ORGANOCHLORINE PESTICIDES AND POLYCHLORINATED BIPHENYLS (PCBs)

SW-846 Method 8081 or 8080

Table 1A. Summary of Holding Times and Preservation for Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs)

Analytical Parameter [a]	**Technical and Contract Holding Times**	**Preservation**
Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) in Water Samples	Technical for Extraction: 7 days from collection; Contract for Extraction: 5 days from receipt at laboratory Technical and Contract for Analysis: 40 days from extraction	Cool to 4EC ±2EC
Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) in Soil Samples	Technical for Extraction: 14 days from collection; Contract for Extraction: 10 days from receipt at laboratory Technical and Contract for Analysis: 40 days from extraction	Cool to 4EC ±2EC

[a] Individual target compounds are listed in Table 1B.

Data Calculations and Reporting Units:

Calculate the calibration factors (CF) of single component pesticides according to Section 7.4.2 of SW-846 Method 8000A. Calculate sample results using the analyte CFs from the midpoint standard of the associated initial calibration curve. Perform sample quantitation for multiple components pesticides according to Section 7.6 of SW-846 Method 8080A or 8081.

Report water sample results in concentration units of micrograms per liter (Fg/L). Report soil sample results on a dry-weight basis in micrograms per kilogram (Fg/kg).

For rounding results, adhere to the following rules:
a)If the number following those to be retained is less than 5, round down;
b)If the number following those to be retained is greater than 5, round up; or
c)If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculation in the data package.

TABLE 1B. Target Compound List, CAS Numbers, and Contract Required Quantitation Limits (CRQL) for SW-846 Method 8081 or Method 8080

COMPOUND	CAS No.	CRQL Water µg/L	CRQL Soil µg/kg
alpha-BHC	319-84-6	0.05	2
beta-BHC	319-85-7	0.05	2
delta-BHC	319-86-8	0.05	2
gamma-BHC (Lindane)	58-89-9	0.05	2
Heptachlor	76-44-8	0.05	2
Aldrin	309-00-2	0.05	2
Heptachlor epoxide	1024-57-3	0.05	2
Endosulfan I	959-98-8	0.05	2
Dieldrin	60-57-1	0.1	3
4,4'-DDE	72-55-9	0.1	3
Endrin	72-20-8	0.1	3
Endosulfan II	33213-65-9	0.1	3
4,4'-DDD	72-54-8	0.1	3
Endosulfan sulfate	1031-07-8	0.1	3
4,4'-DDT	50-29-3	0.1	3
Methoxychlor	72-43-5	0.5	17
Endrin ketone	53494-70-5	0.1	3
Endrin aldehyde	7421-93-4	0.1	3
alpha-Chlordane	5103-71-9	0.05	2
gamma-Chlordane	5103-74-2	0.05	2
Toxaphene	8001-35-2	5	170
Aroclor-1016	12674-11-2	1	33
Aroclor-1221	11104-28-2	2	67
Aroclor-1232	11141-16-5	1	33
Aroclor-1242	53469-21-9	1	33
Aroclor-1248	12672-29-6	1	33
Aroclor-1254	11097-69-1	1	33
Aroclor-1260	11096-82-5	1	3

Table 2. Summary of Calibration Procedures for Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) by SW-846 Method 8081 or 8080

Calibration Element	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (minimum blank + 3 points for each analyte) (ICAL) [a, b,c]	Initially; whenever required, due to failure of CCV	RSD for CFs #20% (#30% for Surrogate compounds)	1. Terminate analysis 2. Re-calibrate and verify before sample analysis
Continuing Calibration Verification (CCV) at midpoint of ICAL	Beginning of each day, after every 10 samples, and end of run	%D between CF of CCV and avg CFs from ICAL #25%	1. Re-calibrate and verify 2. Re-analyze samples back to last good CCV
Endrin and 4,4'-DDT Breakdown	Beginning and end of analytical sequence	#20% each or #30% combined	1. Investigate source of the problem and document 2. If either Endrin, 4,4'-DDT, or their breakdown products were detected, re-analyze the samples

[a] The ICAL low standard must be above but near the CRQL. The low ICAL standard must have a signal to noise ratio $5:1. If this requirement cannot be met, the laboratory must submit a MDL study as part of the data package.

[b] ICAL Prepare initial calibration individual standard mixtures A and B (IND A and IND B) containing the single component pesticides specified in Table 9 of SW-846 Method 8081 at three concentration levels. For multiple response pesticides, including toxaphene and Aroclors (except 1016 and 1260), prepare separate initial calibration standards at the following concentration levels: Aroclors (except 1221)at 100 ng/mL; Aroclor-1221 at 200 ng/mL; and toxaphene at 500 ng/mL. Aroclor-1016 and Aroclor-1260 may be combined into a single standard solution. Spike all calibration standards with the surrogate compounds discussed in Table 3 at a concentration of 20 ng/mL.

[c] Report the retention time window for each analyte. For multiple component pesticides, calculate the retention time window for 5 major peaks from the initial calibration standard analysis.

Determine retention time windows for both single and multiple component pesticides using the following guidelines:

Column Type	Retention Time Window in Minutes
Packed Column	#± 2%
Mega bore or wide bore capillary column	@ ±0.05 for tetrachloro-m-xylene through Aldrin @ ±0.07 for compounds which elute after Aldrin @ ±0.1 for decachlorobiphenyl

Table 3. Summary of Internal Quality Control Procedures for Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) by SW-846 Method 8081 or 8080

QC Element	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	One per Batch or SDG[a] (1 per 20 samples minimum)	< CRQL for each compound	1. Investigate source of contamination and document 2. Re-extract and re-analyze all samples processed with a non-compliant method blank
Surrogate [b]	Every standard, sample, method blank and QC sample at 10 times CRQL	60-150% of expected value	1. Re-analyze all samples with non-compliant surrogate recoveries
Matrix Spike and Matrix Spike Duplicate (MS/MSD)[c]	One MS/MSD set per batch or SDG (1 MS/MSD set per 20 samples minimum)	50-135% of expected value; #30 RPD between MS and MSD	1. Address in narrative

[a] SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within a case; or each 14 calendar day period during which field samples in a case are received.

[b] Spike each standard, sample, and blank with 1mL of a solution containing 0.2 Fg/mL each of tetrachloro-m-xylene and decachlorobiphenyl

[c] Spike MS/MSD samples with 1mL of a solution containing the following compounds and levels:

Target compound	Concentration (Fg/mL)	Target Compound	Concentration (Fg/mL)
?-BHC	0.5	Heptachlor	0.5
4,4'-DDT	1.0	Aldrin	0.5
Endrin	1.0	Dieldrin	1.0

Dilute and re-analyze samples with one or more analytes at concentrations exceeding the range of the calibration curve. Results for such re-analyses should fall within the mid-range of the calibration curve. Report results and submit documentation for both analyses.

Second column confirmation is required for all positive results. Perform confirmation analyses on a column of a phase different from that used for quantitation. Confirmation analyses must meet all instrument calibration criteria and blank acceptance criteria specified in Table 2, above.