Standard Operating Procedure for:

EXTRACTION OF PESTICIDES AND POLYCHLORINATED BIPHENYLS (PCBs) FROM WATER, WASTES, SOIL/SEDIMENT, AND FILTERS FOR SUBSEQUENT ANALYSIS BY GC OR GC/MS TECHNIQUES



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CHEMISTRY SECTION
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1. SCOPE AND APPLICATION

1.1 This method describes the extraction from waters, sediment, PUFs, filters and wastes of select organophosphorus, organonitrogen, organohalide pesticides, and PCB's. It is based on extraction procedures described in EPA Methods 608, 608.2, 617, 1656, 614, 614.1, 619, 622, 622.1, 633, 645, 8081B, 8082A, 8141B, 3510C, 3540C, 3550C, 3580A, 3620C, 3640A, 3660B, 3665A.

1.2 The sample extraction and concentration steps in this method are essentially the same for both halogenated and nitrogen phosphorus containing pesticides. Thus in many instances, a single sample extraction may recover nitrogen, phosphorus, and halogen-containing pesticides.

1.3 This procedure can be used for all samples with one of the following Laboratory Information Management System (LIMS) test IDs: S-PEST-NP, WAS-PST-NP, W-NP-AA-SF, W-PC-AA-SF, W-PESTNP-R, W-PCB-R, W-PST-CL-R, W-PSNP-TQ, W-PSCL-TQ, W-TR-TQ, S-PSCL-TQ, S-PCB-TQ, S-CT-CI, WS-CT-CI., WS-PSCL-TQ, WS-PCB-TQ.

2. SUMMARY OF THE METHOD

2.1 For water samples (based on EPA 3510C, 608, 608.2, 617, 1656, 614, 614.1, 619, 622, 622.1, 633, 645,), a whole one-liter bottle of sample is extracted with methylene chloride. The resulting extract is concentrated and solvent exchanged into iso-octane. The sample extract is then ready for analyses by capillary gas chromatography with electron capture or nitrogen-phosphorus or mass spectrometry based detection.

2.2 For sediment samples (based on EPA 3550C), thirty grams of sample are extracted with a 1:1 solution of methylene chloride: acetone. The resulting extract is exchanged to iso-octane and concentrated. The sample extract is then ready for analyses by capillary gas chromatography with electron capture, nitrogen-phosphorus or mass spectrometry detection. Part of the sample extract is further cleaned up by the appropriate clean up procedures described in this SOP for Chlorinated Pesticides analysis.

2.3 For waste samples (based on EPA 3580C), approximately one gram of sample is weighed into a calibrated tube and diluted to 10 mL with iso-octane. The resulting extract, if necessary, is then subjected to the appropriate clean up procedures described in this SOP. The sample extract is then ready for analyses by capillary gas chromatography with electron capture, nitrogen-phosphorus detection or mass spectrometry detection.

2.4 Filter samples are extracted by Soxhlet extraction (based on EPA 3540C) using methylene chloride and made up to 1mL in iso-octane and analyzed by capillary gas chromatography with electron capture or nitrogen-phosphorus detection.

2.5 Any sample extract may undergo GPC (EPA 3640A), Florisil (EPA 3620C), or Sulfuric Acid clean up (EPA 3665A) if necessary.

3. APPARATUS AND EQUIPMENT

3.1 Separatory funnels - 2 L and 1 L with Teflon stopcocks and caps

3.2 125mL & 250mL Erlenmeyer flasks with B24/40 Std. joint with stopper

3.3 Graduated cylinders 100mL and 2000 mL

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3.4 Concentrator tube, Kuderna-Danish 15mL with ground glass stopper

3.5 Evaporative flask, K&D-500mL, attached to concentrator tube with clips

3.6 Snyder column, K&D - three-ball macro

3.7 Centrifuge tubes - 10 to 15mL with standard joint B, or screw cap

3.8 Boiling chips - approximately 10/40 mesh

3.9 S-Evap® -KD Solvent evaporator system (Organomation Associates, Inc)

3.10 Separatory funnel tumblers with speed control

3.11 Balances

3.11.1 Analytical, accurate to 0.0001g

3.11.2 Top-loading, accurate to 0.1g

3.12 Glass stirring rods (long enough to fit down into a 2-liter separatory funnel)

3.13 Ultrasonic water bath Fisher Scientific FS 220 H, Output 250 W

3.14 Centrifuge (for centrifuging 500mL bottles and 15mL tubes)

3.15 Metal spatulas

3.16 Beakers, 400mL

3.17 Volumetric pipets, 0.5, 1, 2, 5, 10mL

3.18 Volumetric flasks, 5, 10, 25, 50, 100, 200, 250, 500, 1000mL

3.19 Disposable Pasteur pipets

3.20 N-Evap® -116, nitrogen-evaporator (Organomation Associates, Inc)

3.21 GPC:

3.21.1 Autosampler: Shimadzu SIL-10A with SCL-10A System controller

3.21.2 Fraction Collector: ISCO Foxy 200XT

3.21.3 GPC column: Phenomenex Envirosep ABC (350 x 21.2 mm) with (60 x 21.2 mm) pre column

3.22 Glass wool

3.23 Buchner funnels, 9 cm

3.24 Filtration flasks, 500mL with rubber adapter

3.25 Whatman 42 filtration circles 9 cm

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3.26 Aluminum weighing pans, for dry weight determination

3.27 Brinkman Dispensette 20-100mL or a Tippet dispenser with a 60mL bulb

3.28 Long stem powder funnel

3.29 Glass extraction columns

3.30 Aluminum foil

3.31 Culture tubes 16 x 150 mm and 25 x 150 mm (50mL)

3.32 254-nm fixed wavelength ultraviolet detector

3.33 Round bottom flask with B24/40 Std. joint – 250, 500mL

3.34 Soxhlet extractor- ROT-X-TRACT, (Organomation Associates, Inc)

3.35 Soxhlet extractor body with 55/50 top joint and 24/40 bottom joint. For PUF samples 45/50 and 24/40.

3.36 Drying column - Chromatographic column, approximately 400 mm long x 19 mm ID with coarse frit filter disc

3.38 Cleaned PUF Plugs 6 cm OD x 7.6 cm from Supelco.

3.39 Vacuum Desiccator for storing clean PUFs

4. REAGENTS AND CHEMICALS

4.1 Reagent water – Distilled or Deionized water.

4.2 Well water - Free of interferences, used for laboratory blanks and laboratory fortified blanks

4.3 Solvents:

4.3.1 Methylene chloride - Fisher Scientific Pesticide grade or HPLC Grade

4.3.2 Hexane - Fisher HPLC Grade or Baxter UV or GC/MS Grade and distilled for PUF sample extraction

4.3.3 Toluene, Fisher HPLC Grade or Baxter UV or GC/MS Grade

4.3.4 Acetone - Fisher Pesticide Grade or HPLC Grade and distilled for PUF sample extraction

4.3.5 Benzene, Fisher Optima or Pesticide/GC-MS grade

4.3.6 Iso-Octane Fisher Scientific, Pesticide Grade

4.4 Sodium Sulfate - (ALS) granular and powder, anhydrous. Purify by heating at 460 °C for 8 hours or overnight, and store in a largemouth bottle with cap.

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4.5 Florisil 1g (6mL) - Sep-Pak Vac Florisil Cartridge, J&W brand or equivalent

4.6 Tetrabutylammonium hydrogen sulfate, AR

4.7 Sodium sulfite, AR

4.8 GPC calibration solution: Prepare a solution in methylene chloride that contains the following analytes in the concentration listed below (for standard preparation procedure see DEP SOP GC-029-1, Ref. 15.16):

Analyte	mg/mL
Corn oil	12.5
Bis-2-ethylhexyl phthalate	0.5
Perylene	0.01
Sulfur	0.04

4.9 Sodium chloride - AR, granular and powder, anhydrous. Purify by heating at 460 °C for 8 hours or overnight, and store in a largemouth bottle with cap.

4.10 Sulfuric acid, concentrated

4.11 Sulfuric acid 1:1 (v/v). Dilute equal volumes of sulfuric acid and DI water, adding small aliquots of concentrated sulfuric acid in water and cooling the mixture. The temperature must stay close to room temperature all the time.

4.12 10N Sodium hydroxide (NaOH) solution - Dissolve 40g of NaOH (ACS) in DI water and dilute to 100mL

4.13 **Spiking solutions** (see standards preparation procedure for more details in DEP SOPs GC-011-5 (ECD), Ref. 15.13, GC-012-3 (NPD, Ref. 15.14) , GC-033-1 (Ref. 15.20, Gc-034-1 (Ref 15.21) and SOP GC-035-1 (Ref 15.22).

4.13.1 NPD surrogate spiking solution: 5.0 mg/L NMX use 200 µL for 2 mL final voluand 1 mL for 10 mL final volume. .

4.13.2 PSCL surrogate spiking solution 200 ug/L TMX/DCB each; - use 250 µL for water samples for GC ECD if the final volume of the sample extract is to be 2mL. The same surrogate spike is used for GC/MS analyses.

4.13.3 NPD Spike Solutions; use 1mL for 10 ml final volume or 200 uL for 2 mL final volume

4.13.3.1 NPD-A: 500-3000 ug/L
4.13.3.2 NPD-B: 1000-6000 ug/L

4.13.4 ECD SpikeSolutions; use 200 uL for 2 mL final volume

4.13.4.1 ECD-A: 100-500 ug/L;
4.13.4.2 ECD-B: 250-2500 ug/L;
4.13.4.3 PCB1016 & 1260: 2.5 mg/L; Chlordane Tech.: 2.5 mg/L; Toxaphene: 10.0 mg/L;

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4.13.5 Spiking solutions for the GC/MS/MS tests

4.13.5.1 NP-TQ surrogate spike solution 100 ug/L of Simazine-d10 and Malathion-d7 – use 50 uL for 0.25 mL final volume.
4.13.5.2 NP-TQ spike solution 50-400 ug/L – use 50 uL for 0.25 mL final volume.
4.13.5.3 PSCL surrogate spike solution 200 ug/L of TMX and DCB – use 200 uL for 2.0 mL final volume. For W-PSCL-TQ use 50 ul for 0.5 mL final volume. The same surrogate spike is used for ECD analysis….
4.13.5.4 CL-TQ spike solution 200-1000 ug/L use 200 uL/ for 2 mL final volume. For W-PSCL-TQ use 100 uL for 0.5 mL final volume. 200 ppb Dieldrin spike may also be used for this test.
4.13.5.5 PCB 1060 spike 2.5 mg/L – use 160 uL for 2 mL final volume.
4.13.5.6 T-CL-TQ spike solution use 200 ug/L for 2 mL final volume.
4.13.5.7 TR-TQ tracers surrogate spike solution 250 ug/L- use 50 uL for 0.25 mL final volume.
4.13.5.8 TR-TQ spike solution 250 ug/L – use 100 uL for 0.25 mL final volume.

4.13.6 Spiking solutions for the GC/MS NCI tests

4.13.6.1 Chlordane spike at 2500 ug/L – use 80 uL for 2.0 mL final volume
4.13.6.2 Toxaphene spike at 10000 ug/L – use 200 uL for 2.0 mL final volume
4.13.6.3 Chlordane spike 10xdil – use 160 uL, for S-PQL spike only, use 80 uL for T-PQL spike only.
4.13.6.4 Toxaphene spike 10xdil – use 400 uL for S-PQL spike only, use 200 uL for T-PQL spike only
4.13.6.5 CL-TQ surrogate spike solution 200 ug/L of TMX and DCB – use 200 uL for 2.0 mL final volume.

5. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

5.1 Water samples are collected in one liter, glass amber bottles with Teflon-lined screw caps. They are stored at 4 °C. All water samples (except for environmental tracers, test ID: W-TR-TQ) must be extracted within 7 days of collection. Environmental tracers samples must be extracted within 14 days. If samples are to be analyzed for Aldrin and residual Chlorine is present, then add 80 mg of Sodium Thiosulfate per 1 L of sample and mix well.

5.2 Sediment samples are collected in 250-500-1000mL glass jars with Teflon-lined screw caps. Samples are stored at 4 °C. All sediment samples must be prepared within 14 days of collection.

5.3 Waste samples may be collected in a variety of containers, depending on the nature of the waste. All samples are stored at 4 °C and must be extracted within 14 days of collection.

5.4 Filter samples are collected in 250-500mL glass jars with Teflon-lined screw caps. Samples are stored at 4 °C and must be extracted within 14 days of collection.

6. WATER SAMPLE PREPARATION PROCEDURE

(Based on EPA 3510C for RCRA and/or GC/MS/MS samples and the 600 series methods mentioned in 1.1 for CWA samples)

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NOTE: A SECOND PERSON MUST WITNESS THE ADDITION OF SPIKE STANDARD TO THE PROPER SAMPLES AND VERIFY THE CORRECT VOLUME AND CONCENTRATION, AND INITIAL IN THE SAMPLE PREPARATION REPORT.

6.1 CHLORINATED/NITROGEN PHOSPHORUS PESTICIDES AND PCB's (for analysis by GC/ECD and/or GC/NPD)

6.1.1 Weigh the sample bottle before and after taking your sample aliquot or mark the water meniscus with tape on the side of the sample bottle for later determination of sample volume. Pour the entire sample into a 2-liter separatory funnel. Use 1L of well water for blanks and laboratory-fortified blanks. The well water for blanks and LFBs is pre-cleaned by extraction with 60 mL of methylene chloride. When weighing, assume sample has a density of 1.0g/mL. If in doubt, i.e., if sample contains a significant amount of oils or solvent, weigh an exact volume, 5-10mL to determine density). Measure the same extraction volume for matrix spike, matrix spike duplicate and sample spiked, usually 1000mL, using a 2-liter volumetric cylinder. For each extraction batch perform a single method blank, two LFBs and duplicate matrix spike (if enough sample volume is available). Additionally, if an extraction batch contains more than 10 water samples run under Clean Water Act (CWA; NPDES complaint), an additional water sample needs to be spiked. Water samples run under the Clean Water Act will be logged in under test IDs: W-NP-AA-SF, andW-PC-AA-SF.

6.1.2 Using a clean glass rod or Pasteur pipet, transfer a few drops of the sample to a piece of wide range pH paper. Record pH in the logbook. For methods 8081 and 8082 the pH must be in the range of 5-9. For 8141 pH must be "as received" (See EPA 3510C ref.15.5) and adjust the pH if necessary using 1:1 (v/v) sulfuric acid or 10.N sodium hydroxide.

6.1.3 Add 200 uL of 10x diluted ECD/NPD surrogate spike mix to all samples and method blank. Add the required analyte spikes (check work order for specifics, rotate spikes on routine basis) to the laboratory-fortified blanks and matrix spikes.

6.1.4 Add 60mL of methylene chloride to the sample bottle, and swirl for 30 seconds to rinse the inner surface. Transfer this rinsate to the separatory funnel.

6.1.5 Stopper each funnel and secure the tumbler lid.

6.1.6 Turn on the tumbler slowly for a half turn, stopping with the funnels upside down. Slowly open the stopcocks to vent the pressure. Close the stopcocks.

6.1.7 Repeat step 6.1.6 at least three more times, turning one full turn then two full turns then four full turns, respectively, before venting. Continue until the amount of gas escaping is minimal.

6.1.8 Turn on the tumbler again and set the speed to about 30-35 turns per minute. Tumble for three minutes.

6.1.9 Stop the tumbler with the funnels upright (stopcocks down) and lock it in place.

6.1.10 Remove the stoppers from the separatory funnels and allow the methylene chloride and water layers to separate for at least 10 min. If an emulsion forms, follow one or all of these techniques to break up:

- Allow separatory funnel to stand several additional minutes

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- Gently agitate emulsion with a long glass-stirring rod
- Sonicate the emulsion
- Centrifuge the emulsion
- Add solid NaCl and shake
- Add more methylene chloride
- Filter emulsion through glass wool.

6.1.11 Filter the bottom methylene chloride layer through an extraction column fitted with a glass wool plug into a 250mL Erlenmeyer flask.

6.1.12 Rinse the funnel with 2 x 5mL of methylene chloride and collect through the same column. This should be done only for the first tumbling.

6.1.13 Repeat steps 6.1.6- 6.1.11 for a total of three extractions.

6.1.14 Rinse the extraction column into the flask with 20mL of methylene chloride.

6.1.15 If any water is collected, try to remove it in one of the following ways:

- Pour the extract back into the same separatory funnel, and collect the bottom (organic) layer. Rinse the top (aqueous) layer with a few milliliters of methylene chloride, and collect the bottom layer again.

- Use a Pasteur pipet to remove the top (aqueous) layer.

- Sprinkle granular Na_2SO_4 into the extract to trap the water droplets. Carefully decant methylene chloride layer into K&D set up without Na_2SO_4. Rinse flask three times with 5mL of methylene chloride and add it to the K&D set up.

6.1.16 Assemble a Kuderna-Danish (K&D) solvent evaporator by attaching a 15mL concentrator tube to a 500mL evaporative flask, using clips to attach. Rinse the assembly with methylene chloride and discard the rinsate.

6.1.17 Prewet a Snyder column by adding about 1mL of methylene chloride to the top.

6.1.18 Transfer extract into K&D, using methylene chloride to rinse the flask three times, 5mL each. Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column.

6.1.19 Place the K&D apparatus on a hot water bath (70 °C) so that the concentrator tube is partially immersed in the hot water.

6.1.20 Concentrate to 3-4mL and then remove K&D apparatus from water bath and allow it to cool.

6.1.21 Carefully disconnect Snyder column, and then rinse flask with 1 or 2mL of methylene chloride into tube. Carefully disconnect K&D flask from tube. MAKE SURE CONCENTRATOR TUBE IS LABELED WITH THE CORRECT SAMPLE NUMBER!!

6.1.22 Setup a Nitrogen Evaporator (N-Evap™) by turning on nitrogen-gas, heater (40 °C bath temperature), and attaching clean needles.

6.1.23 Open the valves for each needle and check for required flow of Nitrogen with blank solvent in a centrifuge tube.

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6.1.24 Before transferring the sample extract (after K&D concentration) to a centrifuge tube, calibrate the tube to 4mL and to 0.50 mL. Measure 2mL of liquid with a gastight syringe and add to an empty, dry, graduated centrifuge tube. Mark where the meniscus is (for example, if 2.0mL of liquid fills the tube to the 2.1mL graduation mark) and also write this on the side of the tube as 2.1/2.0.After determining the true volume of 2mL on this tube, use the meniscuses that you have marked, rather than the original graduations on the tube.

6.1.25 If samples are to be analyzed only by NPD solvent exchange to isooctane and concentrate to a final volume of 2 mL .Otherwise concentrate to 4mL in methylene chloride. Add internal standard for NPD analysis. For ECD, all samples in the set must undergo GPC cleanup. Filter 1.5 mL out of 4mL extract through 0.45µm filter into a 1.5mL Autosampler vial. Inject 1.0 mL in GPC column for cleanup followed by 1 gram Florisil cleanup (see sec. 11.4). Solvent exchange and make up to final volume of 0.50mL with iso-octane in a calibrated centrifuge tube. For NPD, take 1mL of extract, add 1mL iso-octane, N-blow down to 0.5mL and make up to final volume of 0.50mL in iso-octane.

6.2 **DIELDRIN WITH ULTRA LOW DETECTION LIMITS**
((for analysis by GC/MS/MS) LIMS test: W-PSCL-TQ

For Dieldrin only extractions (samples logged in for W-PSCL-TQ test only) extract as in section 6.1 with the following exceptions:

6.2.1 When spiking, use 50 uL of CL-TQ surrogate mix and 100 uL of CL-TQ spike mixture (or 100 uL of 200 ppb Dieldrin spike).

6.2.2 Solvent exchange the extract to isooctane and concentrate to 0.4 mL. Enter the final volume as 0.50 mL in the extraction report to account for the internal standard addition. No cleanup is necessary.

For Dieldrin + other Chlorinated Pesticides extractions (samples logged in for W-PSCL-TQ and W-PC-AA-SF tests sharing one bottle) extract as in section 6.1 with the following exceptions:

6.2.3 Use surrogate/spikes amounts as for the W-PC-AA-SF extractions. For pesticides spikes different than ECDA additionally spike 100 uL of Dieldrin spike.

6.2.4 Concentrate to 4mL in methylene chloride. Split the extract into two equal parts. With the first part proceed like for a regular W-PC-AA-SF extraction without the NPD test. Subject the extract to GPC/Florisil cleanup as described in 6.1.25 and submit for GC-ECD analysis. Due to limited extract volume this extraction pathway does not allow for the simultaneous preparation for the GC-NPD analysis.
For the second half of the extract solvent exchange to isooctane and concentrate to 0.2 mL. Enter the final volume as 0.50 mL in the extraction report to account for the extract split and internal standard addition.

6.3 **NITROGEN/PHOSPHORUS PESTICIDES WITH ULTRA LOW DETECTION LIMITS**
(for analysis by GC/MS/MS) LIMS Test: W-PSNP- TQ

Extract as in section 6.1 with the following exceptions:

6.4.1 When spiking, use spike mixtures (surrogates and analytes) specially prepared for GC/MS/MS analysis. Add 50 uL of surrogates spike (Simazine-d10 and Malathion-

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d7 at 100 ug/L each to all samples, and 50 uL of required analyte spike to LFBs and Matrix Spikes.

6.4.2 DI water, rather than well water, is used to prepare extraction blanks and LFBs due to trace levels of herbicides, mostly atrazine, usually found in well water. The pH of DI water is adjusted to exactly 7 by adding about six drops of 12% solution of sodium bicarbonate. The pH adjustment prevents random and sometimes significant loses of certain analytes, primarily Hexazinone.

6.3.3 Solvent exchange the extract to isooctane and concentrate to 0.2 mL. Enter the final volume as 0.25 mL in the extraction report to account for the internal standard addition.

6.4 **ENVIRONMENTAL TRACERS**

By GC/MS/MS, LIMS test; W-TR-TQ

Extract as in section 6.3, with the following differences:

6.4.1 When spiking, use spike mixtures (surrogates and analytes) specially prepared for GC/MS/MS analysis. Add 50 uL of tracers surrogates spike (Dibutyl butylphosphonate and Terphenyl-d14 at 250 ug/L each) to all samples, and 100 uL of the tracers spike (each analyte at 250 ug/L) to LFBs and Matrix Spikes.

6.4.2 Extreme care has to be exercised by all the laboratory staff involved in preparation and analysis to limit background contamination with personal care products and flame retardants.. Three method blanks are to be extracted with each sample batch to account for variability in background contamination. So called “dry blanks” should be performed since even the cleanest DI water would usually show higher background levels of environmental tracers than the “dry blanks”. To extract the so-called “dry blanks” perform three separate Blank samples without any water matrix added.

7. SEDIMENT SAMPLES (BASED ON EPA 3550C)

Sediment samples for chlorinated pesticides analysis using GC/MS/MS (test ID: S-PSCL-TQ, S-PCB-TQ) or GC/MS-NCI (test ID: S-CT-CI). And/or Nitrogen phosphorus pesticides by GC-NPD (test ID: S-PEST-NP)
NOTE: A SECOND PERSON MUST WITNESS THE ADDITION OF SPIKE STANDARD TO THE PROPER SAMPLES AND VERIFY THE CORRECT VOLUME AND CONCENTRATION, AND INITIAL IN THE LOG BOOK.

7.1 If the samples have not already been homogenized, homogenize each sample by mixing it thoroughly using a metal spatula. For most samples, you will need to remove the sample from the jar and place it on a sheet of aluminum foil (dull side up, shiny side down) in order to homogenize it fully. Do not use aluminum foil if the sample is to be analyzed for metals. Consult your supervisor for directions if obvious non-representative material is found in the sample.

7.2 Perform a dry weight determination on all samples. This determination is currently performed by the Metals Groups for all sediment samples coming in our Laboratory

7.3 Weigh approximately 30g ± 0.1g of wet sediment sample into a 400mL beaker. Record the weight.

7.4 For GC/NPD analysis add 1 mL of the NPD surrogate (NMX). to all samples Add required analyte spikes (check work order for specifics) to the Laboratory Fortified

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Blanks and Matrix Spikes. For NPD analysis add 1000 uL of NPDA or NPDB+C spike depending on rotation schedule. When spiking for chlorinated pesticides/PCBs tests, use spike mixtures (surrogates and analytes) specially prepared for GC/MS/MS analysis. Add 200 uL of surrogates spike (TMX and DCB at 200 ug/mL) to all samples, and 200 uL of required analyte spike to LFBs and Matrix Spikes. For PCBs analysis add 160 uL of PCB 1060 spike. For Chlordane and Toxaphene analysis using S-CT-CI test use regular ECD spike solutions as indicated in 4.13.6. An additional PQL spike has to be prepared for each batch of samples for this test. If S-PCB-TQ test is requested Chlordane and Toxaphene may be be spiked simulteneaously with PCB-1060.

7.5 Add 60g anhydrous powdered sodium sulfate and mix well. Wet samples should be mixed more frequently than dry samples, until they are sandy and free flowing. DO NOT ADD MORE Na_2SO_4.

7. 6 Immediately add 100mL of 1:1 methylene chloride: acetone to the sample and cover with aluminum foil. Sonicate for three minutes.

7.7 Set up for vacuum filtration using a 500mL filtration flask, 9 cm Buchner funnel and Whatman number 42 9-cm filter paper. Rinse the filtration setup with methylene chloride and discard the solvent to waste.

7.8 Decant the sample extract through the Whatman # 42 filter paper.

7.9 Repeat steps 7.1.6. -7.1.8. two more times, filtering extracts through the Buchner funnel and collecting in the flask. After the third time transfer the sediment from the beaker to the Buchner funnel.

7.10 Rinse the beaker, funnel and soil cake with methylene chloride, adding rinses to the flask.

7.11 Assemble a Kuderna-Danish (K&D) concentrator by attaching a 15mL concentrator tube to a 500mL evaporative flask, using clips to attach. Rinse the assembly with methylene chloride and discard the rinsate.

7.12 Pre-wet a Snyder column by adding about 1mL of methylene chloride to the top.

7.13 Transfer extract into K&D, using methylene chloride to rinse the flask three times 5mL each. Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column.

7.14 If any solid particles are present in the extract, then filter through Na_2SO_4 funnel. If any water droplets are separated, then sprinkle granular Na_2SO_4 into the extract to trap the water droplets. Carefully decant into K&D set up without collecting Na_2SO_4. Rinse flask three times with 5mL of methylene chloride and add it to the K&D set up.

7.15 Place the K&D apparatus on a hot water bath (80 to 85 °C) so that the concentrator tube is partially immersed in the hot water.

7.16 Concentrate to 3-4mL then remove K&D apparatus from water bath and allow it to cool.

7.17 Carefully disconnect Snyder column, and then rinse flask with 1 or 2mL of methylene chloride into tube. Carefully disconnect K&D flask from tube. MAKE SURE

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CONCENTRATOR TUBE IS LABELED WITH THE CORRECT SAMPLE NUMBER!!

7.18 Setup N-Evap® by turning on nitrogen-gas, heater (40 °C bath temperature set to about 1.5), and attaching clean needles.

7.19 Open the valves for each needle and check for required flow of Nitrogen with blank solvent in a centrifuge tube.

7.20 Place concentrator or centrifuge tubes into N-Evap® tray, then carefully pull needles down to approximately 2 inches from solvent level. Make sure N_2 flow is adequate, but be careful NOT to blow N_2 too vigorously into sample, causing splashing.

7.21 Transfer the extract to the centrifuge tube with a Pasteur pipet. Rinse the concentrator tube twice with 1mL of methylene chloride and N-blow down to 2mL and make up to 10mL in methylene chloride.

7.22 If only chlorinated pesticides tests are requested blow down the extract to 2.0 mL final volume. Subject 1.0 mL of extract to GPC/Florisil clean up. Inject 0.8 mL of extract on the GPC column; after the entire clean up procedure is completed concentrate the sample to 0.7 ml in isooctane to account for the internal standard addition. For sulfuric acid resistant analytes perform sulfuric acid clean up followed by TBA using 1 mL of the original extract. Blow down to 0.9 mL to account for the internal standard addition and submit for

7.23 If the GC/ NPD analysis is requested the 10 mL methylene chloride extract needs to be split in half. For NPD analysis take 1mL of extract out of 5mL NPD portion and solvent exchange to iso-octane. The Chlorinated pesticides 5 mL extract should be further concentrated to 1 mL and then subjected to the GPC/1 g florisil clean up as described above in 7. 7.24

7.24 **Save the unused sample extract in the original tube with the remaining volume clearly marked with initials and date.**

8. WASTE SAMPLE (BASED ON EPA 3580C)

Test ID: WAS-PST-NP, WS-PSCL-TQ, WS-PCB-TQ, WS-CT-CI

This procedure is for samples soluble in organic solvents. For an extraction batch of no more than 20 samples, make one Method Blank, one or two Laboratory Fortified Blanks, one Sample Matrix Spike (MS) and one Sample Matrix Duplicate (MSD). Multiphase samples must be prepared by the phase separation method (Lab SOP CM-027-1).

8.1 For each sample and Matrix Spike, weigh approximately 1g of sample into a 15 mL centrifuge tube (record weight to the nearest 0.001g).

8.2 Weigh 1g of a reagent having a similar matrix as the sample, into centrifuge tubes (e.g. corn oil for oil samples etc.) for blanks and lab fortified blanks.

8.3 For GC/NPD analysis add 1 mL of the NPD surrogate. to all samples .Add required analyte spikes (check work order for specifics) to the Laboratory Fortified Blanks and Matrix Spikes.For NPD analysis add 1000 uL of NPDA or NPDB+C spike depending on rotation schedule. When spiking for chlorinated pesticides/PCBs tests, use spike mixtures (surrogates and analytes) specially prepared for GC/MS/MS analysis. Add 1000 uL of surrogates spike (TMX and DCB at 200 ug/mL) to all samples, and 1000

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uL of required analyte spike to LFBs and Matrix Spikes. For PCBs analysis add 800 uL of PCB 1060 spike. For Chlordane and Toxaphene analysis using S-CT-CI test use 5x the amounts of regular ECD spike solutions as indicated in 4.13.6. An additional PQL spike has to be prepared for each batch of samples for this test. If S-PCB-TQ test is requested Chlordane and toxaphene should be spiked simulteneaously with PCB-1060.

8.4 Dilute each sample to 10mL with iso-octane. Cap tightly and vortex. Perform appropriate cleanup procedures as needed (GPC, Florisil, Sulfuric Acid). Proceed with GC analysis.

9. EXTRACTION OF FILTERSAMPLES (BASED ON EPA 3540C)

9.1 FILTER SAMPLES - BY SOXHLET EXTRACTION

9.1.1 Fill 500mL round-bottom flask with 250mL of methylene chloride, set in a support ring, and then add four or five boiling chips to flask. Fit an extractor body on top of flask.

9.1.2 Fold the filter paper into quarters, and then push the filter to the bottom of the extractor body using forceps.

9.1.3 Spike at this point with the assigned spiking solution and volume and surrogates spike solution.

9.1.4 Place the assembled extractor on the Rot-x-tract apparatus for 18 hours at 75 °C.

9.1.5 After 18 hours, remove the extractor from the apparatus, rinsing all parts into the extractor with methylene chloride. Tilt the extractor to empty its contents into the attached 500mL flask, remove the body from the flask and rinse the joint with methylene chloride, catching the rinsate in the flask.

9.1.6 Transfer the extract to an assembled K&D apparatus, **add fresh boiling chips** and attach a Snyder column.

9.1.7 Concentrate to 3 to 4mL and transfer extract in to a pre-calibrated centrifuge tube with methylene chloride and 1mL of iso-octane.

9.1.8 N-blow down to 0.4mL and bring to a final volume of 1.0 mL with iso-octane.

10. ELECTRONIC SAMPLE PREPARATION LOGS

Each sample preparation must be recorded by the preparation team in the electronic Prep Worksheet Manager module of LIMS. For pesticides/tracers analyses there are several, ready- made, partially prefilled templates in LIMS. The template names are identical with LIMS prep IDs:
SOP-GC-002 - (GC/ECD/NPD water tests + W-PSCL-TQ
3510C (TQ) – W-PSNP-TQ
3510C (TR) – W-TR-TQ
3550C (GC) – S-PEST-NP
3550C (TQ) – S-PSCL-TQ, S-PCB-TQ, S-CT-CI
3580A (GC) – WAS-PST-NP
3580A (TQ) – WS-PSCL-TQ, WS-PCB-TQ, WS-CT-CI
USGS-O-1126-95 –W-PCL-SQ, W-PCL-SQ-R
Many fields in both the header and the grid are prefilled and seldom change. They need, however, to be always checked for correctness.

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Header fields in the electronic preparation logs:

- **Worksheet ID#:** *this is assigned automatically by LIMS. Example 3510C (TQ)_41*
- **Prep ID:** *LIMS prep ID defined in LIMS and connected with test ID. Each test ID may only be connected to one Prep ID, but many test IDs may use the same prep method.*
- **Prepared By**:
- **Prep Start Date/Time**:*This will be the prep date in LIMS reports*
- **Spike Witness**;
- **Prep Completion Date/Time**:
- **Date/Time Reviewed**:
- **Reviewed By**:
- **Balance ID**: *pre-entered , usually the same*
- **Spike 1:** *Std tracker SN; this is usually surrogate*
- **Spike 2:** *Std tracker SN; this is the first of the spikes used*
- **Spike 3:** *Std tracker SN; this is the second of the spikes used*
- **Spike 4:** *Std tracker SN; this is the third of the spikes used*
- **Spike 5:** *Std tracker SN; this is the fourth of the spikes used*
- **Spike 6:** *Std tracker SN; this is the fifth of the spikes used*
- -different numbers of spikes may be applicable for specific extractions
- **Water bath temp:** *in deg C*
- **Final Extract Solvent:** *Isooctane_ lot number*
- **Solvent 1** *Methylene Chloride_ lot number*
- **Solvent 2** *Acetone_ lot number*
- **Solvent 3** *Hexane_ lot number*
- **Solvent 4** *Isooctane_ lot number*
- Different numbers of solvents may be used in different procedures
- **Reagent 1-** ***any reagent that may be used in the preparation***
- **Cleanup procedures:** *GPC File: 100914GPC_3*
- **Prep comments:** *enter other chemicals/supplies here with lot numbers; example Ottowa sand., Florisil lot# etc.*
- **Internal standard:** *enter the ISTD information like this: ISTD SN: 53803 10uL, 10-10-2014 IC*

Grid Fields in the electronic preparation Logs

Prep Batch ID: ***consecutive "P" number assigned by LIMS in the form of P123456. The same number has to be used for sample reporting in QC manager.*****Sample ID**: *the sample IDs are selected in LIMS when the worksheet is created.; for QC samples those rows are typically pre-filled in the template*
Bottle #: *Water samples only; entered as ¼ - bottle 1 of*

- **Lab pH**: *The pH of the sample measured with wide range pH ; waters only.test strips.*
- **Sample ID**; *the laboratory sample Id assigned in LIMS*
- **Test ID**: *The analysis requested for the sample.*
- **Matrix:** *matrix assigned in LIMS*
- **Sample Wt (g)/ Volume (mL) extracted**: *For water samples the volume extracted is determined by subtracting the mass of the empty sample bottle from the mass of the sample bottle + sample. For QC samples required volume is measured using a graduated cylinder. For other sample types the mass of the sample itself is measured e.g. 30g wet sample for soil/sediment 1g for waste samples.*

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- **% solid** – *percent solid content as determined by the metals group. This value is imported with the sample info.*

- **Sample Dry Weight –** *automatically calculated by the software*

- **Final extract Volume (mL**: The volume of the sample extract after full concentration step; 2 mL for regular water/sediment/waste sample extraction
  - **Clean-up procedure**: *The clean-up procedure used, if applicable. Write down anything done differently than SOP requirements.*
  - **Spike 1:** *volume of surrogate spike added*
  - **Spike 2:** *volume of the first spike added*

- **Comments:** *This section should include any observations regarding unusual appearance or odor of the sample, any dilutions made during preparations or any other remarks about the preparation*.

11. CLEANUP PROCEDURES

11.1 Cleanup procedures may not be necessary for a relatively clean sample matrix. If particular circumstances demand the use of a cleanup procedure, select from the procedures described below. All sample cleanup preparations shall be recorded in the electronic preparation worksheet.

11.2 TETRABUTYLAMMONIUM - SULFITE (TBA-SULFUR) (used for sulfur cleanup) (BASED ON EPA 3660B)

11.2.1 Prepare a solution of tetrabutylammonium hydrogen sulfate by dissolving 3.39g of tetrabutylammonium hydrogen sulfate in 100mL of deionized water. To remove impurities, extract this solution three times with 20mL portions of hexane. Discard the hexane extracts. Add 25g sodium sulfite to the water solution. Store the resulting solution, which is saturated with sodium sulfite, in an amber bottle with a Teflon-lined screw cap. Label date and initial. Enter the reagent preparation into the DEP standard/reagent tracker in LIMS. This solution can be stored at room temperature for one month. Enter the preparation in the standard preparation tracker under reagent category and obtain a Serila Number. Print the label and affix to the reagent container.

11.2.2 To remove sulfur from sample extract, transfer 2mLs of sample extract (in iso-octane or hexane) to a 40mL clear vial or a centrifuge tube with a Teflon-lined screw cap. Add 1mL of TBA - Sulfite reagent and 2mL of 2-propanol. Cap the vial and shake for 1 minute.

11.2.3 If the sample is colorless or if the initial color is unchanged, and if clear crystals (precipitated sodium sulfite) are observed, sufficient sodium sulfite is present. If the precipitated sodium sulfite disappears, add more crystalline sodium sulfite in approximately 100mg portions until a solid residue remains after repeated shaking.

11.2.4 Add 5mL distilled water and shake for at least 1 minute. Allow the sample to stand for 5-10 minutes and centrifuge. Transfer the top hexane layer to a 2mL GC vial or to a centrifuge tube for analysis.

11.3 GPC CLEANUP PROCEDURE (BASED ON EPA 3640A)

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GPC Cleanup procedures are applied to all sediment samples, group AA water samples and may be used for any sample if needed. See SOP GC-029-1 for details (Ref. 15.16).

11.4 ONE GRAM FLORISIL CARTRIDGE CLEAN UP (BASED ON EPA 3620C)

11.4.1 This procedure is routinely used for Chlorinated Pesticides and PCB. Clean vacuum manifold valves and needles with acetone.

11.4.2 For each sample place a properly labeled one-gram Florisil cartridge into the vacuum manifold.

11.4.3 Wash each cartridge with 5mL of 10% acetone in hexane.

11.4.4 Apply and adjust vacuum if needed (vacuum is not usually needed), but do not let the cartridges go to dryness.

11.4.5 Place a rack containing labeled 15mL centrifuge tubes inside the manifold, and replace the manifold top. Make sure that each cartridge position empties into the appropriate centrifuge tube.

11.4.6 Transfer crude extract (approximately 1mL) to the top frit of the appropriate Florisil cartridge.

11.4.7 Rinse each tube with 3mL of hexane-acetone (90:10) solution. Add rinsate to cartridge. Repeat twice and finally rinse each cartridge twice with 1mL of hexane.

11.4.8 N-blow down to 0.8mL and make up to 1mL in iso-octane. The final volume should result in a concentration equal to that of the extract initially taken for clean up.

11.4.9 If sulfur clean up is required, proceed to "TBA-Sulfur clean up (Sec. 11.2)". Transfer final extract to a labeled GC vial.

11.5 SULFURIC ACID CLEAN UP (USED FOR SAMPLES BEING ANALYZED FOR THE ACID RESISTANT PESTICIDES) (BASED ON EPA 3665A)

NOTE: This cleanup will destroy some pesticides. Do sulfuric acid clean up on part of the sample. DO NOT TREAT ENTIRE SAMPLE WITH ACID!!! See Lab SOP GC-011-5. for analytes amenable to H_2SO_4 cleanup.

11.5.1 Add 8mL of concentrated sulfuric acid to 2mL of sample extract (in iso-octane) in a screw-top culture tube. Be sure the top of the tube has no chips or cracks.

11.5.2 Shake for 30 seconds, allow extract to settle for five minutes and then centrifuge for five minutes.

11.5.3 If the bottom layer is colored, then transfer the top layer to a tube containing 8mL of fresh sulfuric acid and repeat step 11.6.2. This step may need to be repeated until a colorless acid layer is observed.

11.5.4 Transfer the supernatant into a clean centrifuge tube. Extreme care must be exercised at this point. All of the sulfuric acid layer must be avoided during transfer, as this layer will destroy the GC. Processing the extract through 1g Florisil column (Sec. 11.4) will eliminate this danger.

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11.5.5 All sediment extracts from concentrated sulfuric acid clean up without GPC need TBA-SULFUR clean up (Sec. 11.2).

12. SAMPLE ANALYSIS PROCEDURE

Reference applicable analysis SOPs: GC-011-5, GC-012-3., GC-027-1 , GC-033-1 and GC-034-1 and Gc-035-1 (See Ref. 15.13, 15.14, 15.15, 15.20, 15.21, 15.22).

13. QUALITY CONTROL

See QA/QC Lab SOP GC-001-2 (See Ref. 15.17).

14. SAFETY/HAZARDOUS WASTE MANAGEMENT

14.1 Wear safety glasses, gloves and a lab coat at all times while performing sample preparation! Methylene chloride will burn any skin it contacts; so wear nitrile gloves (Latex gloves will not stand up to methylene chloride.).

14.2 Handle all the sample extraction, solvent transfer, concentration, sample preparation, and clean up in a hood at all times. Avoid breathing methylene chloride vapors. All extractions MUST be prepared in a fume hood. When not working directly with samples, pull the hood sash down to the bottom. Do NOT lift the hood sash above the halfway point (marked with a red line) while tumbling samples.

14.3 Discard all methylene chloride waste in the appropriate Chlorinated Waste bottles. Keep chlorinated and non-chlorinated waste solvents separate. If mixed, record the volume of each and dispose with chlorinated waste.

14.4 Dispose of leftover and expired neat standards into a sealed, labeled container.

14.5 Dispose of all the stock standards, working standards and used sample extracts into a labeled chlorinated waste container.

14.6 Dispose of all used GC vials containing standards or samples into a labeled chlorinated waste container.

14.7 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available.

14.8 A reference file of material data handling sheets is kept in the office area, across from the entrance to the tech room (room A316J). Refer to this file for any material you handle. Update this file upon receiving new chemicals or reagents. The MSDS for many chemicals can also be viewed on-line, at http://hazard.com/MSDS/

14.9 Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when an analyst handles high concentrations of these toxic compounds.

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14.10 Dispose of all unwanted, broken glassware into a broken glassware disposal box. Inspect every piece of glassware. Do not use any that are chipped, cracked, etched, or scratched. Glassware with minor damage should be stored for repair.

14.11 See Safety-Waste SOP OG-001 for further details (See 15.17).

15 REFERENCES

1. EPA 608 Methods For Organic Chemical Analysis Of Municipal And Industrial Wastewater. Organochlorine Pesticides And PCBs. 40 C.F.R. Appendix To Part 136, 2008.
2. EPA 8081B Organochlorine Pesticides by Gas Chromatography; Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods, SW 846; 3rd Edition, Update IV, January 3, 2008.
3. EPA 8082A, Polychlorinated Biphenyls (PCBs) by Gas Chromatography; Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods, SW 846; 3rd Edition, Update IV, January 3, 2008
4. EPA 8141B Organophosphorus Compounds by Gas Chromatography, Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods, SW 846; 3rd Edition, Update IV, January 3, 2008
5. EPA 3510C Separatory Funnel Liquid-Liquid Extraction, SW 846; 3rd Edition, Update IV, January 3, 2008.
6. EPA 3540C Soxhlet Extraction, SW 846; 3rd Edition, Update IV, January 3, 2008.
7. EPA 3550C Ultrasonic Extraction, SW 846; 3rd Edition, Update IV, January 3, 2008.
8. EPA 3580A Waste Dilution, SW 846; 3rd Edition, Update IV, January 3, 2008.
9. EPA 3620C Florosil Cleanup, SW 846; 3rd Edition, Update IV, January 3, 2008.
10. EPA 3640A Gel-Permeation Cleanup, SW 846; 3rd Edition, Update IV, January 3, 2008.
11. EPA 3665A Sulfuric Acid Cleanup, SW 846; 3rd Edition, Update IV, January 3, 2008.
12. EPA TO-10A Determination of Pesticides and Polychlorinated Biphenyls in ambient air using low volume Polyurethane Foam (PUF) sampling followed by Gas Chromatographic/Multi-Detector Detection (GC/MD), January 1999.
13. DEP SOP GC-011-5, Analysis of Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) in water, soil/sediment, and wastes by Gas Chromatography with Electron Capture Detection (GC/ECD).
14. DEP SOP GC-012-3, Analysis of Organonitrogen and Organophosphorus Pesticides in water, soil/sediment, and wastes by Gas-Chromatography with Nitrogen-Phosphorus Detection (GC/NPD).
15. DEP SOP GC-027-1, Analysis of Organochlorine Pesticides and PCBs in Polyurethane Foam Filters for Ambient Air Analysis by Gas Chromatography with Electron Capture Detection (GC/ECD).
16. DEP SOP GC-029-1, Gel-Permeation Chromatography (GPC) cleanup procedure for Chlorinated Pesticides and PCBs Analysis (based on EPA Method 3535A).
17. DEP SOP GC-001-2, Quality Assurance/Quality Control in the GC Pesticides Laboratory.
18. DEP SOP OG-001-3, Safety and Waste management practices in the Organic Analysis Group (Volatile, Semi-Volatile, and Pesticides Laboratories).
19. DEP SOP CM-027, Result Calculations for Multi-Phasic Samples
20. DEP SOP GC-033-1, Analysis Pesticides and PCBs in Water and Soil Samples Using GC/MS/MS.
21. DEP-SOP GC-034-1, Analysis of Chlordane and toxaphene in Soil using GC/MS with Negative Chemical Ionization.
22. DEP-SOP GC-035-1, Analysis of Environmental Tracers in Water Samples Using GC/MS/MS.

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APPENDIX OF SIGNIFICANT CHANGES

7/18/2013: The following changes were made:

1) Paragraph 1.3; Test names S-PSCL-TQ and S-PCB-TQ added
2) Paragraph 4.13 Spiking solutions concentrations were updated; spiking descriptions for the triple quad tests were added;
3) Paragraph 7.2 Description for the extraction for the triple quad tests, including spiking, was added.

9/9/2013: The following changes were made;

1) Addition of S-CT-CI test throughout the text
2) Addition of well water matrix pre-treatment (6.1.1)
3) Addition of DI water blank matrix and its treatment (6.3.2)

10/22/2013 The following changes were made:

1) Added requirement to spike additional sample if more than ten CWA samples are extracted in one batch. (6.1.1)
2) Added requirement to include GPC instrument number/EZChrom sequence info on the preparation log. (6.4 and 7.3)

10/20/2014:

1) Addition of newly validated tests throughout the text
2) Corrections to spiking procedures and concentrations
3) Addition of Electronic Preparation Logs

10/30/2015:

1) Removal of PUFs extraction – no longer performed.
2) Corrections to spiking procedures and concentrations
3) Removal of % solid determination –performed by the Metals Group.
4) Removed section 11.4.10. The Florisil checks were only performed long time ago for bulk florisil.

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EXAMPLE OF ELECTRONIC PREPARATION LOG

Printed: 10/17/2014 **SOP-GC-002_1485** Page 1 of 2

Worksheet ID: SOP-GC-002_1485
Prep ID: SOP-GC-002
Prepared By: GHAFFARI_R
Prep Start Date/Time: 10/09/2014 09:00
Spike Witness: SIDDELL_F
Prep Completion Date/Time: 10/10/2014 11:00
Date/Time Reviewed: 10/10/2014 08:45
Reviewed By: GHAFFARI_R
Balance ID: Adventurer ProAV4101c SN#8029281092
Spike 1: 53619 SURR SPIKE
Spike 2: 53399 TOXA STOCK
Spike 3: 56719 NPDB+C SPIKE
Spike 4:
Equipment ID(s):
Water Bath Temperature (C): 70 C
Final Extract Solvent: Isooctane_115919
Solvent 1: Methylene chloride_145389
Solvent 2: Acetone_136059
Solvent 3: n-Hexane_130600
Solvent 4: Isooctane_115919
Reagent 1:
Cleanup Procedures: GPC File:100914GPC_3
Prep Comments: Florisil lot# 734460-930973
Internal Standard: ISTD Sn 53803 10uL, 10-10-2014, IC

Prep Batch ID	Bottle #	Lab pH	Sample ID	Test ID	Matrix Type	Sample	Final Extract	Sample Cleanup	Spike 1	Spike 2	Spike 3	Spike 4
P275003 10X dil. surr spk		7	MBLK	W-NP-AA-SF	FDEP WELL	1000	2		200uL			
P275003 10X dil. surr spk		7	MBLK	W-PC-AA-SF	FDEP WELL	1000	2	GPC/1g Florisil	200uL			
P275003 10X dil. surr spk		7	LFB_1	W-PC-AA-SF	FDEP WELL	1000	2	GPC/1g Florisil	200uL	200uL		
P275003 10X dil. surr spk		7	LFB_1	W-PC-AA-SF	FDEP WELL	1000	2	GPC/1g Florisil	200uL	200uL		
P275003 10X dil. surr spk	2/4	8	MS-1641903	W-PC-AA-SF	W-EFFLUENT	1000	2	GPC/1g Florisil	200uL	200uL		
P275003 10X dil. surr spk	3/4	8	MSD-1641903	W-PC-AA-SF	W-EFFLUENT	1000	2	GPC/1g Florisil	200uL	200uL		
P275003 10X dil. surr spk	2/3	6	MSD-1652415	W-PC-AA-SF	W-SURF-FRH	1000	2	GPC/1g Florisil	200uL	200uL		
P275003 10X dil. surr spk		7	LFB-1	W-NP-AA-SF	FDEP WELL	1000	2		200uL		200uL	
P275003 10X dil. surr spk		7	LFB-2	W-NP-AA-SF	FDEP WELL	1000	2		200uL		200uL	
P275003 10X dil. surr spk	2/4	8	MS-1641903	W-NP-AA-SF	W-EFFLUENT	1000	2		200uL		200uL	
P275003	3/4	8	MSD-1641903	W-NP-AA-SF	W-EFFLUENT	1000	2		200uL		200uL	

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Printed: 10/17/2014 **SOP-GC-002_1485** Page 2 of 2

Prep Batch ID	Bottle #	Lab pH	Sample ID	Test ID	Matrix Type	Sample	Final Extract	Sample Cleanup	Spike 1	Spike 2	Spike 3	Spike 4
10X dil. surr spk												
P275003	2/3	6	MSD-1642415	W-NP-AA-SF	W-SURF-FRH	1000	2		200uL		200uL	
10X dil. surr spk												
P275003	1/4	8	1641810	W-NP-AA-SF	W-EFFLUENT	1040	2		200 uL			
10X dil surr spk												
P275003	1/2	5	1641811	W-NP-AA-SF	W-FIELD-BK	1040	2		200 uL			
10X dil surr spk												
P275003	1/4	8	1641903	W-NP-AA-SF	W-EFFLUENT	1000	2		200 uL			
10X dil surr spk												
P275003	1/2	5	1641904	W-NP-AA-SF	DI-WATER	1041	2		200 uL			
10X dil surr spk												
P275003	1/4	6	1642092	W-NP-AA-SF	W-EFFLUENT	997	2		200 uL			
10X dil surr spk												
P275003	1/2	5	1642093	W-NP-AA-SF	W-FIELD-BK	985	2		200 uL			
10X dil surr spk												
P275003	1/3	6	1642415	W-PC-AA-SF	W-SURF-FRH	1000	2	GPC /1 g Florisil	200 uL			
10X dil surr spk												
P275003	1/2	6	1642416	W-PC-AA-SF	W-SURF-FRH	1039	2	GPC /1 g Florisil	200 uL			
10X dil surr spk												
P275003	1/2	5	1642417	W-PC-AA-SF	W-SURF-FRH	1057	2	GPC /1 g Florisil	200 uL			
10X dil surr spk												
P275003	1/2	7	1642547	W-PC-AA-SF	W-SURF-FRH	1021	2	GPC /1 g Florisil	200 uL			
10X dil surr spk												
P275003	1/2	7	1642548	W-PC-AA-SF	W-SURF-FRH	1039	2	GPC /1 g Florisil	200 uL			
10X dil surr spk												
P275003	1/2	8	1643349	W-PC-AA-SF	W-SURF-FRH	1064	2	GPC /1 g Florisil	200 uL			
10X dil surr spk												

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