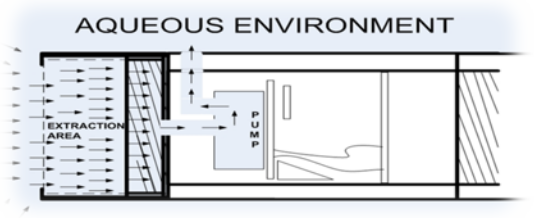
AQUALYTICAL

CLAM Laboratory & Application Notes

January 3, 2013

CLAM Principles of Operation

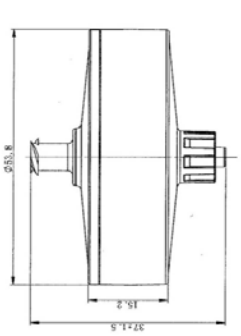


The CLAM is a small submersible extraction sampler, using EPA-approved methodology 3535, utilizing SPE (Solid Phase Extraction) media disks to provide a time integrative, large volume extraction event.

- The CLAM Disk is simply a **convenient holder** of standard laboratory SPE media disks and prefilters.
    - Designed to be easily eluted using the disk syringe Luer Lock System.
    - It provides a rugged disk **storage and shipping vesse**l which can be Luer Lock sealed for sample integrity.

- The water is simply left behind and the pre-extracted disk is submitted to the laboratory for simple solvent elution.
- Saves costs of shipping bottles of water and heavy shipping coolers.
- The sample has been extracted in the field saving the laboratory this time and expense.
- Holding time issues do not apply as the disk is a solid, and can be frozen for up to a year, stopping the hold time clock.
- The extract can be used for most EPA methods and other regulatory methodologies concerning fresh and marine waters.
- In-house IDC's and QA/QC can be performed on the bench, in the field, or both for acceptance criteria compliance.

CLAM Disk Material and Construction



All of our media disk holders are manufactured using high density polypropylene. This is the same material used to construct all of the disposable SPE extraction cartridges, disks and syringes used in the laboratory for over 15 years. The HDPP is resistant to organic, chlorinated and aromatic solvents as well as all acids and bases. The use of the HDPP has not showed evidence of **leaching** except for low levels of phthalates, or **partitioning** of analytes into or off its surface.

- The disks interior design uses HDPP media supporting screens, dispersion baffles, and glass pre-filtration filters to ensure an inert environment.

- The SPE media disks housed in our holders is the same high capacity disks and prefilters used routinely today in laboratories.

- The disks are "proofed" on a batch basis insuring they are free from leaching and produce analytical clean blanks, when properly cleaned and conditioned prior to use.

Media Selection

Solid Phase C-18 Extraction Disks

This media is well established and many EPA methods have incorporated its use, for Pesticides, PAH's, Semi-volatiles, PCB's, Dioxins, Furans, PBDE's and other HRMS methods. EPA1664 gravimetric Oil & Grease may even be run using this media disk.

Solid Phase HLB Extraction Disks

The polymeric HLB (Hydrophilic/Lipophilic Balanced) media in these disks can be used for drinking and waste water applications. Common applications include semi-volatile organic analysis and EPA Method 1694 (pharmaceuticals and personal care products), endocrine disruptors, and other emerging contaminants in wastewater and drinking water. They are self-wetting and will not lose functionality when they dry out.

Lofted Glass Pre-Filtration Disks

This filter assembly uses the triple lofted glass fiber filters to remove suspended sediment from the water. It allows for toxological studies for total and dissolved trace organics, when used in a two stage filter

Specialty Media: Mixed medias and specialized loadings can be arranged with a required minimum order. Some of these include:

- Atlantic™ 8270 One Pass Disk
- Atlantic™ DVB Disk

Cleaning and Conditioning for Deployment

The disks need to be cleaned, and conditioned prior to deployment. The steps involve forcing various solvents through the media disk inlet port, using a 50 ml glass syringe taken to waste. The washing and conditioning sequence are as follows:

1. **Cleaning Step;** 50 ml of DCM, 1 min. residence, followed by 2 syringe volumes of air.
2. **Conditioning Step;** 50ml of Methanol, 2 min. residence, followed by 1 syringe volume of air.
3. **Water Conditioning Step;** 50 ml of DI water, followed by 1 syringe volume of air.
4. If spiked standards are to be added continue to the spiking instruction section, if not, the disks are capped with the Luer Lock plugs provided, and placed in the metal foil pouch for shipment to the field.

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Surrogate and Spiking Instructions

The quality assurance plan or method will often determine if any and what type of spiking standards are to be added for field deployment, for elution or for both.

1. The **field** or **elution** spiking solutions should be made up in a water soluble solvent such as methanol or acetone at a concentration that would use no more the 100 ul of solution per spiking addition.
2. The disk is spiked directly by inserting the spiking syringe needle into the **inlet** and spiking the pre-filtration media.
3. The disk is then rinsed with 50 ml of DI water followed by 1 syringe volume of air, to set the spike onto the media.
4. The disk can then be capped, and is ready for field deployment.
5. If method blanks, trip blanks or laboratory spike/spike duplicates or matrix field spikes are to be used for method compliance, they will be spiked using the same procedure.
6. If the disk has been deployed, and elution standards such as; recovery surrogates, matrix spikes, internal standards, or isotopic dilution standards need to be added, the same procedure is performed prior to elution of the analytes.

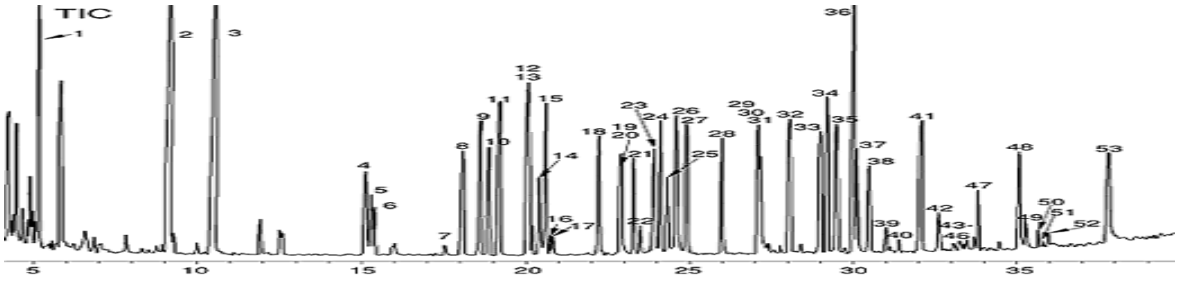
Elution of the CLAM SPE Disks

It is **essential to remove the water** within the SPE disk before the solvent elution. The SPE disk with its enhanced filtration media will retain 5ml or more of water even after vacuum drying. We have found the following method to be simple and effective in removing the water, allowing full media contact to the elution solvent. The procedure simply displaces the water in the disk with methanol, then elutes the disk with DCM. All the elution is collected and partitioned with DI water in a small separatory funnel. The funnel is lightly shaken allowed to settle removing the methanol from the DCM, which is then collected, concentrated and then analyzed (see steps below).

1. With a 50 ml glass syringe slowly elute 50 methanol through the disk into a 250- ml Separatory funnel.
2. The next step is to slowly pass 50 ml of DCM using the same syringe through the disk into the receiving separatory funnel, followed by an additional 25ml of DCM.
3. Add 100 ml DI water adjusted to pH 2 to the separatory funnel, swirl and shake lightly for 30 seconds, this step just removes the methanol from the DCM and partitions it into the water phase.

4. Allow to settle, then draw off the bottom DCM phase into a concentration vessel.
5. The steps are repeated by eluting an additional 50 ml of DCM through the extraction disk, and into the separatory funnel,
6. The total of the DCM extract can be concentrated by KD, nitrogen blow down, or by any other solvent reduction method to produce a final extract volume.
7. The volume of extracted solvent can be exchanged to fit the instrument and analysis type such as; hexane for ECD analysis, or DCM for GC/MS or GC/MS/MS analysis. The extract can then be dried, cleaned up and concentrated to the desired volume required for detection requirements.

CLAM Instrument Data Calculations



Instrument Result (ng/ml) x Solvent Vol. (ml) = Total weight present in (ng)
Total Weight (ng) / Total Volume Water Extracted (L) = Water Conc. (ng/L)

Example: Instrument raw data from ICAL curve = 4.036 ng/ml
Solvent Extract Vol.=2.0 ml
Total Volume Water Extracted=80 L

Calculation: 4.036 ng/ml x 2.0 ml = 8.1 ng

8.1 ng / 80L= Water concentration of 0.101 ng/l

Method Blank and Detection Level Considerations.

The EPA defines the MDL as the "minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. Laboratories determine method detection limits by running 7 replicated analysis, at the low point of their initial calibration, using standard sample deviation. These analysis are matrix, analyst, instrument and method specific.

A one liter volume is usually used to determine the MDL for trace organics in water analysis methods. Once these values are determined they are inserted into the laboratory calculation formula spread sheets or LIMS which will usually apply factors to calculate the sample concentrations which are based on sample volume used, and extract volume concentrations. The MDL and associated field and method blanks will be scaled accordingly for each sample, applying acceptance criteria and determining PQL and RL's and data qualifiers.

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