Productivity Enhancements in Microwave Assisted Extractions (EPA Method 3546) Of Semi Volatile Organic Compounds From Environmental Samples

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Agenda

- Introduction to EPA 3546
- Conventional Extraction Methods
- Microwave Assisted Extraction
- Data Comparison
- Improvements To Increase Throughput
- Summary
- Q&A

What is EPA Method 3546?

- Method for extracting water insoluble or slightly soluble organic compounds from soils, clays, sediments, sludge and solid wastes.
- Employs closed vessel microwaves to achieve the extraction of analytes at high temperatures and pressures
- Recovery equivalent to Soxhlet extraction (EPA method 3540) with – less solvent, shorter time and better precision in most cases

Typical Parameters – EPA 3546

Application: Soils, Clays, Sediments, Sludge, Solid Wastes
Sample Size: 2 – 20 g
Solvent Volume: 25 ml
Temperature: 100 – 115 ^{0}C
Pressure: 50 – 150 psi

The need for EPA 3546…

- Extraction demands
- Data quality
- Representative sampling
- Productivity enhancement
- Cost effectiveness

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Conventional Techniques

Soxhlet:

- Regarded as the standard method
- Inexpensive equipment
- Able to process large amounts of sample
- Separate filtration is unnecessary

Conventional Techniques

Limitations of Soxhlet Extractions:

- Long operation times - ~4 – 48 hours
- Large solvent consumption – 200 – 500 ml
- Large operation cost
- Only extraction capabilities
- High exposure to toxic materials

Conventional Techniques

Sonication:

- Fast method – 30 – 60 min
- Inexpensive equipment
- Able to process large amounts of sample
- Matrix Independent

Conventional Techniques

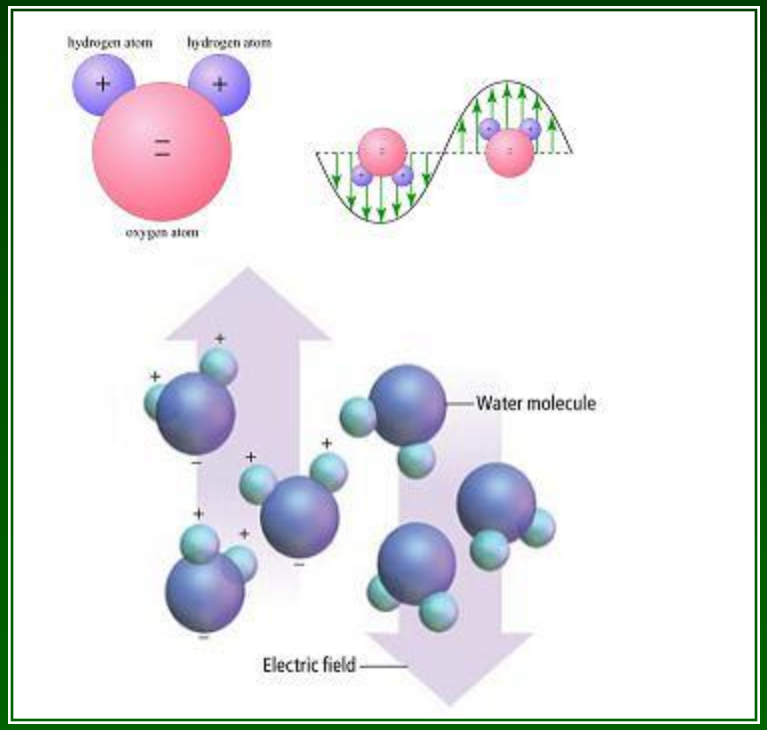
Limitations of Sonication Extractions:

- Large solvent consumption – 200 – 500 ml
- Labor intensive
- Filtration required
- High exposure to toxic materials

Microwave Theory

How Microwaves Work:

- Polar molecules rapidly oscillate with the oscillating electromagnetic field.
- Rotating molecules push, pull, collide other molecules and generate heat



Microwave Extraction

- Specially designed microwaves
- Rotors with multiple vessels
- Samples + mixture of solvents added
- Closed vessels ensure no loss of volatiles
- Concentration
- Analysis

Advantages of Microwave Assisted Extraction

- High throughput
- Reduction of operation time
- Minimum consumption of solvents
- Save of space
- Reduction of operation cost
- No expensive glassware
- Broad range of sample sizes
- Flexible platform (multiple chemistries)
- Conforms with EPA's pollution prevention goals

Milestone's Ethos EX

- Chassis
- Safety features
- Color touch-screen controller
- PID Control

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Improvements to Enhance Productivity – Rotors & Vessels

Milestone Ethos EX Rotors - Specifications

SK-10
Max. temp & pressure **Max. Press.** 100 bar **Max. Temp.** 300°C
Number of Samples 10
Vessel Volume 100 mL
Minimum vol. 10 mL
Max. sample wt. Up to 20 g
Vessel Material TFM
Vessel Technology Vent-and-Reseal

SK-12
Max. temp & pressure **Max. Press.** 35 bar **Max. Temp.** 300°C
Number of Samples 12
Vessel Volume 100 mL
Minimum vol. 10 mL
Max. sample wt. Up to 15 g
Vessel Material TFM
Vessel Technology Vent-and-Reseal

Q-20
Max. temp & pressure **Max. Press.** 40 bar **Max. Temp.** 250°C
Number of Samples 20
Vessel Volume 45 mL
Minimum vol. 3 ml
Max. sample wt. Up to 2 g
Vessel Material TFM
Vessel Technology Vent-and-Reseal

PRO-16/24
Max. temp & pressure **Max. Press.** 30 bar **Max. Temp.** 260°C
Number of Samples 24
Vessel Volume 75 mL
Minimum vol. 10 mL
Max. sample wt. Up to 15 g
Vessel Material TFM
Vessel Technology Vent-and-Reseal

LVR
Max. temp & pressure **Max. Press.** 10 bar **Max. Temp.** 200°C
Number of Samples 6
Vessel Volume 270 mL
Minimum vol. 10 mL
Max. sample wt. Up to 30 g
Vessel Material TFM
Vessel Technology Vent-and-Reseal

MUP-41
Max. temp & pressure **Max. Press.** 30 bar **Max. Temp.** 260°C
Number of Samples 41
Vessel Volume 70 mL
Minimum vol. 10 mL
Max. sample wt. 10 g
Vessel Material TFM or PFA
Vessel Technology Self-regulating

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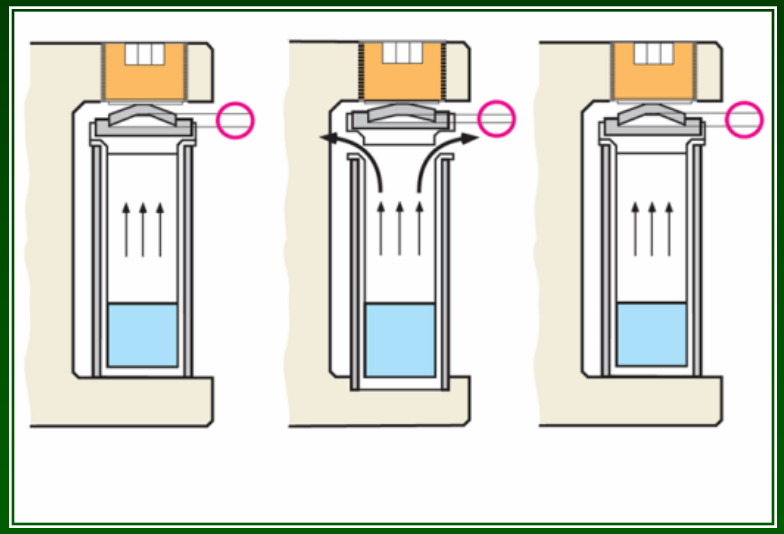
Productivity Enhancement – High Sample Size & Solvent Volume

- Patented by Milestone
- Cap is held in place by a PEEK spring which deforms as pressure pushes the cap upwards
- Overpressure is gently and controllably released
- Pressure is maintained in the vessel even while venting
- No ejection of contents or loss of volatiles



Vent and Reseal Technology

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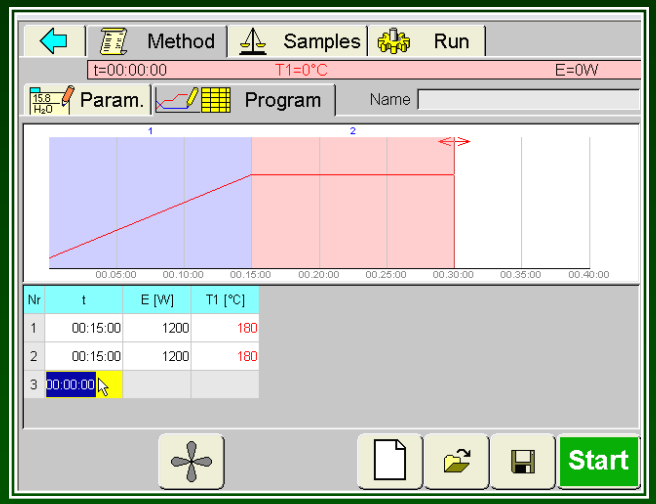
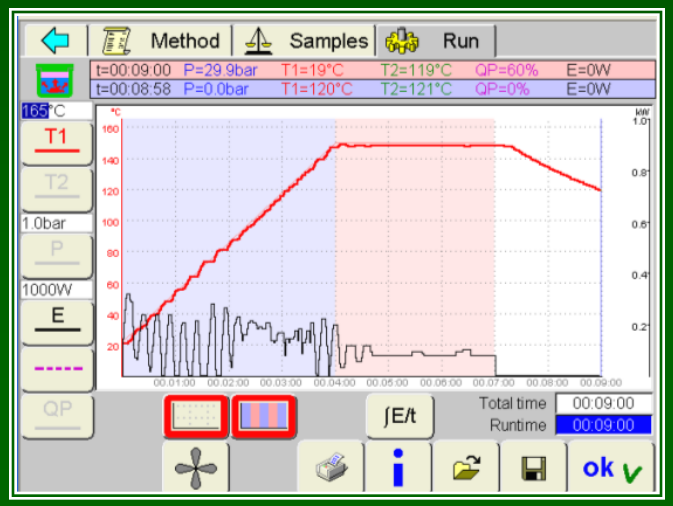
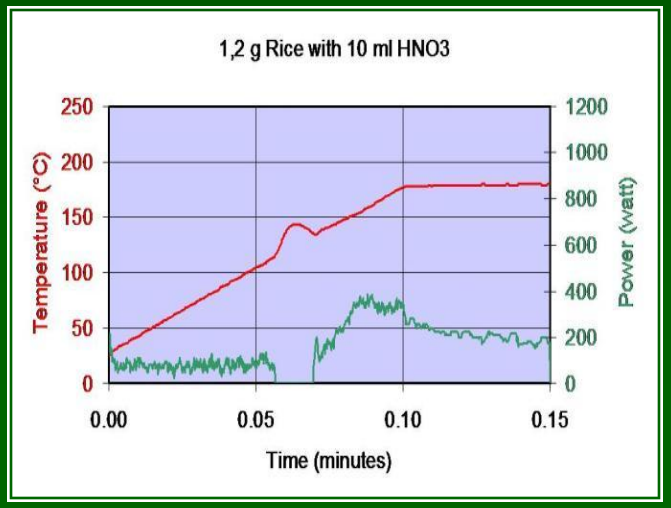
Productivity Enhancement – Temperature Control

- Fiber Optic Probe
- Direct temperature measurement
- PID Control







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Technology Comparison

Soxhlet vs Microwaves

	Method 3540C		Microwave Extraction	
	12 Samples	1000 Samples	12 Samples	1000 Samples
Labor Cost	$140	$11,666	$41	$3,383
Solvent Cost	$108	$9,000	$8.40	$400
Total Cost	$248	$20,666	$49	$3,783
Total Time Required	316 hours	13,167 hours	3.08 hours	257 hours
Total Solvent Consumed	5.4 L	450 L	420 ml	15.96 L

Data Analysis

- Instrument setup
- 15g of Ottawa Sand
- Disposable Glass inserts were used

Run 1:

- 25ml of 1:1 hexane acetone
- 10 min to 100 ^{0}C + 20 min at 100 ^{0}C

Run 2:

- 25 ml of 1:1 DCM acetone
- 10 min to 100 ^{0}C + 20 min at 100 ^{0}C

Further Enhancement – Disposable Glass Vials

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Data Run - 1

- Sample: 15 g soil
- Solvent: 15 ml Hexane + 15 ml Acetone
- Dilution Factor: 1
- Final Volume: 1 ml
- Spikes analyzed in duplicates: Aniline, Phenol, 2-Chlorophenol, 1,4-dichlorobenzene, 1,2-dichlorobenzene, 2-methylphenol, 2-nitrophenol, 2,4-dimethylphenol, 4-chloroanile, 2,4,6-trichlorophenol, 2,4,5-trichlorophenol, 4-nitrophenol, azobenzene, pentachlorophenol, anthracene, fluoroanthene
- Average % Recovery: 78.98%
- Surrogates analyzed: 2-fluorophenol, phenol d-6, nitrobenzene d-5, 2,4,6-tribromophenol, 4-terphenyl – d14
- Average % Recovery: 85.93%

Data Run - 2

- Sample: 15 g soil
- Solvent: 15 ml Hexane + 15 ml Acetone
- Dilution Factor: 1
- Final Volume: 1 ml
- Spikes analyzed in duplicates: Phenol, 2-Chlorophenol, 1,4-dichlorobenzene, 1,2-dichlorobenzene, 2-methylphenol, 2-nitrophenol, 2,4-dimethylphenol, 4-chloroanile, 2,4,6-trichlorophenol, 2,4,5-trichlorophenol, 4-nitrophenol, anthracene, fluoroanthene
- Average % Recovery: 75.45%
- Surrogates analyzed: 2-fluorophenol, phenol d-6, nitrobenzene d-5, 2,4,6-tribromophenol, 4-terphenyl – d14
- Average % Recovery: 75.15%

Advantages of Disposable Glass Vials

- No cleaning step required post extraction
- No need to purchase additional rotors
- Clean environment – low blanks
- Comparable or better recoveries
- Inexpensive

Summary

- Microwave extraction offers significant benefits over traditional extraction techniques.
- Modifications and upgrades are made to enhance productivity – Rotor design, safety features, temperature measurement, software
- The use of disposable glass vials further increase the productivity of a lab – no downtime, no cleaning, great results

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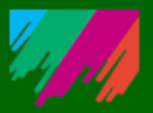
Thank You!

Q&A



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