



Ensuring high quality, safe, productive workflows for Extraction of Soils and Sediments for Semi-Volatile Organics using the Milestone TOTAL WORKFLOW APPROACH

BACKGROUND

As regulations addressing target Semi-Volatile organics in soil matrices drive to lower and lower action levels, laboratories by necessity need to examine preparation workflows to optimize the rate-limiting steps in sample preparation. This, coupled with a focus on Safety and Health and the need for cost reductions provides impetus for the Milestone total workflow solution for sample preparations.

Extraction techniques such as sonication (not allowed for PCB as of Feb 2024), soxhlet, and pressurized fluid extraction (PFE) all present different challenges in terms of safety, quality, productivity, reliability, and cost. Milestone's Total Workflow solution incorporates solvent handling, sample extraction, and sample filtration while eliminating cross contamination and doing all with a small footprint to free up valuable hood space.

Samples are weighed, dried with drying agent, transferred to disposable glass inserts, treated with surrogates (and spikes where appropriate). The solvent system is automatically added to each vessel and transferred to a microwave vessel for extraction according to EPA SW-846 method 3546.

The components of the Milestone Total



Workflow Approach for Organics extractions of soils for Semi-Volatile Organics (BNA, Pesticides, PCBs, TPH-DRO, etc.) include three main components:

1. Automated Solvent addition

- Reduce Solvent Exposure
- Free up Hood space
- Consistent volumes added (+1% accuracy)
- Adaptable to any size vessels
- Inert design/no cross contamination
- Accommodates up to 6 solvent
- Software Driven/ease of use

2. Microwave Extraction

- 30-gram sample size better RLs
- 40 minutes from start to finish for 24 samples
- Disposable glass inserts-no cleaning or cross contamination
- Documentation of each sample extraction

3. Extract filtration

- Filter 24 extracts simultaneously
- Filter through sodium sulfate and 0.45 u filter

DISCUSSION

Most Laboratories spend considerable time and money on optimizing analysis techniques such as GC/MS and GC/ECD. More focus on the preparation processes is needed to reduce the bottlenecks in sample preparation such as extractions.

A focus on employee health and safety is an integral part of any workflow for organic extractions. The ability to reduce exposures to methylene chloride and other solvents is increasingly important as EPA and OSHA focus on methylene chloride use limitations.



Proposal Allows Critical Military, Some Manufacturing and Processing Uses with Strict

OSHA already has strict methylene chloride procedures for users including specific plans and monitoring requirements.

This focus, coupled with State regulations on methylene chloride emissions from fume hoods in laboratories, requires laboratories to reduce their methylene chloride footprint.

The Milestone TOTAL WORKFLOW approach helps laboratories reduce their solvent usage and associated risks, increases productivity, and processes samples more cost effectively and timely.

This workflow approach is depicted below:





EXPERIMENTAL - SOLVENT ADDITION

Our easyFILL system automatically adds solvents to your vessels, safely and accurately (+1%); our Microwave Extraction system safely and efficiently extracts your samples; our Sample Filtration System efficiently filters your extracts. When handling solvent systems for organic extractions, most labs are utilizing 1:1 methylene chloride:acetone or 1:1 hexane:acetone. The handling of these solvents presents a challenge due to the volumes that are necessary to add to any solvent extraction protocol. The Milestone easyFILL system safely and accurately (+1%) delivers solvents individually or as mixtures (0.5 to 50 mL) to any standard extraction vessel. The racks provided come in many configurations and can accommodate most common extraction vessels from all manufacturers. Solvent fumes are vented through a 4" OD exhaust hose into a fume hood.

Proven benefits are:

- Less handling time
- Enhanced safety
- Improved lab efficiency



Figure 2. easyFILL automatic reagent dosing station

EXPERIMENTAL - MICROWAVE EXTRACTION

Soil Extractions using Microwave technology provide a cost-effective, safe, and reliable technique for extractable organics by EPA SW-846 method 3546.

Milestone systems are employed worldwide at over 500 laboratories for organic extractions. The recoveries of BNA compounds, PCBs, Pesticides among others meet or exceed EPA method requirements (EPA 8000d and EPA 3546) and stand up to TNI/NELC and State assessments due to the ability to monitor temperature and pressure in each sample vessel throughout a run.

A Milestone ETHOS X equipped with a fastEX-24 rotor benchtop microwave extraction system, fully compliant with US EPA 3546, was used for these studies. The fastEX-24 rotor works uses 145 mL vessels with 100 mL disposable glass vial inserts. These vessels allow 30 gram of sample to be extracted to assist with your Reporting Limit needs.



Figure 3. ETHOS X with fastEX-24 rotor

Thanks to Milestone's Weflon (patented) technology and contactless temperature control in all positions, the fastEX-24



rotor allows simultaneous extraction of 24 samples ensuring temperature uniformity for all samples and excellent recovery of analytes.

Built-in methods and app notes provide an unmatched ease of use and low operating costs compared to all other extraction techniques currently approved.

All Application notes detailing the recoveries of BNA, PCBs, Pesticides and other extractables are available. Recoveries are summarized in the following table.

| TYPE OF ORGANIC | RECOVERY RANGE | PRECISION-% RSD |
|-----------------------|-------------------|-----------------|
| BNA -8270 list | 69 - 118% R | 4.0 - 14.9 |
| Pesticides- 8081 list | 79 - 120 %R | 1.3 – 10.6 |
| PCBs -8082 list | 75 – 115 %R | 0.2 - 8.3 |
| TPH-DR0 (C10-C28) | 89 – 105 %R | 2.4 – 5.2 |

These data indicate excellent recoveries and precision from both CRMs and Blank Spikes in soil matrices due to the design and workflow of the Milestone systems.



Figure 4. Contactless temperature control in each vessel and documentation of each vessel extraction profile downloadable as .xls, .cvs and .pdf for Batch back-up

Table 1. Recovery ranges by Analysis Group-reflects all typical compounds reported by Environmental Labs

GREATER RETURN ON INVESTMENT

The competitive nature of the environmental analysis market requires today's laboratories to have innovative solutions that provide faster turnaround times. With throughput capabilities of 24 samples in only 40 minutes, the ETHOS X with fastEX-24eT reduces overall analysis costs by increasing productivity. The combination of lower solvent volumes, less maintenance needs, and the use of inexpensive disposable glass vials decreases the cost per sample to enhance your lab's competitiveness and profitability.

| | Soxhlet | Sonication | Pressurized Liquid Extraction | ETHOS X |
|--------------------------------|----------|------------|----------------------------------|---------------------|
| Sample Size (g) | 10-30 | 30 | 10-20 | 2-30 |
| Solvent Volume per sample (mL) | 300-500 | 300-400 | 15-30 | 15-30 |
| Extraction time (min) | Days | Hours | 270 min - 24 samples | 40 min - 24 samples |
| Productivity (8 hours) | Low | Low | Moderate | Very high |
| Initial investment | Low | Low | High | Moderate |
| Cost per sample* | Moderate | Moderate | High | Low |

*Including: solvent - handling - time - productivity - maintenance

Table 2: Comparison of extraction techniques



EXPERIMENTAL – EXTRACT FILTRATION

The final step before extract concentration is the filtration and rinsing of extract disposable vial contents into an evaporation vessel (50 mL or 200 mL, or other appropriate size for your system). Some labs utilize N-EVAP™ systems while others utilize TurboVap™ systems. Very few labs still use Kuderna-Danish concentration techniques. In all cases, the total volume of solvent from the Microwave extraction and rinsing is less than 50 ml. This speeds up the concentration considerably and enhances process recoveries since the concentration is not allowed to proceed for an extended period. In cases where the solvent may need to be exchanged (e.g. to hexane from methylene chloride), the times aved by using lower volume microwave extraction shows time savings due to smaller volumes to exchange over. The Milestone SFS-24 automated filtration system simultaneously filters 24 extracts through anhydrous sodium sulfate and a 0.45 u filter (or other size to fit your need) into the concentration vessel. This operation is done in a hood and the filtration takes only a few minutes, including rinsing and is vacuum assisted. The reservoirs and filters are low cost consumables



Figure 5. SFA-24 automated filtration system

CONCLUSION

The Milestone Total Workflow approach to Extractions provides Laboratories significant advantages ranging from cost savings, performance metrics, risk management and other KPIs.



| TOTAL WORKFLOW SOLUTION: WHY DOES IT MATTER?

- Ability for your lab to do MORE with LESS

- Increased sample capacity for workload variations

- Ability to improve and predict Turnaround times and execute Rush Work

- Improved Data Quality

- Ease of use and workflow implementation make on-boarding new staff seamless

- Risk Avoidance for Laboratory Staff using the easyFILL Reagent dosing station and lower solvent volumes employed





MILESTONE Srl - Via Fatebenefratelli, 1/5 - 24010 Sorisole (BG) - Italy Tel: +39 035 573857 - Fax: +39 035 575498 www.milestonesrl.com - email: analytical@milestonesrl.com