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# Cannabis Testing

Quality You Can Trust

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Cannabinoids

Terpenes

Pesticides

Mycotoxins

Residual Solvents

Trace Metal Analysis

Tools for Sample Preparation  
and Handling



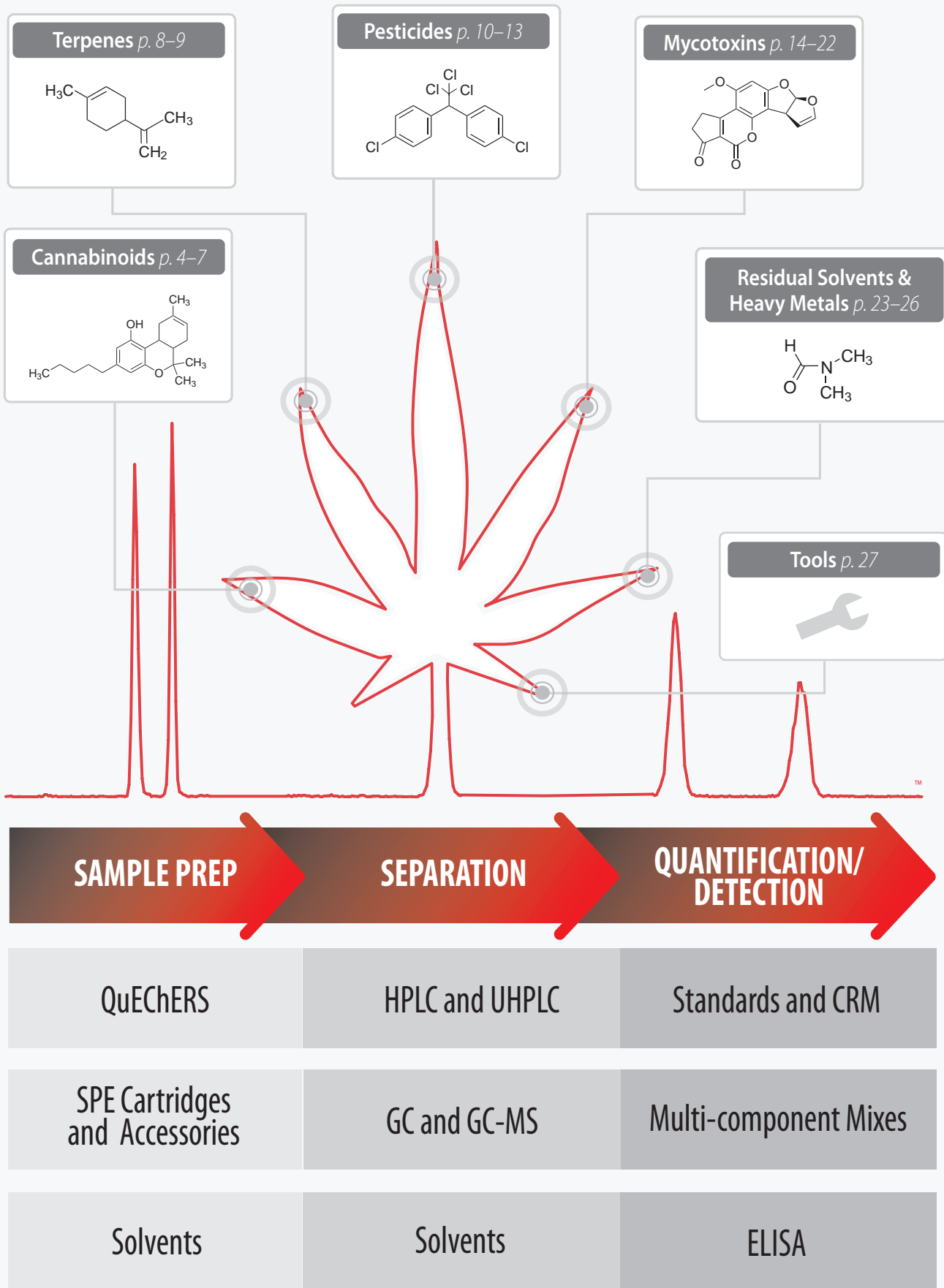
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# CANNABIS TESTING

As of January 2015, 23 states and the District of Columbia of the United States (U.S.) as well as Canada and various European countries, have passed laws allowing Cannabis to be used for medical applications.

Four U.S. states also allow for the recreational use of Cannabis. Currently, laws regulating manufacturing practices and quality standards of Cannabis source materials are few or nonexistent and vary widely among jurisdictions. In order to alleviate patient concerns regarding the efficacy and safety of Cannabis-derived therapeutics, Cannabis quality control testing is mandated in many jurisdictions.

Our Cerilliant® brand pioneered commercially available certified solution standards and U.S. DEA-exempt solution standards of controlled substances, including cannabinoids. We offer the broadest selection of Cannabis Certified Reference Materials (CRMs) and analytical standards for cannabinoids, terpenes, mycotoxins, heavy metals, pesticides, and residual solvents.

This brochure provides a selection of tools and consumables for the scientists performing analytical testing of Cannabis. Included are:

- QuEChERS reagents and SPE tubes for pesticide and other analytes.
- Ascentis® Express U/HPLC columns for cannabinoids, terpenes, pesticides, and other analytes.
- Supel Tox SPE cartridges to simplify mycotoxin analysis. More than 10× faster, simpler, and more reproducible than immunoaffinity columns.

For more information, visit  
[sigma-aldrich.com/cannabis](http://sigma-aldrich.com/cannabis)





# CANNABINOIDS

## STANDARDS, CRMS, COLUMNS, AND SOLVENTS FOR CANNABINOID ANALYSIS

Cannabinoids are a class of psychoactive and nonpsychoactive compounds produced in the Cannabis (marijuana) plant.

In recent years, these compounds have shown potential therapeutic efficacy in the treatment of pain, mood disorders, and inflammatory diseases. Since the concentration-to-potency of cannabinoids in Cannabis can fluctuate through various stages of plant growth and in different plant strains, it is imperative for patients that Cannabis cultivators ensure cannabinoid identity as well as consistent purity and concentration.<sup>1</sup>

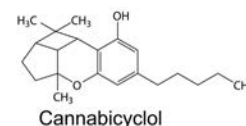
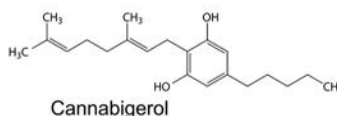
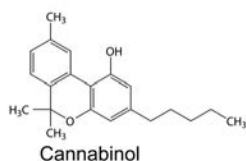
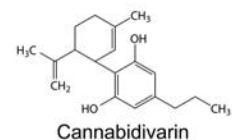
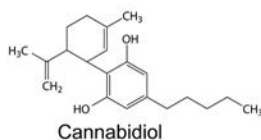
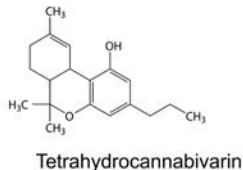
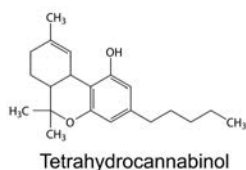
Cannabinoids can be analyzed using multiple methods including separation by GC, LC, or UHPLC, and identification by MS. We offer a complete line of relevant products for cannabinoid potency testing including GC and HPLC columns, solvents, and certified reference materials (CRMs).

Included here are examples relevant to cannabinoid testing.

## Standards and CRMs for Cannabinoid Analysis

### CANNABINOID STANDARDS AND CRMS

#### Structural formulas of main natural cannabinoids



Below is a list of products shown in the examples as well as others of relevance to cannabinoid testing workflows.

Component	Concentration	Solvent	Unit Size	Cat. No.
Cannabidiol-D <sub>3</sub>	100 µg/mL	Methanol	1 mL	C-084
Cannabidiol	1.0 mg/mL	Methanol	1 mL	C-045
Cannabinol	1.0 mg/mL	Methanol	1 mL	C-046
Cannabinol-D <sub>3</sub>	100 µg/mL	Methanol	1 mL	C-115
Cannabidivarin (CBDV)	1.0 mg/mL	Methanol	1 mL	C-140
Cannabigerol (CBG)	1.0 mg/mL	Methanol	1 mL	C-141
Cannabigerolic acid (CBGA)	1.0 mg/mL	Methanol	1 mL	C-142
Cannabichromene (CBC)	1.0 mg/mL	Methanol	1 mL	C-143
Cannabidiolic acid (CBDA)	1.0 mg/mL	Methanol	1 mL	C-144
(-)-Δ <sup>9</sup> -THC-D <sub>3</sub>	100 µg/mL	Methanol	1 mL	T-003
(-)-Δ <sup>9</sup> -THC	1.0 mg/mL	Methanol	1 mL	T-005
(-)-Δ <sup>9</sup> -THC-D <sub>3</sub>	1.0 mg/mL	Methanol	1 mL	T-011
(-)-Δ <sup>8</sup> -THC	1.0 mg/mL	Methanol	1 mL	T-032
exo-THC	1.0 mg/mL	Methanol	1 mL	T-033
Δ <sup>9</sup> -Tetrahydrocannabinolic acid A (THCA-A)	1.0 mg/mL	Methanol	1 mL	T-093
Tetrahydrocannabivarin (THCV)	1.0 mg/mL	Methanol	1 mL	T-094

#### References

1. Elsohly MA. *Marijuana and the Cannabinoids*. New Jersey, Humana Press Inc., 2007.

# Analysis of Cannabinoids by HPLC

## ASCENTIS EXPRESS COLUMNS FOR FAST HPLC

The rapid growth of the Cannabis testing market has prompted the development of equally rapid methods to analyze the active ingredients. Shown here are separations of various cannabinoids on Ascentis® HPLC columns. Cerilliant® CRMs provided reliable identification and quantification.

### Key Features and Benefits

- Maximize speed with sharp peaks even at ultra-high flow rates
- Extend column lifetime with rugged Fused-Core® particles
- Suitable for all HPLC, UHPLC, and LC-MS instruments
- Achieve UHPLC performance on traditional HPLC systems
- Available in 2.0, 2.7, and 5 µm particles

Based on innovative Fused-Core® particle technology, Ascentis® Express 2.7 µm provides the high speed and high efficiency of sub-2 µm particles, but at approximately half the backpressure for the same column length. This lower pressure means that Ascentis Express can be run on conventional HPLC and LC-MS systems, as well as midpressure or ultra-high pressure (UHPLC) systems. Lower pressure also means longer columns can be used for additional resolving power. Ascentis Express 2.7 µm particles offer these benefits over sub-2 µm particles, along with excellent column lifetime.

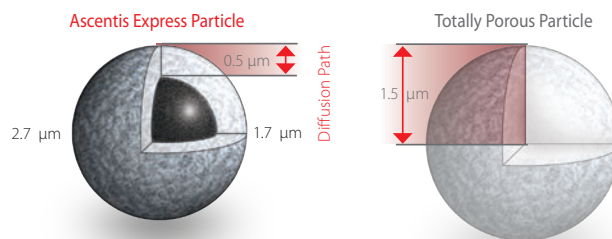
At the heart of Ascentis Express is the Fused-Core particle which comprises a solid core and a porous shell. Compared to totally porous particles, the Fused-Core particles have a much shorter diffusion path because of the solid core. This partial porosity reduces axial dispersion of solutes and minimizes peak broadening. Other features, such as a very tight particle size distribution and high packing density, result in Ascentis Express 2.7 µm particle columns that are capable of 240,000 N/m and higher: comparable to the efficiency of sub-2 µm particle columns and nearly twice the efficiency possible with 3 µm particles. The increased efficiency of Ascentis Express columns enables you to use shorter columns and save solvents.

- Ascentis Express 2 µm particles provide an optimized solution for high-throughput small molecule analysis.
- Ascentis Express 5 µm particles give exceptional "pressure-per-performance" ratio compared to both 5 µm and 3 µm totally porous particles.

### References

1. Elsohly MA. *Marijuana and the Cannabinoids*. New Jersey, Humana Press Inc., 2007.

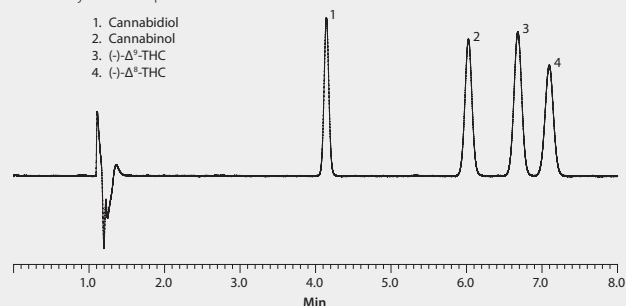
## Comparison of Fused-Core and Standard HPLC Particle (2.7 µm particle shown)



Shown below is the separation of Cannabis compounds on an Ascentis Express RP-Amide column. The selectivity, and therefore resolution, is unique to the amide bonded phase chemistry.

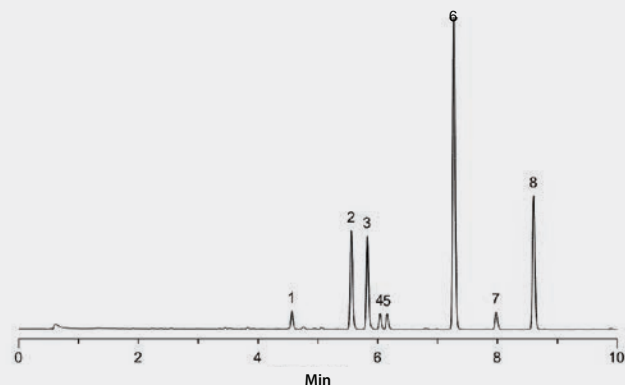
### Figure 1. HPLC Analysis of Cannabinoids using Ascentis® Express RP-Amide

column: Ascentis Express RP-Amide, 15 cm x 4.6 mm I.D., 5 µm particles (50774-U)  
mobile phase: 5 mM ammonium acetate (pH 4.5 with acetic acid) in 20:80, acetonitrile: water  
flow rate: 1.0 mL/min  
pressure: 1450 psi (100 bar)  
column temp.: 35 °C  
detector: UV, 214 nm  
injection: 5 µL



### Figure 2. HPLC Analysis of Cannabinoids using Ascentis® C18

column: Ascentis Express C18 10 cm x 2.1 mm, 2.0 µm particle size (50813-U)  
mobile phase: [A] 0.1% formic acid; [B] 0.1 % formic acid in acetonitrile  
flow rate: 0.4 mL/min  
pressure: 6300 psi  
column temp.: 35 °C  
detector: UV 280 nm  
injection: 1 µL



## Ascentis Express HPLC Columns (2.7 µm)

Component	Cat. No.
C18, 5 cm × 2.1 mm I.D.	53822-U
C18, 10 cm × 2.1 mm I.D.	53823-U
RP-Amide, 10 cm × 2.1 mm I.D.	53913-U
Phenyl-Hexyl 10 cm × 2.1 mm I.D.	53336-U
Phenyl-Hexyl 15 cm × 2.1 mm I.D.	53338-U

## HPLC & LC-MS Solvents

For a comprehensive overview on our solvents for HPLC & LC-MS applications, the LiChrosolv® & LiChrosolv® hypergrade for MS product lines, please visit us at [sigma-aldrich.com/lcms-solvents](http://sigma-aldrich.com/lcms-solvents)

# Analysis of Cannabinoids by GC

GC, with either flame ionization or MS, can be used for cannabinoid detection. Derivatization, typically silylation, is required for determination of cannabinoid acid species. We offer a full line of derivatization reagents in addition to a full line of GC columns. Below is a selection of our most popular products.

### Derivatization Reagents

Component	Cat. No.
Silylation Sampler Kit	505846
<i>N</i> -Methyl- <i>N</i> -(trimethylsilyl)trifluoroacetamide	69479
BSA Derivatization Grade	33037-U
BSTFA, Derivatization Grade for GC derivatization	33084
BSTFA + TMCS, 99:1	33148
HMDS + TMCS, 3:1:9 (Sylon™ HTP)	33038
TMSI, Derivatization Grade p	33068-U
Boron trifluoride-methanol solution	15716

## SLB-5MS CAPILLARY GC COLUMNS

- **Application:** The 5% phenyl equivalent phase provides a boiling point elution order with a slight increase in selectivity, especially for aromatic compounds. The low bleed characteristics, inertness, and durable nature make it the column of choice for anywhere a low bleed non-polar column is required
- **USP Code:** This column meets USP G27 and G36 requirements
- **Phase:** Bonded and highly crosslinked; silphenylene polymer virtually equivalent in polarity to poly(5% diphenyl/95% dimethyl siloxane)
- **Temp. Limits:** ≤0.32 mm I.D.: -60 °C to 340 °C (isothermal) or 360 °C (programmed)
- **Temp. Limits:** ≥0.53 mm I.D.: -60 °C to 330 °C (isothermal) or 340 °C (programmed)

### GC Columns

Component	Cat. No.
SLB-5ms, 10 m × 0.10 mm I.D., 0.10 µm	28465-U
SLB-5ms, 15 m × 0.10 mm I.D., 0.10 µm	28466-U
SLB-5ms, 20 m × 0.18 mm I.D., 0.18 µm	28564-U
SLB-5ms, 20 m × 0.18 mm I.D., 0.36 µm	28576-U
SLB-5ms, 30 m × 0.25 mm I.D., 0.25 µm	28471-U
SLB-5ms, 30 m × 0.25 mm I.D., 0.50 µm	28473-U



# Analysis of Cannabinoids by Thin Layer Chromatography

## THIN LAYER CHROMATOGRAPHY REAGENTS

Thin Layer Chromatography (TLC) has been used for many years to identify the presence of cannabinoids due to the speed and simplicity of the approach. We offer a full range of products for use in TLC. Below is a selection of our most popular products.

### LC Glass Plates from EMD/Merck KGaA

Length × Width	Layer Thickness	Binder / Indicator	Cat. No.
10 cm × 20 cm	250 µm	binder Polymeric / fluorescent indicator	Z293016
10 cm × 20 cm	250 µm	binder Polymeric / fluorescent indicator: No	Z292966
2.5 cm × 7.5 cm	250 µm	binder Polymeric / fluorescent indicator	Z740213
2.5 cm × 7.5 cm	250 µm	binder Polymeric / fluorescent indicator	Z740214
20 cm × 20 cm	250 µm	binder Polymeric / fluorescent indicator: No	Z292974
20 cm × 20 cm	250 µm	binder Polymeric / fluorescent indicator	Z293024
20 cm × 20 cm	250 µm	binder Polymeric / fluorescent indicator	Z293032
20 cm × 20 cm	NA	binder: No / fluorescent indicator	Z740215
5 cm × 10 cm	250 µm	binder Polymeric / fluorescent indicator	Z292990
5 cm × 10 cm	250 µm	binder Polymeric / fluorescent indicator	Z740212

For more TLC plates, please also visit us at [sigma-aldrich.com/tlc](https://sigma-aldrich.com/tlc)

### Solvents and Reagents for TLC

Description	Cat. No.
Chloroform, anhydrous, contains amylenes as stabilizer, ≥99%	372978
1,1-Dichloroethane	36967
Xylenes	214736
Dioxane	296309
Toluene	244511
Diethylamine	471216
Fast Blue BB Salt hemi(zinc chloride) salt	44670

# TERPENES

## STANDARDS, CRMS, COLUMNS, AND REAGENTS FOR TERPENE ANALYSIS

Terpenes are the primary aromatic constituents of Cannabis resin and essential oils. These compounds vary in type and concentration among different genetic lineages of Cannabis and have been shown to modulate and modify the therapeutic and psychoactive effects of cannabinoids.<sup>1</sup> Therefore, the analysis of terpenes is critical to ensuring Cannabis strain identity and medical efficacy.

Similar to cannabinoids, terpenes can be analyzed using multiple methods including separation by GC or LC and identification by MS. We offer a complete line of relevant products for terpene analysis for any matrix such as GC and HPLC columns, solvents, standards and certified reference materials.



## Standards and CRMs

Component	Unit Size	Cat. No.
Borneol*	100 mg	PHY89583 <sup>†</sup>
(+)-Fenchol	1 g	46198
Camphene*	100 mg	PHY80063 <sup>†</sup>
<i>trans</i> (-)-Caryophyllene	1 mL	75541
Cineole/Eucalyptol*	100 mg	PHY89195 <sup>†</sup>
(-)- $\beta$ -Elemene	25 mg	63965
<i>trans</i> - $\beta$ -Farnesene	1 mL	73492
Geraniol	1 mL, 5 mL	48798
Guaiol	250 mg	29242
Isoborneol	25 g, 50 g	I13901
Limonene	1 mL, 5 mL	62128
Linalool*	100 mg	PHY80885 <sup>†</sup>
Menthol*	100 mg	PHY89517 <sup>†</sup>
Mycrene	100 mg, 500 mg	64643
Nerol	1 mL	50949
Sabinene Hydrate	500 mg, 5 g	96573
Terpinolene	100 mL, 500 mL	86485
Valencene	25 g, 100 g	W34430
$\gamma$ -Terpinene	1 mL, 5 mL	86476
(-)- $\alpha$ -Bisabolol	1 mL	95426
(-)- $\alpha$ -Cedrene	1 mL	22133
Guaiene	250 mg	S456306
$\alpha$ -Humulene	1 mL, 5 mL	53675
$\alpha$ -Pinene*	100 mg	PHY89257 <sup>†</sup>
$\alpha$ -Terpinene	1 mL, 5 mL	86473
$\alpha$ -Terpineol*	100 mg	PHY89872 <sup>†</sup>
$\beta$ -Caryophyllene*	100 mg	PHY80717 <sup>†</sup>
$\beta$ -Eudesmol	10 mg, 50 mg	17790
$\beta$ -Pinene*	100 mg	PHY89335 <sup>†</sup>
$\Delta^3$ -Carene*	100 mg	PHY80088 <sup>†</sup>

<sup>†</sup>Distributed product – Products will be provided with the manufacturer's COA. Distributed items are available directly through Cerilliant in the U.S. and Canada. Supelco® and Cerilliant brands available through [sigma-aldrich.com](https://www.sigma-aldrich.com).

\*Cerilliant® Product

### Reference

1. Russo EB, "Taming THC: potential cannabis synergy and phytocannabinoid-terpenoid entourage effects." *Br J Pharmacol*. 2011 Aug;163(7):1344-64. doi: 10.1111/j.14765381.2011.01238.x



# Analysis of Terpenes by HPLC

## ASCENTIS EXPRESS COLUMNS FOR FAST HPLC

### Ascentis Express HPLC Columns (2.7 µm)

Component	Cat. No.
C18, 5 cm × 2.1 mm I.D.	53822-U
C18, 10 cm × 2.1 mm I.D.	53823-U
RP-Amide, 10 cm × 2.1 mm I.D.	53913-U
Phenyl-Hexyl 10 cm × 2.1 mm I.D.	53336-U
Phenyl-Hexyl 15 cm × 2.1 mm I.D.	53338-U

## Analysis of Terpenes by GC

GC, with either flame ionization or MS, can be used for terpene detection. Derivatization, typically silylation, is required for determination of relevant species. We offer a full line of derivatization reagents in addition to a full line of GC columns. Below is a selection of our most popular products.

- SLB-5ms 5% Phenyl-equivalent GC Capillary Columns provide classic boiling point elution order, with low bleed, inertness and durability. The classic workhorse nonpolar GC column.
- SLB-IL60 Ionic Liquid GC Capillary columns have more polar selectivity and higher maximum temperatures than PEG/wax columns.

### Derivatization Reagents

Component	Cat. No.
Silylation Sampler Kit	505846
<i>N</i> -Methyl- <i>N</i> -(trimethylsilyl)trifluoroacetamide	69479
BSA Derivatization Grade	33037-U
BSTFA, Derivatization Grade for GC derivatization	33084
BSTFA + TMCS, 99:1	33148
HMDS + TMCS, 3:1:9 (Sylon™ HTP)	33038
TMSI, Derivatization Grade p	33068-U
Boron trifluoride-methanol solution	15716

### GC Columns

Component	Cat. No.
SLB-5ms, 10 m × 0.10 mm I.D., 0.10 µm	28465-U
SLB-5ms, 15 m × 0.10 mm I.D., 0.10 µm	28466-U
SLB-5ms, 20 m × 0.18 mm I.D., 0.18 µm	28564-U
SLB-5ms, 20 m × 0.18 mm I.D., 0.36 µm	28576-U
SLB-5ms, 30 m × 0.25 mm I.D., 0.25 µm	28471-U
SLB-5ms, 30 m × 0.25 mm I.D., 0.50 µm	28473-U
SLB-IL60, 15 m × 0.10 mm I.D., 0.08 µm	29503-U
SLB-IL60, 20 m × 0.18 mm I.D., 0.14 µm	29504-U
SLB-IL60, 30 m × 0.25 mm I.D., 0.20 µm	29505-U



# PESTICIDE RESIDUE ANALYSIS

## SAMPLE PREPARATION, REAGENTS, STANDARDS, CRMS, COLUMNS, AND SOLVENTS FOR PESTICIDE ANALYSIS

Testing for pesticide residues is required in many states and strongly recommended in others. Cannabis is a challenging matrix for pesticide residue testing because of the oily nature of the plant and the high chlorophyll content. The QuEChERS extraction method followed by bulk SPE cleanup can efficiently and effectively extract pesticide residues from plant matrices resulting in increased accuracy of pesticide determination. Our analytical and chromatography division has been a pioneer in the development of SPE technology and was one of the first commercial providers of QuEChERS products.

## QuEChERS and SPE

### QUICK AND SIMPLE CLEANUP OF SAMPLES PRIOR TO CHROMATOGRAPHIC ANALYSIS



#### Features and Benefits

- Efficient and economic sample cleanup
- Pre-weighed amounts of sorbents and salts save labor and time
- High-purity reagents
- Convenient and reliable in ready-to-use 2 mL, 12 mL, and 15 mL centrifuge tubes

In QuEChERS methodology, the use of loose extraction salts and cleanup sorbents in combination with shaking and centrifugation results in a **Quick, Easy, Cheap, Effective, Rugged and Safe** sample cleanup technique. The QuEChERS method has emerged as a sample prep technique popular in the area of multi-residue pesticide analysis in food and agricultural products, and is formalized in method EN15662:2008 and AOAC 2007.01.<sup>1-2</sup>

The Supel™ QuE line of vials and centrifuge tubes contains pre-determined amounts of salts and SPE sorbents to support the most common method configurations used today for QuEChERS. The sorbents and their uses are listed in **Table 1**.

**Table 1. QuEChERS Cleanup Sorbents and Uses**

Sorbent	For the removal of:
Zirconia on silica	Pigments and lipids or fats
PSA on silica	Sugars, organic acids, fatty acids and polar pigments
C18 on silica	Lipids or fats
Carbon	Pigments

#### Supel QuE QuEChERS Products

Component	Cat. No.
Acetate Tube, pk of 50	55234-U
Citrate Extraction Tube, pk of 50	55227-U
Empty Tube, 50 mL, pk of 50	55248-U
QuEChERS Shaker and Rack Starter Kit	55278-U
Supel QuE PSA/C18/ENVI-Carb 2 mL Tube, pk of 100	55289-U
Supel QuE PSA/C18/ENVI-Carb 12 mL Tube, pk of 50	55286-U
Supel Que PSA/ENVI-Carb (EN) Tube 2	55176-U

#### Supel SPE Products

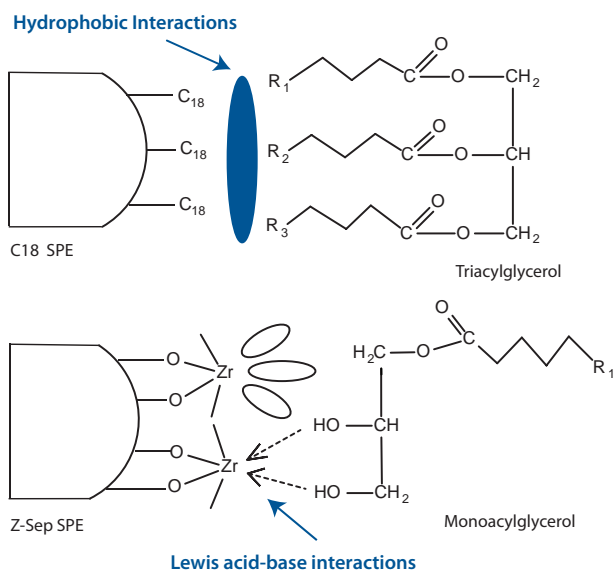
Component	Cat. No.
Supel™ Sphere Carbon/NH <sub>2</sub> SPE Tube	54283-U
Visiprep™ SPE Vacuum Manifold	57044
Disposable Liners for Visiprep DL Manifolds	57059

## Z-SEP SORBENTS: LIPID AND PIGMENT REMOVAL IN DIFFICULT MATRICES

- Significantly diminish fatty matrix interferences and some color due to pigments
- Provide more robust LC-MS and GC-MS methods by eliminating problematic matrix interferences
- Can replace C18 and PSA phases in current methods without additional method development

The patented zirconia-coated silica particles of Supel QuE Z-Sep sorbents selectively remove more fat and color from sample extracts than traditional phases for QuEChERS methods. Lipid retention is based on two synergetic interactions, as illustrated in **Figure 1**. The makeup and use of each sorbent in the Z-Sep family is summarized in **Table 2**.

**Figure 1. Retention Mechanism for Fats on Supel QuE Z-Sep Sorbents**



**Table 2. Supel QuE Z-Sep Sorbents, Composition and Uses**

Sorbent	Composition	For use with:
Z-Sep	Zirconia bonded to silica	Highly hydrophobic analytes such as PAHs and PCBs
Z-Sep/C18	Combination of Z-Sep and Discovery® DSC-18 particles	Samples with less than 15% fat content
Z-Sep+	Zirconia and C18 dual bonded to silica	Samples with greater than 15% fat content

### Z-Sep Sorbents

	Cat. No.		
	2 mL	15 mL	12 mL
Supel QuE Z-Sep Tube	55411-U	55491-U	55403-U
Supel QuE Z-Sep/MgSO <sub>4</sub>	55417-U	55503-U	55407-U
Supel QuE Z-Sep/C18 Tube	55284-U	55506-U	55401-U
Supel QuE Z-Sep+ Tube	55408-U	55486-U	55296-U
Supel QuE Z-Sep+/MgSO <sub>4</sub>	55414-U	55511-U	55406-U



# Columns for Pesticide Residue Analysis

## Ascentis® Express Columns for Fast HPLC

### Key Features and Benefits

- Maximize speed with sharp peaks even at ultra-high flow rates
- Extend column lifetime with rugged Fused-Core® particles
- Suitable for all HPLC, UHPLC, and LC-MS instruments
- Achieve UHPLC performance on traditional HPLC systems
- Available in 2.0, 2.7, and 5 µm particles

Based on innovative Fused-Core® particle technology, Ascentis® Express 2.7 µm provides the high speed and high efficiency of sub-2 µm particles, but at approximately half the backpressure for the same column length. This lower pressure means that Ascentis Express can be run on conventional HPLC and LC-MS systems, as well as midpressure or ultra-high pressure (UHPLC) systems. Lower pressure also means longer columns can be used for additional resolving power. Ascentis Express 2.7 µm particles offer these benefits over sub-2 µm particles, along with excellent column lifetime.

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- Ascentis Express 2 µm particles provide an optimized solution for high-throughput small molecule analysis.
- Ascentis Express 5 µm particles give exceptional “pressure-per-performance” ratio compared to both 5 µm and 3 µm totally porous particles.

### Ascentis Express HPLC Columns (2.7 µm)

Component	Cat. No.
C18, 5 cm × 2.1 mm I.D.	53822-U
C18, 10 cm × 2.1 mm I.D.	53823-U
C8, 10 cm × 2.1 mm I.D.	53832-U
RP-Amide, 10 cm × 2.1 mm I.D.	53913-U

### HPLC & LC-MS Solvents

For a comprehensive overview on our solvent for HPLC & LC-MS applications please visit us at [sigma-aldrich.com/lcms-solvents](https://sigma-aldrich.com/lcms-solvents)

## SLB®-5MS CAPILLARY GC COLUMNS

- **Application:** The 5% phenyl equivalent phase provides a boiling point elution order with a slight increase in selectivity, especially for aromatic compounds. The low bleed characteristics, inertness, and durable nature make it the column of choice anywhere a low bleed non-polar column is required
- **USP Code:** This column meets USP G27 and G36 requirements
- **Phase:** Bonded and highly crosslinked; silphenylene polymer virtually equivalent in polarity to poly(5% diphenyl/95% dimethyl siloxane)
- **Temp. Limits:** ≤0.32 mm I.D.: -60 °C to 340 °C (isothermal) or 360 °C (programmed)
- **Temp. Limits:** ≥0.53 mm I.D.: -60 °C to 330 °C (isothermal) or 340 °C (programmed)

### GC Columns

Component	Cat. No.
SLB-5ms, 10 m × 0.10 mm I.D., 0.10 µm	28465-U
SLB-5ms, 15 m × 0.10 mm I.D., 0.10 µm	28466-U
SLB-5ms, 20 m × 0.18 mm I.D., 0.18 µm	28564-U
SLB-5ms, 20 m × 0.18 mm I.D., 0.36 µm	28576-U
SLB-5ms, 30 m × 0.25 mm I.D., 0.25 µm	28471-U
SLB-5ms, 30 m × 0.25 mm I.D., 0.50 µm	28473-U
SLB-5ms, 30 m × 0.53 mm I.D., 0.50 µm	28541-U
SLB-5ms, 30 m × 0.53 mm I.D., 1.0 µm	28559-U

## Solvents for Pesticide Residue Analysis

To see our GC solvent offer, please visit us at [sigma-aldrich.com/GC-ECD-FID-MS-solvents](https://sigma-aldrich.com/GC-ECD-FID-MS-solvents)

For our HPLC & LC-MS solvent offer, please visit us at [sigma-aldrich.com/LCMS-solvents](https://sigma-aldrich.com/LCMS-solvents)

To learn more about our complete analytical reagent & solvent offer visit us at [sigma-aldrich.com/brighter](https://sigma-aldrich.com/brighter)



# Standards and CRMs

We offer an extensive line of pesticide certified reference materials (CRMs) and analytical grade standards, including for those pesticides most routinely monitored in Cannabis testing applications. The use of pesticides during any stage of Cannabis cultivation is prohibited in the United States and in many European countries.<sup>1</sup> To comply with regulatory agencies and to ensure the safety and therapeutic efficacy of Cannabis, Cannabis crops as well as Cannabis-derived products must be monitored for pesticide residues.

## Certified Reference Material

Component	Unit Size	Cat. No.
Aldrin	100 mg	49000-U
α-BHC	1,000 µg/mL	40100-U
β-BHC	50 mg	48494
δ-BHC	1,000 µg/mL	40103-U
Cypermethrin	50 mg	51991
p,p'-DDE	100 mg	34587
p,p'-DDT	1 g	31041
Deltamethrin	50 mg	05995
Diazinon	50 mg	68486
α-Endosulfan	100 mg	45468
β-Endosulfan	100 mg	33385
Endosulfan ether	100 mg	36673
D <sub>4</sub> -Endosulfan sulfate	100 mg	36676
Endosulfan I (α isomer)	1 g	SCE-003
β-Zearalanol	1 µg/mL in Acetonitrile	35407
Endosulfan II (β isomer)	25 mg	48578
Endosulfan sulfate	100 mg	36676
Malathion	50 mg	91481

## Reference

1. Sullivan N, Elzinga S, and Raber JC. "Determination of Pesticide Residues in Cannabis Smoke." *Journal of Toxicology*, vol. 2013, Article ID 378168, 6 pages, 2013. doi:10.1155/2013/378168

All pesticide CRMs are designed, produced, and verified for accuracy and stability in accordance with ISO/IEC 17025 and ISO Guide 34.

For more information and a list of our comprehensive offering of over 1,300 pesticide standards and our certified reference standards, visit:

[sigma-aldrich.com/pesticides](http://sigma-aldrich.com/pesticides)

## Analytical Standards

Component	Unit Size	Cat. No.
Acetamiprid	100 mg	39246
Acetamiprid-D <sub>3</sub>	50 mg	33674
Abamectin	100 mg	31732
α-BHC	1,000 µg/mL in Methanol	40100-U
Bifenthrin	100 ng/µL, in 2 mL Acetonitrile	36993
Bifenthrin	50 mg	32504
Carbaryl	100 ng/µL, in 10 mL Cyclohexane	36856
Dicofol	100 ng/µL, in 2 mL Methanol	45848
Imidacloprid	100 ng/µL, in 2 mL Acetonitrile	46341
Indoxacarb	25 mg	33969
Myclobutanil	100 mg	34360
Paclobutrazol	250 mg	46046
Permethrin	250 mg	45614
Propiconazole	250 mg	N13576
Pyridaben	25 mg	46047
Resmethrin	250 mg	45655
Tebuconazol	250 mg	32013
Tetramethrin	250 mg	45618
τ-Fluvalinate	100 mg	N13263
Thiamethoxam	100 mg	37924
Thiamethoxam-D <sub>3</sub>	25 mg	38176

# MYCOTOXINS ANALYSIS

## MATRIX PROCESSING, SAMPLE PREPARATION, REAGENTS, COLUMNS, AND KITS FOR MYCOTOXIN ANALYSIS

Due to unique morphological characteristics, medical Cannabis plants are more susceptible to fungal contamination than Cannabis plants grown for non-medical use.<sup>1</sup> The presence of fungi on Cannabis can result in the production of toxic, secondary fungal metabolites known as mycotoxins. These toxins, even in minute amounts at the ppb level, can pose a serious threat to pulmonary or immunocompromised patients.<sup>2</sup>

Mycotoxins are a diverse group of compounds comprised of hundreds of secondary metabolic products from various fungal species.

- Mycotoxins are increasingly prevalent
- Occur during growth, harvest, transportation, processing, or storage
- Several mycotoxins show marked toxicity in humans

The sensitive and accurate detection of very low levels of these compounds is critical to identify contaminated product.

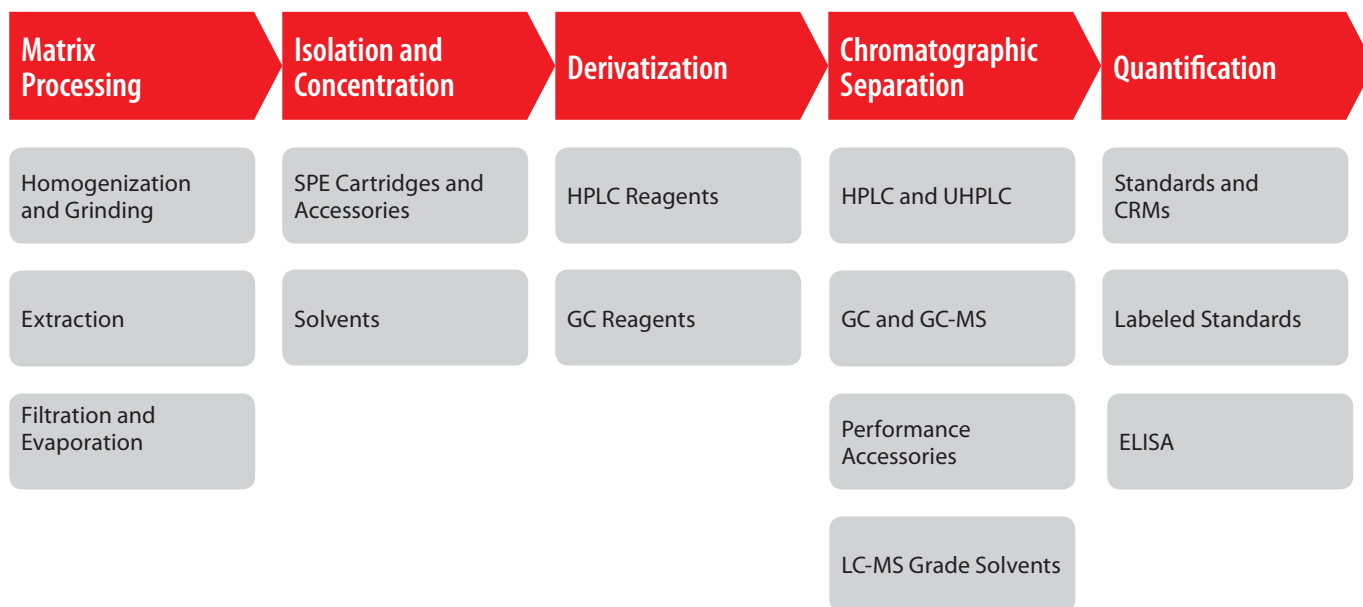
Chromatographic methods such as GC and HPLC are most commonly used for analysis, usually preceded by a number of operations such as sampling, sample preparation, extraction, and cleanup.

In recent years, we have developed a comprehensive range of products and methods for the analysis of mycotoxins.

Contact us if you need support with your application; we will be delighted to help.

## Mycotoxin Analysis Workflow

Our portfolio includes valuable products for the entire mycotoxin analysis workflow.



### References

1. Elsohly MA. *Marijuana and the Cannabinoids*. New Jersey, Human Press Inc., 2007
2. <http://www.fda.gov/FoodGuidanceRegulation/GuidanceDocumentsRegulatoryInformation/ChemicalContaminantsMetalsNaturalToxinsPesticides/ucm077969.htm>

# Mycotoxin ELISA Kits

Enzyme-linked immunosorbent assay (ELISA) is a simple and easy method for the analysis of mycotoxins. We have a comprehensive range of ELISA kits covering all major mycotoxin categories which is complementary to our analytical chromatography offering to quantify mycotoxins.

- Rapid and highly sensitive tests (30–90 minutes)
- Limit of detection down to 5 ppt
- Cost-effective compared to other tests on a cost-per-test basis
- No matrix effects

## ELISA Kits

Component	Cat. No.
Aflatoxin B <sub>1</sub> Low Matrix ELISA Kit	SE120002
Aflatoxin M <sub>1</sub> ELISA Kit for Urine	SE120005
Deoxynivalenol ELISA Kit	SE120009
Fumonisin ELISA Kit	SE120010
High Sensitivity Aflatoxin M <sub>1</sub> ELISA Kit	SE120004
Ochratoxin A ELISA Kit	SE120014
Rapid Aflatoxin B <sub>1</sub> ELISA Kit	SE120001
Rapid Aflatoxin M <sub>1</sub> ELISA Kit	SE120003
Total Aflatoxin ELISA Kit	SE120006
Total Aflatoxin ELISA Kit Low Matrix (Qual.)	SE120008
Total Aflatoxin ELISA Kit Low Matrix (Quan.)	SE120007
Zearalenone ELISA Kit	SE120016

# Isolation/Concentration (SPE)

## SUPEL™ TOX CARTRIDGES – INCREASE THROUGHPUT TEN-FOLD

The need for a quick, simplistic sample cleanup approach prior to chromatographic mycotoxin analysis has brought about SPE cartridges that significantly decrease sample prep time, increase reproducibility and are more user friendly as compared to immunoaffinity columns.



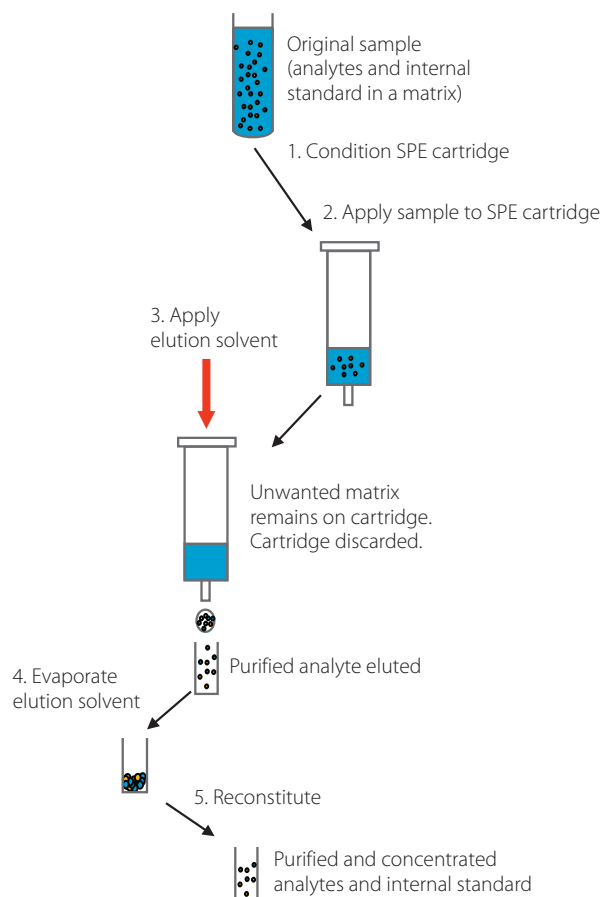
Unlike the multiple step “bind and elute” strategy implemented when using immunoaffinity columns, the Supel Tox AflaZea, DON, and Tricho SPE cartridges employ an “interference removal” strategy which saves time by eliminating wash steps prior to elution of aflatoxin and zearalenone, deoxynivalenol, and tricothecenes (type A and B), respectively. Cartridges removing interferences associated with analysis of fumonisins (B<sub>1</sub> and B<sub>2</sub>) and ochratoxin A are also available as a part of our Supel Tox product offering.

## Features and Benefits

- Remove interferences associated with mycotoxin analysis
- Better reproducibility than immunoaffinity columns
- Sample preparation time is up to ten-fold less than that of immunoaffinity columns (fewer steps)
- Straightforward, cost-effective, and quick methodology requiring little additional method development (generic method)
- Improved shelf life over immunoaffinity columns due to the thermally stable format. No refrigeration is required for shipping and storage.

Purification in six minutes with >85% recovery and <5% RSD for Aflatoxins in peanut paste. Easy to handle and store, no refrigeration required.

## Interference Removal Principle



## Comparison of Supel Tox AflaZea SPE to Immunoaffinity for Aflatoxins B<sub>1</sub>, B<sub>2</sub>, G<sub>1</sub> and G<sub>2</sub> in Peanut Paste

	Immunoaffinity	Supel Tox AflaZea SPE Cartridge
Sample Prep Time (post-extraction to pre-analysis)	<ul style="list-style-type: none"> <li>• 60 minutes</li> <li>• 8 samples/day (if processing 1 at a time)</li> </ul>	<ul style="list-style-type: none"> <li>• <b>6 minutes</b></li> <li>• 80 samples/day (if processing 1 at a time)</li> </ul>
Ease of Use	<ul style="list-style-type: none"> <li>• Large volumes of liquid</li> <li>• Controlled drop rates</li> <li>• Numerous complicated steps</li> <li>• Additional buffer salts required</li> <li>• Must be refrigerated, brought to room temp before use</li> </ul>	<ul style="list-style-type: none"> <li>• Small volumes of liquid</li> <li>• Vacuum filtration used</li> <li>• Steps few and not complicated</li> <li>• No additional reagents required</li> <li>• Column does not require special storage conditions</li> </ul>

## SPE ACCESSORIES

Visiprep™ DL (Disposable Liner) Vacuum Manifold eliminates the possibility of cross-contamination when processing a new sample on the same port.

To avoid long decontamination of your vacuum manifolds, use our Visiprep DL.



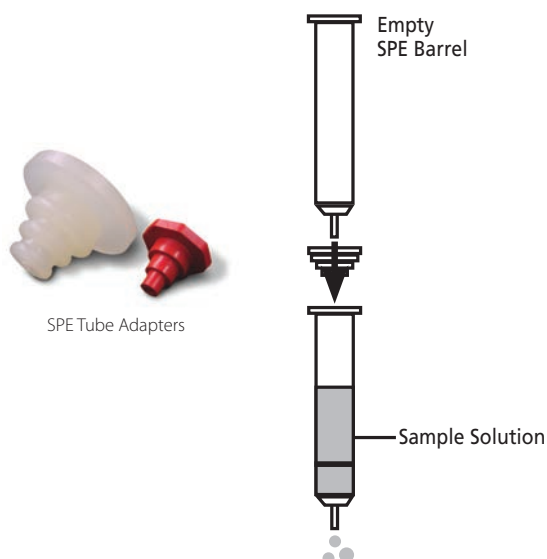
The liner consists of a PP female Luer hub that attaches to the SPE and thin-walled PTFE tubing that is threaded through the SPE port. This ensures that all SPE port and valve surfaces coming in contact with the sample can be replaced following each extraction.

Description	Cat. No.
DL (Disposable Liner), 12-port model	57044
DL (Disposable Liner), 24-port model	57265
Disposable Liners for Visiprep DL Manifolds (included with 57044 and 57265) - PTFE, pk of 100	57059
SPE Tube Adapters for 1, 3, 6 mL tubes, pk of 12	57020-U
Empty PP SPE Tube, 20 mL, pk of 12	57021

## Features and Benefits

- Patented screw-type valves within each SPE port for precise flow control
- Glass basin will not dissolve, fog, or discolor when exposed to solvents
- Leg covers enable cover to rest on work surface after removed from the manifold
- Various PP vessel racks for numerous type of glassware

To apply large volumes of sample, use our SPE tube adapters and reservoirs.





# Standards and CRMs

We offer a wide variety of certified reference materials (CRMs) and analytical standards for the mycotoxins most routinely monitored in medical Cannabis testing applications.

## Certified Reference Material

Component	Concentration	Solvent	Unit Size	Cat. No.
Aflatoxin B <sub>1</sub>	3.79 µg/g	Acetonitrile	4 mL	ERMAC057
Aflatoxin B <sub>1</sub>	3 µg/mL	Benzene: Acetonitrile (98:2)	1 mL	CRM46323
Aflatoxin B <sub>1</sub>	20 µg/mL	Methanol	1 mL	CRM44647
Aflatoxin B <sub>2</sub>	3.80 µg/g	Acetonitrile	4 mL	ERMAC058
Aflatoxin B <sub>2</sub>	3 µg/mL	Benzene: Acetonitrile (98:2)	1 mL	CRM46324
Aflatoxin G <sub>1</sub>	3.78 µg/g	Acetonitrile	4 mL	ERMAC059
Aflatoxin G <sub>1</sub>	3 µg/mL	Benzene: Acetonitrile (98:2)	1 mL	CRM46325
Aflatoxin G <sub>2</sub>	3.80 µg/g	Acetonitrile	4 mL	ERMAC060
Aflatoxin G <sub>2</sub>	3 µg/mL	Benzene: Acetonitrile (98:2)	1 mL	CRM46326
Aflatoxin M <sub>1</sub>	10 µg/mL	Acetonitrile	1 mL	CRM46319
Ochratoxin A	50 µg/mL	Benzene: Acetic Acid (99:1)	1 mL	CRM46912
Zearalanol	50 µg/mL	Acetonitrile	1 mL	CRM46916
Zearalenone	9.95 µg/g	Acetonitrile	4 mL	ERMAC699

## Analytical Standards: Acetonitrile Solvent

Component	Concentration	Unit Size	Cat. No.
Aflatoxin B <sub>1</sub>	2 µg/mL	2 mL	34029
Aflatoxin B <sub>1</sub> - <sup>13</sup> C <sub>17</sub>	0.5 µg/mL	1 mL	32764
Aflatoxin B <sub>2</sub>	0.5 µg/mL	2 mL	34034
Aflatoxin B <sub>2</sub> - <sup>13</sup> C <sub>17</sub>	0.5 µg/mL	1 mL	32771
Aflatoxin G <sub>1</sub>	2 µg/mL	2 mL	34032
Aflatoxin G <sub>1</sub> - <sup>13</sup> C <sub>17</sub>	0.5 µg/mL	1 mL	32772
Aflatoxin G <sub>2</sub>	0.5 µg/mL	2 mL	34033
Aflatoxin G <sub>2</sub> - <sup>13</sup> C <sub>17</sub>	0.5 µg/mL	1 mL	32777
Aflatoxin M <sub>1</sub>	0.5 µg/mL	2 mL	34031
Ochratoxin A	10 µg/mL	2 mL	34037
Ochratoxin B	10 µg/mL	2 mL	32411
α-Zearalanol	10 µg/mL	1 mL	35405
α-Zearalenol	10 µg/mL	1 mL	35406
β-Zearalanol	10 µg/mL	1 mL	35407
β-Zearalenol	10 µg/mL	1 mL	35409
Zearalenone- <sup>13</sup> C <sub>18</sub>	25 µg/mL	1 mL	32758



# Derivatization

## DERIVATIZATION FOR HPLC ANALYSIS

Derivatization is a suitable tool to make detection possible or to improve the detectability of an analyte. As a result, a modified analyte is produced emitting fluorescent light that is proportional to the amount of the analyte in the sample. When choosing an appropriate derivatization agent, a number of criteria should be considered:

- Derivatization should be rapid and quantitative
- By-products and excess reagents should not interfere with the formation of fluorescent light
- The fluorophore must possess intense absorption bands
- The reagent must be stable
- The analyte must be reactive with the derivatization agent
- The derivatizing agent and the by-products should not be fluorescent

The sensitivity of HPLC determination of trichothecenes is limited as many compounds show only minimal fluorescent or ultraviolet absorbing properties. Those with a conjugated C=O double bonding system (C9 and C10 and keto group at C8) can be detected, but derivatization is often applied as well, especially when detection is performed at ppb levels, because of interferences from impurities.

Description	Cat. No.
<i>N</i> -Heptafluorobutyrylimidazole (HFBI)	74382
<i>N</i> -Heptafluorobutyrylimidazole (HFBI)	74382
Sylon™ BTZ (1 × 25 mL)	33031-U
Sylon BTZ (144 × 0.1 mL)	33151
Sylon BTZ (20 × 1 mL)	33030
<i>o</i> -phthalaldehyde for fluorescence, ≥99.0% (HPLC)	79760
<i>o</i> -phthalaldehyde for fluorescence, ≥99.0% (HPLC)	79760

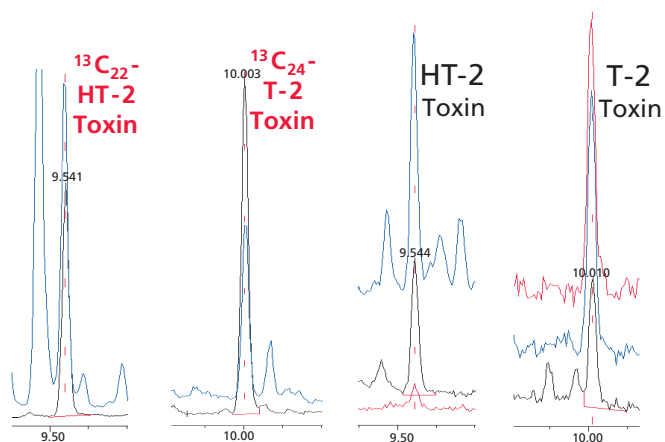
## DERIVATIZATION FOR GC ANALYSIS

When using GC, derivatization is necessary to provide volatility. Different derivatization reagents are available and chosen based on the analyte functional groups. Most methods are based on trimethylsilylation or fluoroacylation.

- A-trichothecenes are derivatized with heptafluorobutyrylimidazole (HFBI).
- Derivatization mixtures for B-trichothecenes include Tri-Sil® TBT and Sylon™ BTZ, which contain TMSI (40 ± 5%), BSA (35 ± 5%), and TMCS (25 ± 5%). This is to avoid two peaks caused by incomplete derivatization.
- Generally, fumonisins are derivatized with ortho-phthalaldehyde (OPA).

## SILYLATION USING MSTFA

Typically when using silylation, the excess of the silylating reagent had to be quenched by water followed by a re-extraction of the analyte. However, by using *N*-methyl-*N*-(trimethylsilyl) trifluoroacetamide (MSTFA) containing 1% trimethylchlorosilane as a silylating reagent, one can eliminate the water-quenching and the re-extraction steps because the relatively low boiling point (131 °C) allows for the use of MSTFA as a solvent for splitless injection.



MSTFA eliminates the water-quenching and re-extraction steps.

For more information or to request a Derivatization Reagents Brochure, visit [sigma-aldrich.com/derivatization](https://sigma-aldrich.com/derivatization)

# Chromatographic Analysis

Coupling a suitable extraction with the right cleanup procedure, an optimized chromatographic separation, and a selective derivatization can achieve an optimum level of specificity for reliable quantification.

Depending on lab resources, mono-residues or multi-residues analysis can be performed. GC can be used with ECD, FID, or MS detection or HPLC with fluorescence, UV, or MS/MS detection.

## GAS CHROMATOGRAPHY

For GC analysis, a robust stationary phase with medium polarity such as a poly (diphenyl 35%/dimethylsiloxane 65%) phase is required to withstand the effects of the silylating agent and temperature. A very good separation of T-2 toxin and HT toxin can be achieved with the application of a fast-temperature program. MS detection with selected ion monitoring (SIM) enables detection limits in the range of 2–5 ppb for HT-2 toxin and T-2 toxin, even in complex matrixes. As claimed by EU Guideline 96/23/EG, the identities of the toxins were confirmed, not only by the retention time, but also by the three SIM ions. A qualifier ion was measured for each internal standard to ensure peak purity.

### SPB®-35 Capillary GC Columns

Description	Cat. No.
30 m × 0.25 mm, I.D. 0.25 µm	24092
60 m × 0.25 mm, I.D. 0.25 µm	28568-U
30 m × 0.32 mm, I.D. 0.25 µm	24094
30 m × 0.53 mm, I.D. 1.00 µm	25335

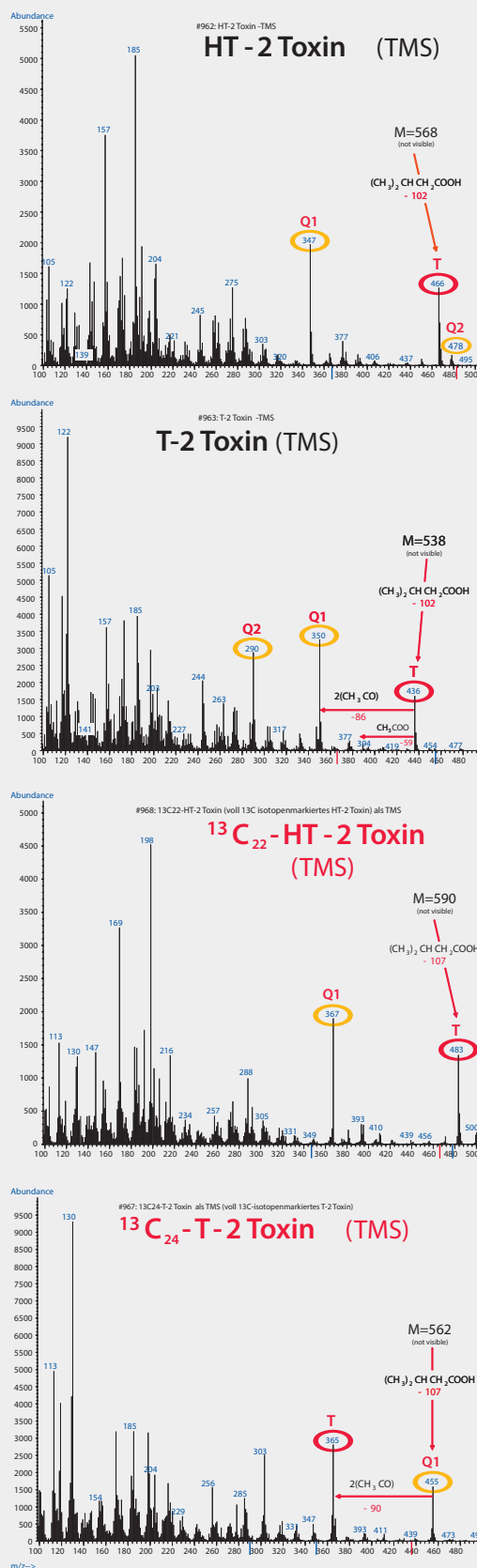
## GC-MS Method for Mycotoxin Analysis Using <sup>13</sup>C-labeled Mycotoxin Derivatives Using IDMS

Andreas Breidbach and Wolfgang Brodacz developed a new GC/MS method using fully <sup>13</sup>C-isotope-labeled analogues of T-2 toxin and HT-2 toxin that allows for the detection of these toxins at concentrations as low as 2–5 ppb.

Isotopic dilution mass spectrometry (IDMS) takes advantage of the fact the chemical and physical properties of <sup>13</sup>C isotope-labeled analogues are nearly identical to those of non-labeled analytes. This means that their behavior in the workup is essentially the same, but the labeled and non-labeled analogues can still be distinguished by mass spectrometry (Figure 1).

- Labeled mycotoxins allow IDMS for increased precision and accuracy
- Simple sample handling
- Addition of the standard after sample extraction compensates for matrix effects and inaccuracies

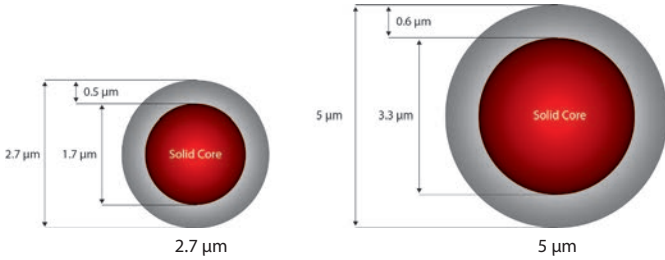
Figure 1. EI-Mass spectra of the TMS-derivatives of unlabeled and fully <sup>13</sup>C-isotope-labeled T-2 Toxin and HT-2 Toxin



# LIQUID CHROMATOGRAPHY

## Analysis of Mycotoxins by LC/MS/MS

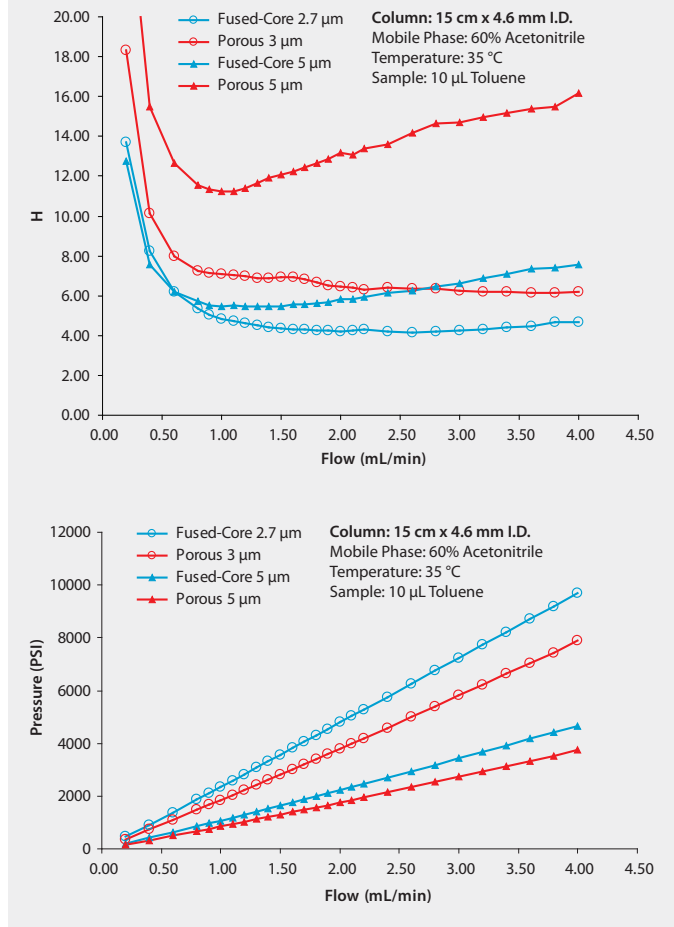
The sensitive and accurate detection of very low levels of mycotoxins is critical to identify contaminated products. LC/MS/MS is a popular analytical technique for this purpose. The combination of LC with MS/MS detection allows the quantification of multiple mycotoxins in the same run. Several different phase selectivities are required to achieve this separation. Using different phases can also decrease the analysis speed.



The 5 µm Ascentis® Express Fused-Core® particle achieves much higher efficiency than fully porous 5 µm particles at comparable pressures. It operates at the same efficiency levels or better than 3 µm particles and is well-suited for high performance with routine 400 bar instruments, while maintaining good analyte retention and loading capacity.

These columns are used at a higher flow rate than the equivalent porous ones, without sacrificing efficiency, on your conventional HPLC system. The tables below demonstrate ways to accelerate systems and save solvent. However, to analyze multiple mycotoxins in the same run, one could face problems in the resolution of some on C18 columns.

Figure 2. Effect of Flow Rate on Performance and Pressure



Current Method		Efficiency (p/col)				
Pressure	Flow Rate	Column Length	5 µm Particles Totally Porous	3 µm Particles Totally Porous	1.8 µm Particles Totally Porous	Ascentis Express Fused-Core 5 µm
4,000 psi	1.0 mL/min	5 cm	4,500	6,000	<b>max. 12,000</b>	7,000
		10 cm	9,000	12,000		15,300
		15 cm	13,500	18,000		<b>max. 23,000</b>
		20 cm	18,000	<b>max. 24,000</b>		
		25 cm	<b>max. 22,500</b>			

- Red box: Back Pressure reaches the limits of conventional HPLC systems
- Green box: Back Pressure adapted to conventional HPLC systems

Analyze 15 mycotoxins in the same run in less than 10 minutes, decrease your solvent consumption by 3-fold without the need of a UHPLC system.

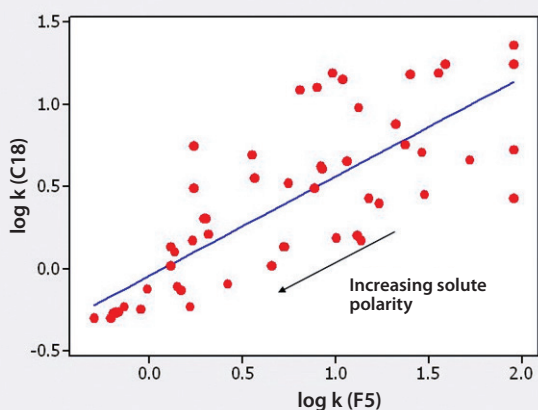
Particle Size	Flow Rate	Column Length	Efficiency (N)	Retention Time
5 µm	1.0 mL/min	25 cm	22,500	25.0 min
5 µm FC	1.0 mL/min	15 cm	23,000	8.0 min
2.7 µm	2.4 mL/min	10 cm	24,000	4.2 min

FC = Fused Core

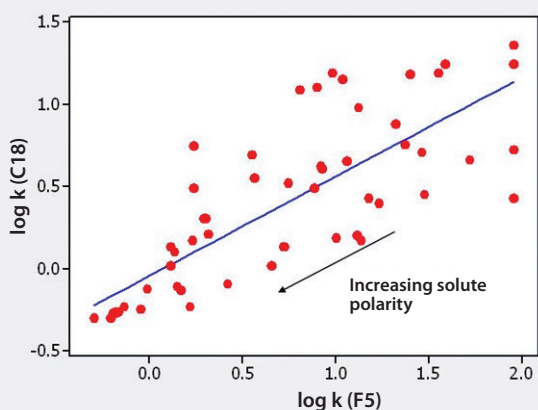


Improvements in resolution can also be achieved by changing the selectivity of the column stationary phase. Although we offer seven different chemistries within the Ascentis® Express range, the F5 and RP-Amide are especially efficient for the analysis of mycotoxins because they provide both polar and ionic interactions. These two columns present orthogonality versus C18 and have been used to separate 15 mycotoxins in the same analytical run with MS/MS detection (Figures 3–5).

**Figure 3. Selectivity differences between Ascentis Express chemistries: RP-Amide vs. C18**



**Figure 4. Selectivity differences between Ascentis Express chemistries: C18 vs. F5**



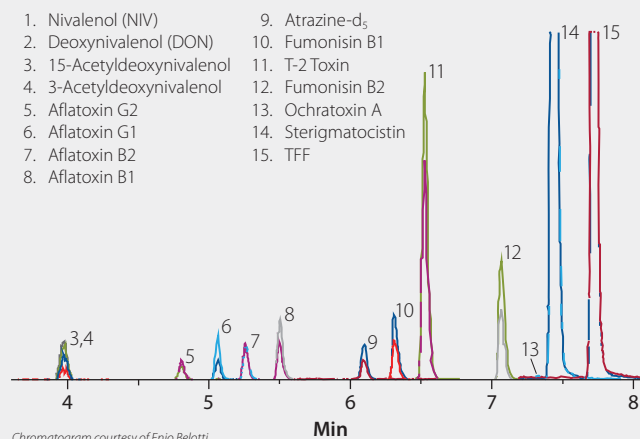
### Ascentis Express Columns

Description	Cat. No.
C18, 10 cm × 2.1 mm I.D., 2.7 μm	<a href="#">53823-U</a>
C18, 15 cm × 2.1 mm I.D., 2.7 μm	<a href="#">53825-U</a>
RP-Amide, 10 cm × 2.1 mm I.D., 2.7 μm	<a href="#">53913-U</a>
RP-Amide, 15 cm × 2.1 mm I.D., 2.7 μm	<a href="#">53914-U</a>
F5, 10 cm × 2.1 mm I.D., 2.7 μm	<a href="#">53569-U</a>
F5, 15 cm × 2.1 mm I.D., 2.7 μm	<a href="#">53571-U</a>

**Figure 5.**

### Ascentis Express RP-Amide

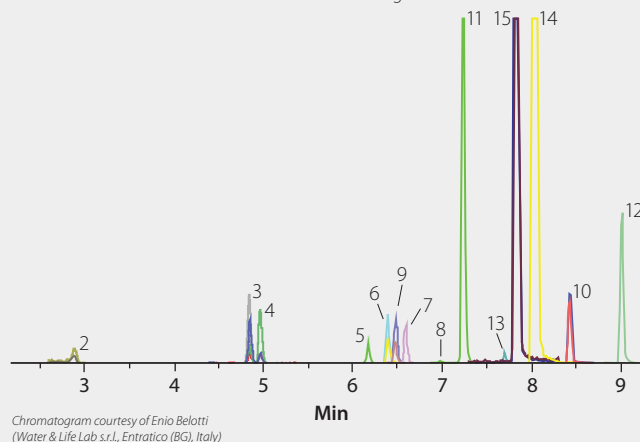
sample/matrix: 5 grams of cereal spiked with 15 mycotoxins; extract with 20 mL acetonitrile:1% formic acid in water (75:25); shake for 1 min; centrifuge; filter through a 0.45 μm syringe filter  
 column: Ascentis Express RP-Amide, 10 cm × 2.1 mm I.D., 2.7 μm particles ([53913-U](#))  
 mobile phase: (A) 1 mM ammonium acetate, 0.5% acetic acid in water; (B) 1 mM ammonium acetate, 0.5% acetic acid in methanol  
 gradient: 0 min: 5% B; 0.5 min: 10% B; 12 min: 95% B; 15 min: 95% B  
 flow rate: 400 μL/min  
 column temp.: 40 °C  
 detector: MS/MS, ESI(+) and ESI (-)  
 injection: 2 μL



### Ascentis Express F5

column: Ascentis Express F5, 10 cm × 2.1 mm I.D., 2.7 μm particles ([53569-U](#))

Peak IDs and all other conditions the same as Figure 1.



For additional details on the Ascentis Express HPLC column range or to request an application note, visit [sigma-aldrich.com/express](http://sigma-aldrich.com/express)

## SOLVENTS FOR SPE, LLE, AND ANALYSIS

We offer a comprehensive range of high-quality solvents for dedicated analytical applications.

Each part of the workflow requires different grades of solvent. The table below provides the suggested solvent grade that should be used within each stage of an application.

### Minimum Grade Suitability

Application	Grade
SPE	LiChrosolv®, SupraSolv®, UniSolv®
HPLC	LiChrosolv®
LC-MS	LiChrosolv® hypergrade for LC-MS
GC and GC-MS applications	SupraSolv®, UniSolv®

Complete details and specifications for these solvents can be found at [sigma-aldrich.com/solvents](http://sigma-aldrich.com/solvents)

To use the solvent selector tool, visit [sigma-aldrich.com/solvent\\_selector](http://sigma-aldrich.com/solvent_selector)

Choose the right quality of solvent for accurate and reproducible results. Depending on your detection mode, use the corresponding solvent for the extraction step.

### Common Extraction Solvents

Mycotoxins	Extraction Solvents
Aflatoxins	Acetonitrile/water Methanol/water
Type A Trichothecenes	Acetonitrile/water Methanol/water
Type B Trichothecenes	Acetonitrile/water Water/PEG Chloroform/methanol
Zearalenone	Ethyl acetate Methanol Acetonitrile Chloroform and mixtures thereof
Moniliformin	Methanol Acetonitrile/water Water Water/tetrabutylammonium hydroxide (TBAH)
Beauvericin	Acetonitrile/water Methanol
Ochratoxin A	Methyl <i>tert</i> -butyl ether (MTBE) Chloroform Acetonitrile/water mixtures of toluene/HCl, MgCl <sub>2</sub>
Fumonisin	Methanol/water (3:1) Acetonitrile/water (1:1)
Patulin	Ethyl acetate Acetone



# RESIDUAL SOLVENTS

## COLUMNS, SOLVENTS, AND CRMS FOR RESIDUAL SOLVENT ANALYSIS

Cannabis concentrates and blends are often extracted using organic solvents that can negatively impact product quality, potency, and human health. These solvents are difficult to visually detect and necessitate the use of chromatographic techniques such as GC Headspace.

We offer a complete line of products for the analysis of residual solvents in Cannabis testing including columns, solvents, and certified reference materials (CRMs).

Residual solvent CRMs are designed, produced, and verified for accuracy and stability in accordance with ISO/IEC 17025 and ISO Guide 34. Cerilliant® residual solvent CRMs are also NIST Traceable, manufactured in accordance with ISO 13485, and compliant with ISO 17511 and ISO 15194.

## Residual Analysis Optimized for Static Headspace GC Applications

Static headspace GC (GC-SH) is a technique used to concentrate volatile analytes prior to analysis. It can improve detection of low levels of volatile analytes and minimizes matrix interference by eliminating the need to inject the sample directly.

### HEADSPACE GRADE SOLVENTS

When developing a GC-SH method, such parameters as sample solvent, extraction temperature, extraction time, sample volume and headspace volume are optimized. The used solvent is playing here a pivotal role.

#### GC-HS Solvents

Description	Boiling Points	Cat. No.
Water for headspace gas chromatography SupraSolv®	100	1.00577
N,N-Dimethylformamide for headspace gas chromatography SupraSolv®	155	1.00202
N,N-Dimethylacetamide for headspace gas chromatography SupraSolv®	165	1.00399
Dimethyl sulfoxide for headspace gas chromatography SupraSolv®	189	1.01900
1-Methyl-2-pyrrolidone for headspace gas chromatography SupraSolv®	202	1.02497
Benzyl alcohol for headspace gas chromatography SupraSolv®	203	1.02695

SupraSolv® headspace is our range of solvents specially designed for headspace gas chromatography. Headspace GC is the preferred method for the analysis of residual solvents in actives, excipients, and drug products according to Ph Eur and USP.

Accurate analysis with headspace gas chromatography requires the use of very pure solvents with extremely low concentrations of the defined residual solvents. We ensure this high purity through special, state-of-the-art production processes, and we specify for our high-purity SupraSolv® headspace solvents the concentrations of all the residual solvents classified in the relevant ICH guideline Q3C "Impurities: Guideline for Residual Solvents".

Moreover, we perform application tests on every single batch of SupraSolv® headspace – thus we are able to consistently deliver you the reliability, accuracy and analytical safety you need.

# Standards and CRMs

## Multicomponent Alcohol Mix

Components	Description	Cat. No.
<ul style="list-style-type: none"> <li>Acetone</li> <li>Ethanol</li> <li>Isopropanol</li> <li>Methanol</li> </ul>	in Water Each at 100 µg/mL; Unit Size: 3 × 1.2 mL	<a href="#">A-076</a>
	in Water Each at 5 µg/mL; Unit Size: 3 × 1.2 mL	<a href="#">A-057</a>
	in Water Each at 1,000 µg/mL; Unit Size: 3 × 1.2 mL	<a href="#">A-056</a>
	in Water Each at 4,000 µg/mL; Unit Size: 3 × 1.2 mL	<a href="#">A-061</a>

## Residual Solvents Mixture – Class I

Components	Description	Cat. No.
<ul style="list-style-type: none"> <li>Benzene, Carbon tetrachloride</li> <li>1,1-Dichloroethene</li> <li>1,2-Dichloroethane</li> <li>1,1,1-Trichloroethane</li> </ul>	in DMSO Each at Varied Concentrations Unit Size: 3 × 1.2 mL	<a href="#">PHR1063</a>

## Residual Solvents Mixture – Class IIA

Components	Description	Cat. No.
<ul style="list-style-type: none"> <li>Acetonitrile (2.05 mg/mL)</li> <li>Chlorobenzene (1.8 mg/mL)</li> <li><i>trans</i>-1,2-Dichloroethylene</li> <li><i>cis</i>-1,2-Dichloroethene</li> <li>Dichloromethane</li> <li>1,4-Dioxane</li> <li>Ethylbenzene</li> <li>Methanol</li> <li>Methylcyclohexane</li> <li>Tetrahydrofuran</li> <li>Toluene</li> <li><i>p</i>-Xylene</li> <li><i>m</i>-Xylene</li> <li><i>o</i>-Xylene</li> </ul>	in DMSO Each at Varied Concentrations Unit Size: 3 × 1.2 mL	<a href="#">PHR1064</a>

## Residual Solvents Mixture – Class IIB

Components	Description	Cat. No.
<ul style="list-style-type: none"> <li>Chloroform (60 µg/mL)</li> <li>Nitromethane (50 µg/mL)</li> <li>Hexane (290 µg/mL)</li> <li>Tetralin (100 µg/mL)</li> <li>1,2-Dimethoxyethane (100 µg/mL)</li> <li>Pyridine (200 µg/mL)</li> <li>3-Methyl-2-pentanone (50 µg/mL)</li> <li>Trichloroethylene (80 µg/mL)</li> </ul>	in DMSO Each at Varied Concentrations Unit Size: 3 × 1.2 mL	<a href="#">PHR1065</a>

## Residual Solvents Mixture – Class IIC

Components	Description	Cat. No.
<ul style="list-style-type: none"> <li><i>N,N</i>-Dimethylacetamide (5.45 mg/mL)</li> <li>Formamide (1.1 mg/mL)</li> <li>2-Ethoxyethanol (0.8 mg/mL)</li> <li><i>N</i>-Methylpyrrolidone (2.65 mg/mL)</li> <li><i>N,N</i>-Dimethylformamide (4.4 mg/mL)</li> <li>2-Methoxyethanol (0.25 mg/mL)</li> <li>Ethylene glycol (3.1 mg/mL)</li> <li>Sulfolane (0.8 mg/mL)</li> </ul>	in DMSO Each at Varied Concentrations Unit Size: 3 × 1.2 mL	<a href="#">PHR1066</a>



# TRACE METAL ANALYSIS

Certain heavy metals are toxicants that cause adverse effects on human health. Toxic heavy metals such as arsenic, cadmium, lead, and mercury are persistent once released into the environment and can accumulate in Cannabis plants. Cannabis-based products such as edibles, oils, tinctures and salves must, therefore, be tested for the presence of heavy metals to ensure patient safety and product quality. We offer an extensive line of products for the heavy metals most routinely monitored in medical Cannabis testing applications.

TraceCERT® CRMs are produced and certified in accordance with ISO Guide 34 and ISO/IEC 17025.

## Standards and CRMs

For a complete list of the heavy metal analytical standards we offer, visit:

[sigma-aldrich.com/inorganiccrm](https://sigma-aldrich.com/inorganiccrm)

### TraceCERT® Standard Solutions for ICP

Component	Concentration	Solvent	Unit Size	Cat. No.
Arsenic (As)	1,000 mg/L	Nitric Acid	100 mL	01969
Cadmium (Cd)	1,000 mg/L	Nitric Acid	100 mL	36379
Lead (Pb)	1,000 mg/L	Nitric Acid	100 mL	41318
Mercury (Hg)	1,000 mg/L	Nitric Acid	100 mL	28941

## High Purity Reagents for Ultra-Trace Analysis

### Suprapur® and Ultrapur

Suprapur® acids and bases are ideal for trace elemental analysis e.g. AAS and ICP in the ppb range. Ultrapur acids and bases meet the highest demands of ultra-trace analysis in the ppt range. A selection is shown below.

For a comprehensive product overview please refer to "Sampling and Sample Preparation/Digestion and Dissolution Reagents (Acids, Bases, Salts)" at [sigma-aldrich.com/traceanalysis](https://sigma-aldrich.com/traceanalysis)

Product Description	Cat. No.
Acetic acid (glacial) 100% Suprapur®	1.00066
Ammonia solution 25 % Suprapur®	1.05428
Hydrobromic acid 47% Suprapur®	1.00306
Hydrochloric acid 30% Suprapur®	1.00318
Hydrogen peroxide 30% Suprapur®	1.07298
Nitric acid 65% Suprapur®	1.00441
ortho-Phosphoric acid 85% Suprapur®	1.00552
Sulfuric acid 96% Suprapur®	1.00714
Nitric acid 60% Ultrapur	1.01518
Water Ultrapur	1.01262

### TraceCERT® Multi-element Standard Solution I for ICP

Components	Description	Cat. No.
<ul style="list-style-type: none"><li>Aluminum (Al), 50 mg/L</li><li>Boron (B), 50 mg/L</li><li>Barium (Ba), 10 mg/L</li><li>Sodium (Na), 50 mg/L</li><li>Bismuth (Bi), 100 mg/L</li><li>Calcium (Ca), 10 mg/L</li><li>Cadmium (Cd), 10 mg/L</li></ul>	<ul style="list-style-type: none"><li>Nickel (Ni), 50 mg/L</li><li>Cobalt (Co), 10 mg/L</li><li>Iron (Fe), 10 mg/L</li><li>Magnesium (Mg), 10 mg/L</li><li>Lead (Pb), 100 mg/L</li><li>Chromium (Cr), 50 mg/L</li><li>Potassium (K), 100 mg/L</li></ul>	<ul style="list-style-type: none"><li>Manganese (Mn), 10 mg/L</li><li>Strontium (Sr), 10 mg/L</li><li>Copper (Cu), 10 mg/L</li><li>Lithium (Li), 50 mg/L</li><li>Molybdenum (Mo), 50 mg/L</li><li>Thallium (Tl), 50 mg/L</li><li>Zinc (Zn), 10 mg/L</li></ul>
in 10% Nitric Acid at Varied Concentrations Unit Size: 100 mL		90243

### TraceCERT® Multi-element Standard Solution IV for ICP

Components	Description	Cat. No.
<ul style="list-style-type: none"><li>Aluminum (Al), 40 mg/L</li><li>Zinc (Zn), 10 mg/L</li><li>Chromium (Cr), 20 mg/L</li><li>Iron (Fe), 100 mg/L</li><li>Arsenic (As), 40 mg/L</li><li>Beryllium (Be), 10 mg/L</li></ul>	<ul style="list-style-type: none"><li>Cobalt (Co), 10 mg/L</li><li>Lead (Pb), 40 mg/L</li><li>Barium (Ba), 40 mg/L</li><li>Boron (B), 100 mg/L</li><li>Copper (Cu), 20 mg/L</li><li>Manganese (Mn), 10 mg/L</li></ul>	<ul style="list-style-type: none"><li>Nickel (Ni), 20 mg/L</li><li>Cadmium (Cd), 10 mg/L</li><li>Thallium (Tl), 100 mg/L</li><li>Selenium (Se), 100 mg/L</li><li>Zinc (Zn), 100 mg/L</li><li>Vanadium (V), 40 mg/L</li></ul>
in 10% Nitric Acid at Varied Concentrations Unit Size: 100 mL		51844



# Lab Equipment Suitable for Inorganic Trace Analysis

## NALGENE® FEP BOTTLES

- Maximum chemical and corrosion resistance at high and low temperatures
- Excellent for trace metal analysis, storage of high-purity samples, and use with organic solvents
- Autoclavable. Leakproof Tefzel® ETFE screw closures

Description	Cat. No.
Narrow-mouth, capacity 30 mL, clear bottle	<a href="#">Z130230</a>
Narrow-mouth, capacity 60 mL, clear bottle	<a href="#">Z130249</a>
Narrow-mouth, capacity 125 mL, clear bottle	<a href="#">Z130265</a>
Wide-mouth, capacity 125 mL, clear bottle	<a href="#">Z130370</a>



## MILLEX® SYRINGE FILTER UNITS

- Pore size 0.45µm
- Non-sterile, disposable

Description	Cat. No.
PVDF membrane	<a href="#">Z227366</a>
PTFE membrane	<a href="#">Z227412</a>



## FORTUNA® UNIVERSAL OPTIFIX® HF DISPENSERS

- For dispensing hydrofluoric acid in addition to normal aqueous solutions, acids and crystallizing liquids
- Valve block made of PTFE, no metal springs in the valve = no corrosion

Description	Cat. No.
HF model, 2–10 mL volume	<a href="#">Z260266</a>
1–5 mL dispensing volume	<a href="#">Z678783</a>



# TOOLS FOR SAMPLE PREPARATION AND HANDLING

## Matrix Processing

A range of well-developed techniques is available. The criteria for choosing a suitable method include available time and equipment, specificity, and sensitivity.

### HOMOGENIZATION AND GRINDING



#### Homogenization

Homogenization is an important first step because mycotoxins are formed by mold that can occur in isolated pockets of materials. To ensure accurate test results, the sample should be representative of the lot from which is was obtained.

Description	Cat. No.
IKA ULTRA-TURRAX disperser tubes, DT-20 dispersing tube with rotor-stator element, 25/CS	Z722375

#### ULTRA-TURRAX Tube Drive Workstation



The workstation consists of an ULTRA-TURRAX Tube Drive: 2 x ST-20 (Z722383), 2 x DT-20 (Z722367), a removal hook for disengagement of the rotor-stator unit, 2 x BMT-20 G / S (Z722405 and Z722421) and a power supply.

The workstation provides a unique and universal dispersing, stirring, homogenizing and grinding system, with hermetically sealable and disposable sample tubes.

- Disperse, stir, homogenize, and grind using a single drive unit
- Hermetically sealable disposable sample tubes eliminate cross-contamination
- Gamma-sterilized tubes
- Tubes (2–15 mL and 15–50 mL) with pierceable membrane covers
- Anti-locking function and chemical-resistant plastic



#### Grinding

The grinding of solid samples is essential to ensure precise analysis. Proper grinding leads to the homogeneity and desired fineness of the sample.

The type of mill used depends on the properties of the matrix and the quantity of the sample.

For example, brittle materials are ground with a beater, fibrous materials with a blade and hard materials are ground with a special metal cutter while small and large sample quantities are generally ground with a batch or inline mills, respectively.

A continuously operating grinder with a powerful drive, an easy-to-clean working surface made of stainless steel, and easily changeable heads.

Description	Cat. No.
MF 10 basic Microfine grinder drive – grinding heads not included with delivery	Z645176
MF 10.1 Cutting-grinding head (for crushing fibrous substances)	Z645249
MF 10.2 Impact grinding head (for crushing brittle, hard materials)	Z645257

#### Waring Laboratory

Description	Cat. No.
Waring® laboratory blender one speed, capacity 1.2 L, glass container	Z272205
Waring® laboratory blender one speed, capacity 1 L, stainless steel container	Z272213
Waring® laboratory blender variable speed, capacity 1.2 L, glass container	Z272183
Waring® laboratory blender variable speed, capacity 1 L, stainless steel container	Z272191
Waring® laboratory blender three speed, heavy duty capacity 4 L, stainless steel container	Z272221



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