample weight/volume will also cause the RLs/ LLOQs to be raised. It is the responsibility of the ~~environmental professional or EP to specify to the laboratory the detection limits required for the samples~~ data user, in concert with the laboratory, to establish the range and required RL/LLOQ for the target analytes to meet Remediation Standard Regulations ("RSR") criteria. To meet the ~~detection~~ limits, it may be necessary to modify the analytical method by using increased sample volume or mass. In such cases the modifications must be noted in the laboratory report narrative.

~~Sample container type, preservation requirements, and holding times for waters, soils, and sediments are presented in Table 2A of this document.~~

## 1.4.21 General Quality Control Requirements ~~*for Determinative Chromatography Methods*~~

This protocol is restricted to use by, or under the supervision of, analysts experienced in the use of GC instrumentation as a quantitative tool and skilled in the interpretation of gas chromatograms for pesticides.

Refer to SW-846 Method 8000 for general quality control ("QC") requirements for all chromatographic methods, including SW-846 Method 8081. These requirements ~~insure~~ensure that each laboratory maintain a formal quality assurance ("QA") program and records to document the quality of all chromatographic data~~.~~ and be certified by the Connecticut Department of Public Health for the analysis performed. QC procedures necessary to evaluate the GC system operation may be found in SW-846 Method 8000~~, Section 7.0~~, and include evaluation of retention time windows, initial and verification of instrument calibrations and chromatographic performance of sample analyses. Instrument quality control and method performance requirements for the GC system may be found in SW-846 Method 8081~~, Sections 8.0 and 9.0, respectively~~.

### ~~1.1.2 General Quality Control Requirements~~

The minimum requirements for a formal QA program include Initial Demonstration of ~~laboratory Proficiency~~ Capability ("IDOC"), ongoing analysis of standards and blanks to confirm acceptable continuing performance, and analysis of laboratory control samples ("LCS") and/ or matrix spikes ("MS") to assess ~~precision and~~ accuracy and LCS duplicates ("LCSD") and matrix spike duplicates ("MSD") to assess precision. The use of site-specific MS/MSD's is highly recommended. Evaluation of sample matrix effects on compound recovery is key to making ~~good~~ informed decisions. Percent recovery data from site-specific samples allow the environmental professional ("EP") to make informed decisions regarding contamination levels at the site. Batch MS/MSD results do not give any indication of site-specific matrix interferences or analytical problems related to the specific site matrices and are in general discouraged. Field, rinsate, or other blanks should not be used for MS/MSD's.

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~~1.4.3 Site Specific Matrix Spike (MS), Matrix Spike Duplicate (MSD) Samples~~

~~It is strongly recommended that site specific MS/MSD samples be analyzed from each site, and each matrix type sampled. Percent recovery data from site specific samples allow the EP to make informed decisions regarding contamination levels at the site. Batch MS/MSD results do not give any indication of site specific matrix interferences or analytical problems related to the specific site matrices and are in general discouraged. Non-site specific MS/MSD's should not be reported for the RCP's. Additionally trip blanks, field blanks, rinsate blanks, etc. should not be used for MS/MSD's.~~

Laboratories must document and have on file an ~~Initial Demonstration of Proficiency~~ IDOC for each combination of sample preparation and determinative method being used. An IDOC must be completed and documented when a method is initially started up, whenever a method is substantially modified, or new laboratory staff is trained to perform this method. These data must meet or ~~exceed~~ fall within the performance standards as presented in Section 1.4~~1.5~~ and Table 1A of this RCP. See ~~Section 8.4 of~~ SW-846 Method 8000 for the procedure. The IDOC must include the following elements provided in Table 4.0:

**Table ~~1.1~~4.0: IDOC Requirements**

| QC Element | Performance Criteria |
|---|---|
| Initial Calibration | Table 1A |
| Continuing Calibration | Table 1A |
| Method Blanks | Table 1A |
| Average Recovery | Table 1A |
| % Relative Standard Deviation | Table 1A |
| Surrogate Recovery | Table 1A |
| Internal Standards | Table 1A |

Because of the extensive analyte list and number of QC elements associated with the IDOC, it should be expected that one or more analytes might not meet the performance standards for one or more QC elements. The laboratory should make every effort to find and correct the problem and repeat the analysis. All non-conforming analytes along with the laboratory acceptance criteria should be noted in the ~~Initial Demonstration of Proficiency~~ IDOC data. This information should be kept on-file at the laboratory.

Laboratories are required to generate laboratory specific performance criteria for LCS compound recovery limits, matrix spike/matrix spike duplicate compound recovery and ~~precision~~ relative percent difference ("RPD") limits, and surrogate recovery limits. These limits must be ~~meet or exceed~~ equal to or fall within the limits specified in Table 1A.

## 1.4.~~2~~3 Specific QA/QC Requirements and Performance Standards for SW-846 Method 8081

Specific QA/QC requirements and performance standards for SW-846 Method 8081 are presented in Table 1A. Strict compliance with the QA/QC requirements and performance standards for this method, as well as satisfying other analytical and reporting requirements will provide the ~~environmental professional (~~EP~~)~~ with "Reasonable Confidence" regarding the usability of analytical data to support ~~DEP~~ environmental decisions. The concept of "Reasonable Confidence" is explained on the CT Department of Energy and Environmental Protection ("DEEP") website.

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While optional, parties electing to utilize these protocols will be assured that agency reviewers will, generally, accept "Reasonable Confidence" data~~, will be generally accepted by agency reviewers. In order to~~. To achieve "Reasonable Confidence" parties must:

1. Comply with the applicable QC analytical requirements prescribed in Table 1A for this test procedure;

2. Evaluate and narrate all protocol non-compliances and implement, as necessary, ~~compliance with~~required corrective actions and analytical response actions for all non-conforming analytical performance standards~~ prescribed in Table 1A for this test method~~; and

3. ~~Adopt the reporting formats and elements specified in Section 1.7 of this method.~~ Retain reported and unreported analytical data and information for a period of 10 years.

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**Table 1A: Specific QA/QC Requirements and Performance Standards for Method 8081**

| Required ~~QA~~QC Parameter Column 1 | Data Quality Objective Column 2 | Required Performance Standard Column 3 | Required Deliverable Column 4 | Required~~Recommended~~ Corrective Action Column 5 | Required Analytical Response Action Column 6 | Rationale for Changes (removed from final version) |
|---|---|---|---|---|---|---|
| Initial Demonstration of Capability ("IDOC") | Laboratory Analytical Accuracy & Precision | (1) Must be performed prior to using method on samples.<br>(2) Must be performed for each matrix.<br>(3) Must contain all target analytes.<br>(4) Must follow procedure in SW-846 8000. | No | Refer to SW-846 8000 and Section 1.4.2 of this protocol. | NA | Group accepted MA language with exceptions of references to specific method version and sections. |
| Retention Windows | Laboratory Analytical Accuracy | (1) Prior to or during an initial calibration when a new column is installed.<br>(2) Calculate according to Method 8000 ~~Section 7.6~~. | No | NA | NA | Removed references to specific method version and sections. |
| Endrin and DDT Breakdown | Laboratory Analytical Accuracy | (1) Before samples are analyzed and at the beginning of each 12-hour shift~~clock~~.<br>(2) % breakdown must be ≤15% ~~for each compound~~ and must be evaluated using peak areas ~~Section 8.4.6 of Method 8081~~. | Yes | Perform injection port/column maintenance. Recalibrate if necessary. | If criteria is not met, perform instrument maintenance, recalibrate instrument, and reanalyze samples.<br>~~Report exceedances in narrative~~ | Column 3: Removed references to specific method version and sections.<br><br>Column 6: Clarified instrument maintenance must be done and criteria met before reporting results. |
| Initial Calibration | Laboratory Analytical Accuracy | (1) Must be analyzed with dual columns at least once prior to analyzing samples, when initial calibration verification (ICV) or continuing calibration does not | No | (1) Recalibrate as required by method.<br>(2) If recalculated concentrations from the lowest calibration standard | Sample analysis cannot proceed without a valid initial calibration. Report non-conforming compounds (%RSD >20, r | Group accepted MA language.<br><br>Column 3, Item 1: Added |

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| Required ~~QA~~QC Parameter Column 1 | Data Quality Objective Column 2 | Required Performance Standard Column 3 | Required Deliverable Column 4 | Required~~Recommended~~ Corrective Action Column 5 | Required Analytical Response Action Column 6 | Rationale for Changes (removed from final version) |
|---|---|---|---|---|---|---|
| | | meet the performance standards, and when major instrument maintenance is performed.<br>(2) Minimum of 5 standards (or if non-linear regression used). ~~for single response pesticides. (Note 1)~~<br>(3) Low standard must be ~~at~~ ≤RL/LLOQ.<br>(4) % RSD ≤20%, r≤0.99 (linear regression), $r^2$≤0.99 (non-linear regression) for each single-component pesticide.<br>(5) If %RSD >20, linear or non-linear regression must be used.<br>(6) Must contain all single-component pesticides.<br>(7) Multi-component analytes: Analysis of a single standard at expected mid-point of calibration range.<br>(8) Calibration must be performed under the same conditions as the samples.<br>(9) If linear or non-linear regression used, verify the RL/LLOQ by recalculating concentrations in lowest calibration standard using the final calibration curve; recoveries must be 70-130%.<br>(10) If curves are used, curve must NOT be forced through origin. Must use additional standards as specified in Method 8000~~, Section 7.5~~. | | are outside of 70-130% recovery range, either:<br>(a) The RL/LLOQ limit must be reported as an estimated value or;<br>(b) The RL/LLOQ must be raised to the concentration of the next highest calibration standard that exhibits acceptable recoveries when recalculated using the final calibration curve. | > 0.99, $r^2$ >0.99) in laboratory report narrative. ~~If avg Rf or linear regression not used~~ If non-linear regression (i.e., quadratic equation) is used for calibration, this must be noted in the laboratory report narrative along with the compounds affected. | language requiring calibration of dual column for method clarification.<br><br>Column 3: Removed references to specific method version and sections. |

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|---|---|---|---|---|---|---|
| | | ~~6) Curves must be verified with independent ICV prior to sample analysis.~~ | | | | |
| Initial Calibration Verification ("ICV") | Laboratory Analytical Accuracy | (1) Immediately after each initial calibration.<br>(2) Concentration level near midpoint of curve.<br>(3) Prepared using standard source different than used for initial calibration.<br>(4) Must contain all single-component pesticides.<br>(5) Percent recoveries must be between 80-120% for each target analyte. | No | Locate source of problem; recalibrate if >10% of all analytes are outside of criteria. | If recovery is outside of 80-120% for any analyte, report non-conforming compounds in laboratory report narrative. | Group accepted MA language. |
| Continuing Calibration Verification ("CCV~~AL~~") | Laboratory Analytical Accuracy | (1) Prior to samples, every 12 hours or every ≤20 field samples, whichever is more frequent, and at the end of analytical sequence<br><br>NOTE: if internal standard calibration used, the CCV at the end of the analytical sequence is not required.<br><br>(2) Concentration near midpoint of curve.<br>(3) Must contain all single-component pesticides.<br>(4) Multi-component~~response~~ analytes~~pesticides~~ must be verified with a one-point standard within 12 hours of being detected in a sample.<br>(5) %D must be ≤20~~15~~ for each target analyte. | No | (1) Perform instrument maintenance, reanalyze CCV~~AL~~ and/or recalibrate as required by method.<br>(2) Reanalyze "associated samples" if beginning or ending CCV~~AL~~ exhibited low response ~~and associated pesticides not detected in samples.~~.<br>(3) Reanalyze "associated samples" if beginning or ending CCV~~AL~~ exhibited high response and associated pesticides were detected in the "associated samples".<br><br>NOTE: "Associated samples" refers to all samples analyzed since the last acceptable CCV. | Report non-conforming compounds (%D>20) and associated samples in laboratory report narrative. | Column 3, Item 2: Group accepted MA language with the exception of the concentration requirement for the CCAL. Group maintained RCP language. |

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|---|---|---|---|---|---|---|
| | | (6) Verify that all analytes fall within retention time windows. (7) Area count of internal standard in CCV must be within ±50% of the average area count in the associated initial calibration. | | | | |
| Method Blank ("MB") | Laboratory Method Sensitivity (contamination evaluation) | (1) Extract with every batch or every ≤20 field samples, whichever is more frequent (2) Matrix-specific (e.g., water, soil) (3) Target analytes must be ≤½ ~~<~~RL/LLOQ. | Yes | (1) If concentration of contaminant in sample is <10x concentration in blank, locate source of contamination; correct problem; re-extract and re-analyze method blank and associated samples. (2) No corrective action required if concentration of contaminant in sample is >10x concentration in blank or if contaminant not detected in sample.<br><br>~~Locate source of contamination and correct problem. Reanalyze method blank.~~<br>~~Re-extract samples if method blank contaminated~~ | (1) If sample re-extraction is not possible, report non-conformance in laboratory report narrative. (2) ~~All results for compounds present in method blank must be "B" flagged if detected in samples associated with the method blank.~~If contamination of method blanks is suspected or present, the lab, using a "B", or some other convention, should qualify the sample results. Blank contamination should also be documented in laboratory report narrative. (3) If re-extraction is performed within holding time ~~report only compliant data. If re-extraction~~ and yields acceptable method blank results, the lab may report | Group accepted MA language. |

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|---|---|---|---|---|---|---|
| | | | | | results of the re-extraction only.<br>(4) If the re-extraction is performed outside of holding time, the lab must report all sets of data. | |
| Laboratory Control Sample ("LCS") | Laboratory Method Accuracy | (1) Extracted with every batch or every ≤20 field samples whichever is more frequent.<br>(2) Concentration level near midpoint of curve.<br>(3) Must contain all single-component pesticides.<br>(4) Matrix-specific (e.g., water, soil)<br>(5) ~~Laboratory determined~~ Percent recoveries must be between 40-140% ~~except for difficult analytes, which must be between 30-140% recovery..~~<br>(6) Must be prepared in a water-miscible solvent (e.g., acetone, methanol).<br>(7) Standard source different from initial calibration source. | Yes | (1) Locate source of problem; re-extract and re-analyze LCS and associated samples if >10% of all analytes are outside of criteria.<br>~~Recalculate the percent recoveries~~<br><br>~~Reanalyze the LCS~~<br>(2) If ≤10% of compounds are outside of the acceptance criteria, re-extraction is not required as long as recoveries are >10%.<br>(3) If >10% of compounds are above~~outside~~ the acceptance criteria (>140%), re-extraction is not required if affected compounds were not detected in associated samples.<br>~~Re-extract LCS and samples if >10% compounds outside acceptance criteria and no MS/MSD with acceptable criteria~~<br>(4) If MS/MSD in same batch, compare to determine if the problem isolated to LCS. | (1) If re-extraction is not possible, report non-conformances in laboratory report narrative.<br>(2) If recovery is outside of 40-140% for any analyte, report non-conforming compounds in laboratory report narrative.<br>~~2) Individual laboratories must identify and document problem analytes that routinely fall outside the limits. Any exceedances must be noted in narrative. Data to support laboratory problem compounds kept on file at lab for review during audit~~(3) If re-extraction is performed within holding time and yields acceptable LCS results, the lab may report results of the re-extraction only. ~~report only compliant data.~~ | Column 3, Item 5: Group decided to maintain "lab-determined" limit language within body of text under IDOC.<br><br>Removed language referencing "difficult analytes" as that language is not applicable to this method. |

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| Required ~~QA~~QC Parameter Column 1 | Data Quality Objective Column 2 | Required Performance Standard Column 3 | Required Deliverable Column 4 | Required~~Recommended~~ Corrective Action Column 5 | Required Analytical Response Action Column 6 | Rationale for Changes (removed from final version) |
|---|---|---|---|---|---|---|
| | | | | | (5) If the re-extraction is performed outside of holding time and yields acceptable LCS results, the lab must report all sets of data. | |
| LCS Duplicate ("LCSD") | Laboratory Analytical Accuracy & Precision | (1) Extracted with every batch or every ≤20 field samples, whichever is more frequent.<br>(2) Concentration level near midpoint of curve.<br>(3) Must contain all single-component pesticides.<br>(4) Matrix-specific (e.g., water, soil).<br>(5) Percent recoveries must be between 40-140% for target analytes.<br>(6) RPDs must be ≤20% for waters and ≤30% for solids.<br>(7) Must be prepared in a water-miscible solvent (e.g., acetone, methanol). | Yes | (1) Locate source of problem; re-extract and re-analyze LCS and associated samples if >10% of all analytes are outside of recovery acceptance criteria.<br>(2) If ≤10% of compounds are outside of the recovery acceptance criteria, re-extraction is not required as long as recoveries are >10%.<br>(3) If >10% of compounds are above the recovery acceptance criteria (>140%), re-extraction is not required if affected compounds were not detected in associated samples. | (1) If sample re-extraction is not possible, report non-conformance in laboratory report narrative.<br>(2) if recovery is outside of 40-140% for any analyte or if RPD is outside of criteria, report non-conforming compounds in laboratory report narrative.<br>(3) If re-extraction is performed within holding time and yields acceptable LCS results, the lab may report results of the re-extraction only.<br>(4) If the re-extraction is performed outside of holding time and yields acceptable LCS results, the lab must report all sets of data. | Group accepted MA language. |
| Matrix Spike / Matrix Spike Duplicate ("MS/MSD") (Site Specific) | Method Accuracy & Precision in Sample Matrix | (1) Every ≤20 field samples (at discretion of lab or at request of data user).<br>(2) Must contain all single-component ~~response~~ pesticides. | Yes (ONLY when requested by data user~~*If requested by EP~~) | Check LCS; If recoveries are acceptable in LCS, narrate non-conformance.<br>~~If compounds out compare to LCS; if LCS recoveries in note in narrative; if LCS~~ | Note non-conformances ~~outliers~~ in laboratory report narrative. | Group accepted MA language. |

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| Required ~~QA~~QC Parameter Column 1 | Data Quality Objective Column 2 | Required Performance Standard Column 3 | Required Deliverable Column 4 | Required~~Recommended~~ Corrective Action Column 5 | Required Analytical Response Action Column 6 | Rationale for Changes (removed from final version) |
|---|---|---|---|---|---|---|
| | | (3) ~~Laboratory determined~~ Percent recoveries between 30-150%<br>(4) Matrix-specific (e.g., aqueous, soil).<br>(5) Concentration level near midpoint ~~lower part~~ of curve.<br>(6) RPDs ≤20% for waters and ≤30% for solids.~~for single response pesticides~~<br>(7) RPD's ≤30% for multi-component pesticides.<br>(8) Must be prepared in a water-miscible solvent (e.g., acetone, methanol).<br>(9) Field blanks, trip blanks, etc. cannot be used for MS/MSD's | | ~~compounds out note in narrative probable lab error~~ | | |
| Surrogates | Method Accuracy in Sample Matrix | (1) Minimum of 2 surrogates,~~compounds~~ one that elutes at beginning of GC run and one that elutes at end of GC run. ~~across retention times of GC run~~Recommended surrogates: TCMX and DCB.<br>(2) ~~Recovery limits lab generated~~ Percent recoveries must be between 30-150% for both surrogates~~compounds~~ on both columns.<br>~~3) Labs must develop own in-house limits, which fall within 30-150% limits.~~ | Yes (report surrogate recoveries from both columns) | (1) If both surrogates outside limits on one column only reanalyze sample.<br>(2) If the same surrogate is outside of limits on both columns:<br>(a) Re-extract the sample if surrogate recoveries are low and there is no chromatographic interference.<br>(b) Re-extract the sample if surrogate recoveries were detected and high pesticides were detected in the sample.<br><br>NOTES:<br>(i) If surrogate recoveries are high and target analytes are | (1) Report recoveries outside of 30-150% in laboratory report narrative.<br>(2) If re-extraction ~~or reanalysis confirms matrix interference~~ yields similar surrogate non-conformances, the lab must report results of both the initial extraction and the re-extraction ~~report all results.~~<br>(3) If re-extraction is performed within holding time and yields acceptable surrogate recoveries, the lab may report results of the re-extraction only. | Group accepted MA language.<br><br>Column 3, Item 3: Group decided to maintain "lab generated" limit language within body of text under IDOC. |

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|---|---|---|---|---|---|---|
| | | | | not detected in sample, re-extraction is not required.<br>(ii) If chromatographic interference is present and surrogate recovery would cause rejection of data (i.e., <10%), reanalyze sample on dilution.<br>(iii) If a surrogate is diluted to a concentration below that of the lowest calibration standard, re-extraction and/or reanalysis is not required.~~no recovery criteria~~ | (4) If re-extraction is performed outside of the holding time and yields acceptable surrogate recoveries, the lab must report results of both the initial extraction and re-extraction.<br>(5) If the sample is not re-extracted due to chromatographic interference, the lab must provide the chromatogram in the data report. | |
| Internal Standards (if employed) | Laboratory Analytical & Method Accuracy in Sample Matrix | (1) Minimum of 1 Recommended internal standard: DCB.<br>(2) Area counts in samples must be between 50-200% of the area counts in the associated continuing calibration standard.<br>(3) Retention times of internal standards must be within ±30 seconds of retention times in associated continuing calibration standard. | No | If internal standard is outside of limits, reanalyze sample unless chromatographic interference present<br><br>NOTE: If chromatographic interference is present and internal standard area would cause rejection of data (i.e., <20%), reanalyze sample on dilution. | (1) Report non-conformances in laboratory report narrative. Include actual recovery of internal standard and provide summary of analytes quantitated of using the internal standard.<br>(2) If reanalysis yields similar internal standard non-conformances, the lab must report results of both analyses.<br>(3) If reanalysis is performed within holding time and yields acceptable internal standard recoveries, the lab may report results of the reanalysis only.<br>(4) If reanalysis is performed outside of the | Group accepted MA language. |

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|---|---|---|---|---|---|---|
| | | | | | holding time and yields acceptable internal standard recoveries, the lab must report results of both analyses.<br>(5) If sample is not reanalyzed due to chromatographic interference, the lab must provide the chromatogram in the data report. | |
| Identification and Quantitation | Inter-laboratory Consistency | (1) Peak area is the expected default to be used for quantitation of pesticides under most circumstances. Regardless, if peak area or peak height is used, the same method used for quantitation of samples must also be used for calibration standards.<br>(2) The lab must ~~should~~ use the average calibration factor, response factor, linear or non-linear regression curve generated from the associated initial calibration for quantitation of each single-component pesticide.<br>(3) Secondary column analysis: Laboratory must utilize a second dissimilar column to confirm positive results ~~above the RL~~. The lab must report the higher of the two results. All required QC parameters (e.g., calibrations, LCSs, etc.) must | No | If the RPD between the dual column results is >40%:<br>(i) determine potential interference;<br>(ii) re-analyze sample on dilution; or<br>(iii) additional sample cleanup techniques may be warranted; or<br>(iv) re-extract sample and re-analyze.<br><br>~~N/A~~ | When the RPD between the dual column results is:<br>(1) <40% and there is no obvious matrix interference, the higher value shall be reported.<br>(2) <40% and there is obvious matrix interference, the lower value shall be reported and the results shall be flagged with a "P".<br>(3) >40% and there is no obvious matrix interference, the higher value shall be reported and the results shall be flagged with a "P".<br>(4) >40% and there is obvious matrix interference, the lower value shall be reported and the results shall be flagged with a "P".<br>~~If the RPD between the results for the two~~ | Group accepted MA language with the exception of:<br><br>(1) MA language regarding significant figures because CT does not advise reporting values <RL/LLOQ; and<br><br>(2) The proposed language in Columns 5 and 6.<br><br>Column 5: Group added corrective action language for clarification. |

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|---|---|---|---|---|---|---|
| | | be met on secondary column as well.<br>(4) For multi-~~response~~ component pesticides, quantitate as per ~~Section 7.6~~ Method 8081.<br>(5) Do no report concentrations below the RL/LLOQ.<br>~~Note: If a high RPD between the two columns can be definitely attributed to matrix interference, report the lower value and note in the narrative with an explanation.~~ | | | ~~columns exceeds 40%, the laboratory must flag the results with a "P" suffix and note in narrative.~~<br><br>All non-conformances must be noted in the laboratory report narrative.<br><br>If avg. CF or RF or linear regression not used (e.g. quadratic equation), must note list of affected compounds in laboratory report narrative. | Column 6: New language proposed by workgroup to maintain protective and conservative approach. |
| General Reporting | NA | (1) The laboratory must only report values ≥ the sample-specific RL/LLOQ<br>(2) Concentrations below the RL/LLOQ should be reported as "ND" with the same specific RL/LLOQ also reported.<br>(3) Dilutions- if diluted and undiluted analyses are performed, the lab should report ~~report results for both sets of data.~~ results for the lowest dilution within the valid calibration range for each analyte. The associated QC (e.g., method blanks, surrogates, etc.) for each analysis must be reported.<br>~~Compounds that exceed the~~ | NA | NA | (1) Complete analytical documentation for diluted and undiluted analyses must documented in laboratory report narrative and be maintained in laboratory records.<br>(2) The performance of dilutions must be documented in the laboratory report narrative or on the report form. Unless due to elevated concentrations of target compounds, reasons for dilutions must be explained in laboratory report narrative.<br>(3) If samples are not properly preserved or are | Group accepted CAM language.<br><br>Column 6: Audit requirement language was removed as CT DEEP does not conduct laboratory audits. |

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| Required ~~QA~~QC Parameter Column 1 | Data Quality Objective Column 2 | Required Performance Standard Column 3 | Required Deliverable Column 4 | Required~~Recommended~~ Corrective Action Column 5 | Required Analytical Response Action Column 6 | Rationale for Changes (removed from final version) |
|---|---|---|---|---|---|---|
| | | ~~linear range should be flagged ("E" flag). Do not report more than 2 sets of data/sample.~~<br>~~4) If a dilution is performed, the highest detected analyte must be in the upper 60% of the calibration curve, unless there are non-target analytes whose concentrations are so high as to cause damage to the instrumentation~~<br><br>NOTE: Labs shall not perform dilutions on samples due to sulfur interference. Labs must employ a cleanup technique to reduce the presence of sulfur interference.<br><br>(4) Results for soils/sediments must be reported on a dry-weight basis. | | | not received with an acceptable cooler temp, note the non-conformances in the laboratory report narrative.<br>(4) If samples are extracted and/or analyzed outside of the holding time, note the non-conformances in the laboratory report narrative. | |

If the RL/LLOQ is estimated due to unacceptable recovery of the lowest standard, the RL has not been achieved; Question 5b of the "Reasonable Confidence Protocol Laboratory Analysis QA/QC Certification Form" must be answered "NO" and this must be addressed in the laboratory report narrative.

~~Notes for Table 1A:~~

~~* Refers to latest promulgated version of SW-846 Method 8081.~~ ~~r = Correlation Coefficient~~
~~GC/MS = Gas Chromatography/Mass Spectrometry~~ ~~RPD = Relative Percent Difference~~
~~CCC = Calibration Check Compound~~
~~%RSD = Relative Percent Standard Deviation~~ ~~N/A = Not Applicable~~
~~EP = Environmental Professional~~

~~Note 1: Six standards are required for a quadratic equation calibration curve, and seven are required for a polynomial fit. In either case the correlation coefficient must be ≥ 0.990~~

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## ~~1.4.4~~1.5 Special Analytical Considerations for Multi-Response Pesticides

The identification of multi-component mixtures (i.e., chlordane or toxaphene) is not based on a single peak, but rather on the characteristic peaks that comprise the "fingerprint" of the mixture, using both the retention times and shapes of the indicator peaks. If, based on site history, multi-component chlorinated pesticides are contaminants of concern; it is the responsibility of the EP to request that these multi-component analyte spikes be included in the LCS and MS/MSD's. Multi-component mixtures are not routinely included in LCS or MS/MSD's.

DDT and endrin are easily degraded in the injection port. Breakdown occurs when the injection port liner is contaminated with high boiling residue from sample injection or when the injector contains metal fittings. The potential for DDT and endrin breakdown should be evaluated before samples are analyzed and at the beginning of each 12-hour shift as described in SW-846 Method 8081.

A linear or non-linear calibration model must not be used to compensate for detector saturation or to avoid proper instrument maintenance. As such, linear or non-linear regression must not be employed for initial calibration calculations that typically meet percent relative standard deviation ("%RSD") requirements specified in Table 1A.

Because of the variable solubility, extraction efficiency and analytical sensitivity of the different compounds that are potentially analyzable by SW-846 Method 8081, the recovery ranges presented in Table ~~II B-1~~1A of this RCP for laboratory control samples, matrix spikes, and surrogates should be considered general upper/lower acceptance limits when a single extraction procedure is utilized to prepare the extract for subsequent analysis. ~~It is essential that laboratory-specific performance criteria for LCS and surrogate recoveries also be calculated and documented as described in SW-846 Method 8000B, Section 8.7. When experience indicates that the criteria recommended in specific methods are frequently not met for some analytes and/or matrices, the in-house performance criteria will be a means of documenting these repeated exceedances. Laboratories are encouraged to actively monitor pertinent quality control performance standards described in Table II B-1 to assess analytical trends (i.e., systematic bias, etc) and improve overall method performance by preempting potential non-conformances.~~

## 1.~~5~~6 Analyte List for SW-846 Method 8081

The ~~Connecticut DEP (DEP)~~DEEP analyte list for SW-846 Method 8081 is presented in Table 1B. The compounds listed are readily determined by Method 8081. Most of the compounds listed have Connecticut ~~Remediation Standard~~RSR Criteria or are listed in the Approved Criteria for Additional Polluting Substances.

**Table 1B: Analyte List For SW-846 Method 8081**

| Analyte | CAS Number |
|---|---|
| Alachlor | 15972608 |
| Aldrin | 309002 |
| α-BHC | 319846 |
| β-BHC | 319857 |
| γ-BHC (Lindane) | 58899 |
| δ-BHC | 319868 |
| Chlordane (technical) | 57749 |
| 4,4'-DDD | 72548 |
| 4,4'-DDE | 72559 |

| Analyte | CAS Number |
|---|---|
| 4,4'-DDT | 50293 |
| Dieldrin[1] | 60571 |
| Endosulfan I | 959988 |
| Endosulfan II | 33213659 |
| Endosulfan Sulfate | 1031078 |
| Endrin | 72208 |
| Endrin Aldehyde | 7421934 |
| Endrin Ketone | 53494705 |
| Heptachlor | 76448 |
| Heptachlor Epoxide | 1024573 |
| Hexachlorobenzene | 118741 |
| Methoxychlor | 72435 |
| Toxaphene | 8001352 |
| [1]Aqueous RSR limit for Dieldrin (0.002 µg/L) may not be achievable | |

## 1.~~5~~6.1 Additional Reporting Requirements for SW-846 Method 8081

While it is not necessary to request and report all the analytes listed in Table 1B to obtain Reasonable Confidence status, it is necessary to document such a limitation, for site characterization and data representativeness considerations. ~~DEP~~DEEP strongly recommends that full list of analytes be reported during the initial stages of a site investigation and/or at sites with an unknown or complicated history of chemical usage or storage.

In cases where a shortened list of analytes is selected, the laboratory must still meet the method specific quality control requirements and performance standards associated with the requested analytes list to obtain Reasonable Confidence.

## *1.~~6~~7 Routine Reporting Deliverables for Method 8081*

The following table (Table ~~1.2~~5.0) lists the routine report deliverables. Note that while laboratories are not required to report certain items, they must keep the data on file and may be required to report all items in special circumstances.

**Table 5.0~~1.2~~: Report Deliverables**

| Parameter | Deliverable | Comments |
|---|---|---|
| Retention Time Windows | NO | |
| Endrin/DDT Breakdown | YES | Note non-conformances in laboratory report narrative |
| Initial Calibration | NO | Note non-conformances in laboratory report narrative |
| Continuing Calibration | NO | Note non-conformances in laboratory report narrative |
| Method Blanks | YES | Note non-conformances in laboratory report narrative. Flag all positive sample results above RL with "B" flag. |

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| Parameter | Deliverable | Comments |
|---|---|---|
| Lab Control Sample/Lab Control Sample Duplicate | YES | Note non-conformances in laboratory report narrative |
| Site Specific Matrix Spike/ Matrix Spike Duplicate | YES (If analyzed) | Note non-conformances in laboratory report narrative |
| Internal Standards (if used) | NO | Note non-conformances in laboratory report narrative |
| Identification and Quantitation | NO | Note non-conformances in laboratory report narrative |
| Surrogate Recoveries | YES | Note non-conformances in laboratory report narrative |
| General Reporting Issues | YES | Note non-conformances in laboratory report narrative |
| QA/QC Certification Form | YES | Signed by laboratory director or their designee. |
| Chain-of-Custody Form | YES | Signed by sample collector, courier, and laboratory. |

### 1.~~6~~7.1 Reporting and Flagging of Results

The following rules apply to reporting results:

- Non-Detects: Report all non-detects and results below the reporting limit as "ND" (Not Detected at the specified ~~reporting limit~~RL/LLOQ). The ~~reporting limit~~RL/LLOQ for each compound in each sample must be listed on the report ~~and take into account~~, based on the lowest calibration standard, the exact sample mass, any dilution factors, percent moisture, etc.

- Compounds detected above the ~~reporting limit~~RL/LLOQ in blanks and found in samples, also above the reporting limit, shall be flagged with a "B" suffix (e.g., 25B).

- When the results from dual columns have a RPD >40%, the results shall be qualified with a "P" flag as described in Table 1A, see identification and quantitation entry.

- All soil/sediment results shall be reported on a dry weight basis.

## *1.8 Sample Containers and Preservation*

Table 6.0 identifies the type of containers, preservation requirements, and holding times dependent upon analyte and matrix.

**Table 6.0~~2A~~: Sample Containers, Preservation, and Holding Times**

| Matrix | Container[1] | Preservative[2] | Holding Time |
|---|---|---|---|
| Aqueous with no chlorine present | 1-liter amber glass bottle with Teflon line cap | Store at 4 ± 2º C, but not frozen. | 7 days to extraction. 40 days from extraction to analysis. |
| Aqueous with chlorine present | 1-liter amber glass bottle with Teflon line cap | Neutralize chlorine with either 25 mg ascorbic acid or 3 mg sodium thiosulfate. | 7 days to extraction. 40 days from extraction to analysis. |

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| | | | |
|---|---|---|---|
| | | Store at 4 ± 2º C, but not frozen. | |
| Soil/Sediment samples. | 250 mL amber glass jar with Teflon lined cap. | Cool to 4 ± 2º C | 14 days to extraction. 40 days from extraction to analysis.<br><br>Up to one year for samples frozen within 48 hours of collection.[3] |
| High Concentration Waste Samples | Collect in amber glass jar with Teflon lined cap. | Cool 4 ± 2º C. | 14 days to extraction. 40 days from extraction to analysis. |

[1]The number of sampling containers specified is not a requirement. For specific analyses, the collection of multiple sample containers is encouraged to avoid resampling if sample is consumed or compromised during shipping and/or analysis.

[2]If samples were received by the laboratory on the same day of collection and were stored and transported to the laboratory on ice, cooler temperatures above 6ºC are acceptable.

[3]If the freezing option is selected the sample must be frozen within 48 hours of collection. The holding time recommences when thawing begins. The total holding time is calculated from the time of collection to freezing plus the time allowed for thawing. The total elapsed time must be less than 14 days.