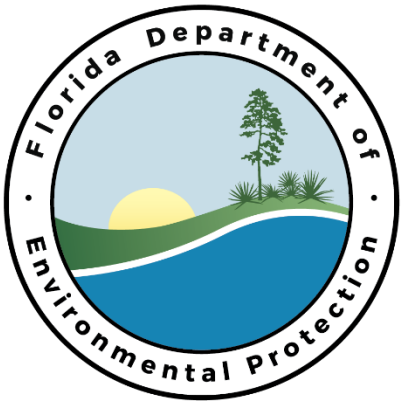
Standard Operating Procedure for:

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



DEPARTMENT OF ENVIRONMENTAL PROTECTION
CHEMISTRY SECTION
TALLAHASSEE FLORIDA

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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 3510C, 3540C, 3550C, 3580A, 3620C, 3640A, 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: W-PSNP-TQ, W-TR-TQ, S-PSNP-TQ, S-PSCL-TQ, S-PCB-TQ, S-CT-CI, WS-CT-CI., WS-PSCL-TQ, WS-PCB-TQ and WS-PSNP-TQ. Liquid - Liquid extraction is also applicable to TCLP and SPLP extracts analyzed using tests TCLP-CL-SQ, TCLP-CT-CI, SPLP-CL-SQ, SPLP-PCBTQ, SPLP-CT-CI and SPLP-NP-TQ.

2. SUMMARY OF THE METHOD

2.1 For water samples (based on EPA 3510C) 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 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 solvent exchanged to iso-octane and concentrated. Unless cleanup is required, the sample extract is then ready for analyses by GC/MS/MS. Part of the sample extract is further cleaned up by the appropriate clean up procedures described in this SOP for GC/MS 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 analysis.

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

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

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

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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 Horn Sonicators (Fisher Scientific/Qsonica) FB705/CL334, maximum output 705W, ¾ inch horn tip.

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

3.15 Metal spatulas

3.16 Beakers, 400mL and Mayo jars, 16oz.

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 Phenogel (300 x 21.2 mm) with (50 x 21.2 mm) guard 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

3.26 Aluminum weighing pans, for dry weight determination

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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 GC Resolve grade

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

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

4.3.4 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.

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

4.7 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):

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

4.8 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.

4.9 Sulfuric acid, concentrated

4.10 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.

4.11 10N Sodium hydroxide (NaOH) solution - Dissolve 40g of NaOH (ACS) in DI water and dilute to 100mL

4.12 **Spiking solutions** (see standards preparation procedure for more details in DEP SOPs GC-033-1 (Ref. 15.20, Gc-034-1 (Ref 15.21) and SOP GC-035-1 (Ref 15.22).

4.12.1 Spiking solutions for the GC/MS/MS tests

4.12.1.1 NP-TQ surrogate spike solution: 100 ug/L of Simazine-d10 and Terbufos-d10 and 500 ug/L of caffeine-d9. Use 50 uL for 0.25 mL final volume (water) and 400 uL for 2.0 mL final volume (soil).

4.12.1.2 NP-TQ spike solution 20-320 ug/L – use 50 uL for 0.25 mL final volume (water) and 400 uL for 2.0 mL final volume (soil)

4.12.1.3 PSCL surrogate spike solution 200 ug/L of TMX, PCNB and DCB – use 200 uL for 2.0 mL final volume.

4.12.1.4 PSCL spike solution 200-1000 ug/L use 160 uL/ for 2 mL final volume.

4.12.1.5 PCB 1060 spike 2000 ug/L – use 160 uL for 2 mL final volume.

4.12.1.6 T-CL-TQ spike 100 ug/L - solution use 200 uL for 2 mL final volume.

4.12.1.7 TR-TQ tracers surrogate spike solution 250 ug/L- use 50 uL for 0.25 mL final volume.

4.12.1.8 TR-TQ spike solution 250 ug/L – use 100 uL for 0.25 mL final volume.

4.12.2 Spiking solutions for the GC/MS NCI tests (soil and tissue)

4.12.2.1 Chlordane/Toxaphene spike at 1000/6000 ug/L – use 320 uL for 2.0 mL final volume (LFBs and matrix spikes) and 80 uL for PQLs.

4.12.2.2 Toxaphene spike at 8000 ug/L, tissues only. – use 100 uL for 2.0 mL final volume (LFBs and matrix spikes), and 25 ul for Tissue PQLs

4.12.2.3 PSCL surrogate spike solution 200 ug/L of TMX, PCNB 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 6 °C or less. All water samples (except for environmental tracers, test ID:

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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 6 °C or less. 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 6 °C or less 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 6 °C or less and must be extracted within 14 days of collection.

5.5 All sample extracts have to be stored at 6 °C or less.

6. WATER SAMPLE PREPARATION PROCEDURE

(Based on EPA 3510C)

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 NITROGEN/PHOSPHORUS PESTICIDES WITH ULTRA LOW DETECTION LIMITS

(for analysis by GC/MS/MS) LIMS Test: W-PSNP- TQ

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 DI water for blanks and laboratory-fortified blanks. The DI water for blanks and LFBs is pre-cleaned by extraction with 60 mL of methylene chloride. When weighing, assume sample has a density of 1.0 g/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). For matrix spike and matrix spike duplicate, weigh the sample bottle before and after pouring the entire sample into the 2-liter separatory funnel. For each extraction batch perform a single method blank, two LFBs and duplicate matrix spike (if enough sample volume is available).

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. The pH must be in the range of 5-9. Adjust the pH if necessary using 1:1 (v/v) sulfuric acid or 10.N sodium hydroxide. 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 300 ul of 10% of sodium bicarbonate. The pH adjustment prevents random and sometimes significant loses of certain analytes, primarily Hexazinon

6.1.3 Add appropriate surrogate spikes (as per 4.13) to all samples and method blank. Add the required analyte spikes add 50 ul of surrogate spike to all samples, and 50 u; of required analyte spike to LFBs and Matrix Spikes.

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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
- 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.

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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 (67 °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 (30 °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.

6.1.24 Before transferring the sample extract (after K&D concentration) to a centrifuge tube, calibrate the tube to 0.20mL with fresh Iso-octane and mark the meniscus. 0.05 mL fresh acetone and mark the meniscus to 0.20mL.measure the 0.20mL of liquid with a pipette syringe at the marked meniscus is the true volume of 0.20mL on this tube, rather than using the original graduations on the tube. Enter the final volume as 0.25 mL in the extraction report to account for the internal standard addition.

6.2 **DIELDRIN WITH ULTRA LOW DETECTION LIMITS**
((for analysis by GC/MS/MS) LIMS test: W-DIELDRIN see SOP GC-036-1

6.3 **ENVIRONMENTAL TRACERS**
By GC/MS/MS, LIMS test; W-TR-TQ

Extract as in section 6.3, with the following differences:

6.3.1 When spiking, use spike mixtures (surrogates and analytes) specially prepared for GC/MS/MS analysis. Add 50 uL of tracer 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.

6.3.2 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)

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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). Nitrogen Phosphorus pesticides by GC/MS/MS (test ID: S-PSNP-TQ)
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 LOGBOOK.

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 into GC Laboratory.

7.3 Weigh approximately 30g ± 0.1g of wet sediment sample into a 16oz Mayo jar. Record the weight.

7.4 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

7.5 Add surrogates and spiking solutions as per chapter 4.13 earlier in this SOP.. If test S-CT-CI is requested and additional PQL has to be prepared. If S-PCB-TQ test is also requested Chlordane and Toxaphene may be spiked simultaneously with PCB-1060.

7. 6 Immediately add 100mL of 1:1 methylene chloride: acetone to the sample and sonicate for three minutes using horn sonicators. The sonicators are programmed to run at 100% amplitude, with duty cycle set at 50% (1s on, 1s off).

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 7.6 – 7.8 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 jar, 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.

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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 (70 to 75 °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 CONCENTRATOR TUBE IS LABELED WITH THE CORRECT SAMPLE NUMBER!!

7.18 Setup N-Evap® by turning on nitrogen-gas, heater (30 °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 thrice with 1mL of methylene chloride and N-blow down to 2mL; concentrator tube should be calibrated to 1mL/2mL (2mL final volume of extract).

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 using pure methylene chloride as eluting solvent; 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 using 1 ml of the original extract. Blow down to 0.9 mL to account for the internal standard addition and submit for analysis. If S-PSNP-TQ test is requested process the other 1 mL of extract through GPC clean-up using 3% methanol in Methylene Chloride as eluting solvent. Inject 0.8 mL on the GPC column. After the clean-up is completed concentrate the sample to 0.6 mL to account for the internal standard addition. No Florisil clean-up is performed for NP pesticides.

7.23 **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: WS-PSNP-TQ, 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).

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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 spiking follow the procedure specified for soil samples in 7.5 and 4.13 but increase each spiking volume by the factor of 5. This is due to the final volume being 10 mL rather than 2 mL like for soils.

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 FILTER SAMPLES (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
3510C (TQ) – W-PSNP-TQ
3510C (TR) – W-TR-TQ
3550C (TQ) – S-PSCL-TQ, S-PCB-TQ, S-CT-CI, S-PSNP-TQ
3580A (TQ) – WS-PSCL-TQ, WS-PCB-TQ, WS-CT-CI, WS-PSNP-TQ
3535A (GC) –W-PCB-TQ-R, W-PCL-SQ-R, W-CT-CI-R, W-DIELDRIN

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Many fields in both the header and the grid are prefilled and seldom change. They need, however, to be always checked for correctness.

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**:
- Equipment ID: GPC SN: Sonicator SN; K&D SN, N-EVAP SN.
- **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: GPC1/200410*
- **Prep comments:** *enter other chemicals/supplies here with lot numbers; example Ottawa sand., Florisil lot# , pH paper lot #, Filter Paper Cat#, GPC Puradisc 25PP Filter lot#,Glasswool lot#, 10% Sodium Bicarbonate 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*

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- **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.*

- **% 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; 0.25 mL for water samples, 2 mL for sediment and 10mLfor waste sample extraction.

- Sonicator ID: list Sonicator used (A1, A2, B1, B2, C1, C2) for each sample
  - **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 GPC CLEANUP PROCEDURE (BASED ON EPA 3640A)

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

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

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

11.3.2 For each sample place a properly labeled one-gram Florisil cartridge into the vacuum manifold. Place one additional Florisil cartridge for Florisil blank.

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

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

11.3.5 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.

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

11.3.7 Add 3x3 mL of hexane-acetone (90:10) solution followed by 2x1 ml of hexane to the original sample tube and transfer each time to cartridge as it elutes.

11.3.8 N-blow down to 0.7mL after solvent exchange with 1mL in iso-octane. The final volume should result in a concentration equal to that of the extract initially taken for cleanup, taking account the internal standard addition.

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

11.4 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.

11.4.1 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.

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

11.4.3 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.

11.4.4 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.

11.4.5 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-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

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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.

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

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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 Florisil 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.

5/2/2016:

1) Addition of sonicator horns per TNI audit requirement.
2) Addition of Florisil blank.

4/21/2017:

1) Addition of test S-PSNP-TQ/WS-PSNP-TQ.
2) Corrected spike volumes and concentration.

2/01/2018:

According to internal audit recommendations added SN of K&D as well as N-EVAPs to the prep worksheets. The worksheets will also show which device was actually used under “bath temp” entry. Additionally, for soil extractions, the worksheets will indicate which horn was used ie: A1 means sonicator A left horn, A2 – sonicator A right horn.

Corrected spike concentrations; removed test W-PSCL-TQ.
Updated the example of electronic worksheet.

1/25/2019:

Updated spiking procedures, removed retired tests. Sample extract storage temperature changed to 6C or less.

1/30/2020:

Removed preparation of samples for W-PC-AA-SF and W-NP-AA-SF (ECD and NPD)

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1/28/2021:

Removed Tetrabutylammonium sulfite cleanup procedure. It is no longer performed and not applicable to current sample preparation protocols.

3/12/2021:

Corrected section 7.9 as per CERP audit recommendation of 2/26/2021.

EXAMPLE OF ELECTRONIC PREPARATION LOG

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Worksheet ID: 3550C (TQ)_83
Prep ID: 3550C (TQ)
Prepared By: SIDDELL_F
Prep Start Date/Time: 1/18/2018 09:00
Spike Witness: BURGOS_J
Prep Completion Date/Time: 1/25/2018 16:10
Date/Time Reviewed: 1/25/2018 16:16
Reviewed By: SIDDELL_F
Balance ID: OP-AV4101C-092
Spike 1: 86614 SURR SPK
Spike 2: 86179 PSCL_SPK
Spike 3: 86123 PCB1060 QCS SPK
Spike 4: 87384 TOXA SPK
Spike 5: 87385 CHLD SPK
Spike 6:
Spike 7:
Equipment ID(s): Sonicator A: 90030L-04-16/2016021085, Sonicator B: 90028L-04-16/2016021131, Sonicator C: 90026L-04-16/2016021111, KD A SN: 61269, KD B: SN: 61257, N-EVAP A SN: 19352, N-EVAP B SN: 21560
GPC ID: GPC-1 SN:L20344655343; GPC-2 SN: C21324950147; GPC-3 C21325050149
Cleanup Procedures: GPC seq: 011818GPC2
Water Bath Temperature (C): KD B 80C, N-EVAP A 30C
Final Extract Solvent: Isooctane_163098
Solvent 1: Methylene chloride_177204
Solvent 2: Acetone_170941
Solvent 3: n-Hexane_158159
Solvent 4: Isooctane_163098
Reagent 1:
Reagent 2:
Prep Comments: Florisil lot# 1002464-3442026, Sodium Sulfate lot# 161563, Ottawa Sand lot# 164859
Internal Standard: 80ul IS added SN # 86862 01/26/18 VTN

Prep Batch	Sample ID	Test ID	Matrix Type	% Solids	Sample Wet	Sample Dry	Final	Sonicator ID	Sample	Spike 1	Spike 2	Spike 3	Spike 4	Spike 5
P338766	MBLK	S-CT-CI	OTTAWA	100	30.0	30	2	A1	GPC/1 g	200 uL				
P338766	MBLK	S-PCB-TQ	OTTAWA	100	30.0	30	2	A1	GPC/1 g	200 uL				
P338766	MBLK	S-PSCL-TQ	OTTAWA	100	30.0	30	2	A1	GPC/1 g	200 uL				
P338766	GPC BLK#1								GPC/1g					
P338766	GPC BLK#2								GPC/1g					
P338766	FLO BLK								1g Florisil					
P338766	PQL	S-CT-CI	OTTAWA	100	30.1	30.1	2	A2	GPC/1 g	200 uL			50 uL	20 uL
P338766	MDLV-PCB	S-PCB-TQ	OTTAWA	100	30.1	30.1	2	A2	GPC/1 g	200 uL		40 uL		
P338766	MDLV-CL1 5x diluted Spike	S-PSCL-TQ	OTTAWA	100	30.1	30.1	2	B1	GPC/1 g	200 uL	45 uL			
P338766	MDLV-CL2 5x diluted Spike	S-PSCL-TQ	OTTAWA	100	30.2	30.2	2	B2	GPC/1 g	200 uL	160 uL			
P338766	MDLV-CL3 5x diluted Spike	S-PSCL-TQ	OTTAWA	100	30.3	30.3	2	A2	GPC/1 g	200 uL	400 uL			
P338766	LFB_1	S-PSCL-TQ	OTTAWA	100	30.1	30.1	2	B1	GPC/1 g	200 uL	160			

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Prep Batch	Sample ID	Test ID	Matrix Type	% Solids	Sample Wet	Sample Dry	Final	Sonicator ID	Sample	Spike 1	Spike 2	Spike 3	Spike 4	Spike 5
Insufficient sample to perform matrix duplicate spikes.														
P338766	LFB_2	S-PSCL-TQ	OTTAWA	100	30.1	30.1	2	B2	GPC/1 g	200 uL	160			
P338766	LFB_3	S-CT-CI	OTTAWA	100	30.0	30	2	C1	GPC/1 g	200 uL			200 uL	80 uL
Insufficient sample to perform matrix duplicate spikes.														
P338766	LFB_3	S-PCB-TQ	OTTAWA	100	30.0	30	2	C1	GPC/1 g	200 uL		160 uL		
P338766	LFB_4	S-CT-CI	OTTAWA	100	30.0	30	2	C2	GPC/1 g	200 uL			200 uL	80 uL
P338766	LFB_4	S-PCB-TQ	OTTAWA	100	30.0	30	2	C2	GPC/1 g	200 uL		160 uL		
P338766	M1_1959102	S-PSCL-TQ	S-OTHER	95.8	10.0	9.58	2	C1	GPC/1 g	200 uL	160			
P338766	M3_1959102	S-CT-CI	S-OTHER	95.8	10.0	9.58	2	C2	GPC/1 g	200 uL			200 uL	80 uL
P338766	M3_1959102	S-PCB-TQ	S-OTHER	95.8	10.0	9.58	2	C2	GPC/1 g	200 uL		160 uL		
P338766	1959102	S-CT-CI	S-OTHER	95.8	10.0	9.58	2	A1	GPC/1 g	200 uL				
P338766	1959102	S-PCB-TQ	S-OTHER	95.8	10.0	9.58	2	A1	GPC/1 g	200 uL				
P338766	1959102	S-PSCL-TQ	S-OTHER	95.8	10.0	9.58	2	A1	GPC/1 g	200 uL				
P338766	1959103	S-CT-CI	S-OTHER	48.9	10.0	4.89	2	A1	GPC/1 g	200 uL				
P338766	1959103	S-PCB-TQ	S-OTHER	48.9	10.0	4.89	2	A1	GPC/1 g	200 uL				
P338766	1959103	S-PSCL-TQ	S-OTHER	48.9	10.0	4.89	2	A1	GPC/1 g	200 uL				
P338766	1959104	S-CT-CI	S-OTHER	91.0	10.0	9.1	2	A2	GPC/1 g	200 uL				
P338766	1959104	S-PCB-TQ	S-OTHER	91.0	10.0	9.1	2	A2	GPC/1 g	200 uL				
P338766	1959104	S-PSCL-TQ	S-OTHER	91.0	10.0	9.1	2	A2	GPC/1 g	200 uL				

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