Automated Sample Preparation Tools in Routine Laboratory Practice

Elsamoul Hamdnalla
Thermo Fisher Scientific
Outline

- Overview Sample Preparation
- Automated Sample Preparation in Ion Chromatography
- Accelerated Solvent Extraction
- Introduction to the ASE 350
- Automated Sample Preparation in GCMS - GCMS/MS by Tri Plus RSH
Workflow for Analysis of Pesticide Residues

Very rarely can analytical samples be analyzed directly without prior treatment.

Sample preparation is a labor-intensive and time-consuming work.
Importance for sample prep in GC&GCMS

- A significant amount of the time spent in a chromatographic analysis is dedicated to sample preparation.
- About 30% of possible sources of error are due to sample processing (sample loss, contamination, modification,..)

Challenges in GC&GCMS sample prep

- Time consuming step in GC&GCMS workflow
- Request of improved analysis speed and precision
- Strong demand for automated sample prep techniques
- Increase sample throughput and labs’ productivity
Benefits of Sample Preparation for GC and GC-MS

Top Problems in Sample Preparation

- Cleanliness
  - Reduce matrix interferences
- Concentrate sample
  - Improves sensitivity
  - Relatively clean matrices
- Compatibility to analysis
  - Improves sensitivity / selectivity
  - Compatibility with separation process, i.e. reduce polarity or increase volatility of analyte
- Ensure system robustness
  - Longer column lifetimes
  - Less maintenance on detector
  - Syringes less likely to block
  - Less contamination

Top problems in sample preparation

*from* Trends in Sample Preparation LCGC North America 2013, vol 31, Issue 3
Automated Sample Preparation in IC … 😊

In the Press…

I'M BORED--YOU KEEP TELLING THE SAME STORIES OVER AND OVER AGAIN!

The Column
21 February 2011 Volume 7 Issue 3
How good is your sample preparation?
When Do You Need Sample Pretreatment?

- No better column for the job
- No alternative detector
- Shortened retention times, poor peak efficiency
- Poor resolution
- Poor reproducibility
- Electrochemical detector electrode fouling
- Irregular baseline
- Interference with analyte signal
- High-Low ratio is too high
Manual Sample Preparation – Cartridges

Interfering Ions
• Poor IC performance
• Short consumables lifetime
• High cost of ownership

Solution – Thermo Scientific™ Dionex™ Matrix Elimination Cartridges
• Eliminate interfering ions
• Automated and manual methods
• Superior IC performance
• Extended lifetime of consumables
• Lower cost of ownership
## Sample Pretreatment Phase Chemistries

<table>
<thead>
<tr>
<th>Phase</th>
<th>Functionality</th>
<th>Retention Mechanism</th>
<th>Application</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Anion-exchange Bicarbonate form</td>
<td>Ion exchange</td>
<td>Remove anions, increase pH</td>
</tr>
<tr>
<td>Ag</td>
<td>Sulfonated resin Silver form</td>
<td>Ion exchange and Precipitation</td>
<td>Remove chloride</td>
</tr>
<tr>
<td>Ba</td>
<td>Sulfonated resin Barium form</td>
<td>Ion exchange and Precipitation</td>
<td>Remove sulfate</td>
</tr>
<tr>
<td>H</td>
<td>Sulfonated resin Acid form</td>
<td>Ion exchange</td>
<td>Remove cations, reduce pH</td>
</tr>
<tr>
<td>M</td>
<td>Iminodiacetate Ammonium form</td>
<td>Chelation</td>
<td>Remove transition metals</td>
</tr>
<tr>
<td>Na</td>
<td>Sulfonated resin Sodium form</td>
<td>Ion exchange</td>
<td>Remove cations, no pH change</td>
</tr>
<tr>
<td>P</td>
<td>Poly-vinylpyrrolidone</td>
<td>H-bonding/ Complexation</td>
<td>Remove phenols, azo dyes, humic acids</td>
</tr>
<tr>
<td>RP</td>
<td>Poly-divinylbenzene</td>
<td>Adsorption</td>
<td>Remove neutral hydrophobics</td>
</tr>
</tbody>
</table>
Determination of TFA in 20% HCl

- **Column:** IonPac AS4A-SC with Guard
- **Eluant:**
  - 1.7 mmol/L NaHCO₃
  - 1.8 mmol/L Na₂CO₃
- **Flow:** 2 mL/min
- **Detection:** Suppressed Conductivity
- **Injection vol.:** 25 µL
Determination of TFA in 20% HCl

**Column:** IonPac AS4A-SC with Guard

**Eluant:**
- 1,7 mmol/L NaHCO₃
- 1,8 mmol/L Na₂CO₃

**Flow:** 2 mL/min

**Detection:** Suppressed Conductivity

**Injection vol.:** 25 µL

**TFA Concentration in original Solution:** 3.7 mg/L
Automated Sample Preparation

- Anions in Organic Solvents
- Anions and Cations in Hydrogen Peroxide
- Anions in Weak Acids
- Anions and Cations in Acids and Bases
- Removing Dissolved Organic Matter
- Automation Using Dionex InGuardCartridges
- Inline Filtration
- pH and Conductivity Measurement
- Summary
Anions from Water Insoluble Drug

Instrument: Thermo Scientific™Dionex™ ICS-3000 and AS
Concentrator: UTAC-ULP1 (5 x 23 mm)
Column: Thermo Scientific™Dionex™ IonPac™ AG15 (mm)/ AS15 (2 mm)
RFIC-EG: KOH Gradient
Temperature: 30°C
Flow Rate: 0.4 mL/min
Inj. Vol.: 100 µL
Detection: Suppressed conductivity
Sample: Drug formulation in Methanol
Procedure: 1. Sample passed through UTAC
2. Anions collected; drug passed to waste
3. UTAC flushed with water from AS to remove MeOH
4. Anions injected onto column
Automated Sample Preparation in GC/LC

The Challenge for Analysis

1.5 mL GC Autosampler Vial

How do we get analytes out of these samples?
The Answer is Sample Preparation

• Extraction
  • Removes analytes from the sample
  • Eliminates compounds that interfere with the analysis (Clean Up)

• Evaporation
  • Concentrates extracted analytes for analysis
  • Evaporates extracted samples for re-constitution

• Most time consuming part of analytical procedure (>60%)*

• Single largest source of errors in the workflow (>30%)**

Extraction Techniques

- Liquid Liquid extraction
- Solid liquid extraction (Quechers)
- Pressurized extraction (ASE)
- Heated extraction – soxhlett

**Scope:**
- Extracting of target analytes from matrix with high recovery
- Removal of matrix
Now . . . Accelerated Solvent Extraction - ASE™

- Automates sample preparation for solid and semisolid samples using solvents at elevated temperatures

- Operates above the boiling point of extraction solvents by using elevated pressure

- Walk-away system that extracts and clean up to 24 samples unattended

- Well established and proven technique that is superior to Soxhlet and approved for U.S. EPA Method 3545A

Thermo Scientific™ Dionex™ ASE™ 350 Accelerated Solvent Extractor system
How does Accelerated Solvent Extraction work?

- Cell loaded into oven: 1 min
- Fill, heat, equilibrate: 5-9 min
- Static extraction: 3-5 min
- Fresh solvent rinse: 0.5 min
- Nitrogen purge: 1-2 min
- Filtered extract: 15-20 min

Total Time: 1-2 min

Diagram:
- Solvent
- Mixing Valve
- Pump
- Relief Valve
- Oven
- Pretreated matrix
- In cell clean-up
- Solvent extraction
- Static valve
- Collection Bottle

Diagram label:
- Static cycle

Diagram organization:
- ThermoFisher Scientific
ASE in-line Clean Up + Use of Adsorbents Improves Selectivity

Schematic of the Cell

Preparation of the Extraction Cell for the Selective Extraction of PCBs from Fish Meal

Preparation of the Extraction Cell for the Selective Extraction of Perchlorate from Vegetation
### ASE in-line Cleanup

<table>
<thead>
<tr>
<th>Adsorbent and Uses</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbon</td>
<td>Removes organics and nonpolar compounds</td>
</tr>
<tr>
<td>Copper</td>
<td>Removes sulfur</td>
</tr>
<tr>
<td>Ion-exchange Resins</td>
<td>Removes organics, ionic interferences for IC and IC/MS analysis</td>
</tr>
<tr>
<td>C\textsubscript{18} Resin</td>
<td>Removes organics, polar compounds, lipids, colors</td>
</tr>
<tr>
<td>Acid-impregnated Silica Gel</td>
<td>Removes lipids</td>
</tr>
<tr>
<td>Alumina</td>
<td>Removes nonpolar lipids, colors</td>
</tr>
<tr>
<td>Florisil</td>
<td>Removes nonpolar lipids</td>
</tr>
<tr>
<td>Silica Gel</td>
<td>Removes nonpolar lipids</td>
</tr>
<tr>
<td>Country</td>
<td>Method/Standard Description</td>
</tr>
<tr>
<td>-------------</td>
<td>-------------------------------------------------------------------------------------------</td>
</tr>
<tr>
<td>United States</td>
<td>U.S. EPA Method 3545A (OCP, OPP, BNA, TPH, PCDD, herbicides and semi-volatiles)</td>
</tr>
<tr>
<td></td>
<td>U.S. EPA Method 8267 (Toxaphene)</td>
</tr>
<tr>
<td></td>
<td>U.S. EPA Method 6860 (Perchlorate)</td>
</tr>
<tr>
<td></td>
<td>NOAA Method NWFS-NWFSC-59 (Hydrocarbons)</td>
</tr>
<tr>
<td></td>
<td>ASTM D-7210 (Polymer Additives)</td>
</tr>
<tr>
<td>China</td>
<td>Method GB/T 19649-2006 for 475 pesticides in grains and grain products</td>
</tr>
<tr>
<td></td>
<td>Method GB/T 23376-2009, pesticides in tea leaves</td>
</tr>
<tr>
<td></td>
<td>Method GB/T22996-2008, ginsenosides in ginseng</td>
</tr>
<tr>
<td>Mexico</td>
<td>National Standard NMX-AA-146-SCFI-2008 for PAHs in soils and sediments</td>
</tr>
<tr>
<td>Germany</td>
<td>Method L00.00-34 for pesticides in foodstuffs</td>
</tr>
</tbody>
</table>
## Global Industries Using ASE

<table>
<thead>
<tr>
<th>Water Treatment Plants</th>
<th>Rubber &amp; Polymers</th>
<th>Natural Products</th>
</tr>
</thead>
<tbody>
<tr>
<td>Contract Laboratories</td>
<td>Government Agencies</td>
<td>Dietary Supplements</td>
</tr>
<tr>
<td>Biotech/Pharmaceutical</td>
<td>Food and Beverage</td>
<td>Thermo Fisher Scientific</td>
</tr>
<tr>
<td>Industry</td>
<td>Analyte</td>
<td>Determinative Step</td>
</tr>
<tr>
<td>-------------------</td>
<td>--------------------------------------</td>
<td>--------------------</td>
</tr>
<tr>
<td>Environmental</td>
<td>Polyaromatic Hydrocarbons (PAHs)</td>
<td>GC-MS</td>
</tr>
<tr>
<td></td>
<td>Polychlorinated Biphenyls (PCBs)</td>
<td>GC-ECD</td>
</tr>
<tr>
<td></td>
<td>Dioxins and Furans</td>
<td>GC-MS/MS</td>
</tr>
<tr>
<td></td>
<td>Total Petroleum Hydrocarbons (TPH)</td>
<td>GC-FID</td>
</tr>
<tr>
<td>Base, Neutral, Acids (BNAs)</td>
<td>GC-MS</td>
<td></td>
</tr>
<tr>
<td>Food</td>
<td>Fat Content</td>
<td>Gravimetric</td>
</tr>
<tr>
<td></td>
<td>Oil Content</td>
<td>Gravimetric</td>
</tr>
<tr>
<td></td>
<td>Pesticide Residues</td>
<td>GC-MS</td>
</tr>
<tr>
<td></td>
<td>Acrylamide</td>
<td>LC-MS</td>
</tr>
<tr>
<td>Natural Products</td>
<td>Herbal Marker Compounds</td>
<td>LC-UV</td>
</tr>
<tr>
<td></td>
<td>Active Ingredients in Herbal Supplements</td>
<td>LC-UV</td>
</tr>
<tr>
<td>Chemical</td>
<td>Polymer Additives</td>
<td>LC-UV</td>
</tr>
<tr>
<td></td>
<td>Bioalcohol</td>
<td>Gravimetric</td>
</tr>
<tr>
<td>Pharma</td>
<td>Leachables &amp; Extractables</td>
<td>LC-MS/MS</td>
</tr>
<tr>
<td></td>
<td>Active Ingredients</td>
<td>LC-UV</td>
</tr>
</tbody>
</table>
### Comparison to Other Techniques (U.S. EPA Methods)

<table>
<thead>
<tr>
<th>U.S. EPA Method</th>
<th>Technique</th>
<th>Solvent Used Per Sample</th>
<th>Extraction Time Per Sample</th>
</tr>
</thead>
<tbody>
<tr>
<td>3545A</td>
<td>ASE</td>
<td>15 – 45 mL</td>
<td>10-15 min</td>
</tr>
<tr>
<td>3540</td>
<td>Soxhlet</td>
<td>300 – 500 mL</td>
<td>18 hours</td>
</tr>
<tr>
<td>3541</td>
<td>Automated Soxhlet</td>
<td>50 mL</td>
<td>2 hours</td>
</tr>
<tr>
<td>8151</td>
<td>Shaker</td>
<td>300 mL</td>
<td>2 hours</td>
</tr>
<tr>
<td>3546</td>
<td>Microwave*</td>
<td>25 mL</td>
<td>15 min</td>
</tr>
</tbody>
</table>

*Requires additional cooling and filtering steps (~ 45 min/sample)
Selecting The Best “Automated” Sample Handling and Introduction Technique

- Tri Plus RSH
- Liquid HS
- ITEX
- SPME
- Arrow Prep

- Tri Plus 300 HS
- Valve & Loop

- Purge & Trap

- Thermal Desorption
XZY Fully Scalable Robotic Autosampler for Routine and Research

- Large Volume Wash Station
- Large Solvent Station
- Vortexer for Intensive sample mix
- Barcode reader for sample tracking
- Peltier or liquid heating or cooling of samples
- More sample trays for higher capacity
- Agitator and heated oven for HS, SPME or sample prep

- XYZ robotic, fully scalable autosampler
  - Liquid
  - Headspace
- SPME & SPME Arrow
- ITEX-DHS
- Dedicated prepcycles
- Large capacity sample trays
- Bar Code Reader
- No carryover
Unattended Syringe/Tool Exchange During a Sequence

**Liquid**
- Magnetic transportation for 2 ml vials
- Large magnetic ring for 10/20 ml Vials

**Headspace**
- Syringe temperature: 40°C up to 50°C, in 1°C steps
- Syringe flush with inert gas flow through X-Y-Z axis

**SPME**
- SPME syringe fiber tool includes a combination of 4 different fibers for starting-up

**ITEX-DHS**
- TriPlus RSH is compatible with a wide range of syringe volumes & tools
- ATC station enables unattended syringe exchange during a sequence
- ATC (Automatic Tool change) Station for automatic recognition and syringe exchanges during operation

**Other Features**
- Magnetic transportation for 2 ml vials
- Large magnetic ring for 10/20 ml Vials
- Syringe temperature: 40°C up to 50°C, in 1°C steps
- Syringe flush with inert gas flow through X-Y-Z axis
- SPME syringe fiber tool includes a combination of 4 different fibers for starting-up
- Magnetic transportation for 2 ml vials
- Large magnetic ring for 10/20 ml Vials
- Syringe temperature: 40°C up to 50°C, in 1°C steps
- Syringe flush with inert gas flow through X-Y-Z axis
- SPME syringe fiber tool includes a combination of 4 different fibers for starting-up
- Magnetic transportation for 2 ml vials
- Large magnetic ring for 10/20 ml Vials
- Syringe temperature: 40°C up to 50°C, in 1°C steps
- Syringe flush with inert gas flow through X-Y-Z axis
- SPME syringe fiber tool includes a combination of 4 different fibers for starting-up
Why is Headspace GC a Good Sampling Technique?

- Sample preparation technique that extracts and concentrates the compounds of interest from unwanted nonvolatile material.
- Excellent for qualitative or quantitative analysis of volatile species in liquids and solids.
- Viscous liquids are particularly appropriate for headspace analysis.
- Liquids which contain high boiling or insoluble components:
  - Blood, Paint, or Adhesives.
  - Volatile Organic Compounds (VOC) from wastewater and contaminated land samples.
  - Residual solvents in packaging and pharmaceuticals.
  - Blood alcohol and toxicology screening.
  - Aroma components from food and beverages.
- Easy to Use, Time & cost efficient with ability to reproducibly automate.
- Assay a variety of sample matrices.
- The solvent chosen must firstly completely dissolve the sample and analytes of interest.
Unwanted Non-Volatile Material Stays Behind

Syringe temperature: 40°C up to 150°C, in 1°C steps
Syringe flush with inert gas flow through X-Y-Z axis

Step 1
Sample reaches equilibrium

Step 2
Sample is extracted from headspace

Step 3
Sample is injected
**Principle of Static Headspace**

- **Equilibrium**
- **Response**
- **Time**

\[
C_m = KC_g
\]

Or:

\[
K = \frac{C_m}{C_g}
\]

*K* is the measure of analyte distribution between phases under specific conditions.

*Cm* = concentration matrix and *Cg* = concentration in gas.

When the equilibrium is reached, *K* is constant.

---

**Key Points**

- *Cm* and *Cg* represent concentrations in the matrix and gas phases, respectively.
- *K* is the equilibrium constant, indicating the proportionality between the concentrations in the matrix and gas phases.
- The principle is used in static headspace analysis to quantify analytes in a sample matrix through gas phase analysis.

---

**ThermoFisher Scientific**

31
Rapid and efficient sample enrichment of volatile and semi-volatile compounds from solid, liquid and gaseous samples
ITEX Dynamic Headspace

...Simplicity and handiness

- Easy sample handling
- No loop, transfer line, or switching valve
- Easy access to the trap and syringe

Straightforward troubleshooting and maintenance

Minimum instrument downtime

ITEX Dynamic Headspace Specifications

<table>
<thead>
<tr>
<th>Specification</th>
<th>Range/Details</th>
</tr>
</thead>
<tbody>
<tr>
<td>Trap temperature range</td>
<td>40 °C - 350 °C</td>
</tr>
<tr>
<td>Syringe temperature range</td>
<td>40 °C - 150 °C</td>
</tr>
<tr>
<td>Extraction flow rates</td>
<td>10 µL/s - 1000 µL/s</td>
</tr>
<tr>
<td>Extraction stroke cycles (strokes/sample)</td>
<td>0 - 1000</td>
</tr>
<tr>
<td>Extraction volume</td>
<td>0 µL - 1300 µL</td>
</tr>
<tr>
<td>Incubation time</td>
<td>up to 600 min</td>
</tr>
<tr>
<td>Standard adsorption material</td>
<td>Tenax TA 80/100 mesh</td>
</tr>
<tr>
<td>Water removal step with adjustable trap temperatures</td>
<td>40 °C - 150 °C</td>
</tr>
<tr>
<td>Ventilated trap for quick sample processing</td>
<td>Fast cooling of trap &lt;2 minutes</td>
</tr>
</tbody>
</table>
Purge & Trap sensitivity without the pitfalls of P&T systems

10 ng/L MegaMix Standard (Restek PN 30432, EPA 502.2, 54 components) and subsequent Blank Injection

EPA 502.2: VOC’s in water, Purge & Trap Sensitivity without the problems of Purge & Trap (carryover, contamination, foaming)

Syringe-only concept: No sample loops, seals, connectors, transfer lines, or switching valves that could be contaminated

• No transfer line → no limitation to a dedicated injection port
• Rapid transfer of the compounds in a narrow band to the GC, GCMS system
• Compatible with any injector type
## ITEX Dynamic Headspace – Repeatability data

### VOCs in water, 50 ppb - Repeatability of 9 replicated extractions

<table>
<thead>
<tr>
<th></th>
<th>Benze ne</th>
<th>Tolue ne</th>
<th>Ethyl benzene</th>
<th>o-Xylen e</th>
<th>Styre ne</th>
<th>Brom o benze ne</th>
<th>1,2,4 trimethyl benzene</th>
<th>1,3,5 trimethyl benzene</th>
<th>n-buthyl benzene</th>
<th>P-isopropyl toluene</th>
<th>1,2,4 trichloro benzene</th>
<th>1,2,3 trichloro benzene</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.35</td>
<td>0.44</td>
<td>0.47</td>
<td>0.47</td>
<td>0.37</td>
<td>0.15</td>
<td>0.37</td>
<td>0.36</td>
<td>0.27</td>
<td>0.18</td>
<td>0.04</td>
<td>0.05</td>
</tr>
<tr>
<td>2</td>
<td>0.35</td>
<td>0.45</td>
<td>0.49</td>
<td>0.48</td>
<td>0.38</td>
<td>0.15</td>
<td>0.39</td>
<td>0.37</td>
<td>0.28</td>
<td>0.19</td>
<td>0.04</td>
<td>0.05</td>
</tr>
<tr>
<td>3</td>
<td>0.35</td>
<td>0.46</td>
<td>0.49</td>
<td>0.49</td>
<td>0.38</td>
<td>0.15</td>
<td>0.39</td>
<td>0.38</td>
<td>0.29</td>
<td>0.19</td>
<td>0.04</td>
<td>0.05</td>
</tr>
<tr>
<td>4</td>
<td>0.35</td>
<td>0.45</td>
<td>0.49</td>
<td>0.48</td>
<td>0.38</td>
<td>0.15</td>
<td>0.39</td>
<td>0.37</td>
<td>0.28</td>
<td>0.18</td>
<td>0.04</td>
<td>0.05</td>
</tr>
<tr>
<td>5</td>
<td>0.35</td>
<td>0.46</td>
<td>0.49</td>
<td>0.49</td>
<td>0.38</td>
<td>0.15</td>
<td>0.39</td>
<td>0.38</td>
<td>0.29</td>
<td>0.19</td>
<td>0.04</td>
<td>0.05</td>
</tr>
<tr>
<td>6</td>
<td>0.35</td>
<td>0.46</td>
<td>0.49</td>
<td>0.48</td>
<td>0.38</td>
<td>0.15</td>
<td>0.39</td>
<td>0.37</td>
<td>0.28</td>
<td>0.19</td>
<td>0.04</td>
<td>0.05</td>
</tr>
<tr>
<td>7</td>
<td>0.35</td>
<td>0.46</td>
<td>0.49</td>
<td>0.48</td>
<td>0.38</td>
<td>0.15</td>
<td>0.39</td>
<td>0.37</td>
<td>0.29</td>
<td>0.19</td>
<td>0.04</td>
<td>0.05</td>
</tr>
<tr>
<td>8</td>
<td>0.35</td>
<td>0.46</td>
<td>0.49</td>
<td>0.48</td>
<td>0.38</td>
<td>0.15</td>
<td>0.39</td>
<td>0.37</td>
<td>0.28</td>
<td>0.18</td>
<td>0.04</td>
<td>0.05</td>
</tr>
<tr>
<td>9</td>
<td>0.36</td>
<td>0.46</td>
<td>0.49</td>
<td>0.48</td>
<td>0.38</td>
<td>0.15</td>
<td>0.39</td>
<td>0.37</td>
<td>0.29</td>
<td>0.19</td>
<td>0.04</td>
<td>0.05</td>
</tr>
<tr>
<td>Avg</td>
<td>0.35</td>
<td>0.45</td>
<td>0.49</td>
<td>0.48</td>
<td>0.38</td>
<td>0.15</td>
<td>0.39</td>
<td>0.37</td>
<td>0.28</td>
<td>0.19</td>
<td>0.04</td>
<td>0.05</td>
</tr>
<tr>
<td>Std Dev</td>
<td>0.00</td>
<td>0.01</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.01</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>RSD %</td>
<td>0.78</td>
<td>1.17</td>
<td>1.43</td>
<td>1.32</td>
<td>0.92</td>
<td>0.74</td>
<td>1.36</td>
<td>1.12</td>
<td>1.58</td>
<td>1.51</td>
<td>0.98</td>
<td>0.95</td>
</tr>
</tbody>
</table>

![Graph showing VOCs in water, 50 ppb - Repeatability of 9 replicated extractions](image.png)
IEX Dynamic Headspace Benefits

- **Ease to use and automated**
  - Rapid and efficient sample enrichment of volatile and semi-volatile compounds from solid, liquid and gaseous samples
  - Determination of VOCs and semi-VOCs at “P&T” limit of detection

- **Scalable sensitivity, from screening to trace analysis**
  - Combination with different injection techniques on the same platform
  - Adapt the best injection mode to sample requirements
  - Single or multi-layer microtraps use industry standard sorbent materials

- **Exploitation of unattended injection mode change during a sequence**
  - ATC (Automatic Tool Change) Station for automatic recognition and syringe exchanges during operation
    - Intelligent Sequence operation of Chromleon CDS

- **Exploitation of combined advanced sample handling and sample preparation capabilities**
  - Dilution, derivatization, standard addition, internal standard additions, etc.
SPME Technology

- Developed by Pawliszyn and co-workers in 1990
- Solvent free sample preparation technique uses a fused silica fiber coated with a stationary phase attached to a modified microsyringe
- Suitable for both headspace and direct-immersion sampling
- Enables isolation of volatile and semi-volatile analytes
  - reduced solvent use
  - sampling + extraction in 1 step
  - small sample size
  - wide range of matrices

Fast Simple Cheap

ThermoFisher Scientific
Features and Benefits of SPME

**SPME advantages...**

- High sample recovery
- Quantitative recovery
- Good linearity and precision in a wide concentration range
- Flexibility and selectivity
- No sample handling and complete automated
- Limited investment, no extra space needed
- No solvent cost and disposal

**...and benefits to the customer**

- High method sensitivity, in the ppt/ppb range
- Wide application range
- Lower cost/analysis
- High sample throughput and productivity
SPME Applications

Environmental
- VOCs in water & soil
- PAHs, PCBs in drinking water & soil
- BTEX, gasoline in water & soil
- Phenols, aromatic amines in water
- MTBE in water
- 2-MIB and geosmin in water & soil
- Chemical warfare agents in water
- Herbicides, Pesticides in water & soil
- Fatty Acid in water & sludges

Food/Beverages/Flavor
- Flavor profiling in food & beverages
- VOC in fruit juices, fruit and vegetables
- Sulfide and Disulfide in wine and beer
- TCA in wine
- Pesticides in food
- Caffeine in tea, coffee
- Off-flavor in food & beverages

Industrial
- Solvents in polymers
- Solvents in water-based coatings
- Acetaldehyde in PET bottles
- Residual solvents in packaging

Natural product/Drugs/Pharma
- Terpenoids in herbs, essential oils
- Phenols, volatiles, flavors in tobacco
- Pheromones
- Pesticides in natural products
- Residual Solvents in pharma products
Off-odor compounds in water, headspace extraction

Relative Headspace extraction efficiency (measured as amount extracted after 30min) for off-flavor compounds in water at 100 ng/L with DVB fibers. 100 μm DVB SPME Arrow compared to 100 μm DVB SPME Fiber.
Total Workflow with Fast Simplified Sample Prep

Integrated Workflow Solutions for Sample Preparation
Where Can I Find Out More?

Customer Testimonials

Educational Webinars

Three Year Warranty

Cost Savings Calculator

Application Notes & Brochures

www.thermofisher.com/samplepreparation
Thank You!

Elsamoul.hamdnallamohammed
@thermofisher.com