Vacuum Assisted Sorbent Extraction (VASE) Thermal Desorption-GC-MS: A Robust, Solvent-Free Technique for Chemical Residue Analysis

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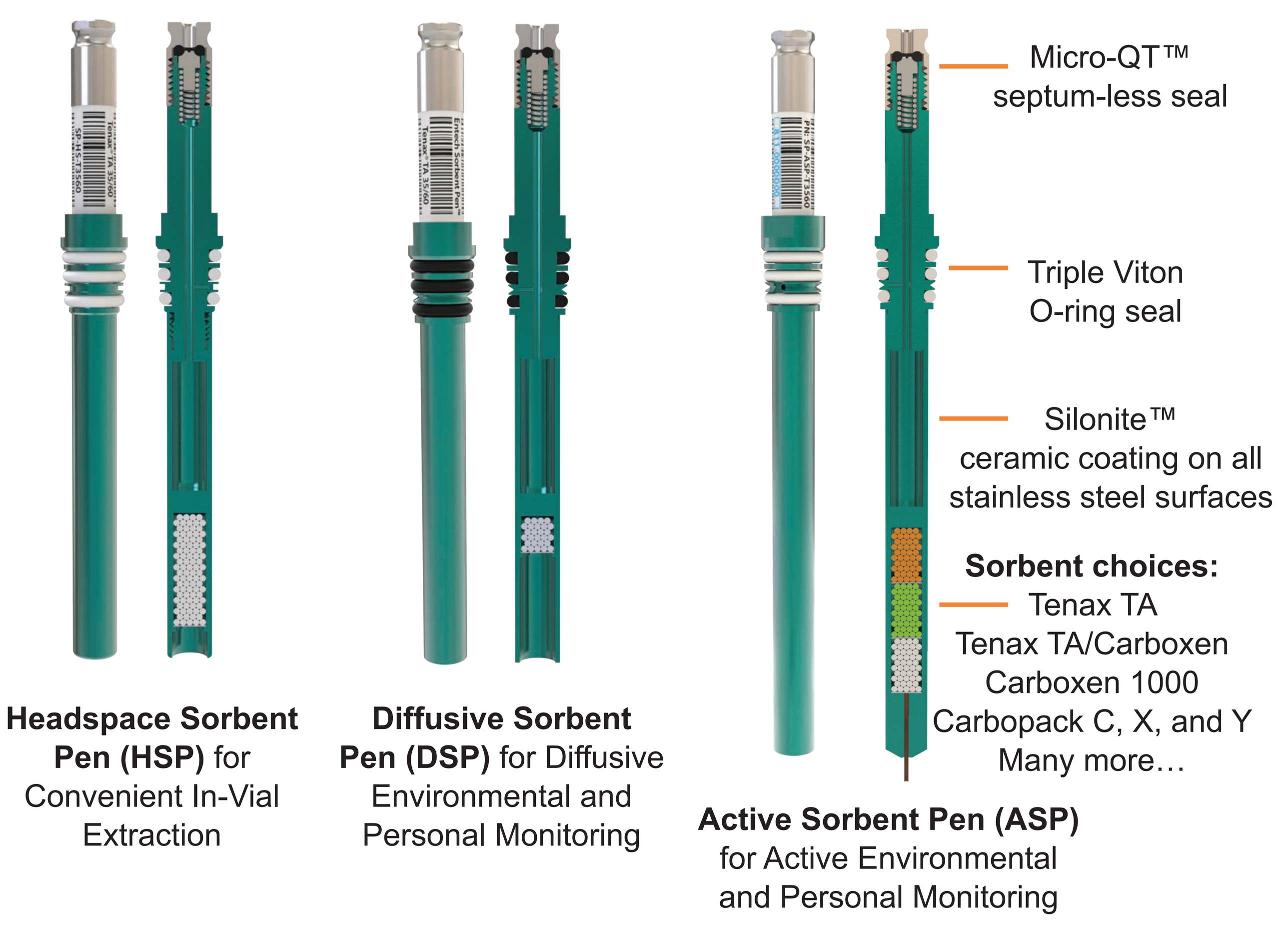
Overview

- In the 30 years since the invention of solid phase microextraction (Belardi & Pawliszyn, 1989), headspace (HS) extraction has developed into a powerful family of techniques for both targeted and untargeted chemical analysis of volatile and semi volatile compounds.
- We recently developed a static HS sampling approach, termed **vacuum assisted sorbent extraction (VASE)**, in which the sample is positioned in a disposable sample vial and placed in a reduced pressure environment with a packed **headspace sorbent pen (HSP)**.
- Here we describe the methodology behind VASE as well as the application of the technique to examine the chemical composition of a wide assortment of liquid and solid samples.
- Example applications are shown in the areas of environmental contamination, cannabis consumer products (e.g., edibles, flower), forensics, and alcoholic beverages.

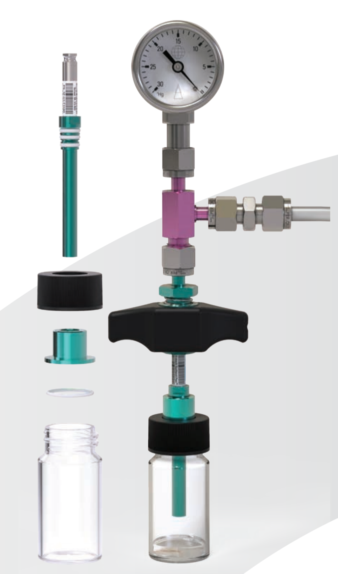
Sorbent Pen Technology



Sorbent Pen (SP) schematic, showing the Micro-QT vacuum seal and the internal sorbent bed. The SPs come in three different configurations and can be packed with a variety of sorbents, including multi-component beds of varying physical properties.

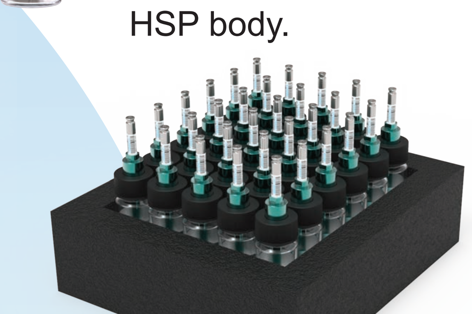
Vacuum Assisted Sorbent Extraction (VASE) & Thermal Desorption GC-MS

Step 1 Sample Preparation & Vial Evacuation
The sample is placed in a glass vial and brought under vacuum (~1/30 atm) using a 2-stage diaphragm pump. Sample vials from 2 - 1000 mL are available however the typical sample size is less than 1 mL. Reduced pressure extraction helps to promote less volatile compounds into the headspace for capture on the sorbent bed.

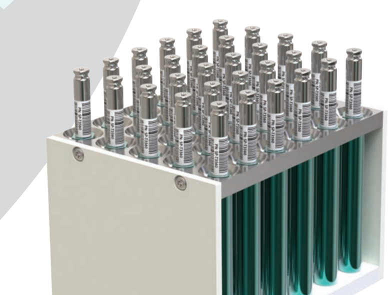


Step 2 Diffusive Extraction with Optional Agitation and Heat
The samples are heated and agitated while under vacuum. Diffusive headspace extraction conditions promote analyte adherence to the front of the sorbent bed, prevents volatile compound breakthrough, enables flash desorption, and promotes tight chromatography. Typical extractions occur in less than 24 h.

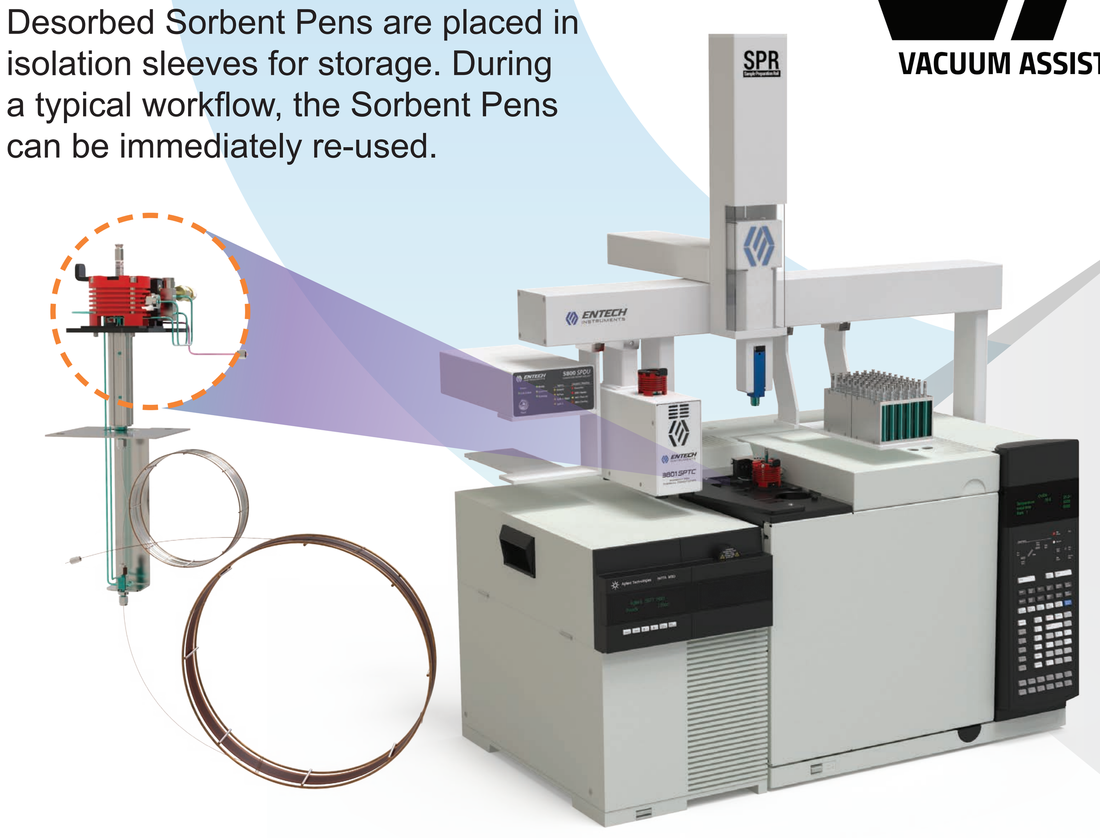
Step 3 Water Management
The sample vials are placed onto a chilled block to remove moisture from the sorbent bed and HSP body.



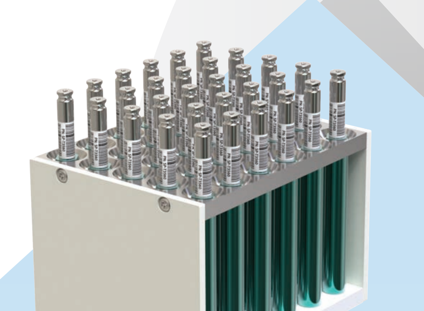
Step 4 Storage
Sorbent Pens containing extracted sample are placed in isolation sleeves, where they can be stored for more than 1 week prior to TD-GC-MS.



Step 5 Direct Thermal Desorption GC-MS
Sorbent Pen Thermal Desorption Unit (SPDU), Sorbent Pen Thermal Conditioner (SPTC) and Sample Preparation Rail (SPR) positioned on top of a GC-MS. The SPR transfers Sorbent Pens between the SPDU, the SPTC, and air-tight isolation sleeves. The SPDU is connected directly to a capillary column held within the GC-oven, which helps to minimize the flow path and mitigate analyte loss and carryover. Important optimization parameters for desorption include the temperature and duration of the preheat, desorption, and bakeout.

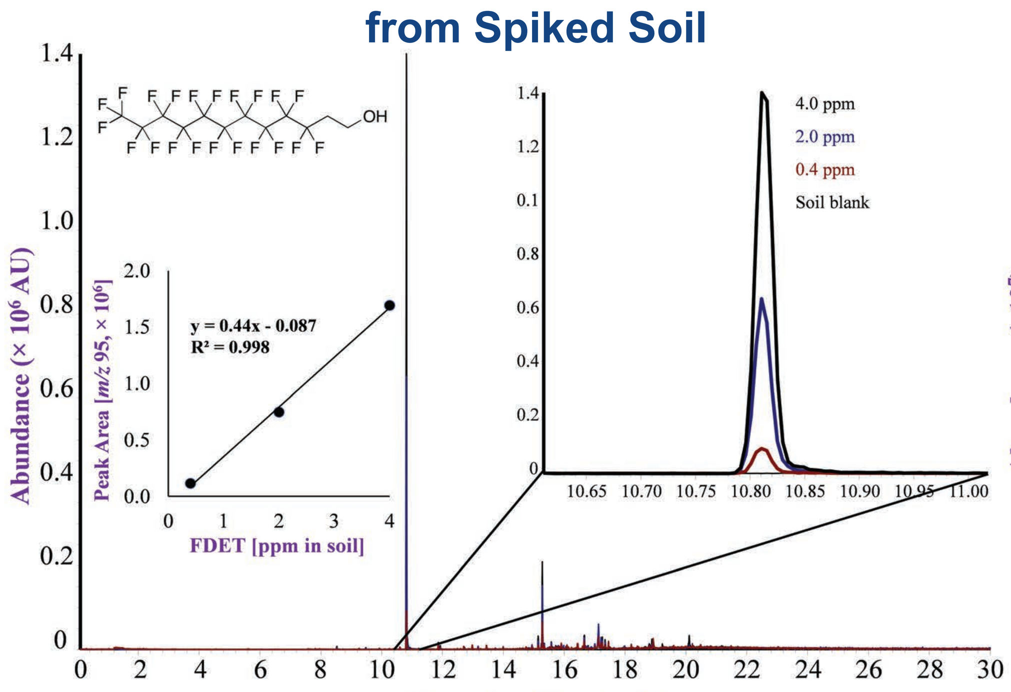


Step 6 Clean Pen Storage
Desorbed Sorbent Pens are placed in isolation sleeves for storage. During a typical workflow, the Sorbent Pens can be immediately re-used.

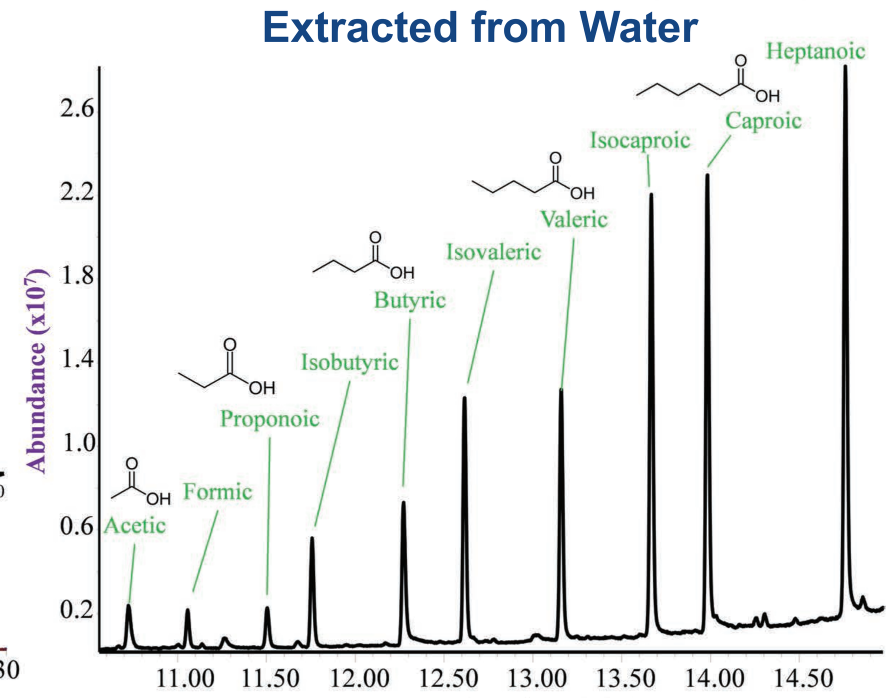


Environmental Contamination: PFAS & Fatty Acids

2-(Perfluorodecyl)ethanol (FDET) Extracted from Spiked Soil

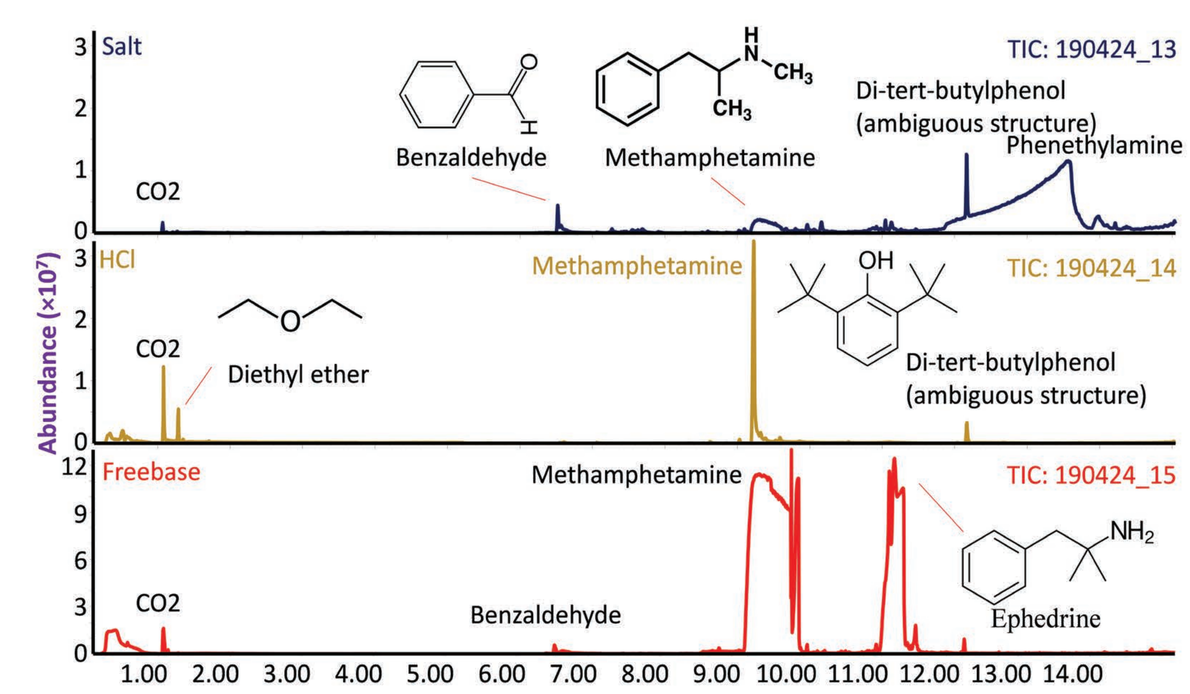


Short Chain Fatty Acids (SCFAs) Extracted from Water

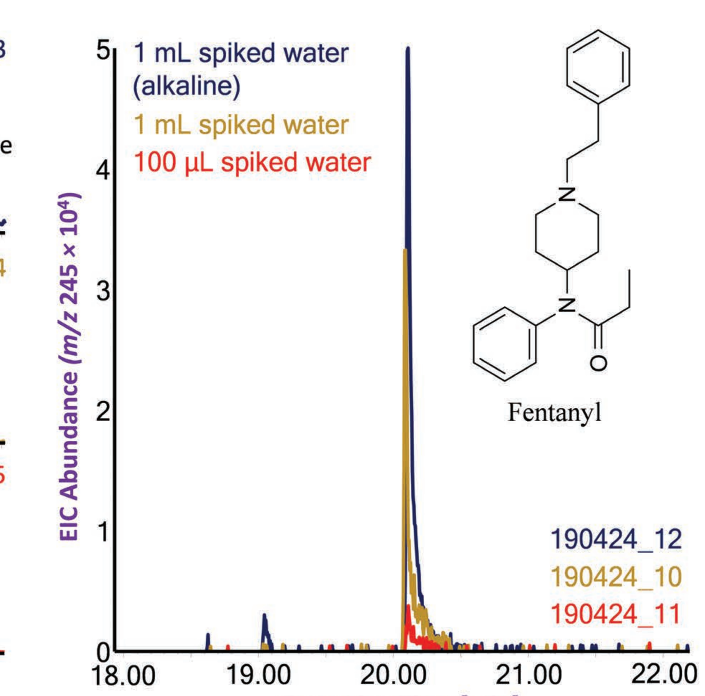


Methamphetamine & Fentanyl

Methamphetamine Extracted from Water

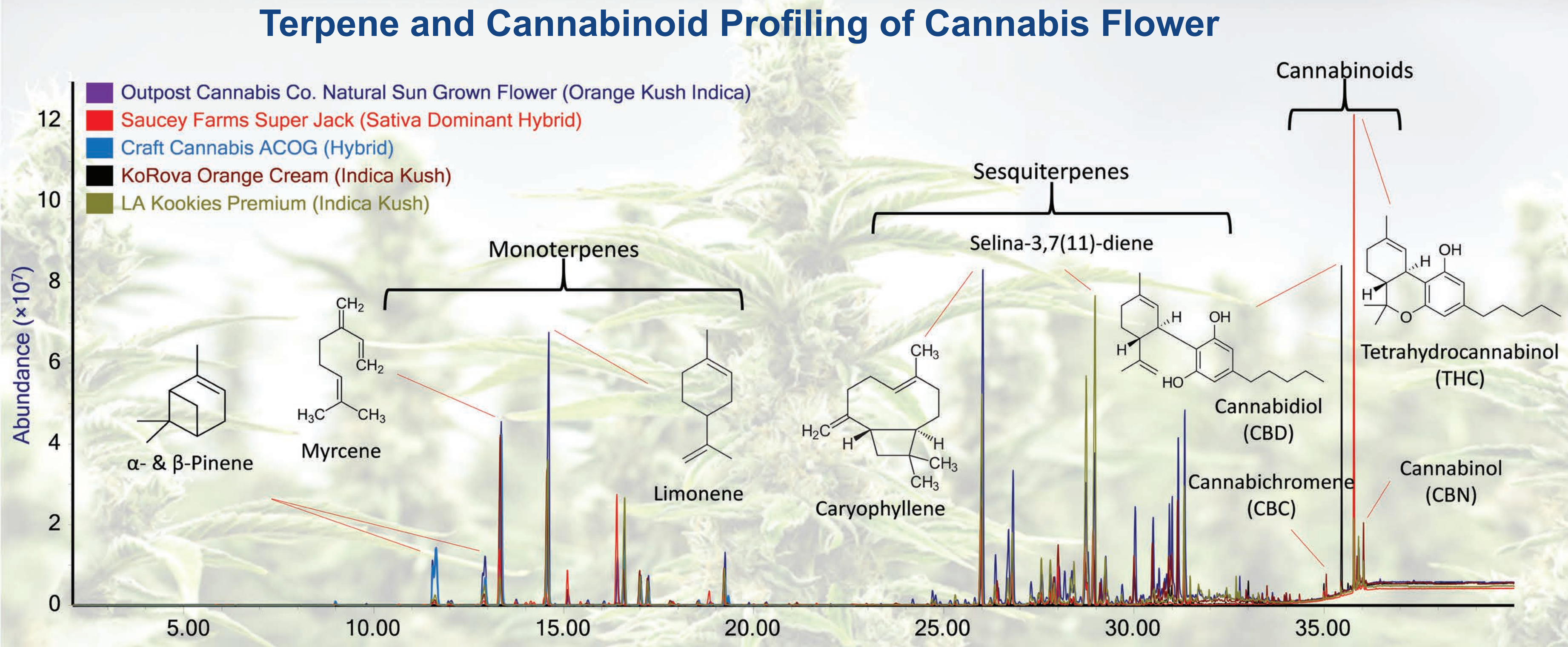


Fentanyl from Water

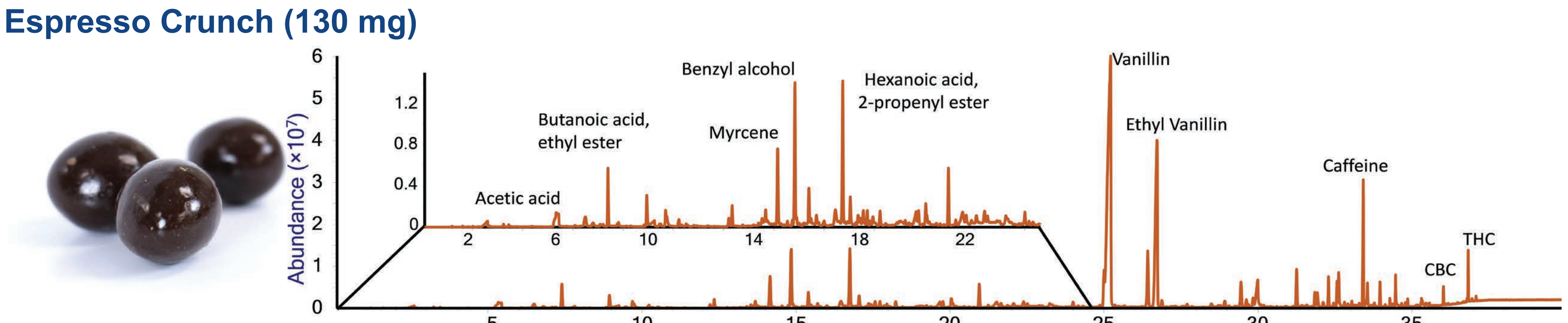


Cannabis & Cannabis Infused Consumer Products

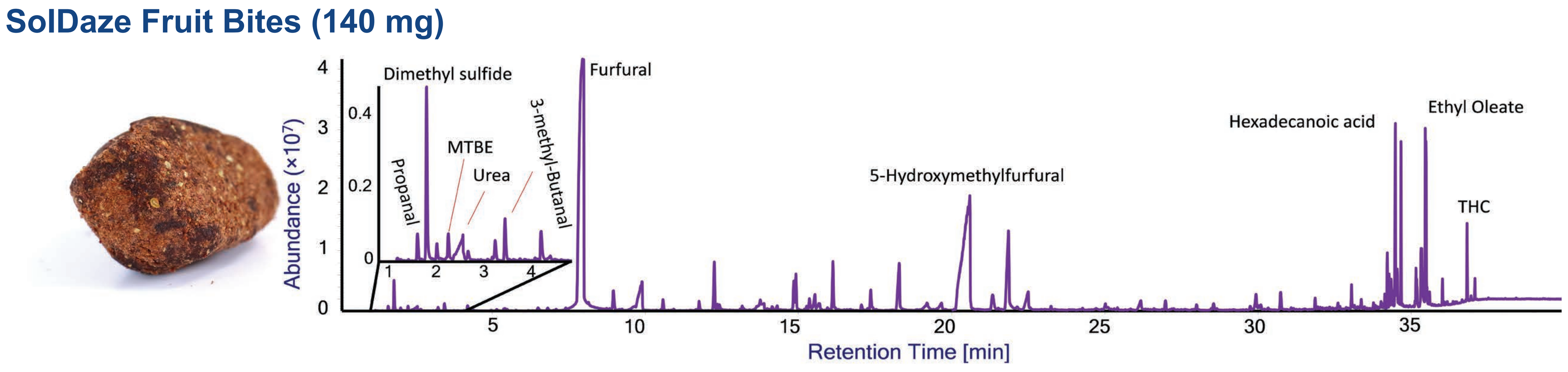
Terpene and Cannabinoid Profiling of Cannabis Flower



Espresso Crunch (130 mg)

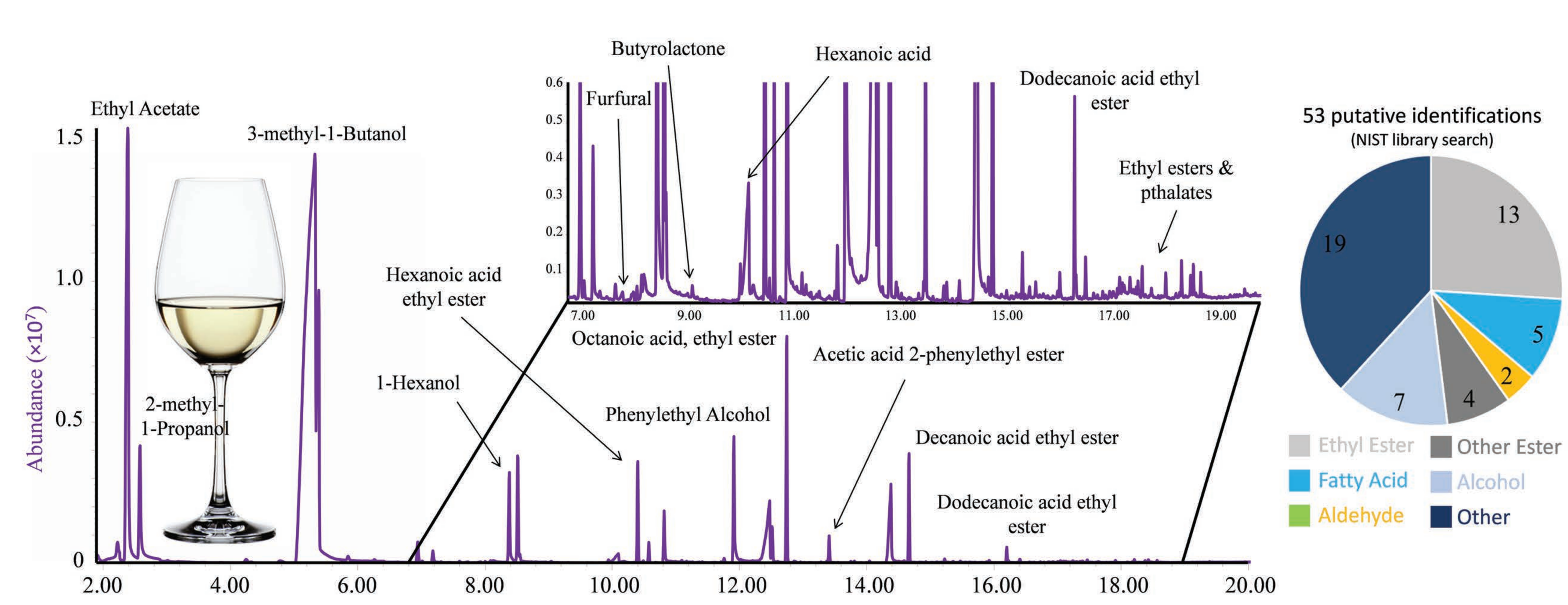


SolDaze Fruit Bites (140 mg)



Wine

500 µL Sauvignon Blanc Wine (diluted to 5 mL with 300 mg/mL NaCl)



Conclusions

- The experimental approach for a vacuum assisted headspace extraction technique, VASE, is described.
- Example applications are shown in the areas of environmental contamination, drugs of abuse, alcoholic beverages, cannabis, and cannabis infused consumer products.
- Our results show that – when combined with thermal desorption GC-MS – VASE is an exceptional tool for quantitative and qualitative analysis of both endogenous compounds and residual adulterants.

References

R.G. Belardi & J. Pawliszyn, "The application of chemically modified fused silica fibers in the extraction of organics from water matrix samples and their rapid transfer to capillary columns", Water Pollut. Res. J. Can. 24 (1989) 179-191

Acknowledgments

The authors express their gratitude to the entire Entech staff, including the excellent engineers, machinists, software developers, assemblers, and graphic designers. We are also grateful for the contributions of Prof. Jarrad Wagner and Austin Ciesielski from Oklahoma State University.