STANDARD OPERATING PROCEDURE
TITLE: Extraction of Organochlorine, Organophosphorus, PCBs, and Pyrethroids Pesticides in Water Samples (Separatory Funnel)

REVISION HISTORY		
Revision #	**Summary of Changes**	**Date**
2	Eliminated solvent exchange into petroleum ether. Changed to concentration using nitrogen. Added additional techniques to improve quantitative transfer. Quality Control sections were updated.	07/16/14
1	Reformatted and separated extraction from instrument analysis. Added Safety sections. Combined pesticides groups into a single procedure. Added tables for working spike solutions. Required muffling of sodium sulfate prior to use. Added additional solvent rinses of sample containers and during extract transfers. Added specific responsibilities of the spike witness. Added information to be recorded for an extraction.	07/10/13
0	Initial release as part of SOP WPCL-GC-010.	06/10/2010

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1.0 Scope and Application

1.1 This method describes the sample extraction procedure for one or all of the following pesticide groups:

1.1.1 Selected organophosphorus pesticides(OPP).

1.1.2 Selected organochlorine pesticides (OCH).

1.1.3 Selected PCB congeners.

1.1.4 Pyrethroids.

1.1.5 See Table 1 through 4 for target analytes and routine spiking concentrations.

1.2 This method is applicable to groundwaters, surface waters, and waste waters. Marine waters have not been validated by WPCL using this method.

1.3 Each pesticide group may be extracted separately by this method. If more than one analysis is requested, a single extraction should be completed.

1.4 Only analysts who have been trained according to WPCL-QA-005 Training may be allowed to generate client data using this procedure.

2.0 Summary of Method.

2.1 A measured volume of sample (1000 mL) is extracted with methylene chloride (DCM) using a separatory funnel. The DCM extract is dried with sodium sulfate, concentrated using Kuderna-Danish (K-D) apparatus with additional evaporation using nitrogen gas. The concentrated extract is adjusted to 2.0 mL with iso-octane. The extracts are analyzed by gas chromatography or GC/MS/MS using conditions which permit the separation and measurement of the target analytes in the extracts.

2.2 Internal standards are added to the extracts to be analyzed by GC/MS/MS.

2.3 Lower reporting limits may be achieved through additional reduction of the final extract volume. Spiking levels may be adjusted to ensure that QC quantification is within the calibration curve range.

3.0 Interferences and Comments.

3.1 Organophosphorus pesticides+pyrethroids, organochlorine pesticides + polychlorinated biphenyls may be analyzed from the same final 2 mL extract; however, additional 1000 mL sample volumes must be provided for each QC sample type for each analysis; 4 @1000 mLs bottles must be provided for OPPs. Ex: From 2 @ 1 liter bottle, Sample A is analyzed for OPP, pyrethroids, OCH, PCBs. If an

MS/MSD is required to meet QC frequency criteria for each analysis on Sample A, then 4 @1 L for OPPs, 2 @ 1L for OCH, 2@ 1L for PCBs, and 2 @ 1L for pyrethroid (total: 12 @ 1 liter bottles) are needed.

3.2 Interferences in analyses may be encountered in very dirty samples. Extract cleanup may be needed to aid in the elimination or reduction of these interferences. Florisil column cleanup or Gel Permeation Chromatography (GPC) procedures will be used as needed.

3.3 Solvents, reagents, glassware, and other sample processing hardware may cause GC artifacts and/or elevated baselines, resulting in the misinterpretation of chromatograms. All materials should be demonstrated to be free from interferences under the conditions of the analysis by running method blanks initially and with each sample lot. High purity reagents and pesticide residue-grade solvents are required and are commercially available.

3.4 An effective way of cleaning laboratory glassware is by rinsing with polar and non-polar solvents before use. The cleaning procedure used must be tested by analyzing procedural blanks prior to analyzing samples.

3.4.1 Glassware washed with Alconox, rinsed with DI water, then air dried may also be baked at 400°C or higher for at least 4 hours.

3.5 Phthalates are common laboratory contaminants that are used widely as plasticizers. Sources of phthalate contamination include plastic lab-ware, plastic tubing, plastic gloves, plastic coated glassware clamps, and have been found as a contaminant in Na_2SO_4. Polytetrafluoroethylene (PTFE) can be used instead of polypropylene or polyethylene to minimize this potential source of contamination. However, use of PTFE lab-ware will not necessarily preclude all phthalate contamination. Na_2SO_4 will be baked in a muffle furnace at 450°C for at least 4.5 hours. Do not use plastic labware.

3.6 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source.

3.7 Care must be taken during the K-D step to ensure that compounds are not lost during evaporation. The compounds dimethoate, demeton-s, and disulfoton can be used as indicator compounds.

4.0 Sample Preservation and Holding Times

4.1 Store samples at <6°C protected from light until extraction.

4.2 Extract samples within 7 days of collection. Water samples may be discarded 30 days after the report is sent to the client. Dispose according to WPCL-EH-049.

4.3 If samples cannot be extracted within 3 days after lab receipt, add about 10 mLs of methylene chloride to all samples. Document the addition and solvent lot number on the chain-of-custody forms or sample log in checklist.

4.4 Extracts must be analyzed within 40 days of the extraction.

4.5 Store extracts at $\leq$ 6°C.

5.0 Safety

5.1 The toxicity or carcinogenicity of each compound or reagent used in this method has not been precisely determined. However, each chemical compound and sample should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level. The laboratory is responsible for maintaining a current file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets should be made available to all personnel involved in this procedure. It is the responsibility of the analyst to read the MSDS as part of the training process.

5.2 Wear gloves, lab coats, safety glasses or face shields while processing samples. All processes must be performed in an operating hood.

5.3 Wear a face shield while performing any operations involving vacuum.

5.4 Wash your hands at the completion of sample processing.

5.5 Dispose of waste solvents and spiking solutions according to WPCL-EH-049 "Disposal of Hazardous Wastes."

5.6 The following chemicals have the potential to be highly toxic or hazardous. For details, read the MSDS associated with each chemical.

5.6.1 Methylene chloride AKA dichloromethane (DCM).

5.6.2 Iso-octane (ISO).

5.6.3 Acetone.

6.0 Equipment and Supplies

6.1 Separatory funnel. 2000-ml, with TFE-fluorocarbon stopcock, ground glass or TEF stopper.

6.2 Automatic shaker designed to fit 2 liter separatory funnels with rpm and timer controls.

6.3 Beakers. Borosilicate glass, 400 mL or 250 mL Amber glass bottles with caps.

6.4 Watch glasses.

6.5 Glass wool. Pyrex - solvent washed prior to use.

6.6 Forceps rinsed with methylene chloride prior to use.

6.7 Kuderna-Danish (K-D) Apparatus.

6.7.1 Concentrator tube. 15 mL, graduate (Kontes K0570012-0500, or equivalent). A ground stopper, 19/22 joint, is used to prevent evaporation of extracts.

6.7.2 Evaporation flask. 500 mL (Kontes K-570050-0500, or equivalent), attached to concentrator tube with blue clamp (Kontes K-662750-0012).

6.7.3 Snyder column. Three ball (Kontes K-503000-0121, or equivalent).

6.7.4 Boiling chips. Chemware PTFE Ultra Pure (PN 26397-103)-before use, rinse 3 times with methylene chloride, then let the solvent evaporate. Note that boiling chips can be a significant source of contamination if not properly cleaned.

6.8 Water bath. Organomation S-EVAP-KD, thermostatically controlled with stainless steel cover to fit K-D apparatus, installed in a fume hood.

6.9 GC vials. GC autosampler vials, borosilicate glass, 2 mL with PTFE-lined screw cap.

6.10 Analytical balance. Capable of weighing 0.1 mg.

6.11 Drying oven.

6.12 Disposable Pasteur Pipettes. 2 mL, rinsed with solvents before use.

6.13 Glass filter funnel. Fluted, 75 mm or larger.

6.14 Graduated cylinder. 1000 ml, 250 mL and 100 mL.

6.15 Sodium Sulfate (Na_2SO_4)-Anhydrous granular grade. Bake in a muffle furnace at 450°C for about 4 hours. Cool, and store in cleaned, glass jars. Rinse with DCM prior to use.

6.16 Nitrogen, pre-purified (99.9999% grade) or better`.

6.17 Nitrogen blow down apparatus

7.0 Reagents and Standards

7.1 Reagent water (“DI water”) is defined as water in which an interferent is not observed at method detection limit of each parameter of interest. ASTM Type I water (distilled, carbon filtered, and polished) will meet the definition of reagent water.

7.2 Acetone, methylene chloride (DCM), isooctane. Pesticide residue quality or equivalent or better.

7.3 Stock standards. Individual stock standards are purchased as certified solutions as well as premixed solutions. See Tables 1 through Tables 4

8.0 Calibration and Standardization.

8.1 Verify and record refrigerator and freezer temperatures using calibrated thermometers.

8.2 Verify and record water bath temperatures on extraction benchsheets or in logbooks.

8.3 Compare final extract volumes to aliquots delivered by volumetric glassware.

9.0 Procedure

9.1 Turn the water bath on and set at 74°C. The range should be within 70°C to 78°C.

9.2 Remove water samples from refrigerator to allow samples to equilibrate to room temperature.

9.2.1 Compare the sample ID, collection date, collection time and requested analyses listed on the bottle label to the chain-of-custody (COC) copy.

9.2.2 If there are any discrepancies, notify the Section Lead or the Sample Receiving Lead for clarification. If any corrections are made on the COC, correct all COC copies.

9.2.3 Remove spikes and surrogate solutions from the refrigerator and allow to equilibrate to room temperature.

9.2.4 Solvent rinse separatory funnels, beakers, and all needed glassware with DCM (three times).

9.2.1 Make tape labels and log an entry for the extraction in the logbook. Prepare an extraction benchsheet or extraction logbook page to record sample and extraction information. The extraction benchsheet must include:

9.2.1.1 Customer/client name.

9.2.1.2 Lab number (L-number).

9.2.1.3 Sample number, sample ID.

9.2.1.4 Test name.

9.2.1.5 Extraction date.

9.2.1.6 Analyst name.

9.2.1.7 Spike witness.

9.2.1.8 Initial sample volume.

9.2.1.9 Final extract volume.

9.2.1.10 Lot number and brand of any filters used.

9.2.1.11 Solvent lot numbers used.

9.2.1.12 Final solvent.

9.2.1.13 Quality control lot/tracking number.

9.2.1.14 Spike solution ID number and concentration.

9.2.1.15 Spike solution volume added.

9.2.1.16 Surrogate solution ID number and concentration.

9.2.1.17 Surrogate solution volume added.

9.2.1.18 If added. Internal standard volume, ID, initials, date added

9.2.2 Pour 1 liter of sample into a pre-cleaned 2-liter separatory funnel. Replace the empty bottle in front of the associated separatory funnel.

9.2.3 If a sample was preserved with DCM, allow the sample to settle for a couple of minutes. Collect the DCM in a cleaned, labeled beaker. This aliquot will be added to the solvent extracts collected in step 9.5 or 9.6.

9.2.4 For Method Blank(MBLK), Lab Control Sample(LCS), Lab Control Sample Duplicate(LCSD), use 1 liter DI water each.

9.2.5 For matrix spikes (MS/MSD each) measure 1 liter of sample. Add pesticide spiking solutions according to the analysis of interest as described in 9.2.6.

9.2.6 Add surrogate solution to every sample and quality control.

9.2.6.1 Universal Surrogate: add 1 mL of (20 ng/mL in acetone DBOB/ DBCE/1000 ng/mL TPP). This surrogate is added to every sample requesting pyrethroids or OCH or PCBs or OPP.

9.2.6.2 OCH: In addition to the Universal Surrogate, add 1 mL of (20 ng/mL in acetone PCB 207).

9.2.6.3 PCBs: In addition to the Universal Surrogate, add 1 mL of (20 ng/mL in acetone PCB 207). Note that this compound is also in the spike solution.

9.2.7 Immediately add spiking solution to the LCS, LCSD, MS, MSD.

9.2.7.1 OPP: Individual 1 liter samples are needed for each spike mixture:

9.2.7.1.1 Add 1.0 mL of OP Mix A (Table 1)

9.2.7.1.2 Add 1.0 mL of OP Mix B. (Table 1)

9.2.7.2 Pyrethroids: add 1 mL Pyrethroid Spike (Table 2)

9.2.7.3 OCH: add 1 mL OCH Spike (Table 3)

9.2.7.4 PCBs: add 1 mL PCB Spike. (Table 4)

9.2.8 A witness is required to confirm that samples have been spiked properly.

9.2.8.1 The spike witness must verify that the spike solutions are not expired, are the proper pesticide group, are the proper concentration and are documented in the standards log.

9.2.8.2 The witness must verify that the correct volume was added to all samples.

9.2.8.3 The witness must sign and date the extraction records.

9.3 Add 40 ml of methylene chloride (DCM) to the empty bottle, replace the cap and rinse the bottle. Pour the DCM into the separatory funnel and repeat 2 more times using 40 mL of DCM each time for a total of 120 mL. Pour 120 mLs of DCM directly into the separatory funnels for the MBLK, LCS, LCSD.

9.4 Cap and strap each separatory funnel. Rotate/spin the samples several spins, then stop and gently vent to release excess pressure without loss of sample. Spin samples for 10 minutes using a timer. After 10 minutes, remove the cap and let stand to allow layers to separate. Allow organic layer to separate from the water phase for no more than 5 minutes..

9.5 Collect the methylene chloride extract in a 400 ml beaker or back into the corresponding sample bottle. Alternatively, collect the extract in 250 mL amber glass bottles if the KD step will not be performed on the same day.

9.6 Add a second 120 ml volume of methylene chloride to the separatory funnel and repeat the extraction procedure a second time, combining the extracts in the beaker or corresponding sample bottle.

9.7 Prepare a solvent-rinsed filter funnel with a plug of pre-cleaned glass wool in the bottom of the funnel. Cover with about two inches of baked and solvent-rinsed sodium sulfate.

9.8 Set up and label pre-cleaned K-D flasks with concentrator tubes and attach with a blue clamp on test tube racks in the fume hood. Place the funnel assembly in the top of the K-D flask.

9.9 Pour the combined extracts from the beaker through sodium sulfate into the K-D flask. Rinse the beaker with about 10 mL of DCM and add this rinse to the sodium sulfate. Allow to drain. Repeat with another 2x10 mL DCM rinse. Rinse the sodium sulfate with an additional portion of DCM (3x ~10-20 mL) for a total of 6 rinses.

9.10 Add 0.5 ml iso-octane as “keeper” and a solvent rinsed micro-boiling chip to each K-D flask. Place a Snyder column on the K-D flask and place the assembly on the hot water bath set at 70-78 ^{o}C.

9.11 Evaporate solvent on the hot water bath. When the apparent volume of solvent in the concentrator tube is 10-15 mLmL, add about 5 mL of iso-octane through the top of the Snyder column slowly.

9.12 Check vials for the presence of water. If present consult with the Section Leader.

9.13 When the reflux line falls below the top of the Snyder column, the K-D apparatus should be removed from the hot water bath. Quickly pour about 5 mLs of iso-octane through the top of the Snyder column.

9.13.1 Spin the K_D apparatus for about 10 seconds.

9.13.2 Rinse the outside of the K_D apparatus with acetone to dry the outside of the KD apparatus. Take care that acetone does not seep into the concentrator tube.Let sit for 5 minutes. Remove the Snyder column.

9.14 Remove the concentrator tube from the K-D apparatus

9.15 Adjust the nitrogen stream to a gentle flow rate. Look for a slight dimple on the solvent tube surface. Too rigorous a flow rate will result in loss of target analytes.Submerge the concentrator tube in a 50°C water bath. Concentrate to about 0.5-1.0 mLs.

9.16 Quantitatively transfer the concentrated extract to a clean, labeled test tube.

9.17 Rinse the concentrator tube with about 0.5 mL of iso-octane and transfer rinsate to the labeled test tube. Repeat.

9.18 Adjust to a final volume of 2 mL with iso-octane.

9.18.1 One mL is used for pyrethroids.

9.18.2 The remaining milliliter is reserved for OCH, OPP, PCBs.

9.19 Mix the tube contents by tapping the bottom of the tube causing a vortex which will rinse the sides of the tube. A Vortex Genie mixer may be used for this step.

9.20 Transfer extract to labeled clear amber glass GC vials then cap.

9.21 Mark the top of the meniscus in the vial with an arrow pointing down to the extract surface. Place extracts in a refrigerator for storage until analysis or cleanup, if necessary.

9.22 If extract clean-up is required, follow WPCL-PR-054 Florisil Cleanup of Water Samples and/or WPCL-PR-055 GPC Cleanup of Water Samples.

10.0 Quality Control

10.1 A method blank, laboratory blank, two reference standards, laboratory control sample, matrix spike, matrix spike duplicate and sample duplicate should be

analyzed for every 20 samples, sample batch or different sample matrix. Refer to the attachments for acceptance criteria and corrective actions.

10.1.1 **Batch.** A batch is 20 or fewer samples of similar matrix processed together at the same time. A batch will share the same reagent lots, procedure, time period, analysts, and quality controls. A batch is defined before the start of processing.

10.1.2 **Method Blank**: Include one blank with every batch of samples. Include all reagents and perform all procedures on the blank as performed for all samples included in the batch. The blank must be treated just like a regular sample. If processing aqueous samples, use the volume of lab-quality water specified in the SOP.

10.1.3 **Laboratory Control Sample (LCS)-** Analytes of known identity and concentration are added to a lab-clean matrix then processed as a sample. Provides evidence that the procedure will perform as validated in the absence of matrix effects. Use a specified volume of lab-quality water as the background matrix. At least one LCS is included with every batch.

10.1.4 **Laboratory Control Sample Duplicate (LCSD)-** A second LCS. Processed the same as an LCS but included when there is insufficient field sample to provide a matrix spike/matrix spike duplicate pair. The spike pair provides a measure of batch processing consistency

10.1.5 **Matrix Spike(MS) –** Matrix spike is a subsample of a field sample where the analyst adds known analytes in known concentrations prior to lab processing with the batch.

10.1.6 **Matrix Spike Duplicate (MSD) –** Matrix spike duplicate, processed the same as a MS.

10.2 Method Detection Limits (MDL)

10.2.1 Method Detection Limit (MDL) refers to the lowest concentration of the analyte that a method can detect reliably. To determine the MDL, 7 lab Milli-Q water samples are spiked and processed through the entire method along with a blank. The standard deviation derived from the spiked sample recoveries were used to calculate the MDL for each analyte using the following equation:

$$MDL = tS$$

Where t is the Student t test value for the 99% confidence level with n-1 degrees of freedom and S denotes the standard deviation obtained from n replicate analyses. For the n=7 replicates used to determine the MDL, t=3.143.

10.3 Reporting Limit (RL).

10.3.1 Reporting limit (RL) refers to a level at which reliable quantitative results may be obtained. The MDL is used as a guide to determine the RL. The RL is typically chosen in a range 1-10 times the MDL.

10.4 Method Validation

10.4.1 Refer to WPCL-QA-006 Validation and Method Detection Limit Studies.

10.5 Control Charts and Limits

10.5.1 Control charts are generated initially using the data from the method validation. The upper and lower warning and control limits are set at ± 2 and 3 standard deviations of the average percent recovery.

10.6 If quality control samples indicate that contamination or poor recoveries are due to errors during the extraction, all affected samples associated with failed controls will be re-extracted in a new batch if there is sufficient sample volume available. The new batch must include a new set of quality control samples.

11.0 References

11.1 Method 3510, Separatory Funnel Liquid-Liquid Extraction, USEPA-OSW, SW-846, Revision 3, 1996.

11.2 WPCL-EH-049, Handling of Wastes.

11.3 WPCL-QA-003 Training

12.0 Attachments

12.1 Table 1: Organophosphorus pesticides.

12.2 Table 2: 25 ppb Pyrethroids Spike.

12.3 Table 3: 20 ppb Organochlorine pesticides spike.

12.4 Table 4: 20 ppb PCB Congeners spike.

12.5 Table 5: Corrective Actions and Acceptance Criteria.

Table 1: Organophosphorus Pesticides

CAS Number	Analyte	Working Spike Concentration ng/mL in Acetone
7786-34-7	Mevinphos	1000 A
13194-48-4	Ethoprop	1000 A
3689-24-5	Sulfotepp	400 A
298-02-2	Phorate	1000 A
333-41-5	Diazinon	400 A
298-04-4	Disulfoton	1000 A
60-51-5	Dimethoate	2000 A
299-84-3	Ronnel (Fenchlorphos)	1000 A
298-00-0	Parathion, methyl	1000 A
2921-88-2	Chlorpyrifos	400 A
121-75-5	Malathion	1000 A
34643-46-4	Tokuthion(Prothiofos)	1000 A
22248-79-9	Tetrachlorvinphos (Stirophos,Gardona)	1000 A
950-37-8	Methidathion	1000 A
35400-43-2	Bolstar(Sulprofos)	2000 A
732-11-6	Phosmet	2000 A
86-50-0	Azinphos-methyl	2000 A
56-72-4	Coumaphos	4000 A
786-19-6	Carbophenothion	1000 A
13071-79-9	Terbufos	400 A
944-22-9	Fonophos (Dyfonate)	400 A
8065-48-3	Demeton-S	1000 B
297-97-2	Thionazin (Zinophos)	400 B
97-17-6	Dichlorofenthion	400 B
122-14-5	Fenitrothion	400 B
56-38-2	Parathion, ethyl	400 B
470-90-6	Chlorfenvinphos	400 B
563-12-2	Ethion	400 B
52-85-7	Famphur	400 B
21609-90-5	Leptophos	400 B
2642-71-9	Azinphos-ethyl	800 B
5598-13-0	Chlorpyrifos methyl	400 B
327-98-0	Trichloronate	400 B
55-38-9	Fenthion	400 B
115-90-2	Fensulfothion	2000 B

A = OP Mix A
B = OP Mix B

Table 2:"25 ppb Pyrethroids Spike"

CAS Number	ANALYTE NAME	Working Spike Concentration ng/mL in Acetone
82657-04-3	Bifenthrin	5
68359-37-5	Cyfluthrin, total	25
52315-07-8	Cypermethrin, total	25
52918-63-5/66841-25-6	Deltamethrin/Tralomethrin	25
66230-04-4/51630-58-1	Esfenvalerate/Fenvalerate, total	10
64257-84-7	Fenpropathrin	5
54774-45-7	Permethrin, cis-	25
51877-74-8	Permethrin, trans-	50
91465-08-6	Cyhalothrin, lambda, total	10

Table 3: "20 ppb Organochlorine Pesticides Spike"

CAS Number	ANALYTE NAME	Working Spike Concentration ng/mL in Acetone
309-00-2	Aldrin	20
5103-71-9	Chlordane, cis	20
5103-74-2	Chlordane, trans	20
1861-32-1	Dacthal	20
53-19-0	DDD(o,p')	20
72-54-8	DDD(p,p')	20
3424-82-6	DDE(o,p')	20
72-55-9	DDE(p,p')	20
1022-22-6	DDMU(p,p')	20
789-02-6	DDT(o,p')	20
50-29-3	DDT(p,p')	40
60-57-1	Dieldrin	20
959-98-8	Endosulfan I	20
33213-65-9	Endosulfan II	20
1031-07-8	Endosulfan sulfate	20
72-20-8	Endrin	20
7421-93-4	Endrin Aldehyde	40
53494-70-5	Endrin Ketone	40
319-84-6	HCH, alpha	20
319-85-7	HCH, beta	20
58-89-9	HCH, gamma	20
319-86-8	HCH, delta	20
76-44-8	Heptachlor	20
1024-57-3	Heptachlor epoxide	20
118-74-1	Hexachlorobenzene	10
72-43-5	Methoxychlor	20
2385-85-5	Mirex	20
5103-73-1	Nonachlor, cis	20
39765-80-5	Nonachlor, trans	20
19666-30-9	Oxadiazon	20
27304-13-8	Oxychlordane	20

116-29-0	Tedion	20

Table 4: "20 ppb PCBs Spike"

CAS Number	CONGENER NUMBER	Working Spike Concentration ng/mL in Acetone
16605-91-7	PCB 005	100
34883-43-7	PCB 008	20
2050-68-2	PCB 015	100
37680-65-2	PCB 018	20
38444-76-7	PCB 027	20
7012-37-5	PCB 028	20
15862-07-4	PCB 029	20
16606-02-3	PCB 031	20
38444-86-9	PCB 033	20
41464-39-5	PCB 044	20
41464-40-8	PCB 049	20
35693-99-3	PCB 052	20
41464-43-1	PCB 056	20
33025-41-1	PCB 060	20
32598-10-0	PCB 066	20
32598-11-1	PCB 070	20
32690-93-0	PCB 074	20
32598-13-3	PCB 077	20
38380-02-8	PCB 087	20
38379-99-6	PCB 095	20
41464-51-1	PCB 097	20
38380-01-7	PCB 099	20
37680-73-2	PCB 101	20
32598-14-4	PCB 105	20
38380-03-9	PCB 110	20
74472-37-0	PCB 114	20

CAS Number	CONGENER NUMBER	Working Spike Concentration ng/mL in Acetone
31508-00-6	PCB 118	20
57465-28-8	PCB 126	20
38380-07-3	PCB 128	20
35694-06-5	PCB 137	20
35065-28-2	PCB 138	20
52712-04-6	PCB 141	20
38380-04-0	PCB 149	20
52663-63-5	PCB 151	20
35065-27-1	PCB 153	20
38380-08-4	PCB 156	20
69782-90-7	PCB 157	20
74472-42-7	PCB 158	20
35065-30-6	PCB 170	20
38411-25-5	PCB 174	20
52663-70-4	PCB 177	20
35065-29-3	PCB 180	20
52663-69-1	PCB 183	20
52663-68-0	PCB 187	20
39635-31-9	PCB 189	20
35694-08-7	PCB 194	20
52663-78-2	PCB 195	20
52663-75-9	PCB 199	20
40186-71-8	PCB 201	20
52663-76-0	PCB 203	20
40186-72-9	PCB 206	20
2051-24-3	PCB 209	20
52663-79-3	PCB 207 (Surrogate)	20

Table 5: Corrective Actions and Acceptance Criteria

QC TYPE	CONTROL	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Batch	Unit of sample processing.	Up to 20 samples of similar matrix, same reagents, equipment, techniques.	Batch is comprised of 20 or fewer field samples.	Include additional controls during processing or reextract.
Method blank.	Indicator of contamination that may be introduced by reagents, equipment during processing.	Every batch.	Must be less than reporting limit or project requirements, whichever is more stringent.	Reanalyze blank to confirm result. Evaluate impact on sample results. Re-extract affected samples as needed.
LCS	Accuracy and recovery of target analytes from a clean, lab matrix.	Every batch.	Must be within control limits.	Reanalyze LCS to confirm result. Evaluate impact on sample results. Low recoveries require re-extraction of the batch.
LCS Duplicate	Accuracy and reproducibility of target analyte recovery in a clean lab matrix	Every batch where a MS/MSD is not processed.	Recoveries must be within control limits. RPD must be within control limits.	Reanalyze LCSD to confirm result. Evaluate impact on sample results. Low recoveries require re-extraction of the batch.
MS	Accuracy and recovery of target analytes in a field sample.	Every batch (assumes sufficient sample).	Recoveries should be within control limits.	Reanalyze MS to confirm result. Review against LCS.
MSD	Accuracy and reproducibility of target analytes in a field sample.	Every batch (assumes sufficient sample).	Recoveries should be within control limits. RPD should be within control limits.	Reanalyze MSD to confirm result. Review against LCS/LCSD.
CRM	Accuracy and recovery of target analytes from a well-characterized matrix.	Every batch as directed by project if commercially available.	Recoveries should be within limits as defined by the project.	Reanalyze CRM to confirm result. Compare against LCS recoveries. Consistent failure requires reextraction of the batch.
Surrogates	Accuracy and recovery of chemically similar compounds in field samples.	Every sample.	Should be within limits.	Reanalyze sample to confirm result. Review against LCS.
ICV/CCV	Instrument drift.	After multipoint calibration, prior to sample analysis after every 10 injections, and end of run.	$\pm$ 20% from expected concentration.	If exceeds acceptance criteria, verify that the standard was not mis-injected, then review bracketed sample results. If CCV response is higher than expected, reanalyze samples with positive detections and surrogate failures. Analyze samples back to the last acceptable CCV. Document decisions with reported results. Recalibrate if ICV/CCV fails.