# Shaker Table Extraction

References: This standard operating procedure (SOP) is a performance-based method. This SOP describes the procedure as developed by Alpha Analytical.

## 1. Scope and Application

**Matrices:** This method is applicable to the extraction of non-volatile and semivolatile organic compounds in solid samples (including soils, sediments, sludges, pads, nets and wastes).

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

Shaker Table extraction is the process of isolating organic compounds of interest from the solid sample matrix. Sample cleanup is highly recommended, since the compounds cannot be analyzed directly without removal of some interferences in the sample matrix. See Section 4.2 for a listing of Alpha Analytical sample cleanup SOPs.

This procedure uses a Shaker Table set at 120 to 150 rotations per minute for agitation of the mixture. Groups of organic compounds such as semivolatile PAHs and petroleum hydrocarbons are separated on the basis of their common solubility in solvents and then analyzed in accordance with the appropriate Alpha Analytical SOP:

- Total Petroleum Hydrocarbons by Gas Chromatography / Flame Ionization Detector (O-003),
- Analysis of Parent and Alkylated Polynuclear Aromatic Hydrocarbons and Selected Heterocyclic Compounds by Gas Chromatography / Mass Spectrometry with Selected ion Monitoring (O-008).

This procedure may be used for the extraction of all types of organic compounds. Because this procedure is performance based, it should only be used for compounds where studies have assessed the precision, accuracy, and sensitivity of the technique relative to the project specific goals.

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 equipment and in the interpretation of data. 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

A 5-30 gram aliquot of solid sample is weighed, then dried by mixing with sodium sulfate, to form a free-flowing powder. Pads and nets are directly extracted without drying.

This mixture is then solvent extracted three times using dichloromethane (DCM, or methylene chloride), the Shaker Table extractor solvent.

The extract is allowed to settle and the solvent is separated from the sample mixture by decanting through a filter funnel packed with pre-baked/clean sodium sulfate and glass wool.

Finally, the extract is concentrated and if necessary, cleanup procedures are performed prior to analysis in accordance with the appropriate Alpha Analytical SOP. See Section 4.2 for the list of Alpha Analytical cleanup SOPs and Section 1 for analytical SOPs.

## 2.1 Method Modifications from Reference

None.

# 3. Reporting Limits

Refer to Analytical SOPs for Reporting Limit information.

# 4. Interferences

**4.1** Solvents, reagents and glassware may introduce interferences. These must be demonstrated to be free of interferences by the analysis of a method blank. See the Alpha Analytical SOP *Reagent, Solvent and Standard Control* (G-008) and *Laboratory Glassware Cleaning* (G-002), for additional details.

**4.2** Many interferences can be removed by sample cleanup. The cleanup methods performed by Alpha Analytical include the following: *Alumina Column Cleanup of Organic Extracts* (OP-009), *Sulfur Cleanup* (OP-007), *Gel-Permeation Chromatography* (OP-006). Only appropriate cleanup techniques must be performed based on the suspected interference and the compounds of interest.

**4.3** Soapy residue may result in basic conditions on glassware and may cause degradation of some compounds. All glassware must be rinsed thoroughly with deionized water and solvent to remove soapy residue. See the Alpha Analytical SOP (G-002) *Laboratory Glassware Cleaning,* for additional details.

**4.4** Phthalate esters can be a major source of contamination if any material containing plasticizers (phthalates) comes in contact with the sample during the extraction process. Use of plastic or any material containing plasticizers (phthalates) should be avoided during extraction or analysis.

# 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.

# 6. Sample Collection, Preservation, Shipping and Handling

## 6.1 Sample Collection

*Solid samples:* A minimum of 100 grams of sample must be collected in a glass jar with a Teflon lined screw cap.

## 6.2 Sample Preservation

The samples must be refrigerated and maintained at 4 ±2°C until extraction and analysis.

## 6.3 Sample Shipping

No special shipping requirements.

## 6.4 Sample Handling

All solid samples must be extracted within 14 days from the date of collection, or up to one year from freeze date (if applicable). The sample extracts must be refrigerated and maintained at 4±2°C until analysis. Sample extracts must be analyzed within 40 days from date of extraction.

# 7. Equipment and Supplies

**7.1 Shaker Table:** New Brunswick Scientific, Innova 2300

**7.2 Drying oven:** Capable of maintaining 105°C and 400°C.

**7.3 Dessicator:** Containing color indicator dessicant.

**7.4 Wide Mouth Amber Jar :** 250 mL

**7.5 Erlenmeyer flasks:** 500mL

**7.6 Stainless steel spatulas.**

## 7.7 Concentration apparatus

**7.7.1 Kuderna-Danish (K-D) concentrator tubes:** 20mL, ground glass.

**7.7.2 Evaporation flasks:** 500mL, that attach to concentrator tubes with clips.

**7.7.3 Snyder columns:** 3-ball macro.

**7.7.4 Teflon boiling chips.**

**7.7.5 Water bath.**

**7.7.6 N-EVAP:** by Organomations, Nitrogen blow-down for micro-concentration.

**7.7.7 Turbo-vap:** With appropriate concentrator tubes by Zymark, or equivalent, as an alternative to the K-D apparatus.

**7.8 Top-loading analytical balance:** Capable of accurately weighing to the nearest 0.01gram.

**7.9 Vials:** 2mL, 4mL and 10mL for transfer of concentrated extract.

**7.10 Syringes:** 10uL - 10mL.

**7.11 Filter funnels**

# 8. Reagents and Standards

Use reagent grade chemicals for all reagents. Deionized (DI) water is ASTM Type II laboratory reagent grade water.

**8.1 Methylene Chloride:** ACS approved, Pesticide grade, see Alpha Analytical SOP *Reagent, Solvent and Standard Control* (G-008) for additional details regarding solvent purity.

**8.2 Sodium Sulfate:** granular, anhydrous. Purified by heating to 400°C for 4 hours.

**8.3 Glass wool:** purified by heating to 400°C for 4 hours.

**8.4 Spiking solutions**: Including the following solutions (see the appropriate Alpha Analytical SOP for details on the correct spike concentrations and spike volumes):

**8.4.1 Surrogate spiking solution**

**8.4.2 Laboratory Control Sample (LCS)/ Matrix Spiking (MS) solution**

**8.4.3 Recovery Internal Standard (RIS)**

# 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.

## 9.1 Blank(s)

A method blank must be prepared in sodium sulfate once per every 20 samples or per extraction batch, whichever is more frequent. If samples will be extracted for a variety of unassociated determinative analyses (*i.e.,* PAH, Pesticide and PCBs within the same extraction batch) a method blank for *each* analysis must be prepared and carried through the same extraction procedures as the samples.

Organic compounds of interest must not be detectable in the method blank at a concentration greater than the reporting limit.

Corrective Action: Extraction of the method blank and all associated samples must be performed until the blank is in control. Samples cannot be analyzed until an acceptable method blank analysis is obtained. Exceptions may be made with approval of the Department Manager,

Laboratory Director or QA Manager, if the samples associated with the out of control method blank are non-detect for the compound of interest, or if sample concentrations are greater than 10x the blank levels. In such cases, the sample results are accepted without corrective action for the high method blank and the client is notified in a project narrative associated with the sample results.

## 9.2 Laboratory Control Sample (LCS)

Laboratory control sample (LCS) and laboratory control samples duplicate (LCSD) must be prepared once per every 20 samples or per extraction batch, whichever is more frequent, in sodium sulfate and spiked with a solution prepared from a second source or lot number, other than the source used to verify the accuracy of the standard curve for the determinative analytical method. The LCS contains all target compounds of interest, and is extracted along with the samples as verification of the accuracy of the entire extraction procedure. If samples will be extracted for a variety of unrelated determinative analyses (*i.e.,* PAH, Pesticide and PCBs within the same extraction batch) a LCS and LCSD for *each* analysis must be prepared and carried through the same procedures as the samples.

The acceptable recovery QC limits are documented in the applicable Alpha Analytical SOPs.

Corrective Action: Analysis according to the appropriate Alpha Analytical SOP must be repeated once to see if an analytical error has occurred. If the LCS/LCSD recovery is still out of control, re-extract and re-analyze the LCS/LCSD and all associated samples. Samples cannot be reported until an acceptable LCS/LCSD is obtained. Exceptions may be made with approval of the Department Manager, Laboratory Director or QA Manager, if the samples associated with the out of control LCS/LCSD are also associated with a matrix spike or matrix spike duplicate pair that is in control. This is an acceptable measure of accuracy of the extraction and analytical procedures. An explanation of this out of control LCS/LCSD recovery must be included in the project narrative to the client and the sample data reported with the acceptable MS results as batch QC.

## 9.3 Initial Calibration Verification (ICV)

Not applicable to this method.

## 9.4 Continuing Calibration Verification (CCV)

Not applicable to this method.

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

Upon Client request, matrix spike / matrix spike duplicate (MS/MSD) samples are performed once per 20 samples (5% frequency). If samples will be extracted for a variety of unrelated determinative analyses (*i.e.,* PAH, Pesticide and PCBs within the same extraction batch) a MS/MSD pair for *each* analysis must be prepared and carried through the same procedures as the samples. If less than 20 samples are prepared in a one-week time frame, a MS/MSD pair will be extracted at the beginning of each week.

The acceptable recovery and %RPD QC limits are documented in the applicable Alpha Analytical SOPs. Calculate the %RPD as described in 9.6.

Corrective Action: Analysis according to the appropriate Alpha Analytical SOP must be repeated once to see if an analytical error has occurred. If the % recovery and/or %RPD still exceeds the control limits and the LCS is compliant; include a project narrative with the results to client noting that there may be potential matrix effects on the accuracy or precision of the reported results as evidenced by MS/MSD recoveries and/or %RPD outside of QC limits.

## 9.6 Laboratory Duplicate

Upon Client request, duplicate analyses (matrix or sample duplicate) are performed once per 20 samples (5% frequency). *For Organic analyses, the matrix duplicate is usually in the form of the matrix spike duplicate, see Section 9.5.*

Acceptable relative percent differences (RPD) of duplicates are documented in the applicable Alpha Analytical SOPs noted in Section 3.1.6. Acceptance criterion is not applicable to sample concentrations less than 5 times the reporting limit. Calculate the RPD as follows:

$$RPD = \frac{R1 - R2}{\frac{[R1 + R2]}{2}} \times 100$$

where:
R1 = sample Replicate #1
R2 = sample Replicate #2

Corrective Action: Analysis according to the appropriate Alpha Analytical SOP must be repeated once to see if an analytical error has occurred. If the % RPD still exceeds the control limits; include a project narrative with the results to client noting that there may be potential matrix effects on the precision of the reported organic results as evidenced by the matrix duplicate % RPD exceedance.

## 9.7 Method-specific Quality Control Samples

### 9.7.1 Surrogate Spike

Surrogate spikes must be added to QC and field samples to evaluate the extraction method performance.

The acceptable surrogate recovery limits are documented in the applicable Alpha Analytical SOPs.

Corrective Action: Analysis according to the appropriate Alpha Analytical SOP must be repeated once to see if an analytical error has occurred. If the % recovery still exceeds the control limits the sample must be re-extracted and re-analyzed to confirm the sample matrix. If *obvious* matrix interferences are noted, consultation with the Department Manager, Laboratory Director or QA Manager may be in order to confirm the need for sample re-extraction. If no re-extraction occurs, the surrogate results and reasons for the no re-extract decision must be discussed in the project narrative to the client.

### 9.7.2 Standard Reference Materials

Standard reference materials (SRM) are available from the National Institute of Standards and Technology (NIST) and are extracted and analyzed with samples on a project specific basis. These are not used as controls, but to evaluate potential matrix effects in associated samples for the target compounds being evaluated.

Acceptance criteria for SRM analysis will vary from project to project depending upon client data quality objectives (DQOs). Generally, ± 35% difference (%D)

based on the true certified values of the target compounds of interest, or 65% - 135% recovery, serve as advisory acceptance criteria.

Corrective Action: Analysis according to the appropriate Alpha Analytical SOP must be repeated once to see if an analytical error has occurred. If the % recovery and/or %D still exceeds the control limits, and the LCS and MS/MSD pair are compliant, include a project narrative with the results to client noting that there may be potential matrix effects on the accuracy or precision of the reported results as evidenced by SRM % recoveries and/or %D values outside of QC limits. These cases are normally isolated to the SRM, if all other controls are within limits.

### 9.8 Method Sequence

- Weigh homogenized sample (5-30g, depending on sample observations and DQO's)
- Dry with sodium sulfate
- Spike samples with surrogates as appropriate
- Add methylene chloride to cover sample
- Begin extraction - serially extract 3X following Procedure in Section 10.
- Sample concentration
- Sample cleanups - as needed or instructed
- Submit at determined pre-injection volume (PIV)

## 10. Procedure

### 10.1 Equipment Set-up

Samples are prioritized by the Department Manager or Preparation Team Leader for extraction based on hold time and client due date. The following steps shall be performed as rapidly as possible to avoid the loss of lighter weight, more volatile compounds.

### 10.2 Initial Calibration

Not applicable to this method.

### 10.3 Equipment Operation and Sample Processing

#### 10.3.1 Sample Extraction

**10.3.1.1** Examine the sample for any signs of heterogeneity. Decant any standing water and discard any rocks, sticks or foreign objects that are not representative of the sample (unless otherwise requested by the client), and mix well. Weigh a 30-gram aliquot of a well-mixed sample into a 250mL wide-mouth amber jar. **Note:** As low as 1 gram and up to 50 grams of sample may be extracted if requested, based on client required reporting limits or other specified instructions and is approved by the Project Manager and Department Manager. Record the weight to the nearest 0.01g. Nets and pads are extracted directly and weight for these samples is determined gravimetrically. See the Alpha Analytical SOP *Gravimetric Determination* (OP-017), for additional details.

**10.3.1.2** Add sodium sulfate incrementally to the sample, and mix well with a methylene chloride rinsed spatula until the sample is “powdery” or “free flowing”. For example, if a 20g-sample aliquot is weighed, then no more than 20g of sodium sulfate must be added to the sample. More sodium sulfate may be needed if the sample is not

"free-flowing". If more is needed, note this on the Sample Preparation Checklist.

**10.3.1.3** The amount of surrogate spike will depend upon the final detection limits, final sample volume and general nature of the sample. For Saturated Hydrocarbon (SHC) and Polynuclear Aromatic Hydrocarbon (PAH) analyses, 100uL of PAH/SHC low level surrogate, and 100uL of low level biomarker surrogate (if required) will be added to the QC samples and all samples that do not require high level spikes. For samples that need high level spikes, 500uL of low level surrogates (PAH/SHC, biomarkers) will be added to samples when required. 100uL LCS spiking solution shall be added to the LCS/LCSD samples and to any matrix spike and matrix spike duplicate samples associated with the extraction batch. If the MS/MSD samples need to be spiked with high level solution, 500uL of the LCS spiking solution will be added. *All sample spiking must be "spike witnessed".*

**10.3.1.4** Add 100mL of methylene chloride to each sample. Use enough solvent to cover the sample during extraction, and add an additional ½ inch of solvent, above the sample.

**10.3.1.5** Place the samples in a container, (*i.e.*, a box), on the Shaker Table and secure. Turn the table on, and set to 120 to 150 rotations per minute. Allow the samples to shake for a minimum of 14 hours.

**10.3.1.6** After a minimum of 14 hours, stop the Shaker Table and transfer the extract solvent, by decanting, into a labeled and solvent rinsed 500-mL Erlenmeyer flasks.

**10.3.1.7** Repeat the extraction steps (starting at 10.3.1.4) twice more with fresh portions of methylene chloride as the extraction solvent for the second and third shakes. The second shake of a minimum of 4 hours long and the third shake of a minimum of 30 minutes. The first and second extractions may be interchanged, but the 30 minute extraction must be the last Combine all three of the resulting extraction portions.

**10.3.1.8** The extract is now ready for concentration.

**10.3.2 Kuderna-Danish (K-D) Concentration**

**10.3.2.1** After all three extractions are complete transfer the extracts through a filter funnel fitted with baked and solvent rinsed glass wool and sodium sulfate, for additional extract drying. Rinse the filter funnel three times with methylene chloride from the laboratory squirt bottles to ensure a complete transfer of the entire extract into a 500 mL K-D Flask with the 20-mL concentrator tube attached to the bottom.

**10.3.2.2** Place the K-D flask on the water bath with 1-2 boiling chips in the bottom of the tube and the macro Snyder column on top. The bath temperature should be 75 ± 5°C. Macro-concentrate the sample to less than 10 mL. This will take approximately 45 minutes. At the proper rate of distillation, the balls of the column will chatter, but the column should not flood with condensed solvent.

**10.3.2.3** If the extract appears extremely viscous and reduces in volume very slowly, a final volume of greater than 10mL may be used to ensure that there is no loss of surrogates or the compounds of interest. See the Department Manager for additional guidance on troublesome matrices.

**10.3.2.4** Remove the sample from the bath and allow it to drain and cool for approximately 10 minutes.

**10.3.2.5** Move the cooled sample, still in the 10-mL concentrator tube, to the N-EVAP unit for micro-concentration, and bring the extract below the final volume required before cleanup. The extract must be concentrated under a *gentle* steady stream of nitrogen. The internal walls of the concentrator tube must be rinsed periodically with the appropriate solvent during evaporation. If the extract appears extremely viscous and reduces in volume very slowly then a final volume of 5-10mL should be used to ensure that there is no loss of surrogates or the compounds of interest. If sample requires solvent exchange, micro concentrate sample to around 1mL and add approximately 5mL of desired solvent. Micro-concentrate back to around 1mL, while occasionally lightly swirling the sample during concentration and add approximately 5mL again and micro-concentrate back to around 1mL while occasionally swirling sample during concentration. **Note**: Micro-concentration may not be needed depending upon the determinative analytical method. Always see the Department Manager for additional guidance when needed.

**10.3.2.6** Transfer the sample extract from the concentrator tube to a vial of the appropriate final volume size. The sample may now undergo any necessary cleanup that may be required prior to analysis. Ensure all samples are volumized to the required volumes using dichloromethane. See Section 4.2 for Alpha Analytical sample cleanup SOP references. If the sample does not require cleanup, it may be directly transferred at the correct final volume, and with 100uL of RIS added at a 1mL pre-injection volume, and 150uL aliquoted and transferred for each analyses along with copies of the sample preparation extraction log, the preparation checklist and the client folder, to the analytical instrument room for analysis.

### 10.4 Continuing Calibration

Not applicable to this method.

### 10.5 Preventive Maintenance

All maintenance records including routine upkeep and outside service visits are maintained in the instrument maintenance logbooks for the Lab-line Orbit Shaker Table.

## 11. Data Evaluation, Calculations and Reporting

Procedures for data and record management for organic extraction must adhere to the Quality Systems Manual, other subordinate documents covering record keeping, and the Alpha Analytical *Document Control* SOP/08-01. All records must be stored in such a manner as to be safe and accessible for at least 10 years.

The extraction bench sheets and other relevant laboratory notebooks must follow the specifications in the Alpha Analytical *Laboratory Notebook Usage Work Instructions* (WI 108-01), and all record keeping and document control practices.

See the appropriate Alpha Analytical SOPs for details on sample analysis, data evaluation, calculations and data reporting.

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

All results for the organic compounds of interests are reportable without qualification if extraction and analytical holding times are met, preservation (including cooler temperatures) are met, all QC criteria defined in the table below are met, and matrix interference is not suspected during extraction or analysis of the samples. If any of the below QC parameters are not met, all associated samples must be evaluated for re-extraction and/or re-analysis.

| QC Parameter | Acceptance Criteria |
|---|---|
| Method Blank | < reporting limit |
| Laboratory Control Sample / Duplicate | See the applicable Alpha Analytical SOP for acceptance criteria |
| Surrogate Recoveries | See the applicable Alpha Analytical SOP for acceptance criteria |
| Matrix Duplicate | See the applicable Alpha Analytical SOP for acceptance criteria |
| Matrix Spike | See the applicable Alpha Analytical SOP for acceptance criteria |
| Matrix Spike Duplicate | See the applicable Alpha Analytical SOP for acceptance criteria |
| Standard Reference Material | See the applicable Alpha Analytical SOP for acceptance criteria |

If non-compliant organic compound results are to be reported, the Department Manager and/or the Laboratory Director, and the QA Manager must approve the reporting of these results. The laboratory Project Manager shall be notified, and may chose to relay the non-compliance to the client, for approval, or other corrective action, such as re-sampling and re-analysis. The analyst or Section Head performing the secondary review initiates the project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

# 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 MDL, LOD, and/or LOQ as outlined in Alpha SOP/08-05. These studies performed by the laboratory are maintained on file for review.

## 13.2 Demonstration of Capability Studies

Refer to Alpha SOP/08-12 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/08-05 MDL/LOD/LOQ Generation

SOP/08-12 IDC/DOC Generation

SOP Waste Management and Disposal (G-006)

SOP Reagent, Solvent and Standard Control (G-008)

SOP Laboratory Glassware Cleaning (G-002)

## 16. Attachments

None.