

AUTOMATED SOLID PHASE EXTRACTION

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The EconoTrace Parallel SPE System is designed to streamline your laboratory's workflow and increase productivity by automating the manual steps in your sample preparation process. The EconoTrace Parallel SPE system automates existing manual SPE techniques and replaces older manual Liquid-Liquid Extraction techniques and outdated semi-automated instruments.

The EconoTrace Parallel SPE system is the only SPE system that combines extraction, drying and concentration into one step – providing a truly automated total sample prep solution for the laboratory. Simply load samples onto the EconoTrace Parallel SPE system to trigger the automated extraction process. After loading the sample onto the SPE cartridge at the set flow rate, the drying step is accomplished using Nitrogen. This drying step replaces manual techniques. The analytes of interest are then eluted directly to the SuperVap Concentrator where the concentration process automatically brings the extract to final volume and places it directly into an autosampler vial, ready for final analysis. Automating these processes into one step ensures the highest quality results in the shortest amount of time and eliminates both human error and the possibility of contamination.



The EconoTrace 3 system runs six samples in parallel and provides direct-to-vial concentration.

Reduces Errors

One-step automated SPE and concentration eliminates human error, saves labor costs and reduces solvent usage while increasing your sample throughput.

Fully Automated

Hyphenates the entire sample prep process—extraction, drying and concentration steps into a one process.

Runs up to eight samples simultaneously.

Automatic sample bottle rinse.

Concentrates samples up to 250 mL directly to a GC vial.

High Speed

The fastest automated sample processing available for SPE cartridges and columns of all sizes.

Runs up to eight samples simultaneously.

Positive pressure pumping for fast, simultaneous loading of samples.

Versatile

Handles a wide range of sample sizes and all matrix types.

Sample sizes from 2 mL to 8 L.

Expandable from one to four modules—two samples per module.

Efficient

Uses all SPE cartridge and column sizes.

Positive pressure pumping for loading samples.

Nitrogen drying.

Compliant

Complies with existing methods that require positive pressure pumping for the precise delivery of sample and solvents.

Dispenses up to six solvents using an HPLC pump to deliver precise volumes and flow rates for sample loading, conditioning and elution.

Easy Documentation

Programs and stores an unlimited number of methods and runs on an SD Card.

Easy method transfer

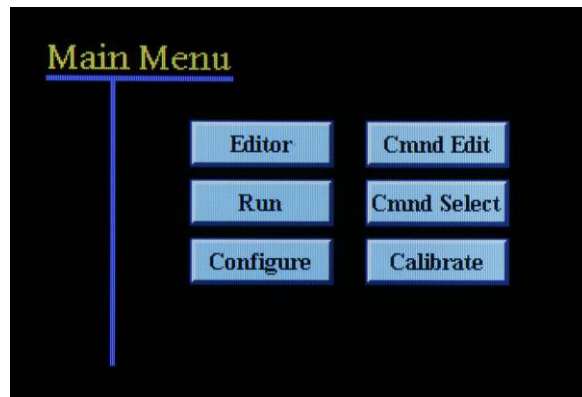
Easy-to-use touch screen.

The EconoTrace Parallel SPE system uses positive pressure pumping for precise and accurate delivery of the sample as well as conditioning, washing and elution solvents. The system is specifically designed to isolate analytes of interest from a wide variety of liquid matrices such as urine, blood, water, milk, beverages. Sample sizes range from 2 mL to 8 L and use the same bottle the sample was collected in. Sample loading rates are programmable. A positive pressure pump is used to load samples onto any SPE cartridge and columns available on the market and it easily handles both clean and tough sample matrices.

The EconoTrace Parallel SPE/SuperVap system concentrates samples up to 250 mL directly to a GC vial. For guaranteed results, especially when dealing with low limits of detection, we recommend using FMS cartridges. Sample Processing Modules can easily be swapped out for service which means your lab will experience zero downtime.



The EconoTrace System is expandable from one to four modules



System control is accomplished via an easy-to-use touch screen.

The SuperVap™ Concentrator is where the concentration process automatically brings the extract to final volume in an autosampler vial, ready for final analysis.



Automatic time-based or endpoint detection for nitrogen shut off for each vessel

Measurements - Concentration / Evaporation vessels in 500 µL, 1 mL, and direct to a GC Vial or to dryness

Specifications

EconoTrace™ Parallel SPE System

| | |
|-------------------|---------------------------------|
| Dimensions: | 15" W x 18" D x 26" H |
| Weight: | 30lbs. |
| Gas Requirements: | Nitrogen - 20 PSI minimum |
| Pump: | Piston Displacement |
| Flow rate: | 0.2 to 15 mL/minute |
| Electrical Input: | 110/220 Volts, 50/60 HZ |
| Controller: | Integrated Touch Screen Control |

SuperVap™ Concentration System

| | |
|-------------------|---------------------------------|
| Dimensions: | 12" W x 13" D x 12" H |
| Weight: | 20 lbs. |
| Gas Requirements: | Nitrogen - 20 PSI minimum |
| Electrical Input: | 110/220 Volts, 50/60 HZ |
| Controller: | Integrated Touch Screen Control |
| Bath: | Dry |

Ordering Information

| Part Number | Description |
|----------------|---|
| EconoTrace / 1 | EconoTrace 2 for running 2 samples simultaneously |
| EconoTrace / 2 | EconoTrace 4 for running 4 samples simultaneously |
| EconoTrace / 3 | EconoTrace 6 for running 6 samples simultaneously |
| EconoTrace / 4 | EconoTrace 8 for running 8 samples simultaneously |

Consumables

| Part Number | Description |
|--------------------------|---|
| SPE-BT1 | SPE Sample Bottle 1L |
| SPE-BTRC | SPE Sample Bottle Rinse Cap |
| SPE-ADP-1 | SPE 1 mL Cartridge Adapter |
| SPE-ADP-3 | SPE 3 mL Cartridge Adapter |
| SPE-ADP-6 | SPE 6 mL Cartridge Adapter |
| SPE-ADP-20 | SPE 20 mL Cartridge Adapter |
| SPE -ADP-35 | SPE 35 mL Cartridge Adapter |
| SPE-PRE-20 | SPE Pre-filter cartridge |
| SPE-CAR1-C ₁₈ | SPE Cartridge 1 gram C ₁₈ |
| SPE-CAR5-C ₁₈ | SPE Cartridge 5 gram C ₁₈ |
| SPE-CAR1-DVB | SPE Cartridge 1 gram DVB |
| SPE-CAR5-DVB | SPE Cartridge 5 gram DVB |
| TFE-TUB-071GR | Teflon Tubing Assembly Flanged any color, 7" |
| TFE-TUB-091GR | Teflon Tubing Assembly Flanged any color, 9" |
| TFE-TUB-161GR | Teflon Tubing Assembly Flanged any color, 16" |
| TFE-TUB-241GR | Teflon Tubing Assembly Flanged any color, 24" |
| PVAP-TUB-200M | Evaporator Glass Tubes |
| PVAP-TUB-200M | GC Evaporator Glass Tubes Standard -GC |
| PVAP-UNI-TF | Teflon GC Vial union |

The TurboTrace Parallel SPE system is designed to streamline your laboratory's workflow and increase productivity by automating the manual steps in your sample preparation process. It is designed for high throughput and tough sample matrices. The TurboTrace Parallel SPE system automates existing manual SPE techniques and replaces older manual Liquid-Liquid Extraction techniques and outdated semi-automated instruments.

The TurboTrace Parallel SPE system combines extraction, drying and concentration into one step – providing a truly automated total sample prep solution for the laboratory. Simply load samples onto the TurboTrace Parallel SPE system then use the touch screen to trigger the automated extraction process. After loading the sample onto the SPE cartridge, the drying step is accomplished using vacuum, nitrogen or both. This drying step replaces manual techniques such as sodium sulfate drying. The analytes of interest are then eluted directly to the SuperVap™ Concentrator where the concentration process automatically brings the extract to final volume in an autosampler vial, ready for final analysis. Automating these processes into one step ensures the highest quality results in the shortest amount of time and eliminates both human error and the possibility of contamination.



The TurboTrace 3 system runs three samples in parallel while providing direct-to-vial concentration.

Reduces Errors

One-step automated SPE and concentration eliminates human error, saves labor costs and reduces solvent usage while increasing your sample throughput.

Fully Automated

Hyphenates the entire sample prep process—extraction, drying and concentration steps—into a one step program.

Runs up to 8 samples simultaneously.

Sample Liquid Level Sensor to detect when the sample has finished loading.

Automatic sample bottle rinse.

Concentrates samples up to 250 mL directly to a GC vial.

High Speed

The fastest automated sample processing available for SPE cartridges and columns.

Vacuum for fast loading of large volume samples.

Versatile

Handles a wide range of sample sizes and all matrix types.

Sample sizes 2 mL to many liters.

Expandable from one to eight modules.

Efficient

Uses all available SPE cartridge and column sizes.

Positive pressure pumping for loading small volume samples.

Nitrogen drying

Compliant

Complies with existing methods that require vacuum, positive pressure pumping for the precise delivery of sample and solvents.

Dispenses up to six solvents using an HPLC pump to deliver precise volumes and flow rates for conditioning and elution.

Easy Documentation

Programs and stores an unlimited number of methods and runs on an SD Card Easy method transfer.

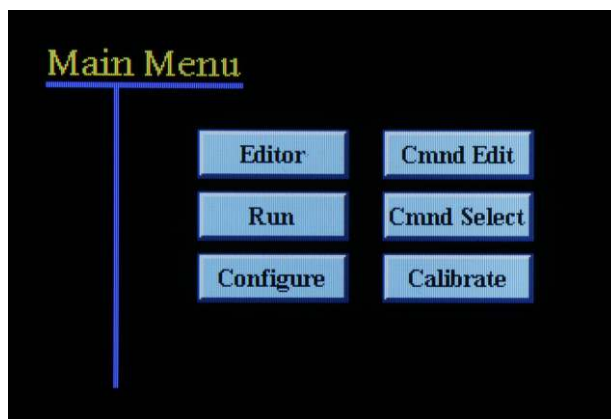
Easy-to-use software

The TurboTrace™ Parallel SPE system incorporates vacuum or positive pressure pump to load samples for compliance with all SPE methods. It uses positive pressure pumping for precise and accurate delivery of conditioning, washing and elution solvents. The TurboTrace Parallel SPE system is specifically designed to isolate analytes of interest from a wide variety of liquid matrices such as urine, blood, water, milk, beverages. Sample sizes range from 2 mL to 8 L and use the same bottle the sample was collected in. Sample loading rates are programmable. Samples are loaded onto the SPE cartridges and columns at unprecedented speeds and the system easily handles both clean and tough sample matrices. A liquid sensor detects when the sample has been loaded, triggering the system to initiate next steps.

The TurboTrace Parallel SPE system concentrates samples up to 250 mL directly to a GC vial. The Sample Processing Module is designed to use all standard formats of SPE cartridges and columns on the market today. For guaranteed results, especially when dealing with low limits of detection, we recommend using FMS prepacked columns and cartridges. Sample Processing Modules can be easily swapped out to perform cleanup column chemistries allowing the lab to use the TurboTrace Parallel SPE system to automate other sample prep processes.



The TurboTrace System is expandable from one to eight modules.



System control is accomplished via an easy-to-use touch screen.

The SuperVap™ Concentrator is where the concentration process automatically brings the extract to final volume in an autosampler vial, ready for final analysis.



Automatic time-based or endpoint detection for nitrogen shut off for each vessel

Measurements - Concentration / Evaporation vessels in 500 µL, 1 mL, and direct to a GC Vial or to dryness

Specifications

TurboTrace™ Parallel SPE System

| | |
|-------------------|---------------------------------|
| Dimensions: | 15" W x 18" D x 26" H |
| Weight: | 30lbs. |
| Gas Requirements: | Nitrogen - 20 PSI minimum |
| Pump: | Piston Displacement |
| Flow rate: | 0.2 to 15 mL/minute |
| Electrical Input: | 110/220 Volts, 50/60 HZ |
| Controller: | Integrated Touch Screen Control |

SuperVap™ Concentration System

| | |
|-------------------|---------------------------------|
| Dimensions: | 12" W x 13" D x 12" H |
| Weight: | 20 lbs. |
| Gas Requirements: | Nitrogen - 20 PSI minimum |
| Electrical Input: | 110/220 Volts, 50/60 HZ |
| Controller: | Integrated Touch Screen Control |
| Bath: | Dry |

Ordering Information

| Part Number | Description |
|----------------|--|
| TurboTrace / 1 | TurboTrace 1 for running one sample |
| TurboTrace / 2 | TurboTrace 2 for running two samples |
| TurboTrace / 3 | TurboTrace 3 for running three samples |
| TurboTrace / 4 | TurboTrace 4 for running four samples |
| TurboTrace / 5 | TurboTrace 5 for running five samples |
| TurboTrace / 6 | TurboTrace 6 for running six samples |
| TurboTrace / 7 | TurboTrace 7 for running seven samples |
| TurboTrace / 8 | TurboTrace 8 for running eight samples |

Consumables

| Part Number | Description |
|--------------------------|---|
| SPE-BT1 | SPE Sample Bottle 1L |
| SPE-BTRC | SPE Sample Bottle Rinse Cap |
| SPE-ADP-1 | SPE 1 mL Cartridge Adapter |
| SPE-ADP-3 | SPE 3 mL Cartridge Adapter |
| SPE-ADP-6 | SPE 6 mL Cartridge Adapter |
| SPE-ADP-20 | SPE 20 mL Cartridge Adapter |
| SPE -ADP-35 | SPE 35 mL Cartridge Adapter |
| SPE-PRE-20 | SPE Pre-filter cartridge |
| SPE-CAR1-C ₁₈ | SPE Cartridge 1 gram C ₁₈ |
| SPE-CAR5-C ₁₈ | SPE Cartridge 5 gram C ₁₈ |
| SPE-CAR1-DVB | SPE Cartridge 1 gram DVB |
| SPE-CAR5-DVB | SPE Cartridge 5 gram DVB |
| TFE-TUB-071GR | Teflon Tubing Assembly Flanged any color, 7" |
| TFE-TUB-091GR | Teflon Tubing Assembly Flanged any color, 9" |
| TFE-TUB-161GR | Teflon Tubing Assembly Flanged any color, 16" |
| TFE-TUB-241GR | Teflon Tubing Assembly Flanged any color, 24" |
| PVAP-TUB-200M | Evaporator Glass Tubes |
| PVAP-TUB-200M | GC Evaporator Glass Tubes Standard -GC |
| PVAP-UNI-TF | Teflon GC Vial union |

NanoTrace™

Low volume Solid Phase Extraction system

Solid Phase Extraction is a powerful technique to separate analytes of interest from matrix interferences in the sample prior to analysis. When performing manual SPE it is difficult to control the sample loading flow rate as well as the drying step, making it difficult to obtain consistent quality and reproducible results.

The quality of manual SPE largely depends on the experience of user and how carefully each step is performed. This is the main reason that the recoveries and precision of manual SPE can be subject to wide range of deviation. To improve the recoveries and precision in SPE process, FMS introduces the NanoTrace automated Solid Phase Extraction system.

The NanoTrace™ is designed to automate the Solid Phase Extraction process, which includes sample loading, cartridge washing, drying and elution step with superior accuracy. The NanoTrace™ is programmable and optimizes recoveries and precision for all SPE applications. The system is modular and expandable. Each module can be loaded with 5 samples and up to 8 modules can be connected together and controlled through the NanoTrace™ controller.

The NanoTrace™ support variety of cartridge and column sizes. User friendly and easy to use editor allows simple programming of flow rate, volume and gives ability to select up to 6 solvents with the ability to deliver up to 4 fractions.



The NanoTrace 3 system runs 8 modules in parallel and each module perform 5 samples sequentially and provides direct-to-vial concentration.

Reduces Errors

One-step automated SPE and concentration eliminates human error, saves labor costs and reduces solvent usage while increasing your sample throughput.

Fully Automated

Hyphenates the entire sample prep process- extraction, drying and concentration steps into a one process.

Increases sample throughput

Automates SPE applications, increases productivity and sample throughput processing available for SPE cartridges and columns of all sizes.

Flow rate and volume programmability

Programmable Positive pressure pumping. Flow rate .02 to 5 ml/min

Versatile

Handles a wide range of sample sizes and all matrix types.

Modular & Expandable

Expandable from one to eight modules for parallel processing each module perform five samples simultaneously.

Efficient

Uses all SPE cartridge and column sizes. Positive pressure pumping for loading samples. Nitrogen drying.

Compliant

Complies with existing methods that require positive pressure pumping for the precise delivery of sample and solvents.

Dispenses up to six solvents using an HPLC pump to deliver precise volumes and flow rates for sample loading, conditioning and elution.

Easy Documentation

Programs and stores an unlimited number of methods and runs on an SD Card

Easy method transfer

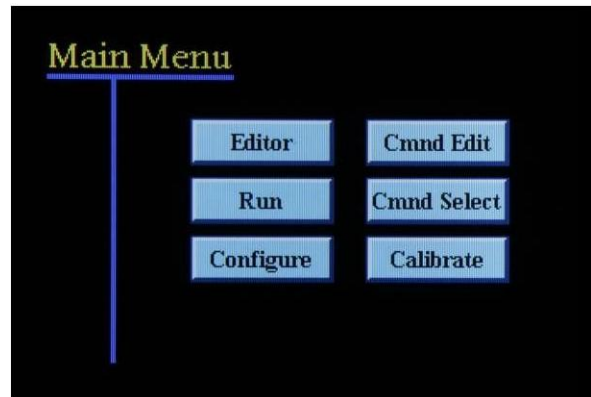
Easy-to-use touch screen

The NanoTrace SPE system uses positive pressure pumping for precise and accurate delivery of the sample as well as conditioning, washing and elution solvents. The system is specifically designed to isolate analytes of interest from a wide variety of liquid matrices such as urine, blood, water, milk, beverages. Small Sample sizes from 0.1 ml can be loaded using positive pressure pump to load samples onto any SPE cartridge.

The NanoTrace SPE system interfaces to the SuperVap concentration system to concentrate extracts directly to a GC vial. For guaranteed results, especially when dealing with low limits of detection, we recommend using FMS cartridges. Sample Processing Modules can easily be swapped out for service which means your lab will experience zero downtime.



The EconoTrace System is expandable from one to four modules



System control is accomplished via an easy-to-use touch screen.

Direct to Vial Concentration



Automated Solid Phase Extraction of EPA 525.2, Semi-Volatile Organic Compounds in Drinking Water.



Introduction

EPA Method 525.2 outlines the procedure for the extraction and analysis of a wide range of organic compounds in water. The extraction method outlines the use of solid phase extraction for water matrix samples employing both cartridges and disks. Consistent with other EPA 500 series methods, EPA 525.2 incorporates a rigid set of QC and acceptance criteria requiring precise and reproducible analytical practices. The potential for error and the variability associated with manual extractions makes the benefits of automating these processes apparent.

The following procedure was developed to demonstrate the benefits of automating EPA Method 525.2 using Waters Oasis[®] HLB cartridges, the FMS SPE and the FMS SuperVap[™] Direct-to-Vial Concentration system. This one step method delivers rapid, consistent and reproducible extraction and concentration of water samples for analysis by GC/MS.

Instrumentation and Consumables

- FMS, Inc. SPE System
- FMS, Inc. SuperVap[™] Concentrator
- FMS, direct to vial concentrator tubes
- Thermo Certified GC/MS auto sampler vials
- 1 gram Waters Oasis[®] HLB Cartridge
- Restek 2 gram Sodium sulfate Cartridge
- Thermo Trace GC w/DSQ MS and AS3000 Autosampler

Reagents

- Fisher Optima^{*} Methanol
- Fisher Optima^{*} Methylene Chloride
- Fisher Optima^{*} Ethyl Acetate
- Fisher HPLC Water
- Fisher Concentrated Sulfuric Acid
- Ultra Scientific Calibration/Spiking solutions (SVM-525, PPM-525E, NPM-525C, NPM-525B, NPM-108B, ISM-510)

Procedure:

Sample Prep

Five, one liter samples are measured out in glass sample collection bottles

Each sample is spiked with 1 mL of 525 spiking solution (dilute of Ultra Scientific standards, 5 ug/mL majority).

Sample PH adjusted to <2 with 1:1 sulfuric acid solution

10 mL Methanol added to each sample bottle

FMS SPE system

1. The HLB cartridges are conditioned with 10 mL Methanol
2. The cartridges are conditioned with 10 mL DI H₂O
3. The samples are loaded across the cartridges via vacuum (~75 mL/min)
4. Sample bottles are automatically rinsed with DI water and the rinse is loaded onto cartridge
5. The cartridges are dried with Nitrogen for 2 minutes
6. The cartridges are eluted with 10 mL of ethyl acetate
7. The cartridges are eluted with 10 mL methylene chloride
8. The cartridges are purged with a Nitrogen push

SuperVap Concentrator system

1. Pre-heat temp: 45 °C
2. Pre-heat time: 20 minutes
3. Heat in Sensor mode: 45 °C
4. Nitrogen Pressure: 10 PSI
5. End point: 1mL

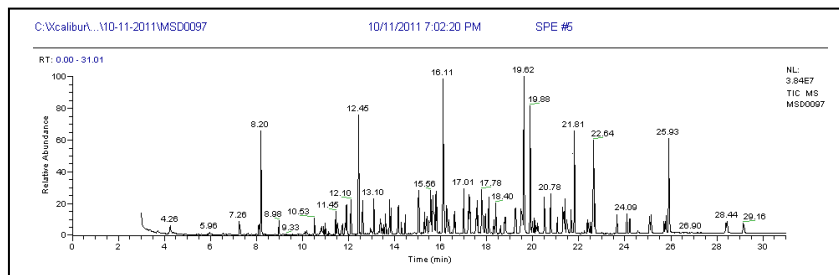


Figure 1: PowerPrep™ SPE and PowerVap™ Concentrator systems.

| Compound | Spike Conc | Mean Rec | STD DEV | | | | |
|--|------------|----------|---------|--------------------------------------|---------|--------|-------|
| Terbufos | 5 µg/L | 107.8% | 4.6% | Diazinon | 5 ug/L | 97.4% | 3.5% |
| Chrysene-d12 | 25 µg/L | 91.1% | 16.8% | Pebulate | 5 ug/L | 106.4% | 6.0% |
| Anthracene | 5 µg/L | 95.1% | 2.3% | Cyanazine | 5 ug/L | 100.7% | 6.2% |
| 2,2',4,4',5,6'-hexachlorobiphenyl | 5 µg/L | 99.7% | 13.4% | 2,2',3,3',4,4',6-heptachlorobiphenyl | 5 ug/L | 103.4% | 15.2% |
| 2-chlorobiphenyl | 5 µg/L | 101.9% | 2.6% | Indeno[1,2,3-cd]pyrene | 5 ug/L | 115.4% | 9.3% |
| Ethoprop | 5 µg/L | 120.2% | 5.3% | bis(2-ethylhexyl)phthalate | 5 ug/L | 109.1% | 15.5% |
| Tricyclazole | 5 µg/L | 138.7% | 9.1% | Isophorone | 5 ug/L | 108.4% | 4.0% |
| DDD | 5 µg/L | 109.0% | 10.1% | Disulfoton | 5 ug/L | 91.0% | 5.5% |
| Cis-Permethrin | 5 µg/L | 98.6% | 9.1% | Mevinphos | 5 ug/L | 109.3% | 6.8% |
| Benzo[a]pyrene | 5 µg/L | 100.8% | 11.3% | Pyrene-d10 | 25 ug/L | 101.0% | 14.4% |
| Propochlor | 5 µg/L | 99.3% | 4.5% | Tebuthiuron | 5 ug/L | 126.1% | 8.6% |
| Pronamide | 5 µg/L | 118.4% | 4.3% | Trifluralin | 5 ug/L | 107.7% | 3.4% |
| Terbutryn | 5 µg/L | 89.6% | 3.1% | Ametryn | 5 ug/L | 122.7% | 5.2% |
| Triademefon | 5 µg/L | 101.6% | 3.7% | Bromacil | 5 ug/L | 115.7% | 1.9% |
| DEF | 50 µg/L | 70.4% | 12.1% | Fenarimol | 5 ug/L | 124.4% | 15.9% |
| DCPA | 5 µg/L | 114.7% | 6.2% | Heptachlor Epoxide | 5 ug/L | 111.9% | 5.6% |
| DDE | 5 µg/L | 101.1% | 10.6% | Trans-nanochlor | 5 ug/L | 97.3% | 7.3% |
| Acenaphthene-d10 | 25 µg/L | 90.1% | 11.5% | Benzo[a]anthracene | 5 ug/L | 99.7% | 11.1% |
| Pyrene | 5 µg/L | 105.7% | 11.9% | Atrazine | 5 ug/L | 120.1% | 7.7% |
| 2,4-dinitrotoluene | 5 µg/L | 110.2% | 7.3% | Trerbacil | 5 ug/L | 113.0% | 13.4% |
| Butylate | 5 µg/L | 119.5% | 5.0% | Etridiazole | 5 ug/L | 108.5% | 4.2% |
| Dieldrin | 5 µg/L | 101.1% | 11.1% | Alpha-BHC | 5 ug/L | 95.9% | 2.4% |
| Perylene-d12 | 25 µg/L | 105.9% | 12.4% | Beta-BHC | 5 ug/L | 105.5% | 5.2% |
| Dibenzo[a,h]anthracene | 5 µg/L | 117.9% | 9.8% | Delta-BHC | 5 ug/L | 102.2% | 5.2% |
| Fenamiphos | 5 µg/L | 109.4% | 7.1% | Alpha Chlordane | 5 ug/L | 100.6% | 10.3% |
| Alachlor | 5 µg/L | 106.8% | 8.6% | Endrin | 5 ug/L | 120.2% | 17.9% |
| Norflurazon | 5 µg/L | 127.7% | 14.5% | Methoxychlor | 5 ug/L | 99.8% | 15.8% |
| Simizine | 5 µg/L | 123.0% | 5.0% | Acenaphthylene | 5 ug/L | 92.6% | 13.0% |
| Phenanthrene-d10 | 25 µg/L | 96.8% | 11.2% | Pentachlorophenol | 5 ug/L | 108.2% | 4.3% |
| Benzo[b]fluoranthene | 5 µg/L | 109.7% | 12.5% | 2,6-dinitrotoluene | 5 ug/L | 111.5% | 7.4% |
| Chrysene | 5 µg/L | 110.3% | 3.1% | Hexachlorobenene | 5 ug/L | 90.9% | 2.8% |
| 2,2',3,3',4,5',6,6'-octachlorobiphenyl | 5 µg/L | 99.2% | 13.5% | Atraton | 5 ug/L | 119.2% | 2.5% |
| Chlorpyrifos | 5 µg/L | 101.6% | 7.7% | Propazine | 5 ug/L | 123.2% | 3.6% |
| Endrin_Aldehyde | 5 µg/L | 112.4% | 22.5% | Napropamide | 5 ug/L | 110.4% | 10.1% |
| Phenanthrene | 5 µg/L | 104.8% | 3.3% | Endosulfan I | 5 ug/L | 121.4% | 4.1% |
| bis(2-ethylhexyl)adipate | 5 µg/L | 106.1% | 8.1% | Benzo[k]fluoranthene | 5 ug/L | 114.9% | 12.5% |
| Diethylphthalate | 5 µg/L | 112.4% | 5.3% | Butylbenzylphthalate | 5 ug/L | 125.3% | 14.0% |
| hexachlorocyclopentadiene | 5 µg/L | 73.7% | 7.2% | 2,4,5-trichlorobiphenyl | 5 ug/L | 104.0% | 2.6% |
| | | | | Dimethylphthalate | 5 ug/L | 105.5% | 6.6% |
| | | | | EPTC | 5 ug/L | 109.4% | 5.9% |



| | | | | | | | |
|---------------------------------|---------|--------|-------|-----------------------------|---------|--------|-------|
| Chloropham | 5 µg/L | 120.6% | 6.7% | Prometryn | 5 µg/L | 122.8% | 6.1% |
| Metolachlor | 5 µg/L | 112.0% | 4.3% | Fluridone | 5 µg/L | 144.4% | 11.5% |
| Stirofos | 5 µg/L | 111.9% | 7.0% | Endosulfan Sulfate | 5 µg/L | 116.7% | 16.1% |
| Aldrin | 5 µg/L | 102.3% | 7.0% | 1-bromo-2-nitrobenzene | 5 µg/L | 91.4% | 6.1% |
| Trans-Permethrin | 5 µg/L | 107.8% | 11.5% | 1,3-dimethyl-2-nitrobenzene | 25 µg/L | 102.8% | 2.7% |
| 2,2',3',4,6-pentachlorobiphenyl | 5 µg/L | 107.1% | 9.3% | Benzo[g,h,i]perylene | 5 µg/L | 117.2% | 9.5% |
| 2,2',4,4'-tetrachlorobiphenyl | 5 µg/L | 97.5% | 5.1% | Di-n-butylphthalate | 5 µg/L | 116.2% | 8.6% |
| Molinate | 5 µg/L | 110.0% | 6.1% | Vernolate | 5 µg/L | 114.0% | 6.1% |
| Cycloate | 5 µg/L | 114.4% | 3.3% | Diphenamid | 5 µg/L | 106.5% | 6.0% |
| Simetryn | 5 µg/L | 123.8% | 4.7% | Dichlorovos | 5 µg/L | 116.8% | 6.6% |
| MGK (B) | 5 µg/L | 120.5% | 5.0% | Heptachlor | 5 µg/L | 97.9% | 6.0% |
| Butachlor | 5 µg/L | 124.9% | 3.6% | Chlorobenzilate | 5 µg/L | 124.6% | 9.1% |
| Chlorothalonil | 5 µg/L | 109.1% | 4.3% | Choroneb | 5 µg/L | 114.9% | 5.0% |
| MGK (A) | 5 µg/L | 120.3% | 0.6% | Prometon | 5 µg/L | 119.1% | 3.6% |
| Gamma-Chlordane | 5 µg/L | 104.8% | 8.2% | Hexazinone | 5 µg/L | 122.4% | 13.1% |
| Triphenylphosphate | 25 µg/L | 119.0% | 10.1% | Gamma-BHC | 5 µg/L | 116.6% | 5.1% |
| Fluorene | 5 µg/L | 108.9% | 6.3% | Endosulfan II | 5 µg/L | 119.0% | 15.9% |
| 2,3-dichlorobiphenyl | 5 µg/L | 98.1% | 1.1% | DDT | 5 µg/L | 93.4% | 11.2% |
| Carboxin | 5 µg/L | 75.0% | 9.0% | | | | |
| Methyl Paraoxon | 5 µg/L | 118.6% | 3.4% | | | | |



Chromatogram of an extracted 525 sample on the DSQ

Conclusions

Reviewing the sample data shows high recoveries for over 100 spiked analytes, demonstrating excellent efficiency for all classes of compounds. Low deviations between runs shows good run-to-run reproducibility. The FMS SPE and SuperVap™ system are suitable for the full automation from sample to vial of EPA Method 525.2. Samples can be taken from collection bottle to GC vial in one quick, consistent, reproducible process that will save laboratories both time and money.

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EPA 522; Analysis of 1,4-Dioxane Drinking Water by Solid Phase Extraction (SPE)



Introduction

EPA 522 defines a laboratory protocol for the extraction for analysis of 1,4-Dioxane (CASRN 123-91-1) in drinking water. The method uses solid phase extraction paired with GC/MS analysis. The usage of selective ion monitoring (SIM) is often utilized to achieve minimum detection levels.

Due to the relatively high volatility of 1,4-Dioxane, the use of a 2 gram coconut charcoal cartridge is required for the retention of 1,4-dioxane from aqueous samples. Cartridges are then eluted with a small portion of methylene chloride, and evaporated to 1 mL. The procedure requires a slow sample loading process where low rates and times must be precise and consistent. By implementing an automated SPE system like the TurboTrace™ System, samples can be extracted in a fully automated, precise, process that frees up extraction chemists to perform other tasks.

Instrumentation and Consumables

Instrumentation

- FMS, Inc. TurboTrace (Solid Phase Extraction) System
- FMS, Inc. SuperVap™ 12-Position Concentrator
- FMS, Inc. direct-to-vial concentrator tubes
- Thermo Trace Ultra GC with SSL Injection Port
- Thermo DSQ Mass Spectrometer

Consumables

- Restek 2g Coconut Charcoal column (Cat# 26032)
- Restek 1,4-dioxane-d8 Standard (Cat# 30614)
- Restek 1,4-dioxane Standard (Cat# 30287)
- Restek Tetrahydrofuran-D8 Standard (Cat# 30112)
- Fisher Optima Grade Methanol
- Fisher Optima Grade Methylene Chloride
- Fisher HPLC Grade Water
- Fisher Anhydrous Sodium Sulfate

Procedure

TurboTrace SPE

1. cartridges conditioned with 10 mL MeOH
2. cartridges conditioned with 10 mL H₂O
3. samples loaded across Cartridges via vacuum (5-10 mL/min)
4. cartridges dried under nitrogen for 5 minutes.
5. sample bottle sprayed with 20 mL methylene chloride.
6. 0 mL methylene chloride spray loaded across cartridge and collected.
7. cartridges eluted with 5 mL methylene chloride
8. eluted solvent nitrogen purged into collection vials.

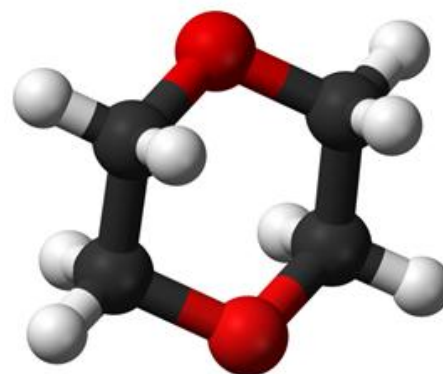


Figure 1: Chemical structure of 1,4-dioxane



Results

Table 1: Results for .5 liter replicates spiked at 1 µg/L

| Replicate | 1,4-dioxane-d8 recovery | 1,4-dioxane conc. (ug/L) | STD DEV |
|-----------|-------------------------|--------------------------|---------|
| 1 | 89.59% | 1.056 | |
| 2 | 84.59% | 0.962 | 0.05 |
| 3 | 82.15% | 1.03 | |



Figure 2: FMS TurboTrace SPE system with the SuperVap concentrator.

Conclusions

Extraction method performance of the FMS TurboTrace system and SuperVap Concentrator resulted in all replicates yielding 1,4-dioxane-d8 recoveries within the 70-130 percent recovery window. Native 1,4-dioxane calculated concentration showed excellent precision and accuracy. The usage of independent sample loading sensors to stop sample loading of individual runs was a critical component to ensuring that no analyte was lost from over drying the cartridges.

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EPA 529; Explosives in Drinking Water by Solid Phase Extraction (SPE)



Introduction

EPA method 529 details the procedure for the extraction and analysis of explosives in finished drinking water. The method uses solid phase extraction of water samples to partition analytes of interest from aqueous samples, and analyzes them by GC/MS analysis.

The method calls for the extraction of 1 liter samples by Solid Phase Extraction (SPE) using a DVB cartridge or disk. Extracts are eluted with Ethyl Acetate and run on a GC/MS system. The use of a PTV injection system is required to optimize the loading of thermally labile compounds such as RDX.

The following details the use of the Fluid Management Systems, Inc TurboTrace™ SPE extraction system to fully automate the extraction procedure contained in EPA 529.

Instrumentation and Consumables

Instrumentation

- FMS, Inc. TurboTrace™ Solid Phase Extraction System
- FMS, Inc. SuperVap™ 12 Position Concentrator
- FMS, direct-to-vial concentrator tubes
- Thermo Trace Ultra GC with PTV Injection Port
- Thermo Polaris Q Mass Spectrometer

Consumables

- FMS DVB Cartridge (1 gram)
- Fisher Optima Grade Methanol
- Fisher Optima Grade Ethyl Acetate
- Fisher HPLC Grade Water
- Fisher Anhydrous Sodium Sulfate

Procedure

TurboTrace SPE System Program

1. Cartridges conditioned with 10 mL MeOH
2. Cartridges conditioned with 10 mL H₂O
3. Samples loaded across cartridges via vacuum
4. Cartridges dried under vacuum for 5 minutes.
5. Sample bottle sprayed with 20 mL Ethyl Acetate.
6. 20 mL Ethyl Acetate spray loaded across cartridge and collected.
7. Cartridges eluted with 5 mL Ethyl Acetate
8. Eluted solvent nitrogen purged directly into FMS SuperVap for concentration**.
9. Total Time ~30 minutes

**Removal of residual water from extract by in-line sodium sulfate filtration.

SuperVap Concentrator

1. Preheat temp: 20 minutes at 45 °C
2. Evap mode w/Sensor temp: 45 °C
3. Nitrogen Pressure: 10 PSI



Figure 1; FMS, Inc TurboTrace SPE system with the SuperVap concentrator.



Results

Table 1; Results for 1 liter replicates spiked at 1 µg/L.

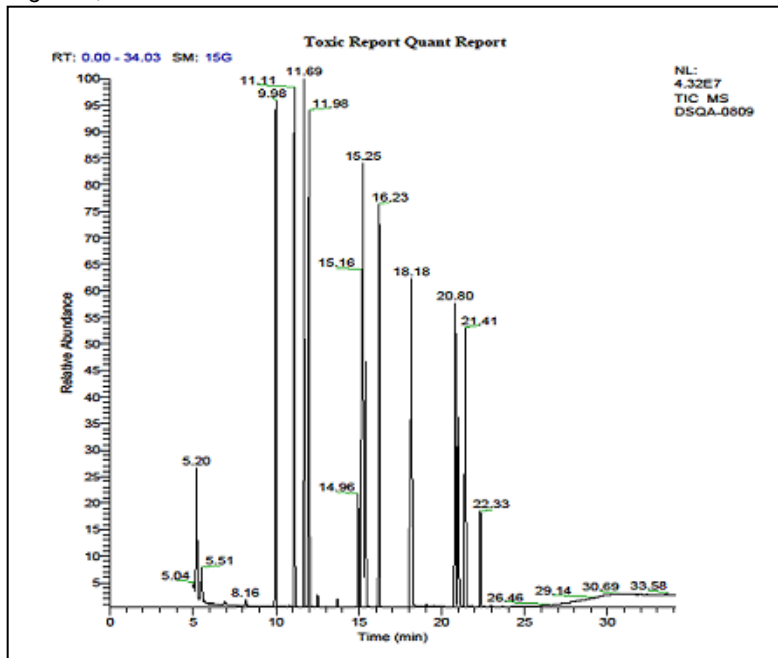
| Compound | Mean Rec. | STD DEV |
|----------------------------|-----------|---------|
| Nitrobenzene | 97.1% | 3.0% |
| 2-Nitrotoluene | 98.8% | 3.8% |
| 3-Nitrotoluene | 99.3% | 3.0% |
| 4-Nitrotoluene | 101.3% | 5.5% |
| 1,3-Dinitrobenzene | 106.8% | 1.5% |
| 2,6-Dinitrotoluene | 95.6% | 2.5% |
| 2,4-Dinitrotoluene | 90.5% | 2.8% |
| 1,3,5-Trinitrobenzene | 80.9% | 2.0% |
| 2,4,6-Trinitrotoluene | 77.4% | 2.5% |
| RDX | 104.6% | 4.7% |
| 4-Amino-4,6-dinitrotoluene | 91.9% | 2.0% |
| 3,5-Dinitroaniline | 88.4% | 1.5% |
| 2-Amino-4,6-dinitrotoluene | 94.8% | 2.4% |
| Tetryl | 77.5% | 5.7% |

Conclusions

The final analysis of the sample replicates yielded recoveries well within the 70-130% limits defined in the method for all analytes. Variation between samples replicates resulted in single digit deviations demonstrating excellent reproducibility.

Totally automated Sample Preparation produce consistent, accurate results and make the FMS TurboTrace SPE and SuperVap Concentration system an ideal solution for drinking water labs currently performing EPA 529 extractions manually..

Figure 2; TIC for EPA 529 LCS



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EPA Method 548.1, Endothall in Drinking Water



Introduction

Endothall is a white, colorless organic solid used primarily as a defoliant and an herbicide. It is widely used to control invasive aquatic exotics like Eurasian milfoil. Regulated by the EPA under the Safe Drinking Water Act, it is classified as a Class II toxin that can cause liver and kidney damage with prolonged exposure, and it can also affect organs of the digestive system.

This application note follows EPA method 548.1 using ion exchange Solid Phase Extraction Cartridges to extract endothall from a water matrix. Traditionally, a lengthy manual process with many conditioning steps is required to use ion exchange cartridges. The following demonstrates how the FMS PowerPrep™ SPE system can automate the conditioning, extraction and elution of samples in a single turnkey process.

Instrumentation & Consumables

- FMS, Inc. PowerPrep™ SPE system
- FMS, Inc. SuperVap™ Concentrator
- FMS, concentrator tubes
- Thermo Trace GC Ultra
- Thermo Polaris Q Mass Spec
- 500 mg Waters Oasis™ Max cartridges
- Fisher Pesticide Grade Methanol
- Fisher Pesticide Grade Methylene Chloride
- Fisher Sulfuric Acid
- Fisher Sodium Hydroxide Pellets
- Fisher Anhydrous Sodium Sulfate

SPE Program

1. Condition Cartridge with Methylene Chloride
2. Condition Cartridge with Methanol
3. Condition Cartridge with DI Water
4. Condition Cartridge with 10% H₂SO₄/MeOH
5. Condition Cartridge with DI Water
6. Condition Cartridge with 1N NaOH
7. Condition Cartridge with DI Water
8. Load Sample (100 mL)
9. Rinse bottle with DI Water and load rinse
10. Rinse Cartridge with Methanol
11. Air dry Cartridge (5 min)
12. Elute Cartridge with 10% H₂SO₄/MeOH
13. Elute Cartridge with Methylene Chloride
14. Nitrogen purge fraction lines

SuperVap Concentrator

1. Pre-heat temp: 40 °C
2. Pre-heat time: 20 minutes
3. Heat in Sensor mode: 40 °C
4. Nitrogen Pressure: 15 PSI



Figure 1: PowerPrep SPE and SuperVap Concentrator systems.



Derivatization /Partition

Following extraction, sample derivatization was conducted following section 11.4 of EPA method 548.1. Extracts were spiked with internal standard following the partition step, and then returned to FMS SuperVap Concentrator equipped with direct-to-GC vial tubes. Following the above parameters, extracts were taken to a 1 mL final volume using the FMS SuperVap Concentrator with direct-to-GC vial tubes and transferred to GC/MS for analysis.

Conclusions

Analysis of sample replicates at the LFB level (Table 1) resulted in excellent recoveries with minimal deviation between runs. Duplicate runs spiked at the method detection limit (Table 2) also displayed precision and reproducibility exceeding manual extraction performance. The conclusion of this study is that the FMS PowerPrep SPE system delivering the extract directly to the SuperVap Concentrator served as an excellent platform for the fully automating EPA method 548.1 delivering consistent and reproducible results.

Table 1. Shows the recoveries and standard deviations of samples extracted and analyzed.

| Replicates | Endothall Concentration | Percent Recovery |
|------------|-------------------------|------------------|
| Sample #1 | 47.6 µg/mL | 95.2% |
| Sample #2 | 49.3 µg/mL | 98.6% |
| Sample #3 | 48 µg/mL | 96.0% |
| | Std Deviation | 2.7% |

Table 2. Shows duplicate analysis of sample spiked at the Method RL.

| Replicates | Endothall Concentration | Percent Recovery |
|--------------|-------------------------|------------------|
| RL Sample #1 | 5.18 µg/mL | 103.6% |
| RL Sample #2 | 5.55 µg/mL | 111.0% |
| | RPD | 4.5% |

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Polycyclic Aromatic Hydrocarbons in Drinking Water by Solid Phase Extraction (SPE)



Introduction

PAHs are hazardous compounds found in petroleum and emissions from fossil fuel consumption. Physically, PAHs are neutral, nonpolar organic molecules that comprise two or more benzene rings arranged in various configurations. Members of this class of compounds exhibit toxic and hazardous properties, and for this reason the U.S. Environmental Protection Agency has included 16 PAHs on its list of priority pollutants to monitor in water and waste.

Analytically, PAHs present challenges for testing labs. Their volatile nature can make them problematic to extract without loss using manual methods. This is especially true for the evaporation/concentration of sample extracts.

The following procedure utilizes the FMS PowerPrep™ SPE extraction system in combination with the SuperVap™ concentrator (Figure 1) to extract PAHs from drinking water.

Instrumentation

- FMS, Inc. PowerPrep SPE (Solid Phase Extraction) System
- FMS, Inc. SuperVap Concentrator
- FMS, direct-to-vial concentrator tubes
- Thermo Trace Ultra GC with DSQ MS

Consumables

- Waters Oasis® HLB (1 gram)
- Fisher Pesticide Grade Methanol
- Fisher Pesticide Grade Methylene Chloride
- Fisher HPLC Grade Water
- Fisher Anhydrous Sodium Sulfate

Procedure

PowerPrep SPE

1. Cartridges conditioned with 10 mL MeOH
2. Cartridges conditioned with 15 mL H₂O
3. Samples loaded across cartridges at ~75 mL/min
4. Sample bottles rinsed with H₂O; Rinse loaded across cartridges.
5. Cartridges dried with N₂ for 1 minute each.
6. Cartridges eluted with 10 mL Methylene Chloride.
7. Cartridges eluted with 5 mL Methylene Chloride.
8. Elution nitrogen purged directly into FMS SuperVap for concentration.

Total Time ~55 minutes

SuperVap Concentrator

1. Preheat temp: 20 minutes at 40 °C
2. Sensor mode / temp: 40 °C
3. Nitrogen Pressure: 10 PSI



Figure 1: PowerPrep SPE and SuperVap Concentrator systems.



Results

Table 1: Results for five, 1 liter replicates spiked at 50 ppb.

| Compound | Mean Rec. | STD DEV |
|-------------------------|------------------|----------------|
| Naphthalene | 86.6% | 4.6% |
| 2-Methylnaphthalene | 88.2% | 3.3% |
| 1-Methylnaphthalene | 83.4% | 4.3% |
| Acenaphthylene | 81.7% | 4.8% |
| Acenaphthene | 92.3% | 6.8% |
| Fluorene | 79.4% | 5.1% |
| Phenanthrene | 87.0% | 3.0% |
| Anthracene | 81.4% | 2.8% |
| Fluoranthene | 93.5% | 2.4% |
| Pyrene | 100.8% | 3.2% |
| Benzo[a]anthracene | 88.3% | 5.1% |
| Chrysene | 93.6% | 6.6% |
| Benzo[b]fluoranthene | 85.5% | 7.0% |
| Benzo[k]fluoranthene | 91.8% | 3.9% |
| Benzo[a]pyrene | 90.5% | 5.1% |
| Indeno[1,2,3-cd]pyrene | 88.9% | 4.0% |
| Dibenzo[a,h]anthracene | 92.3% | 5.2% |
| Benzo[g,h,i]perylene | 88.1% | 3.7% |
| Nitrobenzene-D5 (Surr) | 93.6% | 6.1% |
| 2-Fluorobiphenyl (Surr) | 96.4% | 4.9% |
| p-Terphenyl-d14 (surr) | 81.7% | 5.2% |

Conclusions

Results reported from the PowerPrep™ SPE and SuperVap™ Direct-to-Vial Concentration system (Table 1) show high levels of precision and accuracy for the parallel extraction and concentration of PAHs using the FMS integrated Sample-to-Vial systems. With expanded modules, the PowerPrep SPE system enables the lab to simultaneously extract full batches of sample (up to 30) hands free. Compared to manual extraction processes, this automated technique frees up chemists to focus on increasing sample throughput. The addition of direct to GC vial concentration tubes eliminates the need to manually transfer extract from the concentration tubes to the vials and reduces operator error.

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Dioxin in Drinking Water by One-Step Solid Phase Extraction



Introduction

Dioxins are a group of polyhalogenated compounds which occur as by-products in the manufacture of organochlorides, in the incineration of chlorine-containing substances such as PVC (polyvinyl chloride), in the bleaching of paper, and from natural sources such as volcanoes and forest fires. Aside from being known carcinogens, dioxins and dioxin like compounds have been linked to developmental abnormalities, nervous system, pathology, diabetes, and thyroid disorders as well as damage to the immune system. For these reasons, it is becoming increasingly important to determine dioxin free drinking water and food sources. This procedure demonstrates the use of the FMS SPE system in water samples.

Instrumentation and Consumables

- FMS, Inc. PowerPrep™ SPE system (Solid Phase Extraction)
- FMS, Inc. SuperVap™ Concentrator system
- Waters Oasis® 1 gram HLB Cartridge
- Thermo Fisher Scientific DFS HRGC/MS

Method Summary

PowerPrep SPE

1. Condition Cartridge with MeOH
2. Condition Cartridge with HPLC grade H₂O
3. Load 1 liter of water sample
4. Rinse container and load rinse volume
5. Dry Cartridge
6. Elute sample from Cartridge (DCM)

SuperVap Concentrator

1. Pre-heat temp: 70 °C
2. Pre-heat time: 15 minutes
3. Heat in Sensor mode: 70 °C
4. Nitrogen Pressure: 15 PSI

Procedure

Four, 1 liter water samples were spiked with 100 µL of Cambridge Isotope EDF-4144B Dioxin/Difuran spiking solution. Samples were then loaded onto the FMS SPE system using a vacuum pump and passed across a Waters Oasis HLB cartridge. After loading, samples were then dried using a stream of nitrogen until no residual water was present, and the cartridges were subsequently eluted using methylene chloride. Extracts were eluted directly into the FMS SuperVap Concentrator, spiked with 2 µL dodecane (keeper) and blown to dryness in direct-to-vial tubes. Samples were then re-constituted to 20 µL nonane and injected onto a high-resolution GC/MS system for quantitative analysis.



Figure 1: PowerPrep SPE and SuperVap Concentrator systems.



Results

| Compound | Amount Spiked ng/L | Mean Recovered ng/L | Percent |
|----------------------------------|--------------------|---------------------|----------|
| 2,3,7,8-TCDD (13C12,99%) | 0.250 | 0.1702 | 68.09333 |
| 1,2,3,7,8-PeCDD (13C12,99%) | 0.250 | 0.2474 | 98.95333 |
| 1,2,3,4,7,8-HxCDD (13C12,99%) | 0.600 | 0.6294 | 104.9067 |
| 1,2,3,6,7,8-HxCDD (13C12,99%) | 0.600 | 0.7649 | 127.49 |
| 1,2,3,7,8,9-HxCDD (13C12,99%) | 0.600 | 0.6103 | 101.7233 |
| 1,2,3,4,6,7,8-HpCDD (13C12,99%) | 0.600 | 0.4244 | 70.72667 |
| 1,2,3,4,6,7,9-HpCDD (13C12,99%) | 0.600 | 0.4244 | 70.73 |
| OCDD (13C12,99%) | 1.250 | 0.8123 | 64.98667 |
| 2,3,7,8-TCDF (13C12,99%) | 0.250 | 0.1897 | 75.89333 |
| 1,2,3,7,8-PeCDF (13C12,99%) | 0.250 | 0.2479 | 99.16333 |
| 2,3,4,7,8-PeCDF (13C12,99%) | 0.250 | 0.2136 | 85.44333 |
| 1,2,3,4,7,8-HxCDF (13C12,99%) | 0.625 | 0.6456 | 103.29 |
| 1,2,3,6,7,8-HxCDF (13C12,99%) | 0.625 | 0.6226 | 99.61333 |
| 1,2,3,7,8,9-HxCDF (13C12,99%) | 0.625 | 0.6243 | 99.88 |
| 2,3,4,6,7,8-HxCDF (13C12,99%) | 0.625 | 0.6641 | 106.2533 |
| 1,2,3,4,6,7,8-HpCDF (13C12,99%) | 0.625 | 0.5961 | 95.38333 |
| 1,2,3,4,6,7,9-HpCDF (13C12,99%) | 0.625 | 0.5961 | 95.37333 |
| OCDF (13C12,99%) | 1.250 | 0.7940 | 63.52333 |
| 3,3',4,4'-TCB (13C12, 99%) | 0.250 | 0.2318 | 92.72667 |
| 3,4,4',5'-TCB (13C12, 99%) | 0.250 | 0.2250 | 89.99 |
| 3,3',4,4',5'-PeCB (13C12, 99%) | 0.360 | 0.5509 | 153.02 |
| 3,3',4,4',5,5'-HxCB (13C12, 99%) | 0.480 | 0.8006 | 166.7933 |

Conclusions

The FMS PowerPrep SPE system produces reliable, reproducible results for low level dioxin and dioxin like compounds in drinking water. The use of direct-to-vial concentration tubes eliminates the need for sample transfer of very low volume extracts and the risk of extract loss and contamination.

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Automated One Step Solid Phase Extraction and Concentration of PCBs in Drinking Water



Introduction

PCBs are a group of synthetic organic chemicals that contain 209 individual compounds (known as congeners) with varying harmful effects. PCBs enter the environment in mixtures containing a variety of individual components. Seven types of PCB mixtures include 35% of all the PCBs commercially produced and 98% of PCBs sold throughout the world. PCBs don't burn easily and are good insulating materials. PCBs have been widely used as coolants and lubricants in transformers, capacitors and other electrical equipment. The manufacture of PCBs was stopped due to evidence of the harm they cause when they build up in the environment. Because of their durability and wide-spread industrial use, PCBs have found their way into drinking water supplies.

The PowerPrep™ SPE and SuperVap™ Concentrator systems speed up the sample preparation process for the analysis of PCBs by combining sample prep into a single, automated step.

Instrumentation & Consumables

- FMS, Inc. PowerPrep SPE (Solid Phase Extraction) System
- Waters Oasis® 1 gram HLB SPE cartridge
- FMS, Inc. SuperVap Concentrator
- Thermo Fisher Scientific Polaris Q GCMS

Method Summary

PowerPrep SPE system

1. Condition Cartridge: 10 mL MeOH
2. Condition Cartridge: 10 mL H₂O
3. Load Sample: 15 minutes
4. Rinse bottle: 5 seconds
5. Load rinse: 1 minute
6. Dry Cartridge: 30 minutes
7. Elute Sample: 20 mL DCM

SuperVap Concentrator

1. Pre-heat temp: 55 °C
2. Pre-heat time: 30 minutes
3. Heat in Sensor mode: 65 °C
4. Nitrogen Pressure: 15 PSI

Procedure

Five, 1 liter water samples were each spiked with a mixture of 19 individual PCB congeners at 1 µg/mL each. Samples were also spiked with a 1 µg/mL tetrachloro-m-xylene solution as an extraction surrogate. Using the PowerPrep SPE system, samples were then loaded on pre-wet Oasis HLB cartridges using vacuum to draw the samples across the cartridge. Sample bottles were then automatically rinsed with DI water, after which the rinse was loaded onto the cartridge. Cartridges were dried using a nitrogen stream blown across the cartridge to remove all remaining water (30 minutes). Once dried, the HLB cartridges were eluted with 20 mL of DCM, allowing the cartridge to soak wetted with DCM for 1 minute. DCM was then purged from the cartridge, directly to the FMS SuperVap Concentrator with direct-to-vial tubes (Figure 2). Extracts were blown down to 1 mL final volume using FMS direct-to-vial concentrator tubes. Extracts were then transferred for GC/MS analysis.



Fig. 1: PowerPrep SPE and SuperVap Concentrator systems.



Fig. 2. FMS SuperVap™ Concentrator with direct-to-vial concentrator tubes.

Results

| Congener | Spiked µg/mL | Mean Recovery µg/L | % Rec | STD Dev. |
|----------|--------------|--------------------|--------|----------|
| BZ #1 | 1 | .911 | 91.1% | 0.13152 |
| BZ #5 | 1 | .916 | 91.6% | 0.16226 |
| BZ #18 | 1 | .903 | 90.3% | 0.02089 |
| BZ #31 | 1 | .905 | 90.5% | 0.02668 |
| BZ #44 | 1 | .898 | 89.8% | 0.02869 |
| BZ #52 | 1 | .897 | 89.7% | 0.02518 |
| BZ #66 | 1 | .907 | 90.7% | 0.03209 |
| BZ #87 | 1 | .913 | 91.3% | 0.03405 |
| BZ #101 | 1 | .905 | 90.5% | 0.032893 |
| BZ #110 | 1 | .916 | 91.6% | 0.03250 |
| BZ #138 | 1 | .900 | 90.0% | 0.03206 |
| BZ #141 | 1 | 1.005 | 106.0% | 0.03216 |
| BZ #151 | 1 | .895 | 89.5% | 0.03165 |
| BZ #153 | 1 | .907 | 90.7% | 0.31756 |
| BZ #170 | 1 | .896 | 89.6% | 0.03248 |
| BZ #180 | 1 | .915 | 91.5% | 0.03151 |
| BZ #183 | 1 | .924 | 92.4% | 0.03215 |
| BZ #187 | 1 | .914 | 91.4% | 0.03139 |
| BZ #206 | 1 | .884 | 88.4% | 0.03529 |
| TCMX | 1 | .865 | 86.5% | 0.02589 |

Conclusions

The results demonstrate that the FMS PowerPrep SPE system combined with the FMS SuperVap Concentrator and Waters Oasis cartridges can rapidly and accurately extract PCB samples that produce reproducible recoveries from water samples.

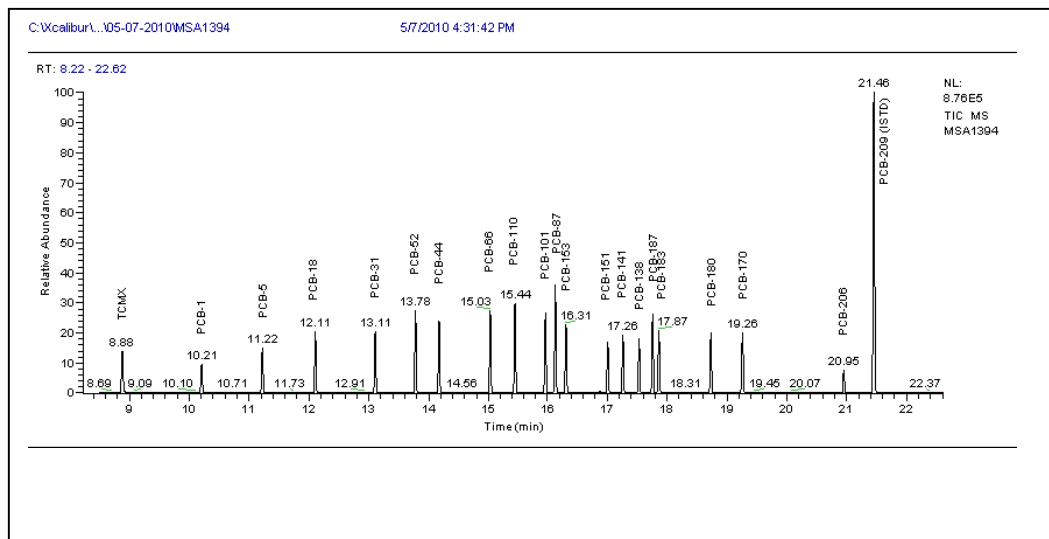


Fig. 3. Results of PCB congeners in sample extract.

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Automated Solid Phase Extraction (SPE) of Organochlorine Pesticides in Water



Introduction

Organochlorine pesticides are man-made organic chemicals with a history of wide spread use in both the United States and globally. Since they tend to persist in the environment, they have found their way into sediments and drinking water supplies posing serious health risks. Organochlorines have a wide range of both acute and chronic health effects, including cancer, neurological damage, and birth defects. Many organochlorines are also suspected endocrine disruptors.

In response to growing health concerns, the United States has banned several of these compounds such as DDT, dieldrin and chlordane. Others are still in use including lindane, endosulfan and methoxychlor.

Liquid/liquid extraction via a sep-funnel is the traditional method used for the extraction of organochlorine pesticides. Not only is it time consuming, it also requires a large volume of chlorinated solvent. The result is high cost and low reproducibility. The automated, solid phase extraction method described below allows for rapid, reproducible extractions using a minimal volume of solvent that produce consistent results.

Instrumentation

- FMS, Inc. PowerPrep™ SPE (Solid Phase Extraction) System
- FMS, Inc. SuperVap™ Concentrator
- FMS, direct-to-vial concentrator tubes
- 1 gram C₁₈ cartridges
- Agilent 7890A GC with uECD

PowerPrep SPE

1. The C₁₈ Cartridge is conditioned with 10 mL methanol
2. The C₁₈ Cartridge is conditioned with 10mL DI H₂O
3. The sample is loaded onto the C₁₈ Cartridge via vacuum
4. The sample bottle auto rinsed loaded on to the C₁₈ cartridge
5. The C₁₈ cartridge is dried with nitrogen
6. Elute with methylene chloride

SuperVap Concentrator

1. Pre-heat temp: 65 °C
2. Pre-heat time: 30 minutes
3. Heat in Sensor mode: 65 °C
4. Nitrogen Pressure: 15 PSI

Procedure: Sample Prep and Extraction

1. Five, 1 liter water samples spiked with 1 mL EPA 8081 surrogate spiking solution (2 analytes)
2. Samples were spiked with EPA 8081 pesticide spiking solution (20 analytes)
3. Samples allowed to equilibrate for 15 minutes
4. Five samples were loaded onto corresponding sample ports on FMS PowerPrep SPE System.
5. The program is initiated to run each sample sequentially.
6. The sample is extracted and automatically transferred to the FMS SuperVap Concentrator with direct-to-vial vessels.
7. The Extracts are concentrated using the SuperVap system to 1 mL, exchanged to Hexane (15 mLs) and re-evaporated to 1 mL.
8. The Extract is removed from the SuperVap system and transferred to Agilent GC for analysis.



Figure 1: PowerPrep SPE and SuperVap Concentrator systems.



Results

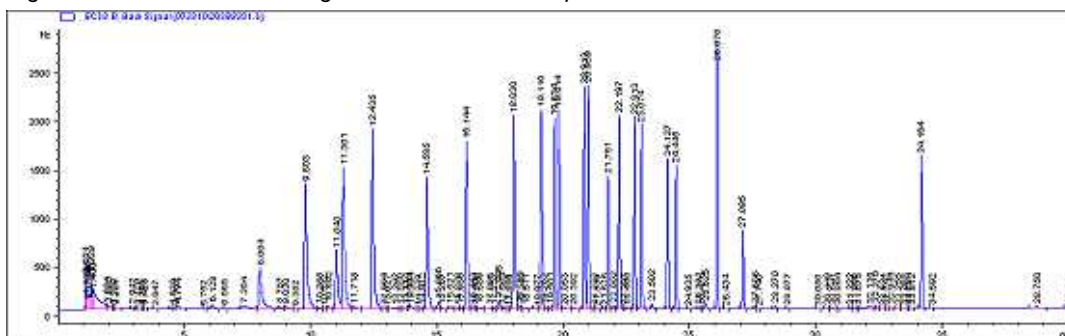
Table 1: Mean recovery and Standard deviation for five replicates

| Compound | Spike Conc. | Avg Rec. | STD Dev. |
|--------------------|-------------|----------|----------|
| TCMX | .1 ug/L | 70.0% | 5.1% |
| Alpha-BHC | .1 ug/L | 81.6% | 2.0% |
| Beta-BHC | .1 ug/L | 93.9% | 4.7% |
| Gamma-BHC | .1 ug/L | 83.1% | 4.7% |
| Delta-BHC | .1 ug/L | 98.9% | 5.9% |
| Heptachlor | .1 ug/L | 82.5% | 5.0% |
| Aldrin | .1 ug/L | 80.0% | 4.5% |
| Heptachlor Epoxide | .1 ug/L | 89.8% | 5.2% |
| Gamma-Chlordane | .1 ug/L | 81.0% | 4.6% |
| Endosulfan I | .1 ug/L | 87.8% | 4.7% |
| Alpha-Chlordane | .1 ug/L | 82.9% | 4.5% |
| Dieldrin | .1 ug/L | 85.9% | 4.7% |
| 4,4'-DDE | .1 ug/L | 84.0% | 4.7% |
| Endrin | .1 ug/L | 70.6% | 5.3% |
| Endosulfan II | .1 ug/L | 90.5% | 4.8% |
| 4,4'-DDD | .1 ug/L | 81.7% | 5.1% |
| Endrin Aldehyde | .1 ug/L | 119.1% | 5.9% |
| Endosulfan Sulfate | .1 ug/L | 95.0% | 5.1% |
| 4,4'-DDT | .1 ug/L | 96.2% | 6.4% |
| Endrin Ketone | .1 ug/L | 110.9% | 5.8% |
| Methoxychlor | .1 ug/L | 92.5% | 6.1% |
| Deca-PCB | .1 ug/L | 77.3% | 4.1% |

Conclusions

The results of five water samples demonstrate the ability of the FMS PowerPrep SPE system to deliver accurate results with excellent reproducibility. The automated SPE Direct-to-Vial Concentration method described is superior to traditional, time-consuming, inconsistent and expensive liquid/liquid extractions. The addition of the FMS SuperVap system equipped with direct-to-vial tubes enables the transfer of samples directly from sample bottles to GC vials in a single extraction process without handling the extract allows the extract to go directly to the GC for analysis.

Figure 2. Shows the chromatogram of a 1L water sample run on uECD detector



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One-Step Extraction and Concentration for Identifying Pharmaceuticals and Personal Care Products in Water, Biosolids and Solids



Introduction

Over the last decade the use of Pharmaceuticals and Personal Care Products (PPCPs) has doubled in the United States. As a result, PPCPs have entered the environment through both human activity and as by-products from manufacturing, agricultural activities, medical use and veterinarian facilities. PPCPs are usually introduced into the environment through the disposal of unused medications into sewer systems and trash. PPCPs tend to be water soluble and do not evaporate under normal temperatures, which is why they end up in soil and water. The full effect of PPCPs on the environment is not fully understood and there is concern about the potential threat they pose to the food chain. Because of the high solubility of most PPCPs, aquatic organisms are most vulnerable. The classes of pharmaceuticals found in these organisms have been linked to slow growth in frogs and the increased feminization of exposed fish. The scope of human exposure to PPCPs from the environment is complicated and increased monitoring is occurring to determine the effect on humans of long-term, low-level exposure to PPCPs.

Due to their persistent nature and toxicity, monitoring water sources for PPCPs is a growing priority for both government agencies and consumers. The following procedure outlines the fully automated, sample-to-vial extraction and concentration of water matrices for the detection of these compounds in one rapid and efficient process.

Instrumentation and Consumables

FMS, Inc. PowerPrep™ SPE system (Solid Phase Extraction)

FMS, Inc. SuperVap™ Concentrator

FMS, Direct-to-Vial concentrator tubes

1 gram Waters Oasis™ HLB cartridge

UPLC, LC/MS.

Procedure: Sample Prep Extraction and Concentration PowerPrep SPE

1. Condition the cartridge with 10 mL of methanol
2. Condition the cartridge with 10 mL of water
3. Load the 1 liter water sample at 100 mL/min
4. Rinse the cartridge
5. Dry the cartridge with nitrogen and vacuum for 20 minutes
6. Elute the cartridge with 15 mL of methanol base fraction
7. Elute the cartridge with 15 mL of methanol 2% formic acid
8. The fractions are directly eluted to the SuperVap Concentrator system

SuperVap Direct-to-Vial Concentration

Pre-heat temp: 40 °C.

Pre-heat time: 10 minutes

Heat in sensor mode: 50 °C

Nitrogen pressure: 15 PSI

Sensor 1 mL Direct to GC vial



Figure 1: PowerPrep SPE and SuperVap Concentrator systems.



Results

Table 1 shows the mean recoveries from the five extracts after analysis from several types of PPCPs in water.

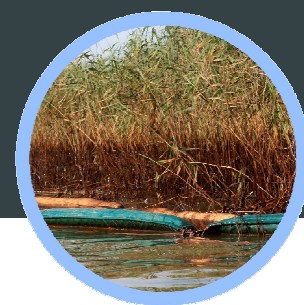
| Compound | Average Recovery |
|---------------------------|------------------|
| Atenolol | 88% |
| Atorvastatin | 81% |
| Avobenzone - A | 97% |
| Avobenzone - B | 92% |
| Ciprofloxacin | 99% |
| Benzophenone-1 | 98% |
| Benzophenone-3 | 94% |
| DEET | 90% |
| 4,4-Dihydroxybenzophenone | 86% |
| Estradiol | 81% |
| Estrone | 84% |
| Naproxen | 95% |
| Methylparaben | 85% |
| Propranolol | 80% |
| Ranitidine | 99% |
| Sulfamethoxazole | 98% |
| Sucralose | 97% |
| TCEP | 86% |
| Trimethoprim | 83% |
| Thiabendazole | 92% |
| Warfarin | 87% |
| Xanthine | 92% |

Conclusions

Analysis of the LC/MS data demonstrates excellent recoveries and reproducibility from a traditionally difficult sample matrix. Adding to the efficiency was the use of nitrogen and vacuum to dry the cartridge and a water free extract that enables a fast concentration step with no loss of analytes. The extract takes 45 minutes to concentrate using the PowerPrep™ SPE system compared to all other drying methods. Using the automated, one-step SPE and Direct-to-Vial Concentration tubes from FMS, Inc. eliminates error-prone manual or semi-automated steps from the sample prep process. No sample transfer is necessary, which allows the sample to be extracted and automatically sent to the SuperVap Concentrator where the final extract is concentrated directly to a vial for LC/MS analysis. This capability eliminates human error, saves time and increases efficiency while producing reproducible, consistent recoveries.

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An MDL study using Automated Solid Phase Extraction of Semi-Volatile Organic Compounds (AB 8270 SIM) in Water



Introduction

EPA Method 8270 is used to determine the concentration of semi-volatile organic compounds in extracts prepared from solid waste matrices such as soils, air sampling media and water samples. Extractions from water samples can be particularly difficult and time consuming due to the problematic analytes that need to be extracted, concentrated and analyzed as well as the large quantities of solvent the manual sample process requires. Laboratories now have to report analytes at lower reporting limits than previously required, so they often need to employ SIM scan variations of the method.

Given the recovery variability of 8270 compounds and the time it takes to conduct manual extractions, complete automation of the sample prep process is often viewed as the Holy Grail of sample prep. The following application outlines the complete automation of a modified 8270 extraction, delivering a complete, out-of-the box MDL study that demonstrates both precision and accuracy for mixed classes of low-level analytes.

Instrumentation and Consumables

- FMS, Inc. PowerPrep™ SPE system
- FMS, Inc. SuperVap™ Concentrator system
- FMS, Direct-to-Vial concentrator tubes
- Thermo Certified GC/MS auto sampler vials
- Waters 1 gram Oasis HLB® Cartridge
- Restek 2 gram Sodium sulfate Cartridge
- Thermo Trace GC w/DSQ MS and AS3000 Autosampler

Reagents

- Fisher Optima* Methanol
- Fisher Optima* Methylene Chloride
- Fisher HPLC Water
- Fisher Conc. Sulfuric Acid
- Restek Cat# 31622 Cal Mix #5
- Restek Cat# 316031 Phthalate mix
- AccuStandard Cat# M-8140-06 Diazinon mix
- Ultra Scientific Custom OPP Mix

Procedure:

Sample Prep

Five, 1 liter samples are measured out in glass sample collection bottles

Each sample is spiked with 1 mL of spiking solution (dilute of Ultra Scientific, Restek and Accustandard solutions)

The sample PH adjusted to <2 with 1:1 sulfuric acid solution

10 mL Methanol added to each sample bottle

PowerPrep SPE system

1. HLB Cartridges are conditioned with 10 mL of Methanol
2. HLB Cartridges are conditioned with 10 mL of DI H₂O
3. Samples are loaded across the HLB Cartridges via vacuum (~75mL/min)
4. Sample bottles are auto rinsed with DI water and the rinse loaded onto the HLB
5. The cartridges are dried with Nitrogen for 2 minutes
6. The HLB cartridges are eluted with 10 mLs of methylene chloride
7. The HLB cartridges are eluted with 10 mLs of methylene chloride
8. Cartridges are purged with N₂
9. Total time: 61.5 minutes

SuperVap Concentrator system

1. Pre-heat temp: 45 °C
2. Pre-heat time: 20 minutes
3. Heat in Sensor mode: 45 °C
4. Nitrogen Pressure: 10 PSI
5. End point: 1mL



Results

Table 1: MDL data compiled over seven replicates.

| Compound | Amount Spiked | SPE | | | | | | | Mean | STD Dev | MDL |
|----------------------------|---------------|--------|--------|--------|--------|--------|--------|--------|-------|---------|--------|
| | | #1 | #2 | #3 | #4 | #5 | #6 | #7 | | | |
| Anthracene | 0.05 | 0.0467 | 0.0510 | 0.0510 | 0.0500 | 0.0499 | 0.0515 | 0.0500 | 0.050 | 0.0016 | 0.0051 |
| bis(2-ethylhexyl)phthalate | 2 | 1.7920 | 1.9400 | 2.4600 | 1.8760 | 2.1200 | 1.9180 | 1.9040 | 2.001 | 0.2252 | 0.7076 |
| dibenzo[a,h]anthracene | 0.05 | 0.0401 | 0.0407 | 0.0438 | 0.0355 | 0.0394 | 0.0404 | 0.0407 | 0.040 | 0.0025 | 0.0077 |
| Chloropyrifos | 0.125 | 0.1450 | 0.1500 | 0.1638 | 0.1363 | 0.1638 | 0.1488 | 0.1475 | 0.151 | 0.0100 | 0.0313 |
| Pyrene | 0.05 | 0.0575 | 0.0580 | 0.0580 | 0.0555 | 0.0565 | 0.0565 | 0.0585 | 0.057 | 0.0011 | 0.0034 |
| Dichlorobenil | 0.125 | 0.0964 | 0.0986 | 0.0998 | 0.0961 | 0.0960 | 0.0976 | 0.1013 | 0.098 | 0.0020 | 0.0063 |
| Dimethylphthalate | 2 | 1.0760 | 0.9840 | 0.9300 | 0.8640 | 0.9580 | 0.9760 | 1.0200 | 0.973 | 0.0670 | 0.2104 |
| Di-n-butylphthalate | 2 | 1.9360 | 1.8960 | 2.0200 | 1.9420 | 1.9500 | 1.9200 | 2.0000 | 1.952 | 0.0437 | 0.1373 |
| benzo[a]anthracene | 0.05 | 0.0449 | 0.0471 | 0.0515 | 0.0463 | 0.0479 | 0.0464 | 0.0447 | 0.047 | 0.0023 | 0.0073 |
| Chrysene | 0.05 | 0.0415 | 0.0428 | 0.0458 | 0.0419 | 0.0425 | 0.0434 | 0.0424 | 0.043 | 0.0014 | 0.0044 |
| indeno[1,2,3-cd]pyrene | 0.05 | 0.0386 | 0.0398 | 0.0420 | 0.0357 | 0.0396 | 0.0396 | 0.0395 | 0.039 | 0.0019 | 0.0059 |
| Phenanthrene | 0.05 | 0.0560 | 0.0545 | 0.0540 | 0.0500 | 0.0525 | 0.0550 | 0.0560 | 0.054 | 0.0021 | 0.0067 |
| benzo[b]fluoranthene | 0.05 | 0.0469 | 0.0489 | 0.0525 | 0.0446 | 0.0462 | 0.0446 | 0.0471 | 0.047 | 0.0028 | 0.0087 |
| 2-methylnaphthalene | 0.05 | 0.0476 | 0.0465 | 0.0425 | 0.0465 | 0.0424 | 0.0461 | 0.0474 | 0.046 | 0.0022 | 0.0069 |
| benzo[a]pyrene | 0.05 | 0.0434 | 0.0459 | 0.0500 | 0.0422 | 0.0429 | 0.0432 | 0.0442 | 0.045 | 0.0027 | 0.0084 |
| Acenaphthylene | 0.05 | 0.0378 | 0.0359 | 0.0317 | 0.0353 | 0.0355 | 0.0379 | 0.0407 | 0.036 | 0.0028 | 0.0088 |
| Malathion | 0.125 | 0.1288 | 0.1500 | 0.1650 | 0.1425 | 0.1600 | 0.1500 | 0.1450 | 0.149 | 0.0119 | 0.0373 |
| Di-n-octylphthalate | 2 | 1.6420 | 1.8580 | 2.3200 | 1.7600 | 1.9840 | 1.8860 | 1.8360 | 1.898 | 0.2143 | 0.6736 |
| Acenaphthene | 0.05 | 0.0431 | 0.0376 | 0.0349 | 0.0369 | 0.0390 | 0.0405 | 0.0468 | 0.040 | 0.0040 | 0.0127 |
| Fluorene | 0.05 | 0.0410 | 0.0384 | 0.0339 | 0.0369 | 0.0375 | 0.0392 | 0.0415 | 0.038 | 0.0026 | 0.0081 |
| benzo[k]fluoranthene | 0.05 | 0.0458 | 0.0479 | 0.0570 | 0.0447 | 0.0475 | 0.0483 | 0.0458 | 0.048 | 0.0041 | 0.0130 |
| Diazanon | 0.05 | 0.0555 | 0.0530 | 0.0565 | 0.0575 | 0.0615 | 0.0560 | 0.0595 | 0.057 | 0.0028 | 0.0087 |
| Fluoranthene | 0.05 | 0.0595 | 0.0585 | 0.0585 | 0.0545 | 0.0585 | 0.0565 | 0.0590 | 0.058 | 0.0017 | 0.0055 |
| Butylbenzylphthalate | 2 | 1.8800 | 2.0800 | 2.4600 | 2.0200 | 2.0800 | 2.0600 | 1.9660 | 2.078 | 0.1832 | 0.5757 |
| Diethylphthalate | 0.05 | 1.8340 | 1.5380 | 1.4020 | 1.4920 | 1.5720 | 1.5860 | 1.8800 | 1.615 | 0.1767 | 0.5553 |
| Naphthalene | 0.05 | 0.0430 | 0.0424 | 0.0408 | 0.0411 | 0.0400 | 0.0416 | 0.0455 | 0.042 | 0.0018 | 0.0057 |
| benzo[g,h,i]perylene | 0.05 | 0.0363 | 0.0355 | 0.0387 | 0.0334 | 0.0375 | 0.0364 | 0.0372 | 0.036 | 0.0017 | 0.0053 |
| Prometon | 0.125 | 0.1375 | 0.1198 | 0.1133 | 0.0984 | 0.1288 | 0.1209 | 0.1350 | 0.122 | 0.0135 | 0.0425 |



Figure 1: FMS PowerPrep SPE system with the SuperVap Concentrator

Conclusions

The results in Table 1 show the recoveries of all seven MDL replicates extracted on the FMS PowerPrep™ SPE/ SuperVap Direct-to-Vial Concentration system. The results demonstrate the ability of the FMS Total Sample Prep approach to deliver precise, consistent recoveries at ultra low level concentrations and with extreme precision. The combination of high recoveries and low standard deviations deliver a low-level MDL that is easy to achieve.

The FMS PowerPrep SPE and SuperVap Direct-to-Vial automated turnkey system is an ideal choice for automating a wide range of compound classes. The system is capable of extracting 5-30 samples in one program which allows the system to grow along with your laboratory's throughput. This turnkey system gives laboratories the ability to rapidly extract and concentrate directly to a vial entire analytical batches with just the click of a mouse.

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The Automated Extraction of Aqueous Samples by Method EPA 8270D Using the TurboTrace™ 8270 SPE System



Introduction

EPA 8270D calls for the extraction for analysis of semi-volatile analytes in various matrices. Target analytes mentioned in the method cover a wide range of compound classes resulting in reporting lists that often approach 100 compounds. In aqueous samples, the combination of compound groups such as phenols, analines, PAHs, phthalates, explosives, pesticides, n-nitrosoamines, and others results in a tedious process of multiple LLE (liquid-liquid extractions) with multiple pH adjustments to extract all the analytes. Large elution volumes (>360 mL of methylene chloride) combined with highly volatile analytes, usually result in low recoveries of target compounds and poor analytical precision.

The use of SPE (Solid Phase Extraction) to automate the extraction process can drastically reduce many of the challenges traditional 8270 extractions pose. By automating the process with the FMS 8270 SPE system, extraction chemists, normally confined to a hood shaking separatory funnels, can be freed up to perform additional tasks.

Instrumentation and Consumables

Instrumentation

- FMS, Inc. TurboTrace™ 8270 SPE system
- FMS, Inc. SuperVap™ concentrator
- FMS, Inc. 200 mL concentrator tubes
- Thermo Trace GC w/DSQ MS

Consumables

- Fisher Pesticide Optima* Methylene Chloride
- Fisher Anhydrous Sodium Sulfate
- Fisher Optima* Methanol
- Fisher HPLC Grade Water
- FMS 1 gram DVB Cartridges
- Fisher Concentrated Sulfuric Acid
- Fisher Sodium Hydroxide

Consumables (continued)

- Restek Resprep 2 gram coconut charcoal cartridges (Cat# 26032)
- Restek 8270 matrix spike (Cat# 33073)
- Restek SV Internal Standard Mix (Cat# 31006)
- Restek B/N Surrogate (Cat# 31024)
- Restek Acid Surrogate (Cat# 31025)
- Restek Benzidines mix (Cat# 31834)

Procedure

Prepare 1 liter samples of DI water

Adjust pH of samples to <2 by adding H₂SO₄ drop wise.

Spike samples with 8270 matrix spike, B/N surrogate, and Acid surrogate spiking solutions

Load samples onto FMS TurboTrace 8270 SPE system.

Affix collection bottle to retain post extraction aqueous sample.

Affix coconut charcoal and DVB with pre-filter cartridges to TurboTrace 8270 system.



Figure 1: FMS TurboTrace SPE system with the SuperVap concentrator.



SPE

1. Cartridges pre-wet with DCM
2. Cartridges conditioned with MeOH
3. Cartridges conditioned with H₂O
4. Samples passed across DVB cartridges at ~20 mL/min via vacuum
5. Cartridges partially dried with N₂ at 15 PSI
6. Sample bottles sprayed with DCM
7. DCM bottle spray loaded across cartridge and collected through in-line NaSO₄
8. Cartridges eluted with additional 10 mL DCM
9. Cartridges purged with N₂ eluting n-Hexane directly to FMS SuperVap concentrator
10. Remove aqueous collection bottle and add NaOH solution to sample mixing thoroughly till pH is >12.
11. Attached sample bottle to system for second pass.
12. DVB Cartridge re-conditioned with MeOH.
13. DVB Cartridge re-conditioned with H₂O
14. Sample passed across both cartridges at ~10 mL/min.
15. Cartridges dried with N₂ for 1 minute each
16. Sample bottle sprayed with DCM
17. DCM bottle spray loaded across DVB cartridge and collected through in-line NaSO₄
18. DVB Cartridge eluted with additional 5 mL DCM
19. Coconut charcoal eluted with 20 mL DCM
20. Cartridges independently purged of residual solvent via N₂ stream.

SuperVap Concentrator

1. Preheat temp: 10 minutes at 40 °C
2. Evap mode: 40 °C
3. Nitrogen Pressure: 10 PSI
4. Evaporate extracts 1 mL*

*Evaporator tubes manually rinsed with DCM to ensure no target analytes adhere to evaporator tube walls.

Internal Standard solution added to extract post evaporation for GC/MS analysis.

Results

Table 1: Results from 6 1L LCS samples (50 µg/L concentration)

| Analyte | Mean |
|-------------------------------|--------|
| Pyridine | 71.63 |
| NDMA | 54.44 |
| 2-Fluorophenol (Surr) | 85.84 |
| Phenol-d5 (Surr) | 78.32 |
| Phenol | 74.6 |
| Aniline | 84.06 |
| bis(2-chloroethyl) ether | 93.3 |
| 2-Chlorophenol | 85.99 |
| 1,3-Dichlorobenzene | 84.86 |
| 1,4-Dichlorobenzene | 86.29 |
| 1,2-Dichlorobenzene | 90.67 |
| benzyl_alcohol | 90.12 |
| 2-methylphenol | 91.32 |
| bis(2-chloroisopropyl) ether | 94.6 |
| N-nitrosodi-n-propylamine | 103.54 |
| 4-methylphenol/3-methylphenol | 93.3 |
| Hexachloroethane | 89.79 |
| Nitrobenzene-d5 (Surr) | 96.27 |
| nitrobenzene | 111.68 |
| isophorone | 94.42 |
| 2-Nitrophenol | 84.56 |
| 2,4-dimethylphenol | 99.46 |
| bis(2-chloroethoxy)methane | 100.55 |
| 2,4-Dichlorophenol | 89.01 |
| 1,2,4-trichlorobenzene | 85.24 |
| Naphthalene | 87.54 |
| 4-Chloroaniline | 80.43 |
| hexachlorobutadiene | 81.3 |
| 4-Chloro-3-methylphenol | 89.26 |
| 2-methylnaphthalene | 103.61 |
| 1-methylnaphthalene | 103.95 |
| hexachlorocyclopentadiene | 52.78 |
| 2,4,6-Trichlorophenol | 78.76 |
| 2,4,5-Trichlorophenol | 79.44 |
| 2-Fluorobiphenyl (Surr) | 99.03 |
| 2-Chloronaphthalene | 84.73 |
| 2-Nitroaniline | 93.23 |
| 1,4-dinitrobenzene | 93 |
| dimethyl phthalate | 97.45 |
| 1,3-dinitrobenzene | 95.21 |



Results (continued)

| Analyte | Mean |
|-----------------------------|--------|
| Acenaphthylene | 89.58 |
| 1,2-dinitrobenzene | 96.45 |
| 3-Nitroaniline | 100.57 |
| 2,6-dinitrotoluene | 101.41 |
| Acenaphthene | 95.77 |
| 2,4-dinitrophenol | 83.84 |
| 4-Nitrophenol | 99.19 |
| dibenzofuran | 94.81 |
| 2,4-dinitrotoluene | 96.66 |
| 2,3,5,6-Tetrachlorophenol | 89.25 |
| 2,3,4,6-Tetrachlorophenol | 87.3 |
| diethyl phthalate | 107.46 |
| Fluorene | 100.96 |
| 4-chlorophenyl phenyl ether | 90.49 |
| 4-Nitroaniline | 112.58 |
| 2-Methyl-4,6-dinitrophenol | 102.85 |
| NDA-NDPA | 89 |
| Azobenzene | 101.4 |
| 2,4,6-Tribromophenol (Surr) | 86.58 |
| 4-bromophenyl phenyl ether | 90.94 |
| Hexachlorobenzene | 92.84 |
| pentachlorophenol | 96.05 |
| Phenanthrene | 99.97 |
| Anthracene | 100.15 |
| Carbazole | 108.6 |
| butyl benzyl phthalate | 117.76 |
| Pyrene | 105.37 |
| Fluoranthene | 98.53 |
| Terphenyl-d14 (Surr) | 102.05 |
| di-n-butyl phthalate | 109.91 |
| benzo[a]anthracene | 106.3 |
| Chrysene | 102.82 |
| bis(2-ethylhexyl)phthalate | 124.46 |
| di-n-octyl_phthalate | 118.02 |
| benzo[b]fluoranthene | 102.23 |
| benzo[k]fluoranthene | 114.84 |
| benzo[a]pyrene | 99.25 |
| indeno[1,2,3-cd]pyrene | 96.23 |
| dibenzo[a,h]anthracene | 94.5 |
| benzo[g,h,i]perylene | 95.43 |
| Benzidine | 88.21 |
| 3,3-dichlorobenzidine | 94.6 |

Conclusions

Evaluation of the performance spikes on the FMS TurboTrace™ 8270 extraction system resulted in average recoveries for nearly all analytes between 70-130%. Combined with the analysis of blank replicates which yielded background concentrations of less than 1 µg/mL for all analytes, the data, when compared to the performance tables within EPA 8270D, shows that automated solid phase extraction is not only equivalent, but in some cases superior to traditional LLE extractions.

Repeatability data gave percent deviations between replicates of less than ±10% for most compounds, and ±20% for all. Fully automated sample loading and eluting of both cartridges independently, and in sequence, eliminates the need to have an extraction chemist separate the cartridges at different stages of the operation. The reduction of human interaction saves time, reduces human error and produces consistent, reproducible results.

When paired with the FMS SuperVap concentrator, the TurboTrace™ 8270 SPE system provides complete, automated sample preparation for aqueous samples by EPA8270D.

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Solid Phase Extraction of Waste Water Samples by EPA 608



Introduction

Solid Phase Extraction of chlorinated pesticides and polychlorinated biphenyls has long been an accepted sample preparation technique for EPA 500 series drinking water samples. Both C₁₈ and DVB cartridges and disks have consistently generated excellent recoveries in drinking water matrices. EPA 600 series methods however, focus on waste water samples that can prove a greater analytical challenge than finished drinking water. The presence of organic materials, surfactants, and other matrix interferences can pose extraction issues for both SPE and traditional LLE (Liquid-Liquid Extraction) techniques. Extract purification is also necessary in most samples, usually involving a Florisil clean-up step.

By using the FMS, Inc. TurboTrace Solid Phase Extraction system together with the PowerPrep sample cleanup and SuperVap concentrator, it is possible to automate the extraction, extract purification and concentration steps of EPA 608.

Instrumentation and Consumables

Instrumentation

- FMS, Inc. TurboTrace SPE (Solid Phase Extraction) System
- FMS, Inc. SuperVap 12 position Concentrator
- FMS, direct-to-vial concentrator tubes
- FMS, Inc Power Prep clean up system
- Agilent 7890 GC with μ ECD

Consumables

- FMS 1 gram C₁₈ cartridges w/pre-filter
- FMS Florisil Columns
- Fisher Pesticide Grade Methanol
- Fisher Pesticide Grade Methylene Chloride
- Fisher HPLC Grade Water
- FMS, Fisher Pesticide Grade Hexanes
- FMS, Fisher Pesticide Grade Acetone

- Fisher Anhydrous Sodium Sulfate Restek Cat #32005; Toxaphene Solution Restek Cat#32009; Aroclor 1242 Solution Restek Cat#32012; Aroclor 1260 Solution Restek Cat# 32291; Organochlorine Pest AB Mix

Procedure

Synthetic waste waters were created following ASTM D5905-96 guidelines.

1 liter of waste water constitutes:

- 60 mL light beer
- .2 grams bleached flour
- 1 gram Sea Salts (Sigma #S-9883)
- .04 grams Kaolin (Sigma #K-7375)
- 10 mL of a .12% Triton-X 100 Surfactant (Sigma #X-100)
- DI water adjusted to 1 L volume

SuperVap Concentration system

1. Pre-heat temp: 45 °C
2. Pre-heat time: 20 minutes
3. Heat in Sensor mode: 45 °C
4. Nitrogen Pressure: 10 PSI
5. End point: 1 mL

TurboTrace SPE System

1. Samples acidified by adding H₂SO₄ drop wise till PH is <2
2. Replicates (3) were spiked with Pesticide, Arochlor and Toxaphene solutions as well as unfortified samples for comparison.



Figure 1. FMS TurboTrace SPE with SuperVap concentrator



3. Sample bottles attached to TurboTrace™ SPE system
3. C₁₈ cartridges w/pre-filters fitted to sample loop.
4. NaSO₄ containing cartridges placed at fraction line termination above collection vials.
5. Cartridges pre-conditioned with 10 mL Methylene Chloride.
6. Cartridges conditioned with 10 mL MeOH
7. Cartridges conditioned with 15 mL H₂O
8. Samples loaded across Cartridges at ~15 mm HG vacuum.
9. Cartridges dried with N₂ for 5 minutes each.
10. Sample bottles rinsed with 25 mL Methylene Chloride.
11. Methylene Chloride rinse loaded across cartridges and collected in fraction vials
12. Cartridges eluted with 10 mL Methylene Chloride.
13. Eluates nitrogen purged through NaSO₄ into collection vials.

SuperVap™ Concentrator

1. Preheat temp: 10 minutes at 45 °C
2. Evap mode w/sensor temp: 40 °C
3. Nitrogen pressure: 5 PS
4. When extracts at 1 mL, 5 mL hexane added to vials.
5. Extracts concentrated under N₂ till 1 mL sensor alarm signals.

PowerPrep Cleanup

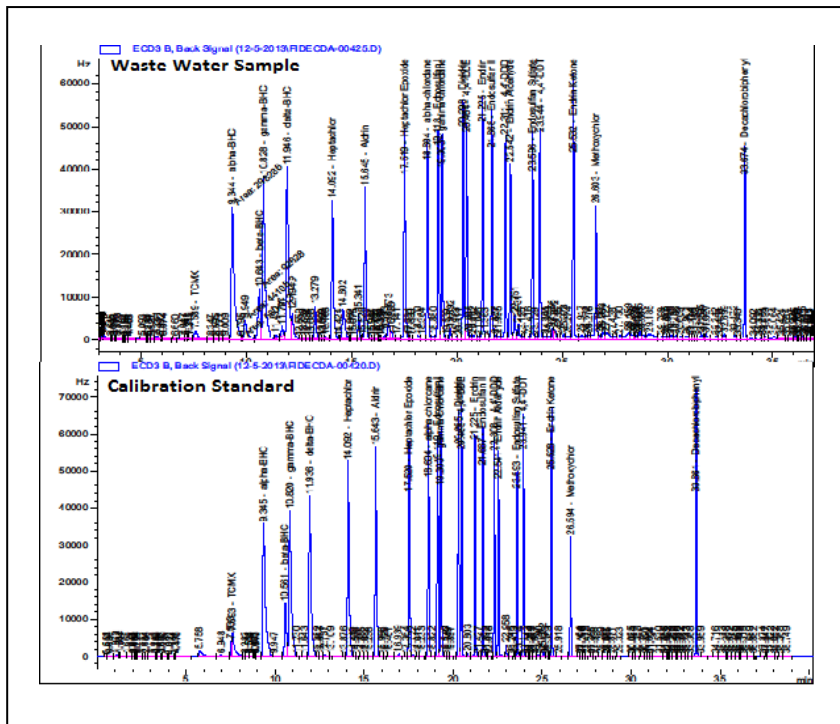
1. Florisil cartridges fitted to Power Prep™ System.
2. Cartridges pre-wet with 10 mL Hexane
3. Sample extracts loaded into Power Prep System.
4. Florisil cartridges eluted with 20 mL Acetone/Hexane (10:90) solution.
5. Elute collected in direct to GC vial tubes and evaporated to 1 mL under same settings as SPE extracts.

Table 1; Results of fortified synthetic waste water samples

| Compound | Mean Rec. | Acc. Limit |
|--------------------|-----------|------------|
| α-BHC | 80.9% | 37-134 |
| β-BHC | 85.5% | 17-147 |
| Lindane | 88.6% | 32-127 |
| δ-BHC | 93.0% | 19-140 |
| Heptachlor | 65.4% | 34-111 |
| Aldrin | 62.5% | 42-122 |
| Heptachlor Epoxide | 81.4% | 37-142 |
| α-Chlordane | 71.8% | 45-119 |
| Endosulfan I | 82.7% | 45-153 |
| γ-Chlordane | 71.8% | 45-110 |
| Dieldrin | 85.0% | 36-146 |
| 4,4'-DDE | 71.8% | 30-145 |
| Endrin | 105.6% | 30-147 |
| Endosulfan II | 90.1% | D-202 |
| Endrin Aldehyde | 75.2% | NA |
| 4,4'-DDD | 84.8% | 31-141 |
| Endosulfan Sulfate | 102.4% | 26-144 |
| 4,4'-DDT | 79.3% | 25-160 |
| Methoxychlor | 112.3% | NA |
| Endrin Ketone | 105.9% | NA |
| Arochlor 1242 | 63.2% | 39-150 |
| Arochlor 1260 | 77.7% | 8-127 |
| Toxaphene | 88.8% | 41-126 |



Figure 2. Overlay of fortified waste water sample with calibration standard.



Conclusions

Analysis of the extracts yielded recoveries for all analytes tested well within EPA 608 acceptance criteria. Using C₁₈ cartridges equipped with pre-filtration sufficiently controlled a steady flow through the cartridge preventing clogging. Paired with PowerPrep Florisil clean-up, extracts were purified of non-target interferences preventing matrix interference of target analytes. The closing degradation check on the GC indicated sample extracts did not “dirty” the GC inlet further demonstrating the efficiency of the PowerPrep system.

By using the TurboTrace™ SPE system, emulsions typically formed by LLE procedures were eliminated, often a major cause of analyte loss. Using inline water removal and automated extract cleanup, extraction chemist interaction with the sample was less, thus reducing the risk of human error in the process. By automating EPA 608 with Solid Phase Extraction, waste water samples (typically a challenging matrix) can be extracted more efficiently than by traditional LLE procedures.

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EPA 625, Base, Neutral and Acid Semi-volatiles in Municipal and Industrial Waste Water by SPE



Introduction

Solid Phase extraction has long been used for the analysis of semi-volatile organics in clean matrices. Methods like EPA 525.3 and EPA 8270D outline performance data for a variety of analytes and products. Due to the unique challenges inherent with waste water matrices laboratories have predominately adhered to LLE (Liquid Liquid Extraction) protocols. Recent advances in packing materials and automated extraction systems have now made once unheard of extractions of matrices commonplace for SPE.

Traditionally involving 6 LLE shakes at both pH 2 and 12, plus heavy emulsions and low recoveries, traditional EPA 625 extractions are time consuming and often result in poor results. By incorporating automated SPE with multi-bed sorbents, 625 samples can be extracted with a single pass procedure without emulsions, centrifuging and hours of manual labor. The FMS TurboTrace Semi-Volatile system is a specialized variant of the TurboTrace system. It is designed to handle multiple cartridges, and provides a fully automated solution for the semi-volatile EPA 625 extraction process.

Instrumentation and Consumables

Instrumentation

Instrumentation

- FMS, Inc. TurboTrace Semi-Volatile SPE system
- FMS, Inc. SuperVap Concentrator
- FMS, Inc. 200 mL concentrator tubes
- Thermo Trace GC w/DSQ MS

Consumables

- Fisher Pesticide Optima* Methylene Chloride
- Fisher Anhydrous Sodium Sulfate
- Fisher Optima* Methanol
- Fisher HPLC Grade Water

- FMS mixed bed 625 cartridges
- Fisher Concentrated Sulfuric Acid
- Fisher Sodium Hydroxide
- Restek Resprep 2 gram coconut charcoal cartridges (Cat# 26032)
- Restek 8270 matrix spike (Cat# 33073)
- Restek SV Internal Standard Mix (Cat# 31006)
- Restek B/N Surrogate (Cat# 31024)
- Restek Acid Surrogate (Cat# 31025)
- Restek Benzidines mix (Cat# 31834)

Procedure

Prepare 1 liter samples of DI water and ASTM D5905-95 synthetic waste water.

Adjust pH of samples to <2 by adding H₂SO₄ drop wise.

Spike samples with matrix spike, B/N surrogate, and acid surrogate spiking solutions

Load samples onto FMS TurboTrace Semi-Volatile SPE system.

Affix coconut charcoal and mixed bed with pre-filter cartridges to TurboTrace Semi-Volatile SPE system



Figure 1. FMS TurboTrace SPE with SuperVap concentrator

**SPE**

1. Cartridges pre-wet with DCM
1. Cartridges conditioned with MeOH
2. Cartridges conditioned with H₂O
3. Samples passed across both cartridges at ~15 mm HG
4. Mixed bed cartridge partially dried with N₂ at 10 PSI
5. Sample bottles sprayed with DCM
6. DCM bottle spray loaded across mixed cartridge and collected as Fraction #1
7. Cartridges eluted with additional 10 mL DCM
8. Cartridges purged with N₂ passing all DCM to collection vials.
9. Mixed bed Cartridge re-conditioned with MeOH.
10. Both cartridges conditioned with a 1% NaOH solution
11. Cartridges independently dried with N₂ for 1 minute each
12. Mixed bed Cartridge eluted with additional 30 mL DCM and collected as Fraction #2
13. Coconut charcoal eluted with 30 mL DCM and added to Fraction #2
14. Cartridges independently purged of residual solvent via N₂ stream.

Fractions passed through NaSO₄ and combined for evaporation.

SuperVap™ Concentrator

1. Preheat temp: 10 minutes at 40 °C
2. Evap mode: 40 °C
3. Nitrogen Pressure: 10 PSI
4. Evaporate extracts 1 mL*

*Evaporator tubes manually rinsed with DCM to ensure no target analytes adhere to evaporator tube walls.

Internal Standard solution added to extract post evaporation for GC/MS analysis.

Table 1; Mean recoveries in synthetic waste water for table #6 analytes from EPA 625

| Analyte | Mean Rec | Acc. Limit |
|------------------------------|----------|------------|
| Acenaphthene | 76.9 | 47-145 |
| Acenaphthylene | 80.6 | 33-145 |
| Aldrin | 84.8 | D-166 |
| Anthracene | 92.1 | 27-133 |
| benzo[a]anthracene | 96.2 | 33-143 |
| benzo[b]fluoranthene | 95.9 | 24-159 |
| benzo[k]fluoranthene | 109.5 | 11-162 |
| benzo[a]pyrene | 92.5 | 17-163 |
| benzo[g,h,i]perylene | 92.5 | D-219 |
| β-BHC | 54.0 | 24-149 |
| δ-BHC | 78.5 | D-110 |
| bis(2-chloroethyl) ether | 80.2 | 12-158 |
| bis(2-chloroethoxy)methane | 79.2 | 33-184 |
| bis(2-chloroisopropyl) ether | 80.5 | 36-166 |
| bis(2-ethylhexyl)phthalate | 108.8 | 8-158 |
| 4-bromophenyl phenyl ether | 82.1 | 53-127 |
| 2-Chloronaphthalene | 71.7 | 60-118 |
| 4-chlorophenyl phenyl ether | 81.8 | 25-158 |
| Chrysene | 99.1 | 17-168 |
| 4,4'-DDE | 59.5 | D-145 |
| 4,4'-DDD | 59.9 | 4-136 |
| 4,4'-DDT | 64.5 | D-203 |
| dibenzo[a,h]anthracene | 92.9 | D-227 |
| di-n-butyl phthalate | 102.2 | 1-118 |
| 1,2-Dichlorobenzene | 73.5 | 32-129 |
| 1,3-Dichlorobenzene | 69.7 | D-172 |
| 1,4-Dichlorobenzene | 69.7 | 20-124 |
| 3,3-dichlorobenzidine | 78.9 | D-262 |
| Dieldrin | 70.1 | 29-136 |
| diethyl phthalate | 97.0 | D-114 |
| dimethyl phthalate | 84.2 | D-112 |
| 2,4-dinitrotoluene | 91.9 | 39-139 |



Table 2; Mean recoveries in synthetic waste water for additional analytes tested

| Analyte | Mean |
|-------------------------------|-------|
| Pyridine | 44.9 |
| NDMA | 30.1 |
| Aniline | 53.8 |
| benzyl_alcohol | 73.2 |
| 2-methylphenol | 63.6 |
| 4-methylphenol/3-methylphenol | 76.4 |
| 4-Chloroaniline | 63.4 |
| 4-Chloro-3-methylphenol | 56.0 |
| 2-methylnaphthalene | 73.2 |
| 1-methylnaphthalene | 71.4 |
| hexachlorocyclopentadiene | 37.5 |
| 2,4,5-Trichlorophenol | 59.3 |
| 2-Nitroaniline | 78.2 |
| 1,4-dinitrobenzene | 80.5 |
| 1,3-dinitrobenzene | 83.1 |
| 1,2-dinitrobenzene | 85.3 |
| 3-Nitroaniline | 71.1 |
| dibenzofuran | 76.4 |
| 2,3,5,6-Tetrachlorophenol | 51.1 |
| 2,3,4,6-Tetrachlorophenol | 52.7 |
| 4-Nitroaniline | 90.3 |
| NDA-NDPA | 86.1 |
| Azobenzene | 85.4 |
| Carbazole | 102.7 |
| butyl benzyl phthalate | 79.0 |
| Benzidine | 67.1 |

Conclusions

Analysis of the analytes from table #6 found in EPA 625 shows that the recoveries for the FMS TurboTrace Semi-Volatile system are all well within the acceptance QC limits for actual waste water matrices. Analysis of additional analytes shows the application is also suitable for analytes beyond those with specified QC criteria by the method.

The ability to efficiently load a waste water matrix in a single pass, with no additional manual pH adjustments or manual steps, makes the FMS TurboTrace Semi-Volatile system a more efficient alternative to traditional LLE methods. By adopting the TurboTrace system, laboratories can eliminate time consuming emulsions and error-prone manual steps from their workflow.

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EPA 1664A; Oil and Grease by Solid Phase Extraction (SPE)



Introduction

EPA 1664A refers to the Solid Phase Extraction (SPE) protocol for extracting hexane extractable oil and grease materials from water samples. The method calls for the passage of a 1 liter aqueous sample across an SPE cartridge or disk, then eluting the cartridge or disk with n-hexane.

The traditional alternative to SPE, LLE (Liquid-Liquid) Extraction, is both time consuming and labor-intensive plus it requires large volumes of solvent. In this automated SPE method, samples are rapidly pulled across a C₁₈ cartridge and then eluted with less than one quarter of the solvent required for eluting the same sample by LLE.

The following application covers the extraction of aqueous samples using the FMS, Inc Turbo Trace system to extract aqueous samples for EPA 1664.

Instrumentation

- FMS, Inc. TurboTrace™ SPE system
- FMS, Inc. SuperVap™ 12 Concentrator
- FMS, Inc. 50 mL direct-to-vial concentrator tubes
- Mettler Toledo analytical balance

Consumables

- Fisher Pesticide Optima* n-Hexane
- Fisher Anhydrous Sodium Sulfate
- Fisher Optima* Methanol
- Fisher HPLC Grade Water
- FMS 2 gram C₁₈ Cartridges
- Restek Oil & Grease Mix (Cat#31954)
- Fisher Concentrated Sulfuric Acid

Procedure

Pre-weigh collection vials

1 liter aqueous samples acidified to PH <2 with H₂SO₄

Samples spiked with varying concentrations of oil and grease spiking solution.

SPE

1. Cartridges pre-wet with Hexane
2. Cartridges conditioned with MeOH
3. Cartridges conditioned with H₂O
4. Samples passed across C₁₈ cartridges at full vacuum
5. Cartridges dried with N₂ at 15 PSI
6. Sample bottles sprayed with n-Hexane
7. N-Hexane bottle spray loaded across cartridge and collected through in-line NaSO₄
8. Cartridges eluted with additional 10 mL n-Hexane
9. Cartridges purged with N₂ eluting n-Hexane directly to FMS SuperVap concentrator

SuperVap concentrator

1. Preheat temp: 20 minutes at 60 °C
2. Evap mode: 60 °C
3. Nitrogen Pressure: 10 PSI
4. Evaporate extracts to total dryness



Figure 1: TurboTrace SPE system with the SuperVap Concentrator.



Results

Table 1; Results of oil and grease LCS samples at three concentrations.

| Sample Concentration | Amount Recovered | % | RPD |
|----------------------|------------------|-------|------|
| 2 mg/L | 2.1 mg/L | 105% | 4.9% |
| 2 mg/L | 2.0 mg/L | 100% | |
| 5 mg/L | 5.0 mg/L | 100% | 0.0% |
| 5 mg/L | 5.0 mg/L | 100% | |
| 10 mg/L | 9.99 mg/L | 99.9% | 1.0% |
| 10 mg/L | 9.89 mg/L | 98.9% | |

Conclusions

Analysis of oil and grease yielded excellent, consistent recoveries with minimal deviations between replicates. Extractions at 2 mg/L near the method MDL (1.4 mg/L), at the ML (5 mg/L) and at 10 mg/L all displayed excellent recoveries.

With a total of 40 mls n-Hexane required for the elution and bottle rinse combined with a total extraction time of <25 minutes from start to elution the Turbo Trace SPE proves to be a far more efficient and economical solution for EPA1664 extractions than LLE. Combined with SuperVap Concentrator, minimal sample manipulation is required eliminating possible recovery loss by human interaction.

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