# Microwave Extraction

Reference Methods: **EPA 3546**, SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III, 1997.

**Method 8082**, Reference: SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III, 1997.

Method for the Determination of Extractable Petroleum Hydrocarbons (EPH), Massachusetts DEP, ORS, BWSC. MADEP-EPH-04-1, Revision 1.1 May 2004.

**CT-ETPH**, Environmental Research Institute – University of Connecticut, March 1999.

**Maine 4.1.25,** Maine Health and Environmental Testing Laboratory, 09/06/1995.

**EPA 8015B (Modified),** SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III, December 1996

Extractable Petroleum Hydrocarbons Methodology, New Jersey Department of Environmental Protection, NJDEP EPH 10/08, Revision 2 October 2009.

## 1. Scope and Application

**Matrices:** Soil, solid, clay, sediment, sludge, solid waste

**Definitions:** Refer to Alpha Analytical Quality Manual.

Method 3546 is a procedure for extracting water insoluble or slightly water soluble organic compounds from soils, clays, sediments, sludges, and solid wastes. The procedure uses microwave energy to produce elevated temperature and pressure conditions (i.e., 75°C and 50 - 175 psi) in a closed vessel containing the sample and organic solvent(s) to achieve analyte recoveries equivalent to those from Soxhlet extraction (Method 3540), using less solvent and taking significantly less time than the Soxhlet procedure.

This method is applicable to the extraction of a variety of semivolatile organic compounds, some of which are: substituted phenols, PCBs, and PCDDs/PCDFs. The extracts are analyzed by the appropriate chromatographic procedure(s).

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the Microwave. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability, analyzing a proficiency test sample and completing the record of training.

After initial demonstration, ongoing demonstration is based on acceptable laboratory performance of at least a quarterly laboratory control sample or acceptable performance from an annual proficiency test sample. A major modification to this procedure requires demonstration of performance. The identification of major method modification requiring performance demonstration is directed by the Quality Assurance Officer and/or Laboratory Director on a case-by-case basis.

# 2. Summary of Method

Samples are prepared for extraction following the LEAN one-piece flow methodology and loaded into the extraction vessel. The appropriate solvent is added to the vessel and sealed. The extraction vessel containing the sample and solvent is heated to the extraction temperature and extracted for 20- 40 minutes. The extraction mixture is allowed to cool. The vessel is opened and the contents are filtered.

The extract is then concentrated and (as needed) exchanged into a solvent compatible with the cleanup or determinative procedure being employed. Extracts are then vialed and transferred to the Analytical Department.

## 2.1 Method Modifications from Reference

None.

# 3. Reporting Limits

Reporting Limit information may be found in the various determinative method SOPs.

# 4. Interferences

**4.1** The most common cause of contamination is from improperly cleaned glassware and lab supplies. All glassware and re-useable extraction equipment (i.e. spatulas, extraction vessels) must be scrupulously cleaned, following the Organic Extraction Glassware Cleaning and Handling SOP.

**4.2** Impurities in solvents and reagents may also yield artifacts and/or interferences that may compromise the results of sample analysis. All of these materials must be demonstrated to be free from interferences under the conditions of extract preparation and analysis by preparing method blanks with each extraction batch. The same solvents and reagents are used for the method blank and the associated samples.

**4.3** Phthalate esters contaminate many types of products used in the laboratory. Plastic materials must not contact the samples or extracts, as phthalates could be easily leached from the plastic. The exception is in the use of various pre-packed reagent cartridges (Florisil, Silica gel) used in the extract cleanup steps. Each new lot of cartridges is checked for contamination, and is monitored on an on-going basis through the analysis of method blanks.

**4.4** Additional specific interference or contamination concerns are addressed in the various analytical SOPs. If necessary, Florisil, Sulfuric Acid, Silica Gel and/or Sulfur cleanup procedures may be employed.

# 5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; 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. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

**5.1** The extraction vessels are at elevated temperatures and pressure after the extraction stage. Allow the vessels to cool before opening and always monitor the temperature and pressure by reconnecting the control vessel to the apparatus prior to opening the vessels.

**5.2** During the heating step, some solvent vapors may escape through the vessel liner/seal cover. Follow the manufacturer's directions regarding the vessel assembly and instrument setup to prevent release of solvent vapors to the laboratory atmosphere.

**5.3** The instrument contains flammable vapor sensors and should be operated with all covers in place and doors closed to ensure proper operation of the sensors. Follow the manufacturer's directions regarding replacement of extraction vessel seals when frequent vapor leaks are detected.

**5.4** Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards or solvents and when washing glassware.

**5.5** All extract concentration steps must be performed in the extraction hoods. All solvent and extract transfers must also be handled in the hood.

**5.6** All expired stock standards, working standards, and spent sample extracts must be placed into the waste bucket in the lab, for future disposal by the Hazardous Waste Manager. The container must be properly labeled with hazard warning labels indicating the container contents.

**5.7** Bottles containing flammable solvents must be stored in the flammables cabinet or in the vented cabinets found under the hoods.

**5.8** All waste solvents must be transferred to the satellite waste storage containers located in the extraction lab. Separate containers are provided for chlorinated and non-chlorinated solvents and must be used accordingly. Under no circumstances are solvents to be poured down the sink drains.

**5.9** Inspect all glassware prior to use. Do not use any glassware that is chipped, cracked or etched if it could present a safety hazard. Damaged glassware is put aside for repair, otherwise discard the piece.

**5.10** All Field Samples must be opened and handled in a hood.

# 6. Sample Collection, Preservation, Shipping and Handling

## 6.1 Sample Collection

Sample collection and preservation requirements are described the in the various analytical method SOPs.

## 6.2 Sample Preservation

None.

## 6.3 Sample Shipping

See applicable Sample Custody SOP.

### 6.4 Sample Handling

All soil samples are stored, refrigerated, in the Custody sample refrigerators. Samples are removed by the analyst immediately prior to sample extraction. The chemist must take custody of the samples by signing them out utilizing the LIMS.

When possible, samples must be homogenized prior to taking the sample aliquot, as described in Section 10.1. After the sample aliquot is removed, the samples are returned to the Sample Bank and placed in the appropriate sample refrigerator. Custody of the samples is transferred utilizing the LIMS.

## 7. Equipment and Supplies

**7.1 Spatulas:** Stainless steel.

**7.2 Beakers:** Stainless Steeel 250mL

**7.3 Mortar and Pestle:** Capable of reducing particle size to <1mm.

**7.4 Kuderna-Danish (K-D) apparatus:**

**7.4.1 Concentrator tube:** 25mL, graduated and calibrated. A ground-glass stopper is used to prevent evaporation of extracts.

**7.4.2 Evaporation flask:** 500mL. Attach to concentrator tube with plastic kek clips.**Snyder column: Th**ree-ball macro.

**7.4.3 Plastic Kek Clips.**

**7.5 Water Bath:** Heated, with concentric ring cover, capable of temperature control (± 5°C). The bath is used in a laboratory hood.

**7.6 Boiling Chips:** Solvent-extracted, approximately 10/40 mesh (silicon carbide or equivalent).

**7.7 Syringe: 1.0mL, 250µL, 25uL, Various sizes for Vialing**,

**7.8 Disposable Borosilicate Transfer Pipets.**

**7.9 Brady labeling system:** Thermal label generator.

**7.10 Glass Funnel.**

**7.11 Glass wool:** SUPELCO, silane treated.

**7.12 Whatman Filter Paper used for filtering all Pesticide/8081.**

**7.13 Graduated Cylinder:** 25 and 50mL.

**7.14 N-EVAP:** Organomation; Various sizes utilized for micro blowdown.

**7.15 Turbo Vap II:** Auto-concentrator; Caliper Life Sciences.

**7.16 Zymark Tubes:** 50mL or 200mL

**7.17 Analytical Balance:** Capable of weighing to 0.01g.

**7.18 Milestone Microwave Laboratory System:**

**7.18.1 Microwave extraction vessels:** With inserts, plugs, and caps. Capable of accommodating 1g to 30g of sample and 30mL of Extraction Solvent. Vessels are transparent to microwave energy, relatively inert to reagents and sample components, and capable of withstanding the temperature and pressure requirements (minimum conditions of 75°C and 200psi) necessary to perform this procedure. Follow the manufacturer's instructions regarding cleaning, handling, and sealing the vessels.

**7.18.2 Extraction Vessel Turntable: and cover.** Used to hold and rotate the extraction vessels during extraction.

**7.18.3 ATC-FO Sensor and Thermowell.** To measure sample temperature (± 2.5°C) and adjust microwave wattage accordingly, up to 1200W.

**7.19 Microwave Accelerated Reaction System (MARS):** CEM Corporation. The temperature performance requirements necessitate that the microwave extraction system be capable of sensing the temperature to within ± 2.0°C and automatically adjusting the microwave field output power within 2 seconds of sensing. Temperature sensor is accurate to ± 2°C and adjustable microwave wattage to 1600W. Temperature feedback control provides the primary performance mechanism for the method.

**7.19.1 Microwave extraction vessels:** With plugs and caps. Capable of accommodating 1g to 30g samples. Vessels are transparent to microwave energy, relatively inert to reagents and sample components, and capable of withstanding the temperature and pressure requirements (minimum conditions of 75°C and 200psi) necessary to perform this procedure. Follow the manufacturer's instructions regarding cleaning, handling, and sealing the vessels.

**7.19.2 Kevlar sleeves.**

**7.19.3 Extraction Vessel Turntable:** Used to hold and rotate the extraction vessels during extraction.

# 8. Reagents and Standards

Reagent grade chemicals are used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

**8.1 Reagent Water:** All references to water in this method refer to reagent water from Alpha's RO water treatment system.

**8.2 Sodium Sulfate ($Na_2SO_4$):** Granular anhydrous; purified by baking at 400ºC for 4 hours in a shallow tray. Store in closed glass containers. All references to sodium sulfate in this method refer to this prepared reagent. Sodium sulfate is also used for filtering.

**8.3 Ottwa Sand:** Purified by baking at 400C for 4 hours in a shallow tray.

**8.4 Hexane:** Pesticide quality.

**8.5 Acetone:** Pesticide quality.

**8.6 Dichloromethane**: Pesticide quality.

**8.7 Nitrogen Gas:** Reagent grade, used to purge and pressurize the extraction cell and as the concentration gas in the Turbovap II auto-concentrator units and the N-EVAP.

**8.8 Spiking Solutions:** Commonly used surrogate and LCS/MS spiking solutions used in the extraction steps are listed in Table 2. Additionally, the Sample Extraction Guide has a complete listing of all surrogate and LCS/MS spiking solutions. The preparation and expiration dates of these solutions are described in the analytical SOPs.

**8.9 Extraction Solvents:** This method has been validated using a 1:1 mixture of hexane and acetone, 1:1 mixture of methylene chloride and acetone, or 100% Methyelene Chloride from matrices such as soil, glass-fibers, and sand. Other solvent systems may have applicability in microwave extraction, provided that at least one component absorbs microwave energy. The choice of extraction solvent will depend on the analytes of interest and no single solvent is universally applicable to all analyte groups. Whatever solvent system is employed, *including* those specifically listed in this method, the analyst *must* demonstrate adequate performance for the analytes of interest, at the levels of interest. Method 8000 describes procedures that may be used to develop performance criteria for such demonstrations as well as for matrix spike and laboratory control sample results.

Hexane is a water-immiscible solvent and acetone is a water-miscible solvent. The purpose of the water-miscible solvent is to facilitate the extraction of wet solids by allowing the mixed solvent to penetrate the layer of water of the surface of the solid particles. The water immiscible solvent extracts organics compounds with similar polarities. The polarity of acetone may also help extract polar analytes in mixed solvent systems. When 100% Methyhlene Chloride is used, water is added as a catalyst to absorb microwave energy for method 8270.

# 9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

Each extraction batch contains various QC samples used to ensure the validity of the sample results. The particular QC elements performed for a given extraction batch are determined by the requirements of the determinative method. The purpose and definition of the QC samples

performed are listed below. Specific QC requirements of the analytical methods are listed in Table 2 and the Sample Extraction Guide.

## 9.1 Blank

Blanks, or method blanks, are measured aliquots of clean matrix (typically sodium sulfate or sand for soil extractions) that are treated identically to the associated samples. Surrogates are added, and the blanks are carried through all stages of the sample extraction, concentration, and cleanup procedures. Blanks serve to ensure that no systematic contamination exists. A blank is extracted with each batch or 20 or less samples.

## 9.2 Laboratory Control Sample / Laboratory Control Sample Duplicate (LCS/LCSD)

LCS samples are measured aliquots of clean matrix (typically sodium sulfate for soil extractions) that are spiked with a solution containing known amounts of target compounds, in addition to the surrogate solution. The LCS is carried through all stages of the sample extraction, concentration, and cleanup procedures. LCS samples serve as batch specific quantitative checks of the extraction. An LCS is extracted with each batch of 20 or less samples.

An LCSD is performed in addition to an LCS for all Massachusetts Contingency Plan (MCP) methods, as well as in lieu of the MS/MSD or Duplicate when there is insufficient sample volume available. The required solutions are listed in Table 2 and the Sample Extraction Guide.

## 9.3 Initial Calibration Verification (ICV)

Not Applicable.

## 9.4 Continuing Calibration Verification (CCV)

Not Applicable.

## 9.5 Matrix Spike / Matrix Spike Duplicate (MS/MSD)

MS and MSDs are field samples spiked with a known quantity of the target analyte(s). They are prepared by taking additional sample aliquots, and adding the appropriate amounts of surrogate and spiking solutions. The MS/MSD are carried through all stages of the sample extraction, concentration, and cleanup procedures. MS samples serve as a measure of extraction accuracy, by allowing the comparison of the found amount(s) of target analyte(s) with the spiked amount(s). An MS/MSD set also allows for the calculation of the extraction precision, by comparing the results of the two samples. Requirements for MS and MSD are listed in Table 2 and the Sample Extraction Guide.

## 9.6 Laboratory Duplicate

Duplicates are laboratory selected replicate samples, prepared by taking an additional sample aliquot of a sample. The duplicate is carried through all stages of the sample extraction, concentration, and cleanup procedures. Duplicates serve as a measure of the extraction precision, by comparing the results of the sample and duplicate. Requirements for Duplicates are listed in Table 2 and the Sample Extraction Guide.

## 9.7 Method-specific Quality Control Samples

### 9.7.1 Surrogates

Surrogates are compounds specified by the analytical method that are added to all samples and QC samples prior to beginning the extraction process. Surrogate recoveries are calculated and serve as a sample specific quantitative check of the

extraction. The various spiking solutions are prepared according to the directions found in the analytical SOPs. The required solutions and volumes used are listed in Table 2 and the Sample Extraction Guide.

### 9.8 Method Sequence

Refer to Section 10.

## 10. Procedure

All soil microwave soil extracts follow the LEAN "one-piece flow". All extraction information is recorded by the chemist performing the work in the ELN (Electronic Lab Notebook) see WI/2517. In addition to recording the extraction, concentration, clean-up and vialing information, the analyst must note the matrix "type" along with any observations, deviations from the procedure, or difficulties encountered with the samples in the comment section of the logbook.

### 10.1 Sample Preparation and Extraction

**10.1.1** Soil Samples are scanned and removed from Sample Login Custody to Oprep Custody. Immediately after scanning, the samples are individual scanned into the ELN to create the Work Group. Labels are printed and placed on the cap of the soil container. See Work Instruction 2421, Labeling and Generating Work Groups and Batches.

**10.1.2** All Glassware is cleaned prior to the Extraction following SOP 1953, Organic Extraction Glassware Cleaning and Handling.

**10.1.3** During the extraction process, each soil or sediment sample is visually inspected. If a sample contains a significant amount of free water, the analyst must contact login or the project manager to determine if the water is to be considered part of the sample. If the water is not to be homogenized with the solid material, decant and discard the water layer. Record this information in the comments section of the ELN.

Any artifacts (rocks, leaves, sticks, or similar materials) are not typically considered part of the soil sample and are not to be included. If necessary, transfer these artifacts to another container prior to homogenizing the sample. Note the presence of sample artifacts in the ELN. Gummy, fibrous, or oily materials not amenable to grinding must be cut, shredded, or otherwise reduced in size to allow mixing and maximum exposure of the sample surfaces for the extraction. Record the sample matrix "type/description" in the comments section of the ELN using the Sample Matrix Description spreadsheet.

**10.1.4** When possible, homogenize the sample well using a spatula by mixing the contents of the sample container. If this is difficult due to sample matrix, describe the non-homogeneity in the ELN.

**10.1.5** The chemist must demonstrate that all equipment used during the extraction process interference-free. This is accomplished through the analysis of a solid matrix (Sodium Sulfate or Ottowa Sand) Method Blank (SB). A Method Blank is extracted with each batch of 20 or less samples.

**10.1.6** Two different microwave systems are eligible for use. Both use the same temperature program and microwave energy, except the Milestone monitors temperature using a probe. Both systems are acceptable for all Extraction Methods.

**10.1.7** **Milestone Operation:** Rinse the reaction vessels, caps, and plugs with Acetone and then DCM or Hexane.

**10.1.7.1** See Work Instruction WI/2421 for proper labeling procedures and one-piece flow operation. Transfer the sample into the reaction vessel from the beaker or

weighing tray. Add the appropriate surrogate and spiking solution. (Refer to Table 2 and the Sample Extraction Guide). Sodium Sulfate is used for the QC substrate for all methods except 8270. Ottowa Sand is used for 8270 QC.

**10.1.7.2** **For all Methods except 8270:** Add 25mL of 1:1 Hexane:Acetone, 1:1 DCM:Acetone or 100% DCM (or the appropriate extraction solvent) to each reaction vessel. (Refer to Table 1).

**10.1.7.3** Place a plug and a cap on each reaction vessel with one exception. One vessel needs to accommodate the temperature probe. This cap does not have the insert screwed into the middle of the cap. Place the thermowell into the vessel and screw on the cap.

**10.1.7.4** Place all labeled vessels onto the carousal making sure that the vessel prepped for the probe is in section one or two. Section one or two allows for easy probe installation. Place the top of the carousel onto the top of the extraction vessels.

**10.1.7.5** Place the carousal on the open microwave door and fit the temperature probe into its receptacle. Place the carousal into the microwave.

**10.1.7.6** On the microwave panel, log in as the “user”(oprep) and load the password (1,2,3). Load the method ”use this method” and click start. The method consists of a 10 minute temperature ramp up to 75 deg. C. Followed by a 30 minute hold step at 75 deg C. Following the extraction, the carousal can be removed to cool.

**10.1.7.7** Once samples have cooled to room temperature, filter through a sodium sulfate funnel and into a labeled KD Flask and concentrator tube. Note- When opening the cap on the Teflon reaction vessel, point away from your body and perform this task in a fume hood.

**10.1.7.8** Proceed to sample concentration and cleanup step as required by the analytical method for the analyte(s) of concern. (Refer to Table 1.)

**10.1.7.9** **For 8270:** See Work Instruction WI/2421 for proper labeling procedures and one-piece flow operation. Transfer the sample into the reaction vessel from the beaker or weighing tray. Note: sodium sulfate is **not** mixed with the sample. For QC samples, Ottowa sand is used, Add the appropriate surrogate and spiking solution. (Refer to Table 2 and the Sample Extraction Guide). After adding the sample to the reaction vessel, using a 1.0mL syringe, add 1.0mL of DI water to the sample.

**10.1.7.10** Add 25mL of 100% DCM to each reaction vessel. (Refer to Table 1).

**10.1.7.11** Place a plug and a cap on each reaction vessel with one exception. One vessel needs to accommodate the temperature probe. This cap does not have the disk screwed into the top of the cap. Place the thermowell into the vessel and screw on the cap

**10.1.7.12** Place all labeled vessels onto the carousal making sure that the vessel prepped for the probe is in section one or two. Section one or two allows for easy probe installation. Place the top of the carousel onto the top of the extraction vessels.

**10.1.7.13** Place the carousal on the open microwave door and fit the temperature probe into its receptacle. Place the carousal into the microwave.

**10.1.7.14** On the microwave panel, log in as the “user”(oprep) and load the password (1,2,3). Load the method ”method 8270” and click start. The method consists of a 10 minute temperature ramp up to 75 deg. C. Followed by a 10 minute hold step at 75 deg C. Following the extraction, the carousal can be removed to cool.

**10.1.7.15** Once samples have cooled to room temperature, filter through a sodium sulfate funnel(using DCM only) and into a labeled KD Flask and concentrator tube. Note- When opening the cap on the Teflon reaction vessel, point away from your body and perform this task in a fume hood.

**10.1.7.16** Proceed to sample concentration and cleanup step as required by the analytical method for the analyte(s) of concern. (Refer to Table 1.)

**10.1.8** **MARS Microwave System Operation:** Rinse the reaction vessels, caps, and plugs with Acetone and then DCM or Hexane. See Work Instruction WI/2421 for proper labeling procedures and one-piece flow operation.

**10.1.8.1** **For all Methods except 8270:** Using the MARS reaction vessel, caps and plugs, repeat steps 10.1.7 -10.1.7.16 for all samples. Note the MARS System does not use an external temperature probe, rather an internal temperature system.

**10.1.8.2** On the front panel of the microwave, select "Use This Method" from the User Directory. Select the appropriate extraction method: "3546 Express"

The following parameters are loaded with 3546 Express method:

- Stage 1: 1600W @ 100% Power.
- Ramp to 75°C for 10 minutes and hold for 30 minutes.
- This stage is followed by a 5 minute Cool Down step.

**10.1.8.3** Remove each reaction vessel from the carousel for cooling.

**10.1.8.4** **Methods 8270:** Using the MARS reaction vessel, caps and plugs, repeat steps 10.1.7.9 -10.1.7.16 for all samples. Note the MARS System does not use an external temperature probe, rather an internal temperature system.

**10.1.8.5** On the front panel of the microwave, select "8270 Soil" from the User Directory. Select the appropriate extraction method: "3546 Express"

The following parameters are loaded with 3546 Express method:

- Stage 1: 1600W @ 100% Power.
- Ramp to 75°C for 10 minutes and hold for 10 minutes.
- This stage is followed by a 5 minute Cool Down step.

**10.1.8.6** Once samples have cooled to room temperature, filter through a sodium sulfate funnel and into a labeled KD Flask with concentrator tube or filter into a Zymark Tube for Turbovap concentration (See Section 10.3). Note- When opening the cap on the Teflon reaction vessel, point away from your body and perform this task in a fume hood.

**10.1.8.7** Proceed to sample concentration and cleanup step as required by the analytical method for the analyte(s) of concern. (Refer to Table 1.)

## 10.2 Sample Concentration: KD Technique

**10.2.1** Attach a three-ball Snyder column to the top of the flask. Place the KD apparatus on a hot water bath (heated to approximately 75$^{o}$C for samples extracted in Methylene Chloride and 95$^{o}$C for samples extracted in Hexane) so that the concentrator tube is partially immersed in the hot water, and so that the entire lower rounded surface of the flask is bathed in hot water vapor. Adjust the position of the apparatus as required to complete the concentration in 30 to 40 minutes. At the proper rate of distillation, the balls

in the column will actively chatter, but the chambers will not flood with solvent. Periodically rinse the internal walls of the concentrator tubes with DCM.

**10.2.2** If a Hexane exchange is required (see Table 1), add 35mL of hexane, when the sample volume reaches approximately 5-10mLs. Add the hexane to the top of the Snyder column while the concentrator is still on the water bath. Continue with the concentration until the extract volume is reduced to below 10mL. Periodically rinse the internal walls of the synder column with 5-10mL of Hexane.

**10.2.3** Remove the KD apparatus from the water bath. Rinse the flask and its lower ground glass joint with 1 to 2 mL of acetone to remove any moisture from the outside of the glassware. Allow to cool for 15minutes. Disassemble the KD apparatus. Move the label from the K-D Flask to the concentrator tube (See WI 2421 Labeling and Generating Work Groups and Batches).

**10.2.4** Place the Concentrator tube on the N-EVAP. The N-EVAP is set at 37°C for samples extracted in Methylene Chloride and 65 °C for samples extracted in Hexane with the nitrogen flow at 5 - 7. Samples remain on N-EVAP until they are reduced to 1mL. **Pesticide Extracts:** Using a syringe or volumetric flask, adjust final volume to 10 mL with hexane.

**10.2.5** The extract is now ready for sample cleanup or vialing (See Table 1). Refer to the relevant Clean-up SOP or proceed with extract vialing (See WI 3827 Extract Vialing Procedure, WI 2426 GC Extract Vialing Procedure and WI 2423 GC/MS Extract Vialing Procedure).

## 10.3 Alternate Concentration Technique

The equipment used for alternate sample concentration is the Zymark TurboVap II, which is a self-contained water bath and nitrogen blow-down. It is equipped with sensors to allow for automatic shutdown when the extract volume goes below 1mL. This technique is generally used for concentration of TPH samples.

**10.3.1** The TurboVap is set at 37°C for samples extracted in Methylene Chloride. The nitrogen pressure is set to 10-15 psi and the endpoint concentration is set to 'Sensor'. The Zymark tube is labeled with the sample I.D.

**10.3.2** Place the labeled Zymark tube(See WI 2421 Labeling and Generating Work Groups and Batches) containing sample into the TurboVap II concentration unit. Start the nitrogen blow-down by pressing the button on the front panel of the unit.

**10.3.3** When the concentration is complete, the nitrogen stream will be automatically shut off. The light on the front panel will flash, and the instrument will beep. Remove the tube from the Zymark concentrator; the final extract volume is 1mL. Refer to the relevant Clean-up SOP or proceed with extract vialing (See WI 3827 Extract Vialing Procedure, WI 2426 GC Extract Vialing Procedure and WI 2423 GC/MS Extract Vialing Procedure).

**10.3.4**

## 10.4 Preventive Maintenance

**10.4.1 Microwave System (MARS and Milestone):**

**10.4.1.1** The instrument must be kept clean. Wipe the inside of microwave with soap and water and dry with a cloth as needed.

**10.4.1.2** All microwave cells, caps and plugs are to be dish washed and rinsed with solvent prior to use, with one exception: Milestone caps are to be rinsed with solvent only, and not placed in the dishwasher. Additionally, the sleeves must be

handled with care. If the edge of the sleeve is dented or chipped it will not hold pressure.

**10.4.2 TurboVap II Concentrators**

**10.4.2.1** Maintain the level of the water bath by adding water daily to fill. Keep unit clean. Avoid solvent spills on or around unit. Clean periodically with a damp cloth.

**10.4.3 Analytical Balance**

**10.4.3.1** All balances are calibrated and serviced every six months by an instrument service company. All service records are kept on file.

**10.4.3.2** Keep balances clean. Brush of any sample spills immediately. Keep the balance doors closed and the balance turned off when not in use.

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# 11. Data Evaluation, Calculations and Reporting

None.

# 12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

**12.1** All Holding time exceedences, improper preservation and Extraction Anomalies are to be reported to a Supervisor or Manager. Non Conformance Reports may need to be issued through the Qualtrax System.

**12.2** If the KD Concentrator tube is allowed to run dry, the sample must be re-extracted.

**12.3** Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

**12.4** Refer to determinative method SOPs for additional Corrective Action information.

# 13. Method Performance

## 13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

## 13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

**13.2.1 Initial (IDC)**

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

**13.2.2 Continuing (DOC)**

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

# 14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

# 15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 DL/LOD/LOQ Generation

SOP/1739 DOC Generation

SOP/14-01 Waste Management and Disposal SOP

SOP/1953 Organic Extraction Glassware Cleaning and Handling

Form 02-50 Sample Cleanup and Vialing Guide

WI/2421 Labeling and Generating Work Groups and Batches

WI/2517 LIMS Electronic Laboratory Notebook Procedure

WI/2423 GC Mass Spec Extract Vialing Procedure

WI/2426 GC Extract Vialing Procedure

WI/3827 Extract Vialing Procedure

Form 02-58 Sample Extraction Guide

# 16. Attachments

Table 1 – Specific Extraction Conditions for Various Determinative Methods

Table 2 – Soil Extraction Guide

# Table 1

## Specific Extraction Conditions for Various Determinative Methods

| LIMS Product Code | Extraction Solvent | Exchange Solvent Required | Typical Final Volume | Appropriate Cleanup Technique |
|---|---|---|---|---|
| 8082 | 1:1 Hexane/Acetone | hexane | 1 mL | Sulfuric acid/ Sulfur |
| 8081 | 1:1 DCM/Acetone | hexane | 10 mL | Florisil |
| 8270 SIM | 1:1 DCM/Acetone | --- | 1mL | --- |
| 8270 | DCM | --- | 1 mL | --- |
| TPH * | DCM | --- | 1 mL | --- |
| EPH | DCM | hexane | 1 mL | Silica gel Fractionation |
| ETPH | DCM | --- | 1 mL | Silica gel |

***TPH includes the following LIMS Products: TPH-DRO and TPH-DRO-D**

# Table 2

## Soil Extraction Guide

| LIMS Product Code | QC * | Solvent | Surrogate Solution | LCS/LCSD MS/MSD Solution |
|---|---|---|---|---|
| 8270 | SB, LCS, LCSD | DCM | 1mL ABN | 1mL MCP ABN |
| MCP 8270 | SB, LCS, LCSD | DCM | 1mL ABN | 1mL MCP ABN |
| PAH | SB, LCS, LCSD | DCM | 1mL ABN | 1mL MCP ABN |
| MCP PAH | SB, LCS, LCSD | DCM | 1mL ABN | 1mL MCP ABN |
| PAH-Low | SB, LCS, LCSD | DCM/ACE | 1mL ABN | 1mL MCP PAH-Low |
| MCP PAH-Low | SB, LCS, LCSD | DCM/ACE | 1mL ABN | 1mL MCP PAH-Low |
| NYTCL/ NJ 8081 | SB, LCS, LCSD | DCM/ACE | 250 uL PEST | 250 uL Pest |
| All other PEST/8081 | SB, LCS, LCSD | DCM/ACE | 1mL PEST | 1mL Pest |
| PCB/8082 | SB, LCS, LCSD | HEX/ACE | 250µL PEST | 250µL PCB |
| MCP PCB | SB, LCS, LCSD | HEX/ACE | 250µL PEST | 250µL PCB |
| EPH | SB, LCS, LCSD | DCM | 1mL EPH | 1mL EPH |
| EPH / PAH-L | SB, LCS, LCSD | DCM | 1mL EPH | 1mL EPH |
| NJ EPH | SB, LCS, LCSD | DCM | 1mL NJ EPH | 1mL NJ EPH |
| ETPH | SB, LCS, MS, Dup | DCM | 1mL DRO | 1mL ETPH |
| TPH * | SB, LCS, Dup | DCM | 1mL DRO | 1mL ETPH |
| ME4125 | SB, LCS, MS, Dup | DCM | 1mL DRO | 1mL ETPH |

**For additional product QC, Solvent, Surrogate and Spike solutions, see Form "Sample Extraction Guide".**

*** TPH includes the following LIMS Products: TPH-DRO and TPH-DRO-D**