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See What's Really There[™]



Flash-VASE

A Superior Vacuum Replacement for Thermal Extraction Analysis

Cannabis, Synthetics, Powders, Packaging, Consumer Products, Extractables and More



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 Flash-VASE - What is it?

Flash-VASE is a next generation Thermal Extraction technique that places the sample in very close proximity to the collection sorbent, and uses static extraction under vacuum to more efficiently and completely recover volatile through semi-volatile compounds. Flash-VASE places the sample within 1-2 cm of the collection sorbent, with no flowing gases to heat up the sorbent during the extraction process like with dynamic headspace techniques, so the collected compounds stay optimally close to the front of the sorbent bed for very fast release to the GC. Extraction under a vacuum allows recovery of chemicals at a lower temperature, reducing or avoiding breakdown of the matrix itself. Flash-VASE has a tremendous number of advantages over other thermal extraction techniques, allowing Chemists to truly "See What's Really There[™]", in a way that keeps the analytical system clean and free from carryover.

Flash-VASE – Technique Selection Guide

Sample Type: Solids/Liquids w/ low volatility matrix Applications: Volatiles/Odors in Packaging, Polymers, Powders, Foams, Synthetics, Natural Products, Heavy Oils Extraction Temperatures: 30° C to 280° C Extraction Times: 2 - 10 Min Typical Operational Mode: Static Vacuum Thermal Extraction BP Range: -50° C to >500° C (depending on extraction temperatures and sorbents used) Vial Sizes: Flash-VASE: 2, 6, 20mL Typical 5800 Mode: SPLIT or SPLIT/SPLITLESS Water Management: SPLIT injection and/or post extraction vial cooling prior to Pen removal

Flash-VASE is intended for samples containing relatively low moisture levels, as the sample is heated anywhere from 30° C to 280° C while in a closed system, so the potential for generating a substantial amount of vapor during Thermal Extractions should be considered. However, some excess water can be temporarily delivered and condensed on the Sorbent Pen, as post extraction cold tray cooling of the vials can be performed to transfer the moisture back into the bottom of the vial while still under vacuum, and prior to removal of the Pens from the vacuum extraction sleeves.

The Flash-VASE module can be altered to perform MA-VASE (Matrix Accelerated VASE) which performs a "reflux in a vial" under vacuum to further accelerate the transfer of organics into the gas phase. Just a few additional components allows both techniques to be performed using a single module, supporting a very wide range of sample types. **See the MA-VASE TID for more information on this exciting and complementary technique**.

Flash-VASE Process



1 Weigh/measure sample into vial, attach vacuum sleeve and Sorbent Pen, and evacuate the Pen/Vial Assembly



2 Place Vial/Pen assemblies in Flash-VASE module and perform extractions while under vacuum by heating sample vial to any temperature between 30-280° C



3 Transfer excess water back into vial using cold tray if needed



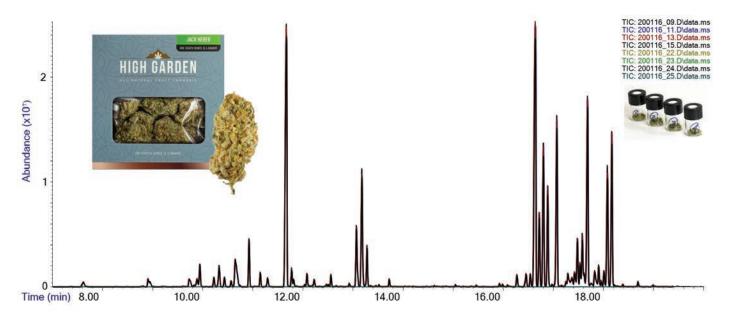
Isolate Sorbent Pens into 30 position tray and desorb using 5800 SPDU either manually or using an SPR40 Rail Autosampler

- Cannabis for analysis of Terpenes and Cannabinoids
- Tobacco for analysis of aromas/flavors
- Plastics for contaminants and unreacted monomers
- Powders for residues and odors
- Dry food products for aromas and contaminants
- Synthetics for regulatory purposes (Automobile Cabin, Foam Outgassing, IAQ Concerns, Micro/Mini Chamber Testing)
- Volatile fractions in oils
- Determination of extractable compounds in "Leachables/ Extractables" analysis

The list of applications is quite extensive, and Flash-VASE can be scaled up to look at outgassing from larger samples as needed, such as in forensic investigations of accelerants in fire debris. Just as in VASE, the presence of a vacuum increases the rate of evolution of volatiles out of materials at lower temperatures, and depending on the matrix or sample surface area, the extraction times can be as short as 2-5 minutes (Cannabis) by heating the sample to 100°- 200° C under vacuum. This provides a whole new opportunity for high speed sample throughput for Flash-VASE compatible applications.

Extreme Precision - By Design

Flash-VASE provides incredibly reproducible results, by eliminating the inconsistencies inherent in dynamically purged Thermal Extractions. Below is an example of 8 replicate Cannabis analyses for Monoterpene and Sesquiterpene profiling, and these 8 runs almost perfectly overlap. The Flash-VASE extraction times in these examples was just 5 minutes, at 100° C. Higher temperatures will yield reproducible recovery of the Cannabinoids present in Cannabis.



The Flash-VASE Workflow

Step 1

Step 2

Step 3

Step 4

Step 6

Step 7

Flash-VASE extractions are typically very fast, although the Entech SPRINT software allows longer extractions at lower or ramped temperatures as needed. The extraction process is simple:

Step 1 Transfer a volume or weight of sampling into a 2.6. or 20mL vial based on the size, concentration of volatiles, and homogeneity of the sample. Step 7 Assemble the vacuum sleeve and retaining nut or cap onto the vial, and insert a clean Sorbent Pen. Perform a guick 10-20 second evacuation of the assembly through the top of the Sorbent Pen. The Vacuum will be retained by the vial/Pen assembly during the extraction. Place the vial into the Flash-VASE Step 6 extraction module, with the extraction module either preheated or heated after vial introduction. Up to 15 Pen/Vial assemblies can be processed in a single batch process. Step 5 After the extraction (2-10 min typical), remove the vials and place in a cold tray to drop the temperature of the vial below that of the Pen to draw any excess water back into the vial. This step is optional if water content in the sample is low. Step 5 (OPTIONAL) Remove and place the Pens in an Craft Cannabis ACOG Hybrid 5 minute VASE at 100°C isolation tray, and analyze using a 15 5800 Sorbent Pen Desorption Unit (x10⁷ 4 10 (5800 SPDU), either one at a time or inalool 3 Abundance automated using the Entech SPR40 rail 5 2 autosampler. 1 33.00 0 5.00 10.00 15.00 20.00 Vacuum sleeves can be cleaned without KoRova Orange Cream Indica Kush solvents using a 3700 Thermal Vacuum 5 minute VASE at 100°C Linalool 40 Cleaning System (3700 TVCS) to remove 5 e (x10° Abundance (x107) 30 any residue from the previous analysis 4 20 3 Cannabidiol 2 Abu 10

10.00

15.00

20.00

Retention Time (min)

25.00

Flash-VASE utilizing the Vacuum X-traction Bar (VXB)

The Vacuum X-traction Bar (VXB) supports sample extraction onto Sorbent Pens and the cleanup of Sorbent Pens and associated extraction hardware (sleeves, O-rings, etc). The VXB allows extractions to be performed in the sample preparation area, with only the extracts on Sorbent Pens brought into the GCMS laboratory. This is similar to how sample preparation techniques are currently performed, except the Entech vacuum extraction methods are easier, cleaner, and more sensitive, all without the use of hazardous solvents. The VXB comes in 30" and 50" sizes, allowing for convenient above the bench management of the Flash-VASE and Pen Evacuation Modules. Other modules may also be connected, such as the 3700 Thermal Vacuum Cleaning System, which is utilized to restore background-free extraction hardware before performing the next set of extractions.



33.00

Step 2

....

Step

Cannabidio

34.00

34.00 Retention Time (mi

Step 3

Flash-VASE shown with Pen/Vial Evacuation Module, Cold Tray, and 3700 Thermal Vacuum Conditioning System to clean up vacuum sleeves and O-rings prior to reuse.

Flash-VASE A Safer and Better Way to do Thermal Extraction

Many systems attempt to perform thermal extraction by placing a sample into a tube and then directly thermally desorbing it into a GC. However, there are several draw-backs associated with this approach:

- 1. Samples may not transfer quickly from the sample to a GC column since chemicals of interest will not release very fast from many matrices, causing poor peak shape unless a secondary trapping or focusing system is used
 - a. Therefore, direct desorption methods often have to heat samples hotter to reduce band broadening, but higher temperatures will increase thermal degradation of the target compounds and the matrix
 - b. Liberated compounds must remain in contact with the matrix longer during a pre-heating step, rather than allowing their removal once they become mobile
 - c. Chemicals must diffuse out of the matrix at positive GC carrier gas pressures which slows down outgassing rates relative to a vacuum thermal extraction approach
- 2. Samples with a high volatiles content can backflash into the GC carrier gas delivery lines, permanently contaminating the lines until the entire injection system is removed and solvent rinsed.
- 3. Many matrices simply cannot be heated to GC injection temperatures, even momentarily, without changing them chemically, potentially creating artifact chemicals that were not in the original sample
- 4. There is no water/moisture management opportunities when directly desorbing a sample into a GCMS
- 5. Loading a sample into a 1/4" glass tube before direct desorption is more difficult than loading it into a vial, and some sample may drop into the desorber or injector, creating a background until removed

Flash-VASE solves all of these problems by performing an offline vacuum thermal extraction, followed by a moisture removal step if necessary while the sample/Pen assembly is still under vacuum in a closed system, simply by cooling the bottom of the sample vial to draw moisture back into the vial before removal and desorption of the Pen. This not only improves the performance of thermal extraction solutions, Flash-VASE may be the only way to perform thermal extraction on many sample matrices.

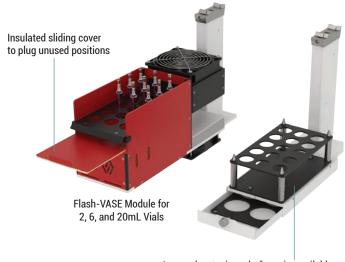
Flash-VASE Improves Performance over Off-Line Dynamic Thermal Extraction

Other thermal extraction systems use small chambers or micro chambers which attempt to thermally extract samples by heating them, flowing a gas over them, and delivering the desorb gas through an outlet port/fitting and into a classical sorbent tube. There are numerous problems associated with this approach as well:

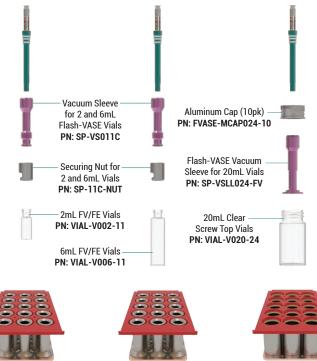
- 1. The cell or chamber can become contaminated when exposed to higher concentration samples
- 2. Contamination of outlet tubing
- 3. Loss of high volatility compounds that breakthrough the sorbent in this "open" system
- 4. Loss of low volatility compounds that adsorb to surfaces prior to reaching the TD tube
- 5. Heating of the front of the sorbent bed can occur when trying to maintain a hot transfer system all the way to the collection sorbent. Hot gas introduced onto a sorbent will allow compounds to penetrate further into the sorbent, resulting in lower thermal desorption recoveries and greater potential for carryover
- 6. Channeling Effects are exhibited while flowing through a sorbent trap, causing reduced recovery, increased carryover, and increased thermal degradation by requiring higher desorption temperatures during analysis

Flash-VASE eliminates these concerns as well. The entire "cell" is the sample vial, which is used once and discarded, so no chance of carryover. There are no transfer lines and connective fittings in the flow path, as the opening of the Sorbent Pen is right at the top of the vial. There is no hot carrier gas to heat up the sorbent, so the penetration of compounds into the sorbent is far less, making their recovery during thermal desorption more complete and at lower desorption temperatures. The Flash-VASE closed system means that even very light compounds will be recovered, as long as they have more affinity for the sorbent in the Pen than they do for the heated sample matrix. Finally, the extraction occurs diffusively, eliminating channeling and all of the negative effects this has on recovery, carryover, and sampler lifetimes.

Flash-VASE Vacuum Extraction Module **Components & Part Numbers**



A sample staging platform is available to support fast transfer in and out of the Flash-VASE module. Snap on tray tops allow changing between 2, 6, and 20mL vials for Flash-VASE, or up to 40/125mL vials when performing MA-VASE



6mL vial Flash-VASE Vial Module PN: VRES-FVVM-6ML



Flash-VASE Vial Module PN: VRES-FVVM-20ML

Ordering Information

(Note: Flash-VASE can be performed using 2, 6, and 20mL vials, typically using the smallest size that still allows required sensitivity and statistical accuracy to be achieved).

Flash-VASE Module Options and Pumps

Description	Qty	Unit	Part #		
Vacuum Thermal Extraction (VTE) of Materials					
Vial Heater Module, VASE Rail Extraction System, 120VAC/60Hz	1	EA	VRES		
Vial Heater Module, VASE Rail Extraction System, 240VAC/50Hz	1	EA	VRES-HV		
2mL vial Flash-VASE Vial Module	1	EA	VRES-FVVM-2ML		
6mL vial Flash-VASE Vial Module	1	EA	VRES-FVVM-6ML		
20mL vial Flash-VASE Vial Module	1	EA	VRES-FVVM-20ML		
2-Stage Oilless Diaphragm Pump, Dual Voltage 120/240VAC, 50-60Hz	1	EA	10-20100		

Vacuum Extraction Vials, Sleeves, Components, Accessories

Description	Qty	Unit	Part #
2mL Flash-VASE Components			
2mL Clear Vials, wide mouth 11mm Crimp Top	1	100pk	VIAL-V002-11
Vacuum Sleeve for 2 and 6mL Flash-VASE Vials	1	10pk	SP-VS011C-10
Securing Nut for 2 and 6mL Vials	1	10pk	SP-11C-NUT-10
2mL Vial Vacuum Sleeve Silicone O-rings	1	30pk	OR-L011S-30
6mL Flash-VASE Components			
6mL Clear Flash-VASE Vials	1	100pk	VIAL-V006-11
Vacuum Sleeve for 2 and 6mL Flash-VASE Vials	1	10pk	SP-VS011C-10
Securing Nut for 2 and 6mL Vials	1	10pk	SP-11C-NUT-10
6mL Vial Vacuum Sleeve Silicone O-rings	1	30pk	OR-L011S-30
20mL Flash-VASE Components			
20mL x 24-400 Clear Screw Top Vials	1	144pk	VIAL-V020-24
20mL x 24-400 Amber Screw Top Vials	1	144pk	VIAL-V020-24-A
Flash-VASE Vacuum Sleeve for 20mL Vials	1	10pk	SP-VSLL024-FV-10
Aluminum Caps for 20mL Vials	1	10pk	FVASE-MCAP024-10
Clean High Temp Silicone O-rings for 20mL vial Vac Sleeves	1	30pk	OR-L024S-30

Vial Platforms and Trays

2mL vial

Flash-VASE Vial Module

PN: VRES-FVVM-2ML

Description	Qty	Unit	Part #
10-Position Platform/Tray for VXB/SPR40, 20mL Vials	1	EA	SP-PF-TRAY10-20ML

Cold Dehydration Tray and Accessories

Description		Unit	Part #
Water Management			
Flash-VASE Cold Tray for Dehydration of 15 each of 2, 6, 20mL Vials	1	EA	SP-HSCOLDTRAY45-FV

VXB - Vacuum X-traction Bars

Description	Qty	Unit	Part #
Vacuum X-traction Bar (30" VXB), allows 1-2 modules to be attached simultaneously	1	EA	VXB-30
Vacuum X-traction Bar (50" VXB), allows 3-4 modules to be attached simultaneously	1	EA	VXB-50
Pen/Vial VXB Evacuation Module	1	EA	VXB-PV-EVAC

Flash-VASE Controllers

Description	Qty	Unit	Part #
Supports Flash-VASE, 3700, & 3830			
VXB Mounted Dual Controller for Flash-VASE 1/2 with SPRINT Interface, 120VAC/60Hz	1	EA	VXB-EMC-VASE-12
VXB Mounted Dual Controller for Flash-VASE 1/2 with SPRINT Interface, 240VAC/50Hz	1	EA	VXB-EMC-VASE-12-HV
VXB Mounted Dual Controller for Flash-VASE 3/4 with SPRINT Interface, 120VAC/60Hz	1	EA	VXB-EMC-VASE-34
VXB Mounted Dual Controller for Flash-VASE 3/4 with SPRINT Interface, 240VAC/50Hz	1	EA	VXB-EMC-VASE-34-HV

Flash-VASE 2/6/20mL Bundles

Part Numbers	F-VASE-2ML	F-VASE-2ML-HV	F-VASE-6ML	F-VASE-6ML-HV	F-VASE-20ML	F-VASE-20ML-HV
VRES	V		V		 ✓ 	
VRES-HV		 ✓ 		 ✓ 		 ✓
VRES-FVVM-2ML	V	 ✓ 				
VRES-FVVM-6ML			V	1		
VRES-FVVM-20ML					1	V
VIAL-V002-11	V	 ✓ 				
VIAL-V006-11			V	 ✓ 		
VIAL-V020-24					1	V
SP-VS011C-10	V	 ✓ 	V	V		
SP-VSLL024-FV-10					1	V
SP-11C-NUT-10	V	 ✓ 	V	 ✓ 		
FVASE-MCAP024-10					×	 ✓
OR-L011S-30	V	 ✓ 	V	 ✓ 		
OR-L024S-30					 ✓ 	V

Note: VXB Rail, Controller, Pump and Evac Tool available separately and required for complete system solution.

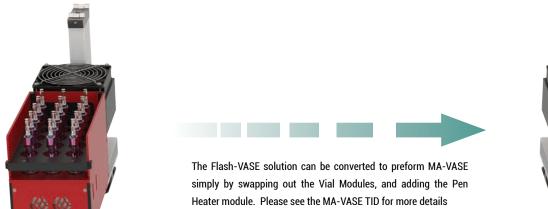
Sorbent Pen Selection Guide

FSP Sorbent Pens - For 2, 6 and 20mL Vial Flash-VASE

Part #	Adsorbent	Range BP	Label Color	Label	Packing Diagram	
SP-FSP-0	Blank / Empty	NA	Yellow	FSPErk FSPErk SPER		
SP-FSP-PDGB-TNX	PDMS Glass Beads + Tenax®	100°C to >500°C	Red / White	Frank Sciller Ver		6
SP-FSP-TNX	Tenax [®] TA	100°C to >450°C	White	Series Series in Prov Termina 193500 SPLSTNR		
SP-FSP-TNX-CPX	Tenax [®] TA + Carbopack™ X	80°C to >450°C	White / Blue	Balach Solary I Nef		2 and 6mL Vials
SP-FSP-TNX-CXN10	Tenax [®] TA + Carboxen [®] 1000	-60°C to >450°C	White / Green	Entransistence of the St Transf A Colonization Straff A Colonizati		
SP-FSP-CUSTOM	End User Defined	NA	Brown	fraida ofern frei Graidae Probet 9-554-005 Tott		
SP-FSP-525	Method 525 FSP	100°C to >500°C	Red / White	birddiarter Ywy Weed Stiff Priferigi		20mL Vials

VASE + Flash-VASE for Leachables/Extractables

The combination of Flash-VASE for measuring the content of extractables in a material, and VASE analysis on a liquid that was exposed to that material, is an ideal way to perform a Leachables/Extractables analysis. Flash-VASE will determine the total "Extractable" composition of volatile chemicals in a sample, often a plastic or plastic/wax lined paper container, and then VASE reveals to what extent these compounds have "leached" into a liquid that was exposed to that material. Examples include food packaging (Phthalates and other regulated contaminants) and pharmaceutical packaging (IV bags and delivery tubing), and other solid and liquid packaging applications.





MA-VASE

Flash-VASE





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

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