



A New Technology, Energized Dispersive Extraction, for the Extraction of Semi-Volatile Organic Compounds from Soil



# Environmental Application

## Extraction of Semi-Volatile Organic Compounds from Soil

### EPA Methods

- Soxhlet – US EPA 3540C
- EDGE – US EPA 3545A
- GCMS Analysis – US EPA 8270

### EDGE

- Solvent – Hexane : Acetone (50:50)
- Solvent Volume – 30 mL (25 mL extraction, 5 mL rinse)
- Temperature – Ramp to 100 °C
- Total Time – 5 minutes

### Soxhlet

- Solvent – Hexane : Acetone (50:50)
- Solvent Volume – 100 mL
- Total Time – 16 - 20 hours

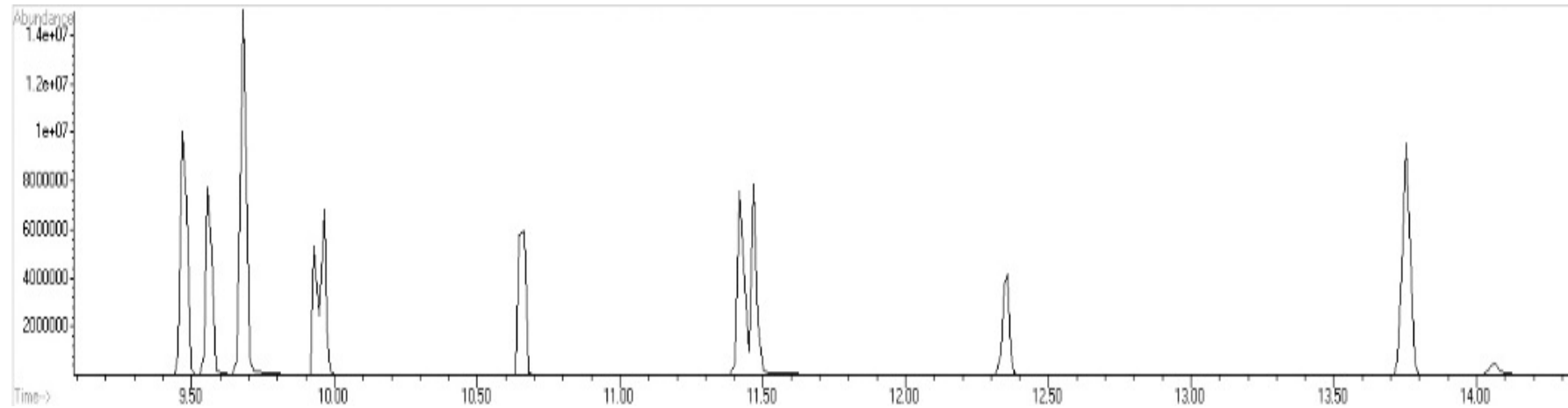
# GCMS Method

- Agilent 7890A GC with a 5986C MSD
- Phenomenex ZB-5MSplus 30 m, 0.25 mm column
- Inlet 250 °C and 1 µL spitless injection
- 0.8 mL/min flow rate using Helium as the carrier gas
- Temperature Profile

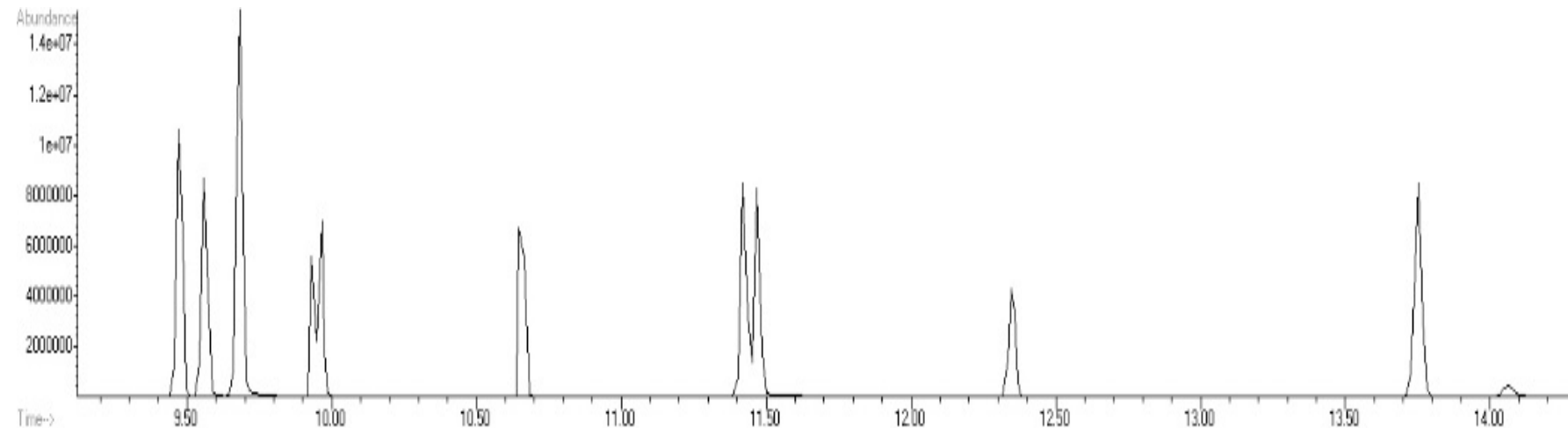
Rate (°C/min)	Temperature (°C)	Hold (min)	Run (min)
	40	0	0
30	260	4	13
6	295	6	26

- Quantitation was based on a 6 point multi-level calibration curve in the concentration interval from 12 ppm to 100 ppm
- Ion Monitoring mode was used to identify each specific compound and from that total area was used for quantifying each peak

# Chromatogram of BNA Compounds



Loam



Clay



# Extracted Soil Samples



# % Recovery Data of Spiked 15 g Soil Samples

Compound	Sand	Loam	Clay
Pyridine	100	88	93
1,4-dichlorobenzene	88	88	96
2-methylphenol	84	95	115
3-methylphenol	90	102	104
hexachlorobutadiene	86	92	97
2,4,6-trichlorophenol	90	105	103
2,4,5-trichlorophenol	89	113	99
2,4-dinitrotoluene	90	102	99
hexachlorobenzene	86	86	81

# % Recovery Data of Spiked 30 g Soil Samples

Compound	Sand	Loam	Clay
Pyridine	78	70	79
1,4-dichlorobenzene	86	95	90
2-methylphenol	83	98	100
3-methylphenol	90	96	91
hexachlorobutadiene	90	91	89
2,4,6-trichlorophenol	93	97	89
2,4,5-trichlorophenol	90	105	82
2,4-dinitrotoluene	80	99	86
hexachlorobenzene	98	85	83

# Recovery Data of 15 g CRM 110-100

Compound	% Soxhlet
2-nitroanaline	94
2,4-dinitrotoluene	114
dibenzofuran	92
fluorene	105
Bis-2-ethylhexyl-phthalate	97

CRM 110-100 purchased from Sigma Aldrich



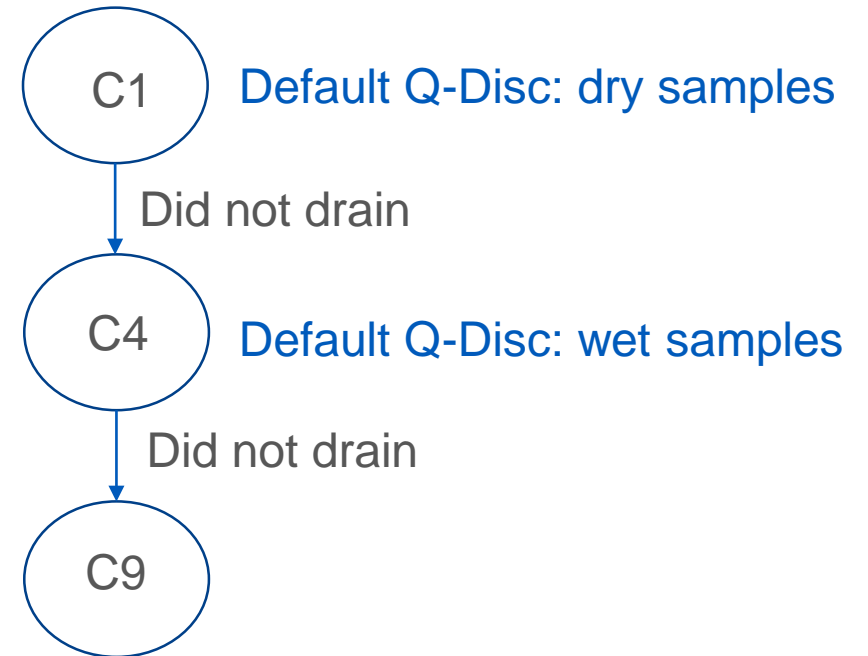
# Environmental Tips



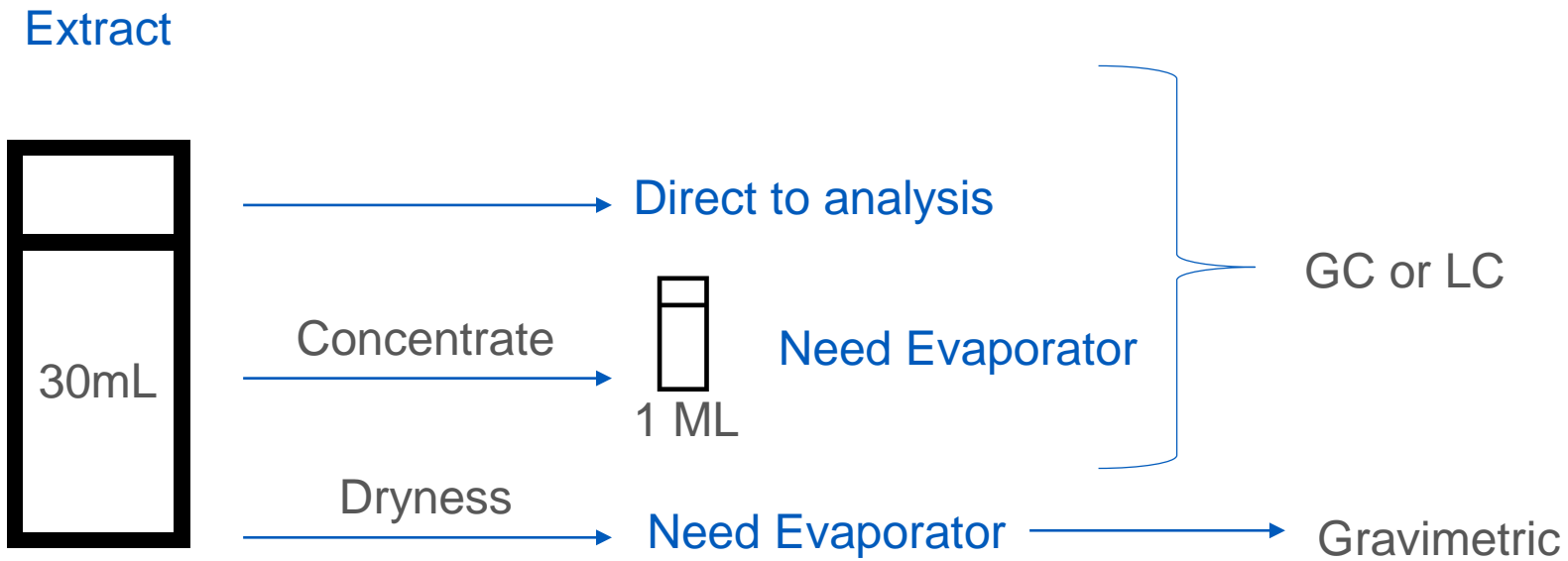
Maximum Fill Line

Add bottom layer of Salt  
for wet samples

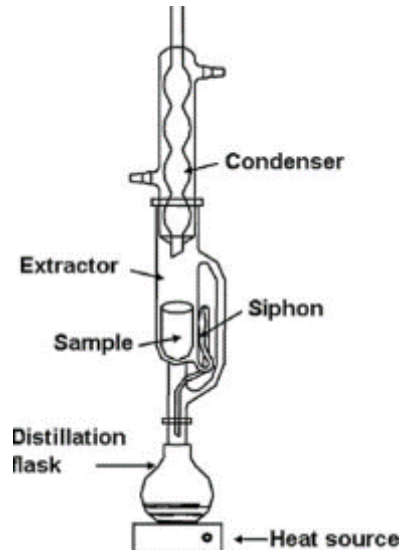
## Q-Disc Selection



# Post Extraction Tips



# Options: Traditional



## Soxhlet

- 150 mL
- 6 hours
- Bank of 8



## Automated Soxhlet

- 90 mL
- 2 hours
- Bank of 6

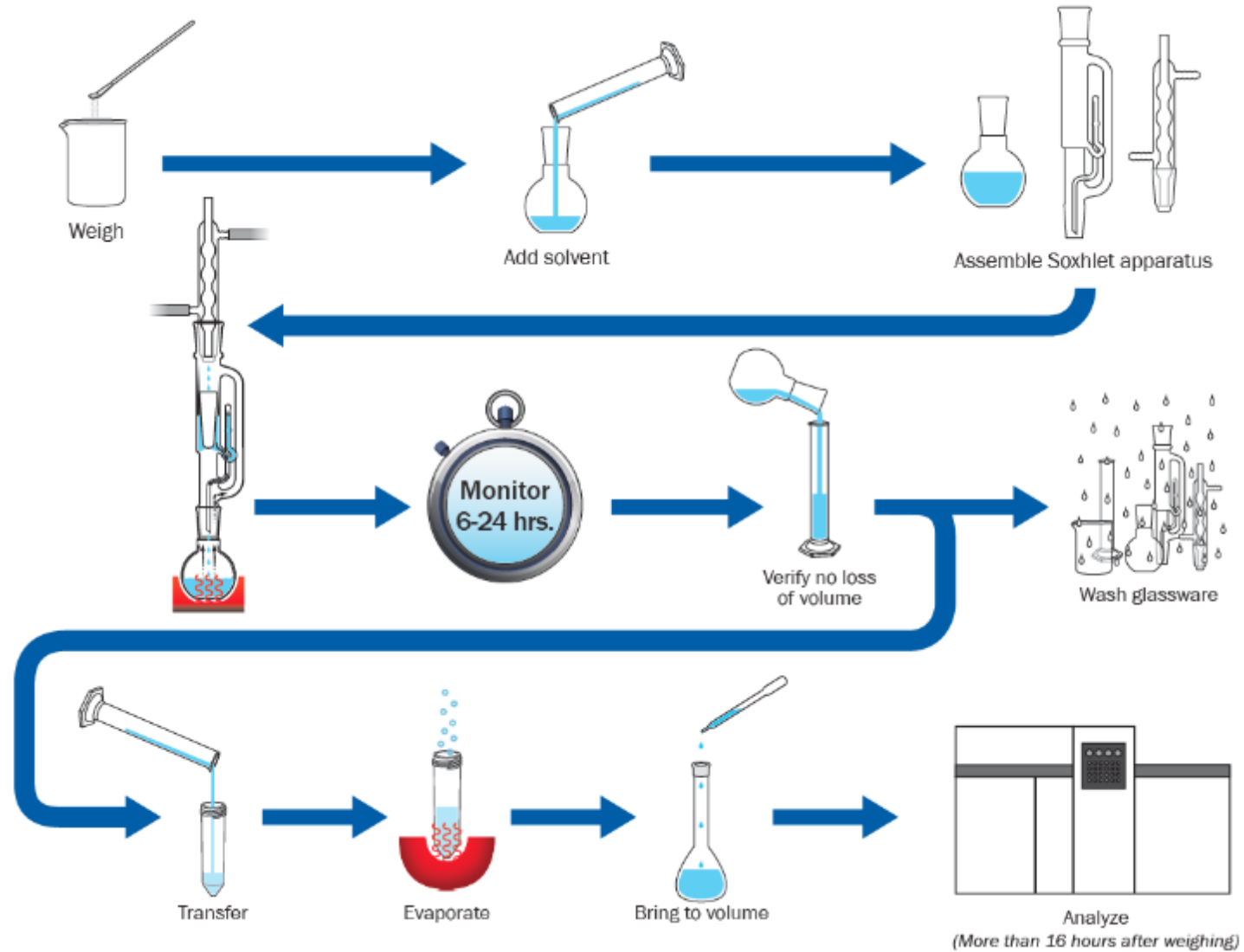


## Ultrasonic

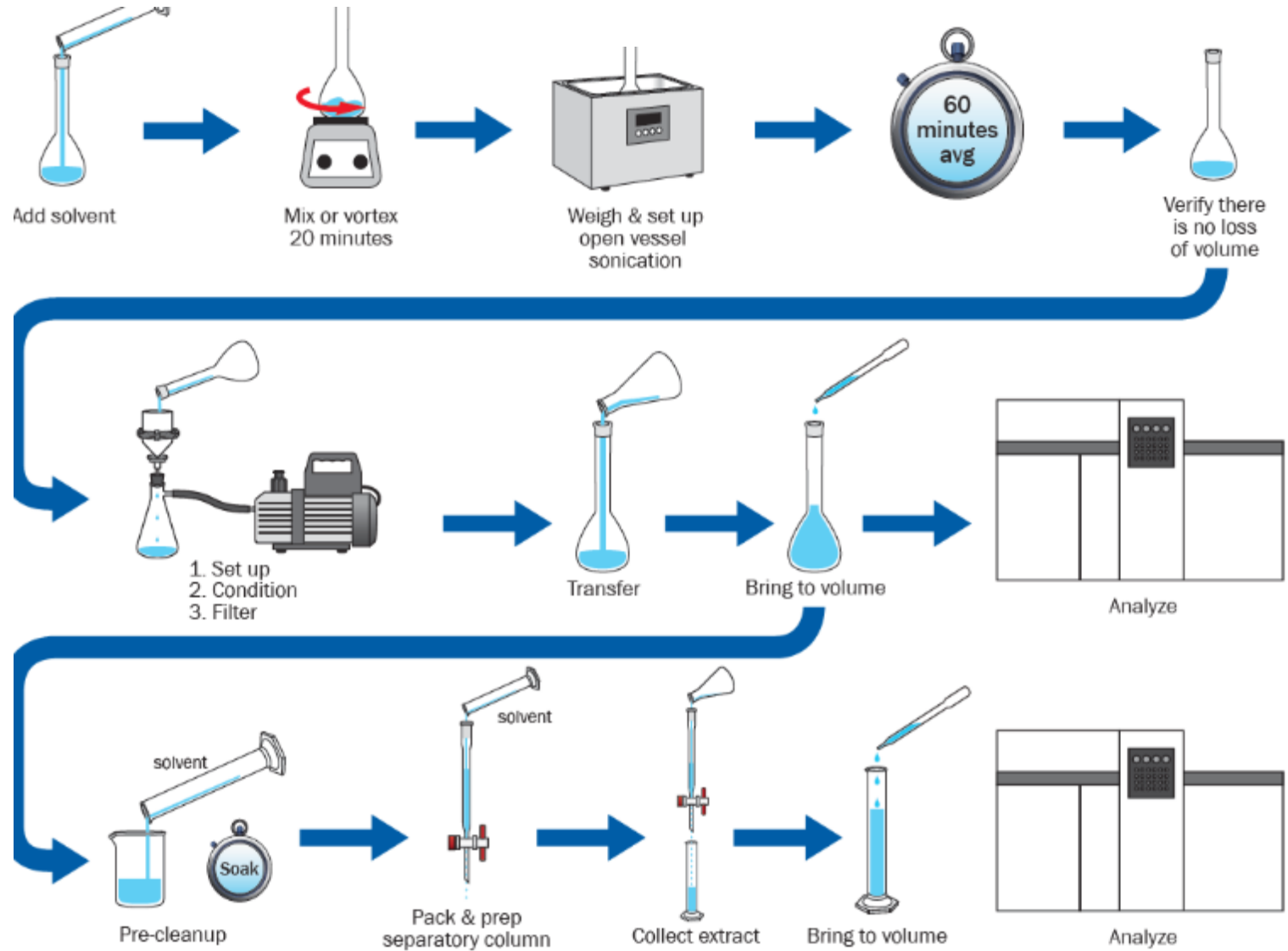
- 300 mL
- 1 hour

Time consuming and lots of solvent usage!

# Soxhlet



# Sonication/Basic Extraction



# Options: Pressurized Fluid Extraction



Dionex ASE  
(Accelerated Solvent Extraction)

- 40 mL
- 30 minutes
- 2 samples per hour



FMS  
(Fluid Management Systems)

- 80 mL
- 22 minutes
- 2 samples per hour

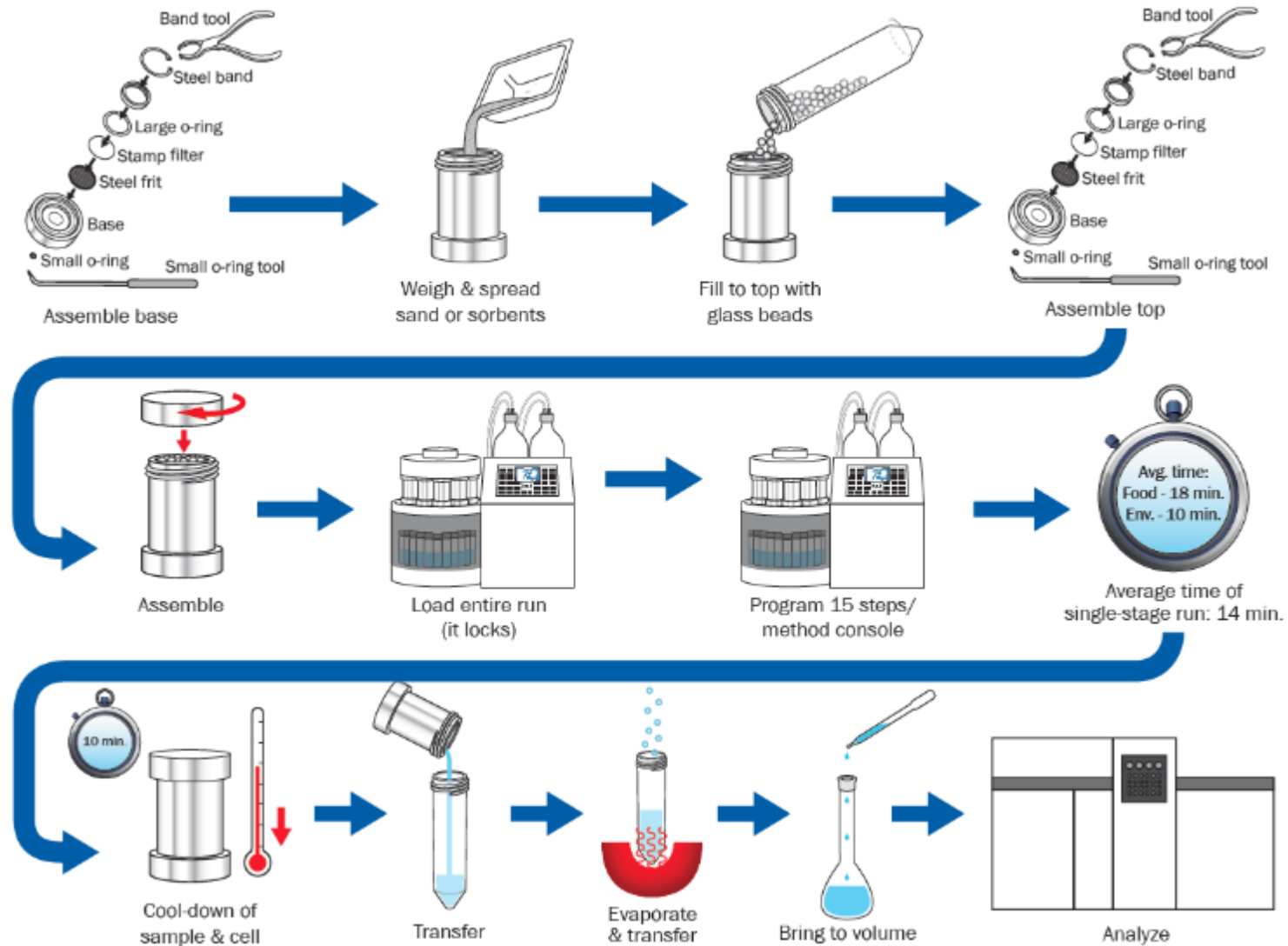


Buchi Speed Extractor

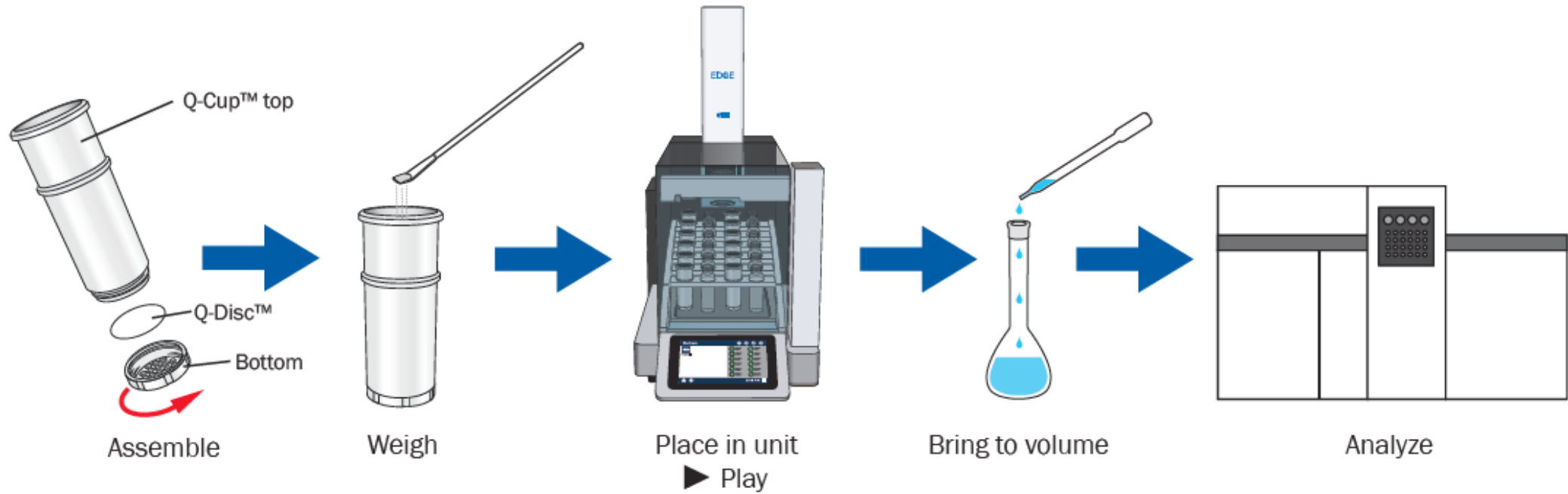
- 60 mL
- 30 minutes
- 12 samples per hour

Large and expensive!

# Pressurized Fluid Extraction



# EDGE Extraction



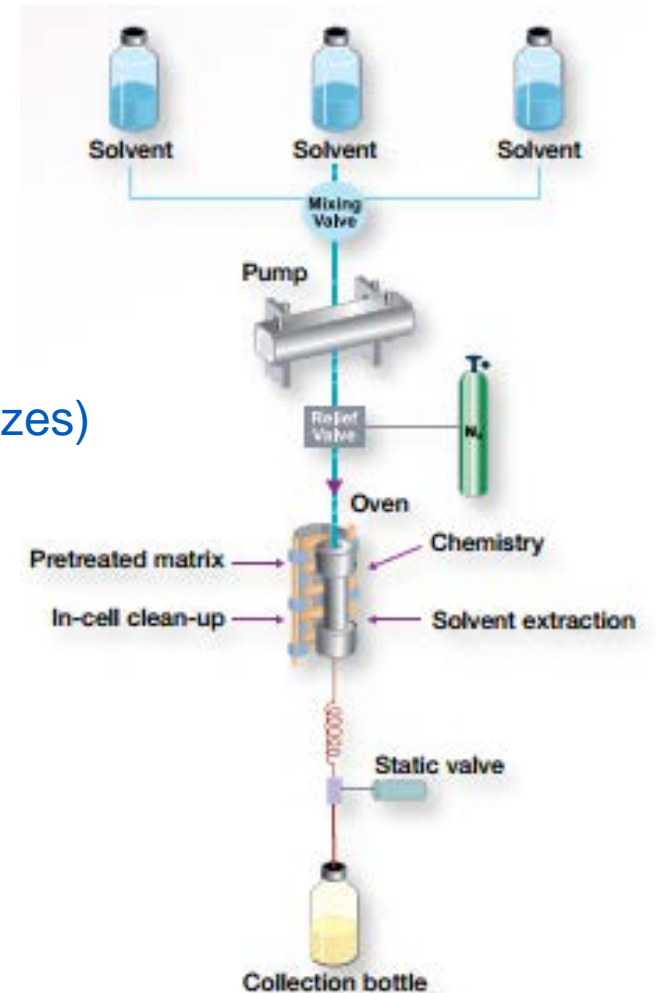


# ASE 350

- Thermo Fisher Scientific Dionex ASE 350 or 150
- Patented (Same for EDGE)
- Solid or semi-solid samples (Same for EDGE)
- Common solvents and elevated temperatures and pressures (Same for EDGE)
- Automatically extract 24 samples (2 EDGE for the price of 1 ASE)
- 1, 5, 10, 22, 34, 66, and 100 mL cell sizes (1 Q-Cup can be used for all samples sizes)

## EDGE Added Features

- 6 solvent lines
- No need for HPLC like pump
- No N<sub>2</sub> required
- Internal thermocouple



# The Difference is Q-Cup Technology

## Q-Cup technology

A simple solution to a complicated problem.

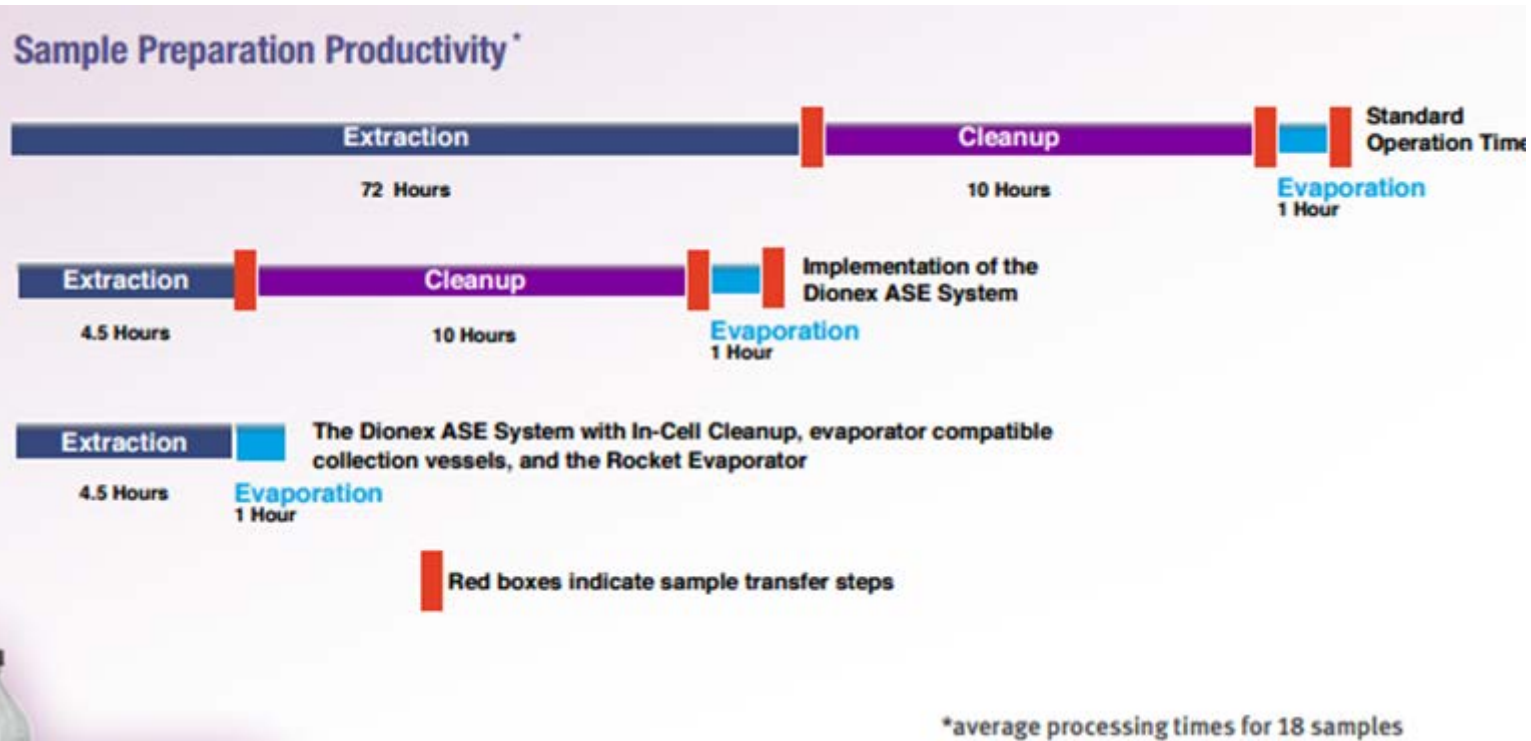
*The Q-Cup™ sample holder is easy to assemble, consisting of just three simple pieces.*

- No packed column required
- No need to run at HPLC like Pressures

# Thermo Fisher Scientific's Comparison

Time Savings	
Technique	Average Extraction Times*
Soxhlet	4–48 h
Automated Soxhlet	1–4 h
Sonication	0.5–1 h
SFE	0.5–2 h
Microwave	0.5–1 h
Dionex ASE 150/350	0.2–0.3 h

Solvent Savings	
Technique	Solvent Usage*
Soxhlet	150–500 mL
Automated Soxhlet	50–100 mL
Sonication	150–200 mL
SFE	5–50 mL
Microwave	25–50 mL
Dionex ASE 150/350	5–200 mL



# 6 Times Faster

## 12 Samples/hr

Includes extraction, rinsing, filtering, cooling & washing

## No Carryover

Technique	Time (minutes)	Solvent Usage (mL)	Cost <sup>1</sup>
EDGE	5	30	\$
QuEChERS	30	30	\$
Pressurized Fluid Extraction	30	35	\$\$\$
Soxhlet	360	150	\$\$
Automated Soxhlet	120	90	\$\$\$
Ultrasonic	60	300	\$\$

<sup>1</sup>Includes instrument cost and running cost



# ASE 350

## **Only extraction time reported in application notes**

Steps not included:

- Sample prep
- Washes between samples
- Heating or cooling
- System set up
- Preheat
- Pressure release
- No access to samples until entire run is complete

# ASE 350: Sample Cell

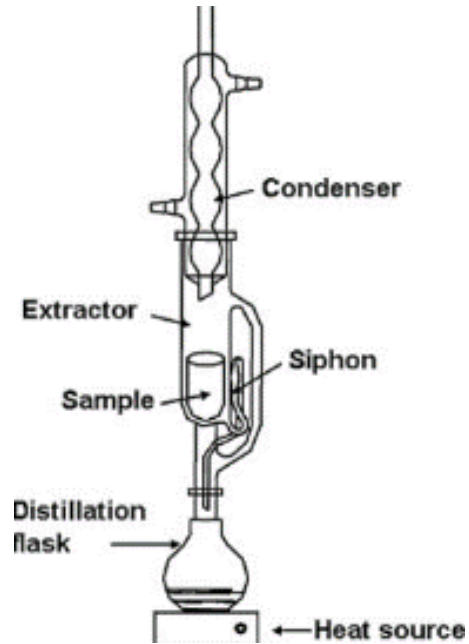
- Each cell contains
  - 1 steel shaft
  - 2 Steel end pieces
  - 2 Tiny rubber o-rings\* (also requires unique tool)
  - 2 Steel frits (10 micron)
  - 2 Large rubber o-rings\*
  - 2 Steel open circular clamps (also requires unique pliers)
  - Enough glass beads to fill the cell void volume

*\*Needs replacement at least every 50 samples run*





# Cost per test comparison



Soxhlet

- \$6.40/test



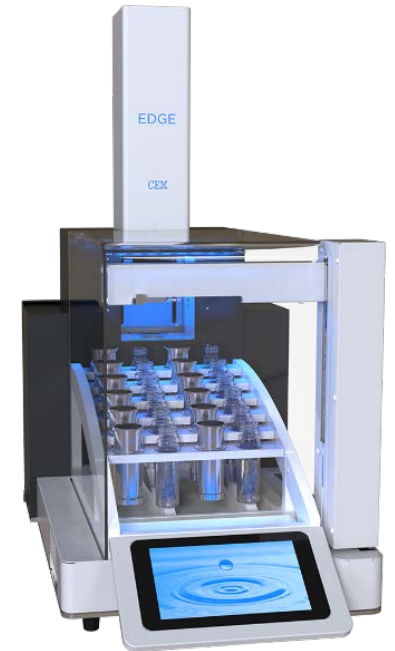
Sonication

- \$.72/test



ASE 350

- \$10.60/test



EDGE

- \$0.75/test



# ASE 350: Issues

## Beyond the obvious drawbacks when compared to EDGE

- Pressures errors due to improper cell construction
- Clogging problems
- Solvent leaking issues
- Dated system, tedious to program





# ASE 350: 52 Application Notes

- **Environmental (15 Application Notes)**
  - Fish, Waste Water, Sludge, Soils, Air Filters, etc.
- **Food (13 Application Notes)**
  - Fats
  - Pesticides/Herbicides in Foods
  - Active Natural Ingredients
  - Acrylamide and Zearalenone Detection
- **Chemicals & Allied Plastics (12 Application Notes)**
  - Plastics/ Textiles
  - Alternative Energy
  - Consumer Products
- **Plant/Herbicides in Animal Tissue (8 Application Notes)**
- **Pharmaceutical/Nutraceutical (3 Application Notes)**

# EDGE | A New World of Sample Preparation

