ENTECH INSTRUMENTS

See What's Really There™

VASE

VACUUM ASSISTED SORBENT EXTRACTION

Recover compounds over a wider volatility range than either SPME or SBSE

Improved Headspace Analysis using Vacuum and high Phase Ratios to maximize recoveries while minimizing matrix effects

VASE
VACUUM ASSISTED SORBENT EXTRACTION

Summary of Emerging Sorbent Pen Extraction Techniques

Entech is proud to present the next generation in headspace extraction solutions that substantially extend the range of recoverable compounds in an increasing number of sample types and matrices while improving sensitivity and reproducibility. These new techniques include VASE, Flash-VASE, MA-VASE, FEVE, and LVSH, which each optimize GC compatible compound recovery based on the matrix being investigated, the range of compounds of interest, and the desire to measure the composition of the liquid/solid matrix vs the equilibrated headspace for accurate aroma analysis. Sorbent Pen extracted samples are then thermally desorbed into a GCMS using the Entech 5800 Sorbent Pen Desorption Unit (5800 SPDU) that "gently yet completely" thermally transfers the sample to a GCMS, using SPLIT or SPLIT-SPLITLESS desorption modes to optimize the delivery of the extract to the GC column.

Introducing VASE - Vacuum Assisted Sorbent Extraction - What is it?

Vacuum Assisted Sorbent Extraction (VASE) is a next generation extraction technique capable of greatly extending the range of extractable compounds over other headspace or even full immersion techniques. Virtually all liquid and solid samples to be analyzed by GC or GCMS are compatible with this exciting technique that was recently made even better by modifying the presentation of the Sorbent Pen to the vacuum headspace that completely prevents the deposition of aerosols on the outside of the Sorbent Pen during rapid agitation. Aerosol contamination is a known issue with HS-SPME and other headspace techniques where splashing of the sample can lead to the deposition of non-volatile compounds on the extraction device, shorting the lifetime of these devices and increasing the production of thermal breakdown products that can be hard to distinguish from compounds that were actually in the original sample.

VASE – Technique Selection Guide

Sample Type: Liquid or Solid

Extraction Temperatures: Ambient to 70° C

Extraction Times: 5 min to 16 hours

Operational Mode: Static Vacuum Extraction in a Closed System, often to full equilibrium

BP Range: -50° C to +500° C

Vial Sizes: 20/40/125mL

Typical 5800 Mode: SPLIT or SPLITLESS

Water Management: Cold Tray Dehydration, and elimination during 5800 desorption both in SPLIT and SPLITLESS Modes

VASE is performed under vacuum which allows compounds to migrate much faster to the extraction device positioned only a few centimeters away, and the tremendous increase in phase over HS-SPME (>100,000x increase in surface area over a PDMS fiber) can virtually eliminate matrix affects thereby dramatically improving sensitivity and extraction reproducibility. Due to the consistency of the VASE technique, multiple Sorbent Pens can be used to extract several samples in parallel to increase the sample throughput with very little "Pen to Pen" variability in the results. Sorbent Pens with 1, 2, or 3 sorbent beds are available based on the desired boiling point range to be recovered, and custom sorbent packings are also available upon request. Analysis of the Sorbent Pen extracts are performed using the 5800 Sorbent Pen Desorption Unit (5800 SPDU), which provides multiple desorption options including SPLIT, SPLITLESS, and SPLITLESS VOC modes to optimize the recovery of compounds of interest.

Applications include:

Water Analysis
- VOCs & SVOCs
- Emerging Contaminants
- Odor Agents

Food Safety
- Nitrosamines
- Acrylamide
- Pesticides/Herbicides
- Carcinogens
- Preservatives

Flavors/Aromas
- Foods
- Beverages
- Alcoholic Beverages
- Taints/Off-Flavors

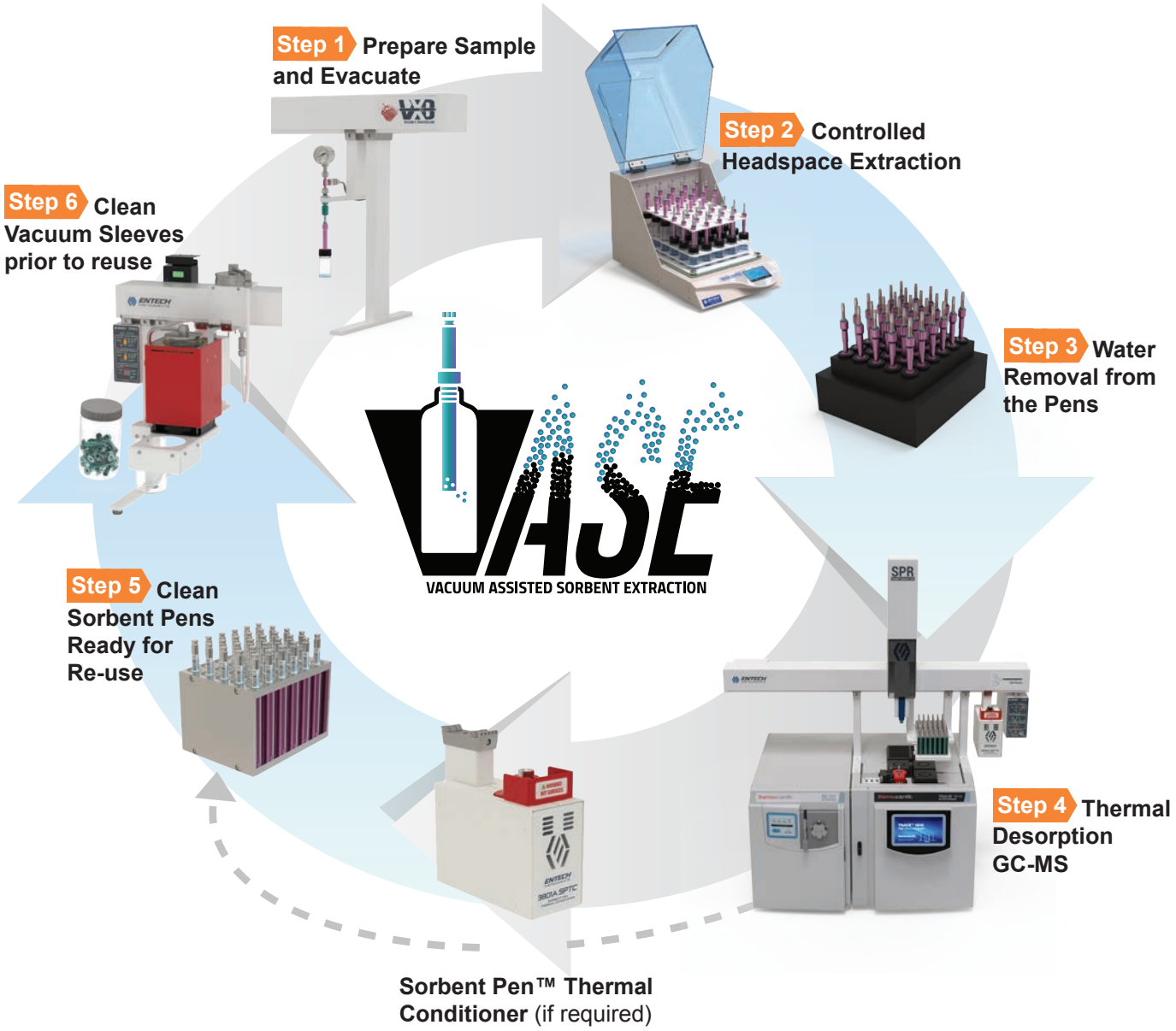
Cannabis
- Pesticide Screening
- Terpene Profiling
- Residual Solvents
- Cannabinoid Potency

Clinical Markers/Drugs In
- Blood
- Urine
- Breath

Misc.
- Odors in Consumer Products
- Residue Drugs/Pharma
- PCBs, PBDEs

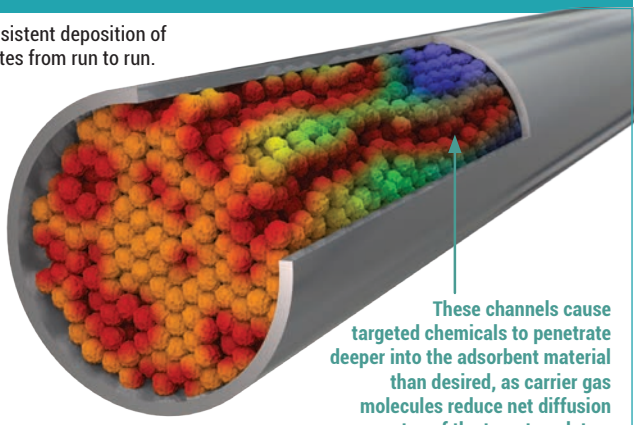
How It Works:

Sorbent Pens can be used literally hundreds of times to perform VASE extractions. To perform VASE, a sample is weighed or measured into a 20, 40, or 125mL vial, a vacuum sleeve is secured to the vial using a high temperature plastic cap, and a Sorbent Pen is inserted with 1 or more sorbents depending on compounds of interest. However, since compounds cannot "break through"" the sorbent during extraction in the closed system created by the VASE process, even a moderate strength sorbent such as Tenax TA can recover much lighter compounds than is possible with dynamic headspace techniques. The Pen/Vial combination is then evacuated for 15-20 seconds to the boiling point of water at 25° C (0.3 psia, or 2KPa). The evacuated assemblies are placed into trays where they are heated and agitated using the 5600 Sorbent Pen Extraction System. After an extraction period of 0.1-16 hours, the Pen/Vial assemblies are removed and placed into a cold tray to quickly withdraw any moisture that partitioned into the Sorbent Pens during extraction. As the assemblies are still under vacuum, the transfer of moisture to the chilled vials occurs in just 10-20 minutes. After isolation into the Sorbent Pen sleeves, the Pens are thermally desorbed into a GCMS using the 5800 Sorbent Pen Desorption Unit. The deposition of compounds near the entrance of the Pens not only leads to a fast delivery to the GC column(s) with less thermal decomposition, but also typically eliminates the need for additional cleaning of the Pens prior to reuse. A 3801A Thermal Conditioner can clean Pens when they are severely overloaded, or before initial use.



Channeling: Limitations of Technologies using Flow-Through Adsorbent Beds

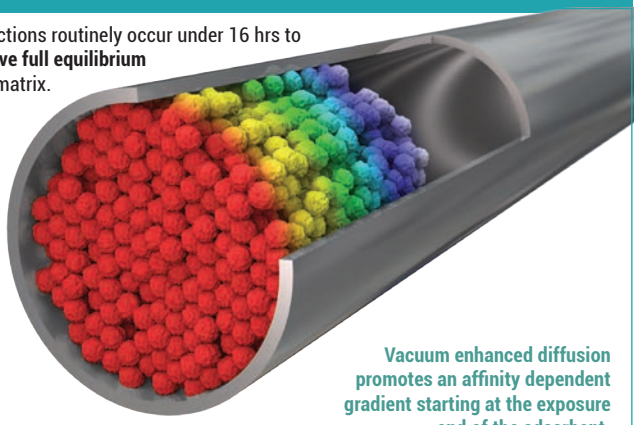
Inconsistent deposition of analytes from run to run.



These channels cause targeted chemicals to penetrate deeper into the adsorbent material than desired, as carrier gas molecules reduce net diffusion rates of the target analytes.

VASE Improves Recovery/Blank Level Performance by Creating a True Affinity Deposition Profile on the Pens

Extractions routinely occur under 16 hrs to **achieve full equilibrium** with matrix.



Vacuum enhanced diffusion promotes an affinity dependent gradient starting at the exposure end of the adsorbent.

Channeling Effect

All materials have a coefficient of thermal expansion that causes them to expand when heated and shrink when cooled. Sorbents used in packed traps are no exception. During dynamic headspace trapping, the channels that form in the cooled adsorbent cause compounds to travel further into the sorbent than would be expected by the affinity of the analyte to the sorbent, creating both recovery and carryover issues with this approach.

The VASE Solution

VASE works without the use of a carrier gas so there is no directional force pushing analytes deep into the sorbent material. The presence or absence of channels within the sorbent during VASE sampling has little to no effect on transmission of compounds further into the sorbent, essentially allowing all components of interest to collect as close to the entrance to the trap as their affinity to the sorbent allows at the trapping temperature. This translates into a faster delivery into a GC during direct thermal desorption for improved chromatography, while reducing the time and temperatures needed to bake out the Pens prior to reuse.

GCMS Analysis of VASE Strawberry Extract

(Over-loaded Pen Still Results in Clean Blanks)

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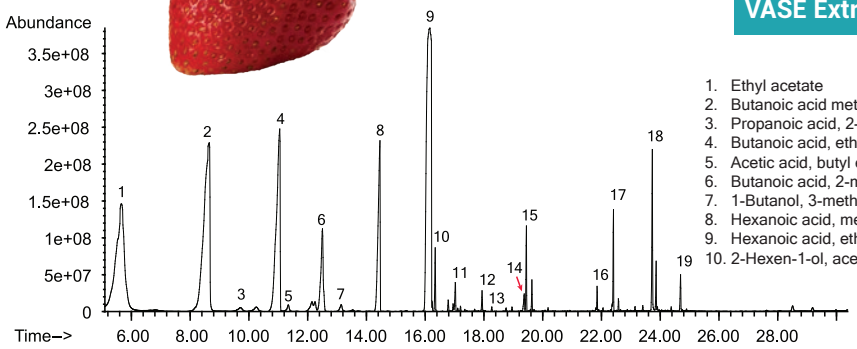
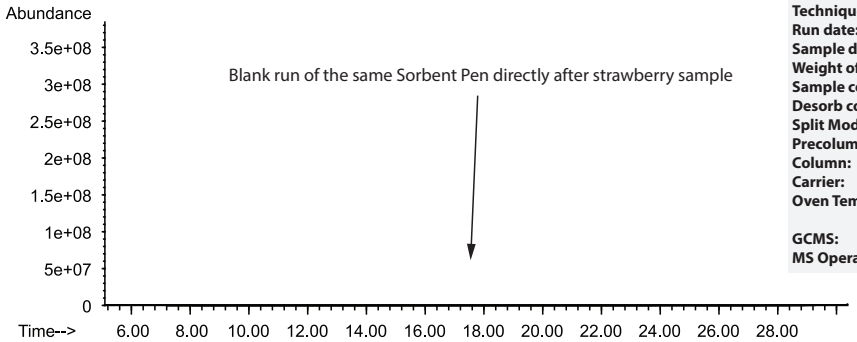
VASE Extraction = Zero Channeling into Sorbent



1. Ethyl acetate
2. Butanoic acid methyl ester
3. Propanoic acid, 2-methyl-, ethyl ester
4. Butanoic acid, ethyl ester
5. Acetic acid, butyl ester
6. Butanoic acid, 2-methyl-, ethyl ester
7. 1-Butanol, 3-methyl-, acetate
8. Hexanoic acid, methyl ester
9. Hexanoic acid, ethyl ester
10. 2-Hexen-1-ol, acetate, (Z)-
11. 3(2H)-Furanone, 4-methoxy-2,5-dimethyl-
12. 1,6-Octadien-3-ol, 3,7-dimethyl-
13. Octanoic acid, methyl ester
14. Methyl salicylate
15. Octanoic acid, ethyl ester
16. Butanoic acid, octyl ester
17. Pentanoic acid, octyl ester
18. 1,6,10-Dodecatrien-3-ol, 3,7,11-trimethyl-
19. .gamma.-Dodecalactone



Blank run of the same Sorbent Pen directly after strawberry sample

Instrument:	5800-SPDU (Sorbent Pen Desorption Unit)
Technique:	VASE (Vacuum Assisted Sorbent Extraction)
Run date:	August 3, 2016
Sample description:	Fresh strawberries
Weight of sample (g):	7.7425
Sample conditions:	vac(30sec) + 15hr equilibration
Desorb conditions:	260°C for 5 min.
Split Mode:	Splitless
Precolumn:	DB1 5m length x 0.25mm ID, 0.25µm film
Column:	DB1 30m length x 0.25mm ID, 0.5µm film
Carrier:	He, 1.5cc/min. constant flow
Oven Temp:	40°C hold 5min., 6°C/min. to 95°C, 10°C/min. to 140°C, 15°C/min. to 325 hold 5min.
GCMS:	Agilent 7890B/5977A
MS Operation:	34-450 amu, 1.8 scans/sec

Newly Updated Sorbent Pen Technology for 2021 and Beyond

VASE was first introduced in 2016, and it has been used by many research groups around the world to improve headspace extractions relative to other sample preparation techniques. However, as with most technologies, feedback from research has led to improvements in the way VASE is performed.

Rather than placing the Sorbent Pen primarily into the headspace of a vial, the recently updated approach only places the opening of the Pen into the vial. This turns out to have a lot of advantages:

1. During agitation, aerosols will undoubtedly form which can cause non-volatiles to be delivered up into the headspace. With the original VASE (and with current SPME applications), aerosols can deposit on the outsides of the Pen (or on the SPME fiber), causing a buildup to form, resulting in both additional carryover potential and artifact formation due to heating of non-volatile and often labile compounds during desorption (Carbohydrates, proteins, etc). The new VASE technique hides all but the entrance of the Pen to the headspace, substantially eliminating the exposure to aerosols created by rapid sample agitation. Excess aerosol formation can lead to reduced SPME fiber lifetimes as well, and with SPME that must perform radial adsorption, there is no way to hide the collection surface from aerosols.

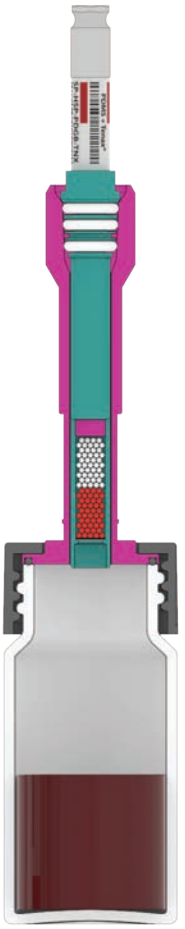
2. **Less carryover of Heavy Compounds**
   Although a repeat thermal desorption from the original HSP Pens after VASE extraction showed no carryover, a VASE extraction of a blank afterwards would show anywhere from 0.1-3% carryover of the heaviest compounds. This was due to deposition of the heavy analytes near the top, outside of the Pen, which does not get as hot during thermal desorption/bakeout. When placed back into a VASE vial at elevated temperatures, low level steam could release these heavy compounds so they could find their way into the sorbent to appear as carryover in the next analysis. The new VASE strategy substantially prevents the heavy compounds from reaching the top, outside of the Pens, virtually eliminating the carryover issue with the original VASE technique.

3. **Less Glassware Exposure** - By placing the Pen at the top of the vial, a 20mL vial allows as much sample to be analyzed as a 40mL vial using the previous approach. However, as can be seen by the included images of the old and new approach, the 20mL vials are 2 times shorter, and when holding the same amount of sample as the 40mL vial when using the original VASE, the exposure to glass in the headspace is 3x less using the new VASE approach. Surface effects caused by the glass are thereby reduced by 3x.

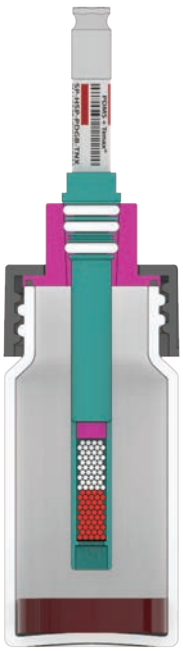
4. **Less Water Collection**
   The new VASE places the sorbent "above" the vial, allowing its temperature to be regulated differently, and sometimes slightly higher than the temperature of the vial in order to reduce or nearly eliminate water collection in the Sorbent Pen. The 5600 Sorbent Pen Extraction Unit has a slightly higher temperature at the top than at the vial platform, resulting in the overall reduction of water collecting on the Pen, and therefore a reduced time needed to dehydrate the Pen/Vial assemblies on a cold plate after the extraction.

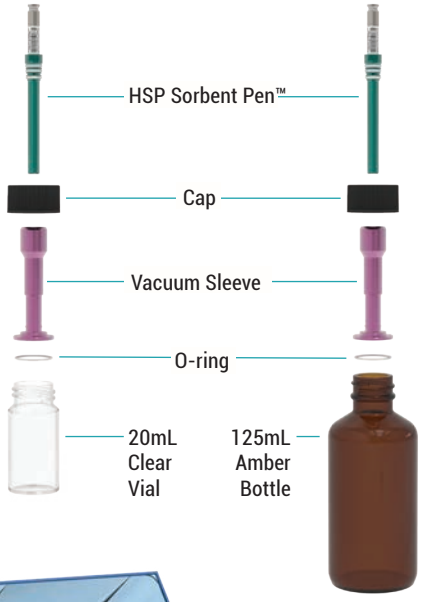
5. **No More Methanol Cleanup**
   The new VASE procedure now allows use of the 3700 Thermal Vacuum Cleaning System to perform vacuum sleeve and even O-ring cleanup fully automated without the use of solvents. See the 3700 brochure for more information on this "green " addition to Entech's vacuum extraction produce line.

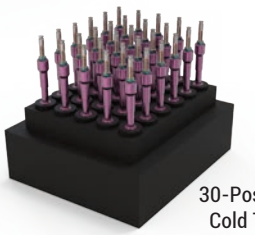
New VASE



Old VASE







HSP Sorbent Pens, Vials, and Vacuum Sleeves

Description	Qty	Unit	Part #
Sorbent Pens™			
HS Sorbent Pen - Tenax TA	1	EA	SP-HSP-TNX
HS Sorbent Pen Tenax® + Carboxen® 1000	1	EA	SP-HSP-TNX-CXN10
HS Sorbent Pen Tenax® + Carbopack™ X	1	EA	SP-HSP-TNX-CPX
HS Sorbent Pen - PDMS Coated Glass Beads + Tenax TA + Carbopack™ X	1	EA	SP-HSP-PDGB-TNX-CPX
HS Sorbent Pen - Blank/Empty	1	EA	SP-HSP-0
HS Sorbent Pen - PDMS Coated Glass Beads + Tenax TA	1	EA	SP-HSP-PDGB-TNX
HS Sorbent Pen O-Rings (upper) 10 pack	4	Pack	SP-OR-SP1-2
HS Sorbent Pen O-Rings (lower) 10 pack	2	Pack	SP-OR-SP3
Glassware, Caps, & Vacuum Sleeves			
20mL Clear Vials (72 ct.)	1	Box	39-75020
40mL Clear Vials (72 ct.)	1	Box	39-75040
125mL Amber Bottles (24 ct.)	1	Box	39-75125AD
Plastic Vial Caps for 20/40/125mL (144 ct)	1	Pack	39-76044B
Vacuum Sleeve Lid Liner for 20/40/125mL, 24mm Screw Top Vials	1	EA	SP-VSLL024S
White Viton O-Rings for 20/40/125mL Vials (10 ct) Low Bleed	1	Pack	SP-OR-L024
Sorbent Pen Ejection Tool	1	EA	SP-PEN-EJECT-TOOL

5600 SPES and Cold Tray Dehydration

The 5600 Sorbent Pen™ Extraction System agitates the samples at 30-300 RPM to speed up transfer of volatiles to the headspace, while optionally heating the sample from ambient +4˚C to 70˚C. Extractions are generally complete in 1–48 hours depending on the application. A 30-Position Cold Tray that is pre-cooled in a lab freezer at about -18˚ C can be used to draw any moisture back out of the Pens prior to Pen removal, isolation, and then GCMS analysis.

Description	Qty	Unit	Part #
Agitation, Extraction, Water Management			
5600 Sorbent Pen Extraction System, 120VAC/60Hz	1	EA	5600-SPES
5600 Sorbent Pen Extraction System, 230VAC/50Hz	1	EA	5600-SPES-HV
30-Position Tray for 20/40mL Vials	1	EA	5600-040TRAY30-2
15-Position Tray for 125mL Amber Bottles	1	EA	5600-125TRAY15-2
30-Position Cold Tray to Dehydrate Pens after 20/40mL Vial Extractions	1	EA	SP-HSCOLDTRAY30-2
15-Position Cold Tray to Dehydrate Pens after 125mL Bottle Extractions	1	EA	SP-HSCOLDTRAY15-2

Sample Preparation & Extraction

Description	Qty	Unit	Part #
Essential Preparation			
30-Position Sorbent Pen Isolation Tray	1	EA	SP-HSP-TRAY30
Vial Evacuation Unit	1	EA	SP-VIAL-EVAC
VXB Vial Evacuation Unit (VXB - Vacuum X-traction Bar)	1	EA	SP-VXB-PV-EVAC
30-0"Hg Vacuum Test Gauge (w/ Female Micro-QT)	1	EA	29-70010QT
Double-Ended Micro-QT Valve	1	EA	MQT-2S
2-Stage Oilless Diaphragm Pump, 120/240VAC, 60/50Hz	1	EA	10-20036

3801A Sorbent Pen Thermal Conditioner

Description	Qty	Unit	Part #
Sorbent Pen Conditioning			
3801A Sorbent Pen Thermal Conditioner, 120VAC/60Hz	1	EA	3801A-SPTC
3801A Sorbent Pen Thermal Conditioner, 230VAC/50Hz	1	EA	3801A-SPTC-HV

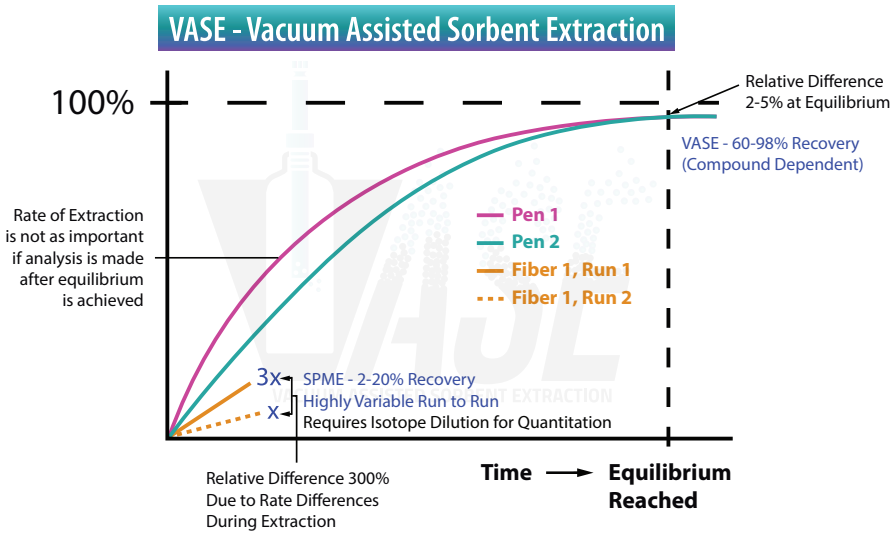
ADVANTAGES OF VASE

Improvements over SPME & Dynamic Headspace Trapping

- Highly reproducible.
- Minimal carryover without the need for a secondary bakeout/cleanup step.
- Durable - hundreds of injections.
- Thousands of time more phase than SPME to eliminate matrix effects on sorbent affinities.
- Operates at or near equilibrium to improve sensitivity and quantitative accuracy.
- Performs exhaustive vacuum extraction of VOCs through SVOCs.
- Unlike SPME, Pens are shielded from exposure to aerosols formed during agitation.
- See taints, odors, additives, flavors & fragrances at levels below previously possible.
- Faster injection rates produces better chromatography and less thermal degradation.
- Rapid injections without cryogen or electronic cooling.
- Higher throughput via parallel off-line extractions.
- Sample at elevated or sub-ambient temperatures as needed.

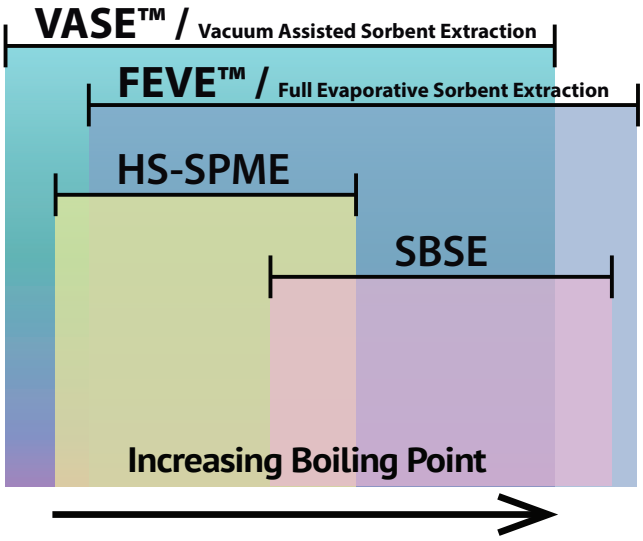
VASE, utilizing Sorbent Pens™, Operates at or Near Equilibrium to Improve Sensitivity and Reproducibility.

- **Operating at or near equilibrium increases sensitivity and reduces run to run variability.**
- **Small changes in VASE extraction conditions result in inconsequential differences in the ultimate recovery at equilibrium, resulting in excellent reproducibility.**
- **Typically no need to use isotope dilution for quantitative measurements.**



VASE and FEVE (Sorbent Pens) vs HS-SPME and SBSE Recovery Relative to Analyte Volatility.

- **Using VASE or FEVE, Sorbent Pens can recover compounds over a wider volatility range than either SPME or SBSE.**
- **Most applications done by either SPME or SBSE can be done more easily and usually with higher sensitivity and accuracy utilizing VASE or FEVE.**



ENTECH INSTRUMENTS

LVSH
LARGE VOLUME STATIC HEADSPACE

VASE
VACUUM ASSISTED SORBENT EXTRACTION

DVS
DIFFUSIVE VIAL SAMPLER

FLASH VASE
VACUUM ASSISTED SORBENT EXTRACTION

FEVE
FULL EVAPORATIVE VACUUM EXTRACTION

Sorbent Pens™ - Achieving the Full Potential of Clean Headspace Extractions in Virtually All Sample Matrices

For additional chromatography data,
see the VASE Featured Chromatograms Booklet.

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