Standard Operating Procedure for Florisil Cleanup of Organic Extracts for Polychlorinated Biphenyls (PCBs) Analysis (TSCA Program)

United States Environmental Protection Agency
Region 5 Chicago Regional Laboratory
536 S Clark Street (ML-10C)
Chicago IL, 60605

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1. Scope and Application
   1.1. Florisil is a commercially available magnesium silicate adsorbent that is suitable for use in the cleanup of organic extracts from environmental samples prior to gas chromatographic analysis. This procedure describes two options for accomplishing the cleanup of extracts: (a) the use of a glass column packed with Florisil; and (b) the use of solid-phase extraction (SPE) cartridges containing Florisil both options from EPA reference method 3620C, with no deviations. This standard operating procedure applies to the separation of polychlorinated biphenyls from interfering compounds and is specific to the TSCA program.
   1.2. In option (a), cleanup is accomplished using a glass chromatographic column packed with activated Florisil and topped with anhydrous sodium sulfate for drying the extract. The PCBs are eluted through the column using hexane. Prior to sample cleanup, the cut-off point is determined by eluting an Aroclor mix that is representative of both the low and high percent chlorine Aroclors (Aroclor 1016/1260 standard mix may be used for this purpose) and demonstrating that the PCBs have been quantitatively recovered.
   1.3. In option (b), cleanup is accomplished using SPE cartridges containing Florisil, usually in 1 g quantities. These cartridges are also available in 0.5 or 2 g quantities of Florisil packing. Each cartridge is washed with solvent immediately prior to use. The analytes are eluted through the cartridge using 9:1 hexane/acetone (v:v). Use of a vacuum manifold is recommended to obtain reproducible results. Prior to sample cleanup, the elution solvent volume is verified by eluting an Aroclor mix (Aroclor 1016/1260) and demonstrating that the PCBs have been quantitatively recovered.

2. Summary of Method
   2.1. The glass chromatographic column is packed with the required amount of Florisil, topped with sodium sulfate and then loaded with the sample extract. The PCBs are eluted with hexane, leaving the interferences on the column. The eluate is made up to a final volume of 10 ml using hexane.
   2.2. The Florisil cartridges are washed with solvent immediately prior to use. The PCBs are eluted through the cartridge using 9:1 hexane/acetone (v:v). Use of a vacuum manifold is recommended to obtain reproducible results. The eluate is then solvent exchanged to hexane and made up to the appropriate final volume prior to other required cleanup procedures.

3. Abbreviations and Definitions
   PCB – Polychlorinated Biphenyl
   SPE – Solid phase extraction

TSCA - Toxic Substances Control Act
CRL - Chicago Regional Laboratory of Region 5, U.S. EPA
SOP - Standard Operating Procedure
SDS – Safety Data Sheet
LIMS - Laboratory Information Management System
MB - Method Blank
LCS/LCS dup - Laboratory Control Sample/Duplicate
MS/MSD- Matrix Spike/Duplicate
QC- Quality Control

4. Health, Safety and Waste Handling
   4.1. Users of this method should operate a formal safety program. Perform this method according to CRL Chemical Hygiene Plan located on the CRL share drive (G:\drive).
   4.2. The toxicity or carcinogenicity of each reagent used in this procedure has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. Exposure to any of these chemicals should be reduced to the lowest possible level by whatever means available. Review Safety Data Sheets (SDS's) for specific physical and health hazards including appropriate personal protective gear (safety glasses, gloves, lab coats) to be used. SDS's may be accessed at www.sigmaaldrich.com.
   4.3. This procedure requires the use of solvents and PCB standards; it must be performed in a hood. **"WARNING"** Samples may contain high concentrations of volatile toxic compounds. Sample containers should be opened in a hood and handled with gloves to prevent exposure
   4.4. After the clean-up and concentration steps are complete, dispose of the solvent waste in a waste container with a green label. Dispose of the used florisil and florisil cartridges in a yellow hazardous waste bag.
   4.5. Waste Handling
      4.5.1. Liquid organic wastes such as waste solvents, extracts and expired standards that are less than 50 ppm PCBs are disposed of in green labeled 5-gallon plastic containers located in a lab fume hood designated as temporary holding area. Organic wastes that are more than 50 ppm PCBs are disposed off in purple labeled 5-gallon plastic containers. Solid, hazardous wastes are placed in properly labeled yellow bags.
      4.5.2. Refer to the *CRL Safety, Health & Environmental Compliance Manual* for more information on waste reduction. Additional information on waste reduction can be found in the CRL Environmental Compliance Plan and additional

information on hazardous waste spills can be found in the CRL Hazardous Materials/Hazardous Waste Contingency Plan.

4.5.3. Report all major spills.

5. Cautions and Interferences

5.1. Method interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts or elevated baselines in gas chromatograms. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

5.1.1. Glassware must be scrupulously clean. See SOP GEN008 for the washing of laboratory glassware/hardware at the CRL. Rinse all glassware once with acetone and twice with hexane before use (10-20 ml each).

5.1.2. The use of high purity reagents and solvents will greatly minimize interference problems.

5.2. Interferences by phthalate esters can pose a major problem when using an electron capture detector. These compounds generally appear in the chromatogram as large late eluting peaks. Avoiding the use of common flexible plastics that contain varying amounts of phthalates will minimize this type of interference problems.

5.3. Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of interferences will vary. Cleanup procedures eliminate certain types of interferences.

6. Equipment and Supplies

The following brands names, suppliers, and part numbers are stated in this SOP for illustrative purposes. No endorsement is implied.

6.1. Glassware– cleaning instruction refer to section 5.1.1

6.1.1. Chromatography columns - plain, glass column, without frits, with an integral reservoir, 200 ml capacity; column ID is 11 mm & length is 250 mm; size 2 PTFE stopcock plug is used to control the flow rate. A plug of glass wool is used to support the column packing material.

6.1.2. Erlenmeyer flasks - glass, 250, 300 ml capacity

6.1.3. Test tubes- glass, 10 ml with Teflon-lined screw caps

6.1.4. Capillary pipettes - disposable, Pasteur type, 1ml

6.1.5. Cylinder, graduated – class A, 500 ml, 1000ml

6.2. Glass wool

6.3. Concentration/evaporation equipment:

6.3.1. TurboVAP 500 Closed-cell concentrators (Zymark, Inc.); or equivalent, consisting of a water bath, motor-fan blade assembly, and condenser, and 500 ml concentrator tubes

6.3.2. TurboVAP LV (Zymark, Inc.); or equivalent - nitrogen evaporator for concentrating smaller volumes of extracts or for exchanging solvents, 50-tube capacity

6.3.3. N-EVAP (Organomation Associates) - nitrogen evaporator for concentrating smaller volumes of extracts or for exchanging solvents

6.4. Vacuum Manifold

6.5. Water chiller - supplies cold water to the TurboVAP 500 concentrator

6.6. Certified thermometer – certified annually

Note: Reference GEN026 for equipment and supplies ordering instructions

7. Reagents and Standards

All of the vendors and part numbers for the reagents and standards listed below are what the CRL currently uses as of the date of this SOP and are listed specifically to facilitate the ordering process. The mention of trade names or commercial products in this SOP is for illustrative purposes only and does not constitute an EPA endorsement or exclusive recommendation for use. Analysts are encouraged to seek out equivalent alternatives to the items listed below. These specific vendors and part numbers are provided examples and other materials of equivalent quality may be substituted without altering this SOP. Refer to CRL SOP GEN026 for instructions and analyst responsibilities when purchasing reagents and standards.

The described preparations of the spiking standards, calibration standards, and QC samples below are not absolute requirements. Rather, the prepared concentrations, spike volumes, and overall calibration range may be tailored to the needs of the project with consideration given to any action limits or regulatory thresholds the samples are being analyzed against and the nature of the samples and any interference present therein.

NOTE: Solvents used for sample preparation must be tracked by a LIMS ID.

7.1. Hexane – pesticide quality {Burdick & Jackson (B&J) or equivalent}

7.2. Acetone – pesticide quality (B&J or equivalent}

7.3.9:1 (v:v) Hexane/Acetone - using a graduated cylinder, mix 900 ml of hexane with 100 ml of acetone.

7.4. Florisil – Supelco or equivalent; PR grade (60/100 mesh). This grade has been activated at 675 °C and is most useful for PCB/pesticide analysis. Before use, activate each batch by heating the Florisil in a glass container, loosely covered with aluminum foil in an oven at 130 °C for at least 16 hours. This is done to drive off any moisture that has been absorbed during storage.

7.5. Florisil cartridges - 6-ml size polypropylene cartridges; 1 g of Florisil is held between two polyethylene frits. (Restek or equivalent)

7.6. Florisil Cleanup Control Standard - an Aroclor mix that is representative of both the low and high percent chlorine Aroclors; Aroclor 1016/1260 standard mix may be used for this purpose - the intermediate standard contains 10 µg/ml of each Aroclor. (See CRL SOP GC010 for preparation of the working A1016/1260 standard.)

Note: Florisil standard expires six months after original preparation date noted on bench sheet and according to LIMS ID assigned.

7.6.1. Florisil Column - 1.00 ml of the above standard (10 µg/ml) is eluted through the florisil column. After it has passed through the column, the eluate is concentrated and brought to a final volume of 10 ml. This results in a final concentration for each Aroclor of 1 µg/ml which is at the midpoint of the 5-point calibration.

7.6.2. Florisil Cartridge - 1.00 ml of the above standard (10 µg/ml) is made up to 10 ml with hexane for a concentration of 1 µg/ml for each Aroclor. In the same manner as the samples, 2.5 ml of the 1 µg/ml mixed aroclor standard is eluted through the florisil cartridge. The eluate is then solvent exchanged back to hexane and subsequently brought to a final volume of 2.5 ml resulting in a final concentration of 1 µg/ml for each Aroclor which is at the midpoint of the 5-point calibration.

7.6.3. Florisil Blank as follows: Take 1 ml of a surrogate spiking solution (0.2 µg/ml) and dilute to 10 ml with hexane. Process the florisil blank in the same manner as the Florisil control standard (9.3.1.4). Refer to section 10.2 for QC frequency.

Note: Florisil Blank standard expires six months after original preparation date noted on bench sheet and according to LIMS ID assigned.

7.7. Sodium sulfate, granular, anhydrous, reagent grade. Purify by heating at 105 °C for 12 hours or more, cool in a desiccator, and store in a glass bottle.

(NOTE: Storing open containers of sodium sulfate in the laboratory may result in contamination. Cover the containers with aluminum foil.)

8. Sample Handling and Preservation
   8.1. The sample extracts should be kept in tightly-capped containers and stored in the sample extract refrigerator at 4 ± 2°C until further processing. Caps should be teflon-lined. Small pieces of aluminum foil may also be used as a liner for the caps to prevent contact with the extract.
   8.2. Cleanup procedures must be performed on the sample extracts as soon as possible after extraction, within the 40 day analysis holding time.

9. Sample Preparation and Analysis
   9.1. Create a florisil cleanup bench sheet through the Laboratory Information Management System (LIMS); be sure to choose the florisil bench sheet format for TSCA samples. On this bench sheet, record the florisil or florisil cartridge supplier and lot number, the amount of extract used for cleanup, standard and blank information, and any other pertinent information. Complete the bench sheet after cleanup is finished.

   9.2. Florisil Column Technique:

   NOTE: Granular Florisil comes in four grades; the differences are a function of the activation temperature. Florisil PR, the most commonly used grade for pesticide residue analysis, has been activated at 675 °C. To drive off any moisture adsorbed during storage and handling, prior to use heat the Florisil in a glass beaker loosely covered with aluminum foil in an oven at 130 °C for at least 12 - 16 hours. Cool before use in a desiccator. To ensure a uniform adsorptive capacity, it is important to use the same batch and source of Florisil to process all the samples and QC samples that are being analyzed as a batch.

   9.2.1. Determination of elution profile: chromatography of Florisil cleanup control standard
      9.2.1.1. Prepare the Florisil column by placing a plug of glass wool at the bottom of the chromatography column that has been fitted with a PTFE stopcock plug. Rinse the column and glass wool with two 25 ml portions

each of acetone and hexane before packing with Florisil. Discard these washings into the appropriate waste reservoirs (see section 4.0)

9.2.1.2. Fill the column with hexane. Add approximately 10 ± 0.5 g of Florisil into the column.

NOTE: This can be done in two ways: (1) by adding the dry Florisil into the column filled with hexane; or (2) by preparing Florisil slurry in hexane and adding, in small increments, into the column containing hexane. The second technique is preferred because it ensures that all the Florisil granules are thoroughly wet. During the addition, open the stopcock just enough to allow the hexane to drip slowly into the Erlenmeyer flask. Do not let the column or any part of the column to dry out.

9.2.1.3. Allow the Florisil to settle during the addition by tapping the column gently with a piece of rubber tubing, taking care not to break the column.

9.2.1.4. When the Florisil has settled and the column sufficiently packed, add anhydrous sodium sulfate to the top of the Florisil to form a layer at least 1 cm deep, but not extending beyond the top of the column and into the reservoir. Allow the hexane to drip until just prior to exposure of the sodium sulfate layer then close the stopcock. Discard the hexane eluate. Rinse up to 10 (more, if necessary) 10-ml graduated cylinders with acetone, and then with hexane, to be used for collecting 10 ml fractions of the eluate.

9.2.1.5. Quantitatively transfer 1 ml of the Florisil cleanup control standard on top of the sodium sulfate in the column. Open the stopcock and allow the hexane to drip just prior to exposure of the sodium sulfate layer. Complete the transfer with two 1-ml rinses with hexane, each time allowing the hexane to drip just until the sodium sulfate layer is nearly exposed (but not dry). This will ensure that the standard is loaded and concentrated on top of the Florisil column before adding the elution solvent.

9.2.1.6. Fill the column reservoir with hexane (usually 200 - 250 ml), open the stopcock and elute at a drip rate of about 5 ml/min into the 10 ml graduated cylinders, collecting 10 ml fractions at a time. Do not allow the column to dry.

9.2.1.7. Concentrate the fractions as described in Sections 9.2.4 or 9.2.5 and analyze by gas chromatography (Refer to CRL SOP GC003) to verify

the cut-off point, i.e. the point where all the Aroclors have been eluted and the interferences start to appear. Note the total volume of hexane that was required to recover all the PCBs. Use this volume to cleanup the sample extracts and QC samples (method blank, LCS/LCSD, MS/MSD).

9.2.2. Florisil column cleanup of the sample extracts

9.2.2.1. Prepare the columns according to 9.2.1.1 - 9.2.1.4. Rinse 250-ml Erlenmeyer flasks with acetone and hexane. Properly label each flask and place beneath the columns.

9.2.2.2. Reduce the volume of the sample extract, from 10 ml volume after extraction, to 1 ml, and quantitatively transfer onto the top of the column. Open the stopcock and allow the hexane to drip just prior to exposure of the sodium sulfate layer. Complete the transfer with two 1-ml rinses with hexane, each time allowing the hexane to drip just until the sodium sulfate layer is nearly exposed (but not dry). This will ensure that the sample is loaded and concentrated on top of the Florisil column before adding the elution solvent to the reservoir.

(**NOTE**: If the sample is known to contain high levels of PCBs, subject only a portion of the extract to the Florisil cleanup. Record the amount of sample that was taken for cleanup and make sure that the dilutions are accounted for in the final calculations. Since the amounts of surrogates and spiking solutions added prior to extraction have been adjusted based on applicable action levels, these must be considered in the determination of final volumes. The analyst must keep track of all the initial preparation parameters and make sure that all of these parameters are accurately reflected in the final calculations.)

9.2.2.3. Add the amount of hexane determined in Section 9.2.1 to the column reservoir and elute at a drip rate of about 5 ml/min into the collection flask. Do not allow the column to dry. Close the stopcock when the pre-determined volume has been collected.

9.2.2.4. Concentrate the eluate using one or more of the procedures described in Sections 9.2.3 – 9.2.5, to a final volume of 10 ml (or to the adjusted amount for highly concentrated samples). Proceed with other required cleanup procedures after concentration.

9.2.3. Concentration using the TURBO-VAP 500 closed-cell concentrator

Description of the apparatus:
The TURBO-VAP 500 closed-cell concentrator is a microprocessor-controlled concentrator that provides automated sample concentration using a helical gas flow.

The closed cell consists of a motor assembly, a cell cap, a condenser and a concentrator tube. The cell cap, which is attached to the motor assembly, encloses the fan blade. The concentrator unit consists primarily of a front panel for controlling the setup and operation, condenser holders and a water bath for warming the sample during concentration. It is equipped with quick-disconnect inlet/outlet for supplying coolant to the condensers and alarm volume control for adjusting the loudness of the buzzer that signals the end-point.

NOTE: For a more detailed description of the apparatus and its operation, refer to TURBO-VAP 500 Closed Cell Concentrator Operator's Manual. A user's guide to daily operation of the instrument can be found in a plastic pocket at the side of the instrument.

9.2.3.1. Fill the water bath such that when the sample tubes are placed in the water bath, the water does not overflow and the level of water in the bath is as high as the initial solvent in the sample tube.

9.2.3.2. Turn the unit **ON** and observe the "Power Up" diagnostics that occurs to make sure the instrument is functioning properly.

9.2.3.3. The following are the recommended operating conditions:

Water bath temperature – ranging from 45- 55°C
NOTE: Check the water bath temperature with a certified thermometer. In the water bath temperatures logbook, record the evaporator ID, the setpoint temperature, the digital temperature readout on the device, and th e certified thermometer ID and temperature.

Fan speed – 6000 rpm (Setting C)
Endpoint selection – Sensor endpoint
NOTE: Ideally, by selecting **SENSOR ENDPOINT**, concentration will proceed until the sensor detects a level of 0.5 or 1 ml depending upon the type of the concentrator tube. The fan speed may be changed if necessary *(This feature does not always work as it should; consequently, causing the*

extract to dry out. It is sometimes necessary to manually start and stop the concentrators.)

9.2.3.4. Set the water bath temperature, fan speed and endpoint selection function specified above.

9.2.3.5. Make sure that the coolant supply and the solvent drain hose are properly connected. Check the solvent waste container. Pour the recovered solvent to a centralized waste collection container (see section 4.0)

9.2.3.6. Turn on the cold water supply (or water chiller, see instructions posted near equipment) connected to the condensers.

9.2.3.7. Rinse the TurboVAP 500 system prior to concentrating sample extracts in order to prevent cross contamination from previous extract, as follows: Pour 50 – 100 ml of acetone into the concentrator tube and place in the water bath. Grasp the motor assembly/cell cap and condenser as a unit, lift up from the mounting bracket and place on the concentrator tube. Start the concentration process by pressing the **START/STOP** button for the cell position used. Run for 5 – 10 minutes. Stop the system and discard any remaining acetone in the concentrator tube into the centralized waste collection container. Proceed to rinse tube with hexane (see section 6.1.1).

9.2.3.8. Transfer the sample extracts into the concentrator tubes (prior to concentrating the sample, rinse the system using a small amount of hexane to avoid cross- contamination with previous samples). Place the tubes in the water bath. Grasp the motor assembly/cell cap and condenser as a unit, lift up from the mounting bracket and place on the concentrator tube.

NOTE: Cover any unused position with plastic closures supplied with the unit to minimize bath water evaporation.

9.2.3.9. Start the concentration process by pressing the **START/STOP** button for each cell position used.

9.2.3.10. When a cell reaches its selected end point, its green **DONE** light will blink and the alarm sounds briefly at 30 second intervals. Silence the alarm by pressing the **START/STOP** button twice.

9.2.3.11. Disassemble the closed cell. **Remove the sample tube promptly from the water bath to avoid further drying up of the extract.**

9.2.3.12. Return the motor/cell cap assembly to its mounting bracket position and the condenser to its holding cup.

9.2.3.13. Transfer the concentrated extract into a test tube. Rinse the bottom of the concentrator tube twice with 2 ml of hexane and transfer the rinses to

the corresponding test tube. Adjust the volume to 10 ml using hexane. If necessary, proceed with further concentration or solvent exchange using the N-EVAP (9.2.4) or TURBO-VAP LV (9.2.5).

9.2.3.14. Clean the concentrator tubes thoroughly for subsequent use.

9.2.3.15. Turn the unit off when no longer in use and replace the plastic closures in each cell position.

9.2.4. Concentration/Solvent Exchange Using the N-EVAP (Organomation Associates)

9.2.4.1. For small adjustments to the final volume, concentrate the extract further using the N-EVAP (Organomation Associates, Inc.) This is done by placing the test tubes in the N-EVAP at approximately 40 °C and using a gentle stream of pure nitrogen applied to the sides of the tube just above the surface of the extract.

9.2.4.2. Rinse down the internal walls of the tube several times with hexane during evaporation. Ensure that the solvent level in the tube is below the level of the water bath to prevent water from condensing into the sample. Do not reduce the extract volume to below 1 ml nor allow it to go to dryness.

9.2.4.3. To solvent exchange, reduce the volume of the extract to between 1 to 2 ml then bring the volume of the extract back up to at least 5 ml using hexane. Repeat this entire process at least two more times to ensure that the solvent has been sufficiently exchanged to hexane.

9.2.4.4. Make the final volume of the extract up to the appropriate volume with hexane (depending on whether florisil column or cartridge was used and the amount of extract that was cleaned up). Proceed with other required cleanup procedures.

9.2.5. Concentration/Solvent Exchange Using the TURBO-VAP LV (Zymark, Inc.)

NOTE: Consult the Operating Manual for the proper operation of this unit. A user's guide to daily operation is located in a plastic pouch at the side of the instrument.

9.2.5.1. Turn ON unit and gas supply. Check the gas pressure (-5 to -10 psi), water bath level using a 10 ml test tube (ensure the water level is just

above sample extract level) and temperature (40 °C). Rinse each gas needle tip with 1-2 ml of hexane prior to starting.

9.2.5.2. Set the evaporation time to 20 minutes with the TIME push wheel. The buzzer will sound when this time expires.

9.2.5.3. Load the 10 ml test tubes containing the extracts into the racks in the evaporator. The test tube must be positioned such that the nozzle supplying the gas for evaporation extends properly into it. There are five manifold rows with 10 nozzles each. Any or all of the rows may be selected by using the tube station pushbuttons.

9.2.5.4. Select the rows that contain sample tubes by pressing the corresponding TUBE STATION pushbuttons. Press the START pushbutton. The unit will run for the duration of the set time. When the evaporation time expires, the buzzer will sound every 30 seconds and the gas will automatically shut off. Lift the cover, add more hexane (if exchanging solvent) and re-start the unit.

(NOTE: **Do not let the solvent go dry**. The optimal operating settings must be determined prior to using this unit by observing the time it takes to evaporate an amount of solvent, approximately equal to the amount of sample to be processed, down to the desired level. To ensure that all the samples will take about the same processing time, start with approximately equal amount of samples.)

9.2.5.5. To solvent exchange, reduce the volume of the extract to between 1 to 2 ml then bring the volume of the extract back up to at least 5 ml using hexane. Repeat this entire process at least two more times to ensure that the solvent has been sufficiently exchanged to hexane.

9.2.5.6. Make the final volume of the extract up to the appropriate volume with hexane. Proceed with other required cleanup procedures.

9.3. Florisil Cartridge Technique:

9.3.1. Determination of elution volume: Chromatography of Florisil control standard.

9.3.1.1. Attach the manifold to a vacuum pump and adjust the pressure to between -10 & -15 psi. Arrange the cartridges on the manifold in the closed-valve position, one cartridge per sample. Place a 10 ml test tube underneath each cartridge.

9.3.1.2. Wash the cartridges with at least 5 ml of hexane/acetone (9:1 v/v). Adjust the vacuum applied to each cartridge so that the flow rate is

approximately equal. Do not allow the solvent level to go below the top of the Florisil otherwise the cartridge will dry up.

9.3.1.3. Release the vacuum; discard the wash solvent and place clean, properly labeled 10 ml collection test tubes under each cartridge. Restore the vacuum but leave the valves in a closed position so as not to dry the cartridges.

9.3.1.4. Dilute 1 ml of the Florisil control standard (at 10 µg/ml) to 10 ml with hexane and add 2.5 ml of this to the top of the cartridge. Elute the PCBs through the cartridge first followed by 8 ml (two 4 ml portions) of 9:1 hexane:acetone (v:v) into the 10 ml collection test tube. Slowly open the valve allowing 8-10 sec between drips then increase the flow rate for the elution to about 2 ml/min (5-6 sec between drips). Once all the valves are open verify the vacuum pressure and elution rate. **(Warning: Do not allow the solvent level to go below the top of the florisil until after the last portion of eluting solvent has been added.)** Refer to section 10.3 for QC frequency.

9.3.1.5. Run a Florisil Blank as follows: Take 1 ml of a surrogate spiking solution (0.2 µg/ml) and dilute to 10 ml with hexane. Process the florisil blank in the same manner as the Florisil control standard (9.3.1.4). Refer to section 10.2 for QC frequency.

9.3.1.6. Solvent exchange the Florisil control standard eluate to hexane using the TurboVap LV (9.2.5.5) or N-Evap (9.2.4.3). When fully exchanged to hexane, adjust to a final volume of 2.5 ml. Inject on the GC and determine % recovery. Alternatively, the cartridge can be eluted a second time with another 8 ml portions of hexane and injected on the GC to determine if PCBs are still present in this eluate. If no PCBs are detected in this eluate, then it can be assumed that all the PCBs were recovered during the first elution and elution volume is valid. The sorrogates must also be recovered in the first eluate.

9.3.1.7. Solvent exchange the Florisil blank, adjust to a final volume of 2.5 ml and analyze on the GC.

9.3.2. <u>Florisil Cartridge Cleanup of Sample Extracts:</u>

9.3.2.1. Using conditioned cartridges as in steps 9.3.1.2 and 9.3.1.3, place 2.5 ml of sample extract on top of the cartridge. Elute the sample extract as in step 9.2.1.4. Repeat for the rest of the sample and QC sample extracts. **(Warning: Do not allow the solvent level to go below the top**

of the florisil until after the last portion of eluting solvent has been added).

9.3.2.2. Concentrate/solvent-exchange the eluates using the TurboVap LV (or equivalent) as described in 9.2.5.5. The final volume should be equal to the initial volume of sample extract eluted through the cartridge, with hexane as the final solvent.

9.3.2.3. Proceed with other required cleanup procedures.

10. Quality Control

10.1. The laboratory is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of method blanks, spiked samples and laboratory control standards to evaluate and document data quality. Ongoing data quality checks are compared with established performance criteria to determine if the results of the analysis meet the criteria. See the analysis SOP (GC003) for the applicable control limits.

10.2. A Florisil cleanup blank to which the required surrogates have been added is processed in the same manner as the samples. A florisil cleanup blank is analyzed with every new batch (different lot number) of florisil or florisil cartridges. The % recoveries of the surrogates are calculated. Target analytes must be less than the CRL MDL. Surrogate recoveries should fall within 70 – 130%.

10.3. A Florisil cleanup control standard is processed in the same manner as the samples. A florisil cleanup control standard is analyzed with every new batch (different lot number) of florisil or florisil cartridges. The % recoveries of the analytes are calculated. The CRL acceptance criteria for cleanup standards are recovery.

11. Data and Records Management

11.1. Raw data and bench sheets are to be submitted with the data package to the CRL data coordinator.

11.2. Reagents and standards LIMS identification numbers used for procedure should be recorded on bench sheets and reviewed and submitted along with data package.

11.3. All reviews are to be performed following the analytical procedure CRL.SOP GC014 and data review procedure CRL.SOP GEN 015.

11.4. All electronic records associated with any data package generated must be archived following CRL.SOP GEN001.

12. Troubleshooting

12.1. For a more detailed description of the apparatus and its operation, refer to TURBO-VAP 500 Closed Cell Concentrator Operator's Manual. A user's guide to daily operation of the instrument can be found in a plastic pocket at the side of the instrument

12.2. Refer to Operating Manual for the proper operation of TURBO-VAP LV (Zymark, Inc.). A user's guide to daily operation is located in a plastic pouch at the side of the instrument.

13. Preventative Maintenance

13.1. Preventative maintenance records and logbooks are kept with the instrument.

14. References

14.1. SW-846 Revision 2 FINAL UPDATE I DEC 1996 METHOD 3620C FLORISIL CLEANUP

14.2. RESTEK Environmental Applications Note#59562

14.3. CRL Quality Management Plan

15. Revision History

Version	Status*	Location of Change History
2	R	Section 4.2 included SDS information.
		Added revision table section 15.
1	R	1st version published to Qualtrax, same as GC014 Rev. 4.0 with minor formatting changes
		Entire document – Added header and adjusted spacing
		Title page – Deleted signature lines
4	R	Editing changes were made throughout the document to improve clarity
		Section 4.0 was combined with section 11.0
		SOP template was reformatted to be in compliance with new GEN006 template.
		Added warning section to 4.2
		Waste handling section was added to 4.4.
		Added recertified thermometer to section 6.6.
		Added vendor order requirements to section 7
		Section added Report all major spills and reference chemical hygiene plan in section 4.0.