# Silica Gel Cleanup Procedure

References: **EPA 3630C,** Test Methods for Evaluating Solid Waste, SW-846, Third Edition, Final Update III, December 1996 (USEPA, Office of Solid Waste and Emergency Response, Washington,

## 1. Scope and Application

**Matrices:** Solid samples for lipids, polymers, proteins, natural resins, cellular components, and fractionation of petroleum

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

This method is recommended for the cleanup of samples from lipids, polymers, proteins, natural resins, cellular components and fractionation of petroleum.

This method can be used to cleanup sample extracts to be analyzed for extractable petroleum hydrocarbons, polynuclear hydrocarbons, derivatized phenolic compounds, organochlorine pesticides, and polychlorinated biphenyls.

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 an experienced analyst. 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 extracted using the appropriate extraction protocol based on the matrix and required analysis parameters. The sample extract is concentrated to an appropriate final volume and vialed accordingly.

### 2.1 Method Modifications from Reference

None.

## 3. Reporting Limits

Refer to individual analytical SOPs.

# 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 *Reagent, Solvent and Standard Control* (G-008) and *Laboratory Glassware Cleaning* (G-002) SOPs for additional details.

**4.2** Many interferences can be removed by sample cleanup. Other cleanup methods performed include the following: *Alumina Column Cleanup of Organic Extracts* (OP-009), *Sulfur Cleanup* (OP-007), *Sulfuric Acid* (OP-010), *GPC Cleanup* (OP-006), and *Amino-propyl Cleanup of Tissues and Sediments* (OP-008). Only appropriate cleanup techniques must be performed based on the suspected interference and the compounds of interest. For example, sulfuric acid cleanup is not applicable to samples requiring pesticide analysis because this rigorous cleanup will destroy the majority of pesticides. Amino-propyl cleanup cannot be used when Endrin aldehyde is a compound of interest as this pesticide is lost during this cleanup.

**4.3** Soapy residue may result in basic conditions on glassware and may cause degradation of the pesticides Aldrin and Heptachlor, and some organophosphorous pesticides. All glassware must be rinsed thoroughly with deionized water and solvent to remove soapy residue. See 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, cleanup, 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.

**5.1** Approved PPE, which includes Safety Glasses, Gloves and Lab Coats, must be worn at ***all*** times when handling samples, reagents, chemicals, or when in the vicinity of others handling these items, so that dermal contact is avoided. All standards, reagents and solvents shall be handled under a hood using the proper PPE. All flammable solvents must be kept in the flammable storage cabinet, and returned to the cabinet immediately after use. When transporting chemicals, use a secure transporting devise and/or secondary outer container. Chemical storage is properly segregated and adequately ventilated to reduce the possibility of hazardous reactions. Chemical storage in work areas shall be kept to a minimum. Storage on bench tops or other work surfaces, except temporary, is not permitted.

**5.2** Spilled samples, solvents, reagents, and water must be cleaned up from bench tops, instruments and autosampler surfaces immediately. A spill is considered a quantity of hazardous material if it is two times greater than the normal working volume. Concentrated

solvents, acids or bases present a moderate to extreme hazard to the skin and mucous membranes. If contact with the skin occurs, immediately flush with large volumes of water. In the case of acidic/basic spills, the *Spill Kit* located in each laboratory shall be utilized before attempting to cleanup the spill. Although procedures are designed to minimize the possibility of an accident, all injuries or accidents, regardless of the nature or severity, are to be reported to the Department Supervisor immediately. If an employee discovers a potentially unsafe condition, this must be reported to the Department Supervisor immediately. No employee should feel compelled to work in a situation where they do not feel entirely informed, trained, or safe.

**5.3** Analytical instrumentation poses the unique possibility of exposure to high voltages. Other than the *routine* instrument maintenance, as listed in the front of every Instrument Maintenance Logbook, at no time shall an instrument operator attempt to maintenance an instrument alone, or without the proper training, supervision or instruction. Caution must always be used in the presence of moving parts (autosamplers) and hot surfaces (injection ports).

**5.4** Compressed gas cylinders shall only be moved with the dolly supplied for this specific purpose. The cap must be on the cylinder while it is being moved. The tank must be secured when in its final position. All spent tanks are to be returned in the same manner, and secured until removed by the vendor. Liquid argon or nitrogen represents a potential cryogenic hazard and safe-handling procedures must be used at all times.

# 6. Sample Collection, Preservation, Shipping and Handling

## 6.1 Sample Collection

Not applicable.

## 6.2 Sample Preservation

Not applicable.

## 6.3 Sample Shipping

Not applicable.

## 6.4 Sample Handling

Samples are stored at 4ºC +/- 2ºC. Extracts expire after 40 days.

# 7. Equipment and Supplies

**7.1 Syringes:** 1mL to 5mL

**7.2 Manifold with vacuum pump for silica cartridge cleanup**

**7.3 Dispensette:** 50mL volumetric cylinder to calibrate automatic pipette at 28mL

**7.4 VOA vials:** 40mL

**7.5 Squeeze bottles of solvent:** to condition cartridges

**7.6 Graduated Cylinder:** 50mL

**7.7 Teflon lined silica cartridge**

### 7.8 Glass Wool: Baked

## 8. Reagents and Standards

### 8.1 Methylene Chloride: Pesticide grade

### 8.2 Hexane: Pesticide grade

### 8.3 Silica Gel: 60-200 mesh. Baked at 110ºC for 24 hours.

### 8.4 Acetone: Pesticide grade

## 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

Not applicable.

### 9.2 Laboratory Control Sample (LCS)

Not applicable.

### 9.3 Initial Calibration Verification (ICV)

Not Applicable

### 9.4 Continuing Calibration Verification (CCV)

Not Applicable

### 9.5 Matrix Spike

Not applicable.

### 9.6 Laboratory Duplicate

Not applicable

### 9.7 Method-specific Quality Control Samples

None

### 9.8 Method Sequence

See Section 10.

## 10. Procedure

### 10.1 Equipment Set-up

#### 10.1.1 Assembling the silica cartridge for the manifold

**10.1.1.1** Put one 25 ml Teflon frit at the bottom of the Teflon coated silica cartridge. Pack down the frit with Teflon rod. Add 5.0g of silica to the cartridge. Continue for each cartridge.

**10.1.1.2 Assembling the silica for glass columns**

10.1.1.2.1 Take a silica glass column and rinse with Acetone and DCM. Cut a pea size piece of baked glass wool and push to the bottom with a DCM rinsed glass rod. Add 5.0 grams of baked silica gel (from a drying oven). Continue for each glass column.

**10.1.2 Condition the silica cartridges**

**10.1.2.1** Setup 6 cartridges on the manifold. Use disposable valve liners in the manifold and put the cartridge on top. Six rinsed vials are then put into the chamber to collect the dirty solvent. The cartridges and reservoirs are then filled with 60mls of methylene chloride, the stopper is opened and the solvent is allowed to draw into the cartridge until it dries. The stopper is then shut. The cartridges and reservoirs are then filled with 60mls of Hexane and the procedure is repeated. ***At this time the cartridge is NOT allowed to go completely dry***. Upon completion, 6 more cartridges are loaded onto the back 6 ports of the manifold and process repeats. Once all 12 cartridges are conditioned, you are ready to silica clean the samples.

**10.1.2.2 Condition for the silica glass columns**

Condition as above for Silica Cartridges in section 10.1.2.1.

## 10.2 Initial Calibration

Not Applicable

## 10.3 Equipment Operation and Sample Processing

10.3.1 Once the cartridges are conditioned (Section 10.1.1) a 50 mL graduated cylinder can be cleaned and filled with 28mL of methylene chloride. Insert a clean, labeled VOA vial into the collection slot in the manifold for each sample.

10.3.2 Next, transfer 1mL of sample into the first cartridge. Release the stop valve on the manifold and drain the sample down until it is even with the top layer of the silica, and then close it. Add 1mL of DCM into the same cartridge and allow to draw in until it is also even with the top of the silica. Now that the sample and the initial DCM are safely in the cartridge add 28mL of methylene chloride to the cartridge. The cartridge can now be allowed to drain into the VOA vial and cleanup is complete when the cartridge goes completely dry.

10.3.3 Repeat section 10.3.1 and 10.3.2 for the remaining samples. If the batch is larger than 12 samples, condition more cartridges as described in section 10.1.1.

10.3.4 It is critical that the silica cartridge does not go dry until the very last step. If the cartridge goes dry at any time during the process, a new cartridge needs to be setup and a new cleanup needs to be started for that sample. This is due to the fact that compounds of interest may be lost or the silica might not work correctly once it goes dry.

**10.3.5 Glass Column Procedure**

10.3.5.1 Once the glass column is conditioned (Section 10.1.2.2.1) take a 50ml graduated cylinder and add 28mls of DCM to the cylinder.

10.3.5.2 Next, transfer 1ml of sample the first glass column. Release the stop cock and drain the sample until it is even with the top of the silica and close the stopcock. Add 1ml of DCM into the same glass column and allow to draw in until it is even with the silica gel. Once done with that add your 28 mls of DCM to the column. The column can now be allowed the drain into the VOA vial and the cleanup is done when the column is dry thoroughly.

10.3.5.3 Repeat section 10.3.5.1 and 10.3.5.2 for all the remaining samples.

### 10.4 Continuing Calibration

Not Applicable

### 10.5 Preventive Maintenance

Not Applicable

## 11. Data Evaluation, Calculations and Reporting

**11.1** Procedures for data and record management for organic extraction must adhere to the Quality Systems Manual, other subordinate documents covering record keeping, and the *Document Control* SOP (G-016). All records must be stored in such a manner as to be safe and accessible for at least 10 years.

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

**11.3** See the appropriate analytical SOPs, for details on sample analysis, data evaluation, calculations and data reporting.

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

Not applicable.

## 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.

These studies are not applicable to this cleanup method.

### 13.2 Demonstration of Capability Studies

Refer to Alpha SOP/08-12 for further information regarding IDC/DOC Generation.

These studies are not applicable to this cleanup method.

#### 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/08-07 Control Limit Generation

SOP/08-12 IDC/DOC Generation

SOP/G-006 Hazardous Waste and Sample Disposal

SOP/G-016 Document Control

WI/108-01 Laboratory Notebook Usage

## 16. Attachments

None.